

US EPA ARCHIVE DOCUMENT

BAS 510 F  
Canola

Processed Food/Feed  
OPPTS 860.1520

PC Code: 128008  
MRID: 45405124

(Meal, Refined Oil, Cleaned Seed, Expeller Crude Oil, Solvent-extracted Crude Oil, Soapstock)  
PMRA a.i. code (CCH)

DACO 6.3

Submission #2001-1027, 1036, 1043



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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Date: July 23/03

DP Barcode: D278386

Petition: 1F06313

Citation: 45405124 Versoi, P.; Abdel-Baky, S. (2001) The Magnitude of BAS 510 F Residues in Canola Seed Processed Fractions: Amended Final Report: Lab Project Number: 58646: 2001/5001064: 2000111. Unpublished study prepared by BASF Agro Research. 98 pages.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 residues of BAS 510 F in the processed commodities of canola seed. In four field trials conducted in Minnesota, North Dakota, and Manitoba, canola plants were cut 20-22 days following the last of two foliar spray applications of a 70% WG formulation at approximately 1.2 lb ai/A/application, for a total rate of 2.38-2.43 lb ai/A (2.67-2.72 kg ai/ha). Canola plants were dried in the field for 0-7 days prior to collection of the seed. Canola seed, bearing BAS 510 F residues of 0.716-2.28 ppm, were processed into meal and refined oil, the currently regulated processed commodities of canola, as well as cleaned canola seed, expeller crude oil, solvent-extracted crude oil and soapstock using simulated commercial processing procedures.

The processing data indicate that residues of BAS 510 F reduce in canola meal (0.263-1.40 ppm; 0.14-0.63x; average processing factor of 0.48x) and concentrate slightly in refined oil (1.35-2.61 ppm; 0.72-1.94x; average processing factor of 1.31x). The observed average processing factor of 1.31x for refined oil is less than the theoretical maximum concentration factor of 3.0x (US EPA Residue Chemistry Test Guidelines, OPPTS 860.1520, Table 3; PMRA Residue Chemistry Guidelines, Dir 98-02, Section 10, Table 3 concentration factor for rapeseed).

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

Canola seed was processed within 57-91 days of harvest, and samples of seed, meal, and refined oil were stored frozen (at less than or equal to -10°C) for up to 167, 90, and 96 days, respectively, prior to analysis. Storage stability data are available to support the storage conditions and intervals of the RAC samples (see DER of MRID 45405109), but no storage stability data are available to support the storage conditions of the processed commodities from the submitted canola seed processing study. Although storage stability data were not provided for residues in canola oil and meal, data were submitted showing stability in peanut oil and meal for 45 days (see MRID 45405122). BAS 510 F was also quite stable under the conditions of processing as evidenced by the results of the peanut processing study (concentrations observed for meal and oil ranged from 6.0 to 11.8x; see MRID 45405122) and the present canola study. In light of those results, storage stability data are not required for canola meal and oil.

The submitted processing study is considered **acceptable** to demonstrate the potential for concentration of BAS 510 F residues in the processed commodities of canola seed.

## 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.

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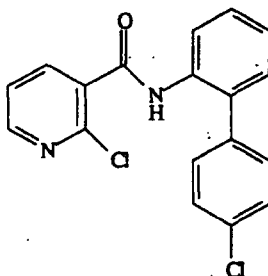
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## 1. Materials and Methods

### 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

Canola seeds were obtained from a total of four field trials conducted during the 2000 growing season. Two trials were conducted in the U.S. in Minnesota (Region 5) and North Dakota (Region 7), and two trials were conducted in Canada in Manitoba (Region 14); refer to the canola field trial DER reviewing MRID 45405116 for further details of the study. Mature canola plants were cut 20-22 days (and allowed to dry in the field 0-7 days prior to collection of canola seed) following the last of two foliar spray applications of a 70% WG formulation at approximately 1.2 lb ai/A/application (1.3 kg ai/ha/application), for a total rate of 2.38-2.43 lb ai/A (2.67-2.72 kg ai/ha). For the U.S. trials, a single bulk-sized sample was collected from each untreated and treated canola seed plot; each bulk sample weighed at least 60 lbs (27 kg). For the Canadian trials, a single bulk-sized sample was collected from each treated plot and a smaller sized sample (weighing at least 1.1 lbs, 0.50 kg) was collected from the control plots. All samples were shipped within 5-21 days of cutting to the Texas A&M University, Food Protein Research and Development Center (Bryan, TX).

Untreated and treated canola seed were dried (if necessary) in an oven (54-71°C) to a moisture content of 7-10%. Light impurities were separated by aspiration, and the remaining sample was screened to separate large and small foreign particles. Canola whole seed was flaked with a gap setting of 0.011-0.015 inches, and the flakes heated to 82-99°C for 10-15 minutes. The flakes

were then pressed in an expeller to release crude oil. Residual crude oil in the press-cake was extracted (3x) with hexane at 45-55°C. After extraction, residual hexane was removed from the press-cake (meal) using forced warm air. The miscella (crude oil/hexane mixture) was separated into crude oil and hexane, and the crude oil heated to 73-90°C. Crude oil from the expeller was combined with the miscella crude oil and then refined. Prior to refining, phosphoric acid was added to the crude oil. After refining, the oil and soapstock were separated and collected.

The processing procedure is representative of a small-scaled commercial processing of canola seed; however, because of the sample size, canola seed was processed using a batch procedure instead of a continuous procedure, as in commercial production. Material balance and process flow charts were provided.

### 1.3. Post-Processing Procedures

All samples of canola seed were stored frozen (temperature unspecified) at the field and at the processor (at less than or equal to -12°C) prior to processing. Canola seed samples were processed within 57-91 days of harvest. After processing samples were shipped frozen to BASF Agricultural Products Center (Research Triangle Park, NC) for analysis.

Matrix	RAC, Processed Commodity or Extract	Storage Temperature (°C) (Analytical Laboratory)	Duration
Canola seed	RAC	<-10	132-167 days (4.3-5.5 months)
	RAC, cleaned	<-10	83-96 days (2.7-3.2 months)
	Expeller crude oil	<-10	84-97 days (2.8-3.2 months)
	Solvent-extracted crude oil	<-10	90-97 days (3.0-3.2 months)
	Meal	<-10	82-90 days (2.7-3.0 months)
	Refined oil	<-10	88-96 days (2.9-3.2 months)
	Soapstock	<-10	83-95 days (2.7-3.1 months)

### 1.4. Analytical Methods

Samples of canola seed (RAC and cleaned RAC) and its processed fractions of crude oil (expeller and solvent-extracted), meal, refined oil, and soapstock were analyzed for residues of BAS 510 F using LC/MS/MS method D9908, the data collection method for plant commodities. Briefly, samples of canola seed, meal, and soapstock were extracted with methanol:water:2N HCl (70:25:5, v:v:v) using Polytron homogenization. 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 evaporated to dryness. Residues were re-dissolved in

ammonium formate:formic acid buffer for analysis by LC/MS/MS; refer to the DER for MRID 45405027 for a complete description of the quantitation procedures.

For oil matrices, samples were extracted by liquid/liquid partitioning using an ACN:hexane mixture. An aliquot of the extract was subjected to cleanup by C18 and silica gel solid phase extraction (SPE). For SPE cleanup, the extract was applied to a C18 SPE column; residues were eluted with dichloromethane and evaporated to dryness. Residues were re-dissolved in dichloromethane:hexane (20:80, v:v) and cleaned up further using silica gel SPE; residues were eluted from the silica gel SPE with 4% ethyl acetate in dichloromethane. The eluate was evaporated to dryness and re-dissolved in ammonium formate:formic acid buffer for analysis by LC/MS/MS.

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 canola seed and its processed commodities. Concurrent recoveries for a broad range of spiking levels are summarized below (Table 2.1).

## 2. Results

Commodity	Fortification Level (ppm)	Recoveries (%)	Mean Recovery $\pm$ SD (%)
Canola seed (RAC)	0.050	92	92
Canola seed, cleaned	1.00	82	82
Expeller crude oil	0.050-3.00	62, 66, 69, 80	69 $\pm$ 8
Solvent-extracted crude oil	0.050, 3.00	71, 75	73
Meal	0.050, 1.00	83, 106	95
Refined oil	0.050, 3.00	73, 82	78
Soapstock	0.050, 1.00	90, 108	99

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

RAC (Trial location)	Processed Commodity	Total Rate (lbs ai/A) [kg ai/ha]	PHI <sup>1</sup> (days)	Residues (ppm)	Processing Factor
Canola seed (McHenry, ND, U.S.)	RAC	2.41 [2.78]	21 (5)	0.716	--
	RAC cleaned			0.557	0.78x
	Crude oil (expeller)			1.11	1.55x
	Crude oil (solvent-extracted)			1.04	1.46x
	Meal			0.371	0.52x
	Refined oil			1.39	1.94x
	Soapstock			0.502	0.70x
Canola seed (Whitewater, MB, Canada)	RAC	2.38 [2.67]	21 (7)	1.87	--
	RAC cleaned			0.997	0.53x
	Crude oil (expeller)			1.51	0.81x
	Crude oil (solvent-extracted)			1.26	0.67x
	Meal			0.263	0.14x
	Refined oil			1.35	0.72x
	Soapstock			0.588	0.31x
Canola seed (Minto, MB, Canada)	RAC	2.43 [2.72]	20 (5)	2.28	--
	RAC cleaned			2.25	0.99x
	Crude oil (expeller)			2.74	1.20x
	Crude oil (solvent-extracted)			2.49	1.09x
	Meal			1.40	0.61x
	Refined oil			2.61	1.15x
	Soapstock			1.77	0.78x
Canola seed (Otertail, MN, U.S.)	RAC	2.40 [2.67]	22 (0)	1.76	--
	RAC cleaned			1.57	0.89x
	Crude oil (expeller)			2.00	1.13x
	Crude oil (solvent-extracted)			1.99	1.13x
	Meal			1.11	0.63x
	Refined oil			2.53	1.44x
	Soapstock			1.13	0.64x

<sup>1</sup> The first number represents the number of days after the last application when canola was cut; the number in parentheses represents the number of days plants were dried in the field prior to collection of canola seed.

Apparent residues were less than the method LOQ (<0.050 ppm) in/on four samples of untreated canola seed (RAC) and two samples each of untreated cleaned canola seed, expeller crude oil, solvent-extracted crude oil, meal, refined oil, and soapstock processed from untreated canola seed.

### 3. Discussion

#### 3.1. Methods

Canola plants were cut 20-22 days following the last of two foliar spray applications of a 70% WG formulation at approximately 1.2 lb ai/A/application (1.34 kg ai/ha/application), with a 5- to 6-day retreatment interval, for a total rate of 2.38-2.43 lb ai/A (2.67-2.72 kg ai/ha). Applications were made using ground equipment in 11.9-21.6 gal/A (133-242 L/ha) of water with a spray adjuvant added. Cut plants were allowed to dry in the field 0-7 days prior to collection of canola seed.

The collected samples were processed into meal and refined oil, the currently regulated processed commodities of canola, as well as cleaned canola seed, expeller crude oil, solvent-extracted crude oil, and soapstock using simulated commercial processing procedures.

Residues of BAS 510 F in/on canola seed (RAC) and its processed commodities (meal, refined oil, cleaned canola seed, expeller crude oil, solvent-extracted crude oil, and soapstock) were quantitated using LC/MS/MS method D9908, the data collection method for plant commodities.

Canola seed was processed within 57-91 days of harvest, and samples of seed, meal, and refined oil were stored frozen (at less than or equal to -10°C) for up to 167, 90, and 96 days, respectively, prior to analysis. Adequate storage stability data are available to support the storage conditions and intervals of the RAC samples (refer to the DER for MRID 45405109), but no storage stability data are available to support the storage conditions of the processed canola seed commodities (meal and refined oil) from the submitted canola seed processing study. However, data on the processed commodities of peanuts to address storage stability are discussed below.

#### 3.2. Results

Residues of BAS 510 F were 0.716-2.28 ppm in/on treated canola seed. The processing data indicate that residues of BAS 510 F reduce in canola meal (0.263-1.40 ppm; 0.14-0.63x; average processing factor of 0.48x) and concentrate slightly in refined oil (1.35-2.61 ppm; 0.72-1.94x; average processing factor of 1.31x).

The observed average processing factor of 1.31x for refined oil is less than the theoretical maximum concentration factor of 3.0x (OPPTS 860.1520, Table 3). HED will consider the



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observed processing factor in determining the need for a tolerance on refined canola oil. The data indicate that residues of BAS 510 F do not concentrate in canola meal processed from canola seed bearing quantifiable BAS 510 F residues. HED will not require a tolerance for canola meal.

Although storage stability data were not provided for residues in canola oil and meal, data were submitted showing stability in peanut oil and meal for 45 days (MRID 45405122). BAS 510 F was also quite stable under the conditions of processing as evidenced by the results of the peanut processing study (concentrations observed for meal and oil ranged from 6.0 to 11.8x; MRID 45405122) and the present canola study. In light of those results, storage stability data are not required for canola meal and oil.

The submitted processing study is considered **acceptable** to demonstrate the potential for concentration of BAS 510 F residues in the processed commodities of canola seed.

#### 4. Deficiencies

None

#### 5. References

None