

*Supplementary Information for*

**Switchable Chirality of Circularly Polarized Luminescence in Dilute Solution Based on the Solvent-dependent Helix Inversion of Poly(quinoxaline-2,3-diyl)s**

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## **1 General**

All reactions were carried out under an atmosphere of nitrogen with magnetic stirring. <sup>1</sup>H NMR spectra were recorded on a Varian 400-MR spectrometer at ambient temperature. <sup>1</sup>H NMR data are reported as follows: chemical shift in ppm downfield from tetramethylsilane ( $\delta$  scale), multiplicity (s = singlet, d = doublet, t = triplet, q =

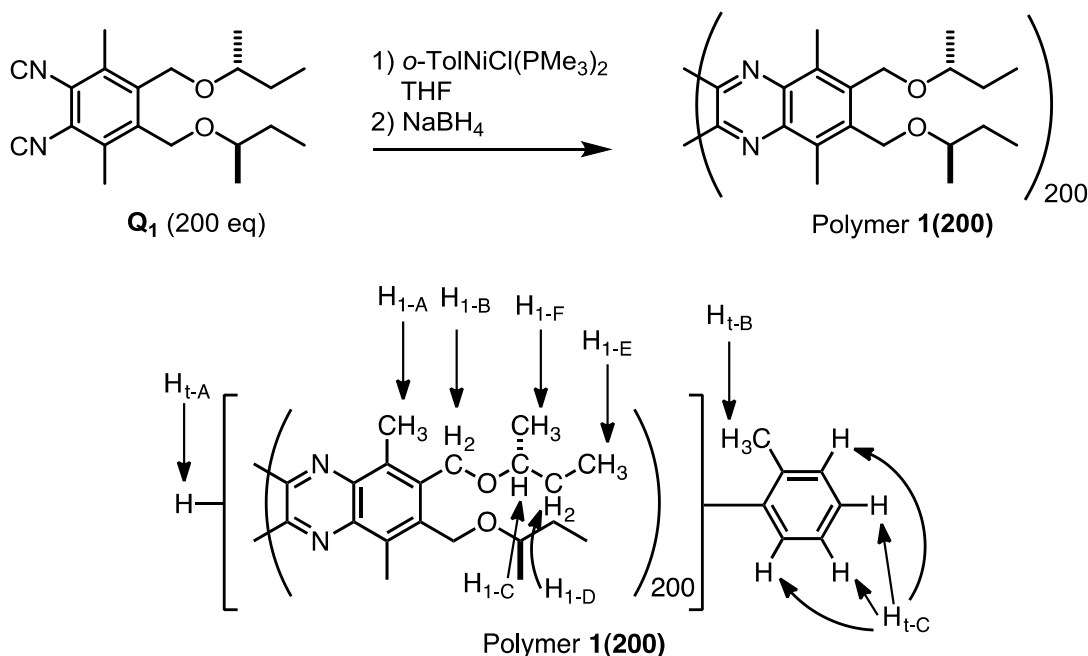
quartet, quint = quintet, sex = sextet, m = multiplet and br = broad), coupling constant (Hz), and integration. The GPC analysis was carried out with TSKgel G4000H<sub>HR</sub> or TSKgel GMH<sub>XL</sub> (CHCl<sub>3</sub>, polystyrene standards). Preparative GPC was performed on JAI LC-908 equipped with JAIGEL-1H and -2H columns in a series (CHCl<sub>3</sub>). UV spectra were recorded on a JASCO V-500 spectrometer equipped with a JASCO ETC-505T temperature/stirring controller at 20 °C. Circular dichroism (CD) spectra were recorded on a JASCO J-750 spectrometer equipped with a JASCO PTC-423L temperature/stirring controller at 20 °C. Fluorescence spectra were recorded on a JASCO FP-6300 spectrofluorometer. Absolute quantum yields were measured by a Hamamatsu absolute PL quantum yield spectrometer C11347. Circularly polarized luminescence (CPL) spectra were recorded on a JASCO CPL-200S at room temperature. Flash chromatography was performed using a Biotage Isolera One flash purification system with silica gel flash cartridges.

Tetrahydrofuran (THF) was dried and deoxygenized using an alumina/catalyst column system (Glass Contour Co.). *o*-ToINiCl(PMe<sub>3</sub>)<sub>2</sub>,<sup>1</sup> 1,2-bis((*R*)-sec-butoxymethyl)-4,5-diisocyno-3,6-dimethylbenzene **Q1**,<sup>2</sup> 1,2-diisocyno-3,6-dimethyl-4,5-bis((*S*)-2-methylbutoxy)benzene **Q3**,<sup>3</sup> **2(200)**,<sup>4</sup> 3,6-dimethyl-4,5-bis((*S*)-2-methylbutoxy)benzene-1,2-diamine,<sup>3</sup> were prepared according to the reported procedures. Other chemical reagents were purchased from the commercial sources and were used without further purification.

## 2 Experimental Procedures and Spectral Data for New Compounds

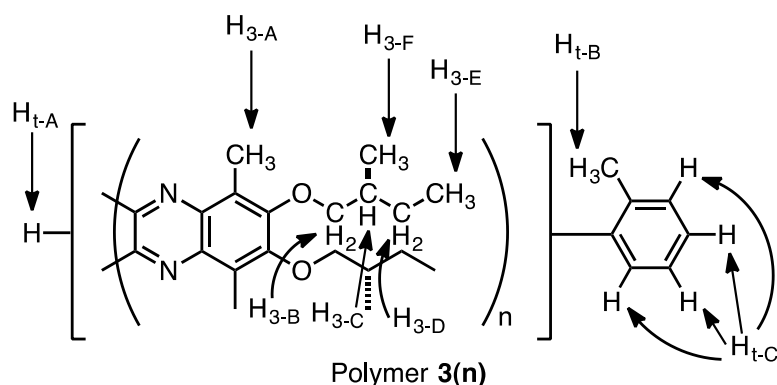
**Synthesis of polymer 1(200):**  $Q_1$  (48.6 mg, 148  $\mu\text{mol}$ ) was dissolved in THF (6 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (13.1 mM, 56.5  $\mu\text{L}$ , 0.740  $\mu\text{mol}$ ) was added to the monomer solution with vigorous stirring. After 48 h, LiBH<sub>4</sub> (0.50 M, 283  $\mu\text{L}$ , 142  $\mu\text{mol}$ ) was added to the reaction mixture and stirred for 2 h. 1N HCl aq (2 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic layer was collected by using ISOLUTE<sup>®</sup> Phase Separator (3 mL), and the solvent was evaporated. The residue was subjected to preparative GPC to give **1(200)** as a beige solid (41.1 mg, 84%). <sup>1</sup>H NMR (CDCl<sub>3</sub>)  $\delta$  4.75 (H<sub>1-B</sub>, 200 $\times$ 4H, br s), 3.40 (H<sub>1-C</sub>, 200 $\times$ 2H, br s), 2.32 (H<sub>1-A</sub> and H<sub>1-B</sub>, (200 $\times$ 6+3)H, br s), 1.85–1.38 (H<sub>1-D</sub>, 200 $\times$ 2H, br m), 1.21 (H<sub>1-F</sub>, 200 $\times$ 6H, br s), 0.87 (H<sub>1-E</sub>, 200 $\times$ 6H, br s), small peaks originated from end-group were observed in 10.02 (H<sub>t-A</sub>, 1H, s) and 7.85–7.67 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol):  $M_n = 6.93 \times 10^4$ ,  $M_w/M_n = 1.11$ .

**Scheme S1.** Synthesis of polymer **1(200)**



**Figure S1.** Structure of polymer **1(200)** with <sup>1</sup>H NMR assignment.

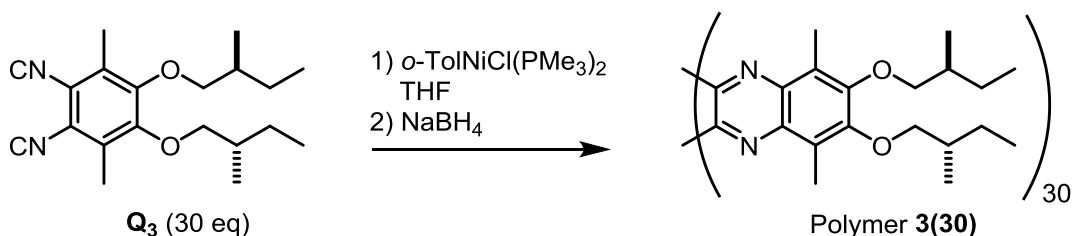
### Synthesis of polymers 3(n):



**Figure S2.** Structure of polymer **3(n)** with  $^1\text{H}$  NMR assignment.

**Synthesis of polymer 3(30):** **Q<sub>3</sub>** (19.71 mg, 60.0  $\mu\text{mol}$ ) was dissolved in THF (2 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 223.3  $\mu\text{L}$ , 2.00  $\mu\text{mol}$ ) was added to the monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (15.9 mg, 420  $\mu\text{mol}$ ) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(30)** as a beige solid (18.4 mg, 94%).  $^1\text{H}$  NMR (CDCl<sub>3</sub>)  $\delta$  4.54–3.25 (H<sub>3-B</sub>, 30 $\times$ 4H, br m), 2.15 (H<sub>3-A</sub> and H<sub>t-B</sub>, (30 $\times$ 6+3)H, br s), 1.83 (H<sub>3-C</sub>, 30 $\times$ 2H, br s), 1.59–1.49 (H<sub>3-D</sub>, 30 $\times$ 2H, br s), 1.27–1.09 (H<sub>3-D</sub>, 30 $\times$ 2H, br m), 1.01 (H<sub>3-F</sub>, 30 $\times$ 6H, d,  $J$  = 6.0 Hz), 0.91 (H<sub>3-E</sub>, 30 $\times$ 6H, t,  $J$  = 6.8 Hz), small peaks originated from end-group were observed in 9.95 (H<sub>t-A</sub>, 1H, s) and 7.83–7.10 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol):  $M_n$  =  $6.81 \times 10^3$ ,  $M_w/M_n$  = 1.13.

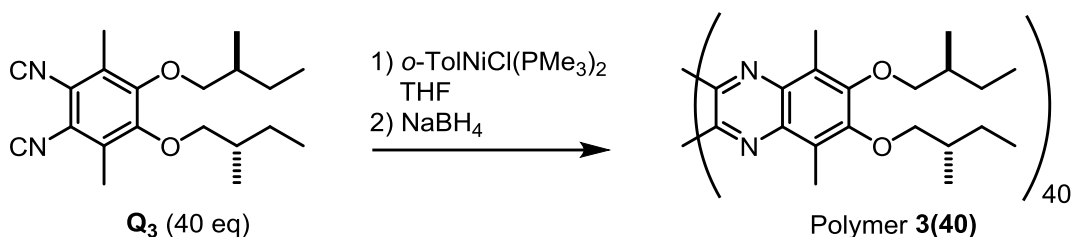
### Scheme S2. Synthesis of polymer 3(30)



**Synthesis of polymer 3(40):** **Q<sub>3</sub>** (19.71 mg, 60.0  $\mu\text{mol}$ ) was dissolved in THF (2 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 167.5  $\mu\text{L}$ , 1.50  $\mu\text{mol}$ ) was added to the

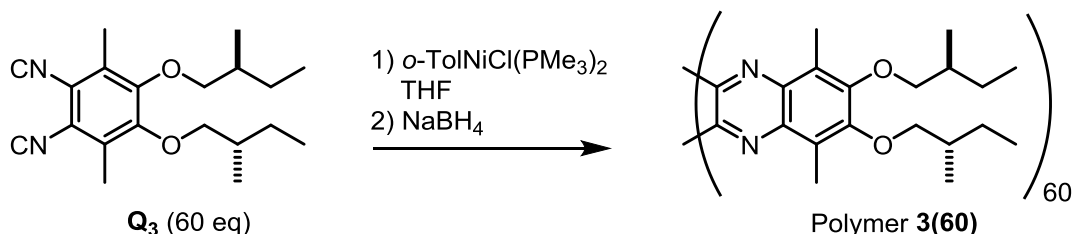
monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (15.9 mg, 420 μmol) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(40)** as a beige solid (17.1 mg, 87%). <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 4.53–3.33 (H<sub>3-B</sub>, 40×4H, br m), 2.16 (H<sub>3-A</sub> and H<sub>t-B</sub>, (40×6+3)H, br s), 1.83 (H<sub>3-C</sub>, 40×2H, br s), 1.55 (H<sub>1-D</sub>, 40×2H, br s), 1.29–1.08 (H<sub>3-D</sub>, 40×2H, br m), 1.02 (H<sub>3-F</sub>, 40×6H, d, *J* = 6.0 Hz), 0.91 (H<sub>3-E</sub>, 40×6H, t, *J* = 6.8 Hz), small peaks originated from end-group were observed in 9.94 (H<sub>t-A</sub>, 1H, s) and 7.84–7.10 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol): *M*<sub>n</sub> = 9.55 × 10<sup>3</sup>, *M*<sub>w</sub>/*M*<sub>n</sub> = 1.12.

**Scheme S3.** Synthesis of polymer **3(40)**



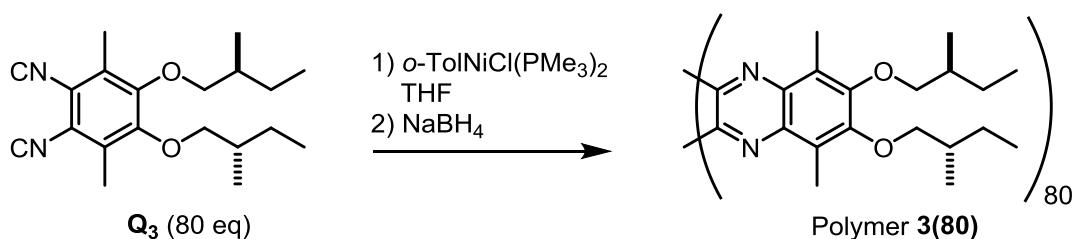
**Synthesis of polymer 3(60):** **Q<sub>3</sub>** (19.71 mg, 60.0 μmol) was dissolved in THF (2 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 111.7 μL, 1.00 μmol) was added to the monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (7.94 mg, 210 μmol) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(60)** as a beige solid (18.3 mg, 93%). <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 4.38–3.20 (H<sub>3-B</sub>, 60×4H, br m), 3.59 (H<sub>3-B</sub>, 60×2H, br s), 2.17 (H<sub>3-A</sub> and H<sub>t-B</sub>, (60×6+3)H, br s), 1.84 (H<sub>3-C</sub>, 60×2H, br s), 1.56 (H<sub>3-D</sub>, 60×2H, br s), 1.35–1.14 (H<sub>3-D</sub>, 60×2H, br m), 1.02 (H<sub>3-F</sub>, 60×6H, d, *J* = 6.0 Hz), 0.92 (H<sub>3-E</sub>, 60×6H, t, *J* = 6.8 Hz), small peaks originated from end-group were observed in 9.94 (H<sub>t-A</sub>, 1H, s) and 7.83–7.11 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol): *M*<sub>n</sub> = 1.41 × 10<sup>4</sup>, *M*<sub>w</sub>/*M*<sub>n</sub> = 1.12.

#### Scheme S4. Synthesis of polymer 3(60)



**Synthesis of polymer 3(80):** **Q<sub>3</sub>** (29.56 mg, 90.0 μmol) was dissolved in THF (3 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 125.6 μL, 1.13 μmol) was added to the monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (8.93 mg, 236 μmol) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(80)** as a beige solid (28.3 mg, 96%). <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 3.83 (H<sub>3-B</sub>, 80×2H, br s), 3.59 (H<sub>3-B</sub>, 80×2H, br s), 2.17 (H<sub>3-A</sub> and H<sub>t-B</sub>, (80×6+3)H, br s), 1.84 (H<sub>3-C</sub>, 80×2H, br s), 1.57 (H<sub>3-D</sub>, 80×2H, br s), 1.32–1.13 (H<sub>3-D</sub>, 80×2H, br m), 1.02 (H<sub>3-F</sub>, 80×6H, d, *J* = 6.0 Hz), 0.92 (H<sub>3-E</sub>, 80×6H, t, *J* = 7.2 Hz), small peaks originated from end-group were observed in 10.00 (H<sub>t-A</sub>, 1H, s) and 7.82–7.11 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol): *M<sub>n</sub>* = 2.36 × 10<sup>4</sup>, *M<sub>w</sub>*/*M<sub>n</sub>* = 1.09.

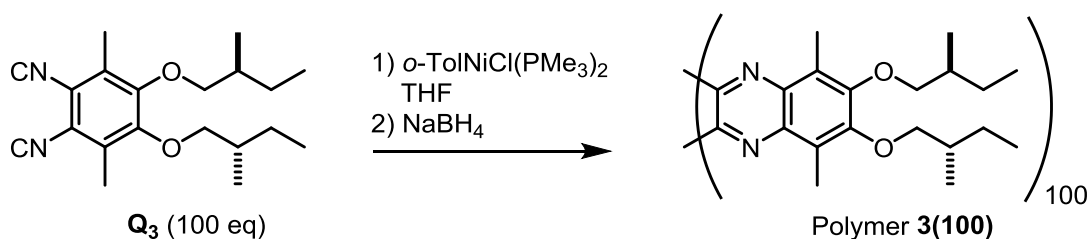
#### Scheme S5. Synthesis of polymer 3(80)



**Synthesis of polymer 3(100):** **Q<sub>3</sub>** (29.56 mg, 90.0 μmol) was dissolved in THF (3 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 100.5 μL, 0.90 μmol) was added to the monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (7.15 mg, 189 μmol) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the

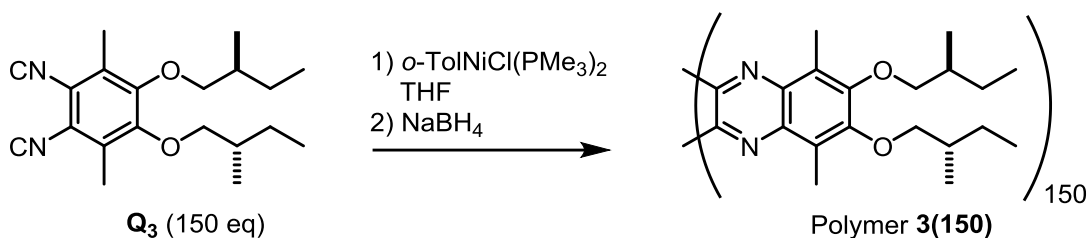
solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(100)** as a beige solid (24.5 mg, 83%). <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 3.83 (H<sub>3-B</sub>, 100×2H, br s), 3.59 (H<sub>3-B</sub>, 100×2H, br s), 2.17 (H<sub>3-A</sub> and H<sub>t-B</sub>, (100×6+3)H, br s), 1.84 (H<sub>3-C</sub>, 100×2H, br s), 1.57 (H<sub>3-D</sub>, 100×2H, br s), 1.30–1.15 (H<sub>3-D</sub>, 100×2H, br m), 1.02 (H<sub>3-F</sub>, 100×6H, d, *J* = 5.6 Hz), 0.92 (H<sub>3-E</sub>, 100×6H, t, *J* = 6.8 Hz), small peaks originated from end-group were observed in 10.06 (H<sub>t-A</sub>, 1H, s) and 7.82–7.11 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol): *M*<sub>n</sub> = 3.09 × 10<sup>4</sup>, *M*<sub>w</sub>/*M*<sub>n</sub> = 1.10.

**Scheme S6. Synthesis of polymer 3(100)**



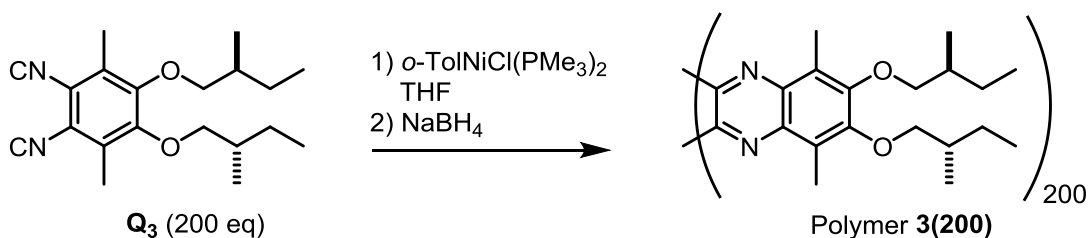
**Synthesis of polymer 3(150):** **Q<sub>3</sub>** (29.56 mg, 90.0 μmol) was dissolved in THF (3 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 67.0 μL, 0.60 μmol) was added to the monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (4.77 mg, 126 μmol) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(150)** as a beige solid (28.1 mg, 95%). <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 3.83 (H<sub>3-B</sub>, 150×2H, br s), 3.59 (H<sub>3-B</sub>, 150×2H, br s), 2.17 (H<sub>3-A</sub> and H<sub>t-B</sub>, (150×6+3)H, br s), 1.84 (H<sub>3-C</sub>, 150×2H, br s), 1.56 (H<sub>3-D</sub>, 150×2H, br s), 1.22 (H<sub>3-D</sub>, 150×2H, br s), 1.02 (H<sub>3-F</sub>, 150×6H, br s), 0.92 (H<sub>3-E</sub>, 150×6H, br s), small peaks originated from end-group were observed in 10.02 (H<sub>t-A</sub>, 1H, s) and 7.82–7.11 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol): *M*<sub>n</sub> = 5.26 × 10<sup>4</sup>, *M*<sub>w</sub>/*M*<sub>n</sub> = 1.08.

### Scheme S7. Synthesis of polymer 3(150)



**Synthesis of polymer 3(200):** **Q<sub>3</sub>** (29.56 mg, 90.0 μmol) was dissolved in THF (3 mL). A THF solution of *o*-TolNiCl(PMe<sub>3</sub>)<sub>2</sub> (8.96 mM, 50.3 μL, 0.450 μmol) was added to the monomer solution with vigorous stirring. After 120 h, NaBH<sub>4</sub> (3.57 mg, 94.5 μmol) was added to the reaction mixture and stirred for 1 h. Water (20 mL) was added to the solution and extracted with CH<sub>2</sub>Cl<sub>2</sub> (20 ml). The organic extract was washed with brine (20 mL), dried over Na<sub>2</sub>SO<sub>4</sub>, and the solvent was evaporated. The residue was subjected to preparative GPC to give **3(200)** as a beige solid (26.1 mg, 88%). <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 3.83 (H<sub>3-B</sub>, 200×2H, br s), 3.59 (H<sub>3-B</sub>, 200×2H, br s), 2.16 (H<sub>3-A</sub> and H<sub>t-B</sub>, (200×6+3)H, br s), 1.84 (H<sub>3-C</sub>, 200×2H, br s), 1.56 (H<sub>3-D</sub>, 200×2H, br s), 1.22 (H<sub>3-D</sub>, 200×2H, br s), 1.02 (H<sub>3-F</sub>, 200×6H, br s), 0.92 (H<sub>3-E</sub>, 200×6H, br s), small peaks originated from end-group were observed in 10.06 (H<sub>t-A</sub>, 1H, s) and 7.83–7.11 ppm (H<sub>t-C</sub>, 4H, m); GPC (CHCl<sub>3</sub>, g/mol):  $M_n = 8.15 \times 10^4$ ,  $M_w/M_n = 1.07$ .

### Scheme S8. Synthesis of polymer 3(200)



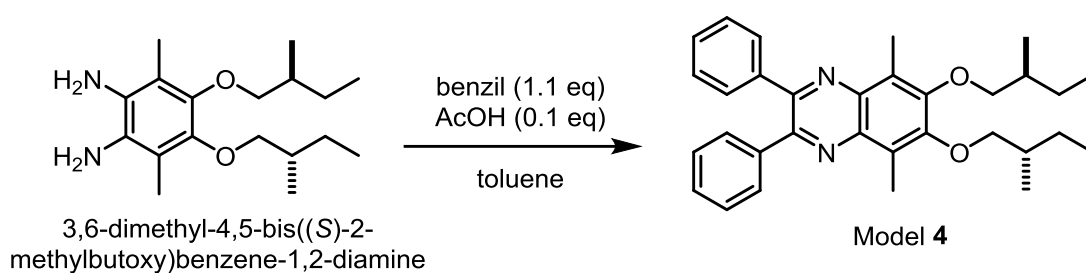
### Synthesis of monomeric model compound 4:

To a solution of 3,6-dimethyl-4,5-bis((*S*)-2-methylbutoxy)benzene-1,2-diamine (92.1 mg, 0.299 mmol) and acetic acid (1.7 μL, 29.9 μmol) in toluene was added benzil (69.1 mg, 0.329 mmol). The mixture was stirred at 120 °C for 41 h. The reaction mixture was concentrated under reduced pressure. The residue was purified with silica gel column chromatography (hexane/AcOEt = 95/5) to give **4** (121.3 mg, 84%) as orange solid. <sup>1</sup>H



NMR (CDCl<sub>3</sub>)  $\delta$  7.58–7.56 (4H, m), 7.35–7.31 (6H, m), 3.95 (2H, dd,  $J = 6.0, 9.2$  Hz), 3.83 (2H, dd,  $J = 6.4, 8.8$  Hz), 2.73 (6H, s), 2.01–1.89 (2H, m), 1.73–1.63 (2H, m), 1.38–1.26 (2H, m), 1.12 (6H, d,  $J = 6.4$  Hz), 1.00 (6H, t,  $J = 7.6$  Hz); <sup>13</sup>C NMR (CDCl<sub>3</sub>):  $\delta$  153.1, 150.0, 139.8, 138.6, 130.1, 128.3, 128.0, 126.0, 78.7, 36.0, 26.3, 16.7, 11.5, 10.0; IR(ATR) 2961, 1348, 1200, 1132, 698 cm<sup>-1</sup>; HRMS(APCI)  $m/z$  calcd for C<sub>32</sub>H<sub>38</sub>N<sub>2</sub>O<sub>2</sub> (M+H<sup>+</sup>): 483.3006, Found: 483.2994; [ $\alpha$ ]<sub>D</sub><sup>24.1</sup> +12.46 (c 1.33, CHCl<sub>3</sub>).

**Scheme S9.** Synthesis of monomeric model compound **4**



### 3 Circularly Polarized Luminescence (CPL) Measurement of Polymers 1(200)–3(200) and Monomeric Model Compound 4

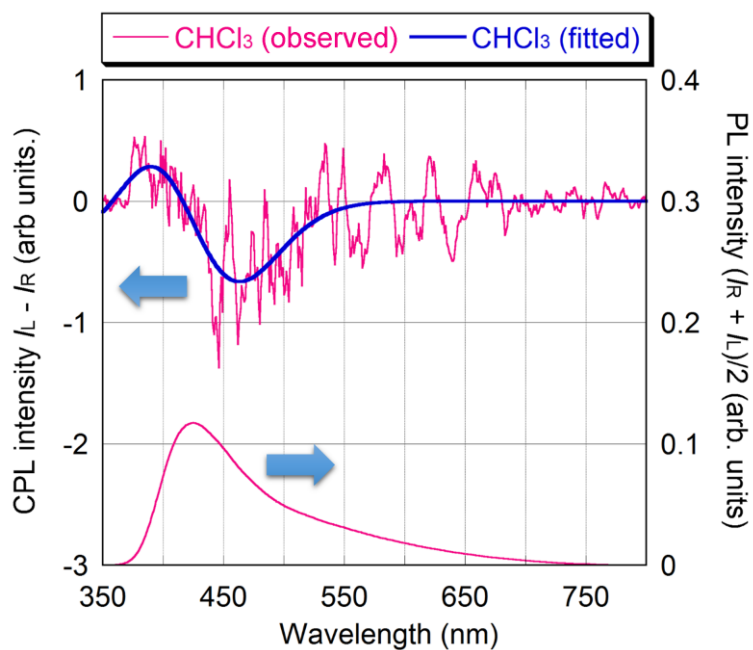
The CPL spectra were recorded at room temperature on a JASCO CPL-200 with an SQ-grade quartz cuvette (a path length of 10 mm). A scanning rate of 50 nm/min, an excitation bandwidth of 3000  $\mu\text{m}$ , a monitoring bandwidth of 3000  $\mu\text{m}$ , a response time of 8 seconds, and 20 times accumulation were employed. The CPL dissymmetry factor  $g_{\text{lum}}$  is defined as  $g_{\text{lum}} = 2(I_L - I_R)/(I_L + I_R)$ , where  $I_L$  and  $I_R$  are the fluorescence intensities of the right- and left-handed circularly polarized light, respectively. The value of  $g_{\text{lum}}$  can be calculated on a JASCO CPL-200 as follows.

$$\begin{aligned} g_{\text{lum}} &= [\text{PL ellipticity (mdeg)}] / [\text{PL intensity (V)}] / (1000 \times 180 / 4\pi) \\ &= [\text{PL ellipticity (mdeg)}] / [\text{PL intensity (V)}] \times (6.98 \times 10^{-5}) \end{aligned}$$

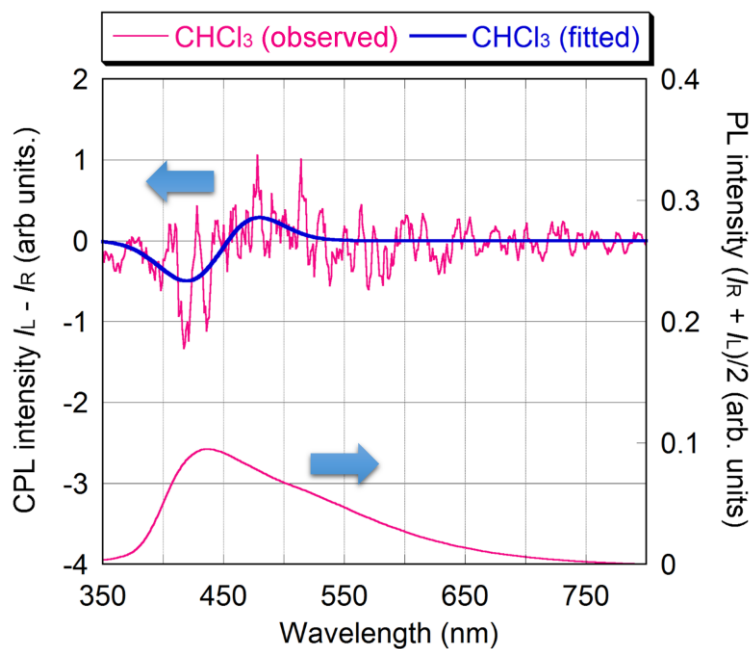
The validity of the coefficient ( $6.98 \times 10^{-5}$ ) was also confirmed by the measurement of previously reported CPL materials<sup>5</sup> (europium(III) ions coordinated by chiral *N,N'*-bis(1-phenylethyl)-2,6-pyridinedicarboxamide in MeCN). For a curve fitting of observed CPL spectra, following equation was adopted on the basis of the supposition that the CPL signals are expressed as a linear combination of two Gaussian distribution functions of the wavenumber.

$$I_L - I_R = [a_1 \times \exp( b_1 / (\lambda - c_1)^2 )] + [a_2 \times \exp( b_2 / (\lambda - c_2)^2 )]$$

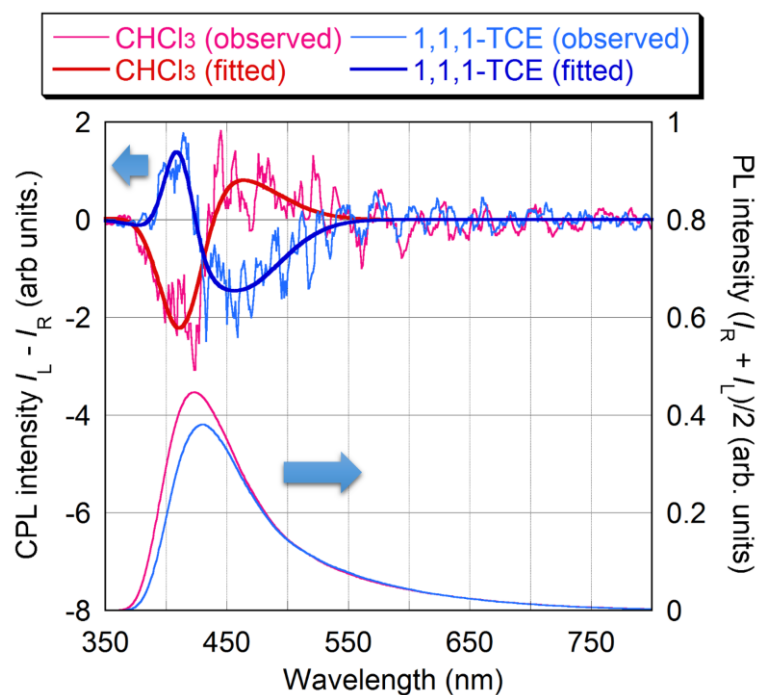
where  $\lambda$  is the wavelength (i.e.  $1/\lambda$  is the wavenumber),  $a_1$ ,  $a_2$ ,  $b_1$ ,  $b_2$ ,  $c_1$ , and  $c_2$  are variables for the curve fitting. Nonlinear least-squares fitting of  $I_L - I_R$  versus  $\lambda$  was performed by using the Solver Function in Microsoft Office Excel 2013. Sums of the squares of the deviation were minimized by varying 6 parameters ( $a_1$ ,  $a_2$ ,  $b_1$ ,  $b_2$ ,  $c_1$ , and  $c_2$ ).



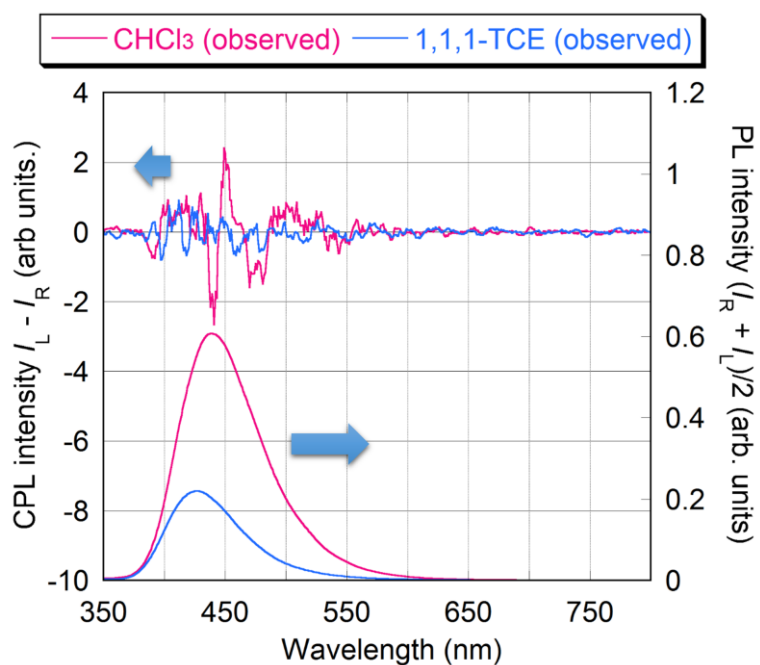
**Figure S3.** CPL and PL spectra of polymer **1(200)** in dilute ( $2.12 \times 10^{-2}$  g/L)  $\text{CHCl}_3$  solution ( $\lambda_{\text{ex}} = 300.0$  nm).



**Figure S4.** CPL and PL spectra of polymer **2(200)** in dilute ( $1.98 \times 10^{-2}$  g/L)  $\text{CHCl}_3$  solution ( $\lambda_{\text{ex}} = 300.0$  nm).



**Figure S5.** CPL and PL spectra of polymer **3(200)** in dilute ( $2.98 \times 10^{-2}$  g/L)  $\text{CHCl}_3$  and 1,1,1-TCE solutions ( $\lambda_{\text{ex}} = 300.0$  nm).



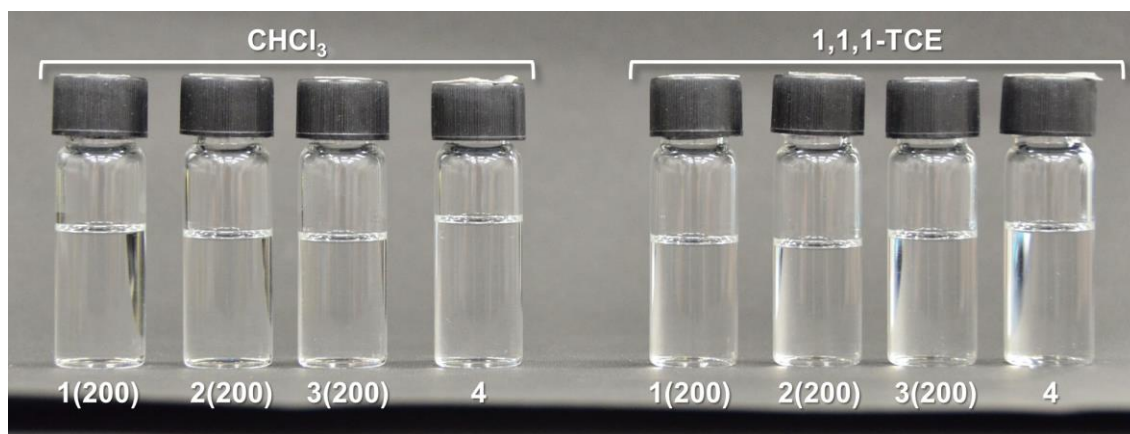
**Figure S6.** CPL and PL spectra of **4** in dilute ( $4.82 \times 10^{-3}$  g/L)  $\text{CHCl}_3$  and 1,1,1-TCE solutions ( $\lambda_{\text{ex}} = 300.0$  nm).

## 4 Absolute Photoluminescence Quantum Yields of New Compounds

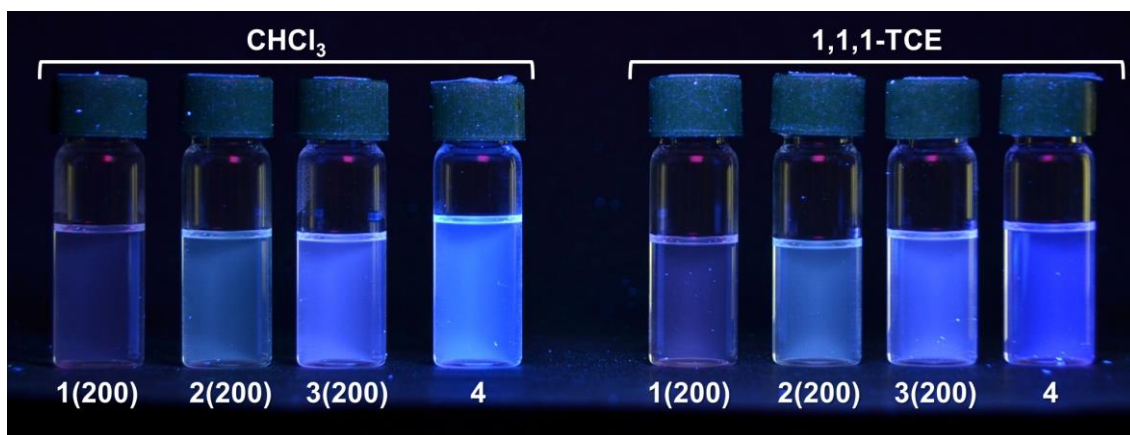
**Table S1.** Absolute photoluminescence (PL) quantum yields of compounds in CHCl<sub>3</sub> or 1,1,1-TCE.

Compound	Solvent	Concentration (g/L)	Excitation wavelength (nm)	PL quantum yield (%)
<b>1(200)</b>	CHCl <sub>3</sub>	$1.60 \times 10^{-2}$	350.0	0.3
<b>1(200)</b>	1,1,1-TCE	$1.60 \times 10^{-2}$	350.0	0.3
<b>2(200)</b>	CHCl <sub>3</sub>	$2.36 \times 10^{-2}$	356.5	0.3
<b>2(200)</b>	1,1,1-TCE	$2.36 \times 10^{-2}$	357.0	0.4
<b>3(200)</b>	CHCl <sub>3</sub>	$2.12 \times 10^{-2}$	356.5	0.7
<b>3(200)</b>	1,1,1-TCE	$2.12 \times 10^{-2}$	359.5	0.8
<b>4</b>	CHCl <sub>3</sub>	$4.82 \times 10^{-3}$	362.0	3.3

## 5 Photograph of 1(200)-3(200) and 4 in dilute $\text{CHCl}_3$ and 1,1,1-TCE solutions

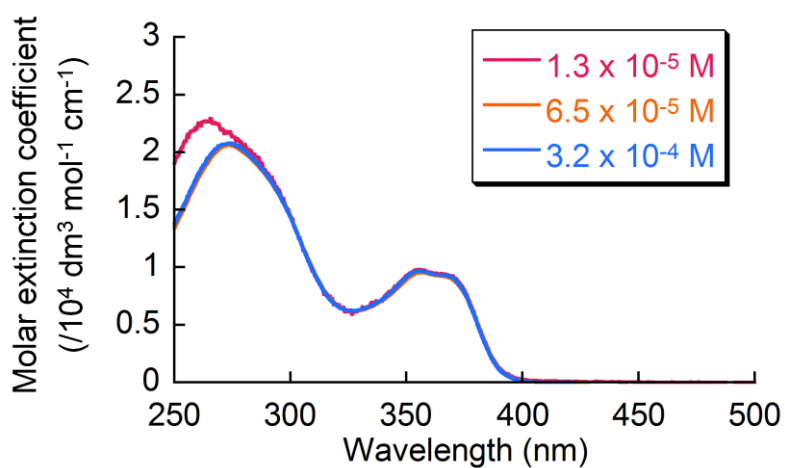


**Figure S7.** Photograph of 1(200)-3(200) and 4 in  $\text{CHCl}_3$  and 1,1,1-TCE ( $1.0 \times 10^{-5}$  M) under ambient light.

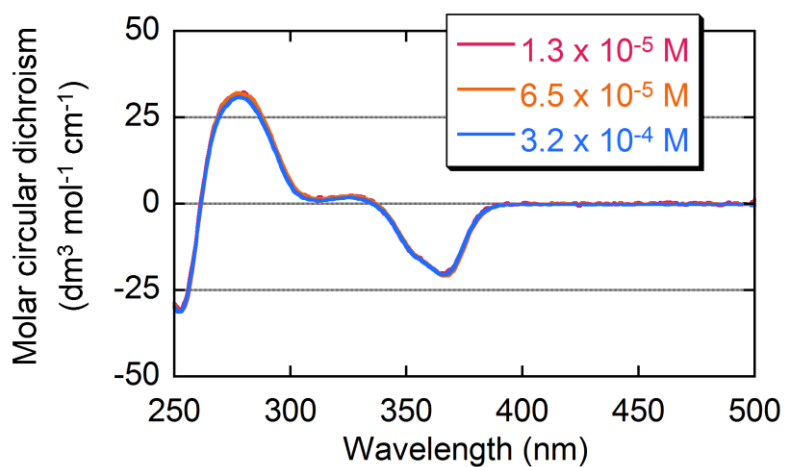


**Figure S8.** Photograph of 1(200)-3(200) and 4 in  $\text{CHCl}_3$  and 1,1,1-TCE ( $1.0 \times 10^{-5}$  M) under UV light irradiation ( $\lambda_{\text{ex}} = 365$  nm).

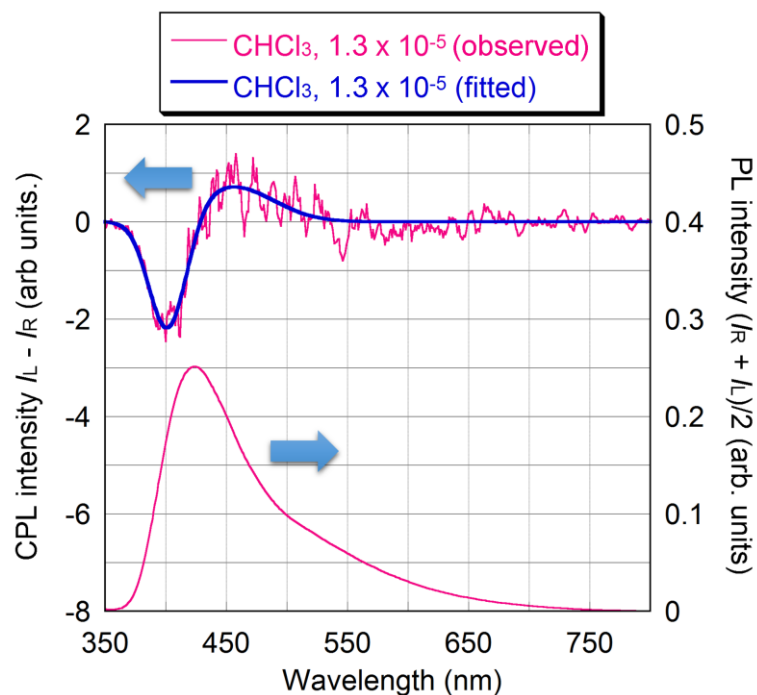
## 6 Effect of Concentration of Polymer 3(200) in CHCl<sub>3</sub> and 1,1,1-TCE Solutions



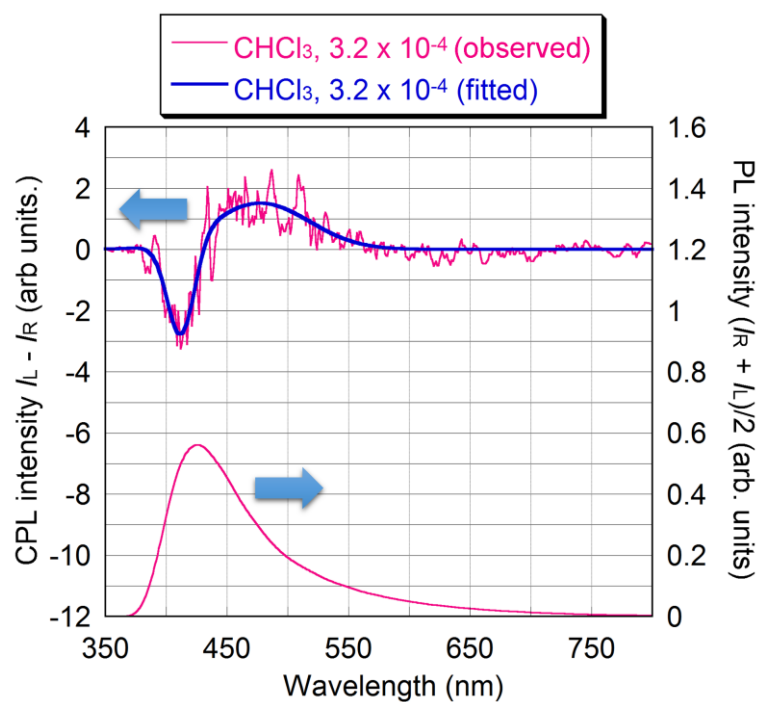
**Figure S9.** UV-absorption spectra of polymer 3(200) at various concentrations ( $1.3 \times 10^{-5}$ ,  $6.5 \times 10^{-5}$ , and  $3.2 \times 10^{-4}$  M) in CHCl<sub>3</sub>.



**Figure S10.** CD spectra of polymer 3(200) at various concentrations ( $1.3 \times 10^{-5}$ ,  $6.5 \times 10^{-5}$ , and  $3.2 \times 10^{-4}$  M) in CHCl<sub>3</sub>.

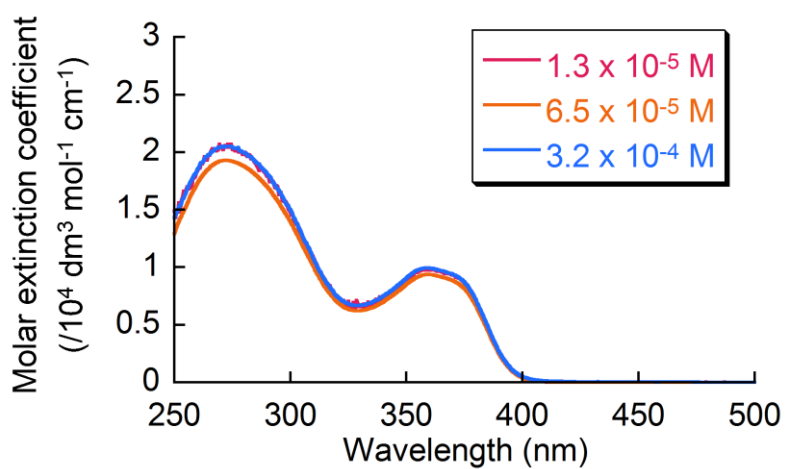


**Figure S11.** CPL and PL spectra of polymer **3(200)** in  $\text{CHCl}_3$  ( $1.3 \times 10^{-5}$  M,  $\lambda_{\text{ex}} = 300.0$  nm).

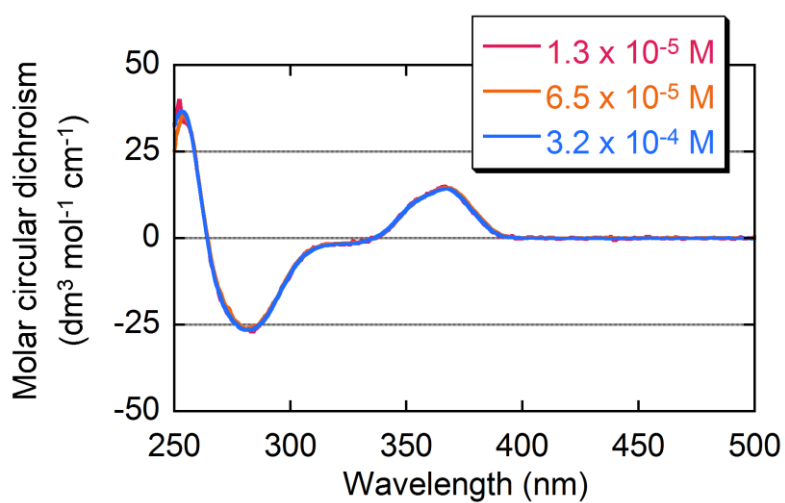


**Figure S12.** CPL and PL spectra of polymer **3(200)** in  $\text{CHCl}_3$  ( $3.2 \times 10^{-4}$  M,  $\lambda_{\text{ex}} = 300.0$  nm).

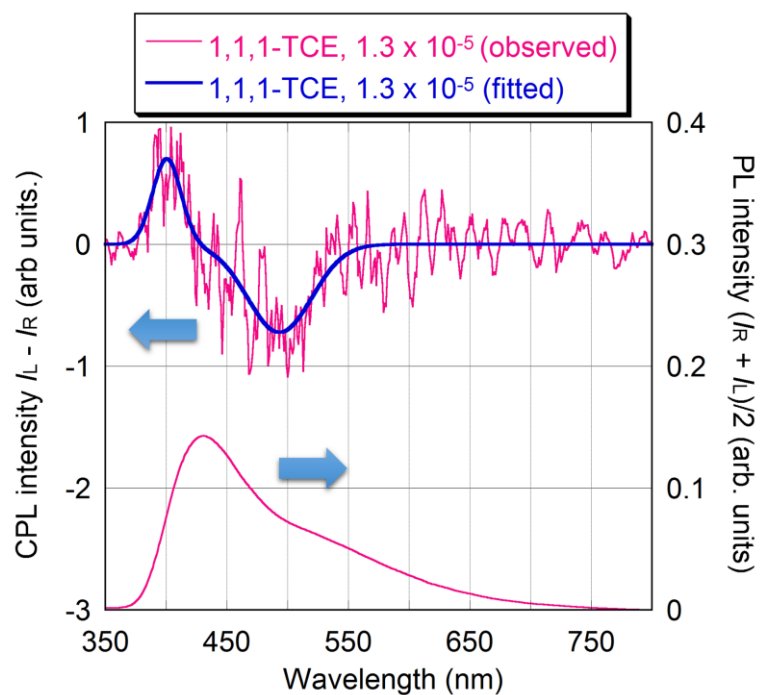




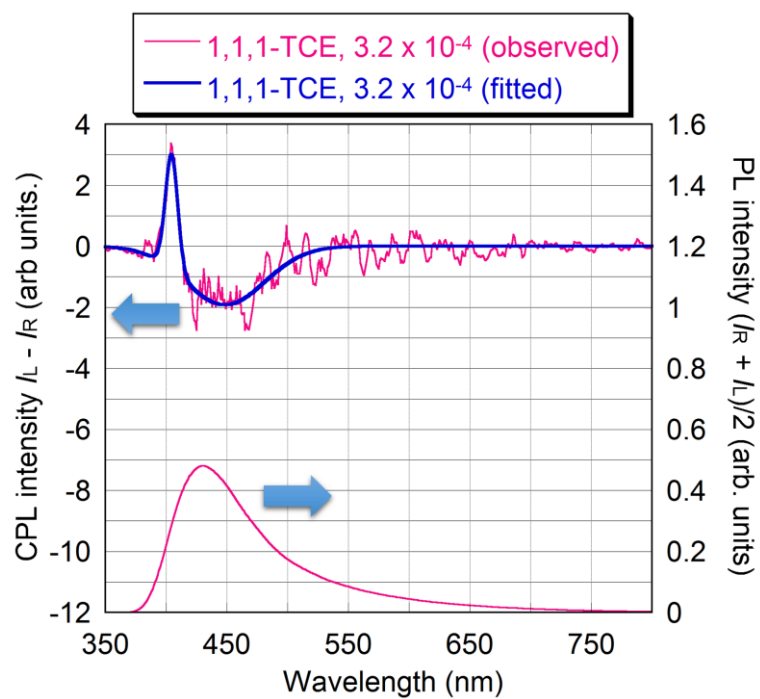
**Figure S13.** UV-absorption spectra of polymer **3(200)** at various concentrations ( $1.3 \times 10^{-5}$ ,  $6.5 \times 10^{-5}$ , and  $3.2 \times 10^{-4} \text{ M}$ ) in 1,1,1-TCE.



**Figure S14.** CD spectra of polymer **3(200)** at various concentrations ( $1.3 \times 10^{-5}$ ,  $6.5 \times 10^{-5}$ , and  $3.2 \times 10^{-4} \text{ M}$ ) in 1,1,1-TCE.

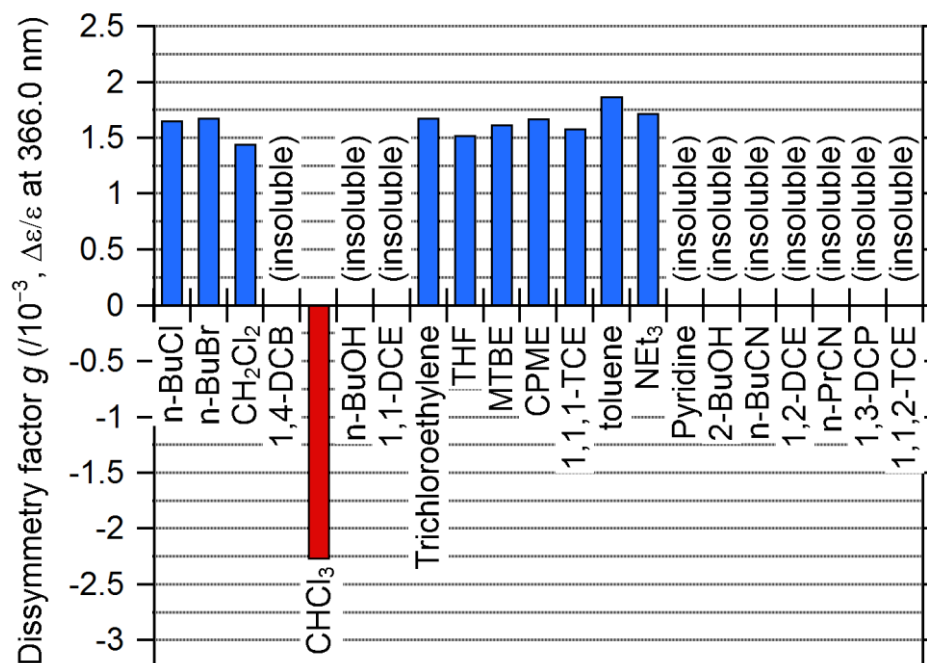


**Figure S15.** CPL and PL spectra of polymer **3(200)** in 1,1,1-TCE ( $1.3 \times 10^{-5}$  M,  $\lambda_{\text{ex}} = 300.0$  nm).



**Figure S16.** CPL and PL spectra of polymer **3(200)** in 1,1,1-TCE ( $3.2 \times 10^{-4}$  M,  $\lambda_{\text{ex}} = 300.0$  nm).

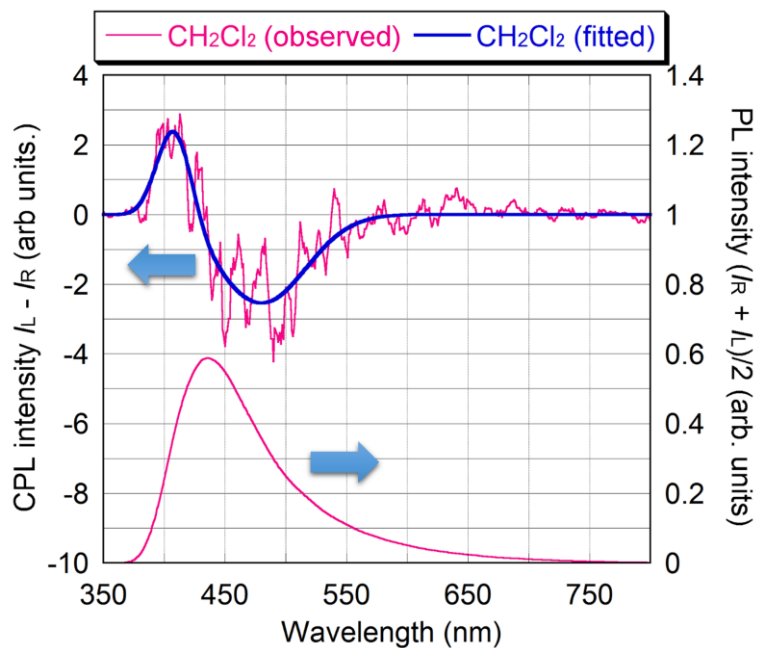
## 7 Optical Properties of Polymer 3(200) in Various Solvents



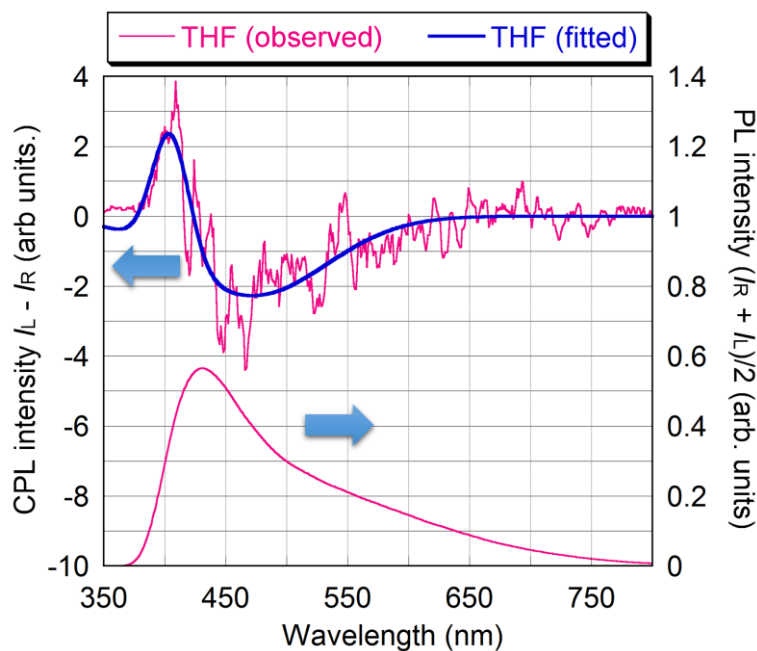
**Figure S17.** Dissymmetry factors  $g$  at 366.0 nm of polymer **3(200)** in various solvents.

DCE: dichloroethane, DCP: dichloropropane, DCB: dichlorobutane, TCE:

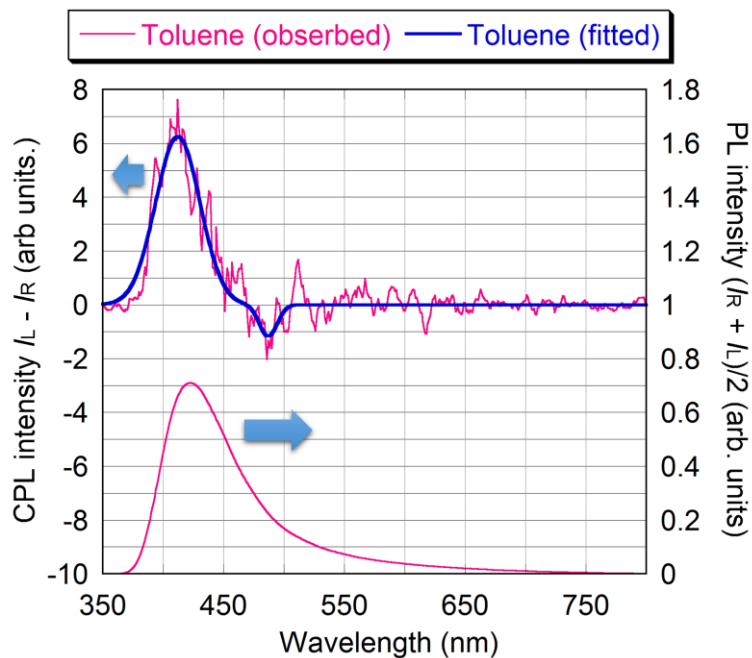
trichloroethane, MTBE: methyl *tert*-butyl ether, and CPME: cyclopentyl methyl ether.



**Figure S18.** CPL and PL spectra of polymer **3(200)** in dilute ( $2.22 \times 10^{-2}$  g/L)  $\text{CH}_2\text{Cl}_2$  solution ( $\lambda_{\text{ex}} = 300.0$  nm).



**Figure S19.** CPL and PL spectra of polymer **3(200)** in dilute ( $2.22 \times 10^{-2}$  g/L) THF solution ( $\lambda_{\text{ex}} = 300.0$  nm).



**Figure S20.** CPL and PL spectra of polymer **3(200)** in dilute ( $2.22 \times 10^{-2}$  g/L) toluene solution ( $\lambda_{\text{ex}} = 300.0$  nm).

## 8 References

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4. Nagata, Y.; Yamada, T.; Adachi, T.; Akai, Y.; Yamamoto, T.; Suginome, M. *J. Am. Chem. Soc.* **2013**, *135*, 10104.
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