PP # 4407 4-3-95

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

APR 3 1995

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP# 4F04407. Sulfentrazone (Sulfentrazone 4F and 75DF Herbicide) for use on soybeans. Review of Residue Data and Analytical Methodology. MRID#s 433454-01 to -03, -14 to -23 and -28 to -32. Barcode D211168. Case 285935. CBTS#s 14993 & 15001.

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Tolerance Petition Section III
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Health Effects Division (7509C)

THRU: E. Zager, Acting Branch Chief
Chemistry Branch I, Tolerance Support
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TO: JoAnne Miller, Product Manager
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And

Jane Smith, Acting Section Head
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FMC has submitted an application 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-1H-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-1H-1,2,4-triazol-1-yl]phenyl] methanesulfonamide). The end use products, Sulfentrazone 4F and 75DF Herbicide, are to be registered for use on soybeans. To cover use on the primary crop, the petitioner has proposed the following tolerances (expressed as parent plus the metabolite 3-hydroxymethyl sulfentrazone):

Soybean Seed	--	0.05 ppm
Aspirated Grain Fractions	--	0.05 ppm

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

Wheat Forage	--	0.10 ppm
Wheat Straw	--	0.10 ppm
Wheat Grain	--	0.10 ppm
Corn Fodder	--	0.10 ppm
Corn Silage	--	0.10 ppm
Corn Grain	--	0.10 ppm

In review of a request for an EUP and temporary tolerances for sulfentrazone on soybeans (PP#3G4272), CBTS identified the deficiencies which must be addressed by the registrant in order for us to be able to recommend in favor of permanent tolerances (Memo, G. Kramer 4/25/94). In the Detailed Considerations section of this Memo, the outstanding deficiencies, listed as presented in the Memo of G. Kramer (4/25/94), are followed by the petitioner's response and our conclusions.

CONCLUSIONS

1. The following product chemistry data requirements remain outstanding: **a)** for GLN § 61-1, One of the impurities (I9) is incorrectly identified. This compound should be identified and the CSF revised to include its chemical name; **b)** for GLN § 62-1, Once commercial production is initiated, the five-batch analysis should be repeated and the CSF revised if necessary; **c)** for GLN § 62-3, The registrant should demonstrate the repeatability (precision), accuracy and linearity of Test Methods AGC 294, 295, 296 and 297 for each impurity measured by the respective method; **d)** for GLN § 62-3, CBTS notes that in the representative chromatogram included with Method AGC 294, Peaks 8, 9, 10 and 11 were listed as being unknowns. All of these peaks were larger than Peak 7 (FMC 119903), an impurity for which certified limits were required. The registrant should provide quantitative data for these compounds and, if present at a level of ≥0.1%, identify the impurity and revise the CSF as required; **e)** for GLN § 62-3, Several peaks were not labelled in the representative chromatogram included with Method AGC 295. The registrant should report whether these peaks are unknowns or are identified impurities accounted for in Method 294. If any are unknowns, then the registrant should provide quantitative data for these compounds and, if present at a level of ≥0.1%, identify the impurity and revise the CSF as required; and **f)** for GLN § 63-13, The registrant should report on the stability of the solid TGAI to sunlight.

2. The following deficiencies in the directions for use were noted: **a)** The proposed crop rotation restrictions are greater than 12 months

in some cases. CBTS considers such restrictions to be impractical in regards to reducing the possibility of residues in rotational crops. The maximum crop rotation interval CBTS will accept in regards to residues is 12 months. However, crop rotation restrictions of longer than 12 months may be retained if necessitated by problems with phytotoxicity. In this case, the label should state that the intervals in excess of 12 months are necessitated by phytotoxicity concerns. **b)** All rotational crops with plantback intervals of one year or less; except corn (10 months), wheat (winter- 4 months, spring 9 months) and soybeans (no restriction); should be removed from list of rotational crops (see conclusion 4b). **c)** No instructions in regards to adjuvant use were included. As adjuvants were not employed in the field residue trials, a label restriction prohibiting their use should be added. **d)** The registrant has not proposed tolerances for soybean forage and hay. Therefore, a label restriction prohibiting the feeding of treated forage and hay to livestock must be included on the label. **A revised Section B is required.**

3a. Sulfentrazone, radiochemically labelled in the phenyl ring or in the triazole ring, was applied to sandy loam soil at a rate of 0.5 lbs. ai/A (1.3X) in a greenhouse. Crops (lettuce, radishes and barley) were seeded 30, 122, 245 and 364 days after treatment (DAT) of the soil with sulfentrazone. The highest residue levels were seen in barley straw (2.98-3.36 ppm at 30 DAT and 0.67-1.83 at 364 DAT). The TRR in lettuce decreased from 0.44-0.65 ppm at 30 DAT to 0.03-0.12 at 364 DAT; the TRR in radish tops, from 0.72-0.87 ppm at 30 DAT to 0.04-0.14 at 364 DAT; the TRR in radish roots, from 0.31-0.34 ppm at 30 DAT to 0.06-0.14 at 364 DAT; the TRR in barley forage, from 1.41-2.07 ppm at 30 DAT to 0.22-0.49 at 364 DAT; and the TRR in barley grain, from 0.04-0.05 ppm at 30 DAT to 0.01-0.03 at 364 DAT. The major metabolites identified were 3-hydroxymethyl sulfentrazone, desmethyl des(difluoromethyl) sulfentrazone, sulfentrazone carboxylic acid/3-desmethyl sulfentrazone and methyl triazole (4-difluoromethyl-3-methyl-1H-1,2,4-triazol-5(4H)-one).

3b. The results of this confined crop rotation study demonstrate that quantifiable residues of 3-hydroxymethyl sulfentrazone are present in all crops planted 1 year after sulfentrazone application. This metabolite was also the major component of the residue identified in soybean seed (30-35% of the TRR). Limited field trials are thus required in order to determine whether rotational crop tolerances are needed and the appropriate plantback intervals.

3c. The nature of the residue in rotational crops can not be considered to be understood due to deficiencies in the characterization of bound residues. Minimal analysis was performed only on the 30 DAT samples. CBTS requests that registrant analyze the bound residues from the 364 DAT samples of barley straw (both phenyl- and triazole-labelled). The methods employed should include treatment with enzymes, surfactants, dilute acid and base and refluxing with 6 N acid and base.

4a. The registrant has submitted the results of five limited field rotational trials for corn and six for winter wheat. No quantifiable residues were observed in field corn so that rotational crop tolerances are not required for corn with a 10 month or greater plantback interval. However, quantifiable residues of 3-hydroxymethyl sulfentrazone were observed in winter wheat forage so that rotational crop tolerances are required for wheat. The required number of field trials required to set rotational crop tolerances is the same as that required to establish primary crop tolerances (i.e., 20 for wheat).

4b. The sulfentrazone label allows rotational barley, peanuts and rice to be planted at 12 months. However, limited field trials are required for these crops in order to determine whether rotational crop tolerances are required. If two limited trials are performed with barley or rice and no quantifiable residues are observed, then it will be concluded that rotational crop tolerances are not required for either crop. Until the required data for rotational barley, rice and peanuts are submitted, all plantback intervals of 1 year or less should be removed from the sulfentrazone label, except for soybeans, wheat and corn.

4c. The registrant has proposed rotational crop tolerances in/on wheat and corn RACs for residues of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone. These tolerances are not required for corn and should be withdrawn. **A revised Section F is required.** This conclusion is contingent on the ability of the registrant to demonstrate the storage stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone residues in corn RACs (see conclusion 12c).

4d. As noted above, tolerances are required for wheat grain, straw, forage and hay. CBTS is unable to comment on the adequacy of the proposed wheat tolerances until receipt and review of the requested residue data. If the wheat field residue data submitted with this petition are to be used for setting rotational crop tolerances, then the registrant must demonstrate the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in wheat RACs (see conclusion 12c). Also, the tolerance expression for wheat should be revised to incorporate the following language: "Tolerances are established for the indirect or inadvertent combined residues of..." Alternatively, the registrant may choose to withdraw the proposed tolerances for wheat RACs and include a prohibition against rotation to wheat on the sulfentrazone labels.

4e. CBTS generally requires that limited rotational crop field trials also be performed on representative root and leafy vegetable crops. As the label does not allow rotation to these crops, we will not require these trials for the sulfentrazone registration on soybeans. However, if the registrant should wish to add uses for sulfentrazone on other crops, then CBTS may require limited rotational crop field trials to be performed on representative root

and leafy vegetable crops.

5. The petitioner must address the following deficiencies in the soybean metabolism study (submitted with the EUP application): **i)** The storage stability of the samples in this study has not been demonstrated. The registrant should report the actual dates of extraction and chromatography. If the samples were stored longer than 6 months prior to analysis, then the registrant must show that the nature of the residue in the samples has not changed during storage by presenting representative chromatographic separations performed early in the study and at the conclusion of the study. If such data do not exist or if significant changes in the metabolite profile occurred during storage, the registrant may be required to repeat this metabolism study. **ii)** Unknown metabolites 2 (0.065-0.077 ppm in hay and 0.061-0.076 ppm in forage), 3 (0.105-0.110 in hay and 0.023-0.088 in forage), 5 (0.045-0.050 ppm in hay and 6 (up to 13.1% of the TRR in seed) accounted for significant portions of the TRR in soybean RACs. The registrant should identify these compounds. **iii)** Significant portions of the TRR in forage and grain were found to be extractable but were not characterized by HPLC (polar metabolites). The registrant should characterize any of these fractions which contain >0.05 ppm or >10% of the TRR (polar extracts of forage, triazole-labelled polar extract of hay and triazole-labelled polar extract of seed). **iv)** Significant portions of the bound residues of hay and forage remained uncharacterized after enzymatic digestions. The registrant should further characterize these bound residues. (Conclusion 4b from Memo, G. Kramer 4/25/94)

6a. Phenyl- and triazole-labelled sulfentrazone were administered orally to lactating goats. The goats were dosed at a rate of 4.9 ppm (phenyl) or 6.0 ppm (triazole). Doses were administered once daily for 10 consecutive days. Tissue and milk residues in excess of 0.01 ppm were observed only in phenyl-labelled kidney (0.013 ppm)

6b. Sulfentrazone *per se* was the predominant component of the kidney residue, accounting for 53.8% of the TRR. The metabolite 3-hydroxymethyl sulfentrazone (7.7% of the TRR) was also identified. 3-Hydroxymethyl sulfentrazone and sulfentrazone carboxylic acid were the major components of the urine and feces residue.

7a. Phenyl- and triazole-labelled sulfentrazone were administered orally to laying hens. The hens were dosed at a rate of 4.7 ppm. Doses were administered once daily for 12 consecutive days. Tissue and egg residues in excess of 0.01 ppm were observed only in egg yolk (0.014 ppm), egg white (0.018 ppm), kidney (0.03 ppm) and liver (0.014 ppm).

7b. Sulfentrazone *per se* was the predominant component of the residue of all samples analyzed, accounting for 27-70% of the TRR. The metabolites 3-hydroxymethyl sulfentrazone (14-33% of the TRR) and 3-desmethyl sulfentrazone (14% of the TRR in liver only) were also identified.

8. CBTS requires that the dietary dosing level for animal metabolism studies be at least 10 ppm. The dosing levels in both the ruminant and poultry studies were well below 10 ppm. However, CBTS will accept these studies since the dose was >10X the estimated maximum dietary burden, the studies were conducted over 10-12 days instead of the usual 3 days, and the majority of the TRR identified in tissues was sulfentrazone and 3-hydroxymethyl sulfentrazone. For any future petition which results in a higher dietary burden, CBTS may require that the poultry and/or ruminant metabolism studies be repeated. Also, if the registrant wishes to propose tolerances for soybean forage and hay, this conclusion will be reevaluated.

9. CBTS will refer to the Metabolism Committee on the toxicological significance of metabolites once the deficiencies associated with plant metabolism studies have been addressed. A decision by CBTS concerning which residues to regulate will then follow. If additional residues are determined to be of regulatory concern, then a revised Section F and additional field studies, analytical methodology, and storage stability data may be needed.

10a. Method P-2811M is the proposed enforcement method for soybeans. Macerated tissue is initially refluxed in acetone/0.25 N HCl. The sample is then cleaned-up using C-8 SPE and silica gel cartridge columns and derivatized with N,O-bis-(trimethylsilyl)-trifluoroacetamide (BSTFA), which converts hydroxymethyl-sulfentrazone to its trimethylsilyl derivative. Analysis was then performed on GC/EC with a Megabore column. This method simultaneously measures both sulfentrazone and hydroxymethyl-sulfentrazone.

10b. An ILV of this method was performed by ADPEN Laboratories. Acceptable recoveries were obtained by the laboratory for all analytes. The method and ILV have been sent to Beltsville for PMV (Memo, G. Kramer 2/16/95). CBTS will withhold a final conclusion on the adequacy of this method as an analytical enforcement method pending receipt of the PMV report.

10c. Reports on Multiresidue testing for both sulfentrazone and 3-hydroxymethyl sulfentrazone have been received and forwarded to FDA (Memo, G. Kramer 2/7/95). Neither compound was recovered by any of the protocols.

10d. A sample from the plant metabolism study was subjected only to the initial hydrolysis step of the proposed enforcement method (see conclusion 10a). Of the TRR, 96% was solubilized by this method. As the conjugated residues were shown to be released by acid hydrolysis with 1 N HCl in the plant metabolism study, CBTS can reach no conclusion on whether conjugated residues are released by the proposed enforcement method. Radiovalidation should be performed by running the entire method on samples from the plant metabolism study.

10e. The registrant has included conditions for use of a MSD

detector in order to confirm the identity of the analytes.

10f. If tolerances are proposed for soybean forage and hay, then enforcement methodology which measures all residues of concern in these RACs will be required. If substantially different from the current method, then the registrant should obtain an ILV for this method(s).

10g. The analytical methods used for rotational wheat RACs differed significantly from the proposed enforcement method for soybeans. CBTS thus requests that these methods be validated by an independent laboratory. Once we receive the ILV, the method will be forwarded to ACL for Agency validation.

11. No analytical method for meat, milk and eggs has been submitted by the registrant. Since no tolerances have been proposed for animal RACs, an analytical enforcement method for animals is not required at this time. If, however, the required ruminant feeding studies (see conclusion 15) demonstrate a potential for transfer of residues to meat or milk, then the registrant will be required to propose tolerances for these RACs and develop the appropriate analytical enforcement methodology. Any required enforcement methods for meat and milk will need successful ILVs and PMVs before being judged to be acceptable by CBTS.

12a. The soybean seed samples from the field residue studies were stored for a maximum of 9 months. The corn silage samples from the limited crop rotation field residue studies were stored for a maximum of 10 months; the corn grain samples, 9 months; the corn fodder samples, 9 months; the wheat grain samples, 14 months; the wheat forage samples, 20 months; and the wheat straw samples, 13 months.

12b. The registrant has demonstrated that residues of sulfentrazone are stable in soybean seed for up to 24 months of frozen storage; and for 3-hydroxymethyl sulfentrazone, 11 months. These results demonstrate storage stability for the purposes of the primary crop field trials.

12c. No data have been supplied on the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in corn and wheat RACs. The registrant must demonstrate the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in corn forage or silage samples for at least 10 months of storage; and fodder samples, for 9 months. The registrant must also demonstrate the stability of 3-desmethyl sulfentrazone in corn grain samples for at least 9 months of storage. The soybean storage stability data for sulfentrazone and 3-hydroxymethyl sulfentrazone can be translated to corn grain. If the wheat field residue data submitted with this petition are to be used for setting rotational crop tolerances, then the registrant must demonstrate the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in wheat forage samples for at least 20 months of

storage; wheat grain samples, for 14 months; and wheat straw samples, for 13 months.

13a. A total 15 field trials were conducted in 10 states located in Regions 2 (3 trials), 4 (5 trials), and 5 (7 trials). The application rate of sulfentrazone 75DF was 0.375 lbs. ai/A (1X) in 12 trials and 0.5 lbs. ai/A (1.3X) in three trials. Preplant incorporation was employed in five trials, preemergence application in six trials and both methods were employed in separate subplots in four trials. Mature soybean seeds were harvested 115-167 days after planting. Seeds were analyzed using Method P-2811M. Residues of sulfentrazone were below the LOD (0.005 ppm) and residues of hydroxymethyl-sulfentrazone were below the LOQ (0.025 ppm) in all samples. Detectable residues of 3-hydroxymethyl sulfentrazone were observed in five trials (maximum residue = 0.022 ppm).

13b. The registrant previously submitted the results of seven acceptable soybean trials which employed the 4F formulation (Memo G. Kramer 4/25/94). Residues of sulfentrazone were below the LOD and residues of 3-hydroxymethyl sulfentrazone were below the LOQ in all samples. Detectable residues of 3-hydroxymethyl sulfentrazone were observed in three trials. Together with the residue data submitted with this petition, the registrant has provided the results of 22 soybean trials, conducted in Regions 2 (5 trials), 4 (6 trials), and 5 (11 trials). These results support the proposed tolerance of 0.05 ppm for the combined residues of sulfentrazone and 3-hydroxymethyl sulfentrazone on soybean seed.

14a. A soybean processing study was reviewed in conjunction with the EUP (Memo, G. Kramer 9/1/94). This processing study was determined to be adequate for the permanent tolerance petition provided that storage stability of 3-hydroxymethyl sulfentrazone was demonstrated. The storage interval for the RAC samples, 5 months, is represented in the storage stability study submitted with this petition. The processed commodities were analyzed within 30 days of preparation. Storage stability has thus been demonstrated for the purposes of the processing study.

14b. Sulfentrazone and 3-hydroxymethyl sulfentrazone residues do not concentrate in processed commodities. Feed/feed additive tolerances for sulfentrazone and 3-hydroxymethyl sulfentrazone are thus not required for this petition. If, however, metabolites other than 3-hydroxymethyl sulfentrazone are determined to be of regulatory significance, then residue data for soybean processed fractions will be required for all such metabolites.

14c. CBTS has determined that a tolerance for aspirated grain fractions is not required as the observed residues in 'grain dust' appear to be the result of soil contamination (Memo, G. Kramer 9/1/94). The proposed tolerance for aspirated grain fractions should thus be withdrawn.

15. The maximum theoretical dietary burden for ruminants associated with soybeans is 0.011 ppm. Based on the results of the ruminant metabolism study, the registrant claims that a conventional feeding study is not required. The dietary burden used in the phenyl-labelled study, 4.9 ppm, is an exaggeration of 445X based on a maximum dietary burden of 0.011 ppm. The maximum tissue residue observed at this level was 0.013 ppm in kidney. However, CBTS has concluded that rotational crop tolerances are required to support rotation to wheat with a 4 month plantback interval. Based on the limited residue data, the theoretical maximum dietary burden associated with rotational wheat would be at least 0.30 ppm. Considering this dietary burden, the ruminant metabolism feeding level represented only a 16X exaggerated rate. CBTS thus concludes that a conventional ruminant feeding study will be required in order to support the establishment of rotational crop tolerances on wheat RACs. Alternatively, the registrant may choose to withdraw the proposed tolerances for wheat RACs and include a prohibition against rotation to wheat on the sulfentrazone labels. Due to the minimal transfer of residues at 445X, CBTS concludes that, based on the soybean seed tolerance only, a conventional feeding study is not required. If in the future, the registrant wishes to propose tolerances for soybean forage and hay, this conclusion will be reevaluated.

16. The maximum theoretical dietary burden for poultry associated with soybeans is 0.02 ppm. Based on the results of the poultry metabolism study, the registrant claims that a conventional feeding study is not required. The dietary burden used in the metabolism studies, 4.7 ppm, is an exaggeration of 235X based on a maximum dietary burden of 0.02 ppm. The maximum tissue residue observed at this level was 0.03 ppm in kidney. However, CBTS has concluded that rotational crop tolerances are required to support rotation to wheat with a 4 month plantback interval. Based on the limited residue data, the theoretical maximum dietary burden associated with rotational wheat would be at least 0.09 ppm. Considering this dietary burden, the feeding level in the poultry metabolism study represented a 52X exaggerated rate. CBTS concludes that a conventional feeding study is not required. However, for any future petition which results in a higher dietary burden, CBTS may require that a poultry feeding study be performed.

17. There is no Codex proposal, nor Canadian or Mexican limits for residues of sulfentrazone and its metabolites in soybeans, wheat or corn. Therefore, a compatibility issue is not relevant to the proposed tolerance. A copy of the IRLS is attached to the memorandum.

RECOMMENDATIONS

CBTS recommends against the proposed tolerances for residues of sulfentrazone and 3-hydroxymethyl sulfentrazone on soybeans and for residues of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone on corn and wheat RACs for reasons detailed in conclusions 1a-f, 2a-d, 3c, 4b-d, 5, 9, 10b, 10d, 10g, 11, 12c, 14b, 14c and 15. A DRES run can (for soybeans only) be initiated at this time.

DETAILED CONSIDERATIONS

Product Chemistry

Deficiency - Conclusion 1a (from Memo, G. Kramer 4/25/94)

1a) for GLN § 61-1: The CSF for the TGAI lists 2.9% as "unknown." Inspection of the chromatographic analysis of the TGAI submitted in conjunction with § 62-3 reveals the presence of at least three significant (>0.1%) impurities which were not identified. Any impurities in the unknown material found at a level above 0.1% must be identified and included in the CSF. Also, the a.i. is not listed under its ANSI systemic name. A revised CSF for the TGAI is thus required.

Petitioner's Response: A revised CSF is included in MRID# 433454-01. See the confidential appendix for details.

CBTS' Conclusion: One of the impurities (I9) is incorrectly identified. This compound should be identified and the CSF revised to include its chemical name.

Deficiency - Conclusion 1b (from Memo, G. Kramer 4/25/94)

1b) for GLN § 61-3, the registrant should identify any impurities in the "unknown" component of the TGAI and discuss their formation, discuss the formation of impurities that might hypothetically occur but were not found in the TGAI, discuss possible degradation products of the TGAI and discuss the potential for starting materials to carry over to the TGAI.

Petitioner's Response: Further discussion of impurities was included in MRID# 433454-01. See the confidential appendix for details.

CBTS' Conclusion: The requested information has been provided. This deficiency is now resolved.

Deficiency - Conclusion 1c (from Memo, G. Kramer 4/25/94)

1c) for GLN § 62-1: The registrant has submitted data from the analysis of only one batch of the TGAI produced by the pilot plant. The registrant should report the results of five batch analyses of sulfentrazone TGAI once it goes into market production. The CSF may need to be revised if the results of the new batch analyses differ from that done previously.

Petitioner's Response: The results of a five-batch analysis were included in MRID# 433454-02. See the confidential appendix for details.

CBTS' Conclusion: The requested information has been provided. This deficiency is now resolved. Once commercial production is initiated, the five-batch analysis should be repeated and the CSF revised if necessary.

Deficiency - Conclusion 1d (from Memo, G. Kramer 4/25/94)

1d) for GLN § 62-2, the registrant should provide a CSF for the TGAI in which all impurities >0.1% are identified and the certified limits are based on the analysis of at least five independent batches.

Petitioner's Response: The results of a five-batch analysis were included in MRID# 433454-02. The certified limits are based on the results of this analysis. See the confidential appendix for details.

CBTS' Conclusion: The requested information has been provided. This deficiency is now resolved.

Deficiency - Conclusion 1e (from Memo, G. Kramer 4/25/94)

1e) for GLN § 62-3, the registrant should demonstrate the repeatability (precision) of the analytical method for the a.i. by reporting the results of at least five determinations of a single sample of the TGAI, and the accuracy of the method performing at least five determinations of the sulfentrazone analytical grade standard.

Petitioner's Response: The registrant has submitted a nonconfidential method, Test Method AGC 274, for determination of the a.i. in the TGAI (MRID# 433454-02). This method involves normal phase HPLC using isocratic elution from a Zorbax-Sil column and UV detection at 280 nm. Validation data were not included.

CBTS' Conclusion: The registrant should demonstrate the repeatability (precision), accuracy and linearity of Test Method AGC 274.

Deficiency - Conclusion 1f (from Memo, G. Kramer 4/25/94)

1f) for GLN § 62-3: The reported precision for one impurity which has a nominal concentration in the 1.0-10.0% range was 24.9%. As the maximum acceptable precision for an impurity in this concentration range is 5%, CBTS concludes that this method is not adequate to enforce the certified limits of the known impurities in the TGAI. The registrant should develop an acceptable analytical method for the identified impurities and demonstrate the repeatability (precision) of the method by reporting the results of at least five determinations of a single sample of the TGAI. The accuracy of the method should also be determined by performing at least five determinations of the analytical grade standards. If other impurities are found at levels >0.1% in the "unknown" component of the TGAI, then validated analytical methods will be required for all such impurities.

Petitioner's Response: The registrant has submitted the following nonconfidential methods for determination of the impurities in the TGAI (MRID# 433454-02):

Test Method AGC 294- This method involves GC using a crossbonded 80% dimethyl-20% diphenyl polysiloxane column and FID detection. This method was used to measure impurities FMC 125117, FMC 114391, FMC 97267, I9, I10, FMC 125175, FMC 122048, and FMC 119903. Validation data were not included.

Test Method AGC 295- This method involves HPLC using a bonded, deactivated C-18 column with gradient elution and UV detection at 230 nm. This method was used to measure impurities FMC 97283, FMC 114391, FMC 97267, FMC 122048, and FMC 114392. In cases where impurities were measured with two different methods, average values were calculated. Validation data were not included.

Test Method AGC 296- This method involves GC using a crossbonded 80% dimethyl-20% diphenyl polysiloxane column and FID detection. This method was used to measure toluene. Linearity was demonstrated over a range of 0.7-1.5 mg/g. Other validation data were not included.

Test Method AGC 297- This method involves gel permeation chromatography to measure "tar" impurities >600 MW. Validation data were not included.

CBTS' Conclusion: The registrant should demonstrate the repeatability (precision), accuracy and linearity of Test Methods AGC 294, 295, 296 and 297 for each impurity measured by the respective method. CBTS also notes that in the representative chromatogram included with Method AGC 294, Peaks 8, 9, 10 and 11 were listed as being unknowns. All of these peaks were larger than Peak 7 (FMC 119903), an impurity for which certified limits were required. The registrant should provide quantitative data for these compounds and, if present at a level of ≥0.1%, identify the impurity and revise the CSF as required. Several peaks were also not labelled in the representative chromatogram included with Method AGC 295. The registrant should report whether these peaks are unknowns or are identified impurities accounted for in Method 294. If any are

unknowns, then the registrant should provide quantitative data for these compounds and, if present at a level of ≥0.1%, identify the impurity and revise the CSF as required.

Deficiency - Conclusion 1g (from Memo, G. Kramer 4/25/94)

1g) for GLN § 63-5, submit data on the melting point of the TGAI.

Petitioner's Response: Using a Fisher-Johns Apparatus, the melting point of sulfentrazone TGAI was determined to be 120-122 °C (MRID# 433454-03).

CBTS' Conclusion: The requested information has been provided. This deficiency is now resolved.

Deficiency - Conclusion 1h (from Memo, G. Kramer 4/25/94)

1h) for GLN § 63-8, submit data on the solubility of the TGAI in nonpolar solvents.

Petitioner's Response: The solubility of the TGAI in organic solvents was determined by the shake flask method (MRID# 433454-03):

Solvent	Solubility, % w/w
Acetone	64
Acetonitrile	18.6
Toluene	0.66
Hexane	0.01

CBTS' Conclusion: The requested information has been provided. This deficiency is now resolved.

Deficiency - Conclusion 1i (from Memo, G. Kramer 4/25/94)

1i) for GLN § 63-13, submit data on the sensitivity of the TGAI to metals, metal ions, elevated temperature and sunlight.

Petitioner's Response: The registrant exposed the solid TGAI to aluminum, stainless steel, ferric sulfate, manganous sulfate and cupric sulfate for 10 days at room temperature and 50 °C (MRID# 433454-03). No evidence of instability was observed at room temperature. At the elevated temperature, instability was observed only in the presence of ferric sulfate (≈4% decrease in sulfentrazone content). The sensitivity to sunlight was not investigated, but

sulfentrazone has been found to be unstable in aqueous photolysis studies.

CBTS' Conclusion: The registrant has not reported on the stability of the solid TGAI to sunlight. **This deficiency remains outstanding.**

Table 1- PRODUCT CHEMISTRY DATA SUMMARY
Chemical No. 129081
Product: Sulfentrazone TGAI

Guideline Number	Requirement	Are Data Requirements Fulfilled?[a]	MRID Number
61-1	Product Identity and Disclosure of Ingredients	N[b]	433454-01
61-2	Beginning Materials and Manufacturing Process	Y	433454-01
61-3	Discussion of Formation of Impurities	Y	433454-01
62-1	Preliminary Analysis	N[b]	433454-02
62-2	Certification of Ingredient Limits	N[c]	433454-02
62-3	Analytical Methods to Verify the Certified Limits	N[d]	433454-02
63-2	Color	Y	419116-03
63-3	Physical State	Y	419116-03
63-4	Odor	Y	419116-03
63-5	Melting Point	Y	433454-03
63-6	Boiling Point	N/A	
63-7	Density, Bulk Density or Specific Gravity	Y	419116-03
63-8	Solubility	Y	419116-03 433454-03
63-9	Vapor Pressure	Y	419116-03
63-10	Dissociation Constant	Y	419116-03
63-11	Octanol/Water Partition Coefficient	Y	419116-03
63-12	pH	Y	419116-03
63-13	Stability	N[e]	433454-03

[a] Y = Yes; N = No; N/A = Not Applicable.
[b] Identification of additional impurities and revised CSF required.
[c] CSF required for the TGAI in which all impurities >0.1% are identified.
[d] Validation data for all methods required.
[e] Data required on the sensitivity of the solid TGAI to sunlight.

Formulation: Sulfentrazone is formulated as Sulfentrazone (F6285) 4F Herbicide, containing 39.6% a.i. by weight and 4 lbs. a.i./gal. and as Sulfentrazone (F6285) 75DF Herbicide, containing 75.0% a.i. by weight.

Proposed Use

Sulfentrazone is applied preemergence, at-plant or preplant soil incorporated (PPI) by ground equipment in a volume of 10-40 gal/A. The application rate is 0.3125-0.375 lbs. ai/A and only one application may be made per season.

The label contains the following rotational crop restrictions: winter wheat, 4 months; spring wheat, 9 months; field corn, 10 months; barley, peanuts, rice and tobacco, 12 months; canola, corn (pop, seed and sweet), cotton and sorghum, 18 months; all other crops, 24 months.

Sulfentrazone may be tank-mixed with Command, Commence, Dual, Frontier, Lasso, Prowl, Sonalan and trifluralin-containing products. Tolerances on soybeans are established for the a.i.s of all of these products.

The following deficiencies in the directions for use were noted: **a)** The proposed crop rotation restrictions are greater than 12 months in some cases. CBTS considers such restrictions to be impractical in regards to reducing the possibility of residues in rotational crops. The maximum crop rotation interval CBTS will accept in regards to residues is 12 months. However, crop rotation restrictions of longer than 12 months may be retained if necessitated by problems with phytotoxicity. In this case, the label should state that the intervals in excess of 12 months are necessitated by phytotoxicity concerns. **b)** All rotational crops with plantback intervals of one year or less; except corn, wheat and soybeans; should be removed from list of rotational crops. **c)** No instructions in regards to adjuvant use were included. As adjuvants were not employed in the field residue trials, a label restriction prohibiting their use should be added. **d)** The registrant has not proposed tolerances for soybean forage and hay. Therefore, a label restriction prohibiting the feeding of treated forage and hay to livestock must be included on the label. **A revised Section B is required.**

Rotational Crop Studies: GLN § 165-1

Deficiency - Conclusion 3 (from Memo, G. Kramer 4/25/94)

3)No rotational crop studies were submitted with this petition. For the purposes of this EUP, crop rotation may be restricted by a label amendment (see above). However, for the permanent tolerance the registrant must submit a confined crop rotation study. The results of this study will be used to determine the appropriate crop rotation restrictions and/or the need for limited field trials.

Submitted with this petition:

Confined Accumulation Studies on Rotational Crops: F6285 Herbicide in Barley, Lettuce and Radish. MRID# 433454-28

In-Life Phase: Sulfentrazone, radiochemically labelled in the aromatic ring (phenyl-UL-^{14}C) or in the triazole ring (carbonyl-^{14}C), was diluted to a specific activity of 5.59 mci/mmol (phenyl) or 5.26 mci/mmol (triazole) and applied to sandy loam soil at a rate of 0.5 lbs. ai/A (1.3X) in a greenhouse. Crops (lettuce, radishes and barley) were seeded 30, 122, 245 and 364 days after treatment (DAT) of the soil with sulfentrazone. All crops were harvested when mature. Immature samples of barley (forage) were also taken.

Crop Residue Quantitation: Crop residues as determined by combustion are shown in Table 2. The highest residue levels were seen in barley straw (2.98-3.36 ppm at 30 DAT and 0.67-1.83 at 364 DAT). The TRR in lettuce decreased from 0.65-0.44 ppm at 30 DAT to 0.03-0.12 at 364 DAT; the TRR in radish tops, from 0.72-0.87 ppm at 30 DAT to 0.04-0.14 at 364 DAT; the TRR in radish roots, from 0.31-0.34 ppm at 30 DAT to 0.06-0.14 at 364 DAT; the TRR in barley forage, from 2.07-1.41 ppm at 30 DAT to 0.22-0.49 at 364 DAT; and the TRR in barley grain, from 0.04-0.05 ppm at 30 DAT to 0.01-0.03 at 364 DAT.

Extraction and Fractionation: Plant samples were homogenized in 1:1 mixture of methanol:water. The methanol:water extracts were further fractionated by the following procedures: Method 1- Extracts of 30 and 122 DAT radish, lettuce and barley forage were partitioned 3X with methylene chloride. The methylene chloride fraction (organosoluble) was concentrated for HPLC analysis. The aqueous fraction was hydrolyzed by refluxing in 1 N HCl. The refluxate was applied to a C-18 SPE column. Residues were eluted with methanol (polar fraction) and water (highly polar fraction). Method 2- Extracts of 30 and 122 DAT mature barley and all 245 and 364 DAT crops were concentrated and applied to a C-18 SPE column. Residues were eluted with methanol and water. Both eluates were hydrolyzed by refluxing in 1 N HCl. The methanol-soluble refluxate was applied to a C-18 SPE column. Residues were eluted with methanol (organosoluble fraction) and water (polar fraction). The aqueous-soluble refluxate was also applied to a C-18 SPE column. Residues were eluted with methanol (polar fraction) and water (highly polar fraction). The total residue of all extracts was thus partitioned into three fractions: organosoluble, polar and highly polar (Tables 3 & 4). High relative levels of bound residues (>10% of the TRR) were observed in radish root, radish tops (364 DAT), lettuce, barley forage (after 30 DAT), barley straw and barley grain.

Table 2- TRR in rotational crops as a result of application of phenyl- or triazole-labelled sulfentrazone to soil at a rate of 0.5 lbs. ai/A. The result are the average of two replicates.

DAT	Crop	RAC	Crop Age* (days)	Label Position (TRR, ppm)	
				Phenyl	Triazole
30	Radish	Top	50	0.716	0.868
		Root	50	0.312	0.343
	Lettuce	Leaf	50	0.651	0.440
	Barley	Forage	50	1.406	2.067
		Straw	243	2.984	3.362
		Grain	243	0.052	0.041
122	Radish	Top	72	0.099	0.132
		Root	72	0.066	0.063
	Lettuce	Leaf	72	0.194	0.110
	Barley	Forage	72	0.350	0.595
		Straw	214	2.725	4.264
		Grain	214	0.035	0.054
245	Radish	Top	74	0.176	0.162
		Root	74	0.044	0.047
	Lettuce	Leaf	74	0.044	0.034
	Barley	Forage	74	0.475	0.329
		Straw	154	1.060	1.705
		Grain	154	0.014	0.035
364	Radish	Top	62	0.042	0.143
		Root	62	0.058	0.139
	Lettuce	Leaf	62	0.115	0.030
	Barley	Forage	62	0.219	0.494
		Straw	236	0.673	1.831
		Grain	236	0.012	0.031

*Age of RAC when harvested

Table 3- Fractioation of TRR in rotational radish and lettuce resulting from application of phenyl- or triazole-labelled sulfentrazone to soil. The result are the average of two replicates.

DAT	RAC	Label	Organosoluble		Polar		Highly Polar		Bound	
			ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
30	Radish Top	Phenyl	0.179	24.9	0.451	63.1	0.004	0.6	0.082	11.5
		Triazole	0.205	24.0	0.446	51.1	0.147	16.8	0.070	8.1
	Radish Root	Phenyl	0.115	37.8	0.066	20.6	0.000	0.0	0.131	41.6
		Triazole	0.122	35.3	0.060	17.7	0.039	11.2	0.122	35.8
	Lettuce	Phenyl	0.116	17.7	0.342	52.4	0.000	0.0	0.193	29.9
		Triazole	0.068	15.3	0.165	37.5	0.077	17.5	0.130	29.7
122	Radish Top	Phenyl	0.048	48.7	0.041	41.9	0.001	1.3	0.008	8.1
		Triazole	0.054	41.5	0.038	27.7	0.034	26.0	0.006	4.8
	Radish Root	Phenyl	0.026	38.8	0.008	11.8	0.002	2.4	0.031	47.1
		Triazole	0.025	43.1	0.004	7.1	0.009	12.7	0.023	37.2
	Lettuce	Phenyl	0.048	24.9	0.104	53.7	0.001	0.7	0.040	20.7
		Triazole	0.019	16.9	0.051	46.8	0.020	18.7	0.019	17.6
245	Radish Top	Phenyl	0.166	94.2	0.002	0.8	0.000	0.0	0.009	4.9
		Triazole	0.108	67.6	0.031	18.4	0.016	10.2	0.006	3.8
	Radish Root	Phenyl	0.028	62.8	0.001	2.1	0.000	0.0	0.016	35.1
		Triazole	0.026	56.3	0.007	15.3	0.000	0.0	0.013	28.5
	Lettuce	Phenyl	0.024	57.5	0.002	4.7	0.000	0.0	0.017	37.8
		Triazole	0.018	54.0	0.001	3.9	0.007	19.4	0.008	22.7
364	Radish Top	Phenyl	0.035	82.8	0.000	0.3	0.000	0.0	0.007	17.5
		Triazole	0.100	70.0	0.008	5.7	0.017	12.2	0.017	12.1
	Radish Root	Phenyl	0.014	24.8	0.000	0.2	0.000	0.0	0.044	75.1
		Triazole	0.030	21.7	0.002	1.2	0.008	5.6	0.099	71.6
	Lettuce	Phenyl	0.077	67.1	0.000	0.1	0.000	0.0	0.038	32.7
		Triazole	0.013	44.7	0.003	10.7	0.005	18.3	0.008	26.4

Table 4- Fractioation of TRR in rotational barley resulting from application of phenyl- or triazole-labelled sulfentrazone to soil. The result are the average of two replicates.

DAT	RAC	Label	Organosoluble		Polar		Highly Polar		Bound	
			ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
30	Barley Forage	Phenyl	0.575	41.0	0.690	49.0	0.007	0.5	0.133	9.5
		Triazole	0.610	29.5	0.717	34.7	0.616	29.8	0.124	6.0
	Barley Straw	Phenyl	2.339	78.4	0.051	1.7	0.046	1.5	0.548	18.3
		Triazole	1.545	45.7	0.238	7.1	1.186	35.6	0.393	11.7
	Barley Grain	Phenyl	0.020	37.8	0.001	2.2	0.002	4.5	0.025	47.4
		Triazole	0.009	21.5	0.002	5.4	0.014	34.6	0.011	28.4
122	Barley Forage	Phenyl	0.131	36.9	0.163	47.2	0.002	0.5	0.054	15.4
		Triazole	0.205	34.4	0.200	33.7	0.140	23.6	0.050	8.4
	Barley Straw	Phenyl	1.508	55.3	0.457	16.8	0.031	1.1	0.686	25.2
		Triazole	1.321	30.3	1.099	26.4	1.267	29.9	0.529	12.4
	Barley Grain	Phenyl	0.014	40.2	0.005	15.4	0.000	0.0	0.013	37.7
		Triazole	0.018	34.3	0.002	2.7	0.019	35.3	0.013	24.2
245	Barley Forage	Phenyl	0.381	80.4	0.010	2.1	0.000	0.0	0.083	17.5
		Triazole	0.207	62.7	0.023	7.1	0.062	18.8	0.038	11.5
	Barley Straw	Phenyl	0.669	63.1	0.065	6.1	0.021	2.0	0.305	28.8
		Triazole	0.488	28.6	0.069	4.1	0.728	42.7	0.420	24.6
	Barley Grain	Phenyl	0.007	52.8	0.000	0.0	0.000	0.0	0.007	47.2
		Triazole	0.011	30.0	0.007	19.3	0.010	27.6	0.008	23.0
364	Barley Forage	Phenyl	0.177	81.4	0.006	2.7	0.002	1.2	0.032	14.7
		Triazole	0.297	60.0	0.049	9.9	0.099	20.2	0.049	10.0
	Barley Straw	Phenyl	0.403	59.9	0.022	3.1	0.026	3.7	0.224	33.3
		Triazole	0.808	43.8	0.133	7.1	0.237	19.2	0.547	29.9
	Barley Grain	Phenyl	-		-		-		-	
		Triazole	-		-		-		-	

- = Not Analyzed

Nature of the Residue: The soluble residues were analyzed by HPLC and the retention times compared with that of sulfentrazone *per se* and standards of possible metabolites (fig. 1, copied from 42 of MRID# 433454-28). Sulfentrazone carboxylic acid and 3-desmethyl sulfentrazone were not determined separately since sulfentrazone

carboxylic acid is decarboxylated to form 3-desmethyl sulfentrazone during acid hydrolysis. The identity of all metabolites found in these samples was confirmed by TLC and GC/MS or NMR.

Nature of the Residue in Radish Tops: The results of HPLC fractionation of triazole- and phenyl-labelled radish top extracts are shown in Tables 5 and 6. 3-Hydroxymethyl sulfentrazone was the major metabolite identified, accounting for 28-37% of the TRR at 30 DAT and 30-49% at 364 DAT. Other major metabolites included desmethyl des(difluoromethyl) sulfentrazone, accounting for 28-37% of the TRR at 30 DAT and 1-4% at 364 DAT, and methyl triazole (4-difluoromethyl-3-methyl-1H-1,2,4-triazol-5(4H)-one), accounting for 16% of the TRR at 30 DAT and 20% at 364 DAT. Minor metabolites identified included sulfentrazone *per se*, sulfentrazone carboxylic acid/3-desmethyl sulfentrazone, and des(methylsulfonyl) sulfentrazone. Unidentified peaks accounted for up to 12.3% of the TRR.

Nature of the Residue in Radish Root: The results of HPLC fractionation of triazole- and phenyl-labelled radish root extracts are shown in Tables 5 and 6. 3-Hydroxymethyl sulfentrazone was the major metabolite identified, accounting for 21-27% of the TRR at 30 DAT and 7-8% at 364 DAT. Other major metabolites included sulfentrazone *per se*, accounting for 11-12% of the TRR at 30 DAT and 2-4% at 364 DAT. Minor metabolites identified included desmethyl des(difluoromethyl) sulfentrazone, sulfentrazone carboxylic acid/3-desmethyl sulfentrazone, methyl triazole and des(methylsulfonyl) sulfentrazone. Unidentified peaks accounted for up to 4.6% of the TRR.

Table 5- Metabolite identification of phenyl-labelled radish residues.

Metabolite	30 DAT		122 DAT		245 DAT		364 DAT	
	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
Radish Tops								
HMS	0.266	37.2	0.032	32.8	0.091	51.6	0.020	48.5
DDS	0.262	36.6	0.029	29.7	0.032	18.3	0.000	0.6
SCA/DMS	0.029	4.1	0.004	3.6	0.018	10.0	0.005	11.5
Sultentrazone	0.009	1.2	0.007	6.8	0.010	5.8	0.003	8.0
DMSS	0.006	0.9	0.002	2.3	-		0.001	2.4
Unknown 30-33	0.013	1.9	0.001	1.2	-		-	
Bound[1]	0.050	7.0	0.008	8.1	0.009	4.9	0.007	17.5
Unassigned[2]	0.081	11.3	0.009	9.1	0.017	9.4	0.005	11.5
Radish Root								
HMS	0.084	26.8	0.010	15.3	0.014	32.5	0.005	8.1
DDS	0.036	11.5	0.004	5.3	0.001	1.7	0.000	0.2
SCA/DMS	0.023	7.2	0.006	8.2	0.004	8.9	0.005	8.0
Sultentrazone	0.033	10.6	0.007	11.1	0.006	14.1	0.002	3.7
DMSS	0.008	2.4	0.002	2.6	0.001	2.3	0.001	1.6
Bound[1]	0.108	34.7	0.031	47.1	0.016	35.1	0.044	75.1
Unassigned[2]	0.020	6.3	0.006	8.5	0.002	5.4	0.002	3.4

[1] 30 DAT samples after acid hydrolysis, [2]Total of unknown HPLC peaks and unanalyzed fractions
- = Not detected
SCA = Sulfentrazone Carboxylic Acid
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DMSS = Des-MethylSulfonyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone

Table 6- Metabolite identification of triazole-labelled radish residues.

Metabolite	30 DAT		122 DAT		245 DAT		364 DAT	
	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
Radish Tops								
HMS	0.240	27.6	0.032	24.6	0.052	31.8	0.043	30.2
DDS	0.242	27.9	0.025	19.0	0.008	5.0	0.006	4.2
SCA/DMS	0.034	4.0	0.001	0.7	0.010	6.1	0.005	3.8
Sultentrazone	0.019	2.2	0.009	6.6	0.003	1.9	0.003	2.4
DMSS	0.010	1.2	0.002	1.2	0.001	0.3	0.003	1.8
Unknown 30-33	0.003	0.3	-		-		-	
MTz	0.137	15.8	0.016	11.9	0.017	10.2	0.028	19.9
Polar B	0.024	2.7	0.000	0.3	0.004	2.6	0.008	5.5
Polar A	0.013	1.5	-		0.020	12.3	0.003	2.1
Bound[1]	0.041	4.7	0.006	4.8	0.006	3.8	0.017	12.1
Unassigned[2]	0.100	11.5	0.029	22.2	0.034	21.1	0.021	15.0
Radish Root								
HMS	0.072	20.9	0.009	14.9	0.009	18.7	0.010	7.0
DDS	0.034	10.0	-		0.001	1.8	0.001	1.0
SCA/DMS	0.017	4.9	0.004	7.0	0.007	14.3	0.004	3.0
Sultentrazone	0.040	11.5	0.008	12.2	0.004	9.1	0.003	1.8
DMSS	0.006	1.9	0.001	1.3	0.001	1.0	0.001	0.7
MTz	0.010	3.0	0.004	7.0	0.001	2.0	0.003	1.8
Polar D	0.016	4.6	0.001	2.2	0.001	1.9	0.001	0.9
Bound[1]	0.101	29.5	0.023	37.2	0.013	28.5	0.099	71.6
Unassigned[2]	0.031	9.0	0.011	17.3	0.010	21.8	0.017	12.2

[1] 30 DAT samples after acid hydrolysis, [2]Total of unknown HPLC peaks and unanalyzed fractions
- = Not detected
SCA = Sulfentrazone Carboxylic Acid
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DMSS = Des-MethylSulfonyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone
MTz = Methyl Triazole

Nature of the Residue in Lettuce: The results of HPLC fractionation of triazole- and phenyl-labelled lettuce extracts are shown in Tables 7 and 8. 3-Hydroxymethyl sulfentrazone was the

major metabolite identified, accounting for 15-18% of the TRR at 30 DAT and 21-41% at 364 DAT. Other major metabolites included desmethyl des(difluoromethyl) sulfentrazone, accounting for 20-33% of the TRR at 30 DAT and 3-4% at 364 DAT. Minor metabolites identified included sulfentrazone *per se*, sulfentrazone carboxylic acid/3-desmethyl sulfentrazone, methyl triazole and des(methylsulfonyl) sulfentrazone. Unidentified peaks accounted for up to 4.9% of the TRR.

Nature of the Residue in Barley Forage: The results of HPLC fractionation of triazole- and phenyl-labelled barley forage extracts are shown in Tables 7 and 8. 3-Hydroxymethyl sulfentrazone was the major metabolite identified in the 245 and 364 DAT samples, accounting for 15-19% of the TRR at 30 DAT and 22-28% at 364 DAT. Sulfentrazone carboxylic acid/3-desmethyl sulfentrazone was the major metabolite identified in the 30 and 122 DAT samples, accounting for 19-31% of the TRR at 30 DAT and 17-29% at 364 DAT. Other major metabolites included desmethyl des(difluoromethyl) sulfentrazone, accounting for 17-26% of the TRR at 30 DAT and 4-6% at 364 DAT, and methyl triazole, accounting for 25% of the TRR at 30 DAT and 17% at 364 DAT. Minor metabolites identified included sulfentrazone *per se* and des(methylsulfonyl) sulfentrazone. Unidentified peaks accounted for up to 8.7% of the TRR.

Table 7- Metabolite identification of phenyl-labelled lettuce leaf and barley forage residues.

Metabolite	30 DAT		122 DAT		245 DAT		364 DAT	
	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
Lettuce								
HMS	0.116	17.8	0.036	18.5	0.009	20.8	0.047	40.8
DDS	0.217	33.3	0.078	40.3	0.008	17.1	0.003	2.6
SCA/DMS	0.003	0.5	0.001	0.4	0.001	2.2	0.004	3.8
Sultentrazone	0.037	5.7	0.011	5.7	0.001	1.8	0.005	4.1
DMSS	0.023	3.5	0.006	3.1	0.003	5.8	0.006	5.3
Unknown 30-33	0.032	4.9	0.002	1.1	-		-	
Unknown 38-39	0.021	3.2	0.001	0.6	-		-	
Bound[1]	0.130	20.0	0.040	20.7	0.017	37.8	0.038	32.7
Unassigned[2]	0.070	10.6	0.011	5.6	0.007	14.7	0.012	10.7
Barley Forage								
HMS	0.272	19.3	0.058	16.6	0.136	28.7	0.062	28.4
DDS	0.364	25.9	0.125	35.7	0.039	8.2	0.009	4.1
SCA/DMS	0.439	31.2	0.054	15.6	0.140	29.6	0.064	29.1
Sultentrazone	0.010	0.7	0.008	2.3	0.009	1.9	0.005	2.2
DMSS	0.079	5.6	0.012	3.5	0.014	3.0	0.007	3.4
Bound[1]	0.091	6.5	0.054	15.4	0.083	17.5	0.032	14.7
Unassigned[2]	0.152	10.8	0.032	9.0	0.053	11.1	0.040	18.1

[1] 30 DAT samples after acid hydrolysis, [2]Total of unknown HPLC peaks and unanalyzed fractions
- = Not detected
SCA = Sulfentrazone Carboxylic Acid
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DMSS = Des-MethylSulfonyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone

Table 8- Metabolite identification of triazole-labelled lettuce leaf and barley forage residues.

Metabolite	30 DAT		122 DAT		245 DAT		364 DAT	
	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
Lettuce								
HMS	0.067	15.3	0.030	27.3	0.009	25.4	0.006	20.5
DDS	0.087	19.9	0.018	16.1	0.004	12.6	0.001	4.1
SCA/DMS	0.004	0.9	0.001	1.3	0.001	1.7	0.001	2.0
Sultentrazone	0.028	6.3	0.005	4.1	0.002	4.5	0.001	2.8
DMSS	0.007	1.5	0.001	0.9	0.001	3.2	0.001	2.1
Unknown 30-33	0.011	2.4	0.001	1.2	-		-	
Unknown 38-39	0.009	2.1	0.002	1.5	-		-	
MTz	0.085	19.3	0.008	7.3	-		0.001	3.2
Bound[1]	0.100	22.6	0.019	17.6	0.008	22.7	0.008	26.4
Unassigned[2]	0.039	8.9	0.021	18.8	0.010	30.1	0.011	38.2
Barley Forage								
HMS	0.303	14.7	0.069	11.6	0.040	12.2	0.111	22.5
DDS	0.354	17.1	0.144	24.1	0.015	4.4	0.029	5.9
SCA/DMS	0.389	18.8	0.088	14.8	0.028	8.4	0.084	16.9
Sultentrazone	0.015	0.7	0.008	1.4	0.003	1.0	0.005	1.0
DMSS	0.073	3.5	0.018	3.0	0.008	2.5	0.007	1.5
MTz	0.515	24.9	0.100	16.9	0.090	27.4	0.084	17.1
Polar B	0.093	4.5	0.041	6.9	0.007	2.2	0.043	8.7
Polar D	0.030	1.5	0.001	0.2	0.011	3.3	0.010	2.1
Bound[1]	0.085	4.1	0.050	8.4	0.038	11.5	0.049	10.0
Unassigned[2]	0.200	9.7	0.049	8.3	0.049	14.8	0.068	13.7

[1] 30 DAT samples after acid hydrolysis, [2]Total of unknown HPLC peaks and unanalyzed fractions
- = Not detected
SCA = Sulfentrazone Carboxylic Acid
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DMSS = Des-MethylSulfonyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone
MTz = Methyl Triazole

Nature of the Residue in Barley Straw: The results of HPLC fractionation of triazole- and phenyl-labelled barley straw extracts are shown in Tables 9 and 10. 3-Hydroxymethyl

sulfentrazone was the major metabolite identified in the 30 DAT samples, accounting for 16-32% of the TRR at 30 DAT and 9-15% at 364 DAT. Sulfentrazone carboxylic acid/3-desmethyl sulfentrazone was the major metabolite identified in the 122, 245 and 364 DAT samples, accounting for 12-29% of the TRR at 30 DAT and 12-23% at 364 DAT. Other major metabolites included methyl triazole, accounting for 25% of the TRR at 30 DAT and 4% at 364 DAT. Minor metabolites identified included sulfentrazone *per se*, desmethyl des(difluoromethyl) sulfentrazone and des(methylsulfonyl) sulfentrazone. Unidentified peaks accounted for up to 17.5% of the TRR.

Nature of the Residue in Barley Grain: The results of HPLC fractionation of triazole- and phenyl-labelled barley grain extracts are shown in Tables 9 and 10. 3-Hydroxymethyl sulfentrazone was the major metabolite identified in the 122 DAT samples, accounting for 7-19% of the TRR at 30 DAT and 3-17% at 245 DAT. Sulfentrazone carboxylic acid/3-desmethyl sulfentrazone was the major metabolite identified in the 30 and 245 DAT samples, accounting for 8-19% of the TRR at 30 DAT and 4-22% at 245 DAT. Other major metabolites included methyl triazole, accounting for 3% of the TRR at 30 DAT and 24% at 245 DAT. Minor metabolites identified included sulfentrazone *per se* and des(methylsulfonyl) sulfentrazone.

Polar Metabolites: Significant amounts (>10% of the TRR and >0.05 ppm) of polar metabolites A and D were observed in the triazole-labelled 245 and 364 DAT barley straw samples (Table 10). These highly polar metabolites were characterized as being closely related to methyl triazole and may represent various oxidation states of the 3-methyl group.

Table 9- Metabolite identification of phenyl-labelled barley straw and grain residues.

Metabolite	30 DAT		122 DAT		245 DAT		364 DAT	
	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
Barley Straw								
HMS	0.959	32.2	0.671	24.6	0.186	17.5	0.099	14.7
DDS	0.045	1.5	0.004	0.1	0.075	7.1	0.044	6.5
SCA/DMS	0.862	28.9	0.760	27.7	0.300	28.3	0.158	23.4
Sultentrazone	0.147	4.9	0.044	1.6	0.021	2.0	0.019	2.8
DMSS	0.167	5.6	0.077	2.8	0.039	3.4	0.019	2.9
Unknown 30-33	0.066	2.2	0.070	2.6	-		0.051	7.6
Unknown 38-40	0.074	2.5	0.020	0.7	-		-	
Unknown 49-50	-		-		-		0.020	2.9
Bound[1]	0.307	10.3	0.686	25.2	0.305	28.8	0.224	33.3
Unassigned[2]	0.357	12.0	0.365	13.4	0.120	11.3	0.040	5.9
Barley Grain								
HMS	0.010	19.1	0.005	14.6	0.002	16.8	NA	NA
SCA/DMS	0.010	19.9	0.005	14.4	0.003	22.0	NA	NA
Sultentrazone	0.001	1.8	0.000	1.3	0.000	2.0	NA	NA
DMSS	0.001	2.7	0.001	2.8	0.001	6.4	NA	NA
Bound[1]	0.010	19.6	0.013	37.7	0.007	47.2	NA	NA
Unassigned[2]	0.018	35.0	0.010	28.1	0.001	5.7	NA	NA

[1] 30 DAT samples after acid hydrolysis, [2]Total of unknown HPLC peaks and unanalyzed fractions
- = Not detected, NA = Not Analyzed
SCA = Sulfentrazone Carboxylic Acid
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DMSS = Des-MethylSulfonyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone

Table 10- Metabolite identification of triazole-labelled barley straw and grain residues.

Metabolite	30 DAT		122 DAT		245 DAT		364 DAT	
	ppm	% TRR	ppm	% TRR	ppm	% TRR	ppm	% TRR
Barley Staw								
HMS	0.549	16.3	0.774	18.1	0.104	6.1	0.170	9.3
DDS	0.040	1.2	0.037	0.9	0.047	2.7	0.054	3.0
SCA/DMS	0.417	12.4	0.773	18.1	0.136	8.0	0.224	12.2
Sultentrazone	0.143	4.3	0.030	0.7	0.020	1.2	0.041	2.2
DMSS	0.100	3.0	0.102	2.4	0.021	1.2	0.033	1.8
Unknown 30-33	0.053	1.6	0.074	1.7	-		0.086	4.7
Unknown 38-40	0.044	1.3	0.027	0.6	-		-	
Unknown 49-50	-		-		-		0.026	1.4
MTz	0.827	24.6	0.927	21.7	0.090	5.3	0.063	3.5
Polar B	0.261	7.8	0.223	5.2	0.096	5.6	0.025	1.4
Polar A	0.032	1.0	-		0.299	17.5	0.243	13.3
Polar D	0.190	5.6	0.031	0.7	0.262	15.3	0.018	1.0
Bound[1]	0.224	6.7	0.529	12.4	0.420	24.6	0.547	29.9
Unassigned[2]	0.482	14.3	0.717	16.8	0.179	10.5	0.300	16.4
Barley Grain								
HMS	0.003	7.3	0.006	11.3	0.001	2.6	NA	NA
SCA/DMS	0.003	8.3	0.006	10.3	0.001	3.7	NA	NA
Sultentrazone	0.001	1.2	0.002	3.7	0.000	1.1	NA	NA
DMSS	0.000	1.1	0.000	0.5	0.001	1.5	NA	NA
MTz	0.001	3.1	0.008	14.0	0.008	23.6	NA	NA
Bound[1]	0.004	10.4	0.013	24.2	0.008	23.0	NA	NA
Unassigned[2]	0.024	59.5	0.015	27.5	0.012	34.6	NA	NA

[1] 30 DAT samples after acid hydrolysis, [2]Total of unknown HPLC peaks and unanalyzed fractions
- = Not detected, NA = Not Analyzed
SCA = Sulfentrazone Carboxylic Acid
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DMSS = Des-MethylSulfonyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone
MTz = Methyl Triazole

Bound Residues: In general, the phenyl-labelled samples had higher levels of bound residues than did the triazole-labelled samples.

Also, soluble cleavage products were detected in the triazole-labelled samples but not in the phenyl-labelled samples, indicating that the phenyl-labelled cleavage products may have been incorporated into the bound residues. The registrant has characterized the bound residues of the 30 DAT samples by sonication and refluxing in 1 N HCl. This procedure released 17-64% of the bound residues (Table 11). The released residues were analyzed by HPLC. The metabolite profile of the residue was similar to that of the soluble residues, with 3-hydroxymethyl sulfentrazone being the major metabolite. These results are incorporated into tables 4-10. The following samples contained significant amounts (>10% of the TRR and >0.05 ppm) of bound residues that were not analyzed: 122 DAT barley forage (phenyl-labelled) and straw (both labels), 245 DAT barley forage (phenyl-labelled) and straw (both labels), and 364 DAT radish root (triazole-labelled) and straw (both labels).

Table 11- Analysis of bound residues of 30 DAT samples by sonication and refluxing in 1 N HCl.

Sample	Label	Initial Residues		Released Residues		% Released
		ppm	% TRR	ppm	% TRR	
Radish Top	Phenyl	0.082	11.5	0.032	4.5	39.0
	Triazole	0.070	8.1	0.029	3.4	41.4
Radish Root	Phenyl	0.131	41.6	0.022	7.0	16.8
	Triazole	0.122	35.8	0.022	6.3	18.0
Lettuce	Phenyl	0.193	29.9	0.065	9.9	33.7
	Triazole	0.130	29.7	0.031	7.1	23.8
Barley Forage	Phenyl	0.133	9.5	0.043	3.1	32.3
	Triazole	0.124	6.0	0.039	1.9	31.5
Barley Straw	Phenyl	0.548	18.3	0.235	8.0	42.9
	Triazole	0.393	11.7	0.170	5.0	43.3
Barley Grain	Phenyl	0.025	47.4	0.015	27.9	60.0
	Triazole	0.011	28.4	0.007	18.0	63.6

Storage Stability: Samples were analyzed within 8 months of harvest. Storage stability was demonstrated by comparison of the analysis of samples performed after 17 months of storage with the initial analysis. Residues in all matrices appeared to be stable

for this storage period.

Discussion: The results of this confined crop rotation study demonstrate that quantifiable residues of 3-hydroxymethyl sulfentrazone are present in all crops planted 1 year after sulfentrazone application. This metabolite was also the major component of the residue identified in soybean seed (30-35% of the TRR). Limited field trials are thus required in order to determine whether rotational crop tolerances are needed and the appropriate plantback intervals.

The nature of the residue in rotational crops can not be considered to be understood due to deficiencies in the characterization of bound residues. Minimal analysis was performed only on the 30 DAT samples. CBTS requests that registrant analyze the bound residues from the 364 DAT samples of barley straw (both phenyl- and triazole-labelled). The methods employed should include treatment with enzymes, surfactants, dilute acid and base and refluxing with 6 N acid and base.

Rotational Crop Studies: GLN § 165-2

Submitted with this petition:

> Determination of the Residue of Sulfentrazone and its Metabolites in/on Winter Wheat as a Rotated Crop following Harvest of F6285 WDG Treated Soybeans. MRID# 433454-29

Four limited field trials were conducted in the states of IL (2), GA and LA in 1992. Sulfentrazone 75 DF was applied at a rate of 0.5 lbs. ai/A (1.3X). Preplant soil incorporation (PPI) was employed in three trials and preemergence application was used in one trial (IL). Soybeans were planted, grown and harvested. Rotational winter wheat was planted 4-6 months after sulfentrazone application. Wheat forage was harvested 30-60 days PHI; wheat grain and straw, 226-295 days PHI. After harvest, samples were stored frozen until analysis. Samples were analyzed for sulfentrazone and 3-desmethyl sulfentrazone by extraction in acetone/0.25 N HCl (70/30, v/v) with refluxing. After filtration, the samples were cleaned-up using C-8 and silica SPE columns. The samples were then analyzed by GC-ECD. Samples of forage and grain were analyzed for 3-hydroxymethyl sulfentrazone by extraction in acetone/0.25 N HCl (3/1, v/v) with refluxing. After filtration, the samples were cleaned-up using C-8 and silica SPE columns. The samples were then derivatized with N,O-bis-(trimethylsilyl)-trifluoroacetamide (BSTFA) and analyzed by GC-ECD. Samples of straw were analyzed for 3-hydroxymethyl sulfentrazone by extraction in acetone/0.25 N HCl (3/1, v/v) with refluxing. After filtration, the samples were cleaned-up using solvent partitioning with dichloromethane. The samples were then derivatized with BSTFA,

cleaned-up using a silica SPE column and analyzed by GC-ECD. Samples of forage were analyzed for desmethyl des(difluoromethyl) sulfentrazone by extraction in acetone/0.25 N HCl (3/1, v/v) with refluxing. After filtration, the samples were cleaned-up using C-18 SPE column chromatography. The samples were then derivatized with iodomethane, cleaned-up using a silica SPE column and analyzed by GC-ECD. The limit of detection (LOD) was reported to be 0.005 ppm for sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone and 0.01 ppm for desmethyl des(difluoromethyl) sulfentrazone. The limit of quantitation (LOQ) was reported to be 0.025 ppm for sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone and 0.05 ppm for desmethyl des(difluoromethyl) sulfentrazone. These methods were validated in wheat forage, straw and grain over a range of 0.025-0.25 ppm. The average recovery for sulfentrazone was 100 ± 13% (n=12); for 3-desmethyl sulfentrazone, 109 ± 16% (n=12); for 3-hydroxymethyl sulfentrazone, 79 ± 10% (n=11); and for desmethyl des(difluoromethyl) sulfentrazone, 103 ± 8% (n=4). Analysis of the treated samples showed that the total of sulfentrazone and its metabolites was a maximum of 0.048 ppm in forage and 0.041 ppm in straw (Table 12). No residues were detected in grain.

Table 12- Results of limited field trials for winter wheat in which Sulfentrazone 75DF was applied to the primary crop at a rate of 0.5 lbs. ai/A. Values of 0.005-0.025 ppm are above the LOD, but below the LOQ.

Location	DAT[1]	RAC	PHI (Days)	Maximum Residue (ppm)				
				Sulfent.	DMS	HMS	DDS	Total
GA[2]	145	Forage	60	0.014	0.013	ND	ND	0.027
		Grain	231	ND	ND	ND	NA	ND
		Straw	231	ND	0.023	0.018	NA	0.041
IL[2]	132	Forage	60	0.008	0.018	ND	ND	0.026
		Grain	296	ND	ND	ND	NA	ND
		Straw	296	ND	ND	ND	NA	ND
IL[3]	132	Forage	60	0.012	0.022	ND	0.014	0.048
		Grain	296	ND	ND	ND	NA	ND
		Straw	296	ND	ND	ND	NA	ND
LA[2]	181	Forage	30	0.010	0.006	0.025	ND	0.041
		Grain	226	ND	ND	ND	NA	ND
		Straw	226	0.006	0.016	0.006	NA	0.028

[1]Days after treatment of soil with sulfentrazone when wheat was planted
[2]Sulfentrazone applied by preplant incorporation, [3]Sulfentrazone applied preemergence
ND = Not Detected; i.e., below the LOD (0.005 ppm for sulfentrazone, HMS and DMS; 0.01 ppm for

DDS).
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone

Submitted with this petition:

Determination of the Residue of Sulfentrazone and its Metabolites in/on Winter Wheat as a Rotated Crop following Harvest of F6285/Treflan WDG Treated Soybeans. MRID# 433454-30

One limited field trial was conducted in IA in 1992. Sulfentrazone/Treflan (trifluralin) WDG was applied at a rate of 0.5 lbs. sulfentrazone/A (1.3X) by PPI. Soybeans were planted, grown and harvested. Rotational winter wheat was planted 127 days after sulfentrazone application. Wheat forage was harvested 60 days PHI; wheat grain and straw, 296 days PHI. After harvest, samples were stored frozen until analysis. Samples were analyzed by the methods described above. These methods were validated in wheat forage, straw and grain over a range of 0.025-0.125 ppm. The average recovery for sulfentrazone was 95 ± 16% (n=3); for 3-desmethyl sulfentrazone, 102 ± 7% (n=3); for 3-hydroxymethyl sulfentrazone, 72 ± 6% (n=6); and for desmethyl des(difluoromethyl) sulfentrazone, 98% (n=1). Analysis of the treated samples showed that the total of sulfentrazone and its metabolites was a maximum of 0.034 ppm in forage (Table 13). No residues were detected in straw and grain.

Table 13- Results of limited field trial for winter wheat in which Sulfentrazone/Treflan was applied to the primary crop at a rate of 0.5 lbs. ai/A for sulfentrazone. Values of 0.005-0.025 ppm are above the LOD, but below the LOQ.

Location	DAT[1]	RAC	PHI (Days)	Maximum Residue (ppm)				
				Sulfent.	DMS	HMS	DDS	Total
IA[2]	127	Forage	60	0.016	0.018	ND	ND	0.034
		Grain	295	ND	ND	ND	NA	ND
		Straw	295	ND	ND	ND	NA	ND

[1]Days after treatment of soil with sulfentrazone when wheat was planted
[2]Sulfentrazone applied by preplant incorporation
ND = Not Detected; i.e., below the LOD (0.005 ppm for sulfentrazone, HMS and DMS; 0.01 ppm for DDS).
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone
DDS = Desmethyl Des(difluoromethyl) Sulfentrazone

Table 14- Results of limited field trials for field corn in which Sulfentrazone 75DF was applied to the primary crop at a rate of 0.5 lbs. ai/A. Values of 0.005-0.025 ppm are above the LOD, but below the LOQ.

Location	DAT[1]	RAC	PHI (Days)	Maximum Residue (ppm)			
				Sulfent.	DMS	HMS	Total
LA	294	Silage	113	ND	ND	ND	ND
		Grain	144	ND	ND	ND	ND
		Fodder	144	ND	0.005	ND	0.005
NE	344	Silage	123	ND	ND	ND	ND
		Grain	158	ND	ND	ND	ND
		Fodder	158	ND	ND	ND	ND
TN	339	Silage	98	ND	0.012	ND	0.012
		Grain	124	ND	ND	ND	ND
		Fodder	124	ND	0.020	ND	0.020
IL	370	Silage	114	ND	ND	ND	ND
		Grain	138	ND	ND	ND	ND
		Fodder	138	ND	ND	ND	ND

[1]Days after treatment of soil with sulfentrazone when wheat was planted
ND = Not Detected; i.e., below the LOD (0.005 ppm).
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone

Submitted with this petition:

Magnitude of the Residue of Sulfentrazone and its Metabolites in/on Field Corn as a Rotated Crop following Harvest of Soybeans Treated with F6285/Command WDG at 0.5 Pound Active Ingredient (F6285) per Acre. MRID# 433454-32

Two limited field trials were conducted in the states of IL and GA in 1992. Sulfentrazone/Command WDG (clomazone) was applied at a rate of 0.5 lbs. sulfentrazone/A (1.3X) by (PPI). Soybeans were planted, grown and harvested. Rotational field corn was planted 10-11 months after sulfentrazone application. Corn silage or forage was harvested 83-90 days PHI; corn grain and fodder, 114-137 days PHI. After harvest, samples were stored frozen until analysis. Samples were analyzed by the methods described above. These methods were validated in corn silage, fodder and grain at 0.025 ppm. The average recovery for sulfentrazone was 89 ± 11% (n=6); for 3-desmethyl sulfentrazone, 105 ± 13% (n=6); and for 3-hydroxymethyl sulfentrazone, 76 ± 5% (n=6). Detectable residues of

sulfentrazone, 3-desmethyl sulfentrazone or 3-hydroxymethyl sulfentrazone were not found in any RAC (Table 15).

Table 15- Results of limited field trials for field corn in which Sulfentrazone/Command WDG was applied to the primary crop at a rate of 0.5 lbs. sulfentrazone/A.

Location	DAT[1]	RAC	PHI (Days)	Maximum Residue (ppm)			
				Sulfent.	DMS	HMS	Total
IL	349	Forage	90	ND	ND	ND	ND
		Grain	137	ND	ND	ND	ND
		Fodder	137	ND	ND	ND	ND
GA	299	Silage	83	ND	ND	ND	ND
		Grain	114	ND	ND	ND	ND
		Fodder	114	ND	ND	ND	ND

[1]Days after treatment of soil with sulfentrazone when wheat was planted
ND = Not Detected; i.e., below the LOD (0.005 ppm).
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone

Conclusions: The registrant has submitted the results of five limited field rotational trials for corn and six for winter wheat. No quantifiable residues were observed in field corn so that rotational crop tolerances are not required for corn with a 10 month or greater plantback interval. However, quantifiable residues of 3-hydroxymethyl sulfentrazone were observed in winter wheat forage so that rotational crop tolerances are required for wheat. The required number of field trials required to set rotational crop tolerances is the same as that required to establish primary crop tolerances (i.e., 20 for wheat- see *EPA Guidance on Number and Location of Domestic Crop Field Trials for Establishment of Pesticide Residue Tolerances, 6/2/94)*.

The sulfentrazone label allows rotational barley, peanuts and rice to be planted at 12 months. However, limited field trials are required for these crops in order to determine whether rotational crop tolerances are required. If two limited trials are performed with barley or rice and no quantifiable residues are observed, then it will be concluded that rotational crop tolerances are not required for either crop. Until the required data for rotational barley, rice and peanuts are submitted, all plantback intervals of 1 year or less should be removed from the sulfentrazone label, except for soybeans, wheat and corn.

The registrant has proposed tolerances in/on wheat and corn RACs

for residues of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone. These tolerances are not required for corn and should be withdrawn. **A revised Section F is required.** This conclusion is contingent on the ability of the registrant to demonstrate the storage stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone residues in corn RACs (see Storage Stability section, below).

As noted above, tolerances are required for wheat grain, straw, forage and hay. CBTS is unable to comment on the adequacy of the proposed wheat tolerances until receipt and review of the requested residue data. Also, the tolerance expression for wheat should be revised to incorporate the following language: "Tolerances are established for the indirect or inadvertent combined residues of..." Alternatively, the registrant may choose to withdraw the proposed tolerances for wheat RACs and include a prohibition against rotation to wheat on the sulfentrazone labels.

Nature of Residue- Plants

Deficiency - Conclusion 4b (from Memo, G. Kramer 4/25/94)

4b) For the permanent tolerance petition, the petitioner must address the following deficiencies in the soybean metabolism study: i) The storage stability of the samples in this study has not been demonstrated. The registrant should report the actual dates of extraction and chromatography. If the samples were stored longer than 6 months prior to analysis, then the registrant must show that the nature of the residue in the samples has not changed during storage by presenting representative chromatographic separations performed early in the study and at the conclusion of the study. If such data do not exist or if significant changes in the metabolite profile occurred during storage, the registrant may be required to repeat this metabolism study. **ii)** Unknown metabolites 2 (0.065-0.077 ppm in hay and 0.061-0.076 ppm in forage), 3 (0.105-0.110 in hay and 0.023-0.088 in forage), 5 (0.045-0.050 ppm in hay and 6 (up to 13.1% of the TRR in seed) accounted for significant portions of the TRR in soybean RACs. The registrant should identify these compounds. **iii)** Significant portions of the TRR in forage and grain were found to be extractable but were not characterized by HPLC (polar metabolites). The registrant should characterize any of these fractions which contain >0.05 ppm or >10% of the TRR (polar extracts of forage, triazole-labelled polar extract of hay and triazole-labelled polar extract of seed). **iv)** Significant portions of the bound residues of hay and forage remained uncharacterized after enzymatic digestions. The registrant should further characterize these bound residues.

Petitioner's Response: None

CBTS' Conclusion: The requested information has not been provided. **This deficiency remains outstanding.**

CBTS will refer to the Metabolism Committee on the toxicological significance of metabolites once the deficiencies associated with

plant metabolism have been addressed. A decision by CBTS concerning which residues to regulate will then follow. A tolerance based on the parent and 3-hydroxymethyl sulfentrazone may not be appropriate; in such an instance a revised Section F and additional field studies, analytical methodology, and storage stability data may be needed.

Nature of Residue- Animals

Deficiency - Conclusion 5 (from Memo, G. Kramer 4/25/94)

5) Metabolism studies for sulfentrazone in ruminants and poultry have not been reported. This data will not be required for this EUP due to the label restrictions against the feeding of treated RACs to livestock and the limited number of acres involved. However, acceptable metabolism studies in ruminants and poultry will be required for the permanent tolerance petition. These studies should utilize sulfentrazone labelled in both rings or separate studies should be performed using [^{14}C]phenyl- and [^{14}C]triazole-labelled sulfentrazone. If there are significant sulfentrazone metabolites (exocons) formed in soybean which are not also formed in animals, then CBTS may also require metabolism studies using any such metabolites.

Ruminants: Submitted with this petition:

> F6285 Dairy Goat Metabolism Study: Tissues, Milk and Excreta. Performing Laboratory: ABC Labs, Inc. MRID# 433454-15

In-Life Phase: [Phenyl(U)-^{14}C]- (20.1 mCi/mmol) and [triazole(carbonyl)-^{14}C]-sulfentrazone (24.0 mCi/mmol) were isotopically diluted, mixed with microgranular cellulose in a dosing capsule and administered orally to lactating goats (weight of 45-60 kg, minimum age 1 year) with the aid of a balling gun. The goats were dosed at a rate of 4.9 ppm (phenyl) or 6.0 ppm (triazole). Doses were administered once daily for 10 consecutive days. The animals were sacrificed approximately 22 hours after administration of the final dose.

Quantitation of Total Radioactivity: Milk was collected twice daily. Tissues were obtained after sacrifice. The distribution of the radioactivity is shown in Table 16. Of the administered radioactivity, 78-94% was recovered in urine. Another 4.5-7.4% was recovered in the feces and <0.04% was recovered in the milk, blood and tissues. The total recovery was over 85%. The TRR in tissues and milk is shown in Table 17. The greatest tissue residues were 0.013 ppm in kidney (phenyl).

Table 16- Total recovery of radioactivity from lactating goats treated with phenyl- or triazole-labelled sulfentrazone for 10 consecutive days.

Fraction	% of Total Radioactivity Administered	
	Phenyl	Triazole
Urine	77.6	93.7
Feces	7.39	4.54
Milk	0.020	0.017
Blood	0.001	<0.001
Tissues	0.011	<0.001
Total	85.02	98.26

Table 17- TRR in goat milk and tissues following treatment with phenyl- or triazole-labelled sulfentrazone for 10 consecutive days.

Fraction	TRR (ppm)	
	Phenyl	Triazole
Liver	0.0068	0.0006
Kidney	0.0130	0.0010
Heart	0.0011	0.0001
Perirenal Fat	0.0004	<0.0001
Omental Fat	0.0007	0.0001
Longissimus Dorsi Muscle	0.0006	<0.0001
Semimembranous Muscle	0.0008	0.0004
Tricep Muscle	0.0003	<0.0001
Milk*	0.0011	0.0011

*Day 2 sample

Extraction and Fractionation: Kidney (phenyl) and feces were extracted in acetonitrile/water (80/20) and the debris removed. The extract was partitioned with ethyl acetate, dividing the residues into three fractions- organic-soluble, aqueous-soluble and

bound. The feces aqueous extract was acidified and further partitioned with ethyl acetate. Urine was partitioned with ethyl acetate, dividing the sample into two fractions- organic-soluble and aqueous-soluble. The aqueous fraction was acidified and further partitioned with ethyl acetate. Of the kidney residue, 69.2% was extractable.

Metabolite Identification: Organic-soluble residues were analyzed by HPLC and TLC and the retention times compared with those of possible metabolites. The identity of metabolites was further confirmed by GC-MS of peaks isolated from the urine samples.

Nature of the Residue in Urine: 3-Hydroxymethyl sulfentrazone was the predominant component of the residue, accounting for 96-98% of the TRR. The metabolite sulfentrazone carboxylic acid was also identified (1.2-2.0% of the TRR).

Nature of the Residue in Feces: 3-Hydroxymethyl sulfentrazone and sulfentrazone carboxylic acid were the only metabolites present in feces, but quantitative data was not reported.

Nature of the Residue in Kidney: Sulfentrazone *per se* was the predominant component of the residue, accounting for 53.8% of the TRR. The metabolite 3-hydroxymethyl sulfentrazone (7.7% of the TRR) was also identified.

Storage Stability: As evidence of storage stability, samples were spiked with labelled sulfentrazone. No evidence of degradation was observed after 174 days of storage. The actual dates of sample extraction and analysis were not provided in this report so that the sample storage interval could not be calculated. However, due to the low levels of radioactivity in the tissues, storage stability is not an issue for this study.

Poultry: Submitted with this petition:

> F6285 Laying Hen Metabolism Study: Tissues, Eggs and Excreta. Performing Laboratory: ABC Labs, Inc. MRID# 433454-14

In-Life Phase: [Phenyl(U)-^{14}C]- (20.1 mCi/mmol) and [triazole(carbonyl)-^{14}C]-sulfentrazone (24.0 mCi/mmol) were isotopically diluted, mixed with microgranular cellulose in a dosing capsule and administered orally to laying hens (weight of 1.3-1.5 kg, age of 26 weeks). Each group (control, phenyl, triazole) contained 15 animals. The hens were dosed at a rate of 4.70 ppm (phenyl) or 4.73 ppm (triazole). Doses were administered once daily for 12 consecutive days. The animals were sacrificed approximately 21-24 hours after administration of the final dose.

Quantitation of Total Radioactivity: Eggs were collected daily.

Tissues were obtained after sacrifice. Of the administered radioactivity, 94-106% was recovered in excreta. The TRR in tissues and eggs is shown in Table 18. The greatest tissue residues were 0.030 ppm in kidney (phenyl).

Table 18- Average TRR in hen excreta, eggs and tissues following treatment with phenyl- or triazole-labelled sulfentrazone for 12 consecutive days.

Fraction	TRR (ppm)	
	Phenyl	Triazole
Excreta	6.21	6.88
Egg White	0.011	0.012
Egg Yolk	0.008	0.007
Liver	0.014	0.007
Kidney	0.030	0.015
Gizzard	0.007	0.004
Fat	0.002	<0.002
Heart	0.007	0.001
Breast Muscle	<0.002	<0.002
Thigh Muscle	0.001	<0.002

*Day 2 sample

Extraction and Fractionation: Liver and egg samples were extracted in acetonitrile/water (80/20) and the debris removed. The extract was partitioned with hexane, dividing the residues into three fractions- organic-soluble, aqueous-soluble and bound. Excreta was extracted in acetonitrile/water (80/20) and the debris removed. The majority of the residue in all samples analyzed was extractable (Table 19).

Table 19- Metabolite identification in hen egg yolks, egg whites and liver

Fraction/ Metabolite	Phenyl		Triazole	
	ppm	% TRR	ppm	% TRR
Egg White				
Total Extractable	0.015	89.8	0.010	73.5
Sulfentrazone	0.010	56.5	0.007	50.2
HMS	0.006	32.9	0.002	17.8
DMS	ND		ND	
Egg Yolk				
Total Extractable	0.012	88.8	NE	
Sulfentrazone	0.010	70.4		
HMS	0.002	14.3		
DMS	ND			
Liver				
Total Extractable	0.009	66.1	NE	
Sulfentrazone	0.003	26.8		
HMS	0.002	17.9		
DMS	0.002	13.5		

ND = Not Detected; NE = Not Extracted
HMS = Hydroxy Methyl Sulfentrazone
DMS = Des-Methyl Sulfentrazone

Metabolite Identification: Soluble residues were analyzed by HPLC and TLC and the retention times compared with those of possible metabolites.

Nature of the Residue in Excreta: 3-Hydroxymethyl sulfentrazone was the predominant component of the residue, accounting for 95-97% of the TRR.

Nature of the Residue in Egg White: Sulfentrazone *per se* was the predominant component of the residue, accounting for 50-56% of the

TRR (Table 19). The metabolite 3-hydroxymethyl sulfentrazone (18-33% of the TRR) was also identified.

Nature of the Residue in Egg Yolk: Sulfentrazone *per se* was the predominant component of the residue, accounting for 70% of the TRR (Table 19). The metabolite 3-hydroxymethyl sulfentrazone (14% of the TRR) was also identified.

Nature of the Residue in Liver: Sulfentrazone *per se* was the predominant component of the residue, accounting for 27% of the TRR (Table 19). The metabolites 3-hydroxymethyl sulfentrazone (18% of the TRR) and 3-desmethyl sulfentrazone (14% of the TRR) were also identified.

Storage Stability: As evidence of storage stability, samples were spiked with labelled sulfentrazone. No evidence of degradation was observed after an unspecified period of storage. The actual dates of sample extraction and analysis were not provided in this report so that the sample storage interval could not be calculated. However, due to the low levels of radioactivity in the tissues, storage stability is not an issue for this study.

Conclusions: CBTS requires that the dietary dosing level for animal metabolism studies be at least 10 ppm. The dosing levels in both the ruminant and poultry studies was well below 10 ppm. However, CBTS will accept these studies since the dose was >10X the estimated maximum dietary burden, the studies were conducted over 10-12 days instead of the usual 3 days, and the majority of the TRR in tissues was identified as sulfentrazone and 3-hydroxymethyl sulfentrazone. For any future petition which results in a higher dietary burden, CBTS may require that the poultry and/or ruminant metabolism studies be repeated. Also, if the registrant wishes to propose tolerances for soybean forage and hay, this conclusion will be reevaluated.

Analytical Methodology- Plants

Method P-2811M (MRID# 429321-09)

Procedure: Macerated tissue is initially refluxed in acetone/0.25 N HCl (75/25, v/v). After filtration, the acetone is removed by evaporation. The sample is then cleaned-up using C-8 SPE and silica gel cartridge columns. The sample is concentrated and derivatized with N,O-bis-(trimethylsilyl)-trifluoroacetamide (BSTFA), which converts hydroxymethyl-sulfentrazone to its trimethylsilyl derivative. Analysis was then performed on GC/EC with a Megabore column. This method simultaneously measures both sulfentrazone and hydroxymethyl-sulfentrazone.

Results: The method was validated with sulfentrazone and hydroxymethyl-sulfentrazone in soybean seed at the reported LOQ, 0.025 ppm. The average recovery was 113 ± 6% (n = 7) for sulfentrazone and 84 ± 12% for hydroxymethyl-sulfentrazone. No method validation using soybean forage and hay was reported. The LOD was reported to be 0.005 ppm.

ILV: An ILV of this method was performed by ADPEN Laboratories. Acceptable recoveries were obtained by the laboratory for all analytes. The method and ILV have been sent to Beltsville for PMV (Memo, G. Kramer 2/16/95). CBTS will withhold a final conclusion on the adequacy of this method as an analytical enforcement method pending receipt of the PMV report. If tolerances are proposed for soybean forage and hay, then enforcement methodology which measures all residues of concern in these RACs will be required. If substantially different from the current method, then the registrant should obtain an ILV for this method(s).

Multiresidue Method Testing: Reports on Multiresidue testing for both sulfentrazone (MRID# 433454-16) and 3-hydroxymethyl sulfentrazone (MRID# 433454-17) have been received and forwarded to FDA (Memo, G. Kramer 2/7/95). Neither compound was recovered by any of the protocols.

Radiovalidation: A sample from the plant metabolism study was subjected only to the initial hydrolysis step of the proposed enforcement method. Of the TRR, 96% was solubilized by this method. As the conjugated residues were shown to be released by acid hydrolysis with 1 N HCl in the plant metabolism study, CBTS can reach no conclusion on whether conjugated residues are released by the proposed enforcement method. Radiovalidation should be performed by running the entire method on samples from the plant metabolism study.

Confirmatory Method: The registrant has included conditions for use of a MSD detector in order to confirm the identity of the analytes.

Rotational Crops: The analytical methods used for rotational wheat RACs differed significantly from the proposed enforcement method for soybeans. CBTS thus requests that these methods be validated by an independent laboratory. Once we receive the ILV, the method will be forwarded to ACL for Agency validation.

Analytical Methodology- Animals

No analytical method has been submitted by the registrant

Since no tolerances have been proposed for animal RACs, an analytical enforcement method for animals is not required at this

time. If, however, the required ruminant feeding studies (see below) demonstrate a potential for transfer of residues to meat or milk, then the registrant will be required to propose tolerances for these RACs and develop the appropriate analytical enforcement methodology. Any required enforcement methods for meat and milk will need successful ILVs and PMVs before being judged to be acceptable by CBTS.

Storage Stability Studies

The soybean seed samples from the field residue studies were stored for a maximum of 9 months. The corn silage samples from the limited rotational crop field residue studies were stored for a maximum of 10 months; the corn grain samples, 9 months; the corn fodder samples, 9 months; the wheat grain samples, 14 months; the wheat forage samples, 20 months; and the wheat straw samples, 13 months.

The registrant has submitted the following storage stability studies:

> Cold Storage Stability of Sulfentrazone in/on Laboratory-Fortified Soybean Seed MRID# 433454-19

Samples of soybean seed were spiked with 0.25 ppm sulfentrazone and stored frozen at -18 °C. Samples were maintained frozen and three subsamples were removed and analyzed at various intervals for sulfentrazone residues using Method P-2811M over the course of 2 years. Each analysis included two freshly fortified controls. The results (Table 20) demonstrate that residues of sulfentrazone are stable during storage in soybean seed for up to 24 months.

Table 20- Average % Recovery of sulfentrazone from fortified soybean seed after storage at -18 °C.

Storage Interval (months)	Fresh Fortification Recovery (%)	Apparent Recovery in Stored Sample (%)	Corrected Recovery in Stored Sample (%)
0	91	104	114
3	89	92	103
6	98	116	118
12	112	104	93
24	102	96	94

Submitted with this petition:

Storage Stability of 3-Hydroxymethyl sulfentrazone in/on Laboratory-Fortified Soybean Seed MRID# 433454-21

Samples of soybean seed were spiked with 0.25 ppm 3-hydroxymethyl sulfentrazone and stored frozen at -18 °C. Samples were maintained frozen and three subsamples were removed and analyzed at various intervals for sulfentrazone residues using Method P-2811M over the course of 2 years. Each analysis included two freshly fortified controls. The results (Table 21) demonstrate that residues of 3-hydroxymethyl sulfentrazone are stable during storage in soybean seed for up to 11 months.

Table 21- Average % Recovery of 3-hydroxymethyl sulfentrazone from fortified soybean seed after storage at -18 °C.

Storage Interval (months)	Fresh Fortification Recovery (%)	Apparent Recovery in Stored Sample (%)	Corrected Recovery in Stored Sample (%)
0	84	100	119
1	97	96	99
3	87	96	110
6	77	96	125
11	95	100	105

Conclusions: The registrant has demonstrated that residues of sulfentrazone are stable in soybean seed for up to 24 months of

frozen storage; and for 3-hydroxymethyl sulfentrazone, 11 months. These results demonstrate storage stability for the purposes of the primary crop field trials. However, no data have been supplied on the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in corn and wheat RACs. The registrant must demonstrate the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in corn silage samples for at least 10 months of storage; and fodder samples, for 9 months. The registrant must also demonstrate the stability of 3-desmethyl sulfentrazone in corn grain samples for at least 9 months of storage. The soybean storage stability data for sulfentrazone and 3-hydroxymethyl sulfentrazone can be translated to corn grain. If the wheat field residue data submitted with this petition is to be used for setting rotational crop tolerances, then the registrant must demonstrate the stability of sulfentrazone, 3-hydroxymethyl sulfentrazone and 3-desmethyl sulfentrazone in wheat forage samples for at least 20 months of storage; wheat grain samples, 14 months; and wheat straw samples, 13 months.

Magnitude of Residue- Plants

Submitted with this petition:

> Magnitude of the Residue of Sulfentrazone and 3-Hydroxymethyl sulfentrazone in/on Soybeans Treated with F6285 75DF MRID# 433454-22

A total 12 field trials were conducted in 1993 in 10 states located in Regions 2 (2 trials), 4 (4 trials), and 5 (6 trials). The application rate of sulfentrazone 75DF was 0.375 lbs. ai/A (1X) in all trials. Preplant incorporation was employed in four trials, preemergence application in five trials and both methods were employed in separate subplots in three trials. The registrant counted the subplots in the latter three trials as separate trials. The spray volumes ranged from 10-20 gal/A. Mature soybean seeds were harvested 115-167 days after planting. Two samples were harvested from each plot. After 5-7 months in storage, seeds were analyzed using Method P-2811M. The method was validated in soybeans at 0.025 ppm. The average recovery for sulfentrazone was 96 ± 19% (n=15); for 3-hydroxymethyl sulfentrazone, 88 ± 15% (n=15). In several cases, interferant peaks were observed in the control samples. The confirmatory method (GC/MSD) was used for these samples. Residues of sulfentrazone were below the LOD (0.005 ppm) and residues of hydroxymethyl-sulfentrazone were below the LOQ (0.025 ppm) in all samples (Table 22). Detectable residues of hydroxymethyl-sulfentrazone were observed in five trials.

Table 22- Results of field residue trials for soybean seed. The application rate was 0.375 lbs. ai/A (1X). Values of 0.005-0.025 ppm are above the LOD, but below the LOQ.

Trial	Application Method	Spray Volume (Gal/A)	PHI (Days)	Maximum Residues (ppm)	
				Sulfentrazone	HMS
IL	PPI	11.5	139	ND	ND
IA	PRE	20	133	ND	0.006
	PPI	20	133	ND	0.005
OH	PRE	15.7	134	ND	0.007
NE	PRE	20	125	ND	0.006
MN	PRE	20	126	ND	ND
AR	PRE	10	140	ND	ND
AR	PPI	20	152	ND	ND
MS	PRE	15	159	ND	ND
	PPI	15	159	ND	ND
GA	PRE	20	167	ND	0.006
GA	PPI	20.5	165	ND	ND
LA	PRE	10	134	ND	ND
	PPI	10	134	ND	ND
MO	PPI	16.7	115	ND	ND

HMS = Hydroxy Methyl Sulfentrazone
ND = Not Detected (<LOD, 0.005 ppm)
PPI = Pre-Plant Incorporated
PRE = Preemergence

Submitted with this petition:

Magnitude of the Residue of FMC 97285 in/on Soybeans Treated with F6285 WDG MRID# 433454-23

A total three field trials were conducted in 1992 in three states located in Regions 2, 4 and 5. The application rate of sulfentrazone 75DF was 0.5 lbs. ai/A (1.3X) in all trials. Preplant incorporation was employed in one trial, preemergence application in one trial and both methods were employed in separate subplots in one trial. The registrant counted the subplots in the latter trial as separate trials. The spray volumes ranged from 17-19 gal/A. Mature soybean seeds were harvested 120-160 days after planting. Two samples were harvested from each plot. After 7-9 months in storage, seeds were analyzed using Method P-2811M. The method was validated in soybeans at 0.025 ppm. The average recovery for sulfentrazone was 102 ± 15% (n=5); for 3-hydroxymethyl sulfentrazone, was 94 ± 12% (n=5). Residues of sulfentrazone were below the LOD (0.005 ppm) and residues of hydroxymethyl-sulfentrazone were below the LOQ (0.025 ppm) in all samples (Table 23). Detectable residues of hydroxymethyl-sulfentrazone were observed in two trials.

Table 23- Results of field residue trials for soybean seed. The application rate was 0.5 lbs. ai/A (1.3X). Values of 0.005-0.025 ppm are above the LOD, but below the LOQ.

Trial	Application Method	Spray Volume (Gal/A)	PHI (Days)	Maximum Residues (ppm)	
				Sulfentrazone	HMS
GA	PPI	19.8	144	ND	**0.022**
IL	PRE	18.6	140	ND	ND
	PPI	18.6	140	ND	ND
LA	PPI	17.0	120	ND	0.009

HMS = Hydroxy Methyl Sulfentrazone
ND = Not Detected (<LOD, 0.005 ppm)
PPI = Pre-Plant Incorporated
PRE = Preemergence

Conclusions: The registrant previously submitted the results of seven acceptable soybean trials which employed the 4F formulation (Memo G. Kramer 4/25/94). Residues of sulfentrazone were below the LOD and residues of 3-hydroxymethyl sulfentrazone were below the LOQ in all samples. Detectable residues of 3-hydroxymethyl sulfentrazone were observed in three trials. Together with the residue data submitted with this petition, the registrant has provided the results of 22 soybean trials, conducted in Regions 2 (5 trials), 4 (6 trials), and 5 (11 trials). These results support the proposed tolerance of 0.05 ppm for the combined residues of sulfentrazone and 3-hydroxymethyl sulfentrazone in/on soybean seed.

Magnitude of the Residue- Processed Fractions

A soybean processing study (MRID# 432782-02) was reviewed in conjunction with the EUP (Memo, G. Kramer 9/1/94). This processing study was determined to be adequate for the permanent tolerance petition provided that storage stability of 3-hydroxymethyl sulfentrazone is demonstrated. The storage interval for the RAC samples, 5 months, is represented in the storage stability study submitted with this petition. The processed commodities were analyzed within 30 days of preparation. Storage stability has thus been demonstrated for the purposes of the processing study.

Conclusions: Sulfentrazone and 3-hydroxymethyl sulfentrazone residues do not appear to concentrate in processed commodities. Feed/feed additive tolerances for sulfentrazone and 3-hydroxymethyl sulfentrazone are thus not required for this petition. If, however, metabolites other than 3-hydroxymethyl sulfentrazone are determined to be of regulatory significance, then residue data for soybean processed fractions will be required for all such

metabolites. CBTS has determined that a tolerance for aspirated grain fractions is not required as the observed residues in 'grain dust' appear to be the result of soil contamination (Memo, G. Kramer 9/1/94). The proposed tolerance for aspirated grain fractions should thus be withdrawn.

Magnitude of the Residue- Ruminants

The maximum theoretical dietary burden associated with soybeans is 0.011 ppm:

Feed Item	% Diet	Proposed Tolerance	% DM	ppm in Diet
Seed	20	0.05 ppm	89	0.011

Note that aspirated grain fractions were not included in this diet as CBTS has concluded that a tolerance on this RAC is not required (Memo, G. Kramer 9/1/94). Based on the results of the ruminant metabolism study, the registrant claims that a conventional feeding study is not required. The dietary burden used in the phenyl-labelled study, 4.9 ppm, is an exaggeration of 445X based on a maximum dietary burden of 0.011 ppm. The maximum tissue residue observed at this level was 0.013 ppm in kidney. However, CBTS has concluded that rotational crop tolerances are required to support rotation to wheat with a 4 month plantback interval. Based on the limited residue data, the theoretical maximum dietary burden associated with rotational wheat would be at least 0.30 ppm:

Feed Item	% Diet	Proposed Tolerance	% DM	ppm in Diet
Wheat Forage	65	0.10 ppm	25	0.26
Wheat Grain	35	0.10 ppm	89	0.04
Total	100			**0.30**

Considering this dietary burden, the ruminant metabolism feeding level represented only a 16X exaggerated rate. CBTS thus concludes that a conventional ruminant feeding study will be required in order to support the establishment of rotational crop tolerances on wheat RACs. Alternatively, the registrant may choose to withdraw the proposed tolerances for wheat RACs and include a prohibition against rotation to wheat on the sulfentrazone labels. Due to the minimal transfer of residues at 445X, CBTS concludes that, <u>based on the soybean seed tolerance only</u>, a conventional feeding study is not required. If in the future, the registrant wishes to propose tolerances for soybean forage and hay, this conclusion will be reevaluated.

Magnitude of the Residue- Poultry

The maximum theoretical dietary burden associated with soybeans is 0.02 ppm:

Feed Item	% Diet	Proposed Tolerance*	ppm in Diet
Meal	40	0.05 ppm	0.02

*Covered by RAC tolerance

Based on the results of the poultry metabolism study, the registrant claims that a conventional feeding study is not required. The dietary burden used in the metabolism studies, 4.7 ppm, is an exaggeration of 235X based on a maximum dietary burden of 0.02 ppm. The maximum tissue residue observed at this level was 0.03 ppm in kidney. However, CBTS has concluded that rotational crop tolerances are required to support rotation to wheat with a 4 month plantback interval. Based on the limited residue data, the theoretical maximum dietary burden associated with rotational wheat would be at least 0.09 ppm:

Feed Item	% Diet	Proposed Tolerance	ppm in Diet
Wheat Forage	82	0.10 ppm	0.082
Soybean Seed	18	0.05 ppm	0.009
Total	100		**0.091**

Considering this dietary burden, the feeding level in the poultry metabolism study represented a 52X exaggerated rate. CBTS concludes that a conventional feeding study is not required. However, for any future petition which results in a higher dietary burden, CBTS may require that a poultry feeding study be performed.

cc (with attachment): PP#4F04407, Kramer, R.F.
cc (without attachment): circ.
RDI: R.B. Perfetti (3/22/95), M.T. Flood (3/28/95)
G.F. Kramer:804T:CM#2:(703)305-5079:7509C

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Attachment: Page 1 of 1

INTERNATIONAL RESIDUE LIMIT STATUS

CHEMICAL Sulfentrazone*

CODEX NO. _____

CODEX STATUS:

[✓] No Codex Proposal Step 6 or Above

Residue (if Step 8): _____

PROPOSED U.S. TOLERANCES:

Petition No. 4F04407

CBTS Reviewer G.F. Kramer

Residue: Parent plus Hydroxymethyl-Sulfentrazone$

Crop(s)	Limit (mg/KG)
Soybeans	0.05
Aspirated Grain Fractions	0.05

CANADIAN LIMITS:

[✓] No Canadian Limits

Residue: _____

Crop(s)	Limit (mg/KG)

MEXICAN LIMITS:

[✓] No Mexican Limits

Residue: _____

Crop(s)	Limit (mg/KG)

NOTES

*FMC 97285 (F6285, N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-methyl-5-oxo-1H-1,2,4-triazol-1-yl]phenyl]methanesulfonamide.

$N-[2,4-dichloro-5-[4-(difluoromethyl)-4,5-dihydro-3-hydroxymethyl-5-oxo-1H-1,2,4-triazol-1-yl]phenyl]methanesulfonamide

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