

Supporting Information

Vilsmeier-Haack Reaction of 1-Cyclopropyl-2-arylethanones

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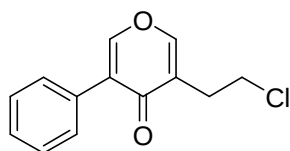
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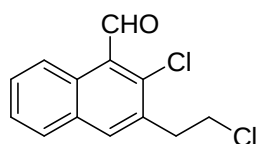
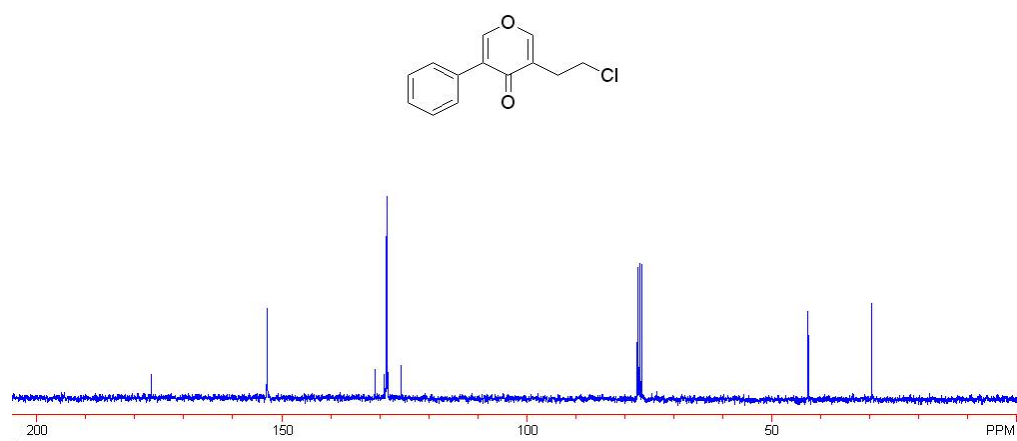
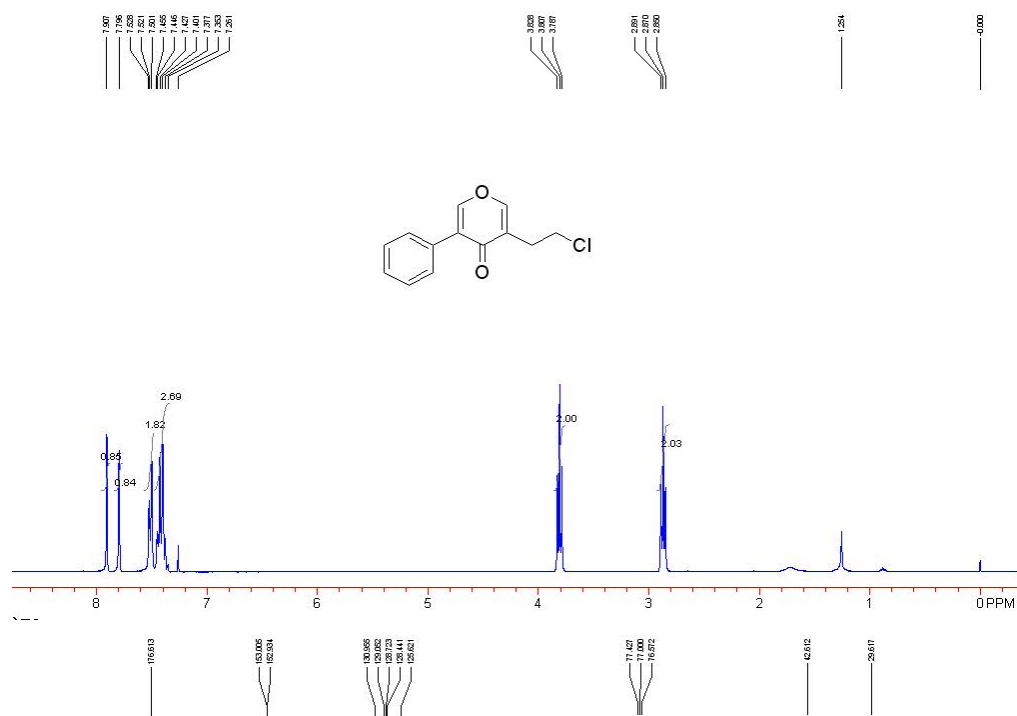
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General remarks. Melting points are uncorrected. ^1H and ^{13}C NMR spectra were recorded at 300 and 75 MHz respectively. Mass and HRMS spectra were recorded by EI methods. Organic solvents used were dried by standard methods when necessary. Satisfactory CHN microanalyses were obtained with an analyzer. Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel coated plates. Flash column chromatography was carried out using silica gel at increased pressure.

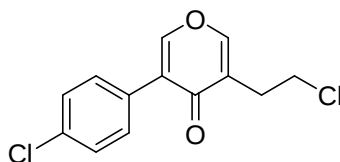
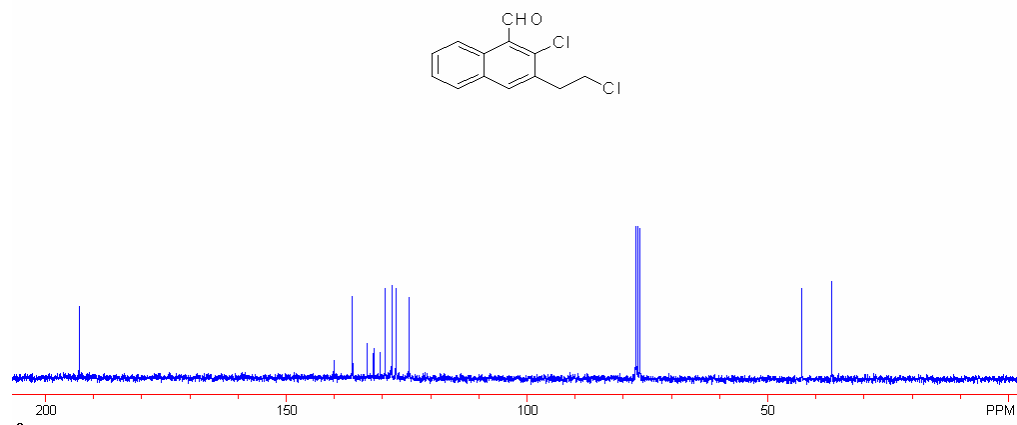
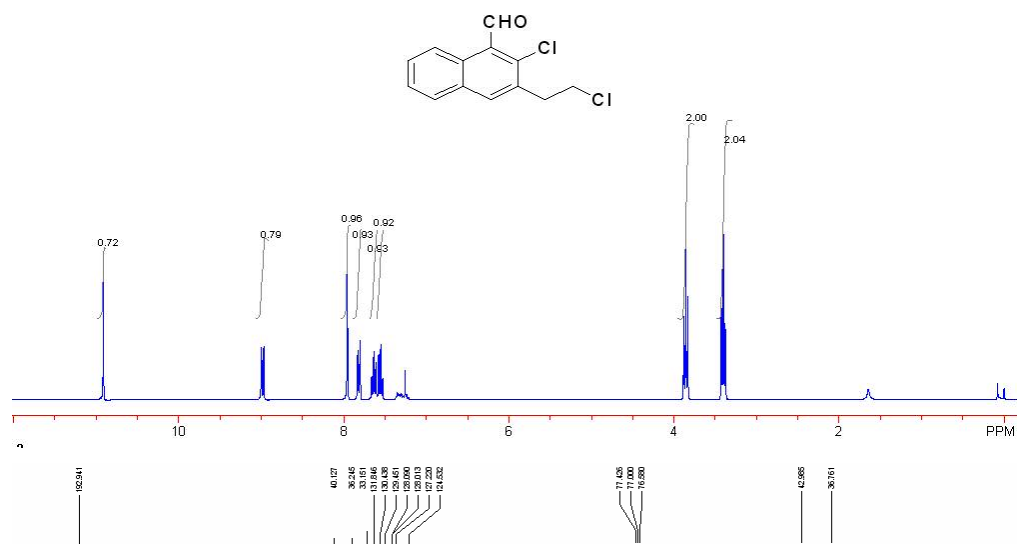
General Procedure for the Reaction of 2a and 3a. The Vilsmeier reagent was prepared by adding POCl_3 (4.5 mmol) dropwise to ice-cold dry DMF (2 mL) under stirring. After ten minutes, to the above Vilsmeier reagent was added 1-cyclopropyl-2-phenylethanone **1a** (48 mg, 0.3 mmol) as a solution in DMF (1.0 mL). The reaction mixture was stirred at 100 °C (120 °C/135 °C) for 20 minutes. Then the mixture was poured into ice-cold water (20 mL) and extracted with dichloromethane (3×20 mL), the combined organic phase was washed with water (3×20 mL), dried over MgSO_4 , filtered. The organic layer was removed under reduced pressure and then the residue was purified by a flash column chromatography.



3-(2-Chloroethyl)-5-phenyl-4H-pyran-4-one (2a). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.87 (t, $J = 6.0$ Hz, 2H, CH_2), 3.81 (t, $J = 6.0$ Hz, 2H, CH_2), 7.38-7.46 (m, 3H, Ar), 7.50-7.53 (m, 2H, Ar), 7.80 (s, 1H), 7.91 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.6, 42.6, 125.6, 128.4, 128.7, 129.0, 131.0, 152.9, 153.0, 176.6; IR (CH_2Cl_2): ν 3082, 2964, 2924, 1646, 1616, 1493, 1448, 1281, 1192, 1047 cm^{-1} ; MS (EI) m/z (%): 234 [M^+] (12.3), 235 (7.0), 200 (15.9), 199 (100), 119 (6.8), 115 (7.3), 102 (16.0), 89 (8.8); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_{11}\text{O}_2\text{Cl}$ (M^+) requires 234.0448, Found: 234.0457.

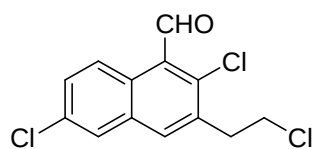
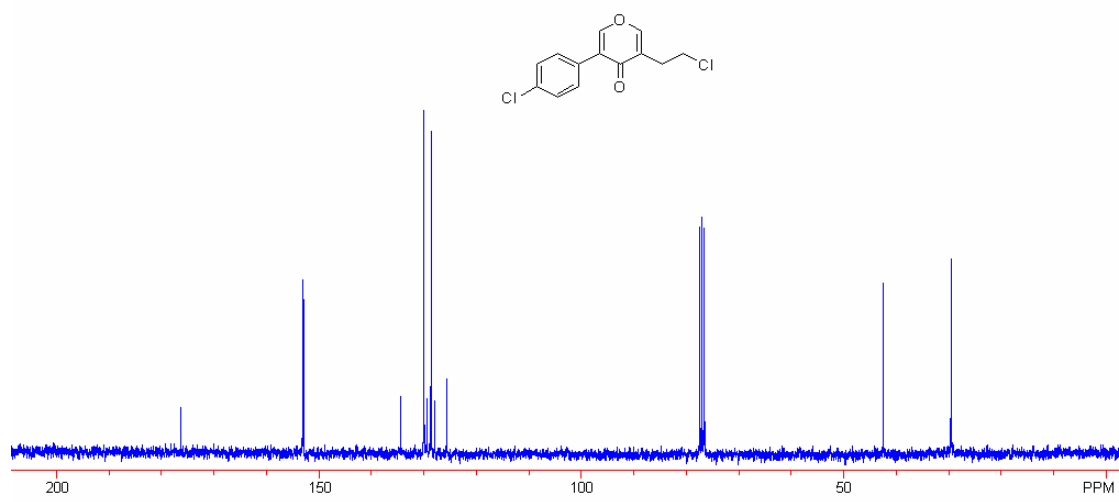
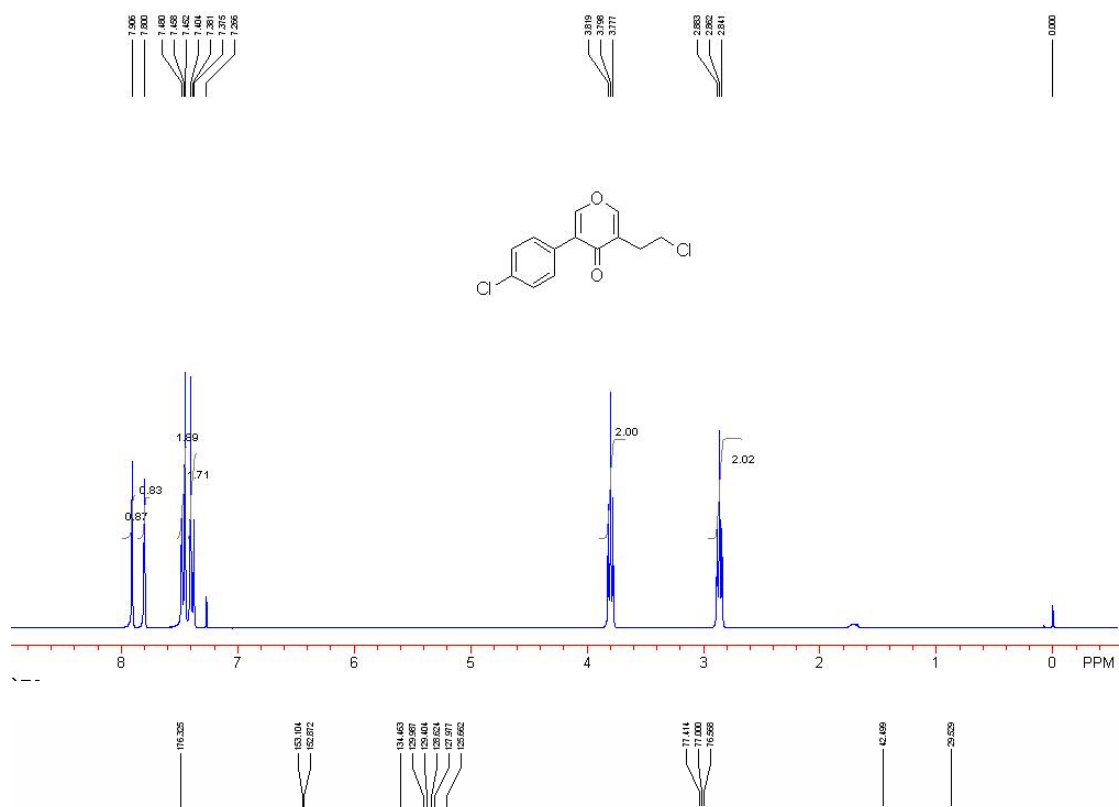


2-Chloro-3-(2-chloroethyl)-1-naphthaldehyde (3a). A yellow solid. m.p. 68-70 °C. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 3.40 (t, *J* = 6.6 Hz, 2H, CH₂), 3.86 (t, *J* = 6.6 Hz, 2H, CH₂), 7.55 (t, *J* = 7.2 Hz, 1H, Ar), 7.64 (td, *J* = 7.2 Hz, *J* = 0.9 Hz, 1H, Ar), 7.82 (d, *J* = 7.8 Hz, 1H, Ar), 7.96 (s, 1H, Ar), 8.98 (d, *J* = 9.0 Hz, 1H, Ar), 10.9 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 36.7, 43.0, 124.5, 127.2, 128.0, 128.1, 129.5, 130.4, 131.8, 133.1, 136.2, 140.1, 192.9; IR (CH₂Cl₂): ν 3058, 2925, 2853, 1690, 1589, 1495, 1454, 1377, 1261, 1069 cm⁻¹; MS (EI) *m/z*

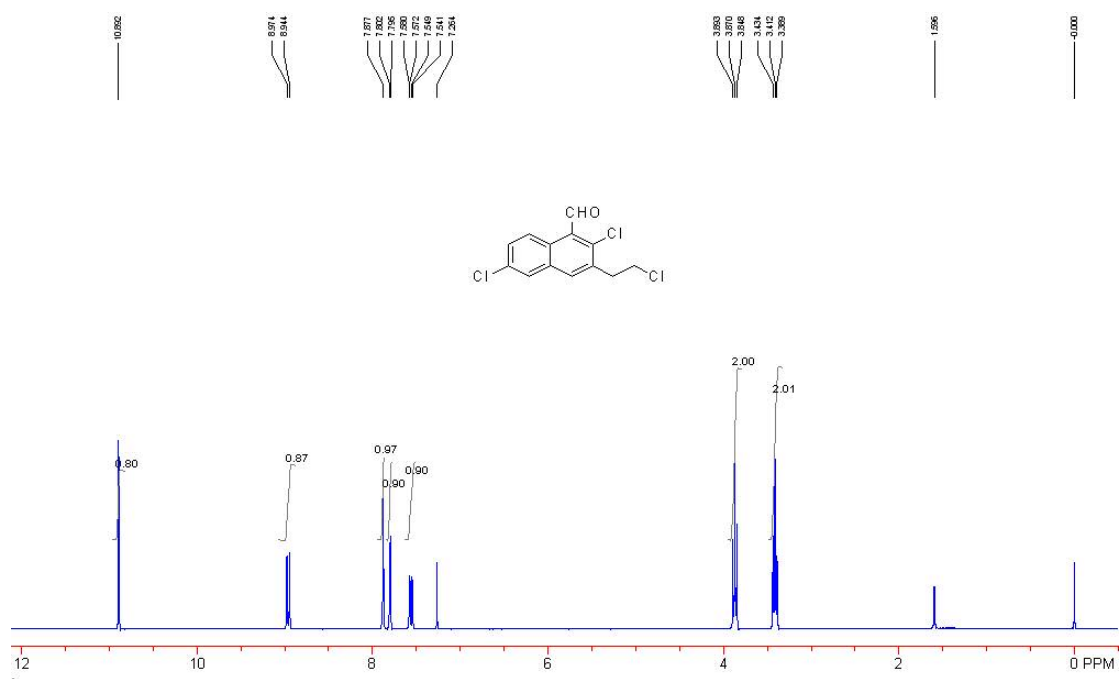


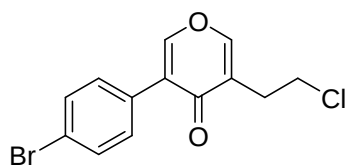
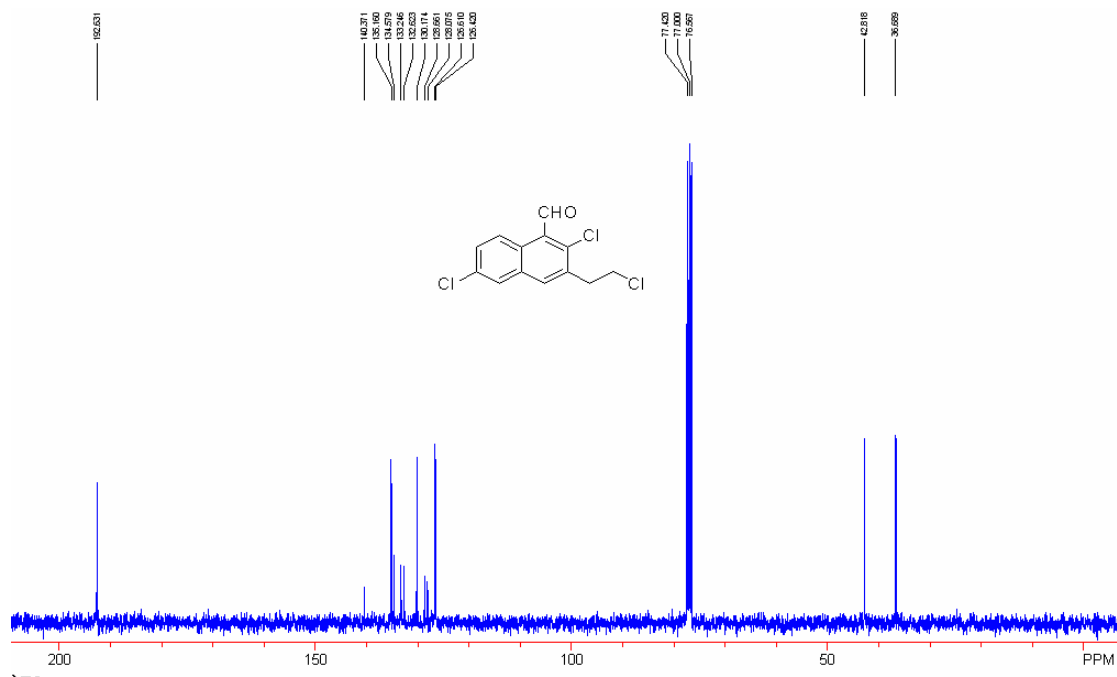
S4

152.9, 153.1, 176.3; IR (CH₂Cl₂): ν 3089, 2962, 2925, 1691, 1655, 1487, 1449, 1286, 1197, 1063 cm⁻¹; MS (EI) m/z (%): 268 [M⁺] (15.1), 235 (42.8), 234 (16.8), 233 (100), 270 (10.1), 136 (12.4), 91 (8.0), 53 (8.1); HRMS (EI) Calcd. for C₁₃H₁₀Cl₂O₂ (M⁺) requires 268.0058, Found: 268.0051.

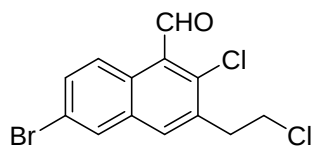
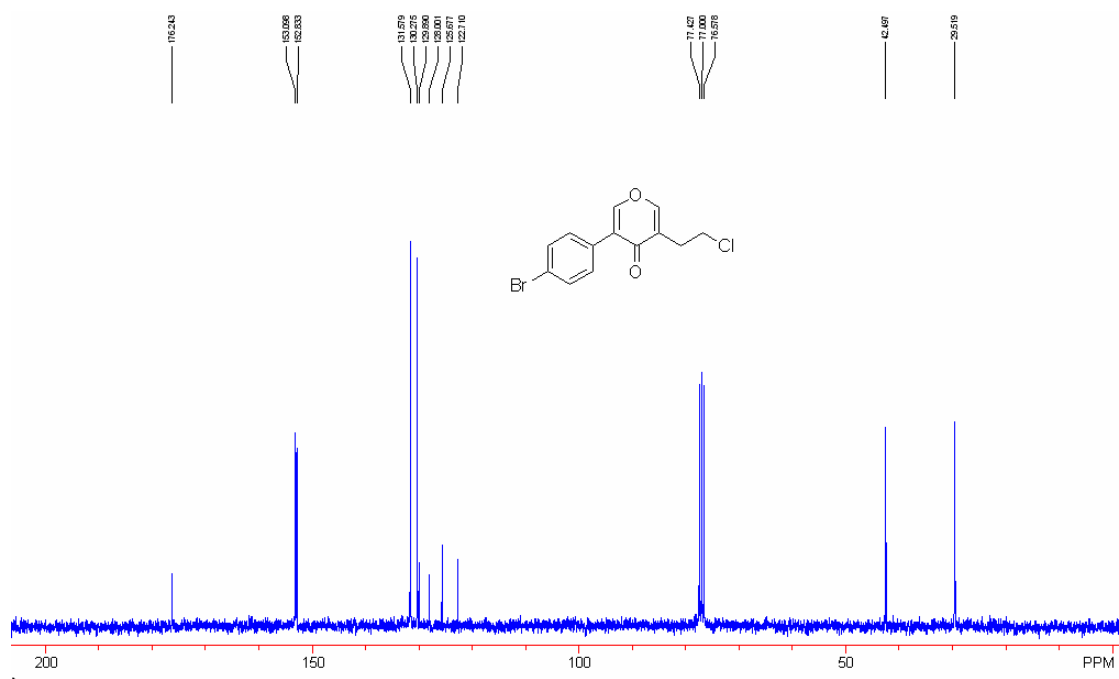
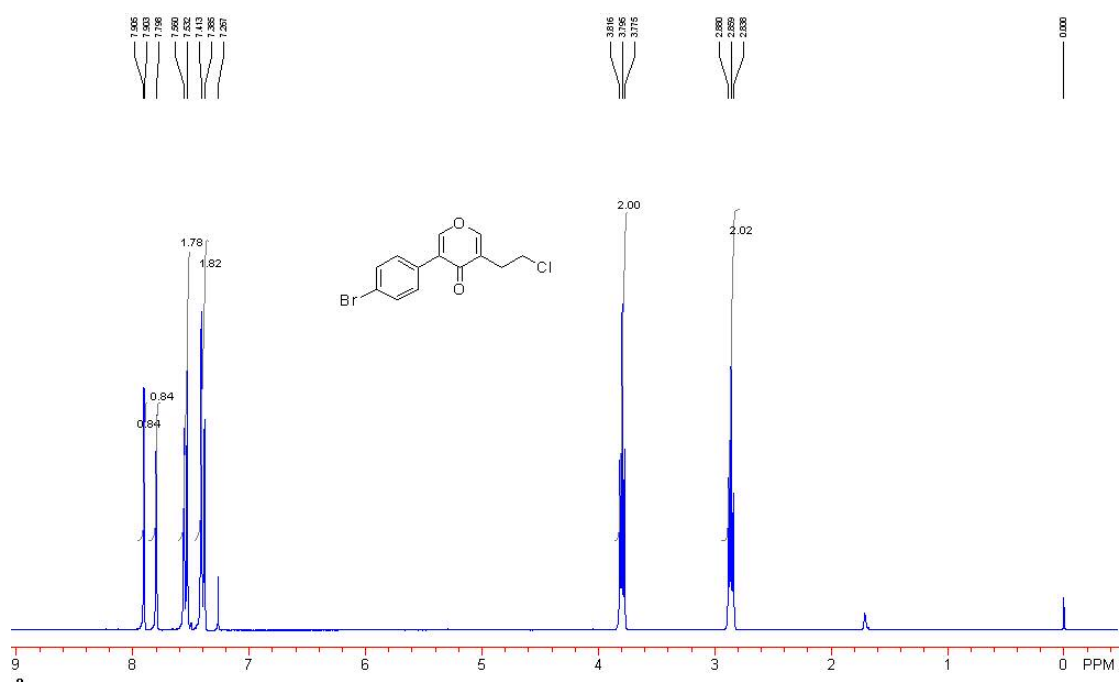


2,6-Dichloro-3-(2-chloroethyl)-1-naphthaldehyde (3b). A yellow solide. m.p. 97-99 °C. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.41 (t, $J = 6.9$ Hz, 2H, CH_2), 3.87 (t, $J = 6.9$ Hz, 2H, CH_2), 7.56 (dd, $J = 9.3$ Hz, $J = 2.4$ Hz, 1H, Ar), 7.80 (d, $J = 2.4$ Hz, 1H, Ar), 7.88 (s, 1H, Ar), 8.96 (d, $J = 9.3$ Hz, 1H, Ar), 10.89 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.7, 42.8, 126.4, 128.1, 128.7, 130.2, 132.6, 133.3, 134.6, 135.2, 140.4, 192.6; IR (CH_2Cl_2): ν 3045, 2927, 2856, 1674, 1582, 1484, 1433, 1349, 1158, 1060 cm^{-1} ; MS (EI) m/z (%): 285 [M^+] (19.1), 288 (62.6), 286 (64.2), 239 (67.0), 237 (100), 209 (34.4), 152 (38.7), 139 (31.2), 75 (24.0), 43 (32.6); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_9\text{OCl}_3$ (M^+) requires 285.9719, Found: 285.9713.



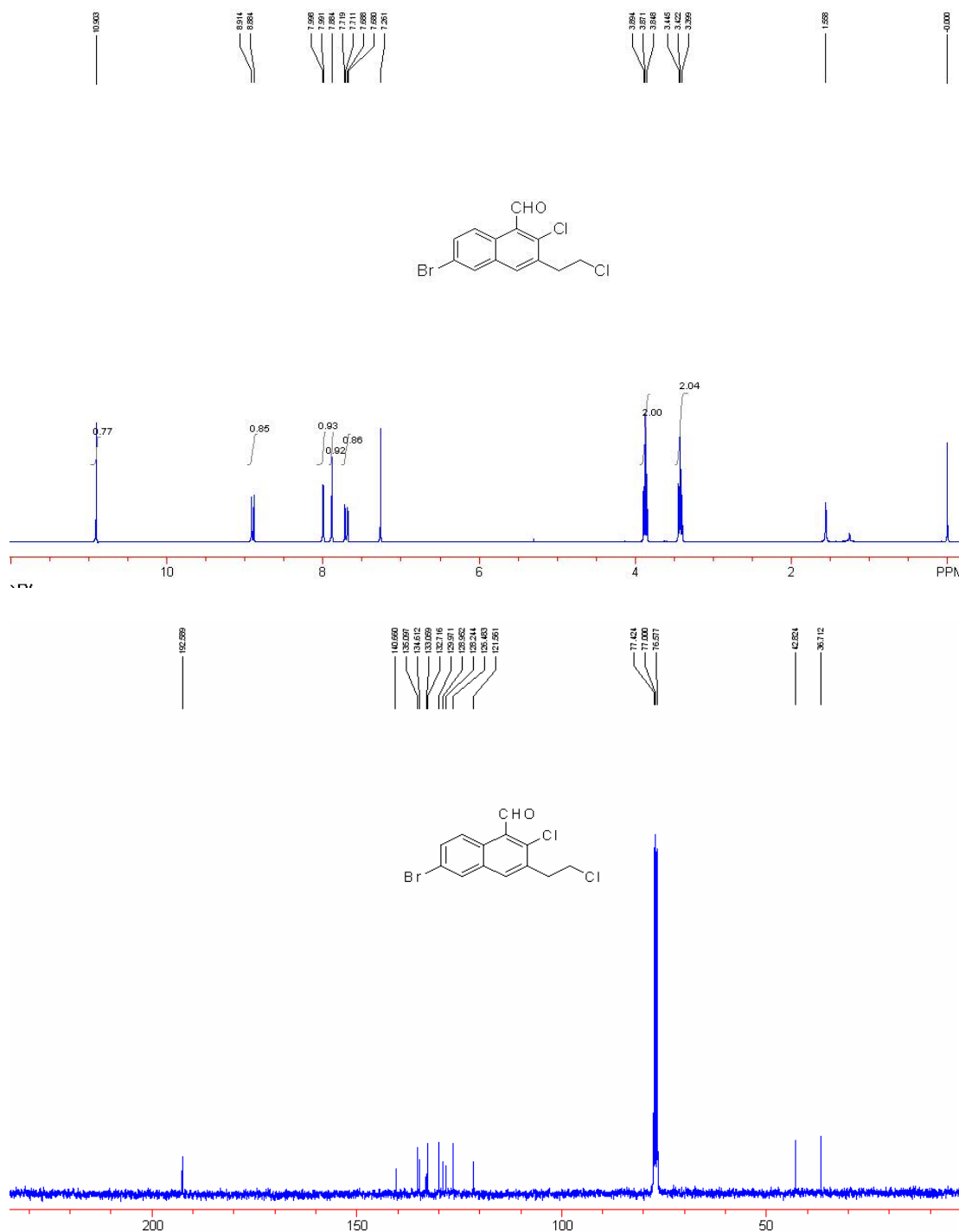


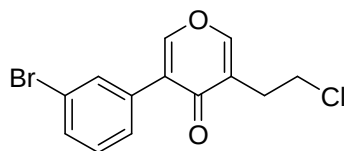
3-(4-Bromophenyl)-5-(2-chloroethyl)-4H-pyran-4-one (2c). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.86 (t, $J = 6.3$ Hz, 2H, CH_2), 3.80 (t, $J = 6.3$ Hz, 2H, CH_2), 7.40 (d, $J = 8.4$ Hz, 2H, Ar), 7.50 (d, $J = 8.4$ Hz, 2H, Ar), 7.80 (s, 1H), 7.90 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.5, 42.5, 122.7, 125.6, 128.0, 129.9, 130.3, 131.6, 152.8, 153.1, 176.3; IR (CH_2Cl_2): ν 3085, 2958, 2925, 1647, 1612, 1491, 1443, 1283, 1192, 1043 cm^{-1} ; MS (EI) m/z (%): 312 [M^+] (15.0), 277 (100), 279 (99.1), 199 (50.0), 197 (23.2), 101 (30.7), 89 (25.2), 75 (30.0), 53 (28.1); Anal. calcd. for $\text{C}_{13}\text{H}_{10}\text{BrClO}_2$: C, 49.79%; H, 3.21%. Found: C, 49.94%; H, 3.44%.



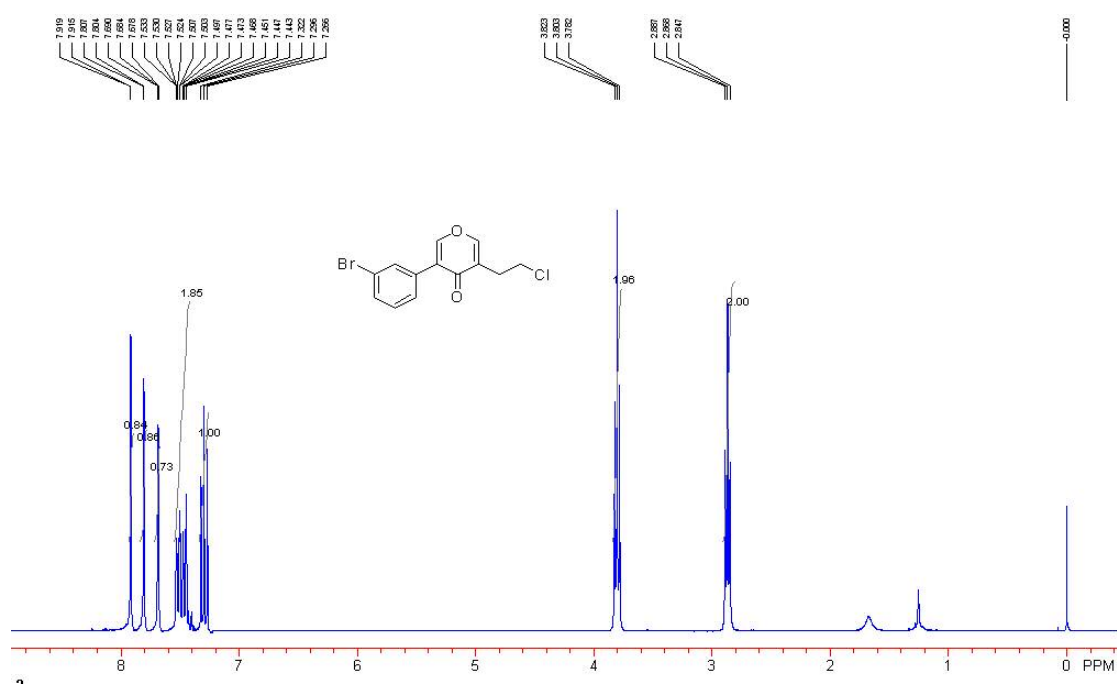
6-Bromo-2-chloro-3-(2-chloroethyl)-1-naphthaldehyde (3c). A yellow solid. m.p. 120-122 °C. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 3.42 (t, *J* = 6.9 Hz, 2H, CH₂), 3.87 (t, *J* = 6.9 Hz, 2H, CH₂), 7.70 (dd, *J* = 9.3 Hz, *J* = 2.4 Hz, 1H, Ar), 7.88 (s, 1H, Ar), 7.99 (d, *J* = 2.4 Hz, 1H, Ar),

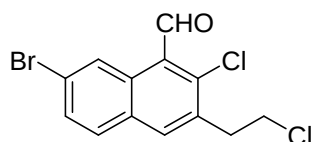
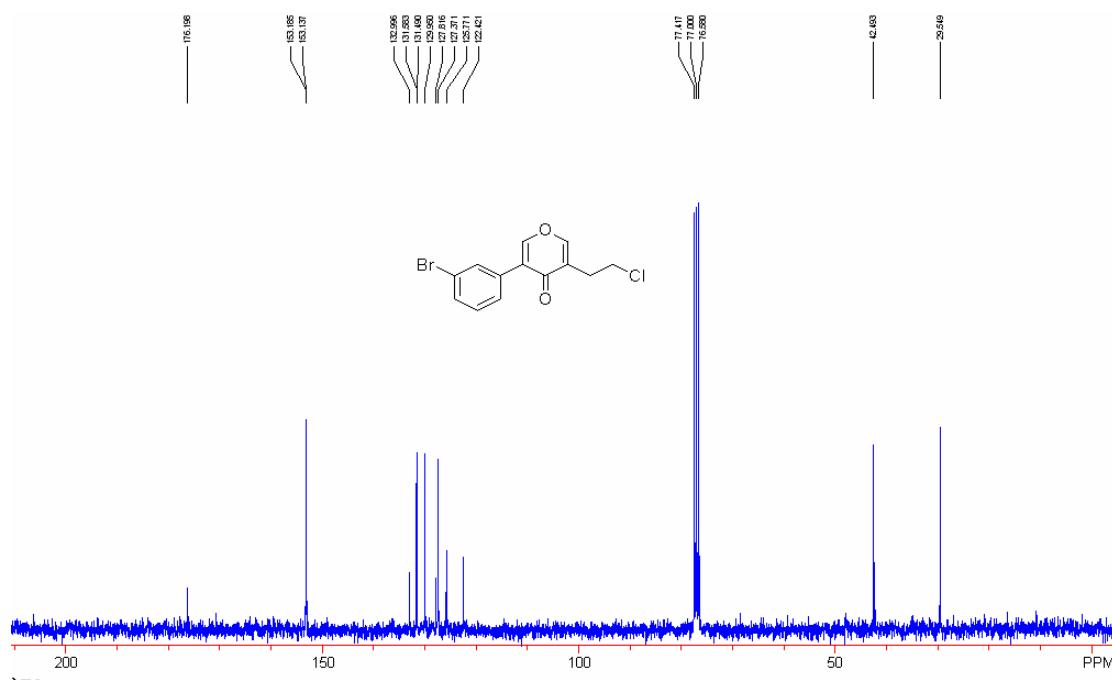
8.90 (d, $J = 9.3$ Hz, 1H, Ar), 10.90 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.7, 42.8, 121.6, 126.5, 128.2, 129.0, 130.0, 132.7, 133.1, 134.6, 135.1, 140.7, 192.6; IR (CH_2Cl_2): ν 3052, 2924, 1678, 1579, 1481, 1435, 1348, 1160, 1057 cm^{-1} ; MS (EI) m/z (%): 329 [M^+] (13.6), 334 (45.9), 332 (100), 330 (63.1), 283 (87.0), 281 (66.6), 253 (40.8), 152 (52.5), 139 (61.3); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_9\text{OCl}_2\text{Br}$ (M^+) requires 329.9214, Found: 329.9218.





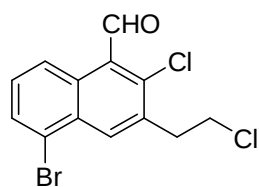
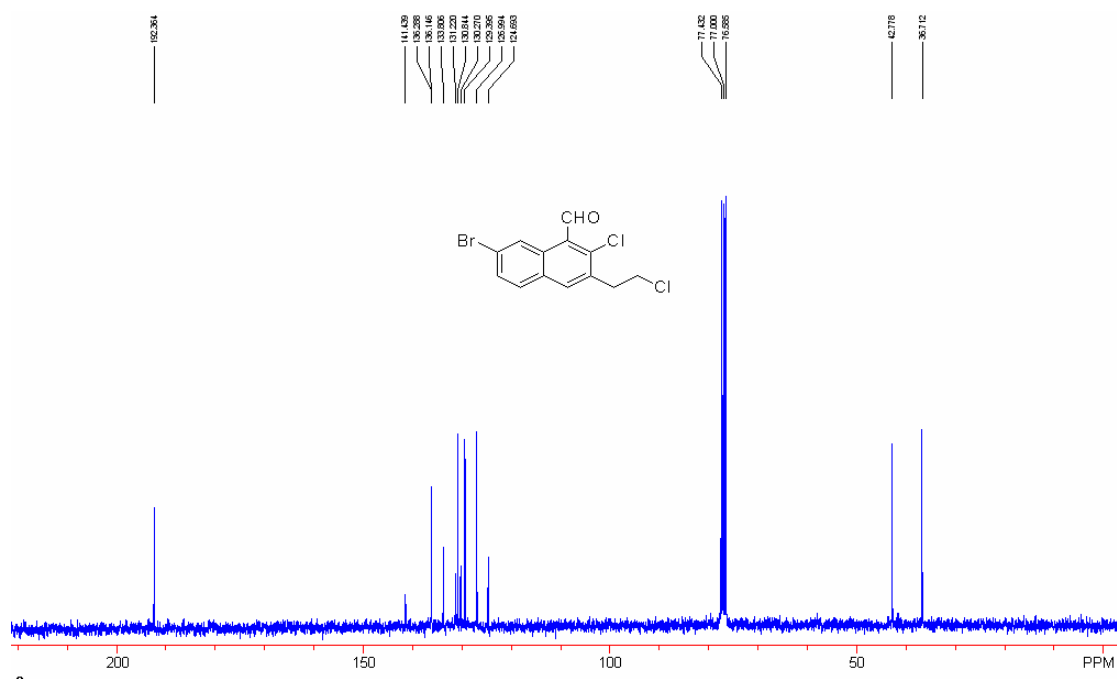
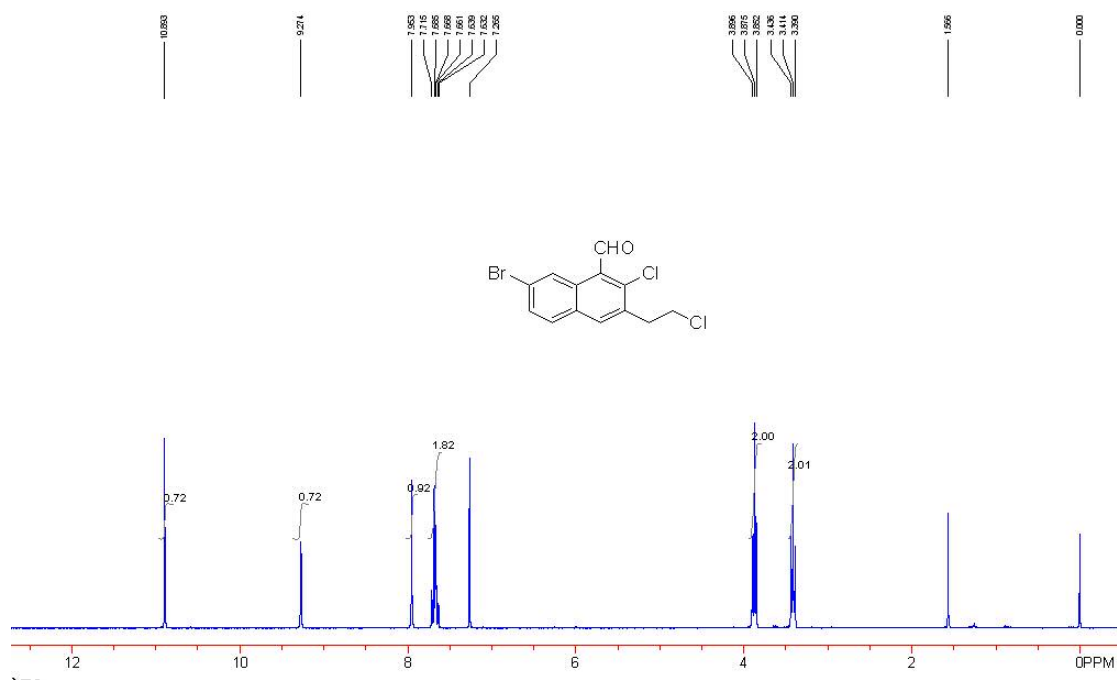
3-(3-Bromophenyl)-5-(2-chloroethyl)-4H-pyran-4-one (2d). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.87 (t, $J = 6.3$ Hz, 2H, CH_2), 3.80 (t, $J = 6.3$ Hz, 2H, CH_2), 7.27-7.32 (m, 1H, Ar), 7.44-7.53 (m, 2H, Ar), 7.68 (t, $J = 1.8$ Hz, 1H, Ar), 7.81 (d, $J = 0.9$ Hz, 1H), 7.92 (d, $J = 0.9$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.5, 42.5, 122.4, 125.8, 127.4, 127.8, 130.0, 131.5, 131.6, 133.0, 153.1, 153.2, 176.2; IR (CH_2Cl_2): ν 3081, 2963, 2924, 1649, 1619, 1476, 1425, 1282, 1192, 1046 cm^{-1} ; MS (EI) m/z (%): 312 [M^+] (14.3), 314 (17.8), 280 (14.6), 279 (84.1), 278 (13.9), 277 (100), 197 (18.1), 89 (15.3); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_{10}\text{BrClO}_2$ (M^+) requires 311.9553, Found: 311.9544.





(major)

7-Bromo-2-chloro-3-(2-chloroethyl)-1-naphthaldehyde (3d). A yellow solid. m.p. 86-88 °C. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 3.41 (t, *J* = 6.9 Hz, 2H, CH₂), 3.88 (t, *J* = 6.9 Hz, 2H, CH₂), 7.63-7.72 (m, 2H, Ar), 7.95 (s, 1H, Ar), 9.27 (s, 1H, Ar), 10.89 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 36.7, 42.8, 124.7, 127.0, 129.4, 130.3, 130.8, 131.2, 133.8, 136.2, 136.3, 141.4, 192.4; IR (CH₂Cl₂): ν 3109, 2958, 2925, 1675, 1580, 1481, 1433, 1346, 1135, 1067 cm⁻¹; MS (EI) *m/z* (%): 330 [M+1⁺] (53.1), 334 (37.2), 333 (35.7), 332 (70.3), 285 (22.3), 283 (100), 253 (27.5), 281 (69.0); HRMS (EI) Calcd. for C₁₃H₉OCl₂Br (M⁺) requires 329.9214, Found: 329.9224.

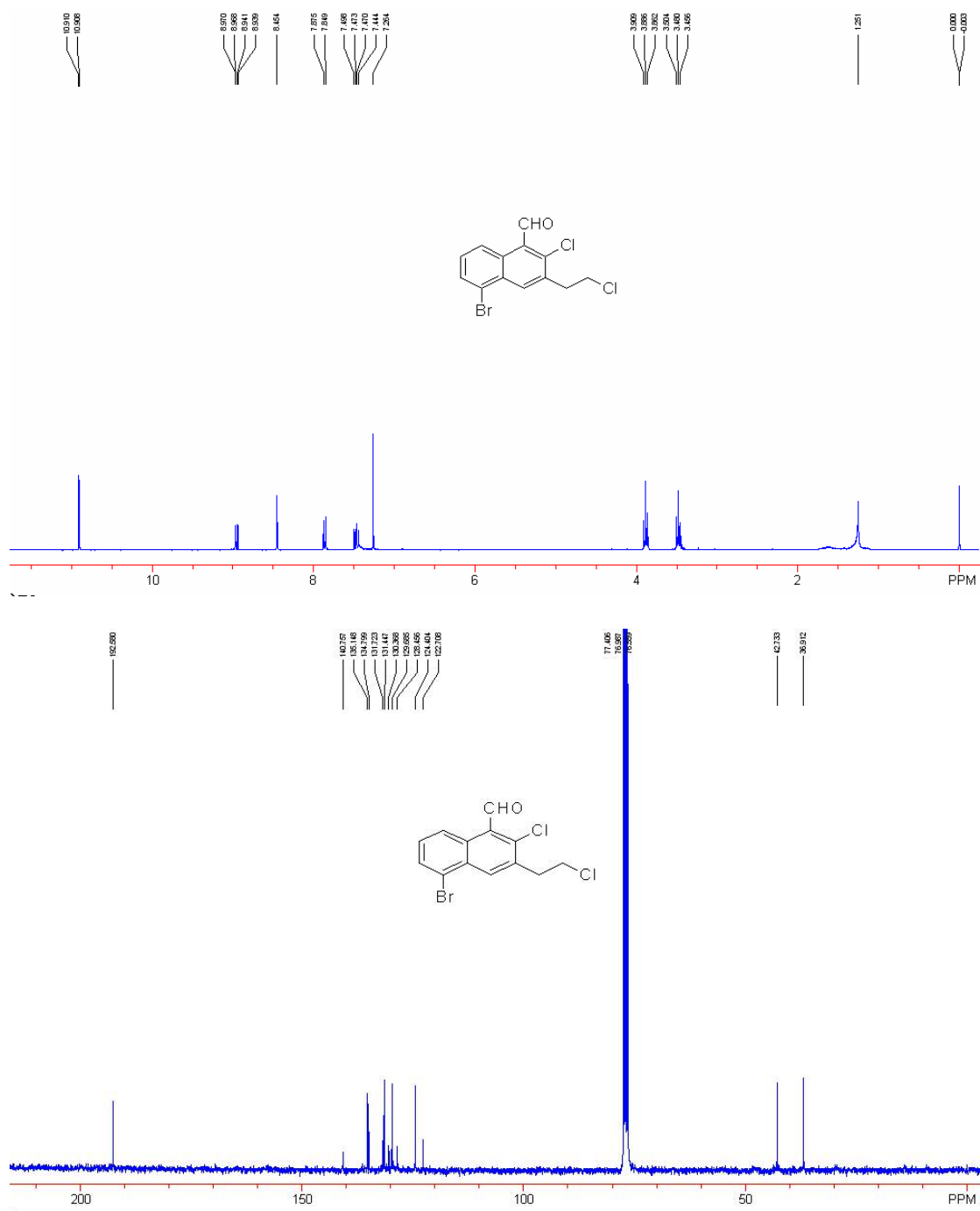


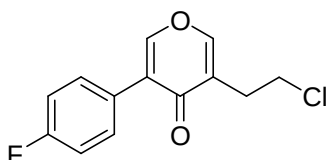
(minor)

5-Bromo-2-chloro-3-(2-chloroethyl)-1-naphthaldehyde (3d'). A yellow solid. m.p. 93-95 °C.

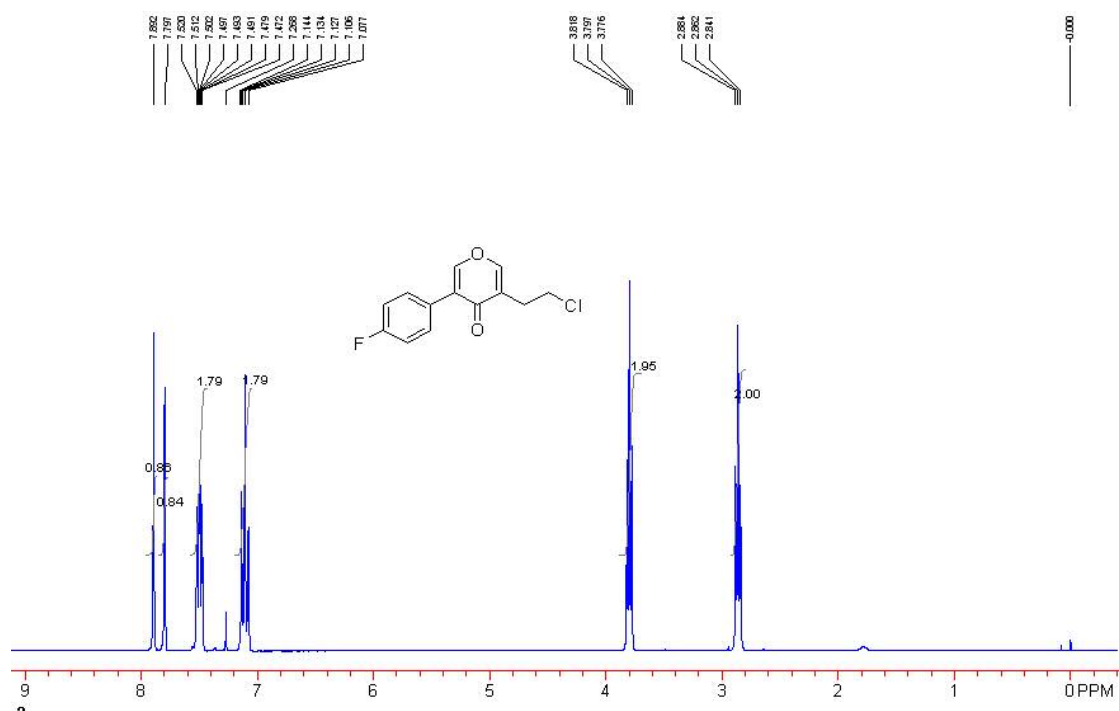
^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.48 (t, J = 6.9 Hz, 2H, CH_2), 3.89 (t, J = 6.9 Hz, 2H,

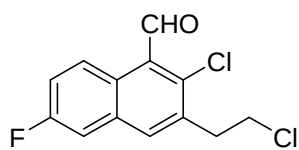
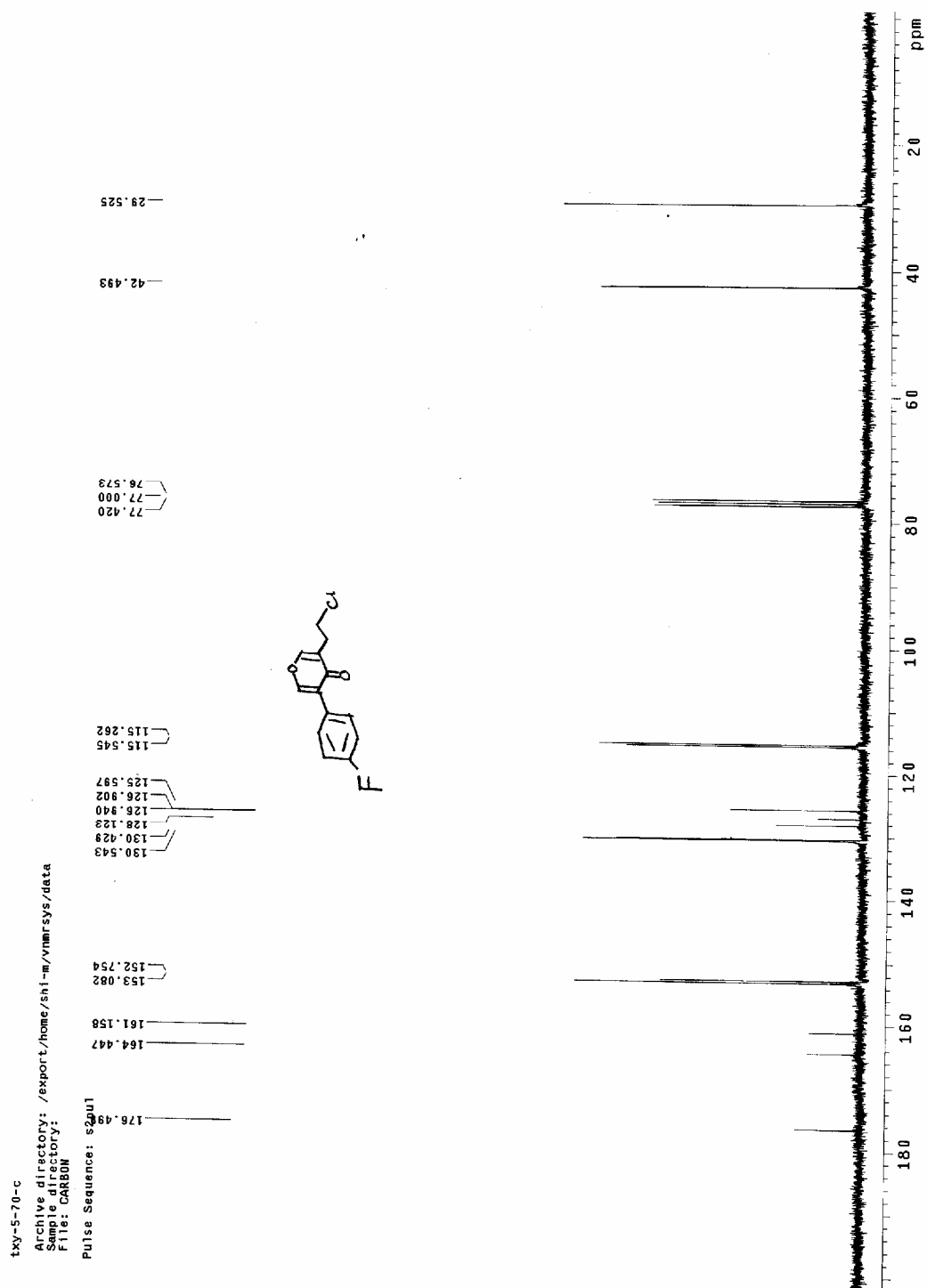
CH₂), 7.47 (dd, *J* = 8.4 Hz, *J* = 7.8 Hz, 1H, Ar), 7.86 (d, *J* = 7.8 Hz, 1H, Ar), 8.45 (s, 1H, Ar), 8.96 (dd, *J* = 8.4 Hz, *J* = 0.6 Hz, 1H, Ar), 10.89 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 36.9, 42.8, 122.7, 124.4, 128.5, 129.7, 130.4, 131.5, 131.7, 134.8, 135.2, 140.8, 192.6; IR (CH₂Cl₂): ν 3077, 2949, 2929, 1686, 1571, 1476, 1421, 1341, 1159, 1078 cm⁻¹; MS (EI) *m/z* (%): 330 [M⁺] (65.2), 332 (85.7), 283 (83.3), 281 (62.3), 253 (63.5), 252 (62.1), 251 (64.2), 105 (100); HRMS (EI) Calcd. for C₁₃H₉OCl₂Br (M⁺) requires 329.9214, Found: 329.9215.





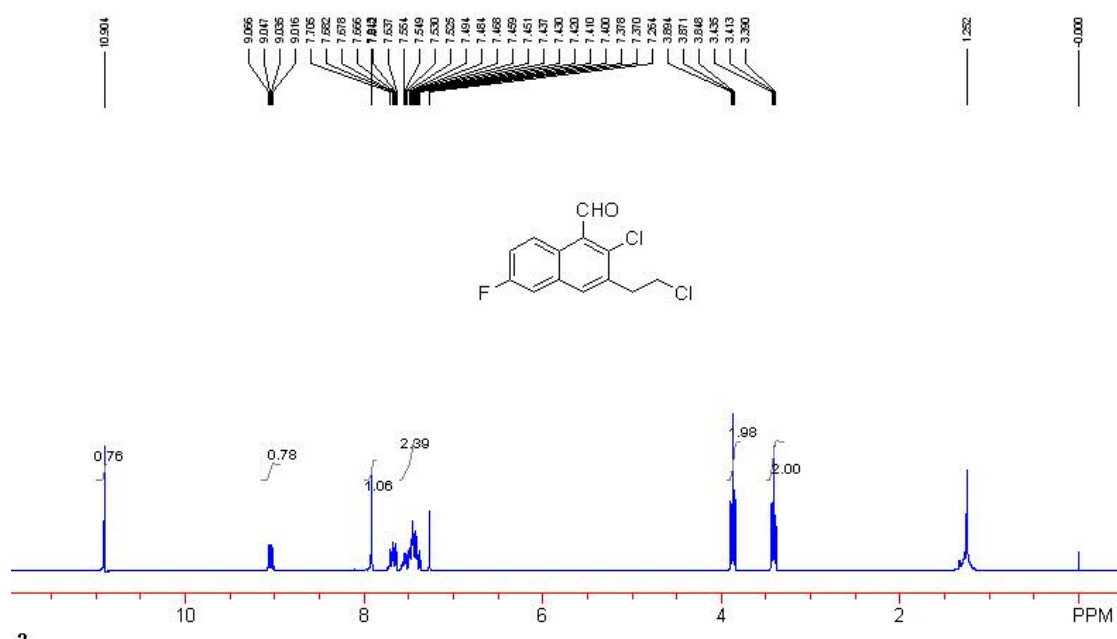
3-(2-Chloroethyl)-5-(4-fluorophenyl)-4H-pyran-4-one (2e). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.86 (t, $J = 6.3$ Hz, 2H, CH_2), 3.80 (t, $J = 6.3$ Hz, 2H, CH_2), 7.11 (t, $J = 8.4$ Hz, 2H, Ar), 7.47-7.52 (m, 2H, Ar), 7.80 (s, 1H), 7.89 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.5, 42.5, 115.4 (d, $J_{\text{C-F}} = 20.6$ Hz), 125.6, 126.9 (d, $J = 2.9$ Hz), 128.1, 130.5 (d, $J = 8.6$ Hz), 152.8, 153.1, 162.8 (d, $J = 246.7$ Hz), 176.5; IR (CH_2Cl_2): ν 3080, 2964, 2924, 1794, 1655, 1618, 1509, 1425, 1286, 1190, 1091 cm^{-1} ; MS (EI) m/z (%): 252 [M^+] (15.3), 218 (13.6), 217 (100), 137 (8.2), 120 (22.7), 108 (9.7), 107 (11.4), 53 (13.2); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_{10}\text{ClFO}_2$ requires 252.0353, Found: 252.0346.

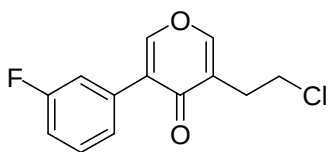
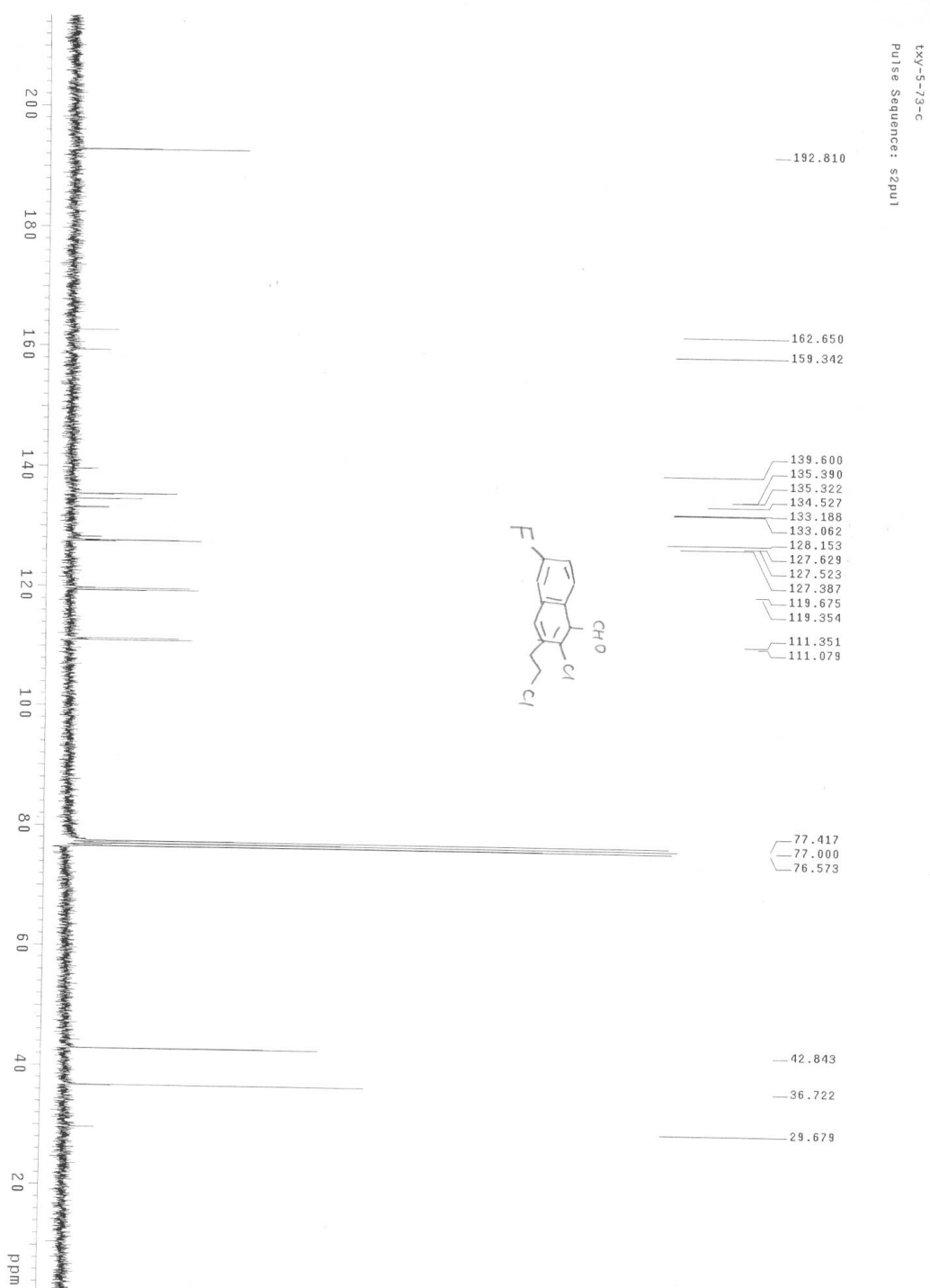




2-Chloro-3-(2-chloroethyl)-6-fluoro-1-naphthaldehyde (3e). A yellow solid. m.p. 90-92 °C.

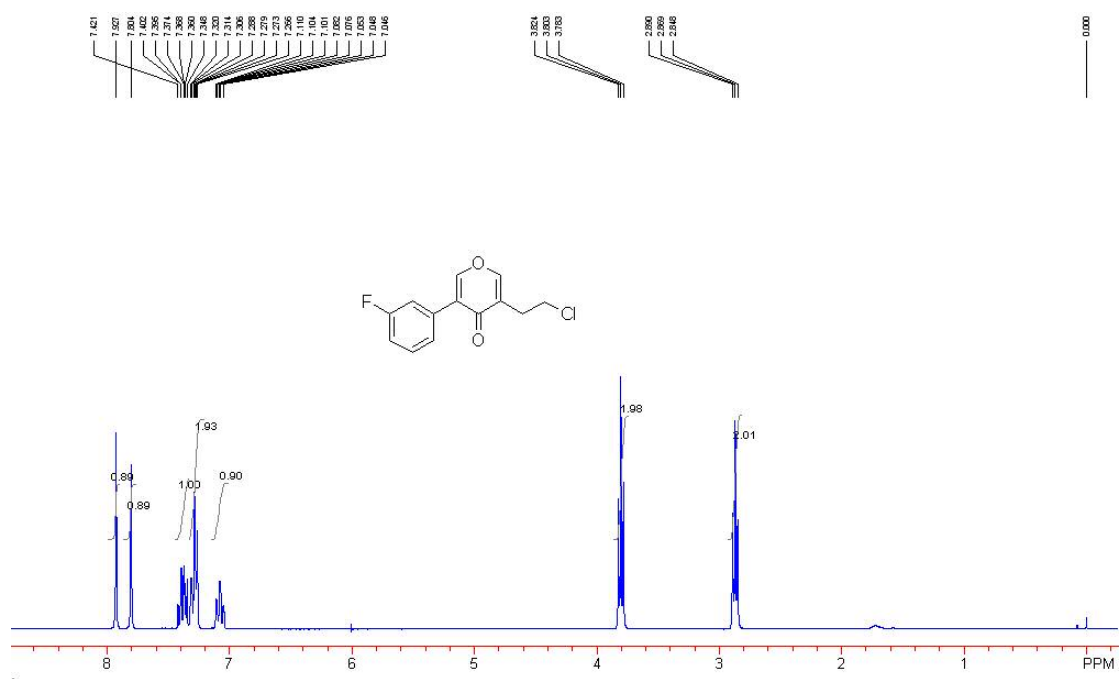
^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.41 (t, $J = 6.9$ Hz, 2H, CH_2), 3.87 (t, $J = 6.9$ Hz, 2H, CH_2), 7.33-7.71 (m, 2H, Ar), 7.91 (s, 1H, Ar), 9.04 (dd, $J = 9.3$ Hz, $J = 5.7$ Hz, 1H, Ar), 10.90 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.7, 42.8, 111.2 (d, $J_{\text{C-F}} = 20.4$ Hz), 119.5 (d, $J_{\text{C-F}} = 24.1$ Hz), 127.4, 127.6 (d, $J_{\text{C-F}} = 8.0$ Hz), 128.2, 133.2 (d, $J_{\text{C-F}} = 9.5$ Hz), 134.5, 135.4 (d, $J_{\text{C-F}} = 5.1$ Hz), 139.6, 161.0 (d, $J_{\text{C-F}} = 248$ Hz), 192.8; IR (CH_2Cl_2): ν 3043, 2920, 2845, 1686, 1561, 1500, 1492, 1344, 1165, 1069 cm^{-1} ; MS (EI) m/z (%): 270 [M^+] (56.9), 269 (58.4), 221 (68.8), 193 (80.6), 170 (100), 157 (76.8), 51 (59.9), 49 (99.9); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_9\text{Cl}_2\text{FO}$ requires 270.0014, Found: 270.0018.

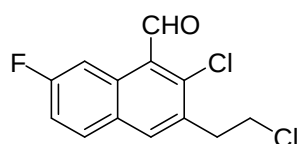
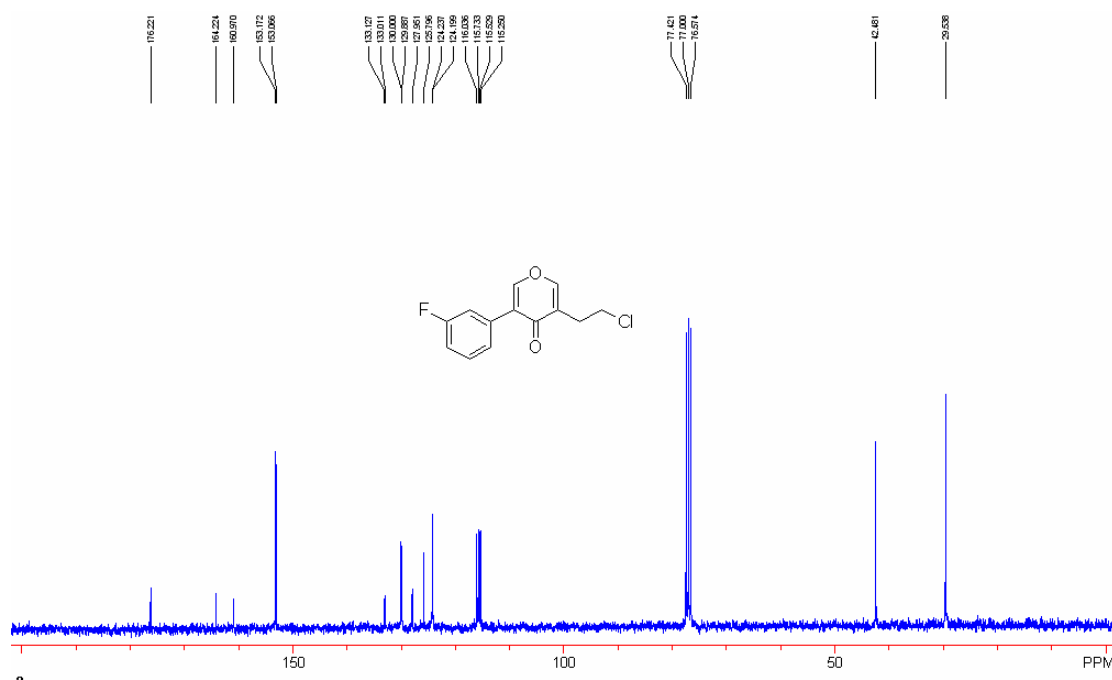




3-(2-Chloroethyl)-5-(3-fluorophenyl)-4H-pyran-4-one (2f). A yellow oil. ¹H NMR (CDCl₃,

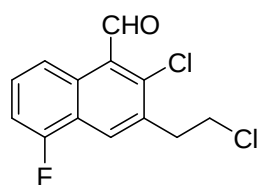
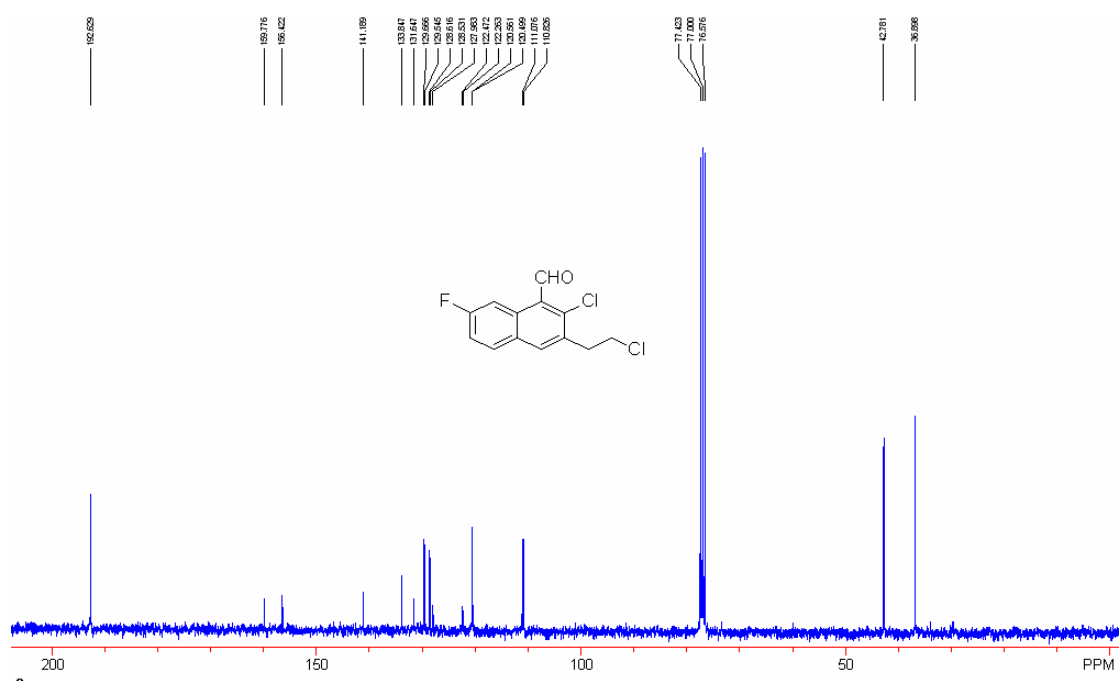
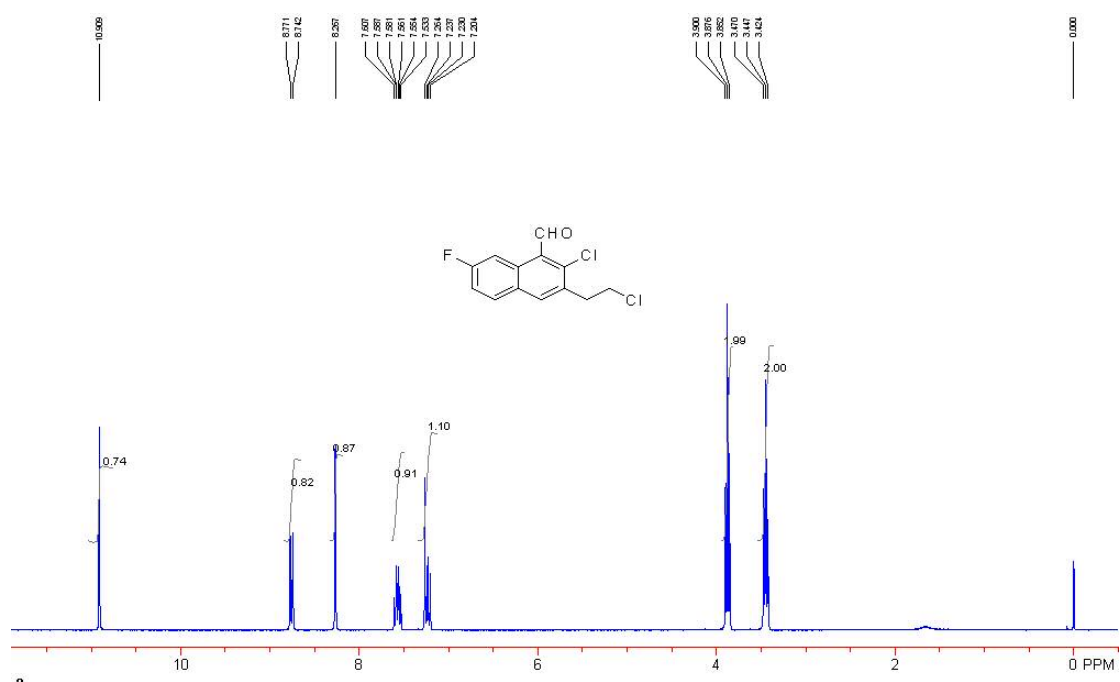
300 MHz, TMS) δ 2.87 (t, $J = 6.3$ Hz, 2H, CH₂), 3.80 (t, $J = 6.3$ Hz, 2H, CH₂), 7.08 (td, $J = 8.7$ Hz, $J = 1.8$ Hz, 1H, Ar), 7.27-7.32 (m, 2H, Ar), 7.35-7.42 (m, 1H, Ar), 7.80 (s, 1H), 7.93 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 29.5, 42.5, 115.4 (d, $J_{C-F} = 21.2$ Hz), 115.9 (d, $J_{C-F} = 22.9$ Hz), 124.2 (d, $J_{C-F} = 2.9$ Hz), 125.8, 128.0, 129.9 (d, $J = 8.0$ Hz), 133.1 (d, $J = 8.6$ Hz), 153.1, 153.2, 162.6 (d, $J = 243.9$ Hz), 176.2; IR (CH₂Cl₂): ν 3080, 2964, 2924, 1794, 1655, 1618, 1509, 1425, 1286, 1190, 1091 cm⁻¹; MS (EI) m/z (%): 252 [M⁺] (14.1), 218 (14.2), 217 (100), 137 (6.7), 133 (6.4), 110 (9.5), 107 (8.6), 53 (8.4); HRMS (EI) Calcd. for C₁₃H₁₀ClFO₂ requires 252.0353, Found: 252.0349.





(major)

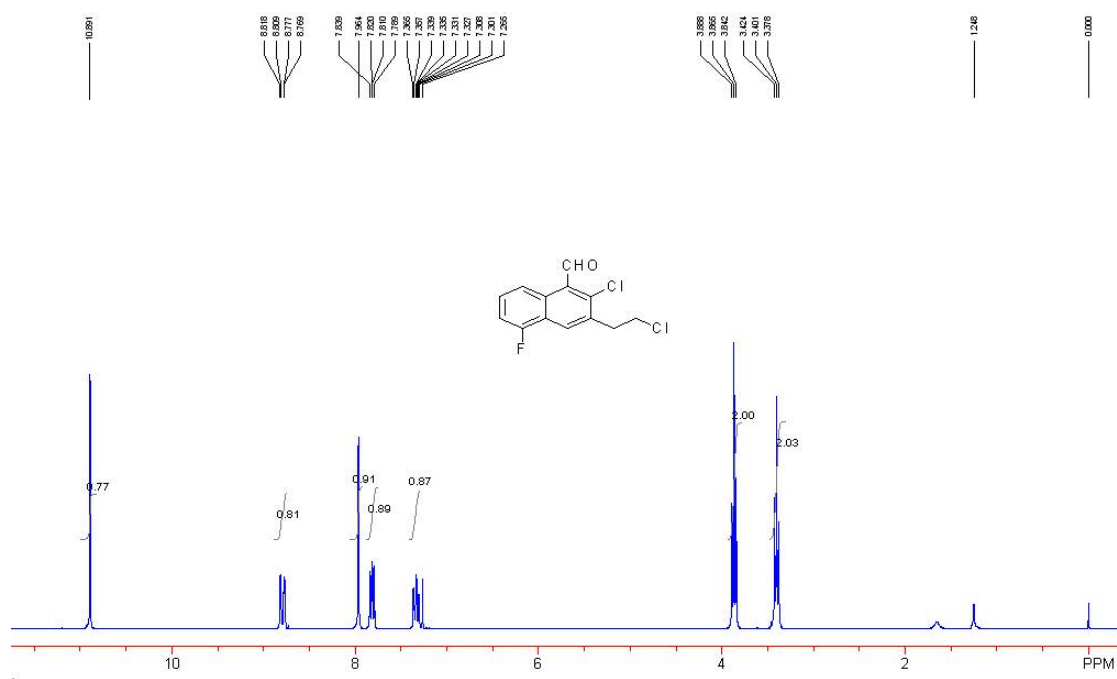
2-Chloro-3-(2-chloroethyl)-5-fluoro-1-naphthaldehyde (3f). A white solid. m.p. 108-110 °C. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 3.45 (t, $J = 6.9$ Hz, 2H, CH_2), 3.88 (t, $J = 6.9$ Hz, 2H, CH_2), 7.23 (td, $J = 9.0$ Hz, $J = 2.1$ Hz, 1H, Ar), 7.57 (qd, $J = 7.2$ Hz, $J = 2.1$ Hz, 1H, Ar), 8.27 (s, 1H, Ar), 8.76 (d, $J = 9.0$ Hz, 1H, Ar), 10.91 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.7, 42.8, 111.0 (d, $J_{\text{C-F}} = 18.8$ Hz), 120.5, 120.6, 122.3 (d, $J_{\text{C-F}} = 15.5$ Hz), 128.6 (d, $J_{\text{C-F}} = 6.3$ Hz), 129.6 (d, $J_{\text{C-F}} = 8.6$ Hz), 131.7, 133.8 (d, $J_{\text{C-F}} = 1.7$ Hz), 141.2, 158.1 (d, $J_{\text{C-F}} = 251.8$ Hz), 192.7; IR (CH_2Cl_2): ν 3070, 2926, 2865, 1693, 1592, 1486, 1450, 1393, 1152, 983 cm^{-1} ; MS (EI) m/z (%): 270 [M^+] (69.8), 272 (41.7), 271 (39.1), 242 (32.8), 223 (36.2), 221 (100), 193 (54.7), 170 (33.8); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_9\text{OCl}_2\text{F}$ (M^+) requires 270.0014, Found: 270.0026.

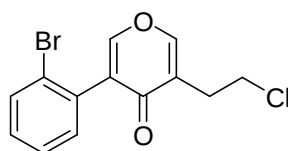
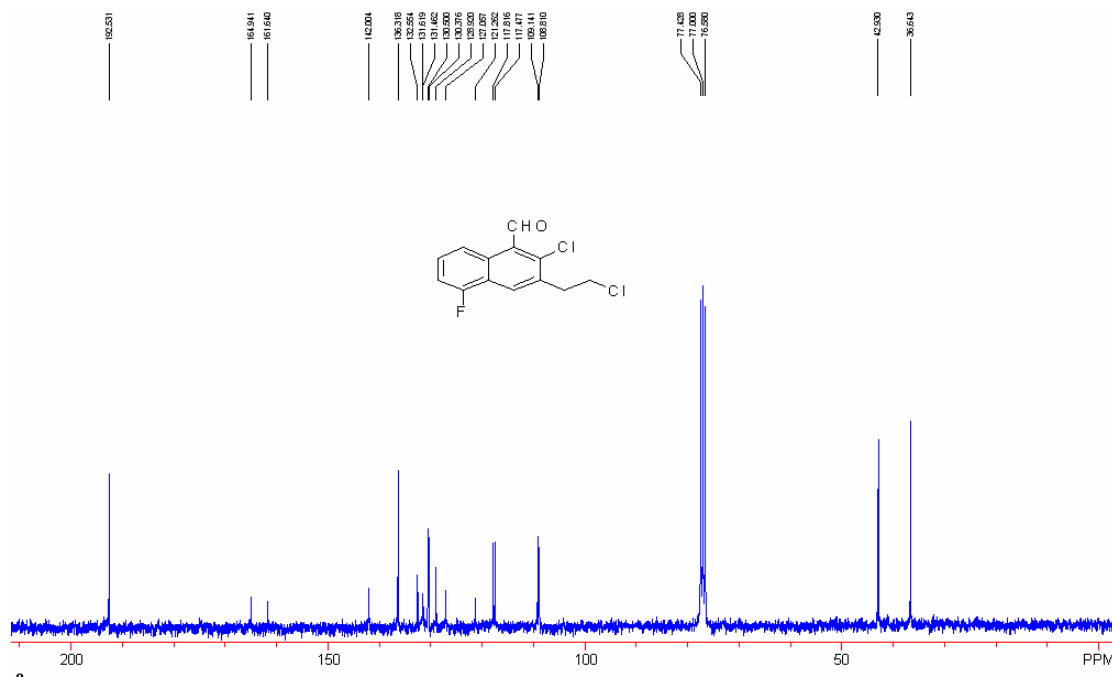


(minor)

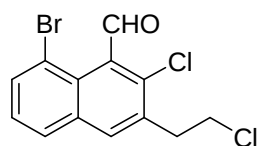
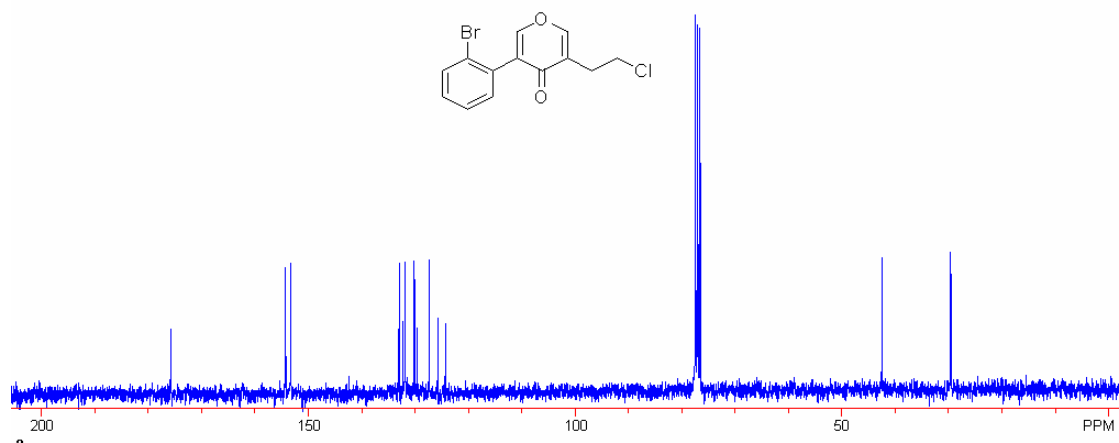
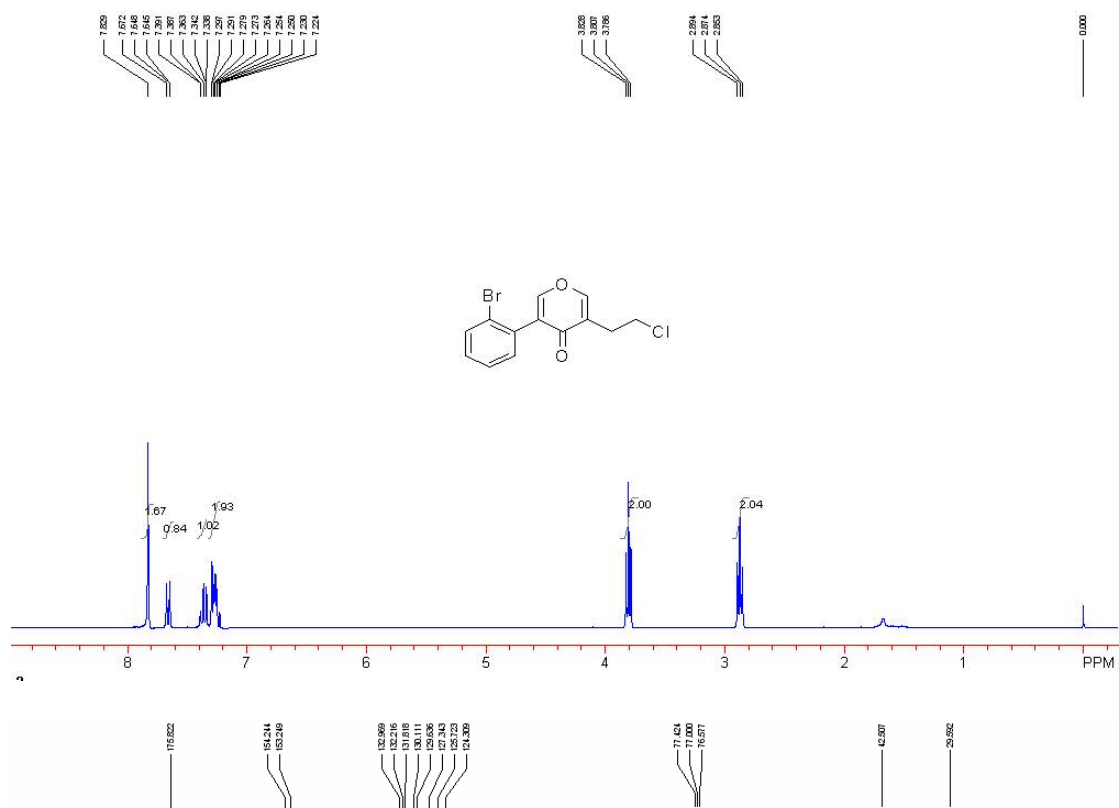
2-Chloro-3-(2-chloroethyl)-7-fluoro-1-naphthaldehyde (3f'). A white solid. m.p. 100-102 °C. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 3.40 (t, *J* = 6.9 Hz, 2H, CH₂), 3.87 (t, *J* = 6.9 Hz, 2H,

CH₂), 7.33 (ddd, $J = 9.0$ Hz, $J = 7.8$ Hz, $J = 2.4$ Hz, 1H, Ar), 7.82 (dd, $J = 9.0$ Hz, $J = 6.9$ Hz, 1H, Ar), 7.96 (s, 1H, Ar), 8.79 (dd, $J = 12.0$ Hz, $J = 2.4$ Hz, 1H, Ar), 10.89 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 36.6, 42.9, 109.0 (d, $J_{C-F} = 25.1$ Hz), 117.7 (d, $J_{C-F} = 25.2$ Hz), 127.1 (d, $J_{C-F} = 5.1$ Hz), 128.9, 130.4 (d, $J_{C-F} = 9.6$ Hz), 131.5 (d, $J_{C-F} = 11.4$ Hz), 132.6, 136.3, 142.0, 163.3 (d, $J_{C-F} = 247.9$ Hz), 192.5; IR (CH₂Cl₂): ν 3097, 3056, 2928, 1681, 1594, 1495, 1441, 1350, 1172, 943 cm⁻¹; MS (EI) m/z (%): 270 [M⁺] (37.8), 272 (23.2), 223 (38.1), 222 (14.9), 221 (100), 193 (22.3), 170 (25.2), 157 (33.7); HRMS (EI) Calcd. for C₁₃H₉OCl₂F (M⁺) requires 270.0014, Found: 270.0004.

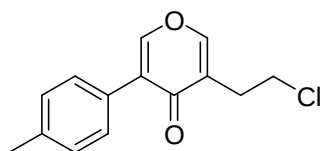




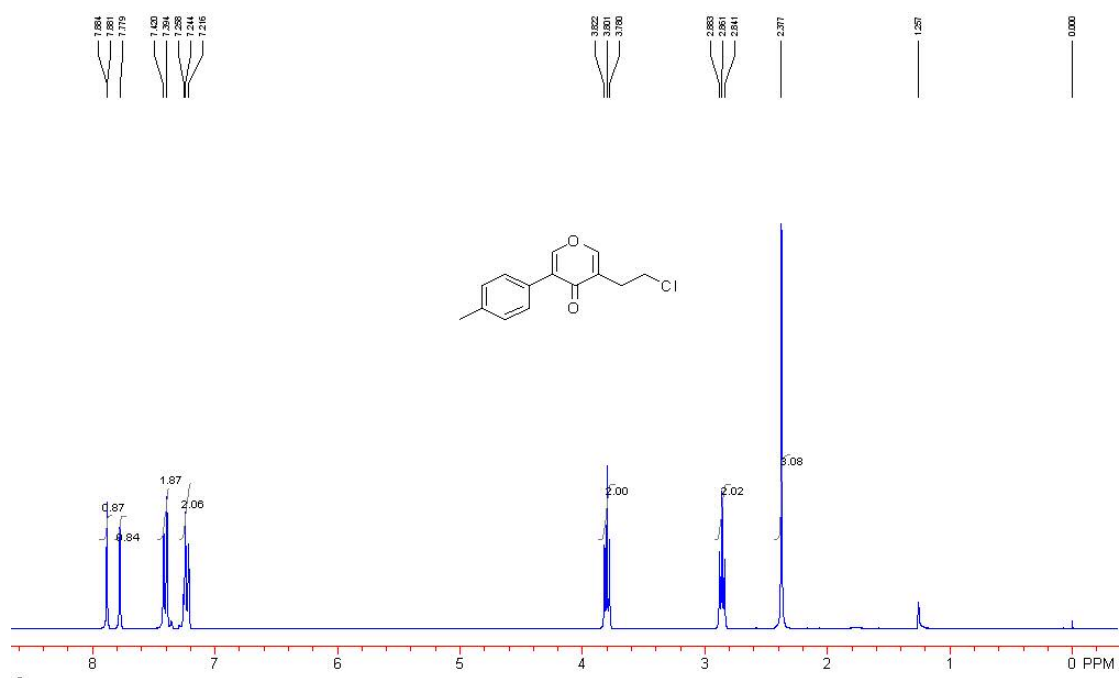
3-(2-Bromophenyl)-5-(2-chloroethyl)-4H-pyran-4-one (2g). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.88 (t, $J = 6.3$ Hz, 2H, CH_2), 3.81 (t, $J = 6.3$ Hz, 2H, CH_2), 7.23-7.30 (m, 2H, Ar), 7.35-7.40 (m, 1H, Ar), 7.66 (d, $J = 7.8$ Hz, 1H, Ar), 7.84 (s, 2H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.6, 42.5, 124.3, 125.7, 127.4, 129.6, 130.1, 131.8, 132.2, 133.0, 153.3, 154.3, 175.8; IR (CH_2Cl_2): ν 3076, 2924, 2853, 1652, 1622, 1471, 1418, 1297, 1192, 1057 cm^{-1} ; MS (EI) m/z (%): 277 [M^+] (2.6), 235 (35.6), 234 (16.4), 233 (100), 197 (17.2), 184 (13.0), 101 (6.6), 89 (5.6), 75 (4.6); HRMS (EI) Calcd. for $\text{C}_{13}\text{H}_{10}\text{BrO}_2$ (M^+) requires 276.9864, Found: 274.9867.

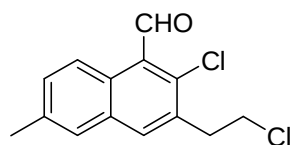
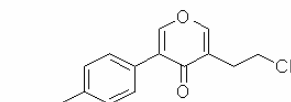


8-Bromo-2-chloro-3-(2-chloroethyl)-1-naphthaldehyde (3g). Trace of this compound was obtained. We cannot get its spectroscopic data.

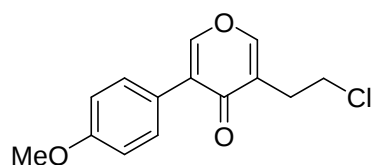
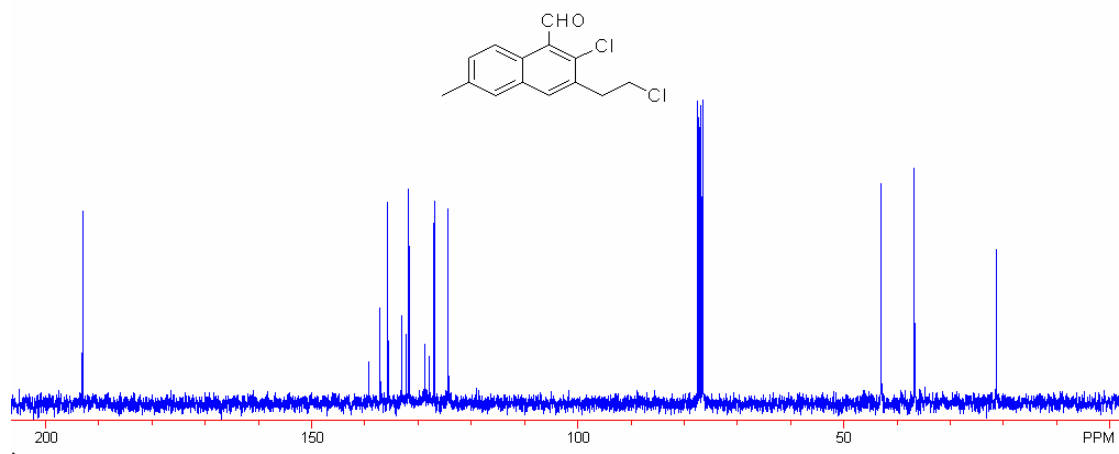
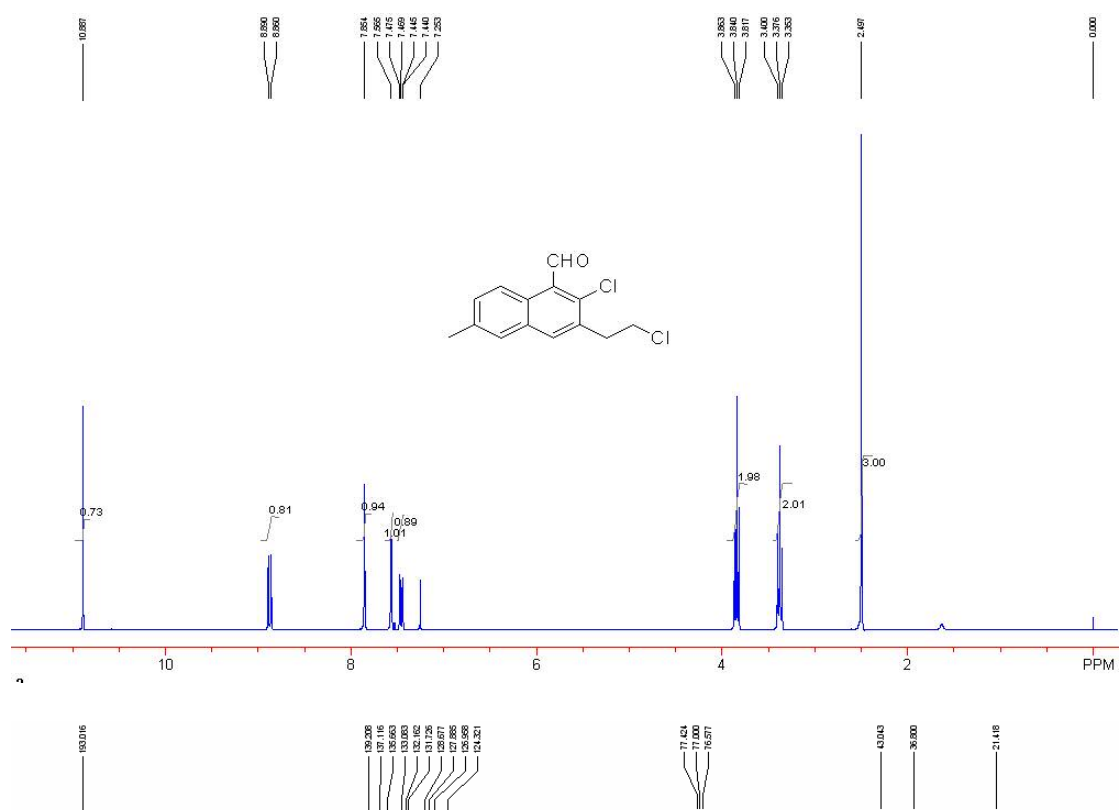


3-(2-Chloroethyl)-5-*p*-tolyl-4*H*-pyran-4-one (2h). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.38 (s, 3H, CH_3), 2.86 (t, $J = 6.3$ Hz, 2H, CH_2), 3.80 (t, $J = 6.3$ Hz, 2H, CH_2), 7.23 (d, $J = 8.4$ Hz, 2H, Ar), 7.41 (d, $J = 8.4$ Hz, 2H, Ar), 7.78 (s, 1H), 7.88 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 21.2, 29.6, 42.6, 125.5, 128.0, 128.6, 129.0, 129.1, 138.4, 152.6, 152.9, 176.7; IR (CH_2Cl_2): ν 3030, 2959, 2868, 1647, 1600, 1493, 1429, 1287, 1190, 1045 cm^{-1} ; MS (EI) m/z (%): 248 [M^+] (18.8), 250 (7.1), 214 (17.2), 213 (100), 115 (17.9), 97 (7.5), 71 (7.1), 57 (9.3); Anal. calcd. for $\text{C}_{14}\text{H}_{13}\text{ClO}_2$: C, 67.61%; H, 5.27%. Found: C, 67.54%; H, 5.15%.



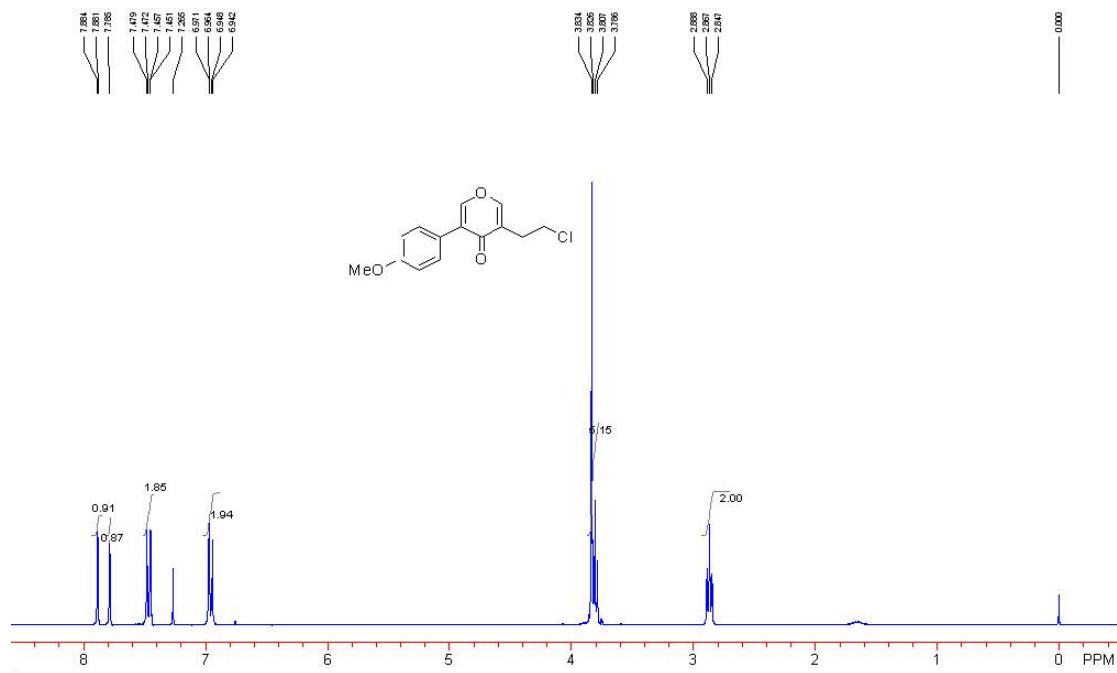


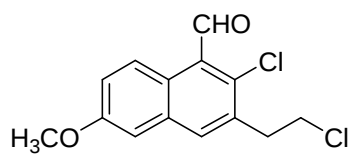
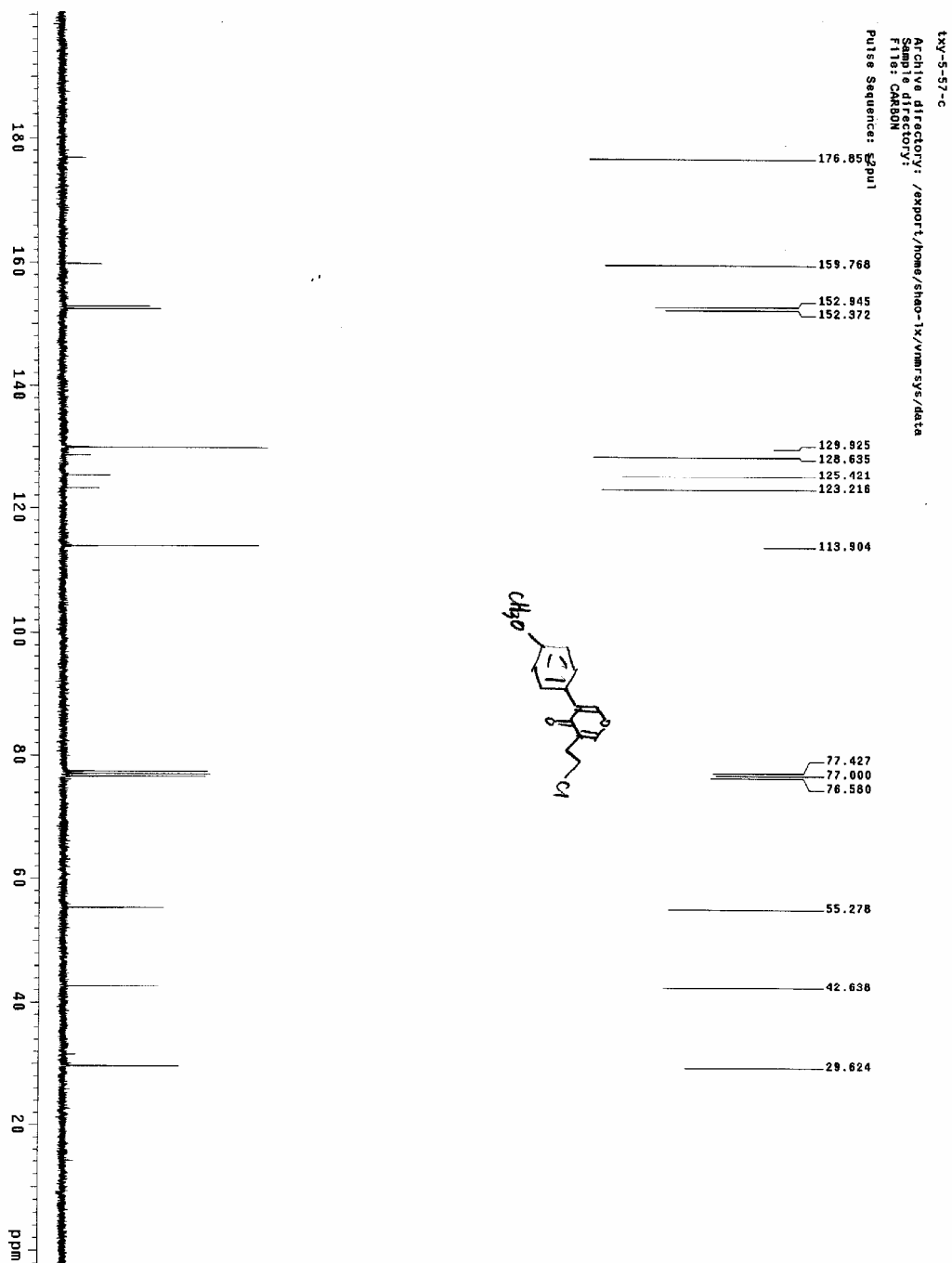
S25



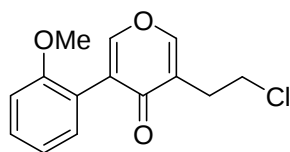
3-(2-Chloroethyl)-5-(4-methoxyphenyl)-4H-pyran-4-one (2i). A yellow oil. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.87 (t, *J* = 6.3 Hz, 2H, CH₂), 3.81 (t, *J* = 6.3 Hz, 2H, CH₂), 3.83 (s, 3H, CH₃), 6.95 (dd, *J* = 6.6 Hz, *J* = 1.8 Hz, 2H, Ar), 7.46 (dd, *J* = 6.6 Hz, *J* = 1.8 Hz, 2H,

Ar), 7.79 (s, 1H), 7.88 (s, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.6, 42.6, 55.3, 113.9, 123.2, 125.4, 128.6, 129.9, 152.4, 152.9, 159.8, 176.9; IR (CH_2Cl_2): ν 3078, 3003, 2960, 2937, 1774, 1647, 1608, 1512, 1464, 1295, 1178, 1092 cm^{-1} ; MS (EI) m/z (%): 264 [M^+] (35.6), 266 (11.6), 230 (13.0), 229 (100), 132 (12.0), 117 (11.0), 89 (21.0), 51 (11.2); HRMS (EI) Calcd. for $\text{C}_{14}\text{H}_{13}\text{O}_3$ ($\text{M}^+ - \text{Cl}$) requires 229.0865, Found: 229.0861.

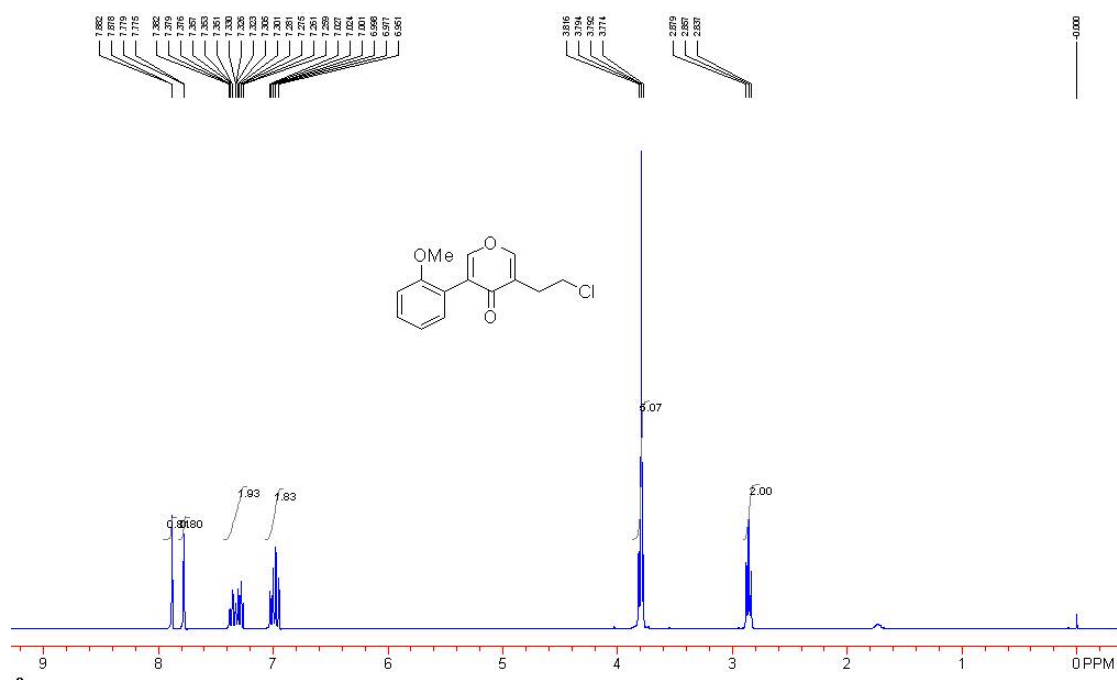


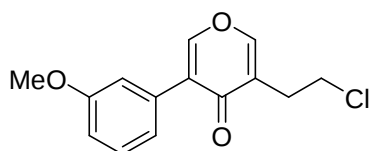
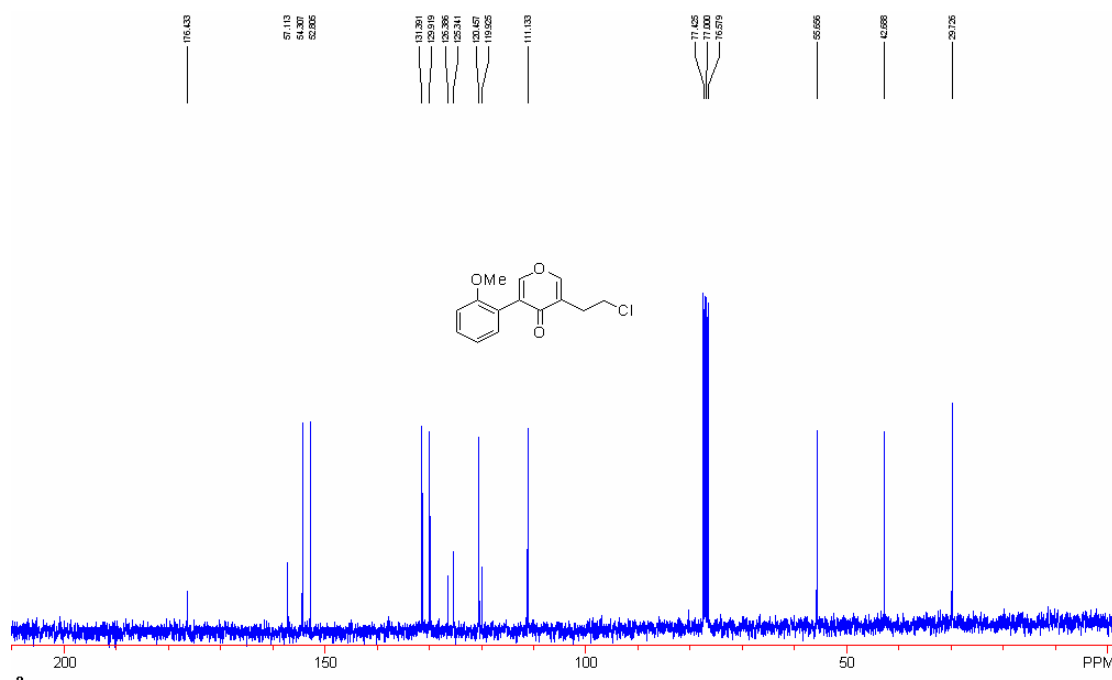


2-Chloro-3-(2-chloroethyl)-6-methoxy-1-naphthaldehyde (3i). Trace of this compound was obtained. We cannot get its spectroscopic data.

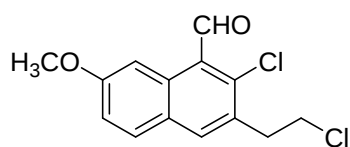
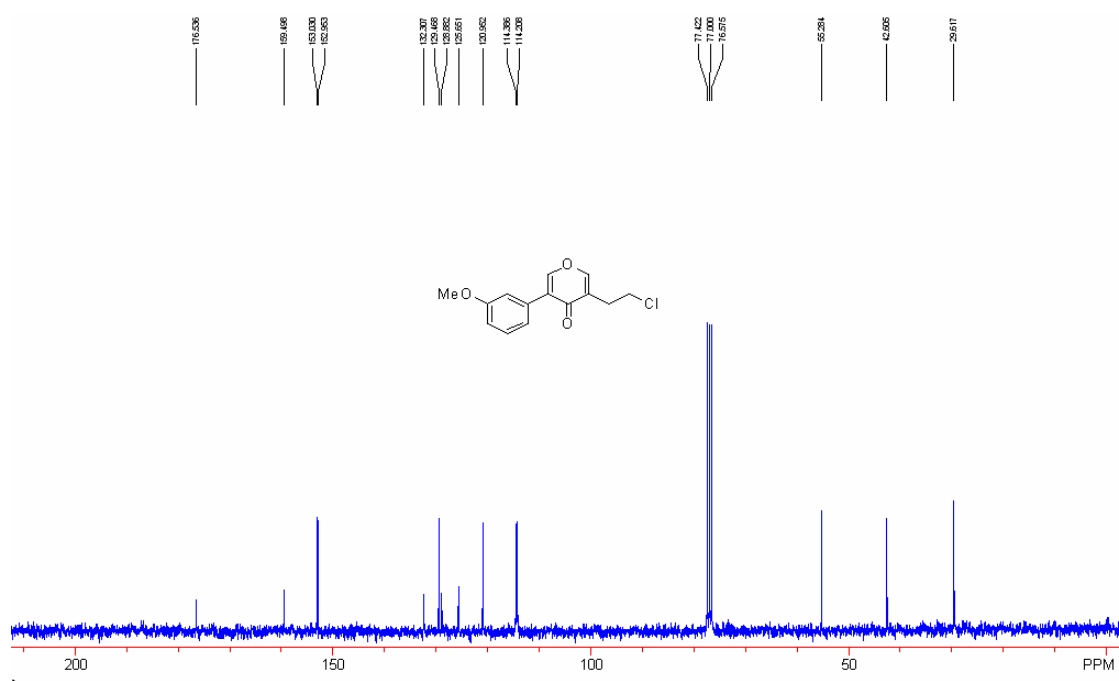
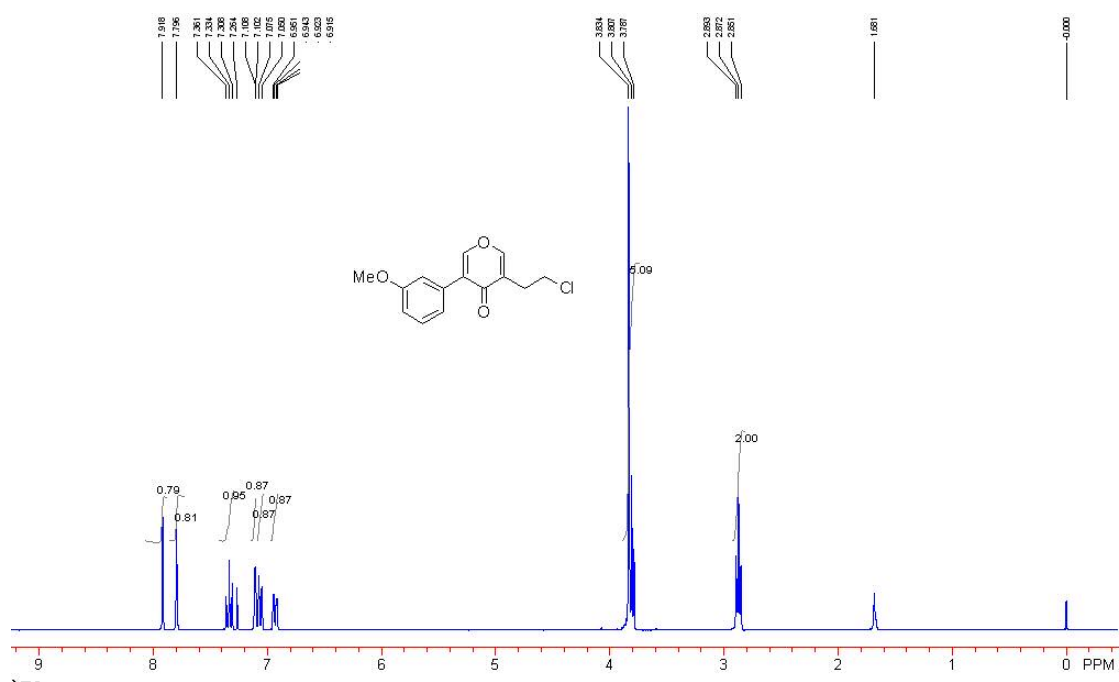


3-(2-Chloroethyl)-5-(2-methoxyphenyl)-4H-pyran-4-one (2j). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.86 (t, $J = 6.3$ Hz, 2H, CH_2), 3.79 (t, $J = 6.3$ Hz, 2H, CH_2), 3.80 (s, 3H, CH_3), 6.95-7.03 (m, 2H, Ar), 7.28-7.38 (m, 2H, Ar), 7.78 (d, $J = 1.2$ Hz, 1H), 7.88 (d, $J = 1.2$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 29.7, 42.7, 55.7, 111.2, 120.0, 120.5, 125.4, 126.4, 130.0, 131.4, 152.8, 154.3, 157.2, 176.5; IR (CH_2Cl_2): ν 3078, 3003, 2961, 2936, 1654, 1624, 1493, 1463, 1297, 1189, 1050 cm^{-1} ; MS (EI) m/z (%): 264 [M^+] (87.4), 266 (32.7), 233 (80.6), 229 (100), 217 (21.5), 215 (74.1), 131 (52.6), 89 (23.2); HRMS (EI) Calcd. for $\text{C}_{14}\text{H}_{13}\text{ClO}_3$ (M^+) requires 264.0553, Found: 264.0553.



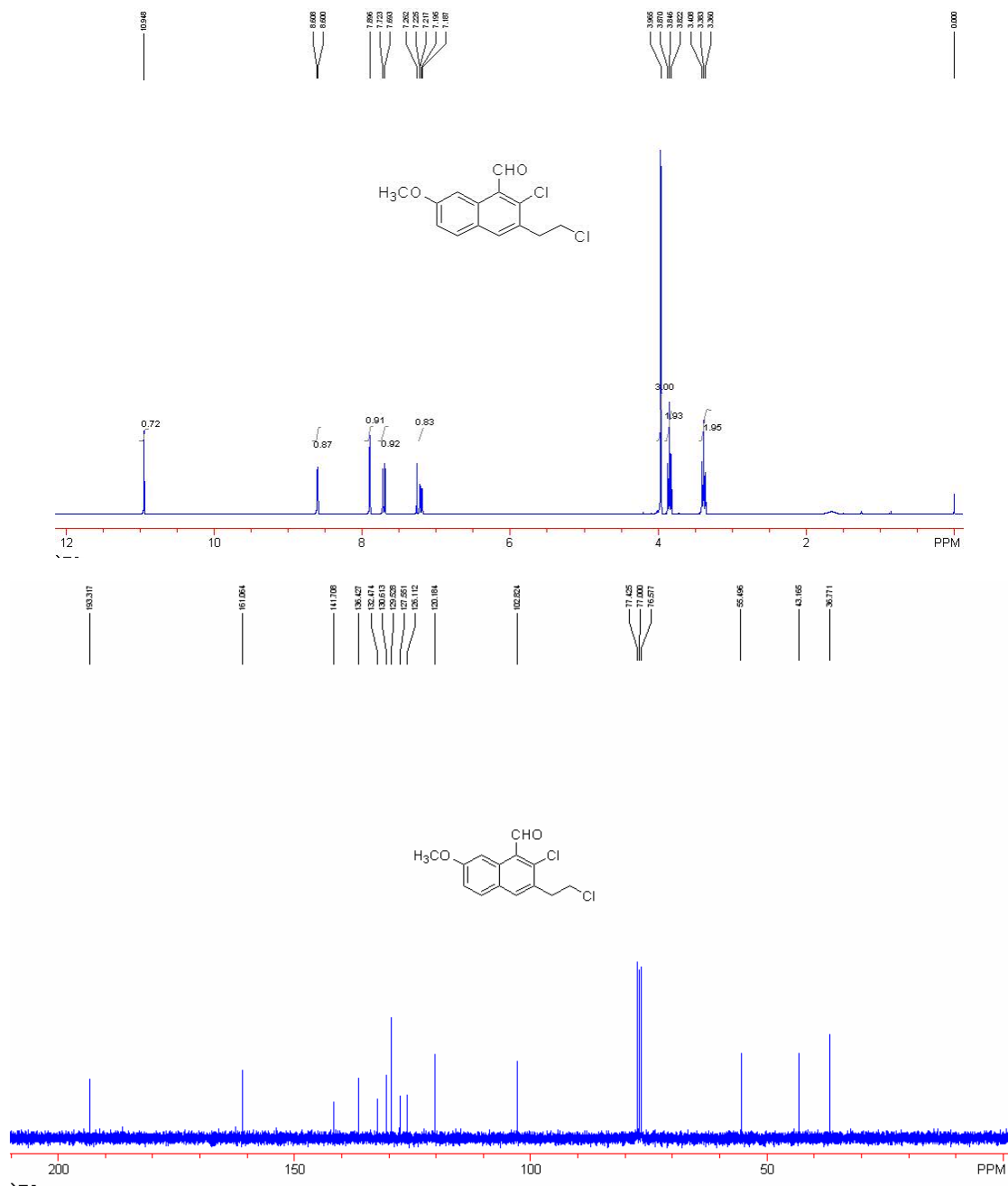


3-(2-Chloroethyl)-5-(3-methoxyphenyl)-4H-pyran-4-one (2k). A yellow oil. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.87 (t, *J* = 6.3 Hz, 2H, CH₂), 3.81 (t, *J* = 6.3 Hz, 2H, CH₂), 3.83 (s, 3H, CH₃), 6.93 (dd, *J* = 8.4 Hz, *J* = 2.4 Hz, 3H, Ar), 7.05-7.11 (m, 2H, Ar), 7.33 (t, *J* = 7.8 Hz, 1H, Ar), 7.79 (s, 1H), 7.92 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 29.6, 42.6, 55.3, 114.2, 114.4, 120.9, 125.7, 128.9, 129.5, 132.3, 152.9, 153.0, 159.5, 176.5; IR (CH₂Cl₂): ν 3081, 2960, 2836, 1649, 1618, 1488, 1420, 1292, 1190, 1039 cm⁻¹; MS (EI) *m/z* (%): 264 [M⁺] (33.0), 266 (8.8), 263 (6.0), 230 (13.6), 229 (100), 149 (4.0), 102 (2.8), 89 (3.0); HRMS (EI) Calcd. for C₁₃H₁₁O₂Cl (M⁺) requires 264.0553, Found: 264.0553.



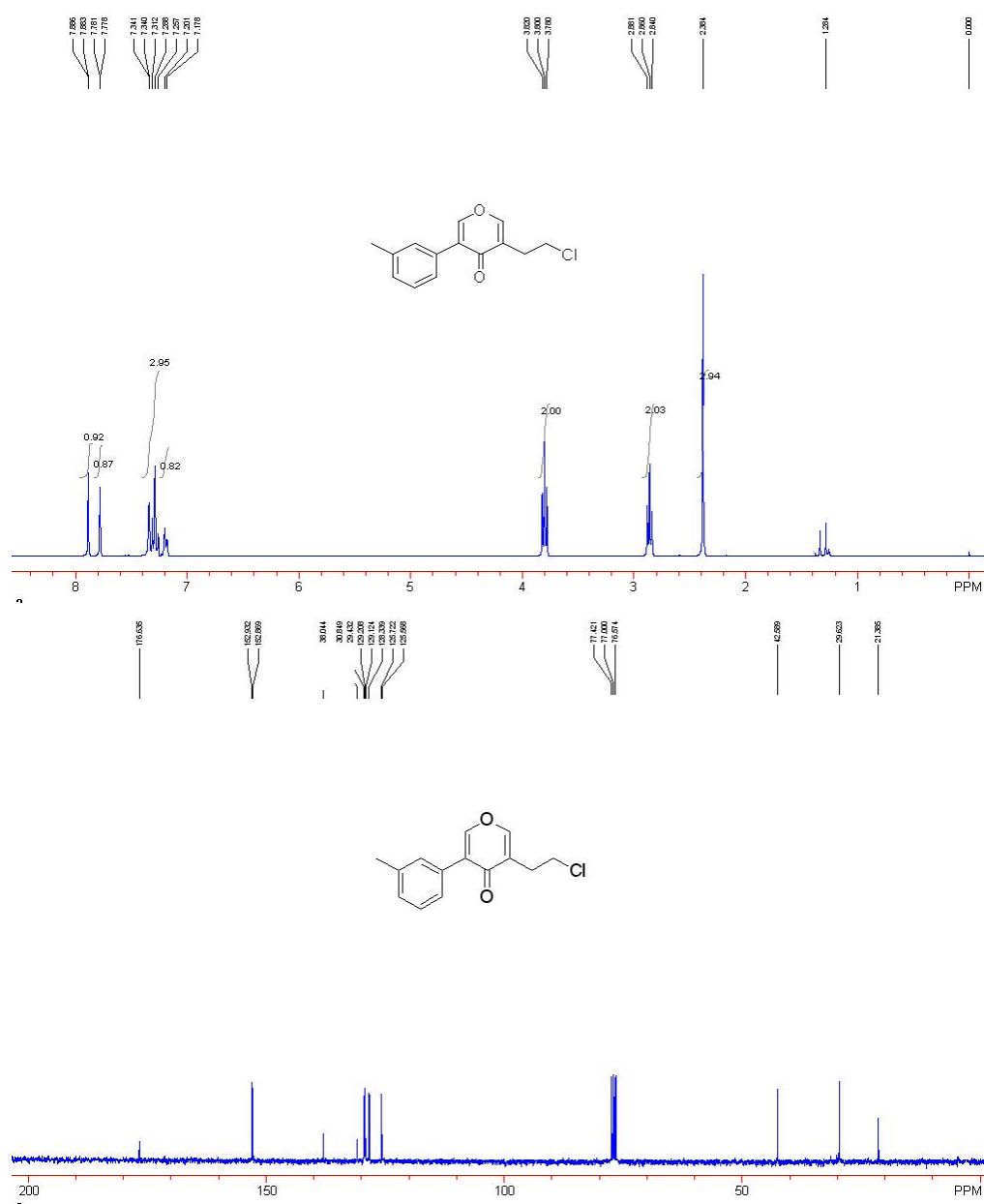
2-Chloro-3-(2-chloroethyl)-7-methoxy-1-naphthaldehyde (3k). A yellow solid. m.p. 94-96 °C. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 3.38 (t, *J* = 7.2 Hz, 2H, CH₂), 3.87 (t, *J* = 7.2 Hz, 2H, CH₂), 3.97 (s, 3H, CH₃), 7.21 (dd, *J* = 9.0 Hz, *J* = 2.4 Hz, 1H, Ar), 7.71 (d, *J* = 9.0 Hz, 1H, Ar), 7.90 (s, 1H, Ar), 8.60 (d, *J* = 2.4 Hz, 1H, Ar), 10.9 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS)

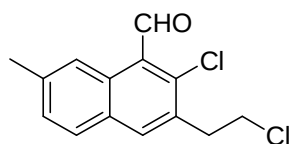
δ 36.8, 43.2, 55.5, 102.8, 120.2, 126.1, 127.6, 129.5, 130.6, 132.5, 136.4, 141.7, 161.1, 193.3;
 IR (CH₂Cl₂): ν 3134, 2923, 2855, 1687, 1620, 1499, 1453, 1380, 1238, 1062 cm⁻¹; MS (EI)
 m/z (%): 282 [M⁺] (38.7), 284 (20.9), 254 (21.9), 235 (34.4), 234 (14.6), 233 (100), 205 (26.0),
 139 (23.8); Anal. calcd. for C₁₄H₁₂Cl₂O₂: C, 59.39%; H, 4.27%. Found: C, 59.29%; H, 4.43%.



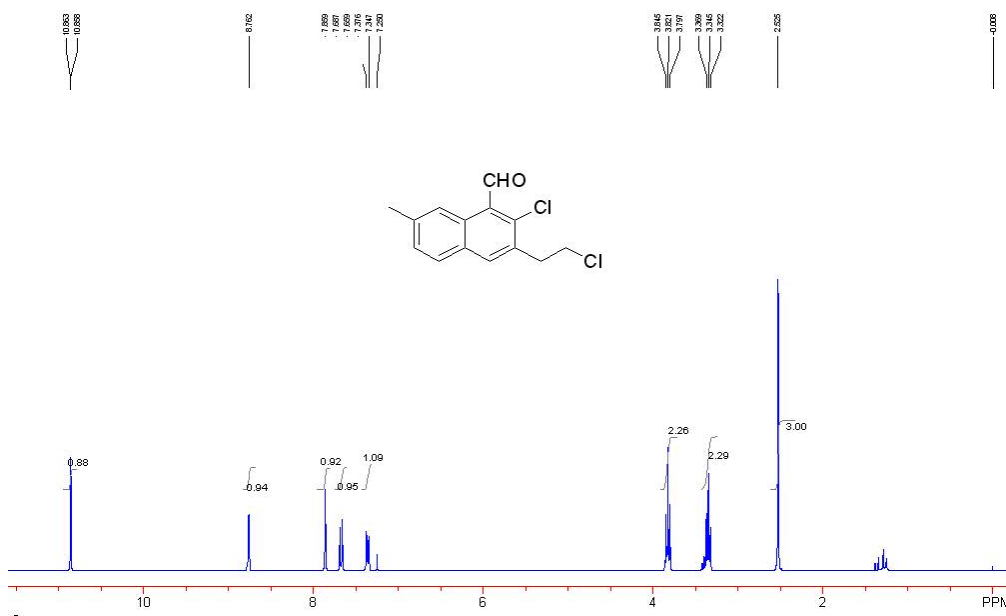
3-(2-Chloroethyl)-5-m-tolyl-4H-pyran-4-one (2l). A yellow oil. ¹H NMR (CDCl₃, 300 MHz,

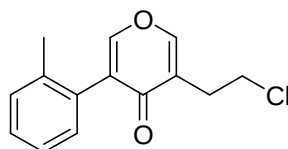
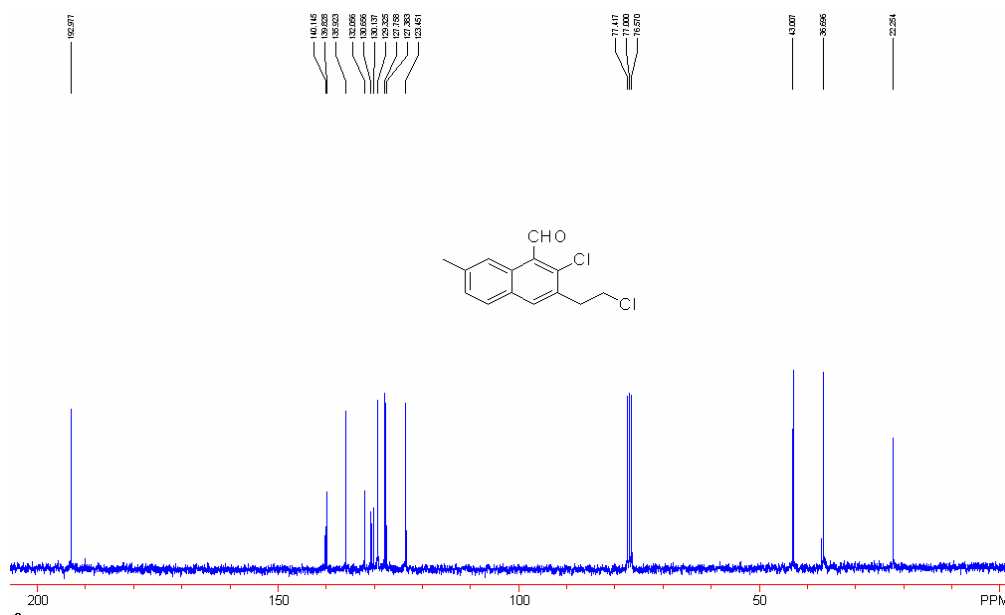
TMS) δ 2.38 (s, 3H, CH₃), 2.86 (t, J = 6.0 Hz, 2H, CH₂), 3.80 (t, J = 6.0 Hz, 2H, CH₂), 7.19 (d, J = 6.9 Hz, 1H, Ar), 7.25-7.34 (m, 3H, Ar), 7.78 (d, J = 0.9 Hz, 1H), 7.88 (d, J = 0.9 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 21.3, 29.6, 42.6, 125.6, 125.7, 128.3, 129.1, 129.2, 129.4, 130.8, 138.0, 152.9, 153.0, 176.6; IR (CH₂Cl₂): ν 3057, 2963, 2864, 1653, 1619, 1488, 1421, 1295, 1178, 1050 cm⁻¹; MS (EI) m/z (%): 248 [M⁺] (18.2), 214 (12.5), 213 (100), 115 (24.6), 77 (9.5), 63 (6.8), 53 (7.2), 51 (8.5); HRMS (EI) Calcd. for C₁₄H₁₃O₂Cl (M⁺) requires 248.0604, Found: 248.0611.



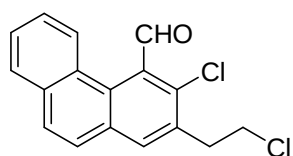
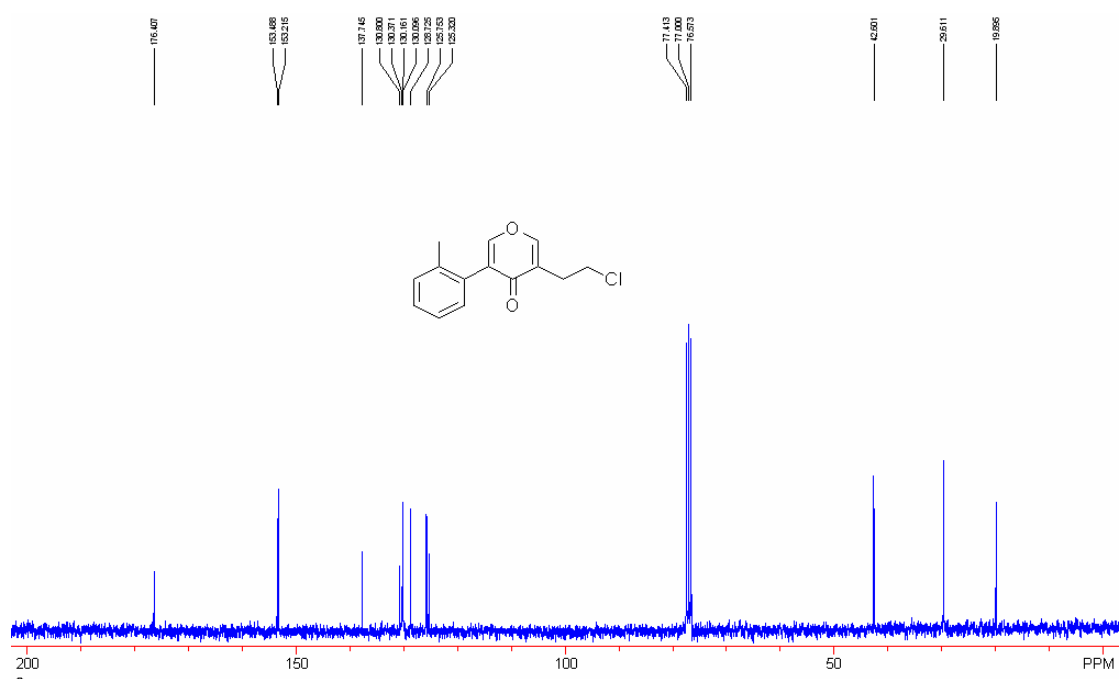
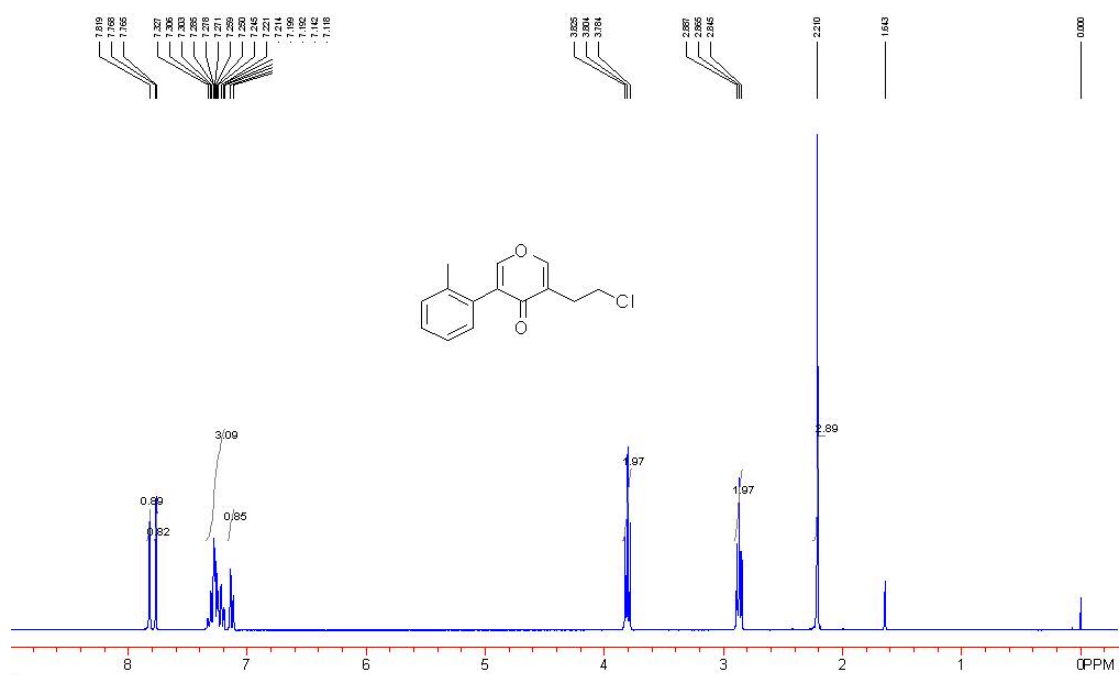


2-Chloro-3-(2-chloroethyl)-7-methyl-1-naphthaldehyde (3l). A yellow oil. ^1H NMR (CDCl_3 , 300 MHz, TMS) δ 2.53 (s, 3H, CH_3), 3.35 (t, $J = 6.9$ Hz, 2H, CH_2), 3.82 (t, $J = 6.9$ Hz, 2H, CH_2), 7.36 (d, $J = 8.4$ Hz, 1H, Ar), 7.67 (d, $J = 8.4$ Hz, 1H, Ar), 7.86 (s, 1H, Ar), 8.76 (s, 1H, Ar), 10.86 (d, $J = 1.5$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 22.3, 36.7, 43.0, 123.5, 127.4, 127.8, 129.3, 130.1, 130.7, 132.1, 135.9, 139.8, 140.2, 193.0; IR (CH_2Cl_2): ν 3053, 2959, 2870, 1624, 1593, 1496, 1439, 1352, 1138, 1068 cm^{-1} ; MS (EI) m/z (%): 266 [M^+] (55.7), 268 (37.4), 251 (38.2), 219 (33.9), 217 (100), 189 (32.9), 153 (30.2), 152 (39.6); HRMS (EI) Calcd. for $\text{C}_{14}\text{H}_{12}\text{OCl}_2$ (M^+) requires 266.0265, Found: 266.0266.



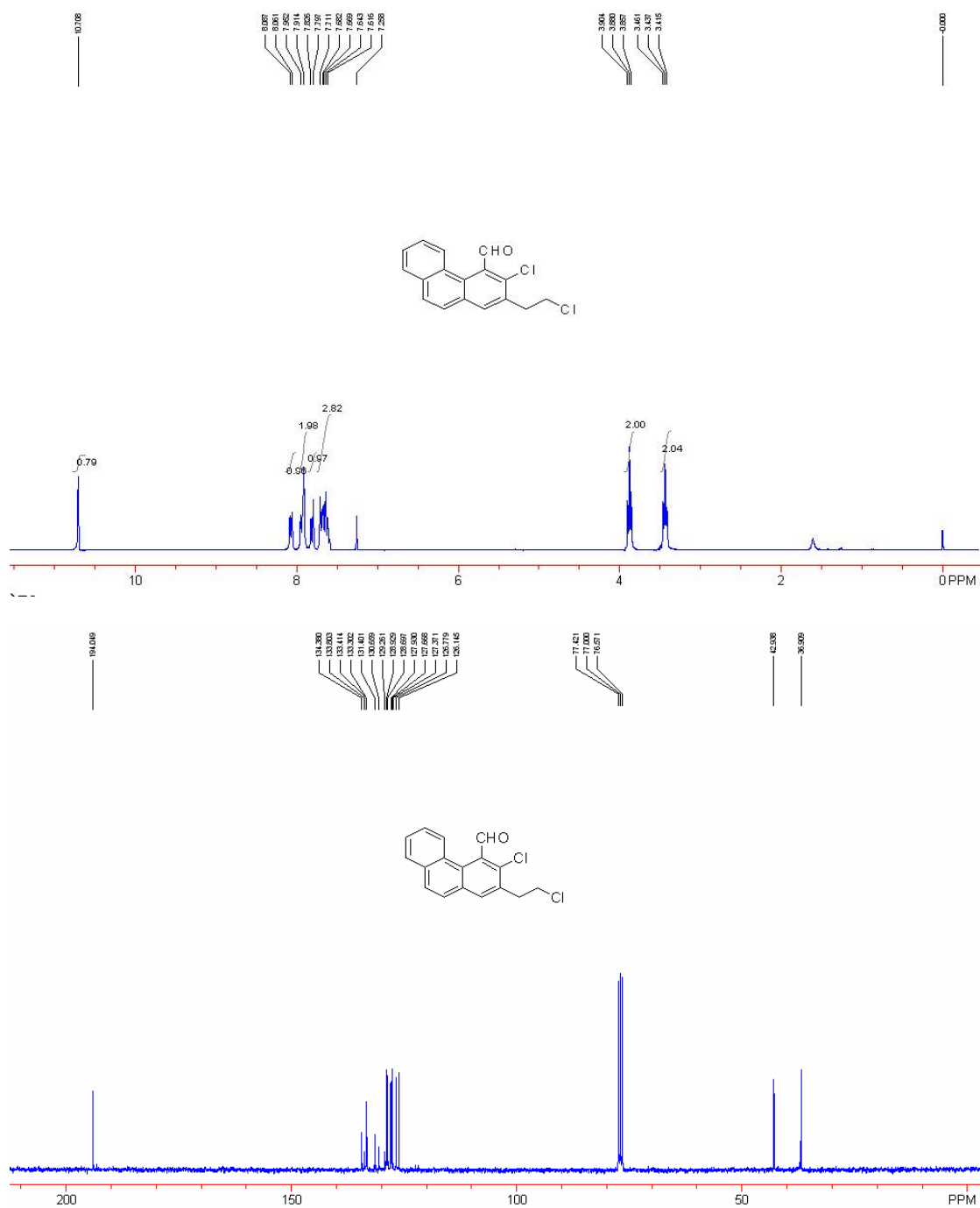


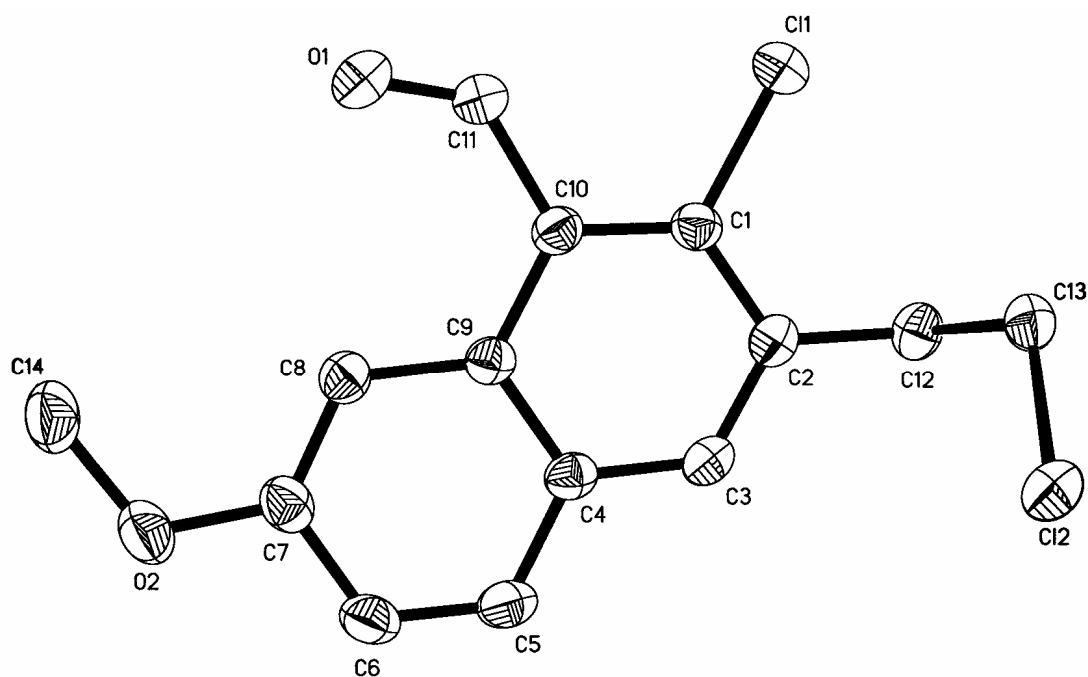
3-(2-Chloroethyl)-5-o-tolyl-4H-pyran-4-one (2m). A yellow oil. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 2.21 (s, 3H, CH₃), 2.87 (t, *J* = 6.3 Hz, 2H, CH₂), 3.80 (t, *J* = 6.3 Hz, 2H, CH₂), 7.13 (d, *J* = 7.2 Hz, 2H, Ar), 7.19-7.33 (m, 3H, Ar), 7.77 (s, 1H), 7.84 (s, 1H); ¹³C NMR (CDCl₃, 75 MHz, TMS) δ 19.9, 29.6, 42.6, 125.3, 125.8, 128.7, 130.1, 130.2, 130.4, 130.8, 137.8, 153.2, 153.5, 176.4; IR (CH₂Cl₂): ν 3072, 2924, 2853, 1652, 1619, 1488, 1420, 1295, 1189, 1038 cm⁻¹; MS (EI) *m/z* (%): 248 [M⁺] (59.2), 213 (46.1), 199 (68.5), 117 (69.1), 116 (30.5), 115 (100), 77 (23.5), 44 (25.6); HRMS (EI) Calcd. for C₁₄H₁₃ClO₂ (M⁺) requires 248.0604, Found: 248.0607.



3-chloro-2-(2-chloroethyl)phenanthrene-4-carbaldehyde (3n). A white solid. m.p. 154-156 °C. ¹H NMR (CDCl₃, 300 MHz, TMS) δ 3.44 (t, *J* = 6.9 Hz, 2H, CH₂), 3.88 (t, *J* = 6.9 Hz, 2H, CH₂), 7.62-7.71 (m, 3H, Ar), 7.81 (dd, *J* = 8.4 Hz, 1.8 Hz, 1H, Ar), 7.91-7.95 (m, 2H, Ar), 9.89 (s, 1H, CHO).

8.07 (d, $J = 8.4$ Hz, 1H, Ar), 10.71 (d, $J = 2.7$ Hz, 1H); ^{13}C NMR (CDCl_3 , 75 MHz, TMS) δ 36.9, 42.9, 126.2, 126.8, 127.4, 127.7, 127.9, 128.7, 128.9, 129.3, 130.7, 131.4, 133.3, 133.4, 133.8, 134.4, 194.1; IR (CH_2Cl_2): ν 3043, 2939, 1701, 1686, 1561, 1491, 1444, 1327, 1129, 1031 cm^{-1} ; MS (EI) m/z (%): 302 [M^+] (69.6), 305 (18.3), 304 (43.4), 303 (71.7), 301 (100), 225 (32.0), 202 (18.3), 189 (24.9); HRMS (EI) Calcd. for $\text{C}_{17}\text{H}_{12}\text{OCl}_2$ (M^+) requires 302.0265, Found: 302.0260.





The crystal data of **3c** have been deposited in CCDC with number 662441. Empirical Formula: $C_{14}H_{12}O_2Cl_2$; Formula Weight: 283.14; Crystal size: 0.483 x 0.301 x 0.190; Crystal Color, Habit: colorless, prismatic; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: $a = 4.4303(9)\text{\AA}$, $b = 10.037(2)\text{\AA}$, $c = 14.798(3)\text{\AA}$, $\alpha = 87.946(4)^\circ$, $\beta = 87.265(4)^\circ$, $\gamma = 79.808(3)^\circ$, $V = 646.7(2)\text{\AA}^3$; Space group: P-1; $Z = 2$; $D_{calc} = 1.454\text{ g/cm}^3$; $F_{000} = 292$; $R1 = 0.0449$, $wR2 = 0.1207$. Diffractometer: Rigaku AFC7R.

Table 1. Crystal data and structure refinement for cd27474.

Identification code	cd27474
Empirical formula	C14 H12 Cl2 O2
Formula weight	283.14
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 4.4303(9) Å alpha = 87.946(4) deg. b = 10.037(2) Å beta = 87.265(4) deg. c = 14.798(3) Å gamma = 79.808(3) deg.
Volume	646.7(2) Å ³
Z, Calculated density	2, 1.454 Mg/m ³
Absorption coefficient	0.492 mm ⁻¹
F(000)	292
Crystal size	0.483 x 0.301 x 0.190 mm
Theta range for data collection	2.06 to 26.00 deg.
Limiting indices	-5<=h<=5, -12<=k<=12, -18<=l<=10
Reflections collected / unique	3521 / 2492 [R(int) = 0.0676]
Completeness to theta = 26.00	97.8 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.75312
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2492 / 0 / 164
Goodness-of-fit on F ²	0.999
Final R indices [I>2sigma(I)]	R1 = 0.0449, wR2 = 0.1207
R indices (all data)	R1 = 0.0515, wR2 = 0.1251
Largest diff. peak and hole	0.307 and -0.259 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd27474. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
C1(1)	6926(2)	4792(1)	6207(1)	64(1)
C1(2)	8523(1)	3587(1)	9412(1)	58(1)
O(1)	11732(5)	7770(2)	5303(1)	81(1)
O(2)	12773(4)	11185(2)	7421(1)	68(1)
C(1)	7455(4)	6050(2)	6943(1)	43(1)
C(2)	6299(4)	5941(2)	7836(1)	42(1)
C(3)	6794(4)	6912(2)	8414(1)	44(1)
C(4)	8363(4)	7966(2)	8150(1)	42(1)
C(5)	8823(5)	8946(2)	8769(1)	53(1)
C(6)	10286(5)	9979(2)	8506(1)	56(1)
C(7)	11388(5)	10086(2)	7605(1)	50(1)
C(8)	11041(4)	9156(2)	6987(1)	46(1)
C(9)	9512(4)	8065(2)	7242(1)	39(1)
C(10)	9014(4)	7069(2)	6624(1)	43(1)
C(11)	9988(6)	7125(2)	5660(1)	59(1)
C(12)	4570(4)	4840(2)	8154(1)	50(1)
C(13)	6543(5)	3494(2)	8397(1)	50(1)
C(14)	13799(7)	11405(3)	6510(2)	74(1)

Table 3. Bond lengths [Å] and angles [deg] for cd27474.

C1(1)-C(1)	1.7494(18)
C1(2)-C(13)	1.789(2)
O(1)-C(11)	1.187(3)
O(2)-C(7)	1.368(2)
O(2)-C(14)	1.425(3)
C(1)-C(10)	1.389(3)
C(1)-C(2)	1.401(3)
C(2)-C(3)	1.372(3)
C(2)-C(12)	1.503(3)
C(3)-C(4)	1.400(3)
C(3)-H(3)	0.9300
C(4)-C(5)	1.414(3)
C(4)-C(9)	1.421(3)
C(5)-C(6)	1.353(3)
C(5)-H(5)	0.9300
C(6)-C(7)	1.405(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.363(3)
C(8)-C(9)	1.417(3)
C(8)-H(8)	0.9300
C(9)-C(10)	1.432(3)
C(10)-C(11)	1.473(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.515(3)
C(12)-H(12A)	0.9700
C(12)-H(12B)	0.9700
C(13)-H(13A)	0.9700
C(13)-H(13B)	0.9700
C(14)-H(14A)	0.9600
C(14)-H(14B)	0.9600
C(14)-H(14C)	0.9600
C(7)-O(2)-C(14)	117.52(17)
C(10)-C(1)-C(2)	123.99(16)
C(10)-C(1)-C1(1)	119.18(14)
C(2)-C(1)-C1(1)	116.81(14)
C(3)-C(2)-C(1)	116.42(17)
C(3)-C(2)-C(12)	120.97(17)
C(1)-C(2)-C(12)	122.61(17)
C(2)-C(3)-C(4)	123.29(16)
C(2)-C(3)-H(3)	118.4
C(4)-C(3)-H(3)	118.4
C(3)-C(4)-C(5)	121.46(17)
C(3)-C(4)-C(9)	119.66(17)
C(5)-C(4)-C(9)	118.88(17)
C(6)-C(5)-C(4)	121.20(19)
C(6)-C(5)-H(5)	119.4
C(4)-C(5)-H(5)	119.4
C(5)-C(6)-C(7)	119.90(19)
C(5)-C(6)-H(6)	120.0
C(7)-C(6)-H(6)	120.0
C(8)-C(7)-O(2)	124.61(19)
C(8)-C(7)-C(6)	121.13(19)
O(2)-C(7)-C(6)	114.26(18)
C(7)-C(8)-C(9)	120.22(18)
C(7)-C(8)-H(8)	119.9
C(9)-C(8)-H(8)	119.9
C(8)-C(9)-C(4)	118.67(17)
C(8)-C(9)-C(10)	123.15(17)
C(4)-C(9)-C(10)	118.16(16)
C(1)-C(10)-C(9)	118.47(16)
C(1)-C(10)-C(11)	119.37(17)
C(9)-C(10)-C(11)	122.11(17)
O(1)-C(11)-C(10)	128.3(2)
O(1)-C(11)-H(11)	115.9
C(10)-C(11)-H(11)	115.9
C(2)-C(12)-C(13)	115.33(16)
C(2)-C(12)-H(12A)	108.4

C(13)-C(12)-H(12A)	108.4
C(2)-C(12)-H(12B)	108.4
C(13)-C(12)-H(12B)	108.4
H(12A)-C(12)-H(12B)	107.5
C(12)-C(13)-Cl(2)	111.48(14)
C(12)-C(13)-H(13A)	109.3
Cl(2)-C(13)-H(13A)	109.3
C(12)-C(13)-H(13B)	109.3
Cl(2)-C(13)-H(13B)	109.3
H(13A)-C(13)-H(13B)	108.0
O(2)-C(14)-H(14A)	109.5
O(2)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
O(2)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd27474.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
C1(1)	90(1)	58(1)	52(1)	-12(1)	0(1)	-30(1)
C1(2)	66(1)	62(1)	47(1)	7(1)	-5(1)	-10(1)
O(1)	117(2)	90(1)	48(1)	-6(1)	19(1)	-50(1)
O(2)	87(1)	51(1)	72(1)	2(1)	-15(1)	-29(1)
C(1)	46(1)	43(1)	39(1)	-3(1)	-5(1)	-7(1)
C(2)	37(1)	44(1)	44(1)	4(1)	-2(1)	-2(1)
C(3)	45(1)	47(1)	36(1)	3(1)	2(1)	0(1)
C(4)	46(1)	40(1)	37(1)	0(1)	-6(1)	1(1)
C(5)	67(1)	50(1)	37(1)	-3(1)	-6(1)	0(1)
C(6)	76(1)	44(1)	50(1)	-7(1)	-16(1)	-8(1)
C(7)	56(1)	40(1)	56(1)	3(1)	-15(1)	-10(1)
C(8)	50(1)	43(1)	43(1)	5(1)	-7(1)	-8(1)
C(9)	40(1)	39(1)	38(1)	2(1)	-7(1)	-2(1)
C(10)	47(1)	44(1)	36(1)	1(1)	-2(1)	-7(1)
C(11)	82(2)	59(1)	40(1)	-5(1)	3(1)	-25(1)
C(12)	42(1)	54(1)	54(1)	6(1)	-1(1)	-11(1)
C(13)	55(1)	46(1)	51(1)	2(1)	-3(1)	-15(1)
C(14)	90(2)	61(1)	78(2)	17(1)	-14(1)	-32(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd27474.

	x	y	z	U(eq)
H(3)	6049	6867	9010	53
H(5)	8106	8880	9367	63
H(6)	10563	10616	8921	68
H(8)	11811	9237	6395	55
H(11)	9126	6586	5282	71
H(12A)	3224	4696	7682	60
H(12B)	3284	5150	8680	60
H(13A)	8027	3229	7904	60
H(13B)	5258	2807	8477	60
H(14A)	15344	10654	6329	111
H(14B)	14646	12222	6464	111
H(14C)	12098	11485	6121	111

Table 6. Torsion angles [deg] for cd27474.

C(10)-C(1)-C(2)-C(3)	-0.4(3)
Cl(1)-C(1)-C(2)-C(3)	178.38(12)
C(10)-C(1)-C(2)-C(12)	178.81(16)
Cl(1)-C(1)-C(2)-C(12)	-2.4(2)
C(1)-C(2)-C(3)-C(4)	-0.1(3)
C(12)-C(2)-C(3)-C(4)	-179.38(16)
C(2)-C(3)-C(4)-C(5)	-179.99(17)
C(2)-C(3)-C(4)-C(9)	0.3(3)
C(3)-C(4)-C(5)-C(6)	-178.64(18)
C(9)-C(4)-C(5)-C(6)	1.0(3)
C(4)-C(5)-C(6)-C(7)	-0.2(3)
C(14)-O(2)-C(7)-C(8)	3.0(3)
C(14)-O(2)-C(7)-C(6)	-176.6(2)
C(5)-C(6)-C(7)-C(8)	-0.8(3)
C(5)-C(6)-C(7)-O(2)	178.88(18)
O(2)-C(7)-C(8)-C(9)	-178.72(17)
C(6)-C(7)-C(8)-C(9)	0.9(3)
C(7)-C(8)-C(9)-C(4)	0.0(3)
C(7)-C(8)-C(9)-C(10)	178.70(17)
C(3)-C(4)-C(9)-C(8)	178.79(16)
C(5)-C(4)-C(9)-C(8)	-0.9(2)
C(3)-C(4)-C(9)-C(10)	0.0(2)
C(5)-C(4)-C(9)-C(10)	-179.70(16)
C(2)-C(1)-C(10)-C(9)	0.7(3)
Cl(1)-C(1)-C(10)-C(9)	-178.05(13)
C(2)-C(1)-C(10)-C(11)	-176.81(18)
Cl(1)-C(1)-C(10)-C(11)	4.4(3)
C(8)-C(9)-C(10)-C(1)	-179.24(16)
C(4)-C(9)-C(10)-C(1)	-0.5(3)
C(8)-C(9)-C(10)-C(11)	-1.8(3)
C(4)-C(9)-C(10)-C(11)	176.99(18)
C(1)-C(10)-C(11)-O(1)	-166.7(2)
C(9)-C(10)-C(11)-O(1)	15.8(4)
C(3)-C(2)-C(12)-C(13)	-98.9(2)
C(1)-C(2)-C(12)-C(13)	81.9(2)
C(2)-C(12)-C(13)-Cl(2)	70.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd27474 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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