Data Evaluation Report on the phototransformation of BAS 510F in water

PMRA Submission Number {.....} EPA MRID Number 45405206

Data Requirement: PMRA Data Code:
EPA DP Barcode: D278387
OECD Data Point:
EPA Guideline: 161-2

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

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

CITATION: von Götz, N. 1999. Aqueous photolysis of BAS 510 F. Unpublished study performed by BASF Aktiengesellschaft, BASF Agricultural Center Limburgerhof, D-67114 Limburgerhof, Germany and sponsored by BASF Corporation,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 1999/11804. Study initiated March 4, 1999 and completed December 3, 1999.

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EXECUTIVE SUMMARY:

The aqueous phototransformation of [pyridine-3-^{14}C]BAS 510 F was studied at 22 ± 1°C in sterile aqueous acetate buffer solutions at pH 5 at an initial concentration of 3 µg a.i./mL under continuous irradiation for 15 days using xenon lamp equipped with a UV filter to remove wavelengths <290 nm. The experiment was conducted in accordance with the US EPA Pesticide Assessment Guidelines, Subdivision N, and in compliance with Good Laboratory Practice Regulations. Test vessels were connected to ethylene glycol, sulfuric acid and NaOH traps for the collection of CO_2 and organic volatiles. Solutions were sampled at 0, 1, 4, 8 and 15 days and directly analyzed by HPLC.

The mass balance was 98.8 ± 2.4% and 97.4 ± 2.3% of the applied amount in the dark and irradiated samples, respectively. At test termination 101.0% of the applied radioactivity was [^{14}C]BAS 510 F in the dark samples. In the irradiated samples, the concentration of the parent compound was 100.0% at day 0 and 94.4% of the applied amount at the end of the study. The decline appeared to be normal variation, since parent was the only compound observed on HPLC chromatograms at day 15. CO_2 totaled 0.1% of the applied and no organic volatiles formed.

In the dark and the irradiated samples, [^{14}C]BAS 510 F was stable in pH 5 acetate buffer; a half-life could not be determined.

The half-life/DT50 for phototransformation was not determined.

An environmental phototransformation half-life was not predicted.

The quantum yield of [^{14}C]BAS 510 F was estimated to be smaller than $\Phi_{ts} = 2.45 \times 10^{-4}$.

Results Synopsis

Test medium:	0.01 M acetate buffer at pH 5
Source of irradiation:	Xenon lamp
Half-life/DT50 for Dark:	Stable
Half-life/DT50 for phototransformation:	Stable

Major transformation products: None
Minor transformation products: None

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

I. MATERIALS AND METHODS

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GUIDELINE FOLLOWED: The study was conducted according to the US EPA Pesticide Assessment Guidelines, Subdivision N, Section 161-2; Commission Directive 94/37/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 Lot/Batch No. Not provided

Radiochemical purity: 99.4% Batch No. 640-2037

Specific activity: 5.16 MBq/mg

Locations of the radio label: Pyridine-3-^{14}C

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	

Parameter	Values	Comments
Stability of compound at room temperature, if provided	Not provided	

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

2) Buffer solution: Buffer solutions were made with deionized water as follows:

Table 1: Description of buffer solutions.

pH	Type of buffer and final molarity	Composition
5	0.01 M acetate buffer	0.2M Acetic acid and 0.2M sodium acetate solutions in deionized water; final concentration of 0.01 mol/L.

Data obtained from p. 13 of the study report.

3) Details of light source:

Table 2: Artificial light source.

Property	Details
Nature of light source	Xenon lamp
Emission wavelength spectrum	290-1220 nm
Light intensity	*ca.* 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. 14 and Appendix 2, p. 24 of the study report.

B. EXPERIMENTAL CONDITIONS:

1) Preliminary Study: No preliminary study was conducted.

2) Experimental Conditions

Table 3: Experimental Parameters

Parameters		**Details**
Duration of the study		15 days
Test concentrations nominal: measured:		 3 µg/L Not reported
Dark controls used (Yes/No)		Yes
Replication	Dark:	2
	Irradiated:	2
Preparation of the test medium:	volume used/treatment:	Approximately 300 µL labeled and 1 mL unlabeled
	method of sterilization:	Prior to use, all glassware was autoclave-sterilized. Prepared buffer solutions were sterilized by filtration.
	co-solvent (name/concentration), if any:	Methanol, 0.26% (1300 µL/500 mL)
Test apparatus (Type/Material/Volume)		Irradiated samples: Eight glass vessels containing ca. 20 mL of test solution were capped with a quartz glass covering and placed into a rectangular thermostated block. Two additional glass vessels were filled with a chemical actinometer solution to determine quantum yield; no volatiles were trapped. Dark control samples: Eight samples were stored in Erlenmeyer flasks in a climatic chamber maintained at 22 ± 1°C.
Details of traps for volatile compounds, if any		Irradiated samples: Each photolysis vessel was equipped with inlet/outlet ports. Moistened, sterilized 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 trap organic volatiles and 0.5 M NaOH to trap CO_2. Dark controls: Volatiles were not collected. Actinometer samples: Volatiles were not collected.
If no traps were used, is the test system closed/open		Dark control systems were closed. Volatiles traps were used with irradiated systems.

Parameters	Details
Is there any indication of the test material adsorbing to the walls of the test apparatus?	Not determined (but no adsorption indicated based on material balances)
Experimental Conditions Temperature; Duration of light/darkness:	 22 ± 1°C 15 days of continuous light
Other details, if any	None

Data obtained from pp. 11, 13-14 of the study report.

3) Supplementary experiments:

Determination of quantum yield: A chemical actinometer system was used to determine the quantum yield for BAS 510 F under the test conditions (p. 13). Aliquots (ca. 20 mL) of a sterile aqueous solution containing 2 x 10^{-5} M paranitroacetophenon and 7.8 x 10^{-2}M pyridine were transferred to two sample vessels, and the samples were irradiated together with the test solutions in the Suntest CPS apparatus. At each sampling interval, aliquots were removed from the irradiated actinometer vessel and analyzed directly using LSC (p. 14). The samples were also analyzed by reverse-phase HPLC as described below for the [^{14}C]BAS 510 F test solutions.

4) Sampling:

Table 4: Sampling details

Observations	Details
Sampling intervals for the parent/transformation products	0, 1, 4, 8, and 15 days
Sampling method	Entire samples collected
Method of sampling volatile compounds, if any	Trapping solutions removed at each sampling interval after time 0.
Sampling intervals/times for: sterility check pH measurement	 Aliquots sampled at 0, 1, 4, 8, and 15 days Not conducted
Sample storage before analysis, if any	Storage intervals and conditions were not reported. Samples were "analyzed as soon as possible" and were kept in a freezer for storage (p. 15).
Other observation, if any (eg: precipitation, color change etc.)	None

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

C. ANALYTICAL METHODS:

Extraction/clean up/concentration methods, if used: Samples were analyzed directly; no extraction/clean up/concentration methods were used.

Total ^{14}C measurement: Aliquots of the irradiated and dark control test solutions were analyzed for total radioactivity by LSC. Aliquots of each trapping solution were analyzed for total radioactivity by LSC.

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 a reverse-phase HPLC using the following operating conditions: Spherisorb ODS column (250 mm x 4.0 mm; 10 µm particle size), 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 reported) and radioactivity detection (pp. 12, 15). The identity of [^{14}C]BAS 510 F was confirmed by chromatographic comparison of the HPLC retention time with that of an unlabeled reference standard of BAS 510 F.

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 parent compound: 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: Irradiated and dark control test solutions were maintained at 22 ± 1°C (p.14); however, temperature records were not provided to confirm that the temperature was maintained throughout the study. It could not be determined if the pH of each test solution was maintained throughout the course of the study since pH was not measured. It was stated that the sterility of the samples during the study period was checked by plant count technique; sterility data were not provided (p. 16).

B. MASS BALANCE: Total radiocarbon recovery was 98.8 ± 2.4% and 97.4 ± 2.3% of the applied amount in the dark and in the irradiated samples, respectively.

Table 6: Phototransformation of [^{14}C]BAS 510 F in pH 5 acetate buffer, expressed as percentage of the applied radioactivity.

Compound		Sampling times (days)				
		0	1	4	8	15
BAS 510 F	irradiated	100.0	98.3	95.2	98.9	94.4
	dark	100.0	97.7	95.0	100.2	101.0
Transformation product	irradiated	Not determined				
	dark					
Unidentified product(s), if any	irradiated					
	dark					
CO_2	irradiated	0.0	0.0	0.1	0.1	0.1
	dark	Not determined				
Volatile organics	irradiated	Not determined				
	dark					
Total % recovery	irradiated	100.0	98.3	95.3	99.0	94.5
	dark	100.0	97.7	95.0	100.2	101.0

Data obtained from Table 1, p. 19 of the study report.

C. TRANSFORMATION OF PARENT COMPOUND: At study termination, 101.0% of the applied radioactivity was [^{14}C]BAS 510 F in the dark samples.

In the irradiated samples, the concentration of the parent compound was 100.0% at day 0 and 94.4% of the applied at day 15. In both irradiated and dark control samples, the concentration of

parent compound varied during the study. There was no pattern of decline in the irradiated samples.

TRANSFORMATION PRODUCTS: In the irradiated samples, at the end of the study, 0.1% of the applied radioactivity was present as CO_2. No other transformation products were identified.

PATHWAY: A transformation pathway was not provided.

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 remained stable in pH 5 acetate buffer in the dark and the irradiated samples; therefore, a half-life could not be determined.

Half-lives/DT50

pH	First order/other half-life			DT50	DT90
	half-life	Regression equation	r^2		
5	Not determined				

The **half-life/DT50 for phototransformation** was not determined by either the study author or the reviewer. BAS 510 F appeared to be stable during the 30 days of the study.

An **environmental phototransformation half-life** was not provided by the study author since BAS 510 F was stable in the irradiated samples.

D. SUPPLEMENTARY EXPERIMENT-RESULTS: The quantum yield (ϕ_{ts}) of [^{14}C]BAS 510 F was estimated to be smaller than $\Phi_{ts} = 2.45 \times 10^{-4}$ (p. 18).

III. STUDY DEFICIENCIES: 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 §161-2.

IV. REVIEWER'S COMMENTS:

1. Representative HPLC chromatograms of day 0 and 15 sample show that no significant degradates were isolated in the test solutions (Figure 2, p. 23).

2. Daily temperature and pH measurements for the study period were not provided.

3. The same light source was used for the irradiation of the actinometer solutions and the treated solutions in each experiment (p. 18). The study author therefore concluded that it was not necessary to standardize the light intensity used during the study (p. 18).

4. The average light intensity of the xenon lamp was measured at approximately 3 mW/cm^2 (p. 14). A graphical presentation of the spectral energy distributions of the xenon lamp and natural sunlight was included in the study report (Appendix 8, p. 27). However, quantified results presented in tabular form should have also been provided.

5. The incubation temperature was 22 ± 1°C rather than the recommended 25 ± 1°C.

6. It was stated that all samples were stored frozen and analyzed as soon as possible (p. 15). Specific storage intervals and conditions for the samples were not provided. However, the test substance was stable in both irradiated and dark controls for the duration of the study.

7. 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.

8. The spectra of the test substance and chemical actinometer ranged from 0 to 0.018 AU and 0 to 0.156 AU, respectively (Appendix 2, p. 24).

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

10. Physical properties of the test substance were not provided in the study report. The water solubility value was obtained from MRID 45405216, included with this submission.

11. 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] Dulin, D. & T. Mill (1982): Development and Evaluation of Sunlight Actinometers. - Environ. Sci. Technol. 16, 815-820.

[2] Timme, G., H. Frehse & V. Laska (1986): Zur statistischen Interpretation und graphischen Darstellung des Abbauverhaltens von Pflanzenschutzmittel-Rückständen II.- Sonderdruck: Pflanzenschutz-Nachrichten Bayer 39/1986, 2, 188-204.

[3] Scharf, J. (1998): Measurement of the Seasonal Progress of the Sunlight Intensity, unpublished BASF report, Reg. Doc. BASF #98/10835.