Supplementary Information

Anisotropic electrical conductivity of n-doped thin-films of polymerizable liquid-crystalline perylene bisimide bearing a triethylene oxide chain and cyclotetrasiloxane rings

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Synthesis of Materials

All ¹H and ¹³CNMR spectra were recorded on a Varian UNITY INOVA400NB spectrometer. FT-IR measurements were conducted on a JASCO FT/IR-660 Plus spectrometer. MALDI-TOF Mass spectra were measured by Bruker Ultraflex III without matrix. Perylene tetracarboxylic acid anhydride and the Karstedt catalyst were purchased from Tokyo Chemical Industry and Gelest Inc., respectively. Zinc acetate, quinoline, and toluene (commercially available from Wako Pure Chemical Industries) were used without purification. Silica gel was purchased from Kanto Chemicals.

N-(1,9-di(1,3,3,5,5,7,7-heptamethyl-1,3,5,7-cyclotetrasiloxan-1-yl)nonan-5-yl)-*N*'-octylperylene-3,4,9,10-tetracarboxylic bisimide (1)

N-N'-bis(1,11-undecan-6-yl)perylene-3,4,9,10-tetracarboxlic acid bisimide (**5**) 0.54 g (0.87 mmol) and 1,3,3,5,5,7,7-heptamethyl-1,3,5,7-cyclotetrasiloxane 0.66 g (2.3 mmol) were dissolved in toluene (30 ml) and 5 μ l of Karstedt catalyst (1,3-divinyl-1,1,3,3-tetramethyldisiloxane platinum (0), 2.1 atom%, xylene solution) was added to the reaction mixture. The solution was refluxed for two hours, and cooled to room temperature. The solvent was evaporated and the obtained red residue was purified by a column chromatography (elutant: dichloromethane). The crude product was dissolved in dichloromethane and the solution was poured into methanol. The red precipitates were filtered. Red powder (0.72 g, 0.61 mmol) was obtained in the yield of 70 %.

¹H NMR (400 MHz, CDCl₃): $\delta = 8.63$ (br, 2H), 8.57 (d, 2H, J = 8.4 Hz), 8.52 (d, 2H, J = 8.4 Hz), 8.49 (d, 2H, J = 8.4 Hz), 5.17 (tt, 2H, J = 9.2, 4.5 Hz), 4.16 (t, 2H, J = 7.2 Hz), 2.10–2.30 (m, 2H), 1.78–2.00 (m, 2H), 1.73 (quint, 2H, J = 6.8 Hz), 1.20–1.46 (m, 24H), 0.86 (t, 3H, J = 6.4 Hz), 0.48 (dd, 4H, J = 7.2, 8.4 Hz), 0.04 (s, 6H), 0.035 (s, 6H), 0.03 (s, 6H), 0.025 (s, 18H), 0.003 (s, 6H) ppm; ¹³C NMR (100 MHz CDCl₃): $\delta = 0.00, 0.93, 0.94, 0.97, 14.3, 17.3, 22.8, 23.2, 27.4, 28.3, 29.4, 29.5,$ 30.8, 32.0, 32.4, 123.0, 123.1, 123.3, 124.2, 126.4, 129.3, 129.6, 131.3, 131.9, 134.3, 134.6, 163.3, $163.4 ppm; IR (ATR): <math>\nu = 2960, 2925, 2859, 1698, 1654, 1595, 1578, 1439, 1405, 1341, 1257, 1049,$ 802, 744, 696, 627, 555, 485 cm⁻¹; exact mass: 1188.42; molecular weight: 1189.95 m/z [M]: 1187.9,1188.9, 1189.9, 1190.9, 1191.9, 1191.9, 1192.9; elemental analysis (%) calculated forC₅₅H₈₄N₂O₁₂Si₈: C, 55.51, H, 7.12, N, 2.35, O, 16.13, Si, 18.88; found: C, 55.5; H, 7.18; N, 2.36.



Mass spectrum of compound 1

N-(1,9-di(1,3,3,5,5,7,7-heptamethyl-1,3,5,7-cyclotetrasiloxan-1-yl)nonan-5-yl)-*N*'-(3,6,9-trioxod ecan-1-yl)perylene-3,4,9,10-tetracarboxylic bisimide (2)

N-N'-bis(1,12-tridecadien-7-yl)perylene-3,4,9,10-tetracarboxlic acid bisimide (**6**) 0.94 g (1.4 mmol) and 1,3,3,5,5,7,7-heptamethyl-1,3,5,7-cyclotetrasiloxane 1.15 g (4.1 mmol) were dissolved in toluene (50 ml) and 10 μ l of Karstedt catalyst (1,3-divinyl-1,1,3,3-tetramethyldisiloxane platinum (0), 2.1 atom%, xylene solution) was added to the reaction mixture. The solution was refluxed for two hours, and cooled to room temperature. The solvent was evaporated and the obtained red residue was purified by a column chromatography (elutant: dichloromethane/methanol (100:1)). The crude product was dissolved in dichloromethane and the solution was poured into methanol. The red precipitates were filtered. Red powder (1.57 g, 1.3 mmol) was obtained in the yield of 89 %.

¹H NMR (400 MHz, CDCl₃): $\delta = 8.62$ (d, 1H, J = 8.0 Hz), 8.59 (d, 1H, J = 8.0 Hz), 8.54 (d, 2H, J = 8.0 Hz), 8.50 (d, 2H, J = 8.0 Hz), 8.46 (d, 2H, J = 8.0 Hz), 5.17 (tt, 1H, J = 3.2, 9.2 Hz), 4.43 (t, 2H, J = 6.0 Hz), 3.85 (t, 2H, J = 6.0 Hz), 3.72 (dd, 2H, J = 5.6, 6.4 Hz), 3.63 (dd, 2H, J = 5.6, 6.4 Hz), 3.58 (dd, 2H, J = 4.8, 6.0 Hz), 3.45 (dd, 2H, J = 4.8, 6.0 Hz), 3.29 (s, 3H), 2.18-2.30 (m, 2H), 1.80-1.94 (m, 2H), 1.24-1.44 (m, 8H), 0.48 (dd, 4H, J = 3.6, 4.8 Hz), 0.04 (s, 6H), 0.04 (s, 6H), 0.03 (s, 6H), 0.02 (s, 18H), 0.00 (s, 6H) ppm; ¹³C NMR (100 MHz CDCl₃): $\delta = 0.58$, 0.94, 0.95, 0.97, 17.3, 23.1, 30.7, 32.4, 39.5, 42.5, 55.0, 59.2, 68.1, 70.3, 70.7, 70.9, 72.1, 123.0, 123.2, 129.5, 129.6, 131.2, 131.5, 131.9, 132.0, 134.4, 134.8, 136.1, 155.6, 161.3, 161.4, 163.5 ppm; IR (ATR): $\nu = 2961.8$, 2924.8, 2862.9, 1695.5, 1654.5, 1594.6, 1577.8, 1437.8, 1404.7, 1341.5, 1257.4, 1050.2, 800.1, 744.4, 695.6, 554.7, 485.8, 432.7 cm⁻¹; exact mass: 1222.39; molecular weight: 1223.92 m/z [M]: 1224.0, 1225.0, 1226.0, 1227.0, 1228.0, 1229.0; elemental analysis (%) calculated for C₅₄H₈₂N₂O₁₅Si₈: C, 52.99, H, 6.75, N, 2.29, O, 19.61, Si, 18.36; found: C, 53.0; H, 6.69; N, 2.33.



Mass spectrum of compound 2

N-(1,8-nonadiene-5-yl)-N'-octylperylene-3,4:9,10-tetracarboxylic bisimide (5)

 N,N^{2} -di(1,8-nonadiene-5-yl)-perylene-3,4:9,10-tetracarboxylic bisimide (**3**) (1.10 g, 1.7 mmol) and crashed sodium hydroxide (1.5 g) were suspended in *tert*-butyl alcohol (30 mL) and heated to 100 °C for 2 h. Acetic acid (30 mL) was added to the solution and the mixture was stirred for 2 h. To the

resulting solution, 2 N hydrochloric acid (10 mL) was added. The dark red precipitates were filtered and washed with methanol. The dark red powder of compound **4** (0.87 g, 1.7 mmol) was obtained. The crude product was used for the next condensation reaction without further purification.

The dark red powder (0.81 g, 1.6 mmol), *n*-octylamine (0.51 g, 4.0 mmol), and zinc(II) acetate (1.02 g, 5.6 mmol) were suspended in quinoline (30 mL) and heated to 150 °C for 2 h. The reaction mixture was cooled, and hydrochloric acid (30%) was added to it. The produced precipitates were filtered and washed with hydrochloric acid (30%), water, and methanol. The crude product was dissolved in dichloromethane, and the insoluble by-products were filtered off. The filtrate was condensed and purified by silica gel column chromatography (eluent: dichloromethane/methanol in 20:1 ratio). The dark red powder was dissolved in dichloromethane and poured onto methanol. The dark red precipitates of compound 5 (0.81 g, 1.3 mmol) were filtered. The yield was 75%. ¹H NMR (400 MHz, CDCl₃): δ = 8.60 (s, 2H, br), 8.52 (d, 2H, J = 8.0 Hz), 8.48 (d, 2H, J = 8.0 Hz), 8.44 (d, 2H, J = 8.0 Hz), 2H, J = 8.0 Hz), 5.82 (ddt, 2H, J = 17.2, 10.4, 6.4 Hz), 5.24 (tt, 1H, J = 9.6, 5.6 Hz), 4.98 (dd, 2H, J = 17.2, 2.0 Hz), 4.89 (dd, 2H, J = 6.4, 2.0 Hz), 4.14 (t, 2H, J = 7.6 Hz), 2.37-2.46 (2H, m), 2.05-2.18 (m, 4H), 1.93-2.02 (m, 2H), 1.71 (quint, 2H, J = 7.2 Hz), 1.20-1.45 (m, 12H), 0.86 (t, 3H, J = 6.8 Hz) ppm; ¹³C NMR (100 MHz CDCl₃): $\delta = 14.3, 22.8, 27.4, 28.3, 29.4, 29.5, 31.3, 31.6, 32.0, 40.9,$ 54.0, 115.1, 123.1, 123.2, 131.4, 134.5, 134.6, 135.0, 135.1, 138.2, 163.4 ppm; IR (ATR): *v* = 2954, 2923, 2853, 1693, 1646, 1593, 1577, 1437, 1403, 1345, 1247, 1168, 1124, 1096, 992, 909, 851, 808, 744, 611, 484, 462, 431, 420, 394 cm⁻¹; Exact Mass: 624.30; molecular weight: 624.77, m/z: 624.0, 625.0, 626.0, 627.0; elemental analysis (%) calculated for C₄₁H₄₀N₂O₄: C, 78.82; H, 6.45; N, 4.48; O, 10.24; found: C, 78.5; H, 6.48; N, 4.51.



N-(1,8-nonadiene-5-yl)-*N*' -(3,6,9-trioxodecane-1-yl)perylene-3,4:9,10-tetracarboxylic bisimide (6)

*N,N*²-di(1,8-nonadiene-5-yl)-perylene-3,4:9,10-tetracarboxylic bisimide (**3**) (1.07 g, 1.7 mmol) and crashed sodium hydroxide (1.0 g) were suspended in *tert*-butyl alcohol (30 mL) and heated to 100 $^{\circ}$ C for 2 h. Acetic acid (30 mL) was added to the solution and the mixture was stirred for 2 h. To the resulting solution, 2 N hydrochloric acid (10 mL) was added. The dark red precipitates were filtered and washed with methanol. The dark red powder of compound **4** (0.87 g, 1.7 mmol) was obtained. The crude product was used for the next condensation reaction without further purification. The dark red powder (0.81 g, 1.6 mmol), 1-amino-3,6,9-trioxodecane (1.81 g, 11 mmol), and zinc(II) acetate (0.44 g, 2.4 mmol) were suspended in quinoline (30 mL) and heated to 150 °C for 2 h. The

reaction mixture was cooled, and hydrochloric acid (30%) was added to it. The produced precipitates were filtered and washed with hydrochloric acid (30%), water, and methanol. The crude product was dissolved in dichloromethane, and the insoluble by-products were filtered off. The filtrate was condensed and purified by silica gel column chromatography (eluent: dichloromethane/methanol in 100:1 ratio). Unreacted material 3 and PTCBI derivatives bearing two triethylene oxide chains can be removed in this process. The dark red powder was dissolved in dichloromethane and poured onto methanol. The dark red precipitates of compound 6 (0.55 g, 0.8 mmol) were filtered. The yield was 52%. ¹H NMR (400 MHz, CDCl₃): δ = 8.58 (s, 2H, br), 8.47 (d, 2H, J = 8.0 Hz), 8.45 (d, 2H, J = 8.0 Hz), 8.38 (d, 2H, J = 8.0 Hz), 5.82 (ddt, 2H, J = 17.2, 10.4, 6.4 Hz), 5.24 (tt, 1H, J = 8.4, 5.6 Hz), 4.98 (dd, 2H, J = 17.2, 3.2 Hz), 4.90 (dd, 2H, J = 10.4, 3.2 Hz), 4.41 (t, 2H, J = 6.0 Hz), 3.84 (t, 2H, J = 0.0 Hz), 3.84 J = 6.0 Hz), 3.73 (t, 2H, J = 4.2 Hz), 3.62 (t, 2H, J = 4.2), 3.58 (dd, 2H, J = 6.0, 4.8 Hz), 3.44 (dd, 2H, 4.8 Hz J = 6.0, 4.8 Hz, 3.29 (3H, s), 2.37-2.47 (m, 2H), 2.06-2.19 (m, 4H), 1.94-2.06 (m, 2H) ppm; ¹³C NMR (100 MHz CDCl₃): δ = 31.3, 31.6, 39.6, 53.9, 59.2, 59.5, 68.1, 70.3, 70.7, 70.8, 72.1, 123.1, 123.2, 131.4, 134.4, 134.6, 138.1, 163.7 ppm; IR (ATR): *v* = 2865, 1691, 1644, 1592, 1576, 1435, 1403, 1337, 1245, 1105, 909, 856, 809, 744, 630, 430, 393 cm⁻¹; elemental analysis (%) calculated for C40H38N2O7: C, 72.93: H, 5.81: N, 4.25: O, 17.00: found: C, 5.80: H, 4.45: N, 4.36.



Characterization of mesophases

The mesomorphic properties of the PTCBI derivatives were studied by differential scanning calorimetry (DSC), polarizing optical microscopy, and X-ray diffraction. A polarizing optical microscope (Olympus DP70) equipped with a hand-made hot stage was used for visual observation of the optical textures. Differential scanning calorimetry (DSC) measurements were conducted with a NETZSCH DSC 204 Phoenix instrument. X-ray diffraction (XRD) measurements were carried out on a Rigaku Rapid II diffractometer with the use of Ni-filtered CuKα radiation.

Figure S1 exhibits DSC thermograms of compounds **1** and **2**. For compound **1**, two mesophases are present during the cooling process. The high-temperature phase appears between 105 and 142 °C. The low-temperature phase is retained as the sample is cooled to -100 °C. During the heating process, the low-temperature phase partially crystallizes at 134 °C and melts to the isotropic phase at 184 °C. Around -50 °C, a glass transition occurs, which is due to the thermal motion of the cyclotetrasiloxane rings. For compound **2**, a single mesophase is present below 176 °C during the cooling process.



Figure S1 DSC thermograms of compounds (a) **1** and (b) **2**. The heating and cooling rates were both $10 \text{ K} \cdot \text{min}^{-1}$.

Production of thin films and in situ polymerization

A toluene solution (10 wt %) of compound 2 was spin-coated on glass substrates. Spin-coating was performed at a rotation speed of 1000 rpm for 10 s, 1500 rpm for 20 s, and 2000 rpm for 30 s. The deposited films were dried under vacuum for one day.

The thin film deposited on the glass substrate was placed in a Petri dish. A small glass dish containing several drops of trifluoromethane sulfonic acid was also placed in the dish which was capped with another Petri dish. The dish was placed in an oven set at 80 °C and kept for 1 h. After exposure to the acid vapors, the thin film was immersed in a toluene solution of triethylamine for 15 min in order to remove absorbed acid from the polymerized thin film.

The thickness of the deposited thin films was determined by using a DEKTAK 6 instrument. UV-Vis absorption spectra were acquired by using a JASCO Ubest V-530iRM instrument.

Measurement of carrier mobility in the LC phases

The electron mobilities were measured by the time-of-flight (TOF) method. A liquid crystal cell was fabricated by combining the two ITO-coated glass plates. The surface of the ITO-coated substrates was cleaned by UV-O₃ treatment. The cell was placed on a hot stage and heated at 140 °C. Compounds **1** and **2** were melted and capillary-filled into the cells. The cell was cooled to 130 °C at a rate of 0.1 °C /min. Dark domains in which columnar stacks were homeotropically aligned were observed under an optical microscope as shown in Figure S2. The liquid crystal cell was placed on a hot stage of the TOF setup. DC voltage was applied to the cell using an electrometer (ADC R8252) and a pulse laser was used to irradiate the cell. The third harmonic of a Nd:YAG laser (Continuum MiniLite II, wavelength = 356 nm, pulse duration: 2 ns) was used as the excitation source, and induced displacement currents were recorded by a digital oscilloscope (Tektronics TDS 3044B) through a serial resistor.



Figure S2 Polarizing optical micrographs of the samples for the TOF measurement at room temperature. (a) Compound 1 and (b) compound 2.

TOF measurements of the electron mobilities of compounds 1 and 2

Figure S5(a) shows the transient photocurrent curves for electrons in the columnar phase of compound **1** at room temperature. The curves show that the field-independent electron mobility exceeds 3×10^{-2} cm²·V⁻¹·s⁻¹. Figure S5(b) shows the transient photocurrent curves for electrons in the dimeric columnar phase of compound **2** at room temperature. The electron mobility was determined to be 2.5×10^{-3} cm²·V⁻¹·s⁻¹ and field-independent. In general, the carrier mobility along the columnar axis is much higher than that perpendicular to the axis. The photocurrent signals were weak compared to those obtained with compound **1**, which is due to the tendency of the columnar aggregates to align homogenously



Figure S3 Transient photocurrent curves for electrons in the (a) columnar phase of compound **1** and (b) smectic phase of compound **2** at room temperature. The sample thickness was 15 μ m and the wavelength of the excitation light was 356 nm.

IR transmission spectra of as-deposited and polymerized thin films of compounds 1 and 2

Figure S3 exhibits IR transmission spectra of as-deposited and polymerized thin films of compound **1** and **2**. Strong absorption bands between 1000 and 1100 cm⁻¹ are attributed to stretching of Si-O bonds. The spectra of the thin films of compounds **1** and **2** do not change during the polymerization process, except for the absorption band between 1000 and 1100 cm⁻¹.



Figure S4 IR transmission spectra of the as-deposited and polymerized thin films of (a) compound **1** and (b) compound **2**.

X-ray diffraction of polymerized thin films

Figure S4 exhibits X-ray diffraction patterns of spin-coated thin films of compounds 1 and 2. In the polymerized film of compound 1, low-angle diffraction peaks became broader although the high-angle peak at $2\theta = 25^{\circ}$ attributed to $\pi - \pi$ stacking remained. In the polymerized thin films, the columnar structure before the polymerization was retained although the LC structure was disordered. In the polymerized thin film of compound 2, low-angle peaks were ambiguous and the high-angle peak at $2\theta = 25^{\circ}$ disappeared although several low-angle peaks remained. The molecular aggregation structure of the polymerized thin film of compound 2 should be disordered during the polymerization process. However the columnar structure should be retained in the polymerized thin film. Due to the limitation of the X-ray diffractometer, diffraction peaks which should appeared lower than $\theta = 2^{\circ}$ because of the dimeric structure could not be observed.



Figure S5 X-ray diffraction patterns of polymerized thin films of compounds **1** and **2** at room temperature. The arrows indicate diffraction peaks suggesting LC structures in the thin film states.

Doping of the thin films of compound 1 and 2

The thin films compounds **1** and **2** were dipped in an aqueous solution of sodium dithionite (0.1 M) and sodium hydroxide (0.1 M). Absorption spectra of the doped thin films were measured in the alkaline solution of sodium dithionite using the ALS SEC 2000 spectrometer system.

Figure S6 exhibits absorption spectra of as-deposited and polymerized thin films of compound 1 dipped in the aqueous solution of sodium dithionate. In contrast to the case of compound 2, the absorption spectrum of the as-deposited and polymerized thin films of compound 1 did not changed in the aqueous solution of sodium dithionite.



Figure S6 Absorption spectra of the (a) as-deposited and (b) polymerized thin films of compound **1** in the aqueous solution of sodium dithionite (0.2 M).

Measurement of electrical conductivities of the thin films of compounds 1 and 2

Patterned ITO substrates with planar electrodes were produced by the etching in concentrate hydrochloric acid. The patterned electrodes were cleaned by the UV-ozone treatment. On the patterned electrode, the toluene solutions of compounds 1 and 2 were spin-coated. When the substrate surface was not treated, we obtained polydomain films as shown in Figure 6. On the friction transferred substrates, uniaxially aligned thin films was obtained.

Figure S7 exhibits I-V characteristics of as-deposited and polymerized thin films of compound **1** for the non-doped and doped states. These films were polydomain samples deposited on non-treated substrates. The electrical conductivities of the as-deposited thin film in the non-doped and doped states were 3.2×10^{-10} Scm⁻¹ and 1.4×10^{-8} Scm⁻¹, respectively. Those of the polymerized thin films in the non-doped and doped states were 1.1×10^{-9} Scm⁻¹ and 5.2×10^{-9} Scm⁻¹, respectively. For the spin-coated thin films of compound **1**, the electrical conductivities increased only by the factor of 44 and 4.7 in the non-polymerized and polymerized states, while they increased by the factor of 10^5 for the thin films of compound **2**.

Figure S8 exhibits I-V characteristics of as-deposited and polymerized thin films of compound 2 for the non-doped and doped states. These films were also deposited on non-treated substrates and

they were polydomain samples. The electrical conductivity of as-deposited and polymerized thin films in the doped states increased from the order of 10^{-10} Scm⁻¹ to 10^{-5} Scm⁻¹.



Figure S7 I-V characteristics of (a) as-deposited and (b) polymerized thin films of compound **1** for non-doped and doped states. The sample thickness was 500 nm, and the electrodes gap was 100 μ m.



Figure S8 Current-voltage characteristics of the as-deposited and polymerized thin films of compound **2** at room temperature.

X-ray diffraction analysis of the columnar phases of compounds 1 and 2

| 1 | (| | (100,) |
|-------------------|-------------------------|-------|--------------|
| 2 <i>θ</i> / deg. | $d (\exp) / \text{\AA}$ | index | d (calc.)/ Å |
| 3.28 | 26.94 | 010 | 27.30 |
| 5.18 | 17.06 | 200 | 17.40 |
| 5.87 | 15.06 | 210 | 14.67 |
| 6.42 | 13.77 | 020 | 13.65 |
| 7.52 | 11.76 | 300 | 11.60 |
| 8.11 | 10.90 | 220 | 10.74 |
| 8.55 | 10.34 | 310 | 10.68 |
| 10.27 | 8.61 | 400 | 8.70 |
| 11.80 | 7.50 | 330 | 7.16 |
| 12.50 | 7.08 | 500 | 6.96 |
| 17.15 | 5.17 | 440 | 5.37 |
| 20.56 | 4.32 | 450 | 4.62 |
| 21.41 | 4.15 | 550 | 4.30 |
| 25.02 | 3.56 | 0 0 1 | 3.56 |

Table S1 Compound **1** at 30 °C (Lattice constants: a = 34.8 Å (= d_{100}), b = 27.3 Å (= d_{010}))

Table S2 Compound **1** at 150 °C (Lattice constants: a = 30.4 Å (= $2/\sqrt{3}d_{100}$))

| 2 <i>θ</i> / deg. | $d(\exp)/\text{\AA}$ | index | d (calc.)/ Å |
|-------------------|----------------------|-------|--------------|
| 3.36 | 26.29 | 100 | 26.30 |
| 5.98 | 14.78 | 110 | 15.18 |
| 9.16 | 9.65 | 210 | 9.94 |
| 10.00 | 8.85 | 300 | 8.77 |
| 11.05 | 8.01 | 220 | 7.59 |
| 12.12 | 7.30 | 310 | 7.29 |
| 13.20 | 6.71 | 400 | 6.58 |
| 14.95 | 5.93 | 320 | 6.03 |
| 15.52 | 5.71 | 410 | 5.74 |
| 17.18 | 5.16 | 500 | 5.26 |
| 20.140 | 4.41 | 600 | 4.38 |
| 22.610 | 3.93 | 440 | 3.80 |

| 2 <i>θ</i> / deg. | <i>d</i> (exp) / Å | index | <i>d</i> (calc.)/ Å |
|-------------------|--------------------|-------|---------------------|
| 2.54 | 34.78 | 100 | 55.80 |
| 3.17 | 27.87 | 200 | 27.90 |
| 5.21 | 16.96 | 110 | 16.87 |
| 5.85 | 15.11 | 210 | 14.95 |
| 6.37 | 13.88 | 400 | 13.95 |
| 7.10 | 12.45 | 310 | 12.80 |
| 7.91 | 11.18 | 500 | 11.16 |
| 8.06 | 10.97 | 410 | 10.96 |
| 8.90 | 9.82 | 510 | 9.44 |
| 9.71 | 9.11 | 600 | 9.30 |
| 10.61 | 8.34 | 220 | 8.44 |
| 11.74 | 7.54 | 420 | 7.47 |
| 12.41 | 7.13 | 520 | 6.93 |
| 25.60 | 3.48 | 0 0 1 | |

Table S3 Compound **2** at 27 °C (Lattice constants: a = 55.80 Å (= d_{100}), b = 17.70 Å (= d_{010}))

| Table S4 Compound 2 at 160 °C (Lattice constants: $a = 58.7$ Å | Å (= d_{100}), $b = 18.5$ Å | $Å (= d_{010}))$ |
|--|--------------------------------|------------------|
|--|--------------------------------|------------------|

| I | | | (100) / |
|-------------------|--------------------|-------|--------------|
| 2 <i>θ</i> / deg. | <i>d</i> (exp) / Å | index | d (calc.)/ Å |
| 3.01 | 29.35 | 200 | 29.35 |
| 5.00 | 17.67 | 110 | 17.67 |
| 6.11 | 14.46 | 400 | 14.67 |
| 9.20 | 9.612 | 600 | 9.78 |