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OPP OFFICIAL RECORDS
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361



Boscalid/7969-197/PC Code 128008/BASF Corporation/7969
DACO 7.3/OPPTS 860.1380/OECD IIA 6.1.1 and IIIA 8.1.1
Storage Stability - Tomato Processed Fractions

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Peer Reviewer William Drew, Chemist, RAB2 *W. Drew* Date: 10/7/2005

This DER was originally prepared under contract by Dynamac Corporation (1910 Sedwick Rd., Building 100, Suite B; Durham, NC 27713; submitted 1/14/2005). The DER has been reviewed by the HED and revised to reflect current OPP policies. {OPPTS 860.1380}

STUDY REPORTS:

46160103 Jordan, J. (2003) BAS 510 F Frozen Storage Stability in Treated Sample of Tomato Paste: Project Number: 2003/5000538. Unpublished study prepared by BASF Corporation. 15 p.

EXECUTIVE SUMMARY:

To generate storage stability data on boscalid residues in tomato paste, paste samples generated and analyzed in conjunction with an earlier tomato processing study (45405126.der, W. Cutchin, 7/2/03) were retained in storage at $\leq -20^{\circ}\text{C}$ for up to 38.4 months. Duplicate stored samples were then reanalyzed after 38.4 months of storage along with a freshly fortified sample.

The LC/MS/MS method (BASF Method Number D9908) used to determine residues of boscalid in/on tomato paste is adequate for data collection. For this method, residues are extracted with methanol:water:2 N HCl, concentrated, cleaned up by partitioning into cyclohexane and silica gel solid phase extraction, and analyzed by LC/MS/MS. The limit of quantitation (LOQ) was 0.05 ppm; the LOD was not reported.

The storage stability data are adequate and indicate that boscalid is relatively stable in frozen ($\leq -20^{\circ}\text{C}$) tomato paste for at least 38 months, declining by $\sim 20\%$ over 3 years. Samples from the tomato processing study (45405126.der, W. Cutchin, 7/2/03) were originally stored frozen for a maximum of 154 days (5 months) prior to the original analysis; therefore, correction of residues for decline during storage is not required.

STUDY/WAIVER ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and parameters used in the study, the storage stability data are classified as scientifically acceptable. The acceptability of this study for regulatory purposes is addressed in the associated U.S. EPA Residue Chemistry Summary Document (D322235).

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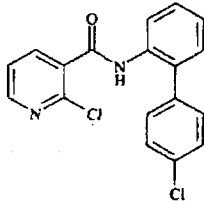
COMPLIANCE:

Signed and dated GLP, quality assurance, and data confidentiality statements were provided. No deviations from regulatory requirements were noted that would impact the study results or their interpretation

A. BACKGROUND INFORMATION

Boscalid is an anilide fungicide that inhibits mitochondrial respiration, thereby inhibiting spore germination, germ tube elongation, mycelial growth, and sporulation of pathogenic fungi on the leaf surface. Permanent tolerances have been established in 40 CFR §180.589 for residues of boscalid in/on numerous plant commodities, ranging from 0.05 ppm in/on peanuts and tuberous and corm vegetables (subgroup 1C) to 35 ppm in/on dried hop cones. Separate tolerances have also been established for indirect or inadvertent residues of boscalid in rotational crops, ranging from 0.05 ppm in several commodities to 8.0 ppm in grass forage, fodder, and hay (group 17). Tolerances for the combined residues of boscalid and its glucuronic acid conjugate are also established on animal commodities, ranging from 0.02 ppm in eggs to 0.35 ppm in meat byproducts of cattle, goats, horses, and sheep. The current storage stability study was submitted to satisfy a deficiency cited in a previous boscalid petition (DP Barcode D278385, M. Nelson, 8/15/03).

The nomenclature and physicochemical properties of boscalid are presented below in Tables A.1. and A.2.

TABLE A.1. Nomenclature of Boscalid	
Compound	
Common name	Boscalid
Company experimental names	BAS 510 F
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 #	188425-85-6
End-use products/EP	70% WDG (Endura™ fungicide; EPA Reg. No. 7969-197)



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Parameter	Value	Reference
Melting point	143.4-143.6°C (TGAI); 142.8-143.8°C (PAI)	D278385, M. Nelson, 8/15/03
pH (23°C)	5.5 (1% solution)	
Density	1.394g/cm ³ (TGAI); 1.381g/cm ³ (PAI)	
Water solubility (20°C)	4.64 mg/L (PAI)	
Solvent solubility (g/100 mL at 20°C)	PAI: 16-20 in acetone; 4-5 in acetonitrile; 4-5 in methanol; 6.7-8 in ethylacetate; 20-25 in dichloromethane; 2-2.5 in toluene; <1 in 1-octanol	
Vapour pressure at 20°C	7 x 10 ⁻⁹ hPa (PAI)	
Dissociation constant (pK _a)	Does not dissociate in water.	
Octanol/water partition coefficient at 21°C Log(K _{ow})	2.96 (PAI)	
UV/visible absorption spectrum	Not available	

B. EXPERIMENTAL DESIGN

B.1. Sample Preparation

A treated sample of tomato paste from the earlier tomato processing study (45405126.der, W. Cutchin, 7/2/03) was retained in frozen (<20° C) storage for up to 38.4 months. The paste sample, which had quantifiable residues of boscalid, was originally analyzed once after 3.4 months of frozen storage and then again in duplicate after 4.5 months of storage. For the analysis after 38 months of storage, the treated paste sample was analyzed in duplicate, along with a control sample, and a single control sample freshly fortified with boscalid at 1.0 ppm.

B.2. Analytical Methodology

Residues of boscalid were determined using an LC/MS/MS method (BASF Method Number D9908). Method D9908 was validated in conjunction with a previous boscalid petition (DP Barcode D278385, M. Nelson, 8/15/03) and deemed acceptable for data collection. A brief description of the method follows.

Residues are extracted with methanol:water:2 N HCl (70:25:5, v/v/v), concentrated, cleaned up by liquid/liquid partitioning into cyclohexane. Residues are then concentrated and cleaned up using silica gel solid phase extraction. Residues are analyzed by LC/MS/MS using the positive ionization mode monitoring ion transitions from *m/z* 343 to 307. Quantitation is obtained using an external calibration curve of boscalid standards. The limit of quantitation (LOQ) is 0.06 ppm for residues of boscalid in/on plant matrices; the LOD was not reported.



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C. RESULTS AND DISCUSSION

The available data indicate that Method D9908 is adequate for determining residues of boscalid in/on tomato paste. In the earlier analyses in the original processing study, the average method validation recoveries were 82% with a standard deviation of 5% from 4 tomato paste samples fortified with boscalid at 0.05-2.0 ppm (Table C.1.1). For the current analysis, the recovery of boscalid was 81% from a control paste sample fortified at 1.0 ppm.

Uncorrected residues of boscalid were 1.68 ppm in treated tomato paste at the 3.5 month analysis and averaged 1.59 ppm at the 4.5 month analysis (Table C.1.2). After 38.4 months of frozen storage, boscalid residues in the same sample averaged 1.30 ppm. As average concurrent recoveries were similar at each analysis (81-82%), residues were not corrected for concurrent recoveries when determining residue decline. Based on residues from the initial analysis, residues in tomato paste decline by approximately 20% over 3 years of frozen storage, or approximately 6% per year. As paste samples were initially analyzed within 5 months, these data indicate that residues in paste do not need to be corrected for decline during storage.

TABLE C.1.1 Summary of Concurrent Recoveries from Tomato Paste for HPLC/MS/MS Method D9908.

Analyte	Matrix	Spiking Level (mg/kg)	Sample size	Recoveries (%)	Mean Recovery ± SD
Boscalid	Tomato Paste	0.05-2.0	4	77-87 ¹	82 ± 5 ¹
		1.0	1	81	NA

¹ Recovery data from a previously reviewed tomato processing study (45405126.der, W. Cutchin, 7/2/03).
 NA = not applicable

TABLE C.1.2 Stability of Boscalid in/on Tomato Fruit Following Storage at ≤-20° C.

Matrix	Spike level (mg/kg) ¹	Storage Interval (months)	Concurrent Fresh Recovery (%)	Residues in Stored Samples (ppm)	Stored Recoveries (%) ²
Tomato Paste	NA	3.4	77-87 (82) ¹	1.68	NA
	NA	4.5		1.62, 1.48 (1.59)	95
	NA	38.4	81	1.31, 1.28 (1.30)	77

¹ Stored samples were treated samples from the tomato processing study (45405126.der, W. Cutchin, 7/2/03).
² Recoveries at 4.5 and 38.4 months are based on residues originally determined after 3.4 months of storage.
 NA = not applicable

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D. CONCLUSION

The storage stability data are adequate and indicate that boscalid is relatively stable in frozen ($\leq -20^{\circ}\text{C}$) tomato paste for at least 38 months, declining by ~20% over 3 years. As samples from the processing study (45405126.der, W. Cutchin, 7/2/03) were stored frozen for a maximum of 154 days (5 months), correction of residue decline during storage is not required.

E. REFERENCES

D278386, PP#0F06313, The Magnitude of BAS 510 F Residues in Tomato Processed Fractions, W. Cutchin, 7/2/03.

D278385, PP#0F06313, BAS 510 F (Common Name: Boscalid), New Fungicide Active Ingredient. Residue Chemistry Summary Document, M. Nelson, 8/15/03.

F. DOCUMENT TRACKING

Petition Number: 1F6313
DP Barcode: D302789
PC Code: 128008

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