

DICAMBA (240)

First draft prepared by Dr. Yukiko Yamada, Ministry of Agriculture, Forestry and Fisheries, Tokyo, Japan

EXPLANATION

Dicamba, a systemic broad-spectrum herbicide, was first evaluated by the 2010 JMPR which estimated an ADI of 0–0.3 mg/kg bw and ARfD of 0.5 mg/kg bw, and recommended the following residue definitions for plant commodities:

Definition of the residue for plant commodities (for compliance with the MRL): *Dicamba*

Definition of the residue (for estimation of dietary intake) for plant commodities: *Sum of dicamba and 5-OH dicamba expressed as dicamba*

Definition of the residue for animal commodities (for compliance with the MRL and for estimation of dietary intake): *Sum of dicamba and DCSA*.

Based on the above residue definition, the 2010 Meeting also recommended maximum residue levels for 21 commodities. These maximum residue levels were adopted as Codex MRLs at the Codex Alimentarius Commission in 2011 (REP 11/CAC, Appendix III, Part 2).

The 2010 JMPR received information on metabolism, method of analysis, storage stability, supervised residue trials and processing studies on soya beans. However, supervised trials were conducted in the USA with PHI of 7 days while the approved US label at that time indicated PHI of 14 days. Since no trials matched the GAP, the Meeting could not estimate a maximum residue level for soya bean (dry).

The label of one formulation, relevant to the trials, was since revised and approved with new PHI of 7 days matching that of the supervised trials. New US GAP allows two different applications: application of 0.56 kg ai/ha as a broadcast made to the soil surface approximately 14 days prior to planting and application of 1.12 kg ai/ha applied 7 days prior to harvest. The maximum total application rate per season is 2.24 kg ai/ha.

In the supervised trials on soya beans provided to the 2010 Meeting, pre-plant application of 0.56 kg ai/ha 14 days before planting and pre-harvest application of 2.24 kg ai/ha 7 days before harvest were made. The pre-harvest application rate was two times the GAP rate. The 2011 Meeting decided to apply the concept of proportionality to the residues from these trials to recommend a maximum residue level.

The Forty-fifth Session of the Codex Committee on Pesticide Residues in 2013 considered the Principles and Guidance for Application of the Proportionality Concept to Estimation of Maximum Residue Limits for Pesticides and agreed to forward the document to the 36th Session of the Commission for adoption and inclusion in the Procedural Manual as an Annex to the Risk Analysis Principles Applied by the Codex Committee on Pesticide Residues. It agreed that desiccants should be excluded from the application of the proportionality concept (REP 13/PR, paras 93 and 98, Annex VIII). The 36th Codex Alimentarius Commission adopted the proposed amendments to the Procedural Manual as above.

The current Meeting received information on new supervised trials on soya beans following the US GAP and analytical methods used for the determination of dicamba and related compounds in the new supervised trials on soya beans

RESIDUE ANALYSIS

Analytical Methods

The 2010 JMPR reviewed a number of analytical methods, mostly employing GC-ECD with GC-MS for confirmation, developed for the determination of dicamba, 5-OH dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA) in plant and animal commodities. Many of these methods developed for plant commodities were applicable to analysis of soya beans (dry).

An analytical method used to determine dicamba, 5-OH dicamba and DCSA arising in newly conducted supervised residue trials on soya beans was provided to the current Meeting. The method is summarized in Table 1 and its details are given below.

Table 1 Summary of analytical method for the determination of residues

Method No. Reference	Matrix tested	Analytes	Method	Limit of Quantitation
BASF Analytical Method No. D0902, Saha, M. (2009) *	Soya bean	Dicamba 5-OH dicamba DCSA	LC-MS/MS	0.01 mg/kg for each analyte, except for 0.05 mg/kg for each analyte in forage and hay (2010 Supervised Field Trials only)

*Validity of the method was tested on corn matrices in the cited separately performed study.

The soya bean samples were homogenized using a Retsch ultra centrifugal milling with liquid nitrogen cooling. The forage and hay samples were processed using a Stephan floor chopper with dry ice. All samples were stored frozen in plastic bags until analysis.

Residues in soya bean homogenate were extracted by hydrolysis in 1N HCl at 90 °C, cooling to room temperature, centrifugation and filtration. The filtrate was diluted with water and an aliquot of the extract was cleaned-up by liquid-liquid partitioning with hexane:ethyl acetate (1:1, v/v) and the organic phase was evaporated to dryness. Residues in dried organic phase were re-dissolved in methanol-water (1:9, v/v) and analysed by LC-MS/MS. MS/MS detection in the negative ionization mode was used to monitor ion transitions from m/z 219→175 for dicamba; m/z 235→155 for 5-OH-dicamba; and m/z 205→161 for DCSA.

For the analysis of the forage and hay samples from the 2010 growing season, a hexane partition was added prior to the hexane:ethyl acetate partition. This additional clean-up step was used due to matrix interferences. Other minor modifications were also made to the method but none of them had significant influence to analytical results.

This method was validated for dicamba, 5-OH dicamba and DCSA in soya bean matrices at the fortification concentration range between 0.01 and 10 mg/kg, in conjunction with the field sample analyses obtained in 2010 and 2012 growing seasons. The results of validation are summarized in Table 2.

The mean recoveries at all fortification levels were 73–100%, within an acceptable range for the three analytes in soya bean seeds with the relative standard deviation well below 20%. On the other hand, the mean recoveries of these three analytes in soya bean forage and hay were mostly between 70 and 109% with the relative standard deviation below 20% except that occasionally at the fortification level of 0.01 mg/kg, the recoveries were lower than 70% and the relative standard deviation around 20% were observed in 2010.

The limit of quantitation (LOQ) of this method is 0.01 mg/kg for the determination of dicamba, 5-OH dicamba and DCSA in soya bean seeds, forage and hay. However, the LOQ was 0.05 mg/kg for these analytes in soya bean forage or hay in the analyses performed in 2010.

Table 2.Recoveries of dicamba, 5-OH dicamba and DCSA in soya bean matrices by the method No.D0902

Soya bean matrix	Analyte	Fortification level (mg/kg)	N	Recoveries (%)	Mean (%)	RSD (%)
Seeds (2010 season)	Dicamba	0.01	6	78, 79, 90, 101, 110, 90	91	13
		1.0	5	74*(75,73), 71, 87*(85,79,96), 81*(80,82), 72	77	9
		10	1*	83*(82,85)	83	-
	5-OH dicamba	0.01	4	65, 63, 81, 82	73	14
		0.05	2	102, 99*(90,107)	100	-
		1.0	1*	85*(83,86)	85	-
		10	1*	94*(91,96)	94	-
	DCSA	0.01	6	90, 90, 92, 102, 76, 71	87	13
		1.0	5	79, 78, 80*(67,93), 79*(79,78), 83	80	3
10		1*	88*(90,87)	88	-	
Seeds (2012 season)	Dicamba	0.01	3	95, 77, 92	88	11
		1.0	3	86, 87, 87	87	1
	5-OH dicamba	0.01	3	82, 83, 78	81	3
		1.0	3	98, 99, 99	99	1
	DCSA	0.01	3	81, 82, 79	81	2
		1.0	3	74, 80, 83	79	6
Forage (2010 season)	Dicamba	0.01	4	67, 70, 61, 71	67	6
		0.05	2	88, 98	93	-
		0.1	1	92	-	-
	5-OH dicamba	0.01	6	72, 68, 86, 88, 94, 83	82	12
		0.05	2	102, 82	92	-
		0.1	2	90, 92	91	-
	DCSA	0.01	6	82, 87, 86, 83, 76, 67	80	10
		0.05	2	105, 69	87	-
		0.1	2	83, 91	87	-
Forage (2012 season)	Dicamba	0.01	2	92, 102	97	-
		1.0	2	101, 103	102	-
	5-OH dicamba	0.01	2	95, 99	97	-
		1.0	2	109, 108	109	-
	DCSA	0.01	2	98, 95	96	-
		1.0	2	103, 101	102	-
Hay (2010 season)	Dicamba	0.01	2	72, 100	86	-
		0.05	2	81, 81	81	-
		1.0	1	73	-	-
	5-OH dicamba	0.01	4	53, 65, 62, 86	66	21
		0.05	4	87, 87, 75, 77	82	7
		1.0	1	93	-	-
	DCSA	0.01	3	55, 57, 71	61	15
		0.05	3	67, 77, 76	73	7
		1.0	1	77	77	-
Hay (2012 season)	Dicamba	0.01	1	74, 95	84	-
		1.0	1	95, 98	97	-
	5-OH dicamba	0.01	1	73,101	87	-
		1.0	1	96,106	101	-
	DCSA	0.01	1	75, 89	82	-
		1.0	1	103, 92	98	-

An asterisk (*) denotes that the result is the mean of duplicate or triplicate analyses of the same fortification sample.

USE PATTERNS

The authorized use on soya beans in the USA is summarized in Table 3.

Table3 Registered uses of dicamba on soya bean (dry) in the USA

Crop	Country	Formulation (g/kg or g/L and type)	F/G/P ^a	Application			PHI (days)
				Method	No. per crop season	min. - max. kg ai/ha per applic. ^b	
Soya bean (dry)	USA	480SL	F	Spray*	1 (pre-plant)	0.14–0.56	NA
					1 (pre-harvest)**	0.28–1.12	7
					Total 2	(2.24/season)	-

^a F = outdoor or field use, G = glasshouse, P = protected

^b Information given on active ingredient referring to dicamba only

NA = not applicable

* Aerial spray is also allowed.

** After soya bean pods have reached mature brown colour and at least 75% leaf drop has occurred. Do not feed soya bean fodder and hay following a pre-harvest application.

The label permits grazing after the pre-plant application while use of soya bean plants for grazing is not a common practice unless good harvest is not expected for some reasons such as natural disasters. The label prohibits feeding the plant materials after pre-harvest application.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The information on new supervised field trials of dicamba on soya beans, conducted in the USA in 2010 and 2012, were received and summarized in Table 4.

All trials were conducted outdoor. Application rates were reported as dicamba acid equivalents expressed in kg ai/ha. Residue concentrations were reported for dicamba, 5-OH dicamba and DCSA expressed in mg dicamba equivalents/kg. Residue concentrations are recorded unadjusted for recoveries or for residue values in control samples. Where duplicate samples were taken from a single plot, individual results are reported, and the calculated average concentration (in parentheses) is used for the estimation of maximum residue level. Where trials were conducted in the same location, with the same or similar varieties, similar formulations or different salt types, and at the same or similar timing, they are not regarded as independent and the highest residues from these trials were used for estimating a maximum residue level and STMR. Although trials included control plots, no control data are recorded in the tables below unless residues in control samples significantly exceeded the LOQ.

Total residues were calculated by summing up the concentrations of dicamba and 5-OH dicamba expressed in mg dicamba equivalents/kg. Residue concentrations found to be below the limit of quantitation (LOQ) or the limit of detection (LOD) were reported as < LOQ or < LOD respectively. Where the concentration of dicamba or 5-OH dicamba was < LOQ, they were regarded to be at the LOQ and where it was < LOD, they are regarded to be 0 for calculation.

Residues from the trials conducted according to maximum GAP and used for the estimation of maximum residues and STMR/HR are underlined.

Pulses

Soya bean

The approved use of dicamba in soya bean consists of two different applications: application of up to 0.56 kg ai/ha as a broadcast made to the soil surface approximately 14 days prior to planting, and/or up to 1.12 kg ai/ha applied 7 days prior to harvest. If both pre-plant and pre-harvest applications are used in one season, the maximum seasonal use rate must not exceed 2.24 kg ai/ha. Soya bean plants are susceptible to dicamba.

2010 Season (bridging trials)

Field trial data were generated for dicamba from four bridging trials (equivalent of 12 tests) on soya bean conducted in EPA Region 5 (Iowa, Minnesota, Nebraska, and South Dakota; one trial each) during the 2010 growing season.

For each trial, one untreated control plot and three “side-by-side” treated plots comparing three different soluble concentrate (SL) formulations of dicamba were established. The formulations tested were: a diglycolamine (DGA) salt of dicamba (480 g ai/L SL); a diethylenetriamine (DETA) salt of dicamba (480 g ai/L SL); and an N,N-bis (3-aminopropyl)-methylamine (BAPMA) salt of dicamba (600 g ai/L SL).

The treated plots received one broadcast application of the designated dicamba formulation to the soil targeting 28 days before planting at 0.56 kg ai/ha and one pre-harvest application targeting 7 days before the harvest of mature soya bean seed at 1.12 kg ai/ha, for a seasonal total of 1.7 kg ai/ha. The actual total application rate was 1.63–1.73 kg ai/ha/season. The applications were made using ground equipment in approximately 181–211 l/ha of water. An adjuvant was included in the spray mixture (0.25% v/v non-ionic spreader/sticker/surfactant of a minimum 80% active ingredient) together with ammonium sulfate liquid fertilizer (2% w/v).

From each untreated plot, single forage and hay (forage dried to less than 20% moisture) samples were collected at the BBCH 16 growth stage (vegetative, 6th trifoliolate leaf unfolded), and a single sample of seed was collected at maturity (BBCH89). From each treated plot, duplicate forage and hay samples were collected at the BBCH 16 growth stage, 66–78 days after treatment (37–50 days after planting), and duplicate samples of seed were collected at maturity (BBCH89), 7 days after the second (last) application.

The soya bean samples were stored frozen (<-10 °C) for maximum of 872 days (29 months) for seed, 957 days (31 months) for forage, and 962 days (32 months) for hay from sample collection to extraction for analysis.

The residues of dicamba and its metabolites 5-OH-dicamba and DCSA in/on soya bean samples were quantitated by LC-MS/MS using BASF analytical method D0902. Acceptable concurrent method validation data for soya bean matrices were obtained for each analyte. In this study, the validated LOQ for residues of dicamba, 5-OH dicamba, and DCSA was 0.01 mg/kg in/on soya bean seed (dry) samples and 0.05 mg/kg in/on soya bean forage and hay samples. Apparent residues of dicamba (each analyte) were < LOQ in/on all untreated control soya bean seed, forage and hay samples. Concurrent analysis of untreated soya bean samples (seed, forage and hay) fortified with a mixture of dicamba, 5-OH-dicamba and DCSA at 0.01, 0.05, 0.1, 1 or 10 mg/kg, resulted in acceptable range of recovery of 70–120% at the stated LOQs with some isolated exceptions.

After only preplant application of three different SL formulations of dicamba, residues of dicamba were below the limit of quantitation of 0.05 mg/kg in all soya bean forage and hay samples harvested at a growth stage targeting BBCH 16.

After one pre-plant and one pre-harvest applications using the different salt types, residues measured in the seed (dry) samples were primarily the parent herbicide and mostly relatively low (< 0.1 mg/kg). However, several samples had significant residues but not in a particular pattern. These residues in the seed suggest that the pod containing the seed may have begun to split allowing residues to contact the seed. Another possibility for the significant residues is contact of the seed with the outer surface of the pods during shelling.

2012 Season

Eight field trials on soya bean were conducted during the 2012 growing season in EPA Region 2 (one trial each in New Jersey and North Carolina), Region 4 (Louisiana, one trial) and Region 5 (Illinois and Wisconsin, one trial each; Iowa, three trials). Each trial location consisted of one untreated and one treated plot. The treated plot received one broadcast pre-plant application of dicamba (a diglycolamine (DGA) salt of dicamba, 480 g dicamba acid equivalents/L SL) at the rate targeting

0.56 kg ai/ha to the soil targeting 28 days prior to planting, followed by one late-season, broadcast foliar application targeting 1.12 kg ai/ha, 7 days prior to harvest of mature seed. The actual total application rate was 1.66–1.74 kg ai/ha/season. The applications were made using ground equipment in approximately 181-219 l/ha of water with the same adjuvant as in 2010 and 2 % w/v ammonium sulfate.

From each untreated plot, single forage and hay (forage dried to less than 20% moisture) samples were collected at the BBCH 16 growth stage, and a single sample of seed was collected at maturity (BBCH89). From each treated plot, duplicate forage and hay samples were collected at the BBCH 16 growth stage, 62–88 days after treatment (33–60 days after planting); and duplicate samples of seed were collected at maturity (BBCH 89), 6 or 7 days after the last application. In one trial (Guthrie, IA), seed samples were harvested at 0, 3, 11 and 14 days after the last application, in addition to the 7 day PHI, to examine residue decline.

The soya bean samples were stored frozen from harvest to extraction, for 88–205 days (2.9 to 6.9 months). Samples were analysed within 6 days of extraction. Adequate storage stability data are available to support the storage conditions and durations for the samples in the subject study.

Soya bean samples (seed, forage and hay) were analysed for residues of dicamba and the metabolites 5-OH-dicamba and DCSA using a modified version of BASF Analytical Method No. D0902. The LOQ for each matrix was 0.01 mg/kg for these three analytes. The limit of detection (LOD) was set at 20% of the LOQ, or 0.002 mg/kg. Apparent residues of dicamba and the metabolites were < LOQ in/on all untreated control soya bean seed, forage and hay samples.

Individual procedural recoveries of dicamba, DCSA and 5-OH-dicamba from soya bean seed, forage and hay samples were within 70–120%. Mean recoveries of each analyte across all soya bean matrices and fortification levels at 0.01 and 1.0 mg/kg ranged from 80 to 103% (4–15% RSD).

After one broadcast preplant plus one late-season pre-harvest application of dicamba 480 g ai/l SL, residues of parent dicamba in soya bean seed (dry) harvested at maturity, 6 or 7 days after the last application, ranged from < 0.01 mg/kg to 0.60 mg/kg and the metabolites 5-OH dicamba and DCSA, were < 0.01 mg/kg in/on the 16 treated soya bean seed (dry) samples in this study.

Data from the residue decline site indicate that dicamba residues in soya bean seed generally declined with increasing pre-harvest intervals.

Residues of dicamba, 5-OH dicamba and DCSA were determined and reported individually. Dicamba concentrations were used for estimating a maximum residue level of dicamba in/on soya bean (dry) and the sum of dicamba and 5-OH dicamba expressed as dicamba was calculated and used for exposure assessment. Where the concentration of 5-OH dicamba was below the LOQ, the value of LOQ was used for the calculation of total concentration. However, where the concentration of 5-OH dicamba was below the LOD, its concentration was assumed to be zero.

The dicamba concentrations used for estimating a maximum residue level of dicamba in/on soya bean (dry) are underlined and the sum of dicamba and 5-OH dicamba used for estimating STMR for exposure assessment are double-underlined in the following Table.

Table4 Residues of dicamba, 5-OH dicamba and DCSA in soya beans from supervised trials conducted in the USA

SOYA BEAN Year, Location (variety) Trial No.	Application					DALT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growth stage	No			Dicamba	5-OH dicamba	Total ^b	DCSA	
<u>US GAP</u>	<u>480 SL</u>	<u>Spray</u>	<u>0.56</u> +/ <u>1.12</u> (<u>max</u> <u>2.24/</u> <u>season)</u>	<u>Pre-</u> <u>plant</u>	<u>1</u>	<u>NA</u>						
			<u>7dbh</u>	<u>1</u>	<u>7</u>							

SOYA BEAN Year, Location (variety) Trial No.	Application				No	DALT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growth stage				Dicamba	5-OH dicamba	Total ^b	DCSA	
2010 York, NE (93Y12) Trial No. R100188	480 SL (DGA)	Pre-plant + foliar	0.56 + 1.15	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	0.10	< 0.01	0.11	< 0.01	Norris, F.A. 2013 BASF# 389561 2012/7005501
								0.11	< 0.01	0.12	< 0.01	
								(0.105)		(0.115)		
	480 SL (DETA)	Pre-plant + foliar	0.56 + 1.13	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	0.09	< 0.01	0.10	< 0.01	
								0.11	< 0.01	0.12	< 0.01	
								(0.10)		(0.11)		
600 SL (BAPMA)	Pre-plant + foliar	0.56 + 1.12	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	0.11	< 0.01	0.12	< 0.01		
							0.13	< 0.01	0.14	< 0.01		
							(0.12)		(0.13)			
2010 Stems, MN (Asgrow AG1002) Trial No. R100189	480 SL (DGA)	Pre-plant + foliar	0.54 + 1.13	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	< 0.01	< 0.01	< 0.02	< 0.01	Norris, F.A. 2013 BASF# 389561 2012/7005501
								< 0.01	< 0.01	< 0.02	< 0.01	
								(<u>< 0.01</u>)		(<u>< 0.02</u>)		
	480 SL (DETA)	Pre-plant + foliar	0.54 + 1.12	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	< 0.01	< 0.01	< 0.02	< 0.01	
								< 0.01	< 0.01	< 0.02	< 0.01	
								(<u>< 0.01</u>)		(<u>< 0.02</u>)		
600 SL (BAPMA)	Pre-plant + foliar	0.57 + 1.13	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	< 0.01	< 0.01	< 0.02	< 0.01		
							< 0.01	< 0.01	< 0.02	< 0.01		
							(<u>< 0.01</u>)		(<u>< 0.02</u>)			
2010 Turner, SD (Hefty 6238359) Trial No. R100190	480 SL (DGA)	Pre-plant + foliar	0.56 + 1.12	pre-plant 30 dbp + 7 dbh (BBCH 96)	2	7	seed	5.01	0.19	5.20	0.16	Norris, F.A. 2013 BASF# 389561 2012/7005501
								5.84	0.23	6.07	0.20	
								(<u>5.42</u>)		(<u>5.64</u>)		
	480 SL (DETA)	Pre-plant + foliar	0.55 + 1.13	pre-plant 30 dbp + 7 dbh (BBCH 96)	2	7	seed	0.08	< 0.01	0.09	< 0.01	
								0.02	< 0.01	0.03	0.05	
								(0.05)		(0.06)		
600 SL (BAPMA)	Pre-plant + foliar	0.55 + 1.08	pre-plant 30 dbp + 7 dbh (BBCH 96)	2	7	seed	0.02	< 0.01	0.03	< 0.01		
							0.03	< 0.01	0.04	< 0.01		
							(0.025)		(0.035)			

SOYA BEAN Year, Location (variety) Trial No.	Application				No	DALT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growth stage				Dicamba	5-OH dicamba	Total ^b	DCSA	
2010 Guthrie, IA (93Y15) Trial No. R100191	480 SL (DGA)	Pre-plant + foliar	0.55 + 1.08	pre-plant 28 dbp + 7 dbh (BBCH 95)	2	7	seed	0.04	< 0.01	0.05	< 0.01	Norris, F.A. 2013 BASF# 389561 2012/7005501
								0.04	< 0.01	0.05	< 0.01	
								(0.04)		(0.05)		
	480 SL (DETA)	Pre-plant + foliar	0.55 + 1.09	pre-plant 28 dbp + 7 dbh (BBCH 95)	2	7	seed	5.75	0.47	6.22	0.81	
								5.50	0.40	5.90	0.78	
								(5.62)		(6.06)		
600 SL (BAPMA)	Pre-plant + foliar	0.59 + 1.14	pre-plant 28 dbp + 7 dbh (BBCH 95)	2	7	seed	0.03	< 0.01	0.04	< 0.01		
							2.63	0.10	2.73	0.04		
							(1.33)		(1.385)			
2012 Hunterdon, NJ (02RM030020 / SC27-C4) Trial No. R120088	480 SL (DGA)	Pre-plant + foliar	0.57 + 1.17	pre-plant 28 dbp + 6 dbh (BBCH 89)	2	6	seed	0.04	< 0.002	0.04	< 0.002	Norris, F.A. 2013 BASF# 430098 2013/7001282
								0.03	< 0.002	0.03	< 0.01	
								(0.035)		(0.035)		
2012 Wayne, NC (S78-G6) Trial No. R120089	480 SL (DGA)	Pre-plant + foliar	0.57 + 1.14	pre-plant 28 dbp + 6 dbh (BBCH 89)	2	6	seed	0.07	< 0.002	0.07	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/7001282
								0.05	< 0.002	0.05	< 0.01	
								(0.06)		(0.06)		
2012 St. Landry Parish, LA (P95Y70) Trial No. R120090	480 SL (DGA)	Pre-plant + foliar	0.56 + 1.10	pre-plant 28 dbp + 7 dbh (BBCH 85)	2	7	seed	0.60	< 0.01	0.61	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/7001282
								0.49	< 0.01	0.50	< 0.01	
								(0.545)		(0.555)		
2012 Keokuk, IA (93Y82) Trial No. R120091	480 SL (DGA)	Pre-plant + foliar	0.56 + 1.13	pre-plant 29 dbp + 7 dbh (BBCH 89)	2	7	seed	0.01	< 0.01	0.02	< 0.002	Norris, F.A. 2013 BASF# 430098 2013/7001282
								< 0.01	< 0.01	< 0.02	< 0.002	
								(0.01)		(0.02)		
2012 Clinton, IL (S39-U2) Trial No. R120092	480 SL (DGA)	Pre-plant + foliar	0.59 + 1.13	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	0.02	< 0.002	0.02	< 0.002	Norris, F.A. 2013 BASF# 430098 2013/7001282
								0.03	< 0.01	0.04	< 0.002	
								(0.025)		(0.03)		
2012 Walworth, WI (AG2031RR) Trial No. R120093	480 SL (DGA)	Pre-plant + foliar	0.56 + 1.12	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	< 0.01	< 0.002	< 0.01	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/7001282
								< 0.01	< 0.002	< 0.01	< 0.01	
								(≤ 0.01)		(≤ 0.01)		
2012 Boone, IA (93Y15) Trial No. R120094	480 SL (DGA)	Pre-plant + foliar	0.57 + 1.14	pre-plant 28 dbp + 7 dbh (BBCH 89)	2	7	seed	0.03	< 0.002	0.03	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/7001282
								0.02	< 0.002	0.02	< 0.01	
								(0.025)		(0.025)		

SOYA BEAN Year, Location (variety) Trial No.	Application				No	DALT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growth stage				Dicamba	5-OH dicamba	Total ^b	DCSA	
2012 Guthrie, IA (93Y15) Trial No. R120095	480 SL (DGA)	Pre-plant + foliar	0.56 + 1.13	pre-plant 28 dbp + 7 dbh	2	0	seed	0.08	< 0.002	0.08	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/7001282
								0.10	< 0.002	0.10	< 0.01	
								< 0.01	< 0.002	< 0.01	< 0.002	
								0.01	< 0.002	0.01	< 0.002	
								0.01	< 0.002	0.01	< 0.002	
								0.01	< 0.002	0.01	< 0.002	
								0.02	< 0.002	0.02	< 0.01	
								0.02	< 0.002	0.02	< 0.01	
							(0.02)		(0.02)			
							0.02	< 0.002	0.02	< 0.01		
							0.01	< 0.002	0.01	< 0.01		

^a Expressed in mg dicamba equivalents/kg. Average values in parentheses.

^b Sum of dicamba and 5-OH dicamba expressed as dicamba.

DALT= days after the last application

dbp = days before planting

dbh = days before harvest

Animal feed stuffs

Soya bean forage and hay

Soya bean forage and hay samples were collected before the second application was made. Therefore, residues in these commodities came from pre-plant application.

Table 3 Residues of dicamba, 5-OH dicamba and DCSA in soya bean forage and hay from supervised trials conducted in the USA

SOYA BEAN Year, Location (variety) Trial No.	Application				No	DA LT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growth stage				Dicamba	5-OH dicamba	Total ^b	DCSA	
US GAP	480 SL	Spray	0.56 +/ or 1.12 (max 2.24/ season)	Pre-plant 7dbh	1	NA						
					1	7						
2010 York, NE (93Y12) Trial No. R100188	480 SL (DGA)	Pre-plant	0.56	pre-plant 28 dbp	1	66	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	66	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	66	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	66	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2010 York, NE (93Y12) Trial No. R100188	480 SL (DETA)	Pre-plant	0.56	pre-plant 28 dbp	1	66	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	66	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	66	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	66	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
Soybean USA, 2010 York, NE (93Y12) Trial No. R100188	600 SL (BAPMA)	Pre-plant	0.56	pre-plant 28 dbp	1	66	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	66	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	66	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	66	Hay	< 0.05	< 0.05	< 0.10	< 0.05	

SOYA BEAN Year, Location (variety) Trial No.	Application					DA LT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growt h stage	No			Dicamba	5-OH dicamba	Total ^b	DCSA	
2010 Turner, SD (Hefty 6238359) Trial No. R100190	480 SL (DGA)	Pre- plant	0.56	pre- plant 30 dbp	1	67	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	67	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	67	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	67	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2010 Turner, SD (Hefty 6238359) Trial No. R100190	480 SL (DETA)	Pre- plant	0.55	pre- plant 30 dbp	1	67	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	67	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	67	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	67	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2010 Turner, SD (Hefty 6238359) Trial No. R100190	600 SL (BAPMA)	Pre- plant	0.55	pre- plant 30 dbp	1	67	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	67	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	67	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	67	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2010 Guthrie, IA (93Y15) Trial No. R100191	480 SL (DGA)	Pre- plant	0.55	pre- plant 28 dbp	1	78	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	78	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	78	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	78	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2010 Guthrie, IA (93Y15) Trial No. R100191	480 SL (DETA)	Pre- plant	0.55	pre- plant 28 dbp	1	78	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	78	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	78	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	78	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2010 Guthrie, IA (93Y15) Trial No. R100191	600 SL (BAPMA)	Pre- plant	0.59	pre- plant 28 dbp	1	78	Forage	< 0.05	< 0.05	< 0.10	< 0.05	Norris, F.A. 2013 BASF# 389561 2012/700550 1
					1	78	Forage	< 0.05	< 0.05	< 0.10	< 0.05	
					1	78	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
					1	78	Hay	< 0.05	< 0.05	< 0.10	< 0.05	
2012 Wayne, NC (S78-G6) Trial No. R120089	480 SL (DGA)	Pre- plant	0.57	pre- plant 28 dbp	1	88	Forage	< 0.002	< 0.002	< 0.01	< 0.002	Norris, F.A. 2013 BASF# 430098 2013/700128 2
					1	88	Forage	< 0.01	< 0.002	< 0.01	< 0.01	
					1	88	Hay	< 0.002	< 0.002	< 0.01	< 0.002	
					1	88	Hay	< 0.002	< 0.002	< 0.01	< 0.002	
2012 Keokuk, IA (93Y82) Trial No. R120091	480 SL (DGA)	Pre- plant	0.56	pre- plant 29 dbp	1	62	Forage	< 0.01	< 0.002	< 0.01	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/700128 2
					1	62	Forage	0.01	0.03	0.04	0.02	
					1	62	Hay	< 0.002	< 0.002	< 0.01	< 0.002	
					1	62	Hay	< 0.002	< 0.002	< 0.01	< 0.002	
2012 Clinton, IL (S39-U2) Trial No. R120092	480 SL (DGA)	Pre- plant	0.59	pre- plant 28 dbp	1	70	Forage	< 0.01	< 0.002	< 0.01	< 0.002	Norris, F.A. 2013 BASF# 430098 2013/700128 2
					1	70	Forage	< 0.002	< 0.002	< 0.01	< 0.002	
					1	70	Hay	< 0.01	< 0.002	< 0.01	< 0.01	
					1	70	Hay	< 0.01	< 0.002	< 0.01	< 0.002	
2012 Walworth, WI (AG2031RR) Trial No. R120093	480 SL (DGA)	Pre- plant	0.56	pre- plant 28 dbp	1	67	Forage	< 0.01	< 0.01	< 0.03	0.01	Norris, F.A. 2013 BASF# 430098 2013/700128 2
					1	67	Forage	0.01	0.02	0.05	0.02	
					1	67	Hay	< 0.002	< 0.01	< 0.03	< 0.002	
					1	67	Hay	< 0.01	< 0.002	< 0.03	< 0.01	
2012 Boone, IA (93Y15) Trial No. R120094	480 SL (DGA)	Pre- plant	0.57	pre- plant 28 dbp	1	80	Forage	0.01	0.02	0.03	0.02	Norris, F.A. 2013 BASF# 430098 2013/700128 2
					1	80	Forage	0.01	< 0.002	0.01	0.01	
					1	80	Hay	< 0.01	< 0.002	< 0.01	< 0.002	
					1	80	Hay	< 0.01	< 0.002	< 0.01	< 0.002	

SOYA BEAN Year, Location (variety) Trial No.	Application					DA LT (d)	Portion analysed	Residues (mg/kg) ^a				Author Report Year Study No. Doc ID.
	Form.	Method	Rate (kg ai/ha)	Growt h stage	No			Dicamba	5-OH dicamba	Total ^b	DCSA	
2012 Guthrie, IA (93Y15) Trial No. R120095	480 SL (DGA)	Pre- plant	0.56	pre- plant 28 dbp	1	81	Forage	< 0.01	< 0.002	< 0.01	< 0.01	Norris, F.A. 2013 BASF# 430098 2013/700128 2
					1	81	Forage	< 0.01	< 0.002	< 0.01	< 0.002	
					1	81	Hay	0.01	< 0.002	0.01	< 0.01	
					1	81	Hay	0.06	< 0.002	0.06	< 0.01	

^a Expressed in mg dicamba equivalents/kg. Average values in parentheses.

^b Sum of dicamba and 5-OH dicamba expressed as dicamba.

dbp = days before planting

FATE OF RESIDUES IN STORAGE AND PROCESSING

The 2010 JMPR received and reviewed information on processing of soya bean to oil. The summary of processing studies on soya bean is transcribed below.

Table 4 Summary of processing studies

Processed Product	Processing factor	
	Dicamba	Total residues
Soya bean		
Meal	0.35	0.36
Hulls	3.9	3.8
Grain dust	676	669
Refined oil	< 0.019	< 0.036

APPRAISAL

Dicamba, a systemic broad-spectrum herbicide, was first evaluated by the 2010 JMPR which estimated an ADI of 0–0.3 mg/kg bw and ARfD of 0.5 mg/kg bw, and recommended the following residue definitions for plant commodities:

Definition of the residue for plant commodities (for compliance with the MRL): *Dicamba*

Definition of the residue for plant commodities (for estimation of dietary intake): *Sum of dicamba and 5-OH dicamba expressed as dicamba*

Definition of the residue for animal commodities (for compliance with the MRL and for estimation of dietary intake): *Sum of dicamba and DCSA expressed as dicamba*

The 2010 Meeting reviewed metabolism, method of analysis, storage stability, supervised residue trials and processing studies of dicamba in soya beans. However, as no supervised trials matched US GAP, the Meeting could not estimate a maximum residue level for soya bean (dry). The 2011 Meeting decided to apply the concept of proportionality to the residues from these trials to recommend a maximum residue level.

The Forty-fifth Session of the Codex Committee on Pesticide Residues in 2013 agreed to the Principles and Guidance for Application of the Proportionality Concept to Estimation of Maximum Residue Limits for Pesticides and further agreed that desiccants should be excluded from the application of the proportionality concept.

The current Meeting received information on new supervised trials on soya beans following the US GAP and analytical methods used for the determination of dicamba and related compounds in the new supervised trials on soya beans.

Method of analysis

The current Meeting received information on the analytical method used for the determination of dicamba, 5-OH dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA) arising in soya bean seeds, forage and hay in newly conducted supervised residue trials. The method analysed these compounds using LC-MS/MS, monitoring ion transitions from m/z 219→175 for dicamba; m/z 235→155 for 5-OH-dicamba; and m/z 205→161 for DCSA.

The method was found suitable for the determination of the three analytes in soya bean seeds, forage and hay with mean recoveries in the acceptable range of 70–110% at the fortification levels between 0.01 and 10 mg/kg.

The limit of quantitation (LOQ) is 0.01 mg/kg for the determination of dicamba, 5-OH dicamba and DCSA in soya bean seeds, forage and hay, except that this LOQ was not achievable in forage or hay for the 2010 season analysis.

Results of supervised residue trials on crops

Soya bean (dry)

A total of twelve supervised residue trials were conducted in the USA in 2010 and 2012.

The approved use of dicamba in soya bean in the USA consists of two different applications: application of up to 0.56 kg ai/ha as a broadcast application made approximately 14 days prior to planting, and/or up to 1.12 kg ai/ha applied to soya bean plants after soya bean pods have reached mature brown color and at least 75% leaf drop has occurred with a PHI of 7 days. If both pre-plant and pre-harvest applications are used in one season, the maximum seasonal use rate must not exceed 2.24 kg ai/ha. After a pre-harvest application soya bean fodder and hay must not be fed to animals.

After one pre-plant and one pre-harvest applications using the three different salt types, residues in the seed (dry) samples were primarily the parent compound and mostly relatively low (< 0.1 mg/kg). However, residue concentrations vary hugely, due possibly to the split of pods causing seeds to come into contact with dicamba itself or dicamba residue on the outer surface of pods. Since all the trials approximated normal agricultural practices, the Meeting decided to use these trial results in estimating a maximum residue level. No significant difference or pattern was observed among the trials using these different formulations.

Residues of dicamba in soya bean (dry) from those trials matching the GAP of the USA were, in rank order (n=12): < 0.01 (2), 0.01, 0.02, 0.025 (2), 0.035, 0.06, 0.12, 0.545, 5.4 and 5.6 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg to replace the previous recommendation of 5 mg/kg. For the purpose of calculating animal dietary burden, the Meeting estimated a median residue of 0.03 mg/kg.

Corresponding total residues (dicamba and 5-OH dicamba) of dicamba were (n=12): < 0.01, < 0.02, 0.02 (2), 0.025, 0.03, 0.035, 0.06, 0.13, 0.555, 5.635 and 6.06 mg/kg.

The Meeting estimated an STMR of 0.033 mg/kg.

Soya bean forage and hay

Soya bean forage and hay samples were collected before the second application was made to avoid abscission. Therefore, residues in these commodities came from a pre-plant application only. The US label prohibits the use of fodder or hay after a pre-harvest application.

The residues from the pre-plant application were expected to be very low as shown in the trials (mostly < 0.05 mg/kg and in one trial 0.06 mg/kg for dicamba) and, as such, residues in soya bean forage and hay from only pre-plant application would not have impacted on the livestock dietary burden. Therefore, the Meeting confirmed the decision of the 2010 and 2011 JMPR that there was no need for estimating a maximum residue level, median residue or highest residue for soya bean fodder and hay.

Fate of residues during processing

The 2010 JMPR received and reviewed information on processing of soya bean seed to oil and meal.

Processed Product	Processing factor		STMR/STMR-P
	Dicamba	Total residues	
Soya bean			0.033
Refined oil	< 0.019	< 0.036	0.001

As there is no concentration of dicamba and 5-OH dicamba observed in refined oil, the estimation of a maximum residue level is not necessary for this commodity.

On the basis of the processing factor of 0.35 for dicamba only, a median residue of 0.0105 mg/kg was calculated for soya bean meal, which may be used as a livestock feed item.

Residue concentration was observed in soya bean hulls and grain dust which may also be used as animal feeds. The processing factors of dicamba only, calculated for these commodities, were 3.9 and 676, respectively. From these factors, median residues in soya bean hulls and grain dust for the estimation of animal burden were calculated to be 0.117 and 20.3 mg/kg, respectively.

Residues in animal commodities

Soya beans and processed soya bean products may be fed to dairy cattle, beef cattle, broilers and layers. The maximum and mean dietary burdens were calculated using the highest residues and median residues of dicamba in commodities for which maximum residue levels were recommended by the 2010 JMPR and current JMPR and their processed products on a basis of the OECD Animal Feeding Table.

Residues of 5-OH dicamba was not included in the calculation of animal dietary burden as its concentrations in animal feeding items were very low and the feeding study with 5-OH dicamba resulted in very low uptake (< 0.01 mg/kg) of 5-OH dicamba into tissues, milk or blood of cattle at a dose equivalent to 59 ppm in the diet.

The resulting maximum and mean dietary burdens to be used for estimating maximum residue levels for commodities of animal origin (both mammals and poultry) were identical to those of the 2010 and 2011 JMPR.

The Meeting concluded that there was no need to re-evaluate maximum residue levels, STMRs or HRs for commodities of animal origin.

RECOMMENDATIONS

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

Definition of the residue for plant commodities (for compliance with the MRL): *Dicamba*.

Definition of the residue for plant commodities (for estimation of dietary intake): *Sum of dicamba and 5-OH dicamba expressed as dicamba*.

Definition of the residue for animal commodities (for compliance with MRLs and for estimation of dietary intake): *Dicamba and 3,6-dichlorosalicylic acid (DCSA) expressed as dicamba*.

Residue is not fat-soluble.

Commodity		Recommended MRL, mg/kg		STMR/STMR-P	HR/HR-P
CCN	Name	New	Previous	mg/kg	mg/kg
VD 0541	Soya bean (dry)	10	5	0.033	-
OR 0541	Soya bean oil, edible	-		0.001	-

For calculating animal dietary burdens

Commodity		Recommended MRL, mg/kg		STMR/STMR-P	HR/HR-P
CCN	Name	New	Previous	mg/kg ^a	mg/kg
	Soya bean (dry)			0.03	
	Soya bean meal			0.0105	
	Soya bean hull	-		0.117	-
	Soya bean grain dust	-		20.3	-

^a residues of dicamba only.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of dicamba were calculated for the 13 GEMS/Food cluster diets using STMRs and STMRPs estimated by the 2010 and current Meeting (see Annex 3 of the 2013 Report). The ADI is 0–0.3 mg/kg bw and the calculated IEDIs were 0–1% of the maximum ADI. The Meeting concluded that the long-term intake of residues of dicamba resulting from the uses considered by the current JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of dicamba were calculated for soya bean and its processed commodity using STMRs/STMR-Ps estimated by the current Meeting (see Annex 4 of the 2013 Report). The ARfD is 0.5 mg/kg and the calculated IESTIs were 0% of the ARfD. The Meeting concluded that the short-term intake of residues of dicamba, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

REFERENCES

Document No.	Author(s)	Year	Title, Report reference, Study number
	FAO/WHO	2010	Pesticide residues in food 2010 - Joint FAO/WHO Meeting on Pesticide Residues - Evaluations Part I-Residues
	FAO/WHO	2011	Pesticide residues in food 2011 - Joint FAO/WHO Meeting on Pesticide Residues -Report
2012/7005501	Norris, F.A	2013	Magnitude of the Residue of Dicamba in Soybean Matrices, Formulation Bridging Study (BASF Study Number 389561, American Agricultural Service Number AA100712)
2013/7001282	Norris, F.A	2013	Magnitude of the Residue of Dicamba in Soybean Matrices (BASF Study Number 430098, American Agricultural Service Number AA120705)