JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

J. Agric. Food Chem. **2005,** *53,* 5079−5083 **5079**

Pesticide Extraction Efficiency of Two Solid Phase Disk Types after Shipping

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An interlaboratory study was conducted to compare pesticide recovery from Empore C_{18} and Speedisks C_{18}XF solid phase extraction disks after shipping. Four pesticides were used for the comparison of the two disk extraction materials: atrazine, diazinon, metolachlor, and tebuconazole. These pesticides were chosen to provide a range of physiochemical properties. Water samples were extracted onto the disk types and shipped to a cooperating laboratory for elution and analysis. The mean recoveries from Empore disks were atrazine, 95%; diazinon, 91%; metolachlor, 92%; and tebuconazole, 83%. The recoveries from Speedisks C_{18}XF were atrazine, 89%; diazinon, 87%; metolachlor, 86%; and tebuconazole, 79%. Means for each of the pesticides using the different disk types were not statistically different ($\alpha = 0.05$), but results were more variable when using Speedisks C_{18}XF as compared to Empore disks. Reasons for the increased variability are discussed, but overall results indicate that Speedisks C_{18}XF could be used as an alternative to Empore disks. Speedisks C_{18}XF are enclosed in a plastic housing, so they can be used more easily in remote sampling sites without the possibility of glassware breakage, no prefiltration of samples is needed, and there are realignment problems that can be associated with the Empore disks.

KEYWORDS: Solid-phase extraction (SPE); pesticides; water; extraction; Empore disks; Speedisks

INTRODUCTION

While essential to maintain high agricultural production (*1*), pesticides have been found to pose serious water quality threats in some settings (*2*). Misuse can result in the contamination of plant, soil, and water resources. The water solubility of some pesticides can result in their movement and contamination of sites distant from their initial use. The concern by the general population over possible contamination of water resources has resulted in a major focus on monitoring pesticide concentrations in water. Government and private agencies as well as individuals demand reliable analytical methods capable of detecting chemical contaminants at trace levels.

Methods for pesticide extraction from environmental water samples have undergone many changes since the development of the first analysis methods were developed for testing water. Initial methods involved liquid−liquid extraction (LLE) requiring large volumes of potentially hazardous organic solvents that ultimately need proper disposal. These methods could only be performed in a laboratory and required the shipment of large

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10.1021/jf050029s CCC: $30.25
Published on Web 05/24/2005

volumes of water in glass containers from collection sites. Glass containers containing water samples could break during transport, and their shipment is expensive. Solid-phase extraction (SPE) methods are an effective alternative to LLE methods (*3*−*6*). SPE methods can reduce organic solvent use, decrease sample preparation time, and may reduce costs. The sorbent or stationary phase in SPE is bonded to a solid support that is configured as a disk, cartridge, or fiber. During filtration using cartridges and disks, the pesticides sorb to the stationary phase and then are eluted with a minimal amount of organic solvent.

Empore SPE disks, a commonly available type of SPE disk, were tested as an alternative to the shipment of water samples in an effort to reduce shipping costs and container breakage. Studies were conducted to determine if sample integrity was maintained when pesticides sorbed to the Empore SPE disks were shipped to an analytical laboratory. Results showed that minimal recovery losses were observed during the shipment of disks (*7*−*9*). Observed losses were pesticide specific when using this two-stage analytical procedure (*8*).

A couple of problems were encountered when using Empore SPE disks for pesticide extraction at one site followed by shipment to another site for elution and analysis. Once removed for shipping, it was impossible to perfectly realign disks onto another laboratory's extraction manifold so that all of the impregnated portions of the disk would be exposed to the elution solvent. Realignment problems resulted in reduced recovery from incomplete pesticide elution. This problem was solved by combining the disks with the elution solvent in screw cap tubes, which were then placed on a shaker (*9*). In addition, surface water with high levels of particulates clogged disks and required a filtration step prior to passing the water sample through the disk.

Speedisks offer an alternative to the use of traditional Empore SPE extraction disks. Speedisks contain the extraction sorbent in a plastic housing, which is placed directly onto an extraction manifold, eliminating the realignment problems previously noted. Various sorbents are available, but C_{18} is most commonly used for the extraction of many pesticides and other pollutants. The XF version of the Speedisks contains a prefilter, eliminating the need for a separate filtration step to remove particulates. The combination provides one-step filtration and extraction. Speedisks can also be used on any manufacturer's extraction manifold with the use of appropriate adaptors.

No data are published that directly compare the Speedisk and traditional SPE disks such as the Empore SPE disks for the extraction of pesticides followed by shipment of the disks to an analytical laboratory. The purpose of this study was to compare the recovery of four pesticides (atrazine, diazinon, metolachlor, and tebuconazole) as a function of disk type (Empore and Speedisk) after shipping fortified disks to another laboratory. The choice of pesticides to use in this study was based on several factors. In urban streams, the percent detection of atrazine, metolachlor, and diazinon was 74, 65, and 50%, respectively (*10*). In stream samples from areas where land use was predominantly agricultural, values were 80, 13, and 68%, respectively. Tebuconazole was not among the target compounds in NAQWA samples. The compound's environmental fate properties (low water solubility and relatively high soil persistence) and widespread use in peanut production indicated that it has potential for runoff and detection in surface water in the southeastern United States (*11*). Validating a method for this compound will help improve monitoring efforts.

MATERIALS AND METHODS

Cooperating Laboratories. Laboratories included Clemson University; Mississippi State University; Southeast Watershed Research Laboratory, USDA (Tifton, GA); Texas A&M University; University of Arkansas; University of Puerto Rico; University of Tennessee−Knoxville; Virginia Tech; and Virginia State University. Laboratories were paired as shipping and receiving facilities. One laboratory was unable to complete the sample analysis in the time frame of the study, and a second laboratory served as the producer of the stock solutions of pesticides used by all of the laboratories associated with the study.

Water Fortification and Extraction Protocol. *Fortification Standards.* The pesticides selected for this study belong to different chemical classes and have different physical properties. They were also some of the most widely detected active ingredients found in surface water (*10*). Atrazine [6-chloro-*N*-ethyl-*N*′-(1-methylethyl)-1,3,5-trazine-2,4-diamine, CAS# 1912-24-9] is a triazine herbicide. Metolachlor [2-chloro-*N*-(2-ethyl-6-methylphenyl)-*N*-(2-methoxy-1-methylethyl)acetamide, CAS# 51218-45-2] is a chloroacetanilide herbicide. Diazinon {*O*,*O*-diethyl *O*-[6-methyl-2-(1-methylethyl)-4-pyrimidinyl]phosphorothioate, CAS# 333-41-5} is an organothiophophate insecticide. Tebuconazole {α-[2-(4-chlorophenyl)ethyl]-α-(1,1-dimethylethyl)-1 *H*-1-2-4-triazole-1-ethanol, CAS# 107534-96-3} is a conazole fungicide. The water solubility ranged from approximately 25 to 500 mg/L. All standards were obtained from Chem Serviece, Inc.

A fortification solution of atrazine, diazinon, metolachlor, and tebuconazole in methanol was prepared (200 μg mL^{-1} each pesticide) by laboratory 5. Portions (≈8 mL) of this solution were placed in two 4 mL borosilicate glass vials sealed with Teflon-lined screw caps and wrapped with sealant film for shipment to each laboratory. Vials were weighed before shipment and after receipt to determine solution loss and ensure integrity of standards. Laboratory 5 did not ship or receive samples.

Chemicals and Extraction Disks. All solvents used in this study were pesticide grade or capillary gas chromatography/mass spectrometry (GC/MS) grade. Sodium sulfate was ACS reagent or environmental residue analysis grade. Each laboratory used a vacuum extraction manifold that accommodated 47 mm Empore C_{18} extraction disks (3M, St. Paul, MN, part no. 2215) and Speedisks C_{18}XF 50 mm disks. (J. T. Baker, Phillipsburg, NJ, part no. 8056-06). The manifolds required adaptors for accommodation of Speedisks. The manifolds used for extraction were not identical, and the exact diameter of the Empore disk, which was exposed to the water sample, was not exactly the same between the different laboratories. Disks, solvents, and chemicals were obtained from laboratory supplies distributors Fisher Scientific (Pittsburgh, PA), VWR International (West Chester, PA), and Sigma-Aldrich Chemicals (St. Louis, MO).

Procedures for Shipping Laboratory. Each facility prepared four fortified Empore samples and one nonfortified control sample using distilled or deionized water. The nonfortified samples and one fortified sample were immediately eluted and analyzed for quality control. Samples were assumed similarly fortified. The analytical results from the nonfortified sample verified that no interfering compounds were added to the disks, and the results from the fortified sample verified that the shipped samples were fortified. Three fortified samples were placed in a desiccator overnight. The following day, disks were placed in individual plastic bags with appropriate labeling using waterproof pens. A calibrated HOBO datalogger (Onset Computer Corporation, Bourne, MA) was placed in the shipment to record temperatures during shipment of disks. Samples were shipped to the partner laboratory by overnight carrier. Sample extraction and shipment were timed such that samples would not be held over a weekend. Disks were extracted within 24 h of receipt. The same procedures were used for the Speedisks.

One nonfortified and one fortified water sample were prepared and extracted onto Empore disks by each receiving laboratory. These samples were eluted and analyzed along with the samples received from the partner laboratory. Nonfortified samples showed no interferences; therefore, it was assumed that no interferences were added to the received samples. The same procedures were used for the Speedisks.

Water Extraction Protocol. The fortification solution (50 μL) was added to each 1 L distilled, deionized water sample, yielding a final

Table 1. GC Conditions for the Various Laboratories Associated with Study

lab no.	1	2	3	4	6	7[a]	8
type GC	HP	Varian	Agilent	Perkin-Elmer	HP	Agilent	Varian
detector	NPD	MS	μECD	NPD	MS	μECD	MS
injection volume (μL)	1	1	2	1	5	1	1
column size, film thickness (m × mm × μm)	30 × 0.53 × 1.2	30 × 0.25 × 0.25	30 × 0.32 × 0.25	30 × 0.25 × 0.25	25 × 0.2 × 0.33	30 × 0.25 × 0.25	30 × 0.25 × 0.25
stationary phase	SE–30	DB-5MS	HP-5	DB-5	Ultra 2	RTX-5	DB-5MS
temp program[b]	110 (1) –15–190 (2) –2–210 (0) –20–235 (0)	80 (0.25) –10–280 (0)	170 (5) –12–260 (5)	150 (1) –8–260 (5)	150 (1)– 10–235 (0) –20–310 (5)	50 (0)– 10–230 (5) –20–280 (10)	150 (1)– 8–260 (5)
injector temp (°C)	230	55 (0.25) –180–250 (0)[c]	225	250	250	250	220
detector temp (°C)[d]	250	260	300	300	300	350	240
flow (mL min^{-1})	2.6	1.1	1.5	1.0	0.4	1.3	1.5

[a] Alternate instrumental analysis used for detection of tebuconazole. Detector: μECD, 280 °C; injection volume, 1 μL. Column: RTX-5, 30 m × 0.25 mm × 0.25 μm. Temperature program: 50 (0)–35–200 (0)–12–250 (30); injector temperature, 250 °C; and flow, 1.0 mL min^{-1}. [b] Temperature program of column: 80 (0.25)–10–280 (0) is read as 80 °C for 0.25 min, followed by an increase of 10 °C per min to 280 °C with a hold time of 0 min. [c] Injector programming available with this instrument is a technique for removing solvent and concentrating analytes at the front of the capillary column without needing to use split injection. It allows larger volumes and therefore amounts to be injected while maintaining column efficiency. [d] Detector temperatures associated with GC/MS units are actually transfer line temperatures.

concentration of 10 μg L^{-1} for each pesticide. Methanol (4 mL) was added to each water sample before extraction to enhance wetting of the C_{18} material conditioned during the extraction process.

The extraction protocol was similar for all participating laboratories. The C_{18} extraction disk or Speedisk C_{18}XF was placed on the manifold, 10 mL of ethyl acetate was added to the disks, and a vacuum was applied. The vacuum was continued for 2 min after all of the ethyl acetate passed through the disk. Methanol (10 mL) was then added to the disk, and the vacuum was applied until a thin film of methanol remained on top of the disk. The disks were not allowed to go dry during this step or any of the following steps until the entire water sample had passed through the disk. A 10 mL portion of water was then applied to the disk and pulled through the disk leaving a thin film of water on top of the disk. A second 10 mL portion of water was applied to the disk in the same manner. The 1 L water sample was then pulled through the disk. The time was recorded at the beginning and end of filtration to determine the flow rate for each sample. Once the water sample had passed through the disk, the vacuum was allowed to pull air through the disk for at least 5 min to allow for a partial drying of the disk.

Extraction of In-House Samples. A glass container was placed in the extraction manifold under the disk, and the sides of the reservoir were rinsed with 5 mL of ethyl acetate. The vacuum was applied to pull approximately 1−10 drops of ethyl acetate through the disk, and the vacuum shut off. After the ethyl acetate was allowed to solvate the disk for 2 min, the ethyl acetate was pulled through the disk by vacuum. An additional 5 mL of ethyl acetate was then added and pulled through the disk. After the ethyl acetate was collected, approximately 3 g of anhydrous sodium sulfate was added to the glass container to remove water from the eluate. The ethyl acetate was transferred to a calibrated test tube. The sodium sulfate was rinsed with an additional 5 mL of ethyl acetate, which was decanted into a calibrated test tube. The ethyl acetate was concentrated to 5.0 mL under a stream of nitrogen in a room temperature water bath. Samples were then transferred to sample vials, sealed with Teflon-lined caps, and stored at <−4 °C until analyzed.

Extraction of Shipped Samples. Because of slight differences in the opening size on the extraction manifolds used in the different laboratories, the extraction of the shipped Empore was handled differently than samples completed in-house. Each Empore disk was extracted with 10 mL of ethyl acetate in a 25 mL culture tube secured with Teflon-lined screw caps by placing them on a shaker for 15 min. The ethyl acetate was decanted into a test tube containing approximately 3 g of anhydrous sodium sulfate. The disk was extracted with an additional 5 mL of ethyl acetate by shaking for 5 min. The first ethyl acetate portion was decanted into a calibrated test tube. The 5 mL of ethyl acetate from the second extraction was used to rinse the sodium sulfate and was then decanted into the calibrated test tube. The sample was concentrated to 5.0 mL under a stream of nitrogen in a room temperature water bath. The sample was then transferred to a sample vial, sealed with a Teflon-lined cap, and stored at <−4 °C until analyzed. This shaking with ethyl acetate procedure was used for the Empore disks to eliminate the problems of slight differences between the openings in different laboratory manifolds and realignment of disks in the manifold. Speedisks were eluted using the same procedure as described above for in-house samples.

GC. The GC conditions for analysis varied among the laboratories involved in the study due to differences in equipment, columns, and detectors. Specific conditions for laboratories associated with the study are reported in **Table 1**.

Statistical Analysis. The experimental design was a completely randomized two factor factorial design with three replications per lab−disk combination. Data from laboratory 8 were not available for tebuconazole. For each compound, the mixed model analysis of variance (ANOVA) for proportion recovered included disk type as a fixed effect and laboratory as a random effect. Hence, the mean proportion recovered depended only on disk type while the variance depended on laboratory, the lab−disk type interaction, and experimental error. On the basis of preliminary descriptive statistics, the variance of experimental error was allowed to differ by disk type. Results were converted from proportions to percentages for presentation purposes. *P* values are presented for each test rather than results of a significance test at a fixed α. The statistical analyses were carried out using SAS version 8 (SAS Institute, Inc., Cary, NC).

RESULTS AND DISCUSSION

Standard and Sample Shipment. Minimal losses were observed during the transport of standards (<0.1% w/w) from the central laboratory preparing the standards and the individual laboratories conducting the experiments. The shipment of identical standards for use at all sites eliminates the variability of standards that would have been observed if laboratories had made their own standards for conduct of experiments.

Previous shipment of disks from one site to another led to questions concerning the temperatures that disks are exposed to during their transport from one site to another for analysis.

Table 2. Mean Percent Recovery of Pesticides from SPE Disks with Standard Errors

lab no.	disk type	atrazine[a]	diazinon[a]	metolachlor[a]	tebuconazole[a]
1	Empore	96 ± 4	95 ± 4	91 ± 4	84 ± 4
	Speedisks	86 ± 4	91 ± 4	81 ± 3	83 ± 3
2	Empore	101 ± 3	93 ± 4	98 ± 2	86 ± 3
	Speedisks	104 ± 5	90 ± 6	99 ± 6	94 ± 6
3	Empore	72 ± 8	66 ± 2	71 ± 2	51 ± 7
	Speedisks	71 ± 3	72 ± 2	74 ± 1	51 ± 2
4	Empore	100 ± 5	93 ± 2	85 ± 2	93 ± 7
	Speedisks	88 ± 10	87 ± 10	79 ± 8	97 ± 11
6	Empore	104 ± 5	98 ± 4	105 ± 4	90 ± 4
	Speedisks	86 ± 6	82 ± 6	85 ± 5	72 ± 7
7	Empore	85 ± 3	87 ± 4	87 ± 3	95 ± 5
	Speedisks	79 ± 2	86 ± 4	80 ± 2	71 ± 13
8[b]	Empore	105 ± 3	102 ± 1	109 ± 3	
	Speedisks[c]	103 ± 16	97 ± 14	98 ± 15	

[a] Mean percent recovery ± standard error based on three replicate samples for each laboratory. [b] Laboratory 8 did not recover tebuconazole. [c] Mean recovery percentages are based on two replications.

Table 3. ANOVA *p* Values for the Test of No Disk Effect on the Mean Percent Recovery for Each Compound and Estimated Mean Percent Recovered

compd	*p* value for no disk effect	percent recovery[a] Empore	percent recovery[a] Speedisks
atrazine	0.1098	94.6 (4.5)	88.7 (4.8)[b]
diazinon	0.1401	90.7 (3.9)[b]	86.5 (4.4)[b]
metolachlor	0.0939	92.2 (4.5)[b]	85.5 (5.0)[b]
tebuconazole	0.4446	83.1 (6.6)[b]	78.6 (7.1)[b]

[a] Estimated standard error of the mean is given in parentheses. [b] Significantly less than 100% recovery at $\alpha = 0.1$ level of significance.

The temperatures observed during the shipment of disks from one laboratory to another did vary some depending on sites. The average maximum and minimum temperatures observed were 38 ± 4 and 9 ± 2 °C, respectively. Temperature differences are dependent on the time of year that samples are shipped, the mode of transport (ground vs air), and temporary storage conditions prior to delivery. All shipping for this study was conducted during March through April when high temperatures were not likely. Investigations into the effect of high temperatures during shipment of disks are presently being conducted to determine its effect on the overall pesticide recovery.

Disk Type. Percent recoveries of the individual pesticides from the two types of SPE disks varied greatly between the different laboratories (**Table 2**). The average recovery for atrazine/Empore disk ranged from 72 to 105% as compared to atrazine/Speedisks of 71 to 104%. Similar recovery percentages were obtained for diazinon/Empore (66−102%) and diazinon/Speedisks (72−97%) and for metolachlor/Empore (71−109%) and metolachlor/Speedisks (74−99%). Tebuconazole analysis was a problem for some laboratories due to excessive peak tailing and low sensitivity when using mass spectrometry or electron capture detection. The overall recoveries were lower for tebuconazole/Empore (51−95%) and tebuconazole/Speedisks (51−97%).

Considering the traditional 5% as the benchmark for statistical significance, no significant difference was observed between the disk types for any of the pesticides tested when results were pooled across laboratories (**Table 3**). All recoveries were significantly less than 100% except for atrazine and metolachlor when using Empore disks. When replacing the traditional 5% with 10% for statistical significance, there is evidence of a difference in mean percent recovery based on disk type for metolachlor and a marginal indication for atrazine recovery. There is still no evidence of a difference in the recoveries for diazinon or tebuconazole based on disk type. At the 10% statistical significance level, only atrazine recovery on Empore disks was not significantly less than 100% recovery. Using the 10% statistical significance level increases the chance of identifying false differences but gives a larger chance of detecting differences that do exist. This level of statistical significance may be needed with the small sample sizes associated with each laboratory.

The variability of the percent recoveries when using Speedisks was significantly higher than the variability of the percent recoveries obtained when using the Empore disks (**Table 4**). The variance was partitioned between the laboratory, laboratory × disk, and error components in an effort to determine the reason for the differences in the variability observed. Some variability is associated with the differing analytical conditions associated with the different laboratories. This variability, however, should have been the same for the Empore and Speedisks. It does not explain the increase variability associated with the Speedisks. Some of the laboratories involved in this study had not used Speedisks previously while all the laboratories had used Empore disks extensively prior to this study. This unfamiliarity with Speedisks may account for the higher variability observed with these samples. In discussions between the cooperating laboratories, two factors were noted as possible reasons for the greater error variance associated with the Speedisks. Some laboratories pulled the vacuum on the Speedisks a shorter period of time after the water passed through the disk prior to removal from the extraction apparatus. This resulted in greater water content in the eluate requiring more anhydrous sodium sulfate in the drying step. An additional difference noted between laboratories was the sample flow rates through the disks due to the variability of individual vacuums. A majority of the variability associated with Empore disks was associated with the laboratory site while the experimental error was higher with the Speedisks.

Table 4. Variance Components as a Percent of the Total Variance and Estimated Total Variance for Percent Recovery for Each Compound and Disk Type

	atrazine		diazinon		metolachlor		tebuconazole	
source	Empore	Speedisk	Empore	Speedisk	Empore	Speedisk	Empore	Speedisk
lab	60.2	47.0	63.9	41.6	75.6	46.6	62.5	43.3
lab × disk	2.3	1.8	<0.1	<0.1	9.6	5.9	12.7	8.8
error	37.5	51.2	36.1	58.4	14.8	47.4	24.7	47.9
total	100.0	100.0	100.1	100.1	100.0	99.9	99.9	100.0
variance of percent recovery	1.90	2.44	1.45	2.23	1.61	2.61	3.16	4.57

Many factors can be associated with the variability of recovery percentages for the pesticides used in this study. Identifiable variables include differences in analytical instrumentation, different people using the same procedure (often slight variations in the way the procedure is conducted occur), different manifolds and associated vacuums, experience of the people conducting the analysis (some laboratories were more experienced in using Empore disks when compared to Speedisks), and temperature during conduct of the experiment. This study showed that at the current time with the laboratories associated with this study the variability was higher with the Speedisks but there were minimal differences between the two disk materials.

Overall results indicate that Speedisks can provide good pesticide recovery when compared to Empore disks. The Speedisks provide more flexibility when extracting pesticides from water samples in field situations since they do not require the glassware associated with the extraction manifolds used with Empore disks and the disk material to which the pesticides are adsorbed is safely held within the plastic housing of the Speedisks, which can be easily placed on various manufacturer's manifolds. Additionally, no prefiltration of turbid samples is required with the Speedisks, which is required when using Empore disks. Field extraction manifolds are currently being constructed using readily available PVC components. The manifolds will be tested using the Speedisks in field situations at remote sites where laboratory facilities may be hours/days away. Studies are also being conducted to determine the effect of temperature during the shipment of disks from one site to another. The development of these procedures will allow for pesticide extraction to be conducted at any remote site where analytical equipment is not readily available without having the expense of shipping water samples to distant laboratories.

ACKNOWLEDGMENT

Appreciation is extended to the excellent technical support staff instrumental in the conduct of this research. Our technical support staff includes the following: Frances Harper, Clyde McNamee, Kathy Carson, and Audie Sciumbato. Without their assistance, this work could not have been conducted. Helpful comments and suggestions by Clarence Watson are also appreciated.

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Received for review January 6, 2005. Revised manuscript received April 27, 2005. Accepted May 1, 2005. This cooperative research was conducted by the S1011 Southern Region Research Project, which received direct support from the respective Agricultural Experiment Stations in each state and from the U.S. Department of Agriculture, Agricultural Research Service. Technical contribution no. 5101 of the South Carolina Agricultural Experiment Station.

JF050029S