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Evaluation of Accelerated Solvent Extraction (ASE) for Analysis of Pesticide Residues in Soil

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Accelerated solvent extraction, or ASE, is a new extraction technique that is similar in principle to Soxhlet extraction, but the use of elevated temperature and pressure with ASE allows the extraction to be completed within a short time and with a small quantity of solvent. In this study, we investigated the effect of residue aging, solvent type, and ASE conditions on the recovery of atrazine and alachlor from different soils and compared the efficiency of ASE with that of Soxhlet and solvent−shake extractions. With ASE, the use of dichloromethane−acetone (1:1, v/v) or methanol as solvent resulted in significantly greater pesticide recovery than hexane. After the residue was aged for >2 weeks, pesticide recovery was significantly influenced by the extraction temperature in ASE vessel, and the recovery increased to 130−140 °C and then decreased. The efficiency of ASE was generally better than that for Soxhlet or shake extraction using methanol−water (4:1, v/v). ASE extraction also consumed considerably less solvent than the other two conventional methods.

Introduction

Extraction is often the most time-consuming step in pesticide residue analysis. Exhaustive methods such as Soxhlet extraction require the use of long time cycles (8−24 h). Alternative methods such as solvent−shake extraction are highly labor intensive because of the use of multiple extraction steps, unless a robotic system is employed (*1*). The sample throughput of these methods is typically low, and the cost is high because of the requirement for intensive human handling. In addition, large amounts of solvents are often needed, and their purchase and waste disposal further add to the overall cost of sample analysis.

In recent years, several new extraction techniques have appeared, one of which is accelerated solvent extraction, or ASE. ASE is similar in principle to Soxhlet extraction, except that elevated temperatures and pressures are used in enclosed vessels, which allows extraction by a small amount of solvent (<50 mL) to be completed in a very short time (<20 min). Early studies showed that hot (>100 °C) and pressurized solvents resulted in improved recovery of PCBs and PAHs from soil (*2*). It was believed that hot and pressurized solvents were able to more effectively solubilize the contaminants and penetrate the sample matrixes. Application of ASE has been reported for the extraction of various organic compounds from different environmental samples, but most applications are found for contaminants of industrial origin, including PAHs (*2*−*10*), PCBs (*11*), phenols (*12*, *13*), dioxins (*14*), and EPA semivolatile organic priority pollutants (*15*). In the studies where method comparisons were made, the performance of ASE was consistently equivalent to or better than conventional methods such as Soxhlet and solvent−shake extractions, as well as new methods such as supercritical fluid extraction and microwave-assisted extraction. ASE application has been reported for the analysis of only a few pesticides in soil, including chlorinated insecticides (*16*, *17*), organophosphorus insecticides (*18*), the herbicide diflufenican [2′,4′-difluoro-2-(α,α,α-trifluro-*m*-tolyloxy) nicotinanilide] (*19*), and the fungicide hexaconazole [(*RS*)-2-(2,4-dichlorophenyl)-1-(1*H*-1,2,4-triazol-1-yl)hexan-2-ol) (*20*). All of these studies have clearly demonstrated the usefulness of ASE. Most studies, however, were conducted using freshly spiked samples (*17*−*19*) or were conducted without method comparison (*16*). It is well-known that as pesticide residues age in soil, the extractability of pesticides decreases. Thus, recovery of pesticides from spiked samples is not necessarily indicative of the ruggedness of an extraction method.

In this study, we evaluated the effect of ASE conditions on the recovery of atrazine [2-chloro-4-(ethylamino)-6-(isopropylamino)-*s*-triazine] and alachlor [2-chloro-2′,6′-diethyl-*N*-methoxymethylacetanilide] residues that were aged in soil for different lengths of time, and compared the extraction efficiency of ASE with that of Soxhlet and solvent−shake extractions. Atrazine and alachlor are two of the most widely used herbicides in the United States, and their parent and metabolic compounds have been frequently detected in groundwater and surface waters. So far, however, there is no report on the development of ASE methods for the extraction of these two herbicides from soil.

TABLE 1. Soil Physical−Chemical Properties

soil	OC (%)	clay (%)	sand (%)	silt (%)	pH[a]
Webster clay loam	3.48	28.8	35.4	35.9	5.2
Waukegan silt loam	3.43	22.4	24.9	52.7	5.5
Linne clay loam	2.51	31.3	36.7	32.0	6.8
Arlington sandy loam	0.92	7.4	74.6	18.0	6.7

[a] Measured with a water/soil ratio of 1:1 (w/w).

Experimental Section

Soils and Chemicals. Four soils from Minnesota and California were used: Webster clay loam (fine loamy, mixed, mesic, typic Endoaquolls; Waseca, MN), Waukegan silt loam (fine silty over sandy or sandy-skeletal, mixed, mesic, typic Hapludolls; Rosemont, MN), Arlington sandy loam (coarse loamy, mixed, thermic, haplic Durixeralf; Riverside, CA), and Linne clay loam (fine loamy, mixed, thermic, calcic pachic Haploxerolls; Paso Robles, CA). Soils were passed through a 2-mm sieve without complete air-drying. The physical−chemical properties of these soils are given in Table 1. Only the Webster clay loam was used for method optimization, while method comparison was performed using all four soils. Standards of atrazine (98% purity) and alachlor (99% purity) were purchased from Chem Service (West Chester, PA), and were used to spike soils in the incubation experiment and as standards in GC quantification.

Incubation Experiment. Soils were treated with atrazine and alachlor at 10 mg kg^{-1} for each pesticide, and then incubated for 2, 8, and 26 weeks to generate aged residue

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samples. The pesticide spiking solution (1000 μg mL^{-1}) was prepared in water containing 10% (v/v) of acetone. The use of 10% acetone was to overcome the limitation of atrazine solubility and is allowed according to U.S. EPA's relevant guidelines for pesticide degradation study. An aliquot of soil (200 g) was thoroughly mixed with 20 mL of the pesticide solution in a beaker, and acetone in the soil was removed by placing the samples in a hood overnight. The treated sample was then mixed with 1800 g of untreated soil, and deionized water was added to adjust the soil water content to 19% for Webster clay loam, Waukegan silt loam, and Arlington sandy loam and to 28% for Linne clay loam. The use of a higher water content for the Linne soil was necessary because of its high content of montmorillonite clay. The treated soil was transferred into 4-L jars, and the jars were loosely covered with aluminum foil. All soil jars were kept at room temperature (21 ± 1 °C), and soil moisture was maintained by adding deionized water when needed.

ASE Method Optimization. The effect of solvent type and ASE operational variables temperature, pressure, and static time on pesticide recovery was evaluated using incubated Webster clay loam. ASE temperature and pressure are conditions at which the extraction cells are held during extraction, while static time is the length of time that the cells are kept at the selected temperature and pressure. At 2, 8, and 26 weeks after treatment, a fraction of the Webster soil was subject to ASE extraction under different conditions. An automated Dionex-200 ASE system (Dionex Co., Sunnyville, CA) was used for all the extractions. Treated soil (20 g, moist weight) was mixed with 10 g of washed silica sand and 3 g of Hydromatrix (ISCO, Lincoln, NE) and then packed into 33-mL stainless steel ASE vessels. The packed vessels were sealed at both ends with circular cellulose filters and then end caps. The use of sand and Hydromatrix was to reduce the void volume and facilitate solvent penetration through the soil matrix. Hydromatrix should also help to reduce the residual water in the final extract.

To determine the effect of solvent type, extraction was conducted using dichloromethane−acetone (1:1, v/v), methanol, or hexane under the same ASE conditions (100 °C, 1500 psi, and 15 min static time). At the end of extraction, N_2 was used to purge the extract into glass collection vials, and the final volume of extract was ~40 mL. Six replicates were used for each solvent type. Extracts were further concentrated to near dryness by rotary evaporation and then redissolved in 10 mL acetone. An aliquot of the final sample was injected into a HP 5890 GC equipped with a nitrogen−phosphorus detector. The analytical conditions on GC were 30 m × 0.53 mm (i.d.) × 1.5 μm (film thickness) DB5 capillary column (J&W Scientific, Folsom, CA), 6 mL min^{-1} flow rate (helium), 240 °C injector temperature, and 210 °C isothermal oven temperature.

After a solvent was selected, a Central Composite design was used to vary temperature (T), pressure (P), and static time (t) on ASE. Temperature was varied from 60 to 140 °C with a 20 °C step, pressure from 500 to 2500 psi with a 500 psi step, and static time from 5 to 25 min with a 5-min step. A quadratic model was used for describing interactions between the response (pesticide concentration) and the variables:

$$C = b_0 + b_1T + b_2P + b_3t + b_4T^2 + b_5TP + b_6P^2 + b_7tT + b_8tP + b_9t^2 \quad (1)$$

where C is the recovered concentration, b_0 is the intercept, and b_i (i = 1, 2, ..., 9) are the fitted coefficients. A total of 20 variable combinations were tested, with the central treatment (100 °C, 1500 psi, and 15 min) replicated six times to give information on method reproducibility. The Response Surface method was used to statistically solve eq 1 and to generate the probability level (P) for each individual coefficient. When a positive correlation ($P < 0.05$) was identified for a given variable, a second optimization experiment was performed to further evaluate the interaction between the response and that variable by using a smaller step. Similar experimental design and regression analysis were previously used for developing SFE and ASE methods (*9, 21*). Such a multivariate optimization scheme is more efficient than separately changing each variable; it also allows nonlinear interactions to be identified.

TABLE 2. Atrazine and Alachlor Concentrations in Soil (mg kg^{-1}) Determined after ASE Extraction Using Different Solvents (Labeling of Different Letters Denotes Difference at $P = 0.05$ for Samples Taken at the Same Time after Treatment)

time of incubation	DCM−acetone	hexane	methanol
	Atrazine		
2 weeks	8.68 ± 0.40^a	7.97 ± 0.15^a	7.36 ± 0.35^a
8 weeks	5.25 ± 0.10^a	3.66 ± 0.15^b	5.05 ± 0.62^a
26 weeks	2.69 ± 0.05^a	2.16 ± 0.19^b	2.63 ± 0.08^a
	Alachlor		
2 weeks	6.66 ± 0.31^a	6.36 ± 0.36^a	6.35 ± 0.14^a
8 weeks	5.10 ± 0.10^a	3.74 ± 0.15^b	5.17 ± 0.75^a
26 weeks	3.62 ± 0.08^a	2.98 ± 0.25^b	3.53 ± 0.12^a

Method Comparison. Method comparison was made between optimized ASE and Soxhlet extractions, and ASE and solvent−shake extractions. Samples for all soil types from the incubation experiment were used for the comparison. The procedures used in solvent−shake extraction followed those published in other studies (*22, 23*), while the conditions adopted for Soxhlet extraction were typical of similar operations. For Soxhlet extraction, 20-g soil samples were refluxed in 300 mL of dichloromethane−acetone (1:1, v/v) for 8 h on a Soxhlet extraction device. The extract was concentrated to near dryness in a rotary evaporator, and then redissolved in 10-mL acetone. For solvent−shake extraction, 10-g soil aliquots were shaken with 20 mL of methanol−water (4:1, v/v) for 1 h, and the supernatant was decanted after the mixture was centrifuged at 10 000 rpm for 15 min. The procedure was repeated two more times, and the volume of the combined extract was decreased to ~15 mL using a rotary evaporator. The aqueous sample was then acidified to pH ≈ 1 with HCl and partitioned three consecutive times with chloroform (30 mL) using a separatory funnel. The combined organic phases were again evaporated to near dryness, and then redissolved in 10 mL of acetone. Six replicates were used for each extraction method, and analysis was conducted under identical chromatographic conditions. The significance level for method comparison was set at $P = 0.05$.

Results and Discussion

Solvent Effect for ASE Extraction. The solvent systems selected for evaluation represent some of the most common solvents used for pesticide residue analysis. The influence of solvent type on pesticide recovery by ASE apparently changed with incubation time (Table 2). Two weeks after treatment, when the residue was still relatively fresh, no difference was observed among the three solvents in their efficiency in extracting atrazine and alachlor. However, after the samples were "aged" for 8 or 26 weeks, pesticide recovery using dichloromethane−acetone or methanol was significantly better than using hexane ($P < 0.05$), while the recovery using dichloromethane−acetone and methanol was not significantly different (Table 2). For instance, after 26 weeks of incubation, recovery of atrazine or alachlor achieved by using

TABLE 3. Correlation Coefficients and Their Probability Levels (*P*) of Atrazine Recovery and ASE Variables in eq (1)

	2-wk incubation		8-wk incubation	
coefficient	coefficient	*P*	coefficient	*P*
b_0	8.34×10^{0}	0.271	4.75×10^{0}	*0.012*[a]
b_1	6.11×10^{-2}	0.500	2.59×10^{-2}	0.199
b_2	-2.67×10^{-1}	0.443	-1.05×10^{-1}	0.175
b_3	-2.68×10^{-4}	0.938	-8.46×10^{-4}	0.268
b_4	-4.00×10^{-4}	0.276	-2.93×10^{-4}	*0.003*
b_5	-1.28×10^{-3}	0.614	1.76×10^{-3}	*0.008*
b_6	-4.37×10^{-3}	0.450	-1.48×10^{-3}	0.246
b_7	-1.80×10^{-6}	0.943	6.99×10^{-6}	0.218
b_8	-1.74×10^{-5}	0.863	6.37×10^{-6}	0.771
b_9	-9.03×10^{-8}	0.874	1.01×10^{-7}	0.418

[a] *P* values in italics indicate significance at $P = 0.05$.

hexane was only 80% of that obtained by using dichloromethane−acetone. The different extraction efficiencies by different solvents may be attributed to the different polarities of these solvents. It also suggests that nonpolar solvents alone should not be used for ASE extraction.

Effects of ASE Operational Conditions. Sample equilibration temperature, pressure, and time are the three most important variables that could potentially affect the extraction efficiency of ASE. Regression analysis using recovered herbicide concentrations in soil as the input for response and the actual ASE conditions as the input for variables in the quadratic model showed that ASE conditions, within the selected ranges, had no effect on the recovery of atrazine or alachlor from the 2-week samples (Table 3). Similar analysis for the 8- and 26-weeks samples showed, however, that the recovery of atrazine and alachlor was significantly influenced by temperature (T^2) as well as the product of temperature and pressure (PT), as shown in Table 3 for atrazine. It is also clear from the regression analysis that the temperature influence was not a simple linear effect.

The above observation indicates that as pesticide residence time in soil increases, more rigorous conditions are needed to extract the pesticide from the sample matrix. The effect of temperature on recovery was further evaluated for the 8- and 26-week samples by varying temperature with a small increment (10 °C) under fixed pressure (1500 psi) and static time (5 min). The reaction of pesticide recovery to temperature variation is shown in Figure 1. As temperature increased, pesticide recovery first increased, and then decreased; this effect was greater for alachlor than for atrazine. The temperature at which maximum recovery was obtained was ~140 °C for atrazine and ~120 °C for alachlor for both the 8- and 26-week soil samples (Figure 1). Further regression analysis showed that under the same pressure and static time conditions, pesticide recovery could be well described by the following relationship simplified from eq1

$$C = a + bTP + cT^2 \qquad (?)$$

where a, b, and c are the coefficients of correlation. As shown in Figure 1, the regression lines closely depict the actual measurements, especially for the 26-week samples.

It was noted that when the temperature was >130 °C, signals other than those attributable to atrazine and alachlor began to appear on the GC chromatograms, and the background noise also increased (Figure 2). This indicates that the use of very high extraction temperatures had resulted in the co-extraction of other organic substances from soil and/or formation of degradation products of the parent compounds. Although not further investigated in this study, the role of pressure for ASE extraction may be important in that a high-pressure keeps the solvent in the liquid phase at a high temperature. Thus, the optimal ASE conditions for the extraction of atrazine and alachlor from aged soil samples when using dichloromethane−acetone should fall into the following ranges: temperature, 100−130 °C; pressure, >1500 psi; static time, ≥5 min.

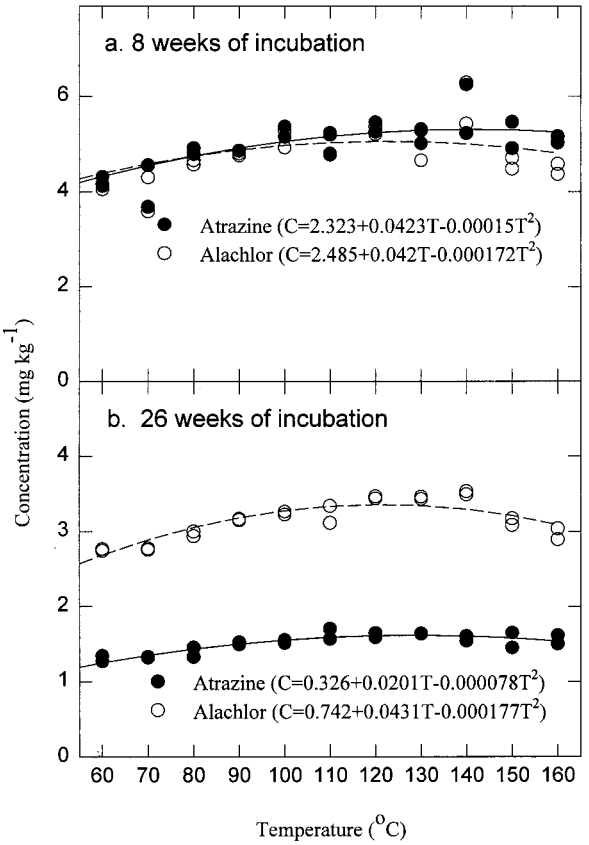

FIGURE 1. Response of atrazine and alachlor concentrations in soil (mg kg^{-1}) determined after accelerated solvent extraction (ASE) using different extraction temperatures. Extraction pressures were 1500 psi, and static time was 15 min.

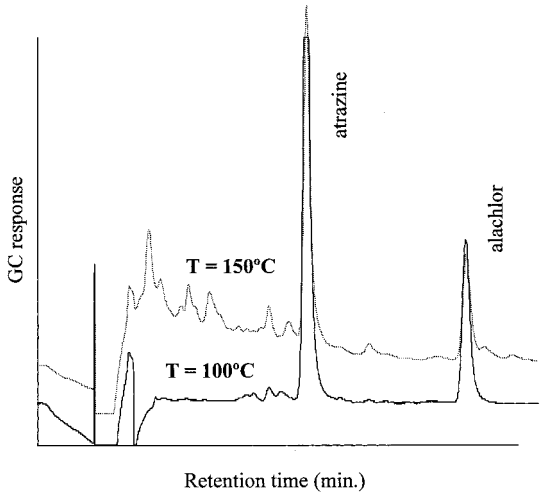

FIGURE 2. Effect of ASE extraction temperature on GC background signals. Samples were Webster clay loam spiked with atrazine and alachlor, incubated at 20 °C for 6 months.

Comparison of ASE with Soxhlet and Solvent−Shake Extractions. Method comparison was made for ASE, Soxhlet, and solvent−shake extractions for their efficiency to recover atrazine and alachlor from all of the four soils that were incubated for different lengths of time. Dichloromethane−

TABLE 4. Atrazine Concentrations in Soil (mg kg^{-1}) Determined after Extraction Using Different Methods (Labeling of Different Letters Denotes Difference at $P = 0.05$ for Samples Taken at the Same Time after Treatment)

treatment	ASE	Soxhlet	shake
2-week Incubation			
Webster CL	8.65 ± 0.21^{a}	9.21 ± 0.12^{a}	8.26 ± 0.39^{a}
Waukegan SL	7.54 ± 0.18^{a}	9.21 ± 0.37^{a}	8.48 ± 0.26^{a}
Linne CL	8.79 ± 0.16^{a}	$7.98 \pm 0.37^{a,b}$	8.02 ± 0.19^{b}
Arlington SL	10.42 ± 0.09^{a}	9.86 ± 0.25^{a}	10.54 ± 0.33^{a}
8-week Incubation			
Webster CL	5.41 ± 0.19^{a}	4.54 ± 0.09^{b}	3.63 ± 0.13^{c}
Waukegan SL	5.02 ± 0.15^{a}	4.34 ± 0.25^{b}	2.96 ± 0.23^{c}
Linne CL	6.72 ± 0.26^{a}	6.29 ± 0.13^{b}	5.38 ± 0.32^{c}
Arlington SL	3.60 ± 0.31^{a}	3.05 ± 0.14^{b}	2.33 ± 0.09^{c}
26-week Incubation			
Webster CL	2.55 ± 0.04^{a}	2.35 ± 0.08^{b}	1.78 ± 0.22^{c}
Waukegan SL	1.41 ± 0.07^{a}	1.10 ± 0.07^{b}	0.73 ± 0.08^{c}
Linne CL	5.85 ± 0.06^{a}	5.36 ± 0.11^{b}	4.30 ± 0.33^{c}
Arlington SL	1.270 ± 0.084^{a}	1.243 ± 0.047^{a}	1.137 ± 0.319^{a}

TABLE 5. Alachlor Concentrations in Soil (mg kg^{-1}) Determined after Extraction Using Different Methods (Labeling of Different Letters Denotes Difference at $P = 0.05$ for Samples Taken at the Same Time after Treatment)

treatment	ASE	Soxhlet	shake
2-week Incubation			
Webster CL	5.67 ± 0.58^{a}	6.42 ± 0.18^{a}	6.98 ± 0.39^{b}
Waukegan SL	5.73 ± 0.38^{a}	5.69 ± 0.25^{a}	6.33 ± 0.20^{b}
Linne CL	2.98 ± 0.11^{a}	3.17 ± 0.09^{ab}	3.21 ± 0.15^{b}
Arlington SL	6.55 ± 0.22^{a}	7.34 ± 0.30^{b}	7.38 ± 0.18^{b}
8-week Incubation			
Webster CL	5.27 ± 0.22^{a}	4.41 ± 0.19^{b}	3.90 ± 0.07^{c}
Waukegan SL	3.85 ± 0.15^{a}	3.19 ± 0.12^{b}	2.54 ± 0.15^{c}
Linne CL	0.62 ± 0.03^{a}	0.50 ± 0.03^{b}	0.46 ± 0.05^{c}
Arlington SL	5.13 ± 0.19^{a}	4.52 ± 0.08^{b}	4.28 ± 0.11^{c}
26-week Incubation			
Webster CL	3.32 ± 0.12^{a}	3.05 ± 0.11^{b}	2.68 ± 0.26^{c}
Waukegan SL	1.84 ± 0.09^{a}	1.70 ± 0.05^{b}	1.21 ± 0.31^{c}
Linne CL	0.29 ± 0.05^{a}	0.25 ± 0.04^{a}	0.37 ± 0.17^{a}
Arlington SL	2.22 ± 0.04^{a}	2.24 ± 0.06^{a}	1.87 ± 0.18^{b}

acetone was used as the solvent for ASE extraction, and the temperature was 100 °C, pressure 1500 psi, and static time 15 min. These conditions were within the optimal ranges that were established in the above experiment. Atrazine concentrations determined after different extractions for the four soils are given in Table 4, and those of alachlor are given in Table 5. As the time of incubation increased, atrazine and alachlor gradually dissipated in soil, and the rate of dissipation was different among different soils. For instance, atrazine was most persistent in Linne clay loam, while in the same soil alachlor largely disappeared 8 weeks after treatment. Herbicide dissipation could be caused by chemical and biochemical transformation of the parent molecule. It is likely that some of the dissipation could also be attributed to decreased extractability due to pesticide aging.

For the 2-week samples, ASE extraction generally resulted in atrazine concentrations that were similar to those achieved by the other two methods for the same soil (Table 4). The only exception was Linne clay loam, from which more atrazine was recovered by ASE than by the solvent−shake method. A similar pattern was not found for the extraction of alachlor from the 2-week samples (Table 5). Alachlor concentrations determined following ASE extraction were generally similar to those obtained after Soxhlet extraction, except for the Arlington soil, from which more alachlor was recovered by Soxhlet extraction than by ASE. Shake extraction using methanol−water outperformed ASE in all soils (Table 5). These results together suggest that when the residue was relatively fresh, drastic conditions such as enhanced solvent temperature were not essential for recovering the pesticide. In fact, methods such as solvent−shake extraction at the ambient temperature may be even better, because the mild conditions during extraction would minimize the loss of analyte due to degradation and/or volatilization.

After the soils were incubated for 8 weeks, a distinctively different pattern was observed for the relative efficiency of the three extraction techniques. For both atrazine and alachlor, the efficiency of ASE was consistently better than that of Soxhlet extraction, which in turn was consistently better than that of solvent−shake extraction (Tables 4 and 5). For instance, in the Webster clay loam, the concentration of atrazine determined using Soxhlet extraction was 84% of that obtained by ASE extraction, while that by solvent−shake extraction was only 67% of that by ASE extraction. For alachlor, the corresponding percentages were 84% and 74%. After the incubation time was further prolonged to 26 weeks, the extraction efficiency demonstrated by the different methods generally still followed ASE > Soxhlet > solvent−shake, with the only exceptions being the extraction of atrazine from the Arlington soil and alachlor from the Linne soil (Tables 4 and 5). Recovery of atrazine from the Arlington soil and alachlor from the Linne soil was similar for the different extraction methods, which in part was due to the large standard deviation associated with the shake method. In Webster clay loam, atrazine concentration determined after Soxhlet extraction was 92% of that achieved by ASE extraction, while that by solvent−shake extraction was only 70% of that by ASE extraction. The corresponding percentages for alachlor recovery from the same soil were 78% and 52%.

The relative extractability of atrazine and alachlor by the different extraction methods for the 8- and 26-week samples again suggests that as pesticides age in soil, harsher conditions are needed to separate the residue from the sample matrix. Because the same solvent system was used for ASE and Soxhlet extractions, the difference in pesticide recovery between these two methods may be directly attributed to their different operational conditions. Under the selected conditions, the samples were in contact with solvent at 100 °C during ASE extraction but at only <60 °C during Soxhlet extraction. It should be also noted that since different solvents were used for ASE and solvent−shake extraction, different post-extraction procedures had to be employed, which may have contributed to the different pesticide concentrations detected. For example, because of the use of water as a part of the extraction agent, repeated partitioning with chloroform had to be performed for solvent−shake extracts. In contrast, sample extracts from ASE extraction were directly concentrated without going through such steps. The extra steps following the solvent−shake extraction could have caused additional loss of pesticides in this study. It can be argued, however, that post-extraction sample preparation is an integral part of an extraction technique, and the simplified procedures following ASE extraction should be considered as one of its meritorious features.

In addition to its improved efficiency, ASE extraction also consumed a much smaller amount of solvent compared to the other two methods. Solvent consumption for ASE extraction (50 mL) was <20% of that for Soxhlet extraction (300 mL), and 25% of that for solvent−shake extraction (~200 mL). Because of its accelerated extraction and the high degree of automation, sample throughput with ASE extraction should also be significantly better than Soxhlet extraction and in some cases better than solvent−shake extraction. Under the conditions used in this study, a total of 48 samples may be extracted on a daily basis using the ASE system. Lower solvent consumption implies reduced cost for solvent purchase and waste disposal, as well as less exposure to solvent vapor by

laboratory personnel. The high sample throughput by ASE will help to meet the requirement for handling the ever-increasing numbers of soil and other environmental samples.

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