Data Evaluation Report on the anaerobic biotransformation of BAS 510 F in water/sediment system

PMRA Submission Number {.....} EPA MRID Number 45405213

Data Requirement: PMRA Data Code:
EPA DP Barcode:
OECD Data Point:
EPA Guideline: 162-3

Test material:
Common name: BAS 510 F
Chemical name
IUPAC: 2-Chloro-*N*-(4-chlorobiphenyl-2-yl)-nicotinamide.
CAS name: 2-Chloro-*N*-(4-chloro[1,1-biphenyl]-2-yl)-3-pyridinecarboxamide.
CAS No: 188425-85-6.
Synonyms: 2-Chloro-*N*-(4'-chlorobiphenyl-2-yl)-nicotinamide.
Nicobifen.
~~BAS 516 02 F.~~
Smiles string:

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CITATION: Paulick, R.C.and M. Baucom. 2001. Anaerobic aquatic metabolism of ^{14}C-BAS 510 F. Unpublished study performed by BASF Corporation, BASF Agro Research, Research Triangle Park, NC and AgVise Laboratories, Northwood, ND. Submitted by BASF Corporation, Research Triangle Park, NC. Laboratory Project Identification: BASF Protocol No. 42385; BASF Registration Document No. 2001/5001021. Study initiated October 12, 1999 and completed March 20, 2001 (p. 11).

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EXECUTIVE SUMMARY

The anaerobic biotransformation of [pyridine-3-^{14}C]- and [diphenyl-U-^{14}C]- labeled BAS 510 F was studied in a pond water/sediment system (water pH 8.4; clay loam soil, pH 7.3, organic matter 4.9%) from Gardner, North Dakota for 361 days in the dark under a nitrogen atmosphere at 20 ± 1°C. [Pyridine-3-^{14}C]- and [U-diphenyl-^{14}C]-labeled BAS 510 F were applied at the rate of 0.27 and 0.26 mg a.i./L, respectively. The sediment/water ratio used was 50 g wet sediment/100 mL pond water. This study was conducted in accordance with the USEPA Subdivision N Guidelines §162-3 and PMRA Guideline T-1-255, DACO 8.2.3.5.6, and in compliance with the USEPA GLP Standards (40 CFR Part 160, 1989). The test system consisted of dishes of treated soil that were contained in glass metabolism towers (not described). To trap volatiles, the air in the towers was periodically evacuated through NaOH trapping solutions and a biological oxidizer. Duplicate samples of each soil were collected after 0, 7, 35, 63, 88, 179, 284 and 361 days of incubation. Single samples were analyzed at all intervals except 284 and 361 days, when duplicate samples were analyzed. The water and sediment phases were separated by centrifugation. The sediment samples were sequentially extracted 2 to 3 times with methanol and 1 to 3 times with methanol and water (1:1, v:v). The water, sediment extracts and extracted sediment were analyzed for total radioactivity using LSC. The water and sediment extracts were analyzed for [^{14}C]BAS 510 F and its transformation products using HPLC; peaks were identified by comparison to reference standards. The identification of BAS 510 F was confirmed using TLC.

The test conditions outlined in the study protocol were maintained throughout the study; redox potentials ranged from -158.2 to -305.2 mV during the study. Mass balances averaged 101.8 ± 2.29% of the applied radioactivity in the pyridine label study and 99.6 ± 3.54% of the applied in the diphenyl label study.

The behavior of [pyridine-3-^{14}C]- and [diphenyl-U-^{14}C]-labeled BAS 510 F in the sediment:water systems was similar, so results are combined in this summary. Extractable [^{14}C]BAS 510 F residues in sediment increased from 2.7-4.9% of the applied at day 0 to a maximum 59.1-61.9% at 35 days posttreatment, then slowly declined to 44.2-43.8% at 361 days. Nonextractable residues increased from 0.6-0.8% of the applied at day 0 to 35.–39.0% at 35 days and 55.0-55.1% at 361 days. At 361 days posttreatment, 4.1-5.6% of the applied was associated with the fulvic acids, 1.2-1.7% with humic acid and 36.9-51.9% with humins. At 361 days, volatilized [^{14}C]residues totaled 0.2% of the applied; [^{14}C]volatiles were not identified.

In the total sediment:water system, [^{14}C]BAS 510 F decreased from 92.2-94.5% of the applied at day 0 to 49.7-51.8% at 179 days posttreatment and 46.2-47.8% at 361 days. The calculated half-life (first-order linear) for the combined data was 385 days; the calculated DT50 (nonlinear) for the entire system was 302-342 days. However, degradation was not observed, and the reported half-life is not a true metabolic half-life. In the water phase, [^{14}C]BAS 510 F decreased from 92.2-94.5% of the applied at day 0 to 50.9-53.1% at 7 days posttreatment, 9.6-14.1% at 35 days and 3.6-4.6% at 361 days. In the sediment phase, [^{14}C]BAS 510 F increased to a maximum 59.1-

61.9% at 35 days posttreatment, then slowly decreased to 41.6-44.2% at 361 days. The water:sediment distribution ratio of [^{14}C]BAS 510 F was 52:36 at 7 days, 12:60 at 35 days, and 4:43 at 361 days.

No transformation products were identified. [^{14}C]Residues other than [^{14}C]BAS 510 F could not be distinguished from background in water or sediment from the [diphenyl-U-^{14}C]BAS 510 F treatment, or in water or sediment from the [pyridine-3-^{14}C]-BAS 510 F treatment except at 361 days, when "others" in the sediment totaled 2.1% of the applied.

A biotransformation pathway for BAS 510 F under anaerobic aquatic conditions was not proposed by the registrant. Based on the study results, BAS 510 F is converted primarily to soil bound residues or remains present as the parent compound.

Results Synopsis:

Test system used: Clay loam sediment flooded with pond water
Half-life in entire system: *ca.* 385 days (r^2 = 0.82) (based on combined data from both labels; affected by a high level of soil bound residues; not a true metabolic half-life since degradation did not occur)
Major transformation product: No major transformation products were identified.
Minor transformation products: No minor transformation products were identified.

[Pyridine-3-14C]
Half-life in water: Initial - *ca.* 13 days (r^2 = 0.988; not a true metabolic half-life since degradation did not occur) and secondary - *ca.* 224 days (r^2=0.75)
Half-life in sediment: *ca.* 693 days (r^2 = 0.95; not a true metabolic half-life since degradation did not occur)
Half-life in entire system: *ca.* 385 days (r^2=0.81; not a true metabolic half-life since degradation did not occur)
DT50 in water: 4.1 days (nonlinear)
DT 75 in water: 15.7 days (nonlinear)
DT50 in entire system: 301 days (nonlinear)
DT75 in entire system: >361 days (nonlinear)

[Diphenyl-U-14C]
Half-life in water: Initial - *ca.* 11 days (r^2 = 1.0; not a true metabolic half-life since degradation did not occur) and secondary - *ca.* 217 days (r^2=0.93)
Half-life in sediment: *ca.* 770 days (r^2 = 0.74; not a true metabolic half-life since degradation did not occur)
Half-life in the entire system: *ca.* 408 days (r^2 = 0.77; not a true metabolic half-life since degradation did not occur)
DT50 in water: 3.6 days (nonlinear)
DT 75 in water: 13.9 days (nonlinear)

DT50 in entire system: 342 days (nonlinear)
DT75 in entire system: >361 days (nonlinear)

Study Acceptability: This study is classified as acceptable and satisfies the guideline data requirement for an anaerobic biotransformation study in a water/sediment system (162-3).

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: This study was conducted in accordance with the USEPA Subdivision N Guideline §162-3 and PMRA Guideline T-1-255, DACO 8.2.3.5.6. No significant deviations from Subdivision N guidelines were noted.

COMPLIANCE: This study was conducted in compliance with USEPA GLP Standards (40 CFR Part 160, 1989; p. 3). Signed and dated Data Confidentiality, GLP, Quality Assurance and certification of authenticity statements were provided (pp. 2-5).

A. MATERIALS:

1. Test Material: [Diphenyl-U-^{14}C]- and [pyridine-3-^{14}C]-labeled BAS 510 F

Chemical Structure:

Description: Solid (p. 15).

Purity:
[Diphenyl-U-^{14}C] label: Radiochemical purity: >98% (prior to dosing, p. 13)
Analytical purity: >96%
Batch No. 641-2017
Specific activity: 376,000 dpm/µg
Location of the label: Uniformly on the biphenyl rings

[Pyridine-3-^{14}C] label: Radiochemical purity: >98% (prior to dosing, p. 13)
Chemical purity: > 97%
Batch No. 640-2037
Specific activity: 310,000 dpm/µg
Location of the label: Carbon 3 of the pyridine ring.

Storage conditions of test chemicals: Storage conditions were not reported.

Physico-chemical properties of ^{14}C-BAS 510 F.

Parameter	Values	Comments
Molecular weight	343.2 g/mol	
Water solubility	4.64 mg/L at 20°C	
Vapour pressure/volatility	Not reported.	
UV absorption	Not reported.	
pK_a	Not reported.	
K_{ow}/log K_{ow}	Not reported.	
Stability of compound at room temperature	Not reported.	

Data obtained from pp. 13 and 23 of study report.

2. Water-sediment collection, storage and properties

Table 1: Description of water collection and storage.

Description	Details
Geographic location	Sediment and water were obtained from a pond located approximately 10 miles from Gardner, North Dakota
Pesticide use history at the collection site	Not reported.
Collection procedures for: Water: Sediment:	 Not reported. Not reported.
Sampling depth for: Water: Sediment:	 Not reported. Not reported.
Storage conditions	Refrigerated until use.
Storage length	16 days.
Preparation of water and sediment samples	Sediment was screened though a 4.74-mm sieve, but was not allowed to dry.

Data obtained from pp. 14-15 of study report.

Table 2: Properties of the water.

Property		Details	
Temperature (°C)		Not reported.	
pH		8.4	
Redox potential (mv)		Initial (0 days)	Final (361 days)
	Diphenyl	-175.1	-274.0 ± 2.97
	Pyridine	-272.0	-281.5 ± 3.18
Oxygen concentration (mg/L)		Initial (0 days)	Final (361 days)
	Diphenyl	0.09	0.14 ± 0.035
	Pyridine	0.03	0.18 ± 0.09
Dissolved organic carbon (%)		Not reported.	
Total dissolved salts (ppm)		432	
Sodium absorption ratio (SAR)		0.52 (p. 57)	
Turbidity (NTU)		20.4	
Hardness ($CaCO_3$/L)		321	
Electrical conductivity (mmhos/cm)		0.78	
Microbial population (CFU/mL of water)*	Actinomycetes	758	
	Fungi	1	
	Bacteria	6670	

Data obtained from pp. 24 and 30 of the study report unless noted.
* Microbial data determined during analysis of physico-chemical properties of water and soil.

Table 3: Properties of the sediment.

Property	Details	
Textural classification	Clay loam	
% sand	24	
% silt	38	
% clay	38	
pH	7.3	
Organic carbon (%)	Not reported.	
Organic matter (%)	4.9	
CEC (meq/100 g)	31.3	
Redox potential (mV)	Initial	Final
	Not reported.	Not reported.
Moisture at 1/3 bar (%)	42.5	
Moisture at 15 bar (%)	28.4	
Bulk density (g/cm^3)	0.98 (disturbed)	
Biomass (mg microbial C/100 g)	Initial	Final
	127.8 µg/g dry wt.	Not reported.

Data obtained from pp. 24 and 55 of study report.

B. EXPERIMENTAL DESIGN:

1. Preliminary experiments: No preliminary experiments were conducted

2. Experimental conditions:

Table 3: Study design.

Parameter	
Duration of the test	361 days after treatment. Flooded sediment was incubated for 52 days prior to treatment to ensure attainment of anaerobic conditions.
Water: Filtered/unfiltered water Type and size of filter used, if any	 Not reported. Not reported.
Amount of sediment and water/ treatment	50 g of wet sediment and 100 mL of water per treatment

Parameter		
Water/sediment ratio		Approximately 2:1 (v:w)
Application rates	Diphenyl	0.26 mg/L (Table 5, p. 27)
	Pyridine	0.27 mg/L (Table 5, p. 27)
Control conditions, if used		No controls were used.
No. of replications	Control, if used	No controls were used.
	Treatments	Duplicate samples were collected at each sampling interval. Only one of the two samples was analyzed at most intervals; the second sample was stored frozen. Both samples collected on days 284 and 361were analyzed.
Test apparatus	Type/material/volume	Glass centrifuge tubes (250 mL) sealed with a Teflon-lined silicon septum and open-topped plastic screw cap (p. 40).
	Details of traps for CO_2 and organic volatile, if any	The headspace of the centrifuge tubes were purged with nitrogen at each sampling interval. The headspace gases were bubbled through two tubes of 2 N NaOH solutions, then through a Harvey Biological Oxidizer for a complete 4-minute cycle. The metabolism apparatus is illustrated on p. 40.
If no traps were used, is the system closed?		Volatile traps were used.
Co-solvent.	Identity	Acetonitrile
	Final concentration	Approximately 0.02% (compared to total system volume)
Test material application	Volume of the test solution used/treatment	25 µL of 1µg a.i./µL test solution per sediment:water sample
	Application method	Test solution was added to the water phase of each test system.
Any indication of the test material adsorbing to the walls of the test apparatus?		Not determined. Mass balances were acceptable and did not indicate material losses due to adsorption to the test vessels.
Microbial biomass/microbial population of the control		Initial / Final
Water:		No controls were used.
Sediment		
Microbial biomass/microbial population of the treated		Initial / Final
Water:		Not determined. / Not determined.
Sediment		Not determined. / Not determined.

Parameter		
Experimental conditions:	Temperature (°C)	20 ± 1°C after dosing
	Continuous darkness (Yes/No)	Yes
Other details, if any		Flooded soils were amended with 0.5% glucose and flushed with nitrogen, then incubated at 25°C for 52 days prior to treatment to ensure attainment of anaerobic conditions.

Data obtained from p. 15 of the study report.

3. Anaerobic conditions: The flooded sediment was amended with 0.5% glucose and flushed with N_2, then incubated at 25°C for 52 days prior to treatment (p. 15). In the two samples analyzed on day 0, the redox potential in the water was -175.1 and -272.0 mV and the dissolved oxygen content was 0.03 and 0.09 mg/L (Table 7, p. 30). After treatment, the samples were flushed with N_2 prior to incubation.

4. Supplementary experiments: In order to facilitate identification of degradates, four additional samples were treated with [pyridine-3-^{14}C]BAS 510 F at 250 µg/sample (p. 15). The soils were incubated under the conditions described for the definitive study.

5. Sampling:

Table 4: Sampling details.

Criteria	Details
Sampling intervals	0, 7, 35, 63, 88, 179, 284 and 361 days
Sampling method	Two dishes of each soil were collected at each sampling interval. At all sampling intervals except the final two, one sample was analyzed and the other sample frozen.
Method of collection of CO_2 and organic volatile compounds	Headspace gases were collected from each sample at each sampling interval.
Sampling intervals/times for: Sterility check, if sterile controls are used: Redox potential:	 Sterile controls were not used. Redox potential, dissolved O_2, and pH were measured in the samples collected at each sampling interval.
Sample storage before analysis	Not reported.. Primary analysis was conducted within 30 days of sampling (p. 21).
Other observations, if any	None.

Data obtained from pp. 15 and 17 from the study report.

C. ANALYTICAL METHODS:

Separation of the sediment and water: The sediment:water systems were centrifuged (15 minutes at 2000 rpm), then the water layer was decanted into a graduated cylinder. Aliquots of the water were analyzed by LSC. (p. 17)

Extraction/clean-up/concentration methods for water and sediment samples: The water was again centrifuged (14,000 rpm), then analyzed directly by HPLC (pp. 17, 41). The sediment was extracted sequentially 1-3 times with 100 mL of methanol:water (1:1, v:v) and 2-3 times with 100 mL of methanol by mechanical shaking (300 strokes/minute) for 45 minutes per extraction. Following each extraction, the mixtures were separated by centrifugation. Portions of each extract were analyzed using LSC. The extracts were combined and evaporated to dryness, and the residues were dissolved in the HPLC mobile phase for analysis (p. 21).

Nonextractable residue determination: Portions of the extracted soils were analyzed for total radioactivity using LSC following combustion (p. 19). The extracted sediment was further extracted twice with 0.1 M sodium hydroxide solution (pp. 17, 41). The alkaline extracts were acidified and the resulting precipitate was removed by centrifugation. Portions of the supernatant (fulvic acid) were analyzed by LSC; the precipitate (humic acid) was redissolved in sodium hydroxide solution and analyzed using LSC. [^{14}C]Residues remaining in the extracted sediment (humin) were quantified by LSC following combustion. In addition, the fulvic acid fraction was extracted with ethyl acetate. The ethyl acetate extract was analyzed using LSC and HPLC, and the extracted solution was analyzed using LSC.

Volatile residue determination: Aliquots of each trapping solution were analyzed for total radioactivity by LSC (p. 18).

Total ^{14}C measurement: Total ^{14}C residues were determined by summing the concentration of residues measured in the water, sediment extracts, extracted sediment, and volatile trapping solutions.

Derivatization method, if used: A derivitization method was not employed.

Identification and quantification of parent compound: Extracts were analyzed by HPLC under the following conditions: YMC-Pack ODS-AQ 120 A column (dimensions and particle size not specified); gradient mobile phase combined A) 0.1% formic acid in water and B) 0.1% formic acid in acetonitrile [% A:B at 0 min. 95:5 (v:v), 5 min 95:5, 15 min 45:55, 25 min 35:65, 31 min 5:95, 36 5:95, 40 min 95:5]; flow rate of 1 mL/minute; UV (254 nm) and IN/US radioactivity detection (p. 16). BAS 510 F was identified by comparison to a reference standard.

Water from the 7-day sample and sediment extracts from the 179-day sample were analyzed using one-dimensional TLC on silica gel plates developed in toluene:acetone:formic acid (80:20:1, v:v:v). Compounds were quantified using a Ambis imaging system and identified by comparison to reference standards.

Identification and quantification of transformation products: Transformation products were separated and quantified as described for the parent. Identification was attempted by comparison to reference standards of 2-(4-chlorophenyl)aniline (BAS No. 363487), 2-hydroxy-N-(4'-chlorobiphenyl-2-yl)-nicotinamide (BAS No. 391572), and 2-chloronicotinic acid (BAS No. 107371) (p. 23).

Detection limits (LOD, LOQ) for the parent compound: Limits of detection for LSC were 60 dpm or 0.007% of the applied radioactivity. The limit of detection for the HPLC on-screen quantification was influenced by the dpm injected and the percent applied radioactivity contained in the fraction. The limit of detection was 0.5-1.0% of the applied radioactivity (p.18)

Detection limits (LOD, LOQ) for the transformation products: Limits of detection were the same as those described for the parent.

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS: Anaerobic conditions were maintained throughout the experiment. Redox potentials ranged from -158 to -305 mV, dissolved oxygen ranged from 0.09 to 4.0 mg/L and the pH ranged from 6.79 to 7.58 (p. 30). It was reported that the temperature was maintained at 20 ± 1°C; supporting records were not provided. The microbial biomass of the system at the start and termination of the study was not determined, but values were reported for the individual phases of the system prior to initiation of the study.

B. MATERIAL BALANCE: Mass balances averaged 101.8 ± 2.29% of the applied radioactivity (range 97.3 to 104.4%) in the pyridine label study and 99.6 ± 3.54% of the applied (range 93.1 to 102.8%) in the diphenyl label study (Table 10, p. 33).

Table 5: Biotransformation of [pyridine-3-^{14}C]-BAS 510 F, expressed as percentage of applied radioactivity (mean ± s.d. when n = 2), in water-sediment system under anaerobic conditions.*

Compound		Sampling times (days)							
		0	7	35	63	88	179	284	361
BAS 510 F	Water	94.5	53.1	14.1	8.3	7.9	4.9	4.1	4.6
	Sediment	NA	35.8	59.1	53.9	53.8	46.9	44.6	41.6
	Entire system	94.5	88.9	73.2	62.2	61.7	51.8	48.7	46.2
"Others"	Water	ND	ND	ND	ND	ND	ND	ND	ND
	Sediment	ND	ND	ND	ND	ND	ND	ND	2.1
Extractable residues	Sediment	4.9	35.8	59.1	53.9	53.8	46.9	44.6± 0.28	43.8± 0.78
Total volatiles[1]	Entire system	---	0.0	0.0	0.0	0.0	0.0	0.1± 0.14	0.2± 0.14
Nonextractable residues	Sediment	0.6	12.0	29.6	35.1	41.1	52.6	53.5± 0.64	55.0± 1.34
Total % recovery	Water	94.5	53.1	14.1	8.3	7.9	4.9	4.1± 0.64	4.6± 0.71
	Sediment	5.5	47.8	88.7	89.0	94.9	99.5	98.1± 0.92	98.7± 0.57
	Entire system	100.0	100.9	102.8	97.3	102.8	104.4	102.7± 0.57	103.5± 1.41

Data obtained from Tables 8-13, pp. 31-36 of study report. Standard deviations and total recovery in sediment were calculated by reviewer.

* At most sampling intervals, only a single sample was analyzed. At 284 and 361 days, duplicate samples were analyzed for total extractable and nonextractable residues and total volatiles to establish the variability of the data.

1 The study author did not distinguish between CO_2 and other volatiles.

NA = Not analyzed

ND = Not detected; individual peaks could not be distinguished from background noise.

Table 6: Biotransformation of [diphenyl-U-^{14}C]BAS 510 F, expressed as percentage of applied radioactivity (mean ± s.d. when n = 2), in water-sediment system under anaerobic conditions.*

Compound		Sampling times (days)							
		0	7	35	63	88	179	284	361
BAS 510 F	Water	92.2	50.9	9.6	8.2	6.9	5.3	3.3	3.6
	Sediment	NA	37.3	61.9	54.0	53.5	44.4	45.9	44.2
	Entire system	92.2	88.2	71.5	62.2	60.4	49.7	49.2	47.8
"Others"	Water	ND	ND	ND	ND	ND	ND	ND	ND
	Sediment	ND	ND	ND	ND	ND	ND	ND	ND
Extractable residues	Sediment	2.7	37.3	61.9	54.0	53.5	44.4	45.9± 0.07	44.2± 0.78
Total volatiles[1]	Entire system	---	0.0	0.0	0.0	0.1	0.4	0.2 ± 0.07	0.2 ± 0.00
Nonextractable residues	Sediment	0.8	10.7	21.6	39.0	42.2	50.9	53.2± 1.20	55.1± 0.21
Total % recovery	Water	92.2	50.9	9.6	8.2	6.9	5.3	3.3 ± 0.28	3.6± 0.35
	Sediment	3.5	48.0	83.5	93.0	96.1	95.3	99.0 ± 1.13	99.2± 0.57
	Entire system	95.7	98.9	93.1	101.1	102.6	100.6	102.3± 1.41	102.8± 0.92

Data obtained from Tables 8-13, pp. 31-36 of study report. Standard deviations and total recovery in sediment were calculated by reviewer.
* At most sampling intervals, only a single sample was analyzed. At 284 and 361 days, duplicate samples were analyzed for total extractable and nonextractable residues and total volatiles to establish the variability of the data.
1 The study author did not distinguish between CO_2 and other volatiles.
NA = Not analyzed
ND = Not detected; individual peaks could not be distinguished from noise.

C. TRANSFORMATION OF PARENT COMPOUND: The rate of dissipation of [pyridine-3-^{14}C]- and [diphenyl-U-^{14}C]-labeled BAS 510 F in the sediment:water systems was similar. In the entire system, [^{14}C]BAS 510 F decreased from 92.2-94.5% of the applied at day 0 to 49.7-51.8% at 179 days posttreatment and 46.2-47.8% at 361 days (Tables 11 and 12, pp. 34-35). In the water phase, [^{14}C]BAS 510 F decreased from 92.2-94.5% of the applied at day 0 to 50.9-53.1% at 7 days posttreatment, 9.6-14.1% at 35 days and 3.6-4.6% at 361 days. In the sediment phase, [^{14}C]BAS 510 F increased to a maximum 59.1-61.9% at 35 days posttreatment, then slowly decreased to 41.6-44.2% at 361 days. The water:sediment distribution ratio of [^{14}C]BAS 510 F was 52:36 at 7 days, 12:60 at 35 days, and 4:43 at 361 days.

The calculated half-life of [pyridine-3-^{14}C]-BAS 510 F in anaerobic water-sediment system determined using linear regression analysis (Excel 2000) was 385 days. The calculated half-life in water was biphasic, with an initial half-life of 13 days and a secondary half-life of 234 days. However, degradation was not observed, and the reported half-lives are not true metabolic half-lives.

The calculated half-life of [diphenyl-U-^{14}C]-BAS 510 F in anaerobic water-sediment system using linear regression analysis (Excel 2000) was 408 days. The calculated half-life in water was biphasic, with an initial half-life of 11 days and a secondary of 217 days. However, degradation was not observed, and the reported half-lives are not true metabolic half-lives.

Half-lives

System			First order Linear			DT50 (days)	DT75 (days)
			Half-life (days)	Regression equation	r^2		
Water	Pyridine label	Initial	13.2	Linear form y = mx + b as lnC= -kt + lnC_0; lnC_0 is initial concentration (b = y intercept), lnC is concentration at time t (y), k is the slope (m), t is time (x) or kt = lnC_0 - lnC. Half-life (t ½) = -(ln 2/k).	0.988	4.1	15.7
		Secondary	223.6		0.753		
	Diphenyl label	Initial	11.0		0.996	3.6	13.9
		Secondary	216.6		0.925		
Sediment	Pyridine label		693.2		0.948	--	--
	Diphenyl label		770.2		0.744	--	--
Entire system	Pyridine label		385.1		0.813	301	>361
	Diphenyl label		407.7		0.767	342	>361
	Combined		385.1		0.815	--	--

Data obtained from pp. 20, 34 and 35. DT50 and DT75 data were calculated by the study author using a nonlinear model (pp. 76-77).

TRANSFORMATION PRODUCTS: No HPLC peaks other than [^{14}C]BAS 510 F could be distinguished from background in water or sediment from the [diphenyl-U-^{14}C]BAS 510 F treatment. No HPLC peaks other than [^{14}C]BAS 510 F could be distinguished from background in water or sediment from the [pyridine-3-^{14}C]-BAS 510 F treatment except at 361 days, when "others" in the sediment totaled 2.1% of the applied. Although the study author did not identify any transformation products, the HPLC chromatograms for 361 days (pp.50-51) show that a small fraction of the residues eluted with 2-hydroxy-N-(4'-chlorobiphenyl-2-yl)-nicotinamide (Reg. No. 391572).

NONEXTRACTABLE AND EXTRACTABLE RESIDUES: Extractable [^{14}C]BAS 510 F residues in sediment increased from 2.7-4.9% of the applied at day 0 to a maximum 59.1-61.9%

at 35 days, then slowly declined to 44.2-43.8% at 361 days. Nonextractable residues increased from 0.6-0.8% of the applied at day 0 to 35.–39.0% at 35 days and 55.0-55.1% at 361 days. At 361 days posttreatment, 4.1-5.6% of the applied was associated with the fulvic acids, 1.2-1.7% with humic acid and 36.9-51.9% with humins (Table 13, p. 36). HPLC analysis of the fulvic acid fraction isolated up to 10 compounds, including BAS 510 F, 2-hydroxy-N-(4'-chlorobiphenyl-2-yl)-nicotinamide, and more polar compounds (pp. 21, 52, 53).

VOLATILIZATION: At 361 days, volatilized [^{14}C]residues totaled 0.2% of the applied (Table 8, p. 31). [^{14}C]Volatiles were not identified.

TRANSFORMATION PATHWAY: A biotransformation pathway for BAS 510 F under anaerobic aquatic conditions was not proposed by the registrant. Based on the study results, BAS 510 F is converted primarily to soil bound residues or remained present as the parent compound.

Table 7: Chemical names and CAS numbers for the transformation products of [^{14}C]BAS 510 F.

Applicant's Code Name	CAS Number	CAS and/or IUPAC Chemical Name(s)	Chemical formula	Molecular weight	SMILES string
		No transformation products were identified by the study author.			

D. SUPPLEMENTARY STUDY- RESULTS: Although some samples were dosed at a higher rate to facilitate the identification of degradates, there is no indication that these samples were ever used.

III. STUDY DEFICIENCIES: No deficiencies were identified. This study is acceptable and can be used to fulfill the anaerobic aquatic metabolism data requirement (§162-3).

IV. REVIEWER'S COMMENTS

1. The study author does not identify any transformation products. However, in the representative chromatograms provided with the study, a peak corresponding to 2-hydroxy-N-(4'-chlorobiphenyl-2-yl)-nicotinamide is identified. Since unidentified extractable residues were isolated only once, at 2.1% of the applied, it is not required that the transformation product be identified. However, it would have been useful if the study author had mentioned this as a tentative identification.

2. In Table 13 (p. 36), the study author identifies the [^{14}C]residues remaining in the sediment after the methanol:water and methanol extractions as "Available." However, the "Available" concentrations reported in this table are different from the concentrations reported for residual [^{14}C]residues in Tables 9 and 10 (pp. 32-33).

3. Although two samples were collected at each sampling interval, only single samples were analyzed at most intervals. Replicate sampling at each sampling interval is necessary so that normal variability can be quantified and outliers can be identified. For the purposes of the half-life determination, the combined data from the different radiolabel studies was utilized since similar patterns of dissipation were observed between the two studies. However, duplicate samples should have been utilized for the analysis of degradates.

4. Volatile organics were not characterized and the presence of CO_2 in the NaOH trapping solutions was not confirmed.

5. The microbiological analysis of the sediment and water was done at the soil analysis lab after field collection. Initial and final microbial biomass concentrations for the anaerobic water and sediment system were not measured, so the biological viability of the system could not be determined.

6. The application rate was reported to be approximately equal to the field rate of 700 g a.i. over a hectare of water 30 cm deep (p. 20).

7. The registrant determined DT50's using the Gustafson-Holden model to describe dissipation kinetics using nonlinear fit. The registrant's DT50 for water dissipation was 4.1 days for the [pyridine-3-^{14}C]-BAS 510 F and 3.6 days for the [diphenyl-U-^{14}C]-BAS 510 F. For the total system, the registrant reported DT50's of 302 days for the [pyridine-3-^{14}C]-BAS 510 F and 342 days for the [diphenyl-U-^{14}C]-BAS 510 F. (p.76 and 77).

8. BAS 510 F chemical name 2-chloro-*N*-(4'-chlorobiphenyl-2-yl)-nicotinamide, as presented in the study report, was identified as the IUPAC name by the Compendium of Pesticide Common Names (http://www.hclrss.demon.co.uk/nicobifen.html). The CAS name 2-chloro-*N*-(4-chloro[1,1-biphenyl]-2-yl)-3-pyridinecarboxamide was also obtained from the Compendium of Pesticide Common Names. The following BAS 510 F synonyms were obtained from USEPA/OPP Chemical Databases (http://www.cdpr.ca.gov/cgi-bin/epa/chemidetriris.pl?pccode=128008 and (http://www.cdpr.ca.gov/cgi-bin/mon/bycode.pl?p_chemcode=5790): 2-chloro-*N*-(4'-chlorobiphenyl-2-yl)-nicotinamide, nicobifen, and BAS 516 02 F.

V. REFERENCES:

1. Gustafson, D.I. and L. Holden. 1990. Nonlinear Pesticide Dissipation in Soil: A new model based on spacial variability. Environ. Sci. Technol., Vol. 24, No. 7, p. 1032-1038.

2. Microcal Software, Inc., Origin 6.0, One Roundhouse Plaza, Northampton, MA 01060.

3. Snedecor, G.W. and W.G. Cochran. 1987. Statistical Methods. The Iowa State University Press. Ames, IA, USA.

4. Wolt, J.D. 1994. Soil Solution Chemistry. John Wiley and Sons, Inc., New York, NY - USA.

5. Wolt, J.D., H.P. Nelson, Jr., C.B. Cleveland, and I. J. van Wesenbeeck. 2001. Biodegradation kinectics for pesticide exposure assessment. Reviews of environmental contamination and toxicology. (Accepted)

6. Sparks, D.L. 1989. Kinetics of soil chemical processes. Academic Press, San Diego, CA - USA.

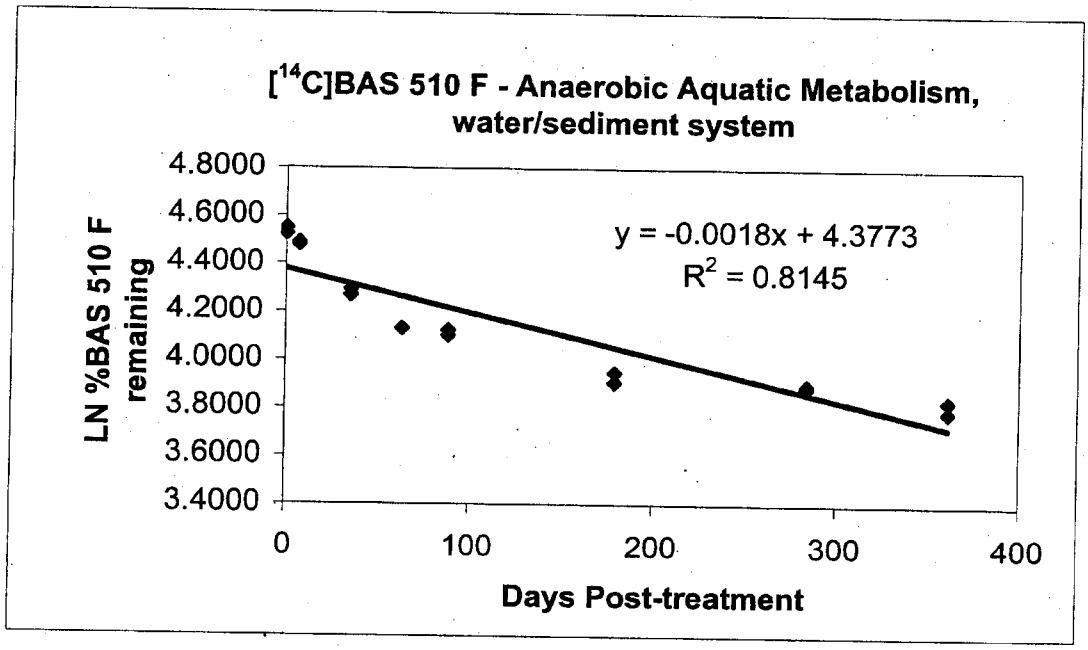
Attachment 1

Excel Spreadsheets

Chemical Name	BAS 510 F	Pyridine and Diphenyl ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Clay loam sediment/water system		

Half-life (days)	385.08

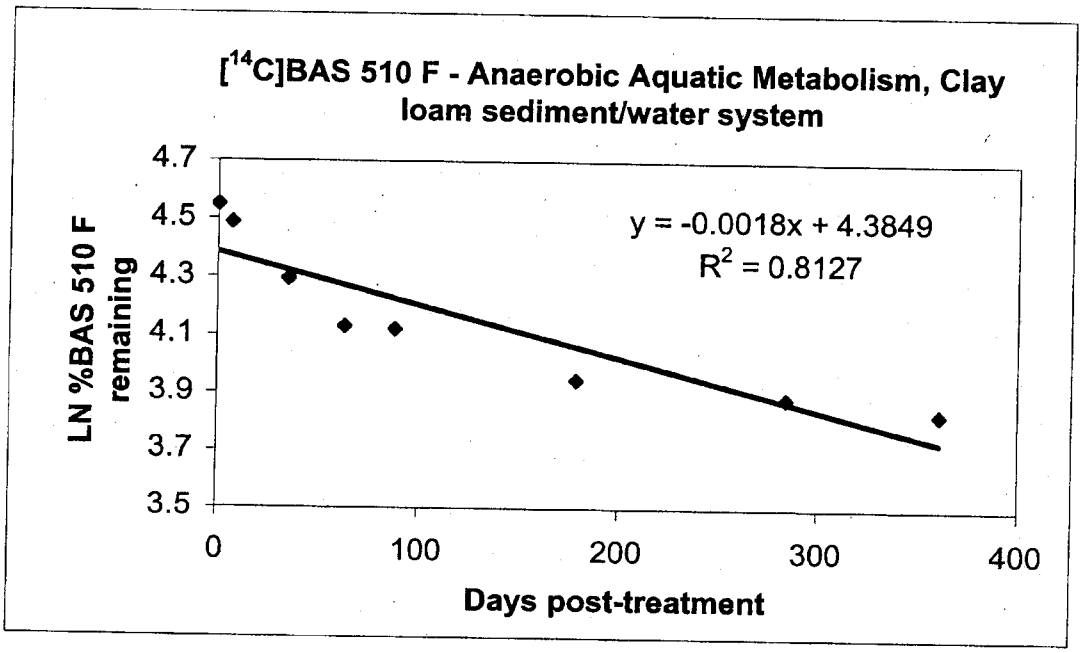
Days post-treatment	% BAS 510 F remaining	LN (%remaining)
0	94.5	4.5486
0	92.2	4.5240
7	88.9	4.4875
7	88.2	4.4796
35	73.2	4.2932
35	71.5	4.2697
63	62.2	4.1304
63	62.2	4.1304
88	61.7	4.1223
88	60.4	4.1010
179	51.8	3.9474
179	49.7	3.9060
284	48.7	3.8857
284	49.2	3.8959
361	46.2	3.8330
361	44.2	3.7887



Chemical Name	BAS 510 F	Pyridine ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Clay loam sediment/water system		

Half-life (days) =	385.08

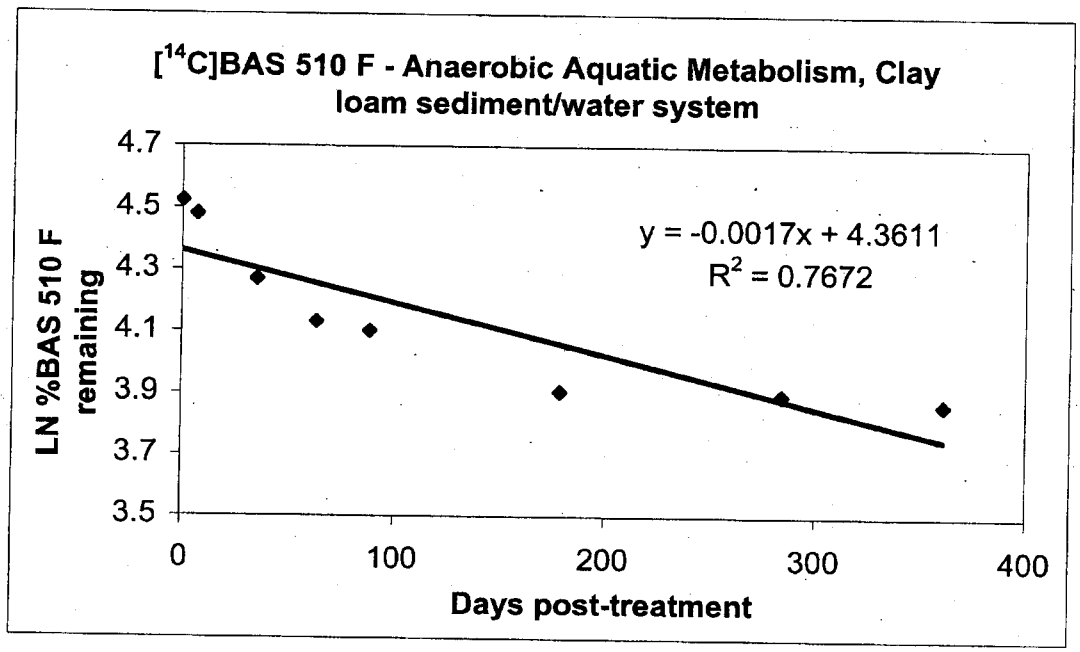
Days post-treatment	% BAS 510 F remaining	LN (%remaining)
0	94.5	4.5486
7	88.9	4.4875
35	73.2	4.2932
63	62.2	4.1304
88	61.7	4.1223
179	51.8	3.9474
284	48.7	3.8857
361	46.2	3.8330



Chemical Name	BAS 510 F	Diphenyl ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Clay loam sediment/water system		

Half-life (days) =	407.73

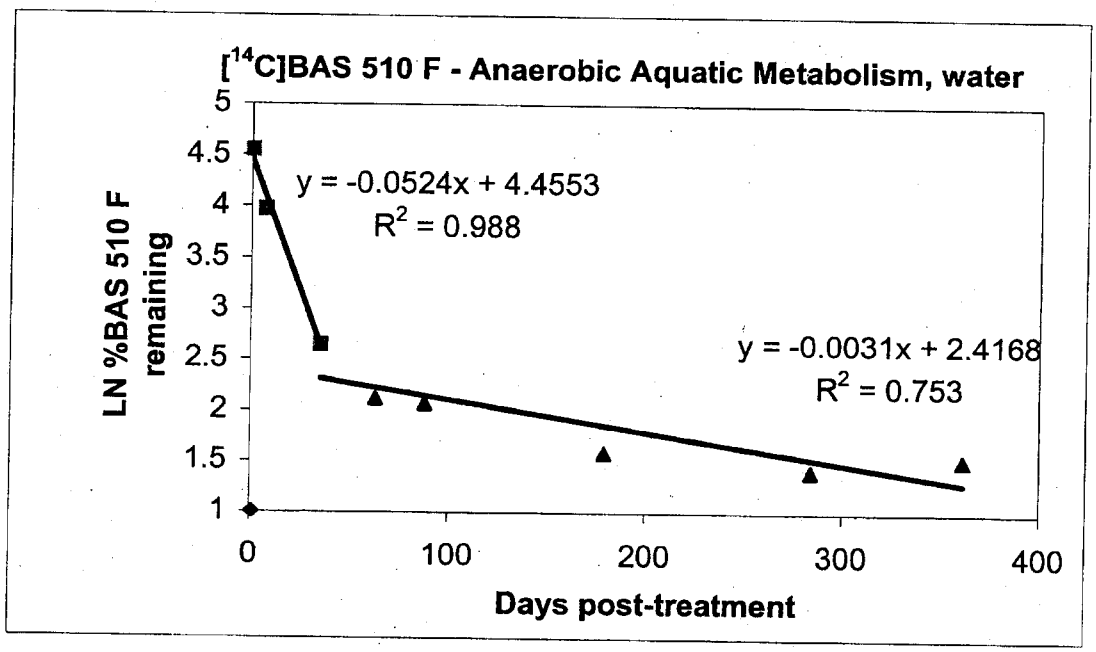
Days post-treatment	% BAS 510 F remaining	LN (%remaining)
0	92.2	4.5240
7	88.2	4.4796
35	71.5	4.2697
63	62.2	4.1304
88	60.4	4.1010
179	49.7	3.9060
284	49.2	3.8959
361	47.8	3.8670



Chemical Name	BAS 510 F	Pyridine ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Water		

	1st	2nd
Half-life (days) =	13.23	223.59

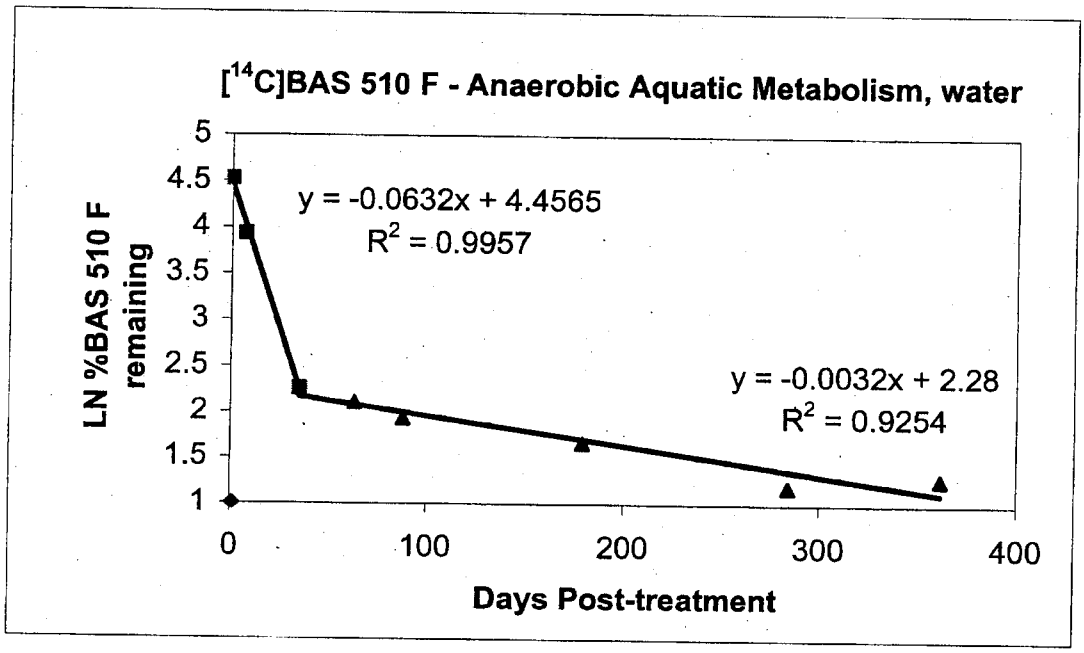
Days post-treatment	% BAS 510 F remaining	LN (%remaining)
0	94.5	4.5486
7	53.1	3.9722
35	14.1	2.6462
63	8.3	2.1163
88	7.9	2.0669
179	4.9	1.5892
284	4.1	1.4110
361	4.6	1.5261



Chemical Name	BAS 510 F	Diphenyl ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Water		

	1st	2nd
Half-life (days)	10.97	216.61

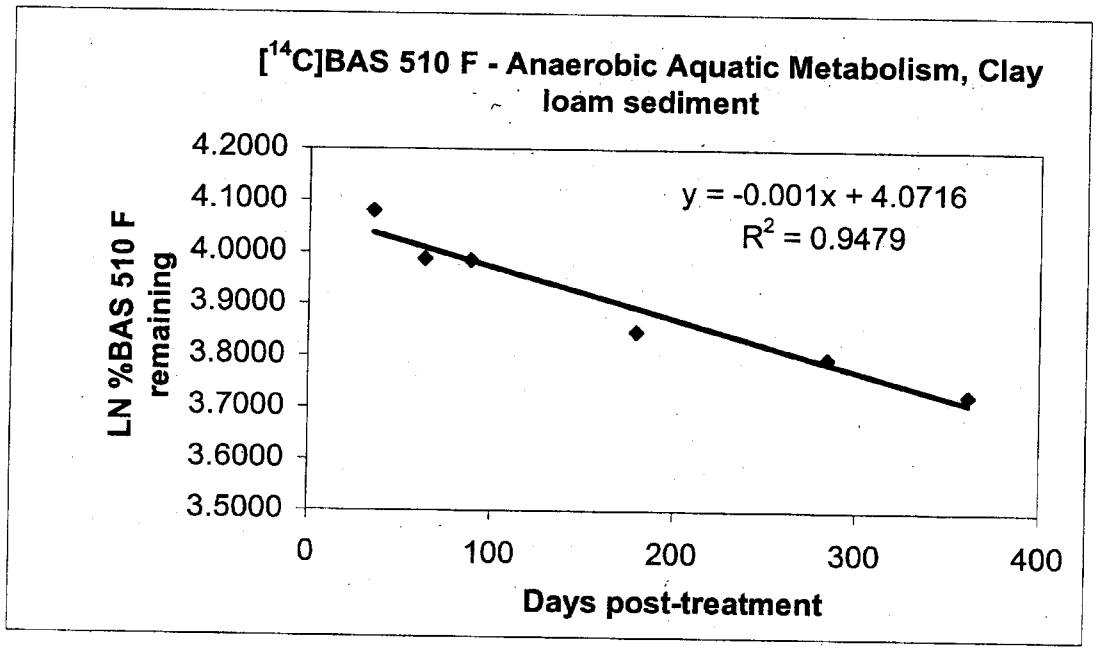
Days post-treatment	% BAS 510 F remaining	LN (%remaining)
0	92.2	4.5240
7	50.9	3.9299
35	9.6	2.2618
63	8.2	2.1041
88	6.9	1.9315
179	5.3	1.6677
284	3.3	1.1939
361	3.6	1.2809



Chemical Name	BAS 510 F	Pyridine ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Clay loam sediment		

Half-life (days) =	693.15	

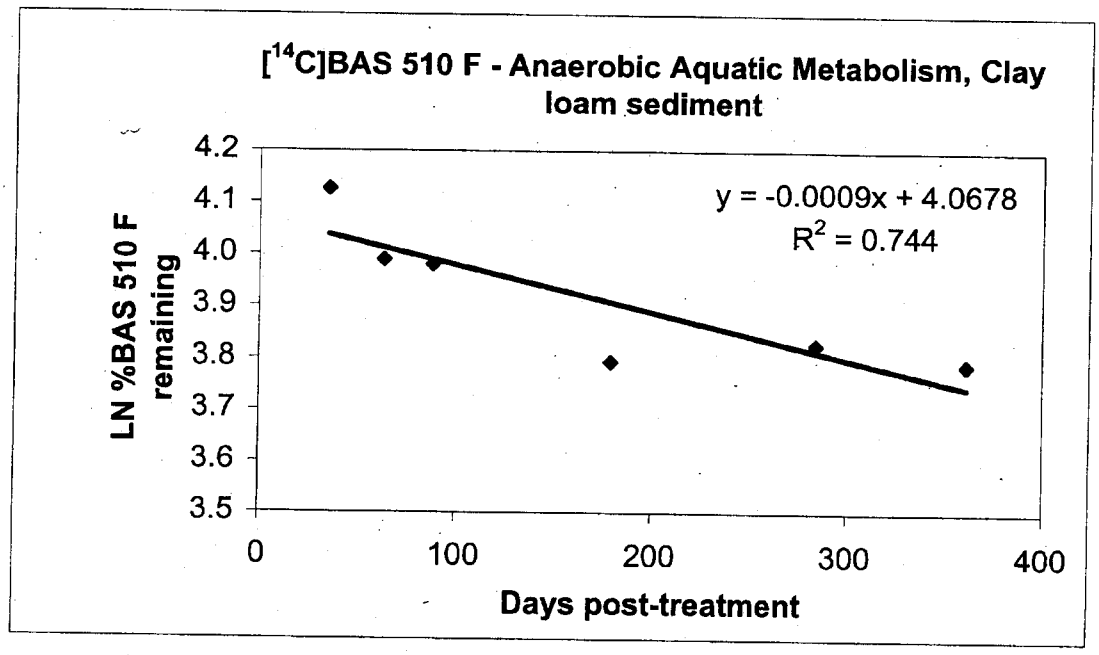
Days post-treatment	% BAS 510 F remaining	LN (%remaining)
35	59.1	4.0792
63	53.9	3.9871
88	53.8	3.9853
179	46.9	3.8480
284	44.6	3.7977
361	41.6	3.7281



Chemical Name	BAS 510 F	Diphenyl ring
PC Code	1280008	
MRID	45405213	
Study No.	162-3	
Clay loam sediment		

Half-life (days) =	770.16	

Days post-treatment	% BAS 510 F remaining	LN (%remaining)
35	61.9	4.1255
63	54	3.9890
88	53.5	3.9797
179	44.4	3.7932
284	45.9	3.8265
361	44.2	3.7887

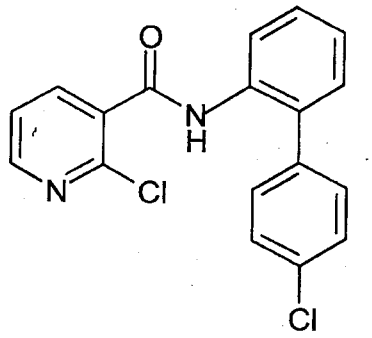


Attachment 2

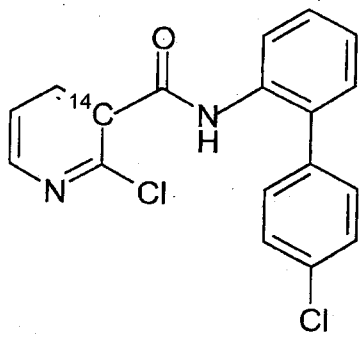
Structures of Parent and Transformation Products

BAS 510 F

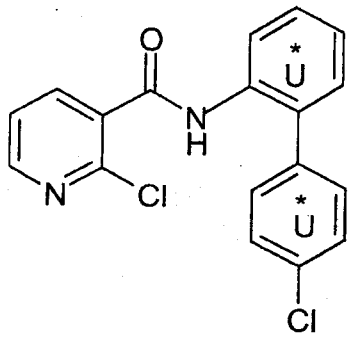
IUPAC name: 2-Chloro-*N*-(4-chlorobiphenyl-2-yl)-nicotinamide.
CAS name: 2-Chloro-*N*-(4-chloro[1,1-biphenyl]-2-yl)-3-pyridinecarboxamide.
CAS No: 188425-85-6.
Synonyms: 2-Chloro-*N*-(4'-chlorobiphenyl-2-yl)-nicotinamide, Nicobifen. BAS 516 02 F.



[Pyridine-3-^{14}C]-labeled BAS 510 F



[Diphenyl-U-^{14}C]-labeled BAS 510 F



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