

Residue Chemistry Review

3/29/1995

Comments:

Subject: New Chemical - Fipronil in or on corn. Results of Petition Method Validation (PMV). MRID# 433234-01.
CBTS# 15316.

Document

Class:

Product

Chem:

Residue 860.1200 Directions for use

Chem: 860.1340 Residue analytical method

860.1550 Proposed tolerances

Biochemicals:

DP Barcode: D213532

MRIDs: 43323401

PC Codes: 129121 1H-Pyrazole-3-carbonitrile, 5-amino-1-(2,6-dichloro-4-(trifluoromethyl)phenyl)-4-((trifluoromethyl)sulfi

Actives/Inerts

CAS #: 120068-37-3

Commodities: Corn; Corn, Field

Administrative 3G04263; 5F04426

#:

Reviewers: G. F. Kramer

Review R. B. Perfetti

Approved on: March 29, 1995

Approver:

WP Document:  - Fipronil_015.wpd

MEMORANDUM

SUBJECT: PP#s 3G04263 & 5F04426. New Chemical - Fipronil in or on corn. **Results of Petition Method Validation (PMV).** MRID# 433234-01. Barcode D213532. Chemical No 129121. CBTS# 15316.

FROM: G.F. Kramer, Ph.D., Chemist
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THRU: R.B. Perfetti, Ph.D., Acting Section Head
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TO: Ann Sibold, Team 10 Reviewer
Rick Keigwin, PM
Registration Division (7505C)

Rhône-Poulenc has submitted an application for permanent tolerances for the insecticide fipronil (5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(1R,S)-(trifluoromethyl)sulfinyl]-1H-pyrazole-3-carbonitrile) and its metabolites MB46136 (5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)sulfonyl]-1H-pyrazole-3-carbonitrile) and MB45950 (5-amino-1-[2,6-dichloro-4-(trifluoromethyl)phenyl]-4-[(trifluoromethyl)thio]-1H-pyrazole-3-carbonitrile) on/in corn. The petitioner has proposed the following tolerances (expressed as parent plus metabolites MB45950 and MB46136):

Corn Grain	--	0.02 ppm		Corn Fodder	--	0.15 ppm
Corn Forage	--	0.15 ppm				

In conjunction with proposed temporary tolerances and an EUP application (PP# 3G04263), CBTS requested that ACL perform a PMV on the following method (Memo, G. Kramer 9/12/94):

Fipronil- Validation of Method of Analysis for Fipronil and Its Metabolites in Field Corn. EC-93-236. 8/27/94. MRID# 433234-01

The results of the PMV and the TMV Pre-review are appended to this memorandum as Attachments 1 & 2.

Results

The average recovery in corn grain was $88.7 \pm 5.6\%$ for fipronil, $97.3 \pm 17.6\%$ for MB45950, $103.2 \pm 9.1\%$ for MB44136, $96.9 \pm 5.5\%$ for RPA105048 and $87.1 \pm 9.2\%$ for RPA200766; in corn forage, was $97.3 \pm 10.8\%$ for fipronil, $74.6 \pm 10.9\%$ for MB45950, $114.6 \pm 10.8\%$ for

MB44136, $112.6 \pm 6.3\%$ for RPA105048 and $115.4 \pm 7.6\%$ for RPA200766; and in corn fodder, was $92.8 \pm 7.7\%$ for fipronil, $105.2 \pm 15.0\%$ for MB45950, $104.5 \pm 6.0\%$ for MB44136, $106.9 \pm 20.6\%$ for RPA105048 and $99.0 \pm 7.2\%$ for RPA200766. One analyst can extract and clean-up six samples in 6 hours.

Conclusions

The recoveries of fipronil and its metabolites are acceptable. The following comments were made by ACL in the PMV results (Memo, M. Law 3/23/95):

- 1) The DB-1 GC column specified by the registrant failed to provide sufficient resolution. ACL used a 0.53 mm DB-1701 megabore column for grain and a 0.32 mm capillary DB-1701 column with splitless injection for forage and fodder. The method should be revised to specify the use of these columns, including the operating conditions employed by ACL.
- 2) Further cleanup of the silica gel and charcoal was required in order to eliminate interfering peaks. The method should be revised to include the clean-up procedures employed by ACL.
- 3) ACL substituted a rotary evaporator for the specified Turbo-Vap II. The method should be revised to include the rotary evaporator as an alternative to the Turbo-Vap II.

This method will be suitable for enforcement purposes once the revisions recommended by ACL are incorporated.

Recommendations

The registrant should submit a revised version of the proposed analytical enforcement method specified in conclusions 1-3. Until the receipt of the standard and the revised method, the requirements for analytical enforcement methodology will remain unfulfilled.

Attachment 1- Memo, M. Law 3/23/95

Attachment 2- Memo, E. Greer, Jr. 10/24/94

cc (with Attachments): M. Clower (FDA, HFS-335)

cc (without Attachment): PP#3G04263, PP#5F04426, S.F., Kramer,
circ., R.F.

RDI: R.B. Perfetti (3/28/95), M.T. Flood (3/28/95), E. Zager
(3/29/95)

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