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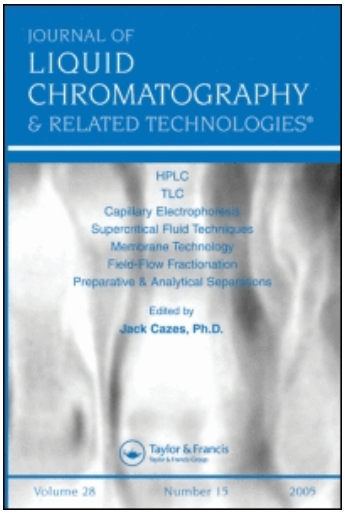

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Tariq Siddique[a]; Zahir A. Zahir[b]; William T. Frankenberger Jr.[a]
[a] Department of Environmental Sciences, University of California, Riverside, California, USA [b] Department of Soil Science, University of Agriculture, Faisalabad, Pakistan

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Reversed-Phase Liquid Chromatographic Method for Analysis of Endosulfan and Its Major Metabolites

Tariq Siddique,[1] Zahir A. Zahir,[2] and William T. Frankenberger Jr.[1,*]

[1]Department of Environmental Sciences, University of California, Riverside, California, USA
[2]Department of Soil Science, University of Agriculture, Faisalabad, Pakistan

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ABSTRACT

A high performance liquid chromatographic (HPLC) method suitable for the analysis of endosulfan and its metabolites in water and soil is described. Endosulfan is a contact and stomach insecticide, which is extremely toxic to fish, birds, and mammals. This method utilizes a μBondapak C_{18} reversed-phase column and acetonitrile : water (70 : 30 v/v) or methanol : water (70 : 30 v/v) as a mobile phase. The maximum absorbance spectrum was obtained at 214 nm. Both mobile phases resolved the endosulfan isomers and their metabolites well (resolution $R_S > 1.5$). Acetonitrile recovered α-endosulfan (106 and 90%)

*Correspondence: William T. Frankenberger Jr., Department of Environmental Sciences, University of California, Riverside, CA 92521, USA; E-mail: william.frankenberger@ucr.edu.

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and β-endosulfan (103 and 80%), while methanol extracted α-endosulfan (91 and 81%) and β-endosulfan (72 and 56%) from water and soil slurry, respectively. Recovery of endosulfan sulfate, endosulfan diol, and endosulfan ether with acetonitrile was 99, 90, and 101% in water and 91, 81, and 90% in a soil slurry, respectively. The method has high selectivity towards the major metabolites of endosulfan. The method was optimized in extraction and chromatographic detection by HPLC.

Key Words: HPLC; α-endosulfan; β-endosulfan; Endosulfan diol; Endosulfan sulfate; Endosulfan ether.

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INTRODUCTION

The chlorinated cyclic sulfite diester endosulfan (6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,3,4-benzo-dioxathiepin-3-oxide) is a cyclodiene insecticide possessing a relatively broad spectrum of activity. It is used extensively, throughout the world, as a contact and stomach insecticide and as an acaricide on field crops, vegetable, and fruit crops.[1] It is extremely toxic to fish and aquatic invertebrates[2] and has been implicated in mammalian gonadal toxicity,[3] genotoxicity,[4] and neurotoxicity.[5] The technical grade of endosulfan is a mixture of two stereoisomers, α- and β-endosulfan (in a ratio of 7 : 3), which are transformed (chemically or biologically) into endosulfan sulfate, endosulfan diol, and endosulfan ether (Fig. 1). Of the above mentioned metabolites, endosulfan sulfate is equally persistent and toxic to mammals as the parent compound, endosulfan.

Several methods have been developed for the estimation of technical-grade endosulfan and its metabolites by gas chromatography (GC) equipped with an electron capture detector (ECD).[6–9] Different organic solvents are used to extract endosulfan but all the procedures involve complex steps for sample extraction and preparation yielding long processing time. Some endosulfan metabolites are polar and need derivatization prior to GC analysis. Endosulfan diol and endosulfan hydroxy ether are more polar than endosulfan, and are derivatized by adding *N*,O-*bis*(trimethylsilyl) acetamide (BSA) to organic extracts. Trimethyl silylation improves the chromatographic behavior of endosulfan metabolites.[7,10,11] The derivatization of samples requires an additional step in the procedure for precise detection of metabolites by GC.

High performance liquid chromatography (HPLC) has been successfully used for the separation and detection of many environmental organic pollutants. High performance liquid chromatography has many advantages over GC because it permits the simultaneous analysis of acidic, basic, neutral, ionic, and thermally unstable pesticides. Moreover, this technique allows the

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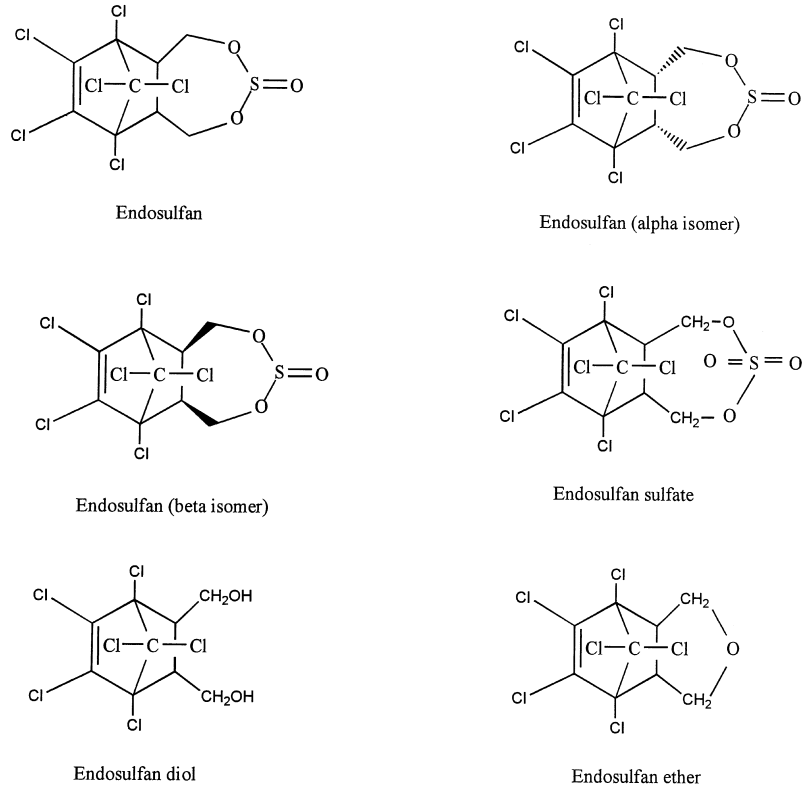

Figure 1. Endosulfan and its major metabolites.

determination of many polar pesticides and metabolites avoiding pre-derivatization and minimizing sample manipulation. Ali and Aboul-Enein[12] reported the determination of DDT and DDD (chlorinated pesticides) by HPLC under reversed-phase conditions. Parrilla et al.[13] separated α- and β-endosulfan and other pesticides from environmental water samples by HPLC on a C_{18} column. They used a combination of isocratic and gradient elution with three solvents (water, acetonitrile, and methanol) to separate and determine pesticides with different solubilities.

This study is the first to report on the detection of endosulfan metabolites by HPLC extracted from soil systems. Our objective was to develop an efficient method for the separation of endosulfan isomers along with its major metabolites. In this paper, we report a novel HPLC method for the detection of endosulfan isomers (α- and β-) and their metabolites (diol, ether, and sulfate) from water and soil slurry.

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EXPERIMENTAL

Reagents and Chemicals

Technical-grade endosulfan (99.5% pure), α-endosulfan (99.5% pure), β-endosulfan (99.3% pure), endosulfan sulfate (98.0% pure), endosulfan diol (99.0% pure), and endosulfan ether (99.0% pure) were purchased from Chem Services Inc. (West Chester, PA). Technical-grade endosulfan (used commercially) is a mixture of two stereoisomers: α-endosulfan and β-endosulfan in a ratio of 7 : 3. Acetonitrile, methanol, and acetone (Burdick & Jackson, 99.9% pure, HPLC, pesticide residue analysis grade) were purchased from VWR Scientific Products, San Diego, CA.

Scanning Absorbance Spectrum of Endosulfan

The Waters dual λ absorbance detector (2487) can measure the spectrum of a sample using either a cuvette or the flow cell. Endosulfan dissolved in acetonitrile was scanned to produce an absorbance spectrum in the range 200–300 nm using a silica cuvette at a detector sensitivity of 2.0 AUFS. Two steps were followed to obtain the endosulfan absorbance spectrum: (i) zero scan, which was the reference scan that characterized the absorbance spectrum of the solvent in the cuvette; and (ii) the sample scan, an absorbance scan of the analyte in the solvent (after subtracting out the zero scan of the solvent) to provide the actual spectrum of the sample. Fifty milligram per liter endosulfan in acetonitrile was used for scanning the absorbance spectrum.

LC Instrumentation and Chromatographic Conditions

LC was performed with a Waters (Milford, MA) liquid chromatography system consisting of binary HPLC pumps (Waters 1525) with a pressure of 6000 p.s.i. and a flow capacity of 10 mL min^{-1}. Compounds were monitored using a two channel UV/VIS dual absorbance detector (Waters 2487), which operates from 190 to 700 nm, with a taperslit flow cell and separated on a μBondapak C_{18} (30 cm × 3.9 mm i.d.) column. The column temperature was ambient and injection was made with a 25 μL SGE syringe (Waters 033381) and a manual injector. Sample filtration was carried out with solvent-compatible Gelman acrodisc syringe filters of 0.20 μm GHP membrane. The mobile phase was acetonitrile : water (70 : 30 v/v) or methanol : water (70 : 30 v/v) at a flow rate of 1 mL min^{-1}. The eluent was degassed by purging with helium. The injection volume was 20 μL and solutes were detected at 214 nm.

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Chromatographic Parameters

The following chromatographic parameters were calculated to assess the quality of the chromatographic separation: capacity factor (k'), defined as:

$$k' = \frac{t_R - t_O}{t_O}$$

where t_R denotes the retention time of the respective compound, and t_O the retention time of the eluent front; and column resolution (R_S) defined as:

$$R_S = \frac{t_{R2} - t_{R1}}{0.5(W_1 + W_2)}$$

where W is the width of the solute peaks at the baseline.

Preparation of Standard Solutions

Stock solutions of endosulfan isomers and their metabolites (1000 mg L^{-1}) were prepared in acetonitrile and methanol, separately, and serially diluted as required. Samples were prefiltered (0.20 μm disc filter) before injection.

Extraction of Endosulfan and Its Metabolites from Water and Soil Slurry

Methanol, acetonitrile, and acetone : acetonitrile were used to assess their extraction efficiency for endosulfan in water and a soil slurry. Ten milliliter of water was added to a 50 mL Erlenmeyer flask. For soil extraction, each flask contained 10 mL of water and 1.5 g of a sandy loam soil (pH 7.73; 0.52% organic carbon). Flasks were spiked with 50 mg L^{-1} of endosulfan dissolved in 30 μL acetone : ethanol. The flasks were then placed on a reciprocating shaker at 180 rpm overnight. Methanol, acetonitrile, and acetone : acetonitrile (50 : 50 v/v), in an amount equal to that present in the flask, were added to different batches and labelled for each solvent. The flasks were shaken on a reciprocating shaker at 180 rpm for one hour. Solid particles were allowed to settle for 30 min and aliquotes of the supernatant were filtered through 0.2 μm syringe filters. This study was carried out in triplicate.

Acetonitrile was used to extract the endosulfan metabolites from water and soil slurry. Flasks were spiked with endosulfan metabolites (endosulfan sulfate, endosulfan diol, and endosulfan ether) at 50 mg L^{-1} of each compound. The

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same procedure, adopted for the extraction of endosulfan isomers, was used to extract the endosulfan metabolites.

RESULTS AND DISCUSSION

Reversed-phase liquid chromatography separates primarily by solubility properties. Acetonitrile : water (70 : 30 v/v) and methanol : water (70 : 30 v/v) were used as mobile phases on a reversed-phase column. High resolution of endosulfan along with its metabolites was achieved with both the mobile phases (Table 2, Fig. 2 and 3). Capacity factors (k') calculated for endosulfan isomers and their metabolites ranged from 1.50 to 4.39 when acetonitrile was used as a mobile phase. Higher k' values for the analytes (ranging between 3.55 and 10.05) using methanol as an eluent resulted in longer retention times. Different retention times of the analytes with both mobile phases are due to different polarities of the two solvents/eluents. Less polar acetonitrile produced shorter retention time with sharp peaks as compared to methanol. Background noise was not a problem with either of the mobile phases.

Wavelengths ranging from 200 to 300 nm were scanned to obtain an absorbance spectrum of endosulfan for effective resolution. The maximum absorbance spectrum was obtained at 214 nm at 2.0 AUFS (Table 1, Fig. 4). Parrilla et al.[13] effectively resolved 21 pesticides including α- and β-endosulfan on a C_{18} reversed-phase column at 212 nm.

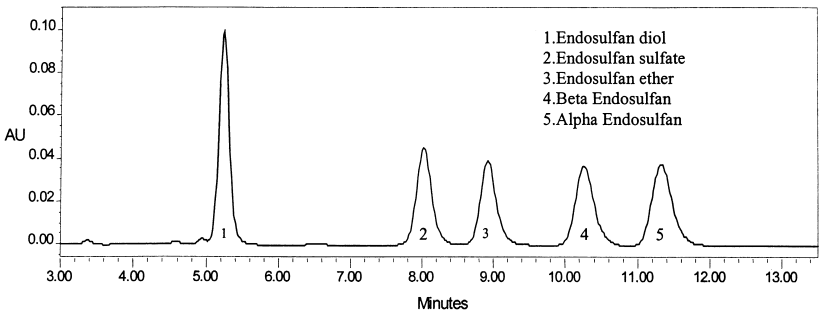

Figure 2. LC chromatogram of endosulfan and its metabolites. Analytical column, μBondapak C_{18}, 10 μm particle size, 3.9 mm × 300 mm dimension; column temperature, ambient; mobile phase, acetonitrile : water (70 : 30 v/v); flow rate, 1.0 mL min^{-1}; sample concentration, 40 mg L^{-1}; injection volume, 20 μL; detection, UV at 214 nm.

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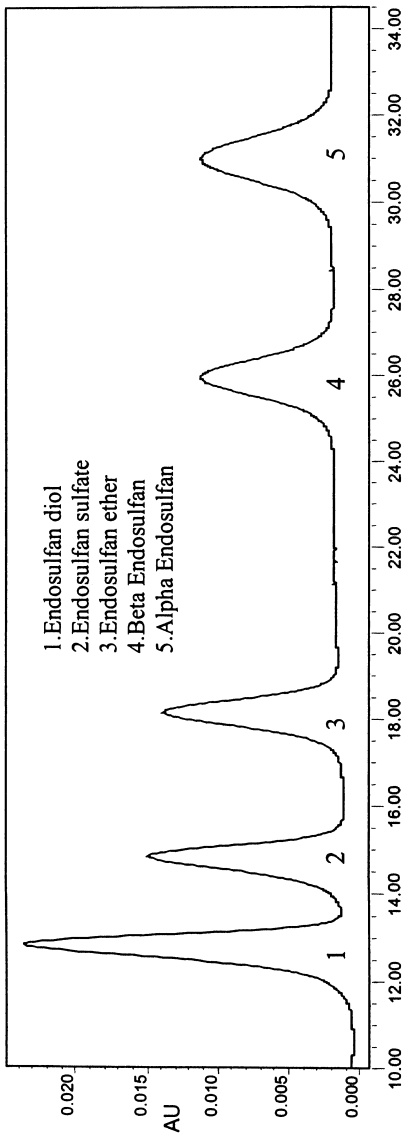

Figure 3. LC chromatogram of endosulfan and its metabolites. Analytical column, μBondapak C_{18}, 10 μm particle size, 3.9 mm × 300 mm dimension; column temperature, ambient; mobile phase, methanol : water (70 : 30 v/v); flow rate, 1.0 mL min^{-1}; sample concentration, 40 mg L^{-1}; injection volume, 20 μL; detection, UV at 214 nm.

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Table 1. Four highest peaks of endosulfan ($50\,mg\,L^{-1}$) in acetonitrile scanned under a UV range of 200–300 nm.

Peak #	Wavelength (λ), nm	Absorbance units (AU)
1	214	0.417
2	219	0.389
3	220	0.281
4	226	0.275

To establish linearity in detection, endosulfan (α- and β-endosulfan) concentrations from 2 to $4000\,mg\,L^{-1}$ were tested on the μBondapak C_{18} column. This broad range of concentrations gave good linearity ($R^2 = 0.99$). The limit of detection (LOD) with a signal-to-noise ratio of 3 was $0.5\,mg\,L^{-1}$.

Acetonitrile, methanol, and acetonitrile : acetone were used to assess their ability to extract endosulfan from water and a soil slurry (Table 3). Since acetonitrile and methanol are frequently used mobile phases in reversed-phase liquid chromatography and acetone can readily solubilize the endosulfan and its metabolites, these three organic solvents were used to extract endosulfan from water and a soil slurry. Results revealed that acetonitrile extracted higher amounts of α- and β-endosulfan from water and the soil slurry compared to methanol. Acetonitrile : acetone (50 : 50 v/v) produced almost the same results as that of acetonitrile in extracting endosulfan isomers from water and the soil slurry. Recovery of α-endosulfan (106 and 90%) and β-endosulfan (103 and 80%) from water and the soil slurry, respectively, was achieved by using acetonitrile as an extractant. Methanol recovered 91 and 81% of the α-endosulfan and 72 and 56% of the β-endosulfan from water and the soil slurry samples, respectively. Less recovery of endosulfan from the soil slurry, as compared to the water samples, is attributed to the soil adsorption capacity. Parkpian et al.[14] reported a strong relationship between endosulfan adsorption and specific soil properties using linear regression techniques. Their results revealed positive correlation coefficients (r) of 0.87, 0.84, and 0.77 for soil organic matter, cation exchange capacity (CEC), and clay contents of soil, respectively.

Acetonitrile effectively extracted all the metabolites (endosulfan sulfate, endosulfan diol, and endosulfan ether) from water and the soil slurry (Table 4). Recovery of endosulfan sulfate and endosulfan ether was 99 and 101% in water and 91 and 90% in the soil slurry, respectively. Endosulfan diol had the lowest recovery of 89.9% in water and 81.1% in soil slurry samples. This is most likely due to different solubility properties of the endosulfan metabolites.

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Table 2. Liquid chromatographic parameters for the determination of endosulfan isomers and their metabolites with acetonitrile and methanol as eluents.

	Acetonitrile (70 : 30 v/v)			Methanol (70 : 30 v/v)		
Compound	Retention time (t_R, min)	Capacity factor (k')	Resolution (R_S)	Retention time (t_R, min)	Capacity factor (k')	Resolution (R_S)
Endosulfan diol	5.25	1.50	6.70 (diol : sulfate)	12.75	3.55	1.58 (diol : sulfate)
Endosulfan sulfate	8.00	2.81	2.14 (sulfate : ether)	14.82	4.29	2.70 (sulfate : ether)
Endosulfan ether	8.93	3.25	2.92 (ether : beta)	18.20	5.50	5.38 (ether : beta)
Beta endosulfan	10.26	3.89	1.98 (beta : alpha)	25.95	8.27	2.76 (beta : alpha)
Alpha endosulfan	11.32	4.39	—	30.94	10.05	—

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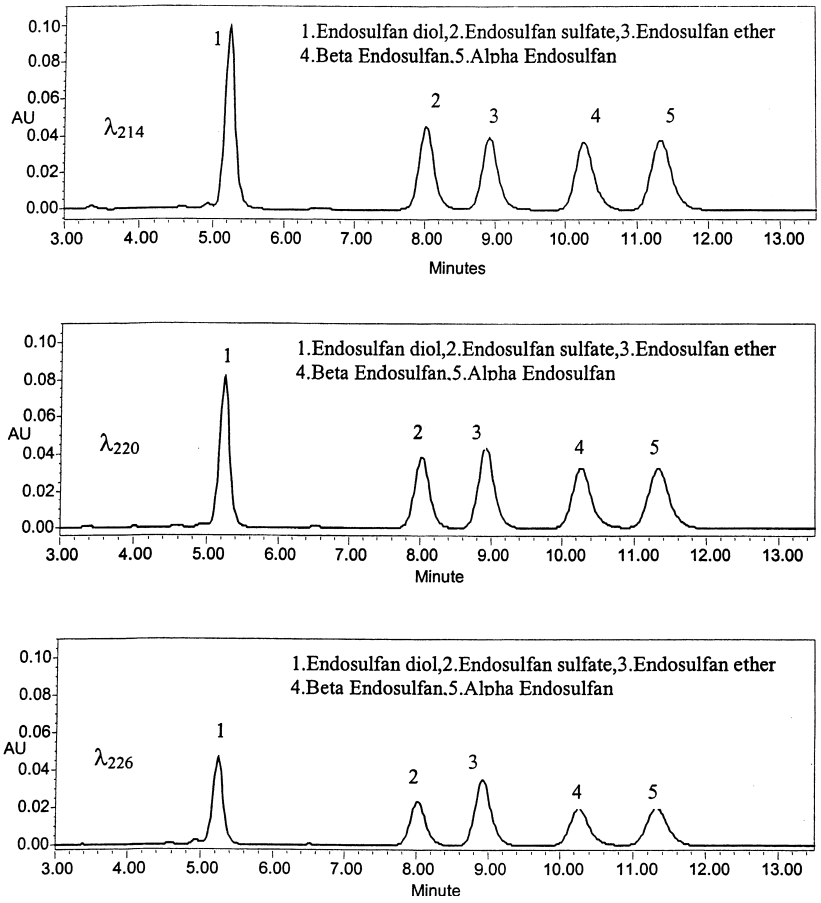

Figure 4. LC chromatograms of endosulfan isomers and their metabolites obtained by injecting 20 μL of 40 mg L^{-1} standard solution at wavelengths of 214, 220, and 226 nm.

CONCLUSIONS

A novel HPLC method described is suitable for the separation of endosulfan and its metabolites in environmental samples. Acetonitrile : water (70 : 30 v/v) and methanol : water (70 : 30 v/v) mobile phases separated all the compounds with high resolution. Acetonitrile gave the highest recovery of endosulfan isomers and metabolites from water and the soil slurry compared to methanol. Acetonitrile was used as a mobile phase for the separation of

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Table 3. Comparative efficiency of different organic solvents used to extract endosulfan from water and soil slurry.

	Liquid				Soil slurry			
	α-Endosulfan		β-Endosulfan		α-Endosulfan		β-Endosulfan	
Organic solvent	$mg\,L^{-1}$	Recovery (%)	$mg\,L^{-1}$	Recovery (%)	$mg\,L^{-1}$	Recovery (%)	$mg\,L^{-1}$	Recovery (%)
Acetonitrile	37.20 (±2.55)	106.39	15.45 (±1.31)	103.05	31.64 (±0.62)	90.49	11.96 (±1.60)	79.77
Methanol	31.88 (±1.59)	91.18	10.85 (±1.49)	72.37	28.42 (±1.11)	81.28	8.46 (±1.96)	56.42
Acetone : acetonitrile	35.69 (±1.97)	102.07	15.17 (±1.12)	101.18	30.63 (±3.26)	87.60	10.69 (±1.34)	71.30

Note: Values in parentheses show standard deviation ($n - 1$).

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Table 4. Recovery of different metabolites of endosulfan from water and soil slurry with acetonitrile.

Extraction system	Endosulfan sulfate		Endosulfan diol		Endosulfan ether	
	mg L^{-1}	Recovery (%)	mg L^{-1}	Recovery (%)	mg L^{-1}	Recovery (%)
Water	49.37 (±2.08)	98.74	44.98 (±1.40)	89.96	50.48 (±1.50)	100.96
Soil slurry	45.33 (±0.75)	90.66	40.74 (±0.13)	81.15	44.84 (±1.29)	89.68

Note: Values in parentheses show standard deviation ($n - 1$).

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endosulfan isomers and their metabolites, as well as, for extraction of these analytes. The highest detection of endosulfan and its metabolites was achieved at 214 nm at 2.0 AUFS (detector sensitivity). The method is simple, selective, reproducible, linear, and sensitive.

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