Supporting Information for:

Ambient Reductive Amination of Levulinic Acid to Pyrrolidones over Pt Nanocatalysts on Porous TiO_2 Nanosheets

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Experimental Section

Materials: Platinum(IV) chloride (99%), levulinate acid (98%), methyl levulinate (97%), ethyl levulinate (98%), butyl levulinate (98%), 2-carboxybenzaldehyde (99%), 2-acetylbenzoic acid (99%), butylamine (99.5%), *n*-octylamine (99%), aminodiphenylmethane (99%), 3-amino-1-propanol (98.5%), 3-methoxypropylamine (99%), *N,N*-diethylethylenediamine (99%), cyclopentylamine (99%), cyclohexylamine (99%), benzylamine (99%), 4-chlorobenzylamine (98%), 3-methoxybenzylamine (98.5%), aniline (99.5%), 4-*tert*-butylaniline (98%), *p*-anisidine (99%), 4-fluoroaniline (99%), 4-chloroaniline (98%), 4-bromoaniline (99%), 4-iodoaniline (98%), 4-aminobenzonitrile (98%), 4-aminoacetophenone (99%), 4-aminoacetanilide (98%), 4-aminostyrene (96%), *p*-toluidine (99%), *o*-toluidine (98.5%), *o*-anisidine (99%), 2,4,6-trimethylaniline (99%), titanium(IV) *n*-butoxide (99%), 4-acetylbutyric acid (96%), and benzylamine (99%) were provided by J&K Scientific Ltd. *n*-Heptylamine (99+%), titanium(IV) oxide (98%), P25 (99%) and potassium nitrate (99+%) were obtained from Acros. Cycloheptylamine was purchased from Aladdin. 3-Aminopentane, methyl 4-aminobenzoate (98%), 4-ethynylaniline (98%), and *m*-toluidine (99%) were provided by Innochem. *n*-Hexane (A.R.), *N,N*-Dimethylformamide (A.R.), Tetrahydrofuran (A.R.), 1,4-Dioxane (A.R.), Acetonitrile (A.R.), Ethanol (A.R.), Isopropanol (A.R.) and Methanol (A.R.) were purchased from

Sinopharm Chemical Reagent Beijing Co. Ltd. Pt/C (5 wt%) was purchased from Alfa Aesar.

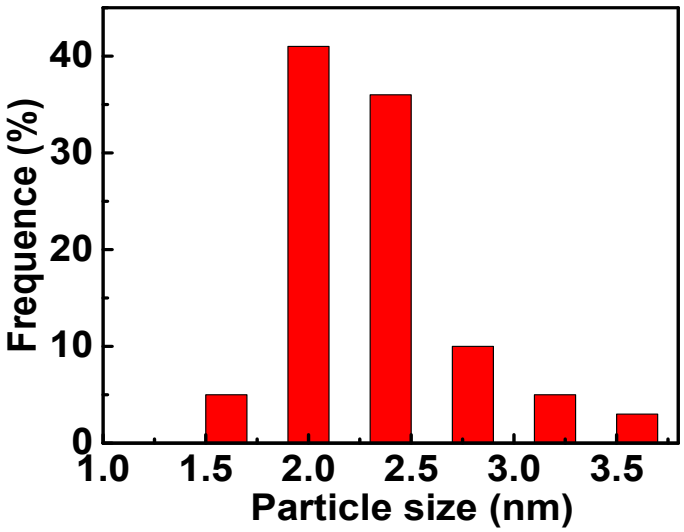


Figure S1. The size distribution of Pt NPs in the prepared Pt/P-TiO_2, and the data were obtained from 100 Pt particles.

Table S1. CO-pulse titration results of the used supported Pt catalysts.

Entry	Catalyst	Pt dispersion (%)	Crystal size of Pt particles (nm)
1	Pt/C	18	5.3
2	Pt/TiO_2	16	5.7
3	Pt/25	17	5.4
4	Pt/P-TiO_2	21	4.8

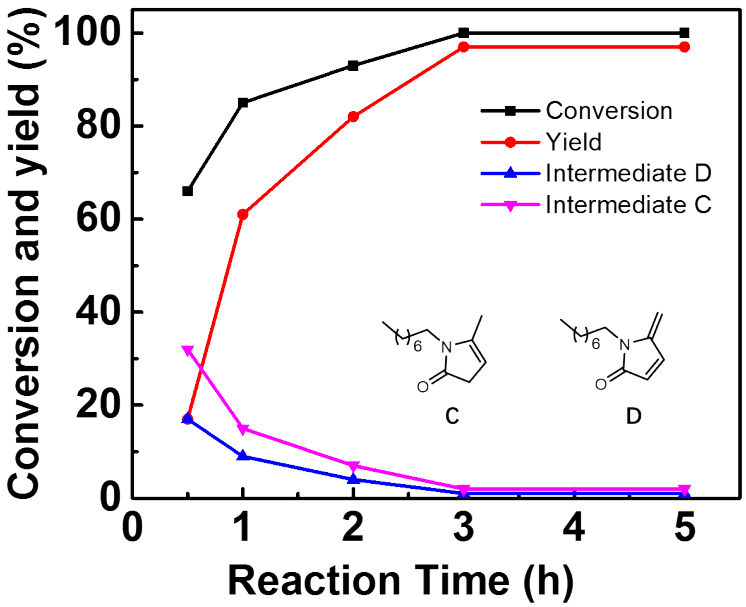


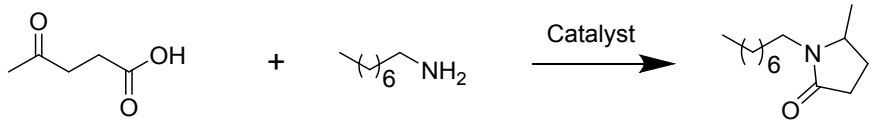
Figure S2. Time-yield plots for the reductive amination of LA with *n*-octylamine. Reaction conditions: *n*-octylamine, 1 mmol; LA, 1 mmol; methanol, 2 mL; hydrogen balloon; room temperature; Pt/P-TiO_2, 0.1 mol% Pt.

Table S2. Reductive amination of LA with *n*-octylamine over various catalysts.

Entry	Catalyst	mol%	Reductant	T (^oC)	Time (h)	Yield (%)	Ref.
1	Ir complex	0.2	HCOOH	80	12	88	1
2[a]	Ir complex	0.05	0.5 MPa H_2	110	16	92	2
3	Ru complex	0.5	HCOOH	80	12	62	3
4[a]	Au/ZrO_2	0.05	HCOOH	130	12	95	4
5	Pt/C	0.86	6.9 MPa H_2	150	18	99	5
6	Pt-MoO_X/TiO_2	0.1	0.3 MPa H_2	100	20	95	6
7	Pt/TiO_2	0.05	1 MPa H_2	120	2	98	7
8	$In(OAc)_3$	1	1 eq $PhSiH_3$	120	24	49	8
9	[BMIm][Lac]	20	2 eq $(ETO)_3SiH$	80	1	94	9
10	Pt/P-TiO_2	0.1	Hydrogen balloon	r.t.	3	97	This work

[a]Amination of LA with *n*-hexylamine.

Table S3. Solvent effect on the reductive amination of LA with *n*-octylamine over Pt/P-TiO_2.[a]



Entry	Solvent	Yield (%)
1	*n*-Hexane	7
2	*N,N*-Dimethylformamide (DMF)	3
3	Tetrahydrofuran (THF)	4
4	1,4-Dioxane	6
5	Acetonitrile	3
6	Methanol	61
7	Ethanol	57
8	Isopropanol	54

[a]Reaction conditions: LA, 1 mmol; *n*-octylamine, 1 mmol; Pt/P-TiO_2, 0.1 mol% Pt; solvent, 2 ml; time, 1 h; temperature, r.t.; H_2 pressure, hydrogen balloon.

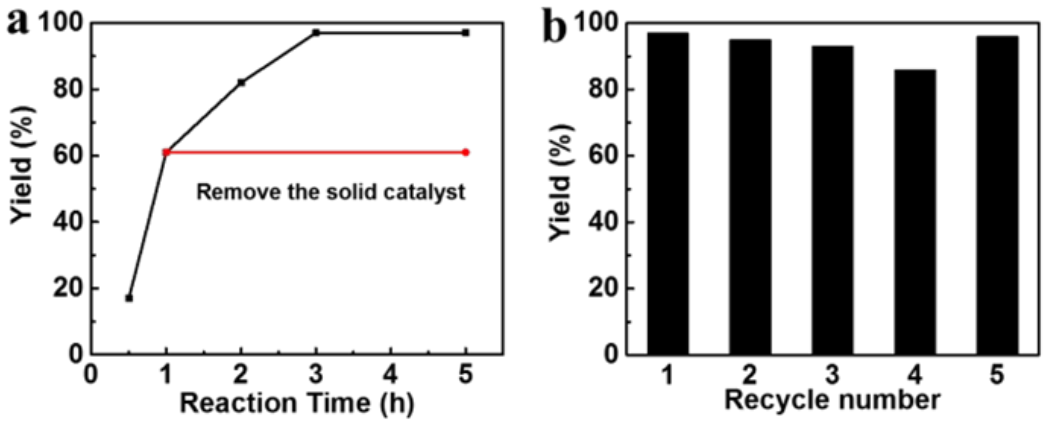


Figure S3. (a) Time-yield plots for reductive amination of LA with *n*-octylamine over Pt/P-TiO_2 (black line) or removing Pt/P-TiO_2 after 1 h (red line); (b) Reusability of the Pt/P-TiO_2. Reaction conditions: LA, 1 mmol; *n*-octylamine, 1 mmol; methanol, 2 mL; Pt/P-TiO_2, 10 mg (0.1 mol% Pt); time, 3 h; rt; hydrogen balloon. In the 5th run, 8 mg Pt/P-TiO_2 was recovered, and 0.8 mmol LA, 0.8 mmol *n*-octylamine and 1.6 mL methanol were used.

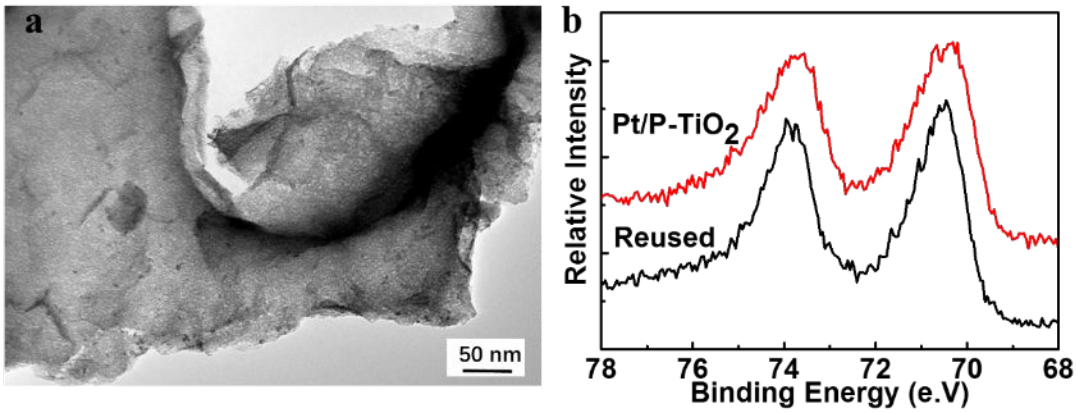


Figure S4. TEM image (a) and XPS spectra of Pt 4f (b) of the Pt/P-TiO_2 after five cycles.

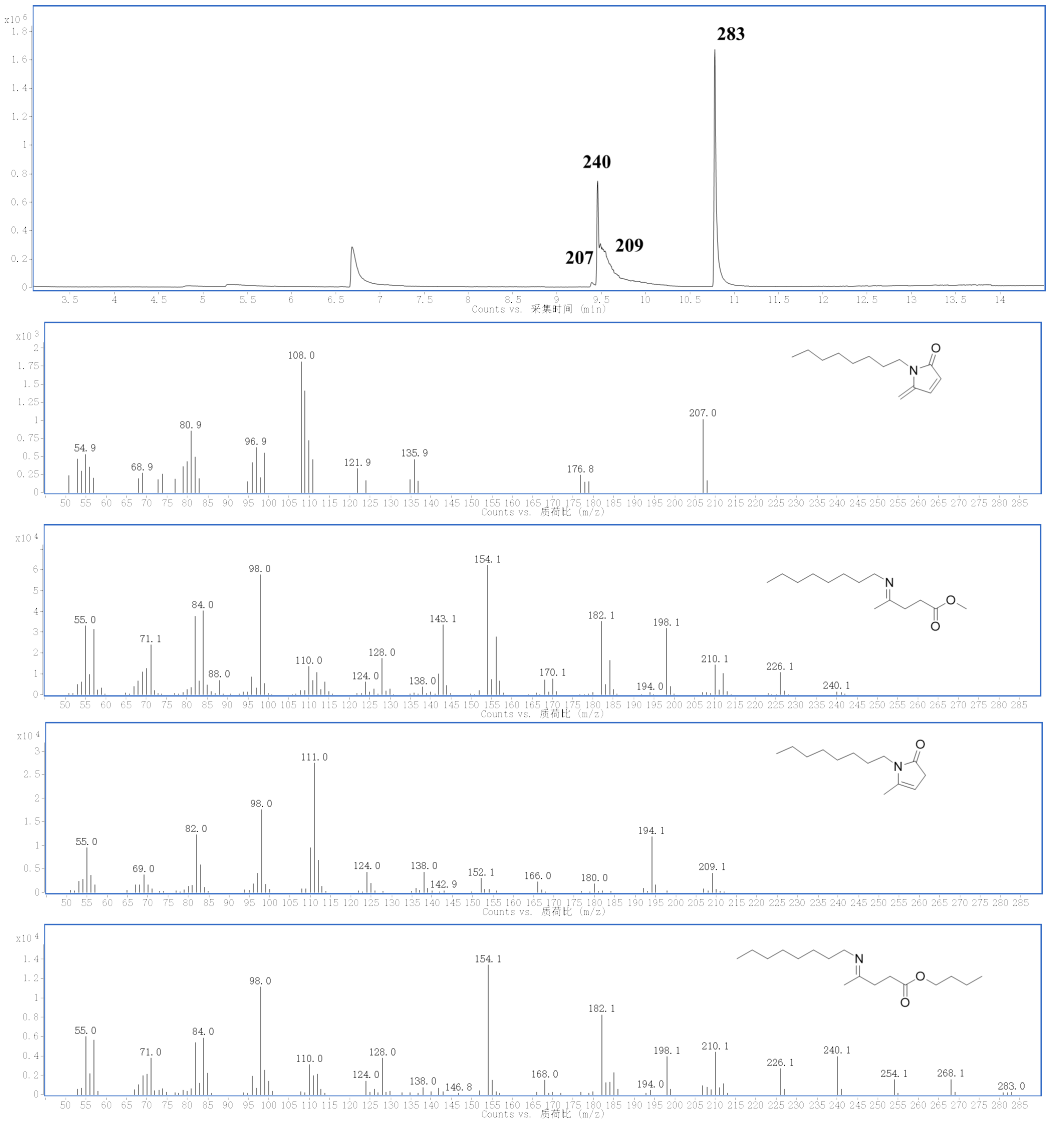


Figure S5. GC-MS spectra of the intermediate generated from butyl levulinate and *n*-octylamine without hydrogen. The probable structure was inserted.

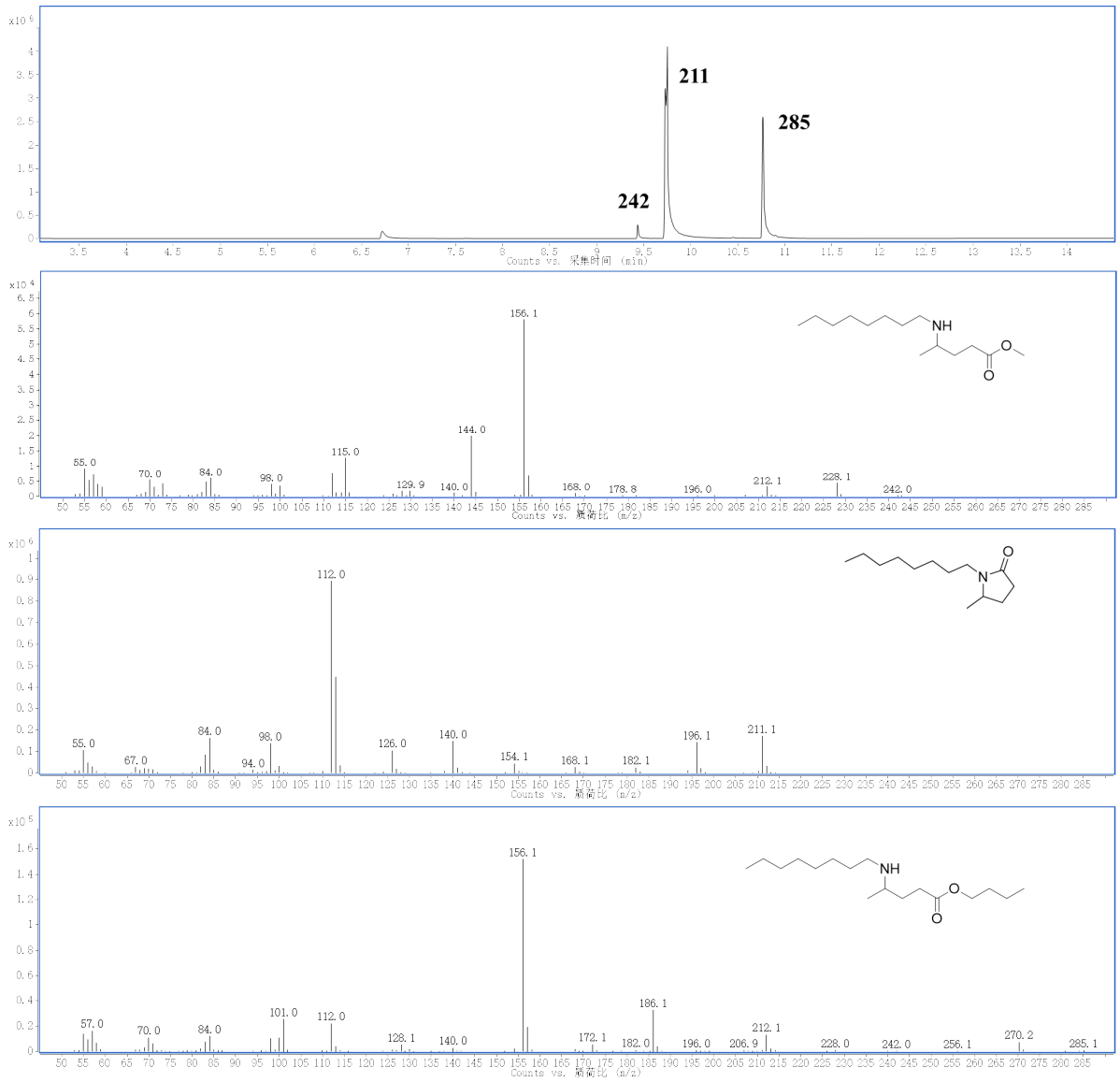


Figure S6. GC-MS spectra of the intermediate generated from butyl levulinate and *n*-octylamine using H_2 as the hydrogen resource under mild conditions. The probable structure was inserted.

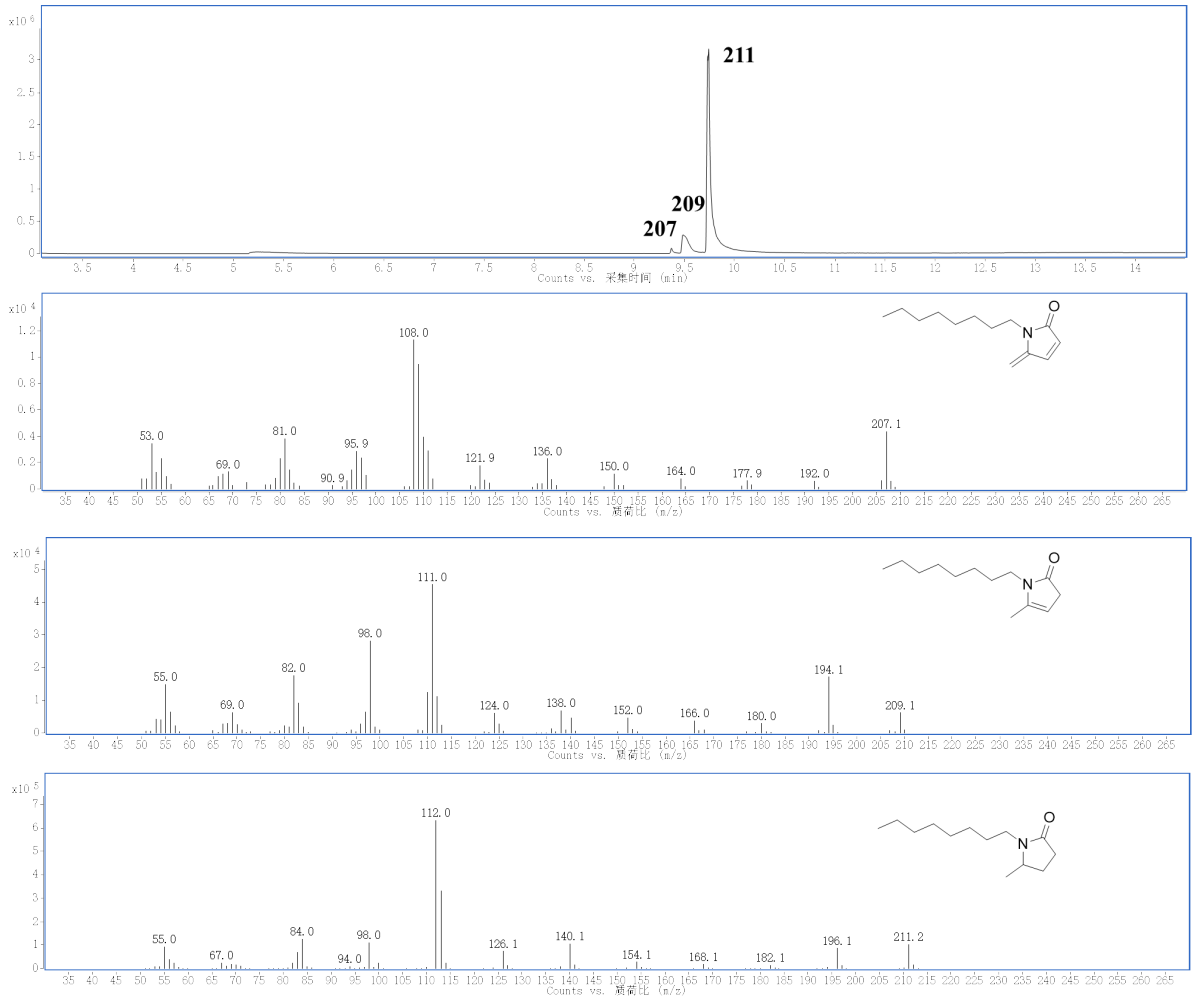


Figure S7. GC-MS spectra of the intermediate generated from LA and *n*-octylamine using H_2 as the hydrogen resource under mild conditions. The probable structure was inserted.

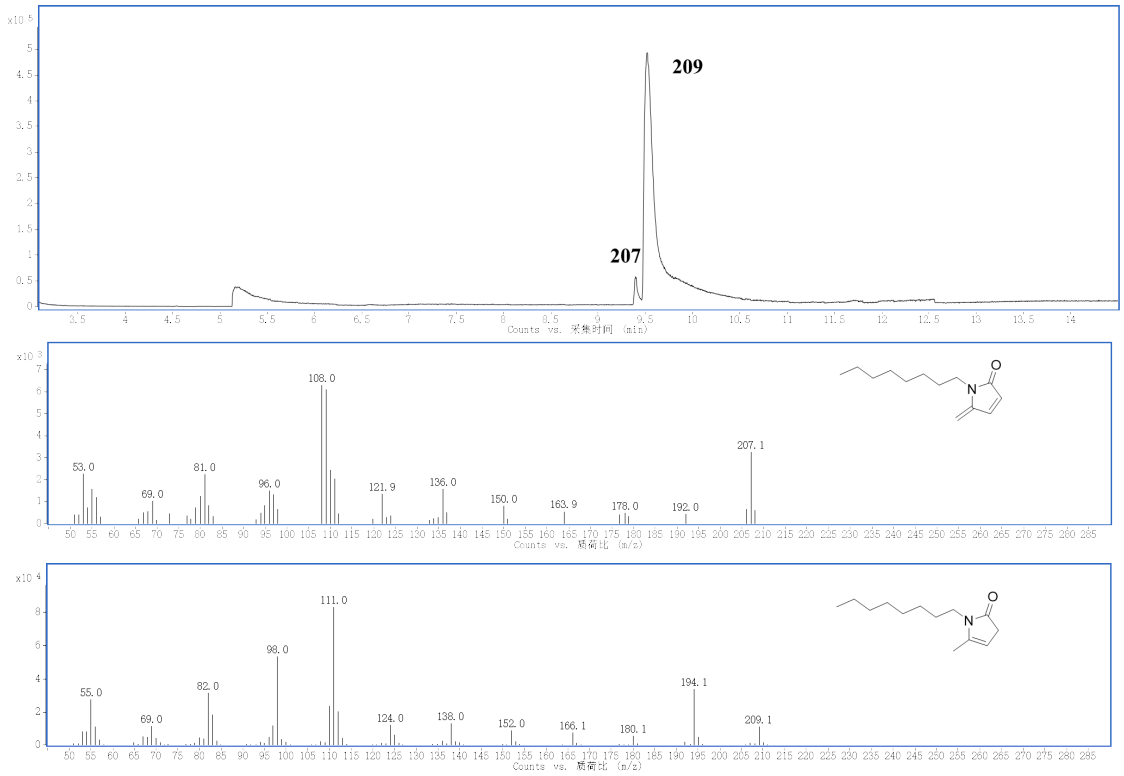


Figure S8. GC-MS spectra of the intermediate generated from LA and *n*-octylamine without hydrogen. The probable structure was inserted

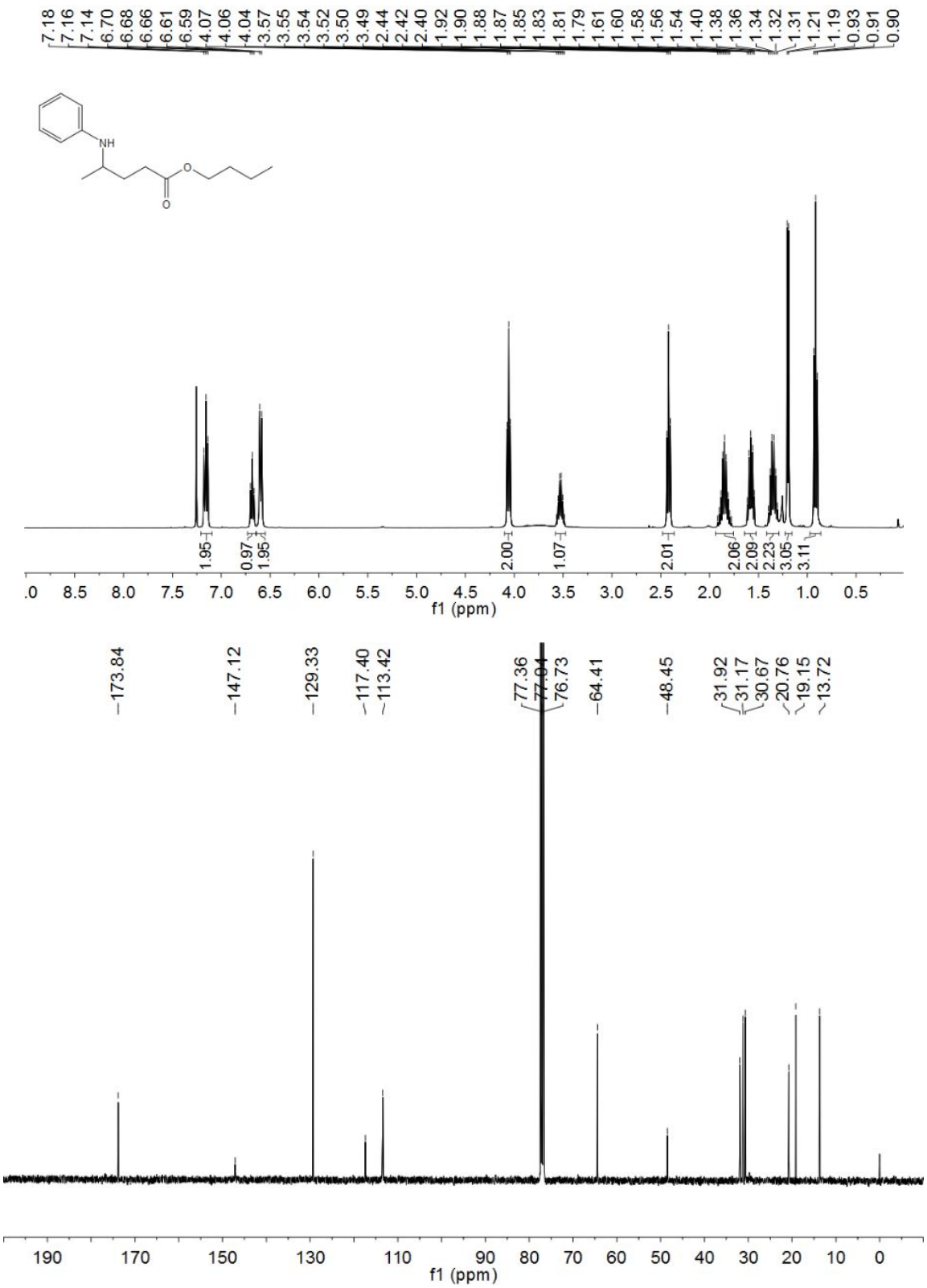
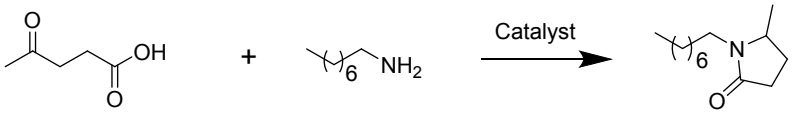
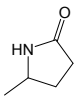


Figure S9. ^{1}H NMR and ^{13}C NMR of the intermediate butyl 4-(phenylamino)pentanoate generated from butyl levulinate and aniline using H_2 as the hydrogen resource under mild conditions.

Table S4. Effect of different additive on the catalytic activity of different catalysts.[a]



Entry	Catalyst	Additive	Yield (%)
1	Pt/TiO_2	KNO_3 (2 mg)	13
2	Pt/P25	KNO_3 (2 mg)	14
3	Pt/TiO_2	No	14
4	Pt/P25	No	16
5	$Pt/P\text{-}TiO_2$	No	61
6	$Pt/P\text{-}TiO_2$	 (1mmol)	63
7	Pt/TiO_2	 (1mmol)	2
8	Pt/P25	 (1mmol)	4

[a]Reaction conditions: LA, 1 mmol; *n*-octylamine, 1 mmol; catalyst, 0.1 mol% Pt; methanol, 2 ml; time, 1 h; temperature, r.t.; H_2 pressure, hydrogen balloon.

Reference:

(1) Wei, Y.; Wang, C.; Jiang, X.; Xue, D.; Li, J.; Xiao, J. *Chem. Commun.* **2013**, *49*, 5408-5410.

(2) Wang, S.; Huang, H.; Bruneau, C.; Fischmeister, C. *ChemSusChem* **2017**, *10*, 4150-4154.

(3) Huang, Y.-B.; Dai, J.-J.; Deng, X.-J.; Qu, Y.-C.; Guo, Q.-X.; Fu, Y. *ChemSusChem* **2011**, *4*, 1578-1581.

(4) Du, X. L.; He, L.; Zhao, S.; Liu, Y. M.; Cao, Y.; He, H. Y.; Fan, K.-N. *Angew. Chem. Int. Ed.* **2011**, *50*, 7815-7819.

(5) Manzer, L. E.; Herkes, F. E. U.S. Patent 2004/0192933A1, **2004**.

(6) Touchy, A. S.; Siddiki, S. M. A. H.; Kon, K.; Shimizu, K. *ACS Catal.* **2014**, *4*, 3045-3050.

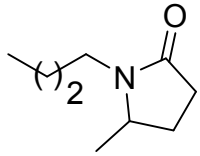
(7) Vidal, J. D.; Climent, M. J.; Concepcion, P.; Corma, A.; Iborra, S.; Sabater, M. J. *ACS Catal.* **2015**, *5*, 5812-5821.

(8) Ogiwara, Y.; Uchiyama, T.; Sakai, N. *Angew. Chem. Int. Ed.* **2016**, *55*, 1864-1867.

(9) Wu, C.; Zhang, H.; Yu, B.; Chen, Y.; Ke, Z.; Guo, S.; Liu, Z. *ACS Catal.* **2017**, *7*, 7772-7776.

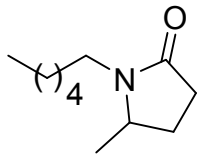
Characterization of the pure products using ^{1}H NMR, ^{13}C NMR, and HRMS:

1-Butyl-5-methylpyrrolidin-2-one (1a)



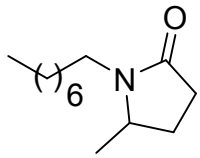
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.69 (dd, *J* = 12.3, 6.2 Hz, 1H), 3.59 (dt, *J* = 18.1, 7.8 Hz, 1H), 2.91 (dd, *J* = 12.7, 6.2 Hz, 1H), 2.48 – 2.27 (m, 2H), 2.18 (tt, *J* = 14.4, 7.1 Hz, 1H), 1.65 – 1.48 (m, 2H), 1.48 – 1.38 (m, 1H), 1.38 – 1.25 (m, 2H), 1.24 – 1.14 (m, 3H), 0.98 – 0.87 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.65, 53.25, 39.73, 30.31, 29.48, 26.78, 20.15, 19.74, 13.77. HRMS (ESI) for $C_9H_{17}NO$ $[M+H]^+$: calc. 156.1383, Found:156.1384. Yellow oil.

1-Hexyl-5-methylpyrrolidin-2-one (1b)



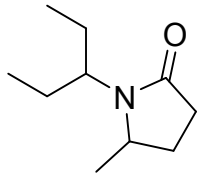
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.68 – 3.57 (m, 1H), 3.57 – 3.46 (m, 1H), 2.84 (ddd, *J* = 13.8, 8.9, 5.1 Hz, 1H), 2.42 – 2.21 (m, 2H), 2.17 – 2.04 (m, 1H), 1.57 – 1.41 (m, 2H), 1.37 (dd, *J* = 14.3, 6.2 Hz, 1H), 1.21 (s, 6H), 1.13 (d, *J* = 6.3 Hz, 3H), 0.81 (t, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.65, 53.26, 40.02, 31.50, 30.31, 27.35, 26.78, 26.61, 22.52, 19.75, 13.98. HRMS (ESI) for $C_{11}H_{21}NO$ $[M+H]^+$: calc. 184.1696, Found: 184.1697. Yellow oil.

1-Octyl-5-methylpyrrolidin-2-one (1c)



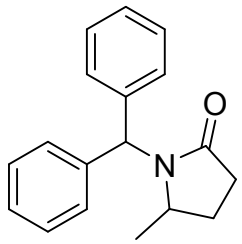
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.70 (dt, *J* = 12.9, 6.4 Hz, 1H), 3.58 (ddd, *J* = 13.9, 9.0, 7.1 Hz, 1H), 2.91 (ddd, *J* = 13.9, 8.9, 5.1 Hz, 1H), 2.48 – 2.26 (m, 2H), 2.24 – 2.09 (m, 1H), 1.62 – 1.48 (m, 2H), 1.44 (dd, *J* = 14.9, 6.9 Hz, 1H), 1.24 (dd, *J* = 27.0, 4.8 Hz, 14H), 0.88 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.62, 53.22, 40.01, 31.76, 30.35, 29.30, 29.19, 27.42, 26.97, 26.79, 22.61, 19.78, 14.06. HRMS (ESI) for $C_{13}H_{25}NO$ $[M+H]^+$: calc. 212.2009, Found: 212.2008. Yellow oil.

5-Methyl-1-(pentan-3-yl)pyrrolidin-2-one (1d)



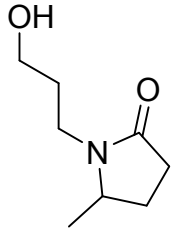
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.78 – 3.55 (m, 2H), 2.49 (ddd, *J* = 16.7, 8.9, 7.9 Hz, 1H), 2.37 – 2.26 (m, 1H), 2.22 – 2.11 (m, 1H), 1.73 – 1.49 (m, 5H), 1.24 (d, *J* = 6.3 Hz, 3H), 0.88 (dt, *J* = 26.4, 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.58, 56.54, 53.23, 30.43, 27.67, 27.18, 24.53, 21.80, 11.43, 11.38. HRMS (ESI) for $C_{10}H_{19}NO$ $[M+H]^+$: calc. 170.1539, Found: 170.1540. Yellow oil.

1-Benzhydryl-5-methylpyrrolidin-2-one (1e)



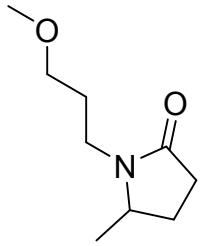
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.23 (m, 10H), 6.49 (s, 1H), 3.67 (dqd, *J* = 12.8, 6.4, 3.5 Hz, 1H), 2.71 – 2.58 (m, 1H), 2.42 (ddd, *J* = 16.8, 9.5, 4.3 Hz, 1H), 2.26 (tt, *J* = 18.7, 7.4 Hz, 1H), 1.75 – 1.64 (m, 1H), 0.90 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.11, 139.43, 138.85, 128.72, 128.65, 128.38, 128.20, 127.40, 127.33, 77.34, 77.02, 76.70, 59.65, 53.53, 30.02, 27.60, 21.23. HRMS (ESI) for $C_{18}H_{19}NO$ $[M+H]^+$: calc. 266.1539, Found: 266.1541. Colorless oil.

1-(3-Hydroxy-propyl)-5-methylpyrrolidin-2-one (1f)



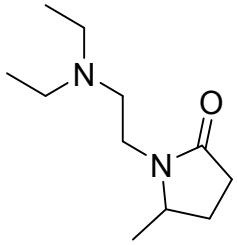
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.69 – 3.59 (m, 1H), 3.58 – 3.41 (m, 3H), 3.27 (dt, *J* = 14.3, 5.8 Hz, 1H), 2.51 – 2.28 (m, 2H), 2.25 – 2.13 (m, 1H), 1.70 – 1.53 (m, 3H), 1.22 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.46, 58.16, 54.40, 36.51, 30.38, 30.07, 27.01, 20.06. HRMS (ESI) for $C_8H_{15}NO_2$ $[M+H]^+$: calc. 158.1176, Found: 158.1177. Colorless oil.

1-(3-Methoxypropyl)-5-methylpyrrolidin-2-one (1g)



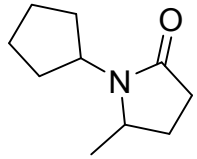
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.70 (dd, *J* = 13.0, 6.4 Hz, 1H), 3.66 – 3.57 (m, 1H), 3.39 (t, *J* = 6.3 Hz, 2H), 3.33 (s, 3H), 3.10 – 3.02 (m, 1H), 2.47 – 2.26 (m, 2H), 2.25 – 2.13 (m, 1H), 1.80 (dtd, *J* = 21.3, 14.0, 7.2 Hz, 2H), 1.64 – 1.53 (m, 1H), 1.22 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.84, 70.31, 58.59, 53.60, 37.42, 30.24, 27.72, 26.81, 19.77. HRMS (ESI) for $C_9H_{17}NO_2$ $[M+H]^+$: calc. 172.1332, Found: 172.1335. Colorless oil.

1-(2-(Diethylamino)ethyl)-5-methylpyrrolidin-2-one (1h)



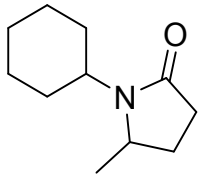
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.83 – 3.72 (m, 1H), 3.70 – 3.59 (m, 1H), 3.03 (ddd, *J* = 13.9, 8.1, 6.1 Hz, 1H), 2.64 – 2.46 (m, 6H), 2.42 – 2.26 (m, 2H), 2.23 – 2.13 (m, 1H), 1.56 (ddt, *J* = 9.5, 7.5, 6.6 Hz, 1H), 1.21 (d, *J* = 6.3 Hz, 3H), 1.02 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.72, 77.34, 77.02, 76.70, 53.86, 50.40, 47.21, 38.30, 30.24, 26.88, 19.87, 11.94. HRMS (ESI) for $C_{11}H_{22}N_2O$ $[M+H]^+$: calc. 199.18049, Found: 199.18049. Colorless oil.

1-Cyclopentyl-5-methylpyrrolidin-2-one (1i)



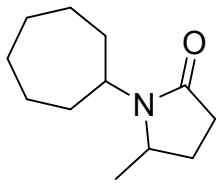
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 – 3.96 (m, 1H), 3.83 – 3.67 (m, 1H), 2.51 – 2.40 (m, 1H), 2.28 (ddd, *J* = 16.6, 9.7, 4.3 Hz, 1H), 2.20 – 2.10 (m, 1H), 1.91 (ddd, *J* = 7.7, 5.3, 3.0 Hz, 1H), 1.82 – 1.51 (m, 8H), 1.23 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.85, 54.09, 53.72, 30.44, 30.16, 28.48, 27.22, 23.78, 23.75, 21.80. HRMS (ESI) for $C_{10}H_{17}NO$ $[M+H]^+$: calc. 168.1383 ,Found: 168.1384. Yellow oil.

1-Cyclohexyl-5-methylpyrrolidin-2-one (1j)



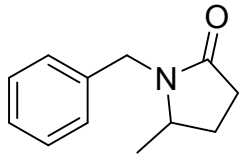
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 – 3.65 (m, 2H), 2.47 (dt, *J* = 17.7, 9.0 Hz, 1H), 2.28 (ddd, *J* = 16.6, 9.7, 4.2 Hz, 1H), 2.20 – 2.05 (m, 1H), 1.88 – 1.72 (m, 3H), 1.69 – 1.43 (m, 5H), 1.42 – 1.20 (m, 5H), 1.19 – 1.04 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.56, 52.91, 52.54, 31.90, 30.37, 30.12, 27.54, 25.99, 25.93, 25.59, 22.43. HRMS (ESI) for $C_{11}H_{19}NO$ $[M+H]^+$: calc. 182.1539 ,Found: 182.1541. Yellow oil.

1-Cycloheptyl-5-methylpyrrolidin-2-one (1k)



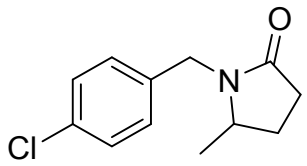
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.94 – 3.66 (m, 2H), 2.51 – 2.35 (m, 1H), 2.33 – 2.20 (m, 1H), 2.18 – 2.02 (m, 1H), 1.92 – 1.38 (m, 14H), 1.23 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.17, 54.72, 53.24, 34.33, 32.28, 30.45, 27.51, 27.42, 27.20, 25.57, 25.36, 22.18. HRMS (ESI) for $C_{12}H_{21}NO$ $[M+H]^+$: calc. 196.1696, Found: 196.1698. Yellow oil.

1-Benzyl-5-methylpyrrolidin-2-one (1l)



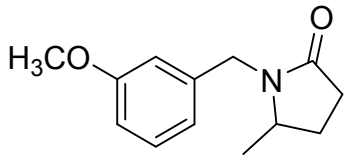
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (dt, *J* = 12.8, 7.2 Hz, 5H), 4.99 (d, *J* = 15.0 Hz, 1H), 4.01 (d, *J* = 15.0 Hz, 1H), 3.55 (dd, *J* = 12.9, 6.4 Hz, 1H), 2.60 – 2.35 (m, 2H), 2.17 (ddd, *J* = 15.9, 10.8, 7.1 Hz, 1H), 1.62 (ddd, *J* = 16.3, 13.1, 6.7 Hz, 1H), 1.18 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.96, 136.86, 128.60, 127.97, 127.39, 52.84, 43.93, 30.25, 26.67, 19.59. HRMS (ESI) for $C_{12}H_{15}NO$ $[M+H]^+$: calc. 190.1226, Found: 190.1227. Yellow oil.

1-(4-Chlorobenzyl)-5-methylpyrrolidin-2-one (1m)



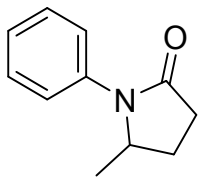
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.4 Hz, 2H), 7.18 (d, *J* = 8.3 Hz, 2H), 4.86 (d, *J* = 15.1 Hz, 1H), 4.01 (d, *J* = 15.1 Hz, 1H), 3.62 – 3.45 (m, 1H), 2.55 – 2.32 (m, 2H), 2.16 (dddd, *J* = 13.1, 9.5, 7.5, 5.8 Hz, 1H), 1.60 (dddd, *J* = 13.1, 9.5, 7.4, 6.0 Hz, 1H), 1.15 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.86, 135.40, 133.02, 129.18, 128.60, 52.82, 43.17, 30.05, 26.55, 19.52. HRMS (ESI) for $C_{12}H_{14}ClNO$ $[M+H]^+$: calc. 224.0837, Found: 224.0839. Yellow oil.

1-(3-Methoxybenzyl)-5-methylpyrrolidin-2-one (1n)



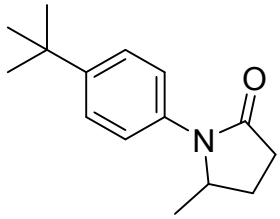
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.19 (m, 1H), 6.82 (dd, *J* = 9.0, 5.3 Hz, 3H), 4.96 (d, *J* = 15.0 Hz, 1H), 3.97 (d, *J* = 15.0 Hz, 1H), 3.81 (s, 3H), 3.56 (dd, *J* = 12.9, 6.5 Hz, 1H), 2.59 – 2.34 (m, 2H), 2.18 (dt, *J* = 9.0, 7.9 Hz, 1H), 1.62 (ddd, *J* = 15.9, 13.1, 6.6 Hz, 1H), 1.18 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.02, 159.90, 138.43, 129.61, 120.29, 113.66, 112.79, 55.23, 52.89, 43.90, 30.23, 26.66, 19.58. HRMS (ESI) for $C_{13}H_{17}NO_2$ $[M+H]^+$: calc. 220.1332, Found: 220.1332. Yellow oil.

5-Methyl-1-phenylpyrrolidin-2-one (2a)



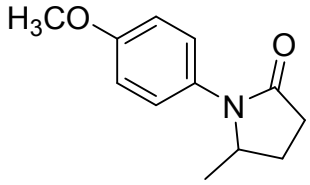
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.33 (m, 4H), 7.24 – 7.15 (m, 1H), 4.30 (dd, *J* = 13.0, 6.4 Hz, 1H), 2.68 – 2.50 (m, 2H), 2.38 (ddd, *J* = 9.9, 9.4, 5.0 Hz, 1H), 1.75 (ddd, *J* = 9.2, 4.5, 1.9 Hz, 1H), 1.21 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.18, 137.58, 128.96, 125.73, 124.03, 55.60, 31.33, 26.74, 20.15. HRMS (ESI) for $C_{11}H_{13}NO$ $[M+H]^+$: calc. 176.1070, Found: 176.1071. White solid, Mp = 47~49 °C.

1-(4-(Tert-butyl)phenyl)-5-methylpyrrolidin-2-one (2b)



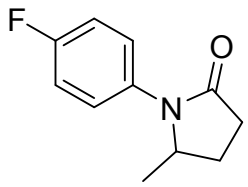
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 8.6 Hz, 2H), 7.27 (d, *J* = 8.6 Hz, 2H), 4.31 – 4.21 (m, 1H), 2.68 – 2.46 (m, 2H), 2.42 – 2.29 (m, 1H), 1.79 – 1.69 (m, 1H), 1.31 (s, 9H), 1.20 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.24, 148.67, 134.85, 125.91, 123.74, 55.75, 34.48, 31.35, 31.32, 26.81, 20.30. HRMS (ESI) for $C_{15}H_{21}NO$ $[M+H]^+$: calc. 232.1696, Found: 232.1696. White solid, Mp = 48~49 °C.

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



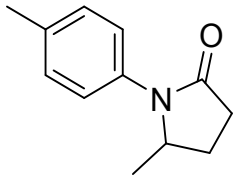
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.21 (m, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 4.20 (dd, *J* = 12.8, 6.4 Hz, 1H), 3.82 (s, 3H), 2.69 – 2.48 (m, 2H), 2.48 – 2.30 (m, 1H), 1.85 – 1.68 (m, 1H), 1.19 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.33, 157.71, 130.39, 126.08, 114.35, 56.14, 55.43, 31.11, 26.84, 20.25. HRMS (ESI) for $C_{12}H_{15}NO_2$ $[M+H]^+$: calc. 206.1176, Found: 206.1177. White solid, Mp = 59~60 °C.

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



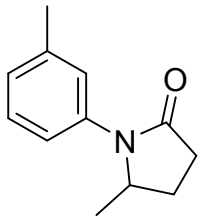
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (dd, *J* = 8.8, 4.9 Hz, 2H), 7.08 (t, *J* = 8.6 Hz, 2H), 4.23 (dd, *J* = 12.9, 6.4 Hz, 1H), 2.64 – 2.51 (m, 2H), 2.42 – 2.35 (m, 1H), 1.79 – 1.72 (m, 1H), 1.19 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.28, 161.66, 159.22, 133.51, 126.04, 125.96, 115.93, 115.71, 55.86, 31.14, 26.75, 20.13. HRMS (ESI) for $C_{11}H_{12}FNO$ $[M+H]^+$: calc. 194.0976, Found: 194.0977. White solid, Mp =74~75 °C.

5-Methyl-1-p-tolylpyrrolidin-2-one (2e)



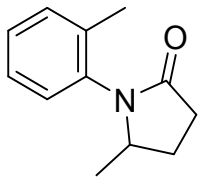
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 – 7.06 (m, 4H), 4.16 (dd, *J* = 13.1, 6.3 Hz, 1H), 2.63 – 2.39 (m, 2H), 2.34 – 2.23 (m, 4H), 1.72 – 1.61 (m, 1H), 1.11 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.27, 135.67, 134.94, 129.61, 124.27, 55.82, 31.25, 26.82, 20.97, 20.20. HRMS (ESI) for $C_{12}H_{15}NO$ $[M+H]^+$: calc. 190.1226, Found: 190.1228. White solid, Mp = 75~76 °C.

1-(3-Methylphenyl)-5-methylpyrrolidin-2-one (2f)



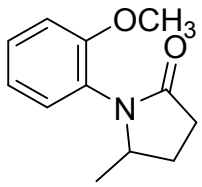
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (t, *J* = 7.7 Hz, 1H), 7.12 (s, 1H), 7.05 (d, *J* = 7.8 Hz, 1H), 6.94 (d, *J* = 7.4 Hz, 1H), 4.18 (dd, *J* = 12.7, 6.3 Hz, 1H), 2.64 – 2.38 (m, 2H), 2.37 – 2.21 (m, 4H), 1.66 (ddd, *J* = 13.0, 11.0, 7.2 Hz, 1H), 1.11 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.11, 138.75, 137.40, 128.69, 126.62, 124.98, 121.14, 55.69, 31.26, 26.70, 21.40, 20.14. HRMS (ESI) for $C_{12}H_{15}NO$ $[M+H]^+$: calc. 190.1226, Found: 190.1228. Colorless oil.

1-(2-Methylphenyl)-5-methylpyrrolidin-2-one (2g)



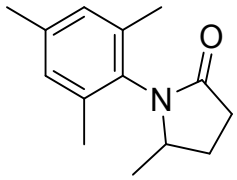
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 1H), 7.26 – 7.20 (m, 2H), 7.10 – 7.04 (m, 1H), 4.08 (d, *J* = 4.6 Hz, 1H), 2.67 – 2.51 (m, 2H), 2.48 – 2.37 (m, 1H), 2.24 (s, 3H), 1.87 – 1.75 (m, 1H), 1.13 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.19, 136.32, 136.00, 131.05, 127.75, 126.59, 56.82, 30.80, 27.75, 20.20, 18.00. HRMS (ESI) for $C_{12}H_{15}NO$ $[M+H]^+$: calc. 190.1226, Found: 190.1227. Colorless oil.

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



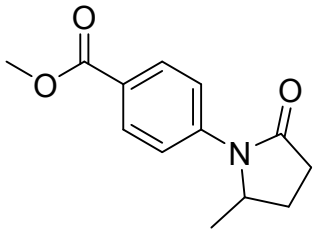
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.24 (m, 1H), 7.15 (dd, *J* = 7.6, 1.1 Hz, 1H), 7.04 – 6.91 (m, 2H), 4.17 (h, *J* = 6.5 Hz, 1H), 3.81 (s, 3H), 2.60 – 2.51 (m, 2H), 2.36 (dt, *J* = 13.5, 7.3 Hz, 1H), 1.80 – 1.66 (m, 1H), 1.07 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.99, 155.21, 130.10, 128.78, 125.72, 120.72, 111.87, 55.76, 55.55, 30.83, 27.65, 20.26. HRMS (ESI) for $C_{12}H_{15}NO_2$ $[M+H]^+$: calc. 206.1176, Found: 206.1176. Brown oil.

1-Mesityl-5-methylpyrrolidin-2-one (2i)



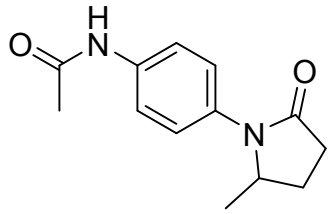
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.91 (d, *J* = 14.1 Hz, 2H), 4.03 (h, *J* = 6.5 Hz, 1H), 2.68 – 2.49 (m, 2H), 2.47 – 2.36 (m, 1H), 2.26 (s, 3H), 2.16 (d, *J* = 14.3 Hz, 6H), 1.81 (dt, *J* = 15.8, 8.5 Hz, 1H), 1.10 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.33, 137.60, 137.02, 135.11, 131.95, 129.44, 129.37, 56.19, 30.70, 28.29, 20.92, 19.62, 18.59, 18.09. HRMS (ESI) for $C_{14}H_{19}NO$ $[M+H]^+$: calc. 218.1539, Found: 218.1539. Colorless oil.

Methyl 4-(2-methyl-5-oxopyrrolidin-1-yl)benzoate (2j)



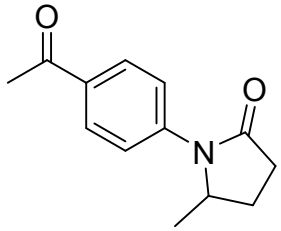
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 8.00 (m, 2H), 7.59 – 7.50 (m, 2H), 4.44 – 4.34 (m, 1H), 3.88 (s, 3H), 2.67 (ddd, *J* = 17.1, 9.4, 7.0 Hz, 1H), 2.53 (ddd, *J* = 17.2, 9.5, 6.4 Hz, 1H), 2.36 (ddt, *J* = 12.7, 9.5, 7.4 Hz, 1H), 1.76 (dddd, *J* = 12.7, 9.4, 6.4, 5.0 Hz, 1H), 1.24 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.31, 166.50, 141.86, 130.40, 126.30, 121.89, 77.34, 77.02, 76.70, 54.96, 52.01, 31.38, 26.34, 19.77. HRMS (ESI) for $C_{13}H_{15}NO_3$ $[M+H]^+$: calc. 234.1125, Found: 234.1127. Pale yellow solid, Mp = 59~60 °C.

N-(4-(2-Methyl-5-oxopyrrolidin-1-yl)phenyl)acetamide (2k)



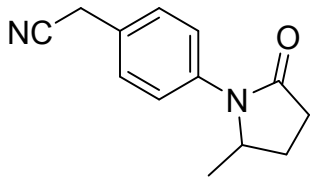
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 (s, 1H), 7.39 (d, *J* = 8.5 Hz, 2H), 7.15 (d, *J* = 8.6 Hz, 2H), 4.27 – 4.14 (m, 1H), 2.69 – 2.48 (m, 2H), 2.38 (ddd, *J* = 15.9, 13.2, 6.8 Hz, 1H), 2.10 (s, 3H), 1.75 (ddd, *J* = 16.1, 13.2, 7.7 Hz, 1H), 1.16 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.70, 168.81, 136.41, 132.87, 125.17, 120.77, 77.34, 77.02, 76.70, 56.29, 31.27, 26.79, 24.20, 20.20. HRMS (ESI) for $C_{13}H_{16}N_2O_2$ $[M+Na]^+$: calc. 255.1104, Found: 255.1102. White solid, Mp = 166~167 °C.

1-(4-Acetylphenyl)-5-methylpyrrolidin-2-one (2l)



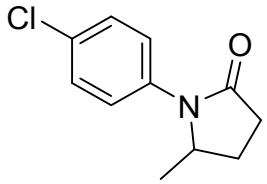
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.7 Hz, 2H), 7.58 (d, *J* = 8.7 Hz, 2H), 4.47 – 4.36 (m, 1H), 2.74 – 2.62 (m, 1H), 2.60 – 2.49 (m, 4H), 2.38 (ddt, *J* = 12.8, 9.3, 7.4 Hz, 1H), 1.84 – 1.73 (m, 1H), 1.26 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 197.02, 174.34, 142.05, 133.45, 129.27, 121.94, 77.34, 77.02, 76.70, 54.93, 31.40, 26.47, 26.37, 19.81. HRMS (ESI) for $C_{13}H_{15}NO_2$ $[M+H]^+$: calc. 218.1176, Found: 218.1175. Colorless oil.

2-(4-(2-Methyl-5-oxopyrrolidin-1-yl)phenyl)acetonitrile (2m)



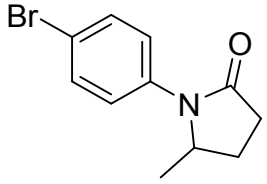
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.5 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 4.36 – 4.26 (m, 1H), 3.74 (s, 2H), 2.72 – 2.60 (m, 1H), 2.60 – 2.49 (m, 1H), 2.38 (ddt, *J* = 13.6, 9.3, 7.0 Hz, 1H), 1.77 (tdd, *J* = 12.8, 9.4, 7.0 Hz, 1H), 1.22 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.28, 137.56, 128.58, 126.93, 124.25, 117.71, 77.34, 77.02, 76.71, 55.38, 31.30, 26.65, 23.19, 20.04. HRMS (ESI) for C13H14N2O $[M+H]^+$: calc. 215.1179, Found: 215.1178. Light yellow oil.

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



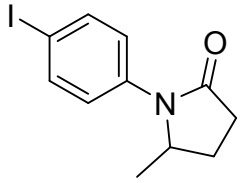
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (s, 4H), 4.28 (dd, *J* = 12.9, 6.4 Hz, 1H), 2.72 – 2.46 (m, 2H), 2.42 – 2.29 (m, 1H), 1.79 – 1.69 (m, 1H), 1.21 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.21, 136.19, 130.89, 129.07, 124.93, 55.44, 31.26, 26.62, 20.02. HRMS (ESI) for $C_{11}H_{12}ClNO$ $[M+H]^+$: calc. 210.0680, Found: 210.0682. Pale yellow solid, Mp = 68~69 °C.

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



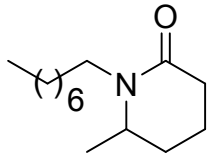
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.7 Hz, 2H), 7.19 – 7.13 (m, 2H), 4.24 – 4.08 (m, 1H), 2.51 (ddd, *J* = 16.0, 9.4, 6.4 Hz, 1H), 2.45 – 2.34 (m, 1H), 2.31 – 2.19 (m, 1H), 1.69 – 1.56 (m, 1H), 1.08 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.15, 136.64, 131.98, 125.16, 118.66, 55.32, 31.23, 26.55, 19.95. HRMS (ESI) for $C_{11}H_{12}BrNO$ $[M+H]^+$: calc. 254.0175, Found: 254.0174. Pale yellow solid, Mp = 57~58 °C.

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



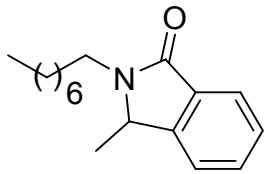
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.66 (m, 2H), 7.20 – 7.13 (m, 2H), 4.35 – 4.26 (m, 1H), 2.71 – 2.51 (m, 2H), 2.45 – 2.33 (m, 1H), 1.77 (dddd, *J* = 12.8, 9.5, 7.1, 5.6 Hz, 1H), 1.21 (d, *J* = 6.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.56, 138.05, 137.14, 125.62, 90.04, 77.34, 77.02, 76.71, 55.64, 31.40, 26.58, 19.97. HRMS (ESI) for $C_{11}H_{12}INO$ $[M+H]^+$: calc. 302.0036, Found: 302.0038. Dark brown oil.

6-Methyl-1-octylpiperidin-2-one



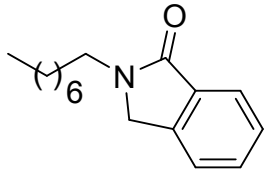
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.74 (ddd, *J* = 13.5, 9.7, 6.1 Hz, 1H), 3.53 (dd, *J* = 10.5, 4.3 Hz, 1H), 2.94 – 2.83 (m, 1H), 2.35 (dd, *J* = 8.5, 5.9 Hz, 2H), 1.94 – 1.80 (m, 2H), 1.75 – 1.43 (m, 4H), 1.25 (dd, *J* = 21.2, 5.3 Hz, 13H), 0.88 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.69, 51.82, 45.07, 32.14, 31.79, 30.17, 29.38, 29.23, 27.65, 27.09, 22.61, 19.96, 17.42, 14.06. HRMS (ESI) for $C_{14}H_{27}NO$ $[M+H]^+$: calc. 226.2165, Found: 226.2167. Yellow oil.

3-Methyl-2-octylisoindolin-1-one



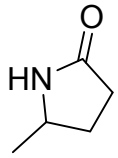
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 1H), 7.46 (t, *J* = 7.4 Hz, 1H), 7.36 (dd, *J* = 15.6, 7.6 Hz, 2H), 4.48 (q, *J* = 6.7 Hz, 1H), 3.94 – 3.81 (m, 1H), 3.13 (ddd, *J* = 13.9, 8.7, 5.1 Hz, 1H), 1.66 – 1.46 (m, 2H), 1.39 (d, *J* = 6.7 Hz, 3H), 1.32 – 1.12 (m, 11H), 0.80 (t, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.85, 146.88, 132.18, 131.18, 128.01, 123.56, 121.79, 55.33, 39.82, 31.78, 29.31, 29.18, 28.50, 27.01, 22.61, 18.16, 14.04. HRMS (ESI) for $C_{17}H_{25}NO$ $[M+H]^+$: calc. 260.2009, Found: 260.2010. Colorless oil.

2-Octylisoindolin-1-one



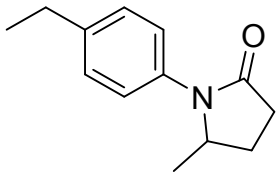
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.4 Hz, 1H), 7.36 (dd, *J* = 15.5, 7.6 Hz, 2H), 4.47 (q, *J* = 6.6 Hz, 1H), 3.97 – 3.80 (m, 1H), 3.13 (ddd, *J* = 13.8, 8.5, 5.2 Hz, 1H), 1.70 – 1.44 (m, 3H), 1.39 (d, *J* = 6.7 Hz, 3H), 1.23 (dd, *J* = 21.2, 8.0 Hz, 11H), 0.79 (t, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.85, 146.88, 132.18, 131.18, 128.01, 123.56, 121.79, 77.33, 77.01, 76.69, 55.33, 39.82, 31.78, 29.31, 29.18, 28.50, 27.01, 22.61, 18.16, 14.04. HRMS (ESI) for $C_{17}H_{25}NO$ $[M+H]^+$: calc. 246.1852, Found: 246.1854. Colorless oil.

5-Methylpyrrolidin-2-one



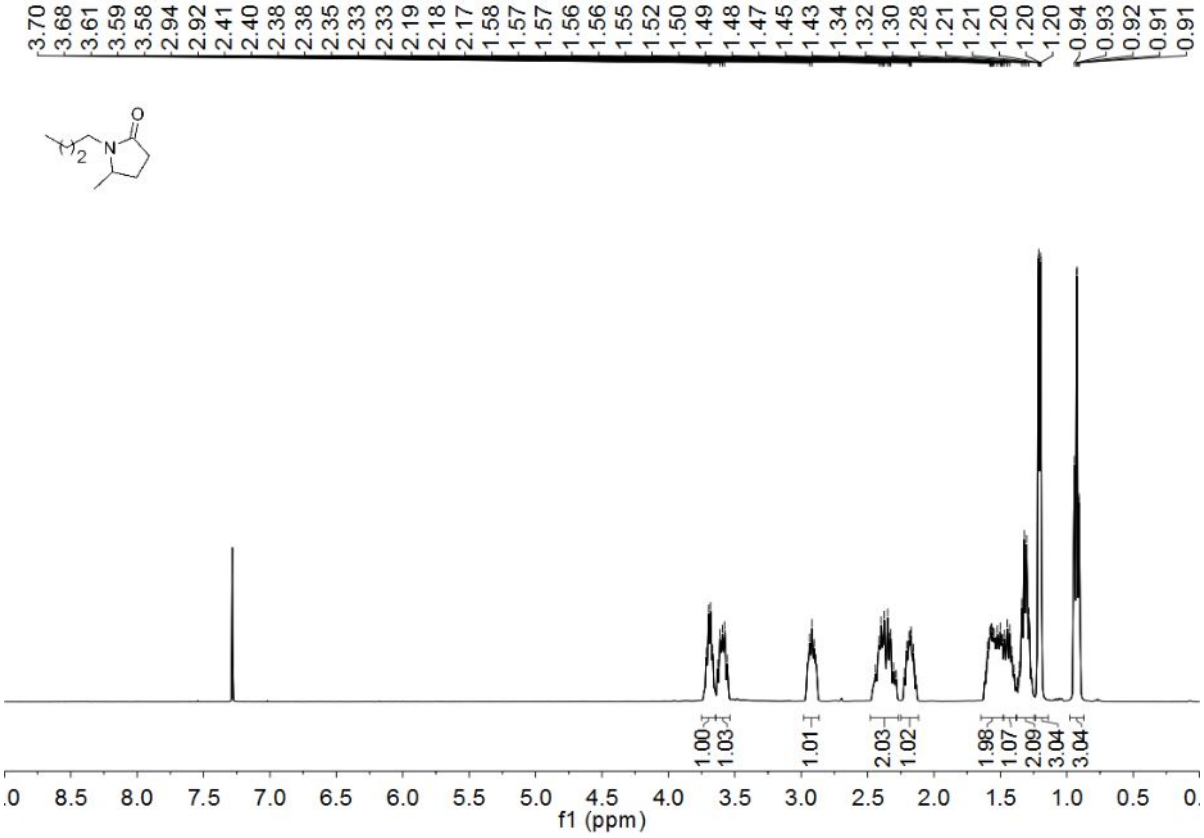
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (s, 1H), 3.86 – 3.73 (m, 1H), 2.41 – 2.30 (m, 2H), 2.30 – 2.21 (m, 1H), 1.72 – 1.57 (m, 1H), 1.23 (d, *J* = 6.3 Hz, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 178.50, 77.34, 77.02, 76.70, 50.01, 30.54, 28.83, 21.85. HRMS (ESI) for C_5H_9NO $[M+Na]^+$: calc. 122.0576, Found: 122.0577. Colorless oil.

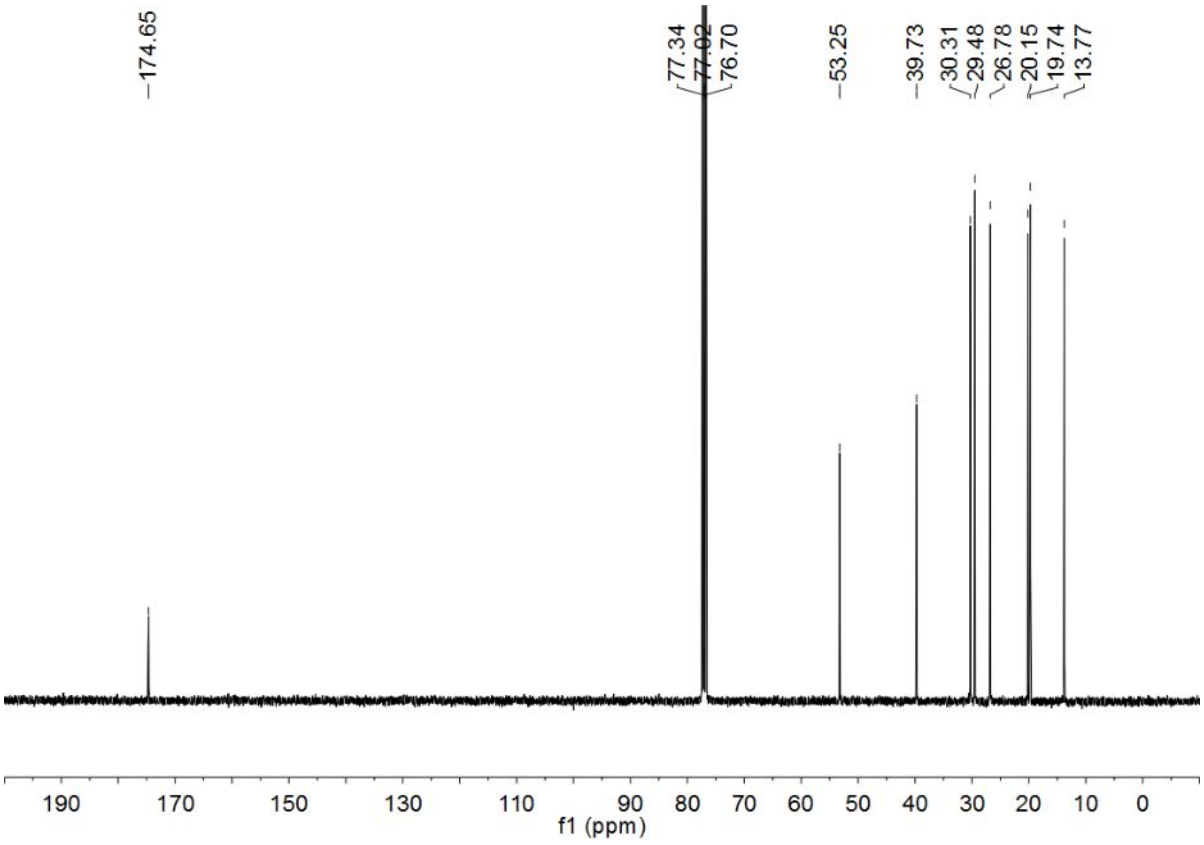
1-(4-ethylphenyl)-5-methylpyrrolidin-2-one

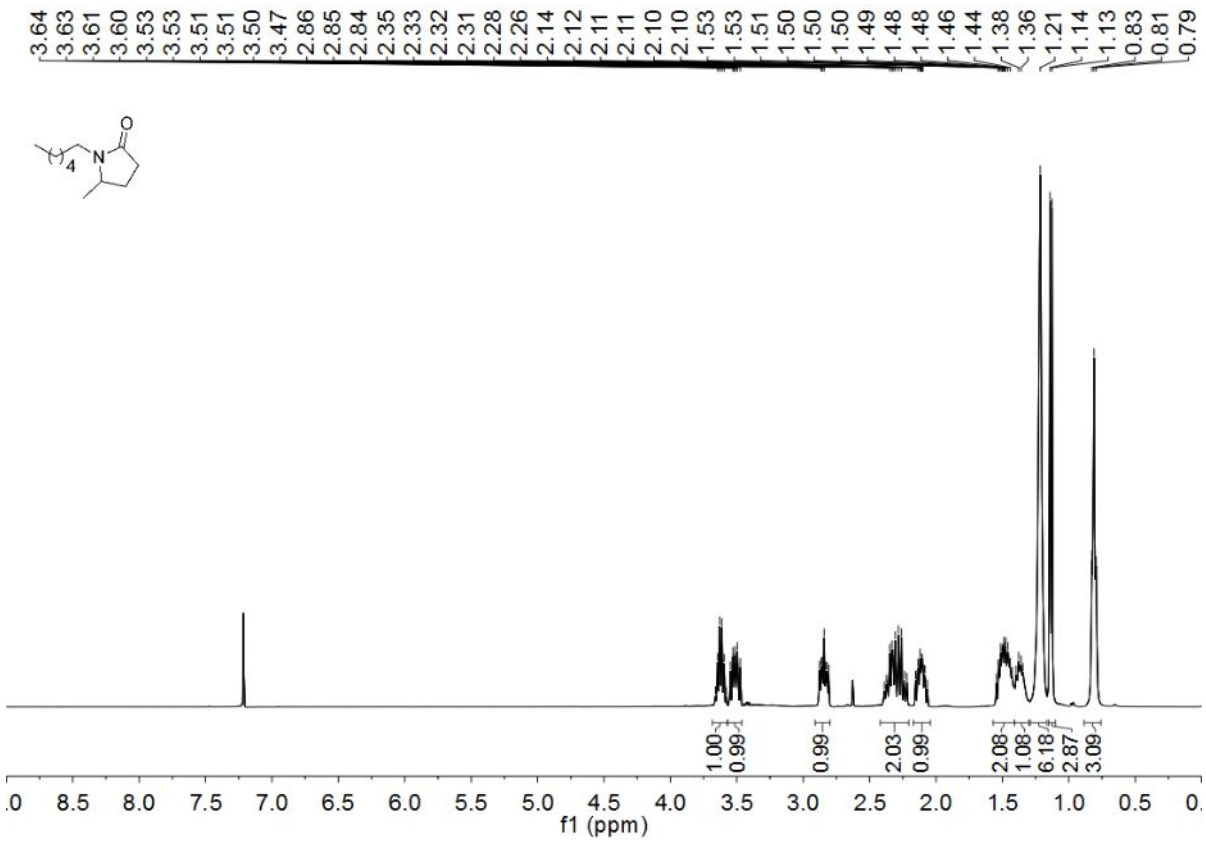


1H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, J = 8.4 Hz, 2H), 7.20 (d, J = 8.4 Hz, 2H), 4.29 – 4.18 (m, 1H), 2.72 – 2.56 (m, 3H), 2.54 – 2.43 (m, 1H), 2.42 – 2.23 (m, 1H), 1.81 – 1.63 (m, 1H), 1.27 – 1.13 (m, 6H). 13C NMR (101 MHz, $CDCl_3$) δ 173.91 (s), 141.58 (s), 134.94 (s), 128.15 (s), 123.96 (s), 77.34 (s), 77.02 (s), 76.70 (s), 55.51 (s), 31.06 (s), 28.16 (s), 26.55 (s), 19.98 (s), 15.27 (s). HRMS (ESI) for $C_{13}H_{18}NO$ $[M+H]^+$: calc. 204.1386, Found: 204.1385. Colorless oil.

^{1}H and ^{13}C NMR spectra of products



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2.35
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2.32
2.31
2.28
2.26
2.14
2.12
2.11
2.11
2.10
2.10
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1.53
1.51
1.50
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1.49
1.48
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0.79
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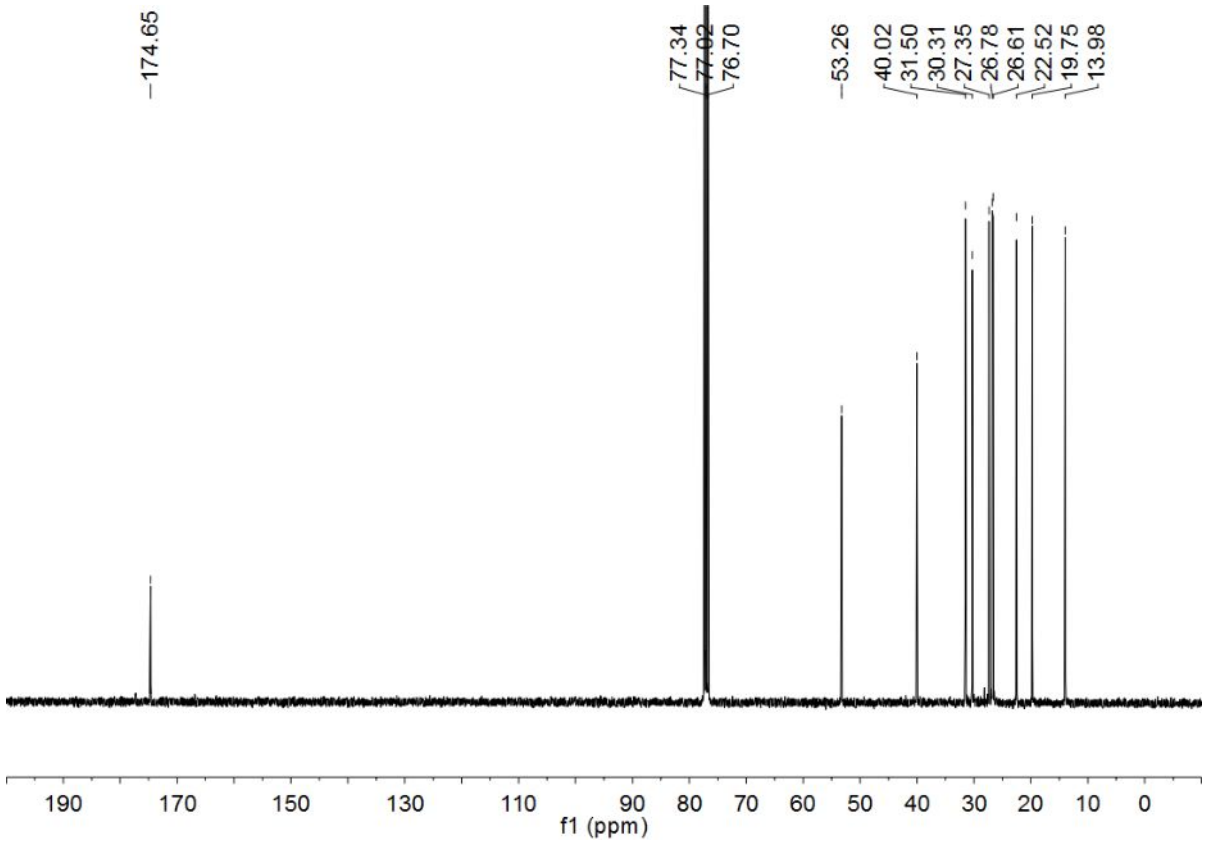
f1 (ppm)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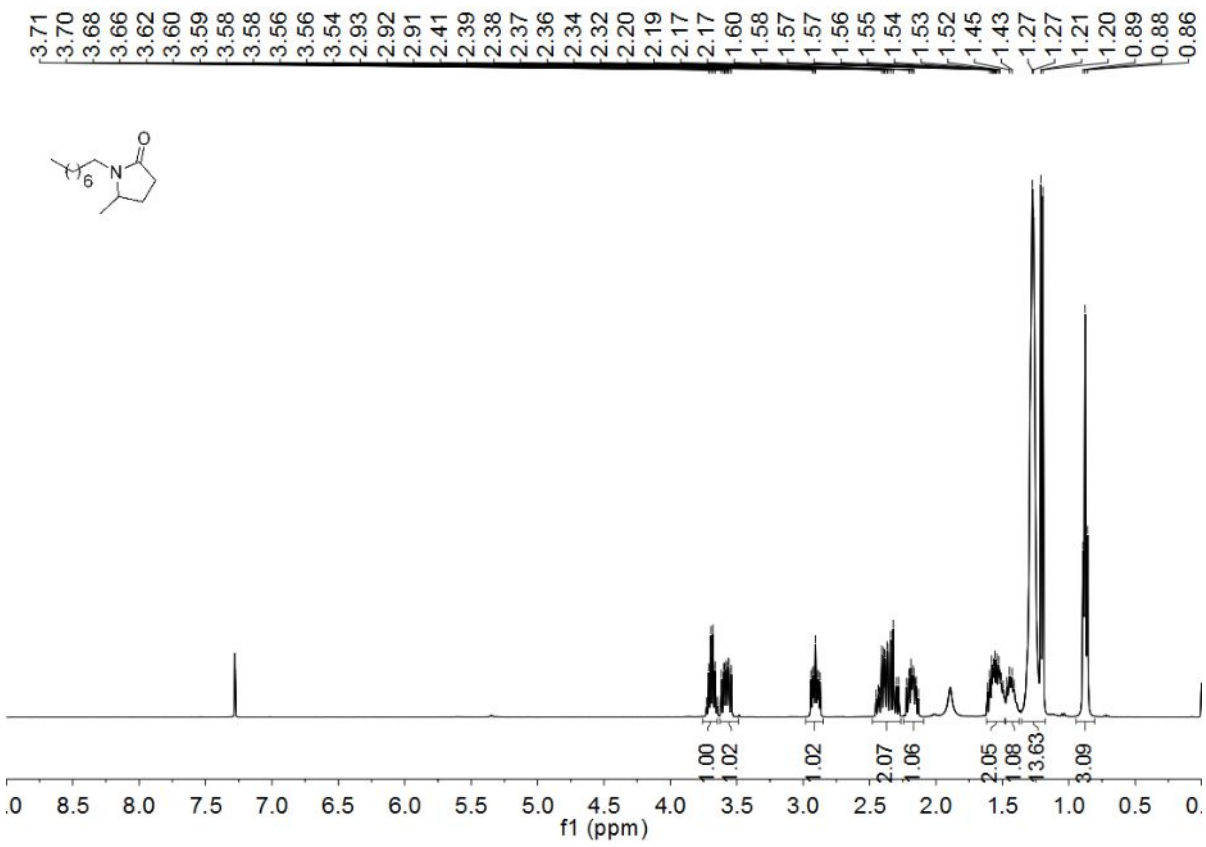
13.98
f1 (ppm)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2.92
2.91
2.41
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2.38
2.37
2.36
2.34
2.32
2.20
2.19
2.17
2.17
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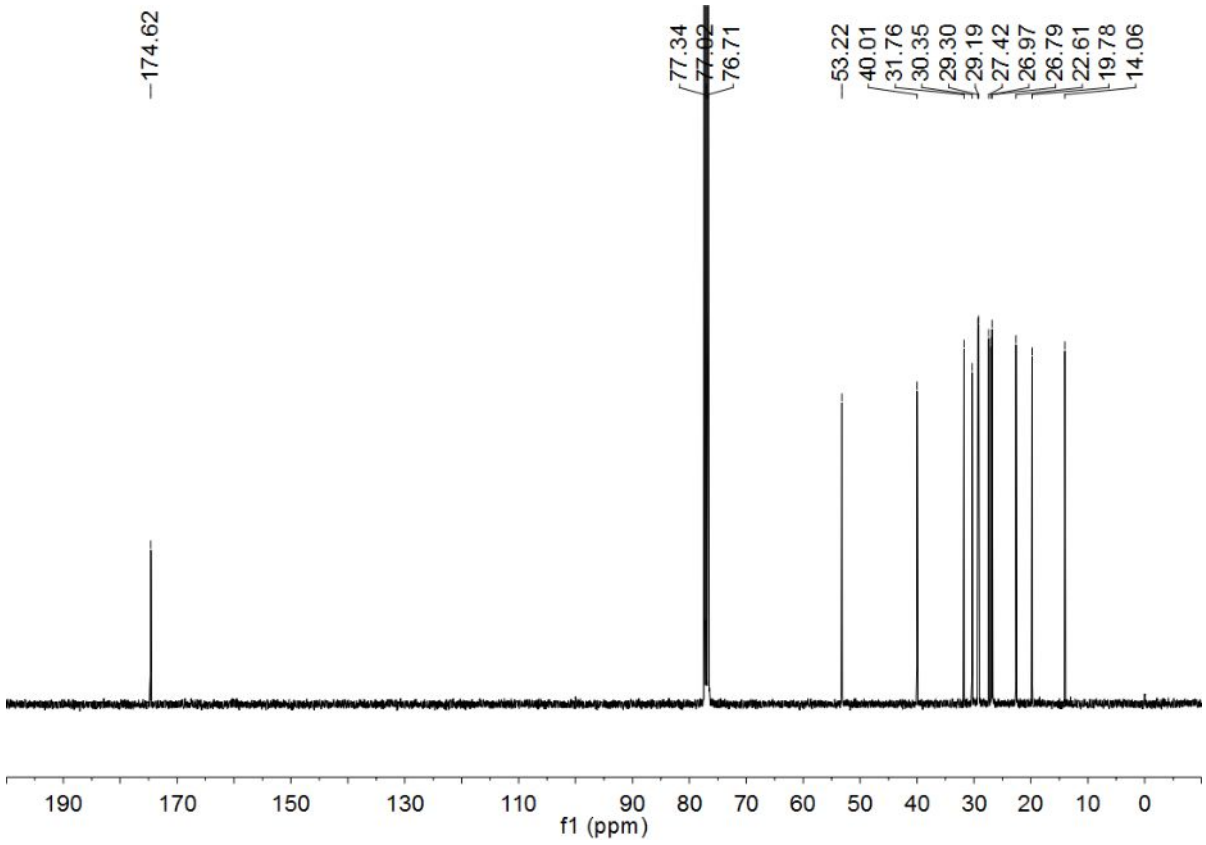
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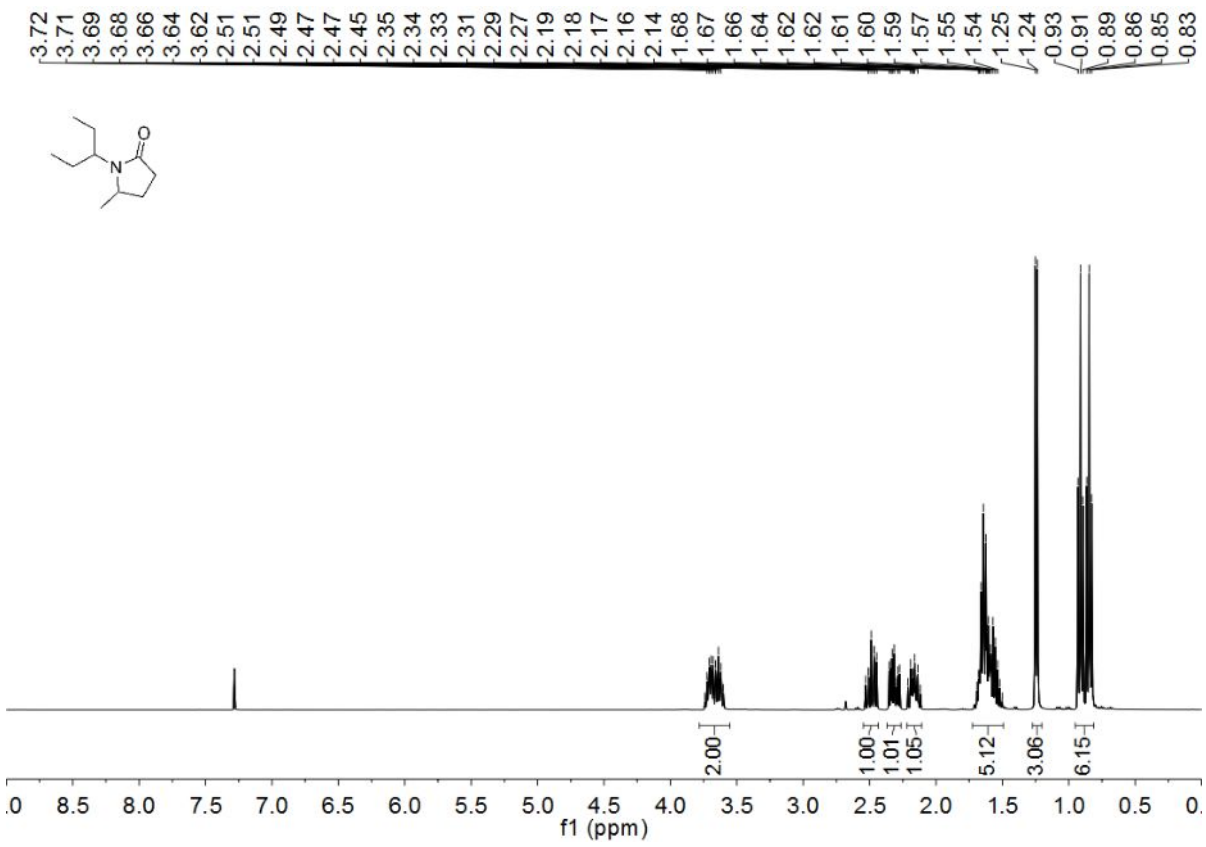
14.06
f1 (ppm)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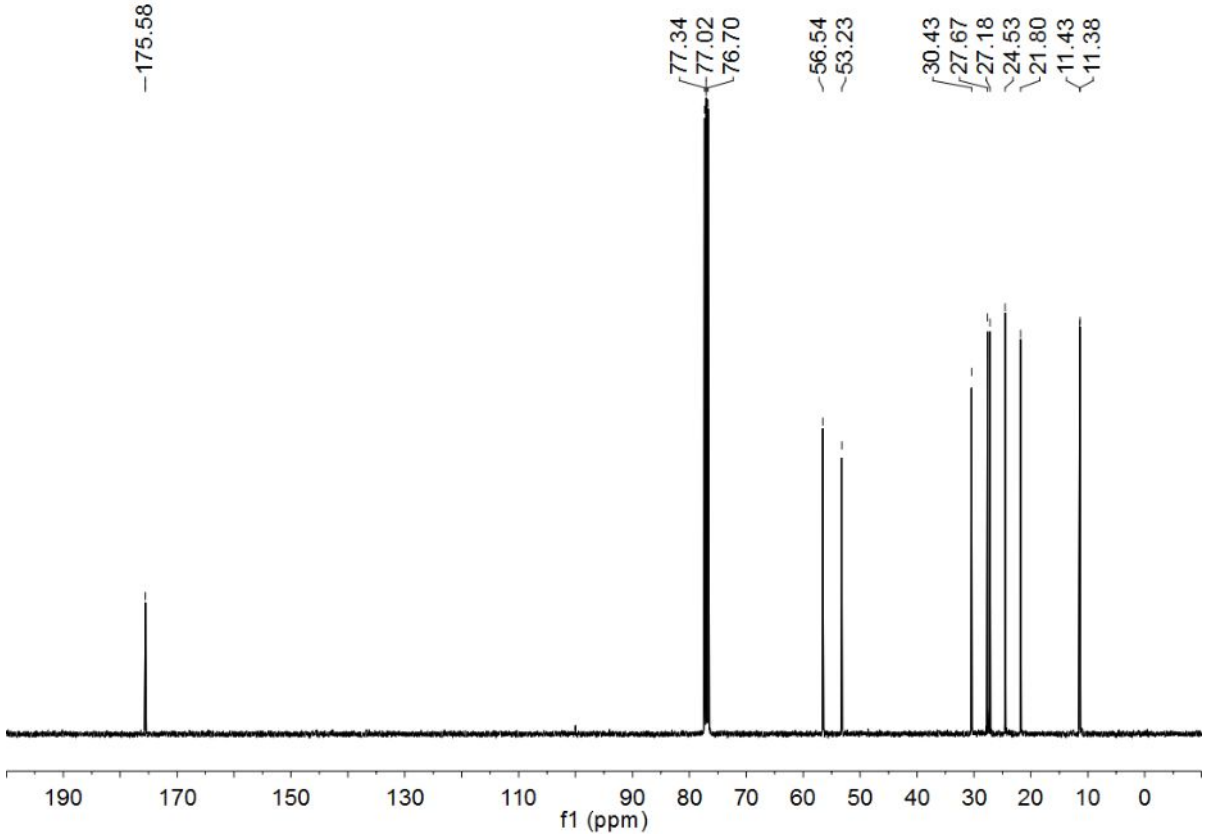
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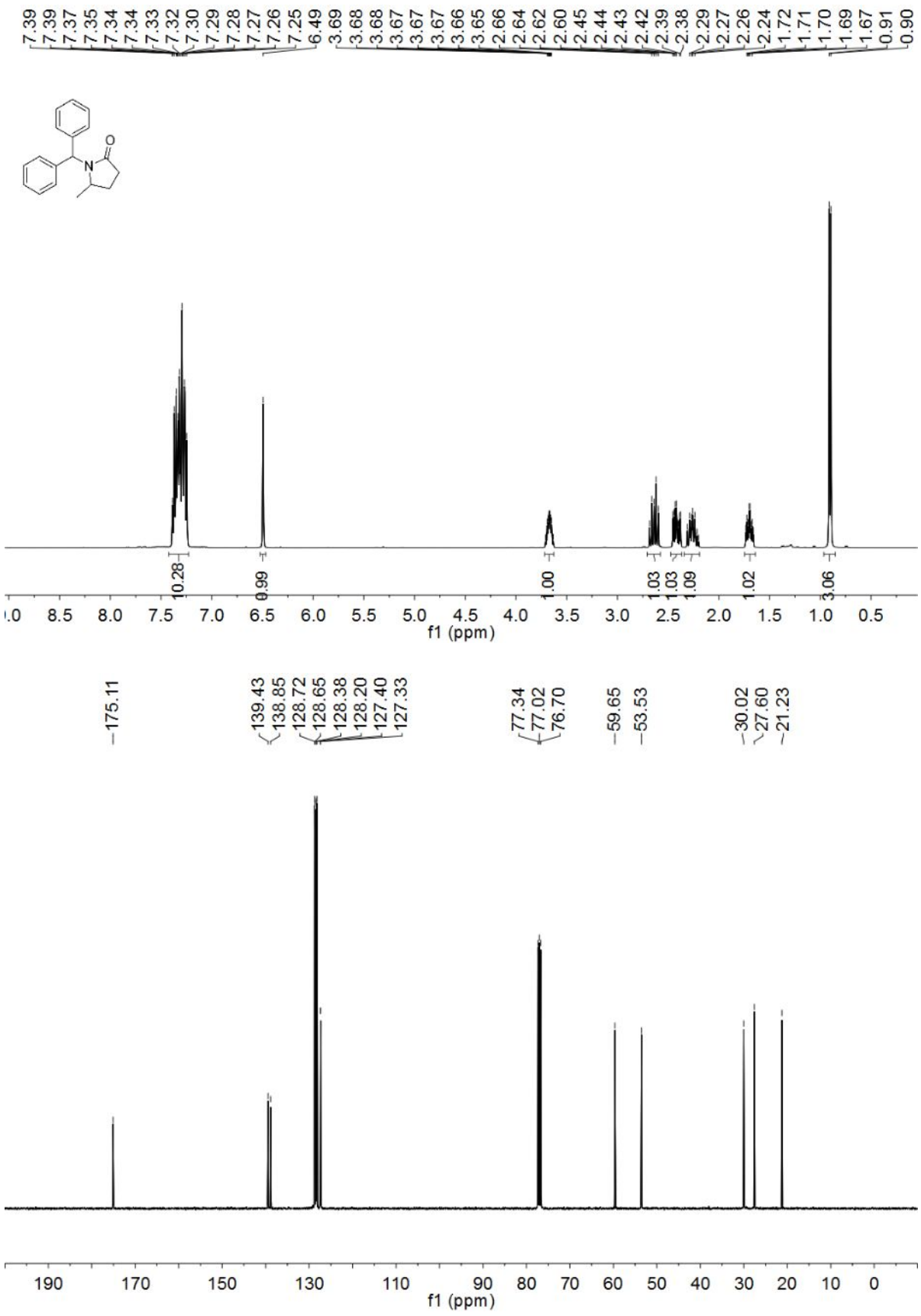
f1 (ppm)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1.02
3.06
f1 (ppm)
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128.65
128.38
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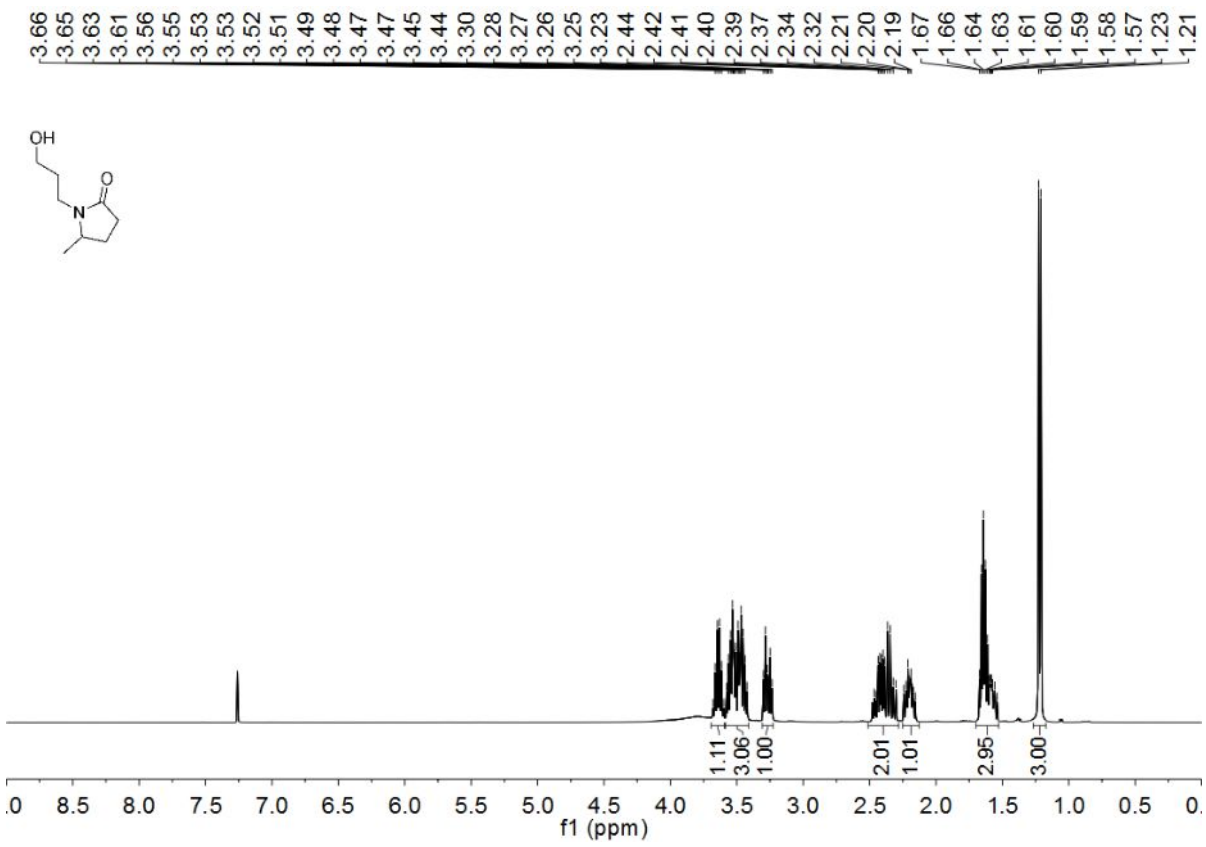
76.70
59.65
53.53
30.02
27.60
21.23
f1 (ppm)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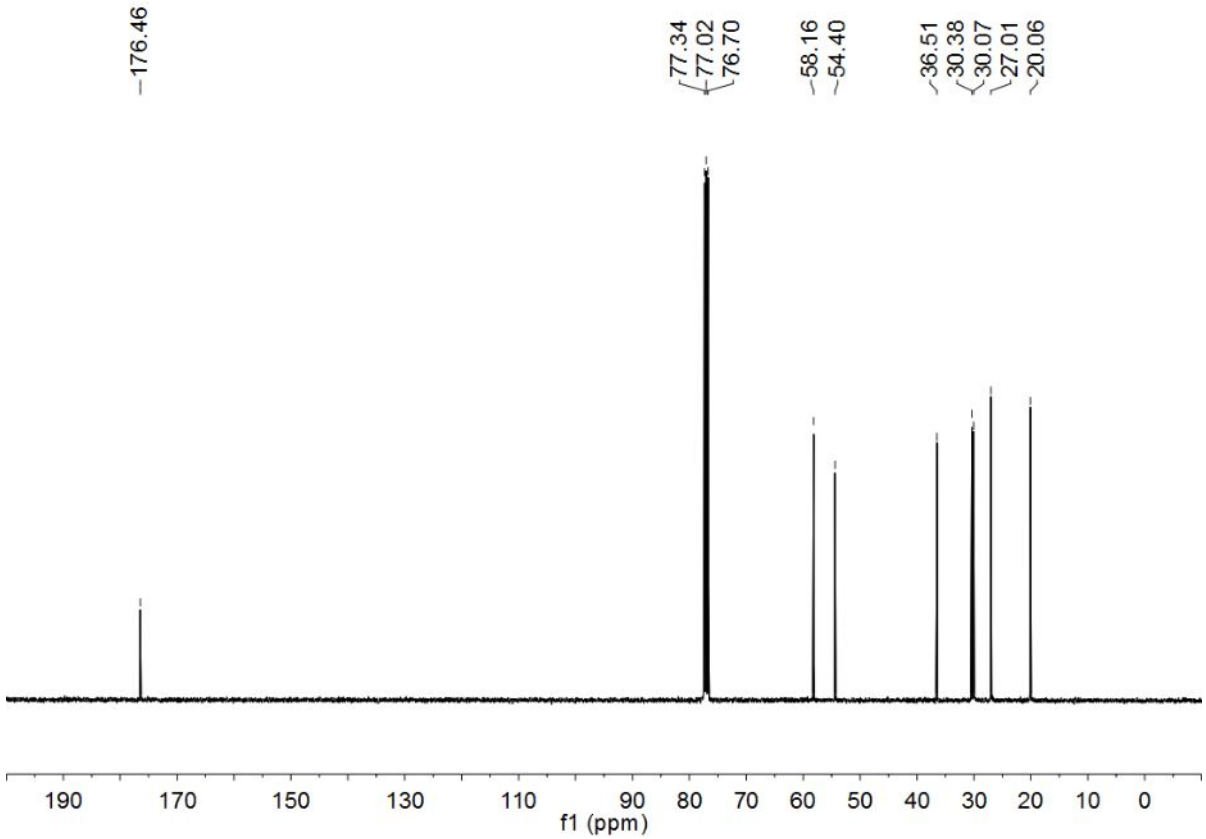
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50
40
30
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10
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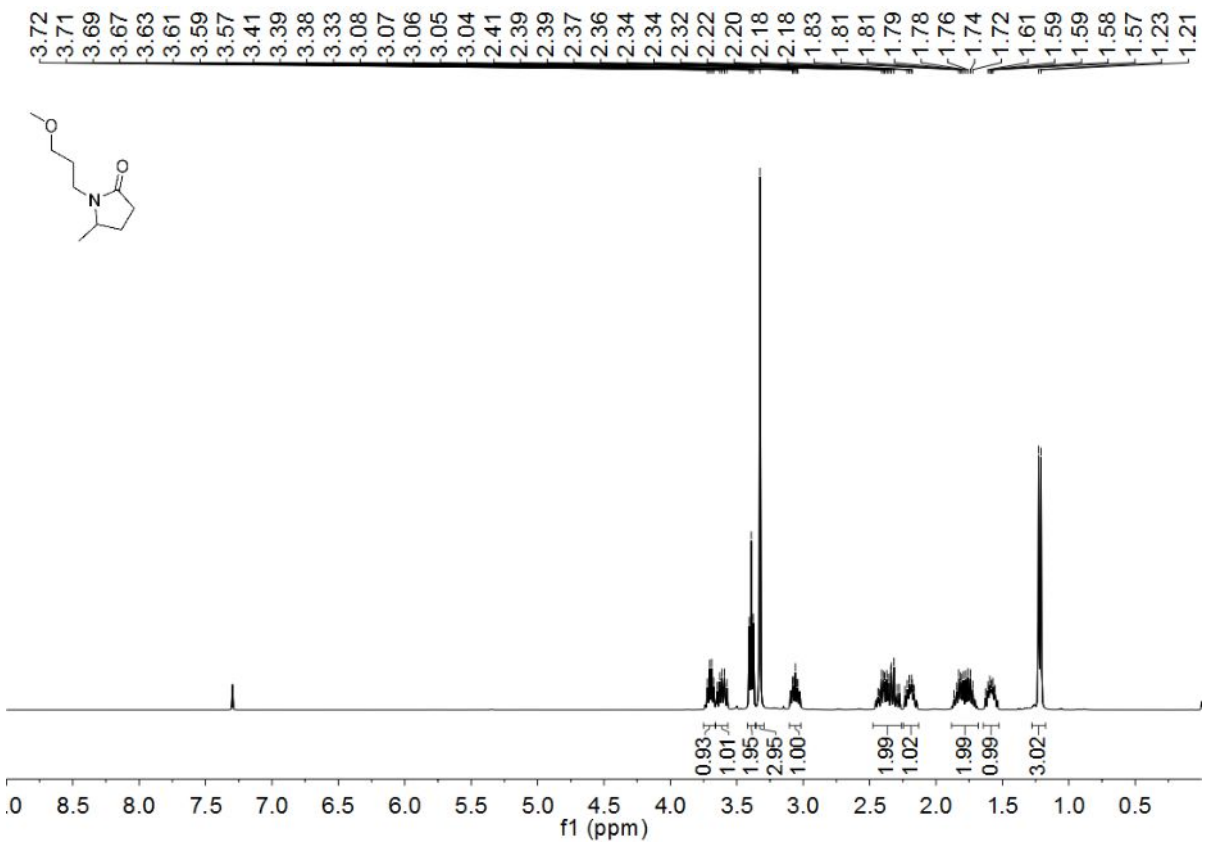
f1 (ppm)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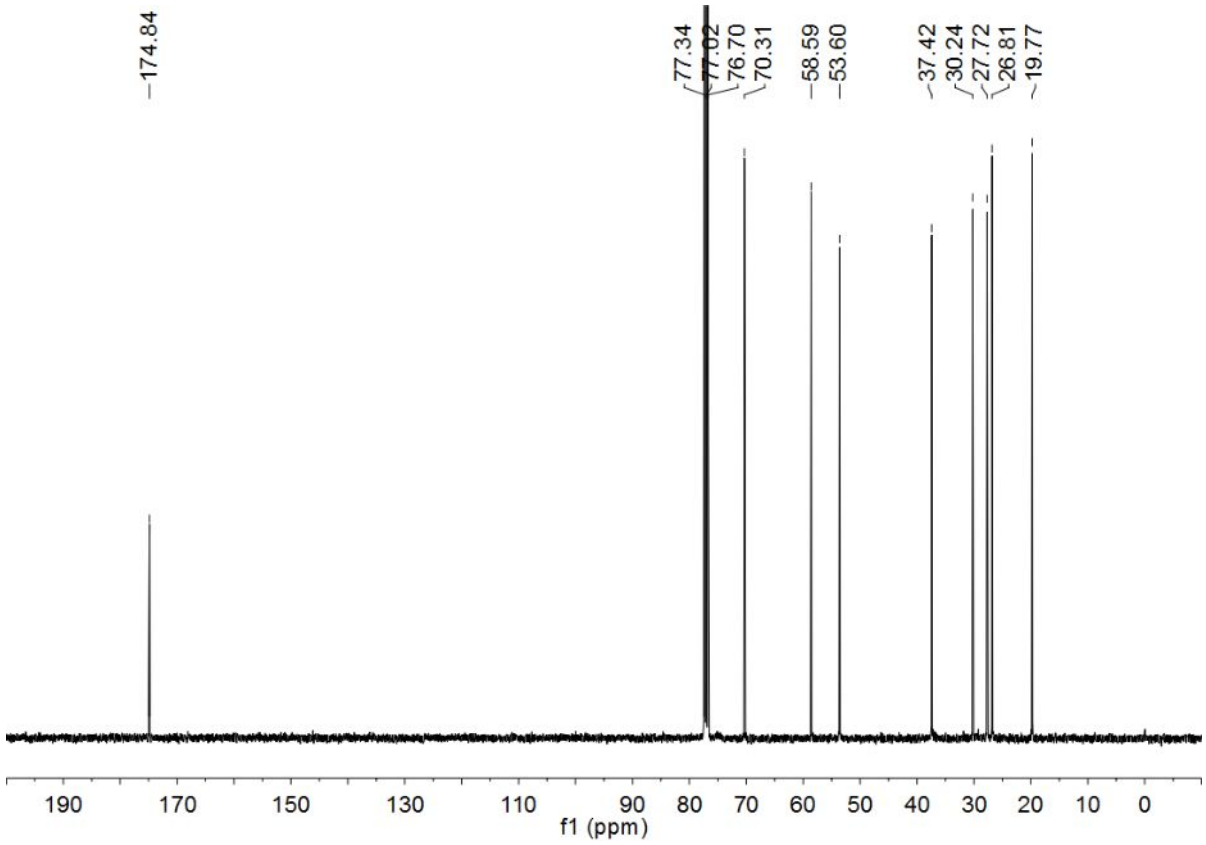
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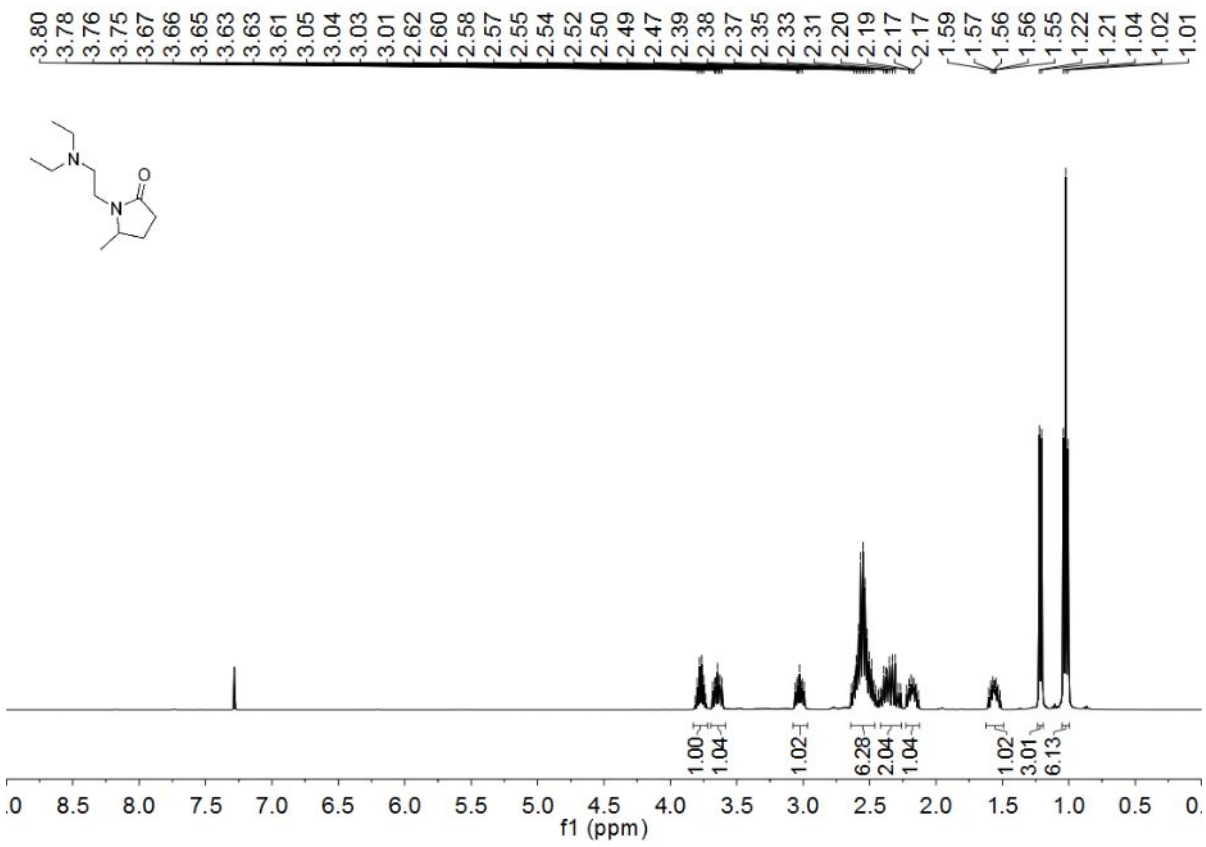
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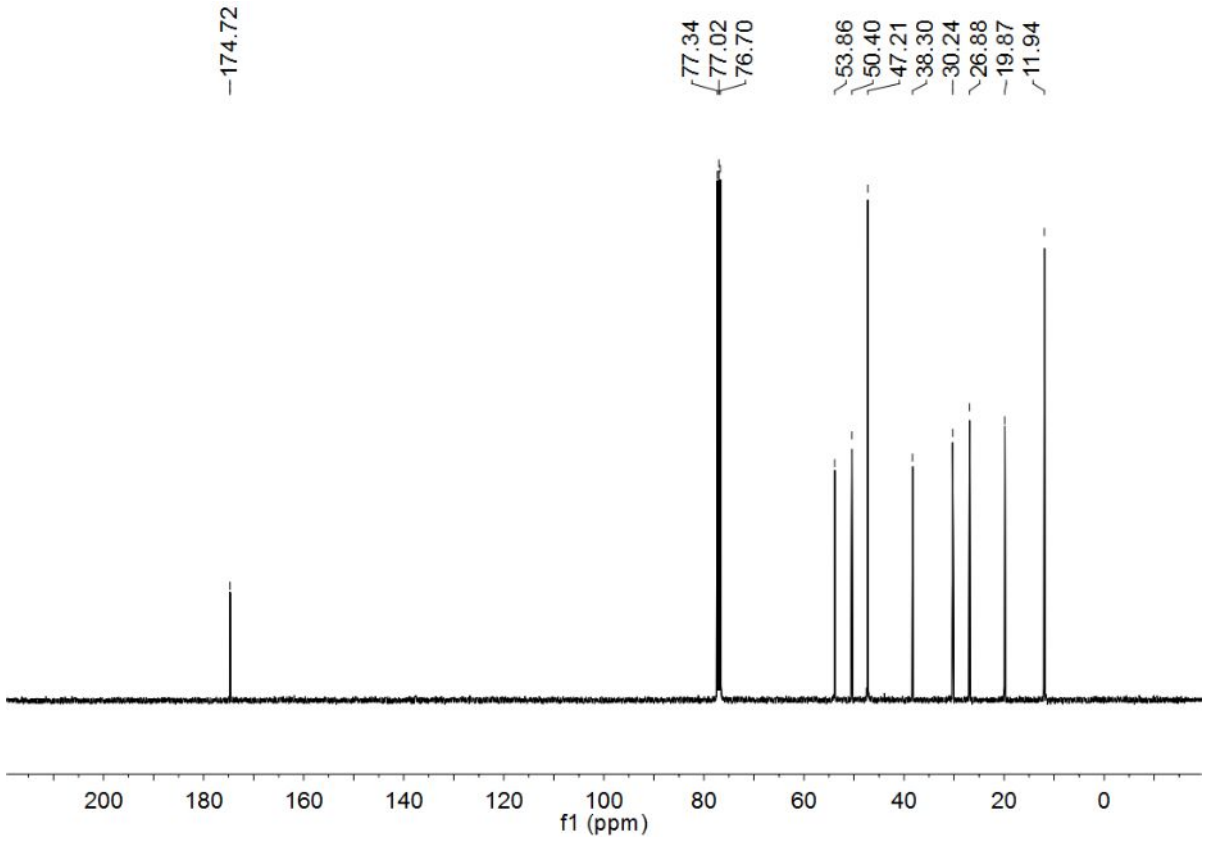
f1 (ppm)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100
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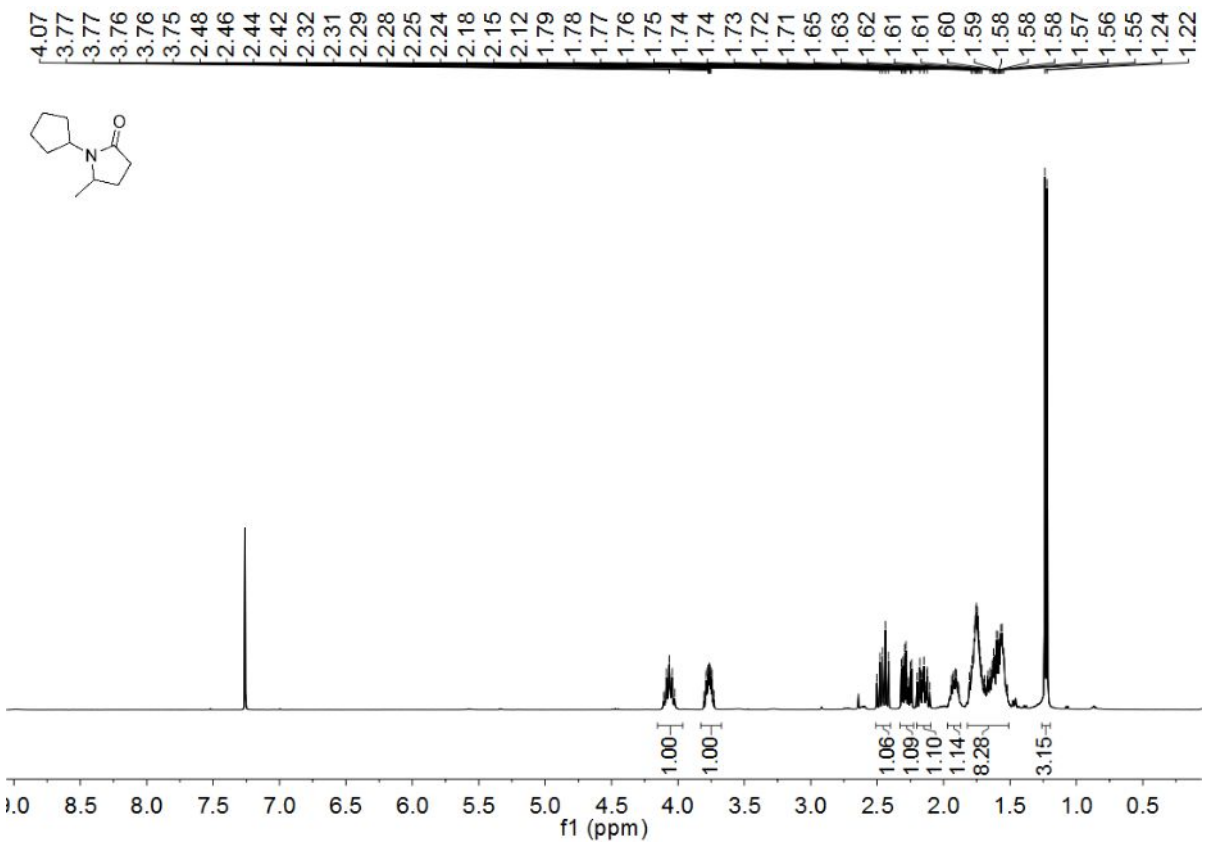
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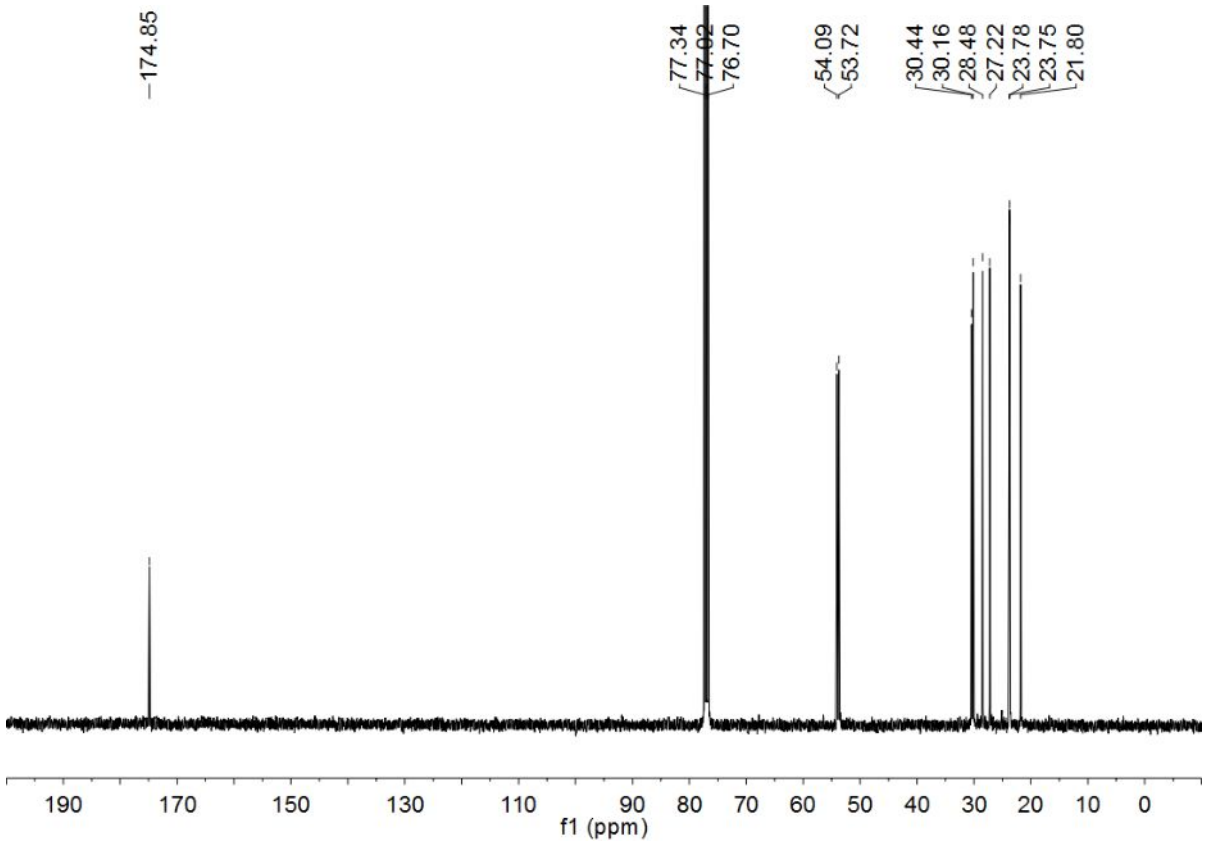
f1 (ppm)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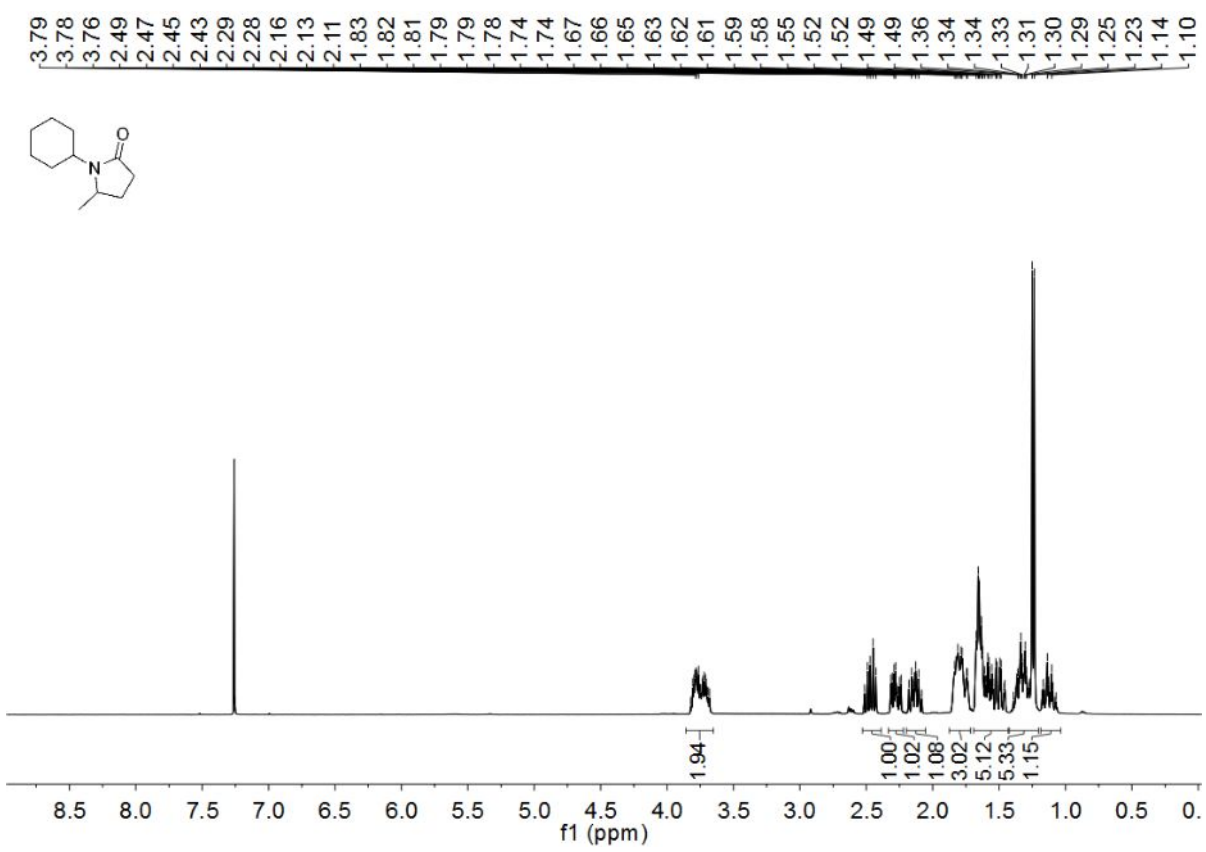
54.09 53.72
30.44 30.16 28.48 27.22 23.78 23.75 21.80
f1 (ppm)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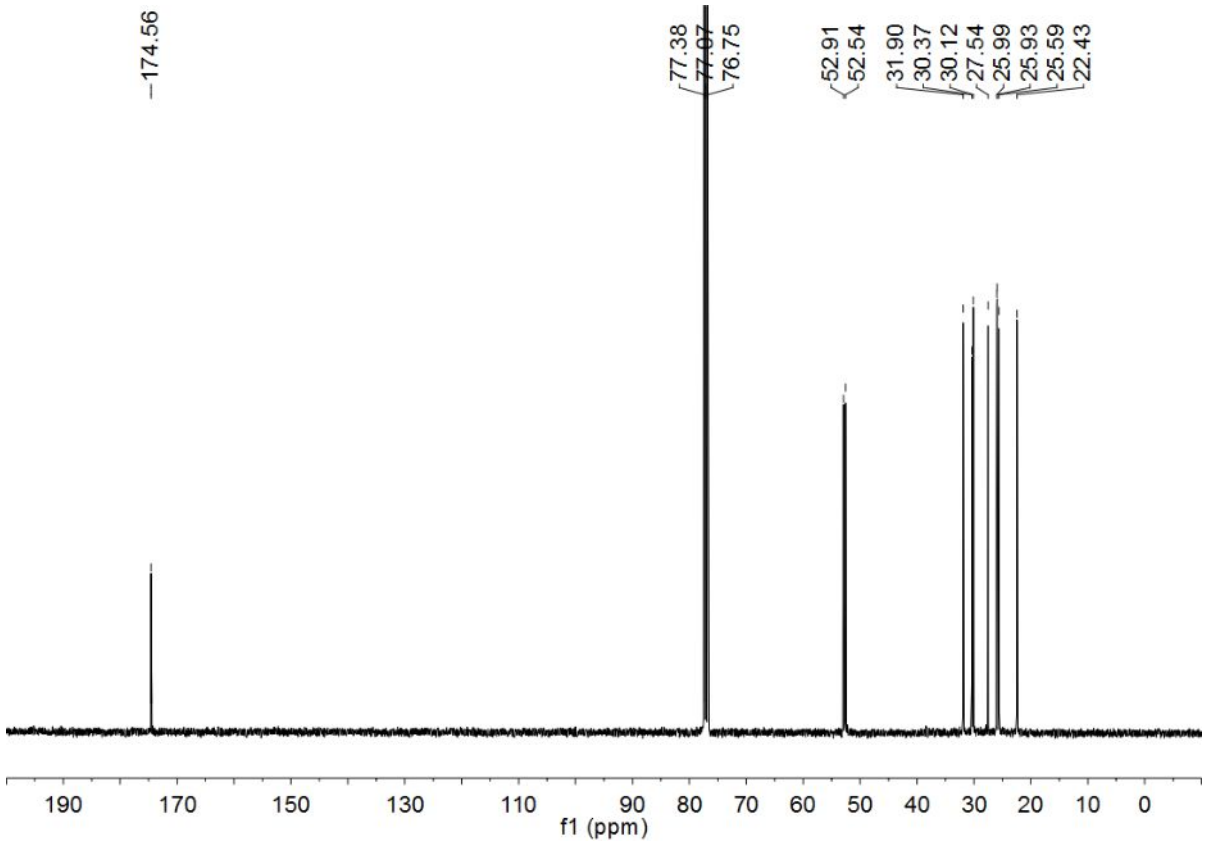
O
N
1.94 1.00 1.02 1.08 3.02 5.12 5.33 1.15
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)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77.38 77.07 76.75
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31.90 30.37 30.12 27.54 25.99 25.93 25.59 22.43
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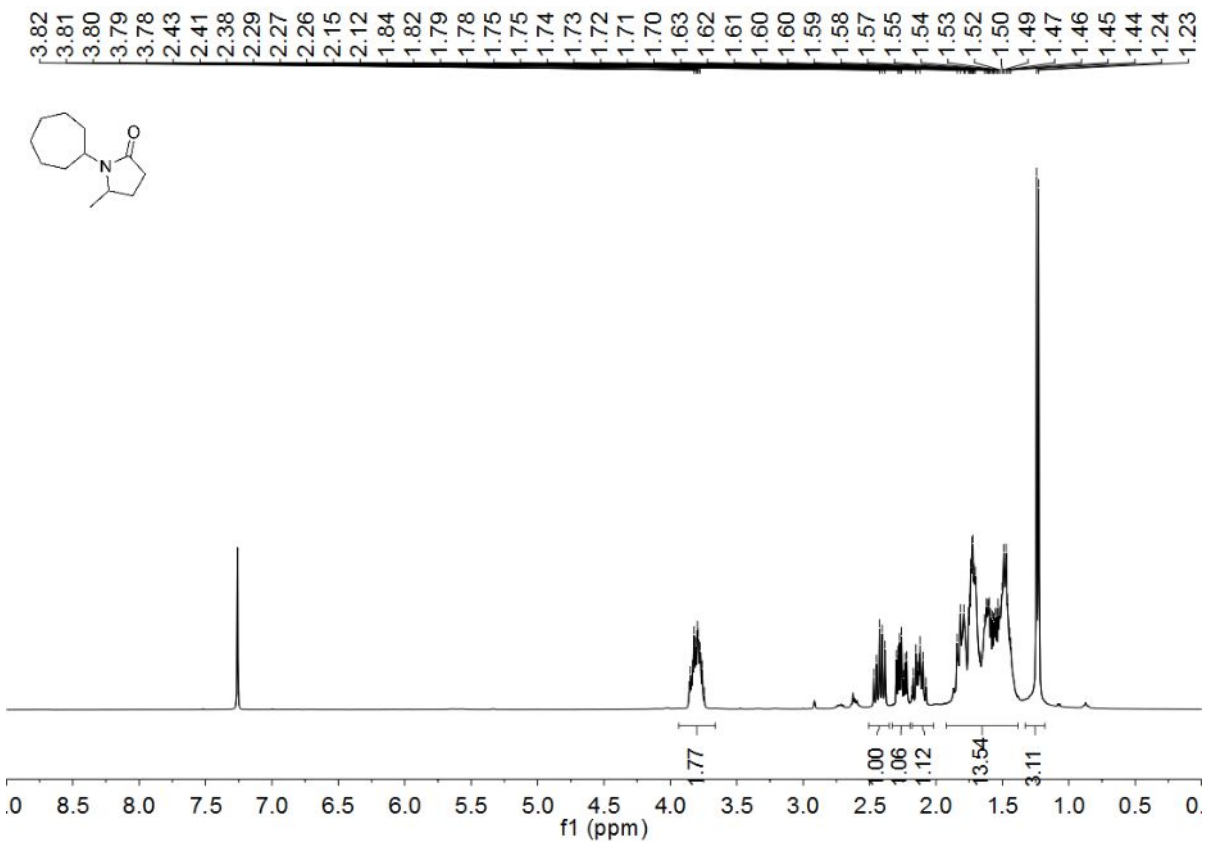
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2.12
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1.82
1.79
1.78
1.75
1.75
1.74
1.73
1.72
1.71
1.70
1.63
1.62
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1.60
1.60
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1.52
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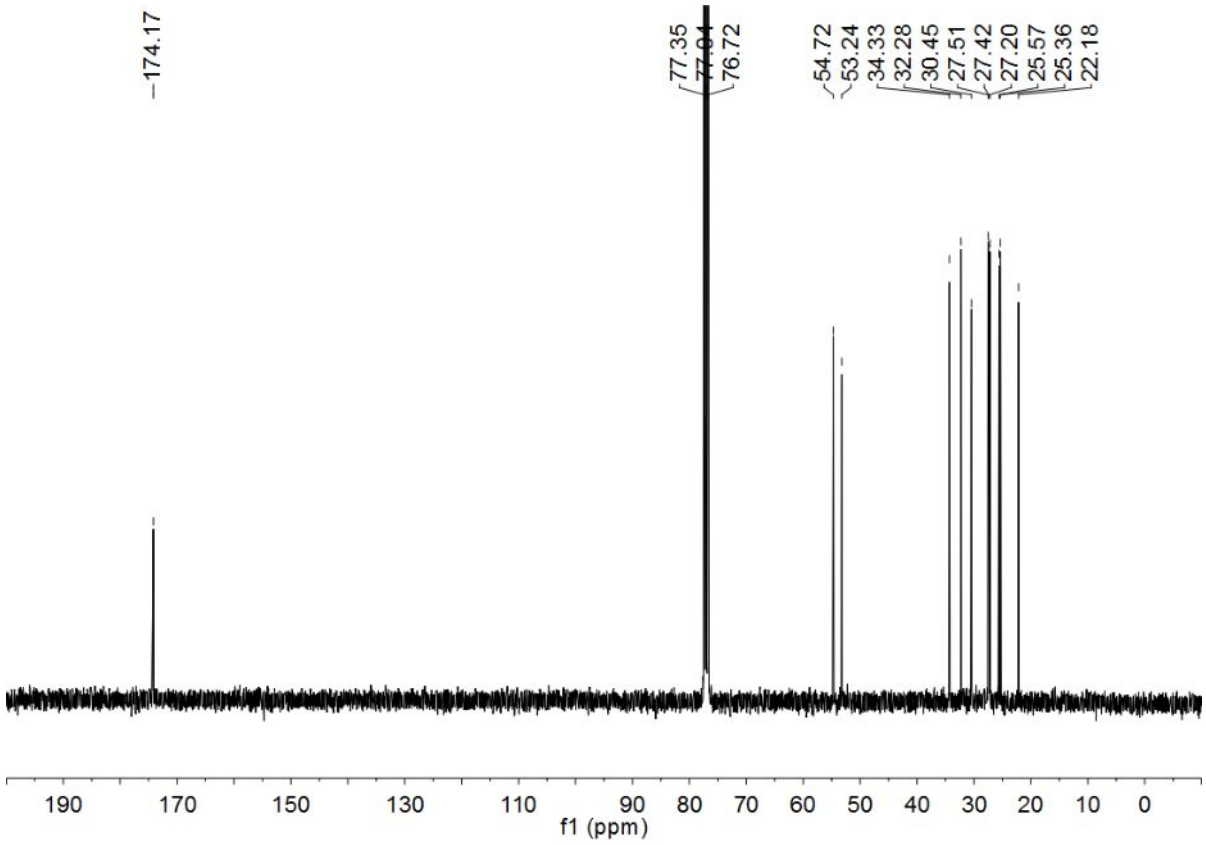
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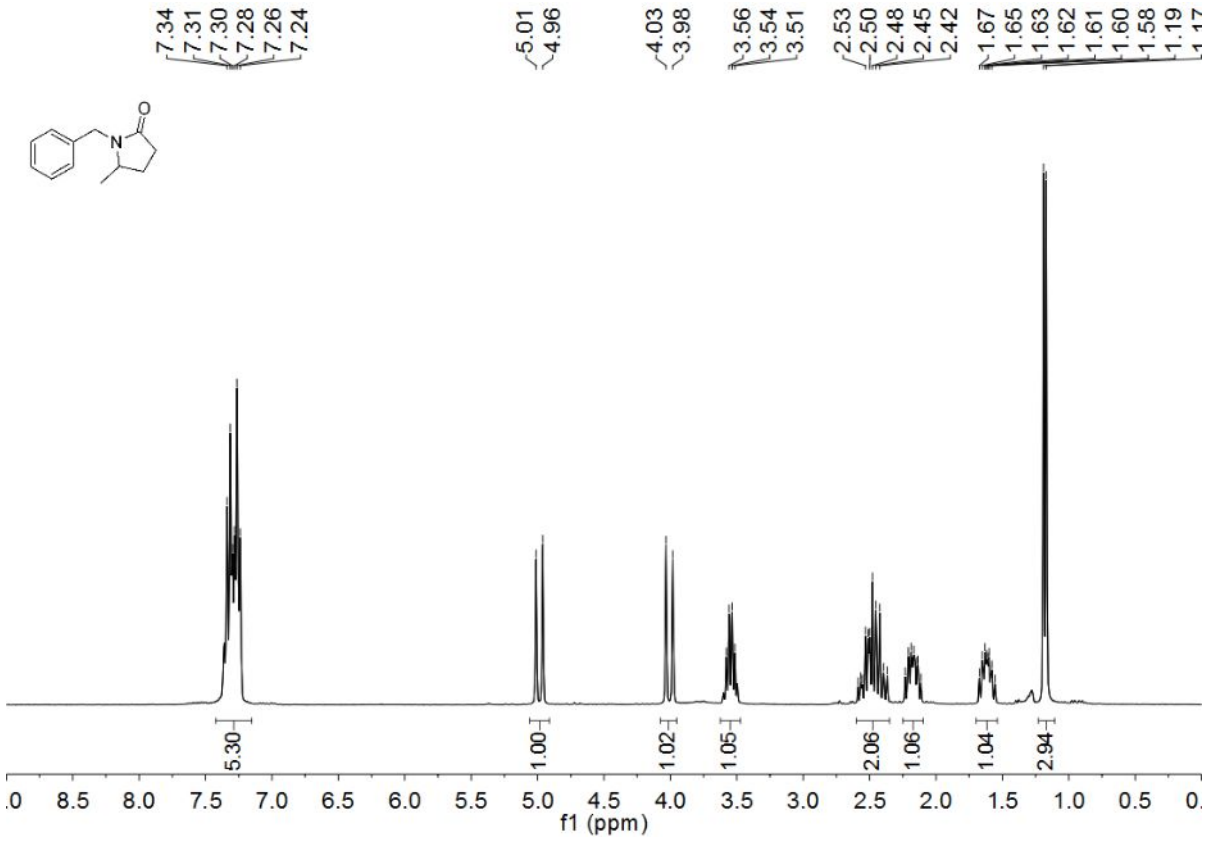
25.57
25.36
22.18
f1 (ppm)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1.63
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1.60
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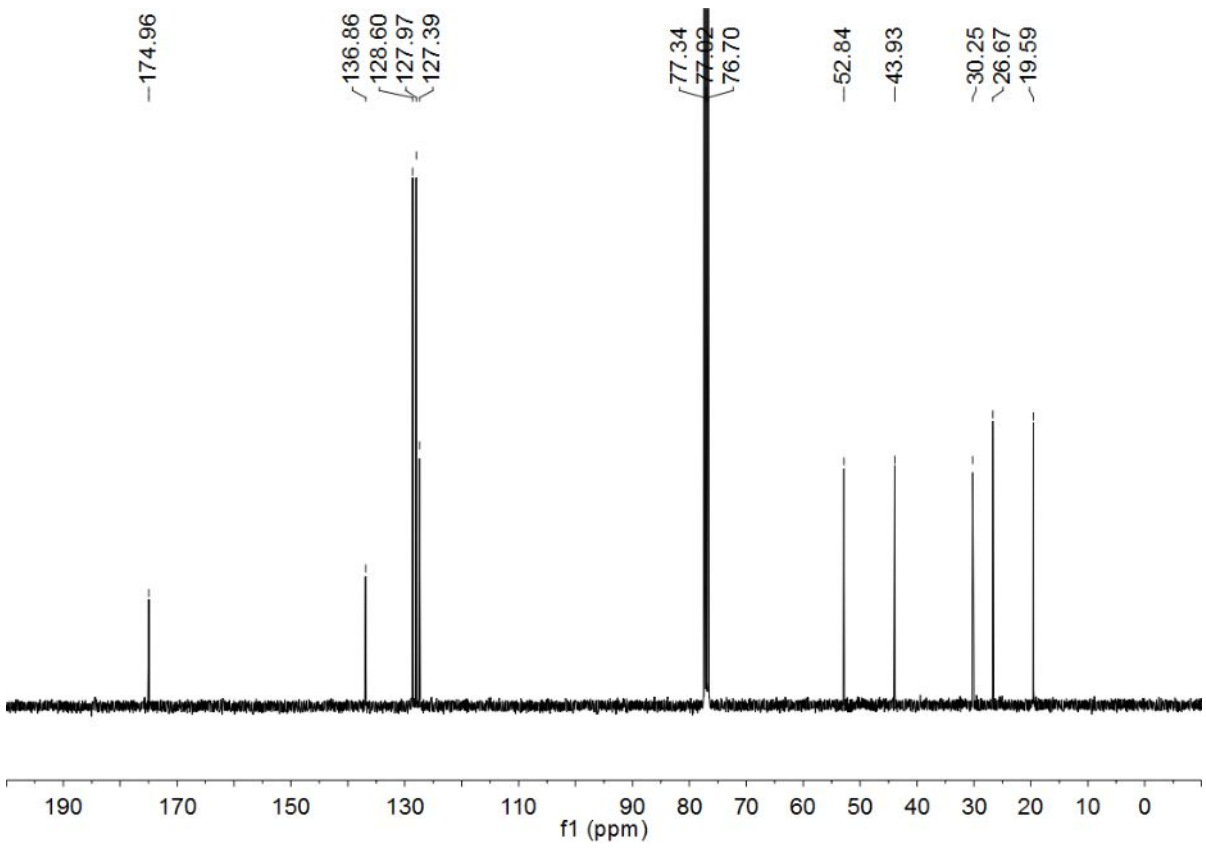
1.04
2.94
f1 (ppm)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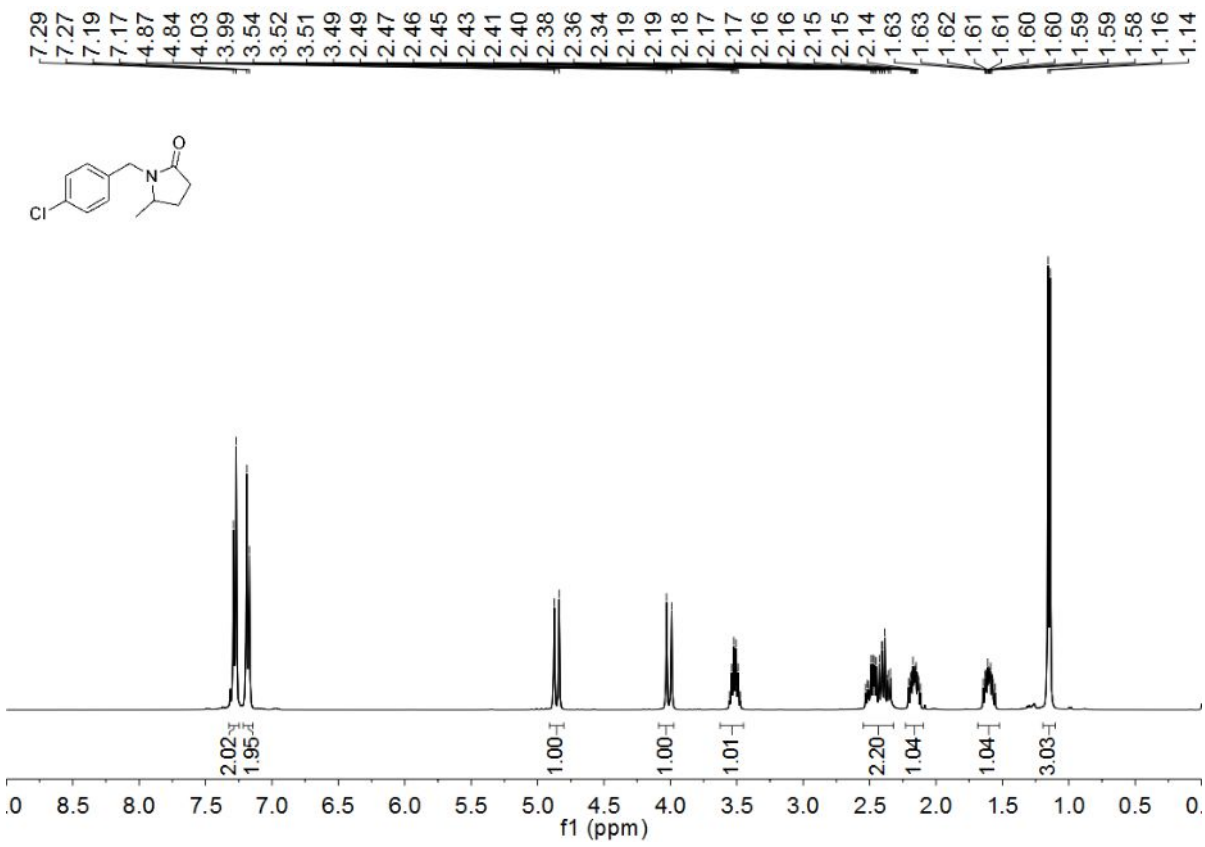
f1 (ppm)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1.58
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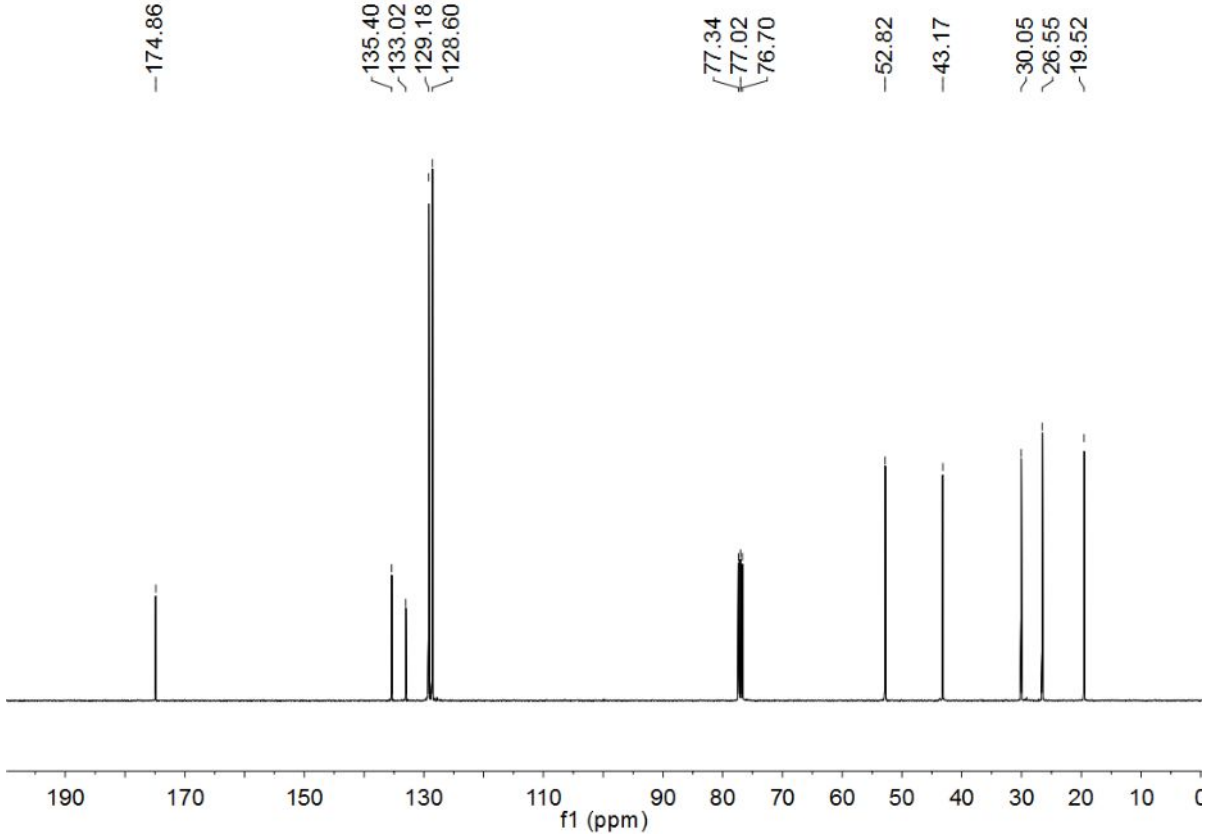
1.04
3.03
f1 (ppm)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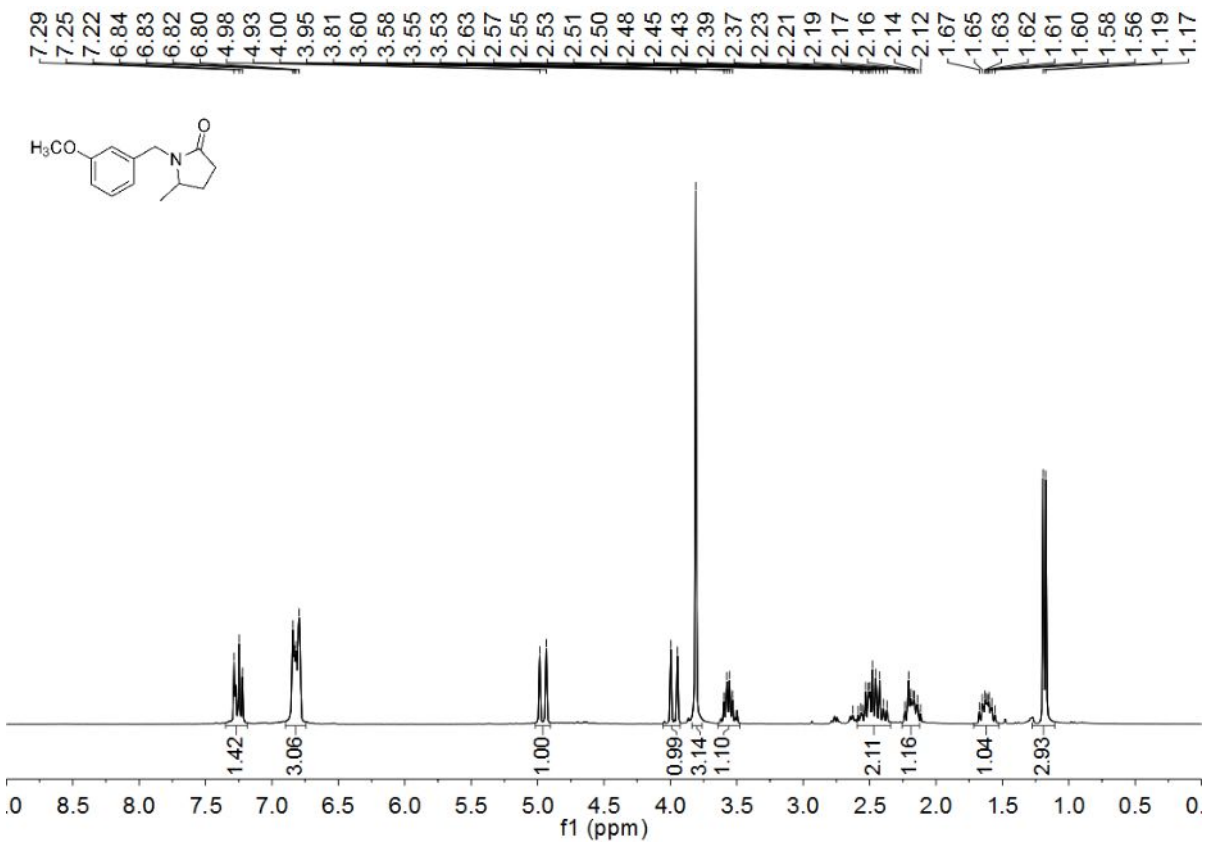
52.82
43.17
30.05
26.55
19.52
f1 (ppm)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3.53
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2.57
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2.53
2.51
2.50
2.48
2.45
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2.14
2.12
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1.65
1.63
1.62
1.61
1.60
1.58
1.56
1.19
1.17
H3CO
O
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0.99
3.14
1.10
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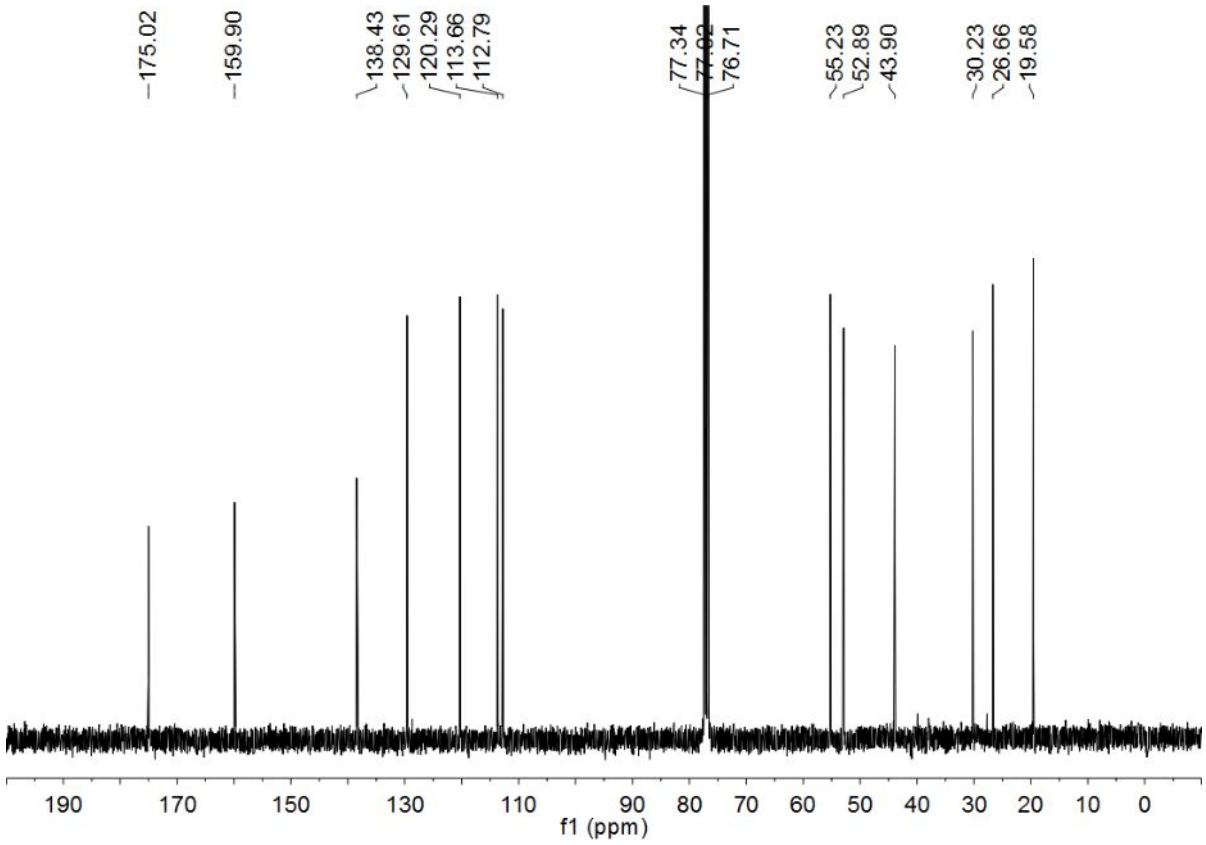
1.16
1.04
2.93
f1 (ppm)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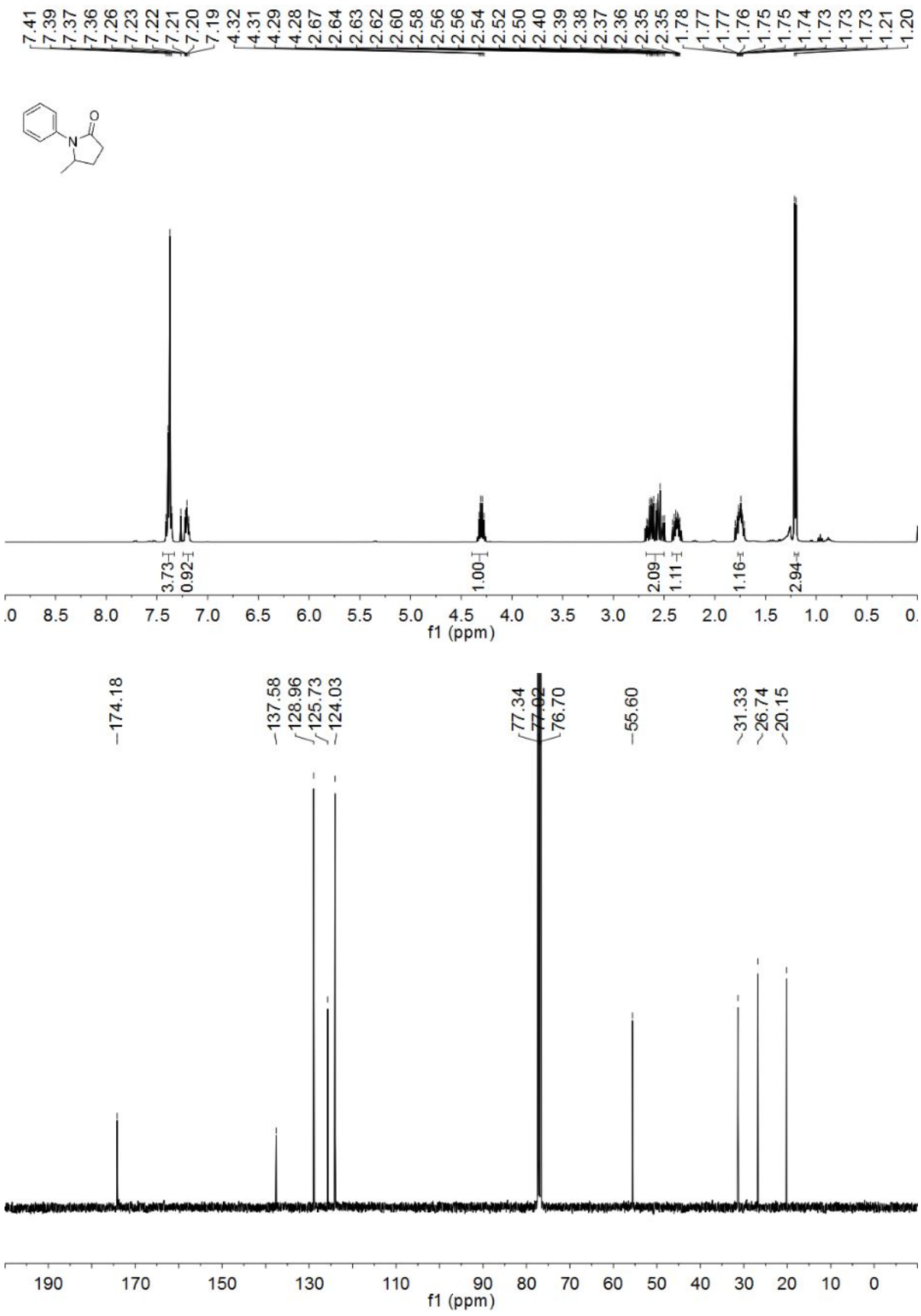
30.23
26.66
19.58
f1 (ppm)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4.28
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2.64
2.63
2.62
2.60
2.58
2.56
2.56
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2.35
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1.77
1.77
1.76
1.75
1.75
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1.73
1.73
1.21
1.20
O
N
3.73
0.92
1.00
2.09
1.11
1.16
2.94
f1 (ppm)
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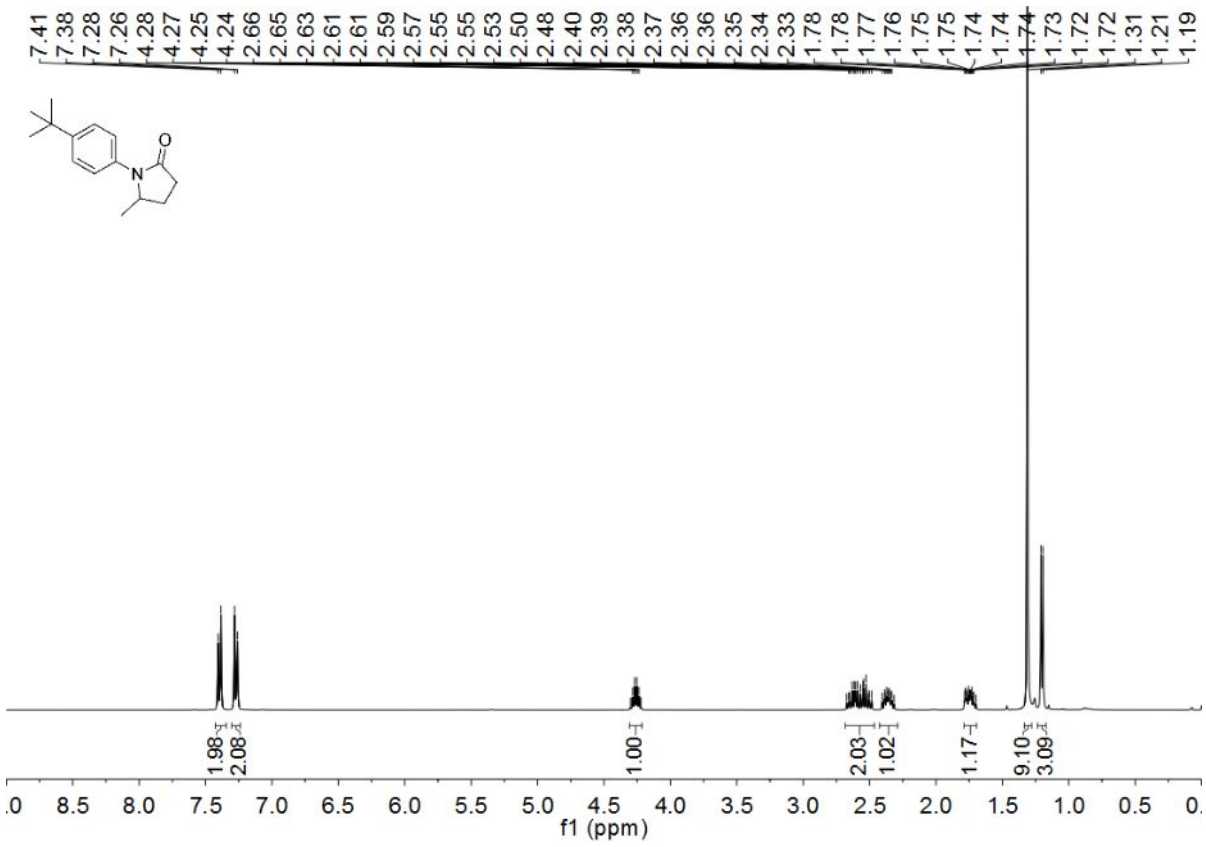
124.03
77.34
77.02
76.70
55.60
31.33
26.74
20.15
f1 (ppm)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O
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1.00
2.03
1.02
1.17
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3.09
8.5
8.0
7.5
7.0
6.5
6.0
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4.0
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3.0
2.5
2.0
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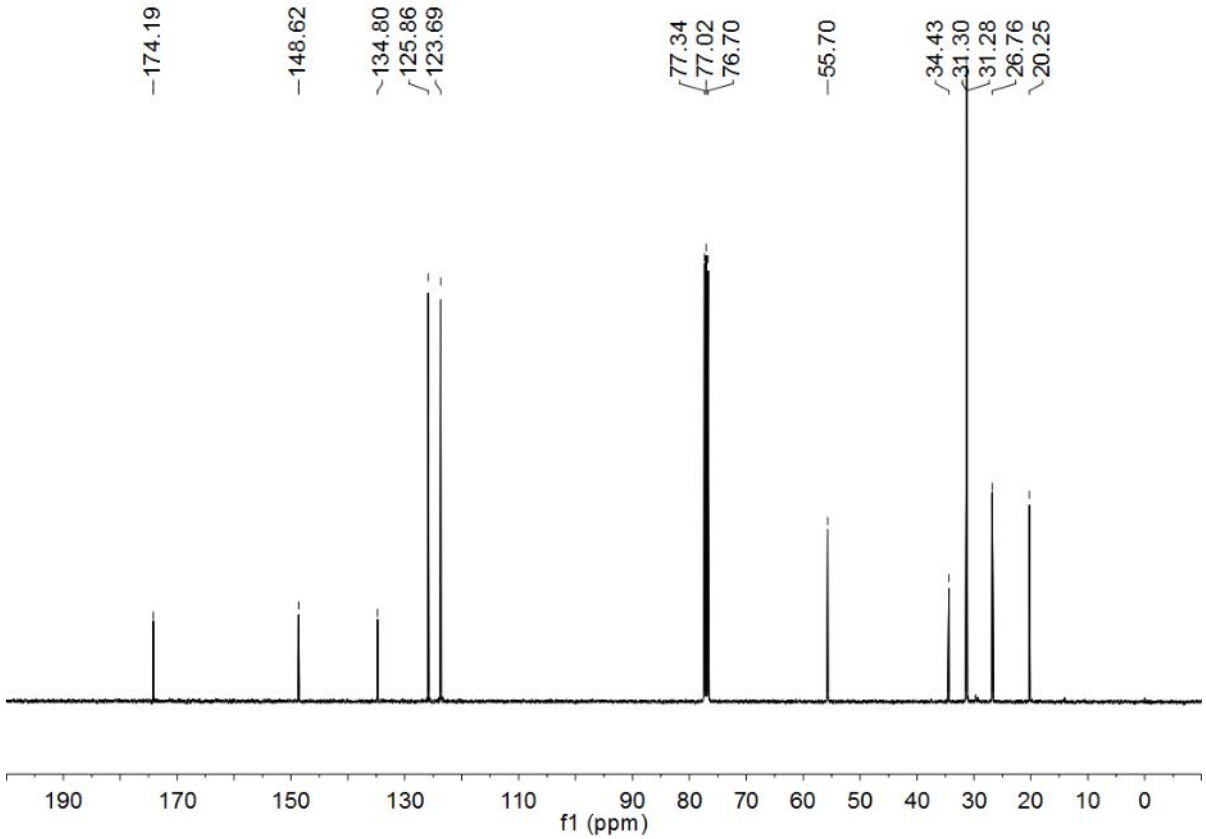
1.0
0.5
f1 (ppm)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76.70
55.70
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31.28
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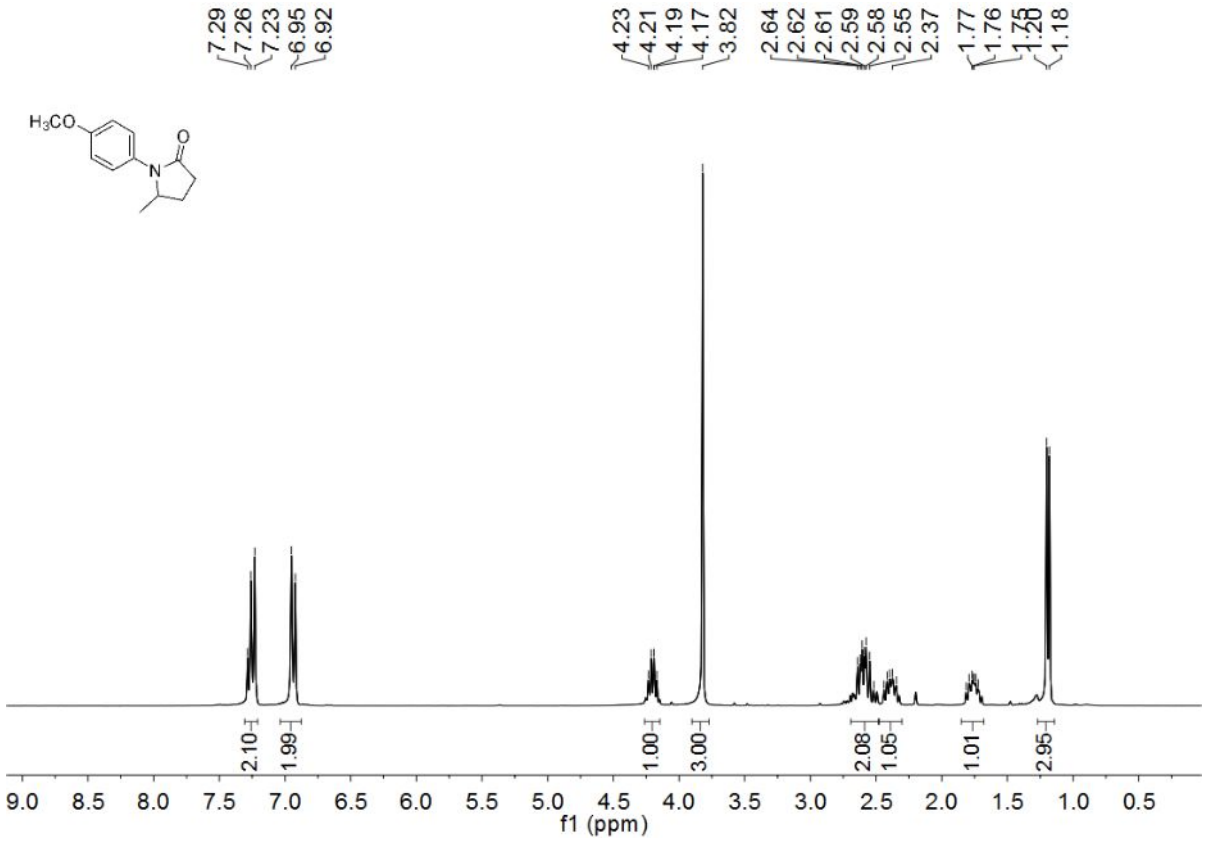
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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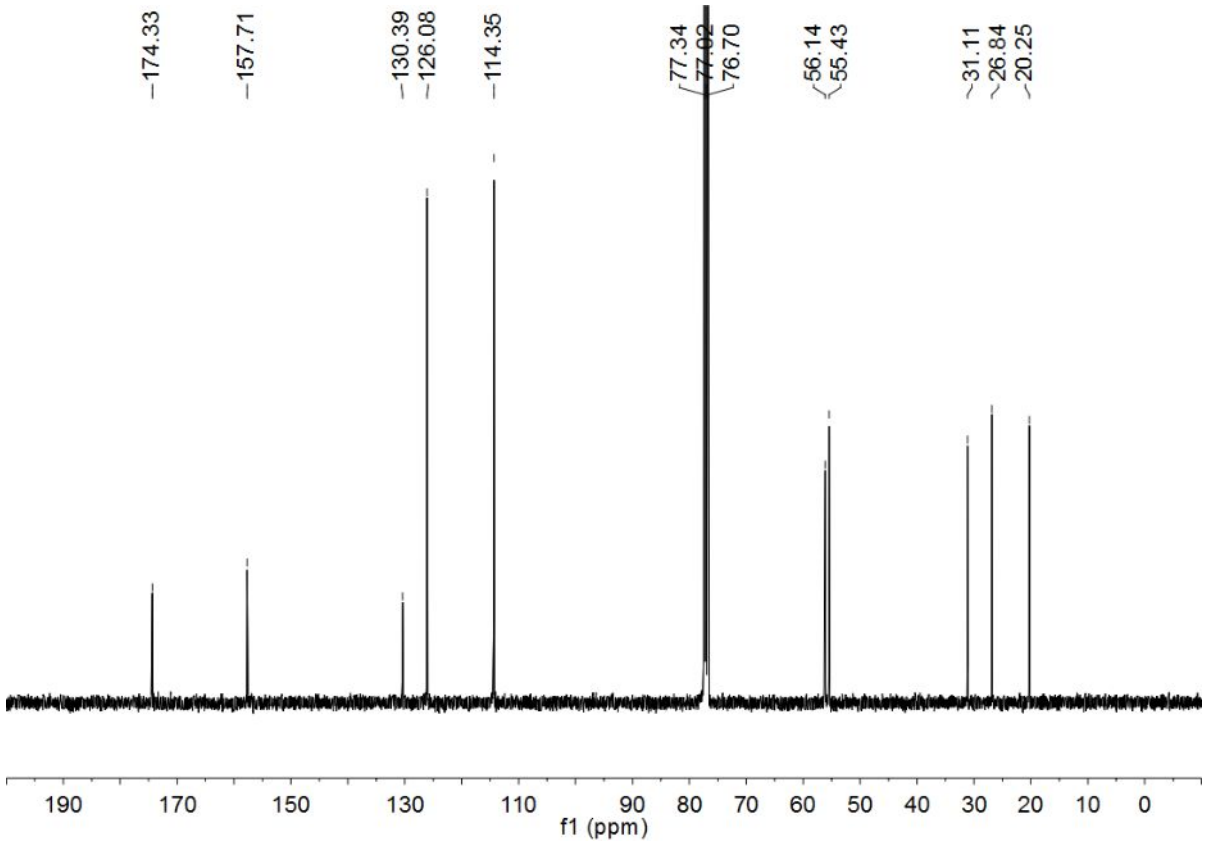
3.82
2.64
2.62
2.61
2.59
2.58
2.55
2.37
1.77
1.76
1.75
1.20
1.18
H3CO
O
N
2.10
1.99
1.00
3.00
2.08
1.05
1.01
2.95
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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77.34
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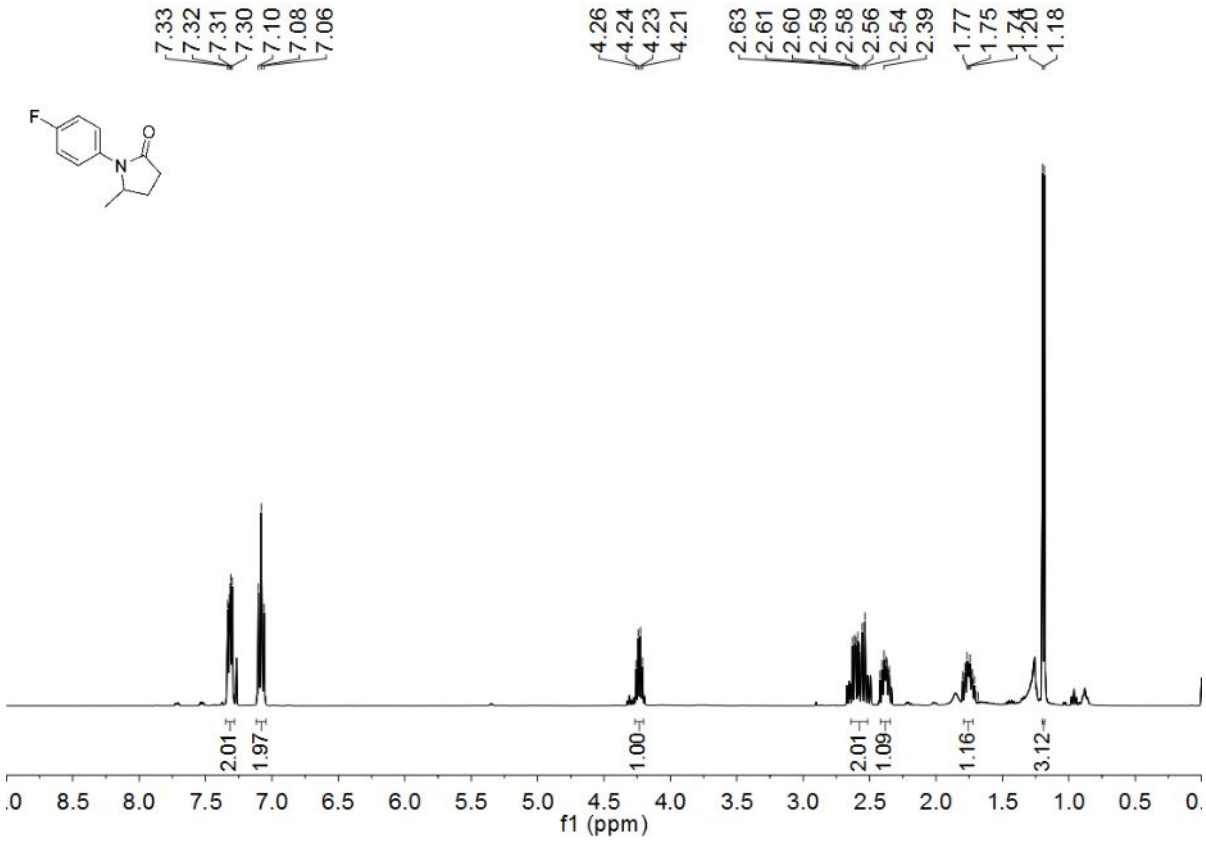
56.14
55.43
31.11
26.84
20.25
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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4.23
4.21
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2.61
2.60
2.59
2.58
2.56
2.54
2.39
1.77
1.75
1.74
1.20
1.18
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O
N
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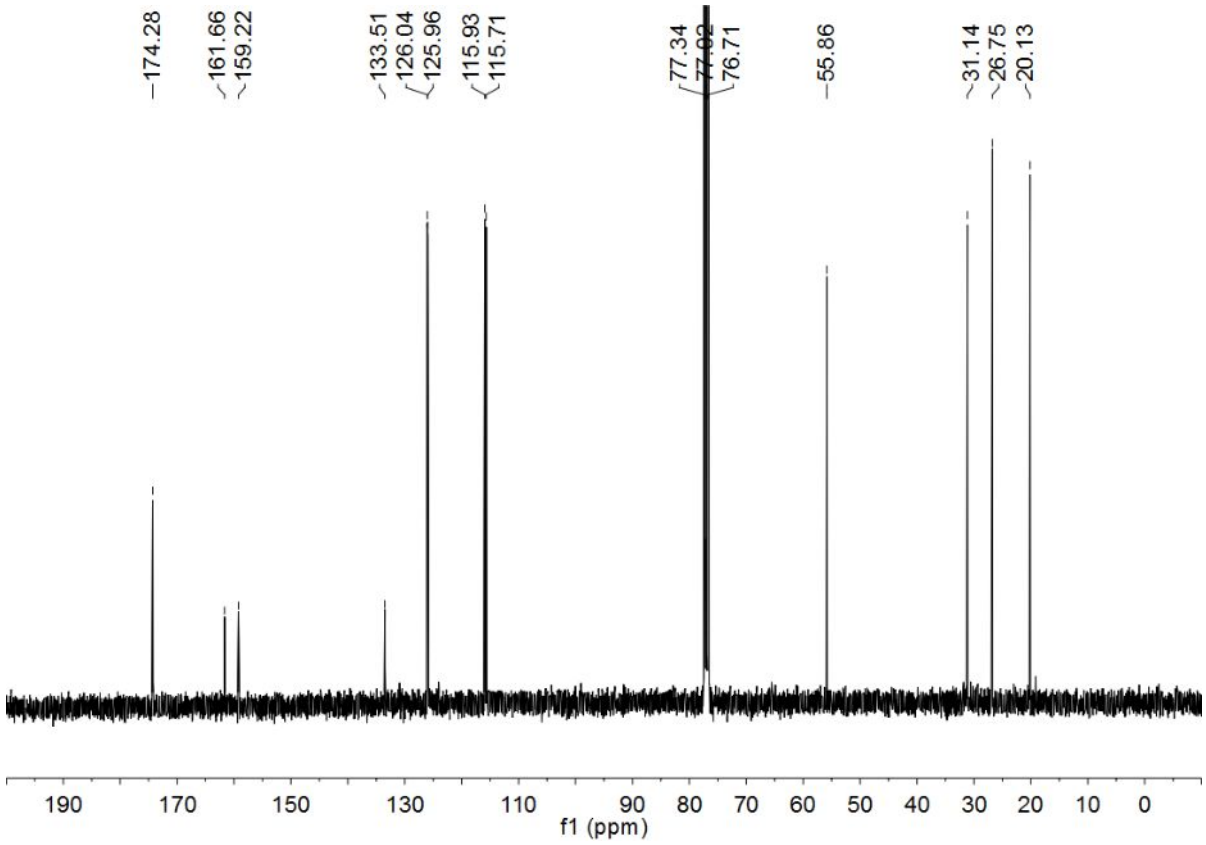
1.00
2.01
1.09
1.16
3.12
f1 (ppm)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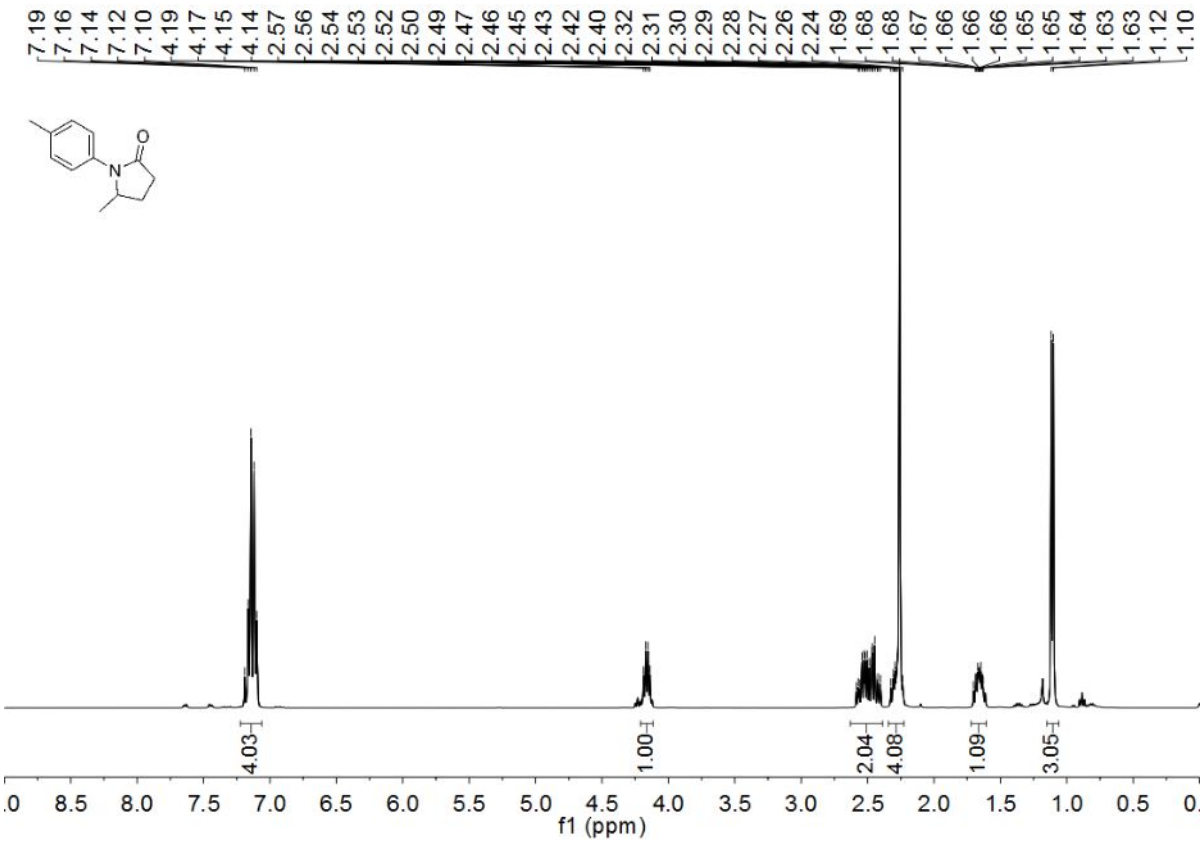
20.13
f1 (ppm)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2.42
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2.26
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O
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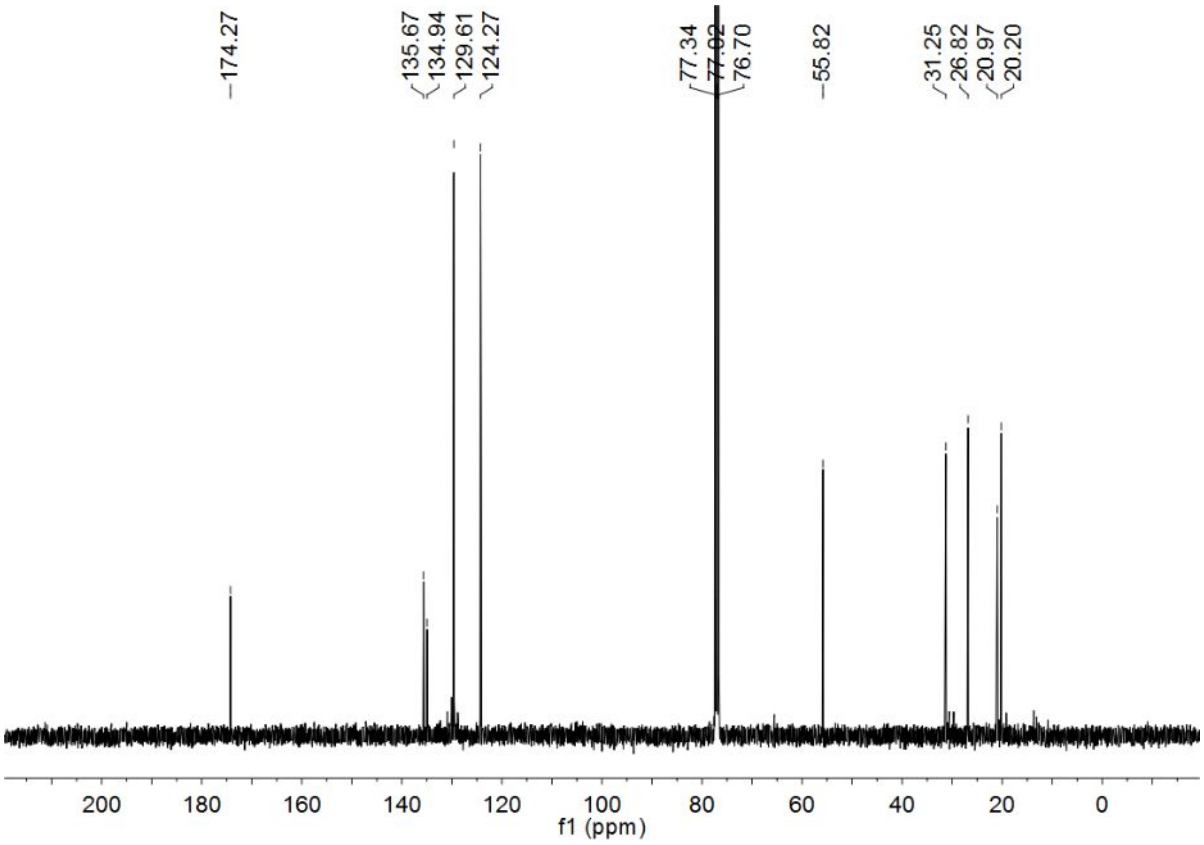
3.05
f1 (ppm)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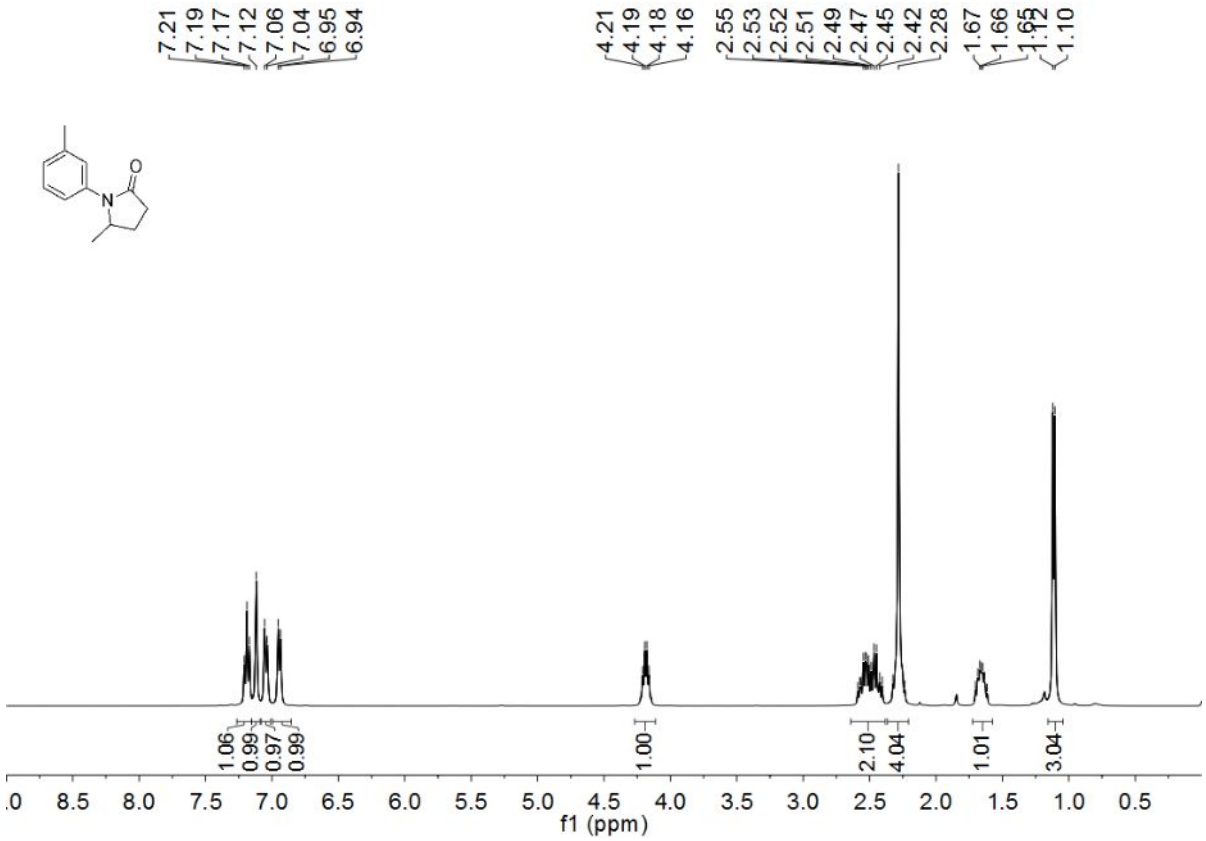
20.20
f1 (ppm)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2.42
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1.66
1.65
1.12
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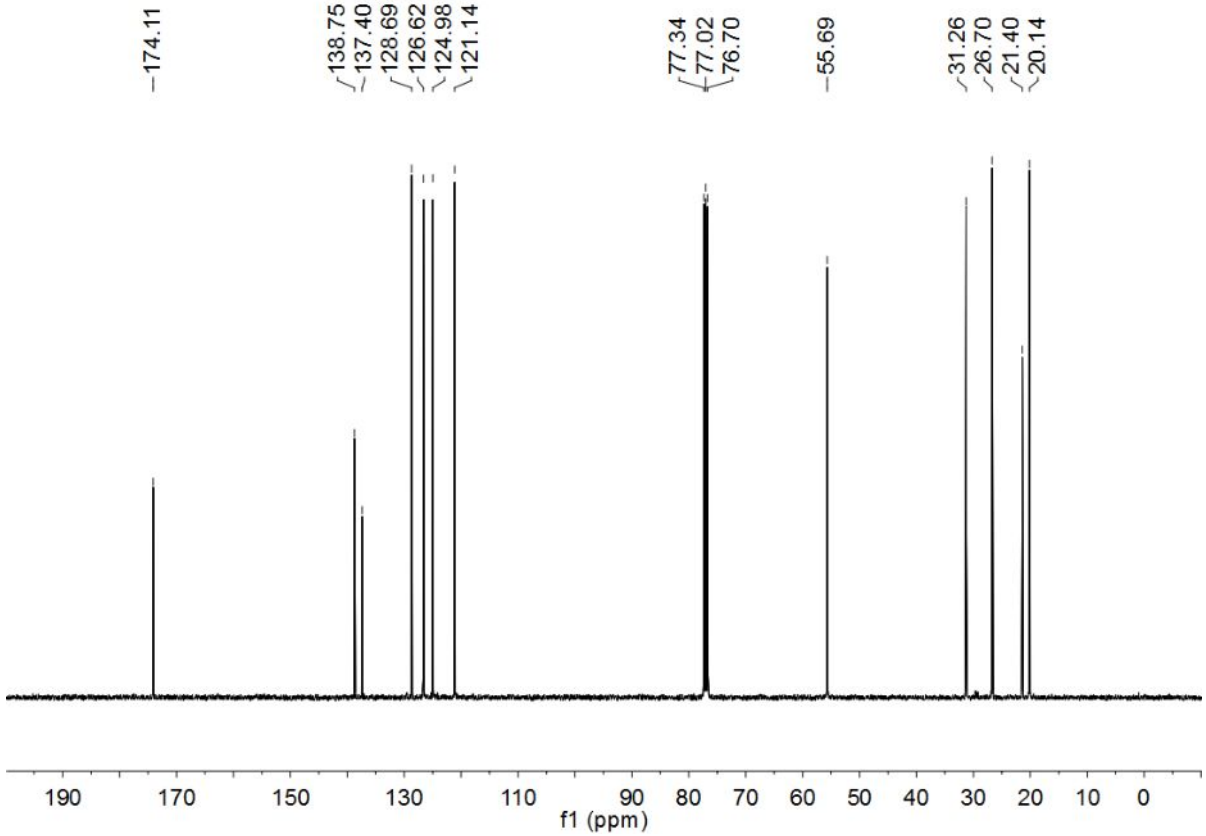
0.99
1.00
2.10
4.04
1.01
3.04
f1 (ppm)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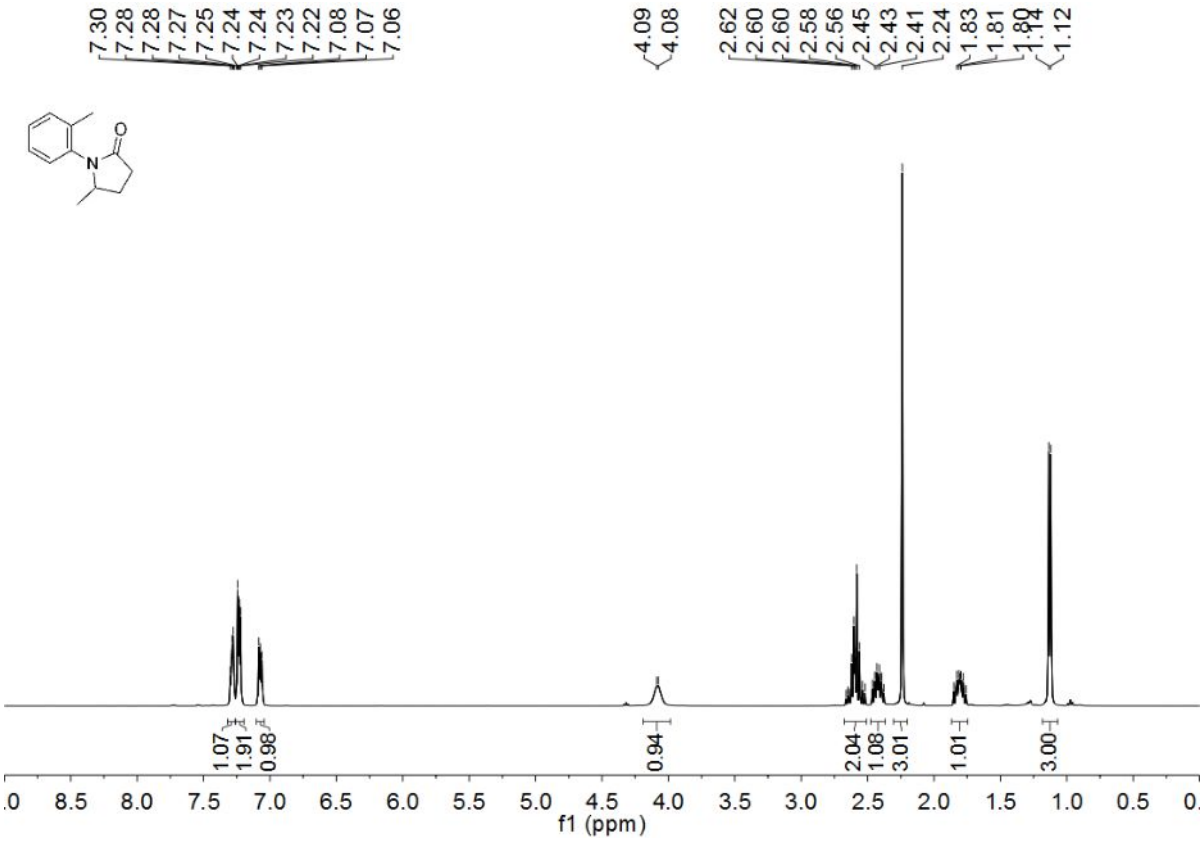
20.14
f1 (ppm)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2.43
2.41
2.24
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1.81
1.80
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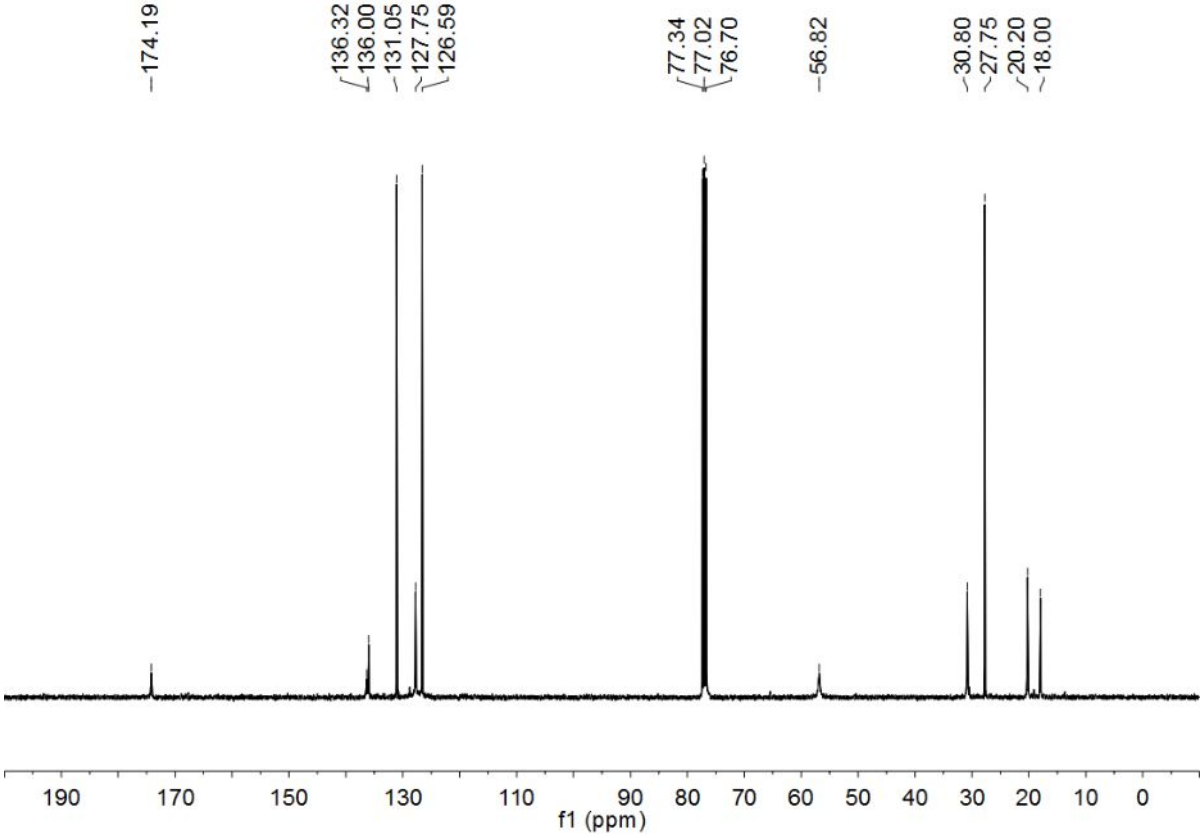
0.94
2.04
1.08
3.01
1.01
3.00
f1 (ppm)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56.82
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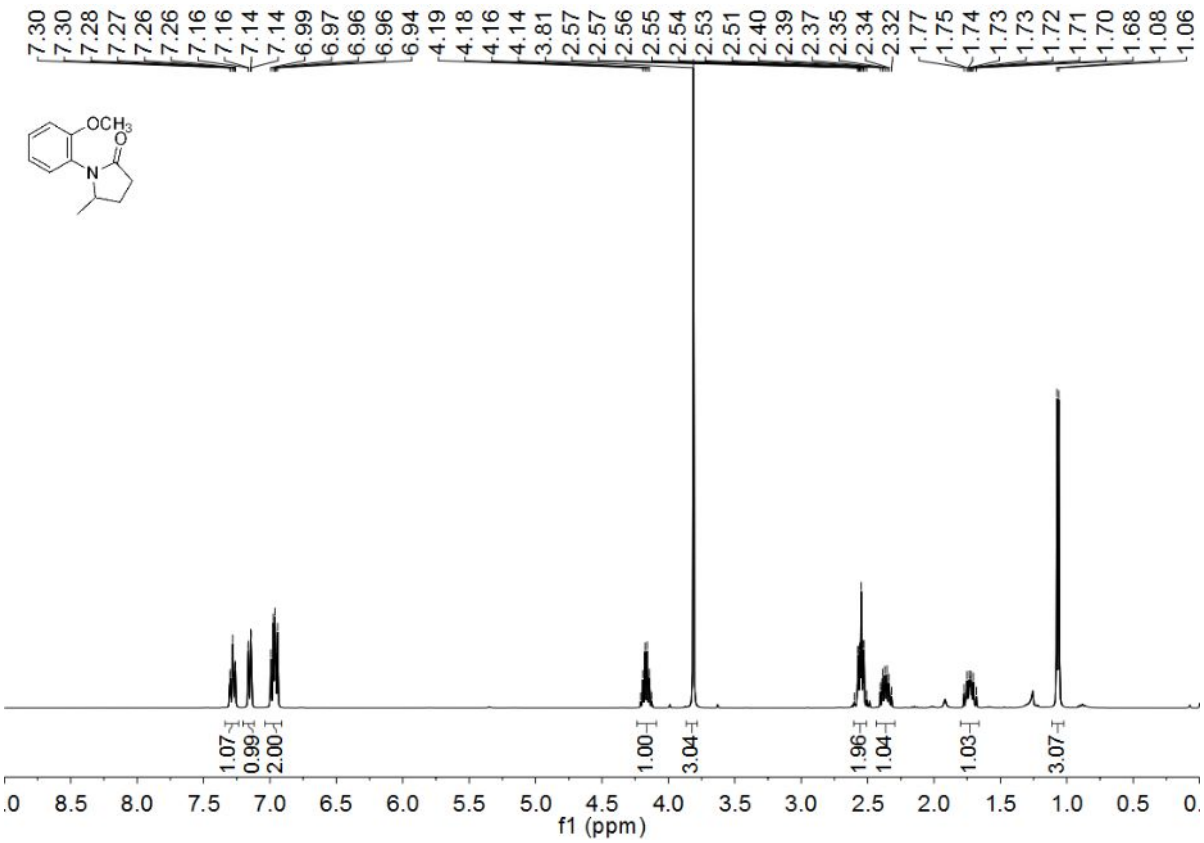
27.75
20.20
18.00
f1 (ppm)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6.96
6.94
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1.73
1.72
1.71
1.70
1.68
1.08
1.06
OCH3
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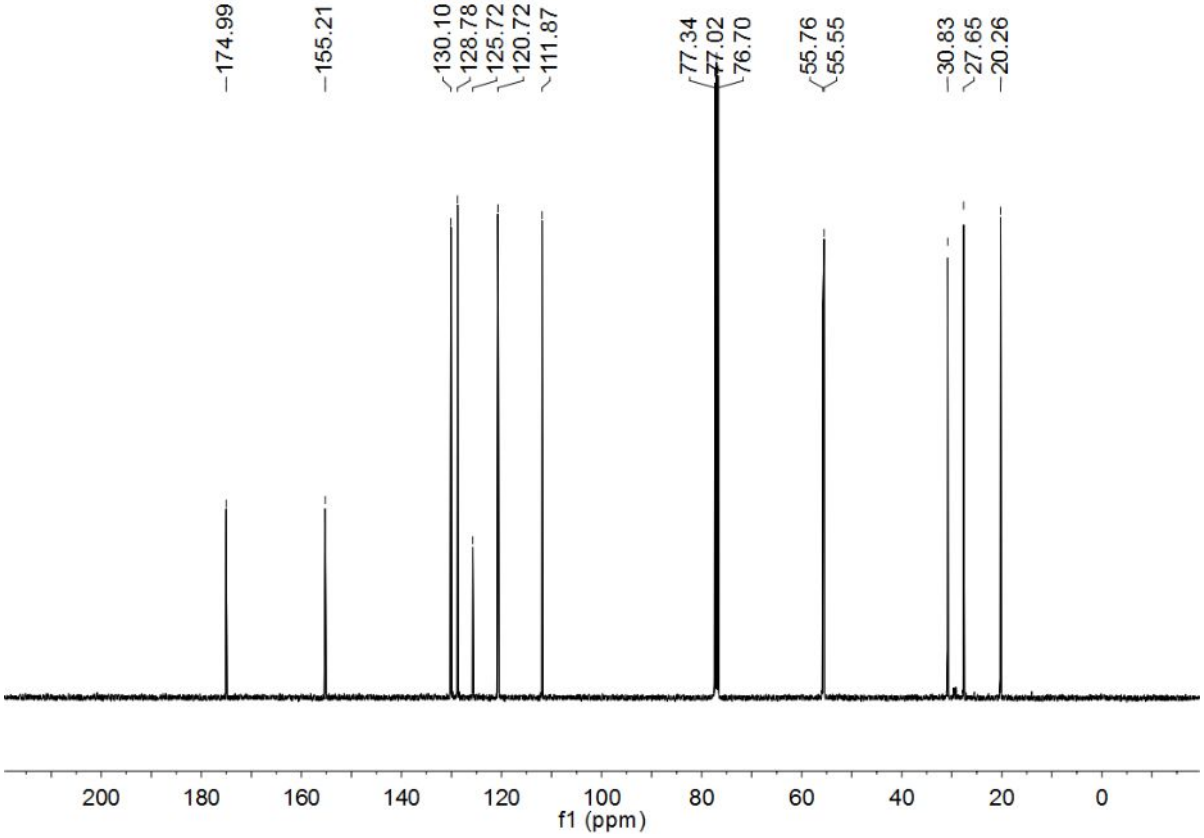
2.00
1.00
3.04
1.96
1.04
1.03
3.07
f1 (ppm)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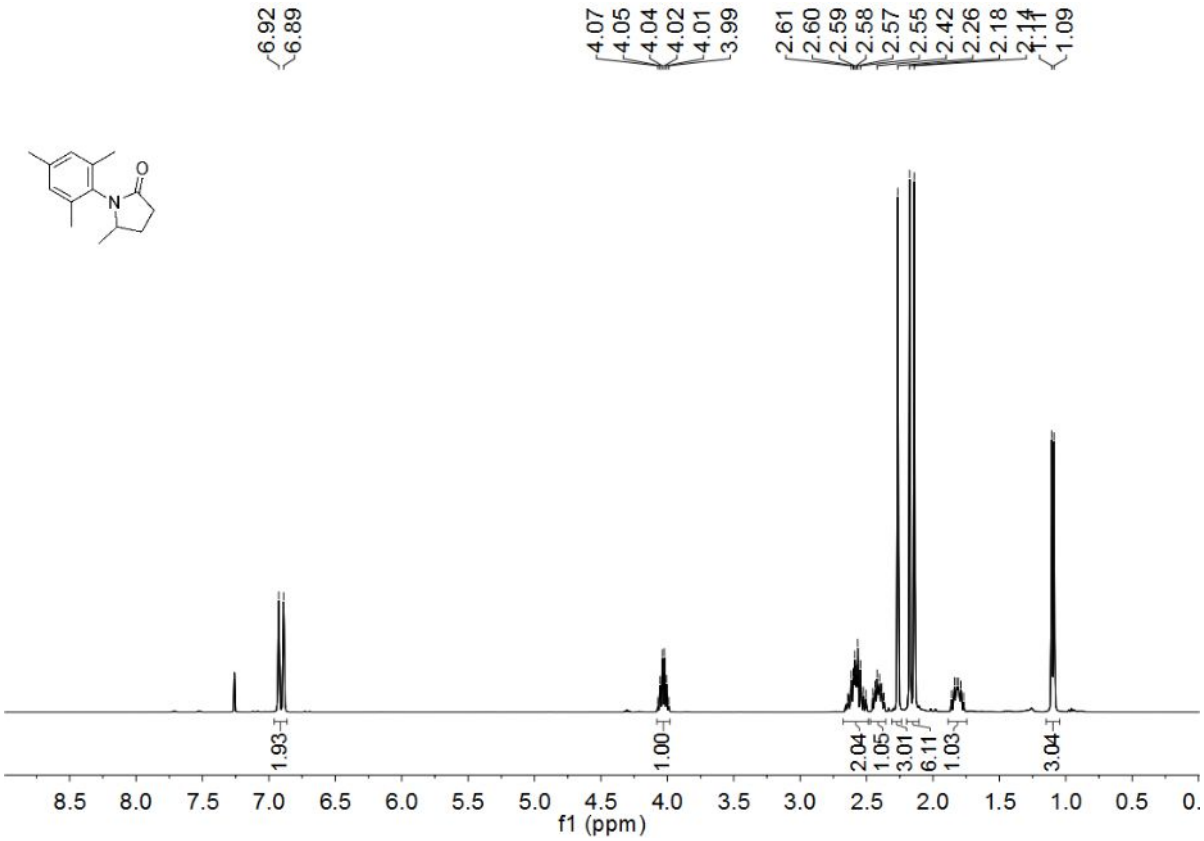
77.02
76.70
55.76
55.55
30.83
27.65
20.26
f1 (ppm)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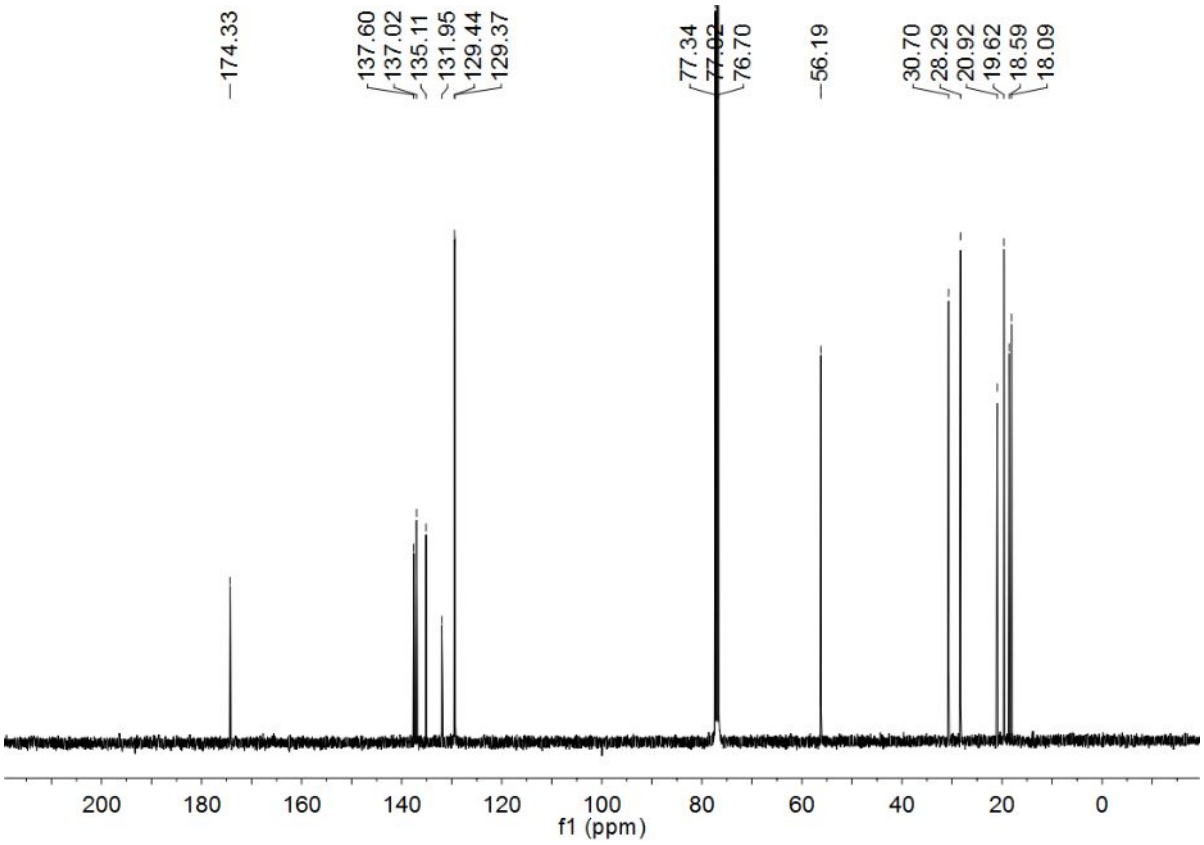
4.02
4.01
3.99
2.61
2.60
2.59
2.58
2.57
2.55
2.42
2.26
2.18
2.14
1.11
1.09
O
N
1.93
1.00
2.04
1.05
3.01
6.11
1.03
3.04
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)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77.02
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56.19
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28.29
20.92
19.62
18.59
18.09
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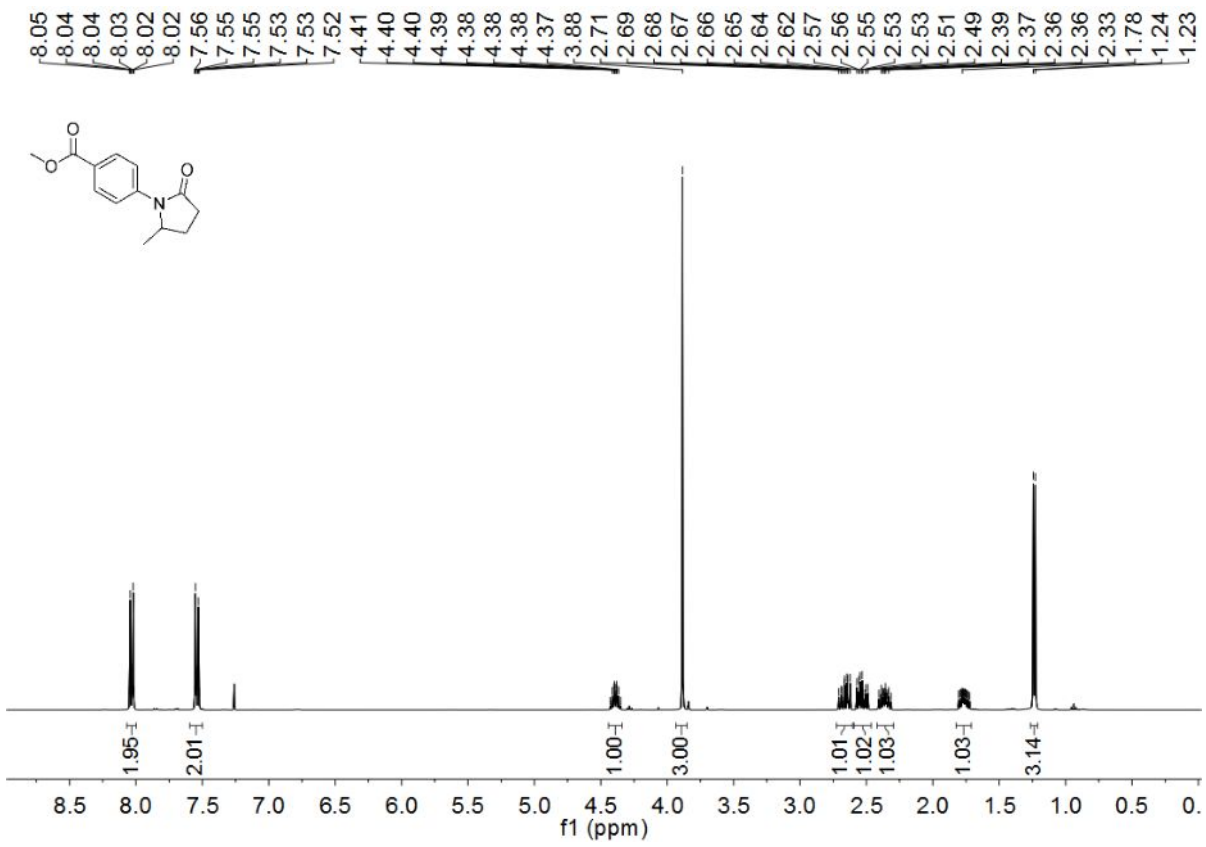
f1 (ppm)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O
O
N
O
1.95
2.01
1.00
3.00
1.01
1.02
1.03
1.03
3.14
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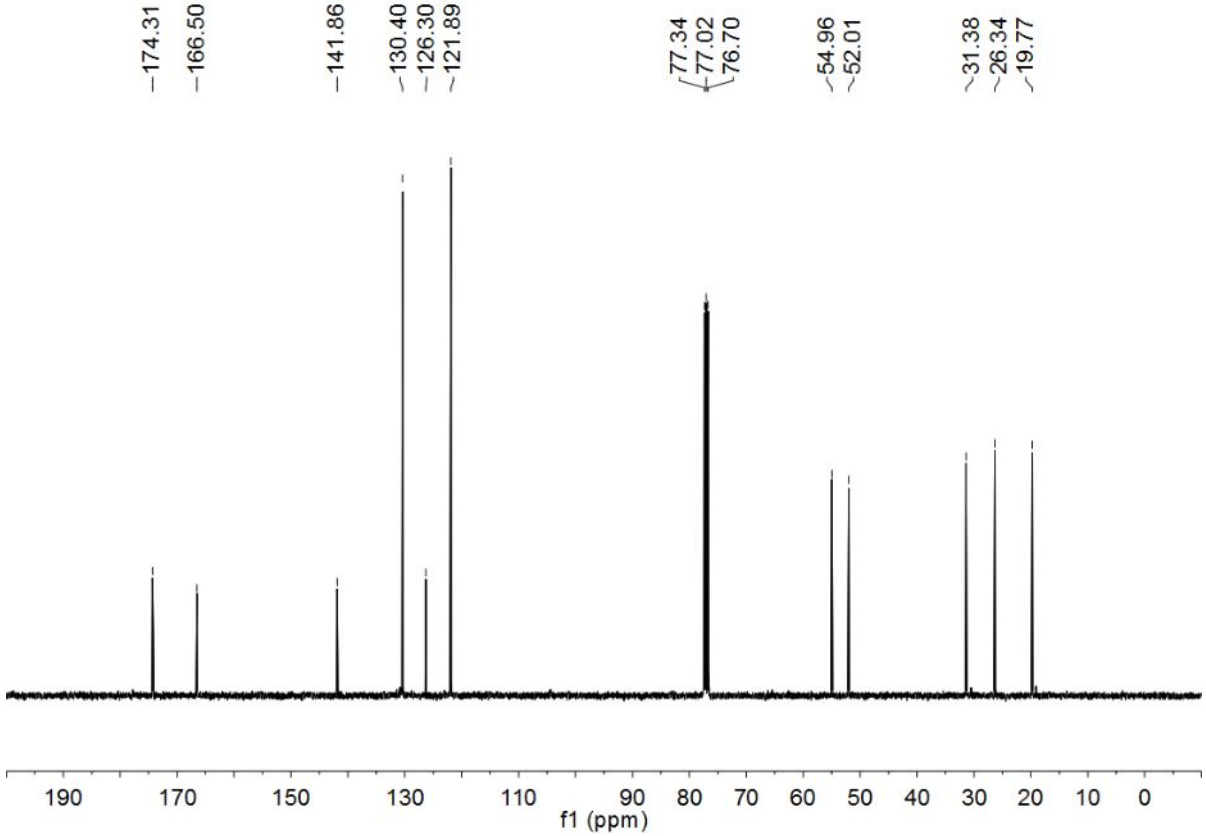
f1 (ppm)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52.01
31.38
26.34
19.77
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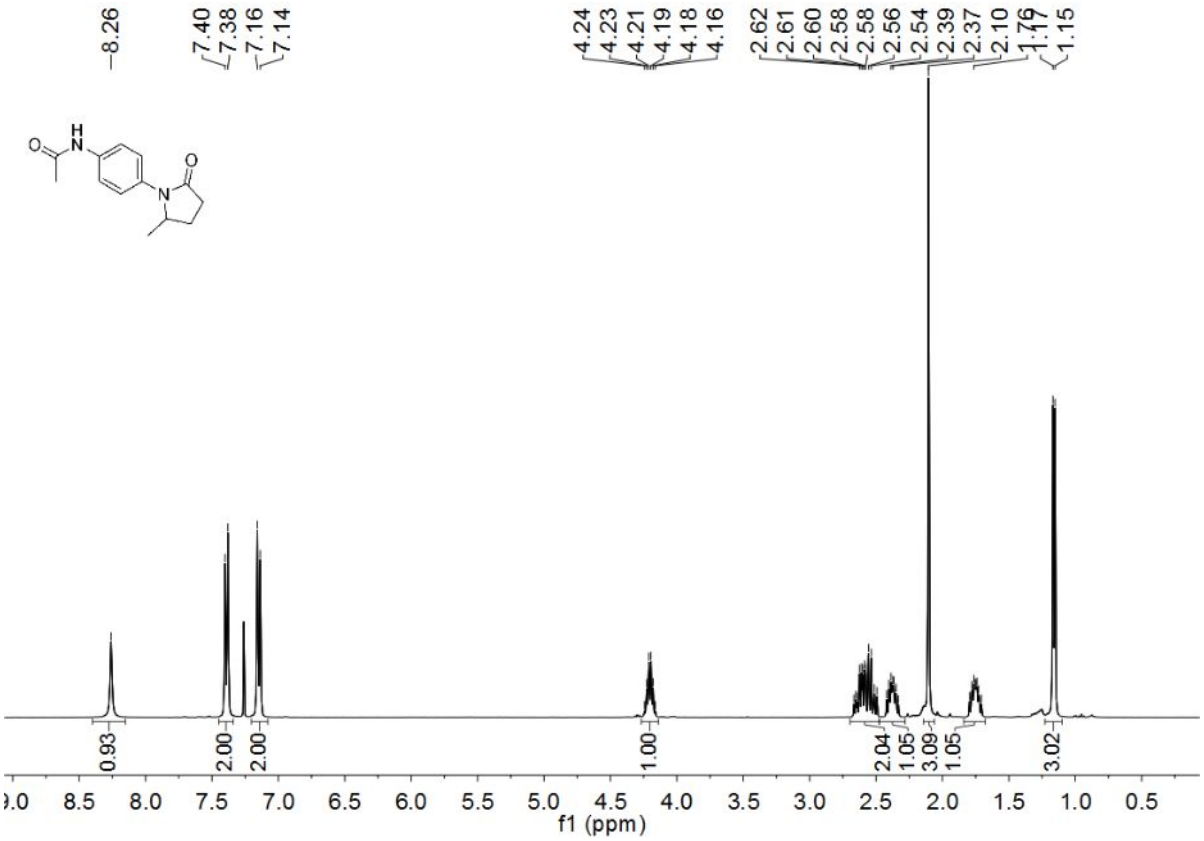
f1 (ppm)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4.18
4.16
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2.61
2.60
2.58
2.58
2.56
2.54
2.39
2.37
2.10
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2.00
1.00
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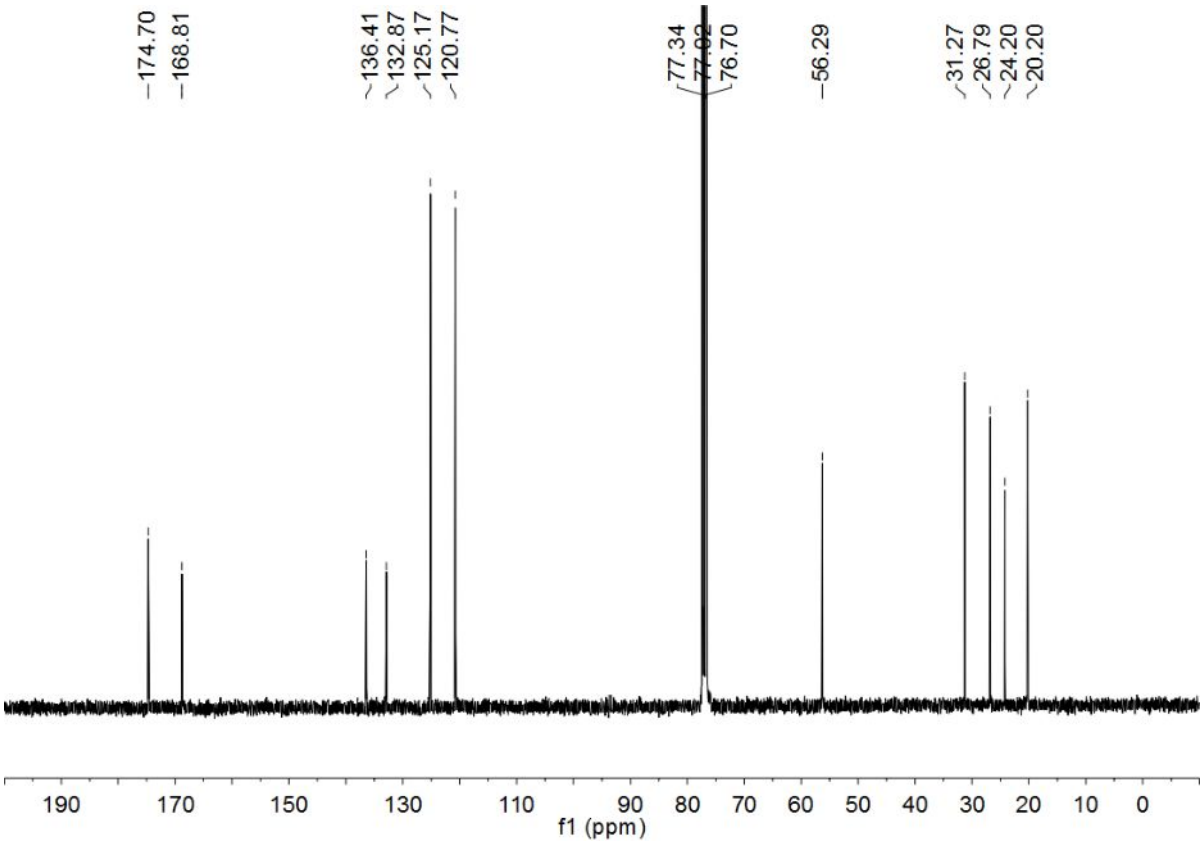
1.05
3.09
1.05
3.02
f1 (ppm)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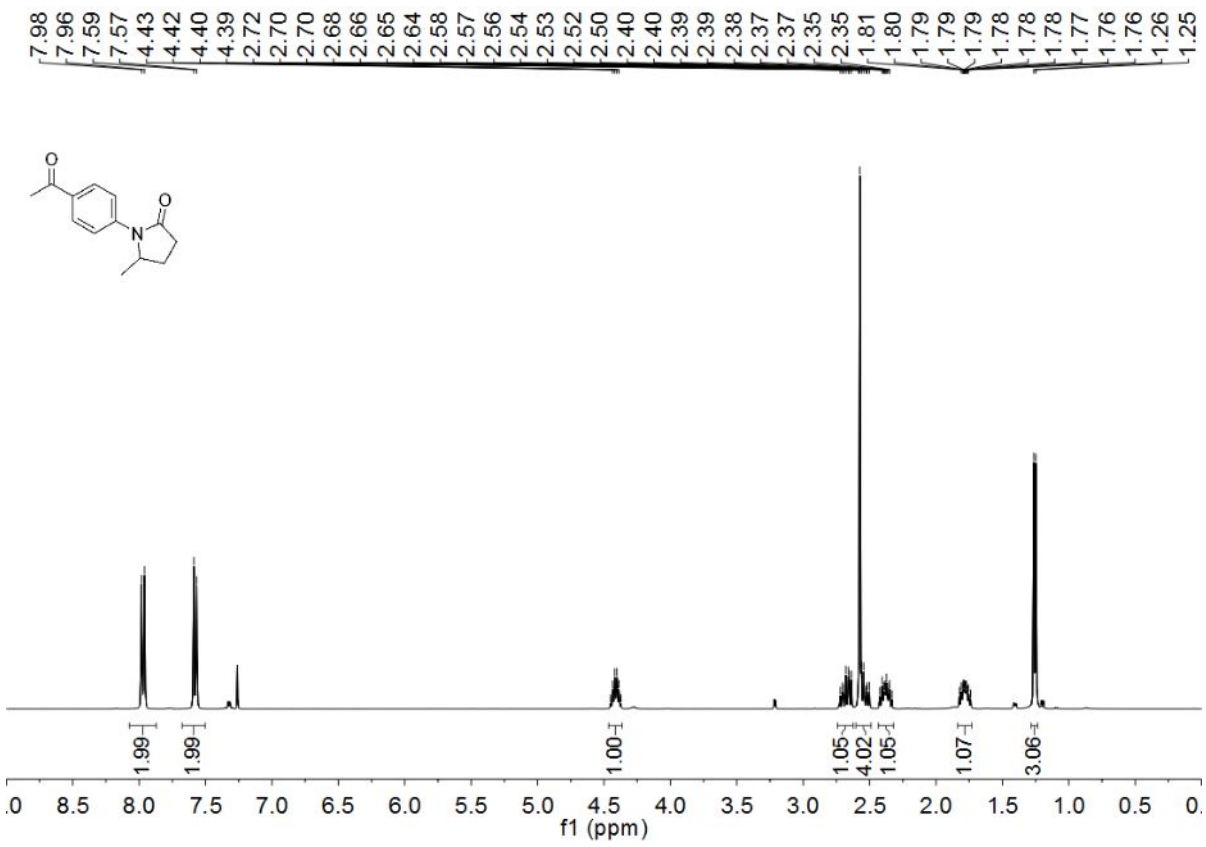
56.29
31.27
26.79
24.20
20.20
f1 (ppm)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7.57
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4.42
4.40
4.39
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2.70
2.70
2.68
2.66
2.65
2.64
2.58
2.57
2.56
2.54
2.53
2.52
2.50
2.40
2.40
2.39
2.39
2.38
2.37
2.37
2.35
2.35
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1.78
1.78
1.78
1.77
1.76
1.76
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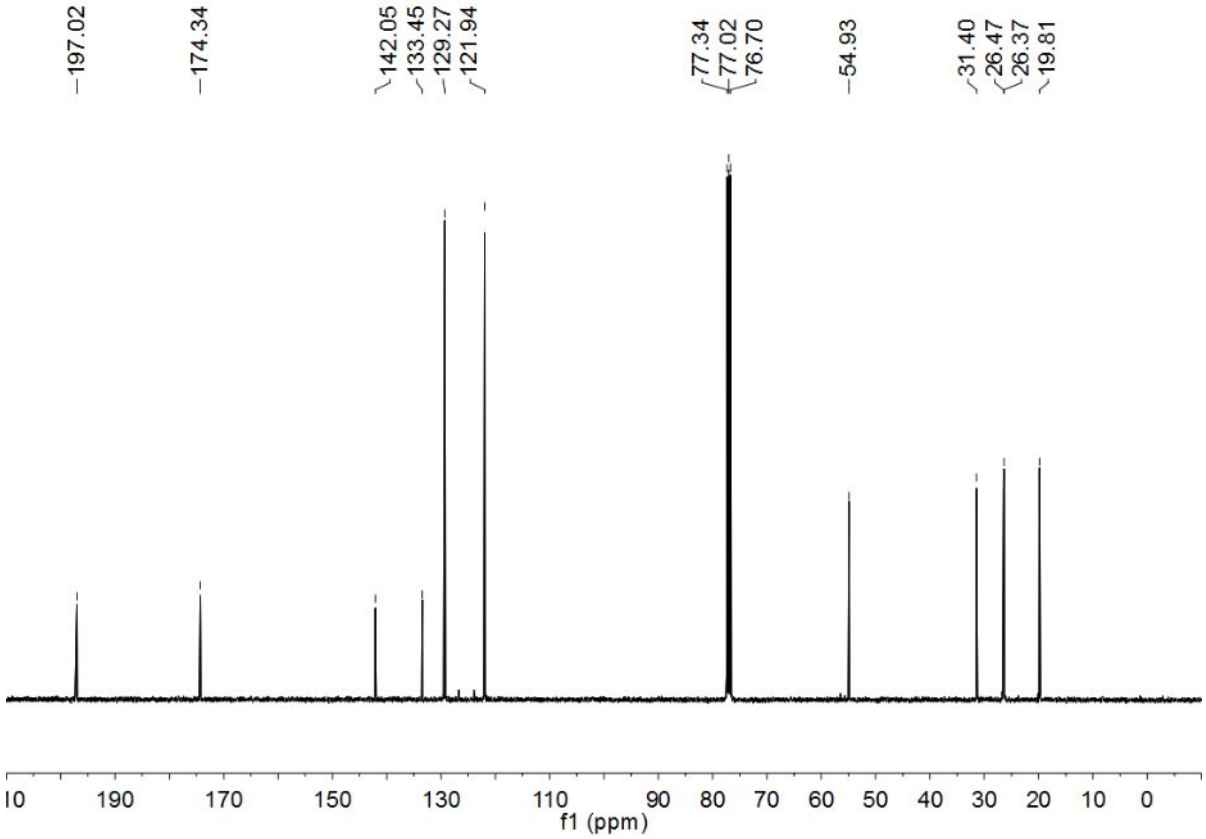
4.02
1.05
1.07
3.06
f1 (ppm)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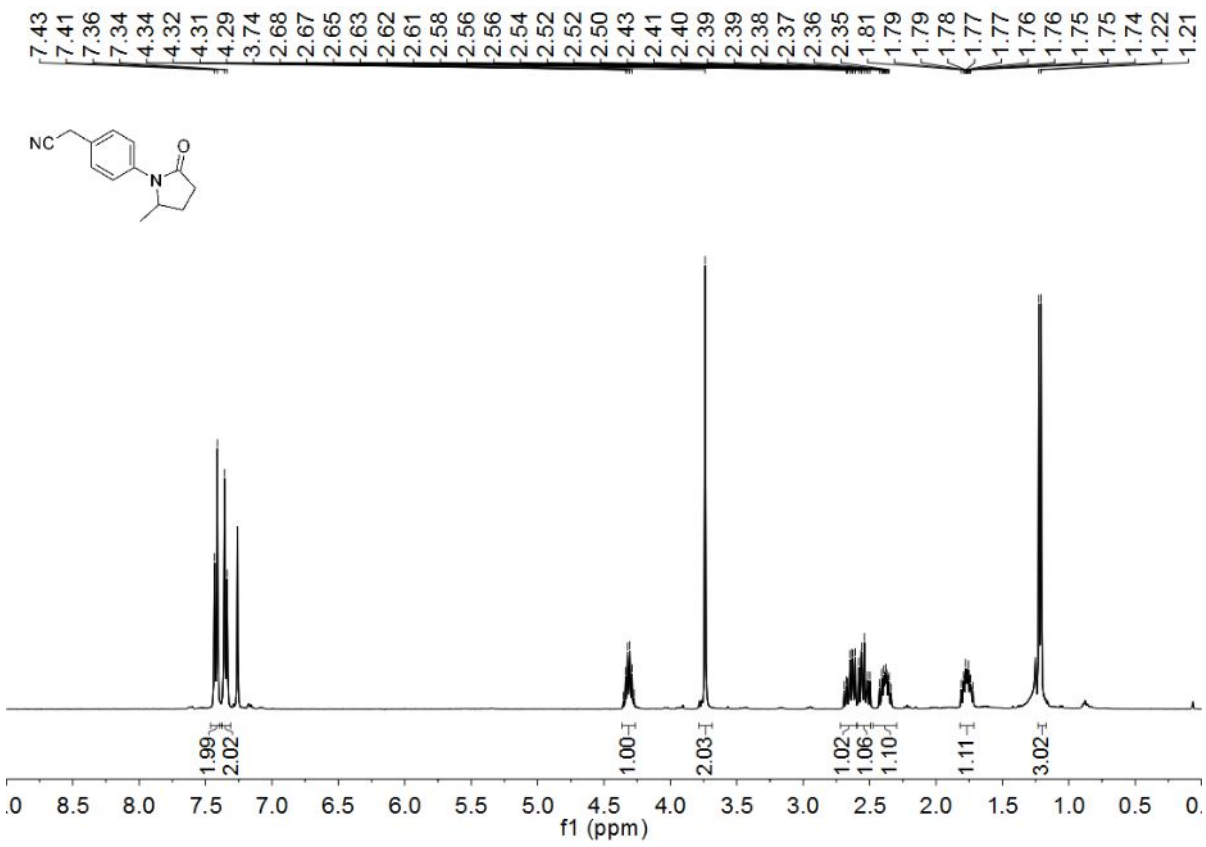
77.02
76.70
54.93
31.40
26.47
26.37
19.81
f1 (ppm)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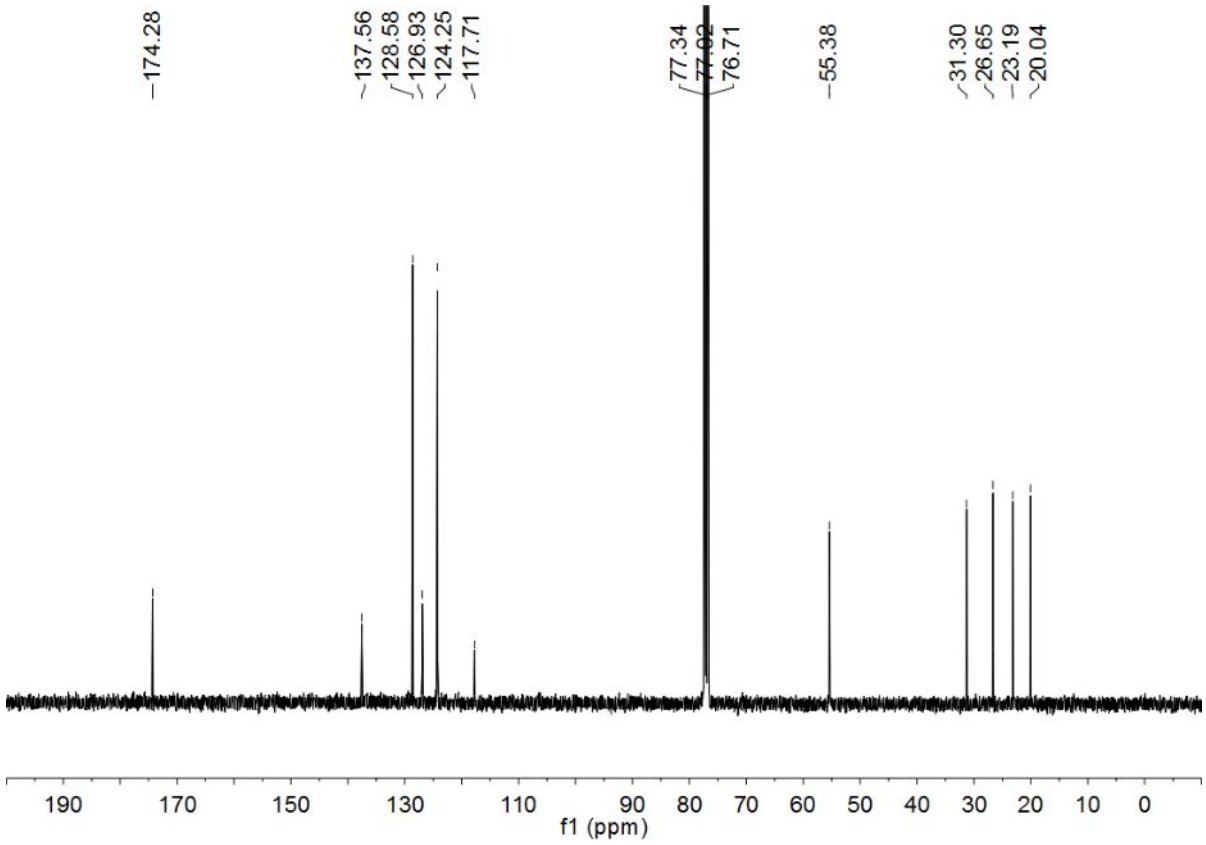
1.00
2.03
1.02
1.06
1.10
1.11
3.02
f1 (ppm)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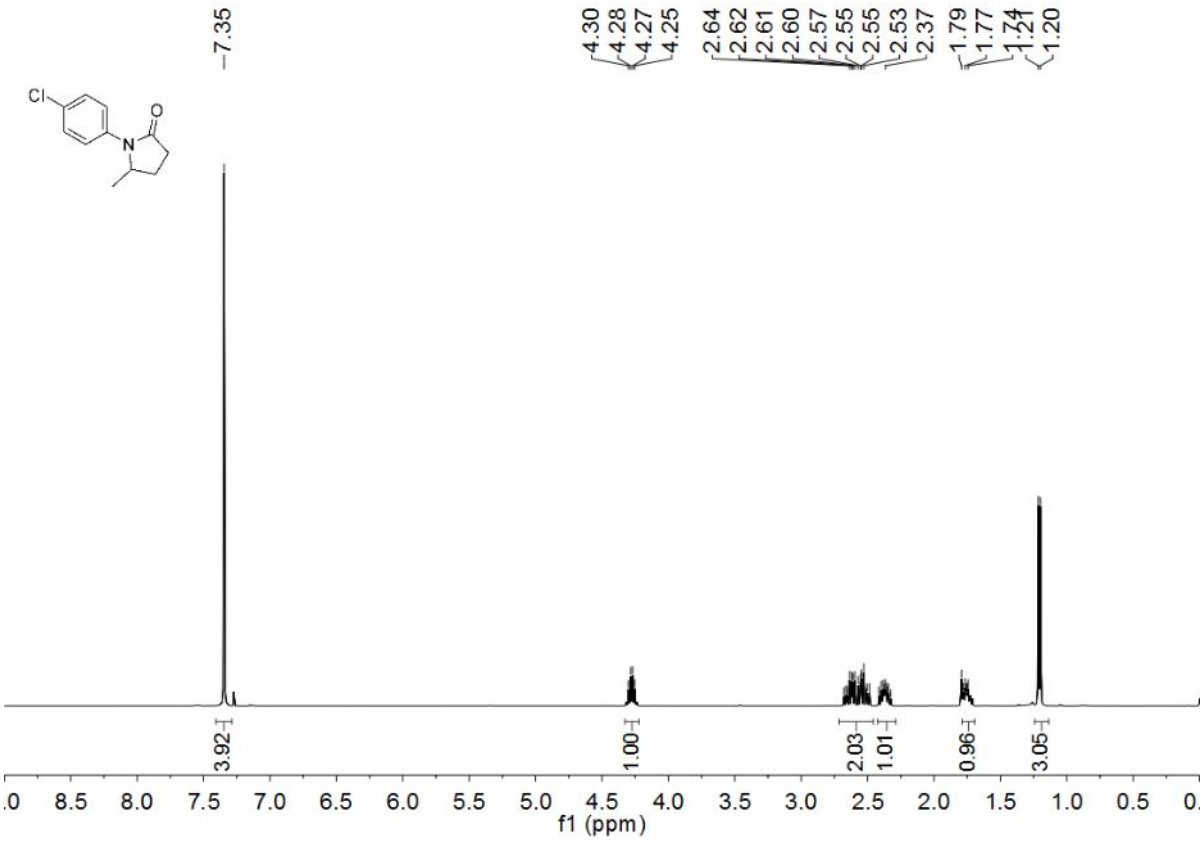
117.71
77.34
77.02
76.71
55.38
31.30
26.65
23.19
20.04
f1 (ppm)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4.28
4.27
4.25
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2.62
2.61
2.60
2.57
2.55
2.55
2.53
2.37
1.79
1.77
1.74
1.21
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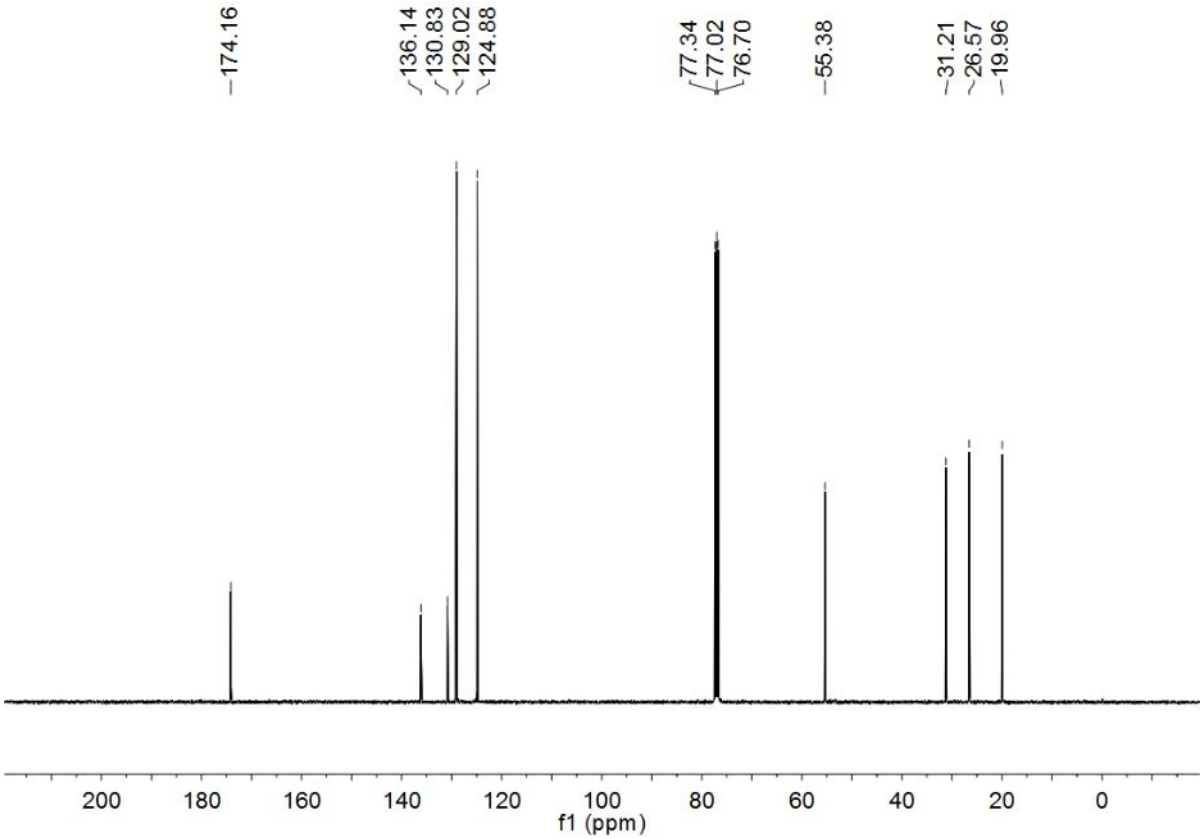
Cl
O
N
3.92
1.00
2.03
1.01
0.96
3.05
f1 (ppm)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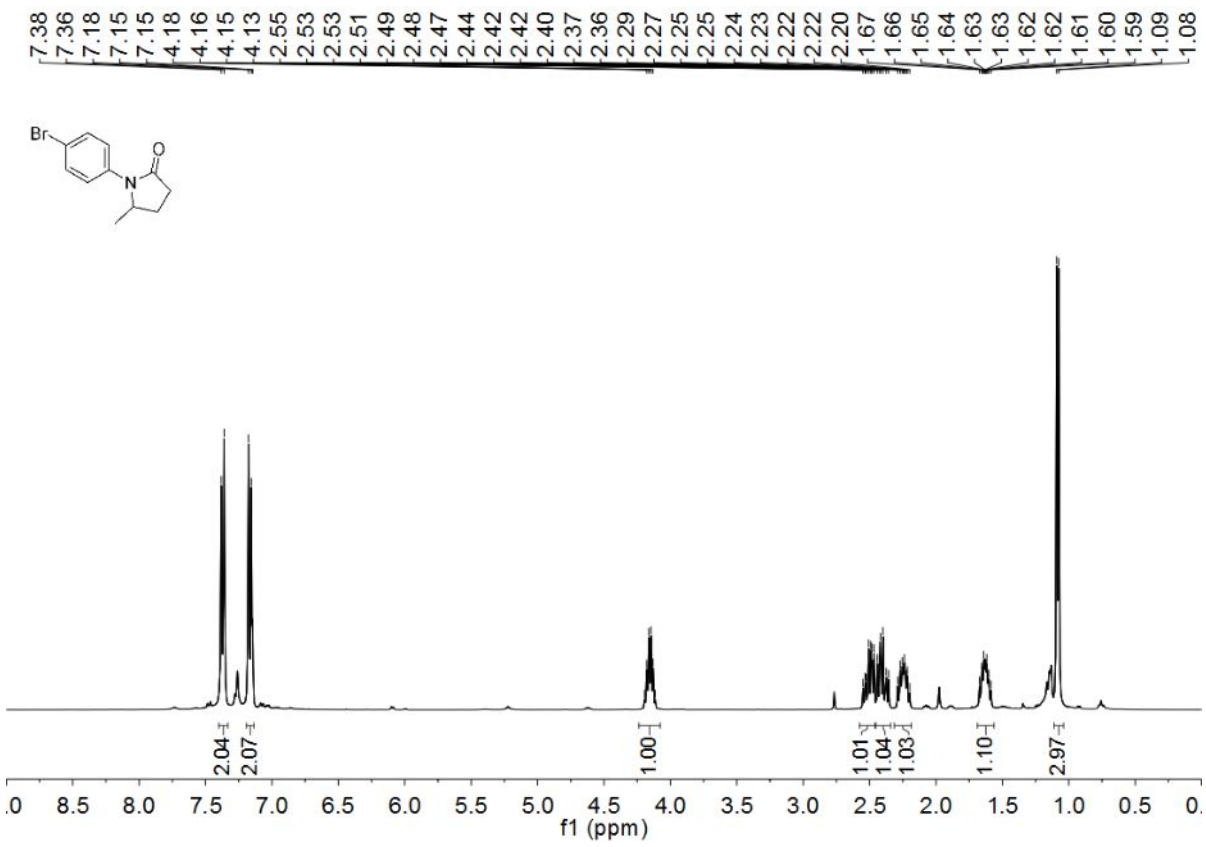
77.34
77.02
76.70
55.38
31.21
26.57
19.96
f1 (ppm)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4.15
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2.53
2.51
2.49
2.48
2.47
2.44
2.42
2.42
2.40
2.37
2.36
2.29
2.27
2.25
2.25
2.24
2.23
2.22
2.22
2.20
1.67
1.66
1.65
1.64
1.63
1.63
1.62
1.62
1.61
1.60
1.59
1.09
1.08
Br
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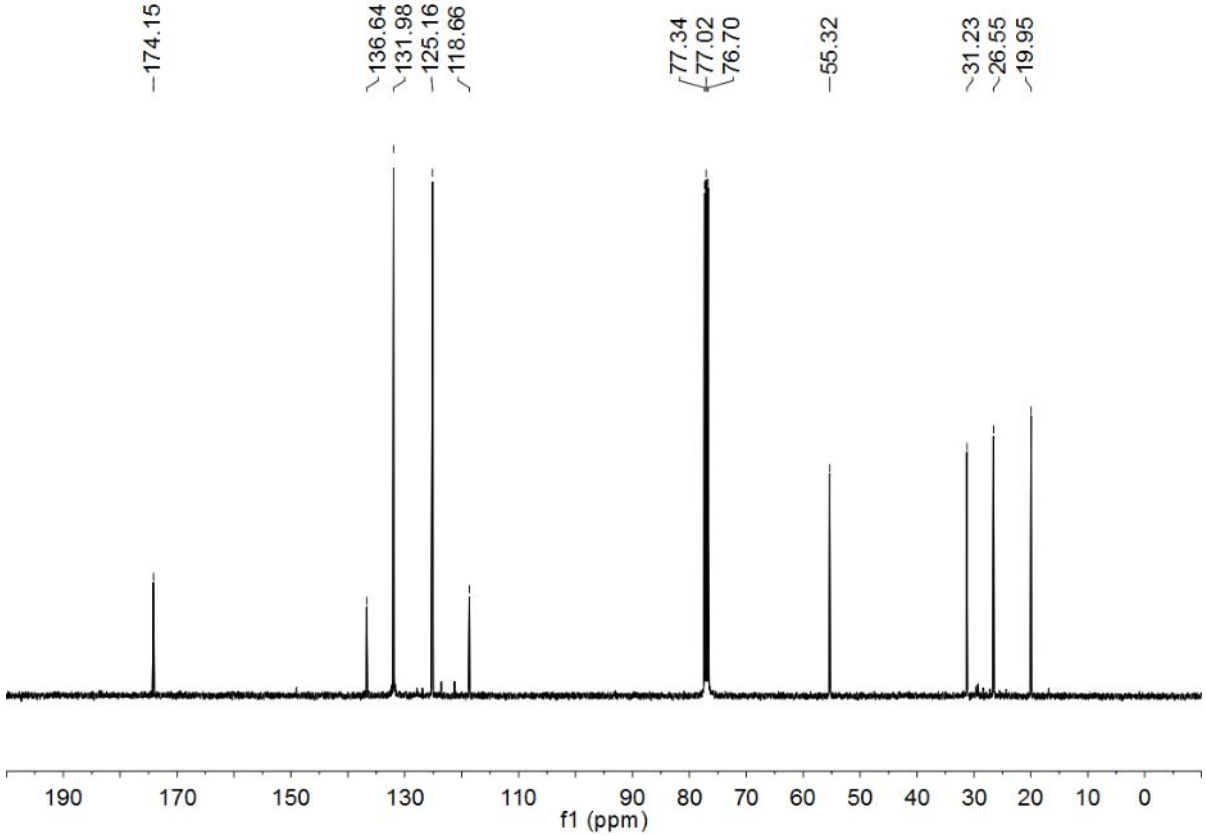
N
2.04
2.07
1.00
1.01
1.04
1.03
1.10
2.97
f1 (ppm)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131.98
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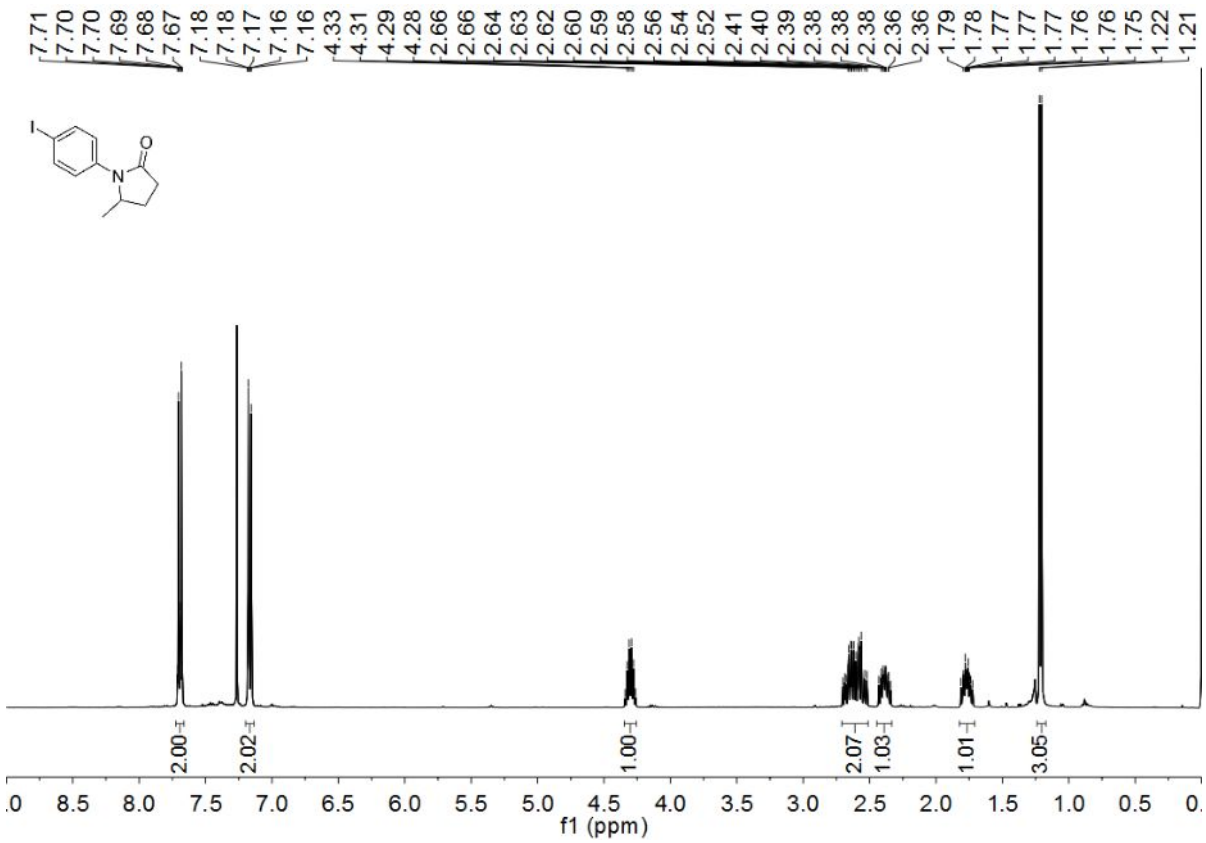
77.34
77.02
76.70
55.32
31.23
26.55
19.95
f1 (ppm)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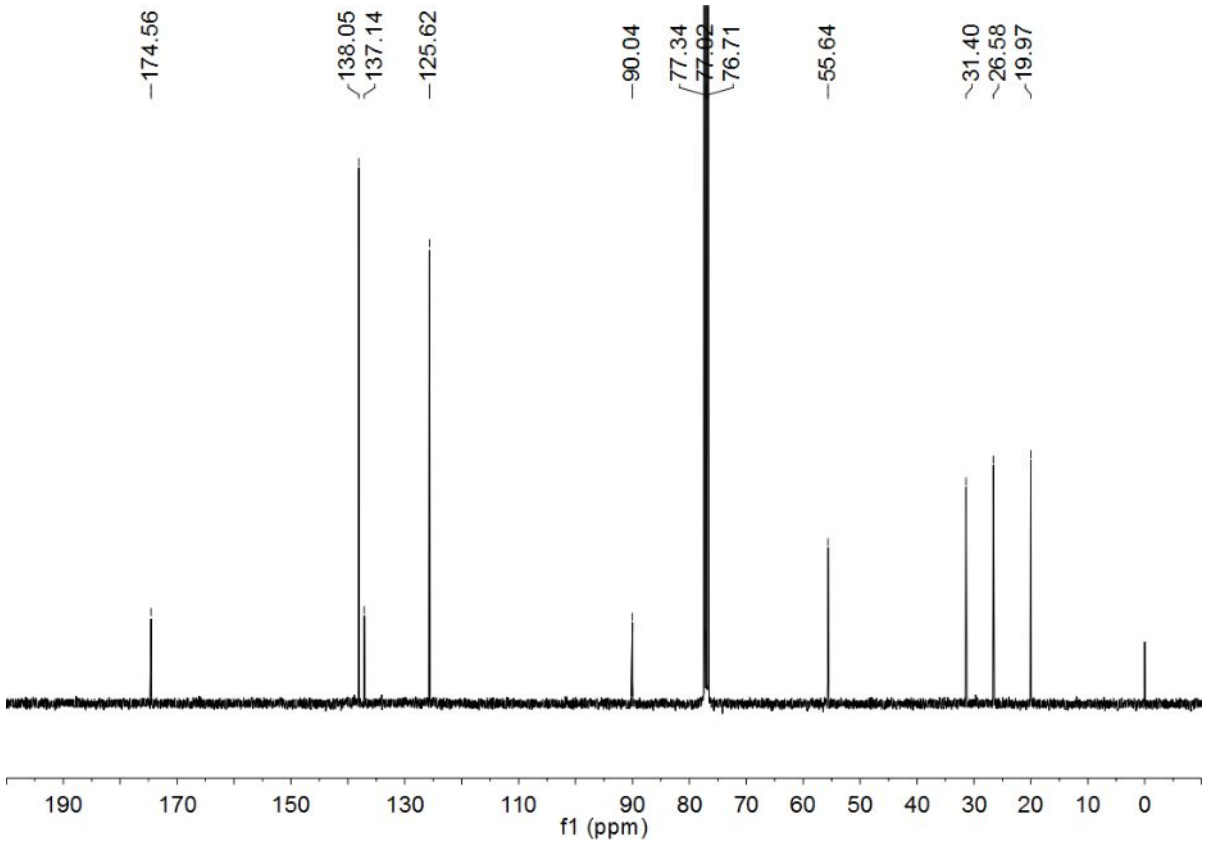
I
O
N
2.00
2.02
1.00
2.07
1.03
1.01
3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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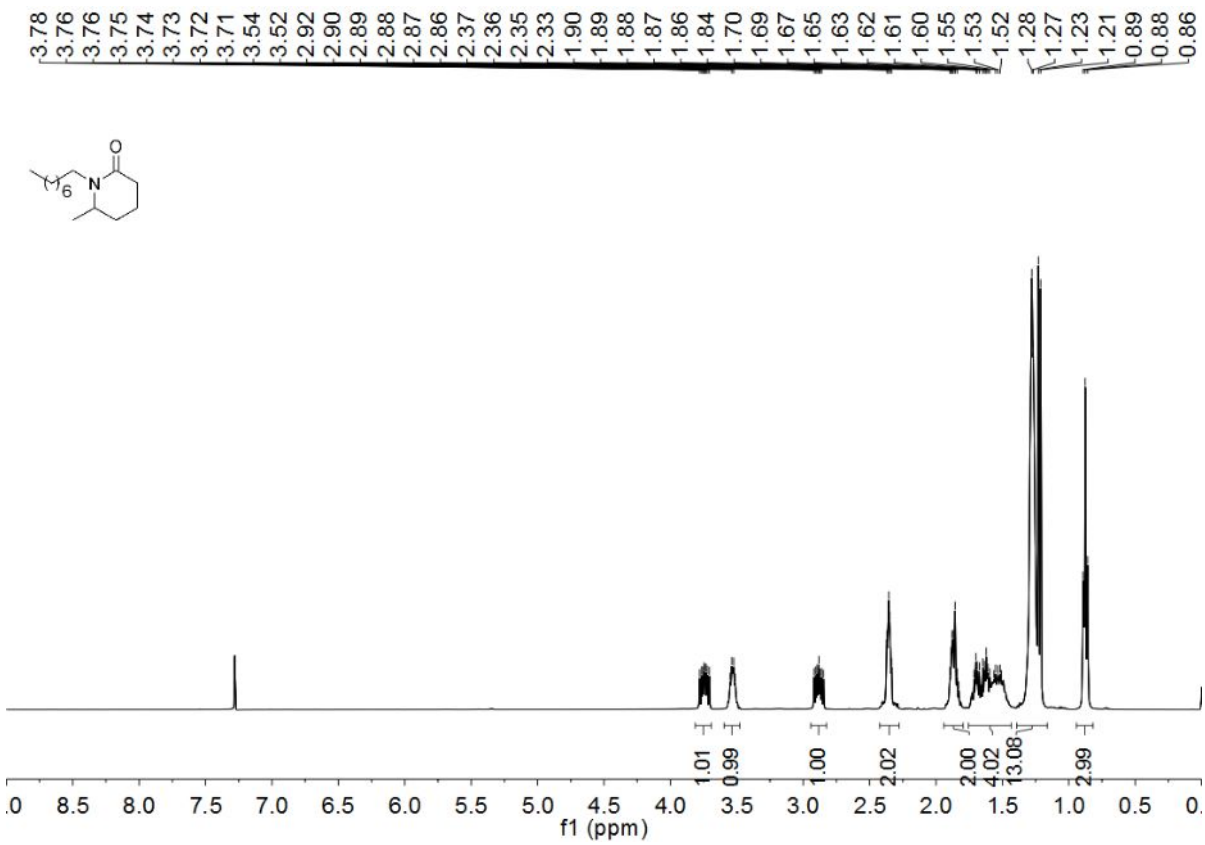
76.71
55.64
31.40
26.58
19.97
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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3.52
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2.89
2.88
2.87
2.86
2.37
2.36
2.35
2.33
1.90
1.89
1.88
1.87
1.86
1.84
1.70
1.69
1.67
1.65
1.63
1.62
1.61
1.60
1.55
1.53
1.52
1.28
1.27
1.23
1.21
0.89
0.88
0.86
O
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6
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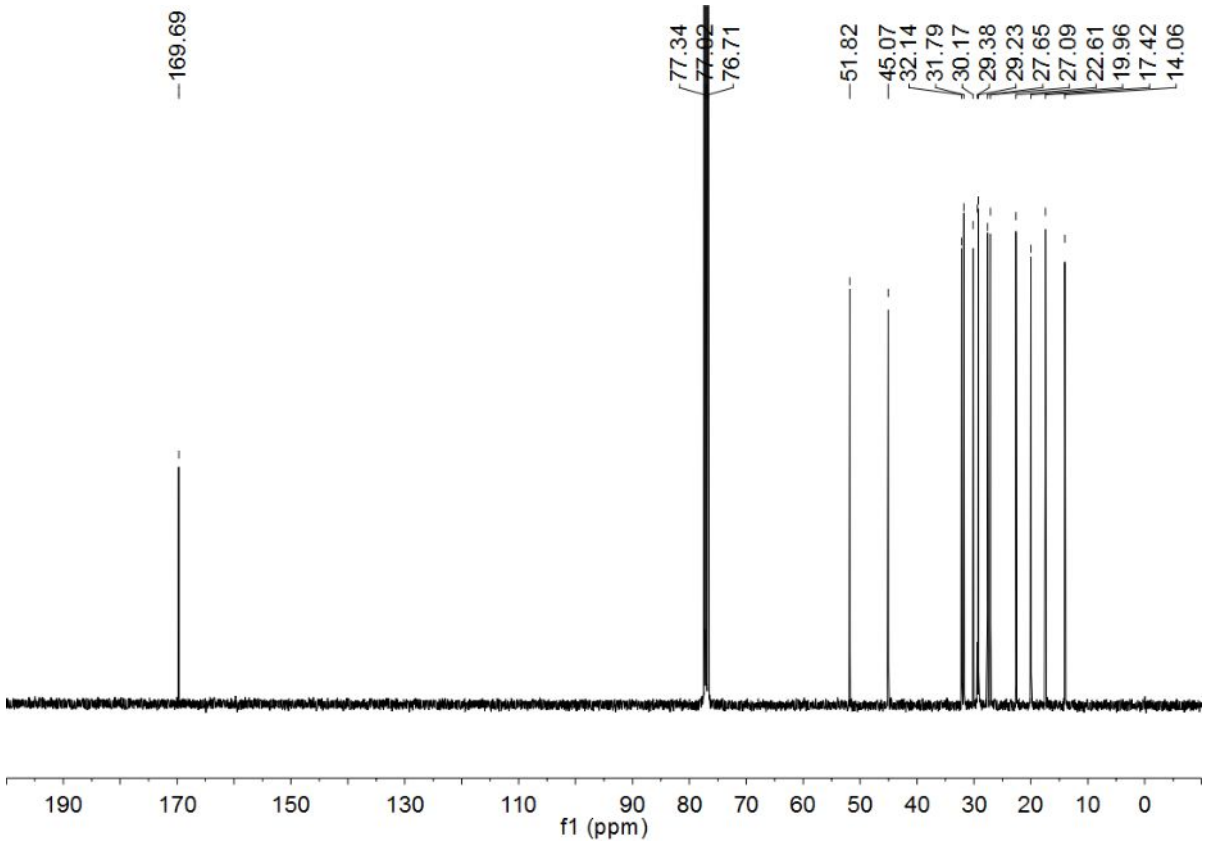
0.99
1.00
2.02
2.00
4.02
13.08
2.99
f1 (ppm)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45.07
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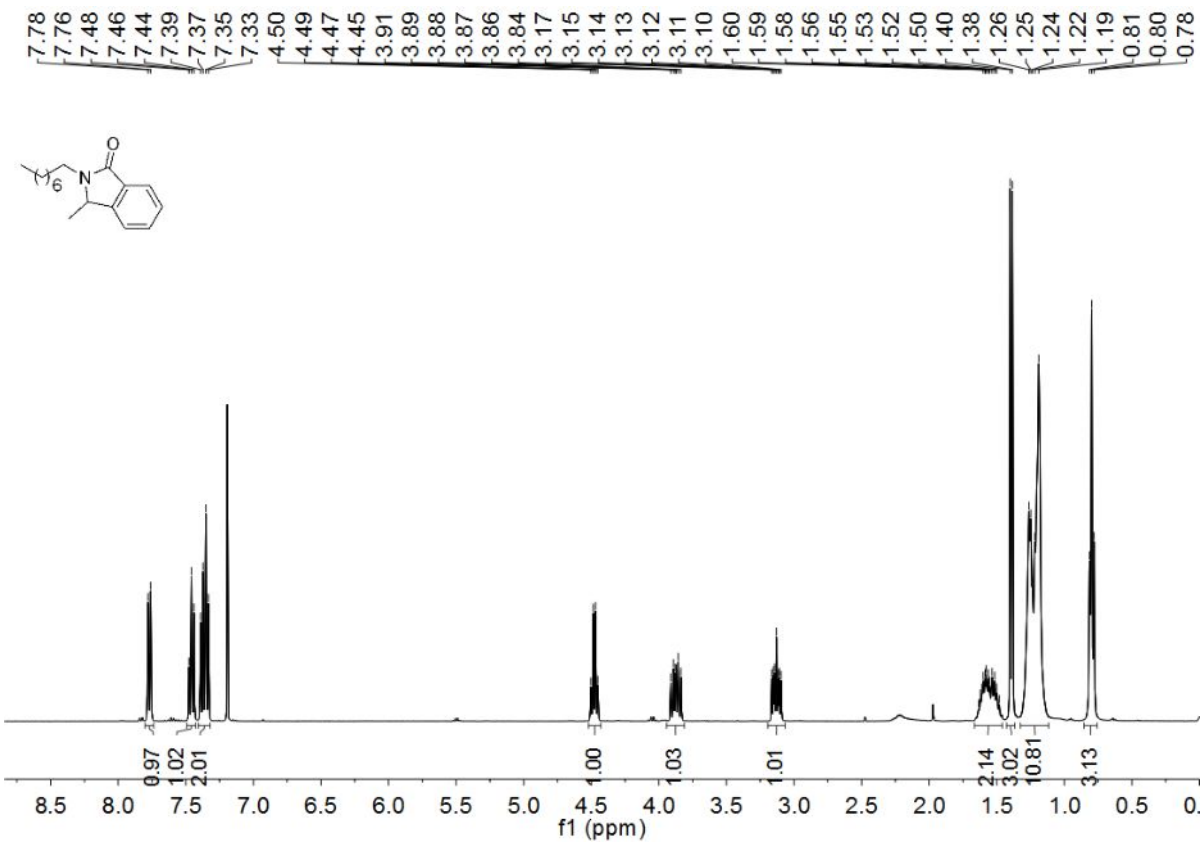
27.09
22.61
19.96
17.42
14.06
f1 (ppm)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4.49
4.47
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3.91
3.89
3.88
3.87
3.86
3.84
3.17
3.15
3.14
3.13
3.12
3.11
3.10
1.60
1.59
1.58
1.56
1.55
1.53
1.52
1.50
1.40
1.38
1.26
1.25
1.24
1.22
1.19
0.81
0.80
0.78
O
N
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0.97
1.02
2.01
1.00
1.03
1.01
2.14
3.02
10.81
3.13
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8.0
7.5
7.0
6.5
6.0
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5.0
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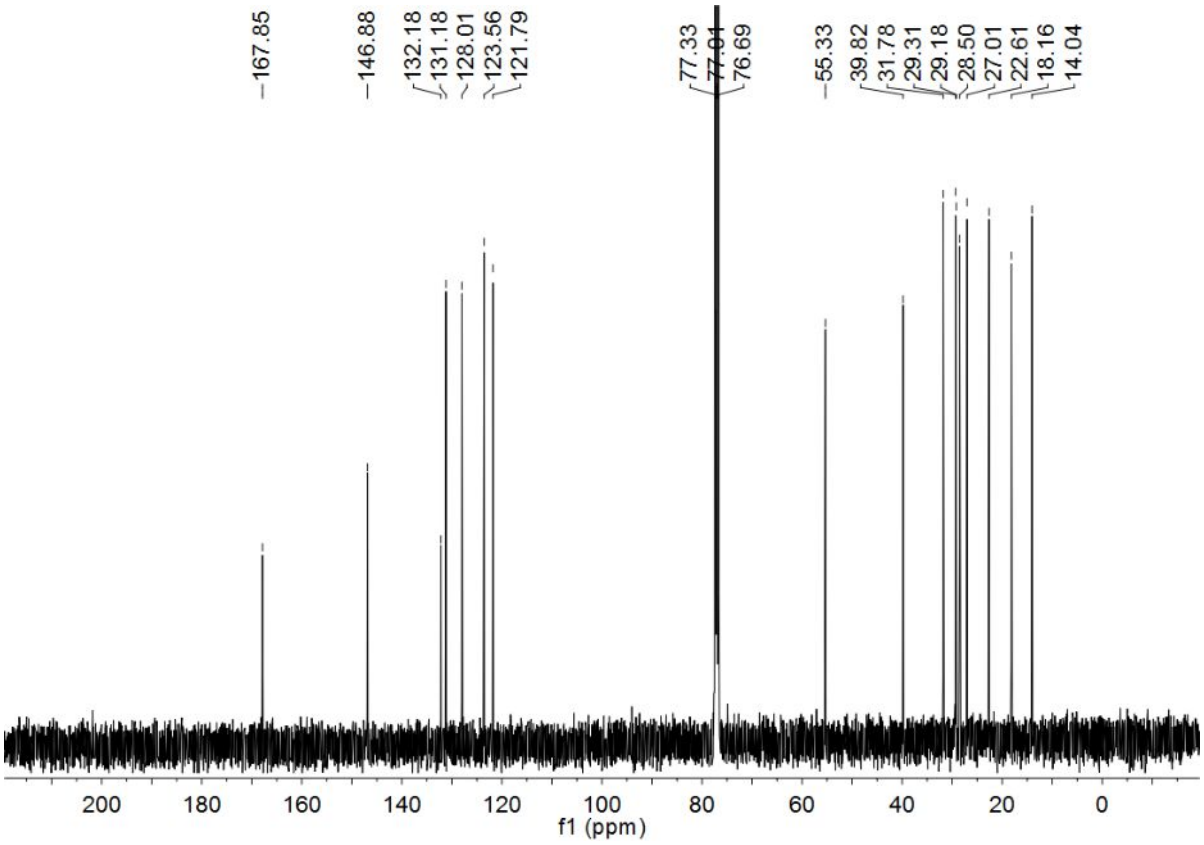
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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39.82
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29.31
29.18
28.50
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18.16
14.04
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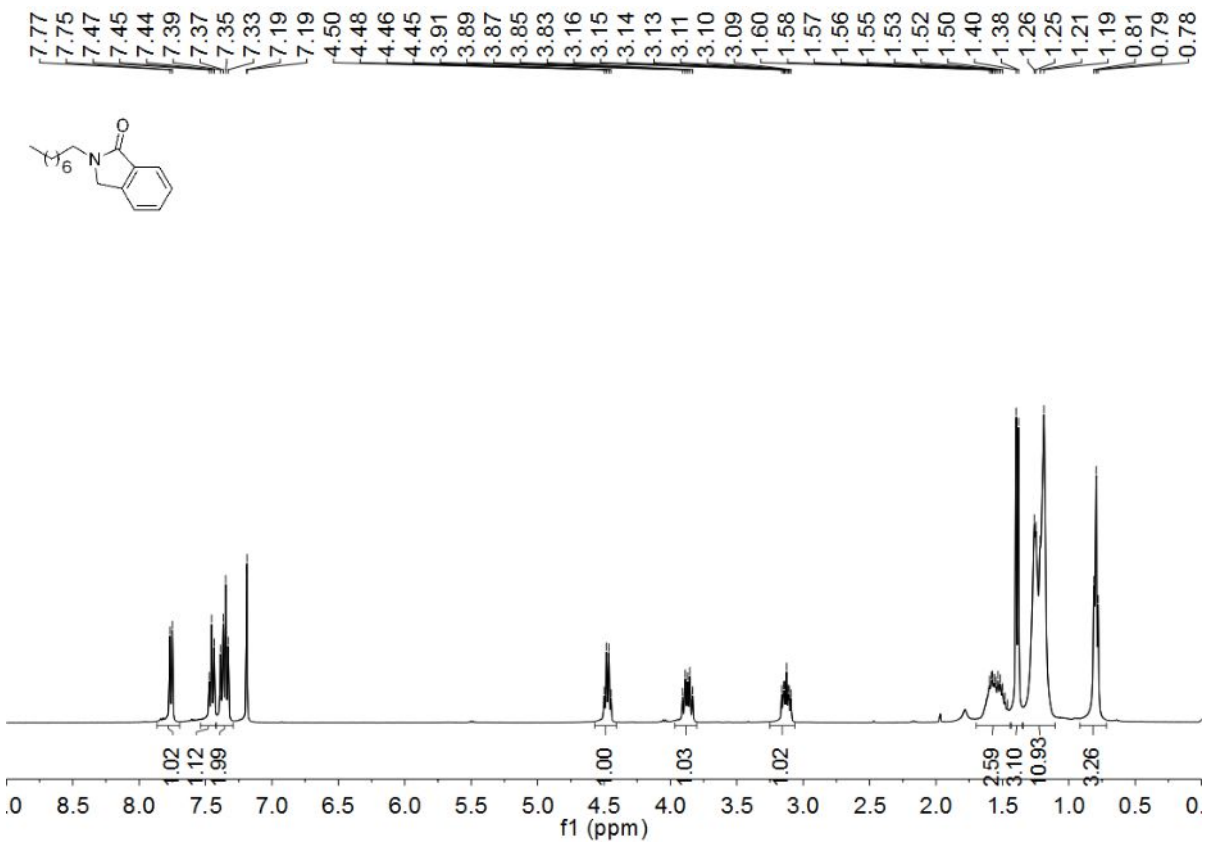
180
160
140
120
100
80
60
40
20
0
f1 (ppm)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7.19
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3.89
3.87
3.85
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3.14
3.13
3.11
3.10
3.09
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1.58
1.57
1.56
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1.53
1.52
1.50
1.40
1.38
1.26
1.25
1.21
1.19
0.81
0.79
0.78
O
N
6
1.02
1.12
1.99
1.00
1.03
1.02
2.59
3.10
10.93
3.26
f1 (ppm)
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141.12
133.18
131.03
127.95
123.67
122.59
77.33
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29.30
29.18
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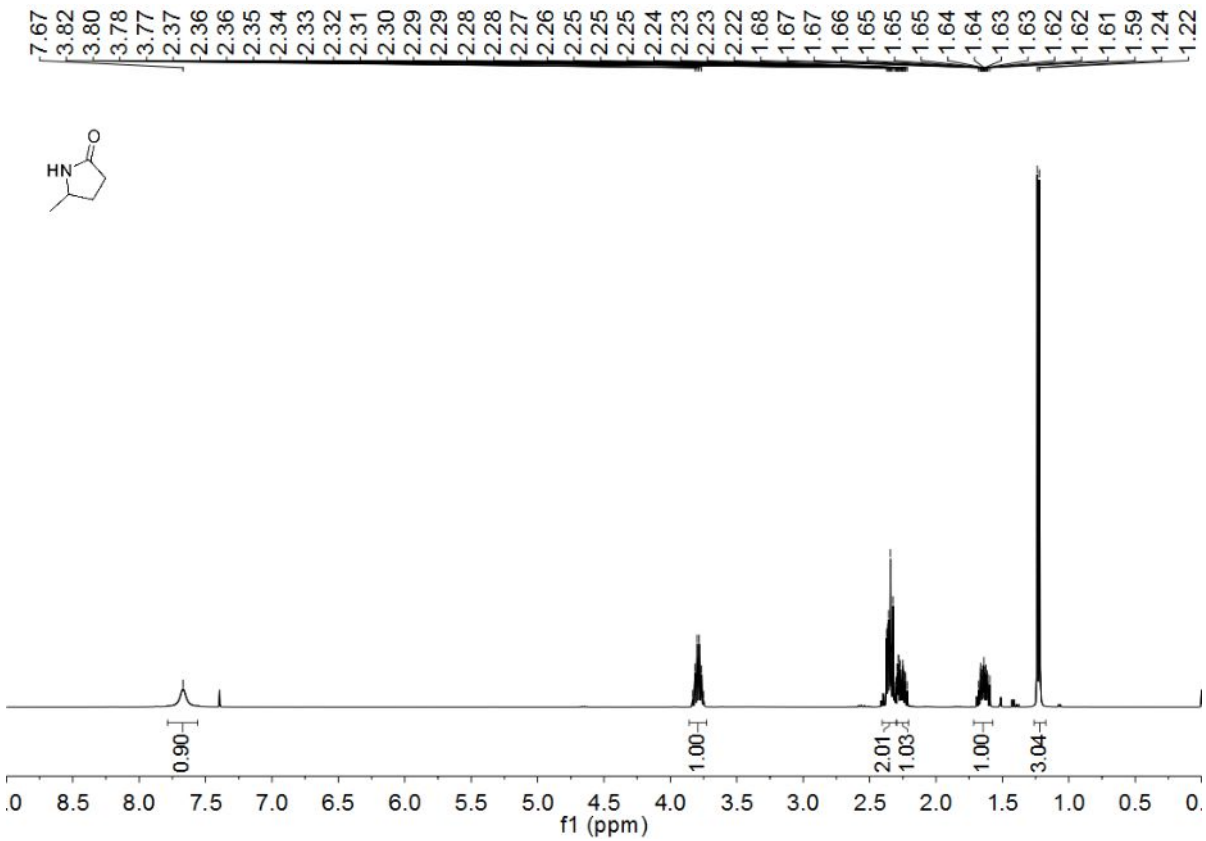
26.90
22.62
14.04
f1 (ppm)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1.62
1.62
1.61
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1.22
HN
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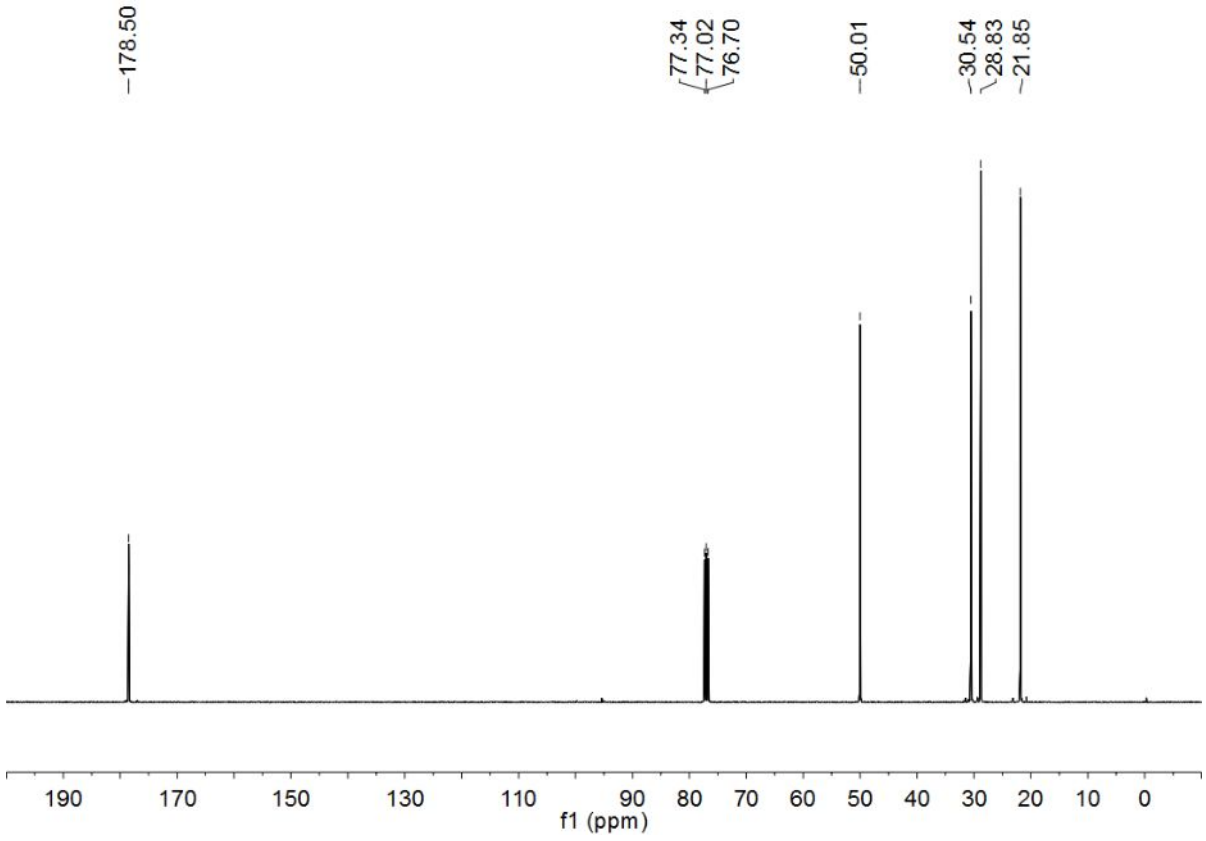
1.03
1.00
3.04
f1 (ppm)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76.70
50.01
30.54
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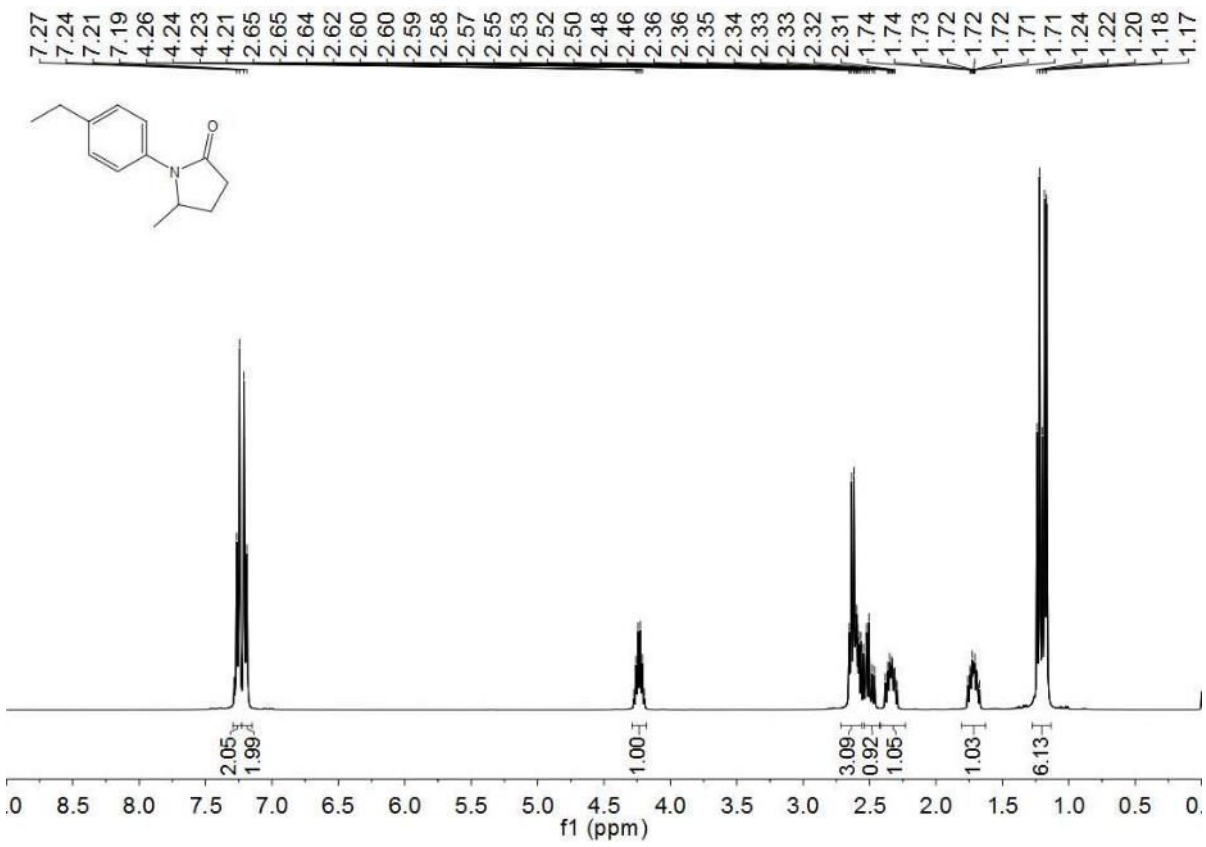
21.85
f1 (ppm)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2.32
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1.74
1.73
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1.72
1.72
1.71
1.71
1.24
1.22
1.20
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3.09
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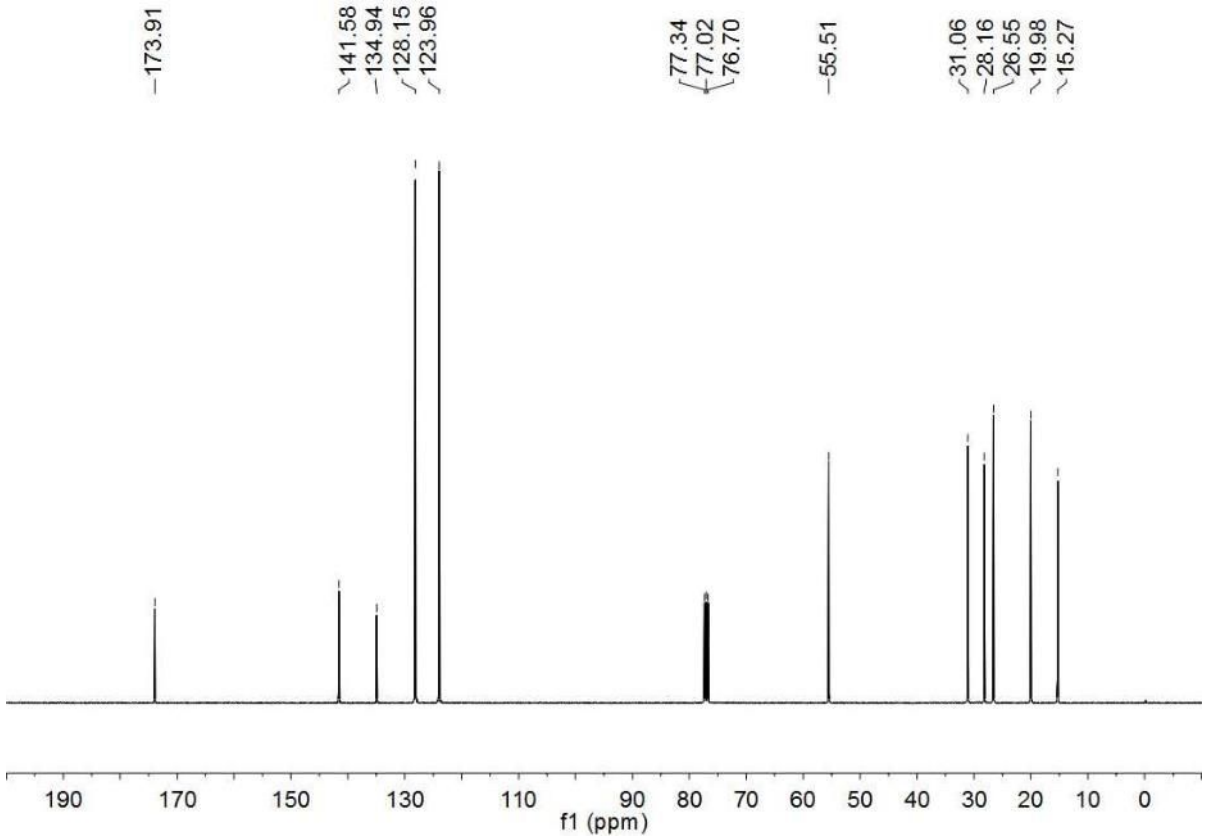
6.13
f1 (ppm)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28.16
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19.98
15.27
f1 (ppm)