

CARBOFURAN (096)**EXPLANATION**

Carbofuran, a systemic acaricide, insecticide and nematicide, was first evaluated in 1967 and reviewed in 1979, 1991 and 1993. The Ad Hoc Working Group on Priorities of the CCPR in 1993 proposed carbofuran for re-evaluation, as the ADI was established in 1982 (ALINORM 93/24A para 251). It was scheduled for toxicological review in 1996 by the 1994 CCPR (ALINORM 95/24 Appendix VI) and for residue review in 1997 by the 1995 CCPR (ALINORM 95/24A, Appendix IV).

The toxicology of carbofuran was re-evaluated by the Joint Meeting in 1996. An ADI of 0-0.002 mg/kg bw was allocated on the basis of the NOAEL for erythrocyte acetylcholinesterase inhibition of 0.22 mg/kg bw per day in a four-week study in dogs and a safety factor of 100. The effect observed was reversible and acute. The previous ADI was 0-0.01 mg/kg bw.

Carbosulfan, the subject of a separate residue re-evaluation at the present Meeting, is metabolized to carbofuran and evaluations of carbofuran residues must account for carbofuran and its metabolites resulting from the use of carbosulfan according to GAP.

IDENTITY

ISO common name: carbofuran

Chemical name:

IUPAC: 2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate

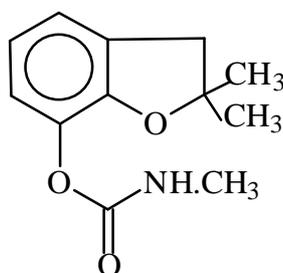
CA: 2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate

CAS No.: 1563-55-2

CIPAC No.: 276

Synonyms: Furadan; Curraterr; Yaltox; FMC 10242

Structural formula:



Molecular formula: $C_{12}H_{15}NO_3$

Molecular weight: 221.26

Physical and chemical properties

Pure active ingredient

Vapour pressure: 6×10^{-7} mm Hg at 25°C (Alvarez, 1989)

Melting point: 153-154°C (USA Standard, 1968)

Octanol/water partition coefficient:

$\log P_{ow}$ 1.3 at 20°C (Brandau, 1975)

Solubility:

g/100 g at 25°C:

acetone	15	(USA Standard, 1968)
acetonitrile	14	
benzene	4	
cyclohexanone	9	
dichloromethane	12	
dimethylformamide	27	
dimethylsulfoxide	25	
ethanol	4	
water	0.035 g/100 ml	(Alvarez, 1987)
xylene	<1	

Specific gravity: 1.18 at 20°C

Hydrolysis:	pH	Temperature, °C	Half-life, h
	25	>20,000	(Alvarez, 1987; Dziedzic 1987)
	3.1	35	>20,000
	3.1	45	>20,000
	6.2	25	≥7,000
	6.2	35	1400
	6.2	45	320
	7.0	25	670
	7.5	25	220
	8.0	25	65
	9.1	25	15
	9.1	35	3.2
	9.1	45	0.76
	9.9	25	2.2
	9.9	35	0.55
	9.9	45	0.16

Photolysis:

Half-life 150 hours in pH 7.0 buffered aqueous solution (5 mg/l) at 25°C when subjected to 300-400 nm radiation with a power of 150 $\mu\text{w}/\text{cm}^2$.

Technical material

Purity: 98%

Melting range: 150-152°C

Stability: Stable under neutral or acid conditions. Unstable in alkaline media.

Formulations

Formulated products containing carbofuran are listed in Table 1.

Table 1. Formulations of carbofuran.

Product	Form.	Active ingredient(s)	% ai
Furadan 75 DB	DP	carbofuran	75
Furadan 85 DB	DP	carbofuran	85
Furadan 3G (Carbo 3G)	GR	carbofuran	3
Furadan 5G or 50G (Carbosip 5G)	GR	carbofuran	5
Furadan 10G	GR	carbofuran	10
Furadan 20F	SC	carbofuran	20
Furadan 35 FS	FS	carbofuran	35
Furadan 4F or 40 F	SC	carbofuran	4
Furadan 47F	SC	carbofuran	47
Furadan 300ST	ST	carbofuran	30
Furadan 310ST (Furazin 310 TS)	ST	carbofuran	31
Furadan 35 or 350	FS	carbofuran	35
Furadan 360	FS	carbofuran	36
Furadan 350SC	SC	carbofuran	35
Curraterr 10G	GR	carbofuran	10
Curraterr 5G	GR	carbofuran	5
Furadan Combi	ST	carbofuran + carbendazim + thiram	27 5 5
Yaltox	ST	carbofuran	

METABOLISM AND ENVIRONMENTAL FATE

Animal Metabolism

The metabolism of [^{14}C]carbofuran has been studied in rats, houseflies, laying hens and lactating goats (Table 2). The carbofuran was uniformly labelled in the phenyl ring in all studies except on houseflies, where [*carbonyl*- ^{14}C]carbofuran was used. Both labels were used in the rat study.

Table 2: Animal metabolism studies on [^{14}C]carbofuran.

Subject	Treatment	References
Rats	4 mg/kg bw, single oral	Dorough, 1968
Houseflies	0.05 $\mu\text{g}/\text{fly}$, topical	
Lactating goats	25 ppm for 7 days ¹	Hoffman and Robinson, 1994a
Laying hens	25 ppm for 7 days ¹	Hoffman and Robinson, 1994b

¹Doses were daily by capsule, equivalent to 25 ppm in feed

Rats. Rats of 200 g each were treated orally with either 0.4 mg per kg bw [*carbonyl*- ^{14}C]carbofuran or 4.0 mg per kg bw [*phenyl*- ^{14}C]carbofuran in a single dose. The urine and faeces were collected and assayed for the total radioactivity. ^{14}C was collected from the rats given the carbonyl label. The cumulative percentages of the administered doses found in air, urine and faeces are shown in Table 3. About 86-90% of the radiolabelled carbofuran was eliminated by 32 hours after treatment. Additional elimination was minimal. About 45% of the radiolabelled carbofuran was eliminated by cleavage of the carbamoyl moiety.

Table 3. Cumulative percentage of administered ^{14}C in air, urine and faeces from orally-dosed rats.

Time after dosing (h)	Cumulative % of administered dose				
	[carbonyl ^{14}C]carbofuran			[phenyl ^{14}C]carbofuran	
	CO_2	Urine	Faeces	Urine	Faeces
2	5.6	2.7	0.0	5.9	0.3
6	31	25	0.8	21	0.5
24	43	37	1.9	72	2.3
32	45	38	2.6	88	2.4
48	45	38	3.8	89	2.5
72	45	38	4.4	91	3.3
96	45	38	4.4	92	3.3
120	45	38	4.4	92	3.3

Urine from the [*phenyl*- ^{14}C]carbofuran-treated rats from the 2-24 hour periods was extracted with an organic solvent. Less than 5% of the radioactivity was organosoluble. The major component identified by TLC in the 24-hour sample was 2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl-hydroxymethylcarbamate, 1.1% of the total radioactivity in the urine. Pooled urine collected for 72 hours from the [*phenyl*- ^{14}C]carbofuran-treated rats was acidified to 0.5 N, boiled for 10 minutes and extracted with chloroform. About 95% of the water-soluble residue was converted to chloroform-soluble material. The compounds tentatively identified by TLC, with their percentages of the total radioactivity in the pooled sample, were 2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl-hydroxymethylcarbamate (3.8%), 3-hydroxy-carbofuran (14%), 2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol (1.4%), 2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol (48%) and 2,3-dihydro-2,2-dimethylbenzofuran-7-ol (20%). Control experiments showed that carbofuran, 2,3-

dihydro-2,2-dimethylbenzofuran-7-ol, 2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol and 2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol were not altered by the treatment.

Houseflies (6 days old) were treated topically with [*carbonyl*-¹⁴C]carbofuran at 0.05 µg per fly and analysed in groups of 100 one hour after application. Surface radioactivity was removed with an acetone rinse. Internal radioactivity was extracted by homogenizing with acetone/water (1:1) and partitioning with chloroform. The vials were rinsed with acetone/water to collect excreted ¹⁴C and the wash was partitioned with chloroform. Water-soluble fractions were hydrolysed with acid and extracted with chloroform. The organic extracts were analysed by TLC, with the results given in Table 4. Identities were not confirmed.

Table 4. Tentative identification of the radiolabelled residue from the topical application of [*carbonyl*-¹⁴C]carbofuran to houseflies.

Compound	% of applied dose		
	Surface residue	Internal residue	Excretion
Carbofuran	23	12	7.1
3-hydroxy-carbofuran	0.3	5.7	0.4
3-hydroxy-carbofuran conjugated ¹	0	11	4.3
2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl hydroxymethylcarbamate	0.2	0.7	0.3
2,3-dihydro-2,2-dimethyl-3-hydroxybenzofuran-7-yl hydroxymethylcarbamate conjugated ¹	0	2.1	0.1
3-oxo-carbofuran	0.2	1.2	2.0
2,3-dihydro-2,2-dimethylbenzofuran-7-yl-N-hydroxymethylcarbamate conjugated ¹	0	1.8	0.2
Total ²	24	34	14

¹Released by mild acid hydrolysis

²An unknown (free and conjugated) accounted for 9% of the applied dose. Also, part of the dose may have been lost as

¹⁴CO₂ from hydrolysis of the carbamate group

Fifteen laying hens (1.34-1.68 kg, randomly divided into groups of 5) each received a capsule containing 3 mg of [¹⁴C]carbofuran on each of 7 consecutive days. Eggs were collected on each day, separated into yolk and whites and pooled by group. Excreta were collected daily and pooled by group. Within 22 hours of the final dose, the hens were killed and samples of breast, thigh, fat with skin, liver and kidneys were collected from each hen and pooled by group.

Most of the administered dose was eliminated in the excreta, with the cumulative percentage of it ranging from an average of 71% on day 1 to 83% on day 7. The distribution of the radiocarbon in the eggs, excreta and tissues is shown in Table 5.

Table 5. Total radioactive residues as cumulative percentage of administered dose and as carbofuran equivalents.¹

Sample	Day	% of applied dose	Total ¹⁴ C as carbofuran, mg/kg
Excreta	1	70.6	
	3	75.2	
	7	82.8	
Egg white	1	0.18	0.032
	3	0.21	0.069
	7	0.27	0.059
Egg yolk	1	0.07	0.027
	3	0.09	0.078

Sample	Day	% of applied dose	Total ¹⁴ C as carbofuran, mg/kg
	7	0.21	0.141
Liver	7	0.11	0.137
Kidneys	7	0.01	0.034
Breast muscle	7	0.02	<0.010
Thigh muscle	7	<0.01	<0.010
Skin and fat	7	<0.01	<0.010
Total recovery	7	83.4	

¹ Average of three groups.

The tissue samples containing >0.01 mg/kg total radioactive residue (TRR) were extracted sequentially with acetonitrile and methanol/water. Egg white was extracted with acetonitrile and egg yolk with a mixture of acetonitrile and hexane. The extractions removed the following percentages of the TRR: egg yolk 91%; egg white 91%; liver 16%; kidneys 41%. The post-extraction solids from the liver and kidneys were treated sequentially with protease, acid and base. Protease released 25% of the TRR from the liver and 19% from the kidneys. Acid and base treatments released an additional 48% from the liver and 28% from the kidneys.

The radiolabelled residues released by solvent extraction and enzyme, acid and base hydrolyses were investigated by normal-phase TLC and reverse-phase HPLC. The characterizations and identifications are shown in Table 6. The structures of the metabolites are given in Figure 1.

Table 6. Characterization and identification of the total radiolabelled residue from the administration of [¹⁴C]carbofuran to hens.

Metabolite or characterization	Liver		Kidneys		Egg white		Egg yolk	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
3-hydroxy-carbofuran	--	-	-	-	-	-	12	0.019
2,3-dihydro-2,2-dimethylbenzofuran-7-ol	5.7 ¹	0.008	4.9 ¹	0.001	-	-	16	0.026
2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol	-	-	-	-	-	-	39	0.062
2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol	-	-	-	-	-	-	8.5	0.014
Phenolic conjugates	-	-	-	-	90	0.060	-	-
Enzyme digestion aqueous fraction	7.3	0.010	4.6	0.002	-	-	4.6	0.007
Mild acid hydrolysis aqueous fraction	3.1	0.004	5.8	0.002	-	-	-	-
Strong acid hydrolysis aqueous fraction	12	0.016	8.2	0.003	-	-	-	-
Mild base hydrolysis aqueous fraction	4.0	0.005	3.6	0.001	-	-	-	-
Polar residues from initial extractions	12	0.016	8.2	0.003	-	-	-	-

¹ conjugated, released by enzyme treatment.

The total radiolabelled residues in the muscle and fat with skin were negligible and the residues in the kidneys, liver and eggs ranged from 0.03 to 0.2 mg/kg. The parent compound was not detected. The metabolic pathway includes oxidation to 3-hydroxy- and 3-keto-carbofuran and hydroxylation to phenolic metabolites. See Figure 2.

[¹⁴C]carbofuran, uniformly labelled in the phenyl ring, was administered orally to 2 goats for 7 consecutive days. The dose was equivalent to 25 mg/kg carbofuran in the feed. Urine, faeces and milk were collected twice daily and pooled. The goats were slaughtered within 24 hours of the final dose and samples of muscle (leg and loin), liver, kidney, omental fat and blood were taken. The distribution of the ¹⁴C is shown in Table 7.

Table 7. Total radioactive residue as cumulative percentage of administered dose and as carbofuran equivalents.¹

Sample	Day	% of applied dose	TRR as carbofuran, mg/kg
Milk	1	0.32	0.010
	3	0.29	0.14
	7	0.30	0.098
Urine	1	95	
	3	90	
	7	88	
Faeces	1	4.1	
	3	5.1	
	7	5.0	
Liver	7	0.025	0.11
Kidneys	7	<0.01	0.18
Leg muscle	7	<0.01	<0.01
Loin muscle	7	<0.01	0.01 ²
Omental fat	7	<0.01	<0.01
Total recovery	7	95	

¹Average of 2 goats

²Goat B only. Goat A was <0.01 mg/kg

Milk (day 5 pm, containing 0.32 mg/kg carbofuran equivalents) was extracted with acetone. Muscle tissue (from goat B), liver and kidneys were sequentially extracted with chloroform and methanol/water. The percentages of the total radioactive residue extracted were milk 99%; muscle 30%; liver 27%; kidney 20%. The post-extraction liver and kidney samples were sequentially treated with protease, mild acid extraction and strong acid hydrolysis. Protease released 41% of the total radioactive residue from the liver and 49% from the kidneys. The mild acid extraction released 12% from the liver and kidneys.

The released radioactive residues were characterized and the components identified by normal-phase TLC and reverse-phase HPLC, with the results shown in Table 8. The structures of the metabolites are given in Figure 1.

Table 8. Characterization and identification of total radioactive residue from the administration of [¹⁴C]carbofuran to lactating goats.

Metabolite or characterization	¹⁴ C, % of the TRR and mg/kg as carbofuran							
	Milk		Muscle		Liver		Kidney	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
carbofuran	0.41	0.001	-	-	-	-	-	-
3-hydroxy-carbofuran	10	0.032	-	-	4.0 ³	0.005	11 ⁶	0.029
2,3-dihydro-2,2-dimethylbenzofuran-7-ol	15	0.048	-	-	2.4 ⁴	0.003	-	-
2,3-dihydro-2,2-dimethylbenzofuran-3,7-diol	6.8 ¹	0.021	-	-	12 ⁵	0.017	16 ⁷	0.042
2,3-dihydro-2,2-dimethyl-3-	32 ²	0.10	-	-	-	-	-	-

Metabolite or characterization	¹⁴ C, % of the TRR and mg/kg as carbofuran							
	Milk		Muscle		Liver		Kidney	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
oxobenzofuran-7-ol								
Aqueous fraction from initial extractions	6.3	0.020	28	0.003	5.0	0.007	3.5	0.009
Aqueous fraction from enzyme digestion	-	-	-	-	16	0.022	13	0.035
Aqueous fraction from mild acid hydrolysis	-	-	-	-	4.5	0.007	5.1	0.014
Aqueous fraction from strong acid hydrolysis	-	-	-	-	6.3	0.009	6.7	0.018
Polar residues (in initial extracts)	22	0.070	-	-	6.9	0.010	17	0.044

¹Including 2% conjugated, released by sulfatase treatment

²Including 29% conjugated, released by sulfatase treatment

³Including 2.2% conjugated, released by protease treatment

⁴Conjugated, released by protease treatment

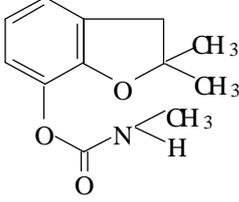
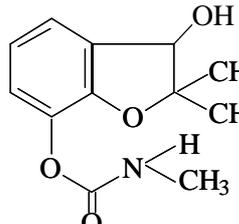
⁵Including 11% conjugated, released by protease treatment

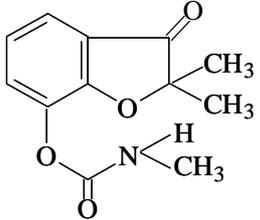
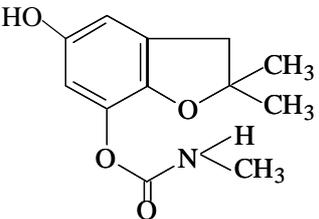
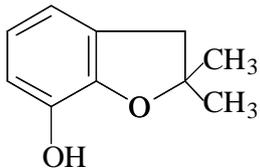
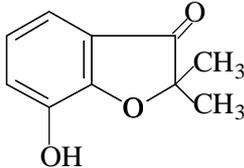
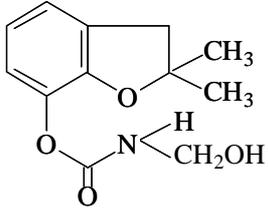
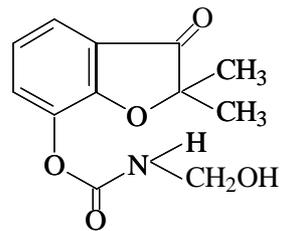
⁶Including 8.2% conjugated, released by protease treatment

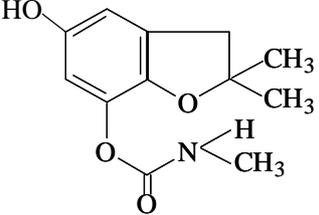
⁷Conjugated, released by protease treatment

The total radioactive residues in the tissues and fat were negligible (≤ 0.01 mg/kg) after the dietary equivalent of 25 mg/kg for 7 days. Residues in the kidneys, liver and milk ranged from 0.09 to 0.39 mg/kg. The identified metabolites are the same as those found in poultry, but the parent compound was also detected in milk. Figure 2 shows the probable metabolic pathways in poultry and ruminants. Two paths are indicated, in which oxidation at C-3 is followed or preceded by hydrolysis of the carbamate linkage. Oxidation of the carbamate methyl was not observed in goats or hens.

Figure 1. Names and chemical structures of carbofuran and its potential metabolites.

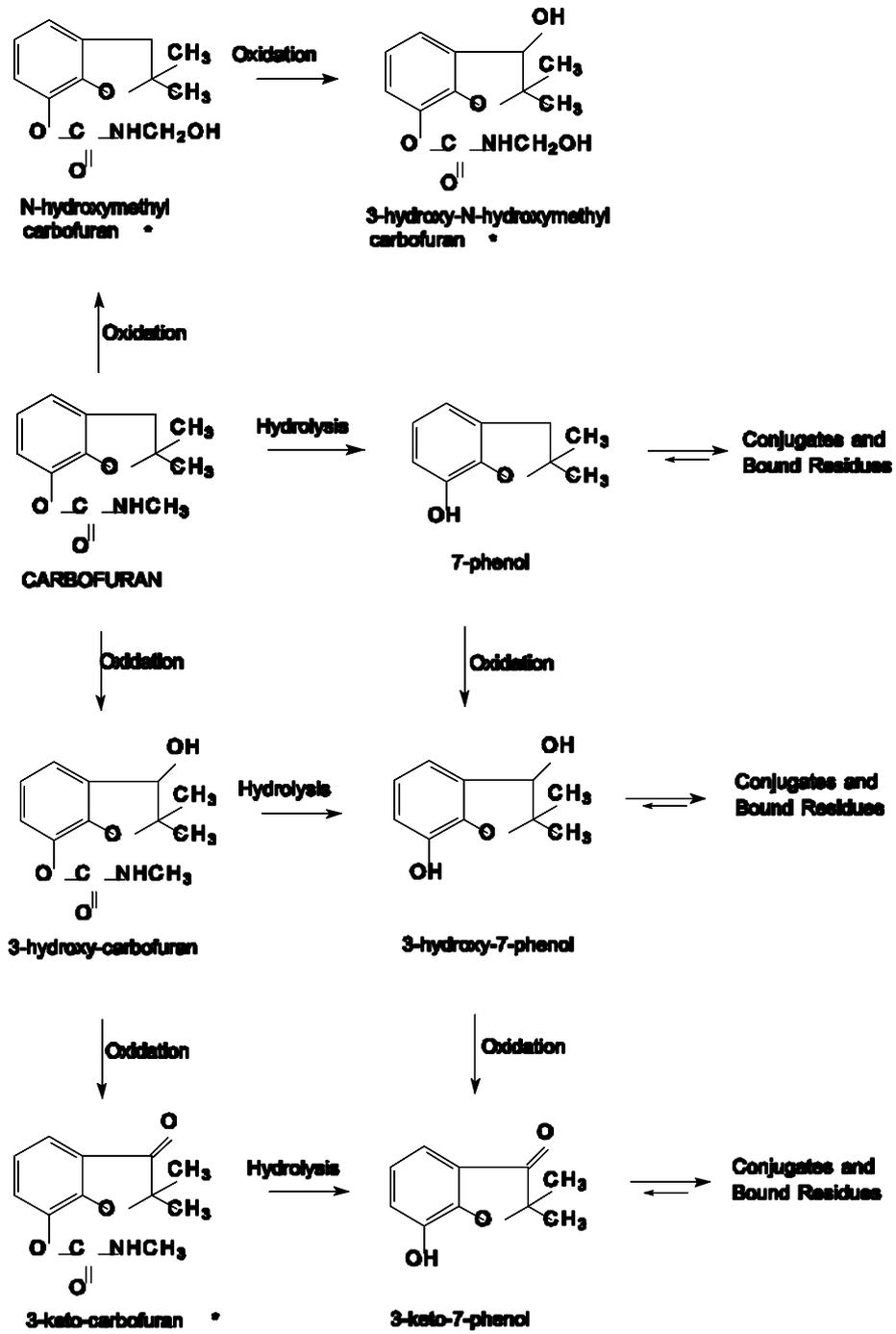
{PRIVATE }Common or derived ¹ name Abbreviation used in Tables FMC number	Chemical name	Structure
Carbofuran CF FMC 10242	2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate	
3-hydroxy-carbofuran 3-OH-CF	2,3-dihydro-3-hydroxy-2,2-dimethylbenzofuran-7-yl methylcarbamate	

{PRIVATE }Common or derived ¹ name Abbreviation used in Tables FMC number	Chemical name	Structure
3-keto-carbofuran 3-K-CF	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-yl methylcarbamate	
5-hydroxy-carbofuran 5-OH-CF FMC 27552	2,3-dihydro-5-hydroxy-2,2-dimethylbenzofuran-7-yl methylcarbamate	
7-phenol 7-P FMC 10272	2,3-dihydro-2,2-dimethylbenzofuran-7-ol	
3-keto-7-phenol 3-K-7-P FMC 16490	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-ol	
N-hydroxymethyl carbofuran N-CH ₂ OH CF FMC 53858	2,3-dihydro-2,2-dimethylbenzofuran-7-yl hydroxymethylcarbamate	
3-keto-N-hydroxymethyl carbofuran 3-K-N-CH ₂ OH CF FMC 53895	2,3-dihydro-2,2-dimethyl-3-oxobenzofuran-7-yl hydroxymethylcarbamate	

{PRIVATE }Common or derived ¹ name Abbreviation used in Tables FMC number	Chemical name	Structure
{PRIVATE } 5-hydroxy-carbofuran 5-OH-CF FMC 27552	2,3-dihydro-2,2-dimethyl-5- hydroxybenzofuran-7-yl N- methylcarbamate	

¹Names such as 3-hydroxy-carbofuran, 7-phenol etc., derived from the common name carbofuran

Figure 2. Proposed biotransformation pathways of carbofuran in poultry and ruminants.



Plant metabolism

Metabolism studies were reported for potatoes, soya beans and maize (field corn). Supplementary information was submitted on the metabolism of radiolabelled carbofuran in several rotational crops.

Potatoes. Greenhouse-grown potato plants, height about 20 cm, were treated with [*phenyl*-¹⁴C]carbofuran (2.65 mCi/mmol, 26,548 dpm/μg) in a single directed application to the soil surface at 7.4 kg ai/ha (Chang, 1994). The [¹⁴C]carbofuran was formulated as a 0.5 kg ai/l flowable formulation (4F) and was diluted with water before application. Immature vines were sampled after 56 days and mature tubers harvested after 104 days. The total radioactive residues were 30.5 mg/kg as carbofuran in the vines and 0.80 mg/kg in the potatoes. Extraction of immature vine and mature potatoes with methanol/water followed by methylene chloride partition of the acidified and concentrated extract yielded 6.0% of the foliage and 22% of the tuber TRR. The aqueous from the methylene chloride partition, containing 87% of the foliage and 61% of the tuber TRR, was sequentially incubated with β-glucosidase (7.9% of the tuber and 51% of the foliage ¹⁴C was organosoluble) and hydrolysed with 0.25 N HCl (32% of the tuber and 14% of the foliage TRR was organosoluble) and 2 N HCl (9.4% of the tuber and 13% of the foliage TRR was organosoluble). The parent compound and metabolites were identified or characterized by reverse-phase HPLC and normal-phase TLC. Tentative identifications were confirmed by GC-MS, both EI and CI. The major metabolite identified in the mature tubers was the 7-phenol (45% of the TRR) and in the foliage 5-hydroxy-carbofuran (34%). The results are shown in Table 9.

Table 9. Identification or characterization of radiolabelled residues in or on potatoes from the application of [*phenyl*-¹⁴C]carbofuran to soil at 7.4 kg ai/ha after plant emergence.

Compound	Mature tuber (104-day PHI)	mg/kg	Immature foliage, 56- day PHI	mg/kg
	% of TRR		% of TRR	
Carbofuran	-	-	3.5%	1.071
3-OH-carbofuran	2.9%	0.023	22.6%	6.906
3-keto-carbofuran	-	-	1.1%	0.324
7-phenol	45.3%	0.361	6.7%	2.044
3-OH-7-phenol	13.4%	0.107	5.4%	1.658
3-keto-7-phenol	6.6%	0.052	9.4%	2.858
5-OH-carbofuran	-	-	34.4%	10.522
Total Identified ¹	68.2% (22% unconjugated)	0.543	83.1% (4.6% unconjugated)	25.383
Other	3.7%	0.029	2.6%	0.807
Polar residues	23.3%	0.185	11.0%	3.354
Unextractable	4.9%	0.039	3.3%	1.002
Total Residues ¹	100.0%	0.80	100.1%	30

¹Results are normalized for recovery (91-101%).

Soya beans. Sandy loam soil in two 61 x 120 x 61 cm boxes was treated with carbofuran uniformly labelled with ¹⁴C in the phenyl ring at 5.5 kg ai/ha in Watsonville, CA, USA. The treatment solution also contained carbofuran labelled with ¹³C on one of the two gem-dimethyl groups and was prepared as a 0.5 kg ai/l flowable formulation (4F) in acetone/water. As applied the solution had a specific activity of 8.03 mCi/mmol. The test material was applied in a 15 cm band to a 1.3 cm deep furrow. Immediately after the application, soya bean seeds were sown in a single row down the middle of the furrow and covered with untreated soil. The soya beans were grown outdoors and samples of forage at 45 days PHI, beans at 139 days and hay at 139 days were collected.

Samples were assayed for the total radioactive carbon by oxidation and liquid scintillation counting. The forage contained 63 mg/kg carbofuran, the beans 0.32 mg/kg and the hay 36 mg/kg. Samples were then extracted with methanol/water (4:1 v/v) and subsamples of the extracts were concentrated and refluxed for one hour with 0.25 N HCl. The product mixtures were extracted with methylene chloride and the residual solids sequentially hydrolysed with 0.25 N HCl (60°C), cellulase, β -glucosidase, amyloglucosidase, pectinase, protease, 6N HCl (60°C) and 2N NaOH (65°C). The solid residues from the hay samples after solvent extraction were solubilized with dioxane/water (3/1 v/v) to release lignin (85°C, 48 hrs). After each hydrolysis the aqueous product solutions were adjusted to pH 2 and extracted with acetonitrile to recover organosoluble residues. The distribution and characterization of the radiolabelled residues are shown in Table 10.

The methanol/water and acid-refluxed methanol/water extracts were analysed by HPLC (reverse-phase) and fractions were collected for radioanalysis. Confirmation was by normal-phase (silica gel) TLC. The main metabolites were identified by GC-MS in both CI and EI modes. Unknown compounds separated by TLC or HPLC were investigated by HPLC-MS. The compounds identified are shown in Table 11.

Table 10. Distribution of the ^{14}C in hydrolysates and extracts of soya bean forage, beans and hay.

Sample	Fraction	[^{14}C]carbofuran equivalents, mg/kg	% of TRR
Forage (63 mg/kg)	Methanol/water extract	50	80
	0.25 N HCl treatment of PES ¹ , organosoluble	1.4	2.3
	Cellulase of PES, organosoluble	0.11	0.18
	B-glucosidase of PES, organosoluble	0.10	0.16
	Amyloglucosidase of PES, organosoluble	0.24	0.38
	Pectinase of PES, organosoluble	0.14	0.22
	Protease of PES, organosoluble	0.84	1.3
	6.0 N HCl treatment of PES, organosoluble	0.82	1.3
	2.0 N NaOH of PES, organosoluble	4.1	6.5
	Final Residual Solid	4.3	6.9
TOTAL	62	99	
Beans (0.32 mg/kg)	Methanol/water extract	0.19	59
	0.25 N HCl treatment of PES ¹ , organosoluble	0.030	9.3
	Cellulase of PES, organosoluble	0.011	3.6
	B-glucosidase of PES, organosoluble	0.003	0.92
	Amyloglucosidase of PES, organosoluble	0.006	1.8
	Pectinase of PES, organosoluble	0.014	4.3
	Protease of PES, organosoluble	0.022	6.9
	6.0 N HCl treatment of PES, organosoluble	0.013	4.0
	2.0 N NaOH of PES, organosoluble	0.019	5.9
	Final Residual Solid	0.019	5.9
TOTAL	0.33	102	
Hay ² (36 mg/kg)	Methanol/water extract	13	35
	0.25 N HCl treatment of PES organosoluble	5.4	15
	Cellulase of PES organosoluble	0.41	1.1
	B-glucosidase of PES organosoluble	0.26	0.72
	Amyloglucosidase of PES organosoluble	0.23	0.65

Sample	Fraction	[¹⁴ C]carbofuran equivalents, mg/kg	% of TRR
	Pectinase of PES organosoluble	0.13	0.36
	Protease of PES Organosoluble	0.17	0.48
	6.0 N HCl treatment of PES organosoluble	0.35	0.97
	2.0 N NaOH of PES organosoluble	0.40	1.1
	Dioxane (lignin release) of PES	2.1	5.9
	Final Residual Solid (before lignin release)	15	43
	TOTAL	35	98

¹Post-extraction

²Moisture content of the hay was not determined: figures refer to undried hay

Table 11. Identification of carbofuran and metabolites in the radiolabelled residue isolated from methanol/water extracts of soya bean seed, forage and hay.

Compound	¹⁴ C, % of TRR and mg/kg as carbofuran											
	Extract		Acid-refluxed extract		Extract		Acid-refluxed extract		Extract		Acid-refluxed extract	
	% TRY	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
carbofuran	11.6	7.3	11.4	7.2	-	-	0.42	0.001	0.30	0.11	0.62	0.22
3-keto carbofuran	1.7	1.1	1.6	1.0	-	-	5.3	0.02	0.41	0.15	-	-
3-hydroxy-carbofuran	10.6	6.6	28	18	0.56	0.002	1.5	0.005	3.2	0.50	7.8	2.8
7-phenol	-	-	1.4	0.90	0.38	0.001	4.0	0.013	0.67	0.24	0.72	0.26
3-keto-7-phenol	1.6	1.0	13	8.1	0.71	0.002	9.2	0.030	4.3	1.6	9.8	3.5
<i>O</i> -glucoside conjugate of 3-hydroxy or 3-keto-7-phenol ¹	16	9.9	3.4	2.1	11	0.036	-	-	3.6	1.3	-	-
2-hydroxymethyl-3-keto carbofuran ²	-	-	3.6	2.2	-	-	0.93	0.03	-	-	0.84	0.30
TOTAL identified	42		62		13		21		12		20	

¹Identification by LC-MS. No comparison with reference standard

²Identification by GC-MS (EI and CI). No comparison with reference standard

Maize. A 1.5 x 1.5 m plot of tilled Crosby Loam soil in Ohio was treated with carbofuran uniformly labelled with ¹⁴C in the phenyl ring at a rate of 8.3 kg ai/ha treated area, equivalent to 3.0 kg ai/ha broadcast (Curry, 1994). The radiolabelled material was isotopically diluted with [¹³C]carbofuran labelled in one of the gem-dimethyl groups and with unlabelled carbofuran to a specific activity of 2.65 mCi/mmol or 26548 dpm/μg. The carbofuran was prepared as a 0.5 kg ai/l flowable formulation (4F) and was mixed with water before application. The test material was sprayed in a 15 cm band on the soil and incorporated to a depth of about 5 cm before planting maize seed (Pioneer Hybrid 3394).

Maize samples were taken at three growth stages: forage (immature stage, 47 days PHI), silage (reproductive stage, 99 days PHI) and stover and grain (mature stage, kernels without cob and husk, 158 days PHI). The samples were assayed for total ¹⁴C by combustion and liquid scintillation counting. Each sample was extracted with methanol/water (1:1 v/v), and the extracts acidified to pH 1 and partitioned with methylene chloride/ether (3:1 v/v). The aqueous fractions from the methylene chloride/ether partitions of the forage and silage samples were divided into two equal portions: one was treated with β-glucosidase and the other was acidified to 0.25 N, refluxed for one hour, and

extracted with methylene chloride/ether. The aqueous layer from this extract of the silage samples was acidified to 1 N, refluxed for one hour, and extracted with methylene chloride/ether.

The post-extraction solids (PES) from the initial methanol/water extractions were refluxed with 0.25 N HCl for one hour. The hydrolysate from the grain was tested to determine the presence of reducing sugars with Benedict's solution and by osazone formation. Both tests indicated reducing sugars. The residue after acid hydrolysis was treated with a surfactant, sodium dodecyl sulfate. The distribution of radioactivity in the various fractions, as determined by liquid scintillation counting, is shown in Table 12.

Table 12. Distribution of the radiolabelled residue in the extracts and hydrolysates of maize grain, forage, silage and stover (fodder) from the pre-plant application of [¹⁴C]carbofuran

Fraction	Grain (0.023 mg/kg)		Forage (0.81 mg/kg)		Stover (0.075 mg/kg)		Silage (0.14 mg/kg)	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Methylene chloride/ether (non-conjugates)	5.8 ¹	0.001	42	0.34	4.4	0.003	4.6	0.006
Acid-released (0.1 n), methylene chloride/ether (aglycones)	-	-	32	0.26	-	-	20	0.028
Glucosidase-released (aglycones)	-	-	19	0.15	-	-	23	0.032
Residual acid aqueous	-	-	8.1	0.066	22	0.016	31 ²	0.036
Acid-released from PES	48 ³	0.011	3.5 (1.0 organo-soluble)	0.028	13 (4.2% organo-soluble)	0.010	9.9 (2.8% organo-soluble)	0.014
Surfactant-released from PES	-	-	1.6	0.013	4.7	0.004	5.1	0.007
Total released residue	48		87		44		71	

¹Methanol/water extract

²1 N HCl treatment of the residual 0.25 N aqueous fraction generated an additional 7.9% of the TRR (0.011mg/kg) of organosoluble residue

³ <1% partitioned into methylene chloride.

The organosoluble fractions from the forage and silage, i.e. the methylene chloride/ether extracts of the acidified methanol/water extract and of the 0.25 N HCl hydrolysate, were analysed by HPLC, TLC and GC-MS. The methylene chloride/ether extract of the 1 N HCl hydrolysate of silage was also analysed. Because of the relatively low levels of radiolabelled residue, the extracts from the grain and stover were not analysed. The identified compounds are shown in Table 13.

Table 13. Carbofuran and its metabolites in organosoluble extracts of maize silage and forage.

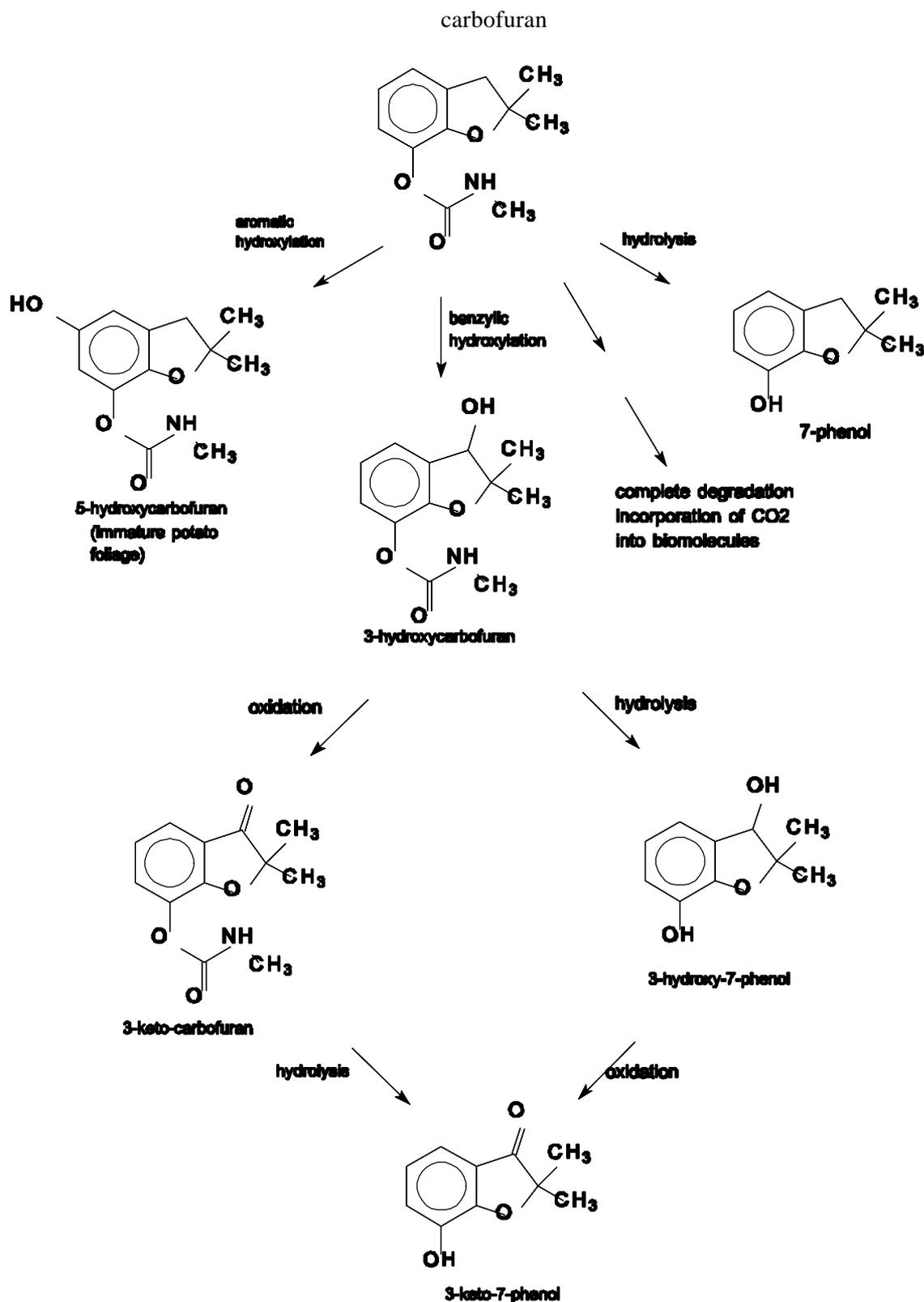
Compound	Forage		Silage	
	% of TRR	mg/kg as carbofuran	% of TRR	mg/kg as carbofuran
carbofuran	14	0.11	0.18	<0.001
carbofuran aglycone	2.4	0.019	2.1	0.003
3-keto-carbofuran	1.6	0.013	-	-
3-keto-carbofuran aglycone	0.28	0.003	0.91	0.001
3-hydroxy-carbofuran	13	0.11	1.3	0.002
3-hydroxy-carbofuran aglycone	9.7	0.078	7.9	0.011
7-phenol	0.47	0.004	0.088	<0.001

Compound	Forage		Silage	
	% of TRR	mg/kg as carbofuran	% of TRR	mg/kg as carbofuran
7-phenol aglycone	7.5	0.060	2.8	<0.001
3-keto-7-phenol	4.8	0.039	1.4	0.002
3-keto-7-phenol aglycone	5.6	0.045	2.4	0.003
3-hydroxy-7-phenol	2.4	0.020	0.88	0.001
3-hydroxy-7-phenol aglycone	3.6	0.029	2.3	0.003
Total	65	0.53	22	0.026

The major components of the radiolabelled residue identified in the forage were carbofuran and 3-hydroxy-carbofuran, free and conjugated and in the silage 3-hydroxy-carbofuran, free and conjugated. The amount of radioactivity that could not be extracted with solvent or released by mild acid hydrolysis increased with the PHI, suggesting incorporation of the radiolabel into plant constituents.

The metabolites in the three crops (maize, potatoes and soya beans) are similar and are consistent with metabolism by hydroxylation and oxidation at C-3 and hydrolysis of the carbamate linkage (C-7). Aromatic hydroxylation was seen only in immature potato foliage. The proposed metabolic pathways are shown in Figure 3.

Figure 3. Proposed metabolic pathways of carbofuran in plants.



Environmental fate in soil

The rate and degree of the aerobic degradation of [¹⁴C]carbofuran and its metabolites in acid and alkaline soils were determined in a study conducted in accordance with US EPA Guidelines (Saxena *et al.*, 1994c). An acidic sandy loam soil (pH 5.7) was collected in Georgia and a portion was made alkaline (pH 7.7) by the addition of lime. The limed soil was incubated for about 2 months at

approximately 25°C until the soil pH and microbial population had reached equilibrium before adding [¹⁴C]carbofuran uniformly labelled in the phenyl ring.

The test system consisted of approximately 50 g of oven-dried soil in a 250-ml flask. The soil samples were fortified with [¹⁴C]carbofuran at a nominal concentration of 3 mg/kg (equivalent to 6.7 kg ai/ha) and incubated at 25 ± 1°C under aerobic conditions in darkness for 365 days. The apparatus included ethylene glycol to trap organic volatiles and sodium hydroxide to trap CO₂.

Duplicate samples were analysed on days 0, 1, 3, 7, 14, 30, 62, 92, 122, 181, 273 and 365, and a third sample was taken at each interval to measure the pH and microbial population. The solutions in the traps were changed and the soil moisture was adjusted periodically. The samples were analysed immediately after collection: the population of aerobic bacteria and the pH were determined, the radioactivity in the traps was counted by LSC, the soils were extracted and analysed by HPLC and the extracted soil was combusted to measure the ¹⁴C. Selected extracts were also analysed by TLC to confirm the identity of [¹⁴C]carbofuran. Mass spectrometry was used to confirm the identities of degradation products which accounted for >10% of the TRR. More than 90% of the applied radioactivity was accounted for in all the samples. A summary of the results is given in Tables 14 and 15.

Table 14. Aerobic degradation of carbofuran in acidic soil.

Day	Mean % of applied ¹⁴ C as							
	carbofuran	3-OH-CF	3-K-7-P	3-K-CF	7-phenol	Volatiles	Soil-bound	Total
0	97.49	0.06	0.16	0.18	0.04	ND	0.40	98.32
1	96.10	0.19	0.31	0.31	ND	0.01	2.91	99.82
3	95.06	0.13	ND	0.29	ND	0.03	4.64	100.15
7	92.68	0.32	0.03	0.70	ND	0.05	5.94	99.71
14	88.89	0.15	0.13	2.10	ND	0.09	8.35	99.71
30	84.30	0.56	0.02	2.08	0.02	0.16	10.96	98.10
62	82.72	ND	ND	2.60	ND	0.29	13.01	98.62
92	74.98	0.56	ND	6.36	ND	0.55	15.00	97.45
122	69.86	ND	ND	7.13	ND	0.94	20.89	98.81
181	58.29	ND	ND	12.41	ND	2.52	24.59	97.80
273	53.85	0.55	ND	11.41	ND	3.98	29.28	99.05
365	43.58	0.63	1.91	11.14	0.33	4.96	35.41	97.95

Table 15. Aerobic degradation of carbofuran in alkaline soil.

Day	Mean % or applied ¹⁴ C as							
	carbofuran	3-OH-	3-K-7-P	3-K-CF	7-phenol	Volatile	Soil-bound	Total
0	96.63	0.36	0.11	0.12	ND	ND	0.52	97.73
1	93.22	0.18	0.33	0.09	0.03	0.01	3.23	97.08
3	91.73	0.10	0.16	0.05	ND	0.02	7.18	99.23
7	87.73	0.79	0.37	0.07	0.28	0.11	9.98	99.32
14	83.00	0.92	0.77	0.13	0.05	0.25	12.98	98.08
30	77.39	0.33	0.20	0.02	ND	0.61	18.00	96.54
62	66.53	0.14	0.12	0.17	ND	1.67	27.48	96.11
92	59.65	ND	ND	ND	0.59	3.18	29.60	93.01
122	25.07	1.32	0.84	0.31	0.32	8.31	55.62	91.78
181	27.14	1.32	0.14	0.22	0.38	10.97	55.95	96.10
273	23.27	0.36	0.24	ND	1.08	14.11	59.23	98.27
365	20.96	0.56	0.26	0.14	0.36	16.60	57.83	96.71

3-OH-CF: 3-hydroxy-carbofuran
3-K-7-P: 3-keto-7-phenol
3-K-CF: 3-keto-carbofuran

The pH of the acidic soil samples showed no significant change during the study and ranged from 5.2 to 5.8. The pH of the alkaline samples remained between 7.4 and 8.0 in most samples but was 7.0 on day 181 and 6.6 on day 273. The microbial population remained viable and stable during the one-year period in both soils.

The only major degradation product (>10% of the applied radioactivity) in the acidic soil extracts was 3-keto-carbofuran, which reached a maximum of 12.41% of the applied radioactivity by day 181 and then decreased to 11.14% by day 365. The structure of 3-keto-carbofuran was confirmed by mass spectrometry and the structure of carbofuran was confirmed by two-dimensional TLC. Radioactivity from the alkaline soil in the NaOH traps was confirmed to be due to $^{14}\text{CO}_2$ by barium chloride precipitation. No degradation products exceeding 10% of the applied radioactivity were detected in the alkaline soil extracts. The other major products of degradation were soil-bound residues in both soils. A maximum of 35.41% (on day 365) and 59.23% (on day 273) of the applied radioactivity was incorporated in bound residues in the extracted acidic and alkaline soils respectively. Fractionation of the bound residues into humic acid, fulvic acid and humin indicated the presence of radioactivity in all three fractions.

The [^{14}C]carbofuran decreased from 97.49% at day 0 to 43.58% on day 365 in the acidic and from 96.63% on day 0 to 20.96% on day 365 in the alkaline samples. The half-life of [^{14}C]carbofuran in the test system calculated according to a first-order rate constant was 321 days and 149 days in the acidic and alkaline soils respectively.

The photodegradation of [^{14}C]carbofuran labelled in the phenyl ring, was studied in accordance with US EPA Guidelines under natural sunlight on a sandy loam soil at a field application rate of 1.7 kg ai/ha at approximately 22°C (McGovern and Shepler, 1989). The soil was sieved (2 mm) and sterilized before treatment. The control soil samples were covered to prevent exposure to light. All samples were placed in temperature-controlled chambers. Ethylene glycol and 10% NaOH were used to trap volatile organic compounds and CO_2 respectively, and air was drawn through both the irradiated and control chambers into separate sets of traps. Duplicate irradiated and control samples were analysed at 0, 3, 8, 15, 22 and 30 days after treatment.

The soil samples were extracted with methanol/water and the extracts assayed by LSC and analysed by HPLC. The remaining soil was combusted and assayed by LSC. More than 90% of the ^{14}C was recovered from all the samples. The ^{14}C from carbofuran decreased to 77% of the applied activity by day 30. The degradation products were the phenol, 3-hydroxy-carbofuran, the 3-hydroxy-7-phenol and CO_2 , each <10% of the applied ^{14}C . Carbofuran was also found to be degraded to the 7-phenol in the dark, showing that the showing that 7-phenol is not (all) photochemically derived. The calculated photolysis half-life of carbofuran was 78 days and the half-life of carbofuran in the dark was 720 days.

Three terrestrial field dissipation studies were in accordance with the US Environmental Agency Pesticides Assessment Guidelines in vineyards in California. In all three Furadan 4F was incorporated into plots of soil at the maximum use rate of 11.2 kg ai/ha. Triplicate soil cores were taken from treated and control plots before and immediately after application, and then at intervals for about a year. The cores were to a depth of 120 cm for the first 14 days and 240 cm thereafter. Each core was divided into 15 cm sections which were composited in groups of three to provide

triplicate samples at each 15 cm depth, which were analysed for carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran.

The first study was in Napa in 1987 (Daly and Tanner, 1988). The treated plot was approximately 30 x 30 m (11 rows of 10 vines) and the control plot was 72 m from the treated plot. The soil cores were taken before and immediately after incorporation and on days 3, 7, 14, 40, 90, 120, 150, 180, 304, 335 and 360. The soil was classified as a loam with the following characteristics.

pH	6.6
% Sand	41
% Silt	13
% Clay	46
CEC (meq/l)	28.7
Bulk density (g/cc)	1.4

The analytical limit of detection was 0.01 mg/kg and the limit of quantification 0.05 mg/kg. More than 77% of the residue in the soil was carbofuran. Residues of 3-keto-carbofuran and 3-hydroxy-carbofuran increased in the 0-15 cm soils for 30 days then decreased to <0.05 mg/kg by 108 days. The average total carbamate residue in the top 15 cm ranged from 3.16 to 6.66 mg/kg during the first 30 days. Quantifiable residues were not detected below the 105-120 cm. depths at any time except on day 30 at a level of 0.73 mg/kg at 120-135 cm. After 181 days the residues were below the limit of quantification at all depths. The first-order half-life calculated from the 0-15 cm depth was 43 days. A summary of the results is given in Table 16.

Table 16. Carbamate residues in soil dissipation study (Napa, 1987-88).

Depth, inches	Mean total carbamate residue, mg/kg										
	Days after application										
	0	5	7	14	30	108	119	150	181	282	388
0-6	3.16	4.65	4.07	6.66	4.45	0.55	0.26	0.15	0.14	(0.02)	(0.04)
6-12	0.29	0.34	0.14	0.05	0.12	0.28	0.10	(0.04)	(0.03)	ND	ND
12-18	0.13	0.07	0.11	(0.02)	ND	0.36	0.19	0.05	(0.02)	ND	ND
18-24	0.09	0.06	0.07	(0.01)	(0.02)	0.11	0.25	0.05	0.05	(0.01)	ND
24-30	0.11	0.08	0.06	(0.02)	ND	0.08	0.16	(0.02)	0.09	(0.01)	ND
30-36	0.08	0.07	0.06	(0.01)	ND	(0.04)	0.10	(0.02)	0.06	(0.01)	ND
36-42	0.07	0.09	(0.04)	ND	(0.01)	(0.02)	0.06	(0.01)	(0.02)	(0.01)	ND
42-48	0.10	0.08	0.06	(0.03)	(0.04)	(0.01)	(0.01)	ND	(0.03)	ND	ND
48-54	NS	NS	ND	NS	0.73	ND	ND	ND	(0.04)	ND	ND
54-60	NS	NS	NS	NS	ND	ND	ND	ND	(0.03)	ND	ND
60-66	NS	NS	NS	NS	ND	ND	ND	ND	(0.03)	ND	ND
66-72	NS	NS	NS	NS	ND						
72-78	NS	NS	NS	NS	(0.01)	ND	ND	ND	ND	ND	ND
78-84	NS	NS	NS	NS	(0.02)	ND	ND	ND	ND	ND	ND
84-90	NS	NS	NS	NS	ND						
90-96	NS	NS	NS	NS	ND	ND	ND	(0.02)	ND	ND	ND

NS: no soil core sample was taken ND: undetectable (<0.01 mg/kg)

Values in parenthesis are estimated, below the limit of quantification (0.05 mg/kg) but above the limit of detection (0.01 mg/kg)

A second study was in Farmersville in 1988-1989 (Herbert, 1989). The treated plot was 36 x 24 m (10 rows of 10 vines) and the control plot was 72 m from the treated plot. Core samples were taken before and immediately after incorporation and on days 3, 7, 14, 40, 90, 120, 150, 180, 304, 335 and 360. The soil was classified as a loam with characteristics shown in Table 17.

Table 17. Soil characteristics, Farmersville dissipation study.

{PRIVATE }Soil depth (inches)	0-12	12-24	24-36	36-48	48-60	60-72	72-84	84-96
PH	7.49	7.55	7.54	7.51	7.55	7.48	7.99	8.01
% Sand	51.4	66.8	69.8	70.8	70.8	68.6	70.6	71.6
% Silt	32.0	18.4	13.4	12.4	11.4	18.2	19.2	17.2
% Clay	16.6	14.8	16.8	16.8	17.8	13.2	10.2	11.2
CEC (meq/l)	10.1	5.1	5.5	5.4	5.5	7.7	8	12.4
% organic matter	1.04	0.34	0.07	0.04	0.06	0.20	0.09	0.10
Bulk density (g/cc)	1.32	1.37	1.40	1.35	1.38	1.47	1.49	1.56

The limit of detection of the analyses was 0.02 and the limit of quantification 0.05 mg/kg. More than 80% of the total residue in the soil was carbofuran. The average total carbamate residue in the 0-15 cm depth ranged from 4.41 to 6.07 mg/kg during the first 14 days, with minimal leaching. The dissipation of residues in this layer in 360 days was significant. Quantifiable residues were not found below 45 cm at any time except on day 40 at a level of 0.08 mg/kg at 120-135 cm and on day 304 at 0.25 mg/kg at 105-120 cm. The first-order half-life calculated from the 0-15 cm soil depth was 23 days. The results are given in Table 18.

Table 18. Carbamate residues in soil dissipation study (Farmersville, 1988-89).

{PRIVATE } E } Depth Inches	Mean total carbamate residue, mg/kg											
	Days after application											
	0	3	7	14	40	90	120	150	180	304	335	360
0-6	5.91	6.07	5.74	4.41	0.92	0.18	(0.02)	0.05	ND	ND	(0.03)	(0.03)
6-12	0.48	0.86	0.81	1.07	1.13	(0.02)	ND	(0.04)	ND	ND	ND	ND
12-18	ND	ND	ND	ND	0.51	(0.02)	ND	0.05	ND	ND	ND	ND
18-24	ND	ND	ND	ND	(0.02)	ND	ND	(0.03)	ND	ND	ND	ND
24-30	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
30-36	ND	ND	ND	ND	ND	ND	ND	(0.02)	ND	0.09	ND	ND
36-42	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.26	(0.02)	ND
42-48	ND	ND	ND	ND	ND	(0.02)	ND	ND	ND	0.25	ND	ND
48-54	NS	NS	NS	NS	0.08	ND	ND	(0.04)	ND	(0.02)	ND	ND
54-60	NS	NS	NS	NS	(0.04)	ND	ND	ND	ND	ND	ND	ND
60-66	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
66-72	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
72-78	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	(0.04)	ND
78-84	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
84-90	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND
90-96	NS	NS	NS	NS	ND	ND	ND	ND	ND	ND	ND	ND

NS: no soil core sample was taken

ND: undetectable (<0.02 mg/kg)

Values in parenthesis are estimated, below the limit of quantification (0.05 mg/kg) but above the limit of detection (0.02 mg/kg)

A third study was conducted in Porterville 1988-1989 (Leppert, 1989). The treated plot was 36 x 24 m (10 rows of 10 vines) and the control plot was 48 m from the treated plot. Core samples were taken before application and on days 0, 3, 7, 14, 50, 61, 90, 120, 150, 180, 307 and 387. The soil was classified as a sandy loam. Its characteristics are shown in Table 19.

Table 19. Soil characteristics, Porterville dissipation study.

Soil Depth (inches)	0-4"	4-8"	8-12"	12-24"	24-36"	36-48"	48-60"	60-72"	72-84"	84-96"
pH	7.27	7.36	7.51	7.52	7.51	7.45	7.41	6.94	6.81	7.24
% sand	64.6	68.8	71.6	75.6	76.2	77.2	66.2	24.2	28.0	32.0

% silt	22.4	18.2	16.4	12.2	12.8	10.8	17.8	47.8	40.0	31.0
% clay	13.0	13.0	12.0	12.2	11.0	12.0	16.0	28.0	32.0	37.0
CEC (meq/l)	4.1	4.0	4.1	5.0	4.4	4.3	6.0	17.3	15.8	17.6
% organic matter	0.38	0.26	0.13	0.2	0.13	0.1	0.2	0.42	0.37	0.15
Bulk density (g/cc)	0.92	0.95	1.05	1.35	1.3	1.32	1.29	1.25	1.32	1.38

The limit of detection was 0.02 mg/kg and the limit of quantification 0.05 mg/kg. More than 80% of the total residue in the soil was carbofuran. The levels of 3-hydroxy-carbofuran did not increase, and it was undetectable by day 50. Residues of 3-keto-carbofuran increased for 14 days in the 15 cm layer, then decreased to undetectable levels by 50 days. The average total carbamate residue in the top 0-15 cm ranged from 4.05 to 5.19 mg/kg during the first 14 days, with minimal leaching. During this period there was little or no rainfall. At the next sampling, day 50, the residues had almost disappeared from the top 15 cm layer with movement of low levels into the lower depths. By 61 days, corresponding to the start of rainfall and irrigation, the residues had permeated the soil strata from 0-240 cm. The levels found, however, were much lower than the original 0-15 cm residues suggesting that various factors such as soil microbial activity, pH and sorption reduced the movement, particularly of oxidized carbamates, through the soil. Quantifiable residues were not found below the 150 cm depth at any time except on days 61 and 150 when residues were found at low levels (0.19 mg/kg) in the 225-240 cm layer. No residues were detectable at any depth after 150 days. A half-life of 13 days was calculated from all of the 0-15 cm residues.

A summary of the results is shown in Table 20.

Table 20. Carbamate residues in soil dissipation study (Porterville, 1988-89).

Depth Inches	Mean total carbamate residue, mg/kg												
	Days after application												
	-1	0	3	7	14	50	61	90	120	150	180	307	387
0-6	ND	5.19	4.36	4.17	4.05	(0.04)	0.06	ND	ND	0.07	ND	ND	ND
6-12	ND	0.75	1.01	0.94	1.40	0.05	ND	ND	ND	0.08	ND	ND	ND
12-18	ND	ND	ND	ND	0.08	0.08	ND	ND	ND	0.06	ND	ND	ND
18-24	ND	ND	ND	ND	ND	0.09	0.05	ND	ND	0.05	ND	ND	ND
24-30	ND	ND	ND	ND	ND	0.08	0.11	ND	ND	(0.04)	ND	ND	ND
30-36	ND	ND	ND	ND	ND	(0.4)	0.15	ND	ND	0.05	ND	ND	ND
36-42	ND	ND	ND	ND	0.11	0.08	0.48	0.07	ND	ND	ND	ND	ND
42-48	ND	ND	ND	ND	0.71	0.12	0.64	0.08	ND	ND	ND	ND	ND
48-54 ¹	ND	NS	NS	NS	NS	(0.04)	0.24	0.07	ND	0.14	ND	ND	ND
54-60	ND	NS	NS	NS	NS	(0.02)	0.22	0.09	ND	0.17	ND	ND	ND
60-66	ND	NS	NS	NS	NS	ND	0.20	(0.02)	ND	0.32	ND	ND	ND
66-72	ND	NS	NS	NS	NS	ND	0.21	ND	ND	0.28	ND	ND	ND
72-78	ND	NS	NS	NS	NS	ND	0.19	(0.02)	ND	0.18	ND	ND	ND
78-84	ND	NS	NS	NS	NS	(0.04)	0.21	(0.04)	ND	0.21	ND	ND	ND
84-90	ND	NS	NS	NS	NS	0.05	0.06	(0.02)	ND	0.19	ND	ND	ND
90-96	ND	NS	NS	NS	NS	(0.02)	0.19	(0.04)	ND	0.17	ND	ND	ND

NS: no soil core sample was taken.

ND: undetectable (<0.02 mg/kg)

Values in parenthesis are estimated, below the limit of quantification (0.05 mg/kg) but above the limit of detection (0.02 mg/kg)

¹The sampling equipment may have caused contamination of the cores below 120 cm. The equipment could take only a 120 cm core, so to take a deeper core the probe had to be reinserted into the same hole after the 0-120 cm core had been taken. In some cases, leaf debris and root fragments were found where there should have been none

Adsorption/desorption was studied in a 0.01 M CaCl₂ solution with [¹⁴C]carbofuran labelled in the phenyl ring (Leppert, 1989). The nominal test concentrations were 0, 0.5, 1.5 and 10 mg/kg. The samples were maintained in an environmentally-controlled chamber at approximately 25°C. Two soils, a silt loam and a sandy loam, were used. Concentrations of carbofuran were estimated in the

aqueous phase by LSC and in the soils by combustion followed by LSC after the desorption phase. Adsorption and desorption constants were determined for the silt loam, but desorption could not be accurately determined for the sandy loam owing to the small amount of test material adsorbed during the adsorption phase. The mass balances for the silt loam and sandy loam were 106% and 104% respectively. The average K_{oc} of 24.7 indicates that carbofuran has the potential to be mobile in the two soils tested. The results are summarized below.

Soil type	% organic carbon	pH	Adsorption		Desorption	
			K_d	K_{oc}	K_d	K_{oc}
Silt loam	1.2%	7.1	0.246	20.5	0.243	20.3
Sandy loam	0.4%	6.5	0.115	28.9		

Column leaching study was conducted to determine the mobility of [*phenyl*- ^{14}C]carbofuran and its degradation products in four agricultural soils (Saxena *et al.*, 1994). The soils were a sandy loam from Georgia (GA), a clay loam and a loam from Ohio, and a sandy loam from California (CA). The CA sandy loam had an organic matter content of less than 1%. The characteristics of the soils are tabulated below.

Source	Type [USDA]	pH	% OM	CEC meq/100g	Sand, %	Silt, %	Clay, %	Bulk density, g/cm ³
Georgia	sandy loam	5.7	1.2	4.3	73	16	11	1.42
Ohio	clay loam	5.8	4.6	24.7	23	38	39	1.13
California	sandy loam	6.8	0.6	5.0	65	28	7	1.39
Ohio	loam	7.6	1.5	13.9	29	46	25	1.12

OM: organic matter

CEC: cation exchange capacity

A preliminary study was conducted to determine the rate of degradation of [^{14}C]carbofuran on each soil type, and hence the sampling intervals and the length of time for the aerobic aging-phase in the definitive study (one half-life or 30 days, whichever was shorter). The four soils were fortified with [^{14}C]carbofuran at a concentration of 3.2 mg/kg and incubated at $25 \pm 1^\circ\text{C}$ under aerobic conditions for 15 days. The soil moisture was maintained at approximately 75% of field capacity throughout the preliminary study. Duplicate soil samples were collected on days 0, 5, 10 and 15. The samples from days 5 and 10 were frozen upon collection. The samples from days 0 and 15 were extracted immediately after collection and the extracts analysed by HPLC. Since less than 20% of the carbofuran in the two sandy loams and the clay loam was degraded by day 15, the sampling points for these soils in the definitive study were days 0, 15, 22 and 30. In the loam soil approximately 41% of the carbofuran was degraded by day 15 so the samples from days 5 and 10 were extracted and analysed to determine the half-life of carbofuran, which was calculated to be 21.9 days. Samples in the definitive study were therefore taken on days 0, 9, 15, 20 and 23.

In the major study each of the four soils was fortified with [*phenyl*- ^{14}C]carbofuran at a concentration of 3.2 mg/kg (equivalent to 6.7 kg ai/ha, which represents the highest single application for row crops) and incubated aerobically at $25 \pm 1^\circ\text{C}$. The soil moisture was maintained at approximately 75% field capacity throughout the study. $^{14}\text{CO}_2$ and other volatile products were trapped and quantified. Duplicate soil samples were extracted immediately upon collection and analysed by HPLC.

The mean recoveries of the applied radioactivity and the half-life of carbofuran in each soil are shown in Table 21.

Table 21. Recoveries of ^{14}C and half-life of carbofuran in four soils.

Soil	^{14}C recovered, %				Half-life, days
	Extracted	Volatile	Bound	Total	
GA sandy loam	87.6	0.1	9.9	97.6	90.8
clay loam	73.1	2.7	22.0	97.8	53.0
CA sandy loam	86.2	2.4	9.2	97.8	99.9
loam	49.4	4.1	42.4	95.9	21.9

The proportion of the recovered radioactivity associated with each of the compounds determined by HPLC is shown in Table 22.

Table 22. Distribution of recovered ^{14}C .

Soil	^{14}C , % of recovered and mg/kg as carbofuran		
	Carbofuran, %	3-OH-carbofuran (% mg/kg)	3-keto-carbofuran (% , mg/kg)
GA sandy loam, day 30	81.6	2.1 (0.07)	1.6 (0.05)
Clay loam, day 30	67.1	0.2 (<0.01)	2.8 (0.09)
CA sandy loam, day 30	79.1	0.8 (0.03)	3.4 (0.11)
Loam, day 23	46.1	0.1 (<0.01)	0.1 (<0.01)

The remaining radioactivity was distributed among soil-bound residues and $^{14}\text{CO}_2$. The identification of 3-ket^oCarbofuran was confirmed by LC-MS.

The remaining soil samples of each soil from the definitive study were combined and used as the aged soil in the leaching study. The soils were packed in columns to a height of 30 cm and the aged soil containing the [^{14}C]carbofuran residues at a nominal concentration of 6.7 kg ai/ha was applied to each column. The columns were maintained at approximately $25 \pm 1^\circ\text{C}$ and leached with 50 column cm of 0.01 N CaCl_2 at an approximate rate of 1.5 cm per hour. Four fractions of leachate (approximately 12.5 cm or 600 ml each) were collected from each column. The leachates and the soils in the columns were assayed for radioactivity. The proportion of the applied radioactivity in the leachates and soil sections were as follows.

% of radioactivity applied to column

	Georgia		California	
	sandy loam	Clay loam	sandy loam	Loam
Aged soil layer	42.5	31.1	13.7	49.2
Total in six soil sections	9.4	26.5	2.8	9.0
Total in leachates	53.4	40.9	78.2	33.2
Mass balance	105.3	98.5	94.7	91.4
K_d	0.73	*	0.25	*

*less than 50% of the applied radioactivity was in the leachate

Leachate fractions that contained >10% of the applied ^{14}C were analysed by HPLC. The identity of carbofuran was also confirmed by two-dimensional TLC. More than 94% of the ^{14}C from all four soils was due to [^{14}C]carbofuran. Minor components (less than 1% of the applied radioactivity) detected in the leachates and/or aged layer sections included 3-keto-carbofuran (0.8%) the 7-phenol (0.2% in GA sandy loam, 0.2% in clay loam, 0.5% in loam), the 3-keto-7-phenol (0.2%) and 3-keto-carbofuran (91.8% in CA sandy loam).

The proportion of leached radioactivity was greatest in the CA sandy loam, followed by GA sandy loam, clay loam and loam. The results indicate that carbofuran and its degradation products have the potential to be mobile in all four soils under the "worst-case" conditions of applying 50 column cm of water.

Environmental fate in water/sediment systems

Cook (1974) studied the hydrolysis of [*phenyl* ^{14}C]carbofuran in aqueous solutions buffered to pH 5, 7 and 9 at a concentration of 2 mg/l. At room temperature (28°C), carbofuran was hydrolytically stable over the 28-day test period at pH 5 and was slowly hydrolysed at pH 7 with a calculated half-life of 26 days. At pH 9 only 20% of the carbofuran remained after 1 day at 26°C and the half-life was 12 hours. At 5°C the half-life was 1.5 days. The hydrolysis product was the 7-phenol.

Degradation in water/sediment systems

In a study in accordance with US EPA Guidelines (Saxena *et al.*, 1994b) the rate and degree of the anaerobic aquatic degradation of [^{14}C]carbofuran was determined in acidic pond water plus sediment systems (approximate pH 5.4) consisting of approximately 82 g wet sediment, equivalent to 50 g oven-dried sediment and 100 ml of pond water in sealed bottles. Test systems were prepared and incubated under anaerobic conditions in the dark at approximately 25°C for at least 30 days (pre-anaerobic incubation) before adding [^{14}C]carbofuran uniformly labelled in the phenyl ring at a nominal concentration of approximately 3 mg/kg (equivalent to 6.7 kg ai/ha) and incubating at 25°C under anaerobic conditions for 12 months in the dark. The test systems were incubated in a "static" anaerobic apparatus that permitted the trapping of organic volatiles and $^{14}\text{CO}_2$. Duplicate samples of sediment plus water were collected immediately after dosing (day 0) and after 1, 3, 7, 14, 31, 60, 98, 122, 183, 273 and 365 days. The samples were flushed with nitrogen to collect organic volatiles, $^{14}\text{CO}_2$ and [^{14}C]methane and the dissolved oxygen content, pH and redox potential of the water samples were determined. The populations of aerobic and anaerobic microbes were also measured. The sediment and water were extracted and analysed by HPLC. The identities of compounds that accounted for more than 10% of the applied radioactivity were confirmed by liquid chromatography-

mass spectrometry (LC-MS). The extracted sediment was combusted to determine the amount of bound radioactivity.

More than >90% of the ^{14}C was accounted for in all the samples. Volatile radioactivity was negligible and reached a maximum of 0.5% by day 273. [^{14}C]carbofuran decreased from 96.2% at day 0 to 24.9% by day 365. One major product, the 7-phenol, reached a maximum of 53.7% by day 365, when the maximum level of 20.4% of the applied radioactivity was observed. Fractionation of the bound residues into humic acid, fulvic acid and humin indicated the presence of radioactivity in all three fractions. The observed redox potential and dissolved oxygen values indicated that anaerobic conditions were maintained. A summary of the results is given in Table 23.

Table 23. Anaerobic degradation in a water/sediment system.

Day	% of ^{14}C as					
	carbofuran	3-keto-7-phenol	7-phenol	Trapped volatiles	Bound residues	Total
0	96.2	ND	0.4	NA	0.3	96.8
1	91.1	ND	0.4	0.1	0.8	92.3
3	90.9	0.1	0.5	0.1	1.1	92.6
7	90.4	0.3	0.9	0.3	3.0	94.8
14	85.9	0.1	0.8	0.2	7.8	94.7
31	75.9	0.4	9.6	0.2	11.8	97.8
60	64.7	0.4	17.9	0.3	11.7	94.9
98	55.5	ND	26.6	0.3	14.3	96.7
122	47.0	ND	30.9	0.3	16.1	94.3
183	41.9	ND	39.3	0.3	16.7	98.2
273	34.0	ND	45.3	0.5	18.8	98.6
365	24.9	ND	53.7	0.2	20.4	99.1

The average half-life of [^{14}C]carbofuran in the test system, assuming first order kinetics, was approximately 189 days.

The rate and degree of aerobic degradation of [^{14}C]carbofuran uniformly labelled in the phenyl ring was determined in an acidic pond water/sediment system (approximate pH 5.4) by Saxena and Marengo (1994). Each vessel contained 50 g of pond sediment and 100 ml of pond water fortified with [^{14}C]carbofuran at a concentration of 3.05 mg/kg (equivalent to 6.7 kg ai/ha which represents the highest single application for row crops) and incubated at $25 \pm 1^\circ\text{C}$ under aerobic conditions for 30 days in darkness. The vessels were connected in pairs to a set of traps (ethylene glycol for organic volatiles, sodium hydroxide for CO_2) and CO_2 -scrubbed humidified air was bubbled through the overlying water of the first vessel of each pair into the water of the second and then into the traps.

Pairs of sediment/water vessels were taken on days 0, 1, 3, 7, 10, 20 and 30 and the contents analysed immediately upon collection. The population of aerobic bacteria, pH, dissolved oxygen content and redox potential of the test system were determined, the radioactivity in the traps was counted by LSC, that in the sediment and water was extracted and the extracts analysed by HPLC and the extracted sediment was combusted to determine bound ^{14}C . Selected extracts were also analysed by TLC to confirm the products detected by HPLC. The identities of compounds accounting for more than 10% of the applied radioactivity were confirmed by LC-MS.

The recovery of applied radioactivity from individual samples was >90% at all times. The distribution of the radioactivity at 0 and 30 days was as follows.

<u>Mean % of applied radioactivity</u>					
	Water	Sediment	Extracted		
Day	layer	extract	sediment	Traps	Total
0	71.07	24.81	3.92	0	99.8
30	15.29	45.86	32.78	1.87	95.8

The first sample in each pair remained acidic throughout the 30-day study with a pH of about 5, and the second sample remained acidic on days 0-10. A shift in the pH of the overlying water in the second vessel of the pair to about 8 was observed at days 20 and 30. A significant difference between the two samples in the degradation of carbofuran was caused by the shift in pH (carbofuran is known to be hydrolysed rapidly at an alkaline pH to the 7-phenol).

The 7-phenol was a major product in the two alkaline samples on days 20 and 30 (23.74 and 17.30% of the applied radioactivity respectively, compared with 0.61 and 1.89% in the first vessels). The other major degradation products were soil-bound residues which accumulated to an average of 32.67% by day 30. Fractionation of the 30-day sediments into humic acid, fulvic acid and humin indicated the presence of radioactivity in all three fractions. Carbofuran decreased to an average of 39.65% by day 30. Minor amounts (<1%) of 3-hydroxy-carbofuran, the 3-keto-7-phenol and 3-keto-carbofuran were detected in all the samples. No unidentified compounds were detected.

An additional study was conducted to determine whether the original arrangements of the vessels in pairs caused the second samples to become alkaline and hence the differences on days 20 and 30. Each vessel, with the same contents as before, was now connected to its own set of traps. Duplicate vessels were collected on days 0, 10, 20 and 30. The contents of the day 0 and day 30 vessels were analysed immediately upon collection as before. The pH, dissolved oxygen and redox potential were measured at days 10 and 20 and the samples were then stored frozen without further analysis.

The mass balance was >90% of the applied radioactivity in the day 0 and day 30 samples. A slight increase in the pH of the overlying water with time was observed in the individual vessels. The pH of the individual water samples at day 0 ranged from 5.23 to 5.50 and a maximum pH of 6.44 was observed in any individual sample during incubation. The 7-phenol was detected at a maximum level of 2.88% and a mean of 2.84% by day 30. No unidentified compounds were detected, the analyses of the duplicate samples at each time interval agreed, and the water samples remained acidic throughout the study. Evidently the connection of the vessels in sequence in the main study caused the pH change to alkaline and the differences between "replicates" on days 20 and 30.

The half-life of carbofuran was calculated by linear regression to be approximately 41 days.

Aqueous photolysis was studied with [¹⁴C]carbofuran labelled in the phenyl ring at a concentration of 20 mg/l in a sterile buffer solution at pH 5 (McGovern and Shepler, 1989a). The samples were exposed to natural sunlight in a water bath at approximately 25°C together with control samples wrapped in aluminum foil. Duplicate irradiated and control samples were analysed 0, 3, 6, 12, 20 and 31 days after treatment. Ethylene glycol and 10% NaOH were used to trap volatile organic compounds and CO₂ respectively. Air was drawn through both the irradiated and control sample tubes into separate sets of traps. All samples were analysed directly by LSC and HPLC. The average recovery of ¹⁴C from all samples was 97.1%. The 7-phenol and CO₂ were the only degradation products observed. The 7-phenol reached a maximum of 3.7% and CO₂ a maximum of 0.3% of the applied ¹⁴C in the irradiated samples. The extrapolated half-life for photolysis was 1200 days, implying a half-life of 450 days in summer daylight conditions. Carbofuran was also found to be

degraded slowly in the dark to the 7-phenol and CO₂ with a half-life of 2100 days, showing that the 7-phenol is not photochemically derived wholly from photolysis.

An aquatic field dissipation study was conducted in the USA in Louisiana and California (Novak, 1987a,b) to determine the distribution of carbofuran and its metabolites in soil, water and rice. Each site consisted of two 20 x 30 m rice plots, one control and one treated, surrounded by a levee. At the Louisiana site, the rice plots were flooded to a depth of 10 cm when the rice plants had reached this height, and the depth of the flood water was maintained between 8 and 21 cm until the rice was mature. The Louisiana plot was treated with Furadan 3G at 0.67 kg ai/ha 19 days after permanent flooding of the planted rice. The California plot was treated with Furadan 5G at 0.56 kg ai/ha immediately after sowing the rice seed before flooding. The plots were flooded to a depth of 20 cm and maintained at 15 to 20 cm. At both sites, an additional plot was planted with crops and irrigated with water from the treated rice plot.

At the Louisiana site, soil core samples were taken from the treated plot to a depth of 64 cm before and 24 hours after application, and then at days 3, 7, 14, 21, 30, 60 and 120. At the California site, soil samples were taken at the same intervals and also at days 162, 196 and 225. The soil cores were taken in the treated plot to a depth of 64 cm during the unflooded phase and 16 cm during the flooded phase. The soil cores were divided and analysed in 8 cm sections. Water samples were taken just before and 8 hours after application at both sites and on days 7, 14, 21, 30, 60 and 99 in Louisiana and 3, 7, 9, 12, 14, 16, 19, 21 and 27 in California. Four 1-l water samples were taken at each sampling at each site.

The soil samples were analysed for carbofuran and 3-hydroxy-carbofuran (Schreier, 1987). The stated limits of determination and detection were 0.1 and 0.02 mg/kg. The analyses showed only low levels (≤ 0.09 mg/kg) of carbofuran at both sites, and 3-hydroxy-carbofuran was not detected in any of the samples. The maximum residue of carbofuran at the California site, 0.09 mg/kg, occurred 7 days after treatment in the 0-8 cm section. Only the 1-60 days sample at the California site were analysed: both analytes were undetectable to a depth of 16 cm at 60 days. The maximum residue of carbofuran at the Louisiana site was 0.04 mg/kg in the 0-8 cm section after 3 days. Only the 1-21 day samples were analysed because both compounds were undetectable to a depth of 16 cm in the 7-21 day samples. The results are shown in Table 24.

Table 24. Aquatic field dissipation: carbofuran and 3-hydroxy-carbofuran in soil and sample.

Site/Interval/Depth	Residue, mg/kg	
	carbofuran	3-hydroxy carbofuran
Louisiana		
Day 1 0-8 cm	(0.04)	ND
Day 1 8-16 cm	ND	ND
Day 3 0-8 cm	(0.04)	ND
Day 3 8-16 cm	ND	ND
Day 7 0-8 cm	ND	ND
Day 7 8-16 cm	ND	ND
Day 14 0-8 cm	ND	ND
Day 14 8-16 cm	ND	ND
Day 21 0-8 cm	ND	ND
Day 21 8-16 cm	ND	ND
California		
Day 1 0-8 cm	(0.05)	ND
Day 1 8-16 cm	ND	ND
Day 3 0-8 cm	(0.06)	ND
Day 3 8-16 cm	ND	ND

Site/Interval/Depth	Residue, mg/kg	
	carbofuran	3-hydroxy carbofuran
Day 7 0-8 cm	(0.09)	ND
Day 7 8-16 cm	ND	ND
Day 14 0-8 cm	(0.07)	ND
Day 14 8-16 cm	ND	ND
Day 21 0-8	0.05	ND
Day 21 8-16 cm	ND	ND
Day 30 0-8 cm	(0.07)	ND
Day 30 8-16 cm	ND	ND
Day 60 0.8 cm	ND	ND

ND: undetectable (<0.02 mg/kg)

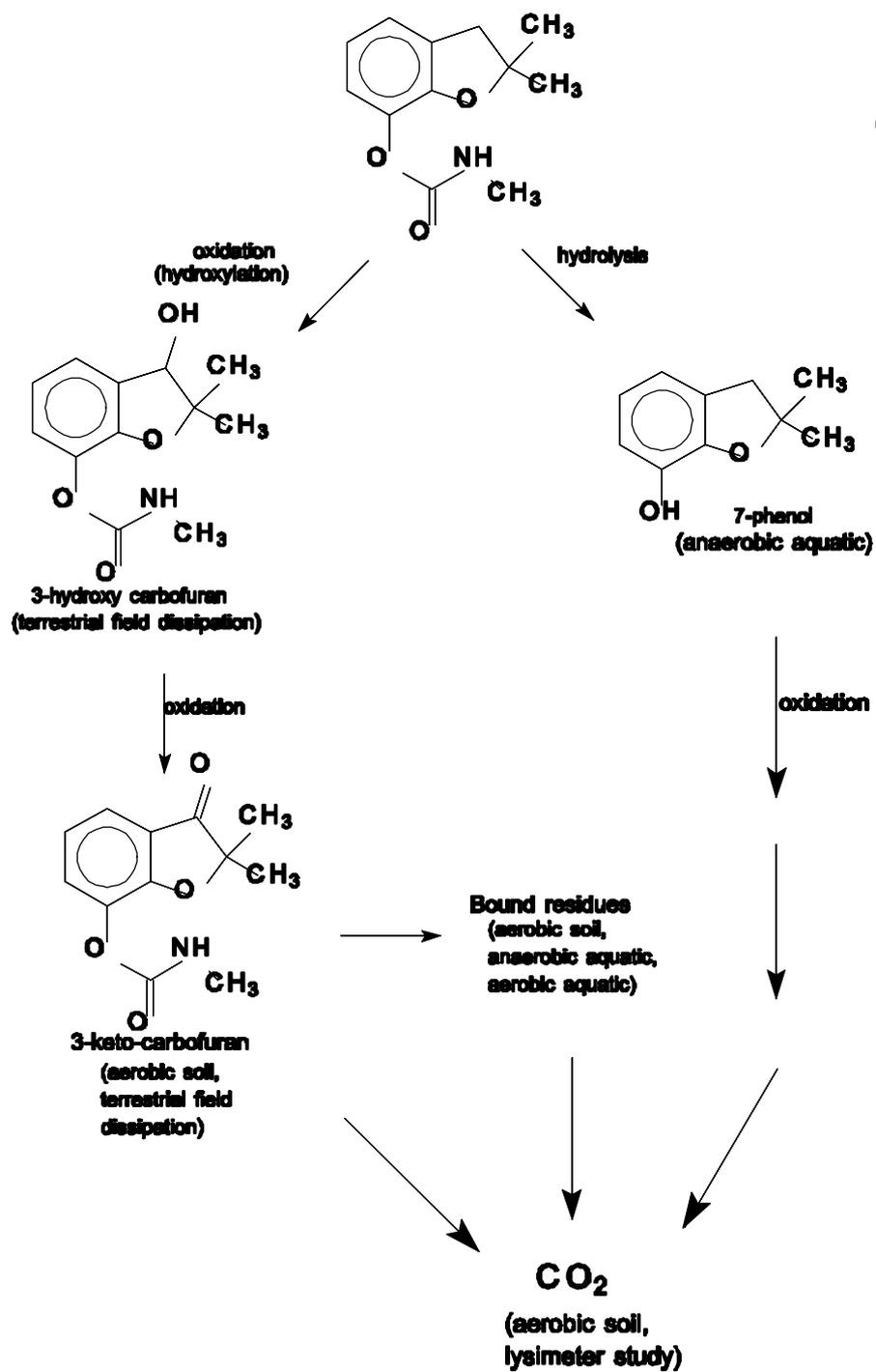
Results in parenthesis are estimated levels below the limit of determination (0.1mg/kg) but above the limit of detection (0.02 mg/kg)

Rice paddy water samples were also analysed for carbofuran and 3-hydroxy-carbofuran (Beauchamp, 1987). The limit of determination was 2.5 $\mu\text{k}/\text{kg}$ and the limit of detection 1 $\mu\text{k}/\text{kg}$. The water from the Louisiana site contained a mean maximum level of carbofuran of 417 $\mu\text{k}/\text{kg}$ after 8 hours, which decreased to 3 $\mu\text{k}/\text{kg}$ after 30 days. 3-Hydroxy-carbofuran was detected only in one sample, at 1.0 $\mu\text{k}/\text{kg}$ on day 7. The water from the California site contained a mean maximum level of carbofuran of 33 $\mu\text{k}/\text{kg}$ after 8 hours which became undetectable by day 27. The half-life of carbofuran in rice paddy water was <10 days at both sites.

Carbofuran dissipated rapidly in soil and water after application to rice plots. Carbofuran was the only residue found in the soil and was undetectable by day 60. In the water, residues of carbofuran were ≤ 3 mg/kg by day 30 and no residues of 3-hydroxy-carbofuran were found except for a level of 1 $\mu\text{k}/\text{kg}$ on day 7. No residues of carbofuran or 3-hydroxy-carbofuran were found in the rice grain or straw.

Proposed degradation pathways of carbofuran in soil and water/sediment systems are shown in Figure 4.

Figure 4. Degradation pathways of carbofuran in soil and water/sediment systems.



Rotational Crops

In a confined crop rotation study [*phenyl*-¹⁴C]carbofuran was applied directly to a silt loam soil at an application rate of 3.4 kg ai/ha, based on a 76 cm row space. Wheat, soya beans and sugar beet were seeded into the treated soil 4 and 12 months after treatment and grown to maturity. Wheat forage, straw and grain, soya bean silage, stems, pods and beans and sugar beet tops and roots from both plantings were assayed separately for ¹⁴C. Table 25 shows that residues above 0.01 carbofuran equivalents were found in all the samples from both plantings.

Table 25. Total radioactive residues in mature rotational crops, as carbofuran.

Crop	Sample	¹⁴ C, mg/kg as carbofuran	
		4 months	12 months
Wheat	Forage	-	1.40
	Straw	54.0	0.30
	Grain	0.60	0.04
Soya bean	Silage	16.0	0.50
	Stem	18.0	0.70
	Pod	5.0	0.10
	Bean	1.0	0.08
Sugar beet	Top	0.40	0.05
	Root	0.20	0.05

Subsamples of each plant part were extracted with methanol/water (1:2) and separated into non-polar and polar fractions which were concentrated and analysed separately to determine the nature of the residues. Conjugated metabolites were hydrolysed with 0.25 N hydrochloric acid. Metabolites were identified by TLC, with co-chromatography with reference standards.

The phenolic metabolites (3-hydroxy-7-phenol, the 3-keto-7-phenol and 7-phenol) were the principal degradation products found in the plants. The carbamates (carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran) constituted a small proportion of the total radioactive residue; none of them individually exceeded 10% of the TRR in any of the crops sown at 4 or 12 months.

A field rotational crop study was conducted with [*phenyl*-¹⁴C]carbofuran applied to the soil at rates of 1.1, 3.4 and 6.7 kg ai/ha. Ten months after treatment, sorghum, soya beans, sugar beet, lettuce, cabbage and wheat were planted in the field and grown to maturity. No ¹⁴C was detectable in mature sorghum, soya beans, sugar beet, wheat grain or lettuce from any of the three application rates. Low levels of the total residue (0.01 mg/kg as carbofuran) were observed in mature cabbage harvested from the 3.4 and 6.7 kg ai/ha treatments. Wheat straw and soya bean stems harvested from the 6.7 kg ai/ha treatment contained 0.21 mg/kg and 0.63 mg/kg respectively, but residues were not detectable at the two lower treatment rates. Residues in the immature crops from the 1.1 and 3.4 kg ai/ha treatments harvested 30 and 58 days after planting were generally below the detection limit. Detectable levels of radiocarbon (0.017-0.084 mg/kg as carbofuran) were found in immature crops from the 6.7 kg ai/ha treatment. Low levels of carbofuran (maximum 0.02 mg/kg) remained in the soil ten months after application.

The results indicate that under normal field use conditions the potential for accumulation of carbofuran into ten-month rotational crops is minimal at application rates of 1.1-6.7 kg ai/ha. The residues from all treatment rates were below the limit of detection in all the edible commodities except cabbage where they were at the limit of detection at the two higher treatment rates.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Schreier (1989) provided a GLC method for the determination of carbofuran, 3-hydroxy-carbofuran and 3-keto-carbofuran in green and dry alfalfa, field corn silage and grain, oranges, peanut nutmeat and hulls, potato tubers, sorghum, sugar beet roots and tops and cow milk and muscle. The weighed sample was macerated, hydrolysed by refluxing for one hour with 0.25 N HCL and filtered. The filtrate was partitioned with dichloromethane, transferred to hexane and cleaned up on a Florisil column. Ethyl acetate was used to elute the compounds of interest. The final solutions were analysed on a 10 or 12 m methyl silicone capillary column, either 0.53 mm with direct injection and a nitrogen-phosphorus detector (NPD) or 0.2 mm with splitless injection and a mass-selective detector (MSD). Calibration was with external standards. Chromatograms and raw data were provided for fortified control samples to demonstrate the limits of determination given in Table 26. Limits of determination were also claimed for milk and muscle (0.5 mg/kg for each analyte) and several plant crops, but no chromatograms or data were submitted.

Table 26. Limits of determination of carbofuran and carbamate metabolites by the method of Schreier (1989).

{PRIVATE }Sample	Detector	Limit of determination, mg/kg		
		Carbofuran	3-hydroxy-carbofuran	3-keto-carbofuran
Maize silage	MSD	1.0	3.0	1.0
Peanut kernels	MSD	0.5	0.5	0.5
Peanut hulls	MSD	2.0	2.0	2.0
Potato	MSD	0.5	0.5	0.5
Sorghum	NPD	1.0	1.0	1.0
Sugar beet tops	NPD	1.0	1.0	1.0

Modifications to the Schreier method, e.g. by Brutschy (1984) included ethoxylation of 3-hydroxy-carbofuran and a procedure for the isolation and determination of the phenol metabolites. For the determination of phenol metabolites the hydrolysed and filtered sample was partitioned with methylene chloride/diethyl ether (3:3), butylated hydroxytoluene (1 ml of 10 mg/kg in methylene chloride) and ethanol were added to the extract and the solution was concentrated to remove methylene chloride, acidified, and refluxed for 45 minutes. It was then partitioned into methylene chloride, which was concentrated, cleaned up on a silica gel solid-phase extraction column conditioned with methanol/water (1:1) and eluted with methylene chloride. The eluate was concentrated and analysed by GLC on a cross-linked dimethyl silicone capillary column operated in the splitless mode. Detection was mass-selective with monitoring of the molecular ions of the 7-phenol, 3-keto-7-phenol and 3-ethoxy-7-phenol (164^+ , 178^+ and 208^+). Calibration was with external standards.

Mollhoff (1975a) described a method for the determination of carbofuran, 3-hydroxy-carbofuran and 3-hydroxy-carbofuran glycoside in plants and soil. Plant samples (cereal grains, potatoes) were macerated with methanol and the macerate filtered, concentrated and extracted with chloroform. The residual aqueous fraction was hydrolysed with acid to convert any glycoside conjugate to 3-hydroxy-carbofuran aglycone and extracted with chloroform. Soil samples were macerated with a mixture of methanol, water and hydrochloric acid and extracted with chloroform. Analyses were by GLC on a packed column (Ucon LB 550 X with 0.5% KOH on Chromosorb G AW DMCS). A limit of determination of 0.1 mg/kg claimed and recoveries were reported for several

crops, but no data were provided. Recoveries of the conjugate were generally unacceptable at or below 0.6 mg/kg. Several acceptable recoveries of the conjugates were reported at 1.0 mg/kg.

Leppert *et al.* (1983) reported a GLC method for the determination of carbosulfan and carbofuran residues in soil, plants and water. Crops with a high water content, e.g. green alfalfa or citrus, were blended with hexane and 2-propanol (2:1), diluted with water and the hexane fraction retained. Soil and crops with a low water content, e.g. hay and straw were blended with methanol and pH 8 buffer and the filtered solution was extracted with methylene chloride. Oily samples, e.g. citrus oil, were diluted with hexane and extracted with acetonitrile. Water samples were extracted with methylene chloride after salting. Various column clean-up procedures were used, including gel permeation, Darco-Attaclay, aluminum oxide and Florisil. After the Darco-Attaclay fractionation, the ethyl acetate eluate was concentrated and treated with ethanol and concentrated HCl to ethoxylate 3-hydroxy-carbofuran. The final extracts were analysed on a packed column of Chromosorb W-HP with nitrogen-selective detection. A limit of determination of 0.1 mg/kg carbofuran was reported for citrus fruit.

Smith (1991) reported a method for the determination of parts-per-billion levels of carbofuran in water. Samples were concentrated on a C-18 solid-phase extraction column, eluted with acidified methanol and analysed with by HPLC on a cyclohexyl column with a UV detector (220 nm). The mobile-phase was a water/acetonitrile gradient. Adequate resolution and sensitivity were demonstrated for a rice-water sample fortified with carbofuran at 11 µg/kg .

Barros (1995) described a multi-residue method for the determination of carbosulfan and its metabolites in or on oranges. In addition to carbosulfan, the method determines carbofuran, 3-keto-carbofuran, and the 3-hydroxy-carbofuran, 3-keto-7-phenol, 7-phenol and 3-hydroxy-7-phenol.

To determine the carbamates, macerated oranges were hydrolysed with 0.25 N HCl under reflux, the mixture was filtered and an aliquot of the filtrate was loaded onto a C-18 solid-phase extraction cartridge conditioned with methanol and 0.25 N HCl. The compounds of interest were eluted with 1% methanol in methylene chloride and passed through an aminopropyl solid-phase cartridge. The final residue was re-dissolved in acetonitrile and analysed by reverse-phase HPLC (C-18) with a post-column reactor and fluorescence detector. The demonstrated limit of determination was 0.03 for each analyte.

To determine the phenols, a separate aliquot of the original filtrate was loaded onto a C-18 cartridge and the dried cartridge was eluted with 5% ethanol in methylene chloride. The phenols were derivatized with pentafluorobenzyl bromide and the 3-hydroxy-7-phenol derivative was ethylated. A final ethanol solution of the analytes was analysed by gas chromatography with a mass-selective detector. The demonstrated limit of determination was 0.03 mg/kg for each analyte.

Geno (1991) reported validation of the Barros method for maize silage. The independent laboratory validation was in accordance with US EPA PR Notice 88-5, 40 CFR Part 160. Control maize silage was fortified with 0.05 or 0.25 mg/kg each of carbofuran, the 3-keto-carbofuran and 3-hydroxy-carbofuran. Adequate recoveries were demonstrated for all three compounds (83-102%, 95-102% and 96-108% respectively). The method was not validated for the phenol metabolites.

Blass and Philipowski (1992) reported a method for the determination of methylcarbamate residues, including carbofuran, by HPLC with post-column reaction. Samples with little or no fat were extracted with methylene chloride/water and fatty samples with acetonitrile saturated with hexane. The latter extract was washed with hexane, concentrated and extracted with methylene chloride. The final organic extracts were cleaned up on an "Extrelut" cartridge. Aqueous extracts

were prepared for the determination of 3-hydroxy-carbofuran. The analytes were separated on a Spherisorb RP 18 column and the eluted methylcarbamates converted in a two-stage reactor to (1-hydroxyethylthio)-2-methylisoindole and the indole measured with a fluorimeter (excitation 340 nm, emission 455 nm). The limit of determination is approximately 0.04 mg/kg for carbofuran and 3-hydroxy-carbofuran. The recoveries given in Table 27 were reported for various samples fortified with carbofuran and 3-hydroxy-carbofuran. Sample chromatograms were provided from barley grain, wheat straw, sugar beet foliage and lettuce.

Table 27. Recoveries of carbofuran and 3-hydroxy-carbofuran by the method of Blass and Philipowski (HPLC with post-column derivatization).

Sample	Fortification, mg/kg	Recovery, %	
		Carbofuran	3-hydroxy-carbofuran
Apple	0.04; 1.0	84; 100	
Beet foliage	0.04; 1.0	76; 85	73; 84
Carrot	0.04; 1.0	94; 94	79; 87
Cherry	0.04; 1.0	90; 85	
Maize grain	0.04; 0.1; 1.0	99; 99; 97	100; 98; 95
Lettuce	0.04; 1.0	92; 95	88; 89
Melon	0.04; 1.0	92; 84	78; 75
Pepper	0.04; 1.0	86; 91	78; 80
Potato	0.04; 1.0	79; 97	78; 80
Rice	0.04; 1.0	91; 91	90; 90
Soya beans	0.04; 1.0	96; 91	101; 98
Wheat straw	0.10; 1.0	102; 95	
Sunflower seed	0.04; 0.1; 1.0	92; 94; 97	86; 78; 94
Barley grain	0.04; 1.0	100; 96	
Asparagus	0.04; 1.0	104; 92	80; 84
Bulb onion	0.04; 1.0	82; 90	74; 83
Tomato	0.04; 1.0	94; 93	86; 76
Sugar beet foliage	0.04; 1.0	85; 91	78; 85
Sugar beet root	0.04; 1.0	90; 90	80; 83

The sponsors claim that the Blass method is the official enforcement screening method for use in Europe (see the official multi-residue methods of The Netherlands, below).

A multi-residue method is published in the US Food and Drug Administration (FDA) Pesticide Analytical Manual (PAM) for determining total residues of carbofuran in food for the enforcement of tolerances.

Chen (1995a) described a method for the determination of carbosulfan and its metabolites, including carbofuran and the metabolites of Figure 1, in ruminant commodities. Milk and tissues are extracted with acetone. The acetone extract is centrifuged and cleaned up by a combination of liquid-liquid extraction, solid-phase extraction and/or gel permeation chromatography. Carbofuran and carbamate metabolites are determined by HPLC with a post-column reactor and fluorescence detector. The phenolic metabolites are extracted and analysed by a similar procedure to that of Barros (GC-MS). Limits of determination of 0.025-0.50 mg/kg were demonstrated for carbofuran and the metabolites in milk and tissues. The recoveries from fortified controls are shown in Table 28.

Table 28. Recovery of carbofuran and its carbamate and phenol metabolites from milk and ruminant tissues by the method of Chen (1995a).

Sample	Fortificn., mg/kg	No. of samples	Recovery, %					
			carbofuran	3-K-CF	3-OH-CF	7-phenol	3-K-7-P	3-OH-7-P
Milk	0.025	19	93 ± 13	92 ± 11	84 ± 13			
Milk	0.025	18				88 ± 12	99 ± 11	104 ± 15
Muscle	0.050	4	88 ± 14	97 ± 17	94 ± 14			
Muscle	0.050	2				70	100	80
Kidney	0.050	2	88	102	72			
Kidney	0.050	2				78	103	91
Kidney	0.50	2				87	114	85
Fat	0.050	3	76 ± 1.5	89 ± 9.2	71 ± 8.6			

Abbreviated compound names: see Figure 1, p.

The Netherlands submitted official multi-residue methods for the determination of carbofuran and 3-hydroxy-carbofuran (The Netherlands, 1997). Fruits, vegetables and potatoes were chopped, homogenized and extracted with acetone/methylene chloride/petroleum ether (1:1:1). Nuts, cereals, oil seeds, tropical seeds and dried fruits were extracted with acetone/methylene chloride (1:1). The extracts were analysed without clean-up, by gas chromatography with electron capture or ion trap detection. The limits of determination were stated to be in the range of 0.01-0.05 mg/kg, with recoveries of >80%. A second, HPLC method, consisted in extraction with acetone, partitioning into methylene chloride/petroleum ether and clean-up on a solid-phase extraction cartridge if necessary. The final extract was analysed on a reverse-phase HPLC column, with post-column hydrolysis and derivatization of the resulting amine with *o*-phthaldialdehyde. Detection was by fluorescence at 340 and 455 nm. The stated limit of determination was 0.005 mg/kg. The HPLC method is essentially that of Blass and Philipowski (1992).

The Netherlands also reported two methods for the determination of residues in field trial samples (The Netherlands, 1997). The GLC method is that of Mollhoff (1975a). A limit of determination of 0.1 mg/kg was reported for carbofuran, metabolites and conjugates. This method was used for strawberries, red cabbage, onions, leek, celery, celeriac, cauliflower, carrots and Brussels sprouts. The second method consisted in extraction with methylene chloride, concentration, clean-up on alumina and determination of carbofuran and 3-hydroxy-carbofuran of a reverse-phase HPLC column with a UV detector (220 nm). The limits of determination were 0.02 mg/kg for carbofuran and 0.01 mg/kg for 3-hydroxy-carbofuran. Recoveries were reported to be 95 ± 6% (n = 6) for carbofuran at fortifications of 0.06-1.1 mg/kg and 85 ± 7% (n = ?) for 3-hydroxy-carbofuran at fortifications of 0.05-0.50 mg/kg.

Stability of pesticide residues in stored analytical samples

Storage stability studies were reported for green and dry alfalfa, maize grain and silage, oranges, peanut kernels and hulls, potatoes, sorghum stalks, sugar beet tops and roots and cow milk and muscle (Schreier, 1989b). Fortified control samples were stored at -18°C for about 2 years. Samples were analysed after intervals of 9-11 and 24-26 months by the method of Schreier (1989a). The results are shown in Table 29. [CLICK HERE to continue](#)

Table 29. Frozen storage stability of carbofuran and its metabolites added to various commodities.

Sample	Analyte	Fort. ¹ mg/kg	Recovery, %	
			9-11 months	24-26 months
Alfalfa, green	carbofuran	1.0	103	90
	3-keto-carbofuran	1.0	105	100
	3-hydroxy-carbofuran	8.0	60	99
Alfalfa, dry	Carbofuran	2.5	86	94
	3-keto-carbofuran	2.5	85	93
	3-hydroxy-carbofuran	20.	80	100
Maize grain	Carbofuran	0.5	94	113
	3-keto-carbofuran	0.5	104	110
	3-hydroxy-carbofuran	0.5	116	115
Maize forage	Carbofuran	1.0	106	105
	3-keto-carbofuran	1.0	113	112
	3-hydroxy-carbofuran	3.0	131	101
Orange (whole)	Carbofuran	0.5	108	107
	3-keto-carbofuran	0.5	104	98
	3-hydroxy-carbofuran	0.5	86	92
Peanut kernels	Carbofuran	0.5	94	83
	3-keto-carbofuran	0.5	90	79
	3-hydroxy-carbofuran	0.5	86	102
Peanut hulls	Carbofuran	2.0	105	116
	3-keto-carbofuran	2.0	105	89
	3-hydroxy-carbofuran	2.0	105	104
Potato, tuber	Carbofuran	0.5	96	86
	3-keto-carbofuran	0.5	104	75
	3-hydroxy-carbofuran	0.5	96	79
Sorghum, stalk	carbofuran	1.0	84	93
	3-keto-carbofuran	1.0	111	87
	3-hydroxy-carbofuran	1.0	78	79
Sugar beet tops	carbofuran	1.0	88	86
	3-keto-carbofuran	1.0	47	46
	3-hydroxy-carbofuran	1.0	85	79
Sugar beet root	carbofuran	0.5	110	99
	3-keto-carbofuran	0.5	94	72
	3-hydroxy-carbofuran	0.5	96	97
Cow milk	carbofuran	0.5	96	97
	3-keto-carbofuran	0.5	106	89
	3-hydroxy-carbofuran	0.5	98	95
Cow muscle	carbofuran	0.5	96	74
	3-keto-carbofuran	0.5	102	72
	3-hydroxy-carbofuran	0.5	90	69

¹The three analytes were combined in fortified samples

Storage stability studies were also conducted with the processed fractions of maize (Schreier, 1990a) and sugar cane. No loss of carbofuran or 3-hydroxy-carbofuran occurred during more than 2 years of frozen storage.

Definition of the residue

MRLs currently refer to the sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran; 3-keto-carbofuran, the 7-phenol, the 3-keto-7-phenol and 3-hydroxy-7-phenol are excluded. Studies of plant and animal metabolism displayed similar metabolic pathways. In ruminants and poultry the parent carbofuran constitutes less than <1% of the residue. The major carbamate metabolite is 3-

hydroxy-carbofuran, but it is only found in certain animal commodities, e.g. 12% of the TRR in egg yolk and 11% in ruminant kidneys. Carbofuran is found in some plant commodities, e.g. 14% of the TRR in maize forage, but 3-hydroxy-carbofuran is generally the predominant carbamate compound. The metabolite 3-keto-carbofuran is not usually detected and contributes little to the total carbamate residue.

Studies of plant metabolism have shown that conjugate(s) of 3-hydroxy-carbofuran can constitute an appreciable proportion of the total residue. For example in soya bean forage 11% of the TRR was free and 28% of the TRR was conjugated (acid-released) 3-hydroxy-carbofuran. In soya beans, 1.5% was free and 3.2% was conjugated 3-hydroxy-carbofuran (Table 3). As the conjugated form might be released after human ingestion, it must be considered as part of the defined residue.

The residue should be defined both for estimates of dietary intake and compliance with MRLs as carbofuran plus 3-hydroxy-carbofuran, free and conjugated, expressed as carbofuran.

USE PATTERN

Carbofuran is a systemic acaricide, nematicide and insecticide, applied to foliage at 0.25-1.0 kg ai/ha, to the furrow at planting at 0.5-4.0 kg/ha to control soil-dwelling and foliar-feeding insects, or broadcast at 6-10 kg/ha to control nematodes. Information on the use patterns on crops (labels and/or summary tables) provided by the sponsors and the governments of Australia and the UK are summarized in Table 30.

RESIDUES RESULTING FROM SUPERVISED TRIALS ARE SUMMARIZED IN TABLE

Data were supplied on at planting, foliar and directed applications of carbofuran to numerous crops, mainly in Australia, Europe, and North, Central and South America.

Residues judged to be from treatments according to GAP and used to estimate maximum residue levels are underlined. Those resulting from the maximum applications consistent with GAP and used to estimate STMRs are double underlined. All residues have been corrected for the average analytical recovery of the compound determined unless otherwise indicated.

Table 30. Summary of information on supervised trials (not necessarily according to GAP) provided by the sponsors.

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Alfalfa	USA	4 F	foliar	2 pts/A; 1.12	2 GPA - aircraft	1-2/cutting 1.1.2 total	28	
Apple	India	3 G		5 g/tree				
Banana	Brazil	350 SC	immersion of the horn type seedling	1.4 g/100 l		1		
Banana	Brazil	350 ST	Seed treatment	0.14 kg/100 L		1		
Banana	Brazil	5 G		3-80 g/hole		2		4 mo. retreatment

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Banana	Cyprus	10 G		1.5-3 g ai/mat			30	
Banana	Cyprus	10 G		1.5-3 g ai/mat			30	
Banana	Cyprus	5 G		1.5-3 g ai/mat			30	
Banana	Cyprus	75 WP		1.5-3 g ai/mat				
Banana	India	3 G		1.5 g/sucker				
Banana	Kenya	10 G		3 g ai/mat		2		
Banana	Philippines	10 G	at planting + 4 mo	1.25-1.5 g/hole; 5g		2		4 mo interval
Banana	Philippines	10 G	base of the plant	2.5- 3 g/mat		4		established plantations
Banana	Philippines	5 G	at planting	1.25-1.5 g/hole; 5 g/hole				at planting + 4months later
Banana	Philippines	5 G		3.0 g/mat		4	0	once every 4 months for established plantations
Banana	Spain	20 F		5.6 kg ai/ha			60	
Banana	Spain	5 G		0.6-0.75kgai/ha			60	
Banana	Iv. Coast	10 G		3 g ai/mat				
Banana	Iv. Coast	4 F		1 g ai/mat			21	
Banana	Iv. Coast	5 G		3 g ai/mat				
Banana	Kenya	3 G		100gai/stool				
Banana	Kenya	5 G		3 g ai/mat		2		
Banana	Malaysia	3 G		0.6 g/tree				apply at base of tree at a distance of 1 foot
Barley	India	3 G	broadcast at plant	1.25 kg/ha				
Bean	Brazil	350 SC	at seeding	0.7-1.05 kg/ha	100-300l/ha	1		
Bean	Brazil	5 G	At planting, incorporated	1-2 kg/ha				
Bean	Cz. Republic	5 G		750 g ai/ha				
Beans	Argentina	10 G	at seedling	0.5 kg/ha				
Beets	Cz. Republic	10 G		1-2 kg ai/ha			>28	
Beets	Cz. Republic	350 F		1.05 kg ai/ha				
Beets	Cz. Republic	5 G		.75-1.5 kgai/ha				
Beets	Poland	5 G		.75 kg ai/ha				
Beets/fodder	Cz. Republic	350 ST		1.4/2.6kgai/100				
Beets/sugar	Cz. Republic	350 ST		1.4/2.6kgai/100				

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Cabbage	Brazil	5 G	in furrow or filed hole	2 kg/ha		1		
Cabbage	Cz. Republic	10 G		2 kg ai/ha				
Cabbage	Cz. Republic	350 F		0.7 kg ai/ha				
Cabbage	Cz. Republic	350 F		0.0525 %				
Cabbage	India	3 G	at planting	1.5 kg/ha				
Canola	USA	10 CR		2.5lbs/A; 0.28				
Carrot	Cyprus	10 G		5-8 kg ai/ha			30	
Carrot	Cyprus	5 G		5-8 kg ai/ha			30	
Carrot	Cyprus	75 WP		5-8 kg ai/ha			30	
Coffee	Brazil	350 SC	gound application	0.35 g/hole				
Coffee	Brazil	5 G	at rains start and end of rains	0.5 - 3* g/hole		2		mechanically or manual application
Coffee	Brazil	5 G	incorporation in soil					* rate depends upon the the transplant age and pest infestation
Coffee	Kenya	10 G		2gai/tree x2				
Coffee	Kenya	5 G		4 g ai/tree				
Coffee	Malaysia	3 G		0.9 g /tree				apply around base of tree at one foot
Coffee	USA	10 G	incorporation in soil	1.5 g/tree		2		PR only. 1st application early winter (Jan/Feb); 2nd late June/July.
Corn (see Argentina maize also)	Argentina	10 G	in furrow, soil application	1-3.5 kg/ha				
Corn	Argentina	10 G						
Corn	Brazil	310 TS	seed treatment	697.5 g/100 kg seeds		1		
Corn	Brazil	350 SC	in furrow at seeding	1.05 -1.4 kg/ha	100-300l/ha	1		
Corn	Brazil	350 ST	Seed treatment	0.7-1.05 kg/100 kg seeds		1		
Corn	Brazil	5 G	At planting, incorporated	1.5-1.75 kg/ha		1		
Corn	Korea	3 G		3 TO 5		1	45	
Corn	USA	10 G	Post planting, band over row, incorporated	10 lbs/A; 1.1		1		row spacing = 40 inch
Corn	USA	10G	Foliar	10 lbs/A; 1.1		2		aerial application
Corn	USA	4 F	at planting	1 qt/A; 1.1		1	30 (forage)	row spacing=40 inch
Corn	USA	4 F	postplant	1 qt/A; 1.1			30 (forage)	no foliar application if soil application with granule formulation 10lbs/A-10 G; 6.7 lbs/A-15 G

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Corn	USA	4 F	foliar	1/2-2 pints/A; 0.28-1.1		2	30 (forage)	1 only if soil application was made.
Cotton	Brazil	350 SC	furrow application at seeding	0.700-1.05 kg/ha	100-300l/ha	1		
Cotton	Brazil	350 ST	Seed treatment	0.7 kg/100 kg seeds		1		
Cotton	Brazil	5 G	in furrow at planting	1.5-3 kg/ha		1		rate depends upon pest type and infestation
Cotton	Bulgaria	10 G		5 kg ai/ha				
Cotton	Bulgaria	350 F		3 kg/100 kg				
Cotton	Bulgaria	350 ST		1.5 kg ai/100kg				
Cotton	China	3 G	in furrow, incorporated	0.675-0.9 kg/ha			60	
Cotton	India	3 G	at planting	1.0 kg/ha				
Cotton	Malaysia	3G	in hole at planting	0.03 g/hole				
Cotton	Spain	5 G		0.6-0.75kgai/ha			60	
Cotton	USA	10 G	in furrow at planting	10 lbs/A; 1.1		1		row spacing = 40 inch
Cotton	USA	4 F	in seed furrow, at planting	1 qt/A; 1.1		1		row spacing = 40 inch
Cranberries	USA (WA)	15 G	soil with rotary spreader	20 lbs/A; 3.4		1	60	incorporate with water sprinkler; do not use with flooding
Cranberries	USA (WA)	10 G	soil with rotary spreader	20 lbs/A; 2.2		2	60	incorporate with water sprinkler; do not use with flooding
Cucurbits	USA	10 G	soil incorporate	20 lbs/A; 2.2				row spacing = 60 inch
Cucurbit	USA	15 G	band application, incorporated	2.245 lbs /1000 linear feet				cucurbit=cucumber, melon, squash and pumpkins; Federal label
Cucurbit	USA	4 F	in furrow	2.4-3.8 oz/1000 row				
Cucurbit	Cyprus	10 G		1-1.5 kg ai/ha			30	
Cucurbit	Cyprus	5 G		1-1.5 kg ai/ha			30	
Cucurbit	Cyprus	75 WP		1-1.5 kg ai/ha			30	
Grape	USA	4F	broadcast to soil, incorporate	2.5gal/A, 11.2.; or 1.5gal/A chemigation, 6.72		1	##	
Grape	USA	4F	drip irrigation	075gal/A; 3.4		1	60	prohibited after May 1. Limited to 2.2 if a postharvest application was made in previous yr

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Grape	USA	10 G	over soil between vines, incorporate	100 lbs/A; 11.2		1	##	
Maize	Bulgaria	350 ST		875 g ai/100 kg				
Maize	Cyprus	10 G		5-8 kg ai/ha			30	
Maize	Cyprus	10 G		5-8 kg ai/ha			30	
Maize	Cyprus	10 G		5-8 kg ai/ha			30	
Maize	Cyprus	5 G		5-8 kg ai/ha			30	
Maize	Cyprus	5 G		5-8 kg ai/ha			30	
Maize	Cyprus	75 WP		5-8 kg ai/ha			30	
Maize	India	3 G	at plant	1.0 kg/ha				
Maize	Makedonia	5 G		1-1.5 kg ai/ha			-	
Maize	Pakistan	3 G	at sowing, in furrow; at whorl	0.24 kg/A		2		irrigate immediately after application
Maize	Poland	5 G		.75 kg ai/ha				
Maize	Poland	5 G		.75 kg ai/ha				
Maize	Former Youg.	350 F		1.4-2.1 kg/ai			-	
Oats/barley	Germany	300 SK	at sowing		4.5		##	
Oats/barley	Argentina	35 TS	seed treatment	0.3 kg/100 seeds	kg 134.4/l			diluted
Pea	India	3 G	at planting	1.0 kg/ha				
Peanut	Korea	3 G			5	1	55	
Peanuts	Brazil	350 SC	furrow application at planting	1.4-1.75 kg/ha	100-300l/ha			
Peanuts	Brazil	5 G	at planting, incorporation in soil	2 kg/ha		1		
Peanuts	China	3 G	furrow application	1.35-2.35 kg/ha			60	
Peppers	India	3 G	at planting	0.5 kg/ha				
Peppers	USA	4 F		3 qt/A; 3.4		2	21	first application at-plant. Arizona only.
Peppers	USA	10 G	side dressing	20 lbs/A, 30; 2.2, 3.4		2	21	
Potato	Argentina	10 G	in furrow	1.5-2.5 kg/ha				appl. at seeding or after planting
Potato	Brazil	350 SC	in furrow	3.5 kg/ha	200l/ha			
Potato	Brazil	5 G	At planting, incorporated	1.5-4 kg/ha		1		
Potato	Cyprus	10 G		4-8 kg ai/ha			30	
Potato	Cyprus	5 G		4-8 kg ai/ha			30	

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Potato	Cyprus	75 WP		4-8 kg ai/ha			30	
Potato	Cyprus	75 WP		4-8 kg ai/ha			30	
Potato	Cz. Republic	350 F		240-350 g ai/ha			30	
Potato	Egypt	10 G		3.25 kg ai/ha			14	
Potato	India	3 G	at planting	2.6 kg/ha				
Potato	Korea	3 G		5 kg/10 A		1	45	
Potato	Makedonia	5 G		1-1.5 kg ai/ha			-	
Potato	Poland	5 G		2 kg ai/ha				
Potato	USA	4 F	Foliar	2 pts/A; 1.1	10 gal/A ground; 3 gal/A aerial	8	14	3 lbs ai/A/season if at plant application made
Potato	USA	4F	at-planting, in furrow	3 qt/A; 3.4		1		0.225lb/1000 feet of row
Potato	USA	4 F		1-2 pints/A	3 GPA aircraft	8/season		
Rice	USA	3 G	air/ground equipment	20 lb/A; 0.67		1/season	60	1 d before or up to 21 d after permanent flooding. State label Expires 9/97
Rice	USA	5 G	air/ground equipment	10 lbs/A; 0.56		1/season		1 d before or 2 or 21 days after flooding. Expires 9/97.
Rice	USA	5G or 2G	preplant, soil incorporated. ground equip	0.56		1		CA only. Expires 8/97
Rice	USA	5G	postplant to soil. ground or aerial	0.56		1	60	CA only
Rice	Argentina	10 G	broadcast	0.75-1 kg/ha				
Rice	Australia	10 G		1 kg/ha		2/season		application at mid-tillerinf and 30-50 d after panicle initiation
Rice	Brazil	310 TS	seed treatment	527 g/100 kg seeds				
Rice	Brazil	350 SC	furrow application	0.700-1.05 kg/ha	400 ml/ha	1		vs irrigation system
Rice	Brazil	350 ST	Seed treatment	0.525 kg/100 kg seeds		1		
Rice	Brazil	350 ST						
Rice	Brazil	5 G		0.75-1 kg/ha		1		irrigated
Rice	China	3 G	broadcast, at seeding, incorporated	0.9-1.35 kg/ha			60	
Rice	India	3 G	at plant	2.0 kg/ha				
Rice	Japan	3G	broadcast	0.9		3	50	
Rice	Korea	3 G		0.09 - 0.12/10a (?)		2	7	2 applications (control 1st and 2nd generation)
Rice	Pakistan	3 G	nursery	0.3 g/m2				2nd application : 5 d before tansplanting
Rice	Pakistan	3 G		240 g/A		2		1st application 25 - 30 days after transplant. 2nd application 50 - 65 days after transplant
Rice	Philippines	3 G	seedbed	90 g/ha			28	
Rice	Thailand	3 G	seedbed	1.9 kg/ha				10 days after seeding
Rye grass	Argentina	35 TS	seed treatment	1.995 kg/100 kg seeds				undiluted

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Rye/wheat	Argentina	35 TS	seed treatment	497 kg/100 seeds	39 g/l			diluted
Small grains	USA	4 F	foliar, before head emerges	1/2 pt/A; 0.28		2/season		do not feed forage
Sorghum	India	3 G	at planting	1.5 kg/ha				
Sorghum	Thailand	3 G	at-planting	1.5 kg/ha				
Sorghum	USA	15 G						
Sorghum	USA	10 G	at-planting	10 lbs/A; 1.1		1		row spacing = 40 inch
Sorghum	USA	4 F		1 pint	20 to 30 GBA	2		State label
Soya bean	Argentina	10 G	in furrow at seedong	1.5 kg/ha				
Soya bean	India	3 G	at planting	2.0 kg/ha				
Soya bean	Philippines	3 G		16.7-33.4 product/ha	kg			
Soya bean	USA	10 G	at planting, incorporated	20 lbs/A; 2.2		1		row spacing = 40 inch
Soya beans	USA	4 F	at planting	3-4 pints/A; 1.7-2.2				row spacing=40 inch
Soya beans	USA	4 F	foliar appl.	0.5pint; 0.28	1 1/2 gal/A aerial; 20 gal /A ground	2/season	21	no foliar appl if treatment at planting
Strawberry	USA	4F	postharvest soil band. Ground equipment		2.2	1		limited to OR, MI, MN, MO, TN, WA, CT, NH, OH, VA, VT. May not be used after Sept. 1 or Oct. 1,
Sugar cane	Australia	10 G	band application, incorporated	3 kg/ha				application 3-5 leaf stage
Sugar cane	Brazil	350 SC	in furrow along planting stick or in bands or streaks in the cane furrow	1.4-1.75 kg/ha	100-300l/ha			
Sugar cane	Brazil	350 SC						
Sugar cane	Brazil	5 G	application around the plant/ in bands or streaks	1.5-3 kg/ha		1		aplication at cane second harvest
Sugar cane	China	3 G	at planting, band application	1.35-2.25 kg/ha			60	
Sugar cane	Pakistan	3 G	at planting/ 30 d. after planting	0.3-0.45 kg/A				
Sugar cane	Philippines	3G	at planting/30 d afr planting	0.3-0.45 kg/A				2 nd application at earthing
Sugar cane	Philippines	5 G	at planting, in furrow and furrow ridge	1-2 kg/ha				
Sugar cane	Thailand	3 G	at planting, in the rows	1.9 kg.ha				

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Sugar cane	USA	4 F	after joint formation, foliar	1.5 pts/A; 0.84		2	17	do not use in Hawaii
Sugar cane	USA	10 G	over stubble cane, incorporated	40 lbs/A; 4.5		1		row spacing = 60 inch. Do NOT use in Hawaii
Sugar beet	Bulgaria	350 ST		875 g ai/100 kg				
Sugar beet	Hungary	10 G		1.5-2 kgaiha				
Sugar beet	Poland	350 ST		44.8g/100,000 s				
Sugar beet	Poland	480 FS		58 LITRE/TON				
Sugar beet	Russia	350 ST		8-10 kg ai/ton			-	
Sugar beet	Former Youg.	350 F		1.4-2.1kgaih			-	
Sugar beet	USA	10 G	at planting, incorporated	20 lbs/A; 2.2				row spacing = 22 inch
Sugar beets	USA	4 F		1 to 2 quarts/A				State label
Sunflower	Bulgaria	300 COMBI		1 kg ai/100 kg				
Sunflower	Bulgaria	350 ST		1.5 kg ai/100kg				
Sunflower	Hungary	10 G		1.5-2 kgaiha				
Sunflower	USA	4F	soil band or in-furrow at planting. ground equipment	1.4 qt/acre; 1.6		1		row spacing = 30 inch. 0.16 pt/1000 feet of row
Sunflower	USA	4F	foliar, ground or aerial	1 pint/A; 0.56	2 gal/A aerial; 10 gal/A ground	4	28	
Sweet corn	USA	4F	at planting, 7 inch band over the row or inject on each side of row with water	2 pt/A; 1.12			30	Forage may not be fed within 30 days of application. Use 2.5 fl oz/1000 linear feet of row.
Sweet corn	USA	4F	foliar	1 pt/A; 0.56	10 gallons/A ground; 2 gallons/A aerial	4	7	Limited to machine-harvested corn. 1st application just prior to silking, with 7 day repeat interval.
Tea	India	3 G	at planting	300 mg/plant				
Tobacco	Argentina	10 G	broadcast/in furrow application	3.5-6 kg/ha				
Tobacco	Australia	10 G	in furrow, incorporated	3.5 kg/ha				
Tobacco	Australia	10 G						
Tobacco	Brazil	350 SC	at planting	1.4-1.75 kg/ha	100-300l/ha	1		
Tobacco	Brazil	5 G	At planting/transplanting, incorporated	0.75-4 kg/ha		1		for granule manual or mechanical application
Tobacco	India	3 G	at planting	4 kg/ha				
Tobacco	Korea	3 G	planting	50 kg/10 a		1		

Label uses:	Country	Form, type	Application type	Application, kg ai/ha or /kg seed (all metric are g or kg ai)	Volume	Application no.	PHI, days	Remarks
Crop								
Tobacco	Pakistan	3 G	nursery, broadcast	0.3 kg/A				15 d before transplanting
Tobacco	Thailand	3 G	at-planting	0.06 g/hill				
Tobacco	USA	4F	pre-transplant, soil incorporated	1.5 gal/A;6.7		1		
Tobacco	USA	10 G	pre-transplant, incorporate	60 lbs/A; 6.7				
Tomato	Argentina	10 G	at transplanting, +1 mo transplanting	1.5 kg/ha		2		
Tomato	Argentina	10 G	in seedbed	3 kg/ha		2		at planting and 15 d before transplanting
Tomato	Brazil	350 SC	in furrow	1.75 kg/ha	200 l/ha	1		
Tomato	Brazil	5 G	at planting/transplanting	0.75-4 kg/ha		1		
Tomato	Brazil	5 G		0.15-0.25 g/ha				
Tomato	India	3 G	at planting	2.5 kg/ha				
Tomato	Malaysia	3 G	at planting, in hole, incorporated	0.09 g /hole		1		
Vege-tables	Cyprus	10 G		4-8 kg ai/ha			30	
Vege-tables	Cyprus	5 G		4-8 kg ai/ha			30	
Vege-tables	Cyprus	75 WP		4-8 kg ai/ha			30	
Vege-tables	Former Youg.	350 F		0.7 kg ai/ha				
Vege-tables	Former Youg.	350 F		0.0525 %				
Wheat	Brazil	350 SC	in furrow at seedling	1.05 -1.4 kg/ha	100-300l/ha			
Wheat	Brazil	5 G	At planting, incorporation in soil	0.75 - 1 kg/ha		1		
Wheat	India	3 G	at planting	3.0 kg/ha				
Wheat	USA	4 F	see small grain					
Wheat/ barley	Australia	360 F	in furrow, at seeding	1.1l/ha	10l/ha			

In the Tables of field trials that follow, a uniform procedure was used to treat residues of the two analytes 3-hydroxy-carbofuran and carbofuran limits at the determination and detection, which may be summarized as follows.

V ₁	V ₂	Total
<LOd	<LOd	LOd

LOd<V ₁ <LOQ	LOd<V ₂ <LOQ	V ₁ V ₂
<LOd	>LOD	V ₂
<LOd	LOd<V ₂ <LOD	V ₂

V₁, V₂: carbofuran or 3-hydroxy-carbofuran

LOd: limit of detection

LOD: limit of determination

Residues below the limit of determination and above the limit of detection are in parentheses.

Furadan 4F was applied as a foliar spray to alfalfa in 28 supervised field trials in the USA (Singer, 1990a). Three applications were made at 1.12 kg ai/ha and at 0.004-0.006 kg/l. Each application was followed by cutting after 28 days. Both green and field-dried samples of alfalfa were analysed for carbofuran and carbamate and phenol metabolites by the method of Schreier. A limit of determination of 0.5 mg/kg was demonstrated for carbofuran, 3-hydroxy-carbofuran, 3-ket°Carbofuran, 7-phenol, 3-hydroxy-7-phenol and 3-keto-7-phenol. The limit of detection was estimated to be 0.1 mg/kg. The results are shown in Table 31. The water contents of the dried alfalfa samples were not determined.

The US GAP conditions are 2 applications, total 1.1 kg ai/ha, with the second application not exceeding 0.56 kg ai/ha. The PHIs are 28 days after a 1.1 kg ai/ha application, 14 days after 0.56 kg ai/ha, and 7 days after 0.28 kg ai/ha.

Table 31. Residues of carbofuran and its metabolites in or on green and dried alfalfa cut 28 days after foliar applications of carbofuran at 1.12 kg ai/ha in the USA.

Location, Year	No. appl ¹	Sample	Carbofuran	Residues, mg/kg						
				3-OH-CF	3-K-CF	Total carb- amates	7- Phenol	3-K-7-P	3-OH-7- P	Total phenols
California, 1986	1	green forage	(0.35)	1.3	<0.1	<u>1.7</u>	(0.17)	0.54	(0.25)	0.96
				Residues, mg/kg						
	1	dried fodder	1.4	6.2	(0.15)	<u>7.6</u>	0.60	1.3	0.90	2.8
	2	green	(0.17)	1.4	<0.1	<u>1.6</u>	(0.15)	0.50	(0.20)	0.85
	2	dried	(0.38)	4.3	<0.1	<u>4.7</u>	0.45	1.2	0.76	2.4
	3	green	(0.10)	1.2	<0.1	<u>1.3</u>	(0.19)	0.68	0.46	1.3
	3	dried	(0.24)	2.8	<0.1	<u>3.0</u>	(0.30)	0.65	0.58	1.5
Pennsylvania, 1987	1	green	<0.1	<0.1	<0.1	<u><0.1</u>	<0.1	(0.13)	<0.1	(0.13)
	1	dried	<0.1	1.2	<0.1	<u>1.2</u>	<0.1	(0.25)	(0.19)	(0.44)
	2	green	<0.1	0.52	<0.1	<u>0.52</u>	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	0.90	<0.1	<u>0.90</u>	<0.1	(0.42)	(0.24)	0.66
	3	green	<0.1	1.2	<0.1	<u>1.2</u>	<0.1	(0.34)	(0.25)	0.59
	3	dried	<0.1	1.4	(0.12)	<u>1.4</u>	<0.1	0.99	0.58	1.6
Ohio, 1987	1	green	<0.1	1.6	<0.1	<u>1.6</u>	(0.14)	0.52	(0.32)	0.98
	1	dried	<0.1	4.5	<0.1	<u>4.5</u>	(0.41)	1.1	1.2	2.7
	2	green	<0.1	<0.1	<0.1	<u><0.1</u>	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	(0.32)	<0.1	<u>(0.32)</u>	<0.1	(0.22)	<0.1	(0.22)
	3	green	<0.5	(0.13)	<0.5	<u>(0.13)</u>	<0.1	<0.1	<0.1	(0.13)
	3	dried	<0.1	(0.28)	<0.1	<u>(0.28)</u>	<0.1	<0.1	<0.1	<0.5
California, 1988	1	green	<0.1	1.8	<0.1	<u>1.8</u>				
	1	dried	(0.41)	2.4	(0.34)	<u>2.8</u>	0.65	1.9	1.2	3.8
	2	green	<0.1	0.92	<0.1	<u>0.92</u>	<0.1	0.55	0.32	0.87
	2	dried	(0.37)	3.0	(0.23)	<u>3.4</u>	0.55	2.0	1.8	4.4
	3	green	(0.34)	1.9	<0.1	<u>2.2</u>	(0.28)	0.62	0.52	1.4
	3	dried	(0.40)	2.2	<0.1	<u>2.6</u>	0.64	1.2	1.3	3.1
Pennsylvania, 1987	1	green	<0.1	1.4	<0.1	<u>1.4</u>	(0.12)	0.50	0.62	1.2

carbofuran

Location, Year	No. appl ¹	Sample	Carbofuran	Residues, mg/kg						
				3-OH-CF	3-K-CF	Total carb-amates	7-Phenol	3-K-7-P	3-OH-7-P	Total phenols
California, 1986	1	green forage	(0.35)	1.3	<0.1	<u>1.7</u>	(0.17)	0.54	(0.25)	0.96
	1	dried	<0.1	5.2	<0.1	<u>5.2</u>	0.55	1.4	0.56	2.5
	2	green	<0.1	1.2	<0.1	<u>1.2</u>	<0.1	(0.46)	<0.1	(0.46)
	2	dried	<0.1	3.8	<0.1	<u>3.8</u>	0.36	1.0	0.72	2.1
	3	green	<0.1	4.3	0.90	<u>4.3</u>	<0.1	1.4	(0.30)	1.7
	3	dried	<0.1	4.2	<0.1	<u>4.2</u>				
Wisconsin, 1986	1	green	<0.1	(0.29)	<0.1	<u>(0.29)</u>	<0.1	<0.1	<0.29	<0.5
	1	dried	<0.1	0.90	<0.1	<u>0.90</u>	(0.17)	(0.30)	(0.26)	0.73
	2	green	<0.1	1.2	<0.1	<u>1.2</u>	<0.1	0.13	(0.32)	(0.45)
	2	dried	<0.1	4.6	(0.28)	<u>4.6</u>	(0.45)	0.96	1.6	3.0
	3	green					<0.1	<0.1	<0.1	<0.1
	3	dried					<0.1	0.59	(0.27)	0.86
Minnesota, 1985	1	green	<0.1	(0.34)	<0.1	<u>(0.34)</u>	<0.1	<0.1	<0.1	<0.1
	1	dried	<0.1	0.64	<0.1	<u>0.64</u>	<0.1	(0.22)	(0.16)	(0.38)
	2	green	<0.1	<0.1	<0.1	<u><0.1</u>	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	<0.1	<0.1	<u><0.1</u>	<0.1	<0.1	<0.1	<0.1
	3	green					<0.1	<0.1	<0.1	<0.1
	3	dried					<0.1	(0.21)	<0.1	(0.21)
Iowa, 1984	1	green	<0.1	(0.38)	<0.1	<u>(0.38)</u>	<0.1	<0.1	<0.1	<0.1
	1	dried	<0.1	0.74	<0.1	<u>0.74</u>	<0.1	(0.23)	<0.1	(0.23)
	2	green	<0.1	<0.1	<0.1	<u><0.1</u>	<0.1	<0.1	<0.1	<0.1
	2	dried	<0.1	<0.1	<0.1	<u><0.1</u>	<0.1	<0.1	<0.1	<0.1
	3	green	<0.1	<0.1	<0.1	<u><0.1</u>				
	3	dried	(0.30)	0.57	<0.1	<u>0.87</u>	<0.1	(0.18)	<0.1	0.18
Nebraska, 1986	1	green	<0.1	0.94	<0.1	<u>0.94</u>	<0.1	(0.36)	(0.37)	0.74
	1	dried	<0.1	1.6	<0.1	<u>1.6</u>	(0.38)	0.90	(0.41)	1.7
	2	green	<0.1	(0.30)	<0.1	<u>(0.30)</u>	<0.1	<0.1	<0.1	<0.1

Location, Year	No. appl ¹	Sample	Carbofuran	Residues, mg/kg						
				3-OH-CF	3-K-CF	Total carb-amates	7-Phenol	3-K-7-P	3-OH-7-P	Total phenols
California, 1986	1	green forage	(0.35)	1.3	<0.1	<u>1.7</u>	(0.17)	0.54	(0.25)	0.96
	2	dried	<0.1	1.2	(0.28)	<u>1.2</u>	(0.28)	(0.48)	(0.32)	1.1

Abbreviated compound names: see Figure 1

Limits of detection, determination 0.1 , 0.5 mg/kg for each analyte

¹The alfalfa was cut 28 days after each application and before the subsequent application

In a separate trial in the USA (Leppert, 1986a), alfalfa in California was treated twice at rates of 0.56 and 0.28 kg ai/acre with Furadan 4F applied as an aerial foliar spray. The PHI was 4 days. Samples of green hay, field-dried hay, meal and finished meal pellets were prepared and analysed by the method of Schreier. Limits of determination were established by determination of recoveries from fortified control samples. The recoveries listed in Table 32 were reported and some supporting chromatograms were included. The trial did not comply with GAP because of the 4-day PHI.

Table 32. Recovery of carbofuran and metabolites from alfalfa by the Schreier Method.

Sample	Spike, mg/kg	Recovery, %					
		Carbofuran	3-OH-CF	3-K-CF	7-Phenol	3-OH-7-P	3-K-7-P
Green hay	0.05			104			
	0.5			69	98	94	102
	2.5	87	105				
Cured hay	0.5			104			
	1.0				71	72	88
	2.5	78	93	109			
Meal	0.2	95	110	90			
	1.0				64	79	83
Pellet	0.2			85			
	1.0				82	87	110
	5.0	85	94	77			

Abbreviated compound names: see Figure 1

Table 33. Residues of carbofuran and metabolites in or on alfalfa harvested 4 days after two applications of Furadan 4F (0.56 and 0.28 kg ai/ha).

Sample	Residue, mg/kg					
	Carbofuran	3-OH-CF	3-K-CF	7-Phenol	3-OH-7-P	3-K-7-P
Green hay	3.6	2.7	0.26	0.31	0.22	0.77
	4.3	4.9	0.63	0.46	0.36	1.4
	3.1	2.4	0.40	0.52	0.45	1.6
Cured hay ¹	19	9.0	0.54	2.2	1.3	1.3
	15	8.9	0.29	1.8	1.2	1.6
	19	8.5	0.62	2.4	2.0	2.2
Meal	18	5.2	0.12	2.9	1.7	1.7
	16	4.5	<0.20	3.8	2.0	1.7
Pellets	14	4.8	0.21	2.8	1.3	1.4
	16	4.8	0.20	2.6	1.3	1.3

Abbreviated compound names: see Figure 1

¹Moisture content not reported.

Maize. Field trial results were reported from Brazil (Sao Paulo University, 1994), France (Mollhoff, 1974), Germany (Mollhoff, 1974) and the USA (Brooks, 1995; Singer, 1990b). The trials represent various combinations of at planting plus foliar treatments and the findings are shown in Table 34. The reports from Brazil, France and Germany consisted of brief summaries and provided inadequate details. They were not suitable for use in estimating maximum residue levels.

The US trials were of two types: an in-furrow application of a 15G formulation at 1.5 kg ai/ha followed by a foliar whorl application of 15G at 1.1 kg ai/ha, and one or two foliar applications of a 4F formulation at 1.1 kg ai/ha. The GAP label conditions specify a soil band, in-furrow, or injection at planting of the F (not G) formulation at 1.12 kg ai/ha (0.090 kg ai/2.54 m row) with a PHI of 30 days for feeding forage, which may be followed by a soil band, side-dress, or basal spray of the F formulation at 1.12 kg ai/ha (2.24 kg ai/ha in South Carolina) with a 30-day restriction on feeding forage, but no other PHI and no limit to the number of treatments. Additionally, two foliar applications may be made at 1.12 kg ai/ha each, with a 30-day PHI, using ground or aerial equipment. The US trials exceed the initial GAP at planting rate by 34% and use an F formulation, which place the trials on the fringe of acceptability. The use of an F or G formulation appears to have no effect on the residue concentrations (see the trials on sweet corn). Only two trials (in Missouri) are within the GAP window.

The other (5) US trials consisted of foliar spray applications of an F formulation at 1.1 kg ai/ha, with PHIs of 102-145 days, far in excess of the 30-day GAP interval. The data could not be evaluated.

Table 34. Total residues of carbamates and phenols in or on maize treated with carbofuran.

Country, Year	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis
		Method/timing	kg ai/ha		Carbamates ¹	Phenols ²	
Brazil 1994	Furadan 350 SC	foliar spray	1.4	30	<0.05 (grain)		Leppert
			2.8	30	<0.05		
Brazil 1993	Furadan 350 TS	seed treatment	1.05 kg ai/100 kg	159	<0.1 (grain)		Leppert
			2.1 kg ai/100 kg	159	<0.1 (grain)		
Brazil 1993	Furadan 50G	in-furrow at-plant	1.75	30 ³	<0.1		Leppert
			3.5	30 ³	<0.1		
France 1973	Curraterr 5G	in-furrow at sowing	0.60	122	<0.1 (cob with grain)		Mollhoff
				163	<0.1 (grain)		
				115	<0.1 (grain)		
Germany 1973	Curraterr 5G	in-furrow at sowing	1.0	115	<0.1 (grain)		Mollhoff
Germany 1976	Curraterr 5G	in-furrow at sowing	0.94	125	0.7, 0.6, 0.8 (silage)		Mollhoff
				153	<0.1, <0.1, <0.1 (grain)	0.4, 0.3, 0.9 (fodder)	
Germany 1975	Curraterr 5G	in-furrow at sowing	0.50	123	0.1, 0.3, 0.2 (silage)		Mollhoff
				143	<0.1, <0.1, <0.1 (grain)		
Germany 1985	Curraterr 5G	in-furrow at sowing	0.62	105	<0.1 (cob at milk to dough)		Mollhoff

Country, Year	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis
		Method/ timing	kg ai/ha		Carbamates ¹	Phenols ²	
					stage)		
Germany 1974	Curraterr 5G	in-furrow at sowing	1.0	138	<0.1 (cob) <0.1 (grain)		Mollhoff
USA (OH) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	69	1.5, <0.1 ⁴ (fodder)	0.62, <0.5 ⁴ (fodder)	Schreier
USA (NC) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	2 x 1.1	65	<0.1 (fodder)	0.70, 0.67 (fodder)	
USA (MO) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	32	<u>1.1</u> , <u>1.0</u> (silage)		
	Furadan 4F	foliar spray	1.1	21	< <u>1.0</u> , <u>1.2</u> (silage)		
USA (MN) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	80	1.5 (silage)	<1.0 (silage)	
	Furadan 4F	foliar spray	1.1	55	1.3 (silage)	<1.0 (silage)	
USA (CA) 1988	Furadan 15G	in-furrow at planting	1.5				
	Furadan 15G	foliar whorl	1.1				
	Furadan 4F	foliar spray	1.1	63	2.0, 2.2 (fodder)	0.80, 0.66 (fodder)	
	Furadan 4F	foliar spray	1.1	63	2.4, 3.1 (fodder)	0.80, 1.4 (fodder)	
USA (IA) 1994	Furadan 4F	foliar spray	1.1	120	<0.03 (grain) <0.1 (fodder)	<0.03 (grain) <0.50 (fodder)	Schreier
USA (IL) 1994	Furadan 4F	foliar spray	1.1	111	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	
USA (NE)	Furadan 4F	foliar spray	1.1	102	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	
USA (MN) 1994	Furadan 4F	foliar spray	1.1	143	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.10 (fodder)	
USA (IN)	Furadan 4F	foliar spray	1.1	125	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	
USA (OH) 1994	Furadan 4F	foliar spray	1.1	124	<0.03 (grain) <0.10 (fodder)	<0.03 (grain) <0.50 (fodder)	

¹Carbofuran + 3-keto-carbofuran + 3-hydroxy-carbofuran

²7-phenol + 3-hydroxy-7-phenol

³30-day period from seed planting to mature crop

⁴Duplicate samples from same plot

Sweet corn (corn-on-the-cob). Field trials in the USA were reported by Martin (1986b, 1987). Thailand reported the GAP conditions used in field trials, but not the results (Thai Industrial Standards Institute, 1997). In 16 side-by-side trials in eight states of the USA, sweet corn was treated at planting with either Furadan granular (15G, 10 G in California) or Furadan flowable, at 3.4 kg ai/ha. Whorl applications were made with 15G (10 G in California) at 1.1 kg ai/ha 3-6 weeks after planting. Four additional foliar applications of the flowable formulation were made at 0.56 kg ai/ha

over a period of 2 to 7 weeks. The total seasonal application was 6.7 kg ai/ha. Ears and husks were harvested 0 and 7 days, and stalk (forage) samples 21 days, after the final treatment. The samples were analysed by the method of Schreier. Carbamates were measured by GC/NPD and phenols were measured by GC-MSD. Limits of determination of 0.5 and 0.03 mg/kg were demonstrated for each analyte in forage and corn ears respectively, with corresponding limits of detection of 0.01 mg/kg. The recoveries reported from stalks at a 0.5 mg/kg fortification (n = 13) were carbofuran 84 ± 13%, 3-keto-carbofuran 100 ± 18%, 3-hydroxy-carbofuran 87 ± 20%, 7-phenol 78 ± 14%, the 3-keto-7-phenol 80 ± 17%, 3-hydroxy-7-phenol 82 ± 19%. The recoveries from corn-on-the-cob at 0.03 mg/kg (n = 4 for carbamates, 5 for phenols) were carbofuran 79 ± 10%, 3-keto-carbofuran 84 ± 22%, 3-hydroxy-carbofuran 85 ± 13%, 7-phenol 77 ± 16%, the 3-keto-7-phenol 94 ± 7%, and 3-hydroxy-7-phenol 86 ± 14%. The limit of determination demonstrated by the analysis of fortified control husk samples was 0.5 mg/kg (n = 7 for carbamates, 6 for phenols). Recoveries were carbofuran 82 ± 20%, 3-keto-carbofuran 91 ± 12%, 3-hydroxy-carbofuran 86 ± 16%, 7-phenol 74 ± 5%, 3-keto-7-phenol 102 ± 17%, and 3-hydroxy-7-phenol 84 ± 13%.

The total carbamate residues in corn-on-the-cob 0 and 7 days after the final application were <0.03 (6), 0.03 (4), 0.04 (4), 0.05 and 0.08 mg/kg. The total phenol residues were ≤0.10 mg/kg. No single carbamate or phenol predominated and there were no differences between the residues from the two treatment programmes.

The residues found in the stalks (forage) and husks are shown in Table 35. Again there were no differences between the residues from the flowable and granular formulations. Sweet corn forage is not an animal feed item, although maize forage is.

US GAP for sweet corn permits application at planting of 1.12 kg ai/ha (90 g/2.54 m of row). Forage may not be fed within 30 days of treatment. Additionally, the F formulation may be applied at 0.56 kg/ai ha, maximum 4 applications per season, with a 7-day PHI. These treatments may not be made if the at planting application exceeded 1.12 kg ai/ha. There is a 21-day restriction on harvesting or feeding stalks.

All trials exceeded the maximum at planting application rate by 240%. The data were not acceptable for the estimation of maximum residue levels.

Thailand reported trials according to GAP, but without data on residues (Thai Industrial Standards Institute, 1977).

Table 35. Residues in or on sweet corn stalks and husks from the application of carbofuran, at planting and foliar, 6.7 kg ai/ha total rate, USA, 1985.

Location	PHI, days	Residue, mg/kg					
		Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Harvest stalks (forage)							
3.4 kg ai/ha 15G at planting, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	8.7	<0.1	(0.3)	0.64	(0.26)	(0.24)
	7	(0.20)	<0.1	(0.39)	(0.20)	(0.24)	(0.29)
	14	<0.1	<0.1	(0.20)	(0.29)	(0.32)	(0.38)
	21	<0.1	<0.1	(0.28)	0.50	<0.1 (0.44)	0.94
California	21	<0.1	<0.1	<0.1	0.70	<0.1	<0.1
Florida	21	<0.1	<0.1	(0.028)	<0.1	<0.1	<0.1
Iowa	21	1.2	<0.1	1.3	1.6	1.3	1.2
Illinois	0	13	<0.1	1.2	1.5	0.74	0.90
	7	<0.1	<0.1	1.5	0.46	1.0	1.2
	14	<0.1	<0.1	1.2	(0.17)	(0.22)	0.50

Location	PHI, days	Residue, mg/kg					
		Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
	21	<0.1	<0.1	1.2	<0.1	<0.1	<0.1
New York	0	4.7	<0.1	0.90	0.62	(0.26)	(0.40)
	7	3.4	<0.1	1.3	0.60	(0.30)	0.52
	14	(0.38)	<0.1	0.52	(0.20)	(0.150)	(0.25)
	21	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Oregon	21	<0.1	<0.1	0.88	(0.30)	0.50	0.56
Wisconsin	0	25 (5.2; 45)	<0.1	1.8	1.8	<0.1	<0.1
	7	0.56	<0.1	0.50	(0.20)	(0.20)	(0.21)
	14	<0.1	<0.1	0.98	(0.24)	(0.28)	(0.31)
	21	<0.1	<0.1	0.45	<0.1	(0.16)	(0.18)
4 kg ai/ha 4F at 3.plant, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	8.3	<0.1	0.62	0.82	0.62	0.63
	7	(0.25)	<0.1	0.60	0.54	0.88	0.76
	14	(0.16)	<0.1	0.86	0.52	0.68	1.0
	21	<0.1	<0.1	0.64	(0.38)	0.71	0.91
California	21	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Florida	21	<0.1	<0.1	0.54	<0.1	<0.1	(0.20)
Iowa	21	(0.48)	<0.1	1.7	1.9	0.93	1.2
Illinois	0	15	<0.1	1.6	1.1	(0.34)	0.52
	7	(0.15)	<0.5	1.4	(0.26)	(0.40)	0.63
	14	<0.1	<0.1	1.2	(0.29)	0.69	0.56
	21	<0.1	<0.1	1.2	<0.1 (0.32)	0.62	0.75
New York	0	4.1 (2.6; 5.7)	<0.1	0.92	0.68	<0.1	(0.32)
	7	1.5	<0.1	0.95	(0.28)	(0.20)	(0.30)
	14	(0.28)	<0.1	(0.32)	<0.1	<0.1	<0.1
	21	(0.15)	<0.1	(0.29)	<0.1	<0.1	<0.1
Oregon	21	<0.1	<0.1	0.67	(0.16)	(0.38)	0.63
Wisconsin	0	17	<0.1	(0.28)	0.90	<0.1	<0.1
	7	0.92	<0.1	0.64	(0.26)	(0.32)	0.59
	14	<0.1	<0.1	1.4	<0.1	<0.1	<0.1
	21	<0.1	<0.1	0.64	<0.1	(0.23)	<0.1
Sweet corn husks							
3.4 kg ai/ha 15G at planting, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	3.7	<0.5	<0.5	(0.27)		
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
California	7	0.94 (0.181; 1.71)	<0.1	<0.1	<0.1	<0.1	<0.1
Iowa	7	2.1	<0.1	<0.1	0.78	<0.1	<0.1
Illinois	0	0.83	<0.1	<0.1	(0.14)	<0.1	<0.1
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
New York	0	0.76	<0.1	<0.1	<0.1	<0.1	<0.1
	7	0.85	<0.1	(0.28)	<0.1	<0.1	<0.1
Oregon	7	(0.16)	<0.	(0.30)	<0.1	<0.1	(0.16)
Wisconsin	0	2.4	<0.1	(0.14)	(0.22)	<0.1	<0.1
	7	(0.18)	<0.1	<0.1	<0.1	<0.1	<0.1
3.4 kg ai/ha 4F at planting, 1.2 kg ai/ha 15G at whorl, 4 x 0.56 kg ai/ha 4F foliar							
Arkansas	0	1.4	<0.1	(0.0.20)	(0.19)	<0.1	<0.1
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
California	7	0.68	<0.1	<0.1	(0.24)	<0.1	<0.1
Iowa	7	4.9	<0.1	(0.46)	<0.1	<0.1	<0.1
Illinois	0	1.4	<0.1	<0.1	(0.18)	<0.1	<0.1
	7	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
New York	0	0.96	<0.1	<0.1	<0.1	<0.1	<0.1

Location	PHI, days	Residue, mg/kg					
		Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
	7	(0.37)	<0.1	(0.26)	(0.18)	<0.1	<0.1
Oregon	7	<0.1	<0.1	(0.18)	<0.1	<0.1	<0.1
Wisconsin	0	2.1	<0.1	(0.18)	(0.22)	<0.1	<0.1

Abbreviated compound names: see Figure 1

Oats. Carbofuran (Curraterr 300 SK) was applied at sowing to oats seeds at the rate of 4.5 kg/ha in 1975 in Germany. No residues were found in the grain at the claimed limit of detection (0.10 mg/kg) when the grain was harvested 112 days after treatment. The straw contained 1.4, 0.7 and 1.0 mg/kg total carbamates. Three varieties (Flamingskron, Luxor, Tiger) were planted in single plots. The method of Mollhoff was utilized to analyse the grain for carbofuran and 3-hydroxy-carbofuran. Only a summary of the studies was submitted, without adequate detail to validate the reported results. The results could not be used to estimate maximum residue levels, and three trials are insufficient, unless supported by data on other small grains.

Rice. Field trial reports on the use of carbofuran on rice were submitted from Australia, Brazil, Japan, the Philippines and the USA. Thailand submitted information on GAP field trials, but no report of the residues found (Thai Industrial Standards Institute, 1997). The results are shown in Table 36. The results from Brazil were reported as summaries with no detail and were not suitable for use in estimating maximum residue levels.

According to information supplied by the sponsors, GAP treatment in the Philippines is with 90 g/ha of a 3 G formulation, with a 28-day PHI. GAP in Brazil allows seed treatment with 350 ST at 0.525 kg/100 kg seed and at planting furrow application of 0.70-1.05 kg ai/ha by irrigation. Australian GAP specifies 2 x 1 kg ai/ha of a 10 G formulation, with the final application 30-50 days after panicle initiation. There is no PHI. US GAP specifies a pre-plant soil incorporation (before flooding) of a 2G or 5G formulation, at 0.56 kg ai/ha (California only). An additional 0.56 kg ai/ha may be applied after planting, with a 60-day PHI. Outside California a 3% G formulation may be applied after flooding to the water at 0.67 kg ai/ha, or the 5% G formulation may be applied at 0.56 kg ai/ha. A PHI is not specified. Most US uses are temporary. Japanese GAP was not available, but that for China specifies 1.35 kg ai/ha of a 3 G formulation broadcast at seeding, with a 60-day PHI.

The US trials were at twice the GAP application rate. The Japanese trials did not compare well with Chinese GAP: the single application rate was 67% of that specified and/or the PHI was 20-40 days (67%) longer. The Philippine trials were at 33 times the GAP application rate and the PHI was excessive, 49-62 days compared with the specified 28 days.

In one of the three Australian trials, the 2 x 1 kg ai/ha GAP treatment is approximated by a single application of 2 kg ai/ha, with a 58-day PHI.

One trial is insufficient for the estimation of a maximum residue level.

Table 36. Total residues of carbamates and phenolic metabolites from the application of carbofuran to rice.

f	Form./ Appln. method	Rate		PHI, days	Residue, mg/kg		Analytical method & LOD, mg/kg	Ref.
		kg ai/ha	kg ai/ha		Carbamates	Phenol		

f	Form./ Appln. method	Rate		PHI, days	Residue, mg/kg		Analytical method & LOD, mg/kg	Ref.
		kg ai/ha	kg ai/hl		Carbamates	Phenol		
Australia (Queensland), 1982/ Starbonnet	Furadan 10G/Broad- cast	2.0	-	58	<0.05 (grain) 0.58 (hulls)		Mollhoff 0.05	Stearns,1982
Australia (Queensland), 1982/ Starbonnet	Furadan 10G/Broad- cast	2 x 0.5	-	57	<0.02 (grain) <0.02 (hulls)		Mollhoff 0.05	Stearns,1982
Australia (Queensland), 1982/ Starbonnet	Furadan 10G/Broad- cast	1.0	-	95	<0.02 (grain) <0.02 (hulls)		Mollhoff 0.05	Stearns,1982
Brazil, 1994/ BR-IRGA 409	Furadan 50G/Broad- cast	1	-	30	<0.02 (grain)		Leppert 0.05 ¹	Sao Paolo U., 1994
Brazil, 1994/ BR-IRGA 409	Furadan 50G/Broad- cast	2	-	30	<0.02 (grain)		Leppert 0.05 ¹	
Brazil, 1993/ Araguaia	Furadan 350 SC/ pulverization in furrow at planting ²	1.05	Not spec.	156	<0.05 (grain)		Leppert 0.1 ¹	
Brazil, 1993/ Araguaia	Furadan 350 SC/ pulverization in furrow at planting ²	2.1	Not spec.	156	<0.05 (grain)		Leppert 0.1 ¹	
Japan, 1974/ Honenwase	Curraterr 3G/ Broadcast	0.9	-	101	<0.01 ³ (grain)		GLC (EC) (no detail) 0.01	Mollhoff,1974
		2 x 0.9	-	82	<0.01 ³ (grain) <0.02 ³ (straw)		GLC (EC) 0.01 0.02	Mollhoff,1974
		3 x 0.9	-	52	<0.01 ³ (grain) <0.02 ³ (straw)		GLC (EC) 0.01 0.02	Mollhoff, 1974
Japan, 1974/ Harebarc	Curraterr 3G/ Broadcast	0.9	-	110	<0.01 ¹ (grain) <0.02 ⁴ (straw)		GLC (EC) 0.01 0.02	Mollhof, 1974
		2 x 0.9	-	95	<0.01 (grain) <0.02 ⁴ (straw)		GLC (EC) 0.01 0.02	Mollhoff, 1974
		3 x 0.9	-	43	(0.01) ⁴ (grain) (0.04) ⁴ (straw)		GLC (EC) 0.01 0.02	Mollhoff, 1974
Philippines, 1971/ Miracle IR-8	Curraterr 3G/ Broadcast	0.99	-	62	<0.1 (grain)		Mollhoff 0.1 ¹	Mollhoff, 1972
		2 x 1.5	-	60	<0.1 (grain)		Mollhoff 0.1 ¹	Mollhoff, 1972
		2 x 1.5	-	49	<0.1 (grain)		Mollhoff 0.1 ¹	Mollhoff, 1972
		2 x 1.5	-	62	<0.1 (grain)		Mollhoff 0.1 ¹	Mollhoff, 1972
USA (Arkansas),	Furadan 4F/	1.1	0.012	117	<0.02	<0.02	Barros	Shevchuk,

f	Form./ Appln. method	Rate		PHI, days	Residue, mg/kg		Analytical method & LOD, mg/kg	Ref.
		kg ai/ha	kg ai/hl		Carbamates	Phenol		
1992/Katy	Broadcast spray, pre- flood				(grain) <0.02 (straw)	(grain) <0.02 (straw)	0.05	1993a
USA (California), 1992/M-201	Furadan 4F/ Broadcast spray, 5 days preplant	1.1	0.012	151	<0.02 (grain) <0.02 (straw)	<0.02 (grain) 0.20 (straw)	Barros 0.05	Shevchuk, 1993a
USA/ (California)/M-201	Furadan 4F/ Broadcast spray, 1 day preplant	1.1	0.006	128	<0.02 (grain) 0.07 (straw)	<0.02 (grain) 0.98 ⁵ (straw)	Barros 0.05	Shevchuk, 1993a
USA (Louisiana), 1992/ Lemont	Furadan 4F Broadcast spray, pre- flood	1.1	0.012	96	<0.02 (grain) 0.02 (straw)	0.13 (grain) 0.32	Barros 0.05	Shevchuk, 1993a
USA (Texas), 1992/ Gulfmont	Furadan 4F Broadcast spray, pre- flood.	1.1	0.008	72	<0.02 (grain) 0.06 (straw)	0.08 (grain) 0.30 ⁶ (straw)	Barros 0.05	Shevchuk, 1993a

¹No data were provided to validate the claimed limit of determination

²The method of application is not consistent with the formulation

³Limits of determination of 0.01 and 0.02 mg/kg for grain and straw respectively are claimed, but recovery is not reported below 0.2 mg/kg

⁴Limits of determination of 0.01 and 0.02 mg/kg for grain and straw respectively are claimed, but recovery is not reported below 0.05 mg/kg

⁵74% 7-phenol

⁶About 50% 7-phenol and 50% 3-hydroxy-7-phenol

Sorghum. Six trials were in the USA, where one application at planting was followed by two foliar applications (Shevchuk, 1994a). Adequate recoveries were demonstrated at 0.03 mg/kg from grain and at 0.1 mg/kg from all substrates. At a single location in India two varieties of sorghum seed were treated with carbofuran and in a separate trial the soil was treated after planting (Rallies, 1981). The results of the trials are shown in Table 37.

Information on GAP was not available for India or a neighbouring nation, so the data from India could not be evaluated. In the USA, GAP conditions include soil-band, in-furrow, or injection application at planting of the F formulation at 1.12 kg ai/ha (1.12 kg ai/3960 m row) in Arizona, Louisiana, Mississippi and Texas. An in-furrow application at 2.8 kg ai/ha may be made in Kansa, Missouri and Nebraska, when grazing or cutting for silage or forage within 75 days of planting is prohibited. Additionally, 2 applications of 0.56 kg ai/ha may be made as a post-emergence foliar directed spray before the head emerges from the boot. Grazing treated fields is prohibited (Louisiana), or there is a 30- or 75-day restriction (Kansa, Nebraska, Mississippi and Texas).

Table 37. Total residues of carbofuran and 3-hydroxy-carbofuran in or on sorghum.

Country, Year	Form.	Application		PHI, days	Residue, mg/kg ¹	Analytical method
		Method, timing	kg ai/ha			
India 1981	Furadan 40F	to seed, 4 days before planting	2.5%	45	0.14	Cook (colorime- tric) ³
			5.0% 10% (w/w)		0.18 0.27 (forage)	
			2.5 5.0	62	0.076 0.12	

Country, Year	Form.	Application		PHI, days	Residue, mg/kg ¹	Analytical method
		Method, timing	kg ai/ha			
			10% (w/w)		0.18 (forage)	
			2.5 5.0 10% (w/w)	76	0.048 0.068 0.072 (forage)	
	Furadan 3G	soil incorporation 2 days after planting	1.0	45	0.14 (forage)	
				62	0.086 (forage)	
				76	0.046 (forage)	
USA 1993	Furadan 4F	in-furrow at planting	1.1	44 Texas 40 Kansas 39 Nebraska 37 Missouri 37 Oklahoma 60 S Dakota	<u>1.2</u> < <u>0.05</u> < <u>0.05</u> <u>0.11</u> <u>0.13</u> < <u>0.05</u> (forage)	Barros
	Furadan 4F	foliar	2 x 0.6 ³	29 Texas 57 Kansas 53 Nebraska 58 Missouri 21 Oklahoma 39 S Dakota	0.06 <0.05 <0.05 <0.05 0.26 0.11 (forage)	Barros
				63 Texas 79 Kansas 69 Nebraska 80 Missouri 59 Oklahoma 91 S. Dakota	<u>(0.06)</u> (fodder) < <u>0.01</u> (grain) < <u>0.10</u> (fodder) < <u>0.01</u> (grain) < <u>(0.07)</u> (forage) < <u>0.01</u> (grain) < <u>0.10</u> (fodder) < <u>0.01</u> (grain) <u>0.20</u> (fodder) < <u>0.01</u> (grain) <u>0.19</u> (forage)	Barros

¹Carbofuran + 3-keto-carbofuran + 3-hydroxy-carbofuran

²No validation or limit of determination data were presented

³The two foliar applications are in addition to the one at-plant application at 1.1 kg ai/ha

Wheat. Supervised field trial results were reported from South Africa and the USA.

In the South African trials (Anon., 1985a) Curraterr 10G or Curraterr 9G (Curraterr 7% + Volaton 2%) were applied to the soil at the time of planting. The application rates were given as 0.03-0.06 g ai/linear metre. Grain samples (180 days PHI) were analysed for carbofuran and 3-hydroxy-carbofuran by an HPLC method. Neither was found at the stated limit of determination, 0.05 mg/kg. The limit of detection was not stated nor were any sample chromatograms supplied. Was not reported for South Africa or neighbouring countries, so the data could not be evaluated. The South African submission was rudimentary and did not contain necessary details.

The US trials (Stearns, 1986a) were conducted at six locations (South Dakota, Colorado, Oregon, Illinois, Washington and Arizona). Two foliar applications were made, one pre-boot and the second 21 days before harvest. Both were with Furadan 4F at 0.28 kg ai/ha. The volume of spray applied per ha was not stated. The mature grain samples were analysed for carbamates and phenolic metabolites by GC-MS (Schreier, 1989a). A limit of determination of 0.05 mg/kg was demonstrated for carbofuran and each of the metabolites. The limit of detection was estimated as 0.02 mg/kg. The 3-hydroxy-7-phenol was found in two trials (Colorado 0.05 mg/kg; Arizona 0.11 mg/kg) but was undetectable in the other four. All the other analytes (carbofuran, 3-keto-carbofuran, 3-hydroxy-carbofuran, 7-phenol) were below the limit of determination in all six locations. Thus, the residues from the six independent trials were (0.04), (0.04), (0.04), (0.04), <0.02 and <0.02 mg/kg.

US trials (Martin, 1985) were also conducted in Illinois with carbofuran as a seed treatment or at planting treatment. Immature spring wheat seedlings were collected 10, 20, 30, 45 and 60 days after emergence and analysed for carbofuran and 3-hydroxy-carbofuran by the method of Schreier. The demonstrated limit of determination was 0.1 mg/kg. The limit of detection was estimated to be 0.02 mg/kg for each analyte. The results are shown in Table 38.

US GAP specifies 2 post-emergence ground or aerial applications of an F formulation at 0.28 kg ai/ha, made before the heads emerge from the boot. Treated forage may not be fed. GAP limited to Nebraska, South Dakota and Wyoming allows the application of the 4F formulation in-furrow at planting to small grains (including barley, oats and wheat) at 1.5 g ai/cm row, with a 15-cm minimum row spacing. The feeding of treated forage is prohibited.

Table 38. Residues of carbofuran and 3-hydroxy-carbofuran in or on immature wheat plants¹ following seed treatment or at planting treatment with carbofuran at 1.0 kg/ha.

-Treatment/Procedure	Days after emergence	Residue, mg/kg	
		Carbofuran	3-OH-CF
Furadan 25 ST, applied as 1% ai to seed	10 (22-day PHI)	3.3	5.6
	20	1.1	5.0
	30	<0.02	0.62
	45	(0.07)	0.68
	60	<0.02	0.46
Furadan 4F, microtube to soil in furrow	10 (22-day PHI)	1.5	1.2
	20	0.60	2.4
	30	<0.02	0.52
	45	<0.02	(0.15)
	60	<0.02	(0.22)
Furadan 5G, in-furrow at planting	10 (22-day PHI)	2.1	2.8
	20	0.58	2.4

-Treatment/Procedure	Days after emergence	Residue, mg/kg	
		Carbofuran	3-OH-CF
	30	<0.1	0.38
	45	<0.1 (0.05)	0.30
	60	<0.1 (0.03)	0.78

¹Not a food or feed item.

Legume Vegetables

Soya beans. Trials were carried out in Brazil, France and the USA. Residues of carbofuran plus 3-hydroxy-carbofuran were below the limits of determination of the methods. Thailand submitted information on GAP for soya beans, but no data from the field trials (Thai Industrial Standard Institute, 1997). The results are given in Table 39.

GAP in Argentina, which can be used to evaluate the Brazilian trials, calls for application of the 10 G formulation in-furrow at planting at 1.5 kg ai/ha. In US GAP the 4F formulation is applied at planting at 1.7-2.0 kg ai/ha with a 100 cm row spacing or, if not used at planting, twice as a foliar spray at 0.28-0.56 kg ai/ha. No PHI is specified. GAP for France is 0.4 kg/ha of a 5% G formulation, but the data presented lacked detail.

Table 39. Total residues of carbofuran and 3-hydroxy-carbofuran in or on soya bean seeds.¹

Country, year, variety	Form.	Application		PHI, days	Residue, mg/kg	Method of analysis, LOD	Ref.
		Method, timing	kg ai/ha)				
Brazil, 1994/ Engopa 201- Gold	5% G	in-furrow at planting	2	75	<0.05	Leppert, 0.1	Anon., 1997 FMC
			4	75	<0.05	Leppert, 0.1	Anon., 1997 FMC
France, 1988/ King	5% G	Soil at planting	0.60	150	<0.04	Blass, 0.04	Anon., 1988
USA (NE) , 1979	Furadan 4F	foliar spray at pod set	0.28	63	<0.05	Mollhoff, 0.1	Cook, 1978
		foliar spray at pod set and at pod maturity	2 x 0.28	36	0.10	Mollhoff, 0.1	Cook, 1978

¹No data were submitted for forage or fodder

Yard-long beans. Thailand submitted information on GAP, but no data on residues (Thai Industrial Standards Institute, 1997).

Root and tuber vegetables

Carrots. The Netherlands submitted summary reports of field trials with Curraterr 200 SC applied to soil in 1980 before sowing carrot seeds (The Netherlands, 1997). The soluble concentrate was applied at 3.6-3.7 kg ai/ha and 3.6-7.5 g ai/l. Samples were analysed by the HPLC method of The Netherlands. No recovery data or storage periods from harvest to analysis were reported. The findings are shown in Table 40. Multiple results are from field replicates.

No GAP was available for The Netherlands or Europe. The data could not be evaluated for the estimation of a maximum residue level.

Table 40. Residues of carbofuran and 3-hydroxy-carbofuran in or on carrot roots from the application of Curraterr 200 SC to the soil at the time of sowing in The Netherlands.

Location/ Year	Rate		PHI, days	Residues, mg/kg		Method of analysis
	kg ai/ha	g ai/l		Carbofuran + 3-OH-CF	3-OH-CF conjugates	
Alkmaar, 1977	3.6	3.6	95	0.05; 0.08; 0.14; 0.26 (mean 0.14)	<0.01; <0.01; <0.01	Netherlands GLC (Mollhoff, 1979a)
Nooruyk- erhouk, 1977	3.6	6.0	118			
Metevik/ 1978	3.7	6.2	102	<0.01 <0.01 < 0.01 (mean <0.01)	<0.01 <0.01 <0.01	
Wageningen, 1980	3.7	7.5	111	<0.02 <0.02 <0.02 <0.02	0.05 0.05 0.06 0.05 (mean 0.05)	Netherlands HPLC
Zaltbommel	3.7	7.5	145	<0.02 <0.02 <0.02 <0.02	<0.01 <0.01 <0.01 <0.01	Netherlands HPLC

Abbreviated compound names: see Figure 1

Celeriac. The Netherlands submitted a summary report of one field trial with Curraterr 200 SC applied to the soil before planting celeriac in 1978. The application rate was 3 kg ai/ha and 5 g ai/l. Mature roots were harvested 158 days after the application and samples were analysed by the method of Molhoff (The Netherlands GLC method). The combined residue of carbofuran and 3-hydroxy-carbofuran was 0.05 mg/kg and the residue of 3-hydroxy-carbofuran conjugates <0.1 mg/kg. The stated limits of determination were 0.05 mg/kg for carbofuran plus 3-hydroxy-carbofuran and 0.1 mg/kg for 3-hydroxy-carbofuran conjugates. No GAP was available for The Netherlands or Europe.

Potatoes. Field trials were reported from Colombia, France, the UK and the USA. The tubers were treated at planting or post-emergence. Results are shown in Table 41.

GAP for Poland (2 kg ai/ha of 5 G) may be used for the evaluation of trials in France and the UK. GAP for Colombia or a neighbour was not available and the data from Colombia could not be considered for the estimation of a maximum residue level. US GAP requires an in-furrow application at planting of a 4F formulation at 3.4 kg ai/ha (Delaware, Pennsylvania, Virginia only). The same formulation may also be used post-emergence at 3.4 kg ai/ha in a shank or band application up to a four-inch rosette potato size. Up to 8 applications of the 4F formulation may be made at 1.1 kg ai/ha with a PHI of 14 days. The maximum foliar application is 3.4 kg ai/ha after an at-plant application, but no foliar applications may be made after a shank or band application.

Table 41. Residues of carbamates and phenolic metabolites in or on white potato tubers from the application of carbofuran.

Country, year, variety	Form./ Applin.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		

Country, year, variety	Form./ Applin.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
France, 1973/ Bintje	Curraterr 5 G in-furrow at planting	1.5	-	154	<0.05 ¹	-	Mollhoff, 0.1	Bayer 1975 Bayer 7155-75
UK, 1977/ Maris Piper	Yaltox 5G in-furrow at planting	5.0	-	101	0.03 ²	-	Mollhoff, 0.01	Bayer 1977 Bayer TCR 155/20-77
Columbia 1984/	Furadan 3G at planting and band. Furadan 3F foliar	3 x 1.0 2 x 1.3 (5.6 total)	not stated	18	<0.02, ³ 0.07, 0.06	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3G at planting and band. Furadan 3F foliar	2 x 1.0 1 x 1.3 (3.3 total)	not stated	69	0.05, 0.06, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3G	3 x 1.0	-	134	0.06, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3F	0.8	not stated	171	<0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1984	Furadan 3G at planting Furadan 3G band post-emergence Furadan 3F band	0.42 2 x 0.18 1.0 (1.78 total)	not stated	28	0.10, 0.06, 0.06	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1985	Furadan 3G at planting Furadan 3F foliar	1.0 2 x 1.0 (3.0 total)	not stated	132	<0.02, <0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1985	Furadan 3G at planting Furadan 3F foliar	1.0 2 x 1.0	not stated	92	<0.02, <0.02, <0.02: <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia, 1985	Furadan 3F foliar	3 x 1.0	not stated	92	<0.02, <0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316

Country, year, variety	Form./ Applin.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
Columbia/ 1985	Furadan 3F foliar	3 x 1.0	not stated	66	0.07, <0.02, <0.02, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
Columbia/ 1985	Furadan 3F foliar	4 x 1.0	not stated	35	<0.02, <0.02, 0.06, <0.02	-	Schreier, 0.05	Martin 1985 FMC P-1316
USA (ID)/ 1991/ Russet Burbank	Furadan 4F banded at hill-up	3.4	0.036	105	<u>0.04</u> , 0.04 (3-OH-CF)	0.23, 0.21	Barros, 0.03	Singer, 1992a
USA (ID)/ 1991/ Russet Burbank	Furadan 4F banded at hill-up	3.4	0.020	105	<0.01, < <u>0.01</u>	0.09, 0.09	Barros, 0.03	Singer, 1992a
USA (ND)/ 1991/ Norchip	Furadan 4F banded at hill-up	3.4	0.036	74	<0.01, < <u>0.01</u>	0.07, 0.07	Barros, 0.03	Singer, 1992a FMC P-2682
USA (OR)/ 1991/ Manona	Furadan 4F banded at hill-up	3.4	0.036	88	<0.01, < <u>0.01</u>	0.11, 0.15	Barros, 0.03	Singer, 1992a FMC P-2682
USA (PA)/ 1991/ Katahdin	Furadan 4F banded at hill-up	3.9	0.042	119	<0.03, < <u>0.03</u>	0.26, 0.31	Barros, 0.03	Singer, 1992a
USA (WA), 1991/ Russet Burbank	Furadan 4F Banded at hill-up	3.4	0.037	126	0.03, <u>0.03</u> (3-OH-CF)	0.48, 0.47 (7-P, 3-K-7-P)	Barros, 0.03	Singer, 1992a

¹Carbofuran + 3-hydroxy-carbofuran + conjugates

²Carbofuran + 3-hydroxy-carbofuran

³Carbofuran + 3-hydroxy-carbofuran + 3-keto-carbofuran

No data were submitted on trials with multiple foliar post emergence applications.

Sugar beet. Field trials were conducted in France, Italy, Germany, the UK and the USA. Applications were at planting and/or foliar and only carbamate residues were determined except in the US trials where the phenol metabolites were also determined.

The European trials were evaluated against the GAP for Hungary (1.5-2 kg ai/ha of 10 G) and Bulgaria (875 g ai/100 kg ST). US GAP is a soil band treatment with the 4F formulation at 2.2 kg ai/ha with a 90-day PHI in Idaho, Oregon, Texas only and a soil band at planting through the six-leaf

stage of the 4F formulation at 0.01 g ai/cm of row with a 90-day PHI in Nebraska only. The results are shown in Table 42.

Table 42. Total residues of carbamates and phenolic metabolites from the application of carbofuran to sugar beet.

Country, Year, Variety	Form./appln.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
France, 1973/?	Curraterr 5G Unknown (?at-plant)	0.66		175	<0.05 (root) <0.05 (foliage)		Mollhoff 0.1	Mollhoff, 1974
France, 1973/?	Curraterr 5G Unknown (?at- plant)	0.68		186	<0.05 (root) <0.05 (foliage)		Mollhoff 0.1	Mollhoff 1974
Germany, 1973/ Poly-Beta	Curraterr 5G In-furrow at- plant	0.50		191	<0.05 (root) <0.05 (foliage)		Mollhoff 0.1	Mollhoff 1974
		1.0		191	<0.05 (root) 0.15 (foliage)			
Germany/ 1984/ Geem 65	500 SC Pelleting (with seed)	0.033 (30 g ai/100,000 pills)		174	<0.05 (root) 0.07 (foliage)		Mollhoff 0.05	Mollhoff 1985
				177	<0.05 (root) <0.05 (foliage)			
Germany/ 1984/ Novadima	500 SC Pelleting (with seed)	0.033 (30 g ai/100,000 pills)		208	<0.05 (root) <0.05 (foliage)			
UK, 1974/ Amono	Curraterr 5G Spreading at planting	0.75		136	<0.05 (root) <0.1 (foliage)		Mollhoff 0.1	Mollhoff 1975
Italy, 1974/Dickman Dima	Curraterr 5G Spreading at planting	0.60		155	<0.05 (root)		Moll-hoof 0.1	Mollhoff 1974
USA (Idaho), 1992/ WS-88	Furadan 4F Banded, postemergence (2-6 leaf)	2.24	0.019	86	0.05 ¹ (foliage)	0.18 ² (foliage) <0.01 (root)	Barros 0.03	Singer, 1992b
USA (Oregon), 1991/ Great North- western 2905	Furadan 4F Banded, post- emergence	2.24	0.017	92	<0.01 (foliage) <0.01 (roots)	0.03 (foliage) <0.03 (roots)	Barros 0.03	Singer, 1992b
USA (Idaho), 1991/ WS-88	Furadan 4F Banded, post- emergence	2.24	0.028	173	<0.01 (foliage) <0.01 (roots)	<0.03 (foliage) <0.03 (roots)	Barros 0.03	Singer, 1992b

Country, Year, Variety	Form./appln.	Rate		PHI, days	Residue, mg/kg		Method of analysis, LOD	Ref.
		kg ai/ha	kg ai/l		Carbamates	Phenols		
USA (Wy- oming), 1991/ Monohikari	Banded, at planting	2.24	0.034	181	<0.01 (foliage) <0.01 (roots)	<0.03 (foliage) <0.03 (roots)	Barros 0.03	Singer, 1992b

¹3-OH-CF, 0.03 mg/kg and 0.07 mg/kg total carbofurans

²About 50% 3-keto-7-phenol, 0.26 mg/kg and 0.10 mg/kg total phenols

Swedes or turnips. Supervised field trials were conducted in France and Norway. The results are shown in Table 43.

No information on GAP was available so the data could not be evaluated for the estimation of a maximum residue level.

Only carbamate residues were determined. The applications were made at planting or early bulb formation. No measurable residues were found in any of the samples.

Table 43. Residues of carbamates in or on swedes from the application of carbofuran.

Country, Year, Variety	Form./ Application	Rate, kg ai/ha	PHI, days	Residue, mg/kg	Method of analysis, LOD ¹	Ref.
France, 1978/ Croissy	Curraterr 5G at planting	1.0	59	<0.05 (tops) <0.05 (roots)	Mollhoff 0.1	Mollhoff 1979
UK, 1977/ Acme (rutabaga)	Yaltox 5G spread at early to mid bulb formation	1.25	40	<0.01 (root)	Mollhoff 0.01	Anon. 1977
Norway, 1982/ (rutabaga)	Curraterr 5G	1.25	133	<0.05 (root, carbofuran) (0.05) (root, 3-hydroxy- carbofuran)	Mollhoff 0.1	Mollhoff 1983
Norway, 1982/ (rutabaga)	Curraterr 5G post-emergence after thinning	1.25	98	<0.05 (root, carbofuran) (0.05) (root, 3-hydroxy- carbofuran)	Mollhoff 0.1	Mollhoff 1983
Norway, 1982/ (rutabaga)	Curraterr 5G at planting	2.5	133	<0.05 (root, carbofuran) (0.05) (root, 3-hydroxy- carbofuran)	Mollhoff 0.1	Mollhoff 1983

¹No data were submitted to support the claimed limits of determination

Cotton seed (SO 691). Field trials were carried out in Brazil and the USA. The Meeting was informed that trials were in progress (1996-1997) in Southern Europe.

The trials in Brazil were with a single post-emergence foliar treatment of cotton plants with Furadan 350 SC at 1.0 or 2.1 kg/ha, both at about 600 l/ha, or a single post-emergence application along the plant rows with Furadan 50G at 2.5 or 5 kg/ha. In all cases the PHI was 45 days. Delinted

cotton seeds were analysed by the method of Leppert. It was claimed that the method was validated at 0.1 mg/kg with 81% recovery, but no data were provided. The residues of carbofuran and 3-hydroxy-carbofuran were below the limit of determination, 0.1 mg/kg, in all four trials. The method does not include a hydrolysis step to release conjugated carbamates.

GAP for Brazil specifies an in-furrow treatment at planting with the 5 G formulation at 1.5-3 kg ai/ha or the 350 SC formulation at 0.7-1.05 kg ai/ha. There is also a seed treatment at 0.7 kg ai/100 kg seed with 350 ST formulation. The above trials therefore did not comply with GAP.

In two other trials in Brazil (San Paulo University, 1994) cotton seed (IAC 20) was treated with Furadan 350 TS at rates of 0.70 and 1.4 kg ai/100 kg seed. The seeds were planted in 1994 at an unstated rate of seeding and mature cotton seeds were harvested 154 days after treatment. The delinted seeds were analysed by the method of Leppert. Carbofuran and 3-hydroxy-carbofuran were below the limit of determination, 0.1 mg/kg. These trials complied with GAP.

The US trials (Shevchuk, 1993) were in California, Arizona, Texas, Mississippi and Louisiana with two broadcast foliar applications of Furadan 4F. Carbofuran and its carbamate and phenol metabolites were determined in delinted cotton seed by the method of Barros. A mass-selective detector was used for the phenols. Limits of determination were established for carbofuran at 0.1 mg/kg (76 ± 4% recovery), 3-hydroxy-carbofuran at 0.1 mg/kg (71 ± 1% recovery), 3-keto-carbofuran (77 ± 4% recovery), the 7-phenol at 0.2 mg/kg (85 ± 10% recovery), 3-keto-7-phenol at 0.2 mg/kg (107 ± 13% recovery) and 3-hydroxy-carbofuran at 0.2 mg/kg (73 ± 17% recovery). Recoveries of the carbamates at 0.05 mg/kg showed poor precision. Limits of detection of 0.01 mg/kg for the carbamates and 0.05 mg/kg for the phenols were claimed. The results of the trials are shown in Table 44. No results were reported for cotton fodder.

US GAP requires in-furrow treatment at planting with the 4F formulation at 0.14 or 1.12 kg ai/ha. The feeding of cotton forage is prohibited. The reported trials are not according to GAP as they are post-emergence treatments with PHIs of about 30 days.

Table 44. Residues of carbofuran and its carbamate and phenol metabolites in or on delinted cotton seed from the foliar application of Furadan 4F to cotton plants.

Location, Year, Variety	Rate		PHI, days	Residue, mg/kg							
	kg ai/ha	g ai/l		carbofuran	3-K-CF	3-OH-CF	total carbamates	7-phenol	3-K-7-P	3-OH-7-P	total phenols
Louisiana, 1992, Deltapine 5415	2 x 0.28	3	27	<0.05	<0.05	<0.05		<0.1	<0.1	<0.1	<0.1
Mississippi, 1992/ DES 119	2 x 0.28	3	27	<0.05	<0.05	<0.05		<0.1	<0.1	<0.1	<0.1
Texas, 1992/ GSC 71+	2 x 0.28	4	27	<0.05	<0.05	<0.05	<0.05	<0.1	<0.1	<0.1	<0.1
Texas/ 1992/ Paymaster HS 200	0.28 2.8 (3.1 total)	4	27	(0.05)	<0.05	(0.02)	(0.07)	<0.1	<0.1	<0.1	<0.1
Arizona, 1992/ DPL 5461	2 x 0.28	3	27	(0.06)	<0.05	<0.05	(0.06)	<0.1	<0.1	<0.1	<0.1

Location, Year, Variety	Rate		PHI, days	Residue, mg/kg							
	kg ai/ha	g ai/l		carbofuran	3-K-CF	3-OH-CF	total carbamates	7-phenol	3-K-7-P	3-OH-7-P	total phenols
California, 1992/ Germaines GC-510	2 x 0.28	3	27	<0.05	<0.05	<0.05	<0.05	<0.1	<0.1	<0.1	<0.1

Abbreviated compound names: see Figure 1

Peanuts. Field trials were reported from Brazil and the USA. In Brazil, peanut plants were sprayed 80 days after planting with Furadan 350 SC at 1.75 or 3.0 kg/ha, 2.4 and 4.2 g/l respectively (Sao Paolo University, 1994b). Peanuts with hulls were harvested 14 days after the treatment. The peanuts were shelled and the carbofuran residues in the kernels determined by the method of Leppert. Recoveries of $78 \pm 6\%$ at 0.1 mg/kg were reported, without data. No carbofuran (<0.1 mg/kg) was found in the two samples. The data could not be used for the estimation of a maximum residue level because adequate details were not provided. Brazilian GAP is an application at planting of the 350 SC formulation at 1.4-1.8 kg ai/ha (100-300 l/ha) or the 5 G formulation at 2 kg ai/ha.

Thailand submitted information on GAP, but none on residues (Thai Industrial Standards Institute, 1997).

Fourteen supervised field trials were reported from the USA (Helt, 1980, Nelson, 1981), in which peanut fields were treated at pegging, and in the 1981 trials at planting. Residues of carbofuran and 3-hydroxy-carbofuran were determined in or on kernels and hulls by the method of Schreier, with an NPD only. Limits of determination of 0.05 mg/kg or 0.1 mg/kg for carbofuran and 3-hydroxy-carbofuran on peanut kernels and 0.10 mg/kg or 0.20 mg/kg for carbofuran and 3-hydroxy-carbofuran on hulls were demonstrated by the analysis of fortified controls, with the following recoveries: carbofuran on kernels 88% at 0.05 mg/kg, $78 \pm 6\%$ (n = 4) at 0.10 mg/kg; carbofuran on hulls 76% at 0.10 mg/kg, 74% at 0.20 mg/kg; 3-hydroxy-carbofuran on peanuts 72% at 0.05 mg/kg, $83 \pm 15\%$ (n = 4) 94% at 0.10 mg/kg, 75% at 0.20 mg/kg. The results of the analyses are shown in Table 45. There are no registered US uses.

Table 45. Residues in or on peanut kernels and hulls from the application of carbofuran at planting and/or postemergence.

Location, Year, Variety	Form.	Application		PHI, days	Sample	Residue, mg/kg	
		kg ai/ha	Timing, method			Carbofuran	3-OH-CF
Alabama, 1979/	Furadan 10 G	2.2	Pegging 30-36 cm band	83	kernels	<0.02	<0.02
					hulls	(0.06)	0.30
Georgia, 1979/	Furadan 10 G	2.2	Pegging 30-36 cm band	92	kernels	<0.02	<0.02
					hulls	(0.03)	(0.08)
North Carolina/ 1979/ Florigiant	Furadan 10 G	2.2	Pegging 30-36 cm band	80	kernels	(0.02)	0.09
					hulls	0.12	0.62
Oklahoma / 1979/ Runner	Furadan 10 G	2.2	Pegging 30-36 cm band	121	kernels	<0.02	<0.02

Location, Year, Variety	Form.	Application		PHI, days	Sample	Residue, mg/kg	
		kg ai/ha	Timing, method			Carbofuran	3-OH-CF
					hulls	(0.04)	<0.05
Oklahoma, 1979/ Runner	Furadan 10 G	2.2	Pegging 30-36 cm band	120	kernels	(0.01)	<0.02
					hulls	(0.03)	0.14
Virginia, 1979/Virginia 61R	Furadan 10 G	2.2	Pegging 30 -36 cm band	64	kernels	<0.02	<0.02
					hulls	(0.06)	0.10
Virginia, 1979 Florigiant	Furadan 10 G	2.2	Pegging 30-36 cm band	82	kernels	(0.01)	<0.02
					hulls	(0.03)	<0.05
Georgia, 1980/ Florunner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels	<0.02	0.22
					hulls	1.2	1.8
Georgia, 1980/Florunner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	50	kernels	<0.02	(0.06)
					hulls	0.48	0.60
Georgia, 1980/ Florunner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels	(0.08)	(0.06)
					hulls	0.74	0.35
Georgia, 1980/Runner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels	(0.02)	(0.08)
					hulls	0.22	0.22
Georgia, 1980/ Runner	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels	0.11	0.42
					hulls	2.6	1.5
North Carolina/ 1980/ Florigiant	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels	(0.04)	0.10
					hulls	0.36	0.40
North Carolina/ 1980/ Florigiant	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36 cm band	60	kernels	(0.04)	<0.02
					hulls	0.32	(0.08)
North Carolina/ 1980/ NC 6	Furadan 10 G	1.1 2.2 2.2 (5.5 total)	In-furrow at plant Band at plant Pegging 30 -36	60	kernels	(0.04)	<0.05
					hulls	(0.12)	(0.12)

Location, Year, Variety	Form.	Application		PHI, days	Sample	Residue, mg/kg	
		kg ai/ha	Timing, method			Carbofuran	3-OH-CF
			cm band				

Abbreviated compound names: see Figure 1

Rape (canola). Field trials were carried out in Canada and France. The trials in Canada included seed, at-plant and post-emergence foliar treatments. The treatments in France were at planting.

No GAP was reported for France, other European countries or Canada, but temporary GAP was reported for the USA where the 10% G formulation may be applied at 0.28 kg ai/ha by soil incorporation at planting. The use is limited to Minnesota, Montana, North Dakota and Washington, states bordering on or near Canada.

In Canada, rape seed was treated with Furadan 5G at planting in Manitoba and Alberta at 0.28 kg/ha (Leppert, 1980a). One or two additional applications of Furadan 4.8F were made at various growth stages (2 leaf, 3-4 inch, post-podding, post-flowering) at rates of 0.28 kg/ha for all stages except post-flowering at 0.14 kg/ha. The PHIs ranged from 23 days (post-flowering) to 108 days (at planting only). Rape seed was collected at normal harvest and analysed by the method of Leppert. Limits of determination of 0.09 mg/kg were demonstrated for carbofuran and 3-hydroxy-carbofuran, which were detectable (>0.01 mg/kg) but not measurable in one of eleven trials. The trial was in Manitoba and involved an at-plant application, a foliar application at the 3-4 inch growth stage, and a foliar application after podding, all at 0.28 kg ai/kg, with a 39-day PHI. Carbofuran was detected at an estimated 0.02 mg/kg and 3-hydroxy-carbofuran at an estimated 0.01 mg/kg, the total residue estimated as 0.03 mg/kg.

In Saskatchewan and Manitoba, Canada, rape seeds were treated with carbofuran (Furadan 350 ST) at 12 and 24 g ai/kg seed (Leppert, 1984). In 6 of the 7 trials, the treatment mixture also contained carbendazim and thiram. The seeds were grown to mature plants and the seeds from these were harvested (PHI 79-127 days) and analysed for carbofuran and 3-hydroxy-carbofuran by the Leppert method. A limit of determination of 0.1 mg/kg was demonstrated for each analyte. Carbofuran was detected at 0.01 mg/kg in one trial (127-day PHI). Neither carbofuran nor 3-hydroxy-carbofuran were detected in the remaining 6 trials.

As the Canadian trials did not comply with the temporary US GAP they could not be evaluated for the estimation of a maximum residue level.

In the French trials in 1976 (Ministry of Agriculture, 1977) carbofuran (5% granular) was incorporated into the soil when the seed was sown at rates of 0.68, 0.75 and 1 kg/ha, with PHIs of 275, 300 and 280 days. Seeds were analysed by an undefined semi-quantitative technique. It was claimed that carbofuran and 3-hydroxy-carbofuran were absent at limits of 0.005 mg/kg and 0.010 mg/kg respectively. GAP for France specifies 0.45 kg ai/ha of a 5% G formulation.

In four trials in 1979 in France (Anon., 1997) Curraterr 5G was applied in the furrow at rates of 0.9, 0.95 and 0.95 kg ai/ha. The PHIs were 345-364 days. Rape seed samples and straw were analysed by the method of Mollhoff and limits of determination of 0.2 mg/kg were demonstrated for carbofuran and 3-hydroxy-carbofuran on both seed and straw. The residues were undetectable in the seed and 0.20-0.24 mg/kg in the straw. The moisture content of the straw was not determined.

Sunflower. Field trials were reported from Canada, France and the USA. The Meeting was informed that trials were in progress (1996-1997) in northern and southern Europe.

No GAP was reported for France or Canada, but GAP in the USA, which may be applicable to Canada, specifies an in-furrow application of the 4F formulation at 3.1 kg ai/ha at planting and four foliar applications at 0.56 kg ai/ha with a 28-day PHI.

In six trials in Manitoba and Saskatchewan, Canada (Leppert, 1980b) sunflower plants 30-60 cm in 1-2 ft height were treated with one or two foliar applications of Furadan 4.8F at 0.28 or one at 0.56 kg/ha. The PHIs ranged from 91 to 111 days. Sunflower seeds were harvested at maturity and analysed by the method of Leppert. Limits of determination of 0.05 mg/kg were demonstrated for carbofuran (76% and 86% recovery) and 3-hydroxy-carbofuran (92 and 92%). No carbofuran or 3-hydroxy-carbofuran was found at or above 0.05 mg/kg. Carbofuran was detected in all samples at estimated concentrations of 0.01-0.03 mg/kg and 3-hydroxy-carbofuran was detected at 0.01 mg/kg in a single sample from the application of 0.56 kg/ha to 60 cm high plants after a 101-day PHI.

In France (Anon., 1977) carbofuran (5G) was applied to the seedbed line at the planting of sunflowers. The application rate was 0.40 kg ai/ha and the PHI 135 days. Residues of 0.02 mg/kg carbofuran and <0.05 mg/kg 3-hydroxy-carbofuran were reported, but the method of analysis was described as semi-quantitative and no details of it were provided.

In the USA (Brutschy, 1984) Furadan 4F or 15G applied as a band at cultivation at 1.1 kg ai/ha was followed by four foliar applications of Furadan 4F, each at 0.56 kg ai/ha. Seeds were collected at maturity and analysed by the method of Schreier. The phenol metabolites were determined by GC-MSD. Limits of determination of 0.05 mg/kg were demonstrated for each analyte by the analysis of triplicate fortified control samples, with the following recoveries: carbofuran 66, 50, 64%, 3-hydroxy-carbofuran 68, 62, 68%, the 7-phenol 68, 72, 56%, the 3-keto-7-phenol 98, 86, 80% and the 3-hydroxy-7-phenol 84, 84, 66%. The recoveries of the carbamates were low, about 60%, over the entire tested range of 0.05-0.20 mg/kg. The trial results are shown in Table 46. The maximum total residue was 0.65 mg/kg. Although the trials were according GAP, none were at the maximum at-plant application rate. The later season foliar applications, which were at the maximum rate, are more likely to have contributed most to the carbamate residues.

Table 46. Residues of carbofuran, 3-hydroxy-carbofuran and phenolic metabolites in or on sunflower seeds from the treatment of sunflowers with carbofuran.

Location, Year, Variety	Application		PHI, days	Residue, mg/kg						
	Form.	kg ai/ha		CF	3-OH-CF	Total carb.	7-P	3-K-7-P	3-OH-7-P	Total phenols
Kansas, 1983/ Oil	15G	1 x 1.1	61	0.06	0.05	0.11	(0.02)	(0.02)	(0.04)	(0.08)
	4F	4 x 0.56								
Arkansas, 1983/ Sunbred 265	15G	1 x 1.1	52	(0.04)	(0.02)	(0.06)	(0.02)	(0.02)	(0.04)	(0.08)
	4F	4 x 0.56								
Minnesota, 1983/ Sigco Dwarf-Oil	15G	1 x 1.1	42	0.05	(0.02)	0.07	(0.02)	(0.02)	(0.02)	(0.06)
	4F	4 x 0.56								

Location, Year, Variety	Application		PHI, days	Residue, mg/kg						
	Form.	kg ai/ha		CF	3-OH-CF	Total carb.	7-P	3-K-7-P	3-OH-7-P	Total phenols
	4F	1 x 1.1 4 x 0.56	42	(0.04)	(0.02)	(0.06)	<0.02	<0.02	(0.02)	(0.02)
Illinois, 1983/ Oil	15G 4F	1 x 1.1 4 x 0.56	53	0.30	0.12	0.42	0.06	0.06	0.11	0.23
	4F	1 x 1.1 4 x 0.56	53	0.28	0.11	0.39	(0.04)	0.05	0.08	0.17
North Dakota, 1983/ Cargill 205-Oil	15G 4F	1 x 1.1 4 x 0.56	50	0.10	(0.02)	0.12	(0.02)	(0.02)	(0.02)	(0.06)
	4F	1 x 1.1 4 x 0.56	50	0.06	(0.02)	0.08	<0.02	(0.02)	(0.01)	(0.03)
Illinois, 1983/ Confectionery	15G 4F	1 x 1.1 4 x 0.56	53	0.21	0.08	0.29	0.06	0.06	0.08	0.20
	4F	1 x 1.1 4 x 0.56	53	0.18	0.08	0.26	0.05	0.05	0.06	0.16

Abbreviated compound names: see Figure 1

Leeks. The Netherlands provided the results of two field trials, one each in Waandenburg and Huissen, in 1977 (Ministry of Health, Welfare and Sport, 1997). Curraterr 200 SC was applied to the soil before planting at 4.4 kg ai/ha, 7.4 g/l. The mature crop was harvested 118 or 125 days after application. Leek bulbs were analysed by the method of Mollhoff. No limits of determination were stated. Results were reported as the sum of carbofuran, 3-hydroxy-carbofuran and conjugates of 3-hydroxy-carbofuran. In the Huissen trial the residues were <0.1 mg/kg (0.07, 0.07 mg/kg) and in the Waandenburg trial 0.13 mg/kg (0.15, 0.12, 0.12 mg/kg). No GAP was reported for The Netherlands or a neighbouring nation.

Onions. The Netherlands reported the results of three field trials in 1977 and 1978. Mature samples were analysed by the method of Mollhoff and limits of determination of 0.1 mg/kg were claimed for carbofuran plus 3-hydroxy-carbofuran and for conjugates of 3-hydroxy-carbofuran. The results are shown in Table 47. No GAP was reported.

Table 47. Residues of carbamate in onions after application of carbofuran.

Location, Year	Form.	Application			PHI, days	Carbofuran 3-OH-CF, mg/kg	Conjugate of 3-OH-CF, mg/kg <0.1
		Timing	Rate, kg ai/ha	Rate, kg ai/l			
Nieuu Vossemeir, 1978	Curraterr 5G	after sowing	1.5	-	176	<0.1	<0.1
Willemstad, 1978	Curraterr 5G	after sowing	1.5	-	158	<0.1	<0.1
Zwingelspoon, 1977	Curraterr 200 SC	before sowing	5	8.4	90	<0.1	

Abbreviated compound names: see Figure 1

Celery. The Netherlands provided the results of two trials in 1978 in Berghen (trial 1) and Schayk (trial 2). Curraterr 200 SC was applied at 3.12 kg ai/ha, 5.2 g/l, to the soil one day before planting celery (goudgele relfblekende). Celery was harvested 84 days (trial 1) or 90 days (trial 2) after the treatment and samples were analysed by the method of Mollhoff. No limits of determination were stated. The combined residue of carbofuran and 3-hydroxy-carbofuran averaged 0.21 mg/kg (0.21, 0.18, 0.24 mg/kg) in trial 1 and 0.15 mg/kg (0.21, 0.12, 0.13 mg/kg) in trial 2. In both trials the conjugate of 3-hydroxy-carbofuran was <0.1 mg/kg (not detected). No GAP was reported for celery.

Tomatoes. Field trials were conducted in Brazil (Anon., 1994), Canada (Hawk, 1975), France (Anon., 1986b), Mexico (Shuttleworth, 1975) and the USA (Hawk, 1974). Thailand reported field trials according to GAP but without data or results (Thai Industrial Standards Institute, 1997). The findings are shown in Table 48.

GAP for Brazil specifies the use of the 350 SC formulation in-furrow at planting at 1.75 kg ai/ha or the 5 G at planting or transplanting at 4 kg ai/ha. There is no US GAP. GAP was not reported for Mexico, Canada or France.

Table 48. Residues of carbofuran and 3-hydroxy-carbofuran in or on tomatoes from the application of carbofuran to tomato plants.

Location, Year, Variety	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis	Recovery, mg/kg/%	
		Method	kg/ha		CF	3-OH-CF		CF	3-OH-CF
Brazil, 1993/ Santa Clara	Furadan 350 SC	spray to soil around plant (200 l/ha)	3.5	60	<u><0.05</u>		Leppert	0.1/89	
			7.0	60	<0.05				
Brazil, 1993/ Santa Clara	Furadan 50G	broadcast and soil incorporation	2.6	60	<0.05		Leppert	0.1/89	
			5.2	60	<0.05				
Brazil, 1993/ Roma VF	Furadan 50G	broadcast and soil incorporation	4	60	<u><0.05</u>		Leppert	0.1/89	
			8	60	<0.05		Leppert	0.1/89	

Location, Year, Variety	Form.	Application		PHI, days	Residue, mg/kg		Method of analysis	Recovery, mg/kg/%	
		Method	kg/ha		CF	3-OH- CF		CF	3-OH-CF
Canada (Ontario), 1974/	Furadan 4.8F	foliar spray	0.56	83	<0.05	<0.05	Schreier	0.1/70, 85	0.1/93
		foliar spray	2 x 0.27	1	0.25	<0.05			
				3	<0.05	<0.05			
				6	<0.05	<0.05			
				10	<0.05	<0.05			
				27	<0.05	<0.05			
France, 1986/ Cam-Root	Curraterr MG (5%)	spread on seed beds	1.5	116	<0.05	<0.25 (conjugate)	Mollhoff		
France, 1986/ Lerica, F1 hybrid	Curraterr MG (5%)	spread on ground with 2-4 leaves on plants	0.75	98	<0.05	<0.25	Mollhoff		
France, 1986/ Variety ACE SS	Curraterr MG (5%)	Spread on ground before fruit formation	1	47	<0.05	<0.25	Mollhoff		
France, 1986/ Arimex	Curraterr MG (5%)	Spreading on 20 cm strips, at 2nd cluster, L 25 stage	1	29	<0.05	<0.25 (conjugate)	Mollhoff		
Mexico (Valle de Culia-can)/ 1975/	Furadan 3G	Spread, sidedress incorporated	2	7	<0.05 (9 plots) 0.056 (1 plot)	<0.05	Schreier	0.05/90	0.1/96, 80
	Furadan 75WP	Foliar	3 x 1 (total 5)						
				14	<0.05 (1 detect, 9 no detects)	<0.05 (10 no detects)			
USA/ (Florida), 1971/	Furadan 4F	Foliar spray	5 x 1.2	7	<0.05	<0.05	Schreier	0.1/100, 94, 80	0.1/109, 94, 64
USA / (Virginia, Maryland, Ohio), 1971	Furadan 10G	Banded at transplant	1 x 1.1	57 (VA) 77 (OH) 133 (MD)	<0.105	<0.05	Schreier	0.1/100, 94, 80	0.1/109, 94, 64

Abbreviated compound names: see Figure 1

Peppers, Chilli. Two trials were conducted, one each in California and New Mexico (Kim, 1995a). Furadan 4F was applied to the soil immediately before planting peppers at 1.2 kg ai/ha (5.9 g ai/l) in California (variety Jalapeno M) and at 1.1 kg ai/ha (3.5 g ai/ha) in New Mexico (variety NM 64). After approximately 2 months in California and 4 months in New Mexico, Furadan 4F was applied as a side-dressing (directed spray). The application rates were 1.7 kg/ha (9.0 g ai/l) in California and 1.7 kg/ha (14 g ai/l) in New Mexico. Mature peppers were harvested after a PHI of 28 days in both states.

The trials reflect the maximum US GAP.

The samples were analysed by the method of Barros. Limits of determination of 0.05 mg/kg were demonstrated for all the analytes, with the following recoveries from fortified control samples: carbofuran 94%, 3-keto-carbofuran 82%, 3-hydroxy-carbofuran 94%, 7-phenol 69%, 3-keto-7-phenol 91% and 3-hydroxy-7-phenol 96%. No carbamates were detected in any sample. The 7-phenol was detected below the limit of determination in both the California and New Mexico samples, at estimated levels of 0.02 and 0.04 mg/kg respectively. The 3-keto-7-phenol and the 3-hydroxy-7-phenol were also detected below the limits of determination in the New Mexico samples, both at 0.02 mg/kg.

Peppers, Sweet. Pepper plants were treated in 1974 with Furadan 4F 5 x 0.56 kg ai/ha in Ontario, Canada (Bednar and Stanovick, 1974). The PHI varied from 1 to 3 days. The samples were analysed by the method of Schreier, with limits of determination of 0.1 mg/kg for both carbofuran (78% recovery) and 3-hydroxy-carbofuran (97% recovery). At PHIs of 1, 2 and 3 days the carbofuran concentration was 0.26 mg/kg maximum (0.18 mg/kg average), 0.20 mg/kg maximum (0.18 mg/kg average) and 0.17 mg/kg maximum (0.15 mg/kg average) respectively. Residues of 3-hydroxy-carbofuran were <0.10 mg/kg at all PHIs.

Pepper plants in the USA (Kim, 1995b) were treated in 1994 either with Furadan 4F at 1.1 kg ai/ha at transplanting in-furrow or 4 weeks after transplanting as a side-dress. A second application was made after an interval of 1-3 months with side-dressing at 1.7 kg ai/ha (2.0 kg ai/ha in Florida). Bell peppers were collected at maturity 28 days after the final treatment from four plots in Florida, California, Texas and New Jersey and analysed by the method of Barros. The limit of determination was 0.05 mg/kg for all analytes as shown by the following recoveries from fortified control samples: carbofuran 84 ± 9%, 3-keto-carbofuran 77 ± 3%, 3-hydroxy-carbofuran 78 ± 7%, 7-phenol 90%, 3-keto-7-phenol 111% and 3-hydroxy-7-phenol 94%.

Canadian GAP was not reported and US GAP does not reflect the conditions of either the Canadian or US trials. US GAP specifies two applications at 3.4 kg ai/ha, 1 at planting and a second 3-4 weeks later as a side-dress. The PHI is 21 days and the use is restricted to Arizona. The trials did not comply with GAP and two trials are inadequate to estimate a maximum residue level.

The LOD for all analytes was 0.05 mg/kg, with an estimated limit of detection of 0.01 mg/kg. The carbamate metabolites 3-keto-carbofuran and 3-hydroxy-carbofuran were not detected in any sample, and carbofuran was detected only in the California sample, at an estimated 0.01 mg/kg. The phenols were detected in all samples, but only the 7-phenol was quantifiable, in one Florida sample at 0.10 mg/kg. The maximum total residue was 0.21 mg/kg (Florida, in-furrow + side-dress). All others were ≤0.05 mg/kg.

Cucumbers. Twenty eight supervised field trials were conducted in the USA in Florida, Illinois, Virginia, Michigan, New York, Arkansas and California (Grigor and Tegriss, 1987a). Furadan 15G was applied as an in-furrow treatment at planting (1.1 kg ai/ha or 3.4 kg ai/ha), or Furadan 4F was applied to the soil as a band at planting. Cucumbers were collected at maturity, at PHIs ranging from 44 to 67 days, and analysed by the method of Schreier, with the method extended to the determination of the phenol metabolites by GC-MSD. Limits of quantification of 0.05 mg/kg for each analyte were established by the analysis of replicate fortified controls. The recoveries (mean of 8) were as follows: carbofuran 93 ± 15%, 3-keto-carbofuran, 104 ± 16%, 3-hydroxy-carbofuran, 74 ± 1%, 7-phenol, 86 ± 19, 3-keto-7-phenol, 101 ± 16%, and 3-hydroxy-7-phenol, 84 ± 15%. The limits of detection were estimated as 0.02 mg/kg for the carbamates and 0.01 mg/kg for the phenols. Total

residues as high as 0.6 mg/kg were found in one of two duplicate samples from Illinois. The results are shown in Table 49.

US GAP for cucumbers specifies application of the 10 or 15G formulation at planting, soil band incorporated at 2.2 kg ai/ha, or of the 4F formulation at 1.7 kg ai/ha. No PHI is specified. Although the trials were not conducted at the maximum GAP rates, they were at rates above and below the maximum with similar results. It can be concluded that residues from treatments according to GAP would be similar.

Thailand submitted information on trials according to GAP, but without data (Thai Industrial Standards Institute, 1997).

Table 49. Residues of carbofuran and carbamate and phenol metabolites in or on cucumbers following at planting treatment with Furadan 15G or Furadan 4F.

Location, Year, Variety	Form.	Rate, kg ai/ha	PHI, days	Residue, mg/kg					
				Carbo- furan	3-K-CF	3-OH- CF	7-phenol	3-K-7-P	3-OH-7- P
Florida, 1984/ Poinsetta 76	4F	1.1	67	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	4F	3.4	67	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	1.1	67	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	67	(0.023)	<0.02	<0.02	<0.02	<0.02	<0.02
Illinois, 1984/ SMR 58	4F	1.1	53	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
	4F	3.4	53	<0.02	<0.02	<0.02	<0.02	0.02	<0.02
	15G	1.1	53	<0.02	<0.02	<0.02	<0.02	0.027)	<0.02
	15G	3.4	53	0.17 (0.27, 0.071)	<0.02	<0.02	(0.024)	(0.24, 0.11)	(0.018)
Virginia, 1984/	4F	1.1	65	0.13	<0.02	<0.02	<0.02	0.07	<0.02
	4F	3.4	65	0.21	<0.02	0.02	<0.02	0.092	<0.02
	15G	1.1	65	(0.030)	<0.02	<0.02	<0.02	<0.02	<0.02
	15G	3.4	65	0.12	<0.02	<0.02	<0.02	0.054	<0.02
Michigan, 1984/ Chicago Pickling	4F	1.1	52	0.15	<0.02	<0.02	<0.02	0.17 (0.22, 0.12)	<0.02
	4F	3.4	52	0.12 (0.091, 0.16)	<0.02	<0.02	<0.02	0.13 (0.088, 0.18)	<0.02
	15G	1.1	52	0.1	<0.02	<0.02	<0.02	(0.044)	<0.02
	15G	3.4	52	0.12 (0.086, 0.15)	<0.02	<0.02	<0.02	0.14	<0.02

Location, Year, Variety	Form.	Rate, kg ai/ha	PHI, days	Residue, mg/kg					
				Carbo- furan	3-K-CF	3-OH- CF	7-phenol	3-K-7-P	3-OH-7- P
New York, 1984/ Victory	4F	1.1	66	<u>(0.028)</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
	4F	3.4	66	<u><0.02</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
	15G	1.1	66	<u><0.02</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
	15G	3.4	66	<u>0.060</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
Arkansas, 1984/ Green Star	4F	1.1	44	<u>0.05</u>	<0.02	<u><0.02</u>	<0.02	0.022)	<0.02
	4F	3.4	44	<u>0.12</u>	<0.02	<u><0.02</u>	<0.02	0.076	<0.02
	15G	1.1	44	<u><0.02</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
	15G	3.4	44	<u>0.098</u>	(0.026)	<u><0.02</u>	<0.02	0.081 (0.062, 0.10)	<0.02
California, 1984/ Poinsetta 76	4F	1	52	<u><0.02</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
	4F	3.4	52	<u><0.02</u>	0.002	<u><0.02</u>	<0.02	<0.02	<0.02
	15G	1	<0.02	<u><0.02</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02
	15G	3.4	<0.02	<u><0.02</u>	<0.02	<u><0.02</u>	<0.02	<0.02	<0.02

¹Furadan 4F was applied as an 18 cm band to the soil at planting. Concentration (kg ai/hl) not stated. Furadan 15G was applied to the furrow at planting

Cantaloupes. In supervised field trials reported from the USA in Florida, Illinois, Virginia, Michigan, New York, Arkansas and California, ± trials per state, Furadan 15G was applied in-furrow at planting (1.1 kg ai/ha or 3.4 kg ai/ha), or Furadan 4F was applied to the soil as ± band at planting (Grigor and Tegriss, 1987b). Samples were collected at maturity, with PHIs of 60 to 92 days, and analysed by the method of Schreier, the phenols by GC-MSD. Limits of determination of 0.05 mg/kg for each analyte were established by the analysis of fortified controls, with recoveries from ± replicates of carbofuran 85 ± 15%, 3-keto-carbofuran 94 ± 15%, 3-hydroxy-carbofuran 68 ± 7%, 7-phenol 71 ± 12%, 3-keto-7-phenol 102 ± 12%, and 3-hydroxy-7-phenol 80 ± 13%. Limits of detection were estimated as 0.02 mg/kg for the carbamates and 0.01 mg/kg for the phenols. The maximum total residue encountered was 0.12 mg/kg. No residues were detectable in or on the Florida samples (88-day PHI) and the residues were not quantifiable in or on any of the Arkansas (60-day PHI) or Virginia samples; one Arkansas sample (4F, 3.4 kg ai/ha) showed an estimated residue of 0.04 mg/kg carbofuran, estimated as ≤0.04 mg/kg, and all Virginia samples contained traces of carbofuran, estimated at 0.02 mg/kg. One of the duplicated samples from the 3.4 kg/ha treatments with 15G and 4F in Illinois contained quantifiable residues, 0.11 mg/kg and 0.081 mg/kg respectively. The major component was 3-hydroxy-carbofuran. In Michigan, treatment at 1.1 kg/ha yielded no quantifiable residues (<0.05 mg/kg) and treatment at 3.4 kg/ha yielded total residues of 0.10 mg/kg from the 4F formulation and 0.50 mg/kg in one of the duplicate from the 15G. The major components were 3-hydroxy-carbofuran and the 3-hydroxy-7-phenol. All New York samples contained carbofuran, ranging from 0.05 to 0.095 mg/kg. California samples from the 1.1 kg ai/ha applications (92 days PHI) also contained carbofuran, 0.05-0.11 mg/kg.

US GAP for cantaloupes is the same as for cucumbers: application of the 10 or 15G formulation at planting, soil band incorporated at 2.2 kg ai/ha, or of the 4F formulation at 1.7 kg ai/ha, no PHI specified. Although the trials were not at the maximum GAP rates, they included rates above and below the maximum, with similar results. It can be concluded that the residues from GAP treatments would also be similar.

Thailand submitted information on GAP trials, but without data on residues (Thai Industrial Standards Institute, 1997).

Summer squash. In trials in Florida, Illinois, Indiana, Michigan, New York, Arkansas and California (Grigor and Tegriss, 1987c) Furadan 15G was applied in-furrow, or Furadan 4F to the soil as \pm band, at planting at 1.1 or 3.4 kg ai/ha. Samples were collected at maturity, at PHIs ranging from 49 to 69 days, and analysed by the method of Schreier, with the same limits of determination (0.05 mg/kg) and detection (0.02 and 0.1 mg/kg) as for cantaloupes. Recoveries from fortified samples ($n \pm 8$) were as follows: carbofuran $86 \pm 16\%$, 3-keto-carbofuran $98 \pm 13\%$, 3-hydroxy-carbofuran $68 \pm 9\%$, 7-phenol 74 ± 6 , 3-keto-7-phenol $112 \pm 12\%$, and 3-hydroxy-7-phenol $85 \pm 11\%$.

One Florida sample contained unquantifiable residues (15G, 3.4 kg ai/ha, estimated 0.04 mg/kg carbofuran and 3-keto-7-phenol) and residues were undetectable in the others (52-day PHI). One New York sample (15G, 3.4 kg ai/ha) contained an estimated 0.02 mg/kg of the 3-keto-7-phenol, and the remaining samples had no detectable residues (69-day PHI). Residues were undetectable in all the California samples (49-day PHI). All except one of the samples from Michigan (PHI 61 days, Yellow Crookedneck variety) contained residues of carbofuran only, 0.05-0.09 mg/kg. The exception contained unquantifiable carbofuran and 3-keto-carbofuran. All samples from Indiana (PHI 53 days, President Elite variety) contained detectable residues of carbofuran (estimated as 0.02-0.05 mg/kg) and the samples from the 15G applications contained total residues from 0.10 to 0.20 mg/kg, with each of the three phenols contributing 0.02-0.03 mg/kg and 3-hydroxy-carbofuran 0.07 mg/kg from the 3.4 kg ai/ha treatment and 0.05 mg/kg from 1.1 kg ai/ha. All Illinois samples contained detectable and quantifiable residues, except from the 1.1 kg ai/ha 15G treatment. Residues of the carbamates were 0.06, 0.08 and 0.10 mg/kg. All samples from Arkansas (PHI 37 days, Golden Girl variety) contained residues in the range 0.094-0.26 mg/kg, consisting of carbofuran only, except from the 15G formulation applied at 3.4 kg ai/ha where about 25% of the residue (0.26 mg/kg total) was 3-hydroxy-carbofuran.

US GAP for summer squash is the same as for cucumbers and cantaloupes. Again the squash trials were at rates above and below the maximum GAP rate with similar results, and residues from GAP applications would also be similar.

Thailand again submitted GAP information but no residue data (Thai Industrial Standards Institute, 1997).

Coffee beans. Supervised field trials were reported from Brazil and the USA.

In two trials in 1994-5 (Brooks, 1996c) in major coffee-growing regions of Brazil (Sao Paulo and Minas Gerais, Catuai variety) Furadan 5G was applied twice at 1.5 g ai/bush. The first application was 30-60 days after flowering and the second approximately 6 months later, with a 29-day PHI. The granules were applied as a band round the bases of the coffee bushes. The coffee cherries were harvested at maturity and sun-dried. The green beans were depulped by a commercial process and analysed by the method of Barros. Limits of determination of 0.05 mg/kg were established for carbofuran and the carbamate and phenol metabolites by the determination of recoveries from fortified controls. The average recoveries (means of duplicates) were carbofuran 76%, 3-keto-

carbofuran 63%, 3-hydroxy-carbofuran 81%, 7-phenol 79%, 3-keto-7-phenol 99%, 3-hydroxy-7-phenol 73%. The recovery of 3-keto-carbofuran improved at a 0.5 mg/kg to 72%.

Duplicate samples were analysed from each of the two locations. At Sao Paulo the 3-hydroxy-7-phenol was found at 0.08 mg/kg in both samples, the 7-phenol and 3-keto-7-phenol were detected but <0.05 mg/kg, and carbofuran and 3-hydroxy-carbofuran were undetectable (<0.02 mg/kg). In the Minas Gerais samples, the 3-hydroxy-7-phenol was found at 0.16 and 0.13 mg/kg, 3-hydroxy-carbofuran and the 7-phenol were detected at estimated concentrations of 0.03 mg/kg each, and carbofuran was undetectable.

In Brazil, samples of beans from coffee bushes treated with a foliar spray of Furadan 350 SC at 2.1 or 4.2 g ai/bush, 200 l/ha, in 1994 (Sao Paulo University, 1994) were analysed for carbofuran by the method of Leppert. The PHI was 90 days. A recovery of $79 \pm 6\%$ was reported at 0.1 mg/kg fortification, but no details were provided. Carbofuran was below the limit of determination (<0.1 mg/kg).

GAP in Brazil specifies application of 0.35 g ai/tree of the 350 SC formulation or application of 0.5-3 g ai/tree of the 5G; the timing is not specified. GAP for foliar treatment was not reported.

Four supervised field trials were conducted on the islands of Kauai, Hawaii and Oahu in the USA (Brooks, 1996a). Two applications of Furadan 5G were made in each trial at 1.5 g ai/tree, the first after flowering and the second after an interval of 5-6 months. For each treatment, approximately 30 g of the granular formulation was applied to the soil near the base of each tree and "minially incorporated (less than 1 cm)". The PHIs were 28-29 days. The treated cherries were harvested at maturity, dried and shelled by the commercial wet method. The beans were analysed by the method of Barros. A limit of determination of 0.05 mg/kg for each analyte was established by the analysis of fortified controls but precision was generally poor. Recoveries were carbofuran (n = 9) $88 \pm 16\%$, 3-keto-carbofuran (n = 8) $105 \pm 13\%$, 3-hydroxy-carbofuran (n = 10) $88 \pm 17\%$, 7-phenol (n = 6) $64 \pm 12\%$, 3-keto-7-phenol (n = 6) $92 \pm 14\%$ and 3-hydroxy-7-phenol (n = 6) $72 \pm 13\%$. The results are shown in Table 50.

GAP in the USA specifies the application of a 10% G formulation at 1.7 g ai/tree twice each year to the base of coffee trees in Puerto Rico only. The application rate in the trials was within 10% of the GAP rate and the results could be used to estimate a maximum residue level.

Table 50. Residues of carbamates and phenols in or on green coffee beans from the application of Furadan 5G (2 x 1.5 g ai/tree) to the base of coffee plants, 28-29-day PHI.

Location/Year/ Variety	Residue, mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Kauai, 1995/ Yellow catuai	<0.02	(0.02)	(0.02)	0.16	(0.03)	0.33
Kauai, 1995/ Yellow catuai	<0.02	<0.02	0.25	0.24	0.10	0.64
Kauai, 1995/ Yellow catuai	<0.02	<0.02	0.79	0.32	0.20	1.08
Hawaii, 1995/ Guatamalan	<0.02	<0.02	0.08	(0.04)	(0.02)	0.22
Hawaii, 1995/ Guatamalan	<0.02	<0.02	0.12	(0.04)	(0.02)	0.28

Location/Year/ Variety	Residue, mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Oahu, 1995/ Guatamalan	<u>≤0.02</u>	<0.02	<u>≤0.02</u>	(0.01)	<0.052	(0.02)

Abbreviated compound names: see Figure 1

Head cabbage. The Netherlands reported two supervised field trials in which Curraterr 200 SC was applied to head cabbage in 1977 at rates of 0.04 and 0.25 g ai/plant, 0.40 g ai/l water. The mode of application was not described. The PHI was 69 days for the lower application rate and 155 days for the higher. Mature heads were analysed by the method of Mollhoff. All residues were <0.1 mg/kg, the stated limit of determination. GAP was not reported for The Netherlands or a neighbouring country.

Brussels sprouts. The Netherlands reported two supervised field trials with the application of Curraterr 200 EC to Brussels sprouts in 1977 at a rate of 0.025 g ai/plant, 0.25 g ai/l water. Mature sprouts were harvested 126-127 days after the treatment and analysed by the method of Mollhoff. All residues were <0.1 mg/kg, the stated limit of determination. GAP was not reported.

Cauliflower. The Netherlands reported five trials. Curraterr 200 SC was applied to cauliflower plants at rates of 0.025 or 0.038 g ai/plant, and 0.25 or 0.38 g ai/l water. The mode of treatment was not described. Mature crops were harvested 61, 69 or 71 days after the application and analysed by the method of Mollhoff. The residues of conjugates of 3-hydroxy-carbofuran were <0.1 mg/kg, the stated limit of determination. The combined residues of carbofuran and 3-hydroxy-carbofuran were 0.2, <0.1, 0.18, 0.22 and <0.1 mg/kg. GAP was not reported.

Kohlrabi. Germany submitted the results of two field trials with single applications of Curraterr-Granulat GR (50 g ai/kg) at a rate of 0.645 g/m. The rate per area and the type of application were not reported. It is implied that residues were determined as carbofuran plus 3-hydroxy-carbofuran and as 3-hydroxy-carbofuran conjugates. Limits of determination and sample chromatograms were not supplied. In the Oldenburger trial, the application was made 38 days after planting and the carbamate and conjugate residues were <0.1 mg/kg at a 27-day PHI and <0.05 mg/kg after 40 and 54 days. In the Braunschweig trial, the treatment was 52 days after planting and residues of carbofuran plus 3-hydroxy-carbofuran were 2.99 mg/kg at a PHI of 25 days, 0.28 mg/kg at 36 days and 0.17 mg/kg at 52 days. The residues of the conjugates at the same PHIs were 0.7, <0.05 and 0.11 mg/kg. GAP was not reported.

Grapes. Field trials were reported from Germany (Federal Biological Research Centre for Agriculture and Forestry, 1996), Mexico (Fullmer, 1977) and the USA (Pejovich, 1984).

In Germany Curraterr-Granulat GR (50 g ai/kg) was applied to mature grape vines in 1986 at four locations and grapes were harvested at intervals of 0-79 days. The growth stages at treatment were described as stages 27-33 and the treatment was 2 x 0.5 g ai/vine; the rate per area was not reported. The PHIs of the analytical samples are significantly shorter than the crop harvest interval, suggesting that immature crops may have been sampled. Samples were analysed by an undisclosed method and it was not stated whether carbofuran only or carbofuran plus certain metabolites were determined. The results are shown in Table 51. GAP was not reported for Germany or the European Union.

Table 51. Residues in or on grapes resulting from the treatment of vines with a granular formulation of carbofuran in Germany in 1986 at 2 x 0.5 g ai/vine.

Location	PHI, days	Residue ¹ , mg/kg
Weinsberg	0	0.04
	21	0.08
	42	0.12
Bernkastel-Kues	0	0.09
	22	<0.04
	79	<0.04
Neustadt-W	0	<0.04
	21	>0.04
	35	<0.04
	56	<0.04
Marienthal	70	0.11
	0	<0.04
	21	0.26
	35	0.09
	56	0.12
	70	0.1

¹The constituents of the residue were not reported

In Mexico, Furadan 5G was applied once to vineyard soil at 10 or 20 kg ai/ha in three trials. Samples of mature grapes were collected 123 days or 43 days after the treatment and analysed by the method of Schreier with limits of determination of 0.1 mg/kg for both carbofuran and 3-hydroxy-carbofuran. The recoveries from single spiked samples were 109% and 70% for carbofuran. The results are shown in Table 52.

GAP for Mexico was not reported, but GAP in the neighbouring USA (California only) requires 11.2 kg ai/ha of the 4F formulation, soil-incorporated after harvest with a PHI of 200 days. Pre-harvest drip irrigation of the same formulation may be made at 3.4 kg ai/ha with a 60-day PHI. The PHI of 123 days in one of the trials was within 40% of the US PHI and the 10 kg ai/ha rate was about 90% of the US rate.

Table 52. Residues of carbofuran in or on grapes from single applications of Furadan 5g to vineyard soil in Torreon, Mexico.

kg ai/ha	PHI, days	carbofuran, mg/kg	3-hydroxy-carbofuran, mg/kg
10	123	<0.1	<0.1
10	43	0.24	0.66

		(0.22; 0.27)	(0.52; 0.80)
20	43	1.1 (1.3; 0.93)	1.5 (1.5; 1.5)

In the USA, vineyards were treated 3 or 5 times by drip irrigation with Furadan 4F at 1.1 or 2.2 kg ai/ha/application at four locations in California in 1983-1984. Grapes taken at normal harvest were analysed by the method of Schreier. An MSD was used for the quantification of the carbamate residues and an NPD for the phenols. Limits of determination were demonstrated at 0.1 mg/kg by the analysis of fortified controls, with recoveries from triplicate analyses of carbofuran 78% ± 4.9%, 3-hydroxy-carbofuran 81% ± 6.2%, 7-phenol 89% ± 4.0%, 3-keto-7-phenol 101% ± 2.1%, and 3-hydroxy-7-phenol 63% ± 4.4%. Recoveries at 0.05 mg/kg were not acceptable and results at such concentrations would be semi-quantitative. The results are shown in Table 53. The maximum total carbamates residue was 0.15 mg/kg and the maximum combined residue of carbamates and phenols was 0.32 mg/kg.

Some trials corresponded to US GAP for post-harvest treatment of vineyards. The maximum total trial rate was 110% of the GAP rate and the PHIs were about 200 days. Some trials also complied with US GAP for pre-harvest treatments (3.4 kg ai/ha, 60-day PHI).

Table 53. Residues in or on grapes from the application of Furadan 4F by drip irrigation to vineyards in California, 1983-4.

Trial No., Location	Variety	Application			PHI, days	Residue, mg/kg				
		kg ai/ha	No.	Total kg ai/ha season		CF	3-OH- CF	7-phenol	3-K-7-P	3-OH- 7-P
RRA-038 Thermal	Perlette	4.5 2.2	1 3	11	218	<0.05	<0.05	<0.05	<0.05	<0.05
RRA-041 Thermal	Cardinal	4.5 2.2	1 3	11	209	<0.05	<0.05	<0.05	<0.05	<0.05
RRA-057 Thermal	Thompson Seedless	2.2	3	6.7	238	<0.5	<0.05	<0.05	<0.05	<0.05
RRA-100 Lost Hills	Perlette	2.2	3	6.7	266	<0.05	<0.05	<0.05	<0.05	<0.05
RRA-113 Madera	Thompson Seedless	2.2	3	6.7	256	<0.05	(0.03)	<0.05	<0.05	<0.05
RRA-241 Soledad	Merlot	2.2	6	13	294	<0.05	<0.05	(0.02)	(0.02)	<0.2
RRA-039 Thermal	Perlette	3.4	1	3.4	54	<0.05	0.1	(0.04)	(0.04)	(.07)
RRA-042 Thermal	Cardinal	1.1	3	3.4	54	<0.05	(0.06)	(0.03)	(.03)	(0.03)
RRA-046 Thermal	Perlette	1.1	3	3.4	60	<0.05	(0.06)	(0.04)	(0.04)	<0.1 (0.04)
RRA-048 Thermal	Cardinal	1.1	3	3.4	60	<0.05	<0.05 (0.03)	<0.05 (0.02)	<0.05 (0.02)	<0.1 (0.02)

Trial No., Location	Variety	Application			PHI, days	Residue, mg/kg				
		kg ai/ha	No.	Total kg ai/ha season		CF	3-OH- CF	7-phenol	3-K-7-P	3-OH- 7-P
RRA-059 Thermal	Thompson Seedless	1.1	3	3.4	59	<0.05	0.1	(0.06)	(0.05)	0.11
RRA-102 Lost Hills	Perlette	1.1	3	3.4	60	<0.05	0.12	(0.06)	<0.05 (0.04)	<0.1 (0.08)
RRA-111 Madera	Thompson Seedless	1.1	3	3.4	63	(0.02)	(0.08)	(0.04)	<0.05 (0.02)	<0.1 (0.02)
RRA-243 Soledad	Merlot	1.1	3	3.4	139	<0.05	(0.03)	(0.02)	(0.02)	<0.1 (0.02)
RRA-063 Thermal	Thompson Seedless	2.2 1.1	3 2	9	59	<0.05	(0.09)	(0.05)	(0.04)	(0.09)
RRA-105 Lost Hills	Perlette	2.2 1.1	3 2	9	60	<0.05	(0.08)	(0.04)	<0.05 (0.04)	<0.05 (0.05)
RRA-115 Madera	Thompson Seedless	2.2 1.1	3 2	9	63	(0.03)	0.10 (0.12; 0.07)	(0.04)	(0.02)	(0.02)
RRA-248 Soledad	Merlot	2.2 1.1	32	9	153	<0.05	(0.06)	(0.04)	(0.03)	<0.1 (0.05)

Abbreviated compound names: see Figure 1

Strawberries. Supervised field trials were conducted in France, The Netherlands, the UK and the USA.

In France (Anon., 1997) Curraterr 5 MG (microgranulate) was applied once or twice in 1982 to strawberry plants as a band. Ripe fruits were harvested and analysed by the method of Molhoff. Recoveries from fortified control samples were reported without details (3-hydroxy-carbofuran at 0.1 mg/kg, 90% and 99%, carbofuran at 0.05 mg/kg 36%, 41% and 47%; at 0.5 mg/kg 49%, 46% and 46%). A limit of determination was not adequately established for carbofuran and any results for this analyte are semi-quantitative. The results are shown in Table 54.

Table 54. Residues of carbofuran and 3-hydroxy-carbofuran in or on strawberries from the application of Curraterr 5 MG in France, 1982.

Variety	Application			PHI, days	Residue, mg/kg	
	Stage	Method	Kg ai/ha		Carbofuran ¹	3-OH-CF
Red Gauntlet	Before bloom	Band (0.90 m)	1.0	48	(<0.05)	<0.1
Tago	Before bloom Fruit-bearing plants	Band (0.90 m) Band over 2 rows	1.3 0.89	13	(0.4)	0.50 (0.46, 0.54)
Tago	Fruit-bearing plants	Band (0.90 m)	0.89	13	(0.06)	0.20
Tago	Before bloom Fruit-bearing plants	Band (0.90 m) Band over 2 rows	0.89	13	(0.20)	0.26

			0.89			
Tago	Before bloom. Fruit-bearing plants	Band (0.90 m). Band over 2 rows	0.44	13	(0.08)	0.37 (0.30; 0.43)
			0.89			
Red Gauntlet	Before bloom.	Band (0.90 m).	2.0	48	(0.06)	0.10

Abbreviated compound names: see Figure 1

¹Limit of determination was not established. Results are estimates only

In three field trials with post-harvest application of carbofuran to strawberry plants in the UK (Bagnall, 1986) Yaltox 5G was applied to three-year-old plants arranged in matted-bed rows with a Horstine Microband Applicator at 1.5 or 2.0 kg ai/ha. Mature strawberries were harvested the following season. The PHIs ranged from 309 to 316 days. The method of Mollhoff was utilized, but the analytes were derivatized to 2,4-dinitrophenyl ethers (Cook *et al.*, 1977). Limits of detection of 0.05 mg/kg for carbofuran and 0.1 mg/kg for 3-hydroxy-carbofuran were claimed, but no recovery data or chromatograms were supplied. The manufacturer's submission listed recovery information (85% for carbofuran, 74% for 3-hydroxy-carbofuran, fortification level not reported), but not in the field trial report. None of the strawberry samples contained detectable residues of carbofuran or 3-hydroxy-carbofuran.

The Netherlands provided a summary report of a field trial conducted in 1977. Curraterr 5G was applied to strawberry plants 2 weeks after transplanting at rates of 5 or 10 kg ai/ha. Mature fruit were harvested 272 days after treatment and analysed by the method of Molhoff. The results are shown in Table 55.

Table 55. Residues of carbofuran and 3-hydroxy-carbofuran in or on strawberries from the application of Curraterr 5G at 5 or 10 kg ai/ha, 272-day PHI. The Netherlands, 1978.

Application, kg ai/ha	carbofuran + 3-OH-CF, mg/kg	Conjugates of 3-OH-CF, mg/kg
5	0.08; 0.13; 0.11 (0.14 mean)	0.13; 0.15; 0.17 (mean 0.15)
10	0.37; 0.18; <0.1 (0.22 mean)	0.34; 0.19; 0.20 (mean 0.24)

Abbreviated compound names: see Figure 1

Three field trials with the post-harvest application of Furadan 4F to strawberry plants as a foliar spray were reported from the USA (Shevchuk, 1995a). The trials were in New York, Michigan and Virginia at 2.2 kg ai/ha, 12 g/l in New York and Virginia and 19 g/l in Michigan. Strawberries were harvested at maturity the following season and analysed by the method of Barros. The PHIs ranged from 225 days to 270 days. The limits of determination were established by the analysis of fortified controls in triplicate. The following recoveries were reported at 0.05 mg/kg: carbofuran 79 ± 3.0%, 3-keto-carbofuran 87 ± 10%, 3-hydroxy-carbofuran 79 ± 6.0%, 7-phenol 109 ± 5.0%, 3-keto-7-phenol 85 ± 1.0%, and 3-hydroxy-7-phenol 57 ± 3%. No analyte was detected (<0.02 mg/kg) in any sample.

GAP was reported only for the USA, where the soil may be treated post-harvest at 2.2 kg ai/ha with the 4F formulation after 1 October. The use is limited to Oregon, Michigan, Minnesota, Missouri, Tennessee and Washington. The US trials reported complied with this GAP.

Bananas. Supervised field trials were reported from Central America, South America and Spain.

Eight supervised field trials were conducted in the 1985-1986 growing season in Costa Rica, Honduras, Mexico, Ecuador and Guatemala (Leppert, 1986b). Furadan 10G (5G in Mexico) was applied twice at rates of 8.1-11 kg ai/ha, except in Costa Rica where the two applications were at 3.8 kg ai/ha, at intervals of about 6 months. Samples of bananas taken at 10, 30, 60, 90 and 120 days after the second application were analysed by the method of Schreier with an NPD for carbamates and an MSD for phenols. The pulp and peel were analysed separately for carbamates but whole fruit were analysed for phenols. The following recoveries were reported, without supporting data, from replicated samples spiked at 0.05 mg/kg, carbofuran (n = 6) $74 \pm 10\%$, 3-keto-carbofuran (n = 6) $86 \pm 21\%$, 3-hydroxy-carbofuran (n = 6) $86 \pm 21\%$, 7-phenol (n = 5) $79 \pm 10\%$, 3-keto-7-phenol (n = 5) $96 \pm 17\%$, 3-hydroxy-7-phenol (n = 5) $78 \pm 12\%$. The precision was unacceptable at 0.05 mg/kg for 3-keto- and 3-hydroxy-carbofuran, but acceptable accuracy and precision were demonstrated at 0.1 mg/kg: $79 \pm 10\%$ (n = 10) and $76 \pm 8\%$ (n = 11) respectively.

In all eight trials at all PHIs, the carbofuran and metabolite residues were below the limits of determination (<0.05 mg/kg for carbofuran, and the phenols, <0.1 mg/kg for 3-keto-carbofuran and 3-hydroxy-carbofuran). In the Mexico trial, 3-keto-carbofuran and 3-hydroxy-7-phenol were detected at estimated levels of 0.02 and 0.04 mg/kg respectively. In the Costa Rica trial, each of the phenols was detected at one or more PHIs; the estimated maximum total concentration was 0.04 mg/kg.

Summary results (Sao Paolo University, 1994e) were reported from a 1993 field trial in Brazil that involved single applications of Furadan 350SC to the soil round banana plants at rates of 4.2 and 8.4 g ai/plant. Samples were harvested 90 days after treatment and analysed for carbofuran by the method of Leppert. A limit of determination of 0.1 mg/kg was claimed. No residues were found. No details were provided in the very short summary.

Summary results were provided from a field trial in Spain in 1986 (Anon., 1986a). Curraterr 350SC was applied by irrigation to banana plants at 16 kg ai/ha in 50,000 l water/ha. Mature fruits were harvested 61 days after treatment and analysed for carbofuran and 3-hydroxy-carbofuran by the method of Molhoff. A limit of determination of 0.05 mg/kg was claimed. No residues were detected in either pulp or peel.

GAP was reported only for Spain, where 5G is applied at 0.6-0.75 kg ai/ha and 20F at 5.6 kg ai/ha, both with 60-day PHIs. The trial in Spain was at a much higher application rate, but the data could be used because no residues were detected (<0.02 mg/kg).

Thailand submitted GLP field trial information, but no report on residues (Thai Industrial Standards Institute, 1997).

Sugar cane. Four supervised field trials were conducted in Brazil (Sao Paulo University, 1994c) and in the USA (Shevchuk, 1992).

In Brazil separate plots were treated with 3 or 6 kg ai/ha of Furadan 50G (50 g ai/kg) or 1.75 or 3.5 kg ai/ha of Furadan 350SC (350 g ai/l), applied to the soil on the plant row about 5 months after planting. The cane was harvested 90 days after treatment. Samples were analysed by the method of Leppert. A limit of determination of 0.1 mg/kg was claimed, with a recovery of $86 \pm 3\%$ at unspecified fortification concentration(s). All samples contained <0.1 mg/kg.

In the USA, Furadan 4F was applied to sugar cane in six trials in three States. The first application was made in-furrow at planting at 1.1 kg ai/ha, 3.9-5.9 g ai/l. Two additional applications were made as aerial foliar sprays after joint formation at 0.84 kg ai/ha (9.0 g ai/l), a total of 2.8 kg ai/ha. The final application was 30 days before harvest. Samples were analysed by the method of Barros. A limit of determination of 0.03 mg/kg was demonstrated for each analyte by the analysis of 10 fortified controls with the following recoveries: carbofuran $91 \pm 7\%$, 3-hydroxy-carbofuran $85 \pm 10\%$, 3-keto-carbofuran $86 \pm 10\%$, 7-phenol $101 \pm 15\%$, 3-hydroxy-7-phenol $104 \pm 16\%$, and 3-keto-7-phenol $102 \pm 14\%$. The results are shown in Table 56.

Table 56 . Residues of carbofuran and metabolites in or on sugar cane from the application of Furadan 4F at planting (1.1 kg ai/ha) and foliar (2 x 0.84 kg ai/ha), 30-day PHI. USA, 1990-91.

State	Residue, $\mu\text{g/kg}$					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Florida ¹	0.05, 0.05	<0.01, <0.01	<0.01, <0.01	(0.01), (0.01)	<0.01, <0.01	<0.01, <0.01
Florida ¹	0.06, 0.06	<0.01 <0.01	<0.01, <0.01	(0.01), <0.01	<0.01 <0.01	<0.01, <0.01
Florida ¹	<u>0.05, 0.05</u>	<u><0.01, <0.01</u>	<u><0.01, <0.01</u>	<u>(0.01),</u> <u>(0.02)</u>	<u><0.01,</u> <u><0.01</u>	<u><0.01,</u> <u><0.01</u>
Florida ¹	0.04, 0.04	<0.01, <0.01	(0.02), <0.01	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01
Louisiana ²	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01
Louisiana ²	0.04, 0.06	<0.01, <0.01	<0.01, <0.01	(0.02), (0.02)	<0.01, <0.01	(0.01), (0.01)
Louisiana ³	(0.02), (0.02)	<0.01, <0.01	<0.01, <0.01	(0.02), (0.02)	<0.01, <0.01	<0.01 <0.01
Louisiana ³	(0.01), (0.02)	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	(0.01), (0.01)
Texas ⁴	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01
Texas ⁴	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	<0.01, <0.01	(0.01), <0.01

Abbreviated compound names: see Figure 1

¹Four trials at same location ²Two trials at same location ³Two trials at same location ⁴Two trials at separate locations

GAP for sugar cane in Brazil specifies 1.4-1.75 kg ai/ha (100-300 l/ha) of the 350 SC formulation applied in furrow with a planting stick or in bands or 1.5-3 kg/ha of the G formulation at 1.5-3 kg ai/ha applied in bands about the plants at second harvest. In US GAP for the 4F formulation no more than 2 foliar applications of 0.84 kg ai/ha each are made, with the use limited to the mainland, with a 17-day PHI. The 10G formulation may also be applied early in the season at 4.5 kg ai/ha with soil incorporation. None of the US trials complied with GAP, as an additional at-plant application was made at 1.1 kg ai/ha, but the residues were generally at or below the LOD.

Animal transfer studies

In a poultry feeding study in the USA in 1968 (Cook, 1968) three groups of 30 laying pullets were subdivided into three groups of 10 hens each for dosing or feeding at levels equivalent to 0.05, 0.5 or 5.0 ppm in the diet. The first group was fed carbofuran, the second group 3-hydroxy-carbofuran and the third alfalfa that had been treated with carbofuran. The dosing period was 10 consecutive weeks, during which eggs were collected periodically for analysis. At the end of the period, the pullets were killed and tissues taken for analysis. The period of frozen storage before extraction and analysis was not disclosed.

Eggs without shell, muscle, gizzard and liver were extracted with acetone. The extract was concentrated, hydrolysed with 0.25 N HCl and extracted with methylene chloride. The solvent was evaporated and the residue dissolved in acetonitrile and partitioned with hexane. The acetonitrile was evaporated and the residue dissolved in methylene chloride and cleaned up with Nuchar-Attaclay and silica gel. The final extract was analysed by GLC on a packed column with a microcoulometric nitrogen detector.

Fortified control samples were analysed to demonstrate the limits of determination, but the results are not acceptable because extensive corrections were made for column efficiency, typically 60%. The results of dosing with carbofuran and 3-hydroxy-carbofuran at 5 ppm are shown in Table 57. Residues were detected only in the gizzards of hens dosed with carbofuran and were below the LOD.

Table 57. Residues in poultry tissues and eggs from the oral dosing of hens with carbofuran or 3-hydroxy-carbofuran at the equivalent 5 ppm in the diet for 70 consecutive days and the recovery of analytes from control samples fortified at 0.05 mg/kg.

Sample	Compound	Analytical recovery, % ¹	carbofuran, mg/kg	3-hydroxy-carbofuran, mg/kg
Egg	carbofuran	51	<0.05 (14 and 56 days)	<0.05
		58 58		
	3-hydroxy-carbofuran	38	<0.05 (14 and 56 days)	<0.05
		35		
		33		
Muscle	carbofuran	63	<0.05	<0.05
	3-hydroxy-carbofuran	52	<0.05	<0.05
Liver	carbofuran	46	<0.05	<0.05
	3-hydroxy-carbofuran	55	<0.05	<0.05

Sample	Compound	Analytical recovery, % ¹	carbofuran, mg/kg	3-hydroxy-carbofuran, mg/kg
Gizzard	carbofuran	64	<0.05 (0.01)	<0.05 (0.01)
	3-hydroxy-carbofuran	60	<0.05	<0.05

¹Column efficiency corrections (81% for carbofuran, 59% for 3-hydroxy-carbofuran in eggs) were removed

In a feeding study in the USA with cows in 1994 (Chen, 1995a) carbosulfan (not carbofuran) was fed to lactating dairy cattle for 28 consecutive days at rates equivalent to 1, 3, 10 and 50 mg/kg in the diet. The study is fully described in the monograph on carbosulfan. In summary, carbofuran was not found in any milk, skim milk, cream or tissue samples at any of the 4 feeding concentrations, where the limit of detection was estimated as 0.005 mg/kg for milk and 0.010 mg/kg for tissues and cream. The metabolite 3-keto-carbofuran was detected only in one liver sample at 0.023 mg/kg from the 50 ppm group, and 3-hydroxy-carbofuran was detected in most milk samples from the 50 ppm group, at 0.007-0.030 mg/kg, and in one from the 10 ppm group (day 4, 0.007 mg/kg). Total carbamate residues reached a plateau at about 0.03 mg/kg from days 1 to 21. At the 50 ppm feeding level 3-hydroxy-carbofuran was detected in the kidneys (0.090, 0.13 mg/kg), liver (0.047, 0.060 mg/kg) and muscle (0.020, 0.030 mg/kg), but not in fat. In the 10 ppm group the 7-phenol (0.057 mg/kg) and 3-hydroxy-7-phenol (0.012 mg/kg) were found in the kidneys.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No data were submitted.

In processing

Sorghum. A processing study on sorghum was reported from the USA (Shevchuk and Singer, 1994). In Texas and Kansas, Furadan 4F was applied in-furrow at planting at 3.6 kg ai/ha and as two broadcast foliar sprays at 2.8 kg ai/ha. The total seasonal application of 9.2 kg ai/ha was four times the GAP limit. The PHI was 63 days in Texas and 87 days in Kansas. Grain samples from both locations were analysed by the method of Barros. The Texas grain sample contained no detectable residue of carbofuran (<0.01 mg/kg). Both 3-hydroxy-carbofuran and the 3-hydroxy-7-phenol were detected at about 0.01 mg/kg. No residues were detected in two of three Kansas grain samples; the 3-hydroxy-7-phenol was detected at about 0.01 mg/kg in one sample. Only the Texas grain was processed.

The grain was dried and cleaned by aspiration and screening, then abrasively milled thereby removing the bran and generating decorticated seeds and grits. Eighteen kg of raw grain yielded 17.1 kg dried grain and 16.8 kg cleaned seed, of which 6.8 kg was dry-milled to 4.2 kg decorticated grain, 1.1 kg bran and 1.3 kg grits. Limits of determination of 0.03 mg/kg were established for carbofuran and each metabolite by the analysis of fortified control samples of grain, decorticated seed, grits and bran. The minimum recovery was 65% of 0.03 mg/kg 7-phenol from grain and the maximum 109% of 0.03 mg/kg 3-hydroxy-carbofuran from bran. No residues were detected (<0.01 mg/kg) in decorticated seed or grits. The 7-phenol and 3-hydroxy-7-phenol were found in bran at estimated concentrations of 0.02 mg/kg each. [CLICK HERE to continue](#)

Sugar beet. In a sugar beet processing study in the USA (Stearns, 1986d) Furadan 15G at 4.5 kg ai/ha was applied once to the soil in an 18-cm band at planting in Colorado in 1985. The beets were harvested 176 days after application and samples were analysed by the method of Barros. Limits of determination of 0.05 mg/kg were demonstrated for carbofuran and each metabolite by the analysis of fortified controls. The recoveries at 0.05 mg/kg (single samples) were carbofuran 84%, 3-keto-carbofuran 84%, 3-hydroxy-carbofuran 92%, 7-phenol 56%, 3-keto-7-phenol 100%, and 3-hydroxy-7-phenol 100%. The recovery of the 7-phenol was improved at 0.10 mg/kg to 66%. No carbofuran or metabolite was detected in any sample, where the limit of detection was estimated to be 0.01 mg/kg for the carbamates and 0.02 mg/kg for the phenols. Representative chromatograms were provided.

The beets were commercially processed into cossettes, dehydrated pulp, molasses and sugar. The analysis of fortified controls showed a limit of determination of 0.05 mg/kg for each analyte in each type of sample. The minimum recovery was 62% (7-phenol in sugar) and the maximum 138% (3-keto-7-phenol in molasses). Carbamates and phenols were undetectable (<0.01 mg/kg carbamates, <0.02 mg/kg phenols) in cossettes and dehydrated pulp. The 3-keto-7-phenol was detected in molasses and sugar at an estimated concentration of 0.03 mg/kg. A concentration factor could not be calculated as the raw commodity did not have detectable residues.

Potatoes. In a processing study in the USA (Shevchuk, 1995b) Furadan 4F was applied to a plot in Washington State in 1993, one in-furrow at planting (6.7 kg ai/ha, 36 g/l) and three times as a broadcast spray post-emergence at 2.2 kg ai/ha, 12 g/l. The total application was 13.4 kg ai/ha, twice the GAP rate. The PHI was 21 days. The potatoes were processed in a laboratory-scale simulation of commercial processes into chips and granules (dehydrated potatoes). Samples of tubers, chips, granules, wet peel and dry peel were analysed by the method of Barros, with limits of determination and detection of 0.05 and 0.01 mg/kg for each analyte in each commodity. The limit of detection was corroborated by sample chromatograms. The results are shown in Table 58. The phenol metabolites were concentrated 1.7 times in potato chips and 5 times in both granules and dry peel. Processing factors could not be calculated for the carbamates, because none of the samples contained quantifiable levels.

Table 58. Carbofuran and metabolites in or on processed potato products after treatment of a Washington potato plot at 13.4 kg ai/ha.

Sample	Residue, mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K 7-P	3-OH-7-P
Tubers	<0.05	<0.05	<0.05	<0.05 (0.03)	<0.05	<0.05
Chips	<0.05	<0.05	<0.05	0.05	<0.05	<0.05 (0.01)
Granules	<0.05	<0.05	<0.05	0.14	(0.01)	<0.05 (0.02)
Wet peel	<0.05	<0.05	<0.05	<0.05 (0.02)	<0.05	<0.05
Dry peel	<0.05	<0.05	<0.05	0.12	<0.05 (0.02)	<0.05 (0.03)

Maize. A processing study on field corn (maize) was reported from the USA (Schreier, 1990b). A plot in Illinois was treated with Furadan 15G in-furrow at planting at 4.4 kg ai/ha. This was followed by one foliar treatment at the whorl stage with Furadan 15G at 3.4 kg ai/ha and two applications of

Furadan 4F at 3.4 kg ai/ha, a total application of 15 kg ai/ha (3 times the GAP rate); PHI was 42 days. The maize was processed by both wet and dry milling procedures and residues were determined in grain, grits, meal, flour, crude oil and refined (edible) oil after dry milling, and in starch, crude oil and refined oil after wet milling. Maize and the dry (non-oily) products were analysed for carbamates by the GLC method of Schreier with an NPD. The method of Leppert with GC-MS was used for the determination of carbamates and phenols in oils and of phenols in dry products. The limit of determination for each analyte in each commodity was shown to be 0.03 mg/kg by the analysis of fortified control samples and the limit of detection was estimated to be 0.01 mg/kg. The recoveries from fortified control samples and the results of the trial are shown in Table 59. No concentration of carbofuran or its metabolites was found in any processed fraction.

Table 59. Carbofuran and metabolites in or on maize and its milling fractions after treatment of a corn field with Furadan 15G and 4F at a total rate of 15 kg ai/ha, PHI 43 days. Illinois, USA.

Sample	Residue, mg/kg					
	Recovery from controls fortified at 0.03 mg/kg					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Grain	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	103	109	109	(0.01) 84	90	(0.03) 77
Dry Milling						
Medium Grits	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	99	93	94	69	67	(0.02) 62
Meal	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	79	104	83	96	98	(0.01) 85
Flour	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	115	117	81	60	61	(0.01) 60
Crude Oil	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	61	98	96	52	91	91
Refined oil	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	68	92	63	64	84	76
Wet Milling						
Starch	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	97	105	76	67	70	66
Crude Oil	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	75	99	93	71	96	101
Refined oil	<0.03	<0.03	<0.03	<0.03	<0.03	<0.03
	91	111	91	63	89	80

In a second processing study (wet milling only) in the USA (Brooks and Arabinick, 1995) Furadan 4F was applied to maize in Iowa in 1994 at planting in-furrow at 4.4 kg ai/ha (81 g/l), at the whorl stage at 3.4 kg ai/ha (32 g/l), and as two broadcast foliar applications, each at 3.4 kg ai/ha (20 g/l). The total application was 15 kg ai/ha (3 times the GAP rate) and the PHI was 67 days. The

residues in mature corn and starch were determined by the method of Barros, with limits of determination established at 0.03 mg/kg by the analysis of fortified controls. Recoveries from grain ranged from 72% (0.03 mg/kg 3-hydroxy-7-phenol) to 105% (0.03 mg/kg 3-hydroxy-carbofuran), and from starch from 68% (0.03 mg/kg 7-phenol to 123%; 0.03 mg/kg 3-hydroxy-7-phenol). The grain was found to contain detectable amounts of 3-hydroxy-carbofuran (estimated at 0.01 mg/kg) and quantifiable amounts of 3-hydroxy-7-phenol, 0.03 mg/kg. The starch contained no detectable residues, with the limit of detection estimated to be 0.01 mg/kg for each analyte. Carbamate residues were reduced in processing maize to starch, but a factor could not be determined.

Rice. A processing study was conducted in the USA (Shevchuk, 1995a). Rice in an Arkansas field was treated with Furadan 3G at 3.4 kg ai/ha (5 times the GAP rate) on the first day of permanent flooding, or about one month after planting. Mature rice was harvested 110 days after the treatment and processed by a batch procedure that closely followed standard commercial practice into polished rice, hulls and bran. The treated rough rice (110 kg) was dried (105 kg), aspirated and screened, yielding 23 kg of rough rice. This was dehulled and separated into hulls (3.9 kg), brown rice (18 kg) and unhulled rice (0.41 kg). The brown rice was debranned, yielding bran (2.4 kg) and white milled rice (16 kg). The rough rice and the processed fractions were analysed by the method of Barros. Duplicate control samples fortified at 0.03 mg/kg were analysed to establish the limits of determination. The lowest recovery was 62% for carbofuran in hulls and the highest 124% for 3-hydroxy-7-phenol in grain. Treated grain contained detectable amounts of 3-hydroxy-carbofuran (0.02 mg/kg) and the 3-hydroxy-7-phenol (0.02 mg/kg) and a quantifiable amount of the 7-phenol (0.05 mg/kg). Polished rice contained no detectable residues. Bran contained detectable levels of 3-keto-7-phenol (0.02 mg/kg, 2-fold concentration) and quantifiable levels of 7-phenol (0.42 mg/kg, 8-fold concentration) and 3-hydroxy-7-phenol (0.04 mg/kg, 2-fold concentration). Hulls contained quantifiable concentrations of carbofuran (0.02 mg/kg, 2-fold concentration), 3-keto-carbofuran (0.02 mg/kg, 2-fold concentration), 3-hydroxy-carbofuran (0.05 mg/kg, 4-fold concentration), 7-phenol (0.10 mg/kg, 2-fold concentration), 3-keto-7-phenol (0.03 mg/kg, 3-fold concentration), 3-hydroxy-7-phenol (0.04 mg/kg, 2-fold concentration). Processing factors could not be determined for the carbamates.

Sunflowers. In two processing studies in the USA (Tilka, 1981,1982) plots in North Dakota (Interstate 4 Variety, oil seed type) and Minnesota (Royal Hybrid Variety, confectionary type) were treated at planting with Furadan 10 G at 2.2 kg ai/ha and the plants were treated four times with foliar aerial applications of Furadan 4F at 0.56 kg ai/ha. A total application of 4.5 kg ai/ha. The North Dakota crop was harvested 26 days after the last application and the Minnesota crop after 50 days. The seeds were processed by simulated commercial procedures. Confectionary seeds were cracked and separated into hulls and kernels. Oil seed kernels were extracted to obtain crude oil and extracted meal. The crude oil was refined, bleached and deodorized to yield edible oil and soapstock. The seed and processed fractions were analysed for carbofuran and 3-hydroxy-carbofuran by the method of Schreier with the clean-up procedures of Leppert. Control samples were fortified at 0.05 mg/kg (0.1 mg/kg for confectionary seed) and analysed by the trial method. The limit of detection was estimated as 0.01 mg/kg for carbofuran and 3-hydroxy-carbofuran in all samples except soapstock, where it was 0.02 mg/kg. The results are shown in Table 60. The residue levels increased slightly in hulls and meal only.

Table 60. Carbofuran and 3-hydroxy-carbofuran in processed fractions of sunflower seed, and recoveries from fortified control samples.

Fraction	Residue, mg/kg, and [processing factor]		Recovery, %	
	Carbofuran	3-OH -CF	Carbofuran	3-OH -CF

	Residue, mg/kg, and [processing factor]		Recovery, %	
Oil seed	0.10	0.05	76 (0.05 mg/kg)	68 (0.05 mg/kg)
Edible oil	<0.05 (0.01) [0.1]	<0.05 [1]	88 (0.05 mg/kg)	100 (0.05 mg/kg)
Hulls	0.12 [1.2]	0.05 [1]	80 (0.05 mg/kg)	72 (0.05 mg/kg)
Soapstock	<0.05 [0.2]	<0.05 [1]	58 (0.05 mg/kg)	100 (0.05 mg/kg)
Extracted meal	0.10 [1]	0.09 [1.8]	68 (0.05 mg/kg)	76 (0.05 mg/kg)
Confectionary seed	<0.1 [0.06]	<0.1 [0.02]	93 (0.10 mg/kg)	67 (0.10 mg/kg)
Hulls	0.07 [1.2]	<0.05	-	-

Abbreviated compound names: see Figure 1

Cotton. In a processing study in the USA. (Shevchuk, 1994b) Texas cotton was treated with Furadan 4F in two broadcast foliar applications, each at 1.4 kg ai/ha (19 g/l), a total of 5 times the GAP rate. The cotton was harvested at the bloom and bolls growth stage, 27 days after the second treatment. Ginned and delinted cotton seed, hulls, meal and crude oil were analysed for carbofuran and its carbamate and phenol metabolites by the method of Barros. Soapstock was analysed for the phenol metabolites only. The method was validated at 0.03 mg/kg for each analyte in each sample. The recoveries from duplicate controls fortified at 0.03 mg/kg and the results of the trial are shown in Table 61. Sample chromatograms were included for the phenol but not for the carbamate determinations. There was no concentration of residues except in soapstock, where the 7-phenol was concentrated by a factor of about 7 from an estimated 0.01 mg/kg.

In the processing operation 48.2 kg cotton yielded 15.8 kg kernels and 6.45 kg hulls and the kernels yielded 3.7 kg crude oil and 11.4 kg meal.

Table 61. Carbofuran and metabolites in processed fractions of cotton seed after the foliar application of Furadan 4F (2 x 1.4 kg ai/ha, 27-day PHI), and recoveries from fortified control samples.

Sample	Residue, mg/kg					
	[Processing factor]					
	Recovery, %					
	carbofuran	3-K-CF	3-OH -CF	7-Phenol	3-K 7-P	3-OH-7-P
Ginned cotton seed	0.06	<0.03	0.19	<0.03 (0.01)	<0.03	<0.03
	73, 75	81, 102	56, 68	106, 106	94, 97	64, 66
Delinted cotton seed	0.03 [0.5]	<0.03	<0.03 (0.01) [0.05]	<0.03	<0.03	<0.03
	84, 88	111, 112	63, 72	90, 95	103, 113	73, 81
Hulls	<0.03 (0.02) [0.4]	<0.03	<0.03 (0.01) [0.05]	<0.03	<0.03	<0.03
	72, 82	69, 75	70, 71	77, 93	96, 101	61, 71

Meal	<0.03 (0.02) [0.4] 90, 99	<0.03 85, 86	<0.03 (0.01) [0.05] 68, 71	<0.03 84, 91	<0.03 122, 129	<0.03 122, 128
Crude oil	<0.03 (0.02) [0.4] 90, 101	<0.03 103, 112	<0.03 [0.05] 70, 75	<0.03 (0.01) 60, 61	<0.03 74, 79	<0.03 84, 85
Soapstock	-	-	-	0.07 [7] 57, 64	<0.03 84, 94	<0.03 78, 92

Abbreviated compound names: see Figure 1

Sugar cane. A processing study was carried out in El Salvador (Stearns, 1986c). In the 1985-1986 growing season, ratoon sugar cane was treated twice with Furadan 10G at 2.5 kg ai/ha, as a banded treatment after the 1985 harvest and as a broadcast application about 6 months later, the total application of 5 kg ai/ha being 1.8 times the GAP rate. Mature sugar cane harvested 169 days after the second treatment was processed into brown sugar and molasses, but the process was not described and the molasses were not defined as either blackstrap or edible molasses. Brown sugar was also not defined, but was presumably unrefined sugar, not the commercially available brown sugar. The samples were analysed by the method of Schreier, with an MSD for the phenols and an NPD for the carbamates. Limits of determination were established for the processed fractions but not for the raw cane. Acceptable recoveries were reported at 0.05 mg/kg fortification for the carbamates from brown sugar and molasses and the phenols from molasses and at 0.10 mg/kg for the phenols from brown sugar. Limited chromatographic information was provided. The analyses showed no residues of carbofuran, 3-keto-carbofuran or 3-hydroxy-carbofuran in the cane, molasses or brown sugar. The 7-phenol was reported as 0.05 mg/kg in cane, 0.06 mg/kg in molasses (1.2-fold concentration) and 0.12 mg/kg in brown sugar (2.4-fold concentration). The residues of the 3-keto-7-phenol were 0.03 mg/kg in cane, 0.06 mg/kg in molasses (2-fold concentration) and 0.08 mg/kg in brown sugar (2.7-fold concentration), and of the 3-hydroxy-7-phenol 0.05 mg/kg in cane, 0.08 mg/kg (1.6-fold concentration) in both molasses and brown sugar. A limit of detection of 0.02 mg/kg was claimed for all analytes. Processing factors for the carbamate residues could not be estimated as neither the raw nor the processed commodities contained carbamates.

Coffee. In a processing study reported from Minas Gerais, Brazil (Brooks, 1996b) Furadan 5G was applied twice at 3.0 g ai/bush to the soil round coffee plants (Catuai, 1400 cova/ha), the total of 6.0 g ai/bush being twice the GAP rate. The first application (in 1994) was 30 days after flowering and the second (1995) about 6 months later. The PHI was 30 days. The green coffee beans were processed into instant coffee and ground roasted coffee in a laboratory scale operation designed to reflect commercial processing. Green beans (13.8 kg) were roasted (177-221 °C hot air for 6 minutes) and a proportion was ground. The remaining beans were brewed and the extract freeze-dried. The spent grounds were press-brewed and the brew added to the extract. The green beans, ground coffee and instant coffee were analysed by the method of Barros. Limits of determination of 0.05 mg/kg for carbofuran and its metabolites on coffee beans only were demonstrated by the analysis of fortified control beans in triplicate. The ranges of recovery were reported as carbofuran 72-94%; 3-keto-carbofuran 64-97%, 3-hydroxy-carbofuran 92-96%, 7-phenol 92-128%, 3-keto-7-phenol 106-138%, and 3-hydroxy-7-phenol 52-82%. The limit of detection was claimed to be 0.01 mg/kg for each analyte. Green bean coffee had a measurable residue of 3-hydroxy-carbofuran, 0.22 mg/kg. Neither instant coffee nor roasted beans contained carbamates (<0.01 mg/kg, processing factor 0.05). Both

the green beans and the processed commodities contained all the three phenol metabolites with higher levels in the processed commodities. The phenolic residues are shown in Table 62.

Table 62. Residues of phenol metabolites in or on green beans, roasted coffee and instant coffee from the application of Furadan 5G to the soil around coffee bushes in Brazil, 2 x 3 g ai/bush, 30-day PHI.

Commodity	7-phenol		3-keto-7-phenol		3-hydroxy-7-phenol	
	Residue, mg/kg	Processing factor	Residue, mg/kg	Processing factor	Residue, mg/kg	Processing factor
Green beans	0.14	-	0.07	-	0.28	-
Ground roast	0.17	1.2	0.08	1.1	0.45	1.6
Instant	0.47	3.4	0.21	3.0	0.43	1.5

Pimento peppers. In a processing study in the USA (Anon., 1971). Furadan 10 G was applied to pimento pepper plots in Delaware in two side-dress treatments at 2.2 and 3.4 kg ai/ha. Pimentos were harvested at maturity and pickled by an undefined method. Residues of carbofuran and 3-hydroxy-carbofuran were determined by the method of Schreier, using a gas chromatograph equipped with a Coulson nitrogen detection system. The limits of determination were established by the analysis of fortified peppers and pickled peppers. At 0.05 mg/kg the recoveries from peppers were 114 and 132% for carbofuran and 94 and 70% for 3-hydroxy-carbofuran. At 0.20 mg/kg the recoveries from fortified pickled peppers (6 replicates) were $89 \pm 9.5\%$ for carbofuran and $62 \pm 10\%$ for 3-hydroxy-carbofuran. Finite residues were found on the raw peppers which were reduced by pickling. The results are shown in Table 63.

Table 63. Residues of carbofuran and 3-hydroxy-carbofuran in or on pimento peppers and pickled peppers from the application of Furadan 10 G as a banded treatment (2.2 + 3.4 kg ai/ha) in 1971 in Delaware, USA.

PHI, days	Residue, mg/kg			
	Raw peppers		Pickled peppers	
	carbofuran	3-hydroxy-carbofuran	carbofuran	3-hydroxy-carbofuran
21	0.35	0.13	0.19	0.08
	0.35	0.23	0.19	0.08
39	0.10	0.15	<0.2	<0.2

Grapes. Furadan 4F was applied as a broadcast treatment at the GAP rate of 11 kg ai/ha (29 g/l) to grape plants (Pinot Blanc) in California in 1985 (Stearns, 1986b). Grapes were harvested at maturity after a PHI of 198 days and processed into juice and wet and dry pomace. No information was supplied on the processing. The samples were analysed by the method of Schreier, carbamates being determined with an NPD and phenols with an MSD. Limits of determination were demonstrated by the analysis of duplicate control samples of grapes fortified at 0.10 mg/kg and juice and dry pomace at 0.05 mg/kg with carbofuran and each of the carbamate and phenol metabolites. Levels of detection

of 0.01 and 0.02 mg/kg were claimed for the carbamates and phenols respectively. The results are shown in Table 64. Residues were not concentrated in the juice but were concentrated in dry pomace (3-hydroxy-carbofuran 2.8-fold and total phenols 3.9-fold).

Table 64. Residues of carbofuran and its metabolites in grapes, juice and pomace from the application of Furadan 4F, 11 kg ai/ha, 198-day PHI and recoveries from fortified control samples.

Commodity	Residues, mg/kg					
	Recoveries, % ¹					
	Carbofuran	3-K-CF	3-OH-CF	7-Phenol	3-K-7-P	3-OH-7-P
Grapes	<0.1	<0.1	0.20	<0.1	0.1	0.14
	83 (76, 90)	98 (78, 117)	98 (79, 117)	(0.07) 87	93	0
Juice	<0.05	<0.05	0.18	<0.05	0.07	0.12
	84 (84, 84)	90 (86, 94)	98 (102, 94)	(0.04) 89 (96, 82)	96 (104, 87)	89 (97, 81)
Dry Pomace	<0.05	<0.05	0.56	0.32	0.44	0.44
	(0.01) 67 (62, 72)	(0.02) 68 (66, 70)	67 (62, 72)	84 (82, 85)	104 (103, 105)	75 (74, 76)

¹Grapes fortified at 0.1 mg/kg, juice and pomace at 0.05 mg/kg with each analyte

Residues in the edible portion of food commodities

No data were submitted.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

A farm gate study was submitted from Korea (Chon Chae-gu, 1996) in which seven domestic commodities were analysed for carbofuran in the period April 1995-January 1996. A total of 210 samples of rice, carrots, maize, green onions, potatoes, peanuts and garlic were collected from farms (only brown rice) or markets (all commodities except rice) near the sites of production in various growing regions of the country. Residues were analysed by the method of Leppert, but the ethoxylation step was omitted. A 10 m RSL-300TM or a 25 m methyl silicone capillary column was used with a nitrogen-phosphorus detector. Calibration was by external standards and only carbofuran was determined, although several sample chromatograms showed a peak labelled 3-hydroxy-carbofuran. Results are shown in Table 65.

Table 65. Monitoring of carbofuran residues in seven domestic commodities in Korea, 1995-1996.

Commodity	No. of samples	No. of detections ¹	Detection frequency, %	Carbofuran, mg/kg	Recoveries	
					Fortification, mg/kg	%
Brown rice	60	1	1.7	<0.5 (0.06)	0.5	106, 107, 108
Garlic	40	2	5.0	<0.1 (0.07, 0.13)	0.1	95.5, 91.3, 95.8, 96.1, 97.6, 94.9
Peanuts	20	0	0	<0.1	0.1	98.4, 92.4, 96.2
Potato	10	0	0	<0.1	0.1	100.1, 92.6, 97.3
Green onions	40	0	0	<0.25	0.25	81.9, 83.5, 83.4
Carrots	20	2	10	<0.25 (0.015, 0.015)	0.25	92.0, 92.8, 91.2
Corn (maize)	20	0	0	<0.25	0.25	95.0, 94.5, 94.0

¹ Limit of detection estimated at 0.05 mg/kg for rice, 0.0125 mg/kg for carrots, maize and green onions and 0.02 mg/kg for garlic, peanuts and potatoes. These limits were based on injections of standards and do not reflect the effect of the matrix

NATIONAL MAXIMUM RESIDUE LIMITS

Maximum residue limits, which have been established in 31 countries and the EU are shown below.

Country	Commodity	MRL, mg/kg	Remark
Argentina	Bean	0.1	
	Eggs	0.05	
	Fat	0.05	
	Maize grain	0.1	
	Meat	0.05	
	Meat by-products	0.05	
	Milk	0.05	
	Potato	0.5	
	Sorghum grain	0.1	
	Sweet corn	0.1	
Australia	Tomato	0.1	
			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Animal feed	2	
	Banana	0.1	
	Eggs	0.05*	
	Meat	0.05*	
	Meat by-products	0.05*	
	Milk	0.05*	
	Poultry meat	0.05*	
	Rice	0.2	
Austria	Sugar cane	0.1*	
	Wheat	0.2	
			Carbofuran, 3-hydroxy-carbofuran and its conjugates, expressed as carbofuran
	Beet, Sugar	0.2	

Country	Commodity	MRL, mg/kg	Remark
	Coffee		
	Grape	0.2	
	Maize	0.2	
	Meat	0.05	
	Milk	0.05	
	Sunflower seed	0.1	
	Potato	0.5	
	Turnips	1	
Belgium			Carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Plant commodities	0	<0.1 mg/kg
Brazil	Coffee	0.1	
	Cotton seed	0.1	
	Peanut	0.1	
	Rice	0.2	
Canada			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Alfalfa	0.1	Negligible tolerance
	Banana	0.1	Negligible tolerance
	Barley	0.1	Negligible tolerance
	Beet, Sugar	0.1	Negligible tolerance
	Clover	0.1	Negligible tolerance
	Coffee	0.1	Negligible tolerance
	Cucumber	0.1	Negligible tolerance
	Eggs	0.1	Negligible tolerance
	Grape	0.1	Negligible tolerance
	Maize	0.1	Negligible tolerance
	Meat	0.1	Negligible tolerance
	Melon	0.1	Negligible tolerance
	Milk	0.1	Negligible tolerance
	Oats	0.1	Negligible tolerance
	Peanut	0.1	Negligible tolerance
	Pepper, Sweet	0.5	
	Potato	0.5	
	Pumpkin	0.1	Negligible tolerance
	Rape seed	0.1	Negligible tolerance
	Rice	0.1	Negligible tolerance
	Rutabaga	0.5	
	Strawberry	0.4	
	Sunflower	0.1	Negligible tolerance
	Tomato	0.1	Negligible tolerance
	Wheat	0.1	Negligible tolerance
Chile			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Animal fat	0.05*	
	Barley	0.1*	
	Beat, sugar	0.1*	
	Mammalian, meat	0.05*	
	Milk	0.05*	
	Oilseed	0.1*	
	Potato	0.5	
	Rice, husked	0.2	
	Tomato	0.1*	
	Wheat	0.1*	
Cyprus	Banana	0.1	
	Beets, sugar	0.1	
	Cereals	0.1	

Country	Commodity	MRL, mg/kg	Remark
	Meat	0.05	
	Milk	0.05	
	Potato	0.05	
	Rice	0.02	
	Strawberry	0.1	
	Tomato	0.1	
Denmark			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Banana	0.1	
	Potato	0.5	
	Turnips, swedes	0.1	
European Union			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Banana	0.1*	T
	Cereals	0.1*	
	Cotton seed	0.1*	T
	Cucurbits	0.1*	
	Egg products	0.1*	
	Eggs	0.1*	T
	Grape	0.1*	T
	Meat	0.1*	T
	Meat by-products	0.1*	
	Meat, preparations of	0.1*	
	Melon	0.1*	T
	Milk	0.1*	
	Milk products	0.1*	
	Oats	0.1*	T
	Peanut	0.1*	T
	Potato	0.1*	T
	Rape seed	0.1*	T
	Rice	0.1*	T
	Rubus species (cane fruit)	0.1*	
	Rutabaga	0.01	T
	Soya	0.1*	T
	Strawberry	0.1*	T
	Sunflower seed	0.1*	T
	Sweet corn	0.1*	T
	Turnip, edible	0.01	T
Finland			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Potato	0.5	
France			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Maize forage	0.5	T
	Maize grain	0.1	
	Rape	0.5	
	Soya	0.2	
	Strawberry	0.5	
	Sunflower	0.5	
	Sweet corn	0.5	
	Sweet corn, forage	0.5	T
Germany			Carbofuran, 3-hydroxy-carbofuran and its conjugates, expressed as carbofuran
	Animal fat	0.05	
	Beet, Sugar	0.2	
	Egg products	0.05	
	Eggs	0.05	

Country	Commodity	MRL, mg/kg	Remark
	Meat	0.05	
	Meat, preparations of	0.05	
	Milk	0.05	
	Milk products	0.05	
	Other plant commodities	0.1	
	Potato	0.5	
Hungary	Other plant commodities	0.1	
India			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Cereal grains	0.1	
	Fruit	0.1	
	Oilseed	0.1	
	Sugar cane	0.1	
	Vegetables	0.1	
	Meat	0.1	Fat basis
	Milk	0.05	Fat basis
	Milk products	0.05	Fat basis
	Poultry	0.1	
Italy	Beet, Sugar	0.1	
	Maize	0.1	
	Potato	0.1	
Kenya	Alfalfa		T
	Alfalfa hay	20	T
	Banana	0.1	T
	Barley	0.1	T
	Beet, Sugar, leaf	0.2	T
	Beet, Sugar, root	0.1	T
	Cattle fat	0.05*	T
	Cattle meat	0.05*	T
	Cattle meat by-products	0.05*	T
	Coffee	0.1	T
	Goat fat	0.05*	T
	Goat meat	0.05*	T
	Goat meat by-products	0.05*	T
	Horse fat	0.05*	T
	Horse meat	0.05*	T
	Horse meat by-products	0.05*	T
	Maize	0.1	T
	Maize, forage		T
	Milk	0.05*	T
	Oats	0.1	T
	Oilseed	0.1	T
	Peanut kernel	0.1	T
	Pig fat	0.05*	T
	Pig meat	0.05*	T
	Pig meat by-products	0.05*	T
	Potato	0.5	T
	Rice, husked	0.2	T
	Sheep fat	0.05*	T
	Sheep meat	0.05*	T
	Sheep meat by-products	0.05*	T
	Sorghum	0.1	T
	Soya	0.2	T
	Strawberry	0.1	T
	Sugar cane	0.1	T
	Sweet corn kernels	0.1	T
	Tomato	0.1	T

Country	Commodity	MRL, mg/kg	Remark
	Wheat	0.1	T
Luxembourg	Maize	0.1	Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
Malaysia	Banana	0.1	
	Grape	0.4	
	Strawberry	0.5	
Mexico	Alfalfa	10	
	Alfalfa hay	40	
	Banana	0.1	
	Barley	0.2	
	Coffee	0.1	
	Cucumber	0.4	
	Grape	0.4	
	Maize	0.1	
	Melon	0.4	
	Oats	0.2	
	Peanut	4	
	Pepper, Cayenne	1	
	Pepper, Sweet	1	
	Potato	2	
	Rice	0.2	
	Sorghum	0.1	
	Soya	1	
	Strawberry	0.5	
	Sugar cane	0.1	
	Wheat	0.2	
Netherlands			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Potato	0.5	
	Rice	0.2	
	Soya	0.2	
	Strawberry	0.2	
	Other plant commodities	0.1*	
Paraguay	Rice	0.2	
	Tomato	0.1	
Portugal	Potato	0.5	Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
South Africa			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Cruciferae	0.5	
	Maize	0.1	
	Maize forage	0.2	
	Potato	0.05	
	Sorghum	0.1	
	Sugar cane	0.1	
	Sunflower seed	0.1	
	Wheat	0.1	
South Korea	Banana	0.1	
	Barley	0.1	
	Coffee	0.1	
	Cotton seed	0.1	
	Cucumber	0.5	
	Grape	0.5	
	Maize	0.1	
	Oats	0.1	
	Peanut	0.5	
	Potato	0.5	

Country	Commodity	MRL, mg/kg	Remark
	Pumpkin	0.5	
	Rice	0.2	
	Sorghum grain	0.1	
	Soya	0.2	
	Strawberry	0.1	
	Sunflower seed	0.1	
	Tomato	0.1	
	Wheat	0.1	
Spain			Sum of carbofuran, carbosulfan and 3-hydroxy-carbofuran, expressed as carbofuran
	Sweet corn	0.1*	
	Cotton seed	0.1*	
	Cucurbits with edible peel	0.1*	
	Cucurbits with inedible peel	0.1*	
	Forage crops and straw	0.1*	
	Grape	0.1*	
	Maize forage	2	
	Oats	0.1*	
	Oil seed	0.1*	
	Other pulses	0.1*	
	Peanut	0.1*	
	Potato	0.2	
	Rape seed	0.1*	
	Rice	0.1*	
	Rubus species (cane fruit)	0.1*	
	Rutabaga	0.1*	
	Solanaceae (peppers)	0.1*	
	Sorghum forage	2	
	Soya	0.1*	
	Stimulant plants (coffee)	0.1*	
	Strawberry	0.1*	
	Sugar plants	0.1*	
	Sunflower seed	0.1*	
	Turnip, edible	0.1*	
Sri Lanka	Banana	0.2	
	Gourd	1	
Sweden			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Fruit	0.1	
	Potato	0.5	
	Other vegetables	0.1	
Switzerland	Beet, Sugar	0.05*	
	Maize	0.05*	
UK			Sum of carbofuran and 3-hydroxy-carbofuran, expressed as carbofuran
	Banana	0.1*	
	Barley	0.1*	
	Beet, Sugar	0.1*	
	Cereals exc. rice	0.1*	
	Cucumber	0.1*	
	Cucurbits	0.1*	
	Eggs	0.1*	
	Fat	0.1*	
	Grape	0.1*	
	Maize	0.1*	
	Meat	0.1*	
	Meat, preparations of	0.1*	

Country	Commodity	MRL, mg/kg	Remark
	Milk	0.1*	
	Milk products	0.1*	
	Oil seed	0.1*	
	Pepper, Sweet	0.1*	
	Rye	0.1*	
	Squash	0.1*	
	Tomato	0.1*	
	Wheat	0.1*	
	Wine grape	0.1*	
Uruguay	Potato	0.5	
	Rice	0.2	
	Tomato	0.1	
USA			Carbofuran, 3-hydroxy-carbofuran
	Alfalfa		
	Alfalfa hay	20	
	Banana	0.1	
	Barley grain	0.1	
	Barley straw	1	
	Beet, Sugar	0.1	
	Beet, Sugar, top or leaves	1	
	Cattle fat	0.02	
	Cattle meat	0.02	
	Cattle meat by-products	0.02	
	Coffee	0.1	
	Cotton seed	0.2	
	Cucumber	0.2	
	Goat fat	0.02	
	Goat meat	0.02	
	Goat meat by-products	0.02	
	Gourd	0.6	
	Grape	0.2	
	Grape pomace, dried	1.5	F
	Grape, raisin	1	F
	Grape, raisin waste	3	F
	Horse fat	0.02	
	Horse meat	0.02	
	Horse meat by-products	0.02	
	Maize fodder		
	Maize forage		
	Maize grain	0.1	
	Maize, fresh	0.2	
	Melon	0.2	
	Milk	0.02	
	Oat grain	0.1	
	Oat straw	1	
	Peanut	1.5	
	Peanut hull	8	
	Peanut soapstock	3	F
	Pepper, Cayenne	0.2	
	Pepper, Sweet	0.2	
	Pig fat	0.02	
	Pig meat	0.02	
	Pig meat by-products	0.02	
	Popcorn grain	0.1	
	Potato	1	
	Pumpkin	0.6	
	Rape, canola seed	0.2 T	Until 22/02/98

Country	Commodity	MRL, mg/kg	Remark
	Rice	0.2	
	Rice straw	0.2	
	Sheep fat	0.02	
	Sheep meat	0.02	
	Sheep meat by-products	0.02	
	Sorghum fodder	0.5	
	Sorghum forage	0.5	
	Sorghum grain	0.1	
	Soya	0.2	
	Soya forage	20	
	Soya hay	20	
	Soya soapstock	1	F
	Strawberry	0.2	
	Sugar cane	0.1	
	Sunflower meal	0.6	F
	Sunflower seed	0.5	
	Sunflower soapstock	0.5	F
	Sunflower, hull	0.6	F
	Sweet corn, fresh	0.2	
	Wheat grain	0.1	
	Wheat straw	1	

*At or about the limit of determination

F: Food-additive tolerance

T: Temporary

APPRAISAL

Carbofuran, 2,3-dihydro-2,2-dimethylbenzofuran-7-yl methylcarbamate, is a widely used insecticide, nematocide, and acaricide. Its uses include seed treatment, at-plant soil application, and directed or foliar applications. A periodic review of the toxicology of carbofuran was carried out by the 1996 JMPR and the present evaluation is a periodic review of its residue and analytical aspects.

Carbosulfan produces carbofuran as a major metabolite. The periodic review of carbosulfan at the present Meeting includes an evaluation of its use on citrus fruit. In evaluating carbofuran, account was taken of its residues arising from the use of carbosulfan on citrus.

Animal metabolism

Studies were provided by the sponsors on rats, houseflies, laying hens, and lactating goats. The metabolism is similar in all species and consists of oxidation at the C-3 position and hydrolysis of the carbamate ester. The major metabolites observed in the urine from rats treated orally with single doses of carbonyl- or phenyl-labelled [¹⁴C]carbofuran were 3-hydroxycarbofuran (14%), 3-ketocarbofuran (48%), the 7-phenol (20%), and the 3-hydroxy-7-phenol (1.4%). The major compounds found from the topical treatment of houseflies with radiolabelled carbofuran were carbofuran (12% internal), 3-hydroxycarbofuran (6%), and conjugated 3-hydroxycarbofuran (11%).

Hens were given 3 mg of phenyl-labelled [¹⁴C]carbofuran for 7 consecutive days, about 2 mg/kg bw/day, equivalent to about 25 ppm in the feed. Eggs and tissues were collected and subjected to a series of extractions and hydrolyses. The residues in muscle and fat were negligible, and radiolabelled residues in the kidneys, liver, and eggs ranged from 0.03 to 0.15 mg/kg expressed as carbofuran. The major metabolite found in eggs was the 3-hydroxy-7-phenol (39% of the TRR).

About 5% of the TRR in the liver and kidneys was identified as the 7-phenol, and significant proportions were characterized as releasable by treatment with protease or strong acid.

[¹⁴C]Carbofuran, uniformly labelled in the phenyl ring, was administered orally to goats for 7 consecutive days at a rate equivalent to 25 ppm carbofuran in the diet. Milk and excreta were collected daily, and tissues were taken within 24 hours of the final dosing. The total radioactive residue in the milk remained fairly constant (0.10 mg/kg), and residues in the fat and tissues were negligible (<0.01 mg/kg). The milk and tissues were extracted with a series of solvents and subjected to enzymatic and acid/base hydrolyses. The major metabolites released and subsequently identified in the milk were 3-hydroxycarbofuran (10% of the TRR), the 7-phenol (15% of the TRR), and the 3-keto-7-phenol (32% of the TRR). Protease released 13% and 16% of the TRR from the kidneys and liver respectively. Major metabolites in the kidneys were 3-hydroxycarbofuran (11% of the TRR) and the 3-hydroxy-7-phenol (16% of the TRR, enzyme-released).

Plant metabolism

Studies were reported on potatoes, soya beans, and maize. The major metabolites identified in potato tubers were the 7-phenol (45% of the TRR) and the 3-hydroxy-7-phenol (13%). Immature foliage contained 3-hydroxycarbofuran (23% of the TRR) and a metabolite unique to the potato, 5-hydroxycarbofuran (34%). In soya bean forage (45-day PHI), the major compounds were identified as carbofuran (11% of the TRR) and 3-hydroxycarbofuran (28%). At a longer pre-harvest interval (139 days), the beans showed a substantial residue (40% of the TRR) releasable only by enzymes and acid and base hydrolyses. Only 12-13% the residue in the beans and hay was identified. The major metabolites in the beans were 3-ketocarbofuran (5% of the TRR) and the 3-keto-7-phenol (9%). The main compounds identified in maize forage were carbofuran and 3-hydroxycarbofuran (14% and 13% of the TRR).

The metabolites identified or characterized in the plants are consistent with hydroxylation at C-3 and hydrolysis of the carbamate, as in animals. Substantial conjugation of the metabolites and incorporation of the radiolabel into plant constituents occur.

The Meeting concluded that the animal and plant metabolism studies were fully adequate and showed a common metabolic pathway.

Environmental fate

Studies were reported on aerobic soil degradation, aerobic and anaerobic aquatic degradation, soil photolysis, terrestrial field dissipation, aqueous photolysis, and aquatic field dissipation.

The major pathway of degradation of [¹⁴C]carbofuran in aerobic soil was by hydroxylation and oxidation at the C-3 position, yielding 3-hydroxycarbofuran and 3-ketocarbofuran. The half-life of carbofuran was calculated to be 320 days under acidic conditions and 150 days under alkaline conditions.

In an anaerobic water/sediment study more than 50% of the [¹⁴C]carbofuran was converted to the 7-phenol, which was also a major product of anaerobic aquatic degradation where the carbofuran half-life was 120 days.

The aerobic aquatic half-life in a water/sediment system at pH 5.4 was 40 days.

The photolysis half-life of carbofuran in soil was about 78 days. Carbofuran is photolytically stable in aqueous solution, with a half-life of 450-1200 days.

From the soil dissipation studies it was determined that the half-life of carbofuran at a 0-6 inch depth was 13-43 days. The aquatic field dissipation study showed a carbofuran half-life of <10 days for carbofuran in rice paddy water. Thus, transfer of carbofuran via irrigation water is not anticipated to be a serious concern.

It was shown that carbofuran can be leached from four different types of soil under vigorous conditions.

The Meeting concluded that carbofuran is readily degraded in aquatic systems and that it is somewhat persistent in soil. Degradation in soil and water involves hydroxylation at the C-3 carbon and hydrolysis of the carbamate.

Methods of residue analysis

The methods of analysis are adequate for monitoring and for use in supervised trials, and at least one multi-residue method exists which is suitable for monitoring and enforcement.

The commonly used HPLC method involves solvent extraction of the homogenized sample, purification on a solid-phase extraction column, and determination on a reverse-phase column. A post-column reactor converts the eluted methylcarbamates to an indole, which is measured fluorimetrically. The method has a demonstrated limit of determination of about 0.05 mg/kg for carbofuran, 3-hydroxycarbofuran, and 3-ketocarbofuran. The limit of determination in milk is 0.025 mg/kg. A variation of the method involves initial hydrolysis of the homogenized sample with 0.25 N HCl to release any conjugates.

Several GLC methods exist for the determination of the carbamate metabolites. A macerated sample is refluxed with 0.25 N HCl, partitioned into methylene chloride, and purified on a Florisil column. A methyl silicone capillary column and a nitrogen-phosphorus or mass spectrometric detector are used. The method may be modified by ethylating the 3-hydroxycarbofuran. Limits of determination of 0.05 to 0.10 mg/kg were demonstrated.

In an older variation of the GLC method the initial extraction of the sample is with methanol/chloroform. The residual aqueous fraction is then hydrolysed with acid. A limit of determination of 0.1 mg/kg is claimed, but recoveries of the conjugate of 3-hydroxycarbofuran were generally unacceptable below 1 mg/kg. A variation of this method did not include acid hydrolysis, and the limit of determination for carbofuran and 3-hydroxycarbofuran was 0.1 mg/kg.

Stability of residues in stored analytical samples

Information was submitted on the stability of carbofuran, 3-hydroxycarbofuran, and 3-ketocarbofuran in or on several diverse raw agricultural commodities. The Meeting concluded that carbofuran and its carbamate metabolites are stable for at least 2 years in or on frozen plant commodities and milk, and for 1 year in meat.

Definition of the residue

The residue is defined for compliance with MRLs as the sum of carbofuran and 3-hydroxycarbofuran, expressed as carbofuran. For the estimation of dietary intake the residue should be defined as the sum of carbofuran, free 3-hydroxycarbofuran and conjugated 3-

hydroxycarbofuran, expressed as carbofuran. The metabolism studies on soya beans and maize showed that the concentration of conjugated 3-hydroxycarbofuran was equal to or greater than that of 3-hydroxycarbofuran. For example, in soya bean forage (63 mg/kg of ^{14}C expressed as carbofuran) the free 3-hydroxycarbofuran was 11% of the TRR and the conjugated (acid-released) 3-hydroxycarbofuran was 17%. In the beans the concentrations were approximately equal. Where the analytical method used for a field trial did not include an acid hydrolysis step (refluxing with 0.1 N HCl) to release conjugates of 3-hydroxycarbofuran, the results were not used in the determination of the STMR levels.

Supervised trials

Residue trials were reported on numerous crops: alfalfa, bananas, Brussels sprouts, cantaloupes, cauliflower, celeriac, celery, coffee, cucumbers, grapes, head cabbages, kohlrabi, leeks, maize, oilseed plants (cotton, sunflower, rape, peanuts), onions, peppers, potatoes, rice, sorghum, soya beans, strawberries, sugar beet, sugar cane, summer squash, sweet corn, tomatoes, turnips, and wheat.

Fruits

Citrus fruits. Residues of carbofuran, 3-hydroxycarbofuran, and 3-ketocarbofuran may occur on citrus from the use of carbosulfan. On the basis of the concurrent review of carbosulfan the Meeting estimated a maximum residue level for carbofuran plus 3-hydroxycarbofuran in oranges of 0.5 mg/kg, and an STMR of 0.1 mg/kg.

Grapes. Field trials in the USA, Germany, and Mexico were reported. The four trials in Germany were not considered because the residue determined and the maturity of the crop samples were not clearly explained; the report consisted only of a simple summary. US GAP was used to evaluate the trials in Mexico and the USA (11.2 kg ai/ha of 4 F formulation, applied after harvest with a PHI of 200 days and soil-incorporated; pre-harvest drip irrigation with 4F at 3.4 kg ai/ha, 60-day PHI). One US and three Mexican trials complied with GAP for the vine treatment after harvest, and one US trial with GAP for the pre-harvest treatment. The residues were <0.05 mg/kg in all five trials, but five trials were considered to be insufficient for the estimation of a maximum residue level.

Strawberries. Supervised field trials were reported from France (0.89-1 kg ai/ha, PHI 13-48 days), the UK (2 kg ai/ha, 300-day PHI), and the USA (2.2 kg ai/ha, 250-day PHI). The results constituted two distinct sets, one for the after-harvest application to vines (UK and USA) where residues were below the limit of determination, 0.05-0.1 mg/kg, and the other with residues from <0.1 to 0.94 mg/kg (France). No information on GAP was provided for France or the UK or a neighbouring nation. The US trials conformed to US GAP, 2.2 kg ai/ha applied post-harvest after 1 October. The residues in the three trials were all 0.02 mg/kg. The results were insufficient to estimate a maximum residue level and the Meeting recommended the withdrawal of the existing CXL (0.1* mg/kg).

Bananas. Field trials in Spain, Central America and South America with the application of carbofuran to banana trees were reported. No residues of carbofuran plus 3-hydroxycarbofuran (<0.02-<0.10 mg/kg, n = 8) were found in any trial. GAP was available only for Spain, where the trial was according to GAP and undetectable residues were <0.02 mg/kg. Because none of the trials, some of which were at higher rates than GAP, yielded detectable residues the Meeting estimated a maximum residue level of 0.1* mg/kg, the same as the existing CXL, and an STMR of 0.1 mg/kg.

Vegetables

Leeks. Curaterr 200 SC was applied to the soil before planting leeks at two locations in The Netherlands. Carbamate residues were above the limit of determination in one trial, with a maximum of 0.15 mg/kg. The number of trials was inadequate to estimate a maximum residue level.

Onions. Curaterr 5G or 200 SC was applied to onions at three locations after or before sowing. The carbamate residues were below the limit of determination. There were too few trials to estimate a maximum residue level. The Meeting recommended withdrawal of the existing CXL for bulb onion (0.1* mg/kg).

Head cabbages. Two supervised field trials were reported for the application of Curaterr 200 SC to head cabbage in The Netherlands. No residues were detected (<0.1 mg/kg). Two trials are too few for the estimation of a maximum residue level and the Meeting recommended the withdrawal of the existing CXL (0.5 mg/kg).

Brussels sprouts. Again only two trials in The Netherlands were reported. The Meeting recommended the withdrawal of the existing CXL (2 mg/kg).

Cauliflower. Five trials were carried out in The Netherlands with Curaterr 200 SC applied to cauliflower plants at 0.038 g ai/plant. The mode and timing of the application were not reported. No GAP was available for The Netherlands or other EU country and the data could not be evaluated. The Meeting recommended the withdrawal of the existing CXL (0.2 mg/kg).

Kohlrabi. Two field trials were carried out in Germany with single applications of a granular formulation at 0.64 g/m 38 and 52 days after planting but no GAP was reported. The Meeting recommended the withdrawal of the existing CXL (0.1* mg/kg).

Cucumbers. Field trials were carried out in the USA. US GAP specifies the at-plant application of 2.2 kg ai/ha of a G formulation or 1.7 kg ai/ha of an F formulation. The trials were conducted at 1.1 and 3.4 kg ai/ha with both the 15 G and 4 F formulations. The lower rate is below maximum GAP and the higher exceeds it. The results from the two rates were comparable and could therefore be used to represent the GAP rate. The residues from the 1.1 kg ai/ha rate were 0.02 (6), 0.04, 0.05, 0.08, 0.09, 0.15 (2), 0.16 and 0.21 mg/kg (n = 14), and those from the 3.4 kg ai/ha rate were 0.02 (4), 0.04 (2), 0.05 (2), 0.13, 0.16, 0.18, 0.21, 0.26 and 0.29 mg/kg, n = 14. The STMR for the 3.4 kg ai/ha rate is 0.05 mg/kg, and that for the 1.1 kg ai/ha rate 0.045 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.05 mg/kg from the combined results.

Cantaloupes. Supervised field trials in the USA were reported, with the application of Furadan 15G or 4F to cantaloupes at planting, with PHIs of 60-92 days. The application rates were 1.1 or 3.4 kg ai/ha. Four trials were conducted in each of seven states. GAP specifies at-plant application of the G formulation at 2.2 kg ai/ha or the F formulation at 1.7 kg ai/ha. Some trials were below and others above maximum GAP. The results from the high and low application rates were similar, and could be used to represent residues resulting from GAP applications. The residues from the 1.1 kg ai/ha rate were 0.02 (8), 0.05 (2), 0.11 (3) and 0.13 mg/kg (n = 14), and those from the higher rate 0.02 (7), 0.05 (5), 0.11 and 0.12 mg/kg (n = 14). The STMR for the 1.1 kg ai/ha rate would be 0.02 mg/kg, and for the 3.4 kg ai/ha rate 0.035 mg/kg. Combining the distributions, the STMR is 0.02 mg/kg. The Meeting estimated a maximum residue level of 0.2 mg/kg.

Summer squash. GAP in the USA is the same as for cucumbers and cantaloupes. Supervised field trials were carried out in seven states of the USA with the at-plant application of carbofuran 15G and 4F formulations at 1.1 and 3.4 kg ai/ha, some therefore below and some above maximum GAP. The results from the high and low application rates were similar, and the trials may be taken to represent

applications according to GAP. The residues in rank order from 1.1 kg ai/ha were 0.02 (7), 0.05 (2), 0.07, 0.10, 0.11, 0.13 and 0.26 mg/kg (n = 14), and from 3.4 kg ai/ha 0.02 (5), 0.04, 0.06 (3), 0.07, 0.08, 0.09, 0.12 and 0.15 mg/kg (n = 14). The STMR for the 1.1 kg ai/ha rate is 0.035 mg/kg, and for both 3.4 kg ai/ha and for all the trials combined 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.05 mg/kg.

Peppers (hot). Carbofuran was applied to the soil before planting hot peppers in two trials in the USA, with a second post-emergence side-dress application. The trials were according to, but not at the maximum, US GAP. No maximum residue level could be estimated.

Peppers (sweet). Furadan 4F was applied to sweet peppers in Canada and the USA. GAP was not available for Canada. US GAP specifies two applications of a 4 F formulation, one at-plant and the second as a side-dress, with a 21-day PHI. Each application is 3.4 kg ai/ha. The Canadian applications were in excess of US GAP at 5 x 0.56 kg ai/ha, 1-3-day PHI, and the results were not evaluated. In the US trials the application rate was ≤50% of the maximum GAP rate. The Meeting concluded that the data were insufficient to estimate a maximum residue level.

Tomatoes. Field trials were carried out in Brazil, Canada, France, Mexico, and the USA. The government of Thailand provided information on field trial conditions but did not include any analytical results. Most of the treatments were with a granular formulation applied to the soil round the plants. No GAP was reported for France, Mexico, or Canada, and the results from these countries could not be evaluated. There is no GAP in the USA. Two trials in Brazil which complied with GAP gave results of 0.05 mg/kg, but two samples are not enough to estimate a maximum residue level. The Meeting recommended withdrawal of the existing CXL (5 mg/kg).

Sweet corn (corn-on-the-cob). The findings of sixteen field trials on sweet corn were submitted from the USA. A combination of at-planting, at whorl, and foliar applications were made with granular and flowable formulations in accordance with the current label, at the maximum rate and with a minimum PHI. The commodity analysed was corn and cob, less husk. GAP was followed (1.12 kg ai/ha at-plant, followed by 4 foliar applications, each 0.56 kg ai/ha, 7-day PHI), and the total carbamate residues (carbofuran + 3-hydroxycarbofuran) in rank order were <0.03 (6), 0.03 (4), 0.04 (4), 0.05 and 0.08 mg/kg (n = 16). The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg.

Soya beans. Trials were reported from Brazil, France, and the USA. Brazilian GAP specifies at-plant application of a 10% G at 1.5 kg ai/ha. US GAP allows 2.0 kg ai/ha at-plant or 2 applications at 0.56 kg ai/ha/application. No information on GAP was available for France or a neighbouring country. Only two trials according to GAP were reported, one from Brazil and the other from the USA. The residue in Brazil was below the limit of detection (0.05 mg/kg) and that in the USA was at the limit of determination (0.10 mg/kg). Two results are inadequate to estimate a maximum residue level, and the Meeting recommended withdrawal of the existing CXL for soya bean (dry) of 0.2 mg/kg.

Yard-long beans. The government of Thailand submitted a description of the in-field aspects of trials on yard-long beans but included no residue data. The Meeting took no action.

Carrots. The government of The Netherlands reported six field trials with at-plant application of an SC formulation to carrots. No GAP is available for The Netherlands or an EU country. The Meeting recommended withdrawal of the existing CXL (0.5 mg/kg).

Celeriac. The government of The Netherlands reported the results of one field trial with the application of an SC carbofuran formulation to celeriac. No GAP was reported and one trial is inadequate even for a very minor crop.

Potatoes. Field trials were carried out in Colombia, France, the UK and the USA. Applications according to GAP range from at-planting in Europe to banded treatment at hill-up and multiple foliar sprays in the USA. No GAP was available for Colombia, and the trials there were not evaluated. France and the UK each reported one trial in accordance with GAP. Six trials in the USA complied with the appropriate GAP, 3.4 kg ai/ha at-plant and 8 foliar applications at 1.1 kg ai/ha each, PHI 17 days. The residues in the whole tubers in the eight trials were <0.01 (3), <0.03 (2), 0.03, 0.04 and <0.05 mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.03 mg/kg.

Sugar beet. Field trials were carried out in France, Italy, Germany, the UK and the USA. European GAP specifies 2 kg ai/ha at planting, and US GAP early post-emergence foliar treatment (2.2 kg ai/ha, 90-day PHI). Five trials were at the maximum GAP rate and minimum PHI. The residues on the foliage were <0.01 (3), 0.05 and 0.15 mg/kg and in the roots <0.01 (4) mg/kg. The data were insufficient to estimate a maximum residue level. The Meeting recommended withdrawal of the existing CXLs for sugar beet and sugar beet leaves or tops.

Turnips. Five field trials on the application of carbofuran to turnips in France, the UK and Norway were reported. No information on GAP was available, and the Meeting could take no action.

Celery. In two field trials in The Netherlands carbofuran was applied to the soil immediately before planting. The Meeting could not estimate a maximum residue level.

Cereal grains

Maize. Reports of trials in Brazil, France, Germany and the USA were submitted. The trials represented a combination of at-planting (France, USA, Germany) and foliar (USA, Brazil) treatments. The reports from Brazil, France, and Germany were abbreviated summaries and did not provide the detail required to evaluate the trials. The results were not used in attempting to estimate a maximum residue level and an STMR.

Eleven trials were conducted in the USA, but only two residues in silage were from trials according to current GAP. In the trials with at-plant applications the rate was 34% higher than the GAP rate and a granular formulation was used in place of the specified soluble concentrate. All the samples of forage, fodder and grain were harvested well outside the GAP PHI (>30% deviation). The two silage residues (1.1 and 1.2 mg/kg) were insufficient to estimate a maximum residue level or an STMR, nor could the Meeting estimate maximum residue levels for maize, maize fodder or maize forage. It therefore recommended withdrawal of the CXLs for maize and maize fodder.

Oats. Field trials on 3 varieties at one location were reported from Germany. The treatment was at-planting, and no residues (<0.10 mg/kg) were found in the oats. The number of trials was inadequate and the report consisted of a short summary that lacked the detail required for evaluation. The Meeting recommended withdrawal of the existing CXL (0.1* mg/kg).

Rice. Field trials in Australia, Brazil, Japan, the Philippines, and the USA were reported. The Brazilian summary report lacked the detail needed to evaluate the trials. The trials in the USA, Japan, and Philippines were not according to GAP. Only one trial in Australia accorded with GAP. The Meeting recommended withdrawal of the existing CXL (0.2 mg/kg).

Sorghum. See Sorghum forage etc., below.

Wheat. Field trials in South Africa and the USA were reported. Information on GAP was not available for the at-plant trials in South Africa. The six US trials were at the maximum GAP rate, with two foliar treatments and a 21-day PHI. The total carbamate residues in the grain in rank order were 0.02, 0.02 and 0.04 (4) mg/kg. The Meeting concluded that six trials were insufficient to estimate a maximum residue level and recommended withdrawal of the existing CXL (0.1* mg/kg).

Other crops

Sugar cane. Supervised field trials with the application of carbofuran to sugar cane were carried out in Brazil and the USA. In Brazil, the carbofuran (G or SC) was applied as a soil treatment about 5 months after planting. The PHI was 90 days. No residues were found (<0.1 mg/kg) in the four trials, two of which complied with GAP and two were at twice the GAP rate. In five trials in three states of the USA with the 4F formulation an in-furrow application at planting (1.1 kg ai/ha) was followed by two aerial foliar applications (2 x 0.84 kg ai/ha), with a 30-day PHI. This was according to GAP, and the maximum carbofuran residue was 0.06 mg/kg. The Meeting estimated a maximum residue level of 0.1* mg/kg, the existing CXL and the practical limit of quantification, and an STMR of 0.1 mg/kg.

Oilseed (cotton, sunflower, peanut, rape). Field trials in the USA and Brazil on cotton were reported, and the sponsor stated that trials were now in progress in southern Europe. The trials in Brazil were with seed treatment or a single post-emergence foliar treatment (2.1 kg ai/ha, 45-day PHI). The US trials involved two foliar applications of a flowable formulation (2 x 0.28 kg ai/ha). Neither set of trials complied with the relevant GAP, which is for at-plant use in both countries.

Trials on peanuts in Brazil and the USA were reported. The government of Thailand submitted information on field trials but no data on residues. Carbofuran was applied to peanut plants in two trials in Brazil as a foliar spray (1.75 or 3 kg ai/ha) with a 14-day PHI. In 14 US trials, peanut fields were treated at pegging. In some cases an initial treatment was also made at planting. The maximum carbamate residue was 0.53 mg/kg. Most of the US trials (80%) showed no quantifiable residues. GAP in Brazil is for at-plant treatment, and the USA has no GAP for the use of carbofuran on peanuts. Neither the Brazilian nor the US results could be used to estimate a maximum residue level.

Field trials on rape (canola) were carried out in Canada (seed treatment, at-plant, post-emergence) and France (at-plant). No GAP was reported for Canada or France, and the Canadian trials did not comply with US GAP. The trials could not be evaluated.

Field trials on sunflowers were carried out in Canada, France and the USA. The trials in France were discounted, because the method of analysis was described as semi-quantitative and was not explained. The US trials were not according to GAP; the PHI was >150% of the GAP PHI of 28 days, and the at-plant application was below the maximum rate. Six trials in Canada complied with maximum US GAP and all the residues were 0.04 mg/kg. The Meeting estimated a maximum residue level for sunflower seed of 0.1* mg/kg and an STMR of 0.1 mg/kg, but concluded that the trials were inadequate to support an MRL for oilseed and recommended withdrawal of the existing CXL (0.1* mg/kg).

Coffee. Two field trials in Brazil and four in the USA, all according to national GAP, on the application of carbofuran to coffee bushes were reported. The use patterns are quite similar in both countries. GAP in Brazil specifies 0.35 g ai/tree of SC formulation or 0.5-3 g ai/tree of G formulation, and US GAP specifies two applications of 1.7 g ai/tree, 10 G formulation. The residues in rank order

were 0.02 (3), 0.08, 0.12 and 0.79 mg/kg (n = 6). The Meeting estimated a maximum residue level of 1 mg/kg and an STMR of 0.10 mg/kg. The two Brazilian residues of 0.02 mg/kg were not used for the estimation of the STMR because the analysis did not include a hydrolysis step to release conjugated 3-hydroxycarbofuran.

Alfalfa. Three field trials in each of seven states in the USA were according to the current maximum use rate and minimum PHI. Green forage and fodder were analysed. The carbamate residues in the fodder ranged from the limit of detection (<0.1 mg/kg) to 7.6 mg/kg. The trials involved foliar application of a flowable formulation at 1.12 kg ai/ha with a 28-day PHI. The maximum residues of carbofuran plus 3-hydroxycarbofuran in each trial in rank order were <0.1 (2), 0.28, 0.32, 0.64, 0.74, 0.87, 0.90, 0.92, 1.2, 1.4, 1.5, 1.6, 2.6, 2.8, 3.0, 3.4, 3.8, 4.2, 4.5, 4.6, 4.7, 5.2 and 7.6 mg/kg (n = 24). The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 1.6 mg/kg. The residues in the green forage in rank order were <0.1 (5), 0.13, 0.29, 0.30, 0.34, 0.38, 0.52, 0.92, 0.94, 1.2 (3), 1.3, 1.4, 1.6 (2), 1.7, 1.8, 2.2 and 4.3 mg/kg (n = 24). The Meeting estimated a maximum residue level of 10 mg/kg and an STMR of 0.93 mg/kg.

Sorghum forage (green), sorghum straw and fodder, dry. Six trials in India and six in the USA were reported. The trials in India were with seed treatment or at-plant treatment, whereas the US trials were at-plant plus two foliar applications, with a total rate of 2.3 kg ai/ha. GAP was not available for India or a neighbouring country. In the US trials the residues in rank order were 0.055 (6), 0.06, 0.07, 0.11 (2), 0.13, 0.19, 0.26 and 1.2 mg/kg (n = 14) in sorghum forage (green), 0.05 (2), 0.06 and 0.20 mg/kg (n = 4) in sorghum fodder, and <0.01 (5) mg/kg in sorghum grain. The Meeting estimated maximum residue levels of 2 mg/kg for forage, 0.5 mg/kg for fodder, and 0.1* mg/kg for grain, with respective STMRs of 0.065 mg/kg, 0.055 mg/kg and 0.01 mg/kg. Although no residues were found in the grain at an estimated limit of detection of 0.01 mg/kg, the practical limit of quantification for carbofuran and 3-hydroxycarbofuran individually in plant commodities is 0.1 mg/kg.

Barley, egg plant, hops (dry), mustard seeds, peaches, pears. No trials were reported. The Meeting recommended withdrawal of the existing CXLs.

Feeding studies on poultry and cows were reported. The poultry study was defective because although residues were reported as <0.05 mg/kg from feeding 5 ppm in the diet, the uncertainties surrounding the method of analysis cast doubts on the reliability of the results. A study conducted over 7 days at 25 ppm however showed negligible concentrations of radiolabelled residue (<0.01 mg/kg as carbofuran) in muscle and fat and a residue of 0.15 mg/kg in eggs, of which the carbamate content was below 20%. Potential poultry feed items include small grain (maize, barley, oats, wheat, sorghum, 80% of the diet) and alfalfa meal (10% of the diet). Thus, the diet might contain 80% x the 0.04 mg/kg STMR of maize + 10% x the 1.2 mg/kg STMR of alfalfa hay = 0.15 mg/kg. Note that this includes a commodity (maize) for which the withdrawal of a CXL has been recommended. Residues of carbofuran and its carbamate metabolites in poultry commodities are unlikely from such feeding levels. The Meeting concluded that MRLs are not needed for poultry commodities.

The ruminant feeding study was conducted with carbosulfan, not carbofuran. Carbosulfan is metabolized rapidly to carbofuran in ruminants, and the carbofuran is converted to 3-hydroxycarbofuran and phenol metabolites. Goats were fed carbosulfan at a level of 50 ppm in the diet for 28 days. The milk contained no detectable residues of carbosulfan on days 1-4, but it was present at very low concentrations, 0.005-0.011 mg/kg, from days 7 to 27. Carbofuran was detected on day 4 at a maximum concentration of 0.006 mg/kg and on day 7 at a maximum of 0.008 mg/kg. The carbofuran metabolite 3-hydroxycarbofuran appeared on day 1 (0.022 mg/kg) and continued through day 27 (0.013 mg/kg). The tissues contained no detectable residues of carbosulfan or carbofuran, but 0.060 mg/kg of 3-hydroxycarbofuran was found in the liver and 0.13 mg/kg in

kidney. The Meeting concluded that feeding with carbosulfan may be substituted for feeding with carbofuran.

The study of metabolism in goats, conducted for 7 consecutive days with 25 ppm carbofuran in the feed, revealed no radiolabelled residues (<0.01 mg/kg as carbofuran) in the muscles or fat. Significant residues occurred in the milk (0.14 mg/kg) and in the liver and kidneys (0.11, 0.18 mg/kg). About 50% of the TRR in the milk was shown not to include carbamate compounds, and about 11% was carbofuran plus 3-hydroxycarbofuran (0.02 mg/kg). The kidneys and liver each contained <15% carbamates (0.02 mg/kg).

On the basis of the MRLs recommended by the present Meeting, the ruminant diet would contain no more than 2 mg/kg of carbofuran plus 3-hydroxycarbofuran. This is based on a diet containing 80% of alfalfa fodder (0.8 x the STMR of 1.6 mg/kg = 1.3 mg/kg). Owing to the substantial number of MRLs recommended for withdrawal there are few animal feed items. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.05 mg/kg for residues (as defined above) in various animal products and milks.

Processing

Studies were conducted with sorghum, sugar beet, potatoes, maize, rice, sunflowers, cotton seed, sugar cane, coffee, pimento peppers, and grapes. Most of them were of limited value because the raw agricultural commodities contained carbamate residues below the limit of detection or between the limit of detection and the limit of determination. In most cases the same applied to the processed commodities. On the basis of the recommendations of the Meeting, processing studies would be appropriate for coffee, potatoes, sunflowers, and sugar cane. The sugar cane and potato processing studies, with applications at 1.8 times and twice the GAP rate respectively, were inadequate because there were no residues in the raw agricultural commodities. The sunflower processing study was acceptable: the residue was unchanged in the edible oil and increased in the hulls and extracted meal by factors of 1.2 and 1.8 respectively. The coffee processing study showed a reduction factor of approximately 0.05-fold for instant and roast coffee. The value is approximate because the residues in the processed commodities were at the limit of detection.

RECOMMENDATIONS[AFM1]

The Meeting estimated the maximum residues and STMR residues listed below. The maximum residue levels are recommended for use as MRLs.

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Recommended MRL or ERL (mg/kg)		STMR (mg/kg)	
			Name	New	Previous	New
Carbofuran** (096)	0.002	AL 1020	Alfalfa fodder	10	20	1.6
		AL 1021	Alfalfa forage (green)	10	5	0.93
		FI 0327	Banana	0.1*	0.1*	0.1
		GC 0640	Barley	W	0.1*	
		VB 0402	Brussels sprouts	W	2	
		VB 0041	Cabbages, Head	W	0.5	
		VC 4199	Cantaloupe	0.2	-	0.02
		VR 0577	Carrot	W	0.5	
		MF 0812	Cattle fat	0.05*	0.05*	0.05

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Recommended MRL or ERL (mg/kg)		STMR (mg/kg)	
			Name	New	Previous	New
		VB 0404	Cauliflower	W	0.2	
		DM 0001	Citrus molasses ¹			0.11 P
		AB 0001	Citrus pulp, dry ¹	2	-	0.29
		SB 0716	Coffee beans	1	0.1*	0.1
			Coffee, Instant			0.005 P ¹
		SM 0716	Coffee, Roast			0.005 P
		VC 0424	Cucumber	0.3	-	0.05
		MO 0096	Edible offal of cattle, goats, horses, pigs and sheep	0.05*	0.05*	0.05
		VO 0440	Egg plant	W	0.1*	
		MF 0814	Goat fat	0.05*	0.05*	0.05
		DH 1100	Hops, dry	W	5	
		MF 0816	Horse fat	0.05*	0.05*	0.05
		VB 0405	Kohlrabi	W	0.1*	
		VL 0482	Lettuce, Head	W	0.1*	
		GC 0645	Maize	W	0.1*	
		AS 0645	Maize fodder	W	5	
		MM 0096	Meat of cattle, goats, horses, pigs and sheep	0.05*	0.05*	0.05
		ML 0106	Milks	0.05*	0.05*	0.05
		SO 0090	Mustard seed	W	0.1*	
		GC 0647	Oats	W	0.1*	
		SO 0088	Oilseed	W	0.1*	
		VA 0385	Onion, Bulb	W	0.1*	
		FC 0004	Oranges, Sweet, Sour ¹	0.5	-	0.1
		JF 0004	Orange juice ¹			0.001
		FS 0247	Peach	W	0.1*	
		FP 0230	Pear	W	0.1*	
		MF 0818	Pig fat	0.05*	0.05*	0.05
		VR 0589	Potato	0.1	0.5	0.03
		CM 0649	Rice, Husked	W	0.2	
		MF 0822	Sheep fat	0.05*	0.05*	0.05
		GC 0651	Sorghum	0.1*	0.1*	0.01
		AF 0651	Sorghum forage (green)	2	-	0.065
		AS 0651	Sorghum straw and fodder, dry	0.5	-	0.055
		VD 0541	Soya bean, dry	W	0.2	
		VC 0431	Squash, Summer	0.3	-	0.05
		FB 0275	Strawberry	W	0.1*	
		VR 0596	Sugar beet	W	0.1*	
		AV 0596	Sugar beet leaves or tops	W	0.2	
		GS 0659	Sugar cane	0.1*	0.1*	0.1
		SO 0702	Sunflower seed	0.1*	0.1* ²	0.1
		VO 1275	Sweet corn (kernels)	W	0.1*	
		VO 0447	Sweet corn (corn-on-the -cob)	0.1	-	0.03
		VO 0448	Tomato	W	0.1*	

Pesticide (Codex ref. No.)	ADI (mg/kg bw)	Commodity CCN	Recommended MRL or ERL (mg/kg)		STMR (mg/kg)	
			Name	New	Previous	New
		GC 0654	Wheat	W	0.1*	

FURTHER WORK OR INFORMATION

Desirable

1. A feeding study with cows fed carbofuran.
2. Processing studies on potatoes and sugar cane. Exaggerated treatment rates (five- to tenfold) should be used to obtain weathered residues in or on the raw agricultural commodities.

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