DIQUAT (031)

EXPLANATION

Diquat, evaluated at the Joint Meetings in 1970, 1972, 1976, 1977 and 1978 is included in the CCPR periodic review programme. Maximum residue levels were estimated for a number of commodities, on the basis of commodities submitted to these Meetings.

Additional updated information on GAP and the results of supervised trials were presented by the manufacturers and several European countries to allow a comprehensive review of all the MRLs previously recommended, to accommodate the use of diquat for the pre-harvest desiccation of food crops. Residue data on additional crops have also become available.

IDENTITY

ISO common name: diquat

Chemical names

(IUPAC): 9,10-dihydro-8a,10a-diazoniaphenanthrene ion

(C.A.): 6,7-dihydrodipyrido[1,2-*a*:2′,1′-*c*]pyrazinediium
Present as the dibromide salt.

CAS Registry No: 2764-72-9 (ion)
85-00-7 (dibromide)

CIPAC No: 0055

Synonyms: Reglone (R)

Structural formula:

Molecular formula: $C_{12}H_{12}N_2$; dibromide $C_{12}H_{12}Br_2N_2$

Molecular weight: 184.2 (ion); 344.0 (dibromide)

Physical and chemical properties

Pure active ingredient (diquat dibromide)

Vapour pressure :	not measurable; < 10^{-8} kPa (25°C)	
Melting point :	325°C (dec)	
Octanol/water partition coefficient:	log P_{ow} -4.6 (20°C)	
Solubility: water	718 g/l	
methanol	25 g/l	
acetone, dichloromethane, toluene, ethyl acetate	<0.1 g/l	
Specific gravity:	1.61 g/cm^3 (25°C)	
Hydrolysis:	pH 5 stable	
	pH 7 stable	
	pH 9 slight hydrolysis	
Photolysis: low sensitivity to UV and sunlight		

Technical material (aqueous solution)

Purity:	268 g diquat ion/l (on average)
Specific gravity:	1.26 g/l
Stability: stable	

Formulations

Soluble concentrate (SL)

USE PATTERN

Diquat is a non-selective contact herbicide and crop desiccant. It is not readily translocated and is rendered biologically inactive by adsorption onto organic matter and clay minerals in soil. It is thus not mobile in soil or available for root uptake.

On a global basis, one third of the diquat sold is used as a total weedkiller. Herbicidal use patterns include weed control either pre-planting, pre-crop emergence or even early post-crop emergence, and weed control by directed or inter-row spray between the rows of established arable and tree crops. The regions of West Europe, Australia and Japan consume 90% of the diquat used for herbicidal purposes. Whilst many registrations exist for the use of diquat alone as a herbicide and are recorded in the label information displayed in Table 1-C, in commercial practice the product is seldom used without paraquat, either in a tank-mix or as a pre-formulated mixture. The commercial product most widely used is 'Reglone', an aqueous formulation containing 20% (w/v) of diquat cation.

When diquat is used as a herbicide, contamination may occasionally arise when spray is misdirected, or when young seedlings emerge through dense swards of sprayed herbage containing

diquat residues. In such cases severe contamination will kill or severely scorch the plants, and small residues (below 0.5 mg/kg) have been detected in the foliage of some crops (e.g. oats and maize) 7-8 weeks after application and in the roots of some root crops, e.g. carrots, where up to 0.07 mg/kg diquat residues were detected 14 days after application (Ref. 141). Such residues most likely arise from soil contamination. However, the great majority of crops treated in this way show no detectable residues (<0.05 mg/kg) in edible parts when harvested from 1-4 months later (FAO/WHO, 1973; Edwards, 1977; Kennedy, 1986d).

Pre-harvest desiccation of a wide range of seed and fodder crops accounts for the use of most of the diquat sold (two-thirds of the global volume). The regions of N. America and Europe (West, Central, East Europe and CIS) use 90% of the product going into the crop desiccation sector. A substantial proportion of this material is used for the desiccation of potato haulms, oilseed rape, sunflower, linseed, legume and pulse crops.

When diquat is used as a desiccant, the interval between application and harvest usually varies between 3 and 21 days. As the product is sprayed directly onto the crop, significant residues are present in the crop at harvest. The registered desiccation uses in internationally traded food or fodder crops and commodities are given in Table 1-A. The residues resulting from these use patterns are shown in Tables 2-13.

Registrations for the desiccation of crops for seed purposes only are given in Table 1-B. Since the seed is not intended for human or animal consumption, residue values for these commodities are not tabulated.

Diquat may also be used as an aquatic herbicide for the control of free-floating and submerged aquatic weeds in ponds, lakes and irrigation ditches. In this situation diquat-treated water may be used to irrigate established crops, either by overhead irrigation or via irrigation channels.

In the former instance, small residues have been found in the crops when residues in the overhead irrigation water had not declined to sufficiently low concentrations (Calderbank, 1972). When crops are irrigated overhead at a nominal water concentration of 0.01 mg/l diquat, no residues are found even within one day of treatment (Fujie, 1989a,b).

In the second situation, diquat-treated water (0.01 mg/l) from irrigation ditches was used to flood rice fields four times during the growing season. No diquat residues (<0.01 mg/kg) were found in the rice grain or straw at harvest (Fujie, 1988g).

Table 1A. Registered uses of diquat for desiccation of food and fodder crops.

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
Alfalfa	Argentina	1	0.3-0.5	200-400	3-7	
	Bulgaria	1	0.6		7-10	
	CIS	1	0.4-0.8			
	France	1	0.4-0.6			
	Germany	1	0.3			
	Israel	1	0.4-0.6		3-4	

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Japan	1	0.6-1.0			+ WC
	Mexico	1	0.4	400-600	3	hay, G
	Romania	1	0.6-1.0			
	Saudi Arabia	1	0.8	200-300		G
	United Arab Em.	1	0.4-0.6	200-300		G
	USA	1	0.3	50-100		G
	Yugoslavia	1	1.0-1.2			
Barley	Cuba	1	0.6-0.8		4-7	fodder
	Korea, North	1	0.7	1400		+ WC, G
Barley (L)**	New Zealand	1	0.6-0.8	+ WC		
	UK	1	0.4-0.8	200-500	4	+ WC, G
Beans	Brazil	1	0.3-0.6	200-300/30-40	1	G/A
	Poland	1	0.6-1.2	400-600		G
Bean, dwarf	Netherlands	1	0.4-0.6	200-400		G
Bean, adzuki	Canada	1	0.4	225-550		G
Bean, field	Australia	1	0.4-0.6			
	Bulgaria	1	0.6			
	Czech Republic	1	0.5-0.8	10		grain
	Germany	1	0.6	1000	5	G
	Guatemala	1	0.4-0.6			
	Netherlands	1	0.4-0.6	200-400		G
	New Zealand	1	0.6-0.8	5-8		
	Poland	1	0.5-0.6	400-600		G
Bean, field	UK	1	0.6	200-500	4-7	G
Bean, Haricot	France	1	0.6-0.8			G
Beetroot	Poland	1	0.8-1.2	400-600		G
Beet, Sugar	Sweden	1	0.4-0.8			
Cereals	Australia	1	0.2-0.6			+ WC
	Chile	1	0.5-0.7		10-15	+ WC
	Saudi Arabia	1	0.5	200-300	5	+ WC
Cereals (L)	Austria	1	0.4-0.6	1000	7	+ WC, G
	Belgium	1	0.4-0.8	200-400	7	+ WC, G
	Czech Republic	1	0.2-0.3		7	

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	France	1	0.4-0.6		7	
	Netherlands	1	0.6-0.8	200-400	4	
	Argentina	1	0.3-0.5	>20/200-400		A/G
Clover	Argentina	1	0.3-0.5	200-400	3-7	G
	Czech Republic	1	0.3		6	hay
	United Arab Em.	1	0.4-0.6	200-300		G
Cotton	Australia	1	0.4-0.6			
	Spain	1	0.4-0.6	300-1000		G
Grass	Israel	1	0.4-0.6			
	CIS	1	2.0-3.0			hay
Grass (Dog's tooth)	United Arab Em.	1	0.4-0.6	200-300		G
Legumes	Argentina	1	0.3-0.6	200-400	3-7	G
	Chile	1	0.4-0.6		3-7	
	Czech Republic	1	0.6-0.8		6	
Lentil	Canada	1	0.3-0.4	225-550	4-7	G
	Canada	1	0.3-0.55	>45		A
	Argentina	1	0.3-0.5	>20/200-400		pulses, A/G
Linseed	Argentina	1	0.3-0.5	>20/200-400		A/G
	Australia	1	0.4-0.6			
	Canada	1	0.3-0.4	225-550		G
	Canada	1	0.4-0.55	>45		A
	Cuba	1	0.4-0.6		4-7	
	Czech Republic	1	0.5-0.8		4	
	France	1	0.4-0.6			
	Italy	1	0.2-0.7	300-800	30	G
	Sweden	1	0.4-0.8			
Maize	Argentina	1	0.4-0.5	>20		A
	Chile	1	0.3-0.5		7-10	
	Cuba	1	0.6-0.8		4-7	fodder
	Guatemala	1	0.8			
	Spain	1	0.3-0.8	1500		harvest aid, G
	Spain	1	0.3-0.8	100		harvest aid, A
	United Arab Em.	1	0.4-0.6	200-300		feed corn, G

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
Oats (L)	New Zealand	1	0.6-0.8			
	UK	1	0.4-0.8	200-500	4	+ WC, RC, G
Pea, field or fodder	Australia	1	0.4-0.6			
	Belgium	1	0.6-1.0	200-400	4-7	G
	Bulgaria	1	0.6			
	Canada	1	0.3-0.4	225-550		G
	Canada	1	0.3-0.55	225-550		A
	Czech Republic	1	0.5-0.8		6	
	France	1	0.4-0.6			
	Germany	1	0.6	1000	5	G
	Netherlands	1	0.4-0.6	200-400		G
	New Zealand	1	0.6-0.8		5-8	
	Poland	1	0.5-0.6	400-600		G
	UK	1	0.4-0.6	200-500	7-10	G
Potato	Australia	1	0.6-0.8		7	
	Austria	1	0.8	1000	10	ware
	Belgium	1	0.6-1.0	400-600	4	haulm, G
	Brazil	1	0.3-0.5	200-300/30-40	7	G/A
	Bulgaria	1	0.4-0.6			
	Canada	1-2	0.3-0.85	550-1100	14	G
	Chile	1	0.4-0.6	400	3-7	G
	CIS	1	0.4			
	Cuba	1	1.0			seed
	Cuba	1	0.6-0.8			ware
	Denmark	1	0.6-1.0			
	France	1	1.0			ware
	Germany	1	0.5		10	ware
	Greece	1	1.0	500		stem, G
	Guatemala	1	0.6-0.8			
	Israel	1	0.6-1.0			
	Italy	1	0.8-1.3	300-800	30	G
	Japan	2	0.4-0.6		7	
	Korea, North	1	1.0	1000		G

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Mexico	1	0.9-1.3	400-600		G
	Morocco	1	0.6-0.8	200-500		ware, G
	Netherlands	2	0.4-1.0	500-600	14-18	ware/ind., G
	New Zealand	2	0.3-0.8			
	Norway	1	0.5			
	Poland	1	0.8-1.0	400-600	10-14	G
	Portugal	1	0.6-0.8	600-800	4-7	ware, G
	Romania	1	0.6-0.8		12-15	ware
	Spain	1	0.3-0.8	300-1000		ware, G
	Sweden	1	0.4			
	Switzerland	1	0.7-1.0	1000		G
	United Arab Em.	1	0.6-0.8	200-300		ware, G
	UK	2	0.4-0.8	200-500		G
	USA	2	0.3	75-375/20-40	7	G/A
	Yugoslavia	1	0.4-1.0		10	
Pulses	Netherlands	1	0.4-0.6	200-400		
Rape	Argentina	1	0.3-0.5	>20		A
	Australia	1	0.4-0.6		4	
	Belgium	1	0.6-1.0	200-400	4-7	G
	Canada	1	0.3-0.4	225-550	14	G
	Chile	1	0.3-0.5	300	7-10	G
	Cuba	1	0.4-0.6		4-7	
	Germany	1	0.4-0.6		5	
	Netherlands	1	0.6		2-6	
	Norway	1	0.4-0.6			
	Poland	1	0.4-0.6	400-600		G
	Sweden	1	0.4-0.8			
	UK	1	0.6	250-500		G
	Yugoslavia	1	0.5-0.7	60-100	5	A
Rape, Summer	Austria	1	0.6	1000	5	+ WC, G
	Denmark	1	0.6			
	Netherlands	1	0.4	200-400		
Rape, Winter	Austria	1	0.4	1000	5	+ WC, G

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Denmark	1	0.6			
Rice	Argentina	1	0.3-0.5	>20	5-7	A
	Australia	1	0.4-0.6		5	
	Brazil	1	0.3-0.6	30-40	7	A
	Bulgaria	1	0.6			
	Cuba	1	0.4-0.5		3-6	
	Greece	1	0.6-0.7	500	2-6	G
	Guatemala	1	0.4-0.5			
	Italy	1	0.2-0.6	300-800	30	G
	Korea, North	1	0.7	1400		G
	Mexico	1	0.3-0.4	60-100		A
	Morocco	1	0.3-0.6	25-76	3-6	A
	Portugal	1	0.3-0.4	100-200/40-80	4-7	G/A
	Romania	1	0.3-0.4		7-14	
Sorghum	Argentina	1	0.3-0.5	200-400/>20	10	G/A
	Australia	1	0.4-0.6			
	CIS	1	0.8			
	Cuba	1	0.4		5-10	
	Guatemala	10.4				
	Mexico	1	0.3-0.4	400-600	14	G
	USA	1	0.3			grain
Soya beans	Argentina	1	0.3-0.5	>20		A
	Australia	1	0.4-0.6		4	
	Brazil	1	0.2-0.4	200-300/30-40	10	G/A
	Bulgaria	1	0.6		7-14	
	Canada	1	0.4-0.5	>45		A
	Canada	1	0.3-0.4	225-550		G
	Cuba	1	0.4-0.6		4-7	
	Czech Republic	1	0.6		6	
	France	1	0.4-0.6			
	Guatemala	1	0.6-0.8			
	Morocco	1	0.4-0.6	200-500		G
	New Zealand	1	0.6-0.8		5-8	

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Romania	1	0.3-0.6		5-7	
	USA	1	0.3-0.56			
	Yugoslavia	1	0.5-0.7	60-100	5	A
Sugar cane	Australia	1	0.4-0.6		4	
	Colombia	1	0.4-0.6			
Sunflower	Argentina	1	0.3-0.5	>20		A
	Australia	1	0.4-0.6		4	
	Bulgaria	1	0.5-0.6		10	
	Canada	1	0.3-0.4	225-550	15-20	A
	Canada	1	0.3-0.55	>45	15-20	A
	Chile	1	0.5-0.7	400	10-15	G
	CIS	1	0.4-0.6			
	Cuba	1	0.4-0.6		5-10	
	Czech Republic	1	0.4-0.6		6	
	France	1	0.4-0.6			
	Hungary	1	0.5	80	7	A
	Israel	1	0.6		21	
	Morocco	1	0.4-0.6	25-76		A
	Poland	1	0.6-0.8	400-600		G
	Romania	1	0.3-0.6		10-14	
	South Africa	1	0.3	30		A
	Spain	1	0.4-0.6	300-1000		G
	Turkey	1		200-500	14	A
	Yugoslavia	1	0.5-0.7	60-100	5	A
Wheat	Argentina	1	0.3-0.5	>400	7-14	G
	Cuba	1	0.6-0.8		4-7	fodder
Wheat (L)	New Zealand	1	0.6-0.8			

* A = aerial application
G = ground application
RC = regrowth control
WC = + weed control
** L = lodged

Table 1B. Registered uses of diquat for desiccation of crops for seed purposes.

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
Alfalfa	Brazil	1	0.6-0.8	200-300/30-40		G/A
	Canada	1	0.4-0.65	225-550	7	G
	CIS	1	0.4-0.8			G
	Cuba	1	0.6		2-4	
	Czech Republic	1	0.6-0.8		3-5	
	Germany	1	0.3			
	Greece	1	0.6-0.7	500	2-6	G
	Italy	1	0.2-0.6	300-800	30	G
	Mexico	1	0.5-0.9	400-600	3	G
	Morocco	1	0.6-0.8	200-500		G
	Netherlands	1	0.4-0.8	200-400		
	New Zealand	1	0.6			
Bean	CIS	1	0.8-1.0			
	Czech Republic	1	0.6		6	
	Cuba	1	0.4-0.6		4-7	field
	Czech Republic	1	0.4		6	green
Beets	Netherlands	1	1.0	800	5	
	Czech Republic	1	0.8-1.0		4-7	fodder, sugar
	CIS	1	0.8-1.2			fodder, table
	CIS	1	1.0-2.0			sugar
	Czech Republic	1	0.8-1.0		4-7	sugar
	Greece	1	0.6-0.7	500	2-6	sugar, G
	Israel	1	0.4-0.6		2-4	sugar
	Morocco	1	0.8-2.0	200-500		sugar, G
	Poland	1	1.2-1.6	400-600		sugar, G
	Poland	1	1.2-1.6	400-600		fodder, G
Cabbage	CIS	1	0.4-0.8			
Canary	Netherlands	1	0.4-0.8	200-400		
Carrot	CIS	1	0.5-0.6			
	Czech Republic	1	0.8		7	
	Norway	1	0.5			
	Poland	1	0.6-1.0	400-600		G
Clover	Bulgaria	1	0.8-1.0			

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	CIS	1	0.4-0.8			
	Cuba	1	0.6		2-4	
	Czech Republic	1	0.5-0.7		3-5	
	Denmark	1	0.5			
	Germany	1	0.3			
	Morocco	1	0.6-0.8	200-500		G
	New Zealand	1	0.6-0.8			
	Netherlands	1	0.4-0.6	200-400		
	Norway	1	0.4-0.6			
	UK	1	0.4-0.6	200-500		G
	USA	1	0.3	20-40		A
(Egyptian)	United Arab Em.	1	0.4-0.6	200-300		G
Cotton	Australia	1	0.4-0.6			
	Spain	1	0.4-0.6	300-1000		G
Crop	Poland	1	0.6-0.8	400-600		field, G
Flower	Netherlands	1	0.4-0.8	200-400		
Grass	Brazil	1	0.6-0.8	200-300/30-40		G/A
Legumes	Australia	1	0.3-0.6			
	Canada	1	0.4-0.65	225-550	7	
Linseed	UK	1	0.6	300-500		G
Lupin	Australia	1	0.4-0.6			
	Denmark	1	0.6			
	Poland	1	0.6-1.2	400-600		G
Maize	Italy	1	0.2-0.7	300-800	30	G
Mustard	Canada	1	0.3-0.4	225-550		G
	Canada	1	0.4-0.55	>45	14	A
	Cuba	1	0.4-0.6		4-7	
	Netherlands	1	0.4-0.8	200-400		
Onion	Czech Republic	1	0.8		6-10	
	Norway	1	0.5			
Ornamental	Netherlands	1	0.4-0.8			
Parsley	Norway	1	0.5			
Peas	Cuba	1	0.4-0.6		4-7	field

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Norway	1	0.4-0.6			field
Poppy	Netherlands	1	0.4-0.8	200-400		
Potato	Algeria	1	0.6-0.8			
	Austria	1	1.0	1000	10	G
	Czech Republic	1	0.4-1.0		7-14	
	France	2	0.5			
	Germany	1	1.0		10	
	Morocco	1	1.0	200-500		G
	Netherlands	1	1.0	500		after flail, G
	Netherlands	2	1.0 + 0.6-1.0	500		G
	Norway	1	0.4-0.6			
	Portugal	1	1.0	600-800	4-7	G
	Romania	1	1.0		20-30	
	Spain	1	0.3-0.8	300-1000		G
	United Arab Em.	1	1.0	200-300		G
Pulses	Netherlands	1	0.4-0.6	200-400		
Radish	CIS	1	0.8-1.0			
	Czech Republic	1	0.8		6	
	Denmark	1	0.5			
	Poland	1	0.8-1.2	400-600		G
Rape	Netherlands	1	0.4 -0.6	200-400		
Sage	CIS	1	0.3			G
Sorghum	Brazil	1	0.6-0.8	200-300/30-40		G/A
Spinach	Denmark	1	0.5			
	Poland	1	0.7-0.8	400-600		G
Vegetables	Netherlands	1	0.4-0.8	200-400		
Wheat	Italy	1	0.2-0.6	300-800	30	G

* A = aerial application
G = ground application

Table 1C. Registered uses of diquat for weed control.

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
Apple	Canada	1	1.1			
Alfalfa	Argentina	1	0.04	300-400		Cuscuta, A
	Austria	1	0.6-0.8	1000		+ Desic., G
	Cuba	1	0.8			Cuscuta
	Czech Republic	1	0.1		6	Cuscuta
	Greece	1	0.04-0.05	500		Cuscuta, G
	Israel	1	0.4			Cuscuta
	Italy	1	0.3-0.7	300-800	30	G
	Morocco	1	1.0	200-500		Cuscuta, G
	Morocco	1	0.01	10		spot, G
	Spain	1	0.8	1000		G
	Sweden	1	0.2-1.0			
	Turkey	1	1.0	200-500		Cuscuta, G
	United Arab Em.	1	0.8	200-300		Cuscuta, G
Asparagus	Italy	1	0.4-0.82	300-800	30	G
Banana	Costa Rica	1	0.1			
	Guatemala	1	0.3-0.8			
	Spain	1	0.15-0.3			
	Nicaragua	1	0.2			
Barley	Japan	1	0.8			pre-
Beets	Spain	1	0.3-0.8	300-1000		pre-, G
Beet, red	Brazil	1	0.3-0.6	200-300/30-40	1	G/A
Beet, sugar	Czech Republic	1	0.2			
Bulbs	Netherlands		1 drop/plant	N/A		Abnormal plants
Cabbage	Mexico	1	0.3-0.4	400-600		G
Carrot	Netherlands	1	0.6-1.0	300	14, 113	
	Spain	1	0.3-0.8	300-1000		pre-, G
	Switzerland	1	0.6	1000		post-sowing pre-em G
Celery	Spain	1	0.3-0.8	300-1000		pre-, G
Cereals	Saudi Arabia	1	0.4-0.8	200-300		pre-
Citrus	Brazil	1	0.3-0.6	200-300	14	G

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Greece	2	0.04-0.08	500		G
	Italy	1	0.6-1.0	300-800	30	G
	Mexico	1	0.3	400-600		G
	Spain	1	0.3-0.8	300-1000		G
Clover	Brazil	1	0.6-0.8	200-300/30-40		Desic.,seed, G/A
	Canada	1	0.4-0.65	225-550	14	Desic.,seed, G
	Denmark	1	0.5			
	Sweden	1	0.2-1.0			
Coffee	Brazil	1	0.3-0.6	200-300	16	G
	Costa Rica	1	0.1			
	Guatemala	1	0.3-0.9			
	Nicaragua	1	0.2			
Crops, field	Canada	1	0.55-1.1	>300		pre-, G
	Cuba	1	0.3-0.8			pre-
	Cuba	1	0.3-0.8			Inter-row
	Israel	1	0.3-0.5			Inter-row
	Morocco	1	0.3-0.8	200-500		G
	Netherlands	1	0.6	200-1000		G
	Netherlands	1	0.6-1.0			Inter-row
	Netherlands	1	0.8	1000		Senescent crop destruction, G
	Spain	1	0.3-0.8	300-1000		Inter-row
	Tanzania	1	0.3-0.8			Inter-row
	United Arab Em.	1	0.3-0.8	200-300		pre-, G
	United Arab Em.	1	0.8	200-300		Inter-row, G
	UK	1	0.3-0.4	200-500		Inter-row, G
Crops, plantation	Tanzania	1	0.3-0.8			
Direct drill	Argentina	1	0.4-0.6	>200		G
Fruits	Netherlands	1	0.6	600-1000		G
Fruits, tree	Spain	1	0.3-0.8			
Grapes	Austria	1	0.6-1.0	1000		G
	France	1	0.2			Sucker control
	France	1	0.6-0.8			
	Greece	1	0.04-0.08	500		G

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	Italy	1	0.6-1.0	300-800	30	G
	South Africa	1	0.3-1.0	200-750		esp. Capeweed, G
	Spain	1	0.3-0.8	300-1000		G
	Yugoslavia	1	0.8-1.2			
Grass	Italy	1	0.6-1.3	300-800	30	Pasture renew.,G
Hops	Czech Republic	1	0.8			Shoot control
	Czech Republic	1	0.4			Weeds
	UK	1	0.5-0.75	200-500		+ hop stripping
Horticulture	Italy	1	0.4-0.8	300-800	30	G
	Sweden	1	0.2-1.0			
Leeks	Switzerland	1	0.6	1000		post-sowing pre-em, G
Maize	Italy	1	0.6-1.0	300-800	30	Inter-row, G
Medicinal plants	Poland	1	0.4-0.8	400-600		G
Nursery	France	1	0.6-0.8			
Oats	Canada	1	0.22-0.3	225-335		Corn spurry, G
Olives	Greece	2	0.04-0.08	500		G
	Italy	1	0.6-1.0	300-800	30	G
	Spain	1	0.45			
	Spain	1	0.3-0.8	300-1000		pre-, G
	Switzerland	1	0.6	1000		post-sowing, pre-em, G
Onions	Brazil	1	0.3-0.6	200-300	1	G
Orchards	Austria	1	1.0	1000		G
	Belgium	1	0.1	0.1		Sucker cont., G
	Canada	1	1.1	225-550		G
	Czech Republic	1	0.8-1.2			
	France	1	0.6-0.8			
	Israel	1	0.5-1.0			
	Italy	1	0.6-1.0	300-800	30	G
	Japan	1-5	0.6-1.0			
	Netherlands	1	1.0			
	Norway	1	0.005/100m^2			
	Poland	1	0.6-1.0	400-600		G
	Saudi Arabia	1	0.4-0.8	200-300		Inter-row

Crop	Country	Application			PHI, days	Remarks*
		No.	Rate per applic. (kg ai/ha)	Spray Volume (l water/ha)		
	South Africa	1	0.3-1.0	200-750		esp. Capeweed, G
	Spain	1	0.3-0.8	300-1000		G
	Sweden	1	0.2-1.0			
	Switzerland	1-2	0.8	1000		Weeds, G
	Yugoslavia	1	0.8-1.2			
Parsley	Spain	1	0.3-0.8	300-1000		pre-, G
Peach	Brazil	1	0.3-0.6	200-300	14	G
Pepper, sweet	Spain	1	0.3-0.8	300-1000		pre-, G
Poppy	Czech Republic	1	0.8-1.0			
	France	1	0.2			
	Sweden	1	0.2-1.0			
Potato	Denmark	1	0.6			pre/early post
	Sweden	1	0.4			pre/early post
	Switzerland	1	0.8	1000		pre/early post,G
Spent crops	Netherlands	1	0.8	1000		Glasshouse
Strawberry	Sweden	1	0.2-1.0			
Sugar cane	Argentina	1	0.3-0.5	>20		Flowering control, A
	Cuba	1	0.15-0.3			Flowering control
	Guatemala	1	0.15-0.3			Flowering control
	Mexico	1	0.15-0.3	80-100	90	Flowering control, A
	Morocco	1	0.15-0.3	25-76		Flowering control, A
Vegetables	Canada	1	0.55-1.1	>300		pre-, G
	Morocco	1	0.3-0.8	50-100		Inter-row, G
	Poland	1	0.4-0.8	400-600		pre-, G
	Poland	1	0.6-0.8	400-600		G
	Spain	1	0.3-0.8			
	Saudi Arabia	1	0.4-0.8	200-300		Inter-row
Vine	Spain	1	0.3-0.8			
Wheat	Japan	1	0.8			pre-

* A = aerial application
G = ground application

RESIDUES RESULTING FROM SUPERVISED TRIALS

Results of supervised trials using diquat for the pre-harvest desiccation of a variety of crops in different countries are presented in Tables 2-13. The countries involved are indicated in the tables by their ISO international code letters, as follows.

COUNTRY	CODE	COUNTRY	CODE
Argentina	AR	Hungary	HU
Australia	AU	Italy	IT
Brazil	BR	Israel	IL
Bulgaria	BG	Japan	JP
Canada	CA	Netherlands	NL
Chile	CL	New Zealand	NZ
Czechoslovakia	CS	Poland	PL
Denmark	DK	Spain	ES
Germany	DE	Sweden	SE
Finland	FI	UK	GB
France	FR	USA	US

FIgures in parentheses in Tables 2-13 refer to the numbers of individual results.

Earlier data presented for evaluation to the JMPR in the years from 1970 to 1977 are summarized at the top of each table, where available, and are given the appropriate FAO/WHO references. These data are largely contained in the following reports.

Calderbank (1968); Calderbank and Yuen (1963); Calderbank and McKenna (1964); McKenna (1966); Calderbank and Springett (1971); Edwards (1977); Ward (1978).

Beans (Table 2). Normal commercial rates of diquat used for the desiccation of field beans are 0.4-0.6, but also up to 0.8 or 1.0 kg ai/ha. Data evaluated earlier by the JMPR showed that diquat residues on whole beans treated with diquat as a desiccating agent were in the range <0.05-0.57 mg/kg (mean 0.10 mg/kg) (FAO/WHO, 1973).

Comparable results were obtained from trials in France in 1984 when diquat residues in the range <0.1-0.66 mg/kg were found on haricot beans (whole beans) harvested 3-11 days after treatment at the maximum rate (0.8 kg ai/ha) (Culoto, 1985). Similarly, from eight trials in Germany in 1984 and 1985 on fodder beans treated at 0.6 kg ai/ha, residues of diquat of <0.02-0.15 mg/kg were found 3-13 days after treatment (Kennedy, 1986f). Nine of the 21 residues were <0.02 mg/kg in the whole beans. In the case of measured residues there was no decrease observed within the period of 4 to 6 days.

Usually, residues in the bean seed are negligible when the bean is protected by the pod, as shown in trials in Germany in 1979 and 1980. Residues of diquat in the beans were generally <0.01 mg/kg, whilst the pods showed residues in the range 13-16 mg/kg (GDR, 1987).

Table 2. Residues of diquat in beans from supervised trials.

Type, Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Various	0.39-1.34	Whole bean	3-21	<0.05-0.20	38
	0.30-1.0		4-10	<0.05-0.57 (mean 0.10)	39
HARICOT FR/1984	0.8	Whole bean	0	0.56, 0.31	23
			3	0.51, 0.57	
			7	0.60, 0.46	
			10	0.43, 0.66	
	0.8	Whole bean	0	1.7, 6.0	23
			4	0.2, 0.24	
			7	<0.1, <0.1	
			11	<0.1, <0.1	
FIELD BEANS DE/1979	1.0	Bean seed	3	<0.1, <0.1	54
			5	<0.1, <0.1	
		Bean pods	3	13, 15	
			5	15, 16	
FIELD BEANS DE/1980	1.0	Bean seed	3-4	0.2, <0.1	
			5-6	0.2, <0.1	
		Bean pods	3-4	3, 11	
			5-6	5, 12	
FODDER BEANS DE/1984	0.6	Whole bean	4-13	<0.02 (8), 0.03	87
DE/1985	0.6	Whole bean	3-11	<0.02-0.15 (12)	87

Lentils (Table 3). Residue trials were carried out in Canada in 1982 and 1989 using rates ranging from 0.28 to 1.1 kg diquat/ha. The maximum recommended rate for desiccation of mature lentils in Canada is 0.55 kg ai/ha.

Seventeen trials were conducted in Saskatchewan in 1982, the product being applied by air and the lentils harvested 6-19 days later. Residues ranged from 0.03 (limit of determination) to 0.19 mg/kg. There is no apparent correlation between the residue found and the application rate (Oberhemmer, 1983).

In trials in 1989, "Reglone" was applied by ground spray at 0.4, 0.55, 0.8 and 1.1 kg/ha and also by air at 0.55 kg/ha. There were 17 separate trials conducted in Manitoba, Saskatchewan and Alberta. Five trials were excluded from analysis. In every case (18 results) the seed, harvested 3-7 days after application, contained no detectable residues of diquat (<0.05 mg/kg) (Dodsworth, 1990).

In three separate trials in Saskatchewan in 1989, "Reglone" was applied by air at the maximum label rate (0.55 kg diquat/ha) to mature lentils which were harvested within one day and seven days after application. Diquat residues at day 0 were 0.07-1.14 mg/kg and after seven days were in the range

0.07-028 mg/kg (Anderson, 1990).

Table 3. Residues of diquat in lentil seeds from supervised trials.

Country/Year	Rate, kg ai/ha	PHI, days	Residue, mg/kg	Ref.
CA/82	0.28 (air)	12	0.14-0.19 (3)	111
	0.42 (air)	8, 10	0.06-0.11 (8)	
	0.55 (air)	4, 6, 9	<0.03-0.05 (8)	
	0.56 (air)	6, 7, 8	0.09-0.14 (7)	
	0.56 (air)	10, 19	0.07-0.15 (6)	
	0.63 (air)	6, 14	0.06-0.13 (11)	
CA/89	0.4	3-7	<0.05 (3)	25
	0.55	3-7	<0.05 (3)	
	0.8	3-7	<0.05 (3)	
	1.10 (ground)	3-7	<0.05 (3)	
	0.55 (air)	3-7	<0.05 (6)	
CA/89	0.55 (air)	0	0.07, 0.36, 1.1	1
		7	0.07, 0.04, 0.28[1]	

[1] Diquat was not used according to GAP, being applied when a much greater proportion of seed pods were open than would be accepted practice (Ref. 145).

Peas (Table 4). Residue trials on peas have been evaluated by the JMPR in 1970 and 1972 (FAO/WHO, 1971, 1973). Trials evaluated in 1972 showed residues in the range <0.05-0.07, with a mean value of 0.05 mg/kg (FAO/WHO, 1973). The pod usually protects the peas from direct contact with the desiccating chemical so that residues of diquat in the peas themselves are usually undetectable (<0.05 mg/kg) or very low.

Trials were carried out during 1984 and 1985 in Germany in which diquat was applied to peas for fodder at the maximum commercial rate used in Northern Europe (0.6 kg ai/ha). In 1985 residues of diquat were 0.05-0.12 mg/kg 3-5 days after treatment and 0.04-0.15 mg/kg after 6-10 days. 12 results in total. In 1984 residues were similar, viz <0.02-0.10 mg/kg from ten results, 4-12 days after treatment at the same rate (0.6 kg ai/ha) (Kennedy, 1985).

Results of trials in Denmark in 1982 (Swaine, 1983b) and 1986 (Massey, 1987), Finland in 1979, 1980 (Heinanen, 1980) and 1985 (Jarvenen, 1985), France 1981 (Culoto and de Mallmann, 1982) and the UK in 1990 (Earl, 1991c), using the same rate of application (0.6 kg ai/ha), all gave similar results with residues in the peas in the range <0.02-0.10 mg/kg.

In the trials in France and the UK, the pods or haulms, used for animal feed were analysed separately and residues of diquat found to be in the range 1.6-9.4 mg/kg.

Table 4. Residues of diquat in peas from supervised trials.

Country/Year	Rate, kg ai/ha	Sample	PHI, days	Residue, mg/kg	Ref.
DE/84	0.6	Fodder peas	5	0.10	81
			7	0.10	
			12	0.07	
			7	0.04	
			4	0.05	
			7	0.03	
			10	0.05	
			5	<0.02	
			7	0.03	
			11	<0.02	
DE/85	0.6	Fodder peas	5	0.06	81
			7	0.04	
			9	0.04	
			5	0.05	
			7	0.04	
			10	0.04	
			3	0.07	
			5	0.06	
			7	0.06	
			3	0.12	
			6	0.13	
			8	0.15	
DK/82	0.6	Peas	15	<0.02, 0.03	133
			21	<0.02, 0.02	
			26	0.02, 0.05	
DK/86	0.6	Peas	0	0.03	108
			5	<0.02	
			7	<0.02	
			9	<0.02	
			13	<0.02	
			0	0.10	

Country/Year	Rate, kg ai/ha	Sample	PHI, days	Residue, mg/kg	Ref.
			3	0.03	
			7	0.04	
			9	0.05	
			14	0.05	
DK/86	0.6	Peas	0	0.02	108
			3	<0.02	
			7	<0.02	
			11	<0.02	
			14	<0.02	
	0.5	Peas	0	0.09	108
			3	0.03	
			7	0.02	
			10	0.03	
			14	0.03	
			0	0.09	
			3	<0.02	
			7	<0.02	
			10	<0.02	
			14	<0.02	
FI/79	0.6	Peas	13	0.04	62
			13	0.05	
			15	0.08	
FI/80	0.6	Peas	13	<0.1	
FI/85	0.6	Peas	10	0.1	74
FR/81	0.6 x 2	Peas	5	<0.05	24
		Pods		4.8	
	0.6	Peas	4	<0.05	
		Pods		9.4	
	0.6	Peas	8	<0.05	
		Haulm		3.3-5.0	
	0.6	Peas	17	<0.05 (2)	
		Haulm		1.6, 2.1	
GB/90	0.6	Seed	5	0.04	29
		Haulm	5	3.6	

Country/Year	Rate, kg ai/ha	Sample	PHI, days	Residue, mg/kg	Ref.
		Seed	10	0.04	
		Haulm	10	2.1	
		Seed	8	<0.03	
		Haulm	8	3.6	

Soya beans (Table 5). Residue trials using diquat for the pre-harvest desiccation of soya were carried out during 1985 in Brazil (Kennedy, 1986g). Commercial rates of application are normally 0.4-0.6 kg ai/ha. Diquat was applied at 0.5 or 0.75 kg ai/ha and residues were <0.02 mg/kg in samples harvested five days later and 0.08-0.09 mg/kg in samples harvested 10 days after treatment.

Two trials on the desiccation of soya were carried out during 1980 in Bulgaria (Swaine, 1982c). With rates of application of 0.6 and 0.7 kg diquat/ha, residues in the beans were either undetectable (<0.05 mg/kg) or just detectable (0.08 mg/kg) when harvested 12 days after treatment.

Several trials on the desiccation of soya beans with diquat were carried out in Canada (Ontario) in 1971 and 1972 (Chipman, 1971/72). In 1971 rates of application were between 0.28 and 0.84 kg ai/ha, mainly 0.42 or 0.56 kg/ha, and harvesting intervals 5-22 days (usually 5-7 days). Similar rates were applied in 1972 but there was a wider range of harvesting intervals (3-43 days). In all cases in both years, there were no detectable residues in the beans (<0.05 mg/kg) or in the meal or oil prepared from them. Residues in the soya straw were in the range 0.27-11.8 mg/kg.

Two trials were carried out in France in 1985 at rates of 0.6 and 0.8 kg diquat/ha, and samples of beans were taken at intervals ranging from 0 to 8 days after treatment. Initial residues on the immature soya were 0.6-0.9 mg/kg (day 0). By five days, the residues were undetectable (<0.1 mg/kg in this study) (Massenot and Culoto, 1985).

Desiccation trials were carried out in seven States in the USA during 1987 using the maximum rate of application of 0.56 kg ai/ha, and seed was collected seven days after treatment (Fujie, 1988c). Residues of diquat in the beans were similar to those in trials from other countries, mainly <0.01-0.04, but with two higher results (0.09 and 0.16 mg/kg).

Soya bean oil and meal were also analysed for residues of diquat in the trials in Canada in 1972. It was found that 3-43 days after application no residues could be detected in 19 samples each of seed, oil or meal (limits of determination 0.05, 0.04 and 0.05 mg/kg respectively) (Chipman, 1971/72).

Table 5. Residues of diquat in soya beans from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
BR/85	0.5	Seed	5	<0.02	88
			10	0.08	
	0.75		5	<0.02	
			10	0.09	
BG/80	0.6	Seed	12	<0.05 (3), 0.08	129
		Pods	12	0.46-1.3	

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
		Stem	12	6.0-20	
	0.7	Seed	12	<0.05 (3), 0.08	
		Pods	12	0.55-2.4	
		Stem	12	4.5-9.6	
CA/71	0.28-0.84	Seed	5-22	<0.05 (14)	20
		Straw	5-14	0.27-1.64 (10)	
		Oil	5-14	<0.04 (10)	
		Meal	5-22	<0.05 (12)	
CA/72	0.28-0.84	Seed	3-43	<0.05 (19)	20
		Straw	3-43	1.8-11.8 (6)	
		Oil	3-43	<0.04 (19)	
		Meal	3-43	<0.05 (19)	
FR/85	0.6	Seed	0	0.63	107
			2	0.37	
			5	<0.1	
			6	<0.1	
			8	<0.1	
			0	0.62	
			2	<0.1	
			5	<0.1	
			6	<0.1	
			8	<0.1	
	0.8	Seed	0	0.59	
			2	<0.1	
			5	<0.1	
			6	<0.1	
			8	<0.1	
			0	0.91	
			2	0.21	
			5	<0.1	
			6	<0.1	
			8	<0.1	
US/87	0.56	Seed	7	0.09, 0.08	48
				<0.01, <0.01	
				0.15, 0.16	
				0.02, <0.01	

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
				0.04, 0.03	
				0.03, 0.02	
			10	0.03 (2)	

Potatoes (Table 6). Using recommended rates of application (0.6-1.0 kg ai/ha) of diquat for the desiccation of potato haulm prior to harvesting the tubers, residues were <0.01-0.04 mg/kg (FAO/WHO, 1971) and <LOD-0.25 mg/kg (FAO/WHO, 1973) in the tubers . Generally, lower rates (0.4-0.8 kg ai/ha) are used for ware potatoes and the higher rates when the potatoes are for seed.

Residues were mainly at or below 0.02 mg/kg (Calderbank and Yuen, 1963) and, in Canadian trials in 1963, at or below 0.06 mg/kg (Calderbank and McKenna, 1964), and seemed to be independent of the application rate. Environmental factors, including dry soil and high humidity, were responsible for higher residues in some earlier trials which caused stem-end rot in the tubers on storage (Headford and Douglas, 1967). Label directions have now eliminated the cause and occurrence of residues above 0.1 mg/kg.

More recent studies in 1985 in Brazil (Kennedy, 1986e), in 1986 in Germany (Kennedy, 1987), in 1988 in Sweden (Earl and Anderson, 1989), in 1982 and 1990 in the UK (Swaine, 1982e; Earl, 1991a) and the Netherlands (Earl, 1991b; Min. Welfare, Health, 1993) all showed residues in the range <0.01-0.05 mg/kg, mostly 0.02 mg/kg or below, which is in good agreement with the earlier results.

Table 6. Residues of diquat in potatoes from supervised trials.

Type, Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Various	0.56-1.68	Tubers	3-21	<0.01-0.04	38
	0.6-1		4-10	<0.01-0.25*	39
				(mean 0.03)	
BR/85	0.4	Tubers	6	<0.02	86
			9	<0.02	
	0.8		6	<0.02	
			9	<0.02	
	1.6		6	<0.02	
			9	<0.02	
DE/86	0.5	Tubers	10-11	0.01 (5), 0.02	89
	1		8-11	0.01, 0.02 (5)	
GB/82	0.8	Tubers	44	<0.01	131
	0.8 and 0.4		37	<0.01	
	0.4 x 2		37	<0.01	
GB/90	0.8	Tubers	14-28	<0.02 (6), 0.03	27
	0.8 + 0.4		7-20	<0.02 (5), 0.03	
	(7-9 days later)				
	0.9		27	<0.02	27
	0.9 + 0.48		20	<0.02	
	1.6		18	<0.02	
	1.6 + 0.8		11	<0.02	
NL/86	0.4	Tubers	15	0.02 (2), 0.03 (2)	141
			18	<0.01, 0.01 (3)	
NL/90	1	Tubers	14-15	<0.02 (4)	28,141
	1.0 x 2		14-15	<0.02, 0.02 (2),	
	(3 days between applns.)			<0.05	
SE/88	0.2	Tubers	13	<0.01	32
			19	<0.01	
	0.4		13	<0.01	
			19	<0.01	
	0.4 + 0.4		9	0.01	
			16	0.01	

* From 36 results, means 0.03 mg/kg (0.25 mg/kg outliers).

Sugar beet (Not tabulated). No new data were available. Residues in sugar beet were evaluated by the 1972 JMPR. An MRL of 0.1 mg/kg was recommended on the basis of residues in only 2 samples (FAO/WHO, 1973).

Cereals (Tables 7-11)

Extensive data on barley and wheat from trials conducted in the UK, Germany and New Zealand in the period 1963-75 (Calderbank, 1968; Calderbank and McKenna, 1964; Reeve, 1972; Ward, 1978) were evaluated by the JMPR (FAO/WHO, 1971, 1973, 1979). These early data, including the ranges of residues in barley, oats, rice, sorghum and wheat are summarized in Table 7.

Factors which affect the magnitude of the residues are the rate of application, the interval between application and harvest, the degree of protection of the seed, and environmental conditions. Desiccation of cereals is normally carried out using rates of diquat of 0.4-0.8 kg ai/ha.

Table 7. Residues of diquat in cereals from supervised trials in various countries, 1963-78 (summary of early data).

Type	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Barley+	0.56-1.12	Grain	3-21	0.5-4.0	42
Barley+	0.1 -0.84	Grain	2-19	<0.05-5.8	138
				mean 3.3*	
Oats	0.42-1.6	Grain	4-17	0.26-2.2	138
				mean 1.0**	
Wheat+	0.56-1.12	Grain	1-21	<0.05-1.3	38
Wheat+	0.60-1.0	Grain	4-7	<0.05-1.6	39
Rice	0.17-0.61	Dehusked	3-21	<0.05-0.16	39
	0.20-3.0	Dehusked	2-16	<0.05-0.96	138
Sorghum	0.3-1.2	Grain	0-30	<0.05-5.9	39

\+ Both spring and winter varieties
* From approx. 100 results
** From 28 results

Barley (Table 8). Residues of diquat in barley are generally about twice those found in wheat from comparable rates of application and intervals. Thus, rates of application of diquat up to 1 kg/ha resulted in residues in the grain of about 1-2 mg/kg (maximum 4 mg/kg) with pre-harvest intervals of 4-7 days (FAO/WHO, 1979).

In trials in the UK in 1980 at commercial rates of application (0.42-0.56 kg ai/ha), residues in the grain ranged from 0.27 to 1.5 mg/kg when the harvest was 4-18 days after application. At the higher rate of 1.1 kg ai/ha diquat residues were 0.86 and 1.5 mg/kg four and six days later (Swaine, 1982a).

In a further eight trials on laid (lodged) barley in the UK (1982) using the maximum rate (0.8 kg ai/ha), residues of diquat in the grain ranged from 0.36 to 0.88 mg/kg when harvested 7-17 days later. At double the rate (1.6 kg/ha), residues of diquat in the grain were 1.0-1.2 mg/kg 7-17 days after

application and 9.8 mg/kg when harvested three days after treatment (Swaine, 1982b). At the normal PHI, residues of diquat found in these later trials all fall within the MRL (5 mg/kg) recommended by the JMPR in 1972 (FAO/WHO, 1973).

Table 8. Residues of diquat in barley from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
GB/1980	0.42	Grain	8	1.5	127
		Ears	5	2.4	
		Grain	18	0.37	
		Grain	4	0.27	
	0.56	Ears	4	1.6	
		Grain	11	0.37	
		Grain	14	0.48	
	1.1	Grain	4	0.86	
		Grain	6	1.5	
GB/1982	0.80	Grain	0	2.5, 3.6	128
			9-10	0.52, 0.36	
	1.6	Grain	0	5.6, 6.1	
			9-10	1.0, 0.91	
	0.8	Grain	3	1.1	132
			7	0.88	
			17	0.37	
	1.6	Grain	3	9.8	
			7	1.2	
			17	0.99	

Maize (Not tabulated). No new data have become available since residues in maize were evaluated by the 1972 JMPR and an MRL of 0.1 mg/kg recommended (FAO/WHO, 1973).

All the data previously evaluated were from 4 trials in France (1962 and 1973, rates 0.59-1 kg ai/ha), one trial in Switzerland (1964, rates 0.6 and 0.9 kg ai/ha) and 4 trials in South Africa using rates of 0.28 and 0.56 kg ai/ha. In all cases (30 results) residues in the maize seed were below the limit of determination (<0.05 mg/kg, refs. 11, 12, 109).

Oats (Table 9). Commercial application rates for diquat used for the desiccation of oats are 0.4-0.8 kg ai/ha.

Trials were carried out in the UK (England and Scotland) and New Zealand in the period 1963-1973 at rates between 0.2 and 1.57 kg ai/ha with PHIs of 3-17 days.

33 results were recorded from 9 separate trials involving at least 15 sites. Diquat residues from commercial application rates (0.4-0.8 kg ai/ha) were in the range 0.24-1.8 mg/kg, with one higher value (2.2 mg/kg) from a total of 18 results. The mean residue was 0.9 mg/kg. The higher residues tended to reflect the shorter PHIs (3-6 days) and higher rates.

No MRL has previously been recommended by the JMPR, but the residue data now available indicate that residue levels of diquat found on oats are of the same order as those found on wheat from similar application rates.

Table 9. Residues of diquat in oat grain from supervised trials (Ref. 143).

Country/Year	Rate, kg ai/ha	PHI, days	Residue, mg/kg
England	0.14	10	0.24
(1962)	0.6	10	0.7
	0.38	12	1.4
	0.57	12	1.8
	0.76	12	2.2
(1973)	0.93	6	2.2
		7	0.42
		4	1.1
	0.7	17	0.87
Scotland	0.63	3	0.93
(1963)	1.25	3	3
(1963)	0.63	4	1.1, 1.7
	1.25	4	2.4, 3.8
	0.2	12	0.22,0.25
		17	0.13
	0.39	12	0.51,0.72
		17	0.4
(1973)	0.45	7	0.59
	0.78	7	0.51
	0.78	7	0.57
	0.78	7	1.2
	1.57	7	3
	1.57	7	1.3
NZ	0.42	6	0.26,0.95

Wheat (Table 10). Residues in wheat grain 4-7 days after treatment at rates up to 1.0 kg diquat/ha were <0.05-1.6 mg/kg with a mean of 0.61 mg/kg (FAO/WHO, 1973). In trials in France in 1977 at the commercial rate (0.6 kg ai/ha) using various formulations, residues of diquat ranged from <0.05 to 0.12 mg/kg 6-7 days after application, and were 0.27 mg/kg at 12 days. At double the application rate (1.2 mg/kg), residues were 0.66 mg/kg 12 days after application (Culoto, 1977). The higher residues at the longer interval were probably caused by further drying out of the grain.

In two more recent trials (1992) in France, diquat was applied at 0.6 kg ai/ha to lodged wheat. Grain harvested six and ten days later showed residues of diquat of 0.77 and 1.1, and 0.43 and 0.66

mg/kg respectively (Benet and Massenot, 1993).

In trials in the UK during 1982 at the maximum recommended rate (0.8 kg ai/ha), grain residues were 0.13 and 0.23 mg/kg at 1-2 days after application. Residues were approximately doubled (0.20 and 0.44 mg/kg) when double the normal rate was used (Swaine, 1982b).

Five trials were conducted in the UK in 1983, using diquat to control late green tillers in wheat (Kennedy, 1984a). The crop was harvested eight days after treatment at 0.6 kg ai/ha and residues were <0.05 to 0.4 mg/kg, in line with earlier data.

Table 10. Residues of diquat in wheat from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
FR/1977	0.6	Grain	6-7	<0.05, 0.06,	22
				0.1, 0.12	
			12	0.27	
		Straw	6-12	2.7, 3.1, 4, 6, 9.6	
	1.2	Grain	6-7	0.05, 0.12, 0.16, 0.16	
			12	0.66	
	Straw	6-12	5, 6, 6.6, 13.2, 14.1		
FR/1992	0.6	Grain	6	0.77, 1.1	4
			10	0.43, 0.66	
GB/1982	0.8	Grain	1	0.13	128
			2	0.23	
	1.6	Grain	1	0.20	
			2	0.44	
GB/1983	0.6	Grain	8	0.24, 0.19, 0.17, 0.40, <0.05 (2)	79

Rice (Table 11). The results of many trials on the desiccation of rice with diquat (in Argentina, Australia, Brazil, Bulgaria, Fiji, France, Hungary, Italy, Japan, Peru and Portugal in the period 1963-73) were summarized by Ward (1978). At commercial rates (0.3-0.6 kg ai/ha), residues of diquat in the whole grain were in the range <0.05-9.0 mg/kg. Most of the results were below 4 mg/kg. Two high results, 9.0 mg/kg (Japan) and 13.0 mg/kg (Peru), were from high rates of 1.0 and 1.5 kg ai/ha and a pre-harvest interval of six days. Residues in rice straw (Japan and Portugal only) were in the range 0.84-22 mg/kg.

Most of the residue of diquat on rice is removed with the husk, and earlier data evaluated by the JMPR showed residues of diquat in the range <0.05-0.16 mg/kg on the dehusked or polished rice following applications of 0.17-0.60 kg ai/ha (FAO/WHO, 1971, 1973).

In trials in 1986 in Japan, residues of diquat and its major photodecomposition product (TOPPS) were both measured in the grain and straw harvested five and seven days following a maximum rate of 0.47 kg ai/ha (Laws *et al*., 1987a).

Residues of diquat in the grain ranged from 0.04-0.13 mg/kg, in good agreement with earlier

data. Residues of diquat in the straw ranged from 3.5-11.0 mg/kg.

Residues of 1,2,3,4-tetrahydro-1-oxopyrido[1,2a]-5-pyrazinium ion (TOPPS) were 0.02-0.06 mg/kg in the grain and 2.0-2.9 mg/kg in the straw (Laws *et al*., 1987a), so its residues in the grain were about half the corresponding residues of diquat.

Table 11. Residues of diquat and TOPPS in rice from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg		Ref.
				Diquat	TOPPS	
Various	0.17-0.61	Grain + husk	3-21	0.7-5.3	na	12
		De-husked or polished grain	3-21	<0.05	na	38
	0.20-0.40	Grain + husk	3-5	<0.05-6.4	na	39
		Polished grain	3-5	<0.05-0.16	na	
	0.20-3.0	Whole grain	2-16	<0.05-13	na	
		Dehusked grain	2-16	<0.05-0.96	na	138
JP/86	0.47	Whole grain	5	0.06, 0.10	0.03, 0.04	94
			7	0.04, 0.13	0.02, 0.06	
	0.47	Straw	5	4.4, 11.0	2.2, 2.9	
			7	3.5, 7.5	2.0, 2.4	

na = not analysed

Sorghum (Table 12). Earlier data on residues in grain, following the desiccation of sorghum with diquat, were from trials carried out in Mexico (ICI, 1969), Argentina, Dominican Republic, France and Italy (ICI, 1970a) and were evaluated by the JMPR in 1972 (FAO/WHO, 1973). Commercial rates vary from 0.4 to 0.8 kg ai/ha.

Residues of diquat from application rates between 0.3 and 1.2 kg ai/ha covered a fairly wide range, but were mostly 0.2-2.0 mg/kg with pre-harvest intervals from 0 to 30 days. Mean residues of diquat from all trials, involving over 200 separate analyses, were of the order of 0.8 mg/kg. There were two unusually high values from trials in France, viz. 12 mg/kg (application rate 0.5 kg/ha and PHI 15 days) and 5.9 mg/kg (application rate 0.6 kg/ha and PHI 4.5 days).

Trials were conducted more recently (1987) in three different States in the USA, using the maximum rate of 0.56 kg diquat/ha (Fujie, 1988a). Residues of diquat in the grain harvested 7-10 days after treatment were in the range 0.42-1.6 mg/kg, in reasonable agreement with the earlier data.

Table 12. Residues of diquat in sorghum from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Various	0.1-1.2	Grain	0-30	0.2-2.0	68,69
(1968-72)	0.4-0.6		4-10	<0.05-5.9 (mean 0.81)	39
US/87					
Texas	0.56	Grain	10	0.42	46

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Kansas	0.56		7	1.6	
Nebraska	0.56		7	1.1	

Cotton seed. No new data were available. Residues of diquat in cotton seed were evaluated by the 1972 JMPR and an MRL of 1.0 mg/kg was recommended for the seed and 0.1 mg/kg for cotton seed oil (FAO/WHO, 1973).

Rape (Table 13). Residue data on oil seed rape obtained from several countries in Northern Europe (Leahey and Allard 1971; ICI, 1970b, 1972) were reviewed by the 1972 JMPR (FAO/WHO, 1973).

Following rates of application of 0.4-0.6 kg/ha (commercial rates are 0.3-0.6 kg ai/ha), diquat residues in the seed were in the range <0.05-1.5 mg/kg 3-10 days after treatment. Mean residues in the whole seed were of the order of 0.4 mg diquat/kg from over 100 separate determinations.

Trials were carried out in the UK in 1980 in which diquat was applied at 0.56 kg ai/ha at three sites, and the seed harvested 7, 14 and 18 days later. The seed was processed into oil and cake, which were analysed separately. Residues in the cake were 0.31 and 0.35 (7 days), 0.21 and 0.23 (14 days) and 0.07-0.22 (18 days). Residues in the oil were all below 0.1 mg/kg (LOD).

In more recent trials in the UK during 1984, diquat was applied for the desiccation of rape at 0.6 kg ai/ha and the seed harvested from 7 to 20 days after treatment. Residues of diquat found in the seed were in the range 0.03-0.38 mg/kg (Kennedy, 1984b). Residues of a similar magnitude (<0.05-0.48 mg/kg) were found in the seed harvested 5-8 days after treating rape at 0.6 kg ai/ha in Germany in 1987 (Kennedy, 1988).

In two trials conducted in 1980 and 1982 in Finland at a rate of application of 0.6 kg diquat/ha, residues in the seed sampled 7 and 13 days after treatment were 0.6 and 0.7 mg/kg respectively (Heinanen, 1980; Jarvinen, 1983).

Trials were carried out in Sweden in 1984/85 (Ref. 142) in which diquat was applied at 0.8 kg ai/ha, and the seed harvested 13-14 days later. The seed was separated into oil and cake. Residues in the cake were 0.17-2.2 mg/kg and those in the oil were below 0.05 mg/kg, the limit of determination.

All the more recent data (since 1980) on rape seed are thus in line with those reported earlier (FAO/WHO, 1973).

Sunflower seed (Table 13). Commercial rates of diquat used for the desiccation of sunflower are 0.4-0.6 kg ai/ha.

Data evaluated previously by the JMPR were derived mainly from desiccation trials in Canada and France in the period 1962-1970 (FAO/WHO, 1973). Using a rate of application of 0.6 kg ai/ha and pre-harvest intervals of 4-15 days, diquat residues were in the range <0.05-0.20 mg/kg.

Other trials, mainly at 0.4-0.6 kg ai/ha, in the same period (up to 1970) in Canada, France, Australia, Chile, Israel and Morocco, showed residues in the seed ranging from <0.05 to 0.7 mg/kg (ICI, 1970c, 1972).

Two trials at higher rates (1.12 and 1.65 kg ai/ha) gave residues of diquat in the seed harvested 1-20 days after treatment up to 1.1 mg/kg (ICI, 1970c).

Supervised trials were carried out in Brazil in 1990/1991, using 0.3, 0.4 and 0.8 kg diquat/ha. The seed was harvested 7 or 14 days after treatment. Residues of diquat were all at or below 0.01 mg/kg (Kamienski, 1991).

Table 13. Residues of diquat in oilseed crops from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Rape					
N Europe, CA	0.3-0.6	Whole seed	3-10	<0.05-1.5	39,70
before 1972				(mean 0.37)*	72,97
GB/80	0.56	Oil	7,14,18	<0.1 (7)	126
		Cake	7,14,18	0.07-0.35 (7)	
GB/84	0.6	Whole seed	7	0.06, 0.2	80
			10	0.03	
			13	0.21	
			20	0.38, 0.18	
DE/87	0.6	Whole seed	5	0.16, 0.26, 0.48	90
			6	0.14, 0.15	
			7-8	<0.05 (2), 0.09	
		Oil	5-7	<0.05 (8)	
FI/80	0.6	Whole seed	7	0.6	62
FI/82	0.6	Whole seed	13	0.7	73
SE/72**	0.6	Whole seed	13	2.4-4**	142
SE/84-85	0.8	Oil	13-14	<0.05	142
SE/84-85	0.8	Cake	13-14	0.17-2.2	142
Sunflower					
BR/90-91	0.3	Seed	7-14	<0.01-0.01	77
	0.4			<0.01-0.01	
	0.8			<0.01-0.01	
CA/FR/1962-70	0.60	Seed	4-15	<0.05-0.20	39
CA/62-70	0.40	Seed	21	0.6, 1.0 (3)	71
	0.56		2-7	0.26-0.72	
	1.12		1-20	<0.05-0.70	
	1.65		1-20	<0.05-1.1	
AU/62-70	0.55	Seed	12	0.15, 0.20	
CL/62-70	0.58	Seed	23	<0.05	
		Oil	23	<0.05	
FR/62-70	0.58	Seed	15	<0.05-0.13	
	0.60		18	<0.05-0.10	
IL/62-70	0.58	Seed	4	<0.05	
		Oil	4	<0.05 (5)	

* From more than 100 results.
** Outliers, plants were very unripe at treatment.

Fodder and forage crops (Table 14). The results of desiccation trials carried out on alfalfa, grass and clover in the period 1965-1976 in France, the UK and Australia are summarized by Swaine and Hayward (1982). The whole plants were analysed since the whole plants are intended for consumption by animals.

These results supplement earlier published data on diquat residues in silage (Black *et al*., 1966), clover hay (Calderbank and Yuen, 1963; Calderbank *et al*., 1966), lucerne (Univ. Perugia, 1967), laid (lodged) cereals (Ward, 1978; Leahey *et al*., 1973), oil seed cake (Leahey and Allard, 1972) and other desiccated crops used to feed farm animals. Some further data will be found on cereal grain in Table 7, rice (Table 11), peas (Table 4), beans (Table 2) and soya beans (Table 5).

The reason for displaying the range of diquat residues found in fodder and forage is not for the purpose of estimating maximum residue levels for these commodities, but to enable a judgement to be made on whether or not residues are likely to occur in products of animal origin.

A wide range of residues is found in crops intended for animal consumption, depending on a variety of factors including the nature of the crop, rate of application, PHI and environmental conditions. Residues of diquat in cereal grains are usually of the order of 1-2 mg/kg, the highest results being recorded in barley (5.8 mg/kg) and rice (9.0 mg/kg). Residues in whole plants and cereal straws cover a wider range, the highest being recorded for lucerne (96 and 120 mg/kg) treated at 0.42 kg ai/ha and harvested 0-1 days after treatment. More usually, residues on desiccated cereal straw are in the range 5-30 mg/kg (Culoto, 1977; Swaine, 1982d; Ward, 1978).

Peas and beans (whole plants) are sometimes used as forage crops after desiccation with diquat. Residues of diquat in the pods or haulm are invariably lower than those found in desiccated grass, clover and cereal crops (Table 4). Diquat residues in soya bean pods and straw are in the range 0.55-20 mg/kg following application rates of 0.28-0.84 kg ai/ha and intervals of 3-43 days (Table 5).

The implications of feeding farm animals with crops containing high residue levels of diquat and the possibility of the transfer of residues to products (meat, milk, eggs etc.) for human consumption are discussed under "Fate of residues in animals".

Table 14. Residues of diquat in fodder/forage crops from supervised trials.

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
Alfalfa					
FR/67	0.15	Whole	2	0.46	134
			4	30	
			5	0.46	
				2.3	
			9	1.1	
				0.59	
	0.30		2	1.4	
			4	0.9	
			5	1.4	
				95	
			9	0.39	
				2.6	
FR/68	0.60		3	5.4	
			4	<0.05	

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
			8	1.1	
GB/74	0.42	Whole	1	7, 28, 34, 23, 49	
			2	1.9, 14, 11, 16	
			3	4, 8.7, 2	
			35	<0.05 (3)	
GB/75	0.42	Whole	0-1	47, 50, 84, 120	
			1-2	6.1, 26, 94	
I/67	1.12	Whole	3	19-23	136
Tall fescue					
FR/68	0.40	Whole	3	9.5	134
			4	1.6, 6, 8.3	
			7	6.4	
GB/74	0.24	Whole	1	3.6	
			2	1.2	
	0.42		0-1	0.52, 1.5, 3.6	
	0.42		2	1.6, 1.9	
AU/76	0.50	Whole	0	6-28 (6)	
	0.50		1	0.8-22 (6)	
Pasture					
GB/61-63	0.29	Whole	1	12-48	5
	0.29		3-4	2.5-11	
	0.29		7	1.0-5.7	
	0.58		1	32-65	
	0.58		3-4	9-26	
			7	2.5-6.5	
Clover					
GB/61	0.56	Whole	4	10-31	11
	0.56		13-22	5-9	
GB/65	0.5	Whole	1-2	45-67 (6)	16
	0.5		7	34, 35	
GB/66	0.1	Whole	0-1	9-18 (7)	134
	0.2		1	13, 15	
	0.3		1	16, 24	
Silage					
GB/61-62	0.21	Whole	7	9-13	5
GB/62	0.60	Whole	7	27	

Country/Year	Rate, kg ai/ha	Crop part	PHI, days	Residue, mg/kg	Ref.
GB/63-64	0.29	Whole	7	1.4-3.6	
CEREALS:					
Barley					
AU, GB, NL,	0.1-0.84	Grain	2-20	<0.05-5.8	138
NZ/63-73	0.1-0.84	Straw	2-20	1.5-45	
Wheat					
AU, GB, JP, NL,	0.1-1.2	Grain	1-35	<0.05-2.3	138
NZ/63-73	0.1-1.2	Straw	1-35	4.3-31	
GB/81	0.8	Straw	0	30	130
			2	18	
			3	6.9	
			6	8.1	
FR/77	0.6	Straw	6-12	2.7-9.6	22
	1.2	Straw	6-12	5.0-14.1	
Oats					
GB, NZ/63-73	0.2-1.6	Grain	4-17	<0.05-2.2	138
		Straw	4-7	0.51-9.3	
Rice					
11 countries/	0.2-3.0	Grain	2-37	<0.05-9.0	138
63-73		Straw	4-9	0.84-22	
OILSEED CAKE:					
Sunflower					
AU/70	0.55	Cake	12	0.33	71
Rape					
CA/70	0.28-0.55	Cake	10-32	0.31-2.0	70
CZ/70	0.3-0.6	Cake	4-8	0.19-1.4	97
NL/70	0.3-0.6	Cake	5-10	0.24-1.3	97

Animal transfer studies

Diquat residue levels in the meat, organs and milk of cows and sheep and in the meat, organs and eggs of hens have been determined from feeding studies using unlabelled diquat both as an incurred residue on desiccated crops and in fortified diets. The results are summarized in Table 15.

Cows. A number of experiments have been carried out in which unlabelled diquat, as a residue on desiccated fodder, has been fed to cows for prolonged periods, and meat, milk and organs analysed for diquat.

No diquat residues were detectable (usually <0.01 mg/kg) in any of the organs, muscle or milk

from the animals. In another residue transfer study, cows were fed higher (fortified) concentrations of diquat of 20, 50 and 100 ppm in their diets for 30 days. No residues of diquat (<0.02 mg/kg) were detected in the tissues of the animals at slaughter, and none (<0.01 mg/kg) in the milk during the feeding period (Edwards *et al*., 1976).

Sheep. An experiment was carried out in which sheep were fed silage containing residues of 6 and 13 mg/kg diquat for eight consecutive days. Diquat intake and excretion were measured over a three-day period and showed that the amount of diquat excreted in the urine was less than 10% of the intake, while the total diquat excreted in the faeces and urine accounted for only 40-50% of the diquat ingested and diquat was not detectable in the sheep tissues. Diquat was shown to be metabolized when incubated in sheep rumen liquor and in a suspension of sheep faeces, suggesting that the undetected diquat had probably been metabolized by the sheep (Black *et al*., 1966).

Hens. In a residue transfer study using unlabelled diquat, levels of 1, 5 and 10 ppm diquat in the diet were fed to chickens for a period of 28 days.

The tissues analysed included heart, skin, liver, gizzard, muscle and fat. During the treatment period the heart, liver, muscle and fat contained less than 0.005 mg/kg diquat; the skin contained a detectable residue (0.006 mg/kg diquat) only at the highest feeding level on treatment day 21; the gizzards contained detectable residues which ranged from 0.006 mg/kg diquat at the lowest feeding level to 0.022 mg/kg at the highest level.

At the end of a further 7-day period on feed containing no diquat, all tissue samples contained less than 0.005 mg/kg, except the gizzard from the middle feeding level which contained 0.006 mg/kg.

Residues in all egg samples taken throughout the dosing period were below the limit of detection (0.005 mg/kg) (Lai *et al*., 1977).

Table 15. Tissue residues in farm animals dosed with "cold" diquat.

Animal, dose	Residue, mg/kg					Ref.
	Meat	Fat	Kidney	Liver	Milk/ eggs	
Sheep						
6.6 and 13.3 mg/kg in diet for 8 d	na	na	<0.01	<0.01	-	5
Cow						
3.6 mg/kg in diet for 1 month	<0.01	na	<0.01	<0.01	<0.003	5
9-66 mg/kg in diet for 17 d	<0.01	<0.01	<0.01	<0.01	<0.01	16
0.3-12 mg/kg in diet for 59 d	<0.01	<0.01	<0.01	<0.01	na	16
20, 50 and 100 mg/kg in diet for 30 d	<0.02	<0.02	<0.02	<0.02	<0.01	37
50 mg/kg in diet for 31 d	<0.01	<0.01	<0.02	<0.01	<0.001	119
0.2 mg/kg in diet for 185 d	na	na	na	na	<0.01	106
Hens						
10 mg/kg in diet for 6 weeks	<0.05	na	<0.2	<0.05	<0.05	36
1, 5 and 10 mg/kg in diet for 28 d	<0.005	<0.005	na	<0.005	<0.005	93

na = not analysed

FATE OF RESIDUES

The metabolism, degradation and movement of [^{14}C]diquat was studied in animals, plants, soil and water, the compound being labelled in the pyridine rings or the ethylene bridge (Figure 1).

Figure 1. Positions of radiolabels in [^{14}C]diquat ion

(a) pyridine label (b) bridge label
*denotes positions of ^{14}C atoms

A summary of the degradation products formed from diquat, with their chemical names, is given in Table 20, and proposed metabolic pathways are shown in Figures 2, 3, 4 and 5. Some of the subject matter has been previously reviewed (Calderbank and Slade, 1976) and was previously evaluated by the 1970 and 1976 Meetings (FAO/WHO, 1971, 1977).

In animals

(A) Studies with diquat alone

The absorption, distribution, metabolism and excretion of [^{14}C]diquat has been studied in the rat, goat, cow and hen. Details of the studies are given in Part II of the Evaluations - Toxicology. They showed that diquat, given as an oral dose, is poorly absorbed and excreted largely unchanged, mainly in the faeces. More than half of the traces of metabolic products were due to the monopyridone (VII). The results of the metabolism studies are summarized in Table 16 and Figure 2.

Table 16. Percentage of original ^{14}C excreted within 3-7 days after oral doses of [^{14}C]diquat.

Animal	Faeces	Urine	Milk	Eggs	Ref.
Rat	89	6	-	-	Part II - Toxicology
Goat	94	2	0.02	-	56
Cow	-	3	0.015	-	125
	91	0.4	0.004*	-	103
Hen	95-99	-	-	0.03-0.06	102

* Expressed as mg diquat equivalents per litre
- Not measured or not relevant

Figure 2. Degradation of diquat in animals.

Rats. Details of the studies are given in Part II - Toxicology. An oral dose of diquat was poorly absorbed: 89% was excreted largely unchanged in the faeces. Diquat was also the major component in the urine (5% of the dose), with small amounts of diquat monopyridone (0.2%) and diquat dipyridone (0.1%) (Compounds VII and VIII, respectively in Figure 2). Diquat monopyridone (4.5%) was also detected in the faeces. There was no significant retention of ^{14}C in tissues.

Goats. In lactating goats, after administration of labelled diquat as a single dose, only 2% of the dose was excreted in the urine within 7 days. A major part of this radioactivity was due to the monopyridone (VII) with a small amount of TOPPS (II). In the faeces, 70-80% of the original dose was present as diquat with small amounts of Compounds II and VII. The low levels in the milk (0.02%) were largely characterized as diquat, monopyridone, TOPPS and natural incorporation into lactose, fat and protein (Griggs and Davis, 1975).

Cows. Similar results were obtained by administering single or multiple doses of [^{14}C]diquat to lactating cows. Most of the dose (91%) was eliminated rapidly in the faeces with small amounts (3% and 0.4%) in the urine. The small proportion (0.015% of the dose, equivalent to 0.004 mg/kg) excreted in the milk was shown to comprise a mixture of diquat, monopyridone, TOPPS and ^{14}C incorporated into lactose, fat and protein (Stevens and Walley, 1966; Leahey *et al*., 1976).

In a calf, slaughtered one day after dosing with [^{14}C]diquat (5 mg/kg body weight), very little (<0.01 mg/kg) diquat or its metabolites were found in the muscle. The kidney and the liver contained detectable ^{14}C residues, 0.66 and 0.20 mg/kg respectively (Stevens and Walley, 1966).

Hens. When ^{14}C-labelled diquat was fed to laying hens, 95-99% of the radioactivity was eliminated in the faeces. As well as diquat (70-80%), the metabolites II and VII were found (2% and 4%, respectively). The remainder of the radioactivity was not identified. A very small radioactive residue (equivalent to <0.003 mg/kg) was found in eggs collected from the hens. The residue in the yolk was accounted for as diquat (35-39%), monopyridone (61%) and TOPPS (2-7%) (Leahey and Hemingway, 1975).

In a later study three hens were dosed daily for four consecutive days with [^{14}C]diquat at a rate of 2.4 mg diquat ion/kg body weight per day, equivalent to 32 ppm in the diet. All the birds were slaughtered approximately 18 hours after receiving the last dose and the radioactive residues (expressed as diquat equivalents) were determined in egg yolk (<0.001 mg/kg), egg white (0.004 mg/kg), liver (0.030-0.045 mg/kg), kidney (0.042-0.058 mg/kg), muscle (0.003 mg/kg) and fat (0.004 mg/kg).

The radioactive residue in the liver was characterized as mainly diquat (48.0%), with small amounts of TOPPS (II, 1.8%), diquat monopyridone (III, 3.9%) and diquat dipyridone (IV, 3.1%).

The identified components of the radioactive residue in the kidneys were diquat (12.0%), diquat monopyridone (III, 15.1%), TOPPS (II, 3.9%) and diquat dipyridone (IV, 6.6%) (French and Leahey, 1988).

The results of the studies with ^{14}C-labelled and unlabelled diquat are summarized in Table 16. Total ^{14}C residues were extremely low, and residues of diquat below the limit of determination.

Overall, it was concluded by the authors of the above studies that diquat is poorly absorbed by animals and rapidly excreted, largely unchanged in the faeces. Only traces of ^{14}C find their way into tissues, milk and eggs. Diquat constitutes part of the residue, the major metabolite is diquat monopyridone and a significant amount of ^{14}C in milk is incorporated into natural lactose, fat and protein.

Table 17. Tissue residues in farm animals dosed with radiolabelled diquat.

Animal, dose	^{14}C residues expressed as diquat ion, mg/kg					Ref.
	Meat	Fat	Kidney	Liver	Milk/ eggs	
Cow						
Single doses of 5 and 20 mg/kg body weight	<0.01	na	na	na	0.004-0.08	125
Bull calf						
Single dose 5 mg/kg body weight	0.005	<0.01	0.66	0.20	-	125
Cow						
1 g cow/day = 30 mg/kg in diet for 7 days	0.004	0.002	0.08	0.05	0.004	103
Hen						
4-5 mg/kg in diet for 14 days	<0.002	<0.001	0.004	0.0004	<0.0005 (egg white)	101
					≤0.02 (yolk)	
2.4 mg/kg body weight for 4 days	0.003	0.004	0.04-0.06	0.03-0.05	<0.004	43

(B) Studies with diquat plus its photoproducts

In practice, a mixture of diquat and its photoproducts will be the residue actually consumed by animals fed crops such as desiccated cereals and alfalfa. Since the main constituent of the photoproduct residue is an unidentified complex, intransigent mixture, formed from degradation products of diquat and plant constituents (Table 18), several studies have been carried out to investigate the fate of such a mixture when fed to animals.

A crop of barley was desiccated with [^{14}C]diquat and the residues of diquat and its photoproducts in the straw were measured before feeding to rats, a goat, a cow and hens. A typical residue composition (in a total of 25 mg diquat ion equivalents per kg barley) was:

Diquat	20-30%
Diquat monopyridone (VII)	2%
TOPPS (II)	10-15%
Picolinic acid (IV)	2%
Other "photoproducts"	60%

(Leahey, 1974; Leahey *et al*., 1973)

Rats. Treated barley straw (10% of the total diet) was fed to rats for eight days. The residue level in the barley was 25 ì g/g diquat ion equivalent. Five rats were killed after four and eight days on the diet. There was no accumulation of radioactivity in any of the tissues (muscle, fat, kidney and liver) examined. The highest residue (^{14}C as diquat ion, 0.03 ì g/g) was found in the kidney (Leahey, 1974).

In a second experiment, rats were fed a similar diet for 20 days. The maximum residue (^{14}C as diquat ion, 0.03 ì g/g) was again found in the kidney (Leahey *et al*., 1974b).

Goats. Barley straw desiccated with [^{14}C]diquat was fed to goats at levels of 2% and 7% of the daily intake. Virtually all the administered radioactivity, of which 59-62% was associated with unidentified photoproducts, was eliminated within ten days, mainly in the faeces with about 5% in the urine. A small radioactive residue (0.0028 ì g/g diquat ion equivalent) was detected in the milk. This was shown to be

mainly due to incorporation of ^{14}C into the natural milk constituents. Residues of diquat and TOPPS were below 0.0003 ì g/g (Leahey, 1974; Hemingway *et al*., 1973).

Cow. In an extension of the above study, a cow was given a single oral dose of barley straw containing 5.2 mCi of ^{14}C-ring-labelled diquat and its photoproducts on 794 g powdered barley straw. Virtually all the radioactive dose was eliminated from the cow within 10 days, mainly in the faeces. Approximately 0.4% of the dose was excreted in the urine and a small radioactive residue (maximum 0.0014 ì g/g diquat ion equivalent) was detected in the milk. The radioactivity in the milk was shown to be mainly (77-90%) incorporated into the lactose, fats and protein (Hemingway *et al*., 1974).

Hens. As with other animals, residues of diquat and its photoproducts are rapidly excreted by hens. There is only an extremely small transfer of residue to tissues or eggs.

Mature barley plants were sprayed with ^{14}C-ring-labelled diquat and left in sunlight for four days before harvesting. Residues of diquat and its photoproducts on the grain were measured and it was then fed to three hens. The first hen was given a single oral dose, of which 96% was recovered in the faeces within five days. The other two hens were dosed for 11 consecutive days at rates equivalent to 1-1.5 ppm diquat and photoproducts in the total diet. There was a very small transfer of radioactivity into the eggs from these hens, the maxima in the albumen and yolk being 0.0006 and 0.0039 ì g diquat ion equivalents/g. The following radioactive residues were detected in the tissues of one of the hens killed four hours after its final dose (ì g diquat ion equivalents/g).

Muscle	0.00086
Fat	0.0022
Heart	0.00083
Kidney	0.014
Liver	0.0047
Lung	0.0014 (Hughes and Leahey, 1975)

The results of the above feeding studies with [^{14}C]diquat plus photoproducts are shown in Table 18. The authors of the reports conclude that residues of diquat and its photoproducts are mainly excreted by ruminant, non-ruminant and avian species, that the compounds are not accumulated in tissues, milk or eggs and that the extremely low levels that are found are without toxicological significance.

In plants

Diquat undergoes rapid and extensive photochemical degradation on plants. The subject was last reviewed by the 1978 JMPR (FAO/WHO, 1979). Photochemical degradation products of diquat (including those formed in water) are listed in Table 18, and a proposed scheme for the photochemical degradation of diquat on plants is shown in Figure 3.

The degradation of [^{14}C]diquat has been studied on maize, tomato, potato, cereal and rape.

Studies with maize and tomato plants showed that diquat is not metabolically degraded by plants. When these plants were treated with diquat and maintained in darkness, no breakdown of diquat occurred. However, a very rapid loss of diquat took place on similar treated plants exposed to sunlight, and degradation continued after the plants were dead. The author concluded that the breakdown of diquat is a photochemical rather than a metabolic process (Slade and Smith, 1967).

Pyridine-labelled diquat was extensively degraded when applied to wheat or barley plants which were then exposed to sunlight. About 80% of the residue was polar and could be extracted by water or dilute acid. Diquat itself is normally the most important single compound, whilst TOPPS (II) is the most important single photoproduct. No other major well-defined degradation product is formed. The bulk of the residue is an ill-defined high-molecular-weight multicomponent mixture. Diquat monopyridone (VII), picolinamide, picolinic acid and oxalic acid have all been identified as minor degradation products, usually not exceeding 5% of the residue in total (Leahey *et al*., 1973; Cavell *et al*., 1978a,b).

Table 18. Excretion and tissue residues* of ^{14}C after oral doses of [^{14}C]diquat and its photoproducts.

Animal/dose	Faeces	Urine	Muscle	Kidney/ liver	Milk/eggs	Ref.
Rat						
2.5 ppm in diet for 8 d	na	na	<0.006	0.02(k) 0.02(L)	-	96
and 20 d	na	na	<0.007	0.03(k) 0.02(L)	-	100
Goat						
Single dose	93-102%	3-5%	na	na	(0.1%) 0.003 max	64
5 x daily	na na	0.0002		0.002(k) 0.001(l)	<0.001	96
Cow						
Single dose	100%	0.4%	na	na 0.001	(0.08%)	65
Hen						
Single dose	96%	-	na	na	na	67
1.0-1.5 ppm ^{14}C in total diet for 11 d	na	-	0.001	0.01	0.005	67

* Results expressed as mg [^{14}C]diquat equivalents/kg tissue unless stated as % of administered dose.
na not analysed

The various fractions of the residue on wheat, oat and barley plants are shown in Table 19. Thus, the photochemical degradation of diquat on plants parallels that which occurs in water (see below), except that on plants a large proportion (35-70%) of the radioactivity applied as diquat is found as an ill-defined complex mixture of compounds which streaks on thin-layer chromatoplates. The streak is caused by many individual radiocompounds all merging together. Attempts have been made to separate and characterize the components of this fraction (named "photoproducts" in Table 19) with the following results.

(i) The water extracts were chromatographed in 44 different solvent systems using neutral, basic and acidic conditions and on silica, alumina, polyamide and cellulose substrates, but the radioactivity always streaked and was always intimately associated with the natural components (Cavell *et al*., 1978a; Leahey *et al*., 1973).

(ii) Extensive fractionation of the radioactivity using a combination of gel-permeation (size-exclusion) chromatography and ion-exchange chromatography showed that it could be separated into a range of fractions varying in molecular weight from below 700 up to 70,000 daltons. Major fractions eluted from the sephadex gel could be further split into smaller fractions.

Application of some of these fractions to cation exchange resins showed that they were mixtures of non-cationic, weakly cationic and strongly cationic components. By multiple fractionation in this way, it was possible to show that no single component of this complex mixture of photoproducts constituted more than 5% of the total radioactive residue. Acid, base or enzyme hydrolysis could not release the radioactivity from these natural plant constituents or change the chromatographic behaviour of the uncharacterized radioactivity (Cavell *et al*., 1978b; Heath and Leahey, 1989).

(iii) The nature of the photoproduct residue was further investigated by causing a light-induced reaction of diquat with cellulose (filter paper) and with glucose. Similar intractable products were formed, which proved impossible to identify Smith, 1967a; Heath, 1992).

In the experiments with glucose, using [^{14}C]diquat and [^{14}C]glucose separately, the products chromatographed as streaks in a similar manner and demonstrated that ^{14}C from both compounds was incorporated into the reaction products. The author concluded that the diquat free radical, formed by the action of sunlight, was binding covalently to glucose and that this was the type of reaction which took place in plants, with the diquat radical binding to organic plant constituents (Heath, 1992).

On the other hand, Smith (1967a) proposed that the complex photoproducts formed on plants were derived from smaller reactive carbon fragments, such as glyoxal, formaldehyde, formic acid and succindialdehyde, formed by the photolysis of diquat (Figure 3), which would be expected to react immediately with natural plant constituents. Support for this suggestion comes from the fact that TOPPS and other pyridinium compounds, which do not form free radicals, nevertheless interact with cellulose on exposure to light in the same manner as diquat (Smith, 1967a).

Table 19. Radioactive residue* after application of [^{14}C]diquat to wheat and barley plants.

Crop/exposure		diquat	TOPPS	Minor products	"Photoproducts"	Ref.
Wheat/9 days		12-25	10	3-5	50-70	18
Wheat/7 days	Straw	32	6	4	58	61
	Grain	51	5	9	35	
	Chaff	23	9	3	66	
Barley/4 days		36	4	3	47	19
Barley/7 + 14 days	Straw	18-21	7-12	5	40-60	98
	Grain	38	10	-	-	
Barley/4 + 14 days		17-36	4-10	3-5	46-50	19
Oats/7 + 14 days	Straw	56	7	-	20	98
	Grain	26	15	-	50	

* Results expressed as % of recovered ^{14}C in that component.

Figure 3. Proposed scheme for photochemical degradation of diquat in plants.

A calculation of the magnitude of the residue of uncharacterized photoproducts may be obtained from the ^{14}C studies on wheat and barley (Table 19). Extensive data for residue levels of diquat on cereal grain and straw, following field rates of application, are available (FAO/WHO, 1971, 1977, 1979 and Tables 7 and 14). From these data, it is possible to calculate an approximate level of "photoproducts" on the basis of a ratio of 2:1 for grain and 3:1 for straw.

The toxicology of the total plant residue has been assessed by feeding [^{14}C]diquat-treated cereals to animals (see Part II - Toxicology and the previous section Fate of residues in animals). Most of the ^{14}C passes through the animals without being absorbed.

Potatoes. No degradation products of diquat were found in potato tubers harvested 14 days after spraying the tops with [^{14}C]diquat. All the ^{14}C residue in the tubers could be accounted for as diquat (Smith, 1967b).

Rape. After desiccation of rape plants with [^{14}C]diquat, no residues of diquat or its photoproducts were found in the oil when the seeds were harvested seven days later. Analysis of the radioactive extract from the rape meal confirmed that the major proportion (about 80%) of the total residue was unchanged diquat (Leahey and Allard, 1971).

Rice. In two supervised trials in Japan, diquat was applied at a normal commercial rate (0.47 kg ai/ha) to desiccate rice plants. Samples of grain and straw taken 5 or 7 days after treatment were analysed for residues of diquat and TOPPS. Residues of TOPPS were about half those of diquat (see Table 11), e.g. whole grain residues: diquat 0.04-0.13 mg/kg; TOPPS 0.02-0.06 mg/kg (Laws *et al*., 1987a)

Table 20. Diquat and its degradation products.

Cpd. No.	Structure & chemical name	Found in:[1]			
		S	W	P	A
I	Diquat 1,1′-ethylene-2,2′-bipyridyldiylium ion	Y	Y	Y	Y
II		Y	Y	Y	Y

	TOPPS 1,2,3,4-tetrahydro-1-oxopyrido[1,2a]-5-pyrazinium ion				
III	picolinamide pyridine-2-carboxamide	No	Y	Y	Y
IV	picolinic acid pyridine-2-carboxylic acid	No	Y	Y	Y
V	3,4-dihydro-8-hydroxy-2H-pyrido[1,2-a]-pyrazine-1,6-dione	No	T	No	No
VI		No	T	No	?

	3,4-dihydro-2H-pyrido-[1,2-a]pyrazine-1,6-dione				
VII	Diquat monopyridone 6,7-dihydro-4-oxo-dipyrido[1,2-a: 2′,1′-c]-pyrazin-8-ium ion	No	Y	Y	Y
VIII	Diquat dipyridone 6,7-dihydrodipyridon-[1,2-a: 2′,1′-c]pyrazine-4,9-dione	No	No	T	Y

[1] S soil; W water; P plants; A animals
Y yes; T trace

In soil

Degradation

Diquat is very stable under acid conditions, but is readily degraded in alkali above pH 9. Under normal soil conditions, it is thus not susceptible to chemical degradation (Calderbank, 1968).

Although diquat is readily photodegraded on plant foliage there is little photodegradation of diquat residues which reach the soil surface, and there is no loss from volatilization (Joseph and Skidmore, 1987).

Diquat is degraded by several common soil micro-organisms when incubated in culture solution with bacteria (Baldwin and Knight, 1967), fungi (Smith *et al*., 1976) and a common soil yeast (Yang and Funderburk, 1978). In soil, however, diquat is firmly adsorbed to clay colloids and degradation is therefore very slow. Experiments therefore have to be carried out over long periods in order to detect real losses against the usual background variation.

A comprehensive long-term field trial was carried out on a sandy loam soil at Frensham (UK). Field plots were treated with 0, 90, 198 and 720 kg/ha diquat incorporated into the soil to a depth of 15

cm (Wilkinson, 1980).

The persistence and movement of diquat residues in the soil were reported after seven years. A loss of diquat of the order of 5-7% per year was recorded. Soil samples taken from lower depths and adjacent untreated areas showed this loss was not due to vertical or horizontal movement of the residues (Gowman *et al*., 1980). The data were analysed statistically after 14 years. The decline in residues at all rates of application was found by the authors to be significant ($P<0.05$) (Cole *et al*., 1986).

The authors concluded that this rate of degradation, although slow, was sufficient to ensure that diquat residues would not accumulate indefinitely in soil but would reach a plateau level when the amount degraded each year was equal to the amount of new addition (Calderbank, 1989).

Since diquat is readily degraded photochemically on leaf surfaces the amount of diquat reaching the soil is expected to be much less than that applied.

A series of trials was performed in Western Europe from 1987 to 1989 to determine residues of diquat in the soil following a single application of 'Reglone' as a desiccant to a variety of crops (potatoes, oil seed rape, peas and sunflowers). The percentage of diquat found in the soil the following spring was, as expected, variable: on average, 75% of the diquat had been lost by degradation on the crop and in the soil (Anderson and Earl, 1990).

Photoproducts of diquat reaching the soil are subject to more rapid degradation than diquat itself. Thus, [^{14}C]diquat plus a mixture of photoproducts formed in the desiccation of barley were degraded (as evidenced by the evolution of $^{14}CO_2$) in acid or alkaline soils under aerobic conditions. Although the radioactive fractions of the ^{14}C residues were not separated, it was shown that TOPPS (an identified photodegradation product) could be degraded in soil (Hill, 1975).

Picolinamide, another photodegradation product of diquat, has also been shown to be readily degraded by an unidentified soil bacterium, or by its cell-free extracts, and the pathway of degradation of the pyridine ring to maleamate and maleate/fumarate (Figure 5) has been elucidated (Orpin *et al*., 1972).

Adsorption/mobility

The adsorption properties of diquat have been tested in four different soils using a batch equilibrium technique. Equilibrium solutions were analysed chemically and by wheat root bioassay. Very high proportions of diquat were bound to the soils in every case with extremely low concentrations in the aqueous phase, even using equilibration levels in excess of 1000 mg diquat per kg soil. At lower rates of application, no diquat was detected in the solution phase and K_d values of soils were estimated to be greater than 10000 (Riley *et al*., 1972).

Adsorption and desorption Freundlich adsorption coefficients (K_d values) for diquat were determined in five soils and one pond sediment. The K_d values ranged from 15 to 10700 and the desorption constants from 20 to 10800. The author concluded that the adsorption of diquat to soils varies from strongly bound to extremely tightly bound (Pack, 1987).

The release of diquat from two sandy loam soils was tested using calcium chloride solutions. The soils could deactivate from 50 to 1100 mg/kg diquat. Addition of 0.1N calcium chloride failed to release the diquat. Some diquat could be released with 0.5N calcium chloride, but this concentration of Ca^{2+} was itself phytotoxic (Riley and Gratton, 1974).

Diquat was the least mobile of a variety of pesticides on soil thin-layer plates, using three

different soil types (Helling and Turner, 1968).

The possibility of diquat being leached into potable water was tested using a model pond-soil-aquifer system and an extremely sandy soil, with low adsorption capacity, as the soil in the system. No diquat (<0.003 mg/l) was found in any aquifer sample (Pack, 1984).

The extremely low mobility of diquat in the field has been confirmed in long-term trials on a sandy loam soil, in which diquat was applied at rates below and above the capacity of the soil to deactivate it. Diquat was incorporated to a depth of 15 cm. Even after 14 years, the residues of diquat found below 15 cm represented a small proportion of that remaining in the 0-15 cm layer. Residues of diquat detected in the soil below 30 cm were negligible (Cole *et al*., 1986).

Groundwater was also analysed for diquat at two sites in Japan, where the product had been used commercially for 5 and 15 years. No diquat was detectable in the water, the limit of detection being 0.1 mg/l (Kuroda and Ishii, 1985).

The mobility of [^{14}C]diquat plus its plant photoproducts (formed on wheat and barley) was also determined in columns of four different soils using almost 80 cm simulated "rain" over an 11-week period. More than 85% of the total radioactivity remained in the 0-5 cm layer, with less than 10% leaching below 10 cm. The authors concluded that the mobilities of diquat and its photoproducts in soil were very low, and that an application of 1 kg diquat/ha to plants would result in less than 0.01 ì g diquat equivalents/ml in water leaching through the soil (Prashad and Newby, 1976).

Uptake into follow-up crops

When diquat is watered on to soil, there is no uptake of the chemical by the plant roots and consequently no phytotoxicity. This is because the chemical is strongly adsorbed and deactivated by the soil colloids. Diquat is, however, taken up by the roots of plants growing in a material with no adsorbing colloids, such as pure sand, with resulting phytotoxicity (Brian *et al*., 1958).

When diquat is used for general weed control, either before crop emergence or between the rows of established crops, there are no detectable residues in the crop because there is no uptake of diquat by plant roots in the presence of soil (Calderbank, 1968).

Using a very sensitive bioassay system, it has been shown that diquat concentrations in the soil need to be very high indeed, above the strong adsorption capacity, before diquat can be detected in the soil solution and be available for uptake by plant roots (Riley and Gratton, 1974).

A confined rotational crop study was conducted on sandy loam soil treated at 1.12 kg/ha with [^{14}C]pyridine-labelled diquat. After 30, 120 and 365 days, carrots, lettuce and wheat were planted and grown to maturity. Plant and soil samples were taken at immature and mature stages of the plants for analysis. In most cases the ^{14}C concentrations in the mature plants (expressed as mg diquat ion/kg) were below the detection limit (<0.008 mg/kg). The ^{14}C concentration was above the limit of detection in carrot leaves 365 days after treatment (0.017 mg/kg) and in wheat straw after 120 and 365 days (0.022 and 0.024 mg/kg respectively).

The radioactive residues in these plants have been attributed to soil contamination. Immature plants contained ^{14}C concentrations (0.035-0.09 mg/kg) above the detection limit but they were not characterized. The soil contained the bulk of the radioactivity, most of it localized in the 0-7.5 cm soil depth (Lee, 1989).

In a field trial, [^{14}C]diquat was applied at 1 kg/ha to a bare soil (sandy loam) and to grass

cover. It was shown that there was no significant uptake of diquat, or of its decomposition products formed by photochemical degradation on treated vegetation, into grass grown on the soil for three years after treatment (Baldwin and Griggs, 1972).

The most conclusive evidence that diquat is not taken up from soil into crop plants comes from the long-term trials at Frensham, UK. Excessively high rates, 90, 198 and 720 kg/ha, of diquat were applied to a sandy loam soil which was monitored for 14 years. Diquat residues in the vegetation were generally below the limit of determination (<0.05 mg/kg) and this applied even to the plot treated at the highest rate after the diquat had had several years to equilibrate with the soil (Cole *et al.*, 1986).

It has also been shown that the uptake of diquat photoproducts by plants is negligible. Powdered barley, oat straw and potato haulm containing [^{14}C]diquat and its photoproducts was mixed into soils at rates equivalent to an application of approximately 1 kg diquat/ha to the crop. Barley, rape, radish, cabbage, winter wheat, carrot and potato plants grown in the soils generally contained less than 0.005 ì g diquat equivalents/g (Leahey *et al*., 1974a; Leahey and Carpenter, 1975).

In water

A scheme for the photochemical degradation of diquat in water is shown in Figure 4.

Sterile aqueous buffer solutions at pH 5, 7 and 9, containing diquat ion at a concentration of approximately 55 mg/l, were incubated at 25°C in the absence of light for 30 days. The authors concluded that under these conditions diquat is hydrolytically stable. There was no significant decrease in its concentration during the incubation at pH 5 and 7, and a decrease of <10% at pH 9 (Upton *et al*., 1985).

Sterile aqueous solutions of [^{14}C]pyridine-labelled diquat (20.1 mg/l diquat ion) at pH 7 were irradiated with light from a Xenon arc lamp filtered to give a spectrum close to that of natural sunlight. The irradiation, carried out at 25 ± 1°C, approximated to Florida spring sunlight. After 32 days of irradiation, 73% of the applied radioactivity was attributed to diquat. The degradation produced TOPPS (II, 12%) and up to four other components, none of which represented more than 5% of the applied radioactivity. The half-life of diquat in aqueous solution at pH 7, under these sterile conditions, was calculated to be 74 days in Florida spring sunlight (Tegala and Skidmore, 1987).

Figure 4. Proposed scheme for photochemical degradation of diquat in water.

Figure 5. Proposed pathway of oxidation of picolinamide (Orpin *et al*., 1972).

When exposed to natural sunlight under non-sterile conditions the photochemical degradation of diquat is more rapid, and 70% degradation occurred in three weeks. TOPPS (II) was found to be the major degradation product. On further irradiation, this compound is degraded to picolinamide (III) and then via picolinic acid (IV) to volatile fragments. The monopyridone (VII) was formed to only a limited extent (Smith and Grove, 1969).

Picolinamide is known to undergo bacterial oxidation with ring opening to form maleic and fumaric acids (Figure 5) (Orpin *et al*., 1972), and pyridine-2-carboxylic acid has been shown to be similarly hydroxylated under the action of light (Kurokawa *et al*., 1973).

In a later study using ^{14}C-bridge-labelled diquat the dialdehyde glyoxal (IX) was shown to be formed as TOPPS is degraded further to picolinamide. The glyoxal is further oxidized to oxalic acid (X) and also to formic acid (XI) and carbon dioxide (Figure 4) (Cavell *et al*., 1978a).

The minor degradation pathway results in the formation of the pyridones V, VI and VII, which are further degraded to the same volatile fragments. The pyridone VII, whose structure was confirmed by synthesis, is present in only trace amounts but can be detected by its intense fluorescence (Cavell *et al*., 1978a; Calderbank *et al*., 1972).

Diquat is frequently added to natural waters to control submerged and floating aquatic weeds at relatively low concentrations (at or below 1 mg diquat/l). In these situations residues in the water rapidly decline, owing mainly to the absorption of diquat into the aquatic plants where it is firmly bound until the decaying weeds disintegrate into the bottom mud. The diquat is then irreversibly bound to the soil particles, leaving the water free of diquat residues. Half-lives of diquat in natural waters are generally less than 48 hours (Calderbank, 1972).

Stability of residues in stored analytical samples

The stability of diquat residues in macerated samples, stored for six months at deep-freeze temperatures, has been studied in clover seed and hay, sorghum grain, soya beans, carrots, lettuce, potatoes, wheat grain and straw, and rice grain and straw (Fujie, 1988e).

For clover, sorghum and soya beans, field-incurred aged residues were present in the treated samples and the stability was assessed by re-analysis of replicate samples from the treated crops.

For carrots, lettuce, wheat, rice and potatoes, untreated control samples were fortified with a standard solution of diquat cation and the fortified samples analysed at appropriate intervals.

The average recoveries from the various crops after 1- to 6-month storage intervals at -20°C are given in Table 21, which also shows the analytical recoveries from each crop.

Table 21. Stability of diquat residues in stored analytical samples and analytical recoveries.

Crop	Residue level (mg/kg)	% of initial residue ± % cv*	Average recovery from fortified samples ± % cv*
Clover seed	0.25	136 ± 19 (N=8)	97 ± 14 (N=5)
Clover hay	0.50	106 ± 7.9 (N=8)	87 ± 9.7 (N=5)
Sorghum grain	0.25	109 ± 8.8 (N=8)	83 ± 8.7 (N=5)
Soya beans	0.25	98 ± 14 (N=8)	90 ± 13 (N=5)
Carrot root	0.10	82 ± 9.1 (N=6)	79 ± 13 (N=4)
Lettuce	0.10	83 ± 7.4 (N=6)	76 ± 8.1 (N=4)
Wheat grain	0.25	93 ± 5.9 (N=6)	92 ± 4.9 (N=4)
Wheat straw	0.50	82 ± 4.2 (N=6)	85 ± 9.7 (N=4)
Rice grain	0.25	90 ± 2.3 (N=8)	86 ± 7.8 (N=5)
Rice straw	0.50	94 ± 6.0 (N=8)	93 ± 12 (N=5)
Potato tuber	0.10	85 ± 4.4 (N=8)	84 ± 5.2 (N=5)

* % CV = percentage coefficient of variation for N analysed samples. All the samples were analysed after 1, 3 and 6 months storage. The figures in the middle column represent the average recoveries after these periods compared with the initial values.

The author concluded that diquat residues were stable in macerated crop matrices for a minimum of six months when stored at -20°C. The wide range of crops studied, including those with high and low moisture contents, indicates that diquat should be stable in all types of crop (Fujie, 1988e).

Residues of diquat, together with its major photochemical degradation product TOPPS, have been shown to be stable on rice grain and straw for a period of at least five months when stored at -20 ± 5°C (Laws *et al*., 1987b).

In an earlier study, diquat residues on wheat and barley grain were found to be stable when stored for periods up to six months at ambient temperatures or six to eight months at -18 ± 5°C (Bullock, 1980).

In processing

Residues of diquat have been determined in products from the processing of barley, wheat, soya beans, sorghum grain and oilseed crops.

Barley. Diquat may be used for the desiccation of lodged barley intended for animal consumption. Such barley is not usually of the quality required for malting, but some limited data were submitted to the 1978 JMPR showing that residues in beer would be approximately 2-3% of those found in whole grain. With mean residues in barley grain of about 1.7 mg/kg at effective use rates, it can be expected that diquat residues in beer would be of the order of 0.05 mg/l (FAO/WHO, 1971, 1979; Calderbank and Springett, 1971).

Wheat. Wheat desiccated with diquat is intended mainly for animal consumption. Nevertheless, to cater for the possibility that some grain from treated crops might occasionally be used for human purposes, results of processing studies were assessed by the 1978 JMPR.

The average residue in wheat grain following desiccation at effective use rates is close to 0.5 mg/kg. Residue levels in bran are usually about twice those found in the whole grain. The highest residue found in bran was 2.7 mg/kg. Residues in fine offal are comparable to those in grain, and in white flour generally 20-25% of those levels. All samples of white flour contained residues below 0.2 mg/kg (maximum 0.19 mg/kg, average 0.07 mg/kg from 47 results from the UK, Germany and New

Zealand).

The baking process does not affect diquat residue levels; residues in white flour and white bread are essentially the same. Residue levels in wholemeal bread are slightly lower than those in grain owing to the higher moisture content of bread (FAO/WHO, 1971, 1979; Edwards *et al*., 1976b, Ref. 144).

Soya beans. Diquat was applied at 2.8 kg/ha (5 times the recommended rate) to soya beans in Iowa (USA). After seven days, the beans were mechanically harvested and frozen.

The beans were analysed and then carried through a small-scale processing procedure. Two samples of the various fractions were analysed with the following results.

Fraction	Diquat ion, mg/kg
Whole bean (as harvested)	0.24, 0.25
Whole bean (at processing)	0.25, 0.14
Hulls	0.65, 0.50
Meal, solvent extracted	0.18, 0.14
Oil, crude	<0.01, <0.0
Oil, refined	<0.01, <0.01
Soapstock	0.02 0.03

There was a 2.6-fold concentration of diquat residues in the hulls of treated soya beans. There was no concentration in any other fraction and no residues were detectable in the crude or refined oil (Fujie, 1988d).

Sorghum grain. Diquat was applied at 2.8 kg/ha (5 times the recommended rate) to sorghum in Texas (USA) and the grain harvested (and frozen) ten days later.

The grain was analysed and passed through a small-scale procedure for both wet and dry milling. Two separate samples of all the fractions were analysed for diquat with the following results.

Fraction	Diquat ion mg/kg
Dry milling	
Grain	2.6, 2.9
Bran	11, 10
Fine grits	0.92, 0.67
Decorticated grain	0.16, 0.10
Wet milling	
Bran	2.6, 2.4
Starch	0.14, 0.14
Paste	0.37, 0.14

The results show an average four-fold concentration of diquat residue in the bran fraction from dry milling. All other fractions from dry and wet milling show a significant reduction in residue level (Fujie, 1988b).

Oilseeds. With oilseeds such as sunflower, rape and cotton, the diquat residue is concentrated in the cake and there are no detectable residues in the expressed oil (FAO/WHO, 1973; Leahey and Allard,

1971). Some results are shown in Table 13.

METHODS OF RESIDUE ANALYSIS

Diquat

Methods are available for determining diquat in a range of food crops, animal tissue, milk, water and soil. They are mainly based on extracting the compound with 1M sulphuric acid under reflux and passing the extract through a cation-exchange resin, which retains diquat. After washing the column with dilute hydrochloric acid, the diquat is eluted into a small volume of 6M ammonium chloride and determined as the radical ion by spectrophotometry after reduction with alkaline sodium dithionite (Calderbank and Yuen, 1966; Black *et al*., 1966).

Aqueous solutions do not require sulphuric acid extraction and soils require special treatment, viz. refluxing with 6M sulphuric acid, to extract the diquat Pack, 1967; Leary, 1978).

Some modifications or refinements have since been introduced for treatment of various matrices as follows.

Food Crops (Kennedy, 1986a; Earl and Boseley, 1989)

Sample	Sample size	Limit of determination, mg/kg	% recovery expected
Vegetables and fruit (including olives)	250 g	0.01	70-85
Grain and seed (including oilseed crops)	50 g	0.05	50-70
Grass and straw	100 g 25 g	0.05 0.10	70-85 70-85

Other matrices

Sample	Sample size	Limit of determination	% recovery expected	Ref.
Soil	50 g	0.01 mg/kg	85-95	33,83
Animal tissues	25 g	0.05 mg/kg	80-95	31,84
Water	500 ml	1.0 ì g/l	70-100	33
Milk & other liquid samples	1 l 5 l	0.5 ì g/l 0.1 ì g/l	30 26	

An alternative procedure for the analysis of diquat residues in agricultural commodities

involving quantification by gas chromatography has been developed and is based on a published method (Kawase *et al*. 1984).

In summary, the method involves extracting diquat by acid hydrolysis (usually 9M sulphuric acid), clean-up and concentration by ion exchange chromatography, reduction with sodium borohydride, and measurement of one of the diquat reduction products by gas chromatography using a nitrogen-phosphorus flame-ionisation detector (Hamada *et al*., 1987a, 1987b).

Recoveries from various matrices are given below.

Sample	Sample size	Limit of determination mg/kg	% recovery	Ref.
Food crops	50, 25 g*	0.01, 0.02*	108 ± 7 90 ± 9	57 45
Soil	50 g	0.01	83-98	58
Animal tissue	50 g	0.01-0.02	94 ± 15 91 ± 20	44
Water & other liquid samples	250 g	0.004	110 ± 16	75

* Half weight taken for crops with low moisture content.

TOPPS in food crops

The major degradation product TOPPS (II), formed by the action of sunlight on diquat residues on plants, can be extracted from crops together with diquat by refluxing with 0.5M sulphuric acid. After concentration and clean-up on a cation-exchange column, TOPPS is eluted and determined by HPLC with UV detection at 268 nm. Recoveries are shown below (Laws *et al*., 1987a).

Sample	Fortification mg/kg	Mean recovery, % (SD)
Rice grain (50 g)	1.0	81 (11)
	0.1	75 (3)
Rice straw (25 g)	0.1	86 (13)

NATIONAL MAXIMUM RESIDUE LIMITS

The national MRLs shown below, grouped according to country and commodity, were brought to the attention of the Meeting.

Country	Country	MRL, mg/kg
Australia	BARLEY	5
	BEANS	1
	COTTON SEED	1
	FLOUR	0.2
	MAIZE	0.1
	MEAT, meat products	0.05
	MILK, whole	0.01
	OIL, cotton seed	0.05
	OIL, rape seed	0.05
	OIL, sesame	0.05
	OIL, sunflower	0.05
	ONIONS	0.1
	PEAS	0.1
	POPPY SEED	5
	POTATOES	0.2
	RAPE SEED	2
	RICE, in husk	5
	RICE, polished	1
	SORGHUM	2
	SUGAR BEET	0.1
	SUNFLOWER	1
	VEGETABLES	0.05
	WHEAT	2
Austria	EGGS, without shell	0.05
	MEAT	0.05
Belgium	CEREALS, for fodder	0.1
	FRUIT	0.05
	POTATO, tuber	0.05
	RICE, in husk	5
	RICE, polished	0.2
	SORGHUM	2
	VEGETABLES	0.1
Canada	CROPS, other	0.1
	LENTILS	0.2
CIS	PEAS	0.05

Country	Country	MRL, mg/kg
Czechoslovakia	ALFALFA, straw	1.5
France	BEANS	0.1
	FRUIT	0.05
	POTATOES	0.05
	SOYA	0.1
	VEGETABLES	0.1
	WHEAT	1
Germany	BARLEY	5
	CEREALS, processed	1
	CROPS, other	0.05
	HOPS	0.05
	OIL, cole seed	0.1
	OIL, rape seed	0.1
	POTATOES	0.1
	RAPE SEED	2
	RAPE, bird	2
	VEGETABLES	0.1
	WHEAT	2
	WHEAT, bran	3
Hungary	SUNFLOWER	1
Italy	ALFALFA	0.1
	FRUIT	0.05
	MAIZE	0.1
	VEGETABLES	0.1
Japan	FRUIT	0.03
	POTATOES	0.05
	RICE, polished	0.1
	TEA	0.3
	VEGETABLES	0.05
	WHEAT/CEREALS	0.03
Kenya	MAIZE	0.1
	OIL, cotton seed	0.1
	OIL, rape seed	0.1
	OIL, sesame	0.1
	OIL, sunflower	0.1
	RAPE SEED	2

Country	Country	MRL, mg/kg
	RICE, in husk	5
	RICE, polished	0.1
	SORGHUM	2
Netherlands	COTTON SEED	1
	CROPS	0.05
	FRUIT	0.05
	MEAT	0.05
	MILK	0.01
	OIL SEED CROPS	0.1
	PLANT OILS and FAT	0.1
	POPPY SEED	5
	POTATOES	0.05
	PULSES	0.1
	RAPESEED	2
	RICE, husk	5
	RICE, polished	0.2
	SORGHUM	2
	SUNFLOWER SEED	0.5
	VEGETABLES	0.1
	OTHER CEREALS	0.1
	OTHER FOOD COMMODITIES	0.05
Romania	PEAS	0.05
Spain	BANANA	0.05
	CARROTS	0.1
	CELERY	0.1
	CITRUS FRUIT	0.05
Spain	COTTON SEED	0.5
	MAIZE	0.05
	OLIVES	0.05
	ONIONS	0.1
	PARSLEY	0.1
	PEPPER	0.1
	POTATOES	0.1
	SUGAR BEET	0.1
	SUNFLOWER SEED	0.1
	VEGETABLE products	0.05

Country	Country	MRL, mg/kg
	VINE	0.05
	OTHER VEGETABLES	0.1
Sweden	BEANS	0.1
	PEAS	0.1
	POTATOES	0.1
Switzerland	FRUIT	0.02
	VEGETABLES	0.02

APPRAISAL

Diquat, previously evaluated for residues by the JMPR in 1970, 1972, 1976, 1977 and 1978, is included in the CCPR periodic review programme.

Diquat is a non-selective contact herbicide and crop desiccant. It is strongly adsorbed to soil and is not taken up by plant roots. When used as a herbicide to control weeds before planting or emergence, between the rows of established crops, or even just after emergence, no residues (<0.05 mg/kg) are found in the harvested crop. Small residues which may be found occasionally are caused by contamination.

The major use of diquat is for pre-harvest desiccation to aid the harvesting of seed and fodder crops. Residues of diquat are found from this application, mainly from direct contact of the spray with the raw agricultural commodity.

New data from supervised residue trials on crops for which MRLs have previously been recommended were available to the Meeting, together with data on two other crops (soya beans and lentils).

Additional data were also received on diquat residues in processed fractions from sorghum and soya beans, in addition to the previously evaluated trials on wheat, barley and oilseed crops. In wheat, residues in the bran (maximum 2.7 mg/kg) are about twice those found in the grain, while residues in white flour (maximum 0.19 mg/kg) are 20-25% of those in whole grain. The baking process does not affect diquat levels. In soya beans there is a 2.6-fold concentration in the hulls, but no concentration in any other fraction, and no residues are detectable in the crude oil. In oilseed the diquat residue is concentrated in the cake and there are no detectable residues (<0.05 mg/kg) in the expressed oil.

Data were also presented on the stability of residues in crops stored at deep freeze and ambient temperatures. Residues of diquat and its major degradation product, 1,2,3,4-tetrahydro-1-oxopyrido[1,2a]-5-pyrazinium ion, (TOPPS), are stable in crops for a minimum of six months when stored at -20°C.

Analytical methods are based on extraction of diquat by acid hydrolysis and clean-up and concentration by ion exchange chromatography, followed by reduction and measurement of the diquat reduction products by GLC with an NP detector. The limits of determination are 0.004 mg/kg in water, 0.01 mg/kg in soil and 0.02 mg/kg in animal tissues and food crops. In other methods diquat is determined as the radical ion by spectrophotometry with limits of determination of 0.001 mg/kg in water, 0.005 mg/kg in milk, 0.01 mg/kg in soil, vegetables and fruits, 0.05 mg/kg in grain, seeds, oilseed crops, grass and animal tissues, and 0.1 mg/kg in straw.

The metabolism of diquat was studied in laboratory and domestic animals (goats, cows and hens) and also in plants and soil. There is little metabolism in animals and the major products formed have been identified as diquat monopyridone, diquat dipyridone and TOPPS. Degradation in soil is very slow, but significant over a long period. Diquat is very strongly bound in soil.

There is no metabolism of diquat in plants, but photodegradation on plant surfaces and in water is extensive. Photodegradation and strong adsorption to soil thus represent the most important processes for removing or negating the effects of diquat on the environment.

The photodegradation pathway of diquat in water has been elucidated. TOPPS was found to be the major degradation product. On further irradiation, this compound is degraded to picolinamide and then via picolinic acid to volatile fragments. The monopyridone was formed to only a limited extent.

In plants the main photoproduct TOPPS occurs as a residue, 7-14 days after treatment, at roughly half to one-third the level of diquat. Other products of diquat photodegradation on plants appear to be incorporated into natural plant constituents. The Meeting agreed that the residue should be defined as diquat cation, the position taken in previous JMPR reviews.

The new data on residues from supervised trials, together with the information previously reviewed, were evaluated as follows.

Onion, Bulb. No new data were submitted since the evaluation in 1970. The Meeting agreed to withdraw the recommendation of 0.1 mg/kg because the results were too few to estimate a maximum residue level.

The residue data for beans (dry), lentils, peas (dry) and soya beans are mutually supportive and the residues were evaluated together.

Numerous further results from residue trials (8 for bean, 66 for peas, 64 for lentils and 50 for soya beans) from many countries showed residues from <0.01 to 0.2 mg/kg. The Meeting agreed to replace the recommendation for shelled beans and shelled peas by recommendations for beans (dry), lentils, peas (dry) and soya bean (dry) of 0.2 mg/kg. In soya bean hulls the residues ranged from 0.5 to 2.4 mg/kg.

Potatoes. On the basis of a large number of new residue data the Meeting estimated a maximum residue level of 0.05 mg/kg for potatoes to replace the previous recommendation (0.2 mg/kg).

Sugar beet. No new data have been submitted since the last evaluation in 1972. The Meeting agreed to withdraw the recommendation of 0.1 mg/kg because the two results available were not enough to estimate a maximum residue level.

Other vegetables. The Meeting agreed to withdraw the recommendation (0.05* mg/kg) and substitute recommendations for specific vegetables where information on GAP and sufficient valid residue data are available.

Barley. Since the residue situation is well covered by the many results evaluated by earlier Meetings and by additional newer values, the Meeting agreed to maintain the current recommendation of 5 mg/kg.

Maize. No new data have been provided since residues in maize were evaluated by the 1972 JMPR, but more precise references have now been made available to support the original data. In all cases (30 results) residues in maize seed were below the limit of determination (<0.05 mg/kg). The Meeting estimated a maximum residue level for maize of 0.05* mg/kg as being a practical limit of

determination.

Oats. Thirty three results from residue trials carried out in the United Kingdom, Canada and New Zealand in 1963-1973 were submitted. Diquat residues following applications at commercial rates (0.4-0.8 kg ai/ha) were in the range 0.24-1.8 mg/kg, with one higher result (2.2 mg/kg) from a total of 18 results. The mean residue was 0.9 mg/kg. These levels are of the same order as those found on wheat following application at similar rates. The Meeting estimated a maximum residue level of 2 mg/kg for oats.

Rice. Newer residue trials in Japan show residues in paddy rice of 0.02-0.13 mg/kg, but earlier values from trials at commercial rates (0.3-0.6 kg ai/ha) were in the range of <0.05-9 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg for paddy rice to replace the previous recommendation (5 mg/kg). In processing studies on paddy rice treated at exaggerated application rates, residues of 0.96 mg/kg were found in dehusked rice prepared from paddy rice containing diquat at 13 mg/kg. Residues of 0.16 mg/kg were found in polished rice from paddy rice containing 6.4 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg for husked rice to replace the previous recommendation (0.2 mg/kg) and agreed to maintain the current recommendation for polished rice (0.2 mg/kg).

Sorghum. On the basis of earlier residue results and newer data the Meeting agreed to maintain the current recommendation of 2 mg/kg for sorghum.

Wheat. New residue values together with earlier data support the previous MRL. The Meeting agreed to maintain the current recommendations of 2 mg/kg for wheat and wheat wholemeal.

Wheat bran, unprocessed. On the basis of the residues (maximum 2.7 mg/kg) evaluated by the JMPR in 1978 the previous MRL of 5 mg/kg can be supported.

Wheat flour. Wheat milling studies showed that diquat residues in the flour were approximately 20-25% of the residues in the grain. Because the recommendation for wheat is 2 mg/kg, the Meeting estimated a maximum residue level of 0.5 mg/kg.

Cotton seed. No new residue data were available, nor were the original data (14 results) on which the 1972 recommendation for the seed of 1 mg/kg was based submitted for re-evaluation. There was information on only two GAP applications (Spain, Australia) for use as a desiccant in cotton. The Meeting agreed to withdraw the recommendation for cotton seed (1 mg/kg).

Poppy seed. No new residue data were available and the original data were not submitted for re-evaluation. The Meeting agreed to withdraw the recommendation for poppy seed (5 mg/kg).

Rape seed. The previous MRL is supported by newer residue data on the whole seed. The Meeting agreed to maintain the current recommendation of 2 mg/kg for rape seed.

Sunflower seed. Data from Australia, Canada, Chile, France and Israel showed residues in the range <0.05 to 1 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg for sunflower seed to replace the previous recommendation (0.5 mg/kg).

Vegetable oils. One of the uses of diquat is as a pre-harvest desiccant on a range of oilseed crops. The residues in the extracted oils are consistently undetectable (see the individual commodities rape seed oil, soya bean oil, sunflower seed oil). This is to be expected in view of the ionic nature of diquat. Both the underlying science and the available data support the estimation of a group maximum residue level for crude vegetable oils of 0.05* mg/kg as being a practical limit of determination.

Because no residue information was available for the edible oils of cotton seed, rape seed,

sesame seed or sunflower seed, the Meeting agreed to withdraw the respective recommendations, but because residues are not detectable in the crude oils there would be no residues in the edible oils. Similar comments apply to soya bean oil, for which there is no current recommendation.

Meat and edible offal (mammalian). New trials on farm animals (cattle and sheep) showed no measurable residues (<0.02 mg/kg) in tissues when feed containing levels of diquat up to 100 ppm in the diet was fed for 30 days. Maximum residues from alfalfa would be expected up to 95 ppm. The Meeting confirmed the previous maximum residue level estimate of 0.05* mg/kg for cattle meat and edible offal (mammalian) this being a practical limit of determination.

Milks. Diquat residues were not detectable (<0.01 mg/kg) in the milk from cows on feed containing up to 100 ppm diquat. These new results support the previous MRL of 0.01* mg/kg.

Poultry meat and edible offal. Trials on hens showed no measurable residues in poultry (meat and edible offal) when the feed contained 10 ppm diquat. The Meeting estimated a new maximum residue level for poultry meat and edible offal of 0.05* mg/kg, this being a practical limit of determination.

Eggs. Newer investigations indicate that no residues are measurable (<0.01 mg/kg) in eggs from hens consuming feed containing 10 ppm diquat. The Meeting confirmed the previous maximum residue level estimate for eggs of 0.05* mg/kg, a practical limit of determination.

Animal feeds. Results of desiccation trials carried out on alfalfa and clover showed that the recommended MRLs were compatible with the MRLs for animal commodities.

Alfalfa fodder. Results from 6 supervised trials on alfalfa (whole plant) covered a wide range. On the basis of the highest value of 95 mg/kg 3-5 days after treatment at 0.3 kg ai/ha the Meeting estimated a maximum residue level of 100 mg/kg for alfalfa fodder.

Clover. Residues in clover 4-7 days after treatment at 0.5-0.56 kg ai/ha were 10-35 mg/kg. The Meeting estimated a maximum residue level of 50 mg/kg for clover.

RECOMMENDATIONS

On the basis of further data available on residues from supervised trials and current GAP the Meeting concluded that the residue levels listed below are suitable for establishing MRLs.

Definition of the residue: diquat cation.

Commodity		Recommended MRL (mg/kg)		PHI (days)
CCN	Name	New	Previous	
AL 1020	Alfalfa fodder	100	-	3 - 5
GC 0640	Barley	5	5	7 - 14
VP 0062	Beans, shelled	W	0.5	
VD 0071	Beans (dry)	0.2	-	3 - 7
AL 1023	Clover	50	-	4 - 7
SO 0691	Cotton seed	W	1	
OR 0691	Cotton seed oil, edible	W	0.1	
MO 0105	Edible offal (mammalian)	0.05*	0.05*	-

Commodity		Recommended MRL (mg/kg)		PHI (days)
CCN	Name	New	Previous	
PE 0112	Eggs	0.05*	0.05*	-
VD 0533	Lentil (dry)	0.2	-	7 - 19
GC 0645	Maize	0.05*	0.1	-
MM 0095	Meat	0.05*	0.05*	-
ML 0106	Milks	0.01*	0.01*	-
GC 0647	Oats	2	-	7 - 17
VA 0385	Onion, Bulb	W	0.1	
VP 0064	Peas, shelled	W	0.1	
VD 0072	Peas (dry)	0.2	-	3 - 26
SO 0698	Poppy seed	W	5	
VR 0589	Potato	0.05	0.2	6 - 28
PO 0111	Poultry, Edible	0.05*	-	
	offal of			
PM 0110	Poultry meat	0.05*	-	
SO 0495	Rape seed	2	2	5 - 20
OR 0495	Rape seed oil, edible	W[1]	0.1	
GC 0649	Rice	10	5	3 - 21
CM 0649	Rice, husked	1	0.2	3 - 21
CM 1205	Rice, polished	0.2	0.2	3 - 21
OR 0700	Sesame seed oil, edible	W	0.1	-
GC 0651	Sorghum	2	2	7 - 10
VD 0541	Soya bean (dry)	0.2	-	3 - 43
VR 0596	Sugar beet	W	0.1	-
SO 0702	Sunflower seed	1	0.5	2 - 23
OR 0702	Sunflower seed oil, edible	W[1]	0.1	
	Vegetables (except as otherwise listed)	W	0.05*	-
OC 0172	Vegetable oils, crude	0.05*	-	-
GC 0654	Wheat	2	2	6 - 12
CM 0654	Wheat bran, unprocessed	5	5	-
CF 1211	Wheat flour	0.5	0.2	-
CF 1212	Wheat wholemeal	2	2	

W The previous recommendation is withdrawn. [1] The recommendation is replaced by a recommendation for vegetable oils, crude.
* At or about the limit of determination

FURTHER WORK OR INFORMATION

Desirable

Additional data on soya bean oil and soya bean meal.

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