Acta Chim. Slov. **1998**, *45*(1), pp. 1-17

(Received 20.1.1998)

USE OF SOLID-PHASE MICROEXTRACTION IN ANALYSIS OF PESTICIDES IN SOIL

Helena Prosen, Lucija Zupančič-Kralj

Faculty of Chemistry and Chemical Technology, University of Ljubljana, Aškerčceva 5, SLO-1000 Ljubljana, Slovenia

ABSTRACT

Residues of pesticides and their degradation products in soil present a serious problem for crops, soil organisms and humans. For isolation of this type of compounds solid-phase microextraction (SPME) could be used in combination with conventional extraction method. This modern separation method was optimized for extraction of organochlorine and triazine pesticides from soil samples. Analytes were desorbed from the fiber in the injector of a gas chromatograph and determined by either electron capture or mass spectrometric detection. Linearity and limits of detection were tested in the 0.1 – 20.0 ng/g range for organochlorines and 10 – 100 ng/g range for triazines. The method presented could be used for screening of pesticides in contaminated soil samples and offers a simple alternative to established methods of pesticide analysis in soil.

INTRODUCTION

Pesticide analysis in soil represents a problem mainly because of the high level of interfering compounds such as humic and fulvic acids. Moreover, pesticide residues tend to adsorb to soil particles very strongly [1], which poses the question of the

reliability of spikes used as a means of evaluating the recovery of a pesticide from soil by the chosen extraction technique.

The pesticides chosen for this study were the ones most commonly present in soil samples from our area, that is triazines, which are widely applied herbicides, and a group of chlorinated insecticides, banned years ago, the residues of which are still found in environmental samples. The most common method for isolation of these compounds from the soil is extraction with organic solvents [2], followed by extensive clean-up procedures in order to remove interferences prior to analysis. Better recoveries are obtained by use of the Soxhlet apparatus or sonication. Analysis is usually accomplished by gas chromatography - GC [3-8] or by high performance liquid chromatography - HPLC for the triazines [9-10].

However, these extraction methods are becoming increasingly unpopular for two reasons: the time-consuming clean-up procedures with great chances of analyte loses and the high consumption of high purity, expensive organic solvents, which pose a burden to the environment thus offsetting the benefits of pesticide residue analysis [11]. Therefore, new extraction methods are being introduced to this branch of analysis, the most applied of which are supercritical fluid extraction [12], and extraction with organic solvents combined with solid-phase extraction [9-10, 13-16]. These techniques are certainly a great improvement, but in our opinion, the emphasis should be on minimization of the consumption of the organic solvent, if adequate detection limits can be reached [10, 14-16].

Recently a new, completely solventless extraction method called solid-phase microextraction (SPME) has been introduced by Pawliszyn and co-workers [17], using a fused silica fiber coated with various stationary phases, similar to those in GC capillary columns, as an extraction medium. Analytes are subsequently thermally desorbed from the fiber in the GC injector.

Both types of pesticides discussed in the present paper were already found to be amenable to SPME. Organochlorine insecticides have been extracted from different water samples [3-5, 18], as well as triazine herbicides [5-8, 19-20]. The conditions of extraction were found to be important for the best performance of SPME: extraction

time, fiber type, ionic strength of the sample, etc. However, to our knowledge no attempt was made to extract these compounds from soil samples, except a single experiment [8]. In this case, the soil sample was suspended in water and the suspension extracted with an SPME fiber.

In the present work, a combination of classical extraction of soil by an organic solvent and solid-phase microextraction was applied for isolation of pesticides from soil. Subsequent analysis was accomplished by gas chromatography with either electron capture (ECD) or mass spectrometric detection (MSD) in electron impact mode (EI). Possibilities of screening tests and quantitative analysis were assessed and are discussed in the present paper.

EXPERIMENTAL

1. Materials

Triazine standards of 98-99% purity were obtained from Riedel - de Haën (Seelze, Germany). Organochlorine insecticide standards were obtained from Supelco (Bellefonte, USA) in the form of a standard pesticide mix of 16 compounds. Stock standard solutions were prepared in acetonitrile at concentrations of 500 mg/l for triazines and in hexane at concentrations of 0.2 - 1.2 mg/l for organochlorines. They were kept in a refrigerator and were found to be stable for several months. Acetonitrile was “gradient grade”, hexane “for trace organic analysis grade” and acetone “p.a. grade”, all three from Merck (Darmstadt, Germany). LC-grade water was obtained by purifying distilled water with a Milli-Q water purification system (Millipore, Bedford, USA). Sodium chloride was p.a. grade from Kemika (Zagreb, Croatia).

A manual holder for solid-phase microextraction was obtained from Supelco (Bellefonte, USA). SPME fibers for the manual holder were 100 μm polydimethylsiloxane or 85 μm polyacrylate from Supelco.

Capillary columns for gas chromatography were HP-1 12 m x 0.2 mm i.d. x 0.33 μm (GC-ECD) or HP-1 25 m x 0.2 mm i.d. x 0.11 μm (GC-MS) from Hewlett-Packard (Palo Alto, USA).

2. Chromatographic conditions

The gas chromatograph was an HP 6890 equipped with an electron capture detector (ECD) from Hewlett-Packard (Palo Alto, USA). The injection port temperatures were 280°C for triazine or 250°C for organochlorine analysis, and the detector temperature was 320°C. The oven temperature program for triazine analysis was as follows: 70°C hold for 4 min, then ramp at 25°C/min to 140°C, then ramp at 3°C/min to 190°C, then ramp at 10°C/min to 220°C and hold for 10 min. The oven temperature program for organochlorine analysis was as follows: 120°C hold for 3 min, then ramp at 30°C/min to 180°C, then ramp at 3°C/min to 220°C and hold for 2 min. Carrier gas was H_2 at a flow rate of 1 ml/min, make-up gas for ECD was N_2 at a flow rate of 30 ml/min. Injection was in the splitless mode, purge was set on after 4 min (triazines) or 3 min (organochlorines) and purge flow was 15 ml/min. Chromatograms were recorded with an HP 3395 computing integrator (Hewlett-Packard).

For GC-MS analysis, the gas chromatograph was an HP 5890 coupled to an HP 5989A mass spectrometer MS Engine (Hewlett-Packard). The injection port and GC-MS interface temperatures were 220°C and 200°C, respectively. Temperatures of the ion source compartment and quadrupole mass analyzer compartment were 250°C and 100°C. The GC oven temperature program for triazine analysis was the same as above, but the carrier gas was He at a flow rate of 0.7 ml/min. Injection conditions were the same as above.

The ionization mode was electron impact at an electron energy of 70 eV. In the scan mode, spectra were recorded in the m/z range 35-400 amu. In the single ion monitoring (SIM) mode, different m/z ion groups were monitored at different time ranges (see Tables 1 and 2). For quantification, areas of the base peaks (indicated in Table 2) were measured in both scan and SIM modes.

TABLE 1:
Ion groups for single ion monitoring (SIM) of triazines.

time range / min	ions monitored (m/z / amu)
4.0 - 12.9	145, 158, 172, 173, 186, 187, 201
12.9 - 17.0	173,186, 200, 201, 214, 215, 229
17.0 - 30.0	198, 212, 225, 240

TABLE 2:
Ions (m/z) monitored in SIM for different triazines. Those printed in italic were used for comparative quantitation in the scan mode.

compound	ions monitored (m/z / amu)	MW
desisopropylatrazine (DPA)	145, 158, *173*	173.6
desethylatrazine (DEA)	145, *172*, 187	187.6
desethylterbutylazine (DET)	145, *186*, 201	201.7
simazine (SZ)	173, 186, *201*	201.7
atrazine (AZ)	173, *200*, 215	215.7
terbutylazine (TZ)	173, *214*, 229	229.7
cyanazine (CZ)	198, 212, *225*, 240	240.7

3. Soil spiking procedure

Soil samples were partially provided by the Agricultural Institute in Ljubljana and partially collected by us at different contaminated and uncontaminated sites. All samples were air-dried, finely ground and homogenized.

Uncontaminated soil was suspended in an acetone solution of an appropriate amount of pesticides. The suspension was thoroughly stirred, then allowed to air-dry for ca. 24 h. Samples for time-dependent experiments were left in a sunny site to allow weathering of the residues.

4. Solvent extraction procedure

A soil sample (7 g) was weighed into the extraction vessel, 15 ml of acetone was added and the suspension was shaken in an ultrasonic bath for 30 min. The suspension was centrifuged at 3000 rpm for 7 min, the clear supernatant separated and evaporated on a

water bath (approx. 50°C) under a stream of N_2. The dry residue was reconstituted in either 100 µl of acetone for direct injection into the GC or in 3.5 ml (triazines) or 7 ml (organochlorines) of water for subsequent solid-phase microextraction.

5. Solid-phase microextraction procedure

Procedure for SPME was optimized using water spiked with known amount of pesticides. The conditions optimized were:

- type of the SPME fiber: 100 µm polydimethylsiloxane versus 85 µm polyacrylate
- extraction time
- effect of agitation with a magnetic stirrer
- effect of neutral salt addition.

The following SPME conditions were found to be the most efficient for the extraction of organochlorine insecticides from aqueous solution: 100 µm polydimethylsiloxane fiber, extraction time 15 min at a stirring rate of 250 rpm and no addition of salt. For extraction of triazines, the optimal SPME conditions were an 85 µm polyacrylate fiber, extraction time 30 min at a stirring rate of 150 rpm, and the aqueous solution saturated with NaCl prior to extraction. Desorption temperatures are listed under chromatographic conditions.

RESULTS AND DISCUSSION

1. Results of SPME procedure optimization

The solid-phase microextraction procedure was optimized for aqueous solutions of pesticides, separately for both groups.

Firstly, a suitable SPME coating had to be chosen. Polydimethylsiloxane (PDMS) and polyacrylate (PA) stationary phases were tested. The PA coating was more efficient in extracting the more polar triazine herbicides (see Figure 1). The difference between the coatings was even more pronounced for desalkylated triazine degradation products,

which are more polar than their parent compounds. The PDMS coating was better for extraction of organochlorine insecticides, which are less polar compared to triazines.

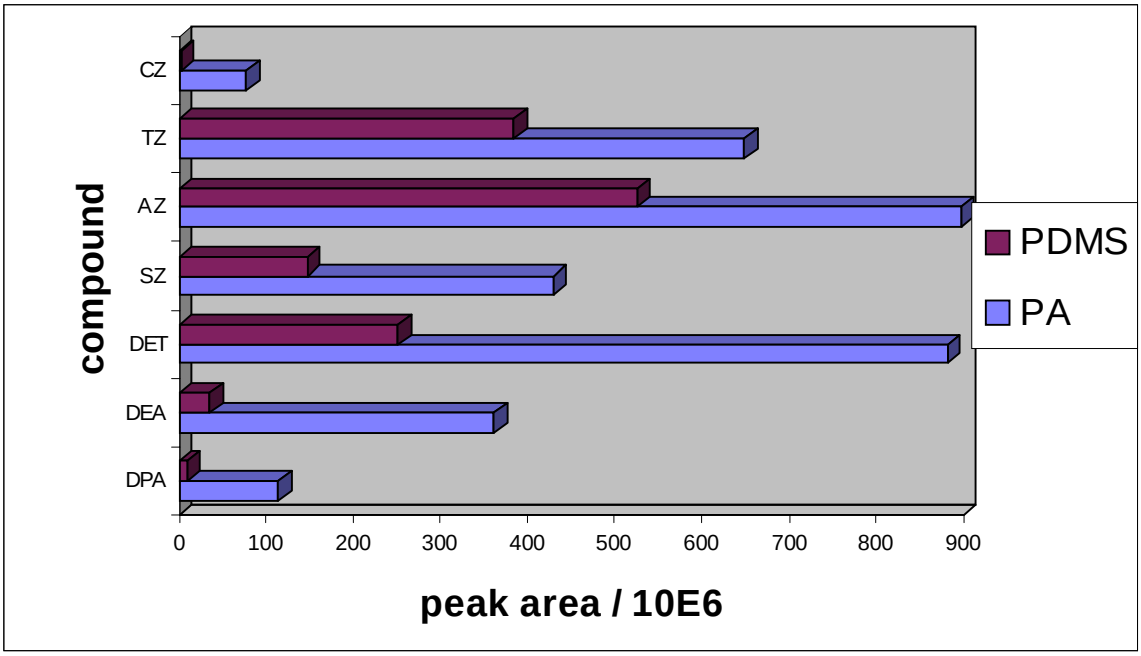

Figure 1: Comparison of polydimethylsiloxane - PDMS (100 μm) and polyacrylate - PA (85 μm) fiber coating for extraction of triazines from aqueous solution, concentration 50 ng/ml. For extraction conditions, see Experimental section. GC-MS detection. Abbrev.: DPA - desisopropylatrazine, DEA - desethylatrazine, DET - desethylterbutylazine, SZ - simazine, AZ - atrazine, TZ - terbutylazine, CZ - cyanazine.

After the initial choice of stationary phase, the effect of extraction time was assayed. This is usually recommended to be as near as possible to the equilibration time, which is much shorter if the sample is agitated. For agitated samples (magnetic stirrer at 100-250 rpm), the dependence of amount of analyte extracted by the fiber on the extraction time is shown in Figure 2. The minimum equilibration time for triazines was reported to be 60-90 min [7] or even up to 120 min [8]. In our experiments, similar results were obtained; however, an extraction time of 30 min was chosen for further work as a compromise between time and detection limits of the method.

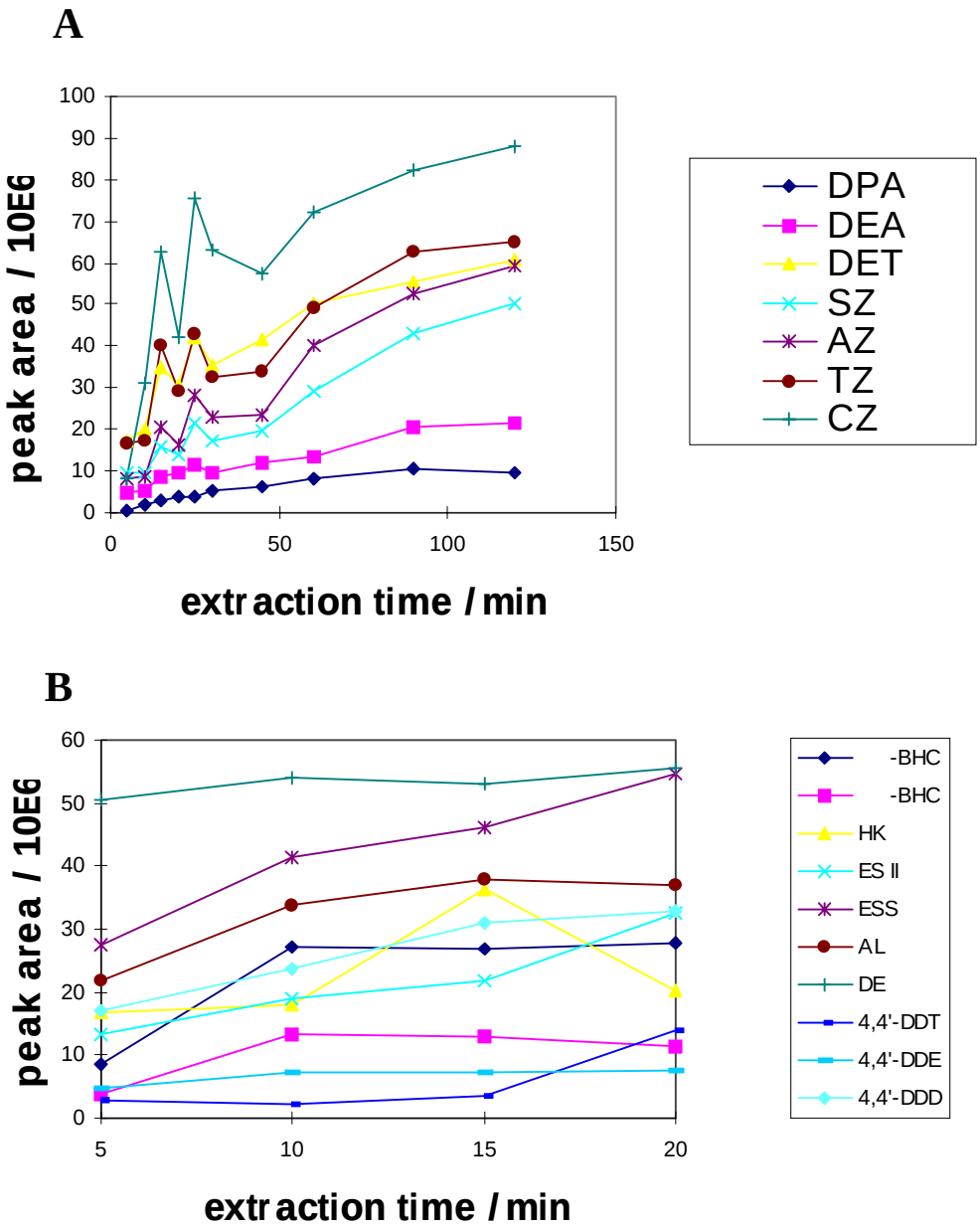

Figure 2: SPME extraction time - response (peak area) diagram for:
A - triazines, 85 μm PA fiber, 100 ng/ml in aqueous solution; for abbreviations, see Figure 1;
B - organochlorines, 100 μm PDMS fiber, 0.2-1.2 ng/ml in aqueous solution. Abbrev.: α-BHC - α-"benzenehexachloride", γ-BHC - lindane, HC - heptachlor, ES II - endosulfan II, ESS - endosulfan sulfate, AL - aldrin, DE - dieldrin.

Organochlorines are much less soluble in water, which means faster adsorption onto the fiber. Equilibration times were reported to range between 15 and 180 min, depending on the chemical structure of the compound, the fastest being for BHC isomers [18]. As is

evident from Figure 2, in our experiments no particular differences were seen between BHC isomers and other organochlorine insecticides analyzed, and therefore 15 min extraction time was chosen as suitable for extraction of these compounds.

Mixing of the sample has an effect on equilibration time and usually also a certain influence on the repeatability of the SPME extraction results. The latter is much less pronounced when using an autosampler, but in manual SPME, the fiber is not likely to be placed in the same position regarding the magnetic stirrer bar in repeated extractions. Thus, the fiber is exposed to different turbulences, resulting in a rather high RSD of the results. The repeatability of SPME for some organochlorines under mixed and non-mixed conditions are shown in Table 3. Except for lindane, the RSD is lower when the sample was not agitated, but the differences are not great enough to justify extraction from unagitated samples as this would mean more time-consuming procedure.

TABLE 3:
Comparison of detector response and RSD (3 extr.) for some organochlorines extracted from aqueous solution - concentration 80 ng/ml with SPME (100 μm polydimethylsiloxane fiber, 15 min) under mixed (magnetic stirrer, 250 rpm) and non-mixed conditions.

	non-mixed conditions		mixed conditions	
compound	peak area / 10^6	RSD	peak area / 10^6	RSD
lindane	1.8 ± 0.6	± 33 %	5.1 ± 0.8	± 16 %
heptachlor	0.5 ± 0.1	± 21 %	5.1 ± 1.6	± 31 %
4,4'-DDE	7.8 ± 1.4	± 18 %	8.7 ± 1.6	± 19 %
endrin	5.9 ± 0.9	± 16 %	9.6 ± 2.1	± 22 %
dieldrin	3.3 ± 0.6	± 18 %	7.1 ± 1.3	± 19 %

The effect of sodium chloride addition to the sample prior to extraction was also tested. As reported before, triazines are better adsorbed onto the fiber in saturated salt solution [19]. Our experiments were conducted on samples with no NaCl added or in saturated solution, and better results were obtained under the later conditions for triazines. For organochlorines, which are strongly hydrophobic compounds, no such effect was observed, and therefore further experiments were made without salt addition. Desorption conditions were chosen from the literature and were not optimized. As no cryofocusing

was available in our gas chromatograph, the desorption time was chosen to be short: 4 min at 220°C for triazines and 3 min at 250°C for organochlorines, while the head of the column was kept at 70°C for triazines and at 120°C for organochlorines. No additional peak broadening was observed under these conditions compared to direct injection and also no carry-over from the fiber. Nevertheless, the fiber was left in the GC injector after opening the purge valve for a further 5-10 min to ensure complete desorption of other contaminants from the fibre.

2. Soil extraction procedure evaluation

Extraction of pesticides from soil by SPME was reported to be successful for screening purposes, if performed from a soil suspension in water [8]. We tried to repeat the experiment with spiked soil, but found the amount of pesticides adsorbed onto the fiber too low for our detection limits, if only 0.5 g of soil were suspended in 5 ml of water. When a thicker suspension was prepared, a new problem arose: the coating of the fiber was brushed off during agitation of the sample which was necessary to maintain the suspension. As the compounds analyzed are not sufficiently volatile to be extracted from the headspace, another approach had to be chosen.

A two-step extraction procedure was designed: first, extraction of pesticides from soil by conventional means, that is, with an organic solvent; second, the solid-phase microextraction of the extraction residue in aqueous solution.

Acetone was chosen for the extraction in the first step, as it was reported before to be efficient [10, 15, 16], as well as because of its rather high volatility and low toxicity. The amount of sample analyzed had to be minimized to reduce solvent consumption. A suitable amount was found to be 7 g of air-dried soil.

The procedure was evaluated by extracting a series of prepared spiked soil samples. In Table 4, extraction recoveries for a group of organochlorines are given separately for the SPME process and the overall recovery of the method. Though the percentage of the analyte extracted by the fiber is extremely small in comparison to other extraction

methods where quantitative extraction is usually achieved, the amount of analyte on the fiber is sufficient to achieve adequate limits of detection.

TABLE 4:
Extraction recoveries (η) for organochlorine pesticides: separately for SPME only and for overall extraction procedure from soil spiked with 2 - 12 ng/g of pesticide. For extraction conditions, see Experimental section.

Compound	η (SPME)	η (overall)	compound	η (SPME)	η (overall)
α-BHC	7.3 %	0.9 %	ES sulfate	8.4 %	0.3 %
β-BHC	4.0 %	0.2 %	aldrin	4.6 %	0.1 %
γ-BHC	5.9 %	0.9 %	endrin	3.3 %	0.1 %
δ-BHC	7.4 %	0.5 %	EN aldehyde	1.6 %	0.04 %
heptachlor	5.1 %	0.2 %	dieldrin	3.6 %	0.1 %
HC epoxide	3.7 %	0.3 %	4,4'-DDT	2.0 %	0.1 %
endosulfan I	5.1 %	0.2 %	4,4'-DDE	0.9 %	0.03 %
endosulfan II	5.6 %	0.2 %	4,4'-DDD	1.5 %	0.03 %

The same soil extract in aqueous solution could be extracted by SPME over five times with good repeatability (3-10%) for triazines, but only twice for organochlorines. In the third extraction, the amount of pesticide extracted was 8-60% of the amount in the previous extractions, depending on the compound.

Another interesting, yet not surprising feature was noticed during our work. It was often observed before that spikes are not equivalent to the native analytes in soil regarding binding to soil particles and thus amenability to extraction. In our case, this was easily confirmed. Figure 3 depicts a comparison of results for triazine herbicides after the complete extraction procedure. Results are compared for soil a few days after spiking with a standard mixture and a few weeks later, after soil was left in a sunny, open site and thus partial "weathering" of the added pesticides was achieved. Though it might be possible that part of analytes could have evaporated from the sample during standing, triazines are not very volatile compounds and it seems much more likely that on weathering, they were adsorbed to soil more strongly or they even formed complexes with some soil components – e.g. humic acids [1], causing poorer extraction recoveries.

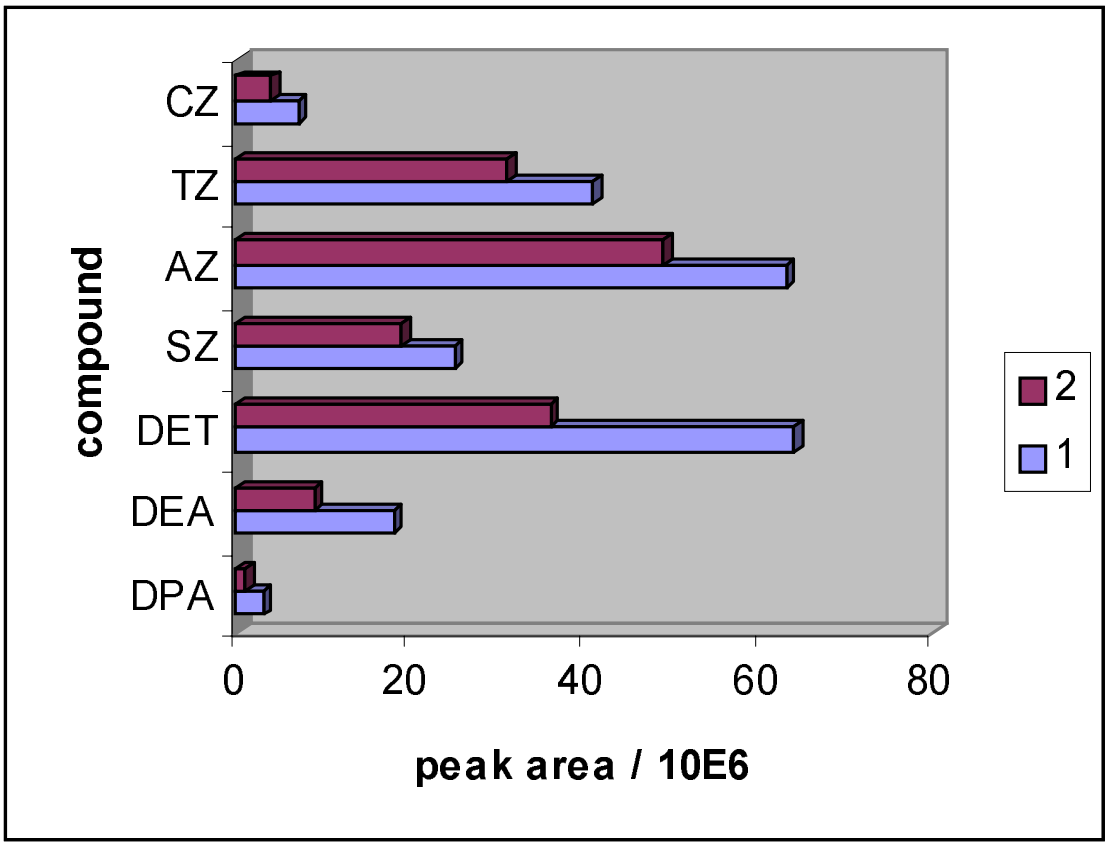

Figure 3: Comparison of peak areas (GC-MS detection) after extraction of triazine herbicides from soil spiked at 30 ng/g level. Extraction a few days after spiking (**1**) and one month after spiking (**2**). For abbreviations, see Figure 1.

3. Discussion of pesticide identification in soil

In Figure 4, ECD chromatograms of organochlorine and triazine pesticides extracted from spiked soil by the proposed method are depicted. Notice the great difference in the spiking level for the two pesticide groups at approximately the same signal / noise level. As expected, organochlorines were very well detected on an electron capture detector; whereas triazines, which were all chloro-triazines, showed a rather poor response. It was thus concluded that the EC detector is not suitable for triazine detection, while for organochlorines, retention times were repeatable enough to allow for identification of individual compounds, though this is not a completely reliable means.

Figure available in printed version only

Figure 4: Chromatograms of SPME soil extracts recorded with electron capture detector. **A** - soil spiked with organochlorines at 1 ng/g level, integrator attenuation 9. **B** - soil spiked with triazines at 50 ng/g level, integrator atten. 10. For abbreviations, see Figures 1 and 2, except: EN - endrin, ENA - endrin aldehyde

Triazine herbicides were detected using a mass spectrometer in the scan mode. Figure 5 depicts a comparison of GC-MS generated chromatograms after injecting an acetone solution of a soil extract and after extracting the same soil extract in water with an SPME fiber. Though the signals in chromatogram are lower in the second case, the chromatogram is much cleaner and the spectra obtained for the analyzed compounds were free of interferences and amenable to computer identification down to the level of approximately 20 ng of pesticide/g of soil.

Figure available in printed version only

Figure 5: Chromatograms of soil extracts recorded with MS in scan mode. Soil spiking level for triazines: 50 ng/g. **A** - soil extract after solvent extraction only, reconstituted in acetone. **B** - soil extract reconstituted in water after extraction with SPME. For abbreviations, see Figure 1.

4. Discussion of pesticide quantification in soil

As said before, part of the investigated compounds is strongly bonded in the soil and cannot be extracted. Thus, the method presented could be used only for the non-bonded part of the pesticides in soil. Some data are presented here regarding the linearity and detection limits. To our knowledge, there are no standard materials for analysis of pesticides in soil, so recoveries, linearity, and detection limits were tested using only spiked samples.

Organochlorines were spiked at the 0.1-20.0 ng/g level. Linearity was quite good ($r > 0.98$ for most compounds), while limits of detection calculated after the method of Winefordner and Long [21] ranged from 0.6 - 5.2 ng/g of soil, except for the analytes showing a poorer response on the ECD (4,4'-DDD, 4,4'-DDT, endosulfan sulfate, endrin aldehyde), which also had to be spiked at a higher level. Some real-life samples were analyzed, including a heavily contaminated field sample containing 30 ± 10 ng/g of DDT.

For triazine analysis, quantification was achieved using single ion monitoring (SIM) mass spectrometric detection. Soil was spiked at the 10-100 ng/g level. The linearity of the method was good ($r > 0.99$), and the limits of detection calculated after the method of Winefordner and Long [21] were 10-15 ng/g, which is acceptable for the level of these compounds usually found in field samples.

Some field samples were analyzed, but levels of triazines were below detection limits except in one case, when a triazine degradation product was found (Figure 6), but was not quantified because of the lack of a suitable standard compound.

Figure available in printed version only

Figure 6: GC-MS (scan mode) generated chromatogram of a real-life soil sample after extraction procedure. A triazine degradation product (marked **X**) was detected (spectrum shown below).

CONCLUSIONS

The method presented offers a simple alternative to established methods of pesticide analysis in soil. In this case SPME is used after transfer of pesticides from soil to aqueous solution, so time-consuming clean-up techniques for organic extracts are eliminated. Though the recoveries cannot be compared to conventional methods, the results show the suitability of the method in terms of identification and quantification of the analytes. These are analyzed by GC with either ECD or MSD. The former is very suitable for organochlorines, while the latter was used to detect triazines, where identification of compounds through mass spectra was possible. To achieve satisfactory limits of detection of target compounds, the SIM mode had to be used. Due to its simplicity and short time of analysis, the described method could be used for quick screening for pesticides in the contaminated soil in GC-MS scan mode.

REFERENCES

[1] A. Heintz, G.A. Reinhardt, *Chemie und Umwelt*, 4th ed., Vieweg Lehrbuch, Germany, 1996, pp. 228.
[2] H.-P. Thier, H. Zeumer, *Manual of pesticide residue analysis* Vol. I, DFG-VCH Publishers, Weinheim, Germany, 1987, pp.265-295.
[3] R. Young, V. Lopez-Avila, and W.F. Beckert, *J. High Resol. Chromatogr.* **1996**, *19*, 247-256.
[4] R.E. Shirey, *J. High Resol. Chromatogr.* **1995**, *18*, 495-499.
[5] A.A. Boyd-Boland, S. Magdic, and J.B. Pawliszyn, *Analyst* **1996,** *121*, 929-937.
[6] R. Eisert, K. Levsen, *J. Am. Soc. Mass Spectrom*. **1995**, *6*, 1119-1130.
[7] R. Eisert, K. Levsen, *Fresenius' J. Anal. Chem*. **1995**, *351*, 555-562.
[8] A.A. Boyd-Boland, J.B. Pawliszyn, *J. Chromatogr. A* **1995**, *704*, 163-172.
[9] R. Schewes, F.X. Maidl, G. Fischbeck, J. Lepschy von Gleissenthall, A. Süss, *J. Chromatogr.* **1993**, *641*, 89-93.
[10] H. Prosen, L. Zupancic-Kralj, J. Marsel, *J. Chromatogr. A* **1995**, *704*, 121-130.
[11] H. Steinwandter, *Fresenius' J. Anal. Chem*. **1992**, *343*, 604-606.
[12] E.G. van der Velde, M. Dietvorst, C.P. Swart, M.R. Ramlal, P.R. Kootstra, *J. Chromatogr. A* **1994**, *683*, 167-174.
[13] H. Weil, K. Haberer, *Fresenius' J. Anal. Chem*. **1991**, *339*, 405-408.
[14] M.S. Mills, E.M. Thurman, *Anal. Chem*. **1992**, *64*, 1985-1990.
[15] M..J. Redondo, M.J. Ruiz, R. Boluda, G. Font, *Chromatographia* **1993**, *36*, 187-190.
[16] M.J. Redondo, M.J. Ruiz, R. Boluda, G. Font, *J. Chromatogr. A* **1996**, *719*, 69-76.
[17] Zh. Zhang, M.J. Yang, J. Pawliszyn, *Anal. Chem*. **1994**, *66*, 844A-853A.
[18] S. Magdic, J.B. Pawliszyn, *J. Chromatogr. A* **1996**, *723*, 111-122.
[19] T.K. Choudhury, K.O. Gerhardt, T.P. Mawhinney, *Environ. Sci. Technol*. **1996**, *30*, 3259-3265.
[20] I.J. Barnabas, J.R. Dean, I.A. Fowlis, S.P. Owen, *J. Chromatogr. A* **1995**, *705*, 305-312.
[21] G.L. Long, J.D.Winefordner, *Anal. Chem*. **1983**, *55*, 713A-724A.

Preostanki pesticidov in njihovih razgradnih produktov v prsti predstavljajo resen problem za posevke, organizme v prsti in ljudi. Za izolacijo te vrste spojin lahko uporabimo mikroekstrakcijo na trdni fazi (solid-phase microextraction – SPME) v kombinaciji z običajno ekstrakcijsko metodo. To moderno separacijsko metodo smo optimizirali za ekstrakcijo kloriranih in triazinskih pesticidov iz vzorcev prsti. Analizirane spojine smo desorbirali z vlakna v injektorju plinskega kromatografa in določili z detektorjem na zajetje elektronov (electron capture detector – ECD) ali z masnim spektrometrom (MS). Linearnost in meje detekcije smo testirali v območju 0.1–20.0 ng/g za klorirane insekticide in v območju 10–100 ng/g za triazine. Predstavljeno metodo lahko uporabimo za pregledno analizo (screening) na pesticide v onesnaženih vzorcih prsti. Omogoča preprostejši pristop k analizi pesticidov v prsti kot že uveljavljene metode.