Electronic Supplementary Information

Direct Carboxamidation of Cyclic 2-Diazo-1,3-diketones by $Rh_2(OAc)_4$-catalyzed Isocyanide Insertion-Hydrolysis

Xinwei He, Zhiyu Yu, Youpeng Zuo, Cheng Yang, Yongjia Shang**

Key Laboratory of Functional Molecular Solids, Ministry of Education, Anhui Laboratory of Molecule-Based Materials (State Key Laboratory Cultivation Base), College of Chemistry and Materials Science, Anhui Normal University, Wuhu 241000, P.R. China

* Corresponging author: *shyj@mail.ahnu.edu.cn, xinweihe@mail.ahnu.edu.cn*

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Experimental section

General comments

Unless otherwise specified, all reagents and starting materials were purchased from commercial sources and used as received, and the solvents were purified and dried using standard procedures. The chromatographic solvents were technical grade and distilled prior to use. Flash chromatography was performed using 200-300 mesh silica gel with the indicated solvent system according to standard techniques. The ^{1}H and ^{13}C NMR data were recorded on 300 MHz and 500 MHz NMR spectrometers, unless otherwise specified. Chemical shifts (δ) in parts per million are reported relative to the residual signals of chloroform (7.26 ppm for ^{1}H and 77.16 ppm for ^{13}C), and all ^{13}C NMR spectra were recorded with proton broadband decoupling and are indicated as $^{13}C\{^{1}H\}$NMR. Multiplicities are described as s (singlet), d (doublet), t (triplet), q (quartet), or m (multiplet), and the coupling constants (J) are reported in Hertz. HRMS analysis on a quadrupole time-of-flight mass spectrometer yielded ion mass/charge (m/z) ratios in atomic mass units. IR spectra were measured as dry films (KBr), and the peaks are reported in terms of wave number (cm^{-1}).

General procedure for the synthesis of *N*-(*tert*-butyl)-2-hydroxy-6-oxocyclohexanecarboxamide (3a). To a stirred solution of 2-diazocyclohexane-1,3-dione **1a** (0.5 mmol), *tert*-butyl isocyanide **2a** (0.5 mmol) and H_2O (1.5 mmol) in acetone (2 mL), $Rh_2(OAc)_4$ (0.025 mmol) was added. The reaction mixture was heated in an oil bath at 60 °C for 6 h. After the reaction was complete, the mixture was cooled to room temperature, extracted with CH_2Cl_2 (3×10 mL), and washed with water. The organic layers were combined, dried over Na_2SO_4, filtered, and evaporated under vacuum. The residue was further purified by flash column chromatography on silica gel (200-300 mesh) with ethyl acetate and petroleum ether (1:8-1:10, v/v) as the elution solvent to give the desired product **3a** in a yield of 71%.

N-(*tert*-butyl)-2-hydroxy-6-oxocyclohexanecarboxamide (**3a**). Yield 71%, Faint yellow solid; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 18.48 (s, 1H), 10.1 (s, 1H), 2.57 (s, 2H) , 2.46 (s, 2H), 1.94 (s, 2H),1.42 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 197.2, 171.6, 102.7, 52.2, 37.9, 33.4, 29.2, 19.8; IR (KBr) ν 3462, 2360, 2341, 1637, 1624, 1400, 1095, 462 cm^{-1}; HRMS (ESI) calcd for $[C_{11}H_{17}NO_3 + H]^+$ 212.1281, found 212.1284.

N-(*tert*-butyl)-2-hydroxy-4-methyl-6-oxocyclohex-1-enecarboxamide (**3b**). Yield 70%, Faint yellow solid; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 18.43 (s, 1H), 10.02 (s, 1H), 2.44-2.61(m, 2H) , 2.23(m, 1H), 2.11-2.33(m, 3H), 1.41(s, 9H), 1.07(d, J = 6.3 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.5, 196.6, 171.5, 102.3, 52.2, 46.1, 41.3, 29.2, 27.2, 21.2; IR (KBr) ν 3471, 2964, 2358, 2341, 2631, 1417, 1384, 1363, 1338, 1219, 592 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{20}NO_3 + H]^+$ 226.1438, found 226.1439.

N-(*tert*-butyl)-2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enecarboxamide (**3c**). Yield 73%, Faint yellow solid; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 18.41 (s, 1H), 9.98 (s, 1H), 2.43 (s, 2H) , 2.31 (s, 2H), 1.42 (s, 9H), 1.07 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.2, 196.0, 171.4, 101.7, 52.2, 51.6, 46.9, 31.2, 29.2, 28.6; IR (KBr) ν 3184, 2956, 1627, 1558, 1473, 1411, 1338, 1267, 1215, 1112, 1039, 891 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{21}NO_3 + H]^+$ 240.1594, found 240.1599.

N-(*tert*-butyl)-2-hydroxy-4-isopropyl-6-oxocyclohex-1-enecarboxamide (**3d**). Yield 74%, Faint yellow solid; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 18.42 (s, 1H), 9.99 (s, 1H), 2.14-2.61 (m, 4H), 1.80-1.89 (m, 1H), 1.60-1.77 (m, 3H),1.41 (s, 9H), 0.93 (d, J = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.9, 197.0, 171.4, 102.3, 52.2, 41.9, 38.4, 37.2, 32.0, 31.2, 29.2, 19.8;

IR (KBr) *v* 3228, 3095, 2958, 2927, 2378, 2349, 2308, 1637, 1566, 1408, 1361, 1330, 1224, 1083, 783 cm^{-1}; HRMS (ESI) calcd for $[C_{14}H_{23}NO_3 + H]^+$ 254.1751, found 254.1756.

N-(*tert*-butyl)-5-hydroxy-3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-carboxamide (**3e**). Yield 69%, Orange-yellow solid; ^1H NMR ($CDCl_3$, 300MHz) δ 18.52 (s, 1H), 10.08 (s, 1H), 7.36 (d, J = 7.5 Hz, 2H), 7.22-7.27 (m, 3H), 3.29-3.40 (m, 1H), 2.68-2.82 (m, 4H), 1.49 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.3, 195.1, 170.0, 141.1, 127.8, 126.1, 125.5, 100.7, 51.0, 43.6, 39.3, 36.2, 27.7; IR (KBr) *v* 3219, 3028, 2980, 2943, 1618, 1543, 1413, 1365, 1342, 1215, 1147, 1045, 1012, 921 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{21}NO_3 + H]^+$ 288.1594, found 288.1599.

N-(*tert*-butyl)-4'-chloro-5-hydroxy-3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-carboxamide (**3f**). Yield 70%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.49 (s, 1H), 10.06 (s, 1H), 7.43 (d, J = 8.7 Hz, 2H), 7.18 (d, J = 8.4 Hz, 2H), 3.27-3.83 (m, 1H), 2.59-2.79 (m, 4H), 1.42 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.2, 196.1, 174.3, 141.0, 133.3, 129.4, 128.3, 102.2, 52.5, 44.8, 40.6, 37.1, 29.2; IR (KBr) *v* 3454, 2980, 1624, 1550, 1492, 1413, 1367, 1342, 1261, 1217, 1091, 1049, 1014, 823 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{20}ClNO_3 + H]^+$ 322.1204, found 322.1200.

N-butyl-2-hydroxy-6-oxocyclohex-1-enecarboxamide (**3g**). Yield 65%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.31 (s, 1H), 9.89 (s, 1H), 3.34 (q, J = 7.2 Hz, 2H), 2.58 (t, J = 6.3 Hz, 2H), 2.46 (t, J = 6.9 Hz, 2H), 1.92-2.00 (m, 3H), 1.96 (d, J = 9.0 Hz, 2H), 1.34-1.41 (m, 3H), 1.24 (s, 2H), 0.92 (t, J = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 196.5, 171.7, 102.7, 39.1, 37.8, 32.9, 31.5, 30.0, 20.4, 19.8, 14.0; IR (KBr) *v* 3460, 2958, 2931, 2360, 2341, 1629, 1566, 1384, 1101, 1049 cm^{-1}; HRMS (ESI) calcd for $[C_{11}H_{17}NO_3 + H]^+$ 212.1281, found 212.1282.

N-butyl-2-hydroxy-4-methyl-6-oxocyclohex-1-enecarboxamide (**3h**). Yield 68%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.25 (s, 1H), 9.86 (s, 1H), 3.34 (q, J = 6.3 Hz, 2H), 2.48-2.63 (m, 2H), 2.15-2.34 (m, 3H), 1.51-1.60 (m, 2H), 1.34-1.40 (m, 2H), 1.06 (d, J = 5.4 Hz, 3H), 0.93 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.2, 195.5, 171.2, 101.8, 45.6, 40.5, 38.6, 31.1, 26.9, 20.8, 20.0, 13.6; IR (KBr) *v* 3462, 3238, 2960, 2929, 2872, 2374, 2345, 1624, 1570, 1261, 1095, 802 cm^{-1}; HRMS (ESI) calcd for $[C_{12}H_{19}NO_3 + H]^+$ 226.1438, found 226.1434.

N-butyl-2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enecarboxamide (**3i**). Yield 70%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.22 (s, 1H), 9.82 (s, 1H), 3.37 (q, J = 6.6 Hz, 2H), 2.45 (s, 2H), 2.32 (s, 2H), 1.51-1.61 (m, 3H), 1.34-1.41 (m, 3H), 1.07 (s, 6H), 0.93 (t, J = 7.2 Hz, 3H), 0.91 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.3, 195.2, 171.5, 101.6, 51.6, 46.5, 39.1, 31.6, 31.3, 30.0, 28.6, 20.5, 14.0; IR (KBr) *v* 3487, 2962, 2360, 2331, 1639, 1384, 1261, 1095, 1022, 802 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{21}NO_3 + H]^+$ 240.1594, found 240.1598.

N-butyl-2-hydroxy-4-isopropyl-6-oxocyclohex-1-enecarboxamide (**3j**). Yield 69%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.23 (s, 1H), 9.83 (s, 1H), 3.37 (q, J = 7.2 Hz, 2H), 2.48-2.62 (m, 2H), 2.15-2.40 (m, 2H), 1.80-1.92 (m, 2H), 1.30-1.41 (m, 2H), 1.25 (m, 2H), 1.24-1.22 (m, 1H), 0.94 (t, J = 6.9 Hz, 3H), 0.86 (t, J = 6.9 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.0, 196.6, 170.6, 102.1, 48.3, 41.9, 38.5, 36.9, 32.9, 32.1, 31.3, 25.8, 24.8, 19.8; IR (KBr) *v* 3238, 2958, 2927, 2872, 2378, 2308, 1624, 1566, 1535, 1261, 1153, 935 cm^{-1}; HRMS (ESI) calcd for $[C_{14}H_{23}NO_3 + H]^+$ 254.1751, found 254.1756.

N-butyl-5-hydroxy-3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-carboxamide (**3k**). Yield 64%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.35 (s, 1H), 9.91 (s, 1H), 7.33-7.38 (m, 2H), 7.21-7.29 (m, 3H), 3.37 (q, J = 6.3 Hz, 2H), 2.84 (d, J = 8.1 Hz, 2H), 2.69-2.72 (m, 2H), 1.54-1.64 (m, 3H), 1.34-1.43 (m, 2H), 1.25-1.20 (m, 2H), 0.94 (t, J = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.3, 195.4, 171.1, 142.0, 128.8, 127.2, 126.5, 101.8, 44.6, 49.9, 38.8, 37.3, 31.1, 20.0,

13.6; IR (KBr) *v* 3502, 2960, 2308, 1631, 1573, 1415, 1099, 798 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{21}NO_3 + H]^+$ 288.1594, found 288.1598.

N-butyl-4'-chloro-5-hydroxy-3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-carboxamide (**3l**). Yield 62%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.34 (s, 1H), 9.90 (s, 1H), 7.29 (d, J = 8.1 Hz, 2H), 7.16 (d, J = 7.8 Hz, 2H), 3.31-3.39 (m, 3H), 2.76-2.82 (m, 2H), 2.64-2.73 (m, 2H), 1.52-1.62 (m, 2H), 1.35-1.44 (m, 2H), 0.93 (t, J = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.8, 195.1, 171.1, 140.5, 132.9, 129.0, 127.9, 101.7, 44.4, 39.8, 38.8, 36.7, 31.1, 20.0, 13.6; IR (KBr) *v* 3454, 2956, 2924, 2852, 2358, 2331, 1639, 1093, 796 cm^{-1}; HRMS (ESI) calcd for $[C_{17}H_{20}ClNO_3 + H]^+$ 322.1204, found 322.1209.

N-cyclohexyl-2-hydroxy-6-oxocyclohex-1-enecarboxamide (**3m**). Yield 70%, Faint yellow solid; 1H NMR ($CDCl_3$, 300 MHz) δ 18.35 (s, 1H), 9.93 (s, 1H), 3.78-3.88 (m, 1H), 2.58 (t, J = 6.3 Hz, 2H), 2.46 (t, J = 6.3 Hz, 2H), 1.88-1.99 (m, 4H), 1.70-1.75 (m, 2H), 1.21-1.44 (m, 7H), 1.33-1.25 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 196.8, 170.7, 102.5, 48.3, 37.9, 33.1, 32.8, 25.8, 24.8, 19.8; IR (KBr) *v* 3473, 2933, 2856, 2360, 2331, 2007, 1631, 1564, 1452, 1415, 1093, 785 cm^{-1}; HRMS (ESI) calcd for $[C_{13}H_{19}NO_3 + H]^+$ 238.1438, found 238.1439.

N-cyclohexyl-2-hydroxy-4-methyl-6-oxocyclohex-1-enecarboxamide (**3n**). Yield 74%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.29 (s, 1H), 9.88 (s, 1H), 3.82-3.86 (m, 1H), 2.44-2.62 (m, 2H), 2.11-2.33 (m, 4H), 1.87-1.91 (m, 3H), 1.64-1.72 (m, 2H), 1.18-1.43 (m, 4H), 1.07 (d, J = 5.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 196.2, 170.6, 102.1, 48.3, 46.1, 41.0, 32.8, 31.2, 27.3, 25.8, 24.8, 21.2; IR (KBr) *v* 3471, 2933, 2856, 2360, 2331, 1631, 1564, 1452, 1421, 1382, 1261, 1083 cm^{-1}; HRMS (ESI) calcd for $[C_{14}H_{21}NO_3 + H]^+$ 252.1594, found 252.1598.

N-cyclohexyl-2-hydroxy-4,4-dimethyl-6-oxocyclohex-1-enecarboxamide (**3o**). Yield 81%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.25 (s, 1H), 9.85 (s, 1H), 3.79-3.87 (m, 1H), 2.44 (s, 2H), 2.32 (s, 2H), 1.89-1.92 (m, 3H), 1.70-1.75 (m, 4H), 1.29-1.41 (s, 4H), 1.07 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.3, 195.5, 170.5, 101.5, 51.6, 48.3, 46.6, 32.9, 31.3, 30.0, 28.6, 25.8, 24.8; IR (KBr) *v* 3452, 2939, 2854, 2358, 2341, 1637, 1566, 1452, 1423, 1384, 1340, 1093 cm^{-1}; HRMS (ESI) calcd for $[C_{15}H_{23}NO_3 + H]^+$ 266.1751, found 166.1755.

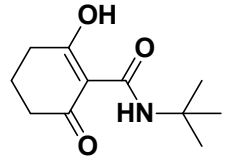
N-cyclohexyl-2-hydroxy-4-isopropyl-6-oxocyclohex-1-enecarboxamide (**3p**). Yield 76%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.27 (s, 1H), 9.86 (s, 1H), 3.78-3.87 (m, 1H), 2.47-2.60 (m, 2H), 2.14-2.39 (m, 2H), 1.87-1.91 (m, 3H), 1.70-1.73 (m, 2H), 1.29-1.44 (m, 7H), 0.93 (d, J = 6.6 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.0, 196.6, 170.5, 102.1, 48.3, 41.9, 38.5, 36.9, 32.9, 32.0, 31.3, 25.8, 24.8, 19.8, 19.7; IR (KBr) *v* 3481, 2939, 2854, 2358, 2341, 1629, 1566, 1423, 1340, 1093, 592 cm^{-1}; HRMS (ESI) calcd for $[C_{16}H_{25}NO_3 + H]^+$ 280.1907, found 280.1909.

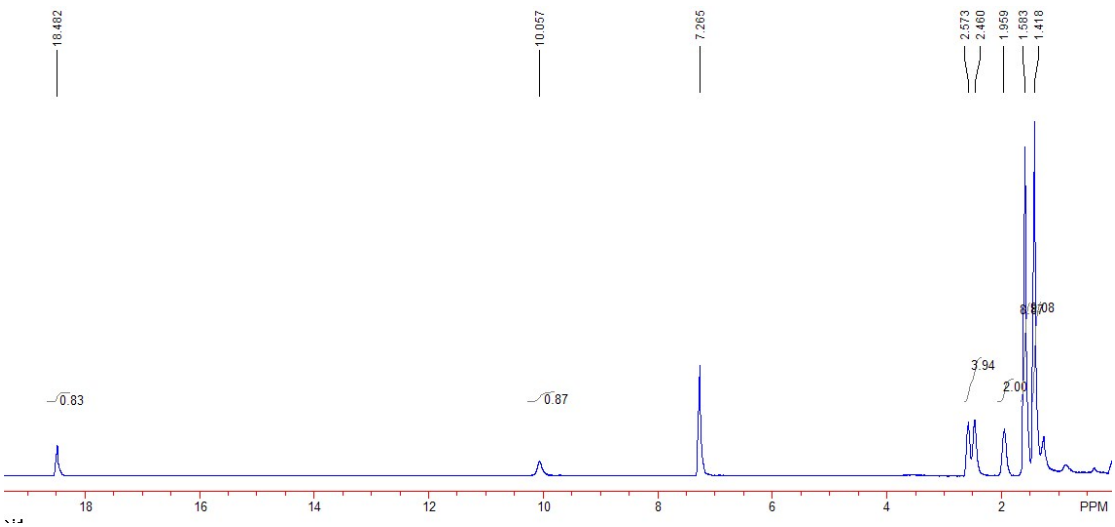
N-cyclohexyl-5-hydroxy-3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-carboxamide (**3q**). Yield 72%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.38 (s, 1H), 9.95 (s, 1H), 7.21-7.38 (m, 5H), 3.83-3.91 (m, 1H), 3.30-3.41 (m, 1H), 2.83 (d, J =8.1 Hz, 2H), 2.68-2.72 (m, 2H), 1.90-1.92 (m, 2H), 1.72-1.76 (m, 2H), 1.31-1.42 (m, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.7, 196.0, 170.6, 142.5, 129.3, 129.0, 127.6, 127.1 126.9, 102.0, 48.4, 45.0, 40.4, 37.7, 32.8, 31.3, 25.8, 24.8; IR (KBr) *v* 3215, 3030, 2929, 2854, 1633, 1562, 1446, 1332, 1257, 1145, 1083, 1010, 912, 763 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{23}NO_3 + H]^+$ 314.1751, found 314.1756.

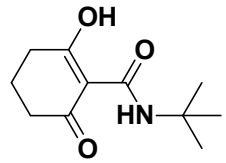
4'-Chloro-*N*-cyclohexyl-5-hydroxy-3-oxo-1,2,3,6-tetrahydro-[1,1'-biphenyl]-4-carboxamide (**3r**). Yield 71%, Faint yellow solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 18.38 (s, 1H), 9.92 (s, 1H), 7.14-7.33 (m, 4H), 3.82-3.89 (m, 1H), 3.28-3.39 (m, 1H), 2.76-2.79 (m, 2H), 2.64-2.69 (m, 2H), 1.90-1.93 (m, 2H), 1.72-1.78 (m, 2H), 1.24-1.41 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.2,

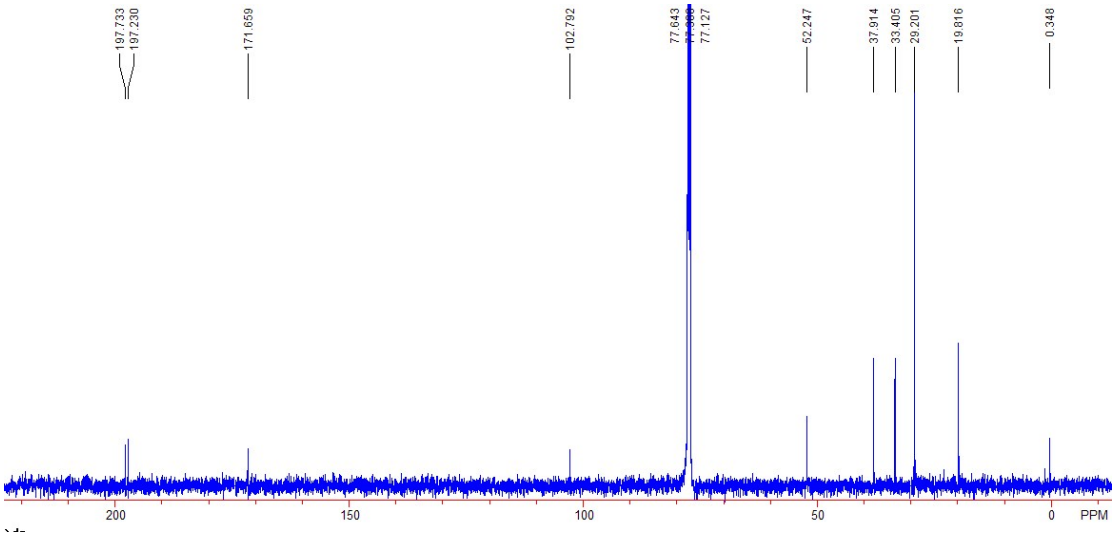
195.8, 170.5, 140.9, 133.3, 129.4, 129.1, 128.4 128.3, 101.9, 48.5, 46.0, 44.8, 40.4, 37.2, 32.8, 31.5, 30.0, 25.7, 24.7; IR (KBr) *v* 3446, 2924, 2852, 2360, 2341, 1629, 1384, 1338, 1259, 1093, 1014, 829 cm^{-1}; HRMS (ESI) calcd for $[C_{19}H_{22}ClNO_3 + H]^+$ 348.1361, found 348.1366.

^{1}H NMR and ^{13}C NMR Spectra of Compound 3a

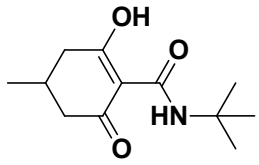


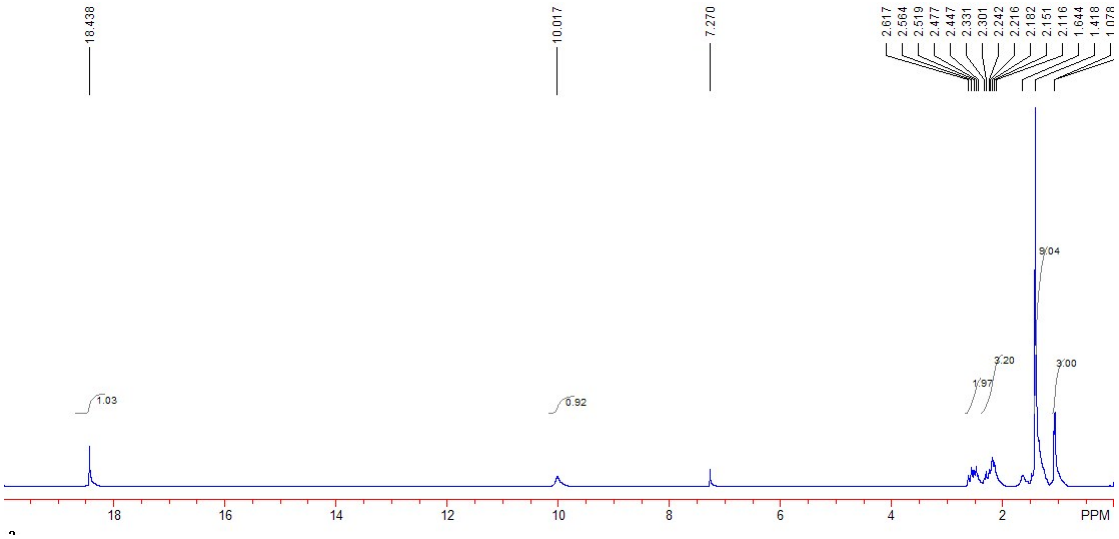


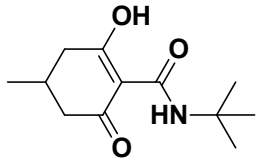


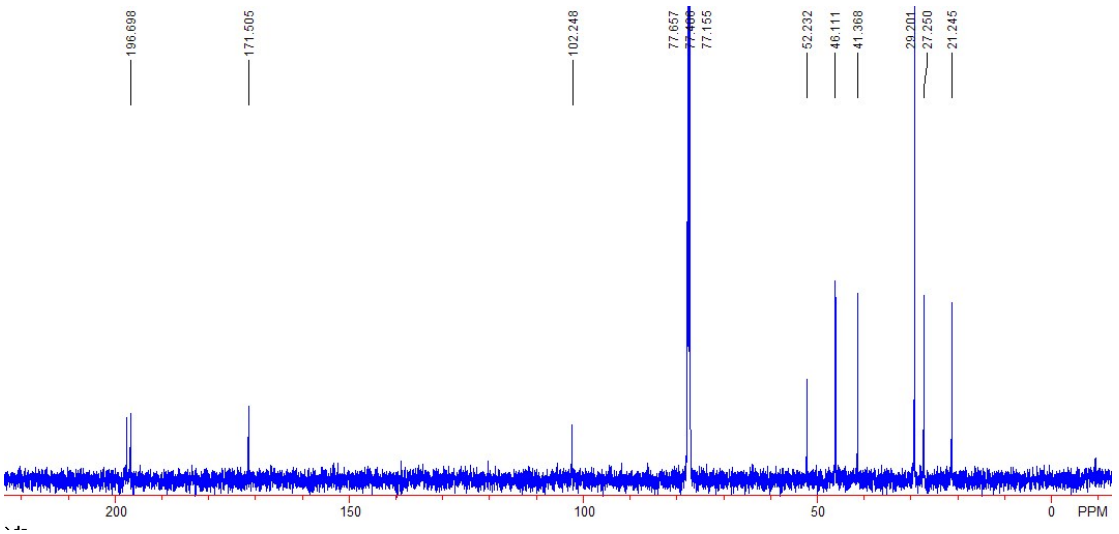


^{1}H NMR and ^{13}C NMR Spectra of Compound 3b

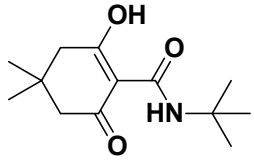


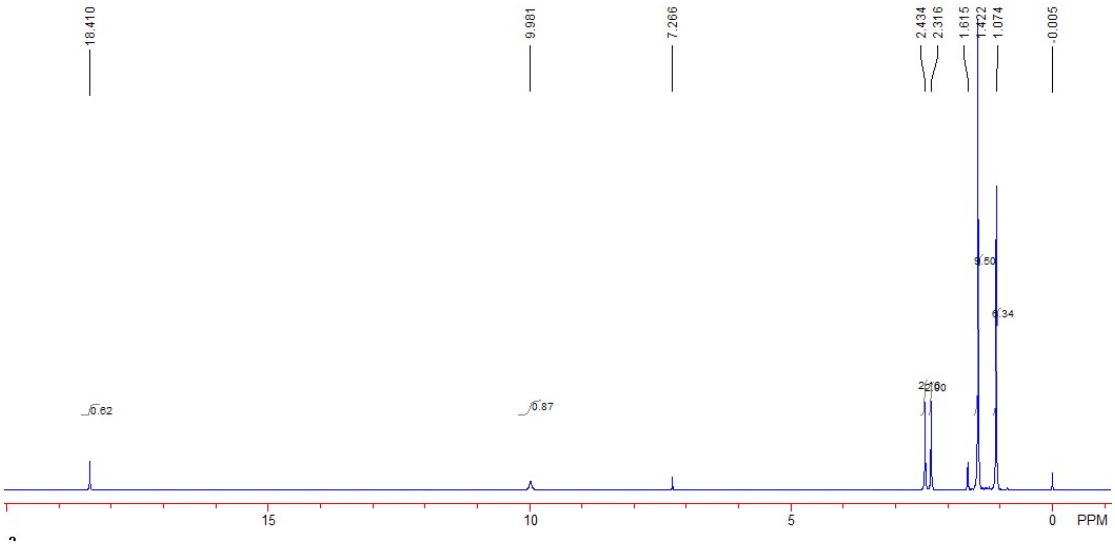


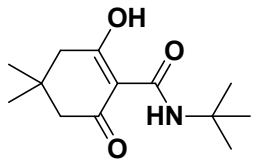


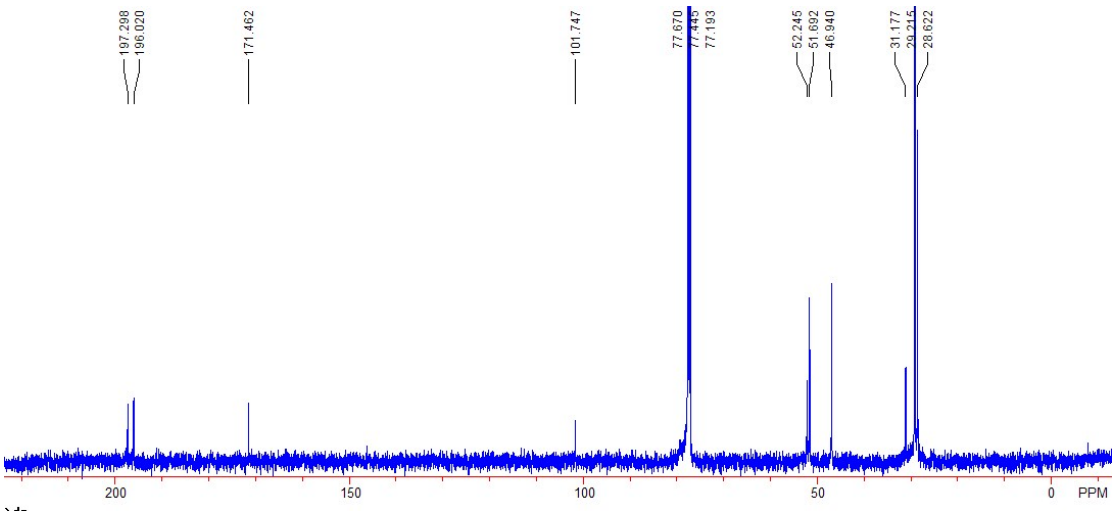


^{1}H NMR and ^{13}C NMR Spectra of Compound 3c

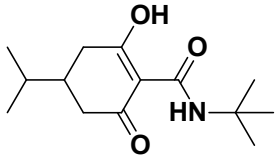


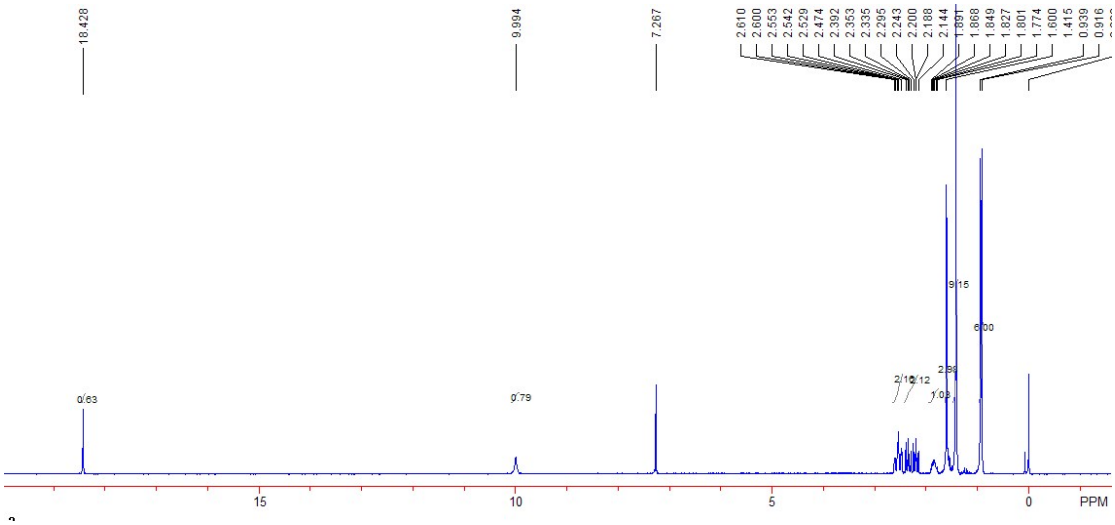


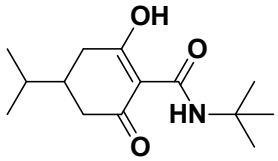


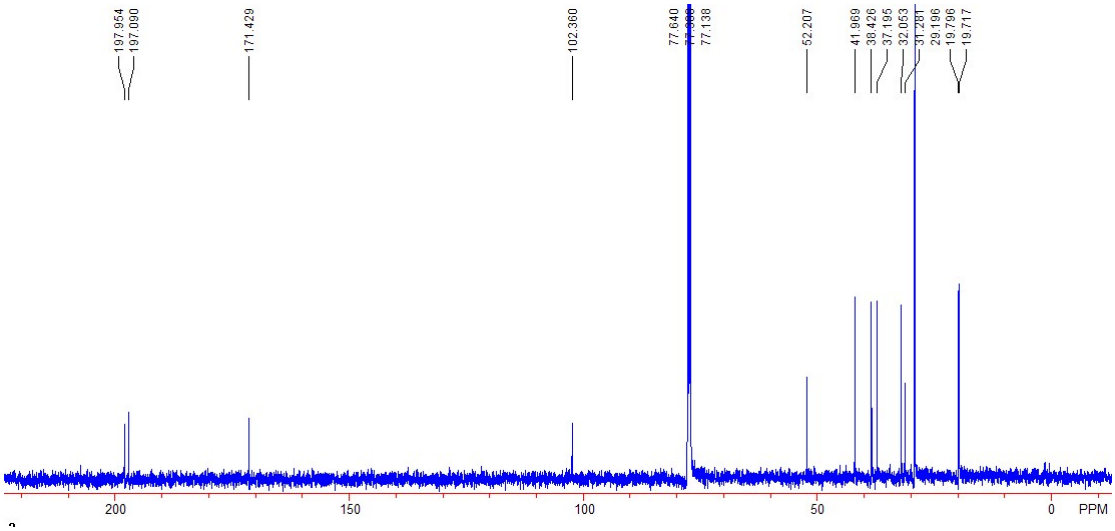


^{1}H NMR and ^{13}C NMR Spectra of Compound 3d

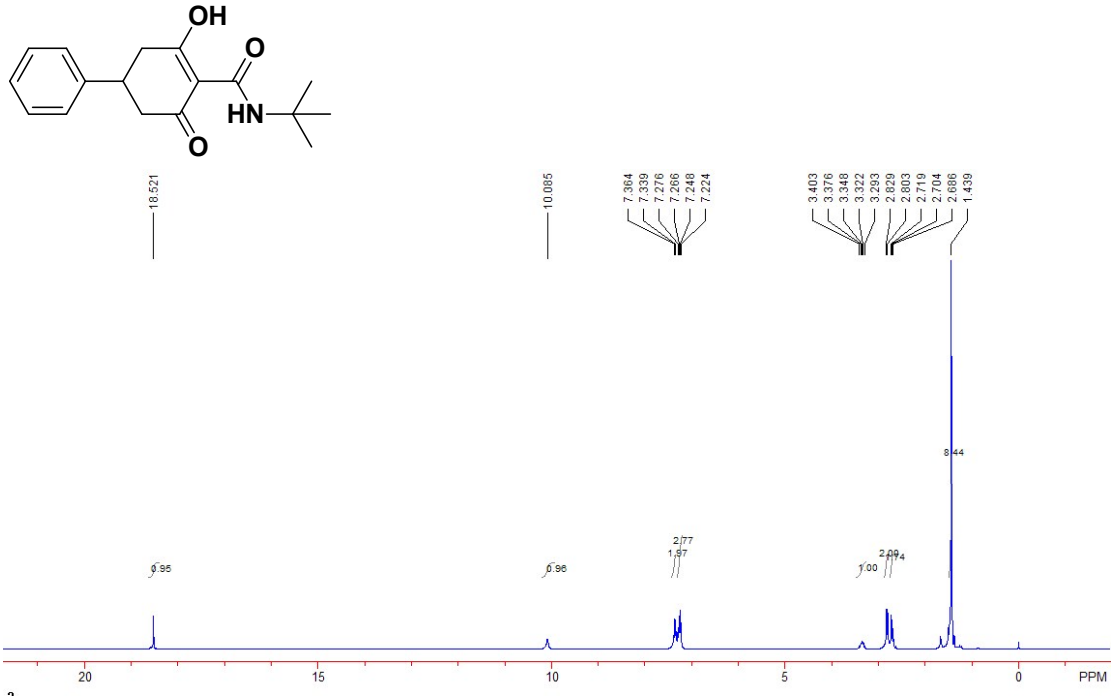


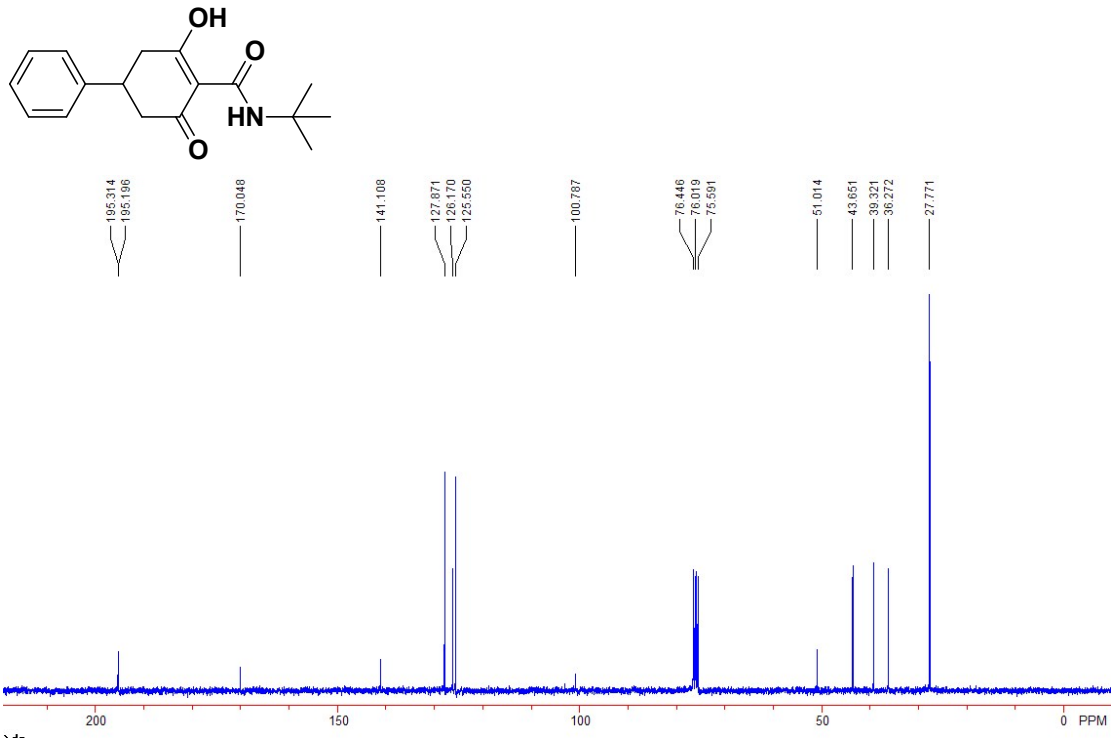




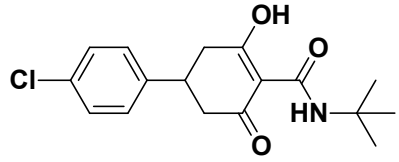


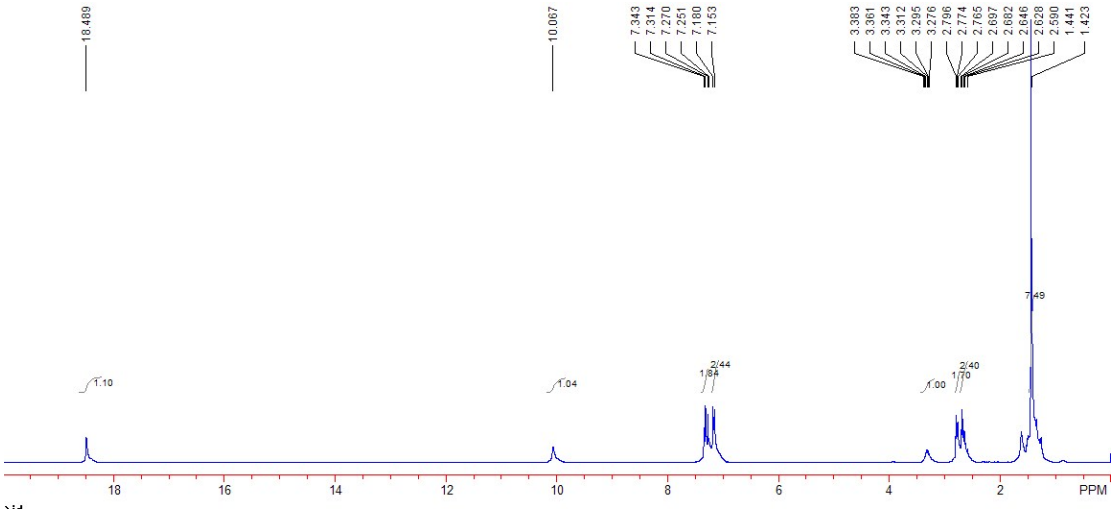
^{1}H NMR and ^{13}C NMR Spectra of Compound 3e

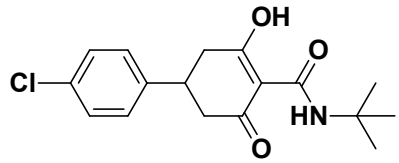


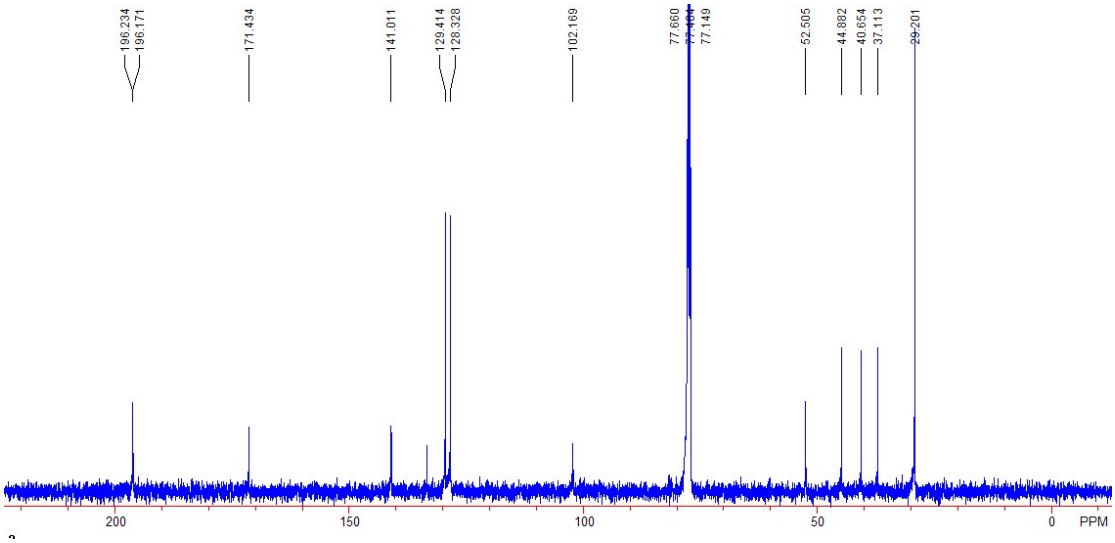


^{1}H NMR and ^{13}C NMR Spectra of Compound 3f

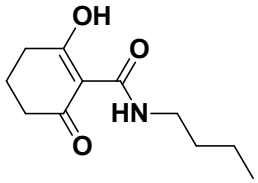


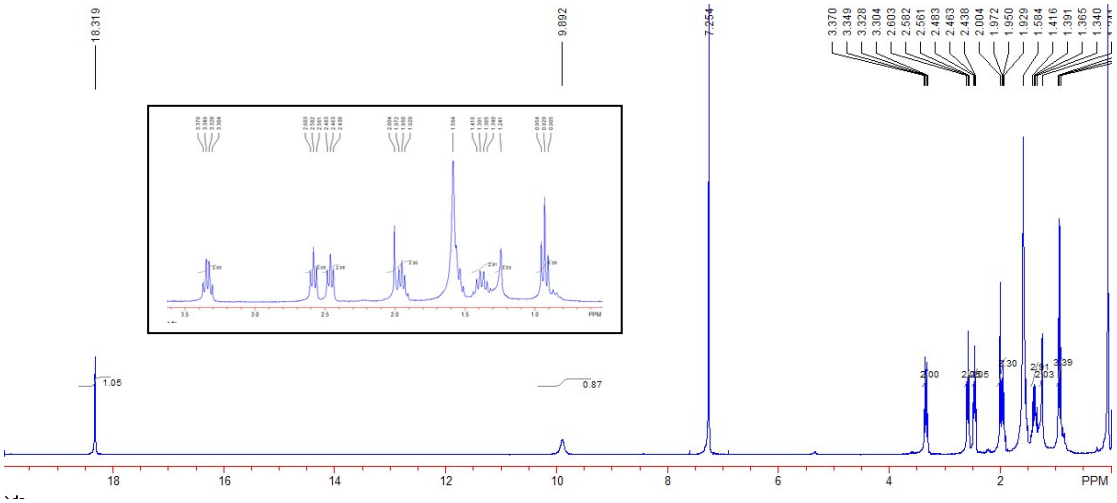


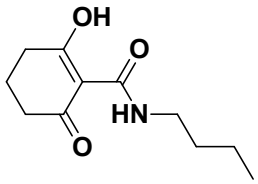


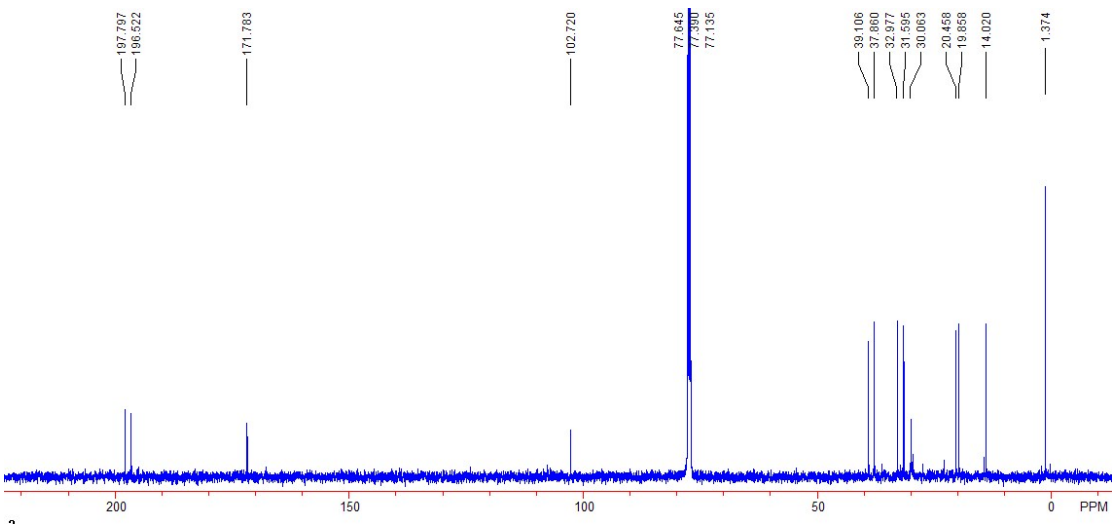


^{1}H NMR and ^{13}C NMR Spectra of Compound 3g

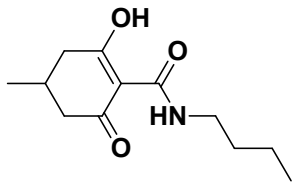


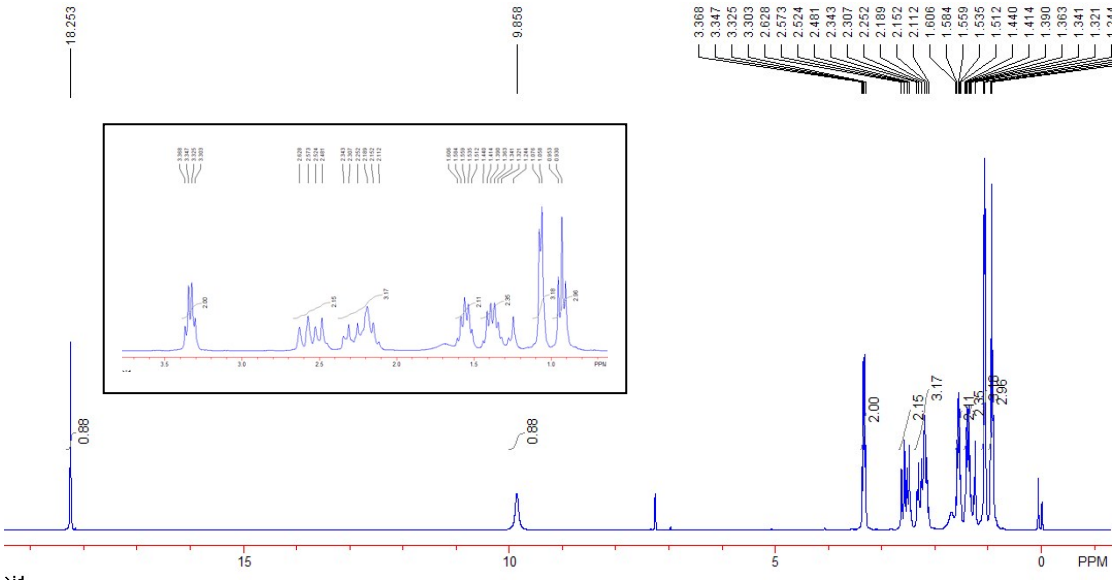


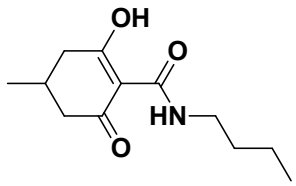


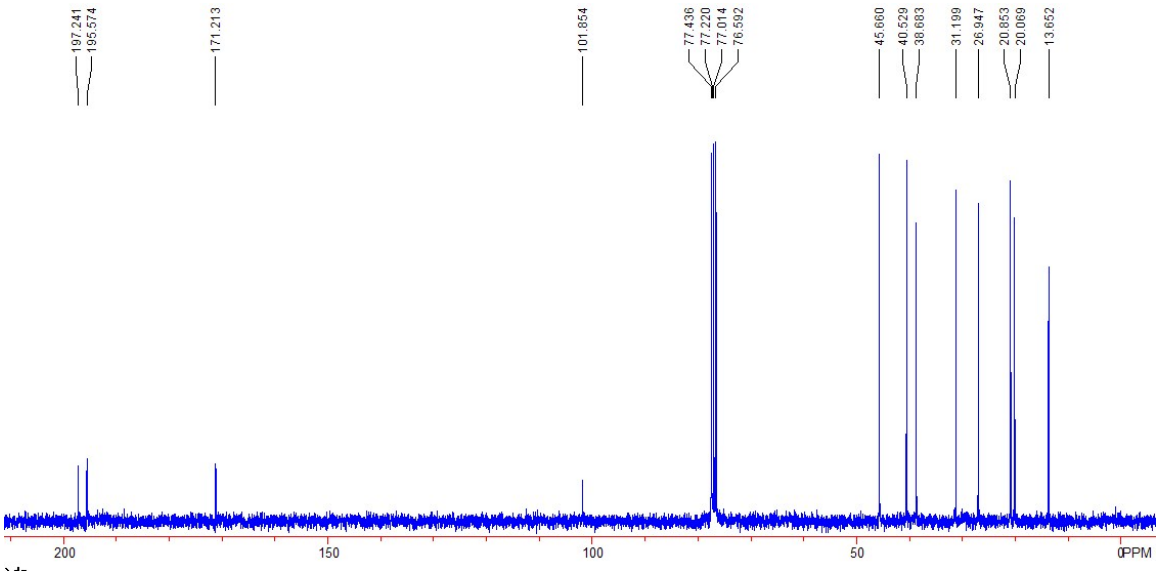


^{1}H NMR and ^{13}C NMR Spectra of Compound 3h

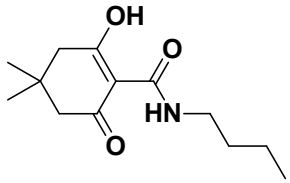


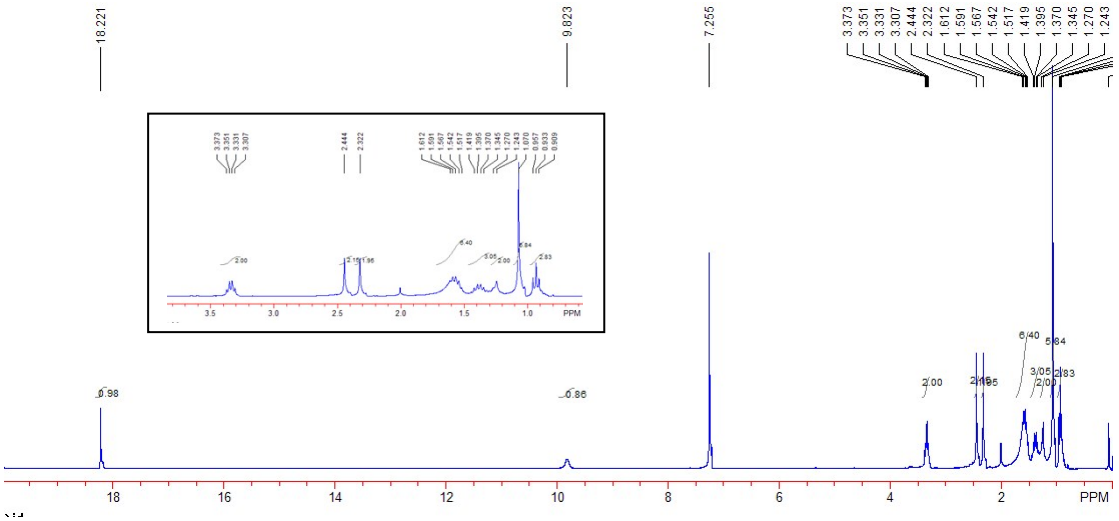


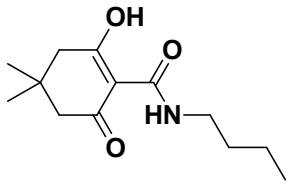


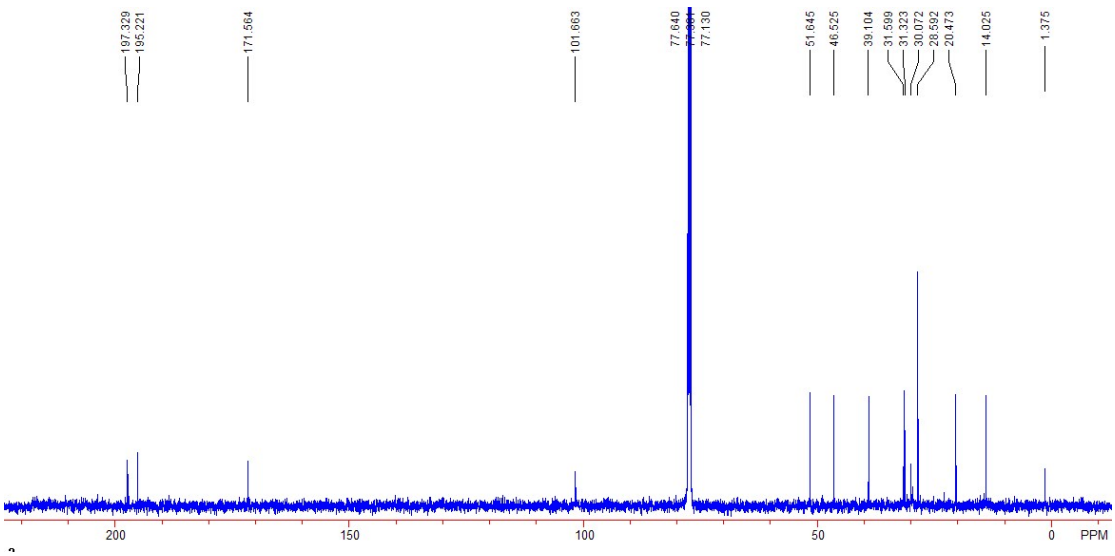


^{1}H NMR and ^{13}C NMR Spectra of Compound 3i

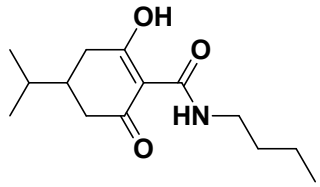


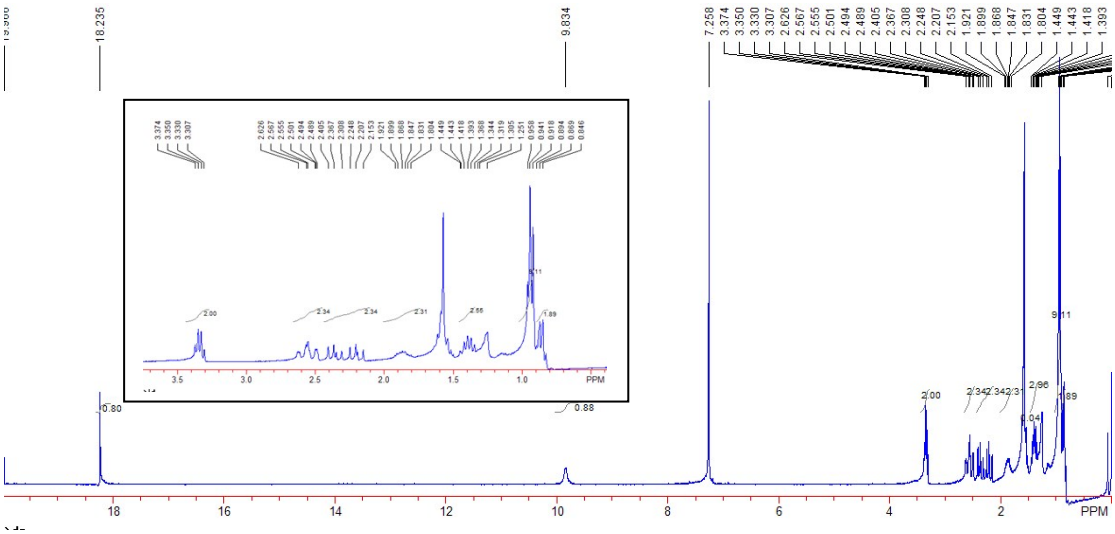


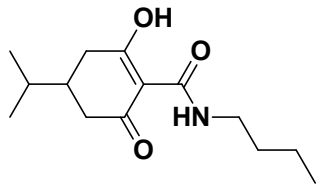


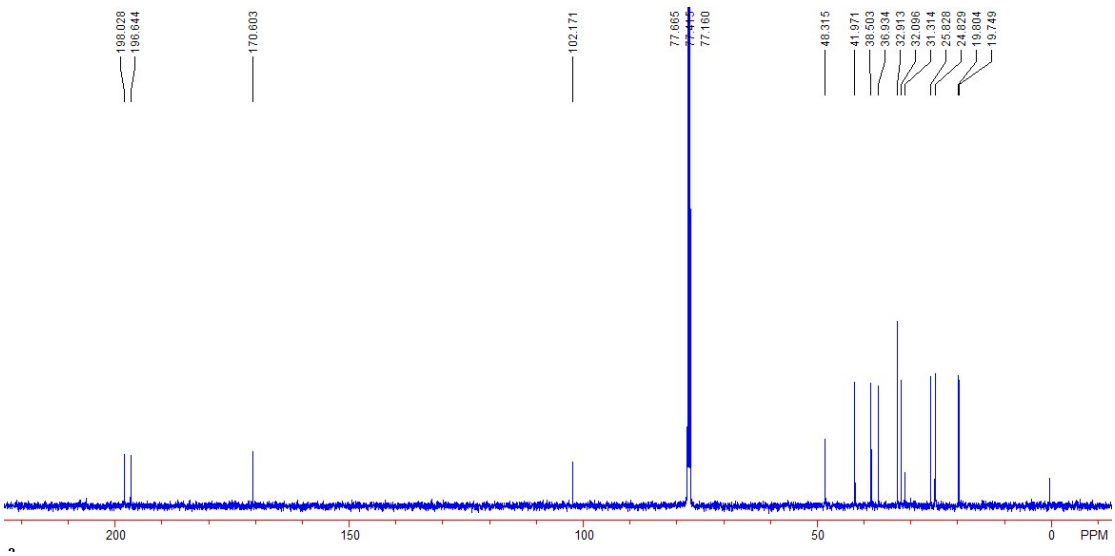


^{1}H NMR and ^{13}C NMR Spectra of Compound 3j

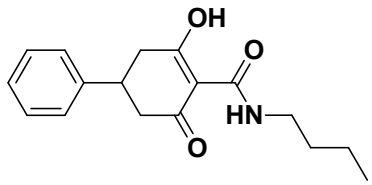


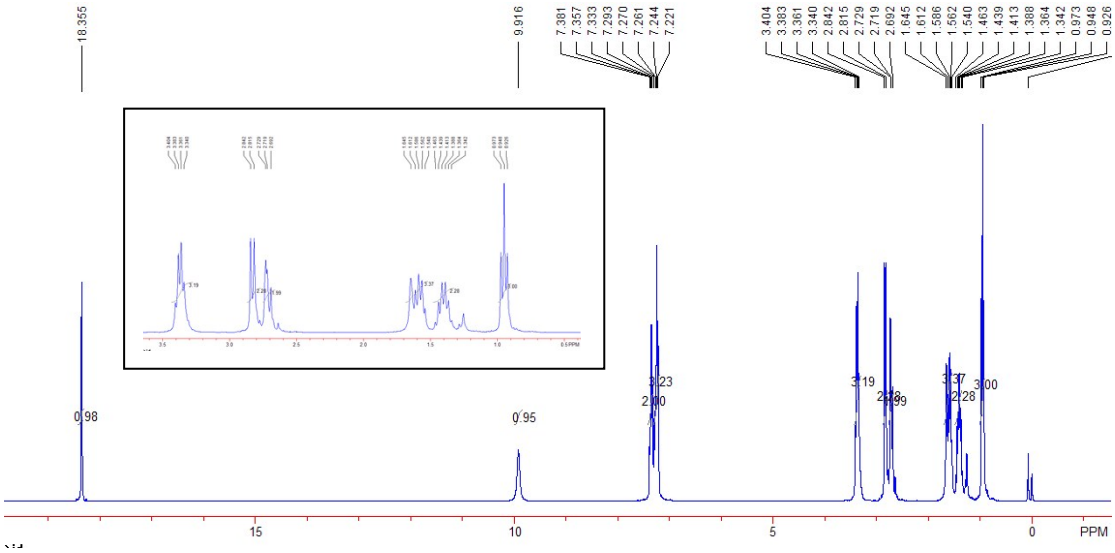


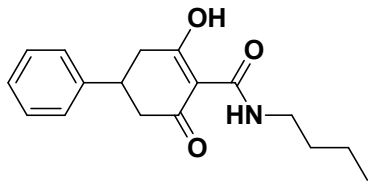


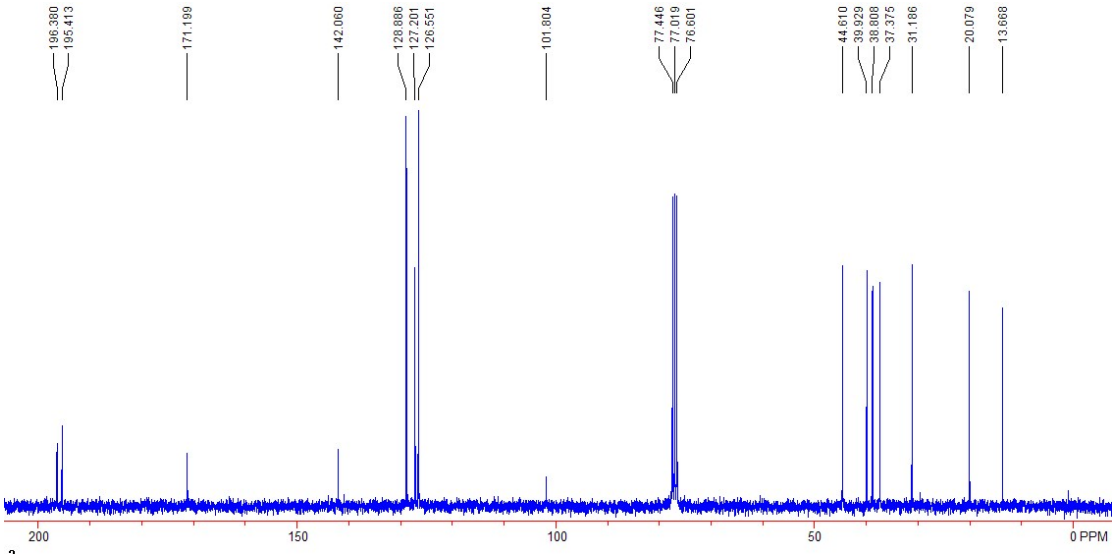


^{1}H NMR and ^{13}C NMR Spectra of Compound 3k

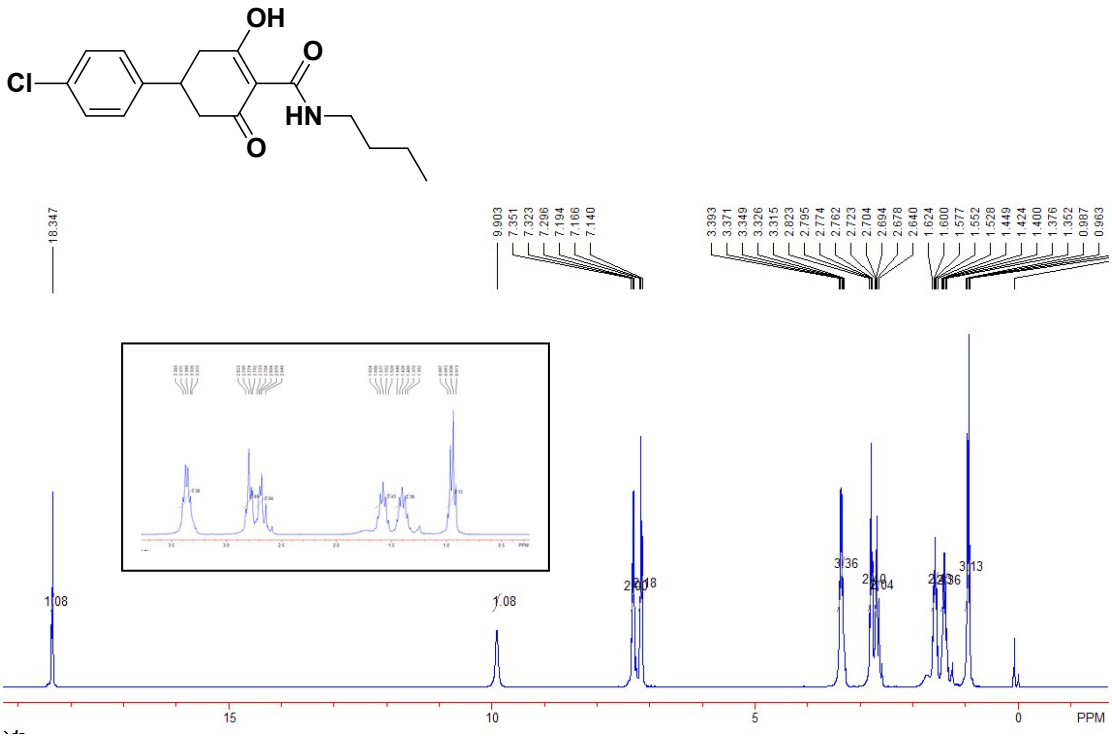


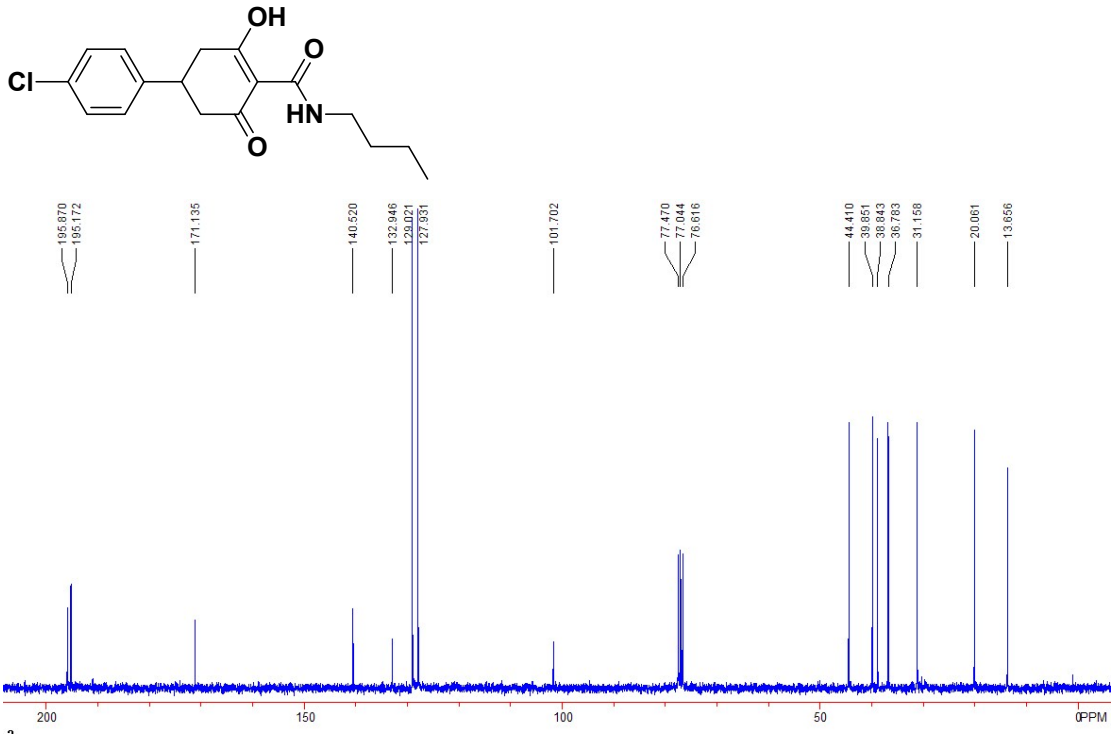




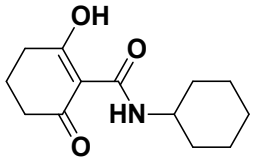


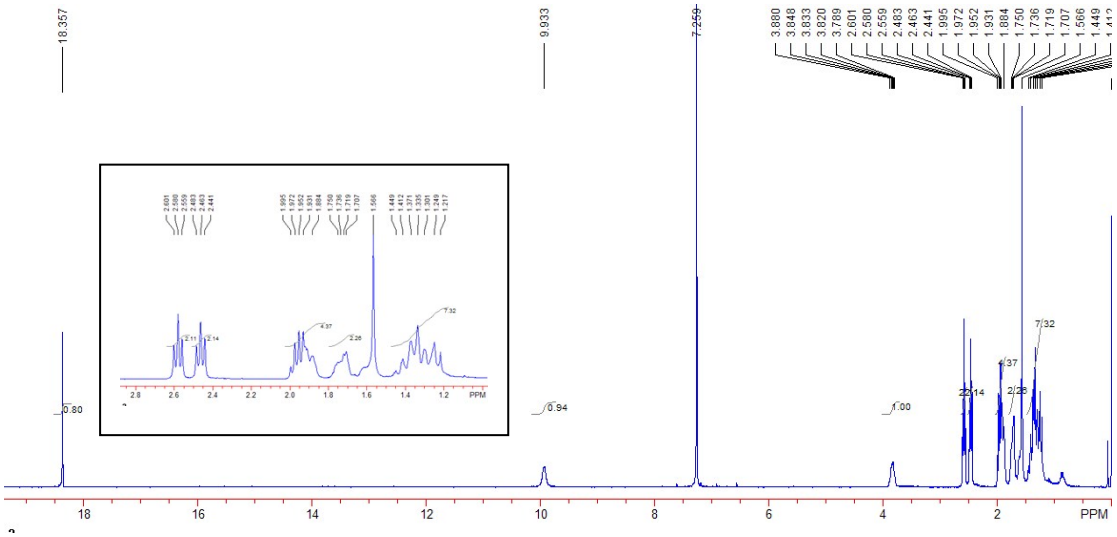
^{1}H NMR and ^{13}C NMR Spectra of Compound 3l

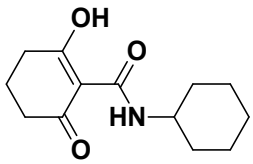


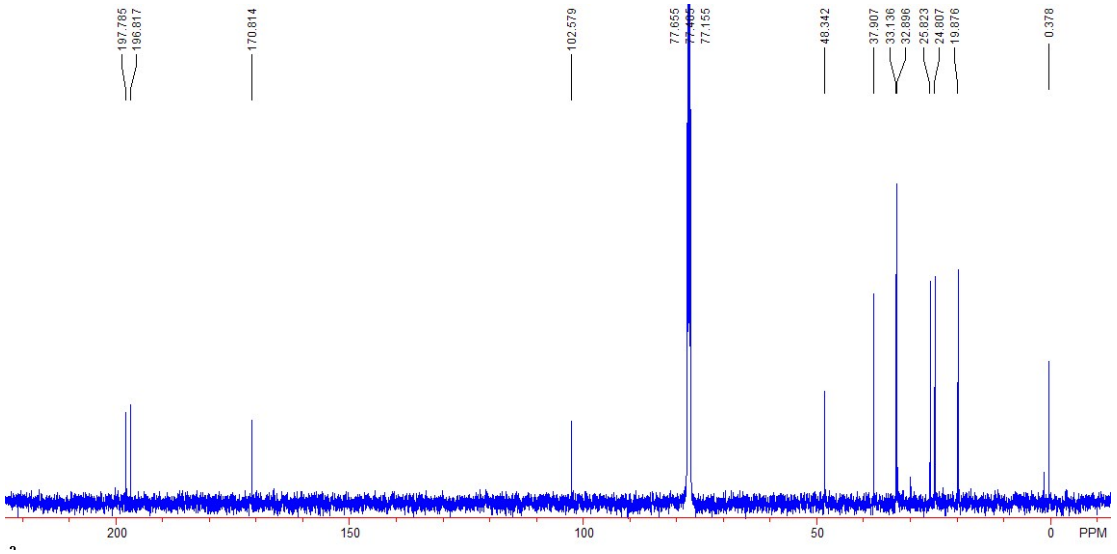


^{1}H NMR and ^{13}C NMR Spectra of Compound 3m

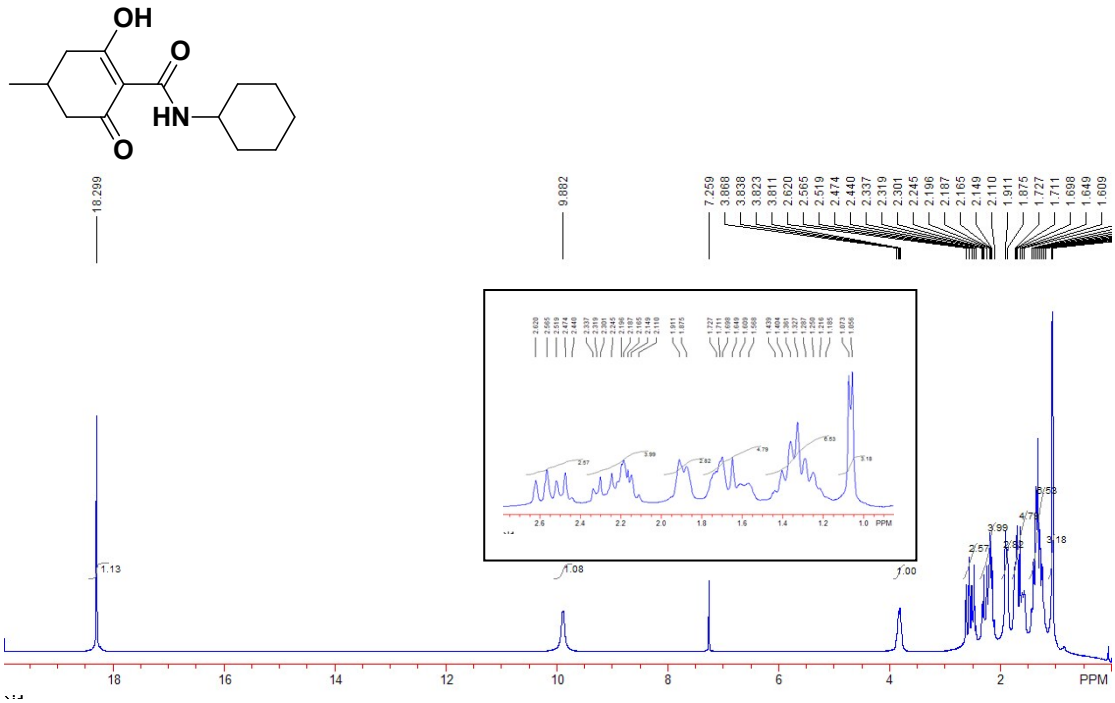


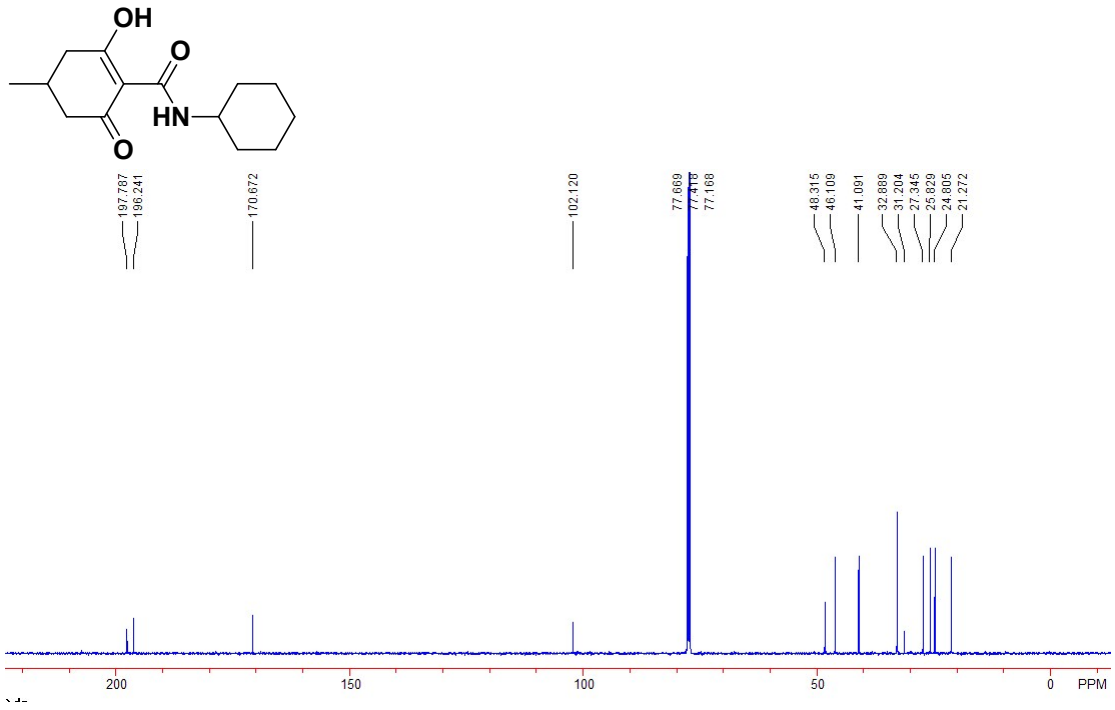




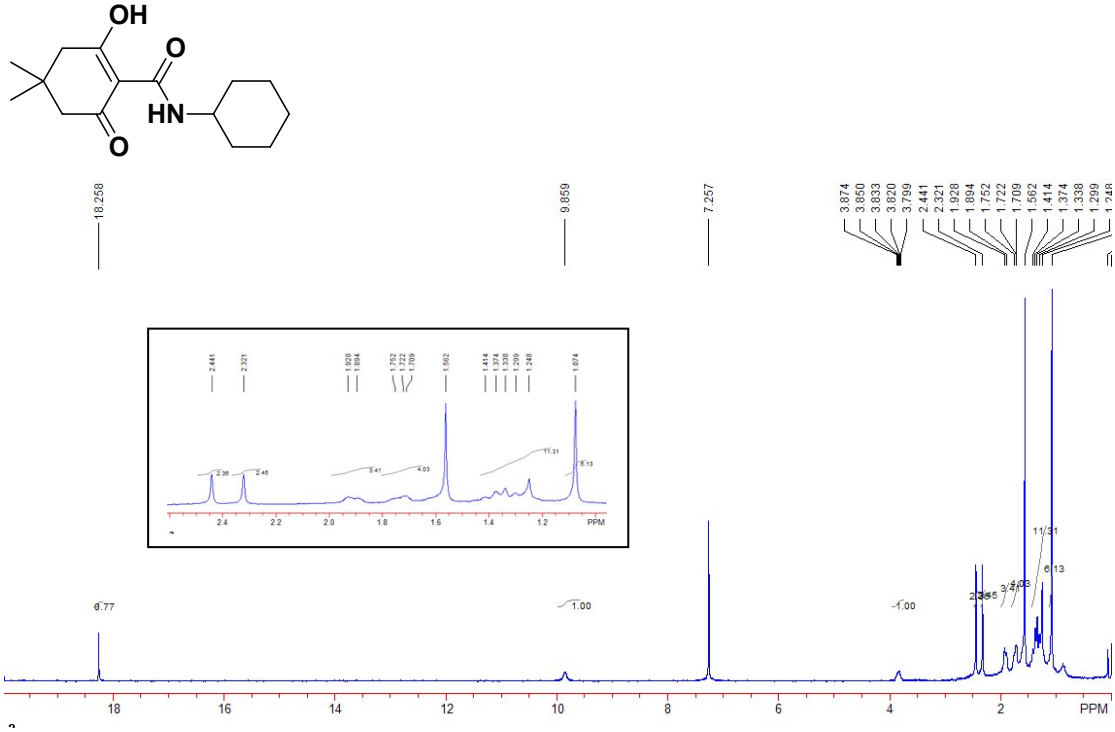


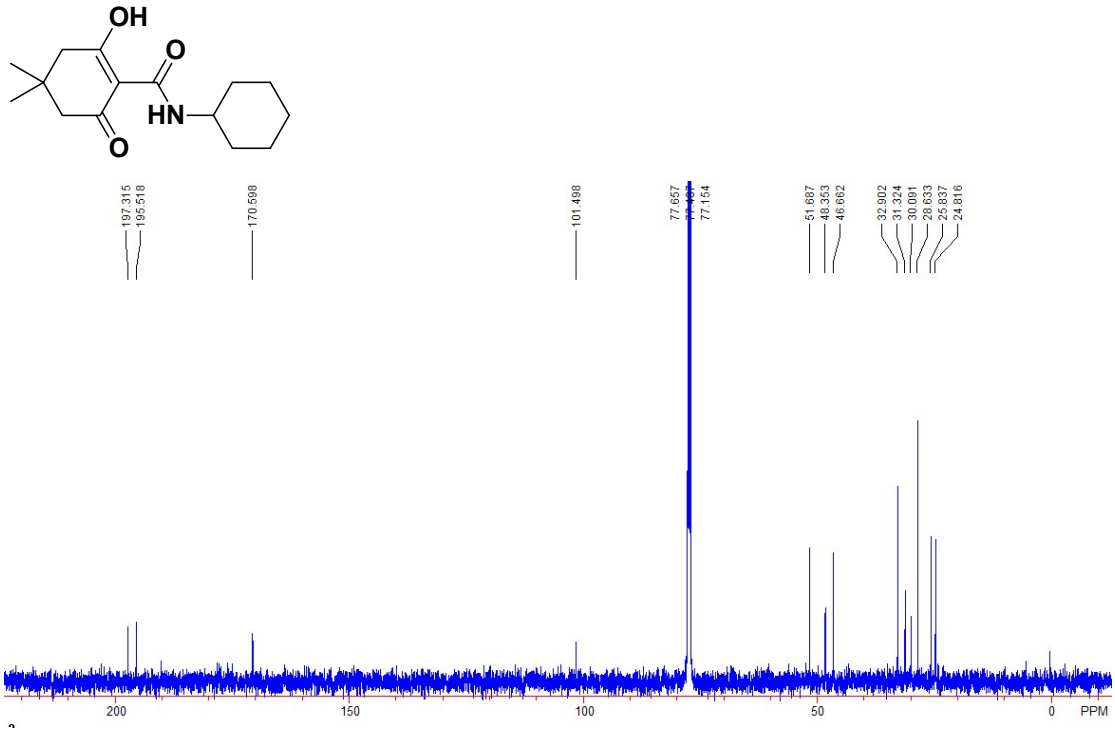
^{1}H NMR and ^{13}C NMR Spectra of Compound 3n



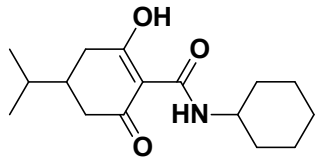


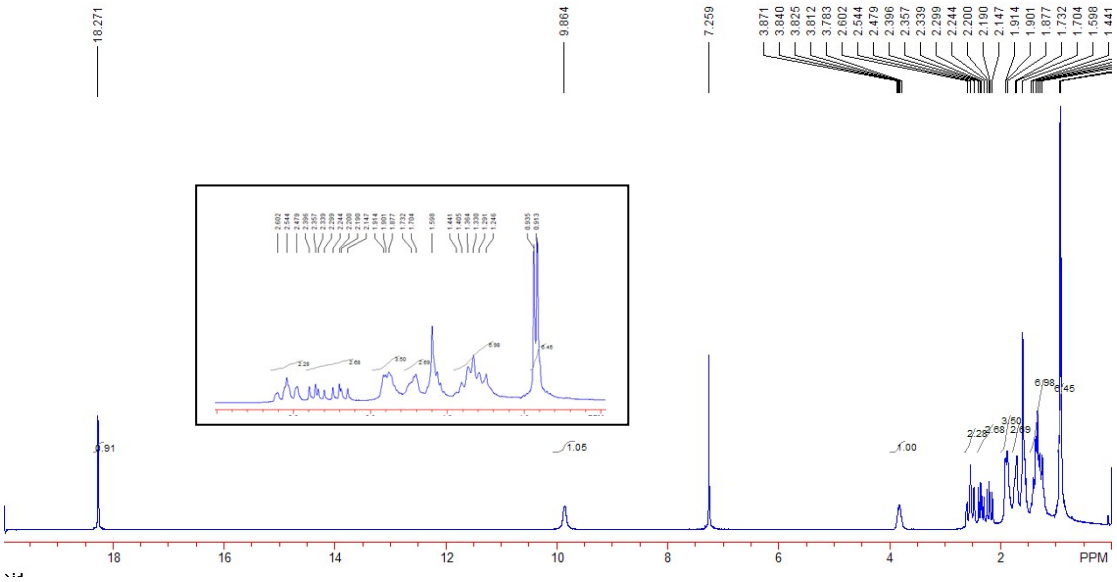
^{1}H NMR and ^{13}C NMR Spectra of Compound 3o

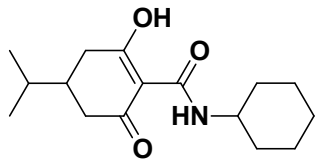


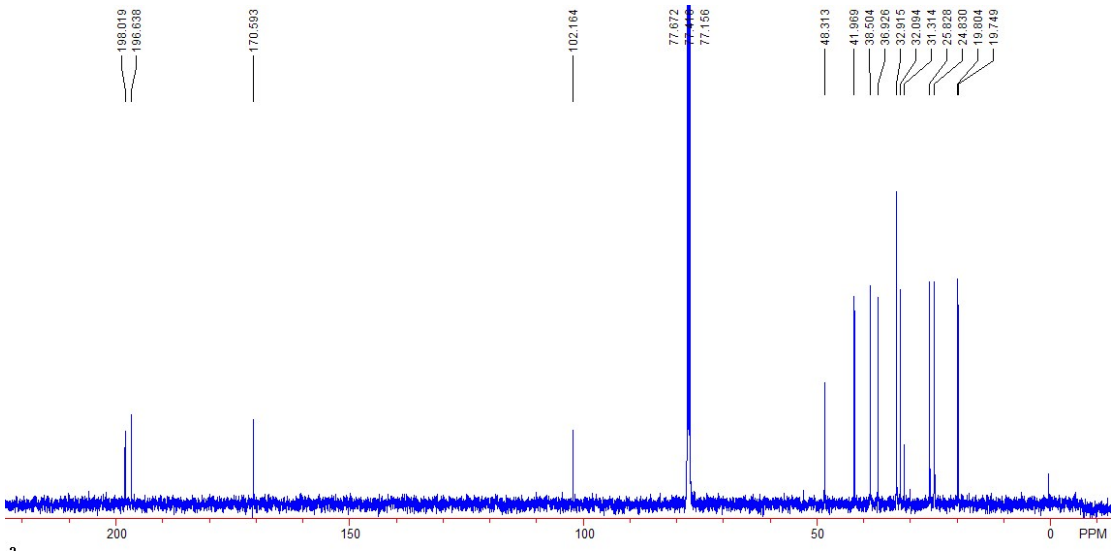


^{1}H NMR and ^{13}C NMR Spectra of Compound 3p

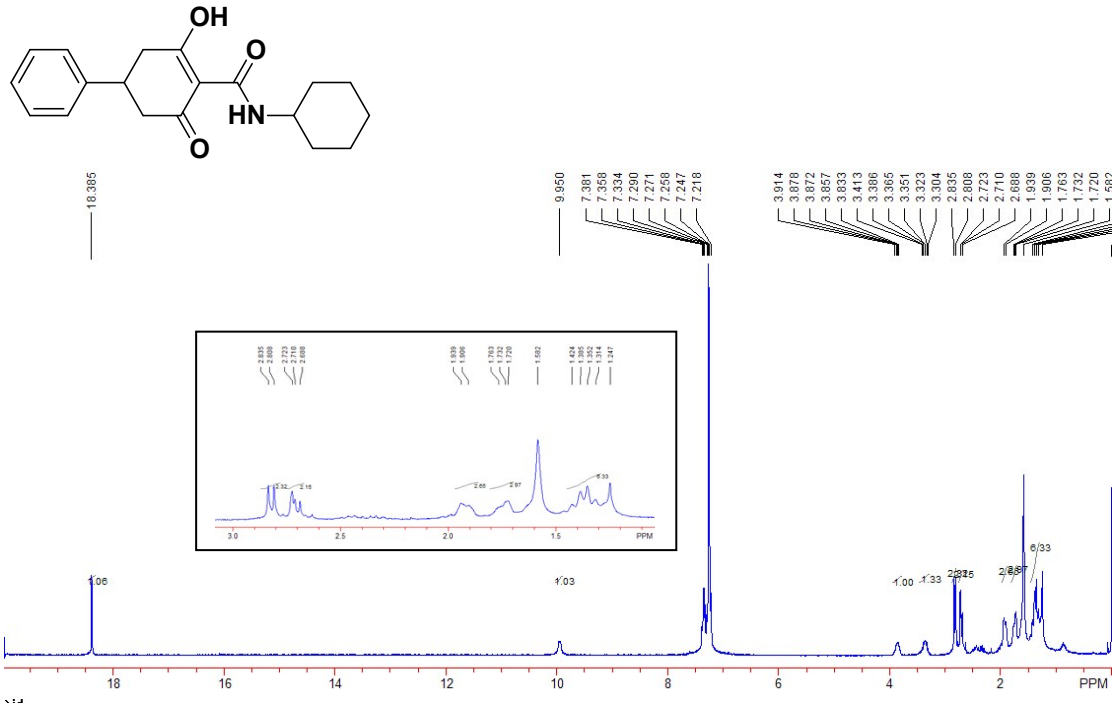


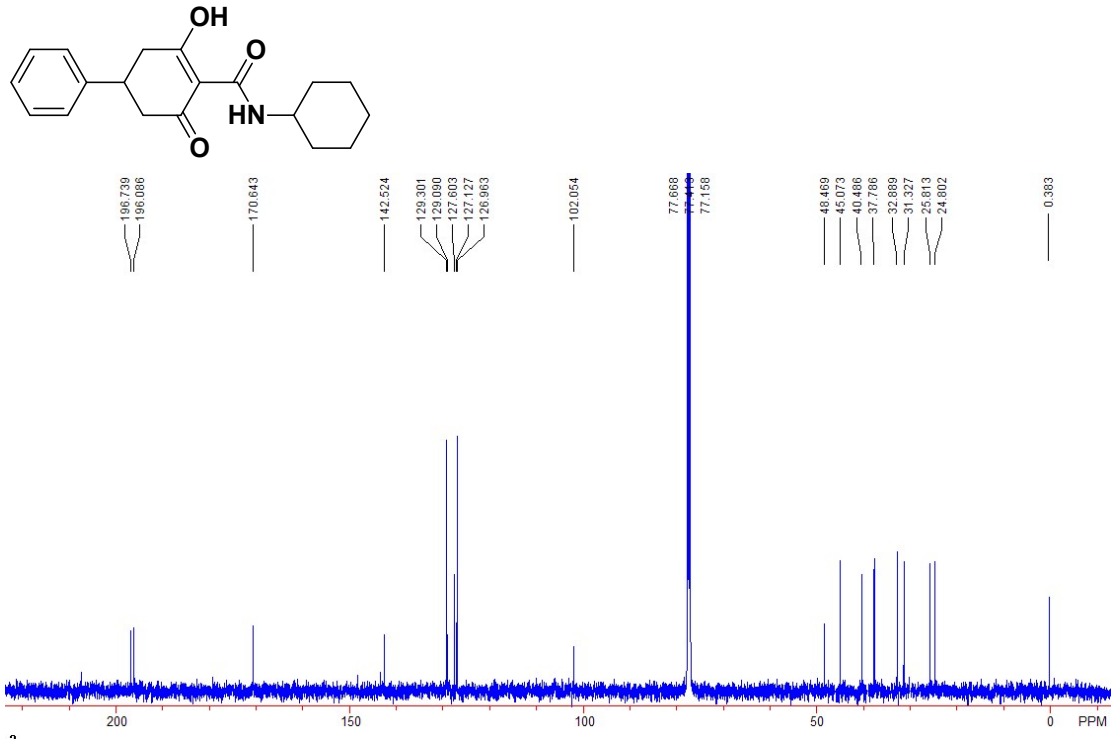




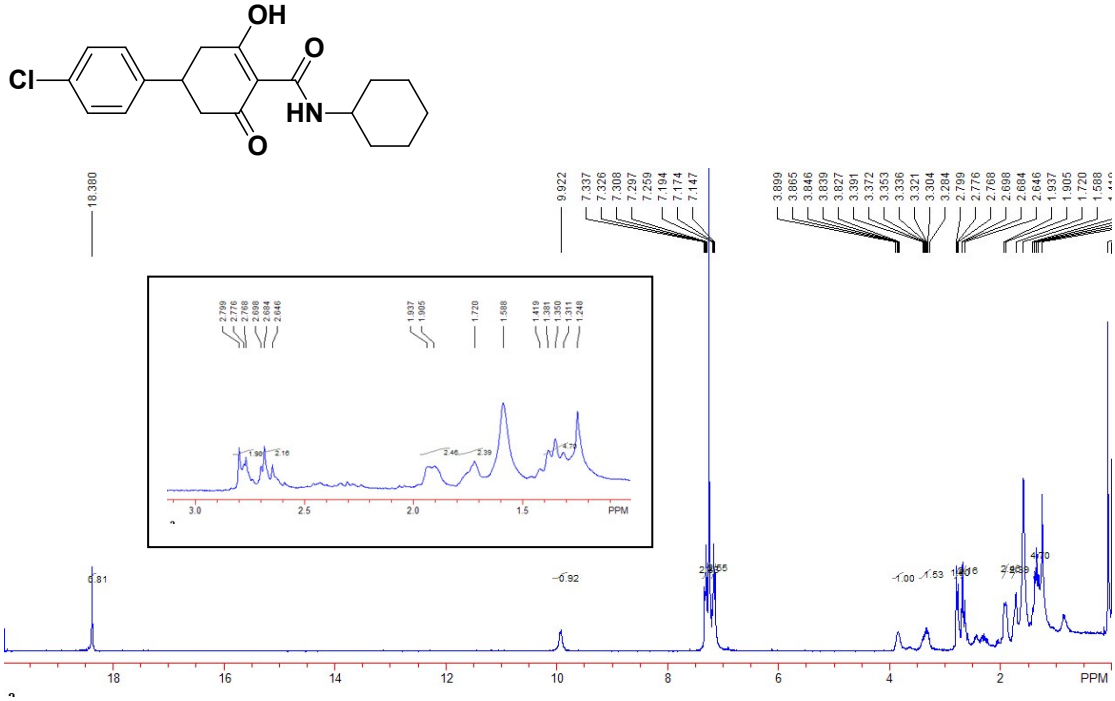


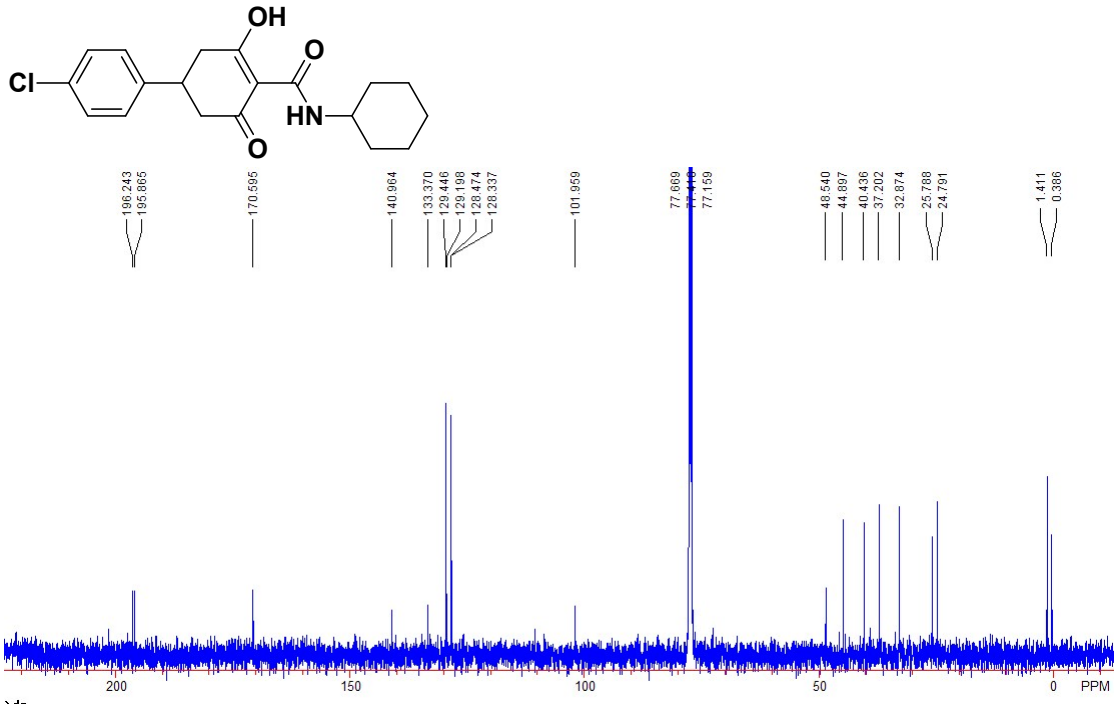
^{1}H NMR and ^{13}C NMR Spectra of Compound 3q





^{1}H NMR and ^{13}C NMR Spectra of Compound 3r





Crystal data and structure refinement for compound 3b.

The diffraction data for **3b** were collected on a Bruker SMART APEX diffractometer using Mo K α (λ = 0.71073 Å). The crystallographic data are listed in Table S1. The CIF deposition number: **CCDC-1448592**.

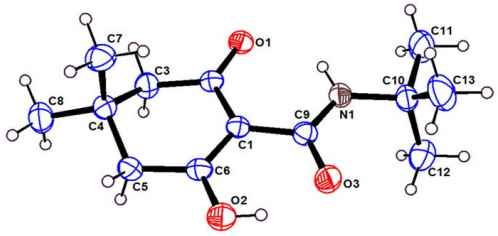


Figure S1. X-ray crystal structure of compound **3b** (The ellipsoid contour probability level in the caption is 30%).

Table S1. Crystal data and structure refinement for **3b**.

Identification code	131225c_0m
Empirical formula	C13 H21 N O3
Formula weight	239.31
Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 11.0499 (14) A alpha = 90 deg.
	b = 11.3287 (14) A beta = 113.0720(10) deg.
	c = 11.7946 (14) A gamma = 90 deg.
Volume	1351.9(3) A^3
Z, Calculated density	4, 1.176 Mg/m^3
Absorption coefficient	0.083 mm^-1
F(000)	520
Crystal size	0.13 x 0.12 x 0.10 mm
Theta range for data collection	2.13 to 27.66 deg.
Limiting indices	-14<=h<=14, -14<=k<=13, -15<=l<=15
Reflections collected / unique	11446 / 3137 [R(int) = 0.0281]
Completeness to theta = 27.76	99.4 %
Absorption correction	None
Max. and min. transmission	0.9918 and 0.9893
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3137 / 0 / 161
Goodness-of-fit on F^2	1.052
Final R indices [I>2sigma(I)]	R1 = 0.0422, wR2 = 0.1160
R indices (all data)	R1 = 0.0558, wR2 = 0.1253
Extinction coefficient	0.045(4)
Largest diff. peak and hole	0.222 and -0.127 e.A^-3