Supplementary Material (ESI) for Journal of Materials Chemistry

Electronic Supplementary Information (ESI)

Reentrant $SmCP_A$ phases: Unusual polymorphism variant SmA–SmC_SP_A–Col_{ob}–SmC_SP_A observed in new bent-core mesogens

H. N. Shreenivasa Murthy, M. Bodyagin, S. Diele, U. Baumeister, G. Pelzl and W. Weissflog*

Martin-Luther-Universitaet Halle-Wittenberg, Institut fuer Physikalische Chemie, Muehlpforte 1, 06108 Halle (Saale) Germany

The experimental procedure to prepare compound **18** is given together with the analytical data of all final products **8, 12, 14, 16, 18, 16-Br.**

4-Chloro-1,3-phenylene bis[4-(4-*n*-octadecyloxyphenyloxycarbonyl)benzoate], 18

4-(4-*n*-Octadecyloxyphenyloxycarbonyl)benzoic acid (0.7 g; 1.37 mmol) was added to a solution of 4-chlororesorcinol (0.1 g; 0.68mmol), DCC (0.23g; 1.25 mmol) and DMAP (20 mg) in about 20 ml of dry dichloromethane. The reaction mixture was stirred for 12 h at room temperature. The solvent was evaporated, and the crude product was repeatedly recrystallised from chloroform/acetonitrile. Yield: 0.5g (65%).

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm); 8.36-8.28 (m, 8H, ArH), 7.57 (d, ^3J = 8.8Hz, 1H, Ar-H), 7.35 (d, ^4J = 2.8Hz, 1H, Ar-H), 7.20 (dd, ^3J = 8.8Hz, ^4J = 2.8Hz, 1H, Ar-H), 7.13(dd, ^3J = 8.8Hz, ^4J = 2.4 Hz, 4H, Ar-H), 6.93 (d, ^3J = 8.8Hz, 4H, Ar-H), 3.95 (t, ^3J = 6.4Hz, 4H, $-OCH_2$), 1.80-1.74 (q, 4H, $-OCH_2\underline{CH_2}$), 1.51-1.25 (m, 60H, CH_2), 0.87 (t, ^3J =6.8Hz, 6H, CH_3); elemental analysis: calculated for $C_{70}H_{93}ClO_{10}$ (%): C 74.41, H 8.30; found C 74.27, H 8.25. MS: m/z 1130 (M^+)

4-Chloro-1,3-phenylene bis[4-(4-n-octyloxyphenyloxycarbonyl)benzoate] **8**

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm); 8.36-8.28 (m, 8H, ArH), 7.57 (d, ^3J = 8.8Hz, 1H, Ar-H), 7.35 (d, ^4J = 2.8Hz, 1H, Ar-H), 7.21 (dd, ^3J = 8.8Hz, ^4J = 2.4Hz, 1H, Ar-H), 7.12(dd, ^3J = 8.8Hz, ^4J = 2.4 Hz, 4H, Ar-H), 6.93 (d, ^3J = 8.8Hz, 4H, Ar-H), 3.96 (t, ^3J = 6.4Hz, 4H, -OCH_2), 1.82-1.75 (q, 4H, -$OCH_2\underline{CH_2}$), 1.55-1.28 (m, 20H, CH_2), 0.88 (t, ^3J =6.4Hz, 6H, CH_3); elemental analysis: calculated for $C_{50}H_{53}ClO_{10}$ (%): C 70.70, H 6.29; found C 70.97, H 6.25.

4-Chloro-1,3-phenylene bis[4-(4-n-dodecyloxyphenyloxycarbonyl)benzoate] **12**

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm); 8.34-8.30 (m, 8H, ArH), 7.57 (d, ^3J = 8.8Hz, 1H, Ar-H), 7.35 (d, ^4J = 2.8Hz, 1H, Ar-H), 7.21 (dd, ^3J = 8.8Hz, ^4J = 2.4Hz, 1H, Ar-H), 7.12(dd, ^3J = 8.8Hz, ^4J = 2.4 Hz, 4H, Ar-H), 6.94 (d, ^3J = 8.8Hz, 4H, Ar-H), 3.96 (t, ^3J = 6.4Hz, 4H, -OCH_2), 1.81-1.76 (q, 4H, -$OCH_2\underline{CH_2}$), 1.54-1.30 (m, 36H, CH_2), 0.88 (t, ^3J =6.8Hz, 6H, CH_3); elemental analysis: calculated for $C_{58}H_{69}ClO_{10}$ (%): C 72.44, H 7.23; found C 72.74, H 7.25.

4-Chloro-1,3-phenylene bis[4-(4-n-tetradecyloxyphenyloxycarbonyl)benzoate] **14**

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm); 8.36-8.28 (m, 8H, ArH), 7.57 (d, ^3J = 8.8Hz, 1H, Ar-H), 7.35 (d, ^4J = 2.8Hz, 1H, Ar-H), 7.21 (dd, ^3J = 8.8Hz, ^4J = 2.8Hz, 1H, Ar-H), 7.12(dd, ^3J = 8.8Hz, ^4J = 2.0 Hz, 4H, Ar-H), 6.93 (d, ^3J = 8.8Hz, 4H, Ar-H), 3.96 (t, ^3J = 6.4Hz, 4H, -OCH_2), 1.81-1.74 (q, 4H, -$OCH_2\underline{CH_2}$), 1.51-1.30 (m, 44H, CH_2), 0.88 (t, ^3J =6.8Hz, 6H, CH_3); elemental analysis: calculated for $C_{62}H_{77}ClO_{10}$ (%): C 73.17, H 7.63; found C 72.93, H 7.50.

4-Chloro-1,3-phenylene bis[4-(4-n-hexaadecyloxyphenyloxycarbonyl)benzoate] **16**

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm); 8.36-8.28 (m, 8H, ArH), 7.57 (d, ^3J = 8.8Hz, 1H, Ar-H), 7.35 (d, ^4J = 2.4Hz, 1H, Ar-H), 7.21 (dd, ^3J = 8.8Hz, ^4J = 2.8Hz, 1H, Ar-H), 7.12(dd, ^3J = 8.8Hz, ^4J = 2.4 Hz, 4H, Ar-H), 6.93 (d, ^3J = 8.8Hz, 4H, Ar-H), 3.96 (t, ^3J = 6.4Hz, 4H, -OCH_2), 1.81-1.74 (q, 4H, -$OCH_2\underline{CH_2}$), 1.57-1.30 (m, 52H, CH_2), 0.88 (t, ^3J =6.8Hz, 6H, CH_3); elemental analysis: calculated for $C_{66}H_{85}ClO_{10}$ (%): C 73.82, H 7.98; found C 73.47, H 7.80.

4-Bromo-1,3-phenylene bis[4-(4-n-hexaadecyloxyphenyloxycarbonyl)benzoate]
16-Br

^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm); 8.37-8.28 (m, 8H, ArH), 7.72 (d, ^{3}J = 8.8Hz, 1H, Ar-H), 7.35 (d, ^{4}J = 2.8Hz, 1H, Ar-H), 7.15 (dd, ^{3}J = 8.8Hz, ^{4}J = 2.4Hz, 1H, Ar-H), 7.12(dd, ^{3}J = 8.8Hz, ^{4}J = 2.0 Hz, 4H, Ar-H), 6.93 (d, ^{3}J = 8.8Hz, 4H, Ar-H), 3.96 (t, ^{3}J = 6.8Hz, 4H, -OCH_2), 1.81-1.74 (q, 4H, -$OCH_2\underline{CH_2}$), 1.57-1.30 (m, 52H, CH_2), 0.87 (t, ^{3}J =6.8Hz, 6H, CH_3); elemental analysis: calculated for $C_{66}H_{85}BrO_{10}$ (%): C 70.89, H 7.66; found C 71.07, H 7.60.