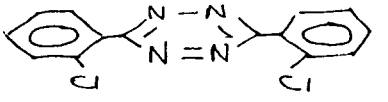
40644901
02
03
04

DATA EVALUATION

1. CHEMICAL:

chemical name: 3,6-bis (2-chlorophenyl)-1,2,4,5-tetrazine
common name: clofentezine
trade name: Apollo SC
structure:



CAS #: 74115-24-5
Shaughnessy #: 125501

2. TEST MATERIAL: n.a.

3. STUDY/ACTION TYPE:

4. STUDY IDENTIFICATION:

Nor-Am Chemical Company. Transmittal Document. undated. received EPA 6/17/88 under MRID # 00406649-00.

Davis, C. and R. Whiteoak. Response to Question Raised in EPA Letter of February 11, 1988 Concerning NOR-AM's Field Soil Dissipation Study (NOR-AM Report W55, EPA Accession Number 262273). performed by NOR-AM Chemical Company, Wilmington, DE. dated 6/14/88. Received EPA 6/17/88 under MRID # 00406649-01.

Snowden, P.J. (W62) Analytical Method for Residues of Clofentezine in Soil (Contains Addendum). performed by FBC Limited/Schering AG, Saffron Walden, Essex, U.K. dated 12/20/85 (original), 6/9/88 (addendum). received EPA 6/17/88 under MRID # 00406649-02.

Peatman, M.H. and P.J. Snowden. (W45) Stability of NC 21314 Residue in Soil During Deep Freeze Storage. performed by FBC Limited/Schering AG, Saffron Walden, Essex, U.K. dated 11/2/88. received EPA 6/17/88 under MRID # 00406649-03.

Davis, C. and P. Carter. Response to EPA Letter of May 24, 1988 Concering [sic] Stability (63-13) and Submittal of Samples (64-1). performed by FBC Limited/Schering AG, Saffron Walden, Essex, U.K. dated 6/14/88). received EPA 6/17/88 under MRID # 00406649-04.

5. REVIEWED BY:

Typed Name: E. Brinson Conerly
Title: Chemist, Review Section 3
Organization: EAB/HED/OPP

E. B. Conerly
8/10/88

2046121

6. APPROVED BY:

Typed Name: Emil Regelman
Title: Supervisory Chemist, Review Section 3
Organization: EAB/HED/OPP

AUG 10 1988

7. CONCLUSIONS:

The applicant has presented satisfactory supplementary data to answer EAB questions. These data together with the original study fulfill the data requirement for field dissipation.

8. RECOMMENDATIONS:

The applicant should be informed of our acceptance of the data for field dissipation.

9. BACKGROUND:

The applicant is responding to EAB questions, which are as follows:

1) EAB question: How were spiked samples handled vs. experimental samples in the orchard application?

Nor-Am response: [NOT VERBATIM] They were fortified just prior to laboratory extracting. Daily analytical runs contained at least one recovery test, and an analysis of unfortified control soil.

EAB response: This information is satisfactory to clarify sample handling. Information discussed below indicates that extractability does not decrease significantly when samples are stored briefly.

2) EPA comment: ... provide any clarification information on extractability of the compound vs. contact time with the soil.

NOR-AM response: ..."...Recovery efficiencies obtained from samples extracted [at various times up to 48 hours] were between 83-102%...In a field soil sampling situation, untreated soil samples would be spiked and frozen shortly thereafter. Our 48-hour period of holding the spiked samples prior to freezing is very unlikely and probably represents a worst case situation....data on frozen storage stability of clofentezine in field treated and laboratory treated soil samples [indicated] recovery efficiency averaged 86.8% over 2 years of frozen storage...."

EPA response: The information is satisfactory to demonstrate that sample storage over the period of the study probably did not materially affect the analytical results.

The data base on clofentezine is as follows:

hydrolysis -- labile -- $t_{1/2}$ from 248.8 hr at pH 4.95 to 34.4 hours at pH 6.98 to 4.3 hr at pH 9.18 -- the principle product is 2-chlorobenzoic (2-chlorobenzylidine) hydrazide, which further degrades to 2-chlorobenzonitrile and 2-chlorobenzamide

photolysis

aqueous -- labile -- $t_{1/2}$ < 7 days at pH 5

soil -- stable -- 85.9% parent remained after 31 days

soil metabolism -- moderately labile -- $t_{1/2}$ 4 -- 12 wks, products were CO_2 and a minor amount of 2-chlorobenzoic acid (interim report--final report is not in the file)

leaching -- no significant leaching of parent or degradation products

soil dissipation -- further information requested, and discussed in this review. On the basis of the preliminary data, no leaching indicated-- $t_{1/2}$ = 32.4 - 83 days

fish bioaccumulation -- accumulation unlikely (EBC 6/27/88)

These data indicate a relatively short-lived, non-mobile compound.

10. DISCUSSION OF INDIVIDUAL TESTS OR STUDIES: below

10.1 A. STUDY IDENTIFICATION

Nor-Am Chemical Company. Transmittal Document. undated. received EPA 6/17/88 under MRID # 00406649-00.

B. MATERIALS AND METHODS: n.a.

C. REPORTED RESULTS: n.a.

D. STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES: n.a.

E. REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS: n.a.

10.2 A. STUDY IDENTIFICATION

Davis, C. and R. Whiteoak. Response to Question Raised in EPA Letter of February 11, 1988 Concerning NOR-AM's Field Soil Dissipation Study (NOR-AM Report W55, EPA Accession Number 262273). performed by NOR-AM Chemical Company, Wilmington, DE. dated 6/14/88. Received EPA 6/17/88 under MRID # 00406649-01.

B. MATERIALS AND METHODS: n.a.

C. REPORTED RESULTS:

"Spiked samples have been extracted after storage at room temperature for periods of up to 48 hours....Recovery efficiencies obtained from samples extracted 1, 4, 8, and 48 hours post fortification were between 83% - 102%. [In another study]...recovery efficiency averaged 86.8% over 2 years of frozen storage.

D. STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES: n.a.

We believe that these data adequately demonstrate that our method of laboratory spiking of soil prior to extraction for analysis is as representative of actual recoveries as is field spiking."

E. REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS: n.a.

We agree. See also the review of Study 3 and Study 4.

10.3 A. STUDY IDENTIFICATION

Snowden, P.J. (W62) Analytical Method for Residues of Clofentezine in Soil (Contains Addendum). performed by FBC Limited/Schering AG, Saffron Walden, Essex, U.K. dated 12/20/85 (original), 6/9/88 (addendum). received EPA 6/17/88 under MRID # 00406649-02.

B. MATERIALS AND METHODS: n.a.

C. REPORTED RESULTS:

"The recovery efficiencies of clofentezine from fortified individual 50 gm portions of untreated soils are shown in table 1. [attached]

Apparent residues found in the respective control (untreated) samples used for fortification have been subtracted in each case. The overall mean recovery efficiency was 94%.

The standard deviation for 9 recovery tests performed at fortification levels ranging from 0.01 to 0.20 mg/kg clofentezine ... was 11%."

Recovery efficiencies ...[at various contact times]... are presented in table 2 [attached]. All recovery effiency values obtained were within the specifications of the analytical method..."

D. STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES:

"...Even after 48 hours, recovery efficiency was comparable to the shorter (1 to 8 hours) intervals tested, confirming that adequate extractability of clofentezine is maintained though [sic] increased contact time."

E. REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS: n.a.

We agree that the applicant appears to have established satisfactory precision and accuracy in this analytical method. THIS REPORT CONTAINS ERRONEOUS MATERIAL ON PG. 13. THE CORRECTION IS ON PG. 20.

5.2 Recovery efficiency

The recovery efficiencies of clofentezine from fortified individual 50 g portions of untreated soils are shown in Table 1.

Apparent residues found in the respective control (untreated) samples used for fortification have been subtracted in each case. The overall mean recovery efficiency was 94%.

5.3 Precision

The standard deviation for 9 recovery tests performed at fortification levels ranging from 0.01 to 0.20 mg/kg clofentezine during December 1985 was 11%.

Table 1

Recovery efficiency of clofentezine from fortified samples

Fortification level (mg/kg)		Recovery efficiency (%)
0.01		118
0.02		99, 84
0.05		87, 83
0.10		94
0.11		92
0.15		91
0.20		98
Overall summary	Mean $\bar{x}$	94
	Std. dev. $\sigma(n-1)$	11
	Number of tests [n]	9

APPENDIX I

Extractability with increased soil contact time

I.1 EXPERIMENTAL

Samples (50 g) of air-dried untreated sandy loam soil were weighed into soxhlet thimbles and moistened with 10 ml of water according to section 4.4.1. Each of these samples was then fortified at a level of 0.2 mg/kg clofentezine by pipetting 1.0 ml from a solution containing 10 µg/ml clofentezine in acetone. The thimbles were then stored at room temperature to await extraction.

At post-fortification intervals of 1, 4, 8 and 48 hours duplicate samples were soxhlet extracted with acetone as prescribed in section 4.4 onwards, with final determination of residues by HPLC monitoring U.V. absorption at 268 nm (section 4.2). All chromatographic data was captured and interpreted by a Beckman CALS laboratory information management system using Peak-Pro software (Appendix II). Results were calculated as described in section 5.6 and 5.7.

I.2 RESULTS AND DISCUSSION

Recovery efficiencies obtained from these tests are presented in Table 2. All values have been corrected for an apparent residue of 0.002 mg/kg found in a non-fortified sample of the soil used in the tests.

Table 2

Recovery efficiency of clofentezine at increased contact times

Contact time (interval between fortification and extraction (hours))	Recovery efficiency (%) (fortification at 0.2 mg/kg)	
	Individual results	Mean
1	101, 75	88
4	95, 91	93
8	86, 89	88
48	88, 82	85

All recovery efficiency values obtained were within the specifications of the analytical method ($x \pm 2\sigma$) as stated in sections 5.2 and 5.3. Even after 48 hours, recovery efficiency was comparable to the shorter (1 to 8 hours) intervals tested, confirming that adequate extractability of clofentezine is maintained though increased soil contact time.

10.4 A. STUDY IDENTIFICATION

Peatman, M.H. and P.J. Snowden. (W45) Stability of NC 21314 Residue in Soil During Deep Freeze Storage. performed by FBC Limited/Schering AG, Saffron Walden, Essex, U.K. dated 11/2/88. received EPA 6/17/88 under MRID # 00406649-03.

B. MATERIALS AND METHODS:

1) Samples were newly spiked, frozen, and analyzed at intervals up to 24 months. [Details attached.]
2) Previously analyzed samples in two different test soils were reanalyzed at intervals of 12, 18, and 24 months.

C. REPORTED RESULTS:

1) In the first study, there appeared to be a first order decline in extractability with a half-time of approximately 3.4 years. [details attached]
2) In the second study, similar results were obtained from the soil used in test one (a sandy loam). The other soil, a clay loam, showed greater extractability -- 85 - 92% of original residues were still extractable after 18 months.

D. STUDY AUTHOR'S CONCLUSIONS/QUALITY ASSURANCE MEASURES: See "Results" above.

E. REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS:

Soil type and length of storage do appear to make some difference in the extractability of clofentezine, but it seems that a relatively long storage period does not compromise the validity of the analyses.

10.5 A. STUDY IDENTIFICATION

Davis, C. and P. Carter. Response to EPA Letter of May 24, 1988 Concering [sic] Stability (63-13) and Submittal of Samples (64-1). performed by FBC Limited/Schering AG, Saffron Walden, Essex, U.K. dated 6/14/88). received EPA 6/17/88 under MRID # 00406649-04.

E. REVIEWER'S DISCUSSION AND INTERPRETATION OF STUDY RESULTS:

This document is a response to RCB comments and has been passed on to that branch.

11. COMPLETION OF ONE-LINER: updated one-liner attached

12. CBI APPENDIX: attached

Table 3

Summary of NC 21 314 residues in laboratory fortified soils

Storage interval (Days)	Mean NC 21 314 residue level (mg/kg)	% of nominal fortification level	
0	0.51	102	4.62497
15	0.52	103	4.63973
29	0.50	100	4.60517
57	0.49	97	4.57971
116	0.46	91	4.51086
177	0.44	89	4.48864
262	0.44	89	4.48864
351	0.39	77	4.34381
373	0.37	74	4.30407
422	0.38	76	4.33073
534	0.36	71	4.26268
718	0.37	74	4.30407

Regression analysis of these data using an equation of the form $y = be^{mx}$ showed a reasonable correlation between residue and time, with a computed correlation coefficient of 0.931, indicating a first order decline of extracted NC 21 314 residues over the 718 day storage period. The computed NC 21 314 half-life was 1200 days (approx. 3.4 years). The appropriate regression line (semi-logarithmic plot) is drawn in Appendix III (Graph 1).

4.3.2 Field treated soils

Mean NC 21 314 residue levels found in all samples at each storage interval are shown in Table 4. Individual analyses, alongside apparent control residues and recovery efficiencies for the respective batch of samples, are presented in Appendix II (Table 8).

$A = 1.59720$

$B = -5.62316 \times 10^{-4}$

$r = -0.92745$

$t_{\frac{1}{2}} = 1232$

3.4 yr

Table 4

Summary of NC 21 314 residues in field treated soils

Original trial (Soil type)	Storage interval (Days)	Mean NC 21 314 residue level (mg/kg)		Percentage of initial day 0 residue (%)	
		Rep* I (II)	Rep II (III)	Rep I (II)	Rep II (III)
Shelford, 1980 (sandy loam)	0	0.96	0.51	100	100
	634	0.71	0.36	74	71
	825	0.67	0.35	70	68
Texas, 1980 (clay loam)	0	0.53	0.61	100	100
	402	0.44	0.55	83	90
	555	0.45	0.56	85	92
	758	0.42	0.55	79	90

* Replicate nos. in brackets refer to Texas 1980 soils.

In the Shelford soil (sandy loam), mean residue levels of 0.96 and 0.51 mg/kg extracted from the two replicates at day 0 of the storage study declined to 0.67 and 0.35 mg/kg, respectively, after a further 2.3 years' deep freeze storage (825 days). This was equivalent to between 68 and 70% of the initial day 0 residue remaining stable/extractable after two years, a very similar result to that obtained in the laboratory fortified study using the same soil (74% after two years).

In the Texas soil (clay loam), mean residue levels of 0.53 and 0.61 mg/kg extracted from the two replicates at day 0 declined to 0.42 and 0.55 mg/kg, respectively, after two years deep freeze storage (758 days), 79 and 90%, respectively of the initial day 0 residue remaining extractable. These results would appear to indicate a higher stability of extractable NC 21 314 residues in clay loam soil during deep freeze storage, with between 85 and 92% of initial residues still present after 18 months' storage.

4.4 Archives

All raw data relating to this study and the final report, will be stored in archives by FBC Limited, Chesterford Park Research Station.

Shaughnessy Number: 125501

Date out of EAB: AUG 10 1988

To: Dennis Edwards/Portia Jenkins
Product Manager
Registration Division (TS 767C)

From: Emil Regelman, Supervisory Chemist
Review Section #3
Exposure Assessment Branch
Hazard Evaluation Division (TS 769C)

Thru: Paul F. Schuda, Chief
Exposure Assessment Branch/HED (TS 769C)

Attached, please find the EAB review of...

Reg./File #: 7F3511

Chemical Name: Clofentezine

Type Product: insecticide

Company Name: Nor-Am Chemical Company

Purpose: submission of additional data in response to comments on field dissipation

Date Received: 06/28/88 Action Code: 231

Date Completed: _____ EAB #(s): 80859

Monitoring Study Requested: _____ Total Reviewing Time: 1.5 day

Monitoring Study Volunteered: _____

Deferrals to: ___Ecological Effects Branch

___Residue Chemistry Branch

___Toxicology Branch