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Low density solvent based dispersive liquid–liquid microextraction with gas chromatography–electron capture detection for the determination of cypermethrin in tissues and blood of cypermethrin treated rats

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ABSTRACT

A simple and rapid method to determine the cypermethrin (CYP) insecticide in rat tissues (kidney, liver and brain) and blood has been developed for the first time using low density solvent-dispersive liquid–liquid microextraction (LDS-DLLME) followed by gas chromatography–electron capture detector (GC–ECD) analysis. Initially, tissue samples containing CYP were homoginized in acetone. Subsequently, homogenate was mixed with *n*-hexane (extraction solvent) and the mixture was rapidly injected into water. The upper *n*-hexane layer was collected in a separate microtube and injected into GC–ECD for analysis. Blood samples were diluted with ultrapure water and subjected to DLLME through similar procedure. Parameters such as type and volume of disperser and extraction solvent, salting out effect and extraction time, which can affect the extraction efficiency of DLLME, were optimized. Method was validated by investigating linearity, precision, recovery, limit of detection (LOD) and quantification (LOQ). LODs in tissue were in the range of 0.043–0.314 ng mg^{-1} and for blood it was 8.6 ng mL^{-1} with a signal to noise ratio of 3:1. LOQs in tissue were in the range of 0.143–1.03 ng mg^{-1} and for blood it was 28.3 ng mL^{-1} with a signal to noise ratio of 10:1. Mean recoveries of CYP at three different concentation levels in all the matrices were found to be in the range of 81.6–103.67%. The results show that, LDS-DLLME coupled with GC–ECD offers a simple, rapid and efficient technique for extraction and determination of CYP in rat tissues and blood samples, which in turn would be useful for toxicological studies of CYP.

1. Introduction

Pyrethroids have replaced the organophosphate and carbamate pesticides due to their low toxicity to mammals and high insecticidal activity. Cypermethrin (CYP), a synthetic pyrethroid insecticide, is commonly used in large scale commercial agricultural applications as well as in consumer products for domestic purposes. CYP is highly toxic to insects and exerts its effect by interacting with sodium channels of nerve cells. CYP is associated with choreoathetosis/salivation syndrome (CS syndrome) which involves nosing, exaggerated jaw opening, increased salivation, progressive development of chewing, muscle tremor, tonic clonic convulsion and death [1]. Various studies have been carried out to understand the toxicity of CYP in different animal models especially rodents [2–5]. Most of the studies utilized laboratory animals (such as rats and mice) to understand the neurotoxicity of CYP. CYP causes neurobehavioral toxicity including decrease in motor activity, decrease in grip strength in rats [6,7].

The analysis of CYP in biological matrices such as tissue and blood is complex and there is a need for sensitive, rapid, accurate and convinient method for the determination of CYP in rat tissues (liver, kidney, brain) and blood samples which will be useful for the toxicological studies involving CYP. Chromatographic techniques such as gas chromatography (GC) and high performance liquid chromatography (HPLC) were the most commonly used analytical instrumention for the determination of CYP in various matrices [8–11]. Many sample preparation techniques have been reported for extraction and preconcentration of CYP from various matrices which include solid phase extraction (SPE) for wines, solid-phase microextraction (SPME) for water and ultrasound assisted-dispersive liquid–liquid microextraction (US-DLLME) for pear juices [10,12–14]. The analysis of CYP in biological matrices needs extensive sample preparation, use of sorbents for cleanup and use of large amount of toxic solvents [15–17]. In recent years,

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SPME has been used as a solvent free miniaturized microextraction and preconcentration technique and widely accepted for its use by research community due to its advantages such as solvent free anlaysis, less sample consumption and simple to use, but these fibers are very fragile and expensive.

DLLME is relatively a newer, environmentally benign, simple and low cost microextraction technique [18]. This technique is based on ternary component solvent system, in which a mixture of disperser and extraction solvents are rapidly injected into the aqueous sample with the help of a syringe. The resultant cloudy solution formed consists of fine particles of extraction solvents which are dispresed into the aqueous phase. This increases the contact surface between the two phases and reduces the extraction time of analytes. DLLME has potential advantages which include the simultaneous extraction and preconcentration of target analyte, cost effectiveness and shorter extraction time when compared to other sample preparation methods repored earlier [18]. Chlorobenzene, dichloromethane, chloroform or trichloroethylene, which have higher density than water, were used as extraction solvents in DLLME. However, use of these solvents limits the complete removal of the upper phase for using the sedimented phase for GC analysis. To overcome this, low densitiy solvents were commonly employed for extraction of analytes from aqueous solution as they settle at upper phase to aqueous phase [19–25]. In recent years, DLLME has been applied to determine several environmental contamiants, such as polycyclic aromatic hydrocarbons, phthalate esters, rhamnolipids, polychlorinated biphenyls, organochlorine pesticides, triazine herbicides, metal ions, and phenols [23,26–32]. Jingjing et al. [13] have reported a method for the determiantion of cypermethrin and permethrin in pear juice by ultrasound assisted DLLME combined with gas chromatography. However, the method has used high density solvent for DLLME extraction and FID detector, which is less sensitive than ECD. Further, to the best of our knowledge, method for the quantitative determiantion of CYP in rat tissues and whole blood using low density solvent dispersive liquid–liquid microextraction (LDS-DLLME) followed by highly sensitive GC–ECD analysis has not been reported. Therefore, we developed a method for accurate and precise determination of CYP in rat tissues (kidney, liver and brain) and blood samples using DLLME/GC–ECD analysis. Further, the method has been successfully applied for the determination of CYP in rat kidney, liver and blood samples.

2. Experimental

2.1. Chemicals and reagents

All chemicals and regents used in this study were of analytical grade, unless otherwise stated. The standard CYP (purity 99.7%) was obtained from Sigma (St. Louis, MO, USA, Lot#SZBA068XV). *n*-hexane, toluene, cyclohexane, ethylacetate, acetone, methanol and acetonitrile were procured from Merck (Darmstadt, Germany). Ultrapure water was produced from Milli-Q water purification system (Millipore, Bedford, MA, USA). A stock solution of CYP of 2000 $\mu g\,mL^{-1}$ was prepared by dissolving 100 mg of CYP in 50 mL of methanol and stored at 4 °C before analysis. Working standard solutions were prepared daily by diluting the stock solution for analysis.

2.2. GC–ECD and GC–MS conditions

The chromatographic analysis was carried out on a PerkinElemer Clarus 500 gas chromatograph coupled with electron capture detector. High purity nitrogen (99.999%) was used as carrier gas and make up gas for ECD at a flow rate of 2 and 30 $mL\,min^{-1}$, respectively. CYP was separated on a DB-5 capillary column (5% phenyl 95% methylpolysiloxane, 30 m × 0.25 mm i.d. × 0.25 μm film thickness). The oven temperature was programmed at 200 °C for 3 min and ramped up to 280 °C at a rate of 45 °C min^{-1}, which was held for 10.3 min (total run time 15 min). The GC injector port was held at 280 °C and operated in split mode at a split ratio of 10:1. Electron capture detector was operated at 375 °C.

Confirmation of CYP in samples was achieved by analyzing the samples on Trace GC Ultra coupled to TSQ Quantum XLS mass spectrometer (Thermo Scientific, FL, USA). The mass spectra of CYP in samples were matched with mass spectra of CYP from NIST library. The oven temperature was initially held at 200 °C (3.0 min) and increased up to 280 °C (10.00 min). Injection was perfomed in splitless mode at a temperature of 280 °C. Helium at a flow rate of 2.0 $mL\,min^{-1}$ was used as carrier gas. Mass spectrometer was operated in full scan mode from mass range of 50 to 500 amu with positive electron impact ionization (70 eV). Transfer line and source temperature were kept at 290° and 220 °C, respectively.

2.3. Animal experiments

The male wistar rats were procured from the animal facility of Indian Institute of Toxicology Research (IITR), Lucknow, India. All animals handling procedures were performed following the regulations of Institutional Animal Ethics Committee, and with its prior approval for using the animals. The animals were housed in a 12-h day and night cycle environment with *ad libitum* availability of diet and water. The three-week-old rats were daily gavaged-treated for 2 weeks with equal volumes of vehicle (corn oil) or cypermethrin (dissolved in corn oil). The dosage of CYP for this study was 10 $mg\,kg^{-1}$. The animals were then sacrificed, tissues dissected from each group and stored at −80 °C till analysis.

2.4. LDS-DLLME procedure

Rat brain, liver and kidney (0.5 g) were homogenized in 5 mL of acetone with Polytron PE 1600E (Kinematica AG) followed by centrifugation at 10,000 rpm for 10 min. After centrifugation, supernatent was collected and concentrated up to 1 mL under a gentle stream of nitrogen. An aliquot of 300 μL of this acetone extract (which itself act as disperser solvent) containing CYP along with 100 μL of *n*-hexane (extraction solvent) were rapidly injected into 2 mL of ultrapure water with the help of a gas tight syringe (Hamilton, USA) and vortexed for 60 s. A cloudy solution of the extraction solvent, disperser solvent and water was formed immediately and CYP was transferred from acetone to *n*-hexane. The upper *n*-hexane layer (~90 μL) was taken in a separate microtube and 1 μL was injected immediately into the GC–ECD for analysis. In case of blood samples, 50 μL of blood was diluted up to 2 mL with ultrapure water and subjected to LDS-DLLME procedure as described above.

2.5. Method validation parameters

Rat brain, liver, kidney and blood samples were spiked with CYP at five different concentration levels. Blood samples were spiked at 100–1000 $ng\,mL^{-1}$. Homogenized brain, liver and kidney samples were spiked from 1.1 to 9.43 $ng\,mg^{-1}$, 0.2 to 1.36 $ng\,mg^{-1}$ and 0.5 to 2.94 $ng\,mg^{-1}$, respectively. All spiked samples were extracted and analyzed following the procedure developed. Sensitivity was evaluated in terms of limit of detection (LOD) and limit of quantification (LOQ). LOD was calculated as the lowest concentration of CYP with a signal to noise ratio of 3:1 and LOQ was calculated at a signal to noise ratio of 10:1. Repeatability and reproducibility were studied at three concentration levels for each matrix and expressed as intra-day and inter-day precision (%RSD), respectively. Intra- and

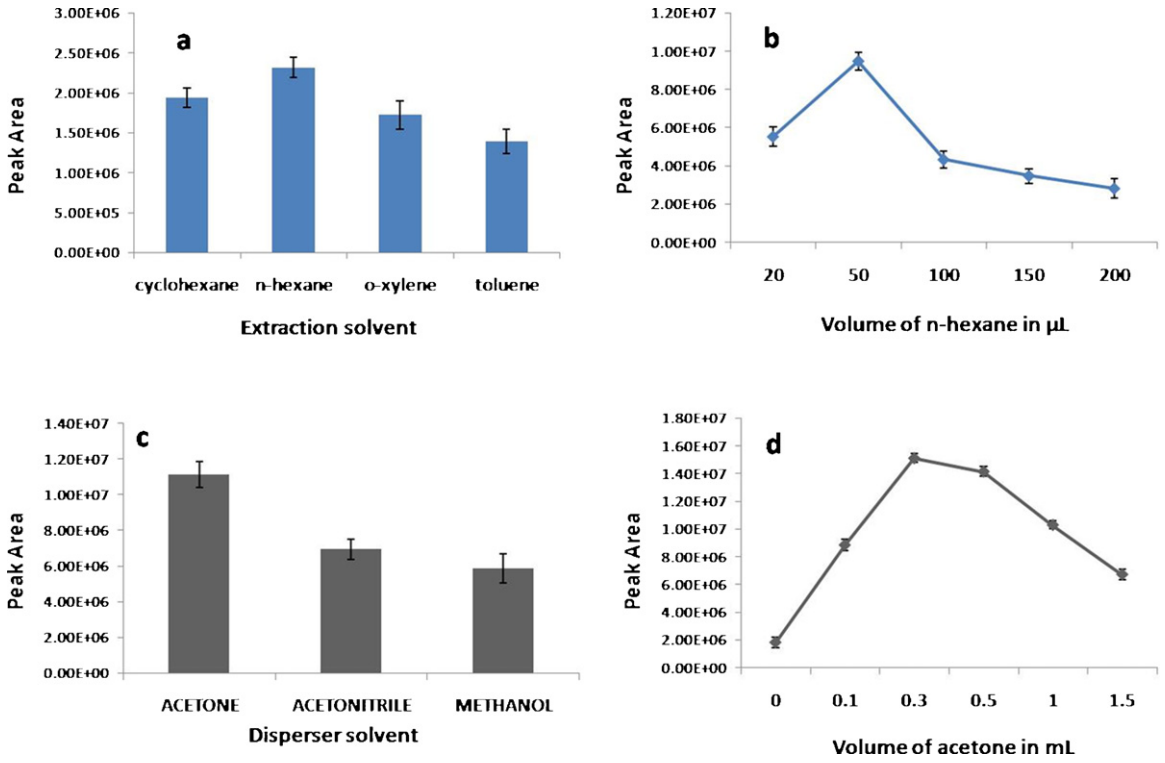

Fig. 1. Extraction parameters of LDS-DLLME procedure: (a) selection of extraction solvent, (b) effect of volume of *n*-hexane (extraction solvent), (c) selection of disperser solvent, (d) effect of volume of acetone (disperser solvent).

inter-day precision was evaluated by analyzing three replicates of each matrix spiked at three concentration levels; i.e. low, middle and high concentration levels of calibration graph and subjected to LDS-DLLME procedure [33].

3. Results and discussion

Several parameters, such as type and volume of disperser and extraction solvents and extraction time, affect the extraction efficiency of DLLME procedure. In order to obtain the best extraction recoveries and enrichment factors from each matrix, all the above mentioned parametes were optimized. All the optimization experiments were performed in triplicate and mean values were reported. Enrichment factor was calculated as $EF = C_{col}/C_0$, where EF, C_{col}, and C_0 are enrichment factor, concentration of analytes in collected phase, and initial concentration of analytes in aqueous sample, respectively [18]. C_{col} was determined by direct injection of CYP in GC–ECD system in the range from 50 to 1000 ng mL^{-1}

3.1. Optimization of extraction solvent and its volume

Type of extraction solvent has a great effect on enrichment factor of target analyte. Various organic solvents were selected on the basis of their density (d) lower than water. In this study, four organic solvents, viz. *n*-hexane ($d = 0.659$ g mL^{-1}), cyclohexane ($d = 0.779$ g mL^{-1}), toluene ($d = 0.865$ g mL^{-1}) and *o*-xylene ($d = 0.88$ g mL^{-1}) were screened. A series of experiments were performed by taking constant volume of extraction solvent (100 µL) and acetonitrile (500 µL) as disperser solvent. Recoveries of each extractant for CYP were compared using different solvents and finally we selected *n*-hexane since this yielded the highest extraction recovery (Fig. 1a).

After selecting *n*-hexane as the extraction solvent, further experiments were performed to optimize the volume of extraction solvent, which is critical for the enrichment factor of DLLME extraction. To determine the optimal volume of extraction solvent, 500 µL of acetonitrile was used as disperser solvent with different volumes of *n*-hexane (20, 50, 100, 150 and 200 µL). The measured area of the analyte increased with the increase in the volume of extraction solvent from 20 to 50 µL and then decreased at volumes higher than 50 µL (Fig. 1b). But, at volumes lesser than 100 µL, collection of the upper phase was difficult as well as easier evaporation of *n*-hexane as reported in the literature [19,23]. Therefore, a volume of 100 µL of *n*-hexane was selected for further experiments.

3.2. Selection of disperser solvent and its volume

Miscibility of disperser solvent in both aqueous sample and extraction solvent is required to achieve rapid extraction of analytes into the extraction solvent. In this study, three commonly used disperser solvents, viz. acetone, acetonitrile and methanol were tested. To select the suitable disperser solvent, 500 µL of methanol, acetronitrile or acetone was mixed with 100 µL of *n*-hexane and the mixture was rapidly injected into the aqueous sample (2 mL) spiked with 100 ng mL^{-1} of CYP and vortexed for 30 s. The peak response was maximum in acetone when compared to methanol and acetonitrile (Fig. 1c). Therefore, acetone was selected as the disperser solvent for further experiments.

Subsequently, the effect of volume of acetone was also studied. A set of experiments were performed by keeping the volume of *n*-hexane constant (100 µL) and by varying the volume of acetone (0, 0.1, 0.3, 0.5, 1 and 1.5 mL). The response of ECD increased from 0.1 to 0.3 mL, but the same decreased with volumes of 0.5 mL or greater (Fig. 1d). Further, volumes of upper organic layer decreased when acetone volume increased from 0.3 to 1.5 mL, probably due to the increasing solubility of *n*-hexane in aqueous layer. The tendency of formation of cloudy solution diminished with the increasing volume of acetone, thereby resulting in lower recoveries. Therefore, on the basis of above results, *n*-hexane and acetone were selected as

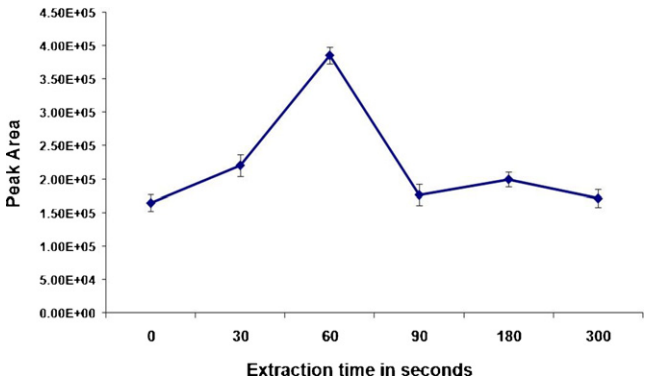

Fig. 2. Effect of extraction time on detector response.

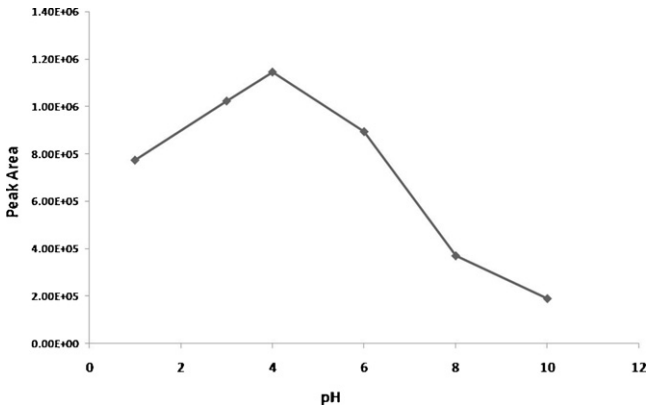

Fig. 3. Effect of pH on extraction efficiency of CYP.

extraction and disperser solvents at volumes of 100 μL and 300 μL, respectively.

3.3. Effect of extraction time

Extraction time is one of the most important parameters, which affects the extraction recovery of the analyte (CYP in this case). In DLLME, the surface area between the extraction solvent and aqueous donor phase is infinitely large, therefore equilibration is achieved very quickly [17]. Extraction time was studied at 0, 30, 60, 90, 180, and 300 s. Highest detector response was obtained, at an extraction time of 60 s, probably due to large contact area between organic phase and water sample that significantly reduces the equilibration time. Extraction time of more than 60 s resulted in decreasing extraction efficiency as evident from Fig. 2. Therefore, an extraction time of 60 s was chosen for all further experiments.

3.4. Effect of ionic strength and pH

Addition of soluble salt into the sample increases the ionic stength of the sample. Generally, salt addition decreases the solubility of target analyte in aqueous solution and increases the partitioning in organic phase. To determine if the salting out effect can enhance the extraction efficiency, different quantities of NaCl (0–10%) were added to the sample while keeping all other experimental conditions constant. No significant imrovement in the extraction recovery of CYP was observed with increasing salt concentration and hence no salt was added in any of the DLLME experiments [13]. pH is an important determinant of the extraction efficiency in DLLME procedure. To investigate the effect of pH on extraction recoveries of CYP from tissue samples, the pH of the sample phase was adjusted from 1 to 10 with 0.1 M HCl and 0.1 M NaOH. The highest extraction recoveries were obtained when the sample pH was kept at 4 (Fig. 3), at higher pH, CYP hydrolyzes in aqueous medium [12,13,34] and no upper layer was obtained.

Finally, the optimized conditions for DLLME procedure were: 2 mL of ultrapure water as dilution solvent, 300 μL of acetone extract as disperser solvent, and 100 μL of *n*-hexane as extraction solvent.

3.5. Method validation

The developed method was validated for linearity, precision, enrichment factors, LOD and LOQ in all the tissue and blood samples studied. Calibration curves for each matrix were constructed by spiking the tissue or blood samples with five different concentrations ranging from 1.1 to 9.43 ng mg^{-1} or 100 to 1000 ng mL^{-1}, respectively. Good linearity for each matrix was observed throughout the concentration range. Precision (intra-day and inter-day) was evaluated by analyzing three replicates of CYP at three different concentration levels (low, middle and high) on the same day and on five different days (Table 1). Intra- and inter-day precision was expressed as percent relative standard deviation (%RSD) and was

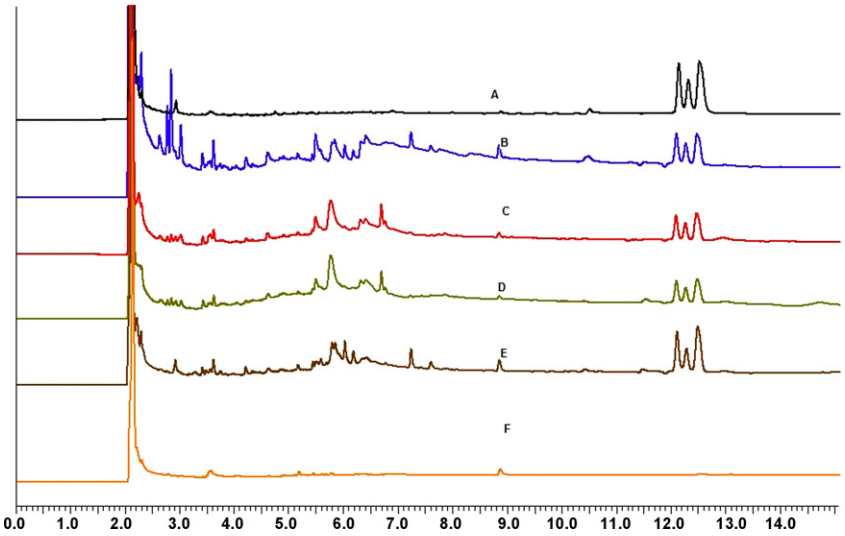

Fig. 4. GC–ECD chromatogram of (A) CYP standard, 500 ng mL^{-1}; (B) CYP in rat brain, 1.1 ng mg^{-1}; (C) CYP in rat kidney, 0.5 ng mg^{-1}; (D) CYP in rat liver, 0.2 ng mg^{-1}; (E) CYP in rat blood, 500 ng mL^{-1}; (F) and a blank run following the same DLLME procedure.

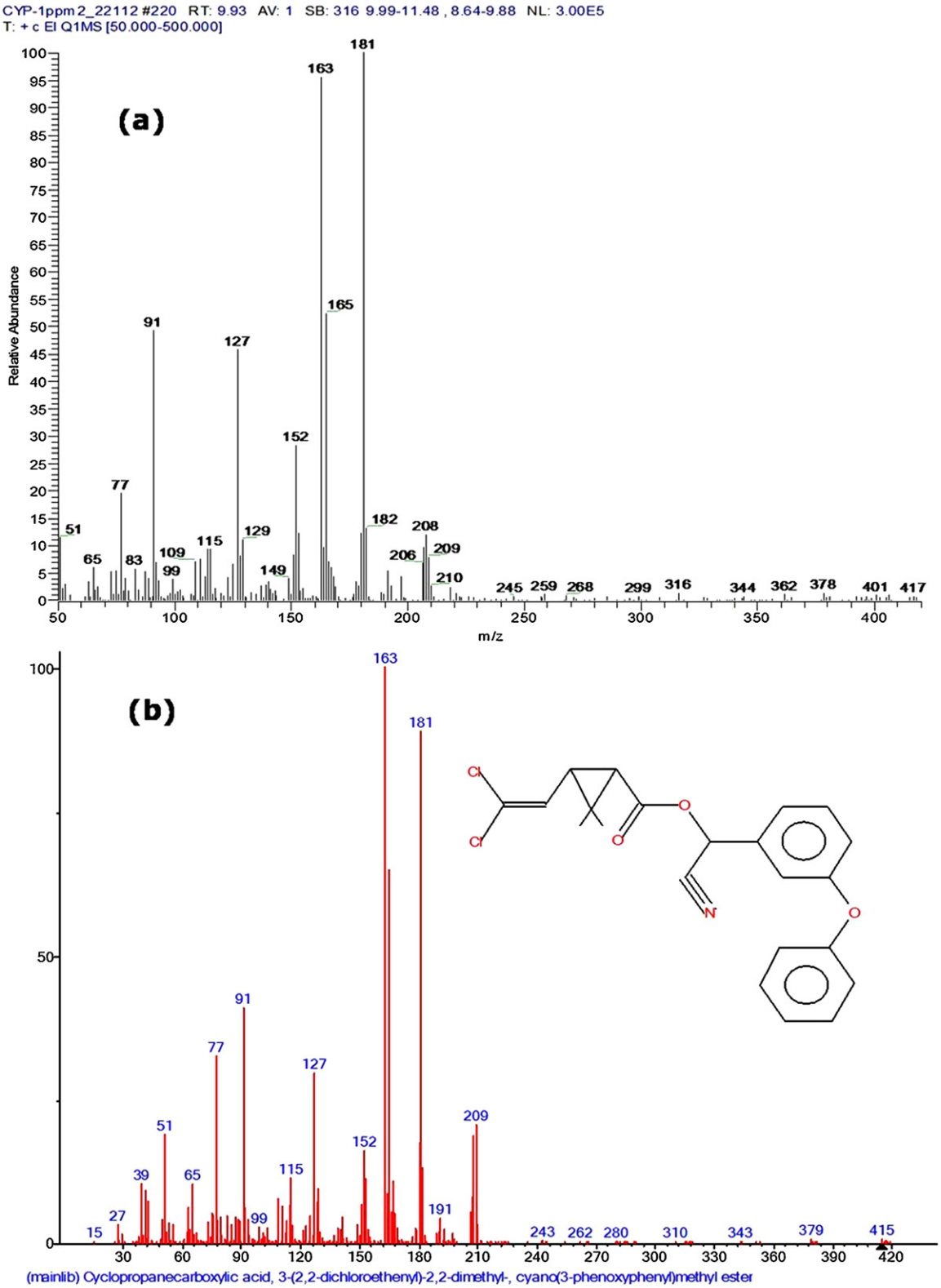

Fig. 5. (a) Mass spectra of standard CYP and (b) mass spectra of CYP from NIST library.

found to be in the range of 1.46–3.61% for intra-day and 4.61–9.17% for inter-day (Table 1). Mean enrichment factors were calculated for each matrix and were found to be in the range of 477–689 for all matrices studied (Table 1). The LODs and LOQs were calculated at a signal to noise ratio of 3 and 10. For blood sample, LOD and LOQ were found to be 8.6 ng mL^{-1} and 28.3 ng mL^{-1}, respectively. For tissue matrices LODs and LOQs were found to be in the range of 0.04–0.3 ng mg^{-1} and 0.143–1.03 ng mg^{-1}, respectively (Table 1). Recoveries of CYP in all four matrices have been studied by spiking the matrix at three different concentration levels of CYP. The mean recoveries of CYP in all matrices were found to be in the range of 81.6–103.67% (Table 2). CYP, at a concentration of 5 ng mg^{-1}, was

Table 1
Method validation parameters for CYP in different matrices.

Matrix	r^2	LOD	LOQ	Precision (%RSD)		EF
				Intra-day	Inter-day	
Blood	0.999	8.6[a]	28.3[a]	1.46	4.61	689
Brain	0.991	0.314[b]	1.03[b]	2.45	8.21	659
Liver	0.999	0.043[b]	0.143[b]	3.61	9.17	477
Kidney	0.994	0.098[b]	0.324[b]	3.37	8.94	517

EF, enrichment factor; RSD, relative standard deviation. All values are mean of triplicate analysis.
[a] Values are expressed ng mL^{-1}.
[b] Values are expressed in ng mg^{-1}.

Table 2
Recoveries of LDS-DLLME procedure.

Matrix	Spiked	Found	%Recovery
Blood[a]	100	81.6	81.6
	500	484	96.8
	1000	900.5	90.05
Brain[b]	1.1	0.942	85.63
	1.8	1.572	87.33
	2.3	2.187	95.08
Liver[b]	0.200	0.164	82
	0.680	0.625	91.91
	1.360	1.41	103.67
Kidney[b]	0.5	0.418	83.6
	1	0.907	90.7
	1.5	1.389	92.6

All values are mean of triplicate analysis.
[a] Values are expressed in ng mL^{-1}.
[b] Values are expressed in ng mg^{-1}.

Table 3
Amount of CYP found in different matrices of treated rats (10 mg kg^{-1} day^{-1}) by LDS-DLLME followed by GC–ECD analysis.

Sample	Concentration of CYP (%RSD)
Blood[a]	139 (2.15)
Liver[b]	0.285 (3.48)
Kidney[b]	0.241 (3.21)

All values are mean of triplicate analysis.
[a] Values are expressed in ng mL^{-1}.
[b] Values are expressed in ng mg^{-1}.

spiked in 0.5 g of tissue samples and homogenized with 20% KCl, water, mixture of water and acetone (1:1, v/v) and acetone. Extraction recoveries were found to be maximum when tissue samples were homogenized with acetone. Fig. 4 shows the GC–ECD chromatogram of CYP in blood, liver, kidney and brain samples spiked at different concentrations. Fig. 5a and b shows the mass spectra of standard CYP dissolved in methanol and NIST library match.

3.6. Application to real samples

To test the reliability and reproducibility of the developed method, LDS-DLLME was applied for the quantitative determination of CYP in blood and tissue samples from male Wistar rats treated with CYP. The rats were treated for 14 days at 10 mg kg^{-1} body weight with CYP. After sacrificing the rats, the samples were kept at −80 °C until analysis. The amount of CYP found in liver and blood samples are depicted in Table 3.

4. Conclusions

A simple and rapid sample preparation methodology has been developed based on LDS-DLLME and applied for the determination of CYP in rat tissues and blood samples. The method described here is easy to perform and cost effective. The developed method may find wide application for the routine analysis of CYP in various biological samples of toxicological interest.

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