



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

**OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEW
EPA SERIES 361**

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM 09/21/2001

SUBJECT: Linuron: Magnitude of Residues in/on Sweet Corn.

Reregistration Case No.: 0047

PC Code: 035506

DP Barcode No.: D276294

MRID No.: 45444101

FROM: John S. Punzi, Ph.D., Chemist
Reregistration Branch II
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THROUGH: Alan Nielsen, Branch Senior Scientist
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TO: Tom Myers, Chemical Review Manager
Special Review and Reregistration Division [7508C]

Attached is a review of the Residue Chemistry Guideline study (860.1500); crop field trials for sweet corn submitted by Griffin Corporation in support of the Reregistration of linuron. The primary review was completed by the Dynamac Corporation under supervision of HED and has undergone secondary review/modification in Reregistration Branch 2 for consistency with current EPA policies.

Conclusions/Recommendations

1. Based on overall method validation and concurrent recovery data, the GC/ECD method, Morse Laboratories SOP Meth-65, Revision No. 3, is adequate for data collection purposes for determination of residues of linuron and its metabolites that can be hydrolyzed to 3,4-DCA in/on sweet corn K+CWHR, forage, and stover. The reported LOQ for linuron residues of concern was 0.01 ppm.
- 2a. Samples were stored frozen between collection and analysis. The intervals between harvest

and residue analysis were 146-203 days (4.8-6.7 months) for sweet corn forage, 141-189 days (4.6-6.2 months) for sweet corn K+CWHR, and 121-163 days (4.0-5.4 months) for sweet corn stover.

- 2b. The petitioner has initiated a storage stability study with sweet corn K+CWHR, forage, and stover. Only the results from the initial (0-day) storage interval were reported in the current submission. The registrant stated that the results from 3- and 6-month storage intervals will be submitted to the Agency when the study is completed.
- 3a. The submitted field trial data are not adequate to support use of linuron on sweet corn. Additional field trial data are required to fulfill geographic representation requirements.
- 3b. Although inadequate, the submitted data indicate that linuron residues of concern were below the LOQ (<0.01 ppm) to 0.048 ppm in/on sweet corn K+CWHR, <0.01 to 2.44 ppm in/on sweet corn forage, and 0.0189 to 4.00 ppm in/on sweet corn stover harvested at maturity following a single soil directed application of the 50% DF or 4 lb/gal FIC formulation at 1.48-1.57 lb ai/A made when sweet corn plants were at least 15 inches tall.
- 3c. If the registrant wishes to reinstate sweet corn uses on their linuron product labels, three additional sweet corn field trials, to be conducted in Regions 1 and 5 (2 trials), must be submitted. In addition, the registrant must submit amended product labels which include the proposed use on sweet corn; the proposed use must be consistent with the submitted field trial data. Finally, the available data indicate that the established tolerances for sweet corn forage and stover would be too low; therefore, the registrant must propose increased tolerances for sweet corn forage and stover.

cc: JSPunzi (RRB2), Linuron Reg. Std. File, Linuron SF, RF, LAN.
RD/T: RRB2 Chem Review Team (09/27/2001), Alan Nielsen (10/02/2001).
7509C:RRB2:John S. Punzi:CM2:Rm 804E:703-305-7727:09/21/2001.

LINURON
PC Code 035506; Case 0047
(DP Barcode D276294)

Registrant's Response to Residue Chemistry Data Requirements

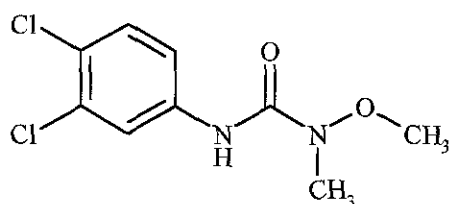
August 23, 2001

Contract No. 68-W-99-053

Submitted to:
U.S. Environmental Protection Agency
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LINURON



PC Code 035506; Case 0047

(DP Barcode D276294)

REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS

BACKGROUND

Griffin Corporation has submitted (2000; MRID 45444101) crop field trial data for sweet corn in support of reregistration of linuron. Additional crop field trial data for sweet corn commodities had been required in the Residue Chemistry Chapter of the Linuron Reregistration Eligibility Decision (DP Barcode D194477, 2/18/94, D. McNeilly and DP Barcode D212230, 2/21/95, S. Hummel). Previously, the registrant elected to cancel uses of linuron on sweet corn instead of submitting residue data (FR notice dated 11/17/95). The registrant is now submitting crop field trial data to satisfy the data requirements specified in the Linuron RED. The Conclusions and Recommendations stated in this review pertain only to the above topic; other residue chemistry data requirements specified in the Linuron Reregistration Eligibility Decision, dated 2/18/94, and Linuron Addendum to RED, dated 2/21/95, are not addressed herein.

The qualitative nature of the residue in plants is adequately understood based on acceptable metabolism studies conducted on corn, soybeans, and potatoes. The qualitative nature of the residue in animals is adequately understood based on acceptable metabolism studies conducted on ruminants and poultry. HED has determined that the residues that need to be regulated in plants and animals should consist of the parent linuron and its related compounds which are convertible to 3,4-dichloroaniline (3,4-DCA), as determined by the enforcement and data collection methods.

Tolerances for residues of linuron in/on various raw agricultural and animal commodities are **currently** expressed in terms of linuron *per se* [3-(3,4-dichlorophenyl)-1-methoxy-1-methylurea] [40 CFR §180.184(a)]; a tolerance with regional restriction has been established for residues of linuron *per se* in/on parsley [40 CFR §180.184(b)]. As stated above, HED has recommended that the linuron tolerance expression for plant and animal commodities be revised to include linuron and its related compounds convertible to 3,4-DCA.

Adequate enforcement methods are available for the determination of linuron in plant and animal commodities. The Pesticide Analytical Manual (PAM) Vol. II lists a colorimetric method (Method I) and a paper chromatographic method (Method II). Residues of diuron may interfere in Method I; a modified version of Method I (H. L. Pease, *Journal of Agric. and Food Chem.*, 1962, Vol. 10, p. 279), which includes a cellulose column step to separate linuron from diuron is available. Both of these methods determine linuron and all metabolites hydrolyzable to 3,4-dichloroaniline and have limits of detection of 0.05 ppm. A GLC/ECD method for linuron residues in/on asparagus from the CA Department of Food and Agriculture has been validated by the Agency and sent to FDA to be published in PAM Vol. II as Method III. This method determines residues of linuron *per se* and the limit of detection is 0.05 ppm. However, this method is inadequate for tolerance enforcement since it does not determine all the residues of concern and uses benzene as the extraction solvent.

As there are no Codex MRLs for residues of linuron, there are no questions with respect to Codex/U.S. tolerance compatibility.

CONCLUSIONS AND RECOMMENDATIONS

OPPTS GLN 860.1340: Residue Analytical Methods - Plant Commodities

1. Based on overall method validation and concurrent recovery data, the GC/ECD method, Morse Laboratories SOP Meth-65, Revision No. 3, is adequate for data collection purposes for determination of residues of linuron and its metabolites that can be hydrolyzed to 3,4-DCA in/on sweet corn K+CWHR, forage, and stover. The reported LOQ for linuron residues of concern was 0.01 ppm.

OPPTS GLN 860.1380: Storage Stability Data

- 2a. Samples were stored frozen between collection and analysis. The intervals between harvest and residue analysis were 146-203 days (4.8-6.7 months) for sweet corn forage, 141-189 days (4.6-6.2 months) for sweet corn K+CWHR, and 121-163 days (4.0-5.4 months) for sweet corn stover.
- 2b. The petitioner has initiated a storage stability study with sweet corn K+CWHR, forage, and stover. Only the results from the initial (0-day) storage interval were reported in the current submission. The registrant stated that the results from 3- and 6-month storage intervals will be submitted to the Agency when the study is completed.

OPPTS GLN 860.1500: Crop Field Trials

- 3a. The submitted field trial data are not adequate to support use of linuron on sweet corn. Additional field trial data are required to fulfill geographic representation requirements.

- 3b. Although inadequate, the submitted data indicate that linuron residues of concern were below the LOQ (<0.01 ppm) to 0.048 ppm in/on sweet corn K+CWHR, <0.01 to 2.44 ppm in/on sweet corn forage, and 0.0189 to 4.00 ppm in/on sweet corn stover harvested at maturity following a single soil directed application of the 50% DF or 4 lb/gal FIC formulation at 1.48-1.57 lb ai/A made when sweet corn plants were at least 15 inches tall.
- 3c. If the registrant wishes to reinstate sweet corn uses on their linuron product labels, three additional sweet corn field trials, to be conducted in Regions 1 and 5 (2 trials), must be submitted. In addition, the registrant must submit amended product labels which include the proposed use on sweet corn; the proposed use must be consistent with the submitted field trial data. Finally, the available data indicate that the established tolerances for sweet corn forage and stover would be too low; therefore, the registrant must propose increased tolerances for sweet corn forage and stover.

DETAILED CONSIDERATIONS

OPPTS GLN 860.1340: Residue Analytical Methods

Data collection methods - plant commodities:

Samples of sweet corn K+CWHR, forage, and stover from the submitted crop field trials (MRID 45444101) were analyzed for residues of linuron and its metabolites hydrolyzable to 3,4-DCA by McKenzie/Wright Laboratories (Phoenix, AZ) using a modification of Morse Laboratories SOP Method No. 65, Revision 3. The method involved hydrolysis of samples by alkaline reflux to convert residues to 3,4-DCA, distillation into concentrated acid, neutralization, and clean up by silica gel column chromatography. Analysis was by GC with an electron capture detector (ECD). The reported LOQ for linuron residues of concern was 0.01 ppm. We note that this method is also used for data collection purposes for diuron, and therefore will not differentiate between residues arising from linuron and residues arising from diuron.

Raw data, sample calculations, and representative chromatograms were submitted, as well as method validation and concurrent method recovery data from the sweet corn field trials. Recovery analyses were conducted using samples fortified with linuron, 15654 (DCPMU), and R915 (DCPU). Recovery data are presented in Table 1. The submitted recovery data indicate that the GC/ECD method is adequate for collection of data for linuron residues of concern from sweet corn commodities.

Table 1. Method validation and concurrent recoveries of residues of linuron, 15654, and R915 from fortified samples of sweet corn commodities analyzed using the GC/ECD method.

Commodity	Fortification Level, ppm	Statistic	Recovery, %		
			Linuron	15654 (DCPMU)	R915 (DCPU)
Method Validation					
Sweet corn K+CWHR	0.01-0.25	Average (%)	86	99	82
		Recovery range (%)	77-96	82-115	72-90
		SD (%)	9.6	17	9.3
		Number	3	3	3
Sweet corn forage	0.01-0.25	Average (%)	90	74	84
		Recovery range (%)	86-93	73-76	70-105
		SD (%)	3.5	1.5	19
		Number	3	3	3
Sweet corn stover	0.01-0.25	Average (%)	105	94	92
		Recovery range (%)	89-118	87-100	81-107
		SD (%)	15	6.6	13
		Number	3	3	3
Concurrent Recovery					
Sweet corn K+CWHR	0.01-0.5	Average (%)	92	89	87
		Recovery range (%)	74-108	77-96	72-98
		SD (%)	15	11	14
		Number	4	3	3
Sweet corn forage	0.01-10 (linuron) 0.01-1.0 (15654 & R915)	Average (%)	93	88	84
		Recovery range (%)	77-101	82-99	77-88
		SD (%)	14	9.8	6.1
		Number	3	3	3
Sweet corn stover	0.01-10 (linuron) 0.01-2.0 (15654) 0.01-1.0 (R915)	Average (%)	97	89	90
		Recovery range (%)	94-100	84-97	74-110
		SD (%)	3.1	7.2	18
		Number	3	3	3

OPPTS GLN 860.1380: Storage Stability Data

Griffin provided adequate information pertaining to storage and handling procedures for samples collected from the sweet corn field trials. The collected sweet corn commodity samples were placed in the freezer within 6 hours after harvest and were shipped via freezer truck to the analytical laboratory, McKenzie/Wright Laboratories (Phoenix, AZ), where they were stored frozen (-29 to -15 C) until analysis. The intervals between harvest and residue analysis were 146-203 days (4.8-6.7 months) for sweet corn forage, 141-189 days (4.6-6.2 months) for sweet corn K+CWHR, and 121-163 days (4.0-5.4 months) for sweet corn stover.

To support the storage intervals and conditions of samples from the sweet corn field trials, Griffin initiated a study to investigate the stability of linuron, 15654, and R915 in sweet corn K+CWHR, forage, and stover during frozen storage (MRID 45444101). Untreated samples of sweet corn commodities were fortified with linuron, 15654, and R915, each at 0.25 ppm. The fortified samples will be stored frozen for 0, ~3, and ~6 months. The registrant included the results of the 0-day interval in the current submission; these results will not be presented here. The registrant stated that the storage stability study is ongoing, and that the results of the remaining storage intervals will be submitted to the Agency when the study is completed.

OPPTS GLN 860.1500: Crop Field Trials

Sweet corn

Established tolerance: A tolerance of 0.25 ppm has been established for residues of linuron *per se* in/on fresh corn (sweet K+CWHR), and tolerances of 1 ppm have been established for residues in/on sweet corn forage and fodder [40 CFR §180.184(a)].

Use patterns registered to Griffin: Currently, there are no registered uses of linuron on sweet corn on Griffin product labels. The registrant requested cancellation of existing sweet corn uses in 1995 (60 FR 57717, 11/17/95).

Discussion of the data: Griffin Corporation has submitted data (2000; MRID 45444101) depicting the magnitude of linuron residues of concern in/on sweet corn K+CWHR, forage, and stover.

A total of nine sweet corn field trials were conducted during the 2000 growing season in Region 1 (PA), Region 2 (NC), Region 3 (FL), Region 5 (IL, OH, and MN), Region 10 (CA), Region 11 (OR), and Region 12 (OR). Five trials (CA, FL, IL, and one OR site) reflected use of the 50% DF formulation (EPA Reg. No. 1812-320; Lorox® DF) and five trials (MN, NC, OH, PA, and one OR site) reflected use of the 4 lb/gal FIC formulation (EPA Reg. No. 1812-245; Linex® 4L). Sweet corn commodities were collected at maturity following a single application of either the 50% DF or 4 lb/gal FIC formulation at 1.48-1.57 lb ai/A. Applications were made directed to the soil surface between the rows when the sweet corn was at least 15 inches tall. All applications were made with a surfactant applied at approximately 0.5% v/v. Applications were made in 21-33 gal/A of spray solution using ground equipment.

The registrant reported that climatic conditions were outside normal ranges in the following instances: (i) cooler than normal temperatures at the PA site; (ii) below normal rainfall at the FL (43% of normal), MN (64% of normal), and Hermiston, OR (33% of normal) trial sites during the entire trial period; (iii) below normal rainfall at the NC (70% of normal), OH (57% of normal), and Corvallis, OR (53% of normal) during one or two months of the study; and (iv) above normal rainfall (65% above normal) at the OH site during two months of the study. The CA, FL, OR (both), and PA sites were irrigated. The registrant stated that the sweet corn crop

was not adversely affected by climatic conditions at any of the sites.

One control and duplicate treated samples of sweet corn forage and K+CWHR were collected from each test site at normal crop maturity, 27-56 days posttreatment. Stover samples were collected after the stalks had dried in the field, 66-80 days posttreatment. The collected sweet corn commodity samples were placed in the freezer within 6 hours after harvest and were shipped via FedEx with dry ice to the analytical laboratory, McKenzie/Wright Laboratories (Phoenix, AZ).

Linuron residues of concern in/on treated and untreated sweet corn commodities were determined using a modification of GC/ECD method, Morse Laboratory Method No. 65, Revision No. 3, dated 8/93. The reported method LOQ was 0.01 ppm.

The results of the sweet corn field trials are presented in Table 2. Apparent linuron residues of concern were below the LOQ (<0.01 ppm) in/on nine samples each of untreated sweet corn forage and K+CWHR and in/on eight samples of untreated stover. One sample of untreated sweet corn stover bore detectable residues at 0.0305 ppm.

The registrant noted that linuron residues of concern in sweet corn commodities from the CA trial were much higher than those from the other field trials (up to 10x the highest residue in the other samples). No reason for the higher residues was found; however, the registrant suspected that contamination, from linuron-containing soil adhering to the lower portion of the corn stalk, may have occurred.

Table 2. Linuron residues of concern, determined as 3,4-DCA, in/on sweet corn commodities treated with a single application of the 50% DF or 4 lb/gal FIC formulation at 1.48-1.57 lb ai/A.

Sweet corn commodity	Test site (EPA Region)	Formulation	Application rate, lb ai/A	PHI, days	Residues, ppm ^a
Forage	Porterville, CA (Region 10)	50% DF	1.52	44	1.28, 2.44
	Bascom, FL (Region 3)	50% DF	1.52	29	0.0587, 0.105
	Wyoming, IL (Region 5)	50% DF	1.48	42	<0.01, 0.0514
	Campbell, MN (Region 5)	4 lb/gal FIC	1.50	53	0.0107, 0.0196
	Lucama, NC (Region 2)	4 lb/gal FIC	1.48	27	0.0515, 0.0672
	New Holland, OH (Region 5)	4 lb/gal FIC	1.57	38	0.0347, 0.0633
	Hermiston, OR (Region 11)	50% DF	1.52	56	0.171, 0.272
	Corvallis, OR (Region 12)	4 lb/gal FIC	1.50	47	0.014, 0.0184
	Germansville, PA (Region 1)	4 lb/gal FIC	1.55	38	0.139, 0.182
K+CWHR	Porterville, CA (Region 10)	50% DF	1.52	44	0.04, 0.048
	Bascom, FL (Region 3)	50% DF	1.52	29	<0.01, <0.01
	Wyoming, IL (Region 5)	50% DF	1.48	42	<0.01, <0.01
	Campbell, MN (Region 5)	4 lb/gal FIC	1.50	53	<0.01, <0.01
	Lucama, NC (Region 2)	4 lb/gal FIC	1.48	27	<0.01, <0.01
	New Holland, OH (Region 5)	4 lb/gal FIC	1.57	38	<0.01, <0.01
	Hermiston, OR (Region 11)	50% DF	1.52	56	<0.01, <0.01
	Corvallis, OR (Region 12)	4 lb/gal FIC	1.50	47	<0.01, <0.01
	Germansville, PA (Region 1)	4 lb/gal FIC	1.55	38	<0.01, <0.01
Stover	Porterville, CA (Region 10)	50% DF	1.52	80	2.67, 4.00
	Bascom, FL (Region 3)	50% DF	1.52	75	0.0695, 0.135
	Wyoming, IL (Region 5)	50% DF	1.48	71	0.0365, 0.434
	Campbell, MN (Region 5)	4 lb/gal FIC	1.50	80	0.0433, 0.0446
	Lucama, NC (Region 2)	4 lb/gal FIC	1.48	72	0.0189, 0.0655
	New Holland, OH (Region 5)	4 lb/gal FIC	1.57	66	0.0819, 0.0953
	Hermiston, OR (Region 11)	50% DF	1.52	70	0.682, 0.853
	Corvallis, OR (Region 12)	4 lb/gal FIC	1.50	75	0.056, 0.0708
	Germansville, PA (Region 1)	4 lb/gal FIC	1.55	75	0.0693, 0.124

^a Each residue value represents a single sample; residue values were not corrected for concurrent method recovery.

Geographic representation of data: Geographic representation of data is inadequate. According to OPPTS GLN 860.1500 (Table 5), a total of 12 field trials for sweet corn must be conducted in Regions 1 (2 trials), 2 (1 trial), 3 (1 trial), 5 (5 trials), 10 (1 trial), 11 (1 trial), and 12 (1 trial). The registrant only conducted 9 field trials, in Regions 1 (1 trial), 2 (1 trial), 3 (1 trial), 5 (3 trials), 10 (1 trial), 11 (1 trial), and 12 (1 trial). The registrant must conduct an additional three sweet corn field trials in Regions 1 and 5 (2 trials).

Because the use of linuron on sweet corn in these field trials is directed to the soil, data may be translated between the DF and FIC formulations.

Conclusions

The submitted field trial data are not adequate to support use of linuron on sweet corn. Additional field trial data are required to fulfill geographic representation requirements.

Although inadequate, the submitted data indicate that linuron residues of concern were below the LOQ (<0.01 ppm) to 0.048 ppm in/on sweet corn K+CWHR, <0.01 to 2.44 ppm in/on sweet corn forage, and 0.0189 to 4.00 ppm in/on sweet corn stover harvested at maturity following a single soil directed application of the 50% DF or 4 lb/gal FIC formulation at 1.48-1.57 lb ai/A made when sweet corn plants were at least 15 inches tall. If the registrant wishes to reinstate sweet corn uses on their linuron product labels, additional crop field trial data must be submitted as noted above. In addition, the registrant must submit amended product labels which include the proposed use on sweet corn; the proposed use must be consistent with the submitted field trial data. Finally, the available data indicate that the established tolerances for sweet corn forage and stover would be too low; therefore, the registrant must propose increased tolerances for sweet corn forage and stover.

EPA MEMORANDA CITED IN THIS REVIEW

DP Barcode:	D194477
Subject:	Linuron Reregistration Eligibility Document (RED).
From:	D. McNeilly
To:	L. Rossi and E. Saito
Dated:	2/18/94
MRID(s):	None

DP Barcode: D212230
Subject: Linuron. (035506) Addendum to RED Residue Chemistry Chapter,
Reregistration Case No. 0047
From: S. Hummel
To: K. Jones
Dated: 2/21/95
MRID(s): None

MASTER RECORD IDENTIFICATION NUMBER

The citation for the MRID document referred to in this review is presented below.

45444101 Willard, T (2000) Magnitude of Linuron Residues in/on Sweet Corn Resulting From a Post-Directed Applications of Lorox DF and Linex 4L: Final Study Report: Lab Project Number: AA000901: GP00-022. Unpublished study prepared by American Agricultural Services, Inc. 199 p.



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