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# Separation of stereoisomers of several furan derivatives by capillary gas chromatography–mass spectrometry, supercritical fluid chromatography, and liquid chromatography using chiral stationary phases

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**Abstract**

The direct separation of several stereoisomers (enantiomers and geometrical isomers) of furan derivatives, important intermediates for the synthesis of physiologically active natural products, was achieved using capillary gas chromatography/mass spectrometry with a per-*O*-methyl-β-cyclodextrin, supercritical fluid chromatography and high-performance liquid chromatography with a tris(3,5-dimethylphenylcarbamate) of cellulose or amylose for the chiral stational phases, respectively. The temperature dependence of the peak resolution ($R_s$) and the retention factor ($k$) over the range of 110–130° was studied using crotyl furfuryl ether in gas chromatography. Successive increases in the $R_s$ value and of the difference between the $k$ value of the *E*-isomer and the $k$ value of the *Z*-isomer were observed when the gradient temperature was decreased. The per-*O*-methyl-β-cyclodextrin column was suitable for use with volatile furan ethers whose molecular masses are between 150 and 180. In conclusion, the separation of thermally unstable furan derivatives was accomplished using supercritical fluid chromatography and high-performance liquid chromatography.


*Keywords:* Stereoisomers; Enantiomer separation; Derivatization; Chiral stationary phases; Chiralcel OD; Chiralpak AD; Furans; Cyclodextrins

## 1. Introduction

Chemically synthesized compounds usually have a very high purity but are often not stereochemically 100% pure. These compounds can contain stereoisomers, such as enantiomers, diastereomers and geometrical isomers. The unambiguous determination of stereoisomeric composition is an important analytical task in the synthesis of these compounds. The determination of the enantiomeric excess (*e.e.*) is of particularly great importance.

Because of its high efficiency, sensitivity and speed, gas chromatography (GC) using chiral stationary phases represents a convenient method for the direct separation of enantiomers. Many useful

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chiral stationary phases have been developed, and the direct separation of enantiomers has been accomplished [1,2]. By using a mass spectrometer as a detector, compounds can be identified with ease and certainty. By calculating the area of each peak appearing on an ion chromatograph, the ratio of isomers in the sample can be determined.

Furan derivatives, classified as terpenes, are important not only because of their widespread occurrence in natural products but also because of their role as versatile intermediates in synthetic processes [3–5]. On the basis of their volatile character, these compounds are suitable for GC analysis.

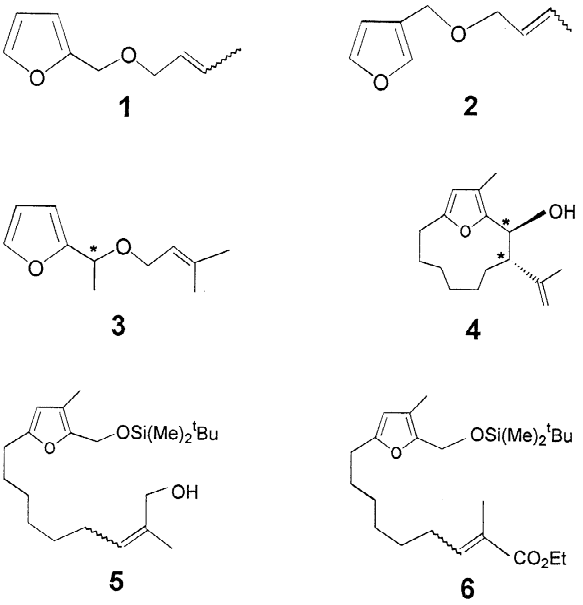
Here, we describe the direct separation of racemates as well as *E*- and *Z*-isomers of furan derivatives using β-cyclodextrin (CD) columns by capillary gas chromatography/mass spectrometry (GC–MS). First, to examine the utility of the columns, separations using per-*O*-methyl-β-CD [6] (β-DEX120) and 2,3-di-*O*-acetyl-6-*O*-*tert*.-butyldimethylsilyl-β-CD (β-DEX225) columns were performed at the same temperature, and their peak resolution ($R_s$) values were compared. Subsequently, *E*- and *Z*-isomers of crotyl furfuryl ether were studied using a β-DEX120 column to examine the temperature dependence of the separation. The relations between the temperature and the $R_s$ and the temperature and the retention factor ($k$) are presented.

Volatility and thermal stability are prerequisites of the GC–MS method, restricting its universal use. Supercritical fluid chromatography (SFC) [7,8] and high-performance liquid chromatography (HPLC) [9,10] represent important complementary methods that can be used for non-volatile or thermally unstable molecules. For furan derivatives, which are not suitable for GC–MS analysis, we used these methods and successfully accomplished their direct separation.

## 2. Experiment

### 2.1. Materials and reagents

The compounds studied (Scheme 1) were a geometrical mixture of crotyl furfuryl ether (**1**), a geometrical mixture of crotyl 3-furylmethyl ether (**2**), racemic 1-(2-furylethyl) prenyl ether (**3**), racemic *anti*-3-isopropenyl-12-methyl-13-oxabicyclo[8.2.1]trideca-1(12),10-dien-2-ol (**4**), a geometrical mixture of 2-*tert*.-butyldimethylsiloxymethyl-5-(9′-hydroxy-8′-methyl-7′-nonenyl)-3-methylfuran (**5**), and a geometrical mixture of 2-*tert*.-butyldimethylsiloxymethyl-5-(8′-ethoxycarbonyl-7′-nonenyl)-3-methylfuran (**6**), which were synthesized according to the usual methods.




Scheme 1. Structures of the compounds.

All solvents, including 2-propanol and *n*-hexane were of HPLC or analytical grade and were purchased from Wako Pure Chemical Industries, Ltd. (Osaka, Japan). The helium used in the GC and the carbon dioxide used in the SFC were both of analytical grade.

### 2.2. Instruments and methods

The $^1$H NMR (270 MHz) spectra for $CDCl_3$ solutions of the compounds were recorded on JNM-LA270 from JEOL (Tokyo, Japan). Chemical shifts were reported using a $\delta$ scale and the internal standard (tetramethylsilane). Mass spectra were measured using a JMS-600W double-focusing (EB geometry) mass spectrometer from JEOL. The sam-

Table 1
Spectral data of the compounds

| Compound | | IR<br>$cm^{-1}$ ($\upsilon$ max) | $^1$H-NMR<br>$\delta$ | MS (EI)<br>$m/z$ |
|---|---|---|---|---|
| **3**<br>(colorless oil) | 1080 | 1.51 (311, d, $J=6.6$ Hz, $CH_3CH$), 1.61 and 1.73 (each 3H, each s, $CH=C(C\underline{H}_3)_2$) 3.8–4.0 (2H, m, $OCH_2$), 4.49 (2H, q, $J=6.6$ Hz, $CH_3C\underline{H}$), 5.33 (1H, br t, $J=7.1$ Hz, =CH), 6.24 (1H, dd, $J=3.3$ and 0.3 Hz, 3-CH), 6.33 (1H, dd, $J=3.3$ and 1.8 Hz, 4-CH), 7.38 (1H, d, $J=1.8$ Hz, 5-CH) | 180 $[M]^+$<br>HRMS: calcd for $C_{11}H_{16}O_2$: 180. 1166<br>Found: 180.1166.<br><br>165 $[M-CH_3]^+$ | |
| **4**<br>(colorless oil) | 3450 | 0.48–1.78 (2H, m, $CH_2$), 1.04–1.38 (4H, m, $2\times CH_2$) 1.50–1.75 (2H, m, $CH_2$), 1.88 (3H, s, $C=CCH_3$), 1.97 (1H, s, 12-$CCH_3$), 1.97–2.06 (2H, m, $CH_2$), 2.11 (111, s, OH), 2.27 (1H, sextet, $J=5.8$ Hz, 3-CH), 2.44–2.66 (211, m, $CH_3$), 4.79 (1H, d, $J=1.5$ Hz, 2-CH), 4.91 (2H, ddd, $J=1.5$ and 5.8 Hz, $CH_2$), 5.83 (1H, s, 11-CH) | 248 $[M1]^+$<br>HRMS: calcd for $C_{16}H_{24}O_2$: 248.1776.<br>Found: 248.1765. | |
| *5*<br>(colorless oil) | 3340 | 0.05 (6H, s, $2\times Si(CH_3)_2$), 0.88 (9H, s, $C(CH_3)_3$), 1.20–1.40 (7H, m, $3\times CH_3$ and OH), 1.58 (2H, q, $J=7.4$ Hz, 2′-$CH_9$), 1.78 (3H, s, 8′-$CCH_3$), 1.96 (3H, s, 3-$CCH_3$), 2.02 (2H, q, $J=6.6$ Hz, 6′-$CH_2$), 2.52 (2H, t, $J=27.3$ Hz, 1′-$CH_2$), 4.10 (211, s, 9′-$CH_2$), 4.54 (2H, s, $CH_2$OTBS), 5.28 (1H, tq, $J=1.2$ and 7.4 Hz, 7′-CH), 5.76 (1H, s, 4-CH) | 380 $[M]^+$<br>HRMS: calcd for $C_{22}H_{40}O_3Si$: 380.2747.<br>Found: 380.2767. | |
| **6**<br>(colorless oil) | 1070<br>and<br>1710 | 0.04 (6H, s, $2\times SiCH_3$), 0.87 (9H, s, $C(CH_3)_3$), 1.28 (3H, t, $J=7.1$ Hz, $CO_2CH_2CH_3$), 1.24–1.48 (6H, m, $3\times CH_9$), 1.59 1.80 (3H, d, $J=1.3$ Hz, 9′-$CH_3$), 1.96 (3H, s, 3-$CCH_3$) 2.14 (2H, q, $J=6.9$ Hz, 6′-$CH_2$), 2.52 (2H, t, $J=7.3$Hz, 1′-$CH_2$), 4.17 (2H, q, $J=7.1$ Hz, $OCH_2CH_3$), 4.54 (2H, s, $CH_2$OTBS), 5.75 (1H, s, 4-H), 6.73 (1H, tq, $J=4.3$ and 6.1 Hz, 7′-H) | 422 $[M]^+$<br>HRMS: calcd for $C_{24}U_{42}O_4Si$: 422.2852.<br>Found: 422.2824. | |

ples were introduced using the direct insertion technique at a probe temperature of 60–300 °C and a rate of 128 °C/min. The resolution for low- and high-resolution MS were set at 500 and 3000, respectively, and perfluorokerosene was used as a standard.

A HP-6890 gas chromatograph system from Agilent (Palo Alto, CA, USA) equipped with a JMS-

BU20 mass spectrometer from JEOL was used. Approximately 10 μl of sample obtained using the head-space procedure for each compound (**1**), (**2**), and (**3**) was injected in the splitless mode. The injection port was set at 220 °C. Helium was used as the carrier gas with a rate of 1 ml/min in the constant flow mode. The mass spectrometer was operated at a filament current of 300 μA, an accelerating voltage of 2.5 kV, an electron energy of 70 V, an ion chamber temperature of 200 °C, and a resolution of 500.

SFC was performed using a SFE/C-201 system from JASCO (Tokyo, Japan) equipped with a MD-910 Multiwavelength detector and a CO-965 Column oven. The gas flow was set at 3 ml/min. Ethanol was added as a modifier to liquid carbon dioxide at a flow-rate of 0.1–0.2 ml/min, and the resulting mixture was pumped into the column and maintained at 45 °C in the column oven. The pressure was controlled by a manual adjustable 880-81 back-pressure regulator. Each compound (**4**), (**5**), and (**6**) was dissolved in ethanol to prepare a 1% solution, and 10 μl of each solution was injected into the column and monitored at a wavelength of 220 nm, enabling the furan-ring to be detected.

HPLC was performed using a HITACHI L-7100 system (Tokyo, Japan) connected to an L-7400 UV absorbance detector operating at 220 nm. The mobile phase was pumped into a column at a flow-rate of 1 ml/min, and the temperature was maintained at 20 °C. Compound (**4**) was dissolved in the same solution as the mobile phase to prepare a 0.05% solution,
5 μl of which was injected.

The ratios of each peak area in the ion chromatogram and the absorption values that were obtained were identical to the ratios of each isomer that were obtained using GC–MS, SFC or HPLC. To clarify these results, the *E*/*Z* ratios were determined by $^{1}$H-NMR spectroscopy and compared.

### 2.3. Columns

β-DEX 120 and β-DEX 225 capillary columns (30 m×0.25 mm internal diameter), coated with 0.25-μm films of per-*O*-methyl-β-CD (20%) and 2,3-di-*O*-acetyl-6-*O*-*tert*.-butyldimethylsilyl-β-CD (25%), respectively, were purchased from Supelco (Bellefonte, PA, USA) and used for the GC experiments.

The enantioselective columns used in the SFC and HPLC were Chiralcel OD [11,12] and Chiralpak AD [13,14] (250×4.6 mm I.D.), respectively, packed with tris(3,5-dimethylphenylcarbamate) derivatives of cellulose and amylose, respectively, and coated on a 10-μm silica-gel support. Both of these columns were purchased from Daicel Chemical Industries, Ltd. (Tokyo, Japan).

## 3. Results and discussion

### 3.1. Preparations of materials

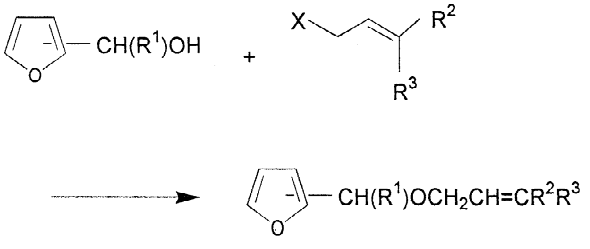
The examined compounds were synthesized in our laboratory. The method used to prepare the 3-methylfuran derivatives (**4**), (**5**), and (**6**) will be published elsewhere. Selected data on the compounds are listed in Table 1.

#### 3.1.1. Crotyl furfuryl ether (*1*) and crotyl 3-furylmethyl ether (*2*)

The synthesis of these compounds was performed using the procedures presented in Scheme 2, as previously described [15,16]. The mixtures of *E*- and *Z*-isomers were studied. The ratio of geometrical mixtures of each furylmethyl ether was determined by $^{1}$H NMR analysis to be *E*:*Z*=85:15, which corresponds to the results obtained with the GC–MS method.

#### 3.1.2. Racemic 1-(2-furylethyl) prenyl ether (*3*)

This compound was synthesized by the reaction of 2-furylethan-1-ol and prenyl chloride. The spectral data are presented in Table 1.




Scheme 2. Preparation of furylmethyl ethers.

*3.1.3. Racemic anti-3-isopropenyl-12-methyl-13-oxabicyclo[8.2.1]trideca-1(12), 10-dien-2-ol (**4**)*

This compound has two stereogenic centers. Here, a mixture of the (2*R**, 3*R**)- and (2*S**, 3*S**)-enantiomers was separated. The spectral data of (2*R**, 3*R**)-enantiomer are shown in Table 1.

*3.1.4. 2-tert.-Butyldimethylsiloxymethyl-5-[9′-hydroxy-8′-methyl-7′-nonenyl]-3-methylfuran (**5**)*

A mixture of 7′*Z*- and 7′*E*-isomers with a ratio of *Z*:*E* = 66:34 was examined. The spectral data of 7′*Z*-isomer are presented in Table 1.

*3.1.5. 2-tert.-Butyldimethylsiloxymethyl-5-[8′-ethoxycarbonyl-7′-nonenyl]-3-methylfuran (**6**)*

A mixture of 7′*E*- and 7′*Z*-isomers with a ratio of *E*:*Z* = 71:29 was studied.

The spectral data of 7′*E*-isomer are listed in Table 1.

*3.2. Separation of E- and Z-isomers of compound (**1**) by capillary GC–MS*

GC–MS was performed in the electron ionization (EI) mode using β-DEX 120 and β-DEX 225 columns for the chiral stationary phases.

*3.2.1. Influence of stationary phases: β-DEX120 vs. β-DEX225 columns*

To examine the use of these columns for the separation of isomers, two types of β-CD columns were examined. A sample (10 μl) obtained by the head-space procedure was injected in the splitless mode, and the column temperature was programmed to increase by 10 °C/min from 110 to 220 °C. Separation was not achieved when the β-DEX225 column was used (Fig. 1). Furthermore, the column temperature at 20 and 1 °C/min were also tried. As a result, the β-DEX120 column was found to be most suitable for the separation of furylmethyl ether isomers. Therefore, further work was performed using only the β-DEX120 column.

*3.2.2. Temperature dependences of peak resolution ($R_s$) and retention factor (k)*

To examine the temperature dependences of the peak resolution ($R_s$) and the retention factor (*k*), five types of gradient temperatures were used in the

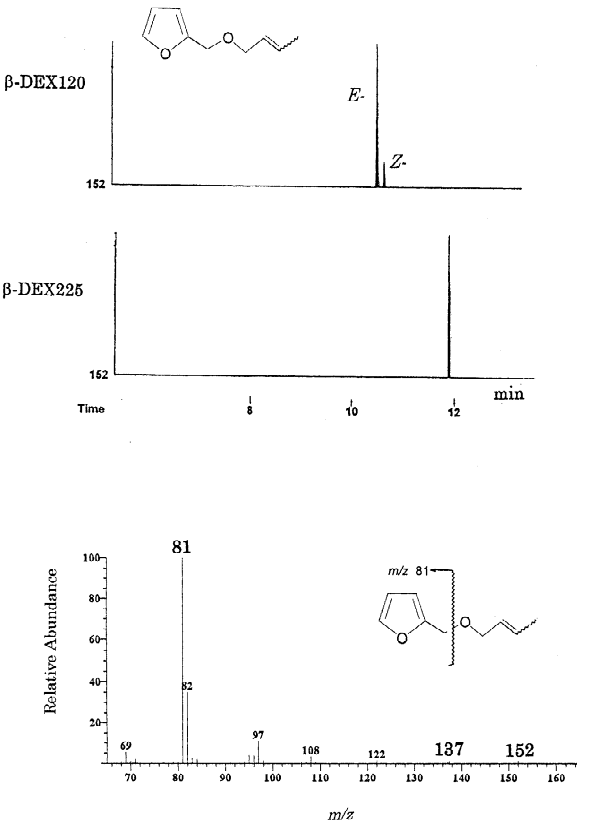



Fig. 1. Separation of *E*- and *Z*-isomers of compound (**1**) by GC–MS at 10 °C/min heating rate with a 30 m×0.25 mm I.D. capillary, 0.25-μm film thickness, (upper) β-DEX 120 column, (middle) β-DEX 225 column, (bottom) MS spectrum of compound (**1**).

β-DEX120 column. A sample (10 μl) obtained by the head-space procedure was injected in the splitless mode, and the column temperature was programmed to increase at 20, 10, 5, 2, and 1 °C/min from 110 to 130 °C. Substantial increases in the $R_s$ values and the difference between the *k*(*E*) and *k*(*Z*) values were observed when the rate of temperature increase was decreased (Fig. 2).

*3.3. Separation of E- and Z-isomers of compound (**2**) by GC–MS*

A sample (10 μl) obtained by the head-space procedure was injected in the splitless mode, and the β-DEX120 column temperature was programmed to

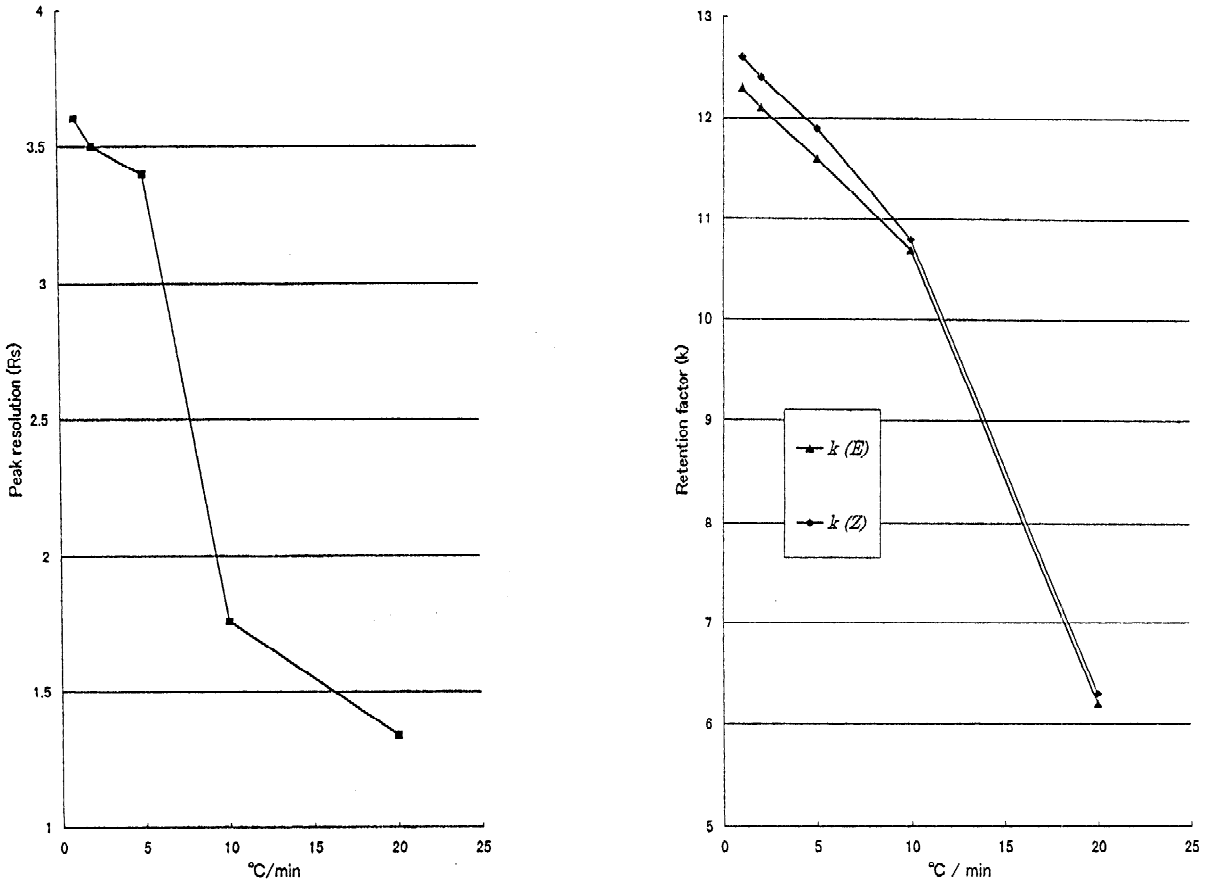



Fig. 2. Dependence of (left) peak resolution ($R_s$) and (right) retention factor ($k$) on gradient temperature between 110 and 130 °C using β-DEX 120 column (30 m×0.25 mm I.D. capillary, 0.25-μm film thickness) with compound (**1**) in GC–MS.

increase from 110 to 130 °C at a rate of 1 °C/min (Fig. 3).

### *3.3.1. Order of elution*

With compounds (**1**) and (**2**), the *E*-isomer was eluted before the *Z*-isomer, indicating that the *Z*-isomer interacts more strongly with per-*O*-methyl-β-CD than the *E*-isomer.

## *3.4. Separation of racemic compound (3) by GC–MS*

Separations were performed using the conditions described above, and the β-DEX120 column enabled a sufficient separation (Fig. 4).

### *3.4.1. Fragment ions in mass spectra*

In the EI-MS, besides the molecular-related ion peak observed at $m/z$ 152 or $m/z$ 180, the base peaks of the fragment ion at $m/z$ 81 or $m/z$ 95 were observed in compounds (**1** and **2**) and (**3**), respectively (Figs. 1, 3, and 4). The base peak at $m/z$ 81 in compound (**1**) was shifted by 14 mass units, which is thought to correspond to the $CH_2$ unit represented by the peak at $m/z$ 95 in compound (**3**), suggesting that a cleavage occurs between the carbon and oxygen bond, as shown in Figs. 1 and 4. Similar results were expected with compound (**2**). To reliably identify the ions at $m/z$ 81 and $m/z$ 95, high-resolution MS was performed. As a result, it was proven that the fragment ion at $m/z$ 81 or $m/z$ 95 were derived from furan derivatives [compound (**1**): $m/z$ 81.03666;

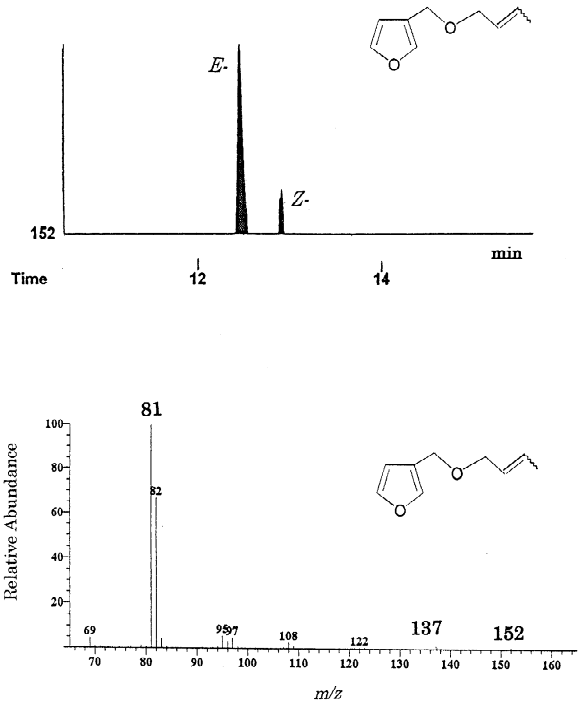



Fig. 3. Separation of *E*- and *Z*-isomers of compound (**2**) by GC–MS at 1 °C/min heating rate between 110 and 130 °C with a 30 m×0.25 mm I.D. capillary, 0.25-μm film thickness, (upper) β-DEX 120 column, (bottom) MS spectrum of compound (**2**).

compound (**2**): *m/z* 81.03479 (Calcd for $C_5H_5O$: 81.03404), compound (**3**): *m/z* 95.04851 (Calcd for $C_6H_7O$: 95.04969].

### *3.5. Separation of racemic anti-compound (4)*

The experiment using GC–MS did not provide a sufficient resolution, but adequate separation was achieved by SFC and HPLC.

#### *3.5.1. SFC*

Ethanol was added as a modifier to liquid carbon dioxide at a rate of 0.2 ml/min, and the resulting mixture was pumped into a column that was maintained at 45 °C in a column oven. The pressure was controlled at 200 kg/cm$^2$. Enantiomer separation of this racemate was accomplished within 5 min using a Chiralcel OD column (Fig. 5).

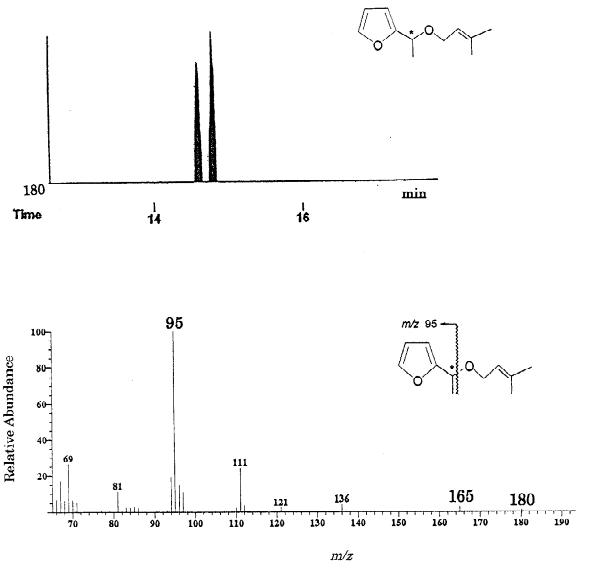



Fig. 4. Separation of enantiomers of compound (**3**) by GC–MS at 1 °C/min between 110 and 130 °C with a 30 m×0.25 mm I.D. capillary, 0.25-μm film thickness, (upper) β-DEX 120 column, (bottom) MS spectrum of compound (**3**).

#### *3.5.2. HPLC*

The mobile phase (2-propanol/*n*-hexane, 5:95 v/v) was pumped into a column at a flow-rate of 1 ml/min and the column was maintained at 20 °C. Five kinds of chiral stationary phases were examined: Chiralcel OD, OB-H (cellulose tribenzoate), OJ (cellulose tris(4-methylbenzoate)), Chiralpak AD, and AS (amylose tris[(*S*)-α-methylbenzylcarbamate]). As the mobile phase, 2-propanol/*n*-hexane (2:98 v/v) was also used. Under the latter condition, the stereoselectivity was reduced when the amount of 2-propanol was decreased because the number of competitive associations of the mobile phase, including isomers with the chiral stationary phases decreased. As a result, the Chiralpak AD column and a mobile phase of 2-propanol/*n*-hexane (5:95 v/v) produced the best resolution, followed by the Chiralcel OD column (Fig. 6). Baseline separations were not achieved using the remaining columns.

#### *3.5.3. SFC vs. HPLC*

Using the Chiralcel OD column, a higher degree of enantiomer separation besides baseline separation was achieved using a supercritical carbon dioxide

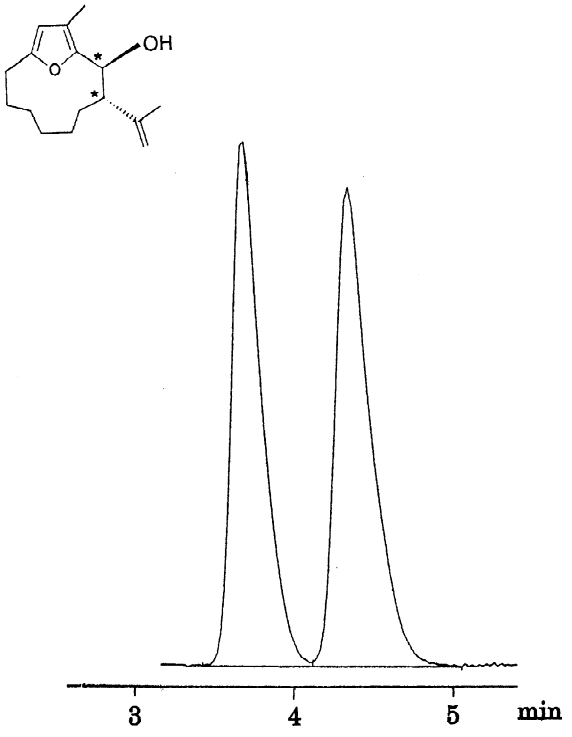



Fig. 5. Separation of enantiomers of *anti*-compound (**4**) by SEC at 45 °C, 3 ml/min carbon dioxide, 200 kg/cm$^2$ pressure, and 0.2 ml/min ethanol as a modifier with a 250×4.6 mm I.D. Chiralcel OD column.

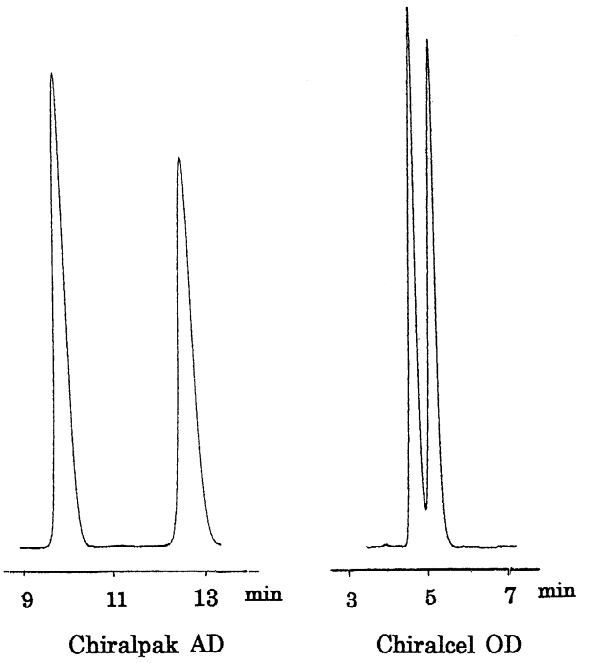



Fig. 6. Separation of enantiomers of anti-compound (**4**) by HPLC with 2-propanol/*n*-hexane, 5:95 *v*/*v* mobile phase at a flow-rate of 1 ml/min. Column: 250×4.6 mm I.D. (left) Chiralpak AD, (right) Chiralcel OD columns.

mobile phase containing ethanol ($R_s = 1.9$), which is more likely to lead to a larger extent of enantioselective association in the chiral stationary phase than 2-propanol/*n*-hexane ($R_s = 1.8$).

### *3.6. Separation of Z- and E-isomers of compound (**5**) by SFC*

Sufficient resolution was not achieved using GC–MS, but separation was accomplished by SFC.

First, ethanol was added as a modifier to liquid carbon dioxide at a rate of 0.2 ml/min and the pressure was controlled at 200 kg/cm$^2$. However, sufficient separation was not obtained. After examining both the rate of modifier addition and the pressure, adequate separation was accomplished using a modifier addition rate of 0.1 ml/min and a pressure of 120 kg/cm$^2$ in the Chiralcel OD column. The resulting mixtures were pumped into a column maintained at 45 °C in a column oven (Fig. 7).

### *3.7. Separation of E- and Z-isomers of compound (**6**) by SFC*

The GC–MS method was not suitable for this compound, and the SFC method was used instead.

The same conditions mentioned above did not provide a sufficient resolution. Consequently, the experiment was performed at a modifier addition rate of 0.15 ml/min and a pressure of 100 kg/cm$^2$ to give the desired separation (Fig. 8).

For 3-methylfuran derivatives, which do not form 12-membered rings, a lower pressure and amount of modifier were used.

## 4. Conclusions

For the direct separations of stereoisomers of

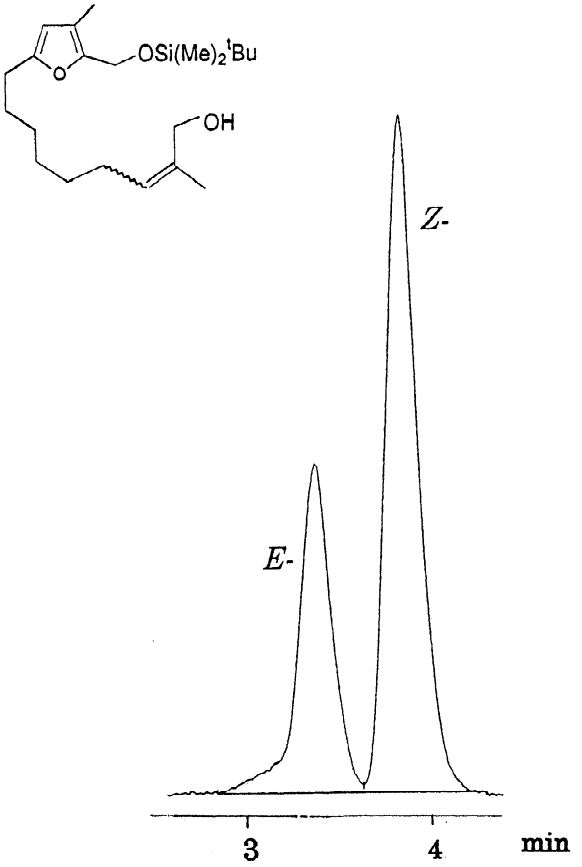



Fig. 7. Separation of *Z*- and *E*-isomers of compound (**5**) by SFC at 45 °C, 3 ml/min carbon dioxide, 120 kg/cm$^2$ pressure, and 0.1 ml/min ethanol as a modifier with a 250×4.6 mm I.D. Chiralcel OD column.

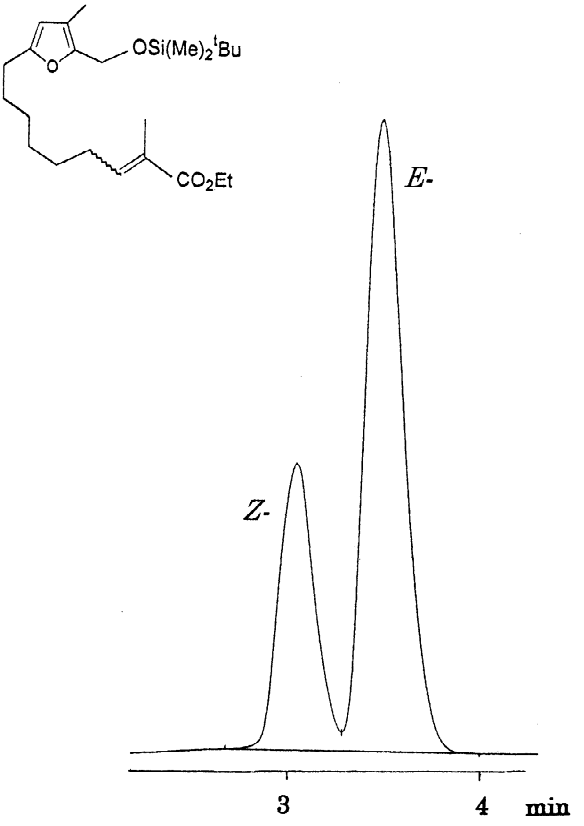



Fig. 8. Separation of *E*- and *Z*-isomers of compound (**6**) by SFC at 45 °C, 3 ml/min carbon dioxide, 100 kg/cm$^2$ pressure, and 0.15 ml/min ethanol as a modifier with a 250×4.6 mm I.D. Chiralcel OD column.

furylmethyl ethers, the GC–MS method is advantageous because of its high-sensitivity, and the small amount of the required sample. Furthermore, the head-space procedure can be used, making the identification of the ions easier and more reliable. β-DEX120 columns are suitable for the separation of ethers whose molecular mass is between 150 and 180. The temperature dependences of the $R_s$ value and the difference in $k$ values between each isomer were observed.

For thermally unstable furan derivatives, SFC and HPLC are applicable. As the chiral stational phases, Chiralcel OD and Chiralpak AD are suitable. Separation by SFC required very short analytical time.

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