Supporting Information

Thermotropic liquid crystals based on 1,8,9,16-tetrasubstituted tetraphenylenes and their structure-property relationship studies

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Experimental Section

All reagents and solvents were reagent grade. Further purification and drying following the guidelines of Armarego[1] were used when necessary. Organic solvents were concentrated under reduced pressure on a rotary evaporator. Chromatographic purification of products was performed on Macherey Nagel Kieselgel 60M (230-400 mesh). Thin-layer chromatography (TLC) was performed on E. Merck silica gel 60 F_{254} (0.1 mm thickness) coated on aluminum plates. Visualization of the developed chromatogram was performed by a spray of 5% w/v dodecamolybdophosphoric acid in ethanol and subsequent heating. Melting points were measured with a Reichert apparatus in Celsius degrees and are uncorrected. Optical rotations were obtained with a Perkin-Elmer model 341 polarimeter, operating at 589 nm. Nuclear magnetic resonance (NMR) spectra were recorded with a Bruker DPX300 NMR spectrometer at 300 MHz (^{1}H) or at 75 MHz (^{13}C) or a Bruker ADVANCE-III NMR spectrometer at 400 MHz (^{1}H) or at 100 MHz (^{13}C). All NMR measurements were carried out at room temperature in deuterated solution and were internally referenced to residual proton solvent signals (note: $CDCl_3$ referenced at δ 7.26 in ^{1}H, and δ 77.16 for central line of the triplet in ^{13}C; Acetone-d_6 referenced at δ 2.05 in ^{1}H, and δ 29.84 for central line of the septet in ^{13}C; DMSO-d_6 referenced at δ 2.50 in ^{1}H, and δ 39.52 for central line of the quintet in ^{13}C; CD_2Cl_2 referenced at δ 5.32 in ^{1}H, and δ 53.10 for central line of the quintet in ^{13}C). Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, brs = broad singlet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, m = multiplet), integration, coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported in terms of chemical shift. Mass spectrometry (MS) and high resolution mass spectrometry (HRMS) were measured on a ThermoFinnigan MAT 95XL and Bruker Autoflex MALDI-TOF MS. X-ray analyses were obtained by SHELXTL PLUS (PC version) with Bruker AXS Kappa ApexII Duo diffractometer unless otherwise stated. Structure solution and refinement were carried out by SHELX-97 suite program.[2] Differential Scanning Calorimetry was carried out by

Perkin Elmer Pyris Diamond DSC. Polarizing Optical Microscopy (POM) was performed by NIKON Polarizing Microscope 501POL and INSTEC STC200 Hot Plate (HCS302). The *in situ* variable temperature diffractographs were recorded on a Bruker D8 ADVANCE diffractometer (HKU) with CuKa X-ray radiation (λ = 1.54146 Å, rated at 1.6 kW) equipped with a modular temperature chamber attachment (Material Research Instruments) operated at 30-200˚C. Scan range = 3-30˚ (2θ), step size = 0.05˚, scan speed = 1 s per step were used. TEM and SAED were performed on a Philips Tecnai G2 20 S-TWIN transmission electron microscope with an accelerating voltage of 200 kV. The TEM images and SAED patterns were taken by Gatan MultiScan Camera Model 794. TEM samples were prepared by depositing a few drops of suspensions on the formvar-coated copper grids and the excess solvent was removed by a piece of filter paper. The SEM images were taken on a Hitachi S-4800 field emission scanning electron microscope operating at 3.0 kV. SEM samples were prepared by drop-casting suspensions onto silicon wafers. Gold sputtering was applied before SEM observations.

Syntheses of Compounds

*1.1. Resolution of 1,8,9,16-Tetrahydroxytetraphenylene (**1**)*

To a solution of compound (±)–**1** (1 g, 2.7 mmol) in dry THF (30 mL) and Et_3N (3.7 mL, 27 mmol), (1*S*)–camphor-10-sulfonyl chloride (3.2 g, 12.2 mmol) was added at 0˚C. The mixture was warmed to room temperature and stirred overnight. The mixture was diluted with water (100 mL) and extracted with CH_2Cl_2 (20 mL × 3). The combined organic layer was washed with aqueous HCl solution (2 M, 30 mL × 2) successively and was then dried over anhydrous Na_2SO_4. After concentration, the residue was purified by column chromatography on silica gel (150 g, EtOAc/toluene, 1:4) to give the two diastereomers. The less polar fraction isolated was diastereomer (*S*,*S*)–**2b** (1.55 g, 47%) as colorless solids: m.p. 167-168˚C (lit:[3] 168-169˚C); ^1H NMR (400 MHz, $CDCl_3$): δ 0.80 (12H, s, 4×CH_3), 1.04 (12H, s, 4×CH_3), 1.35-1.42 (4H, m, 2×CH_2), 1.48-1.55 (4H, m, 2×CH_2), 1.88 (2H, s, CH_2), 1.93 (2H, s, CH_2), 1.95-2.04 (4H, m, 2×CH_2), 2.06-2.08 (4H, m, 2×CH_2), 2.29-2.38 (8H, m, 4×CH_2), 2.99 (4H, d, *J* = 15Hz, 2×SCH_2), 3.15 (4H, d, *J* = 15Hz, 2×SCH_2), 7.30-7.37 (8H, m, ArH), 7.38-7.40 (4H, m, ArH); MS (ESI) *m/z* 1247 $[M+Na]^+$.

The more polar fraction isolated was diastereomer (*R*,*R*)–**2a** (1.48 g, 45%) as colorless solids: m.p. 159-160°C (lit:[3] 160-161°C); ^1H NMR (400 MHz, CD_2Cl_2): δ 0.77 (12H, s, 4×CH_3), 0.94 (12H, s, 4×CH_3), 1.38-1.43 (4H, m, 2×CH_2), 1.52-1.59 (8H, m, 4×CH_2), 1.88 (2H, s, CH_2), 1.89 (2H, s, CH_2), 1.93-2.02 (6H, m, 3×CH_2), 2.05-2.09 (6H, m, 3×CH_2), 2.31-2.38 (4H, m, 2×CH_2), 2.80 (4H, d, *J* = 15Hz, 2×SCH_2), 3.53 (4H, d, *J* = 15Hz, 2×SCH_2), 7.19-7.22 (4H, m, ArH), 7.36-7.41 (8H, m, ArH); MS (ESI) *m/z* 1247 $[M+Na]^+$.

To a suspension of the diastereomer (*R*,*R*)–**2a** or (*S*,*S*)–**2b** (2 g, 1.6 mmol) in methanol (150 mL) was added aqueous KOH solution (3 M, 30 mL). The mixture was heated to reflux overnight. After solvent was evaporated, the mixture was diluted with water (50 mL) and extracted with EtOAc (30 mL × 3) successively. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated. After column chromatography on silica gel (100 g, EtOAc/CH_2Cl_2, 1:8), pure (*R*,*R*)–**1** (571 mg, 94%) or (*S*,*S*)–**1** (590 mg, 97%) was isolated: m.p. 196-197°C (lit:[3] 197-198°C); R_f = 0.31, (EtOAc/*n*-hexanes, 1:4); ^1H NMR (300 MHz, acetone-d_6): δ 3.65 (6H, s, 2×OCH_3), 6.65-6.70 (4H, m, ArH) 6.80 (2H, dd, *J* = 1.2, 7.8Hz, ArH), 6.84 (2H, d, *J* = 8.4Hz, ArH), 7.02 (2H, dd, *J* = 7.8, 7.9Hz, ArH), 7.18 (2H, dd, *J* = 7.8, 7.9Hz, ArH); MS (EI) *m/z* 396 $[M]^+$; (*R*,*R*)–**1**: $[\alpha]_D^{20}$ = +53.3 (*c* = 0.83, MeOH) (lit:[3] $[\alpha]_D^{20}$ = + 55.3 (*c* = 1.07, MeOH)); (*S*,*S*)–**1:** $[\alpha]_D^{20}$ = – 54.8 (*c* = 0.93, MeOH) (lit:[3] $[\alpha]_D^{20}$ = – 55.8 (*c* = 1.05, MeOH)).

*1.2. (R,R)-1,8,9,16-Tetrabutoxytetraphenylene (**3**)*

To a stirring solution of compound (*R*,*R*)–**1** (56 mg, 0.15 mmol) in DMF (1.5 mL), Cs_2CO_3 (247 mg, 0.76 mmol) and TBAB (cat.) were added. After the injection of 1-chlorobutane (80 μL, 0.76 mmol), the mixture was heated to 80°C for 1 hour. The mixture was cooled and was diluted with water (20 mL). The mixture was then extracted by EtOAc (10 mL × 3) and washed with saturated brine solution (50 mL × 2) successively. The combined organic layer was dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by column chromatography on silica gel (5 g, CH_2Cl_2/*n*-hexanes, 1:4) to yield pure (*R*,*R*)–**3** (63 mg, 72%) as pale yellow sticky solids: R_f = 0.25 (CH_2Cl_2/*n*-hexanes, 1:4); ^1H NMR (400 MHz, CD_2Cl_2): δ 0.83 (3H, t, *J* = 7.4Hz, CH_3), 1.18-1.28 (2H, m, CH_2), 1.46-1.53 (2H, m, CH_2), 3.79 (2H, t, *J* = 6.5Hz, OCH_2), 6.76-6.82 (2H, m, ArH), 7.19 (1H, t, *J* = 6.5Hz, ArH); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 13.2, 18.7, 31.0, 68.0, 110.4, 120.1, 126.3, 127.3, 143.1, 155.9; HRMS (EI) *m/z* calcd for $C_{40}H_{48}O_4$ $[M]^+$ 592.3547, found 592.3544; (*R*,*R*)–**3**: $[\alpha]_D^{20}$ = +2.8 (*c* = 0.88, $CHCl_3$).

*1.3. 1,8,9,16-Tetrakis(heptyloxy)tetraphenylene and its (R,R)-Counterpart (**4**)*

Compound (*R*,*R*)–**4** or (±)–**4** was prepared from (*R*,*R*)–**1** or (±)–**1** (59 mg, 0.16 mmol) by using the same preparative method as that for compound (*R*,*R*)–**3**, except using 1-bromoheptane and without adding TBAB.

Column chromatography on silica gel (10 g, CH_2Cl_2/*n*-hexanes, 1:4) gave pure (*R*,*R*)–**4** (111 mg, 92%) as pale yellow waxy solids and (±)–4 was prepared similarly (92% yield): R_f = 0.29 (CH_2Cl_2/*n*-hexanes, 1:4); ^1H NMR (400 MHz, CD_2Cl_2): δ 0.91 (3H, t, *J* = 7.1Hz, CH_3), 1.22-1.32 (8H, m, 4×CH_2), 1.51-1.55 (2H, m, CH_2), 3.79 (2H, t, *J* = 6.5Hz, OCH_2), 6.77-6.83 (2H, m, ArH), 7.19 (1H, dd, *J* = 7.9, 8.0Hz, ArH); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 13.6, 22.3, 25.5, 28.7, 29.0, 31.5, 68.4, 110.4, 120.1, 126.4, 127.3, 143.2, 156.0; HRMS (ESI) *m/z* calcd for $C_{52}H_{72}O_4$ $[M+Na]^+$ 783.5323, found 783.5353; (*R*,*R*)–**4**: $[\alpha]_D^{20}$ = +30.4 (*c* = 0.97, $CHCl_3$).

*1.4. 1,8,9,16-Tetrakis(dodecyloxy)tetraphenylene and its (R,R) Counterpart (**5**)*

Compound (±)–**5** or (*R*,*R*)–**5** was prepared from (±)–**1** or (*R*,*R*)–**1** (50 mg, 0.13 mmol) by using the same preparative method as that for compound (*R*,*R*)–3, except using 1-bromododecane (138 mg, 0.56 mmol).

Column chromatography on silica gel (5 g, CH_2Cl_2/*n*-hexanes, 1:4) gave pure (*R*,*R*)–**5** (128 mg, 91%) as pale yellow waxy solids and (±)–**5** was prepared similarly (80%

yield): R_f = 0.35 (CH_2Cl_2/*n*-hexanes, 1:4); ^{1}H NMR (400 MHz, CD_2Cl_2): δ 0.89-0.92 (3H, m, CH_3), 1.20-1.35 (18H, m, 9×CH_2), 1.49-1.52 (2H, m, CH_2), 3.78 (2H, t, *J* = 6.5Hz, OCH_2), 6.76-6.82 (2H, m, ArH), 7.18 (1H, dd, *J* = 8.0, 8.1Hz, ArH); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 13.6, 22.4, 25.5, 29.0, 29.0, 29.1, 29.3, 29.3, 29.4, 29.4, 31.7, 68.4, 110.4, 120.1, 126.4, 127.3, 143.2, 156.0; HRMS (ESI) *m/z* calcd for $C_{72}H_{112}O_4$ $[M+Na]^+$ 1063.8453, found 1063.8471; (*R*,*R*)–**5:** $[\alpha]_D^{20}$ = +29.8 (*c* = 1.20, $CHCl_3$).

*1.5. 1,8,9,16-Tetrakis(2-(2-methoxyethoxy)ethoxy)tetraphenylene and its (R,R) counterpart (**6**)*

Compound (±)–**6** or (*R*,*R*)–**6** was prepared from (±)–**1** or (*R*,*R*)–**1** (59 mg, 0.16 mmol) by using the same preparative method as that for compound (*R*,*R*)–**3**, except using $H_3CO(CH_2CH_2O)_2Ts$ (193 mg, 0.71 mmol).

Column chromatography on silica gel (5 g, MeOH/$CHCl_3$, 1:40) gave pure (*R*,*R*)–**6** (93 mg, 75%) as pale yellow waxy solids and (±)–**6** was prepared similarly (68% yield): R_f = 0.35 (MeOH/$CHCl_3$, 1:40); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.33 (3H, s, OCH_2), 3.35-3.37 (4H, m, OCH_2), 3.47-3.63 (2H, m OCH_2), 3.63-3.92 (2H, m, OCH_2), 6.73 (1H, d, *J* = 8.1Hz, ArH), 6.80 (1H, d, *J* = 7.5Hz, ArH), 7.13 (1H, dd, *J* = 7.9, 8.0Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 59.1, 69.3, 69.7, 70.7, 72.1, 111.5, 121.1, 126.9, 128.0, 143.5, 156.1; HRMS (ESI) *m/z* calcd for $C_{44}H_{56}O_{12}$ $[M+Na]^+$ 799.3664, found 799.3647; (*R*,*R*)–**6:** $[\alpha]_D^{20}$ = +18.3 (*c* = 0.94, $CHCl_3$).

*1.6. 1,8,9,16-tetrakis(2,5,8,11-tetraoxatridecan-13-yloxy)tetra phenylene (**7**)*

Compound (±)–**7** was prepared from (±)–**1** (31 mg, 0.09 mmol) by using the same preparative method as that for compound (*R*,*R*)–**3**, except using $H_3C\tilde{O}(CH_2CH_2O)_4Ts$ (135 mg, 0.37 mmol).

Column chromatography on silica gel (5 g, MeOH/$CHCl_3$, 1:40) gave pure (±)–**7** (67 mg, 70%) as pale yellow waxy solids: R_f = 0.35 (MeOH/$CHCl_3$, 1:40); ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.36 (3H, s, OCH_2), 3.36 (2H, m, OCH_2), 3.37-3.55 (5H, m, OCH_2), 3.56-3.65 (7H, m, OCH_2), 3.87-3.90 (2H, m OCH_2), 6.73 (1H, d, *J* = 8.1Hz, ArH), 6.79 (1H, d, *J* = 7.0Hz, ArH), 7.13 (1H, dd, *J* = 7.8, 7.9Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 59.1, 69.3, 69.7, 70.6, 70.6, 70.7, 70.7, 70.8, 72.0, 111.7, 121.2, 127.0, 128.0, 143.5, 156.1; HRMS (ESI) *m/z* calcd for $C_{60}H_{88}O_{20}$ [M+Na]$^+$ 1151.5761, found 1151.5774.

*1.7. Dihydrobenzo[h]tetraphenyleno[1,16-bcd][1,6]dioxecine-13,14-diol and its (S,S) Counterpart (**8**)*

To a mixture of compound (±)–**1** (667 mg, 1.65 mmol) and Cs_2CO_3 (805 mg, 2.47 mmol), DMSO (33 mL) was added. The mixture was heated to 80˚C for 30 minutes with stirring. After heating, the mixture was cooled to 15°C. A solution of 1,2-bis(bromomethyl)benzene (480 mg, 1.82 mmol) in DMSO (33 mL) was added to the mixture dropwise by a dropping funnel. After the addition, the mixture was stirred for 30 minutes at room temperature. The mixture was then diluted with water (50 mL) and was extracted with CH_2Cl_2 (60 mL × 3). The combined organic layer was washed with saturated brine solution (200 mL × 2) successively. The organic layer was then dried over anhydrous Na_2SO_4 and concentrated. After column chromatography on silica gel (70 g, EtOAc/CH_2Cl_2, 1:90), pure (±)–**8** (295 mg, 38%) was collected as colorless sticky solids and (*S*,*S*)–**8** was prepared similarly (38% yield): R_f = 0.15 (EtOAc/CH_2Cl_2, 1:90); $[\alpha]_D^{20}$: cannot be determined because of its poor solubility in organic solvent; ^{1}H NMR (300 MHz, DMSO-d_6): δ 5.08 (2H, brs, OCH_2), 5.30 (2H, d, *J* = 11.4Hz, OCH_2), 6.45 (2H, d, *J* = 7.4Hz, ArH), 6.66 (2H, d, *J* = 7.9Hz, ArH), 6.73 (2H, d, *J* = 5.3Hz, ArH), 6.96 (2H, dd, *J* = 7.7, 7.8Hz, ArH), 7.07 (2H, d, *J* = 7.7Hz, ArH), 7.13 (2H, d, *J* = 6.4Hz, ArH), 7.33 (2H, dd, *J* = 3.4, 5.3Hz, ArH), 7.49 (2H, brs, ArH), 8.97 (2H, brs, OH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 113.7, 119.3, 121.7, 124.2, 127.4, 127.8, 128.6, 131.5, 136.3, 143.0, 143.6, 154.4; HRMS (EI) *m/z* calcd for $C_{32}H_{22}O_4$ [M]$^+$ 470.1513, found 470.1508.

*1.8. 8,9-Bis(heptyloxy)tetraphenylene-1,16-diol and its (S,S) Counterpart (**9**)*

To a mixed solution of compound (±)–**8** (217 mg, 0.33 mmol) in THF and EtOH (1:1 *v/v* ratio), palladium black catalyst (10 mol%) was added. The mixture was stirred under H_2 for 2 hours. The mixture was then filtered through celite. The filtrate was concentrated and purified by column chromatography on silica gel (20 g, EtOAc/*n*-hexanes, 1:4) to yield pure (±)–**9** (174 mg, 95%) as colorless solids and (*S,S*)–**9** was prepared similarly (95% yield): m.p. 176-178 °C; R_f = 0.25 (EtOAc/*n*-hexanes, 1:4); ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.87 (6H, t, *J* = 7.2, 2×CH_3), 1.16-1.26 (16H, m, 8×CH_2), 1.43-1.48 (4H, m, 2×CH_2), 3.69-3.74 (4H, m, 2×OCH_2), 4.93 (2H, brs, OH), 6.74 (2H, dd, *J* = 0.6, 8.1Hz, ArH), 6.81 (2H, dd, *J* = 0.9, 7.5Hz, ArH), 6.85 (2H, dd, *J* = 0.9, 8.1Hz, ArH), 6.88 (2H, dd, *J* = 1.2, 7.5Hz, ArH), 7.16 (2H, dd, *J* = 8.1, 8.2Hz, ArH), 7.20 (2H, dd, *J* = 7.7, 7.8Hz, ArH); ^{13}C NMR (75 MHz, $CDCl_3$): δ 14.3, 22.7, 25.9, 29.1, 29.4, 32.0, 68.9, 111.7, 114.5, 120.2, 120.4, 121.5, 126.9, 128.1, 129.8, 142.4, 145.3, 152.3, 156.6; HRMS (EI) *m/z* calcd for $C_{38}H_{44}O_4$ $[M]^+$ 564.3234, found 564.3251; (*S,S*)–**9:** $[\alpha]_D^{20}$ = +5.7 (*c* = 0.83, $CHCl_3$).

*1.9. 8,9-Bis(dodecyloxy)tetraphenylene-1,16-diol (**10**)*

Compound (±)–**10** was prepared from (±)–**8** (115 mg, 0.25 mmol) by using the same preparative method as that for compound (*S,S*)–**9**, except using compound 1-bromododecane (184 mg, 0.74 mmol).

Column chromatography on silica gel (10 g, EtOAc/*n*-hexanes, 1:6) gave pure (±)–**10** (146 mg, 83%) as pale yellow waxy solids: R_f = 0.32 (EtOAc/*n*-hexanes, 1:4); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.89-0.92 (6H, m, 2×CH_3), 1.16-1.34 (36H, m, 18×CH_2), 1.44-1.50 (4H, m, 2×CH_2), 3.68-3.77 (4H, m, 2×OCH_2), 4.93 (2H, brs, OH), 6.74 (2H, d, *J* = 8.2Hz, ArH), 6.82 (4H, d, *J* = 7.8Hz, ArH), 6.88 (2H, d, *J* = 7.4Hz, ArH), 7.14-7.19 (4H, m, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.3, 22.9, 26.0, 29.4, 29.4, 29.5, 29.7, 29.8, 29.8, 29.9, 32.1, 68.9, 111.7, 114.5, 120.2, 120.4, 121.5, 126.9, 128.1, 129.7, 142.4, 145.3, 152.3, 156.6; HRMS (MALDI-TOF) *m/z* calcd for $C_{48}H_{64}O_4$ $[M+Na]^+$ 727.4696, found 727.4655; Anal. Calcd for $C_{48}H_{64}O_4$: C, 81.77; H, 9.15, found C, 81.62; H, 9.20.

*1.10. 1,16-Bis(heptyloxy)-8,9-bis(2-(2-methoxyethoxy) ethoxy)tetraphenylene and its (S,S) counterpart(**11** and (S,S)-**11**)*

To a solution of compound (*S*,*S*)–**11** (102 mg, 0.18 mmol) in DMF (3 mL), Cs_2CO_3 (177 mg, 0.54 mmol) and compound $H_3C\tilde{O}(CH_2CH_2O)_2Ts$ (148 mg, 0.54 mmol) were added subsequently. The mixture was heated to 80˚C for 1 hour. The mixture was cooled and was diluted with water (20 mL). The biphasic mixture was then partitioned between water and EtOAc (10 mL × 3). The combined organic layer was washed with saturated brine solution (50 mL × 2) successively. After dried over anhydrous Na_2SO_4 and concentration, the residue was purified by column chromatography on silica gel (10 g, MeOH/$CHCl_3$, 1:100) to yield pure (*S*,*S*)–**11** (103 mg, 75%) as a pale yellow oil and (±)–**11** was prepared similarly (2-step yield: 60%): $R_f = 0.25$ (MeOH/$CHCl_3$, 1:100); ^{1}H NMR (400 MHz, CD_2Cl_2): δ 0.92 (6H, t, *J* = 7.1Hz, 2×CH_3), 1.19-1.33 (16H, m, 8×CH_2), 1.50-1.56 (4H, m, 2×CH_2), 3.32 (6H, s, 2×OCH_3), 3.40 (8H, brs, 4×OCH_2), 3.52-3.65 (4H, m, 2×OCH_2), 3.80 (4H, t, *J* = 6.5Hz, 2×OCH_2), 3.96-3.98 (4H, m, 2×OCH_2), 6.78-6.88 (8H, m, ArH), 7.19-7.24 (4H, m, ArH); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 13.6, 22.3, 25.4, 28.7, 29.0, 31.5, 58.2, 68.4, 68.7, 69.3, 70.2, 71.6, 110.5, 110.9, 120.1, 120.7, 126.3, 126.3, 127.5, 127.6, 142.9, 143.3, 155.7, 156.0; HRMS (ESI) *m/z* calcd for $C_{48}H_{64}O_8$ $[M]^+$ 791.4493, found 791.4511; (*S*,*S*)–**11:** $[\alpha]_D^{20}$ = –23.2 (*c* = 1.0, $CHCl_3$).

*1.11. 13,13'-(8,9-Bis(dodecyloxy)tetraphenylene-1,16-diyl)bis(oxy)bis(2,5,8,11-tetraoxatridecane) and its (S,S) Counterpart (**12** and (S,S)–**12**)*

Compound (*S*,*S*)–**12** was prepared from (*S*,*S*)–**10** (102 mg, 0.14 mmol) by using the same preparative method as that for compound (*S*,*S*)–**11**, except using $H_3C\tilde{O}(CH_2CH_2O)_4Ts$ (157 mg, 0.43 mmol).

Column chromatography on silica gel (10 g, MeOH/$CHCl_3$, 1:100) gave pure (*S*,*S*)–**12** (121 mg, 80%) as a pale yellow oil and (±)–**12** was prepared similarly (2-steps yield: 61%): $R_f = 0.29$ (MeOH/$CHCl_3$, 1:100); ^{1}H NMR (400 MHz, $CDCl_3$): δ 0.87-0.90 (6H, m, 2×CH_3), 1.15-1.26 (36H, m, 18×CH_2), 1.44 (4H, m, 2×CH_2), 3.37 (10H, brs, 2×OCH_2 and 2×OCH_3), 3.46-3.72 (28H, m, 14×OCH_2), 3.88-3.90 (4H, m, 2×OCH_2), 6.70-6.77 (6H, m, ArH), 6.83 (2H, d, *J* = 7.5Hz, ArH) 7.12 (4H, dd, *J* = 7.8, 7.9Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.2, 22.8, 25.9, 29.4, 29.5, 29.7, 29.7, 29.8, 29.8, 32.0, 59.1, 69.0, 69.4, 69.7, 70.6, 70.6, 70.7, 70.7, 70.8, 72.0, 111.5, 111.6, 120.6, 121.3, 127.1, 127.1, 127.7, 127.8, 143.2, 143.8, 156.0, 156.5; HRMS (ESI) *m/z* calcd for $C_{66}H_{100}O_{12}$ $[M+Na]^+$ 1107.7107, found 1107.7100; (*S*,*S*)–**12:** $[\alpha]_D^{20} = -22.2$ ($c = 0.94$, $CHCl_3$).

Reference

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3. H.-Y. Peng, C.-K. Lam, T. C. W. Mak, Z. Cai, W.-T. Ma, Y.-X. Li, H. N. C. Wong *J. Am. Chem. Soc.* **2005**, *127,* 9603-9611.

Table 1. Crystal data and structure refinement for (±)-11.

Identification code	(±)-11
Empirical formula	C48 H64 O8
Formula weight	768.99
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 12.6286(9) A alpha = 85.812(2) deg. b = 13.2786(10) A beta = 77.077(2) deg. c = 14.6898(10) A gamma = 67.470(2) deg.
Volume	2217.5(3) A^3
Z, Calculated density	2, 1.152 Mg/m^3
Absorption coefficient	0.077 mm^-1
F(000)	832
Crystal size	0.20 x 0.25 x 0.20 mm
Theta range for data collection	1.42 to 26.00 deg.
Limiting indices	-15<=h<=15, -16<=k<=16, -18<=l<=17
Reflections collected / unique	37088 / 8687 [R(int) = 0.0972]
Completeness to theta = 26.00	99.6 %
Absorption correction	None

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8687 / 0 / 505
Goodness-of-fit on F^2	1.002
Final R indices [I>2sigma(I)]	R1 = 0.0670, wR2 = 0.1589
R indices (all data)	R1 = 0.1681, wR2 = 0.2093
Largest diff. peak and hole	0.298 and -0.236 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for p.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	2091(2)	5632(2)	163(1)	58(1)
O(2)	1725(2)	6804(2)	1904(2)	64(1)
O(3)	-116(2)	2918(2)	4393(2)	67(1)
O(4)	-1097(2)	2727(2)	2756(2)	66(1)
O(5)	3869(2)	6548(2)	-194(2)	72(1)
O(6)	4835(3)	8079(3)	277(3)	116(1)
O(7)	344(3)	9109(2)	1931(3)	113(1)
O(8)	-1799(5)	10255(4)	1243(5)	259(4)
C(1)	944(3)	5810(2)	567(2)	49(1)
C(2)	24(3)	6294(3)	107(2)	59(1)
C(3)	-1098(3)	6437(3)	571(3)	64(1)
C(4)	-1325(3)	6109(3)	1476(2)	58(1)
C(5)	-412(3)	5611(2)	1946(2)	47(1)
C(6)	-697(2)	5316(2)	2948(2)	45(1)
C(7)	-1394(3)	6132(3)	3607(2)	58(1)
C(8)	-1662(3)	5877(3)	4529(2)	64(1)
C(9)	-1262(3)	4825(3)	4820(2)	60(1)
C(10)	-574(3)	3998(3)	4176(2)	53(1)
C(11)	-276(2)	4228(2)	3222(2)	46(1)
C(12)	475(3)	3316(2)	2543(2)	49(1)
C(13)	46(3)	2540(2)	2360(2)	55(1)
C(14)	770(4)	1637(3)	1792(2)	68(1)
C(15)	1906(4)	1513(3)	1406(2)	72(1)
C(16)	2350(3)	2272(3)	1568(2)	63(1)
C(17)	1634(3)	3178(2)	2132(2)	50(1)
C(18)	2132(3)	3980(2)	2321(2)	48(1)
C(19)	3044(3)	3638(3)	2796(2)	58(1)

C(20)	3531(3)	4357(3)	2958(2)	67(1)
C(21)	3119(3)	5421(3)	2665(2)	59(1)
C(22)	2215(3)	5762(2)	2204(2)	49(1)
C(23)	1707(2)	5049(2)	2013(2)	43(1)
C(24)	733(3)	5470(2)	1496(2)	44(1)
C(25)	2291(3)	6266(3)	-639(2)	66(1)
C(26)	3561(3)	6095(3)	-891(2)	70(1)
C(27)	5061(3)	6459(3)	-450(3)	82(1)
C(28)	5360(3)	6937(4)	287(3)	86(1)
C(29)	5117(5)	8587(4)	953(5)	153(2)
C(30)	2159(3)	7600(3)	2048(3)	69(1)
C(31)	1533(4)	8610(3)	1580(4)	98(1)
C(32)	-362(6)	8689(4)	1567(6)	178(3)
C(33)	-1372(8)	9267(6)	1466(9)	295(7)
C(34)	-2803(6)	10694(5)	1022(5)	166(3)
C(35)	-375(3)	2599(3)	5335(2)	73(1)
C(36)	178(5)	1383(4)	5377(3)	106(2)
C(37)	1498(7)	971(5)	5191(5)	160(3)
C(38)	2121(8)	793(6)	4297(6)	194(4)
C(39)	3471(9)	379(8)	4147(7)	239(5)
C(40)	4134(11)	103(10)	3416(8)	289(6)
C(41)	5487(8)	-378(9)	3289(7)	264(5)
C(42)	-1482(4)	1839(3)	2780(3)	85(1)
C(43)	-2714(4)	2201(4)	3313(3)	89(1)
C(44)	-2898(3)	2510(3)	4309(3)	82(1)
C(45)	-4157(4)	2898(4)	4833(3)	98(1)
C(46)	-4363(4)	3280(5)	5797(4)	141(2)
C(47)	-5626(6)	3765(8)	6287(6)	229(5)
C(48)	-6073(5)	4529(5)	6850(7)	205(4)

Table 3. Bond lengths [A] and angles [deg] for p.

O(1)-C(1)	1.371(3)
O(1)-C(25)	1.430(4)
O(2)-C(22)	1.366(3)
O(2)-C(30)	1.413(3)
O(3)-C(10)	1.368(4)
O(3)-C(35)	1.422(4)
O(4)-C(13)	1.361(4)
O(4)-C(42)	1.434(4)
O(5)-C(26)	1.417(4)
O(5)-C(27)	1.427(4)
O(6)-C(28)	1.404(5)
O(6)-C(29)	1.417(6)
O(7)-C(31)	1.379(5)
O(7)-C(32)	1.425(5)
O(8)-C(33)	1.263(8)
O(8)-C(34)	1.282(6)
C(1)-C(2)	1.391(4)
C(1)-C(24)	1.406(4)
C(2)-C(3)	1.374(5)
C(2)-H(2A)	0.9300
C(3)-C(4)	1.371(4)
C(3)-H(3A)	0.9300
C(4)-C(5)	1.396(4)
C(4)-H(4A)	0.9300
C(5)-C(24)	1.394(4)
C(5)-C(6)	1.496(4)
C(6)-C(7)	1.389(4)
C(6)-C(11)	1.398(4)
C(7)-C(8)	1.371(4)
C(7)-H(7A)	0.9300
C(8)-C(9)	1.363(5)
C(8)-H(8A)	0.9300
C(9)-C(10)	1.381(4)
C(9)-H(9A)	0.9300

C(10)-C(11)	1.411(4)
C(11)-C(12)	1.495(4)
C(12)-C(17)	1.398(4)
C(12)-C(13)	1.401(4)
C(13)-C(14)	1.391(4)
C(14)-C(15)	1.369(5)
C(14)-H(14A)	0.9300
C(15)-C(16)	1.385(5)
C(15)-H(15A)	0.9300
C(16)-C(17)	1.388(4)
C(16)-H(16A)	0.9300
C(17)-C(18)	1.497(4)
C(18)-C(19)	1.390(4)
C(18)-C(23)	1.393(4)
C(19)-C(20)	1.376(4)
C(19)-H(19A)	0.9300
C(20)-C(21)	1.379(5)
C(20)-H(20A)	0.9300
C(21)-C(22)	1.369(4)
C(21)-H(21A)	0.9300
C(22)-C(23)	1.404(4)
C(23)-C(24)	1.493(4)
C(25)-C(26)	1.493(5)
C(25)-H(25A)	0.9700
C(25)-H(25B)	0.9700
C(26)-H(26A)	0.9700
C(26)-H(26B)	0.9700
C(27)-C(28)	1.478(5)
C(27)-H(27A)	0.9700
C(27)-H(27B)	0.9700
C(28)-H(28A)	0.9700
C(28)-H(28B)	0.9700
C(29)-H(29A)	0.9600
C(29)-H(29B)	0.9600
C(29)-H(29C)	0.9600
C(30)-C(31)	1.481(5)
C(30)-H(30A)	0.9700
C(30)-H(30B)	0.9700

C(31)-H(31A)	0.9700
C(31)-H(31B)	0.9700
C(32)-C(33)	1.247(7)
C(32)-H(32A)	0.9700
C(32)-H(32B)	0.9700
C(33)-H(33A)	0.9700
C(33)-H(33B)	0.9700
C(34)-H(34A)	0.9600
C(34)-H(34B)	0.9600
C(34)-H(34C)	0.9600
C(35)-C(36)	1.497(5)
C(35)-H(35A)	0.9700
C(35)-H(35B)	0.9700
C(36)-C(37)	1.507(7)
C(36)-H(36A)	0.9700
C(36)-H(36B)	0.9700
C(37)-C(38)	1.356(8)
C(37)-H(37A)	0.9700
C(37)-H(37B)	0.9700
C(38)-C(39)	1.547(12)
C(38)-H(38A)	0.9700
C(38)-H(38B)	0.9700
C(39)-C(40)	1.187(11)
C(39)-H(39A)	0.9700
C(39)-H(39B)	0.9700
C(40)-C(41)	1.549(12)
C(40)-H(40A)	0.9700
C(40)-H(40B)	0.9700
C(41)-H(41A)	0.9600
C(41)-H(41B)	0.9600
C(41)-H(41C)	0.9600
C(42)-C(43)	1.484(5)
C(42)-H(42A)	0.9700
C(42)-H(42B)	0.9700
C(43)-C(44)	1.491(5)
C(43)-H(43A)	0.9700
C(43)-H(43B)	0.9700
C(44)-C(45)	1.507(5)

C(44)-H(44A)	0.9700
C(44)-H(44B)	0.9700
C(45)-C(46)	1.473(6)
C(45)-H(45A)	0.9700
C(45)-H(45B)	0.9700
C(46)-C(47)	1.497(7)
C(46)-H(46A)	0.9700
C(46)-H(46B)	0.9700
C(47)-C(48)	1.228(7)
C(47)-H(47A)	0.9700
C(47)-H(47B)	0.9700
C(48)-H(48A)	0.9600
C(48)-H(48B)	0.9600
C(48)-H(48C)	0.9600
C(1)-O(1)-C(25)	116.7(2)
C(22)-O(2)-C(30)	120.3(2)
C(10)-O(3)-C(35)	119.1(3)
C(13)-O(4)-C(42)	117.8(3)
C(26)-O(5)-C(27)	111.6(3)
C(28)-O(6)-C(29)	112.7(4)
C(31)-O(7)-C(32)	114.7(4)
C(33)-O(8)-C(34)	123.1(6)
O(1)-C(1)-C(2)	123.5(3)
O(1)-C(1)-C(24)	115.9(3)
C(2)-C(1)-C(24)	120.6(3)
C(3)-C(2)-C(1)	119.2(3)
C(3)-C(2)-H(2A)	120.4
C(1)-C(2)-H(2A)	120.4
C(4)-C(3)-C(2)	121.3(3)
C(4)-C(3)-H(3A)	119.4
C(2)-C(3)-H(3A)	119.4
C(3)-C(4)-C(5)	120.3(3)
C(3)-C(4)-H(4A)	119.9
C(5)-C(4)-H(4A)	119.9
C(24)-C(5)-C(4)	119.7(3)
C(24)-C(5)-C(6)	121.3(3)
C(4)-C(5)-C(6)	118.9(3)

C(7)-C(6)-C(11)	120.0(3)
C(7)-C(6)-C(5)	119.4(3)
C(11)-C(6)-C(5)	120.6(3)
C(8)-C(7)-C(6)	120.2(3)
C(8)-C(7)-H(7A)	119.9
C(6)-C(7)-H(7A)	119.9
C(9)-C(8)-C(7)	121.2(3)
C(9)-C(8)-H(8A)	119.4
C(7)-C(8)-H(8A)	119.4
C(8)-C(9)-C(10)	119.6(3)
C(8)-C(9)-H(9A)	120.2
C(10)-C(9)-H(9A)	120.2
O(3)-C(10)-C(9)	124.4(3)
O(3)-C(10)-C(11)	114.7(3)
C(9)-C(10)-C(11)	120.9(3)
C(6)-C(11)-C(10)	118.0(3)
C(6)-C(11)-C(12)	122.3(3)
C(10)-C(11)-C(12)	119.6(3)
C(17)-C(12)-C(13)	119.0(3)
C(17)-C(12)-C(11)	121.1(3)
C(13)-C(12)-C(11)	119.8(3)
O(4)-C(13)-C(14)	123.0(3)
O(4)-C(13)-C(12)	116.7(3)
C(14)-C(13)-C(12)	120.3(3)
C(15)-C(14)-C(13)	119.7(3)
C(15)-C(14)-H(14A)	120.2
C(13)-C(14)-H(14A)	120.2
C(14)-C(15)-C(16)	121.2(3)
C(14)-C(15)-H(15A)	119.4
C(16)-C(15)-H(15A)	119.4
C(15)-C(16)-C(17)	119.6(3)
C(15)-C(16)-H(16A)	120.2
C(17)-C(16)-H(16A)	120.2
C(16)-C(17)-C(12)	120.2(3)
C(16)-C(17)-C(18)	119.1(3)
C(12)-C(17)-C(18)	120.6(3)
C(19)-C(18)-C(23)	120.1(3)
C(19)-C(18)-C(17)	119.0(3)

C(23)-C(18)-C(17)	120.9(3)
C(20)-C(19)-C(18)	119.9(3)
C(20)-C(19)-H(19A)	120.0
C(18)-C(19)-H(19A)	120.0
C(19)-C(20)-C(21)	121.0(3)
C(19)-C(20)-H(20A)	119.5
C(21)-C(20)-H(20A)	119.5
C(22)-C(21)-C(20)	119.2(3)
C(22)-C(21)-H(21A)	120.4
C(20)-C(21)-H(21A)	120.4
O(2)-C(22)-C(21)	123.9(3)
O(2)-C(22)-C(23)	114.6(3)
C(21)-C(22)-C(23)	121.5(3)
C(18)-C(23)-C(22)	118.3(3)
C(18)-C(23)-C(24)	122.9(2)
C(22)-C(23)-C(24)	118.8(3)
C(5)-C(24)-C(1)	118.9(3)
C(5)-C(24)-C(23)	120.7(3)
C(1)-C(24)-C(23)	120.2(3)
O(1)-C(25)-C(26)	109.1(3)
O(1)-C(25)-H(25A)	109.9
C(26)-C(25)-H(25A)	109.9
O(1)-C(25)-H(25B)	109.9
C(26)-C(25)-H(25B)	109.9
H(25A)-C(25)-H(25B)	108.3
O(5)-C(26)-C(25)	110.7(3)
O(5)-C(26)-H(26A)	109.5
C(25)-C(26)-H(26A)	109.5
O(5)-C(26)-H(26B)	109.5
C(25)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	108.1
O(5)-C(27)-C(28)	110.6(3)
O(5)-C(27)-H(27A)	109.5
C(28)-C(27)-H(27A)	109.5
O(5)-C(27)-H(27B)	109.5
C(28)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	108.1
O(6)-C(28)-C(27)	110.1(4)

O(6)-C(28)-H(28A)	109.6
C(27)-C(28)-H(28A)	109.6
O(6)-C(28)-H(28B)	109.6
C(27)-C(28)-H(28B)	109.6
H(28A)-C(28)-H(28B)	108.2
O(6)-C(29)-H(29A)	109.5
O(6)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5
O(6)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
O(2)-C(30)-C(31)	108.0(3)
O(2)-C(30)-H(30A)	110.1
C(31)-C(30)-H(30A)	110.1
O(2)-C(30)-H(30B)	110.1
C(31)-C(30)-H(30B)	110.1
H(30A)-C(30)-H(30B)	108.4
O(7)-C(31)-C(30)	116.3(4)
O(7)-C(31)-H(31A)	108.2
C(30)-C(31)-H(31A)	108.2
O(7)-C(31)-H(31B)	108.2
C(30)-C(31)-H(31B)	108.2
H(31A)-C(31)-H(31B)	107.4
C(33)-C(32)-O(7)	122.6(5)
C(33)-C(32)-H(32A)	106.7
O(7)-C(32)-H(32A)	106.7
C(33)-C(32)-H(32B)	106.7
O(7)-C(32)-H(32B)	106.7
H(32A)-C(32)-H(32B)	106.6
C(32)-C(33)-O(8)	130.4(8)
C(32)-C(33)-H(33A)	104.7
O(8)-C(33)-H(33A)	104.7
C(32)-C(33)-H(33B)	104.7
O(8)-C(33)-H(33B)	104.7
H(33A)-C(33)-H(33B)	105.7
O(8)-C(34)-H(34A)	109.5
O(8)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5

O(8)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
O(3)-C(35)-C(36)	108.5(3)
O(3)-C(35)-H(35A)	110.0
C(36)-C(35)-H(35A)	110.0
O(3)-C(35)-H(35B)	110.0
C(36)-C(35)-H(35B)	110.0
H(35A)-C(35)-H(35B)	108.4
C(35)-C(36)-C(37)	112.5(4)
C(35)-C(36)-H(36A)	109.1
C(37)-C(36)-H(36A)	109.1
C(35)-C(36)-H(36B)	109.1
C(37)-C(36)-H(36B)	109.1
H(36A)-C(36)-H(36B)	107.8
C(38)-C(37)-C(36)	119.4(7)
C(38)-C(37)-H(37A)	107.5
C(36)-C(37)-H(37A)	107.5
C(38)-C(37)-H(37B)	107.5
C(36)-C(37)-H(37B)	107.5
H(37A)-C(37)-H(37B)	107.0
C(37)-C(38)-C(39)	117.1(8)
C(37)-C(38)-H(38A)	108.0
C(39)-C(38)-H(38A)	108.0
C(37)-C(38)-H(38B)	108.0
C(39)-C(38)-H(38B)	108.0
H(38A)-C(38)-H(38B)	107.3
C(40)-C(39)-C(38)	124.8(13)
C(40)-C(39)-H(39A)	106.1
C(38)-C(39)-H(39A)	106.1
C(40)-C(39)-H(39B)	106.1
C(38)-C(39)-H(39B)	106.1
H(39A)-C(39)-H(39B)	106.3
C(39)-C(40)-C(41)	124.1(14)
C(39)-C(40)-H(40A)	106.3
C(41)-C(40)-H(40A)	106.3
C(39)-C(40)-H(40B)	106.3
C(41)-C(40)-H(40B)	106.3

H(40A)-C(40)-H(40B)	106.4
C(40)-C(41)-H(41A)	109.5
C(40)-C(41)-H(41B)	109.5
H(41A)-C(41)-H(41B)	109.5
C(40)-C(41)-H(41C)	109.5
H(41A)-C(41)-H(41C)	109.5
H(41B)-C(41)-H(41C)	109.5
O(4)-C(42)-C(43)	108.3(3)
O(4)-C(42)-H(42A)	110.0
C(43)-C(42)-H(42A)	110.0
O(4)-C(42)-H(42B)	110.0
C(43)-C(42)-H(42B)	110.0
H(42A)-C(42)-H(42B)	108.4
C(42)-C(43)-C(44)	115.2(3)
C(42)-C(43)-H(43A)	108.5
C(44)-C(43)-H(43A)	108.5
C(42)-C(43)-H(43B)	108.5
C(44)-C(43)-H(43B)	108.5
H(43A)-C(43)-H(43B)	107.5
C(43)-C(44)-C(45)	114.5(3)
C(43)-C(44)-H(44A)	108.6
C(45)-C(44)-H(44A)	108.6
C(43)-C(44)-H(44B)	108.6
C(45)-C(44)-H(44B)	108.6
H(44A)-C(44)-H(44B)	107.6
C(46)-C(45)-C(44)	115.0(4)
C(46)-C(45)-H(45A)	108.5
C(44)-C(45)-H(45A)	108.5
C(46)-C(45)-H(45B)	108.5
C(44)-C(45)-H(45B)	108.5
H(45A)-C(45)-H(45B)	107.5
C(45)-C(46)-C(47)	115.0(5)
C(45)-C(46)-H(46A)	108.5
C(47)-C(46)-H(46A)	108.5
C(45)-C(46)-H(46B)	108.5
C(47)-C(46)-H(46B)	108.5
H(46A)-C(46)-H(46B)	107.5
C(48)-C(47)-C(46)	126.6(6)

C(48)-C(47)-H(47A)	105.7
C(46)-C(47)-H(47A)	105.7
C(48)-C(47)-H(47B)	105.7
C(46)-C(47)-H(47B)	105.7
H(47A)-C(47)-H(47B)	106.1
C(47)-C(48)-H(48A)	109.5
C(47)-C(48)-H(48B)	109.5
H(48A)-C(48)-H(48B)	109.5
C(47)-C(48)-H(48C)	109.5
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for p.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	63(2)	60(1)	50(1)	13(1)	-12(1)	-24(1)
O(2)	77(2)	49(1)	84(2)	7(1)	-34(1)	-34(1)
O(3)	88(2)	56(2)	50(1)	10(1)	-9(1)	-26(1)
O(4)	77(2)	55(1)	77(2)	-4(1)	-13(1)	-37(1)
O(5)	62(2)	84(2)	63(2)	-1(1)	2(1)	-31(1)
O(6)	106(2)	94(2)	155(3)	2(2)	-46(2)	-36(2)
O(7)	98(2)	62(2)	186(3)	-16(2)	-51(2)	-23(2)
O(8)	231(5)	92(3)	498(10)	-43(4)	-259(7)	0(3)
C(1)	61(2)	41(2)	51(2)	1(2)	-15(2)	-22(2)
C(2)	76(3)	57(2)	52(2)	10(2)	-27(2)	-28(2)
C(3)	70(2)	62(2)	73(3)	12(2)	-36(2)	-29(2)
C(4)	61(2)	52(2)	66(2)	6(2)	-23(2)	-25(2)
C(5)	56(2)	37(2)	54(2)	1(1)	-21(2)	-19(1)
C(6)	48(2)	44(2)	47(2)	-4(2)	-10(2)	-22(2)
C(7)	58(2)	49(2)	68(2)	-3(2)	-16(2)	-19(2)
C(8)	68(2)	60(2)	59(2)	-18(2)	-3(2)	-20(2)
C(9)	66(2)	67(2)	47(2)	-6(2)	-4(2)	-29(2)
C(10)	58(2)	56(2)	50(2)	2(2)	-11(2)	-27(2)
C(11)	53(2)	45(2)	45(2)	-2(2)	-8(2)	-24(2)
C(12)	67(2)	39(2)	41(2)	2(1)	-12(2)	-20(2)
C(13)	73(2)	44(2)	51(2)	2(2)	-13(2)	-25(2)
C(14)	96(3)	48(2)	65(2)	-5(2)	-13(2)	-33(2)
C(15)	99(3)	41(2)	63(2)	-7(2)	-2(2)	-20(2)
C(16)	73(2)	45(2)	57(2)	4(2)	-1(2)	-16(2)
C(17)	63(2)	38(2)	44(2)	6(1)	-8(2)	-16(2)
C(18)	49(2)	50(2)	41(2)	0(2)	-4(2)	-17(2)
C(19)	56(2)	56(2)	52(2)	8(2)	-12(2)	-12(2)
C(20)	55(2)	86(3)	58(2)	3(2)	-21(2)	-20(2)

C(21)	59(2)	67(2)	59(2)	1(2)	-20(2)	-28(2)
C(22)	54(2)	48(2)	47(2)	2(2)	-13(2)	-20(2)
C(23)	47(2)	44(2)	38(2)	3(1)	-9(1)	-19(1)
C(24)	57(2)	35(2)	46(2)	2(1)	-17(2)	-20(1)
C(25)	83(3)	72(2)	45(2)	12(2)	-12(2)	-32(2)
C(26)	83(3)	77(3)	45(2)	7(2)	-2(2)	-32(2)
C(27)	63(3)	95(3)	75(3)	6(2)	1(2)	-26(2)
C(28)	67(3)	89(3)	99(3)	5(3)	-14(2)	-29(2)
C(29)	147(5)	117(4)	207(7)	-39(5)	-60(5)	-42(4)
C(30)	72(2)	58(2)	85(3)	-11(2)	-11(2)	-35(2)
C(31)	104(4)	59(3)	139(4)	6(3)	-22(3)	-42(3)
C(32)	135(5)	74(4)	353(10)	-30(5)	-124(6)	-27(4)
C(33)	212(8)	73(5)	650(20)	-30(8)	-270(12)	-8(5)
C(34)	199(7)	149(6)	186(6)	42(5)	-101(6)	-81(5)
C(35)	96(3)	80(3)	53(2)	18(2)	-19(2)	-45(2)
C(36)	133(4)	84(3)	99(4)	32(3)	-24(3)	-45(3)
C(37)	184(7)	103(4)	144(6)	31(4)	-26(5)	-11(4)
C(38)	198(8)	131(6)	150(7)	20(5)	29(6)	12(5)
C(39)	205(11)	176(8)	200(10)	-1(7)	32(8)	35(7)
C(40)	215(12)	275(13)	260(14)	21(11)	24(10)	-10(10)
C(41)	161(8)	376(15)	231(10)	-37(10)	1(7)	-91(9)
C(42)	110(3)	82(3)	85(3)	-13(2)	-5(2)	-67(3)
C(43)	104(3)	94(3)	95(3)	-9(2)	-23(3)	-64(3)
C(44)	78(3)	95(3)	81(3)	1(2)	-21(2)	-39(2)
C(45)	84(3)	121(4)	106(4)	-3(3)	-29(3)	-50(3)
C(46)	99(4)	229(7)	117(4)	-60(4)	4(3)	-90(4)
C(47)	125(5)	306(11)	274(9)	-184(9)	89(6)	-142(7)
C(48)	101(5)	137(6)	365(13)	-69(7)	15(6)	-56(4)