Data Evaluation Report on the phototransformation of BAS 510 F on soil

PMRA Submission Number {......} EPA MRID Number 45405207

Data Requirement: PMRA Data Code:
EPA DP Barcode: D278387
OECD Data Point:
EPA Guideline: 161-3

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: Nicobifen, ~~BAS 516 02 F~~
SMILES string:

Primary Reviewer: Mary Thomas
Dynamac Corporation
Signature: Mary Thomas
Date: 1/15/02

QC Reviewer: Joan Harlin
Dynamac Corporation
Signature: Joan L. Harlin
Date: 1/15/02

Secondary Reviewer: Cheryl Sutton
EPA
Signature: Cheryl Sutton
Date: 11/02

Company Code: [for PMRA]
Active Code: [for PMRA]
Use Site Category: [for PMRA]
EPA PC Code: 128008

CITATION: von Götz, N. 2000. Soil photolysis of BAS 510 F. Unpublished study performed by BASF Aktiengesellschaft, BASF Agricultural Center Limburgerhof, D-67114 Limburgerhof, Germany and sponsored by BASF Corporation, Agricultural Products, Research Triangle Park, NC. BASF Registration Document Number 2000/1014989. Study initiated October 5, 1999 and completed October 20, 2000.

2017223

EXECUTIVE SUMMARY

The phototransformation of [pyridine-3-^{14}C]BAS 510 F was studied on a German sandy loam soil [pH 7.3, organic carbon 1.9%] at 4.6 µg a.i/g soil for 15 days at 22 ± 1°C and 40% maximum water holding capacity. The experiment was conducted in accordance with the US EPA Pesticide Assessment Guidelines, Subdivision N, Section 161-3, and in compliance with the Good Laboratory Practice Regulations. [^{14}C]BAS 510 F was surface-applied to the soil samples using a Hamilton syringe. The treated samples were continuously irradiated for 15 days using a xenon lamp equipped with a UV filter to remove wavelengths <290 nm. Test vessels were connected to ethylene glycol, sulfuric acid and NaOH traps for the collection of organic volatiles and CO_2, respectively. Samples were taken at 0, 2, 6, 9, 12 and 15 days for the determination of [^{14}C]BAS 510 F and its transformation products. The soil samples were extracted with methanol and methanol:water, and the [^{14}C]residues were analyzed by HPLC.

The mass balance was 102.9 ± 1.4% and 98.2 ± 0.4% in the dark and irradiated samples, respectively. At test termination, 99.2% of the applied radioactivity remained as [^{14}C]BAS 510 F in the dark samples. Metabolite L was ≤0.6% during the study period. At study termination, the evolved CO_2 in the dark samples was 0.1% of the applied amount. Organic volatiles were not formed.

In the irradiated samples, the concentration of [^{14}C]BAS 510 F decreased from 99.4% at day 0 to 90.6% of the applied amount at the end of the study. No major transformation products were detected. Metabolites C and L were ≤1.2% during the study period. In the irradiated samples at study termination, 0.2% of the recovered radioactivity was present as CO_2. Organic volatiles were not formed.

Extractable [^{14}C]residues decreased from 100.0% of the applied amount at Day 0 to 99.9% and 92.6% of the applied at the end of incubation period in the dark and irradiated samples, respectively. Non-extractable [^{14}C]residues in the irradiated samples increased from 0.1% of the applied amount at Day 0 to 5.6 % of the applied at study termination. In the dark controls, the non-extractable radioactivity was 0.1% of the applied amount at day 0 and 2.9% of the applied at study termination.

[^{14}C]BAS 510 F was stable (<10% degradation; half-life of 133 days) in the irradiated samples; a half-life in the dark control could not be determined.

Results Synopsis

Soil type:	sandy loam
Source of irradiation:	xenon lamp
Half-life/DT50 for dark control:	Not determined
Half-life for irradiated samples:	stable (half-life of 133 days)

Major transformation products: None
Minor transformation products: Metabolites C and L, CO_2

Study Acceptability: This study is classified acceptable and satisfies the guideline data requirement for a study on phototransformation on soil.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: The study was conducted according to the US EPA Pesticide Assessment Guidelines, Subdivision N, Section 161-3; EPA Pesticide Reregistration Rejection Rate Analysis; SETAC "Procedure For Assessing the Environmental Fate and Ecotoxicity of Pesticides"; Commission Directive 95/36/EC amending Council Directive 91/414/EEC; and Appendix 1 to § 19a, Section 1, Chemikaliengesetz of 25 July 1994 (Official Bulletin/Federal Republic of Germany, I 1994, P. 1703). No deviations affected the validity of the study.

COMPLIANCE: This study was conducted in compliance with Good Laboratory Practice Regulations; Appendix 1 to § 19a, Section 1, Chemikaliengesetz of 25 July 1994 (Official Bulletin/Federal Republic of Germany, I 1994, P. 1703). Signed and dated GLP, Quality Assurance and Data Confidentiality statements were provided.

A. MATERIALS:

1. Test Material [Pyridine-3-^{14}C]BAS 510 F

Chemical Structure:

Description: Not provided

Purity:
Analytical purity: Not provided
Radiochemical purity: 100% Batch No. 640-2037
Specific activity: 5.17 MBq/mg
Locations of the radiolabel: Carbon 3 of pyridine ring

Storage conditions of test chemicals: Not provided

Physico-chemical properties of BAS 510 F:

Parameter	**Values**	**Comments**
Water solubility	6 mg/L in water at 20°C	
Vapour pressure/volatility	Not provided	
UV absorption	Not provided	
pK_a	Not provided	
K_{ow}	Not provided	
Stability of Compound at room temperature, if provided	Not provided	

Data obtained from MRID 45405216, p. 12 of the study report.

2. Soil Characteristics:

Table 1: Field information and handling procedures.

Information	**Details**
Geographic location	Limburgerhof, Bruch West, Germany
Pesticide use history at the collection site	Not reported
Collection procedures	Not reported
Sampling depth (cm)	Not reported
Storage conditions	Not reported
Storage length	Not reported
Soil preparation (eg: 2 mm sieved; air dried etc.)	2 mm sieved

Data obtained from p. 11 in the study report.

Table 2: Properties of the soil.

Property	**Details**	
Soil texture	Sandy loam	
% sand (50-2000 μm)	63	
% silt (2-50 μm)	27	
% clay (< 2 μm)	10	
pH ($CaCl_2$)	7.3	
Organic carbon (%)	1.9	
CEC (mval/100 g)	9.8	
Soil Moisture	40% of maximum water-holding capacity	
Bulk density (g/cm^3)	Not reported	
Microbial biomass/microbial population (unit)	Initial	Final
	Not reported	Not reported
Soil Taxonomic classification	Not reported	
Soil mapping unit (for EPA)	Not reported	

Data obtained from pp. 11 and 13 of the study report.

3) Details of light source:

Table 3: Artificial light source.

Property	Details
Type of lamp used	Xenon lamp
Emission wavelength spectrum	290-1220 nm
Light intensity	About 3 mW/cm^2
Filters used	UV filter that removed wavelengths <290 nm
Relationship to natural sunlight	Simulated a clear summer day (not further described)

Data obtained from p. 13 and Appendix 1, p. 29 in the study report.

B. EXPERIMENTAL DESIGN

1) Preliminary Study: A preliminary study was not conducted.

2) Experimental Design

Table 4: Experimental design.

Parameter		Details
Duration of the test		15 days
Condition of soil:	Air-dried/fresh:	Fresh
	Sterile/Non-sterile:	Not reported
Test concentrations (µg a.i/g soil) dark and irradiated		4.6
Dark controls used (Yes/No) Is it foil wrapped and kept in darkness		Yes Kept in darkness; stored in climatic chamber
Replications	Dark control:	2
	Irradiated:	2
Identity and concentration of co-solvent:		Methanol, ca. 9% (280 µL/30 g)
Pesticide application	Volume of test solution used/treatment:	280 µL
	Method of application (eg., spraying):	Applied to soil surface using a Hamilton syringe.
	Is the co-solvent evaporated:	No

Parameter		Details
Test apparatus: Type/Material/Volume		For both irradiated and dark control test, 11 small dishes (86 mm x 40 mm x 10 mm) filled with soil were placed in rectangular bowl closed airtight with quartz glass covering.
Details of traps for volatile, if any		Moistened air was drawn (1-2 bubbles/sec) through the vessels, then sequentially through trapping solutions of ethylene glycol and 0.5 M sulfuric acid to collect organic volatiles and 0.5M NaOH to collect CO_2.
If no traps were used, is the system closed/open		Not applicable
Any indication of the test material adsorbing to the walls of the test apparatus		Not determined
Experimental Conditions	Temperature: Temperature maintenance method:	22 ± 1°C The rectangular bowl containing the soil samples was placed inside a Suntest CPS apparatus equipped with a water cooling system and an external air-conditioning apparatus connected to inside of the apparatus. A temperature probe placed in one of the 11 small dishes of soil for both the irradiated and dark control tests to monitor temperature.
	Moisture content: Moisture maintenance method	40% maximum water-holding capacity Soil samples were remoistened with water as needed; samples weighed daily to determine soil moisture content.
	Duration of light/darkness:	15 day continuous irradiation
Other details, if any		None

Data obtained from pp. 11-14 and Figure 3, p. 25 in the study report.

3) Supplementary experiments: Supplementary experiments were not conducted.

4) Sampling:

Table 5: Sampling details.

Criteria	Details
Sampling intervals of soil samples	0, 2, 6, 9, 12 and 15 days
Sampling method	Duplicate whole sample
Method of sampling CO_2 and volatile organic compounds, if any	Entire trapping solution sampled

Criteria	Details
sampling intervals/times for: sterility check, if any moisture content:	Not determined Daily
Sample storage before analysis	Specific intervals not reported. Samples "analysed as soon as possible." For storage, samples kept in freezer.
Other observations, if any	None

Data obtained from pp. 14 and 16 in the study report.

C. ANALYTICAL METHODS

Extraction/clean up/concentration methods: Soil samples were sequentially extracted three times each with methanol (80 mL), then methanol:water (80 mL; 1:1, v:v). The suspensions were manually shaken for 10 minutes, then centrifuged. The corresponding methanol and methanol:water extracts were pooled, respectively, the volume adjusted, and refrigerated prior to analysis. The methanol and methanol:water extracts were concentrated by rotary evaporation at 35°C, and the residues were redissolved in 10 mL methanol or methanol:water (1:1, v:v), respectively, prior to HPLC analysis.

Non-extractable residue determination: Not applicable.

Total ^{14}C measurement: Aliquots of the extracts and trapping solutions were analyzed for total ^{14}C by LSC. Extracted soil samples were air dried and then homogenized by grinding in a mill, and triplicate subsamples were analyzed for total radioactivity by LSC following combustion.

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

Identification and quantification of parent compound: Identification and quantification of the parent compound were performed by reverse-phase HPLC using the following operating conditions: Spherisorb ODS II column (250 mm x 4.0 mm; 10 µm particle size), C18 (ODS) column (4 mm x 3 mm), gradient mobile phase combining (A) water:acetonitrile:formic acid (950:50:1, v:v:v), (B) water:acetonitrile:formic acid (50:950:1, v:v:v) [B (0%) at 0 min, acquisition on, B (100%) at 60 min, B (100%) at 65 min, acquisition off], flow rate 1.0 mL/minute; and UV (wavelength not specified) and radioactivity detection (p. 15). The samples were co-chromatographed with an unlabeled reference standard of BAS 510 F. The identity of the parent compound was confirmed by chromatographic comparison of the HPLC retention time of a reference standard.

Identification and quantification of transformation products: Identification and quantification of transformation products were performed as described for the parent compound. Reference standards for potential degradation products were not identified. Radioactivity recovered in the sodium hydroxide trapping solutions was assumed to be $^{14}CO_2$; a confirmational method was not employed.

Detection limits (LOD, LOQ) for BAS 510 F: Detection limits for parent compound were not reported.

Detection limits (LOD, LOQ) for the transformation products: Detection limits for transformation products were not reported.

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS: Soil moisture was maintained at 40% of maximum water-holding capacity during the study. It was stated that the treated soils were maintained at the reported 22 ± 1°C throughout the study; temperature records were not provided.

B. MASS BALANCE: Total radiocarbon recovery ranged from 100.1% to 103.6 ± 2.1% and 98.2 ± 0.4% to 103.7 ± 0.1% of the applied radioactivity in the dark and in the irradiated samples, respectively.

Table 6: Phototransformation of [pyridine-3-^{14}C]BAS 510 F, expressed as percentage of applied radioactivity, on sandy loam soil.

Compound		**Sampling times (days)**					
		0	2	6	9	12	15
BAS 510 F*	irradiated	99.3 ± 1.0	98.7 ± 2.6	96.7 ± 0.1	97.1 ± 0.3	95.2 ± 0.9	90.6 ± 0.1
	dark	99.3 ± 1.0	98.1 ± 0.0	98.6 ± 0.7	100.1 ± 0.9	99.4 ± 2.0	99.2 ± 2.3
Metabolite C	irradiated	0.0	0.1	0.6	0.6	1.2	0.6
	dark	0.0	0.0	0.0	0.0	0.1	0.0
Metabolite L	irradiated	0.6	0.8	0.8	0.9	0.9	0.5
	dark	0.6	0.6	0.5	0.4	0.2	0.3
Unidentified radioactivity	irradiated	Not determined					
	dark						
Total extractable residues*	irradiated	100.0	100.1 ± 2.5	98.7 ± 0.5	99.3 ± 0.1	98.5 ± 1.3	92.6 ± 0.3
	dark	100.0	99.1 ± 0.2	99.4 ± 0.4	100.6 ± 0.8	100.4 ± 1.8	99.9 ± 1.8

Compound		Sampling times (days)					
		0	2	6	9	12	15
Non-extractable residues*	irradiated	0.2 ± 0.1	2.1 ± 0.4	3.9 ± 0.2	4.5 ± 0.3	4.5 ± 0.6	5.6 ± 0.2
	dark	0.1	1.4 ± 0.0	2.3 ± 0.1	2.3 ± 0.5	3.2 ± 0.3	2.9 ± 0.3
CO_2	irradiated	0.0	0.0	0.1	0.1	0.1	0.2
	dark	0.0	0.0	0.0	0.0	0.1	0.1
Volatile organic compounds	irradiated	Not determined					
	dark						
Total % recovery*	irradiated	100.1 ± 1.1	102.2 ± 2.1	102.6 ± 0.3	103.7 ± 0.1	103.0 ± 0.7	98.2 ± 0.4
	dark	100.1	100.5 ± 0.2	101.7 ± 0.5	103.0 ± 1.3	103.6 ± 2.1	102.9 ± 1.4

Data obtained from Tables 1-2, p. 20, Tables 5-6, p. 22, Appendices 3-8, pp. 30-35 in the study report.
* Reviewer calculated the mean and standard deviation using Excel program with the data obtained from Appendices 3-8, pp. 30-35 in the study report.

C. TRANSFORMATION OF PARENT COMPOUND: In the dark control at test termination, 99.2% of the applied radioactivity remained as [^{14}C]BAS 510 F. Metabolite L was ≤0.6% during the study period. At study termination, the evolved CO_2 in the dark samples was 0.1% of the applied radioactivity. Organic volatiles were not formed.

In the irradiated samples, the concentration of [^{14}C]BAS 510 F decreased from 99.4% at day 0 to 90.6% of the applied radioactivity at the end of the study. No major transformation products were detected. Metabolites C and L were ≤1.2% during the study period. In the irradiated samples, 0.2% of the recovered radioactivity was present as CO_2 at study termination. Organic volatiles were not detected.

NON-EXTRACTABLE AND EXTRACTABLE RESIDUES: Extractable [^{14}C]residues decreased from 100.0% of the applied amount at Day 0 to 99.8% and 92.6% of the applied at the end of incubation period in the dark and irradiated samples, respectively. Non-extractable [^{14}C]residues in the irradiated samples increased from 0.1% of the applied amount at Day 0 to 5.5% of the applied at study termination. In the dark samples, the non-extractable [^{14}C]residues were 0.1% of the applied amount at day 0 and 2.9% of the applied at study termination.

PATHWAY: The pathway of phototransformation of [pyridine-3-^{14}C]BAS 510 F on soil could not be determined.

Table 7: Chemical names and CAS numbers for the transformation products of [pyridine-3-^{14}C]BAS 510 F.

Applicant's Code Name	CAS Number	CAS and/or IUPAC Chemical Name(s)	Chemical formula	Molecular weight	SMILES string
Not applicable					

HALF-LIFE: [pyridine-3-^{14}C]BAS 510 F was stable (<10 degradation) in the irradiated soil sample (half-life of 133 days).

Half-life

Test system	First order/other half-life		Half-life (days)
	Regression equation	r^2	
Dark	Not determined		
Irradiated	Not provided	0.8281	133

D. SUPPLEMENTARY EXPERIMENT-RESULTS: Supplementary experiments were not conducted.

III. STUDY DEFICIENCIES: The objective of this study was to study the photolytic degradation of [^{14}C]BAS 510 F on sandy loam soil. None of the study deficiencies noted are considered to be of sufficient concern to cause the study to be judged scientifically invalid. The study fulfills Subdivision N Guideline §161-3.

IV. REVIEWER'S COMMENTS:

1. The reviewer-calculated half-life of 133 days is of limited value because the data have been extrapolated far beyond the duration of the experiment.
2. The soil was maintained at 40% of the maximum water holding capacity during the study. Subdivision N guidelines specify that soil moisture be maintained at maintained at 75% of 0.33 bar moisture content throughout the study period.
3. Complete details of the soil properties were not provided. The soil bulk density and microbial biomass at the beginning and end of the study were not reported.
4. The distance from the light source to the soil surface was not reported. It was not specified at what distance from the light source intensity measurements were taken. In addition, the intensity of the artificial light source prior to and after the study was completed was not determined.
5. The adsorption spectrum of the test substance in solution was not provided.
6. Physical properties of the test substance were not provided in the study report.

7. The test soil used was a foreign soil. However, the test soil characteristics were similar to those of soil found at the intended application sites in the United States. In addition, the test soil was classified according to the USDA Soil Textural Classification System.

8. A soil series name for the German test soil was not reported.

9. Radioactivity present in the sodium hydroxide trapping solutions was assumed to be $^{14}CO_2$; a method to confirm the identity of $^{14}CO_2$ was not employed. The presence of $^{14}CO_2$ in the sodium hydroxide trapping solutions should have been confirmed using a method such as barium chloride precipitation.

10. The application rate used in the study was based on a recommended field application rate of 700 g/ha. The amount of test substance applied to the soil was approximately 4.6 µg/g dry soil (p. 13).

11. The test soil was reported to be the same as the test soil used in a soil metabolism study (p. 11); that study was not referenced in the present study report.

12. The study author stated that the study was conducted using only one radiolabel since degradation was <10% of the total applied radioactivity (p. 10).

13. Details of the storage conditions used for the soils samples and sample extracts were not provided. It was stated that all samples were stored frozen and analyzed as soon as possible (p. 16). It was also stated that the methanol and methanol:water extracts were refrigerated prior to analysis (p. 14). Specific storage intervals and conditions for the samples and sample extracts should have been provided.

14. The limits of detection and quantitation for the HPLC methods were not reported. Limits of detection and quantitation should be reported to allow the reviewer to evaluate the adequacy of the test method.

15. The water solubility was reported to be 6 mg/L in water at 20°C mg ai/L in MRID 45405216.

16. 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: The following references were cited in the study:

[1] Scharf, J., Measurement of the Seasonal Progress of the Sunlight Intensity, unpublished BASF report, Reg. Doc. BASF #98/10835.

[2] Walker, A., and Crout, N., Modelmaker, User Manual, Version 3-Cherwell Scientific Publishing Limited, Oxford, 1997.

Chemical Name	BAS 510 F
PC Code	128008
MRID	45405207
Guideline No.	161-3
Label	Pyridine-3-^{14}C

Half-life (days) = **133**

Irradiated samples

Days Posttreatment	[^{14}C]BAS 510 F (Mean Percent of Applied)	Ln ([^{14}C]BAS 510 F)
0	99.4	4.599152114
2	98.7	4.592084946
6	96.7	4.571613402
9	97.1	4.575741375
12	95.2	4.555979942
15	90.6	4.506454213