

Supporting Information

Me₃SiI-Promoted Reaction of Salicylic Aldehydes with Ketones: A Facile Way to Construct Benzopyranic [2,3-*b*]Ketal and Spiroketal

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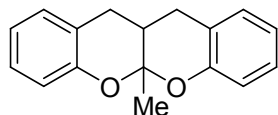
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General remarks. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker AM-300 or AM-400 spectrophotometers. Infrared spectra were recorded on a Perkin-Elmer PE-983 spectrometer with absorption in cm^{-1} . Flash column chromatography was performed using 300-400 mesh silica gel. For thin-layer chromatography (TLC), silica gel plates (Huanghai GF₂₅₄) were used. Mass spectra were recorded by EI and ESI, and HRMS were measured on a HP-5989 instrument.

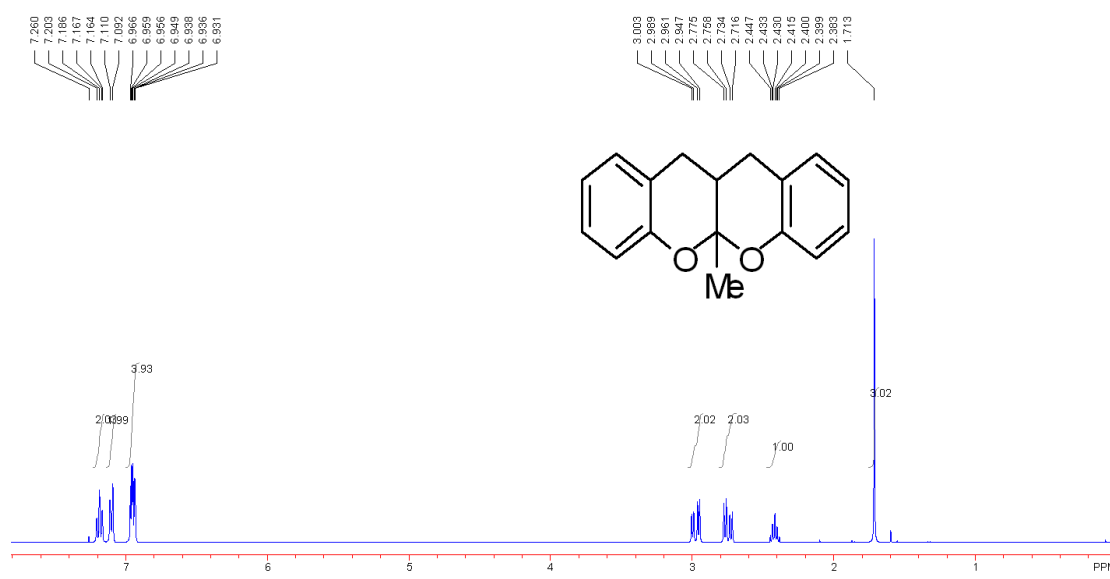
General procedure for the Me₃SiI-promoted reaction of salicylic aldehyde with ketone.

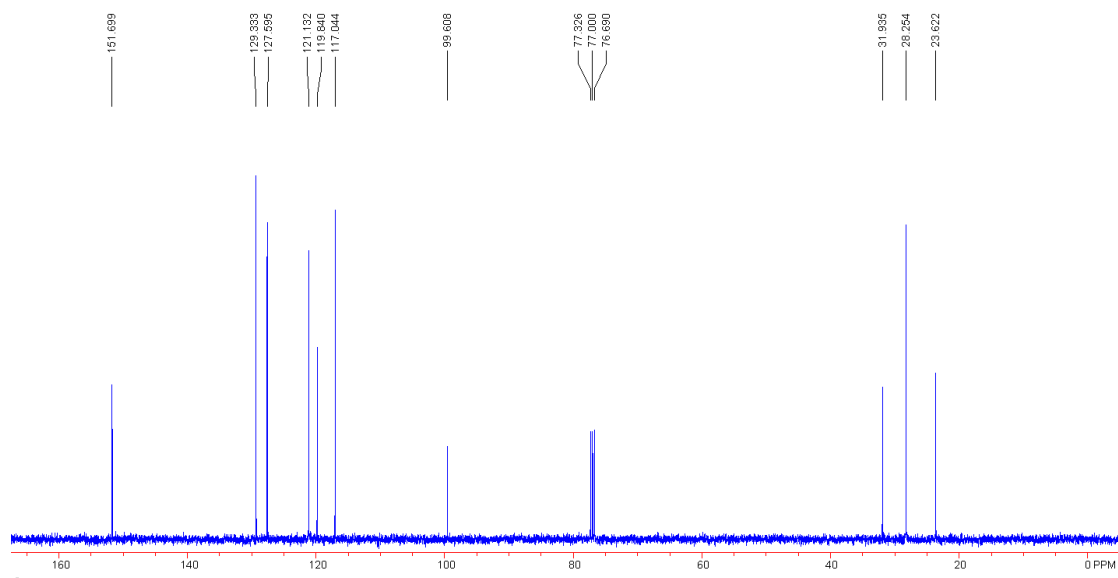
To a stirred mixture of Me₃SiCl (10.0 mmol), NaI (10.0 mmol), ketone (3.0 mmol) and CH₃CN (5 mL) was added salicylic aldehyde **2** (1.0 mmol) at ice-bath temperature. The reaction mixture was stirred at room temperature for 24 h. After addition of aqueous Na₂S₂O₃ to the reaction mixture, the organic layer was extracted with dichloromethane, washed with brine, dried over MgSO₄, and concentrated under reduced pressure. The residue was purified by chromatography on silica gel to obtain the corresponding benzopyranic ketal.

5a-Methyl-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3aa**.

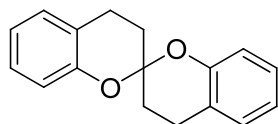


White solid; Mp. 165.8-166.0 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.71 (s, 3H), 2.38-2.45 (m, 1H), 2.75 (dd, *J* = 6.8, 16.4 Hz, 2H), 2.98 (dd, *J* = 5.6, 16.8 Hz, 2H), 6.93-6.97 (m, 4H), 7.10 (d, *J* = 7.2 Hz, 2H), 7.19 (d, *J* = 6.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 23.6, 28.3, 31.9, 99.6, 117.0, 119.8, 121.1, 127.6, 129.3, 151.7; MS (ESI) *m/z* (%): 253.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for C₁₇H₁₇O₂: 253.1229; Found: 253.1220.



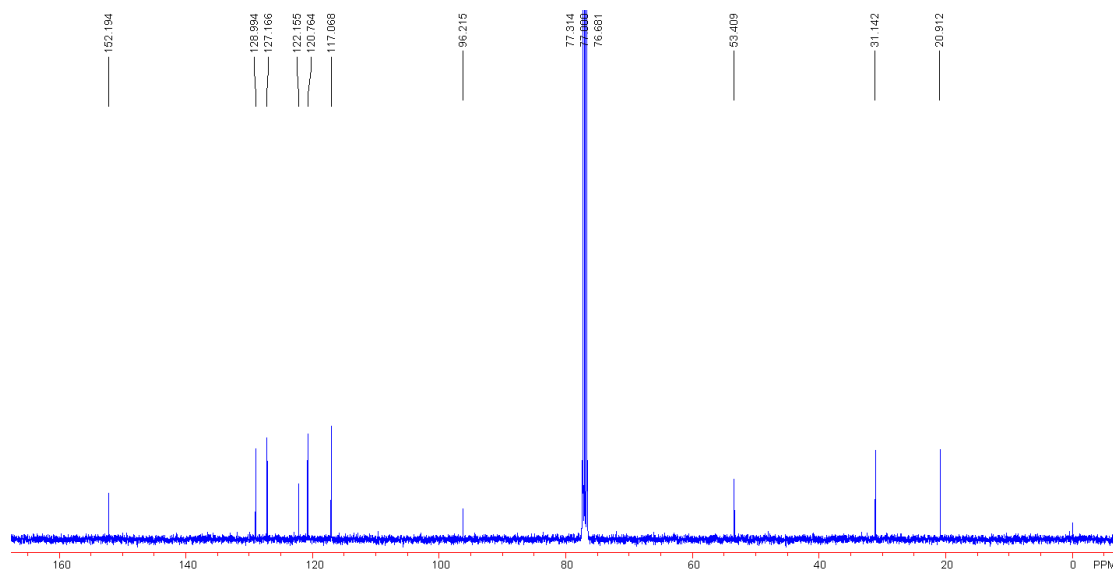


2,2'-Spirobi[chroman] **4aa**.^[1]

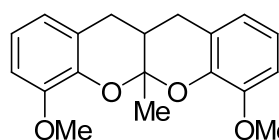


White solid; ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.93-2.01 (m, 2H), 2.20-2.26 (m, 2H), 2.72-2.78 (m, 2H), 3.24-3.33 (m, 2H), 6.73 (dd, *J* = 1.2, 8.4 Hz, 2H), 6.88 (dt, *J* = 1.2, 7.2 Hz, 2H), 7.06 (t, *J* = 7.6 Hz, 2H), 7.12 (d, *J* = 7.6 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 20.9, 31.1, 53.4, 96.2, 117.1, 120.8, 122.2, 127.2, 129.0, 152.2.

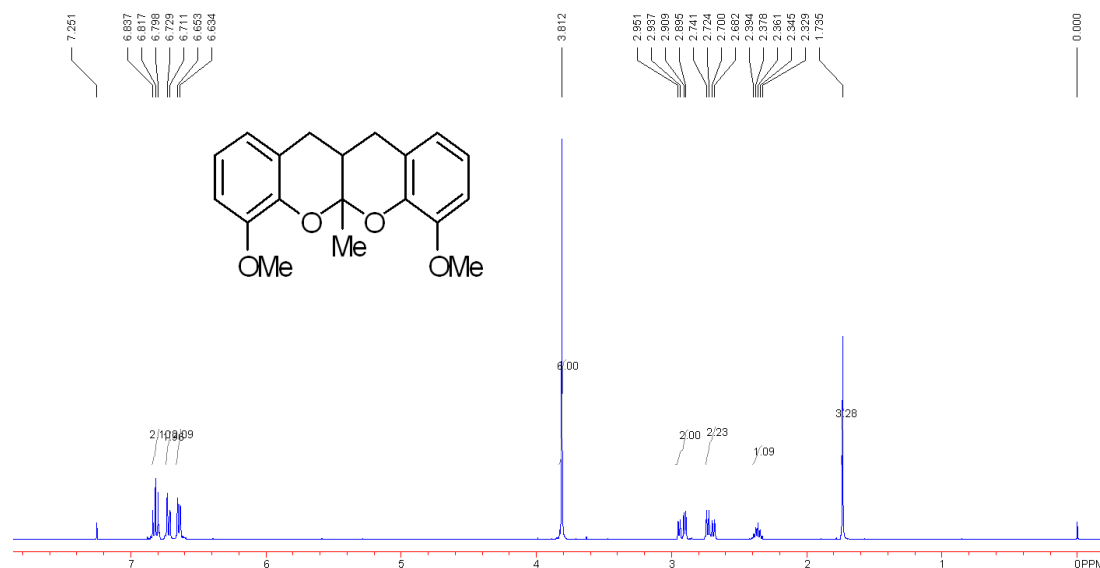


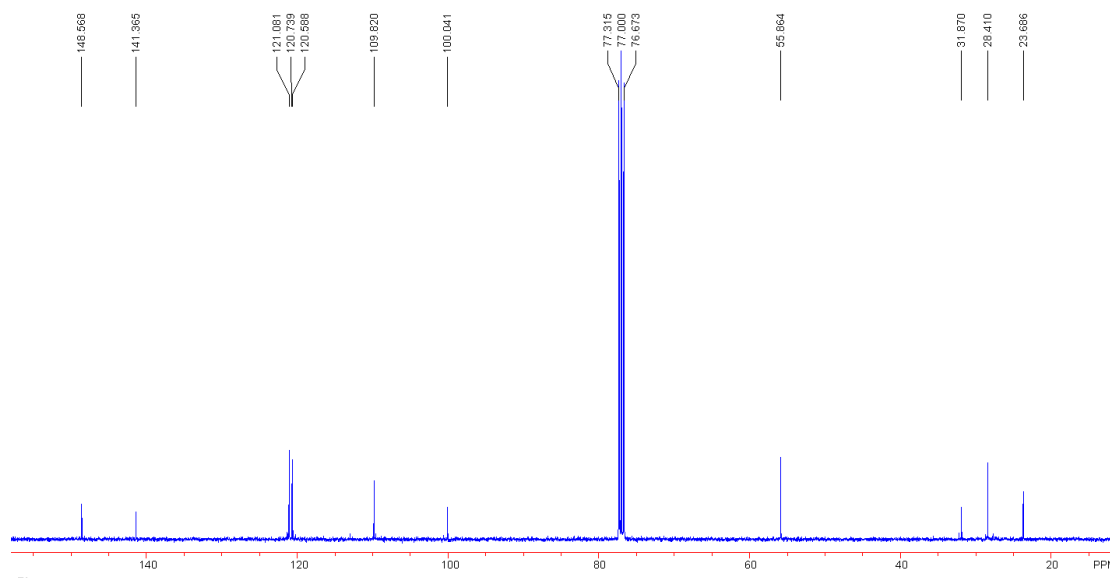


4,7-Dimethoxy-5a-methyl-5a,11,11a,12-tetrahydrochromeno[2,3-*b*]chromene **3ab**.

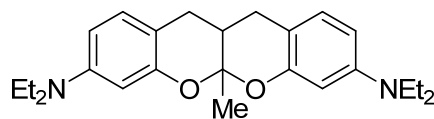


White solid; Mp. 171.5-171.8 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.74 (s, 3H), 2.33-2.39 (m, 1H), 2.71 (dd, *J* = 6.8, 16.4 Hz, 2H), 2.93 (dd, *J* = 5.6, 16.8 Hz, 2H), 3.81 (s, 6H), 6.64 (d, *J* = 7.6 Hz, 2H), 6.72 (d, *J* = 7.2 Hz, 2H), 6.82 (t, *J* = 8.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 23.7, 28.4, 31.9, 55.9, 100.0, 109.8, 120.6, 120.7, 121.1, 141.4, 148.6; MS (ESI) *m/z* (%): 313.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for C₁₉H₂₁O₄: 313.1440; Found: 313.1444.

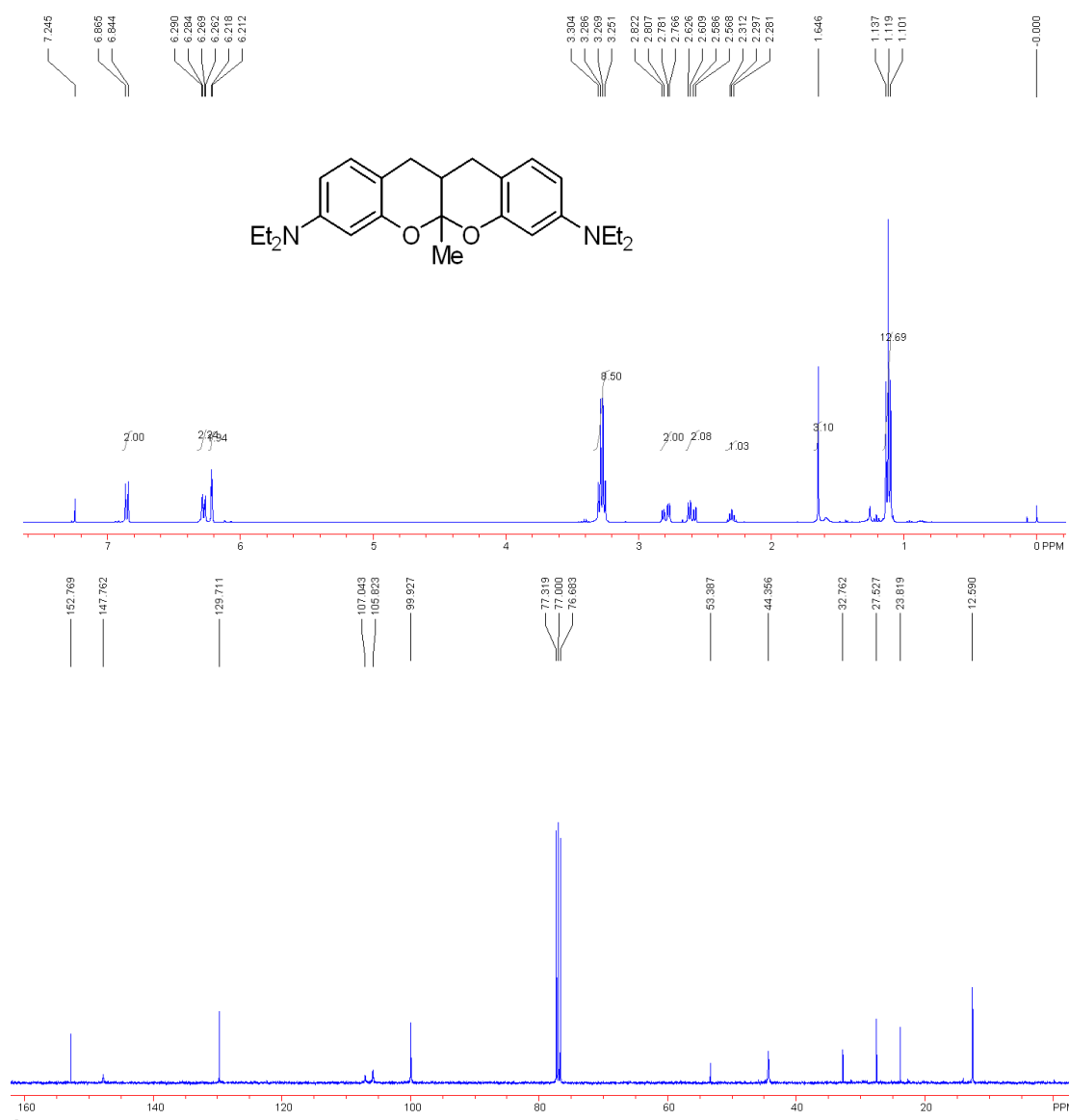




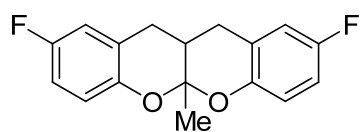
*N*³,*N*³,*N*⁸,*N*⁸-Tetraethyl-5a-methyl-5a,11,11a,12-tetrahydrochromeno[2,3-*b*]chromene-3,8-diamine **3ac**.



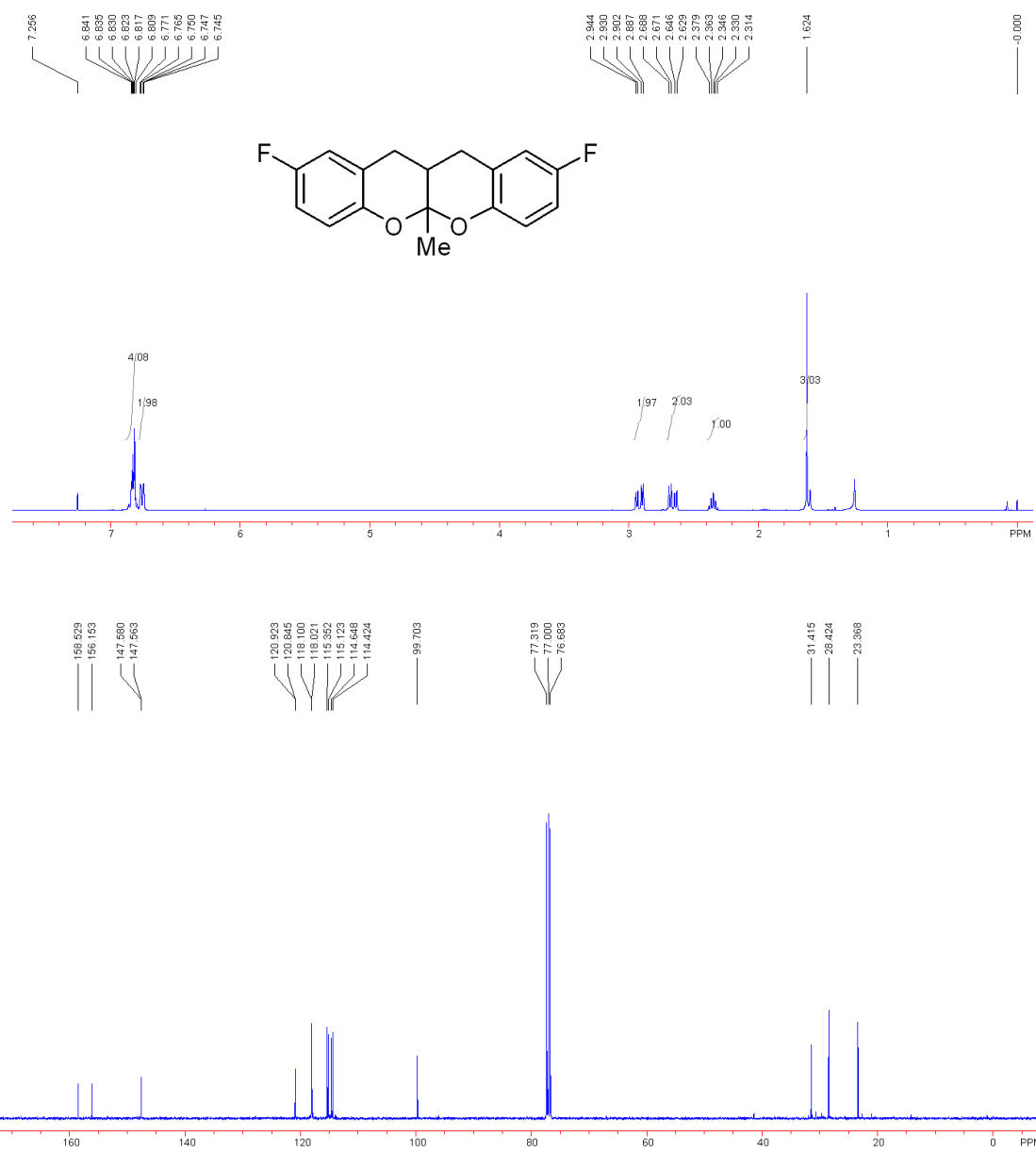
Light yellow liquid; ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.12 (t, *J* = 7.2 Hz, 12H), 1.65 (s, 3H), 2.26-2.33 (m, 1H), 2.60 (dd, *J* = 6.8, 16.0 Hz, 2H), 2.79 (dd, *J* = 6.0, 16.4 Hz, 2H), 3.28 (dd, *J* = 7.2, 14.0 Hz, 8H), 6.22 (d, *J* = 2.4 Hz, 2H), 6.28 (dd, *J* = 2.4, 8.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 12.6, 23.8, 27.5, 32.8, 44.4, 53.4, 99.9, 105.8, 107.0, 129.7, 147.8, 152.8; MS (ESI) *m/z* (%): 395.3 (M + H, 100); HRMS (Micromass LCT) Calcd. for C₂₅H₂₅N₂O₂:395.2699; Found: 395.2691.



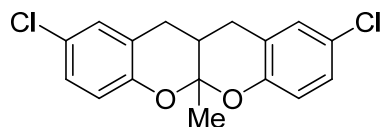
2,9-Difluoro-5a-methyl-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3ad**.



White solid; Mp. 120.1-120.5 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.62 (s, 3H), 2.31-2.38 (m, 1H), 2.66 (dd, $J = 6.8, 16.8$ Hz, 2H), 2.92 (dd, $J = 5.2, 16.8$ Hz, 2H), 6.75-6.77 (m, 2H), 6.81-6.84 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 234.4, 28.4, 31.4, 99.7, 114.5 (d, $J = 22.4$ Hz), 115.2 (d, $J = 22.9$ Hz), 118.1 (d, $J = 7.9$ Hz), 120.9 (d, $J = 7.8$ Hz), 147.6 (d, $J = 1.7$ Hz), 156.3 (d, $J = 37.6$ Hz); MS (EI) m/z (%): 288.1 (M, 50); HRMS (Micromass LCT) Calcd. for $\text{C}_{17}\text{H}_{14}\text{F}_2\text{O}_2$: 288.0962; Found: 288.0954.

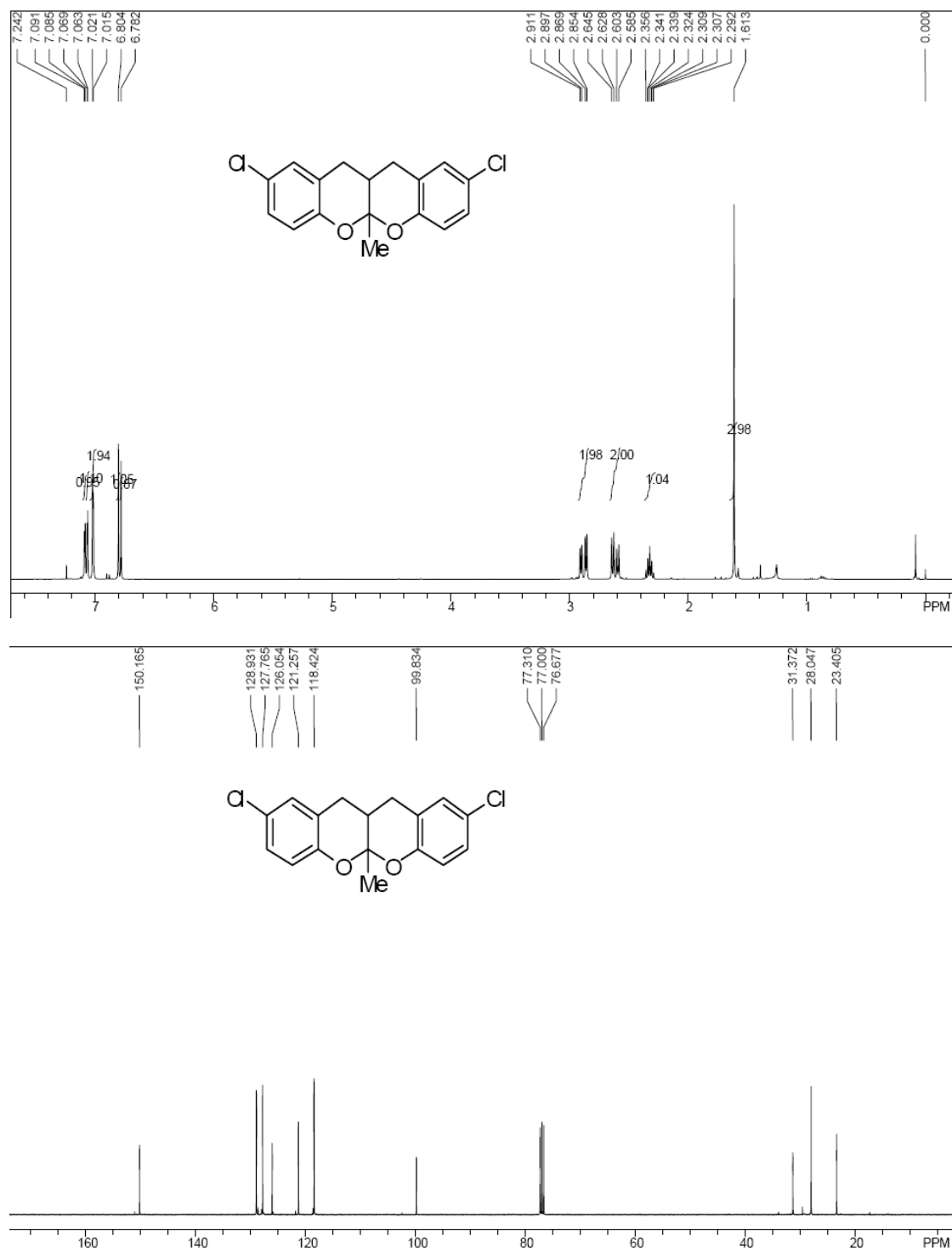


2,9-Dichloro-5a-methyl-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3ae**.

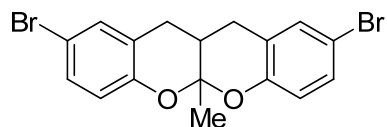


White solid; Mp. 140.7-141.0 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.61 (s, 3H), 2.29-2.36 (m, 1H), 2.62 (dd, $J = 6.8, 16.8$ Hz, 2H), 2.88 (dd, $J = 5.6, 16.8$ Hz, 2H), 6.79 (d, $J = 8.8$ Hz, 2H), 7.02 (d, $J = 2.4$ Hz, 2H), 7.07 (dd, $J = 2.4, 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 23.4, 28.0, 31.4, 99.8,

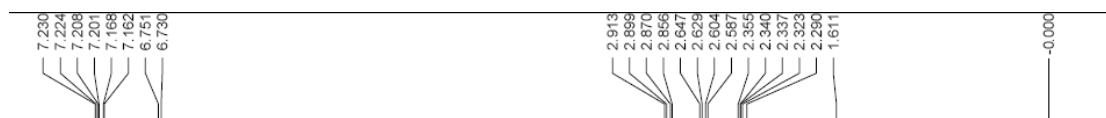
118.4, 121.3, 126.1, 127.8, 128.9, 150.2; MS (ESI) m/z (%): 343.0 (M + Na, 100);
HRMS (Micromass LCT) Calcd. for $C_{17}H_{14}Cl_2O_2Na$: 343.0269; Found: 343.0268.

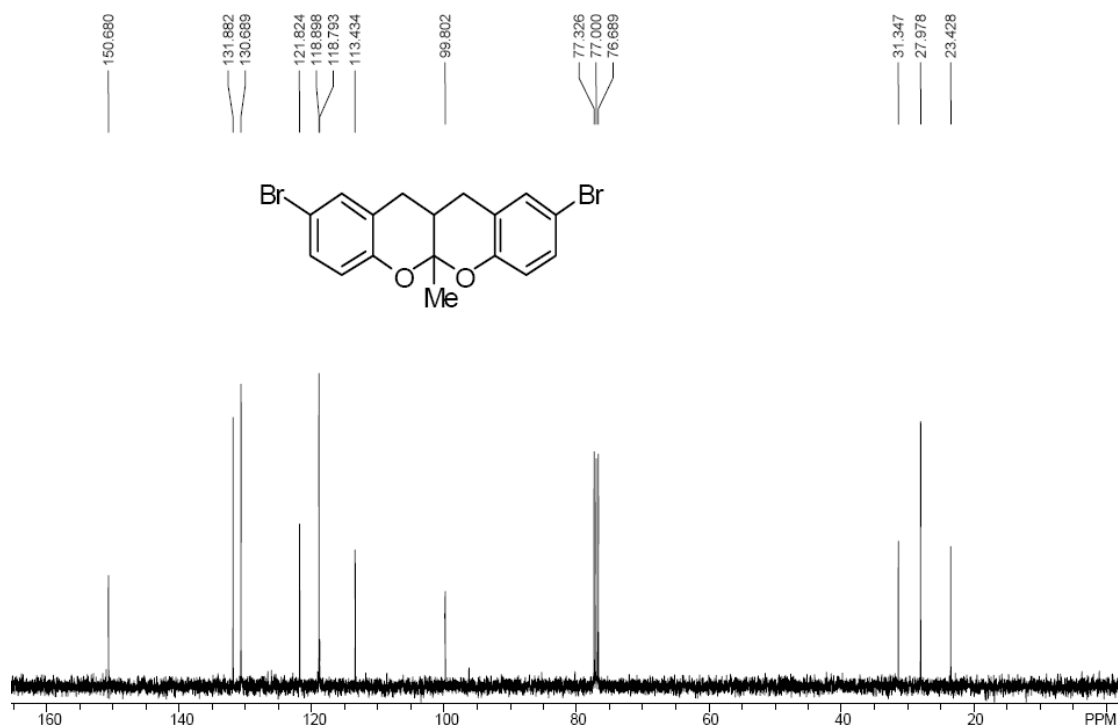


2,9-Dibromo-5a-methyl-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3af**.

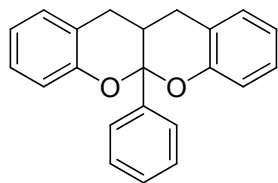


White solid; Mp. 162.2-162.8 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.61 (s, 3H), 2.29-2.36 (m, 1H), 2.62 (dd, $J = 6.8, 17.2$ Hz, 2H), 2.88 (dd, $J = 5.6, 17.2$ Hz, 2H), 6.74 (d, $J = 8.4$ Hz, 2H), 7.17 (d, $J = 2.4$ Hz, 2H), 7.22 (dd, $J = 2.4, 8.8$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 23.4, 28.0, 31.3, 99.8, 113.4, 118.8, 118.9, 121.8, 130.7, 131.9, 150.7; MS (ESI) m/z (%): 407.9 (M + H, 30); HRMS (Micromass LCT) Calcd. for $\text{C}_{17}\text{H}_{14}\text{Br}_2\text{O}_2$: 407.9361; Found: 407.9366.

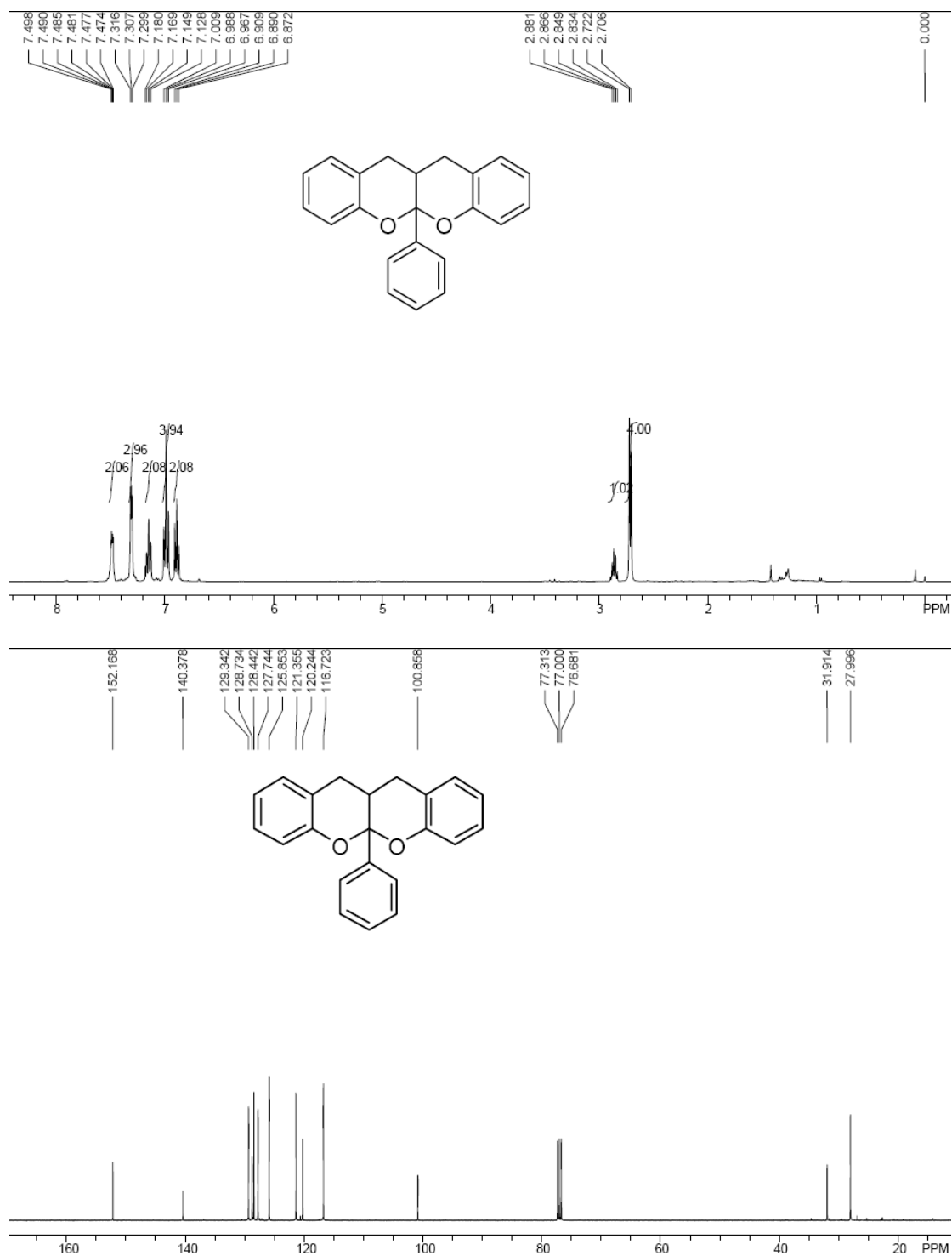




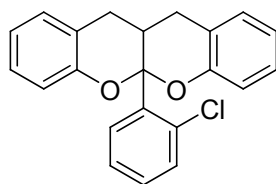
5a-Phenyl-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3ba**.



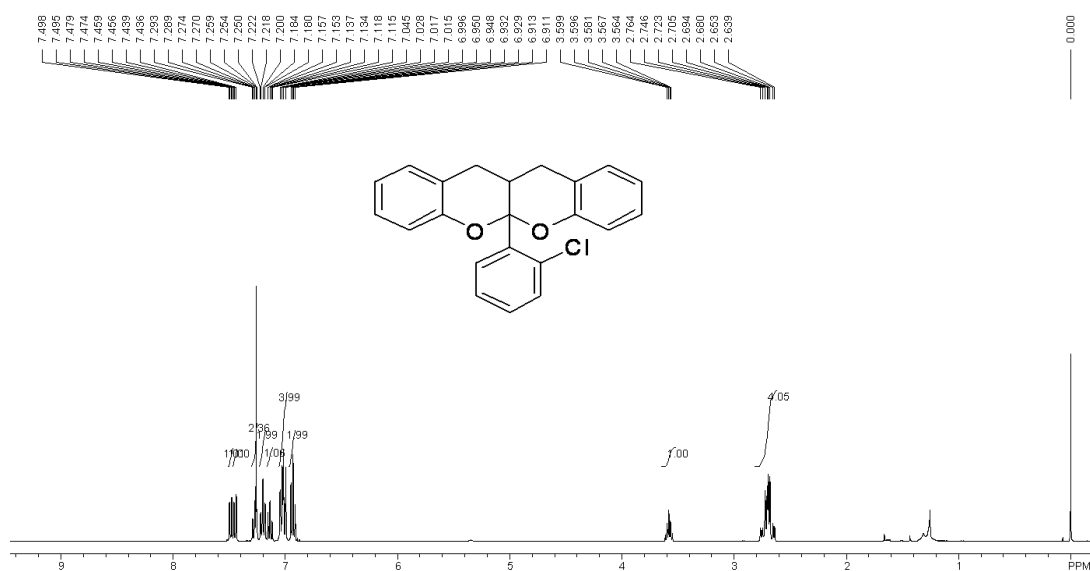
White solid; Mp. 130.5-131.0°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.72 (d, $J = 6.4$ Hz, 4H), 2.83-2.88 (m, 1H), 6.89 (t, $J = 7.2$ Hz, 2H), 6.99 (t, $J = 8.4$ Hz, 4H), 7.4 (t, $J = 8.4$ Hz, 2H), 7.31 (t, $J = 3.2$ Hz, 3H), 7.47-7.50 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 28.0, 31.9, 100.9, 116.7, 120.2, 121.4, 125.9, 127.7, 128.4, 128.7, 129.3, 140.4, 152.2; MS (ESI) m/z (%): 315.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{22}\text{H}_{19}\text{O}_2$: 315.1385; Found: 315.1381.

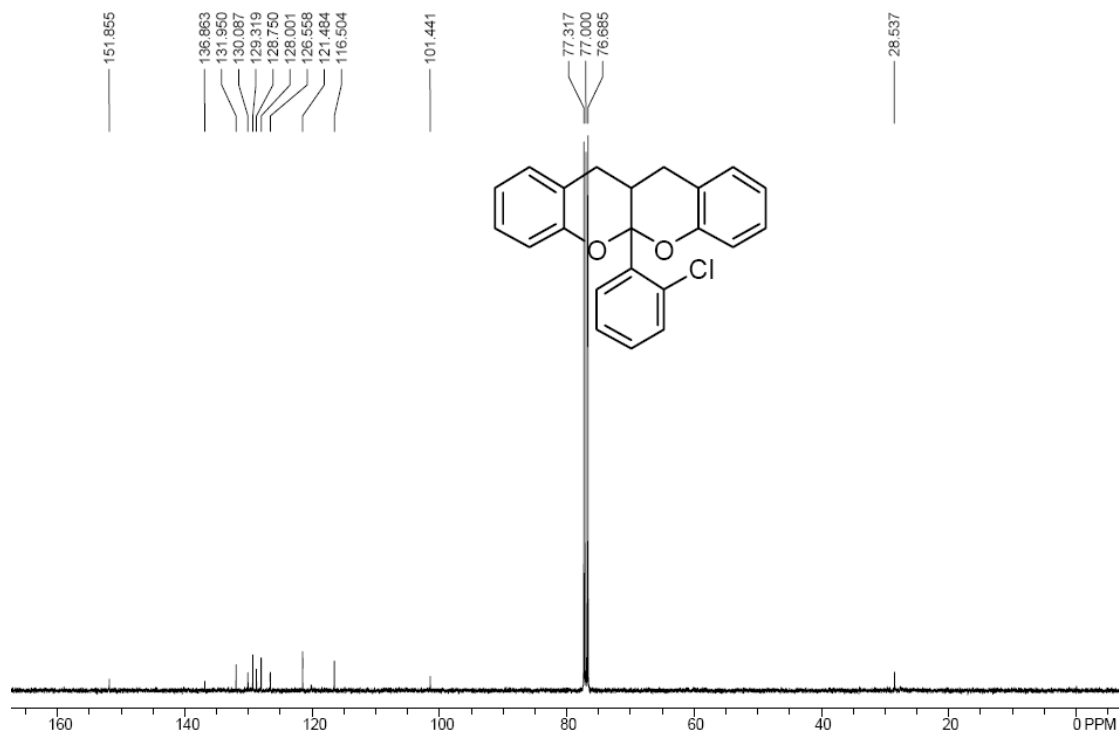


5a-(2-Chlorophenyl)-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3ca**.

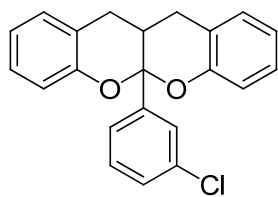


White solid; Mp. 189.0-189.5 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.64-2.76 (m, 4H), 3.56-3.60 (m, 1H), 6.93 (dt, $J = 1.2, 7.2$ Hz, 2H), 7.00-7.05 (m, 4H), 7.14 (dt, $J = 1.6, 8.0$ Hz, 1H), 7.20 (dt, $J = 1.6, 8.0$ Hz, 2H), 7.25-7.29 (m, 1H), 7.45 (dd, $J = 1.2, 8.0$ Hz, 1H), 7.49 (dd, $J = 1.2, 8.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 28.5, 101.4, 116.5, 121.5, 126.6, 128.0, 128.8, 129.3, 130.1, 132.0, 136.9, 151.9; MS (ESI) m/z (%): 350.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{22}\text{H}_{18}\text{ClO}_2$: 349.0995; Found: 349.0988.

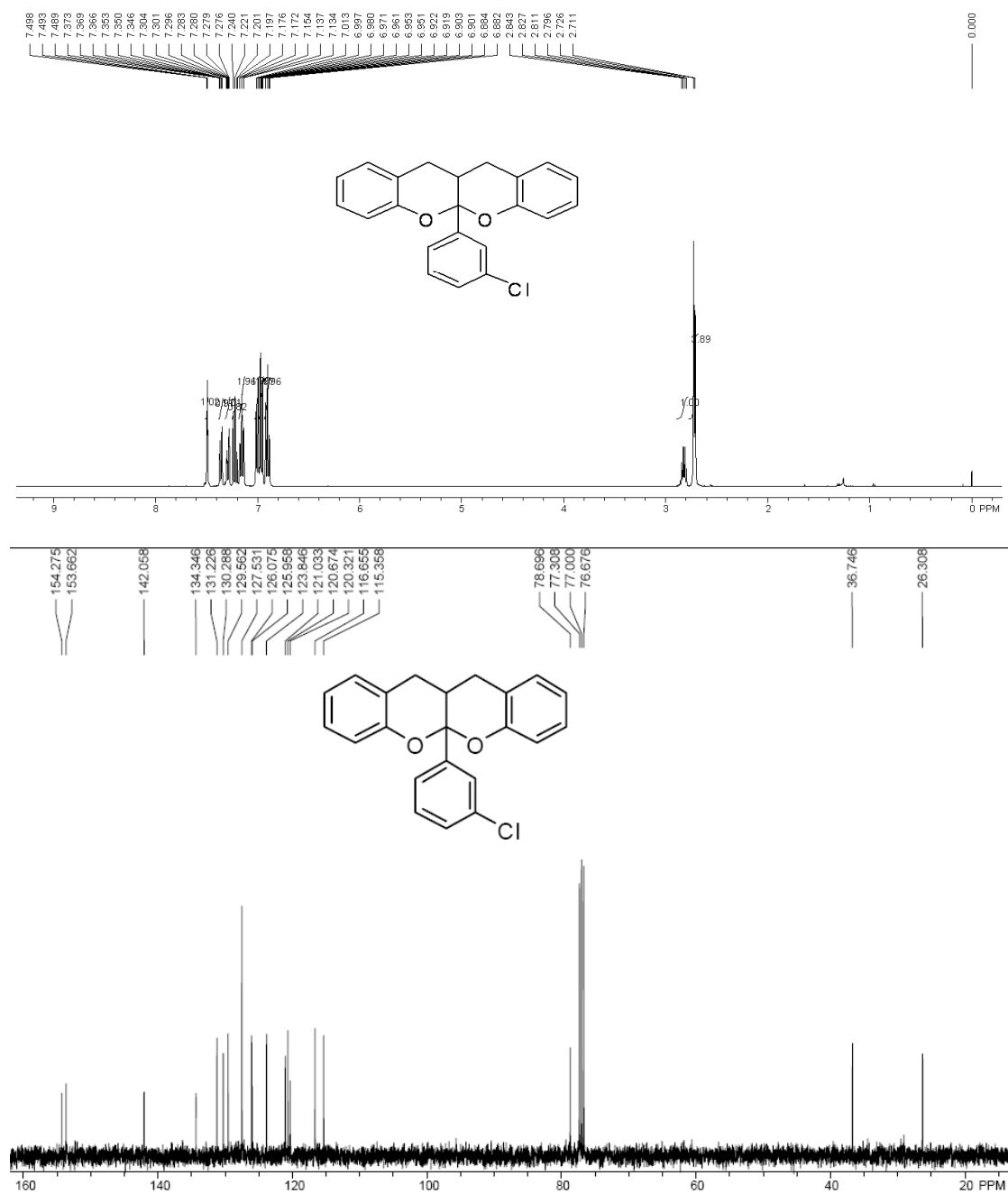




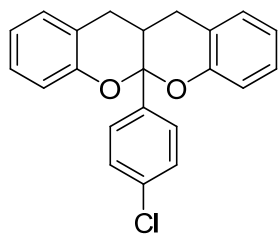
5a-(3-Chlorophenyl)-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3da**



White solid; Mp. 164.2-164.7 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.72 (d, $J = 6.0$ Hz, 4H), 2.80-2.84 (m, 1H), 6.90 (dt, $J = 1.2, 7.6$ Hz, 2H), 6.96 (d, $J = 8.0$ Hz, 2H), 7.01 (d, $J = 6.4$ Hz, 2H), 7.15 (dt, $J = 2.4, 8.8$ Hz, 2H), 7.22 (t, $J = 7.6$ Hz, 1H), 7.28-7.30 (m, 1H), 7.36 (dt, $J = 2.4, 7.6$ Hz, 1H), 7.49 (t, $J = 2.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 26.3, 36.7, 78.7, 115.4, 116.7, 120.3, 120.7, 121.0, 123.8, 126.0, 126.1, 127.5, 129.6, 130.3, 131.2, 134.3, 142.1, 153.7, 154.3; MS (ESI) m/z (%): 350.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{22}\text{H}_{18}\text{ClO}_2$: 349.0995; Found: 349.0984.

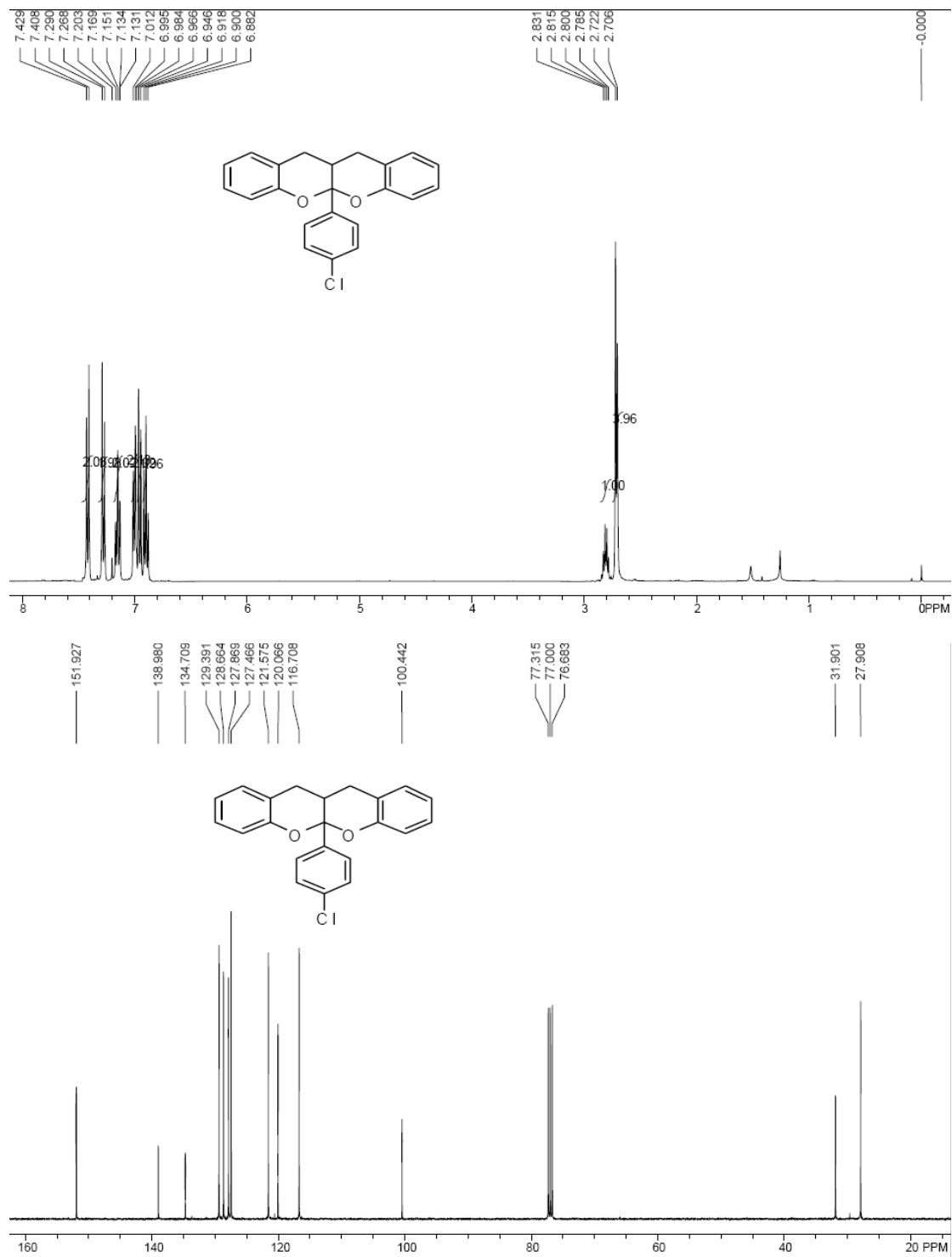


5a-(4-Chlorophenyl)-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3ea**

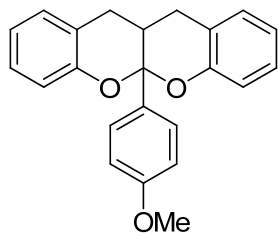


White solid; Mp. 138.1-138.5 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 2.71 (d, *J* = 6.4 Hz, 4H), 2.79-2.83 (m, 1H), 6.90 (t, *J* = 7.2 Hz, 2H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.00 (d, *J* = 6.8 Hz, 2H), 7.15 (dt, *J* = 1.2, 8.0 Hz, 2H), 7.28 (d, *J* =

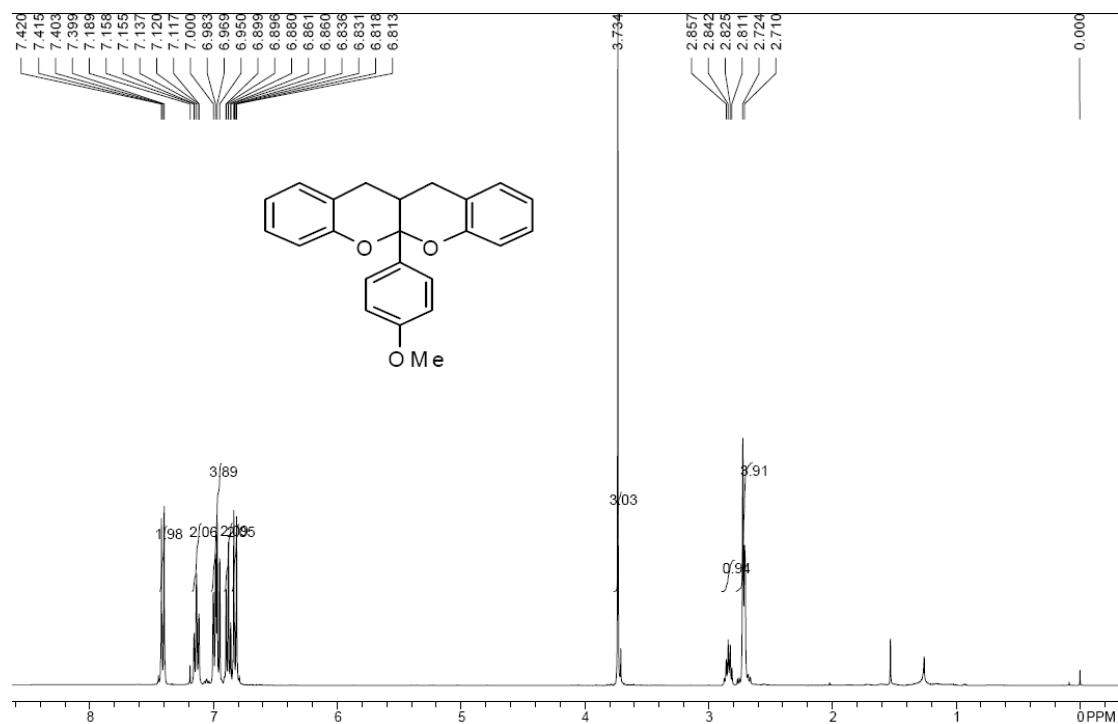
8.8 Hz, 2H), 7.42 (d, $J = 8.4$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 27.9, 31.9, 100.4, 116.7, 120.1, 121.6, 127.5, 127.9, 128.7, 129.4, 134.7, 139.0, 151.9; MS (ESI) m/z (%): 350.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{13}\text{H}_{14}\text{ClO}_3$: 349.0995; Found: 349.0990.

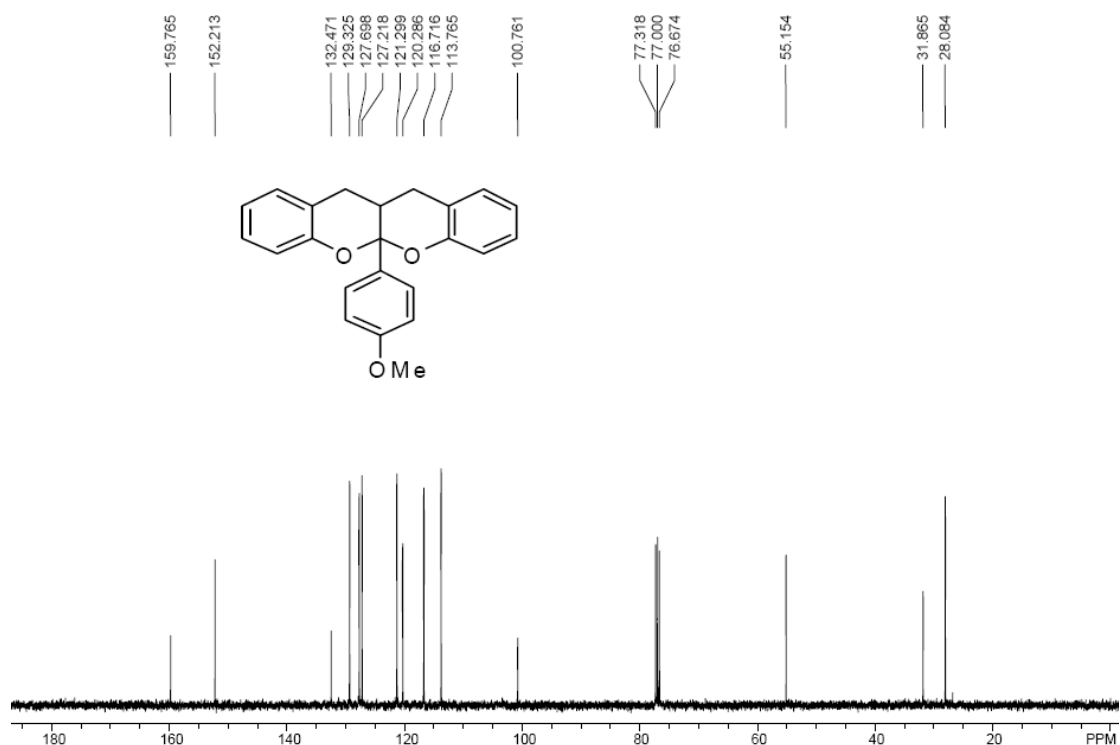


5a-(4-Methoxyphenyl)-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3fa**

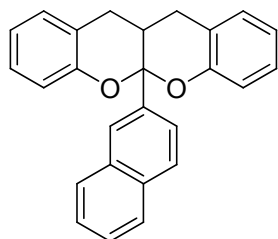


White solid; Mp. 146.8-147.1 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.72 (d, $J = 5.6$ Hz, 4H), 2.81-2.86 (m, 1H), 3.73 (s, 3H), 6.82 (dd, $J = 2.0$, 7.2 Hz, 2H), 6.88 (dt, $J = 0.8$, 7.6 Hz, 2H), 6.96 (d, $J = 8.4$ Hz, 2H), 6.99 (d, $J = 6.8$ Hz, 2H), 7.14 (dt, $J = 1.2$, 8.4 Hz, 2H), 7.41 (dd, $J = 2.0$, 6.8 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 28.1, 31.9, 55.2, 100.8, 113.8, 116.7, 120.3, 121.3, 127.2, 127.7, 129.3, 132.5, 152.2, 159.8; MS (ESI) m/z (%): 345.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{13}\text{H}_{14}\text{ClO}_3$: 345.1491; Found: 345.1495.

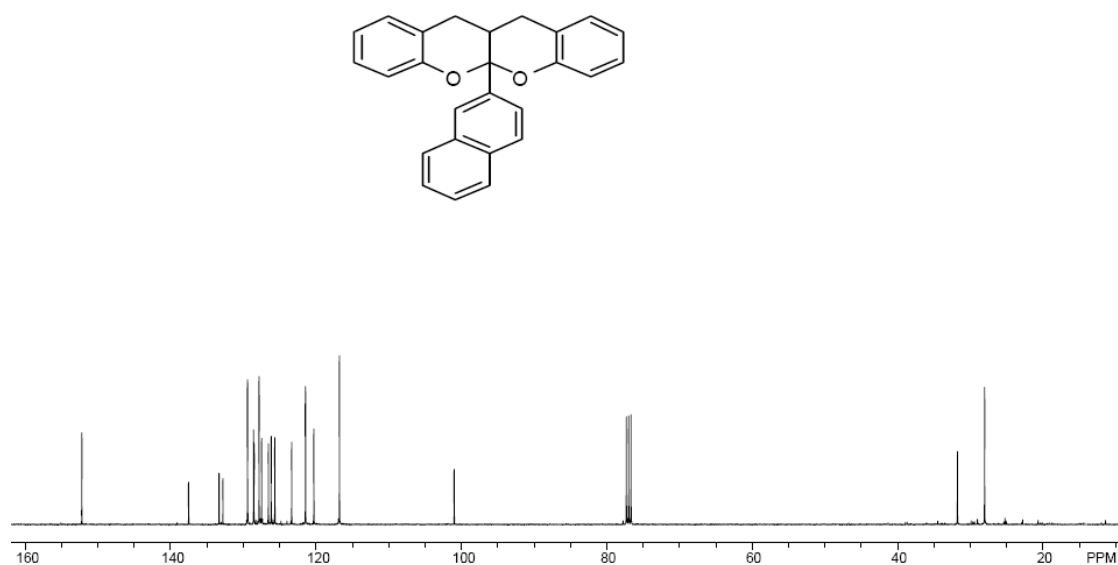
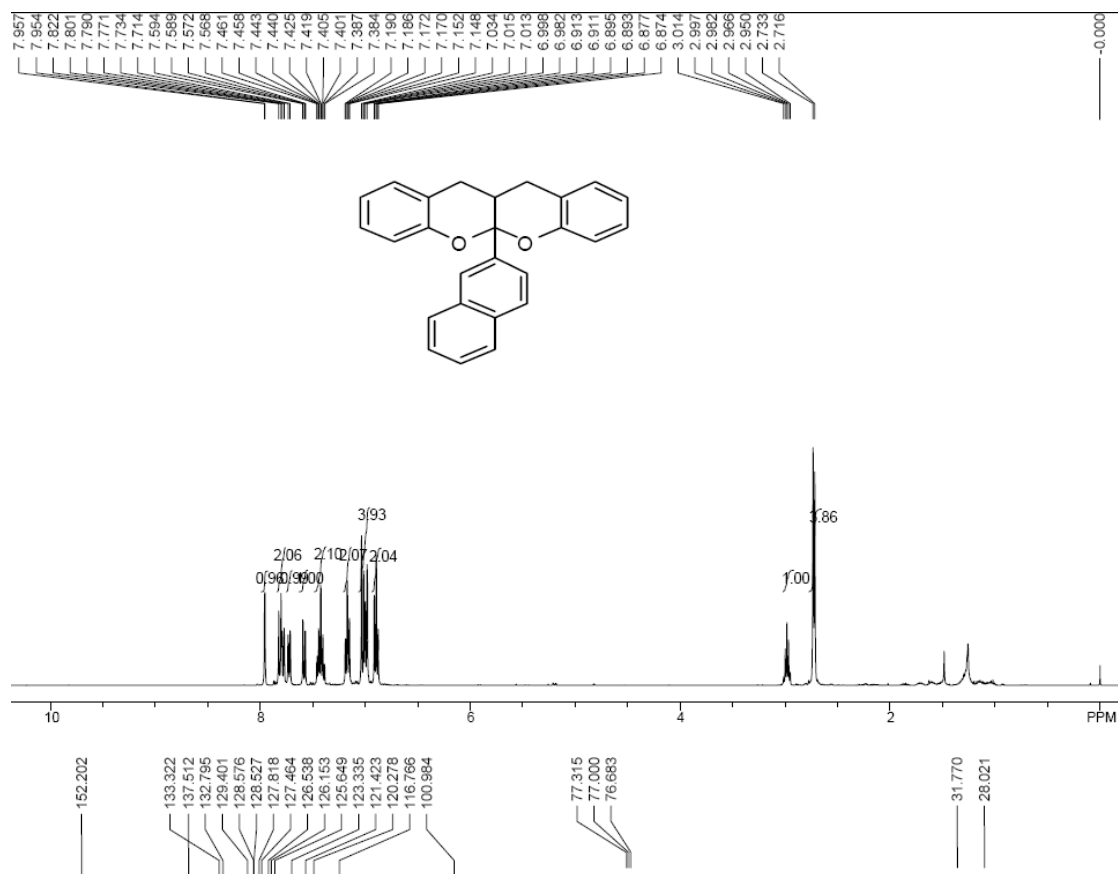




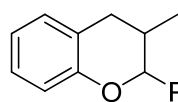
5a-(Naphthalen-2-yl)-5a,11,11a,12-tetrahydrochromeno[2,3-b]chromene **3ga**



White solid; Mp. 172.1-172.6°C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.72 (d, $J = 6.8$ Hz, 4H), 2.95-3.01 (m, 1H), 6.89 (dt, $J = 1.2, 7.6$ Hz, 2H), 6.99 (d, $J = 6.4$ Hz, 2H), 7.02 (d, $J = 8.4$ Hz, 2H), 7.17 (dt, $J = 1.6, 8.8$ Hz, 2H), 7.39-7.46 (m, 2H), 7.58 (dd, $J = 1.6, 8.4$ Hz, 1H), 7.72 (d, $J = 8.0$ Hz, 1H), 7.78 (d, $J = 7.6$ Hz, 1H), 7.81 (d, $J = 8.4$ Hz, 1H), 7.96 (d, $J = 1.2$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 28.0, 31.8, 101.0, 116.8, 120.3, 121.4, 123.3, 125.6, 126.1, 126.5, 127.5, 127.8, 128.6, 129.4, 132.8, 133.3, 137.5, 152.2; MS (ESI) m/z (%): 365.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{13}\text{H}_{14}\text{ClO}_3$: 365.1542; Found: 365.1543.

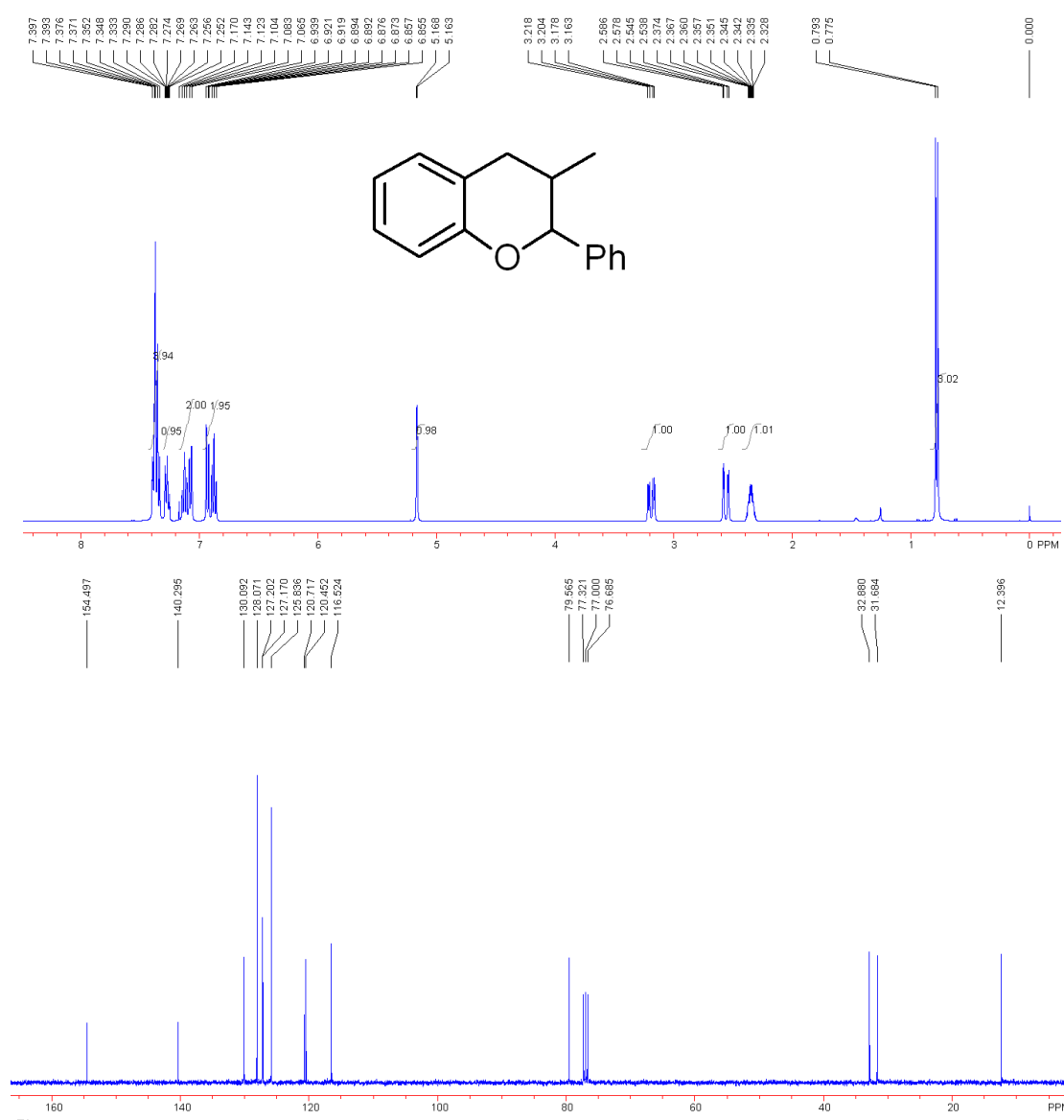


3-Methyl-2-phenylchroman **3ha**^[2]

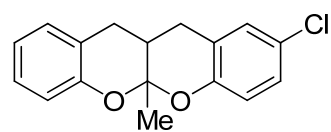


Colorless liquid; ¹H NMR (400 MHz, CDCl₃, TMS) δ 0.78 (d, *J* = 7.2 Hz, 3H), 2.33-2.37 (m, 1H), 2.56 (dd, *J* = 3.2, 16.4 Hz, 1H), 3.19 (dd, *J* = 5.6, 16.0 Hz, 1H), 5.17 (d, *J* = 2.0 Hz, 1H), 6.87 (dt, *J* = 0.8, 7.2 Hz, 1H), 6.93 (dd, *J* = 0.8,

8.0 Hz, 1H), 7.07 (d, $J = 7.2$ Hz, 1H), 7.12 (t, $J = 8.0$ Hz, 1H), 7.25-7.29 (m, 1H), 7.33-7.40 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 12.4, 31.7, 32.9, 79.6, 116.5, 120.5, 120.7, 125.8, 127.17, 127.20, 128.1, 130.1, 140.3, 154.5; MS (ESI) m/z (%): 224.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{16}\text{H}_{16}\text{O}$: 224.1201; Found: 224.1204.

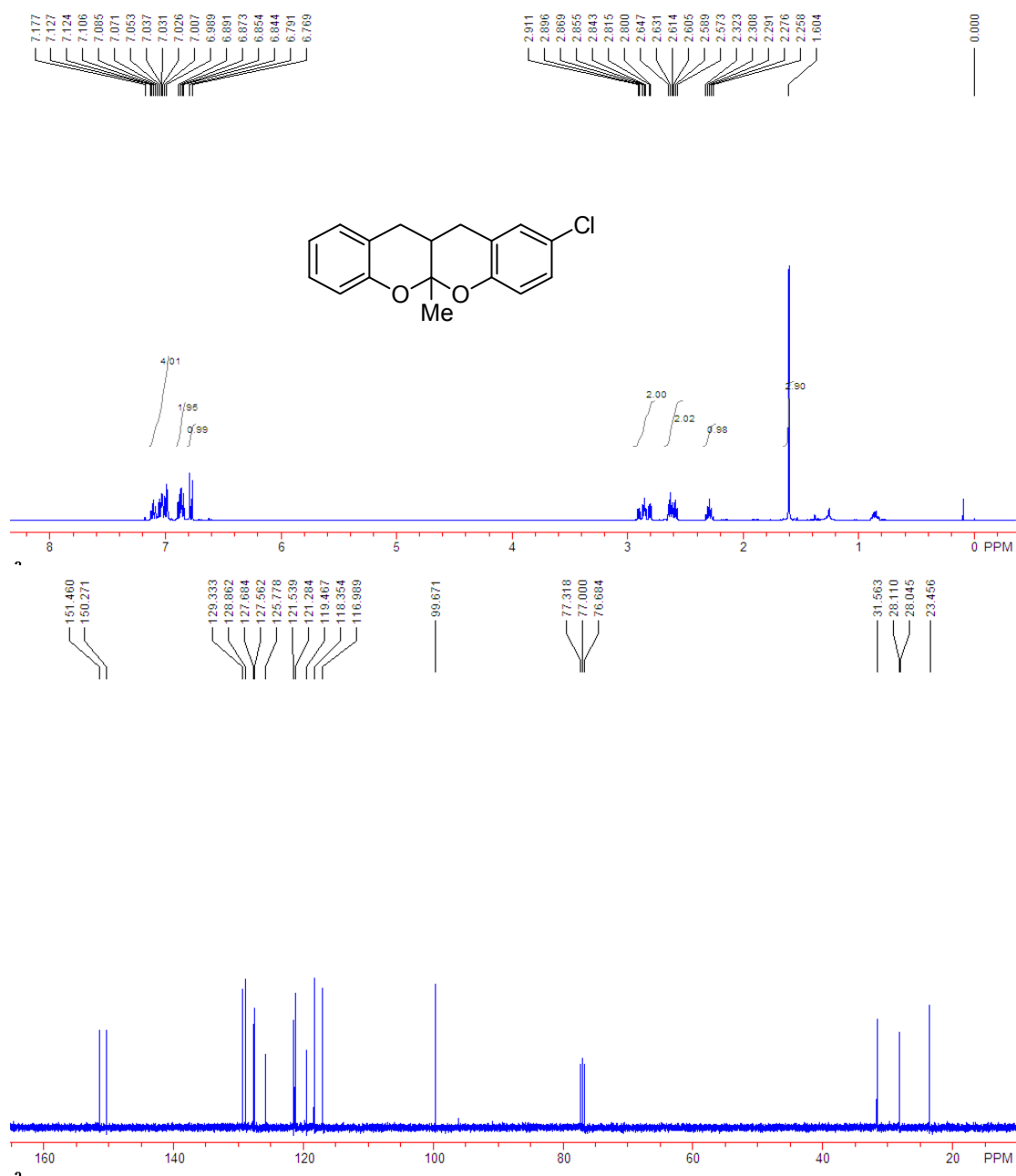


2-Chloro-5a-methyl-5a,11,11a,12-tetrahydrochromeno[2,3-*b*]chromene **3aa'**

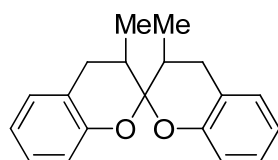


White solid; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.60 (s, 3H), 2.26-2.32 (m, 1H), 2.57-2.65 (m, 2H), 2.80-2.91 (m, 2H), 6.78 (d, $J = 8.8$ Hz,

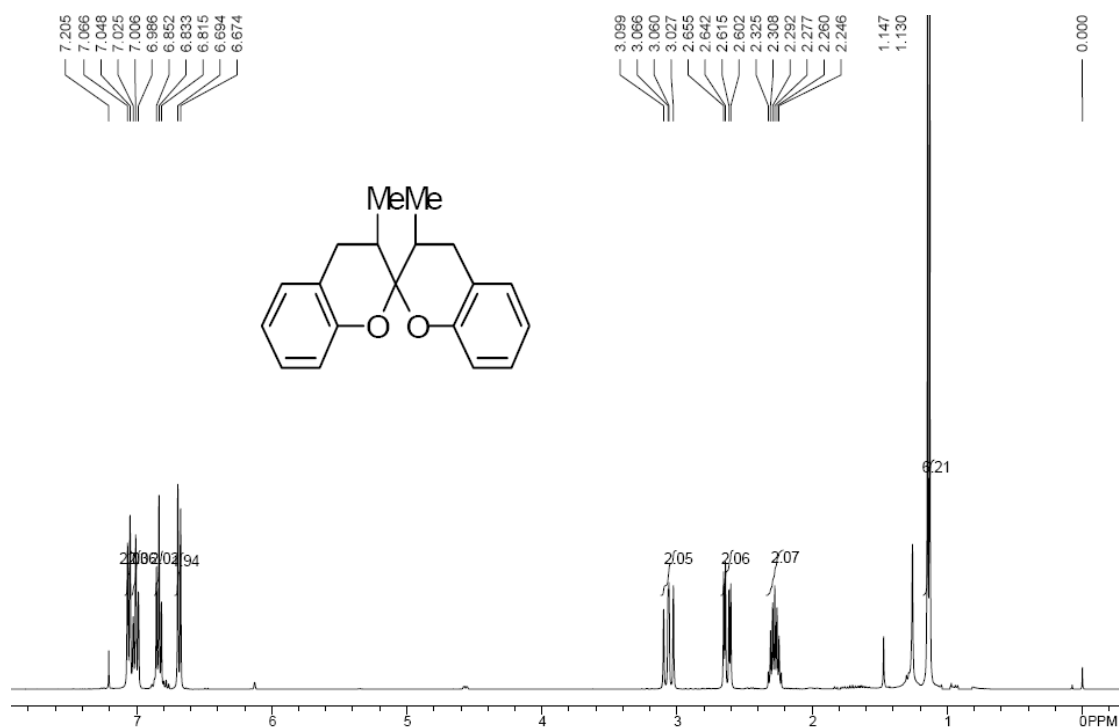
1H), 6.84-6.89 (m, 2H), 6.98-7.12 (m, 4H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ
23.5, 28.0, 28.1, 31.6, 99.7, 117.0, 118.4, 119.5, 121.3, 121.5, 125.8, 127.6, 127.7,
128.9, 129.3, 150.3, 151.5; MS (EI) *m/z* (%): 286.1 (M, 65); HRMS (TOF MS EI)
Calcd. for C₁₇H₁₅ClO₂: 286.0761, Found: 286.0761.

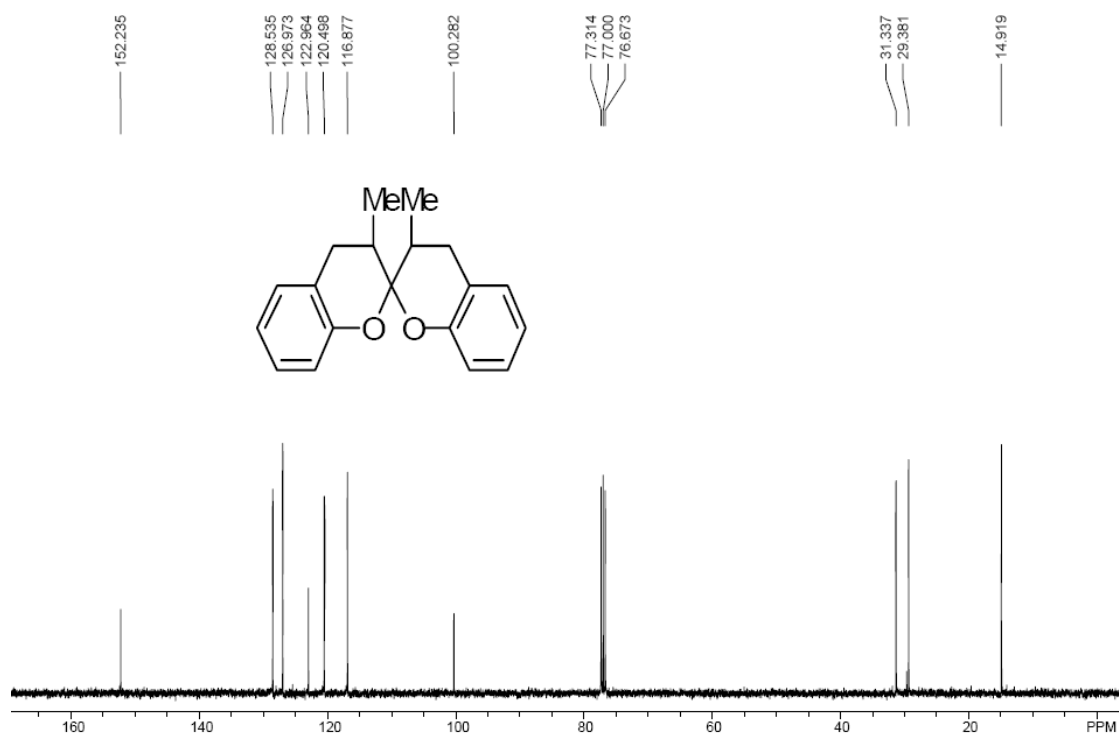


3,3'-Dimethyl-2,2'-spirobi[chroman] **4ia** ^[2]

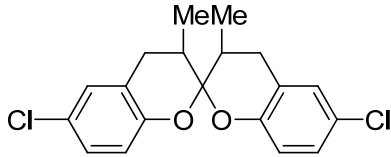


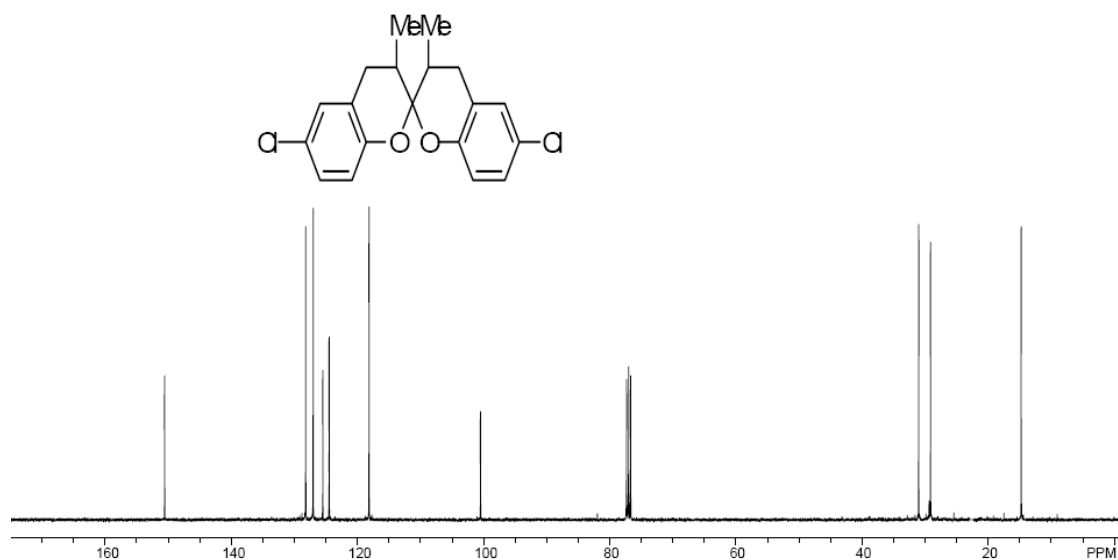
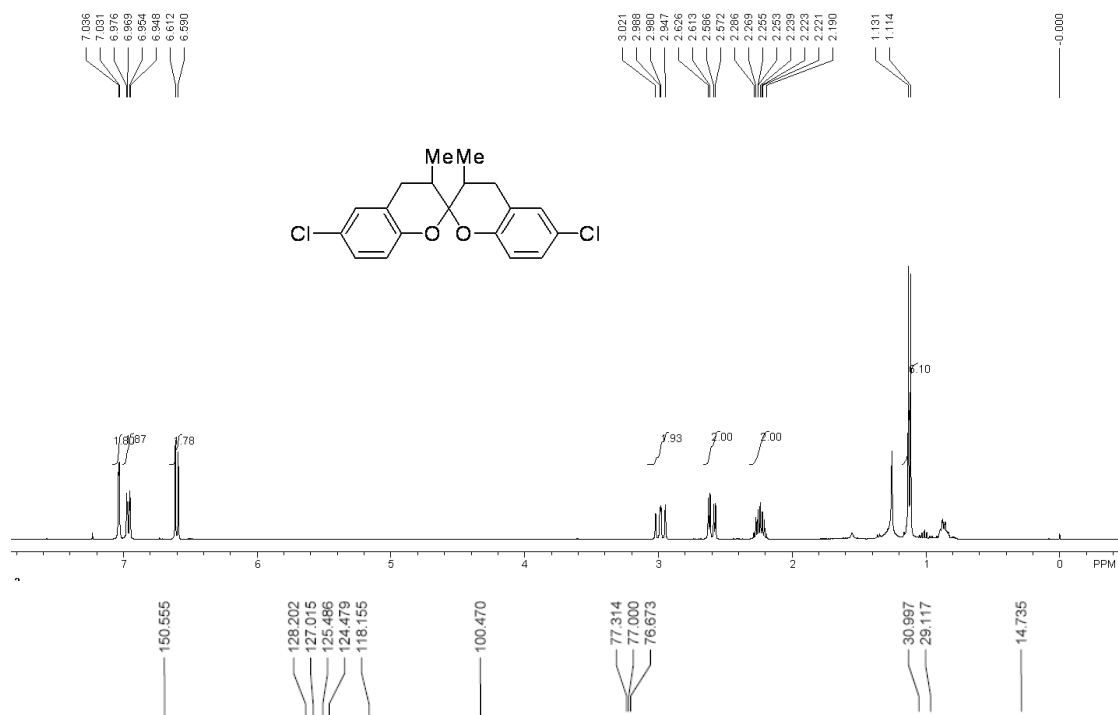
White solid; Mp. 131.0-131.5 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.14 (d, $J = 6.8$ Hz, 6H), 2.25-2.33 (m, 2H), 2.63 (dd, $J = 5.2, 16.0$ Hz, 2H), 3.06 (dd, $J = 13.2, 15.6$ Hz, 2H), 6.68 (d, $J = 8.0$ Hz, 2H), 6.83 (t, $J = 7.2$ Hz, 2H), 7.01 (t, $J = 8.0$ Hz, 2H), 7.06 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 14.9, 29.4, 31.3, 100.3, 116.9, 120.5, 123.0, 127.0, 128.5, 152.2; MS (ESI) m/z (%): 281.2 (M + H, 65); HRMS (Micromass LCT) Calcd. for $\text{C}_{19}\text{H}_{21}\text{O}_2$: 281.1542; Found: 281.1530.





6,6'-Dichloro-3,3'-dimethyl-2,2'-spirobichroman] **4ie**


White solid; Mp. 147.8-148.0 °C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.12 (d, *J* = 6.8 Hz, 6H), 2.19-2.29 (m, 2H), 2.60 (dd, *J* = 5.2, 16.0 Hz, 2H), 2.98 (dd, *J* = 13.2, 16.4 Hz, 2H), 6.60 (d, *J* = 8.8 Hz, 2H), 6.96 (dd, *J* = 2.8, 8.8 Hz, 2H), 7.03 (d, *J* = 2.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 14.7, 29.1, 31.0, 100.5, 118.2, 124.5, 125.5, 127.0, 128.2, 150.6; MS (EI) *m/z* (%): 348.1 (M, 45); HRMS (Micromass EI) Calcd. for C₁₉H₁₈Cl₂O₂: 348.0684; Found: 348.0680.



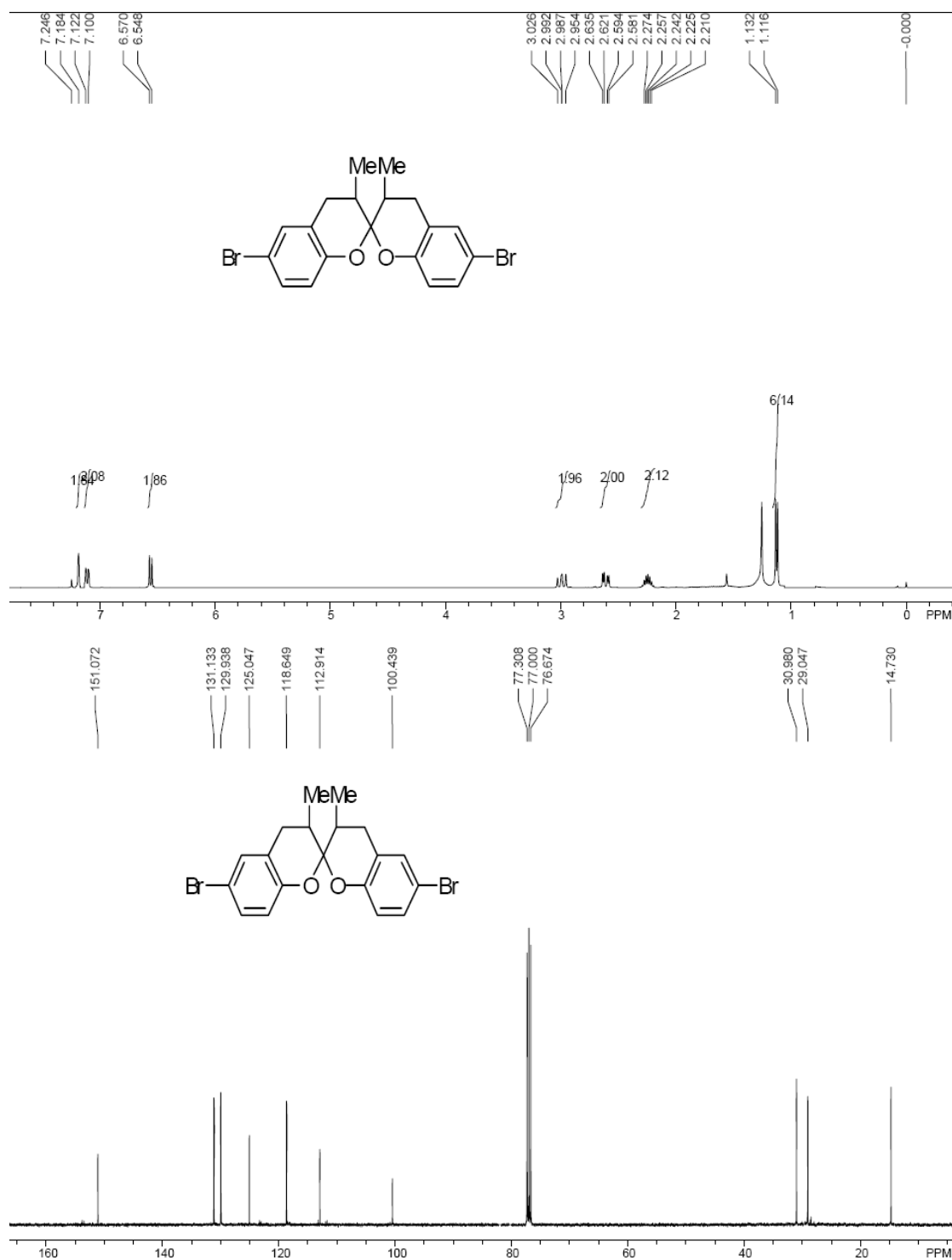
6,6'-Dibromo-3,3'-dimethyl-2,2'-spirobi[chroman] **4if**

Chemical structure of 6,6'-dibromo-3,3'-dimethyl-2,2'-spirobi[chroman] (**4if**):

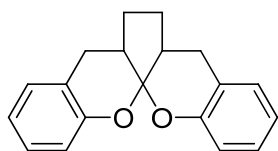
C[C@]12CCc3ccc(Br)cc3O1C2(Br)c4ccc(Br)cc4O

White solid; Mp. 159.7-160.2°C; ¹H NMR (400 MHz, CDCl₃, TMS) δ 1.12 (d, *J* = 6.4 Hz, 6H), 2.21-2.27 (m, 2H), 2.61 (dd, *J* = 5.6, 16.4 Hz, 2H), 2.99 (dd, *J* = 13.6, 15.6 Hz, 2H), 6.56 (d, *J* = 8.8 Hz, 2H), 7.11 (d, *J* = 8.8 Hz, 2H), 7.18 (s, 2H); ¹³C NMR (100 MHz, CDCl₃, TMS) δ 14.7, 29.0, 31.0, 100.4, 112.9,

118.6, 125.0, 129.9, 131.1, 151.1; MS (EI) m/z (%): 436.0 (M, 45); HRMS
(Micromass EI) Calcd. for $C_{18}H_{19}Br_2O_2$: 435.9674; Found: 435.9680.

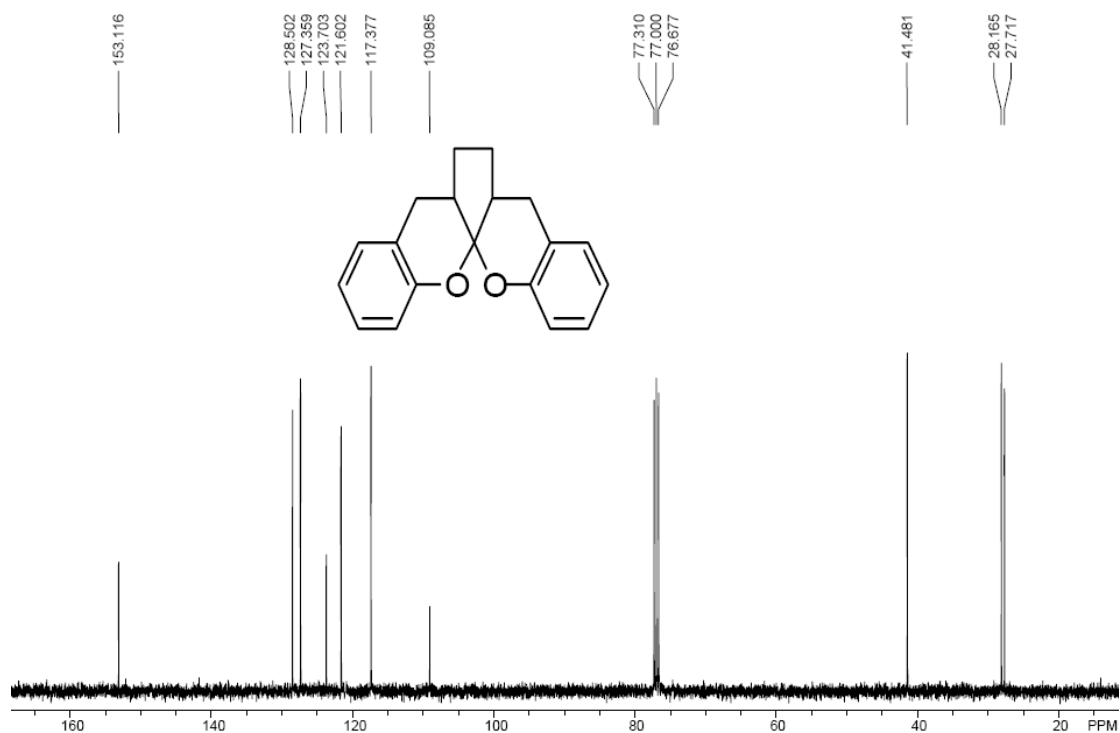


Spiroketal **4ja**^[3]

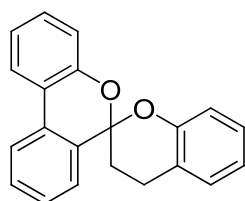


White solid; Mp. 133.8-1340.2 °C; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 1.48-1.54 (m, 2H), 1.97-2.04 (m, 2H), 2.46-2.53 (m, 2H), 2.61 (dd, $J = 7.6$, 15.6 Hz, 2H), 3.00 (dd, $J = 6.4$, 15.6 Hz, 2H), 6.81 (d, $J = 8.4$ Hz, 2H), 6.91-6.95 (m, 2H), 7.10-7.13 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 27.7, 28.2, 41.5, 109.1, 117.4, 121.6, 123.7, 127.4, 128.5, 153.1; MS (ESI) m/z (%): 279.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{19}\text{H}_{19}\text{O}_2$: 279.1385; Found: 279.1384.

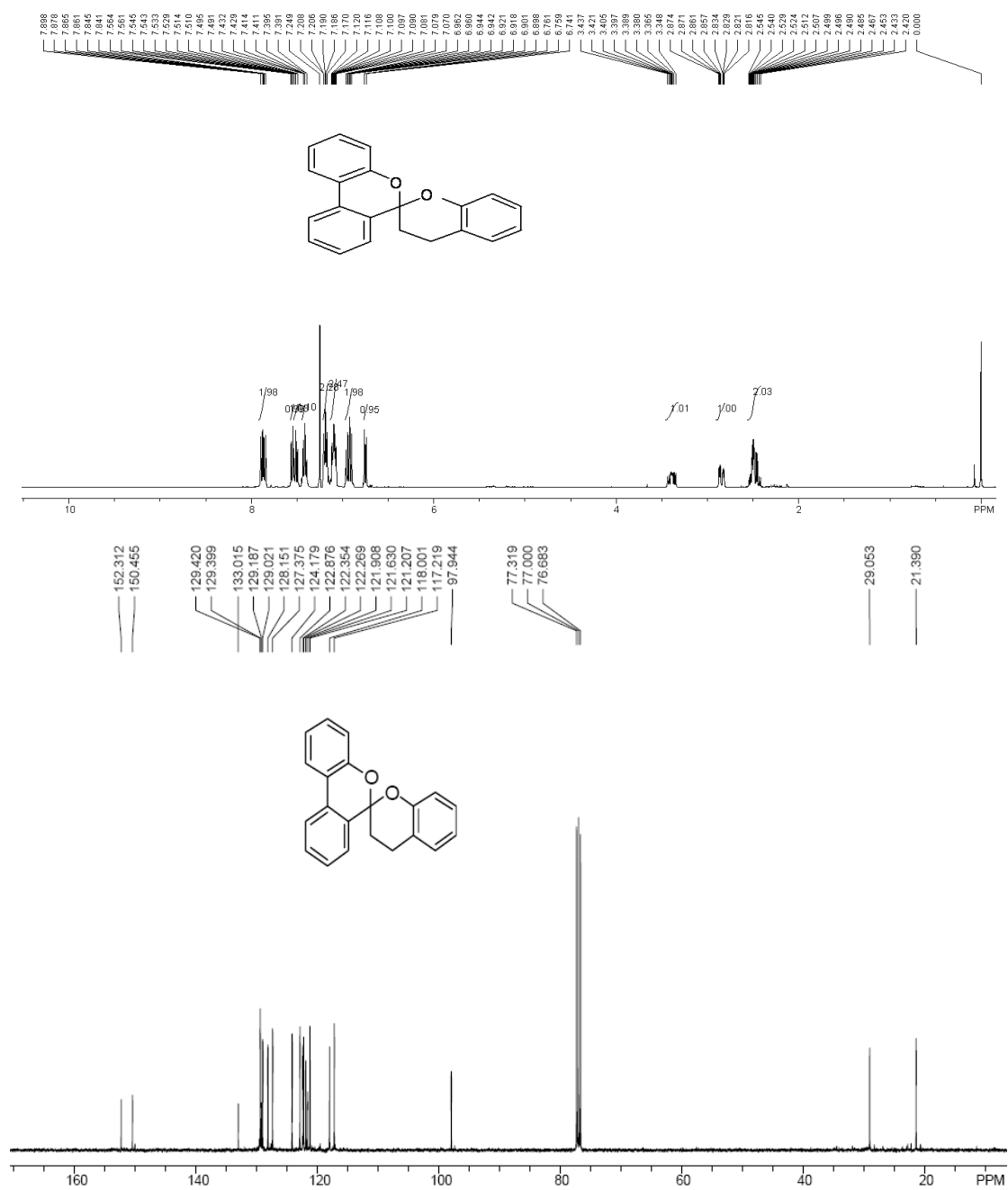




Spiro[benzo[*c*]chromene-6,2'-chroman] **6**



Colorless liquid; ^1H NMR (400 MHz, CDCl_3 , TMS) δ 2.42-2.55 (m, 2H), 2.82-2.87 (m, 1H), 3.35-3.44 (m, 1H), 6.75 (dd, $J = 0.8, 8.0$ Hz, 1H), 6.91 (dd, $J = 1.2, 8.0$ Hz, 1H), 6.95 (dd, $J = 0.8, 7.2$ Hz, 1H), 7.07-7.12 (m, 2H), 7.41 (dt, $J = 1.2, 7.2$ Hz, 1H), 7.51 (dt, $J = 1.6, 7.6$ Hz, 1H), 7.55 (dd, $J = 1.2, 7.6$ Hz, 1H), 7.85 (dt, $J = 1.6, 8.0$ Hz, 1H), 7.89 (d, $J = 8.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3 , TMS) δ 21.4, 29.1, 97.9, 117.2, 118.0, 121.2, 121.6, 121.9, 122.3, 122.4, 122.9, 124.2, 127.4, 128.2, 129.0, 129.2, 129.39, 129.42, 133.0, 150.5, 152.3; MS (ESI) m/z (%): 301.1 (M + H, 100); HRMS (Micromass LCT) Calcd. for $\text{C}_{21}\text{H}_{17}\text{O}_2$: 301.1229; Found: 301.1228.



Reference

1. (a) M. A. Brimble, C. L. Flowers, M. Trzoss, K. Y. Tsang, *Tetrahedron* **2006**, *62*, 5883-5896; (b) G. Zhou, D. Zheng, S. Da, Z. Xie, Y. Li, *Tetrahedron Lett.* **2006**, *47*, 3349-3352.
2. J. D. Chambers, J. Crawford, H. W. R. Williams, C. Dufresne, J. Scheigetz, M. A. Bernstein, C. K. Lau, *Canadian Journal of Chemistry* **1992**, *70*, 1717-1732.

3. (a) Y. Xin, H. Jiang, J. Zhao, S. Zhu, *Tetrahedron* **2008**, *64*, 9315-9319; (b) X. Wang, Z. Han, Z. Wang, K. Ding, *Angew. Chem. Int. Ed.* **2012**, *51*, 936-940.