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

Analytical Chemistry Branch
Environmental Science Center
Fort George G. Meade, MD 20755-5350

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

September 17, 2004

MEMORANDUM

SUBJECT: PP# 3F6542. Review of Residue Analytical Method Data Submitted to Support Food Tolerances for Penoxsulam.

PC Code: 119031
DP Barcode: D303172
MRIDs: 45830714 (enforcement method report)
45830715 (ILV report)
ACL Project #: B04-22

FROM: Charles Stafford, Team Leader *Charles Stafford*
Analytical Chemistry Branch
Biological and Economic Analysis Division (7503C)

THRU: Fred Siegelman, Chief *Fred Siegelman*
Analytical Chemistry Branch
Biological and Economic Analysis Division (7503C)

TO: William Cutchin, Chemist
Science Information Management Branch (SIMB)
Health Effects Division (7509C)

and

Philip Errico, Product Manager
Herbicide Branch
Registration Division (7505C)

INTRODUCTION

The Analytical Chemistry Branch (ACB) was requested by HED/SIMB to conduct a laboratory tolerance method validation (TMV) of an enforcement method submitted by Dow AgroSciences LLC to support tolerances for the new chemical penoxsulam. A TMV was requested for rice grain and rice straw commodities in a memo from W. Cutchin to F. Siegelman dated 7/19/2004. ACB has reviewed the proposed enforcement analytical method, ILV data, and supporting data for penoxsulam.

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ANALYTICAL METHODS AND DOCUMENTATION

MRID 45830714 - Study Title: Validation Report for Method GRM 01.25 - Determination of Residues of XDE-638 in Rice and Rice Processed Products by Liquid Chromatography with Tandem Mass Spectrometry. Author: M. J. Hastings. Study Completed: June 20, 2002. Lab Study ID: 010097. Study (pages 20-41) contains Method GRM 01.25, Dated April 27, 2002. Performing Lab: Regulatory Laboratories-Indianapolis Lab, Dow Agrosciences LLC.

MRID 45830715 - Study Title: Independent Laboratory Validation of Dow AgroSciences LLC Method GRM 01.25 - Determination of XDE-638 in Rice and Rice Processed Products by Liquid Chromatography with Tandem Mass Spectrometry Detection. Author: Clark D. Chickering. Study Completed: July 16, 2002. Performing Lab: ABC Laboratories. Submitting Lab: Dow AgroSciences LLC. Lab Study ID: Dow AgroSciences Protocol No. 020038; ABC Laboratories Report #47421.

RECOMMENDATIONS

1. The ACB recommends that based on our review of the proposed enforcement method and supporting method data, without laboratory validation, Method GRM 01.25 appears to meet the OPPTS 860.1340 Residue Chemistry Test Guidelines for an acceptable enforcement method provided Recommendation 2(a) is adopted. The ACB also recommends that the method does not need to be validated in an Agency laboratory for the following reasons:
 - A. Sufficient method validation data was submitted for Method GRM 01.25 to show that the proposed method is adequate for enforcement of tolerances.
 - B. Method GRM 01.25 was successfully validated by an independent laboratory.
2. The ACB recommends that the method be revised by the petitioner as follows:
 - a. The method identifies penoxsulam residues using only one LC/MS/MS parent-product ion transition. We recommend the petitioner add information to the method which documents either one additional ion transition, or a different chromatographic column/mobile-phase combination as a confirmatory option to reduce the possibility of false positive residues.
3. The ILV study suggested that several corrections and modifications should be made to the method. Dow responded to those suggested changes in a letter from Rafael Herrera to Joanne Miller dated 8/23/2004. The ACB was asked to respond to the ILV suggestions and Dow's followup response. ACB's recommendations are:
 - #1. Dow method step 9.3.9 has a typographical error and Dow agreed to revise the method to delete the extraneous word "dilute". We concur.
 - #2. a) The ILV report states concerns with potential cross-contamination of sample wells using the recommended equipment. We concur with Dow's explanation that the potential problem was with the ILV lab's use of a non-compatible evaporator. We don't believe the method needs to be revised in this regard.

b) The ILV states that the method did not specify to dilute samples outside the instrument calibration range. Dow points out that the instruction was stated in the method. We don't believe the method needs to be revised in this regard.

c) The ILV lab states that there were no instructions for collecting fractions to calibrate the cleanup columns. Dow points out that the instructions were stated in the method. We don't believe the method needs to be revised in this regard.

d) The ILV report questions a statement regarding "purifying" the extract while performing calibration of the cleanup columns. We concur with Dow's responses. We don't believe the method needs to be revised in this regard.

e) The ILV report notes an error in calculating ppm found. Dow has agreed to revise the method to eliminate this discrepancy. We concur.

f) The ILV report states that the Dow recovery tables should indicate that recoveries were corrected by subtracting interferences found in control materials. Dow states that the table footnote explains that recoveries were corrected if applicable. In fact, no corrections were made since the controls were blank. We don't believe the method needs to be revised in this regard.

COMMENTS

1. Method GRM 01.25 gives an equation for calculating net percent recovery during method validation by subtracting the interference found in clean, control materials. The OPPTS guidelines do not allow an enforcement method to require the use of clean, control materials to perform sample analysis. However, the equation given for calculating residue values in actual samples does not correct for interferences found in control materials, and therefore the equations in this section are acceptable.