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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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EXPEDITE

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: 010501 - Dicofol
Rohm and Haas Response to Registration Standard
Residue Data and Feeding Studies
EPA File Symbol 707-ENE
[MRID Nos. 400420-01 to -02 and -09 to -31, RCB No. 2578]

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THRU: Edward Zager, Section Head
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This review is being expedited at the request of E. Tinsworth, Director, Registration Division. The expedite request includes review of studies from this submission not previously reviewed. Our earlier review (S. Hummel, 5/27/87) included plant and animal metabolism, analytical methods, and screening of residue data for residues reported on the day of application. The expedited due date is 10/19/87.

The following data are included in this review.

MRID NO.	STUDY TITLE AND REPORT NO.
400420-01	Determination of the Octanol/Water Coefficient of 14C-p,p'-Dicofol, Rohm and Haas Technical Report No. 310-86-36, A. M. Tillman (Rohm and Haas) and D. Teeter (Analytical Bio-Chemistry Laboratories), June 9, 1986.

MRID NO.	STUDY TITLE AND REPORT NO.
400420-02	Determination of the Octanol/Water Coefficient of 14C-o,p'-Dicofol Rohm and Haas Technical Report No. 310-86-37, A. M. Tillman (Rohm and Haas) and D. Teeter (Analytical Bio-Chemistry Laboratories), June 9, 1986.
400420-09	Interim Report On the Stability of o,p'-Dicofol in Cottonseed Products under Frozen Storage Conditions After 18 Months, Rohm and Haas Technical Report No. 310-86-51, R. J. Pollock (Analytical Development Corporation) and C. K. Hofmann (Rohm and Haas), October, 1986.
400420-10	Interim Report on the Stability of o,p'-Dicofol in Cottonseed Products under Frozen Storage Conditions (10 Months), Rohm and Haas Technical Report No. 310-85-46, R. J. Pollock (Analytical Development Corporation) and C. K. Hofmann (Rohm and Haas), October, 1986.
400420-11	A Study on the Stability of Dicofol and its o,p' Isomer (o,p'-Dicofol) on Citrus in A Frozen Storage Environment: One Year Report, Rohm and Haas Technical Report No. 310-86-24, C. K. Hofmann (Rohm and Haas), July, 1986.
400420-12	Kelthane Residues in Citrus, Rohm and Haas Technical Report No. 31A-86-81, Lorna S. Mazza, 1986 (date not specified).
400420-13	Kelthane Residues in Citrus, Rohm and Haas Analytical Report No. 31A-86-85, Lorna S. Mazza, 1986 (date not specified).
400420-14	Kelthane Residues in Apples, Rohm and Haas Analytical Report No. 31A-86-68, Lorna S. Mazza, 1986 (date not specified).
400420-15	Kelthane Residues in Pears, Rohm and Haas Analytical Report No. 31A-86-79, Lorna S. Mazza, 1986 (date not specified).
400420-16	Kelthane Residues in Pears, Rohm and Haas Analytical Report No. 31A-86-87, Lorna S. Mazza, 1986 (date not specified).

MRID NO.	STUDY TITLE AND REPORT NO.
400420-17	Kelthane Residues in Dry Beans, Rohm and Haas Analytical Report No. 31A-86-64, Lorna S. Mazza, 1986 (date not specified).
400420-18	Kelthane Residues in Melons, Rohm and Haas Analytical Report No. 31A-86-55, Lorna S. Mazza, 1986 (date not specified).
400420-19	Kelthane Residues in Melons, Rohm and Haas Analytical Report No. 31A-86-88, Lorna S. Mazza, 1986 (date not specified).
400420-20	Kelthane Residues in Cucumbers, Rohm and Haas Analytical Report No. 31A-86-86, Lorna S. Mazza, 1986 (date not specified).
400420-21	Kelthane Residues in Squash, Rohm and Haas Analytical Report No. 31A-86-89, Lorna S. Mazza, 1986 (date not specified).
400420-22	Kelthane Residues in Pecans, Rohm and Haas Analytical Report No. 31A-86-83, Lorna S. Mazza, 1986 (date not specified).
400420-23	Kelthane Residues in Walnuts, Rohm and Haas Analytical Report No. 31A-86-84, Lorna S. Mazza, 1986 (date not specified).
400420-24	Kelthane Residues in Grapes, Rohm and Haas Analytical Report No. 31A-86-90, Lorna S. Mazza, 1986 (date not specified).
400420-25	Kelthane Residues in Cottonseed, Rohm and Haas Analytical Report No. 31A-86-76, Lorna S. Mazza, 1986 (date not specified).
400420-26	Kelthane Residues in Processed Apple, Rohm and Haas Technical Report No. 31D-86-48, L. S. Mazza, 1986 (date not specified).
400420-27	Kelthane Residues in Processed Cotton Rohm and Haas Technical Report No. 310-86-42, L. S. Mazza, 1986 (date not specified).
400420-28	Kelthane Residues in Processed Grape Products Rohm and Haas Technical Report No. 310-86-66 L. S. Mazza, 1986 (date not specified).

MRID NO.	STUDY TITLE AND REPORT NO.
400420-29	Kelthane Residues in Processed Citrus Products Rohm and Haas Technical Report No. 310-86-67 L. S. Mazza, 1986 (date not specified).
400420-30	A Feeding Study with Cows Dosed with Technical Kelthane - Preliminary Report on the Analysis of Tissue and Milk Samples, Rohm and Haas Technical Report No. 310-86-57, A. M. Tillman (Rohm and Haas), L. Predmore and S. Shaffer (Analytical Bio-Chemistry Laboratories), November, 1986.
400420-31	A Feeding Study with Hens Dosed with Technical Kelthane - Preliminary Report on the Analysis of Tissue and Egg Samples, Rohm and Haas Technical Report No. 310-86-56, A. M. Tillman (Rohm and Haas), C. Jameson and S. Shaffer (Analytical Bio-Chemistry Laboratories), November, 1986.

Residue studies were not submitted for hops, spent hops, apricots, nectarines, peaches, caneberries, cherries, plum (fresh prunes), snap beans, lima beans (succulent), tomatoes, peppers, figs, field corn, alfalfa, clover, processed tomatoes, bean forage and hay or bean cannery waste, corn forage and fodder, cottonseed forage, and cotton gin trash. These studies are still needed. Alternatively, these crops may be removed from all dicofol labels. We note that a protocol for residue studies on succulent beans and hops has been received from J. R. Simplot Company (L. Cheng, memo of 9/25/87, RCB No. 2579).

Residue studies were not submitted for mint and strawberries. However, the Registration Standard concluded that additional residue studies were not needed for mint and strawberries, provided that plant metabolism studies show that dicofol, per se, is the residue of concern.

TOLERANCES

Tolerances for dicofol [1,1-bis(p-chlorophenyl)-2,2,2-trichloroethanol] have been established (40 CFR 180.163) on a variety of crops. The tolerances are expressed in terms of dicofol, per se. No tolerances have been established on meat, milk, poultry, or eggs. No food or feed additive tolerances have been established.

CONCLUSIONS

1. A tolerance reassessment cannot be done at this time. Registration Standard data deficiencies must be resolved before a tolerance reassessment can be done. Metabolism issues must be resolved. Registered and proposed uses must be supported by residue data. Residue data have not been submitted for all types of formulations on all crops. If dicofol is determined to be the sole residue of concern in plants, the deficiencies in the residue data resolved, and only formulations and types of applications for which data have been submitted are to be supported, then increased tolerances will be needed for apples, pears, and grapes. Tolerances could possibly be lowered for melons, cucumbers, squash, pecans, and walnuts. No conclusion can be made regarding tolerances in dry beans and cottonseed due to severe deficiencies in the submitted residue data.

Food and feed additive tolerances will be needed for a number of commodities. The estimated tolerance levels are in parentheses following the commodity. Food additive tolerances will be needed for citrus peel (100 ppm) and oil (1000 ppm), raisins (100 ppm), and cotton-seed oil (level cannot be determined). Feed additive tolerances will be needed for apple pomace (250 ppm), raisin waste (100 ppm), and grape pomace (20 ppm). No conclusion can be made on the need for food and feed additive tolerances for cottonseed hulls, meal, or soapstock, since cottonseed with non-detectable residues were processed. See Residue data section for further information.

Since no residue data have been submitted for apricots, nectarines, peaches, caneberries, cherries, plum (fresh prunes), tomatoes, peppers, figs, field corn, alfalfa, or clover, tolerances for these crops should be revoked. (Another registrant has submitted protocols for residue field trials on succulent beans and hops.)

Tolerances for meat, milk, poultry and eggs will be needed. However, we cannot determine appropriate tolerance levels until metabolism issues are resolved, and until complete animal feeding studies are submitted.

2. The directions for use on all registered labeling and all proposed labeling must be changed to reflect the uses for which residue data were submitted or residue data must be submitted for all uses on registered and proposed labels. See further discussion in Conclusion 6 (Residue data).

All labeled uses must be supported by residue data or the labels amended to reflect the maximum use supported by residue data. Residue data must be submitted to support the

maximum number of applications allowed on the label, or the maximum number of applications allowed on the label must be changed to reflect the use supported by the submitted residue data. A maximum number of applications per season or a maximum quantity of pesticide to be applied per season must be specified on the label. A minimum interval between applications must be added to the labels. Product labels must be changed to allow dilute sprays and ground application only or residue data must be submitted for both dilute and concentrate sprays, ground and aerial application, as allowed on product labels. Use directions for orchards must be changed to account for the variability in tree sizes. Several options for orchard labeling are shown in Attachment 1 of our previous review (S. Hummel, 5/27/87). Grazing restrictions must also be added for orchards. Suggested language is "Do not allow livestock to graze in treated areas or feed on orchard cover crops." Alternatively, tolerances may be proposed for orchard cover crops and grazing allowed only on orchard cover crops for which tolerances have been established.

Product labels must be changed to allow use only of formulations for which residue data have been received or residue data must be supplied for each type of formulation to be used, i.e., Emulsifiable Concentrate (EC), Wettable Powder (WP) or Flowable Concentrate (F), Granular (G), and Dust (D).

No residue data on an emulsifiable concentrate formulation were submitted for apples, pears, grapes, melons, cucumbers, or squash. Data are required for the emulsifiable concentrate formulation on these crops. Alternatively, these crops may be removed from labels for EC products.

No residue data from the use of a wettable powder or flowable formulation were submitted for citrus, dry beans, pecans, walnuts, and cottonseed. Residue data are required for the wettable powder or flowable formulation on these crops. Alternatively, these crops may be removed from proposed wettable powder and flowable formulation labels.

Residue studies were not submitted for mint and strawberries. However, the Registration Standard concluded that additional residue studies were not needed for mint and strawberries, provided that plant metabolism studies show that dicofol, <u>per se</u>, is the residue of concern.

The volume of spray used in the residue field trials was not reported in the residue field trial reports. The volume of spray must be reported for each residue field trial. Residue data will support only the volume (dilute, concentrate, ULV) and type of application for which data were supplied. (Data on dilute applications support dilute

applications on the label; data on concentrate applications support concentrate applications on the label; etc.)

The following uses appear to be supported by residue data, pending resolution of the deficiencies in the residue data, as discussed in the residue data section of this review. The rates, PHI, and type of formulation which appear to be supported are tabulated below.

Table 2

Ground Application Uses Supported by Residue Data [1/]

Crop	Formulation	Maximum Rate	PHI (days)
Citrus[2/]	EC	3 x 6 lb ai/A	7
Apples[2/]	F	3 x 2.25 lb ai/A	7
Pears[2/]	F	3 x 3 lb ai/A	7
Grapes[2/]	F	2 x 1.2 lb ai/A	7
Dry Beans[3/]	F	2 x 1.5 lb ai/A	20
Melons	F	3 x 0.6 lb ai/A	2
Cucumbers	F	3 x 0.6 lb ai/A	2
Squash	F	4 x 0.6 lb ai/A	2
Pecans	EC	2 x 2 lb ai/A	7
Walnuts	EC	2 x 2 lb ai/A	7
Cottonseed [4/]	EC	none supported	

1/ Pending resolution of residue data deficiencies and establishment of higher tolerances. The volume of spray (dilute, concentrate, ULV) supported by the residue data is not known.

2/ Higher tolerance needed.

3/ Tolerance for bean forage and hay needed (or tolerance for bean cannery waste). Additional data needed for dry beans (without pods).

4/ Delinted cottonseeds were analyzed. No data were submitted for undelinted cottonseed.

Complete residue data must be submitted for aerial applications to all crops or aerial applications should be prohibited on the labels.

3. The metabolism of dicofol in plants is not adequately understood. The grapefruit, cottonseed, and bean metabolism studies submitted are incomplete. Dicofol was reported as the major residue, but its identification was not confirmed. A

full discussion of the metabolism studies was included in our previous review (S. Hummel, 5/27/87).

About 40% of the residue in grapefruit was not identified (10% or more "polar compounds", 30% not extracted). We deferred to TOX and EEB on the need for further identification of the 40% unidentified residue.

4. The metabolism of dicofol in lactating goats and laying hens may be considered adequately understood, depending on TOX and EEB considerations. We await a response from TOX and EEB on our deferrals.

The major dicofol metabolite reported in lactating goats and laying hens is FW-152 (1,1-bis(chlorophenyl)-2,2-dichloroethanol). Minor metabolites are dichlorobenzophenone and dichlorobenzhydrol. Unmetabolized dicofol is also present. Little if any dicofol is metabolized to DDE in lactating goats or laying hens.

Only 50% of the TRR was extracted from goat and poultry liver. Base and enzyme hydrolysis of liver were not attempted. Egg yolks were 70% extracted. Other tissues, egg whites, and whole eggs were 80 to 100% extracted. We noted for TOX and EEB that base and enzyme hydrolysis could have increased the extraction of radioactive residues from liver and egg yolk.

In rats, the major metabolite was FW-152. FW-152 was further metabolized to dichlorobenzophenone (DCBP) and dichlorobenzoic acid (DCBA). Very little DDE was found (W. Phang, TOX, memo of 5/27/87).

Note to TOX: The rat metabolite identified as dichlorobenzoic acid (DCBA) is incorrectly named. The structure given corresponds to 2,2-bis(chlorophenyl)-2-hydroxyacetic acid.

5. Final conclusions cannot be made on the adequacy of the analytical methodology to determine the residue of concern until the plant and animal metabolism of dicofol are adequately understood. Plant methodology was discussed in our previous review. Deficiencies from our previous review are still outstanding. (S. Hummel, 5/27/87).

5a. Methodology for animal products was included in this submission. The sample workup is similar to the PAM I method for chlorinated hydrocarbons. The submitted methodology can only be adequate if the residue of concern in meat, milk, poultry, and eggs is determined to consist of dicofol, dichlorobenzophenone, and FW-152 (1,1-bis(chlorophenyl)-2,2-dichloroethanol. Our final conclusion on the animal product methodology is reserved

until the animal metabolism is considered adequately understood.

5b. The analytical methodology used for the development of residue data must be submitted or a reference to the method used (for methods already in our files) must be submitted. We assume that the analytical method used for the residue data was TR-310-86-74. However, this was not clear.

6. Submitted residue data included analyses for only p,p'- and o,p'- dicofol. We cannot conclude that these data are adequate until the metabolism issues and discrepancies between the residue data and labeling are resolved.

Residue data were submitted for citrus, apples, pears, grapes, dry beans, melons, cucumbers, summer squash, pecans, walnuts, and cotton. A number of deficiencies were found to be common to all studies. The common deficiencies are discussed here rather than for each individual study.

6a. No analysis of the pesticide formulation used in the field trials was included. This analysis is required for each field trial to determine if any impurities may present a residue problem.

6b. The interval between treatments is needed for each field trial.

6c. The volume of spray solution used per acre is needed so that we may determine whether dilute or concentrate sprays were used.

6d. Clarification is needed of the number of samples from each location. It is unclear if several (2-4) samples from each location were taken, and the results averaged, or if one sample was taken from each location, with several analyses of that sample and the results averaged. Results from separate samples should not be averaged.

6e. Complete sample storage information is needed for each sample from the time of harvest until analysis.

6f. The analytical method used for the residue data must be identified and submitted. The analyses were reportedly done be a method based on Rohm and Haas TR 36-81-05. Neither the method used, nor Rohm and Haas TR 36-81-05 were included.

6g. Complete sample calculations must be submitted. Sample calculations were reportedly included in Rohm and

Haas TR # 36-81-05, which was not included in this submission.

6h. Recoveries should be determined each time samples are analyzed and at levels similar to those expected in the crop samples. These recoveries should be used to correct the analytical results for recovery.

6i. Residues on many crops increased with increasing PHI. For this reason, we question the sampling techniques used. The registrant should completely describe all sampling and subsampling techniques used.

6j. No data on an emulsifiable concentrate formulation were submitted for apples, pears, grapes, melons, cucumbers, or squash. Data are required for the emulsifiable formulation on these crops. Alternatively, these crops may be removed from labels for EC products.

6k. No data from the use of a wettable powder or flowable formulation were submitted for citrus, dry beans, pecans, walnuts, and cottonseed. Data are required for the wettable powder or flowable formulation on these crops. Alternatively, these crops may be removed from proposed labels for wettable powder or flowable formulations.

6l. Residue studies were not submitted for hops, spent hops, apricots, nectarines, peaches, caneberries, cherries, plum (fresh prunes), snap beans, lima beans (succulent), tomatoes, peppers, figs, field corn, alfalfa, clover, processed tomatoes, bean forage and hay or bean cannery waste, corn forage and fodder, and cottonseed forage. These studies were required by the Registration Standard, and are still needed. The PM should take appropriate action regarding the non-submission of these data.

6m. Residue studies were not submitted for mint and strawberries. However, the Registration Standard concluded that additional residue studies were not needed for mint and strawberries, provided that plant metabolism studies show that dicofol, per se, is the residue of concern.

7. Conclusions on residue data for specific crops.

7a. Additional residue data for oranges are needed from FL. Additional residue data on lemons are needed from AZ to support the use of ground applications of the EC formulation on lemons. With the submission of additional residue on oranges from FL and lemons from AZ, and

additional information and clarification of the residue data submitted, we could conclude that up to three ground applications of the EC formulation to citrus at rates up to 6 lb ai/A with a 7 day PHI are supported, and the existing tolerance of 10 ppm will not be exceeded, provided that dicofol, per se, is the sole residue of concern. Other formulations and aerial applications are not supported.

7b. Dicofol residues concentrated in citrus peel and citrus oil. Based on the processing data submitted, food additive tolerances are needed for citrus peel and citrus oil. Tolerances of 100 ppm and 1000 ppm may be appropriate, assuming that the citrus tolerance will remain 10 ppm.

7c. Additional residue data on apples are needed from VA/NC with multiple applications at 3 lb ai/A to support three ground applications of the flowable formulation at 3 lb ai/A (7 day PHI). An increased tolerance is needed as well; 10 ppm may be appropriate, if no other uses (formulations, higher rates, etc.) on apples are to be supported, and if dicofol is determined to be the sole residue of concern.

7d. Dicofol residues are reduced in apple juice, which is a human food, and concentrated in apple pomace, which is an animal feed. A feed additive tolerance will be needed for apple pomace, based on the concentration in dry apple pomace. A tolerance of 250 ppm may be appropriate, based on an estimated need for a 10 ppm tolerance in apples.

7e. We could conclude that three ground applications of dicofol (WP of F only) at 3 lb ai/A with a 7 day PHI is supported in pears, provided the deficiencies listed above in conclusion 6 are resolved. An increased tolerance in pears is needed; 10 ppm may be appropriate, provided that only three ground applications of the flowable formulation at 3 lb ai/A (7 day PHI) is to be supported, and provided that dicofol is determined to be the sole residue of concern.

7f. We could conclude that a rate of 2 applications of dicofol (WP of F only) at 1.2 lb ai/A (14 day PHI) is supported, provided the deficiencies discussed above in conclusion 6 are resolved. The existing tolerance in grapes is exceeded and must be increased, even with only 2 applications at 1.2 ppm. A tolerance of 10 ppm in grapes may be appropriate, provided only two ground applications of the flowable formulation at 1.2 lb ai/A

is to be supported, and provided that dicofol is determined to be the sole residue of concern.

7g. Dicofol residues are reduced in grape juice and wine. Dicofol residues concentrate in raisin waste and grape pomace. Feed additive tolerances will be needed for these grape processed commodities. Feed additive tolerances of 100 ppm for raisin waste and 20 ppm for grape pomace may be appropriate. A food additive tolerance of 100 ppm for raisins may be appropriate, depending on the tolerance established for grapes.

7h. Residue data for dry beans are needed from MI, CA, NE, ND, ID, and CO. Either data on dry bean forage and hay or data on dry bean cannery waste are needed. The principal varieties of dry beans are navy beans (24%), great northern beans (11%), pinto beans (35%), and red kidney beans (7%). These varities should be represented by residue data to maintain registration on dry beans. The dry bean (without pod) should be analyzed. (Succulent beans are analyzed with pods.)

7i. The submitted cottonseed residue data do not support any use of dicofol on cotton since the seeds were delinted before analysis. The rac is undelinted cottonseed. A single field trial with aerial application would not be sufficient to maintain registration of aerial application. Additional cottonseed residue data where the undelinted cottonseed are analyzed, are needed for each type of application (ground, aerial, ULV), and for each type of formulation to be used.

7j. Dicofol residues concentrated in cottonseed oil. A Food additive tolerance will be necessary. An appropriate level cannot be determined since cottonseed samples with non-detectable residues were processed. We can make no conclusions about concentration of dicofol in hulls, meal, and soapstock, since cottonseed samples with non-detectable residues were processed. An additional processing study is needed for cottonseed, where cottonseed with detectable residues is processed.

7k. We could concluded that the use of three applications of dicofol (WP or F only) on squash at 0.6 lb ai/A (PHI 2 days) is supported, provided that the deficiencies discussed above in conclusion 6 are resolved. The existing tolerance of 5 ppm for squash will be adequate, and potentially could be lowered to 2 ppm, providing three ground applications of the flowable formulation at 0.6 lb ai/A with a PHI of 2 days is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

7l. Additional residue data for cucumbers is needed from WI/MI. The existing tolerance of 5 ppm in cucumbers may be adequate and could possibly be lowered to 1 ppm, provided that additional residue data are submitted from WI/MI, that two ground applications of the WP or F at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

7m. We could conclude that the use of two ground applications of dicofol (WP or F only) at 2 lb ai/A (PHI 2 days) is supported, provided the deficiencies discussed above in conclusion 6 are resolved. The existing tolerance of 5 ppm in melons may be adequate and could possibly be lowered to 1 ppm, provided that two ground applications of the WP or F at 2 lb ai/A and a PHI of 2 days is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

7o. We could conclude that the use of two ground applications of dicofol (EC only) to pecans at 2 lb ai/A are supported, provided the deficiencies in the residue data discussed in conclusion 6 are resolved. The existing tolerance of 5 ppm in pecans may be adequate and could possibly be lowered to 0.1 ppm, provided that two ground applications of the EC at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

7p. We could conclude that the use of two ground applications of dicofol (EC only) to walnuts at 2 lb ai/A are supported, provided the deficiencies in the residue data discussed in conclusion 6 are resolved. The existing tolerance of 5 ppm in walnuts may be adequate and could possibly be lowered to 0.1 ppm, provided that two ground applications of the EC at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

8. The cattle and poultry feeding studies submitted are not complete and are not acceptable. The complete study (all three feeding levels) must be submitted, along with storage stability data. Although the feeding studies are not complete, we are able to determine that tolerances for cattle and poultry products will be necessary. However, without the complete study, and complete residue data, we cannot determine appropriate tolerance levels.

The feeding studies cannot be accepted until TOX and EEB concur that the metabolism of dicofol in animals is adequately understood. The registrant should note our calculations of maximum dietary intake for livestock based on current tolerances.

9. Compatibility with CODEX was discussed in the Registration Standard. This issue will be discussed further when the metabolism issues are resolved and the residue data reviewed in full.

RECOMMENDATIONS

We recommend that the registrant be informed of the remaining Registration Standard data gaps and advised to fill the data gaps. We recommend that the PM take appropriate action regarding non-receipt of required data.

We recommend that the PM inform TOX of our note in conclusion 4 regarding the chemical name of one rat metabolite.

Detailed Considerations

TOLERANCES

Registration Standard Conclusions on Tolerances

A tolerance reassessment will be performed when the Registration Standard data deficiencies are resolved. If the requested additional residue data indicate the presence of residues of DDTr in or on raw agricultural commodities resulting from the registered uses of dicofol on these raw agricultural commodities, then residue tolerances for DDTr for these commodities may be required. Two uses, for alfalfa and clover seed crops, are designated as food uses of dicofol and require tolerances under the Federal Food Drug and Cosmetic Act.

Current Conclusions on Tolerance Reassessment

Plant and animal metabolism issues have not been resolved. (See S. Hummel memo of 5/27/87.) Consequently, a tolerance reassessment cannot be completed at this time. Additionally, residue data have not been submitted for all types of formulations on all crops. If dicofol is determined to be the sole residue of concern in plants, the deficiencies in the residue data resolved, and only formulations and types of applications for which data have been submitted are to be supported, then increased tolerances will be needed for apples, pears, and grapes. Tolerances could possibly be lowered for melons, cucumbers, squash, pecans, and walnuts. The tolerance for citrus appears to be adequate. The tolerance for citrus appears to be adequate. No conclusion can be made regarding tolerances in dry beans and cottonseed due to severe deficiencies in the submitted residue data.

Food additive tolerances will be needed for citrus peel (100 ppm) and oil (1000 ppm), raisins (100 ppm), and cottonseed oil (level cannot be determined). Feed additive tolerances will be needed for apple pomace (250 ppm), raisin waste (100 ppm), and grape pomace (20 ppm). No conclusion can be made on the need for food and feed additive tolerances for cottonseed hulls, meal, or soapstock, since cottonseed with non-detectable residues were processed. See Residue data section for further information.

Since no residue data have been submitted for apricots, nectarines, peaches, caneberries, cherries, plum (fresh prunes), tomatoes, peppers, figs, field corn, alfalfa, or clover, tolerances for these crops should be revoked. (Another registrant has submitted protocols for residue field trials on succulent beans and hops.)

Tolerances for meat, milk, poultry and eggs will be needed. However, we cannot determine appropriate tolerance levels until metabolism issues are resolved, and until complete animal feeding studies are submitted.

REGISTERED USES

The registered uses of dicofol products and established tolerances are summarized below in Table 1. Both dilute and concentrate sprays may be used. More detail can be found in the Registration Standard.

TABLE 1

REGISTERED USES

CROP	RATE(lb ai/A)	#APPLICATIONS	PHI (days)	TOLERANCE (ppm)	LIMITATIONS
Hops	0.52-1.5	1 or more	7	30	feeding restriction (Rohm and Haas)- not practical
Mint Hay	0.65-1.2	1	30	25	feeding restriction - hay & spent hay- not practical
Apricots, Nectarines, and Peaches	1.05-3.2	1 or more	14	10	30 day interval (Rohm and Haas)
Grapefruit, Kumquats, lemons, limes, oranges, and tangerines	1.6-8.0	1 or more	7	10	follow directions of State Ag. Experiment Station
apples, crabapples, pears, and quinces	1.0-4.0	1 - 2 (Rohm and Haas-1 or more)	7	5	one label-10-14 day intervals; one label-30 day intervals; Rohm and Haas- 7-10 day interval
blackberries, boysenberries, dewberries, loganberries, and raspberries	0.45-1.2	1 or more	2	5	

CROP	RATE(lb ai/A)	#APPLI-CATIONS	PHI (days)	TOLERANCE (ppm)	LIMITATIONS
cherries	1.2-3.2	1 or more	7	5	30 days between applications
plums (prunes)	1.4-2.0	1 or more	7	5	30 days between applications
beans (dry), snap beans and lima beans (succulent)	0.3-1.5	not listed	7-45	5	feeding restriction
cantaloupes, melons, muskmelons, and watermelons,	0.17-1.5	1 or more	2	5	
pumpkins, winter squash,and summer squash	0.17-1.5	1 or more	2	5	feeding restriction
cucumbers	0.3-1.5 0.3-0.7	1 or more 1 or more	21 2	5	
bushnuts, butternuts, chestnuts, hazelnuts, hickory nuts,pecans, walnuts, and filberts	1.6-4.0	1 or more	14	5	feeding restriction-husks
tomatoes	0.3-1.5 0.3-0.7	1 or more 1 or more	21 2	5	feeding restriction - (not practical)
eggplants, peppers, and pimentos	0.6-1.5	1 or more	2	5	
grapes	0.45-1.5	1 or more	7	5	
figs	1.4-2.0	1 or more	7	5	feeding restriction-husks (not needed)
strawberries	0.4-2.4	2-3	2	5	10-20 days between applications
cottonseed	0.8-1.6	1 or more	14	0.1	feeding restriction - gin trash (not practical) some labels-feeding restriction-forage

CROP	RATE(lb ai/A)	#APPLI-CATIONS	PHI (days)	TOLERANCE (ppm)	LIMITATIONS
alfalfa, clover	1.0-1.5	1 or more	-	none	grown for seed feeding restriction (not practical)
field corn*	0.74-1.5	1 or more	45	none	Do not apply after ears begin to form; feeding restriction (not practical)

*No tolerance has been issued for dicofol residues on field corn. However, uses for dicofol on field corn appear on registered State labels, and 3.2% of the total pounds of dicofol is used on field corn (memo from Bruce A. Kapner, 1/27/84).

Note: The current label feeding restrictions for hops, mint hay, tomatoes, cottonseed, alfalfa and clover grown for seed, and field corn, are not practical. Thus label changes and residue and processing data are needed on these commodities, which are animal feed items, since there is a possibility of transfer of residues to meat, milk, poultry, and eggs.

LABELING

Registration Standard Labeling Requirements

The Registration Standard stated that label restrictions will depend on data yet to be submitted and that the Agency may, after review of data to be submitted in response to the Standard, require additional revision to current labels and may impose additional label requirements.

Rohm and Haas Response

No revised labeling was included in this submission.

RCB Comments/Conclusions on Registered and Proposed Uses

All labeled uses must be supported by residue data or the labels amended to reflect the maximum use supported by residue data. Residue data must be submitted to support the maximum number of applications allowed on the label, or the maximum number of applications allowed on the label must be changed to reflect the use supported by the submitted residue data. A maximum number of applications per season or a maximum quantity of pesticide to be applied per season must be specified on the label. A minimum interval between

applications must be added to the labels. Product labels must be changed to allow dilute sprays and ground application only or residue data must be submitted for both dilute and concentrate sprays, ground and aerial application, as allowed on product labels. Use directions for orchards must be changed to account for the variability in tree sizes. (Several options for orchard labeling are shown in Attachment 1 of our previous review (S. Hummel, 5/27/87)). Grazing restrictions must also be added for orchards. Suggested language is "Do not allow livestock to graze in treated areas or feed on orchard cover crops." Alternatively, tolerances may be proposed for orchard cover crops and grazing allowed only on orchard cover crops for which tolerances have been established.

Product labels must be changed to allow use only of formulations for which residue data have been received or residue data must be supplied for each type of formulation to be used, i.e., Emulsifiable Concentrate (EC), Wettable Powder (WP) or Flowable Concentrate (F), Granular (G), and Dust (D).

No residue data on an emulsifiable concentrate formulation were submitted for apples, pears, grapes, melons, cucumbers, or squash. Data are required for the emulsifiable concentrate formulation on these crops. Alternatively, these crops may be removed from labels for EC products.

No residue data from the use of a wettable powder or flowable formulation were submitted for citrus, dry beans, pecans, walnuts, and cottonseed. Residue data are required for the wettable powder or flowable formulation on these crops. Alternatively, these crops may be removed from proposed wettable powder and flowable formulation labels.

Residue studies were not submitted for mint and strawberries. However, the Registration Standard concluded that additional residue studies were not needed for mint and strawberries, provided that plant metabolism studies show that dicofol, per se, is the residue of concern.

The volume of spray used in the residue field trials was not reported in the residue field trial reports. The volume of spray must be reported for each residue field trial. Residue data will support only the volume (dilute, concentrate, ULV) and type of application for which data were supplied. (Data on dilute applications support dilute applications on the label; data on concentrate applications support concentrate applications on the label; etc.)

Revised labels were not included in this submission. Consequently, this deficiency remains outstanding.

Comments on Specific Crops

These comments on specific crops are related to residue data included in this submission. The submitted residue data appear to support these uses, pending resolution of the deficiencies in the residue data, as discussed in the residue data section of this review.

We have tabulated the rates and formulations used for developing residue data for this submission. The rates, PHI, and type of formulation used in these residue studies are tabulated below.

Table 2

Ground Application Uses Supported by Residue Data [1/]

Crop	Formulation	Maximum Rate	PHI (days)
Citrus[2/]	EC	3 x 6 lb ai/A	7
Apples[2/]	F	3 x 2.25 lb ai/A	7
Pears[2/]	F	3 x 3 lb ai/A	7
Grapes[2/]	F	2 x 1.2 lb ai/A	7
Dry Beans[3/]	F	2 x 1.5 lb ai/A	20
Melons	F	3 x 0.6 lb ai/A	2
Cucumbers	F	3 x 0.6 lb ai/A	2
Squash	F	4 x 0.6 lb ai/A	2
Pecans	EC	2 x 2 lb ai/A	7
Walnuts	EC	2 x 2 lb ai/A	7
Cottonseed [4/]	EC	none supported	

[1/] Pending resolution of residue data deficiencies and establishment of higher tolerances. The volume of spray (dilute, concentrate, ULV) supported by the residue data is not known.

[2/] Higher tolerance needed.

[3/] Tolerance for bean forage and hay needed (or tolerance for bean cannery waste). Additional data needed for dry beans (without pods).

[4/] Delinted cottonseeds were analyzed. No data were submitted for undelinted cottonseed.

The maximum rates tabulated above are the maximum rates that appear to be supported, pending resolution of the deficiencies in the submitted residue data. (See discussion starting on p. 20 of this review. These data cannot support formulations of a different type than was used in the residue

field trials. Only ground or hand applications were used in generating these data (except for one field trial for cottonseed and several field trials for dry beans with aerial application). Thus, data must be submitted for aerial applications or aerial applications should be prohibited on the labels.

FORMULATION

There are currently three registered end use products containing dicofol, all 4 lb/gal EC, including Kelthane MF. Additionally, Rohm and Haas has several pending applications for new registration of end use products: Kelthane EC (4 lb/gal), Kelthane 4F (4 lb/gal flowable), and Kelthane 35 (35% WP). (D. Edwards, PM#12, personal communication). The active ingredient in Kelthane is the o,p'- and p,p'- isomers of dicofol.

MANUFACTURING PROCESS AND IMPURITIES

Rohm and Haas and Makhteshim Agan have submitted manufacturing processes for their dicofol technicals. These manufacturing processes have been discussed in previous Product Chemistry Reviews. The manufacturing process was discussed in general terms in the Registration Standard (12/30/83) and in Sittig. Briefly, DDT is chlorinated, producing Cl-DDT. The Cl-DDT is hydrolyzed in acidic solution, producing dicofol. Dicofol contains a mixture of isomers, approximately 1:4 o,p' to p,p', approximately the same ratio as the ratio of isomers in the DDT starting material.

Dicofol products are known to contain DDT and related impurities, including DDE, DDD, and Cl-DDT. DDT related compounds are referred to as DDTr. The Dicofol Special Review was concluded with a cancellation notice (51 FR 19508, May 29, 1986), cancelling all dicofol products unless their registrations were amended to include an upper certified limit of no more than 2.5% DDTr in the technical product. By January 1, 1989, dicofol products may contain no more than 0.1 % DDTr in the technical.

OCTANOL-WATER PARTITION COEFFICIENT

Rohm and Haas submitted data on the ontanol water partition coefficients of p,p' and o,p'-dicofol. (MRID Nos. 400420-01 and 400420-02) The dpm (proportional to concentration) of ^{14}C dicofol was measured with a Beckman Model 3801 Liqud Scintillation Counter. Three concentration

levels were tested. Raw data were included in the report. The following was reported for the octanol-water partition coefficient.

Compound	$K_{o/w}$	Log P
p,p'-dicofol	1.9×10^4	4.28
o,p'-dicofol	3.0×10^4	4.48

PLANT AND ANIMAL METABOLISM

No adequate plant metabolism studies for dicofol had been submitted prior to the publication of the Registration Standard. Two metabolites had previously been reported in mint oil (see PP#6F0472), 4,4'-dichlorobenzophenone, and 4,4'-dichlorobenzhydrol (MRID No. 00004321). There had been some evidence for conversion of dicofol to polar metabolites (MRID No. 05006528). These polar metabolites were not identified.

Plant and animal metabolism were discussed in our previous review (S. Hummel, 5/27/87, RCB No. 1869). Rat metabolism was reviewed by TOX (W. Phang, memo of 5/27/87).

Dicofol was reported as the major residue in crops. However, dicofol was not unequivocally identified in any plant metabolism study. In grapefruit peel, 40% of the residue remains unidentified (10% or more polar compounds, 30% not extracted). Enzyme hydrolysis, which may have released additional residues, was not attempted in grapefruit peel. None of the radioactivity in cottonseed was unequivocally identified. Up to 50% of the radioactivity remained unextracted. Enzyme hydrolysis, which may have released this activity, was not attempted.

The major dicofol metabolite in lactating goats and laying hens is 1,1-bis-(chlorophenyl)-2,2-dichloroethanol (FW-152). Minor metabolites are dichlorobenzophenone and dichlorobenzhydrol. Unmetabolized dicofol is also present. Little if any dicofol is metabolized to DDE in lactating goats. However, only 50% of the total radioactive residue (TRR) was extracted from goat and hen liver. Enzyme hydrolysis of goat liver was not attempted. Neither base nor enzyme hydrolysis of laying hen liver was attempted. Egg yolks were 70% extracted. Other tissues were 80 to 100% extracted. We are awaiting the results of our deferral to TOX and EEB on the need for additional analysis of goat and hen liver and egg yolk. Base and enzyme hydrolysis may have released additional activity.

In rats, the major metabolite was FW-152. FW-152 was further metabolized to dichlorobenzophenone (DCBP) and dichlorobenzoic acid (DCBA). Very little DDE was found (W. Phang, TOX, memo of 5/27/87).

Note to TOX: The rat metabolite identified as dichlorobenzoic acid (DCBA) is incorrectly named. The structure given corresponds to 2,2-bis(chlorophenyl)-2-hydroxyacetic acid.

ANALYTICAL METHODOLOGY

Plant Methodology

The analytical method included in this submission was titled, "A Residue Analytical Method for p,p'-Dicofol and o,p'-Dicofol," Rohm and Haas Technical Report No. 310-86-74, C. K. Hofmann, November, 1986. (MRID No. 400420-08). The copy of this method is stamped, "Property of Rohm and Haas Company, Philadelphia" on every page. For that reason, the method cannot be accepted. A "clean" copy of the analytical method is required, i.e., a copy with no no claim of confidentiality. The method is similar to the PAM I method for chlorinated hydrocarbons.

The method involves several different sample workups, depending on the crop to be analyzed, followed by GC analysis for p,p'- and o,p'- dicofol. No metabolites or degradates are analyzed.

The sample workup involves extraction with isooctane (orange peel, cottonseed), acetonitrile (orange pulp, whole orange), or acetone and isooctane (corn husks and cobs, corn kernels, and beans). For other crops, the sample workup from Rohm and Haas TR # 36-81-05 is to be followed. A copy of this report was not included, and is needed for review.

An aliquot of the initial extract is evaporated to an oily residue by rotary evaporation at 60C, and reconstituted with petroleum ether. The sample is partitioned into acetonitrile, and partitioned back into petroleum ether with the additional of NaCl. The pet ether extract is washed with NaCl solution, and dried with Na_2SO_4. The pet ether extract is evaporated to an oily residue and reconstituted with a known volume of pet ether. The extract is cleaned up on a florisil column. The column is eluted with 6% ethyl ether/petroleum ether. The first eluate is discarded. The column is then eluted with 15% ethyl ether/petroleum ether. This eluate is collected, evaporated to dryness by rotary evaporation at 60C, and reconstituted with isooctane for GC analysis.

A 6' x 2 mm i.d. column, packed with 5% OV-17 on 80/100 mesh Gas Chrom Q (Supelco), is used, along with an Electron Capture Detector (ECD). The column is maintained at 200C for the analysis and then ramped to 250C, apparently to clean out the column between analyses. The injector was maintained at 240C, and the detector at 350C. The retention time of p,p'-dicofol by this method was 7.3 minutes, and the retention time of o,p'-dicofol, approximately 6.3 minutes. Peak heights were used for calculation. Recoveries were calculated by adjusting for control values. Residues were corrected for recovery. The sensitivity of the method was reported to be 0.01 ppm for both dicofol isomers.

Recoveries were reported for a number of commodities and listed in our previous review. Average recoveries ranged from 63 to 130%, and typically were about 90%. Low recoveries were reported for crude cottonseed oil, corn hulls, and grape waste.

Conclusions and comments on the plant methodology was included in our previous review (S. Hummel, 5/27/87).

Animal Product Methodology

Separate methodology was submitted for the animal feeding studies. These methods are found in MRID Nos. 400420-30 and -31.

Tissues are extracted three times with acidified methylene chloride. The solvent is removed by rotary evaporation. The samples are reconstiduted with 50:50 methylene chloride:cyclohexane. The samples were then cleaned up on a florisil column. The column was eluted with 6% acidified ethyl ether in petroleum ether, and the first eluate discarded. The column was then eluted with 15% acidified ethyl ether in petroleum ether. The volume of the eluate was reduced by rotary evaporation, and reconstituted with acidic methanol (0.05% acetic acid) for HPLC analysis. Two 10u 25 cm C_{18} columns connected in series and UV detection at 230 nm were used for the analysis of dicofol and DCBP. The mobile phase was 85:15 acetonitrile:water. GC/ECD, using a 30m DB-5 column, H_2 carrier gas and temperature programming were used for the analysis of FW-152. For GC analysis, the methanol solvent was removed with a stream of nitrogen. The sample was reconstituted in isooctane.

Recoveries were reported as presented in Table 3. The range over which recoveries were reported is given as ppm range. The average recovery is presented as $\bar{x} \pm$ S.D., where S.D. is the standard deviation of the recoveries. The number of analyses, N, is given as a footnote to the table.

TABLE 3

Method Validation Recoveries.

Dicofol

Sample Type	o,p' ppm Range	o,p' $\bar{x}$ ±S.D.	p,p' ppm Range	p,p' $\bar{x}$ ±S.D.
Milk	0.01-6.0	106 ±19.2[a]	0.01-6.0	109 ±15.0[a]
Muscle	0.05-1.0	83.7 ±4.08[d]	0.05-1.0	81.2 ±4.73[d]
Kidney	0.05-1.0	91.4 ±18.4[e]	0.05-1.0	90.6 ±13.2[e]
Liver	0.05-5.0	103 ±6.25[e]	0.05-5.0	87.5 ±12.1[e]
Fat	0.15-16	97.6 ±4.24[e]	0.15-16	96.8 ±4.63[e]

DCBP

Sample Type	o,p' ppm Range	o,p' $\bar{x}$ ±S.D.	p,p' ppm Range	p,p' $\bar{x}$ ±S.D.
Milk	0.01-6.0	98.6 ±16.3[a]	0.01-6.0	94.6 ±19.0[a]
Muscle	0.05-1.0	82.6 ±4.53[d]	0.05-1.0	79.9 ±3.50[d]
Kidney	0.05-1.0	80.0 ±7.66[e]	0.05-1.0	74.6 ±10.6[e]
Liver	0.05-5.0	89.7 ±5.12[e]	0.05-5.0	92.1 ±4.45[e]
Fat	0.15-16	84.2 ±5.94[e]	0.15-16	82.1 ±8.16[e]

FW-152

Sample Type	o,p' ppm Range	o,p' $\bar{x}$ ±S.D.	p,p' ppm Range	p,p' $\bar{x}$ ±S.D.
Milk	0.01-6.0	96.8 ±15.6[b]	0.01-6.0	96.9 ±14.7[c]
Muscle	0.05-1.0	75.0 ±6.07[d]	0.05-1.0	75.8 ±9.86[d]
Kidney	0.05-1.0	97.6 ±9.39[e]	0.05-1.0	106 ±15.1[e]
Liver	0.05-5.0	86.0 ±10.3[e]	0.05-5.0	88.3 ±10.7[e]
Fat	0.15-100	90.2 ±14.3[e]	0.15-100	93.2 ±16.9[e]

[a] N = 12; [b] N = 10; [c] N = 9; [d] N = 6; [e] N = 8

TABLE 3 continued

Method Validation Recoveries.

Dicofol

Sample Type	o,p' ppm Range	o,p' $\bar{x}$ ±S.D.	p,p' ppm Range	p,p' $\bar{x}$ ±S.D.
Whole Egg	0.03-1.0	89.5 ±14.9[a]	0.03-1.0	97.3 ±27.3[a]
Muscle	0.05-1.0	103 ±5.20[c]	0.05-1.0	87.4 ±3.41[c]
Kidney	0.05-3.0	116 ±25.7[c]	0.05-3.0	105 ±21.2[d]
Fat	0.15-5.0	96.1 ±17.8[c]	0.15-5.0	97.8 ±8.77[c]

DCBP

Sample Type	o,p' ppm Range	o,p' $\bar{x}$ ±S.D.	p,p' ppm Range	p,p' $\bar{x}$ ±S.D.
Whole Egg	0.03-1.0	107 ±26.4[a]	0.05-1.0	91.3 ±17.0[b]
Muscle	0.05-1.0	87.2 ±2.25[c]	0.05-1.0	92.6 ±7.44[c]
Kidney	0.05-3.0	89.6 ±18.0[d]	0.05-3.0	92.8 ±16.8[d]
Fat	0.15-5.0	87.9 ±14.2[c]	0.50-5.0	90.2 ±6.00[f]

FW-152

Sample Type	o,p' ppm Range	o,p' $\bar{x}$ ±S.D.	p,p' ppm Range	p,p' $\bar{x}$ ±S.D.
Whole Egg	0.03-1.0	88.0 ±23.9[a]	0.03-1.0	93.0 ±24.5[a]
Muscle	0.05-1.0	92.3 ±11.8[c]	0.05-1.0	94.9 ±13.5[c]
Kidney	0.05-3.0	105 ±5.80[c]	0.05-3.0	102 ±11.0[e]
Fat	0.15-5.0	85.6 ±12.5[e]	0.15-5.0	83.0 ±17.5[e]

[a] N = 10
[b] N = 8
[c] N = 6
[d] N = 7
[e] N = 5
[f] N = 4

UNOFFICIAL

RCB Comments/Conclusions on Animal Products Methodology

The sample workup is similar to the PAM I method for chlorinated hydrocarbons. The submitted methodology can only be adequate if the residue of concern in meat, milk, poultry, and eggs is determined to consist of dicofol, dichlorobenzophenone, and FW-152 (1,1-bis-(chlorophenyl)-2,2-dichloroethanol). Our final conclusion on the animal product methodology is reserved until the animal metabolism is considered adequately understood.

Methodology Used for Residue Data

The residue analyses were reportedly done by a method based on Rohm and Haas TR-36-81-05. Neither the method used, nor Rohm and Haas TR-36-81-05 were included in this submission.

We would assume that the analytical method used was TR-310-86-74, which was included in this submission. However, the analytical method used needs to be clarified. A copy of TR-36-81-05 will still be needed, since sample preparations from this method are used for Method # TR-310-86-74.

RCB Comments/Conclusions on Methodology Used for Residue Data

The analytical methodology used for the development of residue data must be submitted or a reference to the method used (for methods already in our files) must be submitted. This is a deficiency.

STORAGE STABILITY DATA

Registration Standard Data Gap

> All residue studies should be supported by storage stability studies of samples held in storage before analysis. Handling history of the samples should accompany all of the residue studies.

Current Submission

Storage Stability data were submitted for citrus (MRID No. 400420-11) and cottonseed products (MRID No. 400420-09 and -10). The need for storage stability data in meat, milk, poultry and eggs was mentioned in the animal feeding study reports (MRID Nos. 400420-30 and 400420-31). However, storage stability data for animal products were not included in this submission.

Citrus samples were fortified at 1 ppm, and stored one year at -15C in a freezer used for the storage of field trial samples (MRID No. 400420-11). Analytical method No. 31L-83-10 was reportedly used for the analyses. The method was reportedly included in this submission. However, only a very brief summary of the method was included. A copy of the analytical method used must be submitted.

Samples were extracted with acetonitrile, back extracted with petroleum ether. The organic layer was dried with sodium sulfate, and the solvent removed by rotary evaporation. The samples were analyzed by HPLC. Additional details of the method were not included. Sample calculations were reportedly done in accordance with Method TR 31L-83-10, and were not included. HPLC chromatograms were included. Results for both o,p' and p,p'- dicofol varied $\pm$10% over the one year period of storage. No pattern of degradation was seen.

Cottonseed products (whole seeds, hulls, meal, refined oil) were fortified at 1 ppm and cottonseed soapstock was fortified at 2 ppm. Samples analyzed for p,p'-dicofol were stored for 18 months (MRID No. 400420-09). Samples analyzed for o,p'-dicofol were stored for 10 months (MRID No. 400420-10). One additional data point is planned for 2 years storage. Samples were reportedly analyzed by Method TR 35F-81-05 (the same method as was used for the residue data). The method was alternatively described as GC and as HPLC. GC data (chromatograms) were included. We note that the GC chromatograms were labeled as HPLC chromatograms. The GC conditions written on the chromatograms indicated that the analysis used a 6' OV-17 column (on Gas Chrom Q 80/100 mesh) at 195C, and a ^{63}Ni EC detector. And the chromatograms were on strip chart recorder paper, typically used for GC analyses. Consequently, the analytical method used for this report needs to be clarified.

Analytical results in cottonseed products were quite variable. Results varied up to $\pm$ 20% from the spiked amount from one analysis to the next. Residues in refined oil showed a steady decline in residues. Residues of p,p'-dicofol decreased 16% in 26 weeks and 24% in 78 weeks. Residues of o,p'-dicofol decreased 32% in 26 weeks and 22% in 42 weeks. Residues in cottonseed hulls declined sharply in week 78 of the study for p,p'-dicofol (-17%) and week 42 of the study for o,p'-dicofol (-33%).

RCB Comments/Conclusions on Storage Stability Data

No degradation was noted in citrus. The analytical method used is needed before we can conclude that the citrus storage stability study is acceptable.

Significant degradation was found in cottonseed hulls and oil. We cannot concur with the registrant that these results are anomalous. Results were variable for other cottonseed fractions.

FIELD RESIDUE DATA

Registration Standard Data Gaps

Although for most crops, residue data were submitted; due to the changes of application rates and pre-harvest intervals, these outdated data are not adequate to support the registered uses under present day standards, and the established tolerances are not supported.

Residue data will be required for all crops (except mint and strawberries) reflecting the maximum registered application rate, in samples taken at intervals after the application in order to establish a time lapse degradation pattern (decline curve) for the residues. The residue studies should include multiple applications, ground and aircraft application equipment, representation of formulations used, geographical representation, as well as effects of climatological conditions (rain, wind, sun, etc.).

Processing studies will be required showing the amount of residue in the processed commodities apple pomace, tomato pomace, tomato waste, citrus pulp, citrus oil, grape pomace, raisin waste, cottonseed byproducts, i.e., cottonseed meal and hulls, cottonseed oil, etc.

If a concentration of residues is indicated to the extent that the residue level exceeds that of the tolerance level established for the r.a.c., a food additive tolerance for the byproduct will be required.

Residue studies are required reflecting the registered application rate on crop feed items, i.e., forage, hays, stalks, stover, vines, cottonseed linters etc., and if residues are present, adequate tolerances should be established.

Current Submission

Residue data were submitted for citrus, apples, pears, grapes, dry beans, melons, cucumbers, summer squash, pecans, walnuts, and cotton. Field trial reports consisted of a very brief summary and some raw data, including chromatograms and an incomplete summary sheet for each field trial. Residue

an incomplete summary sheet for each field trial. Residue data on the individual crops are discussed below. A number of deficiencies were found to be common to all studies. The common deficiencies are discussed here rather than for each individual study.

The composition of the pesticide formulation used in the field trials was not included in the submission. This analysis is required for each field trial to determine if any impurities may present a residue problem to determine if any impurities may present a residue problem.

Field trial plots were treated two to three times, but only one treatment date was given in the raw data or in the report, apparently the last treatment, since the difference between this date and the harvest date corresponds to the PHI. The interval between treatments was not given either. This information is required. Planting dates, one treatment date, and the harvest date were given. The interval between treatments is needed for each field trial.

The data do not indicate whether dilute or concentrate sprays were used. This information (and volume of spray solution used per acre) is needed for each field trial.

It is unclear if several (2-4) samples from each location were taken, and the results averaged, or if one sample was taken from each location, with several analyses of that sample and the results averaged. Results from separate samples should not be averaged.

Samples were shipped in dry ice from Rohm and Haas to Analytical Development Corporation, where the analyses were done. However, no information on the storage of samples from harvest until receipt by Rohm and Haas was included in any of the residue reports. Processed products were reportedly frozen from the time of processing until shipment to Rohm and Haas by air freight. No information was included on the temperature of the samples when shipped, or on the dates of shipping, processing, storage, and analysis. Complete sample storage information is needed for each sample from the time of harvest until analysis.

The analyses were reportedly done be a method based on Rohm and Haas TR 36-81-05. Neither the method used, nor Rohm and Haas TR 36-81-05 were included. Residue data cannot be accepted until the analytical method used is submitted and reviewed.

Sample calculations were reportedly included in Rohm and Haas TR # 36-81-05, which was not included in this submission. According to the standard curves included with the residue

data, calculations were done by origin constrained regression analysis.

Although recoveries were determined at the time the samples were analyzed, an average recovery of unknown origin, but presumably from the method validation, was used to correct the results for recovery. The same average recovery was also used to correct samples of different processed commodities. Recoveries should be determined each time samples are analyzed and at levels similar to those expected in the crop samples. These recoveries should be used to correct the analytical results for recovery.

We note a lot of duplication of data. Identical standard curves and chromatograms of standards used in the generation of the standard curve are repeated for each sample analyzed in each report. Except for chromatograms of standards, the chromatograms were not dated.

Residues on many crops increased with increasing PHI. For this reason, we question the sampling techniques used. The registrant should completely describe all sampling and subsampling techniques used.

No data on an emulsifiable concentrate formulation were submitted for apples, pears, grapes, melons, cucumbers, or squash. Data are required for the emulsifiable formulation on these crops. Alternatively, these crops may be removed from labels for EC products.

No data from the use of a wettable powder or flowable formulation were submitted for citrus, dry beans, pecans, walnuts, and cottonseed. Data are required for the wettable powder or flowable formulation on these crops. Alternatively, these crops may be removed from proposed labels for wettable powder or flowable formulations.

Residue studies were not submitted for hops, spent hops, apricots, nectarines, peaches, caneberries, cherries, plum (fresh prunes), snap beans, lima beans (succulent), tomatoes, peppers, figs, field corn, alfalfa, clover, processed tomatoes, bean forage and hay or bean cannery waste, corn forage and fodder, and cottonseed forage. These studies were required by the Registration Standard, and are still needed. The PM should take appropriate action regarding the non-submission of these data.

Residue studies were not submitted for mint and strawberries. However, the Registration Standard concluded that additional residue studies were not needed for mint and strawberries, provided that plant metabolism studies

show that dicofol, per se, is the residue of concern.

Information on Specific Crops

Citrus-Grapefruit.. (MRID NO. 400420-12 and -13). Three ground applications of Kelthane MF were made at 6 lb ai/A in six locations in FL (2), CA (2), and TX (2). Grapefruit are grown in AZ (4%), CA (14%), FL (76%), and TX (6%). PHI's ranged from 7 to 21 days. The maximum labeled rate is unlimited applications of 8 lb ai/A. Both ground and aerial applications are permitted on the labels. No information was given regarding the volume of spray used per acre. Samples were stored up to a year before analysis. The residues reported ranged from 0.84 ppm to 5.17 ppm (7 day PHI). Residues found in control samples ranged from non-detectable to 0.03 ppm. Geographical representation for ground application of the EC formulation to grapefruit is adequate.

Citrus-Oranges. Three ground applications of Kelthane MF were made at 6 lb ai/A in five locations in CA (4), and TX. Oranges are grown in AZ (1%), CA (29%), FL (69%), and TX (2%). PHI's ranged from 7 to 21 days. The maximum labeled rate is unlimited applications of 8 lb ai/A with a 7 day PHI. Both ground and aerial applications are permitted on the labels. No information was given regarding the volume of spray used per acre. Samples were stored up to a year before analysis. Residues reported ranged from 0.36 ppm to 3.16 ppm (7 day PHI). Residues in control samples were non-detectable. We note that the table for residues in orange samples is labeled as residues in lemons. The varieties, however, correspond to varieties of oranges, not lemons. Geographical representation for ground application of the EC formulation to oranges is not adequate. Additional residue data for oranges are needed from FL.

Citrus-Lemons. Three ground applications of Kelthane MF were made at 6 lb ai/A in three locations in FL (2) and CA. Two ground applications of Kelthane MF were made at 6 lb ai/A in two additional locations in FL. Lemons are grown in AZ (19%) and CA (29%). PHI's ranged from 0 to 21 days. The maximum labeled rate is unlimited applications of 8 lb ai/A with a 7 day PHI. Both ground and aerial applications are permitted on the labels. No information was given regarding the volume of spray used per acre. Samples were stored up to a year before analysis. Residues reported ranged from 0.70 ppm to 1.25 ppm. Residues in control samples were non-detectable. The maximum residue was found in a sample from FL with a 21 day PHI. Residues did not necessarily decrease with increasing PHI. Additional residue data on lemons are needed from AZ to support the use of ground applications of the EC formulation on lemons.

Citrus-Tangelo. Three ground applications of Kelthane MF were made at 6 lb ai/A in one locations in FL. PHI's ranged from 0 to 21 days. The maximum labeled rate is unlimited applications of 8 lb ai/A. Both ground and aerial applications are permitted on the labels. No information was given regarding the volume of spray used per acre. Samples were stored up to a year before analysis. Residues reported ranged from 0.30 ppm to 1.48 ppm (7 day PHI). Residues were non-detectable in control samples.

The maximum residue reported in citrus from ground application of the EC formulation was 5.17 ppm. With the submission of additional residue on oranges from FL and lemons from AZ, and additional information and clarification of the residue data submitted, we could conclude that up to three ground applications of the EC formulation to citrus at rates up to 6 lb ai/A with a 7 day PHI are supported, and the existing tolerance of 10 ppm will not be exceeded, provided that dicofol, per se, is the sole residue of concern. Other formulations and aerial applications are not supported.

Citrus Processing Data. (MRID NO. 400420-29). Two trials in one location in CA were used for a processing study. Valencia oranges were treated with three ground applications of Kelthane MF at 6 or 12 lb ai/A. Samples were harvested with a 7 day PHI. Oranges were processed into concentrate, dried fines, dried peel, fines, juice, molasses, oil, and wet peel in a citrus product fractionation pilot plant at California State Polytechnic University. A brief description of the fractionation process was submitted. Samples were shipped in dry ice to Rohm and Haas and subsequently to Analytical Development Corporation for analysis.

The fruit was washed, and juice extracted. An emulsion of oil, water, peel and frit is filtered, heated to 120 F, and enzymes added to separate the oil from the emulsion. The oil is separated using a separatory funnel. The peel, rag, frit, and seeds are deposited into a peel hopper. The peel is shredded. As the peel leaves the shredder, a slurry of lime is added. The limed peel is pressed to produce molasses. Pulp is dried to approximately 8% moisture. Dry peel is passed through a cyclone separator, cooled, and collected. Dry fines are collected from the bottom of the cyclone separator. No mention was made of any refining of the citrus oil.

The following results were reported for the citrus processing study. Control samples of orange processed commodities had low or non-detectable residues, except for oil (1.03 ppm).

Table 4

Dicofol Concentration/Reduction Factors in Oranges

Commodity	Residue (ppm)		Concentration/reduction factor	
unwashed fruit	3.79	4.46	1.00	1.00
washed fruit	0.44	2.55	0.12	0.57
wet peel	3.32	8.35	0.88	1.87
dried peel	7.27	24.05	1.92	5.39
dried fines	7.84	29.50	2.07	6.61
fines	7.75	10.93	2.04	2.45
concentrate	0.05	0.05	0.01	0.01
juice	<0.01	<0.01	<0.01	<0.01
molasses	<0.01	<0.01	<0.01	<0.01
oil	141.50	393.00	37.34	88.12

Regulated fractions of citrus are dried pulp, peel, oil, molasses, and juice. Pulp, and molasses are animal feeds. Peel, oil, and juice are human foods. Based on the processing data submitted, food additive tolerances are needed for citrus peel and citrus oil. Food and feed additive tolerance levels are determined by multiplying the rac tolerance by the maximum concentration factor. Tolerances of 100 ppm (10 ppm x 6.6x = 66 ppm) and 1000 ppm (10 ppm x 88x = 880 ppm) may be appropriate, for citrus peel and oil, respectively, assuming that the citrus tolerance will remain 10 ppm.

Apples. (MRID No. 400420-14). Three ground or hand applications of Kelthane 4F were made at the rate of 3.00, 2.25, or 1.88 lb ai/A, in an unspecified volume of spray solution per acre. The lower rates were used in VA (1.88 lb ai/A) and NY (2.25 lb ai/A). The higher rate (3 lb ai/A) was used in WA, NJ, MI, OR, PA, and CA. Apples are grown in WA (35%), NY (12%), MI (9%), PA (7%), CA (6%), VA (6%), NC (4%). PHI's ranged from 7 to 21 days. Residues did not necessarily decline with increasing PHI. Residues reported ranged from 0.71 ppm to 5.54 ppm (7 day PHI). Residues in control samples ranged from non-detectable to 0.04 ppm. Additional residue data on apples are needed from VA/NC with multiple applications at 3 lb ai/A to support three ground applications of the flowable formulation at 3 lb ai/A. An increased tolerance is needed as well; 10 ppm may be appropriate, if no other uses (formulations, higher rates, etc.) on apples are to be supported, and if dicofol is determined to be the sole residue of concern.

Apple processing study. (MRID No. 400420-26). Four samples of apples from the field trials discussed above were processed into juice, wet pomace and dry pomace. The apples

were ground into mush, and the mush pressed to yield juice. The juice was then held as fresh juice, frozen juice, or pasteurized juice. Pasteurization was done in a heat exchanger at 190F. The pasteurized juice was then canned. Wet pomace was also obtained from the press, and was reground. The ground pomace was dried to a 73% weight reduction, cooled 10 minutes and held in a container for 1-2 days to equilibrate, then packed and stored at -10F. The processed samples were packed n dry ice and shipped to Rohm and Haas and then to Analytical Development Corporation for analysis.

The following results were reported for processed fractions of apples. (See Table 5.) Control samples had low or non-detectable residues.

Table 5

Dicofol Concentration/Reduction Factors in Apples

Commodity	Residue (ppm)				
fruit	1.33	1.49	1.63	4.32	3.46
wet pomace	8.35	7.50	14.00	37.20	--
dry pomace	22.70	26.55	41.15	60.73	30.57
juice	<0.01	0.01	<0.01	0.02	0.06
Commodity	**Concentration/reduction factor**				
fruit	1.00	1.00	1.00	1.00	1.00
wet pomace	6.28	5.03	8.59	8.61	--
dry pomace	17.07	17.82	25.25	14.06	7.08
juice	<0.01	0.01	<0.01	<0.01	0.02

Dicofol residues are reduced in apple juice, which is a human food, and concentrated in apple pomace, which is an animal feed. A feed additive tolerance will be needed for apple pomace, based on the concentration in dry apple pomace. A tolerance of 250 ppm may be appropriate, based on an estimated need for a 10 ppm tolerance in apples. (10 ppm x 25x = 250 ppm).

Pears. (MRID No. 400420-15 and -16). Three ground applications of Kelthane 4F were made at the rate of 3 lb ai/A in three locations in CA, MI, and OR. Three ground applications of Kelthane 35W were made at the rate of 3 lb ai/A in one location in WA. Pears are grown in CA (44%), WA (29%), OR (21%), NY (3%), and MI (2%). The geographical representation of these data is adequate.

The volume of spray solution used per acre was not given. The dates of application were not given. We cannot determine

the interval between applications. PHI's ranged from 6 to 21 days. The maximum label rate is multiple applications of 4 lb ai/A. The label PHI is 7 days. Residues reported ranged from 0.27 ppm to 8.81 ppm. Residues in control samples ranged from non-detectable to 0.27 ppm. The maximum residue was found in a D'Anjou pear in OR (6 day PHI). Residues did not necessarily decrease with increasing PHI. An increased tolerance is needed; 10 ppm may be appropriate, provided that only three ground applications of the flowable or wettable powder formulations at 3 lb ai/A are to be supported, and provided that dicofol is determined to be the sole residue of concern.

Grapes. (MRID No. 400420-24). Residue data were submitted from CA (4 trials), OR, MI, PA, and NC (1 trial each). Grapes are grown in CA (90%), NY (4%), WA (3%), PA (1%), and MI (1%). The geographical representation of the submitted residue data is adequate. Ground applications of Kelthane 4F were made. Several application rates and numbers of applications were used. Some field trials used ground two applications at 1.2 lb ai/A (4 trials in CA, 1 trial in OR and NC). One used two ground applications at 2.0 lb ai/A (MI). One used 5 ground applications at 1.2 lb ai/A (PA). The label rate is multiple applications of 1.5 lb ai/A with a 7 day PHI. Residues often increased with increasing PHI.

Residues reported in grapes treated with 2 ground applications at 1.2 lb ai/A ranged from 0.27 ppm to 5.19 ppm (data from Pacific Northwest and NC only) The maximum residue was found in a sample with a PHI of 21 days. The maximum residue in the single trial in MI with 2 ground applications at 2 lb ai/A was 3.45 ppm (14 day PHI). The maximum residue reported in the single trial with 5 ground applications at 1.21 ppm in PA, was 8.63 ppm. Residues in control samples ranged from non-detectable to 0.27 ppm. Even with only 2 applications at 1.2 ppm, the existing tolerance in grapes is exceeded and must be increased. A tolerance of 10 ppm in grapes may be appropriate, provided only two ground applications of the flowable formulation at 1.2 lb ai/A is to be supported, and provided that dicofol is determined to be the sole residue of concern.

Grape Processing Study. (MRID No. 400420-28). Grape samples from the field trials discussed above were processed at California State University (Fresno). Both raisin and wine processing were done. A cryptic flow chart of the processes used were included in the submission. Raisin processing included production of raisins, stems, and raisin waste. Wine processing included production of juice, wine, lees (sediment), and pomace (skins).

The wine processing was described as follows. Sulfur

The grapes are crushed and pressed to separate the juice from the skins for white wine. The juice is fermented for one week and the sediment (lees) removed. For red wine, the skins and juice are fermented together with sugar added. The skins are separated from the juice (or the fermented wine) as pomace.

The results of the grape processing study are presented below in Table 6.

Table 6

Dicofol Concentration/Reduction Factors in Grapes

Commodity	Residue (ppm)		
fruit	1.00	0.62	0.27
raisin	3.64	5.96	--
waste	5.83	--	--
stem	--	3.60	0.58
juice	--	0.24	0.02
wine	--	<0.01	<0.01
wine lees	--	--	0.52
wet pomace	--	--	0.80
dry pomace	--	--	0.47
	Concentration/reduction factor		
fruit	1.00	1.00	1.00
raisin	3.64	9.61	--
waste	5.83	--	--
stem	--	5.81	0.94
juice	--	0.39	0.03
wine	--	<0.01	<0.01
wine lees	--	--	0.84
wet pomace	--	--	1.29
dry pomace	--	--	0.76

Regulated processed commodities are raisins, raisin waste, pomace, and juice. Dicofol residues are reduced in grape juice and wine. Dicofol concentrates in raisin waste and grape pomace. Feed additive tolerances will be needed for these grape processed commodities. Feed additive tolerances of 100 ppm for raisin waste (10 ppm x 5.8x = 58 ppm) and 20 ppm for grape pomace (10 ppm x 1.3x = 13 ppm) may be appropriate. A food additive tolerance of 100 ppm for raisins may be appropriate, depending on the tolerance established for grapes.

Dry Beans. (MRID NO. 400420-17). Two applications of Kelthane MF were made at 1.5 lb ai/A in nine locations in ID, CA, and NY. Five varieties of dry beans (apparently all red kidney beans) were grown. PHI's ranged from 19 to 40 days. In some locations, the bean was analyzed with the pod. In

some locations, the shelled bean was analyzed. In one study, beans and pods were analyzed separately. No data on bean forage and hay were submitted. No data on bean cannery waste were submitted. We note an increase in the residue reported with increasing PHI. This should be explained.

Residues reported ranged from 0.27 ppm to 4.6 ppm in beans w/ pod from aerial application, 0.27 ppm to 2.7 ppm in beans w/pod from ground applications, 21.0 to 21.8 ppm in bean pods (one location, varying PHI's), and non-detectable to 0.04 ppm in beans without pods from CA data. The rac is the dry bean without pod. (The rac for succulent beans is the bean with pod.)

The label rate is multiple applications at 1.5 lb ai/A with a 7 to 45 day PHI, depending on location. Feeding of bean forage and hay is restricted. Dry beans are grown primarily in MI (20%), CA (15%), NE (15%), ND (12%), ID (12%), CO (11%). Smaller amounts are grown in WY (4%), WA (3%), MN (3%), and NY (2%). Residue data for dry beans are needed from MI, CA, NE, ND, ID, and CO. Either data on dry bean forage and hay or data on dry bean cannery waste are needed. The principal varieties of dry beans are navy beans (24%), great northern beans (11%), pinto beans (35%), and red kidney beans (7%). These varities should be represented by residue data to maintain registration on dry beans.

Cotton. (MRID NO. 400420-25). Eight locations in four states were treated with Kelthane MF. Different application rates were used. Seven trials were done with 2 ground applications at 1.5 lb ai/A (CA (5 locations), TX (1 location), MS (1 location)). Two samples were taken from two locations in CA. One trial in AR had two ground applications at 1 lb ai/A. One locations in CA had one aerial application at 1.5 lb ai/A. PHI's ranged from 28 to 67 days. Most locations had samples harvested within 34 days after the last application. Cotton was ginned at Rohm and Haas Spring House Laboratories. Cottonseeds were delinted with concentrated sulfuric acid, washed with lime solution, rinced with running tap water, drained and dried at 120-130C prior to analysis. Residues reported were non-detectable (LOD 0.01 ppm) in all but one sample. One sample had a reported residue of 0.05 ppm dicofol (all p,p'-dicofol).

The principal cotton growing areas are TX (29%), CA (22%), MS (12%), AZ (9%), LA (8%), and AR (5%). Residue data are needed from TX, CA/AZ, and LA/AR/MS. The maximum label rate is multiple applications of 1.6 lb ai/A with a 14 day PHI. The submitted cottonseed residue data do not support any use of dicofol on cotton since the seeds were delinted before analysis. The rac is undelinted cottonseed. A single field trial with aerial application would not be sufficient to

maintain registration of aerial application. Additional cottonseed residue data are needed for each type of application (ground, aerial, ULV), and for each type of formulation to be used.

Cotton Processing study. (MRID NO. 400420-27). Two samples from the residue field trials were processed at Texas A&M. The cotton was ginned, yielding cottonseed, lint, and gin trash. The seeds were linted, yielding linters, motes, and delinted seeds. Cotton seed was then decorticated to separate the hulls from the kernels. The kernels were flaked to a thickness of about 0.01 inch, and extracted with hexane. The meal was desolventized with warm air. The crude oil was separated from the hexane, alkali refined with NaOH, refrigerated, and then filtered. The soapstock and refined oil were frozen until shipped to Rohm and Haas.

Two delinted cottonseed samples with non-detectable residues were processed. Apparently the cotton gin trash and undelinted cottonseeds were not analyzed. Non-detectable residues were found in the cottonseed hulls, meal, and soapstock. Detectable residues were found in both the crude oil and the refined oil of both samples. Residues were reported as 0.05 ppm and 0.08 ppm in the crude oil samples and 0.06 and 0.09 ppm in the refined oil samples. Dicofol concentrates in crude and refined oil. A Food additive tolerance will be necessary. An appropriate level cannot be determined since cottonseed samples with non-detectable residues were processed. We can make no conclusions about concentration of dicofol in hulls, meal, and soapstock, since cottonseed samples with non-detectable residues were processed.

Summer Squash. (MRID No. 400420-21). Three or four hand or ground applications of Kelthane 4F were made at 0.60 lb ai/A at nine locations in six states. Residue data were submitted from FL, CA, TX, GA, MS, and PA. Squash is grown primarily in CA (31%), FL (22%), NJ (9%), MA (6%), TX (6%), and NC (3%). (See Fruit and Vegetable Facts and Pointers, United Fresh Fruit and Vegetable Association, Alexandria, VA). The residue data adequately reflect the US production of squash. Whole squash were analyzed. Residues reported ranged from 0.07 ppm to 1.05 ppm at a PHI of 2 days. Residues in control samples were non-detectable. The existing tolerance of 5 ppm for squash will be adequate, and potentially could be lowered to 2 ppm, providing three ground applications of the flowable formulation at 0.6 lb ai/A with a PHI of 2 days is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

Cucumbers. (MRID NO. 400420-20). Ten residue field trials were conducted in seven states, TX, FL, CA, MS, GA, PA,

trials were conducted in seven states, TX, FL, CA, MS, GA, PA, and NC. Cucumbers are grown in MI (19%), NC (14%), WI (10%), CA (9%), OH (9%), TX (7%), SC (6%), MD (4%), OR (4%), and FL (3%). Additional residue data will be needed from WI/MI. Three ground or hand applications of Kelthane 4F were made at 0.60 lb ai/A. Samples were harvested with PHI's of 2-3 days. Residues reported ranged from 0.05 ppm to 0.45 ppm at a PHI of 2 days. Residues in control samples was non-detectable. The existing tolerance of 5 ppm in cucumbers may be adequate and could possibly be lowered to 1 ppm, provided that additional residue data are submitted from WI/MI, that two ground applications of the WP or F at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

Melons. (MRID No. 400420-18 and -19). Nine field trials were conducted in five states (TX, CA, MS, PA, and FL). Melons are grown in CA (69%), AZ (11%), TX (10%), CO (2%), GA (2%), IN (2%), MI (1%), OH (1%), and SC (1%). (1972 Statistics from Untied Fresh Fruit and Vegetable Facts and Pointers, 1973.) The submitted data adequately reflect the geographic areas in which melons are grown.

Three ground or hand applications of Kelthane 4F were made at 0.6 lb ai/A at approximately weekly intervals. In one trial in CA, 2 hand applications at 1.13 lb ai/A were made at a 6 week interval. PHI's were 0 to 10 days. Residues reported in canteloupe ranged from non-detectable to 0.35 ppm at a PHI of 2 days. A residue of 0.11 ppm was found in the one sample treated at 1.13 lb ai/A. Residues in control samples were non-detectable.

The maximum label rate for use on melons is multiple applications at 1.5 lb ai/A with a PHI of 2 days. The existing tolerance of 5 ppm in melons may be adequate and could possibly be lowered to 1 ppm, provided that two ground applications of the WP or F at 2 lb ai/A and a PHI of 2 days is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

Pecans. (MRID No. 400420-22). Two ground applications of Kelthane MF (EC) were made at 2.0 lb ai/A in two states, GA and TX. These states comprise 52 and 25% of the annual US pecan production. The areas represented by these states comprise 91% of the annual US pecan production. This is adequate geographical representation. Samples were harvested with PHI's ranging from 0 to 7 days. Nut meal (without hulls) was analyzed. No residues were reported in any pecan sample (LOD 0.01 ppm). The existing tolerance of 5 ppm in pecans may be adequate and could possibly be lowered to 0.1 ppm, provided that two ground applications of the EC at 2 lb ai/A is the only use to be supported, and provided that dicofol is

and NC. Cucumbers are grown in MI (19%), NC (14%), WI (10%), CA (9%), OH (9%), TX (7%), SC (6%), MD (4%), OR (4%), and FL (3%). Additional residue data will be needed from WI/MI. Three ground or hand applications of Kelthane 4F were made at 0.60 lb ai/A. Samples were harvested with PHI's of 2-3 days. Residues reported ranged from 0.05 ppm to 0.45 ppm at a PHI of 2 days. Residues in control samples was non-detectable. The existing tolerance of 5 ppm in cucumbers may be adequate and could possibly be lowered to 1 ppm, provided that additional residue data are submitted from WI/MI, that two ground applications of the WP or F at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

Melons. (MRID No. 400420-18 and -19). Nine field trials were conducted in five states (TX, CA, MS, PA, and FL). Melons are grown in CA (69%), AZ (11%), TX (10%), CO (2%), GA (2%), IN (2%), MI (1%), OH (1%), and SC (1%). (1972 Statistics from Untied Fresh Fruit and Vegetable Facts and Pointers, 1973.) The submitted data adequately reflect the geographic areas in which melons are grown.

Three ground or hand applications of Kelthane 4F were made at 0.6 lb ai/A at approximately weekly intervals. In one trial in CA, 2 hand applications at 1.13 lb ai/A were made at a 6 week interval. PHI's were 0 to 10 days. Residues reported in canteloupe ranged from non-detectable to 0.35 ppm at a PHI of 2 days. A residue of 0.11 ppm was found in the one sample treated at 1.13 lb ai/A. Residues in control samples were non-detectable.

The maximum label rate for use on melons is multiple applications at 1.5 lb ai/A with a PHI of 2 days. The existing tolerance of 5 ppm in melons may be adequate and could possibly be lowered to 1 ppm, provided that two ground applications of the WP or F at 2 lb ai/A and a PHI of 2 days is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

Pecans. (MRID No. 400420-22). Two ground applications of Kelthane MF (EC) were made at 2.0 lb ai/A in two states, GA and TX. These states comprise 52 and 25% of the annual US pecan production. The areas represented by these states comprise 91% of the annual US pecan production. This is adequate geographical representation. Samples were harvested with PHI's ranging from 0 to 7 days. Nut meal (without hulls) was analyzed. No residues were reported in any pecan sample (LOD 0.01 ppm). The existing tolerance of 5 ppm in pecans may be adequate and could possibly be lowered to 0.1 ppm, provided that two ground applications of the EC at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

Walnuts. Six field trials were conducted in two locations in California. Essentially all walnuts grown in the US are grown in California. Two hand applications of Kelthane MF were made at 2 lb ai/A. Samples were harvested with PHI's of 7 days. Nuts were removed from the shells and air dried overnight. Nuts were then ground with dry ice, the dry ice sublimed in the freezer, and the samples shipped in dry ice to Analytical Development Corporation for analysis. No detectable residues were reported in any walnut sample (LOD 0.01 ppm). The existing tolerance of 5 ppm in walnuts may be adequate and could possibly be lowered to 0.1 ppm, provided that two ground applications of the EC at 2 lb ai/A is the only use to be supported, and provided that dicofol is determined to be the sole residue of concern.

MEAT, MILK, POULTRY, AND EGGS

Registration Standard Data Gaps

Conventional animal feeding studies with large ruminants and poultry will be required to establish the extent of transfer of residues to meat and milk, poultry and eggs. These studies must be conducted at feeding levels which reflect 1x, 3x, and 10x those of the established tolerances for commodities and/or byproducts used as livestock feed, and at rates at which these commodities and/or byproducts are fed.

Current Submission

Feeding studies were submitted for cattle and poultry. These feeding studies cannot be accepted until the metabolism of dicofol in animals is adequately understood.

Our calculation of dietary intake of dicofol for cattle and poultry, based on current tolerances, is presented below. Note that spent hops, spent mint hay, and bean cannery waste are processed commodities, and thus, not under grower control. Therefore, feeding restrictions for these commodities on dicofol labels would not be practical and these commodities have been included in the livestock diets.

Table 7

DIETARY INTAKE OF DICOFOL IN BEEF CATTLE

Feed Item	Tolerance (ppm)	% in diet	Contribution (ppm)
Apple Pomace	250	50	125.
Raisin Waste	100	10	10.
Hops, spent	30	5	1.5
Mint, spent hay	25	25	6.25
Citrus pulp	10	10	1.0
			233 PPM

Table 8

DIETARY INTAKE OF DICOFOL IN DAIRY CATTLE

Feed Item	Tolerance (ppm)	% in diet	Contribution (ppm)
Apple Pomace	250	25	62.5
Raisin Waste	100	10	10.
Hops, spent	30	5	1.5
Mint, spent hay	25	60	15.
			89. PPM

Table 9

DIETARY INTAKE OF DICOFOL IN POULTRY

Feed Item	Tolerance (ppm)	% in diet	Contribution (ppm)
Apple Pomace	250	5	12.5
Bean Seed	5	15	0.45
Tomato Pomace	5	3	0.15
Grape Pomace	20	3	6.
Cottonseed Meal	0.1	10	0.01
Soapstock	0.1	5	0.005
			19.1 PPM

Cattle Feeding Study (MRID No. 400420-30)

Three groups of four cows were dosed with dicofol by capsule for 28 days. An additional four cows served as controls. The feeding levels were 10, 30, and 100 ppm, based on feed consumption of 19 kg food per day. The actual doses were given and were close to the nominal doses. The test material was reported to contain

95% dicofol. However, no analysis of the test material was given. This is required.

Residues of dicofol did not plateau in the milk within the 28 day study period. Three of the four cows in each group were sacrificed on day 29 (one day after the last dose). The fourth cow in each group was sacrificed after a seven day depuration. Samples were analyzed for o,p'- and p,p'- isomers of dicofol, DCBP, and FW-152 (1,1-bis(p-chlorophenyl)-2,2-dichloroethanol). The submitted report contained only selected samples from the 100 ppm feeding level group. No results were reported from the 10ppm and the 30 ppm feeding level groups. Thus the report is incomplete. Additionally, no storage stability data were submitted for meat commodities. These data are required. We note that the need for storage stability data in meat commodites is mentioned in both the cow feeding study and the hen feeding study report.

The maximum residues in meat and milk were reported as found in Table 10.

Table 10

Maximum Residues in Meat and Milk (ppm)

	dicofol		DCBP		FW-152	
	o,p'	p,p'	o,p'	p,p'	o,p'	p,p'
milk	<0.01	1.14	<0.01	<0.01	<0.01	0.13
beef						
muscle	<0.05	0.63	<0.05	<0.05	<0.05	4.32
kidney	<0.05	0.35	<0.05	<0.05	0.21	2.16
liver	<0.05	<0.05	<0.05	<0.05	0.23	2.82
fat	<0.15	<0.15	<0.15	0.68	2.87	54.5

Hen Feeding Study (MRID No. 400420-31)

Three groups of ten white leghorn hens were dosed for 28 days with dicofol. An additional ten hens served as controls. Dosing was done at 5 ppm, 15 ppm, and 50 ppm, using capsules. The dose was based on a feed comsumption of 115 g per day. The actual dose was calculated and was close to the nominal dose. Eggs were collected about every other day, and pooled by dose group. On four days, the eggs were separated into yolk and white. Six hens from each group were sacrificed one day after the last treatment. Two from each group were sacrificed after a 1 week depuration, and the remaining 2 birds were sacrificed after a 2 week depuration. The researchers reported that some soft shelled eggs were produced (eggs without calcium deposition). They reported that the incidence of soft shelled eggs could not be correlated to the

treatment dose. (pp 015-016, MRID No. 400420-31). EEB should be informed of this observation. Necropsy results were reported as well.

Residues did not plateau in eggs during the 28 day study period. Only results from the 50 ppm feeding level were included in the submitted report. Storage stability data and data from the other two feeding levels were not included. The report indicates that a storage stability study in hen tissues was in progress. Raw data and chromatograms of egg and tissue samples were included in the report.

The residues found in eggs and poultry tissues were reported as found below in Table 11.

Table 11

Maximum Residues in Poultry and Eggs (ppm)

	dicofol		DCBP		FW-152	
	o,p'	p,p'	o,p'	p,p'	o,p'	p,p'
eggs	<0.03	0.62	<0.03	0.76	<0.03	0.06
chicken						
breast	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
thigh	<0.05	0.23	<0.05	0.08	<0.05	<0.05
gizzard	<0.05	0.23	<0.05	0.08	<0.05	<0.05
heart	<0.05	0.50	<0.05	0.08	<0.05	0.05
kidney	<0.05	0.29	<0.05	0.08	<0.05	<0.05
fat	<0.15	3.13	<0.05	<0.05	<0.05	0.16

RCB Comments on Cattle and Poultry Feeding studies

The cattle and poultry feeding studies submitted are not complete and are not acceptable. The complete study (all three feeding levels) must be submitted, along with storage stability data. Although the feeding studies are not complete, we are able to determine that tolerances for cattle and poultry products will be necessary, since dicofol residues transfer to meat, milk, poultry, and eggs. However, without the complete study, and complete data and resolution of metabolism issues, we cannot determine appropriate tolerance levels.

OTHER CONSIDERATIONS

Compatibility with CODEX was discussed in the Registration Standard. This issue will be discussed further when the metabolism issues are resolved and the residue data deficiencies are resolved.

cc: circu, R.F., S. Hummel, Dicofol Special Review File (S. Hummel), dicofol S.F., dicofol Reg. Standard File (Boodee) TOX, EAB, EEB (L. Turner), SRB (B. Kapner), PMSD/ISB
RDI:EZ:10/19/87:KHA:10/19/87
TS-769:RCB:SVH:svh:CM#2:RM810:10/19/87