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

MAY 16 1985

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP# 5F3180. Cyromazine on lettuce. Evaluation of Method Trial Report Dated May 8, 1985.

FROM: Nancy Dodd, Chemist
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THRU: Charles L. Trichilo, Ph.D.,Chief
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TO: Timothy A. Gardner, Product Manager (17)
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and

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EPA's Analytical Chemistry Section (COB, BUD) has completed a method trial of cyromazine (N-cyclopropyl-1,3,5-triazine-2,4,6-triamine) and melamine (1,3,5-triazine-2,4,6-triamine) on lettuce (method AG-408) as requested (PP# 5F3180, C. Deyrup, Ph.D., March 13, 1985).

When lettuce samples were fortified with 4 ppm cyromazine and 2 ppm melamine and subjected to one ion exchange cleanup step, recoveries were 94.5 and 98.5 percent (average 96.5 percent) cyromazine and 88.2 and 105.9 percent (average 97 percent) melamine. Recoveries for corresponding samples fortified at 8 ppm cyromazine and 2 ppm melamine were 81.0 and 89.4 percent (average 85 percent) cyromazine and 80.4 and 87.2 percent (average 84 percent) melamine. Corresponding control values were 0.16 and 0.18 ppm cyromazine and 0.06 and 0.07 ppm melamine.

When lettuce samples were fortified with 4 ppm cyromazine and 1 ppm melamine and taken through the complete method (i.e., multiple cleanup steps), recoveries were 78.6 and 87.3 percent (average 83 percent) cyromazine and 89.2 and 96.1 percent (average 93 percent) melamine. Recoveries for corresponding samples fortified at 8 ppm cyromazine and 2 ppm melamine were 67.4 and 92.8 percent (average 80 percent) cyromazine and 76.5 and 89.7 percent (average 83 percent) melamine. Control values were reported as (< 0.05 ppm) for samples subjected to the complete method (multiple cleanup steps).

Slight modifications to the method were the following (May 8, 1985, memo of Mark Law, ACS, COB):

> "A 125ml round bottom flask was used wherever the method called for a 250ml round bottom. The contents of the flask never exceed 30 ml, and since the flask is rinsed with 10ml of solvent, recoveries could be improved with less glass surface to rinse. The final step in the clean up is to "evaporate the contents to dryness, then make to final volume for HPLC analysis". This should read "evaporate to aqueous..." because there is approximately 4ml of water present at this stage, which cannot be evaporated on a rotovap. I evaporated to aqueous then transferred to a graduated centrifuge tube by pipette and used acetonitrile rinses of the flask to bring the sample to its final volume. These last comments should be put into the method."

Also, a Dowex 1-X8 Anion Exchange Resin, 50 to 100 mesh, was used in place of the Bio-Rex 9 Anion Exchange Resin, 50 to 100 mesh, which is no longer manufactured (conversation with Ron Thomas at EPA's Beltsville lab on May 9, 1985).

Conclusions

RCB finds method No. AG-408 (including minor revisions) adequate as an enforcement method for analysis of cyromazine in lettuce and celery. The detectability of the method on lettuce is 0.2 ppm cyromazine and 0.3 ppm melamine based on submitted residue data for lettuce (PP# 5F3180, C. Deyrup, March 20, 1985). The detectability of the method may be lowered to 0.05 ppm (reported by COB, BUD) by the use of multiple cleanup steps.

Minor revisions to the method were as follows:

1. A 125 ml round bottom flask was used wherever a 250 ml round bottom flask was specified in the method.

2. In the final step of the cleanup, instead of following the directions to "evaporate the contents to dryness, then make to final volume for HPLC analysis" the solution was evaporated to aqueous, transferred to a graduated centrifuge tube by pipette, and brought to final volume with acetonitrile rinses of the flask.

3. A Dowex 1-X8 Anion Exchange Resin, 50 to 100 mesh, was used in place of the Bio-Rex 9 Anion Exchange Resin, 50 to 100 mesh, which is no longer manufactured.

RCB concludes that Deficiency #2d listed in the review of PP# 5F3180 (C. Deyrup, Ph.D., March 20, 1985) is resolved. All other deficiencies related to the proposed lettuce and celery tolerances were previously resolved. (Refer to above review dated March 20, 1985.)

Recommendations

RCB recommends that the three modifications to the method No. AG-408 listed in the conclusions above be incorporated into the method.

TOX and EAB considerations permitting, RCB recommends that the proposed permanent tolerances for cyromazine (N-cyclopropyl-1,3,5-triazine-2,4,6-triamine) and its principal metabolite melamine (1,3,5-triazine-2,4,6-triamine) be established on head lettuce at 5 ppm and celery at 10 ppm.

cc:RF,Circu, Reviewer, TOX, EAB, EEB, PP#5F3180, FDA, Robert Thompson, PMSD-ISB, M. Bradley
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