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

MAY 4 1994

MEMORANDUM:

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

SUBJECT: Sodium Acifluorfen (114402). Nature of the Residue in Rice 171-4(a); MRID #431820-01; CBRS 13509; DP Barcode 201623; Supplemental data to upgrade Rice Metabolism Study.

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Data were submitted in response to previous reviews, which concluded that additional work was required (CBRS 10199, 12/9/92, J. Abbotts and CBRS 12380, 11/3/93, J. Abbotts). The present submission specifically addresses deficiency 2d for Qualitative Nature of the Residue in Rice. A brief chronology of activity concerning deficiency 2d follows:

Deficiency 2d (CBRS 10199, 12/9/92, J. Abbotts):

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

Registrants response (1993; MRIDs 42865801 and 42865802) :

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.

Agency comment (CBRS 12380, 11/3/93, J. Abbotts):

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



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Registrants current submission (1994: MRID 431820-01):

Registrant submitted results of a study entitled: Identification of Residues From [CF3-Ring UL 14C-] in Rice Hulls. (Supplemental Study to that in ABC Report 35986, MRID No. 42368 02). This study is briefly described below:

Materials: Rice hulls from plants treated (0.43 lb. ai/A pre-emergence) with C-14 labelled acifluorfen were analyzed. Samples were obtained from ABC Laboratories Study ID: 35986 (1992; MRID 423683-02), which was previously reviewed in connection with CBRS 10199 (12/9/92, J. Abbotts) and CBRS 12380 (11/3/93, J. Abbotts). Twenty grams of rice hulls were available for the present study.

Extraction: Rice hulls were macerated with 3 parts water and allowed to stand for 3 hours. Three parts acetone were added and maceration was repeated. After filtration hulls were macerated twice with 4 parts 25% aqueous acetone. Acetone was evaporated, aqueous residue adjusted to pH 3, and extracted 3 times with 1.5 parts ethyl acetate. Portions of aqueous residue were made 2 N with HCL or KOH and kept at 110C for 3 hours. Resulting solutions were extracted twice with ethyl acetate at low pH.

Characterization: Radioactive components were characterized by Liquid Scintillation counting (Beckman LS 150) and combustion analysis (Harvey Biological Oxidizer).

Identification: Radioactive components were separated using four separate TLC systems:

	Plate	Solvent	
A:	Silica Gel	Ethyl acetate	60
	Preabsorbent	Hexzane	39
	Layer	Acetic Acid	1
B:	Silica Gel	Toluene	70
	Preabsorbent	Methanol	30
	Layer		
C:	C-18	Metanol	75
		Water	24
		Acetic Acid	1
D:	Silica Gel	Ethyl Acetate	60
	Aluminium	Hexzane	39
		Acetic Acid	1

Radioactive components were co-chromatographed with non-radioactive reference standards of:

Aciflourfen
Descarboxy Acifluorfen
Desnitro Acifluorfen
Amino acifluorfen
Acetamide

Non-labelled standards were visualized by "quenching of the fluorescence of a dye in the layer introduced through UV light." Radioactive materials were detected by a "Ambis 100 radioanalytical imaging system."

Study Results:

Fractionation of Radioactive Components in rice hulls (0.15 ppm acifluorfen equivalence)

Initial extraction of Rice Hulls (0.15 ppm)

Aqueous acetone	80.4% (0.12 ppm)
non-extracted	19.6% (0.03 ppm)

Partition of Aqueous extract (0.12 ppm)

Aqueous #1	61.3% (0.07 ppm)
Ethyl Acetate #1	19.1% (0.02 ppm)

Acid Hydrolysis; Aliquot of Aqueous #1 (0.12 ppm)

Aqueous #2	00.0% (0.00 ppm)
Ethyl acetate #2	40.8% (0.05 ppm)

Base Hydrolysis; Aliquot of Aqueous #1 (0.12 ppm)

Aqueous #3	10.8% (0.01 ppm)
Ethyl acetate #3	49.3% (0.06 ppm)

TLC analysis performed on ethyl acetate fractions #1 and #3 are summarized in table 1 and 2 below. TLC system D was not successful because without a pre-absorbent layer on the plate the spotting of a sufficient amount of material was not possible.

Table 1: TLC Analysis of Ethyl acetate fraction #1: (0.02 ppm)

TLC/system	Rf	Acifluorfen %TRR(ppm)	Rf	Major Unknown %TRR(ppm)
A	0.23	5.3%(0.008)	0.15	13.8%(0.021)
B	0.48	7.0%(0.011)	0.37	12.1%(0.018)
C	0.53	6.2%(0.009)	0.75	8.3%(0.012)

Table 2: TLC Analysis of Ethyl acetate fraction #3: (0.06 ppm)

TLC/system	Rf	Unknown 1 %TRR(ppm)	Rf	Unknown 2 %TRR(ppm)
C	0.02	21.5%(0.03)	0.08	20.3%(0.03)

Conclusion/recommendation:

Registrant has examined organosoluble fraction of rice hulls for acifluorfen. Results showed 5.3 to 7.0% (0.008 to 0.011 ppm) of the organosoluble residues from rice hulls to be acifluorfen. Deficiency 2d (CBRS 10199, 12/9/92, J. Abbotts) is resolved.

cc:Circ, RF, Sodium Acifluorfen List B File, SF.
RDI:MMetzger:05/02/94:EZager:05/02/94
H7509C:CBRS:FSuhre:05/03/94