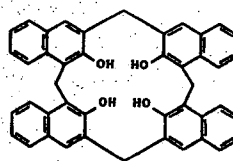


SUPPLEMENTARY INFORMATION

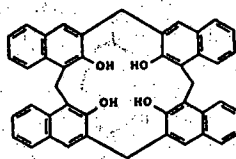
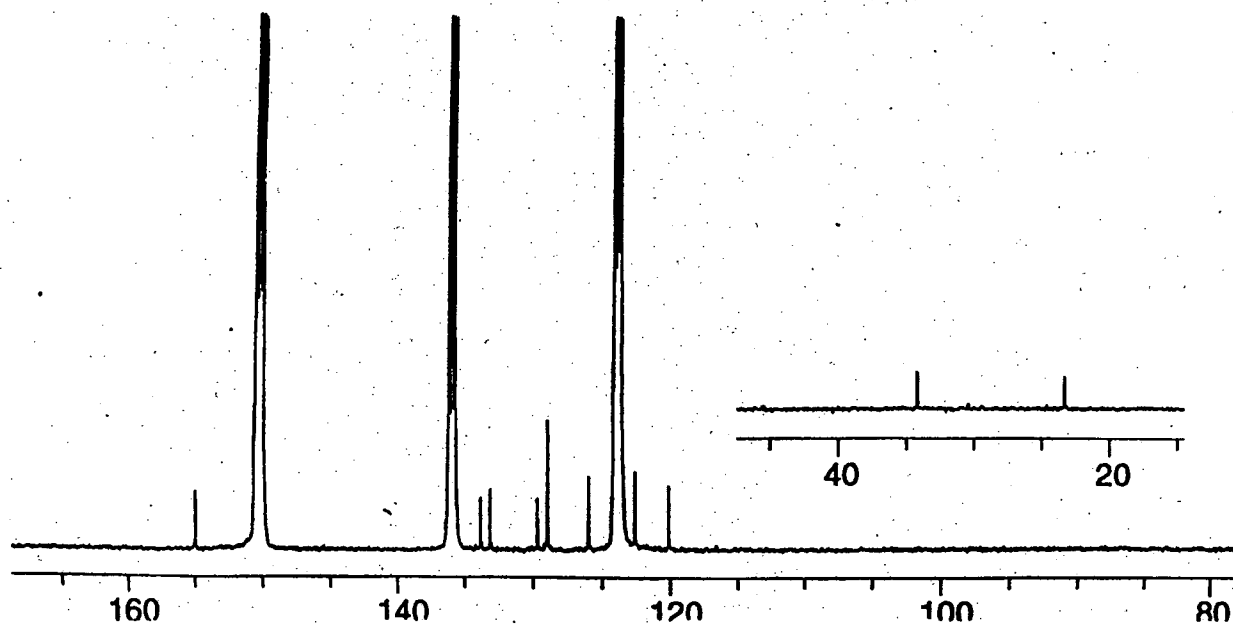
¹H and ¹³C NMR spectra of compounds **2-14a/d**.....Pages S1-S17

Additional Experimental Section details.....Pages S18-S27

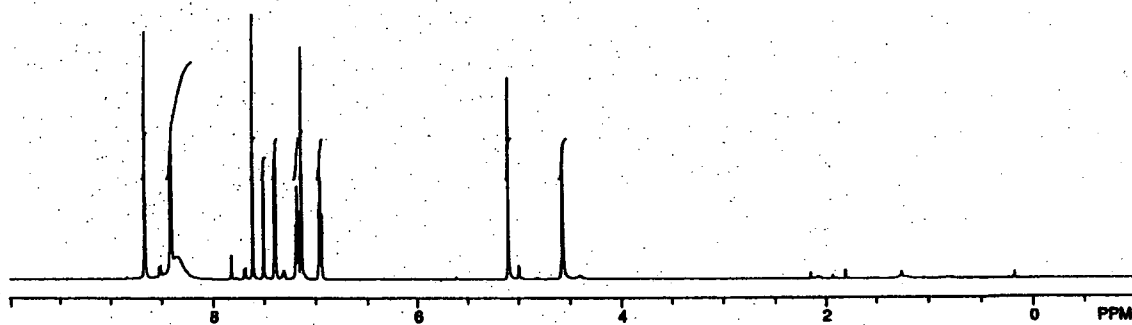
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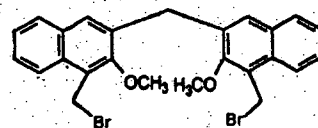
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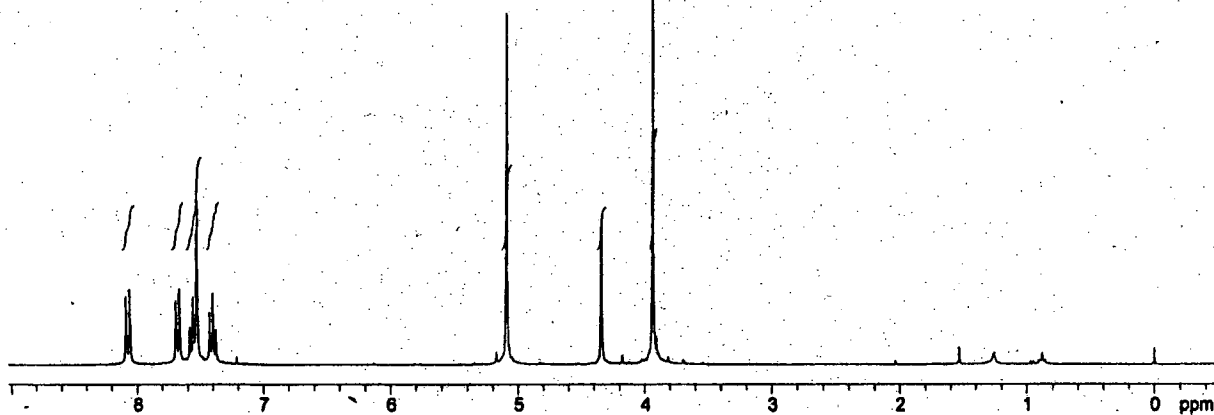
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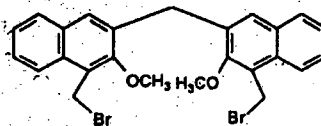
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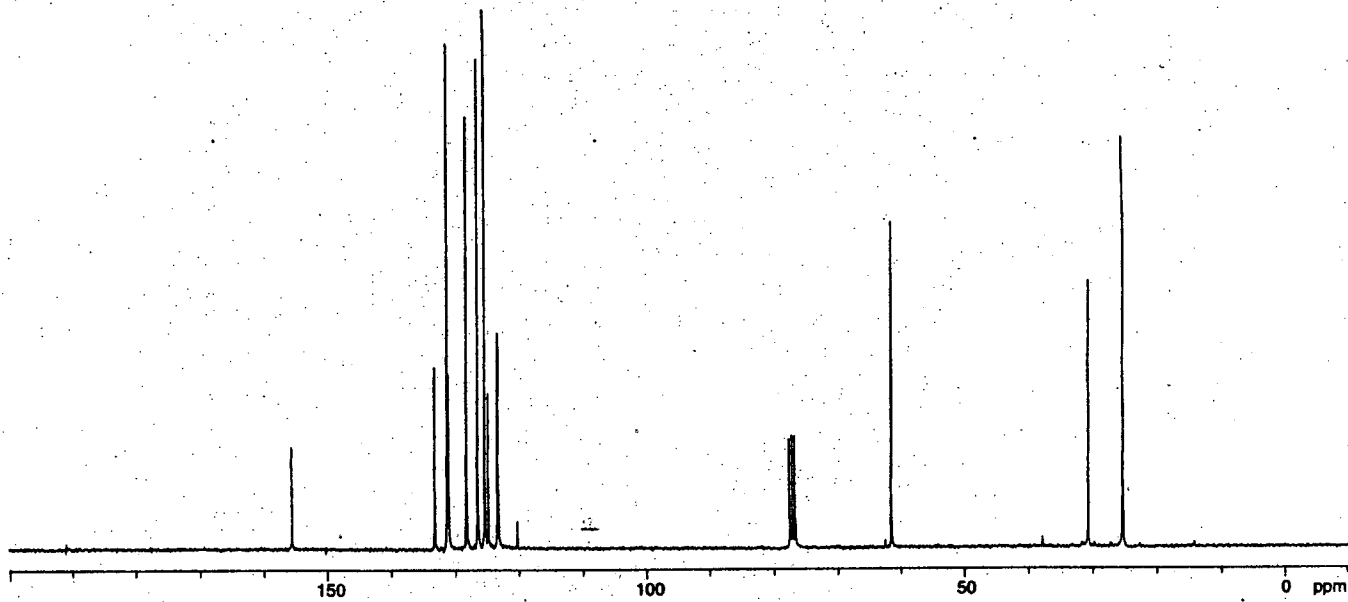
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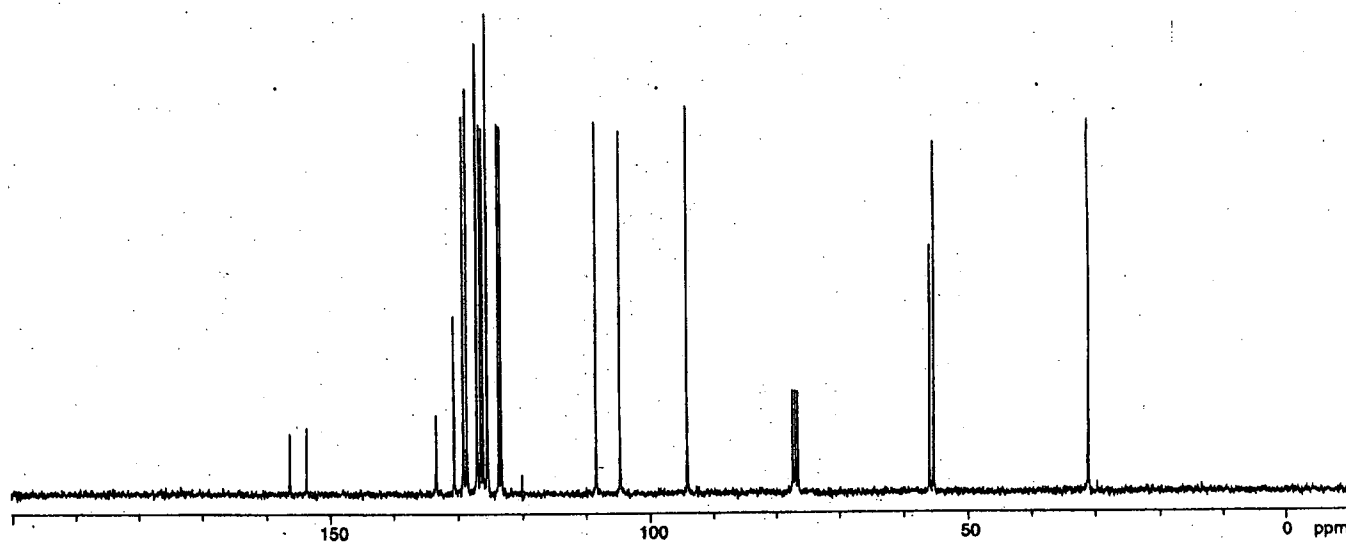
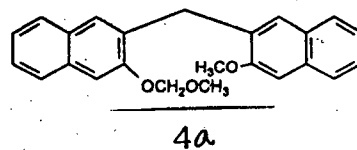
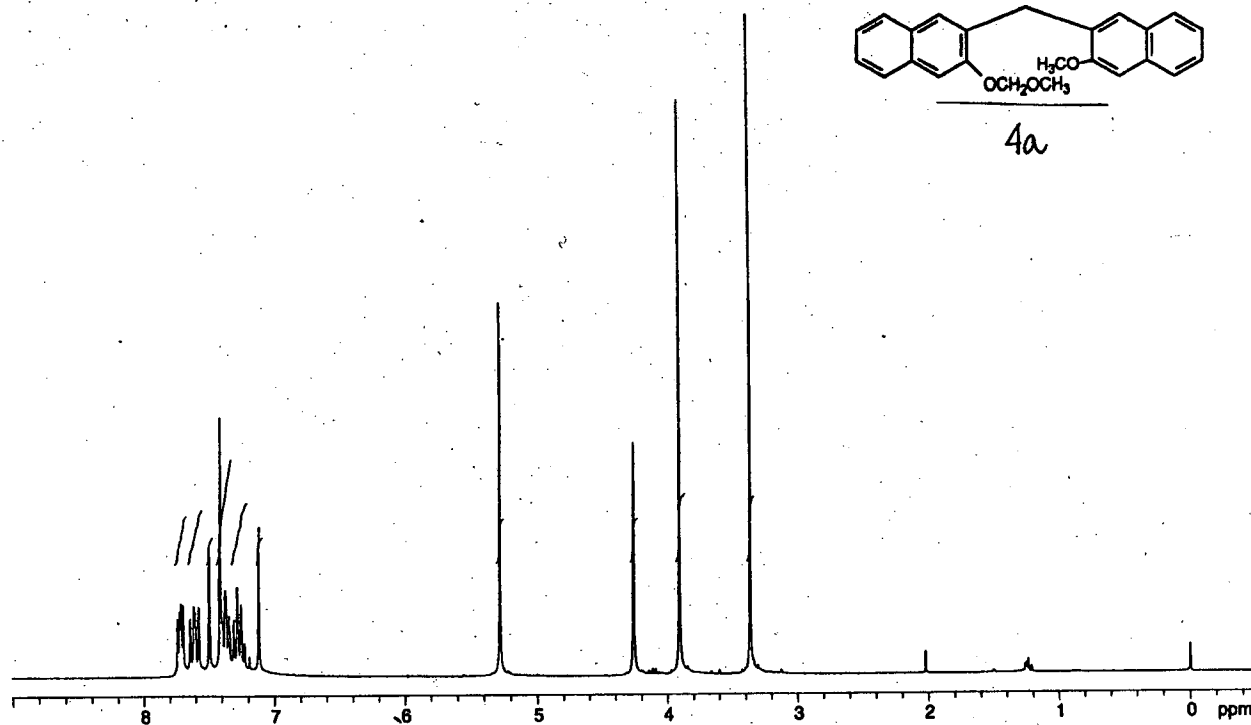
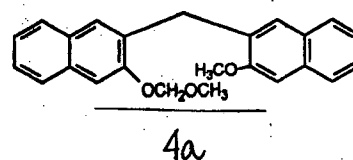
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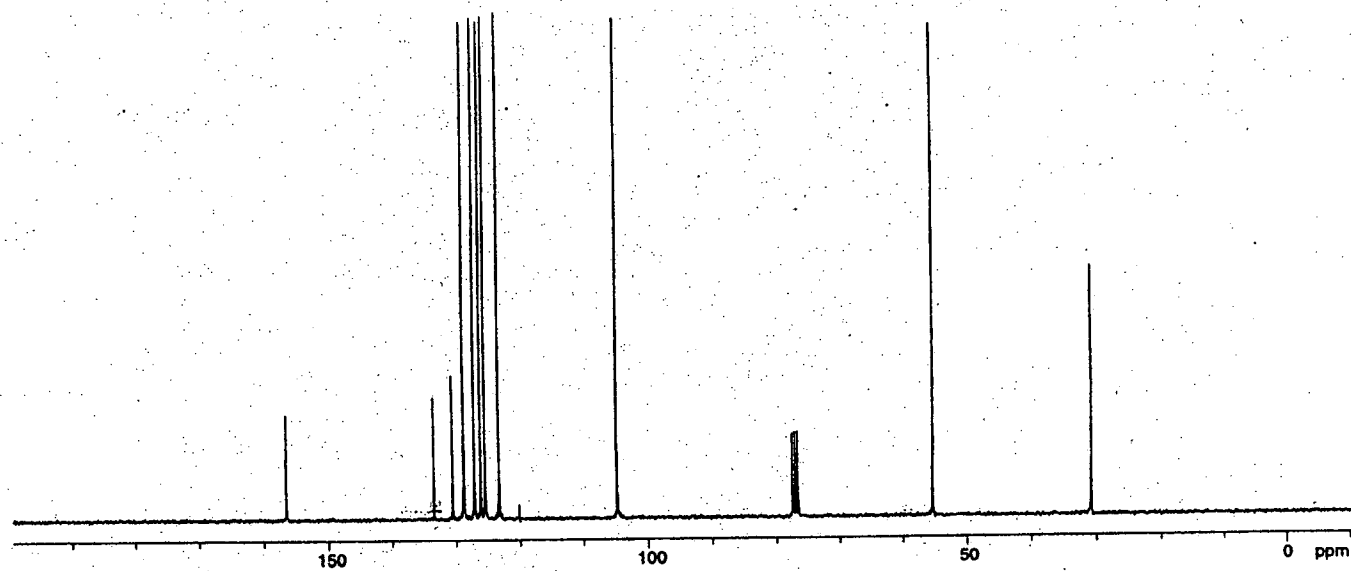
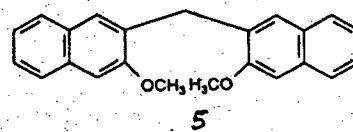
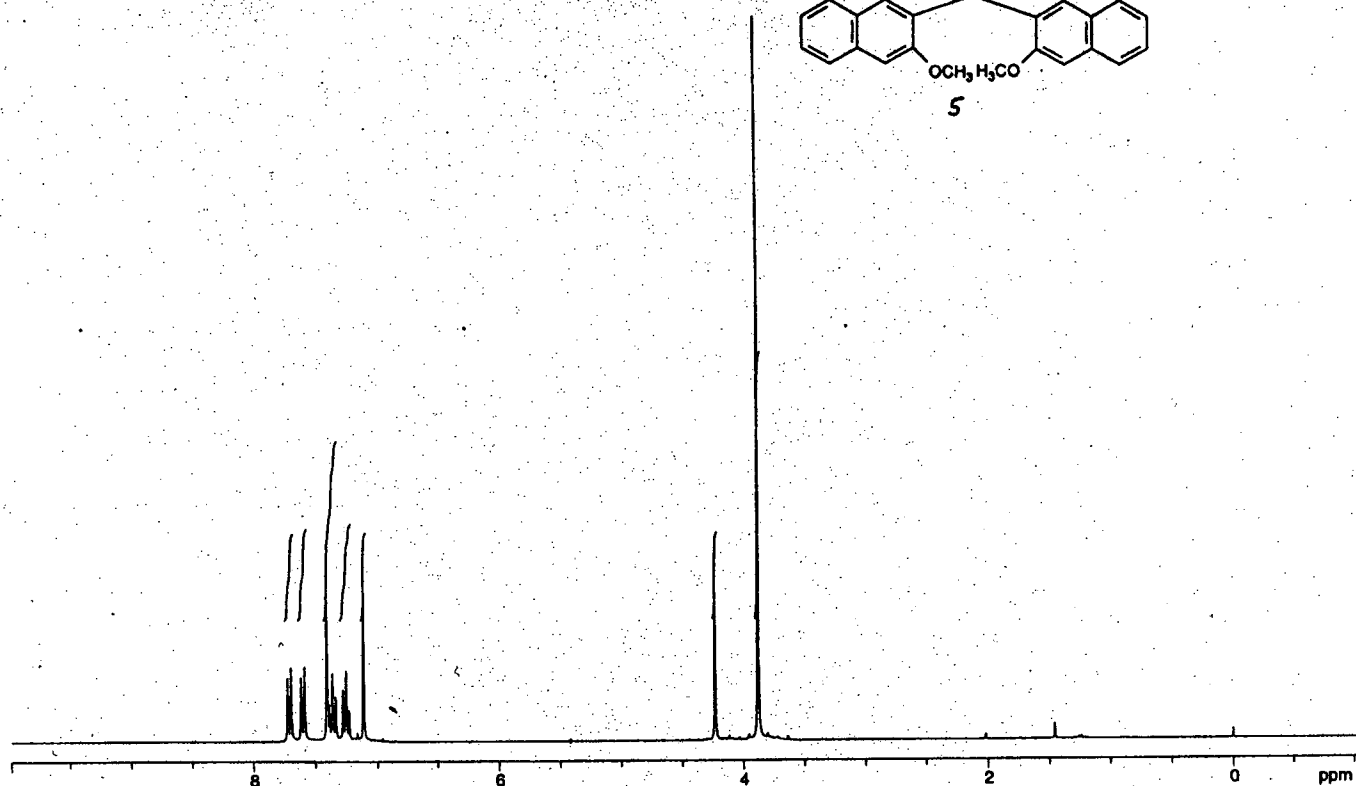
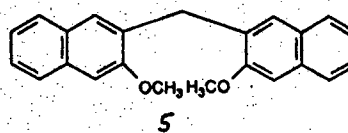
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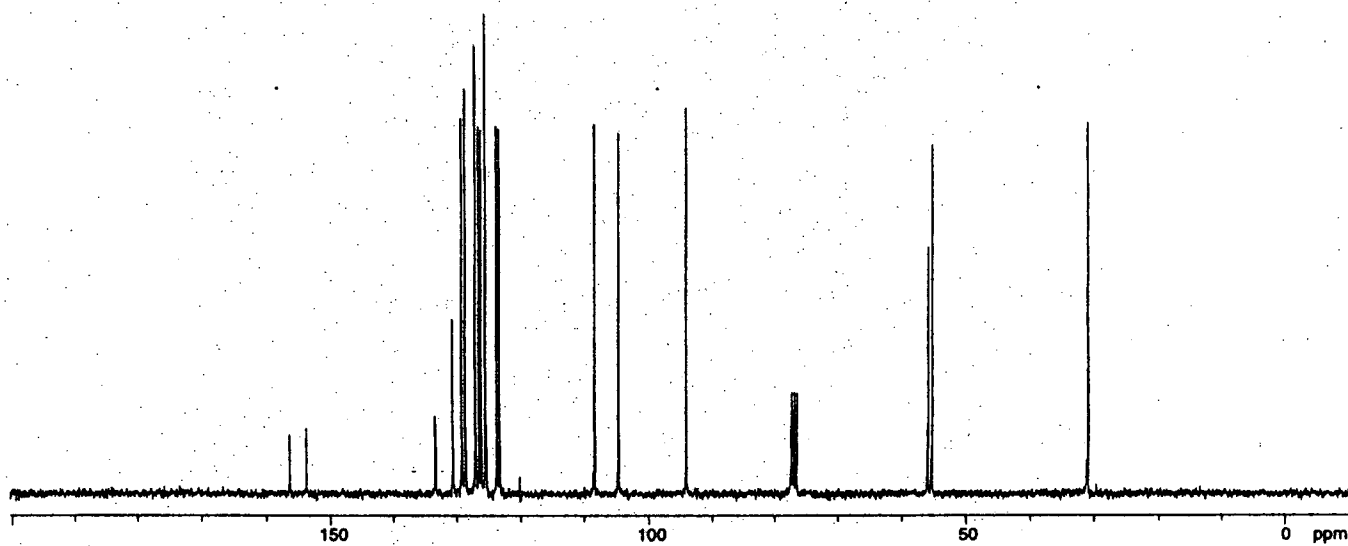
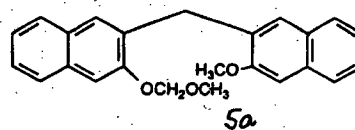
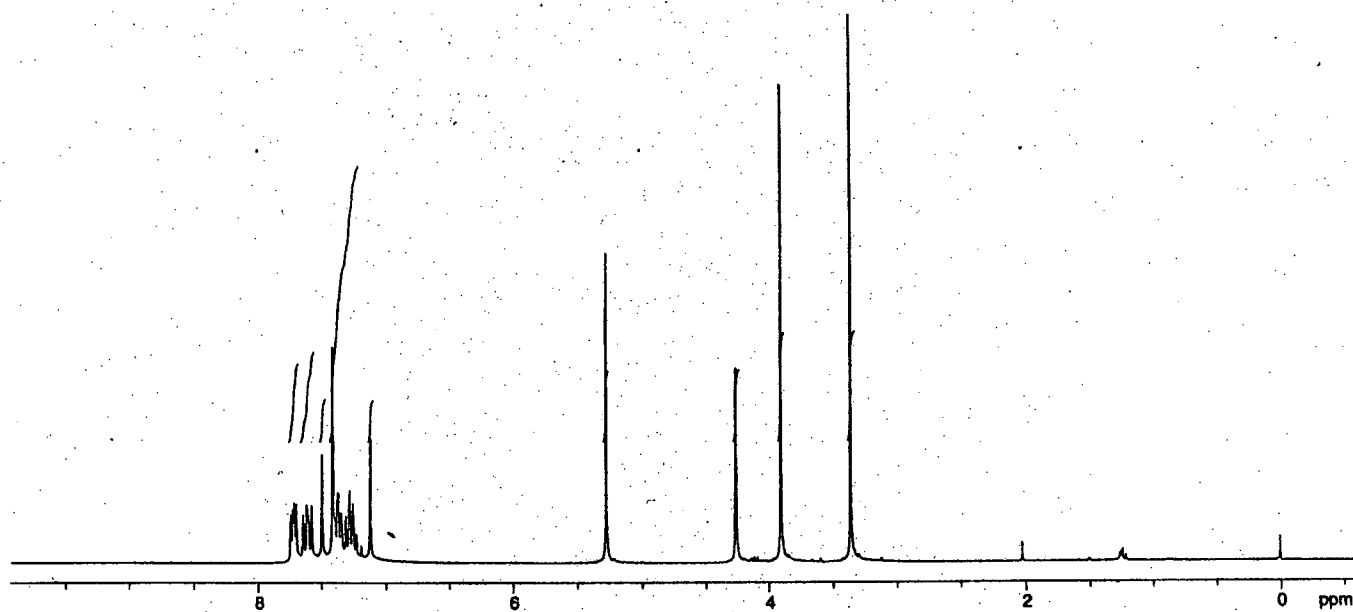
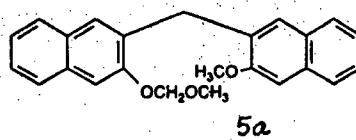
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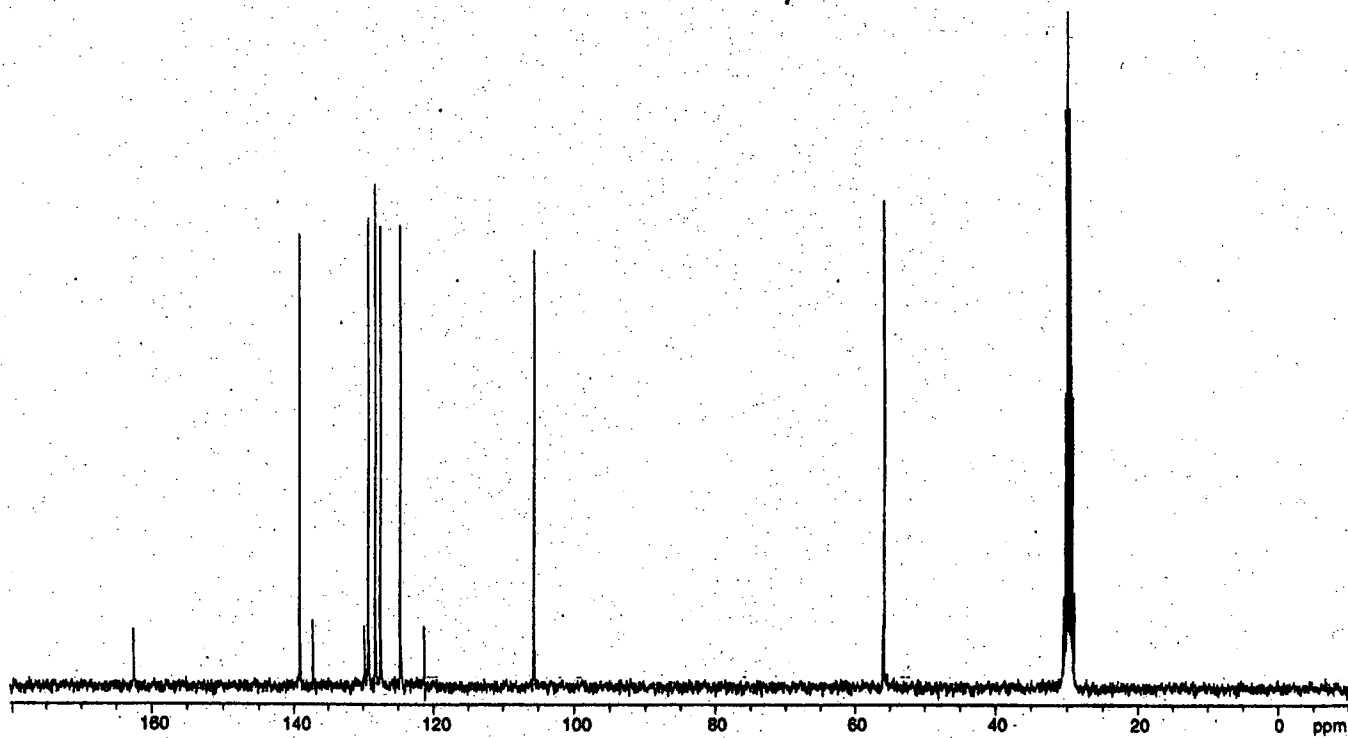
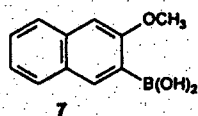
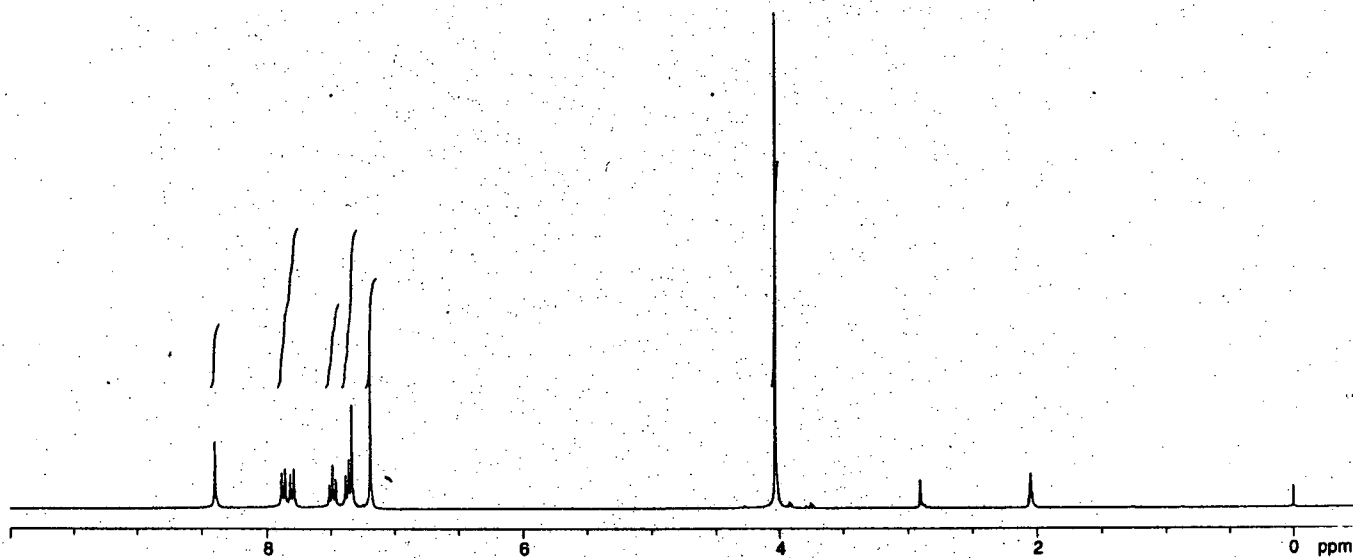
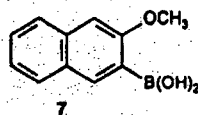
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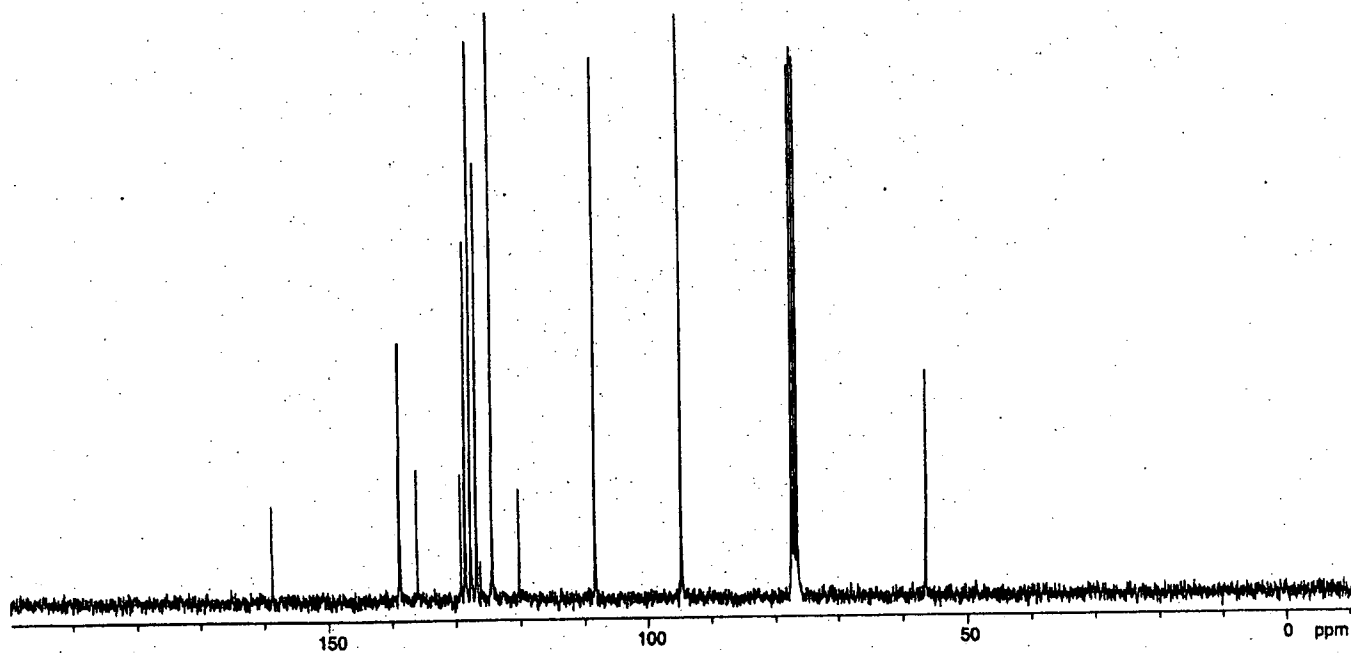
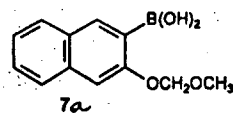
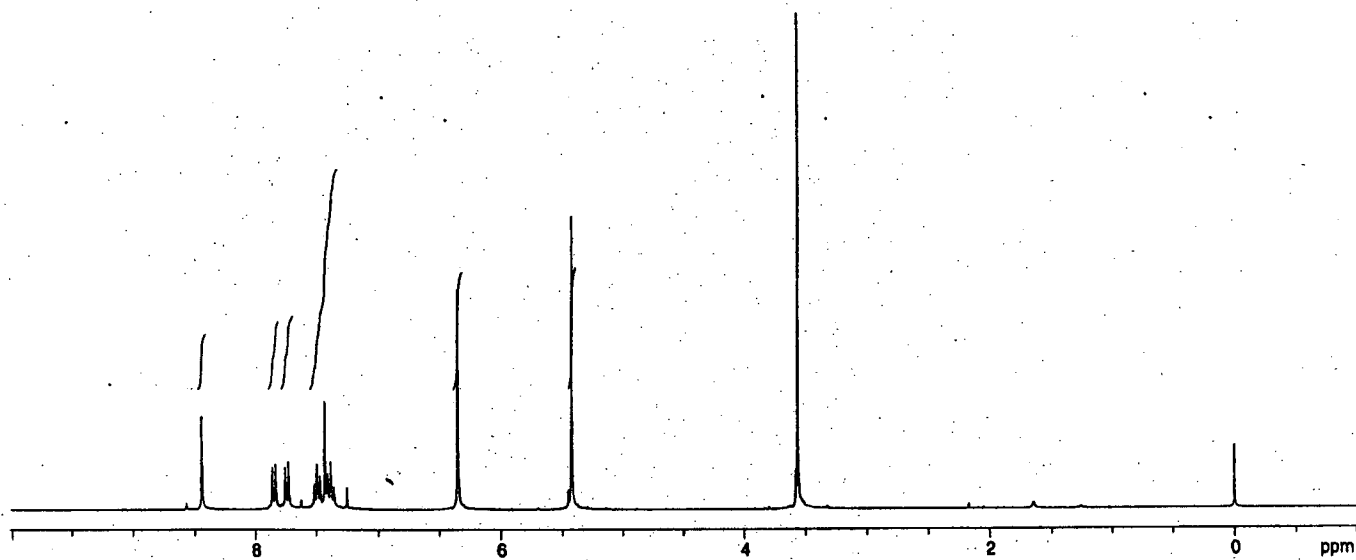
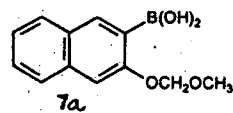
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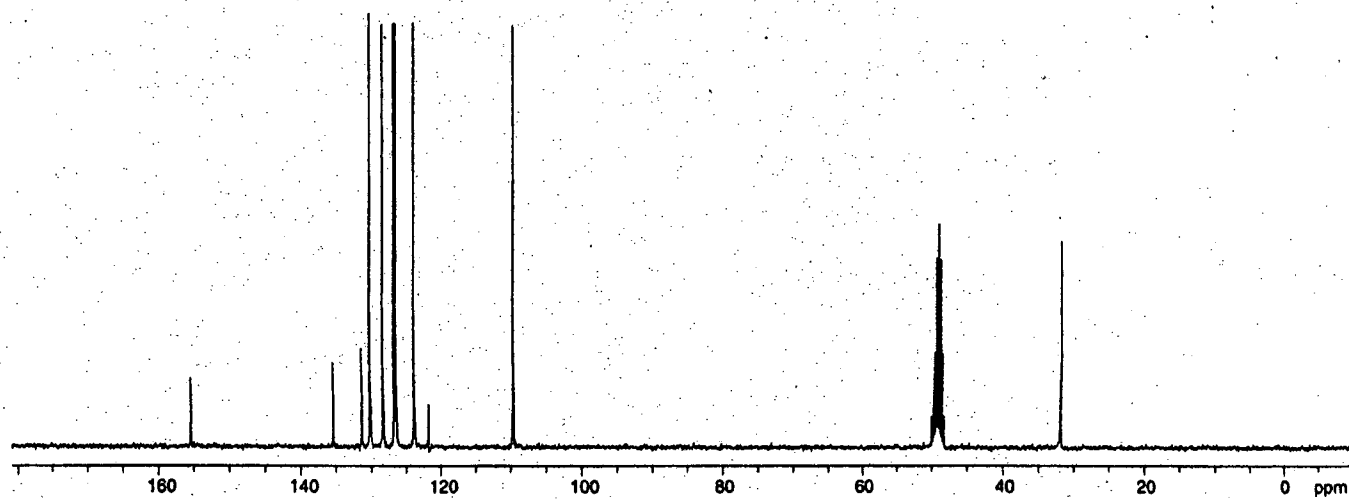
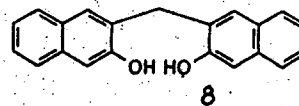
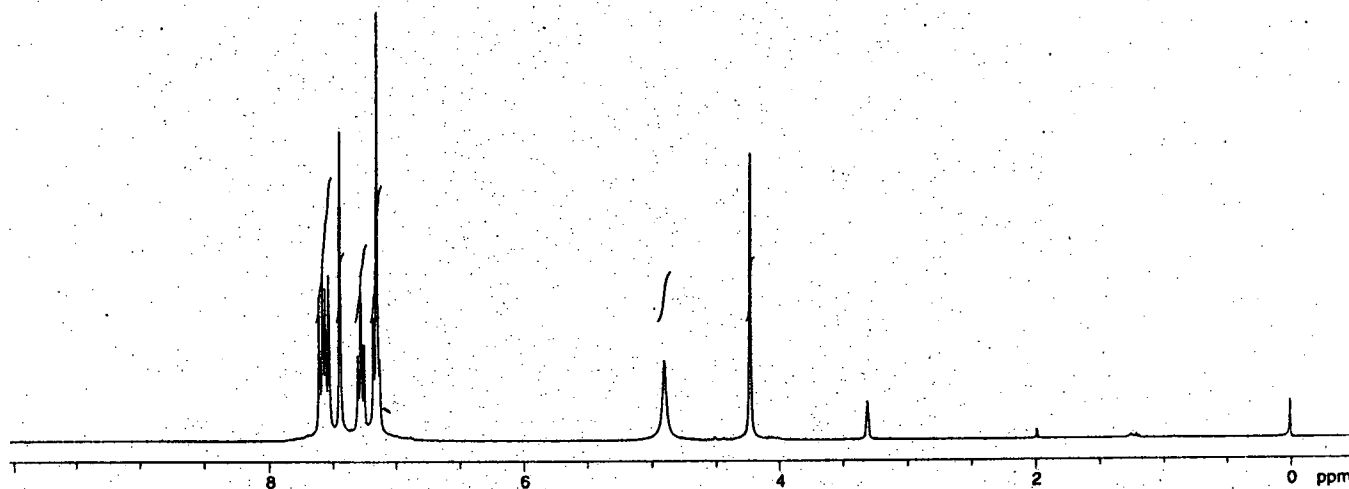
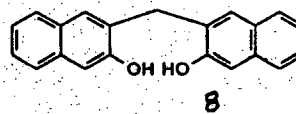
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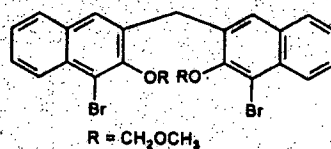
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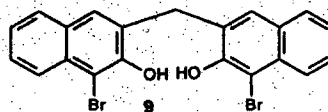
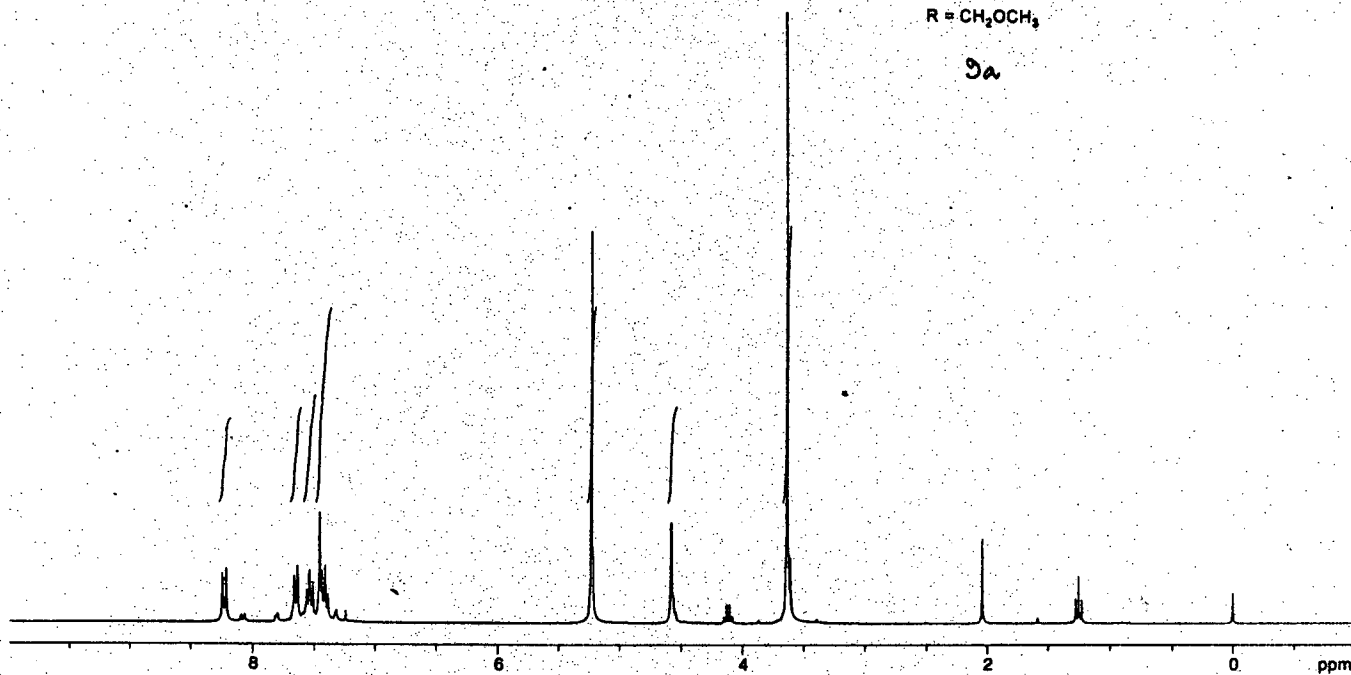
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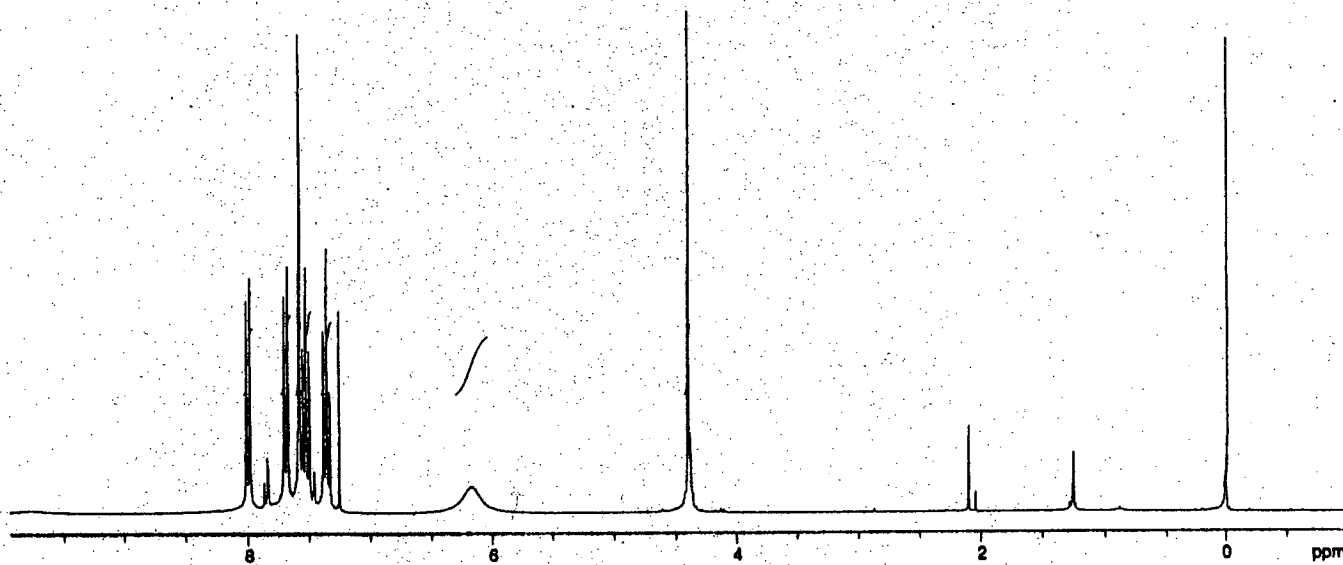
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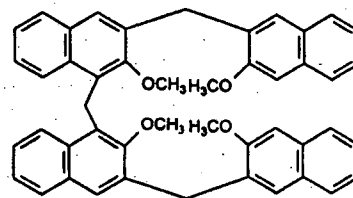
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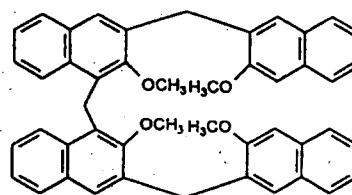
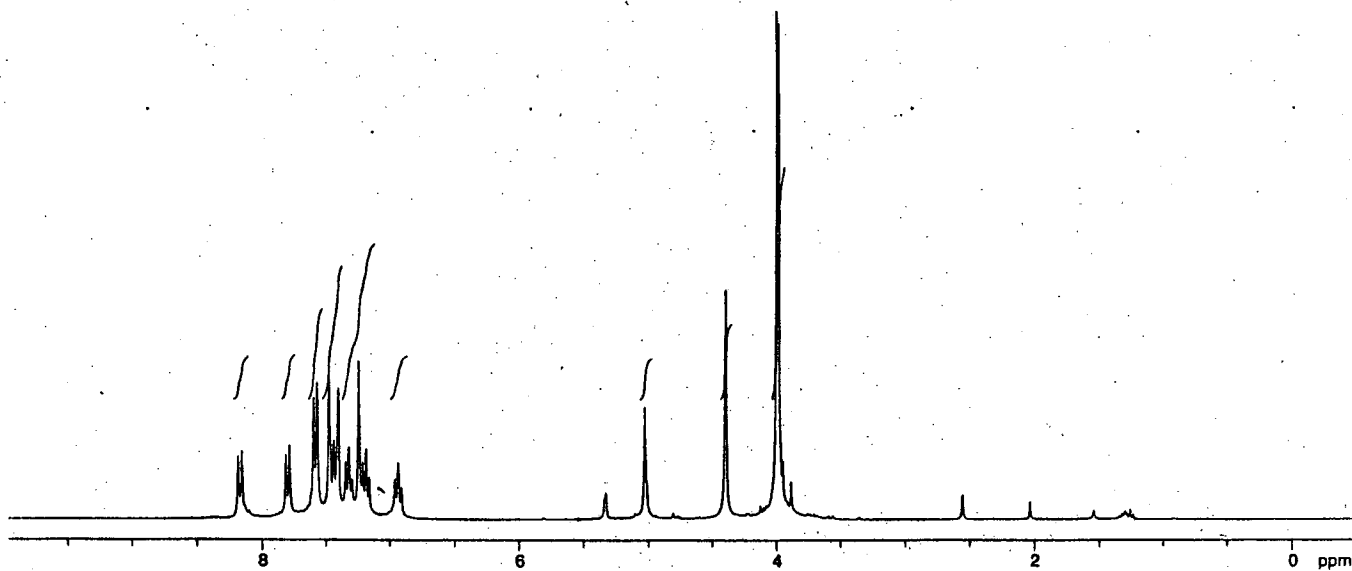
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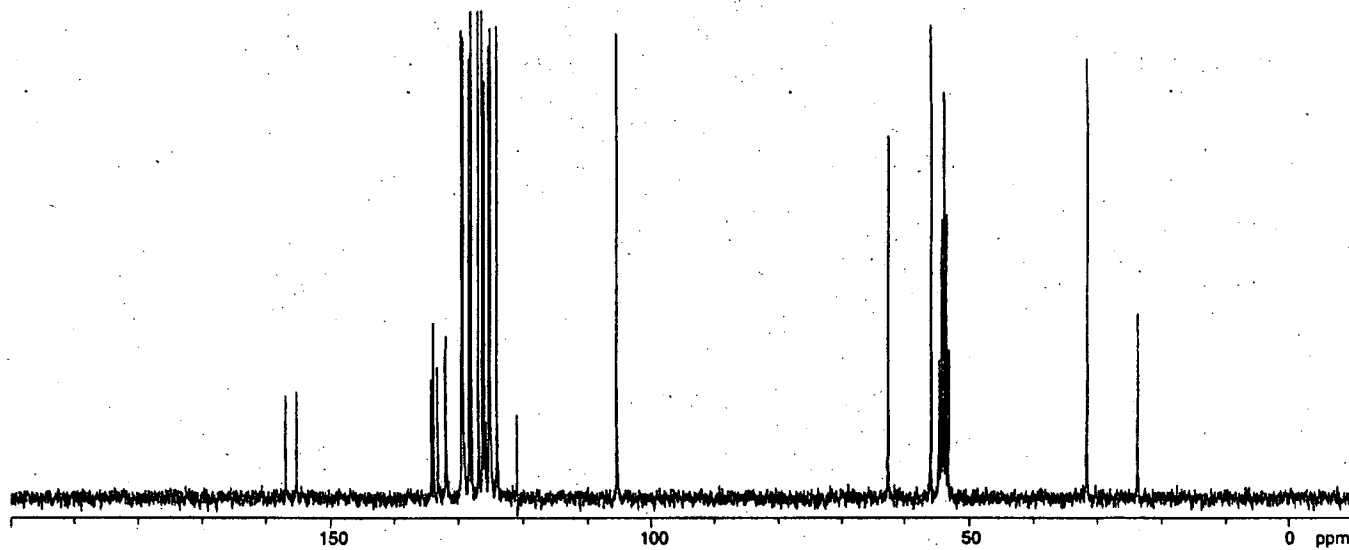
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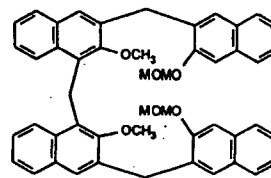
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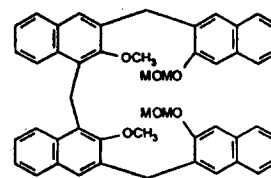
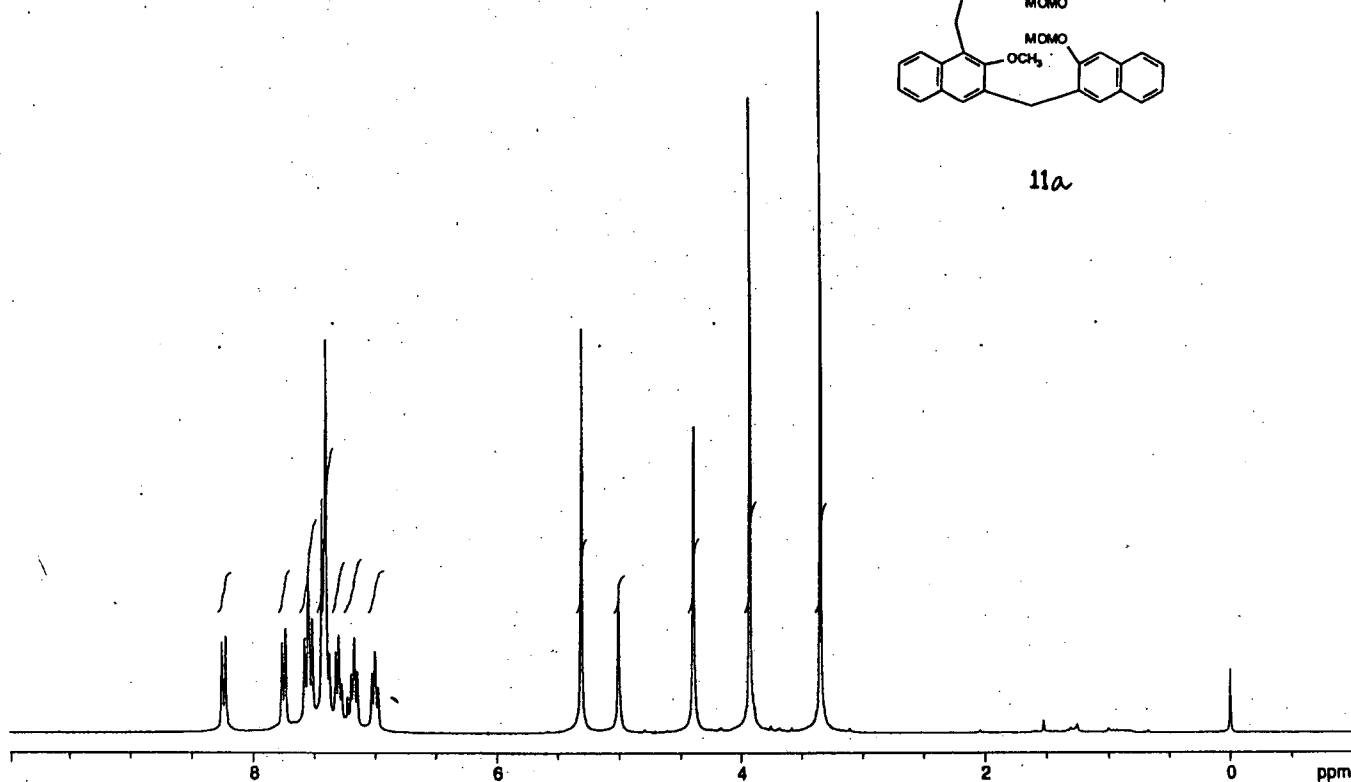
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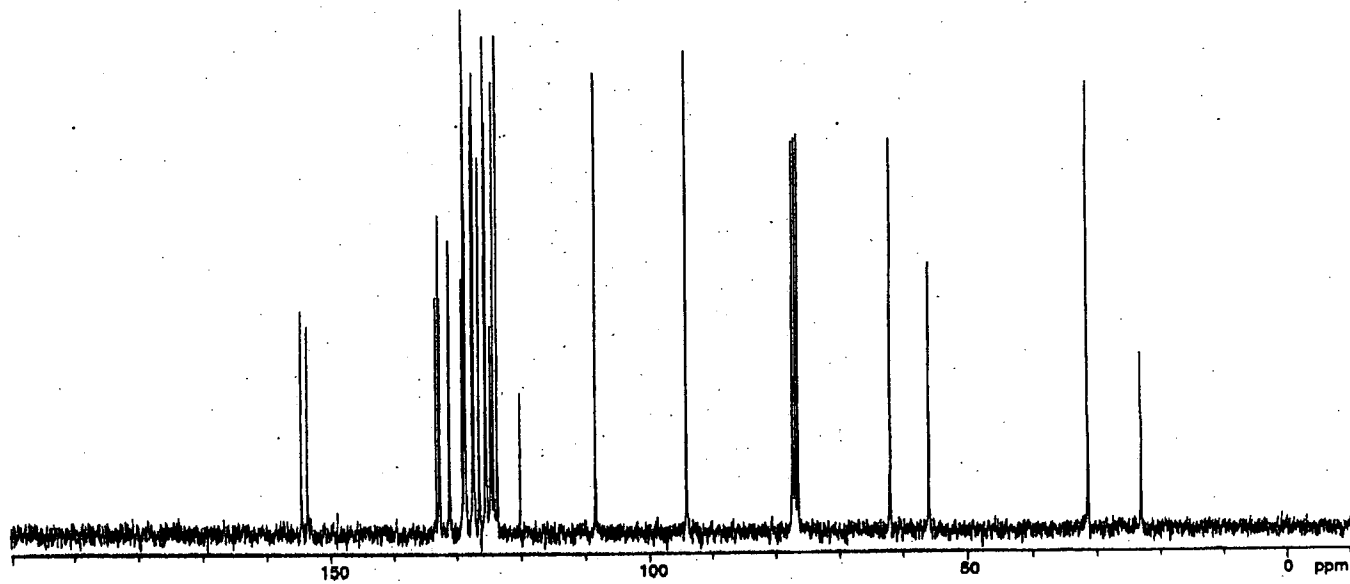
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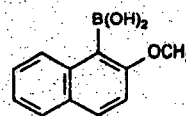
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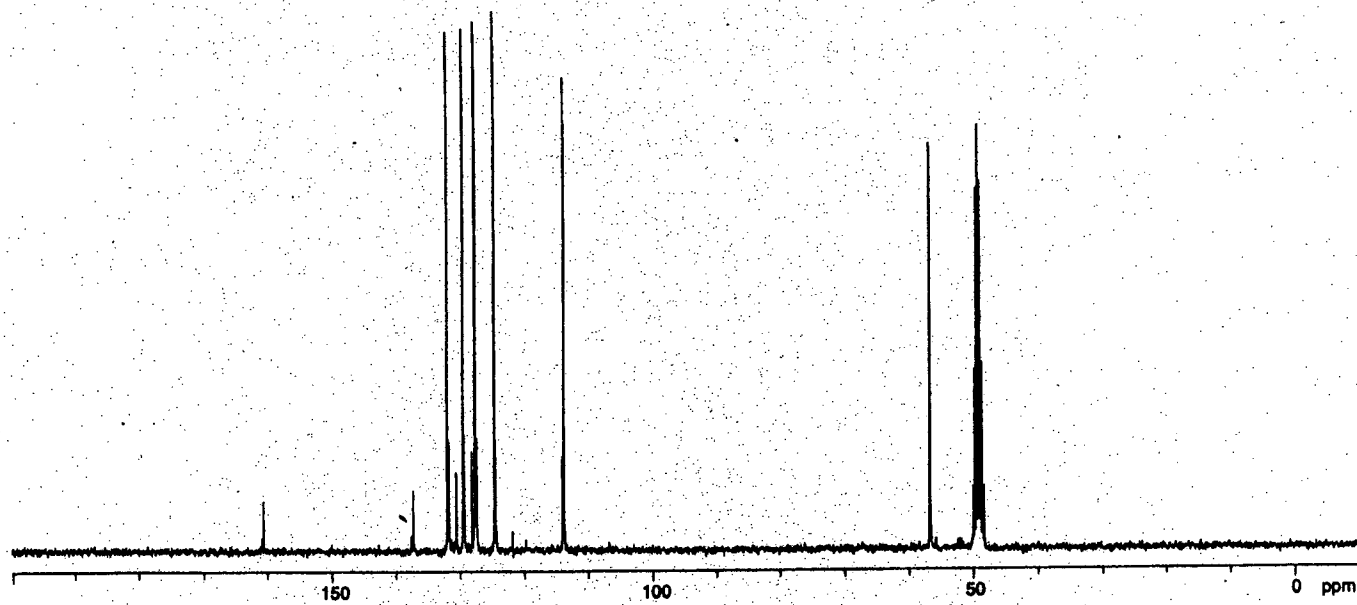
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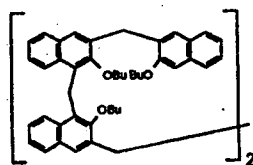
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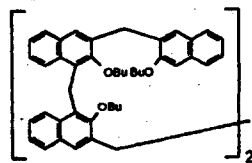
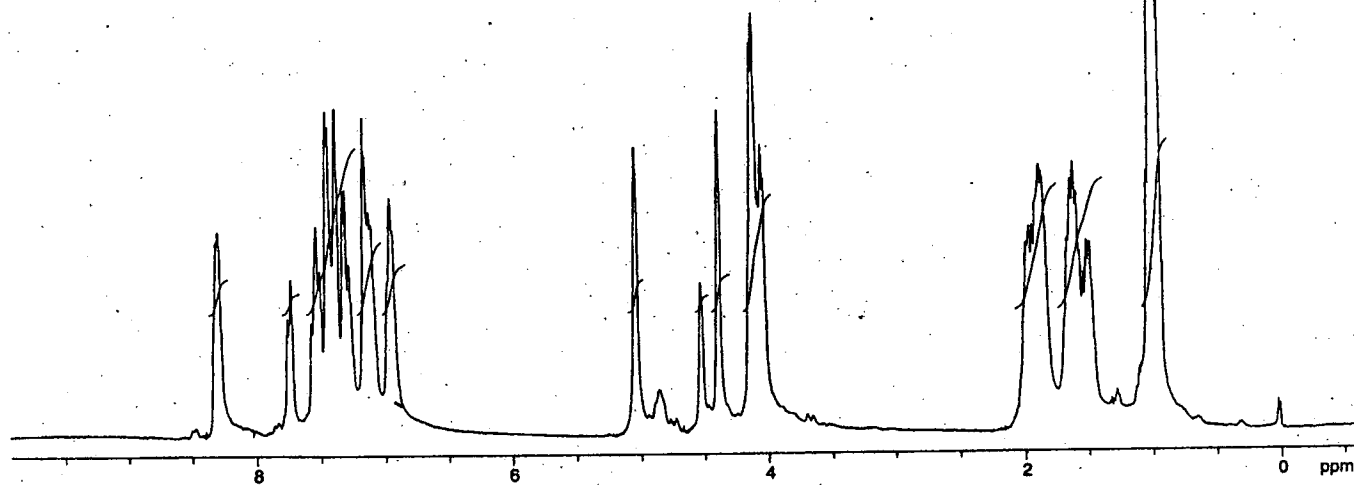
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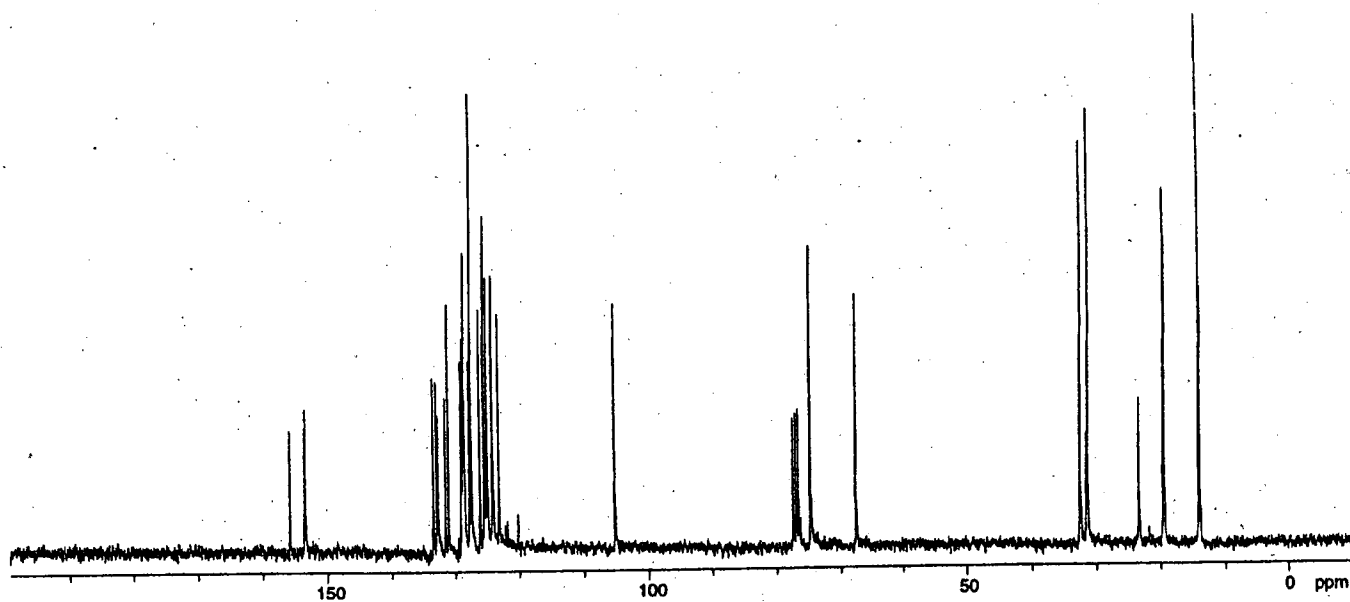
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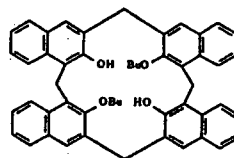
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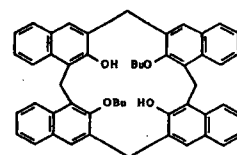
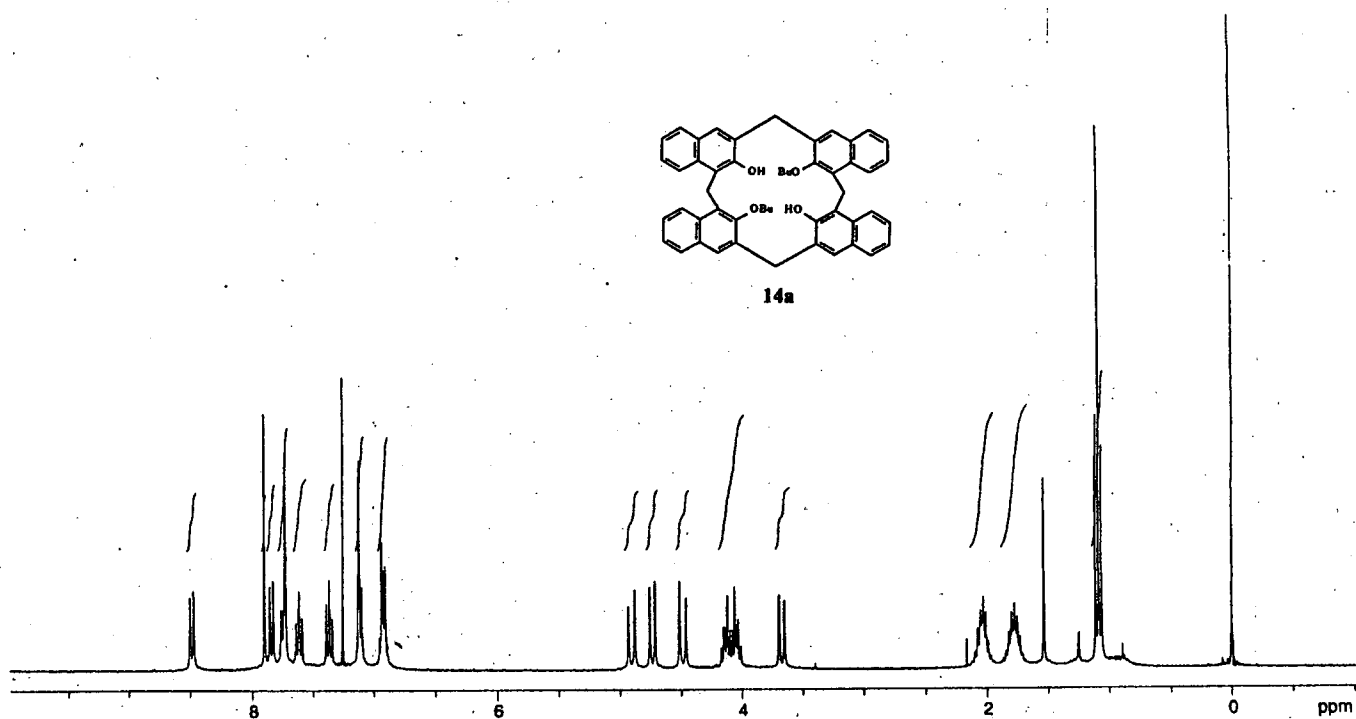
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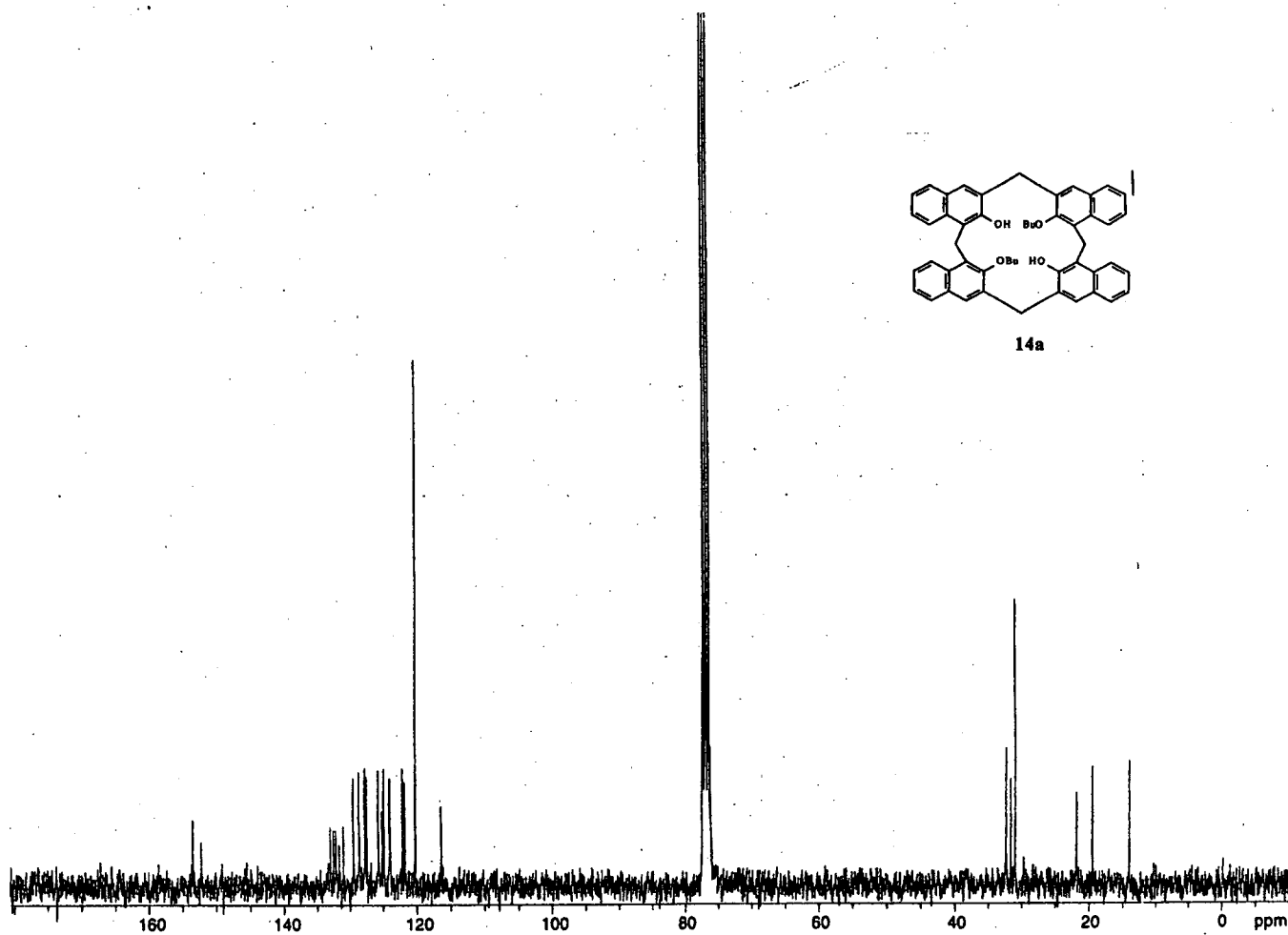
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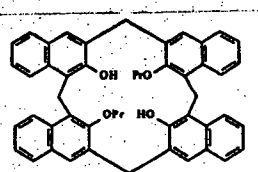
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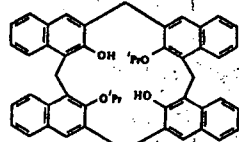
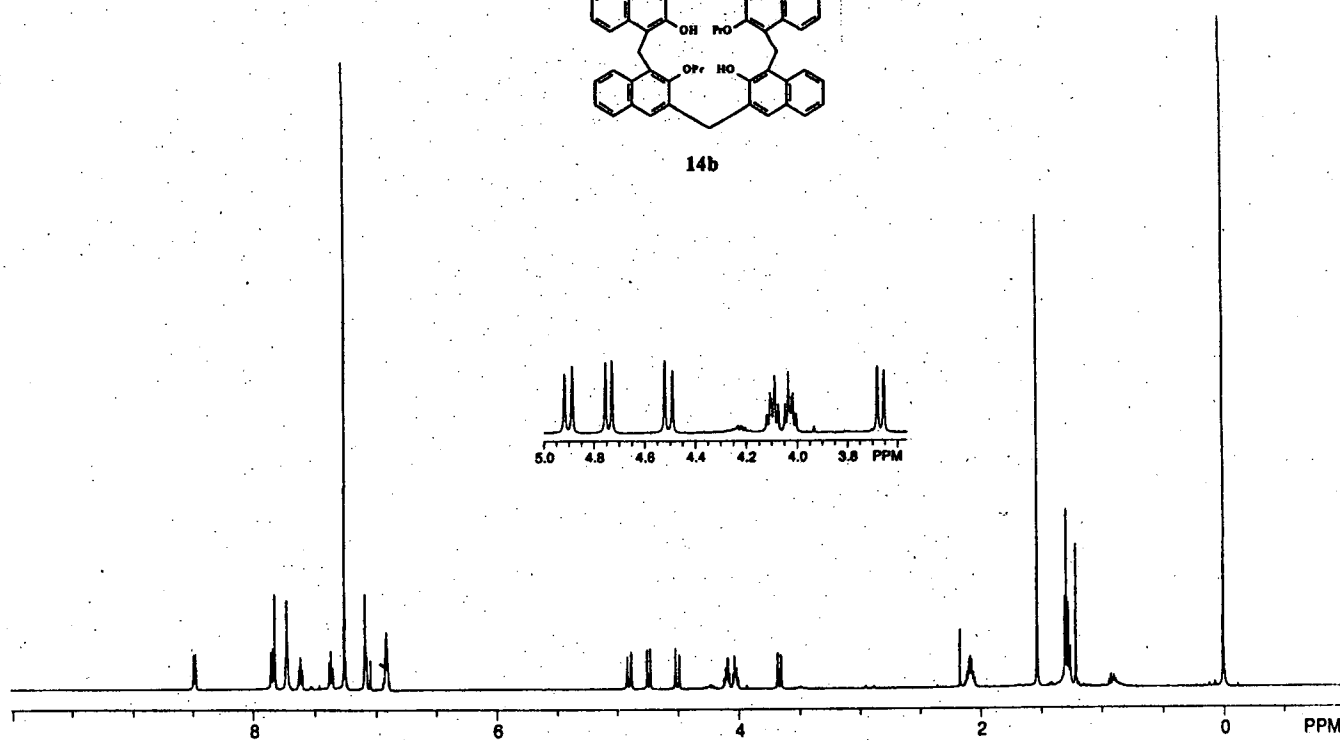
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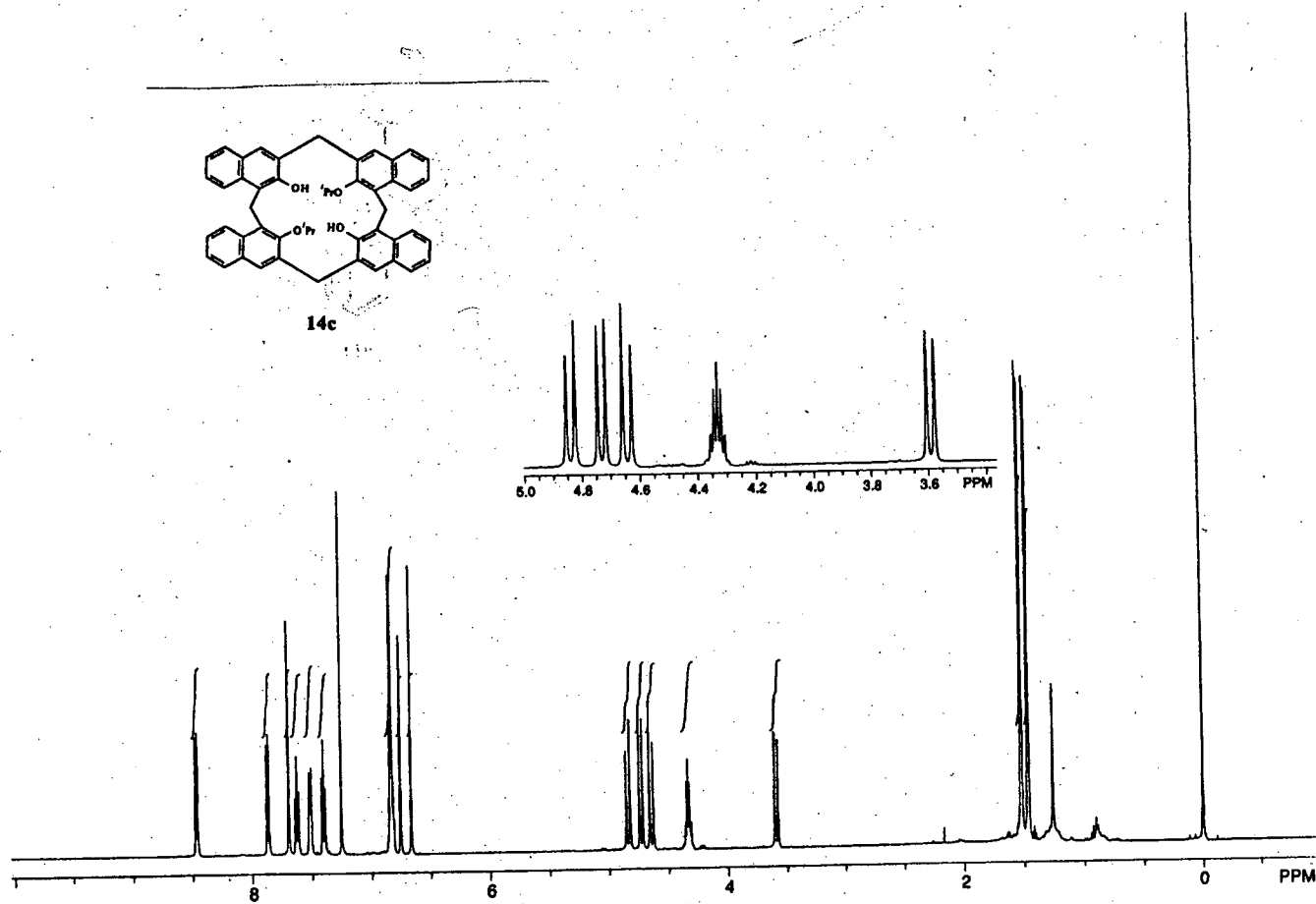
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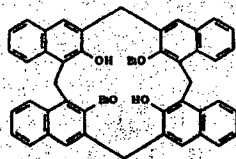
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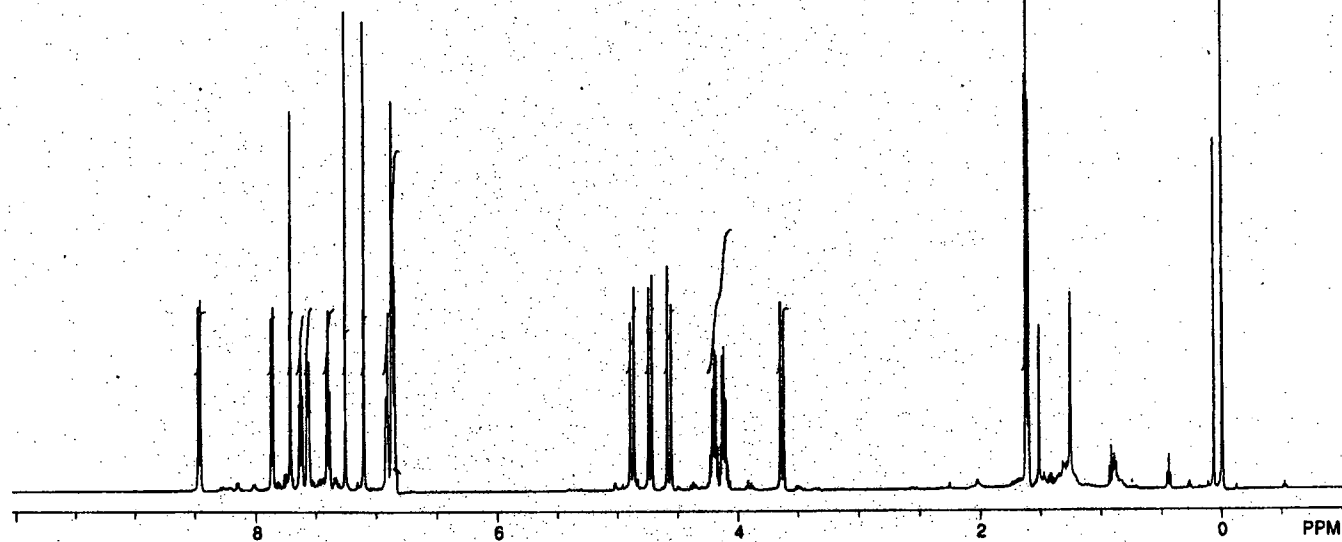
14c



S17



14d



Experimental Section

All the reactions were carried out under argon unless otherwise mentioned. DMF was distilled over MgSO_4 under argon; THF was freshly distilled over sodium and benzophenone under nitrogen prior to use. All other reagents were purchased from commercial suppliers. Unless otherwise indicated, ^1H and ^{13}C NMR spectra were conducted using CDCl_3 as solvent and were recorded at 300 and 75.5 MHz, respectively, using TMS as an internal standard. For a typical modified Suzuki-Miyaura cross-coupling procedure see ref 8.

Bis(1-bromomethyl-2-methoxy-3-naphthyl)methane (4). To a solution of **5** (984 mg, 3.00 mmol) in acetic acid (30 mL) was added aqueous 37% formaldehyde solution (0.50 mL, 15 mmol), and 30% HBr in acetic acid (1.2 mL, 15 mmol). The mixture was stirred at rt for 3 d, during which time a colourless precipitate was formed. The reaction was stirred for another 24 h, then the solid was filtered and washed with water to remove any acid. The residue was purified by flash column chromatography, eluting with 10:90 ethyl acetate-hexane to afford **4** (1.2 g, 77%) as a colourless solid: mp 143-145 °C; ^1H NMR (500 MHz, CDCl_3) δ 3.94 (s, 6H), 4.34 (s, 2H), 5.09 (s, 4H), 7.40 (t, $J = 7.8$ Hz, 2H), 7.45 - 7.58 (m, 4H), 7.68 (d, $J = 8.1$ Hz, 2H), 8.08 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (125 MHz, CDCl_3) δ 25.3, 30.7, 61.6, 123.3, 123.4, 124.8, 125.4, 126.5, 128.3, 131.1, 131.3, 133.3, 155.6; MS (m/z): 515 (M^+ , ^{81}Br , ^{81}Br , 2), 513 (M^+ , ^{79}Br , ^{79}Br , 3), 436 (12), 435 (37), 434 (12), 432 (38), 355 (27), 338 (21), 323 (12), 308 (14); HRMS M^+ 515.9945, calcd for $\text{C}_{25}\text{H}_{22}^{81}\text{Br}^{81}\text{BrO}_2$ 515.9946.

Bis(2-methoxy-3-naphthyl)methane (5). Compound **5** was obtained in 89% yield (7.9 g) as colourless crystals from the reaction of **6** with **7** using the modified Suzuki-Miyaura cross-coupling procedure: mp 145-46 °C; ^1H NMR δ 3.88 (s, 6H), 4.23 (s, 2H), 7.11 (s, 2H), 7.25 (t, $J = 7.4$ Hz, 2H), 7.36 (t, $J = 7.5$ Hz, 2H), 7.41 (s, 2H), 7.60 (d, $J = 8.0$ Hz, 2H), 7.71 (d, $J = 8.1$ Hz,

2H); ^{13}C NMR δ 30.8, 55.3, 104.8, 123.4, 125.6, 126.3, 127.2, 128.8, 128.9, 130.6, 133.5, 156.6; MS (m/z): 328 (M^+ , 47), 281 (7), 265 (3), 234 (100); HRMS M^+ 328.1461, calcd for $\text{C}_{23}\text{H}_{20}\text{O}_2$ 328.1463.

(2-Methoxy-3-naphthyl)-(2-methoxymethoxy-3'-naphthyl)methane (5a). Compound **5a** was obtained in 79% yield (0.72g) as a colourless solid from the reaction between **6** and **7a** using the Suzuki-Miyaura cross-coupling procedure: mp 101-103 °C; ^1H NMR δ 3.37 (s, 3H), 3.91 (s, 3H), 4.26 (s, 2H), 5.28 (s, 2H), 7.12 (s, 1H), 7.28 (t, $J = 7.8$ Hz, 2H), 7.35 - 7.46 (m, 4H), 7.49 (s, 1H), 7.58 - 7.65 (m, 2H), 7.68 - 7.75 (m, 2H); ^{13}C NMR δ 31.0, 55.3, 56.0, 94.1, 104.7, 108.5, 123.4, 123.8, 125.5(2), 125.6, 126.2, 126.6(2), 127.1, 127.2, 128.7, 129.2, 129.3, 130.6, 133.3, 133.4, 153.8, 156.5; MS (m/z): 359 ($\text{M}^+ + 1$, 2), 358 (M^+ , 11), 326 (21), 313 (14), 295 (7), 281(10), 265 (3), 253 (3), 228 (18), 197 (10), 188 (12).

3-Methoxy-2-naphthaleneboronic acid (7). To a solution of 2-methoxynaphthalene (8.3 g, 53 mmol) in anhydrous THF (350 mL) at rt was added dropwise *n*-BuLi (37.1 mL, 63.0 mmol). The reaction mixture was stirred for 2.5 h, then cooled to -78 °C, and trimethylborate (4.2 mL, 76 mmol) was added dropwise over a period of 30 min. The reaction was warmed to rt over 6 h, then aqueous 10% HCl (50 mL) was added. The mixture was stirred at rt for another 30 min. After the solvent was evaporated, the slurry was diluted with CH_2Cl_2 , washed with saturated aqueous NH_4Cl , dried over MgSO_4 and filtered. After the solvent was evaporated, the residue was crystallized from benzene-hexane to afford **7** (9.0 g, 85%) as colourless crystals: mp 129-130 °C; ^1H NMR (acetone- d_6) δ 4.04 (s, 3H), 7.19 (s, 2H), 7.34 - 7.37 (m, 2H), 7.49 (t, $J = 7.4$ Hz, 1H), 7.81 (d, $J = 8.2$ Hz, 1H), 7.85 (d, $J = 7.4$ Hz, 1H), 8.40 (s, 1H); ^{13}C NMR δ 52.3, 102.0, 117.6, 121.8, 124.5, 125.5, 126.1, 133.5, 135.4, 158.8; MS (m/z): 552 (anhydride of boronic acid,

2), 203 (13), 202 (M^+ , 100), 184 (11), 159 (16), 141 (32), 126 (21), 115 (15); HRMS M^+ 202.0827, calcd for $C_{11}H_{11}BO_3$ 202.0801.

3-Methoxymethoxy-2-naphthaleneboronic acid (7a). To a solution of 2-methoxymethoxynaphthalene (1.3 g, 6.7 mmol) in anhydrous THF (50 mL) at -78°C was added *tert*-BuLi (4.8 mL, 8.1 mmol). The yellow solution was stirred for 3 h at -78°C , after which time trimethylborate (1.1 mL, 10 mmol) was added slowly. The mixture was slowly warmed to rt and stirred overnight. After quenching the reaction with saturated aqueous NH_4Cl (20 mL), the organic solvent was removed under reduced pressure. The residue was dissolved in CH_2Cl_2 . The solution was washed with saturated aqueous NH_4Cl (2x25 mL), dried over MgSO_4 and filtered, and the solvent was evaporated. The product was crystallized from ethyl acetate-hexane to afford **7a** (1.2 g, 75%) as yellow crystals: ^1H NMR δ 3.56 (s, 3H), 5.42 (s, 2H), 6.35 (s, 2H, OH, D_2O exchangeable), 7.38 (t, $J = 7.5$ Hz, 1H), 7.43 (s, 1H), 7.49 (t, $J = 7.5$ Hz, 1H), 7.74 (d, $J = 8.2$ Hz, 1H), 7.85 (d, $J = 8.1$ Hz, 1H), 8.44 (s, 1H, H-1); ^{13}C NMR δ 56.5, 94.8, 108.4, 120.3, 124.4, 126.8, 127.7, 128.6, 129.2, 136.0, 138.8, 138.9, 158.7; MS (m/z): 232 (M^+ , 3), 214 (3), 199 (2), 184 (16), 170 (9), 156 (8).

Bis(2-hydroxy-3-naphthyl)methane (8). To a solution of **5** (0.98 g, 3.0 mmol) in anhydrous CH_2Cl_2 (15.0 mL) at 0°C , was added dropwise a solution of BBr_3 (1M in CH_2Cl_2 , 10.5 mL, 10.5 mmol). The brown solution was stirred at rt for 16 h, then aqueous 10% HCl (5.0 mL) was added, and the mixture was stirred at rt for another 30 min. The organic layer was washed with saturated aqueous NH_4Cl (3 x 15 mL), dried over MgSO_4 , filtered and the solvent was evaporated under reduce pressure. The brown solid was purified by flash column chromatography eluting with 30:70 ethyl acetate-hexane to afford **8** (0.86 g, 95%) as a brown solid: mp $185\text{-}87^\circ\text{C}$; ^1H NMR (CD_3OD) δ 4.23 (s, 2H), 4.90 (s, 2H), 7.12 - 7.19 (m, 4H), 7.28

(dd, $J = 1.9, 6.9$ Hz, 2H), 7.45 (s, 2H), 7.55 (d, $J = 8.1$ Hz, 2H), 7.59 (d, $J = 8.1$ Hz, 2H); ^{13}C NMR (CD_3OD) δ 30.3, 108.2, 120.3, 122.3, 124.9, 125.2, 126.8, 128.6, 129.8, 133.8, 153.9; MS (m/z): 300 (M^+ , 100), 281 (8), 252 (10), 157 (95); HRMS M^+ 300.1160, calcd for $\text{C}_{21}\text{H}_{16}\text{O}_2$ 300.1149.

Bis(1-bromo-2-hydroxy-3-naphthyl)methane (9). To a solution of **8** (0.90 g, 3.0 mmol) in acetic acid (20.0 mL), a solution of Br_2 (0.31 mL, 6.0 mmol) in acetic acid (5.0 mL) was added dropwise over period of 5 min. The mixture was stirred at rt for 4 h, after which time, a second equivalent of Br_2 was added and the reaction was stirred a further 16 h. The mixture was poured into water (100 mL), and the organic layer was extracted with ethyl acetate (3 x 50 mL). The combined organic layers were washed with aqueous 10% NaHSO_3 (20 mL), then brine (20 mL), dried over MgSO_4 , and finally the solvent was evaporated. The residue was washed several times with hexane to afford **9** (1.30 g, 95%) as a yellow solid was used in the next step as the compound turns brown: mp 160-163 °C; ^1H NMR δ 4.40 (s, 2H), 6.18 (s, 2H, OH, D_2O exchangeable), 7.35 (t, $J = 8.1$ Hz, 2H), 7.52 (t, $J = 8.3$ Hz, 2H), 7.58 (s, 2H), 7.68 (d, $J = 8.1$ Hz, 2H), 7.99 (d, $J = 8.3$ Hz, 2H).

Bis(1-bromo-2-methoxymethoxy-3-naphthyl)methane (9a). To a solution of **9** (1.30 g, 2.83 mmol) in THF (40 mL) at 0 °C, NaH (327 mg, 8.52 mmol) was added. The mixture was stirred for 30 min, chloromethyl methyl ether (0.65 mL, 8.52 mmol) was added dropwise, and the mixture was stirred at rt for 12 h. The reaction was quenched with water, and the THF was removed under reduced pressure. The residue was diluted with ethyl acetate, washed with water, dried over MgSO_4 and the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography eluting with 20:80 ethyl acetate-hexane to afford as a viscous oil **9a** in 95% yield: ^1H NMR δ 3.65 (s, 6H), 4.62 (s, 2H), 5.21 (s, 4H), 7.39 - 7.47 (m,

4H), 7.55 (dd, $J = 2.1, 8.5$ Hz, 2H), 7.63 (d, $J = 8.4$ Hz, 2H), 8.24 (d, $J = 8.8$ Hz, 2H); MS (m/z): 546 (M^+ , 1), 469 (7), 438 (2), 390 (3). This product was subjected to the usual metalation conditions using *n*-BuLi in THF and trimethylborate. The product **3a** obtained from **9a** was used without purification, in the Suzuki-Miyaura cross-coupling reaction conditions with **4**.

Bis{(2-methoxy-3-[3'-naphthyl-(2'-methoxy)methyl]naphthyl)methane (11). Compound **11** was obtained in 58% yield (193 mg) as colourless crystals from the reaction between **10**^{6a} and **7** using the modified Suzuki-Miyaura cross-coupling procedure: mp 241-244 °C; ¹H NMR (CD₂Cl₂) δ 3.98 (s, 6H), 3.99 (s, 6H), 4.39 (s, 4H), 5.03 (s, 2H), 6.81 (t, $J = 7.4$ Hz, 2H), 7.17 (t, $J = 7.5$ Hz, 4H), 7.24 (s, 2H), 7.33 (t, $J = 7.4$, 2H), 7.38 - 7.51 (m, 6H), 7.58 (d, $J = 8.0$ Hz, 2H), 7.80 (s, $J = 8.2$ Hz, 2H), 8.17 (d, $J = 8.5$ Hz, 2H); ¹³C NMR (CD₂Cl₂) δ 20.3, 28.3, 52.7, 59.4, 102.1, 117.6, 120.6, 121.6, 121.9, 122.3, 122.6, 122.9, 123.5, 124.6, 125.0, 125.9, 126.1, 128.5, 128.7, 129.8, 130.5, 130.8, 151.9, 153.6; MS (m/z): 668 (M^+ , 32), 621 (3), 512 (4), 497 (3), 465 (3), 340 (21), 327 (33), 311 (29); HRMS M^+ 668.2936 calcd for C₄₇H₄₀O₄ 668.2924.

Bis{(2-methoxy)-3-[3'-naphthyl-(2'-methoxymethoxy)methyl]naphthyl)methane (11a). Compound **11a** was obtained in 43% yield (0.54 g) as a yellow fluffy solid from the reaction between **10** and **7a** using the modified Suzuki-Miyaura cross-coupling procedure: mp 78-80 °C; ¹H NMR δ 3.35 (s, 6H), 3.90 (s, 6H), 4.40 (s, 4H), 5.01 (s, 2H), 5.3 (s, 4H), 7.00 (t, $J = 7.7$ Hz, 2H), 7.17 (t, $J = 7.4$ Hz, 2H), 7.29 (t, $J = 7.4$ Hz, 2H), 7.37 - 7.44 (m, 7H), 7.57 (t, $J = 9.4$ Hz, 5H), 7.75 (d, $J = 7.8$ Hz, 2H), 8.23 (d, $J = 8.4$ Hz, 2H); ¹³C NMR δ 23.1, 31.4, 56.0, 62.1, 94.2, 108.5, 120.3, 123.8, 124.4, 124.8, 124.9, 125.5, 125.7, 126.6, 127.4, 127.7, 128.7, 129.2, 131.2, 131.3, 132.7, 132.9, 133.4, 153.6, 154.6; MS (m/z): 698 (3), 665 (10), 652 (6), 633 (4), 591 (2), 575 (5), 495 (2), 463 (2), 371 (22), 339 (30), 325 (25).

Bis{(2-methoxy)-3-[1-naphthyl-(2'-methoxy)methyl]naphthyl}methane (12). Tetranaphthyl compound **12** was obtained in 23% yield (76 mg) as a colourless solid from the reaction of **4** with **13** using the modified Suzuki-Miyaura cross-coupling procedure: mp 101-103 °C; ¹H NMR (500 MHz, CDCl₃) δ 3.83 (s, 6H), 3.96 (s, 6H), 4.49 (s, 2H), 4.99 (s, 4H), 7.06 (t, *J* = 7.2 Hz, 2H), 7.14 (t, *J* = 7.3 Hz, 2H), 7.21 (m, 4H), 7.29 (d, *J* = 9.3 Hz, 2H), 7.40 (s, 2H), 7.53 (m, 2H), 7.65 (d, *J* = 8.1 Hz, 2H), 7.69 (d, *J* = 9.0 Hz, 2H), 8.12 (d, *J* = 2.5 Hz, 2H), 8.14 (d, *J* = 8.3 Hz, 2H); ¹³C NMR (125 MHz, CD₂Cl₂) δ 23.2, 32.2, 57.4, 62.5, 114.3, 123.8, 123.9, 124.9, 125.0, 125.4, 125.7, 127.0, 128.6, 128.8, 128.9, 129.2, 130.1, 130.2, 131.9, 133.5, 134.4, 154.8, 155.5; HRMS M⁺ 668.2948, calcd for C₄₇H₄₀O₄ 668.2924.

2-Methoxy-1-naphthaleneboronic acid (13). To a solution of 1-bromo-2-methoxynaphthalene (10.0 g, 42.2 mmol) in anhydrous THF (250 mL) at -78 °C was added dropwise, *n*-BuLi (1.6 M in hexane, 29.0 mL, 46.4 mmol). The reaction solution was stirred for 45 min at -78 °C, then trimethyl borate (5.80 mL, 50.6 mmol) was added dropwise and the mixture allowed to warm to rt over a 6 h period. Aqueous 10% HCl (40 mL) was added and the mixture stirred at rt for another 30 min. After the solvent was evaporated, the slurry was diluted with CH₂Cl₂ (100 mL), washed with saturated aqueous NH₄Cl, brine and dried over MgSO₄ and filtered. The solvent was evaporated and the crude product was crystallized from ethyl acetate-hexane to afford **13** (5.56 g, 65%) as colourless crystals: mp 109-112 °C; ¹H NMR (CD₃OD) δ 3.89 (s, 3H), 4.90 (s, 2H), 7.25 - 7.34 (m, 2H), 7.42 (t, *J* = 8.2 Hz, 1H), 7.51 (d, *J* = 8.9 Hz, 1H), 7.78 (d, *J* = 8.3 Hz, 1H), 7.87 (d, *J* = 8.6 Hz, 1H); ¹³C NMR (CD₃OD) δ 56.7, 113.8, 124.6, 127.6, 127.7, 128.2, 129.3, 129.5, 132.0, 137.3, 160.6; MS (*m/z*): 202 (M⁺, 100), 186 (5), 172 (4), 158 (63), 141 (26), 126 (25), 115 (62); HRMS M⁺ 202.0796, calcd for C₁₁H₁₁BO₃ 202.0801.

34,36-Dibutyloxy-6H,14H,22H,30H-5,31:7,13:15,21:23,29-tetramethenotetrabenzo [a,f,m,r]cyclotetracosene-33,35-diol (14a). To a solution of the crude reaction product **2** (725 mg) in anhydrous DMF (10 mL) was added NaH (60 % suspension in oil, 223 mg, 5.81 mmol). The brown solution was stirred at rt for 30 min, then 1-bromobutane (0.31 mL, 7.0 mmol) was added. The mixture was stirred at 80 °C for 16 h, cooled to rt and diluted with ethyl acetate. The organic solution was washed several times with water, then with aqueous 10% HCl, brine, dried over MgSO₄, filtered and the solvent was evaporated to afford brown crude product, which was purified by preparative TLC using 10:90 ethyl acetate-hexane. TLC analysis of this material showed the presence of two products which were separated by preparative TLC using benzene-petroleum ether 40:60 to afford **14** (300 mg) as a yellow solid: mp >300 °C; ¹H NMR δ 0.93 - 1.02 (m, 18H), 1.43 - 1.69 (m, 12H), 1.81 - 2.01 (m, 12H), 4.01 - 4.16 (m, 12H), 4.37 (s, 4H), 4.51 (s, 2H), 5.02 (s, 4H), 6.94 (m, 4H), 7.10 (m, 4H), 7.16 (s, 2H), 7.27 - 7.44 (m, 12H), 7.53 (d, *J* = 8.7 Hz, 4H), 7.74 (d, *J* = 8.1 Hz, 2H), 8.28 (m, 4H); ¹³C NMR δ 13.9, 14.1, 19.4, 19.5, 19.6, 23.3, 31.3, 31.6, 32.5, 32.6, 67.6, 74.7, 105.2, 120.3, 124.1, 124.2, 124.9, 125.0, 125.4, 125.5, 126.2, 126.3, 127.5, 127.8, 128.5, 128.6, 128.8, 128.9, 129.0, 129.1, 131.1, 131.6, 132.7, 132.8, 133.0, 133.5, 133.6, 153.4, 153.5, 155.8; and **14a** (20 mg, 3%): mp >320 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.08 (t, *J* = 7.3 Hz, 6H), 1.71-1.83 (m, 4H), 2.01 (m, 4H), 3.67 (d, *J* = 12.9 Hz, 2H), 4.04 (m, 2H), 4.14 (m, 2H), 4.50 (d, *J* = 15.3 Hz, 2H), 4.73 (d, *J* = 12.9 Hz, 2H), 4.90 (d, *J* = 15.2 Hz, 2H), 6.90 (m, 4H), 7.09 - 7.12 (m, 4H), 7.36 (dd, *J* = 1.2, 7.8 Hz, 2H), 7.61 (dd, *J* = 1.2, 8.4 Hz, 2H), 7.73-7.77 (m, 4H), 7.84 (dd, *J* = 1.2, 8.0 Hz, 2H), 7.90 (s, 2H), 8.48 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 14.1, 19.6, 21.9, 30.9, 31.6, 32.2, 116.5, 121.9, 122.2, 124.0, 124.1,

124.9, 125.2, 125.8, 127.5, 127.9, 128.6, 128.7, 129.5, 131.1, 131.7, 132.2, 132.6, 133.1, 152.2, 153.4; (+) FAB MS (m/z): 736 (M^+ , 2, 100), 677 (2), 661 (22), 605 (65), 587 (19), 325 (70), 252 (95).

34,36-Dipropoxy-6H,14H,22H,30H-5,31:7,13:15,21:23,29-tetramethenotetrabenzo [a,f,m,r]cyclotetracosene-33,35-diol (14b). To a solution of the crude reaction product **2** (485 mg) in anhydrous DMF (15 mL) at rt, NaH (60 % suspension in oil, 0.280 mg, 7.49 mmol) was added. The resulting slurry was stirred at rt for 30 min, then 1-iodopropane (0.40 mL, 4.49 mmol) was added. The reaction mixture was stirred for 3 d. After the DMF was removed under reduced pressure, the brown residue was diluted with ethyl acetate, washed with saturated aqueous NH_4Cl , then brine, dried over $MgSO_4$, filtered and finally the solvent was evaporated. The crude product was purified by preparative TLC eluting with 30:70 CH_2Cl_2 -petroleum ether to afford brown solid. This product was further purified by preparative TLC eluting with 90:10 benzene-hexane to afford **14b** (20 mg, 4%) as a brown solid: mp >300 °C (decomposition); 1H NMR (500 MHz, $CDCl_3$) δ 1.29 (t, $J = 7.5$ Hz, 6H), 2.09 (m, 4H), 3.67 (d, $J = 13.0$ Hz, 2H), 4.03 (m, 2H), 4.10 (m, 2H), 4.50 (d, $J = 15.5$ Hz, 2H), 4.73 (d, $J = 13.0$ Hz, 2H), 4.90 (d, $J = 15.2$ Hz, 2H), 6.90 (m, 4H), 7.09 (d, $J = 9.0$ Hz, 4H), 7.37 (t, $J = 8.0$ Hz, 2H), 7.62 (t, $J = 8.0$ Hz, 2H), 7.73 (t, $J = 9.0$ Hz, 4H), 7.82 (s, 2H), 7.84 (d, $J = 8.0$ Hz, 2H), 8.48 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 10.8, 21.9, 23.5, 30.9, 77.9, 116.7, 121.9, 122.2, 124.0, 124.1, 124.9, 125.3, 125.8, 127.5, 127.8, 128.6, 127.7, 129.5, 131.1, 131.7, 132.3, 132.6, 133.2, 152.2, 153.4; FAB MS (m/z): 708 (M^+ , 100), 664 (41), 605 (37), 312 (73).

34,36-Diisopropoxy-6H,14H,22H,30H-5,31:7,13:15,21:23,29-tetramethenotetrabenzo [a,f,m,r]cyclotetracosene-33,35-diol (14c). To a solution of

the crude reaction product **2** (310 mg) in anhydrous DMF (10 mL) at rt, was added NaH (60% suspension in oil, 46 mg, 1.2 mmol). The resulting slurry was stirred at rt for 30 min, then 2-iodopropane (0.15 mL, 1.5 mmol) was added. The mixture was stirred for 48 h. After the DMF was removed under reduced pressure, the residue was diluted with ethyl acetate, washed with aqueous 10% HCl, brine, dried over MgSO₄, filtered and the solvent was evaporated. The crude product was purified by preparative TLC eluting with 10:90 ethyl acetate-hexane to afford a brown solid (110 mg). The ¹H NMR spectrum showed the expected product signals and other signals which could not be identified. This solid was again purified by preparative TLC eluting with 10:90 dioxane-pentane to afford **14c** (20 mg, 6%): mp >320 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.47 (d, *J* = 6.1 Hz, 6H), 1.52 (d, *J* = 6.1 Hz, 6H), 3.60 (d, *J* = 13.6 Hz, 2H), 4.29 - 4.37 (sept, *J* = 6.0 Hz, 2H), 4.63 (d, *J* = 15.4 Hz, 2H), 4.73 (d, *J* = 13.5 Hz, 2H), 4.84 (d, *J* = 15.4 Hz, 2H), 6.74 (s, 2H), 6.79 (s, 2H), 6.84 (m, 6H), 7.40 (m, 2H), 7.54 (m, 2H), 7.64 (m, 2H), 7.69 (s, 2H), 7.86 (d, *J* = 8.1 Hz, 2H), 8.47 (d, *J* = 8.7 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 22.1, 22.3, 22.7, 32.2, 78.6, 118.1, 121.8, 122.4, 123.9, 124.7, 125.6, 125.9, 127.4, 127.6, 128.6, 128.7, 130.8, 131.1, 132.4, 132.8, 134.2, 151.3, 152.7; FAB MS (*m/z*): 708 (M⁺, 100), 664 (41), 605 (37), 312 (73).

34,36-Diethyloxy-6H,14H,22H,30H-5,31:7,13:15,21:23,29-tetramethenotetra benzo [a,f,m,r]cyclotetracosene-33,35-diol (14d). To a suspension of the crude reaction product **2** (570 mg) in dry CH₃CN (20 mL), powdered anhydrous K₂CO₃ (2.5 g, 9.1 mmol) and iodoethane (0.73 mL, 9.1 mmol) was added and the mixture heated at reflux for 16 h. The reaction mixture was cooled to rt, solid material was filtered, and the solvent was evaporated under reduce pressure. The residue was diluted with CHCl₃ (25

mL), washed with aqueous 10% HCl (15 mL), brine, dried over MgSO₄, and filtered. After the solvent was evaporated, the residue was purified by preparative TLC eluting with 30:70 benzene-hexane to afford **14d** (52 mg, 8%) as a brown solid: mp >300 °C; ¹H NMR (500 MHz, CDCl₃) δ 1.61 (t, 7.0 Hz, 6H), 3.63 (d, *J* = 13.2, Hz, 2H), 4.07 - 4.15 (m, 2H), 4.17-4.22 (m, 2H), 4.57 (d, *J* = 15.4 Hz, 2H), 4.73 (d, *J* = 13.2 Hz, 2H), 4.88 (d, *J* = 15.3 Hz, 2H), 6.91 (m, 2H), 7.10 (s, 2H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.56 (d, *J* = 9.4 Hz, 2H), 7.63 (t, *J* = 7.6 Hz, 2H), 7.71 (s, 2H), 7.86 (d, *J* = 8.1 Hz, 2H), 8.46 (d, *J* = 8.6 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃) δ 15.5, 29.7, 31.6, 72.1, 117.4, 120.3, 121.8, 122.4, 123.9, 124.9, 125.1, 125.9, 127.4, 127.6, 128.7, 129.1, 130.9, 131.3, 132.3, 132.7, 133.4, 152.4, 153.0; FAB MS (*m/z*): 680 (M⁺, 24), 679 (48), 633 (60), 605 (100), 587 (60).

X-ray crystal data for 14d: (toluene) C₄₈H₄₀O₄·2C₇H₈; monoclinic, space group C2/c (#15), *Z* value = 4, *a* = 13.840(1) Å, *b* = 17.001(1) Å, *c* = 20.287(2) Å, β = 103.090(2)°, *V* = 4649.4(6) Å³, *D*_{calc} = 1.236 g/cm³, *F*₀₀₀ = 1840.00, μ(MoKα) = 0.75 cm⁻¹, crystal dimension = 0.45 x 0.25 x 0.14 mm. Intensity data were measured at 193 K on a Bruker P4/CCD diffractometer with graphite monochromated Mo-Kα (*l* = 0.71073 Å) radiation to 2θ_{max} (deg) = -80±1°C; 11374 reflections which were collected, 4776 were unique (*R*_{int} = 0.044) with *I* > 2.00σ(*I*); Final *R*₁ and *wR*₂ values were 0.055 and 0.154, respectively, *gof* = 1.03.