Residue Chemistry Review

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
Chem: 860.1200 Directions for use
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)sulfinyl) pyrazole (Fipronil)
Actives/Inerts
CAS #: 120068-37-3
Commodities: Corn; Corn, Field
Administrative #: 3G04263; 5F04426

Reviewers: G. F. Kramer
Review Approver: R. B. Perfetti
Approved on: March 29, 1995

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.

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THRU: R.B. Perfetti, Ph.D., Acting Section Head
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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 ± 5.6% for fipronil, 97.3 ± 17.6% for MB45950, 103.2 ± 9.1% for MB44136, 96.9 ± 5.5% for RPA105048 and 87.1 ± 9.2% for RPA200766; in corn forage, was 97.3 ± 10.8% for fipronil, 74.6 ± 10.9% for MB45950, 114.6 ± 10.8% for
MB44136, 112.6 ± 6.3% for RPA105048 and 115.4 ± 7.6% for RPA200766; and in corn fodder, was 92.8 ± 7.7% for fipronil, 105.2 ± 15.0% for MB45950, 104.5 ± 6.0% for MB44136, 106.9 ± 20.6% for RPA105048 and 99.0 ± 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)
c (without Attachment): PP#3G04263, PP#5F04426, S.F., Kramer, circ., R.F.
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