Factors Affecting Vapor Loss of Trifluralin from Soil

William F. Spencer* and Mark M. Cliath

Trifluralin vapor pressure increased approximately five times for each 10-degree increase in temperature between 20 and 40°. Vapor density in Gila silt loam increased as trifluralin concentration increased and reached a saturated vapor density equal to that of trifluralin without soil at approximately 73 μg/g when the soil water content was 19%. Vapor density was markedly less in drier soils. Vapor density was inversely related to soil organic matter content. Volatilization rates measured by trapping vaporized trifluralin indicated that trifluralin vaporized more rapidly when surface applied than when incorporated into the soil. For example, when 14 kg/ha was incorporated into 10 cm of soil, the loss rate during the first 24 hr was only 0.0517 kg/ha per day. Surface applications of 1–10 kg/ha to a wet soil surface resulted in loss rates up to 4.0 kg/ha per day. Application to a dry soil surface resulted in essentially no volatilization.

Trifluralin (α,α,α-trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine), a selective preemergence herbicide, is used extensively throughout the United States to control a wide variety of grasses and broadleaf weeds. Its effectiveness is greatly increased by incorporation into the soil because of its tendency to volatilize or to decompose photochemically (Pieczarka et al., 1962; Savage and Barrentine, 1969; Fenster et al., 1971; Fink, 1972; Standifer and Thomas, 1965). The chemical and physical properties and degradation pathways for trifluralin were discussed by Probst et al. (1967), Probst and Tepe (1969), and Parr and Smith (1973). Trifluralin is subject to rapid photodecomposition in the presence of ultraviolet light (Probst and Tepe, 1969; Wright and Warren, 1965; Harrison and Anderson, 1970). Standifer and Thomas (1965) found that trifluralin is generally most herbicidally effective in dry soil but tends to desorb in wet soil. Bardsley et al. (1967) found that the toxicity and persistence of trifluralin were related to the organic matter content of the soil. Hollist and Foy (1971) reported that organic matter effectively reduced phytotoxicity and that trifluralin did not adsorb on the internal surfaces of montmorillonite. They also reported that increased soil water contents appeared to block the active sites for trifluralin adsorption on montmorillonite.

Bardsley et al. (1968), Ketchersid et al. (1969), Parochetti and Hein (1973), and Parr and Smith (1973) reported that volatilization was a significant pathway for loss of trifluralin from soil. Bardsley et al. (1968) used a charcoal-coated beaker inverted over the treated surface as a vapor trap to evaluate trifluralin losses from soils. They found that vaporization losses were related to concentration, time, mode of application, and soil water content. Ketchersid et al. (1969), who estimated volatilization of trifluralin by analyzing air samples above the treated soil, reported that volatilization was influenced more by soil water content than by soil types.

This paper reports some effects of temperature, trifluralin concentration, soil water content, soil organic matter, and soil incorporation on vapor density and volatilization rate of trifluralin from soil.

EXPERIMENTAL SECTION

Vapor Density and Desorption Isotherms for Trifluralin. Vapor density or apparent vapor pressure of trifluralin, with and without soil, was determined by a gas saturation method in which N_2 gas was passed through trifluralin-treated sand or soil slowly enough to ensure equi-

librium vapor saturation. The apparatus and procedures were similar to those previously described (Spencer and Cliath, 1969, 1970a, 1972). For vapor pressure without soil, 4 g of technical grade trifluralin (99.5% pure) in 50 ml of hexane was added to 1100 g of silica sand with an atomizer. After evaporating the hexane and placing the sand in 6 × 43 cm glass columns, dry N_2 gas was passed through the columns at flow rates of approximately 3–5 ml/min until sufficient trifluralin had been trapped in hexane for analysis by glc. The amount of trifluralin in the hexane was quantified by peak heights, using a Beckman GC-5 gas–liquid chromatograph with a nonradioactive electron-capture detector. Gas chromatographic conditions were as follows: glass column (1.83 m × 3 mm i.d.) packed with 2% DC-200 (12,500 cSt) on Anakrom ABS (80–90 mesh); helium carrier gas-flow rate, 50 ml/min; injection port temperature, 200°; column temperature, 180°; detector temperature, 250°.

For vapor density in soil, the effects of trifluralin concentration and soil water content were studied when various rates of trifluralin were added to Gila silt loam. To determine soil organic matter effects, vapor density of trifluralin was measured at 10 μg/g of trifluralin in Imperial clay, Gila silt loam, and Kentwood sandy loam with the soil water contents of all soils adjusted to the soil water tension (approximately 400 millibars), equivalent to 19% water in Gila silt loam. Imperial clay, Gila silt loam, and Kentwood sandy loam contain 0.20, 0.58, and 1.62% organic matter and 68, 18.4, and 10.0% clay, respectively.

Soil columns were prepared in the same way as for previous studies (Spencer and Cliath, 1969, 1970b, 1972). To 1100 g of autoclaved soil, technical grade trifluralin was applied by spray atomization in hexane to provide soil concentrations from approximately 2.5 to 80 μg/g. After evaporating the hexane, the soil water content was adjusted to various levels by adding measured amounts of water with an atomizer for soil water contents of 6% or less and by adding ice at below-freezing temperatures for higher water contents. Vapor density measurements were initiated approximately 24 hr after water additions. Trifluralin and water contents of soil samples were determined at the start and the end of each series of vapor density runs on individual soil columns.

Volatilization of Trifluralin from Soil. Volatilization rates were measured in a vapor trapping system by passing N_2 gas over trifluralin-treated Gila silt loam at 30°. The apparatus and procedures have been described (Spencer and Cliath, 1973). The apparatus included a 10-cm long soil column in which water could be supplied through a glass frit at the bottom of the column. The relative humidity of N_2 passing over the soil surface was varied to control water loss rates. The flow rate of 800 ml/

min resulted in a change of atmosphere in the head space over the soil about once every 3 sec. The soil column was 4.78 cm in diameter and the head space above the soil was 36 ml; consequently, the average linear speed of the air over the soil surface was 0.017 cm/sec (0.039 mile/hr). This rate of air exchange is probably less than that encountered under most field conditions. The volatilization rate of soil-incorporated trifluralin was measured in the absence and presence of evaporating water to evaluate the effect of water loss on volatilization rate. The volatilization rate of surface-applied trifluralin was measured as affected by application rate, soil water content, and water loss rate as related to relative humidity of the gas stream.

For volatilization rate of soil-incorporated trifluralin, technical grade trifluralin was applied in hexane to autoclaved Gila silt loam at the rate of 10 μg/g. The hexane was evaporated and the treated soil packed in the glass column as described by Spencer and Cliath (1973). With the soil packed to a bulk density of 1.4 g/cm^3 in the 10-cm column, 10 μg of trifluralin/g of soil was equivalent to 14 kg/ha. The soil water content was approximately 30.5% near the surface during the period of measurement. Volatilization was measured over a 48-day period with the soil surface moist and the relative humidity of the N$_2$ gas adjusted at 100% for the first 6 days, then adjusted to 75, 100, 50, or 0% during subsequent periods. The volatilized trifluralin was trapped in either hexane or ethylene glycol, depending on the length of the measurement period.

For the surface applications, the glass column was filled with Gila silt loam and was wetted from the bottom at a water tension of 50 millibars, or approximately 30.5% soil water content. Trifluralin at 1, 2, or 10 kg/ha was applied dropwise in 0.1 ml of hexane to the wet soil surface using a tlc syringe. A pantograph template controlled positioning and placement of the drops on the soil surface. Since the soil columns were placed in a constant-temperature incubator at 30° before adding trifluralin, volatilization measurements began immediately after the trifluralin was added. The intermediate 2-kg/ha rate was used also to compare volatilization at 100 and 50% relative humidity and to measure volatilization from an air-dry soil surface. Since volatilization rates were very low from the air-dry soil surface, one soil column was wetted from the bottom after 8 or 9 hr and volatilization was measured after the delayed wetting of the soil surface. The trifluralin and water contents of the soil columns were measured after each volatilization test was completed.

Vapor density and volatilization rates were measured in dark, constant-temperature incubators, and all other procedures were carried out under weak incandescent light to prevent photodecomposition of trifluralin by fluorescent lamps.

RESULTS AND DISCUSSION

Vapor Density and Desorption Isotherms for Trifluralin. The vapor density and apparent vapor pressure of trifluralin at three temperatures are shown in Table I. The apparent vapor pressure was linearly related to temperature by the equation log P = 17.318 − 6344/T with ΔH_v (heat of vaporization) = 29 kcal/mol. Probst et al. (1967) reported that the vapor pressure of trifluralin was 1.99 × 10^{-4} mm at 29.5°. The data in Table I can be used to calculate vapor pressures at other temperatures encountered under field conditions.

The effect of concentration on vapor density of trifluralin in Gila silt loam at 6 and 19% soil water content at 30° is shown in Figure 1. No differences in trifluralin concentrations or soil water contents were measurable between the start and end of a series of vapor density runs. However, the trifluralin concentrations and soil water contents were slightly lower than the planned application rates because of losses during the atomizer spray application. The

Table I. Vapor Density and Apparent Vapor Pressure of Trifluralin at Different Temperatures

Temp, °C	Vapor density, μg/l.	Vapor pressure,[a] mm × 10^{-4}
20	0.889 ± 0.016	0.485 ± 0.009
30	4.29 ± 0.09	2.42 ± 0.05
40	19.94 ± 0.30	11.59 ± 0.17

[a] Calculated from vapor density (d) with the equation $P = d(RT/M)$, where P is the vapor pressure, R is the molar gas constant, T is the absolute temperature, and M is the molecular weight.
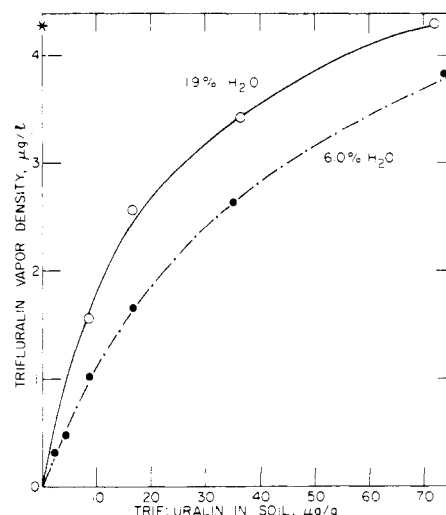

Figure 1. Vapor density of trifluralin as related to concentration in Gila silt loam at 6 and 19% soil water contents and 30°. The asterisk indicates vapor density of trifluralin without soil.

data reported here are based on the trifluralin concentrations and soil water contents measured rather than the amounts supposedly applied. Vapor density increased rapidly as trifluralin concentration increased, and at 19% water a saturated vapor density equal to that of trifluralin without soil was reached at approximately 73 μg/g. For a saturated vapor at 6% water, the projected trifluralin concentration needed was approximately 95 μg/g. Obviously, the volatility of trifluralin would be greater at 19% than at 6% soil water content. For example, at 10 μg/g the vapor density is 50% greater at 19% than at 6% soil water content.

Figure 2 shows the effect of a wider range of soil water contents on trifluralin vapor density at two trifluralin concentrations. Vapor density was markedly reduced in the drier soil at both trifluralin soil concentrations (8.4 and 72 μg/g), but the vapor density began to decrease at a higher soil water percentage at 8.4 than at 72 μg/g. When the soil water content reached 6%, the vapor density at both trifluralin concentrations was significantly reduced. Air drying the Gila silt loam to approximately 2% water content reduced the vapor density to extremely low values. For example, at soil trifluralin concentrations of 2.1–35 μg/g, the vapor density in air-dry soil ranged only from 0.36 to 0.65 ng/l. At 19% soil water content, the trifluralin vapor density, or potential volatility, was 3000–5000 times greater than when the soil was air dry. As the soil water content was reduced, trifluralin vapor density began to decrease at a higher soil water content than was observed for dieldrin or lindane. Vapor densities of dieldrin at 100 μg/g (Spencer et al., 1969) or lindane at 10 μg/g (Spencer and Cliath, 1970b) in Gila silt loam did not decrease until the soil water content was reduced below approximately
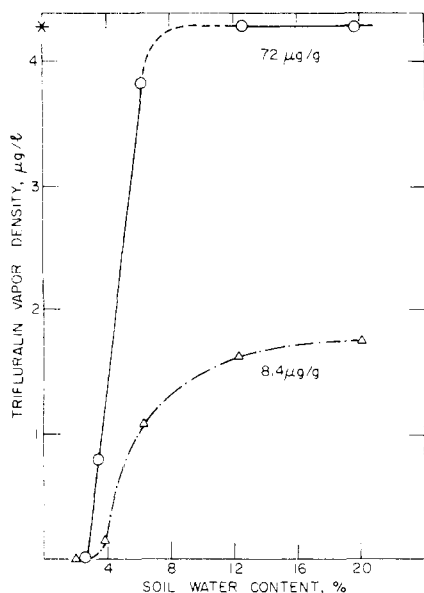
Figure 2. Effect of soil water content on vapor density of trifluralin in Gila silt loam at 8.4 and 72 μg/g of trifluralin and 30°.

Figure 3. Volatilization rate of soil-incorporated trifluralin as related to time and relative humidity of the N_2 gas passing over the soil surface at 30° with 10 μg/g of trifluralin mixed in Gila silt loam at 50 millibars of water suction (expanded scale first day only).

one molecular layer of water equivalent to approximately 2.7% water in Gila silt loam.

Vapor densities at 10 μg/g of trifluralin in three soils varying in organic matter (O.M.) content were 3.19, 1.73, and 0.62 μg/l. in Imperial clay (0.20% O.M.), Gila silt loam (0.58% O.M.), and Kentwood sandy loam (1.62% O.M.), respectively. Thus, vapor density decreased as O.M. content increased. Since the clay content was inversely related to the O.M. content of the three soils, these results indicate that clay plays a minor role in trifluralin adsorption in wet soil. Evidently, trifluralin adsorption, consequently vapor density or potential volatility, is a function of the O.M. content of the soil, at least in wet soils. Spencer (1970) reported that vapor density of dieldrin was inversely related to the O.M. content of the same three soils.

The decreasing vapor density of trifluralin with increasing soil O.M. content is consistent with data reported by others. Weise *et al.* (1971) reported that the toxicity and persistence of trifluralin in four soils were related to the O.M. content of the soils. Segraves *et al.* (1973) found that in 21 soils the total carbon content of the soil was highly associated with trifluralin activity—91% of the variability in response to rate of trifluralin could be accounted for by total carbon content of the soil, whereas only 55% of the variability was associated with clay content.

Volatilization of Trifluralin from Soil. The volatilization rate of soil-incorporated trifluralin as related to time and water loss rate over a 48-day period is shown in Figure 3. The maximum volatilization rate during the first 15 min was 4.19 μg/cm² per day. This dropped to less than 0.30 μg/cm² per day within 24 hr. The total loss for the first 24 hr was 0.517 μg/cm² (0.0517 kg/ha) or 0.36% of that applied. The total loss by volatilization during the 48-day period was 3.42 μg/cm² (0.342 kg/ha) or only 2.45% of that applied. The rapid decrease in volatilization rate of soil-incorporated pesticides with time was also observed with lindane and dieldrin (Spencer and Cliath, 1973). The decreased volatilization of soil-incorporated trifluralin was caused by depletion of trifluralin at the soil surface and not by anaerobic degradation of trifluralin in the soil as reported by Parr and Smith (1973). The Gila silt loam was autoclaved which effectively retarded microbial degradation during the test period.
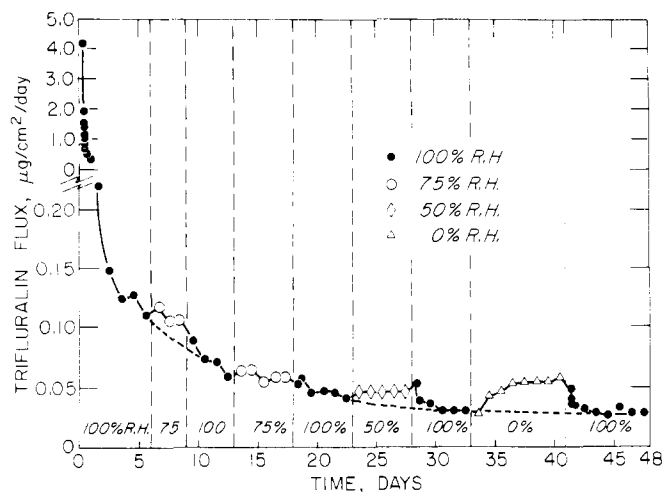
Volatilization rates were somewhat higher at the lower relative humidities, indicating that evaporating water slightly increased the volatilization rate of trifluralin by the wick effect of transporting trifluralin to the surface in evaporating water. This was most apparent at 50 and 0% relative humidity when water was evaporating at the higher rates, compared to no water loss at 100% relative humidity. The effect of evaporating water on the loss of trifluralin was not nearly as great as was observed with lindane (Spencer and Cliath, 1973). At 100% relative humidity, the trifluralin volatilization rate leveled off at flux values approaching 0.025 μg/cm² per day (0.0025 kg/ha per day). When a period of 100% relative humidity followed a drying period at 0 or 50% relative humidity, the volatilization rate of trifluralin did not increase as did that of dieldrin (Spencer and Cliath, 1973). This indicates that trifluralin volatilized from the surface at approximately the same rate as it moved upward in the evaporating water and, unlike dieldrin, did not accumulate at the surface during drying.

Trifluralin volatilized much more rapidly when applied on the soil surface than when mixed into the soil. Figure 4 shows volatilization of trifluralin applied on the wet surface of Gila silt loam at the rate of 1, 2, and 10 kg/ha with the N_2 passing over the soil surface at 100% relative humidity. Initially, volatilization was almost the same for all three application rates, but volatilization for the 1- and 2-kg/ha applications decreased much more rapidly than for 10 kg/ha with time due to more rapid surface depletion. The maximum volatilization rate from each surface treatment was approximately 40 μg/cm² per day (4 kg/ha per day) compared with a volatilization rate of 0.0517 kg/ha per day for the first 24 hr after soil incorporation. With surface applications, the volatilization rate during the initial period of measurement (either 15 or 30 min) was lower than that measured during the second or third volatilization period. This was probably caused by a reduced trifluralin vapor pressure in the presence of hexane before its complete evaporation during the first few minutes of measurement.

Figure 5 shows volatilization rates of surface-applied trifluralin at 2 kg/ha when applied (a) to a wet soil with the N_2 gas at 100% relative humidity, (b) to a wet soil with the N_2 at 50% relative humidity, and (c) to an air-dry soil which was wetted after 8 to 9 hr of volatilization. With the wet soil surface, passing 50 and 100% relative humidity air over the soil resulted in no significant differ-
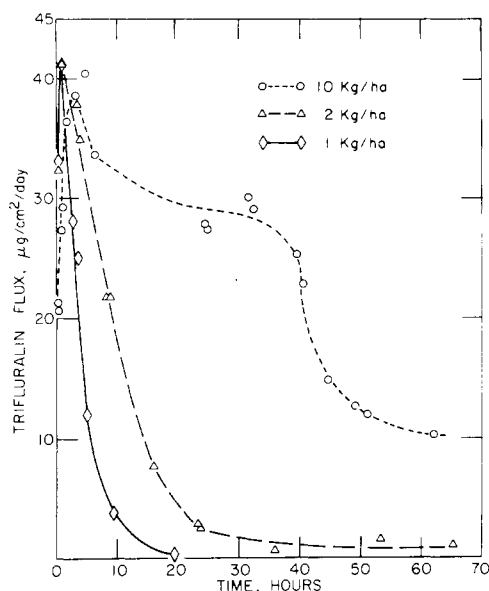
Figure 4. Volatilization of trifluralin at 30° and 100% relative humidity applied on a wet soil surface of Gila silt loam at 10, 2, and 1 kg/ha.
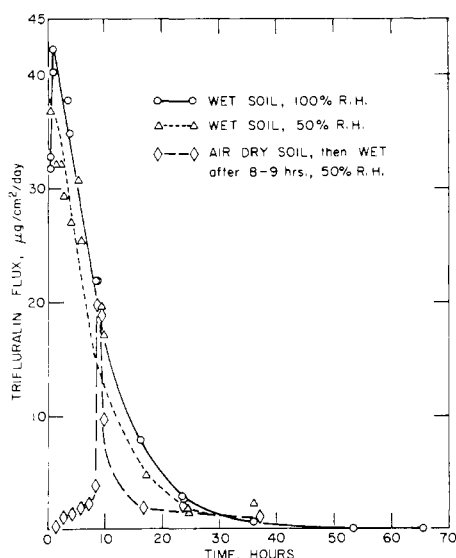

Figure 5. Volatilization rates of surface-applied trifluralin at 2 kg/ha as related to relative humidity of the N_2 gas and the water content of the soil surface.

ralin applied to wet soils were essentially vaporized in less than 24 hr. The rapid vapor loss from surface applications to moist soils indicates that trifluralin must be incorporated immediately to prevent excessive loss. However, when applied to a dry soil surface, volatilization is extremely low and incorporation probably can be delayed until a more convenient time. Trifluralin concentration and soil water content effects on vapor density and volatilization are consistent with data on trifluralin activity and dissipation rates reported by other investigators (Standifer and Thomas, 1965; Bardsley et al., 1967, 1968; Hollist and Foy, 1971).

The high volatilization rates from surface applications indicate that vapor loss is the major reason for the lack of herbicidal activity from surface applications, but does not rule out the possibility of photochemical decomposition being a significant pathway for loss of surface-applied trifluralin under some conditions. Ultraviolet light from sunlight probably decomposes trifluralin on the soil surface, as well as in the vapor phase following volatilization.

The exact relation between volatilization rates measured in laboratory columns and those expected in the field is unknown. In the field, volatilization rates from surface applications probably would be higher than those reported herein because of higher rates of air exchange under most field conditions. The gas over the soil columns was exchanged, on the average, once every 3 sec which is equivalent to a linear flow rate of only 0.039 mile/hr. This is relatively low compared to windspeeds encountered in most agricultural areas. Consequently, for a wet soil, the maximum volatilization rate of surface-applied trifluralin (approximately 4 kg/ha per day) is probably below the maximum rates that would occur under many field conditions. However, volatilization rates of soil-incorporated trifluralin in the field probably would be similar to those reported here since the rate of loss would be controlled more by the rate of trifluralin movement to the soil surface than by the rate of air exchange over the soil surface.

High volatilization rates, such as those observed from surface applications of trifluralin, undoubtedly occur with most pesticides, but at differing rates depending on their vapor pressures (Spencer et al., 1973). Many pesticides applied to foliage and nonabsorbing surfaces have vapor pressures similar to trifluralin; therefore, similar volatilization rates could be expected. Such high rates of volatilization contribute to the short-term effectiveness of many insecticides, the low use-efficiency of some herbicides, and the environmental contamination with pesticides.

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ences in volatilization rate. When trifluralin was applied to an air-dry soil surface, very low volatilization rates resulted. Vapor loss during the first hour was 0.0014 kg/ha from the dry surface application as compared with vapor losses of 0.142 and 0.153 kg/ha for the wet surface applications. The volatilization rate increased between 1 and 8 hr before the soil was wetted, because of water vapor adsorption by the soil from the 50% relative humidity carrier gas. When the dry soil was wetted, volatilization increased to 19.7 $\mu g/cm^2$ per day (1.97 kg/ha per day). In a duplicate column when 2 kg/ha of trifluralin was surface applied to the air-dry soil, without subsequent wetting, essentially all of the trifluralin was extracted from the soil column after 2.25 days of volatilization.

When trifluralin is surface applied to a moist soil, trifluralin concentrations are above that needed for a saturated vapor, and volatilization is at a rate similar to unadsorbed trifluralin. For example, 1 and 2 kg/ha of triflu-

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Translocation and Metabolism of [14C]Phorate as Affected by Percolating Water in a Model Soil-Plant Ecosystem

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A model ecosystem consisting of soils, plants, and water was utilized with corn grown in a [14C]phorate treated soil layer on top of an insecticide-free soil, while water was periodically percolated through this system. Utilizing different soil types and moist soil through which no water was percolated, the interrelationships of movement and metabolism of [14C]phorate in soils, the effects of percolating water on these processes, and the penetration, translocation, and metabolism of the insecticides in corn were investigated. Under both percolating and nonpercolating conditions, 12% of the applied radiocarbon had moved into the lower layers of agricultural soils as phorate sulfoxide and phorate sulfone. Phorate still present in the upper layers of all soils moved only in a quartz sand. Water percolated through a quartz sand, a Plainfield sand, and a Plainfield sand-silt loam mixture contained 16.4, 2.8, and 1.8% of the applied radioactivity, respectively, identified as phorate (with quartz sand only), phorate sulfoxide, and phorate sulfone. Although all soils still contained phorate, only its metabolites were found in plants. Roots contained primarily phorate sulfone and some phorate sulfoxide, while greens also contained phoratoxon sulfoxide and phoratoxon sulfone. It appears that roots of corn plants absorbed phorate from the soil and metabolized it into its sulfoxide and sulfone or absorbed these metabolites directly from the soil, then translocated them into the greens, where their further oxidation into their oxygen analogs (phosphorothiolates) presumably occurred. Roots of plants grown in a quartz sand contained 2.5-5 times more phorate-derived materials than those grown in an agricultural soil. However, the amounts of 14C-labeled materials that had been translocated into the corn greens were similar with all soils, indicating that the uptake and translocation of chemicals from soil are to a large extent governed by physiological processes of the corn plant itself.

The insecticide phorate (*O,O*-diethyl *S*-(ethylthiomethyl)phosphorodithoate) is often applied to soil for the control of soil insects, such as corn rootworms and wireworms. Its conversion in soils into its sulfoxide and sulfone has been described by Getzin and Chapman (1960) under greenhouse and laboratory conditions, while Lichtenstein (1966) and Suett (1971) recovered these metabolites under field conditions from soils previously treated with phorate. Lichtenstein *et al.* (1973) also studied the effects of application methods on the persistence and metabolism of the insecticide in an agricultural soil under field conditions. The movement of phorate and/or its metabolites in soils from the application site to other areas has received relatively little attention. Based on bioassay results, Patterson and Rawlins (1968) reported that after the application of insecticide granules to soil columns, "very little phorate was translocated through more than 3-in. of soil." Schulz *et al.* (1973) showed that phorate residues moved under field conditions both vertically and horizontally after band applications of the granular insecticide at the rates of 5 and 10 lb/acre, and that contrary to findings with emulsifiable concentrates, phorate persisted longer.

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The effects of percolating water on the persistence and movement of a soil-applied insecticide present another problem, since the chemical could be removed with water to areas where it might be unwanted. Another aspect of mobility pertains to the penetration of insecticide soil residues into roots of crop plants and their subsequent translocation and metabolism within the plant system. The metabolism of phorate in some plants was studied by Metcalf *et al.* (1957) with cotton and lemon leaves after topical application of [32P]phorate to the leaves, or by "cutting stems of mature cotton leaves and placing them into a water dispersion of the compound." Bowman and Casida (1957) treated seeds of cotton and peas with [32P]phorate and analyzed the foliage of the plants that grew from these seeds. In both studies the major metabolites found were phorate sulfoxide, phorate sulfone, phoratoxon sulfoxide, and phoratoxon sulfone. Corn silage and carrots grown in a phorate treated soil under field conditions contained small residues of phorate sulfone (Lichtenstein *et al.*, 1973). Potato tubers grown in the same soils did not contain measurable amounts of insecticide residues.

This present study was conducted with a model system that consisted of soils, corn plants, and water. With this entity we attempted to investigate the interrelationships of movement and metabolism of [14C]phorate in different soil types, the effects of water percolating through these