11-18-92

Residue Chemistry Review

Comments:	
Subject:	PP#2F4109 -- Lambda-cyhalothrin for Use in/on Field, Pop and Seed Corn.; CBTS #s 9685, 10231. MRID #'s 422537-01 through 422537-04, 423879-01.; PP#2F4114 -- Lambda-cyhalothrin for Use in/on Peanuts. CBTS # 9773. MRID #'s 422732-01 through -03. PP#7F3560/7H5543 -- Lambda-cyhalothrin for Use in/on Wheat, Sweet Corn, Sunflowers. PP#1F3992 -- Lambda-cyhalothrin for Use in/on Grain Sorghum.
Document Class:	
Product Chem:	
Residue Chem:	860.1200 Directions for use 860.1340 Residue analytical method 860.1380 Storage stability data 860.1480 Meat/milk/poultry/eggs 860.1500 Crop field trials 860.1520 Processed food/feed 860.1550 Proposed tolerances
Biochemicals:	
DP Barcode:	D176545, D180584, D177078
MRIDs:	42253701, 42253702, 42253703, 42253704, 42387901, 42273201, 42273202, 42273203
PC Codes:	128897 lambda-Cyhalothrin
Commodities:	Corn, Field; Corn, pop; Corn; Peanut; Corn, sweet; Sunflower; Wheat; Sorghum, Grain; Cattle, Fat; Cattle, Meat; Cattle, MBYP; Goat, fat; Goat, MBYP; Goat, Meat; Hog, Fat; Hog, MBYP; Hog, Meat; Horse, Fat; Horse, MBYP; Horse, Meat; Sheep, Fat; Sheep, MBYP; Sheep, Meat; Milk, fat; Poultry, fat; Poultry, MBYP; Poultry, Meat; Egg
Administrative #:	1F03992; 2F04109; 2F04114; 7F03560; 7H05543
Reviewers:	Michael T. Flood
Review Approver:	Debra Edwards Approved on: November 18, 1992
DOS File Name:	CYHALOTH.007
WP Document:	 Cyhaloth.0

MEMORANDUM

SUBJECT: **PP#2F4109** -- Lambda-cyhalothrin for Use in/on Field, Pop and Seed Corn.

DP Barcodes: D176545, D180584. CBTS #s 9685, 10231.
MRID #'s 422537-01 through 422537-04, 423879-01.

PP#2F4114 -- Lambda-cyhalothrin for Use in/on Peanuts.

DP Barcode: D177078. CBTS # 9773.
MRID #'s 422732-01 through -03.

PP#7F3560/7H5543 -- Lambda-cyhalothrin for Use in/on Wheat, Sweet Corn, Sunflowers.

PP#1F3992 -- Lambda-cyhalothrin for Use in/on Grain Sorghum.

FROM: Michael T. Flood, Ph.D., Chemist
Tolerance Petition Section II
Chemistry Branch I -- Tolerance Support
Health Effects Division (H7509C)

THROUGH: Debra F. Edwards, Ph.D., Acting Chief
Chemistry Branch I -- Tolerance Support
Health Effects Division (H7509C)

TO: G. LaRocca/A. Heyward, PM 15
Insecticide-Rodenticide Branch
Registration Division (H7505C)

and

Toxicology Branch I
Health Effects Division (H7509C)

With accompanying letter dated 2/28/92, ICI Agricultural Products has submitted a petition in which the following tolerances on <u>field corn</u> raw agricultural commodities (RACS) are proposed for lambda-cyhalothrin, per se:

Corn, fodder	3.0 ppm
Corn, grain, field, pop and seed	0.05 ppm
Corn, grain dust	0.1 ppm

Corn, silage	1.0 ppm

Tolerances of 0.05 ppm and 5.0 ppm have been proposed for the combined residues of lambda-cyhalothrin and its epimer in/on sweet corn (K + CWHR) and sweet corn forage, respectively, as a result of PP#7F3560/7H5543 (see our memo of 4/9/92). A permanent food additive tolerance of 10 ppm for residues of lambda-cyhalothrin, per se in/on dried hops has been established under 40 CFR 185.1310. However, CBTS has recommended that this tolerance expression also include the epimer. In its 3/23/91 memo for FAP#0H5599, the petition which resulted in the permanent tolerance for dried hops, CBTS also recommended that the following permanent tolerances be established for lambda-cyhalothrin and its epimer:

Fat of cattle, goats, horses and sheep	0.02 ppm
Meat and meat byproducts of cattle, goats, horses and sheep	0.01 ppm
Milkfat (reflecting 0.01 ppm in whole milk) whole milk)	0.25 ppm

CBTS has also recommended that the Chemical Abstracts name for lambda-cyhalothrin, [1α(S*), 3α(Z)]-(±)-cyano(3-phenoxyphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylate, be replaced with the IUPAC name:

A 1:1 mixture of

(S)-α-cyano-3-phenoxybenzyl (1R)-cis-3-(Z-2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

and

(R)-α-cyano-3-phenoxybenzyl (1S)-cis-3-(Z-2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

In this nomenclature the name of the epimer of lambda-cyhalothrin would be:

A 1:1 mixture of

(S)-α-cyano-3-phenoxybenzyl (1S)-cis-3-(Z-2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

and

(R)-α-cyano-3-phenoxybenzyl (1R)-cis-3-(Z-2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

Currently active petitions include the following:

PP#7F3560/7H5543, for use in/on wheat, sweet corn and sunflowers.

PP#1F3952/1H5607, for use in/on broccoli, cabbage and tomatoes.

PP#9F3770, for dermal application to beef cattle.

PP#7F3488, for use in/on soybeans.

Our most recent review of PP#7F3560/7H5543 and PP#1F3992 is dated 9/1/92. PP#7F3488 is being reviewed in a memo concurrent with this one.

The present submission of PP#2F4109 consists of residue data on field corn and popcorn, residue data on processed products, and the independent laboratory validation of ICI's method for lambda-cyhalothrin in animal products.

By hand delivered letter dated 7/8/92, ICI has also submitted results of a 24 month storage stability study on metabolites of lambda-cyhalothrin. Also included in this submission is a revised Section F for the various lambda-cyhalothrin petitions reviewed in our 9/19/91 memo.

With cover letter dated 3/31/92, ICI has submitted a new petition for use of lambda-cyhalothrin in/on peanuts. The submission includes a magnitude of residue studies on peanuts and a processing study.

The following tolerances are proposed:

Peanut, hulls	0.05 ppm
Peanut, nutmeats	0.05 ppm

Summary of Deficiencies Remaining to Be Resolved for PP#7F3560/7H5543 and PP#1F3992

Revised Section F should be submitted, as requested in our memo of 9/1/92.

Summary of Deficiencies Remaining to Be Resolved for PP#2F4109 and PP#2F4114

Storage stability data for metabolites in peanut meat and hulls. Storage stability data for parent and metabolites in corn and peanut processed products. (Conclusions 3c, 3d)

Revised Section F. (Conclusions 4a, 4b and 5a)

4

Conclusions

1. The nature of the residue in plants and animals is adequately understood. The residue to be regulated is lambda-cyhalothrin, per se, and its epimer.

2a. Analytical methods are available for enforcement. ICI Method 81 for lambda-cyhalothrin + epimer in plants and Method 86 for these residues in animal products have undergone successful EPA method validation.

2b. Because at the present time lambda-cyhalothrin metabolites need not appear in the tolerance expression, EPA method validation for the analytical methods is not necessary. However, should it become necessary to regulate the metabolites, analytical method ARAM 96-1, for residues of metabolites in animal products, should be revised to incorporate comments made in the independent laboratory validation.

2c. Recoveries of cyhalothrin have been obtained under FDA's multiresidue protocols. Because lambda-cyhalothrin and its epimer are isomers that comprise a part of cyhalothrin, they should behave similarly under these protocols.

3a. Storage stability data for metabolites in a number of plant matrices support analyses in plant matrices for periods up to two years. Deficiencies in storage stability data noted for sweet corn (PP#7F3560/7H5543) and broccoli, cabbage and tomatoes (PP#1F3952/1H5607) are resolved.

3b. Storage stability data for parent and metabolites are available to support the residue analyses on field, pop and seed corn, forage and fodder.

3c. Storage stability data are adequate to support the residue analyses for parent in peanut meat and hulls; however storage stability data on metabolites for periods up to 31 months is necessary. Such a study is in progress.

3d. Storage stability data are lacking in support of the analyses for parent and metabolites in the processed commodities of corn, peanuts and soybeans (PP#7F3488). Stability of parent and metabolites in three processed commodities must be demonstrated for periods up to 17 months. Because available storage stability data on racs indicate a potential problem with peanuts, two of these processed commodities should be peanut meal and peanut oil. If parent or a metabolite is not stable in

these matrices, stability data in other processed commodities may also be required.

4a. Based on submitted residue data, appropriate tolerances for corn grain, forage and fodder are 0.05, 1.0 ppm and 1.0 ppm, respectively. At this time, we do not consider results which include the fonofos/lambda-cyhalothrin premix applications as relevant to the present submission. However, registration of this latter formulation may require additional residue data. ICI Americas should submit a revised Section F in which the above tolerances are proposed.

Because lambda-cyhalothrin residues in corn grain were non-detected, we do not consider a tolerance for these residues in/on grain dust to be necessary. The petitioner should submit a revised Section F in which the proposed tolerance on grain dust is omitted.

4b. Based on submitted residue data for residues of lambda-cyhalothrin in/on peanut meats and hulls, the proposed tolerances of 0.05 ppm for these commodities is appropriate. However, the tolerance for "peanuts, nutmeats" should be expressed simply as "peanuts". This conclusion is provisional, pending submission of adequate storage stability data on metabolites.

5a. Lambda-cyhalothrin concentrates when corn grain is processed into flour. The petitioner should submit a revised Section F in which a food additive tolerance of 0.15 ppm is proposed for this commodity. This conclusion is provisional pending submission of adequate storage stability data.

5b. Food/feed additive tolerances are not necessary for peanut processed commodities. Although parent concentrates when peanuts are processed into crude and refined oil, levels in bleached, deodorized oil are non-detectable. This conclusion is provisional pending submission of adequate storage stability data.

6. Tolerances recommended for meat, milk, poultry and eggs in our memo of 9/1/92 (PP#7F3560/7H5543) remain unaffected by the residue data submitted in the present petitions.

7. An International Residue Limit status sheet is appended to this review. There are Codex limits for cyhalothrin on other commodities. No compatibility problem exists for the RACS of this petition.

Recommendation

CBTS recommends against the proposed tolerances for reasons given in Conclusions 3c,4b (storage stability data on metabolites); 3d,5a,5b (storage stability data for parent and metabolite residues in processed products); 4a,4b,5a (revised Section F)

Detailed Considerations

The detailed manufacturing process for lambda-cyhalothrin was submitted in PP#6F3318 (MRID # 401820-01) and discussed in S. Brooks' (now S. Willett's) memo of 9/29/87. ICIA0321 (lambda-cyhalothrin) Technical (EPA Reg. No. 10182-131) contains a minimum 81% a.i. Karate® EC Insecticide (EPA Reg. No. 10182-96), the formulation to be registered for the proposed use, contains 13.1% a.i. (One pound a.i. per gallon, or 0.0078 lb. a.i./ fl. oz.) A Confidential Statement of Formula dated 1/28/92 is given in the administrative volume of this submission.

The petitioner notes that two formulations were used in developing the field corn residue data -- in addition to the EC, the WG formulation, a 12% wettable granular formulation. ICI does not intend to seek a registration for the WG formulation. According to the registrant, when mixed with water, the EC and WG formulations yield spray solutions with equal active ingredient concentrations.

"Given the strong similarity of the spray solutions of the EC and WG formulations, essentially identical residue results are anticipated from the use of the two formulations. This strong similarity was demonstrated."

ICI will be submitting an application for registration of a lambda-cyhalothrin/fonofos premix for use on field corn. The formulation is granular and contains 20.0% fonofos and 0.4% lambda-cyhalothrin. Although the present petition is to support the foliar use of lambda-cyhalothrin, some premix data are included. (Refer to the residue chemistry section of this review.)

Proposed Use

Corn, Field, Seed, Pop. The EC formulation (Karate®) may be ground or air applied at intervals of 3-7 days. Do not apply within 21 days of harvest. Do not apply more than 0.06 lb ai/A (0.48 pints) after silk initiation. Do not apply more than 0.03 lb ai/A (0.24 pints) after corn has reached the milk stage (yellow kernels with milky fluid). Do not apply more than 0.12 lb ai/A/season.

Directions for use of the fonofos/lambda-cyhalothrin premix (DYFONATE/0321) have also been submitted. The premix may be applied immediately prior to planting or at planting or at cultivation. The application rate is 6 oz. per 1000 linear feet, which is also the seasonal maximum limit. DYFONATE/0321 is applied in a 7 inch wide band. When applied during cultivation, do not apply within 45 days of harvest nor feed or graze livestock within 30 days of treatment.

The use directions for these corn racs differ significantly from the directions for use with sweet corn. With sweet corn, the maximum individual application rate is 0.04 lb ai/A, the maximum seasonal application rate is 0.48 lb ai/A, and the minimum PHI is 1 day. (See our 9/19/91 memo for PP#7F3560.)

Peanuts. Lambda-cyhalothrin in the EC formulation may be applied by ground or air at levels of 0.015-0.03 lb ai/A at intervals required by scouting, usually 7 or more days. Do not apply within 14 days of harvest. Do not apply more than 0.12 lb ai/A/season. Do not graze livestock in treated areas. Do not use treated vines or hay for animal feed.

Nature of the Residue

The nature of the residue in plants is adequately understood (PP#7F3560/7H5543, M. Flood, memo of 1/22/92). Lambda-cyhalothrin is metabolized by cleavage of the ester linkage to form cyclopropanecarboxylic acids and the corresponding phenoxybenzoic acids or alcohols. In most cases the parent compound is the principal constituent of the residue. A cabbage metabolism study indicated that in that rac the cis- and trans-cyclopropanecarboxylic acids were the major constituents.

CB-1 and TB-1 have decided that the plant metabolites need not appear in the tolerance expression at this time due to lack of toxicological concern and low concentrations found from residue studies. (See memo from P. Hurley to M. Flood, 1/3/92.) The residue to be regulated is lambda-cyhalothrin and its epimer.

The nature of the residue in animals is adequately understood. Lambda-cyhalothrin is the major component of the residue, except for kidney and liver of ruminants and liver of poultry. In addition to the plant metabolites, 3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2-hydroxymethyl-2-methylcyclopropanecarboxylic acid (OH-CPA) and 4-hydroxy-3-phenoxybenzoic acid (4'-OH-3PBAcid) may be present in significant quantities. Although OH-CPA has not been definitively identified as a rat metabolite, a residue transfer study in which cows were fed dietary levels of 8, 25 or 80 ppm lambda-cyhalothrin demonstrated that at the maximum level of lambda-cyhalothrin in the diet (≤ 8 ppm) OH-CPA levels in tissue would not exceed 0.01

8.

ppm (M. Flood, memo of 8/31/92). As in the case with plants, the residue to be regulated is lambda-cyhalothrin and its epimer.

Analytical Methods

Plants

The analytical method used to determine the magnitude of the residues of lambda-cyhalothrin and its epimer in plant matrices is ICI Method 81, first described in MRID # 400540-01. The method has successfully undergone EPA Method Validation for soybeans (PP#6F3318/PP#7F3488, E. Greer, memo of 9/30/87). Ground samples are extracted with acetone:hexane (1:1 v/v) and after several purification steps are analyzed by capillary GC. A more complete description of the method is given in our 9/19/91 memo for PP#7F3560/7H5543, PP#1F3952/1H5607, FAP#0H5599.

The analytical method used to determine the magnitude of the residues of the plant metabolites of lambda-cyhalothrin is given as Appendix 5.2 to the field corn magnitude of the residue report (MRID # 422537-03). The method was originally submitted in FAP#0H5599 under the title "Method for Analysis of Lambda-Cyhalothrin Metabolites in Hops" and discussed in detail in our 9/19/91 memo. In brief, samples are extracted with acetonitrile and then acetonitrile: pH 9 water 1:1 v/v. Parent and epimer are removed by C18 solid phase extraction and the metabolites are refluxed in 2N HCl for two hours to hydrolyze conjugates. Jones Reagent is added to oxidize alcohols to the corresponding acids and after several partitioning steps the metabolites are derivatized with trifluoroacetic anhydride and trifluoroethanol and analyzed by capillary GC using a mass selective detector.

Animals

The analytical method used to determine residues of lambda-cyhalothrin in meat, milk, poultry and eggs is ICI Method 86, reviewed in M. Firestone's memo of 1/22/86 (PP#6F3318). Lambda-cyhalothrin and its epimer are extracted from milk or animal tissue with 50 percent acetone:hexane. After removal of the aqueous layer, the organic fraction is cleaned up with a Florisil column prior to determination by GC using a ^{63}Ni electron capture detector. This method has undergone successful EPA Method Validation (PP#6F3318/7F3488, E. Greer, memo of 9/30/87; S. Brooks, memo of 10/30/87).

The analytical method used to determine residues of metabolites in meat, milk, poultry and eggs is ICI ARAM 96/1, dated March, 1990. An earlier version of this method, dated October, 1988, was used in the poultry residue transfer study (PP#1F3952, MRID # 417935-01). Both methods are described in detail in our memo of 9/19/91.

In the later version, milk, eggs, muscle and fat are extracted with acetonitrile, liver and kidney are extracted with either acetonitrile:1 M HCl (1:1) for CPA or methanol for 3-PBAcid and 4'-OH-3-PBAcid. Lambda-cyhalothrin and epimer are removed by C18 solid phase extraction, and the remaining solution is adjusted to 4 M HCl and refluxed for 4 hours. The hydrolysate is extracted with dichloromethane and the solution divided into two equal portions. One half is used for 4'-OH-3-PBAcid analysis; the other is used for CPA and 3-PBAcid analyses. After purification on an aminopropyl bonded silica column (for 4'-OH-3-PBAcid) or a C18 bonded silica column (other metabolites), the first metabolite is analyzed by HPLC using an electrochemical detector; and the metabolites in the second solution are derivatized using benzyl bromide. After purification on a Florisil column, the benzyl esters are quantitated by GC-MS-SIM.

The independent laboratory validation for ARAM 96-1 has been submitted with this petition. (We note that independent lab validation for metabolite analyses is only required if the metabolites must be regulated and EPA Method Validation is necessary. In this case, therefore, independent validation is not required as long as CB and TB do not consider it necessary that metabolites appear in the tolerance expression.)

The independent laboratory validation study was done at Battelle Laboratories, Columbus, Ohio. The following report was submitted:

> "Method Validation of ARAM 96-1 for Residues of Lambda-Cyhalothrin Metabolites in Animal Products," M. G. Schweitzer, 1/8/92, Battelle Study No.: SC900049. (MRID # 422537-02)

Battelle conducted its validation on liver only -- the matrix considered the most difficult to analyze. The laboratory initially assumed that after the separate extractions on liver -- acetonitrile:HCl or methanol -- the extracts should be combined prior to purification on the C18 SPE column. When this procedure was followed, however, the SPE column was overloaded and acceptable recoveries could not be obtained. Acceptable recoveries resulted only when each analyte was fortified and analyzed independently. Under these conditions the method required about 16 man-hours to complete one sample set.

Battelle has also recommended a slight modification in the derivatization step, although it "was not a problem per se".

Recoveries at 0.05 μg/g averaged 102.2%, 74.0% and 80.0% for 4'-OH-3-PBAcid, CPA and 3-PBAcid, respectively. Corresponding recoveries at 0.20 μg/g were 86.2%, 96.4% and 103.8%.

Comment

It is not clear from the report whether it was necessary to separately fortify and extract 4'-OH-3-PBAcid and 3-PBAcid. At this time no further action on the part of the registrant is necessary; however, should EPA Method Validation ever be necessary, ICI should amend its method to account for the above difficulties prior to our validation attempt.

Multiresidue Testing. The petitioner has determined recoveries of cyhalothrin under FDA's multiresidue protocols (PP#7F3488, S. Willett, memo of 3/15/88). ICI's report of recoveries of metabolites CPA and 3-PBAcid were recently forwarded to FDA (M. Flood, memo of 9/19/91).

Storage Stability

Lambda-cyhalothrin residues were shown to be stable in peach, pea, oil seed rape, wheat grain, sugarbeet root, cottonseed, apple, cabbage and potato when stored at -18°C for periods up to 26 months (M. Flood, memo of 9/19/91). Storage stability of lambda-cyhalothrin in animal matrices has been adequately demonstrated. See PP#7F3488, S. Willett, memo of 8/13/87 and N. Dodd, memo of 3/15/88 and PP#9F3770, M. Flood, memo of 1/25/90. Our review of 9/19/91 contains a complete summary of available storage stability data for lambda-cyhalothrin. We note that the available data for this compound would also cover the epimer.

Storage stability of CPA, 3-PBAcid and 3-PBAlcohol has been the subject of an ongoing study. Stability at 3 months in various matrices was reported in our 9/19/91 memo; stability at one year in these same matrices was discussed in our 3/23/92 memo for FAP#0H5599. ICI has now submitted the two year update:

> "Part II: Interim Report on Storage Stability of Pyrethroid Metabolites in Raw Agricultural Commodities (24-month Interval)," C.L. Eckstein and P.D. Francis, 6/30/92, Report No. RR 92-029B, Performing Laboratory: ICI Americas Western Research Center, Richmond, CA. (MRID # 423879-01)

In this study CPA, 3-PBAcid and 3-PBAlcohol as well as DCVA [cis,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane-carboxylic acid, a metabolite of permethrin] were analyzed in the following raw agricultural commodities at -20±5°C: apples, cabbage, corn fodder, corn forage, cotton seed, lettuce, peanut hulls, peanut meats, sorghum grain, soybeans, sugar beets, tomatoes and tobacco. All samples were fortified at 0.10 µg/g at zero-day. Each value reported is the average of the analyses of duplicate stored samples. Reported values were corrected for recovery of newly fortified control samples run at the time of analysis if the recovery of the fortified control sample was less

than 100%.

In most cases metabolite levels after two years were greater than 80% of the day-zero levels. The following exceptions are noted in Table 1.

Table 1

RACs Showing Significant Decline in Lambda-Cyhalothrin Metabolite Levels over a 24-Month Period

Metabolite		Average in µg/g			
	RAC	0-Day	3-Month	12-Month	24-Month
CPA	Peanut Meats	0.099	0.101	0.066	0.072
	Tomatoes	0.099	0.079	0.072	0.066
3-PBAcid	Cabbage	0.113	0.101	0.085	0.065
	Peanut Meats	0.092	0.096	0.067	0.072
	Tomatoes	0.109	0.069	0.077	0.072
3-PBAlcohol	Apples	0.106	0.106	0.080	0.078

In our opinion the observed decline in these RACS is within acceptable limits. The study will be extended to 36 months. Deficiencies in storage stability data, as noted in our 9/19/91 memo, are resolved for sweet corn (PP#7F3560/7H5543) and broccoli, cabbage and tomatoes (PP#1F3952/1H5607).

Residue Data

Field Corn and Popcorn. Residue data have been submitted in the following report:

> "ICIA0321: Magnitude of the Residue Study on Field Corn and Popcorn;" J.C. McKay and P.D. Francis; 9/30/91; Report No. RR 90-428B; Performing Laboratories: Huntingdon Analytical Services, Middleport, NY; Pharmacology and Toxicology Research Laboratory-West, Inc., Richmond, CA. (MRID # 422537-03)

Seventeen field trials with field corn were conducted in 1988 in IL, IN, IA, MI, MN, MO, NE, NC, OH, SD, and WI. According to Agricultural Statistics, 1988, these states produced 85.0% of the 1986 U.S. production of corn grain. In addition two trials with popcorn were held in IN and MO. Four applications of lambda-cyhalothrin were made at 0.03 lb ai/A: (1) from 5 days prior to planting up to 10 days after emergence, (2) at V6 vegetative stage, (3) at silking, and (4) at about 21 days before harvest (19-39 days). Forage samples were collected when the corn was at tasseling after two applications. Silage samples

12

were collected when corn was in the soft dough stage after three applications. Two to three plots were included in each trial -- three if both the EC and WG formulations were applied. One plot served as control. Aerial application (2-5 gallons of water per acre) was made in four trials.

Samples were placed on dry ice within 0.5 to 20 hours after collection. Analyses for parent and epimer were carried out at Huntingdon Analytical Services. Samples were extracted 325-477 days after harvest and analyzed 1-50 days after extraction. (Four grain samples were analyzed 50 days after extraction.) We previously noted that ICI has demonstrated the stability of parent in extracts for periods of 33-42 days. It is our opinion that available storage stability data are sufficient to support the analyses. Analyses for metabolites were done at Pharmacology and Toxicology Research Laboratory-West. Samples were extracted 583-678 days after harvest and analyzed 1-2 days after extraction. Available storage stability data are sufficient to support these analyses.

Percent recoveries of lambda-cyhalothrin from untreated forage, silage, grain and fodder averaged 98.7±19.9%, 90.1±18%, 90.9±10.6% and 91.2±15.6%, respectively. Fortification levels varied from 0.013 ppm to 4.2 ppm. Corresponding percent recoveries of the epimer were 99.8±19.2%, 92.5±17.3%, 90.9±10.6% and 86.4±10.7% at fortification levels of 0.017 ppm to 5.6 ppm.

Percent recoveries of CPA from untreated forage, silage, fodder and grain averaged 86.2±13.2%, 88.5±11.1%, 83.2±11.1% and 79.2±7.6%, respectively, at a fortification level of 0.05 ppm. Percent recoveries for 3-PBAcid were 81.5±10.7%, 82% (n=1), 80.2±5.6% in forage, silage, and fodder, respectively, at a fortification level of 0.05 ppm. Percent recoveries for 3-PB Alcohol were 89.3±7.1%, 100% (n=1), and 86.5±7.2% for silage, fodder and grain, respectively, at the same fortification level.

Results are given in the following tables.

Table 2

Residue Data for Lambda-cyhalothrin (ICIA0321), Epimer (R157836), CPA (PP890) and 3-PBAcid on Field Corn Forage

Location	Formu-lation	Appln. Rate (lb. ai/A)	PHI (days)	ICIA0321 (mg/kg)	R157836 (mg/kg)	PP890 (mg/kg)	3-PBAcid (mg/kg)
W. Liberty, IA	EC	2 x 0.03	32	0.04, 0.03	0.01, <0.01	0.01	<0.01
	WG			0.03	<0.01		
Galien, MI	WG		21	0.02	<0.01		
Mulkeytown, IL	EC		25	<0.01	<0.01	<0.01	<0.01
	WG		25	<0.01	<0.01		
Owatonna, MN	EC		27	0.01	<0.01	<0.01	<0.01
	WG			<0.01, <0.01	<0.01, <0.01		
Chariton, MO	WG		27	0.01	<0.01		
Kearney, NE	EC		31	<0.01	<0.01	<0.01	<0.01
	WG		31	<0.01	<0.01		
Martinsville, IN	EC		42	<0.01	<0.01	<0.01	<0.01
	WG		42	<0.01	<0.01		
Wooster, OH	WG		35	0.01	<0.01		
Waterloo, WI	EC		22	0.01	<0.01	<0.01	<0.01
	WG		22	0.01	<0.01		
Sioux Falls, SD	WG		32	0.03, 0.02	<0.01, <0.01		
Goldsboro, NC	WG		43	<0.01, <0.01	<0.01, <0.01		
Goldsboro, NC	EC		38	<0.01, <0.01	<0.01, <0.01	<0.01	<0.01
W.Des Moines, IA	EC		36	<0.01	<0.01	<0.01	<0.01
Wood River, NE	WG		22	<0.01	<0.01		
Geneseo, IL	WG		33	<0.01	<0.01		

Table 3

Residue Data for Lambda-cyhalothrin (ICIA0321), Epimer (R157836), CPA (PP890) and 3-PBAcid on Field Corn Silage

Location	Formu-lation	Appn. Rate (lb ai/A)	PHI (days)	ICIA0321 (mg/kg)	R157836 (mg/kg)	PP890 (mg/kg)	3-PBAcid (mg/kg)
W. Liberty, IA	EC	3 x 0.03	18	0.06	<0.01	0.01	0.01
	WG		18	0.12	<0.01		
Galien, MI	WG		22	0.02	<0.01		
Mulkeytown, IL	EC		26	0.17	0.02	0.02	0.01
	WG		26	<0.01	<0.01		
Owatonna, MN	EC		36	0.02, 0.02	<0.01, <0.01	0.01	<0.01
	WG		36	0.02	<0.01		
Kearney, NE	EC		27	0.04	<0.01	<0.01	<0.01
	WG		27	0.02	0.02		
Martinsville, IN	EC		40	0.05, 0.05, 0.02	<0.01, <0.01. <0.01	0.01	<0.01
	WG		40	<0.01	<0.01		
Wooster, OH	WG		1	0.10, 0.07	<0.01, <0.01		
Waterloo, WI	EC		37	0.02	<0.01	0.01	0.01
	WG		37	0.01, 0.01	<0.01, <0.01		
Sioux Falls, SD	WG		21	0.14	0.02		
Goldsboro, NC	WG		17	0.05	<0.01		
W.Des Moines, IA	EC		25	0.56	0.04	0.03, 0.03	0.02, 0.03
Wood River, NE	WG		39	0.01	<0.01		
Geneseo, IL	WG		14	<0.01	<0.01		

Table 4

Residue Data for Lambda-cyhalothrin (ICIA0321), Epimer (R157836), CPA (PP890) and 3-PBAcid on Field Corn Fodder

Location	Formu-lation	Appl. Rate (lb ai/A)	PHI (days)	ICIA0321 (mg/kg)	R157836 (mg/kg)	PP890 (mg/kg)	3-PBAcid (mg/kg)
W. Liberty, IA	EC	4 x 0.03	21	0.22	0.02	0.04	0.02
	WG		21	0.10	<0.01		
Galien, MI	WG		21	0.15	0.01		
Mulkeytown, IL	EC		21	0.24	0.02	0.02, 0.03	0.01, 0.01
	WG		21	0.19	0.02		
Owatonna, MN	EC		21	0.23	0.04	0.03, 0.03	0.01, 0.01
	WG		21	<0.01	<0.01		
Cariton, MO	WG		21	0.34	0.05		
Kearney, NE	EC		25	0.20	0.02	0.01, 0.02	0.01, 0.01
	WG		25	0.13	<0.01		
Martinsville, IN	EC		39	0.13	0.02	<0.01	<0.01
	WG		39	0.11	0.02		
Wooster, OH	WG		25	0.28	0.03		
Waterloo, WI	EC		21	0.40	0.06	0.04, 0.08	0.04, 0.04
	WG		21	0.13	0.01		
Sioux Falls, SD	WG		21	0.23	0.04		
Goldsboro, NC	WG		20	0.05	<0.01		
Goldsboro, NC	EC		21	0.19	0.03	0.02, 0.04	0.01, 0.01
W.Des Moines, IA	EC		21	<0.01	<0.01	<0.01	<0.01
Wood River, NE	WG		24	0.12	0.01		
Geneseo, IL	WG		19	0.18	0.03		

Corn grain, sampled at the same time as fodder, failed to show any residue (<0.01) for all analytes.

In two field trials, lambda-cyhalothrin was applied to popcorn. Results are given in Table 5.

Table 5

Residues of Lambda-cyhalothrin (ICIA0321), Epimer (R157836), CPA (PP890) and 3-PBAcid on Popcorn Grain, Forage and Fodder

Location	RAC	Formu-lation	Appln. Rate (lb ai/A)	PHI (days)	ICIA0321 (mg/kg)	R157836 (mg/kg)	PP890 (mg/kg)	3-PBAcid (mg/kg)
Hebron, IN	Forage	EC	2 x 0.03	32	<0.01	<0.01	<0.01	<0.01
	Fodder		4 x 0.03	21	0.14	0.02	0.01	0.01
	Grain		4 x 0.03	21	<0.01	<0.01	<0.01	<0.01
Chariton, MO	Forage	WG	2 x 0.03	27	0.01	<0.01		
	Fodder		4 x 0.03	21	0.18	0.03		
	Grain		4 x 0.03	21	<0.01	<0.01		

Based on these data, the appropriate tolerances are 1.0 ppm for forage and fodder and 0.05 for grain. ICI has proposed a tolerance of 3.0 ppm for fodder based on preliminary results submitted in MRID # 422537-01 (this petition). Seven field trials were carried out in IA, IL, IN, MN, NE, PA, WI. Field corn plots received five applications instead of four. The additional application was made at planting by in-furrow treatment with a premix of 20% fonofos + 0.4% lambda-cyhalothrin at a rate of 6 oz premix/1000 linear row feet with a 20 inch row spacing. This would correspond to 0.04 lb lambda-cyhalothrin per acre. The total application was therefore 0.16 lb lambda-cyhalothrin/A. (Note that the lambda-cyhalothrin applied from the premix is erroneously listed as 0.004 lb/A in ICI's report.) ICI's results, which have not been formally submitted, show that the average residue of lambda-cyhalothrin on corn fodder is 0.62 ppm and the maximum residue found was 2.22 ppm. The average epimer residue was 0.08 ppm. ICI believes that the 2.22 ppm value "could have been obtained from the foliar treatment alone, and ICI is therefore considering this value in assessing a proposed tolerance".

Comment

CBTS is reluctant to use these preliminary data in assessing

the field corn tolerance, for we are unable to verify their accuracy. However, examination of the data for forage and fodder reviewed above shows a definite correlation with residue levels and number of applications. It is not surprising that five applications would produce higher residue levels. Although pyrethroids are mostly non-systemic, in-furrow applications could produce locally higher concentrations than general broadcast applications. We conclude that the appropriate tolerance for field corn forage and fodder should be 1.0 ppm. ICI should submit a revised Section F in which a tolerance of 1.0 ppm for residues of lambda-cyhalothrin in/on corn fodder is proposed. Registration of an additional soil treatment using the fonofos/lambda-cyhalothrin premix may require field trials in addition to the seven already carried out.

Grain Dust

Residue data on corn grain dust have not been submitted. Instead, ICI argues that the concentration factor for lambda-cyhalothrin residues on sorghum grain to residues on grain dust is at least as great as the concentration factor for residues on field corn grain to residues on field corn grain dust. The reasoning is as follows:

1. Lambda-cyhalothrin is foliarly applied to both field corn and sorghum.

2. The pesticide is non-systemic, so residues are primarily on the crop surface.

3. Field corn grain is protected from lambda-cyhalothrin residues by the corn husk.

4. Late season applications of lambda-cyhalothrin to sorghum result in direct treatment of the grain. Sorghum grain is not protected from residues by any type of plant sheath, whereas corn grain is protected by the husk.

Comment

It is our opinion that because residues on grain are non-detectable, neither residue data on grain dust nor a tolerance for lambda-cyhalothrin on grain dust should be required. If in the future the pesticide is used in such a manner that detectable residues are expected on grain, then grain dust data will be required.

ICI should revise its Section F to delete the proposed tolerance on grain dust.

Processing Study

The following corn processing study was submitted:

"ICIA0321 (Lambda cyhalothrin) - Magnitude of the Residue Study on Processed Field Corn Products," C.L. Grant and P.D. Francis, 11/7/91, Lab. Project ID 0321-88-PR-03, Report No. RR 91-027B. (MRID # 422537-04)

The analytical chemistry was done at ICI Americas Inc.'s Western Research Center in Richmond, CA and Huntingdon Analytical Services in Middleport, NY. The corn was processed at the Food Protein Research and Development Center, Texas A&M, College Station, TX.

Field corn grown near Waverly, NE was treated at 4 x 0.3 lb ai/A -- 10 times the maximum proposed use level. The first application was made at the time of planting; the last application was made 23 days before harvest. Samples were dried for 16 days at 60° - 75°F, then transported to Texas for processing. Prior to processing, a subsample was sent to ICI for analysis. Residues found on field corn grain were lambda-cyhalothrin <0.02 mg/kg, R157836 <0.01, PP890 0.01 mg/kg and 3-PBAcid, <0.01.

Samples were shipped unfrozen to Texas A&M where they were placed in a freezer (20±10°C) until processing, 7 months later. Grain was wet milled into hulls, germ, gluten, coarse gluten starch, starch, expelled crude oil, refined oil, refined bleached oil, refined bleached deodorized oil, expelled presscake, solvent extracted presscake and soapstock. Grain was dry milled into large, medium and small grits; hulls; germ; coarse meal; meal; flour; solvent extracted presscake; expelled presscake; refined oil; expelled crude oil; solvent extracted crude oil; refined bleached deodorized oil; and soapstock. A description of the two procedures -- with flow diagrams -- is given in the petition.

Samples were analyzed for parent and epimer 3-5 months after processing and for metabolites 15-17 months after processing. **There are no storage stability data available for corn processed fractions.**

Percent recoveries of lambda-cyhalothrin and epimer from corn processed fractions at fortification levels of 0.042 ppm and 0.42 ppm for parent and 0.057 and 0.57 ppm for epimer averaged 90.6±15.9% and 87.7±15.5%, respectively. Percent recoveries of 3-PBAcid/3-PBAlcohol and CPA (PP890) averaged 88.1±11.4% and 87.8±16.8%, respectively. Analyses for parent and epimer were done at Huntingdon Analytical Services; analyses for metabolites were done at ICI's Western Research Center, although no explicit statement is made.

Dry Mill Processing. No parent was found in treated corn grain at a level of 0.02 ppm. Parent was found in flour at 0.03 ppm.

Neither epimer nor 3-PBAcid were detected in corn grain or any processed product. PP890 was found in treated corn grain at 0.01 ppm. Concentration occurred in crude oil - 0.02 ppm. Levels in refined oil were non-detected.

Wet Mill Processing. Residues of lambda-cyhalothrin, epimer, or 3-PBAcid were not detected in grain or any processed fraction. PP890 was not detected in grain (<0.01 ppm) but was found in crude oil at a level of 0.03 ppm. Residues in refined oil were non-detected.

Comment

Concentration of parent occurs in flour. Since the reported limit of detection in grain was 0.01 ppm, 0.03 ppm was found in flour, and the RAC (grain) tolerance is 0.05 ppm, the appropriate food additive tolerance would be (0.03/0.01) x 0.05 = 0.15 ppm. **ICI should submit a revised Section F in which this food additive tolerance is proposed for corn flour.** (The proposed tolerance on grain -- 0.05 ppm -- is higher than the limit of quantitation. If it becomes necessary to determine "anticipated residues" for lambda-cyhalothrin, we will use the limit of quantitation or limit of detection as a more realistic estimate of average dietary exposure.)

Peanuts. ICI has submitted the following report:

"Lambda Cyhalothrin (ICIA0321): Magnitude of the Residue Study on Peanuts;" J.C. McKay; 5/16/91; Performing Laboratories: ICI Americas Inc.'s Western Research Center, Richmond, CA, Huntingdon Analytical Services, Middleport, NY and Pharmacology and Toxicology Research Laboratory-West, Inc., Richmond, CA; Laboratory Project ID Nos. 6-321-87-01, -02, -03; Report Number RR 90-420B. (MRID # 422732-02)

Twelve field trials were conducted during 1987 in GA(3), TX(2), AL(2), NC(2), VA, OK, and FL. According to Agricultural Statistics, 1988, these states produced over 98% of peanuts grown in the U.S. in 1986. Peanut plots received four applications of the EC formulation at an application rate of 0.03 lb ai/A. The pesticide was applied in 8.0 to 27.9 gallons of water per acre for ground applications and 1 to 4 gallons of water per acre for aerial applications. PHI's varied from 13 to 28 days (average - about 18 days). Samples were frozen prior to shipping to wither the Eastern Research Center in NC or the Western Research Center in CA.

In addition, trials were carried out in AL and NC at an application rate of 4 x 0.12 lb ai/A (4X). Samples were taken 20 or 21 days after final application.

Frozen whole peanut samples were separated into nut meats and hulls. Analysis for residues of parent and epimer were conducted at Huntingdon Analytical Services; analysis for residues of PP890, 3-PBAcid and 3-PBAlcohol were conducted by the Pharmacology and Toxicology Research Laboratory-West, Inc. Nutmeat and hull samples were analyzed for parent and epimer 17-20 months after sampling and for metabolites 30-31 months after analysis. **Available storage stability data support the parent and epimer analyses but not the metabolite analyses.** The reported 24 month storage stability study for metabolites is an ongoing study and, presumably, additional data will be forthcoming.

Percent recoveries from peanut hulls fortified with lambda-cyhalothrin at 0.021 ppm or 0.21 ppm averaged 88.1±8.0%. Percent recoveries from peanut meats at these two fortification levels averaged 84.0±5.4%. Percent recoveries of epimer at fortification levels of 0.028 ppm or 0.28 ppm averaged 90.5±8.9% for hulls and 84.8±8.5% for meats. Recoveries of PP890, 3-PBAcid and 3-PBAlcohol at 0.05 ppm averaged 98.7±21.4, 75.0±4.1% and 83.6±9.7%, respectively. (The averages include individual results from nutmeat and hulls.)

Neither lambda-cyhalothrin nor its epimer could be determined in any sample (treated at a 1X rate)at a level of 0.02 ppm. Neither PP890 nor 3-PBAcid (which includes PBAlcohol) could be detected at a level of 0.01 ppm. (Samples from six field trials were analyzed for metabolites.)

Detectable residues were found in samples treated at the 4X rate. Lambda-cyhalothrin was found at 0.07 ppm in hulls from the NC trial; PP890 was found at 0.013 ppm in hulls from the AL trial and at 0.03 ppm in the NC trial. Epimer was detected at 0.01 ppm in hulls from the NC trial. 3-PBAcid remained non-detected.

The residue data support the proposed tolerances of 0.05 ppm for residues of lambda-cyhalothrin (+ epimer) in/on peanut meat and peanut hulls.

Processing Study

The following peanut processing study was submitted:

"Lambda-cyhalothrin - Magnitude of the Residue Study on Processed Peanut Products;" J.C. McKay and P.D. Francis; 11/7/91; Performing Laboratory: ICI Americas Inc., Western Research Center, Richmond, CA; Laboratory Project ID's 6-321-87-03, 0321-90-PR-02; Report No. RR 91-047B. (MRID # 422732-03).

Peanuts from the NC trial treated at the 4X rate were

processed at the Food Protein Research and Development Center, Texas A&M University, College Station, TX. Peanut meats were processed into meal; crude oil; refined oil; refined, bleached and deodorized oil; and soapstock. Samples were analyzed 217-400 days after processing. As noted in the corn processing study, storage stability data are lacking on processed products.

Percent recoveries of lambda-cyhalothrin fortified into the various processed fractions at levels of 0.0258 ppm to 0.0813 ppm averaged 97.8±18.6%. Percent recoveries of epimer at levels of 0.0197 to 0.107 ppm averaged 100.7±14.6%. Corresponding recoveries of PP890, 3-PBAcid and 3-PBAlcohol at levels of 0.02 ppm or 0.05 ppm averaged 86.3±13.6, 78.8±4.0% and 67.9±7.0%, respectively. Submitted chromatograms are acceptable.

Concentration of lambda-cyhalothrin was observed in crude and refined oil. Because levels in nutmeat were <0.01 ppm, it is impossible to determine an accurate concentration factor. Levels in crude and refined oil were 0.01 ppm and 0.02 ppm, respectively. Levels in refined, deodorized and bleached oil were <0.01 ppm. Since food oils are typically bleached and deodorized (steam stripping under vacuum), we conclude that a food additive tolerance is not necessary.

PP890 levels in soapstock -- 0.02 ppm -- imply that the metabolite concentrates from an initial level of <0.01 ppm. The petitioner speculates that rather than concentrate, PP890 is formed from base hydrolysis of lambda-cyhalothrin in refined oil in the preparation of soapstock. At any rate, because these metabolites are not being regulated at this time, no feed additive tolerance is necessary.

Storage Stability

Presently there are three active petitions having analyses for processed products -- in addition to the two reviewed in this memo PP#7F3488 includes analyses for soybean processed products. To satisfy storage stability requirements for the analyses of these processed commodities, storage stability data are necessary on three commodities, two of which should be peanut oil (crude or refined) and peanut meal. Storage stability data reviewed earlier in this memo indicates a possible stability problem for metabolites in peanut products. If lambda-cyhalothrin, epimer, or any metabolite proves to be unstable in any of these commodities, additional storage stability data generated using other processed commodities may be necessary.

Meat, Milk, Poultry and Eggs

In our 9/1/92 memo for PP#7F3560/7H5543 the following tolerances for residues of lambda-cyhalothrin and its epimer

22

in/on animal commodities were required:

Fat of cattle, goats, hogs, horses and sheep.....	3.0	ppm
Meat and mbyp of cattle, goats, hogs, horses and sheep	0.2	ppm
Meat, mbyp and eggs of poultry	0.01	ppm
Fat of poultry	0.02	ppm
Milk, fat (reflecting 0.2 ppm in whole milk).....	5.0	ppm

The cattle tolerances were determined from diets containing 25% of sweet corn forage, for which the proposed tolerance is 5.0 ppm. A diet consisting of field corn forage instead of sweet corn forage would produce lower exposure to lambda-cyhalothrin. For this reason proposed meat and milk tolerances remain unaffected by this petition. Similarly, the non-detectable residue levels found in corn grain would produce non-detectable residues in poultry products, and hence, the maximum predicted residue would not be affected. The same argument applies to the dietary contribution from peanut commodities.

Other Considerations

An International Residue Limit Status sheet is appended to this review. There are no Canadian or Mexican limits; there are Codex limits for cyhalothrin on other RACs. Compatibility is not an issue.

Attachment: International Residue Limit Status sheet.

cc: SF, RF, Circu., Mike Flood, E. Haeberer, PP#2F4109, PP#2F4114, PP#7F3560, PP#1F3952.

H7509C:CBTS:Reviewer(MTF):CM#2:Rm800A:305-6362:typist(mtf):11/18/92.
RDI:SectionHead:ETHaeberer:11/13/92:BranchSeniorScientist:RALoranger: 11/18/92.