Synthesis of Wholly Aromatic Polyketones Containing Optically Active Macrocycles

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ABSTRACT: Wholly aromatic polyketones **7** containing bisbinaphthyl macrocycles were synthesized *via* P₂O₅-MsOH mediated direct condensation reaction of compound **3** with chlorobenzoic acids **4**, nucleophilic aromatic substitution reaction of the resulting binaphthyl (**5**) with (*S*)-BINOL (**1**) in DMF/toluene, and NiBr₂/Zn mediated aromatic coupling polymerization. The resulting polyketones **7** have excellent thermal resistance and solubility to organic solvents. Cyclic monomers **6** and polymers **7** have higher molar rotations based on optically active cyclic structures than pre-cyclized compounds **5**. [doi:10.1295/polymj.PJ2006159]

KEY WORDS Aromatic Polyketones / Chiral Macrocycles / Nickel Complex / Aromatic Coupling Polymerization / Binaphthyl /

2,2'-Disubstituted 1,1'-binaphthyl derivatives have been applied to asymmetric organic syntheses as asymmetric catalysts, complexes and so on.^{1–3} Binaphthyl-containing polymers such as polyarylenes, polyimides, and polycarbonates have been much reported, which have been applied to polymeric catalysts and so on.^{4–8} We have also reported the syntheses of optically active aromatic polyketones bearing 2,2'-dioxo-1,1'-binaphthyl-6,6'-diyl units *via* nucleophilic aromatic substitution polymerization and nickel complex-mediated aromatic coupling polymerization.^{9–11} The resulting polyketones have high thermal stability and excellent solubility to organic solvents such as CHCl₃ and DMF. Large dihedral angles between two naphthalene rings raise the solubility of the polyketones. Cram *et al.* have reported that cyclic compounds composed of binaphthyls and crown ethers can recognize chiral molecules such as amines, amino acids and amino acid esters.^{12,13} Introduction of cyclic units to optically active molecules will form optically active cavities. This motivated us to develop polymers with rigid cyclic units consisting of optically active binaphthylene units, which will be applied to molecular recognition and separation, in particular, optical resolution.^{14–17} Preliminarily, we have succeeded in the preparation of cyclic aromatic bisbinaphthyls *via* two-step nucleophilic aromatic substitution reaction.¹⁸ Introduction of these cyclic aromatic bis(binaphthyl)s to polyketone backbones will produce polyketones with optically active cavities in the main chains.

In this paper, we would like to report wholly aromatic polyketones containing optically active bis(binaphthyl) macrocycles.

EXPERIMENTAL

General

¹H NMR spectra were recorded on a JEOL JNM-AL-300 (300 MHz). Chemical shifts were expressed in ppm relative to internal standard of Me₄Si (δ , 0.00). ¹³C NMR spectra were recorded on a JEOL JNM-AL-300 (75 MHz) and a JEOL JNM-ECX-400 (100 MHz). Chemical shifts were expressed in ppm relative to internal standard of CDCl₃ (δ , 77.0). IR measurement was recorded on a JEOL FT/IR-4100. Molar rotations ($[\phi]_D^{25}$) were measured in CHCl₃ (5×10^{-5} mol/L) using a JASCO DIP-1000 digital polarimeter. Circular Dichroism (CD) measurement was performed in CHCl₃ (2×10^{-5} mol/L) using a JASCO J820. Cell length was 1 cm. Gel permeation chromatography (GPC) measurements were carried out at a flow rate of 1.0 mL min⁻¹ at 40 °C using CHCl₃ as an eluent on a Shimadzu SPD-6A equipped with a UV detector (300 nm) and a Shodex KD-806M column. Glass transition temperatures (T_g) were determined on the basis of DSC curves. DSC curves were recorded on a Rigaku DSC-8230 differential scanning calorimeter with a heating rate of 10 K min⁻¹. P₂O₅-MsOH mixture was prepared according to Eaton's procedure.¹⁹ 2,2'-Bis(4-fluorobenzoyl-4-phenoxy)-1,1'-binaphthyl (**3**) was prepared according to the previous report.¹⁸

6,6'-Bis(4-chlorobenzoyl)-2,2'-bis(4-fluorobenzoyl-4-phenoxy)-1,1'-binaphthyl **5a**

To a dried 30 mL two-necked flask was added 4-chlorobenzoic acid (**4a**, 1.56 g, 10.0 mmol) and P₂O₅-

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MsOH (12.5 mL). Binaphthyl **3** (1.70 g, 2.5 mmol) was added after the mixture was stirred for 30 min at 80 °C. The reaction mixture was stirred for 72 h at 80 °C. The mixture was quenched by pouring it into water in a beaker. After the mixture was neutralized with 6M aqueous sodium hydroxide, the organic layer was extracted with chloroform three times. The combined extracts were dried over anhydrous MgSO₄. After removal of the drying agent, chloroform was removed under reduced pressure. The crude product was purified by column chromatography to afford disubstituted product **5a** in 84% yield.

¹H NMR δ (300 MHz, CDCl₃): 6.88 (4H, d, J = 8.7 Hz), 7.13 (4H, t, J = 8.7 Hz), 7.39–7.44 (4H, m), 7.50 (4H, d, J = 8.4 Hz), 7.62 (4H, d, J = 8.4 Hz), 7.72 (4H, dd, J = 5.7, 8.4 Hz), 7.81 (2H, dd, J = 1.2, 9.0 Hz), 7.82 (4H, d, J = 8.4 Hz), 8.07 (2H, d, J = 9.0 Hz), 8.33 (2H, d, J = 1.2 Hz) ppm. ¹³C NMR δ (75 MHz, CDCl₃): 115.4 (d, ² J_{CF} = 21.6 Hz), 117.8, 120.4, 122.0, 126.0, 126.9, 128.6, 128.7, 129.6, 131.4, 132.0, 132.2, 132.3 (d, ³ J_{CF} = 9.4 Hz), 133.7, 133.8, 134.0, 135.8, 136.1, 139.0, 153.5, 160.4, 165.3 (d, ¹ J_{CF} = 272 Hz), 193.5, 194.9 ppm. IR ν (neat) 1655, 1596, 1403, 1276, 1239, 1090 cm⁻¹. Anal. Calcd. for C₆₀H₃₄Cl₂F₂O₆: C 75.08%, H 3.57%. Found, C 74.78%, H 3.39%.

6,6'-Bis(3-chlorobenzoyl)-2,2'-bis(4-fluorobenzoyl-4-phenoxy)-1,1'-binaphthyl **5b**

Binaphthyl **5b** was prepared according to synthesis of binaphthyl **5a**. Yield, 82%.

¹H NMR δ (300 MHz, CDCl₃): 6.89 (4H, d, J = 8.7 Hz), 7.13 (4H, t, J = 8.7 Hz), 7.40 (2H, d, J = 9.0 Hz), 7.42 (2H, d, J = 9.0 Hz), 7.47 (2H, d, J = 7.8 Hz), 7.59–7.64 (6H, m), 7.70–7.75 (6H, m), 7.80–7.85 (4H, m), 8.09 (2H, d, J = 9.0 Hz), 8.36 (2H, s) ppm. ¹³C NMR δ (75 MHz, CDCl₃): 115.4 (d, ² J_{CF} = 21.6 Hz), 117.8, 120.4, 122.0, 126.0, 126.9, 128.1, 129.6, 129.7, 129.8, 132.0, 132.1, 132.22, 132.24, 132.3, 132.4 (d, ³ J_{CF} = 8.0 Hz), 133.72, 133.74, 134.7, 136.2, 139.3, 153.6, 160.4, 165.2 (d, ¹ J_{CF} = 252 Hz), 193.7, 194.8 ppm. IR ν (neat) 1656, 1593, 1414, 1274, 1242, 1090 cm⁻¹. Anal. Calcd. for C₆₀H₃₄Cl₂F₂O₆: C 75.08%, H 3.57%. Found, C 75.23%, H 3.36%.

Cyclic Monomer **6a**

To a 30 mL two-necked flask equipped with a reflux condenser and a Dean-Stark trap were added potassium carbonate (410 mg, 3.0 mmol), freshly distilled DMF (5 mL), and toluene (6 mL). A mixed DMF solution (5 mL) of binaphthyl **5a** (192 mg, 0.2 mmol) and (*S*)-BINOL (**1**, 57 mg, 0.2 mmol) was dropped to the flask at 150 °C over 8 h. The reaction mixture was further stirred for 108 h. Solvent was decreased to 5 mL under reduced pressure. The concentrated mixture

was poured into water and the resulting solid was collected by filtrate suction. The collected solid was washed with methanol and extracted with ethyl acetate three times. Ethyl acetate was removed from the extracts under reduced pressure to obtain a pale yellow solid. The resulting residue was purified by silica gel column chromatography (CHCl₃), recycling preparative GPC (CHCl₃), and preparative TLC to give pure **6a** in 29% yield. Recycling preparative GPC separation was carried out at a flow rate of 3.5 mL min⁻¹ using CHCl₃ as an eluent on a JAI LC9201 recycling HPLC system equipped with a JAI UV detector (254 nm) and JAIGEL2.5H and 3H columns.

¹H NMR δ (300 MHz, CDCl₃): 6.49 (4H, d, J = 8.1 Hz), 6.51 (4H, d, J = 8.4 Hz), 7.21–7.24 (10H, m), 7.28 (2H, d, J = 9.0 Hz), 7.36 (2H, d, J = 9.0 Hz), 7.37 (2H, t, J = 8.4 Hz), 7.43 (2H, d, J = 9.0 Hz), 7.50 (2H, t, J = 8.4 Hz), 7.51 (4H, d, J = 8.4 Hz), 7.82 (2H, dd, J = 1.8, 9.0 Hz), 7.86 (4H, d, J = 8.4 Hz), 7.96 (2H, d, J = 7.8 Hz), 7.97 (2H, d, J = 9.0 Hz), 8.09 (2H, d, J = 9.0 Hz), 8.38 (2H, d, J = 1.8 Hz) ppm. ¹³C NMR δ (100 MHz, CDCl₃): 116.2, 116.3, 119.9, 121.0, 122.8, 123.2, 125.5, 125.8, 126.1, 127.0, 127.1, 128.2, 128.8, 130.0, 130.2, 130.8, 130.9, 131.1, 131.5, 131.6, 131.7, 131.9, 132.0, 134.2, 134.3, 135.9, 136.3, 139.1, 149.8, 152.2, 160.2, 161.0, 192.7, 195.1 ppm. IR ν (neat) 1656, 1594, 1242, 1090 cm⁻¹. Anal. Calcd. for C₈₀H₄₆Cl₂O₈: C 79.66%, H 3.84%. Found, C 79.77%, H 3.73%.

Cyclic Monomer **6b**

Monomer **6b** was synthesized according to synthesis of **6a**. Yield, 34%.

¹H NMR δ (300 MHz, CDCl₃): 6.48 (4H, d, J = 9.3 Hz), 6.52 (4H, d, J = 9.3 Hz), 7.21–7.29 (12H, m), 7.33–7.52 (10H, m), 7.60 (2H, d, J = 8.7 Hz), 7.76 (2H, d, J = 7.8 Hz), 7.83 (2H, d, J = 8.7 Hz), 7.87 (2H, s), 7.94 (2H, d, J = 7.8 Hz), 7.96 (2H, d, J = 8.7 Hz), 8.11 (2H, d, J = 9.0 Hz), 8.39 (2H, s) ppm. ¹³C NMR δ (75 MHz, CDCl₃): 116.0, 116.3, 119.9, 121.0, 122.7, 123.2, 125.5, 125.8, 126.1, 127.0, 128.1, 129.7, 129.8, 129.9, 130.0, 130.1, 130.8, 131.0, 131.4, 131.5, 131.6, 132.0, 132.1, 132.2, 132.4, 134.0, 134.2, 134.7, 136.4, 139.2, 149.8, 152.2, 160.2, 160.9, 192.6, 194.8 ppm. IR ν (neat) 1655, 1595, 1241, 1074 cm⁻¹. Anal. Calcd. for C₈₀H₄₆Cl₂O₈: C 79.66%, H 3.84%. Found, C 79.80%, H 3.78%.

Polyketone **7a**

Nickel complex mediated aromatic coupling polymerization of **6a** was performed according to our previous paper.¹⁰ Yield 93%.

¹H NMR δ (300 MHz, CDCl₃): 6.37–6.56 (8H, m), 7.06–7.25 (10H, m), 7.28–7.55 (10H, m), 7.58–7.66 (2H, m), 7.74–8.15 (14H, m), 8.39–8.48 (2H, m) ppm.

^{13}C NMR δ (75 MHz, CDCl_3): 116.2, 119.9, 122.8, 122.9, 123.2, 125.5, 125.7, 125.8, 125.9, 126.0, 127.3, 128.1, 128.4, 130.0, 130.1, 130.2, 130.8, 130.9, 131.0, 131.1, 131.5, 131.6, 131.9, 132.0, 134.3, 134.6, 136.2, 136.3, 137.1, 149.8, 160.2, 160.9, 192.7, 195.7 ppm. IR $\nu(\text{neat})$ 1654, 1595, 1242, 1073 cm^{-1} . Anal. Calcd. for $\text{C}_{80}\text{H}_{46}\text{O}_8$: C 84.64%, H 4.08%. Found, C 83.81%, H 3.96%.

Polyketone **7b**

Yield 89%. ^1H NMR δ (300 MHz, CDCl_3): 6.38–6.54 (8H, m), 7.14–7.24 (8H, m), 7.30–7.41 (8H, m), 7.42–7.54 (4H, m), 7.55–7.65 (2H, m), 7.80–7.99 (10H, m), 8.00–8.11 (2H, m), 8.12–8.19 (2H, m), 8.37–8.48 (2H, m) ppm. ^{13}C NMR δ (75 MHz, CDCl_3): 116.1, 119.9, 120.9, 122.7, 123.1, 125.4, 125.7, 126.0, 127.0, 127.2, 128.1, 128.3, 128.5, 128.5, 129.0, 129.5, 130.0, 130.8, 131.0, 131.2, 131.4, 132.0, 132.1, 132.5, 134.2, 134.4, 134.6, 136.3, 138.4, 140.5, 149.7, 152.0, 160.2, 160.8, 192.6, 195.9 ppm. IR $\nu(\text{neat})$ 1651, 1594, 1241 cm^{-1} . Anal. Calcd. for $\text{C}_{80}\text{H}_{46}\text{O}_8$: C 84.64%, H 4.08%. Found, C 84.02%, H 3.97%.

RESULTS AND DISCUSSION

Synthesis of Pre-Cyclized Products **5**

(*S*)-BINOL (**1**) was allowed to react with six equimolar amounts of 4,4'-difluorobenzophenone (**2**) in the presence of K_2CO_3 in DMF to afford difluoride **3** in 82% yield.^{14,15,18} Next, reaction of difluoride **3** with 4-chlorobenzoic acid (**4a**) was carried out in P_2O_5 -MsOH.¹⁹ According to our previous report,¹¹ the reaction at 80 °C afforded the corresponding disubstituted binaphthyl (**5a**) in good yield. Similarly, disubstituted binaphthyl **5b** was also prepared in good yield (Scheme 1).

Cyclization of **5a** with (*S*)-BINOL (**1**)

The results of cyclization of **5a** with (*S*)-BINOL (**1**) are shown in Table I. When DMF was used as a solvent (run 1), polymerization proceeded preferentially without any production of the cyclized compound (**6a**) even in high-diluted solution. On the other hand, when DMF and toluene were used as cosolvent, cyclized product **6a** was obtained. The yield of product **6a** increased up to 29% by optimization of ratio of DMF/toluene and elongation of reaction time to 108 h (run 4). Consumption of starting material **5a** in run 4 occurred much more slowly than that in run 1. Addition of toluene presumably decreased the polarity of solvent and the reactivity of aryloxy anion, which was advantageous to intermolecular reaction, *i.e.*, cyclization over intramolecular reaction, *i.e.*, oligomerization. Isomeric cyclic monomer **6b** was ob-

Scheme 1. Preparation of pre-cyclized compounds **5a** and **5b**.

Table I. Synthesis of cyclic bis(binaphthyl)s **6a** and **6b**^a

Run	5	DMF/mL	toluene/mL	Time/h	Yield/% ^b
1	5a	15	0	42	0
2		10	2	42	7(6a)
3		10	2	108	23
4		10	6	108	29
5		15	2	42	5
6		15	2	108	15
7	5b	15	2	108	21(6b)
8		10	2	108	22
9		10	6	108	34

^aReaction conditions; **5** (0.2 mmol), (*S*)-**1** (0.2 mmol), K_2CO_3 (3.0 mmol), N_2 . ^bIsolated yield.

tained in a similar manner (run 9).

The structure of cyclic monomers **6a** was identified by ^1H NMR and COSY-NMR spectroscopy. Figures 1 and 2 show ^1H NMR spectra of difluoride **5a** and cyclic monomer **6a**, and the COSY spectrum of **6a**. In the ^1H NMR spectrum of cyclic monomer **6a**, two pairs of doublet signals at 6.49 and 6.51 ppm and a signal at 7.22 ppm were observed. The signal at

Figure 1. ^1H NMR spectra of **5a** (top), **6a** (middle), and **7a** (bottom).

7.22 ppm was strongly correlating with those at 6.49/6.51 ppm in the COSY spectrum of **6a** as depicted in Figure 2. Furthermore, the signal at 7.22 ppm was strongly correlating with that of the carbonyl carbon in the cyclic structure in the HMBC spectrum of **6a**. Accordingly, the signals at 7.22 ppm and 6.49/6.51 ppm were assigned as the protons indicated as H_{a}' and H_{b}' in Figure 2, respectively. These signals shifted upfield as shown in Figure 2. This is probably due to the shielding effect by neighboring aromatic rings based on construction of macrocyclic units. These results support that the resulting compounds have cyclic structures.

NiBr_2/Zn -mediated aromatic coupling polymerization of the resulting monomers **6a** and **6b** afforded the corresponding wholly aromatic polyketones **7a** and **7b**. There was no remarkable difference between ^1H NMR spectra of monomer **6a** and polymer **7a** except that some of signals in polymer **7a** were broadening.

The resulting polymers (**7a** and **7b**) were soluble to chloroform and DMF but insoluble to acetone. Probably, the adequate solubility is due to the suitably twisting structures between naphthalene rings of the resulting polymers, similar to reported aromatic polyketones bearing 2,2'-dioxo-1,1'-binaphthyl-6,6'-diyl units.^{9–11} Glass transition temperatures (T_g) of the polymers **7a** and **7b** were almost as high as those of wholly aromatic polyketones containing 2,2'-dime-

Figure 2. ^1H ^1H -COSY spectrum of macrocycle **6a**.

thoxy-1,1'-binaphthylene units previously reported.¹⁰ Molecular weights were relatively lower than linear wholly aromatic polyketones.¹⁰ The plausible reason is that bulky cyclic units disturb the smooth aromatic coupling polymerization, in particular, the second oxidative addition of aryl chloride to aryl nickel intermediate. For further polymerization, introduction of rod-like linkers to main chains will be requisite.

Molar rotations of **5–7** are shown in Table II. Molar rotations of cyclic monomers **6** and polymers **7** were larger than those of the corresponding pre-cyclized compounds **5**. Molar rotations of polymers **7** were almost the same as those of cyclic monomers **6**. CD spectra of monomer **6a** and polymer **7a** were almost the same and they were larger Cotton effects than pre-cyclized compound **5a** (Figure 3). These results indicate that optically active macrocycles will form in monomers **6** and polymers **7**. However, remarkable change by polymerization was not observed. We could not clear the secondary structures of polymers **7**.

CONCLUSIONS

Cyclic monomers **6a** and **6b** were synthesized through nucleophilic aromatic substitution reaction in high-diluted DMF/toluene solvents. These compounds were purified by using the recycling prepara-

Table II. Nickel complex-mediated aromatic coupling polymerization of cyclic bis(binaphthyl)s **6a** and **6b**

Run	6	Yield	M_n^a	$[\phi]_D^{25}/\text{deg}^b$	T_g^c
		/%		5	7
1	6a	92(7a)	6400(7a)	−50(5a) −702(6a) −748(7a)	178
2	6b	89(7b)	5000(7b)	−48(5b) −277(6b) −284(7b)	176

Reaction conditions: monomer **6** (0.1 mmol), NiBr_2 (0.05 mmol), Zn (0.3 mmol), PPh_3 (0.1 mmol), 2,2'-bpy (0.05 mmol), DMF (0.5 mL), 100 °C, 2 h. ^aDetermined on the basis of GPC using PS standards (column: Shodex KD-806M, eluent: CHCl_3 , UV detector, 300 nm). ^b $[\phi]_D^{25}$ was measured in CHCl_3 (5×10^{-5} mol/L). ^cDetermined on the basis of DSC curves. Heating rate, 10 K/min.

Figure 3. CD spectra of **5a**, **6a** and **7a**.

tive HPLC system with JALGEL 2.5H and 3H columns. Wholly aromatic polyketones **7** were synthesized *via* NiBr_2/Zn mediated aromatic coupling polymerization of the resulting cyclic monomers **6a** and **6b**. The resulting polyketones **7a** and **7b** have excellent thermal resistance and solubility to organic solvents such as CHCl_3 . In addition, the cyclic monomers **6** and polymers **7** have higher molar rotations and showed CD spectra with large Cotton effects based on optically active cyclic structures than pre-cyclized compounds **5**.

The resulting aromatic polyketones **7a** and **7b** have the possibility to fine molecular recognition of optically active molecules under severe conditions such as acidic conditions.

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