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

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: July 2, 2003

Reviewers:

William Cutchin, Chemist — Date: 8/25/03
Reviewer
SIMB/HED (7509C)

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

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

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

DP Barcode: D278386

Petition No.: 1F06313

Citation: 45405117 Haughey, D.; Abdel-Baky, S. (2000) The Magnitude of BAS 510 F Residues in Grapes: Final Report: Lab Project Number: 63892: 2000/5228: 2000110 (ND/5). Unpublished study prepared by BASF Corporation. 56 p.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 has also been peer-reviewed by PMRA/Canada.

Executive Summary

BASF Corporation has submitted field trial data on grapes. Twelve grape trials were conducted in Regions 1 (2 trials; NY), 10 (8 trials; CA), and 11 (2 trials; WA). The number and location of field trials satisfy the US EPA's data requirements with respect to the geographic representation

(1)

of residue data for grapes. For grapes, the PMRA's data requirements have not been met. Four additional trials in zone 5 are required by the PMRA for registration in Canada.

At each test location, the 70% WG formulation of BAS 510 F was applied three times as a foliar spray at ~0.37 lb ai/A/application (0.414 kg ai/ha/application), with a 13- to 14-day retreatment interval, for a total rate of 1.06-1.12 lb ai/A (1.19-1.25 kg ai/ha) using either concentrate (50-79 gal/A; 560-884 l/ha) or dilute (100-159 gal/A; 1120-1781 l/ha) spray volumes. Mature samples were collected at a 14-day posttreatment interval.

Residues of BAS 510 F in/on grapes were quantitated using concurrently 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 103 days storage interval for the samples from the submitted grape field trials.

At the applied total rate of 1.06-1.12 lb ai/A (1.19-1.25 kg ai/ha), residues of BAS 510 F were 0.270-3.104 ppm (concentrate spray) and 0.312-2.156 ppm (dilute spray) in/on mature grapes. No significant differences in the residues were observed between the concentrate and dilute spray applications. The residue decline data for grapes did not demonstrate any trends in BAS 510 F residues at longer posttreatment intervals. Maximum residues were observed at the 14-day posttreatment interval, with lower residues at both shorter and longer posttreatment intervals.

A grape processing study has been submitted separately (see DER of MRID 45405125).

Residue data from the current submission are acceptable to fulfill crop field trial data requirements for grapes in the US. The data submitted do not meet the PMRA's requirements. For Canadian registration, four additional trials to be carried out in Zone 5 are required.

GLP Compliance

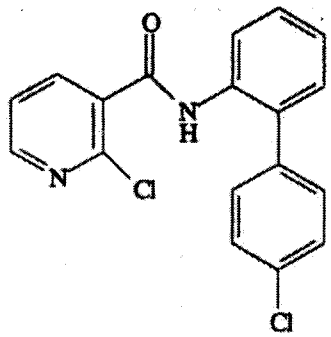
Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. No GLP deviations were reported which would impact the study results or their interpretation.

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
Chemical Structure:



1.2 Trial Locations

TABLE B.1.2. Trial Numbers and Geographical Locations

NAFTA Growing Region	Grapes			
	Canada		US	
	SUB	REQ	SUB	REQ
1			2	2
1A				
2				
3				
4				
5		4		
5A				
5B				
6				
7				
7A				
8				
9				
10			8	8
11	2	1	2	2
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
Total	2	5	12	12

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
1	Yates, NY, 1999	Grapes, Aurora	70% WG	early verasion verasion early maturity	0.37 [0.414]	14	3	Concentrate foliar spray/ 50.2-50.4 [562-564]	1.11 [124]	Activator 90 (0.25%, v:v)	Grapes harvested 14 days after last application (DALA).
1	Yates, NY, 1999	Grapes, Seyval Blau	70% WG	early verasion verasion early maturity	0.37 [0.414]	14	3	Dilute foliar spray/ 100.3-100.7 [1123-1127]	1.11 [1.24]	Activator 90 (0.25%, v:v)	Grapes harvested 14 DALA.
10	Kern, CA, 1999 (decline study)	Grapes, Emperor	70% WG	60% color 70% color 90% color	0.3641-0.3715 [0.408-0.416]	14	3	Concentrate foliar spray/ 75.01-75.86 [840-849]	1.1064 [1.24]	Latron B-1956 (4oz/100gal)	Grapes harvested 0, 7, 14, 21, and 28 DALA.
10	Tulare, CA, 1999	Grapes, Thompson Seedless	70% WG	prior to verasion past verasion 90% sugar	0.32-0.3708 [0.358-0.415]	13-14	3	Dilute foliar spray/ 152.86-154.91 [1712-1735]	1.0608 [1.19]	Latron B-1956 (4oz/100gal)	Grapes harvested 14 DALA.
10	Tulare, CA, 1999	Grapes, Flame Seedless	70% WG	30% color 60% color 90% color	0.3674-0.3751 [0.411-0.420]	13-14	3	Concentrate foliar spray/ 76.70-78.83 [859-883]	1.1113 [1.24]	Latron B-1956 (4oz/100gal)	Grapes harvested 14 DALA.
10	Colusa, CA, 1999	Grapes, Zinfindel	70% WG	fruit development fruit ripening berry development	0.368-0.372 [0.412-0.417]	14	3	Concentrate foliar spray/ 51.3-53.3 [574-597]	1.108 [1.24]	CS-7 (1pt/100gal)	Grapes harvested 14 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
10	Glenn, CA, 1999	Grapes, Zinfindel	70% WG	berry development fruit development fruit development	0.371-0.375 [0.416-0.420]	13-14	3	Dilute foliar spray/ 154-159 [1725-1781]	1.118 [1.25]	CS-7 (1pt/100gal)	Grapes harvested 14 DALA.
10	Fresno, CA, 1999	Grapes, Thompson Seedless	70% WG	early verasion late verasion sweetening berries	0.363-0.366 [0.407-0.410]	14	3	Concentrate foliar spray/ 73.5-74.17 [823-831]	1.093 [1.22]	Latron B-1956 (4oz/100gal)	Grapes harvested 14 DALA.
10	Fresno, CA, 1999	Grapes, Thompson Seedless	70% WG	early verasion late verasion sweetening berries	0.366-0.379 [0.410-0.424]	14	3	Dilute foliar spray/ 148.24-153.6 [1660-1720]	1.117 [1.25]	Latron B-1956 (4oz/100gal)	Grapes harvested 14 DALA.
10	Madera, CA, 1999	Grapes, Thompson Seedless	70% WG	early verasion late verasion sweetening berries	0.37-0.38 [0.414-0.426]	14	3	Dilute foliar spray/ 150.16-153.9 [1682-1724]	1.124 [1.26]	Latron B-1956 (4oz/100gal)	Grapes harvested 14 DALA.
11	Grant, WA, 1999	Grapes, White Riesling	70% WG	12-14 mm berries 14-16 mm berries 14 day PHI	0.369-0.372 [0.413-0.417]	14	3	Concentrate foliar spray/ 74.5 [845.6]	1.113 [1.25]	Latron B-1956 (4.5oz/100gal)	Grapes harvested 14 DALA.
11	Grant, WA, 1999	Grapes, White Riesling	70% WG	50% mature verasion 80% mature	0.37 [0.414]	14	3	Dilute foliar spray/ 149.4-152.0 [1673-1702]	1.11 [1.24]	Mor-Act (2pt/100gal)	Grapes harvested 14 DALA.

1.3. Post-harvest Procedures

A single untreated and duplicate treated samples of mature grapes were harvested from each field trial. Specific harvesting procedures were not described; however, each grape sample weighed ≥2.2 lbs. Additional samples of grapes were collected from the CA trial (Kern 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 2-30 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
Grape	Fruit	<-10	18-103 days (0.6-3.4 months)

1.4. Analytical Methods

Samples of grapes were analyzed for residues of BAS 510 F using LC/MS/MS method D9908, the data collection method for plant commodities. Briefly, samples of grapes 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 grapes. The concurrent recoveries obtained are presented below in Table 2.1.

2. Results

Table 2.1. Summary of Concurrent Analytical Method Validation.

Crop Matrix	Fortification Level (ppm)	Recoveries (%)	Mean Recovery ± SD
Grape	0.05, 1.0	88, 96, 132	105 ± 23

Table 2.2. Residue Data from Crop Field Trials in Grapes with BAS 510 F.

Location (County, State, Year)	Crop Variety	Commodity	Formulation	Total Rate (lbs ai/A) [kg ai/ha]	Spray volume	PHI (days)	BAS 510 F residues (ppm)
Yates, NY, 1999	Aurora	Fruit	70% WG	1.11[1.24]	concentrate	14	2.839, 3.104
Yates, NY, 1999	Seyval Blau	Fruit	70% WG	1.11[1.24]	dilute	14	1.995, 2.156
Kern, CA, 1999 (decline study)	Emperor	Fruit	70% WG	1.106 [1.24]	concentrate	0	0.271, 0.330
						7	0.289, 0.572
						14	0.449, 0.539
						21	0.374, 0.393
						28	0.277, 0.282
Tulare, CA, 1999	Thompson Seedless	Fruit	70% WG	1.06 [1.19]	dilute	14	0.329, 0.386
Tulare, CA, 1999	Flame Seedless	Fruit	70% WG	1.11 [1.24]	concentrate	14	0.315, 0.346
Colusa, CA, 1999	Zinfindel	Fruit	70% WG	1.11 [1.24]	concentrate	14	1.02, 1.97
Glenn, CA, 1999	Zinfindel	Fruit	70% WG	1.12 [1.25]	dilute	14	1.30, 1.46
Fresno, CA, 1999	Thompson Seedless	Fruit	70% WG	1.09 [1.22]	concentrate	14	0.639, 0.662
Fresno, CA, 1999	Thompson Seedless	Fruit	70% WG	1.12 [1.25]	dilute	14	1.09, 1.42
Madera, CA, 1999	Thompson Seedless	Fruit	70% WG	1.12 [1.26]	dilute	14	0.633, 0.668
Grant, WA, 1999	White Riesling	Fruit	70% WG	1.11[1.25]	concentrate	14	0.270, 0.304
Grant, WA, 1999	White Riesling	Fruit	70% WG	1.11[1.24]	dilute	14	0.312, 0.372

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

Commodity	Total Applic. Rate (lb ai/A)	Spray volume	PHI (days)	Residue Levels (ppm)				
				Minimum	Maximum	HAFT	Mean (median)	Std. Dev.
Grape	1.09-1.11	concentrate	14	0.270	3.10	2.97	1.15 [0.651]	[1.09]
	1.06-1.12	dilute	14	0.312	2.16	2.08	1.01 (0.881)	0.657

8

3. Discussion

3.1. Methods

Mature grapes were harvested 14 days following the last of three foliar spray applications of the 70% WG formulation at ~0.37 lb ai/A/application (0.414 ka ai/ha/application), with a 13- to 14-day retreatment interval, for a total rate of 1.06-1.12 lb ai/A (1.19-1.25 kg ai/ha). Applications were made using ground equipment in either a concentrated spray volume (50.2-78.83 gal/A of water; 562-882 l/ha of water) or a dilute spray volume (100.3-159 gal/A of water; 1123-1780 l/ha of water) with a spray adjuvant added. In one trial (Kern County, CA), additional grape samples were collected at 0, 7, 21, and 28 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.

Twelve grape trials were conducted in Regions 1 (2 trials), 10 (8 trials), and 11 (2 trials). Geographic representation of residue data for grapes is adequate. For the US EPA, the number and location of field trials conducted for grapes are in accordance with the guidance requirements (OPPTS 860.1500, Tables 1 and 5). For the PMRA, the number and location (see Table 1.2.2) of the submitted trials does not match the guideline requirements (Dir 98-02).

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

The maximum storage interval from harvest to analysis was 103 days (3.4 months) for grapes. Adequate storage stability data in five diverse matrices (refer to the DER for MRID 45405109) are available to support the storage conditions and intervals of samples from the submitted grape field trials.

3.2. Results

Residues of BAS 510 F were 0.270-3.104 ppm (concentrate spray) and 0.312-2.156 ppm (dilute spray) in/on samples grapes harvested 14 days following the last of three foliar spray applications of the 70% WG formulation at 0.32-0.379 lb ai/A/application (0.358-0.424 kg ai/ha/application), for a total rate of 1.06-1.12 lb ai/A (1.19-1.25 kg ai/ha). No significant differences in the residues were observed between the concentrate and dilute spray applications. Apparent residues of BAS 510 F were less than the method LOQ (<0.05 ppm) in/on 12 samples of untreated grapes. The residue decline data for grapes generated in California demonstrated that the residues of BAS 510 did not degrade in a predictable fashion. Maximum residues were observed at the 14-day posttreatment interval, with lower residues at both shorter and longer posttreatment intervals.

4. Deficiencies

None for US registration. For a Canadian registration, four additional trials to be carried out in Zone 5 are required.

5. References

None