DICAMBA (240)

First draft prepared by Dr M Doherty, Environmental Protection Agency, Washington, USA

EXPLANATION

Dicamba is a systemic broad-spectrum herbicide. It was first evaluated by the JMPR in 2010 (T, R). The most recent residue evaluation was completed in 2013 (R).

The 2010 JMPR established an ADI for dicamba of 0–0.3 mg/kg bw and an ARfD of 0.5 mg/kg bw. The residue definition for compliance with the MRL for plant commodities is parent dicamba. The residue definition for dietary risk assessment for plant commodities is the sum of dicamba and 5-OH dicamba, expressed as dicamba. The residue definition for compliance with the MRL and dietary risk assessment for animal commodities is the sum of dicamba and DCSA, expressed as dicamba. The residue is not fat soluble.

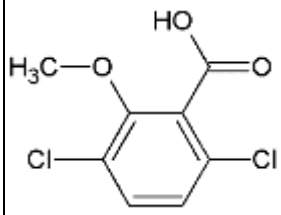
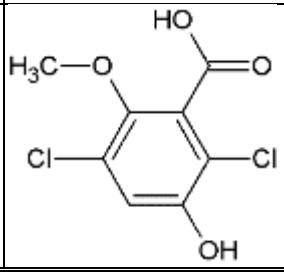
Dicamba was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. Studies submitted by the sponsor include nature of the residue, field trials, and processing studies in soya bean, maize, and cotton, and storage stability in soya bean and cotton.

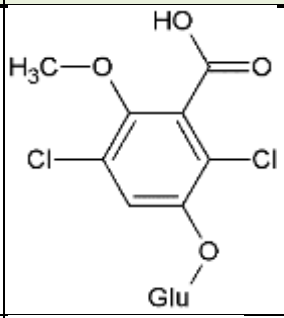
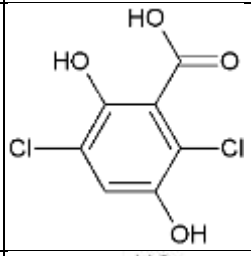
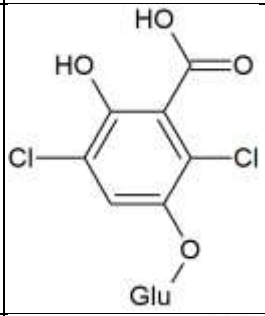
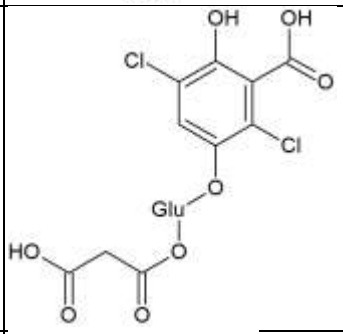
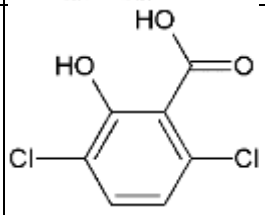
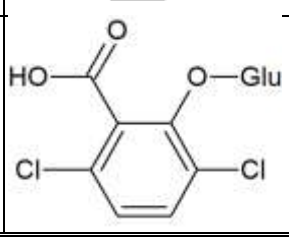
In this document, values in text are rounded to two significant figures; values in tables are presented as provided by the sponsor.

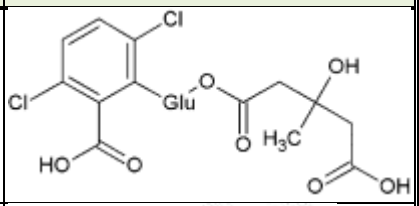
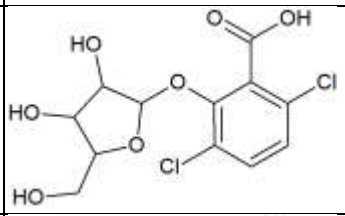
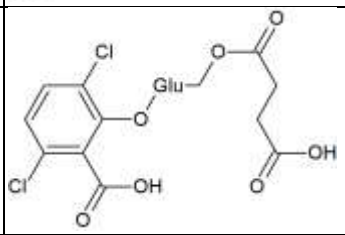
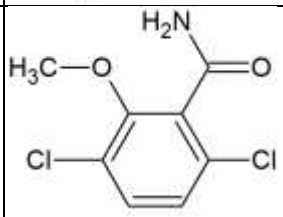
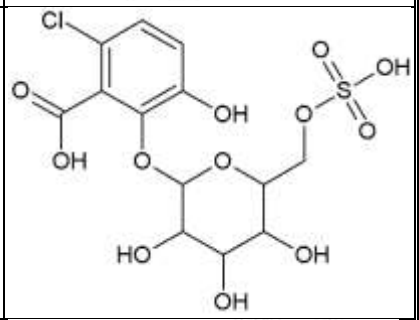
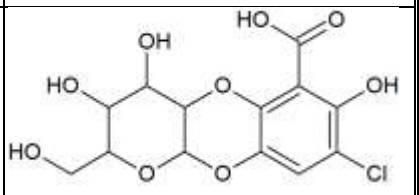
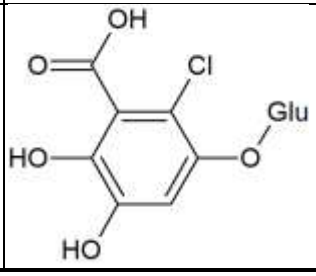
METABOLISM AND ENVIRONMENTAL FATE

The 2019 Extra Meeting received studies depicting the metabolism of dicamba by dicamba-tolerant soya bean, maize, and cotton. Tolerance is conveyed by expression of a dicamba mono-oxygenase protein system that oxidizes dicamba to DCSA. For all studies, dicamba was universally labelled in the phenyl ring, and applications were made either pre-emergence of the crop directly to the soil or post-emergence to the foliage. Control plants were interspersed amongst the treated plants and contained quantifiable levels of radioactivity, indicating potential volatilization and uptake of dicamba or volatile metabolites (e.g., $^{14}CO_2$).

Table 1 Metabolites and degradation products of dicamba observed in dicamba-tolerant crops

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
Dicamba	3,6-dichloro-2-methoxybenzoic acid $C_8H_6Cl_2O_3$ 221	
5-hydroxydicamba (5-OH Dicamba)	2,5-dichloro-3-hydroxy-6-methoxybenzoic acid $C_8H_6Cl_2O_4$ 237	

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
5-OH Dicamba Glucoside	2,5-dichloro-3-(β-D-glucopyranosyloxy)-6-methoxybenzoic acid $C_{14}H_{16}Cl_2O_9$ 399	
DCGA	2,5-dichloro-3,6-dihydroxybenzoic acid $C_7H_4Cl_2O_4$ 223	
DCGA Glucoside	2,5-dichloro-3-(β-D-glucopyranosyloxy)-6-hydroxybenzoic acid $C_{13}H_{14}Cl_2O_9$ 385	
DCGA Malonyl glucoside	3-[[6-O-(2-carboxyacetyl)-β-D-glucopyranosyloxy]-2,5-dichloro-6-hydroxybenzoic acid $C_{16}H_{16}Cl_2O_{12}$ 471	
DCSA	3,6-dichloro-2-hydroxybenzoic acid $C_7H_4Cl_2O_3$ 207	
DCSA Glucoside	3,6-dichloro-2-(β-D-glucopyranosyloxy) benzoic acid $C_{13}H_{14}Cl_2O_8$ 369	

Common or code name	Chemical name molecular formula molar mass, g/mol	Structure
DCSA HMG glucoside	2-[[6-O-(4-carboxy-3-hydroxy-3-methylbutyryl)-β-D-glucopyranosyl]oxy]-3,6-dichlorobenzoic acid $C_{19}H_{22}Cl_2O_{12}$ 513	
DCSA Pentoside	3,6-dichloro-2-(pentosyloxy)benzoic acid $C_{12}H_{12}Cl_2O_7$ 339	
DCSA Succinylglucoside	2-[[6-O-(3-carboxypropanoyl)-β-D-glucopyranosyl]oxy]-3,6-dichlorobenzoic acid $C_{17}H_{18}Cl_2O_{12}$ 485	
Dicamba Amide	3,6-dichloro-2-methoxybenzamide $C_8H_7Cl_2NO_2$ 220	
MCDHBA Glucoside Sulfate	6-Chloro-3-hydroxy-2-(3,4,5-trihydroxy-6-sulfooxymethyltetrahydro-pyran-2-yloxy)-benzoic acid $C_{13}H_{15}ClO_{12}S$ 431	
MCTHBA Cyclic Glucoside	7-Chloro-3,4,6-trihydroxy-2-hydroxymethyl-3,4,4a,9atetrahydro-2H-1,9.10-trioxaanthracene-5-carboxylic acid $C_{13}H_{13}ClO_9$ 349	
MCTHBA Glucoside	2-Chloro-5,6-dihydroxy-3-(β-D-glucopyranosyloxy)-benzoic acid $C_{13}H_{15}ClO_{10}$ 367	

Soya bean

The nature of the residue of dicamba in dicamba-tolerant soya bean was investigated by Miller and Mierkowski (2010; Report MSL0022659). Soya bean plants were grown in 12-inch pots in two greenhouses. Spray application of [phenyl-U-^{14}C]dicamba was made at a target rate of 2.8 kg ai/ha either on the day of planting (pre-emergence; PRE; n=29) or 29 days after planting at BBCH 60 (post-emergence; POE; n=32). Following treatment, the PRE and POE samples were placed in the same greenhouse but were physically separated. Pots with untreated control plants were interspersed amongst the treated plants for each group. Samples were collected as follows: Immature foliage 14 days after planting (PRE only), forage 36 (PRE) or 7 (POE) days after last application (DALA), hay 56 (PRE) or 27 (POE) DALA, and seed 112 (PRE) or 83 (POE) DALA. Samples were processed by grinding and were stored frozen.

Total radioactive residues (TRR) were determined by combustion/liquid scintillation counting (LSC). Residues were extracted with acetonitrile:water (2:3, v/v) from all matrices except seed, for which hexane was used first to extract oils. Seed was then extracted once with acetonitrile and then four times with acetonitrile:water (2:3, v/v). Unextracted radioactivity from the post-extraction solids (PES) was determined by combustion/LSC. Extracts were concentrated by rotary evaporation (analysis of the distillate indicated no significant amount of radioactivity was lost due to volatility). An aliquot of the concentrated aqueous extracts underwent hydrolysis (2 N HCl, ca. 100°C, 2 h). Aqueous extracts and hydrolysates were partitioned against ethyl acetate to assess partitioning behaviour. Specific residues were isolated using preparative HPLC analysis. Identification and characterization of metabolites was accomplished using HPLC-MS/MS as well as HPLC-UV and HPLC-RAD. Samples also underwent derivatization with trimethylsilyldiazomethane to discern the presence of carboxylic acid or phenolic groups and with acetic anhydride/pyridine to discern the presence of hydroxyl or phenolic groups. Finally, specific isolates underwent acid hydrolysis (1 N HCl, ca 100 °C, 1-2 hours), base hydrolysis (1 N NaOH, 58-65 °C, 5 hours) and ß-glucosidase digestion (37 °C, 69 hours) to identify/characterize conjugated metabolites. PES underwent further workup with dilute acid and base extraction, phosphate rinse, α-amylase digestion, protease digestion, EDTA extraction, oxidation with chlorite, hydrolysis with cellulase, and hydrolysis with KOH.

Total radioactive residues (extracted + unextracted) are presented in Table 1 (PRE) and Table 2 (POE). Control plants bore quantifiable levels of radioactivity, indicating potential volatilisation and uptake of dicamba or volatile metabolites. Levels were 0.08/0.28 mg eq/kg in PRE/POE forage and 0.17/0.14 mg eq/kg in PRE/POE seed.

Extractions with acetonitrile:water resulted in 91% TRR extracted from immature foliage, 91/94% from forage (PRE/POE), and 91/95% from hay (PRE/POE). Extraction efficiency was much lower for seed: 59% PRE and 64% POE (combined hexane, acetonitrile, and acetonitrile:water extractions). Further work with the PES showed that the vast majority of the unextracted radioactivity was associated with natural products (starch, lignin, cellulose, etc.).

A summary of extracted residues, PES, and hydrolysis and digestion products is shown in Tables 2 and 3.

Table 2 Summary of the nature of the residues in dicamba-tolerant soya bean following PRE application of dicamba (2.8 kg ai/ha)

	Immature foliage		Forage		Hay		Seed	
TRR, mg eq/kg	3.248		1.433		1.056		0.291	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed						13.87	0.04
Acetonitrile/Water extracts	91.09	2.959	91.21	1.307	90.88	0.96	59.35	0.173
Dicamba	Not Analysed		1.61	0.023	0.85	0.009	0.2	0.001
DCSA			3.19	0.046	1.54	0.016	0.37	0.001
DCSA Glucoside			74.48	1.067	70.81	0.748	11.55	0.034
DCSA HMGglucoside			5.21	0.075	6.67	0.07	8.73	0.025

TRR, mg eq/kg	Immature foliage		Forage		Hay		Seed	
	3.248		1.433		1.056		0.291	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Unk DCSA/DCGA Glucoside			0.55	0.008	0.51	0.005	ND	ND
Unk DCSA/DCGA Conj.			1.26	0.018	1.64	0.017	0.75	0.002
DCGA Glucoside			1.14	0.016	3.45	0.036	1.6	0.005
DCGA Malonyl glucoside			1.4	0.02	0.73	0.008	4.73	0.014
Sugars			0.96	0.014	1.08	0.011	8.42	0.025
Total Unknowns (n=11)[a]			1.39 [0.58]	0.02 [0.008]	3.59 [1.07]	0.038 [0.011]	4.09 [1.26]	0.013 [0.004]
Total Identified or Characterized Metabolites			89.81	1.287	87.29	0.922	50.21	0.146
PES	8.91	0.289	8.79	0.126	9.12	0.096	40.65	0.118
Phosphate	Not Analysed						0.25	0.001
Starch							1.29	0.004
Protein							10.06	0.029
Pectin							3.26	0.009
Cellulose							3.75	0.011
Hemicellulose							13.92	0.04
Unextracted							7.09	0.021

ND = Not detected

[a] Maximum individual values listed in brackets

Table 3 Summary of the nature of the residues in dicamba-tolerant soya bean following POE application of dicamba (2.8 kg ai/ha)

TRR, mg eq/kg	Forage		Hay		Seed	
	134.147		39.149		0.389	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed				10.76	0.042
Acetonitrile/Water extracts	93.79	125.811	95.3	37.308	63.67	0.248
Dicamba	24.21	32.473	12.33	4.828	0.64	0.003
DCSA	4.08	5.473	1.93	0.757	0.46	0.002
DCSA Glucoside	60.32	80.913	67.26	26.333	15.27	0.059
DCSA HMGglucoside	1.14	1.535	2.48	0.97	9.61	0.037
Unk DCSA/DCGA Glucoside	0.12	0.164	ND	ND	ND	ND
Unk DCSA/DCGA Conj.	0.38	0.503	1.75	0.686	0.62	0.002
DCGA Glucoside	0.75	1.007	4.32	1.69	2.07	0.008
DCGA Malonyl glucoside	1.11	1.485	1.61	0.631	4.64	0.018
Sugars	ND	ND	0.49	0.19	9.15	0.036
Total Unknowns (n=11)[a]	1.52 [0.4]	2.038 [0.541]	3.13 [0.95]	1.227 [0.373]	4.27 [0.84]	0.0144 [0.003]
Total Identified or Characterized Metabolites	92.1	123.552	92.17	36.085	53.21	0.207
PES	6.21	8.336	4.7	1.841	36.33	0.141
Phosphate	Not Analysed				1.42	0.006
Starch					1.27	0.005
Protein					8.18	0.032
Pectin					2.78	0.011
Cellulose					3.46	0.013
Hemicellulose					12.9	0.05
Unextracted					7.6	0.03

ND = Not detected

[a] Maximum individual values listed in brackets

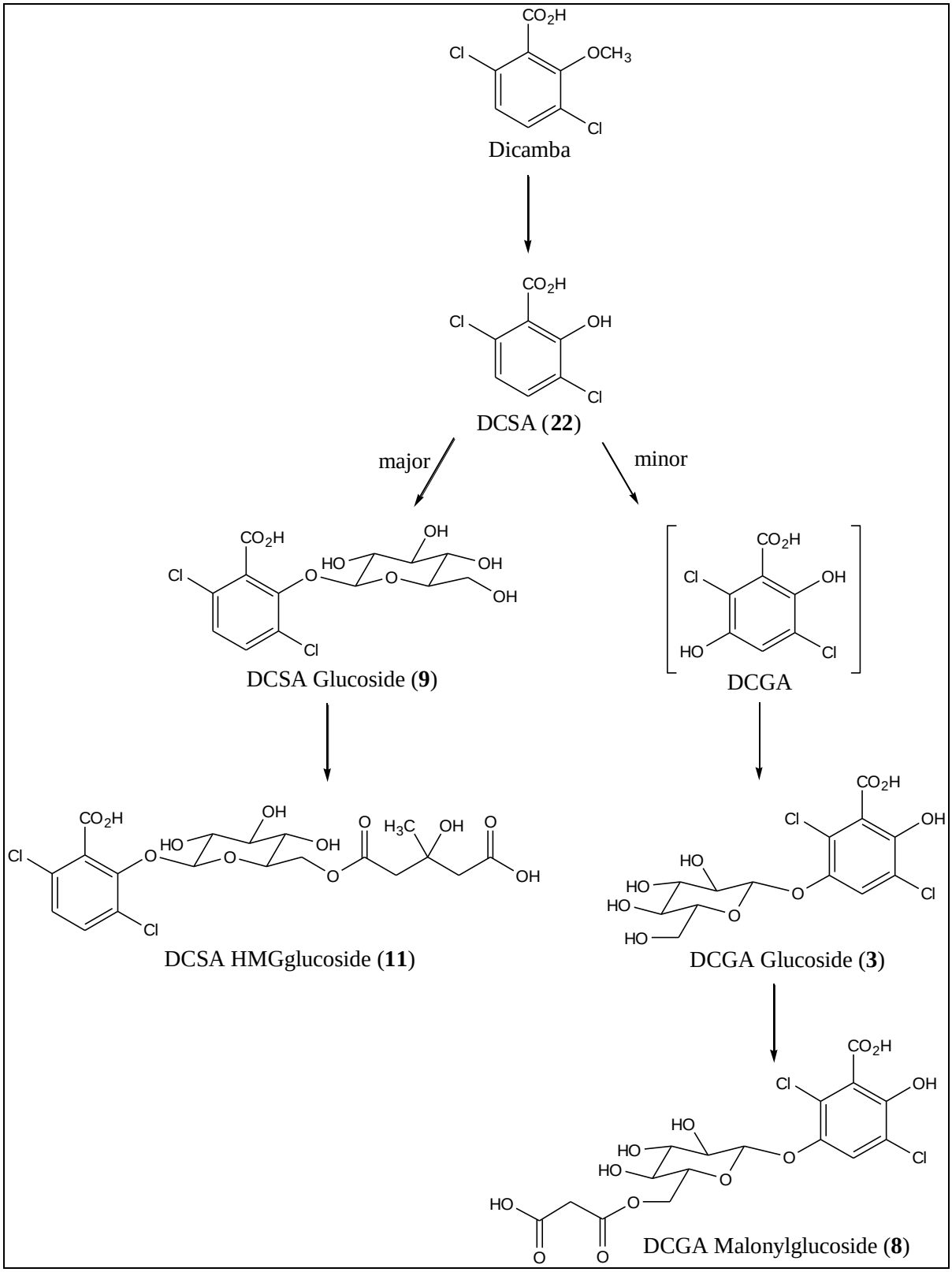


Figure 1 Proposed metabolic pathway of dicamba in dicamba-tolerant soya bean. (Provided by the sponsor)

Maize

The nature of the residue of dicamba in dicamba-tolerant maize was investigated by Adio and Feng (2015; Report MSL0025703). Maize plants were grown in small, outdoor box plots. Application of [phenyl-U-^{14}C]dicamba was made at a target rate of 2.24 kg ai/ha either on the day of planting (pre-emergence; PRE; n=42) or 30 days after planting (post-emergence; POE; n=42). Samples were collected as follows: Immature foliage 19 days after planting (PRE only), forage 80 (PRE) or 50 (POE) days after application, and stover and grain 114 (PRE) or 84 (POE) days after application. Samples were ground and then further homogenized by cryomilling.

Total radioactive residues (TRR) were determined by combustion/LSC. Residues were extracted with acetonitrile:water (2:3, v/v) from all matrices except grain, for which hexane was used first to extract oils (POE sample only). The remaining material was then rinsed with methanol and then extracted with acetonitrile:water (2:3, v/v). Unextracted radioactivity from the PES was determined by combustion/LSC. Extracts were concentrated by rotary evaporation (analysis of the distillate indicated no significant amount of radioactivity was lost due to volatility). An aliquot of the concentrated aqueous extracts underwent hydrolysis (2 N HCl, ca. 100 °C, 2 hours). Aqueous extracts and hydrolysates were partitioned against ethyl acetate to assess partitioning behaviour. Specific residues were isolated using preparative HPLC analysis. Identification and characterization of metabolites was accomplished using HPLC-MS/MS as well as HPLC-UV and HPLC-RAD. Samples also underwent derivatization with trimethylsilyldiazomethane to discern the presence of carboxylic acid or phenolic groups and with acetic anhydride/pyridine to discern the presence of hydroxyl or phenolic groups. Finally, specific isolates underwent acid hydrolysis (1 N HCl, ca 100 °C, 1-2 hours), base hydrolysis (1 N NaOH, ambient temperature, 1 h) and ß-glucosidase digestion (37 °C, 48 hours) to identify/characterize conjugated metabolites. PES underwent further workup with dilute acid and base extraction, phosphate rinse, α-amylase digestion, protease digestion, EDTA extraction, oxidation with chlorite, hydrolysis with cellulase, and hydrolysis with KOH.

Total radioactive residues (extracted + unextracted) from PRE application were 4.5 mg eq/kg in immature foliage, 0.075 mg eq/kg in forage, 0.24 mg eq.kg in stover, and 0.043 mg eq/kg in grain. Following POE application, TRR were 2.2 mg eq/kg in forage, 7.8 mg eq/kg in stover, and 0.062 mg eq/kg in grain. Radioactivity was not quantifiable in control plant matrices.

Extractions with acetonitrile:water resulted in 89% TRR extracted from immature foliage, 76/86% from forage (PRE/POE), and 74/83% from stover (PRE/POE). Extraction efficiency was much lower for grain: 7.4% PRE and 13% POE; inclusion of residues extracted into hexane gives 18% extracted (POE). Of the extracted radioactivity, most was associate with sugars and organic acids (9.4% TRR) and the majority of the remainder was in the form of dicamba-specific residues (e.g., dicamba and free and conjugated forms of 5-hydroxydicamba, DCSA, and DCGA). Further work with the PES showed that the vast majority of the unextracted radioactivity was associated with natural products (starch, lignin, cellulose, etc.).

A summary of extracted residues, PES, and hydrolysis and digestion products is shown in Tables 4 and 5.

Table 4 Summary of the nature of the residues in dicamba-tolerant maize following PRE application of dicamba

TRR, mg eq/kg	Immature foliage		Forage		Stover		Grain	
	4.513		0.075		0.243		0.043	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed						4.64	0.002
Acetonitrile/water extracts	89.13	4.02	76.64	0.057	74.71	0.181	7.36	0.005
Dicamba	3.42	0.154	NQ	NA	0.15	0.0004	NQ	NA
5-OH Dicamba	1.09	0.049	2.01	0.001	1.82	0.004	0.02	<0.0001
5-OH Dicamba Glucoside	1.02	0.046	NQ	NA	0.08	0.0002	NQ	NA
DCSA	3.75	0.169	0.23	0.0002	4.35	0.011	NQ	NA
DCSA Glucoside	53.17	2.4	41.56	0.031	30.52	0.074	0.10	<0.0001
DCSA Pentoside/Unk DCGA Conj.	4.51	0.204	4.87	0.004	5.39	0.013	0.04	<0.0001

TRR, mg eq/kg	Immature foliage		Forage		Stover		Grain	
	4.513		0.075		0.243		0.043	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
DCSA Succinylglucoside	NQ	NA	2.49	0.002	2.97	0.007	0.03	<0.0001
DCGA Glucoside/DCGA Pentosylglucoside	11.2	0.505	3.27	0.002	2.35	0.006	0.14	≤0.0001
Unk DCSA/DCGA Conj.	1.22	0.055	1.46	0.001	2.15	0.005	0.04	<0.0001
Unk DCSA/DCGA Conj.	0.16	0.007	1.17	0.0009	1.68	0.004	NQ	NA
Unk DCSA/DCGA Conj.	0.35	0.016	1.12	0.0008	1.47	0.004	NQ	NA
MCTHBA Glucoside	1.71	0.077	0.60	0.0004	1.43	0.003	0.03	<0.0001
MCTHBA Cyc Glucoside/DCSA HMGglucoside	3.07	0.139	2.41	0.002	2.94	0.007	0.04	<0.0001
Unk MCDHBA conj.	0.64	0.029	0.74	0.0006	0.57	0.001	0.04	<0.0001
Nat. prod. Organic Acids	1.74	0.079	2.43	0.002	0.95	0.002	0.22	0.0001
Sugars	1.12	0.051	6.23	0.005	6.37	0.015	2.50	0.0011
Total Unknowns (n≤12)[a]	0.96 [0.96]	0.043 [0.043]	5.99 [1.76]	0.0041 [0.001]	8.31 [1.96]	0.021 [0.005]	0.09 [0.05]	<0.0003 [<0.0001]
Total Identified or Characterized Metabolites	88.17	3.98	70.59	0.0529	65.19	0.1566	7.84	0.0032
PES	10.88	0.491	22.67	0.017	25.10	0.061	88.37	0.038
Phosphate	Not Analysed		1.80	0.001	3.19	0.008	1.21	0.001
Starch			6.36	0.005	6.76	0.016	21.48	0.009
Protein			1.31	0.001	1.62	0.004	11.51	0.005
Pectin			0.78	0.001	1.09	0.003	3.32	0.001
Lignin			2.31	0.002	3.85	0.009	6.61	0.003
Cellulose			6.27	0.005	5.55	0.013	26.72	0.011
Hemicellulose			4.27	0.003	2.22	0.005	16.67	0.007
Unextracted			0.27	0.0002	1.00	0.0024	0.49	0.0002

NQ = Not Quantified; there was insufficient material to quantify the amount of residue present in the sample.

NA = Not Applicable

[a] Maximum individual values listed in brackets

Table 5 Summary of the nature of the residues in dicamba-tolerant maize following POE application of dicamba

TRR, mg eq/kg	Forage		Stover		Grain	
	2.228		7.826		0.062	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not Analysed				5.31	0.003
Acetonitrile/water extracts	85.82	1.912	83.28	6.518	12.78	0.008
Dicamba	8.64	0.193	6.27	0.491	0.01	<0.0001
5-OH Dicamba	3.01	0.067	3.85	0.301	0.11	≤0.0001
5-OH Dicamba Glucoside	0.36	0.008	0.56	0.044	0.04	<0.0001
DCSA	1.6	0.036	5.09	0.398	0.03	<0.0001
DCSA Glucoside	33.46	0.745	26.94	2.108	0.44	0.0003
DCSA Pentoside/Unk DCGA Conj.	3.65	0.081	3.43	0.269	0.20	0.0001
DCSA Succinylglucoside	3.62	0.081	4.26	0.333	0.12	≤0.0001
DCGA Glucoside/DCGA Pentosylglucoside	3.75	0.084	2.47	0.193	0.39	0.0002
Unk DCSA/DCGA Conj.	2.14	0.048	2.56	0.20	0.16	≤0.0001
Unk DCSA/DCGA Conj.	1.89	0.042	2.36	0.184	0.03	<0.0001
Unk DCSA/DCGA Conj.	1.58	0.035	1.88	0.147	0.04	<0.0001
MCTHBA Glucoside	2.32	0.052	1.87	0.146	0.09	≤0.0001
MCTHBA Cyc Glucoside/DCSA HMGglucoside	3.73	0.083	4.09	0.32	0.14	≤0.0001
Unk MCDHBA conj.	1.23	0.027	1.33	0.104	0.06	<0.0001
Nat. prod. Organic Acids	2.39	0.053	2.61	0.204	1.05	0.0006
Sugars	1.11	0.025	1.49	0.117	3.06	0.0019
Total Unknowns (n≤12)[a]	10.3 [2.27]	0.229 [0.05]	11.5 [2.66]	0.901 [0.208]	0.37 [0.1]	<0.0007 [≤0.0001]
Total Identified or Characterized Metabolites	74.48	1.66	71.06	5.559	11.28	0.0061
PES	14.18	0.316	16.71	1.308	80.65	0.05
Phosphate	2.04	0.045	2.59	0.20	1.67	0.001
Starch	6.43	0.143	7.20	0.56	18.62	0.011

TRR, mg eq/kg	Forage		Stover		Grain	
	2.228		7.826		0.062	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Protein	1.04	0.023	1.61	0.13	8.32	0.005
Pectin	1.05	0.023	1.15	0.09	3.09	0.002
Lignin	2.29	0.051	2.57	0.20	6.70	0.004
Cellulose	0.74	0.017	0.75	0.06	26.59	0.016
Hemicellulose	0.57	0.013	0.76	0.06	15.91	0.01
Unextracted	0.02	0.0005	0.09	0.007	0.69	0.0004

[a] Maximum values listed in brackets

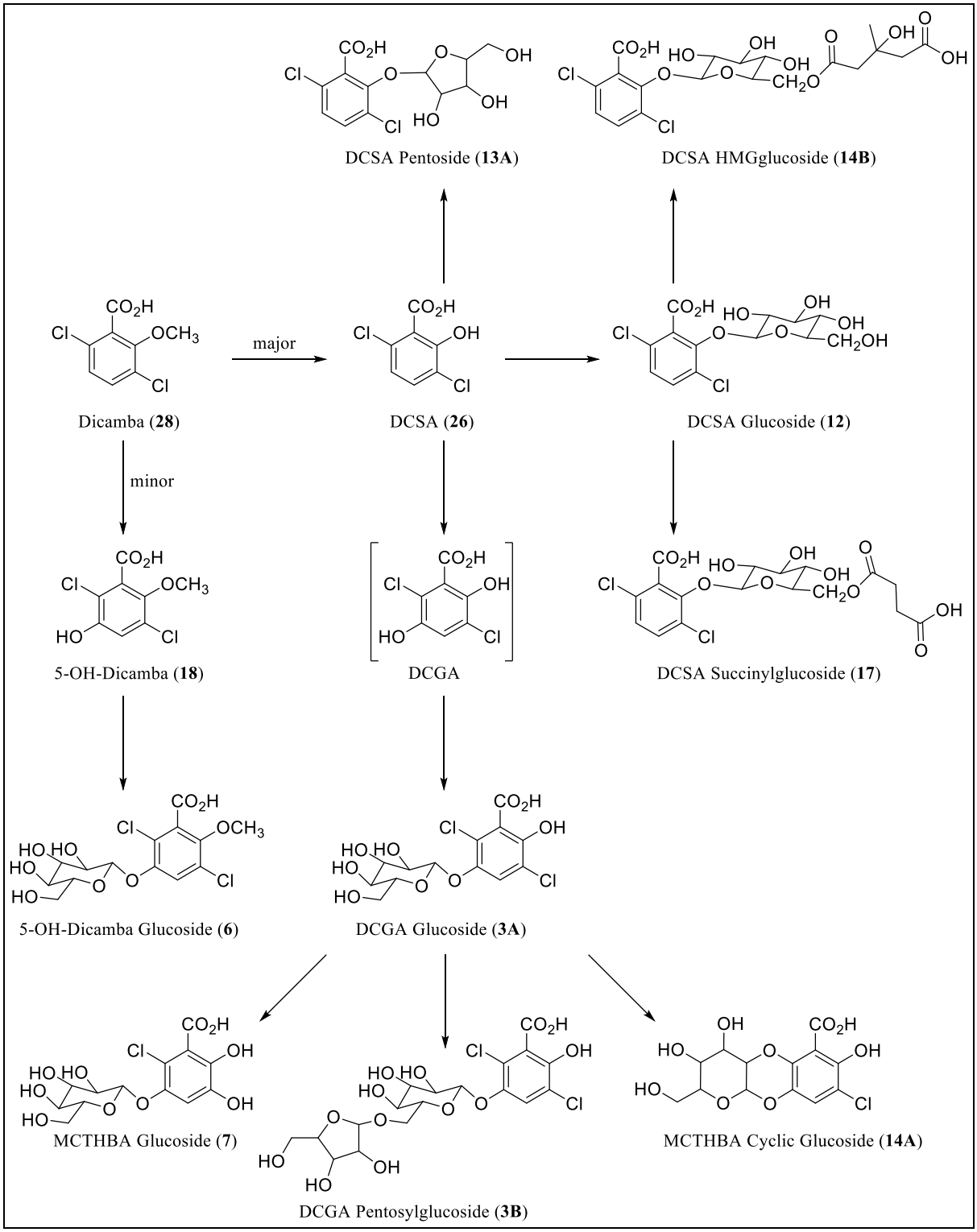


Figure 2 Proposed metabolic pathway of dicamba in dicamba-tolerant maize. (Provided by the sponsor)

Cotton

The nature of the residue of dicamba in dicamba-tolerant cotton was investigated by Whitehead *et al*. (2011; Report MSL0023760). Cotton plants were grown in small, outdoor box plots (~0.84 m^2). Application of [phenyl-U-^{14}C]dicamba was made at a target rate of 2.24 kg ai/ha either on the day of planting (pre-emergence; PRE) or 76 days after planting (post-emergence; POE). Samples of seed cotton (seeds + lint), leaves, and stems were collected 180/104 days after the PRE/POE treatments.

Leaves and stems served as surrogate gin trash samples. The gin trash samples were ground and frozen. Seed cotton samples were ginned to produce undelinted seed, which was homogenized by cryomilling.

Total radioactive residues (TRR) were determined by combustion/LSC. Residues were extracted from gin trash with acetonitrile:water (2:3, v/v). Samples of undelinted seed were extracted sequentially with hexane (three times), acetonitrile (one time), and acetonitrile:water (2:3, v/v; four times). The remaining material was then rinsed with methanol and then extracted with acetonitrile:water (2:3, v/v). Unextracted radioactivity from the PES was determined by combustion/LSC. Extracts were concentrated by rotary evaporation (analysis of the distillate indicated no significant amount of radioactivity was lost due to volatility). An aliquot of the concentrated aqueous extracts underwent hydrolysis (2 N HCl, approximately 100 °C, 2 hours or 2 N NaOH, 60 °C, 4 hours). Aqueous extracts and hydrolysates were partitioned against ethyl acetate to assess partitioning behaviour. Specific residues were isolated using preparative HPLC analysis. Identification and characterization of metabolites was accomplished using HPLC-MS/MS as well as HPLC-UV and HPLC-RAD. Samples also underwent derivatization with trimethylsilyldiazomethane to discern the presence of carboxylic acid or phenolic groups and with acetic anhydride/pyridine to discern the presence of hydroxyl or phenolic groups. Finally, specific isolates underwent acid hydrolysis (1 N HCl, approximately 100 °C, 1–2 hours) and base hydrolysis (1 M NaOH, ambient temperature, 1 hour). PES underwent further workup with dilute acid and base extraction, phosphate rinse, α-amylase digestion, protease digestion, EDTA extraction, oxidation with chlorite, hydrolysis with cellulase, and hydrolysis with KOH.

Total radioactive residues (extracted + unextracted) from PRE application were 0.85 mg eq/kg in gin trash and 0.16 mg eq/kg in undelinted seed. Following POE application, TRRs were 60 mg eq/kg in gin trash and 0.98 mg eq/kg in undelinted seed. Low levels of radioactivity (0.003–0.005 mg eq/kg) were observed in the control boxes, which were approx. 1.8 and 30 m from the PRE and POE treated boxes, respectively. This may be evidence of volatilisation and uptake of dicamba or volatile metabolites from treated plants/boxes to the control plants.

The extraction schemes resulted in 76/71% TRR extracted from gin trash (PRE/POE) and 31/38% from undelinted seed (PRE/POE). Of the material extracted from undelinted seed, 20% and 12% was associated with the hexane fraction for PRE and POE samples, respectively. Further work with the PES showed that the vast majority of the unextracted radioactivity was associated with natural products (starch, lignin, cellulose, etc.).

A summary of extracted residues, PES, and hydrolysis and digestion products is shown in Table 6.

Table 6 Summary of the nature of the residues in dicamba-tolerant cotton following PRE and POE application of dicamba

	PRE				POE			
	Gin trash		Seed		Gin trash		Seed	
TRR, mg eq/kg	0.8493		0.1621		60.0235		0.9778	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Triglycerides (from the hexane-extracted oil fraction)	Not analysed		14.41	0.023	Not analysed		8.8	0.086
Acetonitrile/Water extracts	76.24	0.648	30.84	0.05	71.28	42.785	38.26	0.374
Dicamba	0.5	0.004	0.09	0.0001	4.48	2.691	0.85	0.008
Dicamba Amide	3.22	0.027	0.23	0.0004	ND	ND	ND	ND
DCSA	5.42	0.046	0.06	0.0001	13.39	8.035	1.91	0.019
DCSA Glucoside	27.77	0.236	0.73	0.0012	16.83	10.101	3.42	0.033
DCGA Glucoside	0.68	0.006	0.09	0.0001	2.46	1.476	1.1	0.011
MCTHBA Glucoside [A]	ND	ND	ND	ND	3.33	1.998	0.53	0.005
MCTHBA Cyc Glc	2.94	0.025	ND	ND	2.76	1.656	ND	ND
MCDHBA Glucoside Sulfate	ND	ND	ND	ND	4.71	2.828	ND	ND
MCDHBA Glucoside [a]	ND	ND	ND	ND	0.84	0.505	0.72	0.002
Sugars	8.94	0.076	4.59	0.0074	2.69	1.613	5.61	0.008
Total Unknowns (n=27)[b]	15.97 [2.91]	0.135 [0.025]	4.42 [0.9]	0.0045 [0.0007]	0.86 [0.86]	0.518 [0.518]	4.81 [0.81]	0.049 [0.008]

TRR, mg eq/kg	PRE				POE			
	Gin trash		Seed		Gin trash		Seed	
	0.8493		0.1621		60.0235		0.9778	
Identification	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Identified or Characterized Metabolites	49.49	0.42	19.96	0.032	51.49	30.903	21.69	0.165
PES	23.76	0.202	69.16	0.112	28.72	17.239	61.74	0.604
Phosphate	3.25	0.028	3.87	0.006	6.2	3.72	1.96	0.019
Starch	2.73	0.023	2.79	0.005	7.05	4.234	2.19	0.021
Protein	1.71	0.015	17.71	0.029	2.25	1.35	7.43	0.073
Pectin	1.91	0.016	7.4	0.012	2.66	1.595	4.04	0.039
Lignin	5.28	0.045	6.18	0.01	6.6	3.962	8.43	0.082
Cellulose	0.9	0.008	4.37	0.007	0.62	0.374	3.13	0.031
Hemicellulose	5.35	0.045	14.43	0.023	2.8	1.679	13.49	0.132
Sulfuric acid	Not analysed		5.07	0.008	Not analysed		3.21	0.031
Protein (2nd)			NA	NA			2.67	0.026
Hot DMSO			NA	NA			5.57	0.054
Unextracted	2.62	0.022	7.35	0.012	0.61	0.365	9.66	0.094

ND = Not Found

[a] Identity not confirmed

[b] Maximum individual values listed in brackets

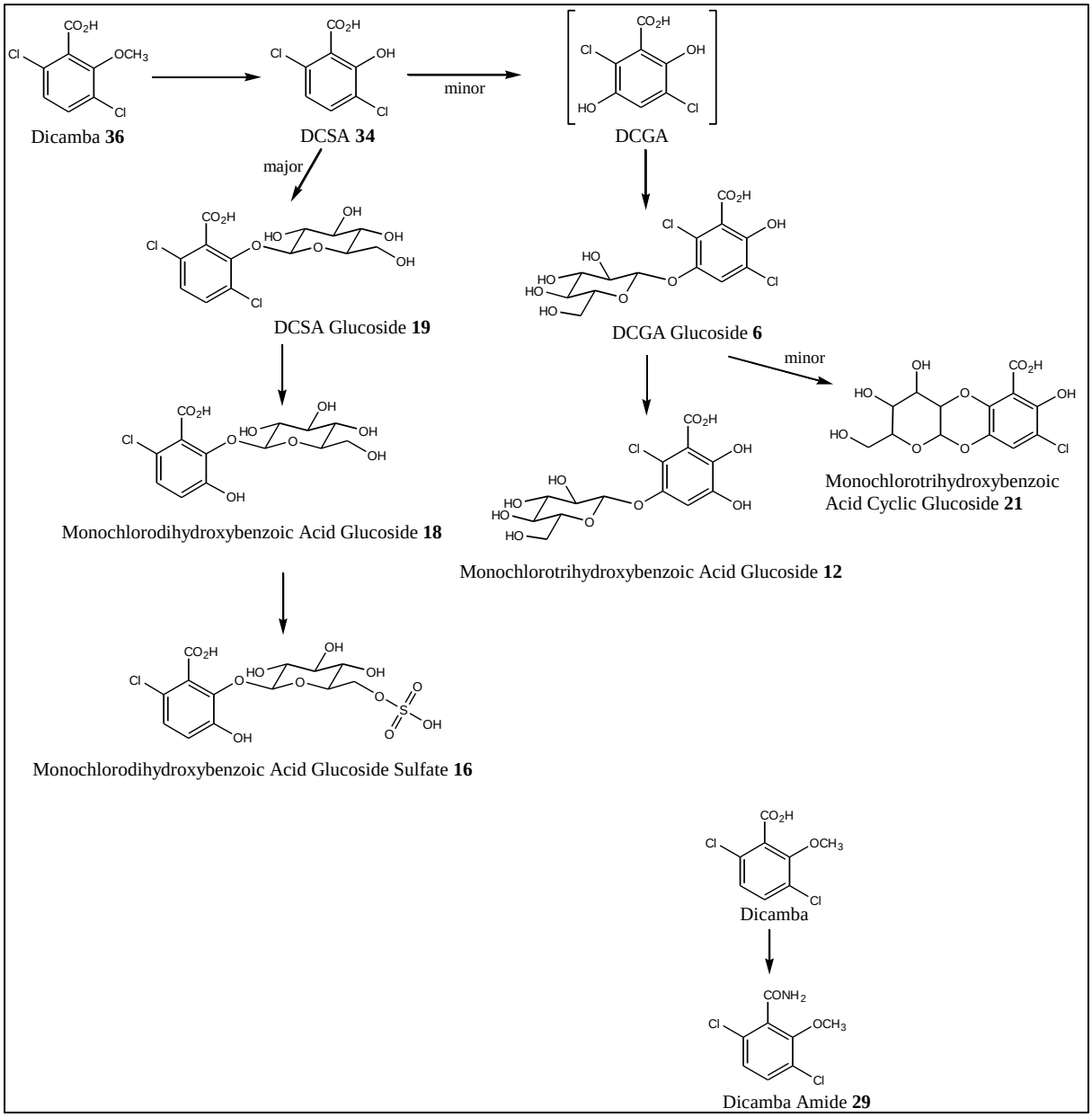


Figure 3 Proposed metabolic pathway of dicamba in dicamba-tolerant cotton. (Provided by the sponsor)

In summary, metabolism of dicamba by the dicamba-tolerant varieties reported above is similar. Demethylation of dicamba results in the formation of DCSA, which is subsequently either conjugated with glucose or hydroxylated to form DCGA, which also undergoes glucose conjugation. The predominant residue is free and glucose-conjugated DCSA, with most of those residues occurring in the conjugated form. In seeds/grain, a large percentage of the radioactive residue was shown to be incorporation of the radiolabelled carbon into natural plant constituents.

RESIDUE ANALYSIS

Analytical Methods

The Meeting received three methods for the analysis of dicamba, one each for dicamba-tolerant soya bean (AG-ME-1321-01), dicamba-tolerant maize (ME-1713), and dicamba-tolerant cotton (AG-ME-1381-01). All three methods are essentially the same and involve extraction of residues into acetonitrile:water, hydrolysis of conjugated metabolites to their free form by 1N HCl digestion at 95 °C

for 1 hour (AG-ME-1321-01, AG-ME-1381-01) or 1.5 hours (ME-1713), clean-up by liquid-liquid partitioning with ethyl acetate:isooctane (1:4, v/v ME-1713 and AG-ME-1381-01; 2:3, v/v AG-ME-1321-01), addition of ^{13}C-labelled internal standards of dicamba, 5-OH dicamba, DCSA, and DCGA, and analysis by LC-MS/MS. The hydrolysis step is similar to that used in the metabolism studies (2N HCl, 100 °C, 2 hours). For soya bean and cotton seed, the method LOQ was determined as the lowest fortification level giving acceptable accuracy (70-120% recovery) and precision (CV ≤ 20%) at that level and all greater levels; for other matrices, the LOQ was determined as the lowest limit of method validation (LLMV). When accuracy and precision were acceptable, but the number of replications was <5, the LOQ was considered to be the fortification level that brought the total number of replicates to at least 5 (see, for example, Table 7, defatted flour).

Soya bean

The analytical method for residues of dicamba in soya bean matrices was validated in seed, forage, hay, and numerous processed commodities (Table 7). Recoveries from samples fortified at 0.01 mg/kg were generally acceptable, with a few exceptions (e.g., 5-OH dicamba in soya bean seed). For some analytes and matrices, acceptable recovery was observed at the 0.005 mg/kg fortification level.

Table 7 Summary of recoveries of dicamba and metabolites from fortified soya bean matrices

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Seed	0 (Control) [a]	7	ND	ND	ND [b]	<0.001
	0.005	7	103 (7.35)	107 (5.18)	63.7 (11.1)	88.0 (5.54)
	0.01	7	99.8 (5.85)	106 (3.55)	66.9 (9.19)	95.9 (2.88)
	0.02	7	97.9 (4.81)	105 (2.39)	71.3 (6.53)	97.4 (3.52)
	0.1	7	97.1 (2.97)	104 (2.12)	102 (2.25)	101 (2.76)
	2	2	87.8	97.3	104	87.7
Forage	0 (Control) [a]	7	ND [b]	0.0014	ND	ND
	0.005	7	97.9 (7.49)	72.6 (5.57)	102 (5.22)	105 (9.30)
	0.01	7	99.3 (4.60)	80.9 (3.10)	110 (7.75)	101 (9.45)
	0.02	7	99.8 (3.52)	88.0 (2.77)	108 (2.42)	100 (5.83)
	0.1	7	105 (2.13)	90.0 (2.51)	106 (3.36)	105 (5.11)
	2	2	95.8	97.4	88.5	92.8
Hay	0 (Control) [a]	7	ND	ND	ND [b]	<0.001
	0.005	7	ND [c]	97.4 (4.55)	77.4 (8.73)	68.4 (2.69)
	0.01	7	86.3 (6.56)	105 (2.94)	80.0 (6.67)	82.9 (3.00)
	0.02	7	95.9 (10.5)	106 (2.01)	84.0 (9.13)	90.5 (1.82)
	0.1	7	105 (4.05)	107 (2.78)	102 (3.94)	100 (3.88)
	2	2	72.5	88.0	89.8	90.0
Hulls	0 (Control) [a]	3	ND [b]	<0.001	<0.001	<0.001
	0.01	2	92.5	115	74.4	78.4
	0.02	4	92.8 (6.80)	109 (1.42)	74.9 (15.3)	86.2 (8.39)
	0.05	3	96.1 (3.81)	104 (8.09)	74.8 (29.3)	88.3 (13.0)
	0.2	1	96.5	99.5	100	86.8
	0.4	2	101	98.3	91.6	90.9
	1.5	1	106	97.9	101	97.9
Defatted flour	0 (Control) [a]	4	<0.001	<0.001	0.007	<0.001
	0.01	2	79.3	102	31.0	83.0
	0.02	2	78.2	98.2	64.4	99.9
	0.05	1	96.3	103	84.3	84.8
	0.2	1	94.1	94.0	91.5	91.0
	0.4	3	103 (5.82)	104 (7.11)	99.4 (2.57)	96.0 (6.10)
	2	2	81.9	89.2	93.5	97.7
	3	1	97.6	92.3	98.5	96.3
Defatted meal	0 (Control) [a]	3	ND [b]	<0.002	<0.001	<0.001
	0.01	2	88.7	92.9	70.7	81.8
	0.02	4	97.5 (5.42)	105 (11.8)	86.6 (10.8)	92.8 (7.42)
	0.05	3	92.8 (10.8)	107 (7.70)	86.5 (9.74)	92.0 7.85)
	0.2	1	94.0	101	101	83.2
	0.4	2	96.7	102	85.7	92.9
	1.5	1	88.0	93.8	95.3	104

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Protein isolate	0 (Control) [a]	4	ND [b]	<0.001	ND	<0.001
	0.01	2	80.5	89.4	86.8	79.3
	0.02	2	87.1	94.7	94.1	82.9
	0.4	2	81.7	92.7	97.7	89.2
	0.8	2	87.1	100	98.0	93.6
	1.5	1	88.1	92.6	101	86.7
Protein concentrate	0 (Control) [a]	3	ND [b]	<0.001	ND	<0.001
	0.01	2	81.0	96.3	80.7	82.7
	0.02	1	90.1	103	84.8	89.2
	0.2	1	90.1	105	106	97.1
	0.4	2	114	97.3	100	95.9
	1.5	1	94.3	90.6	105	89.3
Crude lecithin	0 (Control) [a]	8	<0.001	<0.001	<0.001	ND [b]
	0.01	3	83.5 (18.1)	93.6 (4.90)	84.1 (7.93)	71.3 (7.54)
	0.2	3	86.1 (10.7)	96.7 (8.15)	98.9 (4.05)	78.8 (2.04)
	0.4	2	107	107	107	90.8
	2	2	107	108	103	101
Degummed oil	0 (Control) [a]	4	ND [b]	<0.001	<0.001	<0.001
	0.01	3	98.5 (3.61)	96.0 (3.19)	84.9 (2.65)	70.8 (14.5)
	0.02	3	93.5 (5.16)	99.1 (2.42)	87.7 (2.20)	77.9 (19.6)
	0.05	2	86.8	101	93.7	82.2
	0.4	3	96.5 (12.1)	104 (3.49)	99.3 (8.38)	92.6 (7.20)
	2	2	103	99.4	101	105
Refined oil	0 (Control) [a]	4	<0.001	<0.001	<0.003	<0.002
	0.01	3	88.9 (4.31)	95.1 (1.03)	80.4 (9.65)	63.6 (5.17)
	0.02	3	87.6 (18.3)	98.2 (5.38)	86.7 (7.48)	73.2 (7.96)
	0.05	2	91.1	96.2	100	79.3
	0.2	1	105	99.7	90.0	94.5
	0.4	3	104 (8.32)	103 (4.94)	102 (8.76)	99.4 (5.29)
	2	2	102	107	100	103
Soymilk	0 (Control) [a]	4	ND [b]	ND	<0.001	<0.001
	0.01	3	95.1 (6.26)	101 (5.10)	83.5 (1.52)	72.6 (4.58)
	0.02	3	96.2 (11.5)	103 (8.58)	88.9 (7.30)	83.3 (9.56)
	0.05	2	83.9	104	94.5	86.5
	0.2	1	64.0	105	87.5	107
	0.4	3	90.7 (11.2)	94.1 (5.58)	92.9 (2.38)	87.9 (7.92)
	2	2	77.7	97.8	92.4	95.2
Tofu	0 (Control)[a]	2	ND [b]	ND	<0.001	<0.001
	0.01	2	77.7	109	92.7	77.8
	0.02	1	80.5	101	96.4	78.2
	0.05	1	80.9	103	100	92.6
	0.2	1	97.6	91.6	94.7	85.6
	0.4	3	81.2 (16.3)	82.5 (13.4)	84.5 (11.7)	85.7 (11.8)
	1.5	1	91.1	80.5	83.3	93.3

[a] Values reported for unspiked control samples are mg/kg

[b] ND = not detected in any sample

[c] Mean residue in extracts stored 72 hrs was 0.0055 mg/kg (RSD = 0.1%)

Maize

The analytical method for residues of dicamba in maize matrices was validated in grain, forage, and stover (Table 8).

Table 8 Summary of recoveries of dicamba and metabolites from fortified maize matrices

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Grain	0.01	6	106 (8.8)	106 (3.3)	99 (15)	91 (12)

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
	0.1	6	97 (5.9)	100 (6)	101 (9.5)	95 (3.8)
	0.5	5	101 (2.8)	105 (2.6)	102 (9)	110 (5)
Forage	0.01	6	98 (12)	106 (4.1)	107 (10)	95 (11)
	0.1	6	98 (5.7)	93 (8.3)	91 (10)	78 (7)
	0.5	6	98 (2.8	95 (9.9)	95 (12)	92 (4.8)
Stover	0.01	6	83 (16)	108 (4.2)	117 (1.6)	95 (14) [97 (17)] [a]
	0.1	6	99 (3.6)	98 (4.2)	97 (7.3)	77 (4.2) [101 (4.7)] [a]
	0.5	6	98 (4.7)	96 (5.7)	101 (6.3)	97 (7.3) [106 (4.3)] [a]

[a] Values in square brackets are from samples prepared by an alternate preparation which includes clean-up through a 96-well filter plate

Cotton

The analytical method for residues of dicamba in cotton matrices was validated in undelinted seed, gin trash, hulls, meal, and refined oil (Table 9). For Method AG-ME-1381-01, recoveries and relative standard deviations were acceptable at fortifications of 0.02 mg/kg and greater for all commodities and analytes. Recoveries and relative standard deviations were also acceptable at 0.005 mg/kg in undelinted seed for DCSA, 5-OH dicamba, and DCGA. For MethodME-1713, recoveries and standard deviations were acceptable at 0.01 mg/kg in undelinted seed for all analytes.

Table 9 Summary of recoveries of dicamba and metabolites from fortified cotton matrices

Matrix	Fortification Level (mg/kg)	n	Mean recovery (%) (% RSD)			
			Dicamba	DCSA	5-OH Dicamba	DCGA
Method AG-ME-1381-01						
Undelinted seed	0 (Control) [a]	2	ND [b]	<0.001	<0.001	<0.002
	0.005	5	128 (38.4)	101 (18.5)	82.8 (25.5)	114 (18.9)
	0.01	5	115 (25.1)	89.8 (14.7)	94.2 (16.2)	95.7 (12.0)
	0.02	5	113 (10.5)	93.9 (7.38)	108 (9.26)	87.1 (9.75)
	0.2	5	96.1 (2.25)	98.9 (4.08)	102 (2.90)	88.9 (4.81)
	10	5	89.6 (14.1)	91.6 (6.94)	87.1 (15.3)	88.6 (10.1)
Gin trash	0 (Control) [a]	2	ND [b]	ND [b]	0.010	0.012
	0.04	5	104 (14.6)	79.5 (6.14)	106 (10.1)	80.2 (14.0)
	0.4	5	99.5 (2.73)	89.0 (4.15)	111 (3.28)	97.0 (2.10)
	10	5	86.7 (3.55)	102 (7.40)	99.0 (10.1)	101 (4.30)
Hulls	0 (Control) [a]	2	<0.003	ND [b]	ND [b]	<0.003
	0.02	5	100 (3.38)	100 (6.17)	91.0 (11.5)	116 (2.94)
	0.2	5	100 (2.11)	97.4 (4.05)	108 (6.80)	85.2 (2.83)
Meal	0 (Control) [a]	2	<0.003	<0.003	ND [b]	ND [b]
	0.02	5	104(5.11)	96.5(4.52)	88.1(16.1)	102(3.21)
	0.2	5	96.9 (2.46)	86.2 (4.35)	93.4 (5.28)	86.0 (1.74)
Refined oil	0 (Control) [a]	2	<0.004	ND [b]	ND [b]	ND [b]
	0.02	5	97.6(1.22)	95.2(5.33)	90.8(8.82)	103(5.51)
	0.2	5	99.7 (2.85)	103 (2.01)	102 (8.78)	91.8 (3.07)
Method ME-1713						
Undelinted seed	0.01	6	102 (5.7)	97 (4)	98 (12)	94 (3.9)
	0.1	6	102 (3.3)	97 (1.2)	98 (5.8)	76 (8)
	0.5	6	103 (5.6)	100 (4.8)	101 (6.4)	83 (5.1)

[a] Values reported for unspiked control samples are mg/kg

[b] ND = not detected in any sample

In summary, the submitted analytical methods are suitable for the analysis of dicamba, 5-OH dicamba, and free and conjugated forms of both DCSA and DCGA in the commodities tested.

Stability of pesticide residues in stored analytical samples

The Meeting received data reporting on the stability of dicamba, 5-OH dicamba, DCSA, and DCGA in stored samples of soya bean (M. Mueth and J. Foster, 2012, Report MSL0027420) and cotton (D. Maher and J. Foster, 2012, Report MSL0023058). For both crops, stability was evaluated by using samples from field trials bearing incurred residues of dicamba and metabolites. A single sample for each matrix was kept frozen under conditions mimicking the storage conditions for residue samples and analysing the residues from those samples over time (0–24 months for soya bean seed and 0-9 months for cotton undelinted seed). Samples were analysed in duplicate and the analytical methods included a hydrolysis step; therefore, residues reported as DCSA and DCGA include both free and conjugated forms.

Soya bean

Residues in soya bean matrices were analysed using Method AG-ME-1321-01. Procedural recoveries for soya bean matrices were between 70 and 120% for all analytes-matrices except dicamba in forage (140% at 0.4 mg/kg, Day 0 sample set and 146% at 0.5 mg/kg, Day 61 sample set), dicamba in hay (127% at 0.4 mg/kg, Day 0 sample set), and DCGA (66% at 4 mg/kg, Days 54–61 sample set).

Residues of 5-OH dicamba were < 0.02 mg/kg in all samples, including Day 0; therefore, storage stability for that compound could not be evaluated. For dicamba, DCSA (free and conjugated) and DCGA (free and conjugated), residues from the storage stability samples are summarized in Table 10.

Table 10 Storage stability of dicamba, DCSA, and DCGA in soya bean matrices

Storage Period, months	Dicamba		DCSA		DCGA	
	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0
Forage						
0	0.650, 0.675 [0.6625]	100	33.1, 33.1 [33.1]	100	5.10, 4.82 [4.96]	100
2	1.17, 1.15 [1.16]	175	35.2, 36.3 [35.8]	108	5.12, 4.34 [4.73]	95
3	0.674, 0.614 [0.644]	97	38.8, 37.7 [38.2]	116	5.56, 5.58 [5.57]	112
6	0.676, 0.696 [0.686]	104	36.4, 35.4 [35.9]	108	2.96, 3.11 [3.04]	61
10	0.689, 0.739 [0.714]	108	36.2, 38.5 [37.4]	113	3.24, 3.13 [3.18]	64
12	0.683, 0.709 [0.696]	105	38.1, 35.7 [36.9]	111	3.23, 3.09 [3.16]	64
18	0.660, 0.711 [0.686]	103	40.2, 38.0 [39.1]	118	2.66, 2.28 [2.47]	50
24	0.760, 0.727 [0.744]	112	39.7, 33.1 [36.4]	110	1.80, 1.85 [1.82]	37
Hay						
0	0.129, 0.127 [0.128]	100	109, 117 [113]	100	7.92, 8.47 [8.20]	100
2	0.147, 0.140 [0.144]	112	92.3, 94.5 [93.4]	83	8.12, 8.24 [8.18]	100
3	0.126, 0.131 [0.128]	100	107, 108 [107.5]	95	9.59, 9.54 [9.56]	117
6	0.133, 0.122 [0.128]	100	112, 106 [109]	96	7.12, 7.32 [7.22]	88
10	0.121, 0.098 [0.110]	86	80.7, 92.1 [86.4]	76	6.40, 9.23 [7.82]	95
12	0.171, 0.193 [0.182]	142	88.8, 96.4 [92.6]	82	7.83, 8.76 [8.30]	101
18	0.145, 0.154 [0.150]	117	116, 123 [119.5]	106	7.95, 7.96 [7.96]	97
24	0.200, 0.227 [0.214]	167	109, 114 [111.5]	99	8.47, 8.56 [8.52]	104
Seed						
0	<0.02, <0.02 [<0.02]	--	0.512, 0.566 [0.539]	100	0.244, 0.261 [0.252]	100
2	<0.02, <0.02 [<0.02]	--	0.553, 0.564 [0.558]	103	0.289, 0.315 [0.302]	120
3	<0.02, <0.02 [<0.02]	--	0.590, 0.564 [0.577]	107	0.309, 0.303 [0.306]	121
6	<0.02, <0.02 [<0.02]	--	0.549, 0.540 [0.544]	101	0.243, 0.234 [0.238]	94
10	<0.02, <0.02 [<0.02]	--	0.552, 0.479 [0.516]	96	0.337, 0.339 [0.338]	134
12	<0.02, <0.02 [<0.02]	--	0.560, 0.568 [0.564]	105	0.375, 0.376 [0.376]	149
18	<0.02, <0.02 [<0.02]	--	0.604, 0.580 [0.592]	110	0.288, 0.303 [0.296]	117
24	<0.02, <0.02 [<0.02]	--	0.518, 0.536 [0.527]	98	0.306, 0.313 [0.310]	123

Cotton

Residues in undelinted cotton seed were analysed using Method AG-ME-1381-01, and procedural recoveries ranged from 74 to 133% for all analytes-matrices. Quantifiable residues were observed in 2 of 6 dicamba procedural recovery control samples, 6 of 6 DCSA samples, and 5 of 6 DCGA samples.

The values in the table do not include corrections for residues in the control samples. If corrections are made, the recoveries ranged from 71 to 122%.

Residues of 5-OH dicamba were <0.02 mg/kg in all samples, including Day 0; therefore, storage stability for that compound could not be evaluated. For dicamba, DCSA (free and conjugated) and DCGA (free and conjugated), residues from the storage stability samples are summarized in Table 11.

Table 11 Storage stability of dicamba, DCSA, and DCGA in cotton undelinted seeds

Storage Period, days	Dicamba		DCSA		DCGA	
	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0	mg/kg [mean]	% of Day 0
0	1.01, 0.91, 0.78, 0.83, 0.78, 0.82 [0.85]	100	0.24, 0.24, 0.24, 0.22, 0.24, 0.23 [0.23]	100	0.13, 0.12, 0.12, 0.13, 0.11, 0.12 [0.12]	100
1	0.82, 0.76, 0.79, 0.75 [0.78]	92	0.22, 0.21, 0.23, 0.24 [0.22]	96	0.14, 0.14, 0.16, 0.15 [0.14]	117
2	0.7, 0.89, 0.9, 0.88 [0.84]	99	0.2, 0.24, 0.22, 0.25 [0.23]	100	0.12, 0.13, 0.13, 0.14 [0.13]	108
4	0.94, 0.82, 0.88, 1 [0.91]	107	0.21, 0.2, 0.19, 0.21 [0.2]	87	0.14, 0.13, 0.12, 0.12 [0.13]	108
6	0.54, 0.62, 0.7, 0.72, 0.64, 0.72 [0.66]	78	0.12, 0.18, 0.18, 0.17, 0.18, 0.18 [0.17]	74	0.13, 0.14, 0.15, 0.13, 0.12, 0.14 [0.13]	108
9	0.72, 0.7, 0.8, 0.75, 0.72, 0.71 [0.73]	86	0.2, 0.17, 0.18, 0.21, 0.18, 0.18 [0.19]	83	0.14, 0.13, 0.14, 0.15, 0.14, 0.13 [0.14]	117

In soya beans residues of dicamba and DCSA (incl. conjugates) are stable for at least 2 years in forage, hay, and seed. Residues of DCGA (incl. conjugates) showed a decline in forage during frozen storage, with stability demonstrated for only up to approximately 3 months; DCGA residues were stable in hay and seed for at least 2 years.

Residues of dicamba, DCSA, and DCGA (incl. conjugates) were stable for at least 9 months in undelinted cotton seed.

USE PATTERN

Registered labels describing the uses of dicamba on soya bean, maize, and cotton were provided to the Meeting (Table 12).

Table 12 Registered Uses of Dicamba on Dicamba-Tolerant Crops Submitted to the 2019 Extra JMPR

Crop	Country	Formulation		Application					PHI (days)
		g ai/L	Type	Timing	Method	kg ai/ha	Water L/ha	Number	
Pulses									
Soya bean	Canada	480 (formulated as diglycol-amine salt)	SL	Pre-plant or pre crop emergence	Broadcast	0.28-0.6	100-220	1 or more 0.6 kg ai/ha rate is to be used only once in a season and should be used replant, pre-emergence or in-crop early post-emergence. 7-day retreatment interval 1.18 kg ai/ha annual maximum	7 (forage) 13 (hay)
				Post crop emergence up to 8-leaf stage or 76 cm in height	Broadcast	0.28-0.6			

Crop	Country	Formulation		Application					PHI (days)
		g ai/L	Type	Timing	Method	kg ai/ha	Water L/ha	Number	
	USA	350 (formulated as diglycol-amine salt)	SL	Pre-plant, at-plant, or pre crop emergence	Broadcast	0.56 – 1.12	140	1 or more, not more than 1.12 kg ai/ha pre-emergence 7-day retreatment interval	None
				Post crop emergence up to and incl. R1 growth stage [BBCH 60]	Broadcast	0.56		Up to 2, not more than 1.12 kg ai/ha total post-emergence 7-day retreatment interval 2.24 kg ai/ha annual maximum	
Cereals									
Maize	Canada	480 (formulated as diglycol-amine salt)	SL	Pre-plant or pre crop emergence	Broadcast	0.28-0.6	100-220	1 or more The 0.6 kg ai/ha rate is to be used only once in a season and should be used pre-plant, pre-emergence or in-crop early post-emergence. 7-day retreatment interval	None
				Post crop emergence up to 8-leaf stage or 76 cm in height	Broadcast	0.28-0.6		1.18 kg ai/ha annual maximum	
Oilseeds									
Cotton	USA	350 (formulated as diglycol-amine salt)	SL	Pre-plant, at-plant, or pre crop emergence	Broadcast	0.56 – 1.12	140	1 or more, not more than 1.12 kg ai/ha pre-emergence 7-day retreatment interval	7
				Post crop emergence	Broadcast	0.56		1 or more, not more than 1.12 kg ai/ha total post-emergence 7-day retreatment interval 2.24 kg ai/ha annual maximum	

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received data from supervised residue trials conducted on dicamba-tolerant soya bean, maize, and cotton.

The field trial reports included method validation data, as recoveries from spiked samples at levels reflecting those observed in the field trial samples; dates from critical events during the study, including application, harvest, storage, and analysis; as well as detailed information on the field site and

treatment parameters. Analytical reports were sufficiently detailed and included example chromatograms and example calculations. Samples were analysed by the method described above for plant commodities.

The field trial study designs included control plots. Measured residues from control plots were < LOQ) and are not included in the summary tables in this evaluation. All values in the summary tables are reported in terms of dicamba equivalents. Factors for converting to dicamba equivalents are based on the molecular weights of dicamba (221.04 g/mol), 5-OH dicamba (237.04 g/mol), DCSA (207.01 g/mol), and DCGA (223.01 g/mol). The factors are 0.932 for 5-OH dicamba, 1.068 for DCSA, and 0.991 for DCGA.

When calculating average residues, values below the LOQ were assumed to be at the LOQ, and residues are denoted as being <LOQ only when all samples from a plot were <LOQ. In the summary tables, residue values leading to maximum residue estimations are double underlined, residues used for dietary risk estimation are underlined, and the highest individual values selected for estimating dietary intake are bolded.

When combining residues for estimation of maximum residue levels, values listed as <LOQ are assumed to be LOQ, and the combined residue is listed as '<' only when both residues were below their respective LOQs. When combining residues for risk assessment, residues of dicamba in soya bean seed and maize grain that were reported as <LOQ were assumed to be zero based on the results of metabolism and field trial studies. Similarly, residues of 5-OH dicamba reported as <LOQ were assumed to be 0 in all soya bean and cotton commodities.

Supervised trials for dicamba:

Category	Crop	Commodity	Table
Pulses	Soya bean	Seed (VD 0541)	13
Cereal	Maize	Grain (GC 0645)	14
Oilseeds	Cotton	Seed (SO 0691)	15
Feeds	Soya bean	Forage (AL 1265) and hay	16
	Maize	Forage (AF 0645) and stover	17
	Cotton	Gin trash	18

Pulses

Soya bean

Twenty-two residue trials were conducted on dicamba-tolerant soya beans (MON 87708 variety) in major soya bean-growing areas of the USA during the 2008 growing season (S. Moran and J. Foster, 2010, Report MSL0023061). Trials consisted of one control plot and one or more of the following treatment regimens:

Pre-emergence at 1.12 kg/ha + BBCH 14 at 0.56 kg/ha + BBCH 60 at 0.56 kg/ha,

BBCH 14 at 1.12 kg/ha + BBCH 60 at 1.12 kg/ha, or

Pre-emergence at 1.12 kg/ha + BBCH 60 at 2.24 kg/ha.

Treatments were made with dicamba formulated as either the diglycolamine salt or the monoethanolamine salt. Soya bean seed (without the pod) samples were harvested at maturity (73–98 days after the last application). Samples consisting of 1 kg of seed harvested from at least 12 separate areas of the plot were placed into frozen storage within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis.

Dicamba, 5-hydroxydicamba, DCSA, and DCGA were extracted and analysed using the method described above. Samples were stored for a maximum of 158 days prior to extraction and no more than three days passed between extraction and analysis for residues; residues were shown to be

stable for at least 72 hours. Concurrent recoveries ranged from 74 to 110% across all analytes and fortification levels (0.01 mg/kg to 5 mg/kg except DCGA at 2 mg/kg (121%) and 5 mg/kg (124%). Relative standard deviations across all fortification levels were 5 to 23% for dicamba, 4.7 to 39% for 5-hydroxydicamba, 1.8 to 13% for DCSA, and 6 to 19% for DCGA.

Table 13 Results of dicamba residue trials in dicamba-tolerant soya bean seed (variety MON 87708) in the USA (2008 growing season; Report MSL0023061)

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + up to 2 post, 7-day retreatment interval	1.12 + 0.56 + 0.56	140	Last application BBCH 60	--	--	--	--	--	--
AR Proctor, Arkansas (MEA)	Pre (--) BBCH 14 (34) BBCH 60 (8)	1.12 0.56 0.56	189 190 190	89	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.034, 0.047 [0.040]	0.011, 0.011 [0.011]	0.039, 0.052 [0.045]	0.044, 0.057 [0.051]
	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	89	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.076, 0.062 [0.069]	0.015, 0.018 [0.016]	0.081, 0.067 [0.074]	0.091, 0.080 [0.086]
(DGA)	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	89	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.076, 0.073 [0.075]	0.023, 0.021 [0.022]	0.081, 0.078 [0.080]	0.099, 0.094 [0.097]
GA Montezuma, Georgia (MEA)	Pre (--) BBCH 14 (33) BBCH 60 (21)	1.11 0.56 0.56	191 187 186	77	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.44, 0.44 [0.44]	0.13, 0.13 [0.13]	0.44, 0.44 [0.44]	0.56, **0.57** [0.56]
IA-1 Richland, Iowa (MEA)	Pre (--) BBCH 14 (23) BBCH 60 (22)	1.13 0.51 0.57	184 189 185	80	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.012, 0.014 [0.013]	0.012, 0.012 [0.012]	0.017, 0.019 [0.018]	0.024, 0.026 [0.025]
	Pre (--) BBCH 14 (22)	1.1 1.12	189 188	73	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.020, 0.019 [0.020]	0.024, 0.023 [0.023]	0.025, 0.024 [0.025]	0.044, 0.042 [0.043]
				80	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.019, 0.012 [0.016]	0.016, 0.016 [0.016]	0.024, 0.017 [0.021]	0.036, 0.028 [0.032]
				87	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.019, 0.020 [0.020]	0.021, 0.023 [0.022]	0.024, 0.025 [0.025]	0.041, 0.043 [0.042]
				94	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.017, 0.021 [0.019]	0.020, 0.026 [0.023]	0.022, 0.026 [0.024]	0.037, 0.046 [0.041]
IA-2 Hedrick, Iowa (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (19)	1.12 0.58 0.56	187 189 183	95	<0.005, <0.005 [<0.005]	0.019, 0.019 [0.019]	0.011, 0.011 [0.011]	0.018, 0.014 [0.016]	0.016, 0.016 [0.016]	0.048, 0.044 [0.046]
IL-1 Wyoming, Illinois (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (21)	1.13 0.56 0.56	190 188 184	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.017, 0.010 [0.013]	0.018, 0.011 [0.014]	0.022, 0.015 [0.018]	0.035, 0.021 [0.028]
IL-2 Carlyle, Illinois (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (14)	1.15 0.57 0.56	190 195 185	74	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.048, 0.050 [0.049]	0.026, 0.023 [0.025]	0.053, 0.055 [0.054]	0.074, 0.073 [0.074]
	Pre (--) BBCH 14 (14)	1.11 1.11	191 182	74	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.11, 0.12 [0.11]	0.063, 0.066 [0.064]	0.12, 0.12 [0.12]	0.18, 0.18 [0.18]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(DGA)	Pre (--) BBCH 14 (14)	1.13 1.14	193 188	74	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.099, 0.11 [0.10]	0.058, 0.063 [0.060]	0.10, 0.11 [0.11]	0.16, 0.17 [0.16]
IN Rockville, Indiana (MEA)	Pre (--) BBCH 14 (21) BBCH 60 (21)	1.12 0.56 0.55	189 186 176	73	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.045, 0.041 [0.043]	0.038, 0.050 [0.044]	0.050, 0.046 [0.048]	0.082, 0.091 [0.087]
KS-1 Cunningham, Kansas (MEA)	Pre (--) BBCH 14 (28) BBCH 60 (8)	1.11 0.55 0.57	187 191 198	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.024, 0.020 [0.022]	0.011, 0.011 [0.011]	0.029, 0.025 [0.027]	0.034, 0.031 [0.032]
	Pre (--) BBCH 14 (8)	1.1 1.15	193 200	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.023, 0.045 [0.034]	0.017, 0.019 [0.018]	0.028, 0.050 [0.039]	0.040, 0.064 [0.052]
(DGA)	Pre (--) BBCH 14 (8)	1.11 1.18	193 205	95	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.049, 0.053 [0.051]	0.021, 0.020 [0.021]	0.054, 0.058 [0.056]	0.070, 0.073 [0.072]
KS-2 Hudson, Kansas (MEA)	Pre (--) BBCH 14 (32) BBCH 60 (13)	1.14 0.55 0.58	191 184 193	77	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.013, 0.016 [0.014]	0.016, 0.015 [0.016]	0.018, 0.021 [0.019]	0.028, 0.031 [0.030]
LA Washington, Louisiana (MEA)	Pre (--) BBCH 14 (15) BBCH 60 (15)	1.11 0.55 0.57	188 199 187	85	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.021, 0.017 [0.019]	0.011, 0.011 [0.011]	0.026, 0.022 [0.024]	0.031, 0.028 [0.029]
MI Conklin, Michigan (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (26)	1.12 0.56 0.56	184 186 186	88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.095, 0.090 [0.092]	0.061, 0.055 [0.058]	0.100, 0.095 [0.097]	0.16, 0.15 [0.15]
MN-1 Campbell, Minnesota (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (17)	1.12 0.56 0.56	187 187 187	78	<0.005, 0.012 [0.0088]	<0.019, 0.019 [0.019]	0.054, 0.056 [0.055]	0.043, 0.045 [0.044]	0.059, 0.068 [0.063]	0.096, 0.13 [0.11]
	Pre (--) BBCH 14 (17)	1.12 1.13	188 188	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.082, 0.079 [0.081]	0.081, 0.079 [0.080]	0.087, 0.084 [0.086]	0.16, 0.16 [0.16]
				88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.076, 0.074 [0.075]	0.061, 0.055 [0.058]	0.081, 0.079 [0.080]	0.14, 0.13 [0.13]
				92	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.082, 0.075 [0.079]	0.081, 0.080 [0.081]	0.087, 0.080 [0.084]	0.16, 0.16 [0.16]
				100	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.073, 0.071 [0.072]	0.072, 0.071 [0.072]	0.078, 0.076 [0.077]	0.14, 0.14 [0.14]
MN-2 Fergus Falls, Minnesota (MEA)	Pre (--) BBCH 14 (27) BBCH 60 (18)	1.12 0.56 0.56	188 188 187	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.071, 0.073 [0.072]	0.048, 0.053 [0.050]	0.076, 0.078 [0.077]	0.12, 0.13 [0.12]
	Pre (--) BBCH 14 (18)	1.12 1.13	187 188	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.16, 0.16 [0.16]	0.12, 0.12 [0.12]	0.17, 0.17 [0.17]	0.28, 0.29 [0.28]
(DGA)	Pre (--) BBCH 14 (18)	1.12 1.12	188 187	78	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.20, 0.18 [0.19]	0.14, 0.12 [0.13]	0.21, 0.18 [0.20]	0.34, 0.30 [0.32]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
MO Fisk, Missouri (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (17)	1.12 0.56 0.57	186 189 189	81	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.020, 0.025 [0.023]	0.014, 0.016 [0.015]	0.025, 0.030 [0.028]	0.033, 0.041 [0.037]
ND-1 Carrington, North Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (29)	1.12 0.56 0.57	188 187 188	87	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.059, 0.051 [0.055]	0.048, 0.041 [0.044]	0.064, 0.056 [0.060]	0.11, 0.092 [0.099]
NE-1 York, Nebraska (MEA)	Pre (--) BBCH 14 (26) BBCH 60 (21)	1.13 0.56 0.56	188 187 187	87	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.028, 0.025 [0.027]	0.030, 0.029 [0.029]	0.033, 0.030 [0.032]	0.058, 0.054 [0.056]
NE-2 Osceola, Nebraska (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (18)	1.1 0.56 0.57	182 187 188	86	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.029, 0.032 [0.031]	0.015, 0.016 [0.016]	0.034, 0.037 [0.036]	0.044, 0.049 [0.046]
SC Elko, South Carolina (MEA)	Pre (--) BBCH 14 (30) BBCH 60 (9)	1.12 0.56 0.56	189 193 186	88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.020, 0.022 [0.021]	0.011, 0.011 [0.011]	0.025, 0.027 [0.026]	0.030, 0.032 [0.031]
SD-1 Centerville, South Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (17)	1.09 0.55 0.56	180 184 185	76	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.12, 0.12 [0.12]	0.056, 0.051 [0.053]	0.13, 0.12 [0.12]	0.18, 0.17 [0.17]
SD-2 Britton, South Dakota (MEA)	Pre (--) BBCH 14 (32) BBCH 60 (10)	1.12 0.56 0.56	186 187 187	88	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.046, 0.050 [0.048]	0.011, 0.011 [0.011]	0.051, 0.055 [0.053]	0.057, 0.060 [0.058]
WI-1 Delavan, Wisconsin (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (15)	1.12 0.52 0.56	183 173 188	85	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.079, 0.075 [0.077]	0.071, 0.064 [0.068]	0.084, 0.080 [0.082]	0.15, 0.14 [0.14]
WI-2 Fitchburg, Wisconsin (MEA)	Pre (--) BBCH 14 (29) BBCH 60 (6)	0.56+0 .57 0.57 0.56	195, 186 188 190	98	<0.005, <0.005 [<0.005]	<0.019, <0.019 [<0.019]	0.012, 0.0093 [0.011]	0.011, 0.011 [0.011]	0.017, 0.014 [0.016]	0.023, 0.020 [0.021]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Cereal grains

Maize

Twenty-two residue trials were conducted on dicamba-tolerant maize (MON 87419 variety) in major maize-growing areas of the USA during the 2013 growing season (E. Urbanczyk-Wochniak, 2015, Report MSL0026526). Trials consisted of one control plot and one or more of the following treatment regimens:

Pre-emergence at 1.12 kg ai/ha + BBCH 14-16 at 0.56 kg ai/ha + BBCH 18 at 0.56 kg ai/ha,

BBCH 12-14 at 0.56 kg ai/ha + BBCH 14-16 at 0.56 kg ai/ha + BBCH 18 at 0.56 kg ai/ha + 122 cm [BBCH 19] at 0.56 kg ai/ha, or

Pre-emergence at 6 kg ai/ha + BBCH 14-16 at 3 kg ai/ha + BBCH 18 at 3 kg ai/ha.

For all regimens, application retreatment intervals were at a minimum of seven days. Treatments were made with dicamba formulated as the diglycolamine salt. Maize grain samples were harvested at maturity (64-132 days after the last application). Samples consisting of 1 kg of seed harvested from at least 12 separate areas of the plot were placed into frozen storage within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis.

Samples were stored for a maximum of 147 days prior to extraction and no more than two days passed between extraction and analysis for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above. Concurrent recoveries ranged from 89 to 107% across all analytes and fortification levels (0.01 mg/kg to ca. 0.5 mg/kg). Relative standard deviations across all analytes and fortification levels were 5.1 to 18%.

Table 14 Results of dicamba residue trials in dicamba-tolerant maize grain (variety MON 87419) in the USA (2013 growing season; Report MSL0026526)

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP Canada	1 pre + 1 post up to BBCH 18	0.6+ 0.6	100	30	--	--	--	--	--	--
01PA Germansville, Pennsylvania	Pre (--) BBCH 14-15 (23) BBCH 17-18 (14)	1.14 0.57 0.57	192 190 191	113	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 14-15 (7) BBCH 17-18 (14) BBCH 19 (8)	0.58 0.58 0.58 0.56	194 193 195 189	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
02GA Chula, Georgia	Pre (--) BBCH 15 (27) BBCH 18 (12)	1.11 0.54 0.57	182 185 183	86	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (14) BBCH 18 (12) BBCH 19 (10)	0.56 0.55 0.55 0.57	183 188 178 195	76	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
03NC Belvidere, North Carolina	Pre (--) BBCH 15-16 (28) BBCH 18 (14)	1.16 0.58 0.56	193 186 188	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15-16 (7) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	183 181 187 188	82	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
04TX Uvalde, Texas	BBCH 0 BBCH 15-16 (47) BBCH 18 (7)	1.11 0.56 0.56	187 185 187	71	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.039, 0.031 [0.035]	0.021, 0.020 [0.020]	0.049, 0.041 [0.045]	0.070, 0.061 [0.066]
	BBCH 12 BBCH 15-16 (23) BBCH 18 (7) BBCH 51 (7)	0.57 0.56 0.56 0.57	188 188 187 189	64	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.033, 0.027 [0.030]	0.023, 0.017 [0.020]	0.043, 0.037 [0.040]	0.065, 0.054 [0.060]
05MI Wright, Michigan	Pre (--) BBCH 14 (34) BBCH 18 (13)	1.14 0.56 0.56	187 187 189	121	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 14 (9) BBCH 18 (13) BBCH 19 (11)	0.56 0.56 0.56 0.56	186 186 190 189	110	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
06KS Troy, Kansas	BBCH 5 BBCH 13 (22) BBCH 18 (8)	1.13 0.57 0.65	190 195 221	132	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 13 (9) BBCH 18 (8)	0.58 0.57 0.57	195 194 195	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 39 (13)	0.57	196							
07KS St. John, Kansas	BBCH 1 BBCH 16 (32) BBCH 18 (7)	1.12 0.57 0.55	185 188 187	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 16 (18) BBCH 18 (7) BBCH 19 (21)	0.57 0.56 0.55 0.58	188 185 185 191	70	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
08IA Black Hawk, Iowa	Pre (--) BBCH 15 (35) BBCH 18 (13)	1.11 0.55 0.56	186 183 186	106	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (12) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.55 0.56	191 186 183 189	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
09IL Carlyle, Illinois	Pre (--) BBCH 15-16 (32) BBCH 18 (13)	1.13 0.57 0.56	196 194 193	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 14 BBCH 15-16 (9) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.56 0.56	179 190 193 191	84	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
10IL Highland, Illinois	Pre (--) BBCH 15 (25) BBCH 18 (16)	1.16 0.57 0.56	196 182 194	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (7) BBCH 18 (16) BBCH 18 (9)	0.58 0.57 0.56 0.56	194 182 192 193	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
11IL Camp Grove, Illinois	BBCH 0 BBCH 14 (30) BBCH 18 (9)	1.13 0.56 0.56	188 185 183	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 14 (7) BBCH 18 (9) BBCH 19 (15)	0.55 0.56 0.55 0.57	178 182 181 190	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
12IL Stewardson, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (12)	1.12 0.55 0.55	188 180 180	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (11) BBCH 18 (12) BBCH 19 (11)	0.56 0.56 0.56 0.56	192 184 184 187	94	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
13IL Duvall, Illinois	BBCH 3 BBCH 16 (25) BBCH 18 (16)	1.12 0.57 0.56	190 192 192	108	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 16 (7) BBCH 18 (16) BBCH 24 (9)	0.56 0.58 0.57 0.57	190 196 196 189	96	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
14IL Highland, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (14)	1.12 0.57 0.57	195 195 195	97	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (14) BBCH 18 (14) BBCH 39 (9)	0.56 0.57 0.56 0.57	194 194 195 191	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
15IN Pickard, Indiana	BBCH 0 BBCH 15 (29) BBCH 18 (12)	1.16 0.56 0.56	194 188 193	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]

Trial No. Location (Salt) [a]	Application			DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 12 BBCH 15 (15) BBCH 18 (12) BBCH 19 (14)	0.57 0.56 0.57 0.56	193 188 195 187	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
16MO Kirksville, Missouri	Pre (--) BBCH 14 (26) BBCH 18 (14)	1.11 0.56 0.56	186 188 184	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 14 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	189 187 185 192	82	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
17MO Broseley, Missouri	BBCH 0 BBCH 5 (21) BBCH 16 (9)	1.12 0.57 0.56	187 188 187	95	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 14 BBCH 16 (7) BBCH 18 (9) BBCH 19 (7)	0.57 0.56 0.56 0.57	188 187 187 188	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
18NE Tabor, Nebraska	BBCH 0 BBCH 15 (32) BBCH 18 (8)	1.11 0.56 0.57	178 179 184	118	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (18) BBCH 18 (8) BBCH 19 (7)	0.57 0.56 0.57 0.56	184 180 182 188	111	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
19NE Henderson, Nebraska	BBCH 0 BBCH 16 (34) BBCH 19 (7)	1.1 0.55 0.56	191 191 191	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 16 (8) BBCH 19 (7) BBCH 19 (7)	0.55 0.56 0.57 0.56	190 194 191 194	92	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
20NE Brunswick, Nebraska	BBCH 0 BBCH 15 (30) BBCH 18 (7)	1.09 0.55 0.56	188 190 192	101	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 12 BBCH 15 (10) BBCH 18 (7) BBCH 19 (13)	0.56 0.56 0.56 0.56	190 191 191 194	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
21SD Bushnell, S. Dakota	BBCH 0 BBCH 16 (32) BBCH 18 (7)	1.12 0.58 0.56	188 193 182	117	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 16 (12) BBCH 18 (7) BBCH 19 (7)	0.56 0.56 0.57 0.56	196 187 188 183	110	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
22WI Richmond, Wisconsin	Pre (--) BBCH 15 (33) BBCH 18 (14)	1.11 0.56 0.56	187 184 181	120	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]
	BBCH 13 BBCH 15 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.56	189 185 181 187	113	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	<0.03, <0.03 [<0.03]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Oilseeds

Cotton

Thirteen residue trials were conducted in major cotton growing areas of the USA during the 2010 growing season (D. Maher and J. Foster, 2010, Report MSL0024072). Trials consisted of one control plot and one or more of the following treatment regimens:

Pre-emergence at 1.12 kg/ha + BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha,

Pre-emergence at 1.12 kg/ha + BBCH 80 at 0.56 kg/ha + BBCH 99 at 0.56 kg/ha (7 days preharvest), or

BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha + BBCH 80 at 0.56 kg/ha + BBCH 99 at 0.56 kg/ha (7 days preharvest).

Treatments were made with dicamba formulated as either the diglycolamine salt (Treatments 1-3 above) or the monoethanolamine salt (Treatment 3 only). Cotton seed (undelinted) samples were harvested at maturity. Samples consisting of 1 kg of seed were placed into frozen storage within four hours of collection and remained frozen during transportation to the analytical facility and prior to analysis.

Samples were stored for a maximum of 169 days prior to extraction and no more than two days passed between extraction and analysis for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above. Concurrent recoveries ranged from 85 to 108% across all analytes and fortification levels (0.02 mg/kg to 5 mg/kg (0.2 mg/kg 5-hydroxydicamba)). Relative standard deviations across all analytes and fortification levels were 4.3 to 17%.

Table 15 Results of dicamba residue trials in dicamba-tolerant cotton seed (variety MON 88701) in the USA (2010 growing season; Report MSL0024072)

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post with a 7-day retreatment interval	1.12 + 0.56 + 0.56	140	7	--	--	--	--	--	--
AR1 Proctor, Arkansas (DGA)	ns (--) ns (29) Mid-bloom (38)	1.1 0.56 0.56	188 187 188	70	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.053, 0.043 [0.048]	0.030, 0.030 [0.030]	0.073, 0.063 [0.068]	0.10, 0.092 [0.098]
	ns (--) ns (99) 95% Open bolls (31)	1.1 0.56 0.56	188 188 188	7	1.2, 0.59 [0.90]	<0.0093, <0.0093 [<0.0093]	0.11, 0.053 [0.080]	0.040, 0.030 [0.035]	1.3, 0.64 [0.98]	1.3, 0.67 [1.0]
	ns (--) ns (38) ns (32) 95% Open bolls (31)	0.56 0.56 0.56 0.56	188 189 188 188	7	0.33, 0.58 [0.45]	<0.0093, <0.0093 [<0.0093]	0.053, 0.075 [0.064]	0.040, 0.050 [0.045]	0.38, 0.65 [0.52]	0.42, 0.70 [0.56]
(MEA)	ns (--) ns (38) ns (32) 95% Open bolls (31)	0.56 0.56 0.56 0.56	187 189 189 188	7	0.72, 0.51 [0.62]	<0.0093, <0.0093 [<0.0093]	0.18, 0.064 [0.12]	0.079, 0.040 [0.059]	0.90, 0.57 [0.74]	0.98, 0.61 [0.80]
CA1 Porterville, California (DGA)	ns (--) ns (48) BBCH 65 (43)	1.1 0.56 0.57	192 190 188	81	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (136) BBCH 97 (28)	1.1 0.57 0.57	190 189 191	8	0.18, 0.19 [0.18]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.021 [0.013]	<0.0053, <0.0053 [<0.0053]	0.19, 0.21 [0.20]	0.19, 0.22 [0.20]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	ns (--) ns (43) ns (45) BBCH 97 (28)	0.56 0.58 0.57 0.57	189 190 189 189	8	0.47, 0.39 [0.43]	<0.0093, <0.0093 [<0.0093]	0.096, 0.043 [0.069]	0.040, 0.020 [0.030]	0.57, 0.43 [0.50]	0.61, 0.45 [0.53]
(MEA)	ns (--) ns (43) ns (45) BBCH 97 (28)	0.56 0.58 0.56 0.56	189 189 188 187	8	0.23, 0.17 [0.20]	<0.0093, <0.0093 [<0.0093]	0.064, 0.021 [0.043]	0.020, 0.020 [0.020]	0.29, 0.19 [0.24]	0.31, 0.21 [0.26]
CA2 Visalia, California (DGA)	ns (--) ns (41) BBCH 65 (49)	1.1 0.56 0.57	192 188 194	103	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (178) BBCH 85 (7)	1.1 0.57 0.56	191 190 192	8	0.66, 0.64 [0.65]	<0.0093, <0.0093 [<0.0093]	0.021, 0.053 [0.037]	<0.0053, <0.0053 [<0.0053]	0.68, 0.69 [0.69]	0.69, 0.70 [0.69]
	ns (--) ns (49) ns (88) BBCH 85 (7)	0.57 0.56 0.57 0.57	193 190 191 195	8	1.0, 0.82 [0.94]	<0.0093, <0.0093 [<0.0093]	0.15, 0.17 [0.16]	0.030, 0.030 [0.030]	1.2, 0.99 [1.1]	1.2, 1.0 [1.1]
GA1 Chula, Georgia (DGA)	ns (--) ns (26) 15 Days after white flower (43)	1.1 0.56 0.57	192 192 189	84	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (104) 90% Open bolls (42)	1.1 0.58 0.56	193 191 191	7	0.76, 1.3 [1.0]	<0.0093, <0.0093 [<0.0093]	0.021, 0.021 [0.021]	<0.0053, <0.0053 [<0.0053]	0.78, 1.3 [1.1]	0.79, 1.3 [1.1]
	ns (--) ns (43) ns (35) 90% Open bolls (42)	0.57 0.56 0.57 0.56	195 187 185 191	7	0.80, 0.71 [0.76]	<0.0093, <0.0093 [<0.0093]	0.032, 0.021 [0.027]	0.030, 0.020 [0.025]	0.83, 0.73 [0.78]	0.86, 0.75 [0.81]
(MEA)	ns (--) ns (43) ns (35) 90% Open bolls (42)	0.57 0.56 0.56 0.56	195 187 182 189	7	0.68, 0.50 [0.59]	<0.0093, <0.0093 [<0.0093]	0.021, 0.021 [0.021]	0.020, 0.020 [0.020]	0.70, 0.52 [0.61]	0.72, 0.54 [0.63]
LA1 Cheneyville, Louisiana (DGA)	ns (--) ns (27) Mid-bloom (36)	1.1 0.57 0.57	192 178 180	73	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (97) BBCH 88 (33)	1.1 0.56 0.58	195 188 202	7	0.33, 0.45 [0.39]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	0.020, 0.020 [0.020]	0.34, 0.46 [0.40]	0.36, 0.48 [0.42]
	ns (--) ns (36) ns (34) BBCH 88 (33)	0.58 0.57 0.57 0.57	180 181 192 198	7	0.82, 0.65 [0.74]	<0.0093, <0.0093 [<0.0093]	0.032, 0.064 [0.048]	0.030, 0.030 [0.030]	0.85, 0.71 [0.78]	0.88, 0.74 [0.81]
MO1 Fisk, Missouri (DGA)	ns (--) ns (29) BBCH 65 (42)	1.1 0.56 0.56	187 187 187	79	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.021, 0.021 [0.021]	0.020, 0.020 [0.020]	0.041, 0.041 [0.041]	0.061, 0.061 [0.061]
	ns (--) ns (113) BBCH 89 (30)	1.1 0.56 0.56	187 187 187	7	0.97, 1.0 [0.98]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.064 [0.035]	0.020, <0.0053 [0.012]	0.98, 1.1 [1.0]	1.0, 1.1 [1.0]
	ns (--) ns (42) ns (42) BBCH 89 (30)	0.57 0.56 0.56 0.56	190 188 187 187	7	0.56, 1.2 [0.86]	<0.0093, <0.0093 [<0.0093]	0.021, 0.032 [0.027]	0.020, 0.020 [0.020]	0.58, 1.2 [0.89]	0.60, 1.2 [0.91]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
OK1 Hinton, Oklahoma (DGA)	ns (--) ns (24) BBCH 65 (49)	1.1 0.57 0.56	176 190 188	68	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.030, 0.030 [0.030]	0.052, 0.052 [0.052]	0.082, 0.082 [0.082]
	ns (--) ns (107) BBCH 89 (27)	1.1 0.56 0.57	178 185 193	8	0.33, 0.23 [0.28]	<0.0093, <0.0093 [<0.0093]	0.085, 0.032 [0.059]	0.050, 0.030 [0.040]	0.42, 0.26 [0.34]	0.46, 0.29 [0.38]
	ns (--) ns (49) ns (34) BBCH 89 (27)	0.57 0.55 0.57 0.57	190 186 189 193	8	0.35, 0.18 [0.26]	<0.0093, <0.0093 [<0.0093]	0.12, 0.075 [0.096]	0.089, 0.050 [0.069]	0.47, 0.25 [0.36]	0.56, 0.30 [0.43]
(MEA)	ns (--) ns (49) ns (34) BBCH 89 (27)	0.56 0.55 0.56 0.56	185 185 184 190	8	0.23, 0.23 [0.23]	<0.0093, <0.0093 [<0.0093]	0.021, 0.075 [0.048]	0.030, 0.040 [0.035]	0.25, 0.30 [0.28]	0.28, 0.34 [0.31]
OK2 Dill City, Oklahoma (DGA)	ns (--) ns (28) BBCH 65 (49)	1.1 0.56 0.55	178 188 184	82	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.032, <0.0053 [0.019]	0.030, 0.020 [0.025]	0.052, 0.025 [0.039]	0.082, 0.045 [0.063]
	ns (--) ns (109) BBCH 97 (43)	1.1 0.56 0.56	180 190 188	7	0.070, 0.060 [0.065]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.075, 0.065 [0.070]	0.080, 0.070 [0.075]
	ns (--) ns (49) BBCH 97 (32) ns (43)	0.57 0.56 0.56 0.55	189 188 190 184	7	0.12, 0.16 [0.14]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.020, 0.030 [0.025]	0.15, 0.19 [0.17]	0.17, 0.22 [0.20]
SC1 Elko, South Carolina (DGA)	ns (--) ns (29) BBCH 65 (38)	1.1 0.56 0.56	190 195 193	99	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
				105	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
				112	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
				119	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (102) BBCH 95 (63)	1.1 0.56 0.56	189 195 187	7	0.18, 0.060 [0.12]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.021 [0.013]	<0.0053, 0.020 [0.012]	0.19, 0.081 [0.13]	0.19, 0.10 [0.15]
	ns (--) ns (38) ns (35) BBCH 95 (63)	0.56 0.56 0.56 0.56	194 193 193 186	1	0.21, 0.31 [0.26]	<0.0093, <0.0093 [<0.0093]	0.021, 0.032 [0.027]	0.020, 0.030 [0.025]	0.23, 0.34 [0.29]	0.25, 0.37 [0.31]
				7	0.14, 0.090 [0.12]	<0.0093, <0.0093 [<0.0093]	0.053, 0.021 [0.037]	0.059, 0.020 [0.040]	0.19, 0.11 [0.15]	0.25, 0.13 [0.19]
				14	0.090, 0.070 [0.080]	<0.0093, <0.0093 [<0.0093]	0.064, 0.032 [0.048]	0.099, 0.050 [0.074]	0.15, 0.10 [0.13]	0.25, 0.15 [0.20]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				21	0.040, 0.080 [0.060]	<0.0093, <0.0093 [<0.0093]	0.096, 0.032 [0.064]	0.11, 0.040 [0.074]	0.14, 0.11 [0.12]	0.25, 0.15 [0.20]
TX1 Raymondville, Texas (DGA)	ns (--) ns (36) BBCH 65-67 (46)	1.1 0.58 0.58	190 194 195	43	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.032, <0.0053 [0.019]	0.020, 0.020 [0.020]	0.052, 0.025 [0.039]	0.072, 0.045 [0.059]
				49	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.021, <0.0053 [0.013]	<0.0053, 0.020 [0.012]	0.041, 0.025 [0.033]	0.046, 0.045 [0.046]
				56	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, 0.032 [0.019]	<0.0053, 0.020 [0.012]	0.025, 0.052 [0.039]	0.030, 0.072 [0.051]
				63	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.021, <0.0053 [0.013]	0.020, <0.0053 [0.012]	0.041, 0.025 [0.033]	0.061, 0.030 [0.046]
	ns (--) ns (119) BBCH 87 (5)	1.1 0.58 0.58	191 194 193	7	0.58, 0.19 [0.38]	<0.0093, <0.0093 [<0.0093]	0.075, <0.0053 [0.040]	<0.0053, <0.0053 [<0.0053]	0.65, 0.20 [0.43]	0.66, 0.20 [0.43]
	ns (--) ns (46) ns (37) BBCH 87 (5)	0.58 0.58 0.58 0.58	194 195 194 195	1	0.28, 0.23 [0.26]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.030, 0.030 [0.030]	0.31, 0.26 [0.29]	0.34, 0.29 [0.32]
				7	0.19, 0.17 [0.18]	<0.0093, <0.0093 [<0.0093]	0.096, 0.043 [0.069]	0.030, 0.020 [0.025]	0.29, 0.21 [0.25]	0.32, 0.23 [0.27]
				14	0.070, 0.040 [0.055]	<0.0093, <0.0093 [<0.0093]	0.043, 0.032 [0.037]	0.030, 0.020 [0.025]	0.11, 0.072 [0.092]	0.14, 0.092 [0.12]
				21	0.030, 0.040 [0.035]	<0.0093, <0.0093 [<0.0093]	0.032, 0.032 [0.032]	0.030, 0.030 [0.030]	0.062, 0.072 [0.067]	0.092, 0.10 [0.097]
TX2 Levelland, Texas (DGA)	ns (--) ns (36) BBCH 65 (41)	1.1 0.57 0.56	191 193 188	84	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.25, 0.25 [0.25]	0.11, 0.11 [0.11]	0.27, 0.27 [0.27]	0.37, 0.37 [0.37]
	ns (--) ns (118) BBCH 89 (37)	1.1 0.55 0.55	190 184 185	6	0.84, 1.3 [1.1]	<0.0093, <0.0093 [<0.0093]	0.12, 0.21 [0.17]	0.069, 0.050 [0.059]	0.96, 1.5 [1.2]	1.0, 1.6 [1.3]
	ns (--) ns (41) ns (41) BBCH 89 (37)	0.55 0.55 0.56 0.55	187 187 191 187	6	1.3, 1.5 [1.4]	<0.0093, <0.0093 [<0.0093]	0.28, 0.29 [0.28]	0.11, 0.17 [0.14]	1.6, 1.8 [1.7]	1.7, 2.0 [1.8]
TX3 Wolfforth, Texas (DGA)	ns (--) ns (41) BBCH 65 (36)	1.1 0.57 0.55	188 194 185	84	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	0.11, 0.043 [0.075]	0.069, 0.040 [0.055]	0.13, 0.063 [0.095]	0.20, 0.10 [0.15]
	ns (--) ns (125) BBCH 89 (30)	1.1 0.55 0.55	189 187 184	6	2.0, 0.80 [1.4]	<0.0093, <0.0093 [<0.0093]	0.17, 0.17 [0.17]	0.069, 0.099 [0.084]	2.1, 0.97 [1.6]	2.2, 1.1 [1.6]
	ns (--) ns (36) ns (48) BBCH 89 (30)	0.56 0.56 0.56 0.56	189 188 189 188	6	1.3, 1.1 [1.2]	<0.0093, <0.0093 [<0.0093]	0.12, 0.17 [0.14]	0.079, 0.12 [0.099]	1.4, 1.3 [1.3]	1.5, 1.4 [1.4]

Trial No. Location (Salt) [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
TX4 Uvalde, Texas (DGA)	ns (--) ns (49) BBCH 65 (47)	1.1 0.56 0.56	189 188 186	71	<0.02, <0.02 [<0.02]	<0.0093, <0.0093 [<0.0093]	<0.0053, <0.0053 [<0.0053]	<0.0053, <0.0053 [<0.0053]	0.025, 0.025 [0.025]	0.030, 0.030 [0.030]
	ns (--) ns (132) BBCH 89 (29)	1.1 0.58 0.55	186 201 184	6	0.86, 1.2 [1.0]	<0.0093, 0.019 [0.014]	0.032, 0.043 [0.037]	0.020, 0.030 [0.025]	0.89, 1.3 [1.1]	0.91, 1.3 [1.1]
	ns (--) ns (47) ns (36) BBCH 89 (29)	0.56 0.55 0.55 0.57	187 184 192 189	6	0.49, 0.44 [0.46]	<0.0093, <0.0093 [<0.0093]	0.032, 0.043 [0.037]	0.030, 0.030 [0.030]	0.52, 0.48 [0.50]	0.55, 0.51 [0.53]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Legume animal feeds

Soya bean

Twenty-two residue trials were conducted on dicamba-tolerant soya beans (MON 87708 variety) as described above (Pulses, soya bean; Report MSL0023061). From those trials, samples of soya bean forage and hay were harvested 7–10 and 14–24 DALA, respectively. Samples of hay were dried in the field to a moisture content of 10–20%.

Soya bean forage samples were stored frozen for 119 to 292 days, and hay samples for up to 283 days. Residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA were analysed using the method described above. Average concurrent recoveries from forage across all four analytes and across fortifications from 0.01 to 150 mg/kg ranged from 84 to 125%. Relative standard deviations ranged from 8.1 to 18%. Storage stability data indicate that residues of DCGA in forage are not stable for the storage period experienced by the samples in the study.

Results for soya bean forage and hay are shown in Table 16.

Table 16 Results of dicamba residue trials in dicamba-tolerant soya bean forage and hay (variety MON 87708) in the USA (2008 growing season; Report MSL0023061)

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post with a 7-day retreatment interval	1.12 + 0.56 + 0.56	140	--	7 forage 14 hay	--	--	--	--	--	--
AR Proctor, Arkansas (MEA)	Pre (--) BBCH 14 (34) BBCH 60 (8)	1.12 0.56 0.56	189 190 190	Forage	7	0.051, 0.058 [0.054]	0.0067, 0.0086 [0.0076]	22, 19 [20]	2.0, 1.8 [1.9]	22, 19 [20]	24, 20 [22]
				Hay	15	0.049, 0.052 [0.050]	0.0093, 0.011 [0.0100]	30, 40 [35]	1.9, 2.5 [2.2]	30, 40 [35]	32, 42 [37]
	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	Forage	7	0.17, 0.12 [0.15]	0.018, 0.016 [0.017]	43, 39 [41]	2.2, 2.9 [2.6]	44, 39 [41]	46, 42 [44]
				Hay	15	0.15, 0.11 [0.13]	0.027, 0.031 [0.029]	90, 76 [83]	5.3, 4.3 [4.8]	90, 76 [83]	95, 81 [88]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(DGA)	Pre (--) BBCH 14 (8)	1.12 1.12	190 190	Forage	7	0.16, 0.13 [0.15]	0.021, 0.017 [0.019]	42, 39 [41]	3.0, 2.8 [2.9]	42, 40 [41]	45, 42 [44]
				Hay	15	0.14, 0.17 [0.15]	0.023, 0.032 [0.028]	76, 99 [88]	4.1, 5.4 [4.7]	76, 99 [88]	80, 100 [93]
GA Montezuma, Georgia (MEA)	Pre (--) BBCH 14 (33) BBCH 60 (21)	1.11 0.56 0.56	191 187 186	Forage	8	0.021, 0.021 [0.021]	0.0081, 0.0056 [0.0068]	51, 49 [50]	4.6, 5.9 [5.3]	51, 49 [50]	56, 55 [56]
				Hay	24	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	12, 13 [13]	0.43, 0.37 [0.40]	12, 13 [13]	13, 14 [13]
IA-1 Richland, Iowa (MEA)	Pre (--) BBCH 14 (23) BBCH 60 (22)	1.13 0.51 0.57	184 189 185	Forage	7	0.021, 0.021 [0.021]	0.0047, 0.0047 [0.0047]	14, 14 [14]	1.5, 1.9 [1.7]	14, 14 [14]	16, 16 [16]
				Hay	17	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	36, 32 [34]	3.7, 3.6 [3.7]	36, 32 [34]	40, 36 [38]
	Pre (--) BBCH 14 (22)	1.1 1.12	189 188	Forage	3	0.091, 0.073 [0.082]	0.0074, 0.0065 [0.0069]	49, 46 [47]	5.9, 4.9 [5.4]	49, 46 [47]	55, 51 [53]
					7	0.014, 0.015 [0.014]	0.0049, <0.005 [0.0048]	28, 28 [28]	2.8, 2.8 [2.8]	28, 28 [28]	31, 31 [31]
					10	0.020, 0.010 [0.015]	<0.005, <0.005 [<0.0047]	24, 28 [26]	2.6, 2.6 [2.6]	24, 28 [26]	26, 30 [28]
					14	<0.005, <0.005 [<0.005]	<0.005, <0.005 [<0.0047]	14, 15 [15]	1.9, 1.9 [1.9]	14, 15 [15]	16, 17 [16]
				Hay	17	0.017, 0.021 [0.019]	0.0055, 0.0056 [0.0055]	51, 53 [52]	4.4, 4.2 [4.3]	51, 54 [52]	55, 58 [56]
IA-2 Hedrick, Iowa (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (19)	1.12 0.58 0.56	187 189 183	Forage	8	0.025, 0.030 [0.027]	<0.005, <0.005 [<0.0047]	10, 10 [10]	1.6, 1.7 [1.7]	10, 10 [10]	12, 12 [12]
				Hay	18	0.026, 0.021 [0.023]	0.011, 0.010 [0.011]	23, 24 [24]	6.2, 5.8 [6.0]	24, 24 [24]	30, 30 [30]
IL-1 Wyoming, Illinois (MEA)	Pre (--) BBCH 14 (22) BBCH 60 (21)	1.13 0.56 0.56	190 188 184	Forage	7	0.021, 0.029 [0.025]	<0.005, <0.005 [<0.0047]	14, 13 [14]	1.8, 1.8 [1.8]	14, 13 [14]	16, 15 [15]
				Hay	21	0.027, 0.032 [0.030]	<0.005, <0.005 [<0.0047]	21, 22 [21]	0.42, 0.76 [0.59]	21, 22 [21]	21, 23 [22]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
IL-2 Carlyle, Illinois (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (14)	1.15 0.57 0.56	190 195 185	Forage	8	0.021, 0.021 [0.021]	<0.005, <0.005 [<0.0047]	13, 14 [14]	2.0, 2.2 [2.1]	13, 14 [14]	15, 16 [16]
				Hay	18	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	14, 16 [15]	1.3, 1.4 [1.4]	14, 16 [15]	15, 17 [16]
	Pre (--) BBCH 14 (14)	1.11 1.11	191 182	Forage	8	0.030, 0.032 [0.031]	0.0063, 0.0061 [0.0062]	33, 33 [33]	1.3, 1.4 [1.3]	33, 33 [33]	34, 35 [34]
				Hay	18	0.018, 0.016 [0.017]	<0.005, <0.005 [<0.0047]	37, 36 [36]	2.0, 2.3 [2.1]	37, 36 [36]	38, 39 [39]
(DGA)	Pre (--) BBCH 14 (14)	1.13 1.14	193 188	Forage	8	0.027, 0.021 [0.024]	0.0047, 0.0053 [0.0050]	20, 26 [23]	0.89, 0.97 [0.93]	20, 26 [23]	21, 27 [24]
				Hay	18	<0.010, 0.014 [0.012]	<0.005, 0.0052 [0.0049]	25, 34 [29]	4.4, 2.2 [3.3]	25, 34 [29]	29, 36 [33]
IN Rockville, Indiana (MEA)	Pre (--) BBCH 14 (21) BBCH 60 (21)	1.12 0.56 0.55	189 186 176	Forage	7	1.1, 1.3 [1.2]	0.0082, 0.0095 [0.0089]	17, 15 [16]	2.2, 2.2 [2.2]	18, 16 [17]	20, 18 [19]
				Hay	15	0.28, 0.30 [0.29]	0.0070, 0.0076 [0.0073]	32, 27 [30]	6.4, 4.6 [5.5]	33, 27 [30]	39, 32 [35]
KS-1 Cunningham, Kansas (MEA)	Pre (--) BBCH 14 (28) BBCH 60 (8)	1.11 0.55 0.57	187 191 198	Forage	8	0.021, 0.021 [0.021]	<0.005, 0.0048 [0.0048]	19, 20 [19]	1.2, 1.1 [1.1]	19, 20 [19]	20, 21 [20]
				Hay	21	0.021, 0.017 [0.019]	<0.005, 0.0053 [0.0050]	48, 45 [47]	1.1, 1.5 [1.3]	48, 45 [47]	50, 46 [48]
	Pre (--) BBCH 14 (8)	1.1 1.15	193 200	Forage	8	0.024, 0.020 [0.022]	<0.005, 0.0062 [0.0055]	53, 58 [55]	1.1, 0.90 [1.0]	53, 58 [55]	54, 59 [56]
				Hay	21	0.040, 0.038 [0.039]	0.0093, 0.0070 [0.0082]	140, 120 [130]	2.8, 3.1 [2.9]	140, 120 [130]	140, 120 [130]
(DGA)	Pre (--) BBCH 14 (8)	1.11 1.18	193 205	Forage	8	0.019, 0.016 [0.018]	<0.005, <0.005 [<0.0047]	53, 46 [49]	0.96, 1.0 [0.98]	53, 46 [49]	54, 47 [50]
				Hay	21	0.042, 0.037 [0.040]	0.010, 0.0099 [0.0100]	140, 140 [140]	3.6, 3.7 [3.6]	140, 140 [140]	150, 140 [140]
KS-2 Hudson, Kansas (MEA)	Pre (--) BBCH 14 (32) BBCH 60 (13)	1.14 0.55 0.58	191 184 193	Forage	7	0.021, 0.021 [0.021]	<0.005, <0.005 [<0.0047]	21, 21 [21]	2.2, 2.2 [2.2]	21, 21 [21]	23, 23 [23]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				Hay	18	0.021, 0.027 [0.024]	0.0082, 0.0081 [0.0082]	32, 37 [34]	1.4, 1.3 [1.4]	32, 37 [34]	33, 38 [36]
LA Washington, Louisiana (MEA)	Pre (--) BBCH 14 (15) BBCH 60 (15)	1.11 0.55 0.57	188 199 187	Forage	7	0.095, 0.097 [0.096]	<0.005, 0.0059 [0.0053]	19, 20 [19]	2.2, 2.1 [2.2]	19, 20 [19]	21, 22 [22]
				Hay	20	0.092, 0.11 [0.100]	<0.005, 0.0050 [0.0048]	46, 47 [47]	2.0, 1.9 [2.0]	46, 48 [47]	48, 49 [49]
MI Conklin, Michigan (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (26)	1.12 0.56 0.56	184 186 186	Forage	7	0.92, 1.1 [1.0]	0.0059, 0.0062 [0.0061]	10, 8.9 [9.5]	1.3, 1.4 [1.3]	11, 10.0 [11]	12, 11 [12]
				Hay	14	1.2, 0.86 [1.0]	0.013, 0.0090 [0.011]	22, 18 [20]	3.5, 2.4 [2.9]	23, 19 [21]	26, 21 [24]
MN-1 Campbell, Minnesota (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (17)	1.12 0.56 0.56	187 187 187	Forage	7	0.28, 0.16 [0.22]	<0.005, 0.0047 [0.0047]	14, 14 [14]	1.9, 2.0 [1.9]	14, 14 [14]	16, 16 [16]
				Hay	17	0.021, 0.014 [0.017]	<0.005, <0.005 [<0.0047]	34, 30 [32]	2.3, 2.0 [2.1]	34, 30 [32]	37, 32 [34]
	Pre (--) BBCH 14 (17)	1.12 1.13	188 188	Forage	3	1.1, 0.76 [0.92]	0.0062, 0.0082 [0.0072]	37, 32 [34]	2.9, 2.9 [2.9]	38, 33 [35]	41, 36 [38]
					7	0.45, 0.37 [0.41]	0.0062, <0.005 [0.0055]	26, 24 [25]	3.4, 2.8 [3.1]	27, 25 [26]	30, 27 [29]
					10	0.045, 0.046 [0.046]	<0.005, <0.005 [<0.0047]	22, 22 [22]	2.0, 1.6 [1.8]	22, 22 [22]	24, 24 [24]
					14	0.011, 0.016 [0.014]	<0.005, <0.005 [<0.0047]	16, 20 [18]	1.5, 1.8 [1.6]	16, 20 [18]	18, 21 [20]
				Hay	17	0.036, 0.049 [0.042]	<0.005, 0.0052 [0.0049]	64, 69 [67]	4.2, 4.1 [4.1]	64, 69 [67]	69, 73 [71]
MN-2 Fergus Falls, Minnesota (MEA)	Pre (--) BBCH 14 (27) BBCH 60 (18)	1.12 0.56 0.56	188 188 187	Forage	7	0.28, 0.37 [0.32]	<0.005, <0.005 [<0.0047]	15, 14 [15]	2.6, 2.4 [2.5]	15, 15 [15]	18, 17 [17]
				Hay	18	0.096, 0.13 [0.11]	<0.005, <0.005 [<0.0047]	41, 47 [44]	2.0, 4.1 [3.1]	41, 47 [44]	43, 51 [47]
	Pre (--) BBCH 14 (18)	1.12 1.13	187 188	Forage	7	0.54, 0.80 [0.67]	<0.005, 0.0049 [0.0048]	28, 31 [29]	4.0, 4.0 [4.0]	28, 32 [30]	32, 36 [34]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(DGA)				Hay	18	0.25, 0.29 [0.27]	0.0094, 0.0085 [0.0090]	91, 97 [94]	5.9, 5.9 [5.9]	91, 97 [94]	97, 100 [100]
	Pre (--) BBCH 14 (18)	1.12 1.13	187 188	Forage	7	1.2, 1.1 [1.1]	<0.005, 0.0052 [0.0049]	36, 37 [36]	4.8, 5.2 [5.0]	37, 38 [37]	41, 43 [42]
				Hay	18	0.31, 0.32 [0.32]	0.013, 0.0077 [0.010]	100, 100 [100]	6.3, 6.7 [6.5]	100, 100 [100]	110, 110 [110]
MO Fisk, Missouri (MEA)	Pre (--) BBCH 14 (24) BBCH 60 (17)	1.12 0.56 0.57	186 189 189	Forage	7	0.063, 0.063 [0.063]	0.0056, 0.0047 [0.0051]	18, 16 [17]	0.36, 0.38 [0.37]	18, 16 [17]	19, 17 [18]
				Hay	19	0.014, 0.014 [0.014]	<0.005, <0.005 [<0.0047]	22, 22 [22]	0.77, 0.70 [0.73]	22, 22 [22]	22, 23 [23]
ND-1 Carrington, North Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (29)	1.12 0.56 0.57	188 187 188	Forage	7	0.30, 0.29 [0.30]	<0.005, <0.005 [<0.0047]	12, 13 [12]	0.62, 0.84 [0.73]	12, 13 [13]	13, 14 [13]
				Hay	16	0.20, 0.12 [0.16]	0.0062, <0.005 [0.0054]	27, 26 [26]	4.7, 4.7 [4.7]	27, 26 [27]	32, 31 [31]
NE-1 York, Nebraska (MEA)	Pre (--) BBCH 14 (26) BBCH 60 (21)	1.13 0.56 0.56	188 187 187	Forage	7	0.35, 0.26 [0.31]	0.0059, <0.005 [0.0053]	13, 12 [12]	1.5, 1.4 [1.5]	13, 13 [13]	15, 14 [14]
				Hay	14	0.28, 0.29 [0.29]	0.0062, 0.0074 [0.0068]	33, 37 [35]	1.3, 1.5 [1.4]	34, 38 [36]	35, 39 [37]
NE-2 Osceola, Nebraska (MEA)	Pre (--) BBCH 14 (25) BBCH 60 (18)	1.1 0.56 0.57	182 187 188	Forage	8	0.043, 0.059 [0.051]	0.0074, 0.0055 [0.0064]	14, 14 [14]	1.5, 1.6 [1.6]	14, 14 [14]	15, 16 [16]
				Hay	14	0.014, 0.014 [0.014]	0.0066, 0.010 [0.0084]	38, 38 [38]	3.3, 3.6 [3.5]	38, 38 [38]	41, 42 [42]
SC Elko, South Carolina (MEA)	Pre (--) BBCH 14 (30) BBCH 60 (9)	1.12 0.56 0.56	189 193 186	Forage	7	0.068, 0.068 [0.068]	0.0053, <0.005 [0.0050]	20, 19 [19]	2.3, 2.1 [2.2]	20, 19 [20]	22, 21 [22]
				Hay	20	0.057, 0.065 [0.061]	<0.005, <0.005 [<0.0047]	29, 35 [32]	0.17, 0.18 [0.17]	29, 35 [32]	30, 35 [32]
SD-1 Centerville, South Dakota (MEA)	Pre (--) BBCH 14 (31) BBCH 60 (17)	1.09 0.55 0.56	180 184 185	Forage	7	2.3, 2.6 [2.5]	0.0052, 0.0056 [0.0054]	13, 15 [14]	1.6, 2.9 [2.3]	15, 18 [16]	17, 20 [19]
				Hay	18	0.18, 0.20 [0.19]	0.0047, <0.005 [0.0047]	37, 35 [36]	4.4, 3.7 [4.1]	37, 35 [36]	41, 39 [40]
SD-2 Britton, South Dakota	Pre (--) BBCH 14 (32) BBCH 60 (10)	1.12 0.56 0.56	186 187 187	Forage	7	0.42, 0.44 [0.43]	0.0051, 0.0053 [0.0052]	17, 16 [17]	2.7, 1.8 [2.3]	17, 17 [17]	20, 19 [19]

Trial No. Location Year (Salt) [a]	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
(MEA)											
				Hay	14	0.063, 0.034 [0.048]	<0.005, <0.005 [<0.0047]	31, 30 [30]	2.0, 1.7 [1.8]	31, 30 [30]	33, 32 [32]
WI-1 Delavan, Wisconsin (MEA)	Pre (--) BBCH 14 (35) BBCH 60 (15)	1.12 0.52 0.56	183 173 188	Forage	7	0.56, 0.86 [0.71]	<0.005, <0.005 [<0.0047]	15, 16 [15]	4.0, 3.9 [4.0]	15, 17 [16]	19, 21 [20]
				Hay	18	0.16, 0.21 [0.18]	0.0054, <0.005 [0.0050]	31, 32 [32]	2.4, 2.4 [2.4]	32, 33 [32]	34, 35 [35]
WI-2 Fitchburg, Wisconsin (MEA)	Pre (--) BBCH 14 (29) BBCH 60 (6)	0.56, 0.57 0.57 0.56	195, 186 188 190	Forage	10	0.070, 0.057 [0.064]	<0.005, 0.0051 [0.0049]	16, 15 [15]	1.7, 1.9 [1.8]	16, 15 [15]	17, 17 [17]
				Hay	22	0.22, 0.15 [0.19]	0.0062, 0.0048 [0.0055]	60, 61 [61]	7.1, 7.3 [7.2]	60, 61 [61]	68, 68 [68]

[a] Formulation: MEA = Monoethanolamine salt, DGA = Diglycolamine salt

Straw, fodder, and forage of cereal grains and grasses

Maize

Twenty-two residue trials were conducted on dicamba-tolerant maize (MON 87419 variety) as described above (Cereal grains, maize; Report MSL0026526). From those trials, samples of maize forage and stover were harvested 29-71 and 64-132 DALA, respectively. The moisture content for the stover was not specified.

Maize forage samples were stored frozen for up to 225 days, and stover samples for up to 285 days. Residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA were analysed using the method described above. Average concurrent recoveries from forage and stover across all four analytes and across fortifications from 0.01 to ca. 27 mg/kg depending on the matrix and analyte ranged from 94 to 109%. Relative standard deviations ranged from 3.2 to 16%.

Results for maize forage and hay are shown in Table 17.

Table 17 Results of dicamba residue trials in dicamba-tolerant maize forage and stover (variety MON 87419) in the USA (2013 growing season; Report MSL0026526)

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post	1.12 + 0.56 + 0.56	140	--		--	--	--	--		
01PA Germansville, Pennsylvania	Pre (--) BBCH 14-15 (23) BBCH 17-18 (14)	1.14 0.57 0.57	192 190 191	Forage	68	<0.01, <0.01 [<0.01]	0.052, 0.038 [0.045]	0.86, 0.76 [0.81]	1.6, 1.6 [1.6]	0.87, 0.77 [0.82]	2.6, 2.4 [2.5]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				Stover	107	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.42, 0.41 [0.42]	0.16, 0.22 [0.19]	0.43, 0.42 [0.43]	0.60, 0.65 [0.62]
	BBCH 13 BBCH 14-15 (7) BBCH 17-18 (14) BBCH 19 (8)	0.58 0.58 0.58 0.56	194 193 195 189	Forage	60	<0.01, <0.01 [<0.01]	0.049, 0.018 [0.033]	1.3, 0.82 [1.1]	1.6, 1.5 [1.6]	1.3, 0.83 [1.1]	3.0, 2.4 [2.7]
				Stover	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.67, 0.46 [0.57]	0.34, 0.24 [0.29]	0.68, 0.48 [0.58]	1.0, 0.73 [0.88]
02GA Chula, Georgia	Pre (--) BBCH 15 (27) BBCH 18 (12)	1.11 0.54 0.57	182 185 183	Forage	46	<0.01, <0.01 [<0.01]	0.045, 0.050 [0.048]	2.1, 2.0 [2.0]	1.4, 1.2 [1.3]	2.1, 2.0 [2.0]	3.5, 3.3 [3.4]
				Stover	86	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.29, 0.28 [0.28]	0.17, 0.11 [0.14]	0.30, 0.29 [0.29]	0.48, 0.41 [0.45]
	BBCH 13 BBCH 15 (14) BBCH 18 (12) BBCH 19 (10)	0.56 0.55 0.55 0.57	183 188 178 195	Forage	36	<0.01, <0.01 [<0.01]	0.050, 0.076 [0.063]	2.5, 3.4 [3.0]	1.4, 1.8 [1.6]	2.5, 3.4 [3.0]	3.9, 5.3 [4.6]
				Stover	76	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.35, 0.34 [0.34]	0.21, 0.26 [0.24]	0.36, 0.35 [0.35]	0.58, 0.62 [0.60]
03NC Belvidere, N Carolina	Pre (--) BBCH 15-16 (28) BBCH 18 (14)	1.16 0.58 0.56	193 186 188	Forage	54	<0.01, <0.01 [<0.01]	0.056, 0.050 [0.053]	1.2, 0.25 [0.73]	2.1, 0.43 [1.3]	1.2, 0.26 [0.74]	3.4, 0.73 [2.1]
				Stover	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.22, 0.20 [0.21]	0.12, 0.14 [0.13]	0.23, 0.22 [0.22]	0.36, 0.36 [0.36]
	BBCH 13 BBCH 15-16 (7) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	183 181 187 188	Forage	47	<0.01, <0.01 [<0.01]	<0.01, 0.042 [0.026]	0.24, 0.87 [0.55]	1.8, 2.5 [2.1]	0.25, 0.88 [0.56]	2.0, 3.4 [2.7]
				Stover	82	0.013, <0.01 [0.011]	<0.01, <0.01 [<0.01]	0.16, 0.15 [0.16]	0.052, 0.050 [0.051]	0.17, 0.16 [0.17]	0.24, 0.22 [0.23]
04TX Uvalde, Texas	BBCH 0 BBCH 15-16 (47) BBCH 18 (7)	1.11 0.56 0.56	187 185 187	Forage	36	<0.01, 0.019 [0.014]	0.26, 0.50 [0.38]	4.0, 6.6 [5.3]	1.0, 1.4 [1.2]	4.0, 6.6 [5.3]	5.3, 8.5 [6.9]
				Stover	71	0.066, 0.048 [0.057]	1.6, 0.78 [1.2]	17, 14 [16]	3.0, 3.0 [3.0]	17, 14 [16]	22, 18 [20]
	BBCH 12 BBCH 15-16 (23) BBCH 18 (7) BBCH 51 (7)	0.57 0.56 0.56 0.57	188 188 187 189	Forage	29	0.043, 0.033 [0.038]	0.46, 0.40 [0.43]	7.6, 4.8 [6.2]	1.9, 1.6 [1.7]	7.7, 4.9 [6.3]	10, 6.9 [8.4]
				Stover	64	0.051, 0.10 [0.078]	1.0, 0.91 [0.96]	13, 13 [13]	3.8, 4.4 [4.1]	13, 13 [13]	18, 18 [18]
05MI Wright, Michigan	Pre (--) BBCH 14 (34) BBCH 18 (13)	1.14 0.56 0.56	187 187 189	Forage	71	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.23, 0.42 [0.33]	0.073, 0.19 [0.13]	0.24, 0.43 [0.34]	0.33, 0.63 [0.48]
				Stover	121	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.068, 0.052 [0.060]	0.038, 0.024 [0.031]	0.078, 0.062 [0.070]	0.13, 0.096 [0.11]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 12 BBCH 14 (9) BBCH 18 (13) BBCH 19 (11)	0.56 0.56 0.56 0.56	186 186 190 189	Forage	60	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.18, 0.37 [0.28]	0.066, 0.17 [0.12]	0.19, 0.38 [0.29]	0.27, 0.56 [0.41]
				Stover	110	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.077, 0.054 [0.065]	0.053, 0.030 [0.042]	0.087, 0.064 [0.075]	0.15, 0.10 [0.13]
06KS Troy, Kansas	BBCH 5 BBCH 13 (22) BBCH 18 (8)	1.13 0.57 0.65	190 195 221	Forage	55	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.81, 0.70 [0.76]	0.35, 0.33 [0.34]	0.82, 0.71 [0.77]	1.2, 1.1 [1.1]
				Stover	132	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.032, 0.050 [0.041]	<0.01, 0.022 [0.016]	0.042, 0.060 [0.051]	0.062, 0.093 [0.077]
	BBCH 12 BBCH 13 (9) BBCH 18 (8) BBCH 39 (13)	0.58 0.57 0.57 0.57	195 194 195 196	Forage	42	<0.01, <0.01 [<0.01]	0.034, 0.016 [0.025]	1.8, 1.8 [1.8]	1.8, 1.2 [1.5]	1.8, 1.8 [1.8]	3.7, 3.1 [3.4]
				Stover	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.18, 0.20 [0.19]	0.10, 0.11 [0.11]	0.19, 0.21 [0.20]	0.30, 0.32 [0.31]
07KS St. John, Kansas	BBCH 1 BBCH 16 (32) BBCH 18 (7)	1.12 0.57 0.55	185 188 187	Forage	58	<0.01, <0.01 [<0.01]	0.030, 0.010 [0.020]	0.63, 0.42 [0.53]	1.2, 0.15 [0.68]	0.64, 0.43 [0.54]	1.9, 0.60 [1.2]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.74, 0.53 [0.64]	0.12, 0.080 [0.10]	0.75, 0.54 [0.65]	0.88, 0.63 [0.76]
	BBCH 12 BBCH 16 (18) BBCH 18 (7) BBCH 19 (21)	0.57 0.56 0.55 0.58	188 185 185 191	Forage	37	<0.01, <0.01 [<0.01]	0.019, 0.033 [0.026]	0.81, 1.1 [0.94]	0.45, 0.76 [0.60]	0.82, 1.1 [0.95]	1.3, 1.9 [1.6]
				Stover	70	<0.01, <0.01 [<0.01]	0.016, 0.011 [0.014]	1.5, 1.0 [1.3]	0.29, 0.27 [0.28]	1.5, 1.0 [1.3]	1.8, 1.3 [1.6]
08IA Black Hawk, Iowa	Pre (--) BBCH 15 (35) BBCH 18 (13)	1.11 0.55 0.56	186 183 186	Forage	63	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.32, 0.30 [0.31]	0.39, 0.32 [0.35]	0.33, 0.31 [0.32]	0.72, 0.64 [0.68]
				Stover	106	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.29, 0.40 [0.34]	0.18, 0.21 [0.19]	0.30, 0.41 [0.35]	0.49, 0.62 [0.56]
	BBCH 13 BBCH 15 (12) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.55 0.56	191 186 183 189	Forage	56	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.40, 0.47 [0.43]	0.44, 0.70 [0.57]	0.41, 0.48 [0.44]	0.86, 1.2 [1.0]
				Stover	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.35, 0.35 [0.35]	0.18, 0.20 [0.19]	0.36, 0.36 [0.36]	0.55, 0.56 [0.56]
09IL Carlyle, Illinois	Pre (--) BBCH 15-16 (32) BBCH 18 (13)	1.13 0.57 0.56	196 194 193	Forage	50	<0.01, <0.01 [<0.01]	0.040, 0.067 [0.053]	0.69, 0.72 [0.70]	0.51, 0.72 [0.61]	0.70, 0.73 [0.71]	1.2, 1.5 [1.4]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, 0.014 [0.012]	0.41, 0.48 [0.44]	0.18, 0.25 [0.22]	0.42, 0.49 [0.45]	0.61, 0.75 [0.68]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 14 BBCH 15-16 (9) BBCH 18 (13) BBCH 19 (7)	0.56 0.56 0.56 0.56	179 190 193 191	Forage	43	<0.01, <0.01 [<0.01]	0.083, 0.085 [0.084]	1.6, 1.4 [1.5]	1.4, 1.8 [1.6]	1.6, 1.5 [1.5]	3.1, 3.3 [3.2]
				Stover	84	<0.01, 0.015 [0.012]	<0.01, 0.024 [0.017]	0.62, 0.89 [0.76]	0.48, 0.84 [0.66]	0.63, 0.91 [0.77]	1.1, 1.8 [1.4]
10IL Highland, Illinois	Pre (--) BBCH 15 (25) BBCH 18 (16)	1.16 0.57 0.56	196 182 194	Forage	49	<0.01, <0.01 [<0.01]	0.033, 0.042 [0.038]	0.69, 1.1 [0.89]	0.77, 1.2 [0.98]	0.70, 1.1 [0.90]	1.5, 2.3 [1.9]
				Stover	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.31, 0.48 [0.40]	0.13, 0.18 [0.15]	0.32, 0.49 [0.40]	0.46, 0.68 [0.57]
	BBCH 13 BBCH 15 (7) BBCH 18 (16) BBCH 18 (9)	0.58 0.57 0.56 0.56	194 182 192 193	Forage	40	<0.01, 0.011 [0.011]	0.029, 0.092 [0.061]	0.93, 1.6 [1.3]	1.1, 1.6 [1.4]	0.94, 1.6 [1.3]	2.1, 3.3 [2.7]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.37, 0.66 [0.52]	0.29, 0.35 [0.32]	0.38, 0.67 [0.53]	0.69, 1.0 [0.86]
11IL Camp Grove, Illinois	BBCH 0 BBCH 14 (30) BBCH 18 (9)	1.13 0.56 0.56	188 185 183	Forage	64	<0.01, <0.01 [<0.01]	0.011, <0.01 [0.010]	0.20, 0.14 [0.17]	0.082, 0.070 [0.076]	0.21, 0.15 [0.18]	0.31, 0.23 [0.27]
				Stover	100	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.084, 0.10 [0.094]	0.040, 0.053 [0.046]	0.094, 0.11 [0.10]	0.14, 0.18 [0.16]
	BBCH 13 BBCH 14 (7) BBCH 18 (9) BBCH 19 (15)	0.55 0.56 0.55 0.57	178 182 181 190	Forage	49	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.32, 0.31 [0.31]	0.26, 0.32 [0.29]	0.33, 0.32 [0.32]	0.60, 0.65 [0.62]
				Stover	91	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.36, 0.26 [0.31]	0.21, 0.18 [0.19]	0.37, 0.27 [0.32]	0.59, 0.46 [0.52]
12IL Stewardson, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (12)	1.12 0.55 0.55	188 180 180	Forage	65	<0.01, <0.01 [<0.01]	0.038, 0.022 [0.030]	0.30, 0.31 [0.30]	0.12, 0.82 [0.47]	0.31, 0.32 [0.32]	0.47, 1.2 [0.82]
				Stover	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.025, 0.084 [0.055]	<0.01, 0.034 [0.022]	0.035, 0.094 [0.065]	0.055, 0.14 [0.097]
	BBCH 12 BBCH 15 (11) BBCH 18 (12) BBCH 19 (11)	0.56 0.56 0.56 0.56	192 184 184 187	Forage	54	<0.01, <0.01 [<0.01]	0.21, 0.099 [0.16]	0.62, 0.92 [0.77]	1.3, 1.8 [1.5]	0.63, 0.93 [0.78]	2.2, 2.8 [2.5]
				Stover	94	0.015, <0.01 [0.012]	<0.01, <0.01 [<0.01]	0.39, 0.24 [0.31]	0.13, 0.11 [0.12]	0.40, 0.25 [0.33]	0.54, 0.37 [0.46]
13IL Duvall, Illinois	BBCH 3 BBCH 16 (25) BBCH 18 (16)	1.12 0.57 0.56	190 192 192	Forage	67	<0.01, <0.01 [<0.01]	0.087, 0.024 [0.056]	0.57, 0.51 [0.54]	1.2, 0.49 [0.84]	0.58, 0.52 [0.55]	1.8, 1.0 [1.4]
				Stover	108	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.25, 0.33 [0.29]	0.087, 0.13 [0.11]	0.26, 0.34 [0.30]	0.36, 0.49 [0.42]
	BBCH 12 BBCH 16 (7) BBCH 18 (16)	0.56 0.58 0.57	190 196 196	Forage	55	<0.01, <0.01 [<0.01]	0.036, 0.012 [0.024]	0.49, 0.42 [0.46]	0.57, 0.28 [0.43]	0.50, 0.43 [0.47]	1.1, 0.72 [0.92]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	BBCH 24 (9)	0.57	189	Stover	96	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.40, 0.56 [0.48]	0.33, 0.57 [0.45]	0.41, 0.57 [0.49]	0.75, 1.2 [0.95]
14IL Highland, Illinois	BBCH 0 BBCH 15 (28) BBCH 18 (14)	1.12 0.57 0.57	195 195 195	Forage	63	<0.01, <0.01 [<0.01]	0.030, 0.015 [0.023]	0.68, 0.47 [0.57]	0.60, 0.38 [0.49]	0.69, 0.48 [0.58]	1.3, 0.88 [1.1]
				Stover	97	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.25, 0.16 [0.20]	0.11, 0.089 [0.100]	0.26, 0.17 [0.22]	0.38, 0.27 [0.32]
	BBCH 12 BBCH 15 (14) BBCH 18 (14) BBCH 39 (9)	0.56 0.57 0.56 0.57	194 194 195 191	Forage	54	<0.01, <0.01 [<0.01]	0.058, 0.020 [0.039]	1.3, 1.0 [1.1]	1.1, 0.88 [0.99]	1.3, 1.0 [1.2]	2.4, 1.9 [2.2]
				Stover	88	<0.01, <0.01 [<0.01]	<0.01, 0.010 [0.010]	0.50, 0.83 [0.67]	0.72, 0.73 [0.73]	0.51, 0.84 [0.68]	1.2, 1.6 [1.4]
15IN Pickard, Indiana	BBCH 0 BBCH 15 (29) BBCH 18 (12)	1.16 0.56 0.56	194 188 193	Forage	58	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.14, 0.78 [0.46]	0.15, 0.94 [0.54]	0.15, 0.79 [0.47]	0.31, 1.7 [1.0]
				Stover	119	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.15, 0.23 [0.19]	0.15, 0.28 [0.21]	0.16, 0.24 [0.20]	0.32, 0.52 [0.42]
	BBCH 12 BBCH 15 (15) BBCH 18 (12) BBCH 19 (14)	0.57 0.56 0.57 0.56	193 188 195 187	Forage	44	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.30, 0.11 [0.21]	0.22, 0.050 [0.14]	0.31, 0.12 [0.22]	0.55, 0.18 [0.36]
				Stover	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.22, 0.24 [0.23]	0.19, 0.19 [0.19]	0.23, 0.25 [0.24]	0.43, 0.45 [0.44]
16MO Kirksville, Missouri	Pre (--) BBCH 14 (26) BBCH 18 (14)	1.11 0.56 0.56	186 188 184	Forage	57	<0.01, <0.01 [<0.01]	0.021, 0.015 [0.018]	0.45, 0.43 [0.44]	0.37, 0.27 [0.32]	0.46, 0.44 [0.45]	0.85, 0.73 [0.79]
				Stover	89	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.45, 0.58 [0.52]	0.14, 0.29 [0.21]	0.46, 0.59 [0.53]	0.61, 0.89 [0.75]
	BBCH 12 BBCH 14 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.57	189 187 185 192	Forage	50	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.68, 0.76 [0.72]	0.28, 0.29 [0.28]	0.69, 0.77 [0.73]	0.98, 1.1 [1.0]
				Stover	82	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.51, 0.45 [0.48]	0.19, 0.20 [0.20]	0.52, 0.46 [0.49]	0.72, 0.67 [0.70]
17MO Broseley, Missouri	BBCH 5 BBCH 16 (21) (9)	1.12 0.57 0.56	187 188 187	Forage	68	<0.01, <0.01 [<0.01]	0.011, 0.060 [0.036]	0.34, 1.1 [0.71]	0.063, 0.17 [0.12]	0.35, 1.1 [0.72]	0.42, 1.3 [0.87]
				Stover	95	<0.01, <0.01 [<0.01]	0.015, <0.01 [0.013]	0.58, 0.42 [0.50]	0.14, 0.13 [0.14]	0.59, 0.43 [0.51]	0.75, 0.57 [0.66]
	BBCH 14 BBCH 16 (7) BBCH 18 (9) BBCH 19 (7)	0.57 0.56 0.56 0.57	188 187 187 188	Forage	61	<0.01, <0.01 [<0.01]	<0.01, 0.016 [0.013]	0.42, 0.49 [0.45]	0.046, 0.044 [0.045]	0.43, 0.50 [0.46]	0.48, 0.56 [0.52]
				Stover	88	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.37, 0.28 [0.33]	0.12, 0.11 [0.12]	0.38, 0.29 [0.34]	0.52, 0.41 [0.47]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
18NE Tabor, Nebraska	BBCH 0 BBCH 15 (32) BBCH 18 (8)	1.11 0.56 0.57	178 179 184	Forage	56	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.50, 0.61 [0.55]	0.70, 0.70 [0.70]	0.51, 0.62 [0.56]	1.2, 1.3 [1.3]
				Stover	118	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.12, 0.13 [0.12]	0.10, 0.10 [0.10]	0.12, 0.14 [0.13]	0.24, 0.25 [0.24]
	BBCH 12 BBCH 15 (18) BBCH 18 (8) BBCH 19 (7)	0.57 0.56 0.57 0.56	184 180 182 188	Forage	49	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.53, 0.29 [0.41]	0.39, 0.23 [0.31]	0.54, 0.30 [0.42]	0.94, 0.54 [0.74]
				Stover	111	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.18, 0.15 [0.17]	0.13, 0.16 [0.14]	0.19, 0.16 [0.18]	0.33, 0.33 [0.33]
19NE Henderson, Nebraska	BBCH 0 BBCH 16 (34) BBCH 19 (7)	1.1 0.55 0.56	191 191 191	Forage	53	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	1.6, 1.8 [1.7]	0.96, 0.97 [0.97]	1.6, 1.8 [1.8]	2.6, 2.8 [2.7]
				Stover	99	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	1.1, 0.56 [0.85]	0.21, 0.22 [0.22]	1.2, 0.57 [0.86]	1.4, 0.79 [1.1]
	BBCH 13 BBCH 16 (8) BBCH 19 (7) BBCH 19 (7)	0.55 0.56 0.57 0.56	190 194 191 194	Forage	46	<0.01, <0.01 [<0.01]	0.049, 0.020 [0.034]	2.6, 2.8 [2.7]	1.1, 1.6 [1.4]	2.6, 2.8 [2.7]	3.8, 4.4 [4.1]
				Stover	92	<0.01, <0.01 [<0.01]	0.010, 0.013 [0.012]	1.1, 1.3 [1.2]	0.90, 0.81 [0.86]	1.1, 1.3 [1.2]	2.0, 2.1 [2.1]
20NE Brunswick, Nebraska	BBCH 0 BBCH 15 (30) BBCH 18 (7)	1.09 0.55 0.56	188 190 192	Forage	54	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.65, 0.18 [0.42]	0.50, 0.12 [0.31]	0.66, 0.20 [0.43]	1.2, 0.33 [0.74]
				Stover	101	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.19, 0.052 [0.12]	0.054, 0.020 [0.037]	0.20, 0.062 [0.13]	0.27, 0.093 [0.18]
	BBCH 12 BBCH 15 (10) BBCH 18 (7) BBCH 19 (13)	0.56 0.56 0.56 0.56	190 191 191 194	Forage	41	<0.01, <0.01 [<0.01]	0.026, 0.028 [0.027]	1.8, 0.78 [1.3]	2.9, 0.31 [1.6]	1.9, 0.79 [1.3]	4.8, 1.1 [3.0]
				Stover	88	0.017, 0.015 [0.016]	0.029, 0.032 [0.031]	1.9, 1.7 [1.8]	2.0, 1.4 [1.7]	2.0, 1.7 [1.8]	4.0, 3.1 [3.5]
21SD Bushnell, S. Dakota	BBCH 0 BBCH 16 (32) BBCH 18 (7)	1.12 0.58 0.56	188 193 182	Forage	64	<0.01, <0.01 [<0.01]	0.011, 0.016 [0.013]	1.8, 1.7 [1.7]	1.1, 1.0 [1.1]	1.8, 1.7 [1.7]	2.9, 2.7 [2.8]
				Stover	112	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.23, 0.25 [0.24]	0.091, 0.090 [0.090]	0.24, 0.26 [0.25]	0.34, 0.36 [0.35]
	BBCH 13 BBCH 16 (12) BBCH 18 (7) BBCH 19 (7)	0.56 0.56 0.57 0.56	196 187 188 183	Forage	57	<0.01, <0.01 [<0.01]	<0.01, 0.020 [0.015]	0.38, 1.5 [0.92]	0.19, 0.23 [0.21]	0.39, 1.5 [0.93]	0.58, 1.7 [1.1]
				Stover	105	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.11, 0.14 [0.12]	0.092, 0.081 [0.086]	0.12, 0.15 [0.13]	0.22, 0.24 [0.23]
22WI Richmond, Wisconsin	Pre (--) BBCH 15 (33) BBCH 18 (14)	1.11 0.56 0.56	187 184 181	Forage	67	<0.01, <0.01 [<0.01]	0.013, <0.01 [0.012]	0.49, 0.71 [0.60]	0.29, 0.16 [0.22]	0.50, 0.72 [0.61]	0.80, 0.88 [0.84]

Trial No. Location	Application			Matrix	DALA	Dicamba-equivalent residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha			Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
				Stover	120	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.29, 0.14 [0.21]	0.094, 0.081 [0.088]	0.30, 0.15 [0.22]	0.41, 0.24 [0.32]
	BBCH 13 BBCH 15 (10) BBCH 18 (14) BBCH 19 (7)	0.56 0.56 0.56 0.56	189 185 181 187	Forage	60	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.58, 1.3 [0.95]	0.11, 0.15 [0.13]	0.59, 1.3 [0.96]	0.71, 1.5 [1.1]
				Stover	113	<0.01, <0.01 [<0.01]	<0.01, <0.01 [<0.01]	0.42, 0.32 [0.36]	0.17, 0.15 [0.16]	0.42, 0.32 [0.38]	0.60, 0.49 [0.54]

Oilseeds

Cotton

Three residue trials were conducted on dicamba-tolerant cotton (MON 88701 variety) from which samples of gin trash were collected; a summary of the trials is described above (Oilseeds, cotton; Report MSL0024072).

Gin trash samples were stored frozen for up to 26 days. Residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA were analysed using the method described above. Average concurrent recoveries across all four analytes and across fortifications from 0.04 to ca 100 mg/kg depending on the matrix and analyte ranged from 90 to 121%. Relative standard deviations ranged from 3.4 to 17%.

Results for maize forage and hay are shown in Table 18.

Table 18 Results of dicamba residue trials in dicamba-tolerant cotton gin trash (variety MON 88701) in the USA (2010 growing season; Report MSL0024072)

Trial No. Location Salt [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
Critical GAP USA	1 pre + 2 post with a 7-day retreatment interval	1.12 + 0.56 + 0.56	140	7	--	--	--	--	--	--
OK2 Dill City, Oklahoma (DGA)	ns (--) ns (28) BBCH 65 (49)	1.1 0.56 0.55	178 188 184	82	<0.04, <0.04 [<0.04]	<0.037, <0.037 [<0.037]	0.42, 0.49 [0.45]	0.17, 0.23 [0.20]	0.46, 0.53 [0.49]	0.62, 0.76 [0.69]
	ns (--) ns (49) BBCH 97 (32) ns (43)	0.57 0.56 0.56 0.55	189 188 190 184	7	3.2, 3.1 [3.1]	<0.037, <0.037 [<0.037]	1.8, 2.0 [1.9]	0.49, 0.40 [0.44]	5.0, 5.1 [5.0]	5.5, 5.5 [5.5]
TX2 Levelland, Texas (DGA)	ns (--) ns (36) BBCH 65 (41)	1.1 0.57 0.56	191 193 188	84	<0.04, <0.04 [<0.04]	<0.037, <0.037 [<0.037]	1.8, 1.5 [1.7]	0.42, 0.70 [0.56]	1.9, 1.6 [1.7]	2.3, 2.3 [2.3]
	ns (--) ns (41) ns (41) BBCH 89 (37)	0.55 0.55 0.56 0.55	187 187 191 187	6	16, 13 [15]	<0.037, <0.037 [<0.037]	6.4, 3.2 [4.8]	3.2, 1.5 [2.4]	23, 17 [20]	26, 18 [22]
TX3 Wolfforth, Texas (DGA)	ns (--) ns (41) BBCH 65 (36)	1.1 0.57 0.55	188 194 185	84	<0.04, <0.04 [<0.04]	<0.037, <0.037 [<0.037]	0.91, 0.52 [0.72]	0.32, 0.31 [0.31]	0.95, 0.56 [0.76]	1.3, 0.87 [1.1]

Trial No. Location Salt [a]	Application			DALA	Residues (mg/kg) [Mean]					
	Timing (interval, days)	kg ai/ha	L/ha		Dicamba	5-OH dicamba	DCSA	DCGA	For max. res.	For risk
	ns (--) ns (36) ns (48) BBCH 89 (30)	0.56 0.56 0.56 0.56	189 188 189 188	6	24, 22 [23]	<0.037, <0.037 [<0.037]	6.7, 6.5 [6.6]	4.3, 3.9 [4.1]	30, 29 [30]	35, 33 [34]

[a] Formulation: DGA = Diglycolamine salt

FATE OF RESIDUES DURING PROCESSING

Soya bean

Of the soya bean field trials described above (Report MSL0023061), two were designated for producing seed for processing into hulls, meal, oil, lecithin, flour, protein isolates, soya milk, and tofu. In those trials, soya bean plants were treated at an exaggerated rate consisting of 1.12 kg ai/ha preemergence and 2.24 kg ai/ha at BBCH 60. Soya bean seeds were harvested at maturity and transported frozen to the processing facility.

Processing was done under simulated commercial practices. Seeds were dried to a moisture content of <13.5% followed by cleaning via aspiration and screening. A portion of the cleaned seeds was soaked in water for 12 h, ground and filtered to remove solids, and the resulting soya milk was cooked (ca 93 °C, 10min). Calcium sulfate was slowly added to an aliquot of the cooked soya milk to produce tofu. Soya bean seeds not used for milk/tofu production were processed in a roller mill and aspirated to separate the kernel from the hull. Kernels were adjusted to a moisture content of 13.5%, allowed to equilibrate for 12 hours and then heated and flaked. Flakes were used to produce the remaining processed commodities. Flakes were extruded and turned into collets by steam injection and compression. Following drying, the collets were ground and extracted three times with hexane to extract oil. A portion of the extracted collets were dried, ground, and screened to produce defatted flour. A portion of the defatted flour was freeze dried to produce protein isolate and protein concentrate. A separate portion of the extracted collets were toasted and underwent steam injection to form toasted soya bean meal. Crude oil from the extraction of the collets underwent heated vacuum evaporation to remove the hexane. The crude oil was hydrated and filtered to produce degummed oil and crude lecithin. The degummed oil was processed with NaOH to produce refined oil and soapstock. Finally, the refined oil was bleached and deodorized.

The processed commodities were analysed for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above. Residues of dicamba and 5-hydroxydicamba were below the LOQ (0.013 mg/kg and 0.021 mg/kg, respectively) in all samples of soya bean seed and processed commodities (Table 19).

Table 19 Residues of dicamba metabolites DCSA and DCGA in processed products derived from dicamba-tolerant soya beans (Report MSL0023061)

Matrix	Trial	Residues, mg/kg [mean]			
		Dicamba	5-OH Dicamba	DCSA	DCGA
Seed	NE-1	<0.005	<0.02	0.062, 0.064, 0.062, 0.071 [0.065]	0.055, 0.053, 0.056, 0.055 [0.055]
	WI-1	<0.005	<0.02	0.168, 0.172, 0.167, 0.183 [0.173]	0.139, 0.148, 0.138, 0.144 [0.142]
Hulls	NE-1	<0.02	<0.02	0.082, 0.082, 0.081, 0.082 [0.082]	0.052, 0.055, 0.056, 0.054 [0.054]
	WI-1	<0.02	<0.02	0.251, 0.254, 0.271, 0.276 [0.263]	0.136, 0.144, 0.146, 0.143 [0.142]
Toasted defatted meal	NE-1	<0.02	<0.02	0.076, 0.079, 0.074, 0.076 [0.076]	0.067, 0.068, 0.070, 0.072 [0.069]
	WI-1	<0.02	<0.02	0.254, 0.268, 0.253, 0.266 [0.260]	0.185, 0.198, 0.189, 0.193 [0.191]
Degummed oil	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
RBD oil	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]

Matrix	Trial	Residues, mg/kg [mean]			
		Dicamba	5-OH Dicamba	DCSA	DCGA
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
Crude lecithin	NE-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]
	WI-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]
Defatted flour	NE-1	<0.05	<0.05	0.074, 0.070, 0.071, 0.069 [0.071]	0.068, 0.067, 0.067, 0.066 [0.067]
	WI-1	<0.05	<0.05	0.242, 0.243, 0.239, 0.251 [0.244]	0.187, 0.180, 0.181, 0.178 [0.182]
Protein isolate	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	0.029, 0.028 [0.028]	<0.02, <0.02 [<0.02]
Protein concentrate	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
Soya milk	NE-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
	WI-1	<0.02	<0.02	<0.02, <0.02, <0.02, <0.02 [<0.02]	<0.02, <0.02, <0.02, <0.02 [<0.02]
Tofu	NE-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]
	WI-1	<0.2	<0.2	<0.2, <0.2, <0.2, <0.2 [<0.2]	<0.2, <0.2, <0.2, <0.2 [<0.2]

Table 20 Processing factor for residues of dicamba in soya bean matrices (Report MSL0023061)

Commodity	Mean residues, mg/kg		Processing factors [a]	
	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA
Seed	0.065, 0.173	0.120, 0.315	--	--
Hulls	0.082, 0.263	0.136, 0.405	1.26, 1.52	1.13, 1.29
Toasted defatted meal	0.076, 0.260	0.145, 0.451	1.17, 1.50	1.21, 1.43
Degummed oil	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
RBD oil	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
Crude lecithin	<0.2, <0.2	<0.2, <0.2	<3.08, <1.16	<1.67, <0.63
Defatted flour	0.071, 0.244	0.138, 0.426	1.09, 1.41	1.15, 1.35
Protein isolate	<0.02, 0.028	<0.02, 0.048	<0.31, 0.16	<0.17, 0.15
Protein conc.	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
Soya milk	<0.02, <0.02	<0.02, <0.02	<0.31, <0.12	<0.17, <0.06
Tofu	<0.2, <0.2	<0.2, <0.2	<3.08, <1.16	<1.67, <0.63

[a] Residues of dicamba and 5-OH dicamba are assumed to be zero based on the results of metabolism and field trial data.

Maize

Of the maize field trials described above (Report MSL0026526), two were designated for producing grain for processing into grits, meal, flour starch, and oil. In those trials, maize plants were treated at an exaggerated rate consisting of 5.6 kg ai/ha pre-emergence and 2.8 kg ai/ha each at BBCH 15 and BBCH 18. Maize grain samples were harvested at maturity and transported frozen to the processing facility. Processing was done under simulated commercial practices. Samples were dried to a moisture content of 10–15%. Samples were then cleaned by aspiration and screening.

For dry milling, kernels were adjusted to a moisture content of 21% and then cracked in a disk mill. The resulting stock was dried and then put through a screening process to obtain bran, germ, large grits, grits, meal, and flour. A portion of the germ was heated, flaked, and then submerged three times in hexane to extract the oil. The crude oil was separated from hexane using heated vacuum evaporation. The crude oil was processed with NaOH to produce refined oil and soapstock. Finally, the refined oil was bleached and deodorized.

For wet milling, the grain was steeped in hot water for 22–48 hours, passed through a disc mill, and the resulting germ and hull fractions were separated by water centrifuge. The germ and hull were dried and then separated by aspiration and screening. Starch and gluten were isolated from the processing water by centrifugation. Germ samples were adjusted to a moisture content of 12% and

flaked. The flake was pressed to produce crude oil and presscake; the latter was extracted three times with hexane to liberate the remaining crude oil. Hexane was removed by heated vacuum evaporation and the pressed and extracted crude oil fractions were combined. The crude oil was alkali refined, bleached, and deodorized.

The processed commodities were analysed for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above.

Residues of all four analytes were < 0.01 mg/kg in all samples of grain and processed maize commodities, and processing factors could not be calculated.

Cotton

Of the cotton field trials described above (Report MSL0024072), bulk seed samples were collected from the MO1 and TX4 sites for processing into oil. In those trials cotton plants were treated at rates consisting of either 1.12 kg ai/ha pre-emergence and 0.56 kg each at BBCH 16 and BBCH 60 + 15 days, or 0.56 kg ai/ha each at BBCH 16, BBCH 60 + 15 days, BBCH 80 and BBCH 99 (7 days pre-harvest). Processing was done under simulated commercial practices.

Samples of ginned cotton seed were delinted and cracked to separate the hull from the kernel. Kernels were stored frozen for a maximum of 28 days prior to processing. Kernels were heated and flaked, and the flaked material was processed through an extruder with steam injection. The resulting collets were dried and then extracted three times with hexane. The extract was separated from the meal, which was desolventized and placed into frozen storage. were dried to a moisture content of 10–15%. Samples were then cleaned by aspiration and screening. Crude oil from the extraction of the collets underwent vacuum evaporation to remove the hexane. The crude oil was processed with NaOH to produce refined oil and soapstock. Finally, the refined oil was bleached and deodorized.

The processed commodities were analysed for residues of dicamba, 5-hydroxydicamba, DCSA, and DCGA using the method described above.

Table 21 Residues of dicamba in processed products derived from dicamba-tolerant cotton (Report MSL0024072)

Matrix	Trial	Treatment	Residues, mg/kg			
			Dicamba	5-OH dicamba	DCSA	DCGA
Undelinted Seed	MO1	2[a]	<0.02	<0.01	0.02	<0.005
		4[b]	0.697	<0.01	0.027	0.035
	TX4	2	<0.02	<0.01	<0.005	<0.005
		4	0.602	<0.01	0.048	0.040
Hulls	MO1	2	<0.02	<0.02	0.021	0.022
		4	0.697	<0.02	0.026	0.045
	TX4	2	<0.02	<0.02	0.020	0.013
		4	0.158	<0.02	0.057	0.084
Meal	MO1	2	<0.02	<0.02	<0.02	<0.02
		4	0.217	<0.02	<0.02	0.02
	TX4	2	<0.02	<0.02	0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
Alkali refined oil	MO1	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
	TX4	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
RBD oil	MO1	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02
	TX4	2	<0.02	<0.02	<0.02	<0.02
		4	<0.02	<0.02	<0.02	<0.02

[a] Treatment 2 (Diglycolamine salt): Pre-emergence at 1.12 kg/ha + BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha

[b] Treatment 4 (Diglycolamine salt): BBCH 16 at 0.56 kg/ha + (BBCH 60 + 15 days) at 0.56 kg/ha, + BBCH 80 at 0.56 kg/ha + BBCH 99 at 0.56 kg/ha (7 days pre-harvest)

Since residues from Treatment 2 were <LOQ in all RAC samples of cotton seed, the Meeting calculated processing factors for cotton commodities (Table 22) based on Treatment 4 only.

Table 22 Processing factor for residues of dicamba in cotton seed matrices (Report MSL0024072)

Commodity	Mean residues, mg/kg		Processing factors [mean or best estimate] [a]	
	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA	Dicamba + total DCSA	Dicamba + 5-OH Dicamba + total DCSA + total DCGA
Seed	0.724, 0.650	0.759, 0.690	--	--
Hulls	0.723, 0.215	0.768, 0.299	0.999, 0.331 [0.665]	1.012, 0.433 [0.723]
Meal	0.237, <0.04	0.257, <0.06	0.365, <0.062 [0.365]	0.372, <0.087 [0.372]
Alkali refined oil	<0.04, <0.04	<0.06, <0.06	<0.055, <0.062	<0.079, <0.087
RBD oil	<0.04, <0.04	<0.06, <0.06	<0.055, <0.062	<0.079, <0.087

[a] Residues of 5-OH dicamba are assumed to be zero based on the results of metabolism and field trial data.

RESIDUE AND ANALYTICAL ASPECTS

Dicamba is a systemic broad-spectrum herbicide. It was first evaluated by the JMPR in 2010 (T, R). The latest residue evaluation was conducted in 2013 (R).

The 2010 JMPR established an ADI for dicamba of 0–0.3 mg/kg bw and an ARfD of 0.5 mg/kg bw. Also, the following residue definitions based on metabolism studies with conventional crops have been established:

Definition of the residue for compliance with the MRL for plant commodities: *dicamba;*

Definition of the residue for dietary risk assessment for plant commodities: *sum of dicamba and 5-OH dicamba, expressed as dicamba;*

Definition of the residue for compliance with the MRL and dietary risk assessment for animal commodities: *sum of dicamba and DCSA, expressed as dicamba.*

The residue is not fat soluble.

Dicamba was scheduled at the Fiftieth Session of the CCPR for the evaluation of additional uses by the 2019 Extra JMPR. Studies submitted by the sponsor include nature of the residue studies, field trials, and processing studies in dicamba-tolerant varieties of soya bean, maize, and cotton, and storage stability in soya bean and cotton.

All application rates are expressed as dicamba acid-equivalents.

Plant metabolism

Plant metabolism studies were conducted with dicamba uniformly labelled in the phenyl ring. Treatments were made to dicamba-tolerant varieties of soya bean, maize, and cotton each, either as pre-emergence applications (PRE) on the day of planting or post-emergence of the crop (POE). Tolerance is conveyed by expression of a dicamba mono-oxygenase protein system that oxidizes dicamba to DCSA. The major residue profiles between the two treatment regimens were very similar across all matrices. Since residues were considerably higher following POE treatment than PRE treatment, the Meeting has focused on the POE treatments in its consideration of the plant metabolism studies. Quantifiable levels of radioactivity were observed in the control samples from the metabolism studies. The Meeting is not concerned about this given the interspersing of control plants with treated plants and the metabolism of dicamba to volatile radiolabelled compounds (e.g., $^{14}CO_2$) that could be taken up by the control plants.

In a study illustrating the metabolism of dicamba in dicamba-tolerant soya bean, [phenyl-U-^{14}C]dicamba was applied to greenhouse-grown soya bean either on the day of planting (PRE) or 29 days after planting (DAP) (POE; BBCH 60 (first flowers opened)) at a target rate of 2.8 kg ai/ha.

PRE samples consisted of immature foliage collected 14 DAP (3.2 mg eq/kg TRR), forage harvested 36 DAP (1.4 mg eq/kg), hay harvested 56 DAP (1.1 mg eq/kg) and seed harvested 112 DAP (0.29 mg eq/kg). POE samples were collected as forage 36 DALA (134 mg eq/kg), hay 56 days after last application (DALA) (39 mg eq/kg), and seed 112 DALA (0.39 mg eq/kg).

Extractability of radioactivity into acetonitrile/water solvent was high (>91% TRR) for leafy matrices and lower (ca. 60% TRR) for seeds regardless of the treatment timing. Hexane extracted an additional 11–14% TRR from seeds.

Dicamba was a major predominant residue following POE treatment in forage (24% TRR, 32 mg/kg) and hay (12% TRR, 4.8 mg/kg), but a minor component in seed (0.64% TRR, 0.003 mg/kg). The principal residue in all matrices was DCSA, mostly present as glucoside conjugate. The sum of free DCSA and its conjugates in foliage/hay ranged from 65 to 72% TRR (28-88 mg eq/kg) and represented 25% TRR (0.098 mg eq/kg) in the seed following POE treatment. Other compounds identified in soya bean matrices were conjugated forms of DCGA (1.9–6.7% TRR). In the seeds, a major part of the radioactivity was incorporated into natural products and no dicamba-related compounds occurred at levels exceeding 9.2% TRR or 0.036 mg eq/kg) in the post-extraction solids.

Dicamba-tolerant maize plants grown outdoors were spray treated with [phenyl-U-^{14}C]dicamba at 2.24 kg ai/ha either PRE or POE (30 DAP).

From PRE treatments, immature foliage was harvested 19 DAP (4.5 mg eq/kg), forage 80 DAP (0.075 mg eq/kg), stover 114 DAP (0.24 mg eq/kg), and grain 114 DAP (0.043 mg eq/kg). POE samples consisted of forage 50 DALA (2.2 mg eq/kg) and stover (7.8 mg eq/kg) and grain (0.062 mg eq/kg), each 84 DALA.

Extractability of POE residues into methanol/water was relatively high (>83% TRR) for residues in foliage and low (13% TRR) for residues in grain; hexane extracted an additional 5.3% TRR from grain.

In forage and stover dicamba was a minor residue in all POE matrices, comprising 8.6% TRR (0.19 mg/kg) and 6.3% TRR (0.49 mg/kg), respectively. The principal residue was DCSA, mostly present as glucoside conjugate. The sum of free DCSA and its conjugates represented 42% TRR in foliage (0.94 mg eq/kg) and 40% TRR (3.1 mg eq/kg) in stover. The sum of free and conjugated 5-OH dicamba accounted for <4.5% TRR (0.34 mg/kg). Residues identified as specific DCGA conjugates totalled 3.8% TRR (0.084 mg eq/kg) in forage, 2.5% TRR (0.19 mg eq/kg) in stover, and 0.39% TRR (0.0002 mg eq/kg) in grain; additional DCGA conjugates, totalling 5.6% TRR in forage, 6.8% TRR in stover, and 0.23% TRR in grain, were observed but could not be resolved from DCSA conjugates. Other residues occurred at minor individual levels of < 3.7% TRR (< 0.083 mg eq/kg) in forage and < 5.9% TRR (0.46 mg eq/kg) in stover.

In grain, no extracted residues occurred at >10% TRR or > 0.01 mg eq/kg. The most predominant radioactive residues in grain extracts were sugars (3.1% TRR, 0.0019 mg eq/kg) and natural organic acids (1.1% TRR, 0.0006 mg eq/kg); all other residues were ≤ 0.44% TRR.

Unextracted residues in the PES accounted for 14% TRR in forage, 16% TRR in stover, and 81% TRR in grain. In total, nearly 100% of these residues were comprised of starch, lignin, and phosphate compounds in the foliage and of cellulose, hemicellulose, and starch in the grain.

Following PRE or POE (76 DAP) spray applications of [phenyl-U-^{14}C]dicamba to outdoor grown dicamba-tolerant cotton at 2.24 kg ai/ha., TRR in seed were 0.16 mg eq/kg (PRE) and 0.98 mg eq/kg (POE). TRR in surrogate gin trash (consisting of leaves and stems) were 0.85 mg eq/kg (PRE) and 60 mg eq/kg (POE).

Extractability of POE residues into acetonitrile/water was high for gin trash (71%), but low for seed (38%); an additional 8.8% TRR was extracted from POE cotton seed using hexane.

Dicamba was a minor residue in both seed (0.85% TRR, 0.008 mg/kg) and gin trash (4.5% TRR, 2.7 mg/kg). DCSA glucoside was the predominant residue in both matrices (3.4% TRR, 0.033 mg eq/kg seed; 17% TRR, 10 mg eq/kg gin trash), with free DCSA making up an additional 1.9%

TRR (0.019 mg eq/kg) in seed and 13% TRR (8 mg eq/kg) in gin trash. Sugars accounted for 5.6% TRR in seed and 2.7% TRR in gin trash. All other residues, including DCGA (free and conjugated), were each < 5% TRR for both matrices. Of the 61% TRR in seed PES, approximately two-thirds of the radioactivity was associated with starch, protein, pectin, lignin, cellulose, and hemicellulose. In gin trash PES, approximately 98% of the radioactivity was associated with those natural plant constituents.

Methods of analysis

The analytical methods provided to the meeting were adequately validated for the analysis of dicamba, 5-hydroxydicamba (5-OH dicamba), DCSA, and DCGA in soya bean, maize and cotton matrices. The methods include a hydrolysis step (1 mol/L HCl, 95 °C, 1 or 1.5 hours) that is similar to that used in the metabolism studies (2 mol/L HCl, ca. 100 °C, 2 hours) and adequate to convert conjugated forms of DCSA and DCGA to their free equivalent, which are then determined as the free acid by LC-MS/MS. The LOQs are 0.01 mg/kg or lower in all tested matrices for all analytes except 5-OH dicamba in defatted soya flour (0.05 mg/kg), DCGA in refined soya oil (0.02 mg/kg), and all analytes in cotton hulls, meal, and refined oil (0.02 mg/kg), and gin trash (0.04 mg/kg).

Stability of residues in stored analytical samples

The Meeting received storage stability data for incurred dicamba, DCSA (incl. conjugates) and DCGA (incl. conjugates) in soya bean forage, hay, and seed, and cotton undelinted seed. Samples of soya bean matrices were stored frozen (-10 °C) for approximately 0, 2, 3, 6, 9, 12, 18, and 24 months. Residues of dicamba, DCSA (incl. conjugates) and DCGA (incl. conjugates) were shown to be stable for at least 24 months in all three soya bean matrices, except for DCGA (incl. conjugates) in forage, which was stable up to 3 months. Samples of cotton matrices were stored frozen (-10 °C) for approximately 0, 1, 2, 4, 6, and 9 months. Residues of dicamba, DCSA (incl. conjugates), and DCGA (incl. conjugates) were shown to be stable in cotton undelinted seed for at least 9 months.

Definition of the residue

The 2010 Meeting determined that the definition of the residue for enforcement of dicamba MRLs in conventional crops is dicamba only, noting that DCSA was found only at very low levels. For dietary risk assessment, the 2010 Meeting established a residue definition in plants of the sum of dicamba and 5-OH dicamba, expressed as dicamba.

In all three dicamba-tolerant crops evaluated by the current Meeting, dicamba was a major residue only in soya bean forage and hay and a minor residue in other matrices; in the seeds/grain, dicamba amounted to less than 1% TRR (≤ 0.008 mg/kg). 5-OH dicamba was observed only in maize matrices and only at low levels (free + conjugated < 3.4% in forage, <4.5% TRR in stover, and < 0.15% TRR in grain). The major residue in all matrices was DCSA glucoside, with lesser amounts of free DCSA and other sugar conjugates. In foliage, total DCSA (free + conjugated) accounted for 30 (gin trash) to 72% TRR (soya bean hay). In seeds/grain, total DCSA made up 25% TRR in soya bean, 0.79% TRR in maize grain, and 5.3% TRR in cotton. DCGA was observed only in a conjugated form. Although total DCGA occurred at low total levels (< 6.6% TRR in all matrices), it was a significant portion (13–21%) of the identified residues, especially in seeds and grain.

In supervised field trials on tolerant crops, parent dicamba was generally not present at levels above the LOQ in seeds/grain and only at low levels in forages and hays. Thus, dicamba is not a good marker residue for enforcement in the crops considered by the Meeting. Total DCSA and total DCGA were consistently found at levels >LOQ in field trials, and there was a tendency for total DCSA to be greater than total DCGA. Therefore, the Meeting decided to revise the current residue definition for enforcement of MRLs in soya bean, maize, and cotton commodities to be the sum of dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated), expressed as dicamba. The Meeting noted that this decision may need to be revisited if other dicamba-tolerant crops are considered by future Meetings.

In considering the residue definition for dietary risk assessment, the Meeting confirmed the conclusion from the 2010 Meeting that DCSA and DCGA were considered to have toxicity similar to

or lower than the parent compound. Total residues of DCSA account for the majority of the residues observed in the dicamba-tolerant crops evaluated by the current Meeting. Consequently, the Meeting decided to include free and conjugated DCSA for dietary risk assessment of soya bean, maize, and cotton commodities. Residue data from field trials indicate that exposure to total DCGA may be similar to that of total DCSA and cannot be excluded from consideration of dietary risk assessment of soya bean, maize, and cotton commodities. Therefore, the Meeting decided that the residue definition for risk assessment in soya bean, maize, and cotton commodities should be revised to the sum of dicamba, 2,5-dichloro-3-hydroxy-6-methoxybenzoic acid (5-OH dicamba), 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated) and 2,5-dichloro-3,6-dihydroxybenzoic acid (DCGA; free and conjugated), expressed as dicamba.

Thus, the Meeting agreed to replace the previous definitions for dicamba in plant commodities as follows:

Definition of the residue for compliance with the MRL for soya bean, maize, and cotton commodities: *sum of dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated), expressed as dicamba;* for other plant commodities: *dicamba*.

Definition of the residue for dietary risk assessment for soya bean, maize, and cotton commodities: *sum of dicamba, 2,5-dichloro-3-hydroxy-6-methoxybenzoic acid (5-OH dicamba), 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated) and 2,5-dichloro-3,6-dihydroxybenzoic acid (DCGA; free and conjugated), expressed as dicamba*; for other plant commodities: *sum of dicamba and 5-OH dicamba, expressed as dicamba*.

The residue is not fat-soluble.

The Meeting noted that because of the change to the residue definitions for soya bean, maize, and cotton commodities, all previous recommendations for these commodities needed to be withdrawn and replaced with new recommendations. The changes in definitions of the residue will not influence the numeric values of previous recommendations for conventional crops.

Since the animal dietary burdens are driven by residues arising from the use of dicamba on conventional crops, the definitions for dicamba in animal commodities do not need to be revised.

Results of supervised residue trials on crops

The Meeting received data from supervised residue trials conducted on dicamba-tolerant soya bean, maize, and cotton. All field trials were conducted in the USA, and the results are supported by adequate method and storage stability data, except for DCGA in soya bean forage, for which all samples were stored for approximately 4 to 9 months, which is longer than the demonstrated period of stability of 3 months.

For maximum residue estimation, residues of dicamba or DCSA (free + conjugated) that are <LOQ are assumed to be at the LOQ, and the combined residues are expressed as less than the combined LOQ only when both residues are <LOQ.

For dietary risk estimation, the Meeting noted that in the metabolism studies with dicamba-tolerant crops, dicamba made up 1.9% of the residue definition in soya bean seed and 0.6% in maize grain; furthermore, dicamba residues were reported as <LOQ in all field trial samples of these commodities. Therefore, the Meeting decided that the contribution of dicamba to dietary risk assessment of dicamba-tolerant soya bean seed and maize grain is negligible and could be assumed to be zero. Similarly, 5-OH dicamba was not observed in metabolism studies or field trials in dicamba-tolerant soya bean and cotton commodities and was assumed to be zero; 5-OH dicamba could not be excluded for maize commodities. For the remaining raw commodities considered by the current Meeting, the contribution of dicamba to dietary exposure could not be excluded. Therefore, residues reported as <LOQ in those commodities were assumed to be at the LOQ when deriving total residues for dietary risk assessment.

Estimation of residues for compliance with the MRL and for dietary risk assessment in commodities from dicamba-tolerant varieties for <LOQ residue results

	Residue, mg/kg (reported [assumed])				Combined estimate	
Commodity	Dicamba	5-OH Dicamba	Total DCSA	Total DCGA	Dicamba + DCSA	Dicamba + 5-OH dicamba + DCSA + DCGA
Soya bean seed	<0.005 [0]	<0.02 [0]	<0.005 [0.005]	<0.005 [0.005]	0.005	0.01
Soya bean forage and hay	<0.005 [0.005]	<0.005 [0]	<0.005 [0.005]	<0.005 [0.005]	0.01	0.015
Maize grain	<0.01 [0]	<0.01 [0.01]	<0.01 [0.01]	<0.01 [0.01]	0.01	0.03
Maize forage & stover	<0.01 [0.01]	<0.01 [0.01]	<0.01 [0.01]	<0.01 [0.01]	0.02	0.04
Cotton seed	<0.02 [0.02]	<0.01 [0]	<0.005 [0.005]	<0.005 [0.005]	0.025	0.03
Cotton gin trash	<0.04 [0.04]	<0.04 [0]	<0.04 [0.04]	<0.04 [0.04]	0.08	0.12

Soya bean

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; last application no later than BBCH 60).

Five field trials matching the critical GAP with respect to both application rate and retreatment interval are available. An additional 17 trials were provided that match the GAP for application rate but not for retreatment interval. The meeting noted that for soya bean seed, the retreatment interval used in the submitted studies (6 to 29 days) does not appear to have a significant impact on residues. Therefore, the Meeting decided to consider all trials approximating the critical GAP with respect to application rate. On that basis, there are 22 trials suitable for making residue estimates.

Residues of dicamba in soya bean seed from independent trials for estimation of maximum residues were (n=22): 0.016 (2), 0.018 (2), 0.019, 0.024, 0.026, 0.027, 0.028, 0.032, 0.036, 0.045, 0.048, 0.053, 0.054, 0.060, 0.063, 0.077, 0.082, 0.097, 0.12, and 0.44 mg/kg.

Residues of dicamba in soya bean seed from independent trials for estimation of dietary risk were (n=22): 0.021, 0.025, 0.028, 0.029, 0.030, 0.031, 0.032, 0.037, 0.046 (2), 0.051, 0.056, 0.058, 0.074, 0.087, 0.099, 0.11, 0.12, 0.14, 0.15, 0.17, and 0.56 mg/kg.

The previous recommendation for soya bean (dry) is 10 mg/kg and was derived from a pre-harvest desiccation GAP. As this value accommodates residues in dicamba-tolerant soya bean seeds, the Meeting withdrew the previous maximum residue level recommendation of 10 mg/kg and made a new recommendation of 10 mg/kg for soya bean seed (dry) according to the new residue definition. The Meeting estimated a STMR of 0.0535 mg/kg.

Maize

The critical GAP is from the registration in Canada (one pre-emergent application at 0.58 kg ai/ha and one post-emergent application at 0.6 kg ai/ha with a 30-day PHI). No trials available to the Meeting matched the Canadian GAP.

The Meeting withdrew the previous maximum residue level recommendation of 0.01(*) mg/kg and made a new recommendation of 0.01(*) mg/kg for maize according to the new residue definition. The Meeting confirmed the STMR of 0.02 mg/kg estimated by the 2010 JMPR.

Cotton

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; PHI of 7 days).

Two field trials matching the critical GAP with respect to application rate, retreatment interval, and PHI are available. An additional 11 trials are available that match the rate and PHI but not the retreatment interval. The meeting noted that for cotton seed, the retreatment interval used in the submitted studies (5 to 63 days) does not appear to have a significant impact on residues of dicamba for estimation of residues or for dietary risk assessment. Therefore, the Meeting decided to consider all trials approximating the critical GAP with respect to application rate and PHI. On that basis, there are 13 trials suitable for making residue estimates.

Residues of dicamba in cotton undelinted seed from independent trials for estimation of maximum residues were (n=13): 0.07, 0.13, 0.20, 0.34, 0.40, 0.43, 0.69, 0.98, 1.0, 1.1 (2), 1.2, and 1.6 mg/kg.

Residues of dicamba in cotton undelinted seed from independent trials for estimation of dietary risk were (n=13): 0.075, 0.15, 0.20. 0.38, 0.42, 0.43, 0.69, 1.0 (2), 1.1 (2), 1.3, and 1.6 mg/kg.

The Meeting withdrew the previous maximum residue level recommendation of 0.04 * mg/kg and made a new recommendation of 3 mg/kg for cotton seed according to the new residue definition. The Meeting estimated a STMR of 0.69 mg/kg.

Animal feedstuffs

Soya bean forage and hay

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; PHI = 7 days for forage and 14 days for hay).

The meeting noted that for soya bean forage, the samples were stored for 4 to 9.5 months, which is longer than the period of demonstrated stability (3 months). The Meeting decided that the soya bean forage data could not be used to estimate residues.

Four trials are available approximating the critical GAP for soya bean hay with respect to use pattern and harvest 14 DALA. Although specific residue decline data are not available for soya bean hay, the Meeting noted a tendency for higher residues from trials with harvest >14 DALA than those at 14 DALA. Therefore, the Meeting agreed to consider all trials that were harvested 14–24 DALA for estimating residues.

Residues of dicamba in soya bean hay (as received) from independent trials for estimation of maximum residues were (n=22): 13, 15, *21*, 21, 22, 24, 27, *30*, 30, 32 (3), 34 (2), 35, *36*, 36, *38*, 44, 47 (2), and 61 mg/kg (residues from at-GAP trials in italics).

Residues of dicamba in soya bean hay (as received) from independent trials for estimation of dietary burden were (n=22): 13, 16, 22, 23, *24*, 30, 31, *32*, 32, 34, 35 (2), 36, *37*, 37, 38, 40, *42*, 47, 48, 49, and 68 mg/kg.

The Meeting estimated a maximum residue level for soya bean fodder (dry) of 150 mg/kg (dw; based on a dry matter content of 85% from the OECD feed table), a median residue of 35 mg/kg in hay (as received), and a highest residue of 68 mg/kg (as received).

Maize forage and fodder

The critical GAP is from the registration in Canada (one pre-emergent application at 0.58 kg ai/ha and one post-emergent application at 0.6 kg ai/ha with a 30-day PHI). As noted above under maize grain, no trials matching the critical GAP were available to the Meeting.

Meeting withdrew the previous maximum residue level recommendation of 0.6 mg/kg and made a new recommendation of 0.6 mg/kg (dry weight) for maize fodder (dry) according to the new residue definition. The Meeting confirmed the median residue of 0.06 mg/kg (as received) and highest residue of 0.33 mg/kg (as received) for maize fodder as well as the median residue of 0.16 mg/kg (as received) and highest residue of 0.31 mg/kg (as received) for maize forage estimated by the 2010 JMPR.

Cotton gin trash

The critical GAP is from the registration in the USA (one pre-emergence application at 1.12 kg ai/ha and up to two post-emergence applications at least 7 days apart, each at 0.56 kg ai/ha; PHI of 7 days).

The meeting noted that for cotton gin trash, there appears to be a strong trend for lower residues at increased retreatment intervals used in the submitted studies (30 to 49 days. Therefore, the Meeting decided that the submitted trials for cotton gin trash were not suitable for estimating residues.

Fate of residues during processing

Residues after processing

The Meeting received data depicting the concentration/dilution of residues during processing of soya bean seed, maize grain, and undelinted cotton seed from dicamba-tolerant crops. For all crops, processed commodities were derived using simulated commercial practices. The resulting processing factors and STMR-P estimates for dicamba-tolerant varieties of soya bean and cotton are summarized below; residues were <LOQ in all maize RAC and processed commodity samples.

Raw agricultural commodity	Processed commodity	Processing factors [median/best estimate]		MRL, mg/kg	STMR-P, mg/kg
		Dicamba + DCSA	Dicamba + DCSA + DCGA [a)]		
Soya bean seed MRL = 10 mg/kg STMR = 0.054 mg/kg	Hulls	1.26, 1.52 [1.39]	1.13, 1.29 [1.21]	15	0.065
	Meal	1.17, 1.50 [1.34]	1.21, 1.43 [1.32]	15	0.071
	RBD oil [b)]	<0.31, <0.12	<0.17, <0.06 [0.06]	--	0.0032
	Soya milk	<0.31, <0.12	<0.17, <0.06 [0.06]	--	0.0032
	Tofu	<3.08, <1.16	<1.67, <0.63 [0.63]	--	0.0034
Cotton undelinted seed MRL = 3 mg/kg STMR = 0.69 mg/kg	Hulls	0.999, 0.331 [0.665]	1.01, 0.433 [0.723]	--	0.50
	Meal	0.365, <0.062 [0.365]	0.372, <0.087 [0.372]	--	0.26
	RBD oil [b]	<0.055, <0.062	<0.079, <0.087 [0.079]	--	0.055

a) Residues of 5-OH dicamba were assumed to be zero based on results from metabolism, field trials, and processing studies.

b) Refined, bleached and deodorized

Residues in animal commodities

Estimated maximum and mean dietary burdens of livestock

Dietary burden estimates from the 2010 Meeting have been recalculated to include contributions from commodities grown from dicamba-tolerant soya bean, maize, and cotton considered by the current Meeting. Estimated dietary burdens for Australia, the EU, Japan, and Canada/USA are summarized below. The livestock diets are listed in Annex 6.

Livestock Dietary Burdens (ppm of dry matter diet) for dicamba.

Livestock	Australia		EU		Japan		Canada/USA	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Cattle (beef)	140 [A)]	45 [C)]	77	23	15	5.5	5.7	2.6
Cattle (dairy)	140 [B)]	45 [D)]	85	27	29	8.7	84	30
Poultry (broiler)	1.0	1.0	1.3	1.3	0.86	0.86	1.4	1.4
Poultry (layer)	1.0	1.0	13 [E)]	6.3 [F)]	0.74	0.74	1.4	1.4

A) Highest maximum dietary burden for beef or dairy cattle; suitable for estimating the maximum residue levels for mammalian meat, fat, and offal.

B) Highest maximum dietary burden for dairy cattle; suitable for estimating the maximum residue levels for milk.

C) Highest mean dietary burden for beef or dairy cattle; suitable for estimating STMRs for mammalian meat, fat, and

offal.

D) Highest mean dietary burden for dairy cattle; suitable for estimating the STMR for milk.

E) Highest maximum dietary burden for broiler chickens or laying hens; suitable for estimating the maximum residue levels for poultry meat, fat, offal, and eggs.

F) Highest mean dietary burden for laying hens; suitable for estimating the STMRs for poultry meat, fat, offal, and eggs.

Animal commodity maximum residue levels

The Meeting noted that the dietary burdens for cattle and poultry remain essentially unchanged compared to those derived in 2010 (maximum and mean burdens were 140 and 44 ppm for cattle and 15.6 and 6.0 ppm for poultry; poultry burdens are lower in this assessment due to the removal of some commodities from the current OECD poultry diets); therefore, the Meeting confirmed its previous recommendations for residues in animal commodities.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL for soya bean, maize, and cotton: *sum of dicamba and 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated), expressed as dicamba;* for other plant commodities: *dicamba*.

Definition of the residue for dietary risk assessment for soya bean, maize, and cotton: *sum of dicamba, 2,5-dichloro-3-hydroxy-6-methoxybenzoic acid (5-OH dicamba), 3,6-dichloro-2-hydroxybenzoic acid (DCSA; free and conjugated) and 2,5-dichloro-3,6-dihydroxybenzoic acid (DCGA; free and conjugated), expressed as dicamba*; for other plant commodities: *sum of dicamba and 5-OH dicamba, expressed as dicamba*.

Definition of the residue for compliance with the MRL and for estimation of dietary exposure for animal commodities: *sum of dicamba and DCSA, expressed as dicamba.*

The residue is not fat-soluble.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	Highest Residue mg/kg
CCN	Name	CCN	Previous		
VD 0541	Soya bean (dry)	W [a)]	10		
VD 0541	Soya bean (dry)	10 [a)]	--	0.0535	--
GC 0645	Maize	W [a)]	0.01*		
GC 0645	Maize	0.01* [a)]	--	0.02 [b)]	--
SO 0691	Cotton seed	W [A)]	0.04*		
SO 0691	Cotton seed	3	--	0.69	--
AL 0541	Soya bean fodder (dry)	150 (dw)	--	35 (as)	68 (as)
AB 0541	Soya bean hulls	15	--	0.065	--
AB 1265	Soya bean meal	15	--	0.071	--
AS 0645	Maize fodder (dry)	W [a)]	0.6 (dw)		
AS 0645	Maize fodder (dry)	0.6 (dw) [a)]	--	0.06 (dw) [b)]	0.33 (dw) [b)]
	Soya bean oil	--		0.0032	
	Soya milk	--		0.0032	
	Tofu	--		0.0034	
	Cotton seed oil	--		0.055	
	Maize oil, crude			0.00058 [b)]	
	Cotton seed meal	--		Median: 0.26	
	Maize forage			Median: 0.40 (dw) [b)]	Highest: 0.775 (dw) [b)]

[a)] To withdraw the previous recommendation and replace it with a new one at the same level based on a new residue definition for compliance with the MRL.

[b)] Recommended by 2010 JMPR based on conventional maize

(as) – as received; (dw) – dry weight

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for dicamba is 0–0.3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for dicamba were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2019 Extra JMPR Report.

The IEDIs ranged from 0–1% of the maximum ADI. The Meeting concluded that long-term dietary exposure to residues of dicamba from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for dicamba is 0.5 mg/kg bw. The International Estimate of Short-Term Intakes (IESTIs) for dicamba 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 2019 Extra JMPR Report.

The IESTIs were 0% of the ARfD for both children and for the general population. The Meeting concluded that acute dietary exposure to residues of dicamba from uses considered by the present Meeting is unlikely to present a public health concern.

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