

BAS 510 F
Mint Oil
PMRA a.i. Code (CCH)

Processed Food/Feed
OPPTS 860.1520
DACO 7.4.5

PC Code: 128008
MRID: 45623408
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

Reviewers:

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DP Barcode: D281841 and D297173

Petition: 1F06313

Citation: 45623408 Versoi, P.; Abdel-Baky, S. (2001) The Magnitude of BAS 510 F and BAS 500 F Residues in Mint and Mint Processed Fractions: Final Report: Lab Project Number: 66700: 2001/5002467: F200107. Unpublished study prepared by BASF Agro Research. 95 pages.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 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 mint. In a single field trial conducted in Oregon, mint tops were harvested 7 days following the last of four foliar spray applications of the 70% wettable granule (WG) formulation at 2.0-2.1 lb ai/A/application (2.2-2.4 kg ai/ha/application), with a 6- to 7-day retreatment interval, for a total rate of 8.2 lb ai/A (9.2 kg ai/ha). Mint top samples were cut for hay and dried at the test facility for 2 days prior to collection. Mint hay, bearing BAS 510 F residues of 81.0-103 ppm, were processed into oil, the only currently regulated processed commodity of mint, using simulated commercial processing procedures.

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

Mint hay was processed on the day of collection, and samples of mint hay and mint oil were stored frozen ($\leq -10^{\circ}\text{C}$) for up to 63 and 56 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 12 months frozen storage) and processed oil samples (refer to the DER for MRID 45405122, peanut oil 45 days frozen storage) from the submitted mint processing study.

The processing data indicate that residues of BAS 510 F reduce in oil (0.077-0.190 ppm; processing factor of $<0.01x$). The observed processing factor of $<0.01x$ for mint oil is less than the theoretical maximum concentration factor of 333x (US EPA Residue Chemistry Test Guidelines, OPPTS 860.1520, Table 2; PMRA Dir 98-02, Section 10) for mint oil.

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

The submitted processing study is deemed acceptable. Under the parameters described in the study, residues of BAS 510 F did not concentrate in oil processed from mint hay bearing residues. No tolerance is required for residues of BAS 510 F in mint oil.

GLP Compliance

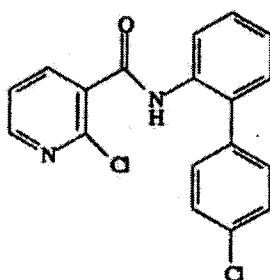
Signed and dated GLP, Quality Assurance, and Data Confidentiality statements were provided. No GLP deviations were reported.

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
Chemical Structure:



BAS 510 F

1.2. Processing Information

Mint hay was obtained from a field trial conducted in Oregon during the 2001 growing season. Mint tops were harvested 7 days following the last of four foliar spray applications of the 70% WG formulation at 2.0-2.1 lb ai/A/application (2.2-2.4 kg ai/ha/application), with a 6- to 7-day retreatment interval, for a total rate of 8.2 lb ai/A (9.2 kg ai/ha). Applications were made using ground equipment in a spray volume of 19.9-21.0 gal/A (22.3-23.5 kg ai/ha) of water with a spray adjuvant added. A single untreated sample and duplicate treated bulk-sized samples were cut for hay and dried passively at the test facility for 2 days prior to collection; each bulk sample weighed ~24 lbs (~11 kg). Samples were shipped at ambient temperatures on the day of collection to the Madras Experiment Station, Oregon State University (Madras, OR) for processing.

Samples of mint hay were processed according to simulated commercial procedures into mint oil. Briefly, the mint hay was steam cooked for 30-40 minutes in a kettle of a peppermint still. The steam and mint oil were condensed at 49° C. Following distillation, the mint oil was separated from the water fraction by titration. The collected mint oil was poured into glass jars. Process flow charts were provided.

1.3. Post-Processing Procedures

Processing of mint hay samples was initiated on the day of collection. After processing, samples of mint hay and processed mint oil were frozen and shipped back to the field facility. The frozen samples were then shipped to BASF Agro Research (Research Triangle Park, NC) for analysis (10 days after collection).

Table 1.3.1. Summary of Storage Conditions.

Matrix	Processed Commodity or Extract	Storage Temperature (°C) (Analytical Laboratory)	Duration
Mint	Hay	<-10	63 days (2.1 months)
	Oil		56 days (1.8 months)

1.4. Analytical Methods

Samples of mint hay and its processed fraction of mint 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 mint hay 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 mint oil samples. Oil samples were diluted with water, purified by steam distillation and cooled. Residues were then partitioned into cyclohexane and purified by silica gel column chromatography as for hay samples. 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 mint hay and oil.

2. Results

Table 2.1. Summary of Concurrent Analytical Method Validation.

Commodity	Fortification Level (ppm)	Recoveries (%)	Mean Recovery (%)
Mint, hay	0.050, 100.0	80, 86	83
Mint, oil	0.050, 0.20	96, 102	99

Table 2.2. Residue Data from Mint Processing Study with BAS 510 F.					
RAC (Trial location)	Processed Commodity	Total Rate (lbs ai/A)	PHI (days)	Residues (ppm) ¹	Processing Factor
Mint (Jefferson, OR)	Mint, hay	8.20	7	81.0, 103.4 (92.2)	—
	Oil			0.077, 0.077 (0.077) 0.182, 0.190 (0.190)	<0.01x

¹ Average of duplicate analyses of a single sample are reported in parentheses.

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

3. Discussion

3.1. Methods

Mint tops were harvested 7 days following the last of four foliar spray applications of the 70% WG formulation at 2.0-2.1 lb ai/A/application (2.2-2.4 kg ai/ha/application), with a 6- to 7-day retreatment interval, for a total rate of 8.2 lb ai/A (9.2 kg ai/ha). Applications were made using ground equipment in a spray volume of 19.9-21.0 gal/A (222.9-235.2 L/ha) of water with a spray adjuvant added. Mint top samples were cut for hay and dried passively at the test facility for 2 days prior to collection. 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 hay samples were processed into mint oil, the only currently regulated processed commodity of mint, using simulated commercial processing procedures.

Residues of BAS 510 F in/on mint hay and its processed commodity, mint oil, were quantitated using LC/MS/MS method D9908, the data collection method for plant commodities. Adequate concurrent method validation data for mint hay and oil were included in the submission.

Mint hay was processed on the day of collection, and samples of mint hay and oil were stored frozen ($\leq -10^{\circ}\text{C}$) for up to 63 and 56 days, respectively, prior to analysis. Adequate storage stability data are available to support the storage conditions and intervals of the hay samples (refer to the DER for MRID 45405109) and processed oil samples (refer to the DER for MRID 45405122, peanut oil) from the submitted mint processing study.

3.2. Results

Residues of BAS 510 F were 81.0-103 ppm in/on treated mint hay. The processing data indicate that residues of BAS 510 F reduce in oil (0.077-0.190 ppm; <0.01x processing factor). The observed processing factor of <0.01x for mint oil is less than the theoretical maximum

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concentration factor of 333x (US EPA Residue Chemistry Test Guidelines, OPPTS 860.1520, Table 2; PMRA Dir 98-02, Section 10) for mint oil.

It was noted that mint hay is not the RAC of mint [mint tops (leaves and stems) are the RAC]. Oil may be processed from either mint hay or mint tops. Based on the submitted processing data and theoretical maximum concentration factor, residues of BAS 510 F in processed oil would not be expected to concentrate from fresh mint tops (RAC).

The submitted processing study is considered acceptable.

4. Deficiencies

None.

5. References

None.