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Task 4. Residue Chemistry.
Registration Standard Update

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PCNB

REGISTRATION STANDARD UPDATE

RESIDUE CHEMISTRY

Task - 4

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PCNB

REGISTRATION STANDARD UPDATE

RESIDUE CHEMISTRY

Task - 4

INTRODUCTION

The updated Index to Pesticide Chemicals dated 3/89 lists registered uses on beans (dry, green, kidney, lima, and snap), broccoli, Brussels sprouts, cabbage, cauliflower, cotton, garlic, peppers, and tomatoes. PCNB is registered for seed treatments on barley, beans, corn, cottonseed, garlic seed cloves, oats, peanuts, peas, potato seed pieces, rice, safflower, sorghum, soybeans, sugar beets, and wheat. PCNB formulations registered for food and feed uses are the 10, 20, 25, 30, and 40% dust (D), the 6.5, 10, and 30% granular (G), the 14, 30, 35, and 75% wettable powder (WP), 23.2, 23.4, 23.8, 23.9, and 24% emulsifiable concentrate (EC), 17% flowable concentrate (FlC), and 10, 17.68, 20, 22.6, 23.1, 23.2, 23.7, 24, and 25% ready-to-use (RTU) formulations.

The PCNB Guidance Document dated 1/87 identifies outstanding data requirements for plant and animal metabolism, analytical methods, storage stability data, residue data on potatoes, garlic, broccoli, Brussels sprouts, cabbage, cauliflower, beans, peppers, tomatoes, bananas including plantains, cottonseed, and peanuts, and data concerning seed treatments. It was determined that a tolerance proposal and supporting residue data are needed for soybean commodities grown from treated seed.

The PCNB Guidance Document dated 1/87 concluded that the available data are insufficient to assess the maximum expected residues of PCNB in or on lettuce, strawberries, alfalfa, and clover resulting from use under intrastate registrations and required tolerance proposals and supporting residue data, or, alternatively, cancellations of the registrations.

In response to the Guidance Document, data have been submitted pertaining to storage stability (MRIDs 40921600 and 40921601), potatoes (MRID 40816701), processed potatoes (MRIDs 40070401, 40088401, and 40457401), broccoli (MRID 40816702), cabbage (MRID 40816703), beans (MRID 40816704), peppers (MRID 40816705), peanuts (MRID 41002507), and seed treatment (MRID 40862901), which have been reviewed or are currently under review by the Agency. Data concerning plant metabolism (MRIDs 41341201, 41341202, and 41341203), animal metabolism (MRIDs 41303301, 41341205, 41303101 and 41341204), and cottonseed (MRID 41341206) are reviewed in this update for their adequacy in fulfilling the outstanding data requirements.

A tolerance of 0.1 ppm has been established for residues of PCNB per se in or on cottonseed (40 CFR 180.291[a]), and tolerances with regional registration of 0.2 ppm have been established for the combined residues of PCNB and its metabolites pentachloroaniline (PCA) and methyl pentachlorophenyl sulfide (MPCPS) in or on collards, kale, and mustard greens (40 CFR 180.291[b]). Interim tolerances of 0.1 ppm are in effect for residues of PCNB in or on bananas, beans, broccoli, Brussels sprouts, cabbage cauliflower, garlic, peppers, potatoes and tomatoes; an interim tolerance of 1 ppm is in effect for peanuts (40 CFR 180.319). Tolerances limiting the amount of the impurity hexachlorobenzene (HCB) have been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on numerous plant and animal commodities (PP#1F1083, Amendment of 1/83).

SUMMARY

The following residue chemistry data are outstanding:

- Data on plant and animal metabolism, residue analytical methods, and storage stability.
- Tolerances must be proposed and supporting residue data submitted for alfalfa, clover, and lettuce for which uses are permitted under intrastate registrations, and corn, soybeans, peas, sugar beets, and wheat for which seed treatments are registered, and bean vines and hay.
- Residue data on potatoes, broccoli, cabbage, cauliflower, Brussels sprouts, beans, peppers, tomatoes, bananas, cottonseed, peanuts, and peanut hulls.
- Processing studies on potatoes, cottonseed, peanuts, corn, and soybeans.

QUALITATIVE NATURE OF THE RESIDUE IN PLANTS

Conclusions:

The qualitative nature of the residue in plants is not adequately understood. The PCNB Guidance Document (1/87) requires data depicting the metabolism of PCNB in peanuts, cabbage, and potatoes. Uniroyal Chemical Co., in its 90-day response to the Guidance Document, requested a time extension for submitting the required plant metabolism data and indicated their intention to submit the data in 7/88; DEB had no objection to the registrant's proposal (DEB No. 2781; 11/16/87). In a second request for a time extension, the registrant stated that the data would be submitted on 12/31/89; DEB recommended in favor of the request

(K. Dockter, DEB No. 4857; 2/21/89). Uniroyal has submitted progress reports for metabolism studies on potatoes, peanuts, and cabbage (1989; MRIDs 41341201, -02, and -03) and has stated their intention to complete the studies in 3/90. According to these reports, data from combustion/radioassay of [¹⁴C]PCNB-treated plants indicate that total radioactive residues were 2.6 ppm in or on potato tubers, 7.5 ppm in or on potato vines, 1520.8 ppm in peanut roots, 42.3 ppm in or on peanut vines, 128.3 ppm in or on peanut shells, and 5.16 ppm in or on peanut nutmeats; data on total radioactivity in or on cabbage were not provided.

Data depicting the uptake and translocation of [¹⁴C]PCNB following registered seed treatments were submitted and are addressed in this review in the section "Crops with Seed Treatments Only." The following additional data are required:

- Data depicting the uptake, distribution, and metabolism of ring-labeled [¹⁴C]PCNB in three dissimilar crops (peanuts, potatoes, and cabbage). A completely characterized test substance representative of technical PCNB used in commercial formulations (including impurities) must be applied under conditions representing normal cropping practices and at rates high enough to permit characterization of ¹⁴C-residues. The identities and quantities of residues in mature plant parts must be determined in order to elucidate terminal residues. Confirmation of the identities of residues using a suitable method such as mass spectrometry (MS) or high-performance liquid chromatography (HPLC) is also required. Representative samples from these studies must also be analyzed by the residue analytical methods developed for data collection and tolerance enforcement to ascertain that the methods are capable of adequately recovering and quantifying all metabolites of concern.

References (used):

MRIDs: 41341201. 41341202. 41341203.

Discussion of the data:

N/A.

QUALITATIVE NATURE OF THE RESIDUE IN ANIMALS

Conclusions:

The qualitative nature of the residue in animals is not adequately understood. The PCNB Guidance Document (1/87) requires data depicting the metabolism of PCNB in ruminants and

poultry. Uniroyal Chemical Co., in its 90-day response to the Guidance Document, requested a time extension for submitting the required animal metabolism data and indicated their intention to submit the data in 7/88; DEB had no objection to the registrant's proposal (DEB No. 2781; 11/16/87). In a second request for a time extension, the registrant stated that the data would be submitted on 12/31/89; DEB recommended in favor of the request (K. Dockter; DEB No. 4857; 2/21/89). Uniroyal has submitted two volumes of data (1989; MRIDs 41303301 and 41341205) pertaining to the metabolism of PCNB in lactating goats. These data do not fulfill the requirement for metabolism data on ruminants because discrepancies exist between the descriptions of the in-life portion of the study and tissue-combustion data (MRID 41303301) and data in the submission containing metabolite characterization data (MRID 41341205). Furthermore, the registrant did not provide adequate data to support the quantifications of metabolites reported. The registrant submitted data on poultry metabolism (1988, 41303101; 1989, 41341204) which were incomplete; the registrant stated their intent to complete the study in 3/90.

The following additional data are required:

- Data depicting the metabolism of ring-labeled [¹⁴C]PCNB in ruminants and poultry. For the poultry study in progress (MRID 41341204), the dietary feeding level, and conditions under which tissue hydrolysis were conducted must be reported. The distribution and identities of residues must be determined in eggs, muscle, liver, fat, and skin. For the submitted study on goats, the registrant must explain the discrepancies in the two submissions and present evidence that the data in MRID 41341205 reflect analysis of tissues from the same animal described in MRID 41303301. In addition, the registrant must report the fraction of the TRR partitioned into each solvent used in the extraction and into bound residue for milk, fat, and liver (and muscle, if warranted by the TRR in muscle), and for each extract that was analyzed by HPLC, the registrant must provide the amount of radioactivity applied in each HPLC analysis and the amount of radioactivity detected in the identified radioactive zones. Data depicting the nature of the residues in swine may also be required if studies with ruminants reveal that the metabolism of PCNB in these animals differs from that in rats. Representative samples from these studies must also be analyzed by the residue analytical methods developed for data collection and tolerance enforcement to ascertain that the methods are capable of adequately recovering and quantifying all metabolites of concern.

References (used):

MRIDs: 41303301. 41341205. 41341204. 41303101.

Discussion of the data:

Goats. Amvac Chemical Corporation (1989; MRID 41303301) submitted data pertaining to the metabolism of PCNB in ruminants. Two lactating goats were dosed orally with uniformly ring-labeled [¹⁴C]PCNB (radiochemical purity: 96.6%) administered in capsules for 5 consecutive days. One goat was dosed at a level of 1.36 g/goat/day (25 mg/kg of body weight, equivalent to 714 ppm in the diet, specific activity of 1,556 dpm/μg) and the other goat was dosed at a level of 2.08 g/goat/day (50 mg/kg of body weight, equivalent to 947 ppm in the diet, specific activity of 1,012 dpm/μg). A third goat served as a control and was administered empty capsules. Milk, urine and feces samples were collected twice daily and were stored frozen pending analysis. The goats were sacrificed 6 hours after the last dose, and samples of blood, muscle, liver, kidneys, fat (renal and omental), bile, urine, and gastrointestinal (GI) tract were collected. Blood was refrigerated until analysis was complete, all other samples were stored frozen pending analysis.

Total Radioactive Residues (TRR)

Tissue samples, feces, and blood were combusted and analyzed for TRR by liquid scintillation spectrometry (LSS); samples of bile, milk, urine, GI tract contents, and wash and pan rinses were analyzed directly by LSS. Recovery of the total dose and distribution of total radioactive residue are given below in Tables 1 and 2.

Table 1. Percent recovery of total dose in matrices of lactating goats administered [¹⁴C]PCNB.

| Matrix | Percent recovery at dietary level of | |
|---------------------|--------------------------------------|---------|
| | 714 ppm | 947 ppm |
| Liver | 0.18 | 0.12 |
| Kidney | 0.02 | 0.02 |
| Muscle | <0.01 | <0.01 |
| Fat (omental) | 0.13 | 0.02 |
| Fat (renal) | 0.12 | <0.01 |
| Milk | 0.34 | 0.21 |
| Blood | <0.01 | <0.01 |
| Bile | 0.03 | 0.02 |
| GI tract + contents | 16.60 | 15.16 |
| Urine (cumulative) | 30.51 | 20.00 |
| Urine (bladder) | 0.13 | ND |
| Feces (cumulative) | 35.90 | 41.02 |
| Pan rinse | 0.20 | 0.44 |
| Total | 84.16 | 77.01 |

Table 2. Total radioactive residues (TRR) in milk and tissues of lactating goats administered [¹⁴C]PCNB.

| Matrix | TRR (ppm as PCNB equivalents) at | |
|---------------|----------------------------------|--------------------|
| | 714 ppm | 947 ppm |
| Liver | 13.371 | 13.353 |
| Kidney | 10.295 | 11.204 |
| Muscle | 0.541 | 0.483 |
| Fat (omental) | 15.162 | 12.792 |
| Fat (renal) | 15.615 | 9.430 |
| Milk | 2.866 ^a | 1.759 ^b |

^a Average obtained by the reviewer from reported TRR values of 0.347-5.981 ppm for days 1-5 of treatment.

^b Average obtained by the reviewer from reported values of 0.223-3.553 ppm for days 1-5 of treatment.

Extraction

Data on the extraction and characterization of PCNB metabolites in goats have been submitted by Uniroyal Chemical Company (1989, MRID 41341205). The Uniroyal data are reviewed below under the assumption that the metabolite characterization performed by

Uniroyal was done in matrices from the goat dosed at a dietary level of 947 ppm and described above.

It is noted, however, that although the Uniroyal study was performed with goats dosed orally with [¹⁴C]PCNB (at 50 mg/kg of body weight) for 5 consecutive days (as reported by Amvac), there is no assurance that both the Uniroyal study (MRID 41341205) and the above Amvac study (MRID 41303301) refer to the same animals. In particular, (i) there is no explicit statement indicating that both studies refer to the same animal; (ii) the only TRR value reported in the Uniroyal study, that for kidney, is 49.1 ppm, whereas the TRR for kidney reported in the Amvac study is 11.2 ppm; (iii) the Uniroyal study reported that liver, kidney, and fat were the only tissues that showed radioactive levels above background, whereas the Amvac study (Table 2) reported a muscle TRR of 0.483 ppm; and (iv) the specific activity of the [¹⁴C]PCNB used in the Uniroyal study was reported as 1,040 dpm/μg, whereas that used in the Amvac study was 1,012 dpm/μg.

Urine samples were passed through a Baker solid phase extraction (SPE) C-18 cartridge, washed with water, eluted from the cartridge with methanol and then analyzed by HPLC.

Milk samples were extracted twice with ethyl acetate. The ethyl acetate extracts were concentrated and partitioned with hexane and acetonitrile (ACN). The hexane and ACN fractions were concentrated to near dryness, and the residues from each fraction were dissolved in methanol and analyzed by HPLC. No other details were given.

Subsamples of liver and kidney were extracted according to the Bligh-Dyer method; however, no details were given other than the indication that methanol:chloroform (2:1, v/v) was used in the extraction. The extracts were passed through C-18 SPE cartridges, washed with water, eluted from the cartridge with methanol, and then analyzed by HPLC. Suspended liver and tissue samples were subjected to proteolysis (P5147 protease) for 18 hours at 37 C to release bound residues. The solubilized material was passed through a C-18 SPE cartridge, washed with water, eluted from the cartridge with methanol, and then analyzed by HPLC.

Fat subsamples were extracted with chloroform. The chloroform was evaporated and the samples were partitioned with hexane and ACN. The ACN fraction was concentrated and analyzed by HPLC. No other details were given.

The distribution of total radioactivity in extracts of liver and kidney is summarized in Table 3. Only 44% of the TRR in liver was extractable into organic solvents. The fate of the remaining 56% of the liver TRR was unaccounted for; an unspecified amount of the unextractable residue became solubilized by protease

treatment. Up to 74% of the TRR of kidney was extractable into organic solvents, the remaining 26% was bound residue. Protease treatment of the bound residue in kidney released up to an additional 19% of the TRR.

Little or no data were available concerning the distribution of radioactivity in extracts of milk and fat. In milk, it was reported that 87% of the radioactivity was extractable into organic solvents, the remaining 13% was unaccounted for. It was not explained how the radioactivity partitioned into the various organic solvents or what fraction of the radioactivity was lost or remained as bound residue.

In fat, it was reported that 84% of the radioactivity partitioned into ACN; the remaining 16% was unaccounted for. The type of fat, omental or renal, was not specified. In addition, it was not explained how the radioactivity partitioned into the various organic solvents or what fraction of the radioactivity was lost or remained as bound residue.

Table 3. Distribution of total radioactivity in extracts of liver and kidney from a lactating goat administered [¹⁴C]PCNB orally.

| Fraction | Percent of total radioactivity in | |
|--------------------------|-----------------------------------|----------------|
| | Liver | Kidney |
| Non-bound | | |
| chloroform | 24 | 46 |
| methanol/water | 20 | 28 |
| Total non-bound | 44 | 74 |
| Bound | | |
| Released by protease | - ^a | 19 |
| Not released by protease | - ^b | 7 ^c |
| Total bound | 56 | 26 |
| Total | 100 | 101 |

^a Unspecified. The author indicated only that the residue bound to the pellet after extraction became water soluble after protease treatment.

^b Unspecified.

^c Calculated by the reviewer, by subtracting reported value for protease-released material from reported total bound.

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Characterization of Residues

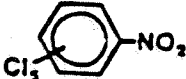
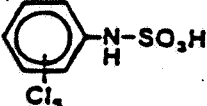
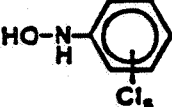
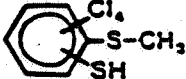

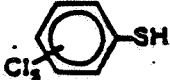
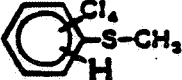
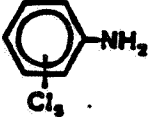
Soluble residues in urine and in extracts of liver, kidney, milk, omental fat, and renal fat were analyzed by high performance liquid chromatography (HPLC). Polar metabolites in liver and kidney were hydrolyzed with 1 M anhydrous methanol/hydrochloric acid at 60 C for 10 minutes prior to analysis by HPLC. A urinary metabolite thought to be metabolite II was hydrolyzed by heating at 80 C in 0.1 N sodium hydroxide for 2 hours. HPLC analysis was conducted using a 20-100% solution of methanol. Metabolite structures were verified by mass spectral analysis. The molecular structures of PCNB and metabolites identified in this study are depicted in Table 4.

The reported results of residue analysis in goat matrices are summarized in Table 5. It is noted that, although [¹⁴C]radioactivity was monitored during analysis and sample radiochromatograms were included in the submission, no experimental data (such as area percent under the radioactivity trace or recoveries of radioactivity after HPLC) were available for review. This omission, coupled with the incomplete data on solvent partition, made it impossible to verify the values for distribution of PCNB metabolites reported in Table 5 and to estimate independently the fractions of TRR lost or unaccounted for.

In particular, as shown in Table 5, the registrant reported that 100% of the TRR in fat was accounted for as metabolite VIII, yet only 84% was accounted for as having been partitioned into ACN and the remaining 16% was unaccounted for. Likewise, the registrant reported that 96% of the TRR in milk was metabolite VIII, yet only 87% of the radioactivity was reported as being extractable into organic solvents and the remaining 13% was unaccounted for. In addition, Table 4 indicates that 101.3% of the TRR in liver was characterized. It is not clear, however, what percentage of the liver TRR was lost, since only 44% (Table 3) was soluble and analysis of the remainder was not reported. In kidney, although 93.2% of the TRR is reported as having been characterized, it is not clear whether the remaining 6.8% was lost or represents uncharacterized bound radioactivity (reported in Table 3 as 7% of the TRR).

No data on characterization of the TRR of muscle was available for review in the Uniroyal submission; no explanation was given for this omission. However, the registrant noted that liver, kidney, and fat were the only tissues that showed radioactivity above background. We note however, that the above Amvac metabolism study (MRID 41303301) reported a TRR of 0.483 ppm in muscle from the high-dose goat (Table 2). Thus, Uniroyal should indicate if the goat used in the Uniroyal study is the same animal that was dosed with 50 mg/kg in the Amvac study.

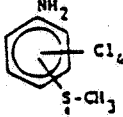
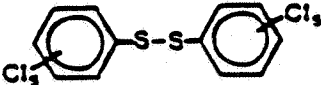
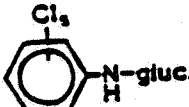
Table 4. PCNB and its metabolites.

| Code | Chemical name Structure | Substrate | MRID Common name |
|-------|---|--|--|
| I. | Pentachloronitrobenzene  | none ^a | 41341205 PCNB |
| II. | Pentachloroaniline sulfamate  | goat urine | 41341205 Metabolite II |
| III. | N-Hydroxypentachloroaniline  | goat liver goat urine | 41341205 41341205 Metabolite III |
| IV. | Tetrachloro(methylthio)thiophenol  | goat liver goat kidney goat urine | 41341205 41341205 41341205 Metabolite IV |
| V. | Pentachloroaniline mercapturic acid conjugate (putative)  | goat urine | 41341205 Metabolite V |
| VI. | Pentachlorothiophenol  | goat kidney | 41341205 Metabolite VI |
| VII. | Tetrachlorothioanisole  | goat liver goat kidney | 41341205 41341205 Metabolite VII |
| VIII. | Pentachloroaniline  | goat liver goat kidney goat omental fat goat renal fat goat milk | 41341205 41341205 41341205 41341205 41341205 PCA, Metabolite VIII |

(Continued)

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Table 4. PCNB and its metabolites (continued).

| Code | Chemical name Structure | Substrate | MRID Common name |
|------|--|---------------------------|---------------------------------------|
| IX. | Tetrachloroaniline methylsulfoxide  | goat kidney | 41341205 Metabolite IX |
| X. | Pentachlorothiophenol dimer  | goat liver | 41341205 Metabolite X |
| XI. | N-Glucuronide of pentachloroaniline  | goat liver goat kidney | 41341205 41341205 Metabolite XI |

^a No unmetabolized PCNB was observed in milk, tissues or urine.

In summary, the metabolites of PCNB in goats are incompletely characterized. Due to incomplete data reporting, levels for metabolites in goat matrices are uncertain. In addition, it is not clear from the available data whether metabolites in muscle should be characterized.

Additional data are required, including: (i) a clarification of whether the goat used in the Uniroyal metabolite characterization study (MRID 41341205) is the same goat dosed at 50 mg/kg of body weight in the Amvac metabolism study (MRID 41303301). If the two goats are different, Uniroyal should submit dosing procedures and other pertinent details of the in-life phase of the study in addition to quantification of the TRR in the various goat matrices. If the two goats are the same, the metabolites in muscle should be characterized; the fraction of the TRR partitioned into each solvent used in the extraction and into bound residue for milk, fat, and liver (and muscle, if warranted by the TRR in muscle); and for each extract that was analyzed by HPLC, provide the amount of radioactivity applied in each HPLC analysis and the amount of radioactivity detected in the identified radioactive zones.

Table 5. Percent distribution of ¹⁴C-residues in matrices of a goat administered [¹⁴C]PCNB orally.

| Metabolite ^a | Percent distribution of goat metabolites in | | | | | |
|-------------------------|---|--------|-------------|-----------|------|-------|
| | Liver | Kidney | Omental Fat | Renal Fat | Milk | Urine |
| II | - ^b | - | - | - | - | 85.0 |
| III | 4.7 | - | - | - | - | 5.0 |
| IV | 1.0 | 4.5 | - | - | - | 6.0 |
| V | - | - | - | - | - | 4.0 |
| VI | 2.9 | 3.3 | - | - | - | - |
| VII | - | 2.1 | - | - | - | - |
| VIII | 17.0 | 26.0 | 100.0 | 100.0 | 96.0 | - |
| IX | - | 2.3 | - | - | - | - |
| X | 2.7 | - | - | - | - | - |
| XI | 73.0 | 55.0 | - | - | - | - |
| Total: | 101.3 | 93.2 | 100.0 | 100.0 | 96.0 | 100.0 |

^a Metabolite codes and the corresponding chemical names and molecular structures are depicted in Table 4.

^b - = Not reported.

Poultry. Uniroyal Chemical Co. (1989; MRID 41341204) submitted preliminary data concerning the metabolism of PCNB in chickens. Laying hens were administered [¹⁴C]PCNB in the diet for 6 days at

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15, 37.5, or 75 mg/day (whether per bird or per kg body weight could not be determined; dietary intake in terms of ppm of residues in the feed was not reported). Eggs and excreta were collected daily and the birds were sacrificed ca. 6 hours after the final dose. Total radioactive residues (TRR) were 10 ppm in abdominal fat, 0.3 ppm in breast muscle, 8.4 ppm in egg yolks, and 0.3 ppm in egg whites from hens dosed at the highest level. TRR in other tissues were not reported nor were data from the lower dosing levels. Tissues were extracted using the Bligh-Dyer technique; the distributions of the ¹⁴C-residues in the extracts and unextracted fractions of tissues and egg yolk are depicted in Table 6.

Table 6. Distribution of total tissue ¹⁴C-activity in extracts and unextracted fractions.

| Tissue | % of TRR in extract/fraction | | | Total |
|---------------|------------------------------|------------|-------------|-------|
| | Aqueous | Chloroform | Unextracted | |
| Breast muscle | 21.9 | 31.8 | 51.3 | 105.0 |
| Thigh muscle | 40.8 | 70.4 | 25.9 | 137.1 |
| Liver | 59.0 | 23.0 | 35.2 | 117.2 |
| Fat | 4.5 | 105.9 | 0.5 | 110.9 |
| Skin | 20.9 | 76.8 | 2.5 | 100.3 |
| Egg yolk | 4.9 | 14.9 | 75.3 | 95.1 |

¹⁴C-Residues were isolated using preparatory thin-layer chromatography (TLC) and high-performance liquid chromatography. Pentachlorophenol, pentachlorothiophenol, pentachloroaniline, and pentachlorothioanisole were tentatively identified in organic extracts of liver. Greater than 75% of the unextracted residues in liver were released by protease, although details of the protease digestion step were not reported. Abdominal fat and egg yolk contained residues corresponding to unchanged PCNB, pentachloroaniline, and thioanisole. Five conjugates of pentachlorothiophenol and two of pentachloroaniline were isolated from the aqueous portion of excreta. No details of extraction or hydrolysis procedures, or descriptions of chromatographic methods were presented. Isolated residues were not quantified and no data from confirmatory analyses were submitted. The registrant states that they plan to submit a complete report in 3/90.

Amvac Chemical Corp. (1988; MRID 41303101) submitted data pertaining to residues of [¹⁴C]PCNB in poultry tissues and eggs. Uniformly ring-labeled [¹⁴C]PCNB (radiochemical purity >98%; specific activity 12.1 mCi/mmol) was mixed with non-labeled PCNB and administered, for 5 consecutive days, to 10 laying hens orally via capsule at 25 and 50 mg/kg of body weight per day, equivalent to a dietary level of 309 and 554 ppm, respectively

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(final specific activity, $1.66-3.33 \times 10^4$ dpm/ug). Eggs were collected twice a day and yolks and whites separated, and excreta was collected once daily. The hens were sacrificed ca. 6 hours after the last dose, and tissues were collected; samples of eggs and tissues were radioassayed by LSS following combustion. Recovery of radioactivity by the radioanalysis procedures was 97-101%.

Excreta contained 65-71% of the total dose. Total radioactive residues (TRR) in egg yolk (collected after the last dose) were 1.22 ppm from the low-dose group and 2.656 ppm from the high dose group; corresponding egg white samples contained 0.017 ppm and 0.038 ppm, respectively. TRR in tissues from hens dosed at low and high levels were, respectively, 1.465 and 1.848 ppm in kidney, 1.973 and 2.415 ppm in liver, 0.127 and 0.145 ppm in thigh muscle, 0.155 and 0.306 ppm in breast muscle, 2.107 and 4.247 ppm in abdominal fat, and 1.124 and 2.181 ppm in skin with fat. The radioactive residues were not extracted or characterized.

RESIDUE ANALYTICAL METHODS

Conclusions:

The PCNB Guidance Document (1/87) requires complete descriptions of analytical methods, including validation data and representative chromatograms, for the detection and quantification of all residues and impurities of concern in or on plant and animal commodities. This requirement remains in effect. It should be noted that the nature of the residue in plants and animals is not adequately understood, and that the adequacy of the existing methodology will be determined following evaluation of the required metabolism studies.

The Pesttrak data base dated 12/13/89 indicates that PCNB is completely recovered (>80%) using Multiresidue Protocol E (both fatty and non-fatty food methods) and Protocol D. The metabolites PCA and MPCPS and the impurity PCB are completely recovered (>80%) using the fatty and non-fatty food methods of Protocol E; HCB is completely recovered from non-fatty foods and partially recovered (50-80%) from fatty foods using Protocol E. PCNB metabolites and impurities must be subjected to analyses using Protocols C and D; therefore, the following additional data are required:

- Data depicting the recovery of PCA, MPCPS, PCB, and HCB using FDA Multiresidue Protocols C and D, published in Pesticide Analytical Manual (PAM) Vol. I and available from the National Technical Information Service (NTIS) under order No. PB 203734/AS.

- The nature of the residue in plants and animals is not adequately understood. If the metabolism studies requested in the sections "Qualitative Nature of the Residue in Plants" and "Qualitative Nature of the Residue in Animals" reveal the presence of additional metabolites of concern, additional validated methods for data collection and tolerance enforcement will be required.

STORAGE STABILITY DATA

Conclusions:

The PCNB Guidance Document (1/87) requires data depicting the storage stability of PCNB, PCA, MPCPS, PCB, and HCB in all animal and plant raw agricultural commodities for which tolerances are proposed. Also required are data depicting the intervals and conditions of sample storage to validate the residue data submitted in support of established or interim tolerances for residues in or potatoes, a brassica leafy vegetable, beans, peppers, tomatoes, cottonseed, and peanuts, and their processed commodities, as well as supporting data depicting the storage stability of PCNB per se. Furthermore, it was specified that all residue data required by the Guidance Document be accompanied by data regarding the length and conditions of sample storage and supporting storage stability data.

In response to the Guidance Document, Uniroyal Chemical Co. (1988; MRIDs 40921600 and 40921601) submitted data pertaining to the storage stability of PCNB, PCA, MPCPS, PCB, and HCB in or on wheat, corn, soybeans, kidney beans, peppers, tomatoes, tomato catsup, and tomato pomace. In an Agency review (M. Flood, DEB No. 4751, 1/29/89), it is concluded that, for those commodities included in the study, correction factors should be applied to all residue values where decline over 6 months exceeds 20%; this category includes corn grain, soybeans, peppers, and tomatoes. For crops for which no storage stability data are available, additional decline data are required unless samples are analyzed within 2 weeks of harvest. Since the data failed to show any pattern of residue decline in related crops, additional data on all pertinent commodities are required.

No data were submitted on the intervals and conditions of residue sample storage called for in the Guidance Document, nor were there any data on the storage stability of residues in animal commodities. The following additional data are required:

- The sample storage conditions and intervals must be supplied for all required and previously submitted residue data for plant and animal commodities. Storage stability data on PCNB and residues of concern in support of previously submitted residue data are required for

only those samples deemed to be useful for tolerance assessment. Data are also required which depict the decline in levels of PCNB residues of concern in commodities stored under the range of conditions and for the range in intervals specified. Crop samples bearing measurable weathered residues or fortified with PCNB residues of concern and fortified meat and milk samples must be analyzed immediately after harvest or fortification and again after storage intervals that allow for reasonable unforeseen delays in sample analysis. In laboratory tests using fortified samples, the pure active ingredient and pure metabolites must be used. However, if field weathered samples are used, the test substance must be a typical end-use product. For additional guidance on conducting storage stability studies, the registrant is referred to an August, 1987 Position Document on the Effects of Storage Validity of Pesticide Residue Data available from NTIS under order no. PB 88112362/AS.

The nature of the residue in plants and animals is not adequately understood. If the requested data on plant and animal metabolism indicate the presence of additional metabolites of toxicological concern, data depicting stability of these residues during storage will be required.

References (used):

MRIDs: 40921600. 40921601.

Discussion of the data:

N/A.

MAGNITUDE OF THE RESIDUE IN PLANTS

Decisions as to the adequacy of established tolerances have been based upon evaluation of the existing data base, including data reviewed for the PCNB Guidance Document dated 1/87 and recently submitted data discussed in Agency reviews. It should be noted that the conclusions stated in this section regarding the adequacy of the established tolerances may change following the submission and review of the required data on plant metabolism and storage stability. The registrant should be urged to complete and submit all required plant metabolism studies prior to initiation of required field trials and processing studies.

The PCNB Guidance Document dated 1/87 identifies outstanding data requirements on potatoes, broccoli, cabbage, lima beans, snap beans, dry beans, peppers, tomatoes, bananas, cottonseed, and

peanuts. Processing studies are required for potatoes, beans, tomatoes, cottonseed, peanuts, and soybeans.

The Guidance Document also noted the need for data reflecting uses permitted by intrastate registrations and required that tolerances be proposed for residues in or on the appropriate commodities. The following requirements remain outstanding:

- Tolerances must be proposed for PCNB residues of concern in or on lettuce, strawberries, alfalfa forage, alfalfa hay, clover forage, and clover hay, and submit appropriate supporting residue data. Alternatively, the registrant may elect to cancel these uses permitted under AZ State Reg. No. N, CA State Reg. Nos. 10972-50043 AA and 10972-50199 AA, and TX State Reg. No. 74.

Data on crops with only seed treatments reviewed for the Guidance Document indicate that soybean plants grown from treated seed bore finite residues of PCNB per se. Thus, tolerance proposals for PCNB residues of concern in or on soybean forage and hay are required along with supporting residue data. Data depicting the potential for uptake and translocation of residues by corn, peas, rice, safflower, sugar beets, and wheat grown from [¹⁴C]PCNB-treated seed are required in order to evaluate the non-food status of these seed-treatment-only uses.

The Guidance Document also required numerous label amendments and clarifications. The following requirements remain outstanding:

- The registrant must amend all pertinent product labels to specify a maximum single application rate (expressed in lb ai/A) and a maximum seasonal rate or number of applications per season; a minimum interval between applications and a PHI must also be specified. The requested data must reflect these specifications.

Since the issuance of the Guidance Document, tolerances with regional registration have been established for residues of PCNB and its metabolites pentachloroaniline (PCA) and methyl pentachlorophenyl sulfide (MPCPS) in or on collards, kale, and mustard greens.

Recently submitted data on storage stability reveal that residues of PCNB and metabolites decline over 20% in corn grain, soybeans, peppers, and tomatoes after 6 months in frozen storage and that decline data are required for all other commodities stored for longer than 2 weeks prior to analysis. The registrant has been instructed to apply correction factors to pertinent residue values based on the decline data for the commodity in question.

It should be noted that the requirements for field trial locations specified in the Guidance Document were based on

Agricultural Statistics, 1984, or 1982 Census of Agriculture, Vol. 1, Part 51. Some of the geographic requirements specified in this review are based on the more recent statistics from the Agricultural Statistics Board, NASS, USDA; Crops Database and Vegetables Summary.

Root and Tuber Vegetables Group

Potatoes

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on potatoes (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on potatoes (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on potatoes following preplant broadcast application of G, WP, and EC formulations at 25 lb ai/A and an in-furrow at planting application using these same formulations each at 11.7 lb ai/A. [We note that the updated EPA Index to Pesticide Chemicals dated 3/89 lists the maximum rate for in-furrow application as 10 lb ai/A.] In response to these requirements, Uniroyal Chemical Co. has submitted one volume of data (1988; MRID 40816701) reflecting broadcast and in-furrow applications of an EC formulation; these data are currently under review by the Agency (R. Perfetti, personal communication, 11/20/89). Two other volumes of data regarding potatoes have been submitted; however, these data are not useful for assessing the established or proposed tolerance because information concerning the location and methods of field application and analytical methodology were not provided (MRIDs 40866014 and 40866015). The following data requirements remain outstanding:

- Data depicting PCNB residues of concern in or on potatoes following preplant broadcast application using a representative G, EC, and WP formulation at 25 lb ai/A and an in-furrow at planting application using these same formulations each at 10 lb ai/A. The tests must be conducted in ID(26%), OR(7%) or WA(17%), CA(5%), MI(3%) or WI(6%), MN(5%) or ND(6%), ME(6%), and FL(2%), since these states represent the major U.S. potato-growing regions accounting for ca. 80% of U.S. production (Agricultural Statistics Board, NASS, USDA, 1988, Crops Database, 1/88).

In addition, the PCNB Guidance Document dated 1/87 requires data on potato chips, granules, and flakes processed from potatoes treated in-furrow at 50 lb ai/A (ca. 5x) in order to obtain measurable weathered residues. In response to this requirement, Uniroyal Chemical Co. submitted potato processing data (1986-1987; MRIDs 40070401 and 40088401) which were reviewed by the Agency and found insufficient to satisfy data requirements because the specified protocol was not followed (L. Cheng, DEB Nos. 1890, 1891, and 1923 dated 2/24/87 and 1965 dated 3/10/78). Additional data were submitted (1987; MRID 40457401) and the Agency concluded that no food or feed additive tolerance is required for processed potato chips, flakes, or granules (N. Gray, DEB No. 3169, 5/17/88). It was noted that since the issuance of the Guidance Document, the requirements for potato processing have been revised to include wet and dry peel, and that additional data on these commodities are required. The review stipulated that the required processing study on wet and dry peel be conducted using formulations representative of technical PCNB containing <0.1% HCB, and that the methodology used be capable of detecting HCB at 0.001 ppm. The registrant then submitted a protocol for the required study which was approved providing that the analytical methodology used would be the same as that used in the previous study (MRID 40457401) and that PCNB would be applied at least 5x the maximum registered rate (M. Flood, DEB No. 4752, 1/10/89). Until these data are received, the following requirement remains outstanding:

- Data depicting the potential for residue concentration of PCNB residues of concern in wet and dry peel processed from potatoes treated at at least 5x the maximum registered rate bearing measurable weathered residues. The formulation(s) used must represent technical PCNB containing <0.1% HCB. Samples must be analyzed using the same methodology as in the previous study (MRID 40457401) validated at 0.001 ppm for HCB. If residues concentrate in either commodity, an appropriate feed additive tolerance will be required.

The Agency has determined that residues of PCNB and metabolites and impurities are likely to exceed 0.1 ppm, based on data on whole potatoes in MRID 40457401 and data currently under review.

References (used):

MRIDs: 40070401. 40088401. 40457401. 40816701.

References (not used):

[The following references do not contain sufficient information to allow evaluation of the data therein.]

MRIDs: 40866014. 40866015.

[The following reference contains data reviewed for the PCNB Guidance Document.]

MRID: 00159016.

Discussion of the data:

N/A.

Bulb Vegetables Group

Garlic

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on garlic (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on garlic (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires the product labeling for in-furrow treatment be amended to specify the spray band width as well as the number of row-feet per field acre. The updated EPA Index to Pesticide Chemicals dated 3/89 lists no such specifications. The following requirement remains outstanding:

- All product labels bearing directions for in-furrow application to garlic must be amended to specify both the spray band width and the number of row-feet per acre.

References (used):

N/A.

Discussion of the data:

N/A.

Brassica Leafy Vegetables Group

Broccoli

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on broccoli (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on broccoli (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on broccoli following pre-transplanting broadcast soil application of the 10% G and 75% WP formulations at 60 lb ai/A followed by at-transplanting application at 40 lb ai/A. Data are also required reflecting at-transplant application at 60 lb ai/A. In response to these requirements, Uniroyal Chemical Co. has submitted one volume of data reflecting broadcast, band, and transplant applications using the 10% G and 75% WP formulations (1988; MRID 40816702); these data are currently under review by the Agency (R. Perfetti, personal communication, 11/20/89). Until it has been determined that sufficient data are available for tolerance assessment, the following requirements remain outstanding:

- Data depicting PCNB residues of concern in or on broccoli resulting from pre-transplanting broadcast incorporated soil application of the 10% G and 75% WP formulations at 60 lb ai/A followed by at-transplanting application at 40 lb ai/A (13,000 row-feet) incorporated into a 14-inch band. Data are also required reflecting at-transplant application at 60 lb ai/A in a transplant solution. Tests must be conducted in CA (90%) and TX(5%) since these states accounted for ca. 95% of the 1986 U.S. broccoli production (Agricultural Statistics Board, NASS, USDA, Vegetables, 1986 Summary, 6/87).

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References (used):

MRID: 40816702.

Discussion of the data:

N/A.

Brussels Sprouts

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on Brussels sprouts (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on Brussels sprouts (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 concludes that the data required for broccoli will, by translation, satisfy the requirement for data on Brussels sprouts.

References (used):

N/A.

Discussion of the data:

N/A.

Cabbage

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on cabbage (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on cabbage (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on cabbage following pre-

transplanting broadcast soil application of the 10% G and 75% WP formulations at 60 lb ai/A followed by at-transplanting application at 40 lb ai/A. Data are also required reflecting at-transplant application at 60 lb ai/A. In response to these requirements, Uniroyal Chemical Co. has submitted one volume of data reflecting broadcast, band, and drench applications using the 10% G and 75% WP formulations (1988; MRID 40816703); these data are currently under review by the Agency (R. Perfetti, personal communication, 11/20/89). Until it has been determined that sufficient data are available for tolerance assessment, the following requirements remain outstanding:

- Data depicting PCNB residues of concern in or on cabbage resulting from pre-transplanting broadcast incorporated soil application of the 10% G and 75% WP formulations at 60 lb ai/A followed by at-transplanting application at 40 lb ai/A (13,000 row-feet) incorporated into a 14-inch band. Data are also required reflecting at-transplant application at 60 lb ai/A in a transplant solution. Tests must be conducted in CA(8%), FL(16%), NY(15%), NC(5%), TX(16%), and WI(9%) since these states accounted for ca. 70% of the 1986 U.S. cabbage production (1982 Census of Agriculture, Vol. I, Part 51, p. 338).

References (used):

MRID: 40816703.

Discussion of the data:

N/A.

Cauliflower

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on cauliflower (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on cauliflower (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 concludes that the data required for broccoli will, by translation, satisfy the requirement for data on cauliflower.

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References (used):

N/A.

Discussion of the data:

N/A.

Collards, kale, and mustard greens

Tolerance(s):

Tolerances with regional registration of 0.2 ppm have been established for the combined residues of PCNB and its metabolites PCA and MPCPS in or on collards, kale, and mustard greens (40 CFR 180.291[b]).

Conclusions:

At the time of the issuance of the PCNB Guidance Document dated 1/87, no tolerance was established for residues of PCNB in or on collards, kale, or mustard greens. In the Agency review of data submitted in support of the current tolerance (MRID 00158795), it was concluded that a tolerance of 0.2 ppm for the combined residues of PCNB, its metabolites PCA and MPCPS, and the impurities PCB and HCB (of which not more than 0.02 ppm is HCB) is appropriate, providing that the use is restricted to GA (W. Chin, DEB Nos. 897 and 1069; 7/17/86). Subsequently, it was concluded that PCB and HCB need not be included in the tolerance definition, since the data indicate that these residues were <0.01 ppm (nondetectable) in or on collard greens (W. Chin, DEB No. 2131; 5/6/87).

References (used):

MRID: 00158795.

Discussion of the data:

N/A.

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Legume Vegetables Group

Beans (succulent and dried)

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on beans (40 CFR 180.319). A tolerance of 0.2 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on beans (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on snap, lima, and dry beans following four applications totalling 7.5 lb ai/A using a D, EC, and WP formulations. Data on cannery waste were also required. The updated Index to Pesticide Chemicals dated 3/89 lists only EC and WP formulations for these uses. In response to these requirements, Uniroyal Chemical Co. has submitted one volume of data reflecting applications of EC and WP formulations to snap, lima, and dry beans (1988; MRID 40816704); these data are currently under review by the Agency (R. Perfetti, personal communication, 11/20/89). Additional submissions contain data reviewed previously for the guidance document (MRIDs 40866004, 40866005, and 40866006) or contain data that are not useful because no details of the field trial were provided and the variety of beans tested was not specified (MRID 00165640).

Until it has been determined that sufficient data are available for tolerance assessment, the following requirements remain outstanding:

- Data depicting PCNB residues of concern in or on snap, lima, and dry beans, and bean vines and hay following four applications of the 75% WP and 2 lb/gal EC formulations; the first application should be made at 1.5 lb ai/A and the subsequent applications must be made at 14-day intervals at 2 lb ai/A. The tests on dry beans must be conducted in CA(12%), CO(10%), ID(11%), MI(21%), NE(13%), and ND(19%); tests on snap beans must be conducted in NY(10%), OR(23%), and WI(41%); and tests on lima beans must be conducted in CA. [Production data in parentheses were obtained from Agricultural Statistics Board, NASS, USDA; 1986 Vegetables Summary.] Based on the residue data, tolerances must be established for PCNB residues of concern in or on bean vines and hay; alternatively, the registrant may propose to add amend

the pertinent product labels to include a feeding restriction.

- Data depicting the potential for concentration of PCNB residues of concern in cannery waste processed from beans bearing measurable weathered residues. If residues concentrate in this commodity, an appropriate feed additive tolerance must be proposed. A tolerance for residues in or on bean vines and hay supported by adequate residue data may substitute for the requirement for a processing study on cannery waste.

References (used):

MRID: 40816704.

References (not used):

MRIDs: 00165640. 40866004. 40866005. 40866006.

Discussion of the data:

N/A.

Fruiting Vegetables (Except Cucurbits) Group

Peppers

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on peppers (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on peppers (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on peppers following in-furrow transplant applications of the 75% WP formulation at 7.5 lb ai/A and in a transplant solution at 34 lb ai/A. In response to these requirements, Uniroyal Chemical Co. has submitted one volume of data reflecting in-furrow and transplant solution applications (1988; MRID 40816705); these data are currently under review by the Agency (R. Perfetti, personal communication, 11/20/89). Until it has been determined that sufficient data are available for tolerance assessment, the following requirements remain outstanding:

- Data depicting PCNB residues of concern in or on peppers resulting from in-furrow transplant applications of the 75% WP formulation at 7.5 lb ai/A and in a transplant solution at 34 lb ai/A. Tests must be conducted in CA(18%), FL(23%), NC(10%), and TX(16%), since these states accounted for ca. 70% of the 1986 U.S. sweet pepper production (1982 Census of Agriculture, Vol. I, Part 51, p. 350).

References (used):

MRID: 40816705.

Discussion of the data:

N/A.

Tomatoes

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on tomatoes (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on tomatoes (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on tomatoes following in-furrow transplant applications of the 75% WP formulation at 7.5 lb ai/A and in a transplant solution at 25.5 lb ai/A. In addition, data on the processed commodities of tomatoes were required. One volume of data was submitted (1978; MRID 40866016); however, the reference contained no residue or processing data. The following requirements remain outstanding:

- Data depicting PCNB residues of concern in or on tomatoes resulting from in-furrow transplant applications of the 75% WP formulation at 7.5 lb ai/A and in a transplant solution at 25.5 lb ai/A. Tests must be conducted in CA(77%), FL(9%), and IN(2%), MI(2%), or OH(5%), since these states accounted for ca. 95% of the 1986 U.S. tomato production (Agricultural Statistics Board, NASS, USDA, 1987, Vegetables, 1986 Summary, pp. 16 and 53).
- Data depicting the potential for concentration of PCNB residues of concern in juice, puree, catsup, and dry

pomace processed from tomatoes bearing measurable weathered residues. If residues concentrate in any commodity, an appropriate food or feed additive tolerance must be proposed.

References (used):

N/A.

References (not used):

MRID: 40866016.

Discussion of the data:

N/A.

Miscellaneous Commodities

Bananas

Tolerance(s):

An interim tolerance of 0.1 ppm is in effect for residues of PCNB per se in or on bananas (40 CFR 180.319). A tolerance of 0.1 ppm (not more than 0.02 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on bananas (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on bananas treated using the 1.63% RTU formulation as a dip. It was noted that residues appeared to concentrate during shipment and, therefore, sampling of bananas after treatment and after arrival in the U.S. should be required. No data have been submitted in response to the Guidance Document; therefore, the following data requirements remain outstanding:

- Data depicting PCNB residues of concern in or on bananas treated using the 1.63% RTU formulation at 1 gallon of product/700-800 stems. The stems should be dipped at the butt and tip end and the remainder of the dose brushed onto cuts and scrapes along the stems. Whole bananas must be sampled immediately after treatment and again after arrival at the U.S. port of entry following intercontinental shipment under representative transport conditions; the data must reflect minimum and maximum anticipated shipment intervals. In addition, copies of the labels for all PCNB products currently used in

countries that export bananas to the U.S. must be submitted.

References (used):

N/A.

Discussion of the data:

N/A.

Cottonseed

Tolerance(s):

A tolerance of 0.1 ppm has been established for residues of PCNB per se in or on cottonseed (40 CFR 180.291[a]).

Use directions and limitations:

The 2/22/89 update of the Index to Pesticide Chemicals identifies various single and multiple active ingredient formulations of PCNB registered for use on cottonseed as: (i) seed treatments in the form of dust, liquid, or slurry; (ii) planter box-delivered seed/soil treatments; and (iii) in-furrow and seed bed treatments.

Seed treatments: The 10-30% D, 20% WP, 2 lb/gal EC, 1.72-1.89 lb/gal FLC, and 1.7-2 lb/gal RTU formulations may be applied at 0.1-0.3 lb/cwt.

Planter box-delivered seed/soil treatments: The 10-30% D formulations are registered for use at 0.3-1 lb ai/A and the 10% G formulation may be applied at 0.3 lb ai/A.

In-furrow and seed bed treatments at planting: The 5-10% G formulations are registered for use at 0.5-5 lb ai/A and the 2 lb/gal EC and 30-70% WP formulations are registered for application at 0.325-2 lb ai/A as an open furrow (over seed) treatment at planting time.

Treated cotton forage may not be grazed by or fed to livestock. The feeding of gin trash from treated cotton is restricted. No PHI has been established.

Conclusions:

The PCNB Guidance Document dated 1/87 concludes that the established tolerance is inadequate, since tolerance exceeding residues were detected in or on cottonseed treated at 0.5x the maximum registered rate. However, it was concluded that the available data were insufficient to determine an appropriate tolerance level, and data are required reflecting in-furrow and surface band application made at planting to cotton using representative G, WP, and EC formulations. Also, the Guidance Document noted that the data available data on processing of cottonseed indicate that residues concentrate 6x in crude oil and that no concentration occurs in meal; however, since no data were available on hulls, refined oil, and soapstock, it was concluded that additional processing data are required.

The data submitted in response to the Guidance Document (MRID 41341206) reflect application of an EC formulation at 1x, although the G formulation was applied at only 0.4x the maximum registered rate of 5 lb ai/A. No data were submitted reflecting application of a WP formulation. The residues of PCNB in or on cottonseed used in the processing study (treated with a G formulation at 10 lb ai/A, or 2x the maximum registered rate) were detectable in only one sample at 0.005 ppm (the limit of detection), and the untreated sample from that test bore residues of 0.008 ppm. PCNB residues in oil were 0.022-0.024 ppm, up to 5x the level in seed, but the oil from untreated samples contained residues of 0.026-0.03 ppm. In order to adequately assess the potential for concentration of residues during processing, additional data are needed reflecting detectable residues resulting from treatment at an exaggerated rate >2x. Therefore, the following additional data are required:

- Data depicting PCNB residues of concern in or on cottonseed resulting from in-furrow and surface band over-the row applications made at-planting using a representative G formulation at 5 lb ai/A and a WP formulation at 2 lb ai/A. Tests must be conducted in AZ(7%), CA(20%), LA(7%), MS(12%), and TX(32%), since these states accounted for ca. 80% of the 1987 U.S. cotton production (Agricultural Statistics Board, NASS, USDA, Crop Database, Jan, 1988).
- Data depicting the potential for concentration of PCNB residues of concern in hulls, refined oil, and soapstock processed from cottonseed bearing measurable weathered residues. If residues concentrate in any commodity, an appropriate food or feed additive tolerance must be proposed. If the requested plant metabolism data reveal residues of concern other than PCNB, PCA, MPCPS, PCB, and HCB, additional processing data on meal and crude oil will be required.

References (used):

MRIDs: 41341206.

Discussion of the data:

Uniroyal Chemical Company, Inc. (1989; MRID 41341206) submitted data from 20 tests conducted in AZ(8), LA(8), and MS(4) depicting residues of PCNB and its metabolites and impurities of concern (PCB, HCB, PCNB, PCA, and pentachlorothioanisole or PCTA) in or on cottonseed harvested at maturity (137-175 days posttreatment) following an in-furrow application at planting of the 2 lb/gal EC and 10% G formulations at 2 lb ai/A (1x the maximum rate for an EC formulation and 0.4x that for a G formulation). Residues in or on 20 samples of cottonseed were: (i) <0.005(nondetectable)-0.012 ppm for PCNB; (ii) <0.005 ppm (nondetectable) each for PCB, HCB, and PCTA; and (iii) <0.005(nondetectable)-0.007 ppm for PCA. Apparent residues <0.005(nondetectable)-0.005 in or on 10 control samples. Data were collected using a GLC/EC method (Method No. CAM-24-73) which is similar to Method I in PAM Vol. II. The limit of detection was 0.005 ppm. Following fortification of 10 samples with each compound at 0.005-0.05 ppm, recoveries were: (i) 90-116% for PCNB; (ii) 76-100% for PCB; (iii) 78-95% for HCB; (iv) 90-110% for PCA; and (v) 89-110% for PCTA. Samples were stored frozen for ca. 169-577 days prior to analysis.

The same submission contained data from a cottonseed processing study. In two tests conducted in MS, cottonseed was harvested at maturity (149 days posttreatment) following an in-furrow application at planting of the 10% G formulation at 10 lb ai/A (2x the maximum registered rate). Residues in or on two samples of cottonseed were <0.005 ppm (nondetectable) for PCB, HCB, PCA, and PCTA; residues of PCNB were <0.005 (nondetectable) -0.005 ppm. Residues of PCNB were 0.022 ppm in refined oil, 0.024 ppm in crude oil, and were <0.005 ppm (nondetectable) in soapstock, reclaimed solvent, hulls, and meal. Residues of PCA (one sample per fraction) were 0.006 ppm in refined oil, soapstock, and crude oil, and were <0.005 ppm (nondetectable) in reclaimed solvent, hulls, and meal. Residues of PCB, HCB, and PCTA (one sample per fraction) were <0.005 ppm (nondetectable) in refined oil, soapstock, crude oil, reclaimed solvent, hulls, and meal. Apparent residues of PCNB in or on control samples (one sample per fraction) were 0.026 ppm in refined oil, 0.030 ppm in crude oil, 0.005 ppm in hull, and <0.005 ppm (nondetectable) in reclaimed solvent and meal. Apparent residues of PCA in or on control samples (one sample per fraction) were 0.006 ppm in refined oil, soapstock, and crude oil, and were <0.005 ppm (nondetectable) in reclaimed solvent, hulls and meal. Apparent residues of PCB, HCB, and PCTA in control samples (one sample per fraction) were <0.005 ppm (nondetectable) each in refined oil, soapstock, crude oil, reclaimed solvent, hulls, and meal. Data

were collected using a GLC/EC method (Method No. CAM-24-73) similar to the GLC/EC method listed in PAM Vol. II as Method I. The limit of detection was stated as 0.005 ppm for each compound, although we note that some control samples of seed, crude oil, and refined oil bore apparent PCNB residues greater than 0.005 ppm. Recovery of PCNB was 92-116% from cottonseed samples fortified at 0.005-0.05 ppm, 98% from soapstock fortified at 0.05 ppm, 132% from refined oil fortified at 0.005 ppm, 95% from crude oil fortified at 0.02 ppm, 80% from hulls fortified at 0.05 ppm, and 84% from meal fortified at 0.05 ppm. Recovery of PCB, HCB, PCA, and PCTA was 70-110% from cottonseed processed commodities fortified at 0.005-0.05 ppm. Samples were stored frozen for unreported intervals prior to analysis.

Geographic representation of the data is adequate since the test states of AZ(7%), LA(7%), and MS(12%) along with the neighboring states of TX(32%), and CA(20%) represent ca. 78% of the U.S. cotton production (Agricultural Statistics Board, NASS, USDA Crop Database, Jan. 1988). However, the available residue data do not reflect current maximum registered use pattern of PCNB in cottonseed because the G formulation was not applied at 1x the maximum registered rate and no data were submitted reflecting registered use of a WP formulation. Furthermore, the processing study is inadequate since residue levels in or on cottonseed were not high enough to assess the potential for concentration of residues. Additional data are required.

Peanuts

Tolerance(s):

An interim tolerance of 1 ppm is in effect for residues of PCNB per se in or on peanuts (40 CFR 180.319). A tolerance of 2 ppm (not more than 0.4 ppm of which is HCB) has been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in or on peanuts, and a tolerance of 5 ppm (not more than 0.3 ppm of which is HCB) has been established for these same residues in or on peanut hulls (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 requires data depicting PCNB residues of concern in or on peanuts and peanut hulls harvested 45 days following surface-banded application made at pegging using the 30% G and 75% WP formulations. Data are also required to support SLN registrations permitting at-pegging application by overhead sprinkler (AL, GA, OK, and TX) and using aerial equipment (TX). In addition, data on processed commodities of peanuts are required.

Uniroyal Chemical Co. has submitted one volume of data from tests using an EC and a FlC formulation applied with overhead irrigation equipment in GA, OK, and VA (1988; MRID 41002507). The data were submitted in support of a request for an experimental-use permit for the new FlC formulation, and were reviewed in that context by the Agency (L. Propst, DEB No. 5217; 5/8/89). The data for the EC formulation indicate that combined residues of PCNB, PCA, MPCPS, PCB, and HCB were <0.025 (nondetectable)-<0.028 ppm in or on three samples from VA and <0.335-1.146 ppm in or on four samples from GA (the data from OK are disregarded due to apparent contamination of plants from untreated plots); the combined residues in or on the GA and VA samples included HCB residues of <0.005 (nondetectable)-0.0061 ppm. With respect to the requirements in the Guidance Document, these data do not satisfy the requirement for soil applications, aerial applications, or overhead sprinkler applications in OK or TX. The data indicate that application of an EC formulation by overhead sprinkler in GA (EPA SLN No. GA810003) and AL (AL800010) is not likely to result in tolerance-exceeding residues.

Additional data from field trials on peanuts were submitted (1977; MRID 40866012), but are not useful for tolerance assessment because application was made >150 days before harvest, the formulation used was not specified, and the method of application was not reported. Three volumes containing processing data were submitted (MRIDs 00165639, 40866011, and 40866013) but are disregarded because spiked samples were used and no commodities of interest were analyzed.

The following requirements remain outstanding:

- Data depicting PCNB residues of concern in or on peanut nutmeats and hulls harvested 45 days following surface-banded application made at pegging using the 30% G and 75% WP formulations each at 10 lb ai/A in separate tests. Tests must be conducted in AL(13%) or GA(44%), NC(11%) or VA(6%), and OK(6%) or TX(12%), since these states accounted for ca. 80% of the 1987 U.S. cotton production (Agricultural Statistics Board, NASS, USDA, Crop Database, Jan, 1988).
- Data depicting PCNB residues of concern in or on peanut nutmeats and hulls harvested 45 days following overhead sprinkler application of the 75% WP and 2 lb/gal EC formulations at 10 lb ai/A, made at pegging. The tests must be conducted in TX to represent the states where this use is permitted. Alternatively, the registrant may elect to cancel these uses permitted under EPA SLN Nos. OK840008, TX790017, and TX840015.
- Data depicting PCNB residues of concern in or on peanut nutmeats and hulls harvested 45 days following aerial

application of the 10% G formulation at 10 lb ai/A made at pegging. The test must be conducted in TX where this use is permitted. Alternatively, the registrant may elect to cancel this use permitted under EPA SLN No. TX780043.

- Data depicting the potential for concentration of PCNB residues of concern in meal, crude oil, refined oil, and soapstock processed from peanuts bearing measurable weathered residues. If residues concentrate in any commodity, an appropriate food or feed additive tolerance must be proposed. If the requested plant metabolism data reveal residues of concern other than PCNB, PCA, MPCPS, PCB, and HCB, additional processing data on meal and crude oil will be required.

References (used):

MRIDs: 41002507.

References (not used):

MRIDs: 00165639. 40866011. 40866012. 40866013.

Discussion of the data:

N/A.

Crops with Seed Treatments Only

The PCNB Guidance Document dated 1/87 concludes that, since finite residues of PCNB per se were detected in or on leaves from soybeans grown from PCNB-treated seed, seed treatment of soybeans must be considered a food use. It is required that tolerances be proposed for PCNB residues of concern in or on soybeans and soybean forage and hay and that supporting residue data be submitted. The Guidance Document also requires data depicting the uptake and translocation of residues into the food and feed commodities of corn, peas, rice, safflower, sugar beets, and wheat grown from seed treated with [¹⁴C]PCNB at the maximum registered seed treatment rate for each crop. In response to the Guidance Document, data were submitted indicating that commodities of corn, peas, sugar beets, soybeans, and wheat grown from treated seed bore detectable residues (1988; MRID 40862901). The Agency has determined that seed treatments using PCNB should not be categorized as non-food uses and that tolerances must be proposed for PCNB residues of concern in or on commodities of these crops (M. Flood, DEB No. 4770; 1/24/89). It was noted that, after the nature of the residue in plants is adequately elucidated, field trials should be conducted to support the proposed tolerances. The Agency review also determined that

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processing studies would not be required on corn and soybeans if application at 28 and 5.4x the maximum registered rates, respectively, resulted in no detectable residues in or on corn grain or soybean seed; residues are not expected to concentrate during the processing of non-fatty commodities such as sugar beets and wheat.

The following additional data are required:

- The registrant must propose tolerances for PCNB residues of concern in or on corn grain, forage, and fodder grown from seed treated using a representative D and an EC or RTU formulation at 0.046 lb ai/100 lb of seed. In one test for each formulation, seed should be treated at 1.29 lb ai/100 lb of seed (28x); if no detectable residues result in grain, a processing study will not be required. The tests must be conducted in CO(2%), GA(1%), IL(17%), IA(18%), NE(11%) and TX(2%), since these states accounted for ca. 50% of 1987 U.S. corn grain production and represent the major U.S. corn-growing regions (Agricultural Statistics Board, NASS, USDA, 1988, Crops Database, 1/88).
- The registrant must propose tolerances for PCNB residues of concern in or on soybean seed, forage, and hay grown from seed treated using a representative D and an EC or RTU formulation at 0.105 lb ai/100 lb of seed. In one test for each formulation, seed should be treated at 0.57 lb ai/100 lb of seed (5.4x); if no detectable residues result in soybeans, a processing study will not be required. The tests must be conducted in IA(18%), LA(2%), MN(10%), MO(8%), OH(8%), and TN(2%), since these states accounted for ca. 50% of 1987 U.S. soybean production and represent the major U.S. soybean-growing regions (Agricultural Statistics Board, NASS, USDA, 1988, Crops Database, 1/88).
- The registrant must propose tolerances for PCNB residues of concern in or on peas (succulent and dried), vines, and hay grown from seed treated using a representative D and an EC or RTU formulation at 0.105 lb ai/100 lb of seed. The tests must be conducted in DE(succulent, 4%), ID(dry, 37%), MI(succulent, 23%), NY(succulent, 4%), WA(dry, 63%; succulent, 20%), and WI(28%), since these states accounted for virtually all of the 1987 U.S. production of succulent and dry peas (Agricultural Statistics Board, NASS, USDA, 1987-1988; Crops Database, 1/88, and Vegetables, 1986 Summary).
- The registrant must propose tolerances for PCNB residues of concern in or on sugar beet roots and tops grown from seed treated using representative D, EC, and FlC

formulations at 0.25 lb ai/100 lb of seed. The tests must be conducted in CA(22%), CO(3%), ID(15%), MI(10%), MN(22%), and OR(2%), since these states accounted for ca. 70% of 1987 U.S. sugar beet production and represent the major U.S. sugar beet-growing regions (Agricultural Statistics Board, NASS, USDA, 1988, Crops Database, 1/88).

- The registrant must propose tolerances for PCNB residues of concern in or on wheat grain, forage, hay, and straw grown from seed treated using a representative D formulation at 0.062 lb ai/bu and a representative EC or RTU formulation at 0.053 lb ai/100 lb of seed. The tests must be conducted in CA(2%), CO(5%), KS(17%), OK(6%), TX(5%), and WA(5%), since these states accounted for ca. 40% of 1987 U.S. wheat production and represent the major U.S. wheat-growing regions (Agricultural Statistics Board, NASS, USDA, 1988, Crops Database, 1/88).

References (used):

MRID: 40862901.

Discussion of the data:

N/A.

MAGNITUDE OF THE RESIDUE IN MEAT, MILK, POULTRY, AND EGGS

Tolerances:

Tolerances of 0.05, 0.15, and 0.05 ppm, respectively, (not more than 0.02, 0.05, and 0.02 ppm of which is HCB) have been proposed for residues of PCNB, its metabolites PCA and MPCPS, and impurities PCB and HCB in the fat, meat, and meat byproducts of cattle, goats, hogs, horses, and sheep; tolerances of 0.05 ppm (not more than 0.02 ppm of which is HCB) have been proposed for these same residues in milk and eggs (PP#1F1083, Amendment of 1/83).

Conclusions:

The PCNB Guidance Document dated 1/87 concludes that the adequacy of the proposed tolerances for residues in animal commodities, the need for tolerances for poultry tissues, and the sufficiency of the supporting data will be assessed following receipt and review of the data requested for plant and animal metabolism and residues on livestock feed items.

In an Agency review of data on processed potatoes (DEB No. 3169, 5/17/88), N. Gray recommended that the following tolerances

(expressed as PCNB, PCA, and HCB) be proposed to cover residues from the feeding of potato waste to livestock: (i) 0.05 ppm in poultry fat; (ii) 0.02 ppm in animal and poultry tissues; (iii) 0.03 ppm in milk fat (representing 0.0012 ppm in whole milk); and (iv) 0.01 ppm in eggs. When the outstanding data requirements for plant and animal metabolism, storage stability data, and residues in or on feed commodities have been fulfilled, the need for and nature of tolerances for residues in animal commodities will be determined.

References (used):

N/A.

Discussion of the data:

N/A.

MASTER RECORD IDENTIFICATION NUMBERS

00158795 Interregional Research Project No. 4 (1986) The Results of Tests on the Amount of PCNB Residues Remaining in or on Collards, Kale and Mustard Greens Including a Description of the Analytical Method Used: Amendment. Unpublished study. 38 p.

40070401 Ball, J., comp. (1987) Magnitude of the Residue in Processed Potatoes PCNB and it's Metabolites and Impurities: Morse Lab. No. 42428 and Uniroyal No. UR1403. Unpublished compilation prepared by Uniroyal Chemical Co., Inc. in cooperation with Morse Laboratories. 81 p.

40088401 Ball, J. (1987) Magnitude of the Residue in Fried Treated Processed Potatoes PCNB and its Metabolites and Impurities: Morse Lab No. 42370 and Uniroyal No. UR1404. Unpublished compilation prepared by Uniroyal Chemical Co., Inc. in cooperation with Morse Laboratories, Inc. 73 p.

40457401 Ball, J. (1987) Magnitude of the Residue Terraclor Residues in Processed Potatoes. Unpublished compilation prepared in cooperation with Michigan State Univ. and Morse Laboratories, Inc. 689 p.

40816701 Ball, J. (1988) Magnitude of the Residue Terraclor and Its Metabolites in Potatoes: Uniroyal Report No. UR 1414. Unpublished study prepared by Morse Laboratories, Inc. in cooperation with Pan-Agricultural Labs, Inc. 245 p.

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