# Mobility and Dissipation of Ethofumesate and Halofenozide in Turfgrass and Bare Soil

D. S. Gardner† and B. E. Branham*,‡

Department of Horticultural and Crop Science, 2021 Coffey Road, The Ohio State University, Columbus, Ohio, 43210-1086 and Department of Natural Resources and Environmental Sciences, 1102 South Goodwin Avenue, University of Illinois, Urbana, Illinois, 61801-4798

The effect of turfgrass cover on the leaching and dissipation of ethofumesate and halofenozide was studied. Sampling cylinders (20 cm diam. × 30 cm long) were placed vertically in plots of creeping bentgrass (*Agrostis palustris* Huds.), tall fescue (*Festuca arundinaceae* Schreb.), or bare soil. ethofumesate [(±)-2-ethoxy-2,3-dihydro-3,3-dimethylbenzofuran-5-yl methansulfonate] was applied at 840 g ai $ha^{-1}$ on September 21, 1997. Halofenozide (*N*-4-chlorobenzoyl-*N*′-benzoyl-*N*′-*tert*-butylhydrazine) was applied at 1680 g ai $ha^{-1}$ on August 30, 1998. Replicate sampling cylinders were removed 2 h after treatment and 4, 8, 16, 32, and 64 days after treatment. Sampling cylinders were sectioned by depths and soil extracts were assayed by HPLC with a pesticide detection limit of 0.01 mg $kg^{-1}$. Turfgrass was divided into verdure and thatch and analyzed separately. ethofumesate leaching in turfgrass was reduced by at least 95% compared to leaching in bare soil. The half-life of ethofumesate in bare soil was 51 days compared to 3 days in turfgrass. Halofenozide showed similar leaching with or without turfgrass. Fifty percent dissipation of halofenozide did not occur within 64 days, regardless of organic matter cover.

**Keywords:** *Leaching; degradation; half-life; pesticides; ethofumesate; halofenozide*

## INTRODUCTION

Ethofumesate [(±)-2-ethoxy-2,3-dihydro-3,3-dimethylbenzofuran-5-yl methansulfonate] is a pre- and postemergence herbicide used to control a range of grasses and broadleaf weeds in several crops (Figure 1). In turfgrass, ethofumesate has been used for the selective postemergence control of annual bluegrass (*Poa annua* L.).

Several researchers have investigated the fate of ethofumesate on agricultural soils. The half-life in a sandy loam or a loam soil was 7.7 or 12.6 weeks, respectively (*1*). A half-life of just over 8 weeks was observed when ethofumesate was applied in September (*2*). Soil moisture is an important factor affecting ethofumesate fate. If applied to dry soil (<2.5% water), chemical degradation and strong adsorption result in significant loss of herbicidal activity (*3*). Under other conditions, e.g. normal soil moisture and warm temperatures, degradation is mostly microbial, and ethofumesate is only moderately sorbed by most soils (*4*).

Little information has been published in the literature on ethofumesate metabolism in plants. Duncan et al. (*5*) indicated that rapid metabolism in plants was the basis for ethofumesate selectivity, but no metabolites were identified in that study.

Halofenozide (*N*-4-chlorobenzoyl-*N*′-benzoyl-*N*′-*tert*-butylhydrazine; Figure 1) is a diacylhydrazine insecticide with a novel mode of action: it mimics the action of the natural insect molting hormone, 20-hydroxy ecdysone. The result is premature molting, resulting in a loss of hemolymph and molting fluid which causes desiccation and death of the larvae. Halofenozide is very selective with significant soil systemic efficacy against scarabid beetle larvae, cutworms, and webworms (*6*). A method has been developed to determine dislodgeable residues of halofenozide from socks or grass clippings (*7*). However, a thorough review of the literature revealed no previous published work on mobility or dissipation of halofenozide in soils or turf.

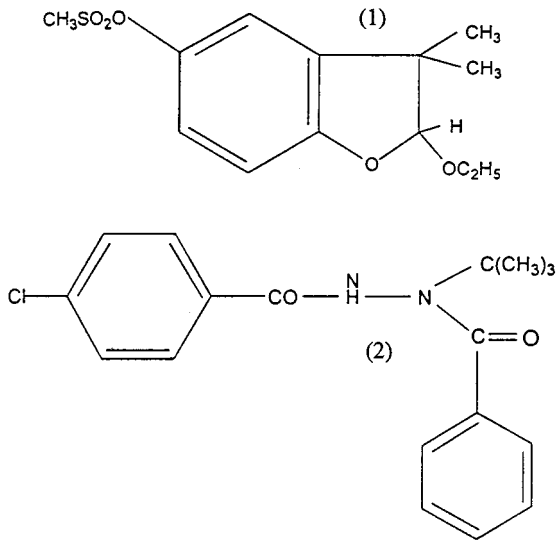


**Figure 1.** Chemical structures of ethofumesate (1) and halofenozide (2).

The use of certain pesticides for turfgrass management has been questioned in recent years because of environmental concerns. However, research suggests that pesticide mobility in turfgrass may be lower than

* To whom correspondence should be addressed. Phone: 217-333-7848. E-mail: bbranham@uiuc.edu.
† The Ohio State University.
‡ University of Illinois.

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in agronomic soils because of retention by thatch (*8, 9*). Several studies have demonstrated that the majority of pesticide residues are contained in thatch (*10−12*). Laboratory studies show that turfgrass leaves and thatch strongly sorb organic compounds and, thus, should have a significant impact on the fate of pesticides applied to turfgrass (*13, 14*). Turfgrass thatch creates an aerobic zone high in microbial activity resulting in enhanced dissipation in the turfgrass root zone (*11, 15*).

The objective of this study was to compare soil mobility and dissipation rates of ethofumesate and halofenozide on turfgrass compared to their mobility and dissipation rates on bare soil. ethofumesate was studied in creeping bentgrass. Halofenozide was studied in creeping bentgrass and tall fescue.

## MATERIALS AND METHODS

**Field Procedures.** Field experiments were conducted in Penneagle cv. creeping bentgrass turf in 1997, and in Penneagle creeping bentgrass and a blend of Montauk, Avanti, and Safari cv. tall fescue turf in 1998, at the University of Illinois Landscape Horticulture Research Center in Urbana, Illinois. The soil was a Flanagan silt loam (fine, smectic, mesic Aquertic, Argiudoll, 52 g $kg^{-1}$ organic matter, 1.38 g $cm^{-3}$ bulk density, and pH 6.5). Creeping bentgrass thatch was 10 to 14 mm thick and had a bulk density of 0.53 g $cm^{-3}$, 241 g $kg^{-1}$ organic matter, and pH 6.7. Tall fescue mat was 2 to 8 mm thick and had a bulk density of 0.58 g $cm^{-3}$, 164 g $kg^{-1}$ organic matter, and pH 6.9. Bare soil plots were prepared by stripping sod from the plot with a sod-cutter.

Sampling cylinders were constructed of 20-cm-diameter schedule 40 poly(vinyl chloride) (PVC) pipe cut into 30-cm lengths and beveled at one end to ease insertion into the soil. Sampling cylinders were inserted vertically into each plot on September 17, 1997 and August 27, 1998 using a hydraulic press (Alden Enterprises, Okemos, MI) attached to a tractor. Both pesticides were applied to plots with no slope making runoff losses unlikely.

Ethofumesate (Prograss EC) was applied on September 21, 1997 at 840 g ai $ha^{-1}$. Halofenozide (Mach 2 SC) was applied on August 30, 1998 at 1680 g ai $ha^{-1}$. The pesticides were applied with a backpack sprayer equipped with a TEEJET 8006E (Spraying Systems Co., Wheaton, IL) nozzle at a height of 36 cm, with an effective spray width of 30 cm. The pesticides were applied in 1120 L water $ha^{-1}$ at 276 kPa. Irrigation (0.4 cm) was applied immediately after treatment.

Creeping bentgrass was mowed three times per week at 1.8 cm with a reel mower, and clippings were collected. Tall fescue was mowed once per week at 8.9 cm with a rotary mower, and clippings were returned. The bare soil and turfgrass plots were irrigated equally as necessary to avoid wilt of the turfgrass (Figure 2).

**Sampling and Analysis of Pesticides.** Sampling cylinders were removed from three replicate blocks of each level of turf cover 2 h after treatment (0 days after treatment, DAT) and 4, 8, 16, 32, and 64 DAT. Sampling dates in 1997 were September 21, September 25, September 29, October 7, October 23, and November 24. Sampling dates in 1998 were August 30, September 3, September 7, September 15, October 1, and November 2. Soil cores were removed by directly cutting the PVC cylinders. Verdure and thatch were separated from the turfgrass-containing cores by using hand shears. Soil sections were separated by using a handsaw. The soil cores were partitioned into sections from depths of 0−1, 1−3, 3−5, 5−15, and 15−30 cm. Samples were weighed, placed in glass mason jars with aluminum-foil-lined lids, and stored at −20 °C until residue analysis. The amount of time required to process the samples on each collection day was about 6 h.

The moisture content of each verdure, thatch, and soil sample was determined by loss in a 60 °C drying oven for 3 days in order to calculate residues on a dry weight basis. Pesticides were extracted from soil, verdure, and thatch.

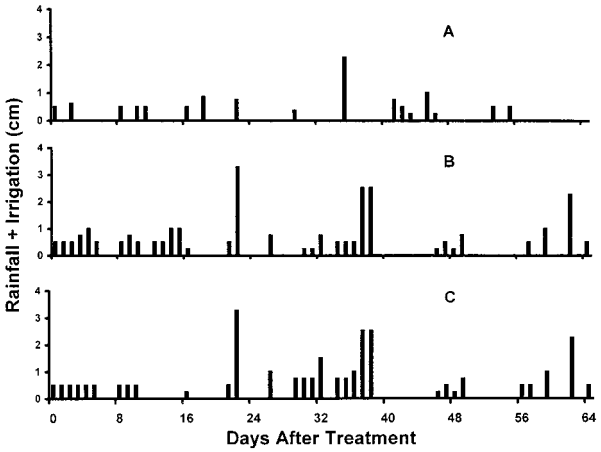


**Figure 2.** Rainfall and irrigation from date of pesticide application to last sample collection date. Pesticide application dates were September 21, 1997 (A), and August 30, 1998 in both creeping bentgrass (B) and tall fescue (C).

Samples were thawed, and representative 20-g samples (3−10 g for verdure) were placed in 500-mL Erlenmeyer flasks. Pesticides were extracted from the samples by shaking with ethyl acetate (100 mL) for 4 h (3 h for soil samples) on a platform shaker at 200 rpm. The extracts were vacuum-filtered through Whatman G6 glass fiber filters.

Ethyl acetate was removed by rotary evaporation at 40 °C. The evaporatory flask was rinsed 3 times with 5 mL of methylene chloride, and the rinsate was transferred to KD tubes. The methylene chloride was evaporated to 2 mL using a reacti-vap (Pierce, Inc., Rockford, IL). The extract was then passed through a 0.45-micron nylon membrane filter (GelmanSciences, Ann Arbor, MI)) and transferred to an autosampler vial for analysis on a high-performance liquid chromatograph (HPLC; Beckman Coulter, Fullerton, CA).

All halofenozide extracts and ethofumesate soil extracts were separated on a 15-cm, 4.6-mm i.d. column with a bonded 5-$\mu$m C-18 phase (Beckman Coulter, Fullerton, CA). Ethofumesate extracted from verdure and thatch was separated on a 25-cm, 4.6-mm i.d. column with a bonded 5-$\mu$m C-18 phase (Phenomenex, Torrence, CA).

Ethofumesate was separated from soil extractives by injecting 40 $\mu$L into a mobile phase of acetonitrile/water (64:36). The flow rate was 1 mL per min. After 3 min the mobile phase was increased to 100% acetonitrile over a 0.5 min period. Ethofumesate eluted with a retention time of 4.9 min. Ethofumesate was separated from verdure and thatch extractives by injecting 40 $\mu$L into a mobile phase of acetonitrile/water (40:60) with a flow rate of 1 mL per min. After 2 min the mobile phase was increased to 100% acetonitrile over a 10 min period. Ethofumesate eluted with a retention time of 13.1 min. Detection of ethofumesate was at 280 nm with a UV−Vis detector (Beckman Coulter, Fullerton, CA).

Halofenozide was separated by injecting 40 $\mu$L into a mobile phase of tetrahydrofuran/acetonitrile/water (20:40:40) with a flow rate of 1 mL per min. Halofenozide eluted with a retention time of 6.8 min. Detection of halofenozide was at 254 nm.

Residues were quantified by peak area measurements in comparison with a 10 $\mu$g $mL^{-1}$ external standard. The limit of detection (3 × background) for both pesticides was 10 $\mu$g $kg^{-1}$. Calibration standards were included after every sixth sample. A control fortified at 1 mg $kg^{-1}$ and a method blank were included with each batch of 22 samples. Ethofumesate recovery from soil, verdure, and thatch samples averaged 97% with a coefficient of variation of 9%. Halofenozide recovery from soil, verdure, and thatch samples averaged 96% with a coefficient of variation of 9%. Controls containing the full range of expected concentrations were also tested in order to establish linearity of response. The amount of ethofumesate or halofenozide remaining in each soil profile was calculated from data on the concentration of pesticide present in a soil core section and the mass of the core section.

**Table 1. Analysis of Variance (ANOVA) on Total Ethofumesate Residues in Verdure, Thatch, and Soil Profile Components on Different Sampling Dates during 1997**

| | | $P > F^b$ | | | | | |
|---|---|---|---|---|---|---|---|
| source[a] | df | day 0 | day 4 | day 8 | day 16 | day 32 | day 64 |
| (*R*)eplication | 2 | 0.3388 | 0.2751 | 0.8420 | 0.3662 | 0.3747 | 0.7920 |
| (*T*)urf cover | 1 | 0.0077 | 0.0513 | 0.0086 | 0.0052 | 0.0169 | 0.0008 |
| $R \times T$ (error *A*) | 2 | | | | | | |
| (*S*)oil depth section | 6 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| $T \times S$ | 4 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| $R \times T \times S$ (error *B*) | 20 | | | | | | |

[a] *T*, bare soil or creeping bentgrass turf; *S*, soil depth section (verdure, thatch, and 0−1, 1−3, 3−5, 5−15, and 15−30 cm). [b] Probability of greater *F* ratio ($P > F$) for surface organic matter content and soil profile components.

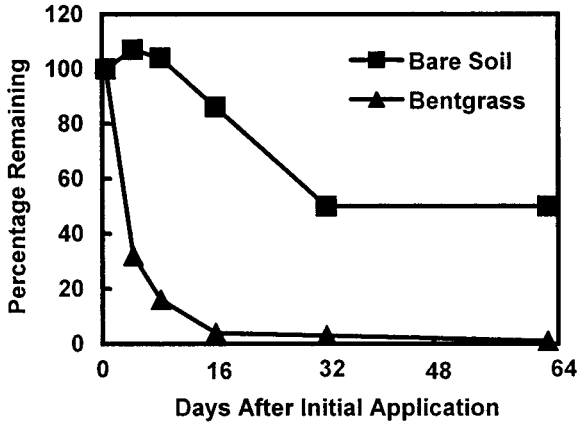


**Figure 3.** Total ethofumesate residues, summed over all sampling depths, recovered from bare soil or creeping bentgrass as a function of sampling time in 1997.

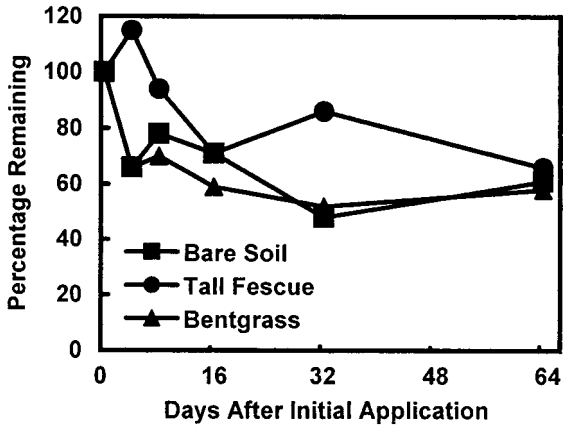


**Figure 4.** Total halofenozide residues, summed over all sampling depths, recovered from bare soil, creeping bentgrass, or tall fescue as a function of sampling time in 1998.

Each experiment was designed as a split-plot with turf cover as the whole-plot and soil depth section as the sub-plot. On each sampling date, data were subjected to analysis of variance (*16*). Half-life values were calculated by regressing the log of pesticide residues remaining in the verdure, thatch, and soil (dry weight basis) versus time. Half-life data for halofenozide was not estimated by this procedure because more than 50% halofenozide remained at 64 DAT.

## RESULTS

**Dissipation.** Ethofumesate exhibited typical 1st order decay kinetics with a rapid initial dissipation followed by a slower rate of dissipation. Ethofumesate dissipated much faster in plots containing turfgrass, with a calculated $t_{1/2}$ of 3 days ($R^2 = 0.99$, $P = 0.007$) (Figure 3). Dissipation rates were much slower in bare soil with a calculated $t_{1/2}$ of 51 days ($R^2 = 0.80$, $P =$ 0.016). No significant difference in dissipation of halofenozide was observed on bare soil or in the presence of turf. The $t_{1/2}$ of halofenozide did not occur within 64 days (Figure 4).

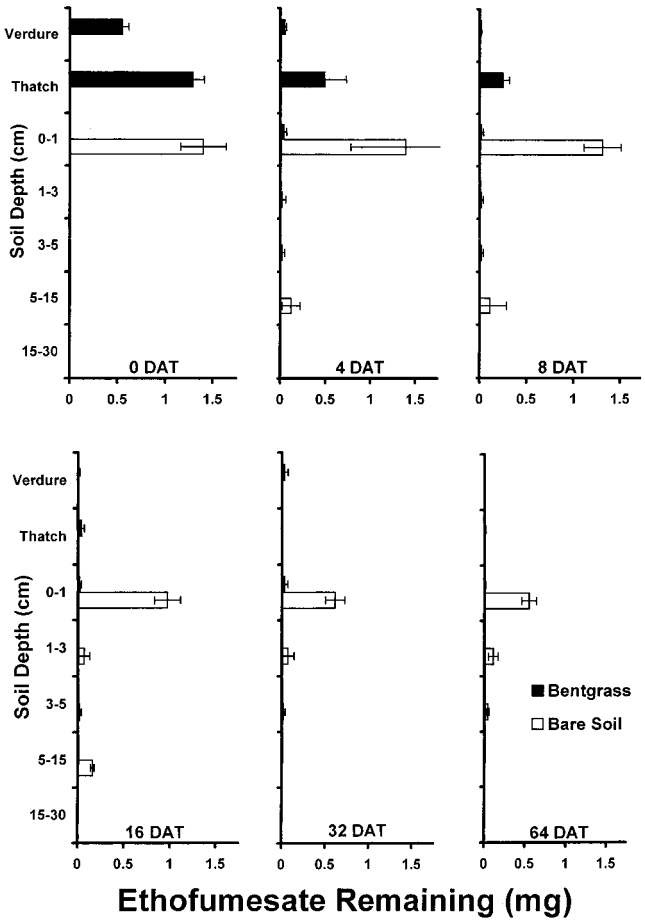


**Figure 5.** Distribution of ethofumesate residues among verdure, thatch, and different soil depths over time in 1997. Horizontal T lines represent standard error of the means.

**Soil Mobility.** The distribution of ethofumesate in the soil profile differed depending on whether application was to turfgrass or bare soil (Table 1). In bare soil, ethofumesate was detected 2 h after application only in the 0−1 cm section (Figure 5). However, at 4, 8, and 16 DAT residues were detected as deep as the 5−15 cm layer. In turfgrass, all residues were contained in the verdure and thatch 2 h after application. No ethofumesate was detected below 0−1 cm on any sampling date and the maximum amount of ethofumesate detected in soil was less than 5% of the total amount applied.

The distribution of halofenozide in the soil profile differed depending on whether the application was to bentgrass, tall fescue, or bare soil at 0 DAT and 4, 8,

**Table 2. Analysis of Variance (ANOVA) on Total Halofenozide Residues in Verdure, Thatch, and Soil Profile Components on Different Sampling Dates during 1998**

| | | $P > F^b$ | | | | | |
|---|---|---|---|---|---|---|---|
| source[a] | df | day 0 | day 4 | day 8 | day 16 | day 32 | day 64 |
| (*R*)eplication | 2 | 0.5577 | 0.0777 | 0.3087 | 0.3828 | 0.8671 | 0.5599 |
| (*T*)urf cover | 2 | 0.0079 | 0.0088 | 0.0148 | 0.0285 | 0.7756 | 0.5147 |
| $R \times T$ (error *A*) | 4 | | | | | | |
| (*S*)oil depth section | 6 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| $T \times S$ | 10 | 0.0001 | 0.0001 | 0.0001 | 0.0001 | 0.0696 | 0.0024 |
| $R \times T \times S$ (error *B*) | 32 | | | | | | |

[a] *T*, bare soil, creeping bentgrass turf, or tall fescue turf; *S*, soil depth section (verdure, thatch, and 0−1, 1−3, 3−5, 5−15, and 15−30 cm). [b] Probability of greater *F* ratio ($P > F$) for surface organic matter content and soil profile components.

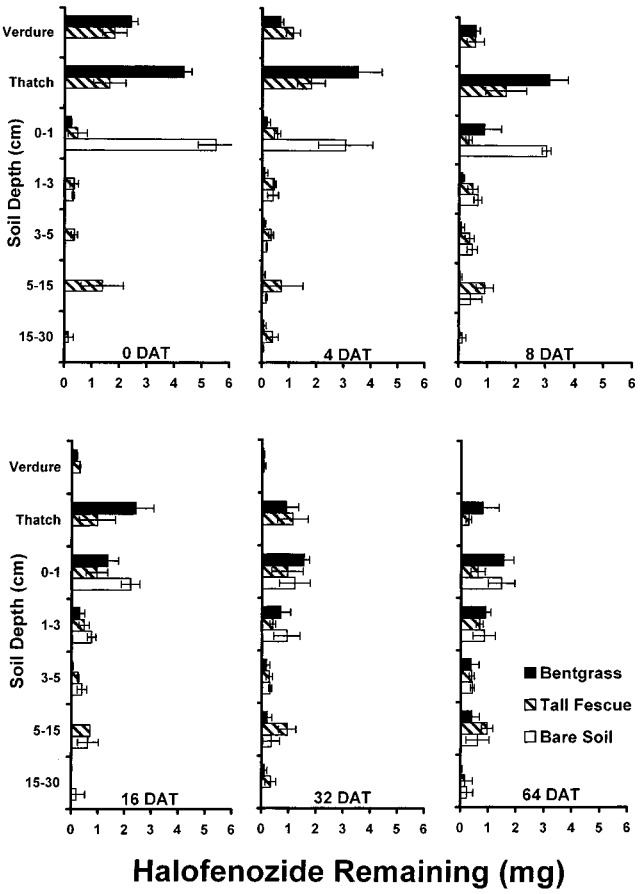


**Figure 6.** Distribution of halofenozide residues among verdure, thatch, and different soil depths over time in 1998. Horizontal T lines represent standard error of the means.

and 16 DAT (Table 2). In soil at 0 DAT, most halofenozide was in the 0−1 cm section, but in bentgrass most of the residues were in the verdure and thatch (Figure 6). In tall fescue at 0 DAT, halofenozide was distributed nearly equally between the verdure, thatch, 0−1, 1−3, 3−5, and 5−15 cm soil sections.

At 4, 8, 16, 32, and 64 DAT, halofenozide applied to bare soil was detected at all sample depths. The majority of the residue was detected in the 0−1 cm layer but this decreased over time because of dissipation and redistribution into the lower soil depths. At 32 and 64 DAT the distribution of halofenozide in the 0−1, 1−3, 3−5, and 5−15 cm soil layers was the same regardless of application to bare soil, bentgrass, or tall fescue.

## DISCUSSION

The dissipation rate of ethofumesate in bare soil was similar to that reported by Wauchope et al. (*17*) but more rapid than that observed in other studies. Dissipation was much more rapid in turfgrass than in bare soil, most likely due to microbial decomposition (*4*). Microbial activity associated with turfgrass thatch results in rapid ethofumesate degradation before significant movement to soil can occur.

McAuliffe and Appleby (*3, 18*) found greater herbicidal activity on wet soils than on dry soils, suggesting another mode of ethofumesate dissipation. In the present study, faster dissipation was observed in thatch than in the 0−1 cm soil layer of the bare soil plots. Although ethofumesate is primarily a preemergence herbicide, the accelerated rate of degradation in turf organic matter essentially eliminates preemergence activity when applied to turf.

Late September is the normal time of ethofumesate application to turf, and the ethofumesate study was initiated on September 21. For the time period between the 32- and 64-DAT sample collections, average air temperature was 3.2 °C and average soil temperature at the 10-cm depth was 6.1 °C. Ethofumesate recovery from bare soil did not differ at 32 or 64 DAT indicating that microbial decomposition rates may have slowed or stopped because of low soil and air temperatures. Schweizer (*1*) observed much more rapid dissipation rates when ethofumesate was applied in March than when applied in November.

Leaching of ethofumesate in bare soil was consistent with that noted in previous studies (*1, 2, 4*). However, leaching of ethofumesate in turfgrass was greatly reduced. Previous researchers correlated the adsorption of ethofumesate in agricultural soils with clay and smectite content, but correlation with soil organic matter content was borderline ($p < 0.10$) insignificant (*19, 20*). Although adsorption and leaching are usually tightly correlated, our research indicates that ethofumesate is degraded so rapidly in turf organic matter that leaching has little chance to be a significant process.

Dissipation and soil mobility of halofenozide were not affected by surface organic matter in this study. Considerable leaching of halofenozide in bare soil was observed. More rainfall was received during the halofenozide study than during the ethofumesate study (Figure 2), aiding vertical movement of halofenozide. Rainfall soon after application may have moved the halofenozide beyond the turf organic matter before much degradation occurred.

Very rapid vertical movement of halofenozide in tall fescue may be due to uptake and root translocation of halofenozide by the turfgrass. Whereas the cores were removed from the field at 2 hours after treatment, up to 6 h was needed to section the cores and collect subsamples. The turfgrass samples had between 2 and 8 h for translocation to occur. Tall fescue typically has

a much deeper root system than creeping bentgrass; it is also possible that creeping bentgrass may not translocate halofenozide as readily as tall fescue. Lack of halofenozide translocation may explain the reduced vertical movement of halofenozide in creeping bentgrass at 0, 4, 8, and 16 DAT as compared to the movement in bare soil or tall fescue. Halofenozide taken up by plants would be unavailable for microbial degradation, which may offset the more rapid degradation expected within the thatch layer (*11*, *15*), leading to a dissipation rate similar to that measured in bare soil.

Previous research has shown that mobility and dissipation of certain pesticides is affected by turfgrass cover (*21*, *22*). The results of this study indicate that ethofumesate behaves very differently when applied to turfgrass compared to its behavior when applied to bare soil. More research is needed to determine if lack of differences seen with halofenozide are due to chemical and physical properties of the pesticide, or if plant uptake and translocation plays a role in the distribution and persistence of halofenozide. Regardless, turf cover appears to have little influence on the distribution and persistence of halofenozide.

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