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HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

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OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Dicamba. Case 0065. PC Code 029801. Residue Data in Sorghum, Wheat, Asparagus, and Sorghum and Sugarcane Processed Fractions. MRID 43245203, 43274501, 43245206, 43425803, 43245204, & 43245205. CB 13882, 13948, 14695. DP Barcode: D204488, D204809, D209229.

FROM: Leung Cheng, Chemist Lee Cheng
Chemistry & Exposure Branch 1
Health Effects Division (7509C)

THROUGH: Francis B. Suhre, Senior Scientist Francis B Suhre
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Health Effects Division (7509C)

TO: Karen Whitby, Chief
Risk Characterization & Analysis Branch
Health Effects Division (7509C)

Attached please find a review of residue data in sorghum, asparagus, and sorghum and sugarcane processed fractions submitted by Sandoz Agro Inc in response to the Dicamba (SRR) Reregistration Standard (6/30/89). These data were reviewed by Dynamac Corporation under the supervision of HED. This information has undergone secondary review in CEB-1 and is consistent with Agency policies.

The submitted wheat residue data (MRID 43274501) have previously been reviewed in connection with PP#6F4604 (CBTS 16431 et al, F. Griffith, 5/2/96), and are not addressed in this memo.

The submitted sorghum residue study is inadequate. Geographical representation is not adequate as no tests were conducted in Regions 2, 4 and 7. Additional field trials are required: grain, forage and stover harvested 30 days after the second of 2 postemergence applications of dicamba each at 0.25 lb ae/A (from Regions 2, 4, and 7); and forage sampled at the late dough stage (Regions 2, 4, 5, 6, 7, and 8). As the available data indicate that the cation has no affect on the level of

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residues, the required studies need only be conducted using a single representative salt formulation. CB recommends that two independently composited samples be taken for each new field trial.

Portions of the asparagus residue data (MRID 43245206 and 43425803) have also been reviewed in connection with PP#6F4604. These data are deemed adequate and the conclusions made in CBTS 16431 et al are still valid. If the registrant wishes to include use in MI, then a tolerance of 3.5 ppm should be proposed for the combined residues of dicamba and DCSA (3,6-dichlorosalicylic acid) in/on asparagus.

The submitted sugarcane processing study is adequate pending submission of adequate supporting storage stability data for sugarcane and molasses. No concentration was observed in refined sugar but dicamba residues concentrate in molasses (24x). Based on the HAFT and the concentration factor, a tolerance of 1 ppm for molasses would be appropriate.

The submitted sorghum processing study is adequate. Residues of dicamba concentrate in aspirated grain fractions. However, the tolerance for aspirated grain fractions is set on the maximum residues found in the grain dust of corn, wheat, sorghum, or soybean. Before a tolerance for aspirated grain fractions can be proposed, data are required from field trials depicting the residues of corn, wheat, and soybean aspirated grain fractions generated from grain treated at the maximum label use rate for each crop and at the minimum PHIs (see OPPTS Test Guidelines, Series 860, Residue Chemistry, 860.1500).

Attachment - Dynamac review of Registrant's Response to Residue Chemistry Data Requirements (CB 13882, 13948, 14695)

cc(with Attachment):Circ, RF, Reg Std File, Cheng
RDI:ResChmTeam:7/8/97:FBSuhre:7/14/97
7509C:CEB-1:LCheng:CM#2:RM804E:7/2/97:[redacted]05:DICAMBA\RACS.2

DICAMBA

Shaughnessy No. 029801. Case No. 0065

(CBRS No. 13882; DP Barcode D204488)
(CBRS No. 13948; DP Barcode D204809)
(CBRS No. 14695; DP Barcode D209229)

REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS

BACKGROUND

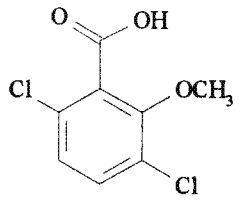
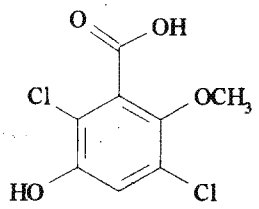
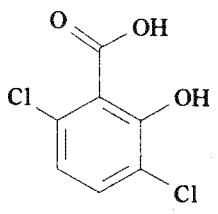
The Dicamba Second Round Review (SRR) dated 6/89 required additional field trial data depicting residues of dicamba and its 5-hydroxy metabolite in/on sorghum, wheat forage and hay, and asparagus. In addition, processing studies were required for sugarcane and sorghum. In response, Sandoz Agro, Inc. has submitted sorghum (1994; MRID 43245203), wheat (1994; MRID 43274501), and asparagus (1994; MRIDs 43245206 and 43425803) field trials and sugarcane (1994; MRID 43245204) and sorghum (1994; MRID 43245205) processing studies. The wheat forage and hay residue data were reviewed in 1996 in connnection with PP#6F4604 (CBTS 16431 et al, F. Griffith, 5/2/96) and are not addressed here. Portions of the asparagus residue data (MRID 43245206 and 43425803) were also reviewed in connection with PP#6F4604 (but the conclusions made in CBTS 16431 are still valid). The pertinent data are reviewed here for their adequacy in fulfilling residue chemistry data requirements. The Conclusions and Recommendations stated herein pertain only to data requirements for magnitude of the residue in plants and plant processed commodities.

The qualitative nature of the residue in plants is adequately understood. The residues of concern in/on plant commodities (except asparagus, soybeans, and soybean forage and hay) are dicamba and its 5-hydroxy metabolite (3,6-dichloro-5-hydroxy-*o*-anisic acid). The residues of concern in/on asparagus, soybeans, and soybean forage and hay are dicamba and its metabolite 3,6-dichloro-2-hydroxybenzoic acid (3,6-dichlorosalicylic acid; DCSA).

Tolerances for residues in/on plants (excluding soybeans, soybean forage and soybean hay) and processed food/feed commodities are currently expressed in terms of the combined residues of dicamba (3,6-dichloro-*o*-anisic acid) and its 5-hydroxy metabolite [40 CFR §180.227(a), §185.1800, and §186.1800]. Tolerances for residues in/on soybeans, soybean forage and soybean hay, and in animal commodities are expressed in terms of the combined residues of dicamba and its DCSA metabolite [40 CFR §180.227(b)]. The chemical structures of dicamba and its metabolites are shown in Figure 1. There are no Codex MRLs for residues of dicamba; therefore, issues of compatibility between U.S. tolerances and Codex MRLs do not exist.

The Pesticide Analytical Manual (PAM) Vol. II lists Method I and II, GC methods with electron capture detection (GC/ECD), for the enforcement of tolerances on dicamba and its metabolite 5-hydroxy dicamba in/on plant commodities and milk.

Figure 1. Chemical names and structures of dicamba and its metabolites.

		
Dicamba (3,6-dichloro-*o*-anisic acid)	**5-hydroxy dicamba** (3,6-dichloro-5-hydroxy-*o*-anisic acid)	**DCSA** (3,6-dichloro-2-hydroxybenzoic acid or 3,6-dichlorosalicylic acid)

CONCLUSIONS AND RECOMMENDATIONS

1a. Sorghum. The submitted sorghum data are inadequate based upon the registrant's intent to support the use of two postemergence applications of dicamba, each at 0.25 lb ae (acid equivalent)/A (0.5 lb ae/A/season) with the second application being made at the soft dough stage. Geographic representation was not adequate as no studies were conducted in Regions 2, 4 and 7 (see OPPTS Test Guidelines, Series 860, Residue Chemistry, 860.1500). Data are required for Regions 2, 4 and 7 depicting the combined residues of dicamba and its 5-hydroxy metabolites in/on sorghum grain and fodder harvested 30 days after the second of two postemergence applications of dicamba each at 0.25 lb. In addition, the available sorghum forage data reflect only the single early season application of dicamba which is 0.5x the proposed application rate. For sorghum forage, residue data are required for sorghum forage following the second of two postemergence applications of dicamba each at 0.25 lb. Tests on sorghum forage are required in the following geographic regions: 2, 4, 5, 6, 7, and 8. As the available data indicate that the counter ion of the salt had no affect on the level of residues, the required studies need only be conducted using a single representative salt formulation. CEB-1 recommends that for each new field trial required, two independently composited samples be taken.

1b. The maximum combined residues of dicamba and its 5-hydroxy metabolite were 2.73 ppm in/on 20 grain samples (30-42 day post-treatment interval; PTI) and 8.2 ppm in/on 20 fodder samples (30-42 day PTI) following two applications of dicamba at the intended 1x rate (0.5 lb ae/A/season). These data indicate that the established 3 ppm for grain and fodder may be appropriate for grain but is too low for fodder (stover). However,

additional data are required before a tolerance assessment can be made for grain, forage, and stover.

1c. In addition, the registrant must (i) indicate consistently on all end-use product labels registered for use on sorghum the maximum use rate, and minimum PHI, and (ii) specify on all labels that aerial applications must be made in >2 GPA or provide the appropriate field trial data for low volume aerial application.

2a. Asparagus. The submitted asparagus field trials are adequate. The maximum combined residues of dicamba and 3,6-dichloro-5-hydroxybenzoic acid (DCSA) was 1.10 ppm in/on 36 asparagus samples grown in CA and WA harvested 24 hours following a single application at 0.5 lb ae/A of the dicamba Na^+ salt (2.0 lb/gal SC/L), dicamba DGA^+ salt (4.0 lb/gal SC/L), or the dicamba DMA^+ salt (4.0 lb/gal SC/L) formulation. The maximum combined residues of dicamba and DCSA was 3.29 ppm in/on 12 asparagus samples grown in MI harvested 24 hours following a single application at 0.5 lb ae/A of the dicamba Na^+, DGA^+, or DMA^+ salt formulation. The 4 lb/gal SC/L DMA^+ salt and the 4 lb/gal SC/L K^+ salt formulations are the only dicamba end-use products currently registered for use on asparagus and the use is restricted to CA, OR, and WA. The field trial data indicate that the residue levels are not dependent on the salt formulation used. No additional data are required.

2b. Since the registrant states in the cover letter he wishes a national use, a tolerance of 3.5 ppm for the combined residues of dicamba and 3,6-dichloro-5-hydroxybenzoic acid in/on asparagus should be proposed (see CBTS 16431 et al, F. Griffith, 5/2/96).

3. Sugarcane. The submitted sugarcane processing study is adequate. Combined residues of dicamba and 5-hydroxy dicamba were 0.054 ppm in sugarcane and 1.32 ppm in molasses indicating a concentration factor of 24x. Pending submission of adequate supporting storage stability data for sugarcane and molasses (5e), these data indicate that the current food/feed additive tolerances of 2.0 ppm for the combined residues of dicamba and its 5-hydroxy metabolite in molasses is too high. Based on the concentration factor and the HAFT, a tolerance of 1 ppm for molasses would be appropriate. The Agency does not currently require residue data on bagasse, but may at a later date. For future reference (pending submission of adequate supporting storage stability data for bagasse), combined residues of dicamba and its 5-hydroxy metabolite appear to concentrate 6.6x in bagasse. No concentration was observed in refined sugar.

4. Aspirated Grain Fractions. The submitted sorghum processing study is adequate. The data indicate that the maximum combined residues of dicamba and 5-hydroxy dicamba are 27.1 ppm in/on aspirated grain fractions generated from sorghum grain treated with a total of 1.5 lb ae/A. A tolerance will be required for aspirated grain fractions as a RAC under 40 CFR §180.227(a). However, the tolerance for aspirated grain fractions is set on the maximum residues found in the grain dust of corn, wheat, sorghum, or soybeans.

Before a tolerance for aspirated grain fractions can be proposed, data are required from field trials depicting the residues of corn, wheat, and soybean aspirated grain fractions generated from grain treated at the maximum label use rate for each crop and at the minimum PHIs (see OPPTS Test Guidelines, Series 860, Residue Chemistry, 860.1500).

5a. Storage Stability. In conjunction with the wheat field trial, the registrant conducted a storage stability study using wheat forage and hay. To demonstrate storage stability of dicamba and 5-hydroxy dicamba in frozen wheat forage and hay, the registrant reanalyzed 11 and 12 selected samples each of hay and forage from the field study after an additional storage interval and compared the results from the two analyses. The initial analyses occurred after 72-357 and 73-105 days of frozen storage for forage and hay, respectively. The reanalyses occurred after an additional 248-283 days of frozen storage. Frozen samples were held at <-17 C. Recoveries after storage averaged 111% for dicamba and 126% for the 5-OH dicamba. The data indicate that residues of dicamba and its 5-hydroxy metabolite are stable in/on frozen wheat forage and hay for up to 258 and 283 days, respectively. Wheat forage and hay samples from the current field study were stored at <-17 C for 4-357 days prior to analysis; however, only 10 of the ~350 samples had a storage interval of >200 days. The available storage stability data adequately support the submitted field trial data.

5b. The sorghum forage, fodder, silage, and grain samples from the current submission were held in frozen (<-17 C) storage for up to 160 days (~5 months) prior to analysis. Data depicting the frozen storage stability of dicamba and 5-hydroxy dicamba in/on sorghum forage and fodder are required to support the current submission. The registrant stated that a 3-year storage stability study for residues of dicamba and its 5-hydroxy metabolite in/on corn forage, silage, grain, and fodder is in progress. For storage stability purposes, the corn data may be used. Adequate storage stability data for grain were submitted with the sorghum processing study.

5c. Adequate storage stability data for sorghum grain were submitted with the sorghum processing study. These data indicate that residues of dicamba and 5-hydroxy dicamba are stable in/on frozen sorghum grain for up to 5 months and are adequate to support storage interval and conditions of the grain and grain dust samples in the current processing study submission (up to 5 months, <-17C) and in the grain samples in the current sorghum field trial (up to 5.3 months, <-17 C). No additional storage stability data are required for sorghum grain or aspirated grain fractions..

5d. The submitted storage stability data for asparagus indicate that residues of dicamba and DCSA are stable in/on asparagus for up to 104 days of frozen (<-12 C) storage and that residues of 5-hydroxy dicamba are stable for up to 119 days of frozen (<-12 C) storage. Samples in the current asparagus studies (1994; MRIDs 43245206 and 43245803) were stored at <-12 C for up to 95 days prior to analysis. No additional storage stability data are required for asparagus.

5e. The submitted storage stability data for sugarcane indicate that residues of dicamba and 5-hydroxy dicamba are stable in white refined sugar stored up to 60 days at <-1 C. However, the data are inadequate to determine the storage stability of dicamba and its metabolite in/on sugarcane, bagasse, and molasses. In the current submission, the RAC sample was stored frozen for up to 58 days prior to analysis. The processed commodities were stored frozen (<-1 C) for up to 64 days prior to analysis. The registrant may use storage stability data on corn forage from the 3-year corn study to support the sugarcane RAC storage interval. Bagasse is not considered a RAC; therefore, supporting storage stability information on bagasse is not required. Storage stability data are required to support the interval and conditions (64 days, <-1 C) of storage of molasses.

DETAILED CONSIDERATIONS

Residue Analytical Methods

In conjunction with the sorghum field trial and processing study, Sandoz (1994; MRIDs 43245203 and 43245205) submitted a method description for the analysis of dicamba and its 5-hydroxy metabolite in/on sorghum forage, silage, grain, fodder, and aspirated grain fractions. The method, AM-0691B, has been previously reviewed and deemed adequate for data collection and tolerance enforcement for residues in/on plant commodities (CBRS No. 12482, DP Barcode D194776, D. Miller, 12/14/93). For the field trial (MRID 43245203), analyses were performed by MVTL Laboratories, Inc., New Ulm, MN. For the processing study (MRID 43245205), analyses were performed by Sandoz Agro, Inc., Des Plaines, IL. Chromatograms and sample calculations were provided.

Briefly, residues of dicamba and 5-hydroxy dicamba are hydrolyzed with 1N HCl for 1.5 hours in a 95 C water bath. The pH is adjusted to ≥8 and readjusted to <1. Residues are extracted with ethyl ether, concentrated, methylated with diazomethane, and cleaned up by silica gel column chromatography prior to analysis by GC/ECD. The limit of detection is 0.01 ppm for each analyte.

For the sorghum field trial, concurrent recovery data were submitted for four to five control samples of each matrix fortified at 0.01 and 0.10 ppm of each dicamba and 5-hydroxy dicamba. Residues of dicamba and its 5-hydroxy metabolite were nondetectable (<0.01 ppm for each analyte) in/on five control samples each of silage, grain, and fodder and four forage control samples. Concurrent recoveries were 68-99% and are detailed in Table 1.

For the sorghum processing study, method recovery data were submitted for four control grain samples fortified at 0.01-1.5 ppm of each dicamba and its 5-hydroxy metabolite. Residues of dicamba and 5-hydroxy dicamba were nondetectable (<0.01 ppm for each analyte) in/on two grain samples. Method recoveries were 80-110% and are detailed in Table 1. In addition, concurrent recovery data were submitted for two samples each of grain and >2030 µm, >1180

µm, >850 µm, >425 µm, <425 µm aspirated grain fractions fortified with dicamba at 5 and 15 ppm with 5-hydroxy dicamba and at 5 and 10 ppm. Concurrent recoveries were 63-127% and are detailed in Table 1.

In conjunction with the sugarcane processing study, Sandoz (1994; MRID 43245204) has submitted a method description for the analysis of dicamba and its 5-hydroxy metabolite in/on sugarcane and its processed fractions. The method, AM-0691B, is identical to the one described above for sorghum. Analyses were performed by Sandoz Agro, Inc., Des Plaines, IL. Chromatograms and sample calculations were provided.

Concurrent recovery data were submitted for four to five control samples of sugarcane, bagasse, molasses, and refined sugar fortified at 0.01-1.0 ppm each with dicamba and its 5-hydroxy metabolite. Residues of dicamba and 5-hydroxy dicamba were nondetectable (<0.01 ppm for each analyte) in/on 1 control of each matrix. Concurrent recoveries were 50-143% and are presented in Table 1.

Table 1. Concurrent and method verification[a] recoveries of dicamba and 5-hydroxy dicamba from fortified control samples.

Crop	Commodity	MRID	Fortification Level (ppm)	Number of Samples	% Recovery [b]	
					Dicamba	5-OH Dicamba
Wheat	forage	43274501	0.01-50 [c]	34	70-110	70-110
	hay	43274501	0.01-10	18	70-96	70-100
Sorghum	forage	43245203	0.01-0.1	4	68-90 (1)	71-97
	silage	43245203	0.01-0.1	5	70-98	70-95
	grain	43245203	0.01-0.1	5	74-94	74-98
	fodder	43245203	0.01-0.1	5	81-99	75-88
	grain[a]	43245205	0.01-1.5	4	86-110	80-89
	grain	43245205	5.0-15.0 [d]	2	75, 95	75, 82
Grain dust	>2030 µm	43245205	5.0-15.0 [d]	2	107, 107	91, 91
	>1180 µm	43245205	5.0-15.0 [d]	2	127, 83	114, 63
	>850 µm	43245205	5.0-15.0 [d]	2	111, 107	96, 96
	>425 µm	43245205	5.0-15.0 [d]	2	112, 111	100, 99
	<425 µm	43245205	5.0-15.0 [d]	2	93, 94	77, 83
Sugarcane	RAC	43245204	0.01, 0.10	4	50-106 (2)	80-107
	bagasse	43245204	0.01-0.2	5	58-100 (1)	63-143 (2)
	molasses	43245204	0.01-1.0	5	76-97	81-102
	refined sugar	43245204	0.01, 0.1	4	62-97	85-102

[a] Method verification recoveries presented only for the sorghum grain samples from MRID 43245205. For all other matrices, only concurrent recovery data are presented.
[b] The number of samples with recoveries outside of the acceptable 70-120% range is listed parenthetically.
[c] Control wheat forage samples were fortified at 0.01-50 ppm of dicamba and 0.01-10 ppm of 5-OH dicamba.
[d] These control samples were fortified with dicamba at 5.0 and 10.0 ppm and with 5-hydroxy dicamba at 5.0 and 15.0 ppm.

In conjunction with the asparagus field trials, Sandoz (1994; MRIDs 43245206 and 43245803) submitted method descriptions for the analysis of dicamba, 5-hydroxy dicamba, and DCSA. Residues were extracted following method AM-0691B as described above for sorghum. Derivatization, clean-up, and analysis of dicamba and DCSA were performed by method AM-0766A. For method AM-0766A, extracted residues are butylated with diazobutane or methylated with diazomethane and cleaned up by silica gel column chromatography prior to analysis by GC/ECD. Confirmation analyses for DCSA and dicamba were performed by GC/MS. The limit of detection is 0.01 ppm each for dicamba and DCSA. Chromatograms and sample calculations were provided. Analyses were performed by Sandoz Agro, Inc., Des Plaines, IL.

Concurrent recovery data were submitted for control samples fortified at 0.01-0.50 ppm of each dicamba, DCSA, and 5-hydroxy dicamba. One to five control samples were fortified at each fortification level. Residues of dicamba, DSCA, and 5-hydroxy dicamba were nondetectable (<0.01 ppm) in/on 13-14 controls. Residues of dicamba were 0.015 and 0.017 ppm in/on two controls and residues of DCSA were 0.011-0.013 ppm in/on three controls. Concurrent recoveries, corrected for residues of DCSA and dicamba in/on controls, were 40-130% and are detailed in Table 2. These data indicate that Method AM-0766A is adequate for data collection on residues of dicamba and DCSA in/on asparagus.

Table 2. Concurrent recoveries of dicamba, 5-hydroxy dicamba, and DCSA from fortified control asparagus samples.

Analyte	MRID	Fortification Level (ppm)	% Recovery [a]
Dicamba	43245206	0.01	40, 70
		0.05	80-88
		0.1	79
		0.5	79, 96
	43425803	0.01	100, 123
		0.02	101
		0.10	103
		0.20	94, 87
		0.50	106, 87
		1.0	82, 97
		3.0	107, 112
DCSA	43245206	0.01	60, 90
		0.05	74-84
		0.1	80
		0.5	81, 102
	43425803	0.01	40-77 [b]
		0.02	73-90
		0.05	84, 96
		0.10	95, 87
5-hydroxy dicamba	43245206	0.01	76
		0.10	84
	43245803	0.01	70-103
		0.02	89-110
		0.05	81, 88
		0.10	107, 130

[a] Recoveries were corrected for apparent residues in the controls.
[b] Three of the four fortified controls had recoveries outside of the acceptable 70-120% range.

Storage Stability Data

In conjunction with the wheat field trial (1994; MRID 43274501), the registrant conducted a storage stability study using wheat forage and hay. To demonstrate storage stability of dicamba and 5-hydroxy dicamba in frozen wheat forage and hay, the registrant reanalyzed 11 and 12 selected samples each of hay and forage from the field study after an additional storage interval and compared the results from the two analyses. The initial analyses occurred after 72-357 and 73-105 days of frozen storage for forage and hay, respectively. The reanalyses occurred after an additional 248-283 days of frozen storage. Frozen samples were held at <-17 C. Results of both analyses are presented in Table 3. The data indicate that residues of dicamba and its 5-hydroxy metabolite are stable in/on frozen wheat forage and hay for up to 258 and 283 days, respectively.

Wheat forage and hay samples from the current field study were stored at <-17 C for 4-357 days prior to analysis; however, only 10 of the ~350 samples had a storage interval of >200 days. The available storage stability data adequately support the submitted field trial data.

Table 3. Storage stability of dicamba and 5-hydroxy dicamba in frozen wheat forage and hay samples bearing measurable weathered residues.

Commodity	Storage interval (days)	# of samples	Dicamba (ppm)			5-OH Dicamba (ppm)		
			Initial analysis	Reanalysis	Percent recovery	Initial analysis	Reanalysis	Percent recovery
Wheat forage	248-258	12	1.3-15.0	0.88-18.0	46-169 (111) [a]	0.8-5.9	0.76-6.2	69-200 (126)
Wheat hay	278-283	11	1.4-8.6	1.4-11.0	70-133 (106)	1.8-11.0	2.1-14.0	68-127 (104)

[a] Values in parentheses are the average storage stability recoveries calculated by the registrant.

In the sorghum field trial study (1994; MRID 43245203), the sorghum forage, fodder, silage, and grain samples were held in frozen (<-17 C) storage for up to 5.3 months prior to analysis. Data depicting the frozen storage stability of dicamba and 5-hydroxy dicamba in/on sorghum forage and fodder are required to support the current submission. The registrant stated that a 3-year storage stability study for residues of dicamba and its 5-hydroxy metabolite in/on corn forage, silage, grain, and fodder is in progress. For storage stability purposes, the corn data may be used for sorghum. Adequate storage stability data for sorghum grain were submitted with the processing study and are discussed below.

In conjunction with the sorghum processing study (1994; MRID 43245205), the registrant submitted data depicting the frozen storage stability of dicamba and its 5-hydroxy metabolite in/on sorghum grain. A grain sample was analyzed within 10 days (day-0) of harvest and again after 5 months of frozen storage. The combined residues of dicamba and 5-hydroxy dicamba were 8.3 ppm in the day-0 sample and 8.8 ppm in the stored sample. These data indicate that residues of dicamba and 5-hydroxy dicamba are stable in/on frozen sorghum grain for up to 5 months and are adequate to support the storage interval and conditions of the grain and grain dust samples in the current submission (up to 5 months, <-17C). No additional storage stability data are required.

In conjunction with the asparagus field trial (1994; MRID 43245206), the registrant submitted supporting storage stability data. Two to three control asparagus samples were fortified at 0.2-0.4 ppm of each dicamba, DCSA, and 5-hydroxy dicamba and analyzed at day-0 and after 104 days of frozen (<-12 C) storage. Freshly fortified samples were also analyzed concurrently at each storage interval. Due to low concurrent recoveries of 5-hydroxy dicamba at the 104-day interval, three fortified control samples were analyzed for 5-hydroxy dicamba after 119 days of

frozen storage. Concurrent recoveries and recoveries after storage are presented in Table 4. These data indicate that residues of dicamba and DCSA are stable in/on asparagus for up to 104 days of frozen (<-12 C) storage and that residues of 5-hydroxy dicamba are stable for up to 119 days of frozen (<-12 C) storage. Samples in the current submissions (1994; MRIDs 43245206 and 43245803) were stored frozen at <-12 C for up to 95 days prior to analysis. No additional storage stability data are required.

Table 4. Recoveries after frozen storage and concurrent recoveries of fortified control asparagus samples

Storage Interval	% Recovery after storage			% Concurrent recovery		
	Dicamba	DCSA	5-OH dicamba	Dicamba	DCSA	5-OH dicamba
0	79.8-91.6	83.3-90.3	65.0-79.9	88.7, 90.2	91.4, 92.6	74.1, 74.2
104	66.7, 82.6	70.3, 90.6	76.2, 85.6	66.0, 74.3	65.4, 75.6	54.1, 63.9
119	NA[a]	NA	80.0-88.5	NA	NA	82.5, 91.0

[a] NA=not analyzed.

In conjunction with the sugarcane processing study, Sandoz (1994; MRID 43245204) submitted storage stability data for residues of dicamba and its 5-hydroxy metabolite in bagasse, molasses, and refined sugar processed from sugarcane. The registrant also submitted storage stability data for the sugarcane RAC; however, the RAC data did not reflect the frozen storage stability of dicamba and 5-hydroxy dicamba in sugarcane. Initial analysis of field samples from a 87-day PTI were compared with stored samples from a 130-day PTI.

For bagasse and final molasses three treated samples were analyzed after 64 days of frozen storage and again after 120 days of frozen storage. For bagasse, the combined residues of dicamba and 5-hydroxy dicamba were 0.356-0.361 and 0.342-0.390 ppm in the 64- and 120-day samples, respectively. For the molasses, combined residues were 1.234-1.359 and 1.030-1.213 ppm in the 64- and 120-day samples, respectively.

For refined sugar, three control samples were fortified at 0.1 ppm of each dicamba and 5-hydroxy dicamba and were analyzed on day-0 and after 60 days of frozen storage. Dicamba recoveries were 95-110% on day-0 and were 94-99% after storage. Recoveries of 5-hydroxy dicamba were 97-121% on day-0 and 115-122% after storage. Stored samples of the RAC and processed commodities were held at <-1 C.

In the current submission, the RAC sample was stored frozen for up to 58 days prior to analysis, and the processed commodities were stored frozen (<-1 C) for up to 64 days prior to analysis. The submitted storage stability data indicate that residues of dicamba and 5-hydroxy dicamba are stable in white refined sugar stored up to 60 days at <-1 C. However, the data are inadequate to determine the storage stability of dicamba and its metabolite in/on sugarcane, bagasse, and

molasses. As mentioned above, the registrant stated that a 3-year storage stability study for residues of dicamba and its 5-hydroxy metabolite in/on corn forage, silage, grain, and fodder is in progress. The registrant may use storage stability data for corn forage to support the sugarcane RAC storage interval. Bagasse is not considered a RAC, therefore, supporting storage stability information for bagasse is not required. Storage stability data are required to support the interval and conditions (64 days, <-1 C) of storage of molasses indicated in the current submission.

Magnitude of the Residue in Plants

Sorghum. The Dicamba SRR dated 6/89 required additional data depicting residues of dicamba and its 5-hydroxy metabolite in or on sorghum grain following a postemergence and a preharvest application of dicamba. In response, the registrant submitted sorghum data, including supporting storage stability information, that was deemed inadequate as it was generated by Craven Laboratories (CBTS No. 8617, DP Barcode D169036, R. Lascola, 1/17/92; CBRS No. none, S. Funk, 10/22/93). In response to a 2/94 DCI for replacement of the Craven data, the registrant submitted a request for a waiver of the data requirements (CBRS No. 13923, DP Barcode 204754, S. Funk, 7/12/94). The Agency denied the waiver request and concluded that data were required from 12 field trials depicting residues following the application of three dicamba formulations (DGA^+ salt SC/L, K salt SC/L, DMA^+ salt SC/L) to sorghum grown in Regions 2 (1), Region 4 (1), Region 5 (4), Region 6 (2), Region 7 (1), and Region 8 (3). For nine of the field trials, a single postemergence application at 0.25 lb ae/A to plants between the three-leaf stage and 15" height were required. For three of the field trials, a preharvest use of dicamba (0.25 lb ae/A) on sorghum grain (soft dough stage) in OK or TX; IL or IN or MI; KS or MO or NE were required for regional registration. Residues of dicamba and its 5-hydroxy metabolite were to be determined on the mature crop (grain, forage, silage, and fodder; S. Funk, 10/22/93). Residue data for silage (see below for revised definition) is no longer required (see Table I, OPPTS Test Guidelines, Series 860, Residue Chemistry, 860.1000).

A tolerance of 3.0 ppm has been established for the combined residues of dicamba and its metabolite 5-hydroxy metabolite in/on each sorghum fodder, forage, and grain. [40 CFR § 180.227 (a)].

A REF's search dated 11/16/94 identified five dicamba end-use products registered to Sandoz for use on sorghum: two DMA^+ salt formulations, a 4.0 lb/gal SC/L and a 1.0 lb/gal SC/L (EPA Reg. Nos. 55947-1 and -24); three K^+ salt formulations: 4 lb/gal SC/L (EPA Reg. No. 55947-38), a 1.1 lb/gal FlC (EPA Reg. No. 55947-157), and a 1.1 lb/gal DF (EPA Reg. No. 55947-39). The one DGA^+ salt formulation is currently registered for use on corn only.

The 4.0 lb/gal SC/L DMA^+ salt and 4 lb/gal SC/L K^+ salt formulations are registered for a broadcast or banded application to sorghum at 0.25 lb ae/A. Only one application per season is permitted and no PHI is indicated. The application must be made before the plants are 15 inches tall. Both labels indicate that a preharvest application to sorghum after the soft dough stage is

permitted in TX and OK at 0.25 lb ae/A with a 30-day PHI. There is a restriction to grazing or feeding treated sorghum forage or silage prior to the mature grain stage on both labels.

The 1.1 lb/gal FlC and 1.1 lb/gal DF K^+ salt formulations are registered for one postemergence band or broadcast application at 0.275 lb ae/A. Only one application per season is permitted. The applications must be made before the plants are 8 inches tall. Grazing or harvesting for livestock feed prior to crop maturity is prohibited. A 30-day PHI is in effect.

The two DMA^+ salt formulations (4.0 and 1.0 lb/gal SC/Ls) and the 4 lb/gal SC/L K^+ salt formulation describe a preplant use pattern on sorghum grown for pasture and hay. The labels are inconsistent regarding maximum application rates and PHIs. The maximum use rate and the minimum PHI indicated on any one label is 0.5 lb ae/A with a 7-day grazing restriction for lactating animals and a 37-day PHI for hay. Maximum seasonal use rates vary from 2-8 lb ae/A and PHIs vary from 7-90 days.

For all labels with a registered use on sorghum, the applications must be made in 3-50 GPA for ground equipment and 1-20 GPA for aerial equipment. The 4.0 lb/gal SC/L DMA^+ salt label specifies that aerial applications are to be made in 2-20 GPA for preharvest use, while all other uses can be applied in 1-10 GPA. All other labels indicate that aerial applications are to be made in >2 GPA.

In a letter (1994; MRID 43274500) accompanying the current submission, the registrant has indicated that the use pattern they are supporting allows for two postemergence applications, one at 0.25 lb ae/A when plants were 15" tall and a second at 0.25 lb ae/A again at the soft dough stage (30-day PHI).

Sandoz submitted data (1994; MRID 43245203) from 40 tests conducted in KS (8), MO (8), NE (8), OK (8), and TX (8) depicting residues of dicamba and its 5-hydroxy metabolite in/on sorghum forage, silage, grain, and fodder. Forage was defined as the whole, green, above ground portion of the plants collected immediately prior to seedhead emergence. Silage was defined as the whole, above ground portion of the plants collected at the soft dough stage (approximates the definition of sorghum forage in Table I). Fodder was defined as the dry, above ground portion of plants collected after the grain has been harvested (defined as sorghum stover in Table I). At each test site, the following four dicamba formulations were used: a 70% DF Na^+ salt, a 4 lb/gal SC/L DGA^+ salt, a 4 lb/gal SC/L K^+ salt, and a 4 lb/gal SC/L DMA^+ salt. At each test site, one or two applications of each of the four dicamba formulations were made at 0.25 lb ae/A/application for total seasonal applications of 0.25 lb ae/A (0.5x) and 0.5 lb ae/A (1x). Applications were made in 19.6-21.5 GPA using ground equipment. The 0.5x plots were treated when the plants were 15" tall using a broadcast application sprayed directly under the canopy (20-109 PTI). The 1x plots received an additional application at 0.25 lb ae/A at the soft dough stage (30-42 PTI).

For the 0.5x tests in MO, NE, OK, and TX, one treated sample of each sorghum forage, silage, grain, and fodder were collected. For the 0.5x tests in KS, one treated sample of each sorghum silage, grain, and fodder were collected. For the 1x tests, one treated sample of sorghum grain and fodder were collected. One control sample of each matrix was collected from each test site and served as the control for both the 0.5x and 1x tests. Samples were stored frozen at <-17 C for 28-160 days prior to analysis. Adequate storage stability data were provided with the sorghum processing study for sorghum grain. Storage stability data for dicamba and 5-OH dicamba in corn commodities can be translated to sorghum forage and fodder.

Geographic representation is inadequate. The submitted field trial data were from tests conducted in Leonard, MO (Region 5), Lincoln, NE (Region 5), Eakly, OK (Region 6), College Station, TX (Region 6), and Dodge City, KS (Region 8). Data are required from tests conducted in Regions 2, 7, and 4.

Following a single postemergence application of a DMA^+, DGA^+, or K^+ salt SC/L formulation or a Na^+ salt DF formulation to 15" tall grain at 0.25 lb ae/A, the combined residues of dicamba and its 5-hydroxy metabolite were 0.029-0.46 ppm in/on 16 forage samples (20-54 day PTI), 0.047-0.27 ppm in/on 20 silage samples (30-61 day PTI), <0.02-0.184 ppm in/on 18 grain samples (81-109 PTI), and <0.02-0.38 ppm in/on 20 fodder samples (81-109 day PTI). Following two applications (one to 15" tall plants and one at the soft dough stage) of each of the four dicamba formulations at 0.25 lb ae/A/application (0.5 lb ae/A/season), the maximum combined residues of dicamba and its 5-hydroxy metabolite were 2.73 ppm in/on 20 grain samples (30-42 PTI) and 8.2 ppm in/on 20 fodder samples (30-42 PTI). Residues of dicamba and its 5-hydroxy metabolite were nondetectable (<0.01 ppm for each analyte) in/on five control samples of each silage, grain, and fodder and four forage control samples.

The submitted sorghum data are inadequate based upon the registrant's intent to support the use of two postemergence applications of dicamba, each at 0.25 lb ae/A (0.5 lb ae/A/season) with the second application being made at the soft dough stage. The available data indicate that the established 3 ppm for grain and fodder may be appropriate for grain and is too low for fodder/stover. However, additional data are required before a tolerance assessment can be made for grain, forage, and fodder/stover.

Geographic representation is not adequate as no studies were conducted in Regions 2, 4 and 7 (see OPPTS Test Guidelines, Series 860, Residue Chemistry, 860.1500). Data are required for Regions 2, 4 and 7 depicting the combined residues of dicamba and its 5-hydroxy metabolites in/on sorghum grain and fodder harvest 30 days after the second of two postemergence applications of dicamba each at 0.25 lb. In addition, the available sorghum forage data reflect only the single early season application of dicamba which is 0.5x the proposed application rate. For sorghum forage, the Agency requires data on forage sampled at the late dough stage, which occurs after the registrant's proposed application at the soft dough stage. Accordingly, residue data are required for sorghum forage following the second of two postemergence applications of dicamba each at 0.25 lb. Tests on sorghum forage are required in the following geographic

regions: 2, 4, 5, 6, 7, and 8. As the available data indicate that the counter ion of the salt had no affect on the level of residues, the required studies need only be conducted using a single representative salt formulation. We recommend that for new each field trial required, two independently composited samples be taken.

In addition, the registrant must (i) indicate consistently on all end-use product labels registered for use on sorghum the maximum use rate, and minimum PHI, and (ii) specify on all labels that aerial applications must be made in >2 GPA or provide the appropriate field trial data for low volume aerial application.

Table 5. Residues of dicamba and its 5-hydroxy metabolite in sorghum commodities harvested following one application at 0.25 lb ae/A (0.5x) of various dicamba salt formulations.

Loc (PTI) [a]	Na^+ Salt			DGA^+ Salt			K^+ Salt			DMA^+ Salt		
	Dicamba	5-OH	Total	Dicamba	5-OH	Total	Dicamba	5-OH	Total	Dicamba	5-OH	Total
Residues (ppm) in Forage												
MO (29)	0.038	0.025	0.063	0.035	0.022	0.057	0.097	0.061	0.158	0.044	0.027	0.071
NE (54)	0.22	0.14	0.36	0.17	0.11	0.28	0.18	0.082	0.262	0.24	0.13	0.37
OK (20)	0.30	0.079	0.379	0.32	0.11	0.43	0.38	0.08	0.46	0.12	0.044	0.164
TX (30)	0.068	0.082	0.15	0.020	0.024	0.044	0.027	0.035	0.062	0.014	0.015	0.029
Residues (ppm) in Silage												
MO (61)	0.037	0.032	0.069	0.031	0.020	0.051	0.065	0.066	0.131	0.035	0.039	0.074
NE (48)	0.11	0.039	0.149	0.17	0.054	0.224	0.20	0.07	0.27	0.20	<0.01	0.21
OK (58)	0.071	0.038	0.109	0.085	0.085	0.17	0.052	0.044	0.096	0.053	0.055	0.108
TX (30)	0.037	<0.01	0.047	0.025	0.032	0.057	0.034	0.040	0.074	0.035	0.033	0.068
KS (56)	0.070	0.099	0.169	0.069	0.110	0.179	0.055	0.077	0.132	0.062	0.093	0.155
Residues (ppm) in Grain												
MO (109)	--[b]	--[b]	--[b]	--[b]	--[b]	--[b]	0.026	<0.01	0.036	0.026	<0.01	0.036
NE (96)	0.028	0.016	0.044	0.045	0.025	0.070	0.084	0.04	0.124	0.12	0.064	0.184
OK (98)	0.028	0.019	0.047	0.024	0.017	0.041	0.016	0.01	0.026	0.017	0.01	0.027
TX (81)	0.013	0.013	0.026	<0.01	<0.01	<0.02	<0.01	<0.01	<0.02	<0.01	0.010	0.02
KS (92)	0.035	0.019	0.054	0.030	0.013	0.043	0.031	0.015	0.046	0.031	0.018	0.049
Residues (ppm) in Fodder												
MO (109)	0.033	0.014	0.047	0.036	<0.01	0.037	0.041	0.022	0.063	<0.01	0.011	0.021
NE (96)	0.12	<0.01	0.13	0.099	<0.01	0.109	0.19	<0.01	0.2	0.16	0.05	0.21
OK (98)	0.22	0.16	0.38	0.22	0.15	0.37	0.081	0.045	0.126	0.14	0.088	0.228
TX (81)	0.012	<0.01	0.022	<0.01	<0.01	<0.02	<0.01	0.027	0.037	<0.01	<0.01	<0.02
KS (92)	0.081	0.11	0.191	0.057	0.13	0.187	0.054	0.084	0.138	0.057	0.11	0.167

[a] PTI is given in days.
[b] Samples lost due to labeling error.

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Table 6. Residues of dicamba and its 5-hydroxy metabolite in sorghum commodities harvested following two applications at 0.25 lb ae/A (0.50 lb ae/A total seasonal rate; 1x) of various dicamba salt formulations.

Loc	Na^+ Salt			DGA^+ Salt			K^+ Salt			DMA^+ Salt		
(PTI) [a]	Dicamba	5-OH	Total	Dicamba	5-OH	Total	Dicamba	5-OH	Total	Dicamba	5-OH	Total
Residues (ppm) in Grain												
MO (42)	1.28	0.22	1.5	0.81	0.30	1.11	0.81	0.33	1.14	0.81	0.27	1.08
NE (42)	0.032	0.015	0.047	0.038	0.021	0.059	0.099	0.048	0.147	0.096	0.048	0.144
OK (30)	0.39	0.15	0.54	0.34	0.17	0.51	0.36	0.17	0.53	0.30	0.19	0.49
TX (31)	0.92	1.77	2.69	0.97	1.76	2.73	0.76	1.24	2.0	0.89	1.57	2.46
KS (30)	0.82	0.43	1.25	0.69	0.35	1.04	0.72	0.41	1.13	1.18	0.52	1.7
Residues (ppm) in Fodder												
MO (42)	0.10	0.053	0.153	0.17	0.10	0.27	0.31	0.13	0.44	0.26	0.11	0.37
NE (42)	0.12	0.04	0.16	0.24	0.051	0.291	0.098	0.034	0.132	0.12	0.044	0.164
OK (30)	5.35	2.87	8.22	4.65	0.12	4.77	4.87	0.064	4.934	1.79	1.08	2.87
TX (31)	1.22	0.061	1.281	0.90	2.01	2.91	0.83	1.95	2.78	1.25	2.81	4.06
KS (30)	0.81	1.45	2.26	0.11	0.24	0.35	0.59	1.11	1.7	0.42	0.70	1.12

[a] PTI is given in days.

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Asparagus. The Dicamba SRR required additional data depicting residues of dicamba and its 5-hydroxy metabolite in/on asparagus. In response, the registrant submitted a draft protocol for asparagus field trials (CBRS Nos. 11617 and 11626, DP Barcodes D189393 and D189595, S. Knizner, 6/11/93). The Agency concluded that the registrant should minimally conduct field trials at two different sites in CA, WA, and MI, for a total of six field trial sites. At each field trial site, the 4 lb/gal SC/L DMA^+ salt, the 4 lb/gal SC/L DGA^+ salt, and the 2 lb/gal SC/L Na^+ salt formulations, in separate experiments, must be applied at the maximum label rate. CBRS also recommended that two independently composited samples be taken and analyzed for residues of dicamba, 5-hydroxy dicamba, and DCSA. Supporting storage stability data were required.

A tolerance of 3.0 ppm has been established for the combined residues of dicamba and its 5-hydroxy metabolite in/on asparagus. [40 CFR § 180.227(a)]. The Dicamba SRR noted that asparagus should be listed under 40 CFR 180.227(b) as the predominant terminal residue is 3,6-dichloro-2-hydroxybenzoic acid, and not 5-hydroxy dicamba.

A REF's search dated 11/16/94 identified two dicamba end-use products registered to Sandoz for use on asparagus: a 4 lb/gal SC/L DMA^+ salt formulation (EPA Reg. No. 55947-1) and a 4 lb/gal SC/L K^+ salt formulation (EPA Reg. No. 55947-38). Both labels specify postemergence broadcast or banded applications in 46-60 GPA. The 4 lb/gal SC/L K^+ salt formulation label species a maximum of one application per season at 0.5 lb ae/A. The 4 lb/gal SC/L DMA^+ salt formulation specifies that multiple applications may be made, with a total seasonal maximum of 0.5 lb ae/A. A 24-hour PHI is in effect. The labels restrict the use to the states of CA, OR, and WA only.

Sandoz submitted data (1994; MRIDs 43245206 and 43425803) from a total of 24 tests conducted in CA (3) and WA (3) in 1993 and in CA (6), MI (6), and WA (6) in 1994 depicting residues of dicamba, 5-hydroxy dicamba, and DCSA in/on asparagus. For the tests conducted in 1994, the 6 tests in each state represent two test locations (1 and 2) with three tests at each site. At each test site, the following three dicamba formulations were used: dicamba Na^+ salt (2.0 lb/gal SC/L), dicamba DGA^+ salt (4.0 lb/gal SC/L), dicamba DMA^+ salt (4.0 lb/gal SC/L). At each test site, one broadcast application of each of the three dicamba formulations was made at 0.5 lb ae/A (1x) in 20-50 GPA of water using ground equipment. Asparagus spears were harvested 24 hours after application. For each test, two treated samples were analyzed for dicamba and DCSA using the GC/ECD method AM-0766A as described earlier in this report. One to two treated samples were analyzed for 5-hydroxy dicamba using method AM-0691B, also previously described. For each test site, one to two control samples were analyzed for 5-hydroxy dicamba and two controls were analyzed for dicamba and DCSA. Samples were stored frozen at <-12 C for 12-95 days prior to analysis. Adequate storage stability data were submitted for these storage interval and conditions. The combined residues are presented in Table 7.

The combined residues of dicamba and its DCSA metabolite were 0.28-3.29 ppm in/on 48 asparagus samples harvested 24 hours following a single application of one of three dicamba salt

SC/L formulations at 0.5 lb ae/A. Residues of 5-hydroxy dicamba were <0.01-0.011 ppm in/on 42 of the treated samples. Residues of dicamba, DSCA, and 5-hydroxy dicamba were nondetectable (<0.01 ppm) in/on 13-14 controls. Residues of dicamba were 0.015 and 0.017 ppm in/on two controls and residues of DCSA were 0.011-0.013 ppm in/on three controls.

Geographic representation is adequate. The current labels indicate that use on asparagus is restricted to the states of CA, OR, and WA only.

The submitted asparagus field trials are adequate. The maximum combined residues of dicamba and DCSA was 1.1 ppm in/on 36 asparagus samples from CA and WA harvested 24 hours following a single application at 0.5 lb ae/A of the Na^+ salt, DGA^+ salt, or the DMA^+ salt of dicamba. The maximum combined residues of dicamba and DCSA was 3.29 ppm in/on 12 asparagus samples grown in MI harvested 24 hours following a single application at 0.5 lb ae/A of the dicamba Na^+, DGA^+, or DMA^+ salt formulation. The field trial data indicate that the residue levels are not dependent on the salt formulation used. No additional data are required.

If the registrant wishes to include use in MI, then a tolerance of 3.5 ppm should be proposed for the combined residues of dicamba and DCSA in/on asparagus (see CBTS 16431 et al, F. Griffith, 5/2/96).

Table 7. Residues of dicamba and DCSA metabolite in/on asparagus following a single application of 0.50 lb ae/A of a DMA^+, a DGA^+, or a Na^+ salt of dicamba.

Location (MRID)	Dicamba (ppm)	DCSA (ppm)	Total Residues (ppm) [a]
DMA^+ Salt			
CA (43245206)	0.387, 0.435	<0.01, 0.017	0.397, 0.452
CA 1 (43425803)	0.578, 0.444	<0.01, <0.01	0.588, 0.454
CA 2 (43425803)	0.422, 0.350	<0.01, <0.01	0.432, 0.360
WA (43245206)	0.560, 0.595	0.035, <0.01	0.595, 0.605
WA 1 (43425803)	1.092, 0.853	0.010, 0.011	1.102, 0.864
WA 2 (43425803)	0.468, 0.794	<0.01, 0.014	0.478, 0.808
MI 1 (43425803)	0.880, 1.064	0.012, 0.010	0.892, 1.074
MI 2 (43425803)	3.112, 3.175	0.026, 0.017	3.138, 3.192
DGA^+ Salt			
CA (43245206)	0.361, 0.452	0.015, 0.011	0.376, 0.463
CA 1 (43425803)	0.524, 0.502	<0.01, <0.01	0.534, 0.512
CA 2 (43425803)	0.493, 0.287	<0.01, <0.01	0.503, 0.297
WA (43245206)	0.781, 0.920	0.071, <0.01	0.852, 0.930
WA 1 (43425803)	0.771, 0.983	<0.01, <0.01	0.781, 0.993
WA 2 (43425803)	0.266, 0.537	<0.01, <0.01	0.276, 0.547
MI 1 (43425803)	0.994, 1.366	0.013, 0.011	1.007, 1.377
MI 2 (43425803)	3.159, 2.421	0.018, <0.01	3.177, 2.431
Na^+ Salt			
CA (43245206)	0.366, 0.399	<0.01-0.010	0.376, 0.409
CA 1 (43425803)	0.465, 0.559	<0.01, <0.01	0.475, 0.569
CA 2 (43425803)	0.281, 0.327	<0.01, <0.01	0.291, 0.337
WA (43245206)	0.496, 0.546	<0.01, 0.016	0.506, 0.562
WA 1 (43425803)	0.604, 0.935	<0.01, 0.012	0.614, 0.947
WA 2 (43425803)	0.607, 0.955	<0.01, <0.01	0.617, 0.965
MI 1 (43425803)	2.326, 1.570	0.027, 0.018	2.353, 1.588
MI 2 (43425803)	3.274, 2.685	0.019, 0.018	3.293, 2.703

[a] The total includes only the dicamba and DCSA values as these are the residues of concern in asparagus.

Magnitude of the Residue in Processed Food/Feed Commodities

Sugarcane. The Dicamba SRR (6/89) required a processing study depicting the combined residues of dicamba and 5-hydroxy dicamba in molasses, refined sugar, and bagasse processed from sugarcane bearing measurable, weathered residues. In response, the registrant submitted a draft protocol for a sugarcane processing study (S. Knizner, 6/11/93). The Agency reiterated the SRR data requirements and also required supporting storage stability data.

A food/feed additive tolerance of 2.0 ppm has been established for the combined residues of dicamba and its 5-hydroxy metabolite in/on sugarcane molasses. [40 CFR § 185.1800 and 186.1800].

A REF's search dated 11/16/94 identified three dicamba end-use products registered to Sandoz for use on sugarcane: two DMA salt formulations, a 4 lb/gal SC/L and a 1 lb/gal SC/L (EPA Reg. Nos. 55947-1 and -24), and a 4 lb/gal SC/L K^+ salt formulation (EPA Reg. No. 55947-38). The two DMA salt SC/L formulation labels indicate that multiple applications at 0.25-2 lb ae/A/application can be made for a total seasonal maximum of 2 lb ae/A. The 4 lb/gal SC/L K^+ salt formulation specifies multiple applications at.0.25-1 lb ae/A/application not to exceed 3 lb ae/A/season. Application of the K^+ salt formulation is not permitted in HI and aerial applications are not permitted in LA. The labels specify that applications must be made prior to the close-in stage of sugarcane, however, exact PHIs are not specified. The applications must be made in 3-50 GPA for ground equipment. For aerial equipment, the 1 lb/gal SC/L DMA^+ salt and the 4 lb/gal SC/L K^+ salt formulations indicate that applications are to be made in 3-10 GPA. The 4.0 lb/gal SC/L DMA^+ salt specifies that applications are to be made in 2-20 GPA for preharvest use, while all other uses can be applied in 1-10 GPA.

In response to the SRR data requirements, Sandoz submitted data (1994; MRID 43245204) from two tests conducted in Opelousas (1) and Washington (1), LA depicting residues of dicamba and its 5-hydroxy metabolite in sugarcane processed fractions. A single broadcast application of the 4.0 lb/gal SC/L DMA^+ salt formulation was applied at 5.0 lbs ae/A (presented as 5x) to sugarcane at layby for both tests. Applications were made in 10 and 11.6 GPA. RAC samples from both sites were harvested and analyzed by Sandoz Agro, Inc., Des Plaines, IL as prequalifier samples. Residues in the RAC (130-day PTI) sample collected from the Washington, LA trial were higher and this sample was the only one processed. The RAC sample was processed within 15 days of harvest following standard commercial practices into bagasse, molasses, and refined sugar by the Audubon Sugar Institute at Louisiana State University in Baton Rouge. The RAC and processed samples were stored frozen (<-1 C) for up to 58 and 64 days, respectively, prior to analysis. The submitted storage stability data are adequate to support the storage interval and conditions for refined sugar. However, additional storage stability data are required for molasses and sugarcane. The submitted storage stability data for bagasse are inadequate, however, these data are not required at this time. See the Storage Stability section of this report for details.

Residues of dicamba and 5-hydroxy dicamba were nondetectable (<0.01 ppm for each analyte) in/on 1 control of each matrix. Residues of dicamba and 5-hydroxy dicamba in the treated sugarcane and its processed commodity samples are detailed in Table 8. Combined residues of dicamba and 5-hydroxy dicamba were 0.054 ppm in sugarcane and 1.320 ppm in molasses indicating a concentration factor of 24x.

The submitted sugarcane processing study is adequate, pending submission of adequate supporting storage stability data for sugarcane and molasses. Based on the field trial data submitted in PP#1F2569, the HAFT was determined to be 0.036 ppm (Accession # 070319, Belle Glade, FL, 3 lb ae/A and PHI of 158 days). Based on the concentration factor of 24.4, a tolerance of 1 ppm for molasses would be appropriate. No concentration of residues was observed in white refined sugar.

Table 8. Residues of dicamba and 5-hydroxy dicamba in/on sugarcane following a single application of a 4 SC/L (DMA^+ salt) at 5.0 lbs ae/A and in its processed commodities.

Commodity	Residues (ppm) [a]			Concentration factor
	Dicamba	5-hydroxy dicamba	Total	
Sugarcane	0.012-0.016 (0.013)	0.038-0.043 (0.040)	0.050-0.059 (0.054)	--
Bagasse	0.210-0.225 (0.220)	0.132-0.140 (0.136)	0.350-0.361 (0.356)	6.6
Molasses	0.537-0.579 (0.549)	0.694-0.856 (0.771)	1.234-1.393 (1.320)	24.4
Refined sugar	ND [b]	ND	ND	--

[a] Average listed parenthetically, used to determine the concentration factors.
[b] ND=nondetectable (<0.01 ppm).

Sorghum. The Dicamba SRR (6/89) required a processing study depicting the combined residues of dicamba and its 5-hydroxy metabolite in milled products (flour and starch) and grain dust from sorghum grain bearing measurable, weathered residues. In response, the registrant submitted a protocol (S. Knizner, 6/11/93) and a rebuttal to the review of the protocol (R. Perfetti, 10/20/93). The Agency concluded that residue data are not required for flour or starch and that data is required for the RAC grain dust. Supporting storage stability data were also required.

In response, Sandoz submitted data (1994; MRID 43245205) from two tests conducted in Peru (1) and Dodge City (1), KS depicting residues of dicamba and its 5-hydroxy metabolite in sorghum aspirated grain fractions. Applications were made at a total of 0.5 lb ae/A (1x) and 1.5 lb ae/A (3x) at both sites. Grain samples from both sites were harvested and analyzed by Sandoz Agro, Inc., Des Plaines, IL as prequalifier samples. Residues in the 3x trial from Dodge City, KS were the highest; therefore, only these grain samples were processed. For the 3x Dodge City trial, two postemergence applications, one to 15" tall sorghum and one after the soft dough stage, of the 4.0 lb/gal SC/L DMA^+ salt formulation were made to sorghum at 0.75 lb ae/A/application for a total season application of 1.5 lb ae/A (3x). Applications were made in 20 GPA and samples were harvested at a 30-day PTI. The 3x treated and control RAC samples were sent via freezer truck from Sandoz Agro, Inc. to the processing facility at the Food Protein R & D Center

in Bryan, TX. The grain samples were in transit for 3 weeks. Grain dust was generated in a steel bucket elevator using a process that was designed to simulate a commercial elevator operation. Upon generation, the grain dust samples were sent to SAI where they were held frozen at <-17 C for up to 2 months prior to analysis. The treated RAC sample was held frozen for 4 months prior to analysis. The submitted storage stability data indicate that residues of dicamba and its 5-hydroxy metabolite are stable in grain and grain dust for the storage intervals reflected in the current submission. One treated grain sample and one of each >2030, >1180, >850, >425, and <425 µm treated grain dust samples were analyzed. One control grain sample was analyzed. Residues of dicamba and 5-hydroxy dicamba were determined using the GC/ECD method AM-0691B.

Residues of dicamba and 5-hydroxy dicamba in/on sorghum grain and aspirated grain fractions are presented in Table 9. The maximum combined residues of dicamba and 5-hydroxy dicamba were 27.1 ppm in/on five grain dust samples ranging in size from <425 µm to >2030 µm generated from grain treated at 3x the stated seasonal rate (1.5 lb ae/A). Residues of dicamba and 5-hydroxy dicamba were nondetectable (<0.01 ppm for each analyte) in/on one control grain sample. Residues of dicamba and 5-hydroxy dicamba were also ≤0.01 ppm in control grain dust samples, except for the >2030µm dust sample which contained apparent dicamba residues of 0.018 ppm.

The submitted sorghum processing study is adequate. The maximum combined residues of dicamba and 5-hydroxy dicamba were 27.1 ppm in/on aspirated grain fractions generated from sorghum grain treated at a total of 1.5 lb ae/A (3x). A tolerance will be required for aspirated grain fractions as a RAC under 40 CFR §180.227. However, the tolerance for aspirated grain fractions is set on the maximum residues found in the grain dust of corn, wheat, sorghum, or soybeans. Before a tolerance for aspirated grain fractions can be proposed, data are required from field trials depicting the residues of corn, wheat, and soybean aspirated grain fractions generated from grain treated at the maximum label use rate for each crop and at the minimum PHIs (see OPPTS Test Guidelines, Series 860, Residue Chemistry, 860.1500).

Table 9. Residues in sorghum grain dust generated from sorghum grain treated with two applications at 0.75 lb ae/A/application for a total season application of 1.5 lb ae/A (3x).

Commodity	Residues (ppm)		
	Dicamba	5-hydroxy dicamba	Total
Sorghum grain	4.8	2.1	6.9
>2030 µm	17.6	9.5	27.1
>1180 µm	16.0	6.0	22.0
>850 µm	10.6	3.4	14.0
>425 µm	11.6	3.2	14.8
<425 µm	10.0	3.5	13.5

MASTER RECORD IDENTIFICATION NUMBER

The citations for the MRID documents used in this review are presented below.

43245203 Laban, S. (1994) Crop Residue Study with Dicamba Formulations on Grain Sorghum: Project No. 480068. Unpublished study prepared by Sandoz Agro, Inc. 684 p.

43245204 Formanski, L. (1994) Dicamba Residue Study on Sugarcane and Sugarcane Processed Fractions: Project No. 4800658. Unpublished study prepared by Sandoz Agro, Inc. 496 p.

43245205 Rosas, M. (1994) Dicamba Residue Study on Sorghum Grain and Sorghum Processed Fractions: Project No. 480068. Unpublished study prepared by Sandoz Agro, Inc. 281 p.

43245206 Clouser, A. (1994) Crop Residue Study with Dicamba Formulations on Asparagus: Project No. 480068. Unpublished study prepared by Sandoz Agro, Inc. 322 p.

43274501 Guirguis, M. (1994) Crop Residue Study with Dicamba Formulations on Wheat (Forage and Hay): Project No. 480068. Unpublished study prepared by Sandoz Agro, Inc. 740 p.

43425803 Clouser, A. (1994) Crop Residue Study with Dicamba Formulations on Asparagus: Project No. 480068. Unpublished study prepared by Sandoz Agro, Inc. 590 p.

AGENCY MEMORANDA CITED IN THIS DOCUMENT

CBRS No.:	13923
DP Barcode:	D204754
Subject:	Sandoz Proposal for the Replacement of Craven Data.
From:	S. Funk
To:	W. Waldrop/J. Mitchell
Date:	7/12/94
MRID(s):	None

CBRS No.:	12482
DP Barcode:	D194776
Subject:	Independent Method Validation.

From: D. Miller
To: J. Mitchell
Date: 12/14/93
MRID(s): 42883201

CBRS No.: None
DP Barcode: None
Subject: Additional Requirements to Replace Craven Data.
From: S. Funk
To: W. Waldrop/J. Mitchell
Date: 10/22/93
MRID(s): None

CBRS No.: 12546
DP Barcode: D195031
Subject: Response to the Dicamba Reregistration Standard
From: R. Perfetti
To: L. Rossi
Date: 10/20/93
MRID(s): None

CBRS No.: 11617 and 11626
DP Barcode: D189393 and D189595
Subject: Protocols for Guidelines and Waiver for FDA Multiresidue Method Testing.
From: S. Knizner
To: J. Mitchell
Date: 6/11/93
MRID(s): None

CBTS No.: 8617
DP Barcode: D169036
Subject: Impact of Craven Laboratories Analytical Data on Registrations
From: R. Lascola
To: R. Taylor
Date: 1/17/92
MRID(s): None

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R111739

Chemical:	**Dicamba**
PC Code:	**029801**
HED File Code	**11000 Chemistry Reviews**
Memo Date:	**07/31/1997**
File ID:	**DPD204488; DPD204809; DPD209229**
Accession Number:	**412-05-0098**

HED Records Reference Center
08/08/2005