

10/26/95

## *Residue Chemistry Review*

**Subject:** Fipronil in or on corn. Results of Petition Method Validation (PMV). MRID# 434011-10. CBTS# 16339.

**Document**

**Class:**

**Product**

**Chem:**

**Residue** 860.1200 Directions for use

**Chem:** 860.1340 Residue analytical method

**Biochemicals:**

**DP Barcode:** D220222

**MRIDs:** 43401110

**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:**

**Administrative** 5F04426

**#:**

**Reviewers:** G. F. Kramer

**Review** Bart Suhre

**Approved on:** October 26, 1995

**Approver:**

**WP Document:**  - Fipronil\_024.wpd

# MEMORANDUM

SUBJECT: PP# 5F04426. Fipronil in or on corn. **Results of  
Petition Method Validation (PMV).** MRID# 434011-10.  
Barcode D220222. Chemical# 129121. CBTS# 16339.

FROM: G.F. Kramer, Ph.D., Chemist  
Tolerance Petition Section I  
Chemistry Branch I, Tolerance Support  
Health Effects Division (7509C)

THRU: F.B. Suhre, Acting Section Head  
Chemistry Branch I, Tolerance Support  
Health Effects Division (7509C)

TO: Rick Keigwin, Product Manager  
Ann Sibold, Team 10 Reviewer  
Registration Division (7505C)

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

Corn Grain	--	0.02 ppm	Corn Fodder	--	0.15 ppm
Corn Forage	--	0.15 ppm	Liver*	--	0.02 ppm
Milk*	--	0.02 ppm	Eggs	--	0.02 ppm
Fat*	--	0.08 ppm	Poultry Skin/Fat	--	0.03 ppm
Muscle*	--	0.02 ppm	Poultry Muscle	--	0.02 ppm
Kidney*	--	0.02 ppm	Poultry Liver	--	0.02 ppm

\*of Dairy Cows

On 5/11/95, CBTS requested that ACL perform a PMV on the following method:

Fipronil- Validation of Method of Analysis for Fipronil and Its Metabolites in Animal Tissues. EC-94-258. 9/9/94. MRID# 434011-10

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

## Results

The average recoveries in milk were  $69.1 \pm 1.3\%$  for MB45950,  $74.0 \pm 1.4\%$  for fipronil and  $79.8 \pm 7.4\%$  for MB46136; in liver,  $80.4 \pm 1.2\%$ ,  $81.8 \pm 0.6\%$  and  $89.5 \pm 5.9\%$ ; and in fat,  $84.9 \pm 2.9\%$ ,  $85.2 \pm$

2.6% and  $91.0 \pm 2.6\%$ . One analyst can extract and clean-up six samples in 24 hours.

### Conclusions

The recoveries of fipronil are acceptable. The following additional comments were made by ACL in the TMV Pre-review (Memo, E. Greer, Jr. 5/30/95):

- 1) The reagent list should be modified to specify the type of celite employed (Celite 503).
- 2) The size of the filter paper and filtration apparatus should be specified.
- 3) A statement should be made in Section 5 as to whether the column is allowed to go dry between additions of eluant.
- 4) A statement should be made in Section 6 to direct the analyst to collect the eluate at the end of this step.
- 5) The calculations include directions to correct for the values found in the untreated controls. As the use of control samples is not permitted in enforcement methods, this equation should be modified by deleting the correction for the untreated sample.

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 as specified in conclusions 1-5. Until the receipt of the revised method, the requirements for analytical enforcement methodology will remain unfulfilled.

Attachment 1- Memo, P. Golden 10/2/95  
Attachment 2- Memo, E. Greer, Jr. 5/30/95

cc (with Attachments): M. Clower (FDA, HFS-335)  
cc (w/o Attachment): PP#5F04426, S.F., Kramer, Circ., R.F., H. Hundley (7503W)  
RDI: F.B. Suhre (10/24/95), M.S. Metzger (10/25/95), R.A. Loranger (10/24/95)  
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