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

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: July 2, 2003

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DP Barcode: D278386

Petition: 1F06313

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Hamm, R.; Veit, P. (2002) Amendment to: Confined Rotational Crop Study with (Carbon 14)-BAS 510 F: Lab Project Number: 41838: 2000/1014862: Unpublished study prepared by BASF Aktiengesellschaft. BASF Document 2002/1004122. 4 p.

Sponsor: BASF Corporation

Background

The information contained herein was compiled by Dynamac Corporation (20440 Century Boulevard, Suite 100, Germantown MD 20874), contractor, under the supervision of RAB2/ HED. This DER has undergone secondary review by PMRA/Canada, and peer review by RAB2, and reflects current HED and Office of Pesticide Programs (OPP) policies.

Executive Summary

BASF Corporation has submitted a confined rotational crop study with BAS 510 F. The in-life and analytical phases of the study were conducted by BASF (Limburgerhof, Germany). Radiolabeled [^{14}C]BAS 510 F, labeled at the 3-position of the pyridine ring or uniformly labeled on the phenyl rings, was applied to bare soil at 1.88 lb ai/A (2100 g ai/ha). Rotational crops, lettuce, radish, and wheat, were planted in treated soil 30, 120, 270, and 365 days following treatment of the soil. Immature wheat forage, and mature lettuce, radish root and top, and wheat straw and grain were harvested from each of the plantback intervals (PBIs). TRR were determined by combustion/LSC. The petitioner used the calculated TRR values (extractable radioactivity plus nonextractable residues) for reporting results. Calculated TRR in diphenyl- and pyridine-label samples ranged 0.028-0.084 and 0.022-0.161 ppm in lettuce, 0.150-0.337 and 0.113-0.343 ppm in radish tops, 0.030-0.098 and 0.017-0.066 ppm in radish roots, 0.265-1.575 and 0.230-0.690 ppm in wheat forage, 1.404-9.826 and 1.614-4.008 ppm in wheat straw, and 0.023-0.243 and 0.147-0.285 ppm in wheat grain, respectively. Material balances, based on combustion of the samples, were 77.2-127.3% and 51.7-112.6% for the diphenyl and pyridine labels, respectively; exceptionally low or high material balances were observed for certain matrices: 230% for pyridine-label lettuce (120-day PBI), 178.5% for pyridine-label wheat straw (120-day PBI), 40.0% for pyridine-label wheat forage (270-day PBI) and 564.6% for pyridine-label wheat grain (270-day PBI).

For both labels, approximately 62-97% of the TRR were extracted using methanol, and aqueous ammonia in some cases, from all rotational crop matrices except wheat grain; only 18-58% of the TRR were extracted from wheat grain using methanol and aqueous ammonia. Nonextractable residues of rotational crops were further fractionated using aqueous ammonia hydrolysis, sodium hydroxide hydrolysis, and/or DMSO extraction, to yield fractions that the petitioner attributed to protein, cellulose, lignin, and/or starch. The methanol extracts were analyzed by HPLC, and the results were confirmed by a second HPLC system. Identifications of the parent and the metabolite M510F61 were confirmed by LC/MS/MS in 30-day PBI wheat straw samples.

A total of 52.6-96.1% of the TRR was identified in **lettuce, radish roots and tops, and wheat forage and straw** from the 30-, 120-, 270-, and 365-day PBIs. The parent, BAS 510 F was the major residue identified in these commodities, accounting for 50.0-96.1% TRR (0.009-3.156 ppm). The glucoside metabolite M510F61 was identified in radish tops (all PBIs), radish roots (120- and 365-day PBIs only), wheat forage (all PBIs), and wheat straw (all PBIs); M510F61 accounted for 0.9-21.2% TRR (0.001-0.423 ppm). Unknown metabolites accounted for up to 22.7% TRR; however, individual peaks that were present at >10% TRR were each <0.04 ppm. Bound residues in certain samples of lettuce, radish tops and roots, and wheat forage and straw were tentatively demonstrated to be due to the incorporation of radioactivity into protein, lignin, and cellulose at levels totaling up to 9.7% TRR. The remaining nonextractable residues were <0.05 ppm and were not further characterized.

A total of 1.9-35.4% of the TRR was identified in **wheat grain** from the 30-, 120-, 270-, and 365-day PBIs. The parent, BAS 510 F, was the only compound identified in wheat grain at all

PBIs, accounting for 9.6-35.4% TRR (0.008-0.028 ppm) in diphenyl-label grain and 1.9-6.1% TRR (0.005-0.015 ppm) in pyridine-label grain. Unknown metabolites accounted for ≤5.6% TRR (≤0.010 ppm). Bound radioactivity in wheat grain was tentatively demonstrated to be due to incorporation of radioactivity into starch, at 0.6-4.3% TRR (0.001-0.004 ppm) for the diphenyl label samples and 36.2-48.4% TRR (0.061-0.118 ppm) for the pyridine label samples, and protein, at 4.9-9.6% TRR (0.002-0.012 ppm) for the diphenyl label samples and 3.1-9.6% TRR (0.005-0.027 ppm) for the pyridine label samples. Nonextractable residues were 30.3-71.6% TRR (0.007-0.174 ppm) in diphenyl-label grain samples and 17.4-26.4% TRR (0.026-0.072 ppm) in pyridine-label grain samples; these residues were not further characterized.

The TRR in soil decreased over the duration of the study, with only the parent identified. Therefore, the petitioner proposed that BAS 510 F is not metabolized in the soil, and that the metabolite M510F61 identified in several crop matrices was a result of metabolism of BAS 510 F in the plants.

The study is **acceptable** to satisfy the data requirement for a confined rotational crop study.

GLP Compliance

Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. The petitioner stated that the study was conducted in accordance with the GLP regulations established in Germany (Appendix 1 to §19a Section 1, Chemikaliengesetz of 25-July-1994; Official Bulletin/Federal Republic of Germany I 1994, p. 1703) instead of U.S. EPA GLP regulations.

1. Materials and Methods

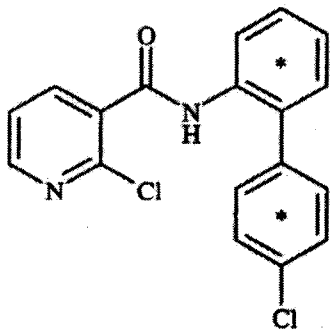
1.1. Substance

Active Ingredient

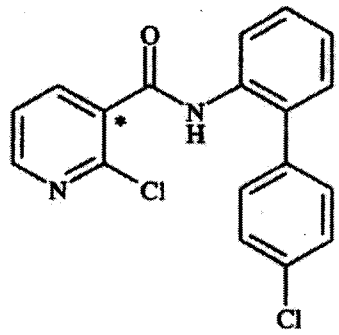
Common Name:	Nicobifen (ISO, proposed)
IUPAC Name:	2-Chloro-N-(4'-chlorobiphenyl-2-yl)nicotinamide
CAS Name:	3-Pyridinecarboxamide, 2-chloro-N-(4'chloro[1,1'-biphenyl]-2-yl)-
CAS Number:	188425-85-6
Company Name:	BAS 510 F
Other Synonyms:	BASF Registry No. 300355

Location of Isotopic Label (diphenyl label): Uniformly labeled in both phenyl rings
Radiochemical Purity: >99% (analyzed by HPLC)
Specific Activity: 376,000 dpm/µg

Location of Isotopic Label (pyridine label): Labeled at the 3-position in the pyridine ring
Radiochemical Purity: >99% (analyzed by HPLC)
Specific Activity: 349,000 dpm/µg



^{14}C-Diphenyl label



^{14}C-Pyridine label

1.2. Crop and Application Information

Types and Varieties of Crops: Head lettuce, radish, and wheat (varieties were not identified)
Growth Environment: Nineteen polyethylene containers containing loamy sand soil were maintained in a growth chamber, vegetation hall, or green house (BASF Agricultural Center, Limburgerhof, Germany).
Conditions: The soil was kept lightly moistened during the study; the temperature and humidity maintained in the growth chamber were not reported. After the soil aging period, ploughing was simulated by either removing the top 20 cm of soil from the containers and mixing in a concrete mixer or breaking up the top layer of soil (~20 cm) with a spade.

1.3. Application

Type of Application: Soil incorporated; the top layer of soil (1 cm), containing the test substance, was uniformly spread over the untreated soil in the containers.
Application Matrix: The radiolabeled test substances were dissolved in acetone and mixed with top layer soil.
Application Rate: 1.88 lb ai/A (2100 g ai/ha)
Number of Applications: One
Plantback Interval(s): 30, 120, 270, and 365 days

1.4. Harvest/Post-harvest Procedures

Lettuce heads were harvested, leaving the roots in the soil, 58-101 days after planting (DAP); radishes were pulled from the soil, 58-114 DAP, and separated into roots and tops; and mature wheat was harvested by cutting the plants just above the soil level, 124-191 DAP, and separated into grain, straw, and chaff. Immature wheat forage was harvested 55-111 DAP; the method of harvest was not specified. All samples were frozen (-18 C) immediately following sampling and remained frozen until analysis by BASF (Limburgerhof, Germany).

Table 1.4.1. Summary of Storage Conditions

Matrix	RAC or Extract	Storage Temperature (°C)	Duration (days)*
Lettuce	Heads	≤-18	60-491
Radish	Roots	≤-18	27-479
	Tops	≤-18	27-479
Wheat	Forage	≤-18	57-476
	Straw	≤-18	29-598
	Chaff	≤-18	Not specified
	Grain	≤-18	29-412

* Storage interval determined from raw data reports. Has not been audited by GLP.

Supporting storage stability data were included in the study. Samples of wheat straw (both labels) were re-extracted and analyzed at the end of the study, after storage for 595 days for diphenyl-label straw and 668 days for pyridine-label straw. The extraction pattern and metabolite profile were similar in the stored samples to those used in the original analysis. In addition, the methanol extracts of wheat straw (both labels) which had been stored for 581 days (diphenyl-label straw) or 640 days (pyridine-label straw) were re-analyzed by HPLC. Again, the metabolite pattern was the same in the stored extracts as in the original analysis. These data support storage of the diphenyl-label samples/extracts for up to ~20 months and storage of the pyridine-label samples/extracts for up to ~22 months prior to completion of analysis.

1.5. Analytical Methods

All rotated crop samples (both labels) were homogenized with liquid nitrogen, and the total radioactive residues (TRR) were determined by combustion/LSC. The limit of quantitation (LOQ) was reported as 0.00148 ppm for pyridine-label wheat straw and 0.00138 ppm for diphenyl-label wheat straw. TRR values were also calculated by the petitioner by summing the radioactivity in the extractable and nonextractable fractions. The calculated TRR values were used for all further calculations.

In general, an aliquot of homogenized plant sample was extracted (3x) with methanol (MeOH), and the MeOH extracts isolated by centrifugation and combined for HPLC analysis. Nonextractable residues of certain matrices (see Tables 2.2.1-2.2.12) were extracted with an

aqueous ammonia solution (concentration not specified); in most cases, the samples were then shaken at 40 C. Concentrated HCl was added to the ammonia extract, which yielded a precipitate (attributed to protein). The aqueous portion (diphenyl labels only) of 30-day PBI wheat forage, straw, and grain; 120-day PBI radish roots and tops, and wheat forage, straw, and grain; 270-day PBI wheat forage, straw, and grain; and 365-day PBI lettuce, and wheat forage, straw, and grain were then partitioned with dichloromethane (DCM).

For selected matrices, the nonextractable residues remaining following extraction with aqueous ammonia and hydrolysis with HCl were subjected to cellulose and lignin fractionations (see Tables 2.2.1-2.2.12 for matrices). An aliquot of the nonextractable residue was refluxed with 10% NaOH for 3 hours and filtered. The remaining residue was washed first with 10% NaOH at 80 C and then with water. The washed residue (attributed to cellulose) was dried for combustion/LSC analysis. The NaOH hydrolysate and NaOH and water washes were combined, and concentrated HCl was added with stirring until the pH dropped to acidic conditions and a precipitate formed. The precipitate (attributed to lignin) was isolated by centrifugation for combustion/LSC analysis.

Nonextractable residues following ammonia extraction of wheat grain were subjected to starch determinations. An aliquot of the nonextractable residue was extracted (2x) with DMSO:water (9:1, v:v) and centrifuged. The extracts were combined, and ethanol was added to precipitate starch (mixture refrigerated overnight). The precipitate and ethanol extract were separated by centrifugation for LSC analysis. For further characterization of the starch fraction, iodine/potassium iodide was added to the ethanol extract as an indicator of the presence or absence of starch.

All MeOH extracts were analyzed by HPLC, using two systems; no other extracts were analyzed. HPLC analyses were conducted with a Hamilton PRP-1 or a Nucleosil C18 semi-preparative column, using a gradient mobile phase of water:acetonitrile:formic acid (950:50:1 and 50:950:1, v:v:v or 950:50:2 and 50:950:2, v:v:v); the detector was not specified. Metabolites were identified by co-chromatography and/or comparison of retention times with those of the reference standards. The following reference standards were listed by the petitioner: ^{14}C-BAS 510 F, ^{14}C-M510F62 [1-(chlorophenyl)-2-aminobenzene[diphenyl-U-^{14}C]], and ^{14}C-M510F47 (2-chloronicotinic acid-[pyridine-3-^{14}C]).

The identification of BAS 510 F isolated from 30-day PBI wheat straw (both labels) and metabolite M510F61 isolated from 30-day PBI wheat straw (diphenyl label) was confirmed by electrospray ionization LC/MS/MS. LC/MS/MS analyses were conducted using a Nucleosil C18 column and a gradient mobile phase similar to that used for HPLC analyses. We note that the petitioner also included an LC/MS/MS chromatogram which indicated the presence of metabolite M510F59 in 30-day PBI wheat straw (diphenyl label).

2. Results

Table 2.1. Total Radioactive Residues in Rotational Crops Planted Following Application of Isotopically Labeled BAS 510 F to Bare Soil at 1.88 lb ai/A.

Label Location	Crop Matrix	Plantback Interval (days)	TRR, ppm		% Mass Balance
			Combustion[1]	Calculation[2]	
Diphenyl label	Lettuce	30	0.064	0.050	78.1%
		120	0.096	0.084	87.5%
		270	0.071	0.067	94.4%
		365	0.022	0.028	127.3%
	Radish tops	30	0.379	0.337	88.9%
		120	0.321	0.294	91.6%
		270	0.174	0.150	86.2%
		365	0.256	0.207	80.9%
	Radish roots	30	0.088	0.072	81.8%
		120	0.055	0.052	94.5%
		270	0.112	0.098	87.5%
		365	0.030	0.030	100.0%
	Wheat forage	30	1.702	1.575	92.5%
		120	1.269	0.980	77.2%
		270	0.525	0.562	107.0%
		365	0.314	0.265	84.4%
	Wheat straw	30	9.687	9.826	101.4%
		120	4.304	3.912	90.9%
		270	3.256	3.226	99.1%
		365	1.527	1.404	91.9%
	Wheat grain	30	0.173	0.166	96.0%
		120	0.253	0.243	96.0%
		270	0.023	0.023	100.0%
		365	0.051	0.048	94.1%
Pyridine label	Lettuce	30	0.041	0.035	85.4%
		120	0.070	0.161	230.0%
		270	0.060	0.031	51.7%
		365	0.023	0.022	95.7%
	Radish tops	30	0.494	0.343	69.4%
		120	0.199	0.211	106.0%
		270	0.227	0.125	55.1%
		365	0.123	0.113	91.9%

Table 2.1. Total Radioactive Residues in Rotational Crops Planted Following Application of Isotopically Labeled BAS 510 F to Bare Soil at 1.88 lb ai/A.

Label Location	Crop Matrix	Plantback Interval (days)	TRR, ppm		% Mass Balance
			Combustion[1]	Calculation[2]	
Pyridine label	Radish roots	30	0.057	0.048	84.2%
		120	0.037	0.038	102.7%
		270	0.032	0.017	53.1%
		365	0.065	0.066	101.5%
	Wheat forage	30	0.613	0.690	112.6%
		120	0.808	0.433	53.6%
		270	0.575	0.230	40.0%
		365	0.433	0.255	58.9%
	Wheat straw	30	3.632	3.609	99.4%
		120	2.246	4.008	178.5%
		270	2.337	1.614	69.1%
		365	3.275	1.925	58.8%
	Wheat grain	30	0.154	0.147	95.5%
		120	0.285	0.285	100.0%
		270	0.048	0.271	564.6%
		365	0.160	0.148	92.5%

[1] As determined by direct combustion/LSC.

[2] Calculated by summing extractable and nonextractable residues. The petitioner used the calculated value for all reported results.

Table 2.2.1. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Lettuce** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.050 ppm)						
MeOH extract	93.8	0.047	BAS 510 F	93.8	0.047	
Nonextractable	6.2	0.003	N/A[1]			
120-day PBI (TRR = 0.084 ppm)						
MeOH extract	89.2	0.075	BAS 510 F	85.2	0.072	Plus 1 unknown at 4.0% TRR (0.003 ppm).
Nonextractable	10.8	0.009	N/A			
270-day PBI (TRR = 0.067 ppm)						
MeOH extract	94.1	0.063	BAS 510 F	94.1	0.063	
Nonextractable	5.9	0.004	N/A			
365-day PBI (TRR = 0.028 ppm)						
MeOH extract	62.8	0.018	BAS 510 F	55.6	0.016	Plus 1 unknown at 7.2% TRR (0.002 ppm).
Nonextractable	37.2	0.010	N/A			Extracted with aqueous ammonia.
Ammonia extract	13.5	0.004	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	2.2	0.001	N/A			
Aqueous (NH_3)	15.4	0.004	N/A			
Precipitate	0	0.0	N/A			Attributed to protein.
Nonextractable	NR[2]	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	16.3	0.005	N/A			
Precipitate	4.5	0.001	N/A			Attributed to lignin.
Solids	5.2	0.001	N/A			Attributed to cellulose.

[1] Not analyzed.
[2] Not reported.

Table 2.2.2. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Radish Tops** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.337 ppm)						
MeOH extract	96.1	0.324	BAS 510 F	90.2	0.304	
			M510F61	5.9	0.020	
Nonextractable	3.9	0.013	N/A[1]			
120-day PBI (TRR = 0.294 ppm)						
MeOH extract	84.4	0.248	BAS 510 F	71.2	0.209	Plus 1 unknown at 13.2% TRR (0.039 ppm).
Nonextractable	15.6	0.046	N/A			Extracted with aqueous ammonia.
Ammonia extract	2.8	0.008	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.4	0.001	N/A			
Aqueous (NH_3)	2.0	0.006	N/A			
Precipitate	0	0.0	N/A			Attributed to protein.
Nonextractable	NR[2]	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	2.6	0.008	N/A			
Precipitate	0.4	0.001	N/A			Attributed to lignin.
Solids	1.2	0.004	N/A			Attributed to cellulose.
270-day PBI (TRR = 0.150 ppm)						
MeOH extract	94.3	0.141	BAS 510 F	73.1	0.109	
			M510F61	21.2	0.032	
Nonextractable	5.7	0.009	N/A			
365-day PBI (TRR = 0.207 ppm)						
MeOH extract	95.2	0.197	BAS 510 F	69.4	0.144	Plus 2 unknowns totaling 10.2% TRR (0.022 ppm); each ≤8.5% TRR (≤0.018 ppm).
			M510F61	15.5	0.032	
Nonextractable	4.8	0.018	N/A			

[1] Not analyzed.
[2] Not reported.

Table 2.2.3. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Radish Roots** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.072 ppm)						
MeOH extract	93.1	0.067	BAS 510 F	89.6	0.064	Plus 1 unknown at 3.5% TRR (0.003 ppm).
Nonextractable	6.9	0.005	N/A[1]			
120-day PBI (TRR = 0.052 ppm)						
MeOH extract	78.7	0.041	BAS 510 F	67.8	0.035	
			M510F61	10.9	0.006	
Nonextractable	21.3	0.011	N/A			Extracted with aqueous ammonia.
Ammonia extract	3.4	0.002	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.9	<0.001	N/A			
Aqueous (NH_3)	2.1	0.001	N/A			
Precipitate	0.3	<0.001	N/A			Attributed to protein.
Nonextractable	NR[2]	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	4.7	0.002	N/A			
Precipitate	1.0	0.001	N/A			Attributed to lignin.
Solids	2.0	0.001	N/A			Attributed to cellulose.
270-day PBI (TRR = 0.098 ppm)						
MeOH extract	92.8	0.091	BAS 510 F	92.8	0.091	
Nonextractable	7.2	0.007	N/A			
365-day PBI (TRR = 0.030 ppm)						
MeOH extract	89.9	0.027	BAS 510 F	78.4	0.024	Plus 1 unknown at 7.5% TRR (0.002 ppm).
			M510F61	4.0	0.001	
Nonextractable	10.1	0.003	N/A			

[1] Not analyzed.
[2] Not reported.

Table 2.2.4. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Forage** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 1.575 ppm)						
MeOH extract	95.5	1.504	BAS 510 F	93.5	1.472	
			M510F61	2.0	0.032	
Nonextractable	4.5	0.071	N/A[1]			Extracted with aqueous ammonia.
Ammonia extract	1.7	0.027	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.6	0.010	N/A			
Aqueous (NH_3)	0.9	0.014	N/A			
Precipitate	1.3	0.021	N/A			Attributed to protein.
Nonextractable	NR[2]	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	0.9	0.015	N/A			Characterized as liquid lignin.
Precipitate	0.6	0.009	N/A			Attributed to lignin.
Solids	0.9	0.014	N/A			Attributed to cellulose.
120-day PBI (TRR = 0.980 ppm)						
MeOH extract	88.5	0.867	BAS 510 F	86.4	0.867	
			M510F61	2.1	0.021	
Nonextractable	11.5	0.113	N/A			Extracted with aqueous ammonia.
Ammonia extract	4.3	0.042	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	2.3	0.022	N/A			
Aqueous (NH_3)	1.6	0.015	N/A			
Precipitate	0.3	0.003	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	2.4	0.023	N/A			
Precipitate	1.5	0.015	N/A			Attributed to lignin.
Solids	1.8	0.018	N/A			Attributed to cellulose.
270-day PBI (TRR = 0.562 ppm)						

Table 2.2.4. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Forage** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
MeOH extract	88.3	0.496	BAS 510 F	62.8	0.352	Plus 2 unknowns totaling 7.5% TRR (0.042 ppm); each at ≤4.2% TRR (≤0.024 ppm).
			M510F61	18.1	0.102	
Nonextractable	11.7	0.066	N/A			Extracted with aqueous ammonia.
Ammonia extract	2.9	0.016	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.6	0.004	N/A			
Aqueous (NH_3)	1.6	0.009	N/A			
Precipitate	0.5	0.003	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	1.3	0.007	N/A			
Precipitate	1.4	0.008	N/A			Attributed to lignin.
Solids	0.3	0.002	N/A			Attributed to cellulose.
365-day PBI (TRR = 0.265 ppm)						
MeOH extract	93.1	0.247	BAS 510 F	75.0	0.199	Plus 1 unknown at 8.3% TRR (0.022 ppm).
			M510F61	9.8	0.026	
Nonextractable	6.9	0.018	N/A			Extracted with aqueous ammonia.
Ammonia extract	3.0	0.008	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.8	0.002	N/A			
Aqueous (NH_3)	1.2	0.003	N/A			
Precipitate	0.3	0.001	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	2.9	0.008	N/A			
Precipitate	1.6	0.004	N/A			Attributed to lignin.
Solids	0.7	0.002	N/A			Attributed to cellulose.

[1] Not analyzed.
[2] Not reported.

Table 2.2.5. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Straw** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 9.826 ppm)						
MeOH extract	85.6	8.414	BAS 510 F	81.3	7.991	
			M510F61	4.3	0.423	
Nonextractable	14.4	1.412	N/A[1]			Extracted with aqueous ammonia.
Ammonia extract	8.2	0.800	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	1.2	0.112	N/A			
Aqueous (NH_3)	5.5	0.538	N/A			
Precipitate	0.2	0.019	N/A			Attributed to protein.
Nonextractable	NR[2]	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	1.8	0.177	N/A			
Precipitate	2.2	0.216	N/A			Attributed to lignin.
Solids	1.2	0.121	N/A			Attributed to cellulose.
120-day PBI (TRR = 3.912 ppm)						
MeOH extract	89.4	3.498	BAS 510 F	84.6	3.311	
			M510F61	4.8	0.187	
Nonextractable	10.6	0.414	N/A			Extracted with aqueous ammonia.
Ammonia extract	5.3	0.206	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.9	0.033	N/A			
Aqueous (NH_3)	3.8	0.149	N/A			
Precipitate	0.4	0.014	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	1.4	0.054	N/A			
Precipitate	1.3	0.052	N/A			Attributed to lignin.
Solids	1.0	0.040	N/A			Attributed to cellulose.
270-day PBI (TRR = 3.226 ppm)						

Table 2.2.5. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Straw** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb **ai/A** to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
MeOH extract	77.1	2.487	BAS 510 F	70.8	2.283	Plus 1 unknown at 5.4% TRR (0.174 ppm).
			M510F61	0.9	0.030	
Nonextractable	22.9	0.739	N/A			Extracted with aqueous ammonia.
Ammonia extract	11.7	0.378	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	3.6	0.117	N/A			
Aqueous (NH_3)	4.7	0.153	N/A			
Precipitate	1.4	0.044	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	2.0	0.066	N/A			
Precipitate	4.6	0.150	N/A			Attributed to lignin.
Solids	3.5	0.114	N/A			Attributed to cellulose.
365-day PBI (TRR = 1.404 ppm)						
MeOH extract	89.3	1.253	BAS 510 F	77.6	1.088	Plus 1 unknown at 10.0% TRR (0.140 ppm).
			M510F61	1.8	0.025	
Nonextractable	10.7	0.151	N/A			Extracted with aqueous ammonia.
Ammonia extract	5.8	0.082	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	1.0	0.014	N/A			
Aqueous (NH_3)	3.1	0.044	N/A			
Precipitate	0.5	0.007	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	1.4	0.019	N/A			
Precipitate	2.5	0.036	N/A			Attributed to lignin.
Solids	1.0	0.014	N/A			Attributed to cellulose.

[1] Not analyzed.
[2] Not reported.

Table 2.2.6. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Grain** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.166 ppm)						
MeOH extract	18.4	0.031	BAS 510 F	16.8	0.028	Plus 1 unknown at 1.6% TRR (0.003 ppm).
Nonextractable	81.6	0.135	N/A[1]			Extracted with aqueous ammonia.
Ammonia extract	16.9	0.028	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	2.1	0.003	N/A			
Aqueous (NH_3)	8.0	0.013	N/A			
Precipitate	6.5	0.011	N/A			Attributed to protein.
Nonextractable	NR[2]	NR	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	5.7	0.009	N/A			
Precipitate	0.6	0.001	N/A			Attributed to starch.
Nonextractable	65.1	0.108	N/A			
120-day PBI (TRR = 0.243 ppm)						
MeOH extract	12.3	0.030	BAS 510 F	9.6	0.023	Plus 1 unknown at 2.7% TRR (0.007 ppm).
Nonextractable	87.8	0.213	N/A			Extracted with aqueous ammonia.
Ammonia extract	13.0	0.032	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	0.3	0.001	N/A			
Aqueous (NH_3)	6.9	0.017	N/A			
Precipitate	4.9	0.012	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	4.6	0.011	N/A			
Precipitate	1.6	0.004	N/A			Attributed to starch.
Nonextractable	71.6	0.174	N/A			
270-day PBI (TRR = 0.023 ppm)						

Table 2.2.6. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Grain** Planted Following Application of **[Diphenyl-U-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
MeOH extract	35.4	0.008	BAS 510 F	35.4	0.008	
Nonextractable	64.6	0.015	N/A			Extracted with aqueous ammonia.
Ammonia extract	22.9	0.005	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	2.3	0.001	N/A			
Aqueous (NH_3)	8.9	0.002	N/A			
Precipitate	9.6	0.002	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	11.3	0.003	N/A			
Precipitate	4.3	0.001	N/A			Attributed to starch.
Nonextractable	30.3	0.007	N/A			
365-day PBI (TRR = 0.048 ppm)						
MeOH extract	25.1	0.012	BAS 510 F	23.6	0.011	Plus 1 unknown at 1.5% TRR (0.001 ppm).
Nonextractable	74.9	0.036	N/A			Extracted with aqueous ammonia.
Ammonia extract	15.2	0.007	N/A			Conc. HCl added, which yielded a precipitate. Aqueous portion partitioned with DCM.
DCM	1.0	<0.001	N/A			
Aqueous (NH_3)	8.1	0.004	N/A			
Precipitate	6.2	0.003	N/A			Attributed to protein.
Nonextractable	NR	NR	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	6.4	0.003	N/A			
Precipitate	2.1	0.001	N/A			Attributed to starch.
Nonextractable	54.2	0.026	N/A			

[1] Not analyzed.
[2] Not reported.

Table 2.2.7. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Lettuce** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.035 ppm)						
MeOH extract	81.2	0.028	BAS 510 F	58.5	0.020	Plus 1 unknown at 22.7% TRR (0.008 ppm).
Nonextractable	18.8	0.007	N/A[1]			Extracted with aqueous ammonia.
Ammonia	1.9	0.001	N/A			
Nonextractable	11.6	0.004	N/A			
120-day PBI (TRR = 0.161 ppm)						
MeOH extract	90.8	0.146	BAS 510 F	90.8	0.146	
Nonextractable	9.2	0.015	N/A			
270-day PBI (TRR = 0.031 ppm)						
MeOH extract	74.5	0.023	BAS 510 F	65.1	0.020	Plus 1 unknown at 9.4% TRR (0.003 ppm).
Nonextractable	25.5	0.008	N/A			
365-day PBI (TRR = 0.022 ppm)						
MeOH extract	76.1	0.017	BAS 510 F	61.6	0.014	Plus 1 unknown at 14.5% TRR (0.003 ppm).
Nonextractable	23.9	0.005	N/A			

[1] Not analyzed.

Table 2.2.8. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Radish Tops** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.343 ppm)						
MeOH extract	92.2	0.317	BAS 510 F	87.6	0.301	
			M510F61	4.6	0.016	
Nonextractable	7.8	0.027	N/A[1]			
120-day PBI (TRR = 0.211 ppm)						
MeOH extract	88.8	0.187	BAS 510 F	81.8	0.172	
			M510F61	7.0	0.015	
Nonextractable	11.2	0.024	N/A			
270-day PBI (TRR = 0.125 ppm)						
MeOH extract	86.1	0.108	BAS 510 F	82.5	0.104	
			M510F61	3.6	0.004	
Nonextractable	13.9	0.017	N/A			

Table 2.2.8. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Radish Tops** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
365-day PBI (TRR = 0.113 ppm)						
MeOH extract	91.1	0.103	BAS 510 F	78.2	0.088	Plus 1 unknown at 1.7% TRR (0.002 ppm).
			M510F61	11.2	0.13	
Nonextractable	8.9	0.010	N/A			

[1] Not analyzed.

Table 2.2.9. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Radish Roots** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.048 ppm)						
MeOH extract	80.7	0.039	BAS 510 F	62.7	0.030	Plus 1 unknown at 18.0% TRR (0.009 ppm).
Nonextractable	19.3	0.009	N/A[1]			Extracted with aqueous ammonia.
Ammonia	1.2	0.001	N/A			
Nonextractable	17.4	0.008	N/A			
120-day PBI (TRR = 0.038 ppm)						
MeOH extract	81.6	0.031	BAS 510 F	60.1	0.023	Plus 1 unknown at 21.5% TRR (0.008 ppm).
Nonextractable	18.4	0.007	N/A			
270-day PBI (TRR = 0.017 ppm)						
MeOH extract	77.1	0.013	BAS 510 F	52.6	0.009	Plus 1 unknown at 24.5% TRR (0.004 ppm).
Nonextractable	22.9	0.004	N/A			
365-day PBI (TRR = 0.066 ppm)						
MeOH extract	91.0	0.060	BAS 510 F	91.0	0.060	
Nonextractable	9.0	0.006	N/A			

[1] Not analyzed.

Table 2.2.10. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Forage** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.690 ppm)						
MeOH extract	93.2	0.643	BAS 510 F	89.8	0.619	
			M510F61	3.4	0.024	
Nonextractable	6.8	0.047	N/A[1]			Extracted with aqueous ammonia.
Ammonia extract	1.5	0.011	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	1.1	0.008	N/A			
Precipitate	<0.1	<0.001	N/A			Attributed to protein.
Nonextractable	5.7	0.039	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	2.7	0.018	N/A			
Precipitate	1.0	0.007	N/A			Attributed to lignin.
Solids	1.2	0.008	N/A			Attributed to cellulose.
120-day PBI (TRR = 0.433 ppm)						
MeOH extract	87.5	0.379	BAS 510 F	87.5	0.379	
Nonextractable	12.5	0.054	N/A			Extracted with aqueous ammonia.
Ammonia extract	1.1	0.005	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	1.5	0.006	N/A			
Precipitate	0.1	<0.001	N/A			Attributed to protein.
Nonextractable	12.9	0.057	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	5.8	0.025	N/A			
Precipitate	1.9	0.008	N/A			Attributed to lignin.
Solids	1.5	0.007	N/A			Attributed to cellulose.
270-day PBI (TRR = 0.230 ppm)						
MeOH extract	97.3	0.224	BAS 510 F	92.8	0.214	Plus 1 unknown at 2.2% TRR (0.005 ppm).
			M510F61	2.3	0.005	
Nonextractable	2.7	0.006	N/A			

Table 2.2.10. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Forage** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
365-day PBI (TRR = 0.255 ppm)						
MeOH extract	83.5	0.213	BAS 510 F	74.7	0.191	Plus 2 unknowns totaling 5.8% TRR (0.015 ppm); each ≤4.0% TRR (≤0.010 ppm).
			M510F61	2.9	0.008	
Nonextractable	16.5	0.042	N/A			

[1] Not analyzed.

Table 2.2.11. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Straw** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 3.609 ppm)						
MeOH extract	90.3	3.258	BAS 510 F	87.5	3.156	
			M510F61	2.8	0.102	
Nonextractable	9.7	0.351	N/A[1]			Extracted with aqueous ammonia.
Ammonia extract	2.5	0.089	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	2.4	0.088	N/A			
Precipitate	0.1	0.004	N/A			Attributed to protein.
Nonextractable	9.2	0.333	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	3.4	0.124	N/A			
Precipitate	4.9	0.176	N/A			Attributed to lignin.
Solids	1.5	0.054	N/A			Attributed to cellulose.
120-day PBI (TRR = 4.008 ppm)						
MeOH extract	67.7	2.715	BAS 510 F	64.8	2.598	
			M510F61	2.9	0.117	
Nonextractable	32.3	1.293	N/A			Extracted with aqueous ammonia.
Ammonia extract	4.2	0.167	N/A			Conc. HCl added, which yielded a precipitate.

Table 2.2.11. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Straw** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
Aqueous (NH_3)	4.1	0.162	N/A			
Precipitate	0.4	0.012	N/A			Attributed to protein.
Nonextractable	19.6	0.783	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	10.1	0.405	N/A			
Precipitate	2.7	0.110	N/A			Attributed to lignin.
Solids	1.9	0.076	N/A			Attributed to cellulose.
270-day PBI (TRR = 1.614 ppm)						
MeOH extract	56.4	0.911	BAS 510 F	50.0	0.808	Plus 1 unknown at 2.0% TRR (0.032 ppm).
			M510F61	4.4	0.071	
Nonextractable	43.6	0.703	N/A			Extracted with aqueous ammonia.
Ammonia extract	5.4	0.087	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	5.2	0.084	N/A			
Precipitate	0.4	0.007	N/A			Attributed to protein.
Nonextractable	20.2	0.326	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.
Aqueous (NaOH)	10.1	0.164	N/A			
Precipitate	2.5	0.041	N/A			Attributed to lignin.
Solids	2.6	0.041	N/A			Attributed to cellulose.
365-day PBI (TRR = 1.925 ppm)						
MeOH extract	77.3	1.488	BAS 510 F	77.3	1.488	
Nonextractable	22.7	0.437	N/A			Extracted with aqueous ammonia.
Ammonia extract	4.8	0.094	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	4.1	0.092	N/A			
Precipitate	0.4	0.008	N/A			Attributed to protein.
Nonextractable	17.7	0.341	N/A			Refluxed with NaOH. Conc. HCl added to the hydrolysate, which yielded a precipitate.

Table 2.2.11. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Straw** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
Aqueous (NaOH)	9.2	0.178	N/A			
Precipitate	3.0	0.057	N/A			Attributed to lignin.
Solids	1.6	0.030	N/A			Attributed to cellulose.

[1] Not analyzed.

Table 2.2.12. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Grain** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
30-day PBI (TRR = 0.147 ppm)						
MeOH extract	11.7	0.017	BAS 510 F	6.1	0.009	Plus 2 unknowns totaling 5.6% TRR (0.009 ppm); each ≤3.8% TRR (≤0.006 ppm).
Nonextractable	88.3	0.130	N/A[1]			Extracted with aqueous ammonia.
Ammonia extract	13.0	0.019	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	11.2	0.016	N/A			
Precipitate	3.1	0.005	N/A			Attributed to protein.
Nonextractable	79.0	0.116	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	6.7	0.010	N/A			
Precipitate	48.4	0.071	N/A			Attributed to starch.
Nonextractable	17.4	0.026	N/A			
120-day PBI (TRR = 0.285 ppm)						
MeOH extract	8.9	0.025	BAS 510 F	5.3	0.015	Plus 1 unknown at 3.6% TRR (0.010 ppm).
Nonextractable	91.1	0.260	N/A			Extracted with aqueous ammonia.
Ammonia extract	17.3	0.049	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	9.1	0.027	N/A			
Precipitate	9.6	0.027	N/A			Attributed to protein.
Nonextractable	55.6	0.159	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	5.5	0.016	N/A			
Precipitate	41.4	0.118	N/A			Attributed to starch.
Nonextractable	22.3	0.064	N/A			

Table 2.2.12. Extraction, Characterization, and Identification of Radioactive Residues in Rotational **Wheat Grain** Planted Following Application of **[Pyridin-3-^{14}C]BAS 510 F** at 1.88 lb ai/A to Bare Soil.

Fraction ID	% TRR	ppm	Residue ID	% TRR	ppm	Comments
270-day PBI (TRR = 0.271 ppm)						
MeOH extract	4.0	0.011	BAS 510 F	1.9	0.005	Plus 1 unknown at 2.1% TRR (0.006 ppm).
Nonextractable	96.0	0.260	N/A			Extracted with aqueous ammonia.
Ammonia extract	13.9	0.038	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	8.8	0.025	N/A			
Precipitate	5.9	0.016	N/A			Attributed to protein.
Nonextractable	58.7	0.159	N/A			Extracted with DMSO/water; ethanol added to the DMSO extract.
Ethanol	6.0	0.016	N/A			
Precipitate	36.2	0.098	N/A			Attributed to starch.
Nonextractable	26.4	0.072	N/A			
365-day PBI (TRR = 0.148 ppm)						
MeOH extract	6.8	0.010	BAS 510 F	4.2	0.006	Plus 1 unknown at 2.6% TRR (0.004 ppm).
Nonextractable	93.2	0.138	N/A			Extracted with aqueous ammonia.
Ammonia extract	12.9	0.019	N/A			Conc. HCl added, which yielded a precipitate.
Aqueous (NH_3)	8.5	0.013	N/A			
Precipitate	5.7	0.008	N/A			Attributed to protein.
Nonextractable	83.0	0.123	N/A			Extracted with DMSO/water; ethanol was added to the DMSO extract.
Ethanol	6.5	0.010	N/A			
Precipitate	41.0	0.061	N/A			Attributed to starch.
Nonextractable	23.3	0.035	N/A			

[1] Not analyzed.

Table 2.3.1. Summary of Characterization and Identification of Radioactive Residues in Lettuce Planted Following Application of Isotopically Labeled BAS 510 F at 1.88 lb ai/A to Bare Soil.

Metabolite or Fraction	30-day PBI		120-day PBI		270-day PBI		365-day PBI	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
Diphenyl label	TRR = 0.050 ppm		TRR = 0.084 ppm		TRR = 0.067 ppm		TRR = 0.028 ppm	
BAS 510 F	93.8	0.047	85.2	0.072	94.1	0.063	55.6	0.016
Unknowns	--	--	4.0	0.003	--	--	7.2	0.002
Aqueous (NH_3)	--	--	--	--	--	--	15.4	0.004
Aqueous (NaOH)	--	--	--	--	--	--	16.3	0.005
DCM fraction	--	--	--	--	--	--	2.2	0.001
Precipitate (lignin)	--	--	--	--	--	--	4.5	0.001
Solids (cellulose)	--	--	--	--	--	--	5.2	0.001
Total Identified (TI)	93.8	0.047	85.2	0.072	94.1	0.063	55.6	0.016
Total Characterized (TC)	--	--	4.0	0.003	--	--	50.8	0.014
Total Extractable (TE)	93.8	0.047	89.2	0.075	94.1	0.063	106.4	0.030
Total Bound (TB)	6.2	0.003	10.8	0.009	5.9	0.004	--	--
% Mass Balance	100		100		100		106.4	
Pyridine label	TRR = 0.035 ppm		TRR = 0.161 ppm		TRR = 0.031 ppm		TRR = 0.022 ppm	
BAS 510 F	58.5	0.020	90.8	0.146	65.1	0.020	61.6	0.014
Unknowns	22.7	0.008	--	--	9.4	0.003	14.5	0.003
Ammonia extract	1.9	0.001	--	--	--	--	--	--
Total Identified (TI)	58.5	0.020	90.8	0.146	65.1	0.020	61.6	0.014
Total Characterized (TC)	24.6	0.009	--	--	9.4	0.003	14.5	0.003
Total Extractable (TE)	83.1	0.029	90.8	0.146	74.5	0.023	76.1	0.017
Total Bound (TB)	11.6	0.004	9.2	0.015	25.5	0.008	23.9	0.005
% Mass Balance	94.7		100		100		100	

TC = Sum of all unidentified, extractable residues
TE = Sum of TI and TC
% Mass Balance = TE %TRR +TB % TRR

Table 2.3.2. Summary of Characterization and Identification of Radioactive Residues in **Radish Tops** Planted Following Application of Isotopically Labeled BAS 510 F at 1.88 lb ai/A to Bare Soil.

Metabolite or Fraction	30-day PBI		120-day PBI		270-day PBI		365-day PBI	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
Diphenyl label	TRR = 0.337 ppm		TRR = 0.294 ppm		TRR = 0.150 ppm		TRR = 0.207 ppm	
BAS 510 F	90.2	0.304	71.2	0.209	73.1	0.109	69.4	0.144
M510F61	5.9	0.020	--	--	21.2	0.032	15.5	0.032
Unknowns	--	--	13.2	0.039	--	--	10.2	0.022
Aqueous (NH_3)	--	--	2.0	0.006	--	--	--	--
Aqueous (NaOH)	--	--	2.6	0.008	--	--	--	--
DCM fraction	--	--	0.4	0.001	--	--	--	--
Precipitate (lignin)	--	--	0.4	0.001	--	--	--	--
Solids (cellulose)	--	--	1.2	0.004	--	--	--	--
Total Identified (TI)	96.1	0.324	71.2	0.209	94.3	0.141	84.9	0.176
Total Characterized (TC)	0	0	19.8	0.059	0	0	10.2	0.022
Total Extractable (TE)	96.1	0.324	91.0	0.268	94.3	0.141	95.1	0.198
Total Bound (TB)	3.9	0.013	--	--	5.7	0.009	4.8	0.018
% Mass Balance	100		91		100		99.9	
Pyridine label	TRR = 0.343 ppm		TRR = 0.211 ppm		TRR = 0.125 ppm		TRR = 0.113 ppm	
BAS 510 F	87.6	0.301	81.8	0.172	82.5	0.104	78.2	0.088
M510F61	4.6	0.016	7.0	0.015	3.6	0.004	11.2	0.013
Unknowns	--	--	--	--	--	--	1.7	0.002
Total Identified (TI)	92.2	0.317	88.8	0.187	86.1	0.108	89.4	0.101
Total Characterized (TC)	--	--	--	--	--	--	1.7	0.002
Total Extractable (TE)	92.2	0.317	88.8	0.187	86.1	0.108	91.1	0.103
Total Bound (TB)	7.8	0.027	11.2	0.024	13.9	0.017	8.9	0.010
% Mass Balance	100		100		100		100	

TC = Sum of all unidentified, extractable residues
TE = Sum of TI and TC
% Mass Balance = TE %TRR +TB % TRR

Table 2.3.3. Summary of Characterization and Identification of Radioactive Residues in **Radish Roots** Planted Following Application of Isotopically Labeled BAS 510 F at 1.88 lb ai/A to Bare Soil.

Metabolite or Fraction	30-day PBI		120-day PBI		270-day PBI		365-day PBI	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
Diphenyl label	TRR = 0.072 ppm		TRR = 0.052 ppm		TRR = 0.098 ppm		TRR = 0.030 ppm	
BAS 510 F	89.6	0.064	67.8	0.035	92.8	0.091	78.4	0.024
M510F61	--	--	10.9	0.006	--	--	4.0	0.001
Unknowns	3.5	0.003	--	--	--	--	7.5	0.002
Aqueous (NaOH)	--	--	4.7	0.002	--	--	--	--
Aqueous (NH_3)	--	--	2.1	0.001	--	--	--	--
DCM fraction	--	--	0.9	<0.001	--	--	--	--
Precipitate (lignin)	--	--	1.0	0.001	--	--	--	--
Precipitate (protein)	--	--	0.3	<0.001	--	--	--	--
Solids (cellulose)	--	--	2.0	0.001	--	--	--	--
Total Identified (TI)	89.6	0.064	78.7	0.041	92.8	0.091	82.4	0.025
Total Characterized (TC)	3.5	0.003	11.0	0.006	--	--	7.5	0.002
Total Extractable (TE)	93.1	0.067	89.7	0.047	92.8	0.091	89.9	0.027
Total Bound (TB)	6.9	0.005	--	--	7.2	0.007	10.1	0.003
% Mass Balance	100		89.7		100		100	
Pyridine label	TRR = 0.048 ppm		TRR = 0.038 ppm		TRR = 0.017 ppm		TRR = 0.066 ppm	
BAS 510 F	62.7	0.030	60.1	0.023	52.6	0.009	91.0	0.060
M510F61	--	--	--	--	--	--	--	--
Unknowns	18.0	0.009	21.5	0.008	24.5	0.004	--	--
Ammonia extract	1.2	0.001	--	--	--	--	--	--
Total Identified (TI)	62.7	0.03	60.1	0.023	52.6	0.009	91.0	0.06
Total Characterized (TC)	19.2	0.01	21.5	0.008	24.5	0.004	--	--
Total Extractable (TE)	81.9	0.04	81.6	0.031	77.1	0.013	91.0	0.06
Total Bound (TB)	17.4	0.008	18.4	0.007	22.9	0.004	9.0	0.006
% Mass Balance	99.3		100		100		100	

TC = Sum of all unidentified, extractable residues
TE = Sum of TI and TC
% Mass Balance = TE %TRR +TB % TRR

Table 2.3.4. Summary of Characterization and Identification of Radioactive Residues in **Wheat Forage** Planted Following Application of Isotopically Labeled BAS 510 F at 1.88 lb ai/A to Bare Soil.

Metabolite or Fraction	30-day PBI		120-day PBI		270-day PBI		365-day PBI	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
Diphenyl label	TRR = 1.575 ppm		TRR = 0.980 ppm		TRR = 0.562 ppm		TRR = 0.265 ppm	
BAS 510 F	93.5	1.472	86.4	0.846	62.8	0.352	75.0	0.199
M510F61	2.0	0.032	2.1	0.021	18.1	0.102	9.8	0.026
Unknowns	--	--	--	--	7.5	0.042	8.3	0.022
Aqueous (NaOH)	0.9	0.015	2.4	0.023	1.3	0.007	2.9	0.008
Aqueous (NH_3)	0.9	0.014	1.6	0.015	1.6	0.009	1.2	0.003
DCM fraction	0.6	0.010	2.3	0.022	0.6	0.004	0.8	0.002
Precipitate (lignin)	0.6	0.009	1.5	0.015	1.4	0.008	1.6	0.004
Precipitate (protein)	1.3	0.021	0.3	0.003	0.5	0.003	0.3	0.001
Solids (cellulose)	0.9	0.014	1.8	0.018	0.3	0.002	0.7	0.002
Total Identified (TI)	95.5	1.504	88.5	0.867	80.9	0.454	84.8	0.225
Total Characterized (TC)	5.2	0.083	9.9	0.096	13.2	0.075	15.8	0.042
Total Extractable (TE)	100.7	1.587	98.4	0.963	94.1	0.529	100.6	0.267
Total Bound (TB)	--	--	--	--	--	--	--	--
% Mass Balance	100.7		98.4		94.1		100.6	
Pyridine label	TRR = 0.690 ppm		TRR = 0.433 ppm		TRR = 0.230 ppm		TRR = 0.255 ppm	
BAS 510 F	89.8	0.619	87.5	0.379	92.8	0.214	74.7	0.191
M510F61	3.4	0.024	--	--	2.3	0.005	2.9	0.008
Unknowns	--	--	--	--	2.2	0.005	5.8	0.015
Aqueous (NaOH)	2.7	0.018	5.8	0.025	--	--	--	--
Aqueous (NH_3)	1.1	0.008	1.5	0.006	--	--	--	--
Precipitate (lignin)	1.0	0.007	1.9	0.008	--	--	--	--
Precipitate (protein)	<0.1	<0.001	0.1	<0.001	--	--	--	--
Solids (cellulose)	1.2	0.008	1.5	0.007	--	--	--	--
Total Identified (TI)	93.2	0.643	87.5	0.379	95.1	0.219	77.6	0.199
Total Characterized (TC)	6.1	0.042	10.8	0.047	2.2	0.01	5.8	0.015
Total Extractable (TE)	99.3	0.685	98.3	0.426	97.3	0.224	83.4	0.214
Total Bound (TB)	--	--	--	--	2.7	0.006	16.5	0.042
% Mass Balance	99.3		98.3		100		99.9	

TC = Sum of all unidentified, extractable residues
TE = Sum of TI and TC
% Mass Balance = TE %TRR +TB % TRR

Table 2.3.5. Summary of Characterization and Identification of Radioactive Residues in **Wheat Straw** Planted Following Application of Isotopically Labeled BAS 510 F at 1.88 lb ai/A to Bare Soil.

Metabolite or Fraction	30-day PBI		120-day PBI		270-day PBI		365-day PBI	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
Diphenyl label	TRR = 9.826 ppm		TRR = 3.912 ppm		TRR = 3.226 ppm		TRR = 1.404 ppm	
BAS 510 F	81.3	7.991	84.6	3.311	70.8	2.283	77.6	1.088
M510F61	4.3	0.423	4.8	0.187	0.9	0.030	1.8	0.025
Unknowns	--	--	--	--	5.4	0.174	10.0	0.140
Aqueous (NaOH)	1.8	0.177	1.4	0.054	2.0	0.066	1.4	0.019
Aqueous (NH_3)	5.5	0.538	3.8	0.149	4.7	0.153	3.1	0.044
DCM fraction	1.2	0.112	0.9	0.033	3.6	0.117	1.0	0.014
Precipitate (lignin)	2.2	0.216	1.3	0.052	4.6	0.150	2.5	0.036
Precipitate (protein)	0.2	0.190	0.4	0.014	1.4	0.044	0.5	0.007
Solids (cellulose)	1.2	0.121	1.0	0.040	3.5	0.114	1.0	0.014
Total Identified (TI)	85.6	8.414	89.4	3.498	71.7	2.313	79.4	1.113
Total Characterized (TC)	12.1	1.354	8.8	0.342	25.2	0.818	19.5	0.274
Total Extractable (TE)	97.7	9.768	98.2	3.84	96.9	3.131	98.9	1.387
Total Bound (TB)	--	--	--	--	--	--	--	--
% Mass Balance	97.7		98.2		96.9		98.9	
Pyridine label	TRR = 3.609 ppm		TRR = 4.008 ppm		TRR = 1.614 ppm		TRR = 1.925 ppm	
BAS 510 F	87.5	3.156	64.8	2.598	50.0	0.808	77.3	1.488
M510F61	2.8	0.102	2.9	0.117	4.4	0.071	--	--
Unknowns	--	--	--	--	2.0	0.032	--	--
Aqueous (NaOH)	3.4	0.124	10.1	0.405	10.1	0.164	9.2	0.178
Aqueous (NH_3)	2.4	0.088	4.1	0.162	5.2	0.084	4.1	0.092
Precipitate (lignin)	4.9	0.176	2.7	0.110	2.5	0.041	3.0	0.057
Precipitate (protein)	0.1	0.004	0.4	0.012	0.4	0.007	0.4	0.008
Solids (cellulose)	1.5	0.054	1.9	0.076	2.6	0.041	1.6	0.030
Total Identified (TI)	90.3	3.258	67.7	2.715	54.4	0.879	77.3	1.488
Total Characterized (TC)	12.3	0.446	19.2	0.765	22.8	0.369	18.3	0.365
Total Extractable (TE)	102.6	3.704	86.9	3.480	77.2	1.248	95.6	1.853
Total Bound (TB)	--	--	--	--	--	--	--	--
% Mass Balance	102.6		86.9		77.2		95.6	

TC = Sum of all unidentified, extractable residues
TE = Sum of TI and TC
% Mass Balance = TE %TRR +TB % TRR

Table 2.3.6. Summary of Characterization and Identification of Radioactive Residues in **Wheat Grain** Planted Following Application of Isotopically Labeled BAS 510 F at 1.88 lb ai/A to Bare Soil.

Metabolite or Fraction	30-day PBI		120-day PBI		270-day PBI		365-day PBI	
	%TRR	ppm	%TRR	ppm	%TRR	ppm	%TRR	ppm
Diphenyl label	TRR = 0.166 ppm		TRR = 0.243 ppm		TRR = 0.023 ppm		TRR = 0.048 ppm	
BAS 510 F	16.8	0.028	9.6	0.023	35.4	0.008	23.6	0.011
Unknowns	1.6	0.003	2.7	0.007	--	--	1.5	0.001
Aqueous (NH_3)	8.0	0.013	6.9	0.017	8.9	0.002	8.1	0.004
DCM fraction	2.1	0.003	0.3	0.001	2.3	0.001	1.0	<0.001
Ethanol fraction	5.7	0.009	4.6	0.011	11.3	0.003	6.4	0.003
Precipitate (protein)	6.5	0.011	4.9	0.012	9.6	0.002	6.2	0.003
Precipitate (starch)	0.6	0.001	1.6	0.004	4.3	0.001	2.1	0.001
Total Identified (TI)	16.8	0.028	9.6	0.023	35.4	0.008	23.6	0.011
Total Characterized (TC)	24.5	0.040	21.0	0.052	36.4	0.009	25.3	0.013
Total Extractable (TE)	41.3	0.068	30.6	0.075	71.8	0.017	48.9	0.024
Total Bound (TB)	65.1	0.108	71.6	0.174	30.3	0.007	54.2	0.026
% Mass Balance	106.4		102.2		102.1		103.1	
Pyridine label	TRR = 0.147 ppm		TRR = 0.285 ppm		TRR = 0.271 ppm		TRR = 0.148 ppm	
BAS 510 F	6.1	0.009	5.3	0.015	1.9	0.005	4.2	0.006
Unknowns	5.6	0.009	3.6	0.010	2.1	0.006	2.6	0.004
Aqueous (NH_3)	11.2	0.016	9.1	0.027	8.8	0.025	8.5	0.013
Ethanol fraction	6.7	0.010	5.5	0.016	6.0	0.016	6.5	0.010
Precipitate (protein)	3.1	0.005	9.6	0.027	5.9	0.016	5.7	0.008
Precipitate (starch)	48.4	0.071	41.4	0.118	36.2	0.098	41.0	0.061
Total Identified (TI)	6.1	0.01	5.3	0.015	1.9	0.01	4.2	0.006
Total Characterized (TC)	75.0	0.111	69.2	0.198	59.0	0.161	64.3	0.096
Total Extractable (TE)	81.1	0.120	74.5	0.213	60.9	0.166	68.5	0.102
Total Bound (TB)	17.4	0.026	22.3	0.064	26.4	0.072	23.3	0.035
% Mass Balance	98.5		96.8		87.3		91.8	

TC = Sum of all unidentified, extractable residues
TE = Sum of TI and TC
% Mass Balance = TE %TRR +TB % TRR.

Figure 1. Proposed Metabolic Fate of BAS 510 F in Rotational Crops.

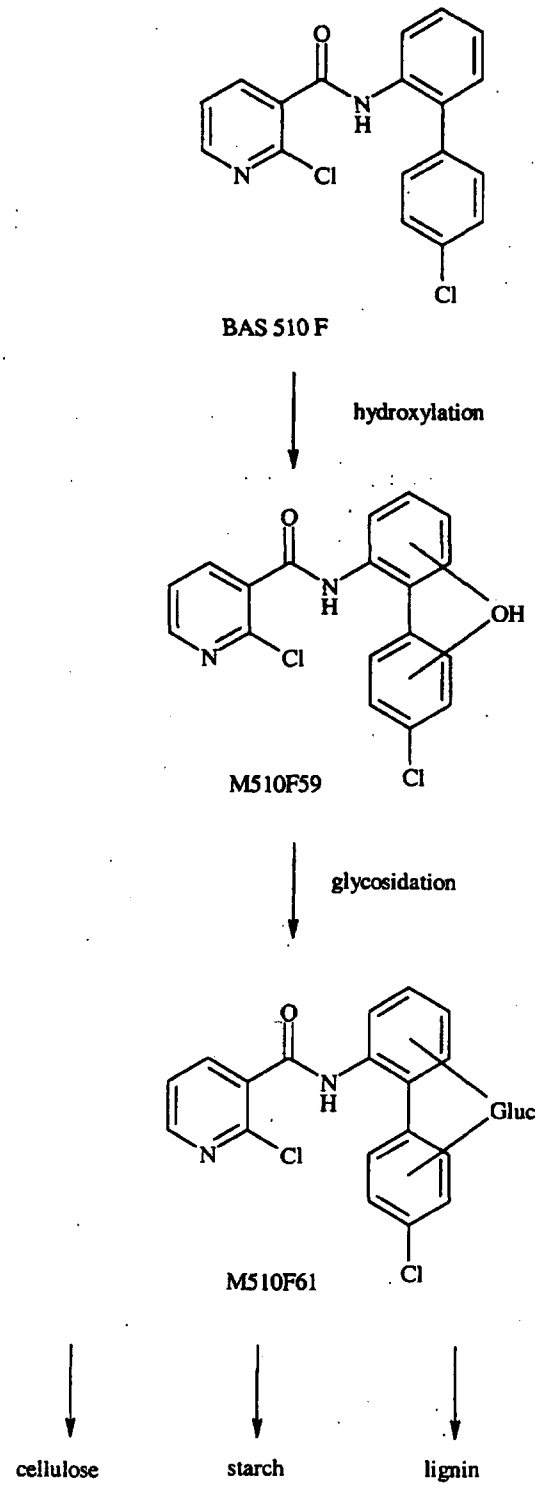
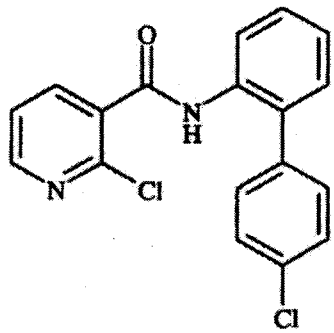
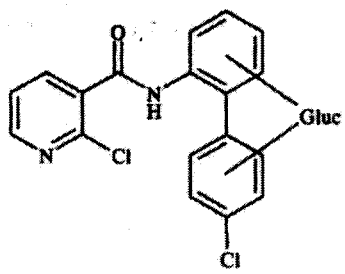


Table 2.4. Metabolites of BAS 510 F in Rotational Crops

Metabolite Identifier	Chemical Name	Structure	Comments
BAS 510 F (Parent compound)	3-Pyridinecarboxamide, 2-chloro-N-(4'chloro[1,1'-biphenyl]-2-yl)-		Identified in all matrices at all PBIs: lettuce, radish tops and roots, and wheat forage, straw, and grain.
M510F61	glucuronide of M510F59		Identified in radish tops (all PBIs) and roots (120- and 365-day PBI only), and wheat forage and straw (all PBIs).

3. Discussion

3.1. Methods

Radiolabeled [^{14}C]BAS 510 F, labeled at the 3-position of the pyridine ring or uniformly labeled on the phenyl rings, was applied to bare soil at 1.88 lb ai/A (2100 g ai/ha). Rotational crops, lettuce, radish, and wheat, were planted in treated soil 30, 120, 270, and 365 days after treatment of the soil. Immature wheat forage, and mature lettuce, radish root and top, and wheat straw and grain were harvested from each of the plantback intervals (PBIs). TRR were determined by combustion/LSC. The petitioner used the calculated TRR values (extractable radioactivity plus nonextractable residues) for reporting results. Material balances, based on combustion of the samples, were 77.2-127.3% and 51.7-112.6% for the diphenyl and pyridine labels, respectively; exceptionally low or high material balances were observed for certain matrices: 7.6% for diphenyl-label grain (120-day PBI), 230% for pyridine-label lettuce (120-day PBI), 178.5% for pyridine-label wheat straw (120-day PBI), 40.0% for pyridine-label wheat forage (270-day PBI) and 564.6% for pyridine-label wheat grain (270-day PBI). The petitioner did not provide any explanations for these values.

For both labels, approximately 62-97% of the TRR were extracted using methanol, and aqueous ammonia in some cases, from all rotational crop matrices except wheat grain; only 18-58% of the TRR were extracted from wheat grain using methanol and aqueous ammonia. Nonextractable residues were further fractionated using aqueous ammonia hydrolysis, sodium hydroxide hydrolysis, and/or DMSO extraction, to yield fractions that the petitioner attributed to protein, cellulose, lignin, and/or starch. The methanol extracts were analyzed by HPLC, and the results were confirmed by a second HPLC system. Identifications of the parent and the metabolite M510F61 were confirmed by LC/MS/MS in 30-day PBI wheat straw samples. In addition, the petitioner analyzed soil samples taken at each of the plantback intervals for TRR by combustion/LSC and for metabolite characterization using HPLC.

The petitioner has made some assumptions about the incorporation of radioactivity into protein, cellulose, lignin, and starch based on fractionation of radioactivity. Although the fractionation pattern indicates that radioactivity may have been incorporated into biomolecules, the petitioner has provided no direct evidence of this incorporation.

3.2. Results

Following an application of [^{14}C]BAS 510 F, labeled at the 3-position of the pyridine ring or uniformly labeled on the phenyl rings, at 1.88 lb ai/A to bare soil, total radioactive residues (TRR) accumulated >0.01 ppm in all commodities of lettuce, radish, and wheat planted 30, 120, 270, and 365 days after treatment. Calculated TRR in diphenyl- and pyridine-label samples ranged 0.028-0.084 and 0.022-0.161 ppm in lettuce, 0.150-0.337 and 0.113-0.343 ppm in radish tops, 0.030-0.098 and 0.017-0.066 ppm in radish roots, 0.265-1.575 and 0.230-0.690 ppm in wheat forage, 1.404-9.826 and 1.614-4.008 ppm in wheat straw, and 0.023-0.243 and 0.147-0.285 ppm in wheat grain, respectively. Radioactivity was lowest in lettuce and radish roots and highest in wheat forage and straw; TRR generally decreased at longer PBIs.

A total of 52.6-96.1% of the TRR was identified in **lettuce, radish roots and tops, and wheat forage and straw** from the 30-, 120-, 270-, and 365-day PBIs. The parent, BAS 510 F was the major residue identified in these commodities, accounting for 50.0-96.1% TRR (0.009-3.156 ppm). The glucoside metabolite M510F61 was identified in radish tops (all PBIs), radish roots (120- and 365-day PBIs only), wheat forage (all PBIs), and wheat straw (all PBIs); M510F61 accounted for 0.9-21.2% TRR (0.001-0.423 ppm). Unknown metabolites accounted for up to 22.7% TRR; however, individual peaks that were present at >10% TRR were each <0.04 ppm. Bound residues in certain samples of lettuce, radish tops and roots, and wheat forage and straw were tentatively demonstrated to be due to the incorporation of radioactivity into protein, lignin, and cellulose at levels totaling up to 9.7% TRR. The remaining nonextractable residues were <0.05 ppm and were not further characterized.

A total of 1.9-35.4% of the TRR was identified in **wheat grain** from the 30-, 120-, 270-, and 365-day PBIs. The parent, BAS 510 F, was the only compound identified in wheat grain at all PBIs, accounting for 9.6-35.4% TRR (0.008-0.028 ppm) in diphenyl-label grain and 1.9-6.1% TRR

(0.005-0.015 ppm) in pyridine-label grain. Unknown metabolites accounted for≤5.6% TRR (≤0.010 ppm). Bound radioactivity in wheat grain was tentatively demonstrated to be due to incorporation of radioactivity into starch, at 0.6-4.3% TRR (0.001-0.004 ppm) for the diphenyl label samples and 36.2-48.4% TRR (0.061-0.118 ppm) for the pyridine label samples, and protein, at 4.9-9.6% TRR (0.002-0.012 ppm) for the diphenyl label samples and 3.1-9.6% TRR (0.005-0.027 ppm) for the pyridine label samples. Nonextractable residues were 30.3-71.6% TRR (0.007-0.174 ppm) in diphenyl-label grain samples and 17.4-26.4% TRR (0.026-0.072 ppm) in pyridine-label grain samples; these residues were not further characterized.

The TRR in soil decreased over the duration of the study, with only the parent identified. Therefore, the petitioner proposed that BAS 510 F is not metabolized in the soil, and that the metabolite M510F61 identified in several crop matrices was a result of metabolism of BAS 510 F in the plants.

4. Deficiencies

The study is acceptable to fulfill the data requirement for a confined rotational crop study.

5. References

None.