PP# 4F04447
10-21-96

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OCT 21 1996

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP# 4F04407. Sulfentrazone (Authority Herbicide) for Use on Soybeans. Amendment of 9/24/96. Revised Section F, Analytical Method, and Reanalysis of Samples. MRID# 441188-01 thru -07. Barcode D230444. CBTS#s 17599. Chemical# 129081. Case 285935.

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FMC has submitted a petition for permanent tolerances for the combined residues of the herbicide sulfentrazone (N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1*H*-1,2,4-triazol-1-yl]phenyl]methanesulfonamide) and its major metabolite 3-hydroxymethyl sulfentrazone (N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-hydroxymethyl-5-oxo-1*H*-1,2,4-triazol-1-yl]phenyl] methanesulfonamide) on soybeans and rotational crops. For residues on the primary crop, the petitioner has proposed the following tolerance (expressed as the combined residues of parent plus the metabolite 3-hydroxymethyl sulfentrazone):

Soybean Seed	--	0.05 ppm

For residues in rotational crops (inadvertent residues), the petitioner has proposed the following tolerances (expressed as the combined residues of parent plus the metabolites 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone [N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-5-oxo-1*H*-1,2,4-triazol-1-yl]phenyl]methanesulfonamide]):

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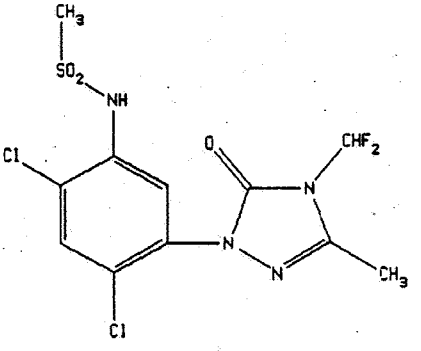
Cereal Grains (excluding sweet corn), Forage	--	0.2 ppm
Cereal Grains (excluding sweet corn), Straw	--	0.6 ppm
Cereal Grains (excluding sweet corn), Hay	--	0.2 ppm
Cereal Grains (excluding sweet corn), Grain	--	0.1 ppm
Cereal Grains (excluding sweet corn), Stover	--	0.1 ppm
Cereal Grains (excluding sweet corn), Bran	--	0.1 ppm
Cereal Grains (excluding sweet corn), Hulls	--	0.2 ppm

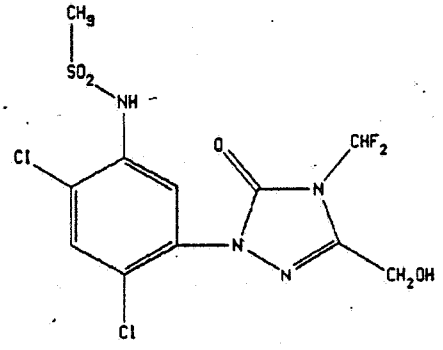
Note that these tolerances were previously proposed on a time-limited basis.

The current amendment addresses deficiencies identified in CBTS's previous reviews (Memos, G. Kramer 7/1/96 & 7/31/96).

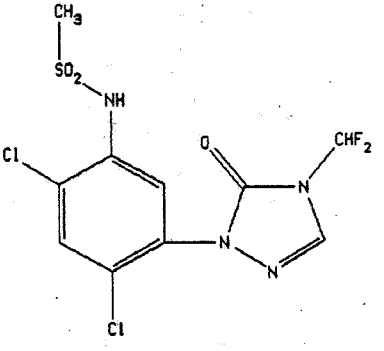
The structures of sulfentrazone and its metabolites are shown below:



Sulfentrazone



3-Hydroxymethyl Sulfentrazone



3-Desmethyl Sulfentrazone

Executive Summary of Chemistry Deficiencies for Permanent Tolerances

- Agency validation of new analytical method for plants.
- Revised Section F.
- Wheat processing study.
- Additional rice residue data.
- Residue data for sorghum aspirated grain fractions.
- May need animal feeding studies.

RECOMMENDATIONS

CBTS continues to recommend against permanent tolerances for residues of sulfentrazone and its metabolites on soybeans, cereal grain RACs and processed commodities for reasons detailed in conclusions 1b, 2b, 3b, 4b, 5, 6, 7 and 8, below.

CONCLUSIONS

1a. The petitioner contends that data for wheat processed fractions are not required as multiplication of the maximum theoretical concentration factor for wheat (8.3X, milled byproducts) by the maximum residue observed in wheat grain (0.007 ppm) results in a value below the proposed cereal grain tolerance (0.1 ppm).

1b. In a cereal grain crop group tolerance, wheat serves as a representative for many other commodities. Thus, any concentration factors observed in a wheat processing study would be applied to the highest average field trial value for any cereal grain (i.e.; 0.068 ppm in rice grain, see below). Also, concentration factors in aspirated grain fractions can exceed several hundred. CBTS thus reiterates that a wheat processing study is required for this petition. If residues are observed to concentrate in bran, then data should also be provided for wheat aspirated grain fractions.

2a. The previously submitted analytical method for plant matrices has been modified to include a more stringent hydrolysis step and by use of a more specific GC detector (ELCD instead of ECD). All three analytes are resolved in a single chromatographic separation.

2b. An ILV of this method was performed by CAL Labs. Acceptable

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recoveries were obtained by the independent laboratory. The revised method has also undergone a successful radiovalidation. The method and ILV will be sent to Beltsville for PMV. CBTS concludes that the revised method is adequate for data gathering purposes. A conclusion on the adequacy of the method for enforcement of the proposed tolerances will be withheld pending satisfactory Agency validation.

3a. Samples from six trials for each crop were reanalyzed with the new method. When reanalyzed with this method, the total residues of sulfentrazone and its metabolites were generally 2-3X higher in soybean seed, 1.5-6X higher in cereal grain forage, 2-5X higher in cereal grain hay, 3-6X higher in cereal grain straw, 1.5-3X higher in cereal grain stover and unchanged in grain. Adequate storage stability data were submitted to confirm the stability of sulfentrazone and its metabolites for the additional storage intervals. Based on these reanalyses, the proposed tolerance level of 0.05 ppm for soybeans is adequate.

3b. Total residues of sulfentrazone and its metabolites were found to concentrate in rice hulls (3.9X) and bran (1.9X). Based on these concentration factors and the current highest average field trial value for cereal grain (0.068 ppm, rice), the appropriate tolerances for cereal grain hulls and bran would be 0.15 ppm and 0.30 ppm, respectively. A final conclusion on the appropriate tolerances for these commodities will be made once the additional rice residue data (see below) and wheat processing study are submitted. A revised Section F will be required.

4a. The petitioner contends that data for sorghum aspirated grain fractions are not required as residues were below the LOQ in all sorghum grain samples.

4b. According to the 860.1500 Guidelines, data for aspirated grain fractions are not required when residues are below the LOQ in grain samples only in situations where the pesticide is applied after the reproductive stage begins. For situations where the pesticide is applied prior to the reproductive stage, data for aspirated grain fractions are not required <u>unless</u> the processing study shows concentration of residues in the outer seed coat. Sulfentrazone residues have been shown to concentrate in rice bran and hulls. CBTS thus reiterates that residue data are required for sorghum aspirated grain fractions

5. The petitioner previously submitted the results of nine rotational rice trials. This number does not correspond to that required for rice as a representative commodity of the cereal grains crop group. CBTS concludes that an additional four rice field trials are required.

6. A conclusion on the appropriate plantback restrictions and on the levels for the permanent tolerances will be withheld pending

submission and review of additional residue data (rice field trials) and processing studies (sorghum aspirated grain fractions and wheat processing).

7. CBTS will reevaluate the need for a cow feeding study once the appropriate tolerance levels are determined for rotational crops by submission of additional residue data and processing studies.

8. HED notes that the Food Quality Protection Act of 1996 has amended and strengthened the standard for establishing tolerances under the FFDCA. OPP is still assessing the full impact of this change in the law on the tolerance-setting process and plans to issue guidelines concerning the establishment of tolerances under the amended statute. All tolerance petitions have to meet the requirements of the FFDCA as amended by the FQPA and OPP may require additional data to determine if the terms of the amended statute are met.

DETAILED CONSIDERATIONS

Deficiency - Conclusion 1c (from Memo, G. Kramer 7/1/96)

1c. The petitioner previously requested a data waiver for the wheat processing study. As residues of sulfentrazone and its metabolites were nondetectable in grain samples from the first five limited field trials, CBTS conditionally recommended in favor of this data waiver request pending resolution of all deficiencies related to the proposed wheat tolerances (Memo, G. Kramer 7/26/95). However, in the residue data submitted with this amendment, detectable residues were found in grain in over one half of the trials. CBTS thus concludes that a wheat processing study will be required for this petition. If concentration of residues is observed in bran, then residue data should also be provided for wheat aspirated grain fractions.

Petitioner's Response: Submission of a data waiver request. The petitioner contends that data for wheat processed fractions are not required as multiplication of the maximum theoretical concentration factor for wheat (8.3X, milled byproducts) by the maximum residue observed in wheat grain (0.007 ppm) results in a value below the proposed cereal grain tolerance (0.1 ppm).

CBTS's Conclusion: In a cereal grain crop group tolerance, wheat serves as a representative for many other commodities. Thus, any concentration factors observed in a wheat processing study would be applied to the highest average field trial value for any cereal grain (i.e.; 0.068 ppm in rice grain, see below). Also, concentration factors in aspirated grain fractions can exceed several hundred. CBTS thus reiterates that a wheat processing study is required for this petition. If residues are observed to concentrate in bran, then data should also be provided for wheat

aspirated grain fractions.

Deficiency - Conclusion 8a (from Memo, G. Kramer 7/1/96)

8a. The petitioner has developed a streamlined method (P-3063M, MRID# 440056-01) which simultaneously measures all three analytes in rotational crops. This method is also very similar to the analytical enforcement method for soybeans. However, in a recent meeting with representatives of FMC, CBTS was informed that the current methodology fails to release a significant portion of the conjugated 3-hydroxymethyl sulfentrazone. The petitioner is in the process of developing a new enforcement method for soybeans and rotational crops. The revised method will be submitted along with an ILV. CBTS will then initiate a PMV. The petitioner has also agreed to reanalyze selected field samples of every RAC associated with this petition using the new method. At least six samples of each RAC will be analyzed, including those which contained the greatest residues when analyzed with the previous methods. Supporting storage stability data will also be provided.

Petitioner's Response: Submission of:

Analytical Methodology for the Determination of Sulfentrazone, 3- Desmethyl Sulfentrazone, and 3-Hydroxymethyl Sulfentrazone in/on Various Matrices. P-3173. Appendix B of MRID# 441188-01.

Independent Laboratory Validation of Analytical Methodology for the Determination of Sulfentrazone, 3- Desmethyl Sulfentrazone, and 3-Hydroxymethyl Sulfentrazone in/on Wheat Forage. Centre Analytical Labs. MRID# 441188-02.

Magnitude of the Residue of Sulfentrazone and 3-Hydroxymethyl Sulfentrazone in Soybean Seed: Re-Analysis Of Selected Trials Using Revised Methodology. MRID# 441188-03.

Magnitude of the Residue of Sulfentrazone and Its Major Metabolites in/on Winter Wheat as a Rotated Crop Following Soybeans Treated with Authority Herbicide: Re-Analysis Of Selected Trials Using Revised Methodology. MRID# 441188-04.

Magnitude of the Residue of Sulfentrazone and Its Major Metabolites in/on Field Corn as a Rotated Crop Following Soybeans Treated with Authority Herbicide: Re-Analysis Of Selected Trials Using Revised Methodology. MRID# 441188-05.

Magnitude of the Residue of Sulfentrazone and Its Major Metabolites in/on Rice as a Rotated Crop Following Soybeans Treated with Authority Herbicide: Re-Analysis Of Selected Trials Using Revised Methodology. MRID# 441188-06.

Magnitude of the Residue of Sulfentrazone and Its Major Metabolites in/on Sorghum as a Rotated Crop Following Soybeans

Treated with Authority Herbicide: Re-Analysis Of Selected Trials Using Revised Methodology. MRID# 441188-07.

Method: The previously submitted method has been modified to include a more stringent hydrolysis step and by use of a more specific GC detector (ELCD instead of ECD). All three analytes are resolved in a single chromatographic separation. The method was validated in cereal grain RACs over a range of 0.025-0.50 ppm. The average recovery for sulfentrazone was 90 ± 9% (n=28); for 3-desmethyl sulfentrazone, 92 ± 18% (n=25); and for 3-hydroxymethyl sulfentrazone, 84 ± 14% (n=28). An ILV of this method was performed by Centre Analytical Labs. The method was validated by the independent lab in wheat forage at 0.025 and 0.20 ppm. The average recovery for sulfentrazone was 87 ± 7% (n=4); for 3-desmethyl sulfentrazone, 100 ± 18% (n=4); and for 3-hydroxymethyl sulfentrazone, 74 ± 3% (n=4).

Soybeans: Samples from six trials were reanalyzed with the new method. The method was validated in soybeans at 0.025 ppm. The average recovery for sulfentrazone was 83 ± 3% (n=3); and for 3-hydroxymethyl sulfentrazone, 82 ± 18% (n=3). The maximum storage interval for the samples was 47 months. Storage stability studies for sulfentrazone and hydroxymethyl sulfentrazone on soybeans have been conducted. Sulfentrazone has shown a pattern of stability for at least 24 months and hydroxymethyl sulfentrazone for at least 11 months (Memo, G. Kramer 4/3/95). Additional storage stability information for hydroxymethyl sulfentrazone was generated during this study. At 38 months, there was still no evidence of decline. The results of reanalysis of the treated samples is shown in Table 1. When reanalyzed with the revised method, the total residues of sulfentrazone and its metabolites were generally 2-3X higher in soybean seed.

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TABLE 1- COMPARISON OF THE MAXIMUM RESIDUES (PPM) OF SULFENTRAZONE AND 3-HYDROXYMETHYL SULFENTRAZONE (HMS) IN/ON SOYBEAN SEEDS: PREVIOUSLY REPORTED VS. RE-ANALYSES WITH THE REVISED METHOD

Previous Study Number		Sulfentrazone		HMS		Total	
Trial #, State	# Analyses	Previous	Revised	Previous	Revised	Previous	Revised
162SOY92R1							
03, NE	2	ND[a]	ND	(0.007)[b]	**(0.022)**	(0.007)	**(0.022)**
07, OH	2	ND	ND	(0.009)	**0.029**	(0.009)	**0.029**
162SOY92R2							
01, GA	2[c]	ND	ND	(0.022)	**(0.006)**	(0.022)	**(0.006)**
04, LA	2	ND	ND	(0.009)	**(0.019)**	(0.009)	**(0.019)**
162SOY93R1							
03, IA	2	ND	ND	(0.006)	**0.036**	(0.006)	**0.036**
05, OH	2	ND	ND	(0.007)	**(0.016)**	(0.007)	**(0.016)**

a ND = Non-detectable (< 0.005 ppm).
b Values in parenthesis are estimates; less than the LOQ (0.025 ppm), but greater than or equal to the LOD.
c Treated samples from 162SOY92R2, trial #01 were analyzed in triplicate with the old method.

Wheat: Samples from six trials were reanalyzed with the new method. The method was validated in wheat RACs at 0.025-0.10 ppm. The average recovery for sulfentrazone was 90 ± 5% (n=8); for 3-desmethyl sulfentrazone, 103 ± 17% (n=8;) and for 3-hydroxymethyl sulfentrazone, 88 ± 17% (n=8). The maximum storage interval for the samples was 34 months. Storage stability studies for sulfentrazone, desmethyl sulfentrazone and hydroxymethyl sulfentrazone on wheat RACs have been conducted. Sulfentrazone, desmethyl sulfentrazone and hydroxymethyl sulfentrazone have shown a pattern of stability for at least 14 months of frozen storage (Memo, G. Kramer 7/1/96). Additional storage stability information for desmethyl sulfentrazone and hydroxymethyl sulfentrazone was generated during this study. At 22 months, there was still no evidence of decline. The results of reanalyses of the treated samples are shown in Table 2. When reanalyzed with the revised method, the total residues of sulfentrazone and its metabolites were generally 1.5-3X higher in wheat forage, 2-5X higher in wheat hay, and 3-6X higher in wheat straw.

TABLE 2- COMPARISON OF THE MAXIMUM RESIDUES (PPM) OF SULFENTRAZONE, DMS AND HMS IN/ON WHEAT MATRICES: PREVIOUSLY REPORTED VS. RE-ANALYSES WITH THE REVISED METHOD

Matrix / Study #	Trial #/ State	# Analyses	Sulfentrazone Previous	Sulfentrazone Revised	DMS Previous	DMS Revised	HMS Previous	HMS Revised	Trial Total Previous	Trial Total Revised
Forage										
162WHW93R1	03/GA	2	(0.007)[a]	ND[b]	0.029	**0.079**	0.052	**0.053**	0.086	**0.132**
162WHW93R1	04/MS	2	(0.016)	ND	0.046	**0.061**	(0.009)	**0.033**	0.065	**0.087**
162WHW94R1	02/LA	2	ND	ND	(0.008)	**0.028**	(0.022)	**0.054**	0.030	**0.082**
162WHW94R1	05/TX	2	ND	ND	(0.007)	**(0.022)**	0.034	**0.037**	0.041	**0.057**
162WHW94R1	07/KS	2	(0.006)	ND	(0.009)	**0.029**	0.026	**0.032**	0.041	**0.061**
162WHW94R3	03/VA	2	(0.005)	ND	(0.017)	**0.038**	(0.023)	**(0.021)**	0.045	**0.059**
Hay										
162WHW93R1	03/GA	0	NS[c]	NS	NS	NS	NS	NS	NS	NS
162WHW93R1	04/MS	0	NS	NS	NS	NS	NS	NS	NS	NS
162WHW94R1	02/LA	2	ND	**(0.014)**	(0.026)	**0.096**	(0.010)	**0.073**	(0.034)	**0.169**
162WHW94R1	05/TX	2	ND	ND	(0.043)	**0.120**	(0.012)	**0.054**	0.055	**0.174**
162WHW94R1	07/KS	2	ND	ND	(0.032)	**0.066**	ND	ND	(0.032)	**0.066**
162WHW94R3	03/VA	2	ND	ND	(0.031)	**0.069**	(0.016)	**(0.030)**	(0.046)	**0.099**
Straw										
162WHW93R1	03/GA	2	0.068	ND	(0.011)	**(0.049)**	(0.011)	**(0.026)**	0.090	**0.065**
162WHW93R1	04/MS	2	0.038	ND	0.030	**(0.011)**	0.029	ND	0.097	**(0.011)**
162WHW94R1	02/LA	2	ND	ND	0.081	**0.494**	(0.034)	**0.105**	0.107	**0.599**
162WHW94R1	05/TX	2	ND	**(0.013)**	0.077	**0.237**	(0.043)	**0.087**	0.120	**0.337**
162WHW94R1	07/KS	2	ND	**(0.011)**	0.069	**0.228**	ND	**(0.038)**	0.069	**0.268**
162WHW94R3	03/VA	2	ND	**(0.012)**	(0.043)	**0.131**	(0.030)	**0.064**	0.073	**0.207**
Grain										
162WHW93R1	03/GA	2	ND	ND	ND	ND	ND	ND	ND	ND
162WHW93R1	04/MS	2	ND	ND	ND	ND	ND	ND	ND	ND
162WHW94R1	02/LA	2	ND	ND	(0.010)	ND	ND	ND	(0.010)	ND
162WHW94R1	05/TX	2	ND	ND	(0.005)	ND	ND	ND	(0.005)	ND
162WHW94R1	07/KS	2	ND	ND	(0.005)	**(0.007)**	ND	ND	(0.005)	**(0.007)**
162WHW94R3	03/VA	2	ND	ND	ND	ND	ND	ND	ND	ND

[a] Values in parentheses are estimates; less than LOQ but greater than or equal to LOD.

[b] ND = Non-detectable (< 0.005 ppm for all grain and forage analyses; < 0.01 ppm for all hay analyses; < 0.01 ppm for all straw analyses except for Study 162WHW93R1 which is < 0.005 ppm).

[c] NS = No Samples were taken. Hay was not required by PAG Subdivision O Table II at the time of this study.

Corn: Samples from six trials were reanalyzed with the new method. The method was validated in corn RACs at 0.025-0.05 ppm. The average recovery for sulfentrazone was 89 ± 9% (n=7); for 3-desmethyl sulfentrazone, 80 ± 8% (n=7); and for 3-hydroxymethyl sulfentrazone, 76 ± 3% (n=7). The maximum storage interval for the samples was 35 months. Storage stability studies for sulfentrazone, desmethyl sulfentrazone and hydroxymethyl sulfentrazone on corn RACs have been conducted. Sulfentrazone, desmethyl sulfentrazone and hydroxymethyl sulfentrazone have shown a pattern of stability for at least 11 months of frozen storage (Memo, G. Kramer 7/1/96). Additional storage stability information for desmethyl sulfentrazone and hydroxymethyl

sulfentrazone was generated during this study. At 24 months, there was still no evidence of decline. The results of reanalyses of the treated samples are shown in Table 3. When reanalyzed with the revised method, the total residues of sulfentrazone and its metabolites were generally higher in fodder and lower in forage.

TABLE 3- COMPARISON OF THE MAXIMUM RESIDUES (PPM) OF SULFENTRAZONE, DMS AND HMS IN/ON FIELD CORN MATRICES: PREVIOUSLY REPORTED VS. RE-ANALYSES WITH THE REVISED METHOD

Matrix	Trial #,		Sulfentrazone		DMS		HMS		Trial Total	
Study #	State	# Analyses	Previous	Revised	Previous	Revised	Previous	Revised	Previous	Revised
Forage										
162COF93R1	03, TN	2	ND[a]	ND	(0.012)	**0.034**	ND	**(0.007)**	(0.012)	**0.041**
162COF94R1	02, IA	2	(0.012)[b]	ND	0.031	**(0.008)**	ND	ND	0.043	**(0.008)**
162COF94R1	03, NE	2	ND	ND	(0.017)	**(0.009)**	(0.007)	ND	(0.024)	**(0.009)**
162COF94R1	04, IL	2	(0.006)	ND	0.054	**(0.016)**	ND	ND	0.054	**(0.016)**
162COF94R1	06, LA	2	(0.005)	ND	ND	ND	ND	ND	(0.005)	ND
162COF95R1	07,MN	2[c]	ND	ND	ND	**(0.009)**	ND	ND	ND	**(0.009)**
Fodder										
162COF93R1	03, TN	2	ND	ND	(0.020)	**0.055**	ND	**(0.005)**	(0.020)	**0.060**
162COF94R1	02, IA	2	ND	ND	ND	**(0.017)**	ND	ND	ND	**(0.017)**
162COF94R1	03, NE	2	ND	ND	ND	**(0.014)**	ND	ND	ND	**(0.014)**
162COF94R1	04, IL	2	ND	ND	ND	**(0.015)**	ND	ND	ND	**(0.015)**
162COF94R1	06, LA	2	ND	ND	(0.028)	**(0.013)**	ND	ND	(0.028)	**(0.013)**
162COF95R1	07,MN	2	ND	ND	(0.006)	**(0.009)**	(0.006)	ND	(0.012)	**(0.009)**
Grain										
162COF93R1	03, TN	2	ND	ND	ND	ND	ND	ND	ND	ND
162COF94R1	02, IA	2	ND	ND	ND	ND	ND	ND	ND	ND
162COF94R1	03, NE	2	ND	ND	ND	ND	ND	ND	ND	ND
162COF94R1	04, IL	2	ND	ND	ND	ND	ND	ND	ND	ND
162COF94R1	06, LA	2	ND	ND	ND	ND	ND	ND	ND	ND
162COF95R1	07,MN	2	ND	ND	ND	ND	ND	ND	ND	ND

a ND = Non-detectable (< 0.005 ppm, <0.01 ppm for 162COF94R1 fodder only).
b Values in parenthesis are estimates; less than the LOQ (0.025 ppm, 0.05 ppm for 162COF94R1 fodder), but greater than the LOD.
c Treated forage samples from 162COF95R1, trial #07 were analyzed in duplicate with the revised method.

Rice: Samples from six trials were reanalyzed with the new method. The method was validated in rice RACs at 0.025-0.50 ppm. The average recovery for sulfentrazone was 101 ± 9% (n=4); for 3-desmethyl sulfentrazone, 107 ± 23% (n=4); and for 3-hydroxymethyl sulfentrazone, 86 ± 7% (n=4). The maximum storage interval for the samples was 24 months. The results of reanalyses of the treated samples are shown in Table 4. When reanalyzed with the revised method, the total residues of sulfentrazone and its metabolites were generally 3-5X higher in straw and unchanged in grain.

TABLE 4- COMPARISON OF THE MAXIMUM RESIDUES (PPM) OF SULFENTRAZONE, DMS AND HMS IN/ON RICE MATRICES: PREVIOUSLY REPORTED VS. RE-ANALYSES WITH THE REVISED METHOD

Matrix	Trial #,		Sulfentrazone		DMS		HMS		Trial Total	
Study #	State	# Analyses	Previous	Revised	Previous	Revised	Previous	Revised	Previous	Revised
Grain										
162RIC94R1	02, AR	2	ND[a]	ND	ND	(0.009)	(0.007)	(0.013)	(0.007)	(0.022)
162RIC94R1	04, MS	2	ND	ND	(0.012)[b]	(0.021)	(0.020)	(0.014)	0.032	0.035
162RIC95R1	02, AR	2	ND	ND	(0.014)	0.028	0.029	(0.016)	0.043	0.044
162RIC95R1	06, LA	2	ND	ND	ND	(0.008)	(0.010)	(0.008)	(0.010)	(0.016)
162RIC95R1	07, MS	2	ND	ND	0.025	0.041	0.052	0.035	0.077	0.076
162RIC95R1	08, TX	2	ND	ND	(0.009)	(0.017)	(0.023)	(0.019)	0.032	0.036
Straw										
162RIC94R1	02, AR	2	ND	ND	(0.017)	0.078	(0.012)	(0.027)	(0.029)	0.105
162RIC94R1	04, MS	2	ND	ND	(0.045)	0.185	(0.021)	(0.028)	0.065	0.203
162RIC95R1	02, AR	2	ND	ND	(0.041)	0.183	(0.022)	0.056	0.063	0.237
162RIC95R1	06, LA	2	ND	ND	(0.013)	0.067	ND	(0.010)	(0.013)	0.077
162RIC95R1	07, MS	2	ND	ND	0.071	0.348	(0.012)	(0.017)	0.083	0.365
162RIC95R1	08, TX	2	ND	ND	(0.030)	0.142	(0.014)	(0.029)	(0.043)	0.171

a ND = Non-detectable (< 0.005 ppm for grain, <0.01 ppm for straw).

b Values in parentheses are estimates; less than the LOQ (0.025 ppm for grain, 0.05 ppm for straw), but greater than or equal to the LOD.

Sorghum: Samples from six trials were reanalyzed with the new method. The method was validated in sorghum RACs at 0.025-0.10 ppm. The average recovery for sulfentrazone was 89 ± 9% (n=6); for 3-desmethyl sulfentrazone, 81 ± 9% (n=6); and for 3-hydroxymethyl sulfentrazone, 88 ± 17% (n=6). The maximum storage interval for the samples was 13 months. The results of reanalyses of the treated samples are shown in Table 5. When reanalyzed with the revised method, the total residues of sulfentrazone and its metabolites were generally 2-6X higher in sorghum forage, and 1.5-3X higher in sorghum fodder.

TABLE 5- COMPARISON OF THE MAXIMUM RESIDUES (PPM) OF SULFENTRAZONE, DMS AND HMS IN/ON SORGHUM MATRICES: PREVIOUSLY REPORTED VS. RE-ANALYSES WITH THE REVISED METHOD

Matrix		Sulfentrazone		DMS		HMS		Trial Total	
Trial, State	# Analyses	Previous	Revised	Previous	Revised	Previous	Revised	Previous	Revised
Previous Study Number: **162SOR95R1**									
Forage									
01, VA	2	ND[a]	ND	(0.017)[b]	0.037	(0.010)	(0.008)	0.023	0.045
02, LA	2	ND	ND	ND	(0.013)	(0.008)	(0.012)	(0.008)	0.025
03, TX	2	ND	ND	(0.016)	0.047	(0.024)	0.041	0.038	0.084
04, TX	2	ND	ND	ND	(0.013)	(0.019)	0.034	(0.019)	0.047
06, NE	2	ND	ND	ND	(0.010)	(0.008)	0.037	(0.008)	0.047
07,KS	2	ND	ND	ND	(0.012)	ND	0.034	ND	0.046
Fodder									
01, VA	2	ND	ND	(0.012)	0.028	(0.006)	(0.012)	(0.018)	0.039
02, LA	2	ND	ND	(0.006)	0.026	(0.009)	(0.018)	(0.015)	0.044
03, TX	2	ND	ND	(0.022)	(0.016)	(0.023)	(0.020)	0.044	0.033
04, TX	2	(0.009)	ND	(0.012)	(0.018)	(0.021)	0.040	0.042	0.058
06, NE	2	ND	ND	ND	(0.012)	(0.018)	0.031	(0.018)	0.043
07,KS	2	ND	ND	(0.012)	(0.024)	(0.013)	(0.009)	0.025	0.034
Grain									
01, VA	2	ND	ND	ND	ND	ND	ND	ND	ND
02, LA	2	ND	ND	ND	ND	(0.006)	ND	(0.006)	ND
03, TX	2	ND	ND	(0.007)	(0.006)	ND	ND	(0.007)	(0.006)
04, TX	2	ND	ND	ND	ND	ND	(0.005)	ND	(0.005)
06, NE	2	ND	ND	ND	(0.007)	ND	ND	ND	(0.007)
07,KS	2	ND	ND	ND	ND	ND	ND	ND	ND

[a] ND = Non-detectable (< 0.005 ppm).

[b] Values in parenthesis are estimates; less than the LOQ (0.025 ppm), but greater than the LOD.

CBTS's Conclusion: Acceptable recoveries were obtained by the independent laboratory. The method and ILV will be sent to Beltsville for PMV. CBTS concludes that the revised method is adequate for data gathering purposes. A conclusion on the adequacy of the method for enforcement of the proposed tolerances will be withheld pending satisfactory Agency validation.

Samples from six trials for each crop were reanalyzed with the new method. When reanalyzed with the revised method, the total residues of sulfentrazone and its metabolites were generally 2-3X higher in soybean seed, 1.5-6X higher in cereal grain forage, 2-5X higher in cereal grain hay, 3-6X higher in cereal grain straw, 1.5-3X higher in cereal grain stover and unchanged in grain. Based on these reanalyses, the proposed tolerance level of 0.05 ppm for soybeans is adequate.

Residues of sulfentrazone and its metabolites have been shown to be stable during storage for up to 38 months is soybeans and for

up to 24 months in cereal grain RACs. The maximum storage intervals for the samples prior to reanalysis was 47 months for soybeans and 35 months for cereal grain RACs. CBTS is willing to extend the results of the storage stability studies to cover the actual storage intervals. Additional storage stability data are thus not required.

Total residues of sulfentrazone and its metabolites were found to concentrate in rice hulls (3.9X) and bran (1.9X) (Memo, G. Kramer 7/1/96). Based on these concentration factors and the current highest average field trial value for cereal grain (0.068 ppm, rice), the appropriate tolerances for cereal grain hulls and bran would be 0.15 ppm and 0.30 ppm, respectively. A final conclusion on the appropriate tolerances for these commodities will be made once the additional rice residue data (see below) and wheat processing study are submitted. A revised Section F will be required.

Deficiency - Conclusion 8b (from Memo, G. Kramer 7/1/96)

8b. Radiovalidation of the new enforcement method for soybeans and rotational crops will be required.

Petitioner's Response: Submission of:

Radiovalidation of Residue Methodology for Sulfentrazone, 3-Hydroxymethyl Sulfentrazone, and 3-Desmethyl Sulfentrazone in/on Barley Forage. MRID# 441188-0x.

The average residues of desmethyl sulfentrazone and hydroxymethyl sulfentrazone were 0.056 and 0.132 ppm, respectively, in barley forage when quantitated by GC/ELCD using the tolerance enforcement methodology. These residue values were 133% and 70% for desmethyl sulfentrazone and hydroxymethyl sulfentrazone, respectively, of the metabolism reanalysis values (0.042 and 0.188 ppm, respectively). No detectable (ND, < 0.005 ppm) residue of sulfentrazone was found using either method. The total residues of desmethyl sulfentrazone and hydroxymethyl sulfentrazone by the tolerance enforcement method (GC/ELCD, 0.188 ppm) were 81% of the metabolism reanalysis values obtained by liquid chromatograph (LC) and specific activity (0.232 ppm). These residue results indicate that similar amounts of desmethyl sulfentrazone and hydroxymethyl sulfentrazone residues in barley forage reported in the metabolism reanalysis were also found using the tolerance enforcement method.

CBTS's Conclusion: The revised method is capable of adequately recovering all analytes of concern. This deficiency is resolved.

Deficiency - Conclusion 10b (from Memo, G. Kramer 7/1/96)

10b. If residue data are submitted for grain sorghum, then data should also be provided for sorghum aspirated grain fractions as concentration of residues has been observed in the bran of another cereal grain (rice).

Petitioner's Response: Submission of a data waiver request. The petitioner contends that data for aspirated grain fractions are not required as residues were below the LOQ in all sorghum grain samples.

CBTS's Conclusion: According to the 860.1500 Guidelines, data for aspirated grain fractions are not required when residues are below the LOQ in grain samples only in situations where the pesticide is applied after the reproductive stage begins. For situations where the pesticide is applied prior to the reproductive stage, data for aspirated grain fractions are not required unless the processing study shows concentration of residues in the outer seed coat. Sulfentrazone residues have been shown to concentrate in rice bran and hulls. CBTS thus reiterates that data are required for sorghum aspirated grain fractions.

Deficiency - Conclusion 2 (from Memo, G. Kramer 7/31/96)

2. The petitioner previously submitted the results of nine rotational rice trials. This number does not correspond to that required for rice as a representative commodity of the cereal grains crop group. CBTS concludes that an additional four rice field trials are required.

Petitioner's Response: none.

CBTS's Conclusion: This deficiency remains outstanding.

Deficiency - Conclusions 3b, 4b, & 5b (from Memo, G. Kramer 7/31/96)

3b. A conclusion on the appropriate levels for permanent tolerances and MRLs will be withheld pending submission and review of additional data.

4b. Residue data are available for all representative commodities of the cereal grains (except sweet corn) crop group. A conclusion on the appropriate plantback restrictions for the permanent tolerances and the unconditional registrations will be withheld pending submission and review of additional data.

5b. However, a final conclusion on the appropriate levels for permanent tolerance will be withheld pending reanalysis of field residue samples with the new enforcement method.

Petitioner's Response: none.

CBTS's Conclusion: This deficiency remains outstanding. A conclusion on the appropriate plantback restrictions and on the levels for the permanent tolerances will be withheld pending submission and review of additional residue data and processing studies (rice field trials) and processing studies (sorghum aspirated grain fractions and wheat processing).

Deficiency - Conclusion 6 (from Memo, G. Kramer 7/31/96)

6. CBTS will reevaluate the need for a cow feeding study once the appropriate tolerance levels are determined for soybeans and rotational crops by reanalysis of field residue samples with the new enforcement method and submission of additional residue data and processing studies.

Petitioner's Response: Submission of a data waiver request.

CBTS's Conclusion: CBTS will reevaluate the need for a cow feeding study once the appropriate tolerance levels are determined for soybeans and rotational crops by submission of additional residue data and processing studies.

cc: PP#4F04407, Kramer, Circ., R.F., J. Miller/D. Morgan (RD, 7505C)
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