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

FEB 3 1992

OFFICE OF
PESTICIDES AND TOXIC
SUBSTANCES

MEMORANDUM

SUBJECT: Uniroyal Chemical Co.: Response to the PCNB
Reregistration Standard: Storage Stability Study (MRID
#'s 41829501 and 41829502, CBRS # 7877, Barcode No.
D163419.)

FROM: R. B. Perfetti, Ph.D., Chemist *RB Perfetti*
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THRU: E. Zager, Chief *E Zager*
Chemistry Branch II: Reregistration Support
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TO: W. Burnam, Acting Chief
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Health Effects Division (H7509C)

and

L. Rossi, Chief
Reregistration Branch
Special Review and Reregistration Division (H7508C)

Attached please find a review of a storage stability study submitted by Uniroyal Chemical Co. in response to the PCNB Reregistration Standard. This data was reviewed by Acurex Corporation under supervision of CBRS, HED.

This information has undergone secondary review in CBRS and has been revised to reflect the Branch policies.

Please see our conclusions in the attachment regarding the acceptability of the data submitted by the Registrant.

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No Residue Data Summary Table has been included with this review owing to the number of other PCNB actions pending.

If you need additional input please advise.

Attachment 1: Review of a PCNB Storage Stability Study.

cc (With Attachment 1): R. B. Perfetti, J. Burrell/C. Furlow (PIB/FOD), PCNB Reregistration Standard File, PCNB Subject File Circ. (7), RF and Acurex.

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**PCNB (CBRS No. 7877)
(Chemical Code 056502)**

TASK 3

**Registrant's Response
to Residue Chemistry Data
Requirements**

September 19, 1991

Contract No. 68-DO-0142

Submitted to:

U.S. Environmental Protection Agency
Arlington, VA 22202

Submitted by:

Acurex Corporation
Environmental Systems Division
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0077

PCNB (CBRS No. 7877; DP Barcode D163419)

(Chemical Code 056502)

REGISTRANTS RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS

TASK - 3

BACKGROUND

The PCNB Guidance Document dated January, 1987 required data depicting the storage stability of pentachloronitrobenzene (PCNB), pentachloroaniline (PCA), pentachlorobenzene (PCB), hexachlorobenzene (HCB), and pentachlorothioanisole (MPCPS) in all animal and plant raw agricultural commodities for which tolerances are proposed. [Tolerances have been proposed to replace the interim tolerances currently in effect for PCNB, *per se* (40 CFR 180.319). The proposed tolerances (PP#1F1083, Amendment of 1/83) are expressed in terms of PCNB, its metabolites PCA and MPCPS, and the impurities PCB and HCB, and specify limits on the amount of HCB in the residue.] Also required were 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 on 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, the Guidance Document required that descriptions of the analytical methodology used be submitted with all required residue data.

In response to the Guidance Document, Uniroyal Chemical Co. (1988; MRID 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 ketchup, and tomato pomace, which were reviewed by the Agency (M. Flood, CBRS No. 4751, 1/29/89). It was concluded that for those commodities included in the study, correction factors should be applied to all residue values where decline over 6 months exceeded 20%; this category included corn grain, soybeans, peppers, and tomatoes. Because the data failed to show any pattern of residue decline in related crops, additional data on all pertinent commodities were required.

Uniroyal Chemical Co. has since submitted data (1991; MRIDs 41829501 and 41829502) concerning storage stability of PCNB and its allied metabolites in or on peanuts and cottonseed. These data, assigned CBRS No. 7877, are reviewed here for adequacy in fulfilling requirements for registration.

CONCLUSIONS

These data satisfy the requirements stated in the Guidance Document regarding storage stability data for peanuts and cottonseed, provided that the data used to support the proposed

tolerances reflect sample storage intervals no longer than 12 months. The data indicate that residues of PCNB and its allied metabolites, PCA, MPCPS, PCB, and HCB are stable in or on peanuts and cottonseed stored at $<0^{\circ}\text{C}$ for up to 12 months. Method MP-PCNB-MA is adequate for the collection of data on residues of PCNB, PCA, MPCPS, PCB, and HCB in or on cottonseed and peanuts.

RECOMMENDATIONS

The registrant should be reminded that data documenting the intervals and conditions of sample storage must accompany all residue data submitted to support tolerances.

DETAILED CONSIDERATIONS

Residue Analytical Methods

Uniroyal Chemical Co. (1991; MRIDs 41829501 and 41829502) submitted data on the stability of residues of PCNB, its metabolites PCA and MPCPS, and the impurities PCB and HCB in peanuts and cottonseed stored at $<0^{\circ}\text{C}$. The data for these storage stability studies were collected by Hazelton Laboratories, Inc. using GLC/EC method MP-PCNB-MA. Samples of plant commodities are dried with anhydrous sodium sulfate and blended with hexane. Samples high in lipids are cleaned up by gel permeation chromatography (GPC). The hexane extract is reduced in volume and methylene chloride:cyclohexane (50:50, v/v) is added. The residues are passed through a GPC column and eluted with methylene chloride:cyclohexane (50:50, v/v). The cleaned up residues are reduced in volume and dissolved in petroleum ether. The residues are then applied to a Florisil column and eluted with ethyl ether:petroleum ether (3:97, v/v). The solvent is evaporated and the residues are reconstituted in isooctane. Analyses are conducted using GLC equipped with a 3% OV-1 or 1.5% SP-2250/1.95% SP-2401 column and electron capture detector. The limit of detection was 0.005 ppm for each compound. Recovery data are presented in Table 2.

Method MP-PCNB-MA is adequate for the collection of data on residues of PCNB, PCA, MPCPS, PCB, and HCB in or on cottonseed and peanuts.

Storage Stability Data

Uniroyal Chemical Co. submitted data (1991; MRID 41829501) depicting the storage stability of PCNB, PCA, PCB, HCB, and MPCPS in or on peanuts. All samples were fortified at 0.2 ppm with PCNB and its allied metabolites. Samples fortified with PCNB and its allied metabolites were stored at $<0^{\circ}\text{C}$ (unspecified) for up to 12 months prior to analysis. Two replicate samples of peanuts were fortified with parent and each metabolite and analyzed at each time interval. Storage intervals, residues found at intervals in stored samples, and concurrent method recoveries are presented in Table 1.

In addition, Uniroyal Chemical Corp. submitted data (1991; MRID 41829502) depicting the stability of PCNB and its metabolites in or on cottonseed. All samples were fortified with PCNB and its metabolites at 0.2 ppm. Samples containing fortified residues of PCNB were stored at $<0^{\circ}\text{C}$ (unspecified) for up to 12 months prior to analysis. Storage intervals, method spike recoveries, and residues found at intervals in stored samples are listed in Table 2.

PCNB residues in samples were determined using GLC/EC method MP-PCNB-MA. The limit of detection was 0.005 ppm for each of the commodities and apparent residues in or on all control samples were <0.005 ppm.

These data indicate that residues of PCNB and its allied metabolites are stable in cottonseed and peanuts stored at $<0^{\circ}\text{C}$ for up to 12 months.

Table 1. Recovery of PCNB and its allied metabolites from peanut samples fortified at 0.2 ppm and stored frozen at $<0^{\circ}\text{C}$ for up to 360 days or fortified at 0.2 ppm on the day of extraction (method recovery).

Fortification Compound	Uncorrected ppm recovery after storage intervals (days)						
	0	30	60	90	120	180	360
PCNB							
storage sample 1	0.171	0.162	0.148	0.170	0.161	0.162	0.191
storage sample 2	0.168	0.169	0.143	0.167	0.156	0.170	0.182
method recovery	0.176, 0.177	0.185	0.131	0.187	0.176	0.186	0.178
PCA							
storage sample 1	0.168	0.164	0.143	0.155	0.165	0.155	0.199
storage sample 2	0.168	0.168	0.121	0.169	0.162	0.170	0.188
method recovery	0.172, 0.178	0.246	0.148	0.173	0.190	0.196	0.173
PCB							
storage sample 1	0.172	0.160	0.125	0.165	0.150	0.162	0.195
storage sample 2	0.170	0.169	0.135	0.162	0.145	0.173	0.183
method recovery	0.176, 0.173	0.175	0.122	0.168	0.162	0.191	0.179
HCB							
storage sample 1	0.161	0.154	0.131	0.163	0.153	0.156	0.197
storage sample 2	0.160	0.160	0.137	0.153	0.146	0.164	0.188
method recovery	0.165, 0.168	0.169	0.123	0.155	0.167	0.184	0.178
MPCPS							
storage sample 1	0.168	0.173	0.137	0.175	0.163	0.165	0.178
storage sample 2	0.167	0.174	0.164	0.166	0.168	0.177	0.184
method recovery	0.174, 0.179	0.184	0.142	0.173	0.189	0.192	0.169

Table 2. Recovery of PCNB and its allied metabolites from cottonseed samples fortified at 0.2 ppm and stored frozen at <0 °C for up to 360 days or fortified at 0.2 ppm on the day of extraction (method recovery).

Fortification Compound	Uncorrected ppm recovery after storage intervals (days)					
	0	30	60	90	180	360
PCNB						
storage sample 1	0.188	0.180	0.182	0.179	0.178	0.190
storage sample 2	0.183	0.186	0.162	0.180	0.174	0.183
method recovery	0.182	0.192	0.208	0.191	0.190	0.189
PCA						
storage sample 1	0.168	0.175	0.199	0.179	0.176	0.198
storage sample 2	0.180	0.184	0.151	0.171	0.174	0.192
method recovery	0.176	0.184	0.206	0.170	0.214	0.179
PCB						
storage sample 1	0.191	0.181	0.181	0.193	0.184	0.198
storage sample 2	0.192	0.184	0.166	0.195	0.181	0.191
method recovery	0.187	0.188	0.197	0.193	0.211	0.195
HCB						
storage sample 1	0.174	0.170	0.168	0.169	0.166	0.175
storage sample 2	0.176	0.172	0.145	0.175	0.162	0.170
method recovery	0.172	0.173	0.183	0.186	0.193	0.171
MPCPS						
storage sample 1	0.160	0.182	0.182	0.189	0.188	0.181
storage sample 2	0.183	0.187	0.166	0.189	0.185	0.196
method recovery	0.183	0.189	0.219	0.168	0.184	0.112