

SUPPORTING INFORMATION

Task-Specific Acidic Ionic Liquid Catalyzed Efficient Synthesis of β -Enaminolactones from Alkynoates and β - Aminoalcohols

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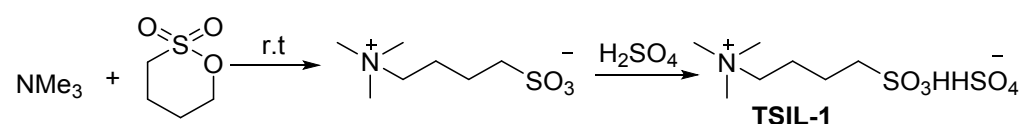
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1. General information

^1H and ^{13}C NMR spectra were recorded on Bruker Avance 400 MHz NMR spectrometer using CDCl_3 as solvent and TMS as an internal standard. Gas chromatograph mass spectra were obtained with a Shimadzu GC-MS-QP5050A spectrometer at an ionization voltage of 70 eV equipped with a DB-WAX capillary column (internal diameter: 0.25 mm, length: 30 m). High-resolution mass spectra (HRMS) were recorded on a Shimadzu LCMS-IT-TOF mass spectrometer. Melting points were obtained with an Electrothermal SGW-X4 microscopy digital melting point apparatus and are uncorrected; IR spectra were recorded on a FTLA2000 spectrometer; Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF254), and was performed under UV light (254 nm). All the new products were further characterized by high resolution mass spectra (HRMS). All commercial reagents and available compounds were obtained from J&K Chemical, TCI, Fluka, Acros, and SCRC, and used without further purification.

Typical procedure for the synthesis and recycle of ionic liquid

The synthesis of acidic ionic liquid [TMBSA] HSO_4 has been carried out using the same method reported in the literature (Scheme 2).¹



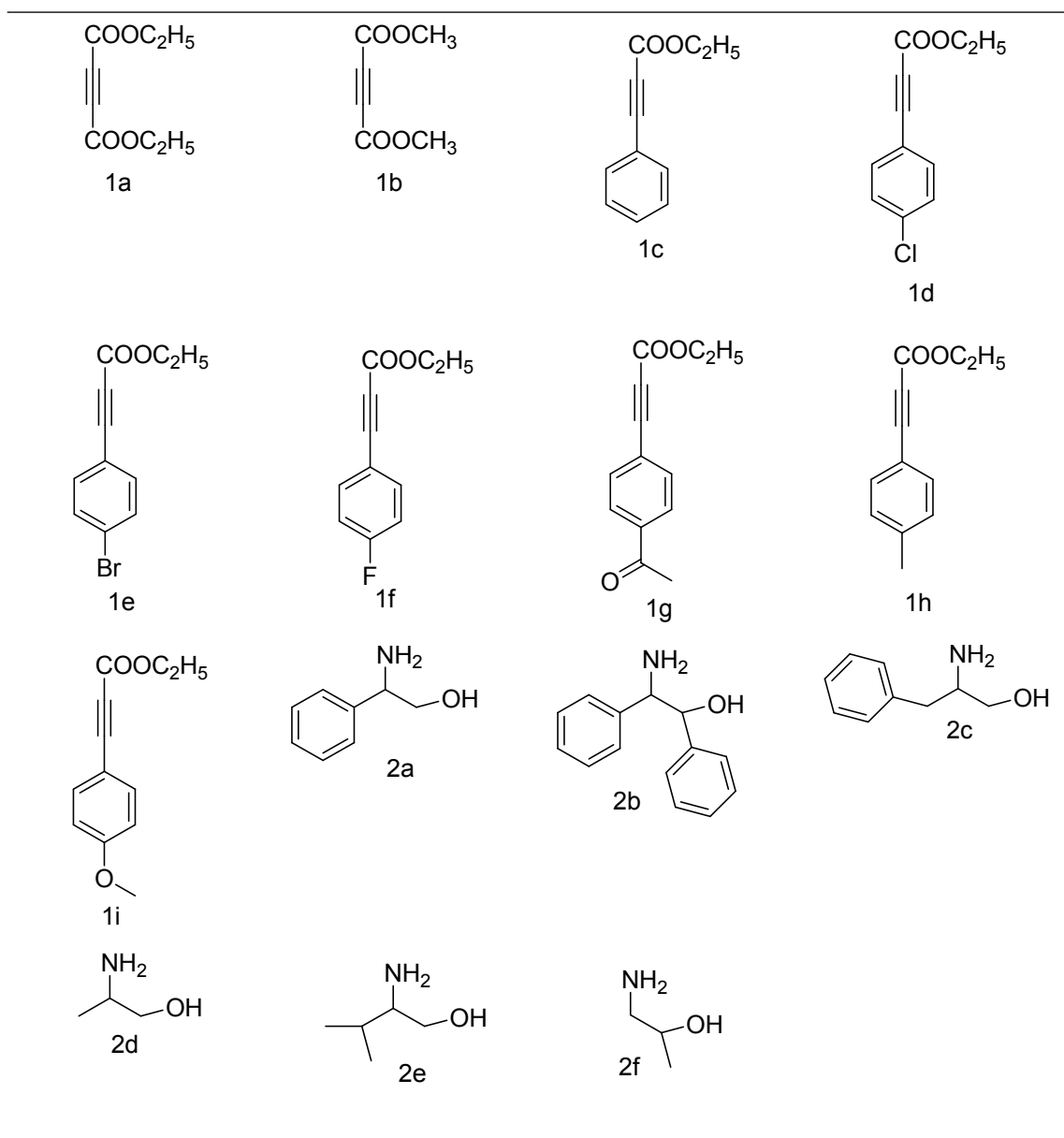
Scheme 2

The ionic liquid in the filtrate was separated from the unreacted starting materials by extracting the filtrate with ether. Then, the water layer containing ionic liquid was vacuum-dried at 75°C for 4 h to remove water, and the ionic liquid was reused.

2. General procedure for synthesis of 3,4-dihydro-1,4-oxazepin-7(2H)-one derivatives

A mixture of dimethyl acetylenedicarboxylate (**1**) (0.5 mmol), 2-amino-2-phenylethanol (**2**) (0.6 mmol), catalyst (10 mol%) in tert-Amyl alcohol (2.0 ml) was

stired at 120°C for 12h under air atmosphere. After completion, the reaction mixture was cooled down to the room temperature. The ionic liquid was separated from the reaction mixture by extraction with water. Then the mixture in organic layer was evaporated under vacuum and the residue was subjected to a column chromatography on silica gel (petroleum ether /ethyl acetate, 8/1) to give the product (**3**) as a yellow viscous liquid.



Scheme S1. Substrates employed for β -enaminolactones derivatives syntheses

3. NMR spectra of the obtained compounds

ethyl 7-oxo-3-phenyl-2,3,4,7-tetrahydro-1,4-oxazepine-5-carboxylate (**3a**)

Yellow oil, yield: 86%. ^1H NMR (400 MHz, CDCl_3) δ 8.48 (s, 1H), 7.34 – 7.26 (m,

5H), 5.67 (s, 1H), 4.68 (dd, $J = 2.8, 4.0$ Hz, 1H), 4.40 (dd, $J = 4.0, 4.0$ Hz, 1H), 4.35 – 4.27 (m, 1H), 4.07 (q, $J = 8.0$ Hz, 2H), 1.19 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 169.94, 160.09, 143.92, 135.75, 129.28, 129.20, 126.75, 91.22, 72.36, 59.80, 52.97, 14.33. IR (KBr): 3307, 2962, 1747, 1662, 1458, 1389, 1264, 1228, 1132, 1027, 768, 699 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{14}\text{H}_{16}\text{NO}_4$ ($[\text{M}+\text{H}]^+$), 262.1074, found 262.1076.

ethyl 7-oxo-2,3-diphenyl-2,3,4,7-tetrahydro-1,4-oxazepine-5-carboxylate (3b)

Yellow oil, yield: 78%. ^1H NMR (400 MHz, CDCl_3) δ 8.73 (s, 1H), 7.46 – 6.90 (m, 10H), 6.83 (d, $J = 8.0$ Hz, 1H), 6.64 (d, $J = 8.0$ Hz, 1H), 5.77 (s, 1H), 4.08 (q, $J = 8.0$ Hz, 2H), 1.20 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.02, 160.99, 143.85, 134.77, 134.09, 128.72, 128.38, 128.18, 128.16, 127.69, 126.64, 90.67, 82.19, 59.87, 57.75, 14.38. IR (KBr): 3307, 2924, 1744, 1662, 1617, 1453, 1249, 1193, 1102, 1032, 771, 699 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{20}\text{H}_{20}\text{NO}_4$ ($[\text{M}+\text{H}]^+$), 338.1387, found 338.1389.

ethyl 3-benzyl-7-oxo-2,3,4,7-tetrahydro-1,4-oxazepine-5-carboxylate (3c)

Yellow oil, yield: 94%. ^1H NMR (400 MHz, CDCl_3) δ 8.30 (s, 1H), 7.29 (t, $J = 8.0$ Hz, 2H), 7.25 – 7.19 (m, 1H), 7.14 (d, $J = 8.0$ Hz, 2H), 5.58 (s, 1H), 4.33 (dd, $J = 1.6, 1.6$ Hz, 1H), 4.19 (dd, $J = 11.0, 8.0$ Hz, 1H), 4.07 (q, $J = 8.0$ Hz, 2H), 3.75 – 3.66 (m, 1H), 2.80 (d, $J = 8.0$ Hz, 2H), 1.19 (t, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 169.82, 160.46, 143.39, 135.53, 129.08, 127.45, 90.80, 70.31, 59.72, 49.79, 38.18, 14.36. IR (KBr): 2926, 1745, 1662, 1618, 1459, 1390, 1353, 1237, 1146, 1032, 770, 702 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{15}\text{H}_{18}\text{NO}_4$ ($[\text{M}+\text{H}]^+$), 276.1230, found 276.1236.

methyl 7-oxo-3-phenyl-2,3,4,7-tetrahydro-1,4-oxazepine-5-carboxylate (3d)

Yellow oil, yield: 80%. ^1H NMR (400 MHz, CDCl_3) δ 8.48 (s, 1H), 7.38 – 7.29 (m, 5H), 5.71 (s, 1H), 4.70 (d, $J = 8.0$ Hz, 1H), 4.43 (d, $J = 8.0$ Hz, 1H), 4.34 (t, $J = 12.0$ Hz, 1H), 3.64 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.36, 160.10, 143.98, 135.59, 129.34, 126.79, 90.90, 72.39, 53.08, 51.09. IR (KBr): 3310, 2920, 2353, 1744, 1666, 1618, 1457, 1388, 1337, 1265, 1229, 1133, 1013, 764, 700 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{13}\text{H}_{14}\text{NO}_4$ ($[\text{M}+\text{H}]^+$), 248.0917, found 248.0912.

methyl 3-benzyl-7-oxo-2,3,4,7-tetrahydro-1,4-oxazepine-5-carboxylate (3e)

Yellow oil, yield: 90%. ¹H NMR (400 MHz, CDCl₃) δ 8.28 (s, 1H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.21 (dd, *J* = 4.0, 4.0 Hz, 1H), 7.14 (d, *J* = 4.0 Hz, 2H), 5.58 (s, 1H), 4.33 (dd, *J* = 4.0, 4.0 Hz, 1H), 4.18 (dd, *J* = 8.0, 8.0 Hz, 1H), 3.74 – 3.67 (m, 1H), 3.60 (s, 3H), 2.79 (d, *J* = 8.0 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 170.17, 160.40, 143.49, 135.46, 129.12, 129.07, 127.49, 90.33, 70.33, 51.03, 49.80, 38.15. IR (KBr): 3306, 2924, 1745, 1666, 1616, 1460, 1388, 1360, 1239, 1147, 1014, 740, 703 cm⁻¹. HRMS *m/z* (ESI⁺) calcd for C₁₄H₁₆NO₄ ([M+H]⁺), 262.1074, found 262.1071.

3,5-diphenyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3f)

Yellow oil, yield: 68%. ¹H NMR (400 MHz, CDCl₃) δ 9.03 (s, 1H), 7.32 – 7.22 (m, 5H), 7.19 (s, 1H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 4.66 (s, 1H), 4.43 – 4.34 (m, 1H), 3.70 (d, *J* = 8.0 Hz, 2H). ¹³C NMR (101 MHz, CDCl₃) δ 170.59, 164.81, 140.02, 135.98, 129.34, 128.72, 128.30, 127.91, 127.57, 126.49, 88.02, 67.29, 59.06. IR (KBr): 3632, 3755, 3693, 3067, 2924, 2857, 2373, 1740, 1580, 1467, 1267, 1176, 1028, 742, 701 cm⁻¹. HRMS *m/z* (ESI⁺) calcd for C₁₇H₁₆NO₂ ([M+H]⁺), 266.1176, found 266.1169.

3-methyl-5-phenyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3g)

Yellow oil, yield: 78%. ¹H NMR (400 MHz, CDCl₃) δ 8.28 (s, 1H), 7.43-7.25 (m, 5H), 4.59 (s, 1H), 3.47 – 3.34 (m, 3H), 1.04 (d, *J* = 8.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 170.52, 164.86, 136.40, 129.33, 128.46, 127.87, 87.20, 67.15, 51.75, 18.57. IR (KBr): 3483, 2978, 2362, 1646, 1478, 1288, 1155, 1041, 775, 703 cm⁻¹. HRMS *m/z* (ESI⁺) calcd for C₁₂H₁₄NO₂ ([M+H]⁺), 204.1019, found 204.1012.

3-isopropyl-5-phenyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3h)

Yellow oil, yield: 58%. ¹H NMR (400 MHz, CDCl₃) δ 8.43 (s, 1H), 7.52-7.20 (m, 5H), 4.59 (s, 1H), 3.60 – 3.31 (m, 2H), 3.21 – 2.93 (m, 1H), 1.77-1.67 (m, 1H), 0.81 (dd, *J* = 2.0, *J* = 4.0 Hz, 6H). ¹³C NMR (101 MHz, CDCl₃) δ 170.53, 166.05, 136.40, 129.21, 128.34, 128.26, 86.93, 64.30, 61.11, 30.55, 19.62. IR (KBr): 3886, 3833, 3754, 3687, 2960, 2365, 1738, 1646, 1593, 1473, 1277, 1172, 1032, 777, 706, 651 cm⁻¹. HRMS *m/z* (ESI⁺) calcd for C₁₅H₁₈NO₄ ([M+H]⁺), 232.1332, found 232.1327.

3-benzyl-5-phenyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3i)

Yellow oil, yield: 68%. ^1H NMR (400 MHz, CDCl_3) δ 8.42 (s, 1H), 7.29-7.22 (m, 2H), 7.19 – 7.13 (m, 5H), 6.91 (dd, $J = 8.0, 8.0$ Hz, 3H), 4.51 (s, 1H), 3.55 – 3.46 (m, 2H), 3.45 – 3.40 (m, 1H), 2.71 (dd, $J = 4.0, 4.0$ Hz, 1H), 2.62 (q, $J = 8.0$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.50, 165.08, 137.91, 136.07, 129.52, 129.14, 128.40, 128.19, 127.87, 126.46, 87.33, 65.30, 57.96, 39.47. IR (KBr): 3869, 3695, 2935, 2338, 1740, 1648, 1591, 1475, 1265, 1174, 1033, 749, 701 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{18}\text{H}_{18}\text{NO}_2$ ($[\text{M}+\text{H}]^+$), 280.1259, found 280.1262.

2-methyl-5-phenyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3j)

Yellow oil, yield: 74%. ^1H NMR (400 MHz, CDCl_3) δ 8.28 (s, 1H), 7.27-7.41 (m, 5H), 4.60 (s, 1H), 3.47 – 3.36 (m, 3H), 1.04 (d, $J = 8.0$ Hz). ^{13}C NMR (101 MHz, CDCl_3) δ 170.52, 164.85, 136.40, 129.33, 128.46, 127.88, 87.27, 67.17, 51.75, 18.56. IR (KBr): 3867, 3762, 3650, 2924, 1740, 1650, 1586, 1475, 1282, 1157, 1039, 740 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{12}\text{H}_{14}\text{NO}_2$ ($[\text{M}+\text{H}]^+$), 204.1019, found 204.1013.

5-(4-chlorophenyl)-3-methyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3k)

Yellow oil, yield: 84%. ^1H NMR (400 MHz, CDCl_3) δ 8.23 (s, 1H), 7.28 (d, d, $J = 8.0, 8.0$ Hz, 4H), 4.56 (s, 1H), 3.50 – 3.28 (m, 3H), 1.02 (d, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.39, 163.51, 135.38, 134.81, 129.28, 128.76, 87.46, 67.03, 51.85, 18.55. IR (KBr): 3449, 2972, 1646, 1609, 1477, 1286, 1158, 1041, 833, 789 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{12}\text{H}_{13}\text{ClNO}_2$ ($[\text{M}+\text{H}]^+$), 238.0629, found 238.0625.

5-(4-bromophenyl)-3-methyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3l)

Yellow oil, yield: 89%. ^1H NMR (400 MHz, CDCl_3) δ 8.23 (s, 1H), 7.45 (d, $J = 8.0$ Hz, 2H), 7.20 (d, $J = 8.0$ Hz, 2H), 4.55 (s, 1H), 3.34 – 3.44 (m, 3H), 1.02 (d, $J = 8.0$ Hz, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.38, 163.51, 135.28, 131.72, 129.54, 123.60, 87.40, 67.02, 51.85, 18.56. IR (KBr): 3449, 2974, 1648, 1605, 1475, 1286, 1173, 1043, 833, 790, 725 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{12}\text{H}_{13}\text{BrNO}_2$ ($[\text{M}+\text{H}]^+$), 282.0124, found 282.0117.

5-(4-chlorophenyl)-3-isopropyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3m)

Yellow oil, yield: 60%. ^1H NMR (400 MHz, CDCl_3) δ 8.39 (s, 1H), 7.35 – 7.25 (m, 4H), 4.57 (s, 1H), 3.48-3.56 (m, 2H), 3.03-3.10 (m, 1H), 1.70 (m, 1H), 0.85 (d, $J = 8.0$ Hz, 3H), 0.76 (d, $J = 4.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.39, 164.72,

135.30, 134.80, 129.68, 128.66, 87.21, 64.31, 61.27, 30.65, 19.62. IR (KBr): 3702, 2925, 1733, 1652, 1600, 1473, 1270, 1176, 1089, 1019, 837, 790, 739 cm^{-1} . HRMS m/z (ESI⁺) calcd for $\text{C}_{14}\text{H}_{17}\text{ClNO}_2$ ([M+H]⁺), 266.0942, found 266.0936.

5-(4-bromophenyl)-3-isopropyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3n)

Yellow oil, yield: 62%. ¹H NMR (400 MHz, CDCl_3) δ 8.39 (s, 1H), 7.45 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 4.56 (s, 1H), 3.56 – 3.47 (m, 2H), 3.10-3.02 (m, 1H), 1.66-1.72 (m, 1H), 0.85 (d, J = 8.0 Hz, 3H), 0.76 (d, J = 4.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl_3) δ 170.39, 164.73, 135.30, 131.62, 129.95, 123.52, 87.17, 64.33, 61.28, 30.66, 19.6. IR (KBr): 3693, 2957, 1733, 1651, 1599, 1472, 1273, 1176, 1070, 1015, 833, 790, 739 cm^{-1} . HRMS m/z (ESI⁺) calcd for $\text{C}_{14}\text{H}_{17}\text{BrNO}_2$ ([M+H]⁺), 310.0437, found 310.0434.

3-methyl-5-(p-tolyl)-3,4-dihydro-1,4-oxazepin-7(2H)-one (3o)

Yellow oil, yield: 72%. ¹H NMR (400 MHz, CDCl_3) δ 8.26 (s, 1H), 7.20 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 4.59 (s, 1H), 3.48 – 3.34 (m, 3H), 2.30 (s, 3H), 1.03 (d, J = 4.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl_3) δ 170.58, 165.06, 139.43, 133.48, 129.13, 127.80, 87.04, 67.17, 51.76, 21.29, 18.54. IR (KBr): 3661, 2925, 1739, 1649, 1594, 1483, 1269, 1155, 1049, 740 cm^{-1} . HRMS m/z (ESI⁺) calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_2$ ([M+H]⁺), 218.1176, found 218.1172.

5-(4-methoxyphenyl)-3-methyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3p)

Yellow oil, yield: 70%. ¹H NMR (400 MHz, CDCl_3) δ 8.22 (s, 1H), 7.26 (d, J = 12.0 Hz, 2H), 6.83 (d, J = 8.0 Hz, 2H), 4.60 (s, 1H), 3.76 (s, 3H), 3.49 – 3.33 (m, 3H), 1.04 (d, J = 8.0 Hz, 3H). ¹³C NMR (101 MHz, CDCl_3) δ 170.57, 164.82, 160.51, 130.92, 129.30, 113.84, 87.15, 67.17, 55.32, 51.86, 18.51. IR (KBr): 3703, 2927, 1600, 1480, 1282, 1161, 1034, 740 cm^{-1} . HRMS m/z (ESI⁺) calcd for $\text{C}_{13}\text{H}_{16}\text{NO}_3$ ([M+H]⁺), 234.1125, found 234.1120.

3-isopropyl-5-(p-tolyl)-3,4-dihydro-1,4-oxazepin-7(2H)-one (3q)

Yellow oil, yield: 56%. ¹H NMR (400 MHz, CDCl_3) δ 8.41 (s, 1H), 7.21 (d, J = 8.0 Hz, 2H), 7.11 (d, J = 8.0 Hz, 2H), 4.59 (s, 1H), 3.56 – 3.49 (m, 2H), 3.14 (m, 1H), 2.30 (s,

3H), 1.72 (m, 1H), 0.86 (d, $J = 8.0$ Hz, 3H), 0.77 (d, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.58, 166.27, 139.29, 133.46, 129.03, 128.17, 86.75, 64.30, 61.12, 30.59, 21.30, 18.16. IR (KBr): 3698, 2926, 1594, 1476, 1286, 1171, 1034, 739 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{15}\text{H}_{20}\text{NO}_2$ ($[\text{M}+\text{H}]^+$), 246.1489, found 246.1483.

3-isopropyl-5-(4-methoxyphenyl)-3,4-dihydro-1,4-oxazepin-7(2H)-one (3r)

Yellow oil, yield: 50%. ^1H NMR (400 MHz, CDCl_3) δ 8.38 (s, 1H), 7.27 (d, $J = 8.0$ Hz, 2H), 6.83 (d, $J = 8.0$ Hz, 2H), 4.59 (s, 1H), 3.76 (s, 3H), 3.51 (d, $J = 4.0$ Hz, 2H), 3.16 (m, 1H), 1.71 (m, 1H), 0.86 (d, $J = 8.0$ Hz, 3H), 0.77 (d, $J = 4.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.59, 166.04, 160.38, 129.66, 128.62, 113.74, 86.81, 64.29, 61.22, 55.30, 30.68, 18.19. IR (KBr): 3698, 1734, 1600, 1475, 1264, 1170, 1030, 740 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{15}\text{H}_{20}\text{NO}_3$ ($[\text{M}+\text{H}]^+$), 262.1438, found 262.1433.

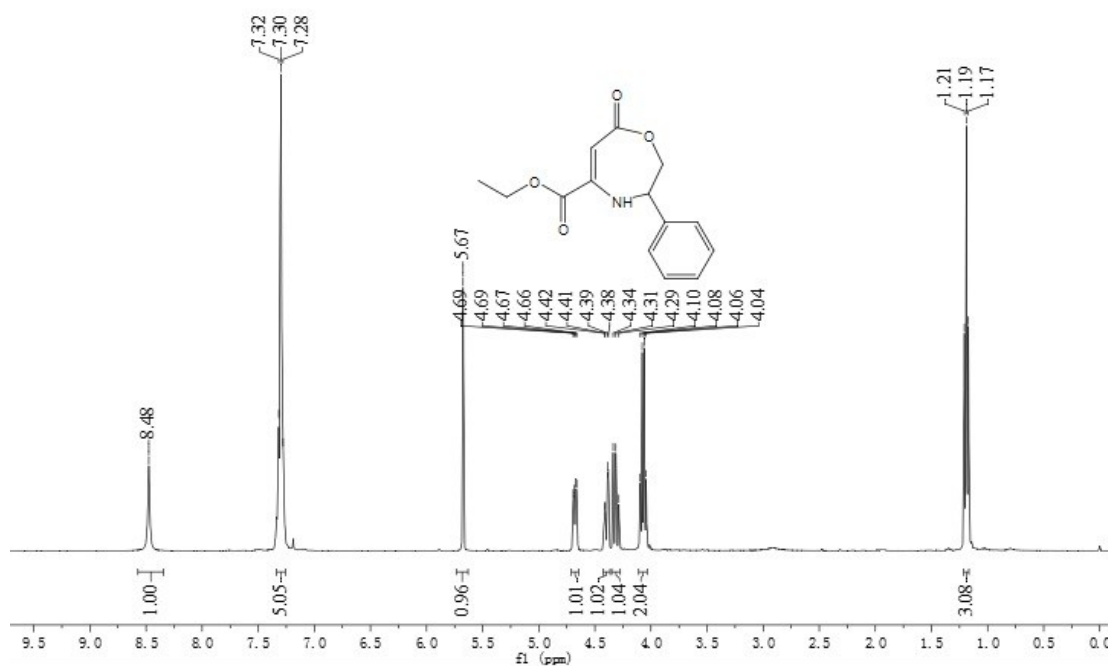
5-(4-fluorophenyl)-3-methyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3s)

Yellow oil, yield: 70%. ^1H NMR (400 MHz, CDCl_3) δ 8.25 (s, 1H), 7.31 (dd, $J = 4.0$, 4.0 Hz, 2H), 7.00 (t, $J = 8.0$ Hz, 2H), 4.55 (s, 1H), 3.40 (m, 3H), 1.02 (d, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.41, 163.72, 163.24 (d, $J = 248$ Hz), 132.40 (d, $J = 3.0$ Hz), 129.80 (d, $J = 9.0$ Hz), 115.53 (d, $J = 21$ Hz), 87.36, 67.01, 51.80, 18.55. IR (KBr): 3700, 2978, 1650, 1607, 1484, 1271, 1226, 1156, 1041, 843, 791, 740 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{12}\text{H}_{13}\text{FNO}_2$ ($[\text{M}+\text{H}]^+$), 222.0925, found 222.0921.

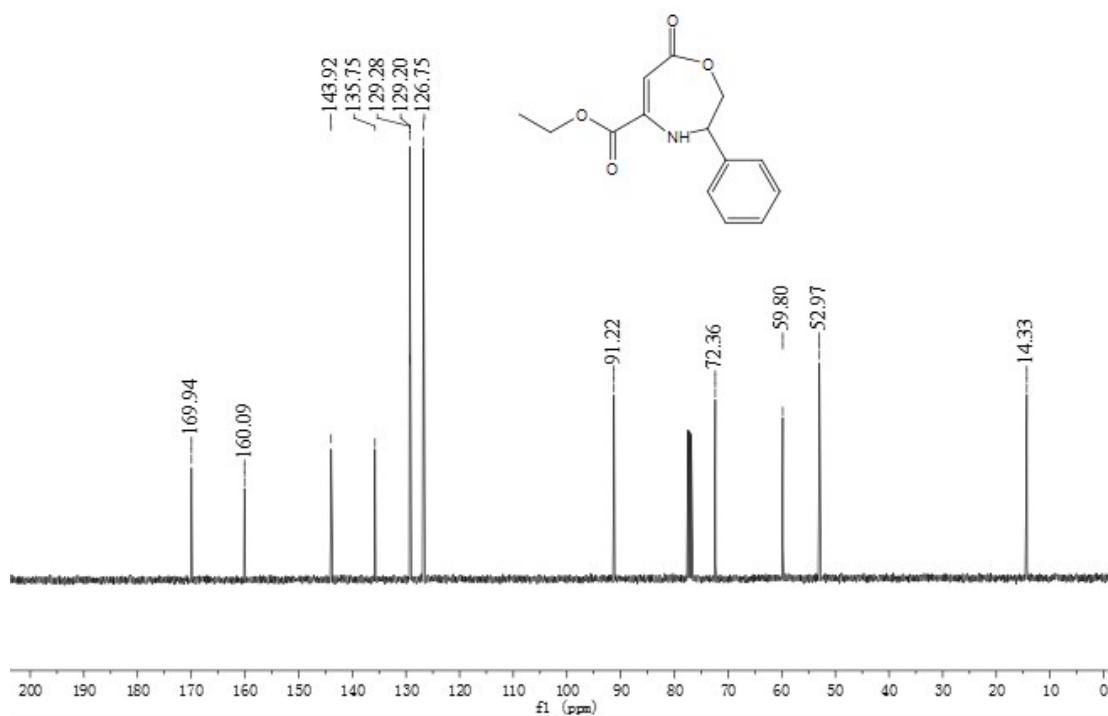
5-(4-acetylphenyl)-3-methyl-3,4-dihydro-1,4-oxazepin-7(2H)-one (3t)

Yellow oil, yield: 75%. ^1H NMR (400 MHz, CDCl_3) δ 8.27 (s, 1H), 7.91 (d, $J = 8.0$ Hz, 2H), 7.43 (d, $J = 8.0$ Hz, 2H), 4.60 (s, 1H), 3.39 (m, 3H), 2.55 (s, 3H), 1.04 (d, $J = 8.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 197.44, 170.26, 163.38, 141.00, 137.66, 128.46, 128.25, 87.81, 67.14, 51.93, 26.66, 18.55. IR (KBr): 3760, 3699, 2925, 2385, 1683, 1590, 1477, 1362, 1265, 1158, 1038, 845, 788, 741 cm^{-1} . HRMS m/z (ESI^+) calcd for $\text{C}_{14}\text{H}_{16}\text{NO}_3$ ($[\text{M}+\text{H}]^+$), 246.1125, found 246.1120.

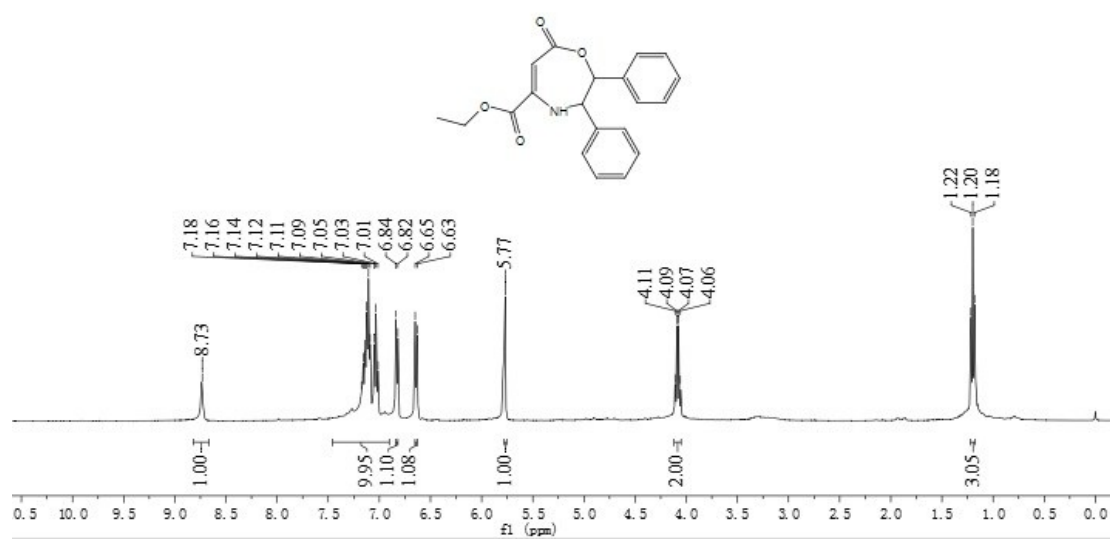
^1H NMR of **3a**



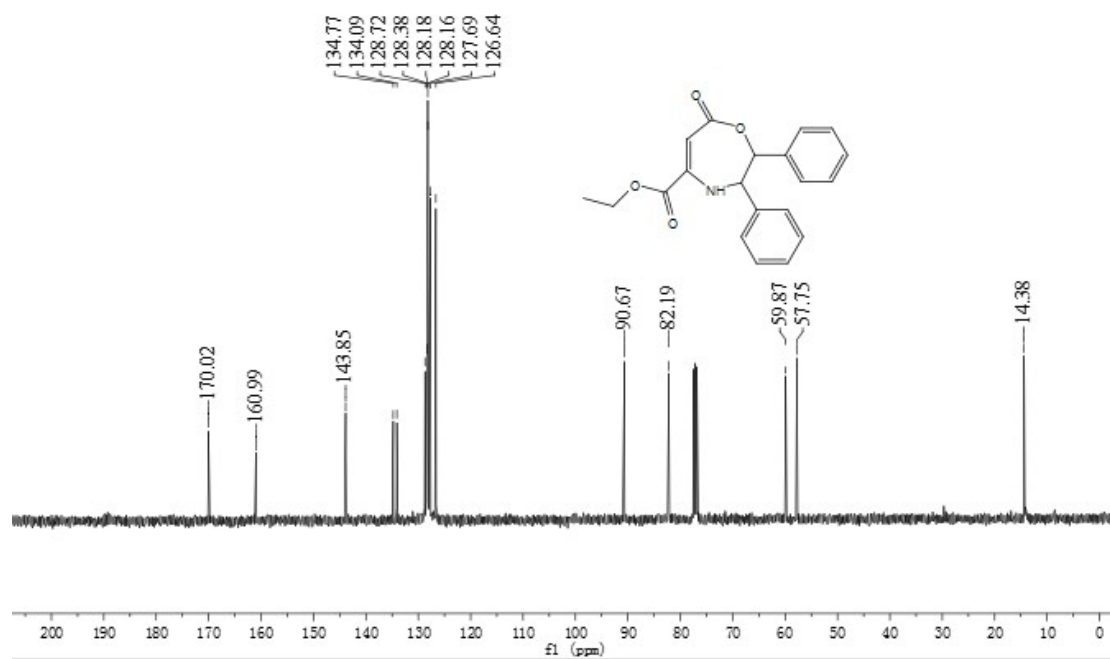
¹³CNMR of 3a



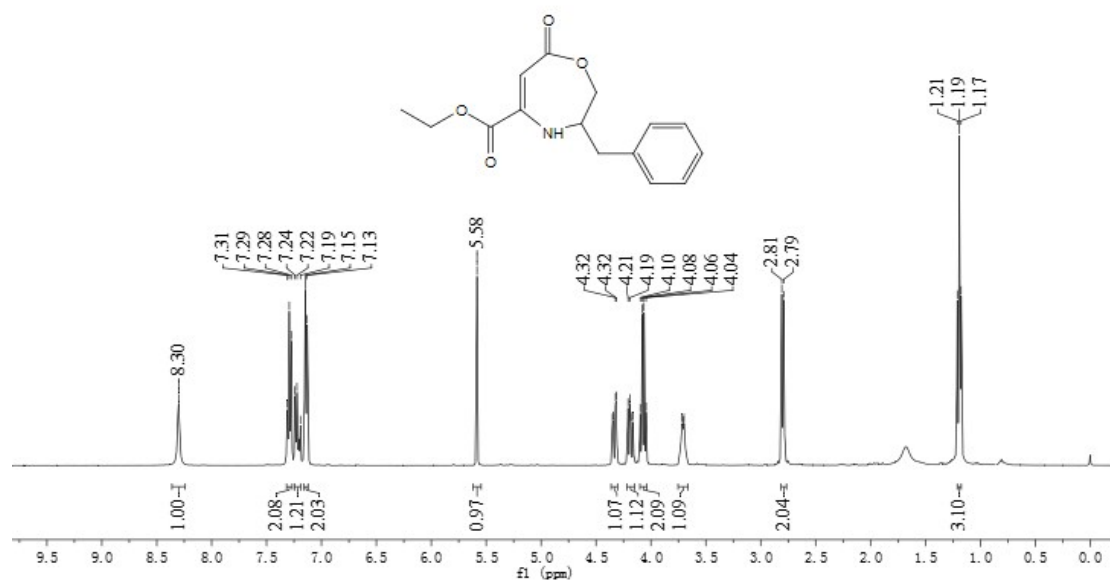
¹H NMR of 3b



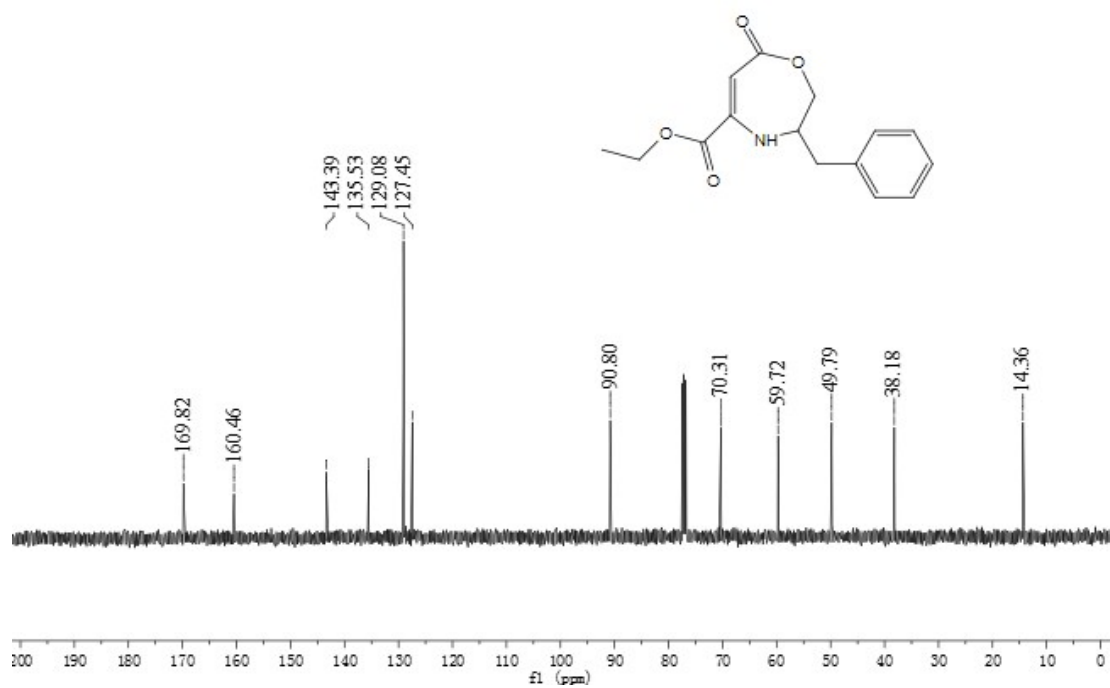
¹³CNMR of **3b**



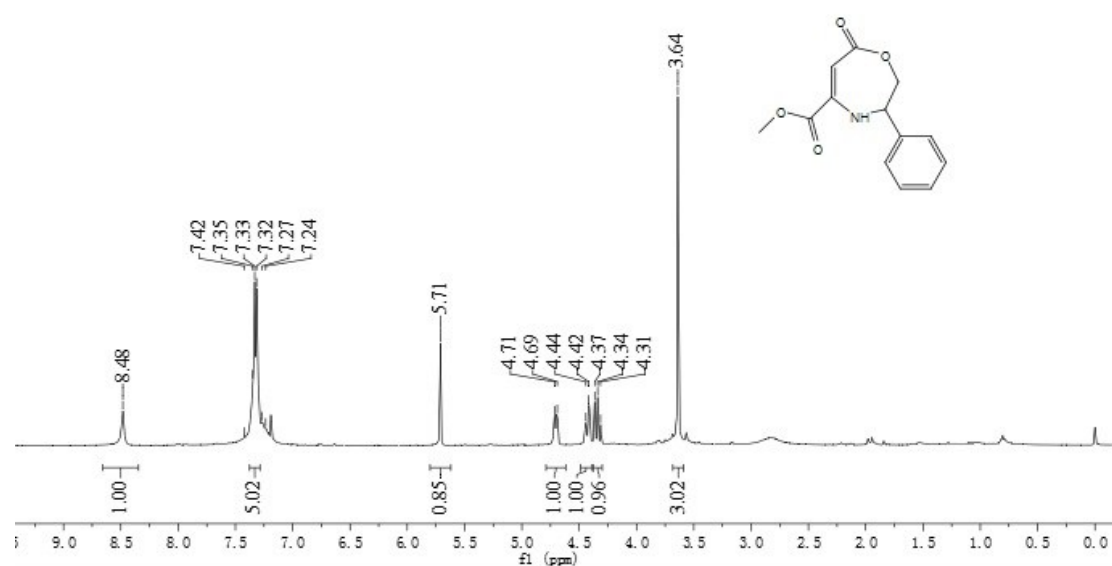
¹H NMR of **3c**



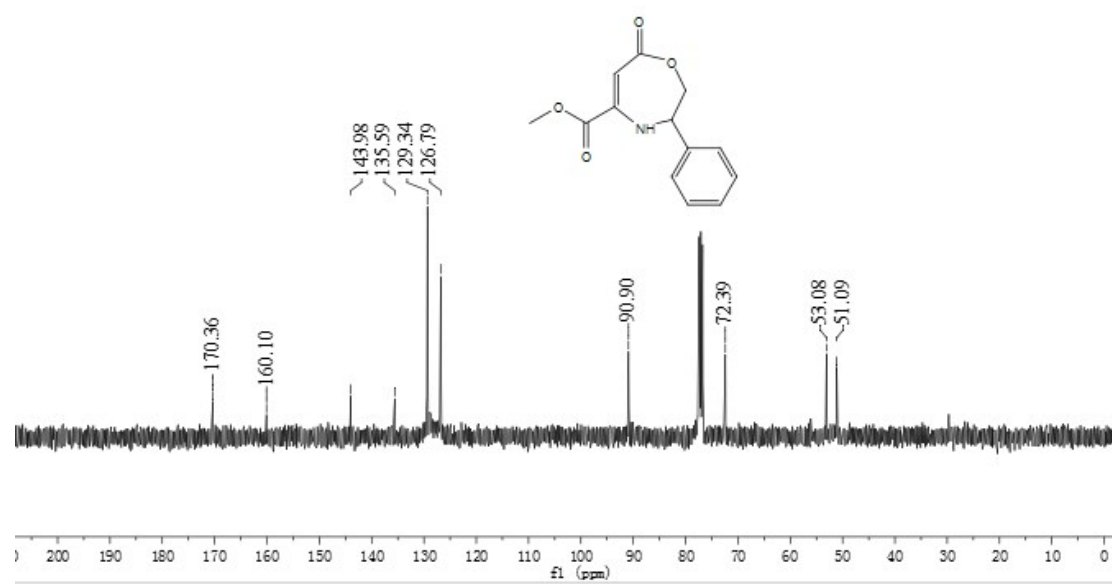
^{13}C NMR of **3c**



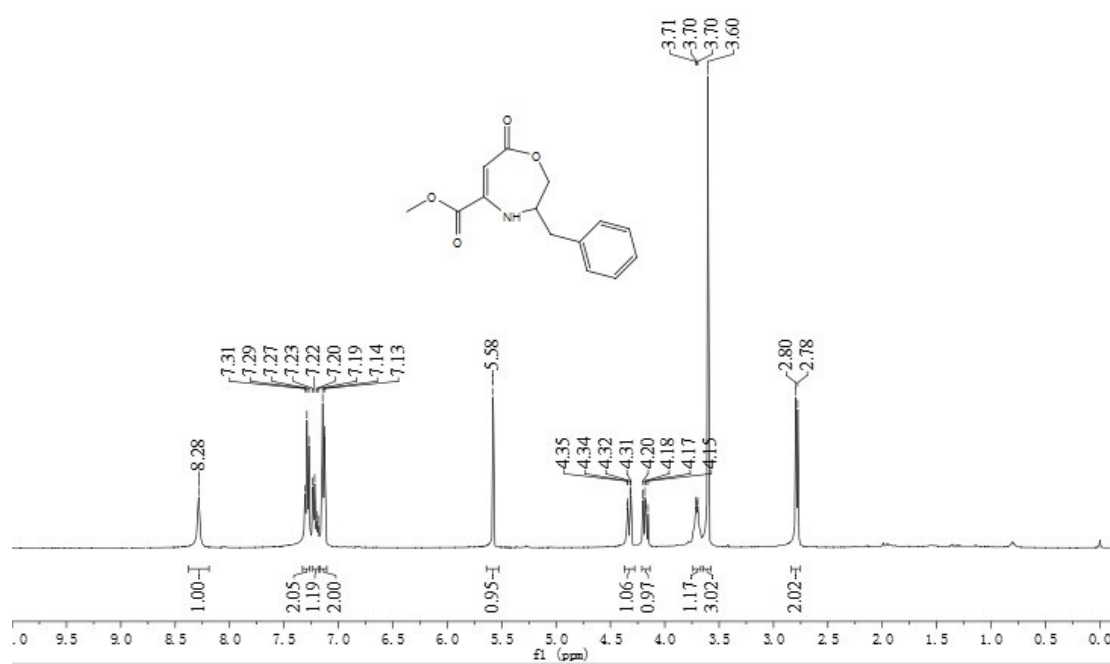
^1H NMR of **3d**



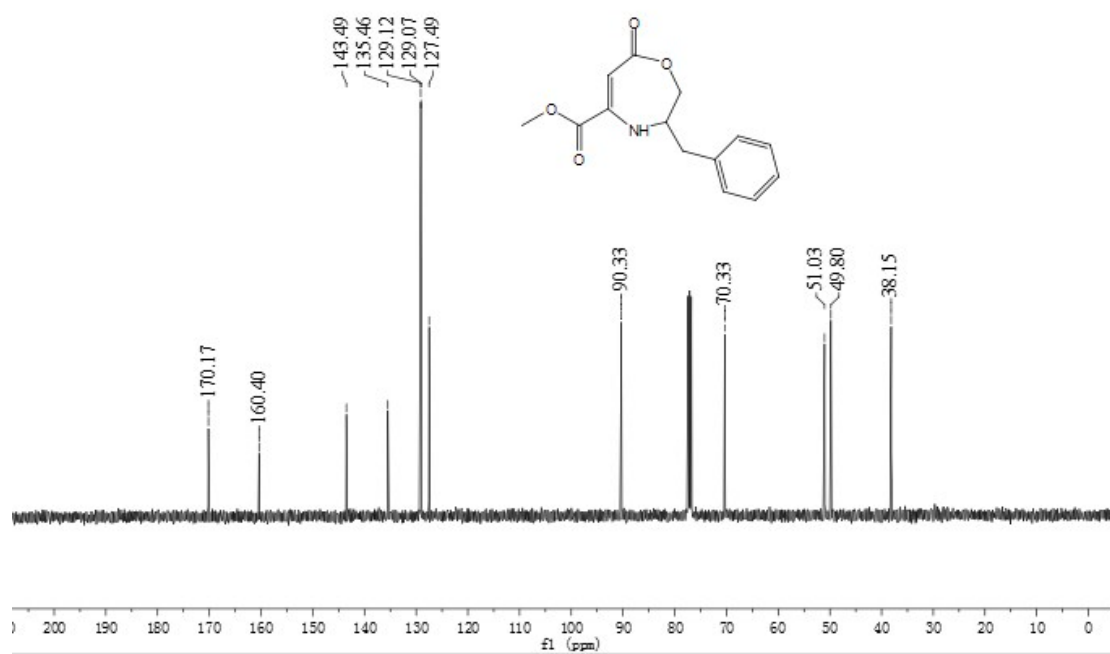
¹³CNMR of **3d**



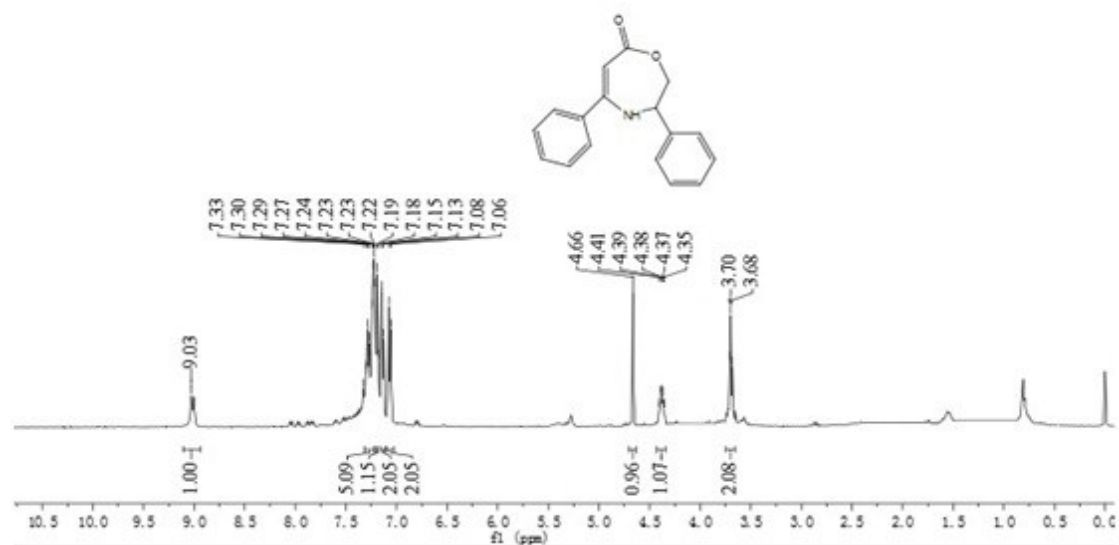
¹H NMR of **3e**



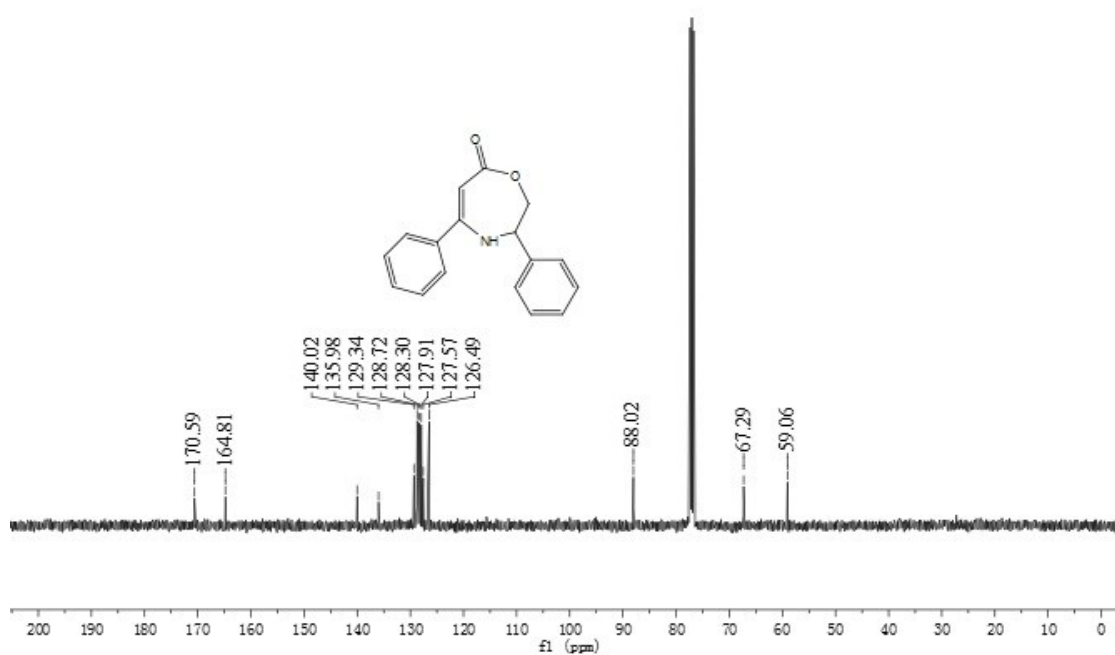
^{13}C NMR of **3e**



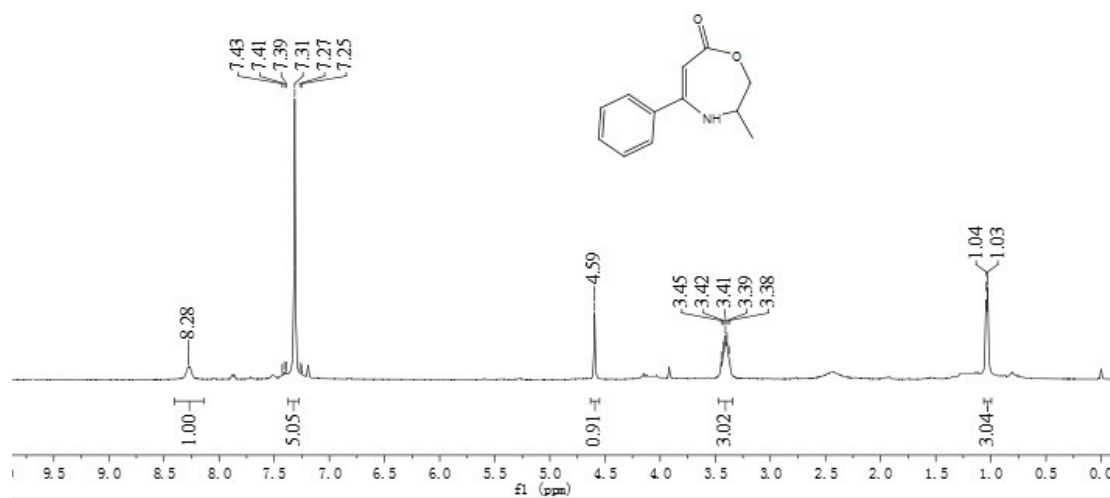
^1H NMR of **3f**



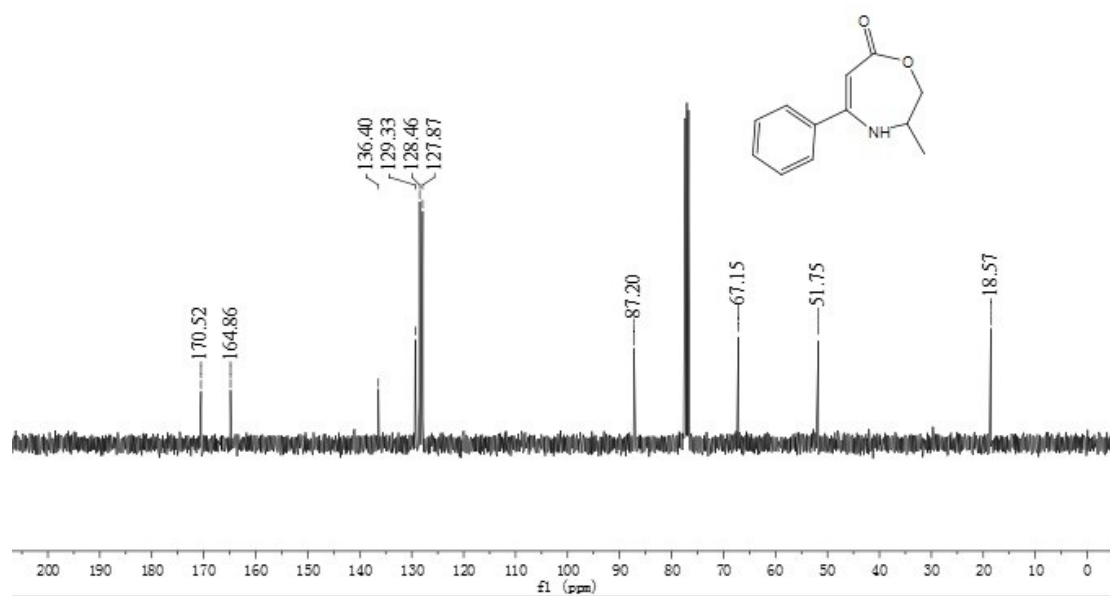
¹³CNMR of **3f**



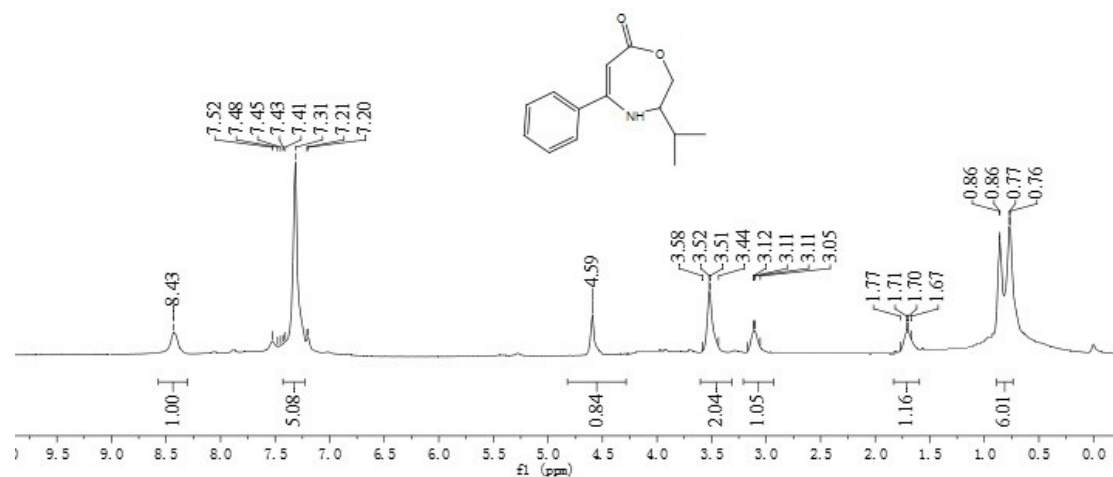
¹H NMR of **3g**



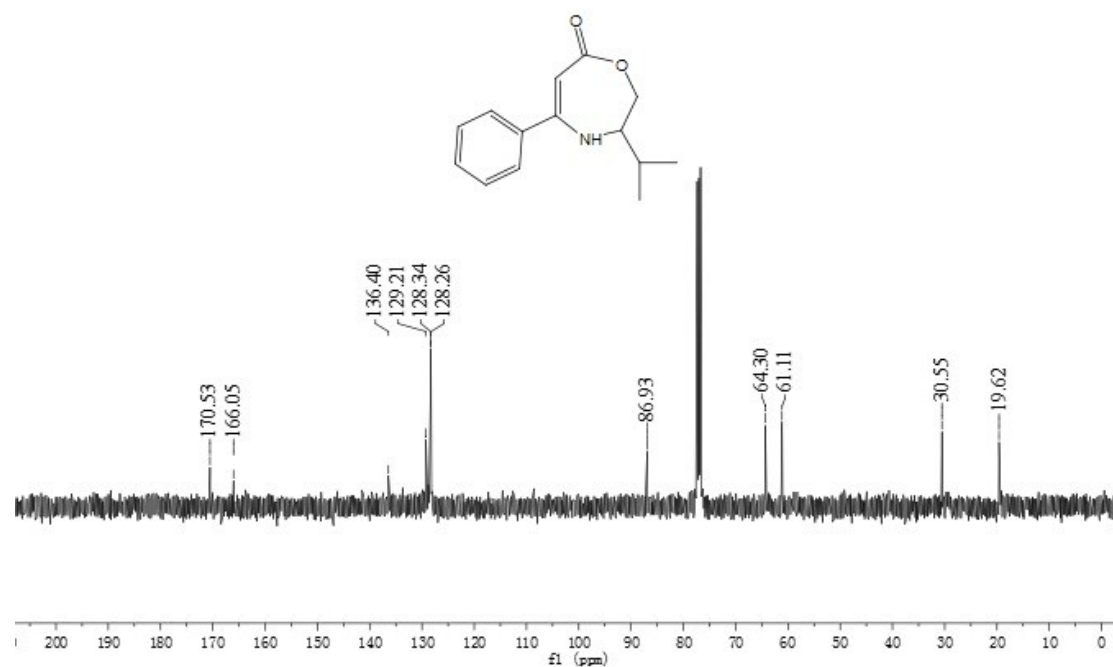
¹³CNMR of **3g**



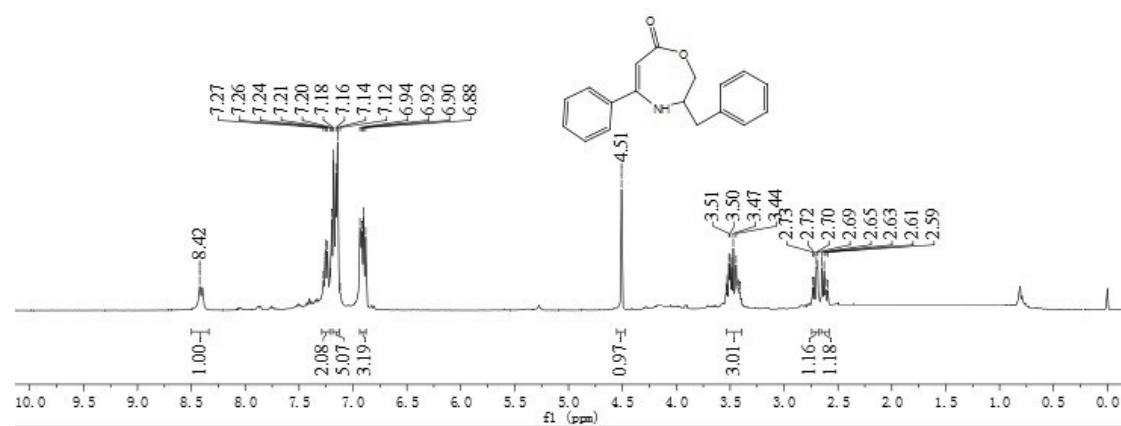
¹H NMR of **3h**



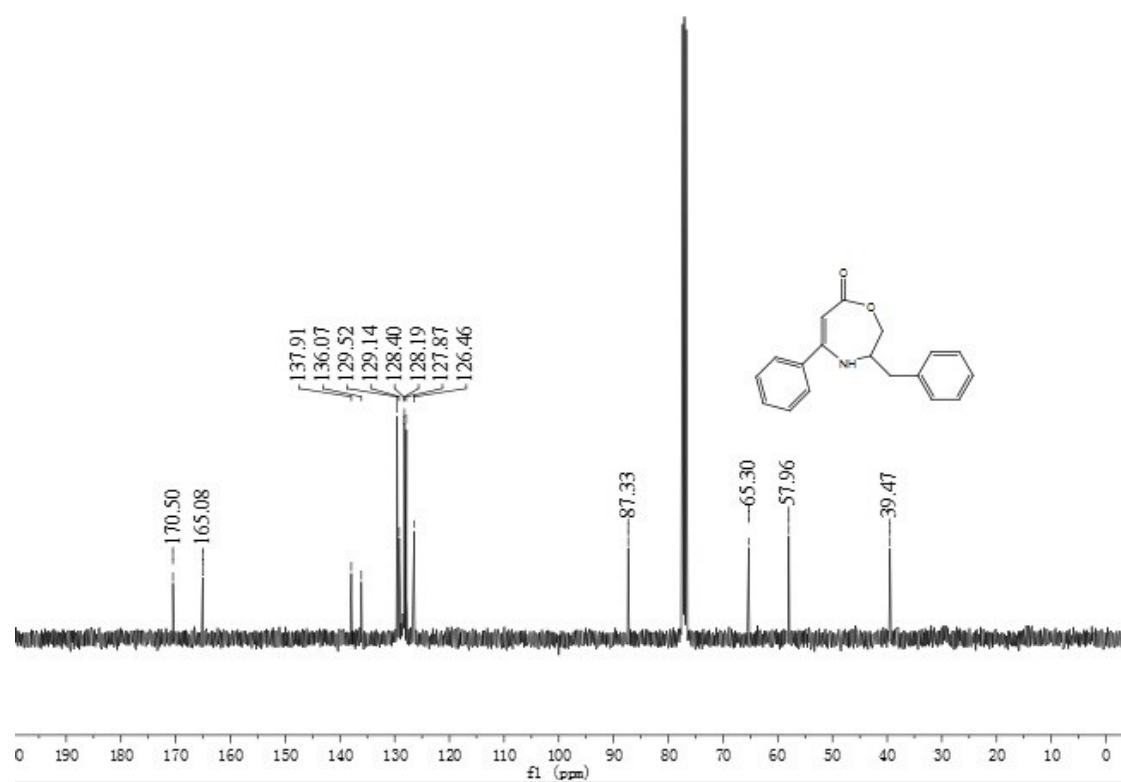
¹³CNMR of **3h**



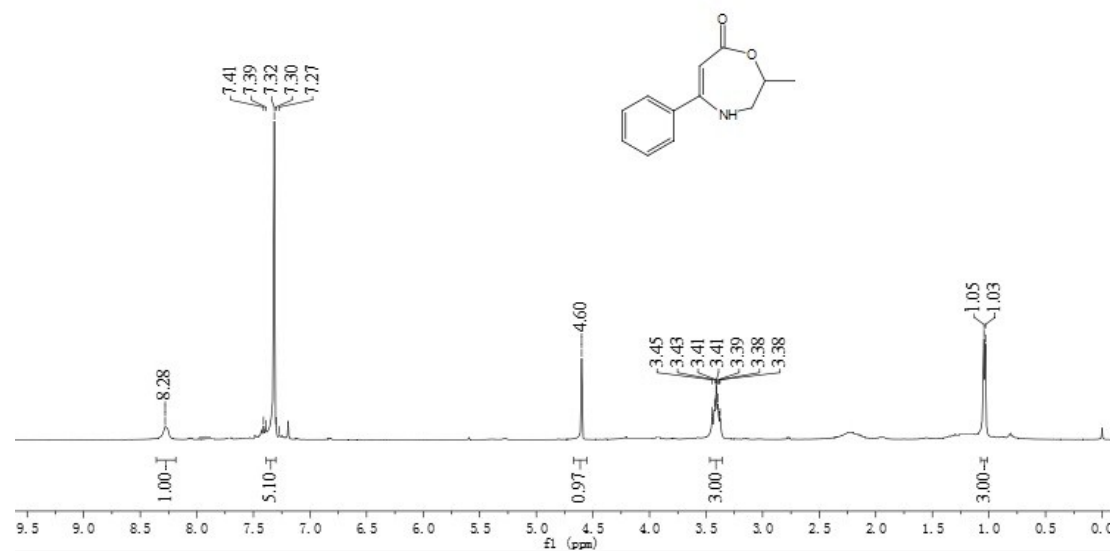
¹H NMR of **3i**



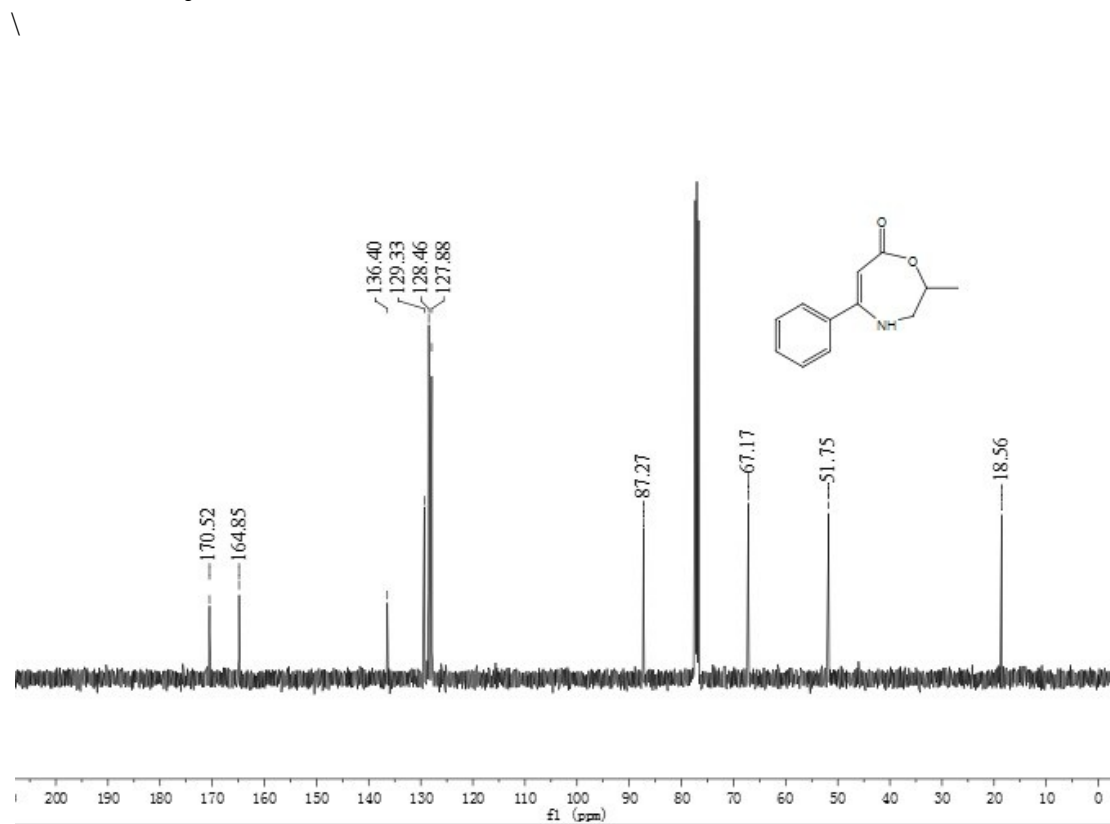
¹³CNMR of **3i**



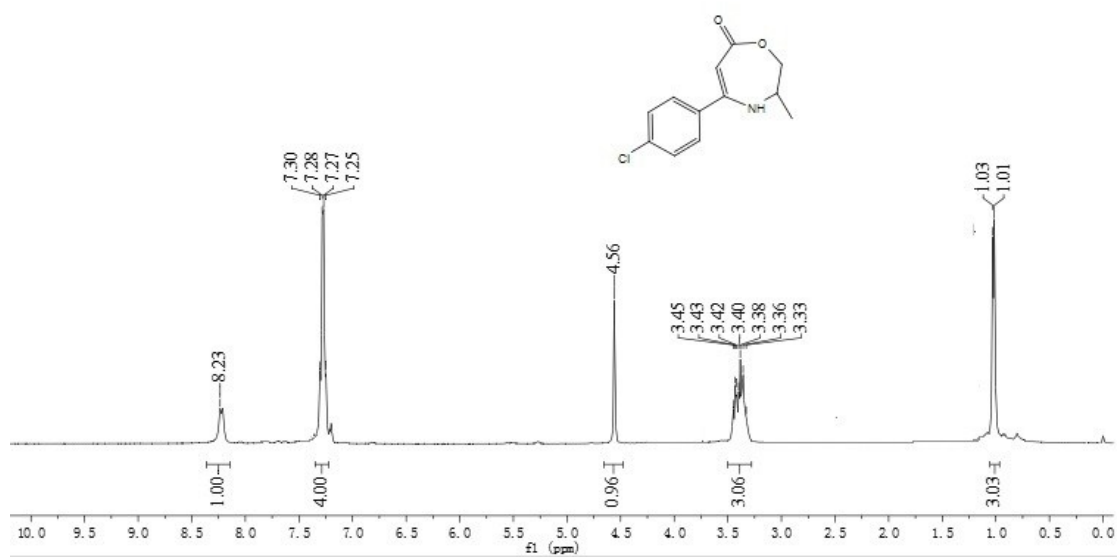
¹H NMR of **3j**



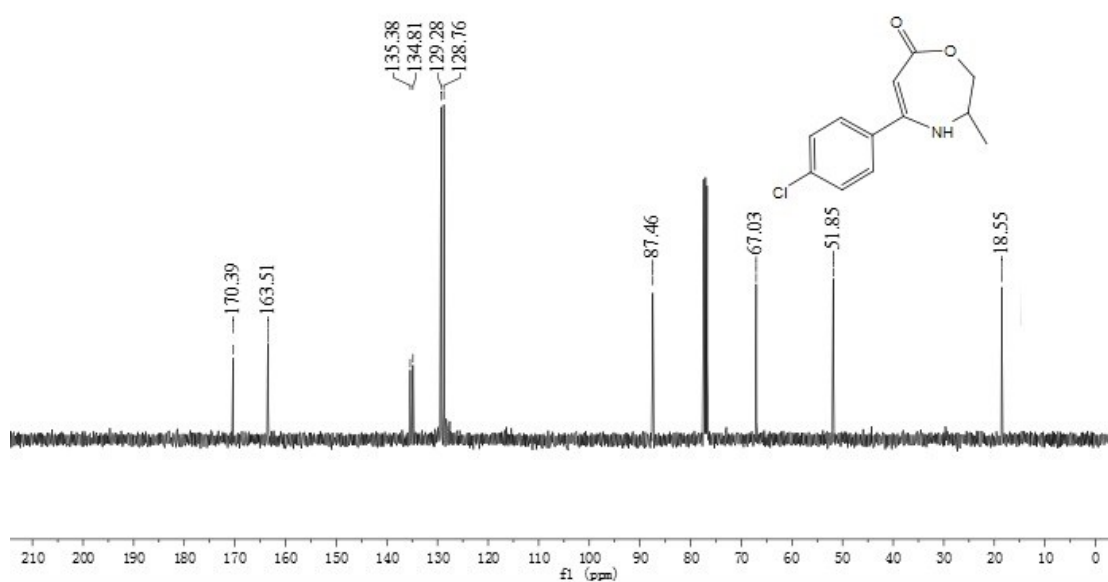
¹³CNMR of **3j**



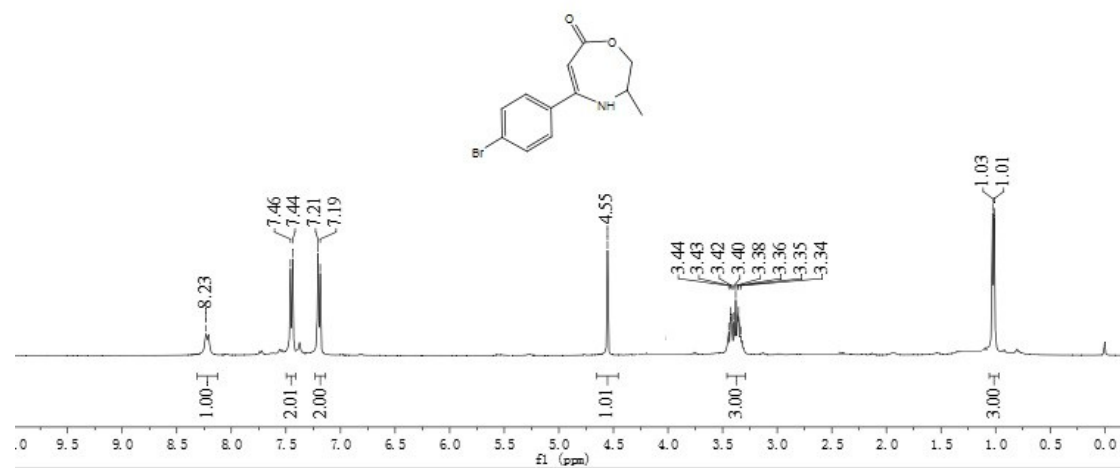
¹H NMR of **3k**



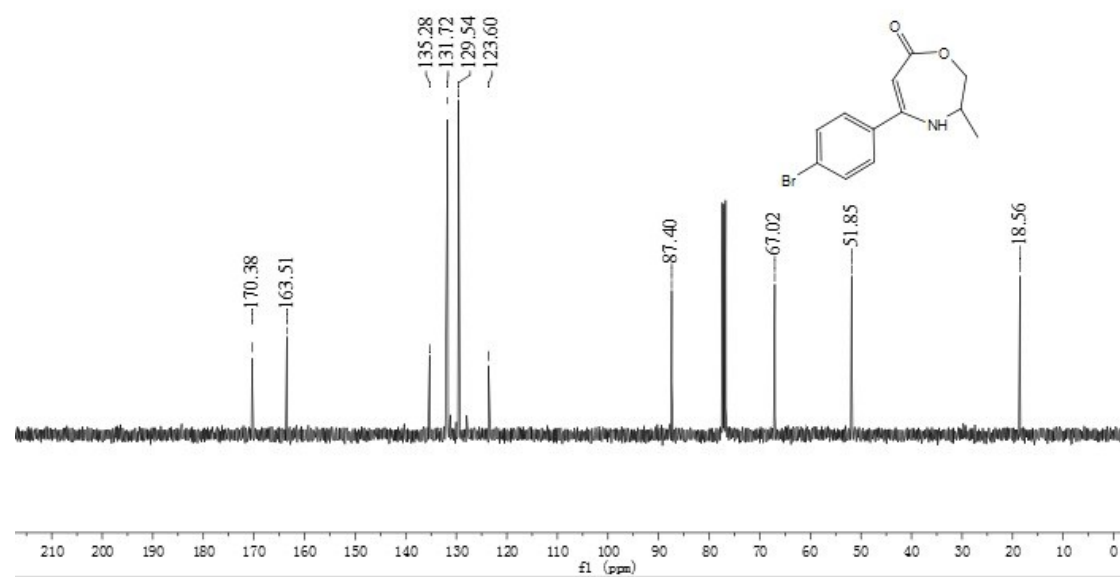
¹³CNMR of 3k



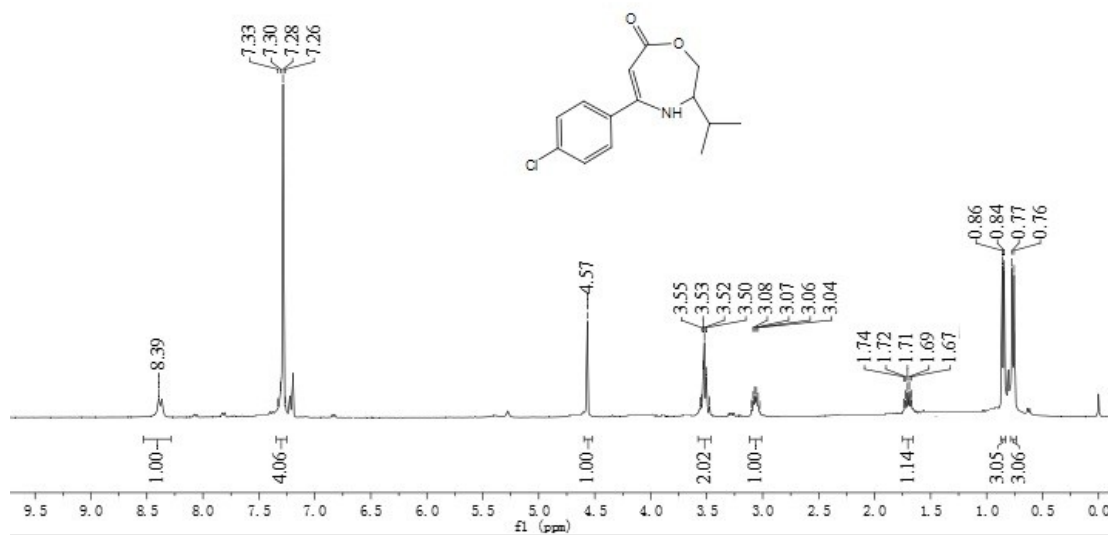
^1H NMR of **31**



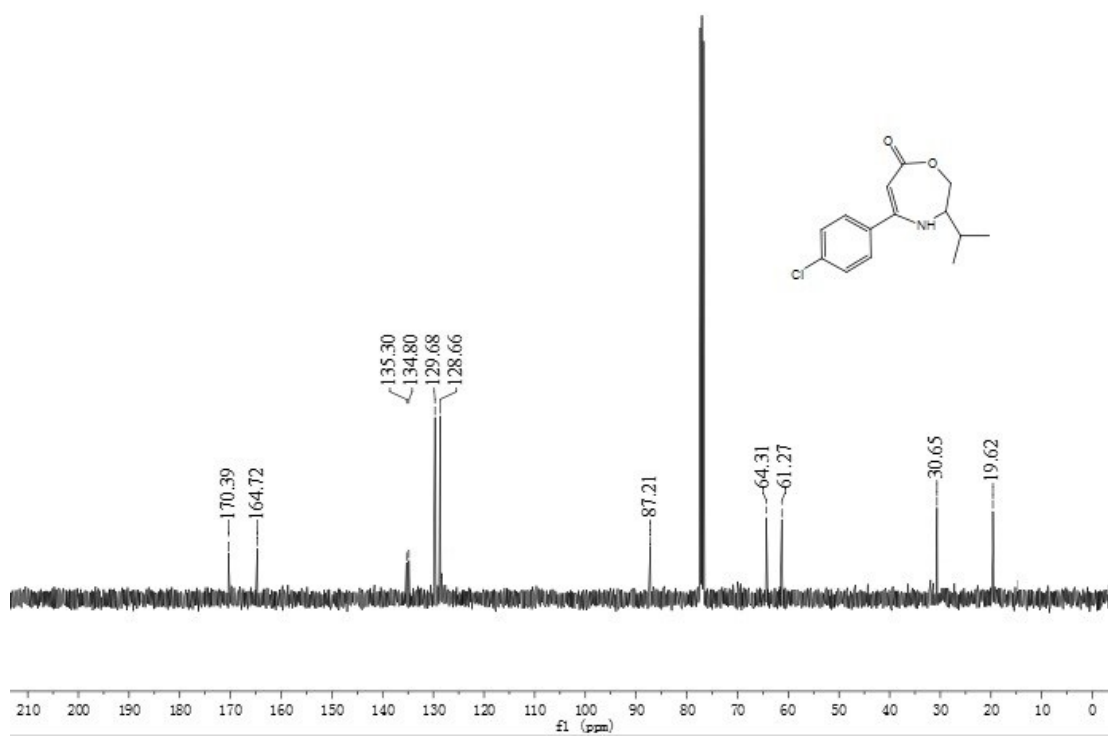
^{13}C NMR of **31**



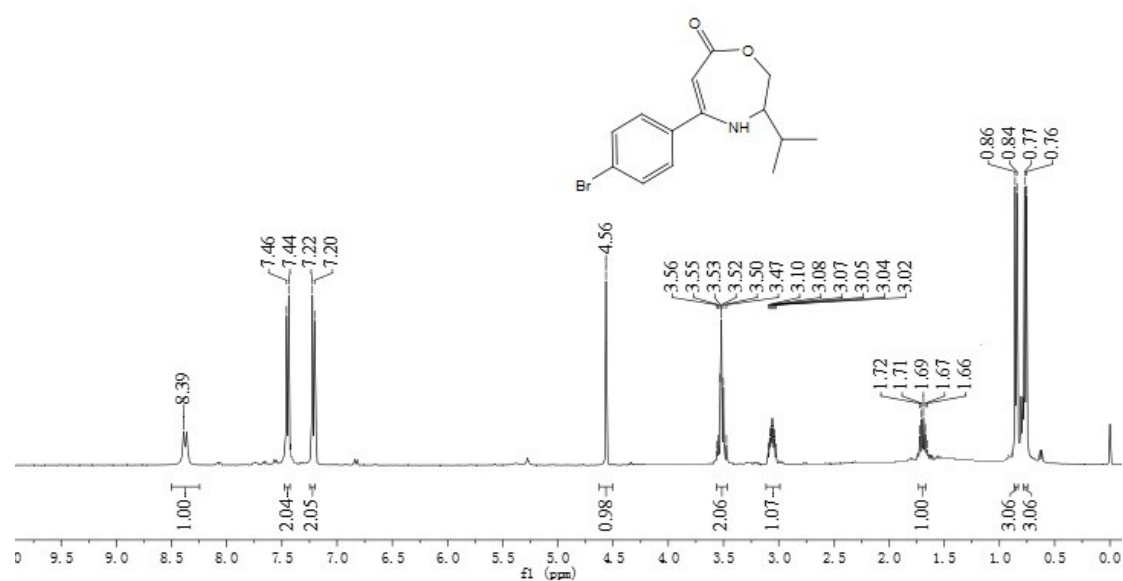
^1H NMR of **3m**



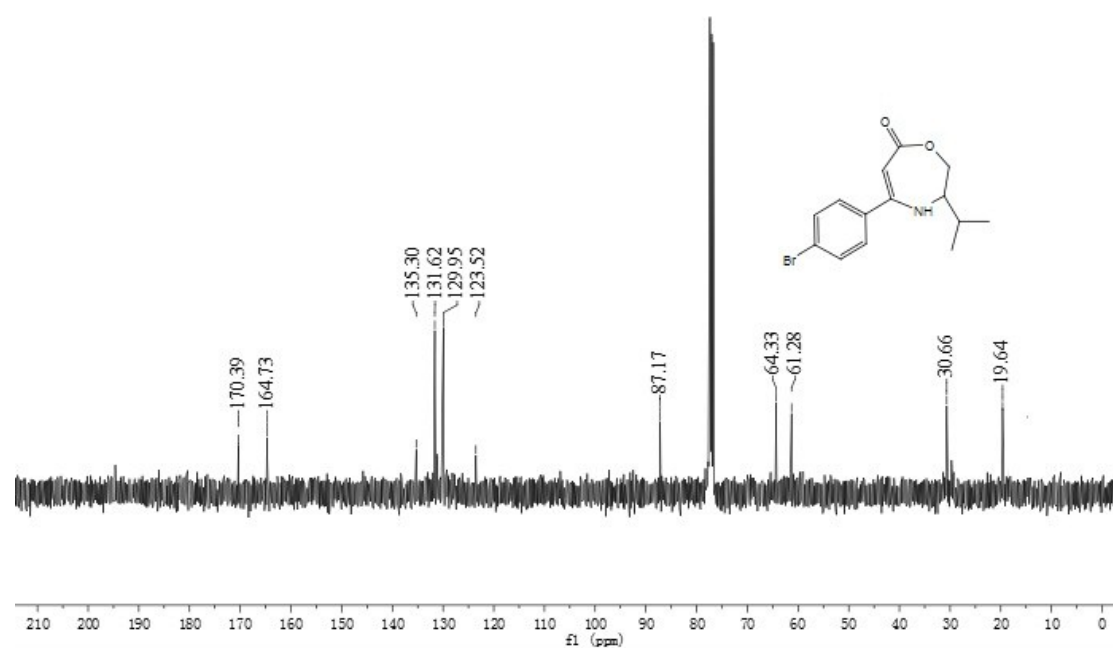
^{13}C NMR of **3m**



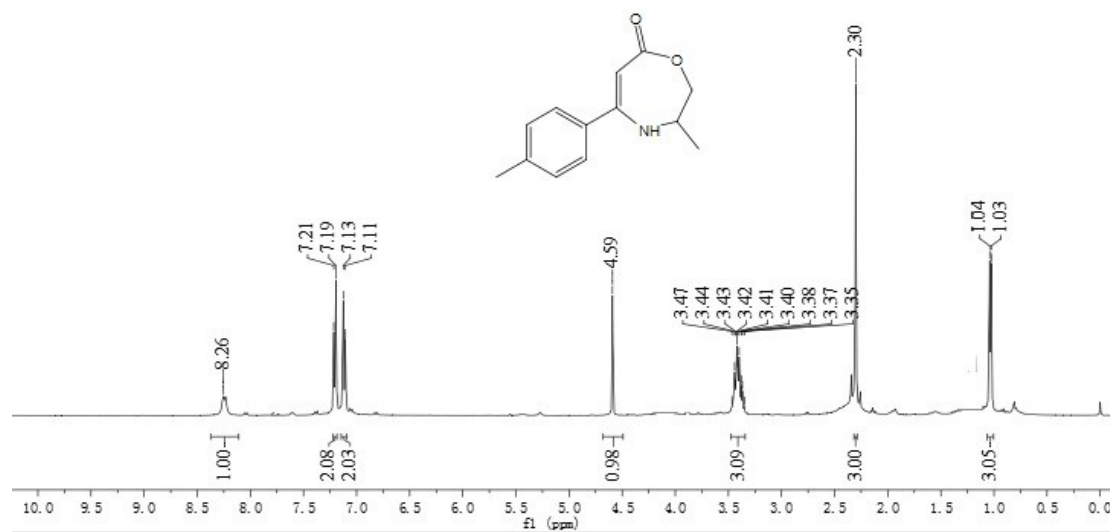
^1H NMR of **3n**



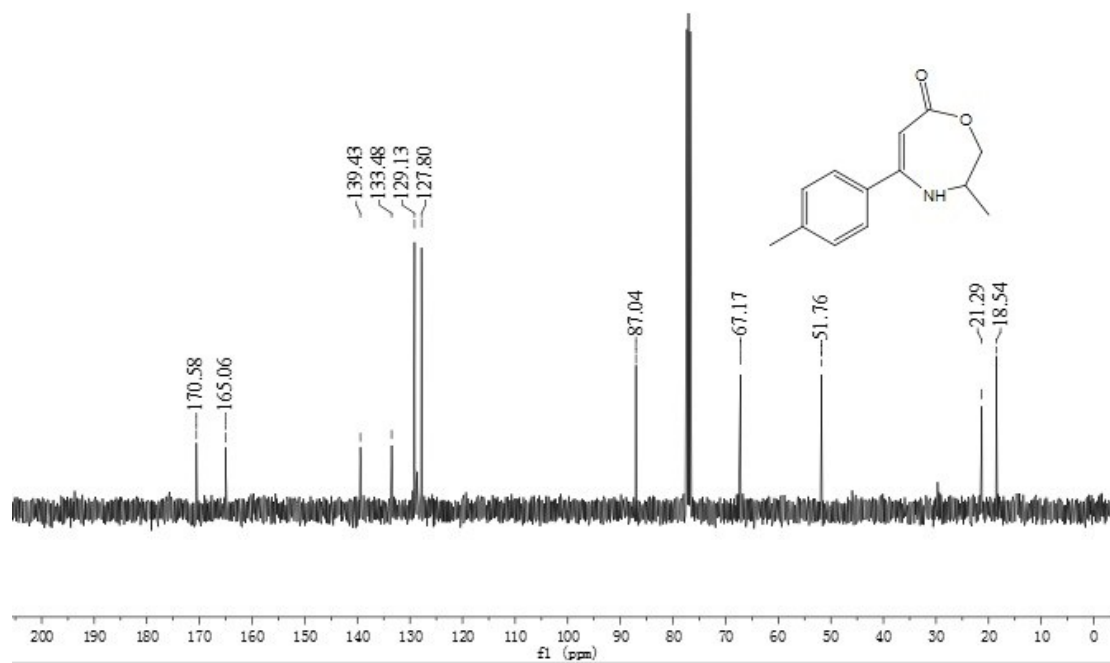
¹³CNMR of **3n**



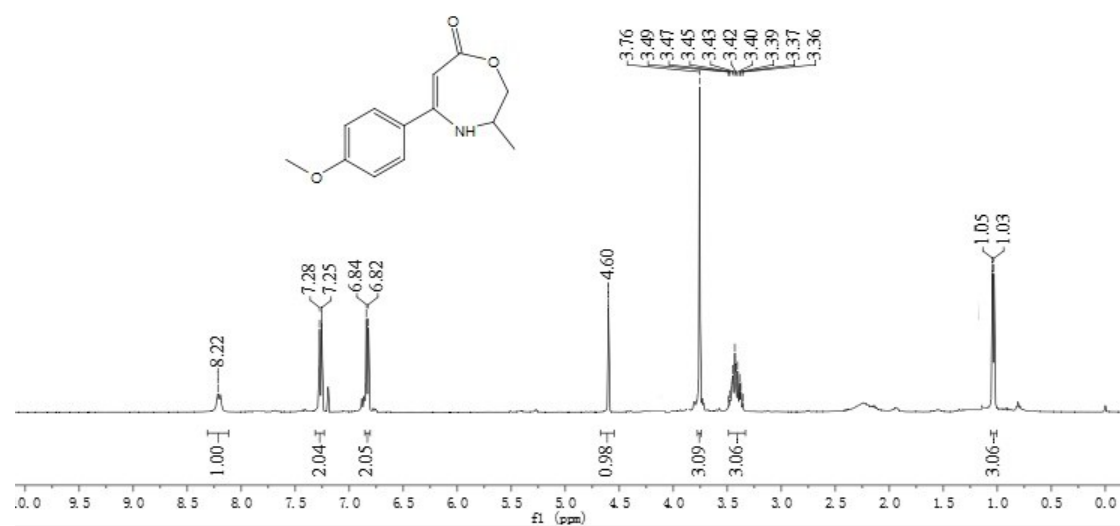
¹H NMR of **3o**



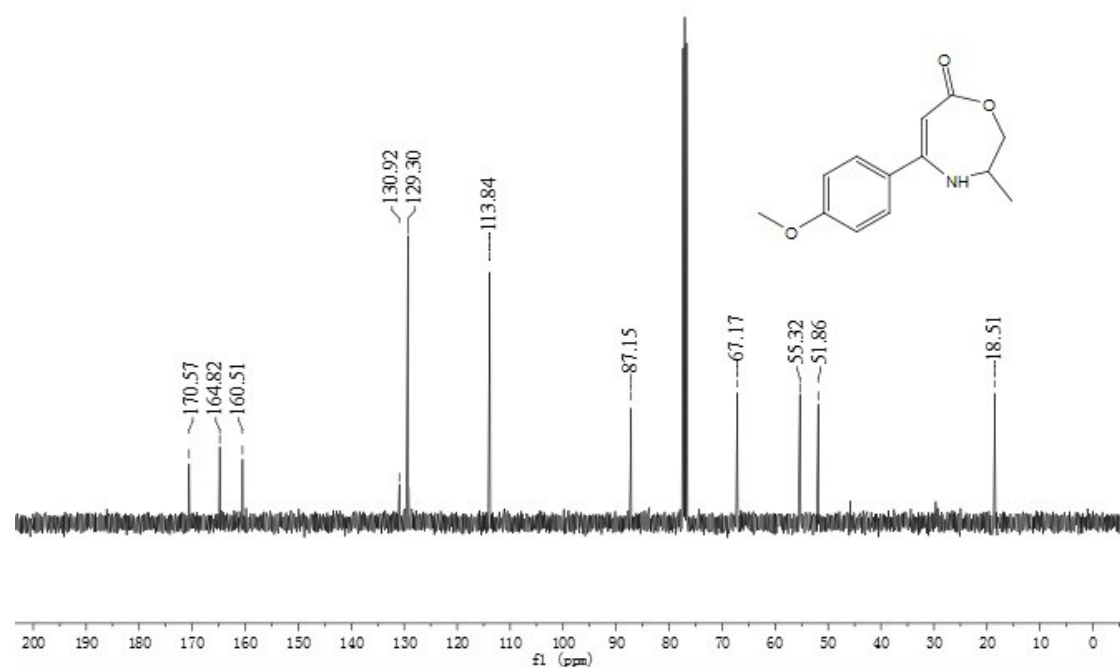
¹³C NMR of **3o**



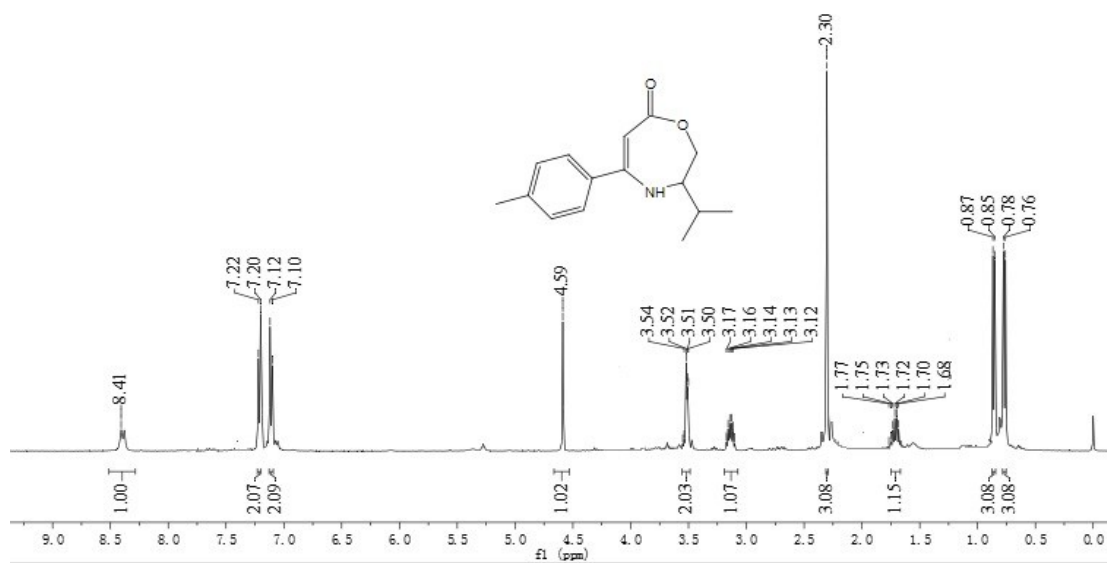
^1H NMR of **3p**



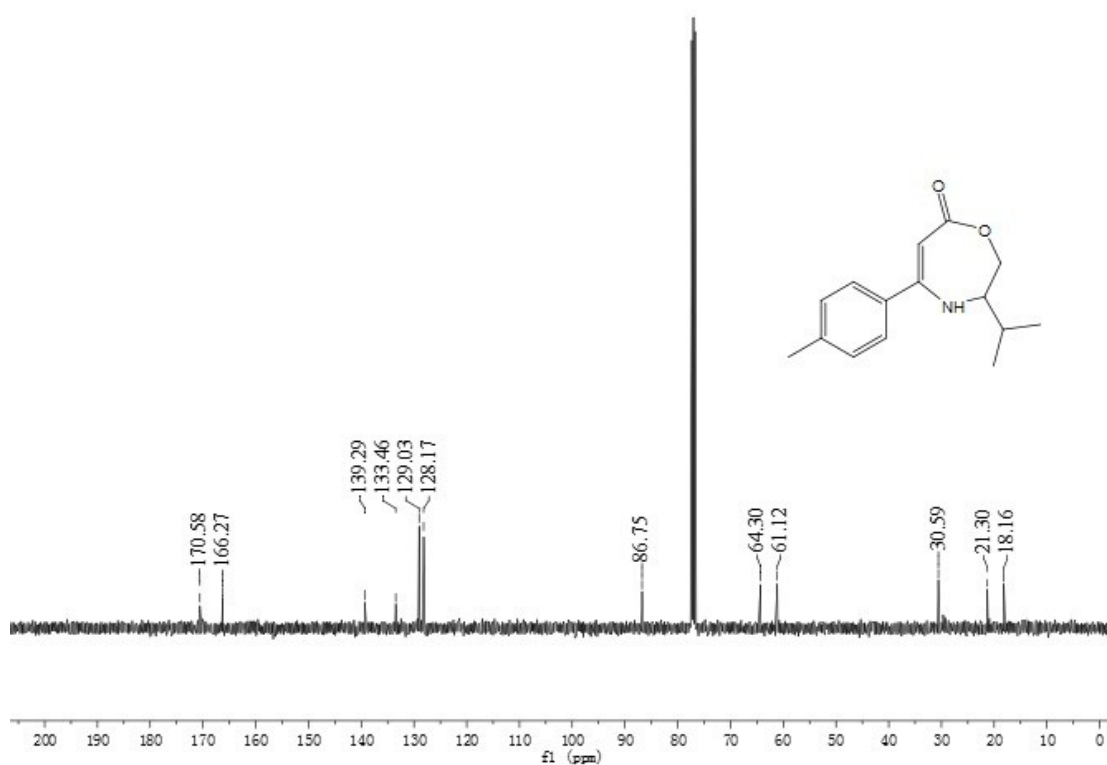
^{13}C NMR of **3p**



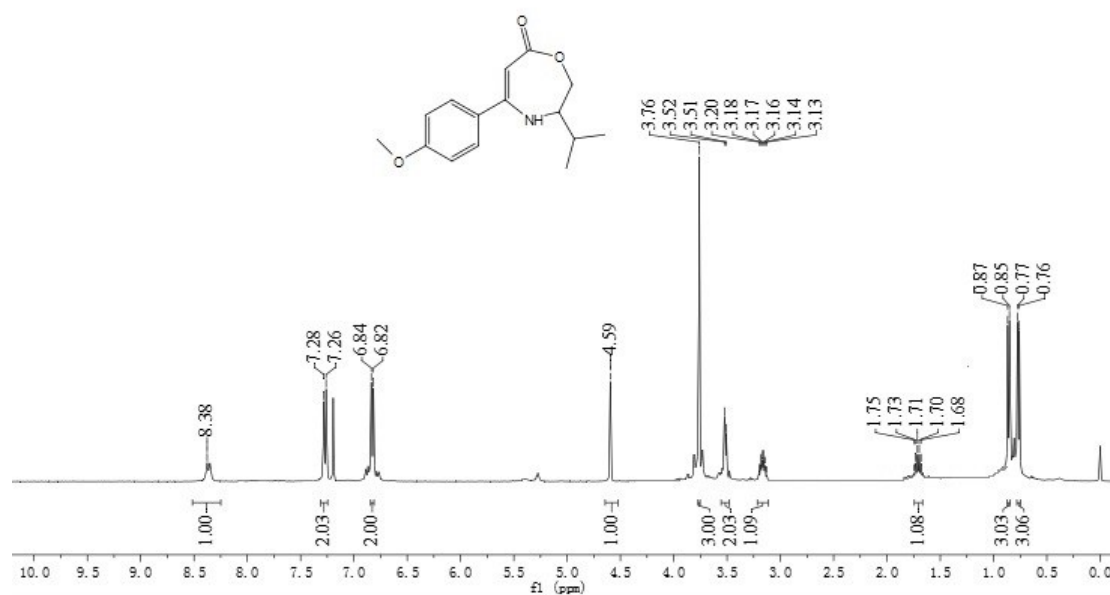
^1H NMR of **3q**



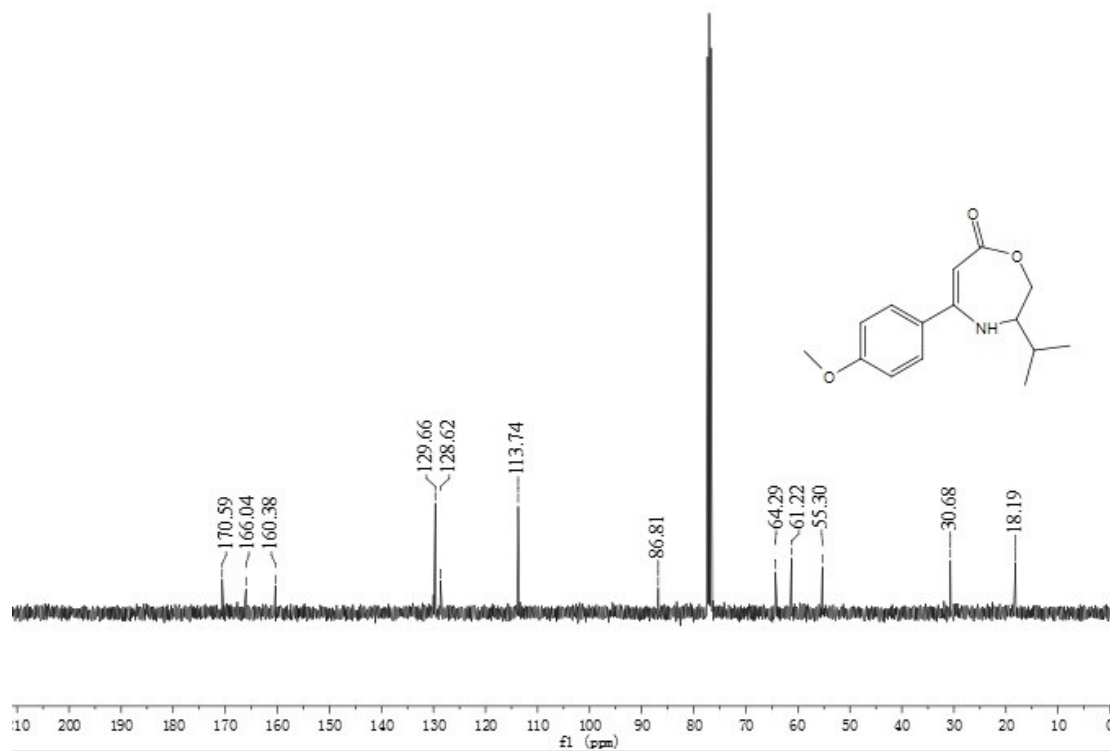
¹³CNMR of **3q**



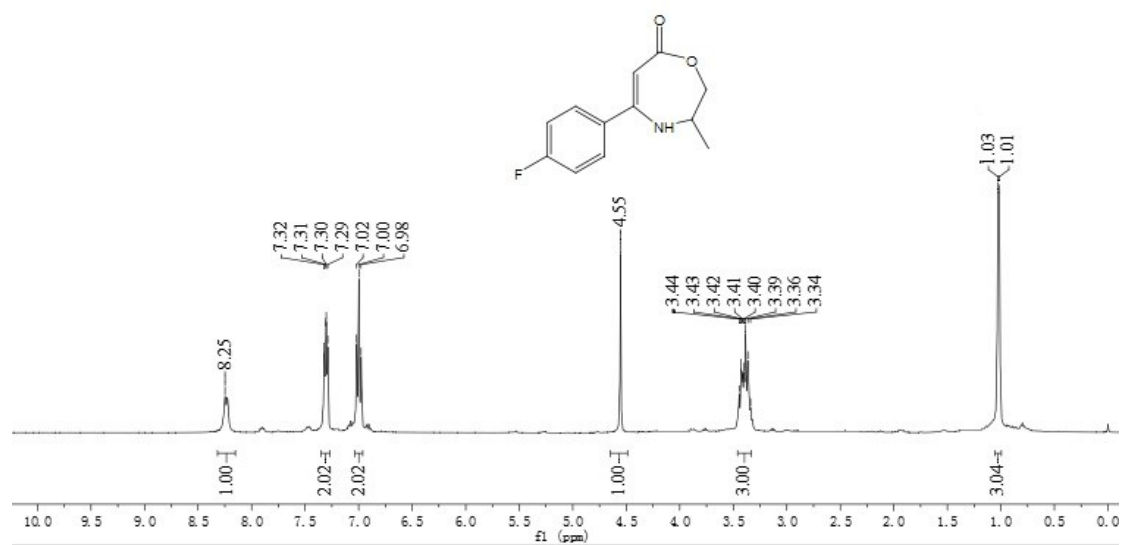
¹H NMR of **3r**



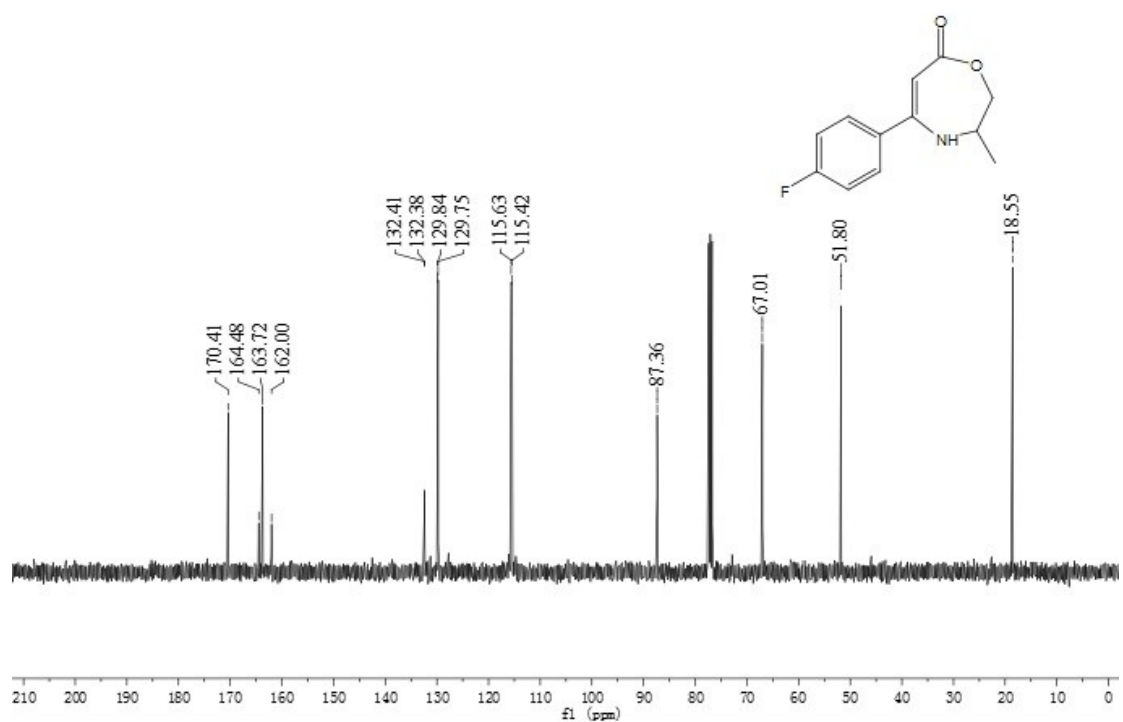
¹³C NMR of **3r**



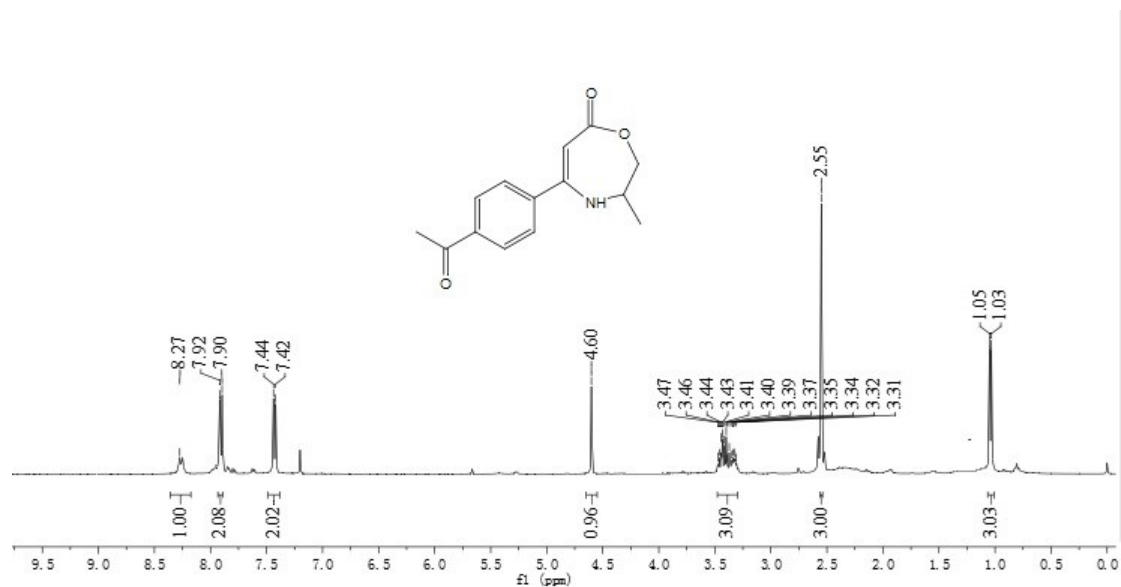
¹H NMR of **3s**



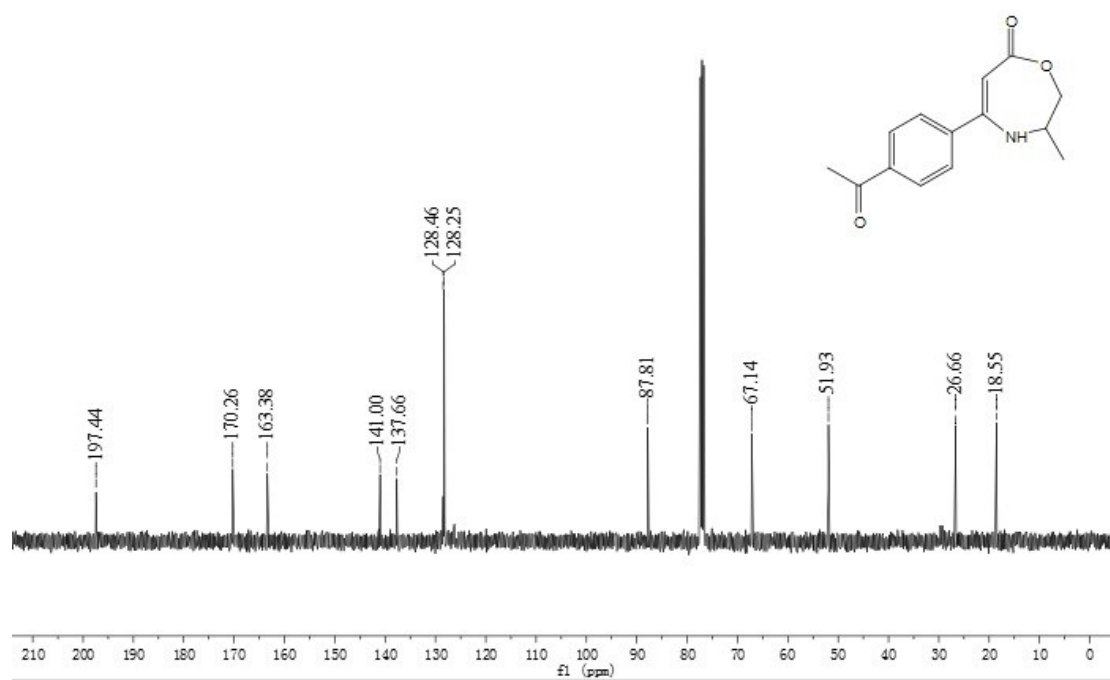
¹³C NMR of **3s**



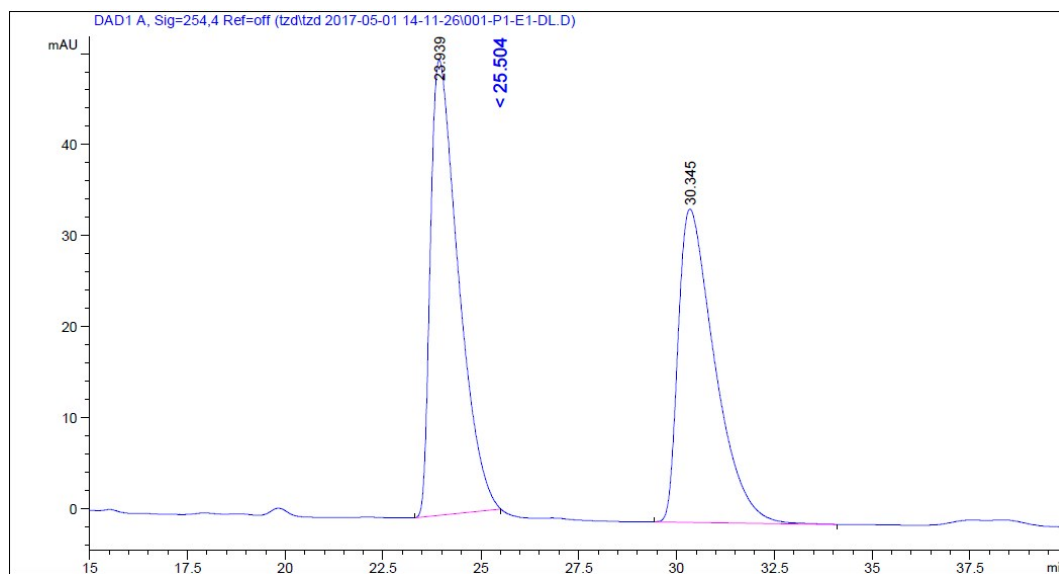
^1H NMR of **3t**



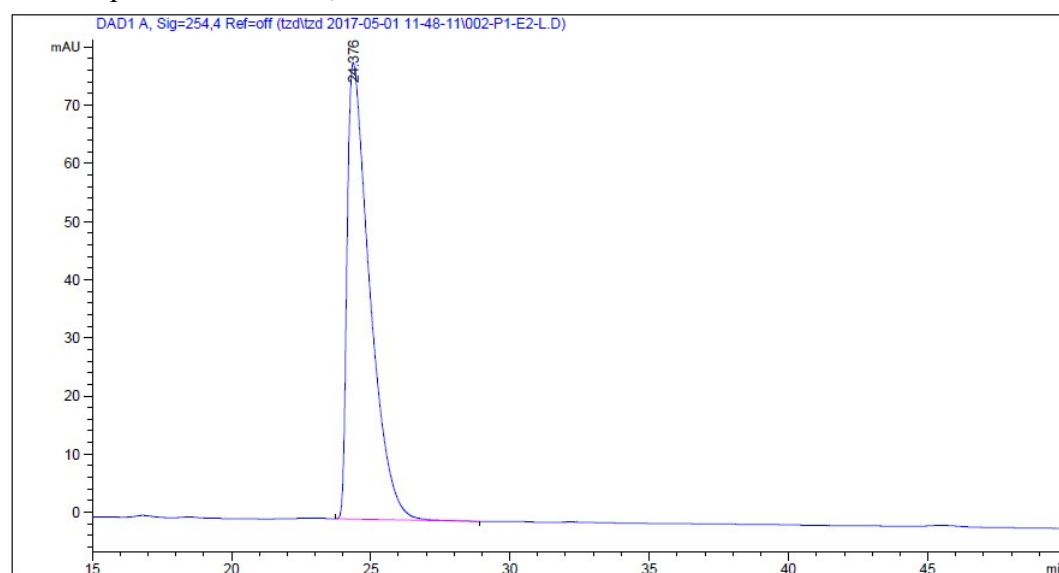
^{13}C NMR of **3t**



HPLC spectra for **3a**



HPLC spectra for enantiopure enam inolactone (S)-(+)-**3a'**



1. D. Fang, J. Luo, X. L. Zhou and Z. L. Liu, *Catal. Lett.*, 2007, **116**, 76-80.