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

NOV 3 1993

MEMORANDUM:

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

SUBJECT: Sodium Acifluorfen, Reregistration. Nature of the Residue in Peanut (MRID No. 42368301) and Rice (MRID No. 42368302).
CBRS No. 12380. DP Barcode No. D194099.

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THRU: Francis B. Suhre, Section Head
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TO: Jay Ellenberger, Chief
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Attached is a review of residue chemistry data on the nature of the residue in peanut and rice, submitted by BASF Corporation in support of reregistration. This information was reviewed by Dynamac Corporation under supervision of CBRS, HED. The data assessment has undergone secondary review in the branch and has been revised to reflect branch policies.

The data submitted was in response to a previous review, which concluded that additional work was required (CBRS 10199, 12/9/92, J. Abbotts). The present review concludes that the present submission clears some deficiencies, and additional work remains necessary to address the remaining deficiencies. The registrant has initiated additional work, including a new peanut metabolism study, to upgrade data submitted to an acceptable status.

If you need additional input please advise.

cc:Circ, Abbotts, RF, Sodium Acifluorfen List B File, SF, Dynamac
RDI:FBSuhre:11/2/93:MSMetzger:11/2/93:EZager:11/2/93
H7509C:CBII-RS:JAbbotts:CM-2:Rm805A:305-6230:11/3/93
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DYNAMAC
CORPORATION
Environmental Services

Final Report

SODIUM ACIFLUORFEN
Shaughnessy No. 114402
(DP Barcode D194099;
CBRS No. 12380; Case No. 2605)

TASK 4
Registrant's Response to Residue Chemistry Data Requirements

October 15, 1993

Contract No. 68-D2-0053

Submitted to:
U.S. Environmental Protection Agency
Arlington, VA 22202

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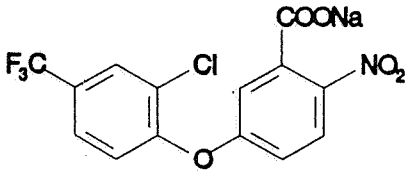
SODIUM ACIFLUORFEN



Shaughnessy No. 114402; Case 2605

(CBRS No. 12380; DP Barcode D194099)

Task 4

REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS

BACKGROUND

In response to the Sodium Acifluorfen Phase 4 Reviews (S. Funk, 2/14/91), BASF Corporation submitted data pertaining to the metabolism of [^{14}C]sodium acifluorfen in peanuts and rice. These data were reviewed by the Agency (CBRS No. 10199, DP Barcode D180455, 12/9/92, J. Abbotts) and were found to be inadequate to satisfy reregistration requirements because of several deficiencies. In response, BASF Corporation has submitted supplemental data (1993; MRIDs 42865801 and 42865802) pertaining to the nature of the residue in peanuts and rice. These supplemental data are reviewed here for their adequacy in fulfilling the outstanding residue chemistry data requirements. The Conclusions and Recommendations stated below apply only to the nature of the residue of sodium acifluorfen in peanuts and rice. No other data requirements in the Sodium Acifluorfen Phase 4 Reviews are addressed herein.

Metabolism studies with poultry and ruminants are currently under review in CBRS (DP Barcodes D192655 and D192899, respectively).

Tolerances for residues of sodium acifluorfen are currently expressed as the combined residues of the sodium salt of acifluorfen (sodium 5-[2-chloro-4-trifluoromethyl)-phenoxy]-2-nitrobenzoic acid) and its metabolites (the corresponding acid, methyl ester, and amino analogs) in/on plant and animal commodities [40 CFR § 180.383]. The Pesticide Analytical Manual (PAM) Vol. II lists Method I, a GLC method with electron capture detection, as available for the enforcement of sodium acifluorfen tolerances. There are no established or proposed Codex MRLs for sodium acifluorfen residues. Therefore, there are no issues of compatibility with respect to U.S. tolerances and Codex MRLs.

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CONCLUSIONS

Conclusions below are numbered to be consistent with the previous review (CBRS 10199, 12/9/92, J. Abbotts):

Qualitative Nature of the Residue in Peanuts:

1a. Registrant reported data on initial sample TRRs and radioactivity in each fraction, which indicate only minor losses during extraction. Conclusion 1a of the previous review is satisfied.

1c. Registrant supplied representative chromatograms from HPLC System II. Conclusion 1c of the previous review is satisfied.

1d. through
1g. Registrant reported that a new peanut metabolism study has been initiated to resolve remaining Conclusions of the previous review.

Qualitative Nature of the Residue in Rice:

2a. Data provided by registrant indicate consistency in HPLC mobility of standards. Conclusion 2a of the previous review is satisfied.

2c. Registrant explained the assignment of residues in rice grain. Conclusion 2c of the previous review is satisfied.

2d. Registrant reported it is conducting additional work in rice hulls in order to satisfy Conclusion 2d of the previous review.

2e. Aciflourfen represents the large majority of TRR in rice straw. Polar peak 1 (total of 18% TRR, 0.36 ppm) represents most of the remaining TRR. If Polar peak 1 cannot be resolved into multiple peaks with a second system (a different HPLC system would be acceptable in this regard), then it should be identified. Other than Polar peak 1, no further work on unidentified peaks or fractions from rice straw is necessary. Conclusion 2e of the previous review is not yet satisfied.

2f. Registrant did not disagree with this Conclusion from the previous review, which reflected an observation that the nature of the residue in rice is not yet adequately understood. This Conclusion can be considered satisfied, but further work in rice is necessary.

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2g. Registrant has provided the storage stability data requested in this Conclusion of the previous review. Relevant storage stability data should also be provided in support of the additional work planned to satisfy Conclusion 2d and recommended to satisfy Conclusion 2e in this review.

RECOMMENDATIONS

Additional work is necessary to upgrade available data on the nature of the residue in plants to an acceptable status. Registrant has initiated a new metabolism study in peanuts to satisfy remaining outstanding Conclusions from the previous review (CBRS 10199, 12/9/92, J. Abbotts). Once the new metabolism study is completed, it will be reviewed with consideration of the data already submitted. Additional work is necessary in rice to satisfy Conclusions 2d and 2e of this review. The generic requirement for radiovalidation of enforcement method(s) will be addressed once the nature of the residue in plants is adequately understood and the residues to be regulated have been determined. The nature of the residue in peanuts and rice remains an outstanding data requirement for reregistration.

Detailed Considerations

Qualitative Nature of the Residue in Peanuts

An Agency memorandum (CBRS No. 10199, DP Barcode D180455, 12/9/92, J. Abbotts) concluded that the qualitative nature of the residue in peanuts is not adequately understood for the following reasons. Conclusions are numbered to be consistent with the previous review:

1a. In reporting radioactivity in each fraction, registrant should also report the initial sample TRR, allowing losses during extraction to be determined.

1b. (Not a deficiency)

1c. Because HPLC System II was used to confirm the identity of metabolites, representative chromatograms from the confirmatory analysis should be provided.

1d. The data provided indicate that metabolism is extensive, and little acifluorfen is found, in mature peanut commodities. However, the data are insufficient to identify the metabolites present in mature commodities; less than 1% of TRR in kernels, 10% of TRR in hulls, and 16% of TRR in vines have been identified. Unidentified polar metabolites in the organic fraction from the ACN:HCl extraction account for up to 43.3% of TRR (0.370 ppm) in vines, and 39.2% of TRR (0.404 ppm) in hulls.

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1e. Because of the small magnitudes of individual chromatographic peaks, further characterization of residues in peanut kernels is not required. In mature hulls and vines, each individual metabolite ≥ 10% of TRR should be identified. Residues extracted by either or both of the ACN:HCl method and the Bligh-Dyer method may be used for identification, but the overall tabulation of identified residues must be reconciled between the two extracts (that is, "double-counting" the same metabolite from both extracts is not allowed). Identification or characterization of residues should be consistent with Additional Guidance for Conducting Plant and Livestock Metabolism Studies, D. Edwards and E. Zager, 7/16/92, a copy of which is attached.

1f. Registrant proposed that metabolism of [^{14}C]sodium acifluorfen in peanut plants proceeds by conversion of the parent compound to form polar and acidic residues, reduction and cleavage of the nitro group, decarboxylation, and cleavage of the diphenyl ether bond. The registrant also believes that ^{14}C-fractions are incorporated into insoluble plant materials. Because polar metabolites and/or conjugates have not been identified, and evidence for incorporation into plant components has not been provided, the proposed metabolic scheme cannot be considered justified.

1g. Storage times and conditions at the analytical laboratory must be provided. If storage times were greater than 6 months between harvest and analysis, evidence should be provided that the profile of residues did not change during the period between collection and final analysis.

1h. Radiovalidation of method(s) using samples from the metabolism study remains an outstanding requirement, pending determination of the residues to be regulated.

In response, BASF has submitted (1993; MRID 42865802) supplemental data addressing these deficiencies. The registrant's response to each Conclusion and the present conclusions are presented below.

Registrant's response to Conclusion 1a: In the original report (1992; MRID 42368301), %TRR in the organosoluble, aqueous-soluble, and non-extractable fractions were presented as normalized values. BASF has recalculated the %TRR data for distribution of radioactivity in peanut matrices without normalization; this information is presented in Tables 1 and 2 of the current review which replace Tables 3 and 4 of the previous Agency review dated 12/9/92.

We find that Conclusion 1a is satisfied. Adequate information on the distribution of radioactivity into the organosoluble, aqueous-soluble, and non-extractable fractions has been submitted. These data indicated that minor losses of radioactivity occurred in the initial extraction of 27-day PTI kernels and in the hydrolysis of vines, hulls, and kernels non-extractable residues.

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Table 1. Distribution of radioactive residues in organic, aqueous, and non-extractable fractions of peanut vines, hulls, and kernels following one or two foliar applications of [^{14}C]sodium acifluorfen at 1x maximum registered single use rate.

Matrices and PTI [a] (days)	TRR as [^{14}C]sodium acifluorfen equivalents [b]						Total Recovery %
	Organic		Aqueous		Non-extractable		
	ppm	%	ppm	%	ppm	%	
Vines							
1st Application							
0	3.49 5.20	97.3 96.8	0.105 0.120	2.9 2.2	0.318 0.382	8.9 7.1	109.1 106.1
22	0.187 0.331	61.4 65.3	0.070 0.122	23.0 23.8	0.057 0.098	18.8 19.0	103.2 107.1
2nd Application							
1	5.330	98.0	0.210	3.7	0.524	9.6	111.3
27	0.370	60.1	0.153	24.9	0.163	26.5	111.5
48	0.267 0.361	52.4 60.1	0.145 0.131	28.5 21.8	0.118 0.157	23.1 26.1	104.0 108.1
62	0.435 0.437	59.9 51.2	0.178 0.238	24.5 27.8	0.200 0.202	27.5 23.6	112.0 102.7
62 [c]	0.067 0.159	9.2 21.9	0.337	46.4	0.204	28.1	105.6
Hulls							
2nd Application							
27	0.250	39.1	0.321	50.4	0.118	18.5	108.1
48	0.417	45.5	0.425	46.3	0.175	19.1	110.9
62	0.561	54.5	0.332	32.2	0.232	22.5	109.2
62 [c]	0.105 0.222	13.0 27.4	0.384	47.5	0.134	16.6	104.5
Kernels							
2nd Application							
27	0.023	18.4	0.041	33.6	0.041	33.9	85.8
48	0.030	19.4	0.052	33.0	0.092	58.6	111.0
62	0.027	18.9	0.057	40.0	0.082	57.2	116.1
62 [c]	0.010 0.027	7.2 19.5	0.059	42.5	0.054	39.1	108.3

[a]Posttreatment interval, days after first or second applications. [b]Radioactivity extracted by ACN:HCl method, unless indicated otherwise. [c]Radioactivity extracted by modified Bligh-Dyer method and partitioned into ethyl acetate and chloroform respectively.

Table 2. Radioactivity released by acid hydrolysis of the aqueous and non-extractable fractions from ACN:HCl extract of peanut vines, hulls, and kernels.

Fractions	TRR as [^{14}C]sodium acifluorfen equivalents					
	Vines		Hulls		Kernels	
	ppm	%	ppm	%	ppm	%
Initial TRR	0.854	100.0	1.031	100.0	0.143	100.0
Organosoluble	**0.437**	**51.2**	**0.561**	**54.5**	**0.027**	**18.9**
Aqueous-soluble	**0.238**	**27.8**	**0.332**	**32.2**	**0.057**	**40.0**
Hydrolyzed/aqueous	0.045	5.2	0.022	2.2	0.016	11.1
Hydrolyzed/EtOAc	0.182	21.3	0.290	28.1	0.037	25.7
Unaccounted [a]	0.011	-	0.020	1.9	0.004	2.8
Non-extractable	**0.202**	**23.6**	**0.232**	**22.5**	**0.082**	**57.2**
Hydrolyzed/aqueous	0.023	2.7	0.014	1.3	0.037	25.9
Hydrolyzed/EtOAc	0.017	2.0	0.039	3.8	0.022	15.1
Unhydrolyzed	0.092	10.7	0.100	9.7	0.012	8.7
Unaccounted[a]	0.070	8.2	0.079	7.7	0.011	7.7

[a] Calculated by the study reviewer.

Registrant's response to Conclusion 1c: BASF has submitted representative copies of HPLC System II chromatograms for the mixture of standards and for TLC Regions A, B, C, and D by UV and LSC/fraction collection detection.

We find that Conclusion 1c is satisfied.

Registrant's response to Conclusions 1d and 1e: The registrant explained that the number of products in the "polar fraction" could not be determined from the data obtained during the study. However, the registrant noted that results of a new metabolism study currently being conducted with peanuts indicate that many polar components and very small amounts of acifluorfen and its related metabolites are found in mature commodities. An effort is being made to characterize these components and to release possible conjugated residues from the polar components.

We conclude that registrant is conducting a new peanut metabolism study to resolve Conclusions 1d and 1e.

Registrant's response to Conclusion 1f: Registrant states that it has no disagreement with this statement. It notes that metabolism is considerable with the peanut plant. The nature of the residue in peanut will not be adequately understood until after submission of the new metabolism study.

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Registrant's response to Conclusion 1g: BASF notes that samples of peanut commodities were stored frozen at <-10 C at the analytical laboratory (Hazelton Laboratories) for up to 596 days prior to extraction. The registrant stated that storage stability of acifluorfen and its metabolites in peanut vine samples for 574 days is demonstrated by the fact that >83% of TRR in three vine samples harvested within one day of application and extracted after 574 days of storage were found to be the parent acifluorfen and its related metabolites. The registrant also stated that the metabolism study with peanuts that is currently in progress should provide additional information regarding storage stability in peanuts.

We find that Conclusion 1g is not satisfied. Dates of analysis and evidence that profiles of residues did not change during the period between collection and final analysis were not provided. Registrant reported that it is conducting a new peanut metabolism study, which should provide additional information on storage stability.

Registrant's response to Conclusion 1h: BASF noted that the acetonitrile:1.0 N hydrochloric acid (7:3, v:v) extraction procedure used for many of the samples in the study is similar to the extraction procedure used in the enforcement method. The metabolism study demonstrated that acifluorfen, amino acifluorfen, descarboxyl-acifluorfen, and desnitro-acifluorfen would be extracted from peanut matrices using the acetonitrile:1.0 N HCl (7:3, v:v) extraction procedure.

We appreciate the information provided by registrant relevant to the enforcement method. CBRS recognizes that the generic requirement for radiovalidation cannot be resolved until after the nature of residue in plants is adequately understood, residues to be regulated have been determined, and the method is validated using samples from metabolism studies.

Qualitative Nature of the Residue in Rice

An Agency memorandum (CBRS No. 10199, DP Barcode D180455, 12/9/92, J. Abbotts), concluded that the qualitative nature of the residue in rice is not adequately understood for the following reasons. Conclusions are numbered to be consistent with the previous review:

2a. The registrant should resolve the discrepancies in the reported HPLC retention times for reference standards of acifluorfen and amino-acifluorfen vs. the reported retention time for acifluorfen in rice samples. This discrepancy could be resolved by the submission of HPLC profiles of reference compounds recorded on the same day as the analysis of metabolites.

2b. (Not a deficiency, provided identification of residues is adequate.)

2c. Registrant should explain in more detail how quantitative assignment of residues in rice grain was made. Assignments should be supported by calculations and data on radioactivity applied to TLC plates and recovered.

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2d. Organosoluble residues from hulls should be examined for the putative acifluorfen peak; identification of acifluorfen by two methods in hull extracts could be translated to grain.

2e. For each identified metabolite (whether tentative or confirmed) in rice straw, complete quantitative data must be provided. These data should be presented in a table which must include the amount (% of TRR and ppm equivalents) for each extractable fraction of rice straw. The amount (% of TRR and ppm equivalents) of unidentified polar metabolites should also be provided. Further characterization or identification of residues should be conducted based on Additional Guidance for metabolism studies. Cleavage by appropriate enzymes should facilitate identification of putative conjugates.

2f. The registrant proposed that [^{14}C]sodium acifluorfen is metabolized in rice by two pathways: (i) a route involving formation of amino acifluorfen by the reduction of nitro group; and (ii) a route involving rapid cleavage of the diphenyl ether bond. Only two metabolites, acifluorfen and amin-acifluorfen, were identified in rice commodities, and the identification of acifluorfen was confirmed only in straw. The proposed metabolic route involving cleavage of the diphenyl ether bond cannot be considered justified until putative conjugated metabolites are identified.

2g. The data presented are insufficient to validate the stability during frozen storage of organosoluble residues in grain, nor aqueous soluble residues in straw. Storage conditions between harvest and homogenization should be provided for grain and straw. Considering the very long storage times, evidence should be provided that the profiles of radioactive residues did not change during storage. If chromatographic profiles at early extraction are not available, then material balances indicating that residue identification and quantitation account for nearly all residues at early extraction will be sufficient evidence for storage stability.

2h. Radiovalidation of method(s) using samples from the metabolism study remains an outstanding data requirement, pending determination of the residues to be regulated.

In response, BASF has submitted (1993; MRID 42865801) supplemental data addressing these deficiencies. The registrant's response to each deficiency and the CBRS conclusions are presented below.

Registrant's response to Conclusion 2a: The registrant states that the discrepancy in retention times between the acifluorfen and amino-acifluorfen reference standards and the acifluorfen and amino-acifluorfen in rice samples was due to the fact that the UV detector associated with the HPLC was located prior to the LSS fraction collector. Non-radioactive reference standards were measured by the UV detector while ^{14}C-residues in collected fractions were measured by LSS. Hence, there was a lag time between the two measurements. To support this, BASF submitted copies of the HPLC chromatograms (UV detection) and radiochromatograms (LSS/fraction collection detection) of [^{14}C]amino

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acifluorfen and [^{14}C]acifluorfen reference standards and rice plant extracts run on the same day. The registrant notes that the HPLC retention times of these two compounds were consistent between runs on different days and are consistent with the identification of acifluorfen as the major plant residue.

We find that Conclusion 2a is satisfied.

Registrant's response to Conclusion 2c: The registrant explained the quantitative assignment of residues in rice grain as follows; equations used for calculation of residues were provided. Acifluorfen residues were quantitated by purification of the organic extract using TLC System A, which yielded two peaks (Rf = 0.34 and 0.67). Following purification of the first peak (Rf = 0.34) using TLC System A, the peak corresponding to acifluorfen (Rf = 0.43) was scraped from the silica plate; analysis of the radioactivity by LSS indicated that the fraction contained 0.0084 ppm.

Released amino acifluorfen residues were quantitated by purifying the residues released by sodium hydroxide and partitioned into ethyl acetate using TLC System A, which yielded two peaks (Rf = 0.52 and 0.86). The first peak (Rf = 0.52) was scraped and analyzed by TLC (solvent B). The peak corresponding to amino acifluorfen (Rf = 0.71) was scraped from the plate and analyzed by LSS which indicated that the peak contained 0.0105 ppm.

The registrant noted that analysis of the aqueous phase following hydrolysis and ethyl acetate extraction revealed non-detectable radioactive residues and that residues in the non-extractable portion following hydrolysis were also non-detectable (the rice grain dissolved in the aqueous sodium hydroxide during hydrolysis).

Extracted amino acifluorfen residues could not be directly quantitated since the fraction containing amino acifluorfen was combined with a rice hull extract prior to identification. However, the registrant quantitated these residues by difference, as follows:

Total radioactive residues in rice grain		0.0273 ppm
minus	extracted acifluorfen	0.0084 ppm
minus	released amino acifluorfen	0.0105 ppm
minus	aqueous NaOH hydrolysate	<MQL
minus	non-extractable after hydrolysis	<MQL
equals	extracted amino acifluorfen	0.008 ppm

We find that Conclusion 2c is satisfied.

Registrant's response to Conclusion 2d: The registrant states that the TLC chromatograms of non-hydrolyzed and hydrolyzed hull extracts are very similar to those of the corresponding grain fractions; an experiment is planned to examine organosoluble residues in hulls for the putative acifluorfen peak.

We conclude that the additional work planned in rice hulls is necessary to resolve Conclusion 2d.

Registrant's response to Conclusion 2e: The registrant presented data showing the distribution of [^{14}C]sodium acifluorfen residues in each extractable fraction of rice straw; these data are represented in Table 3 and a summary of the metabolites found in rice straw is presented in Table 4. Acifluorfen accounted for the majority of the extracted radioactivity (~77% of TRR, 1.54 ppm). Based on the data in Table 3 here, Polar peak 1 accounts for a total of 18.0% of TRR (0.36 ppm). The registrant stated that Polar peak 1 is the only HPLC fraction that could be viewed as needing further work. The registrant noted that this peak is likely a mixture of polar compounds since it eluted at or near the void volume. In addition, the registrant does not believe that enzymatic cleavage would be any more effective than base hydrolysis at releasing putative conjugates or converting these polar compounds into one or more less polar compounds.

CBRS acknowledges that acifluorfen represents the large majority of TRR in rice straw. Polar peak 1 represents most of the remaining TRR. If Polar peak 1 cannot be resolved into multiple peaks with a second system (a different HPLC system would be acceptable in this regard, then it should be identified. Other than Polar peak 1, no further work on unidentified peaks or fractions is necessary.

Table 3. Distribution of total radioactive residues (TRR) in rice straw (TRR = 2.0 ppm) treated with [^{14}C]sodium acifluorfen at 3.4x the maximum registered single use rate and harvested 97 days after the final treatment.

Fraction	% TRR	ppm	Characterization/Identification
Acetone/water	85.3	1.71	Further fractionated.
EtOAc	75.2	1.51	Acifluorfen (75.2% TRR, 1.51 ppm) was identified.
Aqueous	10.1	0.20	Polar peak 1 (10.1% TRR, 0.20 ppm) was resolved.
NaOH/EtOAc	5.4	0.11	Polar peak 1 (5.4% TRR, 0.11 ppm) was resolved.
NaOH/Aqueous	4.0	0.08	Acifluorfen (0.4% TRR, 0.01 ppm) was identified. Polar peak 2 (3.6% TRR, 0.07 ppm) was resolved.
Solids	14.7	0.30	N/A = Not further analyzed.
Aqueous NaCl	2.4	0.05	N/A
EtOAc	1.5	0.03	Acifluorfen (1.2% TRR, 0.02 ppm) was identified. Polar peak 1 (0.3% TRR, 0.01 ppm) was resolved.
Aqueous	0.9	0.02	N/A
Solids	-	-	N/A
Aqueous EDTA	0.5	0.01	N/A
Solids	-	-	N/A
Aqueous NaOH	5.6	0.11	N/A
EtOAc	5.2	0.10	Polar peak 1 (2.3% TRR, 0.04 ppm) was resolved. The remaining 2.9% TRR (0.06 ppm) consisted of at least three components.
Aqueous	1.6	0.03	N/A
Nonextractable	2.4	0.05	N/A

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Table 4. Summary of identification/characterization of radioactive residues in rice straw treated with [^{14}C]sodium acifluorfen at 3.4x the maximum registered single use rate and harvested 97 days after the final treatment.

Metabolite	% TRR	ppm
Identified		
Acifluorfen	76.8	1.54
Characterized		
Polar peak 1	18.0	0.36
Polar peak 2	3.6	0.07
Total	**98.4**	**1.97**
Unanalyzed	5.9	0.12
Non-extractable	2.4	0.05

Registrant's response to Conclusion 2f: Registrant agreed that there is no need to invoke cleavage of the diphenyl ether bond. Registrant noted that there is litle metabolism of acifluorfen in rice straw, and only small amounts of residue are present in grain. This Conclusion can be considered satisfied, but additional work is necessary before the nature of the residue in rice is understood.

Registrant's response to Conclusion 2g: BASF stated that rice grain samples were stored frozen at <-10 C at the analytical laboratory (ABC Laboratories) for approximately six months prior to analysis and concluded that storage stability data for grain residues are not needed. Rice straw samples were stored frozen at <-10 C for up to 50 months prior to extraction. The registrant presented data pertaining to the distribution of radioactivity into the organosoluble, aqueous-soluble, and non-extractable fractions of rice straw after 1, 37, and 50 months of storage which indicated that the %TRR in each fraction was similar at each storage interval. HPLC and TLC analyses of the organosoluble fraction at various storage intervals were presented in the original report. The registrant stated that no analyses of the aqueous-soluble residues of rice straw were conducted early in the study.

We conclude that registrant has provided the storage stability data requested in the earlier review. Relevant storage stability data should also be provided in support of the additional work planned to satisfy Conclusion 2d and recommended to satisfy Conclusion 2e.

Registrant's response to Conclusion 2h: The registrant presented a discussion comparing extraction procedures used in the current rice metabolism study with a (proposed) enforcement method, Rhone-Poulenc Agrochemical Method 160, submitted in MRID 00122722, dated 7/82. The extraction solvents are 1% acetic acid in acetonitrile for Method 160. The registrant stated that the extraction solvents used in the metabolism study, ethyl acetate or ethyl acetate followed by methanol for rice grain samples and acetone or acetone:water for rice straw samples, are very similar to those of Method 160. In addition,

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the registrant noted that the majority of the residues in rice grain and straw were found to be compounds which are very soluble in organic solvents. The registrant additionally noted that problems with a rice storage stability study were recently noted which appear to be method related and that these problems are undergoing investigation.

We appreciate the information provided by registrant relevant to the enforcement method. CBRS recognizes that the generic requirement for radiovalidation cannot be resolved until after the nature of residue in plants is adequately understood, residues to be regulated have been determined, and the method is validated using samples from metabolism studies.

AGENCY MEMORANDA CITED IN THIS REVIEW

CBRS No.: 10199
Subject: Sodium Acifluorfen, Reregistration. BASF Corporation Response to Phase 4 Review. Metabolism in Peanuts and Rice.
From: J. Abbotts
To: J. Ellenberger
Dated: 12/9/92
MRID(s): 42368301 and 42368302

MASTER RECORD IDENTIFICATION NUMBERS

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

42865801 Panek, E.J., Winkler, V.W., and Jordan, R. (1993) Metabolism of [^{14}C]Acifluorfen-Sodium in Rice Supplementary Report to MRID 42368302. BASF Report No.: M9318. BASF Registration Document No. 93/5071. Unpublished study conducted by BASF Corporation. 89 p.

42865802 Panek, E.J., Winkler, V.W., and Jordan, R. (1993) Nature of ^{14}C-Sodium Acifluorfen Residue in Peanuts Supplementary Report to MRID 42368301. BASF Report No.: M9319. BASF Registration Document No. 93/5072. Unpublished study conducted by BASF Corporation. 56 p.