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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460OFFICE OF  
PESTICIDES AND TOXIC  
SUBSTANCES

JAN 4 1991

MEMORANDUM

SUBJECT: PP# 9F3796 Sulfosate (Touchdown<sup>TM</sup>) in or on corn grain, forage and fodder. Evaluation of analytical methods and residue data.

MRID No. 412099-09, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19  
412359-04  
412360-05

DEB No. 6200, 6201, 6202  
HED# 0-0473

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Tolerance Petition Section I  
Chemistry Branch I: Tolerance Support  
Health Effects Division (H7509C)

TO: Robert Taylor/Cynthia Giles, PM25  
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and

Toxicology Branch II  
Herbicides, Fungicides and Antimicrobial Support  
Health Effects Division (H7509C)

THRU: Richard D. Schmitt, Ph.D., Chief  
Chemistry Branch I: Tolerance Support *R. Loranger for*  
Health Effects Division (H7509C)

ICI Americas Inc., Agricultural Products has proposed the establishment of tolerances for the combined residues of the herbicide sulfosate (Touchdown<sup>TM</sup>), N-phosphonomethylglycine, (carboxymethylamino methyl phosphonate) and its metabolite, AMPA (aminomethylphosphonic acid) (calculated as the herbicide) in or on: corn grain @ 0.1 ppm, corn forage @ 0.2 ppm and corn fodder @ 0.2 ppm.

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Sulfosate is a NEW herbicide; there are no established tolerances for the trimethylsulfonium salt of glyphosate. Tolerances have been established for the isopropylamine (Roundup® herbicide) and sodium sesqui salts of glyphosate under 40 CFR 180.364 for grain crops and forage grasses (which included corn grain, fodder and forage under the 40 CFR 180.34 definition prior to 1983). Sulfosate is similar in chemical structure, metabolic breakdown and proposed use to glyphosate.

#### CONCLUSIONS/DEFICIENCIES

1. The product chemistry and manufacture of sulfosate was previously reviewed and found to be adequate by Registration Division when sulfosate was first submitted as a non-food use chemical.
- 2a. The proposed labels recommend the use of ammonium sulfate as an adjuvant. This recommendation must either be deleted from the labels and a revised Section B be submitted or residue data must be generated supporting the use of this adjuvant with sulfosate.
- 2b. The proposed labels are unclear as to the number and type of applications allowed. The labels state that only one application is allowed. Does this mean one pre-emergent or one spot application or one of each is allowed as long as one does not exceed the 4 lbs a.i./A/yr? A revised Section B is required.
- 3a. The corn plant metabolism study submitted for the cation is inadequate to determine the nature of the residue. Additional detail is needed in order to properly evaluate the study. The reviewer should have enough detail to duplicate the calculations used to determine the concentrations of the residues.
- 3b. The corn plant metabolism study submitted for the anion is inadequate to determine the nature of the residue. Additional detail is needed in order to properly evaluate the study. The reviewer should have enough detail to duplicate the calculations used to determine the concentrations of the residues.
- 3c. A soybean plant metabolism study was also submitted with this petition. Although an involved evaluation was not performed, the overall report is superior to the corn metabolism studies in the amount of detail presented. Some details for sample sizes are lacking. A detailed evaluation is being conducted in a current petition for soybeans.
- 4a. Subject to successful completion of the Agency method validation trials, CBTS would consider the methods for the anionic and cationic moieties of sulfosate to be adequate for corn only.

- 4b. Because small residues may be carried into feed items, it is necessary to have available an enforcement method for residues in meat, milk, poultry and eggs. The submitted methods were not validated with these commodities. Both petitioner and independent method validation are required for these commodities.
- 5a. Twelve residue field trials from ten states were submitted all from one growing season. CBTS does not consider either geographical or total representation to be adequate. Additional field trials from the Northeast corn growing region (PA and NY) are required. Residue field trials for each represented state from an additional growing season are also required.
- 5b. Revised labels (Section B) requiring a PHI of 90 days for grain and fodder are required. This will preclude late spot treatments that may raise residue levels. The primary source of residues appears to be the spot treatments.
6. Storage stability data were not submitted for corn, but data from sorghum grain, soybeans, soybean straw and wheat grain were considered to be adequate to be translated to corn. Contingent upon the successful completion of the above required method trials, CBTS concludes that no appreciable losses of the residues of concern occur in frozen storage over a two year period.
7. CBTS finds the corn processing study to be adequate contingent upon no new residues being required to be regulated from the crop metabolism studies and the successful completion of the method validation trials. A food additive tolerance would not be required with the present processing study.
8. Should the application rate or methods be changed in the future or if the results of the required residue studies are not consistent with the old, CBTS may require a new processing study be submitted reflecting these changes. If a new processing study should exhibit concentration of the residue, a food additive tolerance would be required.
9. The submitted laying hen metabolism anion study is inadequate. None of the residues present in tissues have any kind of identification attempt. No attempts were made to determine the nature of the residue in eggs. Further characterization of the nature of the residues in the tissues of laying hens and eggs is required. Although CBTS recognizes the difficulty in determining the nature of residues from a compound that is so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various edible tissues. CBTS normally requires the identification of 90% of the absorbed residues.
10. Feeding studies are required to be carried out for 30 days

on poultry and eggs using unlabeled material and the required analytical method for the anion in order to determine if residues may build up over time. It is CBTS policy that feeding studies should be continued for at least four weeks.

11. The submitted laying hen metabolism cation study is inadequate. None of the residues present in tissues have any kind of identification attempt. No attempts were made to determine the nature of the residue in eggs. Further characterization of the nature of the residues in the tissues of laying hens and eggs is required. Although CBTS recognizes the difficulty in determining the nature of residues from a compound that is so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various edible tissues. CBTS normally requires the identification of 90% of the absorbed residues.

12. Feeding studies are required to be carried out for 30 days on poultry and eggs using unlabeled material and the required analytical method for the cation in order to determine if residues may build up over time. It is CBTS policy that feeding studies should be continued for at least four weeks.

13. The goat anion metabolism study is inadequate. Although CBTS recognizes the difficulty in determining the nature of residues from a compound that is so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces, urine and gut contents) but of that present in the various edible tissues. CBTS normally requires the identification of 90% of the absorbed residues. In this case, only the residues present in kidney have any kind of identification attempt. This is approximately 30% of the residue absorbed into the 100x goat. No attempts were made to determine the nature of the residue in milk.

14. Dairy cattle feeding studies are required to be carried out for 30 days on meat and milk using unlabeled material and the required analytical method for the anion in order to determine if residues may build up over time. It is CBTS policy that feeding studies should be continued for at least four weeks.

15. The goat cation metabolism study is inadequate. Although CBTS recognizes the difficulty in determining the nature of residues from a compound that is so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various edible tissues. CBTS normally requires the identification of 90% of the absorbed residues. In this case, only the residues present in liver have any kind of identification attempt. This is approximately 9% of the residue present in the goat. No attempts were made to determine the

nature of the residue in milk.

16. Dairy cattle feeding studies are required to be carried out for 30 days on meat and milk using unlabeled material and the required analytical method for the anion in order to determine if residues may build up over time. It is CBTS policy that feeding studies should be continued for at least four weeks.

17. An "International Residue Status" sheet is attached. There are no Canadian, Mexican or Codex tolerances for Sulfosate on or in corn. There are Codex limits of 0.1 ppm for corn grain and 100 ppm for corn fodder for glyphosate per se. There is also a Canadian negligible residue type limit of 0.1 ppm for glyphosate per se for "all food crops". There are compatibility problems associated with corn grain and corn fodder due to differences in the residue being regulated (i.e., U.S. tolerance includes the AMPA metabolite).

18. A report on the behavior of TMS, CMP and AMPA in the EPA multiresidue protocols I, II, III and IV, submitted with this petition has been forwarded to the FDA for review.

### RECOMMENDATIONS

CBTS recommends against the requested tolerances because of conclusions 2a, 2b, 3a, 3b, 4b, 5a, 5b, 8, 9, 10, 11, 12, 13 14, 15 and 16.

### DETAILED CONSIDERATIONS

#### MANUFACTURE AND FORMULATION (MRID# 409430-00,01,02,03)

The product chemistry and manufacture was previously reviewed and found to be adequate (memo, 3/17/87, K. Leifer) by Registration Division when sulfosate was first submitted as a non-food use chemical. A letter stating that no additional product chemistry data was necessary was sent (2/15/89, R. Taylor) to the petitioner.

Two formulations are proposed for use:

Touchdown® 4-LC, a 40% a.i. liquid concentrate. Each gallon contains 4 lbs a.i.

Touchdown™ Concentrate a 52.2% a.i. liquid concentrate. Each gallon contains 5.5 lbs a.i.

**PROPOSED USE (MRID# 409430-00)**

Touchdown® is a nonselective systemic herbicide proposed for pre-emergent and spot weed control on corn fields. This formulation is currently approved for use in noncrop areas to control unwanted vegetation.

Touchdown® requires a six hour rain free period after application. Rain occurring within six hours of application may reduce weed control.

Do not apply by aircraft. Do not apply this product through any type of irrigation system.

Touchdown® should be applied to actively growing emerged weeds when they are small. Weeds 6" in height are easiest to control.

A surfactant or wetting agent (approved for crop usage) is required to improve coverage of weed foliage. All surfactants or wetting agents should contain at least 50% active ingredient.

The use of ammonium sulfate is proposed to improve control of weeds. The petition for a tolerance is for the trimethylsulfonium salt and not the ammonium salt. Use of different salts would require additional residue data. When sulfosate is premixed with ammonium sulfate, the ammonium salt will result. CBTS requires this use be removed from the label or that residue data be generated using ammonium sulfate mixed with sulfosate.

Broadcast application of Touchdown® should be made in 10 to 30 gallons of water per acre with conventional spray equipment or in 3 to 10 gallons of water with low volume equipment. Increase volumes if foliage is dense.

Spot applications should use a 1 to 3% a.i. solution of Touchdown® in water. Spray the solution on actively growing foliage until uniformly wet, but not to the point of runoff. Retreat if necessary.

The labels state that wiper applications may be used by mixing 2 gallons Touchdown®-4LC or 2.5 quarts of Touchdown® Concentrate in 2 gallons of water. Apply this mixture to weeds while avoiding contact with desirable vegetation. For improved control, make two applications in opposite directions.

The petitioner supplies no residue data to support a wiper application. CBTS requires a revised label explicitly stating that wiper applications are not allowed.

The following statement appears on the proposed labels in various places for corn:

Do not make more than one application or exceed a total of 4 lbs Touchdown® (4 quarts for 4-LC and 5.8 pints for Concentrate) per acre per year.

It is required for the label to be clarified as to whether this refers to one spot treatment or one pre-emergent treatment or one of each.

Do not plant rotational crops other than corn within one year in a field treated with Touchdown® 4-LC.

CBTS finds the label inadequate. The use of ammonium sulfate is required to be deleted or residue data generated using ammonium sulfate. The proposed wiper use is required to be deleted from the label unless the petitioner supplies residue data for review from wiper applications supporting this use. Additionally, the number of proposed possible types and number of applications per year is unclear. No PHI is proposed on the label. A PHI of 90 days for grain and fodder is required. This will preclude late spot treatments that may raise residue levels. The primary source of residues appears to be the spot treatments. A revised Section B is required.

#### NATURE OF THE RESIDUE (MRID# 412099-9,10)

Two separate metabolism studies were carried out in corn to determine the nature of the residue from both the anionic and cationic portions of the parent molecule. In both studies, the radiolabeled compounds were isotopically diluted with unlabeled material. The purity of this cold material is listed as 56.6% in both studies. Since the final purity of the material used in the studies was >95%, it would appear that the petitioner means that the 56.6% pure a.i. number is pure material diluted with water. This point needs to be clarified.

An aqueous stock solution of 50 ml containing 35 mg of <sup>14</sup>C-trimethylsulfonium carboxymethylaminomethyl phosphonate ([<sup>14</sup>C-TMS]ICIA0224 Specific Activity 20 mCi/mmol) mixed with 226.5 mg of unlabeled a.i. was prepared and 49 ml applied to a container sown with 25 corn seeds at a rate corresponding to an application rate of 4.36 lb/A (4.89 kg/ha). The remaining 1 ml of stock was assayed for purity and concentration. The corn was germinated and grown outdoors for 14 weeks and then transferred to a greenhouse for the additional 8 weeks needed for maturation. The plants were watered each day and fertilized at biweekly intervals. Diazinon insecticide was applied as needed to control ants and aphids.

Samples were harvested at 33, 48 and 154 days after treatment. Both the 33 and 48 day samples were immature plants. No data on the number of plants or weight of the individual samples was provided. Each plant was harvested by cutting the stem approximately 2 cm above the soil. No root samples were taken. The immature samples were divided into stems and leaves. The mature plants were divided into stems, leaves and tassels, husks and silks, kernels, and cobs and shanks. The samples were all frozen in liquid nitrogen and then homogenized in a food processor until a coarse powder was formed. Ten core soil samples were taken from each container. No details as to when these soil samples were taken were submitted. The ten samples were combined and mixed thoroughly. Representative portions of all samples (200-300 mg) were weighed and combusted for radioanalysis. The results of the combustion analysis are listed in Table I. The remaining material was stored at -20°C until further analysis.

Grain samples were further ground to a powder with liquid nitrogen to prevent thawing. The starch was extracted from triplicate aliquots (20 g each) with 87% aqueous DMSO and the layers separated by centrifugation. This extraction was repeated two more times. The remaining pulp was extracted with twice with toluene and twice with 0.5M  $\text{NH}_4\text{HCO}_3$ . It is unclear in the writeup whether these extractions are combined with the DMSO extractions. This point needs to be clarified.

TABLE I.  
Location  $^{14}\text{C}$ -Trimethylsulfonium Metabolism Residues  
SAMPLE ppm TMS equivalents

33 days	
Leaves	0.39
Stems	0.12
Average	0.26
48 days	
Leaves	0.11
Stems	0.024
Average	0.06
154 days (mature)	
Kernels	0.065
Leaves & Tassels	0.10
Stems	0.015
Husks and Silks	0.024
Cobs & Shanks	0.016
Average	0.044
Soil	0.14



Ethanol was added to the DMSO extracts to precipitate the starch and the starch collected by filtration. The starch fractions were combined and dried. Starch (10 g) was hydrolyzed with HCl two separate times and the remaining pulp removed by centrifugation. The glucose formed in the hydrolysis was derivatized to form glucose phenylhydrazone or glucosazone. An aliquot was removed for combustion and LSC analysis and the remaining sample crystallized until constant specific activity. The quantities of sample used in the derivatization as well as the final amount of glucosazone isolated were not given and are required. Confirmation of glucosazone identity was provided by melting point and nmr and mass spectral data. Overall, 83 % of the radioactivity associated with the grain can be accounted for as being incorporated into the glucose of starch.

Frozen leaf samples were further ground in a mortar and pestle with liquid nitrogen to prevent thawing. The tissue samples were extracted with various solvents. Few details are given and are required, such as the identity of the solvents used and the extraction scheme followed. Approximately 32 to 34% of all residues from all samples were extractable and 52 to 65% were bound.

TABLE II.  
[<sup>14</sup>C-TMS]ICIA0224 in Grain  
% of total tissue <sup>14</sup>C

Sample		ppm TMS
Starch	83	0.058
Unknown Aqueous	1.6	0.0011
Unknown Organic	2.6	0.0018
Unknown Bound	11	0.0078
total	98.2	

Two other attempted extraction schemes were followed, both were variations on the standard analytical method presented below. In each case, no sample sizes or weights are given and the residues remain essentially uncharacterized.

Other frozen leaf samples were extracted with either water or EDTA solution. No significant change in extractability was observed. Approximately 27% of the radioactivity was extractable with both the 33 and 48 day samples. No TMS moiety was detected in the 48 and 154 day samples, but the 33 day sample appeared to have a quantity near the limit of detection of 0.0008 ppm. Attempts to characterize the residues are poorly documented in this submission. No quantitative or raw results are given for the incorporation into starch, although these results are mentioned in the submission. No details on any of the individual experiments are given. The quantities or weights of samples used are not given. As a result, it is not possible to determine the

quality of the attempts to characterize the residues in this study. The registrant was able to characterize 0.065 ppm in starch from grain but nothing out of the 0.39 ppm present in leaves. Additional detail and explanation is required.

In the petitioner's table of recoveries of radiolabel from mature leaf pulp, the overall recoveries of  $^{14}\text{C}$  from their hydrolysis scheme are listed as 96 and 85% of the bound residues. No details on sample size or composition are given. Additional detail is required in order to evaluate the study. In all, only the 25% of the total residues present in grain appear to be well characterized. It appears that little characterization was performed after hydrolysis. Additional characterization of the hydrolyzed fractions is required.

A hydroponic study was also submitted. Corn plants were grown hydroponically in a nutrient solution containing 0.5  $\mu\text{g/ml}$  of [ $^{14}\text{C}$ -TMS]ICIA0224 for 21 days. Of the radioactivity taken up into the plant, 90% was water extractable and 97 to 100% was identified as unchanged TMS. Evidently, a three week time frame is too short a period for any extensive metabolism to occur in the plant itself. This is in sharp contrast to the previous study, where virtually none of the radioactivity could be accounted for as starting material. Most of the metabolism appears to occur in the soil prior to uptake by the plant. Little if any, metabolism occurs in the plant in this short time.

More detailed data as explained above are necessary before CBTS can rule on the nature of the residue from the cationic moiety in corn. In particular, data on the sizes of analyzed samples and explanations of where all the numbers in the tables are derived are required. Additional attempts are required to characterize the residues in the plant since only about 25% of the total residues can be considered to be characterized. CBTS normally requires characterization of 90% of the residues. CBTS finds the data on the nature of the residue from the cationic moiety of sulfosate to be inadequate.

An aqueous stock solution of 50 ml containing 81.8 mg of trimethylsulfonium carboxymethylamino[ $^{14}\text{C}$ -methyl]phosphonate ([ $^{14}\text{C}$ -CMPA]ICIA0224 Specific Activity 9.8 mCi/mmol) mixed with 180.2 mg of unlabeled a.i. was prepared and 49 ml applied to a container sown with 25 corn seeds at a rate corresponding to 4.57 lb/A (5.13 kg/ha). The remaining 1 ml of stock was assayed for purity and concentration. The corn was germinated and grown outdoors for 14 weeks and then transferred to a greenhouse for the additional 8 weeks needed for maturation. The plants were watered each day and fertilized at biweekly intervals. Diazinon insecticide was applied as needed to control ants and aphids.

Samples were harvested at 33, 48 and 154 days after treatment. Both the 33 and 48 day samples were immature plants. No data on the number of plants or weight of the individual samples was provided. Each plant was harvested by cutting the stem approximately 2 cm above the soil. No root samples were taken. The immature samples were divided into stems and leaves. The mature plants were divided into stems, leaves and tassels, husks and silks, kernels, and cobs and shanks. The samples were all frozen in liquid nitrogen and then homogenized in a food processor until a coarse powder was formed. Ten core soil samples were taken from each container. No details as to when these soil samples were taken were submitted. The ten samples were combined and mixed thoroughly. Representative portions of all samples (200-300 mg) were weighed and combusted for radioanalysis. The results of the combustion analysis are listed in Table III. The remaining material was stored at -20°C until further analysis.

Grain samples were further ground to a powder with liquid nitrogen to prevent thawing. The starch was extracted from triplicate aliquots (20 g each) with 87% aqueous DMSO and the layers separated by centrifugation. This extraction was repeated two more times. The remaining pulp was extracted twice with toluene and twice with 0.5M  $\text{NH}_4\text{HCO}_3$ . It is unclear in the writeup whether these extractions are combined with the DMSO extractions. This point needs to be clarified.

TABLE III.  
Location  $^{14}\text{C}$ -Anion Metabolism Residues  
SAMPLE ppm CMA equivalents

33 days	
Leaves	0.38
Stems	0.23
Average	0.30
48 days	
Leaves	0.15
Stems	0.066
Average	0.10
154 days (mature)	
Kernels	0.39
Leaves & Tassels	0.67
Stems	0.13
Husks and Silks	0.52
Cobs & Shanks	0.17
Average	0.37
Soil	2.0

Ethanol was added to the DMSO extracts to precipitate the starch and the starch collected by filtration. The starch fractions were combined and dried. Starch (10 g) was hydrolyzed with HCl two separate times and the remaining pulp removed by centrifugation. The glucose formed in the hydrolysis was derivatized to form glucose phenylhydrazone or glucosazone. An aliquot was removed for combustion and LSC analysis and the remaining sample crystallized until constant specific activity. The final amount of glucosazone isolated was not given and is required. Confirmation of glucosazone identity was provided by melting point and nmr and mass spectral data. Overall, 83.7 % of the radioactivity associated with the grain can be accounted for as being incorporated into the glucose of starch.

TABLE IV.  
[<sup>14</sup>C-CMPA]ICIA0224 in Grain  
% of total tissue <sup>14</sup>C

Sample	% of total tissue <sup>14</sup> C	ppm CMPA
Starch	83.7	0.324
Unknown Aqueous	3.2	0.012
Unknown Organic	2.0	0.008
Unknown Bound	<u>13.7</u>	0.053
total	102.4	

Leaf samples from each of the three sampling intervals were extracted with water (3 times), methanol (three times) and hexane (2 times). This resulted in 42%, 2% and 2% respectively of the available <sup>14</sup>C recovered in each soluble fraction for the 33 day sample. Gradually decreasing amounts remained bound as the plants matured, but distribution in the three extractions remained relatively constant. Further attempts at characterization of these residues by tlc, hplc, gel permeation and ion exchange were successful only in demonstrating only a minor portion (approximately 0.4 to 0.9 ppb) of the residues corresponded to CMPA or AMPA standards.

In the petitioner's table of recoveries of radiolabel from mature leaf pulp, the overall recoveries of <sup>14</sup>C from their hydrolysis scheme are listed as 94 and 83% of the bound residues. Approximately 11% is said to be in crude cellulose as glucose. No details as to how this figure was arrived at are given. No details on sample size or composition are given. Additional detail is required in order to evaluate the study. Only the grain residues are adequately characterized. This corresponds to approximately 17% of the total residues. Additional characterization of the residues of the hydrolyzed fractions is required.

A hydroponic study was also submitted. Corn plants were grown hydroponically in a nutrient solution containing 1.3 ppm of [<sup>14</sup>C-CMPA]ICIA0224 for 14 days. Of the radioactivity taken up into

the plant, 97% was water extractable and at least 88% was identified as unchanged CMPA. Evidently, a two week time frame is too short a period for any extensive metabolism to occur in the plant itself. This is in sharp contrast to the previous study, where virtually none of the radioactivity could be accounted for as starting material. Most of the metabolism appears to occur in the soil prior to uptake by the plant. Little, if any metabolism occurs in the plant.

More detailed data as explained above are necessary before CBTS can rule on the nature of the residue from the anionic moiety in corn. In particular, data on the sizes of analyzed samples and explanations of where all the numbers in the tables are derived are required. CBTS finds the data on the nature of the residue from the anionic moiety of sulfosate to be inadequate.

The petitioner might want to use as an example the soybean metabolism report WRC 89-24 which was also submitted along with this petition and still lacking characterization of the residues. This study although not relevant to this petition, contains considerably more detail as to methods, sample sizes, dpm per sample, etc. Although it also is lacking in some details, it is by far superior to either of these submissions. Enough detail should be present in the reports for the reviewer to duplicate all the calculations required to arrive at the final ppm of residues reported for each fraction. Characterization of the residues after hydrolysis would be required.

**ANALYTICAL METHOD** (MRID# 412099-16,17,18 412359-04,05 )

Two analytical methods were submitted for the analysis of the residues of sulfosate, one each for the cationic and anionic portions of the compound. Both were developed at ICI's Western Regional Center and are report numbers: WRC 85-33 (cation) and WRC 85-34R (anion). Both methods have been submitted for a method trial and all subsequent conclusions are subject to a successful outcome of these trials.

The method used to obtain residue data on the trimethylsulfonium cation was ICI's WRC 85-33R: Determination of SC-0224 Cation Residues in Crops, Water and Soil by Gas Chromatography. The detection limits are listed as 0.05 ppm in water, soil and crops and 0.1 ppm for animal feed and forage products.

Briefly, crops are extracted with water with conditions dependent on the water and oil content of that crop. Soil samples are extracted with aqueous potassium hydroxide. The aqueous extracts of all samples may be acidified as in the determination of the anions. Crop samples that have an apparent measurable background

level of trimethylsulfonium (TMS) or dimethylsulfide (DMS) are eluted through a weak cation exchange resin to lower the background. A secondary clean-up step involving elution through a strong cation exchange resin is necessary for those crops that still have an apparent high background level.

The eluate is concentrated to almost dryness by heating and then dealkylated with base at 100°C for 1 hour. The samples are then ready for gas chromatographic analysis. Samples that still have an apparent high background level (>0.05 ppm) are eluted through CC-4 adsorbent prior to chromatography.

Various gas chromatography conditions can be used depending on the columns and type of chromatograph (capillary or packed column) available. Detection is by a flame photometric detector operated in the sulfur mode.

Method validation for corn and corn products was accomplished by fortification with TMS in each crop fraction (see Table III.) and measuring recoveries. Recoveries in corn ranged from 69 to 111% with an average of 97%. Additional crops as well as any animal feeds derived from these crops that were tested include: alfalfa, almonds, apples, apricot, avocado, Bahia grass, bananas, barley, beets (red), Bermuda grass, broccoli, carrots, cherries, clover, coffee beans (green & roasted), cotton, figs, grapes, grapefruit, mangoes, nectarines, oats, oranges, papayas, peaches, pears, pecans, pistachios, plantains, plums, rice, soybeans, sugar beets, sugarcane, sunflower, tangerines, walnuts and wheat. Overall for crops, recoveries ranged from 67 to 125 % with an average of 98.7%. For soil samples, recoveries ranged from 74 to 115 % with an average of 91.5%.

TABLE III.  
Corn Fortification Studies with TMS

Crop	ppm added	ppm background	% recovery
Corn grain (field)	0.23	0	110
	0.23	0	110
Corn Ears (sweet)	0.092	0.013	111
	0.23	0	110
Corn Stover	0.46	0.061	107
	0.46	0	69
	0.092	0	87
	0.23	0.042	109
Corn Meal	0.092	0	104
Corn, Crude Oil	0.092	0	93
Corn, Refined Oil	0.092	0	84
Corn, Soapstock	0.46	0	85
Corn, Silage	0.184	0.038	84

Because residues are present in feed items and animal commodity tolerances are needed items, it is necessary to have an enforcement method for residues in meat, milk, poultry and eggs. The submitted methods were not validated with these commodities. Both petitioner and independent method confirmatory trials are required for these commodities.

Contingent upon the successful completion of the method trials, CBTS considers the method to be adequate for the trimethylsulfonium moiety in crops.

The method used to obtain residue data on the sulfosate anion was ICI's WRC 85-34R: Determination of SC-0224 Anion Residues in Crops, Water and Soil by Liquid Chromatography. The detection limits are listed for CMP and AMPA as 0.01 ppm in water, 0.05 ppm in soil and crops and 0.1 ppm for animal feed and forage products.

Briefly, CMP and AMPA are extracted from crop samples with water under conditions dependent on the water and oil content of the crop and from soil samples with  $\text{NH}_4\text{OH}$ . After extraction, the samples are acidified and crop samples are eluted through a cation exchange resin column. All samples are then evaporated to dryness at  $50^\circ\text{C}$ .

Residues are derivatized with 9-fluorenylmethyl chloroformate in borate buffer, giving fluorescent derivatives. The excess reagent is extracted with ethyl acetate. The residues are chromatographed on hplc using an anion exchange column and fluorescence detection. Regular washings of the hplc column are recommended.

Method validation for corn and corn products was accomplished by fortification with CMP and AMPA in each crop fraction (see Table IV.) and measuring recoveries. Recoveries in corn ranged from 74 to 105% with an average of 92.1% for CMP and from 73 to 102% with an average of 87.3% for AMPA. Additional crops as well as any animal feeds derived from these crops that were tested include: alfalfa, almonds, apples, apricot, avocado, Bahia grass, bananas, barley, beets (red), Bermuda grass, broccoli, carrots, cherries, clover, coffee beans (green & roasted), cotton, figs, grapes, grapefruit, mangoes, nectarines, oats, oranges, papayas, peaches, pears, pecans, pistachios, plantains, plums, rice, soybeans, sugar beets, sugarcane, sunflower, tangerines, walnuts and wheat. Overall for crops, recoveries ranged from 61 to 126% with an average of 94.2% for CMP and from 65 to 135% with an average of 90.5% for AMPA. For soil samples, recoveries ranged from 61 to 111% with an average of 87.1% for CMP and from 79 to 92% with an average of 88.2% for AMPA.

Because residues are present in feed items and animal commodity

tolerances are needed, it is necessary to have an enforcement method for residues in meat, milk, poultry and eggs. The submitted methods were not validated with these commodities. Both petitioner and independent method confirmatory trials are required for these commodities.

A report on the behavior of TMS, CMP and AMPA (MRID# 412099-15) in EPA multiresidue protocols I, II, III and IV, submitted with this petition has been forwarded to the FDA for review (10/29/90, S. Koepke).

Contingent upon the successful completion of the method trials, CBTS considers the method for the anionic moiety of sulfosate to be adequate for corn only.

**TABLE IV.**  
**Corn Fortification Studies**

Crop	ppm added each	ppm background		% recovery	
		CMP	AMPA	CMP	AMPA
Corn grain (field)	0.5	0.0	0.0	92	91
	0.5	0.0	0.0	74	75
Corn Ears (sweet)	0.2	0.0	0.0	92	90
Corn Stover	0.2	0.0	0.0	83	87
	0.5	0.0	0.0	80	94
	1.0	0.1	0.0	104	99
	1.0	0.0	0.0	85	78
Corn Meal	0.2	0.0	0.0	105	75
Corn Silage	0.2	0.0	0.0	93	76
Corn, Crude Oil	0.4	0.02	0.0	102	102
Corn, Refined Oil	0.2	0.0	0.0	97	91
Corn, Soapstock	0.2	0.0	0.01	98	91

**RESIDUE DATA** (MRID# 412099-17,18)

Twelve field trials were carried out in ten different states (California, South Dakota, Nebraska, Minnesota, Iowa[3], Missouri, Wisconsin, Illinois[3], Kentucky and North Carolina) in 1988 using sulfosate on corn (See Figure 1). These states correspond to 69% of all corn production for in the United States (See Figure 2 for areas of major corn production) but only 45% of



corn production for silage and forage (See Figure 3) (Agricultural Statistics 1986). The Northeast corn growing region (New York and Pennsylvania) and Idaho are not represented. CBTS does not consider the overall geographical representation to be adequate. Additional field trials covering the Northeast growing region for corn (PA and NY) are required. CBTS does not consider the total representation to be adequate. Additional field trials for each state are required for an additional growing season.

Ten trials were protocolled as magnitude-of-the-residue and augmented by two processing trials. All 12 field trials utilized either 2½ or 6½ times the recommended maximum application rate of 4 lbs a.i./A/year. Each plot received two applications of sulfosate. The first was applied broadcast at the rate of either 8 or 24 lbs a.i./A followed by spot applications of 2 lbs a.i./A. No trials were performed using a wiper application. Samples were frozen and shipped still frozen to ICI's Western Research Center for later analysis. All samples were analyzed anywhere from 67 to 256 days after harvest.

Residue data for wiper use using a standard measured drip rate and tractor speed or a revised Section B deleting the wiper use from the label is required.

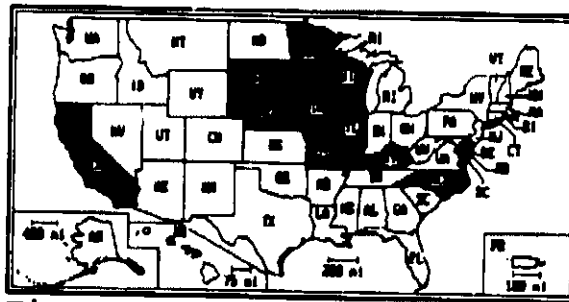


Figure 1 States with residue data.

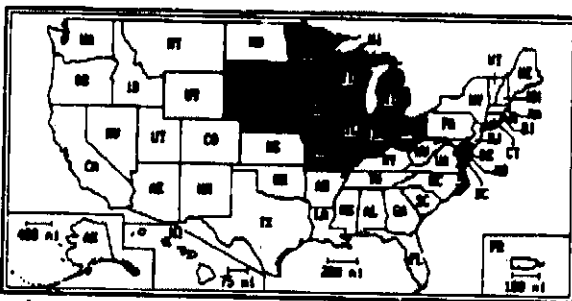


Figure 2 States producing more than 200,000 (1000 bushels) of corn per year.

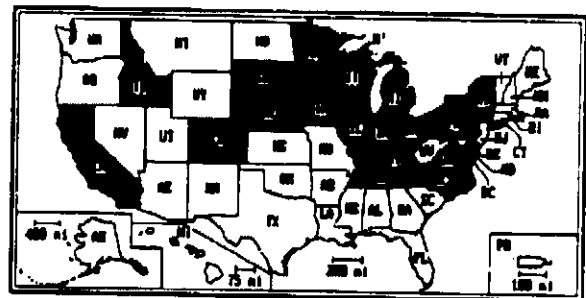


Figure 3 States producing more than 2000 (1000 tons) of corn for silage and forage.

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Only two residue values for TMS and two for CMP were above the limits of detection (Table V.). There were no detectible residues for AMPA. PHI's ranged from 23 to 72 days for forage and from 62 to 121 days for grain and fodder. There appears to be no correlation either between PHI or level of pre-emergent treatment and the level of the residue.

It should be noted that the PHI's in Table V were calculated from the last spot treatment. These data support the imposition of a 90 day PHI. The majority of the data were obtained with PHI's of this length or longer. Any applications after this time may result in higher residues. A revised label specifying a 90 day PHI for corn grain and fodder is required.

Contingent upon the completion of the required analytical method trials, the residue data were successfully validated by the fortification of control samples with 0.1 ppm for forage, 0.1 ppm for fodder and 0.05 ppm for grain with TMS, CMP or AMPA (Table VI.). Recoveries for forage ranged from 79 to 100% for TMS with an average recovery of 85.6%, from 78 to 100% for CMP with an average of 90.0% and from 68 to 95% for AMPA with an average of 79.9%. Recoveries for fodder ranged from 73 to 126% for TMS with an average recovery of 104.5%, from 79 to 140% for CMP with an average of 99.4% and from 53 to 108% for AMPA with an average of 82.5%. Recoveries for grain ranged from 72 to 118% for TMS with an average recovery of 87.8%, from 57 to 108% for CMP with an average of 90.0% and from 70 to 112% for AMPA with an average of 89.1%.

A storage stability study was submitted along with this petition (Table VII). Although corn was not included in this study, CBTS considers that the data available for sorghum, soybeans and wheat are adequate to be translated to corn. Sorghum grain was fortified with 2.3 ppm of TMS, 5.0 ppm of CMP or 5.0 ppm of AMPA and stored frozen for intervals up to 4 years. No losses of analyte greater than experimental error occurred over that time period. Soybeans, soybean straw and wheat grain were fortified with 0.46 ppm of TMS, 1.0 ppm of CMP or 1.0 ppm of AMPA, frozen and stored for intervals up to two years. No losses of analyte greater than experimental error occurred over that time period.

Contingent upon the successful method validation trials for the analytical methods, CBTS considers the storage stability data for the RAC's corn grain, forage and fodder to be adequate.

Corn processing studies were performed by Food Protein Research and Development Center, the Texas A&M University System, College Station, Texas. The processing consisted of a dry-milling and a wet-milling process. The products from each process were frozen and shipped to ICI Americas Western Research Center for analyses.

**Table V.  
Summary of Residue Data**

Location	PHI	Rate (lb a.i. /A)	Sample Type	Magnitude of Residue (ppm)		
				TMS	CMP	AMPA
CA	54	10	Forage	<0.10	<0.10	<0.10
	116		Fodder	<0.10	<0.10	<0.10
	116		Grain	<0.05	<0.05	<0.05
MN	51	10	Forage	<0.10	<0.10	<0.10
	101		Fodder	<0.10	<0.10	<0.10
	101		Grain	<0.05	<0.05	<0.05
NE	56	10	Forage	<0.10	<0.10	<0.10
	89		Fodder	<0.10	<0.10	<0.10
	89		Grain	<0.05	<0.05	<0.05
WI	63	10	Forage	<0.10	<0.10	<0.10
	88		Fodder	<0.10	<0.10	<0.10
	88		Grain	<0.05	<0.05	<0.05
IA	23	10	Forage	<0.10	<0.10	<0.10
	112		Fodder	<0.10	<0.10	<0.10
	112		Grain	<0.05	<0.05	<0.05
MO	56	10	Forage	<0.10	<0.10	<0.10
	98		Fodder	<0.10	<0.10	<0.10
	98		Grain	<0.05	<0.05	<0.05
KY	72	10	Forage	<0.10	<0.10	<0.10
	121		Fodder	<0.10	<0.10	<0.10
	121		Grain	<0.05	<0.05	<0.05
IL	39	10	Forage	<0.10	<0.10	<0.10
	88		Fodder	<0.10	<0.10	<0.10
	88		Grain	<0.05	<0.05	<0.05
NC	37	10	Forage	<0.10	<0.10	<0.10
	72		Fodder	0.131	<0.10	<0.10
	72		Grain	<0.05	<0.05	<0.05
SD	45	10	Forage	<0.10	<0.10	<0.10
	86		Fodder	<0.10	<0.10	<0.10
	86		Grain	<0.05	<0.05	<0.05
IA	88	10	Grain	0.061	0.070	<0.05
	88	26	Grain	<0.05	0.055	<0.05
IL	62	10	Grain	<0.05	<0.05	<0.05
	62	26	Grain	<0.05	<0.05	<0.05

**Table VI.**  
**Summary of Fortification Data**

Location	Amount Standard Added	Sample Type	% Recovery		
			TMS	CMP	AMPA
CA	0.1	Forage	81	90	88
	0.1	Fodder	89	140	84
	0.05	Grain	81	104	80
MN	0.1	Forage	85	80	88
	0.1	Fodder	118	104	70
	0.05	Grain	97	80	112
NE	0.1	Forage	79	99	76
	0.1	Fodder	107	96	108
	0.05	Grain	100	108	80
WI	0.1	Forage	82	100	84
	0.1	Fodder	118	89	75
	0.05	Grain	94	102	80
IA	0.1	Forage	85	87	68
	0.1	Fodder	120	80	53
	0.05	Grain	118	72	97
MO	0.1	Forage	90	95	95
	0.1	Fodder	126	109	84
	0.05	Grain	87	57	93
KY	0.1	Forage	100	78	71
	0.1	Fodder	73	94	85
	0.05	Grain	76	89	70
IL	0.1	Forage	85	87	68
	0.1	Fodder	126	106	79
	0.05	Grain	72	94	108
NC	0.1	Forage	84	97	93
	0.1	Fodder	79	97	94
	0.05	Grain	72	92	88
SD	0.1	Forage	85	87	68
	0.1	Fodder	89	79	93
	0.05	Grain	81	102	83
IA	0.05	Grain	71	86	72
IL	0.05	Grain	113	106	96

**Table VII.**  
**Storage Stability Data**

Matrix/Analyte	Percent Recovery				
	0 Day	6 Month	1 Year	2 Year	4 Year
<u>Sorghum Grain</u>					
TMS fortified at 2.3 ppm	89	NA	85	84	90
CMP fortified at 5.0 ppm	93	NA	90	94	100
AMPA fortified at 5.0 ppm	83	NA	84	69	82
<u>Soybeans</u>					
TMS fortified at 0.46 ppm	86	69	90	102	NA
CMP fortified at 1.0 ppm	100	108	75	104	NA
AMPA fortified at 1.0 ppm	90	86	106	81	NA
<u>Soybean Straw</u>					
TMS fortified at 0.46 ppm	66	74	76	64	NA
CMP fortified at 1.0 ppm	100	102	84	106	NA
AMPA fortified at 1.0 ppm	74	70	93	82	NA
<u>Wheat Grain</u>					
TMS fortified at 0.46 ppm	93	75	74	90	NA
CMP fortified at 1.0 ppm	92	88	82	76	NA
AMPA fortified at 1.0 ppm	98	87	71	70	NA

NA = Not Analyzed

**Table VIII.**  
**Corn Processing Residues**

Sample Type	Application Rate (lb a.i./A)	TMS Residue (ppm)	Conc. Factor (TMS)	CMP Residue (ppm)	Conc. Factor (CMP)	AMPA Residue (ppm)	Conc. Factor (AMPA)
Grain	10.0	0.061	NA	0.070	NA	<0.05	NA
Grain	26.0	<0.05	NA	0.055	NA	<0.05	NA
Grain Dry Process	10.0	<0.05	<0.8	0.064	0.91	<0.05	NA
Grain Dry Process	26.0	0.050	NA	0.065	1.18	<0.05	NA
Grain Wet Process	10.0	0.064	1.05	0.063	0.9	<0.05	NA
Grain Wet Process	26.0	0.050	NA	0.050	0.91	<0.05	NA
Grits Dry Process	10.0	<0.05	<0.8	<0.05	<0.7	<0.05	NA
Grits Dry Process	26.0	<0.05	NA	<0.05	<0.9	<0.05	NA
Meal Dry Process	10.0	<0.05	<0.8	0.070	1.0	<0.05	NA
Meal Dry Process	26.0	<0.05	NA	0.080	1.45	<0.05	NA
Flour Dry Process	10.0	<0.05	<0.8	0.10	1.42	<0.05	NA
Flour Dry Process	26.0	<0.05	NA	0.10	1.82	<0.05	NA

Table VIII. (continued)

Sample Type	Application Rate (lb a.i./A)	TMS Residue (ppm)	Conc. Factor (TMS)	CMP Residue (ppm)	Conc. Factor (CMP)	AMPA Residue (ppm)	Conc. Factor (AMPA)
Crude Oil Dry Process	10.0	<0.05	<0.8	<0.05	<0.7	<0.05	NA
Crude Oil Dry Process	26.0	<0.05	NA	<0.05	<0.9	<0.05	NA
Refined Oil Dry Process	10.0	<0.05	<0.8	<0.05	<0.7	<0.05	NA
Refined Oil Dry Process	26.0	<0.05	NA	<0.05	<0.9	<0.05	NA
Starch Wet Process	10.0	<0.05	<0.8	<0.05	<0.7	<0.05	NA
Starch Wet Process	26.0	<0.05	NA	<0.05	<0.9	<0.05	NA
Crude Oil Wet Process	10.0	<0.05	<0.8	<0.05	<0.7	<0.05	NA
Crude Oil Wet Process	26.0	<0.05	NA	<0.05	<0.9	<0.05	NA
Refined Oil Wet Process	10.0	<0.05	<0.8	<0.05	<0.7	<0.05	NA
Refined Oil Wet Process	26.0	<0.05	NA	<0.05	<0.9	<0.05	NA

NA = Not Applicable

Briefly, the wet milling process consisted of first screening and aspirating the mature, dried grain to remove foreign material. The grain is then steeped in warm water for 48 hours. The steeped corn is ground to separate the germ from the hull and the endosperm. The germ is dried and the oil removed with an expeller. Starch is separated from the remaining products by grinding, washing and finally, by centrifugation. The dry milled products are grits, meal, flour, crude oil and refined oil. These products are obtained by grinding and sieving in a manner similar to the wet milling process without the steeping in warmwater. Both procedures are similar to large-scale commercial operations.

The results (Table VIII.) indicate that in only the corn flour and meal with the dry milling process is there any concentration of residues (1.42 to 1.82 times for CMP residues). These concentration factors are relatively small. In addition, low initial residues are expected from the presented residue studies. Contingent upon the successful completion of the additional required residue studies and no change in use patterns, CBTS finds the submitted processing study to be adequate. A food additive tolerance would not be required from the present study. Should the application rate or methods be changed in the future or if the required residue studies are not consistent with the old, CBTS may require a new processing study be submitted reflecting these changes.

CBTS finds the corn processing study to be adequate contingent upon no new residues being required to be regulated from the crop metabolism studies and the successful completion of the method validation trials.

#### MEAT, MILK, POULTRY AND EGGS (MRID# 412099-11,12,13,14)

A poultry metabolism study was performed using  $^{14}\text{C}$ -sulfosate labeled in the anionic moiety with White Leghorn laying hens. The radiolabeled compound was synthesized at Richmond Research Center of ICI and had a specific activity of 30 mCi/mmol and its radiochemical purity was determined to be 98.2% by tlc coupled with autoradiography.

In addition to the radiolabeled compound, CMPA containing 95% enrichment with  $^{13}\text{C}$  was prepared by Cambridge Isotopes and converted by ICI into sulfosate. This material was used in the dilution of the radiolabeled material for the 100x dose to ease the identification of possible metabolites.

Feeding and the combustion total  $^{14}\text{C}$ -residue studies were performed by Analytical Development Corporation in Monument, Colorado and samples shipped to ICI's Mountain View Research Center in Sunnyvale, California for further analysis.



Fourteen hens were divided into three groups, four each in groups 1 and two with the remaining six in group 3. Group 1 was the untreated control, group 2 was the 1x dose and group 3 was the 100x dose. Overall dosages were based on the expected exposure of a hen consuming 120 g of layer ration containing either 0.87 or 90.8 ppm of CMPA. Actual doses were administered by gelatin capsule containing either 0.104 or 10.9 mg (as calculated from the measured specific activity and volumes of each solution) of sulfosate absorbed into cellulose. An 1.42 ppm calculated low dose would correspond to the hen consuming 68.6 g (the measured average of daily food consumption) of the available food. An 121 ppm calculated high dose would correspond to the hen consuming 85.4 g (the measured average of daily food consumption) of the available food.

**Table VII.**  
**Total Residues in Excreta Samples from <sup>14</sup>C-CMPA Sulfosate**

Group	Dosing Level	Days Post Dosing	ppm	% Dose Recovered
II	1x	1	0.559	19.38
		2	0.765	20.25
		3	0.898	22.41
		4	0.652	<u>20.40</u>
		Total		82.44
III	100x	1	67.8	21.07
		2	51.5	19.99
		3	56.5	22.60
		4	57.3	<u>23.66</u>
		Total		87.32

The hens were treated daily for four days and sacrificed approximately eighteen hours after the last daily dose. Eggs were collected during the dosing period, separated into whites and yolks, composited by group and frozen for later analysis. The shells were discarded. Also during the treatment period, total excreta were collected once daily from each hen, composited by group, weighed and stored frozen. The following samples were collected and composited by group: the entire gastrointestinal tract, liver, kidneys, heart, breast muscle, thigh muscle, composite fat (subcutaneous plus visceral) and the remaining carcass without feathers. The composite samples were weighed and stored frozen until further analysis. Prior to subsampling for combustion analysis, all composite samples were thawed and individually homogenized. The breast muscle, thigh, fat and liver were trimmed of extraneous material, then the samples for each group were ground until homogenous. The kidneys and fat were trimmed and finely chopped with a scalpel. The G.I. tracts and carcasses for each group were chopped with Dry Ice in a

Hobart food chopper. After preparation, each of the above samples was subsampled for combustion analysis and the bulk of the sample refrozen for later analysis.

**Table VIII.**  
**Total Residues in Slaughter Samples  $^{14}\text{C}$ -CMPA Sulfosate**

Group	Dosing Level	Sample Type	ppm	% Dose Recovered
I	Control	Breast Muscle	<0.0010	
		Carcass	<0.0010	
		Fat	<0.0010	
		G.I. Tract	<0.0010	
		Heart	<0.0010	
		Kidney	<0.0010	
		Liver	<0.0010	
		Thigh Muscle	<0.0010	
II	1x	Breast Muscle	<0.0010	0.015
		Carcass	0.0048	0.982
		Fat	<0.0010	0.002
		G.I. Tract	0.2529	8.804
		Heart	0.0013	0.002
		Kidney	0.0272	0.043
		Liver	0.0044	0.036
		Thigh Muscle	0.0011	<u>0.030</u>
		Total		9.914
III	100x	Breast Muscle	<0.0500	0.010
		Carcass	0.2413	0.538
		Fat	<0.0500	0.001
		G.I. Tract	18.1813	6.001
		Heart	0.0894	0.002
		Kidney	1.8127	0.043
		Liver	0.3375	0.033
		Thigh Muscle	0.0520	<u>0.017</u>
		Total		6.645

The composited excreta contained 82.4 (low dose) and 87.3% (high dose) respectively of the applied radioactivity (Table XX). The radioactive components were demonstrated through a lengthy adequately defined isolation procedure to be 89.7% parent CMPA and 6.3% AMPA by two-dimensional tlc and  $^{13}\text{C}$  nmr in the high dose.

The petitioner states that magnitude of the residues in the excreta may actually be underreported since the actual isolated radioactivity corresponded to 104 to 118% of the combustion value. Assuming that the earlier figures in the tables are accurate, then up to 94% of the administered activity may be

found in the excreta.

Table IX.  
Total Residues in Egg Samples <sup>14</sup>C-CMPA Sulfosate

Group	Dosing Level	Sample Type	Days Post Dosing	ppm
I	Control	Whites	1	<0.0010
			2	<0.0010
			3	<0.0010
			4	<0.0010
		Yolks	1	<0.0010
			2	<0.0010
			3	<0.0010
			4	<0.0010
II	1x	Whites	1	<0.0010
			2	<0.0010
			3	<0.0010
			4	<0.0010
		Yolks	1	<0.0010
			2	<0.0010
			3	<0.0010
			4	0.0017
III	100x	Whites	1	<0.0500
			2	<0.0500
			3	0.0568
			4	0.06010
		Yolks	1	<0.0500
			2	<0.0500
			3	<0.0500
			4	0.1168

The contents of the gastrointestinal tract contained 8.8 and 6.0 % of the administered dose, respectively (Table XX). No attempts were made to further characterize the residues in these samples. The edible tissues contained less than 0.1% of dose. The highest residues were found in kidneys with 0.027 ppm from the low dose and 1.81 ppm from the high dose. This result is not unexpected due to the relatively fast absorption and excretion of CMPA. The magnitude of these residues was too small for their nature to be characterized.

The largest percentage of dose outside the gastrointestinal tract is in the carcass, although the overall residue values are small (0.005 and 0.24 ppm). The carcass consists of muscle, fat and

bone and thus reflects the residue composition of all three tissues. Independent measurements of muscle and fat suggest that the majority of the residue is present in bone. In unsubmitted rat studies, ICI reported that CMPA is preferentially adsorbed to or incorporated into the matrix bone tissue. It was suggested that since CMPA is similar in structure to nitrilotriacetic acid, nitrilotri(methylenephosphonic acid) and nitrilodiaceticmethylenephosphonic acid which are good chelators of calcium, that CMPA would also be and this might be the cause of this concentration of residues in the bone tissue. No supporting chemical data were submitted.

Only one egg residue value in the low dose group was above the detection limit (0.0017 ppm) in the yolk. Two values of the egg white (0.0568 and 0.0601 ppm) and one in the yolk (0.1168 ppm) had measurable values in the high dose group. The levels of all the detected residues were too low to identify their nature. However, when compared with high dose residues, the level of the residues continued to rise over the time course. Unlabeled poultry feeding studies are required to be carried out for 30 days in order to determine if residues of CMPA and AMPA in poultry and eggs may continue to build up over time.

Although CBTS recognizes the difficulty in determining the nature of residues from a compound that so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various tissues. CBTS normally requires the identification of 90% of the absorbed residues. In this case, none of the residues present in tissues have any kind of identification attempt. No attempts were made to determine the nature of the residue in eggs.

The submitted laying hen metabolism anion study is inadequate. Further characterization of the nature of the residues in the tissues of laying hens and eggs is required. Additionally, feeding studies are required to be carried out for 30 days on eggs using unlabeled material and the required analytical method for the anion in eggs in order to determine if residues may build up over time. It is CBTS policy that feeding studies should be continued for at least four weeks even if there are no detectable residues or residues plateau prior to that time.

A separate poultry metabolism study was performed using  $^{14}\text{C}$ -sulfosate labeled in the cationic moiety with White Leghorn laying hens. The radiolabeled compound was synthesized at Richmond Research Center of ICI and had a specific activity of 20 mCi/mmol and its radiochemical purity was determined to be 99.7% by tlc coupled with autoradiography.

Feeding and the combustion total  $^{14}\text{C}$ -residue studies were

performed by Analytical Development Corporation in Monument, Colorado and samples shipped to ICI's Mountain View Research Center in Sunnyvale, California for further analysis.

Fourteen hens were divided into three groups, four each in groups 1 and two with the remaining six in group 3. Group 1 was the untreated control, group 2 was the 1x dose and group 3 was the 100x dose. Overall dosages were based on the expected exposure of a hen consuming 120 g of layer ration containing either 0.9 or 87.9 ppm of TMS. Actual doses were administered by gelatin capsule containing either 0.105 or 10.6 mg (as calculated from the measured specific activity and volumes of each solution) of sulfosate absorbed into cellulose. An 1.07 ppm calculated low dose would correspond to the hen consuming 98.1 g (the measured average of daily food consumption) of the available food. A 107 ppm calculated high dose would correspond to the hen consuming 98.8 g (the measured average of daily food consumption) of the available food.

**Table X.**  
**Total Residues in Excreta Samples from <sup>14</sup>C-TMS Sulfosate**

Group	Dosing Level	Days Post Dosing	ppm	% Dose Recovered
II	1x	1	0.397	22.56
		2	0.506	21.95
		3	0.511	24.29
		4	0.648	<u>23.52</u>
		Total		92.32
III	100x	1	55.3	22.60
		2	51.7	23.63
		3	54.1	22.65
		4	65.6	<u>22.94</u>
		Total		91.82

The hens were treated daily for four days and sacrificed approximately eighteen hours after the last daily dose. Eggs were collected during the dosing period, separated into whites and yolks, composited by group and frozen for later analysis. The shells were discarded. Also during the treatment period, total excreta were collected once daily from each hen, composited by group, weighed and stored frozen. The following samples were collected and composited by group: the entire gastrointestinal tract, liver, kidneys, heart, breast muscle, thigh muscle, composite fat (subcutaneous plus visceral) and the remaining carcass without feathers. The composite samples were weighed and stored frozen until further analysis. Prior to subsampling for

combustion analysis, all composite samples were thawed and individually homogenized. The breast muscle, thigh, fat and liver were trimmed of extraneous material, then the samples for each group were ground until homogenous. The kidneys and fat were trimmed and finely chopped with a scalpel. The G.I. tracts and carcasses for each group were chopped with Dry Ice in a Hobart food chopper. After preparation, each of the above samples was subsampled for combustion analysis and the bulk of the sample refrozen for later analysis.

**Table XI.**  
**Total Residues in Slaughter Samples from  $^{14}\text{C}$ -TMS Sulfosate**

Group	Dosing Level	Sample Type	ppm	% Dose Recovered
I	Control	Breast Muscle	<0.0015	
		Carcass	<0.0015	
		Fat	<0.0015	
		G.I. Tract	<0.0015	
		Heart	<0.0015	
		Kidney	<0.0015	
		Liver	<0.0015	
		Thigh Muscle	<0.0015	
II	1x	Breast Muscle	0.0030	0.077
		Carcass	0.0021	0.439
		Fat	<0.0015	0.001
		G.I. Tract	0.0138	0.439
		Heart	0.0019	0.003
		Kidney	0.0039	0.008
		Liver	0.0027	0.021
		Thigh Muscle	0.0030	<u>0.074</u>
		Total		1.062
III	100x	Breast Muscle	0.2317	0.061
		Carcass	0.2308	0.487
		Fat	<0.0500	0.001
		G.I. Tract	3.9126	1.361
		Heart	0.1678	0.003
		Kidney	1.0042	0.020
		Liver	0.4014	0.034
		Thigh Muscle	0.2248	<u>0.071</u>
		Total		2.038

The composited excreta contained 92.32 (low dose) and 91.82 % (high dose) respectively of the applied radioactivity (Table XX). The radioactive components were demonstrated through a lengthy adequately defined isolation procedure to be 99 % parent TMS by tlc in three separate systems and gc-ms of the dealkylated TMS in the high dose.

Table XII.  
Total Residues in Egg Samples from <sup>14</sup>C-TMS Sulfosate

Group	Dosing Level	Sample Type	Days Post Dosing	ppm
I	Control	Whites	1	<0.0015
			2	<0.0015
			3	<0.0015
			4	<0.0015
		Yolks	1	<0.0015
			2	<0.0015
			3	<0.0015
			4	<0.0015
II	1x	Whites	1	<0.0015
			2	<0.0015
			3	<0.0015
			4	<0.0015
		Yolks	1	<0.0015
			2	<0.0015
			3	0.0017
			4	0.0020
III	100x	Whites	1	<0.0500
			2	<0.0500
			3	<0.0500
			4	<0.0500
		Yolks	1	<0.0500
			2	0.0559
			3	0.0742
			4	0.1499

The contents of the gastrointestinal tract contained 0.4 and 1.4 % of the administered dose, respectively (Table XX). No attempts were made to further characterize the residues in these samples. The edible tissues contained less than 0.2% of each dose. The highest residues were found in kidneys with 0.0039 ppm from the low dose and 1.00 ppm from the high dose. This result is not unexpected due to the relatively fast absorption and excretion of TMS. The magnitude of these residues was too small for their nature to be characterized.

The largest percentage of dose outside the gastrointestinal tract is in the carcass, although the overall residue values are small (0.0021 and 0.2308 ppm). The carcass consists of muscle, fat and bone and thus reflects the residue composition of all three tissues. Independent measurements of muscle and fat suggest that

unlike CMPA, the majority of the residue is evenly distributed ( 0.23 ppm in muscle and 0.23 ppm in carcass).

Two egg residue values in the low dose group were above the detection limit (0.0017 and 0.0020 ppm) in the yolk. Three values of the high dose (0.0559, 0.0742 and 0.1499 ppm) had measurable values in the yolk. The levels of all the detected residues were too low to identify their nature. However, the level of the residues continued to rise over the time course. Unlabeled poultry feeding studies are required to be carried out for 30 days in order to determine if residues of TMS in poultry and eggs may continue to build up over time.

Although CBTS recognizes the difficulty in determining the nature of residues from a compound that so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various tissues. CBTS normally requires the identification of 90% of the absorbed residues. In this case, none of the residues present in tissues have any kind of identification attempt. No attempts were made to determine the nature of the residue in eggs.

The submitted hen metabolism cation study is inadequate. Further characterization of the nature of the residues in the tissues of laying hens and eggs is required. Additionally, feeding studies are required to be carried out for 30 days on eggs using unlabeled material and the required analytical method for the anion in eggs in order to determine if residues may build up over time. It is CBTS policy that feeding studies should be continued for at least four weeks even if there are no detectable residues or residues plateau prior to that time.

**Table XIII.**  
**Total <sup>14</sup>C Residues in 100x Goat Whole Milk from <sup>14</sup>C-CMPA Sulfosate**

Days Post Dosing	ppm	% Dose Received
0.5	0.0170	0.001
1.0	0.0324	0.005
1.5	0.0359	0.003
2.0	0.0415	0.004
2.5	0.0468	0.004
3.0	0.0549	0.006
3.5	0.0684	0.004
4.0	0.0623	<u>0.004</u>
	Total	0.032



A goat ruminant metabolism study was performed using  $^{14}\text{C}$ -sulfosate labeled in the anionic moiety with non-pregnant lactating Alpine goats. The radiolabeled compound was synthesized at Richmond Research Center of ICI and had a specific activity of 30 mCi/mmol and its radiochemical purity was determined to be 98.1 to 98.4% by tlc coupled with autoradiography.

In addition to the radiolabeled compound, CMPA containing 95% enrichment with  $^{13}\text{C}$  was prepared by Cambridge Isotopes and converted by ICI into sulfosate. This material was used in the dilution of the radiolabeled material for the 100x dose to ease the identification of possible metabolites.

Feeding and the combustion total  $^{14}\text{C}$ -residue studies were performed by Analytical Development Corporation in Monument, Colorado and samples shipped to ICI's Mountain View Research Center in Sunnyvale, California for further analysis.

Table XIV.  
Total  $^{14}\text{C}$  Residues in 1xA Goat Samples from  $^{14}\text{C}$ -CMPA Sulfosate

Sample Type	ppm	% Dose Received
Carcass	<0.0100	0.322
Fat	<0.0100	
Heart	<0.0100	0.002
Kidney	0.0866	0.163
Large Intestine	0.4281	7.788
Liver	<0.0100	0.033
Muscle	<0.0100	
Small Intestine	0.1458	1.744
Stomach	0.1100	<u>4.702</u>
	Subtotal	14.754
Feces Day 1	0.143	8.416
Feces Day 2	0.411	20.925
Feces Day 3	0.377	25.409
Feces Day 4	0.355	<u>19.774</u>
	Subtotal	74.525
Urine Day 1	0.043	1.533
Urine Day 2	0.148	2.325
Urine Day 3	0.141	3.768
Urine Day 4	0.119	<u>3.226</u>
	Subtotal	10.853
	Total	100.132

Four goats were used in this study, two were dosed at 1 ppm, one at 100 ppm and the remaining goat was used as a negative control.

Each goat received a gelatin capsule containing either cellulose alone or the appropriate dose absorbed into the cellulose twice daily for four days. Each goat was milked twice daily and each milk sample was weighed and stored frozen separately after one gram aliquots were removed for radioanalysis. Urine and feces were collected daily and the 1x goats were placed in a respiration chamber the last day to monitor expiration of expired carbon dioxide.

The goats were slaughtered approximately 16 hours following the last dose and selected tissues and the gut contents homogenized and radioassayed. A 30 ml portion of the milk from the last milking of the 100x goat was separated into skim milk and milk fat for radioassay. Subsamples of urine, feces and kidney tissue were analyzed to determine the metabolic fate of CMPA and the nature of the residues in tissues.

Table XV.  
Total  $^{14}\text{C}$  Residues in 1xB Goat Samples from  $^{14}\text{C}$ -CMPA Sulfosate

Sample Type	ppm	% Dose Received
Carcass	<0.0100	0.198
Fat	<0.0100	
Heart	<0.0100	0.003
Kidney	0.0704	0.168
Large Intestine	0.6097	16.184
Liver	<0.0100	0.033
Muscle	<0.0100	
Small Intestine	0.1353	1.457
Stomach	0.0900	<u>7.738</u>
	Subtotal	25.781
Feces Day 1	0.040	2.452
Feces Day 2	0.312	17.618
Feces Day 3	0.400	22.986
Feces Day 4	0.457	<u>21.223</u>
	Subtotal	64.280
Urine Day 1	0.042	0.615
Urine Day 2	0.112	1.651
Urine Day 3	0.175	2.400
Urine Day 4	0.218	<u>1.975</u>
	Subtotal	6.640
	Total	96.701

Residues in milk from the 1x goats were below the limit of detection (<0.002 ppm sulfosate) and only 0.032% of the dose appeared in milk from the 100x animal (Table XIII). It is difficult to determine whether the magnitude of the residue in

milk has reached a maximum since the study was only carried out for 4 days. The residues in milk were approximately five times more concentrated in the fat fraction (0.2000 ppm vs. 0.0444 ppm sulfosate) than in the skim milk fraction. In all edible tissues of 1x animals except kidneys, residue levels were below the limit of detection (0.01 ppm sulfosate, Tables XIV and XV). The average level of residue in the kidney was 0.078 ppm in the 1x animals. No expiration of labeled carbon dioxide was measured. Overall accountability for the radioactivity is good (96 to 100% Tables XIV, XV and XVI). It is evident that CMPA is poorly absorbed.

In feces, urine and kidney, the major portion of the radioactivity could be assigned to unchanged parent. The three samples were purified by various ion exchange and tlc methods. In feces and urine, the identities of the final peaks were confirmed by  $^{13}\text{C}$  ft-nmr. There was said to be insufficient material isolated from kidney for confirmation in this manner. In all other tissues, no attempts are documented to determine the nature of the residue. No attempt was made to determine the nature of the residue present in the high dose milk.

Table XVI.  
Total  $^{14}\text{C}$  Residues in 100x Goat Samples from  $^{14}\text{C}$ -CMPA Sulfosate

Sample Type	ppm	% Dose Received
Carcass	0.2298	0.449
Fat	<0.0500	
Heart	0.0552	0.002
Kidney	9.7360	0.191
Large Intestine	66.6846	12.715
Liver	0.2628	0.027
Muscle	<0.0500	
Small Intestine	18.5456	1.860
Stomach	8.5164	<u>6.600</u>
	Subtotal	21.894
Feces Day 1	6.30	4.265
Feces Day 2	37.42	19.832
Feces Day 3	44.83	23.581
Feces Day 4	40.35	<u>20.907</u>
	Subtotal	68.584
Urine Day 1	4.990	0.843
Urine Day 2	14.620	2.207
Urine Day 3	23.392	2.322
Urine Day 4	14.581	<u>3.008</u>
	Subtotal	8.380
	Total	98.858

Although CBTS recognizes the difficulty in determining the nature of residues from a compound that so poorly absorbed, it is still necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various tissues. CBTS normally requires the identification of 90% of the absorbed residues. In this case, only the residues present in kidney have any kind of identification attempt. This is approximately 30% of the residue present in the goat. No attempts were made to determine the nature of the residue in milk.

The submitted goat metabolism anion study is inadequate. Additional characterization in the feeding metabolism studies is required in order to determine the nature of the residue in goats. Milk fat, liver and kidney residues should be sufficient to be identified. Additionally, feeding studies are required to be carried out for 30 days feeding with cattle using unlabeled material and the required analytical methods for meat and milk in order to determine if residues may build up over time.

A goat ruminant metabolism study was performed using  $^{14}\text{C}$ -sulfosate labeled in the cationic moiety with non-pregnant lactating Nubian goats. The radiolabeled compound was synthesized at Richmond Research Center of ICI and had a specific activity of 20 mCi/mmol and its radiochemical purity was determined to be 96.8 to 97.2% by tlc coupled with autoradiography.

Feeding and the combustion total  $^{14}\text{C}$ -residue studies were performed by Analytical Development Corporation in Monument, Colorado and samples shipped to ICI's Mountain View Research Center in Sunnyvale, California for further analysis.

Four goats were used in this study, two were dosed at 1 ppm, one at 100 ppm and the remaining goat was used as a negative control. Each goat received a gelatin capsule containing either cellulose alone or the appropriate dose absorbed into the cellulose twice daily for four days. Each goat was milked twice daily and each milk sample was weighed and stored frozen separately after a one gram aliquots were removed for radioanalysis. Urine and feces were collected daily and the 1x goats were placed in a respiration chamber the last day to monitor expiration of expired carbon dioxide.

The goats were slaughtered approximately 16 hours following the last dose and selected tissues and the gut contents homogenized and radioassayed. A 20 ml portion of the milk from the last milking of the treated goats was separated into skim milk and milk fat for radioassay. Subsamples of urine, feces and kidney tissue were analyzed to determine the metabolic fate of sulfosate

and the nature of the residues in tissues.

Residue levels never exceeded 0.035 ppm in the 1x milk samples and were concentrated approximately three fold in the skim milk fraction. Overall milk residue levels did not increase linearly with dose in the 100x sample (approximately 1 ppm) and were concentrated (10x) in the skim milk fraction.

**Table XVII.**  
**Total <sup>14</sup>C TMS Residues in 100x Goat Whole Milk**

Dose	Days Post Dosing	ppm	% Dose Received
1x	0.5	0.0069	0.062
	1.0	0.0190	0.314
	1.5	0.0260	0.265
	2.0	0.0260	0.452
	2.5	0.0284	0.237
	3.0	0.0321	0.511
	3.5	0.0324	0.275
	4.0	0.0315	<u>0.501</u>
		Total	2.617
1x	0.5	0.0071	0.042
	1.0	0.0172	0.148
	1.5	0.0224	0.144
	2.0	0.0253	0.345
	2.5	0.0282	0.219
	3.0	0.0324	0.428
	3.5	0.0321	0.219
	4.0	0.0322	<u>0.276</u>
		Total	1.820
100x	0.5	0.2077	0.014
	1.0	0.6179	0.073
	1.5	0.8591	0.063
	2.0	0.8872	0.111
	2.5	1.1512	0.086
	3.0	1.0747	0.126
	3.5	1.0756	0.079
	4.0	1.0370	<u>0.119</u>
		Total	0.672

Residue levels in the 1x samples never exceeded 0.02 ppm in edible tissues except liver and kidney which were less than 0.055 ppm (Tables XIX to XX). Expired carbon dioxide and volatiles were trapped only for a short period at the end of the experiment and no trapping was done on the 100x animal. The low levels of recovery of radioactivity may be due to untrapped carbon dioxide and volatiles.

**Table XVIII.**  
**Total  $^{14}\text{C}$  TMS Residues in Milk Fractions**

Dose	Sample Type	ppm
1x	Skim Milk	0.0663
	Milk Fat	0.0225
1x	Skim Milk	0.0685
	Milk Fat	0.0229
100x	Skim Milk	6.8971
	Milk Fat	0.5910

**Table XIX.**  
**Total  $^{14}\text{C}$  Residues in 1xA Goat Samples from  $^{14}\text{C}$ -TMS Sulfosate**

Sample Type	ppm	% Dose Received
Carcass	0.0163	4.400
Fat	<0.0100	
Heart	0.0193	0.069
Kidney	0.0447	0.089
Large Intestine	0.0424	0.752
Liver	0.0413	0.465
Muscle	0.0151	
Small Intestine	0.0217	0.228
Stomach	0.0200	<u>1.275</u>
	Subtotal	7.278
Feces Day 1	<0.0100	0.349
Feces Day 2	0.0195	0.988
Feces Day 3	0.0242	1.179
Feces Day 4	0.0281	<u>1.001</u>
	Subtotal	3.418
Urine Day 1	0.3318	5.410
Urine Day 2	0.4631	7.979
Urine Day 3	0.5155	5.808
Urine Day 4	0.4911	<u>6.509</u>
	Subtotal	25.706
	Total	36.402

Distribution of the residues was similar in the 100x animal (Table XXI), but of greater magnitude as should be expected. Residues were highest in the kidney and liver and most values were approximately 1 ppm. Only in the liver was any attempt made to determine the nature of the residue. TMS represents approximately 95.2% of the HCl extractable portion of the residue

in liver. No attempt was made to characterize the nature of the 22% of the radioactivity that was not extracted in this step. No attempts were made to characterize the nature of the residues in any other tissue or in milk. There should be sufficient quantities in order to determine the nature of the residue in these samples. CBTS normally requires characterization of 90% of the residue. Further characterization of the residues is required.

**Table XX.**  
**Total  $^{14}\text{C}$  Residues in 1xB Goat Samples from  $^{14}\text{C}$ -TMS Sulfosate**

Sample Type	ppm	% Dose Received
Carcass	0.0133	3.671
Fat	<0.0100	
Heart	0.0182	0.071
Kidney	0.0409	0.085
Large Intestine	0.0421	0.774
Liver	0.0532	0.643
Muscle	0.0134	
Small Intestine	0.0299	0.128
Stomach	0.0163	<u>0.752</u>
	Subtotal	6.123
Feces Day 1	0.0136	0.463
Feces Day 2	0.0472	1.706
Feces Day 3	0.0506	1.839
Feces Day 4	0.0698	<u>2.463</u>
	Subtotal	6.471
Urine Day 1	0.4241	4.044
Urine Day 2	0.6844	6.619
Urine Day 3	0.5575	7.138
Urine Day 4	0.6531	<u>5.160</u>
	Subtotal	22.962
	Total	35.556

In urine and liver, the major portion of the radioactivity could be assigned to unchanged parent. The two samples were purified by various ion exchange and tlc methods. In urine, the identity of the final peak was confirmed by  $^1\text{H}$  ft-nmr. There was insufficient material isolated from liver for confirmation in this manner. In all other tissues, no attempts are documented to determine the nature of the residue. No attempt was made to determine the nature of the residue present in the milk.

Although CBTS recognizes the difficulty in determining the nature of residues from a compound that so poorly absorbed, it is still

necessary to determine not the nature of the residue in unabsorbed material (such as feces and urine) but of that present in the various tissues. CBTS normally requires the identification of 90% of the absorbed residues. In this case, only the residues present in liver have any kind of identification attempt. This is approximately 9% of the residue present in the goat. No attempts were made to determine the nature of the residue in milk. Further attempts at identification are required.

The submitted goat cation metabolism study is inadequate. Additional feeding metabolism studies or further characterization of the residues in the goat are required in order to determine the nature of the residue in goats. Additionally, feeding studies are required to be carried out with cattle for 30 days using unlabeled material and the required analytical methods for meat and milk in order to determine if residues may build up over time. It is CBTS policy to require such studies when residue levels are too low to detect in short term studies and when such studies are not carried out long enough to reach a maximum or plateau region.

**Table XXI.**  
**Total  $^{14}\text{C}$  Residues in 100x Goat Samples from  $^{14}\text{C}$ -TMS Sulfosate**

Sample Type	ppm	% Dose Received
Carcass	1.4130	3.341
Fat	0.5936	
Heart	1.4663	0.044
Kidney	4.3900	0.075
Large Intestine	2.1753	0.305
Liver	2.2801	0.245
Muscle	1.9345	
Small Intestine	1.2339	0.073
Stomach	1.8885	<u>0.589</u>
	Subtotal	4.672
Feces Day 1	2.1062	0.943
Feces Day 2	3.5781	1.865
Feces Day 3	3.7243	1.756
Feces Day 4	5.5892	<u>3.070</u>
	Subtotal	7.634
Urine Day 1	73.7893	12.570
Urine Day 2	102.2740	15.470
Urine Day 3	98.3398	17.330
Urine Day 4	68.3145	<u>11.036</u>
	Subtotal	56.405
	Total	68.711



**OTHER CONSIDERATIONS**

An "International Residue Status" sheet is attached. There are no Canadian, Mexican or Codex tolerances for Sulfosate on or in corn. There are Codex limits of 0.1 ppm for corn grain and 100 ppm for corn fodder for glyphosate per se. There is also a Canadian negligible residue type limit of 0.1 ppm for glyphosate per se for "all food crops". There are compatibility problems associated with corn grain and corn fodder due to differences in the residue being regulated (i.e., U.S. tolerance includes the AMPA metabolite).

Attachments: International Residue Limit Status Sheet.

CC: S. Koepke (CBTS), PP9F3796, PIB/FOB (C. Furlow),  
Circulation(7), RF, SF, R. Schmitt

H7509C:CBTS:Reviewer(SK):CM#2:Rm810:557-4380:Typist(SK):12/20/90.  
RDI:Section Head: R.S. Quick:12/21/90: Br.Sr.Scientist:R.A.  
Loranger:12/21/90.

J. L. L. 6/27/71

Attachment:

Page 1 of 1INTERNATIONAL RESIDUE LIMIT STATUSCHEMICAL Sulfosate (trimethylsulfonium salt of glyphosate)

CODEX NO. \_\_\_\_\_

CODEX STATUS:☒ No Codex Proposal  
Step 6 or Above *for sulfosate* \*

Residue (if Step 8): \_\_\_\_\_

<u>Crop(s)</u>	<u>Limit</u> <u>(mg/kg)</u>
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PROPOSED U.S. TOLERANCES:Petition No. 9F3796DEB Reviewer S KoepkeResidue: parent, carboxy methyl/aminomethyl phosphonate and aminomethyl phosphonic acids, combined residues.

<u>Crop(s)</u>	<u>Limit</u> <u>(mg/kg)</u>
corn, grain	0.1 ppm
corn, forage	0.2 ppm
corn, fodder	0.2 ppm

CANADIAN LIMITS:☒ No Canadian Limit \*\*

Residue: \_\_\_\_\_

<u>Crop(s)</u>	<u>Limit</u> <u>(mg/kg)</u>
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MEXICAN LIMITS:☒ No Mexican Limit

Residue: \_\_\_\_\_

<u>Crop(s)</u>	<u>Limit</u> <u>(mg/kg)</u>
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**BEST COPY AVAILABLE**NOTES\* There are no limits for glyphosate  
per se on corn grain/fodder (0.1 and 100 ppm respectively, the

Form Revised 1989

\*\* A 0.1/100 Canadian negligible residue  
type limit is established for glyphosate  
per se in "all food crops".Toler for straw and fodders  
of cereal grains.

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