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

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OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: FAP#7H5526 - Carbaryl in or on Processed Commodities (Accession Nos. 262810, 264927; RCB No. 1856) Response to Registration Standard

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and

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THRU: Charles L. Trichilo, Chief
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Union Carbide proposes the following food/feed additive tolerances for residues of the insecticide carbaryl, 1-naphthyl methylcarbamate, in or on the following processed commodities.

Processed Commodity	Proposed Food and Feed Additive Tolerance (ppm)	Established Tolerance (ppm) for RAC* (40 CFR 180.169)
Alfalfa, meal	150	100
Apple, pomace, dry	25	10
Citrus, Molasses	15.0	10
Citrus, peel	15.0	
Citrus, oil	450.0	

Field corn, flour	10.0	5
Field corn meal	10.0	
Grain sorghum, bran	25.0	10
Grapes, juice	15.0	10
Grapes, pomace, dry	15.0	
Grapes, pomace, wet	15.0	
Raisin waste	30.0	
Rice, hulls	25.0	5
Snap beans, cannery waste	20.0	10
Soybean, hulls	10.0	5
Sweet corn, cannery waste	35.0	5
Tomato (pomace)	30.0	10

*RAC (raw agricultural commodity).

The Carbaryl Registration Standard has been issued. In compliance with the Registration Standard, various processing studies are submitted. The studies show that, in some cases, residues in the processed commodity are concentrated, and the level of residues is greater than the level in the corresponding raw agricultural commodity (RAC). As a result, food/feed additive tolerances to cover residues in these processed commodities are required. The above tolerances are proposed to satisfy this requirement.

Conclusions

1. A description of the analytical method used to determine carbaryl residues in the processing studies is not submitted. The method is referenced (Carbaryl-HPLC-Afalfa Method, Union Carbide Project Report No. 33769, June 11, 1985), but is not submitted in this petition. The petitioner should submit copies which are not stamped confidential.

2a. Storage stability studies submitted are inadequate and do not provide a valid basis for determining if carbaryl residues in stored commodities are lost or degraded during storage.

Valid storage stability studies must be submitted. The studies should include analyses of fortified samples before storage as well as during and after storage.

2b. The conclusions drawn concerning the RACs and the processing fractions below are contingent upon the results of the requested storage stability studies.

3. No concentration of residues were noted in potato processing fractions (french fries, potato chips, potato flakes). Therefore, no tolerances are needed for the processing fractions.

4a. To avoid illegal residues in alfalfa and alfalfa hay a 3-day PHI or additional residue data to support the 0-day PHI is needed.

4b. Residues may be concentrated in meal obtained from treated alfalfa, but such concentration is considered insignificant (average 1.07X). Therefore, the proposed feed additive tolerance of 150 ppm for alfalfa meal is not needed and should be withdrawn (see Conclusion 4a above.)

5. Residues are not likely to be concentrated in the tomato processing fractions (juice, puree) to levels greater than those in the RAC. Residues are likely to be concentrated in the tomato pomace to levels greater than the level in the RAC. The proposed feed additive tolerance of 30 ppm is adequate to cover residues in tomato pomace.

6. Residues in apple juice are not likely to exceed the level in the RAC. Residues in the dried apple pomace are likely to exceed the level in the RAC. The proposed 25 ppm feed additive tolerance is adequate.

7. Residues are not likely to be concentrated in dried prunes to levels greater than that in the RAC. Therefore, a food additive tolerance is not needed for processed prunes.

8. Residues are concentrated in the sweet sorghum syrup to levels greater than the level in the RAC. A food additive tolerance of 200 ppm is needed to cover residues in the syrup, and a tolerance proposal should be submitted.

9. Residues in the citrus processing fractions (oil, peel, molasses) are likely to exceed the level in the RAC. The proposed food additive tolerance of 15 ppm for citrus molasses

and citrus peel is adequate to cover residues in these items. The proposed tolerance of 450 ppm for citrus oil is also adequate.

10. Residues in the field corn grain processing fractions (grits, refined oil, and starch) are not concentrated to levels greater than the level in the RAC. Therefore, no food additive tolerances are needed for these items. Residues are concentrated in the flour and the meal. The proposed 10 ppm tolerance for flour and meal is adequate.

11a. Residues of carbaryl are concentrated in the grape processing fractions (wet pomace, dry pomace), and the residue levels in the fractions are greater than the level in the RAC. The proposed feed additive tolerance for grape pomace (wet and dry) adequately covers residues expected in the grape pomace (wet and dry). No significant concentration occurs in grape juice; therefore, the proposed food additive tolerance is not needed and should be withdrawn.

[The above Conclusion is contingent upon resolution of the questions raised in Conclusion 11b below.]

11b. The RAC grapes used for raisin production had residues of 10.6 to 18.8 ppm (average, 14.6 ppm) from two applications at the label rate. (The established tolerance for grapes is 10 ppm.) These data indicate that residue levels much greater than the established tolerance could result in grapes from the maximum registered use. As a result, the established tolerance may not be high enough to cover residues in grapes from the registered use. Therefore, the established tolerance and/or the registered use need to be changed. In either case, additional residue data will be needed. The residue data should reflect the maximum registered uses for grapes and representative grape varieties and adequate geographical locations should be included.

11c. Because of the question raised on the level of carbaryl residues in the RAC grapes, RCB is not able to reach valid conclusions on residue levels expected in the byproducts of grapes (juice, pomace, raisins, raisin trash).

12. Residues in peanut processing fractions are not concentrated, and the residue levels in the byproducts (meal, oil, and soapstock) are less than the level in the RAC peanut. Therefore, no food or feed additive tolerance is needed.

13. Residues are concentrated in the rice hulls when the RAC rice is processed. The proposed feed additive tolerance for the hull is adequate to cover residues expected in the hulls.

14. Residues in snap bean cannery waste are at a higher level than those in the RAC snap beans. A feed additive tolerance is needed to cover such residues. The proposed feed additive tolerance (20.0 ppm) is not adequate. A level of 25 ppm is appropriate and should be proposed.

15. Residue levels in the grain sorghum milling fractions (flour and starch) are less than the level in the RAC grain. Therefore, no feed additive tolerance is needed. The proposed tolerance for the grain sorghum bran is not needed and should be withdrawn.

16. Residues in the soybean processing fractions meal, refined oil, and soapstock are adequately covered by the residue level in the soybean. Residues in the hull are higher than those in the RAC soybeans, and a feed additive tolerance is needed to cover such residues. The proposed 10 ppm tolerance is adequate.

17. Residue levels in sugar beet processing fractions (wet and dried pulp, molasses, and refined sugar) are less than in the RAC sugar beet root. Therefore, no food or feed additive tolerances are needed.

18. The proposed feed additive tolerance of 35 ppm is not adequate to cover residues in sweet corn cannery waste. The residue level was estimated using data from a questionable storage stability procedure. A new tolerance proposal may be needed (see Conclusion 2a).

19. A number of the processing fractions are used as livestock feed items and would result in residues in eggs, milk, and meat. RCB defers conclusions on livestock ingestion levels and residues in meat, milk, and eggs until questions on carbaryl residues in the processing fractions have been resolved.

<u>Recommendation</u>

RCB recommends against the proposed food/feed additive tolerances. A favorable recommendation is contingent upon resolution of questions raised concerning storage stability studies (Conclusion 2a) in general, and residue levels in specific items (Conclusions 1, 2b, 4a, 4b, 8, 11a, 11b, 11c, 14, 15, 18, and 19).

The processing studies are discussed and evaluated below under the appropriate Registration Standard headings. Residues in the samples were determined by the high pressure liquid chromatography (HPLC) procedure (Union Carbide Project Report No. 33769, June 11, 1985). The method determines the parent compound carbaryl and its hydrolysis product 1-naphthol.

158.125 Residue Chemistry
171.4 Magnitude of The Residue: Food/Feed Processing Studies (Project No. 801R11)

Potatoes (File No. 34477, Dated February 25, 1986)

Norgold potatoes, field grown, were treated with two applications of formulated carbaryl at a rate of 8.0 lb ai/A (4X label rate) with 8 days between treatments. The potatoes were harvested on the day of the last application (0-day) and analyzed for carbaryl residues by the HPLC procedure (Union Carbide Project Report No. 33769, June 11, 1985). The residues detected (0.03 ppm) were not enough to support a processing study. In order to provide an adequate level of residues, a potato fortification procedure was used.

The potatoes were dipped in a solution of carbaryl and allowed to air-dry. Treated and untreated samples were analyzed and processed by procedures which simulate commercial processing. The various processing fractions were analyzed, and their residue levels were compared to the level in the RAC potato to determine if concentration of residues had occurred.

For the residue method validation, samples of potatoes, french fries, chips, and flakes were fortified with carbaryl at levels of 0.01 to 1.0 ppm. Recoveries were 65 to 154 percent. Untreated (control) samples had less than 0.01 to 0.02 ppm carbaryl equivalent residues.

No concentration of residues were noted in the potato processing fractions (french fries, potato chips, potato flakes). Therefore, no tolerances are needed for the potato processing fractions.

Alfalfa (File No. 34398, Dated February 14, 1986)

Field-grown alfalfa was treated twice with formulated carbaryl at the rate of 3.0 lb ai/A (2X label rate). The interval between applications was 14 days. The mature forage was cut on the day of the last application. The forage samples, treated and untreated, were analyzed for carbaryl residues. Untreated and treated forage samples were converted to alfalfa meal by a process which simulated commercial processing of alfalfa forage to meal. The treated and untreated meal samples were analyzed for carbaryl residues.

Untreated (control) meal samples had 0.02 to 1.20 ppm (average, 0.83 ppm) carbaryl-equivalent residues. Control forage samples had 0.07 to 2.26 ppm (average, 1.16 ppm) carbaryl-equivalent residues. Control samples of alfalfa meal were fortified with carbaryl at levels of 0.03 to 202 ppm. Recoveries were 73 to 117 percent.

Residues in the treated forage (2 samples) were 263 to 484 ppm (average, 379 ppm). The forage yielded meal with residues of 337 to 376 ppm (average, 357 ppm). The carbaryl residues in the meal showed concentration factors of 0.7 to 1.43 (average, 1.07).

Residues can be concentrated in meal obtained from treated alfalfa. Using a concentration factor of 1.5, Union Carbide has proposed a feed additive tolerance of 150 ppm for alfalfa meal. (Alfalfa has an established tolerance of 100 ppm, §180.169.) RCB considers the residue concentration (average, 1.07X) to be insignificant. As a result, no feed additive tolerance is needed. The proposed 150 ppm tolerance for alfalfa meal should be withdrawn.

The residue levels for the RAC forage and hay in this study consist of unexpectedly high residue levels from exaggerated rates (2X proposed). A review of the alfalfa residue data in the Registration Standard show a similar pattern at exaggerated rates (Registration Standard, June 21, 1982, C.L. Trichilo).

The Registration Standard and a followup memorandum (R.W. Storherr, RCB, June 2, 1983) have indicated that in order to avoid residues greater than the established tolerance, a 3-day PHI or additional residue data should be submitted.

RCB reiterates the need for the 3-day PHI or additional residue data to support the 0-day PHI.

Tomatoes (File No. 34397, Dated February 14, 1986)

Field-grown tomatoes were treated twice with formulated carbaryl at 4.0 lb ai/A (2X the label rate) with 14 days between applications. Mature tomato samples (treated and untreated) were harvested on the day of the second application and analyzed for residues of carbaryl. Treated and untreated tomatoes were processed, and the fractions were analyzed for carbaryl residues.

Untreated (control) tomatoes had less than 0.002 ppm carbaryl-equivalent residues. Control tomato samples were fortified with carbaryl at levels of 0.02 to 20.1 ppm. Recoveries were 85 to 113 percent. Control samples of wet pomace had 0.006 to 0.012 ppm carbaryl-equivalent residues. Control wet pomace samples were fortified with carbaryl at levels of 0.02 to 4.02 ppm. Recoveries were 74 to 101 percent. Control samples of dry pomace had 0.029 to 0.044 ppm carbaryl-equivalent residues. Control samples of dry pomace were fortified with carbaryl at levels of 0.06 to 14.21 ppm. Recoveries were 80 to 106 percent. Control samples of tomato puree and tomato juice had less than 0.002 ppm carbaryl equivalent residues. Control samples of puree and juice were fortified with carbaryl at levels of 0.02 to 4.02 ppm. Recoveries were 94 to 109 percent.

Treated tomatoes (RAC) containing residues of 2.73 to 3.12 ppm (average, 2.93 ppm) were processed to juice, wet and dry pomace, and puree. All fractions except the wet pomace had residue levels less than the level in the whole tomato. Residues in the wet pomace were 2.44X the level in the whole tomato.

Carbaryl residues are concentrated only in the wet pomace. Using a concentration factor of 3.0, Union Carbide has proposed a feed additive tolerance of 30 ppm for tomato pomace. (Tomatoes have an established tolerance of 10 ppm, 40 CFR 180.169.) RCB concludes that the proposed tolerance is adequate.

Apples (File No. 34443, dated February 21, 1986)

Apples were treated twice with formulated carbaryl at a rate of 8 lb ai/A (2X label rate). The interval between applications was 14 days. Apples were harvested on the day of the second application. Treated and untreated (control) samples were analyzed for carbaryl residues. Treated and control samples of apples were then processed, and the processing fractions were analyzed for carbaryl residues.

Untreated (control) apples had carbaryl-equivalent residues of 0.030 to 0.034 ppm. Control apples were fortified with carbaryl at levels of 0.08 to 4.04 ppm. Recoveries were 103 to 118 precent. The control processing fractions (juice, wet pomace, and dry pomace) had 0.080 to 0.319 ppm carbaryl-equivalent residues. The fractions were fortified with carbaryl at levels of 0.08 to 20.2 ppm. Recoveries were 87 to 112 percent.

Carbaryl residues in the whole fruit were 6.56 to 7.14 ppm (average, 6.85 ppm). No concentration of residues occurred in the juice. Residues were concentrated in wet pomace (0.69X to 1.34X; average, 1.02) and dried pomace (average, 2.48X). Based on these data, a feed additive tolerance is needed for apple pomace (dried). A concentration factor of 2.5 is dictated by the data. As a result, a feed additive tolerance of 25 ppm (2.5 x 10 ppm apple tolerance) is warranted.

The petitioner's proposed tolerance of 25 ppm for dried apple pomace is adequate.

Prunes (File No. 34438, Dated February 21, 1986

Prunes were treated twice with formulated carbaryl at a rate of 6.0 lb ai/A. The interval between applications was 14 days. The prunes were harvested on the day of the second application and analyzed for carbaryl residues. Prune samples were processed under conditions that simulated commercial processing. The dried prunes were then examined for carbaryl residues.

Untreated (control) fresh prune samples had less than 0.01 ppm to 0.043 ppm carbaryl-equivalent residues. Control samples of dried prunes had 0.023 to 0.024 ppm carbaryl-equivalent residues. Control samples of fresh or dried prunes were fortified with carbaryl at levels of 0.02 to 10.3 ppm. Recoveries were 71 to 88 percent.

The fresh prunes had carbaryl residues of 4.00 to 4.23 ppm (average, 4.11 ppm). The washed fresh prunes had 0.95 to 1.18 ppm (average, 1.06 ppm) carbaryl residues. The dried prunes had 0.60 to 0.64 ppm (average, 0.62 ppm) carbaryl residues.

The carbaryl residue levels in the dried prunes are much less than the levels in the fresh prunes. The data show that carbaryl residues are not concentrated in the dried prunes. Therefore, the residue level in the fresh prunes will adequately cover residues in the processed prunes. As a result, no food additive tolerance is needed for processed prunes.

Sweet Sorghum (File No. 34413, Dated February 13, 1986)

Sweet sorghum was treated twice with formulated carbaryl at the rate of 4.0 lb ai/A (2X the label rate). The interval between applications was 14 days. The sorghum forage was cut the same day as the last application and analyzed for carbaryl residues. The stalks were processed to raw juice and bagasse. The raw juice was further processed to syrup. The bagasse and syrup were analyzed for carbaryl residues.

Untreated (control) samples of stalks, syrup, and bagasse had less than 0.01 ppm carbaryl-equivalent residues. Control samples of stalks, syrup, and bagasse were fortified with carbaryl at levels of 0.02 to 80.1 ppm. Recoveries were 84 to 113 percent.

The treated stalks had carbaryl residues of 0.98 to 1.62 ppm (average, 1.3 ppm). The syrup had residues of 1.96 to 2.23 ppm (average, 2.10 ppm) and the bagasse had residues of 8.91 to 10.99 ppm (average, 9.95 ppm).

The data show that the residue level in the syrup is greater than the level in the stalk. As a result, carbaryl residues are concentrated in syrup obtained from the treated and processed stalks. The range of concentration factors are 1.2X to 2.3X (average, 1.64X). By using the average concentration factor of 1.64X and allowing for some statistical variation, a concentration factor of 2.0 is estimated.

For the concentration of carbaryl residues in the sorghum stalk, the petitioner has averaged the sample levels and calculated an average stalk level of 1.3 ppm. The petitioner states that this level is less than the level expected from an application rate of 4.0 lb ai/A and a 0-day PHI. (No residue data on treated sorghum stalks are provided to support this statement. The data in this study show four stalk values: 0.98, 1.02, 1.49, and 1.62 ppm; average, 1.3 ppm.)

The petitioner uses the residue levels found in the juice and the bagasse and works backward to the stalk to calculate a level of 12 ppm for the stalk. Using this value and the residue levels found in syrup, the petitioner concludes that residues in the stalk are not concentrated in syrup upon processing. As a result, no food additive tolerance is needed for carbaryl residues in sweet sorghum syrup obtained from field-treated sweet sorghum.

RCB contends that residue levels in or on sweet sorghum should be obtained through analyses of field-treated sorghum samples. The calculation of residue levels for the sorghum from the processing fractions (e.g., juice and bagasse) is not acceptable in lieu of actual residue data.

RCB concludes that the sweet sorghum study shows that residues in the stalk are concentrated in the syrup. Since sorghum forage has an established carbaryl tolerance of 100 ppm (40 CFR 180.169), then a food additive tolerance of 200 ppm is needed to cover residues in sorghum syrup.

Citrus (File No. 34913, Dated August 18, 1986)

Grapefruit

Grapefruit were treated with two ground applications of formulated carbaryl at the rate of 20 lb ai/A (maximum label rate). The interval between applications was 14 days. Samples were collected 1 day after the second treatment and analyzed for carbaryl residues. Samples of grapefruit were processed under conditions that simulated commercial conditions, and the processing fractions were analyzed for carbaryl residues. (The citrus processing procedure is included in the report.)

Untreated (control) samples of whole grapefruit and grapefruit processing fractions (juice, peel, pulp, oil, and molasses) had 0.01 to 0.25 ppm carbaryl-equivalent residues. Control samples of grapefruit and its processing fractions were fortified with carbaryl at levels of 0.04 to 44.20 ppm. Overall recoveries were 73 to 117 percent.

Residues in whole, unwashed grapefruit (RAC) were 9.98 to 11.36 ppm (average, 10.7 ppm). (Washed grapefruit had residues of 4.05 to 5.06 ppm, average, 4.55 ppm.) Each of the processing fractions (juice, wet pulp, dried pulp) had lower residue levels than the grapefruit. As a result no concentrations of residues occurred in these fractions, and no food or feed additive tolerances are needed.

The grapefruit peel had residues of 11.96 to 12.16 ppm (average, 12.06 ppm). Residues are concentrated in the peel, and the concentration factor is 1.1X. A food additive tolerance is needed to cover such residues. A level of 15 ppm is estimated by using a 1.5X concentration factor and the 10 ppm citrus tolerance.

The grapefruit peel oil had residues of 205 to 274 ppm; average, 240 ppm (average concentration factor of 22X). Residues are concentrated in the oil and a food additive tolerance is needed. By using a concentration factor of 25X and the established citrus tolerance of 10 ppm (cf. 40 CFR 180.169), a food additive tolerance of 250 ppm is estimated for the oil.

Residues were occasionally concentrated in the molasses: 1.1X, 0.99X, 1.06X, and 0.93X (average of 1.02X). However, the concentration is not significant, and no food additive tolerance is needed.

Oranges

Oranges were treated with two applications of formulated carbaryl at the rate of 20 lb ai/A, and samples were taken 1 day after the second treatment. The whole oranges were analyzed for carbaryl residues. Oranges were processed as above, and the various fractions were analyzed for carbaryl residues.

Untreated (control) samples of whole oranges and the orange processing fractions (juice, wet pulp, dried pulp, oil, and molasses) had < 0.01 to 0.03 ppm carbaryl-equivalent residues. Control samples of oranges and the processing fractions were fortified with carbaryl at levels of 0.044 to 22.10 ppm. Recoveries were 55 to 116 percent.

Residues in whole unwashed oranges (RAC) were 11.4 to 11.6 ppm; average, 11.5 ppm. (Washed oranges had residues of 4.7 to 6.0 ppm; average, 5.3 ppm.) The processing fractions (juice, wet pulp, dried pulp, and molasses) had residue levels less than the level in the whole orange. As a result, no food or feed additive tolerance is needed for the juice, wet and dried pulp, and molasses.

The oil had carbaryl residues of 292 to 302 ppm (average, 297 ppm). The residues are concentrated in the oil. The concentration factors are 25.2X to 26.5X (average, 25.9X). Using a concentration factor of 30X and the established citrus tolerance of 10 ppm, a level of 300 ppm is estimated for the oil of the orange. A food additive tolerance is warranted to cover such residues.

The orange peel had residues of 13.82 to 15.32 ppm (average, 14.6 ppm). The residues are concentrated in the peel, and show concentration factors of 1.2X to 1.4X (average, 1.3X). Using a concentration factor of 1.5X and the citrus tolerance of 10 ppm, a level of 15 ppm is estimated for the peel. Therefore, a food additive tolerance is needed to cover such residues.

Lemons

Lemons were treated with three applications of formulated carbaryl at the rate of 20 lb ai/A, and samples of lemons were collected the day after the last application. The whole lemons were analyzed for carbaryl residues. Lemons were processed, and the processing fractions were analyzed for carbaryl residues.

Untreated (control) samples of lemons and the processing fractions had < 0.01 to 0.40 ppm carbaryl-equivalent residues. Control samples of lemons and its processing fractions (juice, wet pulp, dried pulp, oil, and molasses) were fortified with carbaryl at levels of 0.04 to 61.14 ppm. Recoveries were 66 to 112 percent.

Residues in whole unwashed lemons were 11.50 to 12.43 ppm (average, 11.97 ppm). (Washed lemons have 10.77 to 11.05 ppm carbaryl residues.) The processing fractions (juice, wet pulp, and dried pulp) had residue levels much less than the level in the whole lemon. Therefore, no food or feed additive tolerance is needed to cover residues in the juice, wet pulp, and dried pulp.

The lemon peel had residues of 13.67 to 14.17 ppm (average, 13.9 ppm). Residues are concentrated in the peel, and the concentration factors are 1.1X to 1.2X. By using a concentration factor of 1.5X and the 10 ppm citrus tolerance, a level of 15 ppm is estimated for the peel. A food additive tolerance is needed to cover such residue.

The lemon oil had residues of 498 to 562 ppm (average, 530 ppm) and shows concentrations of residues of 40X to 49X (average, 44.3X) the levels in the whole lemon. Using a concentration factor of 45X and the established 10 ppm tolerance for citrus, a level of 450 ppm can be estimated for the oil. Therefore, a food additive tolerance is needed to cover such residues.

The molasses had residues of 14.11 to 14.89 (average, 14.5 ppm) and showed concentrations of residues of 1.1X to 1.3X (average, 1.2X). Using a concentration factor of 1.5X and the established 10 ppm tolerance for citrus, a level of 15 ppm can be estimated for the molasses. Therefore, a food additive tolerance is warranted to cover such residues.

Food and feed additive tolerances are required for the processed citrus food/feed items (oil, peel, and molasses).

The following food/feed additive tolerances are warranted by the data.

Citrus oil	450 ppm
Citrus peel	15 ppm
Citrus molasses	15 ppm

The tolerances proposed for citrus peel, citrus oil, and citrus molasses are sufficient to cover residue in these items.

Field Corn (File No. 34914, Dated August 28, 1986)

Field corn was treated with two ground applications of formulated carbaryl at the rate of 13 lb ai/A (2X maximum registered rate). The interval between applications was 14 days. Corn samples were collected on the day of the last application. The grain was removed from the cobs, and half was shipped frozen to the Food Protein Center of Texas A&M University where the grain was analyzed and processed by dry milling. The other half was frozen and sent to the Union Carbide Research and Development Center in Research Triangle Park, North Carolina, where the grain was placed in frozen storage. Grain samples were then shipped frozen to the U.S. Department of Agriculture in Peoria, Illinois where the grain was analyzed and processed by wet milling.

The processing fractions were analyzed for carbaryl residues. The wet milling and dry milling processes are summarized in the study.

Untreated (control) samples of corn grain had 0.001 to 0.002 ppm carbaryl-equivalent residues. Control grain samples were fortified at levels of 0.10 to 2.04 ppm. Recoveries were 64 to 105 percent. The control processing fractions (meal, flour, grits, refined oil, crude oil, germ, and starch) were fortified with carbaryl at levels of 0.04 to 1.02 ppm. Recoveries were 79 to 103 percent. The control processing fractions had less than 0.01 to 0.10 ppm carbaryl-equivalent residues.

The treated corn grain had 1.22 to 2.05 ppm (average, 1.48 ppm) carbaryl residues. The fractions from the dry-milling process had the following residue levels: meal 1.69 to 1.79 ppm (average, 1.74 ppm); flour 2.31 to 2.32 ppm; grits 0.33 to 0.34 ppm; crude oil 3.06 to 3.30 ppm (average, 3.18 ppm); refined oil, 0.22 to 0.23 ppm. For the wet-milling process, the processing fractions had the following residue levels: germ, 3.27 to 3.91 ppm (average, 3.59 ppm); starch, less than 0.01 ppm (no detectable residues).

The residue levels in the meal, grits, refined oil, and starch were not concentrated to levels greater than the level in the grain. Therefore, no food additive tolerances are needed for these items.

Residues were concentrated in the meal (1.29X), the flour (1.71X), crude oil (2.36X), and the germ (1.80X). The crude oil is refined before use, and the refined oil does not have residue levels greater than the level in the grain. The germ itself is not a fraction used as food or feed. As a result, neither the germ nor the crude oil warrants a food additive tolerance. The flour and the meal need food additive tolerances. By using a concentration factor of 2 and the established 5 ppm tolerance for corn, a level of 10 ppm is estimated.

Food additive tolerance are needed for flour and meal. The proposed level of 10 ppm is appropriate for both byproducts.

Grapes (File No. 34691, Dated July 9, 1986)

Grapes were treated with two applications of formulated carbaryl at the rate of 4.0 lb ai/A (2X the label rate). The applications were made 14 days apart. The grapes were harvested on the day of the second application. Samples of grapes were analyzed for carbaryl residues and processed. The processing fractions were analyzed for carbaryl residues. A summary of the processing operation is included in the study.

Untreated (control) samples of grapes and the processing fractions (wet pomace, juice, and dry pomace) had < 0.01 to 0.05 ppm carbaryl-equivalent residues. Control samples of grapes and processing fractions were fortified with carbaryl at levels of 0.021 to 10.3 ppm. Recoveries were 60 to 101 percent.

The grapes had residues of 9.06 to 10.23 ppm (average, 9.69 ppm). The processing fractions had the following levels: juice, 9.67 to 12.02 ppm (average, 10.85 ppm); wet pomace, 13.99 to 14.03 ppm (average, 14.01 ppm); dry pomace, 2.13 to 18.01 (average, 11.47 ppm).

Carbaryl residues are concentrated in all fractions, and the residue levels are greater than the level in the grape.

The average concentration factors calculated for the processing fractions are as follows: juice (1.1X); wet pomace (1.4X); dry pomace (1.2X). The concentration in juice is not considered to be significant. Therefore, residues in the juice will be adequately covered by the level in the RAC. As a result, a food additive tolerance for juice is not warranted, and the proposed juice tolerance should be withdrawn.

By using the concentration factor for pomace and allowing for some statistical variation, a factor of 1.5X is estimated. With the established grape tolerance of 10 ppm (§180.169) and the 1.5X concentration factor, a level of 15 ppm is estimated for grape pomace (wet and dry). The proposed tolerance for grape pomace adequately covers residues expected in grape pomace.

Raisins and Raisin Waste (File No. 34793, dated July 9, 1986

Grapes were treated twice at the rate of 2.0 lb ai/A (maximum label rate), and samples were collected on the day of the second application. (There was a 16-day interval between applications.) Samples of grapes were analyzed for carbaryl residues and processed to raisins by a procedure which simulates commercial processing. (A summary of the process is included in the study.) The raisins were analyzed for residues, and the raisin waste (cull raisins and stems) was also analyzed for residues.

Untreated (control) grapes had 0.01 to 0.09 ppm carbaryl-equivalent residues. Control raisins and raisin waste samples had 0.01 to 0.08 ppm carbaryl-equivalent residues. Control samples of grapes, raisins, and raisin waste were fortified with carbaryl at levels of 0.02 to 10.3 ppm. Recoveries were 60 to 99 percent.

Grapes had carbaryl residues of 10.55 to 18.81 ppm (average, 14.63 ppm) due to treatments with the maximum EPA registered application rate. The established tolerance for carbaryl in grapes is 10 ppm (40 CFR 180.169). These data indicate that residues far greater than the established tolerance could result in grapes from the maximum registered use. As a result, the established tolerance is not high enough to cover residues likely to result from the registered uses.

The following discussion is submitted in the study to explain the unusually high residue levels.

"Grape residues were higher than expected, averaging 14.1 ppm. This result is not to be considered typical since fresh grapes for raisin production are not normally taken for other uses. Grapes grown for other purposes are generally much larger. Their smaller surface area-to-mass ratios should result in lower residues. The 1985 carbaryl grape study in New York supports this conclusion [see the foregoing grape processing study, File No. 34591]. Grapes treated with carbaryl at twice the label rate had carbaryl residues of only 9.7 ppm when harvested the same day as the last application." (The grapes actually had residues of 9.06 to 10.23 ppm, and averaged 9.7 ppm.)

RCB recognizes that the level of pesticide residues in or on grapes from foliar applications may be affected by the size of the grape. However, this factor is taken into consideration since the tolerance established for grapes reflects residue data obtained from a variety of treated grapes.

RCB does not agree with the assertion that ". . . fresh grapes for raisin production are not normally taken for other uses." For example, "the Thompson Seedless may be used for fresh fruit consumption, for canning, for wine production, as well as in the production of raisins" (Food and Food Production Encyclopedia, 1982).

RCB concurs that the established tolerance for grapes and/or the registered use may need to be changed in view of the appearance of carbaryl residues at levels much greater than the tolerance.

If the registered use is to be changed to reflect a preharvest interval (i.e., no harvest is to occur within a given number of days after the last application), then appropriate residue data should support the interval (or PHI).

If the tolerance is changed, then additional residue data for grapes will be needed. The data should reflect the maximum registered uses for grapes, and representative grape varieties should be included.

In view of the question raised on the level of residues of carbaryl in the RAC grapes, RCB is not able to reach valid conclusions on residue levels expected in the byproducts of grapes (juice, pomace, raisins, and raisin trash).

<u>Peanuts (File No. 34912, Dated August 18, 1986)</u>

Peanuts were treated twice with formulated carbaryl at the rate of 6 lb ai/A (about 3X label rate). The applications were 13 days apart. Peanuts were harvested on the day of the last application, and the hulls and the nutmeats were analyzed separately for carbaryl residues.

The report states that residues on the nutmeats were "barely detectable." In order to get residue levels high enough to work with in the processing, nutmeat samples were sprayed with formulated carbaryl and analyzed. Samples of the nutmeats were processed, and the processing fractions (oil, meal, and soapstock) were analyzed for carbaryl residues. (A summary of the processing procedure is included in the study.)

Untreated (control) samples of nutmeats, hulls or shells, and the processing fractions had less than 0.01 to 0.03 ppm carbaryl-equivalent residues. Control samples of nutmeats, shells, and the processing fractions were fortified with carbaryl at levels of 0.04 to 10.19 ppm. Recoveries were 75 to 98 percent.

Nutmeats had 0.024 to 0.314 ppm (average, 0.169 ppm) from one application and 6.92 to 8.48 ppm (average, 7.51 ppm) from the two applications (samples from this group were processed). The shells had 1.23 ppm from the single application. The processing fractions had the following residue levels: meal (0.46 to 1.52 ppm, (average, 1.03 ppm); crude oil (7.77 to 13.43 ppm, (average, 10.64 ppm); refined oil (0.02 to 0.22 ppm, (average, 0.12 ppm); soapstock, less than 0.01 ppm.

The data show that residues in the peanut processing fractions are not concentrated, and the residue levels in the byproducts (meal, oil, and soapstock) are less than the level in the RAC peanut (nutmeat). Therefore, no food or feed additive tolerance is required for the peanut byproducts.

Rice (File No. 34693, Dated March 19, 1986)

Rice was treated with two ground applications of formulated carbaryl at the rate of 4.0 lb ai/A (2X registered rate). The applications were 14 days apart. Samples of rice were collected on the day of the last application and analyzed for carbaryl residues. Samples of rice were processed and the processing fractions were analyzed for carbaryl residues. (A summary of the processing procedure is included in the report.)

Untreated (control) samples of rice and its processing fractions (hulls, bran, and polished rice) had less than 0.01 to 0.04 ppm carbaryl-equivalent residues. Control samples of rice and its processing fractions were fortified with carbaryl at levels of 0.02 to 103 ppm. Recoveries were 72 to 97 percent.

Rough rice had carbaryl residues of 126 to 173 ppm (average, 150 ppm). The processing fractions had the following residue levels: hulls (625 to 630 ppm, average, 628 ppm); bran (116 to 168 ppm, average, 142 ppm); polished rice (1.31 to 1.44 ppm, average, 1.4 ppm).

Residues are concentrated in the hulls. No concentration of residues is noted in the polished rice or the bran. The concentration factor is 4.2X for the hulls. Using the established tolerance of 5 ppm for the RAC rice and the average concentration factor, the residue level is estimated to be 21 ppm. The proposed tolerance of 25 ppm is adequate to cover residues in hulls.

The established tolerance for rice is high enough to cover residues in polished rice and bran.

Snap Beans (File No. 34822, Dated June 3, 1986)

Samples of snap beans were obtained from crops grown in California, Washington, New York, Wisconsin, and North Carolina. The crops had been treated with four applications of formulated carbaryl at the registered rate of 2.0 lb ai/A. The interval between applications was 6 to 13 days. The mature bean samples were collected on the day of the last application and placed in frozen storage (-20 °C) for periods of 156 to 199 days. The samples were analyzed for carbaryl residues. The cannery waste from the beans (cull beans, stems) was also analyzed for carbaryl residues.

Untreated (control) samples of snap beans and snap bean cannery waste had less than 0.01 to 0.04 ppm carbaryl-equivalent residues. Control samples of snap beans and cannery waste were fortified with carbaryl at levels of 0.82 to 4.40 ppm. Recoveries were 75 to 105 percent.

The snap beans had 2.53 to 7.46 ppm (average, 4.43 ppm) carbaryl residues. The cannery waste had 4.69 to 18.87 ppm (average, 9.86 ppm) carbaryl residues. Residue levels in the cannery waste were generally higher than the levels in the snap bean. The concentration factor is 2.2X. Using the concentration factor of 2.2X and the established tolerance of 10 ppm for snap beans and allowing for statistical variation, a level of 25 ppm is calculated for the cannery waste.

Residues in the cannery waste are at a higher level than those in the snap beans. Therefore, a feed additive tolerance is needed to cover such residues. A level of 25 ppm is appropriate and should be proposed.

The proposed tolerance (20 ppm) is not adequate for snap bean cannery waste.

Storage Stability

Untreated (control) snap bean samples were fortified with carbaryl at nominal levels of 0.2 ppm and 8.0 ppm (the fortified samples were not analyzed). The fortified samples were placed in frozen storage (-10 °C) for 183 days. Samples from each fortification level were removed and analyzed for carbaryl residues. The reported stated that one control sample was fortified at 2.2 ppm and also analyzed at this time. However, neither the data nor the purpose for this fortified sample is reported. The recoveries at 183 days were expressed as percent of theoretical (this assumed that the initial concentration levels were analyzed and found to be 0.2 ppm and 8.0 ppm). The recoveries were 61.0 and 65.0 percent at the 0.2 ppm level and 86.1 and 86.6 percent at the 8.0 ppm level. These results indicate losses of 35 to 39 percent at the 0.2 ppm level and 13.4 to 13.9 percent at the 8.0 ppm level after storage at -10 °C for 183 days.

The recoveries at 202 days were 53.5 and 65.5 percent at the 0.2 ppm level and 65.8 and 72.0 percent at the 8.0 ppm fortification level. These results indicate losses of 34.5 to 46.5 percent at the 0.2 ppm level and 28.0 to 34.2 percent losses at the 8.0 ppm level.

The storage stability study indicates that carbaryl is considerably degraded during frozen storage at -10 °C in snap beans for 183 to 202 days. However, the results are questionable since no analyses were performed at the time of sample fortifications. Such analyses would have provided more realistic concentration levels for reference purposes. In view of the foregoing, RCB concludes that the carbaryl storage stability study with snap beans is inadequate. Therefore, valid conclusions on the stability of carbaryl during frozen storage in snap beans are not possible. (See the discussion on Storage Stability which follows below.)

Grain Sorghum (File No. No. 34847, Dated July 31, 1986)

Grain sorghum was treated with two aerial applications of formulated carbaryl at the rate of 4.0 lb ai/A (2X maximum registered rate). The applications were 11 days apart. Samples of grain were collected at 3 days after the last application (the registered use includes a 21-day PHI) and held in frozen storage (-20 °C) for approximately 332 days. The samples were removed from storage and analyzed for carbaryl residues. Grain samples were processed, and the processing fractions (bran, shorts, flour, decorticated grain, and starch) were analyzed for carbaryl residues. Two dry-milling processes and one wet-milling process are summarized and reported to be similar to commercial milling processes.

Untreated (control) samples of sorghum grain and the processing or grain milling fractions had less than 0.01 to 0.05 ppm carbaryl-equivalent residues. Control samples of grain and the processing fractions were fortified with carbaryl at levels of 0.08 to 509.5 ppm. Recoveries were 74 to 94 percent.

The sorghum grain had 1.02 to 1.20 ppm (average, 1.11 ppm) carbaryl residues. The processing fractions (starch, flour, decorticated grain, and shorts) had residue levels of less than 0.02 to 0.74 ppm, which are less than the level in the RAC grain sorghum.

Residues in the bran were 2.30 to 2.95 ppm (average, 2.51 ppm). These levels are greater than the level in the RAC. However, the milling fractions of grain sorghum which are considered for feed additive tolerances are flour and starch. Neither of these items has residue levels greater than the level in the grain. Therefore, no feed additive tolerance is needed.

The tolerance proposed for the bran is not required and should be withdrawn (see memorandum of March 21, 1985, C.L. Trichilo, "Definition of Milled Products for Various Grains.")

<u>Soybeans (File No. 34692, Dated April 17, 1986)</u>

Soybeans were treated with two ground applications of formulated carbaryl at the rate of 5.0 lb ai/A (at least 3.3X registered rate). Soybean samples were collected on the day of the last application and stored at -20 °C. The time between applications was 13 days.

The soybeans were analyzed for carbaryl residues, and samples were processed. The processing operation is summarized in the study. The processing fractions (hulls, meal, crude oil, refined oil, and soapstock) were analyzed for carbaryl residues. The storage intervals were 114 to 121 days.

Untreated (control) samples of soybeans and the processing fractions had less than 0.01 ppm carbaryl-equivalent residues. Control samples of soybeans and the processing fractions were fortified with carbaryl at levels of 0.02 to 8.24 ppm. Recoveries were 56 to 119 percent.

The whole soybeans had an average of 1.5 ppm carbaryl residues. The meal, refined oil, and soapstock had carbaryl residues of less than 0.02 to 0.04 ppm which are considerably less than the level in the soybean. The crude oil had levels of 1.27 to 1.52 ppm (average, 1.40 ppm); however, the crude oil is processed to refined oil. The refined oil had < 0.02 ppm carbaryl residues. Residues in the meal, refined oil, and soapstock would be adequately covered by the residue level in the soybean. Therefore, no food or feed additive tolerance is needed for these processing fractions.

The hulls had carbaryl residues of 1.95 to 2.08 ppm (average, 2.02 ppm). These levels are higher than those in the soybean, and a feed additive tolerance is needed to cover these residues (soybeans have an established tolerance of 5 ppm). Residues in the hulls have concentration factors of 1.3 to 1.4X. The petitioner's proposed tolerance of 10 ppm for the hulls is adequate and will cover residues expected in the hulls.

Sugar Beets (File No. 34818, Dated July 16, 1986)

Sugar beets were treated twice with formulated carbaryl at the rate of 6.0 lb ai/A (4X registered rate) by Mono-Hy Sugar Beet Seed Inc., in Colorado. The interval between applications was 14 days. Sugar beet samples were collected on the day of the second application. Part of the samples was shipped frozen to Union Carbide in North Carolina, and the beets were analyzed 9 days after sampling.

The remaining portion of the sugar beet samples (treated and untreated) was stored at 40 °F for 107 days. The samples were processed, and the processing fractions (wet pulp, dried pulp, refined sugar, and molasses) and whole roots were shipped frozen to Union Carbide in North Carolina. The whole roots and the processing fractions were analyzed for carbaryl residues. (A summary of the processing operation is included in the study.)

Untreated (control) sugar beet roots and the processing fractions had < 0.02 to 0.07 ppm carbaryl-equivalent residues. Control samples of roots and the processing fractions were fortified with carbaryl at levels of 0.02 to 0.88 ppm. Recoveries were 55 to 94 percent.

The sugar beet roots that were not processed and initially shipped to Union Carbide were analyzed 9 days after sampling. The roots had carbaryl residues of 0.23 to 0.30 ppm (average, 0.27 ppm).

The second portion of the sugar beet roots (stored at 40 °F for 107 days) had carbaryl residues of 0.15 to 0.31 ppm (average, 0.23 ppm). The processing fractions had the following residue levels: wet pulp, < 0.02 ppm; dry pulp, < 0.02 ppm; molasses, < 0.02 ppm; refined sugar, < 0.02 ppm. The data show that residues in the processing fractions are much less than in the whole sugar beet root. Therefore, no food or feed additive tolerance is needed.

Sweet Corn (File No. 34830, Dated July 28, 1986).

Sweet corn was grown in plots in Iowa, California, Delaware, and Wisconsin. The crops were treated with four ground applications of formulated carbaryl at the rate of 6.5 lb ai/A (maximum registered rate) with intervals of 6 to 7 days between applications. Corn samples were collected on the day of the last application, and the RAC (corn plus cobs with husks removed) was analyzed for carbaryl residues. Samples of cannery waste (cobs and husks) were also analyzed for carbaryl residues.

Untreated (control) samples of sweet corn and cannery waste had 0.02 to 0.14 ppm carbaryl-equivalent residues. Control samples of sweet corn and cannery waste were fortified with carbaryl at levels of 0.09 to 44.2 ppm. Recoveries were 80 to 93 percent.

Sweet corn had 0.11 to 1.48 ppm (average, 0.53 ppm) carbaryl residues. The sweet corn cannery waste had residues of 16.53 to 21.36 ppm (average, 18.62 ppm).

The samples were held in frozen storage at -20 °C for periods of 214 to 329 days prior to analyses. In order to assess the effect of the frozen storage upon carbaryl residues, a storage stability study was performed. Control chopped sweet corn (ear with husk) samples were fortified with carbaryl at 0.20 ppm and 8.01 ppm. No analyses were performed on these samples prior to storage. The samples were placed in storage at -10 °C for 251 days. The samples were then removed from storage and analyzed. The recovered carbaryl was expressed as a percent of the "initial" (concentration). However, since no analyses were performed before storage, the "initial" was assumed to be 100 percent, or 0.20 ppm and 8.01 ppm. The recovery at the 0.20 ppm level was 55 to 68 percent and 66 to 80 percent at the 8.01 ppm level. These data suggest losses of 32 to 45 percent at the 0.20 ppm level and 20 to 34 percent at the 8.01 ppm level.

Because of the difficulty in achieving a uniform mixture in the fortified sample, the initial concentration before storage was probably less than 0.20 ppm and 8.01 ppm. As a result, the losses, if any, during storage would be much less than those indicated above.

The petitioner contends that "available data from other substrates indicate that carbaryl is generally stable under the conditions of storage used in this study." The petitioner concludes that "the inadvertent omission of analysis of 'zero time' samples is the most likely cause of the apparent loss of carbaryl."

In order to compensate for the apparent loss of residues, the petitioner has proposed a feed additive tolerance of 35 ppm for residues of carbaryl in sweet corn cannery waste.

RCB does not agree that the proposed feed additive tolerance of 35 ppm is adequate to cover residues of carbaryl in sweet corn cannery waste. Correction factors based on questionable data are not an adequate basis for establishing a tolerance. The tolerance should be supported by residue data that are obtained by valid procedures. The validity of the storage stability study with sweet corn cannery waste is questionable, and the tolerance proposal is therefore inadequate.

Storage Stability

Storage stability studies are submitted with the snap bean and the sweet corn processing studies. The purpose of these studies is to show if carbaryl residues in the commodities are degraded during storage and to what extent. The commodities were stored for periods of 183 to 251 days at a temperature of -10 °C. At the end of the storage, the commodities were removed and analyzed for carbaryl residues.

The snap beans showed losses of 35 to 47 percent at the 0.2 ppm level and 28 to 34 percent at the 8.0 ppm level. The sweet corn showed losses of 32 to 45 percent at the 0.2 ppm level and 20 to 34 percent at the 8.0 ppm level.

The studies indicate that carbaryl is considerably degraded in both substrates. However, the results are questionable. No samples were analyzed before storage. The levels in the samples were considered as 100 percent of the fortification levels, and recovery calculations assumed this to be the case. Nevertheless, because of the difficulty in achieving a uniform residue distribution in the fortified samples, the initial concentration before storage was probably less than the nominal levels of 0.2 ppm and 8.0 ppm. Therefore, the losses, if any, during frozen storage would be much less than those indicated above.

The petitioner's rationale is similar to the above discussion. The petitioner contends that "available data from other substrates indicate that carbaryl is generally stable under the conditions of storage used in this study," and concludes that "the inadvertent omission of analysis of 'zero time' samples is the most likely cause of the apparent loss of carbarvl."

In view of the foregoing discussion, RCB concludes that the storage stability studies are inadequate and do not provide a valid basis for determining if carbarvl residues are degraded during storage.

The commodities involved in the processing studies had been stored prior to analyses for periods as long as 329 days and at temperatures down to -20 °C. The residue levels found in these commodities are questionable due to the inadequacies in the storage stability studies. In order to adequately assess the distribution of carbaryl residues in the processing studies, valid storage stability studies must be submitted. The studies should include analyses of fortified samples before storage as well as during and after storage.

Meat, Milk, Poultry, and Eggs

Some of the processing fractions are used as livestock feed items. Carbaryl residues in these feed items would contribute to the residue levels in eggs, milk, and meat of livestock, and this contribution has to be considered. However, the above discussions have raised questions on the residue levels in the processing fractions. Since valid residue levels in the feed items are not known, it is not possible to determine the level of residues likely to be ingested by livestock and deposited in eggs, milk, and meat as warranted under §180.6. Therefore, RCB defers conclusions on livestock ingestion levels and residues in eggs, milk, and meat until questions on carbaryl residues in the processing fractions have been resolved.

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