

Table I. Average Methyl Phoxim Residues in Parts per Million on Soft Red Winter Wheat Milling Fractions after 10-ppm Application of Methyl Phoxim Emulsion Spray on Wheat^a

days of storage	whole wheat	milling fractions		
		bran	shorts	flour
1	9.86 ± 0.24 ^b	32.78 ± 0.46	18.41 ± 0.36	1.71 ± 0.08
7	8.00 ± 0.12	29.06 ± 0.50	17.73 ± 0.44	2.07 ± 0.06
14	7.34 ± 0.08	24.53 ± 0.40	16.54 ± 0.32	1.79 ± 0.05
21	6.88 ± 0.09	21.04 ± 0.26	13.66 ± 0.12	1.22 ± 0.08
30	5.80 ± 0.07	18.21 ± 0.32	11.80 ± 0.08	0.84 ± 0.05
60	4.50 ± 0.08	13.22 ± 0.18	9.01 ± 0.36	0.76 ± 0.04
90	3.48 ± 0.03	9.52 ± 0.22	7.30 ± 0.22	0.70 ± 0.05
180	2.55 ± 0.06	7.72 ± 0.07	4.60 ± 0.12	0.54 ± 0.04
270	1.43 ± 0.02	5.46 ± 0.08	3.46 ± 0.10	0.31 ± 0.05
365	1.12 ± 0.04	4.26 ± 0.06	2.83 ± 0.08	0.19 ± 0.02
control	0	0	0	0

^a Each number is an average of four replicates.

^b Standard deviation.

rate of methyl phoxim from the pericarp to the endosperm is very slow during the 365 days of storage.

The regression models for methyl phoxim residues in wheat and milling fractions were computed using the stepwise regression procedure. The regression models for each fraction of milled wheat treated at 10 ppm methyl phoxim are:

$$Y = 2.2182 + 0.52602(ID) - 0.941032(DS) + 0.000075(DS)^2$$

$$r^2 = 0.933$$

bran

$$\ln Y = 1.466(\ln OD) - 0.01084(SD) + 0.000016(SD)^2$$

$$r^2 = 0.994$$

shorts

$$\ln Y = 1.75(\ln ID) - 0.218(\ln ID)^2 - 0.011(SD) + 0.000017(SD)^2$$

$$r^2 = 0.996$$

flour

$$\ln Y = 0.6055(\ln ID) + 0.342(\ln ID)^2 - 0.0083(SD) + 0.000008(SD)^2$$

$$r^2 = 0.931$$

where Y = residue, ID = initial dose, and DS = days of storage in all models.

The predicted residual values obtained from the models are very close to the observed values. These regression models are useful in calculating methyl phoxim residues at any time during the 365 days of storage.

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Distribution, Movement, and Dissipation of *N*-Nitrosodipropylamine in Soil

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A field soil experiment was conducted to study the distribution, movement, and dissipation of *N*-nitroso[¹⁴C]dipropylamine (NDPA). In two soils NDPA dissipated to less than 1 and 10% of the initially applied amount after 69 days. No significant leaching occurred beyond a soil depth of 20 cm. A laboratory soil degradation study also demonstrated the dissipation of NDPA. The dissipation is attributed to volatilization of NDPA from soil and degradation to volatile and nonvolatile products.

N-Nitrosodipropylamine (NDPA) has been shown to be a trace contaminant in the herbicide trifluralin (Ross et al., 1977) (TREFLAN, Elanco Products Co.). NDPA can be expected to enter the soil environment through application of the herbicide. Tate and Alexander (1975, 1976)

have previously reported laboratory studies to evaluate the stability of nitrosamines in soil and sewage and the formation of nitrosamines in soils fortified with the corresponding secondary amine and nitrite ion.

Ross et al. (1978) and West and Day (1978) did not detect NDPA in soil treated with trifluralin annually for several years. They concluded there was no buildup of NDPA in soil due to trifluralin application. However, the small amounts of NDPA applied to the soil prevented obtaining direct evidence of NDPA dissipation.

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Figure 1. Sample chromatogram equivalent to 0.93 ng of NDPA.

This report is concerned with the stability of NDPA under laboratory and simulated field conditions. In order to aid detection, a series of experiments was conducted using elevated levels of NDPA to assess its distribution, movement, and dissipation in soil.

EXPERIMENTAL SECTION

Chemicals. [^{14}C]NDPA was obtained from New England Nuclear (Boston, MA). The radiochemical purity was determined to be greater than 98% by thin-layer radioautography. The specific activity was 28.0 $\mu\text{Ci}/\text{mg}$. Unlabeled NDPA analytical standard was synthesized by Lilly Research Laboratories.

Samples containing NDPA were handled with extreme care. Undiluted NDPA was stored in a glovebox equipped with a charcoal filter and maintained under negative pressure. All laboratory work on NDPA samples was conducted in a hood with a minimum draw of 150 linear ft/min. Laboratory workers wore heavy rubber gloves. Waste NDPA solutions were decomposed in a hydrogen bromide-acetic acid solution, sealed in a barrel, and buried in a nuclear waste disposal area.

Soils. The soils were air-dried and passed through a No. 12 mesh sieve prior to use. The Amarillo sandy loam was obtained from southern Texas, had a pH of 7.7, and contained 54.6% sand, 34.2% silt, 11.2% clay, and 0.8% organic matter. The Commerce silty clay loam was obtained near Robinsonville, Mississippi, had a pH of 7.5, and contained 8.4% sand, 62.0% silt, 29.6% clay, and 1.8% organic matter. The soil analysis was conducted by A & L Agricultural Laboratories (Memphis, TN).

Determination of NDPA in Soil. NDPA was extracted from soil by shaking with 75:25 (v/v) methanol/water. An aliquot of the extract was added to 5% aqueous sodium chloride and the NDPA was extracted by partitioning with three 20-mL portions of dichloromethane. The fractions were dried by passage through anhydrous sodium sulfate and combined, and the volume was reduced to 5 mL by rotary evaporation under vacuum. The sample solution was transferred to a 10.0-mL volumetric flask and diluted to volume with dichloromethane, and the NDPA content was determined by gas chromatography.

Gas Chromatography. Gas chromatographic assays for NDPA were conducted on a LKB 9000 (LKB Inc., Bromma, Sweden) gas chromatograph-mass spectrometer (GC-MS) equipped with a 1.8 m by 3 mm i.d. borosilicate glass column packed with 5% Carbowax 20M on 80-100 Chromosorb W-HP. The separator temperature was 150 $^{\circ}\text{C}$, the ion source temperature 290 $^{\circ}\text{C}$, the accelerating voltage 3.5 kV, the trap current 60 μA , and the electron energy 20 eV. The carrier gas was helium at a flow rate of 29 mL/min, the injector temperature was 155 $^{\circ}\text{C}$, and the column temperature was 130 $^{\circ}\text{C}$, resulting in a retention time of 1.8 min. The NDPA was measured by

Table I. Efficiencies of Analytical Methods for NDPA in Soil

experiment	soil type	% efficiencies	av
anaerobic soil dissipation	sandy loam	83.7, 85.0, 73.5	81
	silty clay loam	75.5, 76.2, 68.0	73
laboratory leaching	sandy loam	94.4, 94.6, 96.3, 95.4	95
	silty clay loam	91.7, 90.9, 90.4, 91.0	91
field leaching and soil dissipation	sandy loam	91.5, 99.0	95
	silty clay loam	95.0, 90.5	93

employing a single ion detection method. The magnet was adjusted to the molecular ion (m/e 130) and the ion current was displayed on a strip chart recorder. A sample chromatogram is shown in Figure 1.

Radiochemical Measurements. Radiochemical determinations on solutions were made with a Packard Model 3380 Tri-Carb liquid scintillation counter (Packard Instrument Co., Downers Grove, IL). Aqueous or aqueous-methanol samples were dissolved in a scintillation fluid prepared by dissolving 35 g of PPO (2,5-diphenyl-oxazole), 1.75 g of dimethyl-POPOP [1,4-bis[2-(4-methyl-5-phenyloxazolyl)]benzene], and 278 g of naphthalene in 496 mL of toluene, 1490 mL of dioxane, and 1490 mL of ethylene glycol monomethyl ether. All samples were counted for 5 or 10 min and the counting efficiency was determined by [^{14}C]toluene internal standardization. The radioactivity content of soil samples was determined using a combustion furnace (Lindberg, Type 123-4, Watertown, WI) and trapping the $^{14}\text{CO}_2$ in 30:70 (v/v) ethanolamine/ethylene glycol monomethyl ether solution for liquid scintillation counting (LSC).

Thin-Layer Chromatography (TLC). TLC was conducted on Brinkman silica gel plates using hexane/ethyl ether/dichloromethane (40:30:25, v/v/v) as the developing solvent. Radioactivity was detected by radioautography.

Method Efficiencies. The efficiency of the analytical method for NDPA in soil was determined by assay of samples containing known amounts of NDPA before each study. The average method efficiencies used for correcting assay results are presented in Table I.

Laboratory Soil Leaching. The leaching columns were 45-cm sections of 1.0 cm i.d. thin-walled glass tubing mounted in a vertical position. A plug of glass wool was placed in the bottom of the column to retain the soil. Dry soil was added to each column, with gentle tapping, to a height of 30 cm. The sandy loam soil columns required 33 g of soil, while the silty clay loam columns required 28 g. Three columns of each soil type were prepared. One served as a blank and two were treatment replicates.

Each column was treated by pipetting 1.0 mL of water containing 6.50 μg (0.18 μCi) of NDPA onto the top of the column. The columns were leached with the equivalent of 10 cm of rainfall each day for 6 days. The first portion served to wet the column and no leachate was obtained. The next five leachate portions were collected separately and assayed radiochemically for NDPA.

After leaching, the soil columns were divided by breaking the tubes into six 5-cm sections. Each soil section was assayed for methanol-extractable radioactivity.

Anaerobic Soil Dissipation. Ten-gram soil samples were weighed into 25-mL glass screw-cap vials. Two hours prior to treatment with NDPA, the vials were uncapped and placed in a glass desiccator, and the desiccator was purged with carbon dioxide to remove oxygen.

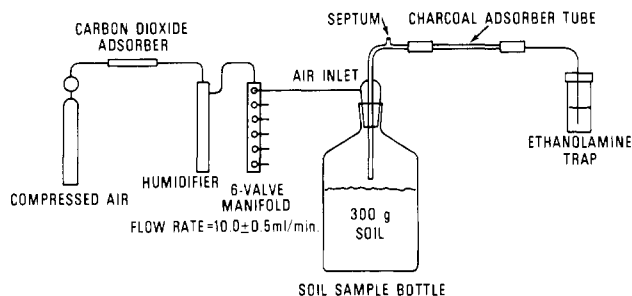


Figure 2. Air flow system for conducting aerobic soil dissipation studies.

The vials remained under the carbon dioxide atmosphere while each was treated with $10.0 \mu\text{g}$ of NDPA ($0.0065 \mu\text{Ci}$) contained in 5.0 mL of deaerated water. The vials were capped immediately and incubated in the dark at 25°C until assay.

Two vials of each soil type were collected at 0, 2, 5, 8, 16, 30, and 60 days after initiation of the experiment and analyzed for total extractable radioactivity and NDPA.

Aerobic Soil Dissipation. The air flow system, soil container, and trapping system are shown in Figure 2. Compressed air was passed through a tube containing Mallcosorb (Mallinckrodt, St. Louis, MO) to remove carbon dioxide and bubbled through water to saturate the air. The air flow rate was maintained at $10.0 \pm 0.5 \text{ mL/min}$ by means of a needle valve. The air stream was passed over the surface of the soil and through two sequential traps containing activated charcoal and ethanolamine, respectively. The charcoal tube was a $3.0 \text{ mm i.d.} \times 10 \text{ cm}$ glass tube containing two sections (200 mg front, 50 mg rear) of "Darco" 20–40 mesh activated charcoal (MC/B, Norwood, OH). Volatile components not trapped on charcoal were passed into a 20-mL liquid scintillation vial containing 10.0 mL of 30:70 ethanolamine/ethylene glycol monoethyl ether.

Two weeks prior to initiation of the experiment, 300-g soil samples were placed in each glass bottle and were treated with 30 mL of water. The soil samples requiring sterilization were autoclaved at 121°C for 16 h. The samples were connected to the flow system, and moist air was passed over the soil. This initial period allowed the soil organisms to reestablish the populations normally found in moist soil.

After this initial period, the soil samples were treated with $10.2 \mu\text{g}$ of NDPA ($0.28 \mu\text{Ci}$) contained in 30 mL of water and immediately reconnected to the flow system. Control soils were treated with 30 mL of water only. The study was conducted in the dark at 22°C and continued for 40 days. At appropriate times, the trapping media were replaced and the used traps immediately assayed for NDPA and/or radioactivity.

NDPA was extracted from the charcoal with dioxane and the NDPA content of the extract was determined by gas chromatography. The radiochemical content of the dioxane extract and the ethanolamine trapping solution was determined by LSC.

In order to verify that charcoal was an effective adsorbent for trapping nitrosamines (Bretschneider and Matz, 1974), the efficiency of adsorption and desorption of NDPA from charcoal samples by dioxane was determined in a separate experiment. A water solution containing NDPA was injected downstream from the soil sample and upstream from the charcoal. After 24 h, the charcoal traps were removed and assayed for radioactivity. The results indicated quantitative trapping of NDPA in the front section and no NDPA in the back section.

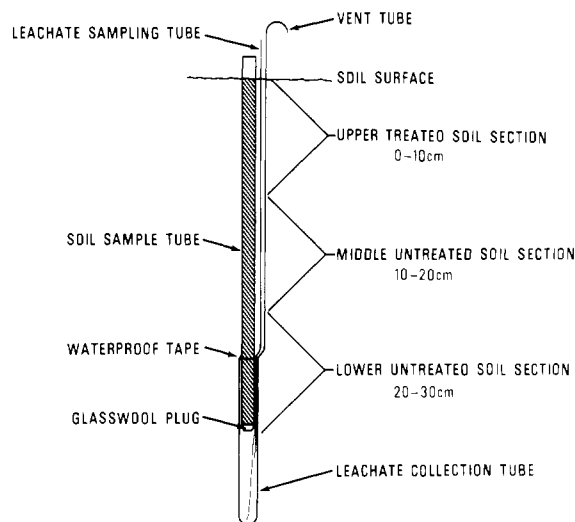


Figure 3. Soil column and leachate collection reservoir for field soil dissipation study.

Field Leaching and Soil Dissipation. The soil sample tube consisted of a $10.0 \text{ mm i.d.} \times 32 \text{ cm}$ section of thin-walled glass tubing partially closed at the bottom end to retain a plug of glass wool. The leachate collection reservoir consisted of a $16.0 \text{ mm i.d.} \times 14 \text{ cm}$ test tube. The vent tube and leachate sample collection tube consisted of 45-cm sections of 2.0 mm i.d. Teflon tubing. The components were assembled as shown in Figure 3 and secured with waterproof tape. The sample tube was filled with a portion of soil equivalent to two-thirds that required for a complete fill. Water (5.0 mL) was added to the column to thoroughly wet the soil.

Portions of soil equivalent to one-third the amount required to fill a soil column were placed in 25-mL screw-cap vials. The soil samples were treated with $10.8 \mu\text{g}$ of NDPA ($0.30 \mu\text{Ci}$) contained in 0.25 mL of acetone. The solvent was allowed to evaporate 1 min, and the samples were capped and thoroughly mixed.

The columns were buried in a level, open field at Greenfield, IN, in a vertical position with only the top 2 cm of the column protruding above the ground. The treated soil was added to complete the filling of the columns to a 30-cm depth.

Leachate samples were collected by syringe as necessary. Soil tubes were collected by completely removing them from the ground at intervals of 0, 15, 24, 69, 111, and 124 days from initiation of the experiment. Two tubes of each soil type were collected and assayed separately. The tubes were divided into three 10-cm sections for assay.

RESULTS

Laboratory Leaching. The data from this experiment are shown in Figure 4 and represent the average of two leaching columns for each soil. More than 88 and 94% of the NDPA was found in the soil leachates after the equivalent of 60-cm simulated rainfall was passed through the silty clay loam and sandy loam soil columns, respectively. NDPA appeared to leach more readily in the sandy loam soil than the silty clay loam.

At the end of the experiment, only 3.1 and 6.0% of the applied radioactivity remained on the silty clay loam and sandy loam soil columns, respectively.

Anaerobic Soil Dissipation. The data in Figure 5 indicated that under anaerobic conditions, NDPA degraded slowly in sandy loam with 77% of the initially applied NDPA remaining after 60 days. The dissipation of NDPA in silty clay loam was faster with a half-life of approximately 47 days. The radiochemical data were

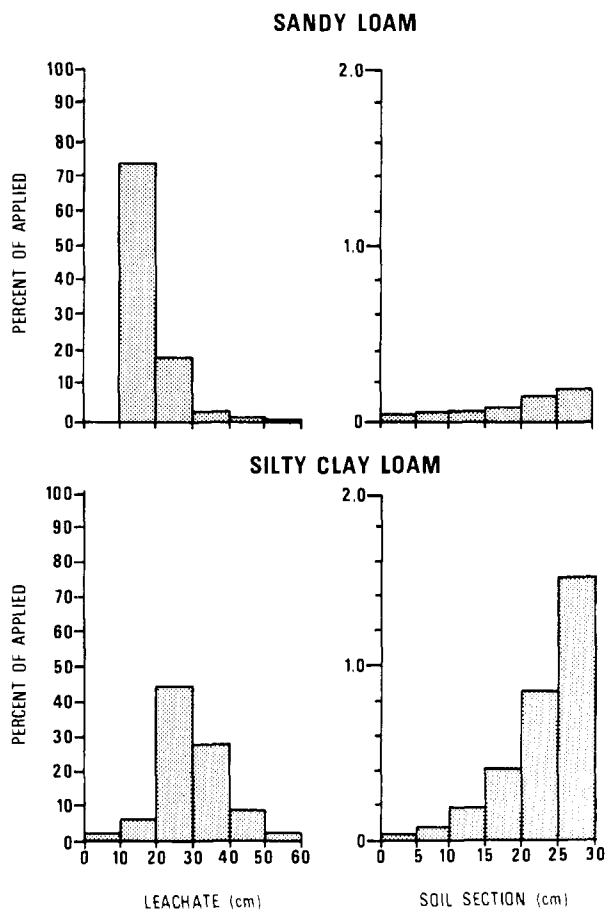


Figure 4. Distribution of radioactivity in leachate fractions and soil sections from laboratory soil leaching study.

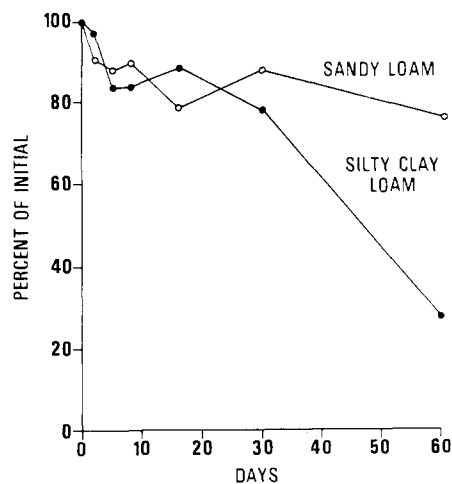


Figure 5. NDPA content of treated soil incubated under anaerobic conditions.

identical with the NDPA assay results and are not shown.

Aerobic Soil Dissipation. The total amounts of NDPA and radioactivity in the charcoal and ethanolamine traps are plotted as a function of time as shown in Figure 6 for the two soils. Charcoal has been shown to be an efficient medium for trapping volatile nitrosamines (Bretschneider and Matz, 1974) and no significant radioactivity was found in the back sections of the charcoal traps. The loss of radioactivity from the soil can be determined by subtracting the radioactivity collected by the two traps as shown in Figure 6. More than 50% of the initially applied radioactivity dissipated from the two soils in 15 days.

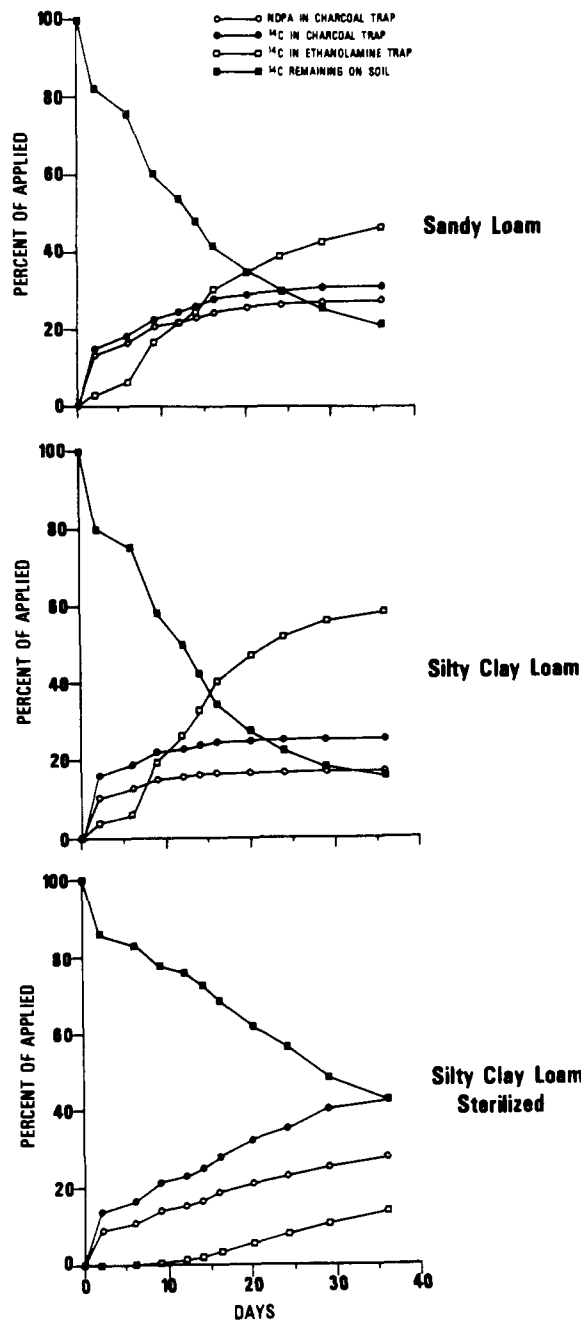


Figure 6. NDPA and radioactivity in charcoal and ethanolamine traps from aerobic soil study.

Most of the initial radioactivity was found in the charcoal trap as a result of volatilization of NDPA from the soil. Volatile degradation products may have also collected on the charcoal since NDPA did not account for all the radioactivity found on the charcoal. After 5 days, the majority of the radioactivity given off from the soil was collected in the ethanolamine trap. Ethanolamine is commonly used for trapping carbon dioxide; however, this does not positively indicate that the radioactivity trapped by the ethanolamine was $^{14}\text{CO}_2$. Oliver et al. (1978) have shown that the rate of radioactivity given off from soil containing radiolabeled NDPA is independent of whether the ^{14}C is in the 1, 2, or 3 position, suggesting that the compound degrades completely in CO_2 . Since NDPA is efficiently trapped by charcoal, the radioactivity in the ethanolamine is presumably due to volatile soil degradation products of NDPA that do not adsorb onto charcoal. The distribution of radioactivity in the traps and soils at the

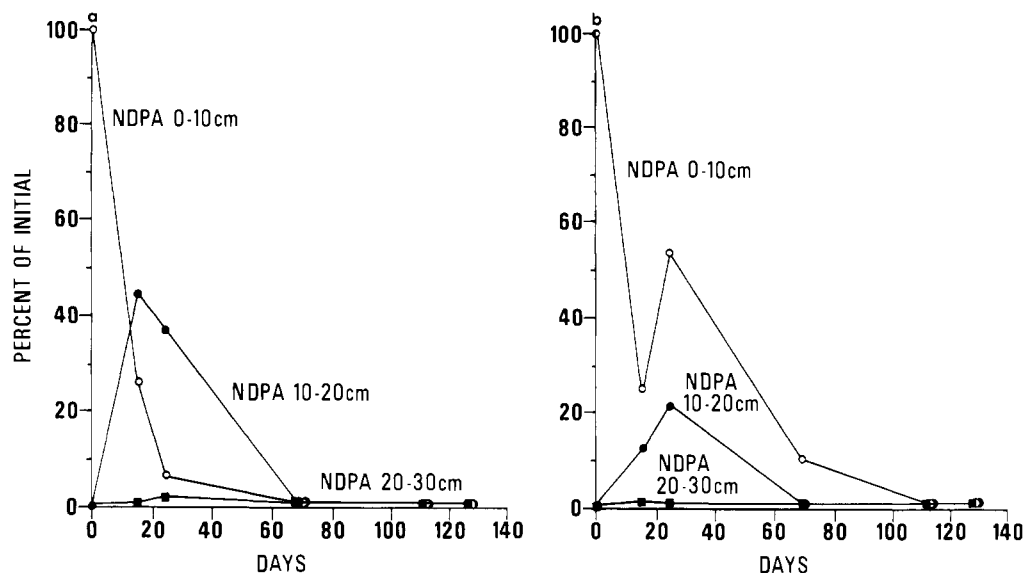


Figure 7. Distribution of NDPA in soil column sections from the field leaching and soil dissipation study: (a) sandy loam soil, (b) silty clay loam soil.

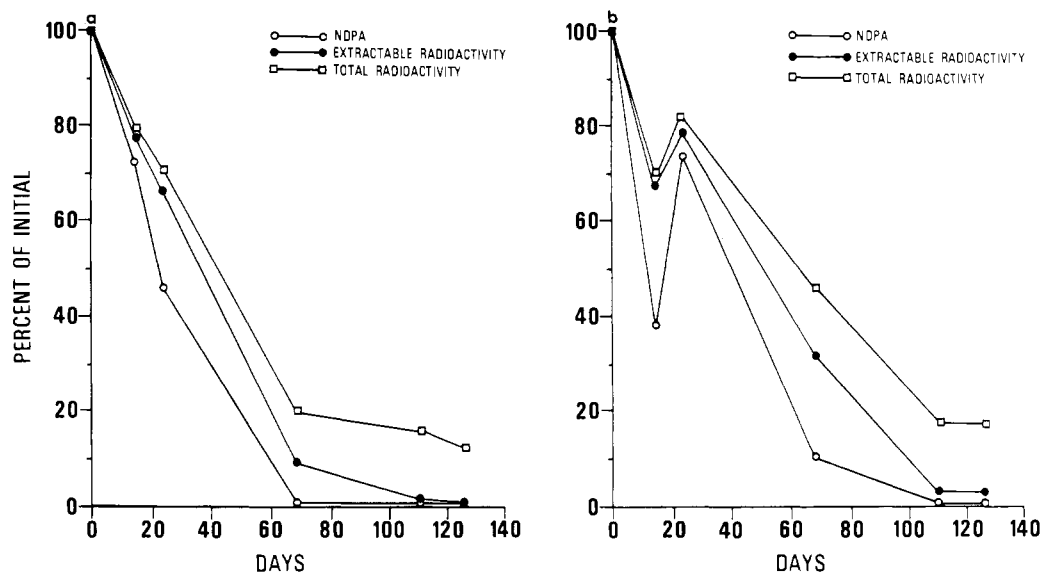


Figure 8. Dissipation of NDPA and radioactivity from the field leaching and soil dissipation study: (a) sandy loam soil, (b) silty clay loam soil.

end of the experiment is shown in Table II. Most of the extractable radioactivity from the soil at the end of the experiment was found to be NDPA.

Autoclaved Soil. Similar data were obtained when the experiment was conducted using autoclaved silty clay loam soil as shown in Figure 6. During the first 10 days of the experiment, almost all the radioactivity given off from the soil was collected on the charcoal. Some of the radioactivity on the charcoal resulted from volatilization of NDPA from the soil, while a significant amount of radioactivity was attributed to volatile degradation products of NDPA which were trapped on the charcoal. The amount of radioactivity collected in the ethanolamine trap increased after the first 10 days. The soil sterilization procedure appeared to inhibit, but not prevent, the degradation of NDPA. No attempt was made to demonstrate the sterility of the soil or to maintain sterility during the study. The distribution of radioactivity in the soil at the end of the experiment is shown in Table II with most of the extractable radioactivity attributed to NDPA. Thin-layer chromatography-radioautography of the soil extracts

Table II. Distribution of Radioactivity at the End of the NDPA Aerobic Soil Dissipation Study

	% of initial application		
	sandy loam	silty clay loam	silty clay loam, auto-claved
charcoal trap	31.4	25.4	43.0
ethanolamine trap	47.1	58.7	14.0
methanol extract	6.3	2.9	45.7
nonextractable	17.1	22.8	7.8
totals	102.0	109.8	110.5

showed only NDPA, and no significant degradation products were found.

Field Dissipation. Rainfall data were collected during this study and are shown in Table III. The distribution and dissipation of NDPA in the three column fractions are shown in Figure 7a,b for the two soils. The NDPA in the 0-10-cm column fraction dissipated rapidly. Some of the NDPA leached into the 10-20-cm column fraction where

Table III. Rainfall during NDPA Field Soil Column Dissipation Experiment

sam- pling no.	date sample collected	total days from initia- tion of experi- ment	rain- fall since last sam- pling period, cm	cum- ulative rain- fall, cm
1	4/12/77	0		0
2	4/27/77	15	4.4	4.4
3	5/06/77	24	2.7	7.1
4	6/20/77	69	6.1	13.2
5	8/01/77	111	20.6	33.8
6	8/16/77	126	6.6	40.4

dissipation of NDPA also occurred rapidly. Little or no radioactivity was observed in the 20–30-cm column fraction and the soil leachates. The disappearance of NDPA and radioactivity from the soil is shown in Figure 8a,b with half the initially applied NDPA dissipated in approximately 21 and 40 days for the sandy loam and silty clay loam soils, respectively.

DISCUSSION

Leaching of NDPA. The laboratory leaching study showed that the compound will leach under rather heavy simulated rainfall conditions, which is consistent with Dean-Raymond and Alexander (1976) who reported on the leachability of dimethylnitrosamine (DMNA). The field study indicated that NDPA did not leach below 20 cm. This suggests that NDPA dissipated readily before extensive leaching occurred and that the high water solubility (approximately 10000 ppm) of NDPA (Mirvish et al., 1976) caused it to move with the soil moisture. During periods of rain, the compound was leached downward, but during periods of drying it may have returned to the soil surface as the water evaporated (Spencer and Cliath, 1973). Some NDPA was observed in the 10–20-cm soil section but the

compound was mixed initially in the 0–10-cm section. The observed differences in leaching between laboratory and field studies demonstrate the value of conducting soil experiments under simulated field conditions whenever possible.

Dissipation of NDPA in Soil. Half-lives of approximately 21 and 40 days for NDPA in sandy loam and silty clay loam soil, respectively, were found in the field dissipation study. Tate and Alexander (1975) reported that NDPA was resistant to microbiological degradation; however, their data for NDPA in Williamson silt loam indicated that half of the initially applied NDPA disappeared in 20–30 days. The aerobic laboratory soil study indicated a half-life of approximately 10–15 days for NDPA in both soils. Oliver et al. (1978) reported a half-life of 2–3 weeks for NDPA in active soils in the laboratory.

These studies demonstrate that the trace quantities of NDPA which have resulted from the application of trifluralin can dissipate from the soil types used in this study.

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Dislodgable Residue of Supracide on Citrus Leaves

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Dislodgable residues of *S*-(2-methoxy-5-oxo- Δ^2 -1,3,4-thiadiazolin-4-yl)methyl] *O,O*-dimethyl phosphorodithioate (Supracide) and a monoxone metabolite were analyzed on citrus leaves at various intervals following application during a dry season (May, 1974) and a wet season (July–August, 1974). Applications were made at one, two, and three times the recommended rate and during the wet season additional applications were made which included oil. Leaves were collected from which leaf disks were punched for analysis. Disks were water/detergent washed for removal of dislodgable residues. Supracide was analyzed by FPD/GC and the monoxone metabolite was quantified by thin-layer chromatography using fly-head cholinesterase. There was a rapid decrease of Supracide and the monoxone metabolite during the first week after application. In those samples in which total residues were checked, total residue paralleled dislodgable residues.

The dislodgable residues of chemicals sprayed in agricultural fields and groves are of high importance with

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respect to field-worker reentry. This is particularly true of organophosphorus compounds with high mammalian toxicity. Spear et al. (1975) have reported work dealing with worker hazard in California citrus treated with parathion. They found the paraoxon accounted for a significant portion of the residue found and that it decayed more slowly than parathion. Dislodgable residue studies by this research group (Leffingwell et al., 1975) have also concerned Ethion and zolone on grape foliage. Westlake