Supporting Information (3 pages)

Novel End-capping of Rotaxane via Diels-Alder Reaction toward the Construction of C_{60}-Terminated [2]Rotaxane

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Spectroscopic data for Rotaxanes **7a-c**, **9**

7a: colorless film. ^{1}H NMR (400 MHz, $CDCl_3$, 294K) δ 7.84-7.82 (m, 2H), 7.56 (br, 2H), 7.35 (d, 2H, *J* = 7.8 Hz), 7.27-7.23 (m, 2H), 7.20 (d, 2H, *J* = 7.8 Hz), 6.88-6.77 (m, 10H), 5.25 (s, 2H), 4.66-4.62 (m, 2H), 4.49-4.45 (m, 2H), 4.13-4.09 (m, 8H), 3.78-3.70 (m, 14H), 3.51-3.44 (m, 8H), 3.20-3.09 (m, 4H), 3.02-2.96 (m, 2H), 2.15 (s, 6H) ppm. IR(NaCl) 2925, 1735, 1593, 1453, 1354, 1253, 1201, 1180, 1123, 1056, 954, 842, 750, 557, 518 cm^{-1}. FAB-MS (matrix; mNBA) m/z = 978.5 $[M-PF_6]^+$. Calcd for $C_{56}H_{68}F_6NO_{14}P\cdot(CHCl_3)_{0.1}$: C, 59.31; H, 6.04; N, 1.23%. Found: C, 59.04; H, 6.02; N, 1.22%.

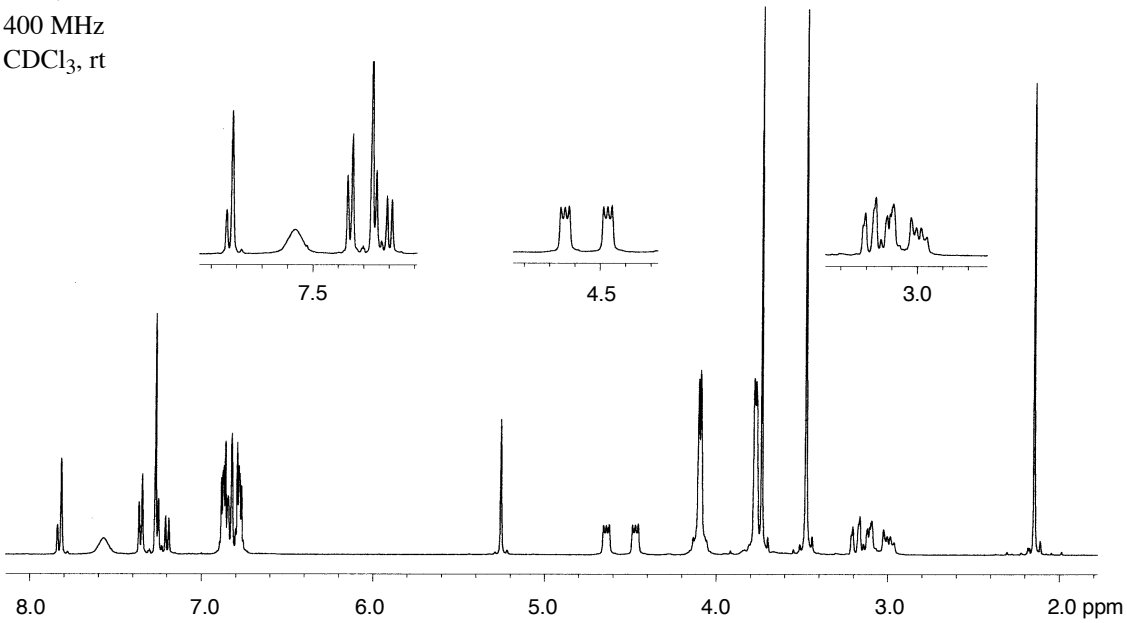
7b: colorless film. ^{1}H NMR (400 MHz, $CDCl_3$, 294K) δ 7.68-7.65 (m, 2H), 7.35-7.26 (m, 5H), 7.14 (d, 1H, *J* = 7.8 Hz), 6.90-6.85 (m, 8H), 4.66-4.63 (m, 2H), 4.24-4.09 (m, 10H), 3.85-3.61 (m, 18H), 3.46-3.41 (m, 6H), 3.20-2.90 (m, 6H), 1.95-1.92 (m, 2H), 1.19 (s, 18H) ppm. IR(NaCl) 2953, 1734, 1595, 1504, 1454, 1362, 1252, 1201, 1123, 1054, 952, 843, 751, 557 cm^{-1}. FAB-MS (matrix; mNBA) m/z = 1000.5 $[M-PF_6]^+$. Calcd for $C_{57}H_{78}F_6NO_{14}P\cdot(CHCl_3)_{0.2}$: C, 58.72; H, 6.74; N, 1.20%. Found: C, 58.72; H, 6.61; N, 1.20%.

7c: colorless film. ^{1}H NMR (400 MHz, $CDCl_3$, 294K) δ 7.91-7.88 (m, 2H), 7.57 (br, 2H), 7.36 (d, 2H, *J* = 8.3 Hz), 7.27 (d, 2H, *J* = 8.3 Hz), 6.88-6.77 (m, 12H), 5.27 (s, 2H), 4.65-4.62 (m, 2H), 4.48-4.45 (m, 2H), 4.14-4.06 (m, 8H), 3.97-3.65 (m, 18H), 3.50-3.44 (m, 8H), 2.14 (s, 6H) ppm. IR(NaCl) 2922, 1721, 1593, 1505, 1454, 1436, 1254, 1123, 1102, 1058, 954, 841, 749, 557 cm^{-1}. FAB-MS (matrix: mNBA) m/z = 976.3 $[M-PF_6]^+$. Calcd for $C_{56}H_{66}F_6NO_{14}P\cdot(CHCl_3)_{0.2}$: C, 58.90; H, 5.82; N, 1.22%. Found: C, 58.54; H, 5.66; N, 1.17%.

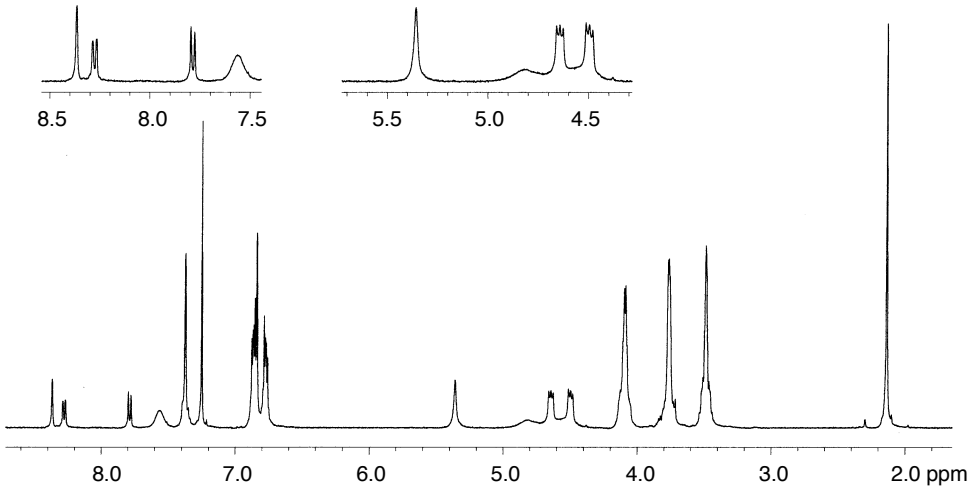
9: black purple solid. ^{1}H NMR (400MHz, $CDCl_3$, 323K) δ 9.05 (s, 1H), 8.93-8.80 (m, 8H), 8.22-7.73 (m, 16H), 7.62-7.50 (m, 4H), 7.36-6.72 (m, 12H), 5.28 (br, 2H), 4.65-4.55 (m, 4H), 4.29-3.40 (m, 28H), 2.23 (s, 6H), 1.61-1.43 (m, 54H) ppm. IR(NaCl) 2962, 2870, 2357, 1717, 1519, 1506, 1456, 1362, 1265, 1101, 845, 733, 667, 557, 527 cm^{-1}. UV-vis($CHCl_3$): 433 (Soret), 555, 596 nm. FAB-MS (matrix; mNBA) m/z = 2609.2 $[M-PF_6]^+$. Calcd for $C_{179}H_{135}F_6N_6O_{11}PZn\cdot(CHCl_3)_{0.25}$: C, 77.27; H, 4.89; N, 3.02%. Found: C, 76.99; H, 5.11; N, 2.90%.

Selected ^{1}H-NMR spectra of Rotaxanes **7a**, **7d**, **9** and UV-vis spectrum of **9**.

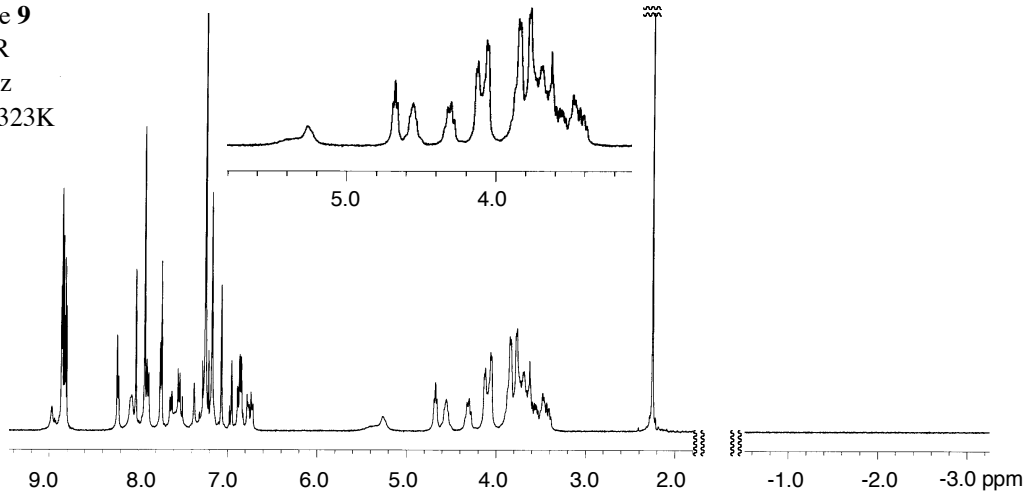
Rotaxane **7a**
^{1}H-NMR
400 MHz
$CDCl_3$, rt

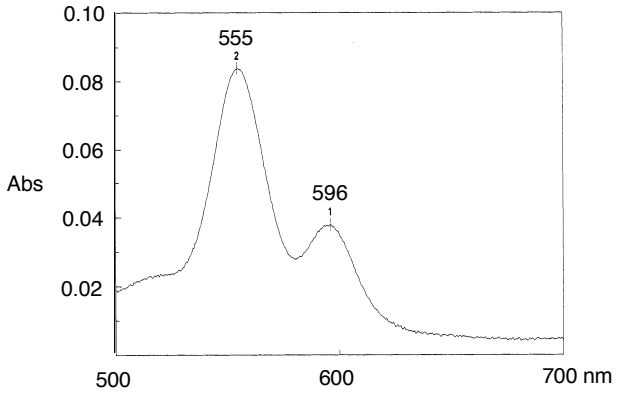


Rotaxane **7d**
^{1}H-NMR
400 MHz
$CDCl_3$, 323K



Rotaxane **9**
^{1}H-NMR
400 MHz
$CDCl_3$, 323K





Rotaxane **9**
UV-vis spectrum in $CDCl_3$, 293K (0.005 mM)