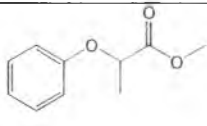
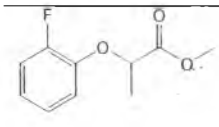
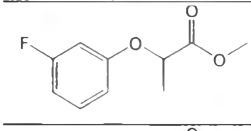
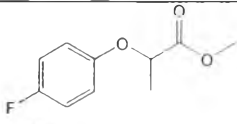
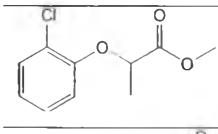
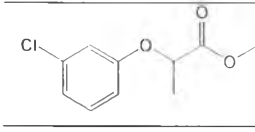
CHAPTER III

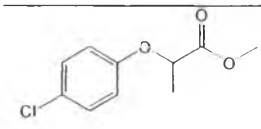
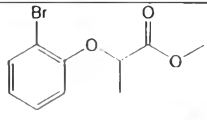
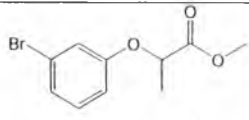
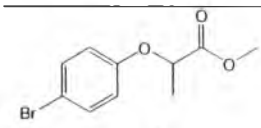
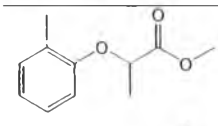
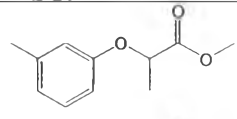
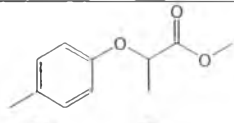
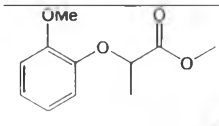
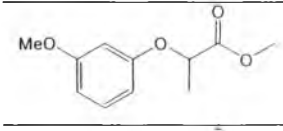
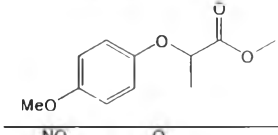
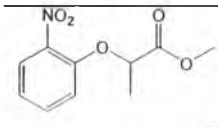
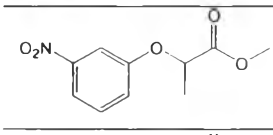
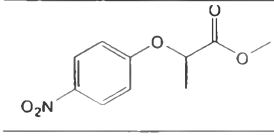
EXPERIMENTAL

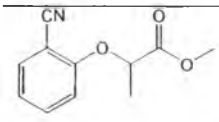
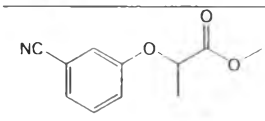
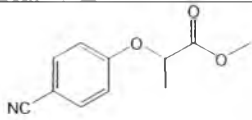
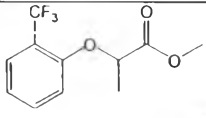
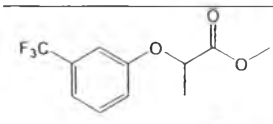
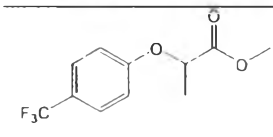
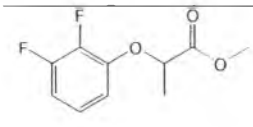
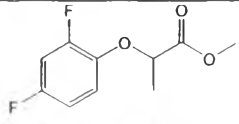
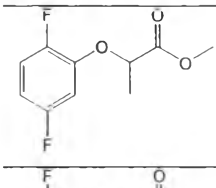
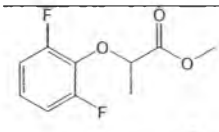
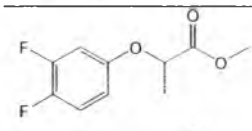
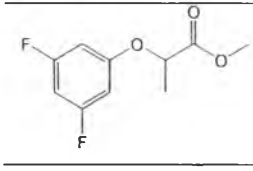
3.1 Phenoxy acid methyl esters (PAMEs)

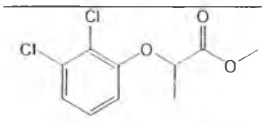
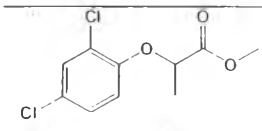
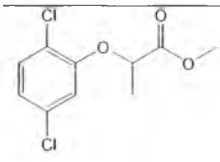
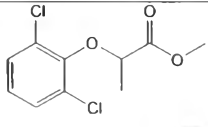
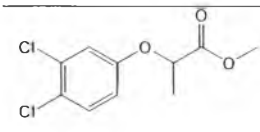
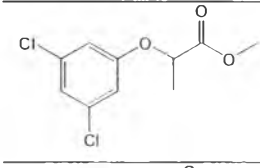
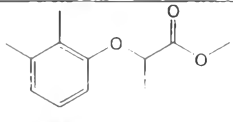
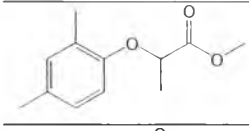
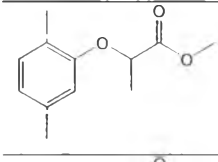
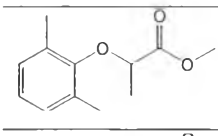
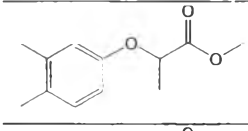
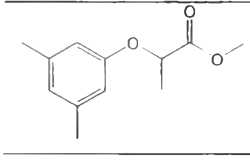
Forty-six PAMEs used in this study were previously synthesized by Rodthongkum [12]. Methyl 2-phenoxypropanoate (H) with no substitution on the aromatic ring was used as a reference analyte. Twenty-four mono-substituted PAMEs, eighteen di-substituted PAMEs and other three substituted PAMEs at different position of the aromatic ring were included to study the effect of number and position of substitution towards enantioselectivity. Structures and abbreviations of all PAMEs used in this study are shown in Table 3.1.

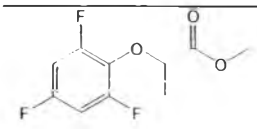
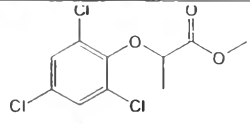
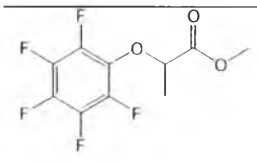
Table 3.1 Structures and abbreviations of PAMEs

chemical structure	abbreviation	compound name
	H	methyl 2-phenoxypropanoate [CAS: 2065-24-9]
mono-substituted PAMEs		
	2F	methyl 2-(2′-fluorophenoxy)propanoate
	3F	methyl 2-(3′-fluorophenoxy)propanoate
	4F	methyl 2-(4′-fluorophenoxy)propanoate
	2Cl	methyl 2-(2′-chlorophenoxy)propanoate
	3Cl	methyl 2-(3′-chlorophenoxy)propanoate

chemical structure	abbreviation	compound name
	4Cl	methyl 2-(4′-chlorophenoxy)propanoate
	2Br	methyl 2-(2′-bromophenoxy)propanoate
	3Br	methyl 2-(3′-bromophenoxy)propanoate
	4Br	methyl 2-(4′-bromophenoxy)propanoate
	2Me	methyl 2-(2′-methylphenoxy)propanoate
	3Me	methyl 2-(3′-methylphenoxy)propanoate
	4Me	methyl 2-(4′-methylphenoxy)propanoate
	2OMe	methyl 2-(2′-methoxyphenoxy)propanoate
	3OMe	methyl 2-(3′-methoxyphenoxy)propanoate
	4OMe	methyl 2-(4′-methoxyphenoxy)propanoate
	$2NO_2$	methyl 2-(2′-nitrophenoxy)propanoate
	$3NO_2$	methyl 2-(3′-nitrophenoxy)propanoate
	$4NO_2$	methyl 2-(4′-nitrophenoxy)propanoate

chemical structure	abbreviation	compound name
	2CN	methyl 2-(2′-cyanophenoxy)propanoate
	3CN	methyl 2-(3′-cyanophenoxy)propanoate
	4CN	methyl 2-(4′-cyanophenoxy)propanoate
	$2CF_3$	methyl 2-(2′-(trifluoromethyl)phenoxy) propanoate
	$3CF_3$	methyl 2-(3′-(trifluoromethyl)phenoxy) propanoate
	$4CF_3$	methyl 2-(4′-(trifluoromethyl)phenoxy) propanoate
di-substituted PAMEs		
	2,3F	methyl 2-(2′,3′-difluorophenoxy)propanoate
	2,4F	methyl 2-(2′,4′-difluorophenoxy)propanoate
	2,5F	methyl 2-(2′,5′-difluorophenoxy)propanoate
	2,6F	methyl 2-(2′,6′-difluorophenoxy)propanoate
	3,4F	methyl 2-(3′,4′-difluorophenoxy)propanoate
	3,5F	methyl 2-(3′,5′-difluorophenoxy)propanoate

chemical structure	abbreviation	compound name
	2,3Cl	methyl 2-(2′,3′-dichlorophenoxy)propanoate
	2,4Cl	methyl 2-(2′,4′-dichlorophenoxy)propanoate
	2,5Cl	methyl 2-(2′,5′-dichlorophenoxy)propanoate
	2,6Cl	methyl 2-(2′,6′-dichlorophenoxy)propanoate
	3,4Cl	methyl 2-(3′,4′-dichlorophenoxy)propanoate
	3,5Cl	methyl 2-(3′,5′-dichlorophenoxy)propanoate
	2,3Me	methyl 2-(2′,3′-dimethylphenoxy) propanoate
	2,4Me	methyl 2-(2′,4′-dimethylphenoxy) propanoate
	2,5Me	methyl 2-(2′,5′-dimethylphenoxy) propanoate
	2,6Me	methyl 2-(2′,6′-dimethylphenoxy) propanoate
	3,4Me	methyl 2-(3′,4′-dimethylphenoxy) propanoate
	3,5Me	methyl 2-(3′,5′-dimethylphenoxy) propanoate

other substituted PAMEs		
	2,4,6F	methyl 2-(2′,4′,6′-trifluorophenoxy) propanoate
	2,4,6Cl	methyl 2-(2′,4′,6′-trichlorophenoxy) propanoate
	pentaF	methyl 2-(2′,3′,4′,5′,6′-pentafluorophenoxy) propanoate

3.2 Gas chromatographic analyses

All GC separations were done on an Agilent 6890 series gas chromatograph equipped with a split injector and a flame ionization detector. Both injector and detector were set at 250 °C. Hydrogen was used as a carrier gas at an average linear velocity of 50 cm/s. Capillary columns of 0.25 mm i.d coated with 0.25 μm thick film of stationary phase were used. Two types of CD derivatives mixed with polysiloxane OV-1701 were used as CSPs.

- column ASiAc : 15.76 m long, contain 30.2% hexakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-α-CD in OV-1701
- column GSiAc : 15.19 m long, contain 36.6% octakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-γ-CD in OV-1701

Each PAME was dissolved in dichloromethane (10-20 mg/mL). Approximately 0.2-0.4 μL of solution was injected at least 2 times with a split ratio of 100:1. All separations were performed isothermally at every 10 °C from 90 °C to 220 °C. Each PAME was analyzed at least at 5 different temperatures. Retention factors (k′) and enantioselectivities (α) of all analytes were calculated from GC chromatograms.