Method for the Analysis of Triadimefon and Ethofumesate from Dislodgeable Foliar Residues on Turfgrass by Solid-Phase Extraction and In-Vial Elution

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Triadimefon, a fungicide, and ethofumesate, an herbicide, are commonly applied to turfgrass in the Pacific Northwest, resulting in foliar residues. A simple and rapid method was developed to determine triadimefon and ethofumesate concentrations from dislodgeable foliar residues on turfgrass. Turfgrass samples were washed, and wash water containing surfactant (a 0.126% solution) was collected for residue analysis. This analytical method utilizes a 25 mm C_8 Empore disk and in-vial elution to quantitatively determine triadimefon and ethofumesate in 170 mL aqueous samples. The analytes were eluted by placing the disk in a 2 mL autosampler vial with 980 μL of ethyl acetate and 20 μL of 2-chlorolepidine, the internal standard, for analysis by GC/MS. The method quantitation limits are 0.29 μg/L for ethofumesate and 0.59 μg/L for triadimefon. The method detection limits are 0.047 μg/L and 0.29 μg/L for ethofumesate and triadimefon, respectively. Concentrations of triadimefon and ethofumesate from dislodgeable foliar residues from a field study are reported.

Keywords: *Empore disks; dislodgeable foliar residues; triadimefon; ethofumesate*

INTRODUCTION

There is a need to better understand pesticide fate in agriculture so that we may assess exposure risks to human health and the environment. Triadimefon and ethofumesate (Figure 1, Table 1) are two semipolar, nonvolatile pesticides commonly applied to turfgrass in the Pacific Northwest, and their application results in foliar residues. Triadimefon is a systemic fungicide used to control rusts in turfgrass (Tomlin, 1997). In plants, it degrades by reducing the carbonyl group to a hydroxyl group to form triadimenol. Triadimefon's soil half-life ranges from 6 to 18 days, and triadimenol's soil half-life ranges from 110 to 375 days (Tomlin, 1997).

Ethofumesate is a selective, systemic herbicide used as a preemergent or postemergent herbicide. It is metabolized in plants to the 2-hydroxy and 2-oxo derivatives, and it is biologically degraded in soil. Its half-life ranges from less than 5 weeks under moist and warm conditions to more than 14 weeks under dry and cold conditions (Tomlin, 1997).

Methods used for the extraction of pesticides from water include liquid−liquid extraction (Bellar and Budde, 1988) and solid-phase extraction (SPE) (Benfenati et al., 1990; Brooks et al., 1990; Patsias and Papadopoulou-Mourkidou, 1996; Johnson et al., 1991). SPE methods are gaining popularity because they are rapid, easily automated, and use less solvent than liquid−liquid extraction techniques. Solid-phase extraction cartridges containing C_8 and C_{18} phases have been used to isolate a number of pesticides from water in an in-line or off-line mode (Noij and van der Kooi, 1995; Junk et al., 1988; Junk and Richard, 1988).

An alternative to the use of liquid−liquid extraction or solid-phase extraction cartridges is the use of solid-phase extraction Empore disks. Empore disks eliminate solvent concentration and exchange steps and reduce organic solvent use compared to solid-phase extraction cartridges. Krueger and Field (1995) developed a technique that eliminated the need for the elution of compounds from disks by placing disks directly into autosampler vials filled with the elution solvent. Variations of this in-vial approach to disk elution were used for the determination of acid herbicides in surface water and groundwater (Field et al., 1997; Field and Monohan, 1995, 1996). In-vial elution was also used to determine surfactants and metabolites in sewage effluent, paper mill effluent, and river water (Krueger and Field, 1995; Field and Reed, 1996).

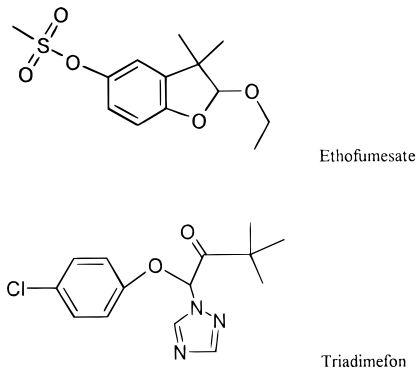

Figure 1. Structures of ethofumesate and triadimefon.

Others have evaluated the use of Empore disks for the extraction of pesticide residues and other pollutants from water (Hagen et al., 1990; Davì et al., 1992; Viana et al., 1996; Bengtsson et al., 1994; McDonnell and Rosenfeld, 1993). One reported advantage of using Empore disks for SPE is that they have a greater cross-sectional area than SPE cartridges, resulting in reduced plugging. Also, the decreased back pressure associated with Empore disks makes higher flow rates possible,

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Table 1. Physical−Chemical Properties for Triadimefon and Ethofumesate[a]

physical/chemical property	triadimefon	ethofumesate
water solubility	64 mg/L (20 °C)	50 mg/L (25 °C)
vapor pressure (mPa)	0.02 (20 °C)	0.12−0.65 (25 °C)
K_{ow} (log P)	3.11	2.70
solvent solubility	moderate (200 g/L), except aliphatics (20 °C)	acetone > methanol > hexane (400−4.67 g/L, 25 °C)

[a] Tomlin (1997).

and because the stationary phase is embedded into a disk format, channeling is avoided (McDonnell and Rosenfeld, 1993; Markell et al., 1991). It has also been noted that using Empore disks eliminates interferences previously associated with the use of SPE cartridges and results in lower detection limits (Hagen et al., 1990; Markell et al., 1991).

Because Empore disks have a small pore size, researchers have often found it necessary to use a prefilter when loading samples containing organic matter or suspended particulates to avoid clogging of the disks. This has been successfully achieved by acidifying the water (Davì et al., 1992), placing prefilters directly on top of Empore disks (Markell et al., 1991; McDonnell and Rosenfeld, 1993; Bengtsson et al., 1994), or by prefiltering water samples and loading the filtrate onto Empore disks (McDonnell and Rosenfeld, 1993; Field et al., 1997).

To the best of our knowledge, evaluation of the use of solid-phase extraction Empore disks has not been explored for the recovery of pesticides, specifically triadimefon and ethofumesate, from dislodgeable foliar residues. The objective of this work was to establish a method for in-vial disk elution with GC/MS analysis for the determination of triadimefon and ethofumesate in dislodgeable foliar residues. This method was validated and applied to the detection of these pesticides in field samples in a study aimed at understanding the fate and distribution of these pesticides in the environment.

MATERIALS AND METHODS

Standards and Reagents. Standards of triadimefon (1-(4-chlorophenoxy)-3,3-dimethyl-1-(1*H*-1,2,4-triazol-1-yl)-2-butanone, 98%) and ethofumesate (2-ethoxy-2,3-dihydro-3,3-dimethylbenzofuran-5-yl methanesulfonate 99%) were obtained from Chem Services (West Chester, PA). The internal standard was 2-chlorolepidine 99% (Aldrich, Milwaukee, WI). All standards were prepared in ethyl acetate. Acetone and methanol (trace residue grade) were purchased from J.T. Baker (Phillipsburg, NJ), and HPLC-grade ethyl acetate was purchased from EM Science (Gibbstown, NJ).

Surfactants. Two surfactants were used during the course of this study. Triton X-100 (octyl phenoxy polyethoxyethanol) was purchased from Sigma (St. Louis, MO) and used for dislodgeable foliar residue washing of turfgrass samples during the first year of sample collection, 1995. Nekal WT-27 or Geropon WT-27 (sodium dioctyl sulfosuccinate) was obtained from Rhône-Poulenc (Cranbury, NJ) and used for samples taken in 1996, the second year of sample collection.

Samples. Pesticide mixtures were applied to a 5000 ft^2 orchard ryegrass turf plot at Lewis Brown Horticulture Farm in Peoria, OR, using a small plot ground rig. Both compounds were applied at the label rate of application; triadimefon was applied at 3.05 kg of active ingredient (ai)/ha (305 mg/m^2), and ethofumesate was applied at 2.5 kg ai/ha (250 mg/m^2). Sample collection and washing of turfgrass were conducted on post-application days 0−3, 5, 7, 14, and 21. The procedure for collecting dislodgeable foliar residues was based on a modified procedure (Iwata et al., 1977). Briefly, turfgrass samples were collected in triplicate from random positions within a 20 m diameter circular test plot. Samples were taken using a 10.8 cm diameter modified golf cup cutter. This resulted in a sample area of 91.5 cm^2. Turfgrass was subsequently separated from thatch and soil and placed in clean 16-oz wide mouth glass jars and capped with an aluminum foil or a Teflon-lined lid. Samples were stored on ice until they were washed within 2 h of collection.

The samples were washed for 20 min on a mechanical shaker in jars containing 150 mL of water and four pipet drops (0.126% solution) of a 1:50 dilution of Triton X-100 or Nekal WT-27 surfactant. The wash water containing detergent was decanted off the washed turfgrass into a 250 mL polypropylene bottle through a funnel with glass wool at the bottom of the cone. The jar was rinsed with an additional 20 mL of water which was added to the polypropylene bottle. The remaining solution was squeezed into the sample bottle from the glass wool with a clean stirring rod. The sample bottle was placed in a −20 °C freezer to await pesticide extraction. Storage stability samples spiked at 5.88 μg/mL were stored along with field samples. Method development samples were collected from the Lewis Brown Horticulture Farm and washed using the technique described above.

Filtration and Solid-Phase Extraction. To remove particulates in dislodgeable foliar residue (DFR) samples, they were filtered prior to solid-phase extraction. Samples were first filtered through a 90 mm S&S 589 Black Ribbon and a Whatman GF/A filter, followed by filtration through a 55 mm GF/F filter. Triadimefon and ethofumesate were extracted from filtered DFR samples with 25 mm C_8 bonded-phase silica Empore disks (Varian, Sugarland, TX) using a modified technique (Field and Monohan, 1995). The disk was placed in a polypropylene filter holder attached to a 75 mL polypropylene reservoir. The disk was rinsed with 8 mL of ethyl acetate that had soaked into the disk for 30 s. Then it was rinsed with 8 mL of methanol that had soaked into the disk for 30 s, followed by 8 mL of double-deionized water (DDI) (Labconco, Kansas City, MO). The two solvents and DDI were drawn through the C_8 disks under a vacuum of 10 mm Hg. Following the ethyl acetate rinse, the disk was allowed to go dry, but following subsequent rinsing with methanol and DDI, the disk remained wet. The sample was then added to the reservoir, and the sample container was rinsed with 10 mL of DDI that was added to the reservoir. Samples were drawn through the disks under 20 mm Hg. Following sample loading and reservoir removal, 60 cm^3 of air was pushed through the disk 3 times to expedite excess water removal. The C_8 disk was then dried for 15−30 min by drawing air through the disk at 20 mm Hg. The disk was removed from the filter holder, folded in half, rolled up, and placed in a 2 mL glass autosampler vial with 980 μL of ethyl acetate and 20 μL of 2-chlorolepidine (2 μg). The vial contents were allowed to equilibrate for at least 4 h prior to analysis.

In-Vial Elution. An experiment was performed to determine the amount of time required for disks to equilibrate in the autosampler vial containing the elution solvent, ethyl acetate (EA). Triadimefon and ethofumesate were extracted from DDI onto a 25 mm C_8 disk, and the disk was placed in the autosampler vial with 1 mL of EA. The autosampler then injected a 1 μL aliquot of the sample at 7.25 min after EA introduction into the autosampler vial with the disk and at intervals of 18.92 min thereafter. A parallel experiment was conducted with a 25 mm C_{18} disk. Autosampler injections occurred at 1.5 min and at intervals of 18.92 min thereafter. The oven program was altered at a later date so that the sample run time increased.

Recovery and Precision. To determine the recovery of triadimefon and ethofumesate from DDI, six 100-mL samples

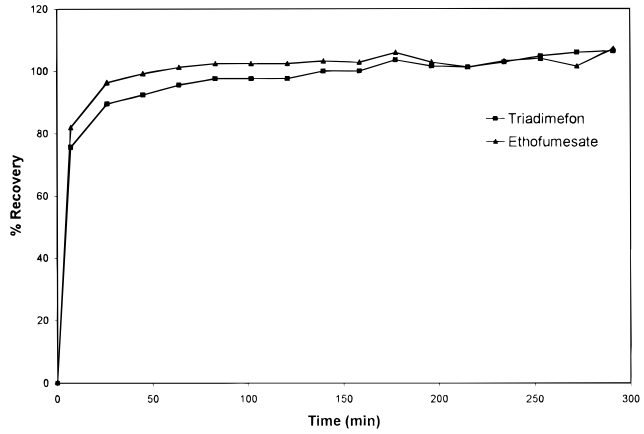

Figure 2. Elution of triadimefon and ethofumesate from a 25 mm C_8 disk with time.

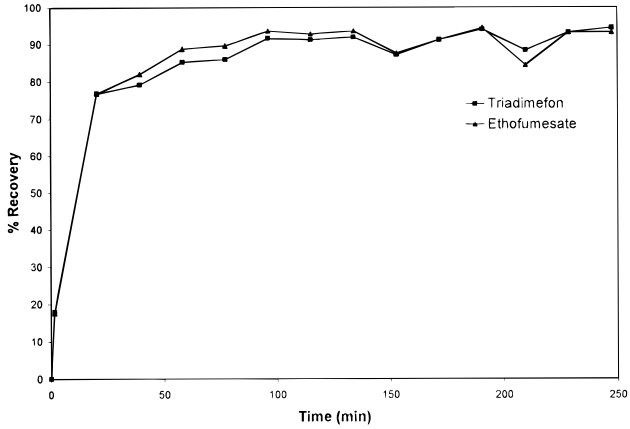

Figure 3. Elution of triadimefon and ethofumesate from a 25 mm C_{18} disk with time.

were spiked to give a final concentration of 59 μg/L of each analyte and filtered and extracted as described above. To determine analyte recovery using the surfactants, this procedure was repeated with six 100-mL DDI samples and Triton X-100 or Nekal WT-27 surfactant. Matrix samples containing undetectable levels of each analyte were used to determine the recovery of ethofumesate and triadimefon for spiked field samples.

The precision of the method was determined by spiking six replicate samples of blank unfiltered matrix (170 mL) to give a final concentration of 5.88 or 11.77 μg/L. The spiked matrix samples were then filtered and extracted.

Gas Chromatography. All samples were separated and analyzed using a Hewlett-Packard model 6890 Series gas chromatograph coupled with a Hewlett-Packard model 5972 mass selective detector (GC/MS). The samples were separated with a DB-17 column (30 m × 0.25 mm × 0.25 μm film thickness; J&W, Folsom, CA). The initial oven temperature was held 1 min at 80 °C and then increased at 5 °C/min to a final temperature of 215 °C, which was held for 3 min. An injection volume of 1 μL was used under pulsed splitless conditions with an injector temperature of 225 °C. The MS was operated under selective ion monitoring (SIM) at a temperature of 280 °C. The retention times (RT) and ions (m/z) monitored for each pesticide are as follows: triadimefon, RT = 28.40 min and m/z = 110, 128, 181, 208; ethofumesate, RT = 29.10 min and m/z = 207, 286, 161, 137; and 2-chlorolepidine RT = 21.05 min and m/z = 115, 142, and 177.

Quantitation. The concentrations of triadimefon and ethofumesate were calculated from a 5−8 point standard calibration curve constructed from the ratio of the peak areas of each analyte to the peak area of 2-chlorolepidine. The concentrations of the analytes in the calibration samples ranged from 0.05 to 10.0 μg/mL, and each vial contained 2.0 μg of 2-chlorolepidine. Calibration curves were linear, and r^2 values were typically 0.995 or greater.

Safety. Only normal laboratory safety is required.

RESULTS AND DISCUSSION

Solid-Phase Extraction. Traditional SPE elution techniques from both cartridges and disks involve adding some quantity of solvent to the cartridge or disk and pulling it through the matrix under vacuum. The in-vial elution approach places the elution solvent and disk together. Therefore, there is some optimum time at which the greatest analyte recovery will be achieved. To determine both the recovery and the rate of analyte elution from disks, samples were repeatedly analyzed for analytes from C_8 and C_{18} disks using GC/MS. Triadimefon and ethofumesate recovery was 106.4−107.2% from the C_8 disks in 3.7 h (Figure 2). Recoveries for triadimefon and ethofumesate from the C_{18} disks were lower (93.2−94.4%) (Figure 3). Since recoveries were greater from the C_8 disk, we felt that it was a more suitable matrix for this method. Samples were typically analyzed after a 4−24 h equilibration period, even though acceptable recoveries were obtained after 3 h.

Table 2. Recovery of Pesticides from Double-Deionized Water (DDI) and Two Surfactant Solutions[a]

	percent recovery ± SD (%RSD)[b]	
matrix	triadimefon	ethofumesate
DDI	98.3 ± 3.8(3.8)	91.8 ± 3.0(3.3)
Triton X-100	98.0 ± 6.1(6.2)	95.3 ± 6.1(6.4)
Nekal WT-27	97.8 ± 7.8(7.9)	95.5 ± 8.3(8.7)

[a] 0.126% solution of surfactant (4 pipet drops). [b] Average ± standard deviation (%RSD) for four replicate samples for each matrix spiked to give a final concentration of 59 μg/L for each analyte.

A breakthrough study was initiated to determine the disk capacity. Replicate samples (170 mL of DDI) spiked with 147 μg/L of the pesticide mix were loaded onto a C_8 disk. The sample was passed through a second C_8 disk, and the amount of analyte contained on the disk was analyzed. No analytes were detected. Therefore, one C_8 disk isolated both analytes from 170 mL of water samples.

Accuracy and Precision. The percent recovery and standard deviation of triadimefon and ethofumesate from 100 mL of DDI, DDI with Triton X-100, and DDI with Nekal WT-27 were determined (Table 2). Triadimefon recovery for four replicate analyses was 98.3 ± 3.8, 98.0 ± 6.1, and 97.8 ± 7.8. Ethofumesate recovery was 91.8 ± 3.0, 95.3 ± 6.1, and 95.5 ± 8.3 from the three matrixes. The relative standard deviations (RSD) were less than 10% for all samples. A two-tailed t-test (95% confidence interval) was performed on data to determine whether there was a significant difference in analyte recoveries in DDI compared to DDI and either surfactant. The statistics showed no significant difference between recoveries from the three matrixes.

Replicate analyses (n = 6) of blank dislodgeable foliar residue samples spiked to give 5.9 μg/L concentrations of each analyte gave recoveries ranging from (95.5 ± 17.0)% for triadimefon to (91.7 ± 7.5)% for ethofumesate (Table 3). Replicate samples (n = 6) spiked to give 11.8 μg/L concentrations of each analyte gave recoveries ranging from (87.0 ± 11.1)% for triadimefon to (94.1 ± 5.9)% for ethofumesate (Table 3). The RSD varied from 6.3% to 17.8%. The greatest variation was observed for triadimefon at the lowest concentration used for the recovery study. Ethofumesate exhibited RSD values

Table 3. Recovery of Pesticides from Blank Dislodgeable Foliar Residue (DFR) Samples

matrix	concentration (μg/L)	percent recovery ± SD (%RSD)[a] triadimefon	ethofumesate
blank DFR	5.9	95.5 ± 17.0(17.8)	91.7 ± 7.5(8.2)
blank DFR	11.8	87.0 ± 11.1(12.8)	94.1 ± 5.9(6.3)

[a] Average ± standard deviation for six replicate samples.

Table 4. Triadimefon and Ethofumesate Concentrations in Dislodgeable Foliar Residue (DFR) Samples Collected in 1996

postapplication day	concentration (μg/L) ± SD[a] triadimefon	ethofumesate
background samples	<dl	<dl
1	4040 ± 1580	2590 ± 1290
2	1850 ± 440	2940 ± 1620
3	2780 ± 1860	1550 ± 360
5	2590 ± 2370	690 ± 411
7	1330 ± 1170	331 ± 138
14	557 ± 728	273 ± 285
21	81.0 ± 71.0	325 ± 47.0

[a] Three replicate samples analyzed. <dl below the detection limit of 0.047 and 0.29 μg/L for ethofumesate and triadimefon, respectively.

below 10% for all samples analyzed in the recovery study. Other researchers have reported that 93% or 85% of ethofumesate is recovered from field water samples (spiked at 0.5 μg/L) extracted by liquid−liquid extraction or SPE, respectively (Patsias et al., 1996). Reported triadimefon recoveries from laboratory water and groundwater using SPE range from 83.1% to 107% (Benfenati et al., 1990; Brooks et al., 1990). Viana et al. (1996) reported 81% triadimefon recovery from spiked water using C_8 disks.

Detection and Quantitation Limits. Detection and quantitation limits were determined by spiking blank dislodgeable foliar residue samples to give final concentrations of 0.047, 0.29, or 0.59 μg/L for each analyte. The detection limit, defined as a signal-to-noise ratio of 3, was 0.047 μg/L for ethofumesate and 0.29 μg/L for triadimefon. The limit of quantitation, defined as a signal-to-noise ratio of 10, was 0.29 μg/L for ethofumesate and 0.59 μg/L for triadimefon.

Turfgrass Samples. Replicate dislodgeable foliar residue samples ($n = 3$, 170 mL) obtained from the field study conducted from June 12 to July 7, 1996 were analyzed for ethofumesate and triadimefon. Triadimefon concentrations ranged from 4040 μg/L on day 1 to 81.0 μg/L on day 21. Ethofumesate concentrations ranged from 2590 μg/L on day 1 to 325 μg/L 21 days following application (Table 4). Triadimefon and ethofumesate recoveries were 90% and 92%, respectively, for storage stability samples spiked at 5880 μg/L.

CONCLUSIONS

A simple and rapid method was developed for the determination of triadimefon and ethofumesate from dislodgeable foliar residues on turfgrass. By using in-vial elution, both sample extraction time and solvent use were decreased. While methods that use Empore disks for the extraction of pesticides from ground and surface water samples are common, use of these disks for the extraction of pesticides from dislodgeable foliar residues has not been previously described.

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