Supporting Information

Lewis Acid Mediated Reactions of 1-Cyclopropyl-2-arylethanone Derivatives with Allenic Ester, Ethyl Acetoacetate, and Methyl Acrylate

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CONTENTS

General Remarks. Melting points are uncorrected. ^{1}H 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 2-Phenyl-1-(1-phenylcyclopropyl)ethanone with Ethyl Buta-2,3-dienoate. 2-Phenyl-1-(1-phenylcyclopropyl)ethanone **1a** (71 mg, 0.3 mmol), ethyl buta-2,3-dienoate (50.4 mg, 0.45 mmol), TMSOTf (54 μL, 0.3 mmol) and DCE (3.0 mL) were added into a Schlenk tube. The reaction mixture was stirred at 60 ^{o}C for 12 h. The solvent was removed under reduced pressure and then the residue was purified by a flash column chromatography (Table 1, entry 1).

General Procedure for the Reaction of 1a with Ethyl Acetoacetate (Ethyl 3-Oxobutanoate). 1-Cyclopropyl-2-phenylethanone **1i** (48 mg, 0.3 mmol), ethyl acetoacetate (ethyl 3-oxobutanoate) (58.5 mg, 0.45 mmol), TMSOTf (54 μL, 0.3 mmol) and DCE (3.0 mL) were added into a Schlenk tube. The reaction mixture was stirred at 60 ^{o}C for 15 h. The solvent was removed under reduced pressure and then the residue was purified by a flash column chromatography (Table 5, entry 1).

General Procedure for the Lewis Acid-Catalyzed Reaction of 1-Cyclopropyl-2-arylethanone 1 with Methyl Acylate.

1-Cyclopropyl-2-phenylethanone **1i** (48 mg, 0.3 mmol), methyl acylate (129 mg, 1.5 mmol), $Bi(OTf)_2Cl$ (162.6 mg, 0.3 mmol) and DCE (3.0 mL) were added into a Schlenk tube. The reaction mixture was stirred at 60 ^{o}C for 12 h. The solvent was removed under reduced

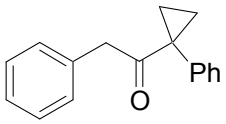
pressure and then the residue was purified by a flash column chromatography (Table 8, entry 1).

The crystal data of **2c** have been deposited in CCDC with number 678290. Empirical Formula: $C_{22}H_{20}O_2$; Formula Weight: 316.38; Crystal size: 0.456 x 0.340 x 0.180; Crystal Color, Habit: colorless, prismatic; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: a = 7.7748(12)Å, b = 12.836(2)Å, c = 17.963(3)Å, α = 97.147(3)^o, β = 93.320(3)^o, γ = 100.103(3)^o, V = 1745.3(5)Å^3; Space group: P-1; Z = 4; D_{calc} = 1.204 g/cm^3; F_{000} = 672; R1 = 0.0664, wR2 = 0.1527. Diffractometer: Rigaku AFC7R.

Although the X-ray crystal structure of **4a** has been reported in our previous paper, which has been deposited in CCDC with number 609488 (R1 = 0.0855, wR2 = 0.2556),[3b] a more precise crystal structure was obtained from the reaction shown in Scheme 4 with R1 = 0.0605, wR2 = 0.1253. Therefore, this new crystal data of **4a** have been again shown below.

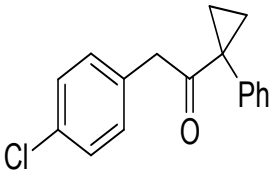
The crystal data of **4a** have been deposited in CCDC with number 669369. Empirical Formula: $C_{19}H_{16}O_3$; Formula Weight: 292.32; Crystal size: 0.428 x 0.327 x 0.119; Crystal Color, Habit: colorless, prismatic; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 10.9030(18)Å, b = 16.171(3)Å, c = 8.8944(15)Å, α = 90^o, β = 109.901(3)^o, γ = 90^o, V = 1474.6(4)Å^3; Space group: P2(1)/c; Z = 4; D_{calc} = 1.317 g/cm^3; F_{000} = 616; R1 = 0.0605, wR2 = 0.1253. Diffractometer: Rigaku AFC7R.

The crystal data of **5c** have been deposited in CCDC with number 635461. Empirical Formula: $C_{14}H_{14}BrO_{2.5}$; Formula Weight: 302.16; Crystal size: 0.503 x 0.483 x 0.437; Crystal Color, Habit: colorless, prismatic; Crystal System: Orthorhombic; Lattice Type: Primitive; Lattice Parameters: a = 45.814(7)Å, b = 9.4317(13)Å, c = 11.6643(16)Å, α = 90^o, β = 90^o, γ = 90^o, V = 5040.2(12)Å^3; Space group: Fdd2; Z = 16; D_{calc} = 1.593 g/cm^3; F_{000} = 2448; R1 = 0.0469, wR2 = 0.0979. Diffractometer: Rigaku AFC7R.

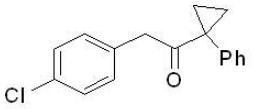


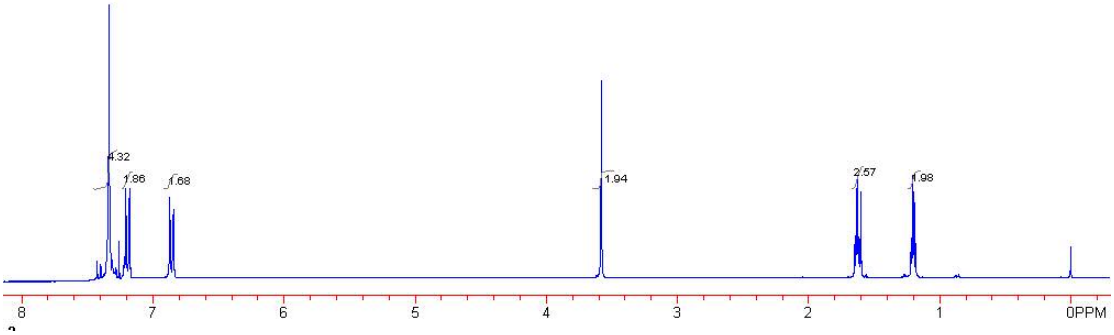
1a

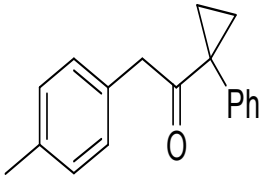
2-Phenyl-1-(1-phenylcyclopropyl)-ethanone (1e). This is a known compound.[1] ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.16-1.19 (m, 2H), 1.61-1.65 (m, 2H), 3.61 (s, 2H), 6.91-6.94 (m, 2H), 7.19-7.23 (m, 3H), 7.31-7.33 (m, 5H); Its spectroscopic data are consistent with those reported in the literature.



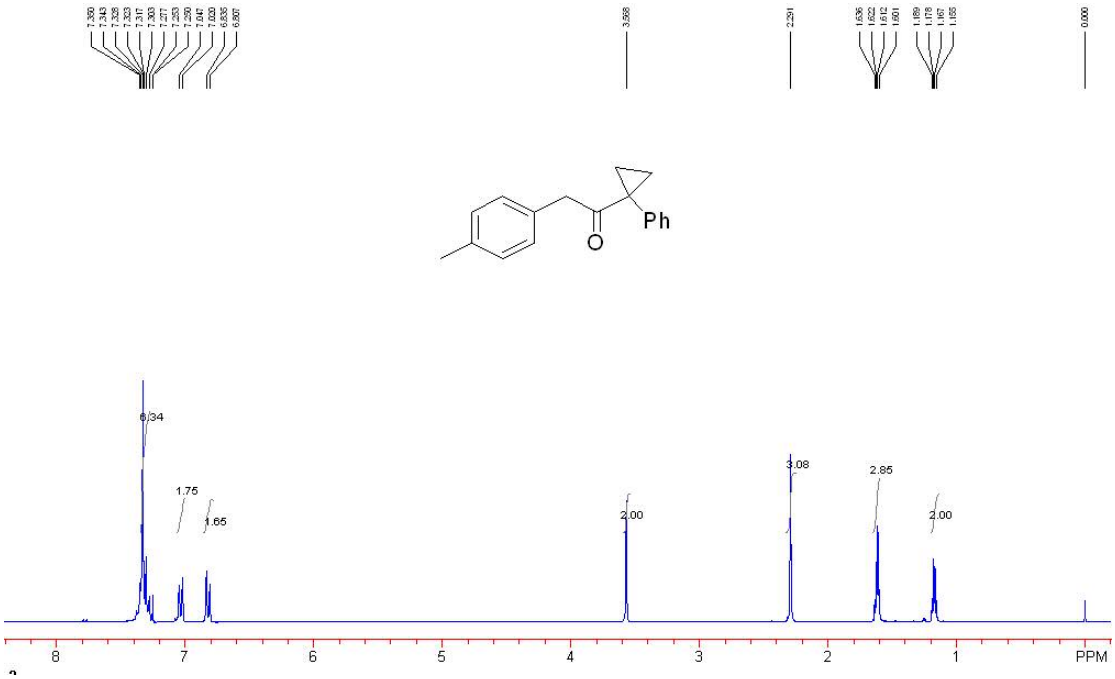
2-(4-Chlorophenyl)-1-(1-phenylcyclopropyl)ethanone 1b. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.20 (dd, J = 7.2 Hz, J = 3.6 Hz, 2H), 1.63 (dd, J = 7.2 Hz, J = 3.6 Hz, 2H), 3.58 (s, 2H), 6.86 (d, J = 8.1 Hz, 2H), 7.19 (d, J = 8.1 Hz, 2H), 7.28-7.40 (m, 5H); IR (CH_2Cl_2): ν 3083, 3026, 2360, 1689, 1600, 1492, 1322, 1059, 1015, 702 cm^{-1}; MS (EI) m/z (%): 270 [M^+] (1.8), 145 (44.7), 125 (9.3), 118 (11.4), 117 (100), 115 (42.3), 91 (18.2), 89 (12.4), 77 (8.3); HRMS (EI) Calcd. for $C_{17}H_{15}OCl$ (M^+) requires 270.0811, Found: 270.0818.



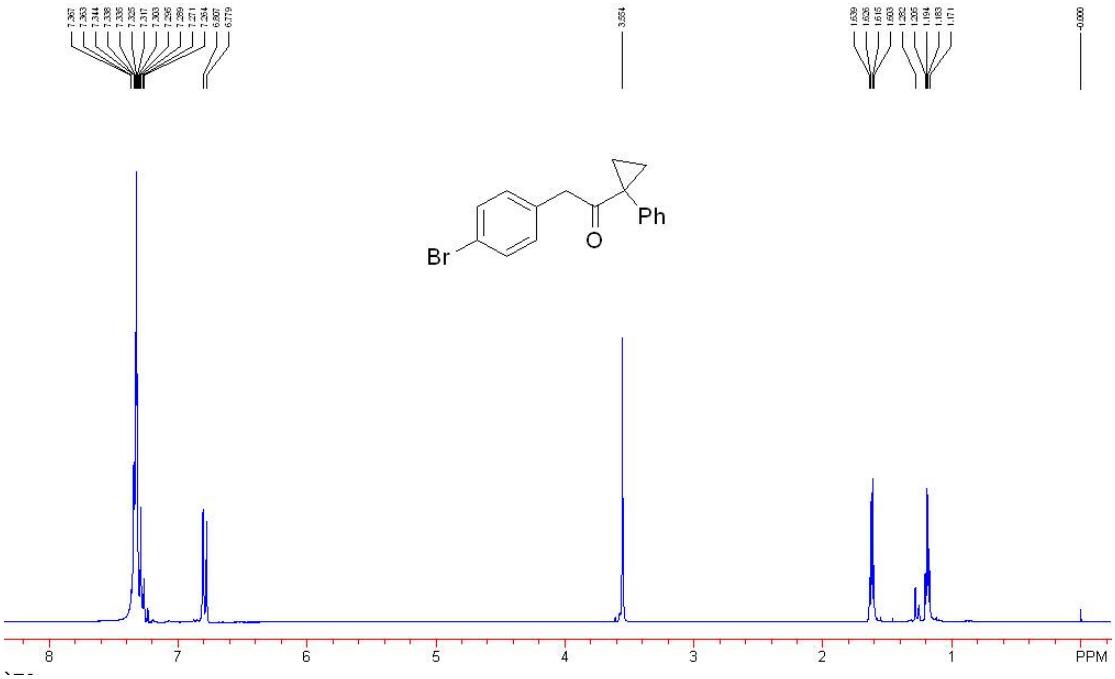


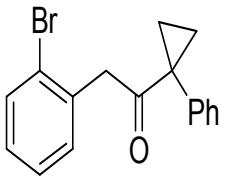


1-(1-Phenylcyclopropyl)-2-p-tolylethanone 1c. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.17 (dd, *J* = 6.9 Hz, *J* = 3.6 Hz, 2H), 1.62 (dd, *J* = 6.9 Hz, *J* = 3.6 Hz, 2H), 3.57 (s, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 7.28-7.35 (m, 5H); IR (CH_2Cl_2): ν 3084, 3024, 2235, 1698, 1601, 1496, 1324, 1059, 1022, 701 cm^{-1}; MS (EI) m/z (%): 250 [M^+] (1.8), 145 (27.9), 119 (40.0), 117 (88.1), 115 (43.5), 105 (100), 91 (42.6), 77 (26.4), 57 (34.3), 43 (39.7); HRMS (EI) Calcd. for $C_{18}H_{18}O$ (M^+) requires 250.1358, Found: 250.1367.

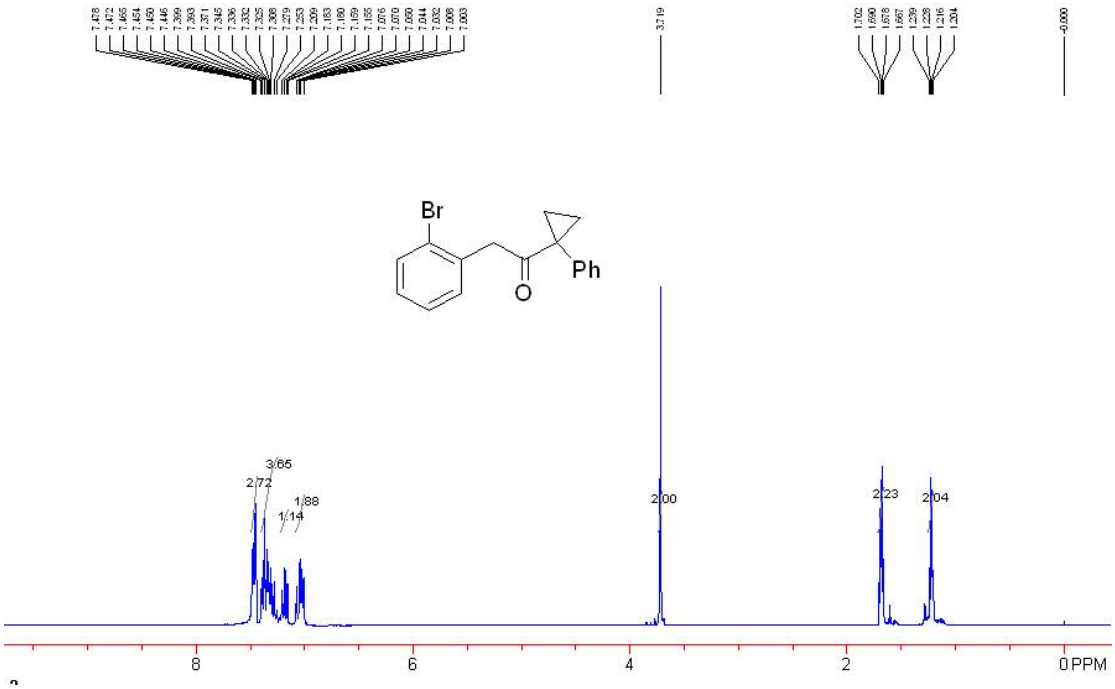


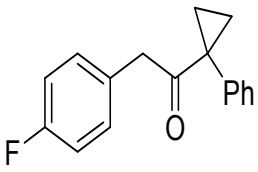
2-(4-Bromophenyl)-1-(1-phenylcyclopropyl)ethanone 1d. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.19 (dd, *J* = 6.9 Hz, *J* = 3.6 Hz, 2H), 1.62 (dd, *J* = 6.9 Hz, *J* = 3.6 Hz, 2H), 3.55 (s, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 7.27-7.37 (m, 7H); IR (CH_2Cl_2): ν 3083, 3026, 2234, 1698, 1601, 1488, 1323, 1059, 1023, 702 cm^{-1}; MS (EI) m/z (%): 314 [M^+] (2.5), 145 (48.7), 118 (10.9), 117 (100), 115 (36.4), 91 (18.0), 90 (15.2), 89 (18.9), 63 (9.7); HRMS (EI) Calcd. for $C_{17}H_{15}OBr$ (M^+) requires 314.0306, Found: 314.0292.



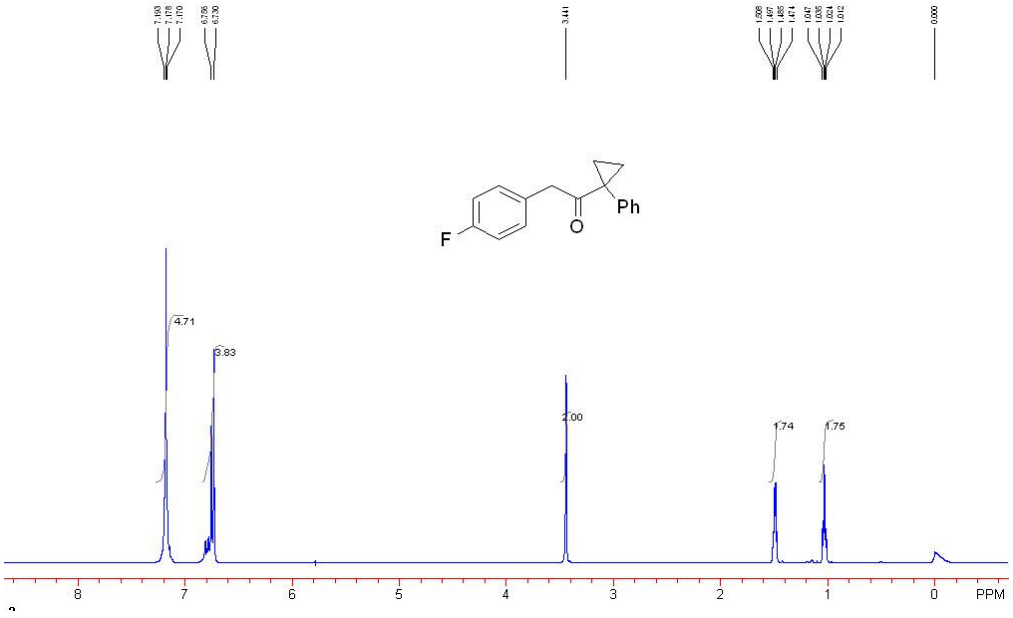


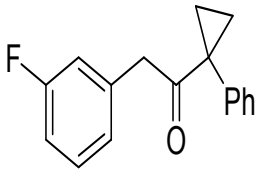
2-(2-Bromophenyl)-1-(1-phenylcyclopropyl)ethanone 1e. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.22 (dd, J = 7.2 Hz, J = 3.3 Hz, 2H), 1.68 (dd, J = 7.2 Hz, J = 3.3 Hz, 2H), 3.72 (s, 2H), 7.00-7.08 (m, 2H), 7.16-7.21 (m, 1H), 7.28-7.40 (m, 3H), 7.45-7.48 (m, 3H); IR (CH_2Cl_2): ν 3058, 3024, 1701, 1600, 1495, 1471, 1323, 1059, 1026, 702 cm^{-1}; MS (EI) m/z (%): 314 [M^+] (1.9), 145 (40.6), 118 (12.7), 117 (100), 115 (37.7), 91 (20.3), 90 (14.4), 89 (18.4), 63 (10.2); HRMS (EI) Calcd. for $C_{17}H_{15}OBr$ (M^+) requires 314.0306, Found: 314.0312.



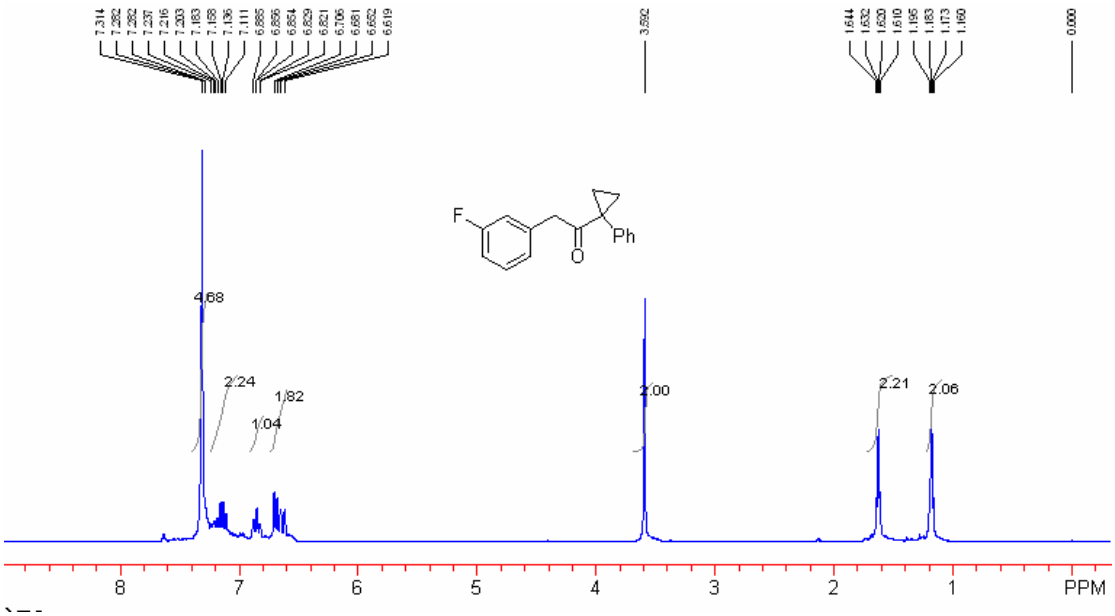


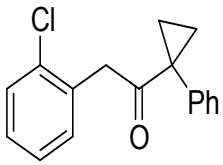
2-(4-Fluorophenyl)-1-(1-phenylcyclopropyl)ethanone 1f. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.03 (dd, J = 7.2 Hz, J = 3.6 Hz, 2H), 1.50 (dd, J = 7.2 Hz, J = 3.6 Hz, 2H), 3.44 (s, 2H), 6.74 (d, J = 7.8 Hz, 4H), 7.17-7.19 (m, 5H); IR (CH_2Cl_2): ν 3059, 3026, 1698, 1602, 1509, 1323, 1060, 1023, 702 cm^{-1}; MS (EI) m/z (%): 254 [M^+] (4.6), 146 (6.4), 145 (50.4), 118 (11.6), 117 (100), 115 (40.1), 109 (22.3), 91 (16.9), 83 (9.1); HRMS (EI) Calcd. for $C_{17}H_{15}OF$ (M^+) requires 254.1107, Found: 254.1113.



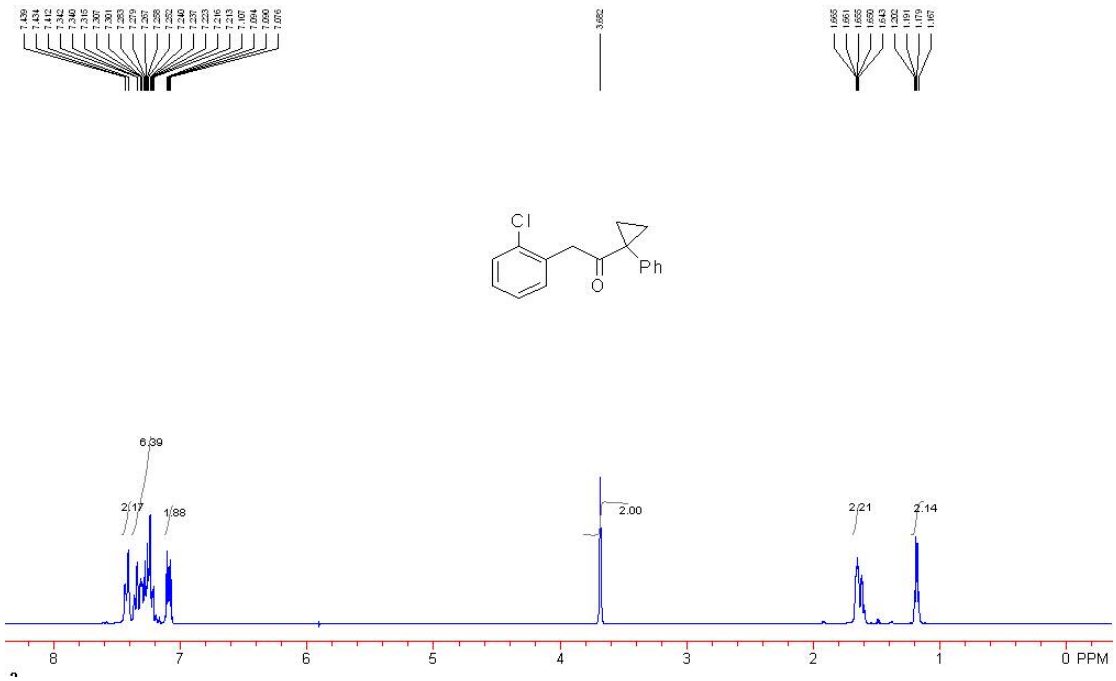


2-(3-Fluorophenyl)-1-(1-phenylcyclopropyl)ethanone 1g. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.18 (dd, J = 6.6 Hz, J = 3.6 Hz, 2H), 1.63 (dd, J = 6.6 Hz, J = 3.6 Hz, 2H), 3.59 (s, 2H), 6.62-6.71 (m, 2H), 6.82-6.89 (m, 1H), 7.11-7.24 (m, 1H), 7.28-7.31 (m, 5H); IR (CH_2Cl_2): ν 3082, 3026, 1700, 1590, 1488, 1323, 1107, 1023, 702 cm^{-1}; MS (EI) m/z (%): 254 [M^+] (5.2), 146 (7.7), 145 (60.7), 118 (10.8), 117 (100), 115 (41.7), 109 (17.8), 91 (15.8), 83 (7.2); HRMS (EI) Calcd. for $C_{17}H_{15}OF$ (M^+) requires 254.1107, Found: 254.1112.





2-(2-Chlorophenyl)-1-(1-phenylcyclopropyl)ethanone 1h. A yellow liquid. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.28 (dd, J = 7.2 Hz, J = 3.6 Hz, 2H), 1.66 (dd, J = 7.2 Hz, J = 3.6 Hz, 2H), 3.68 (s, 2H), 7.08-7.11 (m, 2H), 7.21-7.34 (m, 5H), 7.41-7.44 (m, 2H); IR (CH_2Cl_2): ν 3059, 3025, 1701, 1601, 1495, 1475, 1324, 1060, 1022, 703 cm^{-1}; MS (EI) m/z (%): 270 [M^+] (6.6), 146 (6.8), 145 (67.4), 125 (10.1), 118 (9.1), 117 (100), 115 (23.1), 91 (8.1); HRMS (EI) Calcd. for $C_{17}H_{15}OBr$ (M^+) requires 270.0811, Found: 270.0811.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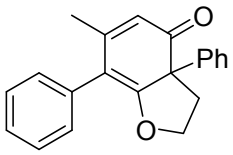
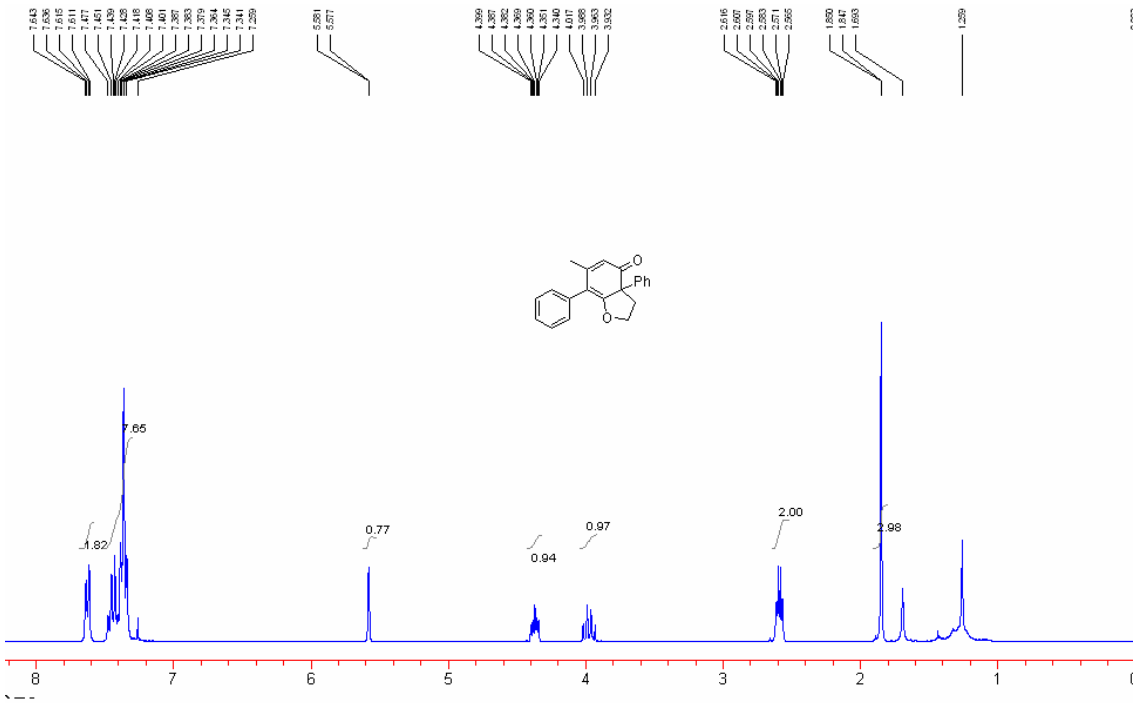
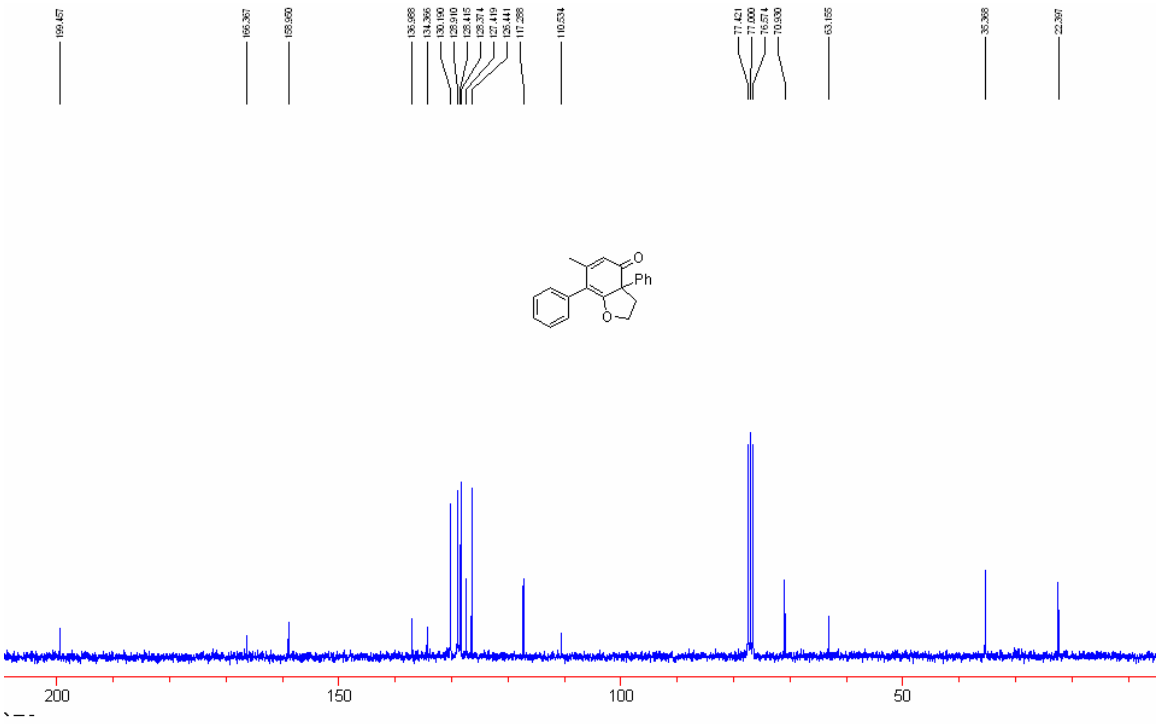
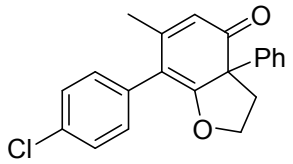


2a

6-Methyl-3a,7-diphenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2a. A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.85 (d, 3H, *J* = 0.9 Hz), 2.56-2.61 (m, 2H), 3.96 (dd, 1H, *J* = 15.8 Hz, *J* = 9.0 Hz), 4.34-4.40 (m, 1H), 5.58 (d, 1H, *J* = 0.9 Hz), 7.34-7.47 (m, 8H), 7.62 (dd, 2H, *J* = 8.0 Hz, *J* = 1.7 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 22.4, 35.4, 63.2, 70.9, 110.5, 117.3, 126.4, 127.4, 128.3, 128.4, 128.9, 130.2, 134.4, 137.0, 159.0, 166.4, 199.5; IR (CH_2Cl_2): ν 3057, 2924, 2851, 1674, 1537, 1492, 1380, 1265, 991 cm^{-1}; MS (EI) m/z (%): 302 [M$^+$] (100), 303 (26.9), 274 (23.3), 259 (21.0), 231 (32.0), 129 (28.8), 128 (24.0), 115 (33.5); HRMS (EI) Calcd. for $C_{21}H_{18}O_2$ (M$^+$) requires 302.1307, Found: 302.1306.

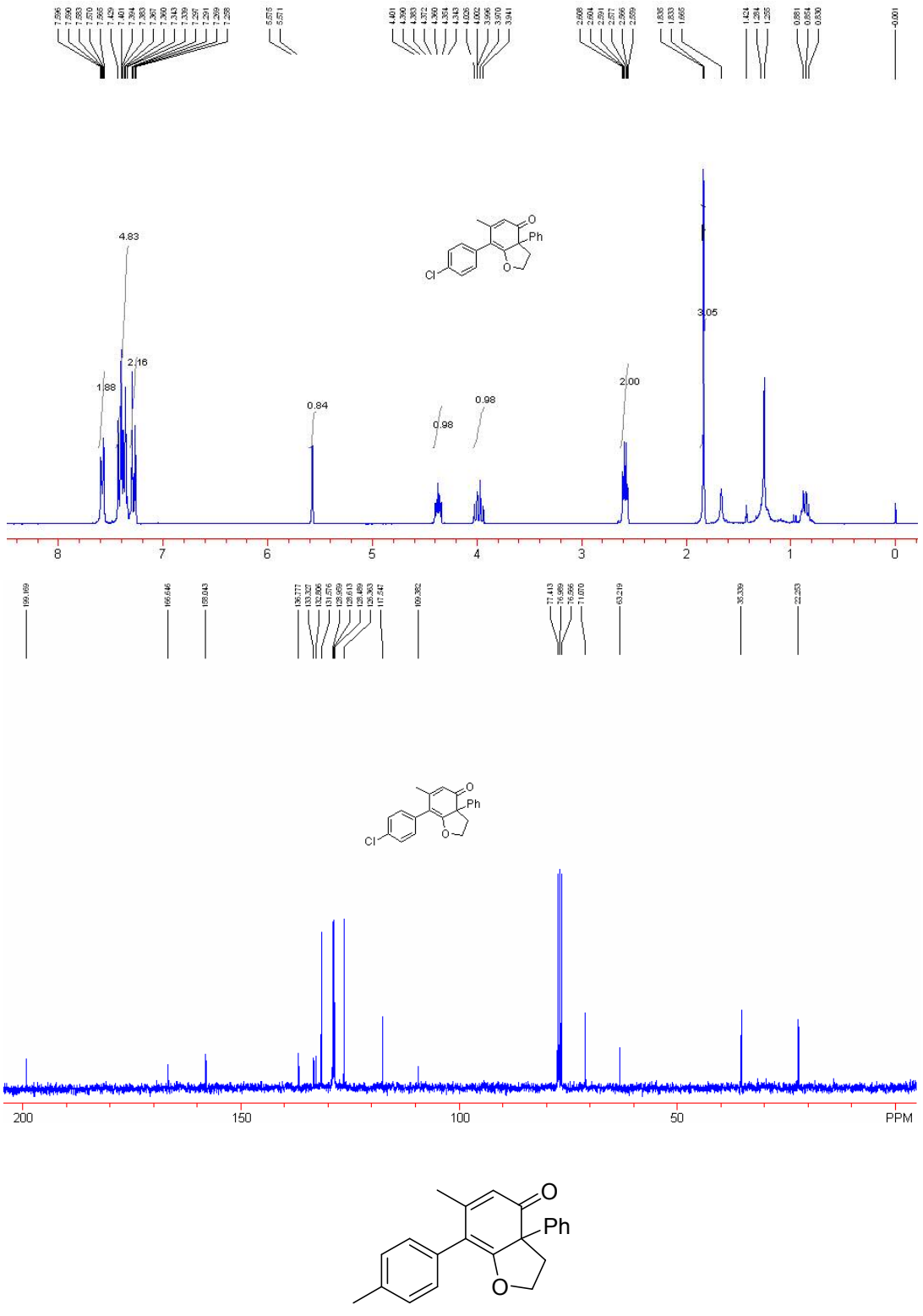






2b

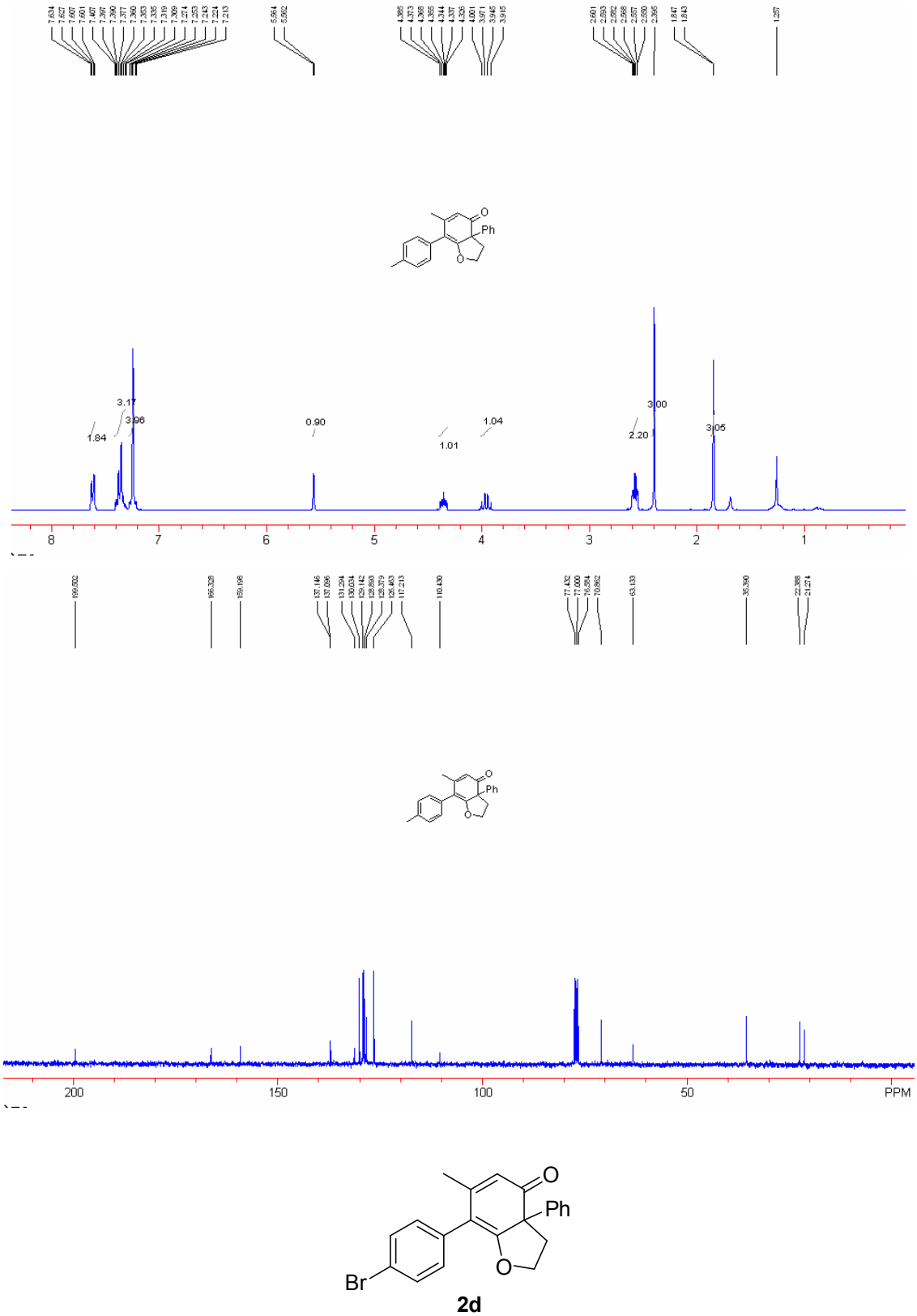
5-(4-Chlorophenyl)-6-methyl-3a-phenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2b. A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.84 (d, J = 0.9 Hz, 3H), 2.56-2.61 (m, 2H), 3.97 (dd, J = 17.7 Hz, J = 9.0 Hz, 1H), 4.34-4.40 (m, 1H), 5.57 (d, J = 0.9 Hz, 1H), 7.28 (dd, J = 7.8 Hz, J = 1.8 Hz, 2H), 7.36-7.43 (m, 5H), 7.58 (dd, J = 7.8 Hz, J = 1.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 22.3, 35.3, 63.2, 71.1, 109.4, 117.6, 126.4, 128.5, 128.6, 129.0, 131.6, 132.8, 133.3, 136.8, 158.1, 166.7, 199.2; IR (CH_2Cl_2): ν 3473, 3060, 2926, 2853, 1734, 1541, 1493, 1380, 1265, 1091 cm^{-1}; MS (EI) m/z (%): 336 [M^+] (100), 338 (31.4), 374 (25.1), 308 (21.0), 139 (23.7), 128 (18.9), 115 (37.4), 44 (21.3); HRMS (EI) Calcd. for $C_{21}H_{17}O_2Cl$ (M^+) requires 336.0917, Found: 336.0929.



2c

6-Methyl-3a-phenyl-7-*p*-tolyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2c. A yellow solid. m.p. 158-160 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.85 (d, 3H, J = 0.9 Hz), 2.40 (s, 3H), 2.55-2.60 (m, 2H), 3.95 (dd, 1H, J = 15.2 Hz, J = 8.7 Hz), 4.33-4.39 (m, 1H), 5.56 (d, 1H, J = 0.9 Hz), 7.21-7.27 (m, 4H), 7.31-7.41 (m, 3H), 7.61 (dd, 2H, J = 7.8 Hz, J = 1.8 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.3, 22.4, 35.4, 63.1, 70.9, 110.4, 117.2, 126.5, 128.4, 128.9, 129.1, 130.0, 131.3, 137.1, 137.2, 159.2, 166.3, 199.5; IR (CH_2Cl_2): ν 3023, 2923, 2853, 1721,

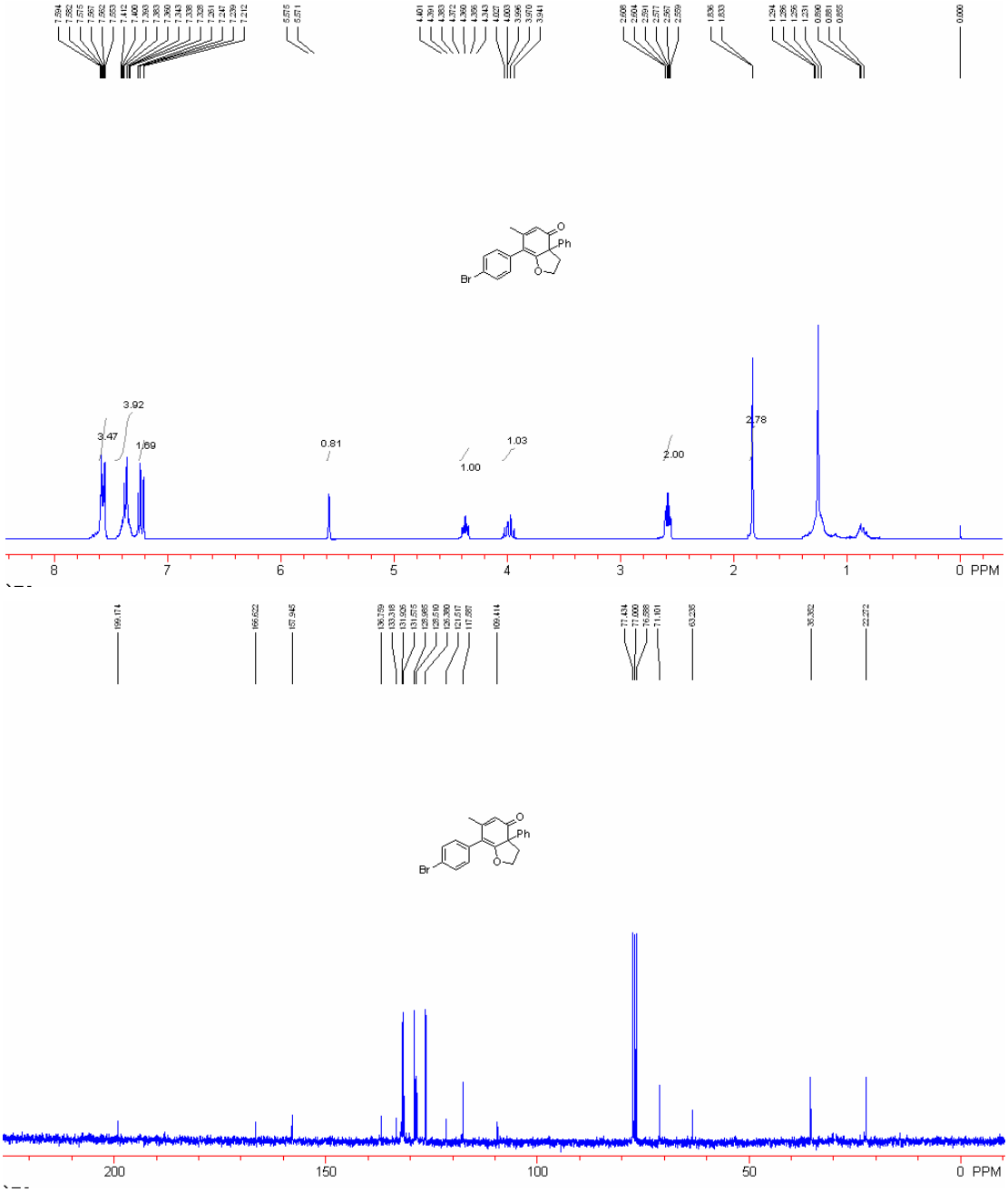
1671, 1512, 1380, 1266, 1030 cm^{-1}; MS (EI) m/z (%): 316 [M^+] (100), 317 (23.1), 288 (19.6), 273 (20.6), 245 (19.4), 128 (15.2), 115 (20.7), 91 (15.3); HRMS (EI) Calcd. for $C_{22}H_{20}O_2$ (M^+) requires 316.1463, Found: 316.1474.

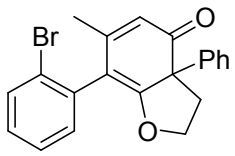


2d

7-(4-Bromophenyl)-6-methyl-3a-phenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2d. A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.84 (d, 3H, J = 0.9 Hz), 2.56-2.61 (m, 2H), 3.98 (dd,

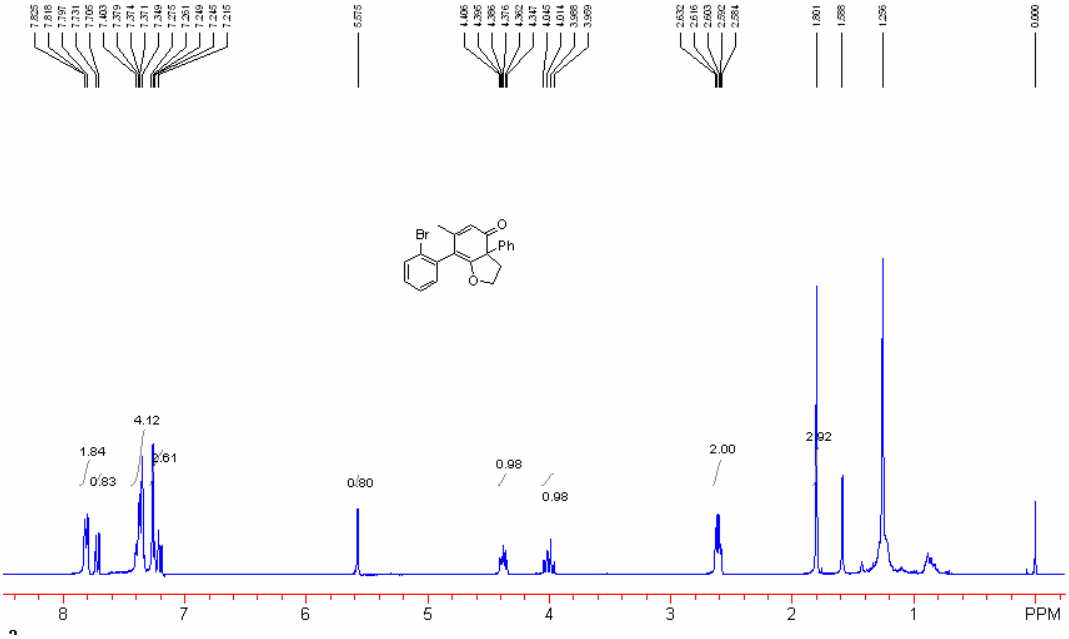
1H, J = 16.5 Hz, J = 9.0 Hz), 4.34-4.40 (m, 1H), 5.57 (d, 1H, J = 0.9 Hz), 7.23 (d, 2H, J = 8.1 Hz), 7.33-7.41 (m, 4H), 7.56 (d, 2H, J = 8.1 Hz), 7.55-7.59 (m, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 22.2, 35.3, 63.2, 71.1, 109.4, 117.6, 121.5, 126.4, 128.5, 129.0, 131.6, 131.9, 133.3, 136.7, 157.9, 166.6, 199.1; IR (CH_2Cl_2): ν 3058, 2924, 2854, 1729, 1672, 1488, 1379, 1265, 1070 cm^{-1}; MS (EI) m/z (%): 380 [M^+] (100), 382 (95.2), 381 (30.6), 352 (30.5), 215 (31.9), 128 (33.9), 115 (51.6), 91 (29.0); HRMS (EI) Calcd. for $C_{21}H_{17}O_2Br$ (M^+) requires 380.0412, Found: 380.0426.

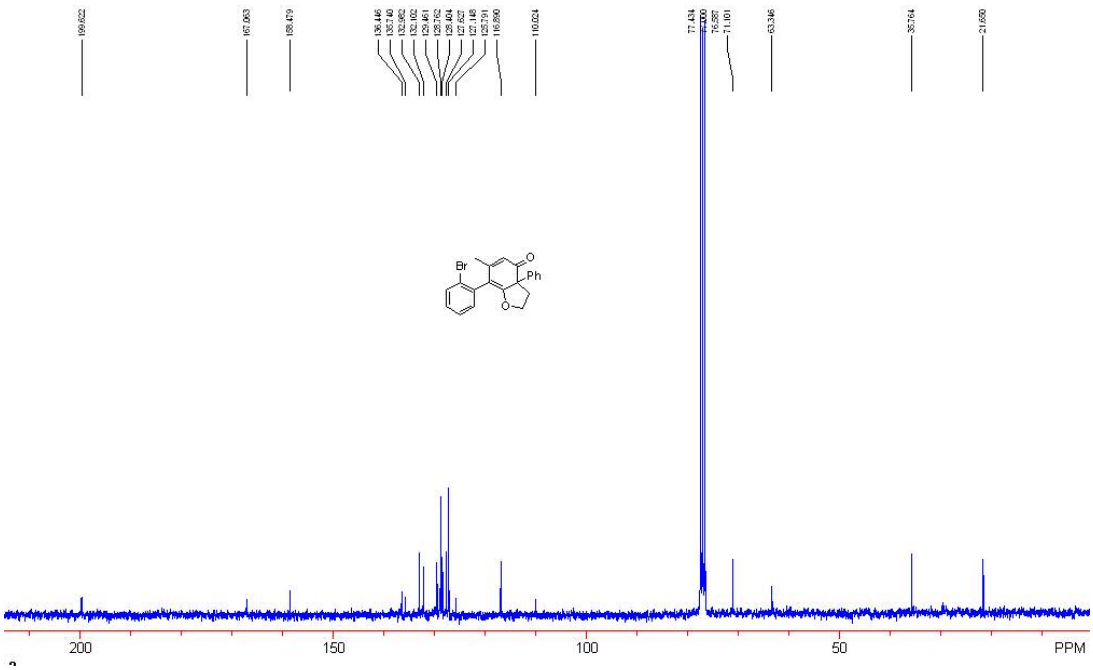


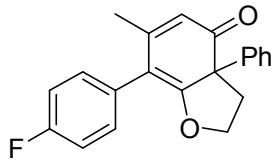


2e

7-(2-Bromophenyl)-6-methyl-3a-phenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2e. A yellow solid. m.p. 160-162 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.80 (s, 3H), 2.58-2.63 (m, 2H), 3.98 (dd, 1H, *J* = 17.1 Hz, *J* = 8.7 Hz), 4.35-4.41 (m, 1H), 5.58 (s, 1H), 7.18-7.25 (m, 2H), 7.33-7.40 (m, 4H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.81 (dd, *J* = 7.8 Hz, *J* = 2.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 35.8, 63.3, 71.1, 110.0, 116.9, 125.8, 127.1, 127.6, 128.4, 128.8, 129.5, 132.1, 133.0, 135.7, 136.5, 158.5, 167.1, 199.6; IR (CH_2Cl_2): ν 3058, 2924, 2853, 1731, 1675, 1492, 1380, 1255, 1071, 762 cm^{-1}; MS (EI) m/z (%): 380 [M^+] (92.3), 382 (89.5), 301 (66.3), 273 (100), 245 (61.8), 215 (57.7), 202 (49.0), 115 (58.8); HRMS (EI) Calcd. for $C_{21}H_{17}O_2Br$ (M^+) requires 380.0412, Found: 380.0417.

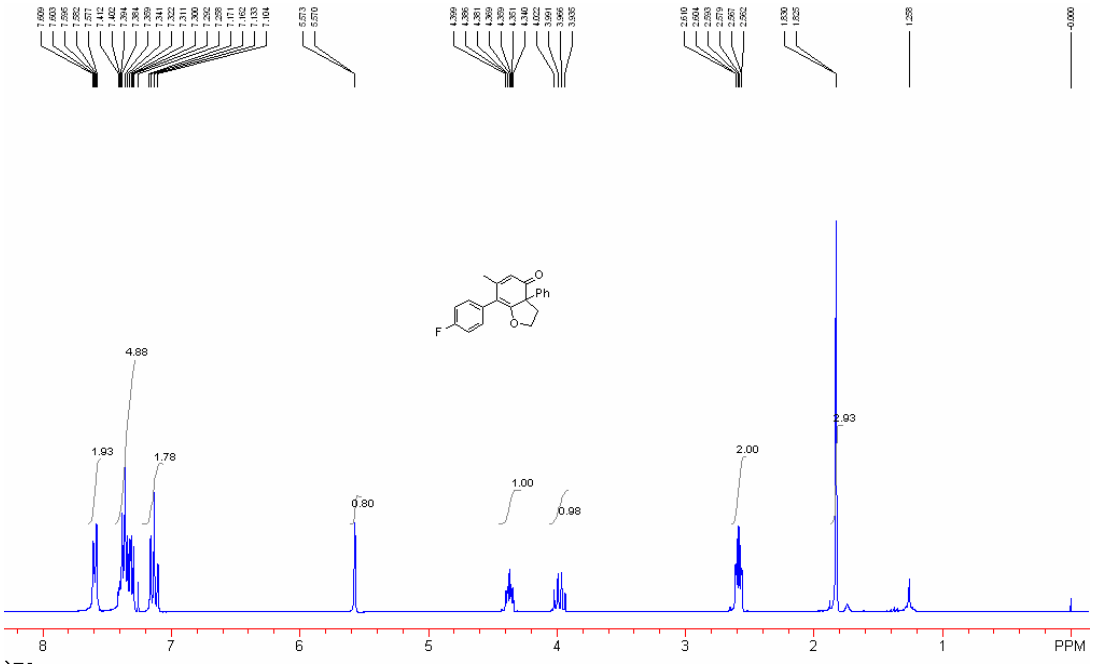


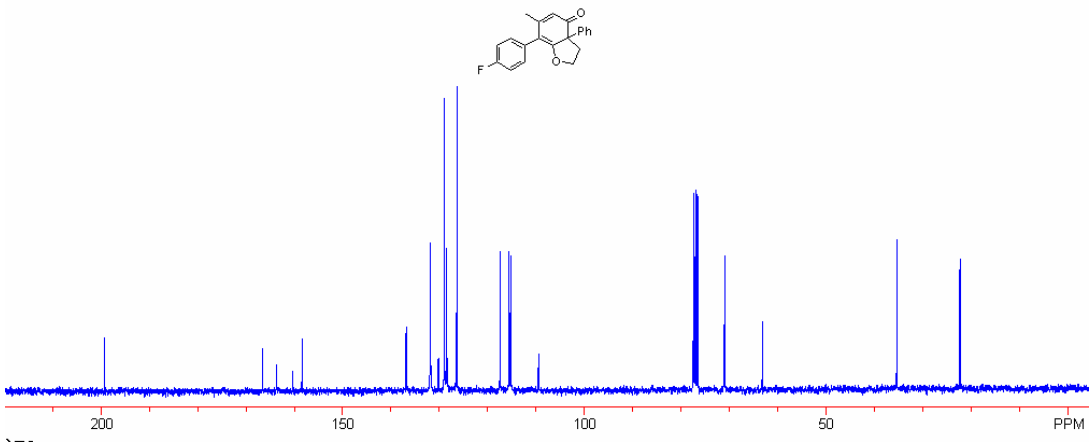


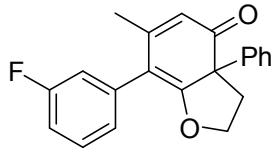


2f

7-(4-Fluorophenyl)-6-methyl-3a-phenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2f. A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.83 (d, 3H, J = 0.9 Hz), 2.56-2.61 (m, 2H), 3.96 (dd, 1H, J = 17.8 Hz, J = 9.3 Hz), 4.34-4.40 (m, 1H), 5.57 (d, 1H, J = 0.9 Hz), 7.13 (dd, J = 8.7 Hz, J = 8.7 Hz, 2H), 7.29-7.41 (m, 5H), 7.59 (dd, J = 7.5 Hz, J = 1.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 22.3, 35.3, 63.2, 71.0, 109.5, 115.4 (d, $J_{C\text{-}F}$ = 21.8 Hz), 117.4, 126.4, 128.5, 128.9, 130.2 (d, $J_{C\text{-}F}$ = 3.5 Hz), 131.9 (d, $J_{C\text{-}F}$ = 8.0 Hz), 136.9, 158.4, 160.4, 165.2 (d, $J_{C\text{-}F}$ = 220.4 Hz), 199.2; IR (CH_2Cl_2): ν 3060, 2962, 2902, 1722, 1671, 1538, 1380, 1266, 1028 cm^{-1}; MS (EI) m/z (%): 320 [M^+] (47.3), 192 (32.6), 145 (55.2), 123 (35.1), 117 (100), 115 (45.3), 109 (26.6), 91 (25.8); HRMS (EI) Calcd. for $C_{21}H_{17}O_2F$ (M^+) requires 320.1213, Found: 320.1221.



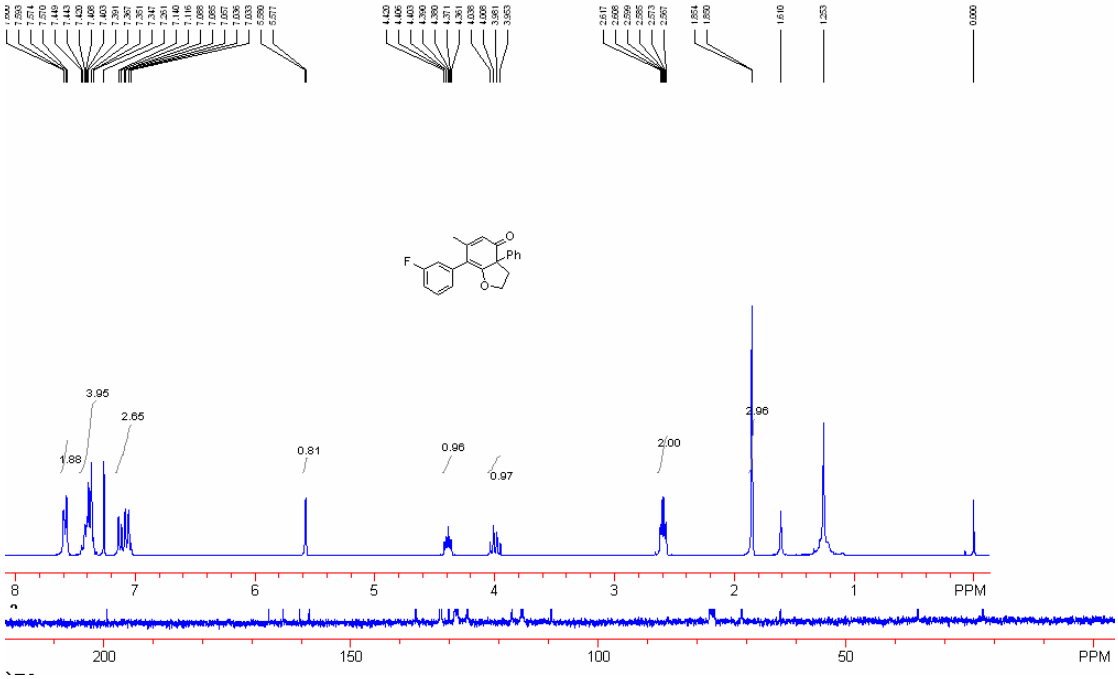


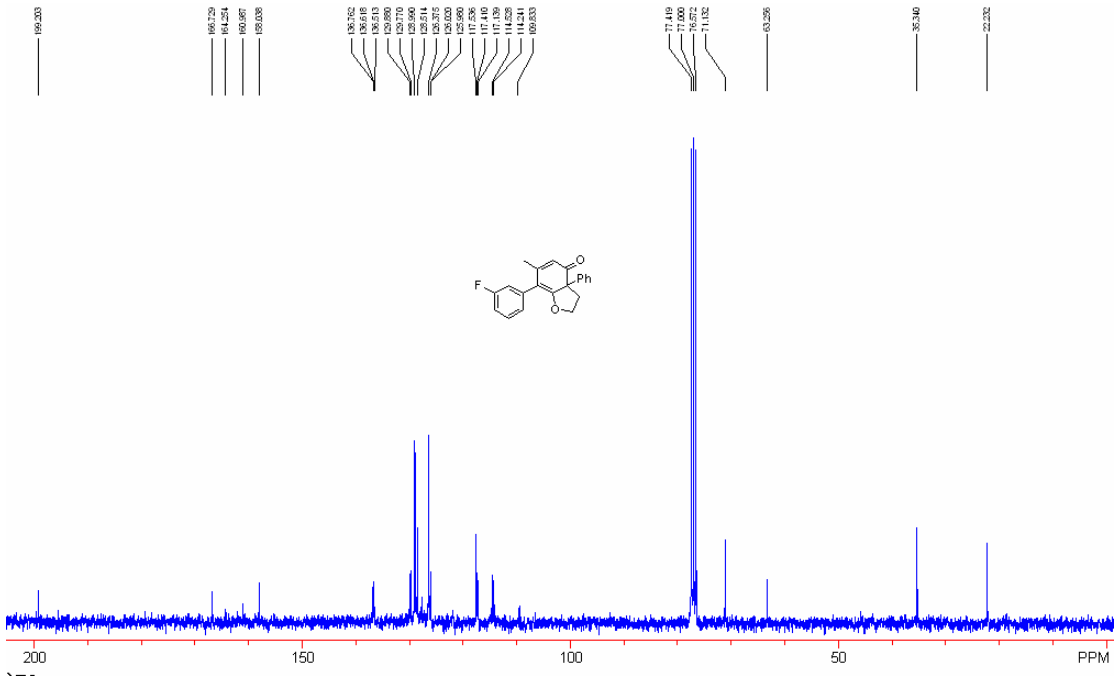


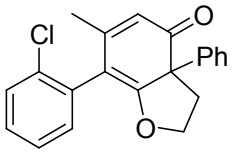
2g

7-(3-Fluorophenyl)-6-methyl-3a-phenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2g. A yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.85 (d, 3H, J = 1.2 Hz), 2.57-2.62 (m, 2H), 3.97 (dd, 1H, J = 15.7 Hz, J = 8.4 Hz), 4.38-4.40 (m, 1H), 5.58 (d, 1H, J = 1.2 Hz), 7.05-7.14 (m, 3H), 7.35-7.42 (m, 4H), 7.58 (dd, J = 7.8 Hz, J = 1.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 22.3, 35.3, 63.2, 71.1, 109.5, 114.4 (d, $J_{C\text{-}F}$ = 20.6 Hz), 117.3 (d, $J_{C\text{-}F}$ = 20.6 Hz), 117.6, 126.0 (d, $J_{C\text{-}F}$ = 2.9 Hz), 126.4, 128.5, 129.0, 129.8 (d, $J_{C\text{-}F}$ = 8.6 Hz), 136.6 (d, $J_{C\text{-}F}$ = 8.0 Hz), 136.8,

158.0, 163.0 (d, $J_{C\text{-}F}$ = 245.0 Hz), 166.7, 199.2; IR (CH_2Cl_2): ν 3061, 2962, 2924, 1723, 1670, 1539, 1377, 1265, 1031 cm^{-1}; MS (EI) m/z (%): 320 [M^+] (100), 192 (60.3), 123 (75.7), 105 (45.4), 95 (34.5), 77 (41.7), 44 (33.5), 43 (38.6); HRMS (EI) Calcd. for $C_{21}H_{17}O_2F$ (M^+) requires 320.1213, Found: 320.1226.



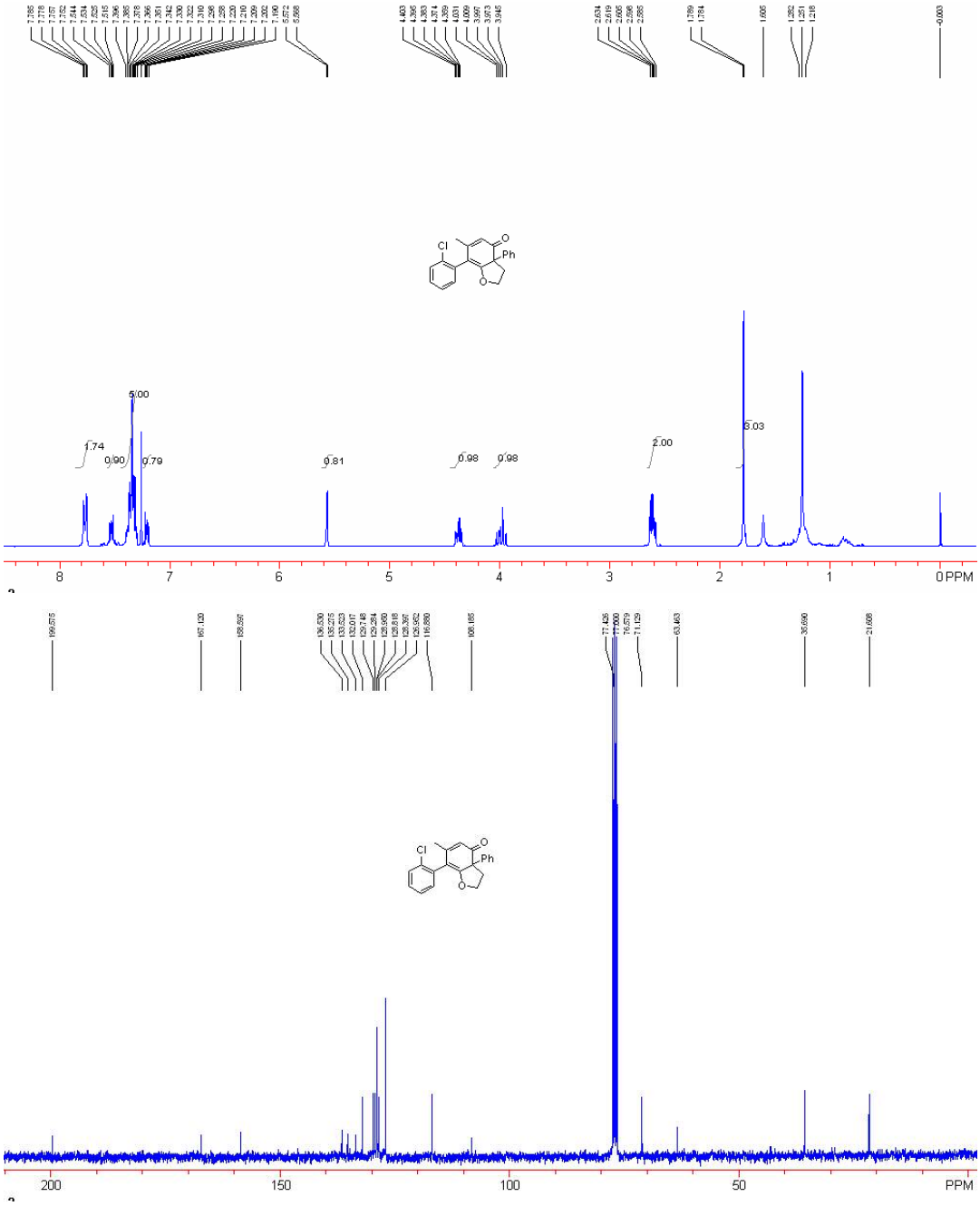


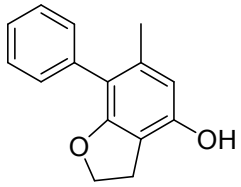


2h

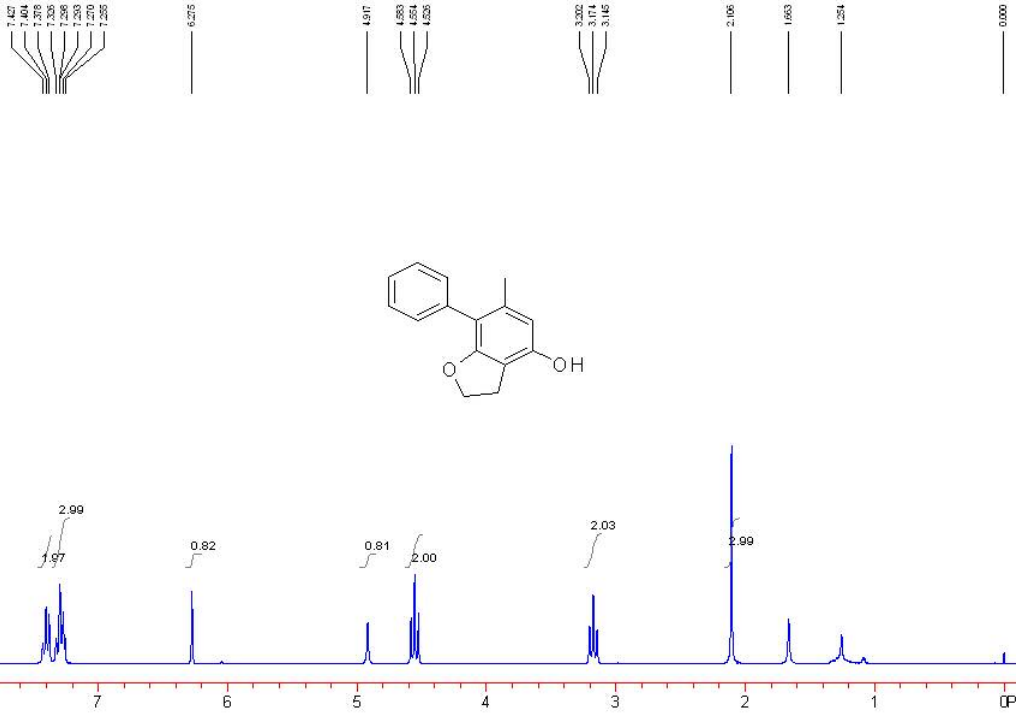
7-(2-Chlorophenyl)-6-methyl-3a-phenyl-3,3a-dihydrobenzofuran-4(2*H*)-one 2h. A yellow

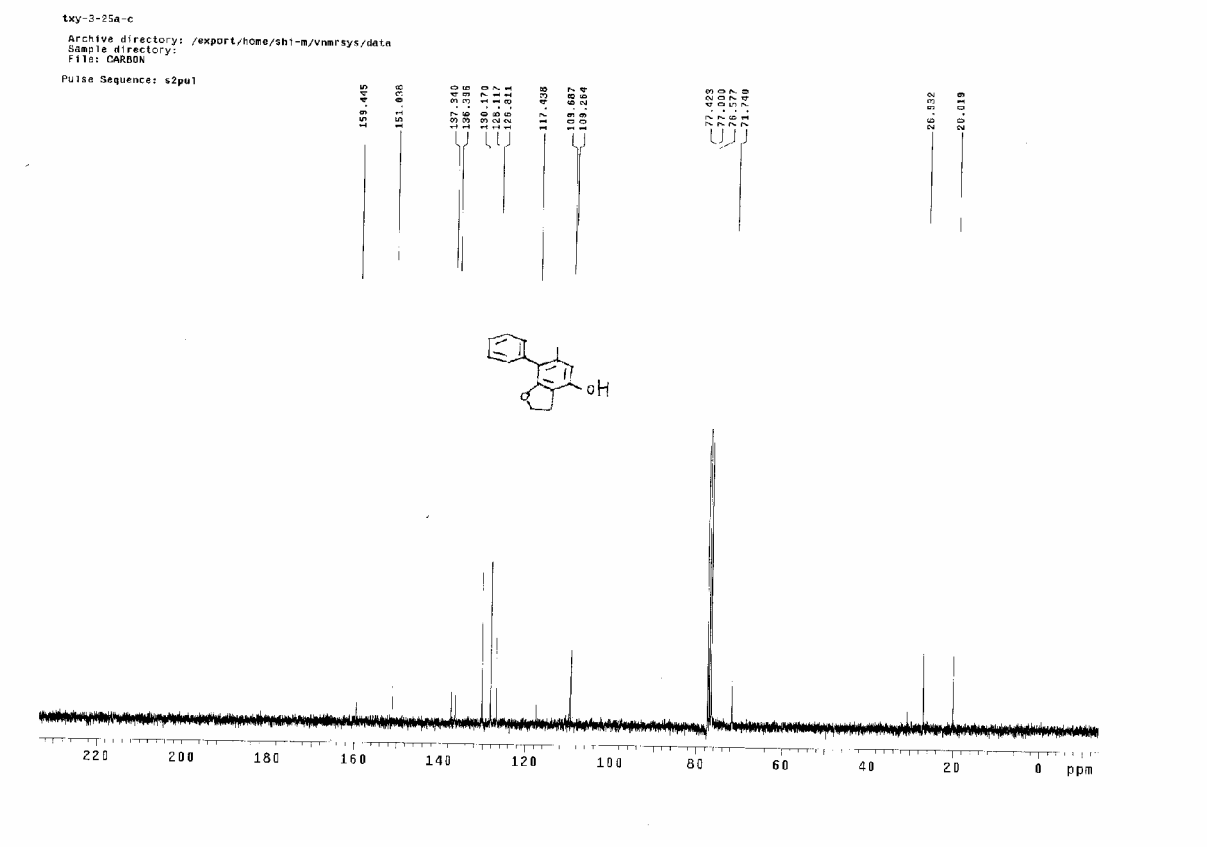
solid. m.p. 166-168 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz, TMS) δ 1.79 (d, *J* = 1.5 Hz, 3H), 2.58-2.64 (m, 2H), 3.96 (dd, 1H, *J* = 17.1 Hz, *J* = 8.7 Hz), 4.35-4.41 (m, 1H), 5.57 (d, *J* = 1.5 Hz, 1H), 7.19-7.22 (m, 1H), 7.31-7.39 (m, 5H), 7.57-7.55 (m, 1H), 7.77 (dd, *J* = 7.8 Hz, *J* = 2.1 Hz, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz, TMS) δ 21.6, 35.7, 63.5, 71.1, 108.2, 116.9, 126.9, 128.4, 128.8, 129.0, 129.3, 129.7, 132.0, 133.5, 135.3, 136.5, 158.6, 167.1, 199.6; IR (CH_2Cl_2): ν 3058, 2924, 2853, 1731, 1675, 1492, 1380, 1255, 1071, 762 cm^{-1}; MS (EI) m/z (%): 380 [M^+] (92.3), 382 (89.5), 301 (66.3), 273 (100), 245 (61.8), 215 (57.7), 202 (49.0), 115 (58.8); HRMS (EI) Calcd. for $C_{21}H_{17}O_2Br$ (M^+) requires 380.0412, Found: 380.0417.

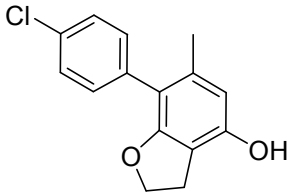




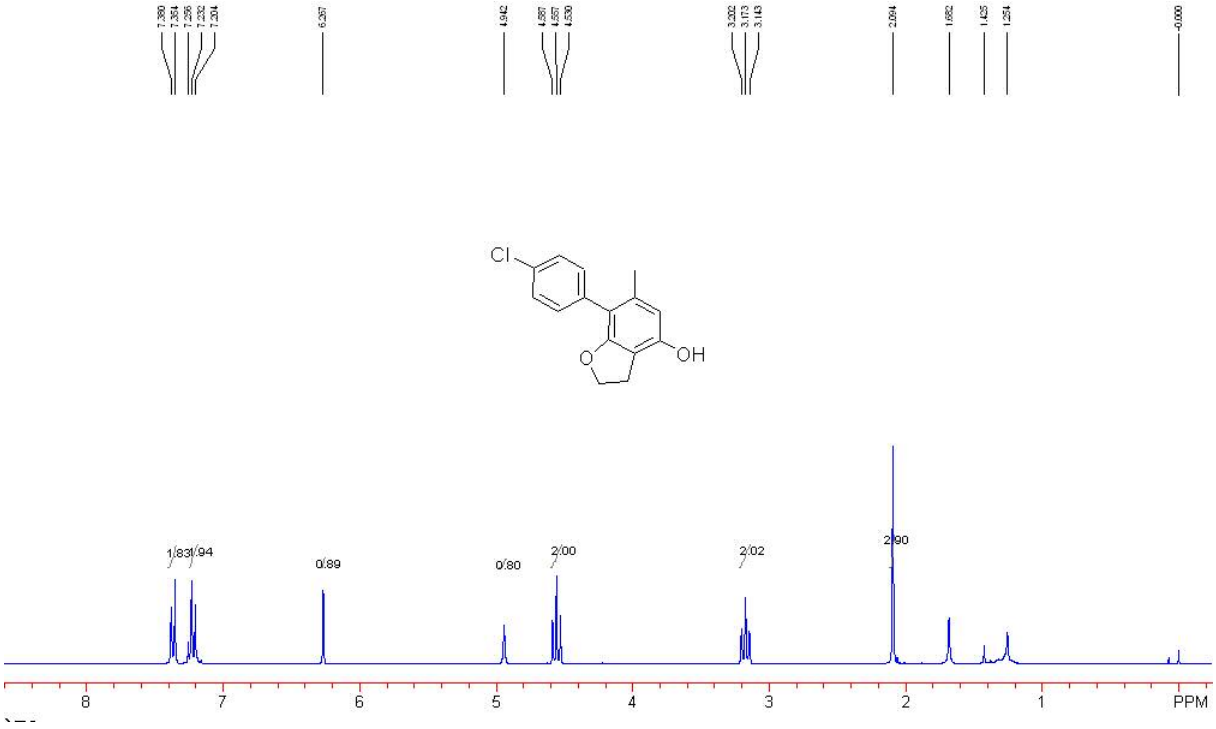
6-Methyl-7-phenyl-2,3-dihydrobenzofuran-4-ol (3a). A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.11 (s, 3H), 3.17 (t, *J* = 8.6 Hz, 2H), 4.56 (t, *J* = 8.6 Hz, 2H), 4.92 (s, 1H), 6.28 (s, 1H), 7.26-7.33 (m, 3H), 7.38-7.43 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0, 27.0, 71.7, 109.3, 109.7, 117.4, 126.8, 128.1, 130.2, 136.4, 137.3, 151.0, 159.4; IR (CH_2Cl_2): ν 3411, 2957, 2856, 1706, 1619, 1453, 1421, 1306, 1126, 1048, 702 cm^{-1}; MS (EI) *m/z* (%): 226 (100) [M^+], 225 (32.2), 211 (26.5), 227 (17.6), 197 (14.9), 165 (10.8), 183 (10.4), 115 (10.2); HRMS (EI) Calcd. for $C_{15}H_{14}O_2$ (M^+) requires 226.0994, Found: 306.0996.

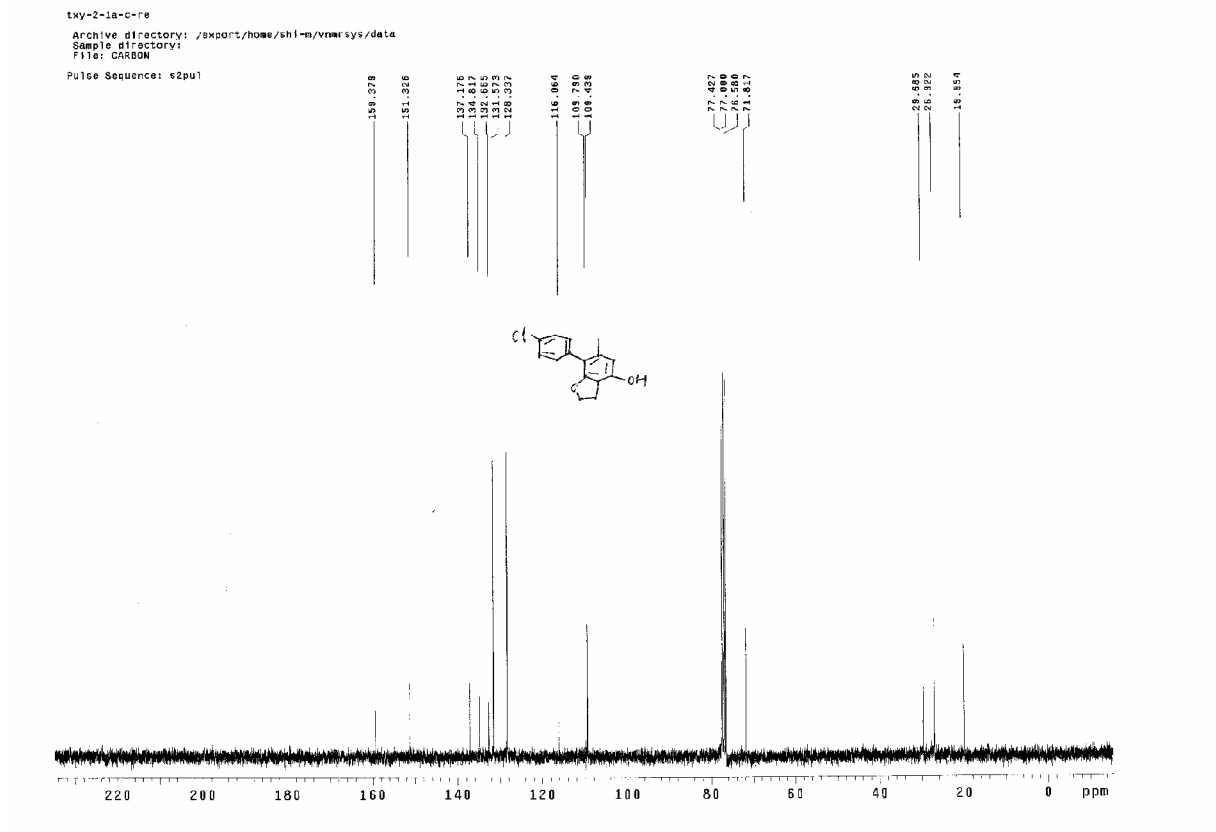


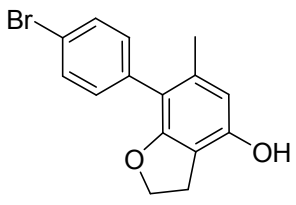




7-(4-Chlorophenyl)-6-methyl-2,3-dihydrobenzofuran-4-ol (3b). A yellow solid. m.p. 194-196 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.09 (s, 3H), 3.17 (t, J = 8.7 Hz, 2H), 4.56 (t, J = 8.7 Hz, 2H), 4.94 (s, 1H), 6.27 (s, 1H), 7.22 (d, J = 8.1 Hz, 2H), 7.36 (d, J = 8.1 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0, 27.0, 71.8, 109.4, 109.8, 116.1, 128.3, 131.6, 132.7, 134.8, 137.2, 151.3, 159.4; IR (CH_2Cl_2): ν 3391, 2969, 2919, 2856, 1619, 1474, 1419, 1311, 1128, 1037, 822 cm^{-1}; MS (EI) *m/z* (%): 260 (100) [M^+], 262 (32.3), 225 (30.0), 261 (18.2), 197 (11.7), 76 (10.1), 224 (8.6), 245 (7.8); HRMS (EI) Calcd. for $C_{15}H_{13}ClO_2$ (M^+) requires 260.0604, 260.0626, Found: 260.0613.

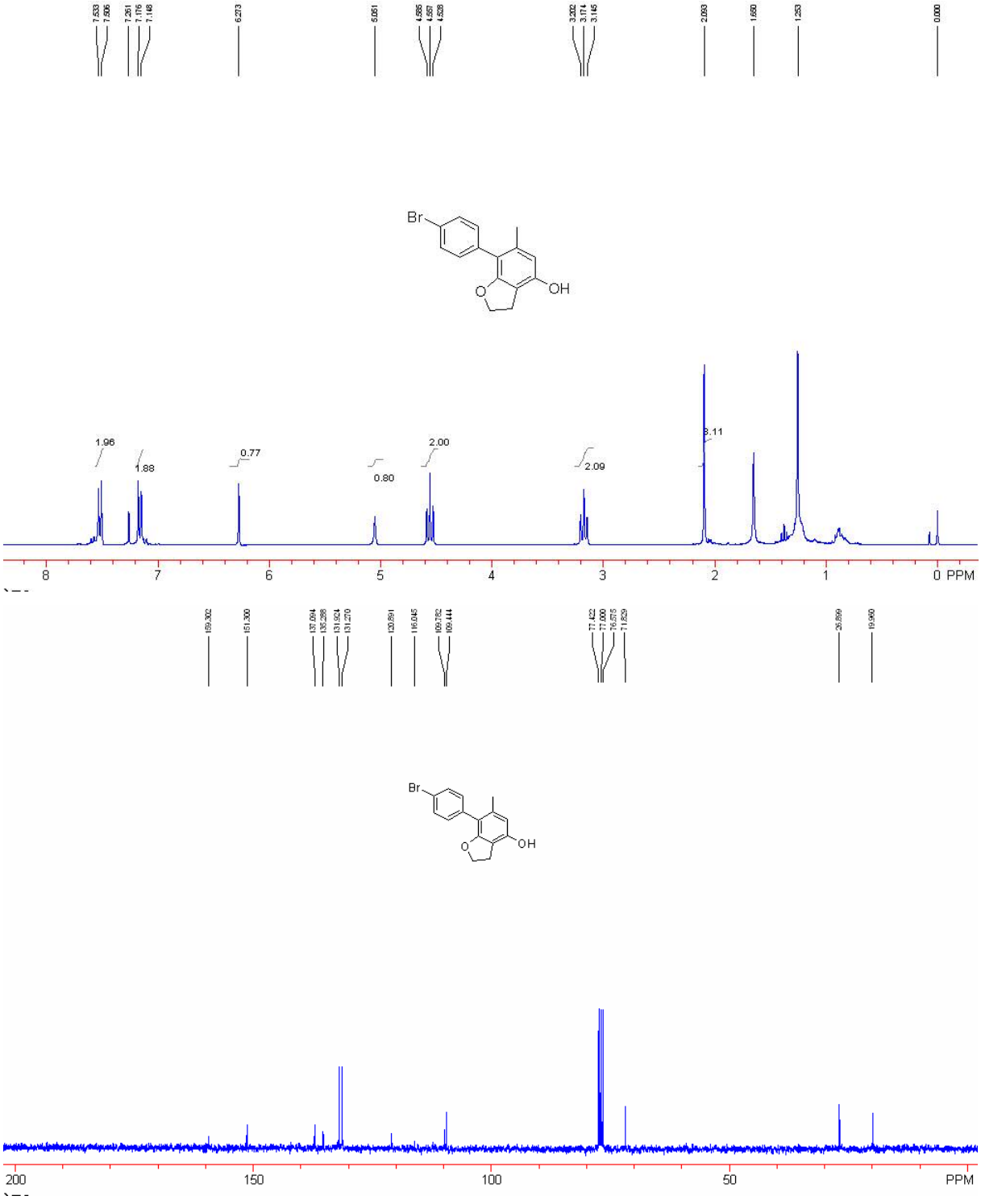


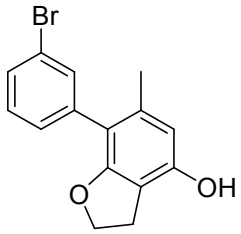




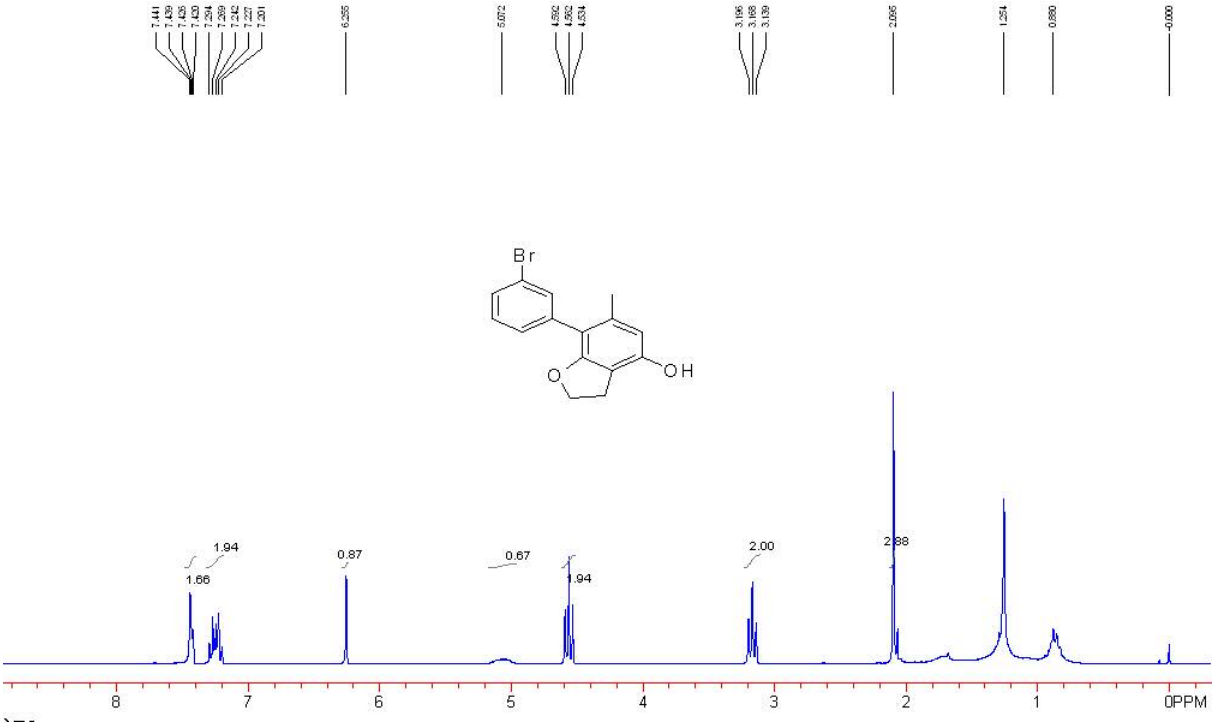
7-(4-Bromophenyl)-6-methyl-2,3-dihydrobenzofuran-4-ol (3c). A yellow solid. m.p.

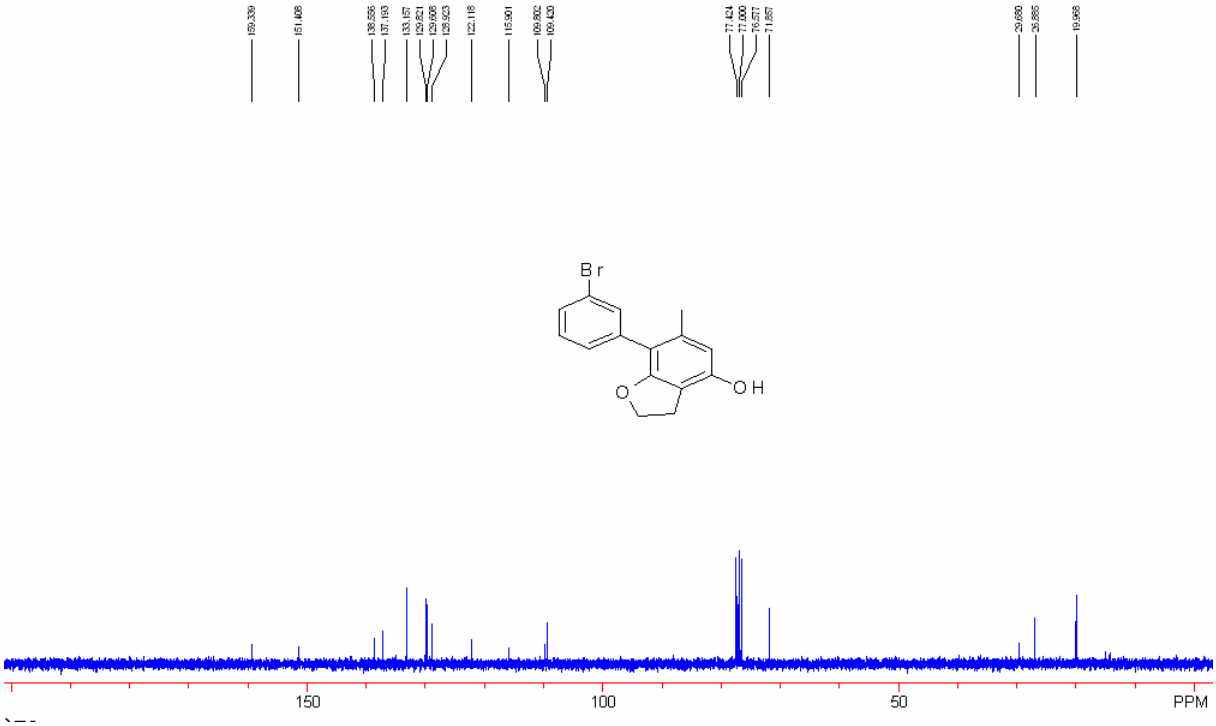
178-180 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.10 (s, 3H), 3.18 (t, *J* = 8.7 Hz, 2H), 4.56 (t, *J* = 8.7 Hz, 2H), 4.93 (s, 1H), 6.28 (s, 1H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.52 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0, 26.9, 71.8, 109.4, 109.8, 116.0, 120.9, 131.3, 131.9, 135.3, 137.1, 151.3, 159.3; IR (CH_2Cl_2): ν 3403, 2957, 2925, 2855, 1708, 1618, 1481, 1453, 1308, 1126, 1048, 818 cm^{-1}; MS (EI) *m/z* (%): 306 (100) [M^{+2}], 304 (98.3) [M^{+}], 225 (33.8), 149 (47.9), 71 (26.1), 57 (37.8), 43 (40.8), 41 (27.8); HRMS (EI) Calcd. for $C_{15}H_{13}BrO_2$ (M^{+}) requires 304.0099, Found: 304.0106.

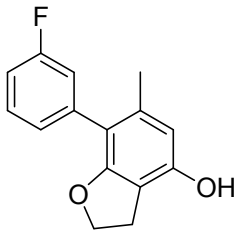




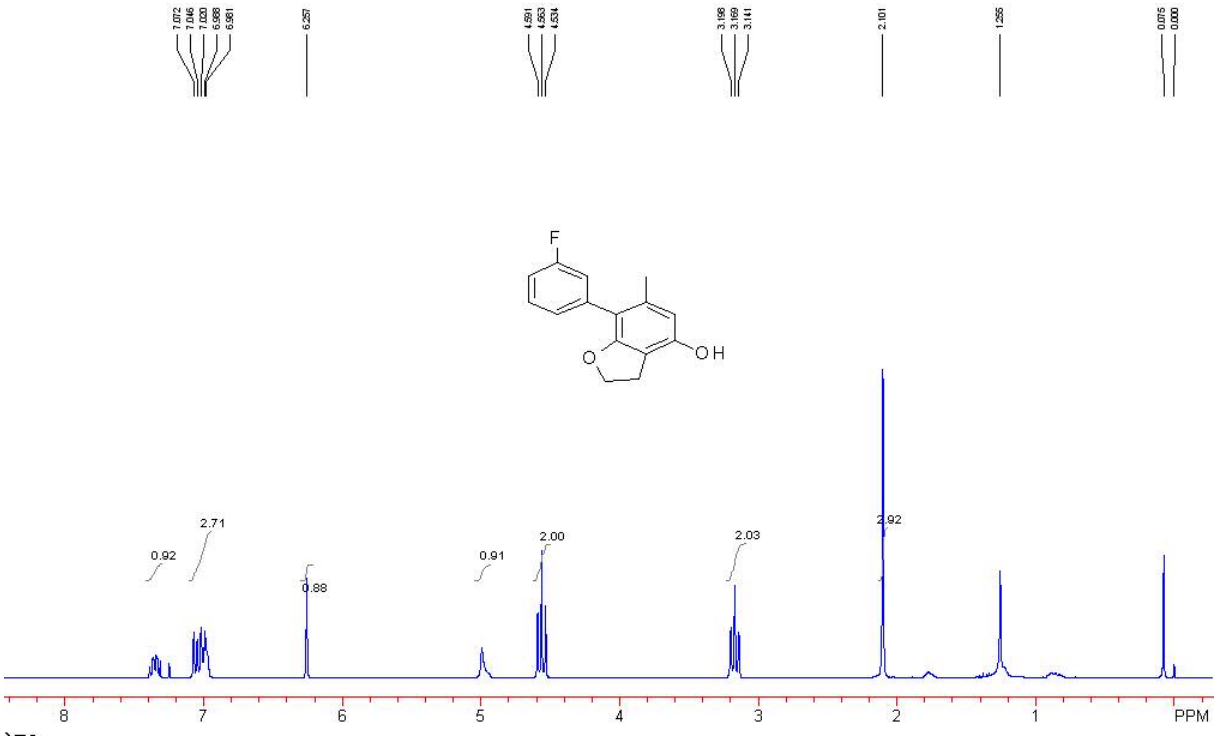
7-(3-Bromophenyl)-6-methyl-2,3-dihydrobenzofuran-4-ol (3d). A yellow solid. m.p. 164-166 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.10 (s, 3H), 3.17 (t, *J* = 8.7 Hz, 2H), 4.57 (t, *J* = 8.7 Hz, 2H), 5.05 (s, 1H), 6.25 (s, 1H), 7.23-7.25 (m, 2H), 7.42-7.44 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0, 26.9, 71.9, 109.4, 109.8, 116.0 122.1, 128.9, 129.6, 129.8, 133.2, 137.2, 138.6, 151.4, 159.3; IR (CH_2Cl_2): ν 3397, 2955, 2924, 2855, 1620, 1453, 1308, 1127, 1024, 784, 692 cm^{-1}; MS (EI) *m/z* (%): 57 (100), 43 (94.9), 306 (74.0), 304 (73.6) [M^+], 41 (68.5), 55 (68.1), 71 (65.2), 69 (57.2); HRMS (EI) Calcd. for $C_{15}H_{13}BrO_2$ (M^+) requires 304.0099, Found: 304.0102.

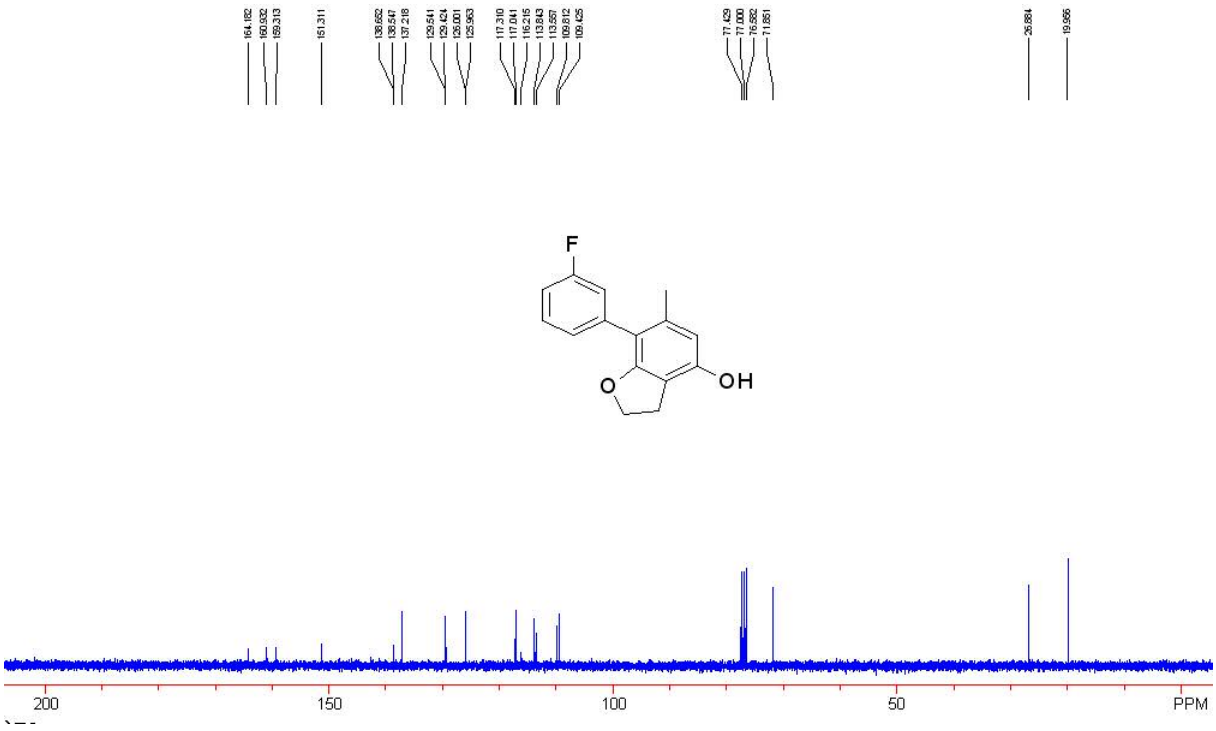


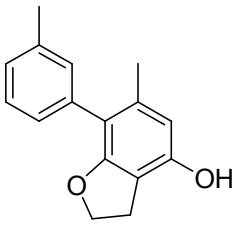




7-(3-Fluorophenyl)-6-methyl-2,3-dihydrobenzofuran-4-ol (3e). A yellow solid. m.p. 124-126 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.10 (s, 3H), 3.17 (t, J = 8.7 Hz, 2H), 4.56 (t, J = 8.7 Hz, 2H), 4.99 (s, 1H), 6.26 (s, 1H), 6.97-7.07 (m, 3H) 7.25-7.37 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0, 26.9, 71.8, 109.4, 109.8, 113.7 (d, $J_{C\text{-}F}$ = 20.8 Hz), 116.2, 117.2 (d, $J_{C\text{-}F}$ = 20.8 Hz), 125.9 (d, $J_{C\text{-}F}$ = 2.8 Hz), 129.5 (d, $J_{C\text{-}F}$ = 8.4 Hz), 137.2, 138.6 (d, $J_{C\text{-}F}$ = 7.8 Hz), 151.3, 159.3, 162.5 (d, $J_{C\text{-}F}$ = 244.1 Hz); ^{19}F NMR ($CDCl_3$, 282 MHz, $CFCl_3$): δ -119.3; IR (CH_2Cl_2): ν 3423, 2963, 2924, 2856, 1702, 1613, 1479, 1453, 1420, 1310, 1262, 1184, 1118, 820 cm^{-1}; MS (EI) *m/z* (%): 244 (100) [M^{+}], 243 (27.6), 229 (18.3), 245 (15.0), 215 (13.2), 183 (7.7), 201 (8.9), 183 (7.7), 173 (6.7); HRMS (EI) Calcd. for $C_{15}H_{13}FO_2$ (M^{+}) requires 244.0900, Found: 244.0899.

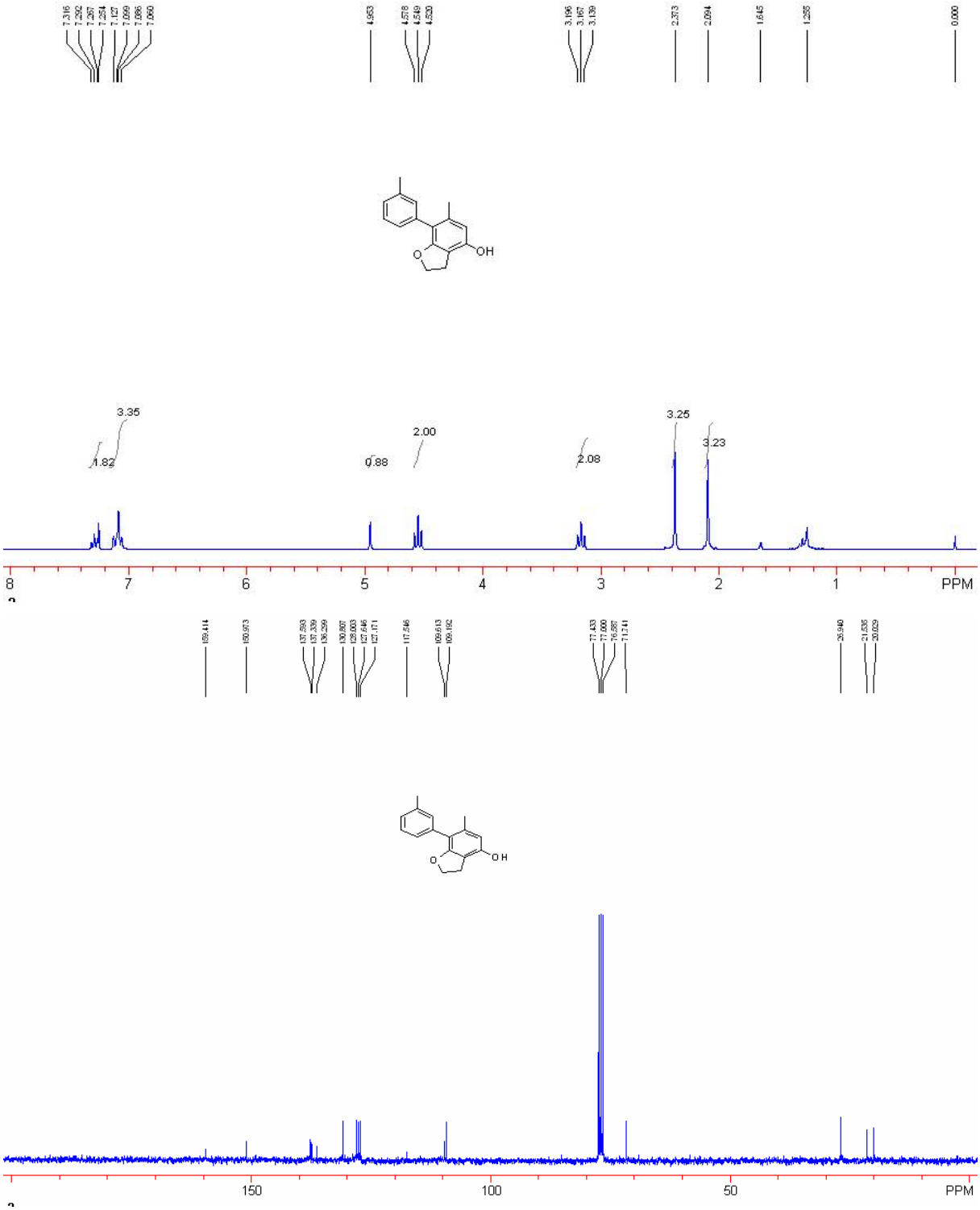


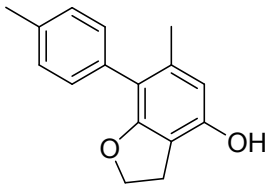




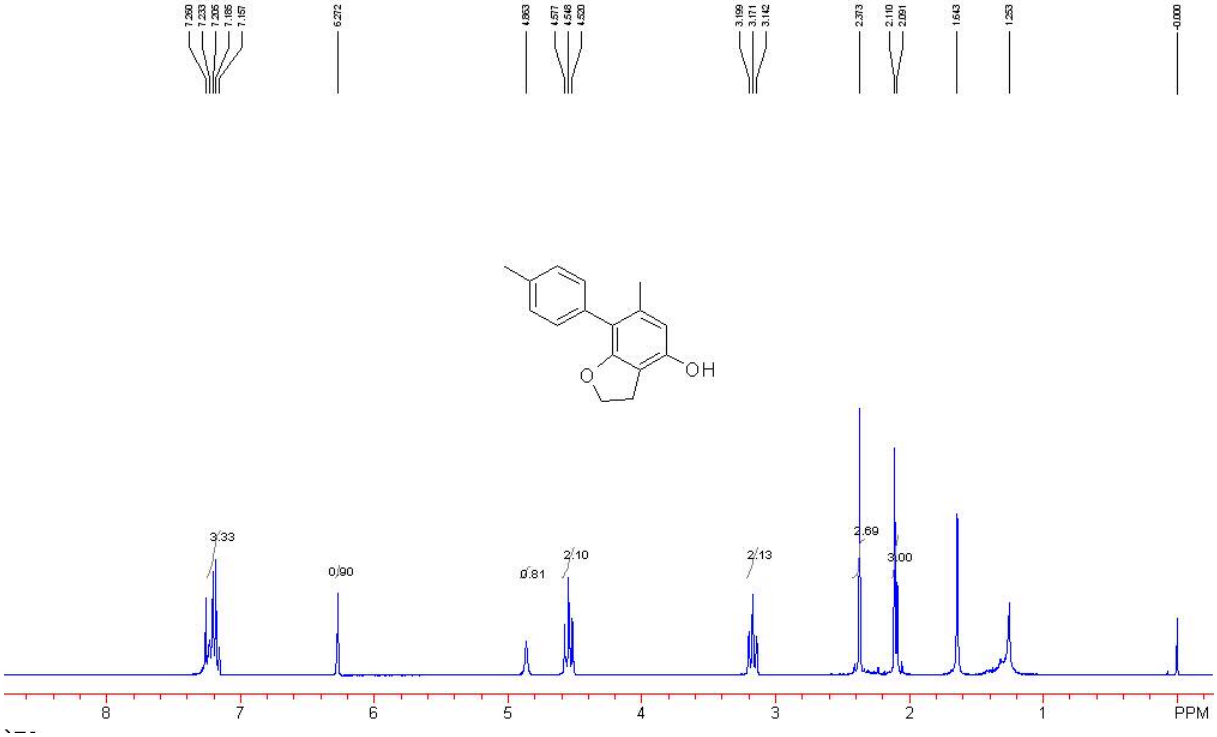
6-Methyl-7-*m*-tolyl-2,3-dihydrobenzofuran-4-ol (3f). A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.09 (s, 3H), 2.37 (s, 3H), 3.17 (t, *J* = 8.7 Hz, 2H), 4.55 (t, *J* = 8.7 Hz, 2H), 4.95 (s, 1H), 7.06-7.13 (m, 3H), 7.25-7.32 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0,

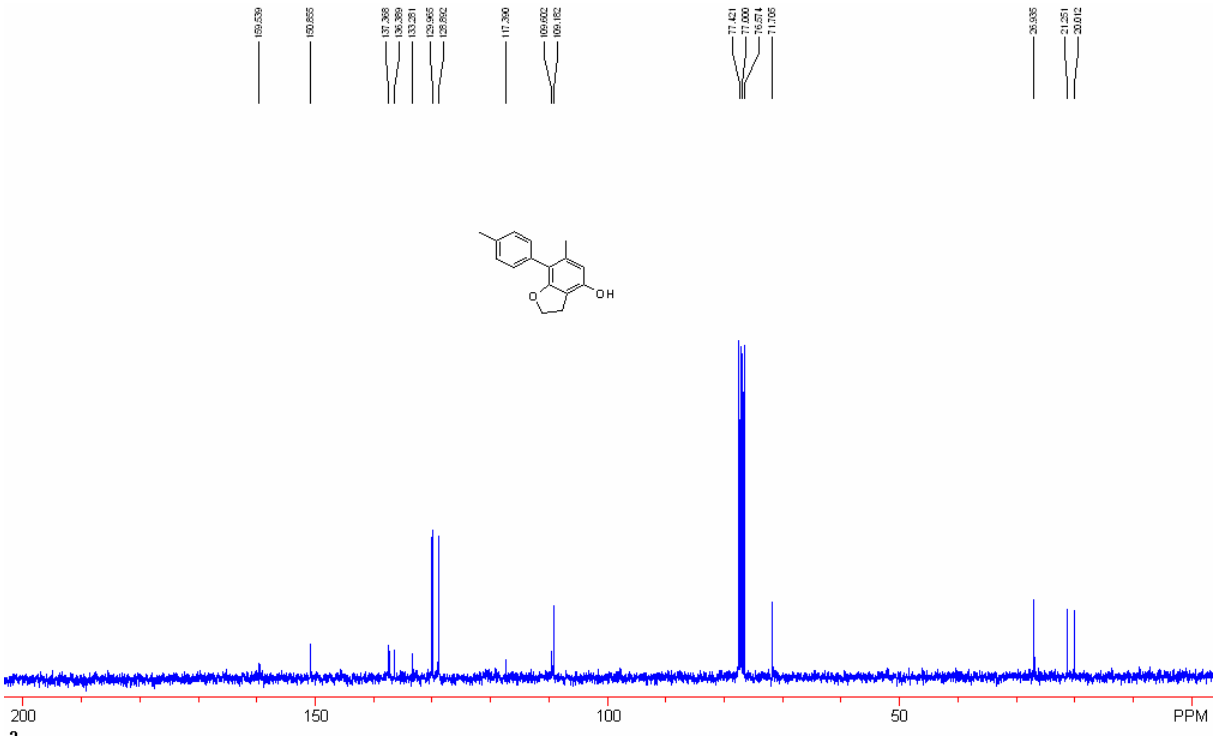
21.3, 26.9, 71.7, 109.2, 109.6, 117.5, 127.2, 127.6, 128.0, 130.8, 136.3, 137.3, 137.6, 151.0, 159.4; IR (CH_2Cl_2): ν 3416, 2959, 2922, 2859, 1731, 1619, 1478, 1453, 1367, 1312, 1125, 1052, 784 cm^{-1}; MS (EI) *m/z* (%): 240 (100) [M^+], 241 (17.9), 225 (35.1), 197 (8.0), 181 (7.7), 165 (8.1), 128 (10.8), 115 (10.9); HRMS (EI) Calcd. for $C_{16}H_{16}O_2$ (M^+) requires 240.1150, Found: 240.1160.

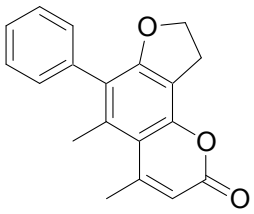




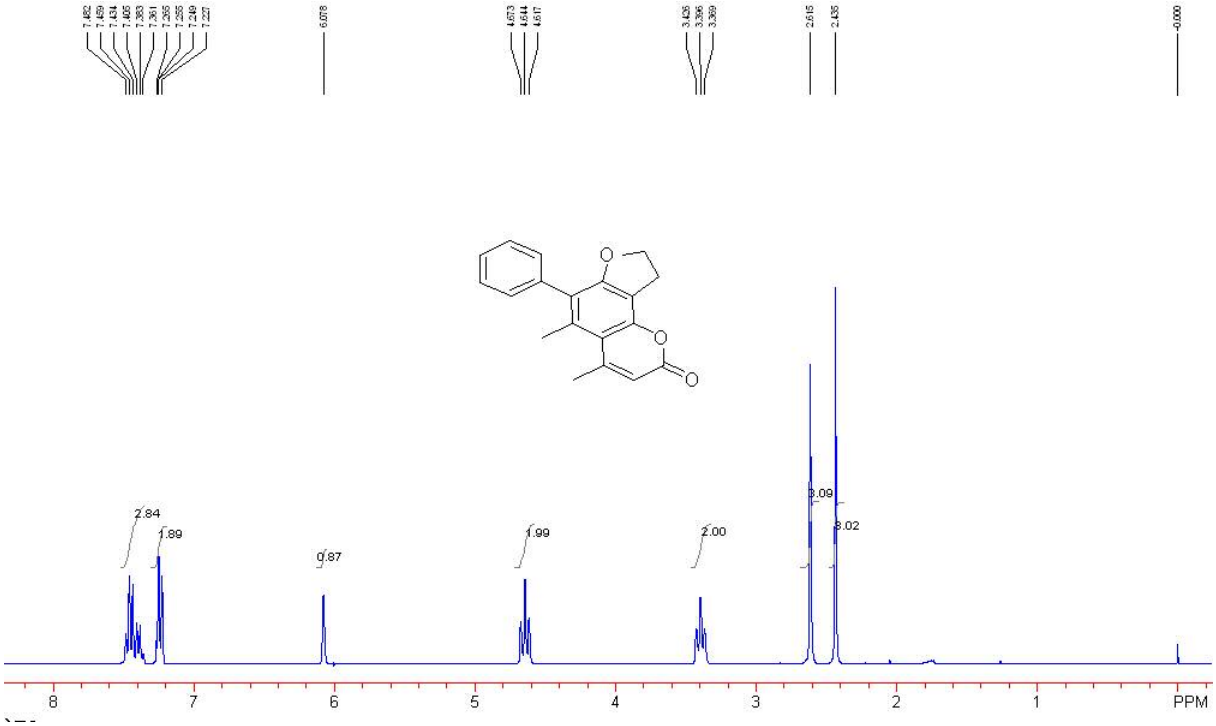
6-Methyl-7-*p*-tolyl-2,3-dihydrobenzofuran-4-ol (3g). A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.10 (s, 3H), 2.38 (s, 3H), 3.17 (t, *J* = 8.7 Hz, 2H), 4.55 (t, *J* = 8.7 Hz, 2H), 4.86 (s, 1H), 6.27 (s, 1H), 7.16-7.26 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.0, 21.3, 26.9, 71.7, 109.2, 109.6, 117.4, 128.9, 130.0, 133.3, 136.4, 137.4, 150.9, 159.6; IR (CH_2Cl_2): ν 3412, 2958, 2922, 2858, 1706, 1619, 1489, 1452, 1424, 1304, 1125, 1047, 815 cm^{-1}; MS (EI) *m/z* (%): 240 (100) [M$^+$], 225 (48.3), 241 (19.5), 239 (16.9), 197 (11.6), 165 (9.3), 211 (9.2), 115 (9.1); HRMS (EI) Calcd. for $C_{16}H_{16}O_2$ (M$^+$) requires 240.1150, Found: 240.1154.







4,5-Dimethyl-6-phenyl-3,4,8,9-tetrahydrofuro[2,3-*h*]chromen-2-one (4a). A white solid. m.p. 222-223 °C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.44 (s, 3H), 2.62 (d, *J* = 0.9 Hz, 3H), 3.40 (t, *J* = 9.0 Hz, 2H), 4.65 (t, *J* = 9.0 Hz, 2H), 6.08 (d, *J* = 0.9 Hz, 1H), 7.23-7.26 (m, 2H), 7.38-7.48 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 20.8, 26.1, 27.3, 72.7, 111.7, 113.2, 113.8, 123.4, 127.6, 128.0, 128.4, 130.1, 130.2, 135.9, 136.1, 151.4, 155.4, 160.7, 161.3; IR (CH_2Cl_2): ν 2974, 2910, 1812, 1728, 1607, 1577, 1295, 1203, 1124, 1084, 1035 cm^{-1}; MS (EI) *m/z* (%): 292 (100) [M^{+}], 264 (54.5), 263 (19.9), 226 (69.6), 225 (20.8), 105 (34.0), 77 (31.7), 51 (19.9); Anal. calcd. for $C_{19}H_{16}O_3$: C, 78.06%; H, 5.52%. Found: C, 77.79%; H, 5.50%.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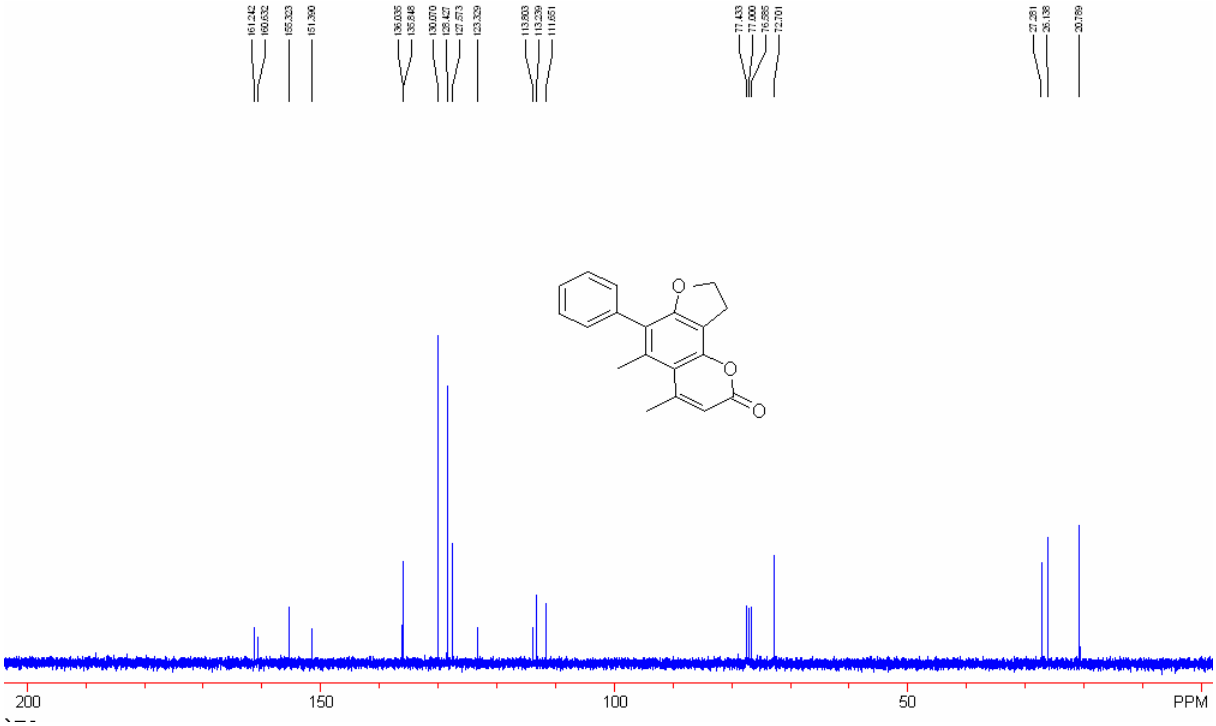
7.434
7.405
7.383
7.361
7.295
7.269
7.227
6.078
4.673
4.644
4.617
3.425
3.396
3.369
2.615
2.435
0.000
2.84
1.89
0.87
1.99
2.00
3.09
3.02
8
7
6
5
4
3
2
1
PPM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130.035
130.070
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123.329
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72.701
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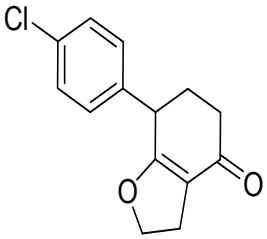
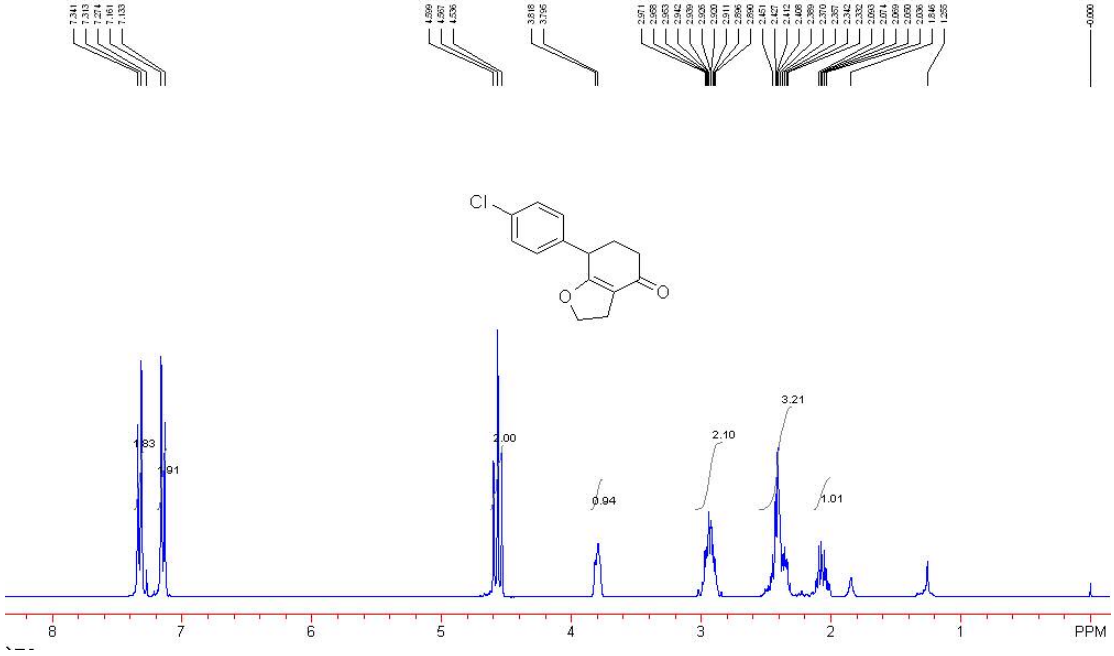
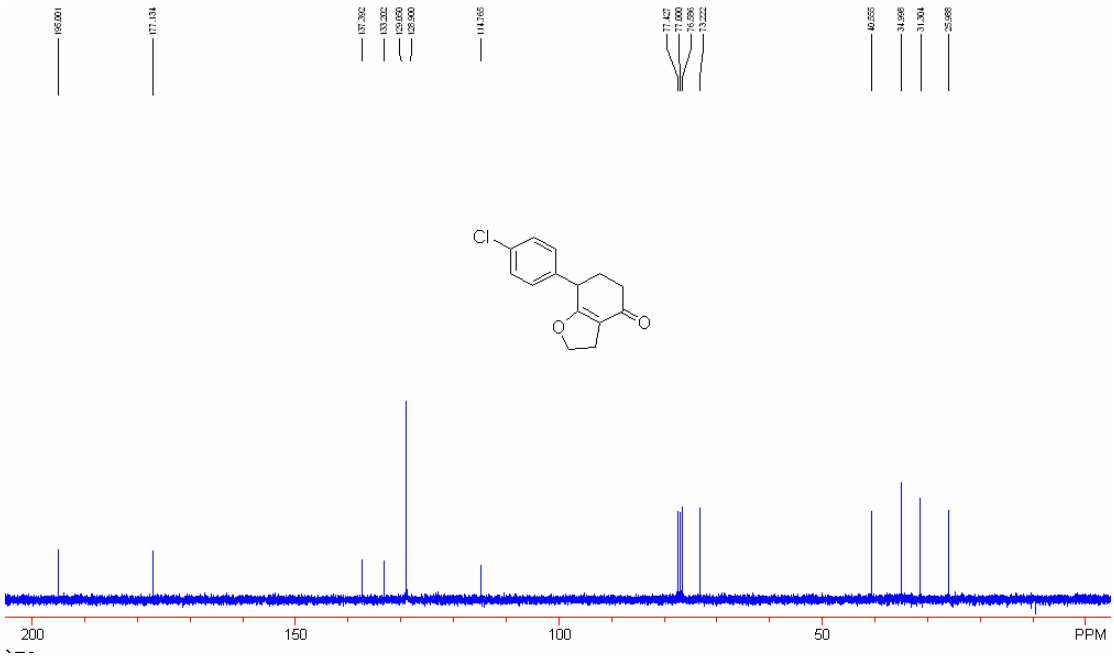
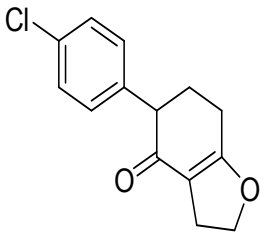
200
150
100
50
PPM



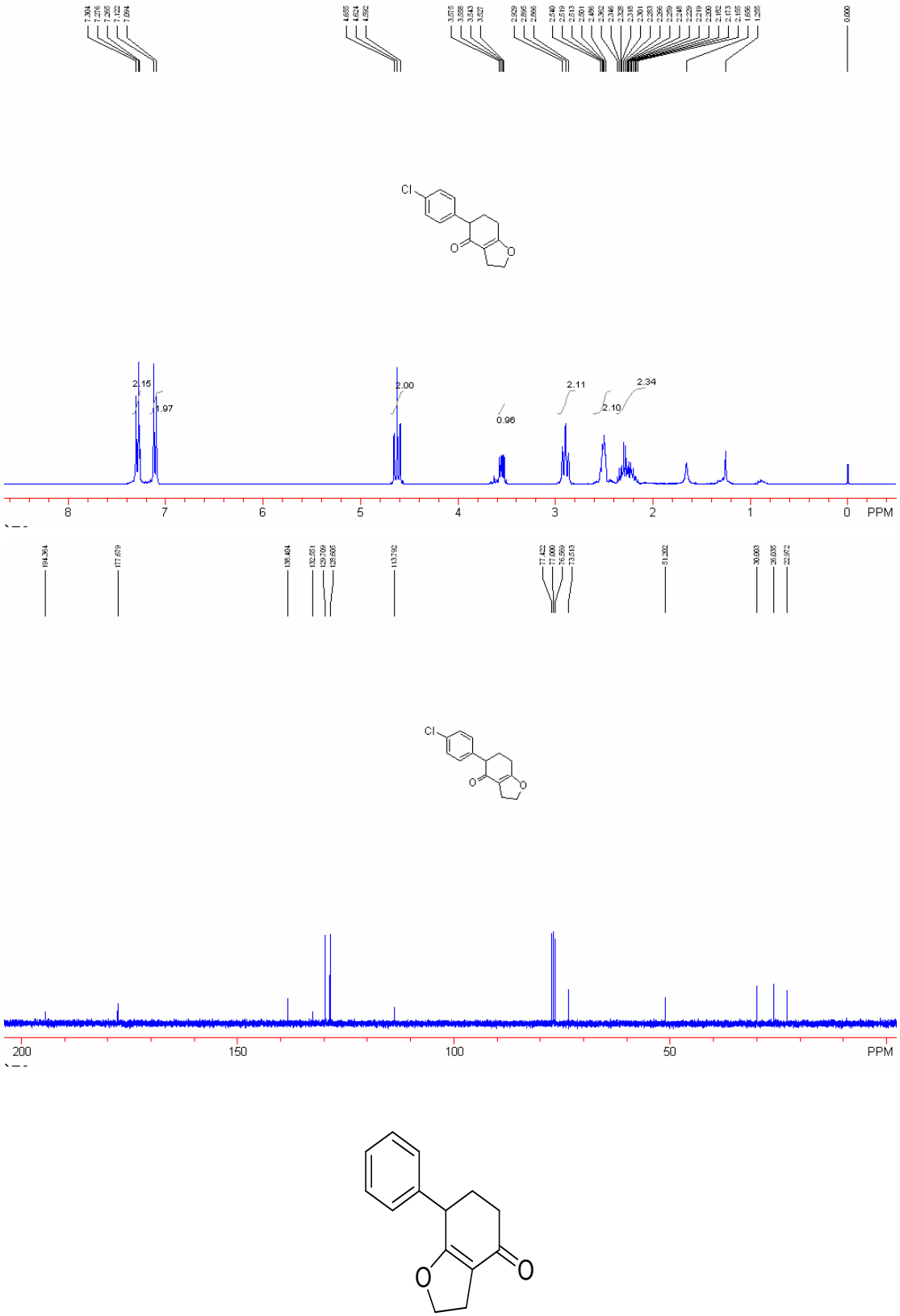
7-(4-Chlorophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5a. A yellow solid. m.p. 122-124 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.85-2.09 (m, 1H), 2.34-2.45 (m, 3H), 2.89-2.97 (m, 2H), 3.79-3.82 (m, 1H), 4.57 (t, *J* = 9.3 Hz, 2H), 7.15 (d, *J* = 8.7 Hz, 2H), 7.33 (d, *J* = 8.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.0, 31.3, 35.0, 40.5, 73.2, 114.8, 128.9, 129.1, 133.2, 137.4, 177.1, 195.0; IR (CH_2Cl_2): ν 2945, 2870, 1650, 1627, 1491, 1397, 1231, 1089, 961, 831 cm^{-1}; MS (EI) *m/z* (%): 248 [M^+] (52.6), 250 (16.1), 247 (100.0), 139 (83.1), 110 (39.3), 69 (69.3), 57 (71.3), 55 (80.2); Anal. calcd. for $C_{14}H_{13}ClO_2 \cdot 1/2H_2O$: C, 65.25%; H, 5.48%. Found: C, 64.94%; H, 5.42%.





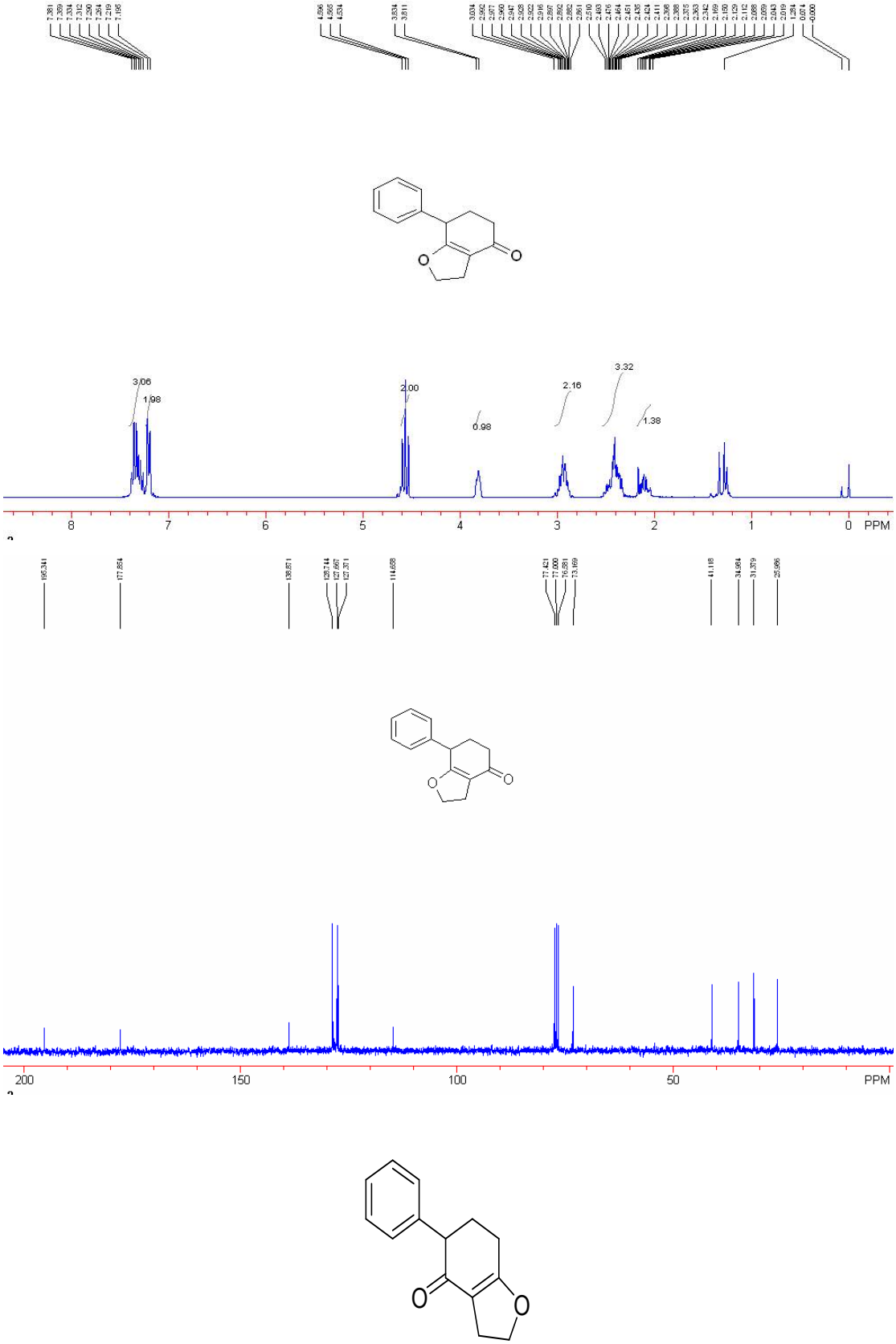


5-(4-Chlorophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6a. A yellow solid. m.p. 135-137 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.20-2.25 (m, 2H), 2.49-2.54 (m, 2H), 2.90 (t, J = 9.0 Hz, 2H), 3.55 (dd, J = 9.0 Hz, J = 7.8 Hz, 1H), 4.63 (t, J = 9.0 Hz, 2H), 7.11 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.0, 26.0, 30.0, 51.2, 73.5, 113.8, 128.6, 129.7, 132.6, 138.4, 177.7, 194.4; IR (CH_2Cl_2): ν 2927, 2870, 1735, 1654, 1638, 1629, 1491, 1407, 1230, 1092, 945, 831; MS (EI) *m/z* (%): 248 [M^+] (15.7), 250 (5.7), 110 (100), 86 (10.0), 84 (16.9), 51 (13.5), 49 (21.7), 44 (15.2); HRMS (EI) Calcd. for $C_{14}H_{13}ClO_2$ (M^+) requires 248.0604, Found: 248.0610.



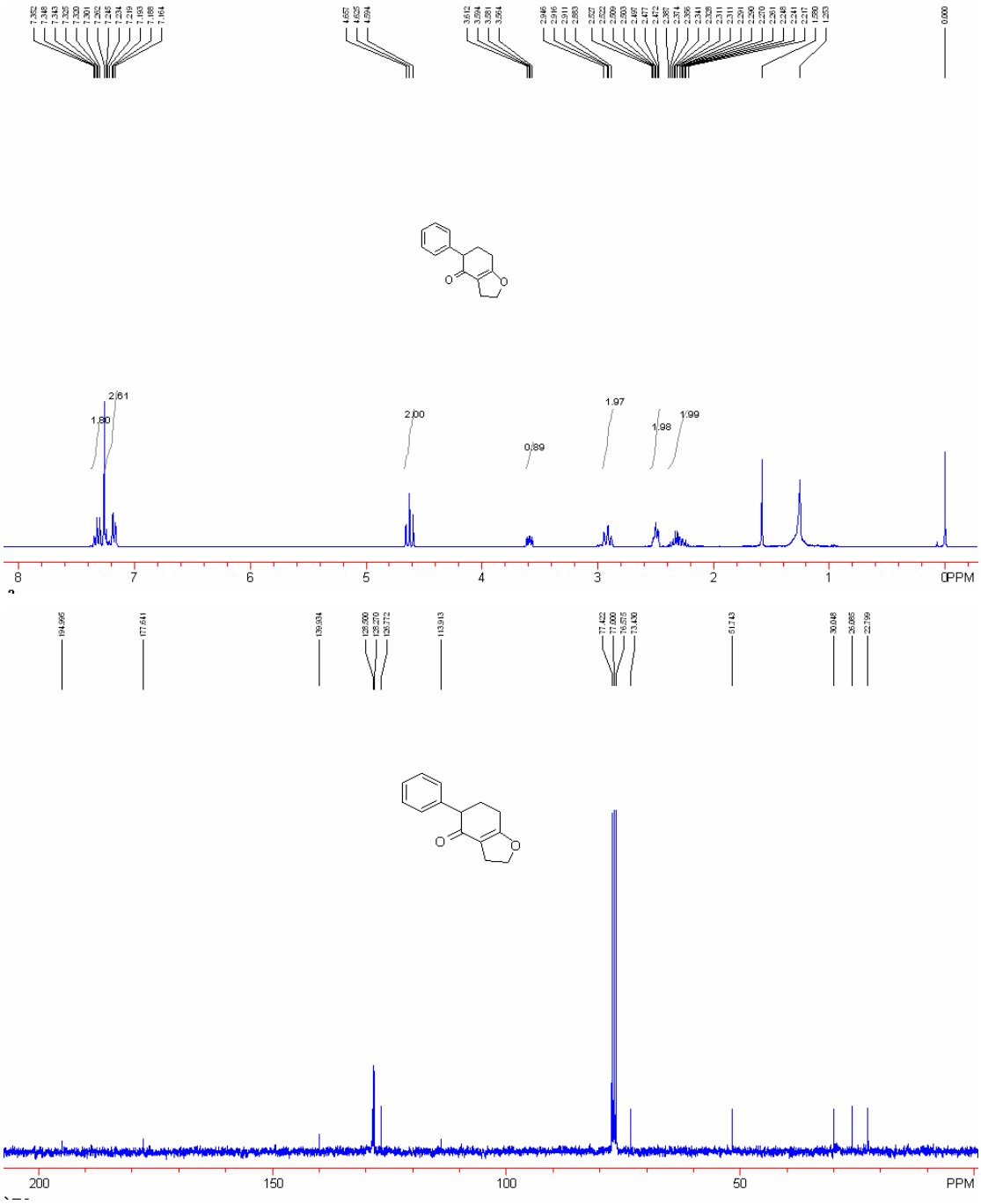
7-Phenyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5b. A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.02-2.17 (m, 1H), 2.34-2.51 (m, 3H), 2.86-2.03 (m, 2H), 3.81-3.83 (m, 1H), 4.57 (t, *J* = 9.3 Hz, 2H), 7.22 (d, *J* = 7.2 Hz, 2H), 7.26-7.38 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.0, 31.4, 35.0, 41.1, 73.2, 114.7, 127.4, 127.7, 128.8, 138.9, 177.9, 195.4; IR (CH_2Cl_2): ν 2953, 2926, 1732, 1651, 1599, 1494, 1409, 1235, 1078, 964, 820 cm^{-1}; MS (EI)

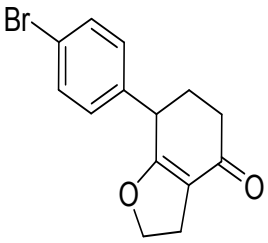
m/z (%): 214 [M^+] (39.8), 186 (40.9), 110 (100.0), 105 (68.6), 77 (50.7), 55 (22.1), 51 (26.3), 43 (17.3); HRMS (EI) Calcd. for $C_{14}H_{14}O_2$ (M^+) requires 214.0994, Found: 214.1010.



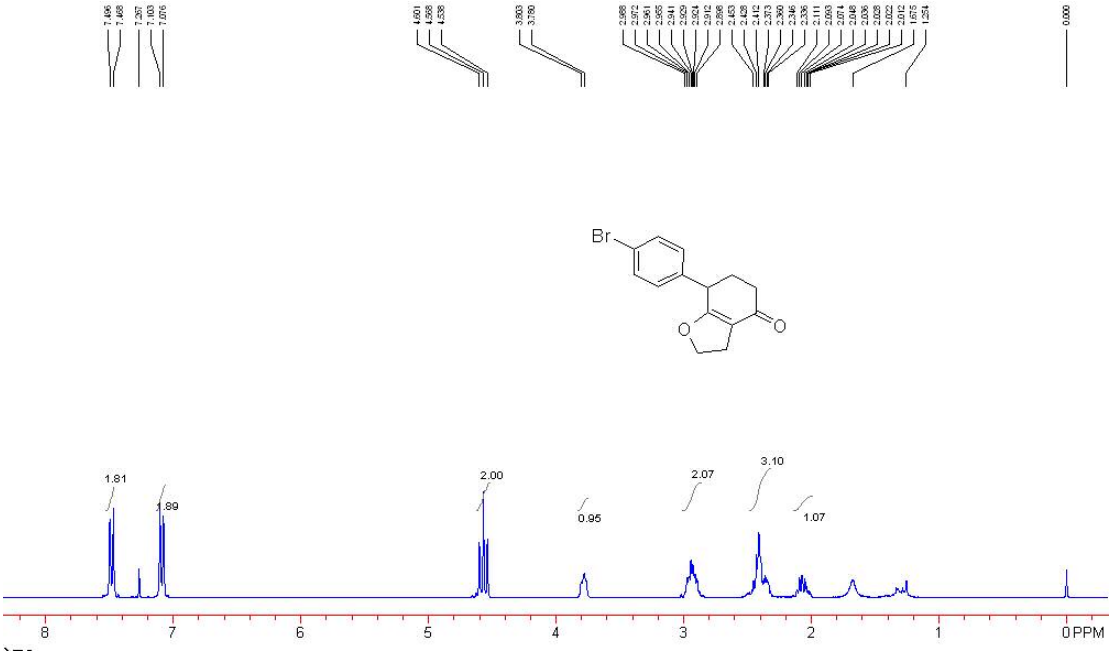
5-Phenyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6b. A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.23-2.39 (m, 2H), 2.47-2.52 (m, 2H), 2.88-2.95 (m, 2H), 3.58 (dd, *J* = 9.0

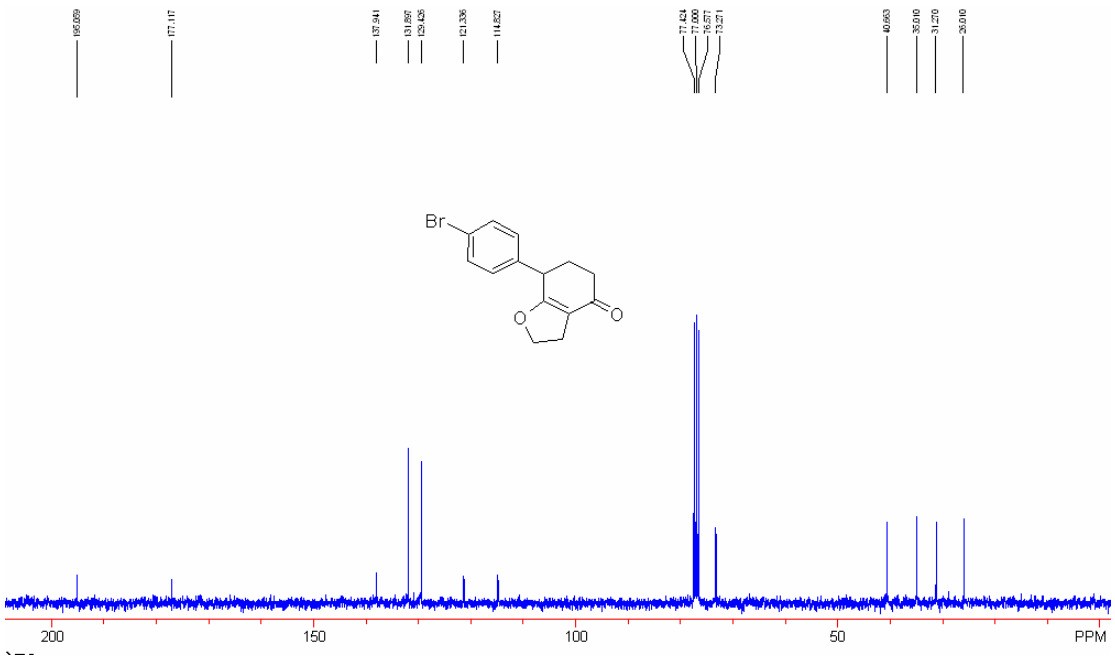
Hz, J = 7.8 Hz, 1H), 4.63 (t, J = 9.6 Hz, 2H), 7.17-7.25 (m, 2H), 7.30-7.35 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 22.8, 26.1, 30.0, 51.7, 73.4, 113.9, 126.8, 128.3, 128.5, 139.9, 177.6, 195.0; IR (CH_2Cl_2): ν 2961, 2853, 1738, 1683, 1597, 1495, 1407, 1262, 1079, 961, 820 cm^{-1}; MS (EI) *m/z* (%): 214 [M^+] (30.1), 186 (11.8), 110 (100), 77 (10.9), 57 (15.6), 54 (10.9), 52 (11.5), 43 (13.3); HRMS (EI) Calcd. for $C_{14}H_{14}O_2$ (M^+) requires 214.0994, Found: 214.1010.

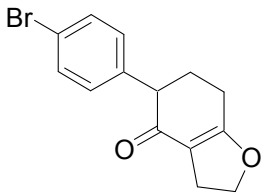




7-(4-Bromophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5c. A yellow solid. m.p. 100-102 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.04-2.09 (m, 1H), 2.33-2.45 (m, 3H), 2.89-2.98 (m, 2H), 3.78-3.80 (m, 1H), 4.57 (t, *J* = 9.6 Hz, 2H), 7.09 (d, *J* = 8.4 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.0, 31.3, 35.0, 40.7, 73.3, 114.8, 121.3, 129.4, 131.9, 137.9, 177.1, 195.1; IR (CH_2Cl_2): ν 2950, 2873, 1743, 1605, 1489, 1405, 1234, 1074, 962, 827 cm^{-1}; MS (EI) *m/z* (%): 292 [M^+] (42.7), 185 (100), 183 (71.1), 181 (96.6) 129 (40.4), 116 (45.2), 110 (62.8), 45 (49.4); HRMS (EI) Calcd. for $C_{14}H_{13}BrO_2$ (M^+) requires 292.0099, Found: 292.0089.

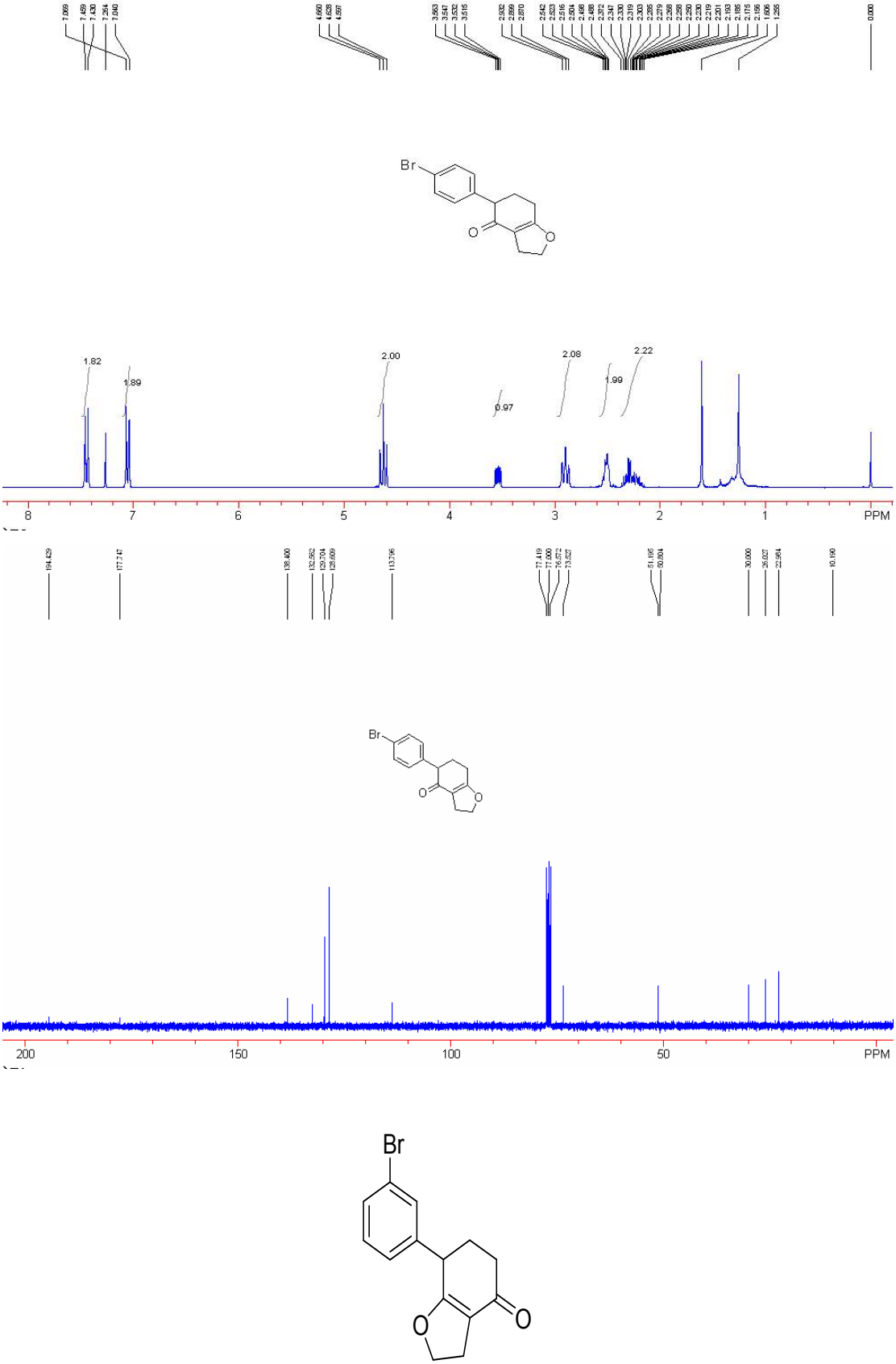






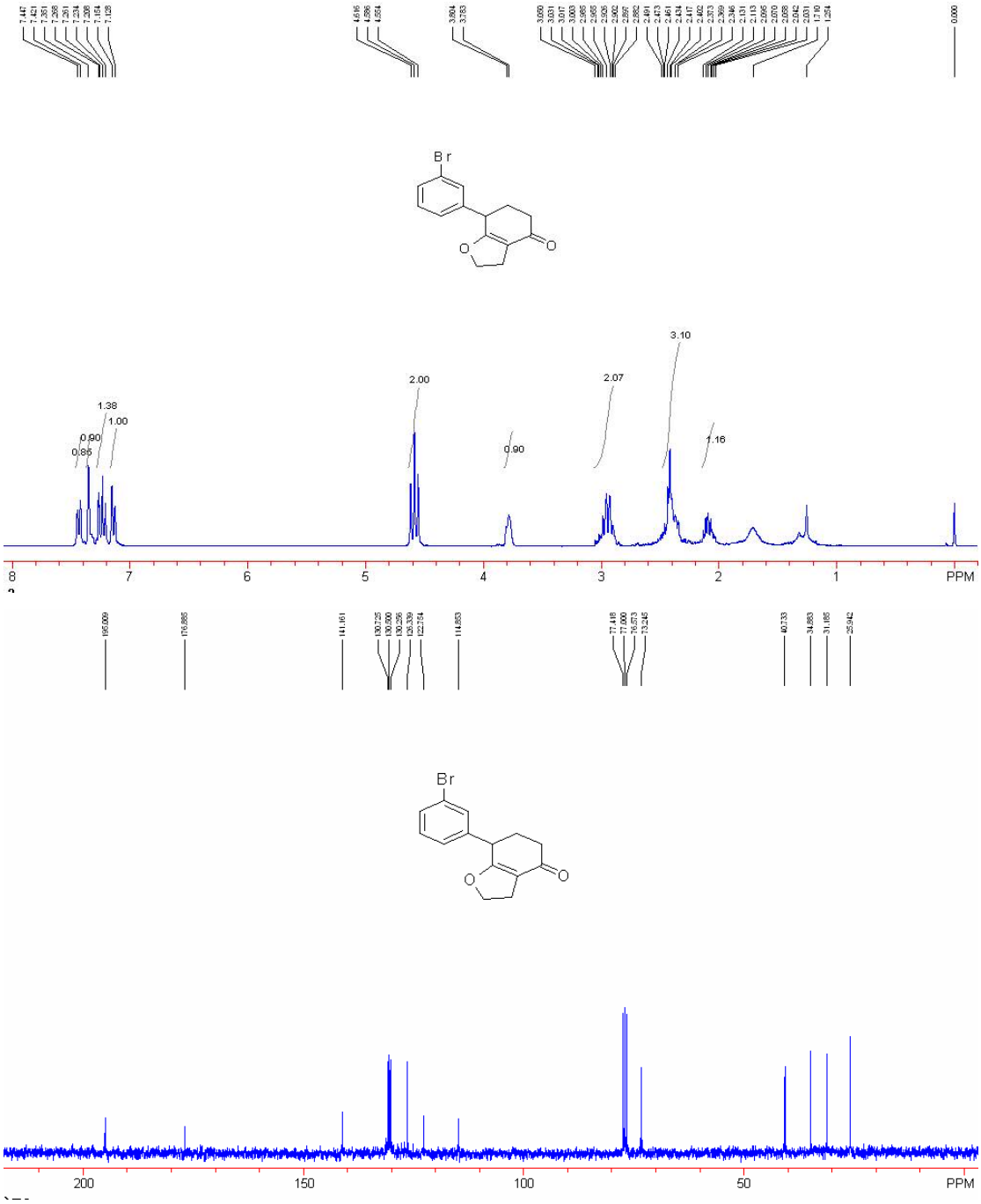
6c

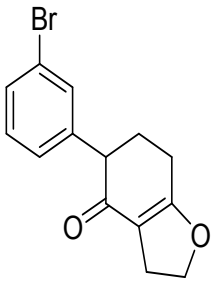
7-(4-Bromophenyl)-2,3,4,5-tetrahydrobenzofuran-6(7*H*)-one 6c. A yellow solid. m.p. 116-118 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.23-2.32 (m, 2H), 2.50-2.52 (m, 2H), 2.90 (t, J = 9.0 Hz, 2H), 3.54 (dd, J = 9.3 Hz, J = 6.1 Hz, 1H), 4.63 (t, J = 9.0 Hz, 2H), 7.05 (d, J = 8.7 Hz, 2H), 7.44 (d, J = 8.7 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.0, 26.0, 30.0, 51.2, 73.5, 113.8, 128.6, 129.7, 132.6, 138.4, 177.7, 194.4; IR (CH_2Cl_2): ν 2947, 2871, 1739, 1620, 1489, 1407, 1230, 1010, 948, 827 cm^{-1}; MS (EI) *m/z* (%): 292 [M^+] (9.9), 294 (9.9), 111 (8.2), 110 (100), 86 (24.6), 84 (36.6), 49 (7.3), 47 (8.4); HRMS (EI) Calcd. for $C_{14}H_{13}BrO_2$ (M^+) requires 292.0099, Found: 292.0102.



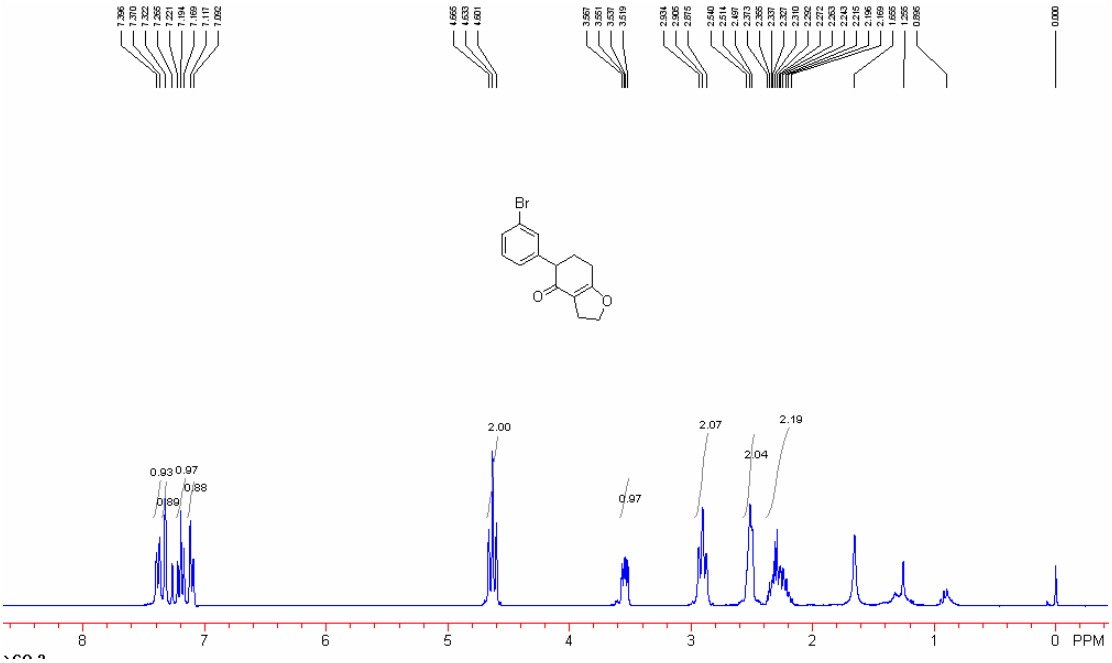
7-(3-Bromophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5d. A yellow solid. m.p. 94-96 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.04-2.13 (m, 1H), 2.35-2.46 (m, 3H), 2.89-2.98 (m, 2H), 3.78-3.80 (m, 1H), 4.59 (t, *J* = 9.6 Hz, 2H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.22 (d, *J* = 7.6 Hz, 1H), 7.35 (s, 1H), 7.44 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS):

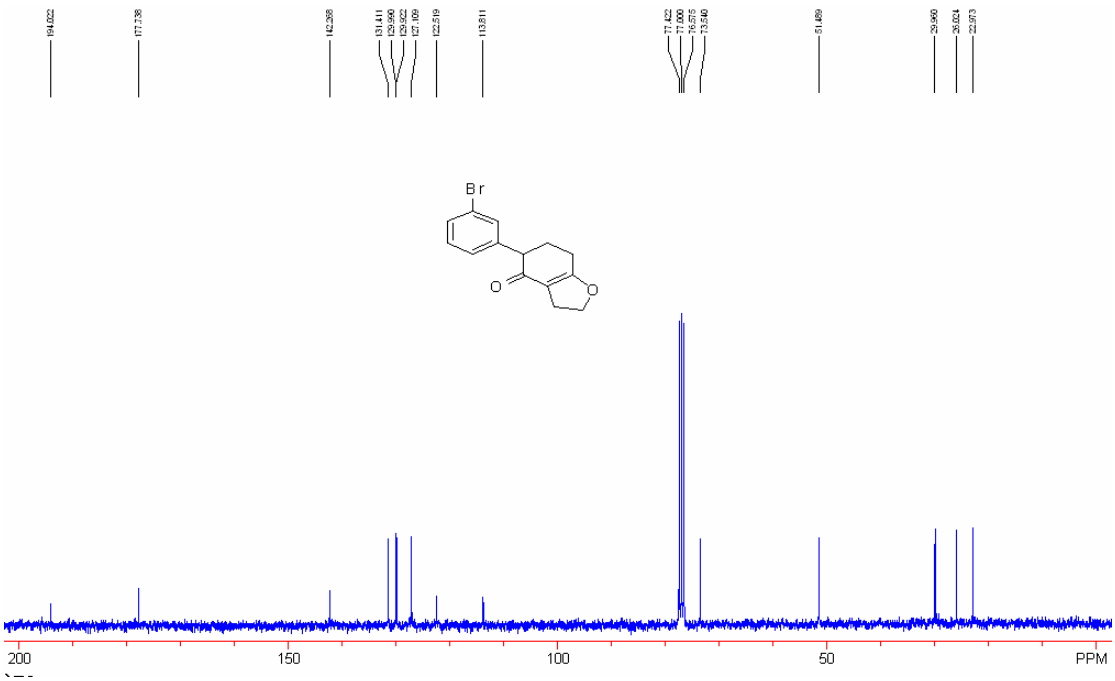
δ 26.0, 31.2, 34.9, 40.7, 73.2, 114.9, 122.8, 126.3, 130.3, 130.5, 130.7, 141.2, 176.9, 195.0; IR (CH_2Cl_2): ν 2927, 2869, 1733, 1593, 1567, 1475, 1402, 1233, 1073, 961 cm^{-1}; MS (EI) *m/z* (%): 292 (60.5), 294 (59.9), 266 (44.7), 264 (45.1), 185 (100), 157 (40.4), 129 (75.0), 128 (39.0); HRMS (EI) Calcd. for $C_{14}H_{13}BrO_2$ (M^+) requires 292.0099, Found: 292.0098.

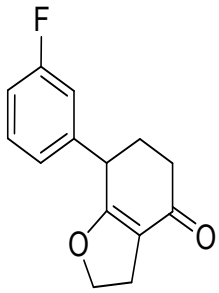




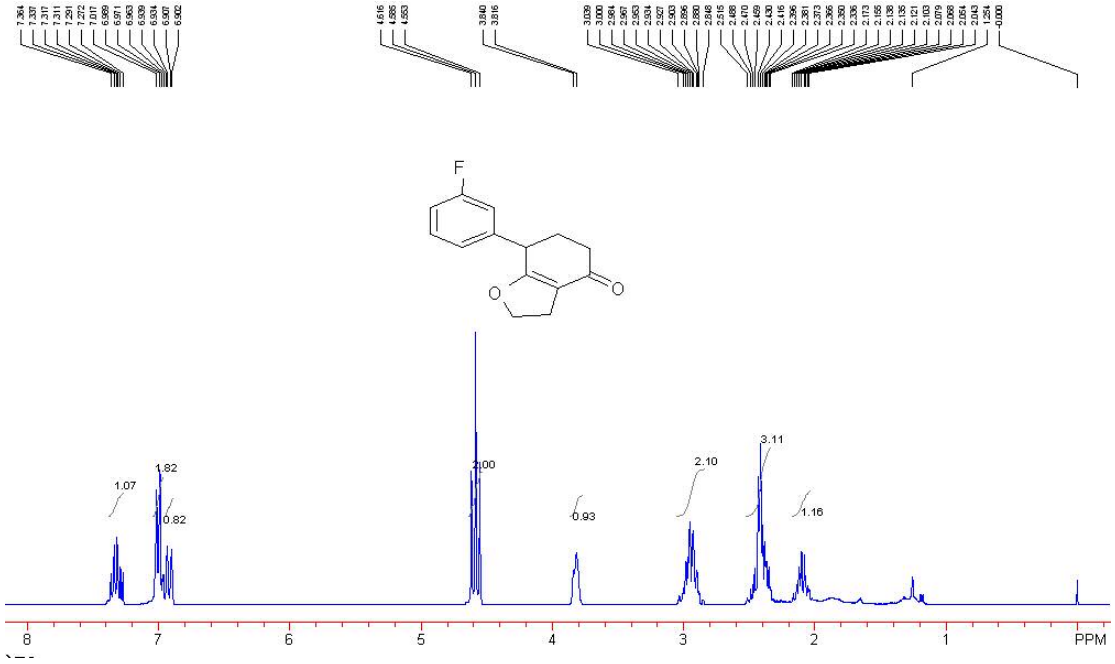
5-(3-Bromophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6d. A yellow solid. m.p. 110-112 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.17-2.37 (m, 2H), 2.50-2.54 (m, 2H), 2.91 (t, *J* = 9.0 Hz, 2H), 3.54 (dd, *J* = 9.3 Hz, *J* = 5.1 Hz, 1H), 4.63 (t, *J* = 9.0 Hz, 2H), 7.11 (d, *J* = 7.5 Hz, 2H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.32 (s, 1H), 7.38 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.0, 26.0, 30.0, 51.5, 73.5, 113.8, 122.5, 127.1, 129.92, 130.0, 131.4, 142.3, 177.7, 194.0; IR (CH_2Cl_2): ν 2928, 2869, 1733, 1635, 1476, 1407, 1229, 1074, 954, 790 cm^{-1}; MS (EI) *m/z* (%): 292 (10.6), 294 (9.9), 111 (7.5), 110 (100), 80 (4.6), 68 (5.0), 54 (6.6), 52 (6.1); HRMS (EI) Calcd. for $C_{14}H_{13}BrO_2$ (M^+) requires 292.0099, Found: 292.0100.

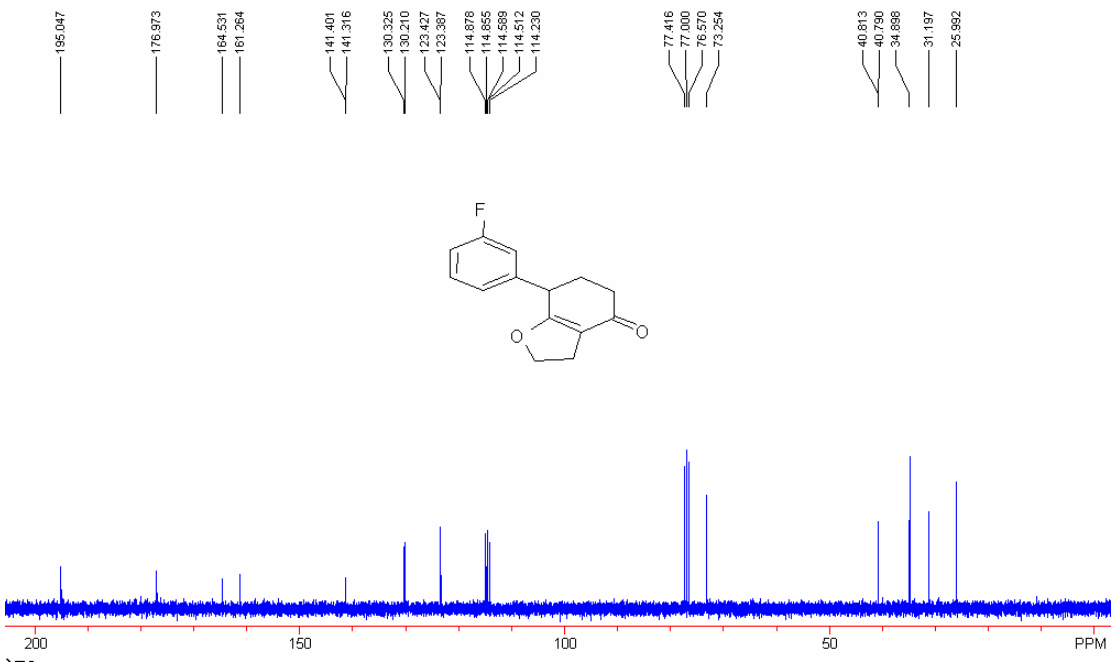


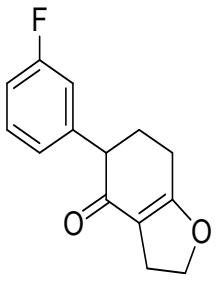




7-(3-Fluorophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5e. A yellow solid. m.p. 91-93 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.04-2.16 (m, 1H), 2.35-2.46 (m, 3H), 2.90-2.98 (m, 2H), 3.82-3.84 (m, 1H), 4.59 (t, J = 9.6 Hz, 2H), 6.90-7.02 (m, 3H), 7.27-7.36 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.0, 31.2, 34.9, 40.8 (d, $J_{C\text{-}F}$ = 1.7 Hz), 73.3, 114.4 (d, $J_{C\text{-}F}$ = 21.1 Hz), 114.7 (d, $J_{C\text{-}F}$ = 21.7 Hz), 114.9, 123.4 (d, $J_{C\text{-}F}$ = 3.0 Hz), 130.3 (d, $J_{C\text{-}F}$ = 8.6 Hz), 141.4 (d, $J_{C\text{-}F}$ = 7.4 Hz), 162.9 (d, $J_{C\text{-}F}$ = 245.0 Hz), 177.8, 195.1; IR (CH_2Cl_2): ν 2929, 2871, 1732, 1612, 1590, 1489, 1402, 1235, 1009, 962, 789 cm^{-1}; MS (EI) *m/z* (%): 232 [M^+] (69.6), 233 (22.0), 204 (100), 203 (23.2), 176 (17.8), 147 (23.6), 133 (20.0), 108 (19.3); HRMS (EI) Calcd. for $C_{14}H_{13}FO_2$ (M^+) requires 232.0900, Found: 232.0905.

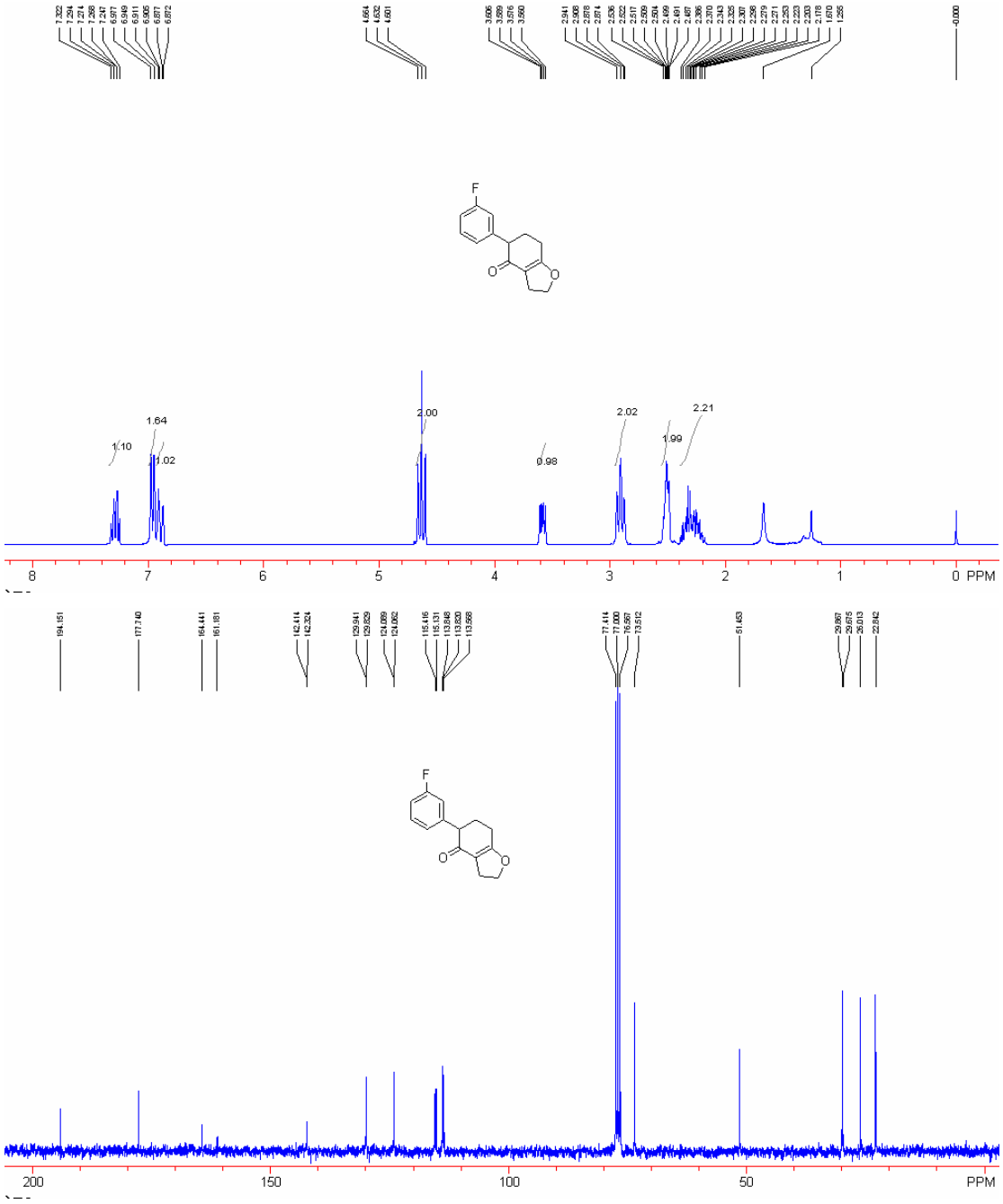


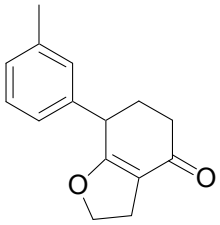




7-(3-Fluorophenyl)-2,3,4,5-tetrahydrobenzofuran-6(7*H*)-one 6e. A yellow solid. m.p. 86-88 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.22-2.37 (m, 2H), 2.49-2.54 (m, 2H), 2.91 (t, J = 9.6 Hz, 2H), 3.58 (dd, J = 9.3 Hz, J = 4.8 Hz, 1H), 4.63 (t, J = 9.6 Hz, 2H), 6.87-6.98 (m, 3H), 7.25-7.32 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 22.8, 26.0, 29.9, 51.5 (d, $J_{C\text{-}F}$ =

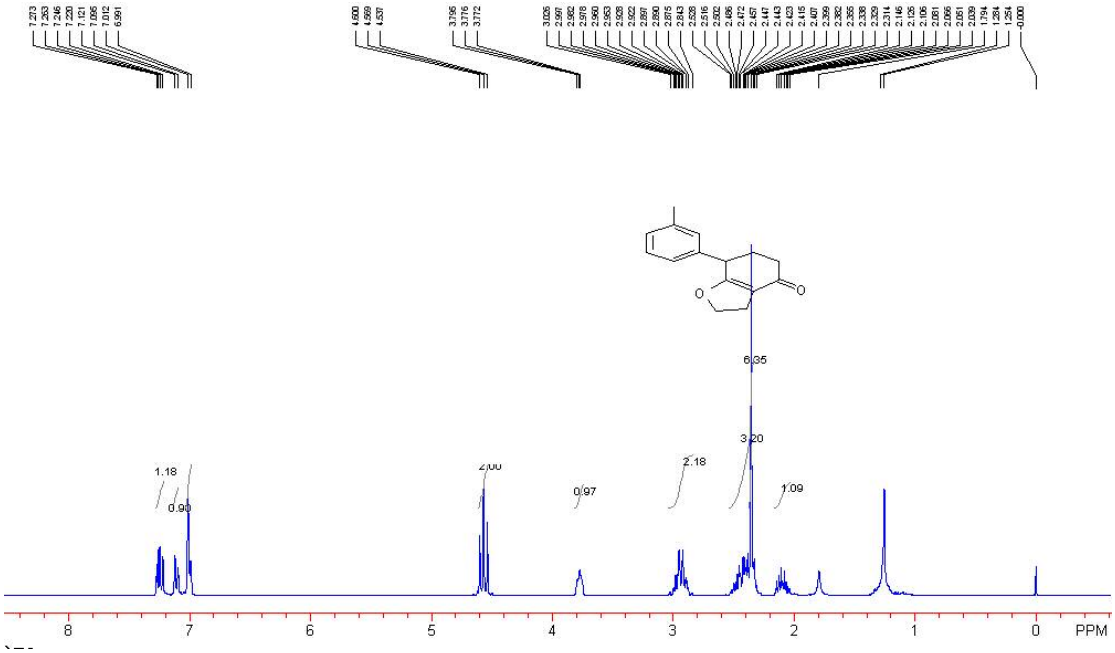
1.1 Hz), 73.5, 113.7 (d, $J_{C\text{-}F}$ = 21.1 Hz), 113.8, 115.3 (d, $J_{C\text{-}F}$ = 21.8 Hz), 124.1 (d, $J_{C\text{-}F}$ = 2.9 Hz), 129.9 (d, $J_{C\text{-}F}$ = 8.6 Hz), 142.4 (d, $J_{C\text{-}F}$ = 6.9 Hz), 162.8 (d, $J_{C\text{-}F}$ = 243.9 Hz), 177.8, 194.2; IR (CH_2Cl_2): ν 2931, 2871, 1733, 1628, 1589, 1407, 1240, 1030, 961, 782 cm^{-1}; MS (EI) *m/z* (%): 232 [M^+] (23.7), 233 (7.3), 111 (7.8), 110 (100), 80 (5.3), 68 (6.1), 54 (7.4), 52 (6.9); HRMS (EI) Calcd. for $C_{14}H_{13}FO_2$ (M^+) requires 232.0900, Found: 232.0903.

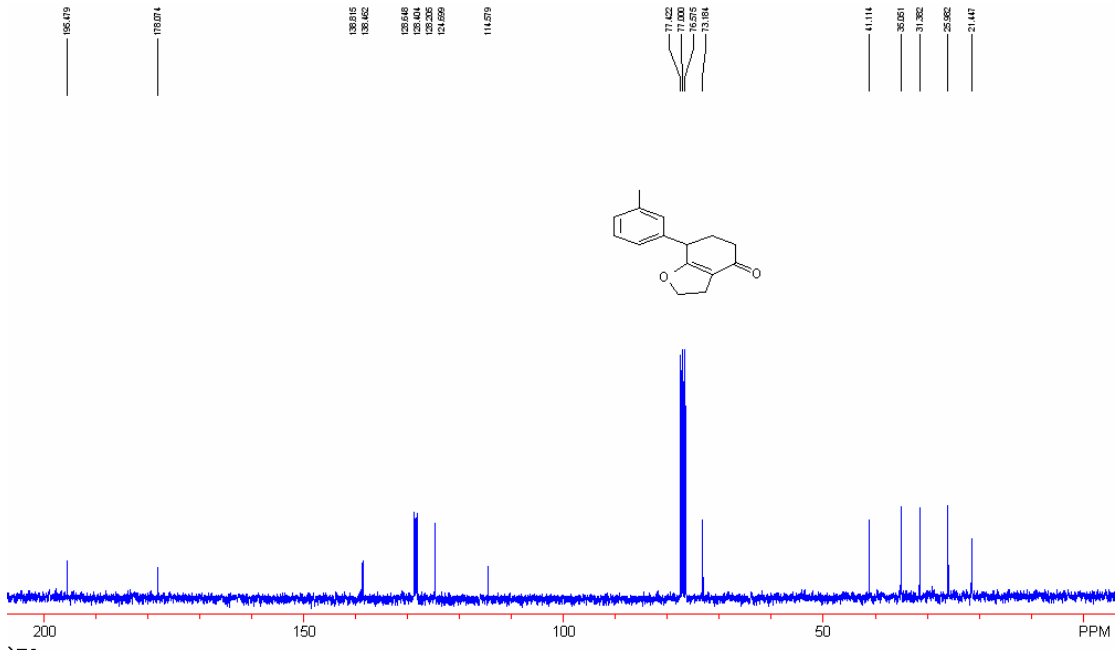


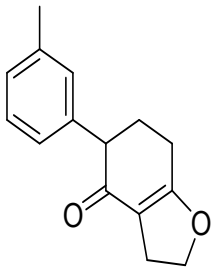


5f

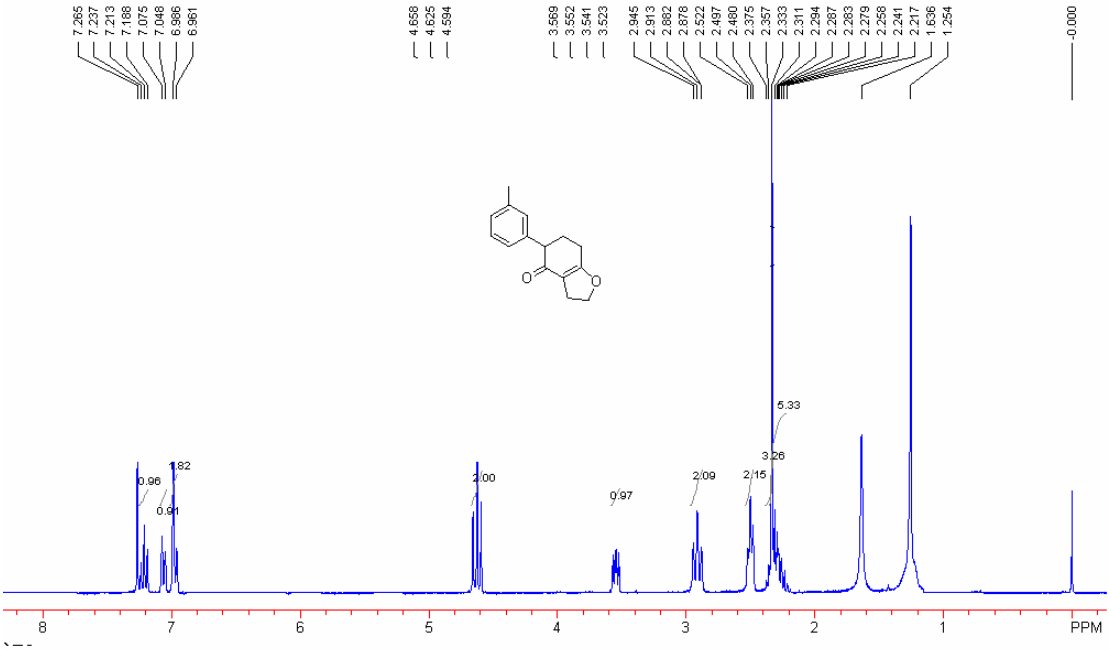
7-m-Tolyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5f. A colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.04-2.15 (m, 1H), 2.31-2.53 (m, 3H), 2.36 (s, 3H), 2.84-3.03 (m, 2H), 3.78-3.80 (m, 1H), 4.57 (t, *J* = 9.6 Hz, 2H), 6.99-7.01 (m, 2H), 7.11 (d, *J* = 7.8 Hz, 1H), 7.26 (t, *J* = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 21.4, 26.0, 31.4, 35.0, 41.1, 73.2, 114.6, 124.7, 128.2, 128.4, 128.7, 138.5, 138.8, 178.1, 195.5; IR (CH_2Cl_2): ν 2925, 2869, 1726, 1624, 1472, 1399, 1233, 1059, 962, 789 cm^{-1}; MS (EI) *m/z* (%): 228 [M^+] (67.8), 229 (18.7), 200 (100), 199 (16.8), 172 (18.8), 129 (23.4), 128 (16.4), 115 (22.0); HRMS (EI) Calcd. for $C_{15}H_{16}O_2$ (M^+) requires 228.1150, Found: 228.1143.

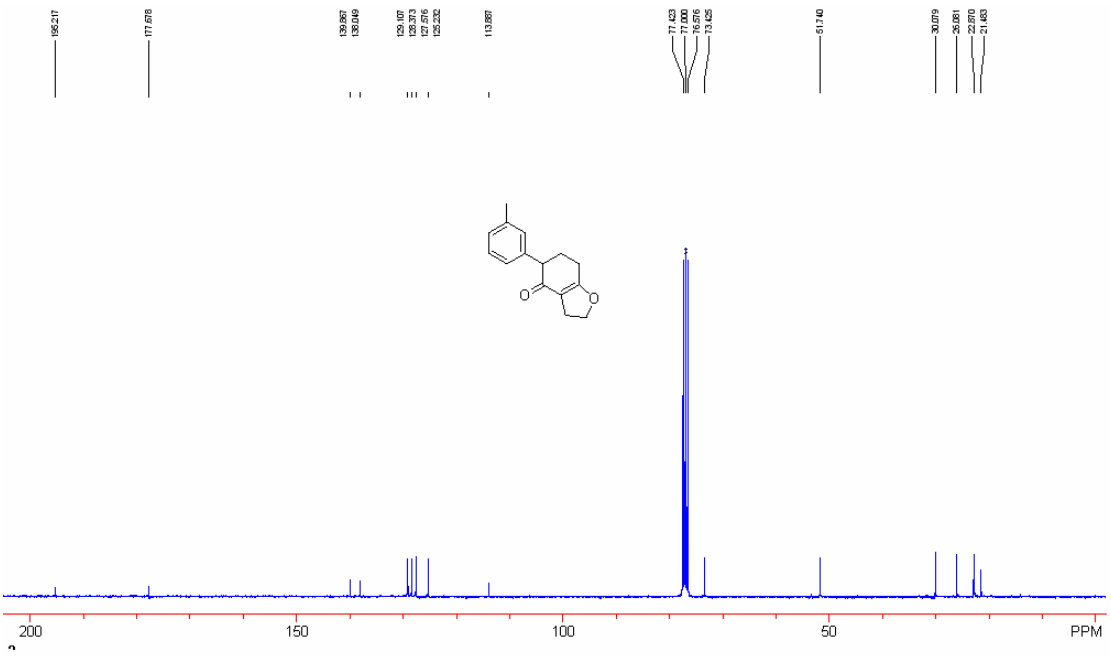


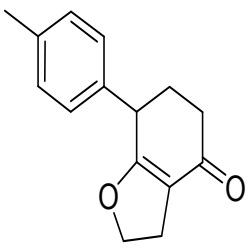




5-m-Tolyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6f. A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.22-2.38 (m, 2H), 2.33 (s, 3H), 2.48-2.52 (m, 2H), 2.88-2.95 (m, 2H), 3.55 (dd, J = 8.4 Hz, J = 5.1 Hz, 1H), 4.63 (t, J = 9.3 Hz, 2H), 6.96-6.99 (m, 2H), 7.06 (d, J = 7.5 Hz, 1H), 7.21 (t, J = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 21.5, 22.9, 26.1, 30.1, 51.7, 73.4, 113.9, 125.2, 127.6, 128.4, 129.1, 138.1, 139.9, 177.7, 195.2; IR (CH_2Cl_2): ν 2922, 2851, 2360, 2343, 1631, 1405, 1220, 1191, 700 cm^{-1}; MS (EI) *m/z* (%): 228 [M^+] (5.2), 169 (33.4), 135 (34.6), 91 (100), 84 (70.0), 69 (72.8), 44 (73.4), 41 (41.2); HRMS (EI) Calcd. for $C_{15}H_{16}O_2$ (M^+) requires 228.1150, Found: 228.1143.

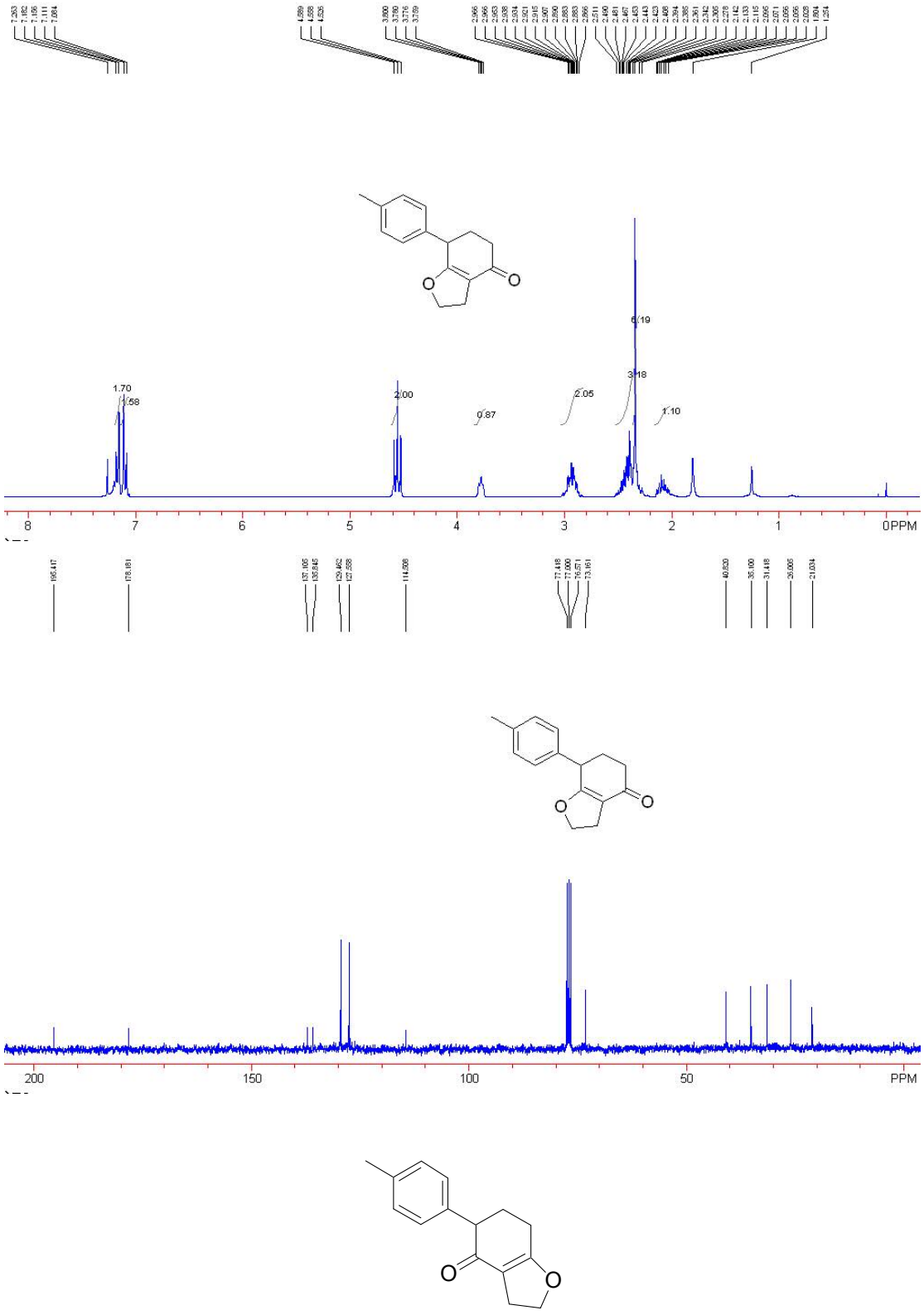






7-*p*-Tolyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5g. A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.01-2.14 (m, 1H), 2.28-2.51 (m, 3H), 2.34 (s, 3H), 2.87-2.97 (m, 2H), 3.76-3.80 (m, 1H), 4.56 (t, J = 9.3 Hz, 2H), 7.10 (d, J = 8.1 Hz, 2H), 7.17 (d, J = 8.1 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 21.0, 26.0, 31.4, 35.1, 40.8, 73.2, 114.5, 127.6, 129.5, 135.9, 137.1, 178.2, 195.4; IR (CH_2Cl_2): ν 2924, 2869, 1735, 1624, 1514, 1450, 1400, 1234,

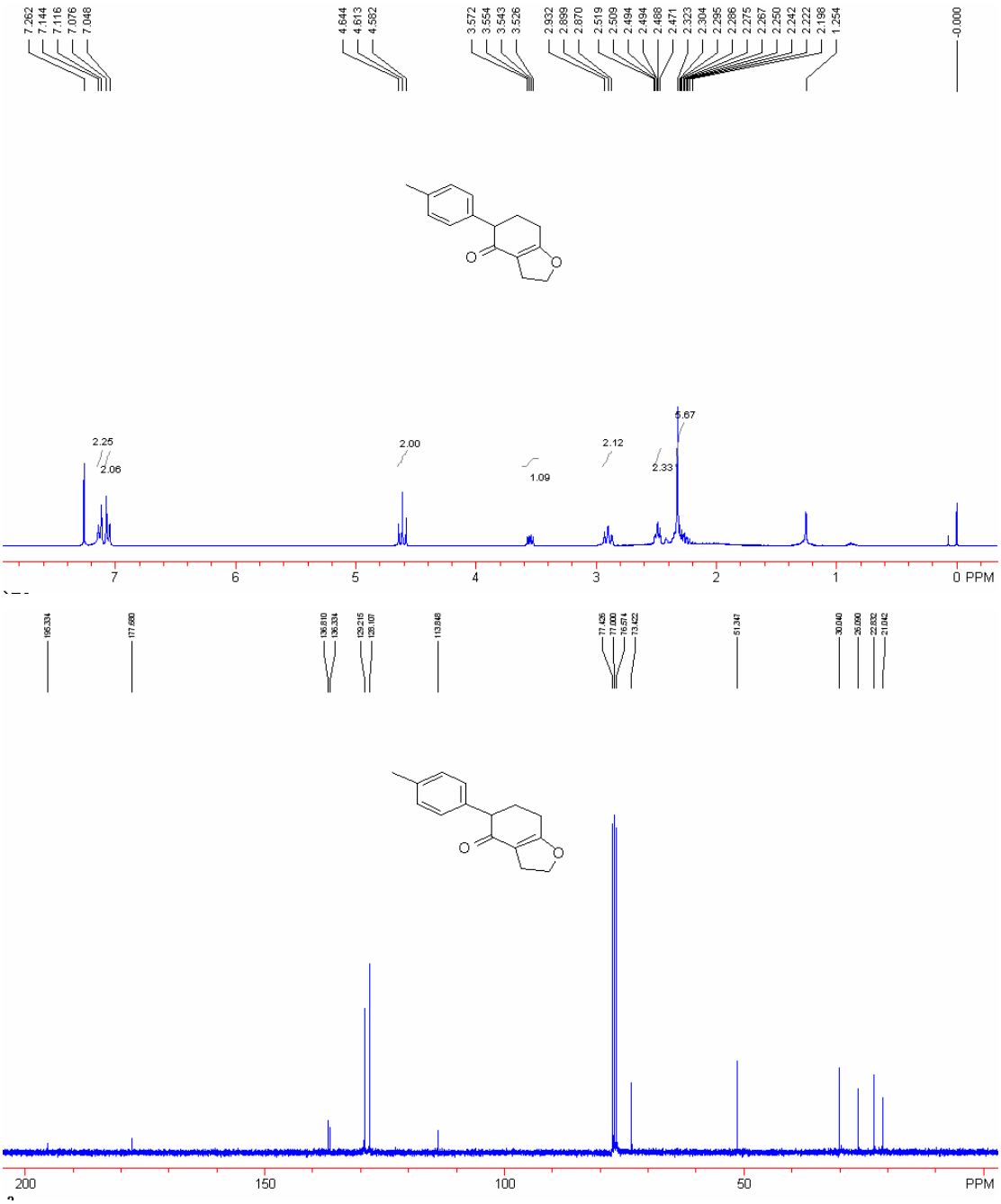
816; MS (EI) *m/z* (%): 228 [M^+] (66.3), 200 (100), 172 (22.7), 129 (25.5), 128 (19.8), 119 (18.1), 115 (27.4), 91 (24.8); HRMS (EI) Calcd. for $C_{15}H_{16}O_2$ (M^+) requires 228.1150, Found: 228.1146.

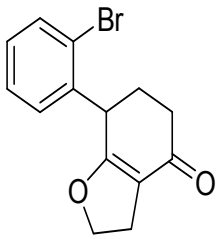


6g

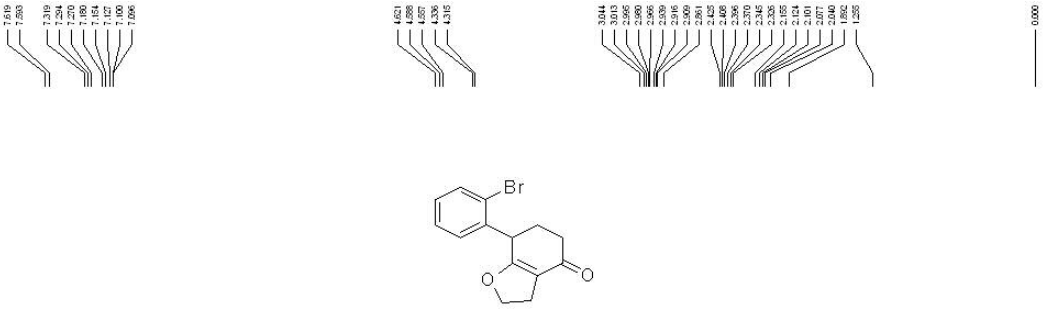
5-p-Tolyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6g. A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.20-2.33 (m, 2H), 2.32 (s, 3H), 2.47-2.52 (m, 2H), 2.91 (t, J = 9.0 Hz, 2H),

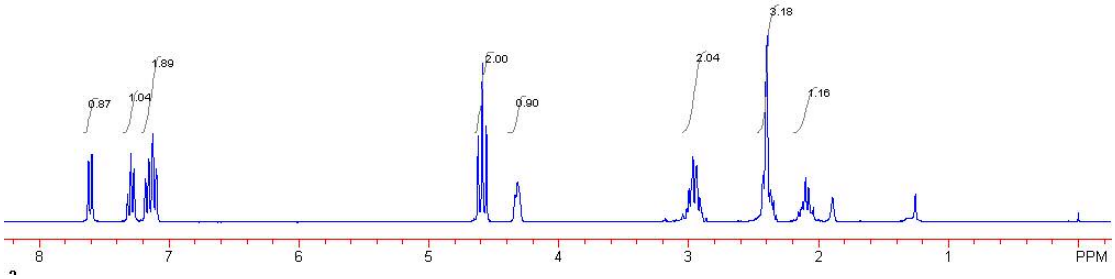
3.55 (dd, J = 8.4 Hz, J = 5.1 Hz, 1H), 4.61 (t, J = 9.0 Hz, 2H), 7.06 (d, J = 8.4 Hz, 2H), 7.13 (d, J = 8.4 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 21.0, 22.8, 26.1, 30.0, 51.3, 73.4, 113.9, 128.1, 129.2, 136.3, 136.8, 177.7, 195.3; IR (CH_2Cl_2): ν 2924, 2869, 1733, 1631, 1514, 1406, 1231, 1063, 996, 817 cm^{-1}; MS (EI) *m/z* (%): 228 [M^+] (24.2), 117 (7.1), 111 (6.9), 110 (100), 91 (8.6), 80 (6.7), 54 (7.2), 52 (6.3); HRMS (EI) Calcd. for $C_{15}H_{16}O_2$ (M^+) requires 228.1150, Found: 228.1149.

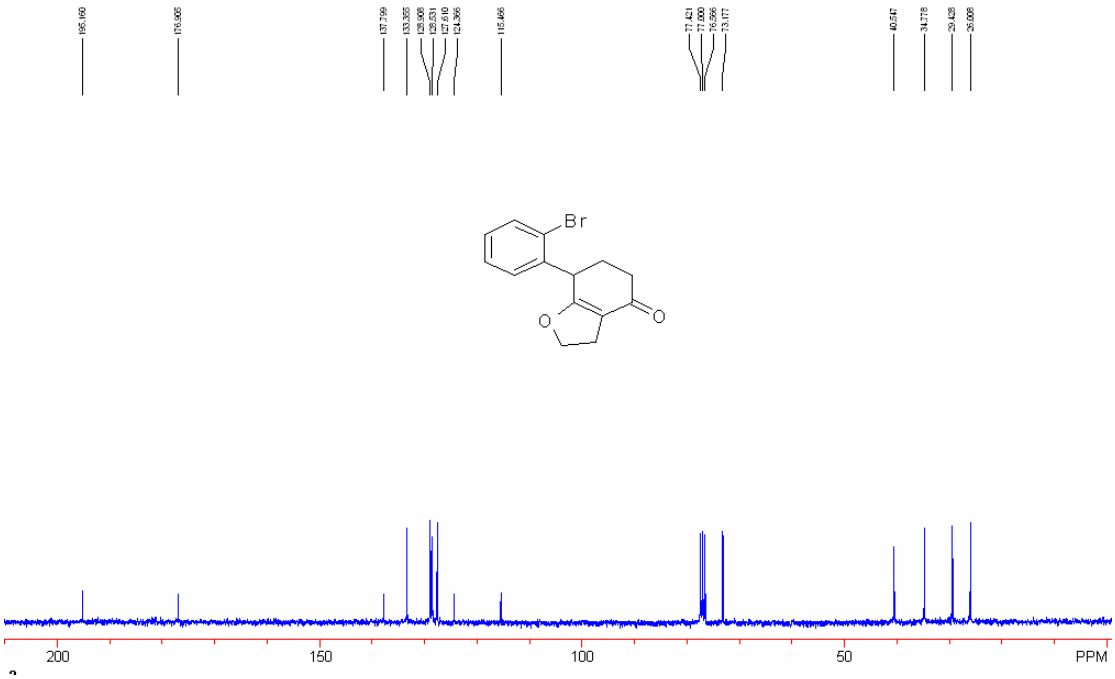


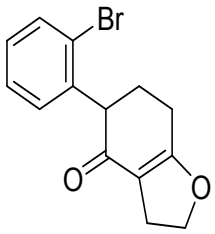


7-(2-Bromophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5h. A yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.04-2.16 (m, 1H), 2.35-2.43 (m, 3H), 2.91-3.05 (m, 2H), 4.32-4.34 (m, 1H), 4.59 (t, *J* = 9.3 Hz, 2H), 7.10-7.18 (m, 2H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.57 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.0, 29.4, 34.8, 40.5, 73.2, 115.5, 124.4, 127.6, 128.5, 128.9, 133.4, 137.8, 176.9, 195.2; IR (CH_2Cl_2): ν 2926, 2869, 1736, 1623, 1566, 1471, 1404, 1231, 1071, 961 cm^{-1}; MS (EI) *m/z* (%): 292 (30.9), 294 (30.8), 185 (100), 157 (27.3), 129 (53.4), 128 (25.4), 115 (16.2), 89 (16.7); HRMS (EI) Calcd. for $C_{14}H_{13}BrO_2$ (M^+) requires 292.0099, Found: 292.0105.

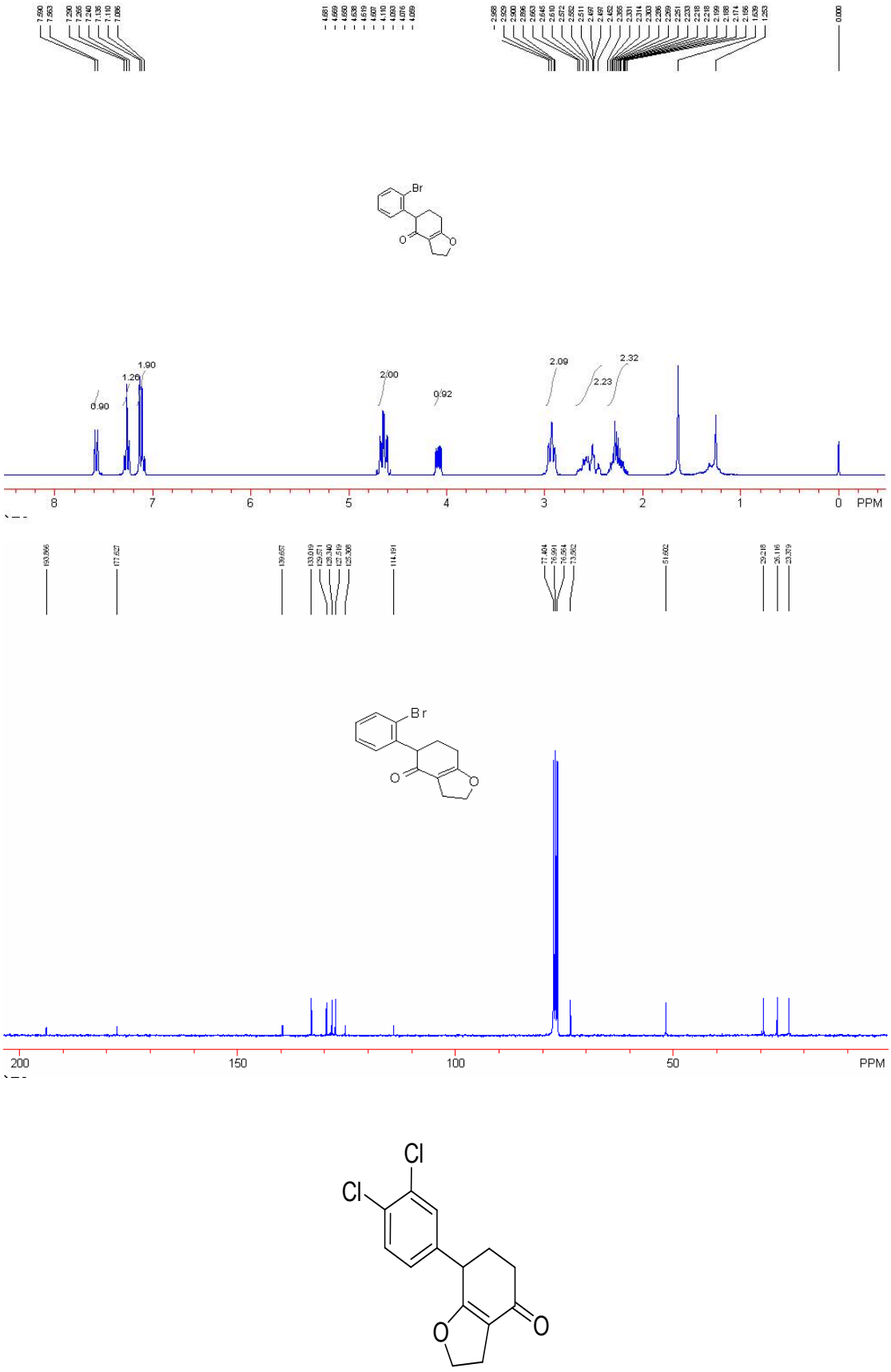






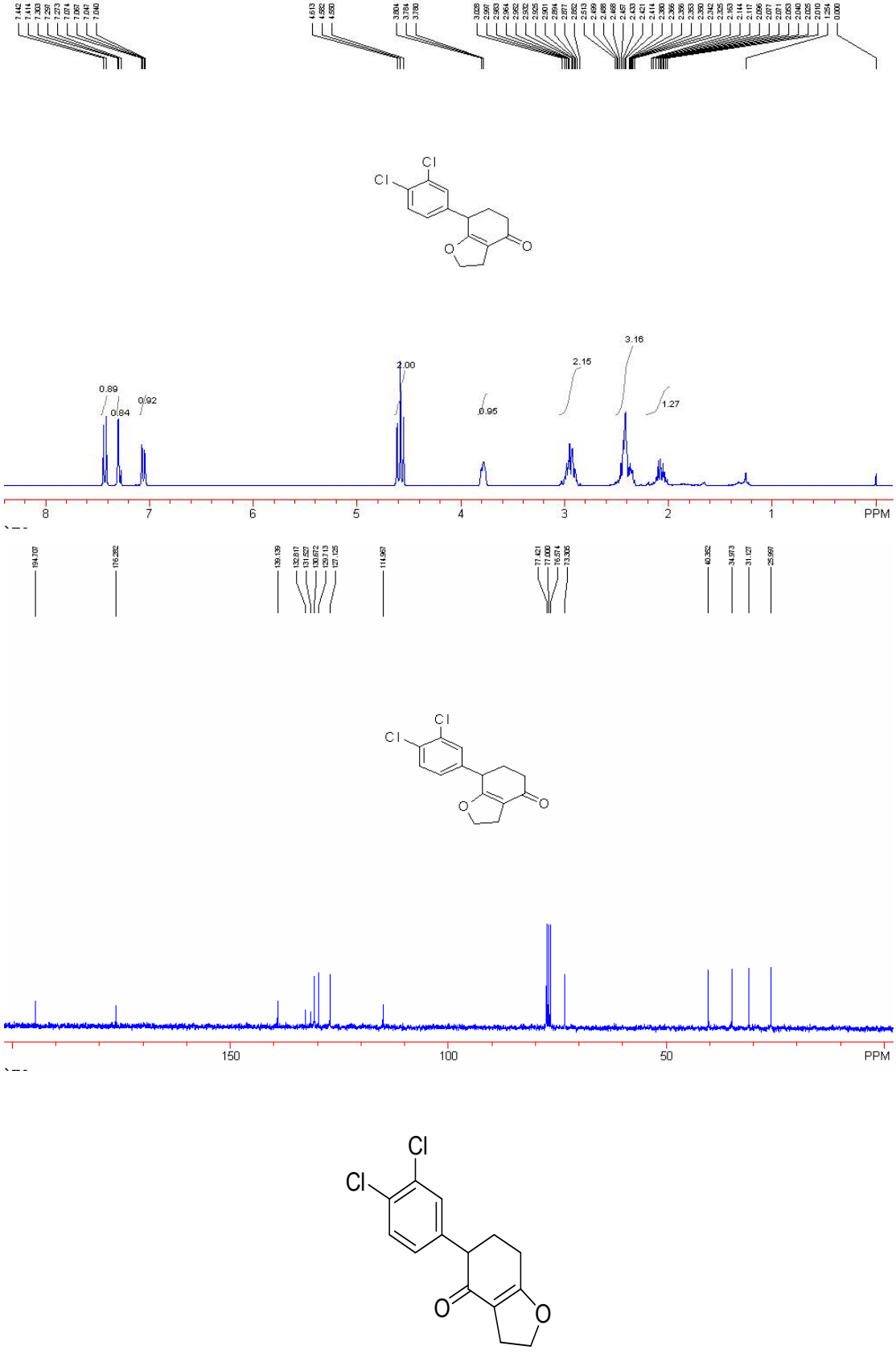


5-(2-Bromophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6h. A colorless liquid. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.16-2.33 (m, 2H), 2.45-2.66 (m, 2H), 2.93 (t, *J* = 9.3 Hz, 2H), 4.09 (dd, *J* = 7.2 Hz, *J* = 5.4 Hz, 1H), 4.64 (td, *J* = 9.3 Hz, *J* = 3.6 Hz, 2H), 7.08-7.14 (m, 2H), 7.27 (t, *J* = 7.5 Hz, 1H), 7.57 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.4, 26.1, 29.2, 51.6, 73.6, 114.2, 125.3, 127.5, 128.4, 129.6, 133.0, 139.7, 177.7, 193.9; IR (CH_2Cl_2): ν 2927, 2870, 1733, 1625, 1474, 1407, 1231, 1023, 950, 750 cm^{-1}; MS (EI) *m/z* (%): 214 (10.3), 213 (69.7), 110 (100), 77 (8.9), 68 (8.8), 55 (8.5), 54 (12.1), 52 (11.8); HRMS (EI) Calcd. for $C_{14}H_{13}BrO_2$ (M^+) requires 292.0099, Found: 292.0092.

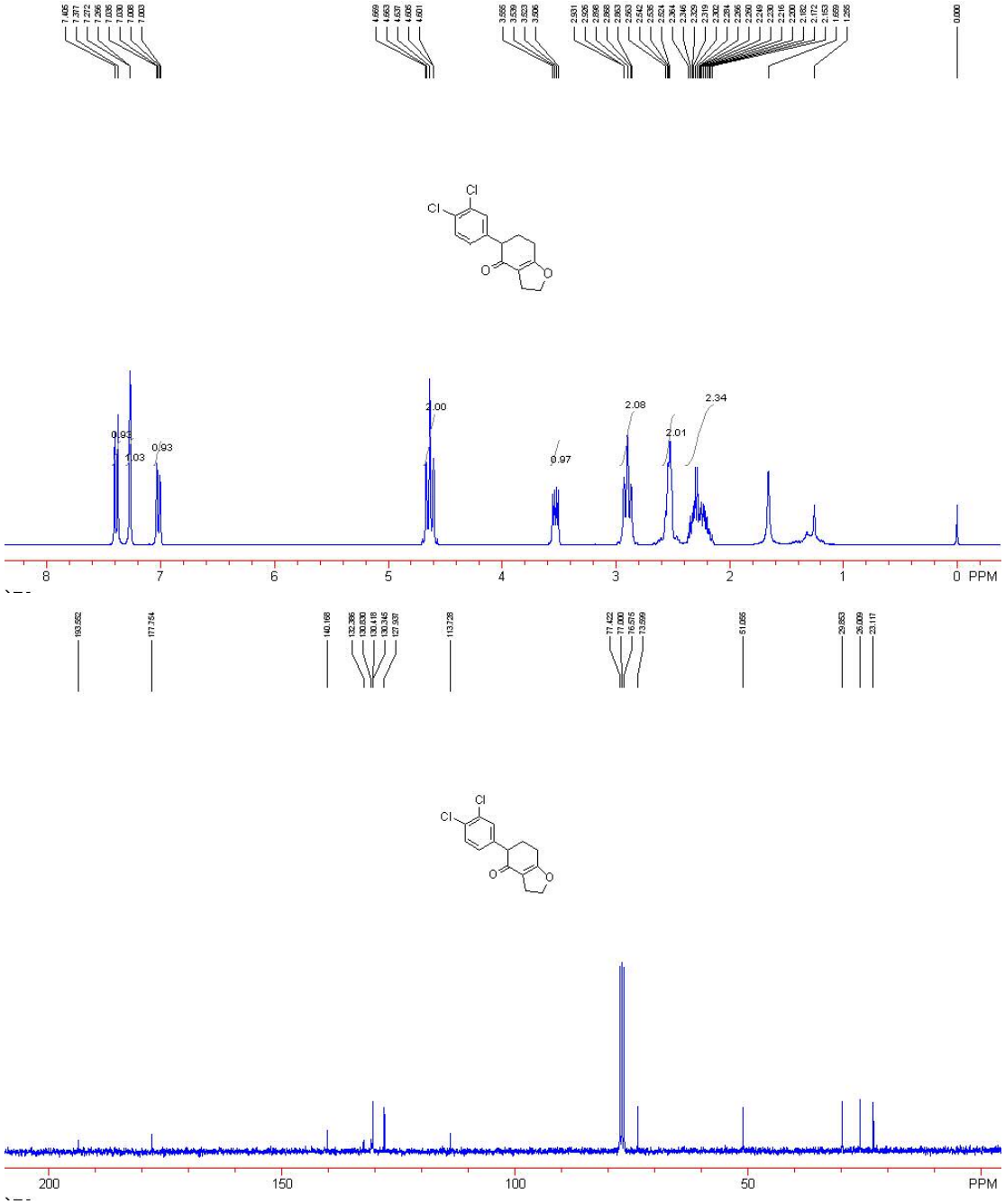


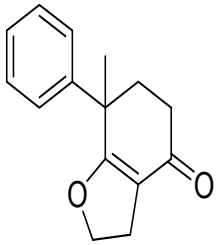
7-(3,4-Dichlorophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5i. A white solid. m.p. 117-119 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.04-2.10 (m, 1H), 2.34-2.46 (m, 3H), 2.89-2.98 (m, 2H), 3.78-3.80 (m, 1H), 4.58 (t, J = 9.6 Hz, 2H), 7.06 (dd, J = 8.1 Hz, J = 2.1 Hz, 1H), 7.30 (d, J = 2.1 Hz, 1H), 7.43 (d, J = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS):

δ 26.0, 31.1, 35.0, 40.3, 73.3, 115.0, 127.1, 129.8, 130.7, 131.5, 132.8, 139.1, 176.3, 194.7; IR (CH_2Cl_2): ν 2928, 2871, 1732, 1652, 1472, 1397, 1231, 1030, 961, 821 cm^{-1}; MS (EI) *m/z* (%): 282 [M^+] (66.8), 284 (40.3), 256 (62.8), 254 (100), 219 (46.5), 191 (21.3), 163 (29.3), 128 (25.6); HRMS (EI) Calcd. for $C_{14}H_{12}Cl_2O_2$ (M^+) requires 282.0214, Found: 282.0211.

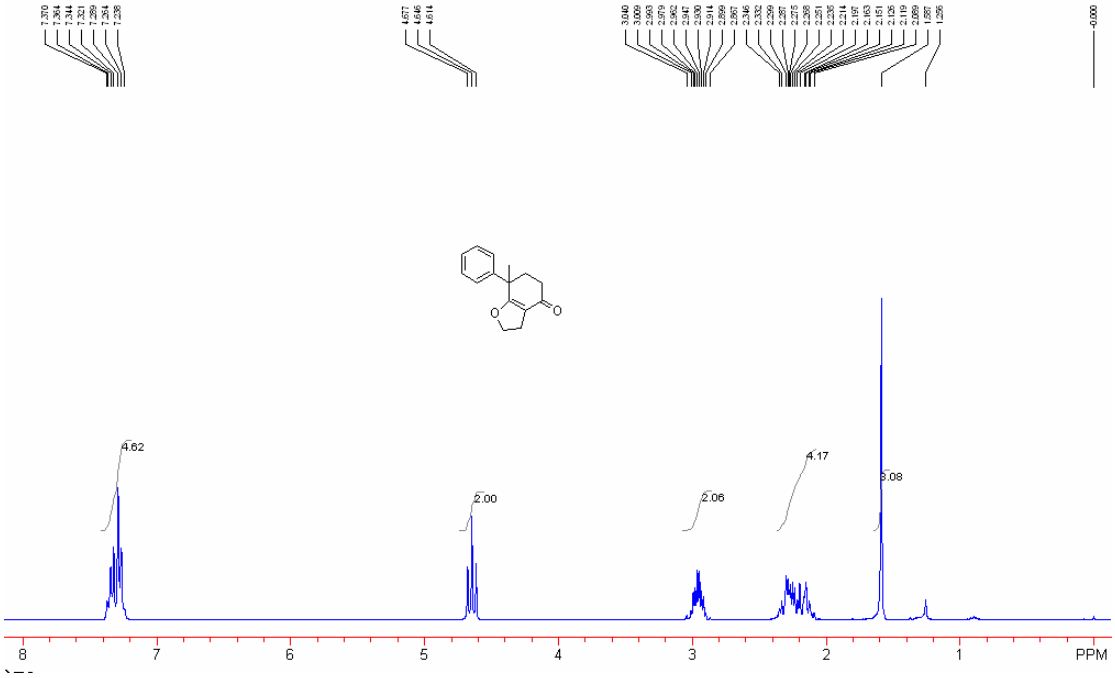


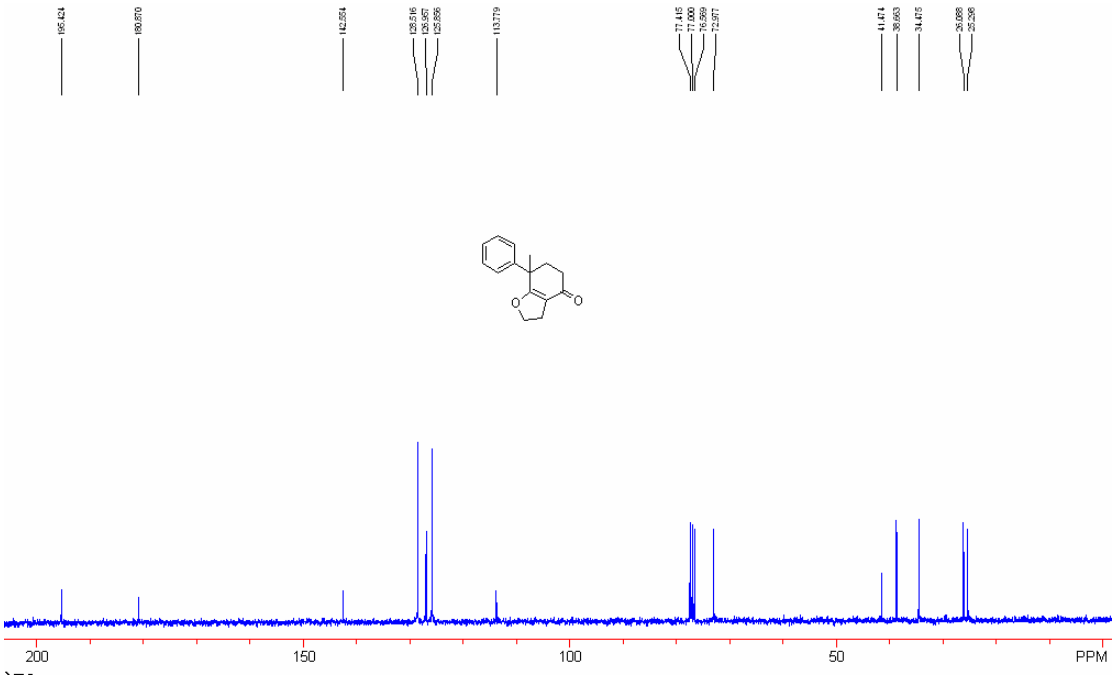
5-(3,4-Dichlorophenyl)-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 6i. A yellow liquid. m.p. 114-116 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.29-2.35 (m, 2H), 2.52-2.56 (m, 2H), 2.90 (t, *J* = 9.0 Hz, 2H), 3.53 (dd, *J* = 9.6 Hz, *J* = 5.1 Hz, 1H), 4.64 (t, *J* = 9.0 Hz, 2H), 7.02 (dd, *J* = 8.1 Hz, *J* = 1.5 Hz, 1H), 7.27 (d, *J* = 1.5 Hz, 1H), 7.39 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 23.1, 26.0, 29.9, 51.1, 73.6, 113.7, 127.9, 130.3, 130.4, 130.8, 132.4, 140.2, 177.8, 193.6; IR (CH_2Cl_2): ν 2930, 2870, 1734, 1636, 1475, 1405, 1230, 1030, 957, 822 cm^{-1}; MS (EI) *m/z* (%): 282 [M^+] (10.1), 284 (6.7), 111 (7.8), 110 (100), 80 (5.4), 68 (6.3), 54 (8.1), 52 (7.3); HRMS (EI) Calcd. for $C_{14}H_{12}Cl_2O_2$ (M^+) requires 282.0214, Found: 282.0212.

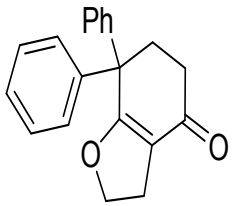




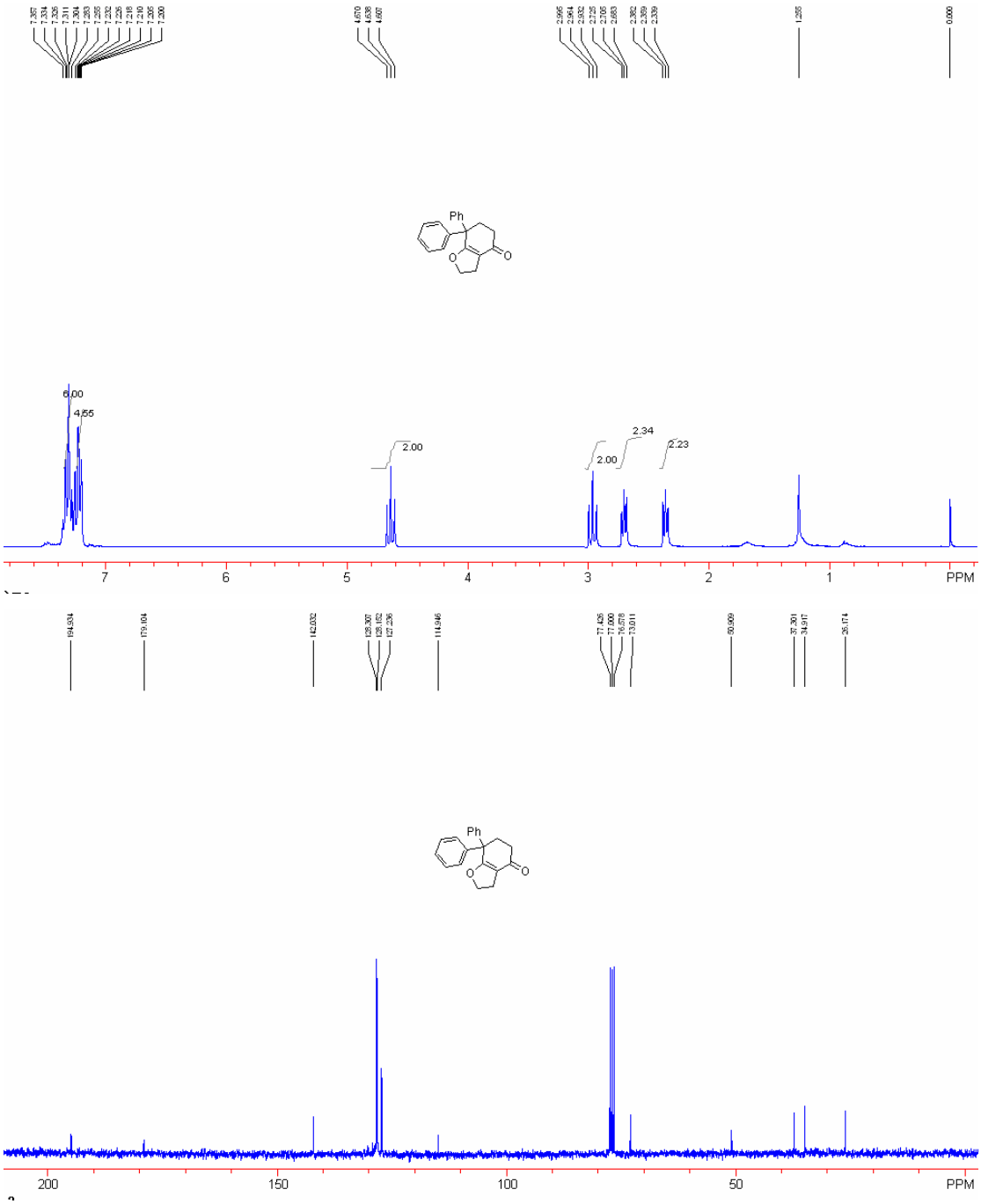
7-Methyl-7-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5j. A yellow solid. m.p. 68-70 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 1.59 (s, 3H), 2.09-2.33 (m, 4H), 2.91-2.99 (td, J = 9.6 Hz, J = 4.8 Hz, 2H), 4.65 (t, J = 9.6 Hz, 2H), 7.29-7.37 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 25.3, 26.1 34.5, 38.7, 41.5, 73.0, 113.8, 125.9, 127.0, 128.5, 142.6, 180.9, 195.4; IR (CH_2Cl_2): ν 3057, 2970, 2872, 1735, 1655, 1582, 1495, 1400, 1229, 1076, 955 cm^{-1}; MS (EI) *m/z* (%): 228 [M^+] (0.77), 155 (10.8), 107 (11.1), 106 (100), 92 (9.1), 91 (60.3), 67 (62.1), 65 (21.3), 56 (16.4); HRMS (EI) Calcd. for $C_{15}H_{16}O_2$ (M^+) requires: 228.1150, found 228.1140.







7,7-Diphenyl-2,3,6,7-tetrahydrobenzofuran-4(5*H*)-one 5k. A white solid. m.p. 128-130 ^{o}C. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ 2.36 (t, *J* = 6.6 Hz, 2H), 2.71 (t, *J* = 6.6 Hz, 2H), 2.97 (t, *J* = 9.6 Hz, 2H), 4.64 (t, *J* = 9.6 Hz, 2H), 7.20-7.26 (m, 4H), 7.29-7.36 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 26.2, 34.9, 37.3, 50.9, 73.0, 115.0, 127.2, 128.2, 128.3, 142.0, 179.1, 195.0; IR (CH_2Cl_2): ν 3057, 2926, 2871, 1724, 1652, 1617, 1494, 1396, 1223, 1064, 965 cm^{-1}; MS (EI) *m/z* (%): 290 [M^+] (93.9), 262 (100), 261 (48.3), 247 (32.9), 185 (42.3), 165 (37.2), 115 (32.6), 105 (30.5); HRMS (EI) Calcd. for $C_{20}H_{18}O_2$ (M^+) requires: 290.1307, found 290.1301.


Ph
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O
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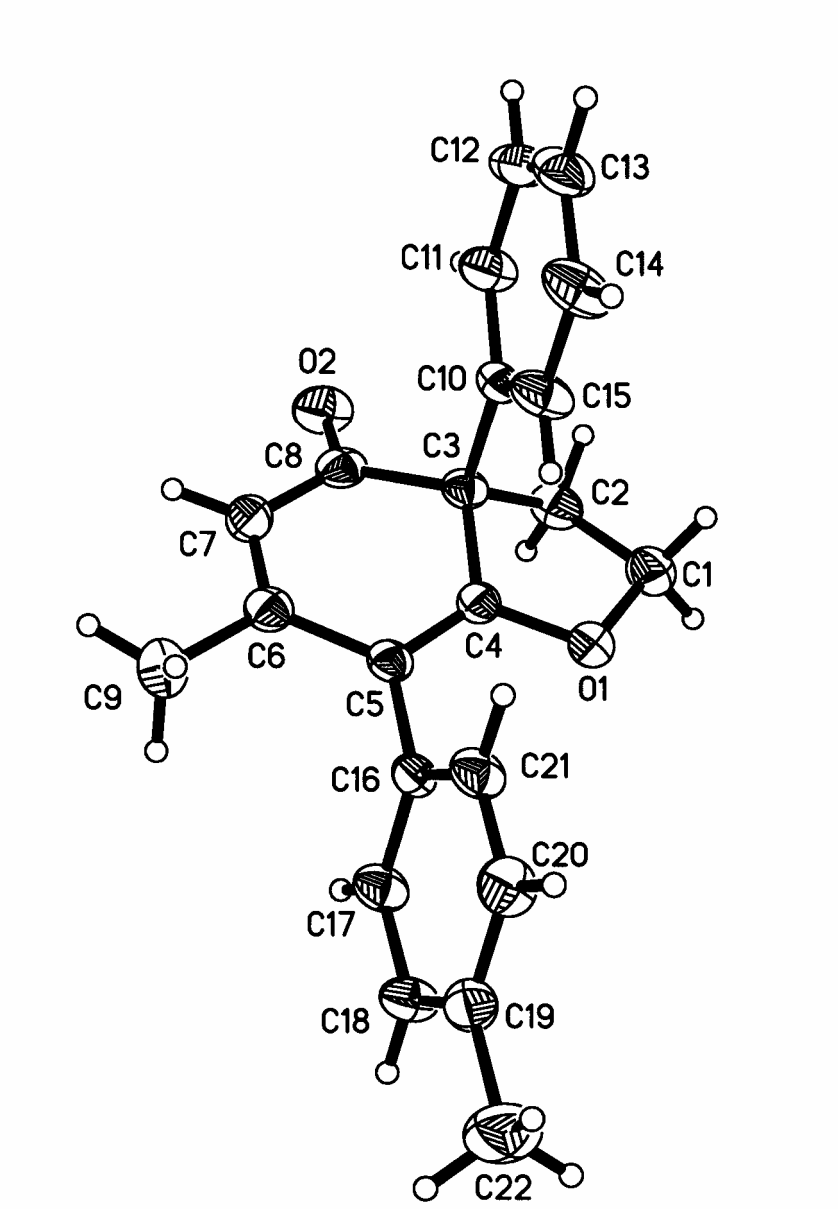
4.55
2.00
2.00
2.34
2.23
7
6
5
4
3
2
1
PPM

Ph
O
O
200
150
100
50
PPM



The crystal data of **2c** have been deposited in CCDC with number 678290. Empirical Formula: $C_{22}H_{20}O_2$; Formula Weight: 316.38; Crystal size: 0.456 x 0.340 x 0.180; Crystal Color, Habit: colorless, prismatic; Crystal System: Triclinic; Lattice Type: Primitive; Lattice Parameters: a = 7.7748(12)Å, b = 12.836(2)Å, c = 17.963(3)Å, α = 97.147(3)^o, β = 93.320(3)^o, γ = 100.103(3)^o, V = 1745.3(5)Å^3; Space group: P-1; Z = 4; D_{calc} = 1.204 g/cm^3; F_{000} = 672; R1 = 0.0664, wR2 = 0.1527. Diffractometer: Rigaku AFC7R.

Table 1. Crystal data and structure refinement for cd2855.

Identification code	cd2855
Empirical formula	C22 H20 O2
Formula weight	316.38
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 7.7748(12) A alpha = 97.147(3) deg. b = 12.836(2) A beta = 93.320(3) deg. c = 17.963(3) A gamma = 100.103(3) deg.
Volume	1745.3(5) A^3
Z, Calculated density	4, 1.204 Mg/m^3
Absorption coefficient	0.076 mm^-1
F(000)	672
Crystal size	0.456 x 0.340 x 0.180 mm
Theta range for data collection	1.86 to 25.50 deg.
Limiting indices	-9<=h<=9, -15<=k<=11, -20<=l<=21
Reflections collected / unique	9333 / 6435 [R(int) = 0.1214]
Completeness to theta = 25.50	98.7 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.7783
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6435 / 0 / 438
Goodness-of-fit on F^2	0.827
Final R indices [I>2sigma(I)]	R1 = 0.0664, wR2 = 0.1527
R indices (all data)	R1 = 0.1216, wR2 = 0.1807
Extinction coefficient	0.0040(9)
Largest diff. peak and hole	0.253 and -0.278 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd2855.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	9828(3)	1000(2)	4384(1)	54(1)
O(2)	4069(3)	-537(2)	3701(1)	69(1)
O(3)	627(3)	5314(2)	1009(1)	52(1)
O(4)	5255(3)	3565(2)	688(1)	73(1)
C(1)	9827(4)	27(2)	3873(2)	60(1)
C(2)	7929(4)	-515(2)	3740(2)	56(1)
C(3)	6998(4)	452(2)	3785(2)	42(1)
C(4)	8175(4)	1200(2)	4383(2)	43(1)
C(5)	7669(4)	1937(2)	4867(2)	42(1)
C(6)	5790(4)	1946(2)	4846(2)	48(1)
C(7)	4606(4)	1172(3)	4424(2)	55(1)
C(8)	5100(4)	281(3)	3965(2)	51(1)
C(9)	5194(4)	2824(3)	5345(2)	71(1)
C(10)	7045(4)	928(2)	3030(2)	42(1)
C(11)	6008(4)	378(3)	2407(2)	60(1)
C(12)	6044(4)	781(3)	1724(2)	69(1)
C(13)	7100(5)	1716(3)	1663(2)	69(1)
C(14)	8152(5)	2262(3)	2274(2)	72(1)
C(15)	8118(4)	1875(2)	2960(2)	61(1)
C(16)	8970(3)	2710(2)	5400(2)	41(1)
C(17)	9147(4)	2635(2)	6162(2)	52(1)
C(18)	10418(4)	3343(2)	6634(2)	56(1)
C(19)	11510(4)	4150(3)	6380(2)	57(1)
C(20)	11336(4)	4225(2)	5615(2)	60(1)
C(21)	10091(4)	3518(2)	5138(2)	53(1)
C(22)	12868(5)	4942(3)	6891(2)	95(1)
C(23)	-84(4)	4184(2)	813(2)	59(1)
C(24)	1457(4)	3656(2)	643(2)	57(1)
C(25)	2940(4)	4380(2)	1165(2)	43(1)
C(26)	2400(4)	5431(2)	1135(1)	41(1)
C(27)	3500(4)	6373(2)	1200(2)	44(1)
C(28)	5362(4)	6338(2)	1238(2)	51(1)
C(29)	5955(4)	5409(3)	1118(2)	56(1)
C(30)	4795(4)	4389(3)	950(2)	50(1)
C(31)	6641(4)	7376(3)	1373(2)	85(1)
C(32)	2874(4)	4068(2)	1975(2)	43(1)
C(33)	3389(4)	3132(2)	2115(2)	54(1)
C(34)	3313(4)	2805(3)	2822(2)	64(1)
C(35)	2704(4)	3416(3)	3386(2)	66(1)
C(36)	2182(4)	4348(3)	3266(2)	65(1)
C(37)	2287(4)	4686(2)	2558(2)	54(1)
C(38)	2818(4)	7388(2)	1203(2)	45(1)
C(39)	2170(4)	7850(3)	1833(2)	69(1)
C(40)	1506(5)	8772(3)	1839(2)	80(1)
C(41)	1458(5)	9277(3)	1216(2)	74(1)
C(42)	2117(5)	8831(3)	582(2)	81(1)
C(43)	2803(5)	7907(2)	572(2)	66(1)
C(44)	736(6)	10301(3)	1218(3)	119(2)

Table 3. Bond lengths [A] and angles [deg] for cd2855.

O(1)-C(4)	1.354(3)
O(1)-C(1)	1.456(3)
O(2)-C(8)	1.227(3)
O(3)-C(26)	1.363(3)
O(3)-C(23)	1.449(3)
O(4)-C(30)	1.223(3)
C(1)-C(2)	1.508(4)
C(1)-H(1A)	0.9700
C(1)-H(1B)	0.9700
C(2)-C(3)	1.539(4)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(4)	1.494(4)
C(3)-C(8)	1.513(4)
C(3)-C(10)	1.554(4)
C(4)-C(5)	1.332(4)
C(5)-C(6)	1.462(4)
C(5)-C(16)	1.491(4)
C(6)-C(7)	1.350(4)
C(6)-C(9)	1.506(4)
C(7)-C(8)	1.446(4)
C(7)-H(7)	0.9300
C(9)-H(9A)	0.9600
C(9)-H(9B)	0.9600
C(9)-H(9C)	0.9600
C(10)-C(15)	1.373(4)
C(10)-C(11)	1.380(4)
C(11)-C(12)	1.390(4)
C(11)-H(11)	0.9300
C(12)-C(13)	1.350(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.367(4)
C(13)-H(13)	0.9300
C(14)-C(15)	1.384(4)
C(14)-H(14)	0.9300
C(15)-H(15)	0.9300
C(16)-C(21)	1.382(4)
C(16)-C(17)	1.384(4)
C(17)-C(18)	1.385(4)
C(17)-H(17)	0.9300
C(18)-C(19)	1.363(4)
C(18)-H(18)	0.9300
C(19)-C(20)	1.390(4)
C(19)-C(22)	1.506(4)
C(20)-C(21)	1.378(4)
C(20)-H(20)	0.9300
C(21)-H(21)	0.9300
C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600
C(22)-H(22C)	0.9600
C(23)-C(24)	1.505(4)
C(23)-H(23A)	0.9700
C(23)-H(23B)	0.9700
C(24)-C(25)	1.534(4)
C(24)-H(24A)	0.9700
C(24)-H(24B)	0.9700
C(25)-C(26)	1.488(4)
C(25)-C(30)	1.513(4)
C(25)-C(32)	1.557(4)
C(26)-C(27)	1.342(4)
C(27)-C(28)	1.454(4)
C(27)-C(38)	1.490(4)
C(28)-C(29)	1.349(4)
C(28)-C(31)	1.501(4)
C(29)-C(30)	1.440(4)
C(29)-H(29)	0.9300
C(31)-H(31A)	0.9600
C(31)-H(31B)	0.9600

C(31)-H(31C)	0.9600
C(32)-C(33)	1.378(4)
C(32)-C(37)	1.383(4)
C(33)-C(34)	1.388(4)
C(33)-H(33)	0.9300
C(34)-C(35)	1.362(4)
C(34)-H(34)	0.9300
C(35)-C(36)	1.365(4)
C(35)-H(35)	0.9300
C(36)-C(37)	1.395(4)
C(36)-H(36)	0.9300
C(37)-H(37)	0.9300
C(38)-C(39)	1.375(4)
C(38)-C(43)	1.384(4)
C(39)-C(40)	1.371(4)
C(39)-H(39)	0.9300
C(40)-C(41)	1.362(5)
C(40)-H(40)	0.9300
C(41)-C(42)	1.377(5)
C(41)-C(44)	1.517(5)
C(42)-C(43)	1.381(4)
C(42)-H(42)	0.9300
C(43)-H(43)	0.9300
C(44)-H(44A)	0.9601
C(44)-H(44B)	0.9601
C(44)-H(44C)	0.9601

C(4)-O(1)-C(1)	108.8(2)
C(26)-O(3)-C(23)	108.0(2)
O(1)-C(1)-C(2)	105.1(2)
O(1)-C(1)-H(1A)	110.7
C(2)-C(1)-H(1A)	110.7
O(1)-C(1)-H(1B)	110.7
C(2)-C(1)-H(1B)	110.7
H(1A)-C(1)-H(1B)	108.8
C(1)-C(2)-C(3)	101.4(2)
C(1)-C(2)-H(2A)	111.5
C(3)-C(2)-H(2A)	111.5
C(1)-C(2)-H(2B)	111.5
C(3)-C(2)-H(2B)	111.5
H(2A)-C(2)-H(2B)	109.3
C(4)-C(3)-C(8)	112.5(2)
C(4)-C(3)-C(2)	100.1(2)
C(8)-C(3)-C(2)	117.8(2)
C(4)-C(3)-C(10)	109.9(2)
C(8)-C(3)-C(10)	105.9(2)
C(2)-C(3)-C(10)	110.5(2)
C(5)-C(4)-O(1)	124.2(3)
C(5)-C(4)-C(3)	125.1(3)
O(1)-C(4)-C(3)	110.6(2)
C(4)-C(5)-C(6)	116.6(3)
C(4)-C(5)-C(16)	121.1(3)
C(6)-C(5)-C(16)	122.3(2)
C(7)-C(6)-C(5)	121.8(3)
C(7)-C(6)-C(9)	120.4(3)
C(5)-C(6)-C(9)	117.8(3)
C(6)-C(7)-C(8)	122.7(3)
C(6)-C(7)-H(7)	118.6
C(8)-C(7)-H(7)	118.6
O(2)-C(8)-C(7)	123.9(3)
O(2)-C(8)-C(3)	121.2(3)
C(7)-C(8)-C(3)	114.8(3)
C(6)-C(9)-H(9A)	109.5
C(6)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(6)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(15)-C(10)-C(11)	118.7(3)
C(15)-C(10)-C(3)	121.9(2)
C(11)-C(10)-C(3)	119.4(3)
C(10)-C(11)-C(12)	120.4(3)

C(10)-C(11)-H(11)	119.8
C(12)-C(11)-H(11)	119.8
C(13)-C(12)-C(11)	120.3(3)
C(13)-C(12)-H(12)	119.8
C(11)-C(12)-H(12)	119.8
C(12)-C(13)-C(14)	119.8(3)
C(12)-C(13)-H(13)	120.1
C(14)-C(13)-H(13)	120.1
C(13)-C(14)-C(15)	120.6(3)
C(13)-C(14)-H(14)	119.7
C(15)-C(14)-H(14)	119.7
C(10)-C(15)-C(14)	120.2(3)
C(10)-C(15)-H(15)	119.9
C(14)-C(15)-H(15)	119.9
C(21)-C(16)-C(17)	117.5(3)
C(21)-C(16)-C(5)	120.2(3)
C(17)-C(16)-C(5)	122.2(3)
C(18)-C(17)-C(16)	120.6(3)
C(18)-C(17)-H(17)	119.7
C(16)-C(17)-H(17)	119.7
C(19)-C(18)-C(17)	122.2(3)
C(19)-C(18)-H(18)	118.9
C(17)-C(18)-H(18)	118.9
C(18)-C(19)-C(20)	117.3(3)
C(18)-C(19)-C(22)	122.8(3)
C(20)-C(19)-C(22)	119.9(3)
C(21)-C(20)-C(19)	121.1(3)
C(21)-C(20)-H(20)	119.5
C(19)-C(20)-H(20)	119.4
C(16)-C(21)-C(20)	121.4(3)
C(16)-C(21)-H(21)	119.3
C(20)-C(21)-H(21)	119.3
C(19)-C(22)-H(22A)	109.5
C(19)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(19)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
O(3)-C(23)-C(24)	105.9(2)
O(3)-C(23)-H(23A)	110.6
C(24)-C(23)-H(23A)	110.5
O(3)-C(23)-H(23B)	110.6
C(24)-C(23)-H(23B)	110.6
H(23A)-C(23)-H(23B)	108.7
C(23)-C(24)-C(25)	101.9(2)
C(23)-C(24)-H(24A)	111.4
C(25)-C(24)-H(24A)	111.4
C(23)-C(24)-H(24B)	111.4
C(25)-C(24)-H(24B)	111.4
H(24A)-C(24)-H(24B)	109.3
C(26)-C(25)-C(30)	112.1(2)
C(26)-C(25)-C(24)	99.8(2)
C(30)-C(25)-C(24)	117.3(2)
C(26)-C(25)-C(32)	110.9(2)
C(30)-C(25)-C(32)	106.8(2)
C(24)-C(25)-C(32)	109.7(2)
C(27)-C(26)-O(3)	123.7(3)
C(27)-C(26)-C(25)	125.1(3)
O(3)-C(26)-C(25)	111.1(2)
C(26)-C(27)-C(28)	116.4(3)
C(26)-C(27)-C(38)	120.8(3)
C(28)-C(27)-C(38)	122.8(3)
C(29)-C(28)-C(27)	122.0(3)
C(29)-C(28)-C(31)	119.8(3)
C(27)-C(28)-C(31)	118.2(3)
C(28)-C(29)-C(30)	122.5(3)
C(28)-C(29)-H(29)	118.7
C(30)-C(29)-H(29)	118.7
O(4)-C(30)-C(29)	124.0(3)
O(4)-C(30)-C(25)	120.8(3)
C(29)-C(30)-C(25)	115.1(3)
C(28)-C(31)-H(31A)	109.5

C(28)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
C(28)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
C(33)-C(32)-C(37)	118.5(3)
C(33)-C(32)-C(25)	119.0(2)
C(37)-C(32)-C(25)	122.5(3)
C(32)-C(33)-C(34)	121.2(3)
C(32)-C(33)-H(33)	119.4
C(34)-C(33)-H(33)	119.4
C(35)-C(34)-C(33)	119.3(3)
C(35)-C(34)-H(34)	120.4
C(33)-C(34)-H(34)	120.3
C(34)-C(35)-C(36)	121.0(3)
C(34)-C(35)-H(35)	119.5
C(36)-C(35)-H(35)	119.5
C(35)-C(36)-C(37)	119.7(3)
C(35)-C(36)-H(36)	120.2
C(37)-C(36)-H(36)	120.2
C(32)-C(37)-C(36)	120.2(3)
C(32)-C(37)-H(37)	119.9
C(36)-C(37)-H(37)	119.9
C(39)-C(38)-C(43)	117.0(3)
C(39)-C(38)-C(27)	121.4(3)
C(43)-C(38)-C(27)	121.6(3)
C(38)-C(39)-C(40)	122.0(3)
C(38)-C(39)-H(39)	119.0
C(40)-C(39)-H(39)	119.0
C(41)-C(40)-C(39)	121.3(3)
C(41)-C(40)-H(40)	119.3
C(39)-C(40)-H(40)	119.3
C(40)-C(41)-C(42)	117.5(3)
C(40)-C(41)-C(44)	121.8(4)
C(42)-C(41)-C(44)	120.8(4)
C(41)-C(42)-C(43)	121.7(4)
C(41)-C(42)-H(42)	119.2
C(43)-C(42)-H(42)	119.2
C(38)-C(43)-C(42)	120.5(3)
C(38)-C(43)-H(43)	119.7
C(42)-C(43)-H(43)	119.7
C(41)-C(44)-H(44A)	109.4
C(41)-C(44)-H(44B)	109.5
H(44A)-C(44)-H(44B)	109.5
C(41)-C(44)-H(44C)	109.5
H(44A)-C(44)-H(44C)	109.5
H(44B)-C(44)-H(44C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd2855.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	51(1)	65(1)	45(1)	0(1)	3(1)	16(1)
O(2)	63(2)	72(2)	61(2)	11(1)	0(1)	-20(1)
O(3)	48(1)	53(1)	57(1)	21(1)	6(1)	11(1)
O(4)	96(2)	77(2)	62(2)	21(1)	33(1)	46(1)
C(1)	65(2)	61(2)	52(2)	-3(2)	6(2)	20(2)
C(2)	72(2)	54(2)	45(2)	9(2)	6(2)	14(2)
C(3)	47(2)	44(2)	34(2)	10(1)	4(1)	2(1)
C(4)	39(2)	56(2)	35(2)	11(1)	5(1)	6(1)
C(5)	45(2)	47(2)	33(2)	10(1)	6(1)	6(1)
C(6)	50(2)	57(2)	39(2)	16(1)	5(1)	8(2)
C(7)	46(2)	76(2)	46(2)	21(2)	8(2)	12(2)
C(8)	50(2)	66(2)	38(2)	22(2)	1(2)	2(2)
C(9)	65(2)	80(3)	70(3)	1(2)	12(2)	25(2)
C(10)	44(2)	51(2)	32(2)	7(1)	8(1)	7(1)
C(11)	66(2)	66(2)	41(2)	8(2)	2(2)	-7(2)
C(12)	69(2)	94(3)	37(2)	9(2)	-3(2)	1(2)
C(13)	93(3)	83(3)	40(2)	22(2)	13(2)	27(2)
C(14)	103(3)	63(2)	48(2)	17(2)	21(2)	-4(2)
C(15)	81(2)	58(2)	38(2)	10(2)	10(2)	-6(2)
C(16)	43(2)	43(2)	35(2)	3(1)	7(1)	7(1)
C(17)	62(2)	51(2)	39(2)	10(1)	8(2)	0(2)
C(18)	66(2)	60(2)	37(2)	6(2)	0(2)	3(2)
C(19)	52(2)	57(2)	56(2)	-2(2)	5(2)	4(2)
C(20)	58(2)	54(2)	63(2)	10(2)	12(2)	-8(2)
C(21)	61(2)	57(2)	40(2)	14(2)	13(2)	2(2)
C(22)	85(3)	94(3)	86(3)	-10(2)	-10(2)	-16(2)
C(23)	64(2)	54(2)	55(2)	15(2)	-2(2)	1(2)
C(24)	78(2)	52(2)	41(2)	13(2)	3(2)	12(2)
C(25)	55(2)	46(2)	32(2)	10(1)	10(1)	14(1)
C(26)	46(2)	51(2)	29(2)	12(1)	12(1)	14(2)
C(27)	55(2)	52(2)	29(2)	14(1)	9(1)	14(2)
C(28)	48(2)	61(2)	45(2)	16(2)	9(1)	9(2)
C(29)	48(2)	75(2)	51(2)	26(2)	16(2)	19(2)
C(30)	62(2)	62(2)	37(2)	19(2)	15(2)	28(2)
C(31)	70(3)	75(3)	108(3)	18(2)	6(2)	2(2)
C(32)	48(2)	49(2)	35(2)	12(1)	9(1)	11(1)
C(33)	68(2)	54(2)	45(2)	14(2)	12(2)	18(2)
C(34)	79(2)	64(2)	61(2)	32(2)	14(2)	23(2)
C(35)	73(2)	84(3)	48(2)	29(2)	10(2)	16(2)
C(36)	78(2)	85(3)	38(2)	16(2)	23(2)	23(2)
C(37)	66(2)	61(2)	42(2)	18(2)	17(2)	21(2)
C(38)	53(2)	46(2)	37(2)	3(1)	7(1)	9(1)
C(39)	91(3)	69(2)	54(2)	12(2)	27(2)	22(2)
C(40)	94(3)	66(3)	82(3)	-9(2)	29(2)	25(2)
C(41)	75(3)	56(2)	88(3)	-7(2)	-7(2)	22(2)
C(42)	126(3)	64(2)	58(2)	4(2)	-13(2)	41(2)
C(43)	109(3)	57(2)	39(2)	4(2)	7(2)	34(2)
C(44)	118(4)	77(3)	143(5)	-27(3)	-20(4)	60(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd2855.

	x	y	z	U(eq)
H(1A)	10525	-427	4098	71
H(1B)	10295	192	3404	71
H(2A)	7592	-958	4127	68
H(2B)	7687	-947	3250	68
H(7)	3424	1213	4427	65
H(9A)	3941	2743	5276	106
H(9B)	5725	3504	5217	106
H(9C)	5535	2784	5862	106
H(11)	5279	-268	2444	72
H(12)	5339	406	1307	83
H(13)	7112	1988	1207	83
H(14)	8896	2899	2229	87
H(15)	8825	2258	3375	73
H(17)	8406	2104	6358	62
H(18)	10529	3264	7141	67
H(20)	12072	4761	5423	72
H(21)	10003	3587	4628	63
H(22A)	12344	5204	7330	122
H(22B)	13314	5527	6629	122
H(22C)	13811	4599	7038	122
H(23A)	-923	4064	376	70
H(23B)	-665	3903	1229	70
H(24A)	1698	3647	119	68
H(24B)	1267	2930	764	68
H(29)	7158	5428	1144	67
H(31A)	7812	7237	1424	128
H(31B)	6406	7791	1826	128
H(31C)	6522	7765	955	128
H(33)	3795	2712	1728	64
H(34)	3674	2174	2910	77
H(35)	2642	3196	3859	79
H(36)	1758	4755	3654	78
H(37)	1961	5329	2479	64
H(39)	2182	7527	2268	83
H(40)	1079	9058	2276	96
H(42)	2100	9160	149	98
H(43)	3258	7631	138	80
H(44A)	-397	10208	1413	134
H(44B)	631	10468	713	134
H(44C)	1516	10873	1528	134

Table 6. Torsion angles [deg] for cd2855.

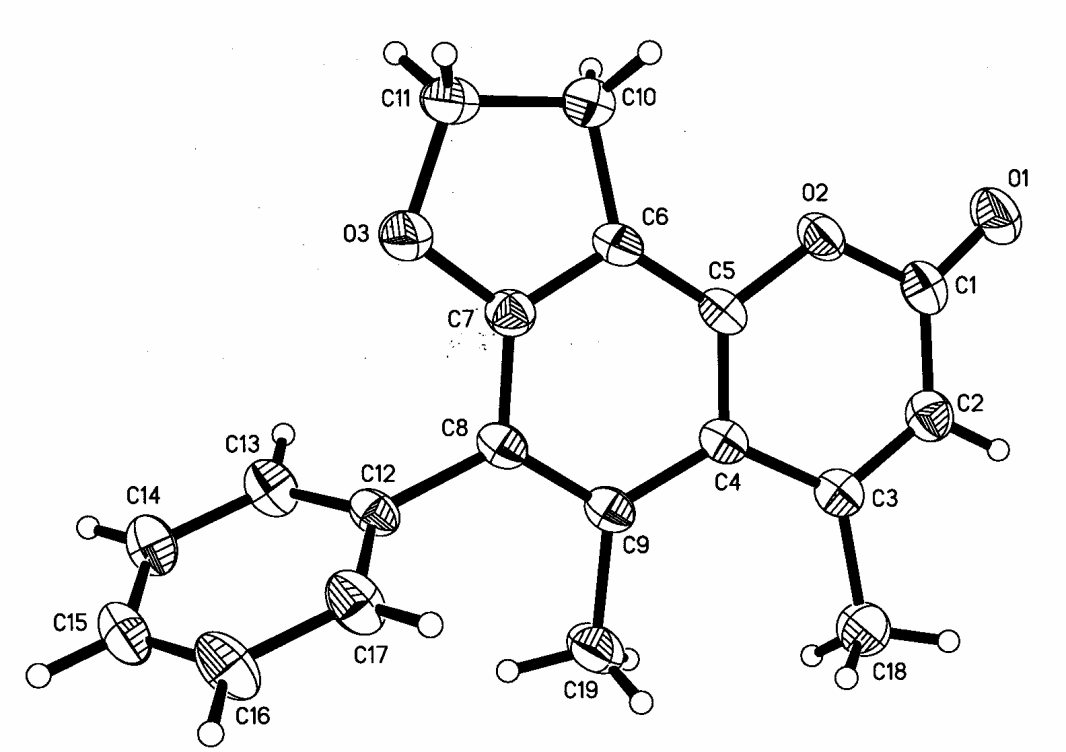
C(4)-O(1)-C(1)-C(2)	-16.5(3)
O(1)-C(1)-C(2)-C(3)	32.8(3)
C(1)-C(2)-C(3)-C(4)	-35.7(3)
C(1)-C(2)-C(3)-C(8)	-158.0(2)
C(1)-C(2)-C(3)-C(10)	80.1(3)
C(1)-O(1)-C(4)-C(5)	169.8(3)
C(1)-O(1)-C(4)-C(3)	-8.0(3)
C(8)-C(3)-C(4)-C(5)	-23.6(4)
C(2)-C(3)-C(4)-C(5)	-149.6(3)
C(10)-C(3)-C(4)-C(5)	94.1(3)
C(8)-C(3)-C(4)-O(1)	154.1(2)
C(2)-C(3)-C(4)-O(1)	28.2(3)
C(10)-C(3)-C(4)-O(1)	-88.2(3)
O(1)-C(4)-C(5)-C(6)	-172.1(2)
C(3)-C(4)-C(5)-C(6)	5.3(4)
O(1)-C(4)-C(5)-C(16)	8.0(5)
C(3)-C(4)-C(5)-C(16)	-174.6(2)
C(4)-C(5)-C(6)-C(7)	7.7(4)
C(16)-C(5)-C(6)-C(7)	-172.4(3)
C(4)-C(5)-C(6)-C(9)	-175.3(3)
C(16)-C(5)-C(6)-C(9)	4.6(4)
C(5)-C(6)-C(7)-C(8)	0.2(5)
C(9)-C(6)-C(7)-C(8)	-176.7(3)
C(6)-C(7)-C(8)-O(2)	164.4(3)
C(6)-C(7)-C(8)-C(3)	-19.3(4)
C(4)-C(3)-C(8)-O(2)	-154.7(3)
C(2)-C(3)-C(8)-O(2)	-39.1(4)
C(10)-C(3)-C(8)-O(2)	85.2(3)
C(4)-C(3)-C(8)-C(7)	28.8(3)
C(2)-C(3)-C(8)-C(7)	144.5(3)
C(10)-C(3)-C(8)-C(7)	-91.2(3)
C(4)-C(3)-C(10)-C(15)	3.1(4)
C(8)-C(3)-C(10)-C(15)	124.9(3)
C(2)-C(3)-C(10)-C(15)	-106.4(3)
C(4)-C(3)-C(10)-C(11)	-178.2(3)
C(8)-C(3)-C(10)-C(11)	-56.4(3)
C(2)-C(3)-C(10)-C(11)	72.3(3)
C(15)-C(10)-C(11)-C(12)	-0.4(5)
C(3)-C(10)-C(11)-C(12)	-179.2(3)
C(10)-C(11)-C(12)-C(13)	0.1(5)
C(11)-C(12)-C(13)-C(14)	0.7(6)
C(12)-C(13)-C(14)-C(15)	-1.3(6)
C(11)-C(10)-C(15)-C(14)	-0.1(5)
C(3)-C(10)-C(15)-C(14)	178.6(3)
C(13)-C(14)-C(15)-C(10)	1.0(5)
C(4)-C(5)-C(16)-C(21)	71.4(4)
C(6)-C(5)-C(16)-C(21)	-108.5(3)
C(4)-C(5)-C(16)-C(17)	-106.6(3)
C(6)-C(5)-C(16)-C(17)	73.5(4)
C(21)-C(16)-C(17)-C(18)	-0.7(4)
C(5)-C(16)-C(17)-C(18)	177.4(3)
C(16)-C(17)-C(18)-C(19)	1.6(5)
C(17)-C(18)-C(19)-C(20)	-1.6(5)
C(17)-C(18)-C(19)-C(22)	178.4(3)
C(18)-C(19)-C(20)-C(21)	0.8(5)
C(22)-C(19)-C(20)-C(21)	-179.2(3)
C(17)-C(16)-C(21)-C(20)	-0.1(4)
C(5)-C(16)-C(21)-C(20)	-178.2(3)
C(19)-C(20)-C(21)-C(16)	0.0(5)
C(26)-O(3)-C(23)-C(24)	-13.8(3)
O(3)-C(23)-C(24)-C(25)	31.0(3)
C(23)-C(24)-C(25)-C(26)	-34.9(3)
C(23)-C(24)-C(25)-C(30)	-156.2(3)
C(23)-C(24)-C(25)-C(32)	81.7(3)
C(23)-O(3)-C(26)-C(27)	168.0(3)
C(23)-O(3)-C(26)-C(25)	-10.3(3)
C(30)-C(25)-C(26)-C(27)	-24.2(4)
C(24)-C(25)-C(26)-C(27)	-149.2(3)

C(32)-C(25)-C(26)-C(27)	95.2(3)
C(30)-C(25)-C(26)-O(3)	154.0(2)
C(24)-C(25)-C(26)-O(3)	29.0(3)
C(32)-C(25)-C(26)-O(3)	-86.6(3)
O(3)-C(26)-C(27)-C(28)	-172.5(2)
C(25)-C(26)-C(27)-C(28)	5.5(4)
O(3)-C(26)-C(27)-C(38)	5.3(4)
C(25)-C(26)-C(27)-C(38)	-176.7(2)
C(26)-C(27)-C(28)-C(29)	8.4(4)
C(38)-C(27)-C(28)-C(29)	-169.5(3)
C(26)-C(27)-C(28)-C(31)	-175.2(3)
C(38)-C(27)-C(28)-C(31)	7.0(4)
C(27)-C(28)-C(29)-C(30)	-0.6(4)
C(31)-C(28)-C(29)-C(30)	-177.0(3)
C(28)-C(29)-C(30)-O(4)	164.3(3)
C(28)-C(29)-C(30)-C(25)	-19.2(4)
C(26)-C(25)-C(30)-O(4)	-154.0(3)
C(24)-C(25)-C(30)-O(4)	-39.3(4)
C(32)-C(25)-C(30)-O(4)	84.3(3)
C(26)-C(25)-C(30)-C(29)	29.4(3)
C(24)-C(25)-C(30)-C(29)	144.1(3)
C(32)-C(25)-C(30)-C(29)	-92.3(3)
C(26)-C(25)-C(32)-C(33)	-177.8(3)
C(30)-C(25)-C(32)-C(33)	-55.4(3)
C(24)-C(25)-C(32)-C(33)	72.8(3)
C(26)-C(25)-C(32)-C(37)	3.3(4)
C(30)-C(25)-C(32)-C(37)	125.8(3)
C(24)-C(25)-C(32)-C(37)	-106.0(3)
C(37)-C(32)-C(33)-C(34)	0.5(5)
C(25)-C(32)-C(33)-C(34)	-178.4(3)
C(32)-C(33)-C(34)-C(35)	0.6(5)
C(33)-C(34)-C(35)-C(36)	-0.5(5)
C(34)-C(35)-C(36)-C(37)	-0.6(5)
C(33)-C(32)-C(37)-C(36)	-1.6(5)
C(25)-C(32)-C(37)-C(36)	177.2(3)
C(35)-C(36)-C(37)-C(32)	1.7(5)
C(26)-C(27)-C(38)-C(39)	77.7(4)
C(28)-C(27)-C(38)-C(39)	-104.6(4)
C(26)-C(27)-C(38)-C(43)	-102.1(4)
C(28)-C(27)-C(38)-C(43)	75.6(4)
C(43)-C(38)-C(39)-C(40)	1.2(5)
C(27)-C(38)-C(39)-C(40)	-178.7(3)
C(38)-C(39)-C(40)-C(41)	0.0(6)
C(39)-C(40)-C(41)-C(42)	-0.5(6)
C(39)-C(40)-C(41)-C(44)	-179.6(3)
C(40)-C(41)-C(42)-C(43)	0.0(6)
C(44)-C(41)-C(42)-C(43)	179.1(3)
C(39)-C(38)-C(43)-C(42)	-1.7(5)
C(27)-C(38)-C(43)-C(42)	178.1(3)
C(41)-C(42)-C(43)-C(38)	1.2(6)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd2855 [A and deg.].

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



The crystal data of **4a** have been deposited in CCDC with number 669369. Empirical Formula: $C_{19}H_{16}O_3$; Formula Weight: 292.32; Crystal size: 0.428 x 0.327 x 0.119; Crystal Color, Habit: colorless, prismatic; Crystal System: Monoclinic; Lattice Type: Primitive; Lattice Parameters: a = 10.9030(18)Å, b = 16.171(3)Å, c = 8.8944(15)Å, $\alpha = 90^o$, $\beta = 109.901(3)^o$, $\gamma = 90^o$, V = 1474.6(4)Å^3; Space group: P2(1)/c; Z = 4; D_{calc} = 1.317 g/cm^3; F_{000} = 616; R1 = 0.0605, wR2 = 0.1253. Diffractometer: Rigaku AFC7R.

Table 1. Crystal data and structure refinement for cd27586.

Identification code	cd27586
Empirical formula	C19 H16 O3
Formula weight	292.32
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 10.9030(18) A alpha = 90 deg. b = 16.171(3) A beta = 109.901(3) deg. c = 8.8944(15) A gamma = 90 deg.
Volume	1474.6(4) A^3
Z, Calculated density	4, 1.317 Mg/m^3
Absorption coefficient	0.088 mm^-1
F(000)	616
Crystal size	0.428 x 0.327 x 0.119 mm
Theta range for data collection	1.99 to 26.99 deg.
Limiting indices	-13<=h<=13, -20<=k<=20, -10<=l<=11
Reflections collected / unique	8607 / 3210 [R(int) = 0.1517]
Completeness to theta = 26.99	99.5 %
Absorption correction	Empirical
Max. and min. transmission	1.0000 and 0.8183
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3210 / 0 / 202
Goodness-of-fit on F^2	0.882
Final R indices [I>2sigma(I)]	R1 = 0.0605, wR2 = 0.1253
R indices (all data)	R1 = 0.1004, wR2 = 0.1397
Extinction coefficient	0.0152(19)
Largest diff. peak and hole	0.236 and -0.205 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd27586.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	11095(2)	11748(1)	8155(2)	69(1)
O(2)	9703(1)	10720(1)	7418(1)	49(1)
O(3)	6652(2)	8662(1)	6488(2)	63(1)
C(1)	10329(2)	11388(1)	7036(3)	50(1)
C(2)	9987(2)	11572(1)	5388(2)	50(1)
C(3)	9053(2)	11171(1)	4212(2)	44(1)
C(4)	8319(2)	10511(1)	4634(2)	39(1)
C(5)	8729(2)	10301(1)	6256(2)	41(1)
C(6)	8157(2)	9681(1)	6845(2)	43(1)
C(7)	7163(2)	9247(1)	5782(2)	44(1)
C(8)	6679(2)	9410(1)	4149(2)	41(1)
C(9)	7243(2)	10051(1)	3568(2)	43(1)
C(10)	8340(2)	9386(2)	8507(2)	60(1)
C(11)	7388(3)	8667(2)	8177(3)	77(1)
C(12)	5581(2)	8888(1)	3108(2)	41(1)
C(13)	4359(2)	8927(1)	3258(2)	54(1)
C(14)	3361(2)	8433(2)	2344(3)	59(1)
C(15)	3548(2)	7897(1)	1273(2)	57(1)
C(16)	4749(2)	7850(2)	1096(3)	65(1)
C(17)	5758(2)	8344(2)	2011(2)	59(1)
C(18)	8857(2)	11438(2)	2527(2)	64(1)
C(19)	6623(3)	10276(2)	1825(2)	72(1)

Table 3. Bond lengths [A] and angles [deg] for cd27586.

O(1)-C(1)	1.209(2)
O(2)-C(1)	1.380(3)
O(2)-C(5)	1.381(2)
O(3)-C(7)	1.356(2)
O(3)-C(11)	1.442(2)
C(1)-C(2)	1.416(3)
C(2)-C(3)	1.352(3)
C(2)-H(2)	0.9300
C(3)-C(4)	1.457(3)
C(3)-C(18)	1.503(3)
C(4)-C(5)	1.399(2)
C(4)-C(9)	1.440(3)
C(5)-C(6)	1.376(3)
C(6)-C(7)	1.365(3)
C(6)-C(10)	1.500(3)
C(7)-C(8)	1.392(2)
C(8)-C(9)	1.391(3)
C(8)-C(12)	1.499(3)
C(9)-C(19)	1.509(3)
C(10)-C(11)	1.518(3)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(12)-C(17)	1.375(3)
C(12)-C(13)	1.385(3)
C(13)-C(14)	1.372(3)
C(13)-H(13)	0.9300
C(14)-C(15)	1.354(3)
C(14)-H(14)	0.9300
C(15)-C(16)	1.373(3)
C(15)-H(15)	0.9300
C(16)-C(17)	1.379(3)
C(16)-H(16)	0.9300
C(17)-H(17)	0.9300
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600

C(1)-O(2)-C(5)	121.20(16)
C(7)-O(3)-C(11)	107.88(16)
O(1)-C(1)-O(2)	115.8(2)
O(1)-C(1)-C(2)	128.0(2)
O(2)-C(1)-C(2)	116.24(18)
C(3)-C(2)-C(1)	124.3(2)
C(3)-C(2)-H(2)	117.9
C(1)-C(2)-H(2)	117.9
C(2)-C(3)-C(4)	119.14(18)
C(2)-C(3)-C(18)	116.6(2)
C(4)-C(3)-C(18)	124.23(18)
C(5)-C(4)-C(9)	117.12(19)
C(5)-C(4)-C(3)	115.66(17)
C(9)-C(4)-C(3)	127.21(17)
C(6)-C(5)-O(2)	113.91(17)
C(6)-C(5)-C(4)	122.90(18)
O(2)-C(5)-C(4)	123.14(19)
C(7)-C(6)-C(5)	117.82(18)
C(7)-C(6)-C(10)	109.1(2)
C(5)-C(6)-C(10)	132.96(18)
O(3)-C(7)-C(6)	113.10(17)
O(3)-C(7)-C(8)	123.09(18)
C(6)-C(7)-C(8)	123.7(2)
C(9)-C(8)-C(7)	118.04(18)
C(9)-C(8)-C(12)	123.44(17)
C(7)-C(8)-C(12)	118.52(19)

C(8)-C(9)-C(4)	120.33(17)
C(8)-C(9)-C(19)	117.43(18)
C(4)-C(9)-C(19)	122.10(19)
C(6)-C(10)-C(11)	101.52(16)
C(6)-C(10)-H(10A)	111.5
C(11)-C(10)-H(10A)	111.5
C(6)-C(10)-H(10B)	111.5
C(11)-C(10)-H(10B)	111.5
H(10A)-C(10)-H(10B)	109.3
O(3)-C(11)-C(10)	107.92(17)
O(3)-C(11)-H(11A)	110.1
C(10)-C(11)-H(11A)	110.1
O(3)-C(11)-H(11B)	110.1
C(10)-C(11)-H(11B)	110.1
H(11A)-C(11)-H(11B)	108.4
C(17)-C(12)-C(13)	117.98(19)
C(17)-C(12)-C(8)	121.55(19)
C(13)-C(12)-C(8)	120.45(18)
C(14)-C(13)-C(12)	120.8(2)
C(14)-C(13)-H(13)	119.6
C(12)-C(13)-H(13)	119.6
C(15)-C(14)-C(13)	120.7(2)
C(15)-C(14)-H(14)	119.6
C(13)-C(14)-H(14)	119.6
C(14)-C(15)-C(16)	119.6(2)
C(14)-C(15)-H(15)	120.2
C(16)-C(15)-H(15)	120.2
C(15)-C(16)-C(17)	120.0(2)
C(15)-C(16)-H(16)	120.0
C(17)-C(16)-H(16)	120.0
C(12)-C(17)-C(16)	120.9(2)
C(12)-C(17)-H(17)	119.6
C(16)-C(17)-H(17)	119.6
C(3)-C(18)-H(18A)	109.5
C(3)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(3)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(9)-C(19)-H(19A)	109.5
C(9)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(9)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd27586.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	59(1)	71(1)	62(1)	-12(1)	4(1)	-21(1)
O(2)	44(1)	52(1)	43(1)	-4(1)	3(1)	-7(1)
O(3)	68(1)	61(1)	48(1)	9(1)	6(1)	-19(1)
C(1)	42(1)	47(1)	56(1)	-8(1)	10(1)	-2(1)
C(2)	48(1)	43(1)	55(1)	1(1)	13(1)	-2(1)
C(3)	46(1)	37(1)	46(1)	0(1)	11(1)	5(1)
C(4)	41(1)	35(1)	38(1)	-1(1)	8(1)	5(1)
C(5)	38(1)	40(1)	38(1)	-5(1)	4(1)	3(1)
C(6)	44(1)	45(1)	34(1)	0(1)	5(1)	2(1)
C(7)	46(1)	41(1)	42(1)	3(1)	10(1)	1(1)
C(8)	41(1)	40(1)	38(1)	-2(1)	6(1)	5(1)
C(9)	43(1)	41(1)	39(1)	-1(1)	7(1)	4(1)
C(10)	61(2)	67(2)	47(1)	5(1)	10(1)	-13(1)
C(11)	88(2)	83(2)	44(1)	13(1)	2(1)	-30(2)
C(12)	41(1)	35(1)	38(1)	-1(1)	2(1)	4(1)
C(13)	48(1)	50(2)	59(1)	-10(1)	12(1)	4(1)
C(14)	42(1)	62(2)	65(2)	-5(1)	9(1)	-3(1)
C(15)	50(2)	43(1)	60(1)	-1(1)	-5(1)	-4(1)
C(16)	59(2)	56(2)	68(2)	-26(1)	5(1)	3(1)
C(17)	45(1)	62(2)	63(2)	-17(1)	10(1)	6(1)
C(18)	70(2)	56(2)	58(2)	11(1)	14(1)	-13(1)
C(19)	75(2)	78(2)	45(1)	9(1)	-3(1)	-21(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd27586.

	x	y	z	U(eq)
H(2)	10435	11997	5096	60
H(10A)	8116	9813	9135	72
H(10B)	9229	9204	9055	72
H(11A)	7858	8151	8488	92
H(11B)	6806	8732	8786	92
H(13)	4213	9293	3986	65
H(14)	2547	8466	2462	71
H(15)	2867	7563	660	68
H(16)	4882	7484	359	78
H(17)	6569	8309	1885	70
H(18A)	8035	11717	2093	95
H(18B)	8863	10960	1888	95
H(18C)	9548	11805	2526	95
H(19A)	7178	10098	1249	108
H(19B)	6507	10864	1724	108
H(19C)	5791	10008	1394	108

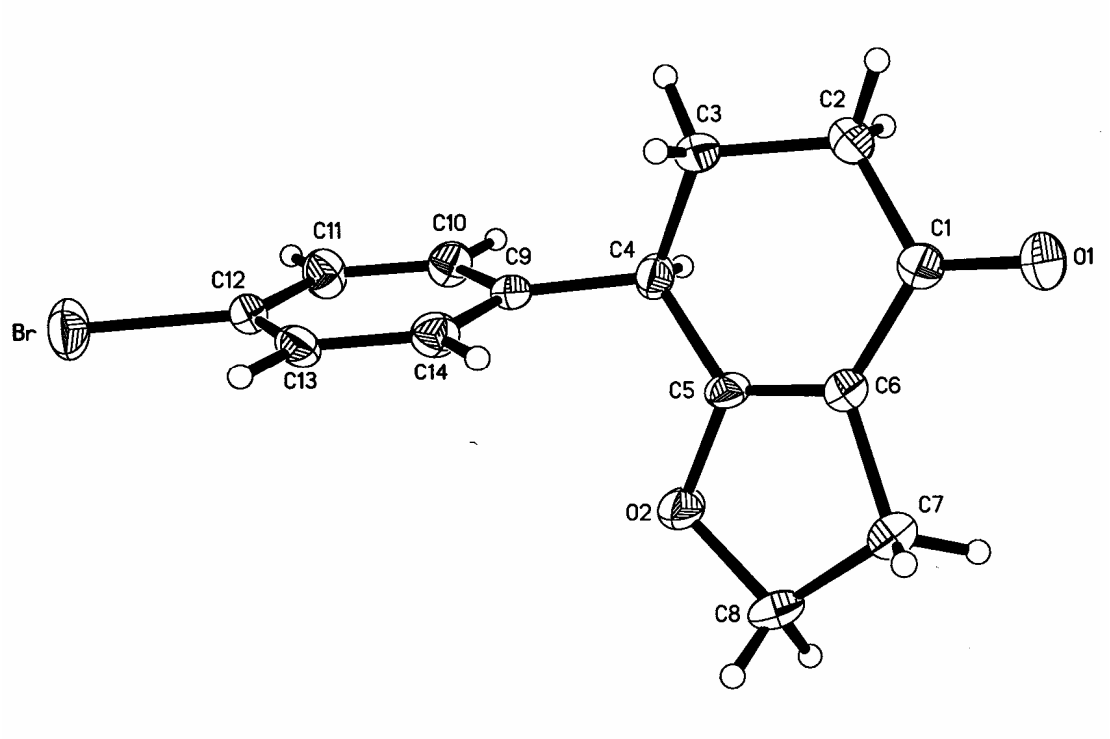
Table 6. Torsion angles [deg] for cd27586.

C(5)-O(2)-C(1)-O(1)	174.62(18)
C(5)-O(2)-C(1)-C(2)	-4.7(3)
O(1)-C(1)-C(2)-C(3)	-175.3(2)
O(2)-C(1)-C(2)-C(3)	3.9(3)
C(1)-C(2)-C(3)-C(4)	1.0(3)
C(1)-C(2)-C(3)-C(18)	-178.18(19)
C(2)-C(3)-C(4)-C(5)	-5.0(3)
C(18)-C(3)-C(4)-C(5)	174.09(19)
C(2)-C(3)-C(4)-C(9)	175.91(19)
C(18)-C(3)-C(4)-C(9)	-5.0(3)
C(1)-O(2)-C(5)-C(6)	-176.66(19)
C(1)-O(2)-C(5)-C(4)	0.6(3)
C(9)-C(4)-C(5)-C(6)	0.5(3)
C(3)-C(4)-C(5)-C(6)	-178.65(19)
C(9)-C(4)-C(5)-O(2)	-176.49(17)
C(3)-C(4)-C(5)-O(2)	4.4(3)
O(2)-C(5)-C(6)-C(7)	178.46(17)
C(4)-C(5)-C(6)-C(7)	1.2(3)
O(2)-C(5)-C(6)-C(10)	2.8(3)
C(4)-C(5)-C(6)-C(10)	-174.5(2)
C(11)-O(3)-C(7)-C(6)	-2.5(3)
C(11)-O(3)-C(7)-C(8)	-179.7(2)
C(5)-C(6)-C(7)-O(3)	-178.82(18)
C(10)-C(6)-C(7)-O(3)	-2.2(3)
C(5)-C(6)-C(7)-C(8)	-1.5(3)
C(10)-C(6)-C(7)-C(8)	175.12(19)
O(3)-C(7)-C(8)-C(9)	177.08(19)
C(6)-C(7)-C(8)-C(9)	0.1(3)
O(3)-C(7)-C(8)-C(12)	-2.9(3)
C(6)-C(7)-C(8)-C(12)	-179.9(2)
C(7)-C(8)-C(9)-C(4)	1.7(3)
C(12)-C(8)-C(9)-C(4)	-178.26(18)
C(7)-C(8)-C(9)-C(19)	-174.10(19)
C(12)-C(8)-C(9)-C(19)	5.9(3)
C(5)-C(4)-C(9)-C(8)	-2.0(3)
C(3)-C(4)-C(9)-C(8)	177.0(2)
C(5)-C(4)-C(9)-C(19)	173.6(2)
C(3)-C(4)-C(9)-C(19)	-7.3(3)
C(7)-C(6)-C(10)-C(11)	5.4(3)
C(5)-C(6)-C(10)-C(11)	-178.6(2)
C(7)-O(3)-C(11)-C(10)	6.0(3)
C(6)-C(10)-C(11)-O(3)	-6.8(3)
C(9)-C(8)-C(12)-C(17)	68.5(3)
C(7)-C(8)-C(12)-C(17)	-111.5(2)
C(9)-C(8)-C(12)-C(13)	-113.2(2)
C(7)-C(8)-C(12)-C(13)	66.8(3)
C(17)-C(12)-C(13)-C(14)	0.4(3)
C(8)-C(12)-C(13)-C(14)	-178.0(2)
C(12)-C(13)-C(14)-C(15)	-0.2(3)
C(13)-C(14)-C(15)-C(16)	-0.2(4)
C(14)-C(15)-C(16)-C(17)	0.2(4)
C(13)-C(12)-C(17)-C(16)	-0.4(3)
C(8)-C(12)-C(17)-C(16)	178.0(2)
C(15)-C(16)-C(17)-C(12)	0.0(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd27586 [A and deg.].

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



The crystal data of **5c** have been deposited in CCDC with number 635461. Empirical Formula: $C_{14}H_{14}BrO_{2.5}$; Formula Weight: 302.16; Crystal size: 0.503 x 0.483 x 0.437; Crystal Color, Habit: colorless, prismatic; Crystal System: Orthorhombic; Lattice Type: Primitive; Lattice Parameters: a = 45.814(7)Å, b = 9.4317(13)Å, c = 11.6643(16)Å, $\alpha = 90^o$, $\beta = 90^o$, $\gamma = 90^o$, V = 5040.2(12)Å^3; Space group: Fdd2; Z = 16; D_{calc} = 1.593 g/cm^3; F_{000} = 2448; R1 = 0.0469, wR2 = 0.0979. Diffractometer: Rigaku AFC7R.

Table 1. Crystal data and structure refinement for cd2754.

Identification code	cd2754
Empirical formula	C14 H14 Br O2.50
Formula weight	302.16
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, Fdd2
Unit cell dimensions	a = 45.814(7) A alpha = 90 deg. b = 9.4317(13) A beta = 90 deg. c = 11.6643(16) A gamma = 90 deg.
Volume	5040.2(12) A^3
Z, Calculated density	16, 1.593 Mg/m^3
Absorption coefficient	3.254 mm^-1
F(000)	2448
Crystal size	0.503 x 0.483 x 0.437 mm
Theta range for data collection	2.81 to 27.50 deg.
Limiting indices	-59<=h<=57, -8<=k<=12, -15<=l<=13
Reflections collected / unique	7284 / 2671 [R(int) = 0.0815]
Completeness to theta = 27.50	99.7 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.71194
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2671 / 2 / 167
Goodness-of-fit on F^2	0.916
Final R indices [I>2sigma(I)]	R1 = 0.0469, wR2 = 0.0979
R indices (all data)	R1 = 0.0612, wR2 = 0.1013
Absolute structure parameter	-0.021(12)
Largest diff. peak and hole	0.425 and -0.623 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for cd2754.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Br	1079(1)	-1647(1)	3017(1)	58(1)
O(1)	98(1)	7746(4)	1778(3)	61(1)
O(2)	627(1)	4915(4)	4198(3)	50(1)
O(3)	0	10000	3366(5)	63(2)
C(1)	278(1)	6795(5)	1897(4)	40(1)
C(2)	444(1)	6227(6)	893(4)	52(1)
C(3)	563(1)	4725(5)	1075(4)	46(1)
C(4)	739(1)	4578(5)	2184(4)	36(1)
C(5)	563(1)	5246(4)	3120(4)	33(1)
C(6)	350(1)	6215(4)	2999(4)	35(1)
C(7)	243(1)	6654(5)	4156(5)	51(1)
C(8)	437(1)	5776(6)	4939(4)	60(2)
C(9)	825(1)	3068(4)	2410(3)	32(1)
C(10)	1106(1)	2612(5)	2238(4)	39(1)
C(11)	1187(1)	1228(5)	2410(4)	44(1)
C(12)	975(1)	280(4)	2768(3)	36(1)
C(13)	694(1)	685(4)	2951(4)	36(1)
C(14)	621(1)	2079(5)	2772(4)	38(1)

Table 3. Bond lengths [A] and angles [deg] for cd2754.

Br-C(12)	1.901(4)
O(1)-C(1)	1.227(5)
O(2)-C(5)	1.329(5)
O(2)-C(8)	1.471(6)
O(3)-H(3)	0.91(2)
C(1)-C(6)	1.436(7)
C(1)-C(2)	1.496(7)
C(2)-C(3)	1.533(7)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(4)	1.529(7)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.496(6)
C(4)-C(9)	1.502(6)
C(4)-H(4)	0.98(4)
C(5)-C(6)	1.343(6)
C(6)-C(7)	1.494(7)
C(7)-C(8)	1.520(8)
C(7)-H(7A)	0.9700
C(7)-H(7B)	0.9700
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(9)-C(10)	1.375(6)
C(9)-C(14)	1.385(6)
C(10)-C(11)	1.373(7)
C(10)-H(10)	0.9300
C(11)-C(12)	1.385(7)
C(11)-H(11)	0.9300
C(12)-C(13)	1.363(6)
C(13)-C(14)	1.373(6)
C(13)-H(13)	0.9300
C(14)-H(14)	0.9300
C(5)-O(2)-C(8)	107.2(4)
O(1)-C(1)-C(6)	122.3(5)
O(1)-C(1)-C(2)	121.1(5)
C(6)-C(1)-C(2)	116.6(4)
C(1)-C(2)-C(3)	113.8(4)
C(1)-C(2)-H(2A)	108.8
C(3)-C(2)-H(2A)	108.8
C(1)-C(2)-H(2B)	108.8
C(3)-C(2)-H(2B)	108.8
H(2A)-C(2)-H(2B)	107.7
C(4)-C(3)-C(2)	112.8(4)
C(4)-C(3)-H(3A)	109.0
C(2)-C(3)-H(3A)	109.0
C(4)-C(3)-H(3B)	109.0
C(2)-C(3)-H(3B)	109.0
H(3A)-C(3)-H(3B)	107.8
C(5)-C(4)-C(9)	114.3(4)
C(5)-C(4)-C(3)	107.3(3)
C(9)-C(4)-C(3)	111.8(4)
C(5)-C(4)-H(4)	107(2)
C(9)-C(4)-H(4)	107(2)
C(3)-C(4)-H(4)	109(2)
O(2)-C(5)-C(6)	114.8(4)
O(2)-C(5)-C(4)	118.2(4)
C(6)-C(5)-C(4)	126.9(4)
C(5)-C(6)-C(1)	121.3(4)
C(5)-C(6)-C(7)	109.4(4)
C(1)-C(6)-C(7)	128.8(4)
C(6)-C(7)-C(8)	101.5(4)
C(6)-C(7)-H(7A)	111.5
C(8)-C(7)-H(7A)	111.5
C(6)-C(7)-H(7B)	111.5
C(8)-C(7)-H(7B)	111.5
H(7A)-C(7)-H(7B)	109.3

O(2)-C(8)-C(7)	107.1(4)
O(2)-C(8)-H(8A)	110.3
C(7)-C(8)-H(8A)	110.3
O(2)-C(8)-H(8B)	110.3
C(7)-C(8)-H(8B)	110.3
H(8A)-C(8)-H(8B)	108.6
C(10)-C(9)-C(14)	117.8(4)
C(10)-C(9)-C(4)	121.1(4)
C(14)-C(9)-C(4)	121.0(4)
C(11)-C(10)-C(9)	122.1(4)
C(11)-C(10)-H(10)	119.0
C(9)-C(10)-H(10)	119.0
C(10)-C(11)-C(12)	117.9(4)
C(10)-C(11)-H(11)	121.1
C(12)-C(11)-H(11)	121.1
C(13)-C(12)-C(11)	122.0(4)
C(13)-C(12)-Br	118.8(3)
C(11)-C(12)-Br	119.2(3)
C(12)-C(13)-C(14)	118.4(4)
C(12)-C(13)-H(13)	120.8
C(14)-C(13)-H(13)	120.8
C(13)-C(14)-C(9)	121.8(4)
C(13)-C(14)-H(14)	119.1
C(9)-C(14)-H(14)	119.1

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for cd2754.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Br	70(1)	34(1)	72(1)	5(1)	-7(1)	9(1)
O(1)	74(2)	57(2)	51(2)	2(2)	-6(2)	35(2)
O(2)	73(2)	50(2)	29(2)	-8(2)	-12(2)	22(2)
O(3)	57(3)	55(4)	78(4)	0	0	5(3)
C(1)	45(3)	40(3)	34(3)	-2(2)	-5(2)	6(2)
C(2)	74(3)	48(3)	35(3)	7(2)	6(2)	23(3)
C(3)	68(3)	43(3)	27(2)	-1(2)	9(2)	17(2)
C(4)	38(2)	29(2)	40(3)	-1(2)	5(2)	5(2)
C(5)	43(2)	33(2)	23(2)	-7(2)	-3(2)	-4(2)
C(6)	41(2)	33(2)	31(2)	-4(2)	0(2)	4(2)
C(7)	74(4)	43(3)	37(3)	-7(2)	6(3)	12(2)
C(8)	97(4)	57(3)	26(2)	-13(2)	5(3)	13(3)
C(9)	40(2)	34(2)	22(2)	-1(2)	1(2)	4(2)
C(10)	37(2)	38(2)	43(2)	-2(2)	8(2)	-3(2)
C(11)	37(2)	43(3)	51(3)	6(2)	4(2)	10(2)
C(12)	51(2)	27(2)	30(2)	2(2)	-3(2)	1(2)
C(13)	44(2)	36(2)	30(2)	7(2)	3(2)	-6(2)
C(14)	35(2)	41(2)	36(3)	0(2)	3(2)	2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for cd2754.

	x	y	z	U(eq)
H(2A)	606	6858	730	63
H(2B)	317	6224	227	63
H(3A)	401	4062	1092	55
H(3B)	687	4474	431	55
H(7A)	39	6419	4259	62
H(7B)	271	7661	4282	62
H(8A)	554	6391	5423	72
H(8B)	319	5170	5425	72
H(10)	1246	3262	1997	47
H(11)	1379	936	2290	52
H(13)	554	34	3192	44
H(14)	429	2368	2896	45
H(3)	70(17)	9340(60)	2870(50)	120(30)
H(4)	919(9)	5120(50)	2110(30)	28(10)

Table 6. Torsion angles [deg] for cd2754.

O(1)-C(1)-C(2)-C(3)	-157.2(5)
C(6)-C(1)-C(2)-C(3)	25.4(7)
C(1)-C(2)-C(3)-C(4)	-52.0(6)
C(2)-C(3)-C(4)-C(5)	47.5(5)
C(2)-C(3)-C(4)-C(9)	173.6(4)
C(8)-O(2)-C(5)-C(6)	-0.8(5)
C(8)-O(2)-C(5)-C(4)	176.4(4)
C(9)-C(4)-C(5)-O(2)	36.9(5)
C(3)-C(4)-C(5)-O(2)	161.5(4)
C(9)-C(4)-C(5)-C(6)	-146.4(4)
C(3)-C(4)-C(5)-C(6)	-21.7(6)
O(2)-C(5)-C(6)-C(1)	173.1(4)
C(4)-C(5)-C(6)-C(1)	-3.8(7)
O(2)-C(5)-C(6)-C(7)	-0.1(5)
C(4)-C(5)-C(6)-C(7)	-177.0(4)
O(1)-C(1)-C(6)-C(5)	-175.2(5)
C(2)-C(1)-C(6)-C(5)	2.2(7)
O(1)-C(1)-C(6)-C(7)	-3.4(8)
C(2)-C(1)-C(6)-C(7)	173.9(5)
C(5)-C(6)-C(7)-C(8)	0.9(5)
C(1)-C(6)-C(7)-C(8)	-171.7(5)
C(5)-O(2)-C(8)-C(7)	1.3(5)
C(6)-C(7)-C(8)-O(2)	-1.3(5)
C(5)-C(4)-C(9)-C(10)	-132.9(4)
C(3)-C(4)-C(9)-C(10)	105.0(5)
C(5)-C(4)-C(9)-C(14)	48.8(5)
C(3)-C(4)-C(9)-C(14)	-73.4(5)
C(14)-C(9)-C(10)-C(11)	0.2(7)
C(4)-C(9)-C(10)-C(11)	-178.2(4)
C(9)-C(10)-C(11)-C(12)	0.0(7)
C(10)-C(11)-C(12)-C(13)	-0.1(7)
C(10)-C(11)-C(12)-Br	-180.0(4)
C(11)-C(12)-C(13)-C(14)	0.1(7)
Br-C(12)-C(13)-C(14)	179.9(3)
C(12)-C(13)-C(14)-C(9)	0.1(7)
C(10)-C(9)-C(14)-C(13)	-0.3(6)
C(4)-C(9)-C(14)-C(13)	178.1(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd2754 [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3)...O(1)	0.91(2)	1.97(3)	2.855(5)	163(8)

Symmetry transformations used to generate equivalent atoms:

Reference:

1) Yang, Y.-H.; Shi, M. *Org. Lett.* **2006**, *8*, 1709-1712.