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

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: July 2, 2003

Reviewers:

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DP Barcode: D281841 and D297173

Petition: 1F06313

Citation: 45623407 Versoi, P.; Abdel-Baky, S. (2002) The Magnitude of BAS 510 F and BAS 500 F Residues in Sunflower and Sunflower Processed Fractions: Final Report: Lab Project Number: 2001/5002552:66710: RCN 2001289. Unpublished study prepared by BASF Agro Research. 113 pages.

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 data evaluation record (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 data depicting the potential for concentration of BAS 510 F residues in the processed commodities of sunflower. In a single field trial conducted in Texas,

sunflower seeds were harvested 21 days following the last of two foliar spray applications of the 70% wettable granule (WG) formulation (containing pyraclostrobin in the tank mix) at approximately 2 lb ai/A/application (2.2 kg ai/ha), with a 6-day retreatment interval, for a total rate of 3.91 lb ai/A (4.38 kg ai/ha). Sunflower seed, bearing BAS 510 F residues of 2.40-4.95 ppm, were processed into meal and oil, the only currently regulated processed commodities of sunflower, using simulated commercial processing procedures.

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

Sunflower seeds were processed within seven days of collection, and samples of sunflower seed (RAC), meal, and oil were stored frozen for up to 26-37 days prior to analysis. Supporting storage stability data are not required because samples from the sunflower processing study were analyzed within approximately one month of collection. It is noted that acceptable storage stability data are available to support the storage conditions and intervals of the seed samples (refer to the DER for MRID 45405109) and processed meal and oil samples (refer to the DER for MRID 45405122, peanut meal and oil) from the submitted sunflower processing study.

The processing data indicate that residues of BAS 510 F reduce in sunflower meal (0.082-0.085 ppm; <0.1x processing factor) and oil (0.059-0.079 ppm; <0.1x processing factor). The theoretical maximum concentration factors are 4.5x and 2.5x (US EPA Residue Chemistry Test Guidelines, OPPTS 860.1520, Table 3; Dir 98-02, Section 10, Table 3) for sunflower meal and oil, respectively.

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 (sunflower seed) bearing residues. **No tolerances are required for residues of BAS 510 F in sunflower processed commodities.**

GLP Compliance

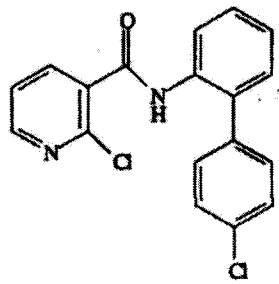
Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. No GLP deviations were reported which would impact the study results or their interpretation.

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:	



BAS 510 F

1.2. Processing Information

Sunflower seeds were obtained from a field trial conducted in Texas during the 2001 growing season. Sunflower seeds were harvested 21 days following the last of two foliar spray applications of the 70% WG formulation at 1.93-1.98 lb ai/A/application (2.16-2.22 ka ai/ha/application), with a 6-day retreatment interval, for a total rate of 3.91 lb ai/A (4.38 kg ai/ha). Applications were made using ground equipment in a spray volume of 48.2-49.4 gal/A (540-553 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 approximately 20-22 lbs (9.05-9.96 kg). Samples were shipped at ambient temperatures on the day of collection to the Texas A&M University, Food Protein Research and Development Center (Bryan, TX) for processing.

Samples of sunflower seed were processed according to simulated commercial procedures into 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 screened to remove unhulled seed. The kernel fraction was dried to a moisture of 7-10% and moisture conditioned to 12%, heated to 88-104°C, and pressed in an expeller to produce crude oil and press-cake. The press-cake was extracted with hexane (3x) and dried with warm air to remove residual solvent from the extracted meal. Hexane was removed from the

crude oil produced by the press-cake extraction by heating (73-90°C) and the resulting oil was combined with the crude oil from the expeller. Crude oil was refined and the refined oil and soapstock separated. The petitioner submitted adequate descriptions and material balance sheets for the processing procedures.

1.3. Post-Processing Procedures

Sunflower seed samples were stored frozen at the processing facility prior to processing into meal and oil; processing was initiated within seven days of collection. After processing, samples of sunflower seed (RAC) and processed meal and oil were frozen and shipped to BASF Agro Research (Research Triangle Park, NC) for analysis, 15 days after collection.

Table 1.3.1. Summary of Storage Conditions.

Matrix	Processed Commodity or Extract	Storage Temperature (°C) (Analytical Laboratory)	Duration
Sunflower	Seed (RAC)	< -10	36 days (1.2 months)
	Meal		26 days (0.9 months)
	Oil		37 days (1.2 months)

1.4. Analytical Methods

Samples of sunflower seed and its processed fractions of 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 sunflower seed 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 dichloromethane. The eluate following silica gel cleanup was then evaporated to dryness and residues were re-dissolved 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. An aliquot of the acetonitrile 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 0.050 ppm for the residues of BAS 510 F in/on sunflower seed, meal, and oil. Concurrent recoveries for a broad range in spiking levels are summarized below (Table 2.1).

2. Results

Table 2.1. Summary of Concurrent Analytical Method Validation.

Commodity	Fortification Level (ppm)	Recoveries (%)	Mean Recovery (%)
Sunflower, seed	0.050, 20.0	104, 108	106
Sunflower, meal	0.050, 20.0	95, 106	101
Sunflower, oil	0.050, 1.00	77, 88	83

Table 2.2. Residue Data from Sunflower Processing Study with BAS 510 F.

Crop (Trial location)	RAC or Processed Commodity	Total Rate (lbs ai/A) [kg ai/ha]	PHI (days)	Residues (ppm)[1]	Processing Factor
Sunflower (Hockley, TX)	Sunflower (RAC)	3.91 [4.38]	21	2.40, 4.95 (3.68)	--
	Meal			0.082, 0.085	<0.1x
	Oil			0.059, 0.079	<0.1x

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

Apparent residues were less than the method LOQ (<0.050 ppm) in/on one sample each of untreated sunflower seed and meal and oil processed from untreated sunflower seed.

3. Discussion

3.1. Methods

Sunflower seeds were harvested 21 days following the last of two foliar spray applications of the 70% WG formulation at approximately 2 lb ai/A/application (2.2 kg ai/ha/application), with a 6-day retreatment interval, for a total rate of 3.91 lb ai/A (4.38 kg ai/ha). Applications were made using ground equipment in a spray volume of 48.2-49.4 gal/A (540-553 L/ha) of water with a spray adjuvant added. It was noted 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 sunflower meal and refined oil, the only currently regulated processed commodities of sunflower, using simulated commercial processing procedures.

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

Sunflower seeds were processed within seven days of collection, and samples of sunflower seed (RAC), meal, and oil were stored frozen for up to 26-37 days prior to analysis. Supporting storage stability data are not required because samples from the sunflower processing study were

analyzed within approximately one month of collection. Acceptable storage stability data are available to support the storage conditions and intervals of the seed samples (refer to the DER for MRID 45405109) and processed meal and oil samples (refer to the DER for MRID 45405122, peanut meal and oil) from the submitted sunflower processing study.

3.2. Results

Residues of BAS 510 F were 2.40-4.95 ppm in/on treated sunflower seed. The processing data indicate that residues of BAS 510 F reduce in sunflower meal (0.082-0.085 ppm; <0.1x processing factor) and oil (0.059-0.079 ppm; <0.1x processing factor). The observed processing factor of <0.1x for sunflower meal and oil is less than the maximum theoretical concentration factors of 4.5x and 2.5x (US EPA Residue Chemistry Test Guidelines, OPPTS 860.1520, Table 3; Dir 98-02, Section 10, Table 3) for sunflower meal and oil, respectively.

4. Deficiencies

None.

5. References

None.