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

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: July 2, 2003

Reviewers:

M.J. Nelson Date: 9.2.03
Maxie Jo Nelson, Chemist
Reviewer
RAB2/HED (7509C)

Date: July 16/03
Henri P. Bietlot, Chemist
Peer reviewer
FREAS, HED, PMRA

R. Loranger Date: 8/15/03
Richard A. Loranger
Branch Senior Scientist
RAB2/HED (7509C)

Date: July 25/03
Ariff Aby
Section Head
FREAS, HED, PMRA

DP Barcode: D278386

Petition No.: PP#1F06313

Citation: 45405118 Versoi, P.; Abdel-Baky, S. (2000) The Magnitude of BAS 510 F Residues in Red Raspberries and Highbush Blueberries: Final Report: Lab Project Number: 2000/5195: 63912: 99277/NY/1. Unpublished study prepared by BASF Corporation. 53 p.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 RAB2, and reflects current HED and Office of Pesticide Programs (OPP) policies. This DER was also peer reviewed by PMRA.

Executive Summary

BASF Corporation has submitted field trial data on the representative crops (highbush blueberry and red raspberry) of the berry crop group (crop group 13). Six blueberry trials were conducted in Regions 1 (1 trial; NY), 2 (2 trials; GA), 5 (2 trials; WI), and 12 (1 trial; OR) and three raspberry trials were conducted in Regions 1 (1 trial; NY) and 12 (2 trials; OR). The number and

(1)

location of field trials satisfy the US EPA's data requirements with respect to the geographic representation of residue data for the berries crop group.

For both high bush and low bush blueberries, the PMRA's data requirements have not been met. Additional trials in both zones 1A (3 trials) and 5A (3trials) are required by the PMRA to support a national registration for blueberries. The PMRA can, however, support a registration for high bush blue berries with the data provided. For raspberries, additional trials (one additional in each of zones 5 and 5B) will be required as a condition of registration in Canada.

At each test location, the 70% WG formulation of BAS 510 F was applied four times as a foliar spray at ~0.37 lb ai/A/application, (0.414 ka ai/ha/application) with a 6- to 9-day retreatment interval, for a total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha/season). Mature samples were collected at a 0-day post-treatment interval. In one raspberry field trial, additional samples were collected at 2, 4, 6, and 8 days following treatment to evaluate residue decline.

Residues of BAS 510 F in/on blueberries and raspberries were quantitated using a validated LC/MS/MS method (D9908), the data collection method for plant commodities. Storage stability data (refer to the DER for MRID 45405109) are available to support the 96 days storage interval for the samples from the submitted berry field trials.

At the applied total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha/season), the ranges of BAS 510F residues in/on treated mature samples were 0.49-2.50 ppm for blueberries and 1.39-3.31 ppm for raspberries. The residue decline data generated in New York State for raspberries indicated that residues of BAS 510 F decreased at longer post-treatment intervals with a half-life of approximately 8 days and complete dissipation anticipated by 15 days.

Residue data from the current submission are acceptable to fulfill the US EPA's crop field trial data requirements by this use pattern for the berry crop group (crop group 13). Sufficient data are available for the PMRA to support a registration on high bush blueberries and a registration on the remaining crops within this crop group, provided additional reside trials in raspberries (one trial in each of zones 5 and 5A) are carried out and submitted as a condition of registration.

GLP Compliance

Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. No GLP deviations were reported which would impact the study results or their interpretation.

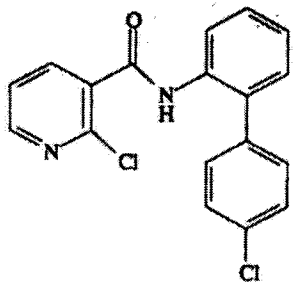
1. Materials and Methods

1.1. Test 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

Structure:



BAS 510 F

TABLE B.1.2.Trial Numbers and Geographical Locations

NAFTA Growing Region	Raspberry, red[1]				Blueberries			
	Canada		US		Canada		US	
	SUB	REQ	SUB	REQ[2]	SUB	REQ	SUB	REQ
1	1		1		1	1	1	1
1A						3		
2					2		2	2
3								
4								
5		1			2		2	2
5A						3		
5B		1						
6								
7								
7A								
8								
9								
10								
11								
12	2	3	2			1		
13								
14								
15								
16								
17								
18								
19								
20								
21								
Total	3	5	3	N/A	5	8	5	5

[1] The representative commodities for berries crop group are any one blackberry or any one raspberry and highbush blueberry.
[2] OPPTS 860.1500 Table 5 does not identify specific regions for the reduced number of raspberry field trials required as a representative crop of the berries crop group. However, trials were conducted for raspberries in Regions 1 and 12, which together account for 82% of raspberry production (OPPTS 860.1500, Table 6).

Table 1.2.2. Crop and Field Trial Information.

EPA Region	Location (County, State, Year)	Crop, Variety	Formul.	Applic. Timing	Applic. Rate (lb ai/A) [kg ai/ha]	Retreat. Intervals (days)	No. of Applics.	Applic. Method/ Applic. Volume (GPA) [l/ha]	Total Applic. Rate (lb ai/A) [kg ai/ha]	Tank Mix Adjuvants	Harvest Procedures
Blueberry, highbush											
1	Yates, NY, 1999	Highbush blueberry, Blue Ray and Blue Crop	70% WG	Verasion (coloration)	0.37-0.39 [0.414-0.437]	6-7	4	Foliar spray/ 60.3-62.6 [675-701]	1.52 [1.70]	Activate Plus (0.0025%, v:v)	Berries harvested 0 days after last application (DALA).
				early maturity							
				early maturity							
				mature berries							
2	Tift, GA, 1999	Highbush blueberry, Tift Blue	70% WG	1% ripe fruit	0.367-0.372 [0.411-0.417]	7	4	Foliar spray/ 53.3-56.8 [597-636]	1.475 [1.652]	Latron B-1956 (1pt/100gal)	Berries harvested 0 DALA.
				10% ripe fruit							
				30% ripe fruit							
				50% ripe fruit							
2	Colquitt, GA, 1999	Highbush blueberry, Climax	70% WG	40% ripe fruit	0.364-0.381 [0.408-0.427]	7	4	Foliar spray/ 54.3-57.0 [608-638]	1.498 [1.678]	Latron B-1956 (1pt/100gal)	Berries harvested 0 DALA.
				70% ripe fruit							
				95% ripe fruit							
				mature fruit							
5	Pience, WI, 1999	Highbush blueberry, Blue Chop	70% WG	0.25" diameter	0.37 [0.414]	6-7	4	Foliar spray/ 49.6-50.43 [555-565]	1.48 [1.658]	X77 (6oz/100gal)	Berries harvested 0 DALA.
				0.5" diameter							
				20% ripe							
				85% ripe							
5	Jackson, WI, 1999	Highbush blueberry, Berkley	70% WG	0.25" diameter	0.37-0.383 [0.414-0.429]	6-9	4	Foliar spray/ 50.5-51.7 [566-579]	1.513 [1.694]	X77 (6oz/100gal)	Berries harvested 0 DALA.
				0.5" diameter							

Table 1.2.2. Crop and Field Trial Information.

EPA Region	Location (County, State, Year)	Crop, Variety	Formul.	Applic. Timing	Applic. Rate (lb ai/A) [kg ai/ha]	Retreat. Intervals (days)	No. of Applics.	Applic. Method/ Applic. Volume (GPA) [l/ha]	Total Applic. Rate (lb ai/A) [kg ai/ha]	Tank Mix Adjuvants	Harvest Procedures
				70% ripe							
				80% ripe							
12	Benton, OR, 1999	Highbush blueberry, Blue Crop	70% WG	1-2 berries in cluster	0.375-0.379 [0.420-0.424]	6-8	4	Foliar spray/ 101.08-102.02 [1131-1142]	1.504 [1.684]	Latron B-1956 (3oz/100gal)	Berries harvested 0 DALA.
				10% ripe							
				50% colored							
				mature							
Raspberry, red											
1	Yates, NY, 1999 (decline study)	Red raspberry, Titau	70% WG	Immature berries	0.37-0.39 [0.414-0.437]	6	4	Foliar spray/ 59.7-63.1 [669-707]	1.51 [1.69]	Activate Plus (0.0025%, v:v)	Berries harvested 0, 2, 4, 6, and 8 DALA.
				0.5" berries							
				0.625" berries							
				mature berries							
12	Washington, OR, 1999	Red raspberry, Meeker	70% WG	Bloom - green fruit	0.368-0.387 [0.412-0.433]	7	4	Foliar spray/ 54.85-57.72 [614-646]	1.4996 [1.68]	Triton AG 98 (4oz/100gal)	Berries harvested 0 DALA.
				Bloom - green fruit							
				green, red fruit							
				mature fruit							
12	Washington, OR, 1999	Red raspberry, Tulamene	70% WG	Bloom - green fruit	0.365-0.384 [0.409-0.43]	7	4	Foliar spray/ 54.42-57.3 [609-642]	1.4885 [1.67]	Triton AG 98 (4oz/100gal)	Berries harvested 0 DALA.

Table 1.2.2. Crop and Field Trial Information.

EPA Region	Location (County, State, Year)	Crop, Variety	Formul.	Applic. Timing	Applic. Rate (lb ai/A) [kg ai/ha]	Retreat. Intervals (days)	No. of Applics.	Applic. Method/ Applic. Volume (GPA) [l/ha]	Total Applic. Rate (lb ai/A) [kg ai/ha]	Tank Mix Adjuvants	Harvest Procedures
				Bloom - green fruit							
				green, red fruit							
				mature fruit							

1.3. Post-harvest Procedures

A single untreated and duplicate treated samples of blueberries and raspberries were harvested from each field trial. Specific harvesting procedures were not described; however, each sample weighed ≥1.1 lbs. Additional samples of raspberries were collected from the NY trial (Yates County) at various time intervals for residue decline samples. Samples were bagged and stored frozen (temperature not specified) on the day of harvest. Samples were shipped frozen within 4-28 days of harvest to BASF Agricultural Products Center (Research Triangle Park, NC) for analysis.

Table 1.3.1. Summary of Storage Conditions

Matrix	RAC or Extract	Storage Temperature (°C) (Analytical Laboratory)	Duration
Blueberry, highbush	Berry	<-10	61-96 days (2.0-3.2 months)
Raspberry, red	Berry	<-10	70-83 days (2.3-2.7 months)

1.4. Analytical Methods

Samples of blueberries and raspberries were analyzed for residues of BAS 510 F using LC/MS/MS method D9908, the data collection method for plant commodities. Briefly, blueberry and raspberry samples were extracted with methanol:water (70:30, v:v) and filtered. An aliquot of the filtrate was cleaned-up using C18 solid phase extraction. Residues were eluted with dichloromethane. The eluate was evaporated and residues were redissolved in ammonium formate:formic acid for analysis by LC/MS/MS; refer to the DER for MRID 45405027 for a complete description of the quantitation procedures. The limit of detection (LOD) was 0.025 ppm, and the validated limit of quantitation (LOQ) was 0.05 ppm for the residues of BAS 510 F in/on blueberries and raspberries. The concurrent recoveries obtained are presented in Table 2.1.

2. Results

Table 2.1. Summary of Concurrent Analytical Method Validation.

Crop Matrix	Fortification Level (ppm)	Recoveries (%)	Mean Recovery
Blueberry, highbush	0.05, 5.0	94, 104	99
Raspberry, red	0.05, 5.0	91, 107	99

Table 2.2. Residue Data from Crop Field Trials in Berries (highbush blueberry and red raspberry) with BAS 510 F.

Location (County, State, Year)	Crop Variety	Commodity	Formulation	Total Rate (lbs ai/A) [kg ai/ha]	PHI (days)	BAS 510 F residues (ppm)
Blueberry, highbush						
Pience, WI, 1999	Blue Chop	Berry	70% WG	1.48 [1.66]	0	0.923, 1.395
Benton, OR, 1999	Blue Crop	Berry	70% WG	1.50 [1.68]	0	0.486, 1.194
Yates, NY, 1999	Blue Ray and Blue Crop	Berry	70% WG	1.52 [1.70]	0	1.142, 1.402
Jackson, WI, 1999	Berkley	Berry	70% WG	1.51 [1.69]	0	1.062, 1.460
Tift, GA, 1999	Tift Blue	Berry	70% WG	1.475 [1.65]	0	1.393, 1.522
Colquitt, GA, 1999	Climax	Berry	70% WG	1.498 [1.68]	0	2.171, 2.503
Raspberry, red						
Yates, NY, 1999 (decline study)	Titau	Berry	70% WG	1.51 [1.69]	0	2.064, 3.308
					2	2.110, 2.529
					4	1.562, 2.288
					6	1.139, 2.024
					8	0.956, 1.525
Washington, OR, 1999	Meeker	Berry	70% WG	1.4996[1.68]	0	1.388, 1.589
Washington, OR, 1999	Tulamene	Berry	70% WG	1.4885[1.68]	0	1.565, 2.431

Table 2.3. Summary of Residue Data from Crop Field Trials in Berries with BAS 510 F.

Commodity	Total Applic. Rate (lb ai/A) [kg ai/ha]	PHI (days)	Residue Levels (ppm)				
			Minimum	Maximum	HAFT	Mean (median)	Std. Dev.
Blueberry, highbush	1.475-1.52 [1.65-1.70]	0	0.486	2.503	2.337	1.388 (1.394)	0.532
Raspberry, red	1.4885-1.51 [1.67-1.69]	0	1.388	3.308	2.686	2.058 (1.826)	0.723

3. Discussion

3.1. Methods

Blueberries and raspberries were harvested on the day (0-day PHI) of the last of four foliar spray applications of the 70% WG formulation at ~0.37 lb ai/A/application (0.41 kg ai/ha/application), with a 6- to 9-day retreatment interval, for a total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha).

Applications were made using ground equipment in a spray volume of 49.6-102.02 gal/A (555-1144 l/ha) of water with a spray adjuvant added. In one trial (Yates County, NY), additional raspberry samples were collected at 2, 4, 6, and 8 days following treatment to evaluate residue decline. We note that the 70% BAS 510 F WG formulation used in the field trials also contained another experimental active ingredient (BAS 500 F; pyraclostrobin) as part of the tank-mix; data for the BAS 500 F active ingredient were submitted separately.

Six blueberry trials were conducted in Regions 1 (1 trial), 2 (2 trials), 5 (2 trials), and 12 (1 trial) and three raspberry trials were conducted in Regions 1 (1 trial) and 12 (2 trials). The number and location of field trials conducted for blueberries and raspberries, as the representative crops of the berry group, are in accordance with the US EPA's guidance requirements (OPPTS 860.1500, Tables 2, 5, and 6). For the PMRA, the number and location of the submitted trials (see Table B.1.2) does not match Canadian guideline requirements (Dir 98-02).

During the course of the field trials, rainfall was reported as normal in the NY and OR trials; below normal in the GA trials; and above normal in the WI trials. Temperatures were reported as above normal in the WI and one of the GA trials, and normal in all the other trials. There was no irrigation in three (NY-1, WI-1, GA-1) of the trials.

Residues of BAS 510 F in/on blueberries and raspberries were quantitated using a concurrently validated LC/MS/MS method (D9908), the data collection method for plant commodities.

Maximum storage intervals of crop samples from harvest to analysis were 96 days (3.2 months) for blueberries and 83 days (2.7 months) for raspberries. Adequate storage stability data (refer to the DER for MRID 45405109) are available in five diverse matrices to support the storage conditions and intervals of samples from the submitted berry field trials.

3.2. Results

In blueberries, residues of BAS 510 F were 0.486-2.503 ppm in/on samples harvested on the day of the last of four foliar spray applications of the 70% WG formulation at 0.364-0.39 lb ai/A/application (0.407-0.437 kg ai/ha/application), for a total rate of 1.48-1.52 lb ai/A (1.66-1.70 kg ai/ha). Apparent residues of BAS 510 F were less than the method LOQ (<0.05 ppm) in/on six samples of untreated blueberries.

In raspberries, residues of BAS 510 F were 1.388-3.308 ppm in/on samples harvested on the day of the last of four foliar spray applications of the 70% WG formulation at 0.3653-0.39 lb ai/A/application (0.408-0.437 kg ai/ha/application), for a total rate of 1.49-1.51 lb ai/A (1.67-1.69 kg ai/ha). Apparent residues of BAS 510 F were less than the method LOQ (<0.05 ppm) in/on three samples of untreated raspberries. The residue decline data generated in New York State for raspberries indicated that residues of BAS 510 F decreased in a linear fashion. The residues decline data fits the linear equation $y=-0.1815x + 2.6763$ with a correlation coefficient (r^2) of 0.9991. The equation predicts a half life of approximately 7 days and a complete dissipation of the residue by approximately 15 days.

4. Deficiencies

None for US registration. Additional field trials are required by PMRA for Canadian registration.

5. References

None