OPTIMIZATION OF ENANTIOMERIC SEPARATION BY GAS CHROMATOGRAPHY USING BETA-CYCLODEXTRIN DERIVATIVES AS STATIONARY PHASES

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การหาภาวะเหมาะที่สุดของการแยกอิแนนทิโอเมอร์ด้วยแก๊สโครมาโทกราฟีที่ใช้อนุพันธ์บีตาไซโคลเดกซ์ทรินเป็นเฟสคงที่

น.ส.สุชาดา ม่วงศรี

วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
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Thesis Title	OPTIMIZATION OF ENANTIOMERIC SEPARATION BY GAS CHROMATOGRAPHY USING BETA-CYCLODEXTRIN DERIVATIVES AS STATIONARY PHASES
By	Miss Suchada Muangsri
Field of Study	Chemistry
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สุชาดา ม่วงศรี : การหาภาวะเหมาะที่สุดของการแยกอิแนนทิโอเมอร์ด้วยแก๊สโครมาโทกราฟีที่ใช้อนุพันธ์บีตาไซโคลเดกซ์ทรินเป็นเฟสคงที่. (OPTIMIZATION OF ENANTIOMERIC SEPARATION BY GAS CHROMATOGRAPHY USING BETA-CYCLODEXTRIN DERIVATIVES AS STATIONARY PHASES) อ.ที่ปรึกษาหลัก : ผศ. ดร.อรุณศิริ ชิตางกูร

ศึกษาการแยกคู่อิแนนทิโอเมอร์ของแอลกอฮอล์จำนวน 72 ชนิด และของเอมีนในรูปอนุพันธ์ไตรฟลูออโรแอซีทิลจำนวน 43 ชนิด ที่มีโครงสร้างแตกต่างกันด้วยแก๊สโครมาโทกราฟี ที่ใช้อนุพันธ์ไซโคลเดกซ์ทริน 2 ชนิดเป็นเฟสคงที่ คือ เฮปตะคิส(2,3-ได-โอ-แอซีทิล-6-โอ-เทอร์ต-บิวทิลไดเมทิลไซลิล)บีตาไซโคลเดกซ์ทริน (หรือ BSiAc) และ เฮปตะคิส(2,3-ได-โอ-เมทิล-6-โอ-เทอร์ต-บิวทิลไดเมทิลไซลิล)บีตาไซโคลเดกซ์ทริน (หรือ BSiMe) โดยวิเคราะห์สารแต่ละชนิดด้วยโปรแกรมอุณหภูมิและหาภาวะอุณหภูมิคงที่ที่เหมาะสุดสำหรับการแยกคู่อิแนนทิโอเมอร์ จากการศึกษาพบว่าสามารถใช้ค่า resolution และ elution temperature ที่ได้จากการวิเคราะห์ด้วยโปรแกรมอุณหภูมิ เป็นแนวทางในการหาภาวะอุณหภูมิคงที่ที่เหมาะสุดสำหรับการแยกคู่อิแนนทิโอเมอร์ได้รวดเร็วขึ้น สำหรับแคพิลลารีคอลัมน์ความยาว 15 เมตร หากได้ค่า resolution น้อยกว่า 1.0 จะไม่สามารถหาภาวะที่แยกคู่อิแนนทิโอเมอร์ได้อย่างสมบูรณ์ แต่หากได้ค่า resolution มากกว่า 3.0 มักจะหาภาวะที่แยกคู่อิแนนทิโอเมอร์ได้อย่างสมบูรณ์ได้และใช้เวลาวิเคราะห์น้อย

สาขาวิชา เคมี ลายมือชื่อนิสิต .....

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Suchada Muangsri : OPTIMIZATION OF ENANTIOMERIC SEPARATION BY GAS CHROMATOGRAPHY USING BETA-CYCLODEXTRIN DERIVATIVES AS STATIONARY PHASES. Advisor: Asst. Prof. AROONSIRI SHITANGKOON, Ph.D.

Enantiomeric separations of seventy-two underivatized chiral alcohols and forty-three trifluoroacetyl derivatives of chiral amines of various structures were investigated by gas chromatography using two types of cyclodextrin-based chiral stationary phases: heptakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-beta-CD (or BSiAc) and heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-beta-CD (or BSiMe). Each compound was analyzed by temperature program and the optimum isothermal condition for each compound was also determined. It was found that resolution and elution temperature obtained from a temperature program run could be used as a guideline for quickly determining the optimum isothermal condition for enantiomeric separation. For a 15-meter long capillary column, if a resolution less than 1.0 was obtained, a complete enantiomeric separation could not be achieved. If resolution greater than 3.0 was obtained, a complete enantiomeric separation at isothermal condition could be generally achieved with short analysis time.

Field of Study: Chemistry Student's Signature .....

Academic Year: 2019 Advisor's Signature .....

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CHAPTER I

INTRODUCTION

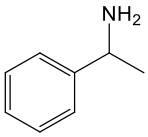
Chiral compounds have two non-superimposable mirror image forms called enantiomers. Enantiomers have identical physical and chemical properties except for the direction of rotating polarized light [1]. Two enantiomers of a chiral compound can show different bioactivity, toxicity or clinical activity. In many cases, the use of a racemic mixture of a chiral drug may be wasteful and may lead to side effect or adverse reactions [2]. Therefore, the use of optically pure active drugs is important and the determination of their enantiomeric purity is required.

Alcohols and amines are important class of compounds for chemical, pharmaceutical and agrochemical industries [3]. Chiral alcohols are natural products and useful intermediates in the synthesis of chiral molecules [4]. For example, warfarin or 1-(4′-hydroxy-3′-coumarinyl)-1-phenyl-3-butanone (**Figure 1.1**) was used in a treatment to prevent blood clots in veins or arteries. (*S*)-warfarin is a more potent anticoagulant than (*R*)-warfarin [5], [6].

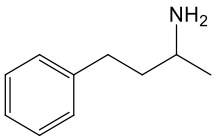
(*R*)-warfarin (*S*)-warfarin

Figure 1.1 Structures of (*R*)-warfarin and (*S*)-warfarin.

In addition, chiral amines are used as medicines, chemical intermediates and chiral reagents for diastereomeric salt formation method [7]. Examples are 1-phenylethylamine and 1-phenyl-3-butylamine (**Figure 1.2**), they are often used as chiral auxiliaries and resolving agents [8], [9].



1-phenylethylamine



1-phenyl-3-butylamine

Figure 1.2 Structures of 1-phenylethylamine and 1-phenyl-3-butylamine.

Gas chromatography (GC) is well-known and popular among many chromatographic techniques for separation of optically active drugs from a racemic mixture. For volatile and thermally stable analytes, GC based on chiral stationary phases (CSP) is preferred because of its high efficiency and sensitivity [10]. There are three major types of chiral GC stationary phases: amino acid derivatives and diamides; chiral metal complexes; and cyclodextrin (CD) derivatives [11]. CDs are chiral molecules having a cavity in their structures. This characteristic enables CD to form an inclusion complex with several types of molecules. Analytes act as a guest and can enter into the cavity of a host (or CD) by physical forces [12], [13]. CD derivatives are among the most commonly used chiral selectors in GC as enantiomeric separation of various types of compounds were reported [14]. Nevertheless, there is no guideline for selection of a suitable chiral stationary phase for the enantiomeric separation. In many cases, determination of GC condition for enantiomeric separation of a new compound is often performed by trial and error.

The objective of this work was therefore to study the enantiomeric separation of alcohols and amines by GC using β-cyclodextrin derivatives as chiral stationary phases. In this study, seventy-two chiral alcohols and forty-three trifluoroacetyl derivatives of chiral amines of various structures were investigated. Two β-cyclodextrin derivatives having different types of substitution were selected. They were heptakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or BSiAc) and heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or BSiMe). The optimum conditions for the complete enantiomeric separation of each chiral analyte on both

columns were determined. In addition, a temperature program run was performed for each chiral analyte on both columns. Chromatographic data obtained from temperature program runs and from the optimum isothermal conditions were studied for their relationship hoping that some information from the temperature program run can be used as a guideline to quickly determine the optimum separation condition.

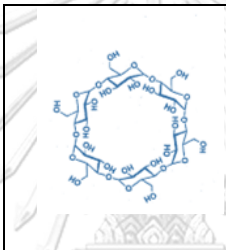
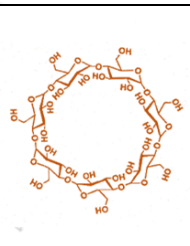
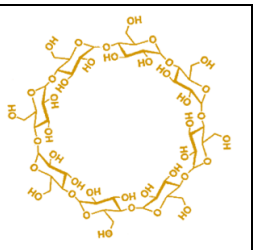
CHAPTER II

THEORY AND LITERATURE REVIEWS

2.1 Cyclodextrin [12], [13], [15]

Cyclodextrins (CDs) are cyclic oligosaccharides composed of several D-glucose units connected by α–(1,4)-glycosidic linkage. The most frequently used CDs are those with 6, 7 and 8 glucose units in their molecules and are called α–, β– and γ–CDs, respectively. Their physical properties are compared in **Table 2.1.**

Table 2.1 Properties of α–, β– and γ–cyclodextrins [15].

			
properties	**α-CD**	**β-CD**	**γ-CD**
number of glucose units	6	7	8
molecule weight (g/mol)	972	1135	1297
solubility in water at 25 °C (%w/v)	14.5	1.85	23.2
outer diameter (Å)	14.6	15.4	17.5
cavity diameter (Å)	4.7-5.3	6.0-6.5	7.5-8.3
height of torus (Å)	7.9	7.9	7.9
cavity volume ($Å^3$)	174	262	427
hydrate H_2O (cavity)	2.0	6.0	8.8
hydrate H_2O (external)	4.4	3.6	5.4

CD has a truncated cone shape with primary hydroxyl groups (C6 position) at the narrower opening and secondary hydroxyl groups (C2 and C3 positions) at the wider opening of the cavity. The interior of a CD is hydrophobic, while the exterior is

relatively hydrophilic (**Figure 2.1**). These characteristics enable the CD to form inclusion complexes with different types of analytes. A nonpolar guest analyte of appropriate size and shape can enter inside the cavity of a host, CD molecule. In addition, CD can interact with analytes with a variety of non-covalent forces, such as van der Waals forces, hydrophobic interactions and other forces, which are responsible for the formation of a stable complex.

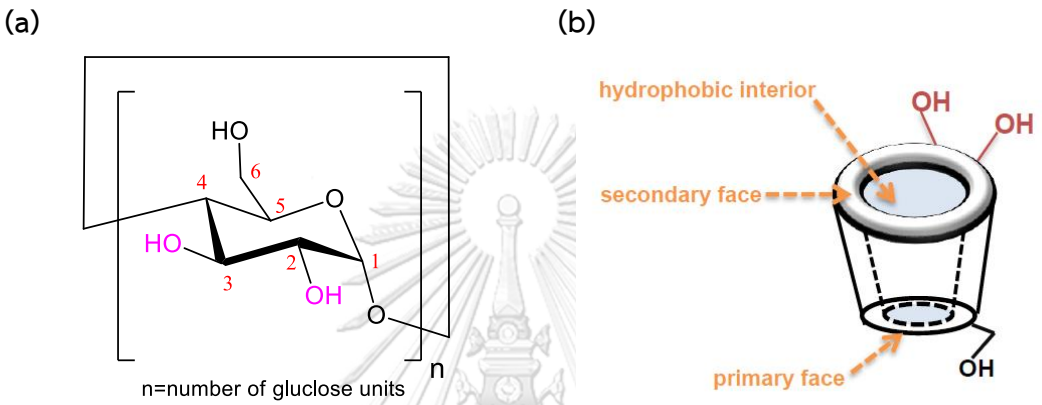


Figure 2.1 (a) Subunit of a CD molecule and (b) shape of CD [14], [16].

2.2 Gas chromatography

Gas chromatography (GC) is a chromatographic method for separating and analyzing compounds that can be vaporized without decomposition. Their sample components can be identified by comparing their characteristic retention time with reference compounds. Chromatograms obtained from GC provide both qualitative and quantitative data. GC provides high resolution, sensitivity and selectivity. GC instrumentation capable of high accuracy and precision control of both temperature and gas flow rate makes it popular for both research and routine works in most industrial and analytical chemistry laboratories [10], [17].

2.2.1 Temperature control [10], [18], [19], [20], [21]

Column temperature in GC is the important factor for successful separation and must be controlled by keeping the column in a thermostatted oven.

Isothermal conditions: The oven temperature is maintained constant throughout the analysis. This condition is mostly suitable for a sample containing components with closely similar boiling points or properties. The temperature can be adjusted to improve the separation or analysis time.

Temperature programs: The oven temperature is varied (increased) during the analysis. Temperature program is very useful for initial screening of unknown samples and is also suitable for analyses of samples containing components with a wide range of volatility. The elution temperature of an analyte can be calculated from its retention time and temperature program condition as shown below.

$$\textbf{elution temperature} = T_i + (\textbf{ramp rate} \times t_R)$$

where T_i = initial temperature and t_R = retention time of the analyte.

Temperature program run provided better peak shapes for most compounds (early eluting and late eluting compounds) over the run, whereas broad peaks for late eluting compounds were usually observed for isothermal runs. However, additional time must be paid for the temperature program run since the column must be equilibrated at initial condition before performing a new run.

2.2.2 Gas chromatographic parameters [10], [21]

retention factor (k')**:** Retention factor of an analyte can be calculated from its retention time (t_R). Large k' value indicated a strong interaction between an analyte and a stationary phase and resulted in long retention.

$$k' = \left(\frac{t_R - t_m}{t_m}\right)$$

selectivity or separation factor (α)**:** The selectivity between two peaks is expressed as a relative adjusted retention and can be calculated from their retention times.

$$\alpha = \frac{k'_2}{k'_1} = \left(\frac{t_{R,2} - t_m}{t_{R,1} - t_m}\right)$$

resolution (Rs)**:** The quality of separation of two peaks can be determined from resolution. Baseline resolution of Gaussian peaks is considered to be achieved when a resolution of 1.5 (Rs=1.5) is obtained.

$$Rs = 1.177\left(\frac{t_{R,2} - t_{R,1}}{W_{h,1} + W_{h,2}}\right)$$

$t_{R,1}$, $t_{R,2}$ = retention time, where $t_{R,2} > t_{R,1}$

$W_{h,1}$, $W_{h,2}$ = peak width at half-height

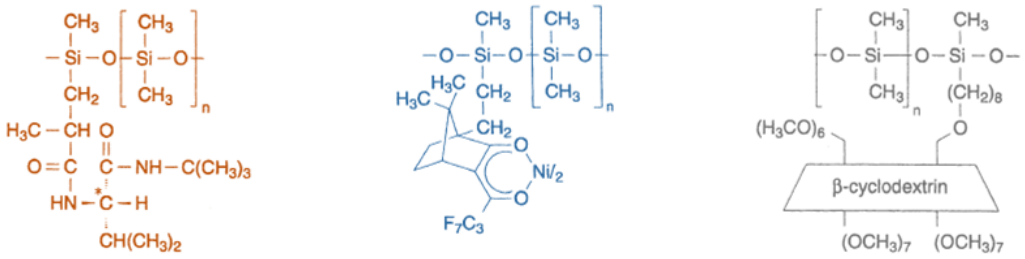
t_m = time for an unretained compound

2.3 Analyses of enantiomers by gas chromatography

The first GC enantiomeric separation using a chiral stationary phase was demonstrated by Gil-Av and coworkers in 1966 [14]. They used derivatives of amino acids as the chiral stationary phase coated on a capillary column. After that, there were many efforts to develop stationary phases containing chiral selectors of different nature with the aim of increasing the universality of a chiral column.

Stationary phases with chiral column selectors for GC can be classified in three categories [11]:

i. amino acid derivatives and diamides with potential for interacting by hydrogen bonds with certain chiral compounds
ii. chiral metal complexes which can coordinate certain analytes
iii. cyclodextrin (CD) derivatives which separate enantiomers mainly by forming host-guest complexes.



Chirasil-Val **Chirasil-nickel(II)** **Chirasil-β-Dex**

Figure 2.2 Major types of chiral stationary phases for GC.

Among three types of chiral GC selectors, it was found that CD-based stationary phases are nowadays the most widely used in direct enantiomer GC separations. CD derivatives can be used to analyze a variety of volatile substances of many areas such as organic chemistry, geochemistry, aroma and fragrances research, and food additives. Therefore, scientists are interested to further develop and study about CDs and derivatives.

From 1989, a large number of publications related to enantiomeric separations were based on the use of β-CD derivatives as chiral GC stationary phases. Several β-CD derivatives showed good selectivities for various types of compounds [22]. Examples of previous works on the use of β-CD derivatives as chiral GC stationary phases are shown as follows:

Schurig and Nowotny [23] studied the effect of polysiloxane in chiral separation. They used heptakis(2,3,6-tri-*O*-methyl)-β-CD mixed in various types of polysiloxanes as chiral stationary phases. It was found that medium polarity polysiloxane, such as OV-1701, provided higher enantioselectivities than other polysiloxanes. In addition, the column had high efficiency and was stable over wide temperature range from 25-250 °C.

Konig et al. [24] studied the characteristics of heptakis(2,3,6-tri-*O*-pentyl)-β-CD as a chiral stationary phase for GC. The results showed that cyanohydrins, polyols, diols, epoxy alcohols and hydroxy acids could be separated at low temperatures.

Beier and Holtje [25] used molecular modeling to investigate the enantioselective binding properties of chiral dihydrofuranones on heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD by capillary GC. It was found that the relationship between the β-CD and dihydrofuranones are host–guest complexes. Results obtained from this study can be used to simulate experimental data for predicting separation order or designing a new type of CD derivative.

Menestrina et al. [26] studied the enantiomeric separation of seven chiral pesticides (mecoprop, dichlorprop, fenoprop and hydroprop esters, fenoxaprop ethyl, metalaxyl and haloxyfop esters). Their separations were performed by GC using

permethyl-β-CD as a chiral selector diluted polysiloxane as a stationary phase. Three types of polysiloxanes were studied: cyanopropylphenyl-1%vinyl-86%methylpolysiloxane(OV-1701-vi); phenyl-1%vinyl-95%methylpolysiloxane (SE-54); and polyethyleneglycol (Carbowax 20 M). The results showed that permethyl-β-CD diluted in nonpolar SE-54 gave highest chiral selectivity among the three types of polysiloxanes.

2.4 Enantiomeric separation of alcohols and amines using GC

Alcohols and amines were analytes of interest. There were several types of CD derivatives used for their enantiomeric separations which can be summarized as in **Table 2.2**.

Table 2.2 Enantiomeric separation of alcohols and amines using GC.

literature	stationary phase and column dimension	chiral analytes	parameters reported
(1990); Li et al. [27]	- 2,6-di-*O*-pentyl-3-*O*-trifluoroacetyl-α–CD (10 m, 0.25 mm i.d. fused-silica capillary column) - 2,6-di-*O*-pentyl-3-*O*-trifluoroacetyl-β-CD (10 m, 0.25 mm i.d. fused-silica capillary column) - 2,6-di-*O*-pentyl-3-*O*-trifluoroacetyl-γ-CD (10 m, 0.25 mm i.d. fused-silica capillary column)	- alcohols - amines (TFA) - diols - polyols - halohydrocarbons - lactones	- temperature - **α** - k'_1
(1994); Krupčík et al. [28]	- OV-1701 (25 m, 0.30 mm i.d. fused-silica capillary column) - heptakis(6-*O*-*tert*-butyldimetylsilyl-2,3-di-*O*-acetyl)-β-CD (25 m, 0.30 mm i.d. fused-silica capillary column)	- secondary alcohols (2-butanol, 2-pentanol and 2-, 3-hexanol) in the forms of methyl, pentyl, Ac and TFA derivatives	- temperature - **α**

literature	stationary phase and column dimension	chiral analytes	parameters reported
(2000); Nie et al. [29]	- heptakis(2,6-di-*O*-nonyl-3-*O*-trifluoroacetyl)-β-CD (20 m, 0.25 mm i.d. fused-silica capillary column) - heptakis(2,6-di-*O*-dodecyl-3-*O*-trifluoroacetyl)-β-CD (20 m, 0.25 mm i.d. fused-silica capillary column)	- phenylethylamine (TFA) - alcohols - diols - carboxylic acids - amino acids - halohydrocarbons - epoxides - ketones	- temperature - α - k'_1 - thermodynamic
(2003); Ghanam et al. [30]	- permethylated β-CD with a new 11-spacer bonded to a polysiloxane backbone (19 m, 0.25 mm i.d. fused-silica capillary column)	- secondary alcohols in form underivatized and Ac derivatives	- temperature - α - t_R (*S*) and (*R*)
(2004); Juvancz et al. [31]	- permethylated-β-CD in polysiloxane (10 m, 0.1 mm i.d. fused-silica capillary column)	- arylalkyl amines (Ac and TFA) - alcohols	- temperature - first elution enantiomer - $t_{R,1}$ - α

literature	stationary phase and column dimension	chiral analytes	parameters reported
(2008); Li et al. [32]	- diproline chiral selector to a methylhydrosiloxane–dimethylsiloxane copolymer (30 m, 0.25 mm i.d. fused-silica capillary column)	- aromatic alcohols - amines (TFA)	- temperature - k'_2 - **α** - Rs
(2012); Oromi-Farrus et al. [4]	- DB-Wax (polyethylene glycol) (30 m, 0.25 mm i.d. fused-silica capillary column)	- acyclic alcohols - cyclic alcohols - diol (underivatized and Ac derivatives)	- temperature - $t_{R,1}$ - **α** - isomer form
(2015); Zhang et al [33]	- homochiral imine-linked POC (CC3-R) in polysiloxane OV-1701 (30 m, 0.25 mm i.d. fused-silica capillary column) - β-DEX 120 capillary column (30 m, 0.25 mm i.d. Supelco Inc.) - Chirasil-L-Val capillary column (25 m, 0.25 mm i.d., Agilent Technologies) - DB-17 capillary column (30 m, 0.25 mm i.d., Agilent Technologies) - HP-5 capillary column (30 m, 0.25 mm i.d., Agilent Technologies)	- alkanes - alcohols - aromatic hydrocarbons - positional isomers of - dichlorobenzenes - dibromobenzenes - chlorotoluenes - chloroanilines - bromoanilines - iodoanilines	- temperature - k'_1 - **α** - Rs

literature	stationary phase and column dimension	chiral analytes	parameters reported
(2019); Wang et al. [34]	- Chiral crystalline sponges (CCSs) in polysiloxane OV-1701 (30 m, 0.32 mm i.d. fused-silica capillary column) - Chiral crystalline sponges (CCSs) with the chiral ligand of (*S*)-mandelic in polysiloxane OV-1701 (30 m, 0.32 mm i.d. fused-silica capillary column) - Cyclosil B capillary column (30 m, 0.32 mm i.d., Agilent Technologies) - Chirasil L-Val capillary column (25 m long ×0.25 mm i.d., Agilent Technologies) - β-DEX 225 capillary column (30 m long ×0.25 mm i.d., Supelco Inc.)	-alcohols -1-phenylethylamine -nitriles -3,7-dimethyl-6-octenoic acid -benzene -lactones	- temperature - separation time - α - Rs

** Ac = acetyl; TFA = trifluoroacetyl

CHAPTER III

EXPERIMENTAL

3.1 Chiral analytes

3.1.1 Chiral alcohols

Chiral alcohols used in this study were previously prepared by Iamsam-ang [35], Konghuirob [36], Jongjitwatana [37] and Toboonpha [38]. Their corresponding ketones and sodium borohydride mixed in ethanol were refluxed for 3 hours. After work-up, the obtained alcohols were characterized by ^{1}H and ^{13}C NMR. Total of 72 chiral alcohols were used in this work and were analyzed by GC without derivatization.

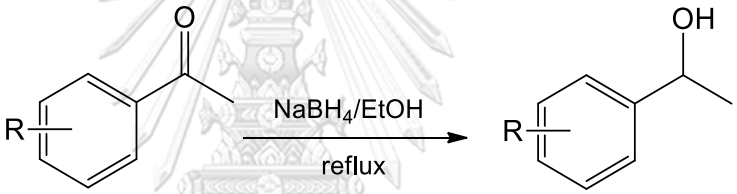
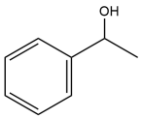
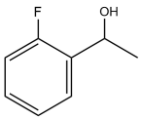
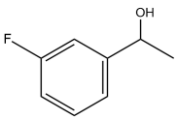
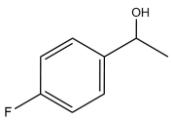
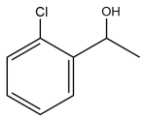
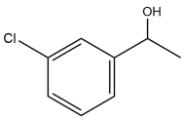
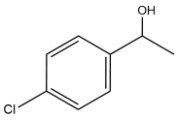
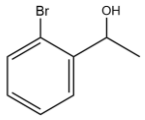
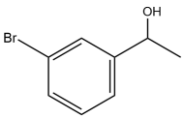
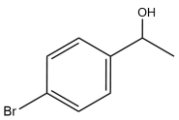
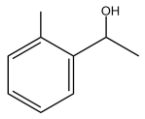
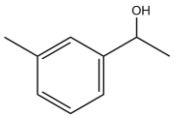
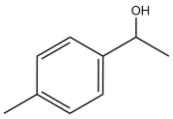
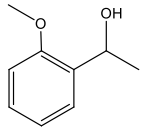
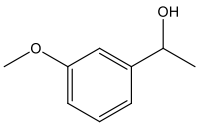
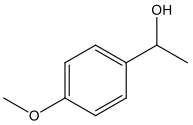
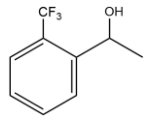
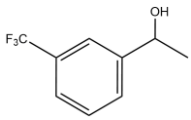
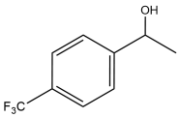
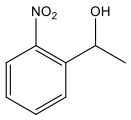
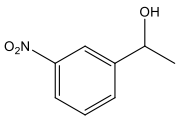
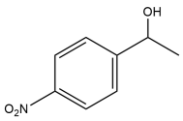
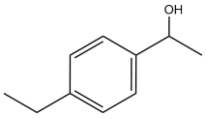
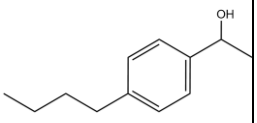
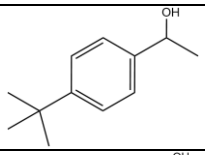
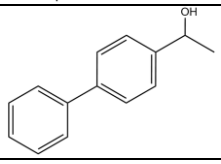
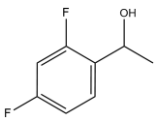
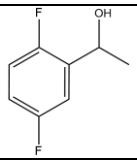
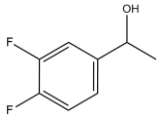
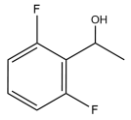
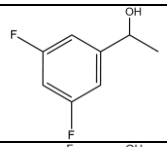
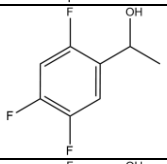
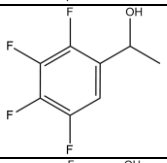
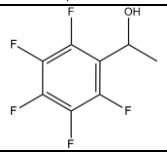
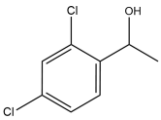
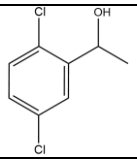
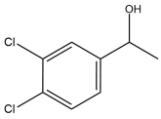
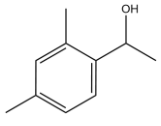


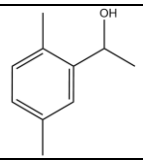
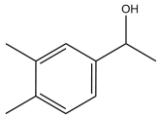
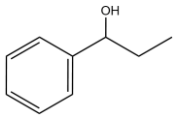
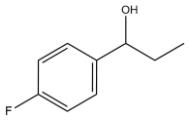
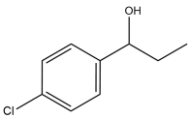
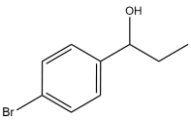
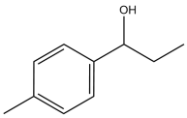
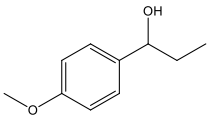
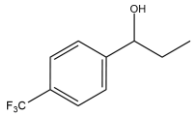
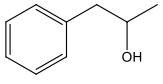
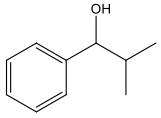
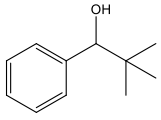
Table 3.1 Abbreviation, structure and name of all alcohols used in this study.

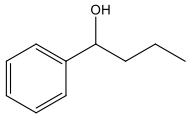
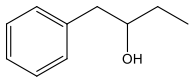
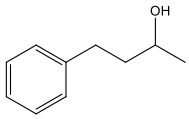
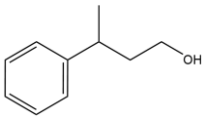
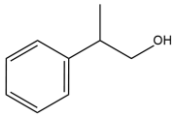
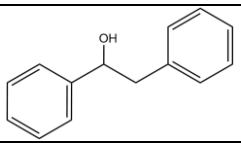
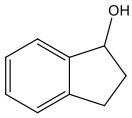
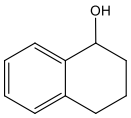
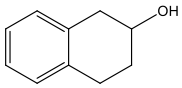
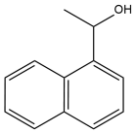
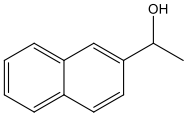
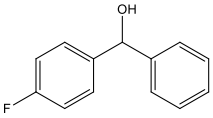
no.	abbreviation	name	structure
L01	PE	1-phenylethanol	
L02	oF-PE	1-(2-fluorophenyl)ethanol	
L03	mF-PE	1-(3-fluorophenyl)ethanol	
L04	pF-PE	1-(4-fluorophenyl)ethanol	

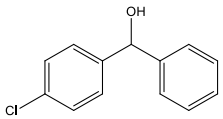
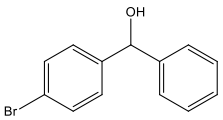
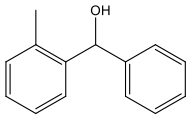
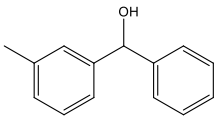
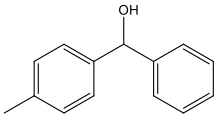
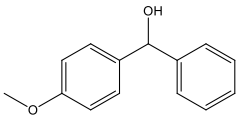
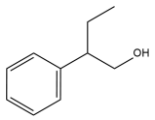
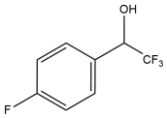
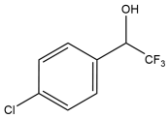
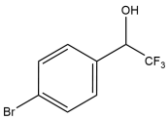
no.	abbreviation	name	structure
L05	oCl-PE	1-(2-chlorophenyl)ethanol	
L06	mCl-PE	1-(3-chlorophenyl)ethanol	
L07	pCl-PE	1-(4-chlorophenyl)ethanol	
L08	oBr-PE	1-(2-bromophenyl)ethanol	
L09	mBr-PE	1-(3-bromophenyl)ethanol	
L10	pBr-PE	1-(4-bromophenyl)ethanol	
L11	oMe-PE	1-(2-methylphenyl)ethanol	
L12	mMe-PE	1-(3-methylphenyl)ethanol	
L13	pME-PE	1-(4-methylphenyl)ethanol	
L14	oOMe-PE	1-(2-methoxyphenyl)ethanol	
L15	mOMe-PE	1-(3-methoxyphenyl)ethanol	
L16	pOMe-PE	1-(4-methoxyphenyl)ethanol	

no.	abbreviation	name	structure
L17	oCF-PE	1-(2-trifluoromethylphenyl)ethanol	
L18	mCF-PE	1-(3-trifluoromethylphenyl)ethanol	
L19	pCF-PE	1-(4-trifluoromethylphenyl)ethanol	
L20	oNO-PE	1-(2-nitrophenyl)ethanol	
L21	mNO-PE	1-(3-nitrophenyl)ethanol	
L22	pNO-PE	1-(4-nitrophenyl)ethanol	
L23	pEt-PE	1-(4-ethylphenyl)ethanol	
L24	pBu-PE	1-(4-butylphenyl)ethanol	
L25	ptBu-PE	1-(4-*tert*-butylphenyl)ethanol	
L26	pPh-PE	1-(4-biphenyl)ethanol	
L27	24F-PE	1-(2,4-difluorophenyl)ethanol	

no.	abbreviation	name	structure
L28	25F-PE	1-(2,5-difluorophenyl)ethanol	
L29	34F-PE	1-(3,4-difluorophenyl)ethanol	
L30	26F-PE	1-(2,6-difluorophenyl)ethanol	
L31	35F-PE	1-(3,5-difluorophenyl)ethanol	
L32	triF-PE	1-(2,4,5-trifluorophenyl)ethanol	
L33	tetraF-PE	1-(2,3,4,5-tetrafluorophenyl)ethanol	
L34	pentaF-PE	1-(pentafluorophenyl)ethanol	
L35	24Cl-PE	1-(2,4-dichlorophenyl)ethanol	
L36	25Cl-PE	1-(2,5-dichlorophenyl)ethanol	
L37	34Cl-PE	1-(3,4-dichlorophenyl)ethanol	
L38	24Me-PE	1-(2,4-dimethylphenyl)ethanol	

no.	abbreviation	name	structure
L39	25Me-PE	1-(2,5-dimethylphenyl)ethanol	
L40	34Me-PE	1-(3,4-dimethylphenyl)ethanol	
L41	PP	1-phenyl-1-propanol	
L42	pF-PP	1-(4-fluorophenyl)propanol	
L43	pCl-PP	1-(4-chlorophenyl)propanol	
L44	pBr-PP	1-(4-bromophenyl)propanol	
L45	pMe-PP	1-(4-methylphenyl)propanol	
L46	pOMe-PP	1-(4-methoxyphenyl)propanol	
L47	pCF3-PP	1-(4-(trifluoromethyl)phenyl)propanol	
L48	1OH	1-phenyl-2-propanol	
L49	2OH	2-methyl-1-phenyl-1-propanol	
L50	3OH	2,2-dimethyl-1-phenyl-1-propanol	

no.	abbreviation	name	structure
L51	4OH	1-phenyl-1-butanol	
L52	5OH	1-phenyl-2-butanol	
L53	6OH	4-phenyl-2-butanol	
L54	7OH	3-phenyl-1-butanol	
L55	8OH	2-phenyl-1-propanol	
L56	DPE	1,2-diphenylethanol	
L57	In	1-indanol	
L58	1-Nap	1,2,3,4-tetrahydro-1-naphthol	
L59	2-Nap	1,2,3,4-tetrahydro-2-naphthol	
L60	1Nap-E	1-(1-naphthyl)ethanol	
L61	2Nap-E	1-(2-naphthyl)ethanol	
L62	pF-Ph	(4-fluorophenyl)(phenyl)-methanol	

no.	abbreviation	name	structure
L63	pCl-Ph	(4-chlorophenyl)(phenyl)-methanol	
L64	pBr-Ph	(4-bromophenyl)(phenyl)-methanol	
L65	oMe-Ph	phenyl-*o*-tolyl-methanol	
L66	mMe-Ph	phenyl-*m*-tolyl-methanol	
L67	pMe-Ph	phenyl-*p*-tolyl-methanol	
L68	pOMe-Ph	(4-methoxyphenyl)(phenyl)-methanol	
L69	2Ph-Bu	2-phenyl-1-butanol	
L70	pF-CF	2,2,2-trifluoro-1-(4-fluorophenyl)ethanol	
L71	pCl-CF	2,2,2-trifluoro-1-(4-chlorophenyl)ethanol	
L72	pBr-CF	2,2,2-trifluoro-1-(4-bromophenyl)ethanol	

3.1.2 Chiral amines

Chiral amines and their derivatives used in this work were previously prepared by Issaraseriruk [39], and Charoenchaiworakit [40]. Their corresponding ketones and titanium(IV) isopropoxide mixed in isopropanol were stirred under purging ammonia gas 5-7 hours. After that, add sodium borohydride and continue stirring for 2 hours. After quenching the reaction and work-up, the obtained amines were characterized by ^{1}H and ^{13}C NMR. Total of 43 chiral amines were used in this work. All amines were derivatized into trifluoroacetyl (TFA) derivatives using trifluoroacetic anhydride (TFAA) before GC analyses.

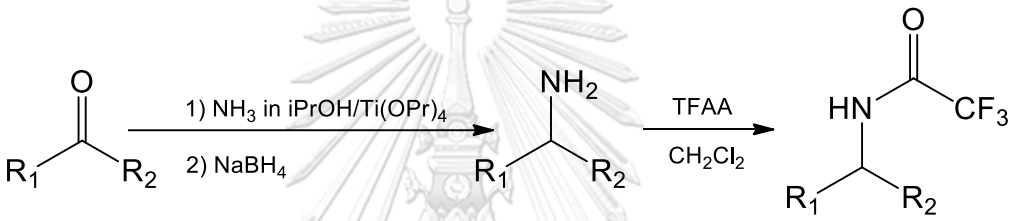
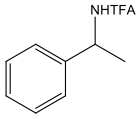
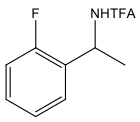
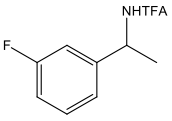
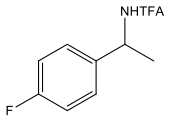
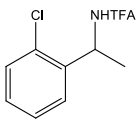
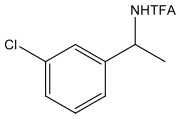
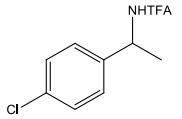
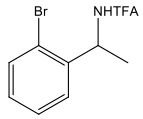
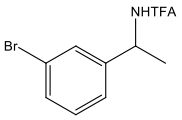
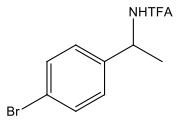
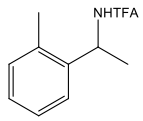
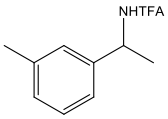
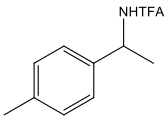
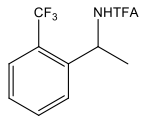
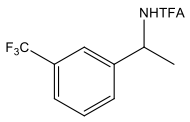
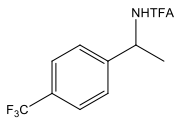
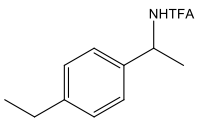
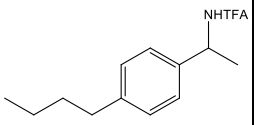
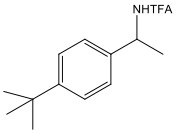
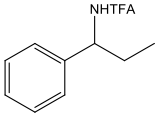
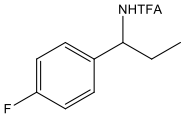
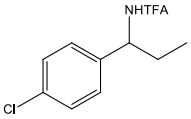
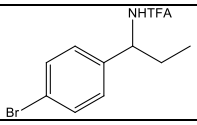
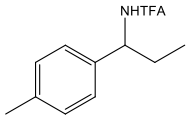
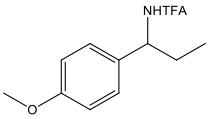
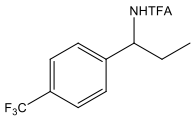
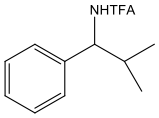
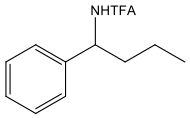
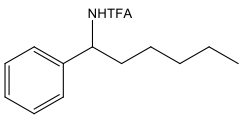
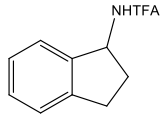
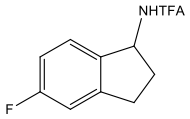
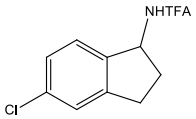
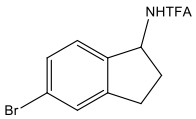
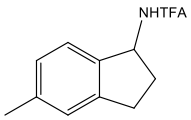
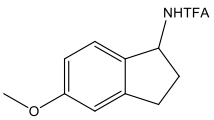
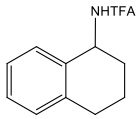
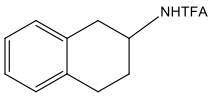
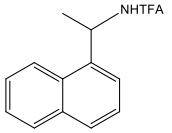
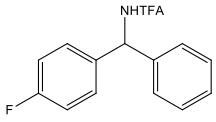
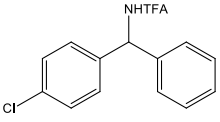
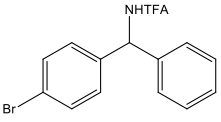


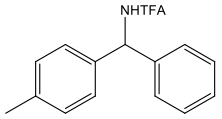
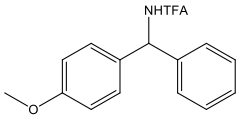
Table 3.2 Abbreviation, structure and name of all amine derivatives used in this study.

no.	abbreviation	name of amine	structure
A01	**1**	1-phenylethylamine	
A02	**oF-1**	1-(2′-fluorophenyl)ethylamine	
A03	**mF-1**	1-(3′-fluorophenyl)ethylamine	
A04	**pF-1**	1-(4′-fluorophenyl)ethylamine	
A05	**oCl-1**	1-(2′-chlorophenyl)ethylamine	

no.	abbreviation	name of amine	structure
A06	mCl-1	1-(3′-chlorophenyl)ethylamine	
A07	pCl-1	1-(4′-chlorophenyl)ethylamine	
A08	oBr-1	1-(2′-bromophenyl)ethylamine	
A09	mBr-1	1-(3′-bromophenyl)ethylamine	
A10	pBr-1	1-(4′-bromophenyl)ethylamine	
A11	oMe-1	1-(2′-methylphenyl)ethylamine	
A12	mMe-1	1-(3′-methylphenyl)ethylamine	
A13	pMe-1	1-(4′-methylphenyl)ethylamine	
A14	oCF-1	1-(2′-trifluoromethylphenyl)ethylamine	
A15	mCF-1	1-(3′-trifluoromethylphenyl)ethylamine	
A16	pCF-1	1-(4′-trifluoromethylphenyl)ethylamine	
A17	pEt	1-(4-ethylphenyl)ethylamine	

no.	abbreviation	name of amine	structure
A18	pBu	1-(4-butylphenyl)ethylamine	
A19	ptBu	1-(4-*tert*-butylphenyl)ethylamine	
A20	2	1-phenylpropylamine	
A21	pF-2	1-(4´-fluorophenyl)propylamine	
A22	pCl-2	1-(4´-chlorophenyl)propylamine	
A23	pBr-2	1-(4´-bromophenyl)propylamine	
A24	pMe-2	1-(4´-methylphenyl)propylamine	
A25	pOMe-2	1-(4´-methoxyphenyl)propylamine	
A26	pCF-2	1-(4´-trifluoromethylphenyl)propylamine	
A27	iBu	2-methyl-1-phenyl-1-propylamine	
A28	3	1-phenylbutylamine	
A29	4	1-phenylhexylamine	

no.	abbreviation	name of amine	structure
A30	A	1-aminoindan	
A31	5F-A	5′-fluoro-1-aminoindan	
A32	5Cl-A	5′-chloro-1-aminoindan	
A33	5Br-A	5′-bromo-1-aminoindan	
A34	5Me-A	5′-methyl-1-aminoindan	
A35	5OMe-A	5′-methoxy-1-aminoindan	
A36	1ATL	1-aminotetralin	
A37	2ATL	2-aminotetralin	
A38	Nap	1-(1-naphthyl)ethylamine	
A39	pF-6	(4-fluorophenyl)(phenyl)methanamine	
A40	pCl-6	(4-chlorophenyl)(phenyl)methanamine	
A41	pBr-6	(4-bromophenyl)(phenyl)methanamine	

no.	abbreviation	name of amine	structure
A42	pMe-6	(4-methylphenyl)(phenyl)methanamine	
A43	pOMe-6	(4-methoxyphenyl)(phenyl)methanamine	

3.2 Gas chromatographic analysis

All chromatographic analyses were performed on an Agilent 7890 B series gas chromatograph using the following conditions:

carrier gas : hydrogen, average linear velocity of 50 cm/sec

injector : split injector, split ratio 100

injector temperature : 250 °C

detector : flame ionization detector (FID)

detector temperature : 250 °C

hydrogen : 40 mL/min

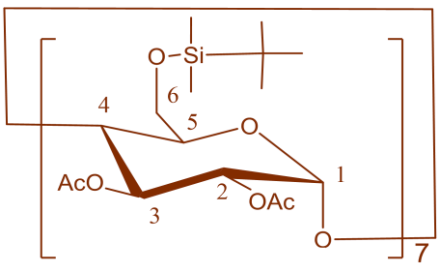
air : 400 mL/min

nitrogen + carrier : 40 mL/min

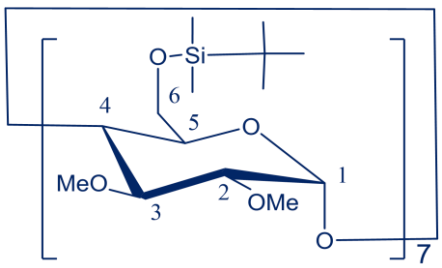
GC column : fused silica capillary column of ~15 m long, 0.25 mm i.d. coated with 0.25 µm thick film of stationary phase

column BSiAc (15.57 m long) : 33.5% heptakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD in polysiloxane OV-1701

column BSiMe (14.91 m long) : 30.0% heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD in polysiloxane OV-1701



BSiAc



BSiMe

Before use, columns were conditioned at 220 °C until a stable baseline was observed. Column efficiency was regularly checked at each working temperature with *n*-alkane. Effciencies above 3,500 plates/m were observed for both columns.

All chiral analytes were dilute in dichloromethane. Each analyte solution (~0.2 µL) was analyzed by both temperature program and isothermal conditions.

3.2.1 GC analysis using temperature program

Each chiral analyte was run under temperature program condition. The column temperature was programmed from 40 °C at the rate of ~3 °C/min (according to Grob) [41], [42]. Elution temperature for each eluted peak was calculated. Resolution (Rs) between two peaks were calculated if enantiomeric separation was observed.

3.2.2 GC analysis using isothermal condition

Each chiral analyte was analyzed, at least in duplicate, at 6-8 different isothermal temperatures of 10 °C interval. Retention factors (k′), enantioselectivity (α), and resolution (Rs) were calculated from chromatograms. Additionally, the column temperature was adjusted for each analyte until a resolution of at least 1.5 and 2.0 were obtained. Their retention factors (k′) at both temperatures were calculated.

CHAPTER IV

RESULTS AND DISCUSSIONS

In this work, enantiomeric separations of seventy-two chiral alcohols and forty-three chiral amines were studied by capillary gas chromatography using two chiral stationary phases: heptakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or BSiAc) mixed in OV-1701 and heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or BSiMe) mixed in OV-1701. Both β-CD derivatives have been proved to be useful chiral stationary phases for different classes of compounds.

Generally, determination of the optimum condition for the separation of enantiomers is time consuming. In many cases, a complete separation of enantiomers may not be obtained. So far, there is still no guideline for separation of enantiomers. In this work, each chiral compound was analyzed using a temperature program run and elution temperatures for all eluted peaks were calculated. When a chiral compound can be separated into their enantiomers, two peaks will be observed and the resolution (Rs) between two peaks will be calculated. Next, an optimum isothermal temperature for a complete separation of enantiomers (with a minimum Rs of 1.5) will be determined. At the optimum condition, retention factor (k′) and enantioselectivity (α) were calculated from chromatograms. Chromatographic data obtained from temperature program runs and from the optimum isothermal conditions were studied for their relationship hoping that some information from the temperature program run can be used as a guideline to quickly determine the optimum separation condition.

4.1 Enantiomeric separation using BSiAc column

4.1.1 Analyses of chiral alcohols by temperature program

Seventy-two chiral alcohols were used in this study and they were directly analyzed without derivatization. Their structures were mainly based on 1-phenylethanols with different type (e.g. F, Cl, Br, Me, OCH_3, CF_3, NO_2) and position (*ortho*, *meta, para*) of substitution. Other structures included mono-substituted 1-phenyl-1-propanols, mono-substituted diphenylmethanols and other alcohols. Each alcohol was analyzed by GC using a temperature program starting from 40 ºC with a rate of 3.21 °C/min [41], [42]. The elution temperatures for all eluted peaks and resolution (Rs) between two enantiomeric peaks were calculated as shown in **Table 4.1**.

Table 4.1 Chromatographic results of 72 chiral alcohols analyzed by BSiAc column (15.57 m long) using a temperature program starting from 40 °C at a rate of 3.21 °C/min.

no.	abbreviation	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
L01	PE	18.929	19.517	0.0554	0.0530	100.76	102.65	6.38
L02	oF-PE	16.886	17.144	0.0612	0.0645	94.20	95.03	2.42
L03	mF-PE	21.625	22.037	0.0542	0.0529	109.42	110.74	4.53
L04	pF-PE	21.626	21.961	0.0562	0.0523	109.42	110.49	3.63
L05	oCl-PE	21.381	21.722	0.0588	0.0548	108.63	109.73	3.53
L06	mCl-PE	27.204	27.646	0.0530	0.0482	127.32	128.74	5.14
L07	pCl-PE	28.245	28.535	0.0516	0.0508	130.67	131.60	3.33
L08	oBr-PE	24.339	24.605	0.0603	0.0596	118.13	118.98	2.61
L09	mBr-PE	29.834	30.241	0.0525	0.0516	135.77	137.07	4.60
L10	pBr-PE	31.140	31.401	0.0539	0.0555	139.96	140.80	2.81
L11	oMe-PE	19.297	19.394	0.0542	0.0575	101.94	102.25	1.02
L12	mMe-PE	20.922	21.407	0.0523	0.0488	107.16	108.72	5.65
L13	pME-PE	22.306	23.037	0.0529	0.0499	111.60	113.95	8.37
L14	oOMe-PE	14.819	14.934	0.0550	0.0516	87.57	87.94	1.27
L15	mOMe-PE	27.823	28.243	0.0544	0.0527	129.31	130.66	4.62

no.	abbreviation	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
L16	pOMe-PE	20.719	21.078	0.0599	0.0594	106.51	107.66	3.54
L17	oCF-PE	14.943	15.425	0.0541	0.0550	87.97	89.51	5.20
L18	mCF-PE	21.267	21.844	0.0526	0.0507	108.27	110.12	6.57
L19	pCF-PE	23.647	23.963	0.0490	0.0480	115.91	116.92	3.83
L20	oNO-PE	31.461	31.836	0.0599	0.0603	140.99	142.19	3.67
L21	mNO-PE	-	38.891	-	0.0594	-	164.84	0
L22	pNO-PE	-	42.491	-	0.0850	-	176.40	0
L23	pEt-PE	24.593	25.106	0.0537	0.0521	118.94	120.59	5.71
L24	pBu-PE	29.905	30.179	0.0572	0.0526	136.00	136.87	2.94
L25	ptBu-PE	27.147	27.304	0.0537	0.0562	127.14	127.65	1.68
L26	pPh-PE	42.345	42.497	0.0565	0.0580	175.93	176.42	1.56
L27	24F-PE	18.157	18.362	0.0534	0.0537	98.28	98.94	2.25
L28	25F-PE	18.982	19.195	0.0540	0.0536	100.93	101.62	2.33
L29	34F-PE	24.760	24.946	0.0485	0.0506	119.48	120.08	2.21
L30	26F-PE	-	14.128	-	0.0878	-	85.35	0
L31	35F-PE	21.997	22.550	0.0485	0.0468	110.61	112.39	6.83
L32	triF-PE	21.252	21.514	0.0500	0.0469	108.22	109.06	3.18
L33	tetraF-PE	21.394	21.462	0.0468	0.0528	108.67	108.89	*(b)
L34	pentaF-PE	18.887	19.223	0.0505	0.0489	100.63	101.71	3.98
L35	24Cl-PE	-	28.626	-	0.0960	-	131.89	0
L36	25Cl-PE	-	29.018	-	0.0425	-	133.15	0
L37	34Cl-PE	34.644	34.965	0.0570	0.0550	151.21	152.24	3.37
L38	24Me-PE	-	22.343	-	0.0900	-	111.72	0
L39	25Me-PE	21.604	21.691	0.0575	0.0624	109.35	109.63	*(b)
L40	34Me-PE	25.213	25.899	0.0522	0.0530	120.93	123.14	7.68
L41	PP	20.159	20.578	0.0569	0.0530	104.71	106.06	4.49
L42	pF-PP	22.945	23.320	0.0507	0.0514	113.65	114.86	4.32
L43	pCl-PP	29.507	29.760	0.0534	0.0497	134.72	135.53	2.89
L44	pBr-PP	32.390	32.598	0.0571	0.0562	143.97	144.64	2.16
L45	pMe-PP	23.229	23.758	0.0550	0.0536	114.57	116.26	5.73
L46	pOMe-PP	29.423	29.740	0.0559	0.0556	134.45	135.47	3.35
L47	pCF3-PP	21.997	22.550	0.0485	0.0468	110.61	112.39	6.83

no.	abbreviation	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
L48	1OH	20.087	20.297	0.0548	0.0564	104.48	105.15	2.22
L49	2OH	-	20.140	-	0.0578	-	104.65	0
L50	3OH	20.850	20.973	0.0589	0.0580	106.93	107.32	1.24
L51	4OH	-	22.518	-	0.0780	-	112.28	0
L52	5OH	-	22.485	-	0.0560	-	112.18	0
L53	6OH	24.817	24.931	0.0527	0.0543	119.66	120.03	1.25
L54	7OH	23.295	23.344	0.0453	0.0639	114.78	114.93	*(b)
L55	8OH	20.341	21.034	0.0589	0.0567	105.29	107.52	7.06
L56	DPE	-	37.933	-	0.0682	-	161.76	0
L57	In	-	23.172	-	0.0752	-	114.38	0
L58	1-Nap	-	25.797	-	0.0880	-	122.81	0
L59	2-Nap	27.553	27.649	0.0549	0.0647	128.45	128.75	*(b)
L60	1Nap-E	-	34.626	-	0.0843	-	151.15	0
L61	2Nap-E	37.227	37.501	0.0539	0.0573	159.50	160.38	2.90
L62	pF-Ph	-	36.816	-	0.0793	-	158.18	0
L63	pCl-Ph	-	42.824	-	0.0930	-	177.47	0
L64	pBr-Ph	-	42.262	-	0.0626	-	175.66	0
L65	oMe-Ph	-	37.567	-	0.0655	-	160.59	0
L66	mMe-Ph	-	37.696	-	0.0645	-	161.00	0
L67	pMe-Ph	-	38.242	-	0.0660	-	162.76	0
L68	pOMe-Ph	-	43.898	-	0.0663	-	180.91	0
L69	2Ph-Bu	21.521	21.838	0.0638	0.0609	109.08	110.10	2.99
L70	pF-CF	24.709	24.913	0.0503	0.0457	119.32	119.97	2.50
L71	pCl- CF	30.297	30.423	0.0499	0.0509	137.25	137.66	1.47
L72	pBr-CF	32.981	33.093	0.0515	0.0498	145.87	146.23	1.30

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

From **Table 4.1**, 10 out of 72 alcohols showed no enantiomeric separation on BSiAc column using temperature program condition. While 42 alcohols could be completely separated (Rs ≥ 1.5) into their enantiomers and 20 alcohols showed

incomplete enantiomeric separation (Rs < 1.5). Next, each chiral alcohol was analyzed by the same column using isothermal condition in order to shorten the analysis time for alcohols with Rs ≥ 1.5 and to improve enantiomeric separation for alcohols with Rs < 1.5.

4.1.2 Analyses of chiral alcohols using isothermal condition

The optimum GC condition was the column temperature which provided the shortest analysis time for the separation of two enantiomers with a minimum Rs of 1.5. Although many alcohols could be separated into their enantiomers using temperature programs, their analysis times were too long. Using the elution temperature obtained from the temperature program run as a starting isothermal temperature for the analysis of each analyte, the column temperature was further adjusted (increased or decreased) until a minimum Rs of 1.5 was obtained. In addition, the column temperature for a separation with Rs of 2.0 was also determined (if possible).

The optimization for the enantiomeric separation of alcohol **pMe-PE** (or **L13**) was demonstrated. Using a temperature program starting from 40 °C with a rate of 3.21 °C/min, **L13** could be well separated into two peaks with retention times of 22.306 and 23.037 minutes and Rs of 8.37 (**Figure 4.1**). The elution temperature of the last eluting peak was calculated to be 113.95 °C (**Table 4.1**).

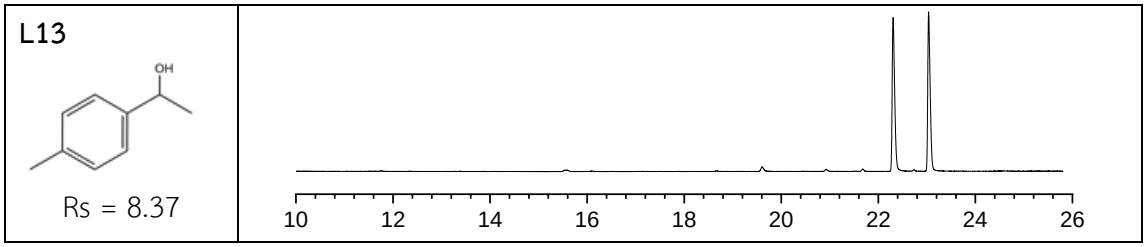


Figure 4.1 Separation of **L13** on BSiAc column using temperature program.

An isothermal temperature of 120 ℃ was then selected as a starting temperature for determination of optimum isothermal condition. At 120 ℃, **L13** were very well separated into two peaks with Rs = 3.60 (**Figure 4.2**). The column temperature was then increased by 10 ℃ to 130 and 140 ℃ to obtain Rs values of 2.20 and 1.38, respectively. The column temperature was further adjusted between 130-140 ℃ in order to obtain Rs values closed to 1.5 and 2.0. It was found that at 137 ℃, **L13** could be completely separated with Rs = 1.57 and analysis time of 2.039 minutes (k'_2 = 2.906) (**Figure 4.2**). It was obvious that the separation of **L13** at its optimum condition was about 11 times faster than using a temperature program. In addition, the isothermal condition for the separation of **L13** with Rs value closed to 2.0 was determined to be 131 ℃ with analysis time of 2.534 minutes (k'_2 = 3.667).

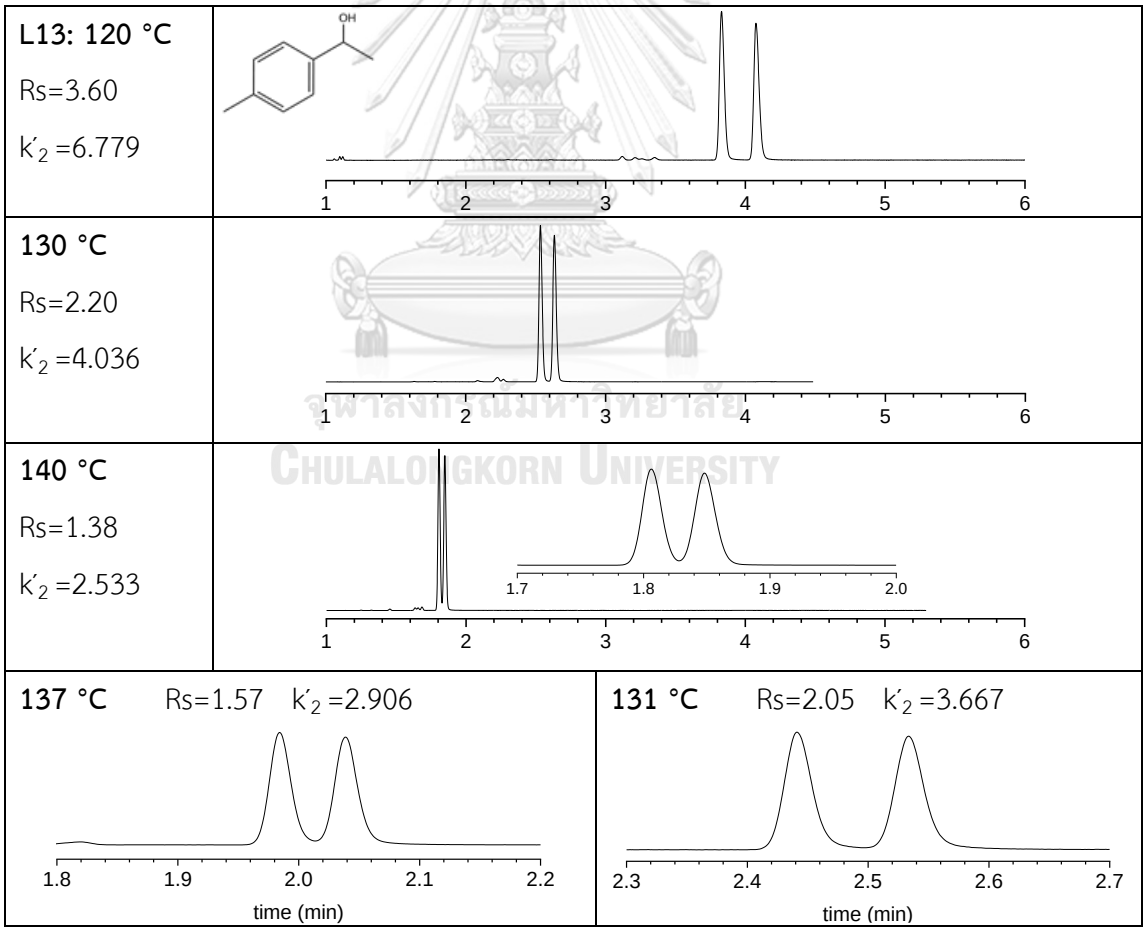


Figure 4.2 Separation of **L13** on BSiAc column using isothermal conditions.

The optimization for the enantiomeric separation of alcohol **ptBu-PE** (or **L25**) was demonstrated in **Figure 4.3**. From a temperature program, **L25** was separated in 27.304 minutes with Rs of 1.68. The elution temperature of the last eluting peak was calculated to be 127.65 ℃ (**Table 4.1**). An isothermal temperature of 120 ℃ was selected, enantiomers of **L25** was not well separated (Rs = 1.04, k'_2 = 16.927). The column temperature was then decreased by 10 ℃ and adjusted until a complete separation was obtained. Alcohol **L25** showed complete separation at 113 ℃ with Rs = 1.51 and analysis time of 13.720 minutes (k'_2 = 25.091) (**Figure 4.3**). The separation of **L25** at its optimum condition was about 2 times faster than using a temperature program. At 108 ℃, **L25** were separated with Rs = 2.08 and analysis time of 18.388 minutes (k'_2 = 33.892).

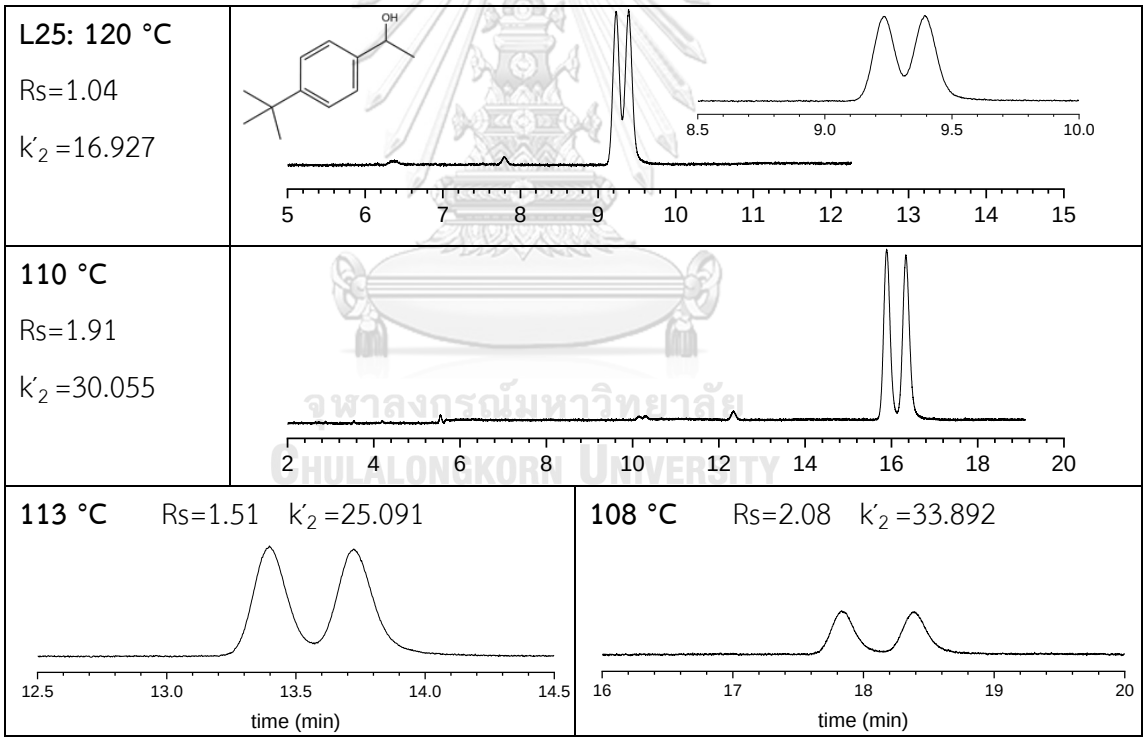


Figure 4.3 Separation of **L25** on BSiAc column using isothermal conditions.

When a temperature program run could not provide a complete enantiomeric separation, isothermal analyses were attempted in a similar fashion. Alcohol **L50** (or **3OH**) was analyzed by temperature program with the analysis time

of 20.973 minutes with Rs of 1.24. The elution temperature of the last eluting peak was calculated to be 107.32 ℃ (**Table 4.1**). Starting with an isothermal temperature of 110 ℃, **L50** was observed as a single peak. The column temperature was then decreased and adjusted until a complete separation was obtained. Alcohol **L50** showed complete separation at 88 ℃ with Rs = 1.52 and analysis time of 17.163 minutes (k'_2 = 31.322) (**Figure 4.4**). Although the complete enantiomeric separation of **L50** was achieved, the k'_2 value of 31.322 was quite large. As a compound retained too long (k' > 20), broader peak shapes may be observed. For this compound, further decrease the column temperature to get a separation with Rs of 2.0 was not attempted.

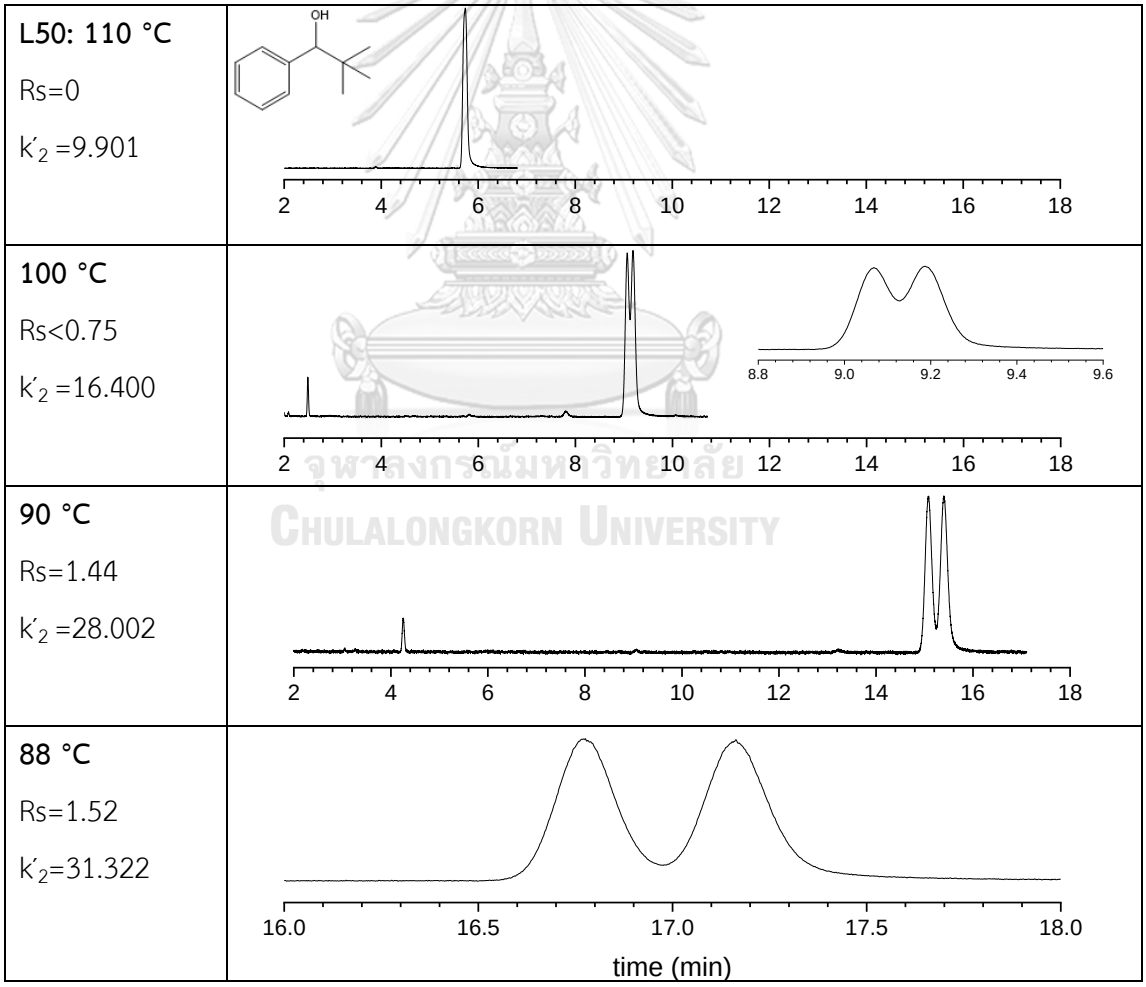


Figure 4.4 Separation of **L50** on BSiAc column using isothermal conditions.

Alcohol **L62** (or **pF-Ph**) was analyzed by temperature program with the analysis time of 36.816 minutes and only one peak was observed (Rs = 0). The elution temperature of the eluting peak was calculated to be 158.18 ℃ (**Table 4.1**). Isothermal analyses were attempted by starting at 160 ℃, **L62** was observed as a single peak. The column temperature was then decreased. At 140 ℃, **L62** was still observed as a single broad peak with long retention with k' =32.770 (**Figure 4.5**). In this case, a complete enantiomeric separation for alcohol **L62** could not be achieved with this stationary phase.

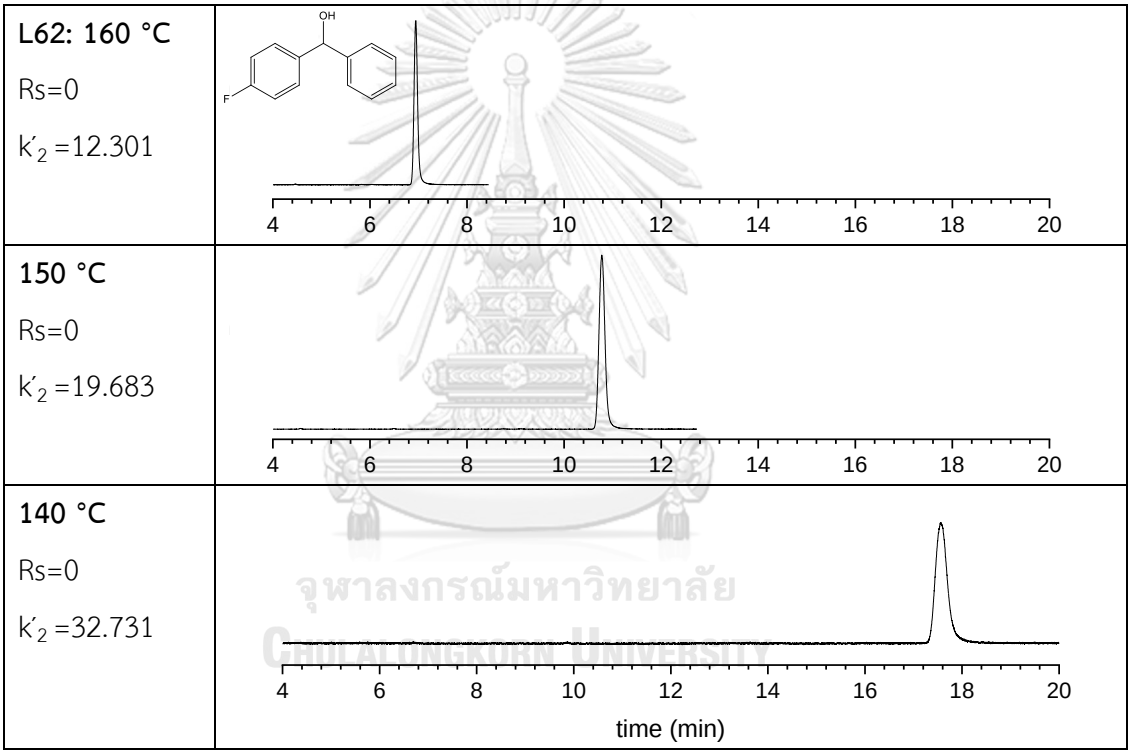


Figure 4.5 Separation of **L62** on BSiAc column using isothermal conditions.

Determination of the optimum isothermal condition for other alcohols was performed. If the retention factor of analyte was greater than 20 and only a single peak was obtained, further analysis was not performed. Chromatographic results for 72 alcohols obtained from temperature program condition and isothermal conditions with Rs values closed to 1.5 and 2.0 were compared in **Table 4.2**.

Table 4.2 Chromatographic results of 72 chiral alcohols analyzed by BSiAc column (15.57 m long) using temperature program and isothermal conditions.

alcohol		temperature program rate 3.21 °C /min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L01	PE	19.517	102.65	6.38	118	2.599	3.960	1.036	1.53	113	3.216	5.137	1.046	2.05
L02	oF-PE	17.144	95.03	2.42	108	3.173	5.021	1.034	1.55	98	4.958	8.408	1.042	2.02
L03	mF-PE	22.037	110.74	4.53	118	3.711	6.069	1.036	1.57	112	5.046	8.611	1.046	2.09
L04	pF-PE	21.961	110.49	3.63	116	4.147	6.899	1.034	1.58	110	5.679	9.797	1.043	2.08
L05	oCl-PE	21.722	109.73	3.53	118	4.596	7.771	1.033	1.51	109	6.772	11.899	1.042	2.08
L06	mCl-PE	27.646	128.74	5.14	135	4.334	7.287	1.033	1.58	130	5.513	9.541	1.042	2.03
L07	pCl-PE	28.535	131.60	3.33	132	5.880	10.221	1.032	1.56	127	7.683	13.662	1.039	2.07
L08	oBr-PE	24.605	118.98	2.61	120	6.615	11.600	1.030	1.51	111	9.952	17.884	1.038	2.02
L09	mBr-PE	30.241	137.07	4.60	141	5.141	8.830	1.030	1.51	135	6.858	12.088	1.040	2.00
L10	pBr-PE	31.401	140.80	2.81	138	7.253	12.842	1.030	1.50	132	9.998	18.044	1.038	2.06
L11	oMe-PE	19.394	102.25	1.02	-	-	-	-	-	-	-	-	-	-
L12	mMe-PE	21.407	108.72	5.65	127	2.527	3.823	1.037	1.59	121	3.213	5.120	1.050	2.37
L13	pME-PE	23.037	113.95	8.37	137	2.039	2.906	1.038	1.57	131	2.534	3.845	1.048	2.05
L14	oOMe-PE	14.934	87.94	1.27	-	-	-	-	-	-	-	-	-	-
L15	mOMe-PE	28.243	130.66	4.62	137	4.424	7.475	1.032	1.51	131	5.900	10.281	1.042	2.02
L16	pOMe-PE	21.078	107.66	3.54	119	3.968	6.573	1.029	1.51	110	5.780	10.010	1.038	2.01
L17	oCF-PE	15.425	89.51	5.20	112	2.336	3.450	1.036	1.54	104	3.122	4.935	1.046	2.04

alcohol		temperature program rate 3.21 °C /min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution temp$_2$	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L18	mCF-PE	21.844	110.12	6.57	124	2.768	4.282	1.035	1.56	119	3.454	5.579	1.044	2.04
L19	pCF-PE	23.963	116.92	3.83	120	4.703	7.975	1.033	1.58	114	6.540	11.457	1.042	2.06
L20	oNO-PE	31.836	142.19	3.67	150	5.093	8.738	1.031	1.56	141	7.453	13.278	1.041	2.00
L21	mNO-PE	38.891	164.84	0	-	-	-	-	-	-	-	-	-	-
L22	pNO-PE	42.492	176.40	0	-	-	-	-	-	-	-	-	-	-
L23	pEt-PE	25.106	120.59	5.71	132	3.459	5.601	1.035	1.53	126	4.496	7.580	1.046	2.03
L24	pBu-PE	30.179	136.87	2.94	132	8.190	14.630	1.031	1.57	126	11.231	20.392	1.040	2.06
L25	ptBu-PE	27.304	127.65	1.68	113	13.720	25.084	1.025	1.50	108	18.388	33.892	1.032	2.08
L26	pPh-PE	42.497	176.42	1.56	158	18.297	33.985	1.023	1.51	-	-	-	-	-
L27	24F-PE	18.362	98.94	2.25	102	4.807	8.104	1.032	1.54	91	8.685	15.480	1.040	2.03
L28	25F-PE	19.195	101.62	2.33	108	4.168	6.939	1.032	1.52	97	7.300	12.878	1.040	2.01
L29	34F-PE	24.946	120.08	2.21	113	8.369	14.971	1.030	1.52	108	11.506	20.916	1.037	2.07
L30	26F-PE	14.128	85.35	0	-	-	-	-	-	-	-	-	-	-
L31	35F-PE	22.550	112.39	6.83	126	2.705	4.162	1.036	1.54	121	3.393	5.475	1.045	2.07
L32	triF-PE	21.514	109.06	3.18	116	3.884	6.398	1.033	1.54	107	6.224	10.855	1.043	2.09
L33	tetraF-PE	21.462	108.89	*(b)	-	-	-	-	-	-	-	-	-	-
L34	pentaF-PE	19.223	101.71	3.98	112	3.222	5.137	1.035	1.54	105	4.542	7.635	1.044	2.05
L35	24Cl-PE	28.626	131.89	0	-	-	-	-	-	-	-	-	-	-
L36	25Cl-PE	29.018	133.15	0	-	-	-	-	-	-	-	-	-	-
L37	34Cl-PE	34.965	152.24	3.37	151	6.912	12.216	1.030	1.5	145	9.386	16.912	1.039	2.05

alcohol		temperature program rate 3.21 °C /min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution temp$_2$	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L38	24Me-PE	22.343	111.72	0	-	-	-	-	-	-	-	-	-	-
L39	25Me-PE	21.691	109.63	*(b)	-	-	-	-	-	-	-	-	-	-
L40	34Me-PE	25.899	123.14	7.68	139	2.875	4.487	1.035	1.52	134	3.509	5.697	1.044	2.01
L41	PP	20.578	106.06	4.49	111	4.485	7.543	1.033	1.52	105	6.019	10.443	1.044	2.09
L42	pF-PP	23.320	114.86	4.32	123	3.856	6.373	1.034	1.59	117	5.150	8.828	1.043	2.06
L43	pCl-PP	29.760	135.53	2.89	132	7.503	13.346	1.031	1.53	126	10.373	18.834	1.038	2.02
L44	pBr-PP	32.598	144.64	2.16	135	10.782	19.616	1.030	1.52	131	13.437	24.692	1.034	2.08
L45	pMe-PP	23.758	116.26	5.73	126	3.768	6.205	1.035	1.58	121	4.705	7.979	1.044	2.09
L46	pOMe-PP	29.740	135.47	3.35	132	7.658	13.642	1.031	1.53	127	9.880	17.855	1.040	2.01
L47	pCF3-PP	22.550	112.39	6.83	124	4.968	8.481	1.032	1.57	118	6.861	12.069	1.040	2.04
L48	1OH	20.297	105.15	2.22	104	6.059	10.497	1.032	1.53	97	8.881	15.788	1.040	2.04
L49	2OH	20.140	104.65	0	-	-	-	-	-	-	-	-	-	-
L50	3OH	20.973	107.32	1.24	88	17.163	31.322	1.024	1.52	-	-	-	-	-
L51	4OH	22.518	112.28	0	-	-	-	-	-	-	-	-	-	-
L52	5OH	22.485	112.18	0	-	-	-	-	-	-	-	-	-	-
L53	6OH	24.931	120.03	1.25	102	16.621	30.479	1.024	1.54	-	-	-	-	-
L54	7OH	23.344	114.93	*(b)	-	-	-	-	-	-	-	-	-	-
L55	8OH	21.034	107.52	7.06	132	2.178	3.156	1.041	1.51	126	2.671	4.088	1.051	2.05
L56	DPE	37.933	161.76	0	-	-	-	-	-	-	-	-	-	-
L57	In	23.172	114.38	0	-	-	-	-	-	-	-	-	-	-

alcohol		temperature program rate 3.21 °C /min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L58	1-Nap	25.797	122.81	0	-	-	-	-	-	-	-	-	-	-
L59	2-Nap	27.649	128.75	*(b)	-	-	-	-	-	-	-	-	-	-
L60	1Nap-E	34.626	151.15	0	-	-	-	-	-	-	-	-	-	-
L61	2Nap-E	37.501	160.38	2.90	152	10.452	18.985	1.031	1.59	149	12.199	22.325	1.035	2.01
L62	pF-Ph	36.816	158.18	0	-	-	-	-	-	-	-	-	-	-
L63	pCl-Ph	42.824	177.47	0	-	-	-	-	-	-	-	-	-	-
L64	pBr-Ph	42.262	175.66	0	-	-	-	-	-	-	-	-	-	-
L65	oMe-Ph	37.567	160.59	0	-	-	-	-	-	-	-	-	-	-
L66	mMe-Ph	37.696	161.00	0	-	-	-	-	-	-	-	-	-	-
L67	pMe-Ph	38.242	162.76	0	-	-	-	-	-	-	-	-	-	-
L68	pOMe-Ph	43.898	180.91	0	-	-	-	-	-	-	-	-	-	-
L69	2Ph-Bu	21.838	110.10	2.99	106	7.681	13.575	1.037	1.52	101	9.873	17.699	1.045	2.02
L70	pF-CF	24.913	119.97	2.50	115	7.366	12.977	1.029	1.51	110	10.044	18.059	1.037	2.02
L71	pCl- CF	30.423	137.66	1.47	125	12.457	22.728	1.024	1.54	-	-	-	-	-
L72	pBr-CF	33.093	146.23	1.30	130	15.761	29.021	1.022	1.53	-	-	-	-	-

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

From **Table 4.2**, 42 alcohols could be completely separated (Rs ≥ 1.5) into their enantiomers by temperature program. Optimum isothermal conditions (Rs ~ 1.5) for all 42 alcohols could be determined and their analysis times using isothermal condition were much shorter than using temperature program. For six alcohols analyzed by temperature program with incomplete separation (1.0 < Rs < 1.5), four of them could be completely separated using isothermal condition with slightly longer retention. Total of 26 alcohols could not be completely separated into their enantiomers either by temperature program or isothermal condition in a reasonable time, which all of them showed no separation or separated with Rs < 1.3 by temperature program.

Based on results obtained from 72 chiral alcohols, resolution values from temperature program runs may be used as a screening condition. If Rs value is less than 1.0, complete enantiomeric separation is likely not possible with this column. Next, temperature program rates of 10.00 and 30.00 °C/min were examined in order to decrease analysis time using a temperature program run. Results obtained from different temperature program rates were compared in **Table 4.3**. When the temperature program rates were changed to 10.00 and 30.00 °C/min, analysis times were decreased by 2-6 times, respectively. Nonetheless, Rs values obtained from high temperature program rates were low or incomplete separations were mostly observed. Therefore, initial temperature program rate based on Grob test [41], [42] was used.

Table 4.3 Chromatographic results of 72 chiral alcohols analyzed by a BSiAc column using different temperature program rates.

alcohol		rate 3.21 °C/min			rate 10.00 °C/min			rate 30.00 °C/min		
no.	abbr.	$t_{R,2}$ (min)	elution temp$_2$ (°C)	Rs	$t_{R,2}$ (min)	elution temp$_2$ (°C)	Rs	$t_{R,2}$ (min)	elution temp$_2$ (°C)	Rs
L01	PE	19.517	102.65	6.38	8.121	121.21	3.51	3.507	145.21	1.55
L02	oF-PE	17.144	95.03	2.42	7.524	115.24	2.07	3.349	140.47	1.12
L03	mF-PE	22.037	110.74	4.53	8.917	129.17	2.60	3.754	152.62	1.13
L04	pF-PE	21.961	110.49	3.63	8.927	129.27	2.13	3.761	152.83	1.29
L05	oCl-PE	21.722	109.73	3.53	9.216	132.16	2.11	3.990	159.70	1.01
L06	mCl-PE	27.646	128.74	5.14	10.830	148.30	2.50	4.449	173.47	1.26
L07	pCl-PE	28.535	131.60	3.33	11.128	151.28	1.73	4.544	176.32	0
L08	oBr-PE	24.605	118.98	2.61	10.220	142.20	1.63	4.338	170.14	0
L09	mBr-PE	30.241	137.07	4.60	11.742	157.42	2.26	4.786	183.58	1.21
L10	pBr-PE	31.401	140.80	2.81	12.118	161.18	1.57	4.899	186.97	1.04
L11	oMe-PE	19.394	102.25	1.02	8.403	124.03	0	3.698	150.94	0
L12	mMe-PE	21.407	108.72	5.65	8.862	128.62	4.51	3.778	153.34	1.80
L13	pME-PE	23.037	113.95	8.37	9.284	132.84	4.91	3.902	157.06	2.15
L14	oOMe-PE	14.934	87.94	1.27	9.578	135.78	0	4.119	163.57	0
L15	mOMe-PE	28.243	130.66	4.62	11.056	150.56	2.37	4.536	176.08	1.66
L16	pOMe-PE	21.078	107.66	3.54	8.970	129.70	2.10	3.903	157.09	1.05
L17	oCF-PE	15.425	89.51	5.20	7.048	110.48	3.36	3.219	136.57	1.62
L18	mCF-PE	21.844	110.12	6.57	8.862	128.62	3.50	3.745	152.35	1.42

alcohol		rate 3.21 °C/min			rate 10.00 °C/min			rate 30.00 °C/min		
no.	abbr.	$t_{R,2}$ (min)	elution temp$_2$ (°C)	Rs	$t_{R,2}$ (min)	elution temp$_2$ (°C)	Rs	$t_{R,2}$ (min)	elution temp$_2$ (°C)	Rs
L19	pCF-PE	23.963	116.92	3.83	9.546	135.46	1.99	3.962	158.86	*(b)
L20	oNO-PE	31.836	142.19	3.67	12.512	165.12	2.05	5.112	193.36	1.18
L21	mNO-PE	38.891	164.84	0	14.657	186.57	0	5.807	214.21	0
L22	pNO-PE	42.491	176.40	0	15.745	197.45	0	6.119	223.57	0
L23	pEt-PE	25.106	120.59	5.71	10.033	140.33	3.19	4.160	164.80	0
L24	pBu-PE	30.179	136.87	2.94	11.798	157.98	1.50	4.820	184.60	0
L25	ptBu-PE	27.304	127.65	1.68	10.904	149.04	*(b)	4.527	175.81	0
L26	pPh-PE	42.497	176.42	1.56	15.965	199.65	*(b)	6.287	228.61	0
L27	24F-PE	18.362	98.94	2.25	7.846	118.46	1.55	3.429	142.87	1.08
L28	25F-PE	19.195	101.62	2.33	8.134	121.34	1.69	3.535	146.05	1.13
L29	34F-PE	24.946	120.08	2.21	9.842	138.42	1.12	4.053	161.59	0
L30	26F-PE	14.128	85.35	0	6.590	105.90	0	3.063	131.89	0
L31	35F-PE	22.550	112.39	6.83	9.037	130.37	3.50	3.788	153.64	1.38
L32	triF-PE	21.514	109.06	3.18	8.785	127.85	1.94	3.702	151.06	0
L33	tetraF-PE	21.462	108.89	*(b)	8.836	128.36	*(b)	3.756	152.68	0
L34	pentaF-PE	19.223	101.71	3.98	8.028	120.28	2.23	3.444	143.32	0
L35	24Cl-PE	28.626	131.89	0	11.483	154.83	0	4.768	183.04	0
L36	25Cl-PE	29.018	133.15	0	11.626	156.26	0	4.816	184.48	0
L37	34Cl-PE	34.965	152.24	3.37	13.304	173.04	1.81	5.318	199.54	0

alcohol		rate 3.21 °C/min			rate 10.00 °C/min			rate 30.00 °C/min		
no.	abbr.	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs
L38	24Me-PE	22.343	111.72	0	9.408	134.08	0	4.054	161.62	0
L39	25Me-PE	21.691	109.63	*(b)	9.229	132.29	0	4.001	160.03	0
L40	34Me-PE	25.899	123.14	7.68	10.273	142.73	4.13	4.270	168.10	1.80
L41	PP	20.578	106.06	4.49	8.582	125.82	2.20	3.689	150.67	0
L42	pF-PP	23.320	114.86	4.32	9.407	134.07	2.06	3.941	158.23	0
L43	pCl-PP	29.760	135.53	2.89	11.601	156.01	1.50	4.733	181.99	0
L44	pBr-PP	32.598	144.64	2.16	12.575	165.75	1.06	5.087	192.61	0
L45	pMe-PP	23.758	116.26	5.73	9.635	136.35	3.09	4.069	162.07	1.18
L46	pOMe-PP	29.740	135.47	3.35	11.679	156.79	1.55	4.786	183.58	0
L47	pCF3-PP	22.550	112.39	6.83	10.034	140.34	1.84	4.150	164.50	0
L48	1OH	20.297	105.15	2.22	8.492	124.92	1.42	3.665	149.95	0
L49	2OH	20.140	104.65	0	8.615	126.15	0	3.768	153.04	0
L50	3OH	20.973	107.32	1.24	8.977	129.77	*(b)	3.917	157.51	0
L51	4OH	22.518	112.28	0	9.345	133.45	0	4.000	160.00	0
L52	5OH	22.485	112.18	0	9.274	132.74	0	3.967	159.01	0
L53	6OH	24.931	120.03	1.25	10.034	140.34	*(b)	4.203	166.09	0
L54	7OH	23.344	114.93	*(b)	9.672	136.72	0	4.132	163.96	0
L55	8OH	21.034	107.52	7.06	8.785	127.85	4.31	3.790	153.70	2.32
L56	DPE	37.933	161.76	0	14.569	185.69	0	5.839	215.17	0

alcohol		rate 3.21 °C/min			rate 10.00 °C/min			rate 30.00 °C/min		
no.	abbr.	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs
L57	In	23.172	114.38	0	9.597	135.97	0	4.092	162.76	0
L58	1-Nap	25.797	122.81	0	10.587	145.87	0	4.477	174.31	0
L59	2-Nap	27.649	128.75	*(b)	11.173	151.73	0	4.669	180.07	0
L60	1Nap-E	34.626	151.15	0	13.581	175.81	0	5.525	205.75	0
L61	2Nap-E	37.501	160.38	2.90	14.260	182.60	1.24	5.686	210.58	0
L62	pF-Ph	36.816	158.18	0	14.195	181.95	0	5.702	211.06	0
L63	pCl-Ph	42.824	177.47	0	16.224	202.24	0	6.415	232.45	0
L64	pBr-Ph	42.262	175.66	0	17.186	211.86	0	6.038	221.14	0
L65	oMe-Ph	37.567	160.59	0	14.570	185.70	0	5.869	216.07	0
L66	mMe-Ph	37.696	161.00	0	14.585	185.85	0	5.860	215.80	0
L67	pMe-Ph	38.242	162.76	0	14.759	187.59	0	5.921	217.63	0
L68	pOMe-Ph	43.898	180.91	0	16.600	206.00	0	6.550	236.50	0
L69	2Ph-Bu	21.838	110.10	2.99	9.196	131.96	1.60	3.975	159.25	0
L70	pF-CF	24.913	119.97	2.50	9.824	138.24	1.20	4.039	161.17	0
L71	pCl- CF	30.423	137.66	1.47	11.719	157.19	*(b)	4.722	181.66	0
L72	pBr-CF	33.093	146.23	1.30	12.634	166.34	*(b)	5.053	191.59	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

4.1.3 Analyses of chiral amines by temperature program and isothermal condition

Forty-three chiral amines were used in this study and they were analyzed as trifluoroacetyl (TFA) derivatives. Amine structures were mainly based on 1-phenylethylamines with different type (e.g. F, Cl, Br, Me, CF_3) and position (*ortho*, *meta, para*) of substitution. Other structures included mono-substituted 1-phenylpropylamines, mono-substituted 1-aminoindanes, mono-substituted diphenylmethanamines and other amines. Each amine was analyzed by GC using a temperature program starting from 40 °C with a rate of 3.21 °C/min, similar to analyses of chiral alcohols in 4.1.1. The elution temperatures for all eluted peaks and resolution (Rs) between two enantiomeric peaks were calculated as shown in **Table 4.4**. The optimum isothermal condition for each amine was also determined using the same procedures as described in 4.1.2. Chromatographic results for 43 amines obtained from temperature program condition and isothermal conditions with Rs values closed to 1.5 and 2.0 were compared in **Table 4.4**.

From **Table 4.4**, 35 amines could be completely separated (Rs ≥ 1.5) into their enantiomers by temperature program. Optimum isothermal conditions (Rs ~ 1.5) for all 35 amines could be determined and their analysis times using isothermal condition were much shorter than using temperature program. For example, amine **A15** eluted at 35.076 minutes by temperature program, but it could be completely separated in less than 1.1 minutes at its optimum isothermal condition of 193 °C (**Figure 4.6**). In addition, separation of amine **A15** with Rs = 2.06 was also obtained in less than 1.2 minutes. For four amines analyzed by temperature program with incomplete separation (1.0 < Rs < 1.5), three of them could be completely separated using isothermal condition with slightly longer retention. Only 5 amines could not be completely separated into their enantiomers either by temperature program or isothermal condition, which all of them showed no separation or were separated with Rs < 1.0 by temperature program.

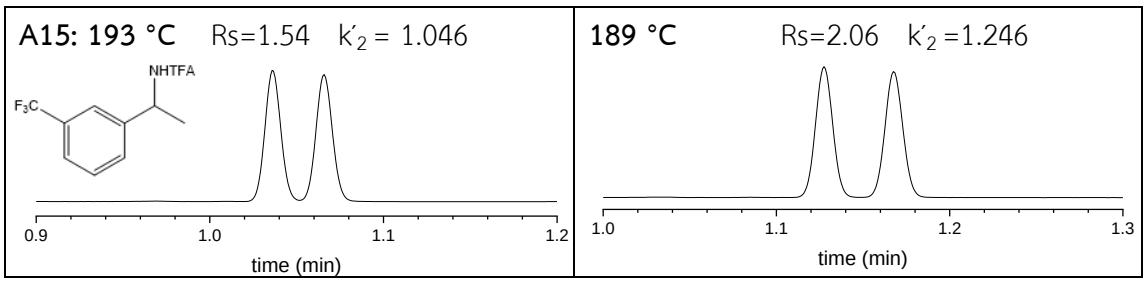


Figure 4.6 Separation of **A15** on BSiAc column using isothermal conditions.

Table 4.4 Chromatographic results of 43 chiral amines analyzed by BSiAc column (15.57 m long) using temperature program and isothermal conditions.

amine		temperature program rate 3.21 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$(°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
A01	1	30.715	138.60	1.10	120	18.769	34.819	1.022	1.59	-	-	-	-	-
A02	oF-1	27.108	127.02	8.27	150	1.971	2.783	1.036	1.53	145	2.373	3.537	1.044	2.00
A03	mF-1	34.168	149.68	4.39	175	1.675	2.215	1.041	1.59	170	2.013	2.864	1.048	2.02
A04	pF-1	34.665	151.27	2.49	151	5.607	9.762	1.030	1.57	145	8.123	14.532	1.037	2.01
A05	oCl-1	31.171	140.06	2.19	130	10.941	19.880	1.026	1.52	-	-	-	-	-
A06	mCl-1	37.861	161.53	10.76	196	1.485	1.850	1.041	1.51	190	1.764	2.386	1.052	2.08
A07	pCl-1	39.850	167.92	2.05	165	7.010	12.455	1.029	1.55	159	10.099	18.384	1.034	2.02
A08	oBr-1	33.474	147.45	1.87	133	13.948	25.618	1.025	1.58	-	-	-	-	-
A09	mBr-1	39.838	167.88	10.30	199	1.766	2.390	1.038	1.52	193	2.126	3.081	1.047	2.05
A10	pBr-1	42.041	174.95	1.23	159	16.158	29.954	1.021	1.50	-	-	-	-	-
A11	oMe-1	30.904	139.20	5.25	163	1.946	2.728	1.037	1.58	158	2.324	3.452	1.045	2.06
A12	mMe-1	30.663	138.43	9.15	166	1.793	2.435	1.038	1.58	161	2.113	3.048	1.045	2.00
A13	pMe-1	31.530	141.21	5.74	160	2.413	3.623	1.033	1.53	154	3.073	4.887	1.041	2.04
A14	oCF-1	27.204	127.32	2.74	133	4.620	7.817	1.030	1.53	123	7.607	13.490	1.039	2.08

amine		temperature program rate 3.21 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$(°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
A15	mCF-1	35.076	152.59	15.09	193	1.066	1.046	1.058	1.54	189	1.168	1.246	1.066	2.06
A16	pCF-1	36.890	158.42	9.49	190	1.334	1.565	1.046	1.58	185	1.558	1.996	1.056	2.05
A17	pEt	33.347	147.04	6.94	167	2.460	3.722	1.033	1.57	162	2.977	4.714	1.041	2.01
A18	pBu	37.606	160.72	4.67	171	3.997	6.672	1.030	1.55	165	5.189	8.998	1.038	2.02
A19	ptBu	35.901	155.24	7.48	176	2.532	3.860	1.033	1.56	171	3.050	4.854	1.041	2.03
A20	2	30.114	136.67	7.47	164	1.844	2.539	1.037	1.51	158	2.254	3.326	1.046	2.07
A21	pF-2	33.845	148.64	8.31	181	1.491	1.862	1.043	1.58	175	1.788	2.432	1.051	2.09
A22	pCl-2	39.206	165.85	6.54	192	2.029	2.894	1.036	1.59	186	2.496	3.791	1.044	2.06
A23	pBr-2	41.454	173.07	5.24	192	2.806	4.386	1.032	1.56	185	3.698	6.112	1.041	2.09
A24	pMe-2	35.562	154.15	12.06	188	1.375	1.639	1.044	1.58	183	1.593	2.058	1.053	2.04
A25	pOMe-2	36.452	157.01	4.43	166	4.414	7.472	1.029	1.58	160	5.746	10.029	1.037	2.02
A26	pCF-2	31.354	140.65	8.34	165	2.233	3.286	1.034	1.54	159	2.754	4.276	1.043	2.08
A27	iBu	29.531	134.79	5.73	152	2.820	4.402	1.033	1.59	146	3.575	5.836	1.041	2.06
A28	3	30.399	137.58	1.65	128	10.443	18.929	1.027	1.55	-	-	-	-	-
A29	4	35.777	154.84	2.80	156	6.180	10.839	1.029	1.56	148	9.242	16.705	1.037	2.09
A30	A	36.246	156.35	11.31	188	1.564	2.002	1.040	1.55	182	1.881	2.610	1.050	2.08
A31	5F-A	39.766	167.65	7.49	193	1.833	2.518	1.036	1.58	187	2.283	3.390	1.046	2.06

amine		temperature program rate 3.21 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$(°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
A32	5Cl-A	44.811	183.84	5.13	199	3.043	4.841	1.032	1.56	194	3.778	6.251	1.039	2.02
A33	5Br-A	47.060	191.06	4.64	205	3.408	5.541	1.031	1.51	199	4.423	7.489	1.039	2.02
A34	5Me-A	37.031	158.87	9.59	185	2.035	2.906	1.035	1.54	180	2.405	3.616	1.044	2.03
A35	5OMe-A	41.546	173.36	4.87	182	4.392	7.430	1.029	1.52	176	5.756	10.048	1.038	2.07
A36	1ATL	36.378	156.77	10.34	182	2.207	3.244	1.035	1.54	178	2.520	3.846	1.042	2.01
A37	2ATL	39.742	167.57	4.60	188	2.594	3.979	1.032	1.53	180	3.546	5.819	1.041	2.05
A38	Nap	40.642	170.46	0	-	-	-	-	-	-	-	-	-	-
A39	pF-6	41.213	172.29	*(b)	-	-	-	-	-	-	-	-	-	-
A40	pCl-6	46.655	189.76	1.18	-	-	-	-	-	-	-	-	-	-
A41	pBr-6	49.198	197.93	1.24	179	19.450	36.332	1.021	1.51	-	-	-	-	-
A42	pMe-6	41.495	173.20	0	-	-	-	-	-	-	-	-	-	-
A43	pOMe-6	46.536	189.38	0	-	-	-	-	-	-	-	-	-	-

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

4.2 Enantiomeric separation using BSiMe column

Another type of β-CD derivative with different polarity from BSiAc, heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or BSiMe), was used as a chiral stationary phase. Enantiomeric separations of seventy-two chiral alcohols and forty-three chiral amines were performed using the same procedures as described in 4.1.1 and 4.1.2.

4.2.1 Analyses of chiral alcohols by temperature program and isothermal condition

From **Table 4.5**, 55 alcohols could be completely separated (Rs ≥ 1.5) into their enantiomers by temperature program. Optimum isothermal conditions (Rs ~ 1.5) for all 55 alcohols could be determined and their analysis times using isothermal condition were much shorter than using temperature program. For seven alcohols analyzed by temperature program with incomplete separation (1.0 < Rs < 1.5), six of them could be completely separated using isothermal condition with slightly longer retention. Eleven alcohols could not be completely separated into their enantiomers either by temperature program or isothermal condition, which all of them showed no separation or were separated with Rs < 1.1 by temperature program.

Analyses of chiral alcohols using BSiMe column were compared to those obtained from BSiAc column (**Table 4.2**). It was found that BSiMe column was suitable for enantiomeric separation of chiral alcohols than BSiAc column, as more alcohols could be completely separated and less alcohols could not be separated on BSiMe column. In addition, complete separations of alcohols on BSiMe column could be obtained in shorter analysis times. Most alcohols that could not be separated on BSiMe column, could not be separated on BSiAc column either. The only exception is alcohol **L31**, it could not be separated on BSiMe column but it showed complete separation with short analysis time (2.705 minutes, Rs = 1.54) on BSiAc column.

Table 4.5 Chromatographic results of 72 chiral alcohols analyzed by BSiMe column (14.91 m long) using temperature program and isothermal conditions.

alcohol		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L01	PE	21.061	110.55	5.30	135	2.162	3.290	1.040	1.55	128	2.794	4.522	1.049	2.06
L02	oF-PE	22.384	114.99	14.06	156	1.178	1.342	1.051	1.57	150	1.377	1.732	1.065	2.09
L03	mF-PE	22.673	115.95	4.19	137	2.396	3.754	1.036	1.56	128	3.439	5.796	1.045	2.03
L04	pF-PE	22.207	114.39	5.45	133	2.697	4.341	1.036	1.55	126	3.575	6.065	1.046	2.09
L05	oCl-PE	29.946	140.32	29.44	192	0.966	0.924	1.077	1.51	186	1.086	1.168	1.093	2.05
L06	mCl-PE	28.615	135.86	3.17	145	4.171	7.276	1.032	1.53	135	6.624	12.117	1.042	2.08
L07	pCl-PE	28.329	134.90	4.58	145	4.170	7.323	1.032	1.53	140	5.173	9.264	1.041	2.01
L08	oBr-PE	33.036	150.67	31.05	206	0.934	0.864	1.072	1.54	200	1.035	1.066	1.085	2.09
L09	mBr-PE	31.339	144.99	2.16	140	8.334	15.503	1.029	1.51	132	12.668	24.036	1.036	2.03
L10	pBr-PE	31.291	144.82	3.14	146	6.421	11.715	1.030	1.55	139	8.920	16.698	1.038	2.04
L11	oMe-PE	26.407	128.46	12.56	162	1.511	2.010	1.046	1.60	157	1.765	2.502	1.056	2.09
L12	mMe-PE	24.177	120.99	6.19	150	1.822	2.615	1.039	1.50	141	2.497	3.974	1.051	2.08
L13	pME-PE	23.124	117.47	7.72	147	1.921	2.812	1.038	1.55	140	2.422	3.825	1.048	2.09
L14	oOMe-PE	28.875	136.73	21.24	192	0.959	0.914	1.068	1.55	187	1.048	1.092	1.079	2.06

alcohol		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L15	mOMe-PE	28.764	136.36	5.04	155	2.822	4.622	1.035	1.52	148	3.724	6.418	1.043	2.03
L16	pOMe-PE	22.079	113.96	2.99	125	4.062	7.012	1.032	1.51	114	6.452	11.726	1.040	2.06
L17	oCF-PE	24.358	121.60	34.14	175	0.829	0.655	1.083	1.53	171	0.886	0.765	1.100	2.01
L18	mCF-PE	22.763	116.26	3.70	130	3.076	5.079	1.033	1.52	121	4.618	8.091	1.042	2.01
L19	pCF-PE	23.481	118.66	7.25	148	1.838	2.647	1.039	1.53	141	2.336	3.626	1.049	2.04
L20	oNO-PE	37.932	167.07	29.61	222	0.971	0.938	1.066	1.57	216	1.080	1.160	1.082	2.00
L21	mNO-PE	38.189	167.93	1.08	-	-	-	-	-	-	-	-	-	-
L22	pNO-PE	39.415	172.04	3.01	141	7.515	13.881	1.029	1.55	164	10.465	19.764	1.038	2.05
L23	pEt-PE	25.853	126.61	3.66	137	4.060	7.056	1.032	1.51	127	6.318	11.462	1.043	2.09
L24	pBu-PE	31.413	145.23	2.87	146	6.494	11.911	1.030	1.54	138	9.517	17.846	1.038	2.06
L25	ptBu-PE	29.861	140.03	1.29	122	16.680	31.903	1.023	1.54	112	29.620	57.193	1.029	2.09
L26	pPh-PE	42.799	183.38	1.39	164	18.980	36.880	1.023	1.54	157	27.645	53.851	1.028	2.06
L27	24F-PE	23.172	117.63	25.01	169	0.910	0.813	1.071	1.51	163	1.023	1.038	1.088	2.08
L28	25F-PE	24.804	123.09	14.32	167	1.046	1.084	1.060	1.54	162	1.179	1.344	1.070	2.02
L29	34F-PE	23.542	118.87	6.80	149	1.785	2.542	1.041	1.58	142	2.260	3.484	1.051	2.09
L30	26F-PE	19.377	104.91	18.47	155	0.988	0.968	1.066	1.56	150	1.090	1.163	1.081	2.03
L31	35F-PE	22.502	115.38	0	-	-	-	-	-	-	-	-	-	-

alcohol		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L32	triF-PE	25.584	125.71	36.31	185	0.783	0.563	1.124	1.53	182	0.811	0.619	1.140	2.01
L33	tetraF-PE	22.404	115.05	6.32	137	2.244	3.452	1.036	1.51	130	2.956	4.900	1.047	2.02
L34	pentaF-PE	19.572	105.57	13.32	151	1.078	1.143	1.053	1.50	144	1.276	1.537	1.068	2.01
L35	24Cl-PE	34.941	157.05	25.78	207	1.091	1.182	1.059	1.69	203	1.181	1.357	1.068	2.07
L36	25Cl-PE	36.864	163.49	26.55	220	0.927	0.850	1.073	1.58	215	1.005	1.006	1.084	2.04
L37	34Cl-PE	34.585	155.86	2.69	154	7.480	13.871	1.029	1.52	147	10.512	19.857	1.036	2.03
L38	24Me-PE	28.803	136.49	14.93	173	1.431	1.856	1.044	1.54	167	1.701	2.388	1.056	2.05
L39	25Me-PE	28.968	137.04	15.15	182	1.148	1.291	1.054	1.59	176	1.322	1.633	1.065	2.04
L40	34Me-PE	27.046	130.60	1.56	117	12.760	24.108	1.026	1.50	110	18.839	36.085	1.032	2.19
L41	PP	23.775	119.65	1.88	108	11.190	20.935	1.027	1.54	102	15.614	29.616	1.034	2.01
L42	pF-PP	25.117	124.14	3.08	125	5.862	10.562	1.032	1.56	119	7.940	14.692	1.040	2.03
L43	pCl-PP	30.850	143.35	2.31	136	9.423	17.659	1.029	1.50	130	12.958	24.659	1.035	2.05
L44	pBr-PP	33.680	152.83	1.35	134	17.700	34.127	1.022	1.50	127	26.137	50.756	1.030	2.07
L45	pMe-PP	25.755	126.28	4.79	141	3.301	5.550	1.033	1.53	132	4.836	8.576	1.044	2.01
L46	pOMe-PP	30.726	142.93	2.26	138	8.435	15.736	1.029	1.56	131	11.986	22.735	1.036	2.01
L47	pCF3-PP	26.139	127.57	4.33	148	2.526	4.022	1.034	1.56	139	3.634	6.210	1.043	2.05
L48	1OH	21.229	111.12	2.90	127	3.174	5.285	1.035	1.51	114	5.477	9.760	1.046	2.02

alcohol		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L49	2OH	25.342	124.90	0	-	-	-	-	-	-	-	-	-	-
L50	3OH	26.709	129.48	3.32	158	1.962	2.901	1.038	1.54	147	2.901	4.756	1.044	2.03
L51	4OH	25.988	127.06	3.15	146	2.780	4.527	1.035	1.54	135	4.358	7.630	1.043	2.04
L52	5OH	23.981	120.34	4.21	145	2.379	3.711	1.036	1.59	135	3.420	5.772	1.045	2.04
L53	6OH	25.327	124.85	1.96	116	9.862	18.413	1.029	1.54	111	12.798	24.143	1.034	2.07
L54	7OH	26.051	127.27	*(b)	-	-	-	-	-	-	-	-	-	-
L55	8OH	23.387	118.35	2.85	135	3.072	5.095	1.034	1.54	120	5.821	10.481	1.043	2.06
L56	DPE	39.501	172.33	*(b)	-	-	-		-	-	-	-	-	-
L57	In	25.896	126.75	*(b)	-	-	-	-	-	-	-	-	-	-
L58	1-Nap	30.334	141.62	2.30	131	11.250	21.323	1.028	1.50	127	13.862	26.341	1.035	2.04
L59	2-Nap	30.904	143.53	0	-	-	-	-	-	-	-	-		-
L60	1Nap-E	38.175	167.89	7.42	206	1.776	2.545	1.039	1.50	197	2.295	3.572	1.050	2.05
L61	2Nap-E	37.157	164.48	2.27	157	10.250	19.384	1.028	1.51	151	13.704	26.190	1.034	2.12
L62	pF-Ph	38.626	169.40	0	-	-	-	-	-	-	-	-	-	-
L63	pCl-Ph	44.057	187.59	1.09	164	23.670	46.155	1.023	1.58	155	39.255	77.042	1.028	2.03
L64	pBr-Ph	46.642	196.25	1.01	169	29.270	57.193	1.021	1.55	159	51.454	101.091	1.027	2.06
L65	oMe-Ph	39.380	171.92	0	-	-	-	-	-	-	-	-	-	-

alcohol		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
L66	mMe-Ph	39.830	173.43	2.24	172	7.657	14.253	1.029	1.52	164	11.176	21.131	1.035	2.02
L67	pMe-Ph	39.989	173.96	1.69	162	12.830	24.562	1.026	1.51	155	18.314	35.410	1.030	2.09
L68	pOMe-Ph	44.335	188.52	1.29	169	19.160	37.173	1.023	1.54	160	30.734	60.101	1.028	2.00
L69	2Ph-Bu	24.771	122.98	1.68	111	11.570	21.725	1.028	1.58	105	15.980	30.272	1.033	2.05
L70	pF-CF	25.138	124.21	1.69	141	2.739	4.435	1.035	1.50	133	10.925	20.464	1.035	2.01
L71	pCl- CF	30.449	142.00	0	-	-	-	-	-	-	-	-	-	-
L72	pBr-CF	33.217	151.28	0	-	-	-	-	-	-	-	-	-	-

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

4.2.2 Analyses of chiral amines by temperature program and isothermal condition

From **Table 4.6**, only 15 amines could be completely separated (Rs ≥ 1.5) into their enantiomers by temperature program. Optimum isothermal conditions (Rs ~ 1.5) for all 15 amines could be determined and their analysis times using isothermal condition were much shorter than using temperature program. For four amines analyzed by temperature program with incomplete separation (1.0 < Rs < 1.5), three of them could be completely separated using isothermal condition with slightly longer retention. Total of 25 amines could not be completely separated into their enantiomers either by temperature program or isothermal condition, which all of them showed no separation or were separated with Rs < 1.10 by temperature program.

Analyses of chiral amines using BSiMe column were compared to those obtained from BSiAc column (**Table 4.4**). It was found that BSiAc column was suitable for enantiomeric separation of chiral alcohols than BSiMe column, as more amines could be completely separated and less amines could not be separated on BSiAc column.

Table 4.6 Chromatographic results of 43 chiral amines analyzed by BSiMe column (14.91 m long) using temperature program and isothermal conditions.

amine		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
A01	1	23.355	118.24	2.66	127	4.266	7.431	1.032	1.56	115	7.338	13.445	1.039	2.07
A02	oF-1	21.527	112.12	5.51	139	2.126	3.218	1.036	1.58	132	2.701	4.349	1.044	2.02
A03	mF-1	25.256	124.61	4.27	147	2.443	3.857	1.035	1.55	139	3.303	5.554	1.042	2.03
A04	pF-1	25.198	124.41	3.61	142	3.008	4.968	1.032	1.54	132	4.508	7.927	1.041	2.06
A05	oCl-1	27.900	133.47	5.87	155	2.740	4.447	1.033	1.54	148	3.537	6.018	1.042	2.02
A06	mCl-1	30.796	143.17	3.24	152	4.519	7.984	1.030	1.54	143	6.716	12.325	1.038	2.00
A07	pCl-1	31.325	144.94	1.04	121	23.077	44.517	1.022	1.58	111	41.267	80.075	1.027	2.09
A08	oBr-1	30.626	142.60	4.23	154	4.180	7.310	1.031	1.58	147	5.587	10.107	1.038	2.01
A09	mBr-1	33.489	152.19	2.42	150	7.625	14.159	1.029	1.59	142	11.196	21.214	1.036	2.08
A10	pBr-1	34.387	155.20	*(a)	-	-	-	-	-	-	-	-	-	-
A11	oMe-1	24.836	123.20	0	-	-	-	-	-	-	-	-	-	-
A12	mMe-1	25.499	125.42	1.26	110	13.886	26.281	1.023	1.53	102	21.764	41.591	1.028	2.00
A13	pMe-1	25.720	126.16	0	-	-	-	-	-	-	-	-	-	-
A14	oCF-1	23.578	118.99	1.15	102	14.922	28.202	1.023	1.50	93	25.366	48.350	1.027	2.00

amine		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
A15	mCF-1	25.766	126.32	1.78	120	8.436	15.639	1.028	1.51	112	13.032	24.603	1.033	2.03
A16	pCF-1	27.135	130.90	*(b)	-	-	-	-	-	-	-	-	-	-
A17	pEt	29.029	137.24	0	-	-	-	-	-	-	-	-	-	-
A18	pBu	34.265	154.79	0	-	-	-	-	-	-	-	-	-	-
A19	ptBu	33.235	151.34	1.59	139	12.244	23.294	1.025	1.54	133	17.115	32.891	1.030	2.06
A20	2	24.975	123.67	0	-	-	-	-	-	-	-	-	-	-
A21	pF-2	26.873	130.02	1.64	120	10.542	19.793	1.025	1.54	114	14.504	27.495	1.029	2.07
A22	pCl-2	32.852	150.05	0	-	-	-	-	-	-	-	-	-	-
A23	pBr-2	35.826	160.02	0	-	-	-	-	-	-	-	-	-	-
A24	pMe-2	28.536	135.60	0	-	-	-	-	-	-	-	-	-	-
A25	pOMe-2	32.773	149.79	0	-	-	-	-	-	-	-	-	-	-
A26	pCF-2	27.552	132.30	0	-	-	-	-	-	-	-	-	-	-
A27	iBu	25.450	125.26	*(b)	-	-	-	-	-	-	-	-	-	-
A28	3	27.182	131.06	*(b)	-	-	-	-	-	-	-	-	-	-
A29	4	32.771	149.78	0	-	-	-	-	-	-	-	-	-	-
A30	A	29.911	140.20	2.41	142	6.148	11.247	1.029	1.53	131	10.390	19.534	1.038	2.04
A31	5F-A	31.511	145.56	2.66	148	5.973	10.898	1.030	1.56	139	9.161	17.213	1.037	2.01

amine		temperature program rate 3.35 °C/min			isothermal									
					Rs ~ 1.5					Rs ~ 2.0				
no.	abbreviation	$t_{R,2}$ (min)	elution $temp_2$ (°C)	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs	temp (°C)	$t_{R,2}$ (min)	k'_2	α	Rs
A32	5Cl-A	37.199	164.62	0	-	-	-	-	-	-	-	-	-	-
A33	5Br-A	40.108	174.36	*(b)	-	-	-	-	-	-	-	-	-	-
A34	5Me-A	31.763	146.41	1.10	-	-	-	-	-	-	-	-	-	-
A35	5OMe-A	36.567	162.50	0	-	-	-	-	-	-	-	-	-	-
A36	1ATL	32.863	150.09	4.99	173	2.715	4.419	1.033	1.54	166	3.489	5.964	1.040	2.00
A37	2ATL	34.272	154.81	1.81	150	8.810	16.550	1.028	1.52	141	13.649	26.081	1.033	2.01
A38	Nap	37.922	167.04	0	-	-	-	-	-	-	-	-	-	-
A39	pF-6	37.993	167.28	0	-	-	-	-	-	-	-	-	-	-
A40	pCl-6	43.565	185.94	0	-	-	-	-	-	-	-	-	-	-
A41	pBr-6	46.279	195.04	0	-	-	-	-	-	-	-	-	-	-
A42	pMe-6	39.579	172.59	0	-	-	-	-	-	-	-	-	-	-
A43	pOMe-6	44.195	188.05	0	-	-	-	-	-	-	-	-	-	-

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0..

4.3 Relationship between chromatographic data obtained from temperature program runs and optimum conditions

Chromatographic data obtained from temperature program runs and from the optimum isothermal conditions were studied for their relationship hoping that some information from temperature program runs can be used as a guideline to quickly determine the optimum separation condition. From **Table 4.2**, alcohol **L13** showed the highest Rs value of 8.37 from a temperature program run with elution temperature of 113.95 °C. Its optimum isothermal condition (Rs = 1.57) was at 137 °C, **L13** showed analysis time of 2.039 minutes with k'_2 = 2.906. The optimum isothermal condition was ~ 23 °C higher than its elution temperature. Chromatographic data (**Table 4.2**) from analyses of chiral alcohols on BSiAc column temperature program runs were sorted by Rs values in decreasing order and were shown in **Figure 4.7**. Chromatographic data from analyses of chiral alcohols on BSiMe column (**Table 4.5**) were treated in a similar fashion and were shown in **Figure 4.8**. Chromatographic data from analyses of chiral amines on BSiAc and BSiMe columns (**Table 4.4** and **Table 4.6**) were treated in a similar fashion and were shown in **Figure 4.9** and **Figure 4.10**, respectively.

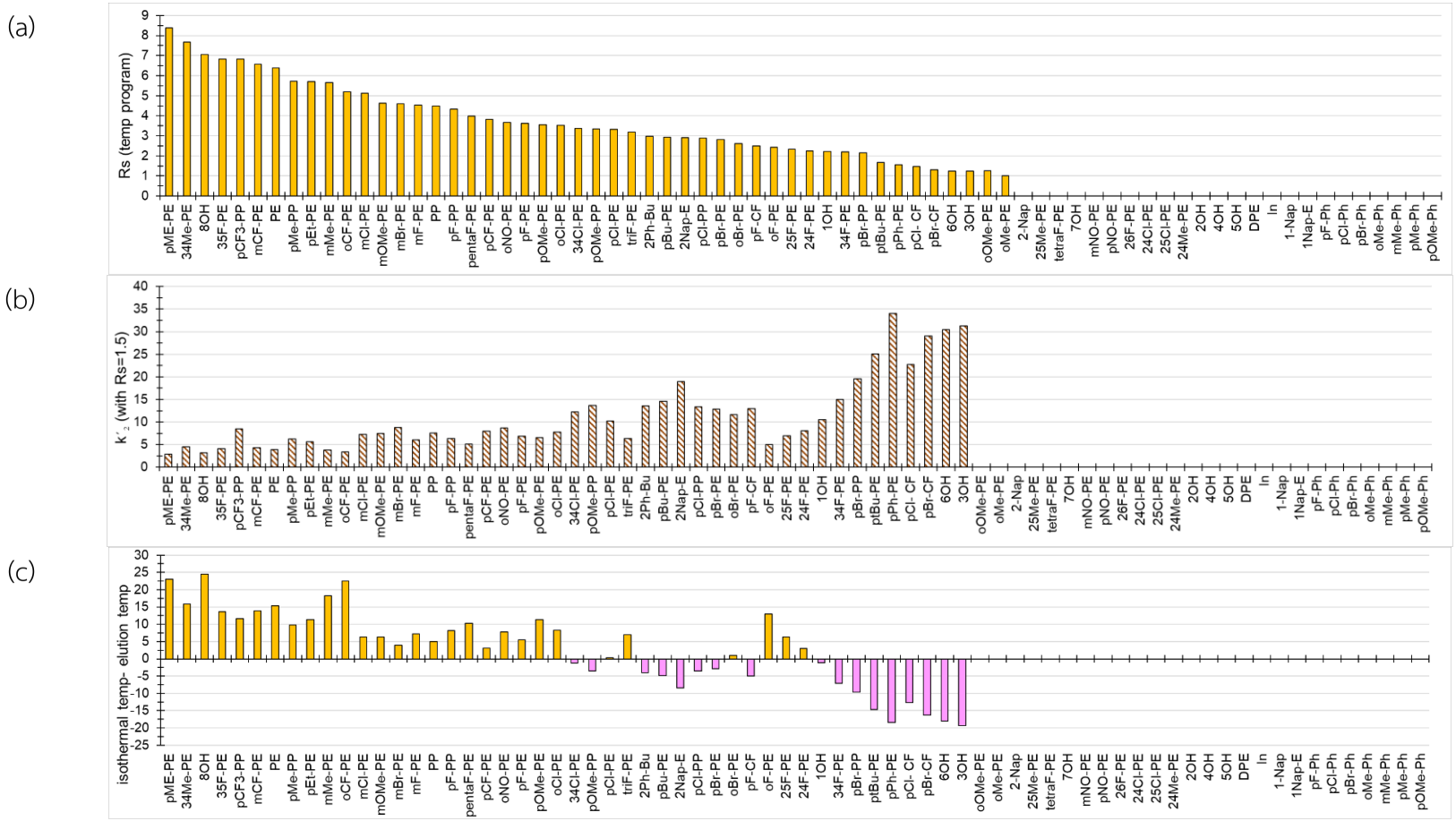


Figure 4.7 Chromatographic results of 72 chiral alcohols analyzed by BSiAc column.

(a) Rs values obtained from temperature program runs in decreasing order; (b) k'_2 values at optimum isothermal condition (Rs ~ 1.5); (c) temperature difference (optimum isothermal temperature – elution temperature).

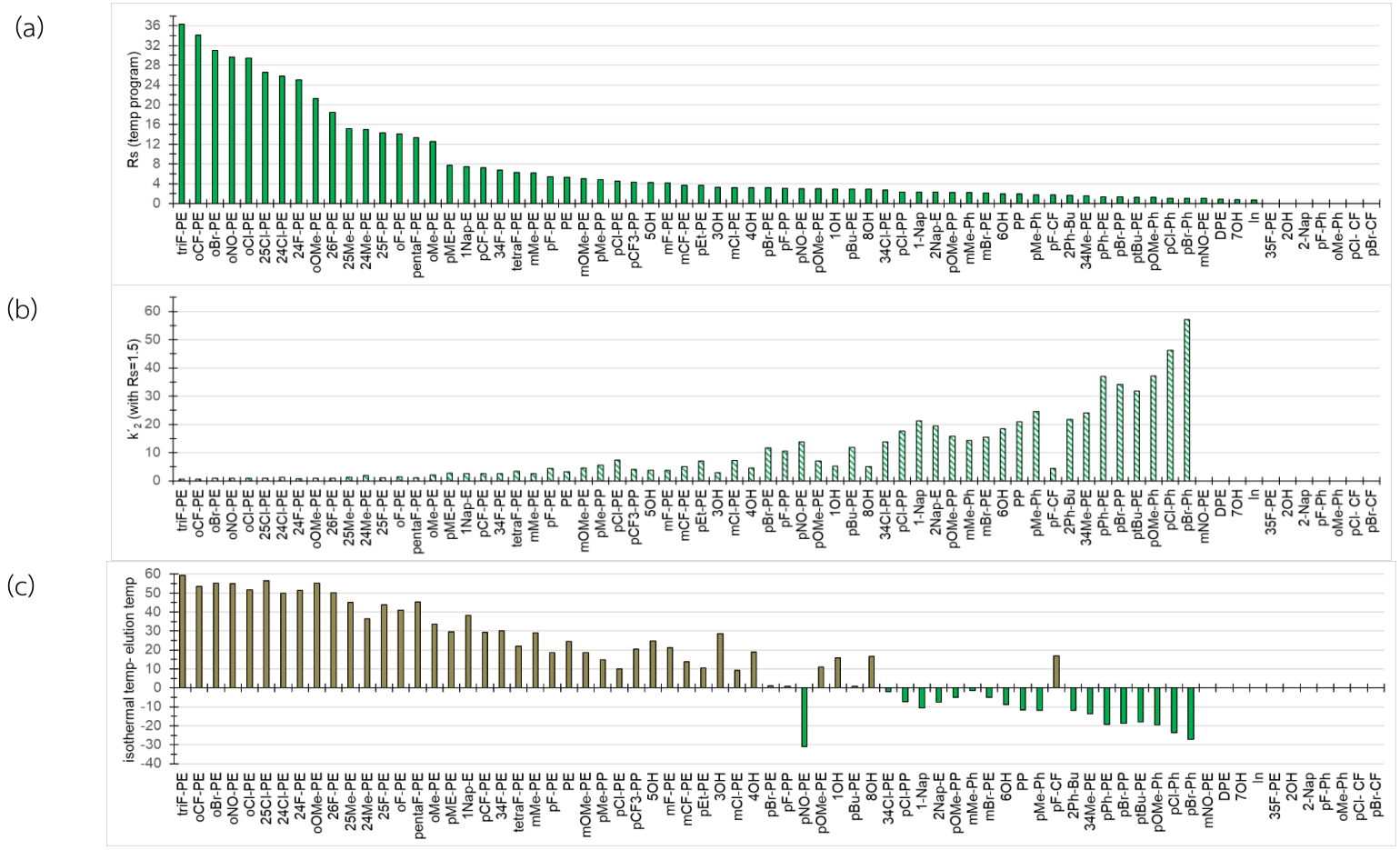


Figure 4.8 Chromatographic results of 72 chiral alcohols analyzed by BSiMe column.

(a) Rs values obtained from temperature program runs in decreasing order; (b) k'_2 values at optimum isothermal condition (Rs ~ 1.5);

(c) temperature difference (optimum isothermal temperature – elution temperature).

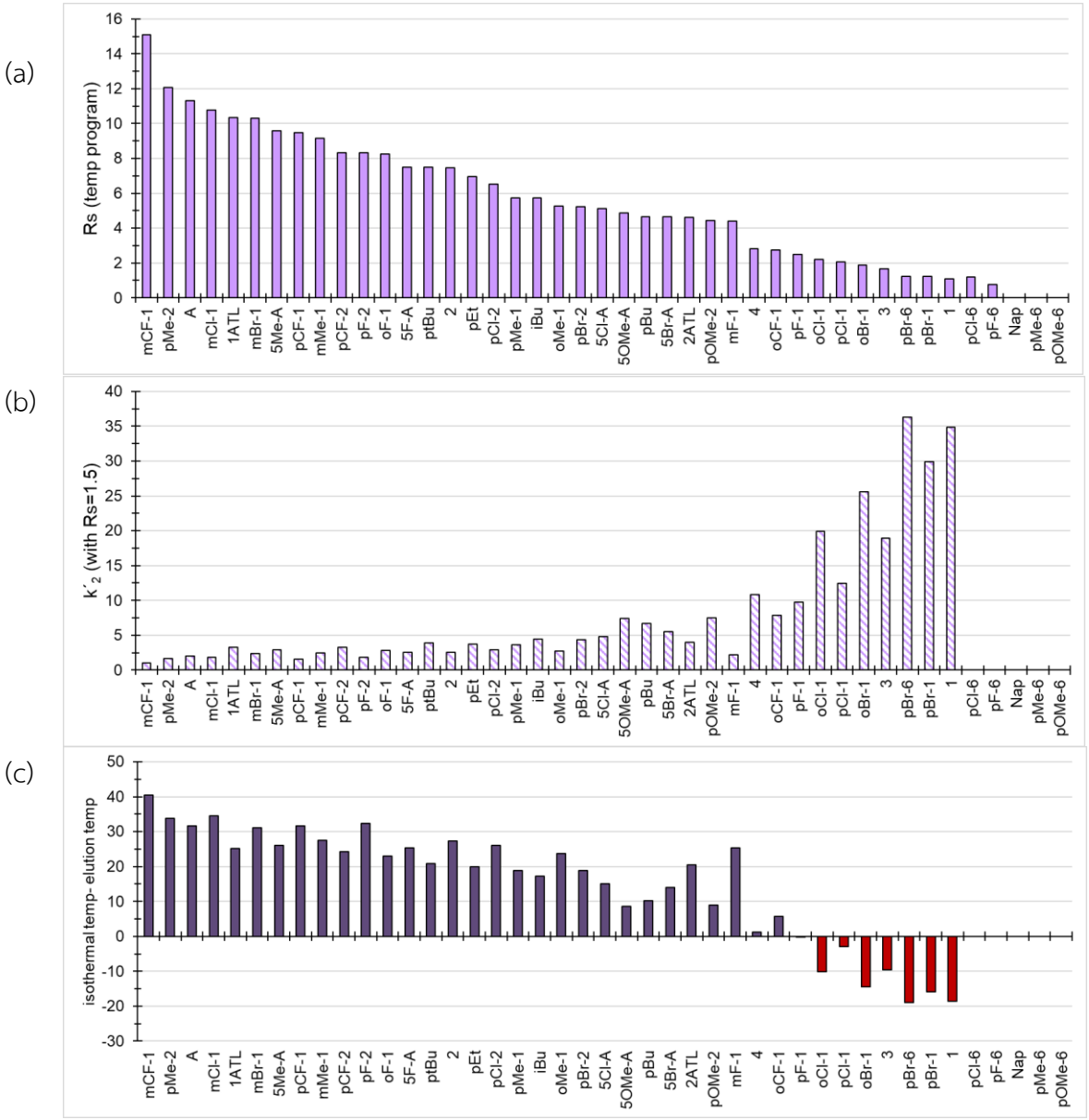


Figure 4.9 Chromatographic results of 43 chiral amines analyzed by BSiAc column.

(a) Rs values obtained from temperature program runs in decreasing order;

(b) k'_2 values at optimum isothermal condition (Rs ~ 1.5);

(c) temperature difference (optimum isothermal temperature – elution temperature).

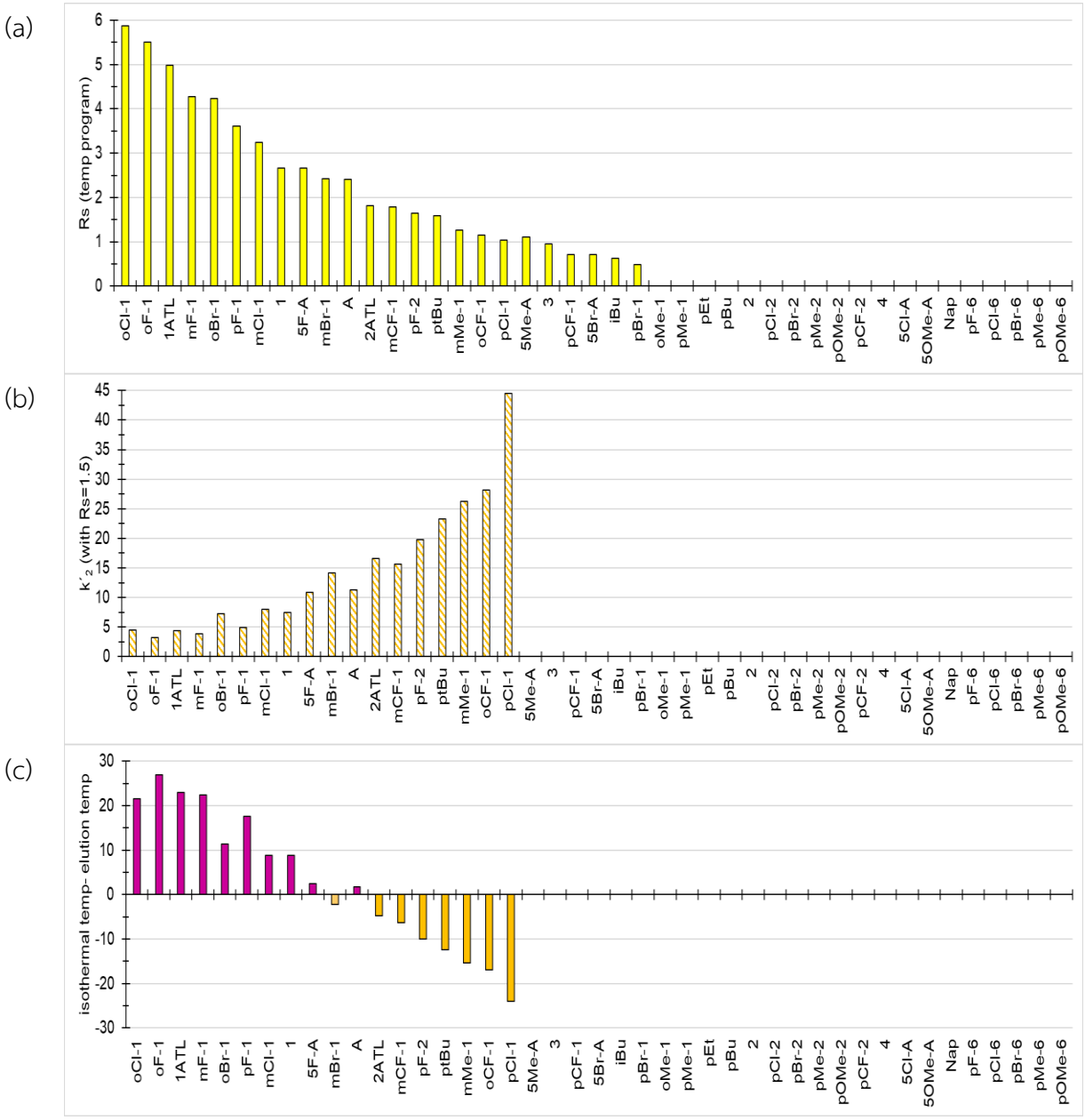


Figure 4.10 Chromatographic results of 43 chiral amines analyzed by BSiMe column.

(a) Rs values obtained from temperature program runs in decreasing order;

(b) k'_2 values at optimum isothermal condition (Rs ~ 1.5);

(c) temperature difference (optimum isothermal temperature – elution temperature).

From **Figures 4.7-4.10**, it can be seen that chiral compounds separated by temperature program with high Rs values could be completely separated by isothermal condition with short analysis time (low k' values). If a Rs < 1.0 was obtained, a complete enantiomeric separation could not be achieved or could be achieved with long retention (k' > 30). If a Rs > 3.0 was obtained, a complete enantiomeric separation could be generally achieved with short analysis time (k' < 10). In addition, the optimum isothermal temperature was higher than the elution temperature obtained from a temperature program run for a compound with high Rs value. Therefore, resolution and elution temperature obtained from a temperature program run could be used as a guideline for quickly determining the optimum isothermal condition.

In this work, alcohol **L32** (or **triF-PE**) analyzed on BSiMe column showed the highest resolution (Rs = 36.31), its optimum isothermal temperature was at 185 °C; 59 °C higher than the elution temperature. A complete enantiomeric separation of alcohol **L32** on BSiMe column could be achieved with the shortest analysis time of 0.783 minute (k'_2=0.563) (**Figure 4.11**).

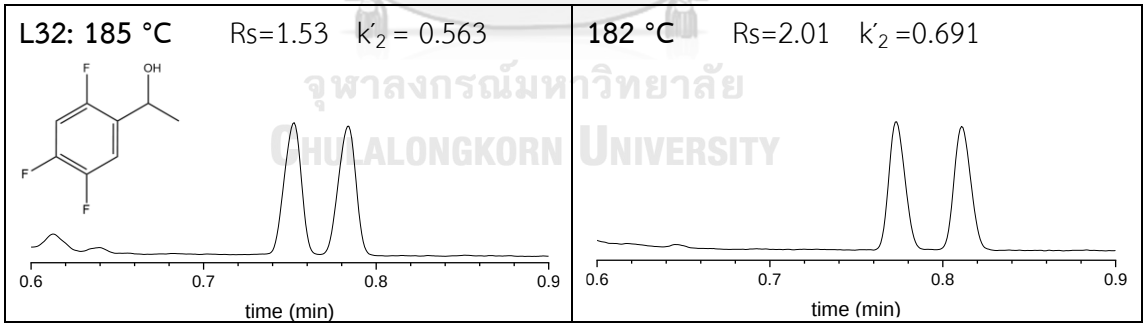


Figure 4.11 Separation of **L32** on BSiMe column using isothermal conditions.

CHAPTER V

CONCLUSION

Enantiomeric separations of seventy-two underivatized chiral alcohols and forty-three trifluoroacetyl derivatives of chiral amines of various structures were investigated by GC. Two β-cyclodextrin derivatives having different types of substitution were used as chiral stationary phases: heptakis(2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or **BSiAc**) and heptakis(2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl)-β-CD (or **BSiMe**). Analyses were performed by temperature program, followed by determination of optimum isothermal condition. The number of compounds that could be completely separated on BSiAc and BSiMe columns were compared in **Table 5.1**. Results from **Table 5.1** suggested that BSiAc column was suitable for the separation of chiral amines (TFA derivatives), while BSiMe column was suitable for the separation of chiral alcohols.

Table 5.1 Enantiomeric separations of chiral alcohols and chiral amines by BSiAc and BSiMe columns.

column	**BSiAc**			**BSiMe**		
	alcohols	amines	total	alcohols	amines	total
analytes	72	43	115	72	43	115
complete separation[(a)]	42	35	77	55	15	70
%	53.8%	81.4%	67.0%	76.4%	34.9%	60.9%
incomplete separation	10	5	15	10	9	19
no separation[(b)]	20	3	23	7	19	26
%	27.8%	7.0%	20.0%	9.7%	44.2%	22.6%

(a) Compounds could be completely separated by temperature program and isothermal conditions.

(b) Compounds showed no separation by temperature program and isothermal conditions.

Based on results in this study, a temperature program run (suggested by Grob test [41], [42]) could be used as a screening condition for the separation of enantiomers. In addition, resolution and elution temperature obtained from a temperature program run could be used as a guideline for quickly determining the optimum isothermal condition. If a resolution was less than 1.0, a complete enantiomeric separation could not be achieved with reasonable retention ($k' \leq 20$); therefore, this stationary phase was not recommended for the separation. If a resolution obtained from a temperature program run was greater than 3.0, a complete enantiomeric separation could be generally achieved with short analysis time ($k' < 10$). An isothermal temperature of at least 10 °C higher than the elution temperature could be used as a starting temperature to quickly reach at its optimum isothermal condition. The higher the resolution obtained, the higher the isothermal temperature could be used and the shorter the retention.

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Appendix A

Table A1 Chromatographic results of 43 chiral amines analyzed by BSiAc column (15.57 m long) using a temperature program starting from 40 °C at a rate of 3.21 °C/min.

no.	abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
A01	1	30.619	30.715	0.0454	0.0576	138.29	138.60	1.10
A02	oF-1	26.438	27.108	0.0487	0.0466	124.87	127.02	8.27
A03	mF-1	33.727	34.168	0.0603	0.0579	148.26	149.68	4.39
A04	pF-1	34.473	34.665	0.0471	0.0437	150.66	151.27	2.49
A05	oCl-1	30.972	31.171	0.0535	0.0534	139.42	140.06	2.19
A06	mCl-1	36.926	37.861	0.0500	0.0523	158.53	161.53	10.76
A07	pCl-1	39.678	39.850	0.0487	0.0499	167.37	167.92	2.05
A08	oBr-1	33.300	33.474	0.0533	0.0563	146.89	147.45	1.87
A09	mBr-1	38.996	39.838	0.0493	0.0469	165.18	167.88	10.30
A10	pBr-1	41.945	42.041	0.0463	0.0452	174.64	174.95	1.23
A11	oMe-1	30.481	30.904	0.0462	0.0487	137.84	139.20	5.25
A12	mMe-1	29.907	30.663	0.0491	0.0481	136.00	138.43	9.15
A13	pMe-1	31.053	31.530	0.0496	0.0482	139.68	141.21	5.74
A14	oCF-1	26.962	27.204	0.0515	0.0526	126.55	127.32	2.74
A15	mCF-1	33.884	35.076	0.0498	0.0432	148.77	152.59	15.09
A16	pCF-1	36.162	36.890	0.0459	0.0444	156.08	158.42	9.49
A17	pEt	32.775	33.347	0.0494	0.0476	145.21	147.04	6.94
A18	pBu	37.195	37.606	0.0521	0.0515	159.40	160.72	4.67
A19	ptBu	35.265	35.901	0.0513	0.0488	153.20	155.24	7.48
A20	2	29.491	30.114	0.0513	0.0469	134.67	136.67	7.47
A21	pF-2	33.158	33.845	0.0481	0.0492	146.44	148.64	8.31
A22	pCl-2	38.641	39.206	0.0527	0.049	164.04	165.85	6.54
A23	pBr-2	41.005	41.454	0.0492	0.0516	171.63	173.07	5.24
A24	pMe-2	34.592	35.562	0.0486	0.0461	151.04	154.15	12.06
A25	pOMe-2	36.056	36.452	0.0537	0.0514	155.74	157.01	4.43
A26	pCF-2	30.630	31.354	0.0534	0.0488	138.32	140.65	8.34

no.	abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
A27	iBu	29.027	29.531	0.0529	0.0507	133.18	134.79	5.73
A28	3	30.251	30.399	0.0525	0.0528	137.11	137.58	1.65
A29	4	35.516	35.777	0.0566	0.0530	154.01	154.84	2.80
A30	A	35.382	36.246	0.0455	0.0444	153.58	156.35	11.31
A31	5F-A	39.180	39.766	0.0461	0.0460	165.77	167.65	7.49
A32	5Cl-A	44.403	44.811	0.0490	0.0447	182.53	183.84	5.13
A33	5Br-A	46.675	47.060	0.0499	0.0477	189.83	191.06	4.64
A34	5Me-A	36.226	37.031	0.0503	0.0485	156.29	158.87	9.59
A35	5OMe-A	41.135	41.546	0.0498	0.0495	172.04	173.36	4.87
A36	1ATL	35.476	36.378	0.0522	0.0505	153.88	156.77	10.34
A37	2ATL	39.353	39.742	0.0502	0.0493	166.32	167.57	4.60
A38	Nap	-	40.642	-	0.0568	-	170.46	0
A39	pF-6	41.133	41.213	0.0579	0.0622	172.04	172.29	*(a)
A40	pCl-6	46.538	46.655	0.0578	0.0592	189.39	189.76	1.18
A41	pBr-6	49.076	49.198	0.0558	0.0598	197.53	197.93	1.24
A42	pMe-6	-	41.495	-	0.0643	-	173.20	0
A43	pOMe-6	-	46.536	-	0.0647	-	189.38	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

Table A2 Chromatographic results of 72 chiral alcohols analyzed by BSiMe column (14.91 m long) using a temperature program starting from 40 °C at a rate of 3.35 °C/min.

No.	Abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
L01	PE	20.514	21.061	0.0616	0.0598	108.72	110.55	5.30
L02	oF-PE	20.998	22.384	0.0610	0.0550	110.34	114.99	14.06
L03	mF-PE	22.281	22.673	0.0543	0.0558	114.64	115.95	4.19
L04	pF-PE	21.679	22.207	0.0571	0.0569	112.62	114.39	5.45
L05	oCl-PE	27.400	29.946	0.0551	0.0467	131.79	140.32	29.44
L06	mCl-PE	28.299	28.615	0.0611	0.0564	134.80	135.86	3.17
L07	pCl-PE	27.884	28.329	0.0571	0.0572	133.41	134.90	4.58
L08	oBr-PE	30.282	33.036	0.0565	0.0479	141.44	150.67	31.05
L09	mBr-PE	31.132	31.339	0.0546	0.0582	144.29	144.99	2.16
L10	pBr-PE	30.979	31.291	0.0595	0.0575	143.78	144.82	3.14
L11	oMe-PE	25.258	26.407	0.0553	0.0524	124.61	128.46	12.56
L12	mMe-PE	23.582	24.177	0.0578	0.0553	119.00	120.99	6.19
L13	pME-PE	22.361	23.124	0.0591	0.0572	114.91	117.47	7.72
L14	oOMe-PE	26.980	28.875	0.0547	0.0503	130.38	136.73	21.24
L15	mOMe-PE	28.269	28.764	0.0584	0.0573	134.70	136.36	5.04
L16	pOMe-PE	21.786	22.079	0.0584	0.0570	112.98	113.96	2.99
L17	oCF-PE	21.341	24.358	0.0553	0.0487	111.49	121.60	34.14
L18	mCF-PE	22.432	22.763	0.0519	0.0534	115.15	116.26	3.70
L19	pCF-PE	22.794	23.481	0.0557	0.0558	116.36	118.66	7.25
L20	oNO-PE	35.306	37.932	0.0545	0.0499	158.28	167.07	29.61
L21	mNO-PE	38.082	38.189	0.0555	0.0609	167.57	167.93	1.08
L22	pNO-PE	39.114	39.415	0.0620	0.0558	171.03	172.04	3.01
L23	pEt-PE	25.470	25.853	0.0618	0.0612	125.32	126.61	3.66
L24	pBu-PE	31.135	31.413	0.0580	0.0560	144.30	145.23	2.87
L25	ptBu-PE	29.738	29.861	0.0558	0.0562	139.62	140.03	1.29
L26	pPh-PE	42.662	42.799	0.0577	0.0582	182.92	183.38	1.39
L27	24F-PE	20.852	23.172	0.0579	0.0513	109.85	117.63	25.01
L28	25F-PE	23.546	24.804	0.0538	0.0496	118.88	123.09	14.32

No.	Abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
L29	34F-PE	22.831	23.542	0.0633	0.0597	116.48	118.87	6.80
L30	26F-PE	17.483	19.377	0.0643	0.0564	98.57	104.91	18.47
L31	35F-PE	-	22.502	-	0.0848	-	115.38	0
L32	triF-PE	21.968	25.584	0.0640	0.0532	113.59	125.71	36.31
L33	tetraF-PE	21.821	22.404	0.0555	0.0531	113.10	115.05	6.32
L34	pentaF-PE	18.344	19.572	0.0553	0.0532	101.45	105.57	13.32
L35	24Cl-PE	32.615	34.941	0.0569	0.0493	149.26	157.05	25.78
L36	25Cl-PE	34.464	36.864	0.0538	0.0526	155.45	163.49	26.55
L37	34Cl-PE	34.325	34.585	0.0589	0.0547	154.99	155.86	2.69
L38	24Me-PE	27.456	28.803	0.0545	0.0517	131.98	136.49	14.93
L39	25Me-PE	27.638	28.968	0.0534	0.0499	132.59	137.04	15.15
L40	34Me-PE	26.895	27.046	0.0560	0.0580	130.10	130.60	1.56
L41	PP	23.601	23.775	0.0546	0.0545	119.06	119.65	1.88
L42	pF-PP	24.804	25.117	0.0608	0.0589	123.09	124.14	3.08
L43	pCl-PP	30.631	30.850	0.0560	0.0556	142.61	143.35	2.31
L44	pBr-PP	33.553	33.680	0.0563	0.0548	152.40	152.83	1.35
L45	pMe-PP	25.293	25.755	0.0559	0.0576	124.73	126.28	4.79
L46	pOMe-PP	30.501	30.726	0.0579	0.0593	142.18	142.93	2.26
L47	pCF3-PP	25.726	26.139	0.0568	0.0555	126.18	127.57	4.33
L48	1OH	20.908	21.229	0.0637	0.0665	110.04	111.12	2.90
L49	2OH	-	25.342	-	0.0542	-	124.90	0
L50	3OH	26.406	26.709	0.0537	0.0536	128.46	129.48	3.32
L51	4OH	25.683	25.988	0.0580	0.0560	126.04	127.06	3.15
L52	5OH	23.553	23.981	0.0609	0.0587	118.90	120.34	4.21
L53	6OH	25.116	25.327	0.0636	0.0632	124.14	124.85	1.96
L54	7OH	25.979	26.051	0.0529	0.0598	127.03	127.27	*(b)
L55	8OH	23.102	23.387	0.0599	0.0578	117.39	118.35	2.85
L56	DPE	39.420	39.501	0.0561	0.0590	172.06	172.33	*(b)
L57	In	25.830	25.896	0.0527	0.0639	126.53	126.75	*(b)
L58	1-Nap	30.109	30.334	0.0570	0.0583	140.87	141.62	2.30
L59	2-Nap	-	30.904	-	0.0825	-	143.53	0

No.	Abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
L60	**1Nap-E**	37.469	38.175	0.0560	0.0560	165.52	167.89	7.42
L61	**2Nap-E**	36.925	37.157	0.0603	0.0602	163.70	164.48	2.27
L62	**pF-Ph**	-	38.626	-	0.0781	-	169.40	0
L63	**pCl-Ph**	43.948	44.057	0.0592	0.0589	187.23	187.59	1.09
L64	**pBr-Ph**	46.537	46.642	0.0598	0.0629	195.90	196.25	1.01
L65	**oMe-Ph**	-	39.380	-	0.0879	-	171.92	0
L66	**mMe-Ph**	39.609	39.830	0.0593	0.0567	172.69	173.43	2.24
L67	**pMe-Ph**	39.826	39.989	0.0560	0.0574	173.42	173.96	1.69
L68	**pOMe-Ph**	44.203	44.335	0.0585	0.0622	188.08	188.52	1.29
L69	**2Ph-Bu**	24.604	24.771	0.0587	0.0585	122.42	122.98	1.68
L70	**pF-CF**	24.989	25.138	0.0513	0.0525	123.71	124.21	1.69
L71	**pCl- CF**	-	30.449	-	0.0576	-	142.00	0
L72	**pBr-CF**	-	33.217	-	0.0253	-	151.28	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

Table A3 Chromatographic results of 43 chiral amines analyzed by BSiMe column (14.91 m long) using a temperature program starting from 40 °C at a rate of 3.35 °C/min.

No.	Abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
A01	1	23.101	23.355	0.0556	0.0567	117.39	118.24	2.66
A02	oF-1	20.984	21.527	0.0579	0.0581	110.30	112.12	5.51
A03	mF-1	24.841	25.256	0.0584	0.0561	123.22	124.61	4.27
A04	pF-1	24.850	25.198	0.0577	0.0559	123.25	124.41	3.61
A05	oCl-1	27.338	27.900	0.0561	0.0565	131.58	133.47	5.87
A06	mCl-1	30.491	30.796	0.0559	0.0548	142.14	143.17	3.24
A07	pCl-1	31.223	31.325	0.0570	0.0589	144.60	144.94	1.04
A08	oBr-1	30.22	30.626	0.0579	0.0550	141.24	142.60	4.23
A09	mBr-1	33.254	33.489	0.0571	0.0574	151.40	152.19	2.42
A10	pBr-1	34.346	34.387	0.0481	0.0509	155.06	155.20	*(a)
A11	oMe-1	-	24.836	-	0.0581	-	123.20	0
A12	mMe-1	25.379	25.499	0.0551	0.0571	125.02	125.42	1.26
A13	pMe-1	-	25.720	-	0.0878	-	126.16	0
A14	oCF-1	23.467	23.578	0.0553	0.0581	118.61	118.99	1.15
A15	mCF-1	25.605	25.766	0.0528	0.0536	125.78	126.32	1.78
A16	pCF-1	27.074	27.135	0.0501	0.0515	130.70	130.90	*(b)
A17	pEt	-	29.029	-	0.0587	-	137.25	0
A18	pBu	-	34.265	-	0.0627	-	154.79	0
A19	ptBu	33.089	33.235	0.0541	0.0541	150.85	151.34	1.59
A20	2	24.975	24.975	0.0600	0.0600	123.67	123.67	0
A21	pF-2	26.717	26.873	0.0556	0.0566	129.50	130.02	1.64
A22	pCl-2	-	32.852	-	0.0602	-	150.05	0
A23	pBr-2	-	35.826	-	0.0596	-	160.02	0
A24	pMe-2	-	28.536	-	0.0696	-	135.60	0
A25	pOMe-2	-	32.773	-	0.0601	-	149.79	0
A26	pCF-2	-	27.552	-	0.0722	-	132.30	0
A27	iBu	25.392	25.450	0.0541	0.0541	125.06	125.26	*(b)
A28	3	27.091	27.182	0.0554	0.0569	130.75	131.06	*(b)

No.	Abbr.	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$ (min)	$W_{h,2}$ (min)	elution $temp_1$ (°C)	elution $temp_2$ (°C)	Rs
A29	4	-	32.771	-	0.0591	-	149.78	0
A30	A	29.672	29.911	0.0587	0.0580	139.40	140.20	2.41
A31	5F-A	31.252	31.511	0.0564	0.0581	144.69	145.56	2.66
A32	5Cl-A	-	37.199	-	0.0603	-	164.62	0
A33	5Br-A	40.041	40.108	0.0529	0.0589	174.14	174.36	*(b)
A34	5Me-A	31.653	31.763	0.0566	0.0611	146.04	146.41	1.10
A35	5OMe-A	-	36.567	-	0.0721	-	162.50	0
A36	1ATL	32.381	32.863	0.0596	0.0542	148.48	150.09	4.99
A37	2ATL	34.089	34.272	0.0594	0.0593	154.20	154.81	1.81
A38	Nap	-	37.922	-	0.0731	-	167.04	0
A39	pF-6	-	37.993	-	0.0587	-	167.28	0
A40	pCl-6	-	43.565	-	0.0633	-	185.94	0
A41	pBr-6	-	46.279	-	0.0728	-	195.03	0
A42	pMe-6	-	39.579	-	0.0755	-	172.59	0
A43	pOMe-6	-	44.195	-	0.0655	-	188.05	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0

Appendix B

Table B1 Chromatographic results of 72 chiral alcohols analyzed by BSiAc column (15.57 m long) using isothermal conditions.

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
L01	PE	130	1.679	1.702	0.0173	0.0187	2.210	2.254	1.020	*(b)
		120	2.348	2.409	0.0265	0.0283	3.481	3.597	1.033	1.31
		110	3.538	3.698	0.0370	0.0394	5.713	6.017	1.053	2.46
		100	5.778	6.207	0.0621	0.0671	9.943	10.756	1.082	3.91
		90	10.19	11.387	0.1044	0.1189	18.207	20.444	1.123	6.26
L02	oF-PE	130	1.501	1.520	0.0149	0.0147	1.870	1.906	1.019	*(b)
		120	2.003	2.041	0.0206	0.0209	2.823	2.895	1.026	1.08
		110	2.851	2.926	0.0281	0.0309	4.410	4.552	1.032	1.50
		100	4.350	4.503	0.0440	0.0480	7.239	7.528	1.040	1.96
		90	7.129	7.450	0.0764	0.0797	12.426	13.030	1.049	2.42
L03	mF-PE	130	2.182	2.212	0.0207	0.0224	3.172	3.229	1.018	*(b)
		120	3.279	3.370	0.0356	0.0375	5.258	5.431	1.033	1.47
		110	5.377	5.616	0.0545	0.0714	9.203	9.657	1.049	2.23
		100	9.550	10.274	0.0956	0.1368	17.087	18.458	1.080	3.67
		90	18.47	20.592	0.1517	0.1699	33.800	37.780	1.118	7.73
L04	pF-PE	130	2.210	2.241	0.0210	0.0227	3.226	3.285	1.018	*(b)
		120	3.333	3.414	0.0338	0.0368	5.361	5.515	1.029	1.35
		110	5.453	5.666	0.0562	0.0626	9.347	9.751	1.043	2.11
		100	9.668	10.241	0.0954	0.1039	17.311	18.396	1.063	3.38
		90	18.47	20.085	0.1519	0.1634	33.787	36.825	1.090	6.02
L05	oCl-PE	140	2.113	2.141	0.0211	0.0224	3.040	3.094	1.018	*(b)
		130	2.880	2.936	0.0296	0.0318	4.507	4.614	1.024	1.07
		120	4.131	4.244	0.0430	0.0450	6.884	7.099	1.031	1.51
		110	6.243	6.475	0.0656	0.0662	10.846	11.287	1.041	2.07
		100	9.921	10.420	0.1169	0.1060	17.790	18.735	1.053	2.63
L06	mCl-PE	150	2.354	2.380	0.0222	0.0239	3.510	3.559	1.014	*(b)
		140	3.410	3.485	0.0332	0.0358	5.520	5.663	1.026	1.28

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	**α**	Rs
		130	5.320	5.522	0.0540	0.0566	9.172	9.558	1.042	2.15
		120	8.932	9.499	0.0926	0.0908	16.046	17.128	1.067	3.64
		110	16.17	17.814	0.1356	0.1487	29.689	32.803	1.105	6.79
L07	pCl-PE	150	2.630	2.659	0.0229	0.0260	4.038	4.094	1.014	*(b)
		140	3.936	4.013	0.0380	0.0409	6.526	6.673	1.023	1.15
		130	6.348	6.547	0.0642	0.0680	11.138	11.518	1.034	1.77
		120	10.97	11.502	0.1115	0.0998	19.941	20.950	1.051	2.95
		110	20.40	21.842	0.1692	0.1739	37.710	40.446	1.073	4.95
L08	oBr-PE	150	2.236	2.255	0.0191	0.0210	3.284	3.320	1.011	*(a)
		140	3.036	3.080	0.0289	0.0298	4.805	4.889	1.018	*(b)
		130	4.321	4.408	0.0427	0.0445	7.262	7.428	1.023	1.17
		120	6.439	6.615	0.0654	0.0731	11.288	11.624	1.030	1.50
		110	10.07	10.437	0.1063	0.0968	18.108	18.805	1.038	2.13
L09	mBr-PE	150	3.453	3.509	0.0327	0.0343	5.615	5.722	1.019	*(b)
		140	5.240	5.388	0.0502	0.0548	9.019	9.302	1.031	1.66
		130	8.532	8.941	0.0844	0.0844	15.314	16.096	1.051	2.85
		120	14.86	16.036	0.1227	0.1299	27.368	29.603	1.082	5.46
		110	27.70	31.143	0.2230	0.2591	51.564	58.095	1.127	8.40
L10	pBr-PE	150	4.055	4.119	0.0377	0.0415	6.768	6.891	1.018	*(b)
		140	6.397	6.560	0.0606	0.0675	11.231	11.543	1.028	1.50
		130	10.80	11.233	0.1036	0.1066	19.660	20.478	1.042	2.40
		120	19.47	20.609	0.1545	0.1647	36.156	38.330	1.060	4.20
		110	37.42	40.630	0.3028	0.3349	70.023	76.097	1.087	5.91
L11	oMe-PE	130	-	2.148	-	0.0235	-	3.107	1.000	0
		120	-	2.996	-	0.0393	-	4.707	1.000	0
		110	-	4.406	-	0.0668	-	7.376	1.000	0
		100	-	6.888	-	0.1211	-	12.095	1.000	0
		90	11.35	11.529	0.1088	0.1164	20.375	20.712	1.017	*(b)
L12	mMe-PE	140	1.640	1.661	0.0169	0.0176	2.136	2.176	1.019	*(b)
		130	2.216	2.270	0.0227	0.0239	3.237	3.340	1.032	1.36

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	**α**	Rs
		120	3.213	3.352	0.0335	0.0394	5.132	5.397	1.052	2.24
		110	5.020	5.394	0.0532	0.0600	8.526	9.235	1.083	3.89
		100	8.451	9.494	0.0924	0.0913	15.006	16.981	1.132	6.68
L13	pMe-PE	140	1.806	1.848	0.0171	0.0188	2.453	2.533	1.033	1.38
		130	2.535	2.636	0.0247	0.0259	3.847	4.040	1.050	2.35
		120	3.828	4.073	0.0378	0.0404	6.305	6.773	1.074	3.69
		110	6.264	6.895	0.0651	0.0713	10.909	12.108	1.110	5.44
		100	11.05	12.742	0.1084	0.1216	19.945	23.133	1.160	8.61
L14	oOMe-PE	140	-	2.404	-	0.0306	-	3.614	1.000	0
		130	-	3.352	-	0.0417	-	5.409	1.000	0
		120	-	4.923	-	0.0675	-	8.377	1.000	0
		110	-	7.597	-	0.1012	-	13.470	1.000	0
		100	-	12.384	-	0.1962	-	22.544	1.000	0
L15	mOMe-PE	150	2.587	2.622	0.0243	0.0257	3.956	4.023	1.017	*(b)
		140	3.794	3.885	0.0366	0.0385	6.254	6.428	1.028	1.43
		130	5.966	6.206	0.0604	0.0629	10.407	10.866	1.044	2.29
		120	10.08	10.740	0.1013	0.1041	18.254	19.513	1.069	3.78
		110	18.39	20.302	0.1528	0.1702	33.966	37.597	1.107	6.96
L16	pOMe-PE	140	1.946	1.965	0.0172	0.0174	2.721	2.757	1.013	*(b)
		130	2.626	2.669	0.0245	0.0247	4.021	4.103	1.020	1.03
		120	3.734	3.824	0.0358	0.0374	6.126	6.298	1.028	1.45
		110	5.600	5.793	0.0541	0.0575	9.646	10.013	1.038	2.04
		100	8.818	9.245	0.0854	0.0887	15.701	16.509	1.052	2.89
L17	oCF-PE	130	1.382	1.400	0.0141	0.0148	1.642	1.677	1.021	*(b)
		120	1.784	1.820	0.0183	0.0182	2.405	2.473	1.029	1.16
		110	2.437	2.510	0.0244	0.0255	3.633	3.772	1.038	1.72
		100	3.512	3.665	0.0363	0.0381	5.652	5.941	1.051	2.42
		90	5.378	5.709	0.0563	0.0608	9.128	9.751	1.068	3.33
L18	mCF-PE	140	1.559	1.575	0.0154	0.0158	1.981	2.011	1.015	*(b)
		130	2.147	2.190	0.0219	0.0230	3.105	3.187	1.026	1.13

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	**α**	Rs
		120	3.188	3.300	0.0319	0.0338	5.084	5.298	1.042	2.01
		110	5.139	5.446	0.0528	0.0571	8.770	9.354	1.067	3.29
		100	8.958	9.836	0.0934	0.0985	15.966	17.629	1.104	5.39
L19	pCF-PE	140	-	1.936	-	0.0362	-	2.716	1.000	0
		130	2.858	2.906	0.0281	0.0309	4.465	4.556	1.021	*(b)
		120	4.572	4.704	0.0469	0.0487	7.725	7.977	1.033	1.63
		110	7.932	8.301	0.0796	0.0840	14.080	14.781	1.050	2.65
		100	14.76	15.811	0.1286	0.1311	26.968	28.945	1.073	4.73
L20	oNO-PE	170	2.502	2.535	0.0242	0.0246	3.793	3.856	1.017	*(b)
		160	3.446	3.513	0.0333	0.0342	5.602	5.730	1.023	1.17
		150	4.981	5.120	0.0492	0.0516	8.542	8.808	1.031	1.62
		140	7.528	7.824	0.0749	0.0796	13.394	13.960	1.042	2.25
		130	11.98	12.650	0.1082	0.1195	21.912	23.187	1.058	3.45
L21	mNO-PE	190	-	2.630	-	0.0280	-	4.048	1.000	0
		180	-	3.708	-	0.0417	-	6.117	1.000	0
		170	-	5.533	-	0.0626	-	9.600	1.000	0
		160	-	8.833	-	0.1041	-	15.921	1.000	0
		150	-	14.865	-	0.1434	-	27.532	1.000	0
L22	pNO-PE	200	-	2.593	-	0.0283	-	3.977	1.000	0
		190	-	3.765	-	0.0406	-	6.226	1.000	0
		180	-	5.763	-	0.0689	-	10.061	1.000	0
		170	-	9.495	-	0.1187	-	17.190	1.000	0
		160	-	16.830	-	0.1998	-	31.241	1.000	0
L23	pEt-PE	140	2.487	2.536	0.0248	0.0256	3.755	3.849	1.025	1.14
		130	3.640	3.761	0.0369	0.0404	5.960	6.191	1.039	1.84
		120	5.731	6.038	0.0574	0.0662	9.937	10.523	1.059	2.92
		110	9.696	10.503	0.0984	0.1060	17.433	18.968	1.088	4.65
		100	17.59	19.816	0.1483	0.1651	32.330	36.530	1.130	8.33
L24	pBU-PE	150	3.628	3.669	0.0319	0.0332	5.950	6.029	1.013	*(b)
		140	5.476	5.582	0.0536	0.0560	9.470	9.673	1.021	1.14

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	**α**	Rs
		130	8.791	9.066	0.0864	0.0933	15.809	16.335	1.033	1.80
		120	15.07	15.816	0.1373	0.1298	27.773	29.183	1.051	3.26
		110	27.63	29.720	0.2270	0.2367	51.540	55.502	1.077	5.29
L25	ptBU-PE	150	-	2.604	-	0.0317	-	3.989	1.000	0
		140	-	3.738	-	0.0575	-	6.175	1.000	0
		130	5.715	5.770	0.0486	0.0568	9.927	10.033	1.011	*(b)
		120	9.233	9.394	0.0941	0.0879	16.620	16.927	1.018	1.04
		110	15.88	16.335	0.1373	0.1377	29.207	30.055	1.029	1.91
L26	pPh-PE	190	-	4.371	-	0.0559	-	7.390	1.000	0
		180	-	6.431	-	0.1053	-	11.344	1.000	0
		170	9.982	10.105	0.0862	0.0920	18.123	18.358	1.013	*(b)
		160	16.18	16.524	0.1329	0.1394	30.010	30.655	1.022	1.46
		150	27.73	28.670	0.2161	0.2196	52.134	53.923	1.034	2.52
L27	24F-PE	130	1.574	1.601	0.0159	0.0173	2.004	2.055	1.026	*(b)
		120	2.182	2.218	0.0209	0.0224	3.156	3.225	1.022	*(b)
		110	3.243	3.316	0.0322	0.0349	5.165	5.304	1.027	1.28
		100	5.164	5.319	0.0530	0.0579	8.780	9.074	1.033	1.65
		90	8.894	9.235	0.0881	0.0949	15.750	16.392	1.041	2.19
L28	25F-PE	130	1.758	1.782	0.0168	0.0178	2.355	2.401	1.019	*(b)
		120	2.461	2.509	0.0243	0.0259	3.688	3.779	1.025	1.13
		110	3.714	3.813	0.0386	0.0406	6.061	6.249	1.031	1.47
		100	6.004	6.210	0.0622	0.0667	10.371	10.761	1.038	1.88
		90	10.46	10.910	0.1079	0.1114	18.701	19.546	1.045	2.41
L29	34F-PE	140	-	2.148	-	0.0317	-	3.123	1.000	0
		130	3.284	3.323	0.0295	0.0364	5.267	5.342	1.014	*(b)
		120	5.441	5.555	0.0545	0.0614	9.364	9.581	1.023	1.16
		110	9.818	10.137	0.0981	0.1003	17.665	18.272	1.034	1.89
		100	18.93	19.858	0.1670	0.1740	34.869	36.610	1.050	3.17
L30	26F-PE	110	-	2.099	-	0.0234	-	2.990	1.000	0
		100	-	2.985	-	0.0349	-	4.653	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		90	-	4.526	-	0.0612	-	7.572	1.000	0
		80	-	7.304	-	0.1118	-	12.755	1.000	0
		70	12.56	12.697	0.1038	0.1182	22.486	22.733	1.011	*(b)
L31	35F-PE	130	2.245	2.295	0.0226	0.0249	3.284	3.380	1.029	1.24
		120	3.431	3.570	0.0351	0.0370	5.535	5.800	1.048	2.27
		110	5.700	6.094	0.0592	0.0648	9.837	10.586	1.076	3.74
		100	10.28	11.430	0.0949	0.0964	18.487	20.648	1.117	7.02
		90	20.05	23.425	0.1618	0.1924	36.770	43.115	1.173	11.2
L32	triF-PE	130	2.100	2.133	0.0232	0.0225	3.008	3.071	1.021	*(b)
		120	3.143	3.219	0.0322	0.0374	4.987	5.131	1.029	1.29
		110	5.113	5.292	0.0534	0.0563	8.721	9.061	1.039	1.92
		100	8.958	9.389	0.0859	0.0841	15.966	16.782	1.051	2.98
		90	16.92	18.012	0.1430	0.1477	30.872	32.921	1.066	4.41
L33	tetraF-PE	130	2.216	2.246	0.0213	0.0224	3.229	3.286	1.018	*(b)
		120	3.310	3.363	0.0329	0.0345	5.305	5.406	1.019	*(b)
		110	5.340	5.436	0.0525	0.0567	9.152	9.335	1.020	1.03
		100	9.263	9.429	0.0863	0.0945	16.544	16.858	1.019	1.08
		90	17.25	17.521	0.1256	0.1490	31.499	31.996	1.016	1.13
L34	pentaF-PE	130	1.594	1.610	0.0160	0.0169	2.042	2.073	1.015	*(b)
		120	2.248	2.292	0.0228	0.0254	3.282	3.366	1.026	1.07
		110	3.439	3.546	0.0362	0.0374	5.538	5.741	1.037	1.71
		100	5.690	5.960	0.0589	0.0638	9.777	10.288	1.052	2.59
		90	10.16	10.853	0.1050	0.1080	18.137	19.439	1.072	3.82
L35	24Cl-PE	160	-	2.452	-	0.0294	-	3.697	1.000	0
		150	-	3.398	-	0.0441	-	5.510	1.000	0
		140	-	4.940	-	0.0735	-	8.482	1.000	0
		130	-	7.555	-	0.1159	-	13.446	1.000	0
		120	-	12.171	-	0.1834	-	22.183	1.000	0
L36	25Cl-PE	160	-	2.574	-	0.0290	-	3.931	1.000	0
		150	-	3.587	-	0.0461	-	5.885	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	**α**	Rs
		140	-	3.252	-	0.0692	-	5.242	1.000	0
		130	-	8.093	-	0.1204	-	14.474	1.000	0
		120	-	13.154	-	0.1964	-	24.055	1.000	0
L37	34Cl-PE	170	3.063	3.090	0.0272	0.0290	4.868	4.920	1.011	*(b)
		160	4.511	4.590	0.0426	0.0472	7.642	7.793	1.020	1.04
		150	7.070	7.278	0.0700	0.0719	12.544	12.943	1.032	1.73
		140	11.83	12.389	0.1116	0.1150	21.621	22.688	1.049	2.90
		130	21.17	22.732	0.1624	0.1821	39.408	42.382	1.075	5.32
L38	24Me-PE	140	-	2.249	-	0.0266	-	3.317	1.000	0
		130	-	3.122	-	0.0418	-	4.969	1.000	0
		120	-	4.569	-	0.0677	-	7.703	1.000	0
		110	-	7.038	-	0.1127	-	12.380	1.000	0
		100	-	11.463	-	0.1658	-	20.793	1.000	0
L39	25Me-PE	140	-	2.150	-	0.0258	-	3.127	1.000	0
		130	-	2.948	-	0.0402	-	4.637	1.000	0
		120	-	4.253	-	0.0675	-	7.101	1.000	0
		110	-	6.456	-	0.1090	-	11.274	1.000	0
		100	10.30	10.426	0.0809	0.0994	18.523	18.746	1.012	*(b)
L40	34Me-PE	150	1.951	1.981	0.0192	0.0198	2.738	2.795	1.021	*(b)
		140	2.703	2.776	0.0258	0.0281	4.168	4.308	1.033	1.59
		130	3.996	4.182	0.0410	0.0424	6.626	6.981	1.054	2.62
		120	6.347	6.839	0.0651	0.0709	11.090	12.027	1.085	4.26
		110	10.85	12.214	0.0979	0.1076	19.644	22.221	1.131	7.76
L41	PP	130	-	2.095	-	0.0399	-	3.006	1.000	0
		120	2.991	3.042	0.0295	0.0325	4.697	4.794	1.021	*(b)
		110	4.571	4.711	0.0464	0.0498	7.690	7.956	1.035	1.71
		100	7.479	7.871	0.0774	0.0854	13.165	13.907	1.056	2.83
		90	13.16	14.288	0.1164	0.1299	23.800	25.908	1.089	5.35
L42	pF-PP	140	1.958	1.982	0.0177	0.0200	2.744	2.790	1.017	*(b)
		130	2.795	2.854	0.0273	0.0299	4.334	4.447	1.026	1.21

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	**α**	Rs
		120	4.300	4.445	0.0422	0.0477	7.190	7.467	1.038	1.90
		110	7.100	7.467	0.0729	0.0802	12.498	13.196	1.056	2.82
		100	12.69	13.662	0.1167	0.1220	23.051	24.875	1.079	4.75
L43	pCl-PP	160	-	2.296	-	0.0356	-	3.398	1.000	0
		150	3.292	3.332	0.0297	0.0326	5.307	5.383	1.014	*(b)
		140	4.994	5.094	0.0486	0.0526	8.549	8.740	1.022	1.16
		130	8.116	8.370	0.0806	0.0857	14.489	14.973	1.033	1.80
		120	13.99	14.649	0.1267	0.1403	25.655	26.903	1.049	2.89
L44	pBr-PP	160	-	3.409	-	0.0608	-	5.531	1.000	0
		150	5.109	5.183	0.0459	0.0554	8.787	8.929	1.016	*(b)
		140	8.132	8.316	0.0775	0.0761	14.549	14.901	1.024	1.41
		130	13.76	14.239	0.1220	0.1187	25.269	26.174	1.036	2.32
		120	24.81	26.141	0.1915	0.2039	46.259	48.792	1.055	3.96
L45	pMe-PP	140	2.208	2.239	0.0207	0.0228	3.222	3.281	1.018	*(b)
		130	3.129	3.205	0.0311	0.0338	4.971	5.116	1.029	1.38
		120	4.732	4.926	0.0473	0.0503	8.013	8.383	1.046	2.34
		110	7.676	8.188	0.0845	0.0837	13.593	14.567	1.072	3.58
		100	13.32	14.715	0.1147	0.1368	24.239	26.869	1.109	6.50
L46	pOMe-PP	150	3.475	3.512	0.0308	0.0328	5.657	5.728	1.013	*(b)
		140	5.180	5.280	0.0498	0.0521	8.904	9.096	1.021	1.16
		130	8.202	8.466	0.0834	0.0852	14.653	15.156	1.034	1.84
		120	13.81	14.538	0.1213	0.1230	25.318	26.691	1.054	3.47
		110	24.75	26.767	0.1968	0.2158	45.970	49.791	1.083	5.75
L47	pCF3-PP	140	2.410	2.440	0.0217	0.0247	3.608	3.665	1.016	*(b)
		130	3.644	3.722	0.0357	0.0379	5.954	6.103	1.025	1.25
		120	5.937	6.139	0.0589	0.0640	10.309	10.693	1.037	1.93
		110	10.45	10.995	0.1065	0.1071	18.884	19.903	1.054	2.95
		100	19.80	21.259	0.1596	0.1709	36.511	39.263	1.075	5.17
L48	1OH	130	-	2.011	-	0.0344	-	2.845	1.000	0
		120	2.866	2.912	0.0289	0.0318	4.459	4.547	1.020	*(b)

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		110	4.391	4.496	0.0456	0.0503	7.332	7.531	1.027	1.29
		100	7.269	7.513	0.0798	0.0799	12.767	13.229	1.036	1.80
		90	13.01	13.604	0.1134	0.1101	23.501	24.620	1.048	3.13
L49	2OH	130	-	2.297	-	0.0243	-	3.392	1.000	0
		120	-	3.240	-	0.0398	-	5.171	1.000	0
		110	-	4.851	-	0.0609	-	8.222	1.000	0
		100	-	7.726	-	0.1021	-	13.688	1.000	0
		90	-	13.089	-	0.1389	-	23.790	1.000	0
L50	3OH	130	2.692	2.692	0.0305	0.0305	4.147	4.147	1.000	0
		120	3.825	3.825	0.0502	0.0502	6.286	6.286	1.000	0
		110	5.738	5.738	0.1009	0.1009	9.909	9.909	1.000	0
		100	9.068	9.187	0.0842	0.0942	16.174	16.400	1.014	*(b)
		90	15.08	15.400	0.1277	0.1317	27.403	28.002	1.022	1.44
L51	4OH	140	-	2.101	-	0.0255	-	3.033	1.000	0
		130	-	2.931	-	0.0367	-	4.604	1.000	0
		120	-	4.344	-	0.0651	-	7.274	1.000	0
		110	-	6.886	-	0.1127	-	12.091	1.000	0
		100	-	11.637	-	0.1732	-	21.082	1.000	0
L52	5OH	140	-	2.018	-	0.0208	-	2.873	1.000	0
		130	-	2.823	-	0.0318	-	4.398	1.000	0
		120	-	4.203	-	0.0507	-	7.006	1.000	0
		110	-	6.701	-	0.0785	-	11.740	1.000	0
		100	-	11.504	-	0.1259	-	20.829	1.000	0
L53	6OH	140	-	2.574	-	0.0346	-	3.940	1.000	0
		130	-	3.800	-	0.0674	-	6.266	1.000	0
		120	6.030	6.101	0.0556	0.0632	10.486	10.621	1.013	*(b)
		110	10.20	10.387	0.0946	0.1045	18.366	18.710	1.019	1.07
		100	18.55	19.024	0.1421	0.1541	34.146	35.030	1.026	1.86
L54	7OH	140	-	2.408	-	0.0361	-	3.622	1.000	0
		130	-	3.388	-	0.0580	-	5.478	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		120	-	5.049	-	0.1028	-	8.617	1.000	0
		110	-	7.992	-	0.1487	-	14.194	1.000	0
		100	-	13.501	-	0.2495	-	24.619	1.000	0
L55	8OH	140	1.684	1.720	0.0164	0.0176	2.220	2.289	1.031	1.25
		130	2.251	2.328	0.0223	0.0251	3.296	3.443	1.045	1.91
		120	3.195	3.361	0.0338	0.0370	5.086	5.402	1.062	2.76
		110	4.837	5.210	0.0537	0.0570	8.178	8.886	1.087	3.97
		100	7.833	8.692	0.0851	0.0939	13.835	15.462	1.118	5.65
L56	DPE	190	-	2.768	-	0.0310	-	4.313	1.000	0
		180	-	3.796	-	0.0435	-	6.286	1.000	0
		170	-	5.532	-	0.0662	-	9.598	1.000	0
		160	-	8.147	-	0.0926	-	14.607	1.000	0
		150	-	12.816	-	0.1297	-	23.599	1.000	0
L57	In	140	-	2.344	-	0.0234	-	3.482	1.014	0
		130	-	3.307	-	0.0336	-	5.311	1.015	0
		120	-	4.945	-	0.0511	-	8.419	1.014	0
		110	-	7.858	-	0.0868	-	13.911	1.013	0
		100	-	13.152	-	0.1679	-	23.956	1.000	0
L58	1-Nap	150	-	2.513	-	0.0390	-	3.823	1.000	0
		140	-	3.480	-	0.0576	-	5.679	1.000	0
		130	-	5.061	-	0.0782	-	8.677	1.000	0
		120	-	7.749	-	0.1207	-	13.760	1.000	0
		110	-	12.487	-	0.1690	-	22.740	1.000	0
L59	2-Nap	150	-	3.044	-	0.0374	-	4.843	1.000	0
		140	-	4.337	-	0.0666	-	7.324	1.000	0
		130	-	6.514	-	0.1034	-	11.455	1.000	0
		120	10.32	10.440	0.0838	0.1116	18.661	18.886	1.012	*(b)
		110	17.09	17.377	0.1303	0.1474	31.438	31.973	1.017	1.20
L60	1Nap-E	180	-	2.802	-	0.0312	-	4.378	1.000	0
		170	-	3.829	-	0.0444	-	6.335	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		160	-	5.459	-	0.0667	-	9.458	1.000	0
		150	-	8.106	-	0.1030	-	14.559	1.000	0
		140	-	12.544	-	0.1409	-	23.077	1.000	0
L61	2Nap-E	170	4.652	4.701	0.0399	0.0428	7.912	8.006	1.012	*(b)
		160	7.028	7.163	0.0692	0.0703	12.464	12.722	1.021	1.14
		150	11.23	11.593	0.1086	0.1050	20.474	21.166	1.034	1.99
		140	19.04	20.029	0.1478	0.1557	35.407	37.296	1.053	3.83
		130	34.35	37.159	0.2679	0.2889	64.571	69.914	1.083	5.92
L62	pF-Ph	180	-	3.310	-	0.0369	-	5.329	1.000	0
		170	-	4.690	-	0.0530	-	7.985	1.000	0
		160	-	6.943	-	0.0801	-	12.301	1.000	0
		150	-	10.776	-	0.1292	-	19.683	1.000	0
		140	-	17.594	-	0.1470	-	32.770	1.000	0
L63	pCl-Ph	210	-	2.646	-	0.0291	-	4.079	1.000	0
		200	-	3.570	-	0.0395	-	5.852	1.000	0
		190	-	4.996	-	0.0560	-	8.589	1.000	0
		180	-	7.280	-	0.0837	-	12.920	1.000	0
		170	-	11.072	-	0.1249	-	20.211	1.000	0
L64	pBr-Ph	220	-	2.709	-	0.0314	-	4.210	1.000	0
		210	-	3.633	-	0.0417	-	5.973	1.000	0
		200	-	5.056	-	0.0587	-	8.704	1.000	0
		190	-	7.297	-	0.0876	-	13.006	1.000	0
		180	-	10.957	-	0.1200	-	20.031	1.000	0
L65	oMe-Ph	190	-	2.875	-	0.0316	-	4.518	1.000	0
		180	-	3.926	-	0.0425	-	6.536	1.000	0
		170	-	5.562	-	0.0612	-	9.655	1.000	0
		160	-	8.192	-	0.0916	-	14.693	1.000	0
		150	-	12.543	-	0.1236	-	23.075	1.000	0
L66	mMe-Ph	190	-	2.849	-	0.0300	-	4.468	1.000	0
		180	-	3.907	-	0.0414	-	6.499	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		170	-	5.559	-	0.0602	-	9.649	1.000	0
		160	-	8.233	-	0.0927	-	14.772	1.000	0
		150	-	12.701	-	0.1324	-	23.378	1.000	0
L67	pMe-Ph	190	-	3.019	-	0.0310	-	4.795	1.000	0
		180	-	4.161	-	0.0430	-	6.987	1.000	0
		170	-	5.971	-	0.0617	-	10.439	1.000	0
		160	-	8.892	-	0.0937	-	16.034	1.000	0
		150	-	13.785	-	0.1344	-	25.459	1.000	0
L68	oOMe-Ph	210	-	2.994	-	0.0322	-	4.747	1.000	0
		200	-	4.117	-	0.0451	-	6.902	1.000	0
		190	-	5.806	-	0.0643	-	10.144	1.000	0
		180	-	8.539	-	0.0972	-	15.327	1.000	0
		170	-	13.042	-	0.1265	-	23.985	1.000	0
L69	2Ph-Bu	130	2.839	2.868	0.0263	0.0329	4.418	4.473	1.013	*(b)
		120	4.073	4.148	0.0431	0.0515	6.758	6.901	1.021	*(b)
		110	6.182	6.364	0.0737	0.0807	10.731	11.076	1.032	1.39
		100	9.964	10.399	0.1078	0.1036	17.871	18.695	1.046	2.42
		90	17.02	18.153	0.1657	0.1697	31.070	33.186	1.068	3.94
L70	pF-CF	140	-	2.121	-	0.0258	-	3.071	1.000	0
		130	3.254	3.283	0.0286	0.0315	5.210	5.265	1.011	*(b)
		120	5.394	5.505	0.0539	0.0564	9.255	9.466	1.023	1.18
		110	9.689	10.030	0.0932	0.0949	17.385	18.032	1.037	2.13
		100	18.78	19.809	0.1468	0.1566	34.580	36.517	1.056	3.97
L71	pCl-CF	160	-	2.249	-	0.0262	-	3.308	1.000	0
		150	-	3.312	-	0.0487	-	5.357	1.000	0
		140	5.308	5.364	0.0444	0.0584	9.169	9.276	1.012	*(b)
		130	9.069	9.239	0.0835	0.0983	16.307	16.632	1.020	1.10
		120	16.66	17.126	0.1280	0.1370	30.683	31.559	1.029	2.05
L72	pBr-CF	160	-	3.335	-	0.0426	-	5.389	1.000	0
		150	-	5.195	-	0.0762	-	8.971	1.000	0
		140	8.695	8.818	0.0761	0.0884	15.625	15.860	1.015	*(b)
		130	15.39	15.721	0.1209	0.1373	28.385	29.002	1.022	1.47
		120	28.91	29.840	0.2357	0.2697	53.977	55.730	1.032	2.15

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

Table B2 Chromatographic results of 43 chiral amines analyzed by BSiAc column (15.57 m long) using isothermal conditions.

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
A01	1	160	-	2.002	-	0.0284	-	2.835	1.000	0
		150	-	3.008	-	0.0438	-	4.762	1.000	0
		140	5.015	5.080	0.0456	0.0522	8.589	8.713	1.014	*(b)
		130	9.152	9.312	0.0849	0.0974	16.466	16.771	1.019	1.03
		120	18.382	18.769	0.1437	0.1556	34.013	34.750	1.022	1.52
A02	oF-1	160	1.439	1.459	0.0133	0.0147	1.762	1.800	1.022	*(b)
		150	1.928	1.978	0.0188	0.0194	2.693	2.789	1.036	1.54
		140	2.787	2.912	0.0274	0.0283	4.329	4.568	1.055	2.64
		130	4.366	4.681	0.0435	0.0471	7.332	7.933	1.082	4.09
		120	7.456	8.295	0.0757	0.0818	13.202	14.800	1.121	6.27
A03	mF-1	180	1.405	1.436	0.0141	0.0146	1.686	1.746	1.035	1.27
		170	1.950	2.017	0.0189	0.0196	2.743	2.871	1.047	2.05
		160	2.978	3.134	0.0289	0.0311	4.716	5.015	1.063	3.06
		150	5.007	5.379	0.0486	0.0535	8.592	9.305	1.083	4.29
		140	9.313	10.228	0.0917	0.0982	16.807	18.556	1.104	5.67
A04	pF-1	170	2.152	2.175	0.0190	0.0212	3.131	3.175	1.014	*(b)
		160	3.369	3.433	0.0324	0.0360	5.466	5.589	1.022	1.10
		150	5.791	5.957	0.0556	0.0603	10.094	10.412	1.032	1.69
		140	10.920	11.368	0.0995	0.1042	19.880	20.736	1.043	2.59
		130	21.941	23.191	0.2334	0.2462	40.872	43.258	1.058	3.07
A05	oCl-1	160	-	2.699	-	0.0274	-	4.170	1.000	0
		150	-	3.987	-	0.0541	-	6.638	1.000	0
		140	6.213	6.294	0.0583	0.0591	10.880	11.034	1.014	*(b)
		130	10.389	10.648	0.0971	0.1005	18.826	19.321	1.026	1.54
		120	18.640	19.453	0.1420	0.1468	34.505	36.053	1.045	3.31
A06	mCl-1	200	1.317	1.345	0.0141	0.0133	1.538	1.592	1.035	1.20
		190	1.702	1.763	0.0160	0.0167	2.273	2.390	1.052	2.20
		180	2.364	2.503	0.0230	0.0238	3.520	3.786	1.076	3.50
		170	3.517	3.841	0.0342	0.0380	5.750	6.372	1.108	5.28
		160	5.675	6.452	0.0555	0.0631	9.893	11.384	1.151	7.71
A07	pCl-1	190	-	2.131	-	0.0322	-	3.098	1.000	0
		180	3.181	3.227	0.0292	0.0347	5.082	5.170	1.017	*(b)
		170	5.194	5.309	0.0478	0.0528	8.969	9.190	1.025	1.35

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		160	9.228	9.514	0.0837	0.0887	16.712	17.261	1.033	1.95
		150	17.750	18.498	0.1360	0.1411	33.004	34.437	1.043	3.18
A08	oBr-1	170	-	2.694	-	0.0266	-	4.171	1.000	0
		160	-	3.865	-	0.0415	-	6.404	1.000	0
		150	-	5.896	-	0.0921	-	10.295	1.000	0
		140	9.437	9.583	0.0889	0.0898	17.044	17.323	1.016	*(b)
		130	16.143	16.612	0.1270	0.1340	29.807	30.702	1.030	2.11
A09	mBr-1	200	1.677	1.719	0.0178	0.1800	2.231	2.312	1.036	*(a)
		190	2.263	2.354	0.0221	0.0230	3.352	3.527	1.052	2.37
		180	3.273	3.482	0.0313	0.0330	5.258	5.658	1.076	3.83
		170	5.051	5.542	0.0477	0.0535	8.695	9.637	1.108	5.71
		160	14.885	15.196	0.1183	0.1259	27.570	28.167	1.022	1.50
A10	pBr-1	200	-	2.075	-	0.0220	-	2.998	1.000	0
		190	-	2.998	-	0.0386	-	4.765	1.000	0
		180	-	4.729	-	0.0677	-	8.059	1.000	0
		170	8.061	8.168	0.0719	0.0741	14.472	14.678	1.014	*(b)
		160	14.885	15.196	0.1183	0.1259	27.570	28.167	1.022	1.50
A11	oMe-1	180	1.204	1.225	0.0121	0.0119	1.302	1.342	1.031	1.03
		170	1.543	1.572	0.0153	0.0157	1.962	2.017	1.028	1.10
		160	2.099	2.163	0.0200	0.0218	3.029	3.152	1.041	1.80
		150	3.087	3.232	0.0305	0.0350	4.914	5.192	1.057	2.61
		140	4.971	5.307	0.0499	0.0526	8.505	9.147	1.076	3.86
A12	mMe-1	180	1.219	1.243	0.0124	0.0126	1.327	1.377	1.035	1.13
		170	1.565	1.598	0.0152	0.0155	2.004	2.067	1.035	1.27
		160	2.122	2.197	0.0201	0.0209	3.073	3.217	1.047	2.15
		150	3.080	3.257	0.0303	0.0317	4.900	5.239	1.069	3.36
		140	4.824	5.260	0.0464	0.0519	8.224	9.057	1.101	5.22
A13	pMe-1	170	1.700	1.725	0.0162	0.0163	2.263	2.311	1.021	*(b)
		160	2.362	2.422	0.0226	0.0234	3.534	3.649	1.033	1.54
		150	3.537	3.683	0.0342	0.0366	5.776	6.056	1.048	2.43
		140	5.758	6.123	0.0559	0.0578	10.010	10.707	1.070	3.78
		130	10.199	11.161	0.0893	0.0888	18.464	20.300	1.099	6.36
A14	oCF-1	150	2.286	2.317	0.0215	0.0216	3.379	3.439	1.018	*(b)
		140	3.328	3.396	0.0333	0.0331	5.363	5.493	1.024	1.21
		130	5.180	5.331	0.0516	0.0543	8.885	9.174	1.032	1.68
		120	8.632	8.974	0.0855	0.0882	15.442	16.093	1.042	2.32

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		110	15.380	16.180	0.1295	0.1306	28.295	29.819	1.054	3.62
A15	mCF-1	200	0.912	0.934	0.0101	0.0103	0.757	0.800	1.056	1.27
		190	1.103	1.140	0.0113	0.0116	1.121	1.192	1.063	1.90
		180	1.427	1.510	0.0146	0.0153	1.728	1.887	1.092	3.27
		170	1.994	2.186	0.0197	0.0221	2.827	3.196	1.130	5.41
		160	3.051	3.506	0.0308	0.0357	4.856	5.729	1.180	8.05
A16	pCF-1	200	1.016	1.042	0.0106	0.0109	0.958	1.008	1.052	1.42
		190	1.299	1.335	0.0136	0.0130	1.498	1.567	1.047	1.59
		180	1.786	1.871	0.0169	0.0190	2.415	2.577	1.067	2.79
		170	2.689	2.893	0.0256	0.0295	4.161	4.553	1.094	4.36
		160	4.456	4.957	0.0457	0.0495	7.553	8.514	1.127	6.19
A17	pEt	180	1.609	1.630	0.0141	0.0155	2.076	2.117	1.019	*(b)
		170	2.172	2.220	0.0204	0.0220	3.169	3.261	1.029	1.33
		160	3.131	3.245	0.0303	0.0309	5.010	5.228	1.044	2.19
		150	4.843	5.127	0.0468	0.0490	8.278	8.822	1.066	3.49
		140	8.077	8.794	0.0778	0.0851	14.444	15.815	1.095	5.18
A18	pBu	190	2.003	2.021	0.0191	0.0199	2.845	2.879	1.012	*(b)
		180	2.780	2.826	0.0268	0.0258	4.315	4.403	1.020	1.03
		170	4.078	4.188	0.0386	0.0401	6.827	7.038	1.031	1.65
		160	6.384	6.656	0.0599	0.0634	11.253	11.775	1.046	2.60
		150	10.679	11.381	0.0928	0.1003	19.458	20.803	1.069	4.28
A19	ptBu	190	1.625	1.645	0.0154	0.0155	2.119	2.157	1.018	*(b)
		180	2.175	2.222	0.0204	0.0207	3.159	3.249	1.028	1.35
		170	3.078	3.186	0.0290	0.0308	4.908	5.115	1.042	2.13
		160	4.648	4.908	0.0455	0.0471	7.921	8.420	1.063	3.30
		150	7.523	8.177	0.0725	0.0805	13.412	14.665	1.093	5.03
A20	2	180	1.207	1.220	0.0120	0.0121	1.308	1.333	1.019	*(b)
		170	1.524	1.554	0.0151	0.0153	1.925	1.983	1.030	1.16
		160	2.041	2.106	0.0197	0.0213	2.917	3.042	1.043	1.87
		150	2.930	3.076	0.0286	0.0304	4.613	4.893	1.061	2.91
		140	4.532	4.870	0.0440	0.0481	7.665	8.312	1.084	4.32
A21	pF-2	190	1.172	1.192	0.0116	0.0130	1.250	1.288	1.031	*(b)
		180	1.498	1.541	0.0148	0.0152	1.864	1.946	1.044	1.69
		170	2.044	2.135	0.0199	0.0206	2.923	3.098	1.060	2.64
		160	3.012	3.218	0.0302	0.0315	4.781	5.177	1.083	3.93
		150	4.832	5.310	0.0478	0.0520	8.257	9.172	1.111	5.64

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	**α**	Rs
A22	pCl-2	190	2.110	2.171	0.0202	0.0207	3.050	3.167	1.038	1.76
		180	3.034	3.168	0.0283	0.0309	4.801	5.057	1.053	2.66
		170	4.674	4.981	0.0453	0.0493	7.971	8.560	1.074	3.82
		160	7.778	8.501	0.0752	0.0824	13.929	15.317	1.100	5.40
		150	13.984	15.753	0.1196	0.1215	25.789	29.178	1.131	8.64
A23	pBr-2	200	2.100	2.138	0.0209	0.0203	3.046	3.119	1.024	1.09
		190	2.950	3.033	0.0280	0.0299	4.662	4.821	1.034	1.69
		180	4.439	4.625	0.0425	0.0440	7.488	7.843	1.047	2.53
		170	7.143	7.575	0.0696	0.0741	12.710	13.539	1.065	3.54
		160	12.336	13.384	0.1091	0.1217	22.678	24.689	1.089	5.34
A24	pMe-2	190	1.279	1.309	0.0123	0.0137	1.455	1.512	1.040	1.36
		180	1.689	1.758	0.0163	0.0178	2.229	2.361	1.059	2.38
		170	2.397	2.560	0.0235	0.0235	3.601	3.914	1.087	4.08
		160	3.697	4.096	0.0360	0.0394	6.096	6.862	1.126	6.23
		150	6.199	7.207	0.0607	0.0706	10.875	12.807	1.178	9.04
A25	oOMe-2	180	2.584	2.619	0.0251	0.0238	3.941	4.008	1.017	*(b)
		170	3.688	3.768	0.0352	0.0354	6.079	6.232	1.025	1.33
		160	5.583	5.770	0.0541	0.0560	9.716	10.075	1.037	2.00
		150	8.967	9.418	0.0868	0.0933	16.178	17.042	1.053	2.95
		140	15.315	16.465	0.1290	0.1352	28.283	30.482	1.078	5.12
A26	pCF-2	180	1.434	1.462	0.0145	0.0142	1.742	1.795	1.031	1.15
		170	1.876	1.914	0.0174	0.0189	2.601	2.674	1.028	1.23
		160	2.579	2.662	0.0249	0.0253	3.950	4.109	1.040	1.95
		150	3.767	3.960	0.0361	0.0386	6.216	6.586	1.059	3.04
		140	5.873	6.337	0.0563	0.0611	10.229	11.117	1.087	4.65
A27	iBu	170	1.581	1.597	0.0164	0.0155	2.035	2.065	1.015	*(b)
		160	2.098	2.137	0.0202	0.0214	3.027	3.102	1.025	1.10
		150	2.966	3.053	0.0296	0.0301	4.682	4.849	1.036	1.72
		140	4.474	4.678	0.0437	0.0447	7.554	7.945	1.052	2.72
		130	7.220	7.720	0.0719	0.0780	12.779	13.733	1.075	3.93
A28	3	160	-	2.511	-	0.0376	-	3.820	1.000	0
		150	3.614	3.661	0.0340	0.0345	5.923	6.013	1.015	*(b)
		140	5.584	5.686	0.0550	0.0567	9.677	9.872	1.020	1.07
		130	9.174	9.391	0.0881	0.0899	16.508	16.922	1.025	1.43
		120	16.121	16.618	0.1326	0.1356	29.707	30.653	1.032	2.18
A29	4	180	2.362	2.382	0.0209	0.0211	3.516	3.554	1.011	*(b)

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		170	3.351	3.402	0.0320	0.0322	5.432	5.530	1.018	*(b)
		160	5.046	5.158	0.0486	0.0504	8.685	8.900	1.025	1.33
		150	8.086	8.346	0.0768	0.0856	14.490	14.989	1.034	1.88
		140	13.828	14.477	0.1081	0.1211	25.440	26.681	1.049	3.33
A30	A	200	1.155	1.178	0.0124	0.0117	1.225	1.270	1.036	1.12
		190	1.454	1.488	0.0145	0.0148	1.791	1.856	1.036	1.37
		180	1.942	2.019	0.0191	0.0197	2.713	2.860	1.054	2.34
		170	2.779	2.962	0.0271	0.0283	4.334	4.685	1.081	3.89
		160	4.311	4.752	0.0432	0.0463	7.274	8.121	1.116	5.80
A31	5F-A	200	1.455	1.480	0.0143	0.0150	1.803	1.852	1.027	1.00
		190	1.982	2.042	0.0192	0.0212	2.804	2.919	1.041	1.75
		180	2.938	3.082	0.0280	0.0309	4.618	4.893	1.060	2.88
		170	4.738	5.099	0.0475	0.0485	8.094	8.787	1.086	4.43
		160	8.333	9.245	0.0820	0.0830	14.994	16.745	1.117	6.51
A32	5Cl-A	210	2.008	2.038	0.0194	0.0198	2.862	2.919	1.020	*(b)
		200	2.858	2.929	0.0276	0.0296	4.507	4.644	1.030	1.46
		190	4.385	4.558	0.0420	0.0438	7.417	7.749	1.045	2.37
		180	7.314	7.746	0.0717	0.0734	12.985	13.811	1.064	3.50
		170	13.167	14.281	0.1073	0.1083	24.273	26.411	1.088	6.08
A33	5Br-A	220	1.978	2.001	0.0181	0.0191	2.797	2.841	1.016	*(b)
		210	2.753	2.810	0.0269	0.0287	4.294	4.404	1.026	1.21
		200	4.096	4.226	0.0402	0.0403	6.892	7.143	1.036	1.90
		190	6.563	6.884	0.0630	0.0660	11.597	12.213	1.053	2.93
		180	11.378	12.170	0.1006	0.1123	20.755	22.270	1.073	4.38
A34	5Me-A	190	1.719	1.752	0.0178	0.0180	2.299	2.363	1.028	1.08
		180	2.334	2.413	0.0227	0.0235	3.463	3.614	1.044	2.01
		170	3.387	3.577	0.0325	0.0325	5.501	5.866	1.066	3.44
		160	5.247	5.718	0.0504	0.0552	9.071	9.975	1.100	5.25
		150	8.800	10.005	0.0804	0.0948	15.858	18.167	1.146	8.10
A35	5OMe-A	200	2.245	2.257	0.0204	0.0205	3.326	3.349	1.007	*(a)
		190	3.138	3.189	0.0294	0.0302	5.023	5.121	1.019	1.01
		180	4.685	4.815	0.0453	0.0470	7.958	8.207	1.031	1.66
		170	7.464	7.806	0.0698	0.0745	13.326	13.983	1.049	2.79
		160	12.693	13.600	0.1143	0.1190	23.363	25.104	1.075	4.58
A36	1ATL	200	1.361	1.370	0.0128	0.0134	1.622	1.640	1.011	*(a)
		190	1.721	1.750	0.0175	0.0177	2.303	2.359	1.024	*(b)

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		180	2.298	2.367	0.0213	0.0229	3.394	3.526	1.039	1.84
		170	3.257	3.417	0.0311	0.0326	5.251	5.559	1.058	2.96
		160	4.888	5.294	0.0486	0.0502	8.382	9.161	1.093	4.84
A37	2ATL	200	1.746	1.773	0.0165	0.0172	2.364	2.416	1.022	*(b)
		190	2.367	2.423	0.0229	0.0234	3.543	3.651	1.030	1.42
		180	3.442	3.561	0.0325	0.0348	5.581	5.809	1.041	2.08
		170	5.365	5.631	0.0525	0.0534	9.298	9.808	1.055	2.96
		160	9.001	9.610	0.0845	0.0907	16.276	17.445	1.072	4.09
A38	Nap	200	-	2.514	-	0.0243	-	3.844	1.000	0
		190	-	3.464	-	0.0333	-	5.649	1.000	0
		180	-	5.012	-	0.0494	-	8.602	1.000	0
		170	-	7.590	-	0.0721	-	13.568	1.000	0
		160	-	12.106	-	0.1092	-	22.236	1.000	0
A39	pF-6	200	-	2.510	-	0.0243	-	3.836	1.000	0
		190	-	3.513	-	0.0374	-	5.743	1.000	0
		180	-	5.185	-	0.0604	-	8.933	1.000	0
		170	-	8.064	-	0.1060	-	14.478	1.000	0
		160	13.116	13.247	0.1123	0.1064	24.175	24.426	1.010	*(b)
A40	pCl-6	220	-	2.511	-	0.0268	-	3.820	1.000	0
		210	-	3.457	-	0.0385	-	5.648	1.000	0
		200	-	4.978	-	0.0716	-	8.592	1.000	0
		190	-	7.559	-	0.1160	-	13.509	1.000	0
		180	11.901	12.075	0.0920	0.1023	21.755	22.088	1.015	1.05
A41	pBr-6	220	-	3.402	-	0.0372	-	5.530	1.000	0
		210	-	4.839	-	0.0640	-	8.306	1.000	0
		200	7.160	7.208	0.0583	0.0609	12.796	12.888	1.007	*(a)
		190	11.129	11.281	0.1051	0.0974	20.361	20.653	1.014	*(b)
		180	18.204	18.570	0.1358	0.1475	33.807	34.507	1.021	1.52
A42	pMe-6	200	-	2.843	-	0.0273	-	4.478	1.000	0
		190	-	3.979	-	0.0387	-	6.637	1.000	0
		180	-	5.831	-	0.0564	-	10.170	1.000	0
		170	-	8.908	-	0.0846	-	16.098	1.000	0
		160	-	14.200	-	0.1112	-	26.255	1.000	0
A43	pOMe-6	220	-	2.670	-	0.0261	-	4.125	1.000	0
		210	-	3.666	-	0.0355	-	6.050	1.000	0
		200	-	5.260	-	0.0516	-	9.135	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		190	-	7.842	-	0.0774	-	14.052	1.000	0
		180	-	12.282	-	0.1171	-	22.529	1.000	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

Table B3 Chromatographic results of 72 chiral alcohols analyzed by BSiMe column (14.91 m long) using isothermal conditions.

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
L01	PE	150	1.343	1.364	0.0145	0.0141	1.670	1.712	1.025	*(b)
		140	1.779	1.824	0.0180	0.0200	2.530	2.619	1.035	1.39
		130	2.498	2.590	0.0272	0.0289	3.947	4.129	1.046	1.93
		120	3.700	3.893	0.0407	0.0427	6.283	6.663	1.060	2.72
		110	5.769	6.179	0.0638	0.0673	10.356	11.163	1.078	3.68
L02	oF-PE	160	1.052	1.076	0.0115	0.0129	1.091	1.139	1.044	1.16
		150	1.322	1.375	0.0132	0.0148	1.628	1.734	1.065	2.23
		140	1.769	1.888	0.0180	0.0200	2.510	2.746	1.094	3.69
		130	2.516	2.780	0.0264	0.0294	3.982	4.505	1.131	5.57
		120	3.783	4.367	0.0419	0.0484	6.447	7.596	1.178	7.61
L03	mF-PE	150	1.524	1.550	0.0152	0.0168	2.030	2.082	1.025	*(b)
		140	2.096	2.150	0.0225	0.0237	3.159	3.266	1.034	1.38
		130	3.059	3.169	0.0322	0.0339	5.057	5.275	1.043	1.96
		120	4.722	4.950	0.0509	0.0533	8.295	8.744	1.054	2.58
		110	7.659	8.140	0.0862	0.0881	14.018	14.961	1.067	3.25
L04	pF-PE	140	2.044	2.088	0.0205	0.0216	3.056	3.143	1.029	1.23
		130	2.937	3.035	0.0313	0.0339	4.816	5.010	1.040	1.77
		120	4.440	4.658	0.0482	0.0505	7.740	8.169	1.055	2.60
		110	7.071	7.564	0.0774	0.0821	12.919	13.890	1.075	3.64
		100	11.749	12.867	0.1122	0.1173	21.947	24.131	1.099	5.73
L05	oCl-PE	200	0.832	0.851	0.0094	0.0103	0.664	0.702	1.057	1.14
		190	0.961	0.999	0.0109	0.0125	0.922	0.998	1.082	1.91
		180	1.159	1.237	0.0136	0.0140	1.295	1.450	1.119	3.33
		170	1.470	1.633	0.0154	0.0169	1.934	2.259	1.168	5.94
		160	1.979	2.322	0.0207	0.0249	2.934	3.616	1.232	8.85
L06	mCl-PE	160	2.283	2.320	0.0236	0.0250	3.539	3.612	1.021	*(b)
		150	3.301	3.380	0.0345	0.0364	5.473	5.627	1.028	1.31
		140	5.059	5.229	0.0538	0.0564	9.038	9.375	1.037	1.82
		130	8.155	8.524	0.0929	0.0958	15.149	15.879	1.048	2.30
		120	13.783	14.610	0.1386	0.1651	26.132	27.760	1.062	3.21
L07	pCl-PE	160	2.325	2.361	0.0220	0.0243	3.622	3.694	1.020	*(b)
		150	3.326	3.408	0.0327	0.0366	5.612	5.775	1.029	1.39
		140	5.008	5.193	0.0519	0.0533	8.937	9.304	1.041	2.07

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		130	7.872	8.290	0.0812	0.0825	14.588	15.416	1.057	3.01
		120	12.914	13.871	0.1217	0.1296	24.421	26.305	1.077	4.48
L08	oBr-PE	210	0.858	0.879	0.0099	0.0103	0.706	0.748	1.059	1.22
		200	0.994	1.037	0.0110	0.0125	0.988	1.074	1.087	2.15
		190	1.197	1.284	0.0134	0.0141	1.394	1.568	1.125	3.72
		180	1.518	1.699	0.0166	0.0181	2.006	2.364	1.179	6.14
		170	2.031	2.409	0.0210	0.0247	3.038	3.789	1.247	9.74
L09	mBr-PE	160	3.357	3.402	0.0324	0.0353	5.674	5.763	1.016	*(b)
		150	5.092	5.191	0.0513	0.0559	9.123	9.320	1.022	1.09
		140	8.140	8.360	0.0817	0.0881	15.151	15.587	1.029	1.52
		130	13.628	14.130	0.1087	0.1311	25.986	26.980	1.038	2.46
		120	23.914	25.047	0.1953	0.2136	46.075	48.305	1.048	3.26
L10	pBr-PE	160	3.501	3.555	0.0335	0.0367	5.960	6.068	1.018	*(b)
		150	5.246	5.371	0.0528	0.0568	9.429	9.678	1.026	1.34
		140	8.223	8.509	0.0836	0.0885	15.315	15.883	1.037	1.96
		130	13.428	14.088	0.1127	0.1184	25.590	26.897	1.051	3.36
		120	22.790	24.355	0.1827	0.1995	43.862	46.943	1.070	4.82
L11	oMe-PE	170	1.201	1.224	0.0123	0.0129	1.397	1.443	1.033	1.07
		160	1.555	1.607	0.0171	0.0179	2.091	2.195	1.049	1.75
		150	2.134	2.254	0.0225	0.0237	3.243	3.481	1.074	3.06
		140	3.118	3.395	0.0339	0.0367	5.187	5.736	1.106	4.62
		130	4.811	5.445	0.0533	0.0607	8.508	9.761	1.147	6.55
L12	mMe-PE	160	1.341	1.366	0.0148	0.0151	1.666	1.716	1.030	*(b)
		150	1.768	1.819	0.0189	0.0194	2.515	2.616	1.040	1.57
		140	2.483	2.587	0.0255	0.0277	3.927	4.133	1.053	2.30
		130	3.703	3.924	0.0404	0.0425	6.333	6.770	1.069	3.14
		120	5.829	6.299	0.0645	0.0691	10.474	11.400	1.088	4.14
L13	pMe-PE	160	1.317	1.337	0.0141	0.0138	1.618	1.658	1.025	*(b)
		150	1.708	1.750	0.0176	0.0186	2.396	2.479	1.035	1.37
		140	2.337	2.426	0.0238	0.0255	3.637	3.813	1.049	2.12
		130	3.368	3.557	0.0355	0.0376	5.669	6.044	1.066	3.04
		120	5.097	5.502	0.0542	0.0593	9.033	9.831	1.088	4.20
L14	oOMe-PE	190	0.959	0.993	0.0106	0.0109	0.918	0.986	1.074	1.86
		180	1.148	1.212	0.0121	0.0126	1.273	1.400	1.100	3.05
		170	1.441	1.566	0.0152	0.0162	1.876	2.126	1.133	4.69
		160	1.911	2.155	0.0205	0.0235	2.792	3.276	1.173	6.53

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		150	2.688	3.185	0.0289	0.0344	4.333	5.319	1.228	9.24
L15	mOMe-PE	170	1.692	1.716	0.0165	0.0176	2.377	2.425	1.020	*(b)
		160	2.300	2.353	0.0233	0.0246	3.573	3.678	1.029	1.30
		150	3.314	3.429	0.0338	0.0362	5.588	5.817	1.041	1.93
		140	5.049	5.301	0.0531	0.0568	9.018	9.518	1.055	2.70
		130	8.103	8.666	0.0879	0.0957	15.014	16.126	1.074	3.61
L16	pOMe-PE	150	1.744	1.765	0.0163	0.0167	2.467	2.509	1.017	*(b)
		140	2.332	2.373	0.0228	0.0236	3.627	3.708	1.022	1.04
		130	3.275	3.354	0.0319	0.0343	5.485	5.642	1.029	1.40
		120	4.827	4.980	0.0478	0.0515	8.502	8.803	1.035	1.81
		110	7.447	7.751	0.0746	0.0796	13.659	14.258	1.044	2.32
L17	oCF-PE	170	0.862	0.900	0.0099	0.0104	0.721	0.796	1.105	2.20
		160	1.033	1.114	0.0126	0.0120	1.054	1.215	1.153	3.88
		150	1.305	1.482	0.0142	0.0156	1.594	1.946	1.221	6.99
		140	1.759	2.151	0.0187	0.0222	2.490	3.268	1.312	11.2
		130	2.531	3.399	0.0275	0.0379	4.002	5.717	1.429	15.6
L18	mCF-PE	150	1.485	1.502	0.0150	0.0161	1.952	1.986	1.017	*(b)
		140	2.041	2.079	0.0200	0.0227	3.050	3.125	1.025	1.05
		130	2.991	3.074	0.0299	0.0328	4.923	5.087	1.033	1.56
		120	4.662	4.843	0.0497	0.0566	8.177	8.533	1.044	2.00
		110	7.704	8.109	0.0845	0.0854	14.165	14.963	1.056	2.81
L19	pCF-PE	160	1.281	1.302	0.0136	0.0142	1.547	1.588	1.027	*(b)
		150	1.681	1.725	0.0178	0.0184	2.342	2.429	1.037	1.43
		140	2.339	2.430	0.0240	0.0260	3.641	3.821	1.050	2.14
		130	3.432	3.626	0.0359	0.0402	5.796	6.180	1.066	3.00
		120	5.298	5.716	0.0552	0.0617	9.429	10.252	1.087	4.21
L20	oNO-PE	220	0.971	1.005	0.017	0.011	0.942	1.010	1.072	1.84
		210	1.156	1.224	0.0134	0.0136	1.298	1.433	1.104	2.96
		200	1.439	1.578	0.0154	0.0174	1.878	2.156	1.148	4.99
		190	1.887	2.175	0.0196	0.0230	2.774	3.350	1.208	7.96
		180	2.621	3.234	0.0276	0.0345	4.221	5.442	1.289	11.6
L21	mNO-PE	190	-	2.888	-	0.0322	-	4.674	1.000	0
		180	-	4.182	-	0.0497	-	7.184	1.000	0
		170	-	6.424	-	0.0929	-	11.596	1.000	0
		160	10.096	10.221	0.0898	0.0977	19.032	19.280	1.013	*(b)
		150	16.615	16.973	0.1564	0.2144	31.966	32.677	1.022	1.14

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
L22	pNO-PE	190	3.377	3.415	0.0303	0.0357	5.754	5.830	1.013	*(b)
		180	4.980	5.074	0.0527	0.0502	8.861	9.048	1.021	1.08
		170	7.641	7.859	0.0774	0.0828	14.251	14.687	1.031	1.60
		160	12.276	12.783	0.1240	0.1230	23.357	24.363	1.043	2.42
		150	20.521	21.724	0.1653	0.1752	39.716	42.103	1.060	4.16
L23	pEt-PE	150	2.429	2.469	0.0242	0.0257	3.829	3.909	1.021	*(b)
		140	3.509	3.596	0.0358	0.0380	5.962	6.135	1.029	1.39
		130	5.319	5.511	0.0586	0.0584	9.533	9.913	1.040	1.93
		120	8.432	8.853	0.0868	0.0899	15.598	16.427	1.053	2.80
		110	13.969	14.932	0.1321	0.1383	26.498	28.394	1.072	4.19
L24	pBU-PE	160	3.522	3.582	0.0338	0.0352	6.002	6.121	1.020	1.02
		150	5.293	5.422	0.0509	0.0551	9.523	9.779	1.027	1.43
		140	8.342	8.625	0.0799	0.0855	15.552	16.113	1.036	2.01
		130	13.733	14.367	0.1127	0.1408	26.302	27.563	1.048	2.94
		120	23.547	24.996	0.1893	0.2035	45.352	48.205	1.063	4.34
L25	ptBU-PE	150	4.187	4.227	0.0380	0.0392	7.324	7.404	1.011	*(b)
		140	6.525	6.619	0.0617	0.0679	11.946	12.133	1.016	*(b)
		130	10.653	10.850	0.1040	0.1056	20.053	20.443	1.019	1.11
		120	18.215	18.656	0.1406	0.1528	34.856	35.724	1.025	1.77
		110	32.362	33.307	0.2542	0.2688	62.705	64.565	1.030	2.13
L26	pPh-PE	190	-	5.744	-	0.0931	-	10.285	1.000	0
		180	8.626	8.728	0.0762	0.0865	16.218	16.421	1.013	*(b)
		170	13.766	14.012	0.1426	0.1494	26.477	26.968	1.019	*(b)
		160	22.916	23.489	0.1845	0.1900	44.559	45.698	1.026	1.80
		150	39.386	40.795	0.3085	0.3391	77.147	79.942	1.036	2.56
L27	24F-PE	170	0.871	0.896	0.0101	0.0104	0.739	0.788	1.068	1.44
		160	1.038	1.091	0.0113	0.0118	1.064	1.169	1.099	2.70
		150	1.306	1.418	0.0141	0.0149	1.596	1.819	1.139	4.55
		140	1.742	1.982	0.0188	0.0207	2.456	2.933	1.194	7.15
		130	2.474	2.995	0.0274	0.0316	3.880	4.907	1.265	10.3
L28	25F-PE	170	0.959	0.984	0.0106	0.0109	0.907	0.956	1.055	1.37
		160	1.190	1.243	0.0137	0.0142	1.366	1.471	1.077	2.24
		150	1.574	1.685	0.0164	0.0182	2.129	2.350	1.104	3.78
		140	2.230	2.468	0.0236	0.0269	3.425	3.897	1.138	5.55
		130	3.381	3.898	0.0369	0.0419	5.669	6.688	1.180	7.72
L29	34F-PE	160	1.286	1.308	0.0130	0.0145	1.557	1.600	1.028	*(b)

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		150	1.685	1.732	0.0184	0.0195	2.350	2.443	1.040	1.46
		140	2.338	2.436	0.0259	0.0258	3.639	3.833	1.053	2.23
		130	3.418	3.626	0.0361	0.0393	5.768	6.180	1.071	3.25
		120	5.290	5.739	0.0585	0.0622	9.434	10.320	1.094	4.38
L30	26F-PE	160	0.881	0.903	0.0103	0.0107	0.751	0.795	1.058	1.23
		150	1.049	1.092	0.0118	0.0132	1.085	1.171	1.079	2.02
		140	1.305	1.394	0.0149	0.0159	1.589	1.766	1.111	3.40
		130	1.718	1.899	0.0188	0.0213	2.402	2.760	1.149	5.31
		120	2.399	2.780	0.0263	0.0315	3.732	4.483	1.201	7.76
L31	35F-PE	140	-	2.113	-	0.0229	-	3.119	1.000	0
		130	-	3.111	-	0.0129	-	5.041	1.000	0
		120	-	4.855	-	0.0552	-	8.391	1.000	0
		110	-	8.026	-	0.1033	-	14.464	1.000	0
		100	-	13.930	-	0.1824	-	25.686	1.000	0
L32	triF-PE	190	0.710	0.733	0.0096	0.0087	0.420	0.466	1.110	1.49
		180	0.793	0.837	0.0112	0.0112	0.570	0.657	1.153	2.31
		170	0.915	1.000	0.0107	0.0118	0.826	0.996	1.205	4.45
		160	1.115	1.286	0.0146	0.0156	1.217	1.557	1.279	6.66
		150	1.429	1.773	0.0164	0.0240	1.835	2.518	1.372	10.0
L33	tetraF-PE	150	1.447	1.467	0.0151	0.0146	1.877	1.917	1.021	*(b)
		140	1.969	2.017	0.0214	0.0223	2.907	3.002	1.033	1.29
		130	2.843	2.953	0.0303	0.0321	4.630	4.848	1.047	2.07
		120	4.360	4.613	0.0482	0.0495	7.600	8.099	1.066	3.05
		110	7.052	7.639	0.0775	0.0795	12.855	14.008	1.090	4.40
L34	pentaF-PE	150	1.071	1.103	0.0114	0.0128	1.129	1.193	1.056	1.56
		140	1.358	1.423	0.0148	0.0154	1.694	1.823	1.076	2.53
		130	1.823	1.957	0.0193	0.0202	2.610	2.875	1.102	3.99
		120	2.605	2.886	0.0280	0.0315	4.138	4.692	1.134	5.56
		110	3.940	4.533	0.0422	0.0491	6.725	7.888	1.173	7.64
L35	24Cl-PE	210	1.006	1.032	0.0109	0.0113	1.000	1.052	1.052	1.38
		200	1.209	1.263	0.0123	0.0140	1.418	1.526	1.076	2.42
		190	1.518	1.631	0.0167	0.0174	2.036	2.262	1.111	3.90
		180	2.012	2.249	0.0206	0.0237	2.984	3.453	1.157	6.30
		170	2.812	3.321	0.0286	0.0336	4.613	5.629	1.220	9.63
L36	25Cl-PE	220	0.896	0.925	0.0174	0.0103	0.792	0.850	1.073	1.23
		210	1.050	1.106	0.0114	0.0129	1.087	1.199	1.102	2.71

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		200	1.285	1.397	0.0142	0.0150	1.570	1.794	1.143	4.51
		190	1.659	1.885	0.0168	0.0193	2.318	2.770	1.195	7.37
		180	2.275	2.741	0.0223	0.0283	3.532	4.460	1.263	10.8
L37	34Cl-PE	170	3.702	3.754	0.0371	0.0391	6.389	6.493	1.016	*(b)
		160	5.575	5.695	0.0561	0.0609	10.083	10.322	1.024	1.21
		150	8.789	9.065	0.0917	0.0888	16.473	17.022	1.033	1.80
		140	14.488	15.125	0.1288	0.1297	27.746	29.010	1.046	2.90
		130	24.895	26.366	0.2028	0.2128	48.297	51.210	1.060	4.17
L38	24Me-PE	180	1.183	1.205	0.0132	0.0124	1.343	1.386	1.032	1.01
		170	1.505	1.555	0.0165	0.0169	2.004	2.104	1.050	1.76
		160	2.028	2.143	0.0206	0.0224	3.032	3.260	1.075	3.15
		150	2.900	3.164	0.0295	0.0329	4.765	5.290	1.110	4.98
		140	4.393	5.000	0.0464	0.0520	7.716	8.921	1.156	7.26
L39	25Me-PE	190	0.962	0.981	0.0106	0.0110	0.916	0.954	1.041	1.04
		180	1.163	1.201	0.0131	0.0124	1.303	1.378	1.058	1.75
		170	1.481	1.558	0.0154	0.0172	1.944	2.097	1.079	2.78
		160	2.001	2.162	0.0203	0.0227	2.978	3.298	1.107	4.41
		150	2.844	3.226	0.0307	0.0333	4.654	5.414	1.163	7.03
L40	34Me-PE	140	4.138	4.182	0.0378	0.0430	7.194	7.281	1.012	*(b)
		130	6.468	6.576	0.0626	0.0717	11.808	12.022	1.018	*(b)
		120	10.640	10.884	0.1037	0.1096	19.945	20.425	1.024	1.35
		110	18.289	18.848	0.1617	0.1562	34.931	36.029	1.031	2.07
		100	32.702	34.023	0.2634	0.2706	62.996	65.581	1.041	2.91
L41	PP	130	-	3.847	-	0.0660	-	6.470	1.000	0
		120	5.934	6.017	0.0573	0.0657	10.704	10.868	1.015	*(b)
		110	9.818	10.050	0.0984	0.1081	18.289	18.745	1.025	1.32
		100	16.944	17.544	0.1410	0.1424	32.159	33.333	1.037	2.49
		90	30.484	32.041	0.2508	0.2667	58.192	61.216	1.052	3.54
L42	pF-PP	140	2.957	2.997	0.0266	0.0339	4.867	4.946	1.016	*(b)
		130	4.525	4.629	0.0451	0.0499	7.960	8.166	1.026	1.29
		120	7.249	7.511	0.0748	0.0890	13.298	13.815	1.039	1.88
		110	12.277	12.928	0.1241	0.1257	23.120	24.399	1.055	3.07
		100	21.504	23.132	0.1838	0.2017	41.082	44.268	1.078	4.97
L43	pCl-PP	150	4.808	4.878	0.0448	0.0526	8.559	8.698	1.016	*(b)
		140	7.584	7.756	0.0754	0.0818	14.048	14.389	1.024	1.29
		130	12.497	12.917	0.1206	0.1293	23.747	24.578	1.035	1.98

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		120	21.165	22.209	0.2199	0.2637	40.746	42.805	1.051	2.54
		110	38.135	40.717	0.3379	0.3713	73.921	78.994	1.069	4.29
L44	pBr-PP	160	-	5.011	-	0.0652	-	8.806	1.000	0
		150	7.698	7.772	0.0722	0.0917	14.304	14.451	1.010	*(b)
		140	12.597	12.815	0.1094	0.1276	23.994	24.427	1.018	1.08
		130	21.483	22.039	0.2330	0.2521	41.541	42.642	1.027	1.35
		120	37.979	39.428	0.3026	0.3192	73.909	76.767	1.039	2.74
L45	pMe-PP	160	1.701	1.720	0.0161	0.0169	2.382	2.419	1.016	*(b)
		150	2.322	2.366	0.0235	0.0258	3.616	3.704	1.024	1.05
		140	3.348	3.446	0.0344	0.0355	5.643	5.837	1.034	1.65
		130	5.076	5.291	0.0539	0.0551	9.051	9.477	1.047	2.32
		120	8.096	8.580	0.0853	0.0897	14.968	15.923	1.064	3.26
L46	pOMe-PP	160	3.210	3.251	0.0301	0.0326	5.382	5.463	1.015	*(b)
		150	4.779	4.866	0.0470	0.0523	8.501	8.674	1.020	1.03
		140	7.471	7.664	0.0762	0.0827	13.823	14.206	1.028	1.43
		130	12.216	12.647	0.1170	0.1160	23.190	24.044	1.037	2.18
		120	20.884	21.880	0.2278	0.2243	40.191	42.156	1.049	2.59
L47	pCF3-PP	150	2.284	2.342	0.0227	0.0243	3.541	3.656	1.033	1.45
		140	3.368	3.490	0.0343	0.0361	5.683	5.924	1.043	2.04
		130	5.233	5.486	0.0531	0.0569	9.362	9.863	1.054	2.71
		120	8.571	9.116	0.0882	0.0912	15.905	16.980	1.068	3.58
		110	14.670	15.869	0.1196	0.1292	27.765	30.116	1.085	5.67
L48	1OH	150	1.478	1.498	0.0149	0.0158	1.938	1.978	1.021	*(b)
		140	1.965	2.004	0.0201	0.0214	2.899	2.976	1.027	1.11
		130	2.757	2.832	0.0300	0.0314	4.459	4.608	1.033	1.44
		120	4.066	4.210	0.0430	0.0467	7.020	7.304	1.040	1.89
		110	6.310	6.594	0.0722	0.0754	11.397	11.955	1.049	2.26
L49	2OH	150	-	2.158	-	0.0228	-	3.290	1.000	0
		140	-	3.145	-	0.0330	-	5.240	1.000	0
		130	-	4.850	-	0.0557	-	8.604	1.000	0
		120	-	7.893	-	0.0865	-	14.568	1.000	0
		110	-	13.429	-	0.0510	-	25.383	1.000	0
L50	3OH	170	1.364	1.391	0.0131	0.0135	1.723	1.776	1.031	1.19
		160	1.794	1.841	0.0184	0.0192	2.567	2.660	1.036	1.47
		150	2.506	2.591	0.0247	0.0267	3.982	4.151	1.042	1.95
		140	3.713	3.870	0.0376	0.0392	6.367	6.679	1.049	2.41

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		130	5.826	6.119	0.0608	0.0634	10.537	11.117	1.055	2.78
L51	4OH	160	1.702	1.733	0.0167	0.0176	2.384	2.445	1.026	1.06
		150	2.343	2.402	0.0237	0.0247	3.658	3.775	1.032	1.43
		140	3.415	3.528	0.0352	0.0364	5.776	6.000	1.039	1.86
		130	5.257	5.480	0.0559	0.0593	9.410	9.851	1.047	2.28
		120	8.520	8.961	0.0858	0.0934	15.805	16.675	1.055	2.90
L52	5OH	160	1.496	1.520	0.0153	0.0160	1.974	2.022	1.024	*(b)
		150	1.977	2.023	0.0208	0.0219	2.923	3.014	1.031	1.27
		140	2.748	2.838	0.0279	0.0292	4.452	4.631	1.040	1.86
		130	4.022	4.198	0.0416	0.0446	6.964	7.313	1.050	2.40
		120	6.175	6.525	0.0658	0.0713	11.179	11.870	1.062	3.00
L53	6OH	140	3.329	3.359	0.0295	0.0331	5.605	5.665	1.011	*(b)
		130	4.998	5.080	0.0469	0.0584	8.897	9.059	1.018	*(b)
		120	7.899	8.089	0.0785	0.0911	14.580	14.955	1.026	1.32
		110	13.050	13.495	0.1323	0.1314	24.639	25.513	1.035	1.99
		100	22.393	23.453	0.1894	0.2041	42.822	44.896	1.048	3.17
L54	7OH	140	-	3.653	-	0.0617	-	6.121	1.000	0
		130	-	5.648	-	0.0960	-	9.967	1.000	0
		120	8.956	9.054	0.0745	0.0880	16.665	16.858	1.012	*(b)
		110	15.306	15.520	0.1841	0.1855	29.071	29.491	1.014	*(b)
		100	27.839	28.313	0.2158	0.2639	52.229	53.136	1.017	1.16
L55	8OH	160	1.369	1.387	0.0131	0.0142	1.722	1.757	1.021	*(b)
		150	1.798	1.832	0.0191	0.0203	2.567	2.635	1.026	1.02
		140	2.485	2.548	0.0249	0.0276	3.931	4.056	1.032	1.41
		130	3.639	3.756	0.0389	0.0413	6.206	6.438	1.037	1.72
		120	5.607	5.830	0.0618	0.0669	10.059	10.499	1.044	2.04
L56	DPE	170	8.004	8.081	0.0673	0.0748	14.976	15.130	1.010	*(b)
		160	12.854	13.024	0.1100	0.1249	24.555	24.893	1.014	*(b)
		150	21.562	21.909	0.1581	0.1813	41.782	42.470	1.016	*(b)
		140	37.776	38.534	0.3818	0.4320	73.952	75.456	1.020	*(b)
		130	69.104	70.654	0.4711	0.5151	135.84	138.91	1.022	1.23
L57	In	140	-	3.824	-	0.0448	-	6.454	1.000	0
		130	-	5.807	-	0.0631	-	10.276	1.000	0
		120	-	9.250	-	0.1144	-	16.892	1.000	0
		110	15.227	15.387	0.1072	0.1444	28.339	28.647	1.011	*(b)
		100	25.915	26.442	0.2041	0.2648	48.551	49.558	1.021	1.32

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
L58	1-Nap	150	-	4.669	-	0.0619	-	8.101	1.000	0
		140	7.135	7.244	0.0679	0.0803	13.129	13.345	1.016	*(b)
		130	11.534	11.867	0.1146	0.1226	21.840	22.499	1.030	1.65
		120	19.403	20.302	0.1660	0.1724	37.270	39.043	1.048	3.13
		110	33.715	36.002	0.2753	0.2877	65.238	69.731	1.069	4.78
L59	2-Nap	170	-	2.227	-	0.0289	-	3.367	1.000	0
		160	-	3.491	-	0.0382	-	5.832	1.000	0
		150	-	5.247	-	0.0570	-	9.228	1.000	0
		140	-	8.165	-	0.0956	-	14.916	1.000	0
		130	-	13.415	-	0.1428	-	25.049	1.000	0
L60	1Nap-E	210	1.573	1.612	0.0162	0.0170	2.127	2.205	1.036	1.38
		200	2.030	2.102	0.0224	0.0215	3.060	3.204	1.047	1.93
		190	2.745	2.881	0.0290	0.0294	4.490	4.762	1.061	2.74
		180	3.907	4.169	0.0407	0.0424	6.737	7.255	1.077	3.71
		170	5.837	6.349	0.0627	0.0654	10.651	11.673	1.096	4.70
L61	2Nap-E	180	3.874	3.908	0.0342	0.0387	6.733	6.800	1.010	*(b)
		170	5.686	5.775	0.0560	0.0615	10.349	10.527	1.017	*(b)
		160	8.711	8.914	0.0840	0.0918	16.318	16.722	1.025	1.36
		150	13.922	14.385	0.1258	0.1370	26.623	27.542	1.035	2.07
		140	23.150	24.205	0.2530	0.2498	44.842	46.931	1.047	2.47
L62	pF-Ph	200	-	2.628	-	0.0271	-	4.173	1.000	0
		190	-	3.595	-	0.0363	-	6.176	1.000	0
		180	-	5.310	-	0.0561	-	9.391	1.000	0
		170	-	8.104	-	0.0841	-	5.871	1.000	0
		160	-	12.953	-	0.1272	-	24.348	1.000	0
L63	pCl-Ph	200	-	2.289	-	0.0243	-	3.506	1.000	0
		190	-	3.171	-	0.0356	-	5.230	1.000	0
		180	-	4.610	-	0.0556	-	8.022	1.000	0
		170	-	7.037	-	0.0923	-	12.798	1.000	0
		160	-	11.275	-	0.1336	-	21.065	1.000	0
L64	pBr-Ph	200	-	4.621	-	0.0808	-	8.096	1.000	0
		190	6.760	6.832	0.0594	0.0666	12.493	12.637	1.012	*(b)
		180	10.508	10.665	0.0969	0.1070	19.932	20.245	1.016	*(b)
		170	17.092	17.418	0.1329	0.1564	33.116	33.766	1.020	1.33
		160	28.961	29.651	0.2048	0.2242	56.577	57.948	1.024	1.89
L65	oMe-Ph	200	-	6.765	-	0.1059	-	12.317	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		190	10.143	10.270	0.0943	0.1001	19.205	19.458	1.013	*(b)
		180	16.241	16.501	0.1382	0.1563	31.353	31.871	1.017	1.04
		170	27.103	27.650	0.2039	0.2330	52.990	54.080	1.021	1.47
		160	47.268	48.474	0.3491	0.3756	93.159	95.562	1.026	1.96
L66	mMe-Ph	180	5.302	5.418	0.0534	0.0545	9.562	9.793	1.024	1.27
		170	8.167	8.399	0.0798	0.0851	15.301	15.764	1.030	1.66
		160	13.212	13.691	0.1284	0.1349	25.266	26.219	1.038	2.14
		150	22.376	23.398	0.1752	0.1876	43.397	45.425	1.047	3.32
		140	39.759	41.940	0.3068	0.3221	77.731	82.050	1.056	4.08
L67	pMe-Ph	190	3.845	3.887	0.0353	0.0381	6.675	6.758	1.013	*(b)
		180	5.626	5.715	0.0537	0.0578	10.207	10.384	1.017	*(b)
		170	8.630	8.805	0.0849	0.0880	16.226	16.575	1.022	1.19
		160	13.831	14.192	0.1195	0.1254	26.497	27.215	1.027	1.73
		150	23.144	23.890	0.1853	0.1940	44.921	46.401	1.033	2.31
L68	oOMe-Ph	200	4.858	4.893	0.0443	0.0429	8.716	8.786	1.008	*(a)
		190	7.189	7.281	0.0662	0.0734	13.349	13.533	1.014	*(b)
		180	11.109	11.295	0.0991	0.1048	21.129	21.500	1.018	1.07
		170	17.875	18.263	0.1600	0.1535	34.679	35.453	1.022	1.46
		160	30.105	30.925	0.2279	0.2366	58.851	60.481	1.028	2.08
L69	2Ph-Bu	140	3.040	3.074	0.0282	0.0325	5.020	5.087	1.013	*(b)
		130	4.542	4.616	0.0461	0.0510	7.994	8.141	1.018	*(b)
		120	7.153	7.309	0.0807	0.0927	13.108	13.416	1.023	1.06
		110	11.836	12.174	0.1006	0.1241	22.253	22.917	1.030	1.77
		100	20.494	21.215	0.1680	0.1732	39.106	40.517	1.036	2.49
L70	pF-CF	160	1.382	1.408	0.0148	0.0152	1.748	1.799	1.030	1.02
		150	1.893	1.939	0.0206	0.0206	2.763	2.855	1.033	1.31
		140	2.775	2.856	0.0284	0.0308	4.495	4.655	1.036	1.61
		130	4.333	4.474	0.0460	0.0488	7.580	7.859	1.037	1.75
		120	7.186	7.433	0.0755	0.0818	13.174	13.661	1.037	1.85
L71	pCl-CF	170	-	2.014	-	0.0271	-	2.949	1.000	0
		160	-	2.900	-	0.0404	-	4.675	1.000	0
		150	-	4.411	-	0.0589	-	7.598	1.000	0
		140	-	7.127	-	0.0799	-	12.893	1.000	0
		130	-	12.054	-	0.1245	-	22.406	1.000	0
L72	pBr-CF	170	-	2.943	-	0.0360	-	4.771	1.000	0
		160	-	4.460	-	0.0529	-	7.728	1.000	0

no.	abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	K'_1	K'_2	α	Rs
		150	-	7.106	-	0.0778	-	12.852	1.000	0
		140	-	11.879	-	0.1172	-	22.066	1.000	0
		130	-	20.410	-	0.1902	-	39.416	1.000	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

Table B4 Chromatographic results of 43 chiral amines analyzed by BSiAc column (14.91 m long) using isothermal conditions.

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
A01	1	140	2.535	2.580	0.0257	0.0256	4.010	4.099	1.022	1.03
		130	3.667	3.757	0.0365	0.0391	6.233	6.410	1.028	1.40
		120	5.608	5.788	0.0560	0.0597	10.061	10.416	1.035	1.83
		110	9.016	9.388	0.0886	0.0979	16.678	17.408	1.044	2.35
		100	15.472	16.273	0.1196	0.1289	28.754	30.294	1.054	3.79
A02	oF-1	140	2.006	2.059	0.0208	0.0204	2.964	3.069	1.035	1.51
		130	2.789	2.896	0.0282	0.0302	4.501	4.712	1.047	2.16
		120	4.096	4.315	0.0418	0.0444	7.079	7.511	1.061	2.99
		110	6.334	6.788	0.0657	0.0713	11.420	12.310	1.078	3.90
		100	10.452	11.438	0.1062	0.1113	19.100	20.996	1.099	5.34
A03	mF-1	150	2.150	2.202	0.0217	0.0227	3.274	3.378	1.032	1.38
		140	3.063	3.169	0.0310	0.0322	5.053	5.263	1.041	1.97
		130	4.607	4.824	0.0461	0.0504	8.087	8.515	1.053	2.65
		120	7.337	7.791	0.0762	0.0821	13.443	14.337	1.066	3.38
		110	12.464	13.445	0.1096	0.1301	23.202	25.107	1.082	4.82
A04	pF-1	150	2.213	2.258	0.0221	0.0218	3.400	3.489	1.026	1.21
		140	3.153	3.242	0.0314	0.0322	5.231	5.407	1.034	1.65
		130	4.728	4.909	0.0478	0.0506	8.325	8.682	1.043	2.17
		120	7.462	7.836	0.0775	0.0787	13.718	14.456	1.054	2.82
		110	12.555	13.344	0.1130	0.1148	23.379	24.911	1.066	4.08
A05	oCl-1	160	2.269	2.320	0.0228	0.0232	3.520	3.622	1.029	1.30
		150	3.171	3.276	0.0307	0.0325	5.304	5.513	1.039	1.96
		140	4.659	4.883	0.0468	0.0494	8.208	8.650	1.054	2.74
		130	7.180	7.666	0.0733	0.0762	13.162	14.120	1.073	3.83
		120	11.773	12.865	0.1054	0.1106	21.860	23.981	1.097	5.95
A06	mCl-1	170	2.292	2.323	0.0217	0.0225	3.575	3.637	1.017	*(b)
		160	3.231	3.295	0.0320	0.0320	5.436	5.564	1.023	1.18
		150	4.779	4.913	0.0475	0.0488	8.501	8.767	1.031	1.64
		140	7.445	7.729	0.0727	0.0768	13.713	14.275	1.041	2.24
		130	12.143	12.767	0.1079	0.1223	22.951	24.181	1.054	3.19
A07	pCl-1	150	-	5.543	-	0.0814	-	9.869	1.000	0
		140	8.639	8.743	0.0818	0.0835	15.906	16.110	1.013	*(b)
		130	13.944	14.173	0.1284	0.1223	26.503	26.955	1.017	1.08

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		120	23.850	24.369	0.1835	0.1938	46.041	47.065	1.022	1.62
		110	42.543	43.655	0.3181	0.3375	82.418	84.598	1.026	2.00
A08	oBr-1	170	2.346	2.379	0.0221	0.0237	3.683	3.749	1.018	*(b)
		160	3.266	3.336	0.0320	0.0338	5.506	5.645	1.025	1.25
		150	4.770	4.918	0.0473	0.0476	8.483	8.777	1.035	1.84
		140	7.301	7.622	0.0739	0.0741	13.457	14.093	1.047	2.55
		130	11.686	12.408	0.1192	0.1095	22.049	23.473	1.065	3.72
A09	mBr-1	170	3.281	3.326	0.0321	0.0322	5.549	5.639	1.016	*(b)
		160	4.815	4.909	0.0465	0.0492	8.592	8.779	1.022	1.16
		150	7.424	7.624	0.0729	0.0776	13.759	14.157	1.029	1.56
		140	11.967	12.403	0.1136	0.1165	22.697	23.560	1.038	2.23
		130	20.191	21.158	0.1584	0.1618	38.824	40.732	1.049	3.55
A10	pBr-1	170	-	3.879	-	0.0411	-	6.621	1.000	0
		160	-	5.798	-	0.0673	-	10.391	1.000	0
		150	-	9.064	-	0.1197	-	16.773	1.000	0
		140	-	14.786	-	0.1938	-	27.823	1.000	0
		130	24.779	25.053	0.1755	0.1992	47.970	48.512	1.011	*(b)
A11	oMe-1	150	-	2.284	-	0.0230	-	3.478	1.000	0
		140	-	3.235	-	0.0345	-	5.306	1.000	0
		130	-	4.828	-	0.0524	-	8.430	1.000	0
		120	-	7.587	-	0.0937	-	13.732	1.000	0
		110	-	12.490	-	0.1369	-	23.252	1.000	0
A12	mMe-1	140	-	3.431	-	0.0517	-	5.714	1.000	0
		130	5.143	5.205	0.0482	0.0505	9.164	9.287	1.013	*(b)
		120	8.154	8.292	0.0771	0.0867	14.988	15.259	1.018	*(b)
		110	13.604	13.906	0.1131	0.1128	25.675	26.267	1.023	1.57
		100	23.857	24.507	0.1805	0.1842	45.505	46.772	1.028	2.10
A13	pMe-1	140	-	3.715	-	0.0418	-	6.242	1.000	0
		130	-	5.602	-	0.0728	-	9.941	1.000	0
		120	-	8.871	-	0.1147	-	16.225	1.000	0
		110	14.441	14.536	0.0999	0.1182	27.371	27.558	1.007	b
		100	25.008	25.307	0.1831	0.1989	47.749	48.331	1.012	*(b)
A14	oCF-1	130	3.742	3.777	0.0345	0.0353	6.395	6.464	1.011	*(b)
		120	5.799	5.880	0.0594	0.0565	10.438	10.598	1.015	*(b)
		110	9.481	9.656	0.0961	0.0964	17.590	17.933	1.020	1.07
		100	16.372	16.754	0.1313	0.1360	30.977	31.723	1.024	1.68

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		90	29.858	30.707	0.2302	0.2310	56.864	58.510	1.029	2.17
A15	mCF-1	150	2.255	2.278	0.0209	0.0211	3.483	3.529	1.013	*(b)
		140	3.275	3.326	0.0322	0.0322	5.485	5.586	1.018	*(b)
		130	5.047	5.152	0.0497	0.0522	8.974	9.182	1.023	1.21
		120	8.225	8.444	0.0814	0.0873	15.223	15.655	1.028	1.53
		110	14.150	14.613	0.1134	0.1184	26.745	27.653	1.034	2.35
A16	pCF-1	140	-	4.154	-	0.0466	-	7.097	1.000	0
		130	-	6.585	-	0.0903	-	11.861	1.000	0
		120	10.840	10.921	0.0741	0.0873	20.381	20.540	1.008	*(b)
		110	19.048	19.306	0.1485	0.1505	36.349	36.855	1.014	1.02
		100	35.118	35.793	0.2615	0.2918	67.456	68.772	1.020	1.44
A17	pEt	160	-	2.706	-	0.0274	-	4.316	1.000	0
		150	-	3.922	-	0.0392	-	6.690	1.000	0
		140	-	5.968	-	0.0573	-	10.634	1.000	0
		130	-	9.546	-	0.0960	-	17.645	1.000	0
		120	-	15.984	-	0.1264	-	30.037	1.000	0
A18	pBu	180	-	2.607	-	0.0262	-	4.132	1.000	0
		170	-	3.725	-	0.0370	-	6.333	1.000	0
		160	-	5.577	-	0.0546	-	9.957	1.000	0
		150	-	8.769	-	0.0882	-	16.194	1.000	0
		140	-	14.447	-	0.1283	-	27.162	1.000	0
A19	ptBu	160	4.360	4.408	0.0398	0.0401	7.651	7.746	1.012	*(b)
		150	6.841	6.955	0.0667	0.0685	12.600	12.827	1.018	*(b)
		140	11.346	11.613	0.1093	0.1052	21.467	21.996	1.025	1.47
		130	19.814	20.438	0.1522	0.1614	38.158	39.391	1.032	2.34
		120	36.306	37.823	0.2766	0.2958	70.469	73.455	1.042	3.12
A20	2	150	-	2.310	-	0.0235	-	3.529	1.000	0
		140	-	3.282	-	0.0343	-	5.398	1.000	0
		130	-	4.913	-	0.0493	-	8.596	1.000	0
		120	-	7.736	-	0.0798	-	14.021	1.000	0
		110	-	12.803	-	0.1111	-	23.860	1.000	0
A21	pF-2	140	4.096	4.142	0.0383	0.0400	7.111	7.202	1.013	*(b)
		130	6.336	6.444	0.0644	0.0644	11.522	11.735	1.019	*(b)
		120	10.297	10.540	0.0906	0.0992	19.310	19.789	1.025	1.51
		110	17.549	18.110	0.1382	0.1393	33.410	34.510	1.033	2.38
		100	31.398	32.705	0.2424	0.2502	60.205	62.752	1.042	3.12

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
A22	pCl-2	180	-	2.261	-	0.0230	-	3.451	1.000	0
		170	-	3.151	-	0.0317	-	5.203	1.000	0
		160	-	4.608	-	0.0467	-	8.053	1.000	0
		150	-	7.056	-	0.0691	-	12.835	1.000	0
		140	-	11.340	-	0.1063	-	21.105	1.000	0
A23	pBr-2	190	-	2.352	-	0.0252	-	3.630	1.000	0
		180	-	3.271	-	0.0332	-	5.426	1.000	0
		170	-	4.766	-	0.0498	-	8.382	1.000	0
		160	-	7.258	-	0.0738	-	13.259	1.000	0
		150	-	11.574	-	0.1127	-	21.694	1.000	0
A24	pMe-2	160	-	2.340	-	0.0271	-	3.597	1.000	0
		150	-	3.399	-	0.0431	-	5.665	1.000	0
		140	-	5.226	-	0.0684	-	9.187	1.000	0
		130	-	8.489	-	0.1149	-	15.580	1.000	0
		120	-	14.516	-	0.1459	-	27.186	1.000	0
A25	oOMe-2	180	-	2.301	-	0.0236	-	3.530	1.000	0
		170	-	3.187	-	0.0315	-	5.274	1.000	0
		160	-	4.636	-	0.0454	-	8.108	1.000	0
		150	-	7.048	-	0.0698	-	12.820	1.000	0
		140	-	11.206	-	0.1087	-	20.844	1.000	0
A26	pCF-2	160	-	2.331	-	0.0255	-	3.580	1.000	0
		150	-	3.284	-	0.0372	-	5.439	1.000	0
		140	-	4.844	-	0.0575	-	8.442	1.000	0
		130	-	7.521	-	0.0895	-	13.689	1.000	0
		120	-	12.229	-	0.1161	-	22.746	1.000	0
A27	iBu	140	-	3.547	-	0.0512	-	5.914	1.000	0
		130	-	5.343	-	0.0760	-	9.436	1.000	0
		120	8.218	8.291	0.0680	0.0775	15.241	15.385	1.009	*(b)
		110	13.611	13.766	0.1088	0.1124	25.741	26.045	1.012	*(b)
		100	23.675	24.002	0.1826	0.1858	45.150	45.788	1.014	1.04
A28	3	140	4.425	4.461	0.0392	0.0407	7.762	7.834	1.009	*(a)
		130	6.833	6.914	0.0651	0.0652	12.477	12.637	1.013	*(b)
		120	11.048	11.217	0.1048	0.1090	20.791	21.124	1.016	*(b)
		110	18.757	19.106	0.1455	0.1519	35.778	36.463	1.019	1.38
		100	33.395	34.141	0.2413	0.2497	64.225	65.682	1.023	1.79
A29	4	180	-	2.220	-	0.0227	-	3.370	1.000	0

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		170	-	3.089	-	0.0296	-	5.081	1.000	0
		160	-	4.508	-	0.0435	-	7.857	1.000	0
		150	-	6.914	-	0.0694	-	12.557	1.000	0
		140	-	11.105	-	0.1120	-	20.647	1.000	0
A30	A	170	2.099	2.121	0.0212	0.0222	3.190	3.234	1.014	*(b)
		160	2.915	2.960	0.0282	0.0296	4.784	4.873	1.019	*(b)
		150	4.264	4.354	0.0416	0.0447	7.460	7.639	1.024	1.23
		140	6.547	6.733	0.0662	0.0702	11.964	12.333	1.031	1.61
		130	10.557	10.951	0.1048	0.1131	19.822	20.600	1.039	2.13
A31	5F-A	170	2.470	2.501	0.0227	0.0247	3.930	3.992	1.016	*(b)
		160	3.536	3.602	0.0341	0.0367	6.016	6.147	1.022	1.10
		150	5.327	5.463	0.0529	0.0552	9.569	9.839	1.028	1.48
		140	8.425	8.714	0.0857	0.0870	15.683	16.255	1.036	1.97
		130	13.968	14.596	0.1316	0.1290	26.550	27.789	1.047	2.84
A32	5Cl-A	190	-	2.746	-	0.0275	-	4.406	1.000	0
		180	-	3.896	-	0.0397	-	6.651	1.000	0
		170	-	5.790	-	0.0579	-	10.398	1.000	0
		160	-	9.016	-	0.0925	-	16.719	1.000	0
		150	-	14.675	-	0.1160	-	27.775	1.000	0
A33	5Br-A	180	-	5.895	-	0.0878	-	10.582	1.000	0
		170	8.929	9.016	0.0818	0.0791	16.822	16.996	1.010	*(b)
		160	14.362	14.536	0.1202	0.1231	27.496	27.841	1.013	*(b)
		150	24.092	24.447	0.1764	0.1961	46.802	47.506	1.015	*(b)
		140	42.259	42.945	0.4129	0.4279	82.681	84.040	1.016	*(b)
A34	5Me-A	160	-	4.003	-	0.0606	-	6.849	1.000	0
		150	5.876	5.942	0.0551	0.0564	10.659	10.790	1.012	*(b)
		140	9.207	9.348	0.0849	0.0943	17.160	17.438	1.016	*(b)
		130	15.056	15.347	0.1379	0.1387	28.696	29.270	1.020	1.24
		120	25.717	26.319	0.2666	0.2767	49.724	50.911	1.024	1.30
A35	5OMe-A	190	-	2.646	-	0.0281	-	4.209	1.000	0
		180	-	3.704	-	0.0420	-	6.277	1.000	0
		170	-	5.409	-	0.0654	-	9.648	1.000	0
		160	-	8.262	-	0.1147	-	15.200	1.000	0
		150	-	13.200	-	0.1059	-	24.882	1.000	0
A36	1ATL	180	2.128	2.171	0.0206	0.0213	3.239	3.325	1.026	1.21
		170	2.931	3.018	0.0284	0.0297	4.850	5.024	1.036	1.76

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		160	4.242	4.418	0.0423	0.0439	7.417	7.766	1.047	2.40
		150	6.451	6.815	0.0626	0.0679	11.800	12.522	1.061	3.28
		140	10.287	11.056	0.0996	0.1044	19.290	20.807	1.079	4.44
A37	2ATL	170	3.732	3.794	0.0363	0.0376	6.449	6.573	1.019	*(b)
		160	5.535	5.654	0.0549	0.0589	9.982	10.218	1.024	1.23
		150	8.590	8.817	0.0854	0.0883	16.044	16.494	1.028	1.54
		140	13.912	14.357	0.1156	0.1276	26.440	27.318	1.033	2.15
		130	23.539	24.428	0.1873	0.1891	45.428	47.181	1.039	2.78
A38	Nap	190	-	3.184	-	0.0350	-	5.268	1.000	0
		180	-	4.525	-	0.0526	-	7.890	1.000	0
		170	-	6.685	-	0.0802	-	12.159	1.000	0
		160	-	10.334	-	0.1208	-	19.627	1.000	0
		150	-	16.677	-	0.1585	-	31.700	1.000	0
A39	pF-6	190	-	3.108	-	0.0298	-	5.118	1.000	0
		180	-	4.436	-	0.0450	-	7.715	1.000	0
		170	-	6.664	-	0.0656	-	12.118	1.000	0
		160	-	10.346	-	0.1012	-	19.286	1.000	0
		150	-	16.845	-	0.1337	-	32.029	1.000	0
A40	pCl-6	210	-	3.184	-	0.0325	-	5.268	1.000	0
		200	-	4.506	-	0.0462	-	7.870	1.000	0
		190	-	6.635	-	0.0680	-	12.061	1.000	0
		180	-	10.195	-	0.0980	-	19.029	1.000	0
		170	-	16.274	-	0.1371	-	31.035	1.000	0
A41	pBr-6	220	-	3.195	-	0.0351	-	5.289	1.000	0
		210	-	4.488	-	0.0479	-	7.835	1.000	0
		200	-	6.579	-	0.0693	-	11.951	1.000	0
		190	-	10.001	-	0.0999	-	18.687	1.000	0
		180	-	15.827	-	0.1589	-	30.094	1.000	0
A42	pMe-6	200	-	2.800	-	0.0282	-	4.512	1.000	0
		190	-	3.906	-	0.0404	-	6.689	1.000	0
		180	-	5.692	-	0.0605	-	10.183	1.000	0
		170	-	8.621	-	0.1005	-	15.970	1.000	0
		160	-	13.588	-	0.1218	-	25.643	1.000	0
A43	pOMe-6	220	-	2.553	-	0.0264	-	4.045	1.000	0
		210	-	3.503	-	0.0355	-	5.902	1.000	0
		200	-	4.984	-	0.0499	-	8.811	1.000	0

No.	Abbr.	temp (°C)	$t_{R,1}$ (min)	$t_{R,2}$ (min)	$W_{h,1}$	$W_{h,2}$	k'_1	k'_2	α	Rs
		190	-	7.379	-	0.0746	-	13.526	1.000	0
		180	-	11.349	-	0.1131	-	21.297	1.000	0

* Incomplete separation was observed but accurate W_h values could not be obtained. Rs was estimated by comparing peaks with reference [43]: where (a) Rs<0.75; (b) Rs=0.75-1.0.

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