Diquat (031)

First draft prepared by Dr Samuel Margerison, Australian Pesticides and Veterinary Medicines Authority, Canberra, Australia

EXPLANATION

Diquat is a herbicide used in a variety of crops and was first reviewed by the 1970 JMPR. The toxicology and residues aspects of diquat were reconsidered by the 2013 JMPR as part of the periodic review program. The 2013 JMPR established an ADI of 0–0.006 mg/kg bw and an ARfD of 0.8 mg/kg bw, and recommended a residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities of diquat cation, along with maximum residue levels in a number of plant and animal commodities.

Diquat was scheduled by the Forty-ninth Session of the CCPR (2017) for evaluation of additional residues data in pulses and cereals. The company sponsor supplied information on the Canadian GAP in pulses and supervised residue trials covering pre-harvest dessication use in dry peas, dry beans, lentils and chickpeas as well as the final report for an interim storage stability study considered by the 2013 JMPR. The company sponsor, together with the government of Australia supplied information on the Australian GAP in cereals, and supervised residue trials on early post-emergent weed control use and pre-harvest weed control use in wheat, barley and oats.

RESIDUE ANALYSIS

Analytical methods

Plant commodities

Samples from the Canadian residue trials in pulses and the Australian residue trials in cereals were analysed using an LC-MS/MS method (based on method number GRM012.03A, with minor modifications), which was evaluated for the 2013 JMPR. Briefly, this method involved extracting homogenised samples by refluxing for approximately 5 hours in sulfuric acid, followed by centrifuging and filtering of a portion of the cooled extract. An aliquot was diluted to volume with ammonium formate (pH 3.7) buffer and acetonitrile, and analysed by LC-MS/MS. The method LOQ was 0.01 mg/kg (as the diquat cation).

Since the commodities for which the method was validated did not include a high protein commodity, validation data in pulses generated as part of the residue studies submitted to the Meeting is tabulated below.

Table 1 Concurrent recoveries for diquat ion from pulse seed matrices

Matrix	Fortification level (ppm)	Individual recoveries (%)	Mean (%RSD) (%)	Reference
Chickpea seed	0.01	92, 97, 100, 101	98 (4.8)	Sagan, 2017-a (TK0225767)
	0.10	93, 100, 101, 101	99 (3.9)	
	Combined		98 (4.1)	
Lentil seed	0.01	97, 98, 101, 110	102 (5.8)	
	0.10	92. 103, 104, 106	101 (6.2)	
	Combined		101 (5.6)	
Dry bean seed	0.01	93, 101, 104, 106, 106	102 (5.3)	
	0.10	96, 100, 102, 103, 105	101 (3.4)	
	Combined		102 (4.2)	
Dry pea seed	0.01	99, 99, 103, 104, 104	102 (2.5)	Sagan, 2017-b (TK0256751)
	0.10	97, 99, 102, 104, 105	101 (3.3)	
	Combined		102 (2.7)	

Additional method verification recovery data was generated prior to analysis of the field samples from the pulse trials.

Table 2 Method verification recovery data for pulse seed matrices

Matrix	Fortification level (ppm)	Individual recoveries (%)	Mean (%RSD)	Reference
Chickpea seed	0.01	92, 95, 96	94 (2.2)	Sagan, 2017-a (TK0225767)
	0.10	91, 92, 97	93 (3.4)	
	3.0	96, 97, 102	98 (3.3)	
Lentil seed	0.01	92, 98, 100	97 (4.3)	

Matrix	Fortification level (ppm)	Individual recoveries (%)	Mean (%RSD)	Reference
	0.10	94, 95, 98	96 (2.2)	
	3.0	96, 97, 98	97 (1.0)	
Dry bean seed	0.01	94, 95, 103	97 (5.1)	
	0.10	97, 99, 100	99 (1.5)	
	3.0	99, 100, 100	100 (0.6)	
Dry pea seed	0.01	73, 84, 88	82 (9.5)	Sagan, 2017-b (TK0256751)
	0.10	102, 103, 107	104 (2.5)	
	0.30	99, 103, 104	102 (2.6)	

The individual recoveries in pulse seeds ranged from 73–110% and are acceptable.

Recovery data generated for the cereal matrices in conjunction with the Australian residue trials are tabulated below.

Table 3 Recovery data from cereal matrices

Matrix	Fortification level (ppm)	Individual recoveries (%)	Mean (%RSD)	Reference
Barley grain	0.01	95, 100, 100, 107, 107, 107	103 (5.1)	Keats, 2017 (AKC-1601)
	1.0	80, 84, 97	87 (10.2)	
	Combined		97 (10.2)	
Barley straw	0.01	96, 97, 98, 100, 104, 106	100 (4.1)	
	2.5	88, 92, 108	96 (10.8)	
	Combined		99 (6.5)	
Barley forage	0.01	80, 94, 105, 107, 108, 109	101 (11)	Addison, 2016 (SYNGENTACROP/ GLP/13/83-1)
	5	101, 101, 102, 102, 103, 104	102 (1.4)	
	Combined		101 (7.7)	
Barley grain	0.01	101, 105, 106, 106, 109, 113	107 (3.7)	
	5	76, 82, 89, 92, 95, 102	89 (10)	
	Combined		98 (12)	
Barley straw	0.01	100, 103, 105, 106, 109, 110	105 (3.5)	
	5	83, 98, 101, 101, 102, 102	98 (7.6)	
	Combined		102 (6.7)	
Oat forage	0.01	89, 96, 99, 100, 100, 102	98 (4.7)	
	5	93, 95, 104, 105, 106, 109	102 (6.2)	
	Combined		100 (5.8)	
Oat grain	0.01	72, 74, 77, 79, 79, 81	77 (4.6)	
	5	92, 99, 102, 104, 109, 110	103 (6.4)	
	Combined		90 (16)	
Oat straw	0.01	75, 75, 79, 80, 81, 84	79 (4.6)	
	5	92, 103, 103, 103, 105, 106	102 (5.0)	
	Combined		90 (14)	
Wheat forage	0.01	79, 85, 93, 100, 102, 103	94 (10)	
	5	86, 103, 103, 104, 104, 108	101 (7.8)	
	Combined		97 (9.6)	
Wheat grain	0.01	77, 77, 83, 91, 94, 100	87 (11)	
	5	72, 72, 101, 103, 104, 108	93 (18)	
	Combined		90 (15)	
Wheat straw	0.01	74, 81, 82, 88, 90, 93	85 (8.2)	
	5	102, 103, 105, 106, 107	105 (1.9)	
	Combined		95 (12)	

Individual recoveries for diquat from cereal forage, straw and grain matrices ranged from 72–110%. Mean recoveries ranged from 77–107%, and all RSD values were <20%.

Stability of pesticide residues in stored analytical samples

The final version of a storage stability study presented as the interim version to the 2013 JMPR was submitted to the Meeting. This study (Langridge, 2013) determined the stability of diquat in spinach, wheat grain, oilseed rape seed, lentil, orange, potato and wheat straw under freezer storage conditions for up to 24 months. These include representatives of the four crop types, predominantly water-, oil-, protein- and starch-containing materials. Crop samples (10 g of spinach, wheat grain, oilseed rape seed, lentil, whole orange, potato and wheat straw) were separately weighed into polypropylene extraction vessels and fortified with known amounts of a standard solution of diquat in water at a target rate of 0.2 mg/kg. The fortified samples were sealed (after allowing any solvent to evaporate) and gently shaken to distribute the analyte before being stored in a temperature monitored freezer at < -18 °C. Method GRM012.03A was used to determine diquat in the crop commodities.

There was no significant decrease (> 30% as compared to the initial value) in the observed residues of diquat in spinach, wheat grain, oilseed rape seed, lentil, whole orange, potato and wheat straw when stored deep frozen at < -18 °C for a period of at least 24 months.

Table 4 Storage stability results for samples spiked with diquat at 0.2 mg/kg

Commodity	Storage Period (months)	Concentration (mg/kg)	Mean Procedural Recovery (%) [a]
Spinach	0	0.22, 0.21, 0.22	107
	3	0.18, 0.18	98
	6	0.21, 0.20	94
	12	0.24, 0.22	116
	18	0.19, 0.21	105
	24	0.17, 0.18	91
Wheat grain	0	0.22, 0.22, 0.20	104
	3	0.19, 0.20	95
	6	0.19, 0.19	94
	12	0.19, 0.23	109
	18	0.18, 0.19	88
	24	0.19, 0.18	97
Wheat straw	0	0.21, 0.21, 0.21	110
	3	0.18, 0.17	85
	6	0.18, 0.19	100
	12	0.23, 0.22	110
	18	0.19, 0.17	97
	24	0.18, 0.17	91
Rape seed	0	0.21, 0.21, 0.22	104
	3	0.23, 0.25	107
	6	0.20, 0.19	92
	12	0.23, 0.22	109
	18	0.21, 0.19	105
	24	0.15, 0.15	69
Lentils	0	0.21, 0.23, 0.22	104
	3	0.14, 0.15	70
	6	0.17, 0.17	80
	12	0.18, 0.19	90
	18	0.17, 0.19	94
	24	0.16, 0.15	69
Orange fruit	0	0.21, 0.21, 0.21	107
	3	0.18, 0.18	95
	6	0.20, 0.20	103
	12	0.22, 0.22	114
	18	0.19, 0.19	86
	24	0.17, 0.17	80
Potato tubers	0	0.22, 0.19, 0.20	100
	3	0.18, 0.21	92
	6	0.20, 0.21	103
	12	0.17, 0.19	78
	18	0.19, 0.20	98
	24	0.20, 0.19	89

a Mean of two recoveries

Additional storage stability data was generated concurrently with the Australian cereal residue trials. Untreated control samples of forage, grain or straw were homogenised and fortified with diquat dibromide at a level of 0.1 mg/kg (as diquat cation), stored frozen under the same conditions as the field samples, and analysed during the same 48-hour period as the analyses of the field samples.

Table 5 Storage stability data for cereal matrices (generated concurrently with the field trial results)

Matrix	Storage time (months)	Recoveries (%)	Reference
Barley grain	2.3	87, 89	Keats, 2017 (AKC-1601)
	8.3	84, 84	
Barley straw	2.3	90, 91	
	8.3	86, 87	
Barley grain	7.5	85, 87, 87, 88, 88 (mean = 87)	Addison, 2016 (SYNGENTACROP/ GLP/13/83-1)
Barley forage	7.5	84, 85, 87, 88, 90 (mean = 87)	
Barley straw	7.5	87, 87, 88, 89, 89 (mean = 88)	
Oat grain	8.0	75, 90, 92, 92, 93 (88)	
Oat forage	8.0	79, 81, 82, 85, 89 (mean = 83)	
Oat straw	8.0	86, 87, 89, 90, 94 (mean = 89)	
Wheat grain	8.2	75, 98, 98, 100, 100 (mean = 94)	
Wheat forage	8.2	87, 94, 96, 97, 97 (mean = 94)	
Wheat straw	8.2	87, 94, 95, 98, 100 (mean = 95)	

Recoveries after storage from samples fortified during the cereal residue studies are within the range 75–100%, and are consistent with the results from the main storage stability study.

USE PATTERN

Information on registered uses made available to this Meeting are shown in Table 2.

Table 6 Registered uses of diquat in pulses and cereals

Crop	Country	Formulation		Application				PHI [days]
		g ai/L[a]	Type	Method	Timing [Interval – days]	Rate [g ai/ ha][b]	Season Max. [g ai/ ha/year] or (no. per crop)	
Pulses – pre-harvest desiccation uses								
Beans – white and red kidney, soya beans and adzuki beans	Canada	240	SL	Foliar (ground application)	-	408	408 (1)	*
	Canada	240	SL	Foliar (aerial application)	-	552	552 (1)	*
Chickpeas	Canada	240	SL	Foliar (ground application)	-	300–408	408 (1)	*
	Canada	240	SL	Foliar (aerial application)	-	408	408 (1)	*
Lentils	Canada	240	SL	Foliar (ground application)	-	408	408 (1)	*
	Canada	240	SL	Foliar (aerial application)	-	552	552 (1)	*
Peas, dry	Canada	240	SL	Foliar (ground application)	-	408	408 (1)	*
	Canada	240	SL	Foliar (aerial application)	-	552	552 (1)	*
Faba beans	Canada	240	SL	Foliar (ground application)	-	300–408	408 (1)	4
	Canada	240	SL	Foliar (aerial application)	-	408–552	552 (1)	4
Beans – white and red kidney, soya beans and adzuki beans	Canada	200	SL	Foliar (ground application)	-	408	408 (1)	*
	Canada	200	SL	Foliar (aerial application)	-	552	552 (1)	*
Chickpeas	Canada	200	SL	Foliar (ground application)	-	300–408	408 (1)	*

Crop	Country	Formulation		Application				PHI [days]
		g ai/L[a]	Type	Method	Timing [Interval – days]	Rate [g ai/ ha][b]	Season Max. [g ai/ ha/year] or (no. per crop)	
	Canada	200	SL	Foliar (aerial application)	-	408	408 (1)	*
Lentils	Canada	200	SL	Foliar (ground application)	-	408	408 (1)	*
	Canada	200	SL	Foliar (aerial application)	-	552	552 (1)	*
Peas, dry	Canada	200	SL	Foliar (ground application)	-	408	408 (1)	*
	Canada	200	SL	Foliar (aerial application)	-	552	552 (1)	*
Faba beans	Canada	240	SL	Foliar (ground application)	-	300–408	408 (1)	4
	Canada	240	SL	Foliar (aerial application)	-	408–552	552 (1)	4
Dry beans Dry peas Mung beans	Australia	200	SL	Ground boom or aerial	Harvest maturity (BBCH 89)	400–600 + 200 mL/100 L Agral or 160 mL/100 L BS 1000 wetting agent	400–600 (1)	G: 1 H: NR
Lentils Chickpeas Faba beans	Australia	200	SL	Ground boom or aerial	Harvest maturity (BBCH 89)	400–600 + 200 mL/100 L Agral or 160 mL/100 L BS 1000 wetting agent	400–600 (1)	G: 1 H: 2
Pigeon peas Soya beans	Australia	200	SL	Ground boom or aerial	Harvest maturity (BBCH 89)	400–600 + 200 mL/100 L Agral or 160 mL/100 L BS 1000 wetting agent	400–600 (1)	G: 1 H: 4
Cereal grains								
Early season weed control uses								
Oats	Australia	200	SL	Ground boom or aerial	BBCH 13–22	140	140 (1)	G: 1 H: NR
Wheat	Australia	200	SL	Ground boom or aerial	BBCH 14–22	140	140 (1)	G: 1 H: NR
Pre-harvest weed control								
Winter cereals (including barley, oats and wheat)	Australia	200	SL	Ground boom or aerial	Harvest maturity (BBCH 89)	200–600 + 200 mL/100 L Agral or 160 mL/100 L BS 1000 wetting agent	200–600 (1)	G: 1 H: NR
Wheat	Australia	200	SL	Ground boom or aerial	Harvest maturity (BBCH 89)	200–600 + 200 mL/100 L Agral or 160 mL/100 L BS 1000 wetting agent	400–600 (1)	G: 1 H: NR

Crop	Country	Formulation		Application				PHI [days]
		g ai/L[a]	Type	Method	Timing [Interval – days]	Rate [g ai/ ha][b]	Season Max. [g ai/ ha/year] or (no. per crop)	
Quinoa (permit)	Australia	200	SL	Ground boom or aerial	Harvest maturity (BBCH 89)	300–600 + 200 mL/100 L Agral or 160 mL/100 L BS 1000 wetting agent	300–600 (1)	G: 1 H: 4

[a] g ai/L diquat present as diquat dibromide

[b] g ai/ha diquat

NR = not required when used as directed.

*Label instruction states that 'harvesting of most crops can commence within 4–10 days after desiccation application'.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised trials for the uses of diquat dibromide on pulses (dry peas, dry beans, chickpeas and lentils) and cereals (wheat, barley and oats).

Trials were well documented with laboratory and field reports. The former included method validation including recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Samples were collected and stored frozen immediately or soon after sampling. Residue values have not been adjusted for recoveries.

Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and dietary risk assessment and are underlined. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in the estimation of maximum residue levels and dietary risk assessment.

For multiple trials on a crop from the same location, the result from the trial yielding the highest residue was utilised for maximum residue level estimation and dietary risk assessment. In this case the trials are separated with a dotted line.

Some relevant field trial data in beans and peas, dry were provided to the 2013 JMPR, and are reproduced below for convenience (Tables 8, 9, 10, 14 and 15).

Group	Commodity	Country/ Countries	Table No.
VD Pulses	Beans, dry	Canada	Table 7, Table 8, Table 9, Table 10
	Chickpeas	Canada	Table 11
	Lentils	Canada	Table 12
	Peas, dry	Canada	Table 13, Table 14, Table 15
GC Cereal grains	Barley	Australia	Table 16, Table 17
	Oats	Australia	Table 18, Table 19
	Wheat	Australia	Table 20, Table 21
Animal feeds	Barley forage	Australia	Table 22
	Oat forage	Australia	Table 23
	Wheat forage	Australia	Table 24
	Barley straw	Australia	Table 25, Table 26
	Oat straw	Australia	Table 27, Table 28
	Wheat straw	Australia	Table 29, Table 30

Pulses

Trials were conducted in Canada in chickpeas, lentils, beans (dry), and peas, dry for the use of diquat as a pre-harvest desiccant (Sagan, 2017-a and Sagan, 2017-b). One foliar broadcast ground application of a 200 g/L (diquat as the dibromide) soluble concentrate formulation was made at each site at a target rate of 500 g ai/ha, in spray volumes of ~300 or ~45 L/ha. Duplicated treated and single untreated control samples of seed were taken at a target interval of 4 days after application, with the exception of two sites for each of the crops which were run as decline trials, and there additional samples (one treated and one control) were collected at 1, 4, 7, and 10-day target intervals after application. Samples of seed were at least 500 g, and mostly >1.0 kg. Samples were frozen on the day of collection and kept frozen (<-10 ºC) during storage, transport, and until analysis (maximum 10 months storage, thus within the 24-month interval verified as stable by the storage stability study for a range of plant matrices).

Samples were analysed using LC-MS/MS (method GRM012.03A). Concurrent recovery data is summarised above in the analytical methods section.

Results for the trials presented to the present Meeting are tabulated below. Except where noted otherwise, results in all untreated control samples were <LOQ.

Additional trial data in beans (dry), peas, dry and soya bean (dry) were considered by the 2013 JMPR. The residue trial data are reproduced below from the 2013 evaluation for convenience.

Beans, dry

Table 7 Residues of diquat cation in dry bean seed after a single foliar ground application close to harvest (Canada, 2015)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Elm Creek, MB, 2015 (T9905)	85–86	481	300	5	0.18 (0.14, 0.21)	Sagan, 2017-a, T759
St-Marc-sur-Richelieu, QC, Canada, 2015 (Etna)	79–87	526	400	1	0.012	Sagan, 2017-a, T760-D
				4	<0.01 (<0.01, <0.01)	
				7	0.01	
				11	<0.01	
Branchton, ON, Canada, 2015 (Zorro)	88–89	520	300	4	0.012	Sagan, 2017-a, T761
Plattsville, ON, Canada, 2015 (Fathom)	88–89	486	300	1	0.01	Sagan, 2017-a, T762-D
				4	0.012 (0.012, 0.011)	
				7	0.019	
				10	0.012	
Moon Lake, SK, Canada, 2015 (Snowdrop)	85–86	499	45	5	0.35 (0.52, 0.18)	Sagan, 2017-a, T763
Taber, AB, Canada, 2015 (AC Island)	88–89	496	300	5	0.070 (0.081, 0.059)	Sagan, 2017-a, T764
Taber, AB, Canada, 2015 (AC Polaris)	87–89	502	300	5	0.18 (0.14, 0.22)	Sagan, 2017-a, T767
Delisle, SK, Canada, 2015 (Snowdrop)	86–88	491	300	4	0.040 (0.030, 0.051)	Sagan, 2017-a, T765
Hanley, SK, Canada, 2015 (Windbreaker)	84–85	498	300	5	0.044 (0.039, 0.050)	Sagan, 2017-a, T766
Hague, SK, Canada, 2015 (T9905)	84–86	510	300	4	0.15 (0.13, 0.17)	Sagan, 2017-a, T768

Except where noted otherwise, no residues above LOQ were found in the untreated control samples.

Table 8 Residues of diquat in beans dry (pre-harvest desiccation) – German trials reproduced from 2013 JMPR

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (days)	Diquat (mg/kg)	Reference
Oldenburg Germany 1984 Hara	1	0.60	600	PH	Seed	5 8 13	< 0.02 < 0.02 < 0.02	PP901/0312
Mörstadt Germany 1984 Kristal	1	0.60	600	PH	Seed	4	< 0.02	PP901/0312
Rheinhessen Germany 1984 Kristal	1	0.60	600	PH	Seed	5 7	< 0.02 < 0.02	PP901/0312
Wankendorf Germany 1984 Hara	1	0.60	600	PH	Seed	5 7 9	< 0.02 < 0.02 0.03	PP901/0312
Wankendorf Germany 1984 Hara	1	0.60	600	BBCH 88	seed	5 7 11	0.08 0.07 0.09	PP901/0312
Lüneburg Germany 1985 Hara	1	0.60	600	BBCH 88	seed	5 7 10	0.15 0.09 0.14	PP901/0312
Bottenbach Germany 1985 Kristall	1	0.60	600	BBCH 86–88	seed	3 5 8	< 0.02 0.03 0.05	PP901/0312
Kappellen-Drusweiler Germany 1985	1	0.60	600	BBCH 88	seed	3 5 7	0.04 0.08 0.06	PP901/0312

Table 9 Residues of diquat in bean (pre-harvest desiccation) – US trials reproduced from 2013 JMPR

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (days)	Diquat (mg/kg)	Reference
Visalia CA USA 1994 Greencrop	1	0.42	91	PH crop fully mature, pods drying	seed	4	< 0.05	PP901/0325
Ault CO USA 1994 Bill Z	1	0.42	93	PH crop mature	seed	4	< 0.05	PP901/0325
Jerome ID USA 1994 Pinto Vofi 196	1	0.42	76	PH crop mature	seed	4	< 0.05	PP901/0325
Bridgeport MI USA 1994 Blackhawk	1	0.42	92	PH crop mature	seed	4	< 0.05 [a]	PP901/0325
Hutchinson MN USA 1994 Montcalm	1	0.42	89	PH crop mature, pods yellow	seed	4	< 0.05	PP901/0325
Madrid NE USA 1994 Vaccaro	1	0.42	93	PH crop mature	seed	4	< 0.05	PP901/0325
Fabius NY USA 1994 Light Red Kidney	1	0.42	93	PH crop mature	seed	4	< 0.05	PP901/0325
Northwood ND USA 1994 Norstar	1	0.42	94	PH crop near maturity, 70% defoliated	seed	4	< 0.05	PP901/0325

[a] Bridgeport MI USA site received 18 mm rain on the day after application

Table 10 Residues in soya beans (pre-harvest desiccation) – French trials reproduced from 2013 JMPR

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	Diquat (mg/kg)	Reference
Villemur France 1985 Sloan	1	0.60	300	Yellow leaves	Seed	0 2 5 6 8	0.63 0.37 < 0.1 < 0.1 < 0.1	PP901/0310
	1	0.80	300	Yellow leaves	Seed	0 2 5 6 8	0.91 0.21 < 0.1 < 0.1 < 0.1	

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	Diquat (mg/kg)	Reference
	1	0.60	300	Yellow leaves	Seed	0 2 5 6 8	0.62 < 0.1 < 0.1 < 0.1 < 0.1	
	1	0.80	300	Yellow leaves	Seed	0 2 5 6 8	0.59 < 0.1 < 0.1 < 0.1 < 0.1	
Avignon France 1994 Goldor	1	0.60		Mature	Oil Cake Seed	4	< 0.05 0.06 0.06 [a]	PP901/0328
Blois France 1994 Maple Arrow	1	0.60		Mature	Oil Cake Seed	4	< 0.05 < 0.05 < 0.05[A]	PP901/0328

[a] Calculated residue (from separate oil and meal determinations)

Chickpeas

Table 11 Residues of diquat cation in chickpea seed after a single foliar ground application close to harvest (Canada, 2015)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		
Elm Creek, MB, Canada, 2015 (CDC Anna)	85–85	479	300	1	0.20	Sagan, 2017-a, T740-D
				5	0.16 (0.17, 0.16) c0.012	
				7	0.089 c0.014	
				9	0.15 c0.041	
Delisle, SK, Canada, 2015 (CDC Frontier)	79–83	468	45	4	0.18 (0.14, 0.21)	Sagan, 2017-a, T741
Vanscoy, SK, Canada, 2015, (CDC Anna)	87–88	518	300	4	0.58 (0.50, 0.65) c0.01	Sagan, 2017-a, T742
Zealandia, SK, Canada, 2015 (CDC Frontier)	79–84	507	300	4	0.38 (0.39, 0.38)	Sagan, 2017-a, T743
Pike Lake, SK, Canada, 2015 (CDC Anna)	81–86	527	300	1	0.79	Sagan, 2017-a, T744-D
				4	0.23 (0.27, 0.19)	
				7	0.20 c0.045	
				10	0.26 c0.026	
Hanley, SK, Canada, 2015 (CDC Orion)	84–85	492	300	5	0.10 (0.10, 0.11)	Sagan, 2017-a, T745
Taber, AB, Canada, 2015 (CDC Anna)	87–89	486	300	5	0.24 (0.31, 0.17)	Sagan, 2017-a, T746
Glenboro, MB, Canada, 2015 (CDC Frontier	79–81	474	300	5	0.32 (0.30, 0.33)	Sagan, 2017-a, T747

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days		
Avonlea, SK, Canada, 2015 (CDC Consul)	82–84	508	300	4	0.070 (0.065, 0.075)	Sagan, 2017-a, T748

Except where noted otherwise, no residues above LOQ were found in the untreated control samples.

Lentils

Table 12 Residues of diquat cation in lentil seed after a single foliar ground application close to harvest (Canada, 2015)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Delisle, SK, Canada, 2015 (CDC Dazil)	81–83	475	300	1	0.27	Sagan, 2017-a, T750-D
				4	0.21 (0.21, 0.21)	
				8	0.19	
				10	0.099	
Vanscoy, SK, Canada, 2015 (CDC Invincible)	83–84	493	45	1	0.54	Sagan, 2017-a, T751-D
				4	0.50 (0.51, 0.49)	
				7	0.44 c0.01	
				10	0.57	
Zealandia, SK, Canada, 2015 (CDC Dazil)	83–85	503	300	4	0.18 (0.13, 0.24)	Sagan, 2017-a, T752
Perdue, SK, Canada, 2015 (Maxim)	82–88	480	300	5	0.070 (0.058, 0.083)	Sagan, 2017-a, T753
Hanley, SK, Canada, 2015 (CDC Invincible)	81–85	501	300	5	0.052 (0.044, 0.059)	Sagan, 2017-a, T754
Broderick, SK, Canada, 2015 (CDC Invincible)	75–81	501	300	4	0.33 (0.26, 0.40) c0.028	Sagan, 2017-a, T755
Glenboro, MB, Canada, 2015 (CDC Invincible)	83–84	470	300	5	0.10 (0.089, 0.12)	Sagan, 2017-a, T756
Hepburn, SK, Canada, 2015 (CDC Invincible)	81–82	474	300	5	0.16 (0.14, 0.18)	Sagan, 2017-a, T757

Except where noted otherwise, no residues above LOQ were found in the untreated control samples.

Peas, dry

Table 13 Residues of diquat cation in dry pea seed after a single foliar ground application close to harvest (Canada, 2015)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Zelandia, SK, Canada, 2015 (CDC Amarillo)	88–89	488	250	1	0.12 c0.013	Sagan, 2017-b, T769-D
				4	0.13 (0.080, 0.18)	
				7	0.032	
				11	0.035	
Delisle, SK, Canada, 2015 (CDC Amarillo)	87–88	479	250	1	0.10 c0.052	Sagan, 2017-b, T770-D

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
				3	0.054 (0.043, 0.064) c0.012	
				7	0.042	
				10	0.034	
Dundurn, SK, Canada, 2015 (CDC Meadow)	86–87	490	250	4	0.020 (0.020, 0.019)	Sagan, 2017-b, T771
Moon Lake, SK, Canada, 2015 (CDC Amarillo)	79–87	513	250	4	0.054 (0.062, 0.045)	Sagan, 2017-b, T772
Minto, MB, Canada, 2015 (Croma)	88–89	514	320	5	0.014 (0.015, 0.014)	Sagan, 2017-b, T773
Blaine Lake, SK, Canada, 2015 (CDC Amarillo)	86–87	510	250	5	0.038 (0.037, 0.039)	Sagan, 2017-b, T774
Hague, SK, Canada, 2015 (CDC Amarillo)	82–84	495	250	4	0.036 (0.045, 0.028) c0.014	Sagan, 2017-b, T775
Glenboro, MB, Canada, 2015 (CDC Meadow)	87–88	480	300	3	0.061 (0.089, 0.033)	Sagan, 2017-b, T776

Except where noted otherwise, no residues above LOQ were found in the untreated control samples.

Table 14 Residues of diquat in peas dry (pre-harvest desiccation) – reproduced from 2013 JMPR evaluation

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (days)	Diquat (mg/kg)	Reference
Wankendorf Germany 1984	1	0.60	600	PH	seed	4 7 10	0.05 0.03 0.05	PP901/0311
Neustadt/Holst Germany 1984	1	0.60	600	PH	seed	5 7 11	< 0.02 0.03 < 0.02	PP901/0311
Morstadt bei Worms Germany 1984 Stehgold	1	0.60	600	PH	seed	5 7 12	0.10 0.10 0.07	PP901/0311
Bröthen/Büchen Germany 1985 Columba	1	0.60	600	PH	seed	5 7 9	0.06 0.04 0.04	PP901/0311
Neustadt/Holst Germany 1985 Birte	1	0.60	600	PH	seed	5 7 10	0.05 0.04 0.04	PP901/0311
Dierbach Germany 1985 Stehgolt	1	0.60	600	PH	seed	3 5 7	0.07 0.06 0.06	PP901/0311
Kapellen-Drusweiler Germany 1985 Bodil	1	0.60	600	PH	seed	3 6 8	0.12 0.13 0.15	PP901/0311
Oberndorf-Hochmössingen Germany 1985 Stehgold	1	0.60	600	PH	Seed	7	0.04	PP901/0311
Slagelse Denmark 1986 Bodil (app 28/7)	1	0.60	400	yellowing	seed	0 3 7 9 14	0.10 0.03 0.04 0.05 0.05	PP901/0315 Day 0 seed 54% moisture
Slagelse Denmark 1986 Bodil (app 6/8)	1	0.60	400	yellowing	seed	0 3 7 9 13	0.03 < 0.02 < 0.02 < 0.02 < 0.02	PP901/0315 Day 0 seed 32% moisture

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (days)	Diquat (mg/kg)	Reference
Slagelse Denmark 1986 Bodil (app 8/8)	1	0.60	400	yellowing	seed	0 3 7 11 14	0.02 < 0.02 < 0.02 < 0.02 < 0.02	PP901/0315 Day 0 seed 14% moisture
Olstykke Denmark 1986 Bodil (app 5/8)	1	0.60	400	yellowing	seed	0 3 7 10 14	0.09 0.03 0.02 0.03 0.03	PP901/0315 Day 0 seed 51% moisture
Olstykke Denmark 1986 Bodil (app 8/8) unclear not two apps? Subsampled for analysis	1	0.60	400	yellowing	seed	0 3 7 10 14	0.09 < 0.02 < 0.02 < 0.02 < 0.02	PP901/0315 Day 0 seed 25% moisture
Dorrington Lincolnshire UK 1990 Helka	1	0.53	300	PGRO/Knott 301–302	seed	5	0.04	PP901/0319 + Agral
Warwickshire UK 1990 Princess	1	0.53	300	PGRO/Knott 301–303	seed	10	0.04	PP901/0319 + Agral
Icklingham East Anglia UK 1990 Solara	1	0.53	300	PGRO/Knott 301–302	seed	8	< 0.03	PP901/0319 + Agral
Dorrington Lincolnshire UK 1992 Baroness	1	0.26	200	PGRO/Knott 301–302	seed	4	< 0.05	PP901/0322 + Agral
	1	0.53	200	PGRO/Knott 301–302	seed	4	< 0.05	
	1	0.53	200	PGRO/Knott 301–302	seed	4	< 0.05	+ Agral
Welton Cuff Lincolnshire UK 1992 Progreta	1	0.26	200	PGRO/Knott 301–302	seed	4	< 0.05	PP901/0322 + Agral
	1	0.53	200	PGRO/Knott 301–302	seed	4	< 0.05	
	1	0.53	200	PGRO/Knott 301–302	seed	4	< 0.05	+ Agral
Sandy Gate Lincolnshire UK 1992 Princess	1	0.26	200	PGRO/Knott 301–302	seed	4	< 0.05	PP901/0322 + Agral
	1	0.53	200	PGRO/Knott 301–302	seed	4	< 0.05	
	1	0.53	200	PGRO/Knott 301–302	seed	4	< 0.05	+ Agral

Table `5 Residues of diquat in peas dry (pre-harvest desiccation) – reproduced from 2013 JMPR evaluation

Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (days)	Diquat (mg/kg)	Reference
Visalia CA USA 1994 Progress #9	1	0.42	90	Mature	Seed	4	0.05	PP901/0324 + NIS
Nezperce ID USA 1994 Columbia	1	0.42	93	Mature	Seed	4	0.09	PP901/0324 + NIS
Milton-Freewater OR USA 1994 Columbia	1	0.42	93	Mature	Seed	4	0.56	PP901/0324 + NIS
Mercedes TX USA 1994 Tracer	1	0.42	84	Mature	Seed	4	0.40 c0.06	PP901/0324 + NIS small sample size 0.23 kg
Walla Walla WA USA 1994 Columbia	1	0.42	93	Mature	Seed	4	0.11	PP901/0324 + NIS
Ridgefield WA USA 1994 Yellow Spring	1	0.42	82	Mature	Seed	4	0.05	PP901/0324 + NIS

Cereal grains

A series of four trials for pre-harvest desiccation treatment in barley was conducted in Australia during the 2016/17 growing season (Keats, 2017). At each site, two treated plots and an untreated control plot were established. A single foliar application of a diquat dibromide soluble concentrate formulation (200 g/L diquat ion) was made to the treated plots at or very close to harvest maturity (BBCH 90–92) at target rates of either 600 or 900 g ai/ha, in a spray volume of ~100 L/ha. A spray adjuvant (alcohol alkoxylate) was incorporated in the spray tank at 1.6 mL/L. Single samples of grain and straw (incorrectly referred to as hay in the study) were collected at target intervals of 0, 3 and 7 days after application from the treated plots, and 7 days only from the control plots. Sample sizes were suitable (1 kg for grain and 0.5 kg for straw) and all samples were frozen within 24 hours of collection and stored frozen (≤ -18 ºC) until analysis, with all analyses being completed within 9 months of sample collection, within the 24-month interval verified as stable by the storage stability study for a range of plant matrices. Residues of diquat were determined by LC-MS/MS, based on method number GRM012.03A.

A series of 16 trials in barley, oats, and wheat was conducted in Australia during the 2014/15 growing season (Addison, 2016), all utilising a diquat dibromide soluble concentrate formulation (200 g/L diquat ion). Spray adjuvants were not included in the tank mixes for any applications. Three treated plots and one untreated control plot were established at each trial site. The first treated plot at each site was treated with two applications at target rates of 140 g ai/ha, the first application scheduled for BBCH 24–31 (4 tillers detectable to first node stage) and the second for approximately 14 days later. The second and third treated plots at each site were treated with a single pre-harvest weed control application at target rates of 600 and 900 g ai/ha respectively.

At all trial sites, grain and straw samples were collected from the pre-harvest dessication treatment plots at intervals of 0–7 days after application. For the plots receiving the early season weed control treatment, grain and straw samples were collected at normal commercial harvest (at a single interval of 0 or 1 day after the pre-harvest application was made to the dessication treatment plots). For a number of the sites, forage samples were collected from the early season weed control treatment plots at target intervals ranging from 0 days after the first application to 14 days after the second application. Untreated control samples of grain and straw were collected at all sites at harvest, at a single interval of 0 or 1 day after the pre-harvest dessication application. At trial sites where forage samples were collected, untreated control samples of forage were collected on the day of each of the two early season weed control applications. In all cases, duplicate samples were collected (one primary and one reserve sample). Sample sizes were generally >1 kg for forage and grain and >0.5 kg for straw. Samples were placed in freezer storage immediately on return from the field site (same day) and kept frozen until analysis, with the exception of a single barley straw sample which arrived at the laboratory thawed (noted in the appropriate table). Grain and straw samples were analysed within 9 months of collection, while forage samples were analysed within 12 months of collection, thus within the 24-month interval verified as stable by the storage stability study for a range of plant matrices. Residues of diquat were determined by LC-MS/MS, based on method number GRM012.03A.

Barley

Table 16 Residues of diquat cation in barley grain after a single pre-harvest weed control application (Australia, 2014 and 2016)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Narrabri, NSW 2390, Australia, 2016 (Gairdner)	90–92	606	100	0	3.9	Keats 2017, AKC-1601-01
				3	2.1	
				7	1.1	
	90–92	909	100	0	6.7	
				3	3.0	
				7	2.0	
Red Creek, SA 5255, Australia, 2016 (Commander)	92	606	100	0	3.8	Keats 2017, AKC-1601-02
				3	2.0	
				7	1.5	
	92	909	100	0	5.6	
				3	2.5	
				7	2.0	
Red Creek, SA 5255, Australia, 2016 (Compass)	92	606	100	0	3.5	Keats 2017, AKC-1601-03
				3	1.8	
				7	1.4	

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
	92	909	100	0	6.9	
				3	2.6	
				7	2.1	
York, WA 6302, Australia, 2016 (Baudin)	92–95	630	105	0	3.5	Keats 2017, AKC-1601-04
				2	2.0	
				7	1.3	
	92–95	990	110	0	6.2	
				2	2.5	
				7	1.7	
Meckering, WA , Australia, 2014 (Hindmarsh)	92	622	113	0	3.9	Addison 2016, 140125
				1	1.8	
				2	0.53	
				7	0.34	
	92	1227	112	0	5.4	
				1	1.7	
				2	0.87	
				7	0.51	
Stockport, SA, Australia, 2014 (Fleet)	90	606	99	0	0.68	Addison 2016, 140128
				1	1.1	
				3	0.49	
				7	0.10	
	90	1188	97	0	1.6	
				1	1.2	
				3	0.77	
				7	0.39	
Undera, VIC, Australia, 2014 (Hindmarsh)	90	597	114	0	3.8	Addison 2016, 140131
				1	1.9	
				3	1.1	
				7	0.39	
	90	1227	117	0	6.0	
				1	3.7	
				3	1.7	
				7	0.50	
Cargo, NSW, Australia, 2014 (Hindmarsh)	90	594	89	0	2.1	Addison 2016, 140135
				1	1.4	
				4	0.15	
				7	0.10	
	90	1188	89	0	3.4	
				1	0.91	
				4	0.45	
				7	0.38	

No residues above LOQ were found in the untreated control samples.

Table 17 Residues of diquat cation in barley grain at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Meckering, WA, Australia, 2014 (Hindmarsh)	2 (15)	24, 24–31	140, 140	102, 114	98	0.09	Addison 2016, 140125
Stockport, SA, Australia, 2014 (Fleet)	2 (15)	24, 24–26	140, 140	95, 100	54	0.04	Addison 2016, 140128
Undera, VIC, Australia, 2014 (Hindmarsh)	2 (13)	24, 54	144, 144	113, 113	61	0.10	Addison 2016, 140131
Cargo, NSW, Australia, 2014 (Hindmarsh)	2 (15)	24, 41	144, 144	87, 90	74	0.14	Addison 2016, 140135

No residues above LOQ were found in the untreated control samples.

Oats

Table 18 Residues of diquat cation in oat grain after a single pre-harvest weed control application (Australia, 2014)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
York, WA, Australia, 2014 (Carrdup)	92	622	111	0	2.1	Addison 2016, 140126
				1	1.2	
				2	0.46	
				7	0.23	
	92	1213	110	0	3.1	
				1	2.0	
				2	0.47	
				7	0.27	
Redbank, SA, Australia, 2014 (Winteroo)	90	576	92	0	1.5	Addison 2016, 140129
				1	0.50	
				3	0.26	
				7	0.13	
	90	1176	94	0	2.0	
				1	0.99	
				3	0.46	
				7	0.15	
Shepparton, VIC, Australia, 2014 (Mitikia)	90	606	112	0	1.3	Addison 2016, 140132
				1	1.6	
				2	0.41	
				6	0.30	
	90	1201	111	0	1.4	
				1	4.5	
				2	1.1	
				6	0.15	
Barry, NSW, Australia, 2014 (Nile)	90	579	87	0	1.5	Addison 2016, 140136
				1	0.98	
				4	0.41	
				7	0.16	

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
	90	1176	88	0	3.1	
				1	1.9	
				4	0.71	
				7	0.35	

No residues above LOQ were found in the untreated control samples.

Table 19 Residues of diquat cation in oat grain at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
York, WA, Australia, 2014 (Carrdup)	2 (15)	24, 37–45	142, 142	115, 108	83	0.19	Addison 2016, 140126
Redbank, SA, Australia, 2014 (Winteroo)	2 (14)	24, 24–26	142, 142	98, 99	99	0.13	Addison 2016, 140129
Shepparton, VIC, Australia, 2014 (Mitikia)	2 (14)	24, 30	141, 141	115, 93	115	0.13	Addison 2016, 140132
Barry, NSW, Australia, 2014 (Nile)	2 (14)	24, 25	138. 138	102, 106	103	0.21	Addison 2016, 140136

No residues above LOQ were found in the untreated control samples.

Wheat

Table 20 Residues of diquat cation in wheat grain after a single pre-harvest weed control application (Australia, 2014)

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Beverley, WA , Australia, 2014 (Mace)	90	602	114	0	1.9	Addison 2016, 140123
				1	1.3	
				3	0.56	
				7	0.26	
	90	1207	114	0	3.6	
				1	2.4	
				3	0.90	
				7	0.33	
York, WA, Australia, 2014 (Mace)	92	598	111	0	1.8	Addison 2016, 140124
				1	1.2	
				2	0.57	
				7	0.24	
	92	1192	111	0	3.3	
				1	2.1	
				2	0.81	
				7	0.38	
Kangaroo Flats, SA, Australia, 2014 (Mace)	90	600	98	0	2.7	Addison 2016, 140127
				1	1.5	
				3	0.78	
				7	0.29	
	90	1176	96	0	4.0	
				1	2.6	

Location, Year (variety)	Application				Residues, mg/kg diquat	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
				3	1.0	
				7	0.37	
Cosgrove, VIC, Australia, 2014 (Phantom)	90	613	113	0	2.2	Addison 2016, 140130
				1	1.3	
				2	0.63	
				6	0.21	
	90	1224	113	0	3.9	
				1	1.7	
				2	0.96	
				6	0.29	
Narrabri, NSW, Australia, 2014 (Lancer)	90	594	84	0	2.3	Addison 2016, 140133
				1	0.86	
				3	0.45	
				7	0.17	
	90	1187	84	0	4.4	
				1	2.3	
				3	0.47	
				7	0.25	
Hobby's Yards, NSW, Australia, 2014 (Gregory)	90	612	92	0	2.8	Addison 2016, 140134
				1	0.84	
				4	0.41	
				7	0.19	
	90	1188	89	0	3.6	
				1	1.9	
				4	0.61	
				7	0.26	
Darling Downs, QLD, Australia, 2014 (Gregory)	90	592	111	0	2.1	Addison 2016, 140137
				1	0.86	
				3	0.28	
				7	0.13	
	90	1186	111	0	3.4	
				1	1.8	
				3	0.41	
				7	0.17	
Cambooya, QLD, Australia, 2014 (Gregory)	90	581	81	0	2.1	Addison 2016, 140138
				1	1.2	
				3	0.20	
				8	0.13	
	90	1140	80	0	3.7	
				1	2.7	
				3	0.29	
				8	0.17	

No residues above LOQ were found in the untreated control samples.

Table 21 Residues of diquat cation in wheat grain at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Beverley, WA, Australia, 2014 (Mace)	2 (15)	24, 24–29	143, 143	106, 114	103	0.26	Addison 2016, 140123
York, WA, Australia, 2014 (Mace)	2 (13)	24–31, 37–52	142, 142	108, 110	78	0.29	Addison 2016, 140124
Kangaroo Flats, SA, Australia, 2014 (Mace)	2 (14)	24, 24–26	141, 141	98, 98	82	0.34	Addison 2016, 140127
Cosgrove, VIC, Australia, 2014 (Phantom)	2 (14)	24, 30	141, 141	112, 95	115	0.28	Addison 2016, 140130
Narrabri, NSW, Australia, 2014 (Lancer)	2 (14)	24, 32	138, 138	84, 84	67	0.30	Addison 2016, 140133
Hobby's Yards, NSW, Australia, 2014 (Gregory)	2 (14)	29, 44	139, 139	111, 110	46	0.04	Addison 2016, 140134
Darling Downs, QLD, Australia, 2014 (Gregory)	2 (14)	24, 32	139, 139	87, 79	82	0.22	Addison 2016, 140137
Cambooya, QLD, Australia, 2014 (Gregory)	2 (14)	24, 24–28	136, 136	80, 92	75	0.14	Addison 2016, 140138

No residues above LOQ were found in the untreated control samples.

Animal feeds

Barley forage

Table 22 Residues of diquat cation in barley forage at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI		
Meckering, WA, Australia, 2014 (Hindmarsh)	2 (15)	24, 24–31	140, 140	102, 114	0 DAA1	12	Addison 2016, 140125
					2 DAA1	5.4	
					8 DAA1	2.2	
					15 DAA1	0.53	
					0 DAA2	28	
					1 DAA2	7.6	
					7 DAA2	3.0	
					15 DAA2	0.87	
Cargo, NSW, Australia, 2014 (Hindmarsh)	2 (15)	24, 41	144, 144	87, 90	0 DAA1	7.1	Addison 2016, 140135
					1 DAA1	2.9	
					7 DAA1	0.72	
					15 DAA1	0.18	
					0 DAA2	6.1	
					1 DAA2	1.9	
					7 DAA2	0.41	
					14 DAA2	0.19	

No residues above LOQ were found in the untreated control samples.

Oat forage

Table 23 Residues of diquat cation in oat forage at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI		
York, WA, Australia, 2014 (Carrdup)	2 (15)	24, 37–45	142, 142	115, 108	0 DAA1	16	Addison 2016, 140126
					1 DAA1	13	
					6 DAA1	2.9	
					15 DAA1	0.49	
					0 DAA2	18	
					1 DAA2	14	
					7 DAA2	2.0	
					14 DAA2	0.34	
Barry, NSW, Australia, 2014 (Nile)	2 (14)	24, 25	138. 138	102, 106	0 DAA1	15	Addison 2016, 140136
					1 DAA1	3.6	
					7 DAA1	0.43	
					14 DAA1	0.36	
					0 DAA2	12	
					1 DAA2	3.1	
					7 DAA2	0.25	
					14 DAA2	0.13	

No residues above LOQ were found in the untreated control samples.

Wheat forage

Table 24 Residues of diquat cation in wheat forage at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI		
York, WA, Australia, 2014 (Mace)	2 (13)	24–31, 37–52	142, 142	108, 110	0 DAA1	23	Addison 2016, 140124
					1 DAA1	13	
					8 DAA 1	1.7	
					13 DAA 1	0.40	
					0 DAA 2	18	
					2 DAA2	11	
					6 DAA 2	2.7	
					15 DAA2	0.56	
Kangaroo Flats, SA, Australia, 2014 (Mace)	2 (14)	24, 24–26	141, 141	98, 98	0 DAA1	18	Addison 2016, 140127
					1 DAA1	15	
					7 DAA1	1.4	

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI		
					14 DAA1	0.42	
					0 DAA2	19	
					1 DAA2	13	
					7 DAA2	2.4	
					14 DAA2	0.74	
Hobby's Yards, NSW, Australia, 2014 (Gregory)	2 (14)	29, 44	139, 139	111, 110	0 DAA1	20	Addison 2016, 140134
					1 DAA1	15	
					7 DAA1	1.6	
					14 DAA1	Not collected	
					0 DAA2	19	
					1 DAA2	11	
					7 DAA2	23	
					13 DAA2	0.35	
Cambooya, QLD, Australia, 2014 (Gregory)	2 (14)	24, 24–28	136, 136	80, 92	0 DAA1	15	Addison 2016, 140138
					1 DAA1	10	
					7 DAA1	0.92	
					14 DAA1	0.29	
					0 DAA2	14	
					1 DAA2	11	
					7 DAA2	2.7	
					14 DAA2	0.34	

No residues above LOQ were found in the untreated control samples.

Barley straw

Table 25 Residues of diquat cation in barley straw after a single pre-harvest weed control application (Australia, 2014 and 2016)

Location, Year (variety)	Application				Residues, mg/kg diquat (dry weight basis)	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Narrabri, NSW 2390, Australia, 2016 (Gairdner)	90–92	611	100	0	33	Keats 2017, AKC-1601-01
				3	26	
				7	20	
	90–92	909	100	0	63	
				3	43	
				7	27	
Red Creek, SA 5255, Australia, 2016 (Commander)	92	606	100	0	24	Keats 2017, AKC-1601-02
				3	25	
				7	14	
	92	909	100	0	37	
				3	32	

Location, Year (variety)	Application				Residues, mg/kg diquat (dry weight basis)	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
				7	18	
Red Creek, SA 5255, Australia, 2016 (Compass)	92	606	100	0	20	Keats 2017, AKC-1601-03
				3	17	
				7	9.3	
	92	909	100	0	34	
				3	25	
				7	16	
York, WA 6302, Australia, 2016 (Baudin)	92–95	630	105	0	29	Keats 2017, AKC-1601-04
				2	23	
				7	18	
	92–95	990	110	0	53	
				2	40	
				7	26	
Meckering, WA , Australia, 2014 (Hindmarsh)	92	622	113	0	12	Addison 2016, 140125
				1	5.3	
				2	4.8	
				7	1.7	
	92	1227	112	0	17	
				1	12	
				2	5.6	
				7	2.2	
Stockport, SA, Australia, 2014 (Fleet)	90	606	99	0	6.7	Addison 2016, 140128
				1	9.7	
				3	6.9	
				7	3.5	
	90	1188	97	0	12	
				1	11	
				3	8.8	
				7	4.4	
Undera, VIC, Australia, 2014 (Hindmarsh)	90	597	114	0	12	Addison 2016, 140131
				1	8.0	
				3	6.2	
				7	4.7	
	90	1227	117	0	18	
				1	14[a]	
				3	9.8	
				7	7.4	
Cargo, NSW, Australia, 2014 (Hindmarsh)	90	594	89	0	7.1	Addison 2016, 140135
				1	4.3	
				4	2.8	
				7	2.0	
	90	1188	89	0	11	
				1	7.0	
				4	4.1	
				7	3.6	

No residues above LOQ were found in the untreated control samples.

[a] Sample arrived at the laboratory thawed.

Table 26 Residues of diquat cation in barley straw at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Meckering, WA, Australia, 2014 (Hindmarsh)	2 (15)	24, 24–31	140, 140	102, 114	98	0.21	Addison 2016, 140125
Stockport, SA, Australia, 2014 (Fleet)	2 (15)	24, 24–26	140, 140	95, 100	54	0.12	Addison 2016, 140128
Undera, VIC, Australia, 2014 (Hindmarsh)	2 (13)	24, 54	144, 144	113, 113	61	0.24	Addison 2016, 140131
Cargo, NSW, Australia, 2014 (Hindmarsh)	2 (15)	24, 41	144, 144	87, 90	74	0.09	Addison 2016, 140135

No residues above LOQ were found in the untreated control samples.

Oat straw

Table 27 Residues of diquat cation in oat straw after a single pre-harvest weed control application (Australia, 2014)

Location, Year (variety)	Application				Residues, mg/kg diquat (dry weight basis)	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
York, WA , Australia, 2014 (Carrdup)	92	622	111	0	4.5	Addison 2016, 140126
				1	2.6	
				2	1.8	
				7	1.3	
	92	1213	110	0	7.3	
				1	5.1	
				2	3.0	
				7	2.3	
Redbank, SA, Australia, 2014 (Winteroo)	90	576	92	0	7.7	Addison 2016, 140129
				1	7.8	
				3	2.8	
				7	1.2	
	90	1176	94	0	13	
				1	11	
				3	5.8	
				7	6.0	
Shepparton, VIC, Australia, 2014 (Mitikia)	90	606	112	0	12	Addison 2016, 140132
				1	4.4	
				2	3.1	
				6	2.9	
	90	1201	111	0	18	
				1	6.4	
				2	5.0	
				6	4.2	
Barry, NSW, Australia, 2014 (Nile)	90	579	87	0	2.8	Addison 2016, 140136
				1	0.96	
				4	0.27	
				7	0.37	

Location, Year (variety)	Application				Residues, mg/kg diquat (dry weight basis)	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
	90	1176	88	0	3.9	
				1	1.3	
				4	0.68	
				7	0.56	

No residues above LOQ were found in the untreated control samples.

Table 28 Residues of diquat cation in oat straw at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
York, WA , Australia, 2014 (Carrdup)	2 (15)	24, 37–45	142, 142	115, 108	83	0.40	Addison 2016, 140126
Redbank, SA, Australia, 2014 (Winteroo)	2 (14)	24, 24–26	142, 142	98, 99	99	0.74	Addison 2016, 140129
Shepparton, VIC, Australia, 2014 (Mitikia)	2 (14)	24, 30	141, 141	115, 93	115	0.85	Addison 2016, 140132
Barry, NSW, Australia, 2014 (Nile)	2 (14)	24, 25	138. 138	102, 106	103	0.36	Addison 2016, 140136

No residues above LOQ were found in the untreated control samples.

Wheat straw

Table 29 Residues of diquat cation in wheat straw after a single pre-harvest weed control application (Australia, 2014)

Location, Year (variety)	Application				Residues, mg/kg diquat (dry weight basis)	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Beverley, WA, Australia, 2014 (Mace)	90	602	114	0	5.6	Addison 2016, 140123
				1	2.8	
				3	2.0	
				7	2.0	
	90	1207	114	0	8.5	
				1	6.0	
				3	3.7	
				7	3.2	
York, WA, Australia, 2014 (Mace)	92	598	111	0	5.9	Addison 2016, 140124
				1	3.6	
				2	2.8	
				7	1.6	
	92	1192	111	0	9.3	
				1	6.6	
				2	4.4	
				7	3.2	
Kangaroo Flats, SA, Australia, 2014 (Mace)	90	600	98	0	8.5	Addison 2016, 140127
				1	4.4	

Location, Year (variety)	Application				Residues, mg/kg diquat (dry weight basis)	Reference
	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
				3	6.1	
				7	4.6	
	90	1176	96	0	13	
				1	12	
				3	9.0	
				7	6.8	
Cosgrove, VIC, Australia, 2014 (Phantom)	90	613	113	0	5.2	Addison 2016, 140130
				1	7.1	
				2	5.6	
				6	3.9	
	90	1224	113	0	8.2	
				1	10	
				2	6.7	
				6	5.8	
Narrabri, NSW, Australia, 2014 (Lancer)	90	594	84	0	6.3	Addison 2016, 140133
				1	5.1	
				3	3.3	
				7	1.8	
	90	1187	84	0	7.3	
				1	8.1	
				3	4.9	
				7	2.8	
Hobby's Yards, NSW, Australia, 2014 (Gregory)	90	612	92	0	4.9	Addison 2016, 140134
				1	3.5	
				4	2.4	
				7	0.84	
	90	1188	89	0	8.1	
				1	5.8	
				4	3.8	
				7	1.3	
Darling Downs, QLD, Australia, 2014 (Gregory)	90	592	111	0	5.7	Addison 2016, 140137
				1	5.2	
				3	4.3	
				7	2.2	
	90	1186	111	0	9.3	
				1	7.5	
				3	6.0	
				7	3.0	
Cambooya, QLD, Australia, 2014 (Gregory)	90	581	81	0	7.6	Addison 2016, 140138
				1	1.8	
				3	1.2	
				8	1.2	
	90	1140	80	0	11	
				1	4.8	
				3	2.0	
				8	1.7	

No residues above LOQ were found in the untreated control samples.

Table 30 Residues of diquat cation in wheat straw at harvest after early season applications (Australia, 2014)

Location, Year (variety)	Application					Residues, mg/kg diquat (dry weight basis)	Reference
	n (RTI, days)	Growth stage (BBCH)	Rate, g ai/ha	Spray volume (L/ha)	PHI, days		
Beverley, WA, Australia, 2014 (Mace)	2 (15)	24, 24–29	143, 143	106, 114	103	0.48	Addison 2016, 140123
York, WA, Australia, 2014 (Mace)	2 (13)	24–31, 37–52	142, 142	108, 110	78	0.57	Addison 2016, 140124
Kangaroo Flats, SA, Australia, 2014 (Mace)	2 (14)	24, 24–26	141, 141	98, 98	82	1.0	Addison 2016, 140127
Cosgrove, VIC, Australia, 2014 (Phantom)	2 (14)	24, 30	141, 141	112, 95	115	0.62	Addison 2016, 140130
Narrabri, NSW, Australia, 2014 (Lancer)	2 (14)	24, 32	138, 138	84, 84	67	0.72	Addison 2016, 140133
Hobby's Yards, NSW, Australia, 2014 (Gregory)	2 (14)	29, 44	139, 139	111, 110	46	0.56	Addison 2016, 140134
Darling Downs, QLD, Australia, 2014 (Gregory)	2 (14)	24, 32	139, 139	87, 79	82	0.36	Addison 2016, 140137
Cambooya, QLD, Australia, 2014 (Gregory)	2 (14)	24, 24-28	136, 136	80, 92	75	0.45	Addison 2016, 140138

No residues above LOQ were found in the untreated control samples.

APPRAISAL

Diquat was first reviewed by the 1970 JMPR. The toxicology and residues aspects of diquat were reconsidered by the 2013 JMPR as part of the periodic review program. The 2013 JMPR established an ADI of 0–0.006 mg/kg bw and an ARfD of 0.8 mg/kg bw, and recommended a residue definition for compliance with the MRL and dietary risk assessment for plant and animal commodities of *diquat cation*, along with maximum residue levels in a number of plant and animal commodities.

Diquat was scheduled by the Forty-ninth Session of the CCPR (2017) for evaluation of additional residues data in pulses and cereals.

Methods of analysis

No new methods of analysis were submitted to the Meeting. In all trials presented to the Meeting, samples were analysed using a LC-MS/MS method (method number GRM012.03A), which was considered by the 2013 JMPR. Additional validation data in pulse matrices (chickpea, lentil, dry bean and dry pea seed) were generated for the current Meeting, since the previous validation data for this method did not include a high protein commodity. Acceptable recovery values were achieved in these matrices over a fortification range of 0.01–3.0 mg/kg.

Stability of residues in stored analytical samples

The Meeting received the final version of a storage stability study provided as an interim report to the 2013 JMPR, together with storage stability data generated concurrently with the cereal residue trials. The final version of the storage stability study confirmed the assessment of the 2013 JMPR, that residues of diquat are stable in a range of commodities (spinach, wheat grain, oilseed rape seed, lentil, whole orange, potato and wheat straw) over 24 months frozen storage. This data covers a sufficient time interval to support the proposition that residues of diquat will have remained stable for the storage times used in the trials presented to the Meeting.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for foliar application of diquat to beans, dry, chickpeas, lentils and peas, dry for pre-harvest desiccation, and for foliar application to barley, oats and wheat for weed control both early in the crop and immediately pre-harvest.

All rates discussed below are expressed in terms of g ai/ha of diquat cation.

Pulses

In the discussion of uses on the label from Canada below, the following applies concerning harvesting of pulses. There is no specific harvest interval on the label, however information on the label regarding harvesting states that 'harvesting can normally commence within 4–10 days of desiccation'. The Meeting considered that 4 days represented the minimum interval likely to be used in practice, as sufficient time is required for effective desiccation or weed control.

Dry beans, Subgroup of

The critical GAP for beans (including white and red kidney beans, soya beans, adzuki beans and faba beans) in Canada is a single aerial application at 552 g ai/ha for pre-harvest desiccation.

Residue trials in beans, dry were conducted in Canada during the 2015 season and matching the Canadian GAP (1 application at 0.87–0.95× the maximum Canadian rate, and with sampling at a 4–5 day PHI). Residues observed in these trials are shown below (*in italics*) together with residues observed in data from JMPR 2013, from trials conducted in Germany in 1984 and the USA in 1994 matching the Canadian GAP.

Residues of diquat in beans, dry after treatment at GAP were (n = 24): *0.01*, *0.012*, *0.019*, < 0.02 (3), *0.040*, *0.044*, < 0.05 (8), 0.05, 0.08, 0.09, *0.15*, 0.15, *0.18* (*2*), and *0.35* mg/kg.

Similarly data was available in the 2013 JMPR for dry soya beans from trials conducted in France matching the Canadian GAP.

Residues of diquat in soya beans, dry after treatment at GAP were (n = 3): < 0.05, 0.06 and < 0.1 mg/kg.

The combined dataset in ranked order is (n = 27) were: 0.01, 0.012, 0.019, < 0.02 (3), 0.040, 0.044, < <u>0.05</u> (9), 0.05, 0.06, 0.08, 0.09, < 0.1, 0.15 (2), 0.18 (2) and 0.35 mg/kg.

The Meeting noted that several commodities in the subgroup of dry beans were covered by the Canadian GAP, and decided to estimate a maximum residue level for the subgroup. The Meeting estimated a maximum residue level of 0.4 mg/kg for diquat in the subgroup of Dry beans, together with a STMR of 0.05 mg/kg. The Meeting withdrew the previous recommendation of 0.2 and 0.3 mg/kg for beans (dry) and soya bean (dry), respectively.

Chickpeas

The critical GAP for chickpeas in Australia is a single application at 600 g ai/ha for pre-harvest crop desiccation, with a 2-day PHI. No trials were available with a 2-day PHI, and only two were available with a 1-day PHI.

The GAP for chickpeas in Canada is a single ground foliar spray application at 408 g ai/ha for pre-harvest desiccation.

Residue trials in chickpeas were conducted in Canada during the 2015 season and matching the Canadian GAP (1 application at 1.1-1.29× the maximum Canadian rate, and with sampling at a 4-5 day PHI).

Residues of diquat in chickpeas after treatment at GAP were (n = 9): 0.070, 0.10, 0.16, 0.18, <u>0.24</u>, 0.26, 0.32, 0.38, and 0.58 mg/kg.

The Meeting estimated a maximum residue level of 0.9 mg/kg, together with a STMR of 0.24 mg/kg, for diquat in chickpea (dry).

Subgroup – Dry peas (except chick-peas)

Lentils

The critical GAP for lentils in Australia is a single application at 600 g ai/ha for pre-harvest crop desiccation, with a 2-day PHI. No trials were available with a 2-day PHI, and only two were available with a 1-day PHI.

The GAP for lentils in Canada is a single aerial application at 552 g ai/ha for pre-harvest desiccation.

Residue trials in lentils were conducted in Canada during the 2015 season and matching the Canadian GAP (1 application at 0.85-0.91× the maximum Canadian rate, and with sampling at a 4-5 day PHI).

Residues of diquat in lentils after treatment at GAP were (n = 8): 0.052, 0.070, 0.10, <u>0.16</u>, <u>0.18</u>, 0.21, 0.33, and 0.57 mg/kg.

Peas, dry

The GAP for dry peas in Australia is a single application at 600 g ai/ha for pre-harvest crop desiccation, with a harvest withholding period not required.

The GAP for peas, dry in Canada is a single application at 552 g ai/ha for pre-harvest desiccation.

Residue trials in peas, dry were conducted in Canada during the 2015 season and matching Canadian GAP (1 application at 0.87–0.93× the maximum Canadian rate, and with sampling at a 4–5 day PHI). Residues observed in these trials are shown below (*in italics*) together with data from JMPR 2013, from trials conducted in Germany in 1984/5 and the UK in 1992, and the USA in 1994 matching the Canadian GAP.

The combined dataset is (n = 21): *0.014*, *0.020*, 0.03, *0.038*, 0.04, < 0.05 (3), 0.05 (4), *0.054*, 0.06 (2), *0.061*, 0.09, 0.10, 0.11, *0.13*, and 0.56 mg/kg.

The Meeting noted that data were available for lentils, and peas, dry and considered a maximum residue level for the subgroup of dry peas (except chickpeas). The Meeting noted that the median residues for the datasets for the two crops differed by less than 5-fold, and noted that the datasets were not statistically similar (Mann-Whitney). The Meeting therefore decided to estimate a maximum residue level for the subgroup of dry peas based on the lentil dataset.

The Meeting estimated a maximum residue level of 0.9 mg/kg, together with a STMR of 0.17 mg/kg, for diquat in the subgroup of dry peas (except chickpeas). The Meeting withdrew the previous recommendation of 0.3 mg/kg for peas (dry).

Cereals

In the discussion of uses on the label from Australia below, the following applies concerning harvesting of cereals after a single pre-harvest application. Where a harvest withholding period is stated as being not required when used as directed, the Meeting considered that at least 4 days would be required for effective weed and crop dry down. The Meeting considered that harvest at a minimum of 4 days was consistent with expected agricultural practice in Australia.

Barley

The GAP for barley in Australia is a single application at 600 g ai/ha made shortly before harvest for weed control. A harvest withholding period is stated as being not required when used as directed. Residue data were available at 2-4 day harvest intervals and at a 7-day harvest interval.

Residues of diquat cation in barley at 4 days after application were (n = 1): 0.15 mg/kg.

Considering that decline data were available for all trial sites, the Meeting noted that for several of the barley trial sites, the interpolated residue level at 4 days after application differed by less than ±25% from the measured level at 2 or 3 days after application.

Residues of diquat cation in barley approximating GAP were (n = 6): 0.15, 0.53, 1.1, 2.0 (2), and 2.1 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg for diquat in barley, together with a STMR of 1.55 mg/kg.

Oats

The GAP for oats in Australia is an early application for weed control at 140 g ai/ha made between the 3-leaf and early tillering stage (BBCH 13–22) plus a pre-harvest weed control application at 600 g ai/ha shortly before harvest.

Residue trials generated in Australia for oats were provided to the Meeting but did not match GAP.

Rye and triticale

The GAP for winter cereals in Australia is a single application at 600 g ai/ha made shortly before harvest for weed control. A harvest withholding period is stated as being not required when used as directed.

The Meeting agreed to use the wheat data to estimate maximum residue levels for rye and triticale.

Considering that decline data were available for all trial sites, the Meeting noted that for several of the trial sites, the interpolated residue level at 4 days after application generally differed by less than ±25% from the measured level at 2 or 3 days after application.

Residues of diquat cation in wheat at 2–4 days after application (at trial sites where values consistent with those expected from use in accordance with GAP were obtained) were (n = 6): 0.28, 0.41, 0.45, 0.56, 0.57 and 0.78 mg/kg.

Based on the wheat data, the Meeting estimated maximum residue levels and STMRs of 1.5 mg/kg and 0.505 mg/kg respectively for rye and triticale.

Wheat

The GAP for wheat in Australia is an early application for weed control at 140 g ai/ha made between the 4-leaf and early tillering stage (BBCH 14–22) plus a pre-harvest weed control application at 600 g ai/ha shortly before harvest.

Residue trials generated in Australia for wheat were provided to the Meeting but did not match GAP.

Animal feeds

Barley forage

Residue data were available from the Australian cereal residue trials for barley forage. However, there is no relevant GAP in barley involving application at or before the forage stage.

Oat forage

The Australian GAP for application of diquat to oats at the forage stage is 1 × 140 g ai/ha application, with a 1-day grazing interval. Trials conducted in Australia in oats included forage sampling; however these trials did not match GAP.

Wheat forage

The Australian GAP for application of diquat to wheat at the forage stage is 1 × 140 g ai/ha application, with a 1-day grazing interval. Trials conducted in Australia in wheat included forage sampling; however these trials did not match GAP.

Barley, rye and triticale straw

The GAP for barley, rye and triticale straw in Australia is a single application at 600 g ai/ha made shortly before harvest for weed control.

Considering that decline data were available for all trial sites, the Meeting noted that for many of the trial sites, the interpolated residue level at 4 days after application differed by less than ±25% from the measured level at 2 or 3 days after application.

The Meeting considered that straws of cereal crops are not distinguished in trade and considered that data for barley, oat and wheat straw matching the GAPs for barley, rye and triticale could be combined for the purpose of obtaining more robust estimates for maximum residue levels.

Residues of diquat cation in barley straw at 2–4 days after application (at trial sites where values consistent with those expected from use in accordance with GAP were obtained) were (n = 5): 2.8, 6.2, 6.9, 23, and 26 mg/kg (on a dry weight basis). Residues of diquat cation in oat straw at 2–4 days after application (at trial sites where values consistent with those expected from use in accordance with GAP were obtained) were (n = 4): 0.27, 1.8, 2.8, and 3.1 mg/kg.

Residues of diquat cation in wheat straw at 2-4 days after application (at trial sites where values consistent with those expected from use in accordance with GAP were obtained) were (n = 8): 1.2, 2.0, 2.4, 2.8, 3.3, 4.3, 5.6 and 6.1 mg/kg.

The Meeting further noted that the median residues of the barley, oat and wheat data sets differed by < 5×, and that the datasets were statistically similar (Kruskal-Wallis), and agreed to combine them for the purpose of estimating maximum residue levels.

The combined data set is (n = 17): 0.27, 1.2, 1.8, 2.0, 2.4, 2.8 (3), 3.1, 3.3, 4.3, 5.6, 6.1, 6.2, 6.9, 23, and 26 mg/kg.

The Meeting estimated maximum residue levels of 40 mg/kg for diquat in barley straw and fodder (dry), rye straw and fodder (dry), and triticale straw and fodder (dry), together with median and highest residues of 3.1 and 26 mg/kg respectively.

Oat straw

The GAP for oat straw in Australia is an early application for weed control at 140 g ai/ha made between the 4-leaf and early tillering stage (BBCH 14–22) plus a pre-harvest weed control application at 600 g ai/ha shortly before harvest.

Residue trials generated in Australia for oats were provided to the Meeting but did not match GAP.

Wheat straw

The GAP for wheat straw in Australia is an early application for weed control at 140 g ai/ha made between the 4-leaf and early tillering stage (BBCH 14–22) plus a pre-harvest weed control application at 600 g ai/ha shortly before harvest.

Residue trials generated in Australia for wheat were provided to the Meeting but did not match GAP.

Fate of residues during processing

No processing studies were provided to this Meeting. The 2013 JMPR considered a processing study in soya bean, and this was used to estimate updated values for processed soya commodities based on the increased STMR covering soya bean RAC estimated by the Meeting.

Summary of selected processing factors for diquat

Raw commodity	Processed commodity	Individual PF	Best estimate PF	$STMR_{RAC}$ (mg/kg)	$STMR_{RAC}$ × PF (mg/kg)	RAC MRL (mg/kg)	Processed commodity MRL
Soya bean	Hulls	2.6 3.6	3.1	0.05	0.155	0.4	1.24
	Meal	0.7 1.0	0.85		0.0425		-
	Oil	< 0.04 < 0.07	< 0.055		< 0.00275		-

Based on the estimated maximum residue level for the RAC of the dry beans subgroup, and the processing factor, the Meeting estimated a maximum residue level of 1.5 mg/kg for soya bean hulls (3.1 × 0.4 = 1.24, rounded up to nearest 'step').

Residues in animal commodities

Farm animal feeding studies

The 2013 JMPR received information on the residue levels arising in tissues and milk when dairy cows were fed a diet containing incurred residues of diquat at dietary levels of 18, 50 and 84 ppm for 30 consecutive days. There were no residues of diquat at or above the LOQ (0.001 mg/kg) in any of the milk samples or at or above the LOQ (0.01 mg/kg) in any of the tissue samples (liver, kidney, fat and muscle) from any of the dose groups throughout the duration of the study.

The 2013 JMPR also received information on the residue levels arising in tissues and eggs, when laying hens were fed a diet containing diquat at total dietary levels of 1, 5 and 10 ppm diquat for 21 or 28 consecutive days. No residues of diquat above the LOQ (< 0.01 mg/kg) were found in any of the egg, fat, muscle, skin, liver or heart samples.

Livestock dietary burden

Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO Manual, are presented in Annex 6 and summarised below.

Summary of livestock dietary burden (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	Max	Mean	max	Mean	max	Mean
Beef cattle	4.0	1.33	17	6.3	29❶	18❸	1.27	1.27
Dairy cattle	6.7	3.0	18	7.2	29❷	14❹	2.25	0.95
Poultry Broiler	1.37	1.37	1.32	1.30	0.34	0.34	0.20	0.20
Poultry Layer	1.37	1.37	5.8❺	3.5❻	0.34	0.34	0.04	0.04

❶ Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimates for mammalian meat

❷ Highest maximum dairy cattle dietary burden suitable for maximum residue level estimates for mammalian milk

❸ Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat.

❹ Highest mean dairy cattle dietary burden suitable for STMR estimates for milk.

❺ Highest maximum poultry dietary burden suitable for maximum residue level estimates for poultry meat and eggs.

❻ Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs.

Animal commodity maximum residue levels

The Meeting noted that at the estimated maximum dietary burdens of 29 and 5.8 ppm for cattle and poultry respectively, no residues are expected in tissues, milk, or eggs. Slight increases in the dietary burdens over those calculated by the 2013 JMPR were noted for dairy cattle and poultry.

The Meeting considered that the recommendations of the 2013 JMPR for maximum residue levels at the LOQ (0.01(*) mg/kg; 0.001(*) mg/kg for milk), together with nil STMR and HR values, for mammalian and poultry meat and offal, milk, and eggs remained appropriate.

The Meeting estimated maximum residue levels of 0.01(*) mg/kg for mammalian and poultry fats, together with STMR and HR values of 0.

RECOMMENDATIONS

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

Definition of the residue for compliance with MRL and for estimation of dietary intake (for animal and plant commodities): *Diquat ion*

The residue is not fat-soluble.

CCN	Commodity name	Recommended maximum residue level, mg/kg		STMR (P), mg/kg	HR (P), mg/kg
		New	Previous		
GC 0640	Barley	5	-	1.55	-
AS 0640	Barley straw and fodder, dry	40 (dw)	-	Median: 3.1 (dw)	Highest: 26 (dw)
VD 0071	Beans, dry	W	0.2		
VD 0524	Chick-pea (dry)	0.9	-	0.24	-
VD 2065	Dry beans, Subgroup of (includes all commodities in this subgroup)	0.4	-	0.05	-
VD 2066	Dry peas, Subgroup of (except chick-pea (dry))	0.9	-	0.17	-
MF 0100	Mammalian fats (except milk fats)	0.01*	-	0	0
VD 0072	Peas (dry)	W	0.3		
PF 0111	Poultry fats	0.01*	-	0	0
GC 0650	Rye	1.5	-	0.505	-
AS 0650	Rye straw and fodder, dry	40 (dw)	-	Median: 3.1 (dw)	Highest: 26 (dw)
VD 4521	Soya bean (dry)	W	0.3		
AB 0541	Soya bean hulls	1.5	-	Median: 0.155	-
GC 0653	Triticale	1.5	-	0.505	-
AS 0653	Triticale straw and fodder, dry	40 (dw)	-	Median: 3.1 (dw)	Highest: 26 (dw)

dw = dry weight

Dietary exposure and feed burden only

Commodity		STMR, STMR-P or median (mg/kg)
CCN	Name	
	Soya bean meal	0.0425
OR 0541	Soya bean oil, refined	0.00275

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for diquat is 0–0.006 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for diquat were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the 2013 and 2018 JMPR. The results are shown in Annex 3 of the 2018 JMPR Report. The IEDIs ranged from 2–30% of the maximum ADI.

The Meeting concluded that long-term dietary exposure to residues of diquat from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for diquat is 0.8 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for diquat were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2018 JMPR Report. The IESTIs varied from 0–7% of the ARfD for children and 0–10% for the general population.

The Meeting concluded that acute dietary exposure to residues of diquat from uses considered by the present Meeting is unlikely to present a public health concern.

REFERENCES

Code	Authors	Year	Title, Report reference
SYNGENTACROP/ GLP/13/83-1	Addison, S.	2016	Determination of Residues of Diquat in Wheat, Barley and Oat Forage, Straw and Seed Following the Early Post-Emergence or Pre-Harvest Application of Reglone Australia 2014, Agrisearch report number SYNGENTACROP/GLP/13/83-1, 23 February 2016, unpublished
AKC-1601	Keats, A.	2017	Determination of Diquat Residues in Barley Crops Following One Application of Reglone Non-Residual Herbicide, Study number AKC-1601, 13 December 2017, unpublished
CEMR-4964	Langridge, G.	2013	Diquat Dibromide – Frozen Storage Stability of Diquat Cation in Spinach, Wheat Grain, Oilseed Rape Seed, Lentil, Orange, Potato and Wheat Straw, Study number CEMR-4964, 4 June 2013, unpublished
TK0225767	Sagan, K.	2017	Diquat SL (A1412H) – Magnitude of the Residues in or on Chickpea, Lentil, and Dry Bean Canada 2015, Study number TK0225767, 13 March 2017, unpublished
TK0256751	Sagan, K.	2017	Fludioxonil/Cyprodinil WG (A9219B) & Diquat SL (A1412H) Magnitude of the Residues in or Dry Pea (Representative Commodities for Crop Group 6C) Canada 2015, Study number 0256751, 15 March 2017, unpublished