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

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: July 2, 2003

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RAB2/HED (7509C)

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DP Barcode: D281841 and D297173

Petition No.: 1F06313

Citations: 45623410 Versoi, P.; Malinsky, D. (2001) The Magnitude of BAS 500 F and BAS 510 F Residues in Soybean Processed Fractions: Final Report: Lab Project Number: 2001/5002529: 66714: RCN 2001214. Unpublished study prepared by BASF Agro Research. 108 p.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

(1)

BASF Corporation has submitted data depicting the potential for concentration of residues of BAS 510 F in the processed commodities of soybean. In a single field trial conducted in NE, soybean seeds were harvested 13 days following the last of two foliar spray applications of the 70% wettable granule (WG) formulation (pyraclostrobin was included in the tank mix) at ~2.5 lb ai/A/application (2.8 kg ai/ha/application), with a 7-day retreatment interval, for a total rate of 4.88 lb ai/A (5.47 kg ai/ha). Soybean seed, bearing BAS 510 F residues of 0.2520-0.3675 ppm, were processed into hulls, meal and oil, the only currently regulated processed commodities of soybean, using simulated commercial processing procedures.

The processing data indicate that residues of BAS 510 F reduced in soybean meal (<0.05 ppm; <0.2x processing factor) and oil (0.115-0.133 ppm; 0.4x processing factor), and concentrated in hulls (0.522-0.563 ppm; 1.7-1.8x processing factors). The observed processing factor of 1.8x for soybean hulls is less than the theoretical maximum concentration factor of 11.3x (OPPTS 860.1520, Table 3, Dir 98-02 Section 10 Table 3).

Residues of BAS 510 F in/on soybean seed and its processed commodities, hulls, meal and oil, were quantitated using LC/MS/MS method D9908, the data collection method for plant commodities. Acceptable concurrent method validation data for soybean seed, hulls, meal, and oil were included in the submission.

Soybean seeds were processed within 13-17 days of collection, and samples of soybean seed (RAC) were stored frozen for up to 24 days, and samples of processed hulls, meal, and oil were stored frozen for up to 8-18 days prior to analysis. Supporting storage stability data were not submitted and are not required because samples from the subject processing study were analyzed within ~1 month of collection.

The submitted processing study is considered adequate to demonstrate the potential for concentration of BAS 510 F residues in the processed commodities of soybeans.

The submitted processing study is deemed acceptable. Under the parameters described in the study, residues of BAS 510 F did not concentrate in meal and oil processed from the RAC (soybean seed) bearing detectable residues; however, **residues concentrated by 1.7-18.x in soybean hulls.** These data suggest that no tolerances are required for residues of BAS 510 F in soybean meal and oil; however, a tolerance for soybean hulls is warranted. When determining the appropriate tolerance for soybean hulls, HED will rely on the observed concentration factor along with the highest average field trial (HAFT) residue.

Extensive field accumulation rotational crop trials on soybeans are reviewed in the DER of MRID 45623412.

GLP Compliance

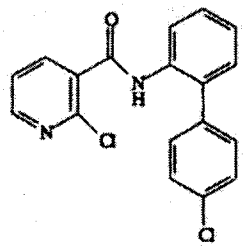
Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. No GLP deviations were reported.

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:	



1.2. Processing Information

Soybean seeds were obtained from a field trial conducted in NE during the 2001 growing season. Soybeans were harvested 13 days following the last of two foliar spray applications of the 70% WG formulation at 2.43-2.45 lb ai/A/application (2.72-2.74 kg ai/ha/application), with a 7-day retreatment interval, for a total rate of 4.88 lb ai/A (5.46 kg ai/ha). Applications were made using ground equipment in a spray volume of 19.1-19.5 gal/A (214-218 l/ha) of water with a spray adjuvant added. A single untreated sample and duplicate treated bulk-sized samples were collected; each bulk sample weighed ≥70 lbs (≥31 kg). Samples were frozen on the day of harvest and shipped frozen the next day to the Texas A&M University, Food Protein Research and Development Center (Bryan, TX) for processing.

Samples of soybean seed were processed according to simulated commercial procedures into hulls, meal, and refined oil. Briefly, the seed was oven dried at 54-71 °C to a moisture content of 7-10% and cleaned by aspiration and mechanical screening. The whole seeds were disc-milled to crack the hull and aspirated to separate the hull and kernel fractions. The kernels were heated to 71-79 °C, flaked, and expanded into collets using steam. The collets were extracted with hexane at 49-60 °C; the material remaining after solvent extraction was the meal fraction. The hexane

extracts were evaporated to yield crude oil, which was then refined with sodium hydroxide and separated into refined oil and soapstock. Adequate descriptions and material balance information were submitted for the processing procedures. The petitioner submitted adequate descriptions and material balance sheets for the processing procedures.

1.3. Post-Processing Procedures

Soybean seed samples were stored frozen (<-10 °C) at the processing facility prior to processing into hulls, meal and oil; processing was completed within 13-17 days of collection. After processing, samples of soybean seed (RAC) and processed hulls, meal, and refined oil were frozen and shipped to BASF Agro Research (Research Triangle Park, NC) for analysis (21 days after collection).

Table 1.3.1. Summary of Storage Conditions.

Matrix	RAC/Processed Commodity or Extract	Storage Temperature (°C) (Analytical Laboratory)	Duration
Soybean	Seed (RAC)	<-10	24 days (0.8 months)
	Hulls		15-16 days (0.5 months)
	Meal		8 days (0.3 months)
	Oil		18 days (0.6 months)

1.4. Analytical Methods

Samples of soybean seed and its processed fractions of hulls, meal, and oil were analyzed for residues of BAS 510 F using LC/MS/MS method D9908, the data collection method for plant commodities. Briefly, samples of soybean seed, hulls, and meal were extracted with methanol:water:2N HCl (70:25:5, v:v:v). An aliquot of the extract was subjected to liquid/liquid partitioning with saturated sodium chloride and cyclohexane. An aliquot of the cyclohexane phase was collected and subjected to further cleanup through a silica gel micro-column; residues were eluted with ethyl acetate in DCM. The eluate following silica gel cleanup was then evaporated to dryness and residues were redissolved in methanol:4 mM ammonium formate and formic acid buffer solution (8:2, v:v) for analysis by LC/MS/MS; refer to the DER for MRID 45405027 for a complete description of the quantitation procedures. The method was modified for analysis of refined oil samples. Oil samples were extracted with hexane and subjected to liquid/liquid partitioning with acetonitrile (ACN). An aliquot of the ACN phase was cleaned up by sequential C18 micro column and silica gel micro-column chromatography. 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 soybean seed, hulls, meal, and oil.

2. Results

Table 2.1. Summary of Concurrent Analytical Method Validation.			
Commodity	Fortification Level (ppm)	Recoveries (%)	Mean Recovery
Soybean, seed	0.05, 5.0	76, 78	77
Soybean, hulls	0.05, 5.0	84, 99	92
Soybean, meal	0.05, 5.0	84, 104	94
Soybean, oil	0.05, 1.0	79, 110	95

Table 2.2. Residue Data from Soybean Processing Study with BAS 510 F.					
Crop (Trial location)	RAC or Processed Commodity	Total Rate (lbs ai/A)	PHI (days)	Residues (ppm) [1]	Processing Factor
Soybean (York, NE)	Seed (RAC)	4.88	13	0.252, 0.368 (0.310)	--
	Hull			0.522, 0.563	1.7x, 1.8x
	Meal			<0.05, <0.05	<0.2x
	Oil			0.115, 0.133	0.4x

[1] Average of two samples are reported in parentheses.

Apparent residues were less than the method LOQ (<0.05 ppm) in/on one sample each of untreated soybean seed and hulls, meal, and oil processed from untreated soybean seed.

3. Discussion

3.1. Methods

Soybeans were harvested 13 days following the last of two foliar spray applications of the 70% WG formulation at ~2.5 lb ai/A/application (≈2.8 kg ai/ha/application), with a 7-day retreatment interval, for a total rate of 4.88 lb ai/A (5.47 kg ai/ha). Applications were made using ground equipment in a spray volume of 19.1-19.5 gal/A (214-219 l/ha) of water with a spray adjuvant added. We note that the 70% BAS 510 F WG formulation used in the field trial also contained another experimental active ingredient (BAS 500 F; pyraclostrobin) as part of the tank-mix; data for the BAS 500 F active ingredient are not reviewed herein.

The collected seed samples were processed into soybean hulls, meal, and refined oil, the only currently regulated processed commodities of soybean, using simulated commercial processing procedures.

Residues of BAS 510 F in/on soybean seed and its processed commodities, hulls, meal and oil, were quantitated using LC/MS/MS method D9908, (the data collection method for plant

commodities). Acceptable concurrent method validation data for soybean seed, hulls, meal, and oil were included in the submission.

Soybean seeds were processed within 13-17 days of collection, and samples of soybean seed (RAC) were stored frozen for up to 24 days, and samples of processed hulls, meal, and oil were stored frozen for up to 8-18 days prior to analysis. Supporting storage stability data are not required because samples from the soybean processing study were analyzed within ~1 month of collection.

3.2. Results

Residues of BAS 510 F were 0.252-0.368 ppm in/on treated soybean seed. The processing data indicate that residues of BAS 510 F reduce in soybean meal (<0.05 ppm; <0.2x processing factor) and oil (0.115-0.133 ppm; 0.4x processing factor), and concentrate in hulls (0.522-0.563 ppm; 1.7-1.8x processing factors). The observed processing factor of 1.8x for soybean hulls is less than the theoretical maximum concentration factor of 11.3x (OPPTS 860.1520, Table 3, Dir 98-02 Section 10 Table 3) for soybean hulls.

Extensive field accumulation rotational crop trials on soybeans are reviewed in the DER of MRID 45623412.

4. Deficiencies

None.

5. References

None.