ALDICARB (117)

EXPLANATION

Aldicarb was first evaluated in 1979, and has been reviewed in 1982, 1985, 1988, 1990 (correction in 1991) and 1993. It is included in the CCPR periodic review programme.

Since the information on residues in potatoes and Brussels sprouts has been recently reviewed (1990 and 1993 respectively), the residues in these commodities were not evaluated by the Meeting.

The manufacturer informed the Meeting that the use pattern on bananas has been changed. A programme of residue trials in accordance with the current use pattern is in progress and data will be provided for evaluation when it has been completed.

The manufacturer and some countries have provided extensive information on the fate of residues, residues resulting from supervised trials, the effects of processing, and analytical methods.

IDENTITY

ISO common name: aldicarb

Chemical name:

IUPAC:2-methyl-2-(methylthio)propionaldehyde O-methylcarbamoyloxime

CA: 2-methyl-2-(methylthio)propanal *O*-[(methylamino)carbonyl]oxime

CAS Number: 116-06-3

Synonyms: OMS 771, ENT 27093, AI3-27093, UC 21149, Temik, Sentry, Tranid

Structural Formula:

Empirical formula: C₇H₁₄N₂O₂S

Molecular Weight: 190.3

Physical and chemical properties

Purity of the active ingredient

Crystalline aldicarb, the active ingredient, is never isolated during manufacture. The manufacturing concentrate, aldicarb solution (referred to as Aldisol), contains 36 to 39% aldicarb. The nominal concentration is 37.5%. Aldisol is used directly in the manufacture of the granular formulations; it is not used as an end-use product. The composition of Aldisol has been provided to the Meeting.

Vapour pressure: 2.90 x 10⁻⁵ mm Hg at 24°C

standard deviation 0.21 x 10⁻⁵

This value supersedes the value reported from previous studies (McDaniel and Weiler, 1987).

Melting point:

Crystallised aldicarb: 100-101°C

Aldisol: Not applicable (liquid)

Boiling point:

Crystallised aldicarb: Decomposes above 100°C

Aldisol: 47-50°C

Density:

Crystallised aldicarb: 1.195 at 25°C Aldisol: 1.22 g/cm³ at 23°C

Henry's Law Constant: 1.23 x 10⁻⁹ atm m³ g.mol⁻¹ (Guyot, 1988c)

Colour:

Crystallised aldicarb: White Aldisol: Light amber

Odour:

Crystallised aldicarb: Slightly sulphurous

Aldisol: Slightly fishy and lachrymatory

Octanol-water partition

coefficient: $K_{ow} = 14.08$ at 25° C

This partition coefficient was found constant at two aldicarb concentrations (5 mM and 0.5 mM) and two octanol/water volume ratios (1/9 and 1/1) (Guyot, 1988a).

Solubility in water

aldicarb (99.5% purity): 5.88 g/l at 25°C(Guyot, 1988b). aldicarb (99% purity):

	10°C	20°C	30°C
pH 5		5.29	
pH 7	3.83	4.93	6.15
pH 9		6.15	

(Offizorz, 1992)

Solubility in organic solvents

The solubilities determined for crystallised aldicarb are shown in Table 1 (Union Carbide Ag. Products Co., 1984).

Table 1. Solubilities of crystalline aldicarb in organic solvents.

Solvent		Solubi	lity, %	
	10°C	25°C	30°C	50°C
Acetone	28	38	43	67
Benzene	9	18	24	49
Butyl acetate		13		
Butyl cellosolve		16		
Butyl ether		<8		
Carbon tetrachloride	2	4	5	25
Chlorobenzene		12		
Chloroform	38	42	44	53
Cyclohexanol		10		
Cyclohexanone		25		
Dichloromethane	40	47	50	60
Dimethyl sulphoxide		30		
Ethyl benzoate		14		
Ethylhexanol		6		
Hexamethylphosphoramide		33		
Hexane		<1		
Isophorone		20		
Mesityl oxide		25		
Methyl ethyl ketone		20		
Methyl isobutyl ketone	13	21	24	42
Methylpyrrolidone		33		
Nitrobenzene		20		
Nitromethane		26		
Propylene dichloride		20		
Toluene	10		12	33
Trichloroethylene		20		

Hydrolysis half-life: 81.34 days at pH 7 and 25°C (Andrawes 1976c)

0.87 day at pH 9 and 25°C

Flammability: Aldicarb, Aldisol and Temik formulations are all non-flammable.

Oxidising or reducing: Neither aldicarb nor any of the accompanying impurities have the tendency

action: to act as oxidizing or reducing agents.

Corrosion characteristics

Crystallised aldicarb: Non-corrosive.

Aldisol: Non-corrosive in anhydrous state.

Formulations

Aldicarb is available commercially only as granular formulations containing 50, 100 or 150 g/kg aldicarb (Trade name Temik). In certain European countries, granular formulations of aldicarb plus lindane are registered for use on sugar beet (Trade names Sentry, Tranid).

METABOLISM AND ENVIRONMENTAL FATE

The fate of aldicarb has been examined in a range of animal and plant species, and in soil and water. Most of the studies mentioned in the following discussion have been previously reviewed by the JMPR in 1979 or 1982. Several new studies have also been provided, including studies of metabolism in goats and poultry, a study of aerobic degradation in soil, a rotational crop study, and a study of photodegradation in aqueous solution.

The designations, chemical names and structures of identified metabolites and degradation products are shown below.

Designation	Chemical Name	Structure
Aldicarb	2-methyl-2-(methylthio)propionaldehyde	CH ₃
	O-methylcarbamoyloxime	CH ₃ -S- C-CH=NO-CO-NH-CH ₃
		CH ₃
Aldicarb sulphoxide	2-methyl-2-(methylsulphinyl)propionaldehyde <i>O</i> -methylcarbamoyloxime	O CH ₃
sulphoxide	methylcarbannoyioxinie	CH ₃ -S- C-CH=NO-CO-NH-CH ₃
		CH ₃
Aldicarb sulphone	2-methyl-2-(methylsulphonyl)propionaldehyde <i>O</i> -methylcarbamoyloxime	O CH ₃
surpriorie	methylcarbanioyioxinie	CH ₃ -S- C-CH=NO-CO-NH-CH ₃
		O CH ₃
Aldicarb	2-methyl-2-(methylthio)propionaldehyde oxime	CH ₃
oxime		CH ₃ -S- C-CH=N-OH
		CH ₃
Aldicarb	2-methyl-2-(methylsulphinyl)propionaldehyde oxime	O CH ₃
oxime sulphoxide		CH ₃ -S- C-CH=N-OH
		CH ₃
Aldicarb	2-methyl-2-(methylsulphonyl)propionaldehyde oxime	O CH ₃
oxime sulphone		CH ₃ -S- C-CH=N-OH
		O CH₃
Aldicarb	2-methyl-2-(methylthio)propionitrile	CH ₃
nitrile		CH ₃ -S- C-C≡N
		CH ₃

Designation	Chemical Name	Structure
Aldicarb	2-methyl-2-(methylsulphinyl)propionitrile	O CH ₃
nitrile sulphoxide		CH ₃ -S- C-C≡N
		CH ₃
Aldicarb	2-methyl-2-(methylsulphonyl)propionitrile	O CH ₃
nitrile sulphone		CH ₃ -S- C-C≡N
		O CH ₃
Aldicarb	2-methyl-2-(methylsulphinyl)propionamide	O CH ₃
amide sulphoxide		CH ₃ -S- C-CO-NH ₂
		СН₃
Aldicarb	2-methyl-2-(methylsulphonyl)propionamide	O CH ₃
amide sulphone		CH ₃ -S- C-CO-NH ₂
		O CH ₃
Aldicarb	2-methyl-2-(methylsulphinyl)propanol	O CH ₃
alcohol sulphoxide		CH ₃ -S- C-CH ₂ OH
		CH ₃
Aldicarb	2-methyl-2-(methylsulphonyl)propanol	O CH ₃
alcohol sulphone		CH ₃ -S- C-CH ₂ OH
		O CH ₃
Aldicarb	2-methyl-2-(methylthio)propionaldehyde	CH ₃
aldehyde		CH ₃ -S- C-CHO
		CH ₃
Aldicarb	2-methyl-2-(methylsulphinyl)propionaldehyde	O CH ₃
aldehyde sulphoxide		CH ₃ -S- C-CHO
		CH ₃
Aldicarb	2-methyl-2-(methylsulphonyl)propionaldehyde	O CH ₃
aldehyde sulphone		CH ₃ -S- C-CHO
		O CH ₃
Aldicarb acid	2-methyl-2-(methylsulphinyl)propionic acid	O CH ₃
sulphoxide		CH₃-S- C-COOH
		CH ₃
Aldicarb acid	2-methyl-2-(methylsulphonyl)propionic acid	O CH ₃
sulphone		CH₃-S- C-COOH
		O CH ₃

Designation	Chemical Name	Structure
	methanesulphonic acid	О
		СН₃-S-ОН
		0

Animal metabolism

The metabolic fate of aldicarb was studied in rats using five separate labelled compounds: *S*-methyl-¹⁴C, *tert*-butyl-¹⁴C, carbonyl-¹⁴C, *N*-methyl-¹⁴C and ³⁵S-methyl (Andrawes *et al.*, 1967; Knaak *et al.*, 1966). The pesticide was administered orally to adult females (Andrawes *et al.*, 1967) or males (Knaak *et al.*, 1966).

Within 5 days 87.5% and 6.6% of [³⁵S]aldicarb administered orally at 0.4 mg/kg was excreted by female rats in the urine and faeces respectively. Only a trace of aldicarb *per se* was found in the excreta, indicating very rapid degradation. The following metabolites were isolated from the urine and quantified by thin-layer chromatography, radioautography and liquid scintillation counting: aldicarb sulphoxide, aldicarb sulphone, aldicarb oxime, oxime sulphoxide, oxime sulphone, nitrile sulphoxide, nitrile sulphone and water-soluble products (Andrawes *et al.*, 1967; Knaak *et al.*, 1966). The relative concentrations of the metabolites excreted in the urine of female rats (Andrawes *et al.*, 1967) are shown in Table 2.

The rapidity with which aldicarb was converted to aldicarb sulphoxide *in vivo* indicated that the overall metabolism of the pesticide depended largely upon the fate of this metabolite. Two labelled preparations of aldicarb sulphoxide were administered orally to female rats and the rate of elimination and the nature of the metabolic products were determined in urine, faeces, and respiratory gases (Andrawes *et al.*, 1967). Analysis of the urine showed that the degradation of aldicarb sulphoxide in rats produced most of the metabolites detected after treatment with aldicarb (Table 3).

A similar study was conducted to determine the metabolism of aldicarb sulphone in rats using the S-methyl-¹⁴C preparation (Andrawes, 1977). Analysis of the urine revealed the same metabolic products as in the urine of rats receiving aldicarb or aldicarb sulphoxide (Andrawes *et al.*, 1967). In addition, several of the previously reported unidentified organo- and water-soluble metabolites were identified and their quantities determined (Table 4).

Female beagle dogs were dosed for 20 days with unlabelled aldicarb at a rate of 0.75 mg/kg/day, given a single dose of [S-methyl-14C]aldicarb on the 21st day and then fed unlabelled material for an additional ten days (Sullivan and Carpenter, 1968b). Elimination via the urine averaged 74% of the applied dose and was essentially complete in 11 days. Of the radioactivity recovered in the urine, 90% was found within the first 24 hours of administration of the radiolabelled compound. The metabolic products identified were essentially the same as in the rat.

Table 2. Relative concentrations of metabolites excreted in the urine during a one-week period by rats treated orally with [35]aldicarb.

Metabolites	Radioactivity (%) at days after treatment			ment
	1	3	5	7
Water-soluble	43.3	33.5	35.1	27.3
Aldicarb oxime	0.48	0.00	0.00	0.00

Metabolites	Radioactivity (%) at days after treatment			ment
	1	3	5	7
Unknown A	0.00	0.96	2.79	2.61
Oxime sulphoxide	14.7	9.33	6.01	4.31
Nitrile sulphoxide	6.22	8.99	2.36	2.00
Nitrile sulphone	1.23	42.4	51.2	61.3
Aldicarb	0.82	0.00	0.00	0.00
Aldicarb sulphoxide	31.8	4.78	2.56	2.48
Aldicarb sulphone	1.50	0.00	0.00	0.00

Table 3. Relative concentrations of metabolites excreted in the urine during a 4-day period by rats treated orally with radiolabelled aldicarb sulphoxide.

Metabolites	Radioactivity (%), at days after treatment			reatment
	1	2	3	4
Water-soluble	45.3	31.2	19.1	23.0
Oxime sulphoxide	16.4	13.1	5.29	4.53
Nitrile sulphoxide	8.17	22.7	14.6	6.99
Nitrile sulphone	1.66	16.8	47.8	52.3
Aldicarb sulphoxide	27.5	8.17	1.24	1.16
Aldicarb sulphone	0.15	0.13	0.09	0.04
Unknown B	0.81	7.93	11.9	12.0

Table 4. Relative concentrations of metabolic products excreted in the urine of rats treated orally with [*S*-methyl-¹⁴C]aldicarb sulphone (Andrawes, 1977).

Metabolites	Radioactivity (%) at days after treatment			reatment
	6	12	24	48
Free Metabolites				
Aldicarb sulphone	69.7	58.8	33.3	25.3
Methylol sulphone	2.3	2.3	1.8	0.3
Oxime sulphone	9.2	10.6	8.7	4.8
Nitrile sulphone	1.2	1.5	1.4	2.3
Amide sulphone	0.3	0.4	0.8	1.0
Alcohol sulphone	1.8	6.5	17.9	14.4
Conjugated metabolites				
Oxime sulphone	1.7	2.1	1.1	1.1
Alcohol sulphone	5.2	5.3	18.0	37.0
Aldehyde sulphone	0.8	0.6	4.4	0.2
Unknowns	0.2	0.8	0.1	1.3
Polar metabolites ¹	7.6	11.1	12.5	12.3

¹ TLC analysis showed this fraction to contain aldicarb sulphone acid and methanesulphonic acid

A lactating cow given a single dose of 0.1 mg/kg/bw of [35 S]aldicarb eliminated over 96% of the radioactivity within 540 hours (Dorough and Ivie, 1968). The percentages of the total dose detected in the urine, milk, and faeces were 90.2, 3.0 and 2.9 respectively.

[S-methyl-14C]aldicarb plus aldicarb sulphone (1:1 molar ratio) were fed to 3 lactating cows at levels of 0.12, 0.6 and 0.12 ppm in the diet for 14 days (Dorough et al., 1970). The proportions of the doses eliminated in the milk, urine and faeces (Table 5) were essentially similar to those found in the single-dose study described above. The radioactive metabolites in the milk and urine from each cow were determined quantitatively at 1, 3, 5, 7, 10, and 14 days after starting to feed the radiolabelled material. The average of these compounds in the milk of Cow 3, fed 1.2 ppm, over the fourteen days is given in Table 6. The relative amounts of the various metabolites were essentially constant with time, and there were no significant variations with dose. The metabolic products in the urine were largely (about 75%) water-soluble, and the organo-extractable products had a similar distribution to that in other animals. The animals were slaughtered 18 hours after the last treatment and twenty-eight tissues from each cow were analysed for radioactivity.

The total radioactive residues, as mg/kg aldicarb equivalents, in five important tissues and in the blood (taken daily and averaged for the 14-day period) are shown in Table 7. Milk production and food and water consumption were essentially constant over the 24 days. Cholinesterase activity in the plasma and red blood cells showed no deviation outside the normal limits over the entire period. Weight records for all the cows were as expected for confined animals.

Table 5. Elimination of radioactivity by cows fed [S-methyl-14C]aldicarb plus aldicarb sulphone (1:1 molar ratio) for 14 days.

Dose (ppm in the diet)	% of applied dose in		
	Milk	Urine	Faeces
0.12	0.9	93.8	3.5
0.6	0.9	91.6	3.0
1.2	1.3	92.1	2.9

Table 6. Average composition of residues in milk from a cow fed 1.2 ppm of radioactive aldicarb plus aldicarb sulphone daily for 14 days.

Metabolite	% of radioactivity in milk	ì g/kg aldicarb equivalents
Aldicarb sulphoxide	3.0	0.4
Aldicarb sulphone	17.2	2.3
Oxime sulphoxide	8.5	1.1
Nitrile sulphoxide	4.6	0.6
Oxime sulphone	7.1	0.9
Nitrile sulphone	27.9	3.7
Water-soluble	0.0	0
Milk solids	15.5	2.1
Unidentified (5)	16.2	2.1

Table 7. Aldicarb equivalents (mg/kg) in tissues and blood of cows fed aldicarb plus aldicarb sulphone.

Sample	Cow 1 (fed 0.12 ppm)	Cow 2 (fed 0.6 ppm)	Cow 3 (fed 1.2 ppm)
Liver	0.029	0.123	0.163

Sample	Cow 1 (fed 0.12 ppm)	Cow 2 (fed 0.6 ppm)	Cow 3 (fed 1.2 ppm)
Kidney	ND	0.006	0.016
Foreleg muscle	ND	0.003	0.004
Hind leg muscle	ND	0.002	0.004
Omental fat	ND	ND	ND
Blood (average over 14 days)	ND	0.008	0.017

ND = <0.004 mg/kg

An additional feeding study was conducted to determine the residue levels in milk and liver from feeding a 1:1 mixture of unlabelled aldicarb sulphoxide and aldicarb sulphone to lactating cows (Romine, 1973). Three lactating cows were used: one was held as a control and the other two were continuously fed a diet containing the mixture. One cow was fed for 32 days and the other for 46 days. The metabolites were fed initially at a level of 1.0 ppm, and upon equilibration of the residues in the milk (after ten days) the intake was increased to 3.0 ppm for nine days and then to 5.0 ppm (13 days for cow no. 2 and 27 days for cow no. 3). Milk samples were analysed daily until the residues reached a plateau. The samples were then analysed every two or three days. The results are summarized in Table 8.

Table 8. Aldicarb residues in milk following dietary feeding of 1.0, 3.0 and 5.0 ppm of a 1:1 mixture of aldicarb sulphoxide and aldicarb sulphone to lactating cows.

Feeding level (ppm)	Aldica	arb equivalents, mg/kg	, range
	Cow 1 (Untreated)	Cow 2	Cow 3
1.0	< 0.002	< 0.002	<0.002
3.0	<0.002	0.0035-0.004	0.0025-0.004
5.0	<0.002	0.006-0.0075	0.005-0.006

The total aldicarb residues in the milk were about 0.1% of the level of metabolites in the feed, which is in agreement with the value of about 0.1-0.2% obtained by Dorough *et al.* (1970) for the total radioactive metabolites in milk from feeding aldicarb. Aldicarb residues were not detected in liver (<0.01 mg/kg) at any of the feeding levels. No build-up of aldicarb residues with time was found as the continuous feeding study was extended to 46 days.

A study was conducted to determine the fate of aldicarb in goats and the nature and magnitude of the residues in urine, milk and tissues (Andrawes and Lee, 1986). Three lactating goats were acclimatized for seven days, after which two were treated with [S-methyl-14C]aldicarb, the third being a control. Radiolabelled aldicarb was administered in gelatin capsules at a level equivalent to 2.5 ppm in the feed (or 0.165 mg/kg bw) for ten days. Neither of the dosed goats displayed any toxic manifestations: the feed consumption, milk production and quantity of excretory products remained stable throughout the experiment. Only a slight reduction in the body weight of one treated goat (from 51 to 50 pounds) was noted. The latter could have resulted from confinement of the animal. The goats were slaughtered within 6-8 hours after the last dose so that high levels of ¹⁴C residues would be present in the tissues for identification and quantification of the metabolites.

Water-soluble conjugates of several of the hydroxylated metabolites were identified in the urine (Tables 9 and 10).

Table 9. Metabolic products found in the urine of goats treated with [S-methyl-14C]aldicarb at a level

equivalent to 2.5 ppm in the diet for ten days.

Metabolite	% of total ¹⁴ C	in samples at intervals	after treatment
	2 days	5 days	10 days
Aldicarb oxime	1.5	7.2	1.2
Aldicarb sulphoxide	3.6	2.6	3.4
Aldicarb oxime sulphoxide	7.9	13.7	6.8
Aldicarb nitrile sulphoxide	5.0	10.1	7.9
Aldicarb amide sulphoxide	1.1	2.0	1.8
Aldicarb sulphone	0.9	0.7	0.8
Aldicarb oxime sulphone	1.7	2.7	0.6
Aldicarb nitrile sulphone	3.6	14.8	11.9
Aldicarb amide sulphone	0.4	2.2	2.6
Aldicarb alcohol sulphone	0.1	0.2	0.1
Unknown	0.3	ND	ND
Water-soluble	74.5	44.2	63.2

ND = None detected

Table 10. Aglycones released by enzyme hydrolysis of water-soluble metabolites in 2-day goat urine.

Aglycones	% of total ¹⁴ C in sample
Aldicarb alcohol	0.4
Aldicarb oxime sulphoxide	7.5
Aldicarb nitrile sulphoxide	13.9
Aldicarb amide sulphoxide	0.4
Aldicarb alcohol sulphoxide	3.5
Aldicarb oxime sulphone	4.9
Aldicarb nitrile sulphone	10.7
Unknown	1.1
Origin of TLC	9.3
Unhydrolysed water-solubles	24.2
Total	75.9

The levels and nature of the residues found in milk and some tissues are shown in Tables 11 and 12.

Table 11. Metabolic products extracted from 5- and 10-day milk from goats treated with $[S-methyl^{-14}C]$ aldicarb at a level equivalent to 2.5 ppm in the diet for ten days.

	5 days am	5 days pm	10 days am	10 days pm
Aldicarb sulphoxide	0.02	0.16	ND	ND
Aldicarb oxime sulphoxide	0.14	1.75	0.15	0.24
Aldicarb nitrile sulphoxide	0.18	0.75	0.28	0.40
Aldicarb alcohol sulphoxide	0.05	0.20	0.04	0.15
Aldicarb sulphone	0.02	0.10	ND	0.01
Aldicarb nitrile sulphone	34.7	37.0	50.1	53.6
Aldicarb amide sulphone	1.19	1.09	1.24	1.60
Aldicarb alcohol sulphone	0.04	0.08	0.04	0.04
Aldicarb sulphone aldehyde	0.37	0.47	0.29	0.22
Unknown 1	0.02	0.15	0.02	0.03
Unknown 2	0.12	0.20	0.05	0.19
Origin of TLC	0.19	0.34	0.19	0.26
Water-soluble	7.95	13.3	10.6	10.1
¹⁴ C unextracted from milk solids by acetonitrile-water	13.0	12.5	12.0	12.2
Total	58.0	68.1	75.0	79.0

ND = None detected

Table 12. Levels and nature of residues extracted from tissues of goats fed [S-methyl-¹⁴C]aldicarb at a level equivalent to 2.5 ppm in the diet for ten days.

Metabolite				[¹⁴ C]a	ldicarb	equivalents	, ì g/kg, i	n		
	Liver	Kidney	Lung	Heart	Brain	Mammary gland	Leg muscle	Loin muscle	Periph- eral fat	Omental fat
Aldicarb oxime	ND	0.26	ND	ND	ND	ND	ND	ND	ND	ND
Aldicarb sulphoxide	1.48	0.34	0.06	0.03	ND	ND	ND	0.10	0.11	ND
Aldicarb oxime sulphoxide	ND	ND	ND	ND	0.20	ND	0.06	0.09	0.06	ND
Aldicarb nitrile sulphoxide	12.9	9.55	0.90	0.58	0.55	0.70	0.58	0.58	0 .35	0.07
Aldicarb amide sulphoxide	ND	0.33	0.09	0.02	0.04	0.20	0.10	0.08		ND
Aldicarb alcohol sulphoxide	0.65	0.87	0.26	0.36	1.00	0.23	0.62	0.68	0. 14 ¹	ND
Aldicarb sulphone	ND	0.12	ND	ND	ND	ND	0.04	ND	ND	ND
Aldicarb oxime sulphone	ND	0.04	0.03	ND	0.12	ND	0.12	0.16	ND	ND
Aldicarb nitrile sulphone	40.3	49.9	41.2	43.1	42.5	42.1	48.4	45.2	22.9	13.1
Aldicarb amide sulphone	1.82	3.00	1.66	1.11	1.23	1.20	1.28	1.68	0.14	ND
Aldicarb alcohol sulphone	0.10	0.54	0.16	0.13	0.28	0.09	0.19	0.17	ND	ND
Aldicarb sulphone aldehyde	0.44	0.51	0.76	0.38	0.26	0.74	0.30	0.42	0. 17	ND
Unknown 1	ND	ND	0.03	ND	0.05	0.05	0.04	ND	ND	0.06
Unknown 2	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Unknown 3	ND	ND	ND	ND	0.04	ND	ND	ND	0.03	ND
Origin of TLC	0.80	4.78	0.42	0.21	0.14	0.31	0.08	0.12	0.21	0.06
Water-soluble	152	73.3	67.4	11.7	11.0	22.4	7.76	7.38	1.00	ND
Unextracted ¹⁴ C ²	309	49.5	201	26.4	12.5	59.3	20.4	10.3	3.93	6.67
Total	519	193	314	84	70	127	80	67	29	20

¹ Average of aldicarb amide sulphoxide and aldicarb alcohol sulphoxide

ND = None detected

Ten hens were treated once with a 1:1 molar mixture of aldicarb and aldicarb sulphone at 0.66 mg/kg bw (Hicks $et\ al.$, 1972). Time sequence analyses of excreta and tissues showed 86% of the administered radioactivity could be accounted for in 7 to 10 days, with 0.007 to 0.061 mg/kg aldicarb equivalents in the eggs and 0.004 to 0.034 mg/kg in various tissues. Characterization of the metabolites in the faeces indicated that the metabolic pathway in poultry is similar to that in other animals; i.e. no aldicarb was observed, but aldicarb sulphoxide , aldicarb sulphone, oxime sulphoxide and other breakdown products were present.

In a continuous feeding study (Hicks *et al.*, 1972) laying white leghorn hens were given daily doses of a 1:1 molar mixture of aldicarb and aldicarb sulphone, each labelled with ¹⁴C in the *S*-methyl group. Three groups of six birds each were treated with 0.005, 0.05 and 1.0 mg/kg/day; on an average consumption of 80 g feed per day per bird, this corresponded to levels of 0.1, 1.0 and 20.0 ppm aldicarb equivalents in the feed.

A fourth group of six hens was maintained as controls and fed a normal diet of commercial laying mash with water *ad libitum*. The 28-day treatment consisted of seven days feeding with unlabelled pesticide followed by 21 days with the radioactive mixture, given in gelatin capsules twice

² Radioactivity remaining in tissue solids after acetonitrile-water extraction

daily. Body weight, feed consumption, egg production, faeces elimination and general health remained essentially constant. In each of the three treated groups, three of the birds were killed 12 hours after the end of feeding and the other three seven days after the last treatment. At the highest level fed, 85% of the total administered radioactivity appeared in the droppings and 5% was found in the eggs. Summarized results of the study are presented in Table 13. The levels of total radioactivity were too low to allow complete identification of the metabolites in the eggs and tissues at the lower dose levels. Characterization of the radioactivity in the eggs from the 20 ppm group is shown in Table 14.

Table 13. Average aldicarb equivalents (mg/kg) in eggs and tissues of laying hens receiving 0.1 ppm, 1.0 ppm and 20 ppm aldicarb equivalents in the feed.

Sample	12 ho	ours after treat	ment at	7 d	ays after treatm	ent at
	0.1 ppm	1.0 ppm	20.0 ppm	0.1 ppm	1.0 ppm	20.0 ppm
Eggs	0.006	0.072	0.79	a	0.014 b	0.23 b
Blood	0.015	0.072	0.76	a	0.030	0.34
Kidney	0.012	0.12	1.38	a	0.026	0.39
Liver	0.011	0.14	1.40	a	0.019	0.36
Breast	a	0.057	0.69	a	0.019	0.28

a: less than 0.005 mg/kg or undetectable

Table 14. Average composition of residues in eggs from chickens fed 20 ppm of a mixture of aldicarb and aldicarb sulphone for 28 days.

Metabolite	% of ¹⁴ C in eggs	mg/kg
Aldicarb sulphoxide	0.1	0.001
Aldicarb sulphone	0.2	0.002
Aldicarb oxime	0.2	0.002
Oxime sulphoxide	3.4	0.027
Oxime sulphone	3.8	0.033
Aldicarb nitrile	0.1	0.001
Nitrile sulphoxide	1.1	0.009
Nitrile sulphone	41.4	0.32
Unidentified	2.8	0.027
Water-soluble	15.4	0.12
Hexane-soluble	18.3	0.14
Solids	13.2	0.10

A study was recently completed according to US EPA re-registration requirements to determine the nature and levels of [*S-methyl-*¹⁴C]aldicarb residues in hen tissues and eggs following oral dosing (Byrd, 1994). Two groups of ten birds were dosed twice daily with [¹⁴C]aldicarb for seven consecutive days at a rate equivalent to 3.5 ppm in the diet and a third group of ten was maintained as controls. Excreta were collected once daily and eggs twice daily. The volatiles (¹⁴CO₂) expired by three of the birds were determined in one treated group.

b: eggs collected on the 7th day, not an average of eggs produced over the 7-day period.

At the end of the dosing period the birds were slaughtered and tissues collected for analysis. Approximately 66.9% of the total radioactivity administered was recovered in one group, and 81.8% in the other. The residues in the treated groups expressed as aldicarb equivalents are shown in Table 15.

The total aldicarb equivalents in yolks, whites and whole eggs are shown in Table 16.

The radioactivity in the edible tissues and eggs was distributed between the acetonitrile/chloroform fraction, hexane fraction, aqueous fraction and the post-extracted solids (PES). In all but the liver, the majority of the radioactivity was organosoluble (hexane or acetonitrile/chloroform fraction). In the edible tissues $\leq 22\%$ of the radioactivity remained in the PES fraction. However, the PES fraction in egg whites contained 32.2% of the total radioactivity.

Aldicarb nitrile sulphone was the most common free metabolite isolated in the organic extracts of liver, muscle, egg yolk and egg white and ranged in total concentration from 0.005 mg/kg aldicarb equivalent in egg yolk to 0.040 mg/kg aldicarb equivalent in muscle. It was the only free metabolite in muscle and egg yolk.

Table 15. Concentrations of ¹⁴C in tissues of hens fed [¹⁴C]aldicarb.

Tissue	Aldica	arb equivalents, m	g/kg
	Group 1	Group 2	Average
Muscle	0.092	0.095	0.094
Fat	0.030	0.024	0.026
Skin with fat	0.082	0.084	0.084
Gastro-intestinal tissue	0.18	0.17	0.17
Kidney	0.31	0.32	0.31
Liver	0.39	0.43	0.42
Gastro-intestinal contents	0.27	0.26	0.26
Red blood cells	0.12	0.12	0.12
Plasma	0.15	0.15	0.15

Sample		-	Aldic	arb equivale	ents, mg/kg,	at day		
	1	2	3	4	5	6	7	Mean
Yolks	0.014	0.029	0.057	0.090	0.12	0.15	0.19	0.09
Whites	0.043	0.081	0.11	0.12	0.13	0.14	0.16	0.11
Whole egg	0.032	0.062	0.09	0.11	0.13	0.15	0.17	0.10

Table 16. Concentration of total [14C]aldicarb equivalents in eggs.

In liver, aldicarb nitrile sulphone was present at a level of 0.028 mg/kg aldicarb equivalents (AE) (6.7% of the total radioactive residue, TRR). However, the major free metabolite in liver was aldicarb acid sulphone which was present at a level of 0.105 mg/kg AE (25.4 % TRR). Two additional minor free metabolites were isolated from the liver: methanesulphonic acid, 0.029 mg/kg AE (7.0% TRR) and aldicarb nitrile sulphoxide, 0.003 mg/kg AE (0.8% TRR).

In addition to the nitrile sulphone, egg white contained measurable levels of aldicarb oxime sulphone (0.003~mg/kg aldicarb equivalents, 2.8%~TRR) and aldicarb oxime sulphoxide (0.004~mg/kg AE, 3.3%~TRR). The radioactivity in the hexane fraction of egg yolk was tentatively identified as being incorporated into natural constituents.

The metabolic pathways proposed for aldicarb in animals are shown in Figure 1 below.

Plant metabolism

The metabolic pathway of aldicarb has been studied in potatoes (Andrawes *et al.*, 1970, 1971b), sugar beet (Andrawes and Bagley, 1969, 1970a,b; Rouchaud *et al.*, 1980, 1981), cotton (Andrawes *et al.*, 1970, 1973; Bartley *et al.*, 1970; Metcalf *et al.*, 1966), peanuts (Andrawes, 1972b), tobacco (Andrawes, 1972a), spearmint and lettuce (Andrawes and Bagley, 1968a). The Meeting was informed by the manufacturer that a new plant metabolism study would be conducted on potatoes and should be completed by the end of 1995. A discussion of individual crop studies is provided in the following sections.

The metabolism of [S-methyl-¹⁴C]aldicarb in potato plants was studied under greenhouse (Andrawes et al., 1970) and field (Andrawes et al., 1971b) conditions. After application of the radiolabelled pesticide to field soil at the rate of 3.4 kg ai/ha in-furrow at planting, the distribution of the total ¹⁴C residue in various plant parts was as shown in Table 17.

Table 17. Distribution of ¹⁴C from [*S-methyl*-¹⁴C]aldicarb in field-grown potato plants after in-furrow application at planting.

Plant part	[¹⁴ C]aldicarb	equivalents, m	g/kg, found at	% of	applied radioact	tivity at
	30 days	60 days	90 days	30 days	60 days	90 days
Foliage	4.7	6.7	4.4	0.56	3.3	2.04
Tubers	-	1.4	0.79	1	0.48	0.95
Roots	3.3	2.5	2.3	0.12	0.16	0.04

|--|

Figure 1. Metabolic pathways proposed for aldicarb in animals.

Thirty days after treatment the highest level of total aldicarb residues was found in the seed piece which contained 36.9 mg/kg aldicarb equivalents. The high level of residues might be expected since seed pieces were placed directly in the treated furrow.

The residues recovered from the potato foliage and tubers are shown in Table 18. Additional information on the nature of the water-soluble metabolites was obtained after treatment of immature tuber buds with the radiolabelled aldicarb (Table 19) (Andrawes *et al.*, 1971b).

Table 18. Radiolabelled components present in the foliage and tubers of potatoes after in-furrow atplanting application of [S-methyl-14C]aldicarb at 3.4 kg ai/ha.

Metabolic products	% of recovered radioacti	vity present at	¹⁴ C-aldicarb equiv	valents, mg/kg
	60 days	90 days	60 days	90 days
Foliage				
Water-soluble ¹	27.2	29.8	1.81	1.30
Aldicarb sulphoxide	22.9	6.6	1.53	0.29
Aldicarb sulphone	43.9	55.9	2.92	2.45
Oxime sulphoxide	0.9	1.1	0.06	0.05
Oxime sulphone	1.6	4.0	0.10	0.18
Origin of TLC	3.6	2.6	0.24	0.11
Tubers				
Water-soluble ¹	30.7	65.7	0.42	0.52
Aldicarb sulphoxide	33.4	4.6	0.46	0.03
Aldicarb sulphone	30.0	10.1	0.42	0.08
Oxime sulphoxide	1.6	11.3	0.02	0.09
Oxime sulphone	4.0	8.0	0.06	0.06
Origin of TLC	0.3	0.3	0.01	0.01

¹ Remaining in aqueous phase after chloroform extraction.

Table 19. Metabolism of [S-methyl-14C]aldicarb by potato tubers.

Metabolic products	% of recovered	d radioactivity
	Organosoluble	Water-soluble
Aldicarb	-	-
Aldicarb sulphoxide	51.6	-
Aldicarb sulphone	2.75	-
Oxime sulphoxide	8.77	0.241
Oxime sulphone	0.77	0.241
Nitrile sulphoxide	7.66	1.09
Nitrile sulphone	1.26	-
Alcohol sulphoxide	0.84	5.221
Alcohol sulphone	ND^2	5.631
Amide sulphoxide	2.57	0.49
Acid sulphoxide	ND ²	0.27

Metabolic products	% of recovered radioactivity		
	Organosoluble	Water-soluble	
Acid sulphone	-	-	
Unknown 1	-	0.27	
Unknown 2	-	0.27	
Unknown 3	-	0.68	
Unknown 4	-	1.19	
Unhydrolysed conjugate	0.43	7.78	

¹ As glycoside conjugate

The metabolism of [S-methyl-¹⁴C]aldicarb in <u>sugar beet</u> plants was studied under greenhouse (Andrawes and Bagley, 1969, 1970a,b) and field (Rouchaud *et al.*, 1980) conditions. In the greenhouse study the pesticide was applied at the rate of 22.4 kg ai/ha,broadcast and incorporated, to two groups of sugar beet plants. One group was in greenhouse artificial soil (peat moss-sand fertilizer mixture, C-mix) and the other in field Norfolk sandy loam soil. The rate of uptake of aldicarb by the plants grown in C-mix was five times that by the plants grown in field soil.

The distribution of the terminal residues found in the maturing sugar beet plants (Andrawes and Bagley, 1970b) is shown in Table 20.

Table 20. Radiolabelled components isolated from sugar beet plants treated with [*S-methyl-*¹⁴C]aldicarb at 22.4 kg ai/ha broadcast and incorporated.

Metabolic products		% of recovered radioactivity				
		Foliage		Roots		
	90 days	140 days	90 days	140 days		
Aldicarb	ND ¹	ND	ND	ND		
Aldicarb sulphoxide	13.5	9.8	12.2	12.7		
Aldicarb sulphone	25.4	30.8	8.8	11.3		
Oxime sulphoxide	0.21	0.10	1.2	1.6		
Oxime sulphone	1.1	1.2	0.34	3.5		
Nitrile sulphoxide	0.62	2.4	ND	1.2		
Nitrile sulphone	0.71	2.4	0.40	0.47		
Alcohol sulphone	8.9	12.4	1.9	4.5		
Origin of TLC	1.3	3.1	1.2	2.7		
Water-solubles	48.3	37.8	74.0	62.1		
Total (mg/kg)	18.3	27.2	2.7	2.5		

¹ None detected

The metabolic fate of aldicarb in <u>cotton</u> plants was studied in a series of greenhouse experiments, following uptake by excised leaves (Coppedge *et al.*, 1967; Metcalf *et al.*, 1966), injection into the petiole of intact leaves (Coppedge *et al.*, 1967), root uptake into young seedlings (Bartley *et al.*, 1970), stem application to large plants (Metcalf *et al.*, 1966) and soil application (Bartley *et al.*, 1970, Coppedge *et al.*, 1967). Under field conditions the metabolic fate was studied by treating

² None detected

individual leaves and stems (Bull, 1968), after soil application at planting, and as a side-dress treatment (Andrawes *et al.*, 1973).

The levels of the metabolic products found in cotton plants after soil application of 1.12 kg ai/ha in-furrow, and 1.12 kg ai/ha at planting plus 2.24 kg ai/ha side-dress 58 days after planting (Andrawes *et al.*, 1973) are shown in Table 21. The composition of the water-soluble fraction (Table 22) was determined in the foliage under greenhouse conditions (Bartley *et al.*, 1970).

Table 21. Radiolabelled components present in the foliage of field-grown cotton after in-furrow and side-dress applications of $[S-methyl-^{14}C]$ aldicarb.

Residue components				Residues	, mg/kg, a	ıt days afte	er treatme	nt		
	9	14	22	37	58	65	72	86	100^{2}	146 ²
1.12 kg ai/ha in-furrow					•	•	•	•		•
Aldicarb	2.2	1.1	1.0	0.4	T^3	T	T	T	Т	T
Aldicarb sulphoxide	147.6	146.8	45.3	13.0	2.5	2.1	0.7	0.2	0.7	0.4
Aldicarb sulphone	14.8	37.7	39.2	12.9	7.3	7.5	2.5	1.1	2.0	0.6
Oxime sulphoxide	0.4	1.1	1.1	1.1	1.2	1.3	0.7	0.2	0.6	0.5
Oxime sulphone	ND^4	1.0	1.4	0.7	0.3	0.4	0.2	T	Т	T
Nitrile sulphoxide	ND	4.8	4.7	2.0	0.1	0.2	0.1	T	Т	T
Nitrile sulphone	ND	ND	ND	Т	T	T	T	T	Т	T
Alcohol sulphone	ND	2.7	1.1	0.4	ND	ND	ND	ND	ND	ND
Origin of TLC	2.2	3.1	0.9	1.0	0.3	T	T	T	Т	0.2
Water-solubles	41.8	43.6	32.1	22.0	8.3	8.7	5.0	0.9	3.8	7.2
Total	209.0	241.9	126.8	53.5	20.0	20.2	9.2	2.4	7.1	8.9
1.12 kg ai/ha in-furrow	plus 2.24	ai/ha side-	-dress	•	•	•	•	•		•
Aldicarb						0.1	0.2	T	Т	T
Aldicarb sulphoxide						12.5	17.9	25.5	8.9	10.8
Aldicarb sulphone						7.2	5.7	16.2	11.7	13.1
Oxime sulphoxide						0.5	0.6	1.1	1.2	2.5
Oxime sulphone						0.1	0.2	0.7	0.5	0.5
Nitrile sulphoxide						T	0.6	2.9	ND	1.1
Nitrile sulphone						T	T	0.7	0.5	1.4
Alcohol sulphone						0.6	0.7	0.5	0.3	0.1
Origin of TLC						0.2	0.3	0.2	0.4	1.5
Water-soluble						12.1	12.9	22.5	19.3	80.8
Total						33.3	39.1	70.3	42.8	111.8

¹ 10% granular formulation applied at the rates shown. Side-dress application 58 days after planting

² Desiccation of foliage began at approximately 90 days

³ Less than 0.1 mg/kg

⁴ None detected

Table 22. Products of metabolism of ¹⁴CH₃S-labelled aldicarb administered by root uptake to cotton plants (Bartley *et al.*, 1970).

Compound	% of recovered radioactivity in extracts at days			
	30		6	0
	Organic	Aqueous	Organic	Aqueous
Aldicarb	1.7	-	-	-
Aldicarb sulphoxide	30.2	a)	12.3	a
Aldicarb sulphone	5.7	-	5.4	-
Oxime sulphoxide	2.1	2.0 b)	-	1.0 b)
Oxime sulphone	0.5	a)	4.6	a)
Nitrile sulphoxide	15.3	a)	10.3	a)
Alcohol sulphoxide	1.3	18.8 b)	-	28.5 b)
Alcohol sulphone	0.9	-	0.7	-
Amide sulphoxide	2.3	1.4	0.7	1.4
Acid sulphoxide		4.4	-	7.5
Acid sulphone	0.8 c)	0.8	-	2.3
Unknowns	-	11.8	0.6	24.7

- a) Minor amount remaining after extraction-value combined with organic
- b) As glycoside conjugate
- c) Acid sulphoxide plus acid sulphone

The uptake, distribution and metabolism of aldicarb in <u>tobacco</u> plants was studied under field (Andrawes, 1972a) and greenhouse (Khasawinah and Hirsh, 1976) conditions, with similar indications of the overall fate of the pesticide. The metabolites were qualitatively similar to those previously reported for other plant species. The small amount of aldicarb (77.8 ì g or 1.14% of the recovered radioactivity) present in the 14-day foliage, which became undetectable thereafter, is noteworthy.

The metabolism and associated residues of [S-methyl-¹⁴C]aldicarb in a 10G formulation were investigated in field-grown peanut plants. The pesticide was applied in one experiment banded and incorporated at planting at the rate of 6.72 kg ai/ha and in another as a side-dress treatment at the pegging stage at the rate of 62 mg ai per plant, equivalent to 6.72 kg ai/ha (Andrawes, 1972b).

Table 23 shows the distribution of the radiolabelled metabolites found in the foliage, roots, pegs, kernels and shells, and Table 24 the distribution of the extracted and unextracted radioactivity at various times after application.

The filter cake from the kernels after the side-dress treatment contained sufficient radioactivity for further testing. More than half of the filter cake residue was extracted by chloroform-acetonitrile (1:1 v/v). This extract was concentrated to an oily residue and reconstituted in acetonitrile from which 80 to 98% of the radioactivity was partitioned into hexane in association with the peanut oil.

The general metabolic profile of aldicarb in plants was described by Andrawes (1982), and the proposed metabolic pathways are shown in Figure 2 below.

Figure 2. Metabolic pathways proposed for aldicarb in plants.

Rotational Crop Studies

Two studies have been conducted to measure the uptake of residues of aldicarb by rotational crops (Hirsh and Sheets, 1977; Hunt, 1992).

The first study consisted in treating a Norfolk sandy loam soil with 5.6 kg ai/ha of [S-methyl-14C] aldicarb and ageing under field conditions for four and 12 months. At the end of these periods, a leafy vegetable (lettuce), a root crop (turnip) and a grain crop (barley) were planted separately in the treated soil. The crops were grown to maturity and analysed periodically. Of the applied [14C] aldicarb, approximately 60% was lost by degradation (as CO₂) and 20% by leaching during the four-month ageing period. Only traces of carbamate residues were detected in the four-month plantings. These ranged from 0.02 to 0.05 mg/kg in all the plant parts, except in barley straw which contained 0.72 mg/kg. Although radioactive residues were detected in the one-year plantings, none were carbamates.

In the second study, covering seven test sites in six states of the USA, Temik was applied to three primary crops, cotton, potatoes and sugar beet, according to registered uses and generally at the maximum use rate. After destruction of the primary crop, secondary crops typical of each growing region were planted back into the treated plots. The rotational crops used were carrot, onion, lettuce, broccoli, cucumber, cantaloupe, tomato, corn, alfalfa, oats, barley and wheat. Plant-back intervals ranged from five to twelve months from the last application.

Residues were found in most crops, especially at the early plant-back interval, but were generally below the limit of quantification (Hunt, 1992). The maximum detectable residues measured in rotational crops and the corresponding residues in soil at planting are summarized in Table 25.

Table 23. Compounds found in field-grown peanut plants 98 days after aldicarb was applied at planting at 6.72 kg ai/ha, banded and incorporated.

Compound	% of recovered radioactivity				
	Foliage	Roots	Kernels	Shells	Pegs
Aldicarb	ND ¹	ND	ND	ND	ND
Aldicarb sulphoxide	5.3	4.0	1.7	3.2	2.6
Aldicarb sulphone	15.1	2.6	3.3	7.1	5.6
Oxime sulphoxide	0.3	0.4	0.1	0.3	0.4
Oxime sulphone	2.8	1.0	0.3	1.5	1.4
Nitrile sulphoxide	0.9	0.7	0.8	1.2	1.8
Nitrile sulphone	0.9	0.7	2.0	2.6	1.9
Alcohol sulphone	6.7	1.2	1.1	2.5	3.1
Origin of TLC	3.1	7.5	0.5	1.2	0.6
Water-soluble	64.9	81.9	90.2	80.4	82.5
¹⁴ C-Aldicarb equivalents (mg/kg)	3.6	1.0	0.6	0.5	0.8

¹ None detected

Table 24. Uptake and 14 C-residue distribution in field-grown peanuts treated with 10G formulation of [S-methyl- 14 C]aldicarb at 6.72 kg ai/ha.

Days after treatment	Plant Part	m	mg/kg aldicarb equivalents			
			unextracted ¹	total	¹⁴ C	
6.7 kg ai/ha ir	corporated at planting					
21	Foliage	173	3.18	177	1.41	
	Roots	18.6	7.17	25.7	0.03	
	Total				1.44	
35	Foliage	127	3.65	131	2.76	
	Roots	12.5	5.00	17.5	0.05	
	Total				2.81	
56	Foliage	60.9	2.02	62.9	7.41	
	Roots	4.45	2.62	7.07	0.08	
	Total				7.49	
98	Foliage	3.56	0.45	4.01	6.26	
	Roots	0.98	0.85	1.81	0.12	
	Pegs	0.80	0.56	1.36	0.07	
	Kernels	0.62	0.42	1.04	0.06	
	Shells	0.51	0.54	1.05	0.07	
	Total				6.58	
126	Foliage	3.47	0.51	3.96	5.78	
	Roots	1.28	1.41	2.69	0.17	
	Pegs	1.53	0.72	2.25	0.11	
	Kernels	0.52	0.33	0.85	0.16	

Days after treatment	Plant Part	mg/kg aldicarb equivalents			% of applied ¹⁴ C
		extracted	unextracted1	total	
	Shells	0.19	0.39	0.58	0.09
	Total				6.31
6.7 kg ai/ha side	dress at pegging stage				
70	Foliage (green)	4.08			
	Foliage (desiccated)	27.4			
	Kernels (green)	2.72	1.78	4.5	
	Kernels (desiccated)	2.84	3.04	5.88	
	Shells (green)	0.75	1.2	1.95	
	Shells (desiccated)	1.42	2.3	3.72	

¹ Radioactivity remaining in the filter cake of plant tissues after three extractions with ethanol-water (1:1 v/v)

Table 25. Maximum aldicarb residues expressed as aldicarb sulphone found in rotational crops in the USA.

Primary crop	Residues in soil at planting of rotational crop, mg/kg	Residues in rotational crop 6-8 months after planting	
		Crop	Residues, mg/kg
Cotton	ND - 0.12	Carrot	0.03
	<0.04 - <0.06	Carrot	0.04
	<0.02 - <0.06	Wheat forage	0.11
		Wheat straw	0.04
	ND - <0.02	Alfalfa	0.06
Sugar beet	0.17 - 0.24	Alfalfa	0.07
	0.17 - 0.24	Barley forage	0.15
	0.17 - 0.24	Wheat forage	0.88
	0.13 -0.27	Wheat forage	0.70

Environmental fate in soil

The fate of aldicarb in soil has been extensively investigated under both laboratory and field conditions. Some representative studies are summarized below.

Coppedge *et al.* (1967) studied the fate of [³⁵S]aldicarb in three types of soil in the laboratory. The three soils varied considerably in organic matter content and pH (Houston clay on 4.2%, pH 8; Norwood silty clay loam on 1%, pH 8; Lakeland fine sand on 0.4%, pH 6.3), and in water-holding capacity and sand, clay and silt content. The approximate half-life of aldicarb was 9, 7 and 12 days in Houston clay, Norwood silty clay loam, and Lakeland fine sand respectively. Four weeks after treatment the soils contained 0.3% (Norwood) to 27.2% (Lakeland) of the applied dose as aldicarb.

Bull *et al.* (1970) conducted further laboratory studies on the fate of radiolabelled aldicarb in sand, loam, clay and muck soils maintained at different moisture levels (0, 50% and 100% of field capacity) and at pH values of 6, 7, and 8. No important differences could be attributed to pH within the range used, but the moisture level was a critical factor. Aldicarb was relatively stable in all the soils when dry, in sand at all moisture levels, and in loam with 50% moisture. The half-life of the total carbamate compounds exceeded 56 days. A moisture level of 50% was optimum for the oxidation of

aldicarb to its sulphoxide and sulphone in loam, clay and muck, and a moisture level of 100% caused a substantially faster rate of decomposition to non-toxic products in the same soils.

Under laboratory conditions, the rates of conversion and degradation of the three carbamates were well described by first-order reaction kinetics (Smelt *et al.*, 1978a,b,c). The rate constants were dependent on the type of soil, the temperature and moisture and the depth from which the sample was taken. In general, degradation was slower in the deeper layers than in the top layers of the soil. The conversion of aldicarb to its sulphoxide and sulphone decreased markedly at lower temperature and moisture content.

Other laboratory studies (Bull *et al.*, 1970; Coppedge *et al.*, 1967; Richey, 1972b; Supak, 1972; Supak *et al.*, 1977) demonstrated that the volatilization of aldicarb and its degradation products was influenced greatly by soil moisture; as the rate of evaporation of water increased, so did the volatilization of radioactivity from the treated soil. The influence of temperature and moisture on the rate of volatilization of aldicarb and its degradation products has been reported by Bull *et al.* (1970). At 25°C and 75°C, 17% and 48% of the applied [35S]aldicarb was lost, respectively, within 24 hours from dry sand. In wet sand (0.2 g water/g sand), the losses for the same temperatures and period were 8% and 89%. Loss of water from the sand closely paralleled the loss of radioactivity at both temperatures.

Losses of aldicarb by volatilization from Houston Black (calcareous) and Beaumont (acid) soils fortified with 1 mg of aldicarb per g soil were studied by Supak (1972, 1977). Carbamates present in the trapped vapours were determined by gas chromatography. Volatilization losses were generally depressed by the presence of water and elevated temperature, which enhanced the chemical decomposition rather than the volatility of aldicarb.

Co-distillation from the moist soil resulted in essentially insignificant losses of carbamate-containing residues, which ranged from 0.01% to 0.08% of the applied dose. As the soil moisture content approached that required for monolayer coverage of the soil surfaces, volatilization effectively ceased. At this point, aldicarb was weakly adsorbed to the clay-oriented water adhering to the external clay surface. Increasing the sample temperature from 23°C to 42°C decreased toxicant volatilization losses from all the systems described above. The inert surface of construction sand does not absorb or bind aldicarb, thus allowing more volatilization than from soils.

Bull *et al.* (1970) tentatively identified the volatile radioactivity as CO₂. A more detailed study (Richey *et al.*, 1977) using *S*-methyl-¹⁴C, *tert*-¹⁴C and *N*-methyl-¹⁴C-labelled aldicarb and a more refined trapping system showed conclusively that the volatile material from the treated soils was indeed ¹⁴CO₂. No carbamates were detected in the volatilized radioactivity.

When aldicarb was incubated in Muskingum silt loam soil (pH 5.4) under aerobic conditions for 30 days followed by 30 and 60 days of anaerobic incubation, its metabolic profile and rate of degradation generally resembled that of aldicarb that was left under continuously aerobic conditions (Sheets and Hirsh, 1976).

The degradation rates of aldicarb were studied in soil samples taken from above and below the soil water table at four locations in The Netherlands (Smelt *et al.*, 1983). Degradation of aldicarb, aldicarb sulphoxide and aldicarb sulphone was significant under both aerobic and anaerobic conditions. The rates of degradation of the sulphoxide and sulphone were faster under anaerobic conditions with half-lives of 5.1 to 131 days in four anaerobic subsoils.

Conversion rates in the aerobic soil layer above the water table were between one eighth and less than 1% of those in the water-saturated layers in the same soil profile. When soil from below the water table was incubated aerobically, the conversion of aldicarb sulphone was drastically reduced. The

opposite was found when an originally aerobic soil was incubated anaerobically. Autoclaving the incubation systems restarted the conversions.

A study of the degradation of aldicarb in aerobic soil was recently conducted according to US EPA Pesticide Assessment Guidelines Subdivision N, Section 162-1 (Das, 1990). A sandy loam soil containing [S-methyl-14C]aldicarb at a concentration of 10.5 mg/kg was incubated in the dark at 25°C. Samples were taken at 0, 1, 2, 3, 4, 5, 7, 14, 21, 30 and 60 days after treatment, extracted and analysed by HPLC. There were two major products, identified as aldicarb sulphoxide and aldicarb sulphone. In the organosoluble extract the sulphoxide reached a maximum concentration of 86.1% at day 14 and the sulphone a maximum concentration of 80.1% at day 21. An unidentified minor product reached a maximum concentration of 2.4% at day 30. Aldicarb was rapidly degraded under aerobic conditions with a calculated half-life of 2.3 days. This study confirmed the earlier soil degradation studies described above.

Experiments with multiple-¹⁴C-labelled aldicarb incubated with different soil types indicated extensive degradation of the molecule to ¹⁴CO₂ (Richey *et al.*, 1977). The rates of ¹⁴CO₂ evolution during the incubation showed a lag period (2 to 5 days) during which no appreciable evolution of ¹⁴CO₂ occurred. Such a phenomenon is a characteristic of the microbial degradation of pesticides.

The effects of soil micro-organisms on the degradation of aldicarb were studied with isolated organisms. Pure cultures of five common soil fungi grown in Czapek-Dox broth were tested for their degradation of aldicarb, and three of the five were tested against aldicarb sulphoxide, the major toxic metabolite (Jones, 1976). The fungi, in decreasing order of their effectiveness in degrading the pesticide, were *Gliocladium catenulatum* > *Penicillium multicolor* = *Cunninghamella elegans* > *Rhizoctonia* sp. > *Trichoderma harzianum*. Aldicarb sulphoxide and water-soluble metabolites were the major products of metabolism. Traces of aldicarb sulphone, aldicarb oxime sulphoxide, aldicarb nitrile sulphoxide, aldicarb oxime sulphone and aldicarb nitrile sulphone were also detected in the organosoluble fraction. The water-soluble metabolites consisted of aldicarb alcohol sulphone and aldicarb amide sulphone as the major products, with moderate amounts of aldicarb alcohol sulphoxide and aldicarb amide sulphoxide, and small amounts of acids presumably derived from aldicarb sulphoxide and aldicarb sulphone. The metabolic pattern of aldicarb sulphoxide was similar to that of aldicarb. The results indicate that the ability to metabolize aldicarb and its sulphoxide is common in soil fungi. Further, the transformation pathway in the fungi appeared to be similar to that in soil, plants and animals.

Environmental fate in water

Aldicarb, being a carbamic acid ester, is susceptible to hydrolysis. The rates of hydrolysis and the nature of the products have been studied in sterile buffer solutions to exclude any microbial action. Aldicarb and its carbamate metabolites, aldicarb sulphoxide and aldicarb sulphone, were found to be stable under acid and neutral (pH 5-7) conditions (Andrawes, 1976a,b,c; Heywood and Barkley, 1965; Lykins, 1969a; Supak, 1972; Tobler, 1970). At pH 8 to pH 9 these carbamates were degraded mainly to their corresponding oximes and nitriles (Andrawes, 1976a,b,c; Tobler, 1970). At pH 9 and 25°C the half-lives of aldicarb, aldicarb sulphoxide and aldicarb sulphone were 74.7, 2.3 and 0.9 days respectively (Andrawes, 1976a,b,c). The order of reactivities appears to follow that of the acidities of the corresponding oximes (Kurtz and Asbury, 1977). The less acidic the oxime, the more stable its methylcarbamoyl derivative.

The hydrolysis half-lives of a 1:1 mixture of aldicarb sulphoxide and sulphone were found by Hansen and Spiegel (1983) to be as follows.

Temperature, °C	pH 5.5	pH 7.5	pH 8.5
5	865	640	50
15	445	240	7

The rates of hydrolysis were further studied at various temperatures and pH values (Andrawes, 1976a; Lemley and Zhong, 1983; Lykins, 1969a; Romine and Chancey, 1982; Speigel, 1982a,b; Stephen, 1969).

The photolysis of aldicarb, aldicarb sulphoxide and aldicarb sulphone in aqueous solutions has been studied in the presence and absence of a triplet sensitizer (Andrawes, 1976d,e,f). In general, the presence of a sensitizer had a minimal effect on the rate of photolysis of the three carbamates. Aldicarb was more susceptible to UV (290 nm) irradiation than the sulphoxide and sulphone. The half-life of aldicarb was 8 to 12 days and aldicarb sulphone 36 to 38 days. In contrast, aldicarb sulphoxide was stable to UV irradiation with only 2% degradation in 14 days.

A study of photodegradation in water was carried out according to the requirements of the US EPA Pesticide Assessment Guidelines Section N: 161-2 (Das, 1991). Sterile water buffered at pH 5 containing [S-methyl-14C]aldicarb at 10.6 mg/kg was exposed to artificial sunlight (510 watts/m², comparable to natural sunlight 548.8 watts/m²) for a total period of 360 hours of continuous radiation. The test solutions were incubated at 25°C.

Samples were analysed by HPLC. The concentrations of aldicarb in non-irradiated solutions changed insignificantly, remaining within the range 95.0 to 98.4% of the original. The concentrations in irradiated solutions decreased from 98.4% to 0.4% of the total radioactivity in 168 hours.

There were two major products: aldicarb oxime, which reached a maximum of 64.6% of the total activity by 168 hours, and aldicarb nitrile which reached a maximum of 48.2% by 360 hours. The identities of the parent and the two major products were established by HPLC and LC-MS and/or GC-MS.

When the period of continuous irradiation was expressed in terms of 12-hour photoperiods ("natural days"), the calculated half-life under irradiated conditions was 4.1 days, each day having 12 hours of irradiation.

Aldicarb is incorporated in soils for pest control and no residues are deposited on the soil surface. Studies of photolysis on soil surfaces have therefore not been conducted.

Only summary information was available on the mobility of the compound in soil (WHO, 1991). It indicated that aldicarb, aldicarb sulphoxide and the sulphone were mobile to different extents. The composition of the residues and their concentrations depended on the soil characters, pH, extent of leaching, and temperature (Coppedge *et al.*, 1977; Bowman, 1988).

Woodham *et al.* (1973a) studied the lateral movement of aldicarb in sandy loam soil. Temik 10G was applied to irrigated and non-irrigated fields at a rate of 16.8 kg/ha at 12.5-15 cm depth between rows of cotton seedlings. Soil samples were collected throughout the growing season from a depth of 15 cm of the treated soil, from the bottom of a creek adjacent to the treated field, and from sites 0.4 and 1.61 km downstream. The aldicarb residue fell to 15% within one month, and no residue was found after four months. No aldicarb was detected in the creek that collected the water drainage.

In a three-year study in a Wisconsin potato field (sandy plain) Fathula *et al.* monitored aldicarb residues in the saturated ground-water zone under fluctuating conditions of temperature, pH, and total hardness of the water. Soils were well-drained sands, loamy sands and sandy loams (with 1-2% organic

matter content). The water table was high, with a depth to the saturated zone between 1.3 and 4.6 m. Sampling wells were bored to a maximum of 7.5 m for ground-water sampling. The results indicated that the presence and persistence of aldicarb residues were dependent on alkalinity and temperature. The movement of residues was lateral as well as vertical. Pacenka et al. (1987) sampled both soil and ground-water from sites on Long Island (New York, USA), where earlier surveys had detected aldicarb and its sulphoxide and sulphone. Three study sites were chosen with shallow (3 m), medium (10 m) and deep (30 m) water tables. All were overlain with sandy soils. Soil cores, driven to the depth of the water table, were taken from a field where aldicarb had been applied to potatoes and from surrounding areas. Ground-water was sampled from 188 wells of varying depth and at different distances from the treated area. The results indicated that the residence time of aldicarb (including the sulphoxide and sulphone) in soil depended on the depth of the water table, and hence the overlying unsaturated zone. In the sites with shallow and medium depth water tables, all aldicarb residues disappeared within three years of the last use of the compound. With deeper unsaturated layers, aldicarb was present at increasing concentrations in soil water from 10 m down to the water table at 30 m. The uppermost 10 m was free of residues. Analysis of ground-water samples showed lateral movement of residues extending 120 to 270 m "downstream" of the source in a single year. It was calculated that the relatively shallow aquifer in the area would flush residues completely from the area over a very long period of time (≤ 100 years).

METHODS OF RESIDUE ANALYSIS

Analytical methods

The toxicologically significant residues containing the carbamoyl group (aldicarb, aldicarb sulphoxide and aldicarb sulphone) are usually extracted from plant foliage, fruits and vegetables with mixed solvents consisting of acetone/water, methanol/water, or dichloromethane/acetone (3:1). Oil is dissolved in hexane and the residues are partitioned into acetonitrile. Soil is extracted with water. Water samples are analysed directly after concentration.

Two methods are commonly used for the determination of aldicarb residues: gas chromatography and HPLC.

<u>Gas chromatography</u>. The extracted residues are oxidized to aldicarb sulphone by adding peracetic acid to the extracting solvent. After clean-up of the extract on a Florisil column, the pesticide residues are determined as aldicarb sulphone by gas chromatography using a flame-photometric detector. The residue is quantified by reference of the peak height or area to a calibration curve derived from the injection of standard solutions of aldicarb sulphone. The limit of determination of the method is approximately 0.02 mg/kg (Rhône-Poulenc, 1988a). In peanut oil an LOD of 0.001 mg/kg was reported (Rhône-Poulenc, 1988e).

<u>High performance liquid chromatography (HPLC)</u>. This procedure is applicable to the determination of total carbamate-containing aldicarb residues in certain fruits and vegetables, soil, and water. The method involves post-column reaction and fluorescence detection. With a C18 reverse-phase column and step gradient elution, the character of the aldicarb residue can be determined because the three carbamates are eluted separately and can be detected and quantified as discrete peaks. The post-column derivatization is based on alkaline hydrolysis to methylamine which is reacted with *o*-phthalaldehyde and mercaptoethanol to yield the fluorescent product 1-(2-hydroxyethyl)thio-2-methylisoindole. The limit of determination for each analyte is about 0.01 mg/kg in plant materials (Rhône-Poulenc, 1990b, 1991a), 0.001 mg/kg in soil (Rhône-Poulenc, 1990a) and 0.1 i g/kg in ground-water (Rhône-Poulenc, 1989c,d).

A list of the currently available published and unpublished methods for the determination of aldicarb, aldicarb sulphoxide and aldicarb sulphone in a variety of agricultural and environmental

substrates is given below. Many of the older Union Carbide methods and published methods for crops have been replaced by the general GLC and HPLC methods described above, but are nevertheless included since they have been referenced in many of the older residue studies.

Enforcement Methods

EPA multi-residue method (Ver Hay, 1992);

German multi-residue methods (DFG Method No. 250-1; DFG Method No. S 25-1.)

Dutch multi-residue method (Ministry of Welfare, 1988a, 1988b)

Published methods

Anon (1973)

Beckmann et al. (1969)

Carey and Helrich (1970)

Unpublished specific methods

Bananas: Union Carbide Corporation (1976a); Rhône-Poulenc (1991) Beans (Dry beans, soya beans): Union Carbide Corporation (1976c)

Coffee: Union Carbide Corporation (1977a)

Citrus pulp: Rhône-Poulenc (1991) Juice: Rhône-Poulenc (1988b)

Cotton seed: Union Carbide Corporation (1971) Cotton seed oil: Union Carbide Corporation (1968)

Grapes: Union Carbide Corporation (1978)

Milk: Rhône-Poulenc (1989a) Peanuts: Rhône-Poulenc (1989b) Peanut Oil: Rhône-Poulenc (1988e) Potatoes: Rhône-Poulenc (1990b).

Processed potato fractions: Union Carbide Corporation (undated a, 1969d)

Soil: Rhône-Poulenc, (1988f, 1990a)

Soya beans: Union Carbide Corporation (1976c)

Sugar beet: Union Carbide Corporation (1969a, 1969c, 1969b, undated b,c, 1969e)

Tobacco: Union Carbide Corporation (1976b,d); Lykins, (1969b, 1972)

Tomatoes: Union Carbide Corporation (1977b)

Water: Rhône-Poulenc (1988d); Tammara and Gustafson, (1992)

Stability of residues in stored analytical samples

Studies of the stability of residues in stored analytical samples have been conducted over several years on a range of substrates.

Data were provided on the stability of residues of aldicarb in frozen citrus fruits for nine months (Union Carbide Corporation, 1976; Tew, 1994), frozen milk and beef liver for six months (Hudson and Romine, 1986), frozen potato processed fractions for six weeks (Tew, 1992a), and frozen soya bean processed fractions for six weeks (Tew, 1992b). These were to support residue studies conducted for US registrations.

USE PATTERN

Aldicarb is a systemic insecticide, nematicide and miticide, available commercially only as low-assay (50, 100 or 150 g/kg) granular formulations. The granules are applied as seed furrow, band, or overall treatments (either pre-plant or at planting), and as post-emergence side-dress treatments at rates from 0.34 to 11.25 kg ai/ha, depending on the crop and the pests to be controlled. The granules must be

incorporated into soil immediately after application. Soil moisture is required to release the active ingredient from the granules, so irrigation or rainfall should follow application.

Aldicarb is rapidly absorbed by the plant's root system and moves throughout the plant primarily in the xylem. Pests feeding on foliage ingest enough aldicarb to kill them. This process, from application to effective control, takes about 24 hours or less depending on, for example, whether there is enough moisture to release the aldicarb, and whether the plant is actively growing and able to absorb the aldicarb rapidly. The length of residual activity (several weeks) depends on factors such as the pest susceptibility and the rate and timing of the application.

As a nematicide, aldicarb kills nematodes on contact in the soil and by systemic action as the nematodes feed on root tissues. Additionally, aldicarb can prevent nematodes from locating roots by interfering with the nematode sensory system. Aldicarb may also prevent reproduction through disorientation of male nematodes so that they cannot locate the females.

Some of the major pests controlled are listed in Table 26.

Table 26. Some of the major pests controlled by aldicarb.

Pest group	Species
Aphids	Aphis spp., Rhopalosiphum maidis, Acyrthosiphon solani, Schizaphis graminum, Myzus persicae, Macrosiphum euphorbiae, Adelges spp.
Beetles	Anthonomus grandis and A. vestitus (Boll weevils) Oulema melanopa (Cereal leaf beetle) Leptinotarsa decimlineata (Colorado Potato Beetle) Epitrix spp. (Flea beetles) Agriotes spp. (Wireworms)
Leafhoppers	Macrosteles fascifrons (Aster leafhopper Empoasca fabae (Potato leafhopper) Circulifer tenellus (Beet Leafhopper)
Leafminers	Nepticula gossypii (Cotton leafminer) Pegomya betae (Sugar beet leafminer) Liriomyza spp. and Phytomyza spp. (Vegetable leafminers)
Mealybugs	Planococcus citri (Citrus mealybug) Pseudococcus spp. (Greenhouse mealybugs)
Mites	Bryobia rubioculus (Brown mite) Panonychus citri (Citrus red mite) Phyllocoptruta oleivora (Citrus rust mite) Panonychus ulmi (European red mite) Tetranychus cinnabarinus (Carmine spider mite) Tetranychus urticae (Two-spotted spider mite)
Nematodes	Dolichodorus spp. Ditylenchus spp. (Bulb and stem nematodes) Radopholus similis (Burrowing nematode) Tylenchulus semipenetrans (Citrus nematode) Heterodera spp. (Cyst nematodes) Xiphinema spp. (Dagger nematode) Pratylenchus spp. (Lesion nematodes) Longidorus spp. (Needle nematodes) Rotylenchulus reniformis (Reniform nematode) Meloidogyne spp. (Root Knot nematodes) Helicotylencus spp. and Rotylenchus spp. (Spiral nematodes) Trichodorus spp. (Stubby Root nematodes)
Plant bugs	Lygus spp. (Lygus bugs) Stephanitis spp. (Lace bugs)

Pest group	Species
Psyllids	Psylla pyricola (Pear psylla) Paratrioza cockerelli (Potato psyllid)
Scale	Coccus spp.
Thrips	Scirtothrips citri (Citrus thrips) Frankliniella spp. Thrips tabaci (Tobacco thrips)
Whiteflies	Aleurothrixus spp. (Citrus blackfly, Woolly whitefly) Dialeurodes citri (Citrus whitefly) Trialeurodes vaporariorum (Greenhouse whitefly) Bemisia tabaci (Sweet potato whitefly)

The typical recommended application is by soil treatment in band or furrow, drilling 5-7.5 cm below the seed line at planting or sowing. In orchards the granules may be applied in a band along the dripline on both sides of or around the trees, or spreading the granules uniformly around them. Application must be followed by immediate and complete incorporation into soil to a depth of about 30-80 mm. Incorporation to a greater depth than 100-150 mm may reduce the efficacy.

The number of applications is usually restricted to one per year in food- and feed-producing plants except bananas, coffee, cotton, macadamia nuts and potatoes.

The timing of applications depends upon the crop, as follows.

In citrus: just before or during the spring flush of foliage growth.

In grapes: just before bud swell in a band, or in the autumn after harvest.

In dry beans, cereals, garlic, onions, peanuts, soya beans, sugar cane and sweet potatoes:

in seed bed or furrow immediately before sowing or at planting.

In cotton: before or at planting; at pin-head squaring as side-dressing.

In potatoes: at planting in furrow, or in emergency in 10-15 cm band over row on top of hill, side-dress

granules on both sides of plant row and 10 cm deep.

In sugar beet: at planting or shortly (1 week) before planting.

In macadamia nuts: at petal fall and 6 weeks later. In pecans: during period from bud break to nut set.

The registered uses of aldicarb around the world are shown in Tables 27-31.

Table 27. Registered uses of aldicarb on fruits.

Crop	Country			Application	1	PHI, days
		No.	Rate per application			
			kg ai/ha	g ai/tree	g ai/100 m row or g ai/m ²	
Bananas ¹	Argentina	2	4.5	1-3		90
	Cameroon	2		2		180
	Egypt	2	2-5			
	Ethiopia	2		2		No PHI
	France	2	4			180
	Ivory Coast	2	4	2		180
	South Africa	2		2.5-3		No PHI
	Portugal					28
	Spain			2-3		100
	Zimbabwe	2		1.5		No PHI
Citrus (bearing)	Argentina	1		22		90
	Australia ²	1	2.1-11.5			180
	Belize ³	1	5.5-10	22.5		
	Brazil	1		19.5		60
	Chile	1		19.5-30		30
	Colombia	1		9-15		30
	Costa Rica	1	5.6			30
	Cyprus	1	4.2-5.25			No PHI
	Egypt	1	6.0			No PHI
	El Salvador	1	5.6-11.25			No PHI
	Greece	1	5.5-8.0	12.5-20		45 (lemons); 120 (oranges, tangerines
	Guatemala	1	5.6-11.25			No PHI
	Honduras	1	5.6			30
	Italy Jamaica ³	1 1	6.0-10.0			180

Crop	Country	Country Application					
		No.	Ra				
			kg ai/ha	g ai/tree	g ai/100 m row or g ai/m ²		
	Mexico	1		30-45		90	
	Morocco	1	7.5-10.5			No PHI	
	Nicaragua	1	5.6-11.25			No PHI	
	Pakistan	1		37.5		No PHI	
	Panama	1	5.6			30	
	Peru	1		15-22.5		No PHI	
	Portugal	1		10-20		14	
	South Africa	1			1.9 g ai/m ²	100 (lemons) 150 (other citrus)	
	Spain	1		15-20		45 (lemons) 100 (others)	
	Uruguay	1		25		No PHI	
	USA	1	5.6			30 (lemons)	
	Venezuela	1		9-15		90	
Citrus (non-bearing)	Australia	NS ⁵	10.5		1.05 g ai/m ²	No PHI	
	Belize	1	0.7-4.0	3-9		No PHI	
	Brazil	1		19.5		60	
	Chile	2			1.5 g ai/m ²	No PHI	
	Israel	1		0.9-6		90	
	South Africa	4			1.9-4.5 g ai/m ²	No PHI	
Grapes (bearing)	Australia	1	2.25			133	
	Egypt	1	4.7			No PHI	
	Peru	1	3-3.75			No PHI	
	South Africa	1			0.75 g ai/m ²	120	
Grapes (non-bearing)	Chile	2	3-3.75	1.5-3		No PHI	
	France	1	20			No PHI	
	Portugal	1	20			No PHI	
Olive	Peru	NS ⁵		35-45		No PHI	
Pineapples	South Africa	1	3.75-4.05			No PHI	
Stone fruit (non-bearing)	Chile	2		1.5-3		No PHI	
	Israel	1		1.05-3		90	
	Mexico	1		45-90		No PHI	
Strawberries (non-bearing)	Ireland ⁴		4			No PHI	
	Netherlands	2-3	3		0.2 g ai/m row	No PHI	
	Poland					No PHI	
	UK		2.8-4			No PHI	

Table 28. Registered uses of aldicarb on vegetables.

Crop	Country		_	Application	PHI, days
		No.	R	ate per application	
			kg ai/ha	g ai/100 m row or g ai/m ²	
Beans (dry)	Argentina	1	0.5		60
	Bolivia	1	0.9-1.95		80
	Brazil	1	1.0-2.0		80
	Costa Rica	1	1.5-1.7		90
	El Salvador	1	1.2-2.25		60
	Guatemala	1	1.2-2.25		60
	Honduras	1	1.5-1.7		90
	Ireland ¹	1	1.0		84
	Mexico	1	0.75-1.2		90
	Nicaragua	1	1.2-3.3		90
	Panama	1	1.5-1.7		90
	UK ¹	1	1.0		84
	USA	1	0.6-2.35		90
Brassicas (Brussels Sprouts, Cabbage, Calabrese, Cauliflower)	Ireland	1		5.1 g ai/100 m row	70
	UK	1		5.1 g ai/100 m row	70
Brassica seed beds	Ireland	1	3.36		No PHI
	UK	1	3.36		No PHI
Brussels sprouts	Netherlands	1	3		No PHI
Carrots	Ireland	1	3.36	3.8 g ai/100 m row	84
	UK	1	3.36	3.8 g ai/100 m row	84
Garlic	Argentina	1	0.5-3		70
	Ireland ¹	1		0.77 g ai/100 m row	No PHI
	Israel	1	3.9		90
	UK ¹	1		0.77 g ai/100 m row	No PHI
Onion	Argentina	1	0.5-3.0		70
	Ireland (Spring and Autumn sown)	1	3.36	2.6-7.7 g ai/100 m row	No PHI ³
	Israel	1	3.9		90
	Netherlands (seed onion)	1	1.5	0.5 ai/100 m row (33 cm row)	No PHI
Parsnips	UK (Spring and Autumn sown)	1	3.36	2.6-7.7 g ai/100 m row	No PHI ³
Peppers	Argentina	1	1.0-3.0		60
Potatoes	Argentina	1	1.0-2.0		90
	Belgium	1	1.0		No PHI
	Brazil	1	1.95-3.9		No PHI
	Canada ⁵	1	1.1-2.25	10-20 g ai/100 m row	90
	Chile ⁶	1	1.05-3.0		90/50
	Colombia	2	1.5-3.0		90

Crop	Country			Application	PHI, days
		No.	R	ate per application	·
			kg ai/ha	g ai/100 m row or g ai/m ²	
	Cyprus	1	4.5-5.25		No PHI
	Czech Republic	1	1.5-5.0		No PHI
	Egypt	1	3.0		No PHI
	El Salvador	1	1.2-3.3		50
	Greece	1	1.2-2.5		90
	Guatemala	1	1.2-3.3		50
	India	1	2.0		60
	Ireland	1	2.24-3.36	4.3-12.8 g ai/100 m row	56
	Israel (for seed)	1	1.95		No PHI
	Italy	1	1.0-1.5		90
	Italy (for seed)	1	5.0		90
	Mexico	1	2.1-3.0		90
	Netherlands ⁷	1	0.75-3.0		No PHI
	Pakistan ⁸	1	1.2		No PHI
	Peru	2	1.5-2.25		No PHI
	South Africa (table potatoes)	1	2.55-5.25	25.5-52.5 g ai/100 m row	120
	South Africa (seed potatoes)	1	7.5	75 g ai/100 m row	120
	Spain	1	1.5-3.0		100
	UK	1	2.24-3.36	4.3-12.8 g ai/100 m row	56
	Uruguay	1	1.0-5.0		90
	USA	2	1.12-3.36		90/50
	Venezuela ⁶	1	2.0		90/50
Soya beans	El Salvador	NS	0.84-3.36		90
-	Guatemala	NS	0.84-3.36		90
	Mexico		0.75-1.2		90
	Nicaragua		0.84-3.36		90
	Panama		0.84-3.36		90
	USA	1	0.84-5.55		90
	Venezuela		2-3		90
Sugar beet	Belgium	1	0.5-1.0		No PHI
	Canada	1	1.1	10 g ai/100 m row	90
	Chile	1	1.5-3.0		No PHI
	Czech Republic	1			No PHI
	Egypt	1	3.2		No PHI
	France	1	1.0		No PHI
	Germany	1		4 g/100 m	No PHI
	Greece	1	0.5-1.6	-	90
	Hungary	1	1.2-1.8		No PHI
	Ireland	1		2.6-5.1 g ai/100 m row	No PHI
	Italy	1	1.0-2.0	, , ,	No PHI

Crop	Country			Application	PHI, days
		No.	R	•	
			kg ai/ha	g ai/100 m row or g ai/m ²	
	Netherlands	14	0.6-2.5		No PHI
	Poland	1	3.0		150
	Spain	1	1.0		100
	Switzerland	1	1.5-2.3		No PHI
	UK	1		2.6-5.1 g ai/100 m row	No PHI
	Uruguay	1	1.0-5.0		No PHI
	USA	1	1.2-4.5		90 120 ⁸
Swedes and turnips	Ireland	1		3.8 g ai/100 m row	70
	UK	1		3.8 g ai/100 m row	70
Sweet potatoes	Argentina	1	1.0-1.5		120
	Costa Rica	1	1.7		75
	El Salvador	1	1.7-3.3		90
	Guatemala	1	1.7-3.3		90
	Honduras	1	1.7		75
	Panama	1	1.7		75
	USA	1	1.7-3.36		120
Tomatoes	Argentina	1	1.0-3.0		60
	Chile	1	1.05-3.0		90
	Egypt		4.8		No PHI
	Ireland ¹	1			42
	Italy	1	1.5-2.0		110
	South Africa	1	3.0	22.5-45 g ai/100 m row	80
	UK ¹	1			42

Approved use pattern, but voluntarily withdrawn from label
 Apply before planting
 Do not harvest onions until they have reached the mature bulb stage

⁴ At sowing
⁵ Aldicarb is registered for use on potatoes but is not currently marketed for that use
⁶ Only one full application per crop. Apply full rate at sowing (PHI 90 days) or at earthing up (PHI 50 days), or apply half at sowing and half at earthing up

7 Apply before planting for ware, starch and seed potatoes

8 For livestock feed

Table 29. Registered uses of aldicarb on grasses

Crop	Country			Application	PHI, days
		No.		Rate per application	
			kg ai/ha	g ai/100 m row or g ai/m ²	
Cereals	Argentina	1	0.3-0.5		60
	Australia	1 ¹	0.375-0.54		No PHI
	France	1	1.0		No PHI
Maize	El Salvador	1	0.95-2.55		90
	France	1	0.5		No PHI
	Guatemala	1	0.95-2.55		90
	Honduras	1	0.95-2.55		90
	Ireland ²	1		2.6-3.8 g ai/100 m row	No PHI
	Nicaragua	1	1.2-3.3		90
	Panama	1	0.95-2.55		90
	South Africa	1	0.7-1.0	10.5-15 g ai/100 m row	56
	UK ²	1		2.6-3.8 g ai/10 m row	No PHI
Sorghum	El Salvador	1	1.2-1.95		90
	Guatemala	1	1.2-1.95		90
	Honduras	1	1.2-1.95		90
	Nicaragua	1	1.2-3.3		90
	Panama	1	1.2-1.95		90
	USA	1	0.6-1.2		90
Sugar cane	Argentina	1	1.5-3.0		70
	Australia	1	2.55		120
	Burkino Faso	1			
	Costa Rica	1	1.7		100
	El Salvador	1	2.25-3.3		120
	Guatemala	1	2.25-3.3		120
	Honduras	1	1.7		100
	Mexico	1	2.1-4.5		90
	Nicaragua	1	2.25-3.3		90
	Panama	1	1.7		100
	South Africa	1	2.25-3.0	27-36 g ai/100 m row	180
	Thailand	1	2.2-3.4		No PHI
	Uruguay	1	1-2		120
	USA	1	2.35-3.36	120	120

¹ Apply into seed furrow at seeding ² Approved use but voluntarily withdrawn from the label

Table 30. Registered uses of aldicarb on nuts and seeds.

Crop	Country			Application		PHI, days		
		No.		Rate per application				
			kg ai/ha	g ai/tree	g ai/100 m row or g ai/m ²			
Coffee	Angola	1	3	2-2.5		No PHI		
	Argentina	1		0.5-2.0		70		
	Brazil	1		0.3-3.0		90		
	Colombia	2		0.75-1.5		90		
	Costa Rica	NS ¹		0.6-0.84		75		
	El Salvador	1	3-4.35			90		
	Guatemala	1	3-4.35			90		
	Honduras	1		0.6-0.84		75		
	Jamaica	1						
	Kenya	1	3	2.25		100		
	Malawi							
	Mexico	1		0.3-3		90		
	Nicaragua	1	3-4.35			90		
	Panama	1		0.6-0.84		75		
	Peru	2		1.5-3		No PHI		
	USA (Puerto Rico)	2		2-3		90		
	Venezuela	2	4.5	1-1.5		90		
Cotton	Argentina	1	0.15-2			60		
	Australia	2 ²	0.45-2.1			No PHI		
	Bolivia	1	0.45-1.95			No PHI		
	Brazil	1	0.45-1.95			No PHI		
	China	2	0.5-2.25			90		
	Colombia	1	0.75-1.05	0.02-0.026		No PHI		
	Costa Rica	2	0.825			75		
	Egypt	2	1.07-2.38			No PHI		
	El Salvador	2	0.3-4.5			90		
	Ethiopia	1	2.7			No PHI		
	Greece	1	0.5-1.1			90		
	Guatemala	2	0.3-4.5			90		
	Honduras	2	0.825			75		
	Israel	2	0.975-3			90		
	Malawi (see South Africa)	2						
	Mexico	2	1.05-3			90		
	Morocco	1	1.5-1.8					
	Pakistan	1	1.2			No PHI		

Crop	Country			Application		PHI, days
		No.		Rate per app	plication]
			kg ai/ha	g ai/tree	g ai/100 m row or g ai/m ²	
	Panama	2	0.825			75
	Peru	1	0.45-2.1			No PHI
	South Africa	2	0.53-1.5		5.25-15 g ai/100 m row	90
	Spain	1	1			100
	Turkey	1	2.5			90
	USA	2	0.7-3.36			90
	Venezuela	1	0.6-1.5			90
	Zimbabwe	1			5.25-15 g ai/100 m row	No PHI
Macadamia Nuts	South Africa	2			0.75 g ai/m ³	100
Oil palm and coconut (nursery stock)	Cameroon	3		0.2		No PHI
	Ivory Coast	3		0.2		No PHI
Oilseed rape	Ireland	1	5-10			No PHI
	UK ³	1	5-10			No PHI
Palm oil	Guinea					
Peanuts	Argentina	1	0.3-1.0			60
	China	1	2-2.9			90
	Cyprus					
	Egypt		2.5			No PHI
	El Salvador	NS	1.2-2.4			90
	Guatemala	NS	1.2-2.4			90
	Panama		1.2-2.4			90
	South Africa		1.6		5.25-15 g ai/100 m row	100
	Uruguay	1	1-5			90
	USA	1	2.35-3.36			90
Pecan	Israel	1		5.25-21		60
	South Africa	1			0.75 g ai/m ³	100
	USA	1	5.55-11.25			No PHI
Sunflower	France		0.5			No PHI

 $^{^1}$ Not specified on label 2 Apply into the seed furrow at seeding and max. 70 days later 3 Approved use, but voluntarily withdrawn from label

Table 31. Registered uses of aldicarb on legume animal feeds and hops.

Crop	Country		Application		
		No.	Rate	•	
			kg ai/ha g ai/100 m row or g ai/m ²		
Alfalfa seed crops only	Argentina	2	0.5		No PHI
Peas (for fodder)	France	1	1.0	1.7 g/100 m row	No PHI
	Ireland ¹	1	1		No PHI

Crop	Country		Application			
		No.	No. Rate per application			
			kg ai/ha	g ai/100 m row or g ai/m ²		
	UK ¹	1	1		No PHI	
Hops	Ireland ¹	1	2.24	5 g ai/100 m row	No PHI	
	UK ¹	1	2.24	5 g ai/100 m row	No PHI	

¹ Approved use, but voluntarily withdrawn from label

RESIDUES RESULTING FROM SUPERVISED TRIALS

Numerous supervised field trials, conducted around the world between 1966 and 1992, were provided for evaluation. Most of the reports had already been submitted to earlier Meetings. In the present evaluation only those trials are discussed which closely approximated the currently recommended uses.

In the analyses the total residues containing the carbamate moiety were usually determined as aldicarb sulphone by GLC with flame-photometric detection in the sulphur mode. Individual residue components were analysed by HPLC in a few recent studies. In some early trials colorimetric methods were also used. These were validated by comparative studies.

Unless otherwise indicated, the residues were not corrected for recoveries or blank values, and are expressed as aldicarb sulphone.

Residues in various crops are summarized in the following tables.

Citrus fruit: 32-37 38-39 Grapes: Bulb vegetables: 40 Brassica vegetables: 41 Pulses: 42-46 Root and tuber vegetables: 47-49 Sorghum: 50 Sugar cane: 51 Pecans: 52-54 Cotton: 55 Peanuts: 56-58

Underlined residues in the Tables are from treatments according to GAP.

Citrus fruits

Aldicarb residues in citrus fruits have been thoroughly investigated. The results from several hundred trials in 13 countries have been submitted for evaluation.

Separate determinations of aldicarb, aldicarb sulphoxide and aldicarb sulphone residues in orange peel and pulp were reported by McDonough and Maitlen (1967). These show the absence of aldicarb, its toxic residue being composed of aldicarb sulphoxide and aldicarb sulphone in the ratio of about 5:1.

Limes at different stages of maturity were picked in the USA from the same tree at the same time and analysed separately in order to study the translocation of residues. The results are given in Table 32.

The variation of residues within orchards was studied in 3 orange and 3 grapefruit orchards in the USA (Hunt 1991a,b). Temik 15G was incorporated at a rate of 5.6 kg ai/ha under the drip lines of the trees. Fruits were harvested 60 days after application and 100 individual fruits from each plot were analysed separately for aldicarb, its sulphoxide and its sulphone. The aldicarb parent compound was detectable only in 11 samples from one orchard where it was below the LOD. No orange samples had quantifiable residues of aldicarb sulphone, although it was detectable in traces in over half the samples. The findings are summarized in Table 33.

Supervised field trials on several varieties of orange were submitted from 7 countries. The residues from trials at recommended and double rates are summarized in Tables 34, 35 and 37.

In Australia samples of green and mature fruits were taken at various intervals after treatment (Table 34). In addition to the tabulated data, residues of 0.04-0.18 mg/kg and 0.07-0.67 mg/kg were measured in mature fruit samples 14-19 and 28-36 days after treatments with 5.7 and 11.25 kg ai/ha respectively (Rhône-Poulenc Rural Australia Pty Ltd., 1988).

Over 70 trials were reported from Spain from the period 1973-1984. Aldicarb was applied at recommended rates (10-20 g ai/tree). The residues were generally at or below 0.02 mg/kg 100 days after treatment. Higher residues were detected in 5 samples with a maximum of 0.1 mg/kg (Anon b).

Table 32. Aldicarb residues in limes at different growth stages.

	_	
Treatment rate,	Days after treatment	Residues (mg/kg) in fruits of average diameter (cm)
kg ai/ha		

		3.8	5	6.4
5.6	34	0.08	0.05	0.05
	63		< 0.02	< 0.02
11.2	34	0.22	0.08	0.05
	63	0.07	0.04	0.04
	91	0.02	0.02	< 0.02
	127	<0.02	< 0.02	< 0.02
22.4	34	0.24	0.34	0.2
	63	0.19	0.05	0.09
	91	0.02	0.04	0.02
	127	< 0.02	< 0.02	< 0.02

Table 33. Residue levels in the pulp of individual oranges and grapefruit.

Residue component and ranges (mg/kg)	Number of fruits containing residues					
	О	Orange orchard		(ard	
	1	2	3	1	2	3
Detectable aldicarb	0	0	11	0	0	0
Detectable aldicarb sulphone (<0.01)	43	1	100	17	84	79
Aldicarb sulphone ≥0.01 (LOD)	0	0	7	0	0	0
Detectable aldicarb sulphoxide	100	0	100	36	100	98
Aldicarb sulphoxide = LOD (0.01)	21	0	17	6	7	10
Aldicarb sulphoxide >0.01, ≤0.02	18	0	20	6	2	4
Aldicarb sulphoxide >0.02, ≤0.03	0	0	17	1	0	0
Aldicarb sulphoxide >0.03, ≤0.04	1	0	15	0	0	0
Aldicarb sulphoxide >0.04, ≤0.05			6			
Aldicarb sulphoxide >0.05, ≤0.1			8			
Aldicarb sulphoxide >0.1, ≤0.12			5			
Aldicarb sulphoxide >0.012, ≤0.15			6			

In 27 field trials in Turkey (1984-85), aldicarb was applied at rates of 15-45 g ai/tree. Residues were measured in the pulp of all samples and in the peel of a few. The residues were ≤ 0.02 mg/kg in all samples taken 124-199 days after treatment (MacDonald *et al.*, 1985a,c).

The residues reported from trials in Australia, Israel, Italy, Morocco and South Africa on oranges and mandarins are summarized in Table 34.

In the USA field trials on oranges between 1974 and 1978 in Arizona, California, Florida and Texas (Anon, 1984) were at rates of 2.3-22.4 kg ai/ha. The method of application included incorporation into 5 cm stripes, 1.2 m bands, spreading under the trees and shanking into water furrows on two sides of trees. The total residues expressed as aldicarb measured in the peel and pulp and in whole fruits from treatments with recommended (5.6 kg ai/ha) and double rates are summarized in Table 35.

Table 34. Aldicarb residues from supervised trials in oranges and clementine mandarins.

Country, Year	Rate, ai, kg/ha or *g/tree or **g/m ²	Sample			Residue	s (mg/kg) at o	lays after ap	oplication		Ref ¹
			47-51	60-70	90-100	110-149	150-162	168-182	≥210 (No. of days)	
Australia 1986	5.7	mature fruit	0.19	0.21	0.13	0.06	0.02	0.02^{2}		1
	11.25	mature fruit	0.64	0.60	0.43	0.40	0.21	0.07^2		
1987	5.7	green or semi- mature fruit				0.06		0.02	0.01	
	11.25					0.09		0.02	0.01	
1987	5.6	green or semi- mature fruit				0.02	<0.01		<0.01	
	11.25					0.04	0.03		< 0.01	
1988	11.25	fruit				<0.01-0.08	0.02- 0.03	0.01	< 0.01	
	22.5	fruit				0.28-0.58	0.06-0.08	<0.01- 0.08	0.03-0.07	
Israel 1976	10	fruit	0.07		ND					2
	20	fruit	0.06		ND					
Italy 1987	10	mandarin							<u><0.01</u> (315)	3
	20	mandarin							<0.01 (315)	
1988	19	mandarin							< <u>0.02</u> (273)	4
1981	10	mandarin						<0.01		
	20	mandarin						< 0.01		
Morocco	3	pulp		0.04 0.08 0.03						5
		peel		< 0.01						
	4.5	mandarin pulp		0.09 0.12 0.05						
		peel		0.05 0.12 0.05						
	4.5	mandarin pulp			0.06 0.03 0.08					
	3	mandarin pulp			0.05 0.03 0.03					
		peel			0.13 0.11 0.05					

Country, Year	Rate, ai, kg/ha or *g/tree or **g/m ²	Sample			Residue	s (mg/kg) at	t days after a	pplication		Ref ¹
			47-51	60-70	90-100	110-149	150-162	168-182	≥210 (No. of days)	
	3.75	pulp			0.21 <0.01 0.08					
		peel			0.82 <0.01 0.29					
	4.5	mandarin pulp			0.07 0.07 0.08					
		peel			0.19 0.27 0.31					
S. Africa 1979	3**	fruit		0.5					0.01 (252)	6
	4.5**	fruit		1.9					0.01 (252)	
	5.7	fruit		0.7					0.02 (252) <0.01 (315)	
	10.4	fruit		1.0					0.01 (252) <0.01 (315)	
	13.8	fruit		0.7					0.02 (252) <0.01 (315)	
	17.9	fruit		2.5					0.04 (252) <0.01 (315)	
	3**	fruit	0.85			0.12			<0.01 ³	
	4.5**	fruit	1.1			0.09			<0.01 ³	
	5.7	fruit	0.65			0.1			<0.01 ³	
	10.4	fruit	3.0			0.2			<0.01 ³	
	13.8	fruit	3.5			0.1				
	17.9	fruit	3.0			0.2			<0.01 ³	
	3**	fruit	0.4	0.3		0.1	0.06		< 0.063	
	4.5**	fruit	0.6	0.48		0.1	0.09		0.06^{3}	
	7.2	fruit	0.3	0.12		0.03	0.06			
	10.8	fruit		0.3		0.09	0.09		0.12^{3}	
	14.4	fruit		0.42		0.01	0.12		0.12^{3}	
	3**	fruit						0.25		
	4.5**	fruit						0.09		
	3**	fruit							0.06 (217)	
	4.5**	fruit							0.04 (217)	
1981	3**	fruit			0.21					
	4.5**	fruit			0.33					
	30*	fruit			0.24		< 0.03			
	45*	fruit			0.03		< 0.03			

Country, Year	Rate, ai, kg/ha or *g/tree or **g/m ²	Sample			Residues	s (mg/kg) at	days after ap	pplication		Ref ¹
			47-51	60-70	90-100	110-149	150-162	168-182	≥210 (No. of days)	
	7.2	fruit			< 0.03		<0.03			
	10.8	fruit			0.03		<0.03			
	14.4	fruit			0.07		<0.03			
	45*	fruit				0.5 0.2				
	45*	fruit				< 0.07				
	3**	fruit				0.03				
	4.5**	fruit				0.03				
	7.2	fruit				< 0.03			0.03^{3}	
	10.4	fruit				< 0.03			<0.01 ³	_
	13.8	fruit				< 0.03			< 0.013	

¹ References:

Table 35. Residues expressed as aldicarb in oranges from supervised trials in the USA.

Rate, kg ai/ha	PHI, days	Sample			Number	of residues in	range, mg/k	g	
			< 0.05	≤0.1	≤0.2	≤0.3	≤0.4	≤0.5	>0.5
5.6	31-35	Peel	1		2		1		
		Pulp	3	1					
		Green fruit					1		
		Ripe fruit	1	3	2				
11.2		Green fruit						1	
		Ripe fruit	9	1	5	1			
5.6 60-70	Peel		1	1				1	
		Pulp	2		1				
		Green fruit		1		1			
		Ripe fruit	3	2	1				
11.2		Green fruit				1	1		
		Ripe fruit	6	6	1	2	1		
5.6	70-80	Peel			1				
		Ripe fruit	1						
11.2				1					
5.6	90-100	Peel		2					
		Pulp	2						
		Green fruit	1			2			

^{1.} Rhône-Poulenc Rural Australia Pty, 1988; 2. Christopher *et al.*, 1977a; 3. Toussaint, 1987; 4. Muller, 1988c; 5. Parsons, 1986; 6. Maybaker, 1982

² Sample was green fruit from next crop ³ Samples were taken at maturity (date not given)

Rate, kg ai/ha	PHI, days	Sample			Number	of residues in	n range, mg/l	kg	
			< 0.05	≤0.1	≤0.2	≤0.3	≤0.4	≤0.5	>0.5
		Ripe fruit	5	2					
11.2		Green fruit	1	2					1
		Ripe fruit	3	9	4				
5.6	101-120	Green fruit	1						
		Ripe fruit							
11.2		Green fruit	1						
		Ripe fruit	2	3					
5.6	121-140	Peel	1	1					
		Pulp	1	1					
		Green fruit			1				
		Ripe fruit		1					
11.2		Green fruit	2			1			2
		Ripe fruit	2		1				
5.6	141-160	Pulp	5						
		Green fruit	1						
11.2		Green fruit	1						2
		Ripe fruit	2		1				
5.6	161-180	Pulp	6						
		Green fruit	2						
11.2		Green fruit	3	1					
		Ripe fruit	1	1					
5.6	181-200	Peel	5						
		Green fruit	2						
		Ripe fruit		1					
11.2		Green fruit	2						
		Ripe fruit			1	1			
5.6	201-250	Peel	1						
		Pulp	1						
		Green fruit	2						
		Ripe fruit	3		2				
11.2		Green fruit	2						
		Ripe fruit	3			2			
5.6	> 250	Ripe fruit	4						
11.2		Green fruit	1						
11.2	>250	Ripe fruit	2						

From 1974 to 1978 supervised field trials in which aldicarb was applied at recommended and higher rates (Anon, 1979, 1984) were carried out in grapefruit orchards (21 trials) in California, Florida and Texas, in lime orchards (18 trials) in Florida, Peru and Mexico, and in <u>lemon</u> orchards in Arizona and California. The residues from recommended and double rates are shown in Table 36.

Grapefruit trees were treated at rates of 20 and 40 g ai/tree in France. Residues were not detectable in either peel or pulp samples 179-264 days after treatment (Muller, 1988a).

In field trials on grapefruit and lemons in South Africa in 1979 aldicarb was applied at rates of 30-75 g ai/tree and 7.5-18 kg ai/ha. The residues detected are shown in Table 37. When aldicarb was applied at 10 kg ai/ha, none of the 19 samples taken from the four trial sites contained detectable residues (<0.03 mg/kg) (Maybaker, 1982).

Six plots of lemons were treated with recommended rates (10-20 g ai/tree) in Spain in 1982-3. The residues were \leq 0.02 mg/kg (limit of determination) in all samples (Anon undated, b)

Aldicarb was applied at rates of 30, 45 and 53 g ai/tree to protect lemon trees in Turkey. Residues of ≤ 0.02 mg/kg were reported in the pulp from 25 trials in which the samples were taken ≥ 87 days after application (Table 37). Higher residues were present at shorter intervals (Macdonald *et al.*, 1985a).

Lime trees were treated with aldicarb in Brazil at rates of 10, 20 (registered) and 40 g ai/tree. Residues in the peel were 0.05, 0.07 and 0.015 mg/kg, respectively, 30 days after application. The corresponding pulp samples contained residues of 0.02, 0.03 and 0.05 mg/kg (Casadei de Batista, 1984).

Whole limes from trees treated with 20 and 40 g ai/tree in France contained residues of <0.02-0.17 mg/kg 74 days or more after the treatment (Table 37).

Table 36. Aldicarb residues from trials in grapefruit, limes and lemons in the USA.

Rate,	PHI, days	Sample	Number of residues in range, mg/kg
kg ai/ha			

			≤0.05	≤0.1	≤0.2	≤0.3	≤0.4	≤0.5
5.6	31-34	Green fruit	2	1				
		Ripe fruit	4	2		1		
11.2		Green fruit	1	1		1		1
		Ripe fruit	3	5				
5.6	42-63	Green fruit	3	1				
		Ripe fruit	1	1	1			
11.2		Green fruit	3	2	2		1	
		Ripe fruit	3	2	3	2		
5.6	90-105	Green fruit	1					
		Ripe fruit	4					
11.2		Green fruit	5	1	2			
		Ripe fruit	3	3	2			
5.6	127-150	Green fruit	2					
		Ripe fruit	1					
11.2		Green fruit	5	1				
		Ripe fruit	4	1				
5.6	183-199	Green fruit	4					
11.2		Green fruit	2					
5.6	230-265	Green fruit	1					
		Ripe fruit	6					
11.2		Green fruit	1					
		Ripe fruit	4					

In 28 trials in Florida tangerine hybrids were treated at 11.2 kg ai/ha at spring flush, or 5.6 kg ai/ha at spring flush and again 100 days later. Samples were taken between 73 and 246 days after application. Residues in the whole fruit were generally below the limit of determination (0.03 mg/kg) with a few exceptions where 0.03 mg/kg, $2 \times 0.04 \text{ mg/kg}$ and 0.05 mg/kg were detected. The application represented a double rate according to the current use pattern.

Trials on mandarins were reported from Italy (Toussaint, 1987), Morocco ((Parsons, 1986), Spain (Anon undated, b) and Turkey (Christopher *et al.*, 1977b,c; Woodhouse *et al.*, 1979a; Macdonald *et al.*, 1985c). Except in the Moroccan trials the residues were at or below the limit of determination (0.02 mg/kg) in mature fruit. The results of the trials in Morocco are included in Table 34 (mandarins and oranges).

Table 37. Aldicarb residues from supervised trials on citrus fruits. Whole fruits analysed except in Turkey, 1984, where pulp was analysed.

Crop	Rate, ai	Residues, mg/kg, at days	Ref.1
Country	kg/ha or		
Year	*g/tree or		
	$**g/m^2$		

		14-19	28-36	43-55	60-79	88-100	100-149	150-162	≥168 (No. of days)	
Grapefruit S. Africa 1979	30*			0.25					0.03	1
	45*			5.4					0.03	
	60*			11.8					0.06	
	7.2								< <u>0.04</u> (168)	
	10.8			2.4					< <u>0.04</u> (168)	
	14.4			3.5					<u><0.04</u> (168)	
	18			7.2					< <u>0.04</u> (168)	
Lemon Spain 1982	10*								0.06 (199)	
	20*								<0.02 (199)	2
	10*								0.05 (199)	
	20*								0.08 (199)	
	10*				0.06					
	20*				0.08					
1983	20*	0.1	0.06		<u>0.05</u>	< 0.01				
	20*	0.15	0.15		0.1	0.05				
	24*		< 0.01		< 0.01	< 0.01				
	24*		< 0.01		< 0.01	< 0.01				
	20*		0.01		<u>0.05</u>					
	20*		0.01		0.2					
S. Africa 1979	3**	1.1							0.01 (197)	1
	4.5**	1.6							0.04 (197)	
	3.3	0.7							0.02 (197)	
	5.1	1.1							0.02 (197)	
	6.8	1.3							0.07 (197)	
	8.6	2.0							0.05 (197)	
	3**								0.12 (175) 0.01 (189)	
	4.5**								0.28 (175) 0.12 (189)	
	3**						0.21		<0.02 (239)	
	4.5**						0.21		<0.02 (239)	
Turkey 1984	30*	0.08^{2}		0.122	0.112					3
	45*	0.31 ²		0.28^{2}	0.16^2					
	53*	0.23^{2}		0.34^{2}	0.18^2					
Limes France 1987	20		0.37	0.36	0.05	0.05	<0.02			4
	40		0.02							
Mexico	30*			0.04	< 0.03	< 0.03	< 0.03			5

Crop Country Year	Rate, ai kg/ha or *g/tree or **g/m ²				Residues	s, mg/kg, a	at days			Ref. ¹		
		14-19	-19 28-36 43-55 60-79 88-100 100-149 150-162 ≥168 (No. of days)									
1981												
	45*			0.03	0.03	0.04	0.04					
	60*			0.05	< 0.03	< 0.03	< 0.03					
	30*			0.05	0.04	<0.03	< 0.03					
	45*			0.34	0.08 0.14 (80 days)	<0.03	0.1					
	60*			0.44	0.2	0.19	< 0.03					
Tangerines Turkey 1975	25*								0.05 (193)	6		
	50*								0.09 (193)			

¹ References:

<u>Grapes</u>. Supervised field trials were reported from eight countries, three of which have registered uses for aldicarb.

In Australia the trials were at 3 locations with recommended and double rates (Table 38).

Five varieties of grape were treated at 8 locations in Chile. Triplicate samples were analysed from each plot. In addition to the trials summarized in Table 38, seven trials were carried out with 4.95 kg ai/ha (Myers, 1983) and three with 3.15 kg ai/ha (Sanchez, 1991). Samples were taken between 112 and 153 days after treatment. No residues were detectable in 28 samples. Two samples contained 0.04 and 0.06 mg/kg.

In Egypt Cabernet and Thompson Seedless grapes (2 sites) were treated at the recommended 4.7 kg ai/ha rate. Residues in grapes taken from the three experimental plots 123-132 days after application were below the limit of determination (0.03 mg/kg) (Adams *et al.*, 1991). The residues detected in trials in Brazil, France and Mexico are also shown in Table 38.

Table 38. Residues of aldicarb in grapes from supervised field trials.

Country Variety Year	Rate kg ai/ha		Residue, m	g/kg, at days af	ter application	1	Ref.
		80-100	110-132	137-159	160-200	217-240	
Australia Gordo 1986	2.25			0.03 (3)1			R-P. AUS 1988
	4.5			0.09 0.14 0.22			
Sultana	2.25			0.01			

^{1.} Maybaker, 1982; 2. Anon, undated b; 3. MacDonald *et al.*, 1985a; 4. Muller, 1988b; 5. Bocanegra, 1982; 6. Dawson, 1976.

² Residues in pulp. All other samples were whole fruit

Country Variety Year	Rate kg ai/ha		Residue, mg	/kg, at days aft	er application		Ref.
		80-100	110-132	137-159	160-200	217-240	
1986				<u>0.02</u> <u>0.03</u>			
	4.5			0.03 0.07 0.08			
Rhine Riesling 1986	2.25					0.02 (2) ² <0.01	
	4.5					0.06 0.02 <0.01	
Gordo 1988	2.25 4.5				<0.02 0.07		
Gordo 1988	4.5			0.04			
Sultana, dried 1988	2.25 4.5		<0.05 <0.05				
Cabernet, 1988	4.5	0.17					
Brazil Jacquet, 1985	2.5	<0.02, 0.02, 0.04 0.03 (2) ¹ 0.04					Casadei de Batista 1984
Chile Thompson	5.2		<0.03				Myers 1982
Ribiers	4.0		< 0.03, 0.05				
Ribiers	7.8				<0.03 0.03 0.06		
Thompson	6.5				<0.03 0.1 (2)		
Thompson	5.2			<0.03 (3)1			
Sauvignon	2.2			$< \underline{0.03} (3)^1$			
Semillon	1.9			< <u>0.03</u> (3) ¹			
Cabernet	1.6				$< \underline{0.03} (3)^1$		
France Ugni Blanc 1990	20			1.6	0.27	0.11	Muller 1990
Mexico 1975	1 g ai/plant 2 3 4		<0.02 <0.02 0.07 0.06				Romine 1976
Thompson 1985	2.25 4.5		$<0.02 (8)^2$ $<0.02 (8)^2$				Ayers 1988
Bola Dolce 1985	3 4 5			<0.02 (4) ² 0.02 (3) ² 0.02 (2) ² 0.06			
Grenache 1985	3 4 5			<0.02 (3) ² <0.02 0.03 0.04 0.03 0.09			

Country Variety Year	Rate kg ai/ha		Residue, mg/	kg, at days aft	er application	1	Ref.
		80-100	110-132	137-159	160-200	217-240	
				0.13			
Sevell	3 4 5			<0.02 (3) ² <0.02 0.03 0.05 <0.02 0.02 (2)			
Bola Dolce 1987	3 3.75 4.5		<0.02 (3) ² <0.02 (3) ² <0.02, <0.02 0.08				Ayers 1988
South Africa Clairette Blanche 1982	3.75 5.55 7.5		<0.05 0.43 0.52		< <u>0.03</u> ³ <u>0.1</u> <u>0.05</u>		U.C. SA
	3.75 x 2 ⁴		0.06		< 0.033		
Cabernet Sauvignon 1982	3.75 5.55 7.5		0.03 0.1 0.26		$\begin{array}{c} 0.06^{3} \\ 0.04 \\ 0.09 \end{array}$		
	3.75 x 2 ⁴		0.05		0.03^{3}		
Semillon 1982	3.75 5.55 7.5		0.16 0.87 0.65				
	3.75 x 2		0.15				
Not specified 1982	3.75 5.55 7.5		0.23 0.30 0.77		0.06 ³ 0.16 0.1		
	3.75 x 2		0.17		< 0.033		

¹ Number of replicate samples. ² Number of trials. ³ Samples taken at harvest. Time between application and harvest not specified. ⁴ 1st treatment at autumn after harvest, 2nd treatment in spring at bud swelling.

R-P AUS: Rhône-Poulenc Rural Australia Pty Ltd (1988); U.C. SA: Union Carbide South Africa Pty Ltd (undated, a)

In supervised trials from 1979 through 1983 at 39 sites in eight States of the USA aldicarb was applied once in a year at a rate of 4.5 kg ai/ha. The treatments were intended to be not less than 120 days before the harvest (Myers, 1984). The results are shown in Table 39.

Table 39. Aldicarb residues in grapes from supervised trials in the USA.

PHI, days			Number of resid	lues in ranges,	mg/kg	
	≤0.01	≤0.05	≤0.1	≤0.2	≤0.3	≤0.4-<0.5
90-109	2	3	4	2		
110-119	5	3	3	4	3	3
120-130	6	9	7	14	9	2
>130	2		2	2	1	

Bulb vegetables

Supervised field trials were reported from Israel, The Netherlands, the UK and Venezuela. The results are presented in Table 40.

Table 40. Aldicarb residues in garlic and onions from supervised trials.

Crop, Country	No (Ye	of trials ar)	Rate, kg ai/ha	Residues	, mg/kg, at d	ays after appli	cation	Ref.
				108-115	138-142	154-162	169-184	
Garlic Dominican Rep.	1	(1991)	3.36				ND	R-P
Venezuela	3	(1991)	3	0.1 0.06 0.07				
Onion Israel	4	(1976)	2	0.01 0.01 <0.01 (2)		0.02 0.02 <0.01 (2)	0.01 <0.01 (2)	Anon a
	4	(1976)	4	0.01 <0.01 (3)		< <u>0.01</u> (4)	0.01 <0.01 (3)	
UK	21	(1970)	2.24 furrow			< <u>0.04</u> <u>0.08</u>		Burrows
	21	(1970)	6.7 broadcast			< <u>0.04</u> <u>0.08</u>		1971
	1	(1970)	1.12+ 1.12 ²		<0.04			
	1	(1970)	2.24		<0.02			
	1	(1970)	4.48		< 0.02			
	2	(1972)	1.12	1.28 0.38 ³			0.02 0.05	
	4	(1992)	7.7 g/100m				< <u>0.01</u> ⁴ (4)	Maycey
Netherlands	2	(1971)	1.5				< 0.05	NL

¹ Normal bulb onions and onions with thick neck were treated. Higher residues are from thick neck onions ² First application in furrow at planting in autumn, second broadcast application at emergence in spring ³ Salad onion

NL The Netherlands (1994)

⁴ For each residue component: parent, sulphoxide and sulphone

R-P Rhône-Poulenc (1991b)

Brassica vegetables

Brussels sprouts planted in furrows treated with aldicarb at 7.7 g ai/m in the UK (Woodhouse *et al.*, 1978a), and at rates of 1, 2 and 3 kg ai/ha in The Netherlands (Netherlands, 1994) were analysed 152-175 days later. The buttons from two UK trials contained residues of 0.01 mg/kg (PHI 155-172 days), while in 8 samples from The Netherlands the residues were <0.003 mg/kg.

The results of supervised field trials on cabbage and cauliflower were reported from the UK. Aldicarb was applied in furrow prior to transplanting five varieties of cauliflower and 4 varieties of cabbage. The results are shown in Table 41.

Table 41. Aldicarb residues in Brassica vegetables from supervised trials in the UK.

Crop, year	Rate, g ai/100 m	PHI, days	Residues, mg/kg	Reference
Cauliflower, 1977	7.7	90	< 0.02	Woodhouse et al., 1978a
1977	7.7	76	<0.02	
1978	5.1	185	0.02	Woodhouse et al., 1979b
1978	7.7	185	0.05	
1978	5.1	56	0.11	
1978	7.7	56	0.09	
1978	5.1	56	0.02	
1978	7.7	81	0.08	
Cabbage, 1977	7.7	141	< <u>0.02</u>	Woodhouse et al., 1978a
1978	5.1	67	0.88	Woodhouse et al., 1979b
1978	7.7	67	2.72	
1978	5.1	81	< 0.01	
1978	7.7	81	0.01	
1978	5.1	7	5.1	
		32	0.52	
		45	0.27	
		62	0.13	
1978	7.7	7	2.58	
		32	0.37	
		45	0.14	
		62	0.27	

Pulses

Trials on beans and peas reported from Argentina, Brazil, France and the UK are shown in Table 42.

Table 42. Aldicarb residues from supervised trials on pulses.

Crop, Country	, Year	Rate, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Reference
Beans, o		0.5	beans ¹	97	< 0.002	Larea 1991b
Brazil	1989	1.95	beans	69	0.76	Santana 1989
		3.9	beans	69	0.67	
France	1988	1.0^{2}	beans	128-151	<0.02 (5)	Muller 1989a
	1989	1.0^{3}	beans	109-123	<0.02 (4)	Muller 1990b
			straw	109-123	<0.04 (3), 0.19	
		1.5 ³	beans	109-123	<0.02 (4)	Muller 1990c
			straw	109-123	<0.04 (2), 0.10, 0.18	
UK	1968	2.24 BC	nearly ripe beans	155	0.06	Anon undated, c
Peas UK	1968	1.7 FR	peas	97	0.02	
	1977	1.1 ⁴ FR	peas	105-115	<0.01, 0.01 (2), 0.02	Woodhouse et al., 1978b
	1978	1.55	peas	101-119	<0.01, 0.01, 0.03	Woodhouse et al., 1979b

Samples taken 15 days before normal harvest.
 Belinda variety was used in 4 trials at 2 sites.
 Sparkle variety was used at 4 sites

In 7 states of the USA 34 field trials were carried out with recommended or 1.5-fold rates on 10 varieties of beans in 1974-1975. In addition to the mature dry beans, immature seeds were also analysed 20 and 40 days before harvest (Union Carbide Corporation, 1979a). The residues in the seeds and forage are shown in Tables 43 and 44.

Table 43. Aldicarb residues in dry beans from supervised trials in the USA, 1974-75.

Rate, kg ai/ha	Residues	, mg/kg, at or before harves	st
	40 days before	20 days before	Harvest
0.84-2.24	0.32	0.04	
	0.28	0.11	
	0.07	< 0.02	
	0.05		0.02
	0.08	<0.02	<0.02
	0.12	0.06	0.02
	<0.02 (4)	<0.02 (7)	<0.02 (17)
3.34	1.3	0.49	
		0.09	0.03
		0.02	0.07
	0.09		<0.02
	0.07	0.02	<0.02
		2.2	0.03

⁵ Puget and Sparkle varieties were treated in 3 trials.

Rate, kg ai/ha	Residues	, mg/kg, at or before harves	t
	40 days before	20 days before	Harvest
	0.29	0.17	0.03

Table 44. Distribution of aldicarb residues in dry bean forage.

Rate, kg ai/ha	Residue, mg/kg, range	Residu	es, mg/kg, at or before	re harvest
		40 days before	20 days before	Harvest
1.12	≤0.5	2	2	7
	≤1	1	1	
	≤2	1		
	≤5		1	
	≤10		1	
	≤20	1		
1.6-2.24	≤0.5	4	6	9
	≤1	2	2	1
	≤2	1	1	
	≤5	3	1	
	≤10		1	
	>30 ≤40	1		
3.36	≤0.5	1	1	3
	≤1			2
	≤2		1	1
	≤5	1	3	1
	≤10	2	2	
	≤20	3	1	

Trials on 8 varieties of soya beans were reported from 8 States of the USA. The treatments were carried out with recommended rates (Union Carbide Corporation, 1979a). In addition to the mature seeds at harvest, immature succulent seeds were shelled from the pods for analysis in order to provide additional assurance of low residues at harvest. The results are shown in Table 45.

Table 45. Aldicarb residues in soya beans from trials in the USA.

Rate, kg ai/ha	Residue, mg/kg, range	Re	sidues, mg/kg, at or befo	ore harvest
		50 days before	25 days before	Harvest
<1.12	≤0.02			3
2.24	≤0.02	5	6	10
3.92	≤0.02	4	7	11
	≤0.05		2	
	≤0.1		1	

Rate, kg ai/ha	Residue, mg/kg, range	Residu	nes, mg/kg, at or before h	narvest
		50 days before	25 days before	Harvest
	≤0.2	1		

The soya bean forage was also sampled before and at harvest. The residues are shown in Table 46.

Table 46. Aldicarb residues in soya bean forage from supervised trials in the USA.

Rate, kg ai/ha	Residue, mg/kg, range	Residue	s, mg/kg, at or befo	re harvest
		50 days before	25 days before	Harvest
2.24	≤0.5	1	9	6
	≤1	5		
	≤2	1		
	≤5	1		
3.92	≤0.5	1	4	6
	≤1	1	1	1
	≤2	2	2	
	≤5	1	2	
	≤10	1		
	≤20	1		

Root and tuber vegetables

<u>Carrots</u> were grown in soil treated with aldicarb at planting in the UK in 1978 (Woodhouse *et al.*, 1979b). The results are shown in Table 47.

Table 47. Aldicarb residues from supervised trials on carrots in the UK.

Rate, g ai/100 m		Residues, mg/kg, at	days after treatment	
	43	55	70	84
2.6	6.0			0.17
3.8	6.5			0.21
5.1	15	1.35	1.51	0.29

<u>Swedes</u> were grown in soil treated at planting with aldicarb at rates of 5.1 g ai/100m (1.3 times GAP) and 7.7 g ai/100 m (twice GAP). Whole plant and root samples were taken 191-222 days after the treatment. Residues were undetectable (<0.01 mg/kg) in all of four root samples and 8 whole plant samples (Woodhouse *et al.*, 1979b). In another trial forage swedes were treated at 1.3 times the recommended rate. Whole plant and root samples taken 129 days after application contained 0.04 and 0.01 mg/kg respectively (Woodhouse *et al.*, 1978b).

Supervised trials on sweet potatoes were carried out in 5 states of the USA between 1968 and

1975. Three varieties of sweet potato were planted on the day of or within 2 weeks of soil treatment. Application rates ranged from the recommended 1.7-3.36 kg ai/ha to 6.72 kg ai/ha. The immature roots, mature roots and vines, were analysed. The residues in the roots are shown in Table 48 (Union Carbide Corporation, 1979a).

Table 48. Aldicarb residues in sweet potato roots from supervised trials in the USA, 1968-75.

Rate, kg ai/ha	Residues, mg/kg, at days after planting ¹					
	90-105	107-114	119-128			
1.7			< <u>0.01</u> (6)			
2.24	0.07	0.04	< <u>0.01</u> (6) <u>0.02</u> <u>0.03</u> (2) <u>0.04</u>			
3.36	0.06 (2) 0.07 0.19	0.02 (2) 0.03 0.04 (2)	< <u>0.01</u> (7) <u>0.04</u> <u>0.05</u> (2)			
4.5			<0.01 (2) 0.01			
5.6	0.14	0.14	0.09			
6.7	0.28 0.48		0.01 0.02 0.06 0.11 0.16			

¹ Number of results in parentheses

The vines of sweet potatoes following treatment with the maximum recommended rate contained residues in the range 0.21-0.89 mg/kg after 90-105 days, and 0.34 mg/kg after 119 days. When a double rate was applied the residues were 1.6-5.1 mg/kg after 90-105 days and 0.74 mg/kg after 119 days.

Supervised trials on sugar beet were reported from Belgium, Denmark, France, Germany, The Netherlands, Spain, the UK and the USA from the period 1967-1990 (Anon, 1992; Orme, 1975; Union Carbide Europe S.A., undated; Muller, 1989b, 1990d,e, 1991a; Union Carbide Corporation, 1970). In European trials aldicarb was applied in furrow, band or broadcast at rates between 1 and 5 kg ai/ha, corresponding to the recommended and double rates. Following application at sowing (GAP) the residues in the roots were at or below 0.04 mg/kg in samples from 76 trials. Broadcast applications over the plants (up to five leaves) did not result in higher residues. In four trials conducted in the UK in 1967-68 higher residues were detected in two samples: 0.05 mg/kg (from a 4.5 kg ai/ha top-dressing application, 182 days) and 0.06 mg/kg (1.12 kg ai/ha in furrow, 159 days). In addition to the mature roots, six immature root samples were analysed. Two of them contained residues above 0.04 mg/kg: 0.05 mg/kg (1.36 kg ai/ha, 139 days) and 0.06 mg/kg (1.8 kg ai/ha, 128 days).

The residues in tops and leaves ranged from 0.03 to 0.65 mg/kg in all of the 79 samples. One immature beet sample from a UK trial in 1968 contained 1.3 mg/kg 91 days after a top-dressing application with 4.5 kg ai/ha. By harvest, the residues in the leaves had declined to 0.02 mg/kg.

Trials were conducted in eight States of the USA from 1966 through 1969 (Union Carbide Corporation, 1970). Aldicarb was applied shortly before or at planting and 1 to 5 times after planting. Rates ranged from 1.68 kg ai/ha to 33.6 kg ai/ha. The residues were measured in leaves and roots from 18 to 238 days after the last application.

Single applications up to 4.5 kg ai/ha with a 90-day or longer PHI, corresponding to the current recommended uses, resulted in residues in roots of \leq 0.02 mg/kg in 23 trials. A double rate (9 kg ai/ha) also gave residues of \leq 0.03 mg/kg at harvest (\geq 140 days after application).

The residues in leaves from treatments corresponding to GAP ranged from <0.1 to 0.93 mg/kg at 120 days or later. After treatments with a double rate the residues in the leaves amounted to 0.98 mg/kg at 160 days. The decrease of residues in roots and leaves is illustrated in Table 49. Analyses of 14 leaf samples showed that the major component of the residue was aldicarb sulphone, amounting to an average of 78%. Aldicarb sulphoxide was present at 22% and aldicarb was not detectable.

A trial with <u>fodder beet</u> was reported from Germany. Following a treatment with 0.9 kg ai/ha the residues in 4 root samples taken after 76-159 days were <0.02 mg/kg, the limit of determination. The residues in the leaves (mg/kg) were 0.53 (day 76), 0.31 (day 104) and 0.05 (day 138).

State	Appl. rate, kg ai/ha		Residues, mg/kg, at days after application						
		60	90	120	138-146	159-163			
Leaves									
Utah	2.24	5.0	< 0.1	0.21	0.13				
			1.4	< <u>0.1</u>	< 0.1				
Idaho		5.2	< 0.1	< <u>0.1</u>	0.13				
Utah	4.5	4.6	0.57	<u>0.61</u>	0.51				
			4.7	< <u>0.1</u>		<0.1			
Idaho	9	21	2.8	0.87	0.55				
Roots									
Utah	2.24	0.17	< <u>0.01</u>	< 0.01					
Idaho	2.24	0.27	< <u>0.01</u>	< 0.01					
	4.5	0.23	0.01	0.01	0.01				

Table 49. Aldicarb residues in sugar beet roots and leaves (USA).

Cereals

9

Supervised trials were conducted on wheat, barley, oats and sorghum in Australia (Woodhouse and Eden, 1977), France (Muller, 1990f,g), Israel (Goloner and Adato, 1985), the UK (Anon undated, c) and the USA (Anon undated, e).

0.08

0.03

0.02

In wheat 12 trials in Australia (Woodhouse and Eden, 1977) and 2 in Israel (Goloner and Adato, 1985) indicated that after application at planting with rates up to 3 kg ai/ha, residues were not present in detectable concentrations (<0.01 or <0.02 mg/kg) in the mature grain.

Wheat straw from the Australian trials contained <0.02-0.03 mg/kg.

1.1

In two trials in Germany winter wheat was grown in soil treated with 4.88-5 kg ai/ha in the previous year to protect sugar beet. The grain and straw did not contain any detectable residues (<0.02 mg/kg for grain and <0.04 mg/kg for straw) (Christopher *et al.*, 1976).

In the grain from <u>barley</u> grown in soil treated with 1 kg ai/ha, the residues were <0.02 in 5 French trials (Muller, 1990f,g) and 1 Australian trial (Woodhouse and Eden, 1977).

In the UK, treatments with exaggerated rates (6.72-11.2 kg ai/ha) resulted in residues of 0.01-0.05 mg/kg in 3 mature barley grain samples and 0.05 mg/kg in one oat sample (Anon, undated c).

In 5 French trials samples of barley straw were also analysed but no residues were detected (<0.04 mg/kg).

Numerous trials on <u>sorghum</u> were reported from the USA from the period 1970-1977 (Union Carbide Corporation, 1979a). Applications at or shortly before planting with rates ranging from 0.56 kg ai/ha to 3.36 kg ai/ha did not give rise to detectable residues (<0.01 mg/kg) in mature grain at the normal harvest (115-151 days after the treatments) in 55 of 60 trials. Following applications with 1.12 kg ai/ha and 2.24 kg ai/ha the residues were 0.03, 0.04 and 0.13 mg/kg, and 0.04 and 0.1 mg/kg respectively.

The residues in green plants declined rapidly. The distribution of the residues is shown in Table 50.

Residues were not detectable (<0.02 mg/kg) in sorghum straw in 16 cases. Detectable residues were present in 5 samples at the following concentrations (application rate, kg ai/ha, and PHI, days, are given in parentheses): 0.03 mg/kg (1.34, 124 d), 0.03 mg/kg (1.12, 158 d), 0.09 mg/kg (0.56, 132), 0.11 mg/kg (1.68, 158), and 0.4 mg/kg (1.12, 132).

Table 50. Distribution of aldicarb residues in sorghum forage from supervised trials carried out in the USA.

Rate, kg ai/ha	Residue ranges, mg/kg		Number of residues in range at intervals (days)					
		30-40	60-70	90-105	≥ 120			
0.56-0.84	≤0.2		1	1	2			
	≤0.5		1					
1.12	≤0.2		11	20	16			
	≤0.5	2	4	9				
	≤1	1	1					
	≤2		1					
	≥10-≤15	1						
2.24	≤0.2		1	2	3			
	≤0.5		1	1				
	≤1		1					
3.36	0.5-≤1		1					

Supervised field trials on <u>maize</u> were reported from France, Germany, Israel, the UK and the USA.

In France treatments were at the maximum recommended and up to 2.5-fold rates. Six trials were at the recommended rate (0.5 kg ai/ha). The whole plants did not contain any detectable residues (<0.02 mg/kg) 123-169 days after application (Mestres, 1980). In further trials with excessive 0.75-1.13 kg ai/ha rates, residues were undetectable in whole plants 100 days or more after application in 9 of 12 samples. The remaining three samples contained 0.06, 0.07 and 0.08 mg/kg 132 days after application (Muller, 1988b, 1989c, 1990h,j).

In 6 trials in Germany aldicarb was applied at 0.7-1 kg/ha. Residues in mature grain were below the LOD of 0.02 mg/kg (Christopher *et al.*, 1976).

In Israel the whole plants were analysed 42 days after application at rates of 2 and 4 kg ai/ha. The residues were ND, ND and 0.11, and ND, ND and 0.37 mg/kg respectively (Goloner *et al.*, 1985).

In 18 trials in the UK 2.6-3.8 g ai/100 m was applied at or shortly before planting. No residues were detected in whole plant samples 141-165 days after application (Woodhouse *et al.*, 1978b, 1979b).

In the USA samples were analysed from 25 trials on 21 varieties of field corn in 13 States representing those areas of the USA in which maize is a significant agronomic crop. Following application with 1.68 kg ai/ha the residues were generally below the limits of determinations (0.02-0.03 mg/kg). Measurable residues (0.02, 0.03 and 0.03 mg/kg) were found in three samples (Union Carbide Agricultural Products Co., 1982a).

The residues in green forage harvested from 60 to 116 days after treatment ranged from <0.02 to 0.34 mg/kg. The mature dry fodder (stover) contained residues of <0.02-0.54 mg/kg.

Data on supervised field trials on sugar cane were submitted from Australia, India, Indonesia and South Africa.

In Australia 4 trials were conducted with application rates of 2.8 and 5.6 kg ai/ha (Union Carbide Australia Ltd, 1977). In India (8 trials, Singh *et al.*, 1981) and Indonesia (2 trials, Christopher *et al.*, 1975) the application rates ranged from 0.75 to 4 kg ai/ha and from 5.5 to 7.5 kg ai/ha respectively. The residues in the leaves and stalks from these 14 trials were below the limits of determination (<0.001-0.003 mg/kg) 315-362 days after the applications.

In South Africa the application rates ranged from 2.24 to 11.2 kg ai/ha (Ponena Chemicals, undated a). The residues are given in Table 51.

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Table 51. Aldicarb	reciditec 1	ın cılgar canı	trom cunervice	d friale in	South Atrica
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Rate, kg ai/ha		Residues, mg/kg, at days after application						
-	60-70	119-123	170-181	187-192	320-330			
Untreated			0.09 <0.02		0.06 0.06			
2.24			< 0.02		<0.02			
3.36			0.05 0.33		<0.02 0.11			
5.67			<0.02 0.09		<0.02 0.04			
11.2			<0.02 0.13		0.06 0.03			
31	<0.05 (2)	<0.05 (3)	0.02 (4)	0.02				
	0.12	0.03 (2)		0.03				
	0.09			0.09				
				0.15				

Nuts and seeds

Supervised trials on pecans were reported from Israel, South Africa and the USA.

In Israel 8, 12, 16 or 20 g ai/tree rates were applied in 12 trials according to the use recommendations (Anon, undated a). In ten of 12 samples of kernels residues were undetectable (<0.08 mg/kg). In two samples taken about 3 months after treatments with 8 and 20 g ai/tree residues of 0.08 mg/kg were detected.

In South Africa 17.3-43.2 g ai/tree, 0.75-1.5 g ai/m² and 7.5 kg ai/ha rates were applied. Samples were taken 104-180 days after application. Residues were <0.03 mg/kg in 11 samples. One sample taken 134 days after a double-rate application contained 0.2 mg/kg (Union Carbide South Africa (Pty) Ltd, undated b).

In 7 States of the USA in a number of trials from 1975 through 1981 the application rates and timing were within the current recommendations. A few trials were also carried out at double rates and with 2 split applications (Union Carbide Agricultural Products Co. Inc., 1982b). The residues found in pecan kernels are shown in Table 52. Pecan shells (with hull removed) were analysed separately, with the results in Table 53. In a few samples the residues in immature pecan hulls were also determined. The meat, shell and hull residues in these are shown in Table 54.

Table 52. Aldicarb residues in pecan kernels from supervised trials at recommended rates¹ in the USA.

PHI, days	Residues², mg/kg, after application at rate (kg ai/ha) ≤2.8							
48	0.08; 0.11							
59	0.06; 0.29; 0	0.23; 0.33; 0.77						
64	0.06; 0.15							
70	<0.03; 0.09;	0.04; 0.03; 0.05; 0.06						
81	<0.03; <0.03	<0.03; <0.03						
96-98	0.10; 0.16; 0	0.18; 0.40; 0.75						
120-121	0.09; 0.15; 0	0.09; 0.12; 0.19; 0.13; 0	0.06; <0.03; <0.	03				
133-138	0.02; 0.05; 0	0.02; 0.02; 0.11; <0.03;	<0.03; <0.03; <	<0.03; 0.22; 0.13; 0	0.26			
	≤2.8	3.36-5.6	6.7-9.0	11.2	22.4	5.6 + 2.8		
106-107		0.17 < 0.03						
133-138						0.09 0.15 0.08		
171-189	<0.01	0.02; 0.01 0.06; 0.08 0.05	0.03 <0.01	0.10; 0.01 0.27; 0.05 0.12; 0.13	0.12; 0.16 0.04; 0.30 0.08			
197	0.02	0.04	0.07; 0.09	0.11				
268		< 0.01		< 0.01				

¹ 22.4 kg ai/ha is double rate, split application is not recommended

¹ Each result is from a separate trial. Number of results in parentheses.

² Corrected for recovery

PHI, days	Residues, mg/kg, after application at rate (kg ai/ha)					
	≤2.24	4.5-5.6	6.7-9.0	11.2	22.4	5.6 + 5.6
59-60			0.12	0.02	0.06	
				0.3		
106-107						0.50
180-189	0.03	0.06 0.61	0.04 0.09	0.27 0.05 1.1	0.55 1.7	
197	0.04	0.13	0.20 0.51	0.12		
268		0.02		0.04		

Table 54. Aldicarb residues in immature pecans 60 days before harvest from supervised trials in the USA in 1976. All samples at 60 days PHI.

Rate, kg ai/ha	Residues, mg/kg, in				
	Kernels	Shells	Hulls		
6.8	0.06	0.12	0.33		
11.2	<0.01	0.02	0.04		
22.4	0.03	0.06	0.11		
5.6	0.12	-	0.59		
11.2	0.08	0.3	0.42		

Oil seeds

Supervised trials on cotton were reported from Australia, Israel and the USA.

In Australia trials were conducted with recommended and 1.5-fold rates applied once either at planting or 90 days before harvest in 1983. In 8 of 9 samples residues were undetectable (<0.01 mg/kg) 91-152 days after application. One sample contained 0.02 mg/kg (Union Carbide Australia Ltd., 1983).

In Israel 5 trials were conducted according to GAP. In samples taken 80-159 days after one or two applications the residues were below the LOD (<0.01 mg/kg) (Anon, undated a).

Numerous trials were conducted in 12 States of the USA during 1966-68. In 1966-67 a colorimetric analytical method was used (LOD = 0.04 mg/kg). Later analyses were by GLC with an FPD (LOD = 0.02 mg/kg). The two procedures were compared by analysing 24 field-treated samples. The study indicated that the colorimetric method gave valid results (Union Carbide Corporation, 1979b).

In 35 trials aldicarb was applied at planting at rates ranging from 0.56 to 4.5 kg ai/ha. No residues were detected in 23 samples. The detectable residues ranged from 0.03 to 0.08 mg/kg 112-188

days after application.

After 63 single side-dress applications with 1.12-5.6 kg ai/ha, 25 samples had undetectable residues. Positive residues ranged from 0.03 to 0.09 mg/kg 60-157 days after application. Treatments with 6.7-8.9 kg ai/ha resulted in residues between <0.02 and 0.08 mg/kg during the same sampling period.

Residues from applying aldicarb at planting and squaring according to use recommendations were <0.02 mg/kg in 14 of 36 trials. Detectable residues ranged from 0.03 to 0.08 mg/kg.

When 2 or 3 side-dressing treatments were applied at a total rate of \leq 6.7 kg ai/ha the residues ranged from <0.02 to 0.08 mg/kg 55-97 days after the last application.

The detectable residues found in the US trials with applications up to 1.66 times the highest recommended rate are shown in Table 55.

A number of trials in the three main <u>peanut</u>-growing areas of the USA (Union Carbide Corporation, 1973) and five trials in Senegal (Muller, 1990j,k, 1991b) were reported. The pesticide is applied in furrow at planting and in the USA peanuts are normally harvested 120-150 days after planting.

The kernels, pods and hay were analysed separately in the Senegalese trials. Residues were not detectable in any of the kernel samples (<0.01 mg/kg). The residues in the hay ranged from 0.03 to 0.15 mg/kg in samples taken 79-100 days after treatments with 1 kg ai/ha at planting.

The residues in whole peanuts at harvest from treatments at planting in the USA are shown in Table 56.

The residues in green peanut vines at various intervals after application as well as in peanut hay at the time of harvest of the peanut crop are shown in Tables 57 and 58.

Table 55. Aldicarb residues in cotton seed following single at-planting or side-dressing applications in supervised trials in the USA.

Days after applicn.	Residues, mg/kg, after application at rate (kg ai/ha) ¹								
	0.56	1.12	2.24	3.36	4.44	5.6			
60-74				0.03; 0.04					
76-78			0.01		0.02; 0.03				
85-90			0.02	0.02	0.04; 0.03				
91-100		0.02	0.02		0.05; 0.09; 0.03; 0.04	0.02			
101-110			0.02; 0.06	0.03; 0.02; 0.01; 0.02; 0.03					
111-120				0.01; 0.03; 0.08					
121-130		0.03*; 0.04; 0.01; 0.02; 0.02; 0.03	0.06; 0.01; 0.05	0.07					
131-140		0.02*; 0.03*	0.04; 0.05						
141-150		0.04*							
151-160		0.03*; 0.08*; 0.05*	0.07						
171-180	0.02								

Days after applicn.		Residues, mg/kg, after application at rate (kg ai/ha) ¹							
	0.56	1.12	2.24	3.36	4.44	5.6			
181-190	0.02		0.03; 0.05*		0.04				

¹ Each residue is from a separate trial

Table 56. Aldicarb residues in whole peanuts at harvest from supervised trials in the USA.

PHI, days		Residues, m	g/kg, after applicatio	n at rate (kg ai/ha) ¹	
	1.12	2.24	3.36	4.5	6.7
90		0.01		0.06	
114		< 0.01		< 0.01	< 0.02
		< 0.01		< 0.01	< 0.01
119		< 0.01		0.01	0.02
121		0.01		0.02	0.02
122	0.04 0.05 0.09				0.16
124				< 0.01	
127		0.01		0.02	
131		0.02		0.02	0.04
138		0.05		0.17	0.15
144		0.01		0.01	0.01
145		0.04		0.08	
146		0.01		0.01	0.01
		0.02		0.02	0.04
148			< 0.01		
150		< 0.01		< 0.01	
153	< 0.01	0.01		0.01	
154			<0.01		
156		<0.01		< 0.01	
157		< 0.01		< 0.01	< 0.01
168		<0.01			
174	<0.01	< 0.01		< 0.01	
196		< 0.01		<0.01	
197				< 0.01	0.04

¹ Each residue is from a separate trial

Table 57. Aldicarb residues in green peanut vines from supervised trials in the USA.

State Rate, kg ai/ha	Residues, mg/kg, at days after treatment				
	50-50	70-80	90-100	120	
Alabama					

^{*} Application at planting

State Rate, kg ai/ha		Residues, mg	g/kg, at days after treat	ment
	50-50	70-80	90-100	120
2.24	7.0	1.8	0.93	0.60
2.24	7.5	0.84	0.23	< 0.02
4.5	14.0	6.2	0.34	0.05
6.7	7.1	9.3	1.7	0.10
Georgia				
2.24	0.65	0.38	0.43	0.16
4.5	2.6	1.5	0.92	0.44
2.24	1.9	0.91	0.18	0.03
4.5	3.5	1.9	0.6	0.09
6.7	8.1	4.9	2.0	0.36
North Carolina				
4.5	6.1	-	0.79	0.50
2.24	7.4	7.1	1.1	0.35
4.5	15.0	12.5	1.1	0.80
6.7	21.5	21.8	3.0	2.7
Texas				
3.36	3.0	0.48	0.15	< 0.01
4.5	10.7	1.6	0.34	< 0.01
6.7	20.4	2.9	1.0	< 0.01
Virginia				
2.24	2.5	0.74	0.3	0.03
4.5	5.5	1.6	1.2	0.11
2.24	1.1	0.24	0.34	0.13
4.5	1.5	0.83	0.50	0.14
6.7	1.1	0.58	0.34	0.14

Table 58. Aldicarb residues in peanut hay at harvest from supervised trials in the USA.

PHI, days	Residues, mg/kg, after treatment at rate (kg ai/ha)				
	≤1.12	2.24	4.5	6.9	
121		<0.05	< 0.05	0.11	
122	0.08 0.05 0.08			0.38	
132		0.14	0.53		
138		1.7	2.6	7.6	
144		<0.05	< 0.05	<0.05	
145		0.74	0.69		
150		0.21	0.35		
153		0.19	0.12		
157		<0.05	<0.05	<0.05	
174	0.08	0.07	0.13		
204		<0.05	<0.05	<0.05	

Seven trials on <u>sunflower</u> were reported from France. Aldicarb was applied at the recommended maximum rate of 0.5 kg ai/ha at planting. The seeds did not contain detectable residues (aldicarb <0.02 mg/kg, sulphoxide <0.05 mg/kg, sulphone <0.05 mg/kg) (Mestres, 1983).

<u>Coffee</u>. Trials were reported from Brazil, Columbia, Costa Rica, Ecuador, Guatemala, Peru and South Africa from the period 1973-1991 (Union Carbide Corporation, 1979c; Romine, 1986: Escobar, 1991; Jones, 1991).

In South American trials between 1974 and 1985 one or two applications with recommended rates (up to 3 g ai/tree, each site consisting of 1 to 4 trees growing in a clump) produced low residues in dried and hulled green coffee beans. Four of 47 samples contained residues above the LOD (0.02 mg/kg) 15 to 274 days after the last application: three of 0.03 mg/kg at 118 to 143 days and one of 0.05 mg/kg at day 186. About a double rate resulted in residues of <0.02 (3), 0.04 (2) and 0.08 mg/kg.

In South Africa applications were made at rates of 10 and 15 g ai/tree. The residues in green coffee berries were respectively 0.08-0.19 mg/kg and 0.05-0.24 mg/kg 95 to 123 days after application.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

<u>Citrus fruits</u>. Aldicarb residues have been determined in various fractions resulting from the commercial processing of treated fruit in pilot plants (McDonough and Maitlen, 1967; Anon, 1984). The residues, expressed as aldicarb, are shown in Table 59. The process is illustrated in Figure 3.

Figure 3. Schematic diagram of pilot-plant processing of oranges (The sampling points are indicated in parentheses).

Table 59. Aldicarb residues in processed fractions of oranges, grapefruit and limes.

Appl. rate, kg ai/ha	Residues, mg/kg as aldicarb								
			Oranges			Grapefruit		Limes	
	22.4	22.4	22.4	22.4	44.8	11.2	22.4	11.2	22.4
Ripe fruit	0.07		0.24	0.09	0.53	0.21	0.27	0.07	0.18
Wet peel	0.08	0.57	0.5	0.09	0.69	0.2	0.36	0.07	0.18
Wet pulp			0.12						
Dry peel	0.04	0.42			1.1	0.28	0.47	0.15	0.37
Dry pulp			0.42	0.06					
Juice	0.06	0.11	0.15	0.08	0.18	< 0.02	0.05	0.04	0.1
Juice, concentrated	0.18	0.04		0.22	0.19				
Press liquor	0.02			0.02	< 0.01	0.17	0.34		
Concentrated press liquor	0.04	0.04			< 0.01				
Oil	0.03	0.02	0.02	0.03		0.02	0.04	< 0.01	< 0.01
Oil water	0.02				0.09				
Molasses			0.04	0.03		0.33	0.36	0.24	0.29

Juice was prepared with a citrus juice extractor by taking 3 x 2 fruits from replicate samples of Valencia oranges treated with aldicarb in Australia. The juices were composited. The residues of aldicarb measured in whole fruit and juice samples are shown in Table 60.

The ratio of residues in the peel and pulp of oranges was also determined in many samples taken at wide intervals between application and sampling (Rhone Poulenc Rural Australia, 1988). Some of the data are included in Table 34.

<u>Grapes</u>. Wine was produced from grapes treated with aldicarb at rates of 2.25 and 4.5 kg ai/ha in Australia (Rhône-Poulenc Rural Australia, 1988). The residues found in the wine are shown in Table 61. The residues in the grapes were not reported.

Eight grape varieties were processed to fresh juice, pomace and wine in the USA from 1976 through 1983 (Myers, 1984; Anon, 1985b). The residues are shown in Table 62.

Table 60. Aldicarb residues measured as sulphone in oranges and juice

Application rate, kg ai/ha	PHI, days	Residues, mg/kg		
		Whole fruit	Juice	
5.7	91	0.06 -0.21	0.11	
	119	<0.01-0.06	0.10	
	150	<0.01-0.06	0.07	
11.25	91	0.11-0.33	0.23	
	119	<0.01-0.14	0.14	

Table 61. Aldicarb residues in wine, 3 months after harvest, from treated Rhine Riesling grapes in Australia.

Rate, kg ai/ha	PHI, months	Residue, mg/kg, in the wine
2.25	4	<0.01 0.023
2.25 4	6.5	<0.01 0.01 0.012 0.016

Table 62. Aldicarb residues in processed grape products in the USA.

Grape variety	Year	Residues, mg/kg, in				
		Grapes	Fresh juice	Pomace	New wine ¹	Aged wine ²
Tokay	1981	0.16	0.11	0.1		
French Colombard	1981	0.17	0.10	0.31		
Thompson Seedless	1981	0.10	0.08	0.09		
Cabernet Sauvignon	1983	0.16	0.13	0.5	0.1	0.1
French Colombard	1979	1.5	1.3	2.1	0.66	0.42
Tokay	1979	0.17	0.09	0.16	0.06	0.05
Carignan	1979	1.2	0.72	0.88	0.36	0.18
Ruby Cabernet	1979	2.5	1.6	2.4	0.85	0.48
Emperor	1979	0.04	0.03	0.03	< 0.02	< 0.02
Pinot Chardonnay	1977	3.3	2.6	3.1	2.3	

¹ Wine from final racking and filtration 42 days after start of fermentation

The residues in both fresh juice and wine made from Cabernet Sauvignon grapes contained aldicarb sulphoxide and sulphone in a 1:1 ratio with no parent aldicarb.

Raisins were prepared from Thompson Seedless grapes grown in California in 1981 (Myers, 1984). The residues are shown in Table 63.

Table 63. Effect on aldicarb residues of drying ripe Thompson Seedless grapes for raisins and the by-product, raisin trash.

	Concn. factor			
Ripe grapes	Dry raisins	Raisins/grapes	Trash/grapes	
0.14	0.06	0.87		
0.06	0.10	0.81		
0.09	0.09	0.93		
Mean: 0.10	0.08	0.87	0.8	8.7

² Wine stored for 71 days at ambient temperature after final filtration

	0.24	0.48	2.6		
	0.22	0.27	2.5		
	0.14	0.42	2.2		
Mean:	0.20	0.39	2.5	2.0	12.5
	0.37	0.26	2.6		
	0.21	0.26	2.9		
	0.23	0.27	2.6		
Mean:	0.27	0.26	2.7	1.0	10.0
	0.06	0.04	0.24		
	0.06	0.04	0.14		
	0.02	ND	0.07		
Mean:	0.05	0.03	0.15	0.6	3.0

<u>Pulses</u>. The effects of preparative procedures and cooking as usually done in the home were studied (Union Carbide Corporation, 1979a). Blackeye peas were soaked and cooked for 3 hours. Portions were analysed in every hour. The initial residue of 0.49 mg/kg decreased to 0.15 mg/kg, 0.07 mg/kg, 0.03 mg/kg, and 0.03 mg/kg in soaked beans and cooked 1-h, 2-h and 3-h samples respectively. The reduction of residues in other varieties was similar (Table 64).

Table 64. Reduction of aldicarb residues during cooking dry beans and peas.

Residue, mg/kg, in dry seed		Residues, mg/kg, in fully-cooked seeds
Blackeye Peas	0.49	0.03
	0.49	0.02
Red kidney beans	0.10	0.01
Field Peas	0.22	0.01

Aldicarb residues were determined in processed fractions of <u>soya beans</u>. The experimental plot was treated with 13.3 kg ai/ha (4 times GAP) to obtain detectable residues in the seed. The mature seed containing 0.034 mg/kg was processed according to EPA Pesticide Assessment Guidelines. The beans were dried at 74°C to about 10% moisture to facilitate hulling, then cracked by roller and the hulls separated with an air separator. Kernels were then flaked by rolling to about 0.25 mm thickness and the oil was extracted with hot (63°C) hexane. Solvent was removed from the extracted meal with forced warm air. The crude oil was recovered by evaporating the hexane in a laboratory evaporator. The free fatty acids were reacted with sodium hydroxide and the refined oil was separated by decanting and filtering (Romine, 1988). The following residues (mg/kg) were detected in the processed fractions: hulls 0.04; meal 0.05; crude oil 0.02; soapstock <0.01; refined oil <0.01. The results are in agreement with an earlier study in which soya bean seed containing 0.01 mg/kg aldicarb was processed. Residues were undetectable in both the oil (<0.005 mg/kg) and the meal (<0.01 mg/kg) (Union Carbide, 1979a).

<u>Sugar beet</u>. Six portions, each approximately 90 kg, of sugar beet roots were processed in a pilot unit simulating commercial practice (Union Carbide Corporation, 1970). The residues found in root samples and in the corresponding diffusion juice are shown in Table 65.

Table 65. Aldicarb residues	(mg/kg) found	l in sugar beet roots and	d the corresi	ponding diffusion juice.

Roots	0.005	0.011	0.027	0.019	0.011	0.006
	0.005	0.012	0.024	0.018	0.009	0.006
Diffusion juice	0.005	0.011	0.017	0.006	0.006	0.006
	0.005	0.01	0.016	0.006	0.006	0.006

Other processed fractions (thin juice, thick juice, dry pulp, wet pulp) contained no detectable residue (<0.005 mg/kg). Diffusion juice was fortified with components of the residue at 13 mg/kg and treated with lime water under conditions simulating plant processing. No detectable residue (<0.005 mg/kg) remained in the juice. No residues were detectable in beet pulp fortified at 0.13 mg/kg after simulated plant drying (150°C for 30 minutes).

<u>Cereals</u>. Two lots of <u>sorghum</u> grain were processed. The residues measured in the processed fractions are shown in Table 66 (Anon, undated e).

Table 66. Aldicarb residues in sorghum grain and processed fractions.

Sample	Residue, mg/kg		
Grain	0.09	0.04	
Bran	0.41	0.04	
Shorts	0.05	0.02	
Flour	0.03	< 0.02	

Field-treated <u>maize</u> grain was dry-milled with a laboratory-scale mill to resemble a typical industrial process. The separated germ was ground to fine meal and extracted with petroleum ether to obtain maize oil. The aldicarb residues were determined in the processed fractions (Union Carbide Agricultural Products Co., 1985). The results are shown in Table 67. The limit of determination was 0.005 mg/kg for grain, hulls and meal.

<u>Sugar cane</u>. Field-treated sugar cane stalks, juice and foliage were analysed separately in 8 trials in India. The residues were below the limit of determination (0.003 mg/kg) in all samples (Singh *et al.*, 1981).

<u>Oil seeds</u>. Cotton seed samples from trials with deliberately exaggerated treatments in four States of the USA were processed to obtain oil, meal and hull fractions. Solvent-extraction of the oil, which is less severe in thermal exposure than the screw-press method, led to somewhat higher residues in the meal. The alkaline refining process degrades all residues containing the carbamate moiety (Union Carbide Corporation, 1979b). The results are given in Table 68.

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Table 6/	/\ Idicarh	racidilac 1	ın r	arocaccad	tractions	Of ma174
Table 67.	Aluicaro	residues i	ш	nocesseu	nacuons	or marze.

Product	Samp	le 1	Sam	ple 2
	% of final solids	Residue, mg/kg	% of final solids	Residue, mg/kg
Grain		0.013		0.014
Meal	80	0.01	81.7	0.009
Hulls	1.4	0.031	1.1	0.038
Germ	18.6		17.2	
Oil		0.005^{1}		0.005^{1}

¹ This apparent residue, found in both the treated and control oil, was present in the petroleum ether used to extract the oil

Table 68. Aldicarb residues in processed fractions of cotton seed.

Product	Residue, mg/kg, in sample ¹					
	A	В	C	D^2	E^2	F
Cotton seed	0.05	0.02	0.06	< 0.02	0.027	0.02
Hulls	0.15	0.03	0.17	0.018	0.024	0.005
Oil (crude extracted)	< 0.003	< 0.003	0.004	-	-	< 0.003
Oil (refined extracted)	< 0.003	< 0.003	< 0.003	-	-	< 0.003
Oil (crude pressed)	< 0.003	< 0.003	0.004	< 0.003	< 0.003	< 0.003
Oil (refined pressed)	< 0.003	< 0.003	< 0.003	-	-	< 0.003
Meal (extracted)	0.014	0.002	0.03	-	-	< 0.003
Meal (pressed)	<0.003	0.002	0.006	<0.003	<0.003	<0.003

¹ Treatment of samples:

A: 4.5 kg ai/ha, 91 days PHI; B: 1.12 + 9 kg ai/ha, 108 days PHI; C: 2.24 + 2.24 + 4.5 kg ai/ha, 82 days PHI; D: 0.56 + 4.6 kg ai/ha, 99 days PHI; E: 0.56 + 4.5 kg ai/ha, 76 days PHI; F: 1.12 + 4.5 kg ai/ha, 104 days PHI

Four lots of field treated <u>peanuts</u> were processed to oil and meal. The residues in the processed fractions are shown in Table 69. (Union Carbide Corporation, 1973).

<u>Coffee</u>. Green coffee containing aldicarb residues of 0.11 mg/kg was processed to yield roasted coffee, spent grounds, and instant coffee. No aldicarb residues were detectable (<0.02 mg/kg) in any of these products (Romine, 1986).

² Samples D and E were pressed but not extracted. The pressed crude oil was not refined

Table 69. Aldicarb residues in processed peanut fractions.

		Res	sidue, mg/kg			
Whole dry nuts	Hulls	Kernels	Meal	Crude oil		
				Screw-pressed	Solvent-extracted	
Alabama						
0.01	0.01	< 0.002				
0.01	0.02	0.007	0.012	<0.003	<0.003	
0.01	0.02	0.002				
Georgia						
0.01	< 0.01	0.003				
0.02	0.02	0.003	< 0.03	<0.03	<0.03	
0.02	0.02	0.04				
North Carolina						
0.05	0.13	0.008				
0.17	0.48	0.017	< 0.003	<0.003	< 0.003	
0.15	0.35	0.013				
Texas						
<0.01	0.02	< 0.01				
<0.01	< 0.01	< 0.01				
0.02	< 0.01	0.01				
Virginia						
0.02	0.03	0.006				
0.02	0.04	0.004	0.005	<0.003	< 0.003	
0.04	0.05	0.006				

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

In 1987, a National Food Survey was developed in the USA in order to assess dietary exposure to aldicarb more accurately (Union Carbide, 1987). The survey was structured to determine the potential dietary exposure to aldicarb and its primary metabolites at the point of purchase.

Five agricultural commodities were analysed: bananas, oranges, potatoes, sweet potatoes and grapefruit. Together, these five foods represent almost 90% of the potential dietary exposure to aldicarb.

The A.C. Nielsen National Food Index was used to provide statistically valid sampling of food stores throughout the USA. The samples represented the top 25 market locations and cities as well as smaller markets. The sampling covered 82% of the total sum spent by retail consumers on these commodities in grocery stores.

Seventy-five locations were selected nation-wide and all major supermarket chains were represented. To account for possible seasonal variations in residue levels, samples were taken in winter (February), spring (May) and autumn (September). Samples of the five commodities included both

fresh and freshly processed foods. All analyses were "blind" in order to avoid bias. The results are shown in Table 70.

Table 70. Aldicarb residues detected in the national food survey in the USA in 1987.

Sample	Season	Distribution of residues ¹	Residues, mg/kg		
			Mean ²	95%≤²	Max.
BANANAS fresh fruit	Winter Spring Autumn	6/75 (8%) 14/75 (17%) 18/75 (24%)	0.011 0.019 0.018	0.022 0.071 0.066	0.051 0.216 0.227
BANANAS	Winter	42/72 (58%)	0.012	0.018	0.029
processed	Spring	30/74 (41%)	0.010	0.013	0.018
infant	Autumn	2/69 (3%)	0.010	0.010	Trace
ORANGES	Winter	0/75 (0%)	$0.010^{3} \\ 0.010^{3} \\ 0.010^{3}$	0.010	ND
fresh	Spring	0/75 (0%)		0.010	ND
fruit	Autumn	0/73 (0%)		0.010	ND
ORANGES	Winter	2/75 (3%)	0.010	0.010	0.7
orange	Spring	11/75 (15%)	0.010	0.010	7
juice	Autumn	9/75 (12%)	0.010	0.010	10
ORANGES	Winter	4/75 (5%)	0.010	0.010	8
infant	Spring	3/75 (4%)	0.010	0.010	7
orange juice	Autumn	12/70 (17%)	0.010	0.010	10
GRAPEFRUIT	Winter	1/75 (1%)	0.010	0.010	3
fresh	Spring	13/75 (17%)	0.012	0.021	46
fruit	Autumn	1/73 (1%)	0.010	0.010	Trace
WHITE POTATOES fresh	Winter	7/75 (9%)	0.010	0.010	15
	Spring	9/75 (12%)	0.010	0.025	46
	Autumn	11/75 (15%)	0.019	0.068	188
SWEET POTATOES fresh	Winter Spring Autumn	0/57 (0%) 1/68 (1%) 7/72 (10%)	0.010^3 0.010 0.012	0.010 0.010 0.027	ND 9 65
SWEET POTATOES processed infant	Winter Spring Autumn	0/75 (0%) 0/75 (0%) 0/69 (0%)	$\begin{array}{c} 0.010^{3} \\ 0.010^{3} \\ 0.010^{3} \end{array}$	0.010 0.010 0.010	ND ND ND

¹ No. of detectable [including <LOD] residues/total no. of samples, and (% of samples with detectable residues)

ND = Not detectable

A residue monitoring programme was conducted in Florida, USA, during 1993 to estimate the potential residues of aldicarb and its major metabolites in fresh-market oranges (Tew, 1994). The design of the survey involved collection of samples of oranges from groves that (1) had been treated with aldicarb, (2) had mature fruit on the tree at harvest, and (3) were intended for the fresh market.

869 individual oranges were analysed, of which 467 contained residues below the limit of determination (LOD) and 244 had no detectable residues. The remaining 158 samples had quantifiable residues with a mean of 0.025 mg/kg and a standard deviation of 0.020 mg/kg. The residues found are shown in Table 71.

The distribution of residues across all fresh-market oranges was gauged from market share estimates of the proportion of the fresh-market oranges treated with aldicarb, and the proportion of the treated area that had mature fruit on the tree at the time of treatment. Data collected by Rhône-Poulenc

² All levels below 0.01 mg/kg, including undetectable residues, were calculated as 0.01 mg/kg

³ No residues were detectable

indicate that 9% (12,744 acres) of the estimated 141,867 acres of fresh-market oranges in Florida and California were treated with aldicarb and that only 1,252 acres were treated when mature oranges were on the trees. Thus an estimated 0.9% of all the fresh-market oranges were from groves treated when mature oranges were on the trees. The authors assumed that all residues on the fruits from the remaining treated trees were half the LOD and that untreated oranges contained no residues. The estimated mean and SD of the residue distribution for all fresh-market oranges would then be 0.0005 mg/kg and 0.002 mg/kg respectively. The proportion of fresh-market oranges expected to have residues > 0.09 mg/kg was estimated to be 0.00003.

Table 71. Aldicarb residues in pulps of individual oranges of known spraying history from Florida, USA.

Rate, PHI, days No. of samples analysed			Resi	dues, mg/kg
			Mean	Max
3.4	16-18	50	0.009	0.03
4.9	26	25	0.007	0.02
4.2	28	44	0.009	0.06
4.2	30	25	0.010	0.03
4.2	35	25	0.010	0.04
3.4	54-56	50	0.006	0.01
5.5	35	50	0.008	0.04
5.5	17-18	50	0.006	0.02
3.4	7	50	0.005	0.01
3.4	7	50	0.005	0.01
5.5	56-61	50	0.006	0.02
3.4	6	50	0.005	0.01
5.5	34-48	50	0.042	0.12
5.0	27	50	0.006	0.02
5.5	58	50	0.014	0.07
4.9	33	50	0.005	0.01
4.2	23	50	0.005	0.01
3.5	26	25	0.006	0.02
5.0	26	50	0.005	0.01
4.8	8	25	0.005	0.01

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

Crop	Country	MRL, mg/kg
Bananas	Argentina	0.01
	Brazil	0.3
	Canada	0.5
	Denmark	0.5

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Crop	Country	MRL, mg/kg
	Finland	0.05
	France	0.2 pulp
	Japan	0.5
	Netherlands	0.5
	South Africa	0.5
	Spain	0.3
	Venezuela	0.3
Beans	Argentina	0.01
	Brazil	0.02
	Germany	0.1
	Japan	0.1
	Netherlands	0.1
	USA	0.1
Brussels sprouts	Belgium	0.05
	Netherlands	0.05*
Cereals (small grain)	Australia	0.02
	France	0.02* grain
	France	0.04 straw
	Japan	0.02
Citrus fruit	Argentina	0.3
	Australia	0.05
	Brazil	0.3
	Canada	0.1
	Denmark	0.2
	Finland	0.2
	France	0.2
	Germany	0.3 fruit
	Germany	0.1 juice
	Italy	0.2
	Japan	0.3
	South Africa	0.2
	Spain	0.2
	USA	0.3 fruit and juice
	USA	0.6 dried pulp
	Venezuela	0.3
Coffee	Argentina	0.1
	Brazil	0.1
	Germany	0.1
	Japan	0.1
	Kenya	0.1
	Netherlands	0.1
II	ľ	

Crop	Country	MRL, mg/kg
	USA (Puerto Rico)	0.1
	Venezuela	0.3
Cotton	Argentina	0.1
	Australia	0.05
	Brazil	0.1
	Germany	0.1
	Japan	0.1
	Netherlands	0.1
	South Africa	0.1 seed
	Spain	0.1 seed
	USA	0.1 seed
	Venezuela	0.1 seed
	Venezuela	0.3 husk
Garlic	Argentina	0.01
Grapes	Australia	0.05
	Japan	0.05
	South Africa	0.2 wine and table
Macadamia nuts	South Africa	0.05
Maize	France	0.05
	Germany	0.05
	Japan	0.05
	UK	0.05 grain
	UK	5 fodder
Meat	Netherlands	0.01
	USA	0.01 cattle, goat, hog, horse and sheep
	USA	0.01 cattle, goat, hog, horse and sheep fat
	USA	0.01 cattle, goat, hog, horse and sheep meat by-products
Milk	Netherlands	0.01
	USA	0.002
Onion	Argentina	0.01
	Denmark	0.05
	Germany	0.05
	Japan	0.05
	Netherlands	0.05*
	UK	0.05
	South Africa	0.2
Peanut	Argentina	0.05
	Germany	0.05
	Japan	0.05 dry
	Netherlands	0.05*
	South Africa	0.1 nut
II	l	T

	T	
Crop	Country	MRL, mg/kg
	South Africa	1.0 hay
	USA	0.5 hulls
	USA	0.05 nut
Peas	France	0.02
Pecan nuts	Japan	0.5
	Netherlands	0.5
	South Africa	0.05
	USA	0.5
Peppers	Argentina	0.01
Pineapples	South Africa	0.05
Potatoes	Argentina	0.01
	Australia	0.2
	Belgium	0.05
	Brazil	1.0
	Canada	0.5
	Denmark	0.2
	Finland	0.05
	Germany	0.5
	Italy	0.2
	Japan	0.5
	Netherlands	0.5
	South Africa	1.0 seed and table
	Spain	0.5
	UK	0.5
	USA	1.0
	Venezuela	1.0
Sorghum	Netherlands	0.2
	USA	0.2 grain
	USA	0.5 fodder
Soya bean	Germany	0.05
*	Japan	0.02
	USA	0.02 bean
	USA	1.0 tops
	Venezuela	0.1
Strawberries	Australia	0.2
	Germany	0.05
	Japan	0.2
	Netherlands	0.02*
Sugar beet	Belgium	0.02*
-	Canada	0.1
	France	0.03* roots

Crop	Country	MRL, mg/kg
	Germany	0.05
	Hungary	0.05
Italy		0.05
	Japan	0.05
	Netherlands	0.02*
	Spain	0.05 roots
	Spain	1.0 leaves
	UK	0.05
	UK	1.0
	USA	0.05 beet
	USA	1.0 tops
Sugar cane	Argentina	0.01
	Australia	0.02
	Japan	0.02
	South Africa	0.1
	USA	0.02
	USA	0.1 fodder and forage
Sweet Potatoes	Argentina	0.01
	Japan	0.1
	Netherlands	0.1
	USA	0.1
Tomatoes	Argentina	0.01
	Italy	0.1
	South Africa	0.2

APPRAISAL

Aldicarb was first evaluated in 1979, and last reviewed in 1993. It is included in the CCPR periodic review programme.

Aldicarb is a systemic insecticide, nematicide and miticide, available commercially only as low-assay (50, 100 or 150 g/kg) granular formulations. To protect field crops the granules are applied in seed furrow, band or overall treatments (either pre-plant or at planting), as well as in post-emergence side-dress treatments. In orchards and vineyards the pesticide is applied at the intensively developing stage of the trees or vines (e.g. at the spring flush of foliage, before bud swell or around bud break) in bands along the row of vines, and along the dripline on both side of or around the trees, or uniformly around the trees. The application rate ranges from 0.34 to 11.25 kg ai/ha, depending on the crop and pests to be controlled. The granules must be incorporated into the soil immediately after application. Soil moisture is required to release the active ingredient from the granules, so irrigation or rainfall should follow application. The number of applications is usually restricted to one per year in food- and feed-producing plants except bananas, coffee, cotton, macadamia nuts and potatoes.

Aldicarb is rapidly absorbed by the plant's root system and moves throughout the plant, mainly in the xylem. The plants are protected for several weeks.

The fate of residues was studied in rats, dogs, cows, goats, hens, various plants and soil.

The basic metabolic pathway appears to be the same in all species studied. Aldicarb is rapidly oxidized to the relatively stable sulphoxide; then, more slowly, a small proportion of the sulphoxide is oxidized to aldicarb sulphone. Aldicarb, aldicarb sulphoxide and aldicarb sulphone are also readily converted to the corresponding oximes and nitriles, which are in turn slowly degraded to the corresponding aldehydes, acids, and alcohols, none of which are toxicologically significant.

Aldicarb sulphoxide and aldicarb sulphone fed separately to rats or in a mixture to dairy cows were eliminated similarly to the parent aldicarb. The metabolites present were the same.

A <u>lactating cow</u> fed [³⁵S]aldicarb as a single dose of 0.1 mg/kg bw eliminated over 96% of the administered radioactivity within 540 hours after treatment. The percentages of the total dose detected in the urine, milk, and faeces were 90.2, 3.0 and 2.9 respectively.

Lactating cows were administered aldicarb and aldicarb sulphone (1:1 molar ratio) labelled with ¹⁴C in the methylthio group at levels of 0.12, 0.6 or 1.2 ppm in the diet for 14 days. The dose level did not alter the magnitude or nature of the residues eliminated daily in the urine, milk and faeces. The percentages of the dose excreted by these routes were 92, 1, and 3 respectively.

Residues in the milk expressed as aldicarb were between about 0.1 and 0.2% of the levels in the feed. About 15% of the radioactive residue in the milk was aldicarb sulphone and about 4% was the sulphoxide. The total aldicarb equivalents in the liver were 0.029, 0.12 and 0.16 mg/kg from the three treatments respectively, when the animals were slaughtered 18 hours after the last treatment. Twenty-six other tissue samples contained either much lower or undetectable residues.

Two <u>lactating goats</u> were administered [*S-methyl-*¹⁴C]aldicarb in gelatin capsules at a level equivalent to 2.5 ppm in the feed (or 0.165 mg/kg bw) for ten days and a third served as a control. The goats were slaughtered within 6-8 hours after the last dose was given. Of the applied dose, 61.2% was eliminated in the urine, 11.3% in the faeces, and 1.1% in the milk. Only 0.2% of the applied dose was found in the respiratory gases and 0.10% in the tissues at the end of the 10-day dosing period. The overall recovery of the applied dose was 74%.

The total ¹⁴C residues in the milk reached a plateau within 7 days and a maximum concentration of 0.1 mg/kg by the end of the treatment period. Tissues contained low concentrations of total ¹⁴C residues with a maximum of 0.52 mg/kg in the liver. No aldicarb, *per se*, was found in the urine, milk or tissues. Only traces of the sulphoxide and sulphone were detected in the milk and some tissues. A maximum of 0.26 ½ g/kg and 1.5 ½ g/kg of the total carbamate residues were found in the milk and liver respectively. No carbamates were detected in the brain, mammary glands or omental fat, and only 0.1 mg/kg of the sulphoxide in the peripheral fat. The largest single component of the ¹⁴C residues in the milk and all the tissues was identified as aldicarb nitrile sulphone, which represented 54% to 68% in the milk and varied in the tissues from 7.7% in the liver to 79% in the peripheral fat. Other metabolites identified in the milk and tissues were non-carbamate products similar to those in the urine. With the exception of aldicarb nitrile sulphoxide, all the non-carbamate metabolites in the milk and tissues were less than 1% of the total ¹⁴C. Aldicarb nitrile sulphoxide appeared to be significant only in the kidney (4.9%), liver (2.5%) and peripheral fat (1.2%). Insignificant amounts (<1.0%) of aldicarb nitrile sulphoxide were detected in all the other tissue and milk samples analysed.

The results of these studies indicate that the rapid excretion of aldicarb and its biotransformation products by dairy animals would prevent the accumulation of residues in milk or tissues.

In a feeding study mature, laying white leghorn hens were given daily doses of a 1:1 molar

mixture of aldicarb and aldicarb sulphone, each labelled with ¹⁴C in the *S*-methyl group. Three groups of six birds each were treated with 0.005, 0.05 or 1.0 mg/kg/day corresponding to levels of 0.1, 1.0 or 20.0 ppm aldicarb equivalents in the feed. In each of the three treated groups, three of the birds were killed 12 hours after the cessation of feeding and the other three seven days after the last treatment. At the highest level fed, 85% of the total administered radioactivity appeared in the droppings and 5% was found in the eggs. The total radioactive residues 12 hours after the last treatment amounted to 0.07 and 0.79 mg/kg in eggs, and 0.06 and 0.69 mg/kg in breast meat at the 1 ppm and 20 ppm feeding levels respectively. The levels of radioactivity in muscle, fat, and skin with fat were comparable to that in breast muscle, but those in liver (0.14 mg/kg and 1.4 mg/kg) and kidney (0.12 mg/kg and 1.4 mg/kg) were higher. The total radioactivity in these organs declined significantly during the seven days after withdrawal of the pesticide. Characterization of the radioactive material in the eggs and tissues showed that the total toxicologically significant residues (containing the carbamate moiety) were present at 0.003 mg/kg after 28 days of continuously feeding 1:1 mixtures of aldicarb plus aldicarb sulphone at the level of 20 ppm in the diet.

In another study ten laying hens were dosed twice daily with [S-methyl-14C]aldicarb for seven consecutive days at a rate equivalent to 3.54 ppm in the diet. At the end of the dosing period, animals were slaughtered and tissues collected for analysis. The highest concentration of [14C]aldicarb in tissues was observed in the liver with an average concentration of 0.42 mg/kg. The kidney contained the next highest concentration with an average of 0.31 mg/kg. Plasma contained an average of 0.15 mg/kg and red blood cells an average of 0.12 mg/kg. Muscle, fat and skin with adhering fat contained less than 0.1 mg/kg. The average concentrations of total aldicarb equivalents in egg yolks and whites over the sevenday dosing period were 0.09 mg/kg and 0.11 mg/kg respectively, giving a calculated average concentration in whole egg of 0.1 mg/kg. Aldicarb nitrile sulphone was the most common free metabolite isolated from liver, muscle, egg yolk and egg white. In liver, it was present at a level of 0.028 mg/kg aldicarb equivalents (6.7% of the total radioactive residue, TRR). However, the major free metabolite in liver was aldicarb sulphone acid which was present at a level of 0.105 mg/kg aldicarb equivalents (25.4% TRR). Two additional minor free metabolites were isolated from the liver: methanesulphonic acid, 0.029 mg/kg aldicarb equivalents (7.0% TRR), and aldicarb nitrile sulphoxide, 0.003 mg/kg aldicarb equivalents (0.8% TRR). No intact carbamates (aldicarb, aldicarb sulphoxide or aldicarb sulphone) were detected in the tissues or eggs.

The metabolic pathways of aldicarb have been studied in potatoes, sugar beet, cotton, peanuts, tobacco, spearmint and lettuce.

The initial step in the metabolism of aldicarb in plants is thio-oxidation by plant enzymes to aldicarb sulphoxide. This conversion occurs rapidly since no parent aldicarb is found in the plant after a few weeks. Aldicarb sulphoxide is subsequently metabolized, mainly by hydrolysis to the oxime sulphoxide. It is also converted to aldicarb sulphone by slow thio-oxidation. Both of these metabolites suffer extensive degradation through hydrolysis, elimination, oxidation, reduction and conjugation reactions to yield the corresponding oximes and the resulting alcohols and their glycoside conjugates, amides, nitriles and carbonic acids. Possible intermediate metabolites are the aldehydes of the sulphoxide and sulphone. The major plant metabolite at harvest in the plants studied, in terms of percentage of the applied dose of aldicarb, was aldicarb alcohol sulphoxide (2-methyl-2-(methylsulphinyl)propanol), which may be conjugated with plant sugars in the form of water-soluble glycosides. No evidence of demethylation or reduction of aldicarb sulphone to aldicarb sulphoxide or aldicarb has been found in any of the plant studies.

There has been no evidence of conjugated carbamate metabolites in plants resulting from aldicarb treatment. Consequently, the only significant terminal carbamate-containing residues in plants following aldicarb treatment are aldicarb sulphoxide and aldicarb sulphone, both of which are toxicologically significant. In foliage, the ratio of aldicarb sulphoxide to aldicarb sulphone changes in favour of the latter with time.

Since the amount of each residue component represents the final yield of continuous uptake and biotransformation processes the half-life of the individual metabolites cannot easily be calculated.

The metabolic pathway for aldicarb is qualitatively similar in all the plant species studied (potato, sugar beet, cotton, peanut and tobacco). An identical pattern of degradation products has been described whether the chemical was introduced by injection, leaf uptake, topical stem application, or soil treatment. The last is the only method of application commercially recommended.

Over the 90-day growing season there was an effective systemic uptake of aldicarb by <u>potato</u> plants from soil which resulted in the accumulation of the residues in the foliage for at least 60 days. Thereafter, the residues declined as a result of the loss of the older leaves and dilution of the residues by plant growth. Aldicarb sulphoxide and sulphone in the tubers amounted to 63% and 14.7% of the total radioactive residue at 60 and 90 days after planting respectively.

In <u>sugar beet</u> roots aldicarb sulphoxide and sulphone amounted to about 21% and 24% of the total radioactive residue at 90 and 140 days after planting and in sugar beet forage the corresponding proportions were 25.6% and 40.6%.

The concentration of total radioactive residues in <u>cotton</u> foliage declined rapidly. The following residues were measured at various intervals after application: 242 mg/kg (14 days); 53.5 (37 days); 9.2 (72 days) and 8.9 mg/kg (146 days).

The uptake by <u>peanut</u> plants from soil was slow. Only 1.4% and 2.8% of the applied radioactivity was detected in the plants at 21 and 35 days respectively. The maximum uptake (7.5%) was reached at 56 days. It should be noted that while the percentage of the applied radioactivity found in the plants continued to increase until 56 days, the concentration of ¹⁴C residues declined after the 21-day sampling because the rate of dilution by plant growth was greater than the rate of uptake from the soil.

The absorbed radioactivity accumulated preferentially in the foliage and at 126 days only 0.25% of the applied material was translocated to the nuts (shells plus kernels). The proportion of unextracted ¹⁴C was higher in the roots, pegs, shells and kernels than in the foliage.

Two studies have been conducted to measure the uptake of residues of aldicarb by <u>rotational crops</u>. The first consisted in treating a Norfolk sandy loam soil with 5.6 kg ai/ha of [S-methyl-\frac{14}{2}C] aldicarb and ageing under field conditions for four- and twelve-month periods. Of the applied [\frac{14}{2}C] aldicarb, approximately 60% was lost by degradation (as CO₂) and 20% by leaching during the four month ageing period. At the end of the period lettuce, turnip and barley were planted separately in the treated soil and grown to maturity. Only traces of carbamate residues were detected in the fourmonth plantings. These ranged from 0.02 to 0.05 mg/kg in all the plant parts except barley straw which contained 0.72 mg/kg.

In the second study covering seven test sites in six States of the USA, aldicarb was applied to three primary crops, cotton, potatoes and sugar beet, according to registered uses and generally at the maximum use rate. Residues were found in most rotational crops especially at the early plant-back intervals ranging from five to twelve months, but the residues were generally below the limit of determination (LOD). The maximum residues (mg/kg aldicarb sulphone) measured in rotational crops were: alfalfa (0.07), barley forage (0.15), carrot (0.04), and wheat forage (0.7). Residues in the other crops (onion, lettuce, broccoli, cucumber, cantaloupe, tomato, maize and oats) were below the LOD.

The fate of aldicarb in <u>soil</u> has been extensively investigated under both laboratory and field conditions. The main transformation pathway involves oxidation to aldicarb sulphoxide as a major

product and to a lesser extent to aldicarb sulphone. These products are degraded to the corresponding oximes and nitriles. Extensive decomposition of the transformation products leads to the formation of carbon dioxide as the major end product.

The experiments showed that soil micro-organisms were active in the degradation of the pesticide and contributed significantly to its short persistence in soil. Aldicarb sulphoxide and water-soluble metabolites were the major products of metabolism by various fungi. Traces of aldicarb sulphone, aldicarb sulphoxide oxime, aldicarb nitrile sulphoxide, aldicarb sulphone oxime and aldicarb nitrile sulphone were also detected in the organosoluble fraction. Water-soluble metabolites consisted of aldicarb alcohol sulphone and amide as major products, moderate amounts of aldicarb alcohol sulphoxide and amide, and small amounts of acids, presumably derived from aldicarb sulphoxide and aldicarb sulphone. The metabolic pattern of aldicarb sulphoxide was similar to that of aldicarb.

The rate of dissipation of aldicarb and its carbamate metabolites in the soil varies, and depends on several factors such as mineral and organic matter contents, nature of the micro-organisms present, temperature and moisture.

No important differences could be attributed to pH within the range 6 to 8. There was however variation among the soil types in their capacity to degrade aldicarb at different moisture levels. It is apparent that the moisture level is a critical factor in the fate of aldicarb in soils. In general, degradation was slower in the deeper layers than in the top layers of the soil. Transformation of aldicarb to its sulphoxide and sulphone decreased markedly at lower temperature and moisture content.

Co-distillation from the moist soil resulted in essentially insignificant losses, which ranged from 0.01% to 0.08% of the applied dose. No carbamates were detected in the volatilized radioactivity.

Under the regular farming and environmental conditions of six States of the USA, the aldicarb sulphoxide and sulphone residues in soil ranged from undetected to 0.27 mg/kg five to ten months after the last application at maximum recommended rates.

In <u>aqueous solution</u> aldicarb was more susceptible than aldicarb sulphoxide and aldicarb sulphone to UV (290 nm) irradiation. The half-life of aldicarb was 8 to 12 days and aldicarb sulphone 36 to 38 days, while aldicarb sulphoxide was stable to UV irradiation with only 2% degradation in 14 days. This photostability was attributed to the lack of light absorption above the 290 nm region by aldicarb sulphoxide. Under field conditions therefore, other environmental agents such as microbes and plant absorption and metabolism are responsible for its ultimate dissipation.

In another study sterile water buffered at pH 5 containing [S-methyl-14C]aldicarb at 10.6 mg/kg was exposed to artificial sunlight comparable to natural sunlight for a total period of 360 hours of continuous irradiation at 25°C. The parent concentrations in unirradiated samples changed insignificantly, remaining within the range 95.0 to 98.4% of the initial level. The concentrations of aldicarb in irradiated samples decreased from 98.4% to 0.4% of the total radioactivity in 168 hours of exposure. There were two major products: aldicarb oxime, which reached a maximum of 64.6% of the total activity by 168 hours, and aldicarb nitrile which reached a maximum of 48.2% by 360 hours.

The movement of aldicarb in soils has been extensively studied under both laboratory and field conditions, but only summary information was provided on leaching and the fate of residues in ground-water. Laboratory studies show that aldicarb residues do not bind significantly to inorganic soil particles, but can be retained to some extent by soil organic matter, so that the rate of movement may be equal to or as little as one-tenth that of the movement of water. The movement of water in the soil under normal agricultural conditions is determined by the rainfall, irrigation, evaporation/transpiration, and the water-holding capacity of the soil. The movement of residues into deeper soil layers or groundwater is influenced by the rate of degradation of the compound in the soil, which is a function of the

pH, moisture, temperature, texture and microbial activity in or of the soil.

Field studies indicated that the half-life of aldicarb residues in shallow and medium-deep ground-water (down to 10 m) ranges from about one month to three years. The residues in ground-water moved laterally as well as vertically. A worst-case calculation indicated that under unfavourable conditions the degradation or effective dilution of residues in ground-water may take up to 100 years. Because of the persistence of the residues in ground-water, appropriate management practices are to be introduced and followed in order to protect ground-water from contamination.

For <u>residue analysis</u>, the toxicologically significant carbamate residues (aldicarb, aldicarb sulphoxide and aldicarb sulphone) are usually extracted from plant foliage, fruits and vegetables with mixed solvents, from oils with hexane followed by partition into acetonitrile, and from soil with water. After clean-up the residues may either be oxidized to aldicarb sulphone and determined by GLC, or separated by HPLC. In the latter case the individual compounds can be detected by a fluorescence detector after post-column derivatization.

The limit of determination by GLC is usually 0.02~mg/kg but lower levels down to 0.001~mg/kg could be achieved in peanut oil.

The HPLC separation and detection provide the advantage of determining the individual toxicologically significant residues with limits of determination of 0.01 mg/kg in plant materials, 0.001 mg/kg in soil and 0.1 i g/kg in ground-water for each residue component.

Since the sum of the toxicologically significant residues has to be calculated the limits of determination are about the same with both methods, which are suitable for regulatory purposes.

Attention has to be paid to the expression of the residue. Since aldicarb sulphone is determined by GLC, the residues found by oxidation and GLC are most frequently expressed as aldicarb sulphone. In HPLC procedures the expression of the total residue should take into account the differences in the molecular masses of the components (aldicarb sulphone/aldicarb = 1.168; aldicarb sulphoxide/aldicarb = 1.084; aldicarb sulphone/aldicarb sulphoxide = 1.077).

In the supervised trials evaluated by the Meeting the residues were generally determined by GLC and the results, with few exceptions, were expressed as aldicarb sulphone.

The typical <u>recommended application</u> is by soil treatment in band or furrow, drilling 5-7.5 cm below the seed line at planting or sowing. Application must be followed by immediate and complete incorporation into soil to a depth of about 30-80 mm.

The Meeting evaluated <u>supervised field trials</u> on citrus fruits, grapes, onions, garlic, cauliflower, cabbage, dry peas, soya beans, carrots, swedes, sweet potatoes, sugar beet, wheat, barley, sorghum, maize, sugar cane, pecans, cotton, peanuts and coffee beans.

Since the residue information for potatoes (1990 JMPR) and Brussels sprouts (1993 JMPR) has been recently reviewed, the residues in these commodities have not been evaluated by the Meeting.

The manufacturer informed the Meeting that owing to changes in the use pattern for <u>bananas</u>, the available residue data for aldicarb do not reflect the currently recommended GAP. A programme of residue trials in accordance with the new use pattern is in progress, and will be made available to the JMPR when it has been completed.

The Meeting therefore withdrew the previous estimate of 0.5 mg/kg.

Citrus fruits. Supervised field trials and specific studies on several varieties of orange, mandarin,

grapefruit, lemon and lime were reported from 13 countries.

The results indicated that the pesticide incorporated into the soil was translocated from the tree roots to the developing fruit and the initial residue was then reduced by growth dilution as the fruit matured. Also, the essentially mature fruit on the tree at the time of treatment did not accumulate the translocating pesticide to the same extent as the smaller more actively growing fruit. Consequently the residues are higher in immature green fruit than in mature fruit on the tree at the same time.

The analysis of orange peel and pulp showed that the parent aldicarb was practically absent, and the toxic residue was composed of aldicarb sulphoxide and aldicarb sulphone in the ratio of about 5:1.

Field trials on <u>oranges</u> in 6 countries were considered. Following application at recommended rates, the residues were generally at or below 0.1 mg/kg 100 days after treatment. However, higher residues up to 0.2 mg/kg were also detected during the period of 31-250 days following the last application. No substantial differences in residue patterns at various time intervals after application were observed.

Residues in <u>lemons</u>, <u>limes</u> and <u>grapefruit</u> were similar, both in magnitude and in the variation of residue levels in the maturing fruit with time after treatment. Since their culture is essentially similar, the residue data from all three crops were considered together for estimating maximum residue levels. The residues in various fruits resulting from recommended application rates up to 56 kg ai/ha showed similar patterns and were at or below 0.1 mg/kg in most of the samples. Two samples contained residues in the range >0.1 to <0.2 mg/kg and two from ≥ 0.2 to <0.3 mg/kg. In trials in the USA with 11.2 kg ai/ha the residues did not exceed 0.3 mg/kg in the mature fruits.

The Meeting took into account the similar growing conditions, use patterns and residue distributions in the different fruits, and the reduction of approximately 20% of the measured residue levels when they are expressed as aldicarb, and agreed to maintain the current recommendation of 0.2 mg/kg for citrus fruits.

Trials on grapes were evaluated from eight countries, four of which have registered uses. The maximum residue in grapes is likely to occur at about 60 days after treatment and then decline slowly. There was no significant difference in the residue levels occurring in various grape varieties from 100 to 130 days.

The residues in both fresh juice and wine made from Cabernet Sauvignon grapes consisted of a 1:1 ratio of aldicarb sulphoxide to sulphone with no parent aldicarb.

The highest residues were observed in trials in the USA and South Africa. The use recommendation has been withdrawn in the USA, so the evaluation was based on South African GAP, where the granules are applied in a band between the rows and the maximum rate of 0.75 g ai/m² is equivalent to 3.75 kg ai/ha taking into account the band and row widths. Although a PHI of 120 days is registered in South Africa, the pesticide is to be applied at the time of bud swelling. The residues measured in harvested grapes (<0.03-0.16 mg/kg) were therefore considered for estimating the maximum residue level. The registered uses in Australia, Egypt and Peru (used as a reference for trials in Chile) lead to lower residues.

The Meeting estimated a maximum residue level of 0.2 mg/kg for grapes.

Several trials on <u>onions</u> were reported from Israel, the UK and The Netherlands, which were in accordance with registered uses. Residues in mature onions ranged from undetected (<0.01-0.04 mg/kg) to 0.08 mg/kg. Two trials on garlic resulted in similar residues.

The Meeting estimated a maximum residue level of 0.1 mg/kg for onions. The data for garlic were not sufficient to estimate a maximum residue level.

The 1990 JMPR confirmed the Codex MRL of 0.5 mg/kg on <u>potato</u> on the basis of available data. In view of the acute toxicity of aldicarb, its inclusion in the periodic review, and the understanding that new data were available the Meeting decided to make the current recommendation temporary pending the reconsideration of all available data reflecting current use patterns. Consequently, the supervised trials reported from The Netherlands will also be evaluated at a future Meeting.

The Meeting noted the residue data on <u>Brussels Sprouts</u> evaluated in 1993, and reaffirmed the previous recommendation of 0.1 mg/kg.

Residues were between <0.02 and 0.11 mg/kg in <u>cauliflowers</u> 56-90 days after soil treatments with recommended and 1.5-fold rates. In <u>cabbages</u> the residues ranged from <0.01 to 2.7 mg/kg within 67-81 days following applications with recommended and 1.5-fold rates. The trials were in the UK in 1977 and 1978.

The Meeting considered the limited data insufficient to estimate a maximum residue level for cabbage or cauliflower.

Trials on <u>dry beans</u> from 4 countries were considered. In Brazilian trials the part of the plant which was analysed was not clearly indicated and the analytical method used had a LOD of 0.2 mg/kg, so the results were not taken into account. Aldicarb residues in the beans declined rapidly during the last few weeks of the season while the beans were developing and drying. In US trials the maximum residue found in dry beans at harvest following recommended application was 0.02 mg/kg. After applications at a 1.5-fold rate the residues at harvest were mostly below the limit of determination (<0.02 mg/kg). Quantifiable residues (3 x 0.03, 0.07 mg/kg) were found in four of 22 samples. The residues in samples from trials in other countries were also generally below the limit of determination.

Residues in dry bean forage at harvest were below 0.5 mg/kg in 21 of 26 samples. The remaining five contained residues of 0.8 mg/kg from the recommended rate and 0.6, 0.88, 1.2 and 2.8 mg/kg from 1.5 times that rate. The green forage 40 days before harvest contained residues in the range 0.03-35 mg/kg. However green and dry forages are restricted for use as animal feed.

The Meeting agreed to maintain the current recommendation of 0.1 mg/kg for beans (dry).

Residues in <u>dry peas</u> were determined in 8 trials in the UK. They were in the same range as in beans (<0.01-0.03 mg/kg). Since no information on GAP for peas was available, the Meeting could not estimate a maximum residue level.

In <u>soya beans</u> in US trials the residues in the immature succulent seeds were low and decreased quickly as the seed matured. In all samples taken at harvest residues were below the limit of determination (0.02 mg/kg) regardless of the method of treatment and including treatments at 2 and 2.3 times the recommended maximum rate.

Residues in the forage at harvest were below 0.1 mg/kg except in one sample which contained 0.56 mg/kg. The residues in green forage may exceed 5 mg/kg, but quickly decline below 0.1 mg/kg. Owing to their value as oil seed, no significant quantities of soya beans are likely to be used as green forage or cut for hay. In addition, the US label states that treated plants must not be used as animal feed.

The Meeting agreed to maintain the current recommendation of 0.02* mg/kg for soya beans

(dry).

<u>Carrots</u> were grown in three plots in the UK following soil treatments with 2.6-5.1 g ai/100 m. Residues declined rapidly in the root and were in the range 0.17-0.29 mg/kg at 84 days after planting.

The Meeting considered the limited data insufficient to estimate a maximum residue level for carrots.

<u>Swedes</u> were grown in soils treated with aldicarb at 1.3-2 times the recommended rate. In one trial the residue was 0.04 mg/kg in the whole plant and 0.01 mg/kg in the root at 129 days, and in four other trials below the limit of determination in all root samples 191-222 days after treatment. Since the samples were taken at much longer intervals than the current 70 day PHI, the residue data could not be used for estimating a maximum residue level.

Supervised trials were carried out on sweet_potato in 5 States of the USA between 1968 and 1975. Application rates included the recommended US rates (1.7-3.36 kg ai/ha and ranged up to 6.72 kg ai/ha. At the registered PHI and dosage the roots contained residues from <0.01 to 0.05 mg/kg. Residues of 0.09 and 0.16 mg/kg were detected after applications with 1.5- and twofold rates. The distribution of the results indicated that residues higher than 0.05 mg/kg residues might also occur when the compound used according to GAP.

The vines of sweet potato following treatment with the maximum recommended rate contained residues in the range 0.21-0.89 mg/kg after 90-105 days, and 0.34 mg/kg after 119 days. When a double rate was applied the residues ranged from 1.6 to 5.1 mg/kg after 90-105 days and 0.74 mg/kg was measured after 119 days.

The Meeting agreed to maintain the current recommendation of 0.1 mg/kg for sweet potato.

Results were considered from field trials on <u>sugar beet</u> in seven European countries and in eight States of the USA. Following application at sowing (GAP) in European trials, the residues in the roots were at or below 0.04 mg/kg in samples from 76 trials. Broadcast applications over the plants (up to five leaves) did not result in higher residues. In four trials in the UK in 1967-68 higher residues were detected in two samples: 0.05 mg/kg (4.5 kg ai/ha, top-dressing application, 182 days PHI) and 0.06 mg/kg (1.12 kg ai/ha in furrow, 159 days). Single applications up to 4.5 kg ai/ha and with a 90-day or longer PHI, corresponding to the current recommended uses in the USA, resulted in residues in the roots of \leq 0.02 mg/kg in 23 trials. Application at a double rate (9 kg ai/ha gave residues of \leq 0.03 mg/kg at harvest (\geq 140 days after application).

The residues in leaves from treatments corresponding to US GAP ranged from <0.1 to 0.93 mg/kg at 120 days after application or later. After treatments at a double rate the residues in the leaves amounted to 0.98 mg/kg at 160 days. The residues in tops and leaves ranged from 0.03 to 0.65 mg/kg in all of the 79 samples from European trials. The residues in the leaves consisted of about 78% aldicarb sulphone and 22% aldicarb sulphoxide. The parent aldicarb could not be detected.

The Meeting agreed to maintain the current recommendations of 0.05* mg/kg for sugar beet root and 1 mg/kg for sugar beet leaves or tops.

In 12 trials in Australia and 2 in Israel on wheat, after application at planting with rates up to 3 kg ai/ha, residues were not present at detectable levels (<0.01 or <0.02 mg/kg) in the mature grain.

In grain from <u>barley</u> grown in soil treated with 1 kg ai/ha, the residues were <0.02 mg/kg in 1 Australian and 5 French trials.

Wheat straw from the Australian trials contained residues of <0.02-0.03 mg/kg. Barley straw samples were also analysed but no residue was detected (<0.04 mg/kg). Rotational crop studies conducted in the USA indicated, however, that wheat and barley planted in soils containing 0.17-0.24 mg/kg aldicarb residues may take up higher residues from the soil, resulting in 0.11-0.88 mg/kg residue levels in green forage.

The Meeting took into consideration the similar residue pattern in wheat and barley and estimated maximum residue levels of 0.02 mg/kg for wheat and barley grain, and 0.05 mg/kg for the straws.

After applications at rates ranging from 0.56 to 3.36 kg ai/ha at or shortly before planting sorghum, residues were undetectable (<0.01 mg/kg) in mature grain in 55 of 60 trials. Following applications with 1.12 and 2.24 kg ai/ha the residues were 0.03, 0.04 and 0.13 mg/kg and 0.04 and 0.1 mg/kg respectively. Residues were undetectable (<0.02 mg/kg) in sorghum straw in 16 cases. Detectable residues were present in 5 samples at the following concentrations from the application rates (kg ai/ha) and PHIs shown in parentheses: 0.03 mg/kg (1.34, 124 days), 0.03 mg/kg (1.12, 158), 0.09 mg/kg (0.56, 132), 0.11 mg/kg (1.68, 158) 0.4 mg/kg (1.12, 132). The residues in straw and dry fodder would be unlikely to exceed 0.5 mg/kg when the compound is used according to GAP.

The Meeting concluded that about 98% of the residues from approved uses would be below 0.1 mg/kg, and recommended lowering the present MRL of 0.2 mg/kg MRL for sorghum grain to 0.1 mg/kg. The previous recommendation of 0.5 mg/kg for sorghum straw and fodder is maintained.

Supervised field trials on <u>maize</u> were reported from France, Germany, Israel, the UK and the USA. Treatments up to 1 kg ai/ha did not give rise to detectable residues in the grain. Applications with 1.7 kg ai/ha resulted in detectable (\geq 0.03 mg/kg) residues in three of 25 trials. The residues in the other samples were below the limit of determination.

The residues in green forage harvested from 60 to 116 days after treatment ranged from <0.02 to 0.34 mg/kg. The dry fodder (stover) contained residues in the range <0.02-0.54 mg/kg.

The Meeting agreed to maintain the current recommendation of 0.05 mg/kg for maize, and recommended new limits of 0.5 mg/kg for maize fodder and maize forage, the latter to replace the previous recommendation of 5 mg/kg.

Supervised field trials on <u>sugar cane</u> were submitted from Australia, India, Indonesia and South Africa. The compound may be used either at or shortly after planting, or after the first harvest. Consequently the minimum time which would elapse between application and harvest is 8 months, but usually over a year. The residues in the leaves and stalks of sugar cane from 14 trials at rates of 0.75-7.75 kg ai/ha were below the limit of determination (<0.001-0.003 mg/kg) 315-362 days after application. In South Africa eleven trials with rates from 2.24 to 3.36 kg ai/ha resulted in residues from <0.02 to 0.33 mg/kg at 170-192 days after application and <0.02 to 0.11 mg/kg after about a year. In these last trials the blank values were up to 0.06 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg for sugar cane, based on a PHI of about a year.

68 trials on <u>pecans</u> have been carried out in Israel, South Africa and the USA with recommended rates. Samples taken at intervals of 48 to 268 days after application generally contained residues below 0.2 mg/kg, with a mean of 0.11 mg/kg. The residues were between 0.2 and 0.3 mg/kg in three samples, and between 0.3 and 0.4 mg/kg in two others. Two samples contained residues of 0.77 and 0.75 mg/kg 59 and 96-98 days after treatment.

Residues in pecan shells ranged from 0.02 mg/kg to 0.61 mg/kg.

The Meeting recommended an increase in the current limit for pecan from $0.5~\mathrm{mg/kg}$ to $1~\mathrm{mg/kg}$.

Numerous field trials on <u>cotton</u> were reported from Australia, Israel and the USA. Applications made in accordance with recommended uses gave no detectable residues in about 75% of the samples. Over 90% of the samples contained <0.07 mg/kg, and a few 0.08 mg/kg. Even when more than one side-dressing application was made with higher rates the residues did not exceed 0.08 mg/kg.

The Meeting agreed to maintain the current recommendation of 0.1 mg/kg for cotton seed.

A large number of trials on <u>peanuts</u> in the USA resulted in residues at or below 0.08 mg/kg in whole peanuts treated according to GAP (the LOD was 0.01 mg/kg). Processing studies in the USA and trials from Senegal showed that the residues in peanut kernels were 5 to 10 times lower than in the whole nuts, so residues may occur up to 0.02 mg/kg in peanut kernels when GAP is followed.

The residues in green peanut vines were 0.65-21.5, 0.48-21.8, 0.15-3.0 and <0.02-2.7 mg/kg about 55, 75, 95 and 120 days after application. The hay contained residues in the range <0.05-2.6 mg/kg at harvest following applications at about the recommended rates.

The Meeting agreed to replace the previous recommendation for peanut kernel (0.05* mg/kg) by 0.02 mg/kg.

Seven trials on sunflower were reported from France. Aldicarb was applied at the maximum recommended rate of 0.5~kg ai/ha at planting. The seeds did not contain detectable residues (aldicarb sulphone <0.05~mg/kg).

The Meeting estimated a maximum residue level of 0.05* mg/kg for sunflower seed.

The residues in dried and hulled green <u>coffee beans</u> from supervised trials in accordance with use patterns in South America were low. Four of 47 samples contained residues above the LOD (0.02 mg/kg) 15 to 274 days after the last application: three were 0.03 mg/kg 118 to 143 days after the last application and the fourth was 0.05 mg/kg at day 186. Application of about double the recommended rate resulted in <0.02 (3), 0.04 (2) and 0.08 mg/kg.

The Meeting agreed to maintain the current recommendation of 0.1 mg/kg for coffee beans.

A feeding study was conducted to determine the residue levels in $\underline{\text{milk}}$ and $\underline{\text{liver}}$ after feeding a 1:1 mixture of unlabelled aldicarb sulphoxide and aldicarb sulphone at 1.0, 3.0 or 5.0 ppm in the diet to lactating cows.

The total toxic aldicarb residues in the milk were about 0.1% of the level of the metabolites in the feed, which is in agreement with the value of about 0.1-0.2% obtained for the total radioactive metabolites in milk from feeding radiolabelled aldicarb and its sulphone. No build-up of toxic aldicarb residues was found during the 46 days of the continuous feeding study.

Aldicarb residues were not detected in liver (<0.01 mg/kg) at any of the dose levels tested. Since the samples were stored frozen for an unreported period, this may have been caused by the decomposition of residues during storage.

In view of the very low concentration or absence of the carbamate metabolites in the meat of

cattle and goats (<0.1% of the feeding level) and eggs (<0.05%), and the maximum estimated residues in potential animal feeds, it is unlikely that detectable residues would ever occur in meat, milk or eggs if the current use patterns are followed, even if some treated plant forages were used as animal feed.

Aldicarb sulphoxide and aldicarb sulphone were shown to be stable under <u>deep-frozen storage</u> for at least nine months in oranges, six months in milk, six weeks in potato processing products (chips, flakes, granules, wet and dry peel) and in soya bean processing products except soapstock. However, in frozen beef liver 85% of the aldicarb sulphoxide and >99% of the aldicarb sulphone were lost within one day.

The <u>effect of processing</u> on residue levels in processed products was studied in citrus fruit, grapes, dry beans and peas, soya beans, sugar beet, sorghum, maize and peanuts.

The commercial washing of citrus fruit did not remove aldicarb residues. The peel generally contained about 3 to 5 times the residue in the pulp, with extremes of 2-9 times. The peel/pulp ratio ranged from 31/69 to 49/51.

The juice contained lower residues than the fresh fruit. The residue levels in juice as a percentage of those in the whole fruit were 34-89% for oranges, <10-18% for grapefruit and 27-57% for limes.

Residues were concentrated when fresh fruit was processed to dried citrus pulp (dried peel) by a factor of about 1.8. Residues in the molasses were at about the same level as in the dried pulp.

Eight grape varieties, containing residues in the range 0.1-2.5 mg/kg, were processed to fresh juice, pomace and wine. The concentration factors (residue in processed product/residue in fresh fruit) ranged between 0.53 and 0.87 for fresh juice, 0.73 and 1.4 for pomace, and 0.19 and 0.63 for wine.

Raisins were prepared from Thompson Seedless grapes grown in California in 1981. Concentration factors ranged from 0.6 to 2.4.

Studies to determine the effects of preparative procedures and cooking as usually done in the home showed a significant reduction of the residues in dry beans and peas. About 85% and over 90% of the residues in the seeds were lost after one hour and three hours cooking respectively. Fully cooked blackeye peas, red kidney beans and field peas contained less than 10% of the original residue in the dry seeds.

There was no concentration of residues on processing soya beans (after treatment at 4 times the recommended rate) to oil. Residues were undetectable in the refined oil (<0.005-0.01 mg/kg). The residues in the hulls and meal were about the same as, or somewhat higher than, in the seed.

Diffusion juice obtained from $\underline{\text{sugar beet}}$ in a pilot unit simulating commercial processing contained residues which were about the same as or somewhat lower than in the corresponding root samples. Other processed fractions (thin juice, dry pulp, wet pulp) contained no detectable residue (<0.005 mg/kg).

When <u>sorghum</u> grain was milled, residues in the flour were about 1/2-1/3, in the shorts about 1/2, and in the bran up to 4 times those in the grain.

Processing field-treated <u>maize</u> to meal and oil caused no concentration of residues. The hulls contained about 2.5 times the residue in the grain. Since the hulls are generally fed to animals only in a mixture with other grain products, their feeding would be unlikely to result in a higher residue intake than feeding grain.

Crude oil processed from field-treated <u>cotton seed</u> contained about 1/10 to 1/15 of the residue in the seed. No residues containing the carbamate moiety were present in refined oil. The residues in the meal were about 1/2-1/3, and those in the hulls up to about 3 times, those in the seed.

Four lots of field-treated <u>peanuts</u> were processed to oil and meal. The residues in the kernels were about 1/5 to 1/10 of those in the whole nuts. The meal contained residues at about the same level as the kernels. Residues were not detectable in pressed or extracted oil (<0.003 mg/kg). The residues in the hulls were about 1 to 3 times those in the whole nuts. Since the average kernel/hull ratio is about 70/30 it may be concluded that most of the residue is generally in the hull.

Green <u>coffee</u> containing aldicarb residues of 0.11 mg/kg was processed to yield roasted coffee, spent grounds, and instant coffee. No aldicarb residues (<0.02 mg/kg) were found in any of these products.

The <u>residues in commodities moving in commerce</u> were studied in two extensive food survey programmes in the USA in order to assess more accurately the dietary exposure to aldicarb at the point of purchase. Seventy-five locations were selected nation-wide. Samples of bananas, oranges, potatoes, sweet potatoes and grapefruit and their processed products were taken in February, May and September during 1987 to cover seasonal variations. At each sampling 69-75 samples of each commodity were analysed. Detectable residues, including those below the limit of determination (LOD), were found in about 15% of the samples of bananas (0.019, 0.175 mg/mg), bananas processed for infant food (0.012, 0.01 mg/kg), orange juice for infants (0.01 mg/kg), and fresh grapefruit. The numbers in parentheses are the mean residue calculated with 0.01 mg/kg assigned to all samples containing <0.01 mg/kg.

The second monitoring programme was conducted in Florida in 1993 to determine the residues in oranges. The design of the survey involved collection of samples of oranges from groves that (1) had been treated with aldicarb, (2) had mature fruit on the tree at harvest, and (3) were intended for the fresh market. Altogether 869 <u>individual</u> oranges were analysed, of which 711 had either less than the LOD (467 oranges) or no detectable residues (244). The remaining 158 samples had quantifiable residues with a mean of 0.025 mg/kg and a standard deviation of 0.020 mg/kg.

From the results of these surveys it is apparent that the potential dietary exposure to aldicarb is significantly overestimated by using either tolerance-based or 95th percentile residue calculations.

RECOMMENDATIONS

On the basis of the data on residues resulting from supervised trials the Meeting concluded that the residue levels listed below (next page) are suitable for use as MRLs.

Definition of the residue: sum of aldicarb, its sulphoxide and its sulphone, expressed as aldicarb.

Commodity		Recommended MRL, mg/kg		PHI on which based, days
CCN	Name	New	Previous	
FI 0327	Banana	W	0.5	
GC 0640	Barley	0.02		>100

Commodity		Recommended MRL, mg/kg		PHI on which based, days
CCN	Name	New	Previous	
AS 0640	Barley straw and fodder, dry	0.05		>100
VD 0071	Beans (dry)	0.1	0.1	>70
VB 0402	Brussels sprouts	0.11	0.1	
FC 0001	Citrus fruits	0.2	0.2	
SB 0716	Coffee beans	0.1	0.1	>70
SO 0691	Cotton seed	0.1	0.1	>80
OR 0691	Cotton seed oil, edible	0.01*		
FB 0269	Grapes	0.2		120
GC 0645	Maize	0.05	0.05	>120
AF 0645	Maize forage	0.5	5	
AS 0645	Maize fodder	0.5		
MM 0095	Meat	0.01*	0.01*	
ML 0106	Milks	0.01*	0.01*	
VA 0385	Onion, Bulb	0.1	0.05*	>150
SO 0697	Peanut	0.02	0.05*	90
OR 0697	Peanut oil, edible	0.01*		
TN 0672	Pecan	1	0.5	>48
VR 0589	Potato	0.5 T	0.5	
GC 0651	Sorghum	0.1	0.2	90
AS 0651	Sorghum straw and fodder, dry	0.5	0.5	120
VD 0541	Soya bean (dry)	0.02*	0.02*	90
VR 0596	Sugar beet	0.05*	0.05*	90
AV 0596	Sugar beet leaves or tops	1	1	120
GS 0659	Sugar cane	0.1		360
SO 0702	Sunflower	0.05*		>90
VR 0508	Sweet potato	0.1	0.1	120
GC 0654	Wheat	0.02		>100
AS 0654	Wheat straw and fodder, dry	0.05		>100

 $^{^{1}}$ Residue data were not considered by the present Meeting. The recommendation of the 1993 JMPR is maintained. 2 Residue data were not considered by the present Meeting. The recommendation of the 1990 JMPR is maintained.

FURTHER WORK OR INFORMATION

Required (by 1996)

Submission of all available data on residues in potatoes from trials reflecting current use patterns.

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