Supporting Information

Lactate-Based Ionic Liquid Catalyzed Reductive Amination/Cyclization of Keto Acids under Mild Conditions: A Metal-Free Route to Synthesize Lactams

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1. General Information

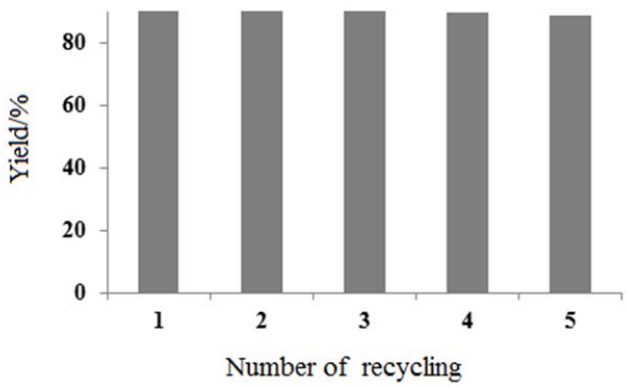
Liquid NMR spectra were recorded on Bruck 400 spectrometer. The composition of the reaction mixture was analyzed by means of GC (Agilent 4890D) with a FID detector and a non polar capillary column (DB - 5) (30 m × 0.25 mm × 0.25 μm). The column oven was temperature programmed with a 2 min initial hold at 323 K, followed by the temperature increase to 528K at a rate of 15 K/min and kept at 538 K for 10 min. High purity nitrogen was used as a carrier gas. The ionic liquids, [DBU][Lac], [tBu_4P][Lac], [Et_4N][Lac] were synthesized via the neutralization of corresponding bases and proton donors according to the reported procedures.[1, 2] and the others were all supplied by Lanzhou Institute of Chemical Physics CAS.

2. Typical procedure for the synthesis of lactams

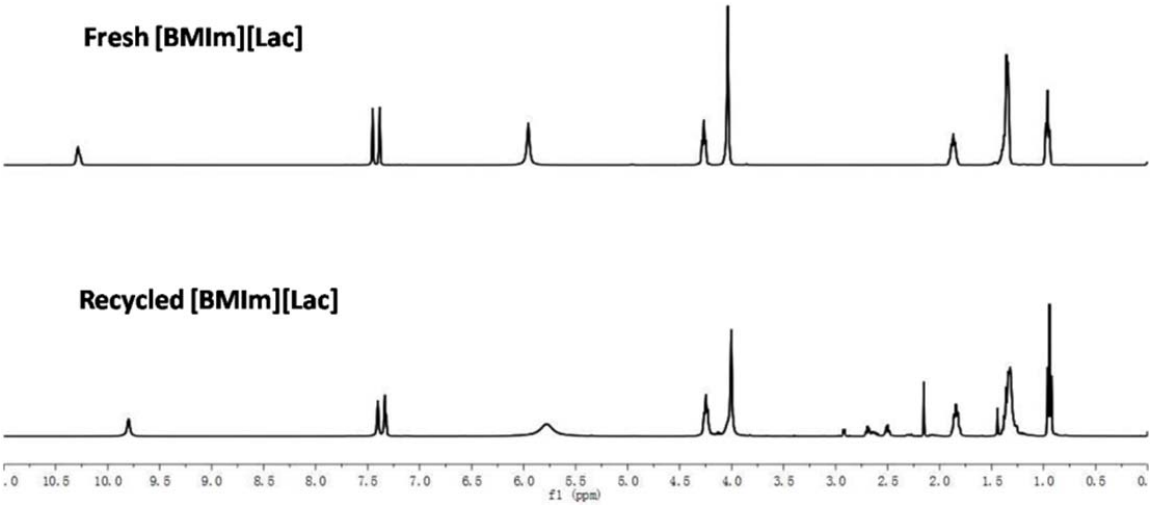
The reductive amination/cyclization of keto acid in the presence of hydrosilane was conducted in a screw-capped vial (15 mL inner volume) equipped with a magnetic stirrer. In a typical experiment to synthesize 5-methyl-1-phenylpyrrolidin-2-one, 1-butyl-3-methylimidazolium lactate [BMIm][Lac] (0.2 mmol), aniline (1 mmol), keto acid (1.0 mmol), $(EtO)_3SiH$ (2 mmol) were added into the reactor successively. Subsequently, the reactor was heated at 80 ^oC for 1 h. And then the reactor was moved to water and cooled to room temperature. Subsequently, the reaction mixture was extracted with diethyl ether for three times to separate the product from the IL. The IL was left in the reactor to be reused for the next run, and the reaction mixture in diethyl ether phase was analyzed by GC using FID to determine the product yield.

Similarly, the other N-substituted lactams were synthesized using the corresponding substituted amines as the substrates at the optimal reaction condition for certain time. The corresponding N-substituted lactams yields were determined by ^1H NMR using mesitylene as an internal standard. And then the crude mixture was extracted by ether for three times, then isolated by column chromatography on silica gel (eluent: petroleum ether and EtOAc with 3-5% triethylamine), and identified by NMR spectra for substrate scope screening.

4. The recyclability and characterization of the catalyst [BMIm][Lac]



(a)



(b)

Figure S1. (a) Recycling of [BMIm][Lac] for the reductive amination of levulinic acid. Reaction conditions: IL (0.2 mmol), aniline (1 mmol), LA (1 mmol), $(EtO)_3SiH$ (2 mmol), 80 ^oC, 1 h. **(b)** ^1H NMR spectra of fresh and recycled [BMIm][Lac], ($CDCl_3$, 298 K).

5. ^{1}H NMR and ^{13}C NMR spectra of $(EtO)_3SiH$, [BMIm][Lac] and their mixture

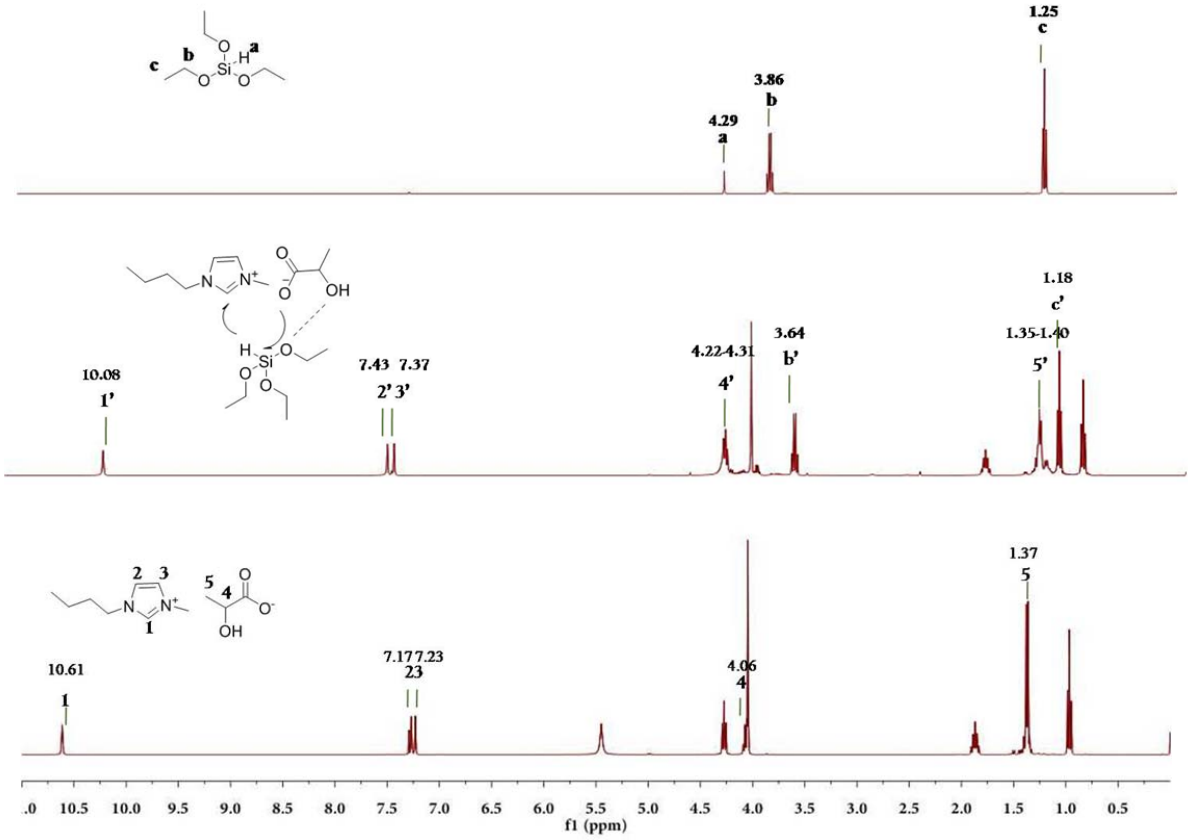


Figure S2. ^{1}H NMR spectra of pure $(EtO)_3SiH$, the mixture of [BMIm][Lac] (0.1 mmol) and $(EtO)_3SiH$ (0.1 mmol) and pure [BMIm][Lac]; ($CDCl_3$, 298 K).

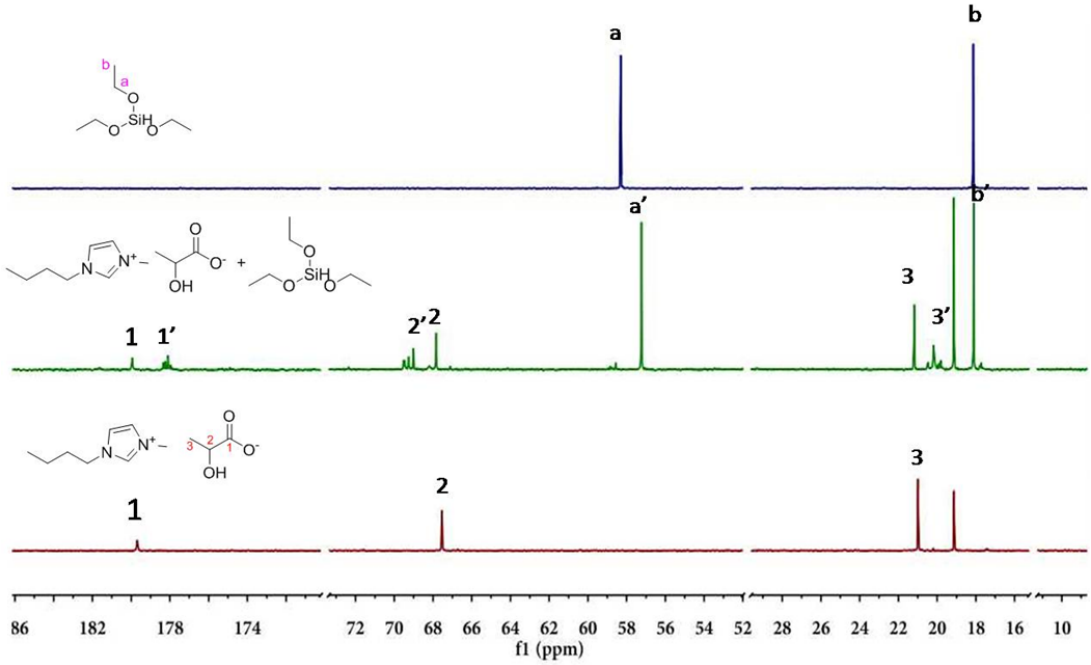


Figure S3. ^{13}C NMR spectra of pure $(EtO)_3SiH$, the mixture of [BMIm][Lac] (0.1 mmol) and $(EtO)_3SiH$ (0.1 mmol) and pure [BMIm][Lac]; ($CDCl_3$, 298 K).

6. ^{1}H NMR spectra of $PhNH_2$, [BMIm][Lac] and their mixture

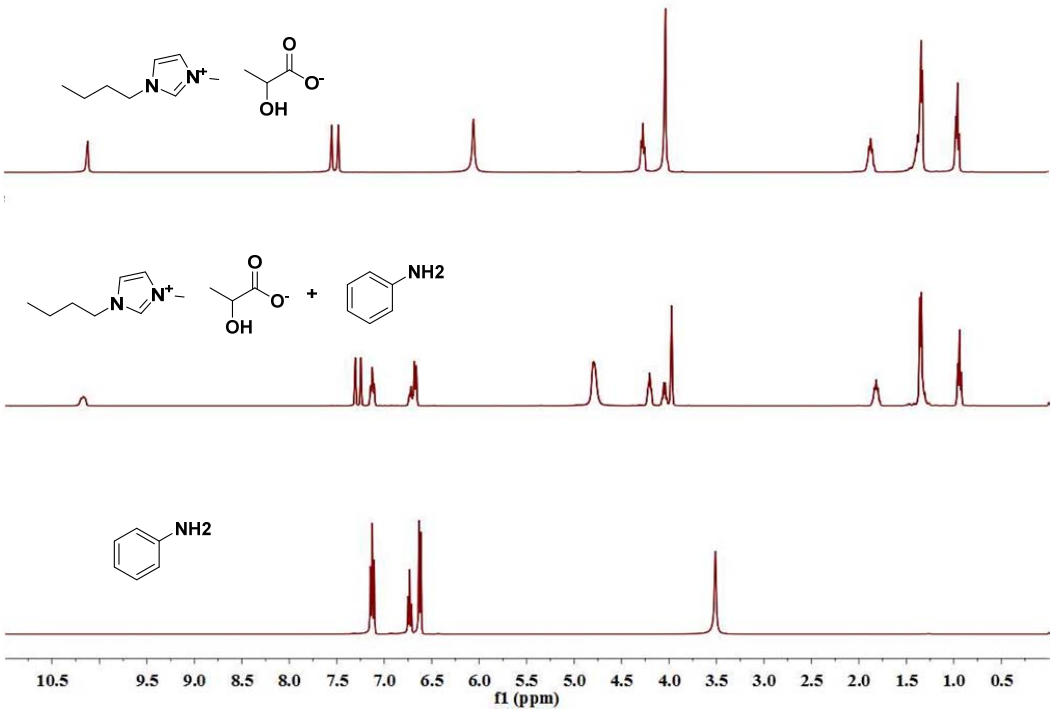


Figure S4. ^{1}H NMR spectra of pure [BMIm][Lac], the mixture of [BMIm][Lac] (0.1 mmol) and $PhNH_2$ (0.1 mmol) and pure $PhNH_2$; ($CDCl_3$, 298 K).

7. ^{1}H NMR spectra of LA, [BMIm][Lac] and their mixture

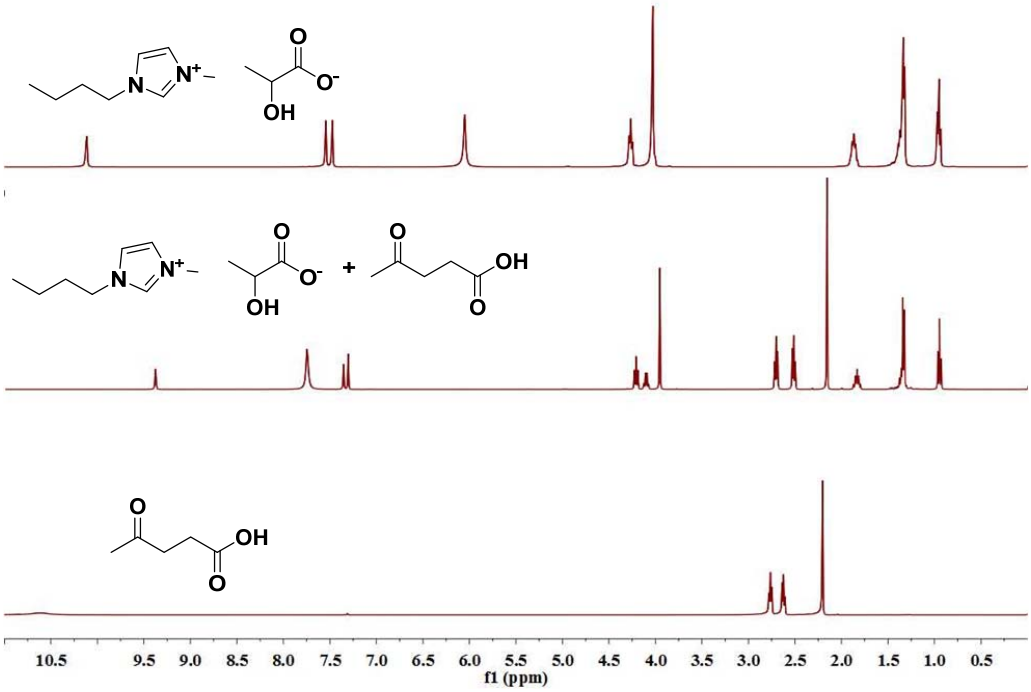
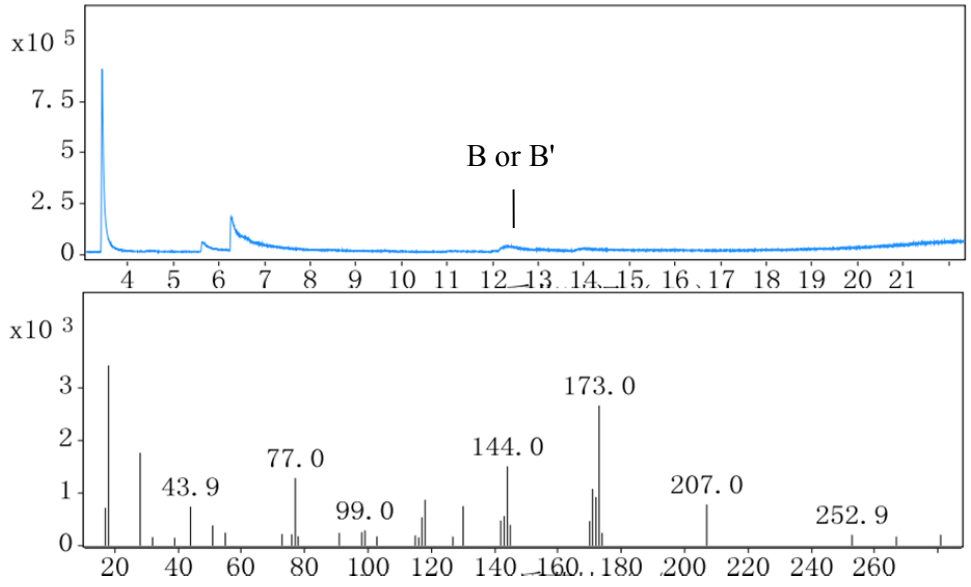
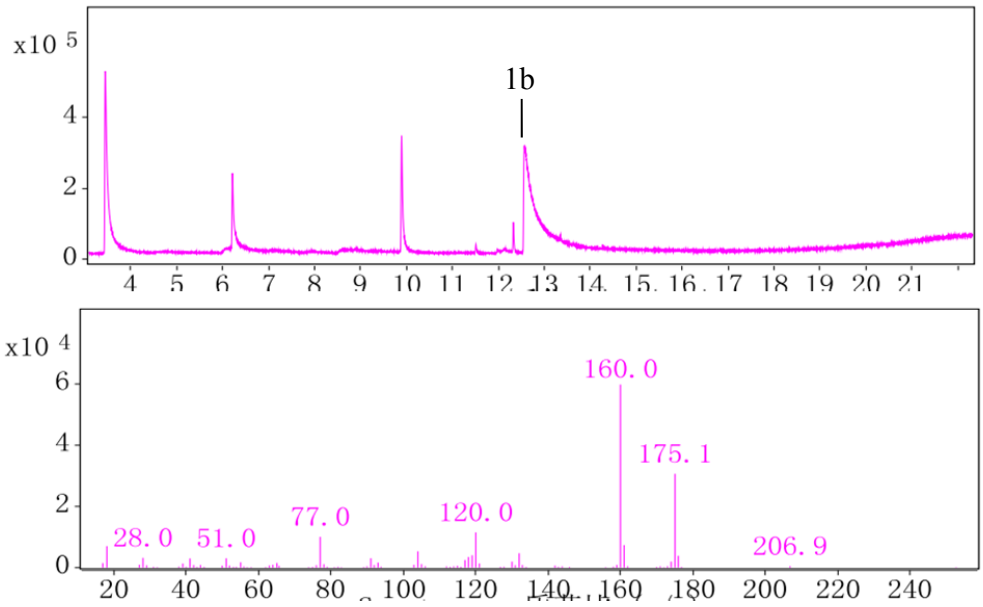


Figure S5. ^{1}H NMR spectra of pure [BMIm][Lac], the mixture of [BMIm][Lac] (0.1 mmol) and LA (0.1 mmol) and pure LA; ($CDCl_3$, 298 K).

8. GC-MS spectra of the reaction solutions of the control experiments..



(a)



(b)

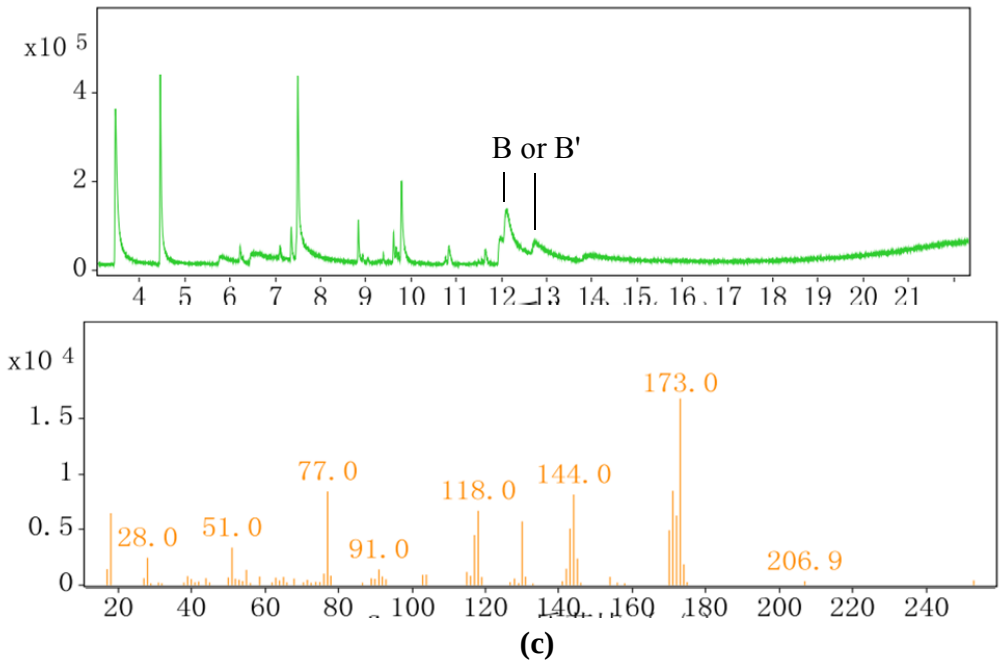
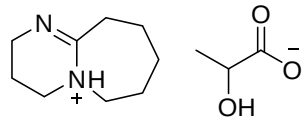


Figure S6. GC-MS spectra of the reaction solutions of the control experiments as illustrated in Scheme 3.**(a)** The reaction solution of aniline(1.0 mmol), LA (1.0 mmol), [BMIm][Lac] (0.2 mmol) at 80 °C for 1 h. **(b)** Adding $(EtO)_3SiH$ to the mixture of (a). **(c)** The reaction solution of aniline (1.0 mmol), LA (1.0 mmol), $(EtO)_3SiH$ (2 mmol) at 80 °C for 1 h. **(d)** Adding [BMIm][Lac] (0.2 mmol) to the mixture of (c).

9. NMR data and spectra of the as-synthesized lactate-based ILs.

1,8-Diazabicyclo[5.4.0]undec-7-ene lactate ([DBU][Lac])



^{1}H NMR (400 MHz, $CDCl_3$) δ 4.02 (q, *J* = 6.8 Hz, 1H), 3.58 – 3.42 (m, 6H), 2.14 – 1.95 (m, 2H), 1.84 – 1.61 (m, 6H), 1.37 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.47, 165.13, 67.09, 53.31, 47.68, 37.18, 31.17, 28.08, 25.91, 23.18, 20.64, 18.69 ppm.

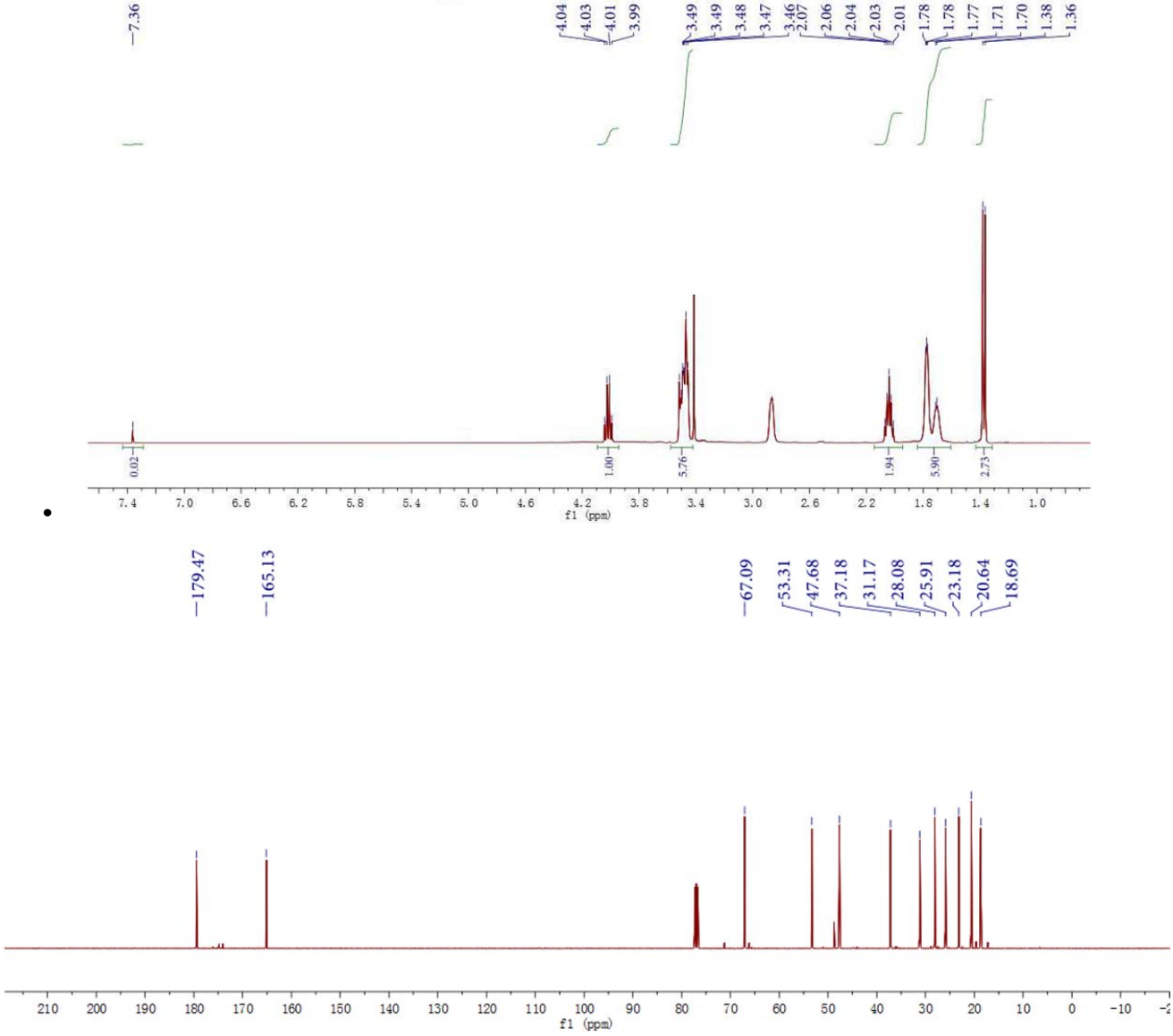
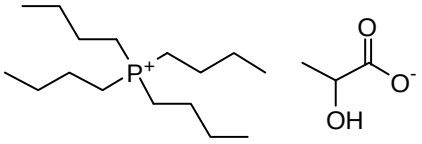
- 

Figure S7. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1,8-diazabicyclo[5.4.0]undec-7-ene lactate

Tetrabutylphosphonium lactate



^{1}H NMR (400 MHz, $CDCl_3$) δ 3.93 (q, *J* = 6.7 Hz, 1H), 2.35 (ddd, *J* = 13.1, 9.8, 6.6 Hz, 8H), 1.63 – 1.46 (m, 16H), 1.35 (d, *J* = 6.7 Hz, 3H), 0.98 (t, *J* = 6.9 Hz, 12H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.25, 68.08, 23.78 (dd, *J* = 22.9, 10.0 Hz), 21.64, 18.82, 18.35, 13.35 ppm.

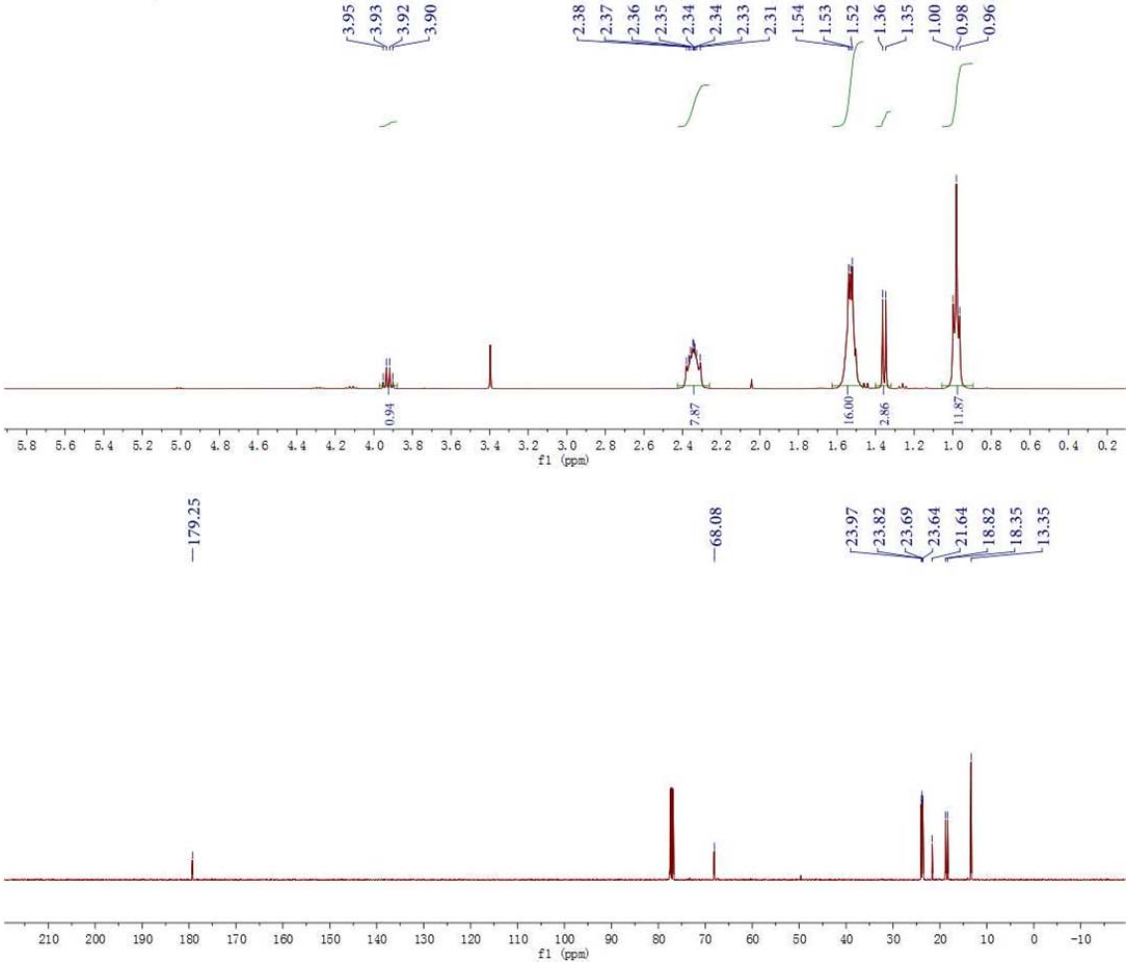
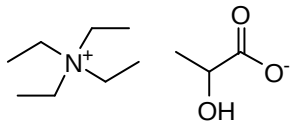


Figure S8. ^{1}H (top) and ^{13}C (bottom) NMR spectra of tetrabutylphosphonium lactate

Tetraethylammonium lactate



^{1}H NMR (400 MHz, $CDCl_3$) δ 3.89 (q, *J* = 6.7 Hz, 1H), 3.38 (q, *J* = 7.3 Hz, 8H), 1.35 (ddd, *J* = 14.1, 7.9, 4.2 Hz, 15H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.25, 68.00, 52.55 – 52.24 (m), 21.59, 7.46 ppm.

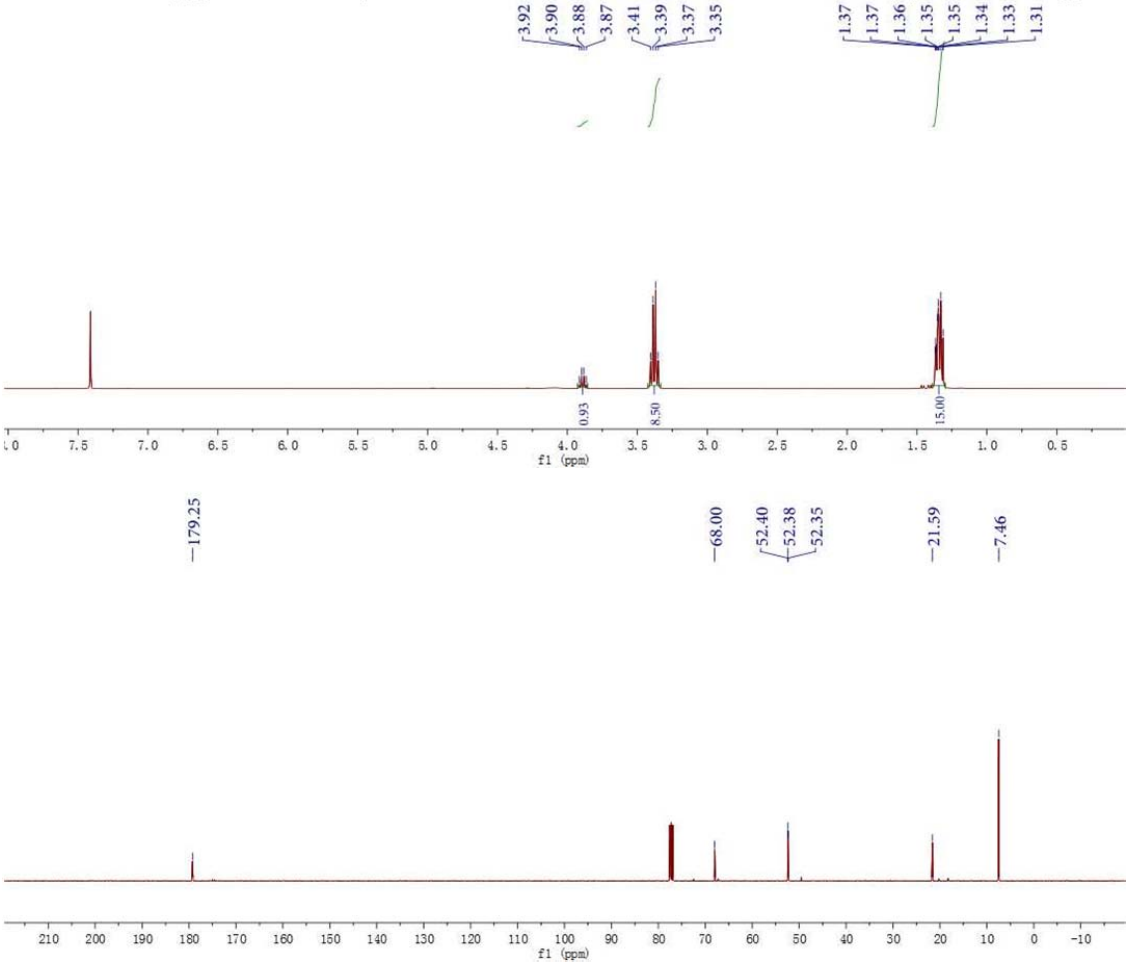
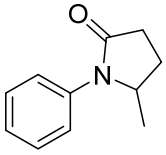


Figure S9. ^{1}H (top) and ^{13}C (bottom) NMR spectra of tetraethylammonium lactate

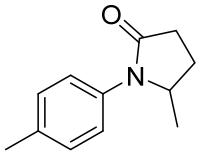
10. NMR data and spectra of the N-substituted lactam products

5-Methyl-1-phenylpyrrolidin-2-one



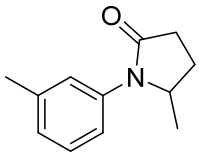
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.31 (m, 4H), 7.20 (dd, *J* = 6.7, 4.7 Hz, 1H), 4.42 – 4.18 (m, 1H), 2.75 – 2.26 (m, 3H), 1.83 – 1.66 (m, 1H), 1.20 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.18, 137.63, 128.97, 125.72, 124.04, 55.61, 31.35, 26.75, 20.16 ppm.

5-Methyl-1-(p-tolyl)pyrrolidin-2-one



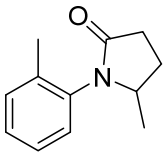
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (q, *J* = 8.4 Hz, 4H), 4.29 – 4.17 (m, 1H), 2.67 – 2.45 (m, 2H), 2.32 (s, 4H), 1.72 (dtd, *J* = 13.0, 9.4, 7.3 Hz, 1H), 1.17 (d, *J* = 6.2 Hz, 3H) ppm.^{13}C NMR (101 MHz, $CDCl_3$) δ 174.14, 135.55, 134.89, 129.52, 129.14, 124.17, 55.71, 31.19, 26.73, 20.89, 20.12 ppm.

5-Methyl-1-(m-tolyl)pyrrolidin-2-one



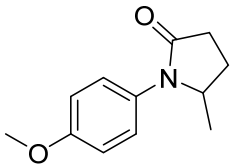
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 15.8, 8.0 Hz, 1H), 7.18 (s, 1H), 7.12 (d, *J* = 8.0 Hz, 1H), 7.02 (d, *J* = 7.5 Hz, 1H), 4.32 – 4.20 (m, 1H), 2.68 – 2.46 (m, 2H), 2.41 – 2.30 (m, 4H), 1.74 (dddd, *J* = 12.8, 9.5, 7.2, 5.9 Hz, 1H), 1.19 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.20, 138.81, 137.48, 128.75, 126.72, 125.10, 121.30, 55.81, 31.32, 26.76, 21.44, 20.19 ppm.

5-Methyl-1-(o-tolyl)pyrrolidin-2-one



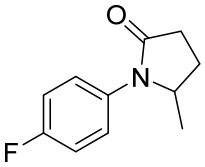
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (dt, *J* = 22.4, 4.6 Hz, 3H), 7.12 – 7.00 (m, 1H), 4.06 (d, *J* = 5.7 Hz, 1H), 2.68 – 2.32 (m, 3H), 2.22 (s, 3H), 1.86 – 1.70 (m, 1H), 1.11 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.19, 136.43, 136.15, 131.14, 127.81, 126.67, 56.87, 30.88, 27.85, 20.29, 18.08 ppm.

1-(4-Methoxyphenyl)-5-methylpyrrolidin-2-one



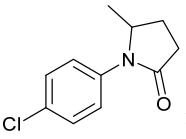
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.19 (m, 2H), 6.94 – 6.88 (m, 2H), 4.23 – 4.11 (m, 1H), 3.80 (s, 3H), 2.67 – 2.45 (m, 2H), 2.35 (dddd, *J* = 13.3, 9.3, 7.3, 6.1 Hz, 1H), 1.79 – 1.66 (m, 1H), 1.17 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.31, 157.72, 130.43, 126.10, 114.37, 56.14, 55.45, 31.13, 26.86, 20.27 ppm.

1-(4-Fluorophenyl)-5-methylpyrrolidin-2-one



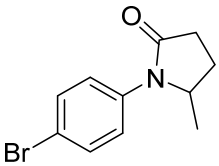
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 2H), 7.12 – 7.01 (m, 2H), 4.30 – 4.15 (m, 1H), 2.69 – 2.45 (m, 2H), 2.37 (dddd, *J* = 13.3, 9.4, 7.3, 6.2 Hz, 1H), 1.78 – 1.70 (m, 1H), 1.18 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.24, 160.43 (d, *J* = 245.4 Hz), 133.54, 125.98 (d, *J* = 8.3 Hz), 115.78 (d, *J* = 22.5 Hz), 55.84, 31.10, 26.73, 20.10 ppm.

1-(4-Chlorophenyl)-5-methylpyrrolidin-2-one



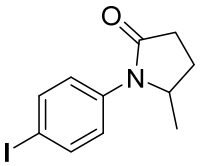
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (s, 4H), 4.27 (dd, *J* = 13.1, 6.2 Hz, 1H), 2.57 (ddd, *J* = 16.7, 9.5, 6.8 Hz, 2H), 2.45 – 2.24 (m, 1H), 1.75 (dddd, *J* = 12.8, 9.5, 7.1, 5.6 Hz, 1H), 1.20 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.17, 136.12, 130.86, 129.01, 124.89, 55.40, 31.18, 26.56, 19.95 ppm.

1-(4-Bromophenyl)-5-methylpyrrolidin-2-one



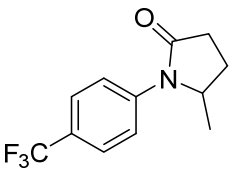
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.32 (m, 2H), 7.32 – 7.16 (m, 2H), 4.19 (dd, *J* = 12.2, 6.0 Hz, 1H), 2.67 – 2.16 (m, 3H), 1.79 – 1.52 (m, 1H), 1.11 (dd, *J* = 6.1, 1.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.98, 136.55, 131.79, 124.99, 118.41, 55.15, 31.08, 26.38, 19.78 ppm.

1-(4-Iodophenyl)-5-methylpyrrolidin-2-one



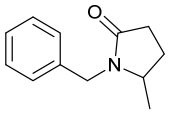
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 8.7 Hz, 2H), 7.17 (d, *J* = 8.7 Hz, 2H), 4.36 – 4.20 (m, 1H), 2.70 – 2.46 (m, 2H), 2.43 – 2.30 (m, 1H), 1.75 (dddd, *J* = 12.7, 9.5, 7.0, 5.6 Hz, 1H), 1.21 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.17, 137.99, 137.43, 125.38, 89.65, 55.25, 31.30, 26.58, 19.98 ppm.

5-Methyl-1-(4-(trifluoromethyl)phenyl)pyrrolidin-2-one



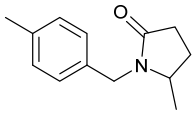
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.7 Hz, 2H), 7.57 (d, *J* = 8.7 Hz, 2H), 4.39 (dd, *J* = 12.9, 6.2 Hz, 1H), 2.75 – 2.33 (m, 4H), 1.91 – 1.74 (m, 2H), 1.25 (d, *J* = 6.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.52, 140.75, 127.19, 126.91 – 126.24 (m), 126.04 (d, *J* = 3.8 Hz), 125.35, 122.83, 55.14, 31.29, 26.45, 19.82 ppm.

1-Benzyl-5-methylpyrrolidin-2-one



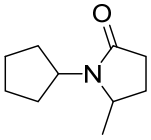
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.13 (m, 5H), 4.96 (d, *J* = 15.0 Hz, 1H), 3.99 (d, *J* = 15.0 Hz, 1H), 3.59 – 3.42 (m, 1H), 2.58 – 2.29 (m, 2H), 2.23 – 2.07 (m, 1H), 1.67 – 1.51 (m, 1H), 1.16 (d, *J* = 6.3 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.94, 136.88, 128.60, 127.97, 127.39, 52.81, 43.90, 30.28, 26.67, 19.60 ppm.

5-Methyl-1-(4-methylbenzyl)pyrrolidin-2-one



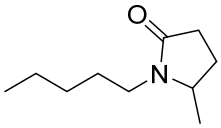
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (s, 4H), 4.93 (d, *J* = 14.9 Hz, 1H), 3.92 (d, *J* = 14.9 Hz, 1H), 3.50 (dd, *J* = 13.5, 6.2 Hz, 1H), 2.43 (ddd, *J* = 25.5, 10.0, 7.0 Hz, 2H), 2.32 (s, 3H), 2.19 – 2.09 (m, 1H), 1.57 (dddd, *J* = 12.9, 9.6, 7.2, 5.9 Hz, 1H), 1.15 (d, *J* = 6.3 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.83, 137.01, 133.76, 129.24, 127.96, 52.68, 43.57, 30.28, 26.62, 21.04, 19.55 ppm.

1-Cyclopentyl-5-methylpyrrolidin-2-one



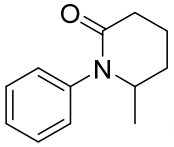
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.14 – 3.98 (m, 1H), 3.77 (ddd, *J* = 7.6, 6.3, 3.4 Hz, 1H), 2.52 – 2.07 (m, 3H), 1.98 – 1.87 (m, 1H), 1.84 – 1.50 (m, 8H), 1.48 – 1.34 (m, 1H), 1.23 (d, *J* = 6.3 Hz, 3H).^{13} C NMR (101 MHz, $CDCl_3$) δ 174.82, 54.16, 53.78, 30.46, 30.17, 28.54, 27.24, 23.82 (d, *J* = 3.1 Hz), 21.80.

5-Methyl-1-pentylpyrrolidin-2-one



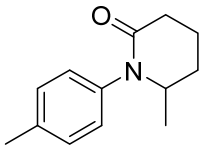
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.63 (dd, *J* = 13.2, 6.3 Hz, 1H), 3.52 (ddd, *J* = 13.8, 9.0, 7.1 Hz, 1H), 2.86 (ddd, *J* = 13.8, 8.9, 5.1 Hz, 1H), 2.41 – 2.20 (m, 2H), 2.12 (dddd, *J* = 13.3, 9.3, 7.4, 6.1 Hz, 1H), 1.58 – 1.34 (m, 3H), 1.34 – 1.05 (m, 7H), 0.83 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.63, 53.23, 39.95, 30.29, 29.08, 27.06, 26.77, 22.34, 19.74, 13.92.

6-Methyl-1-phenylpiperidin-2-one



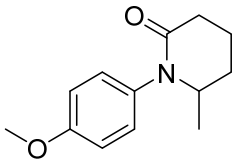
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (t, *J* = 7.7 Hz, 2H), 7.29 – 7.23 (m, 1H), 7.17 – 7.12 (m, 2H), 3.96 – 3.84 (m, 1H), 2.59 – 2.43 (m, 2H), 2.14 – 2.02 (m, 1H), 2.02 – 1.89 (m, 1H), 1.82 (dddd, *J* = 13.3, 9.7, 6.7, 3.0 Hz, 1H), 1.70 (tdd, *J* = 8.5, 6.1, 2.9 Hz, 1H), 1.05 (d, *J* = 6.4 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.35, 141.57, 129.14, 128.11, 127.10, 55.75, 32.76, 30.86, 20.89, 18.33 ppm.

6-Methyl-1-(p-tolyl)piperidin-2-one



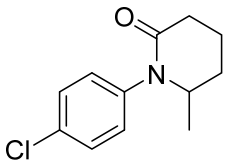
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 3.94 – 3.77 (m, 1H), 2.52 (t, *J* = 6.6 Hz, 2H), 2.34 (s, 3H), 2.15 – 2.03 (m, 1H), 2.03 – 1.90 (m, 1H), 1.90 – 1.76 (m, 1H), 1.76 – 1.65 (m, 1H), 1.06 (d, *J* = 6.4 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.35, 138.95, 136.78, 129.81, 127.82, 55.73, 32.81, 30.86, 21.10, 20.89, 18.36 ppm.

1-(4-Methoxyphenyl)-6-methylpiperidin-2-one



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.7 Hz, 2H), 6.91 (d, *J* = 8.6 Hz, 2H), 3.85 (dd, *J* = 12.1, 6.1 Hz, 1H), 3.80 (s, 3H), 2.52 (t, *J* = 6.6 Hz, 2H), 2.16 – 1.96 (m, 2H), 1.91 – 1.66 (m, 2H), 1.08 (d, *J* = 6.4 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.57, 158.26, 134.18, 128.94, 114.36, 55.84, 55.33, 32.67, 30.77, 20.78, 18.25 ppm.

1-(4-Chlorophenyl)-6-methylpiperidin-2-one



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 3.96 – 3.82 (m, 1H), 2.53 (t, *J* = 6.6 Hz, 2H), 2.18 – 1.65 (m, 4H), 1.07 (d, *J* = 6.4 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.32, 139.99, 132.67, 129.33 (d, *J* = 12.5 Hz), 55.69, 32.69, 30.78, 20.84, 18.27 ppm.

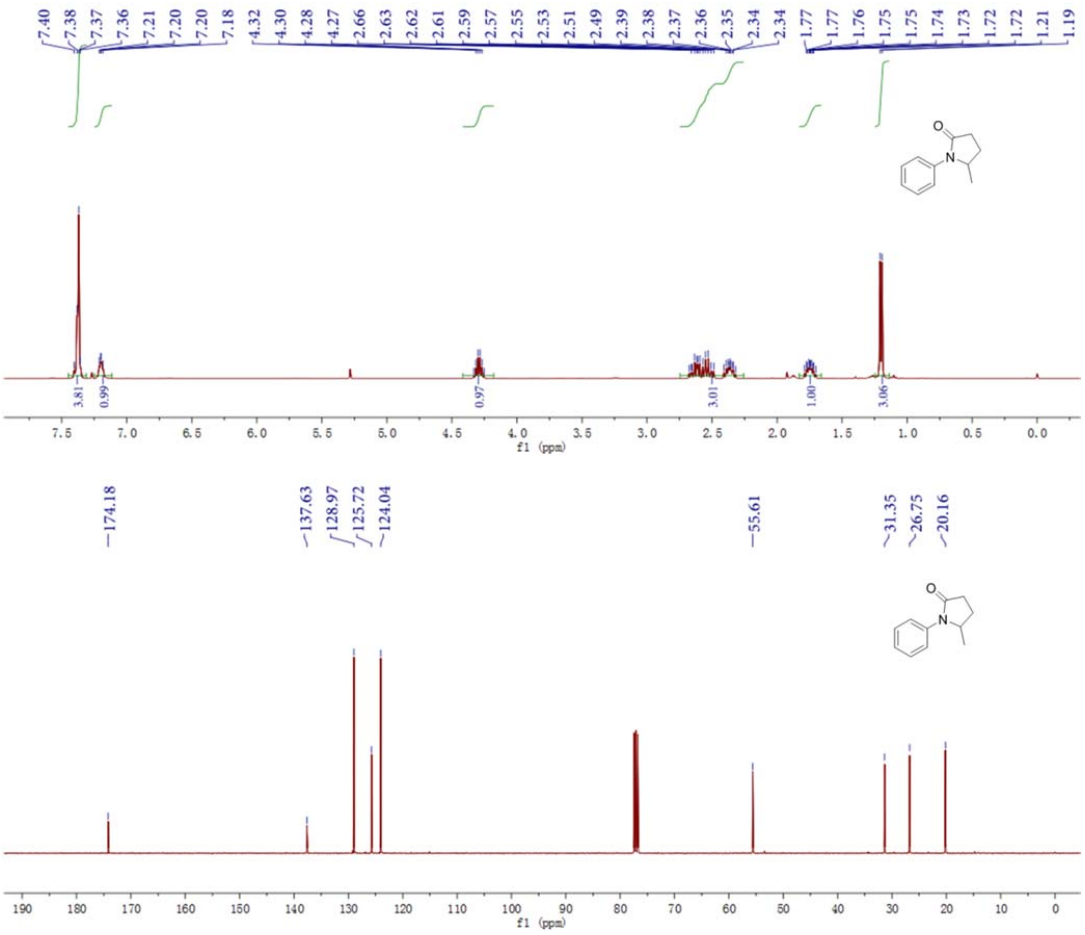


Figure S10. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 5-methyl-1-phenylpyrrolidin-2-one

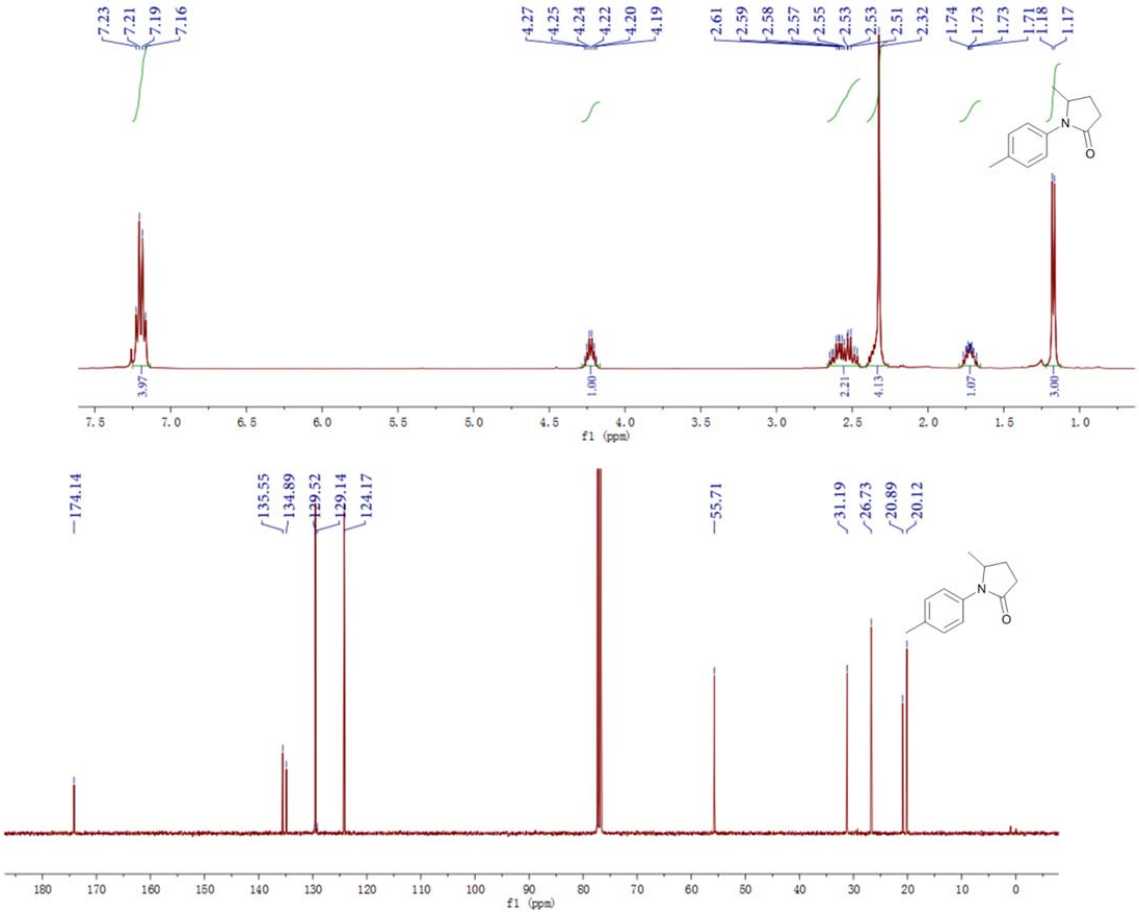


Figure S11. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 5-methyl-1-(p-tolyl)pyrrolidin-2-one

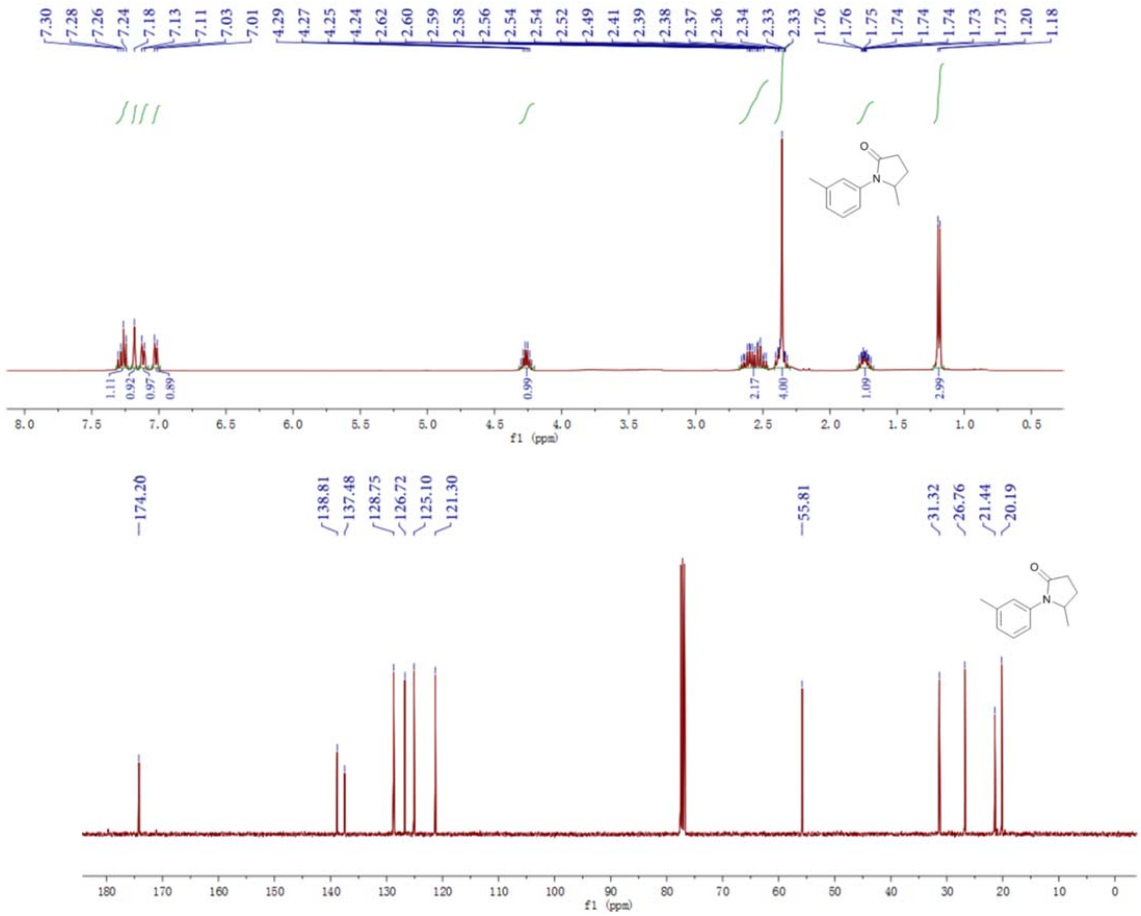


Figure S12. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 5-methyl-1-(m-tolyl)pyrrolidin-2-one

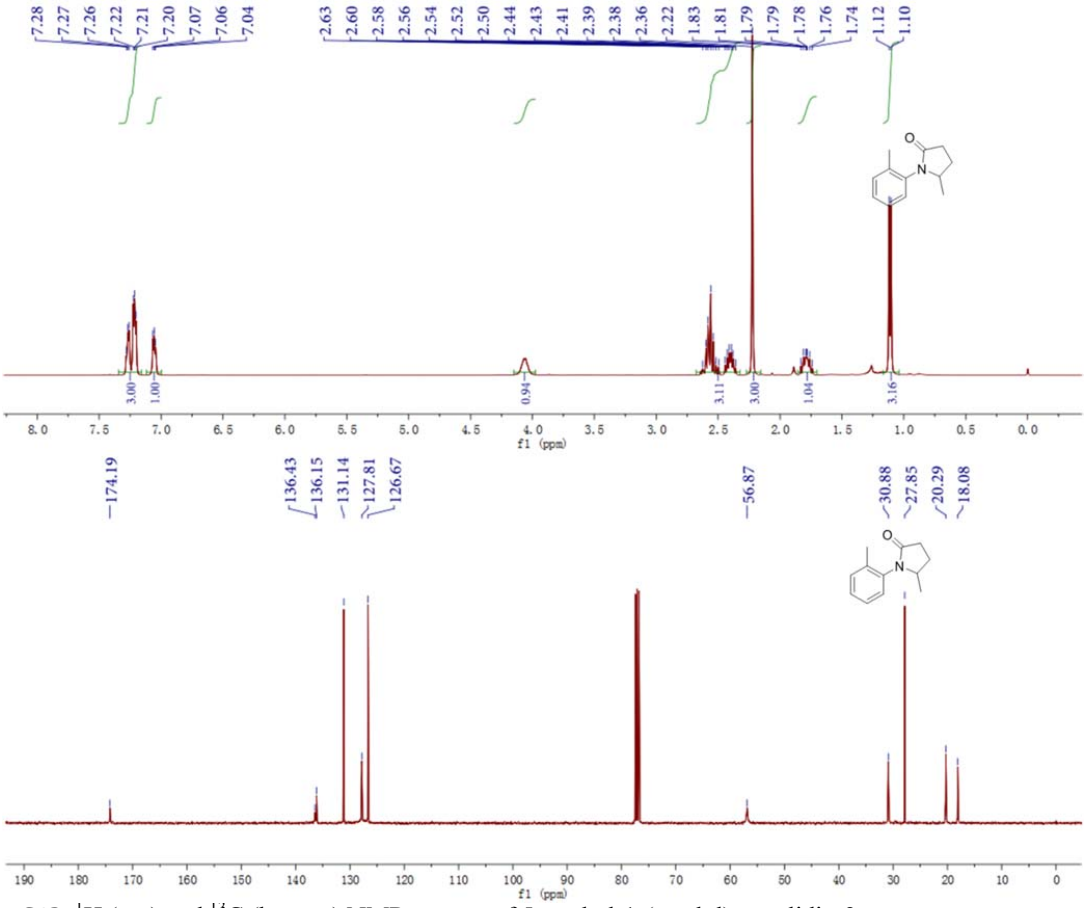


Figure S13. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 5-methyl-1-(o-tolyl)pyrrolidin-2-one

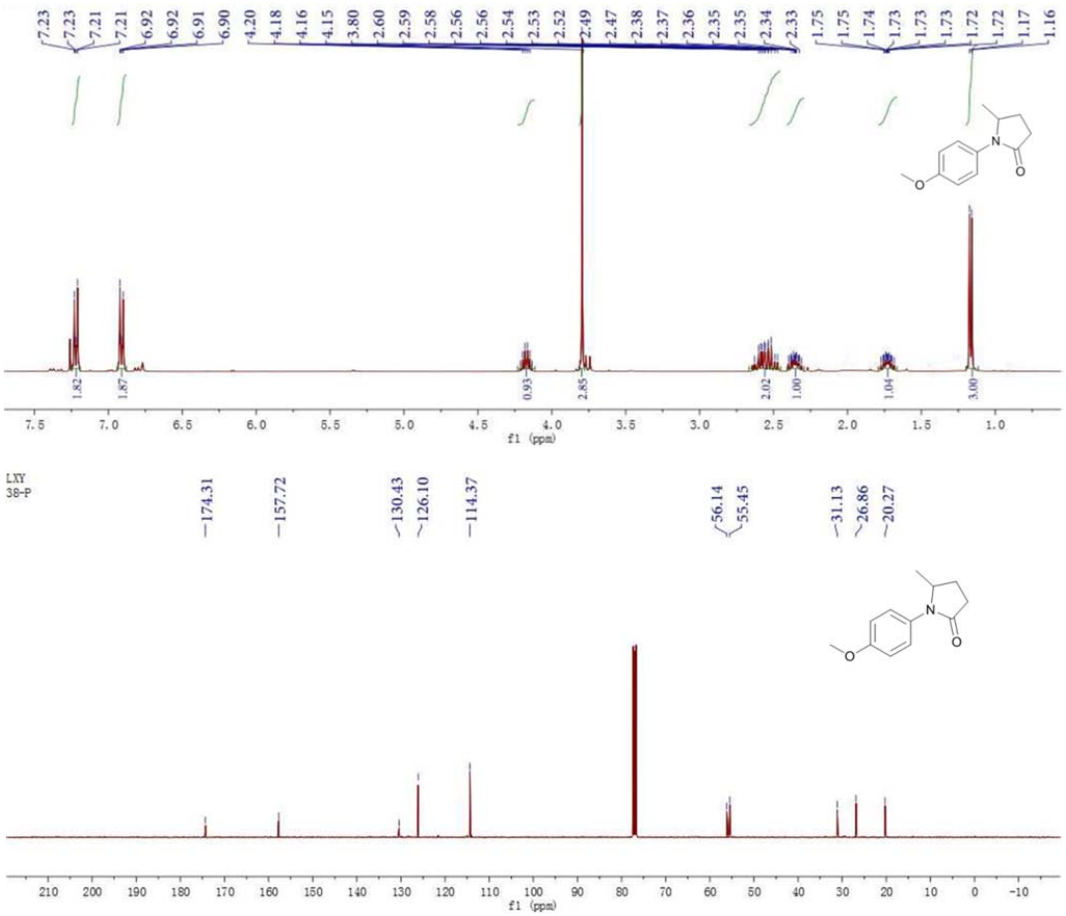


Figure S14. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-(4-methoxyphenyl)-5-methylpyrrolidin-2-one

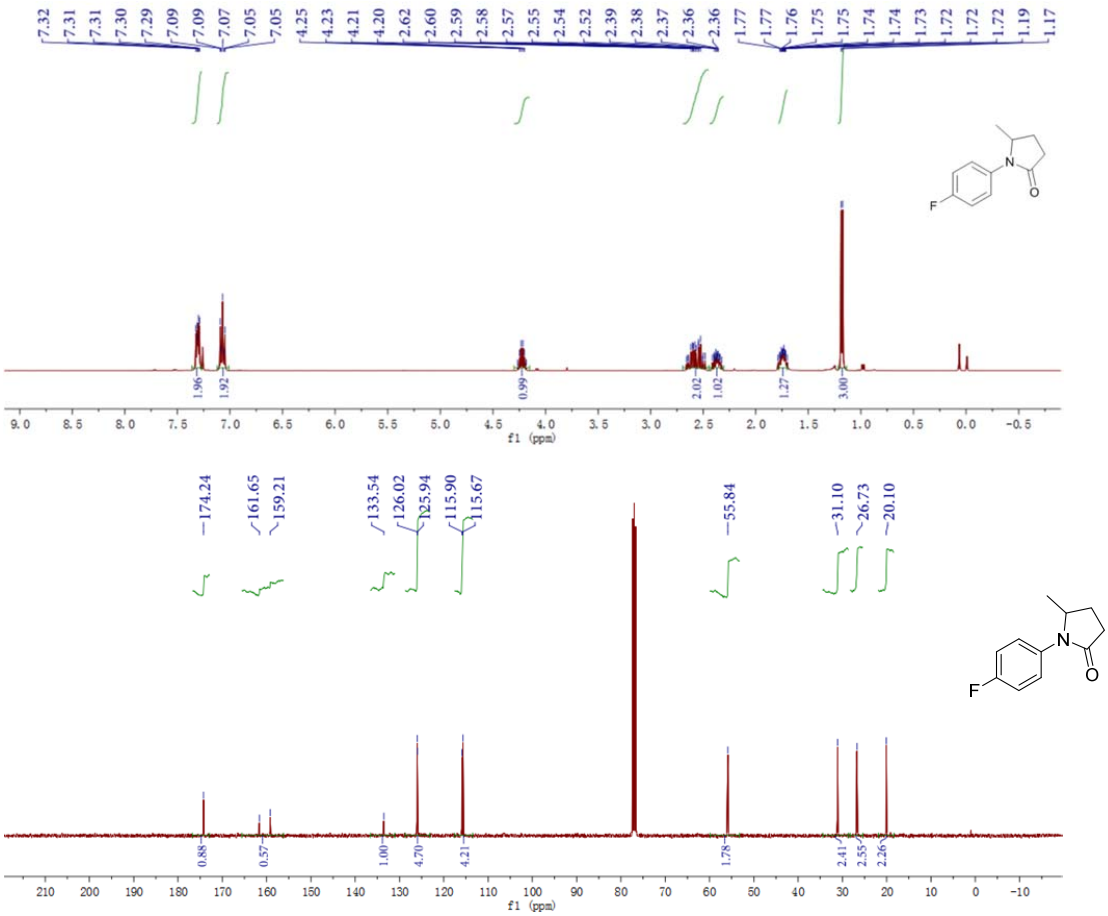


Figure S15. ^{1}H (top), and ^{13}C (bottom) NMR spectra of 1-(4-fluorophenyl)-5-methylpyrrolidin-2-one

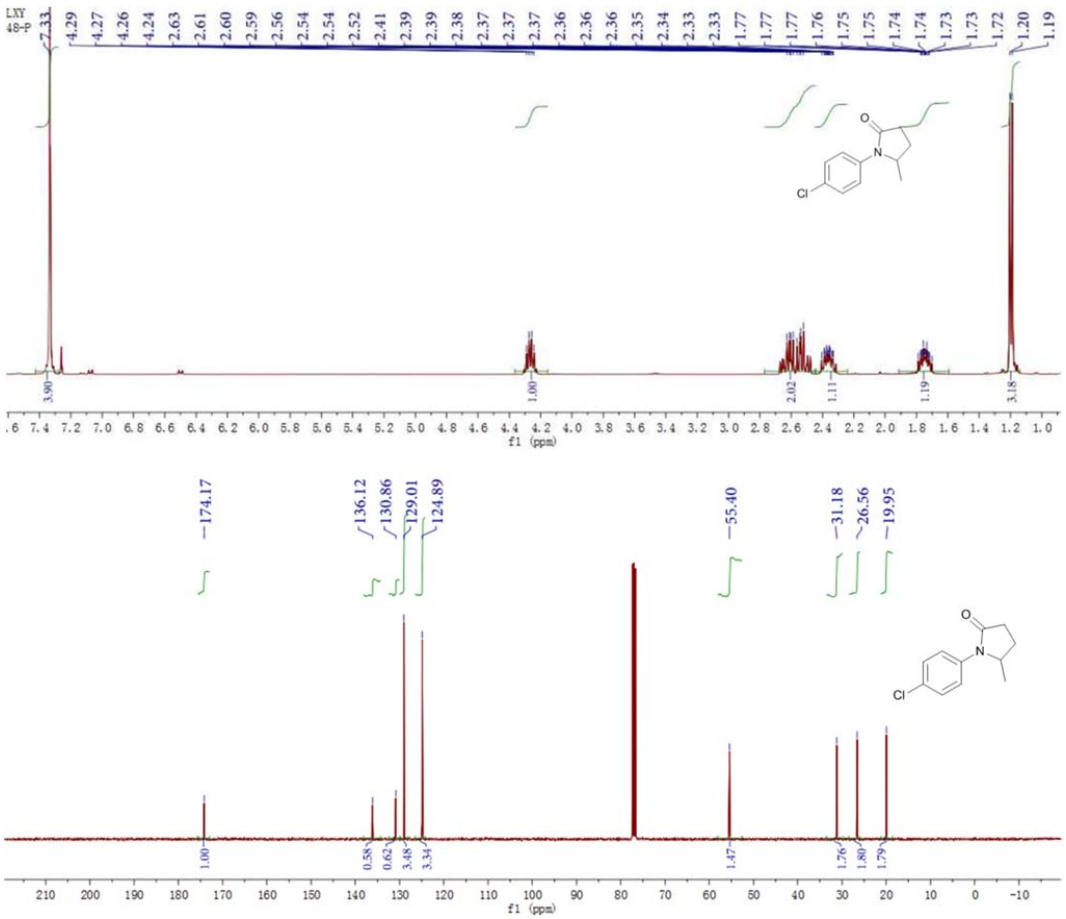


Figure S16. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-(4-chlorophenyl)-5-methylpyrrolidin-2-one

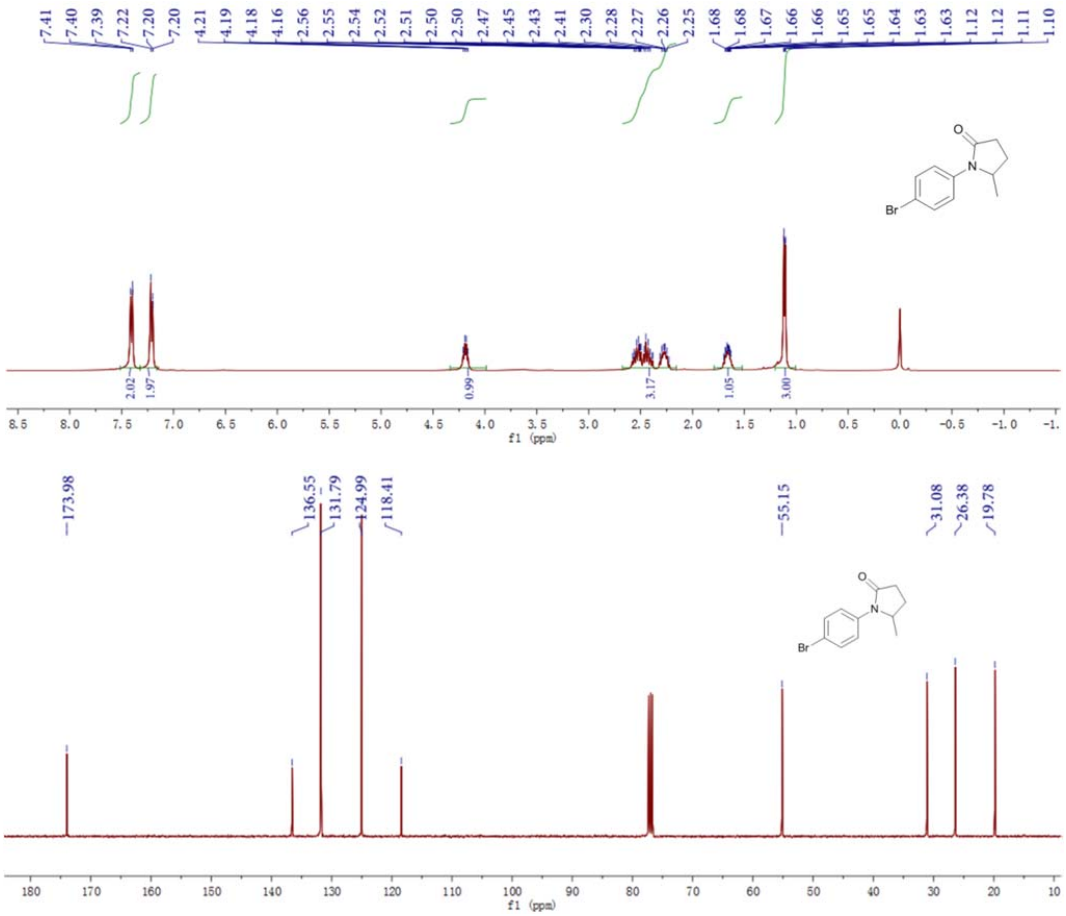


Figure S17. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-(4-bromophenyl)-5-methylpyrrolidin-2-one

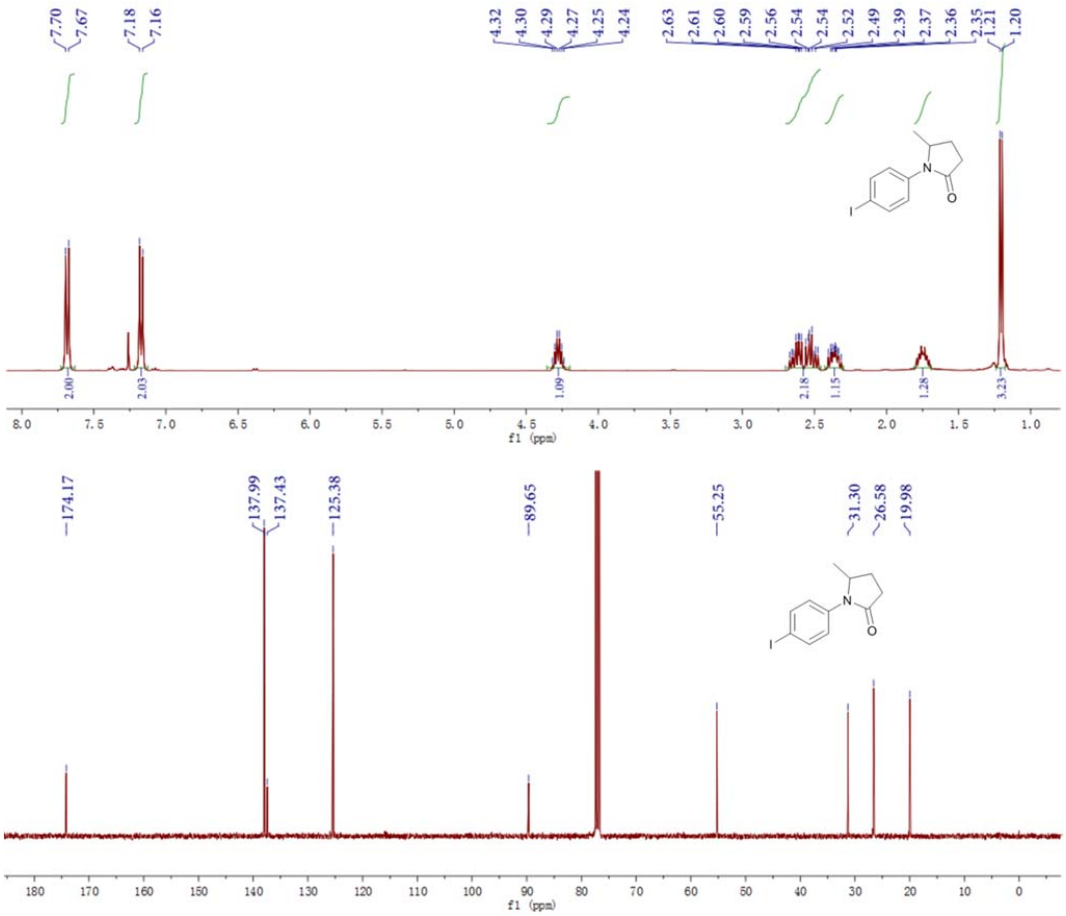


Figure S18. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-(4-iodophenyl)-5-methylpyrrolidin-2-one

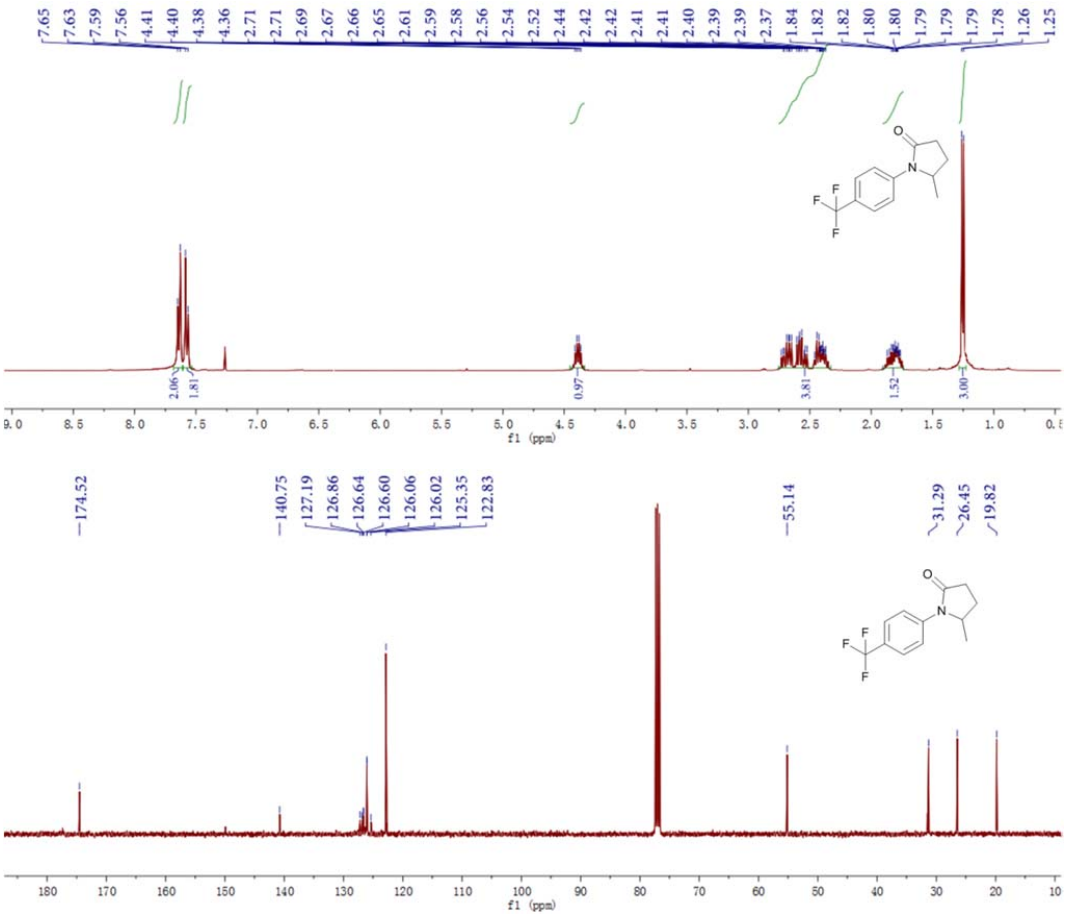


Figure S19. ^{1}H (top), and ^{13}C (bottom) NMR spectra of 5-methyl-1-(4-(trifluoromethyl)phenyl)pyrrolidin-2-one

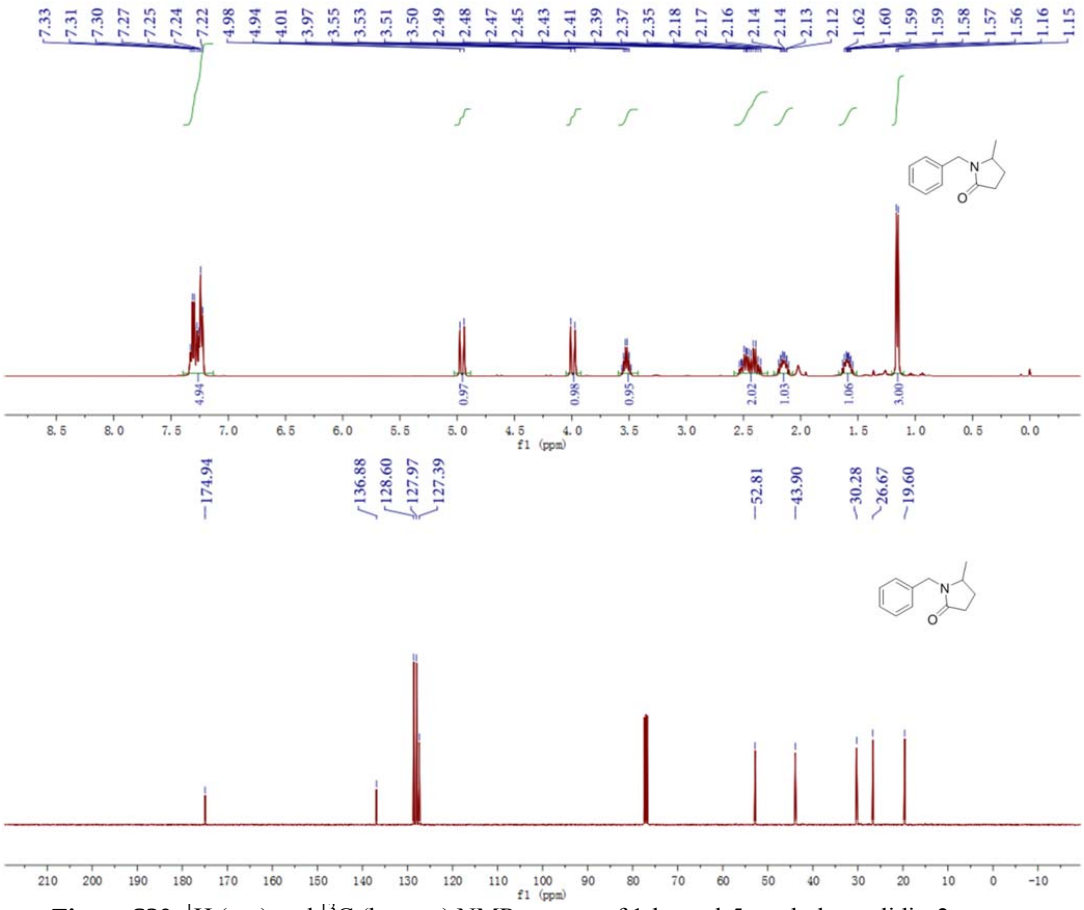


Figure S20. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-benzyl-5-methylpyrrolidin-2-one

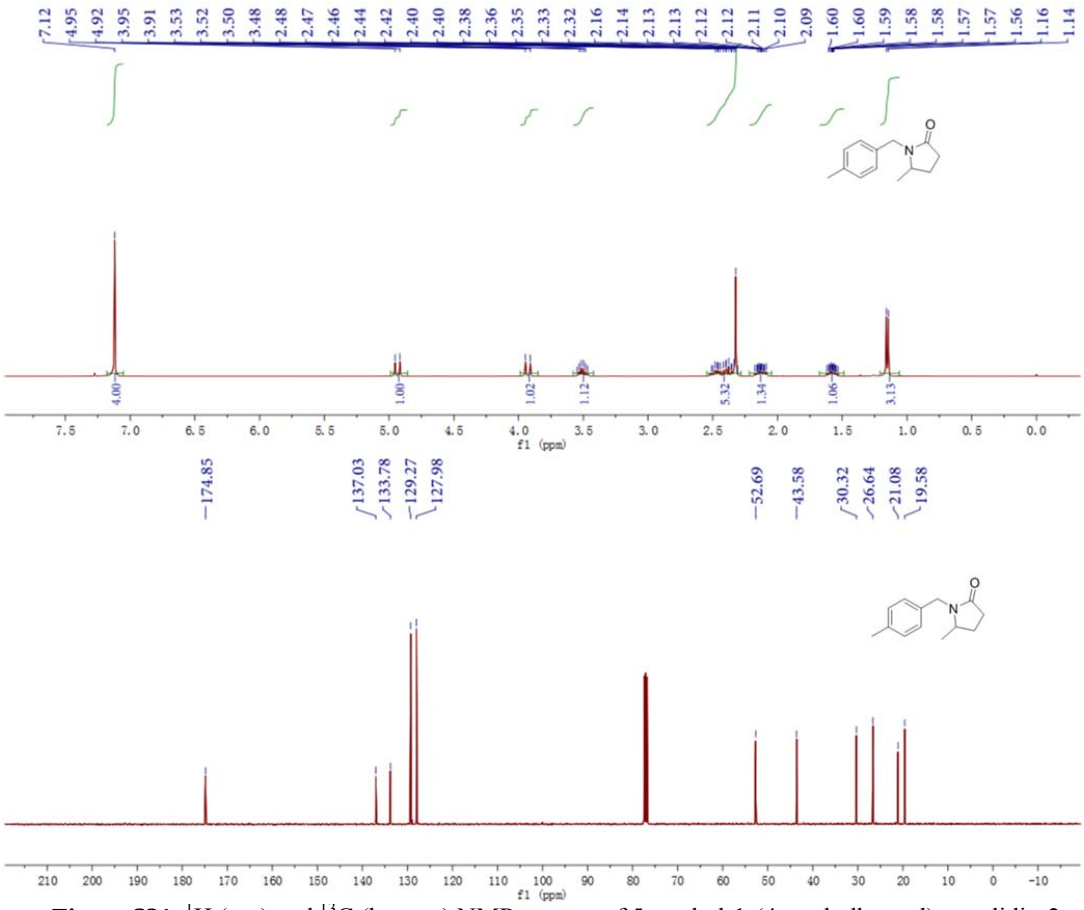


Figure S21. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 5-methyl-1-(4-methylbenzyl)pyrrolidin-2-one

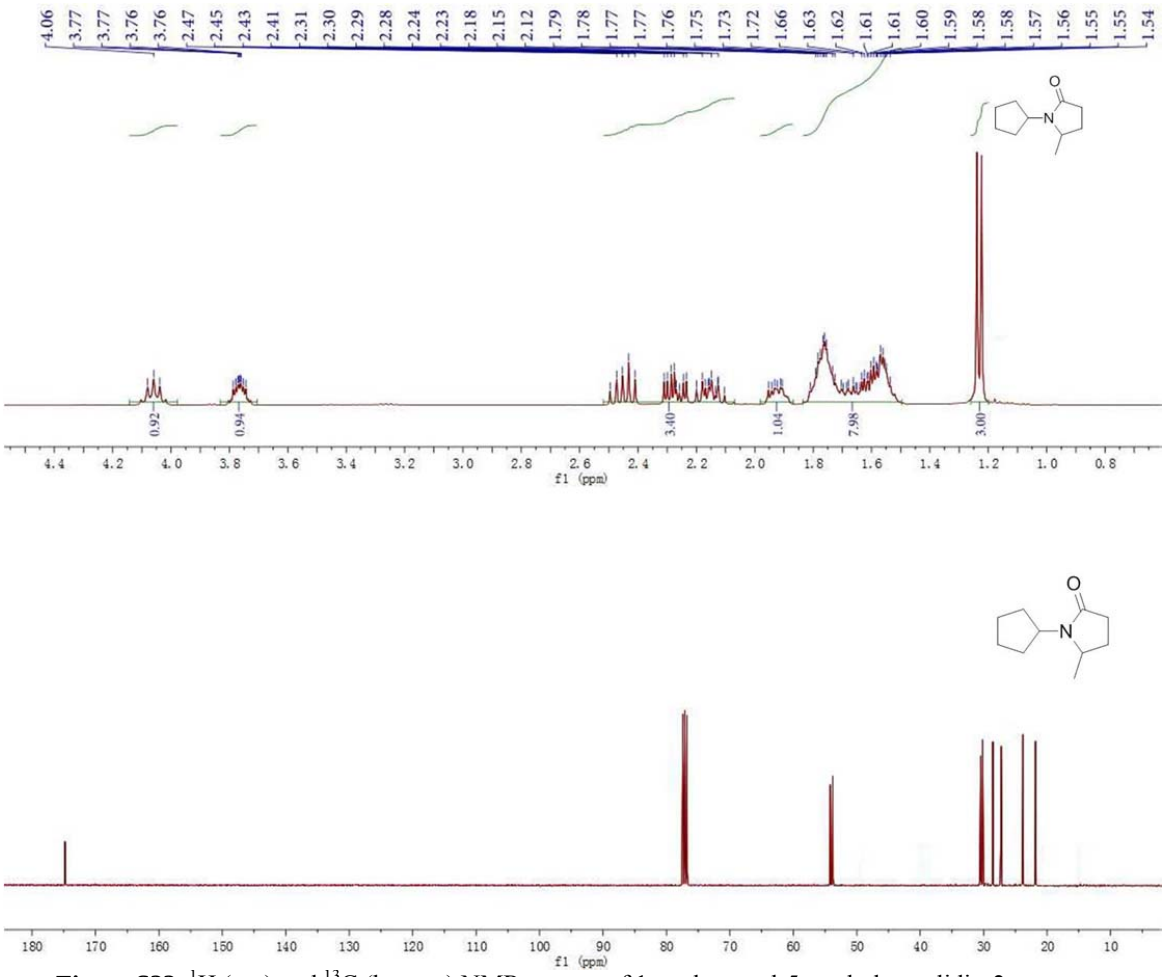


Figure S22. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-cyclopentyl-5-methylpyrrolidin-2-one

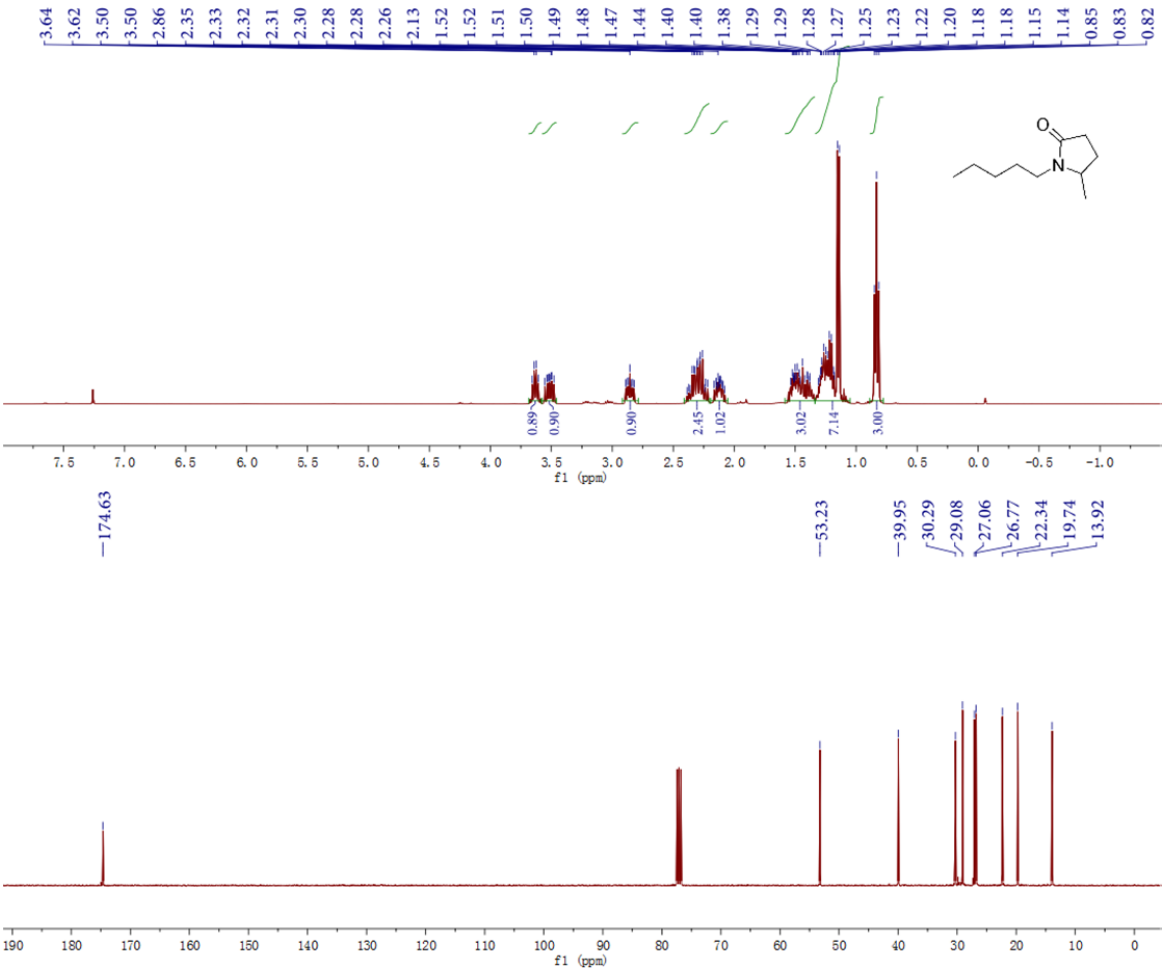


Figure S23. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 5-methyl-1-pentylpyrrolidin-2-one

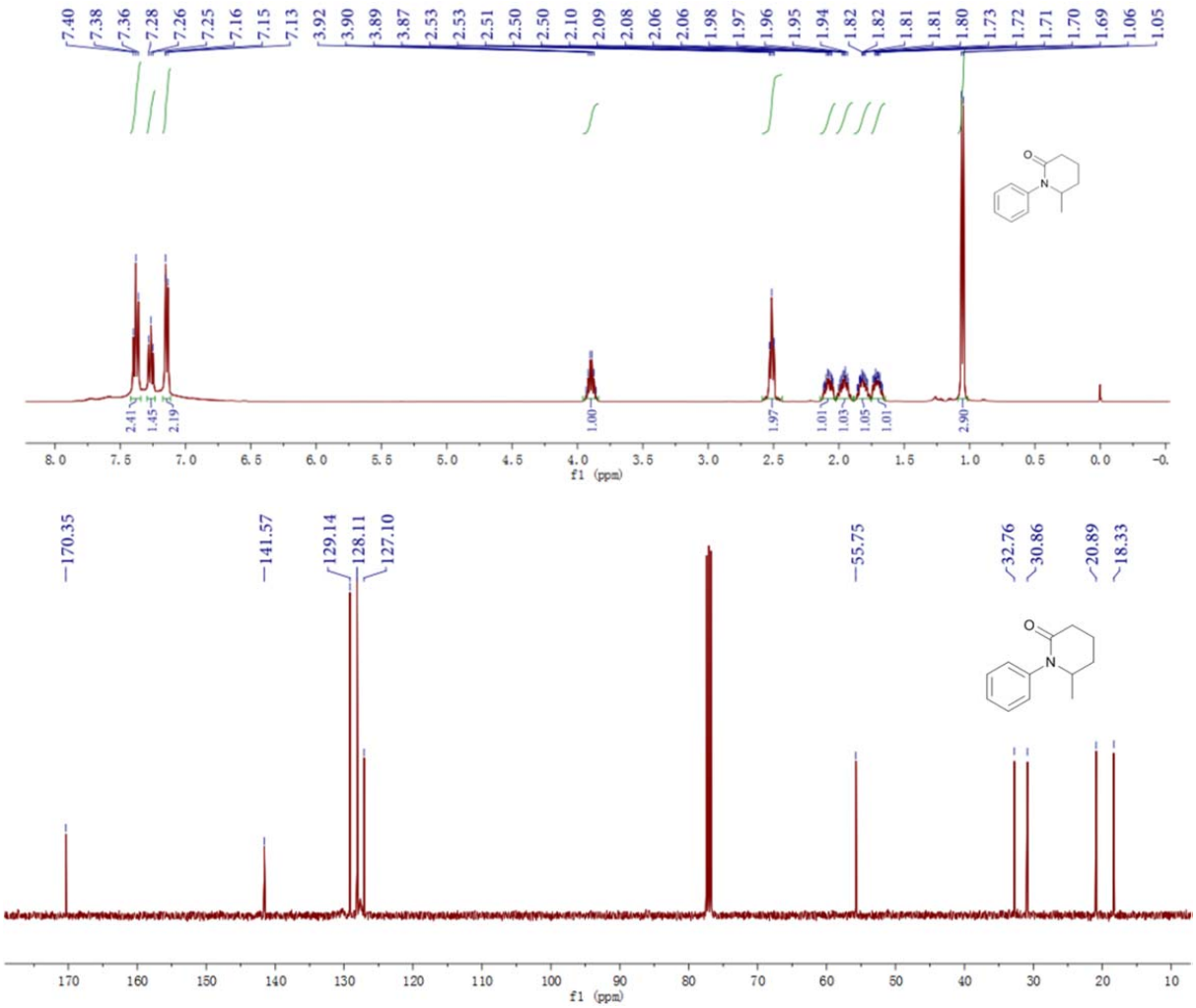


Figure S24. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 6-methyl-1-phenylpiperidin-2-one

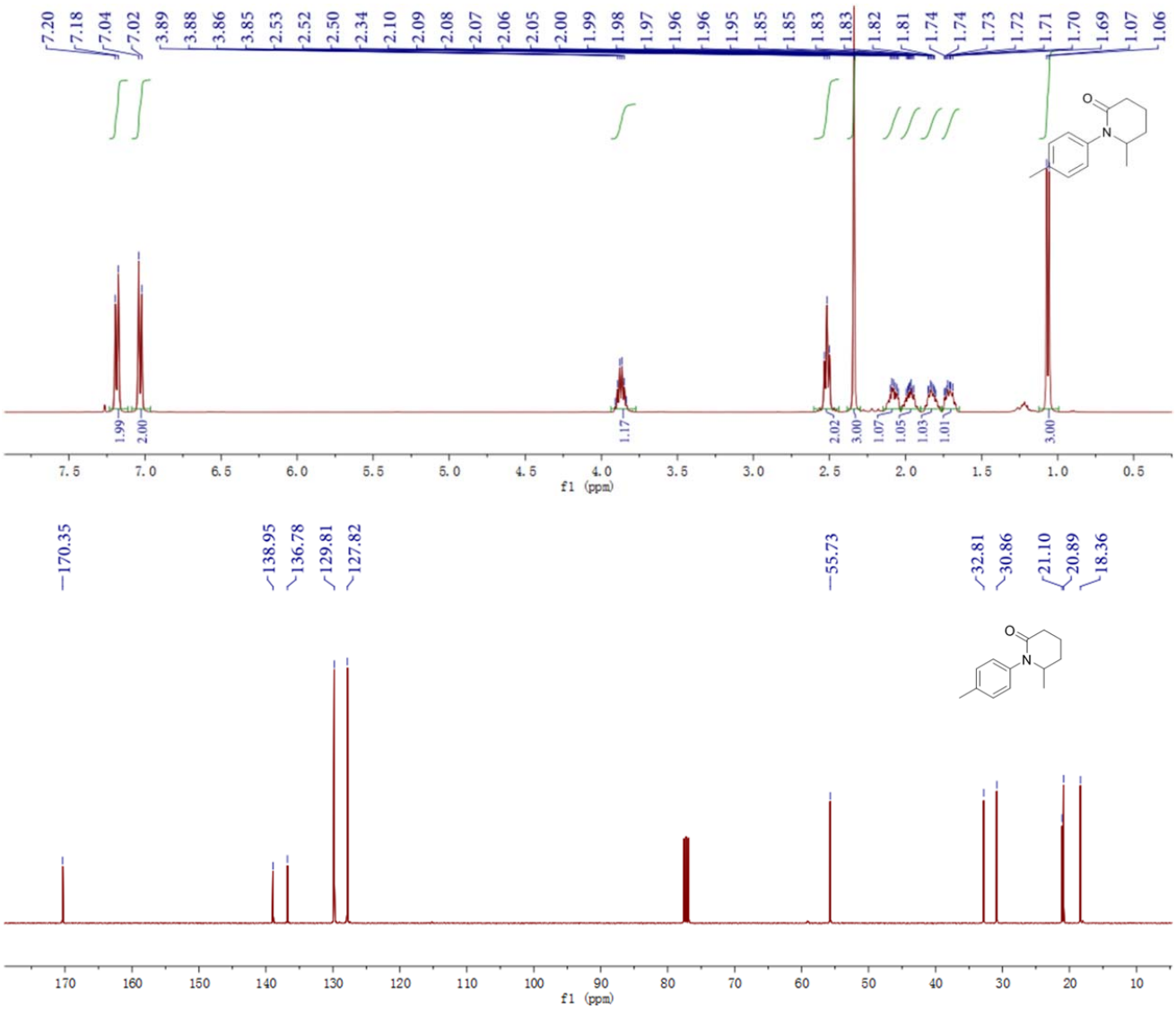


Figure S25. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 6-methyl-1-(p-tolyl)piperidin-2-one

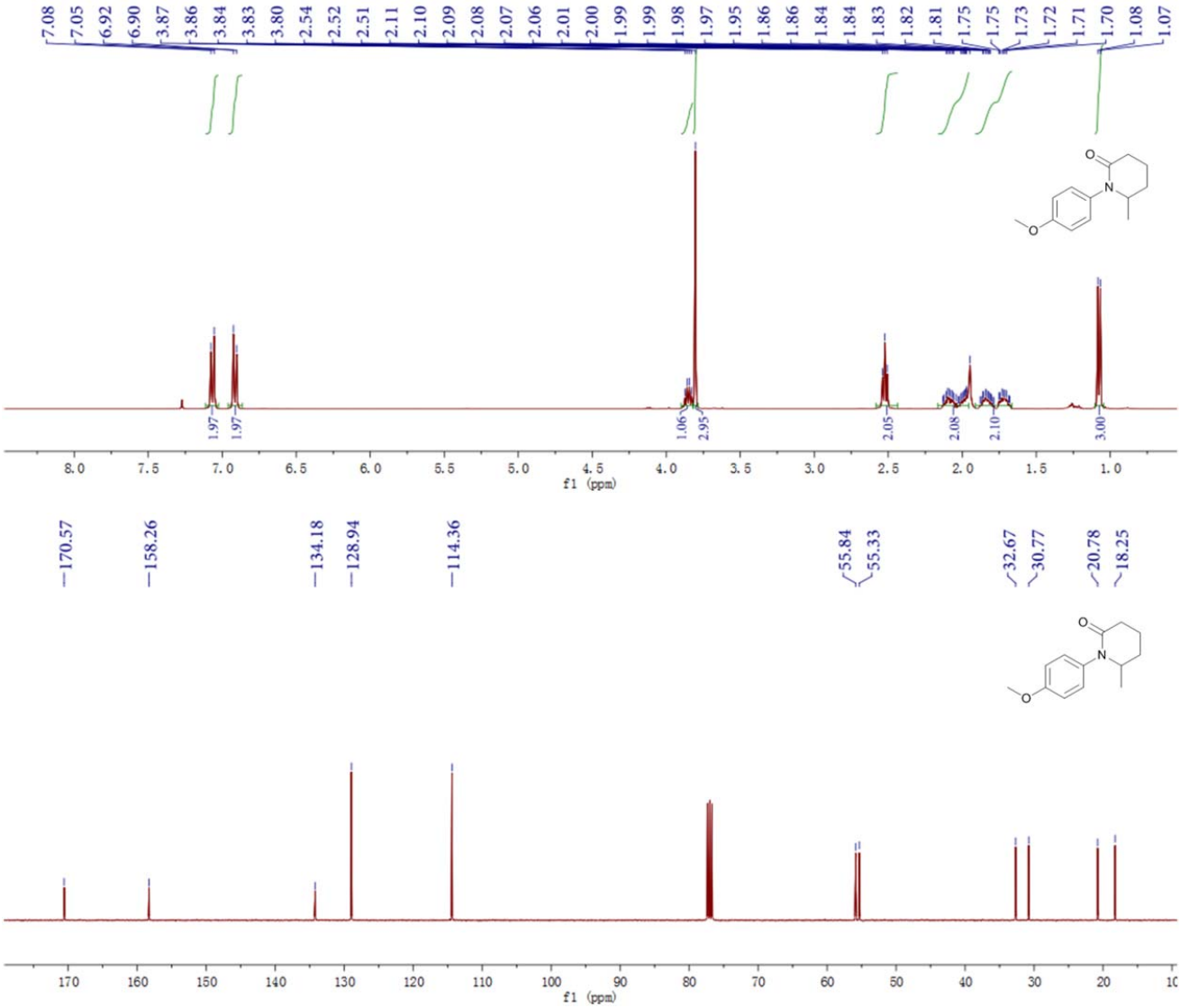


Figure S26. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-(4-methoxyphenyl)-6-methylpiperidin-2-one

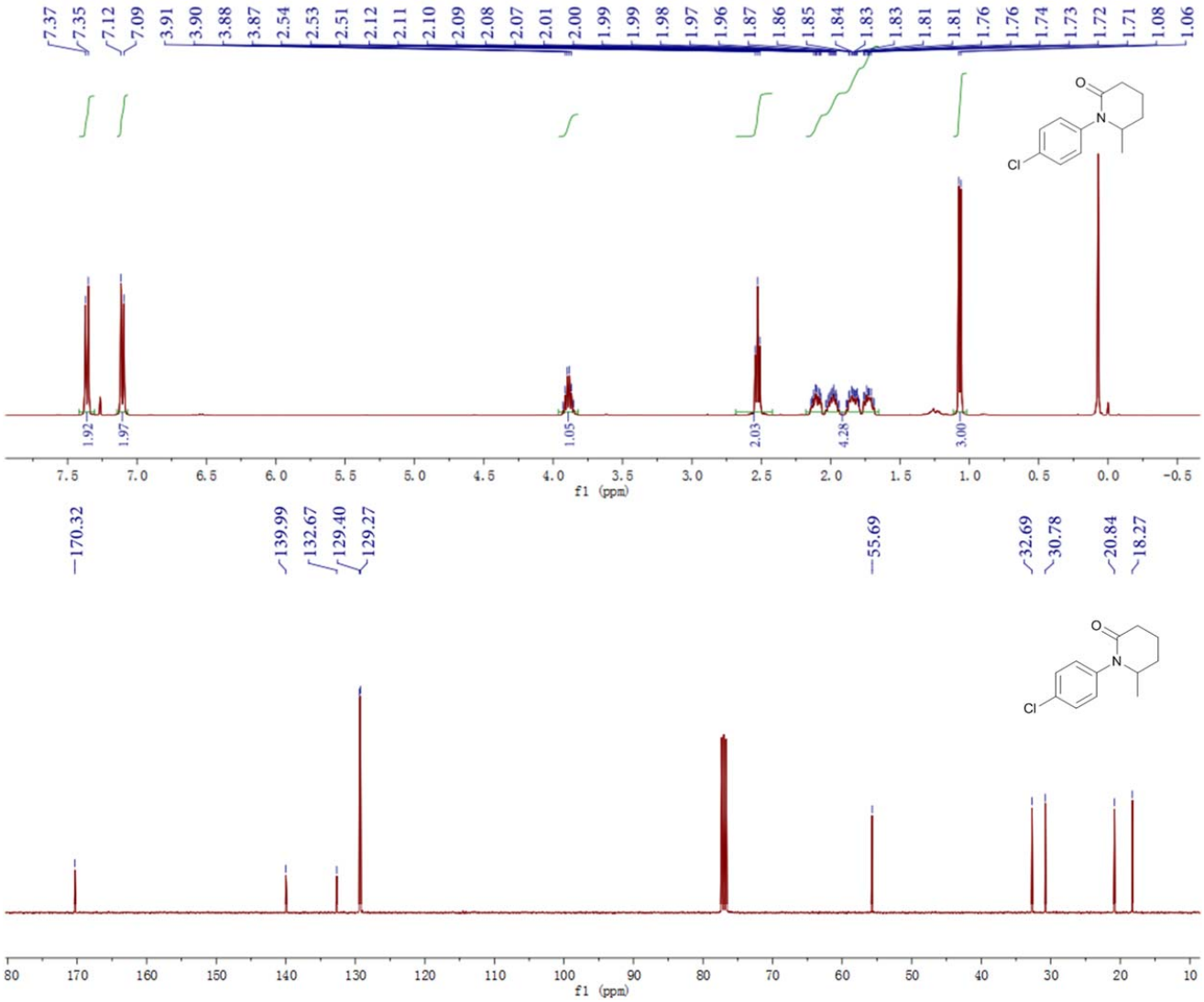


Figure S27. ^{1}H (top) and ^{13}C (bottom) NMR spectra of 1-(4-chlorophenyl)-6-methylpiperidin-2-one

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1. Hayouni, S.;Robert, A.; Ferlin, N.; Amri, H.; Bouquillon, S. *RSC Adv.*, 2016, **6**, 113583-113595.
2. Yu, B.; Zhang, H.; Zhao, Y.; Chen, S.; Xu, J.; Hao, L.; Liu, Z. *ACS Catal.*, 2013, **3**, 2076-2082.