Supplementary Materials

Asymmetric Suzuki-Miyaura cross-coupling of aryl chlorides with enhancement of reaction time and catalyst turnover

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Contents

a) General: ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER-SPECTROSPIN-400 with a 5 mm QNP probe at 400 MHz and 100 MHz, respectively. Chemical shift values, reported in parts per million (ppm), were indirectly referenced to external tetramethylsilane employing resonances due to trace monoprotio-solvent as an internal reference. Optical rotations were taken with a JASCO DIP-370 digital polarimeter. Abbreviations are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. ^{31}P NMR spectra were taken with a BRUKER-SPECTROSPIN-400 at 162 MHz. The ^{31}P NMR data are given relative to external 85% H_3PO_4. Elemental analyses were performed with Yanaco MT-5 CHN-Corder. Mass spectra were reported on a Bruker Daltonics esquire-2000T (for ESI), a JEOL JMS-SX102A (for EI), and JEOL GC-mate II (for FAB). Column chromatography was carried out with silica gel, Silica Gel 60N (Kanto Chemical Co.). Thin-layer chromategraphy analyses were performed on Merck silica gel 60 F_{254}. Reactions were performed under an argon atmosphere unless otherwise noted. Materials were purchased from Kanto Chemicals, Co., Inc., and Wako Pure Chemicals, and Tokyo Chemical Industry Co., LTD., and Acros Organics. All the chemical materials were used without further purification.

b) Materials: In the starting materials for the cross-coupling reactions, arylboronic acid compounds were purchased from Tokyo Chemical Industry Co., LTD. and used without further purification. The dehydrated THF, toluene, and potassium fluoride were purchased from Wako Chemicals, Co., Inc., and used without further purification. Other bases were purchased and used without further purification. The compounds of 2-chloro-3-methoxybenzaldehyde and 2-chloro-3-methoxy benzonitrile were purchased from Tokyo Chemical Industry Co., LTD., and Aldrich, respectively. The palladium source $Pd_2(dba)_3{\cdot}CHCl_3$ (dba; dibenzylideneacetone) was purchased from Strem Chemicals.

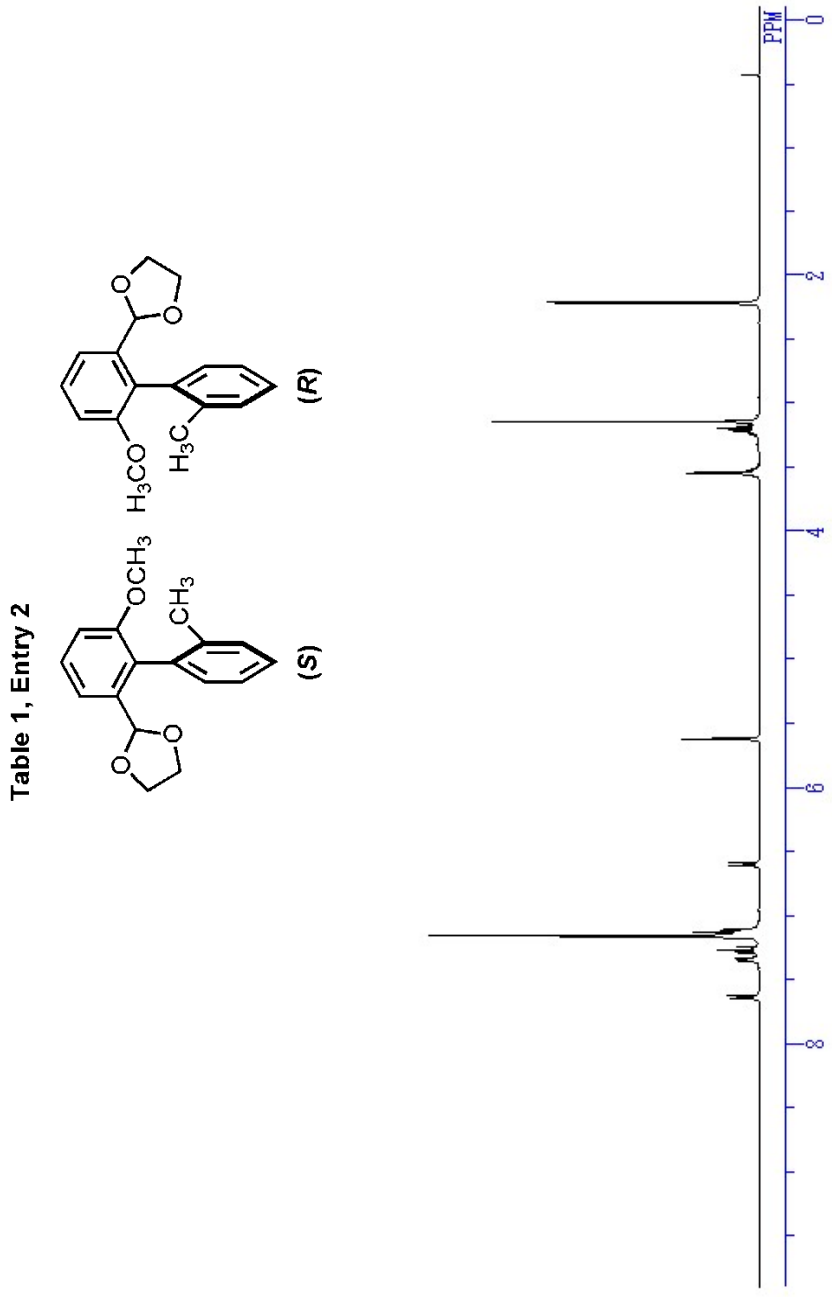
c) **Preparation of 2-(2-chloro-3-methoxyphenyl)-1,3-dioxolane**: To the flask charged with 2-chloro-3-methoxybenzaldehyde (6.80 g, 40 mmol) equipped with Dean-Stark apparatus was added ethylenegrycol (14 mL, 248 mmol) and *para*-toluenesulfonic acid (344 mg, 2 mmol) in distilled benzene (180 mL), and the mixture was refluxed for 16 h, and it was allowed to cool to room temperature. The solvent was thoroughly evaporated, and to the mixture was added EtOAc (100 mL) and water (100 mL), and the resultant solution was extracted with EtOAc (30 mL x 3). Combined organic phase were washed with brine (100 mL) and then dried over Na_2SO_4 and concentrated to give the crude. The crude was purified by silica gel column chromatography (hexane/ CH_2Cl_2 = 2/1) and recrystallized from EtOAc to afford 2-(2-chloro-3-methoxyphenyl)-1,3-dioxolane (6.62 g, 77%) as colorless needles. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 2H), 6.96 (dd, *J* = 7.3, 2.3 Hz, 1H), 6.22 (s, 1H), 4.20 – 4.05 (m, 4H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.2 (s, 1C), 136.9 (s, 1C), 127.4 (d, *J* = 4.0 Hz, 1C), 121.9 (s, 1C), 119.2 (d, *J* = 3.0 Hz, 1C), 119.1 (d, *J* = 2.0 Hz, 1C), 112.7 (s, 1C), 100.8 (d, *J* = 10.0 Hz, 1C), 65.5 (dd, *J* = 10.0, 10.0 Hz, 2C), 56.3 (q, *J* = 8.0 Hz, 1C). MS (EI) *m/z*: 214 (M^+). Anal. Calcd For $C_{10}H_{11}ClO_3$: C, 55.96; H, 5.17. Found: C, 55.75; H, 5.24.

d) **Physical data of phosphonite 2 and 3, for 2:** To a solution of **6** (682 mg, 1.0 mmol) in THF (13 mL) at –78 °C was added *n*-BuLi (0.71 mmol, 1.57 M in hexane) dropwise over 5 min and the mixture was stirred for 5 min. PCl_3 (149 mg, 1.1 mmol) was slowly added over 2 min, and the reaction was allowed to warm to room temperature. After stirring for 2 h, the solvent was thoroughly removed *in vacuo*, and to the residue was added THF (10 mL) and (*R*)-(+)-1,1'-bi-2-naphthol (343 mg, 1.2 mmol), and then Et_3N (212 mg, 2.1 mmol). After stirring for 10 h at ambient temperature, all the volatiles were evaporated. The mixture was dissolved in benzene (100 mL), and washed with water (50 mL), and brine (50 mL), and dried over Na_2SO_4. Purification by silica gel column chromatography gave a desired molecule. Date of **2** is as follows: Yield 43% as a white solid material; $[\alpha]^{25}_D$ + 158.5 (c 1.04, C_6H_6). ^{1}H NMR (400 MHz, C_6D_6) δ 7.78-7.67 (m, 4H), 7.62-7.48 (m, 5H), 7.32-6.93 (m, 15H), 6.87-6.62 (m, 11H), 6.32 (ddd, J = 0.9, 7.6, 7.8 Hz, 1H), 1.94 (s, 3H), 1.80 (s, 3H), 1.79 (s, 3H), 1.76 (s, 3H), 1.75 (s, 3H). ^{13}C NMR (100 MHz, C_6D_6) δ 151.4, 150.21, 150.17, 147.2, 146.8, 142.7, 142.02, 142.01, 141.9, 141.8, 141.4, 139.32, 139.27, 139.1, 139.0, 138.3, 137.9, 135.7, 135.6, 135.3, 135.2, 133.9, 133.7, 133.4, 133.33, 133.29, 132.73, 132.69, 132.53, 132.47, 132.43, 132.39, 132.1, 132.0, 131.4, 130.7, 130.0, 129.5, 129.4, 129.2, 129.0, 128.9, 128.8, 128.6, 128.5, 128.3, 127.7, 127.6, 127.0, 126.8, 126.2, 126.0, 125.5, 125.3, 124.5, 124.4, 123.8, 122.3, 21.5, 21.39, 21.36, 21.35, 21.3. ^{31}P NMR (162 MHz, C_6D_6) δ 178.8. MS (ESI) m/z: 919 ($[M+H]^+$). Anal. Calcd For $C_{67}H_{51}O_2P$: C, 87.56; H, 5.59. Found: C, 87.37; H, 5.65. **for 3:** To a solution of **6** (682 mg, 1.0 mmol) in THF (13 mL) at –78 °C was added *n*-BuLi (1.3 mmol, 1.67 M in hexane) dropwise over 3 min and the mixture was stirred for 10 min. PCl_3 (149 mg, 1.1 mmol) was slowly added over 2 min, and the reaction was allowed to warm to room temperature. After stirring for 2 h, the solvent was thoroughly removed *in vacuo*, and to the residue was added THF (10 mL) and (*R*)-(+)-3,3'-dimethyl-1,1'-bi-2-naphthol (408 mg, 1.3 mmol), and then Et_3N (212 mg, 2.1 mmol). After stirring for 10 h at ambient temperature, all the volatiles were evaporated. The mixture was dissolved in benzene (110 mL), and washed with water (100 mL), and brine (50 mL), and dried over Na_2SO_4. Purification by silica gel column chromatography gave a desired molecule. Date of **3** is as follows: Yield 69% as a white solid material; $[\alpha]^{27}_D$ + 378 (c 1.00, C_6H_6). ^{1}H NMR (400 MHz, C_6D_6) δ 7.69-7.46 (m, 6H), 7.35-6.57 (m, 27H), 6.34 (dd, *J* = 7.4, 7.4 Hz, 1H), 2.81 (s, 3H), 1.90 (s, 3H), 1.79-1.74 (m, 12H), 1.34 (s, 3H). ^{13}C NMR (100 MHz, C_6D_6) δ 150.7, 150.5, 150.4, 147.3, 146.9, 142.9, 141.77, 141.75, 141.44, 141.41, 141.2, 139.6, 139.5, 139.4, 139.2, 139.1, 139.0, 138.9, 135.9, 135.6, 135.5, 135.4, 135.3, 135.4, 133.7, 133.4, 133.2, 133.05, 132.98, 132.9, 132.7, 132.6, 132.4, 132.3, 132.1, 132.0, 131.9, 131.5, 131.1, 130.8, 130.1, 129.9, 129.3, 128.9, 128.0, 126.8, 126.45, 126.38, 126.3, 126.2, 125.8, 125.6, 124.0, 21.7, 21.61, 21.58, 21.5, 18.9 17.9. ^{31}P NMR (162 MHz, C_6D_6) δ 176.4. MS (FAB) *m/z*: 947.77 ($[M+H]^+$). Anal. Calcd For $C_{69}H_{55}O_2P$: C, 87.50; H, 5.85. Found: C, 87.46; H, 5.77.

e) NMR spectra and HPLC charts for Table 1

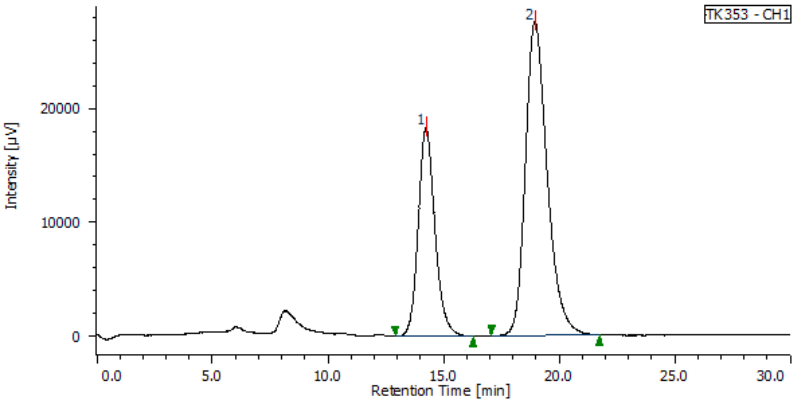
Table 1, entry 2: Purification by silica gel column chromatography (hexane/EtOAc = 9/1) gave a desired biaryl (129 mg, 96%) as white needles. The ee value was determined by HPLC analysis to be 33% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 254 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 14.24 min for (−) with 33.67%, 18.95 min for (+) with 66.33%).

1. NMR spectrum



2. HPLC chart of the biaryl with 33% ee.

クロマトグラム



クロマトグラム情報

ユーザー名	kikuchi
更新日時	2008/12/03 16:07:29
コメント	
HPLC システム名	HPLC
測定日	2008/12/03 15:23:38
注入量	1.00 [μL]
サンプル#	2
プロジェクト名	Guest
取込時間	30.0 [min]
測定シーケンス	20081203
コントロールメソッド	OJH-IPA 75-25 0.5mL
ピークIDテーブル	
検量線テーブル	
追加情報	

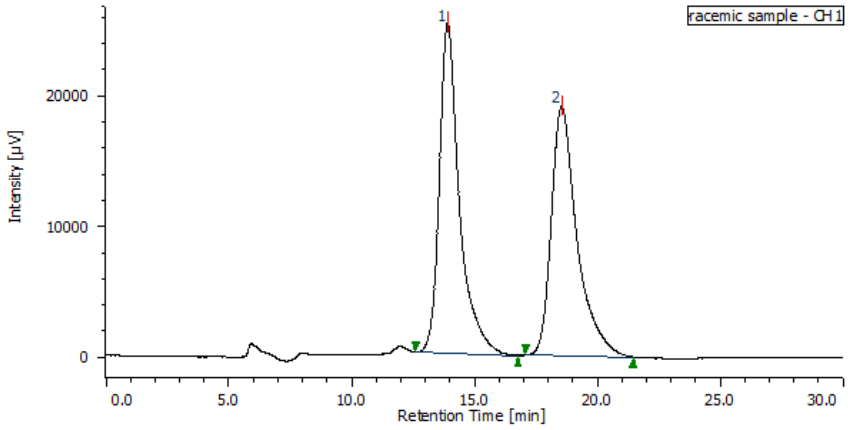
チャンネル情報＋ピーク情報

クロマトグラム名	TK353-CH1
サンプル名	
チャンネル名	UV-2075
データ取り込み間隔	500 [msec]
波形処理メソッド	（マニュアル）
数値計算式	
判定式	

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.242	925020	18337	33.739	39.908	N/A	1993	3.180	1.189	
2	Unknown	1	18.950	1816863	27612	66.261	60.092	N/A	2036	N/A	1.286	

3. HPLC chart of the racemic biaryl.

クロマトグラム



クロマトグラム情報

ユーザー名	kikuchi
更新日時	2008/12/03 16:07:29
コメント	
HPLC システム名	HPLC
測定日	2008/12/03 14:39:36
注入量	1.00 [μL]
サンプル#	1
プロジェクト名	Guest
取込時間	30.0 [min]
測定シーケンス	20081203
コントロールメソッド	OJH-IPA 75-25 0.5mL
ピークIDテーブル	
検量線テーブル	
追加情報	

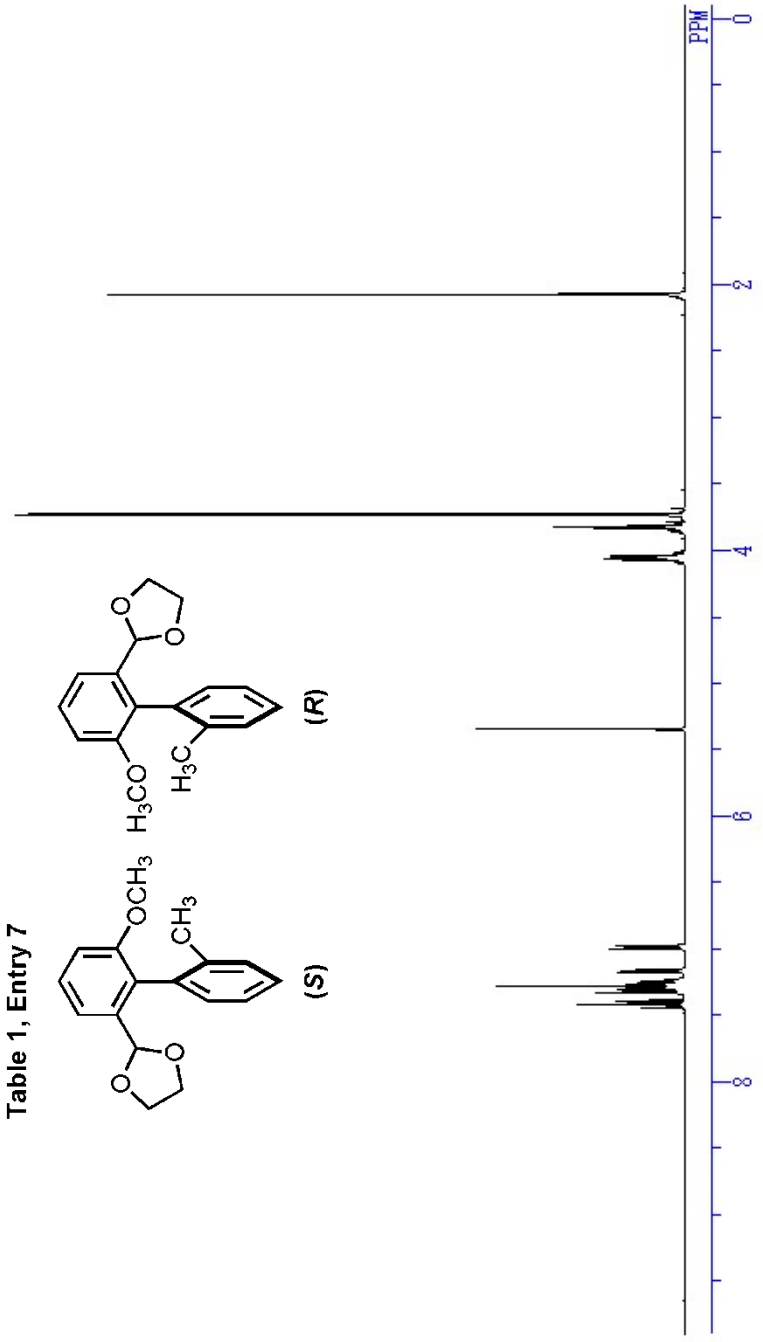
チャンネル情報＋ピーク情報

クロマトグラム名	racemic sample-CH1
サンプル名	
チャンネル名	UV-2075
データ取り込み間隔	500 [msec]
波形処理メソッド	（マニュアル）
数値計算式	
判定式	

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.903	1430715	23262	50.705	56.964	N/A	1735	2.993	1.476	
2	Unknown	1	18.542	1390901	19085	49.294	43.035	N/A	1782	N/A	1.332	

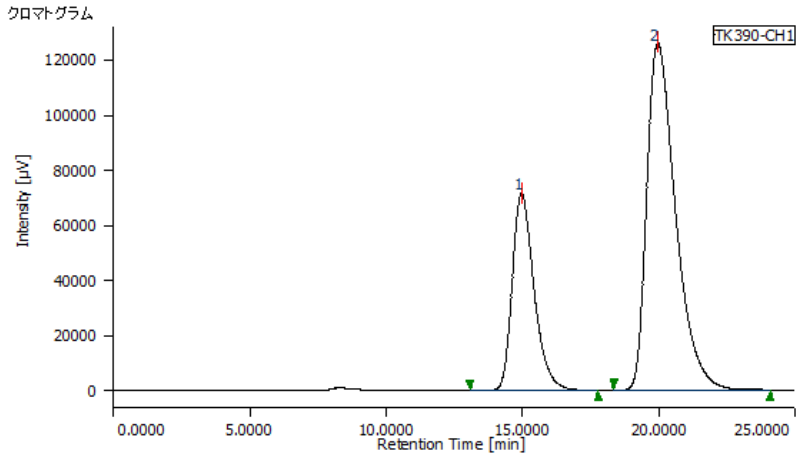
Table 1, entry 7: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (1.33 g, 99%) as white needles. The ee value was determined by HPLC analysis to be 40% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25ºC, retention times: 15.0 min for (−) with 30.0 %, 19.96 min for (+) with 69.98%).

1. NMR spectrum



2. HPLC chart of the biaryl with 40% ee.

20081205 TK390 2009/01/27 18:05:05



クロマトグラム情報

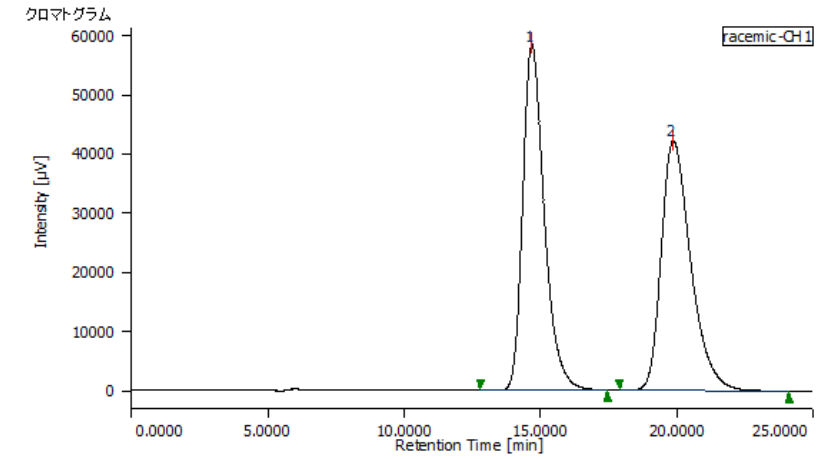
ユーザー名	kamei
更新日時	2009/01/27 17:58:13
コメント	
HPLC システム名	HPLC
測定日	2009/01/27 17:33:13
注入量	2.00 [μL]
サンプル#	2
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.967	3975811	71447	30.017	36.162	N/A	1752	3.017	1.338	
2	Unknown	1	19.938	9269240	126129	69.983	63.838	N/A	1760	N/A	1.350	

3. HPLC chart of a racemic biaryl.

20081205 racemic 2009/01/27 17:35:13



クロマトグラム情報

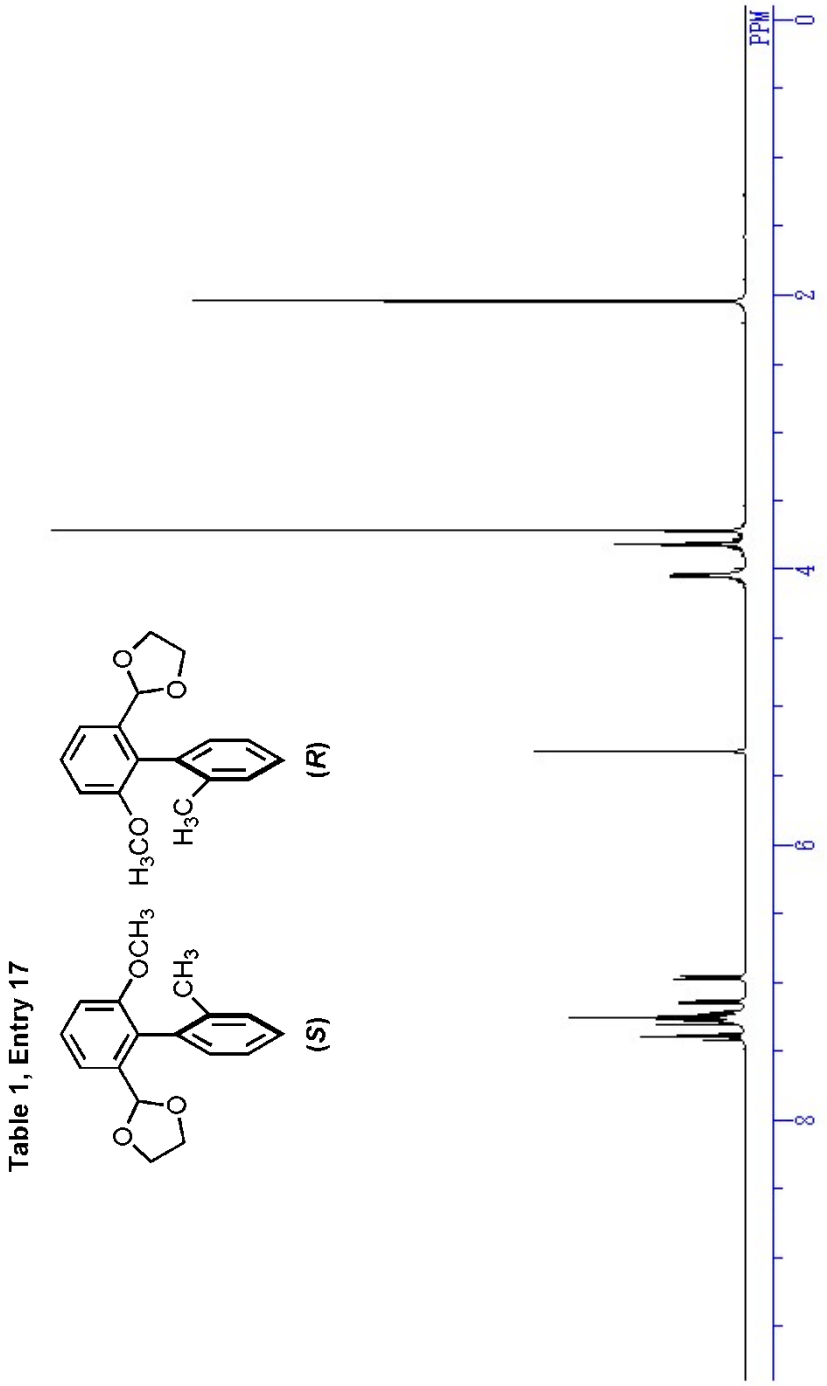
ユーザー名	kamei
更新日時	2009/01/27 17:30:09
コメント	
HPLC システム名	HPLC
測定日	2009/01/27 17:05:09
注入量	2.00 [μL]
サンプル#	1
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.683	3207794	98525	50.230	58.081	N/A	1772	3.126	1.336	
2	Unknown	1	19.867	3178465	42240	49.770	41.919	N/A	1696	N/A	1.403	

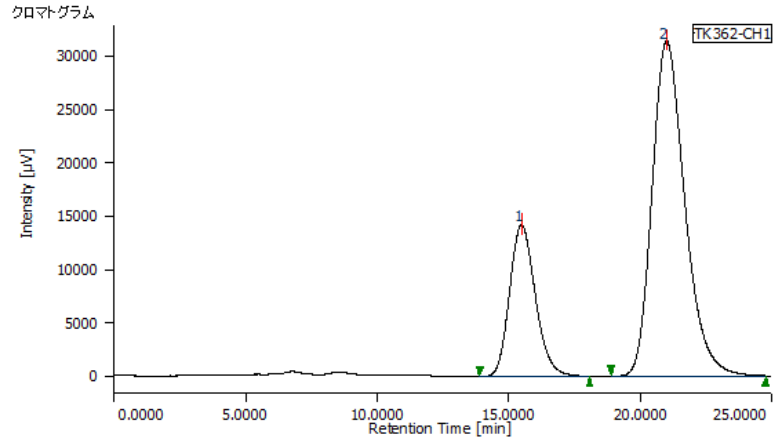
Table 1, entry 6: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (113 mg, 84%) as white needles. The ee was determined by HPLC analysis to be 47% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 15.48 min for (−) with 26.29 %; 20.99 min for (+) with 73.71 %).

1. NMR spectrum



2. HPLC chart of the biaryl with 47% ee.

20081205 TK362 2008/12/16 18:05:34



クロマトグラム情報

ユーザー名	kamei
更新日時	2008/12/16 17:51:53
コメント	
HPLC システム名	HPLC
測定日	2008/12/16 17:23:52
注入量	1.00 [μL]
サンプル#	3
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

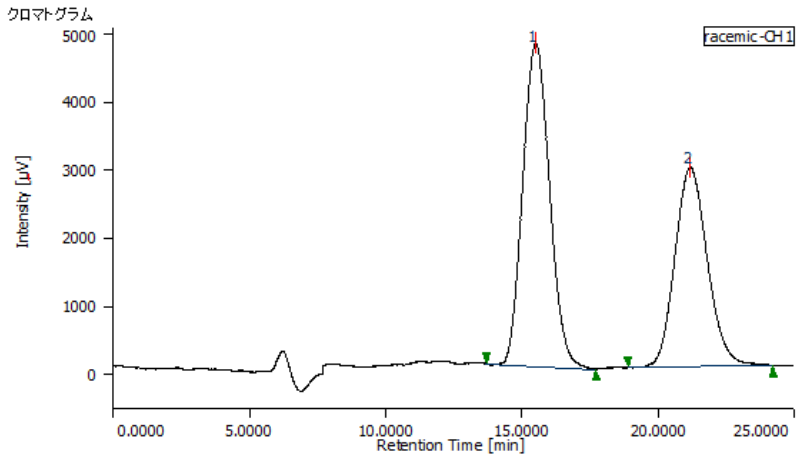
ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.483	968944	14225	26.287	31.157	N/A	1217	2.757	1.190	
2	Unknown	1	20.992	2717144	31431	73.713	68.843	N/A	1418	N/A	1.273	

1 / 1

3. HPLC chart of a racemic biaryl.

20081205 racemic 2008/12/16 18:06:21



クロマトグラム情報

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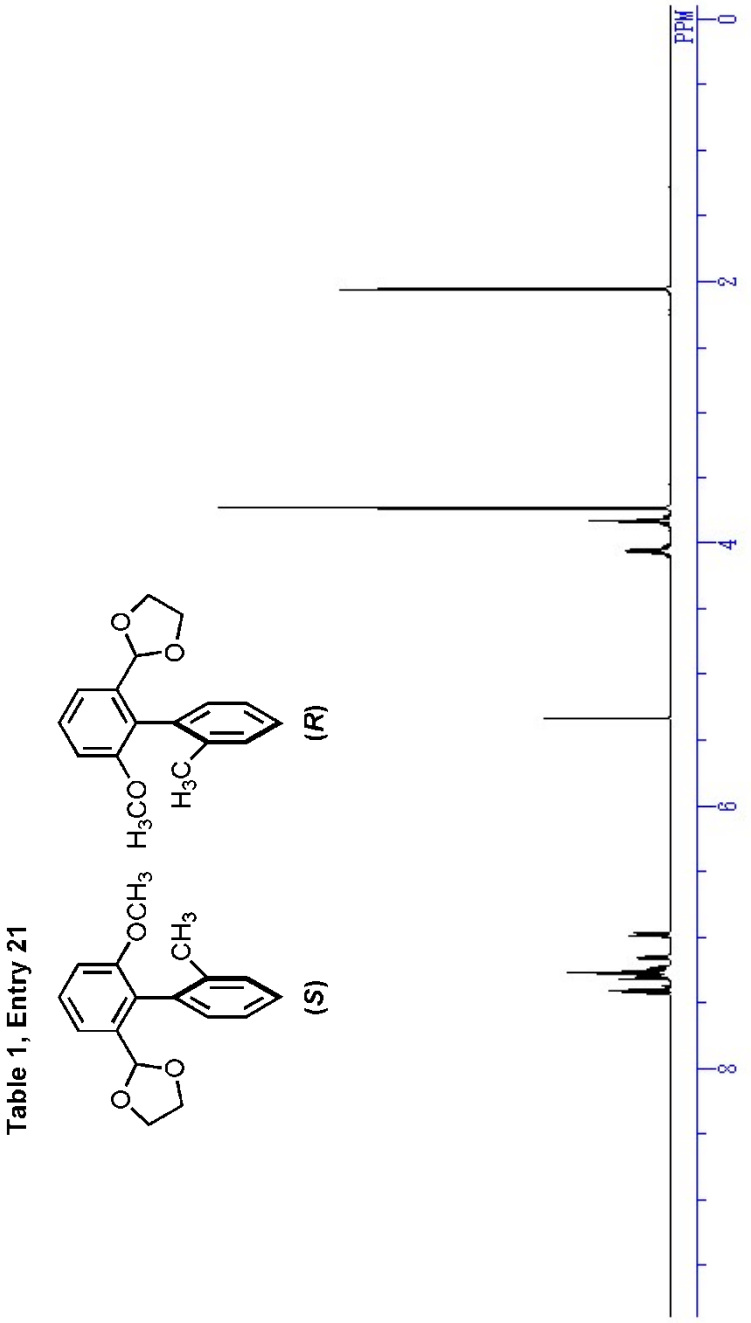
更新日時	2008/12/16 18:03:12
コメント	
HPLC システム名	HPLC
測定日	2008/12/16 16:28:45
注入量	1.00 [μL]
サンプル#	1
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	13.908	324466	4730	57.239	61.781	N/A	1137	2.842	1.108	
2	Unknown	1	21.183	242391	2926	42.761	38.219	N/A	1333	N/A	1.134	

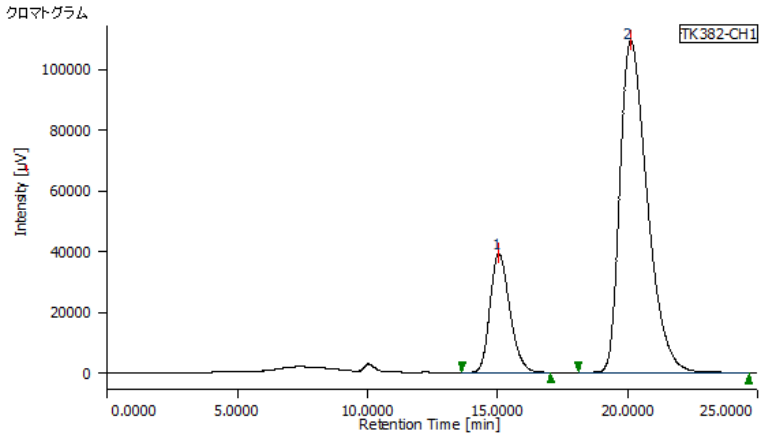
Table 1, entry 9: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (22 mg, 16%) as white needles. The ee was determined by HPLC analysis to be 59% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 15.04 min for (−) with 20.51%, 20.11 min for (+) with 79.49%).

1. NMR spectrum



2. HPLC chart of the biaryl with 59% ee.

20081205 TK382 2009/01/23 18:40:46



クロマトグラム情報

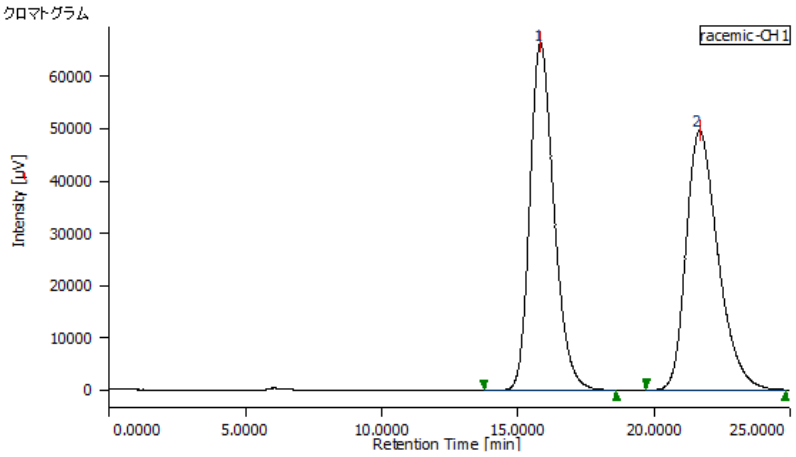
ユーザー名	kamei
更新日時	2009/01/23 18:39:57
コメント	
HPLC システム名	HPLC
測定日	2009/01/23 18:14:58
注入量	2.00 [μL]
サンプル#	4
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.042	2058208	39225	20.511	26.425	N/A	1909	3.098	1.234	
2	Unknown	1	20.117	8015133	109218	79.489	73.575	N/A	1775	N/A	1.448	

3. HPLC chart of a racemic biaryl.

20081205 racemic 2009/01/23 16:01:41



クロマトグラム情報

ユーザー名	kamei
更新日時	2009/01/23 15:58:58
コメント	
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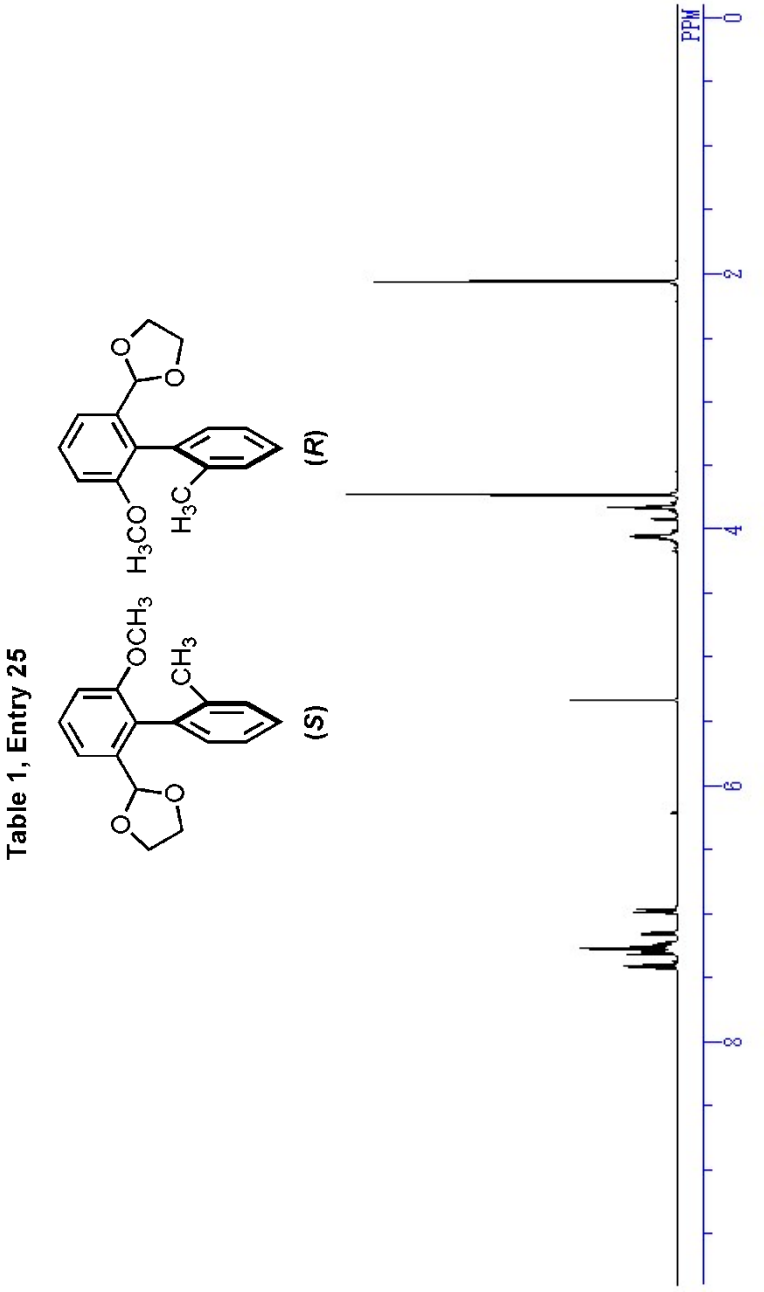
測定日	2009/01/23 15:33:58
注入量	2.00 [μL]
サンプル#	1
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.625	4099920	65413	50.376	57.265	N/A	1574	3.161	1.253	
2	Unknown	1	21.638	4038774	48861	49.624	42.734	N/A	1893	N/A	1.347	

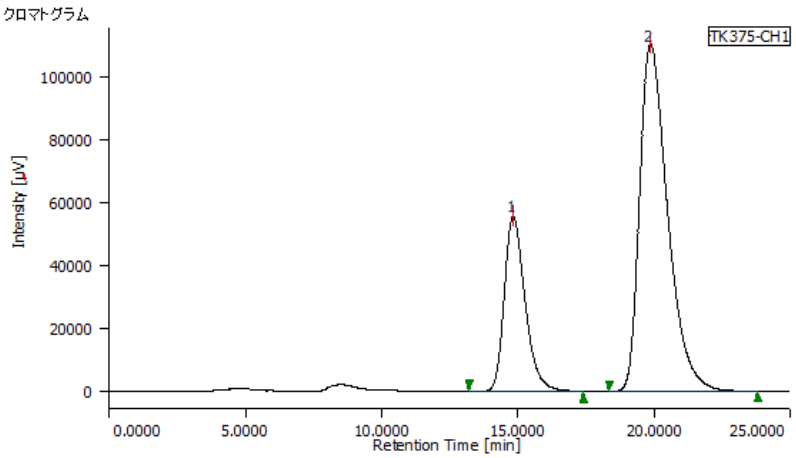
Table 1, entry 11: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (129 mg, 96%) as white needles. The ee value was determined by HPLC analysis to be 46% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 14.83 min for (−) with 27.17%, 19.87 min for (+) with 72.83%).

1. NMR spectrum



2. HPLC chart of the biaryl with 46% ee.

20081205 TK375 2008/01/19 17:47:01



クロマトグラム情報

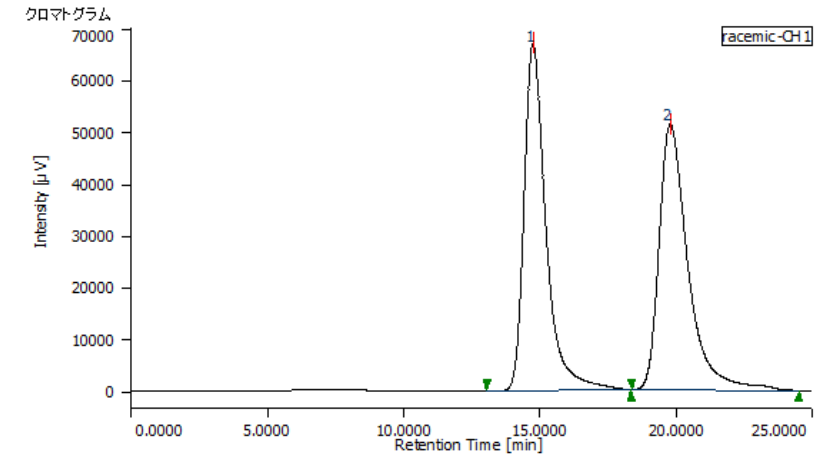
ユーザー名	kamei
更新日時	2008/01/19 16:59:48
コメント	
HPLC システム名	HPLC
測定日	2008/01/19 16:34:48
注入量	2.00 [μL]
サンプル#	3
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.833	2961143	55824	27.169	33.472	N/A	1892	3.142	1.302	
2	Unknown	1	19.867	7937895	110557	72.831	66.528	N/A	1848	N/A	1.498	

3. HPLC chart of a racemic biaryl.

20081205 racemic 2009/01/19 17:51:30



クロマトグラム情報

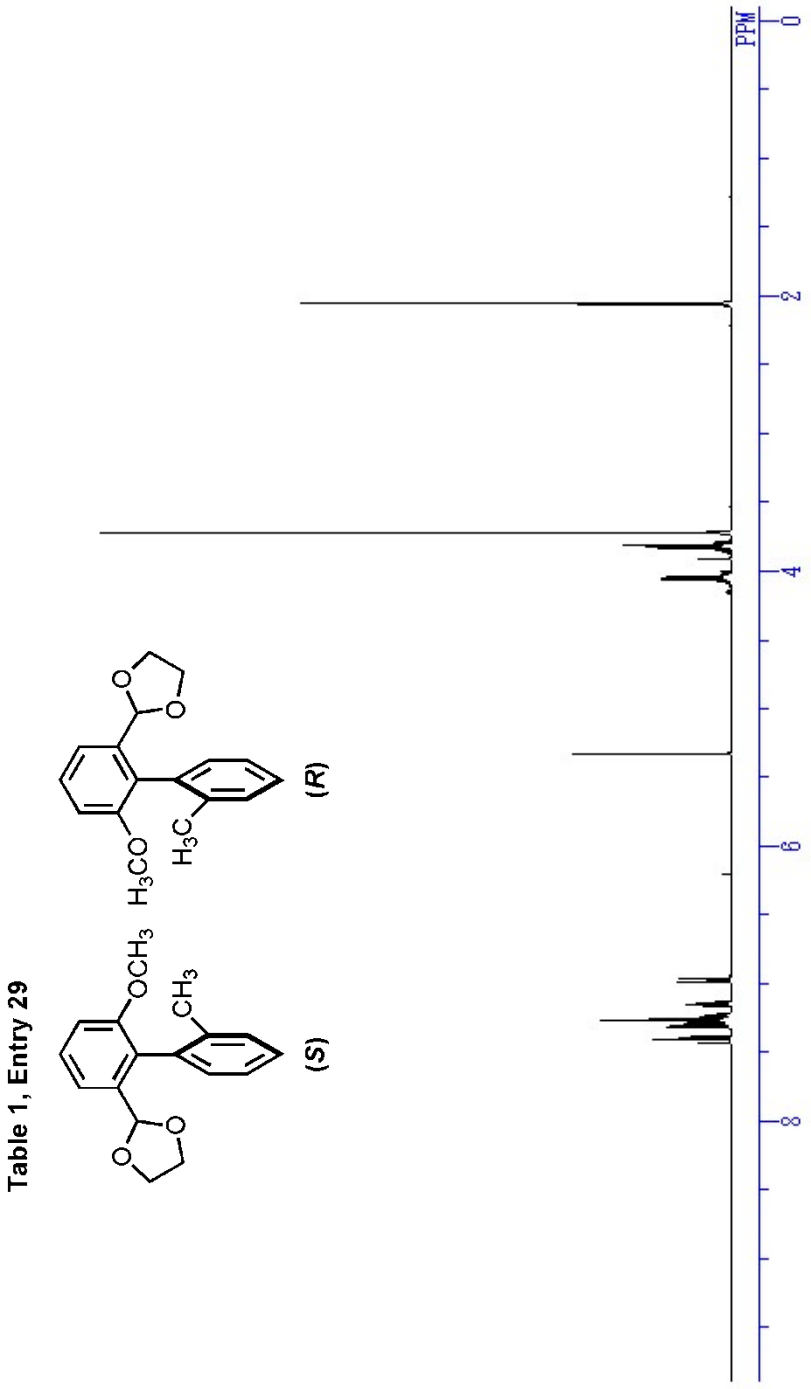
ユーザー名	kamei
更新日時	2009/01/19 16:07:21
コメント	
HPLC システム名	HPLC
測定日	2009/01/19 15:42:21
注入量	2.00 [μL]
サンプル#	1
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.750	3742994	88935	49.663	56.622	N/A	1887	3.199	1.901	
2	Unknown	1	19.783	3793354	51279	50.337	43.378	N/A	1939	N/A	1.992	

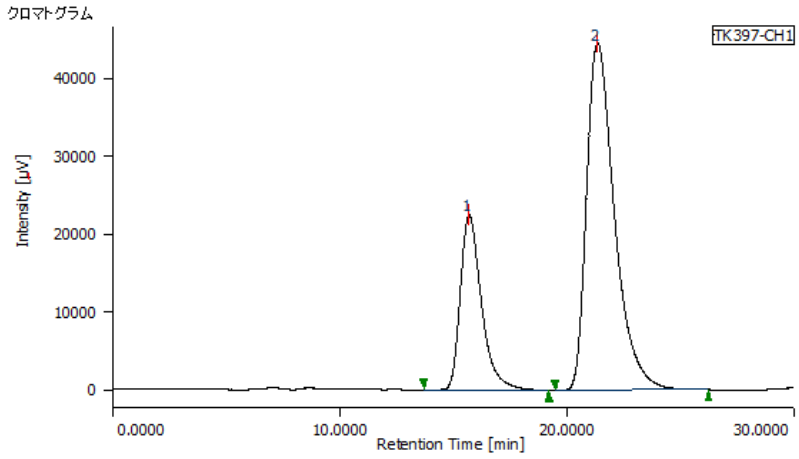
Table 1, entry 14: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (1.24 mg, 92%) as white needles. The ee biaryl was determined by HPLC analysis to be 45% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 15.68 min for (−) with 27.63%, 21.32 min for (+) with 72.37%).

1. NMR spectrum



2. HPLC chart of the biaryl with 45% ee.

20090218 TK397 2009/02/18 15:21:11



クロマトグラム情報

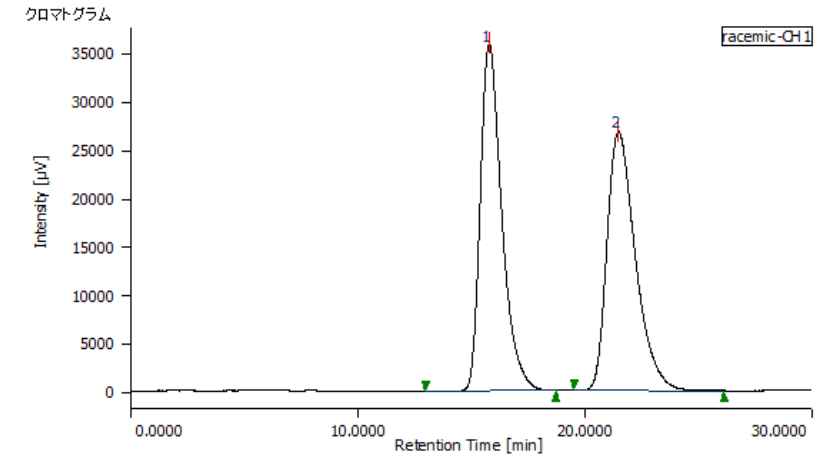
ユーザー名	kamei
更新日時	2009/02/18 15:02:13
コメント	
HPLC システム名	HPLC
測定日	2009/02/18 14:32:13
注入量	2.00 [μL]
サンプル#	3
プロジェクト名	kamei
取込時間	30.0 [min]
測定シーケンス	20090218
コントロールメソッド	H-HPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.675	1456169	22428	27.629	33.473	N/A	1471	2.974	1.450	
2	Unknown	1	21.325	3814191	44576	72.371	66.527	N/A	1337	N/A	1.339	

3. HPLC chart of a racemic biaryl.

20090218 racemic 2009/02/18 16:27:43



クロマトグラム情報

ユーザー名	kamei
更新日時	2009/02/18 16:22:06
コメント	
HPLC システム名	HPLC
測定日	2009/02/18 15:52:07
注入量	2.00 [μL]
サンプル#	1
プロジェクト名	kamei
取込時間	30.0 [min]
測定シーケンス	20090218
コントロールメソッド	H-IPA 75-25 05mL
ピークIDテーブル	
検量線テーブル	
追加情報	

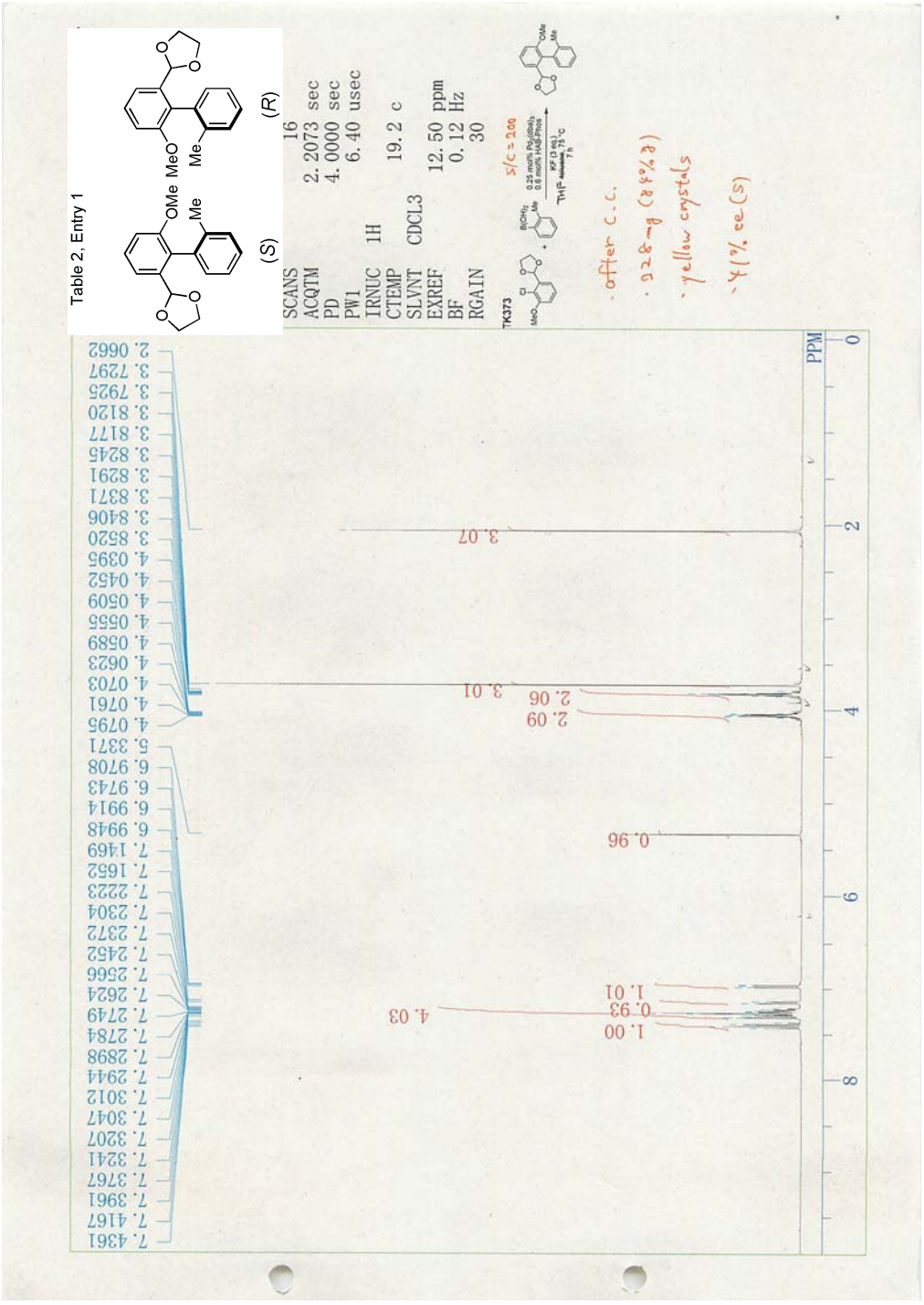
ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV*sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	15.767	2350869	33874	50.362	57.252	N/A	1415	2.918	1.426	
2	Unknown	1	21.456	2328900	26785	49.638	42.748	N/A	1475	N/A	1.437	

NMR spectra and HPLC charts for Table 2

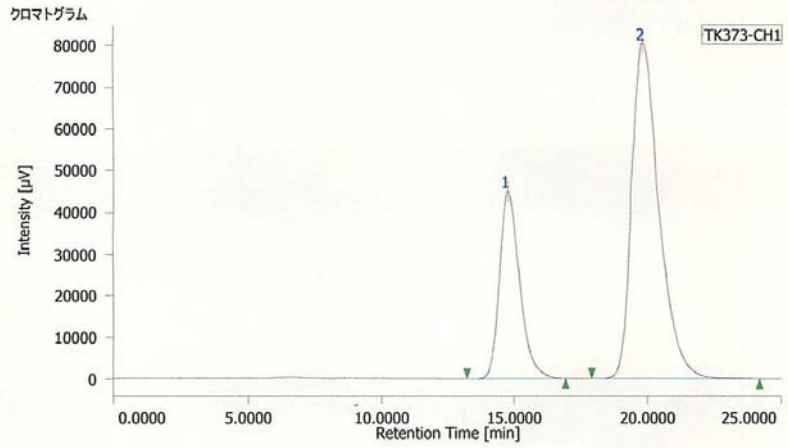
Table 2, entry 1: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (228 mg, 84%) as white needles. The ee value was determined by HPLC analysis to be 41% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times, 14.77 min for (−) with 29.26%, 19.80 min for (+) with 70.74%).

1. NMR spectrum



2. HPLC chart of the biaryl with 41% ee.

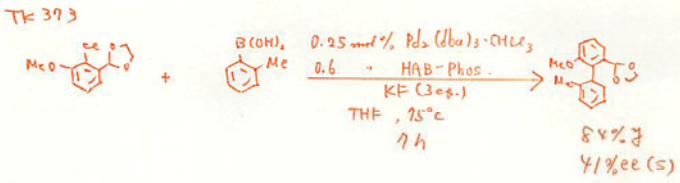
20081205 TK373 2009/01/19 16:36:27



クロマトグラム情報	
ユーザー名	kamei
更新日時	2009/01/19 16:33:33
コメント	
HPLC システム名	HPLC
測定日	2009/01/19 16:08:34
注入量	2.00 [μL]
サンプル#	2
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-IPA 75-25 05mL
ピーク ID テーブル	
検量線テーブル	UV: 270 nm
追加情報	

ピーク情報

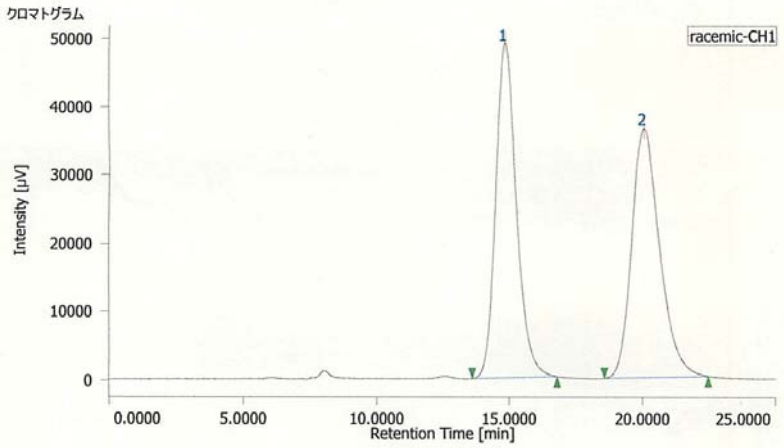
#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.767	2367959	44951	29.259	35.758	N/A	1919	3.193	1.310	
2	Unknown	1	19.800	5725069	80756	70.741	64.242	N/A	1909	N/A	1.448	



1/1

3. HPLC chart of a racemic biaryl.

20081205 racemic 2008/12/05 18:16:37



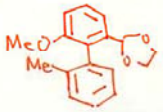
クロマトグラム情報

ユーザー名	kamei
更新日時	2008/12/05 18:14:47
コメント	
HPLC システム名	HPLC
測定日	2008/12/05 17:49:48
注入量	1.00 [μL]
サンプル#	1
プロジェクト名	kamei
取込時間	25.0 [min]
測定シーケンス	20081205
コントロールメソッド	H-IPA 75-25 05mL
ピーク ID テーブル	
検量線テーブル	
追加情報	

ピーク情報

#	ピーク名	CH	tR [min]	面積 [μV·sec]	高さ [μV]	面積%	高さ%	定量値	NTP	分離度	シンメトリー係数	警告
1	Unknown	1	14.833	2662321	49129	50.166	57.407	N/A	1836	3.212	1.253	
2	Unknown	1	20.042	2644687	36451	49.834	42.593	N/A	1844	N/A	1.306	

TK329 - sample



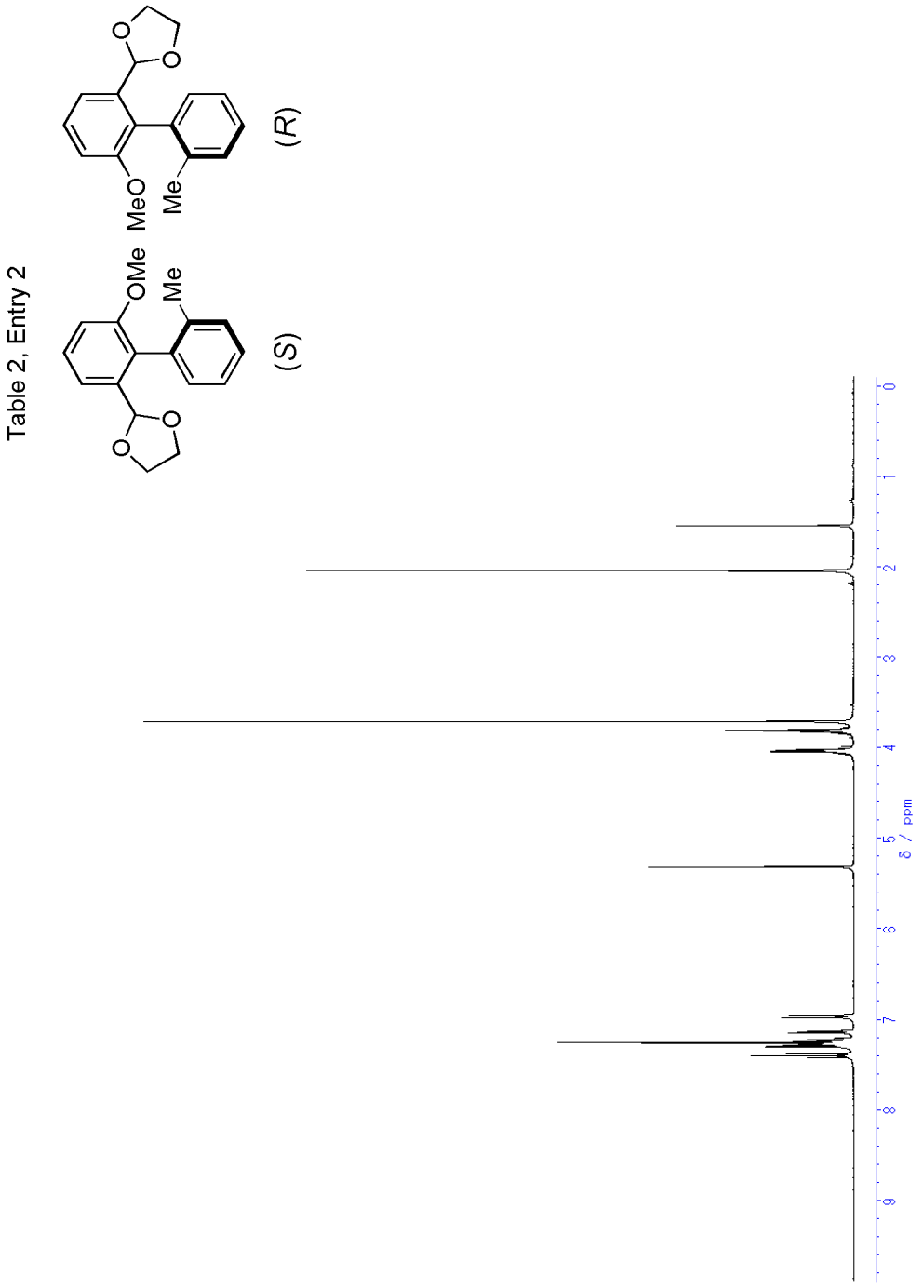
(rac)

after c.c.
recrystals from EtOAc

1/1

Table 2, entry 2: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (213 mg, 79%) as white needles. The ee value was determined by HPLC analysis to be 65% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times, 14.78 min for (−) with 17.54%, 19.24 min for (+) with 82.46%).

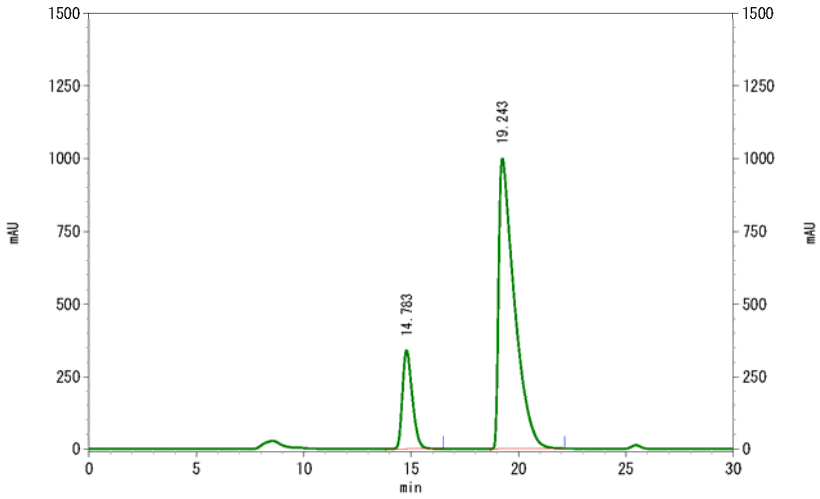
1. NMR spectrum



2. HPLC chart of the biaryl with 65% ee.

sample ID: AS78-01_TM_OJH
date: 2010/02/24 11:10:55
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS78-01_TM_OJH_2010-02-24 11-09-30.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H



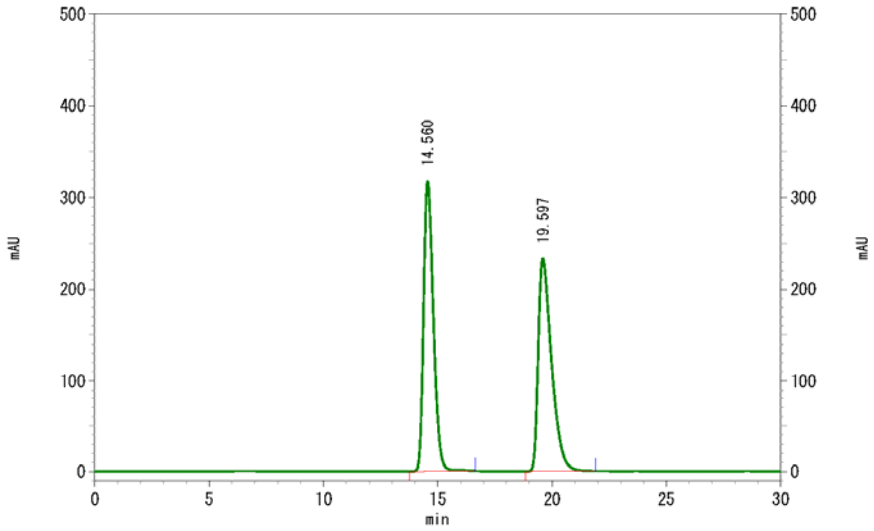
UV結果

保持時間	面積	面積%	開始時間	終了時間
14.783	43349457	17.542	13.82	16.49
19.243	203772562	82.458	18.70	22.13
トータル	247122019	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: TK462-02-racemic_OJH
date: 2009/11/13 19:37:24
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_亀井俊徳¥TK462-02-racemic_OJH_2009-11-13 19-36-59.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25
wave lenght (nm): 270
chiral column: Daicel chiralcel OJ-H

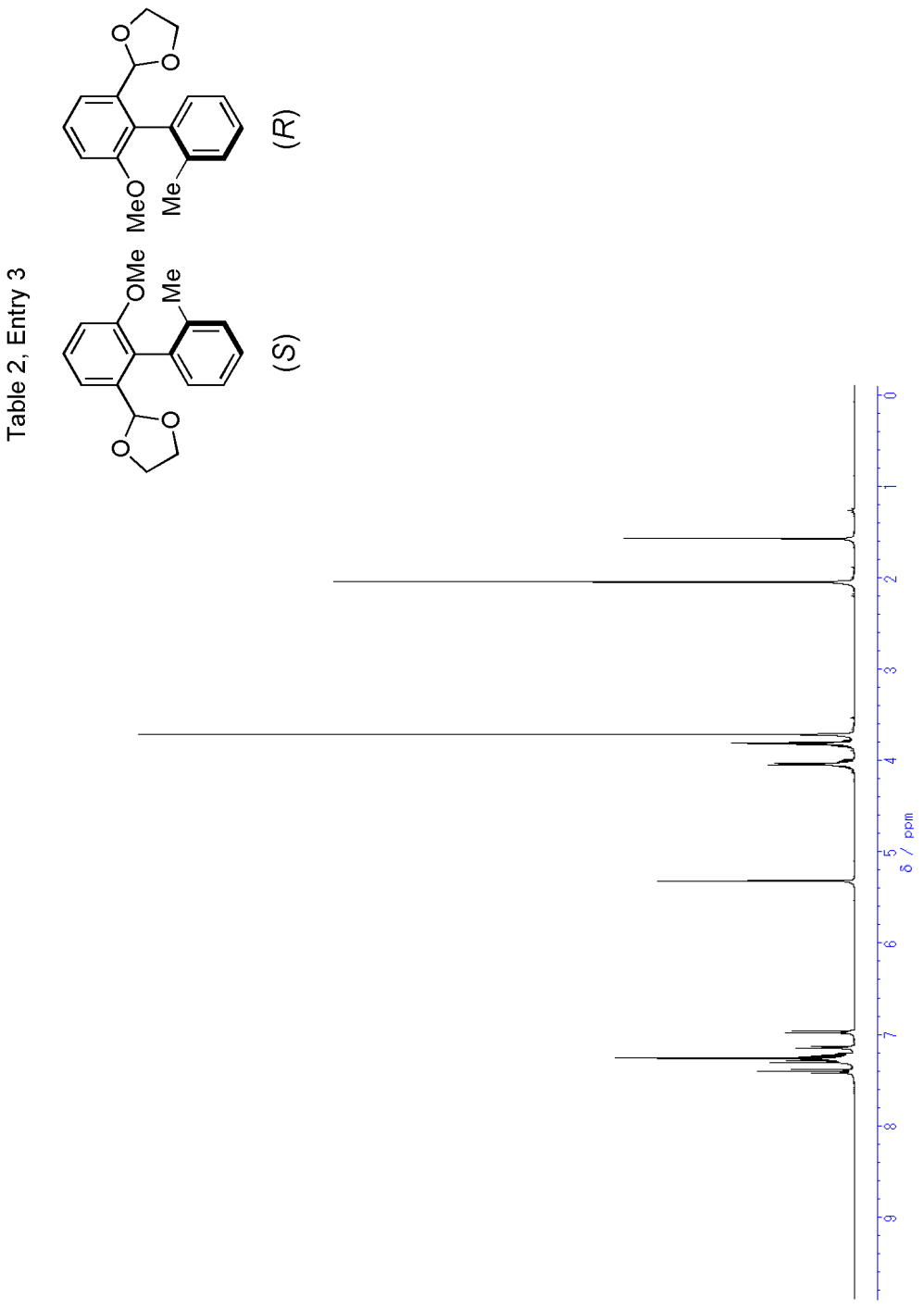


UV結果

保持時間	面積	面積%	開始時間	終了時間
14.560	38061423	50.104	13.76	16.64
19.597	37903034	49.896	18.82	21.90
トータル	75964457	100.000		

Table 2, entry 3: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (243 mg, 90%) as white solid materials. The ee value was determined by HPLC analysis to be 69% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 14.18 min for (−) with 15.46%, 18.68 min for (+) with 84.54%).

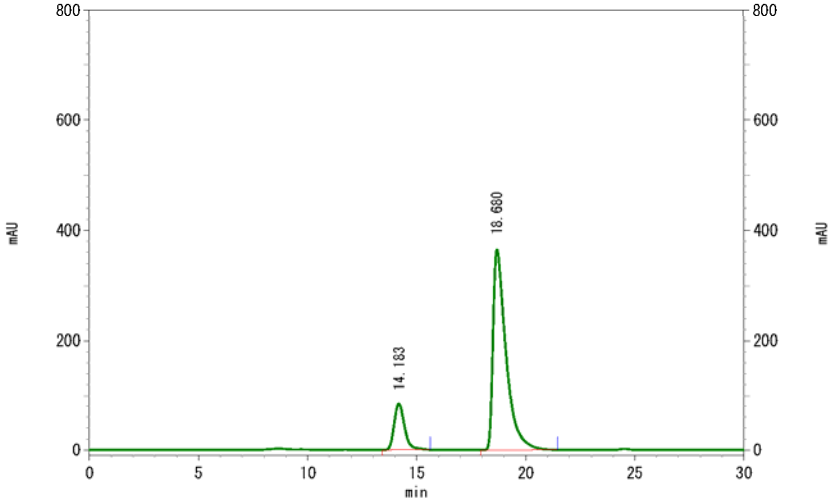
1. NMR spectrum



2. HPLC chart of the biaryl with 69% ee.

sample ID: AS165-02
date: 2010/08/25 18:56:59
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS165-02_2010-08-25 18-56-01.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H



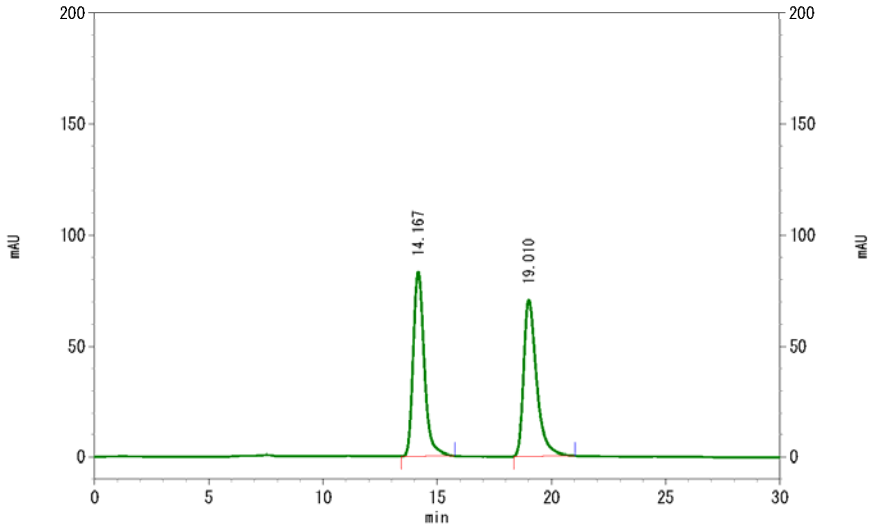
UV結果

保持時間	面積	面積%	開始時間	終了時間
14.183	11080672	15.457	13.44	15.63
18.680	60604799	84.543	17.92	21.44
トータル	71685471	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: AS165test
date: 2010/08/25 15:10:39
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS165test_2010-08-25 15-08-57.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

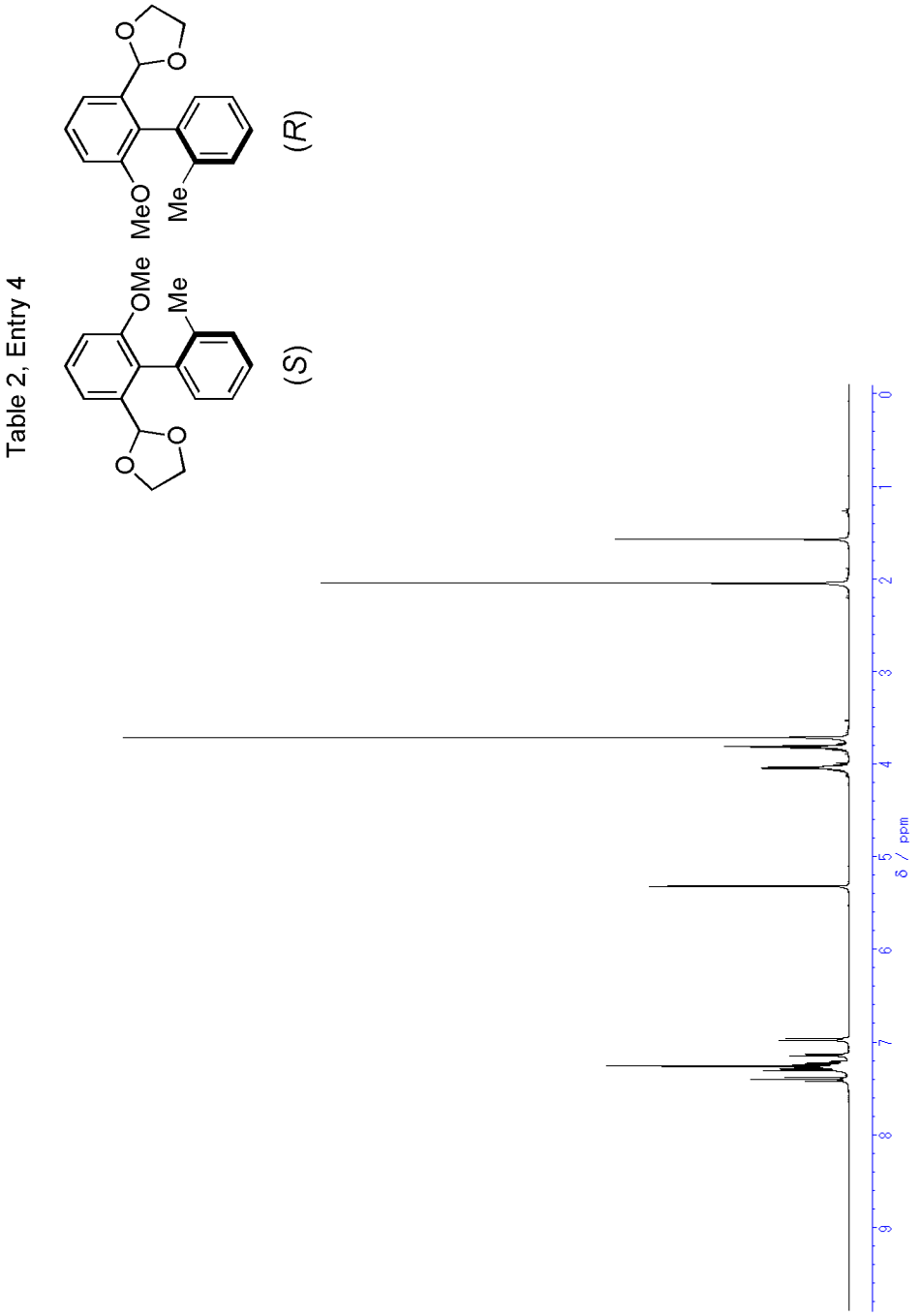


UV結果

保持時間	面積	面積%	開始時間	終了時間
14.167	11244811	50.251	13.44	15.77
19.010	11132485	49.749	18.35	21.02
トータル	22377296	100.000		

Table 2, entry 4: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (252 mg, 94%) as white solid materials. The ee value was determined by HPLC analysis to be 72% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 14.16 min for (−) with 86.02%, 19.18 min for (+) with 13.98%).

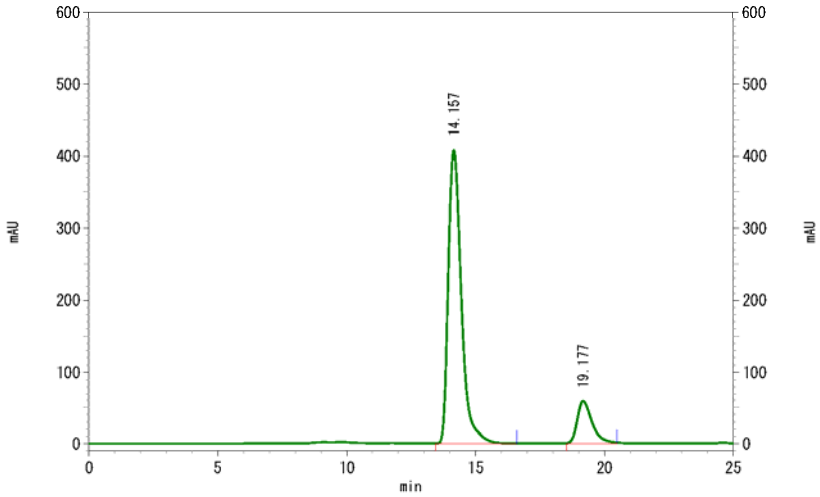
1. NMR spectrum



2. HPLC chart of the biaryl with 72% ee.

sample ID: test4
date: 2010/08/04 22:47:35
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥test4_2010-08-04 22-46-40.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H



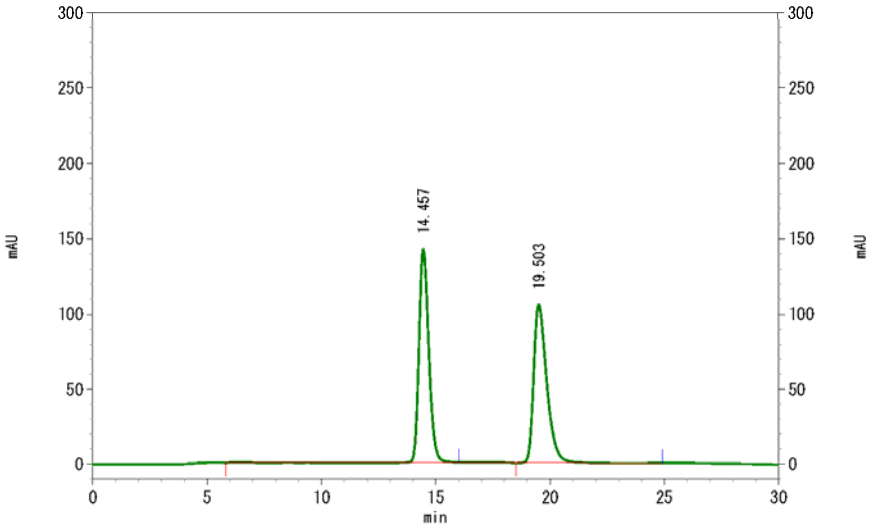
UV結果

保持時間	面積	面積%	開始時間	終了時間
14.157	57402795	86.020	13.44	16.59
19.177	9329104	13.980	18.52	20.47
トータル	66731899	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: TK462-03-OJH-racemic
date: 2009/11/25 11:02:52
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_亀井俊徳¥TK462-03-OJH-racemic_2009-11-25 11-01-00.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OD-H

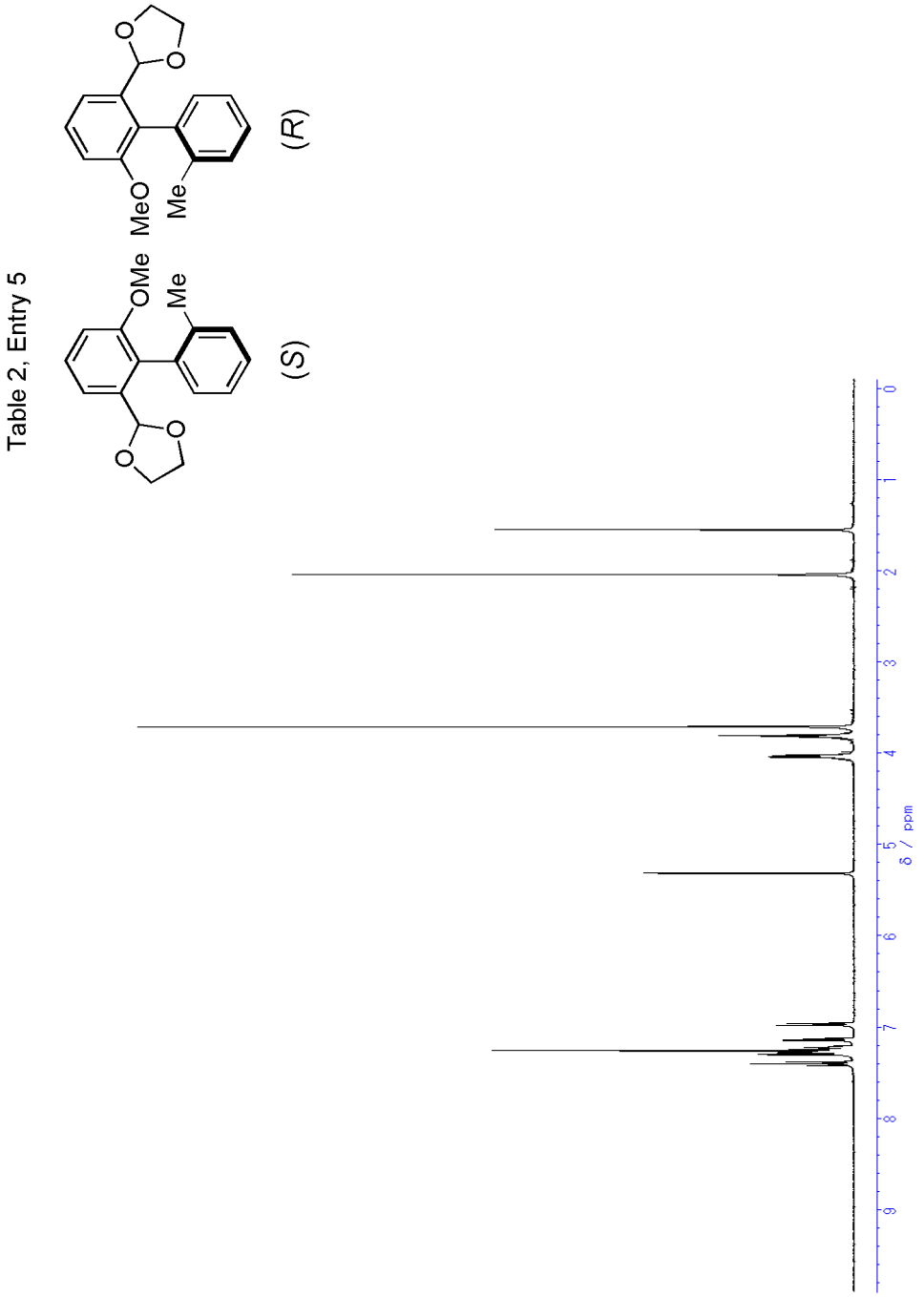


UV結果

	保持時間	面積	面積%	開始時間	終了時間
	14.457	17103672	50.504	5.81	16.03
	19.503	16762439	49.496	18.52	24.90
トータル		33866111	100.000		

Table 2, entry 5: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (251 mg, 93%) as white solid materials. The ee value was determined by HPLC analysis to be 74% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times, 14.2 min for (−) with 13.1%, 18.7 min for (+) with 86.92%).

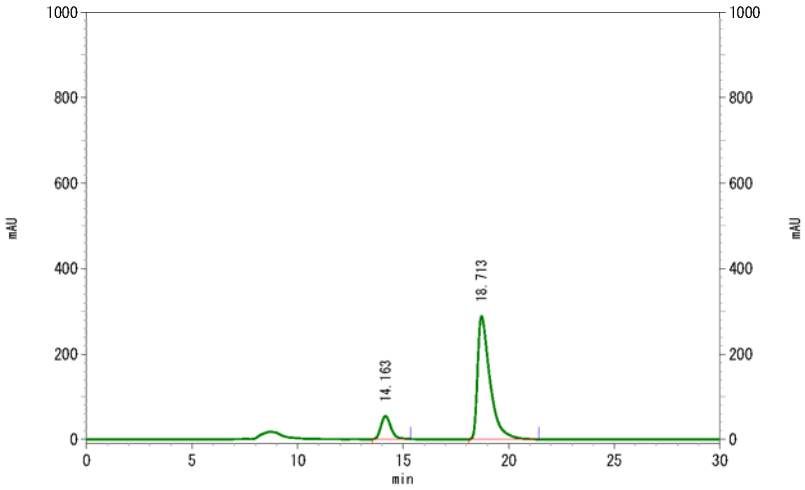
1. NMR spectrum



2. HPLC chart of the biaryl with 74% ee.

sample ID: AS167-01
date: 2010/08/26 16:33:13
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS167-01_2010-08-26 16-31-46.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

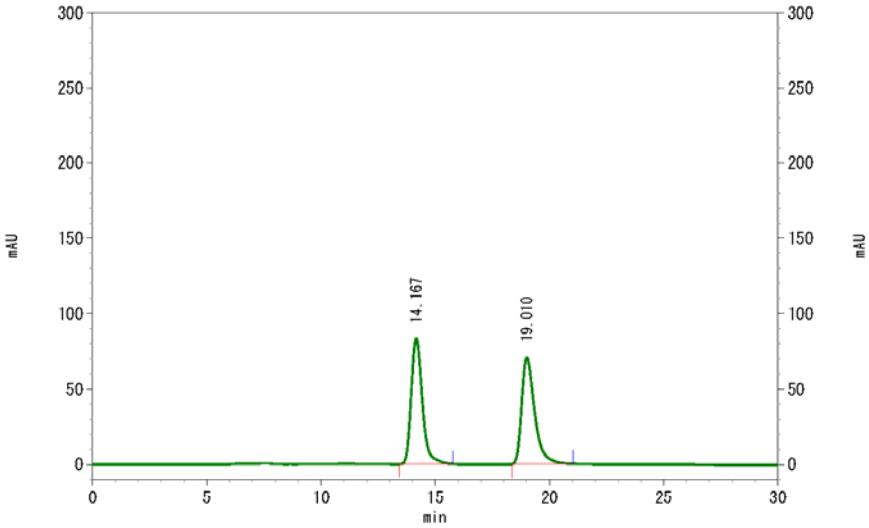


UV結果

	保持時間	面積	面積%	開始時間	終了時間
	14.163	7093315	13.076	13.56	15.37
	18.713	47151768	86.924	18.11	21.43
トータル		54245083	100.000		

3. HPLC chart of a racemic biaryl.

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

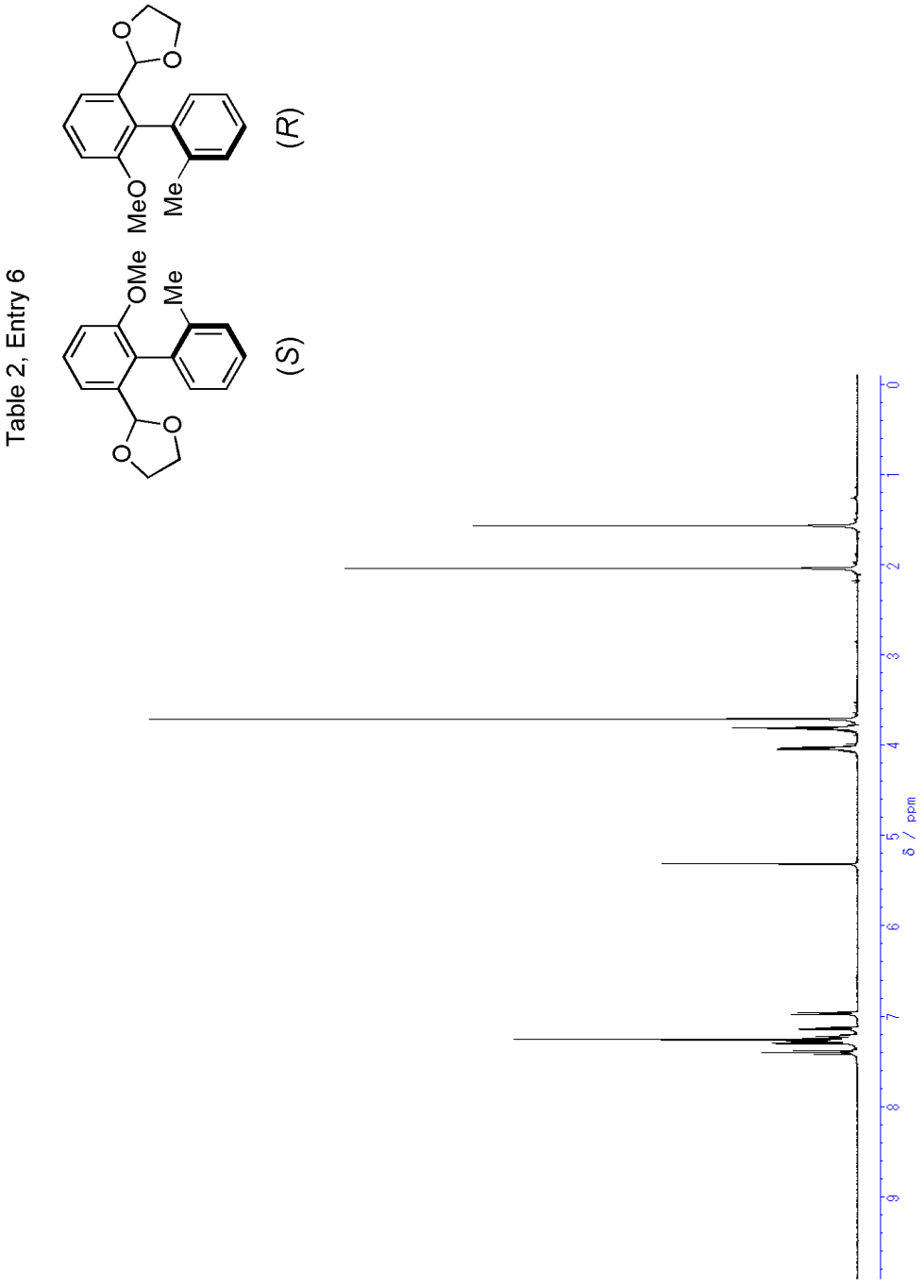


UV結果

保持時間	面積	面積%	開始時間	終了時間
14.167	11244811	50.251	13.44	15.77
19.010	11132485	49.749	18.35	21.02
トータル	22377296	100.000		

Table 2, entry 6: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (246 mg, 91%) as white solid materials. The ee value was determined by HPLC analysis to be 78% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 14.02 min for (−) with 89.07%, 18.98 min for (+) with 10.93%).

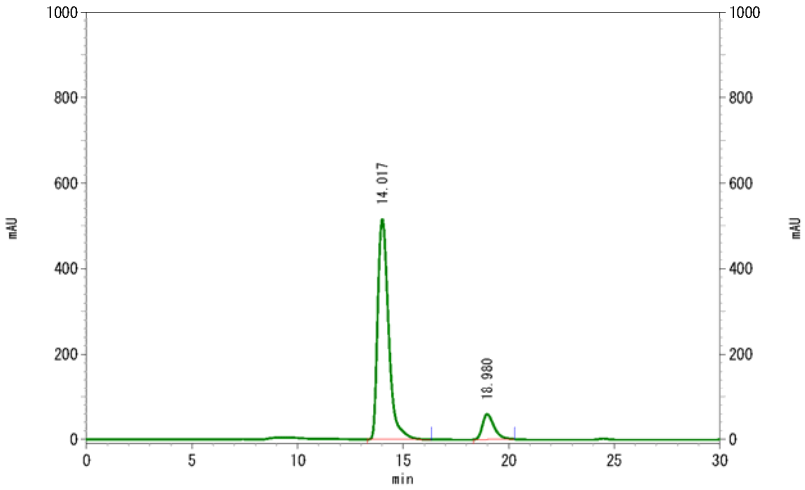
1. NMR spectrum



2. HPLC chart of the biaryl with 78% ee.

sample ID: AS168-01
date: 2010/08/26 15:30:02
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS168-01_2010-08-26 15-29-05.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H



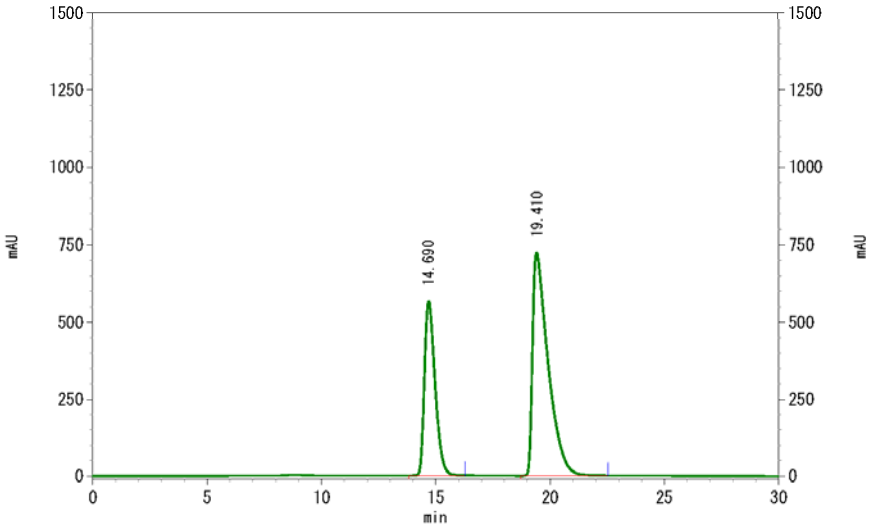
UV結果

保持時間	面積	面積%	開始時間	終了時間
14.017	73832774	89.067	13.32	16.34
18.980	9063423	10.933	18.32	20.29
トータル	82896197	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: AS80_rac_TM_OJH
date: 2010/03/02 11:05:54
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS80_rac_TM_OJH_2010-03-02 11-04-32.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

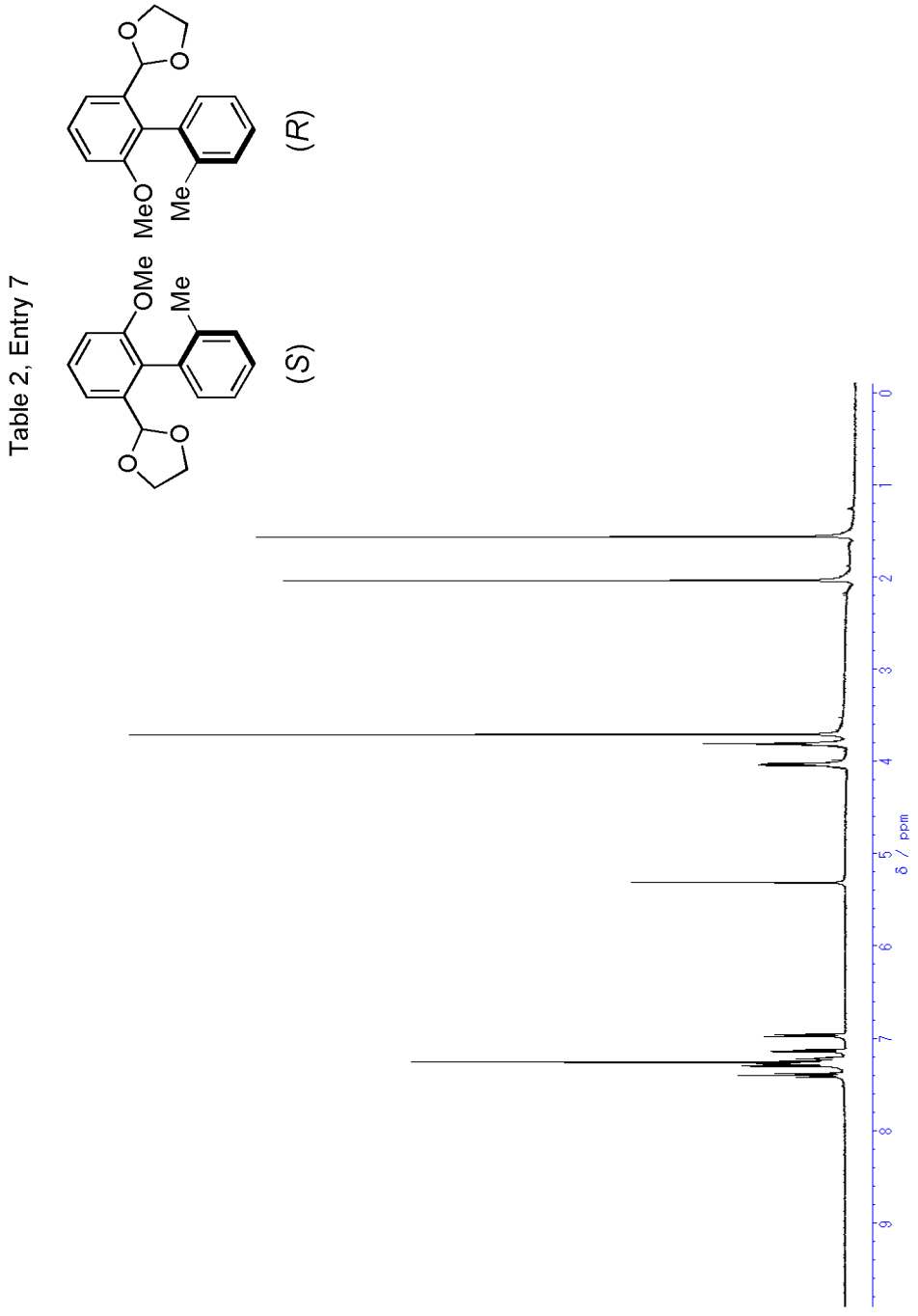


UV結果

	保持時間	面積	面積%	開始時間	終了時間
	14.690	73187259	34.819	13.83	16.29
	19.410	137004581	65.181	18.71	22.55
トータル		210191840	100.000		

Table 2, entry 7: Purification by silica gel column chromatography (hexane/EtOAc/benzene = 8/1/1) gave a desired biaryl (1.0 g, 75%) as white solid materials. The ee value was determined by HPLC analysis to be 76% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 75/25, 270 nm, flow rate 0.5 mL/min, column temperature 25 ºC, retention times: 13.96 min for (−) with 87.78 %, 19.0 min for (+) with 12.22%).

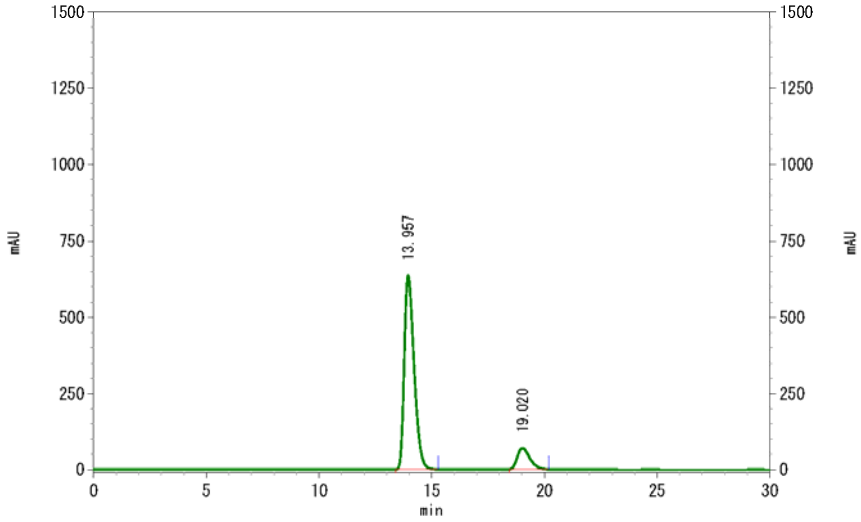
1. NMR spectrum



2. HPLC chart of the biaryl with 76% ee.

sample ID: MM4-01
date: 2010/10/06 11:52:43
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥100930_前田美音¥MM4-01_2010-10-06 11-48-05.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H



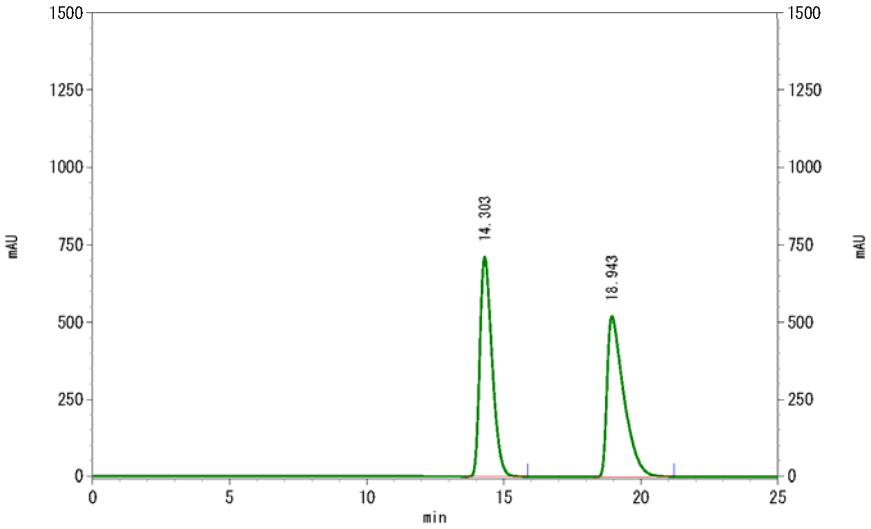
UV結果

保持時間	面積	面積%	開始時間	終了時間
13.957	76953397	87.776	13.39	15.28
19.020	10716508	12.224	18.43	20.19
トータル	87669905	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: MM4-rac02
date: 2010/10/11 10:22:00
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_75_25_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥100930_前田美音¥MM4-rac02_2010-10-11 10-17-55.dat

solvent: Hexane/IPA = 75/25
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

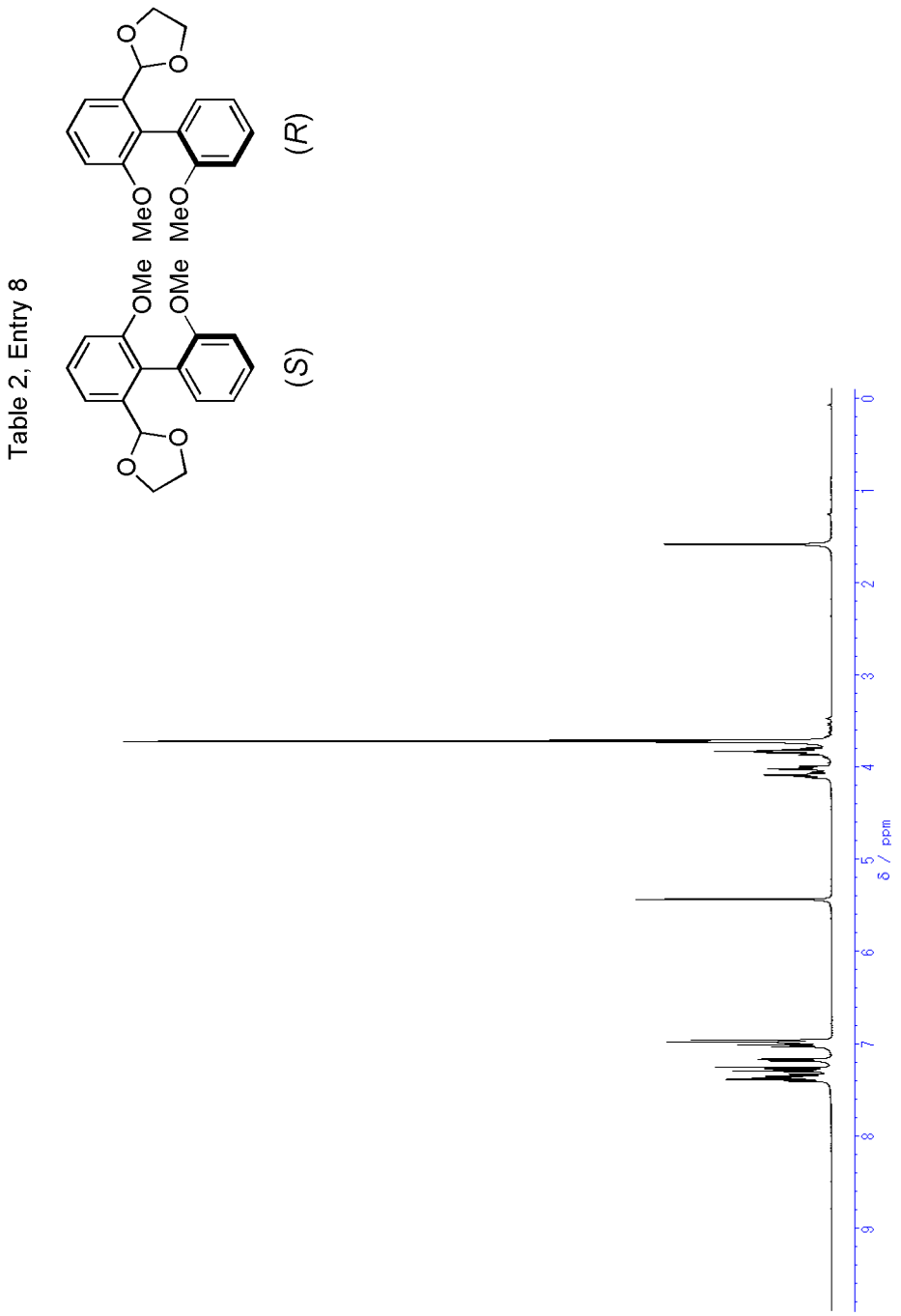


UV結果

保持時間	面積	面積%	開始時間	終了時間
14.303	88385394	49.962	13.62	15.86
18.943	88518447	50.038	18.33	21.20
トータル	176903841	100.000		

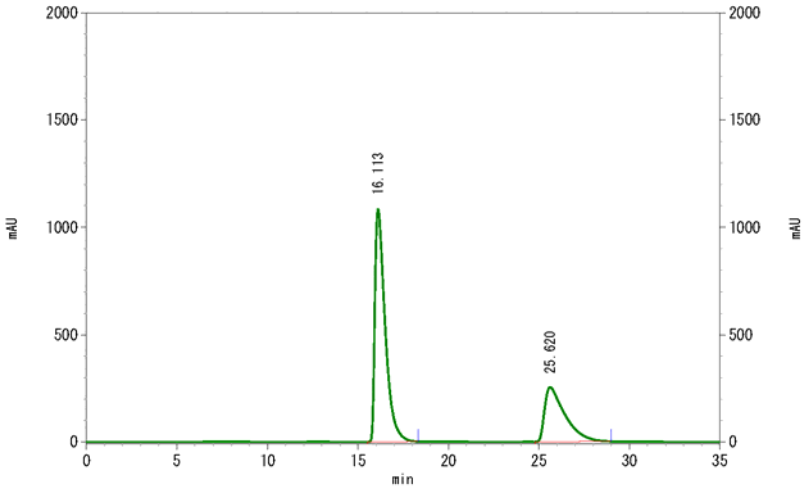
Table 2, entry 8: Purification by silica gel column chromatography (toluene/EtOAc = 19/1) gave a desired biaryl (263 mg, 92%) as white solid materials. The ee value was determined by HPLC analysis to be 37% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 60/40, 270 nm, and flow rate 0.5 mL/min, column temperature 25 ºC, and retention times: 16.11 min with 68.28%, 25.62 min with 31.72%).

1. NMR spectrum



2. HPLC chart of the biaryl with 37% ee.

sample ID: AS170-01
date: 2010/08/31 19:08:56
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_60_40_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS170-01_2010-08-31 19-07-32.dat
solvent: Hexane/IPA = 60/40
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

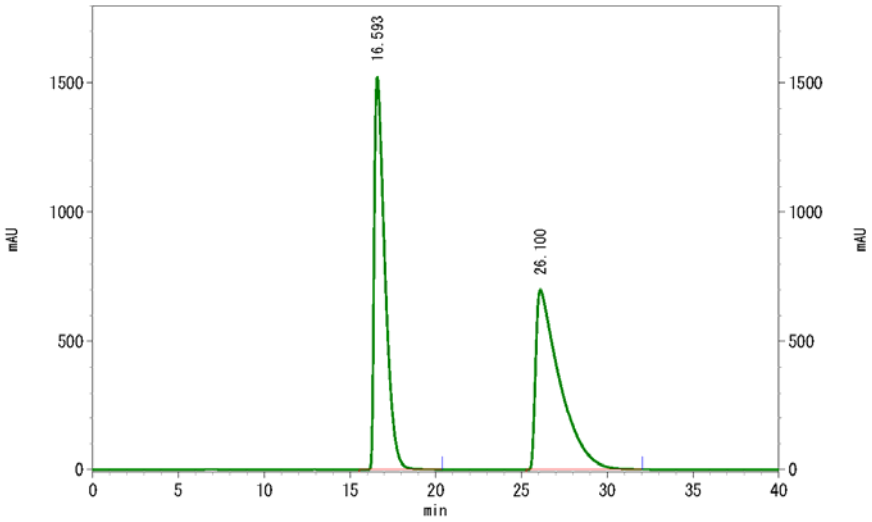


UV結果

	保持時間	面積	面積%	開始時間	終了時間
	16.113	170641088	68.281	15.51	18.35
	25.620	79267215	31.719	24.78	28.99
トータル		249908303	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: AS3-02
date: 2009/11/07 17:29:12
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_60_40_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_亀井俊徳¥AS3-02_OJH_2009-11-07 17-28-41.dat
solvent: Hexane/IPA = 60/40
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

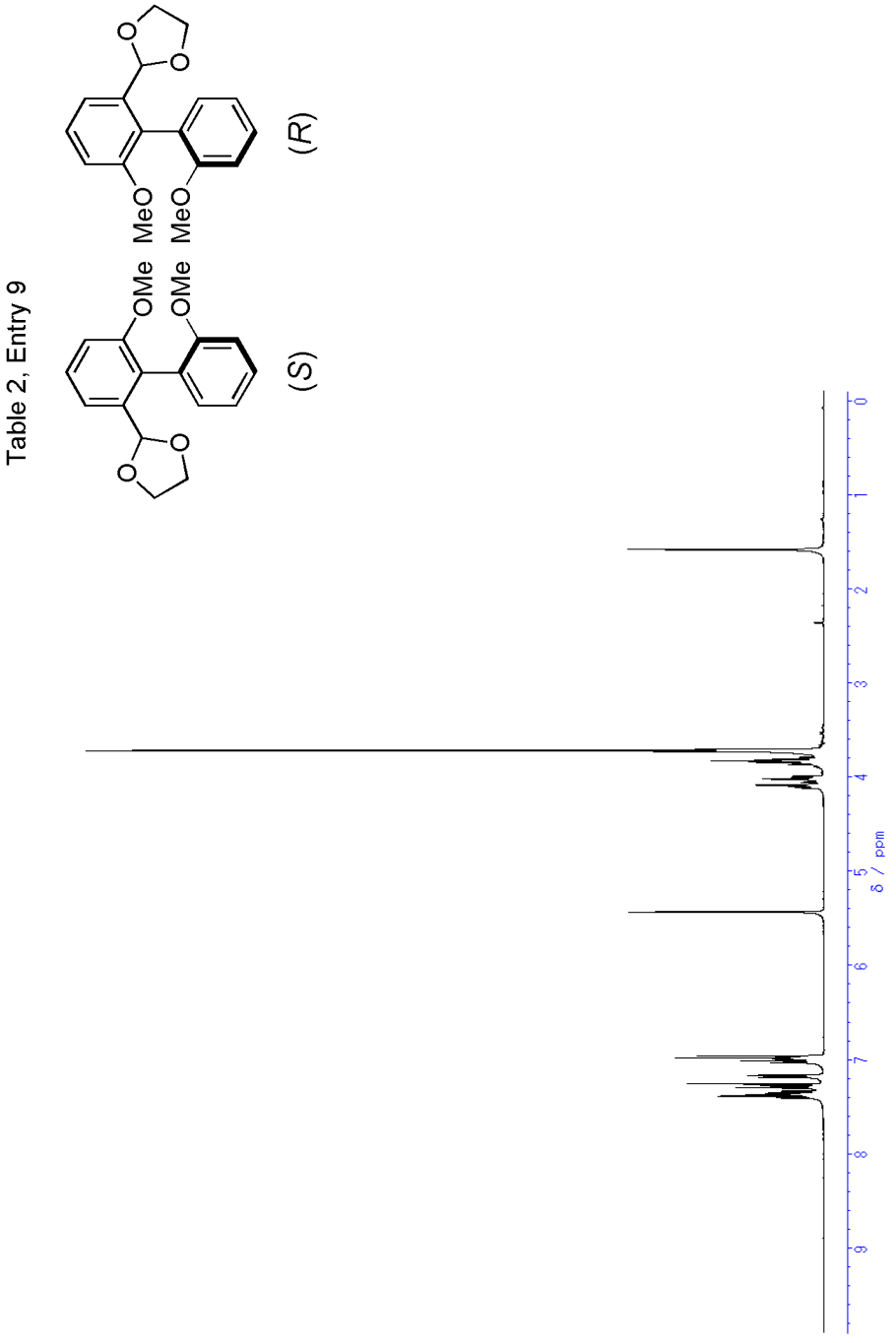


UV結果

保持時間	面積	面積%	開始時間	終了時間
16.593	265298613	49.287	15.54	20.39
26.100	272973921	50.713	25.29	32.06
トータル	538272534	100.000		

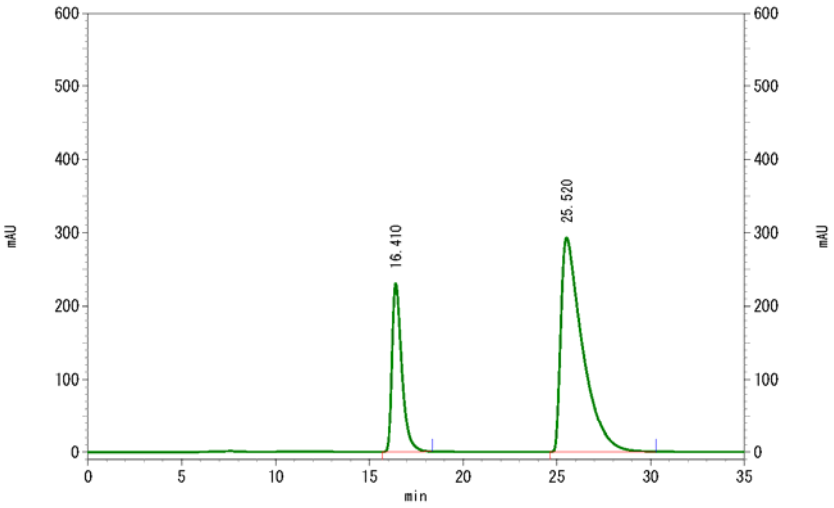
Table 2, entry 9: Purification by silica gel column chromatography (toluene/EtOAc = 19/1) gave a desired biaryl (260 mg, 91%) of $[\alpha]_D^{21}$ = -16.9 (c 0.50, $CDCl_3$) as white solid materials. The ee value was determined by HPLC analysis to be 47% with Daicel Chiralcel OJ (eluted with hexane-*i*PrOH 60/40, 270 nm, and flow rate 0.5 mL/min, column temperature 25 ºC, and retention times: 16.41 min with 26.35%, 25.52 min with 73.65%).

1. NMR spectrum



2. HPLC chart of the biaryl with 47% ee.

sample ID: AS1701-01
date: 2010/08/31 18:13:12
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_60_40_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS1701-01_2010-08-31 18-12-32.dat
solvent: Hexane/IPA = 60/40
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

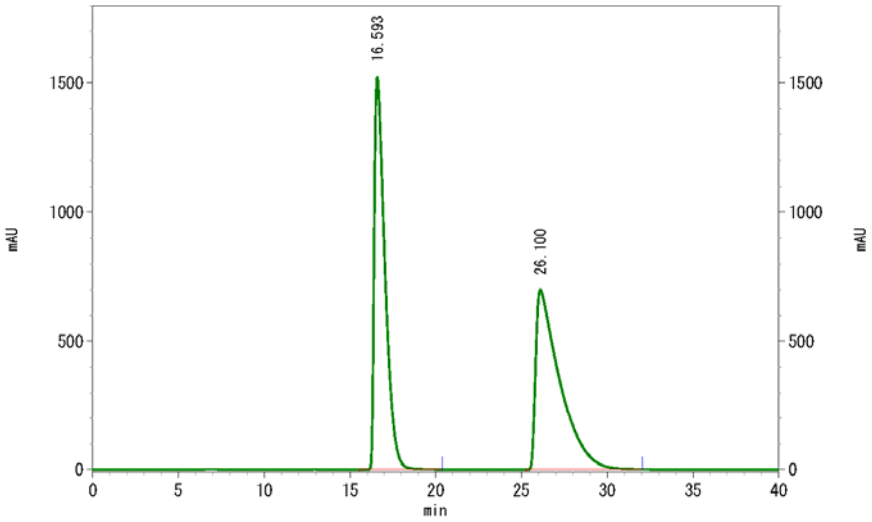


UV結果

	保持時間	面積	面積%	開始時間	終了時間
	16.410	33607334	26.348	15.70	18.38
	25.520	93946009	73.652	24.66	30.28
トータル		127553343	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: AS3-02
date: 2009/11/07 17:29:12
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_60_40_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_亀井俊徳¥AS3-02_OJH_2009-11-07 17-28-41.dat
solvent: Hexane/IPA = 60/40
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OJ-H

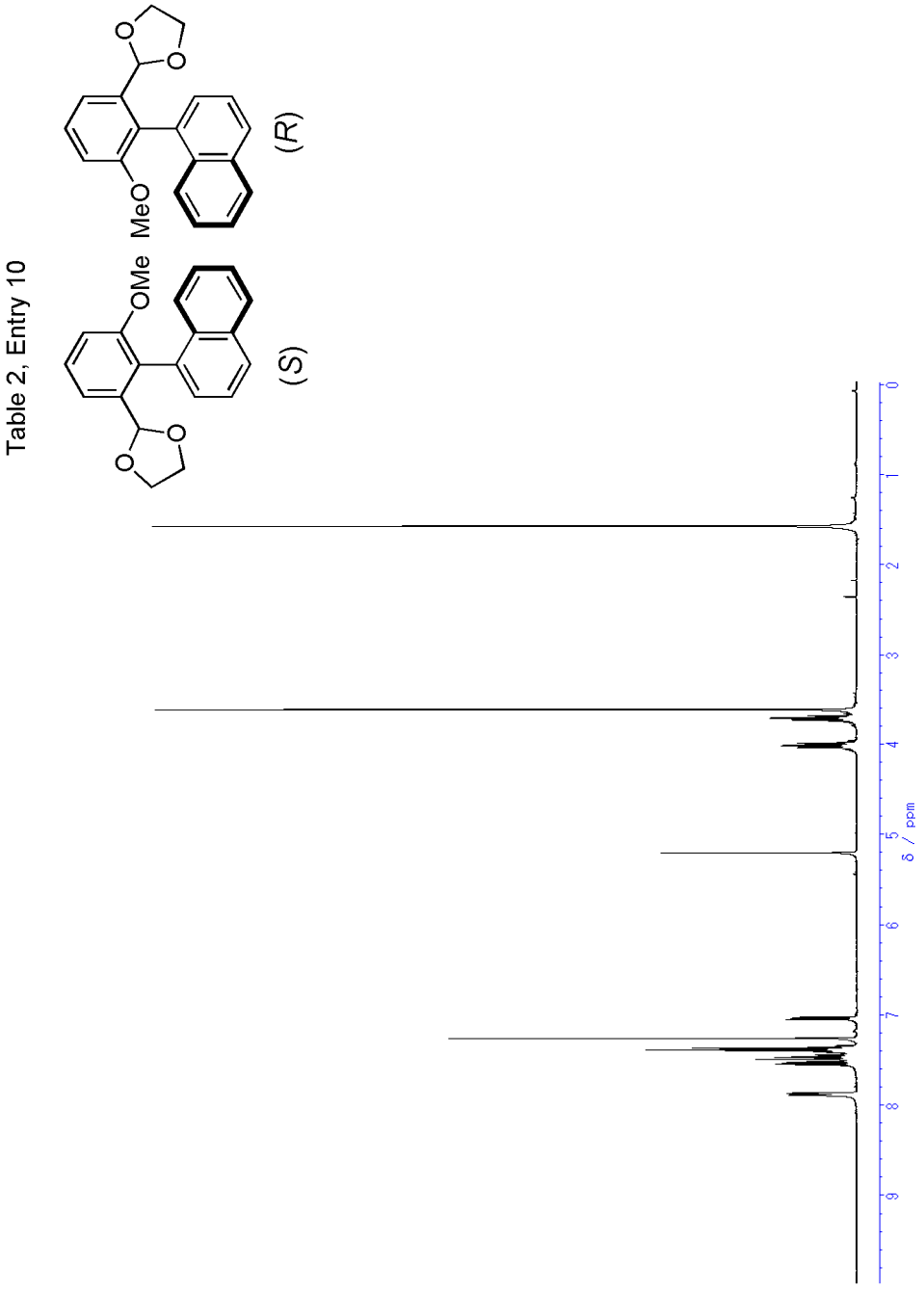


UV結果

保持時間	面積	面積%	開始時間	終了時間
16.593	265298613	49.287	15.54	20.39
26.100	272973921	50.713	25.29	32.06
トータル	538272534	100.000		

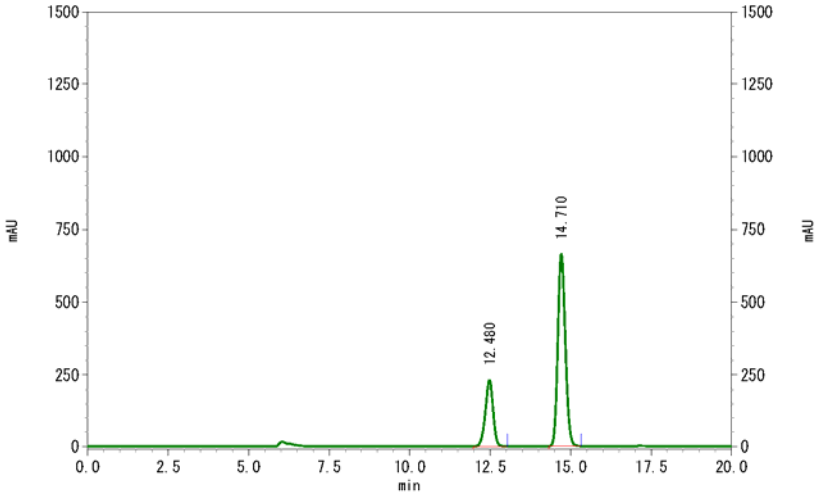
Table 2, entry 10: Purification by silica gel column chromatography (toluene/ methoxycyclopentane = 200/1) and recrystallization gave a desired biaryl (193 mg, 63%) of as white solid materials. The ee value was determined by HPLC analysis to be 47% with Daicel Chiralcel IC-3 (eluted with hexane-*i*PrOH 90/10, 270 nm, and flow rate 0.5 mL/min, column temperature 25 ºC, and retention times: 12.48 min with 26.39%, 14.71 min with 73.61%).

1. NMR spectrum



2. HPLC chart of the biaryl with 47% ee.

sample ID: AS177-01
date: 2010/09/11 11:21:55
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_90_10_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS177-01_2010-09-11 11-20-16.dat
solvent: Hexane/IPA = 90/10
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel IC-3

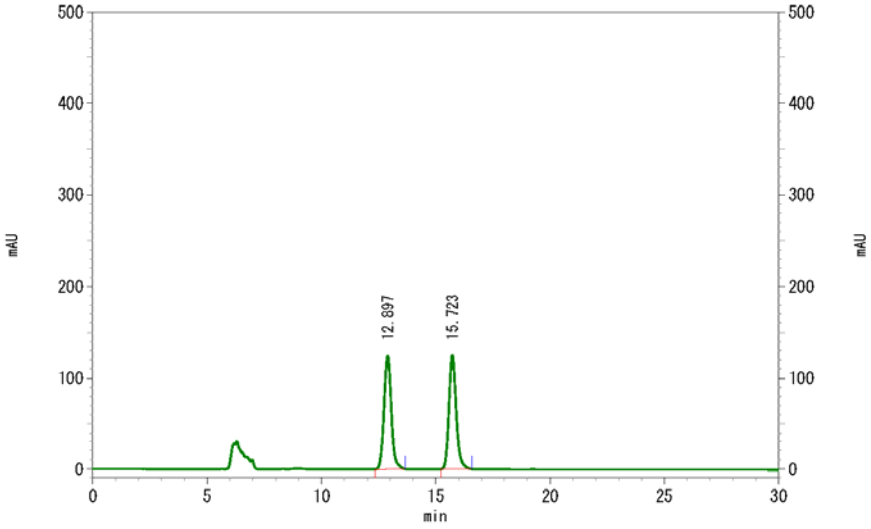


UV結果

保持時間	面積	面積%	開始時間	終了時間
12.480	15515485	26.389	11.98	13.04
14.710	43278841	73.611	14.31	15.32
トータル	58794326	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: AS126-02
date: 2010/06/08 19:29:09
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_90_10_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS126-02_2010-06-08 19-27-43.dat
solvent: Hexane/IPA = 90/10
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel IC-3

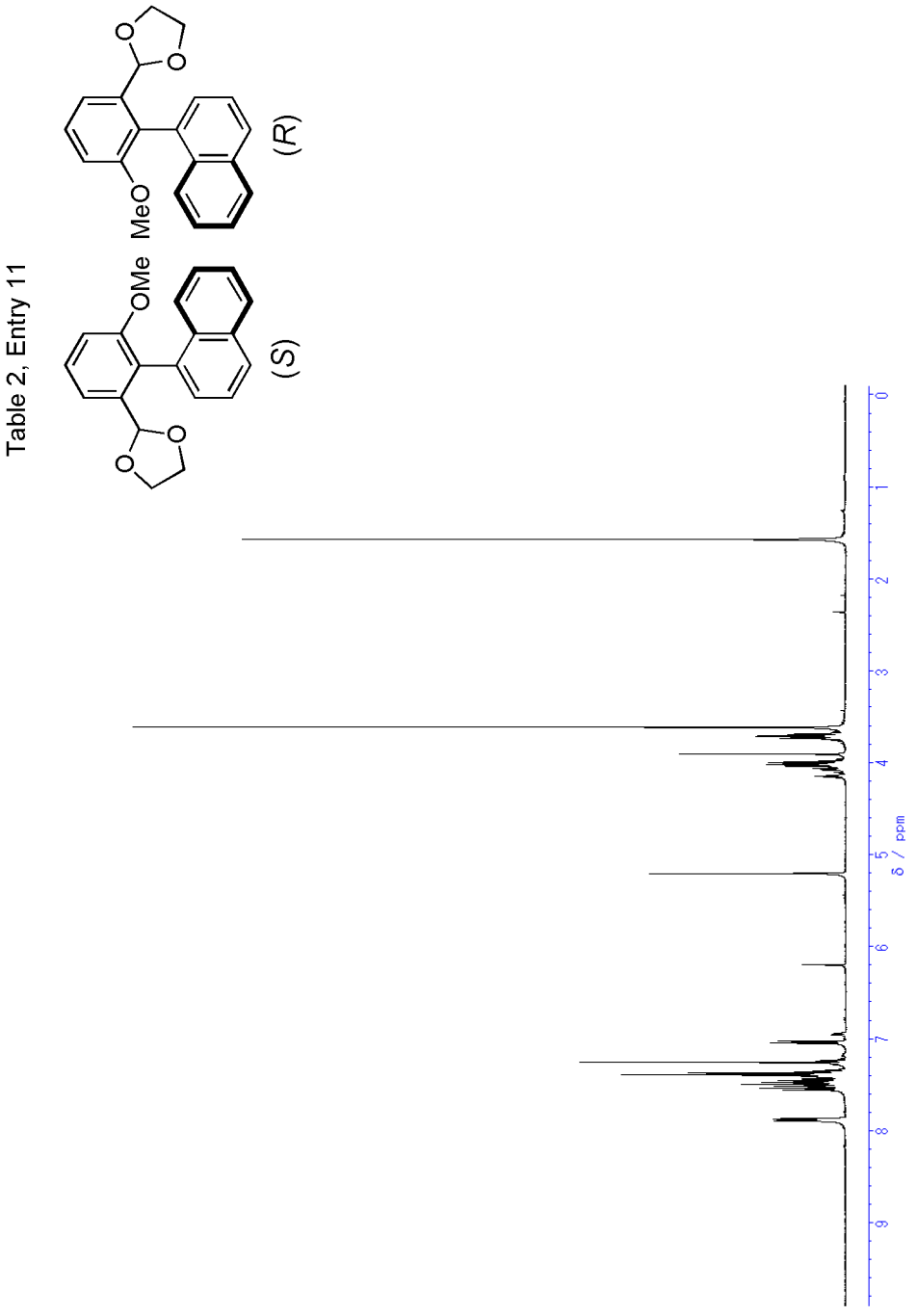


UV結果

保持時間	面積	面積%	開始時間	終了時間
12.897	11096668	50.086	12.34	13.66
15.723	11058542	49.914	15.23	16.59
トータル	22155210	100.000		

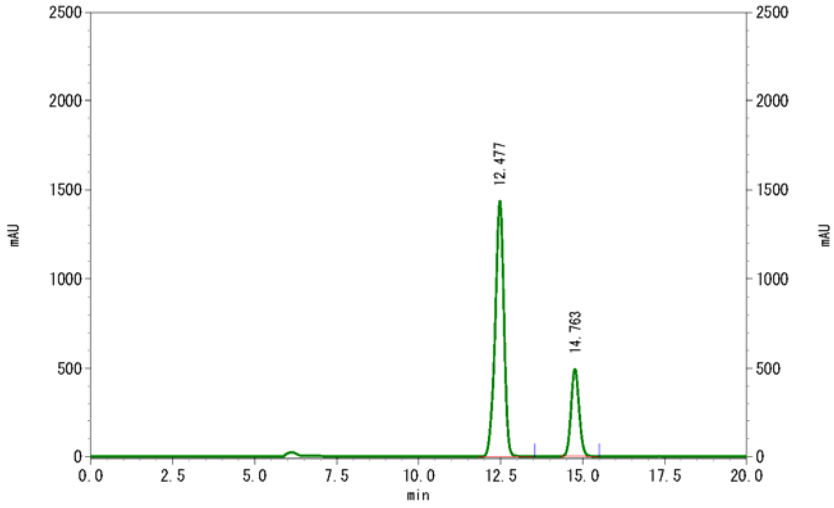
Table 2, entry 11: Purification by silica gel column chromatography (toluene/ methoxycyclopentane = 200/1) and recrystallization gave a desired biaryl (168 mg, 55%) of $[\alpha]_D^{22}$ = -2.24 (c 0.49, $CDCl_3$) as white solid materials. The ee value was determined by HPLC analysis to be 52% with Daicel Chiralcel IC-3 (eluted with hexane-*i*PrOH 90/10, 270 nm, and flow rate 0.5 mL/min, column temperature 25 ºC, and retention times: 12.48 min with 75.99%, 14.76 min with 24.01%).

1. NMR spectrum



2. HPLC chart of the biaryl with 52% ee.

sample ID: AS174-02
date: 2010/09/09 16:52:04
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_90_10_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS174-02_2010-09-09 16-50-43.dat
solvent: Hexane/IPA = 90/10
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel IC-3

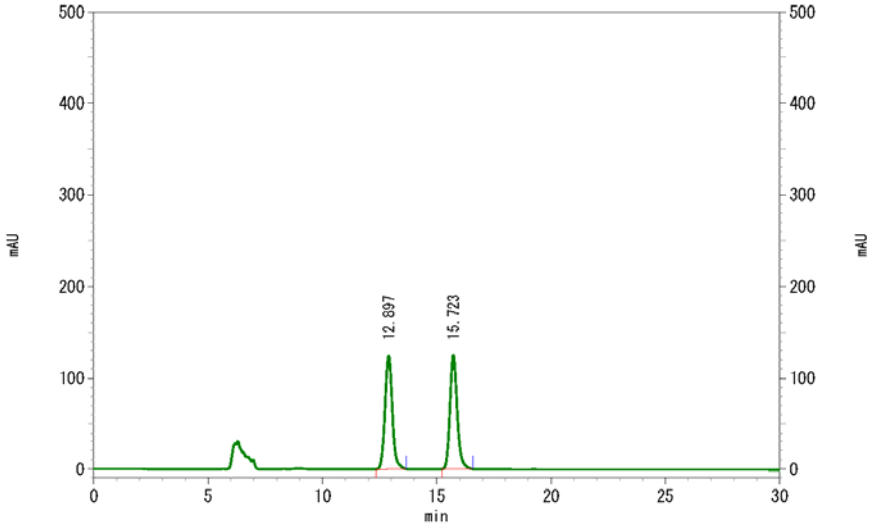


UV結果

保持時間	面積	面積%	開始時間	終了時間
12.477	107276218	75.988	11.85	13.54
14.763	33898285	24.012	14.34	15.51
トータル	141174503	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: AS126-02
date: 2010/06/08 19:29:09
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_90_10_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS126-02_2010-06-08 19-27-43.dat
solvent: Hexane/IPA = 90/10
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel IC-3

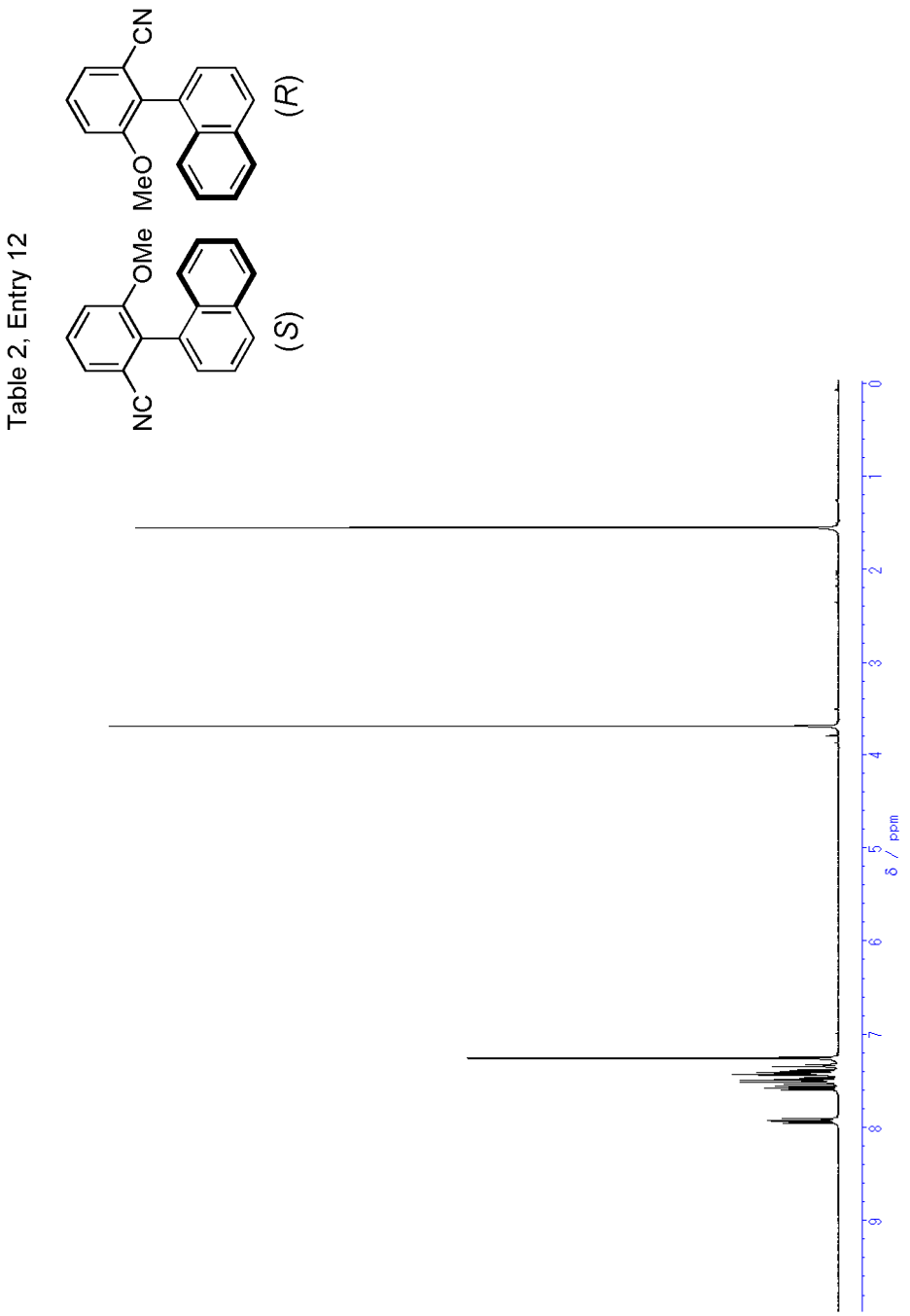


UV結果

保持時間	面積	面積%	開始時間	終了時間
12.897	11096668	50.086	12.34	13.66
15.723	11058542	49.914	15.23	16.59
トータル	22155210	100.000		

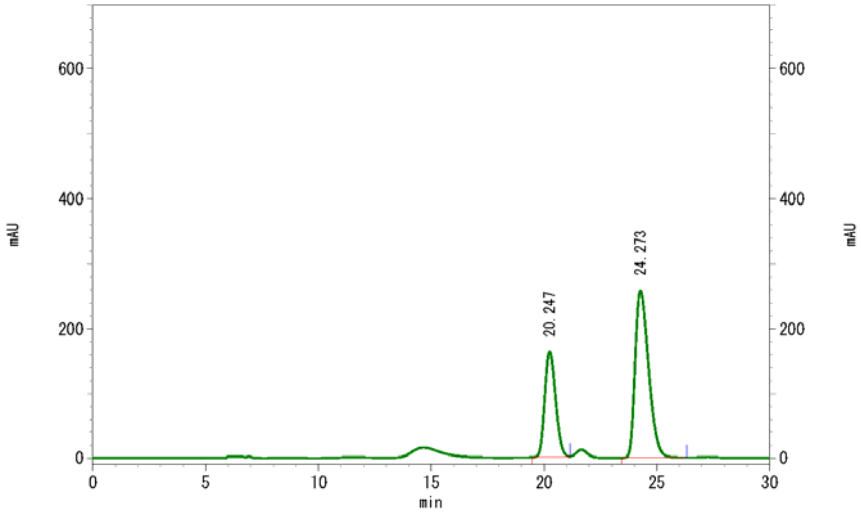
Table 2, entry 12: Purification by silica gel column chromatography (toluene/ Hexane = 1/1) and gave a desired biaryl (225 mg, 87%) of $[\alpha]_D^{22}$ = +39.8 (c 0.50, $CDCl_3$) as white solid materials. The ee value was determined by HPLC analysis to be 33% with Daicel Chiralcel IC-3 (eluted with hexane-*i*PrOH 90/10, 270 nm, and flow rate 0.5 mL/min, column temperature 25 ºC, and retention times: 20.25 min with 33.66%, 24.27 min with 66.34%).

1. NMR spectrum



2. HPLC chart of the biaryl with 33% ee.

sample ID: AS176-01
date: 2010/09/11 16:40:43
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_95_5_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_佐藤明広¥AS176-01_2010-09-11 16-39-47.dat
solvent: Hexane/IPA = 95/5
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave length (nm): 270
chiral column: Daicel chiralcel OD-H

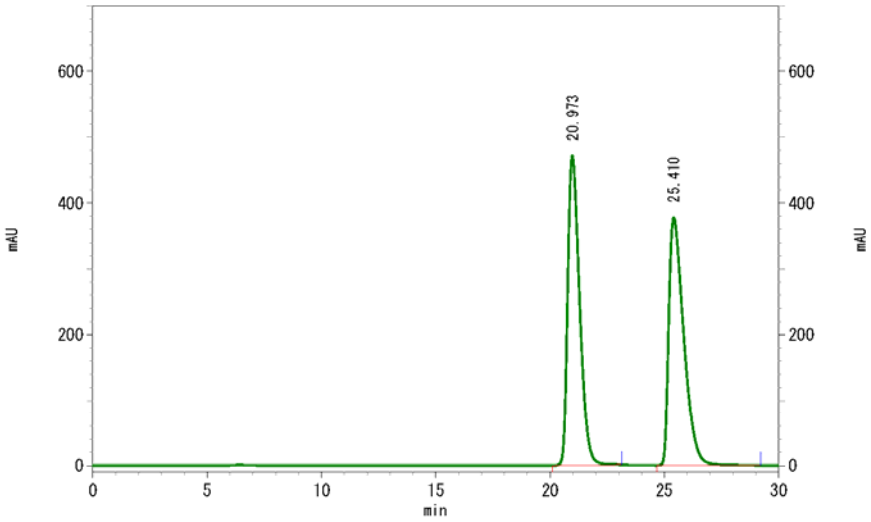


UV結果

保持時間	面積	面積%	開始時間	終了時間
20.247	21807566	33.660	19.47	21.14
24.273	42980673	66.340	23.43	26.32
トータル	64788239	100.000		

3. HPLC chart of a racemic biaryl.

sample ID: TK458-05_OD-H
date: 2009/11/18 13:36:18
method:C:¥EZChrom Elite¥Enterprise¥Projects¥Default¥Method¥H-IPA_95_5_0.5.met
data:C:¥Documents and Settings¥admin¥デスクトップ¥HPLC_亀井俊徳¥TK458-05_OD-H_2009-11-18 13-33-44.dat
solvent: Hexane/IPA = 95/5
flow rate (mL/min): 0.5
temperature (℃): 25.0
wave lenght (nm): 270
chiral column: Daicel chiralcel OD-H



UV結果

保持時間	面積	面積%	開始時間	終了時間
20.973	68264654	49.842	20.10	23.13
25.410	68696413	50.158	24.67	29.21
トータル	136961067	100.000		