File PP#7F0599

September 20, 1967

AF 25-202
(Diamond Alkali Co.)

Petitions Control Branch and
Division of Toxicological Evaluation

Pesticides Branch, Division of Food
Standards and Additives

PP #7F0599, Daconil on various crops.

Attached is the evaluation of the residue data and analytical method in the subject petition from Division of Food Chemistry. We concur in their findings. There is one minor discrepancy on page 10, paragraph 6, regarding the range of residues present on sugar beet crowns vs. roots plus crowns. We have discussed this with Miss Malone and find that this is the way the data were reported by the petitioner. If further action is contemplated on this petition, it would be advisable to have the petitioner clarify how the plant fractions were prepared.

J. G. Cummings

Attachment
(memo 8/28/67 - Malone to Alpert)

cc:
DTE
SCI-R
SCI-F
SCI-OD
DFC
FSA/OD
FSA/PB
PP #7F0599

JGCummings:jrf
9/18/67 & 9/20/67

Jerry Alpert, Chief, PB/FSA/SCI
Through: Howard A. Jones, Chief, PB/DF/SCI

August 28, 1967

Bernadette Malone, PB/DF/SCI

PP #7F0599: Tolerances for Tetrachloroisophthalonitrile (Daconil 2787) on various vegetables. Evaluation of analytical method and residue data.

Diamond Alkali Company proposes tolerances for residues of tetrachloroisophthalonitrile (Daconil 2787), of 15.0 ppm on celery; 7.0 ppm on snap beans; 5.0 ppm on broccoli, brussels sprouts, cabbage, carrots, cauliflower, cucumber, melons (cantaloupe, honeydew, muskmelon, watermelon), pumpkin, summer squash, winter squash, and tomatoes; 1.0 ppm on lima beans and sweet corn; 0.1 ppm on peanuts, white potatoes, and sugar beets.

A petition for a temporary tolerance for 0.1 ppm Daconil on potatoes (PP #7G0516) was presented and approved in September, 1966. The petitioner withdrew his petition in October, 1966, for unstated reasons.

Conclusions

The question of metabolites or degradation products has still not been answered by the petitioner, despite the statement by R. Quick in the 8/26/66 memo on PP #7G0516 that an exploration of the fate of Daconil would be necessary before requesting permanent tolerances. Although studies show that there is no translocation of Daconil (or its possible metabolites) from roots to aerial portions, the incorporation of the metabolite(s) by root crops is not known.

The analytical method is considered adequate for presentation of most of the residue data in the petition (those on celery excepted). For enforcement purposes, evidence showing specificity is required and a better extraction system would be preferable. The uncertainty of the metabolic situation precludes a statement concerning the ability of the method to detect all metabolites. The three metabolites now known would probably not be determined by this method.

Residue data in most cases were acceptable. With certain modifications of directions for use, all requested tolerances would appear to be acceptable on the basis of residue data.

Absence of significant meat and milk transfer data prevent consideration of tolerances on peanuts, lima beans, and sweet corn.

Soil persistence studies indicate that parent Daconil will probably degrade before a subsequent year's planting. Evidence shows that the metabolite hydroxytrichloroisophthalonitrile will replace the Daconil; no soil persistence tests have been done on this compound, however.

Recommendations

1. The metabolic or degradation products of Daconil must be explored further before action can be taken on this petition. Necessary information includes:

   a. Metabolism or degradation of Daconil on the plant.

   b. An analytical method that extracts and detects Daconil and the soil and plant metabolites or degradation products.

   c. Presence or absence of the soil metabolite, hydroxytrichloroisophthalonitrile, on plants and in root crops.

   d. Persistence of the soil metabolite.

2. Specificity of the method for Daconil and metabolites in the presence of other pesticides allowed on these commodities must be shown; a more efficient extraction than the one in this petition is preferred.

3. If the metabolism studies and method were acceptable, recommendations on the requested tolerances on the basis of residue data would be:

   a. Establishment of 5.0 ppm on broccoli, brussels sprouts, cauliflower, cabbage, cucumbers, summer squash, cantaloupe, muskmelons, honeydew melons, winter squash, watermelons, pumpkins, and tomatoes; of 7.0 ppm on snap beans.

   b. Establishment of 0.1 ppm on sugar beets if the petitioner imposes a 14-day PHI or provides evidence that residues will be below tolerance immediately after treatment.

   c. Establishment of 15.0 ppm on celery if the petitioner imposes a 14-day PHI.

   d. Establishment of 5.0 ppm on carrots (without tops) and 0.1 ppm on potatoes if the petitioner presents evidence showing residues would not be transferred to meat or milk. Without this evidence, the tolerances could still be accepted with a restriction against feeding of cull potatoes and carrots and carrot tops to livestock and against the use of carrot tops as food.

e. Establishment of 0.1 ppm on peanuts and 1.0 ppm on lima beans (without pods) and sweet corn if the petitioner presents evidence showing residues would not be transferred to meat or milk. Without this evidence, the tolerances could not be recommended, since restrictions against feeding peanut hulls, lima bean foliage and sweet corn cannery waste are not practical.

Detailed Considerations

Proposed Usage

Daconil is distributed as Daconil 2787 W-75, a wettable powder, containing 75.0% tetrachloroisophthalonitrile (Daconil) and 25.0% inert ingredients. It is applied as a spray in enough water to provide thorough, uniform coverage.

Because of the large number of crops involved, specific usage directions will be given along with the residue data.

Nature of the Residue

Soil metabolism: Experiments with C^{14} ring-labelled Daconil show the appearance of two metabolites in soil as the parent compound degrades. By the time the Daconil had disappeared (46 days) 70-80% of the initial radioactivity was found in a metabolite identified as a salt form (probably) of hydroxytrichloroisophthalonitrile. The second metabolite has not been identified.

Uptake by plants: Studies to determine the extent of incorporation of Daconil by the plant also used the C^{14} labelled fungicide. Measured drops of labelled Daconil in aqueous suspension were placed on aerial plant parts, with physical restrictions to prevent movement of the drops on the surface. In addition, corn seeds and tomato transplants were planted in soil treated with Daconil to measure uptake from the soil. The location of the radioactivity in all these tests was determined by exposure of radiation sensitive emulsions to the harvested plants. Leaves spotted with labelled Daconil and exposed for two to seven days from treatment to harvest showed no translocation of radioactivity to other areas of the plant. Only the roots of corn and tomato plants grown in treated soil indicated the presence of radioactivity, showing a failure of the plant to incorporate Daconil into aerial portions. In root crops such as carrots and sugar beets, this uptake by the roots may be an important consideration.

Animal metabolism: Dogs fed Daconil on a one dose basis excreted 85-90% of the unchanged compound in the feces, usually within 48 hours. No Daconil was found in urine or blood.

Chronic feeding studies using dogs and rats show an inverse relationship between the amount of Daconil ingested and the unchanged compound excreted in the feces. Dogs fed a diet containing 3% Daconil (30,000 ppm) excreted 85% in the feces. Total Cl analyses of these feces showed that all ingested Cl was excreted there; i.e., any metabolites were also in the feces. At 1.5 and 0.15% (15,000 and 1,500 ppm) feeding levels, only 69% and 12% unchanged Daconil was found in feces of the respective animals. No total Cl tests were run on these.

Two minor metabolites were isolated from feces and identified as trichlorodicyanoaniline (probably present in conjugated form) and 2,4,5-trichloroisophthalonitrile. No hydroxytrichloroisophthalonitrile (the soil metabolite) was found. The petitioner has characterized the remaining metabolite(s) as highly polar and strongly hydrophilic, but has not isolated and identified any.

Labelled Daconil fed to rats showed 88.45% excretion in feces and 5.14% in urine over 264 hours. Storage in tissues was negligible: organs, G.I. tract and carcass contained a total of 0.5% of the ingested amount; 0.32% was expired as $C^{14}O_2$.

Plant metabolism: No significant studies of metabolism or degradation on the plants themselves has been presented. Potato peels extracted with acetone and analyzed by TLC purport to show the absence of trichlorodicyanoaniline. It is unlikely, however, that this metabolite would be extracted by acetone were it present. (PP #7G0516 also reports the absence of the postulated metabolites resulting from nitrile hydrolysis, tetrachlorocyanobenzoic acid and tetrachloroisophthalic acid.)

The possible presence of residues of hydroxytrichloroisophthalonitrile on crops has been ignored. This compound can comprise as much as 80% of the soil residue and absence of such data on plants is an unexplained deficiency in this petition.

Analytical Method

Residues of Daconil 2787 are extracted from the sample either by tumbling 500 g of smaller commodities for two hours with 600 ml dichloromethane; rinsing the larger commodities with a stream of dichloromethane; or blending 500 g of a chopped sample with 1L of dichloromethane for one minute and then tumbling for two hours. Extracts from the first two procedures are concentrated for cleanup by column chromatography; in the blending procedure, the extract is filtered before evaporation.

Cleanup is performed on a column of activated alumina by first eluting interferences with 100 ml dichloromethane and then the Daconil with 100 ml 5% acetone/dichloromethane. The sample is evaporated to dryness, taken up in benzene or xylene, and analyzed by either microcoulometric or electron capture gas chromatography.

Chromatograms of standard solutions using both type chromatographs and MC-GLC chromatograms of plant samples were provided; all are satisfactory. No sensitivity limits were suggested, but 0.02 ppm sensitivity by MC-GLC can be estimated.

Most residue data presented were determined after surface extraction. Results of this were shown to be equivalent to those obtained by maceration. Maceration of watery samples by blending with a water-immiscible solvent like dichloromethane is not a preferred extraction technique for weathered pesticides. Pesticide literature (1,2,3,4) points out problems in obtaining efficient extraction. We believe that this method may extract as little as 80% of the compound present. Except for celery, residues presented in this petition are far enough below the tolerances requested so that we can accept them despite this deficiency.

Recovery data were presented on 17 of the crops, using tumbling and/or blending extraction. These were generally satisfactory but fortified samples give no indication of completeness of extraction of weathered residues. In cases where recovery levels did not reflect the requested tolerance, recoveries on other crops could be applied. No data were presented on recoveries from large, surface-rinsed crops (cantaloupe, honeydew, watermelons, pumpkins), presumably because of the difficulty in fortifying such a sample. We are willing to assume such a rinse was sufficient, in light of the surface nature of the residue and the nature of these crops.

Several other pesticides which might possibly interfere with Daconil in analysis have tolerances on some of the crops involved. CIPC, BHC, lindane, and dichlone are all permitted on one or more of these crops and could have GLC retention times interfering with Daconil. Petitioner should show specificity of the method for Daconil and metabolites in the presence of the above pesticides. Similar specificity requirements are necessary for any metabolites in the presence of any other pesticide permitted on the commodities.

The metabolites identified by the petitioner would probably not be extracted and determined by this method. The trichlorodicyanoaniline was extracted with dichloromethane, but from an alkaline aqueous suspension; the 2,4,5-trichloroisophthalonitrile is water soluble. Hydroxytrichloroisophthalonitrile was extracted from soil with acetone:water: conc. H_2SO_4 in 48:1:1 (by volume). Preparation of the trimethylsilyl ether of the latter metabolite is necessary for gas chromatography.

Residue Data, including Recommended Usages

(The dosage in terms of pounds in each case refers to Daconil 2787 W-75 (75% act.) in sufficient water to obtain adequate coverage.) Residue data were obtained after application of Daconil at rates either according to or in excess of those directed. We feel that repeat applications are sufficient in number to include maximum usage.

Carrots: Apply 1.5-2 lb/A when disease threatens; repeat at seven to ten day intervals or as necessary to maintain control.

Residue data reflect both the directions for use and heavier doses; from 1.5-3 lb and from seven to twelve applications. Results indicate that 2 lb is the maximum rate for which residues will be below the requested tolerance of 5.0 ppm. The range of residues on unwashed roots and crown from a plot sprayed with 2 lb/A for nine applications and no days from final treatment to harvest (pre-harvest interval, or PHI) was 2.21-3.44 ppm; mean was 2.78 ppm. As little higher a rate as 2.25 lb, with nine applications, and two days PHI results in residues >5 ppm on unwashed roots and crown. Three lb treatments result in correspondingly higher residues.

Washing reduces the residues in all cases, insuring residues within the requested tolerance.

Blanks ranged from .00-.03 ppm. One set of results on unwashed roots only ranged from .00-.05 ppm; treatment had been seven 3 lb/A applications with seven days PHI. This indicates that most of the residue is on the carrot tops, a logical result for a foliar spray. Establishment of the tolerance on carrots (without tops) would therefore be possible, provided there is a restriction against the use of the tops as food. To establish the tolerance on carrots with tops, additional data would be required showing that residues on tops only would be within the tolerance (see Pesticide Regulations, paragraph 120.1 (j)(6).)

A restriction against the feeding of cull carrots and carrot tops to livestock will be necessary unless data are presented showing a failure of residues to transfer to milk and meat.

Crucifers: Apply 1.5 lb/A after transplants are set in field or the field-seeded crop has reached transplant size and conditions favor disease. Repeat at seven to ten day intervals or as needed. Do not apply to plants in seedbed. The proposed tolerance for crucifers is 5 ppm.

Broccoli treated at 3 lb/A six times contained residues of 0.35-1.57 ppm when harvested with no day PHI, 0.57-2.56 ppm with five-day PHI; and 0.00 at 12 day PHI. All samples were surface extracted.

Brussels sprouts treated five times with 1.5 lb/A contained residues of 1.09-3.54 ppm at no days PHI and less with longer PHI. Treatments with 4.0 lb/A resulted in residues as high as 11.3 ppm; again, a longer PHI resulted in lower residues. Only one sample was extracted by maceration; the remainder were surface extracted. In that one case, however, residues were comparable to the surface residue of sample receiving identical spray treatment.

Surface extraction of total cauliflower heads after 10-3 lb/A sprays and no day PHI gave 0.30-1.92 ppm residues. Both longer PHI and the tying of leaves over the heads for blanching reduced this residue. When curds only were surface extracted with 33 days PHI, residues were 0.03-0.25 ppm.

Field trimmed cabbage sprayed nine times at 2 lb/A with no day PHI contained 0.77-2.67 ppm Daconil. Market trimming reduced these values to negligible.

Recoveries and controls are generally satisfactory for all the crucifers.

Cucumbers: Apply 1.5-3 lb/A, beginning when plants are in first true leaf stage or when conditions are favorable for disease. Repeat at seven-day intervals or as needed.

Surface extractions of cucumbers treated with 1.0-3.0 lb/A for 1-12 applications all result in residues well within the 5.0 ppm requested tolerance (maximum residue was 2.52 ppm, for 12 sprays of 2 lb/A with no days PHI). Some of the sprays contained a spreader sticker, but this does not appear to affect residues significantly.

Water washing of the crop before surface extraction removes most residue (e.g., 0.39 is reduced to 0.02 ppm). Analysis of peelings alone and the remaining peeled portion alone corroborate experimental evidence that the compound is not absorbed -- essentially all the residue is in the peel.

Recoveries are adequate and control samples generally negligible.

Melons and Gourds: Apply 1.5-3 lb/A, beginning when plants are in the first true leaf stage or when conditions favor disease. Repeat at seven-day intervals or as needed. The proposed tolerance for this group is 5 ppm.

Summer squash was treated with 1-3 lb/A for 1-9 applications and surface extracted. All residues ranged from 0.00-2.02 ppm, including those where spreader stickers were added to the spray. Water washing of the sample before surface extraction reduced residues to 0.00-0.39 ppm.

Cantaloupe residues ranged from 0.00-1.12 ppm after 8-11 sprays at 2 lb/A. Surface stripping of the whole melon was used for extraction. Although the maximum rate of 3 lb/A was not studied, it can be safely assumed from the 2 lb/A results and from 3 lb/A results on the other plants in this group that the residues from this dosage would be within the 5.0 ppm tolerance.

Muskmelons were treated with 11 sprays of 1.5 and 3.0 lb/A and harvested the day of the final treatment. Macerated rind gave residues of 1.35-1.70 ppm at the 3 lb/A rate. No residues were found in macerated pulp at either level.

Honeydew melons were surface stripped after 2 and 3 lb/A treatments of seven to ten sprays. All residues were well within the requested tolerance, maximum being 1.64 ppm found after 10-2 lb/A sprays with no day PHI.

Winter squash, surface extracted after 5-10, 2 and 3 lb/A, treatments, gave residues of 0.04-2.26 ppm. Rind macerated after surface extraction gave an additional 0.02-0.13 ppm residue. No residue was found in the pulp.

Watermelons were subjected to surface extraction only. One-three lb/A doses at eight to ten applications resulted in residues ranging from 0.00-0.99 ppm; the higher residues are produced by higher dosage rates and shorter PHI, as is generally true.

Surface extraction of pumpkins treated with 2 lb/A, ten times, showed residues of 1.05-1.79 ppm at no days PHI and 0.76-0.87 ppm at seven days PHI. Further analysis of the no day PHI rind after surface extraction produced 0.08-0.13 ppm residue. No residue was found in edible pulp. As mentioned for cantaloupes, use of 3 lb/A can be assumed to cause no residues exceeding the proposed tolerance.

Control samples for all melons and gourds have none or negligible values. Recoveries were performed on muskmelon and winter squash using the maceration extraction of pulp and rind. Recoveries from rind averaged 62-77.5%; from pulp, 97-102%. No recovery studies were performed with surface stripped extractions, but maceration extraction of the rind after a surface stripping extraction indicates that more than 90% of the rind residue is removed by the surface strip.

Tomatoes: Apply 1.5-3 lb/A, beginning when disease threatens. Repeat at seven to ten day intervals or as needed.

Tomatoes were sprayed with 0.75-3.0 lb/A for 4-18 applications and then surface extracted for analysis. In two cases, anomalous results occurred. After 10 sprays with 1.5 lb/A and one day PHI, five out of six results were close to or at the requested 5.0 ppm tolerance (4.19-5.00 ppm found). With the same treatment and seven day PHI, three out of eight results ranged from 4.72-5.49 ppm. Both these tests were run in Michigan. In Ohio, a plot sprayed six times with 2 lb/A and five day PHI gave residues in four out of eleven tests ranging from 4.11-5.45 ppm. These results seem inconsistent with residues of 0.31-0.57 ppm obtained after five

treatments at 3 lb/A and no day PHI. The latter study was done in California and the difference in climate is a possible reason for the variety in results. A spreader sticker was employed with the spray in the two high residue instances. In many other tests, however, such a substance has been used without leading to higher residues.

Almost 300 results were presented for tomatoes, and only three of these were residues of 5 ppm or above. About 85% of the results showed residues below 3 ppm. In this light, the aberrant results mentioned can probably be discounted as contaminated plots.

Analyses show that water-washing removes 90% of the residues.

Control samples generally show negligible values, and recoveries range from 68-94%.

Peanut: Apply 1.5 lb/A, beginning when disease first appears. Repeat at 10-14 day intervals or as needed. Do not feed peanut hay to livestock. Proposed tolerance is 0.1 ppm.

Residue data are given on meats only and hulls only after seven applications at 1.5 lb/A, with 6 and 21 days PHI. No residue was found in the meat at either date. Hulls with seven days PHI showed 0.42-0.53 ppm residue; at 21 days PHI, levels were reduced to 0.00-0.20 ppm. Pesticide Regulation §120.1 (j)(2) specifies that nut meats only, and not hulls, are to be analyzed for pesticide residues. A no day PHI would most likely result in higher than the seven-day residues on hulls, but other analyses showing lack of absorption indicate that there would be no nut meat residues even with no day PHI.

The use of peanut hulls as a constituent in animal feed presents a problem because the grower lacks the control to restrict the use of hulls as feed. Assuming that the hulls can constitute 1.5-5% of the diet of dairy cattle, a significant residue of Daconil in the total diet could occur. Studies on transfer of residue to meat and milk therefore are necessary before the establishment of a tolerance on peanuts can be considered.

The label restriction against the feeding of peanut hay to livestock is considered a practical one. There also should be no problem involved with peanut meal or peanut oil, since no residues were found in the nut meat.

Recoveries were run at 1.54 ppm on hulls, a level not consistent with the 0.1 tolerance requested. Other recovery studies at 0.04 ppm (on potatoes) and 0.05 ppm (on carrots) gave mean results of 80% and 96% respectively. This would probably indicate successful removal of the lower residues from peanut hulls as well.

The two control tests run gave 0.00 and 0.10 ppm Daconil in the blank hulls.

Potatoes: Apply 1-1.5 lb/A, beginning when plants are six inches high or when disease threatens. Repeat at seven to ten day intervals or as needed.

Residue data are presented on surface extracted and macerated potatoes, potato peelings only and peeled potatoes only, after treatments of 1-3 lb/A with 3-13 applications and 0-23 days PHI. In all cases, results are below the 0.1 ppm requested tolerance. The highest residue found on whole potatoes was 0.07 ppm and more than 80% of the results are below 0.02. The highest residue found at all was 0.15 ppm in the peelings; since the peelings are 10% of the whole potato by weight, this would be 0.015 ppm on the whole potato.

The R. Quick memo (8/26/66) on PP #7G0516 suggested a restriction against feeding treated cull potatoes to livestock to safeguard against transfer to meat and milk. The petitioner agreed to add this restriction to his label at that time. We feel this restriction should again be imposed, unless the petitioner presents information showing failure of the residue to transfer to meat and milk. Recoveries and control samples are satisfactory.

Sugar Beets: Apply 1.5-2 lb/A in 50-125 gal H_2O, beginning when disease threatens. Repeat at 10-14 day intervals or as needed. Do not feed treated sugar beet tops to livestock.

Residue data are presented on roots with and without crowns, crowns only, and tops only. Treatments were four to six sprays of 2 or 3 lb/A. All results on roots less crowns show no residue, so should meet the 0.1 ppm requested tolerance. With crowns, residues are as high as 1.22 ppm. Crowns without leaves contained 0.00-0.57 ppm and tops alone had 6.76-32.0 ppm.

The minimum PHI for which residue data are provided is 14 days. Because of this, the petitioner should either impose a 14 day PHI or provide data on roots with a no day PHI.

Removal of crown and tops by the grower is common agricultural practice, so there should be no problem of residues in processed products such as sugar and sugar beet pulp, as long as the roots have no residue. This practice also makes the restriction against livestock feeding of tops practical and acceptable. Recoveries are satisfactory and residues on control samples negligible.

Sweet corn: Apply 1.5-2 lb/A, beginning when conditions favor disease development. Repeat at four to seven day intervals as required for control. Do not feed treated forage to livestock.

Residue data are provided on the kernels plus cob only at 1.5-3 lb/A, 10-14 applications and 0-14 days PHI. All residues from both surface extraction and maceration range from 0.00-0.13, well within the requested tolerance of 1.0 ppm. Husks from plots treated with 1.5 lb/A 14 times with 14 day PHI and extracted by maceration contained residues of 0.68-12.2 ppm. Since the raw agricultural product is the kernels plus cob with husks removed, this tolerance can easily be met.

The feeding of stalks and leaves to livestock is within the control of the grower and a practical restriction. Sweet corn cannery waste, however, loses it identity and its use cannot be restricted. Therefore, studies of transfer of residue to meat and milk must be provided before a tolerance on sweet corn can be considered. Recoveries on 0.4-1.0 ppm were 72-108%; control values were negligible.

Snap beans: Apply 2-3 lb/A, beginning during early bloom or when disease first threatens. Repeat at weekly intervals as necessary. Do not apply within seven days of harvest. Do not feed treated plant parts to livestock.

Residue data was provided on crops treated with eight applications of 2-3 lb/A, with zero to seven days PHI. (One set of results on beans sprayed at 2 lb/A five times, with no day PHI was disregarded because control values averaged 4.78 ppm and contamination is suspected.) The seven day PHI is necessary to bring residues within the requested tolerance of 7.0 ppm. At 3 lb/A, residues at zero, three and seven days PHI have ranged of 2.75-9.71, 2.55-9.90 and 0.81-4.66 ppm, respectively. At 2 lb/A, maximum residue (occurring with no day PHI) was 3.24 ppm.

The restriction against feeding to livestock is considered practical and acceptable. Except in the case already mentioned, control values were negligible; recoveries were satisfactory.

Lima beans: Apply 2 lb/A, beginning during early bloom stage or when disease first threatens. Repeat at seven-day intervals or as needed. Do not feed treated plant parts to livestock. Proposed tolerance is 1.0 ppm.

Residue data are presented for beans only, beans plus pods, and plants only, sprayed at 1.5-2 lb/A 7-13 times, with 0 and 15 day PHI. Beans only had residues of 0.00-0.39 ppm, pods and beans, 10.1-12.9 ppm, and plants only, 22.0-535 ppm.

The tolerance of 1.0 on lima beans cannot be permitted, because the raw agricultural commodity is the beans plus pod, and residues on these are above this level. A tolerance on lima beans with pods removed and discarded might be considered, however.

The restriction against feeding treated plants to livestock is considered impractical, in view of the usual practice of sending the whole plant to the processor. Because of this, a tolerance on lima beans cannot be allowed unless data are presented showing lack of residue transfer to milk and meat. Such data should reflect actual residues found in lima bean plant parts.

Control samples had one set of inexplicably high values (up to 79.2 ppm) and one recovery of 39%. Other controls were 0.00-0.15 ppm and other recoveries 74-106%.

Celery: Apply 1-3 lb/A, beginning when transplants are set in field. Repeat at three to seven day intervals, depending on the disease involved. Do not apply within seven days of harvest. Remove residue by stripping, trimming, and washing.

Residue data indicate that both the PHI imposed and the directions to strip, trim and wash the celery are necessary, but not always adequate in keeping the residue below the requested tolerance of 15.0 ppm. (This type of handling is a practical direction, being common practice.) Field-trimmed celery from plots sprayed 24 times with 1.5 lb/A contained residues of 9.36-51.7 ppm after no day PHI, and 6.42-14.1 ppm after seven days PHI; field-washed celery from the same plots contained 4.50-20.6 ppm and 7.70-9.36 ppm at zero and seven days, respectively.

At the highest recommended dosage of 3 lb/A, no data are given on field-washed material. This is important because even at the lower rate of 2 lb/A (24 applications), one field-washed sample in the eight presented contained more than the tolerance, i.e., 16.2 ppm. Residues on field-trimmed (but not washed) samples from plots sprayed with 3 lb/A were shown to range from 0.57-14.5 (plus one at 31.3 ppm) after five applications and seven days PHI.

We are not convinced that all residues will be less than 15 ppm at the higher dosage rates of 2 and 3 lb/A. In one to six samples from three different plots, residues on unwashed celery went over the tolerance; other values came very close to tolerance. Even on washed celery, one value, already mentioned, exceeded 15 ppm. Considering the fact that we do not have complete confidence in the extraction method, these values are considered too close to the tolerance to be allowable. We suggest changing the PHI to 14 days if the 15.0 ppm tolerance is desired. This restriction would apparently bring the residue, even on unwashed celery, below 15 ppm. Control plots generally contained negligible residues and recoveries at 0.20-0.67 ppm ranged from 68-125%.

Meat and Milk

The petitioner included a paper published by W. H. Gutenmann and D. J. Lisk in the J. Dairy Science, 49, 1272-1276, showing effects of feeding 5 ppm Daconil to a lactating cow. Daconil residues were not detected in milk, urine or manure (0.03 ppm method sensitivity), nor were there any acid metabolites or conjugates of these found. In vitro tests showed that rumen fluid metabolized Daconil to two unidentified metabolites with GLC retention times shorter than that for Daconil.

This test is not sufficient evidence to show a failure of Daconil residues to transfer to meat and milk. Only one cow was used, no tissue analysis was performed, and a 5 ppm feeding level is not reflective of the residues that might be encountered were cows fed plants or parts of plants treated with Daconil.

In order to consider establishment of a tolerance for peanuts, lima beans and sweet corn, or to permit feeding of the by-products of other crops to beef or dairy animals, we will need data from a more adequate study showing conclusively whether or not Daconil and derived products are transferred to and stored in meat and milk.

Persistence in the Soil

Experiments to determine the degradation of Daconil in soil were performed under both laboratory and field conditions. Laboratory tests showed that the half-life of Daconil under the conditions used ranged from four days to more than 70 days. Soil type appeared to have the greatest effect; eight different types gave half-life results ranging from 4 to 40 days, at the same moisture content and temperature. Both increasing moisture and increasing temperature sped the degradation process, but pH within 6.5-8.0 (the range found in the soils) had no significant effect. Autoclaving the soil sample to kill microorganisms slowed the degradation by about one-half, but did not stop it. Experiments in tightly closed containers proved that the degradation is a chemical or biological process and not a leeching or vaporization.

Georgia, Mississippi, Ohio, and South Dakota were sites of field degradation studies. Georgia and Mississippi tests agreed with laboratory tests, showing half-lives within the range reported in the laboratory and showing that degradation occurred more slowly during colder weather. Ohio and South Dakota tests were of questionable value. The Ohio tests showed that Daconil in treated soils had degraded to the control level by 10 months time, but the control level in the zero to six inch layer was 1.62 ppm at this point. No other control data was given, and it is suspected that contamination of the control had

occurred. Tests done in South Dakota indicate no residue one month after spraying, but as much as 0.2 ppm two months after spraying. Such results weaken the reliability of the information provided. In both these latter cases, half-life estimates fell within the range of laboratory results.

Soil persistence of the known major metabolite, hydroxytrichloroisophthalonitrile, has not been studied.

RD initialed by H.A. Jones

cc: FSA - Mr. Alpert (orig. + 1)
DF - Mr. Jones (2)
Miss Malone
RF

BMalone/brm 8/28/67

References

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