

US EPA ARCHIVE DOCUMENT



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 - Plant Commodities

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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.

STUDY REPORTS:

46351404 Funk, H., Mackenroth, C. (2001) Investigation of the Stability of BAS 510 F in Plant Matrices Under Normal Storage Conditions: Project Number: 41851, 2001/1015028.

Unpublished study prepared by BASF Aktiengesellschaft. 46 p. {OPPTS 860.1380}

46351405 Sasturain, J. (2000) Technical Procedure: Method for the Determination of BAS 480F, BAS 490 F, BAS 500 F, and BAS 510 F in Plant Matrices: Final Report. Project Number: 445/0, 2000/1014864. Unpublished study prepared by BASF Ag Research Station (BASF Aktieng)

16 p. {OPPTS 860.1340}

EXECUTIVE SUMMARY:

The current submission is the final report of a storage stability study, including data from storage intervals of 18 and 24 months. The data from a 12-month interim report of this study were previously reviewed (45405109.der, M. Nelson, 7/2/03). In the current submission, duplicate stored samples of wheat (forage, grain, and straw), rape seed, sugar beet roots, cabbage, peach, and pea fortified with boscalid (0.5 ppm) were analyzed after 18 and 24 months of frozen storage, along with duplicate freshly fortified samples.

The LC/MS/MS method (BASF Method 445/0) used to determine residues of boscalid in/on plant matrices is nearly identical to the previously validated BASF Method D9908 and is adequate for data collection. For this method, residues are extracted with methanol:water:2 N HCl, concentrated, cleaned up using liquid/liquid partitioning into cyclohexane and analyzed by LC/MS/MS. The limit of quantitation (LOQ) was 0.05 ppm in/on each plant matrix; the LOD was not reported.

The storage stability data are adequate and indicate that boscalid is stable under frozen ($\leq -20^{\circ}\text{C}$) conditions in diverse representative crop matrices for at least 24 months. Average corrected recoveries were 76-108% from all frozen plant matrices after 24 months of storage. Samples from the crop field trials, processing studies, and rotational crop studies were stored frozen for a maximum of 12.5 months.



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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 forthcoming U.S. EPA Residue Chemistry Summary Document (D322235).

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 (BAS 510 F) 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 and rotational crop commodities, ranging from 0.05 ppm in/on several commodities to 35 ppm in/on dried hop cones. Tolerances for the combined residues of boscalid and its glucuronic acid conjugate are also established for animal commodities, ranging from 0.02 ppm in eggs to 0.35 ppm in meat byproducts of cattle, goats, horses, and sheep. The current submission is the final 24-month report of a storage stability study; the data from an 12-month interim report of the study was previously reviewed (45405109.der, M. Nelson, 7/2/03).

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

Untreated samples of representative crops (cabbage, oil rape seed, pea, peach, sugar beet root, wheat forage, grain and straw) were fortified with boscalid at 0.5 ppm. Prior to fortification, wheat forage samples were ground, seed and grain samples were sieved and mixed, straw was milled, and sugar beets, cabbage heads, and fruits were cut. Fortified and unfortified samples were stored in polyethylene containers at approximately -20° C in the dark for up to 720 days (24 months).

B.2. Analytical Methodology

Samples were analyzed for residues of boscalid using LC/MS/MS method 445/0 (MRID 46351405). Method 445/0 is nearly identical to BASF Method D9908, which 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 redissolved in methanol:water (80:20, v/v). Residues are analyzed by LC/MS/MS using the positive ionization mode monitoring ion transitions from *m/z* 343 to 271. Quantitation is obtained using an external calibration curve of boscalid standards. The LOQ is 0.05 ppm for residues of boscalid in/on each plant matrix; the LOD was not reported.



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C. RESULTS AND DISCUSSION

The available data indicate that Method 445/0 is adequate for determining residues of boscalid in/on plant matrices. Duplicate freshly fortified samples of each matrix were analyzed for concurrent recoveries. Including all sampling intervals over the course of the study, concurrent method validation recoveries were relatively consist across all commodities fortified with boscalid at 0.5 ppm (Table C.1). Average concurrent recoveries were 87-95% with standard deviations of 7-12%. The overall average recovery was 93% with a standard deviation of 9% from all samples.

Residues of boscalid were stable at $\leq -20^{\circ}$ C for at least 24 months in all plant matrices tested. Average corrected recoveries were 76-108% from all frozen plant matrix samples after 24 months of storage. Using the corrected recoveries over the 24-month study, dissipation curves were generated for each sample matrix. The only notable declines were observed in wheat forage (8% decline) and wheat straw (11% decline). Residues in the remaining matrices showed either a slight increase in residues or a decline of <4%. Samples generated in the crop field trials, processing studies, and rotational crop studies were stored frozen for up to 12.5 months.

Commodity	Storage Interval (days)	Concurrent Spike Recovery (%) ²	Apparent Recovery in Stored Sample (%) ³	Corrected Recovery in Stored Sample (%) ⁴
Beet, sugar, root	0	84.6, 86.8 (85.7)	84, 88	--
	33	95.6, 97.0 (96.3)	90, 94	93, 97 (95)
	96	91.5, 96.2 (93.9)	87, 93	93, 99 (96)
	182	73.2, 96.8 (85.0)	95, 100	112, 117 (115)
	356	94.0, 95.7 (94.9)	98, 99	103, 104 (104)
	566	96.5, 104.1 (100.3)	92, 92	90, 92 (91)
	720	69.8, 73.8 (71.8)	82, 74	114, 102 (108)
Cabbage, white	0	81.7, 101.6 (91.7)	90, 96	--
	33	88.6, 97.0 (92.8)	86, 87	92, 94 (93)
	96	94.0, 120.0 (107.0)	111, 112	104, 104 (104)
	182	85.6, 93.3 (89.5)	93, 95	104, 106 (105)
	356	94.0, 95.0 (94.5)	93, 95	98, 100 (99)
	566	100.9, 94.5 (97.7)	90, 96	92, 98 (95)
	720	83.0	80, 84	96, 100 (98)
Oilrape seed (canola seed)	0	77.1, 110.3 (93.7)	73, 78	--
	33	107.7, 116.0 (111.9)	90, 91	80, 82 (81)
	96	89.1, 90.1 (89.6)	79, 82	88, 91 (90)
	182	94.4, 99.2 (96.8)	85, 87	88, 89 (89)
	356	90.4, 92.7 (91.6)	84, 84	92, 92 (92)
	566	89.8, 93.3 (91.6)	86, 84	94, 92 (93)
	720	77.5, 81.5 (79.5)	60, 60	76, 76 (76)

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Table C.1 Storage Stability of Boscalid in Agricultural Commodities Following Fortification at 0.5 ppm and Storage at $\leq -20^{\circ}$ C for up to 24 Months.¹

Commodity	Storage Interval (days)	Concurrent Spike Recovery (%) ²	Apparent Recovery in Stored Sample (%) ³	Corrected Recovery in Stored Sample (%) ⁴
Pea	0	89.1, 89.2 (89.2)	87, 88	--
	33	92.2, 101.4 (96.8)	94, 96	97, 99 (98)
	96	79.4, 79.4 (79.4)	86, 88	108, 111 (110)
	182	95.4	94, 98	99, 103 (101)
	356	97.5, 101.5 (99.5)	100, 102	101, 102 (102)
	566	94.5, 94.5 (94.5)	80, 86	84, 90 (87)
	720	66.1, 85.6 (75.9)	78, 80	102, 104 (103)
Peach	0	82.8, 87.3 (89.1)	89, 92	--
	33	95.1, 100.2 (97.7)	83, 95	85, 97 (91)
	96	86.9, 91.8 (89.4)	89, 90	99, 101 (100)
	182	94.8, 100.0 (97.4)	97	99
	356	92.8, 94.0 (93.4)	91, 101	97, 108 (103)
	566	83.8, 98.5 (91.2)	82, 86	90, 94 (92)
	720	80.2, 78.5 (79.4)	84, 76	106, 96 (101)
Wheat, forage	0	87.1, 88.5 (87.8)	84, 98	--
	33	82.0, 95.8 (88.9)	80, 80	89, 89 (89)
	96	84.6, 93.5 (89.1)	86, 89	96, 100 (98)
	182	98.6, 101.4 (100.0)	91, 96	91, 96 (94)
	356	74.8, 91.0 (82.9)	88, 90	106, 108 (107)
	566	89.6, 90.6 (90.1)	78, 78	86, 86 (86)
	720	69.0, 83.2 (76.1)	64, 70	84, 92 (88)
Wheat, grain	0	88.7, 96.1 (92.4)	87, 91	--
	33	103.0, 106.7 (104.9)	84, 85	80, 81 (81)
	96	93.8, 97.4 (95.6)	86	90
	182	98.0, 104.2 (101.1)	91, 93	90, 92 (91)
	356	98.2, 99.4 (98.8)	90, 94	92, 95 (94)
	566	99.2, 87.9 (93.6)	88, 90	94, 96 (95)
	720	75.1, 76.0 (75.6)	82, 78	108, 104 (106)
Wheat, straw	0	74.6, 87.4 (81.0)	79, 81	--
	33	68.4, 89.3 (78.9)	82, 84	104, 107 (106)
	96	84.6, 86.1 (85.4)	82, 89	96, 104 (100)
	182	100.9, 106.1 (103.5)	95, 96	91, 93 (92)
	356	92.5, 93.7 (93.1)	92, 92	99, 99 (99)
	566	86.6, 89.4 (88.0)	82, 84	92, 96 (94)
	720	74.1, 85.8 (80.0)	64, 74	80, 92 (86)

¹ Data from 0 to 356 days were reported previously in the interim report of storage stability data (45405109.der, M. Nelson, 7/2/03).

² Average fresh fortification recoveries are presented in parentheses.

³ Residues were reported by the petitioner in mg/kg; % recoveries were calculated by the reviewer.

⁴ Corrected residues were reported by the petitioner in mg/kg; % recoveries were calculated by the reviewer, with averages in parentheses.

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D. CONCLUSION

The storage stability data are adequate and indicate that boscalid is stable under frozen ($\leq -20^{\circ}\text{C}$) conditions in diverse representative crop matrices for at least 24 months. Samples from the crop field trials, processing studies, and rotational crop studies were stored frozen for a maximum of 12.5 months. Therefore, correction of residue values for decline during storage is not required.

E. REFERENCES

D278386, PP#0F06313, Investigation of the Stability of Residues of BAS 510 F in Plant Matrices under Normal Storage Conditions, M. Nelson, 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: D307931
PC Code: 128008

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