

Supporting Information

1,2-Arylalkylation of *N*-(arylsulfonyl)acrylamides with aliphatic aldehyde as alkyl source

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1. General experimental details

General Information: All chemicals were used as received without further purification unless stated otherwise. NMR spectra were recorded at ambient temperature on a 400 MHz NMR spectrometer. Chemical shifts (δ) are given in ppm relative to TMS, the coupling constants J are given in Hz. HRMS were recorded on a TOF LC/MS equipped with electrospray ionization (ESI) probe operating in positive or negative ion mode.

Experimental procedure for the procedure for 1,2-arylalkylation of *N*-(arylsulfonyl)acrylamide with aldehyde: Under N_2 , the mixture of *N*-(arylsulfonyl)acrylamide **1** (0.2 mmol), aldehyde **2** (0.6 mmol), DTBP (0.6 mmol) and PhH (2 mL) were added into the sealed tube. The reaction mixture was vigorously stirred at 120 °C for 20 h. Then, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on silica gel to give the products.

2. Mechanism studies

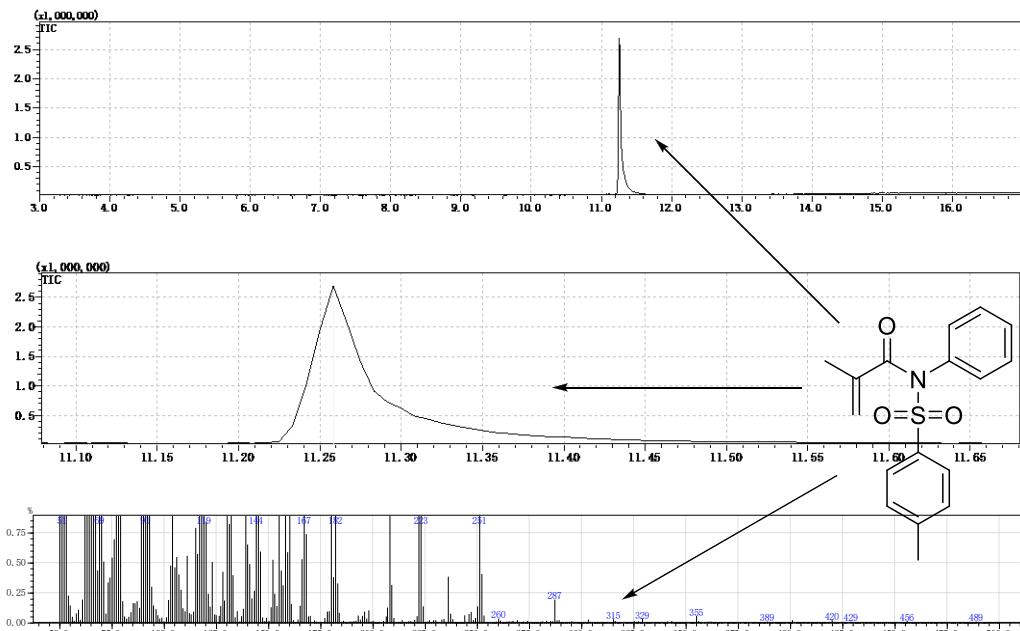
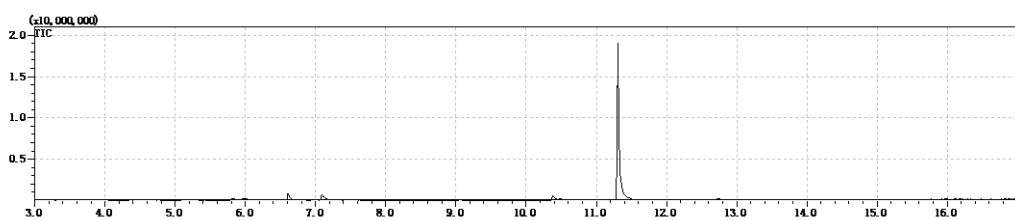


Figure S1 GC-MS spectra of the **1a**



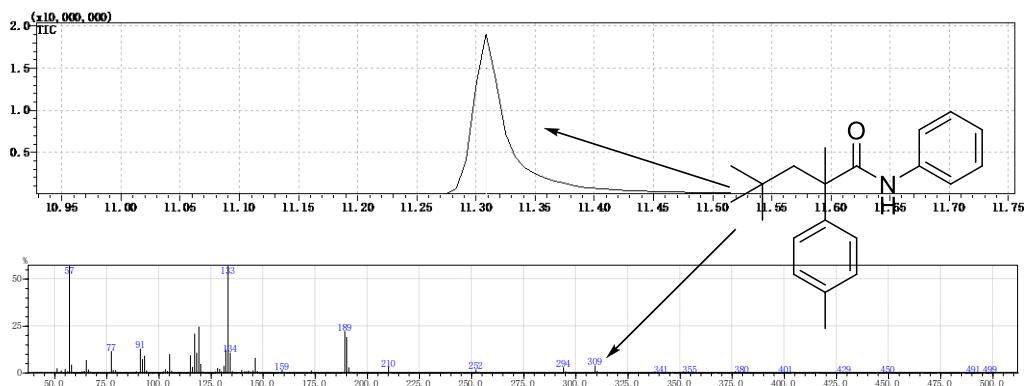


Figure S2 GC-MS spectra of the 3aa

Standard Procedure + TEMPO (3.0 equiv)

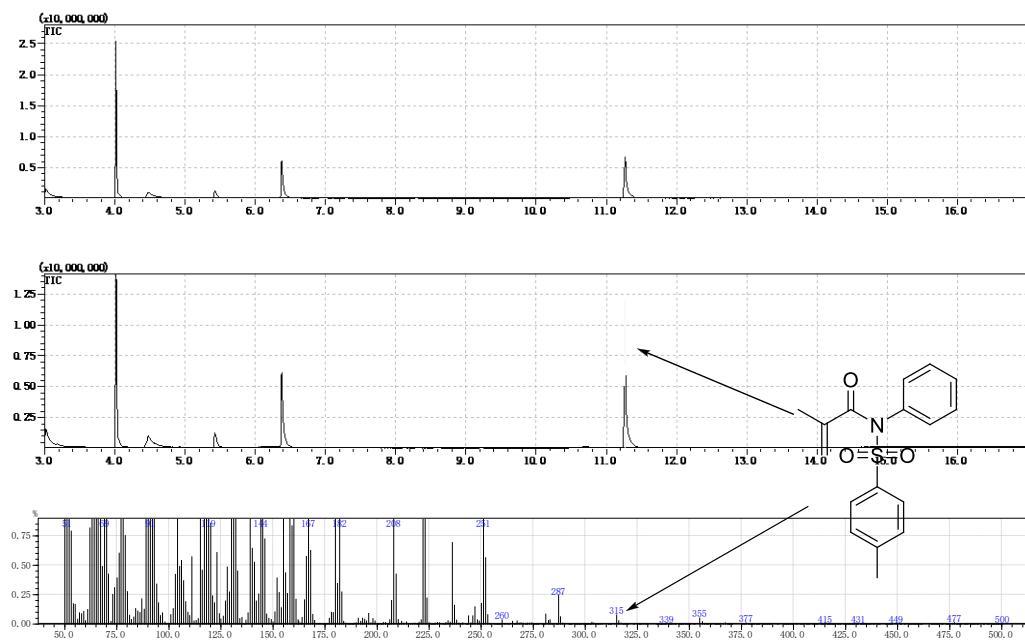
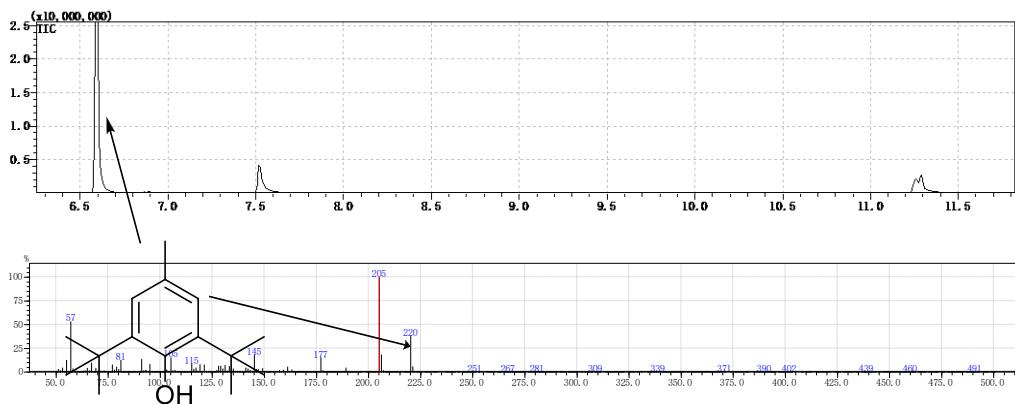


Figure S3 GC-MS spectra of the free radical capture results

Standard Procedure + BHT (3.0 equiv)



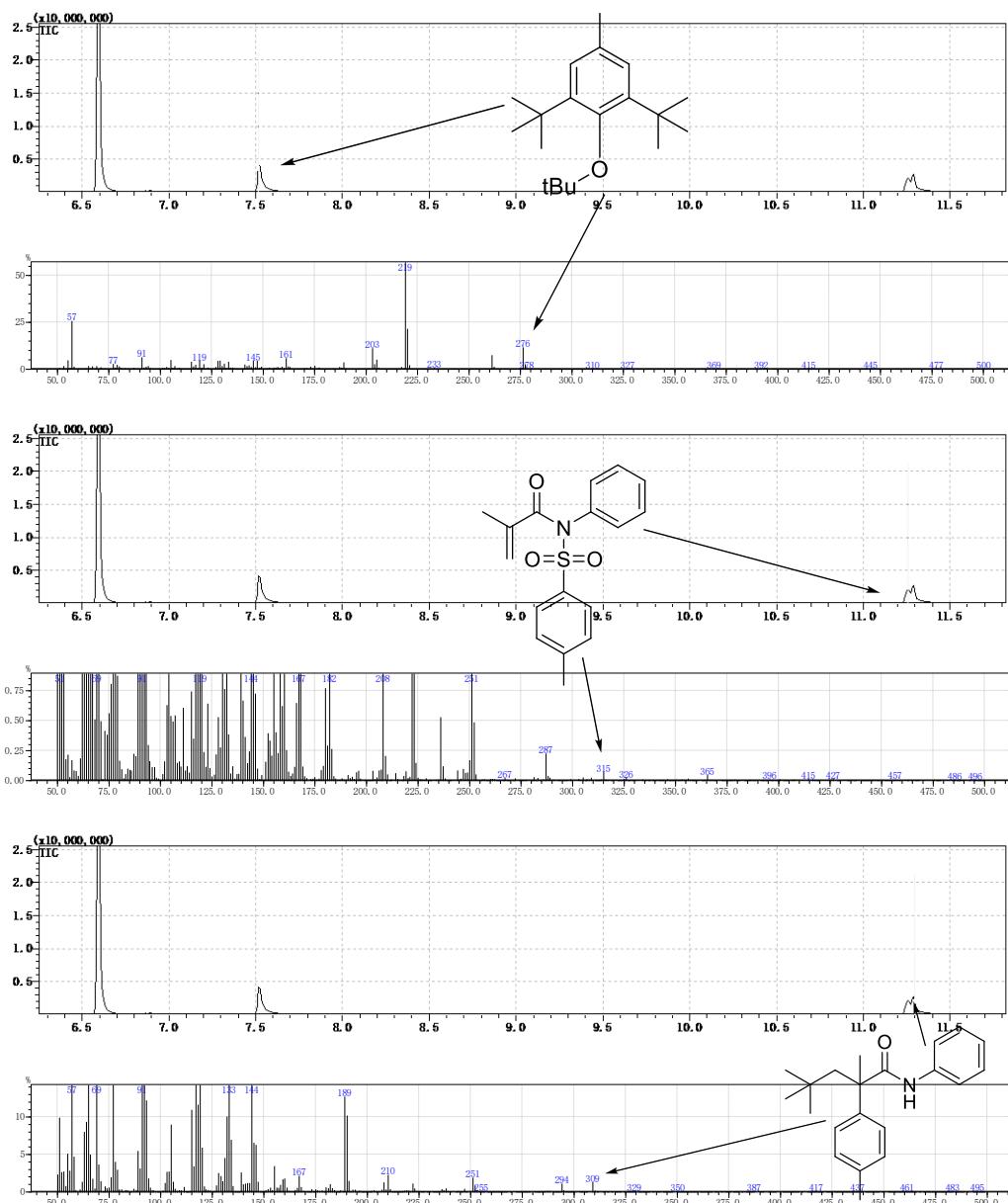


Figure S4 GC-MS spectra of the free radical capture results

3. Test for the evolution of CO and SO₂ gas

A piece of test strip prepared from PMA (phosphomolybdic acid)-PdCl₂ solution¹¹ was put into the reaction tube and sealed. After the completion of the reaction, the color of the test strip was changed from light yellow to dark blue (Figure S5a).

After the completion of the reaction, the reaction mixture was cooled to room temperature. A piece of test strip prepared from magenta solution was put into the reaction tube and sealed. The magenta was fade rapidly (Figure S5b).

¹ A. Verma and S. kumar, *Org. Lett.*, 2016, **18**, 4388.

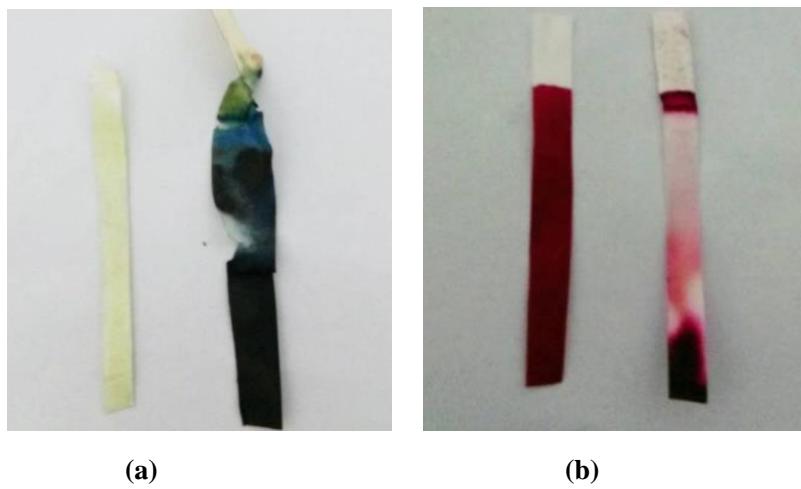
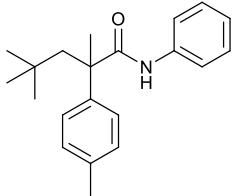


Figure 5 **a).** The PMA-PdCl₂ test strip as prepared (left) and the PMA-PdCl₂ test strip in the reaction tube (right). **b)** The magenta test strip as prepared (left) and the magenta test strip that put into the reaction tube (right).

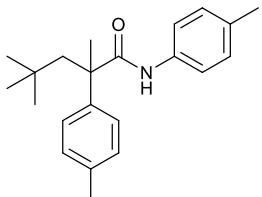
2. Characterization data of the products

2,4,4-trimethyl-N-phenyl-2-(p-tolyl)pentanamide (3aa)



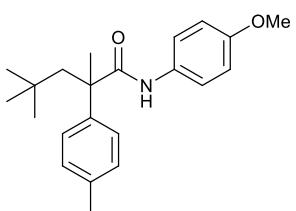
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (71%, 43.9 mg). m.p. 121-123 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.38-7.35 (m, 4H), 7.29-7.25 (m, 2H), 7.20 (d, J = 8.1 Hz, 2H), 7.07 (d, J = 7.3 Hz, 1H), 6.88 (s, 1H), 2.38 (s, 3H), 2.35 (d, J = 14.7 Hz, 1H), 2.15 (d, J = 14.7 Hz, 1H), 1.78 (s, 3H), 0.91 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.9, 141.9, 138.0, 136.9, 129.5, 128.9, 126.9, 124.0, 119.7, 51.5, 50.4, 31.9, 31.7, 25.2, 21.0. IR (cm^{-1}): 3405, 3018, 2951, 2867, 1666, 1597, 1518, 1499, 1436, 1308, 1242, 1177. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{28}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 310.2165, found 310.2167.

2,4,4-trimethyl-N,2-di-p-tolylpentanamide (3ba)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (70%, 45.2 mg). m.p. 145-147 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.33 (d, J = 7.4 Hz, 2H), 7.23 (d, J = 7.5 Hz, 2H), 7.17 (d, J = 7.6 Hz, 2H), 7.06 (d, J = 7.8 Hz, 2H), 6.80 (s, 1H), 2.35 (s, 3H), 2.32 (d, J = 14.9 Hz, 1H), 2.27 (s, 3H), 2.11 (d, J = 14.7 Hz, 1H), 1.75 (s, 3H), 0.88 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.8, 142.0, 136.8, 135.5, 133.6, 129.4, 129.3, 126.9, 119.7, 51.5, 50.4, 31.9, 31.7, 25.2, 21.0, 20.8. IR (cm^{-1}): 3408, 3016, 2950, 2867, 1659, 1594, 1515, 1480, 1402, 1309, 1241, 1191. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{30}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 324.2322, found 324.2323.

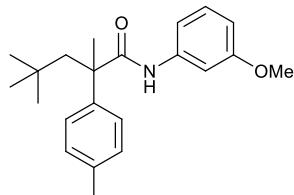
N-(4-methoxyphenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3ca)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a yellow solid (70%, 47.5 mg). m.p. 117-119 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.36 (d, J = 8.3 Hz, 2H), 7.29-7.24 (m, 2H), 7.19 (d, J = 8.1 Hz, 2H), 6.83-6.79 (m, 3H), 3.77 (s, 3H), 2.37 (s, 3H), 2.34 (d,

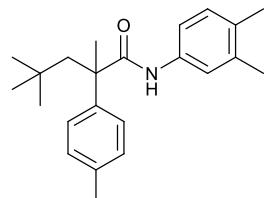
J = 14.7 Hz, 1H), 2.12 (d, *J* = 14.7 Hz, 1H), 1.77 (s, 3H), 0.90 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.8, 156.2, 142.1, 136.8, 131.2, 129.4, 126.9, 121.5, 114.0, 55.5, 51.3, 50.4, 31.9, 31.7, 25.2, 21.0. IR (cm^{-1}): 3405, 3339, 3013, 2951, 2868, 2834, 1659, 1597, 1513, 1466, 1441, 1409, 1365, 1299, 1244, 1179, 1036. HRMS (ESI) *m/z* calcd for $\text{C}_{22}\text{H}_{30}\text{NO}_2$ ($\text{M}+\text{H}$) $^+$ 340.2271, found 340.2272.

N-(3-methoxyphenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3da)



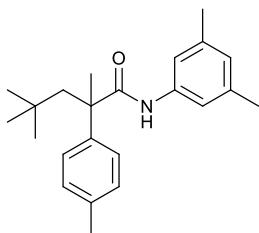
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a light yellow solid (68%, 46.1 mg). m.p. 110-112 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.33 (d, *J* = 8.2 Hz, 2H), 7.24-7.23 (m, 1H), 7.18 (d, *J* = 8.1 Hz, 2H), 7.12 (t, *J* = 8.1 Hz, 1H), 6.85 (s, 1H), 6.71 (dd, *J* = 8.0, 1.2 Hz, 1H), 6.60 (dd, *J* = 8.2, 2.4 Hz, 1H), 3.78 (s, 3H), 2.36 (s, 3H), 2.32 (d, *J* = 14.7 Hz, 1H), 2.13 (d, *J* = 14.7 Hz, 1H), 1.75 (s, 3H), 0.88 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 176.0, 160.1, 141.7, 139.4, 136.9, 129.5, 126.9, 111.6, 110.0, 105.1, 55.3, 51.6, 50.3, 31.9, 31.7, 25.1, 21.0. IR (cm^{-1}): 3404, 3015, 2951, 2865, 1674, 1606, 1525, 1491, 1450, 1424, 1287, 1206, 1157, 1042. HRMS (ESI) *m/z* calcd for $\text{C}_{22}\text{H}_{30}\text{NO}_2$ ($\text{M}+\text{H}$) $^+$ 340.2271, found 340.2273.

N-(3,4-dimethylphenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3ea)



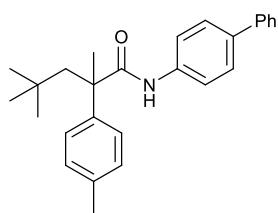
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a yellow liquid (74%, 49.8 mg). m.p. 131-132 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.34 (d, *J* = 8.2 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 3H), 7.09-7.06 (m, 1H), 7.01 (d, *J* = 8.1 Hz, 2H), 6.79 (s, 1H), 2.36 (s, 3H), 2.33 (d, *J* = 14.7 Hz, 1H), 2.21 (s, 3H), 2.19 (s, 3H), 2.12 (d, *J* = 14.7 Hz, 1H), 1.75 (s, 3H), 0.89 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.7, 142.1, 137.1, 136.8, 135.8, 132.3, 129.8, 129.4, 126.9, 120.9, 117.1, 51.5, 50.5, 31.9, 31.7, 25.3, 21.0, 19.9, 19.2. IR (cm^{-1}): 3407, 3342, 3022, 2949, 2866, 1660, 1592, 1514, 1481, 1448, 1403, 1384, 1306, 1210, 1193, 1019. HRMS (ESI) *m/z* calcd for $\text{C}_{23}\text{H}_{32}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 338.2478, found 338.2475.

N-(3,5-dimethylphenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3fa)



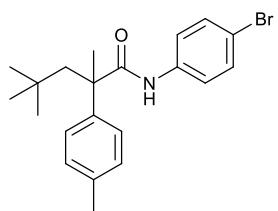
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (51%, 34.4 mg). m.p. 75-77 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.69 (d, J = 8.2 Hz, 1H), 7.38 (d, J = 8.2 Hz, 2H), 7.19 (d, J = 8.1 Hz, 2H), 6.97 (d, J = 8.1 Hz, 1H), 6.88 (s, 1H), 6.68 (s, 1H), 2.37 (d, J = 14.8 Hz, 1H), 2.36 (s, 3H), 2.24 (s, 3H), 2.16 (d, J = 14.8 Hz, 1H), 1.79 (s, 3H), 1.78 (s, 3H), 0.87 (s, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 176.0, 141.8, 136.8, 134.1, 133.5, 130.9, 129.4, 128.1, 127.2, 127.1, 121.9, 51.6, 49.9, 31.9, 31.8, 24.8, 21.0, 20.8, 17.0. IR (cm⁻¹): 3418, 3338, 3020, 2951, 2866, 1682, 1594, 1514, 1479, 1396, 1300, 1265, 1222, 1193, 1018. HRMS (ESI) *m/z* calcd for C₂₃H₃₂NO (M+H)⁺ 338.2478, found 338.2481.

N-[(1,1'-biphenyl)-4-yl]-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3ga)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a yellow liquid (81%, 62.4 mg). m.p. 156-158 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.57-7.51 (m, 4H), 7.47-7.31 (m, 7H), 7.21 (d, J = 8.0 Hz, 2H), 6.87 (s, 1H), 2.39 (s, 3H), 2.36 (d, J = 14.6 Hz, 1H), 2.17 (d, J = 14.6 Hz, 1H), 1.80 (s, 3H), 0.93 (s, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 175.9, 141.9, 140.6, 137.5, 136.95, 136.93, 129.5, 128.8, 127.5, 127.1, 126.9, 126.8, 119.9, 51.6, 50.4, 31.9, 31.8, 25.2, 21.1. IR (cm⁻¹): 3412, 3343, 3028, 2951, 2867, 1678, 1590, 1522, 1486, 1446, 1401, 1311, 1242, 1191, 1019. HRMS (ESI) *m/z* calcd for C₂₇H₃₂NO (M+H)⁺ 386.2478, found 386.2479.

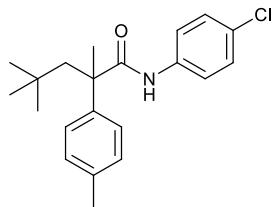
N-(4-bromophenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3ha)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (86%, 66.6 mg). m.p. 159-161 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.37-7.32 (m, 4H), 7.27-7.24 (m, 2H), 7.19 (d, J = 8.0 Hz, 2H), 6.90 (s, 1H), 2.37 (s, 3H), 2.32 (d, J = 14.7 Hz, 1H),

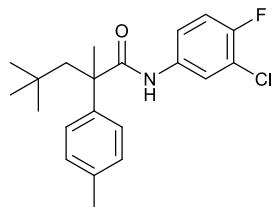
2.13 (d, $J = 14.7$ Hz, 1H), 1.76 (s, 3H), 0.89 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 176.0, 141.6, 137.2, 137.0, 131.8, 129.5, 126.9, 121.3, 116.5, 51.6, 50.3, 31.9, 31.7, 25.1, 21.0. IR (cm^{-1}): 3343, 3025, 2951, 2868, 1663, 1588, 1510, 1489, 1392, 1301, 1240, 1171, 1009. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{27}\text{BrNO} (\text{M}+\text{H})^+$ 388.1271, found 388.1276.

N-(4-chlorophenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3ia)



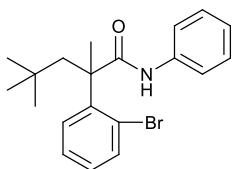
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (71%, 48.7 mg). m.p. 151-152 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.33-6.27 (m, 4H), 7.22-7.17 (m, 4H), 6.84 (s, 1H), 2.35 (s, 3H), 2.30 (d, $J = 14.7$ Hz, 1H), 2.11 (d, $J = 14.7$ Hz, 1H), 1.74 (s, 3H), 0.87 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 176.0, 141.6, 137.0, 136.6, 129.5, 128.9, 128.8, 126.9, 120.9, 51.5, 50.3, 31.9, 31.7, 25.1, 21.0. IR (cm^{-1}): 3402, 3346, 3021, 2952, 2864, 1663, 1592, 1511, 1492, 1395, 1301, 1240, 1174, 1088, 1013. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{27}\text{ClNO} (\text{M}+\text{H})^+$ 344.1776, found 344.1778.

N-(3-chloro-4-fluorophenyl)-2,4,4-trimethyl-2-(p-tolyl)pentanamide (3ja)



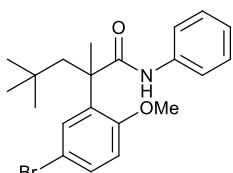
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (70%, 50.5 mg). m.p. 130-131 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.53 (dd, $J = 6.6$, 2.6 Hz, 2H), 7.31 (d, $J = 8.2$ Hz, 2H), 7.18 (d, $J = 8.0$ Hz, 2H), 7.14-7.10 (m, 1H), 7.31 (t, $J = 8.8$ Hz, 1H), 6.85 (s, 1H), 2.36 (s, 3H), 2.29 (d, $J = 14.7$ Hz, 1H), 2.10 (d, $J = 14.7$ Hz, 1H), 1.74 (s, 3H), 0.87 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 176.1, 154.6 (d, $J_{\text{C}-\text{F}} = 244.3$ Hz), 141.4, 137.1, 134.6 (d, $J_{\text{C}-\text{F}} = 3.3$ Hz), 129.6, 126.9, 121.9, 120.9 (d, $J_{\text{C}-\text{F}} = 18.4$ Hz), 119.4 (d, $J_{\text{C}-\text{F}} = 6.7$ Hz), 116.4 (d, $J_{\text{C}-\text{F}} = 21.9$ Hz), 51.5, 50.3, 31.9, 31.7, 25.1, 21.0. IR (cm^{-1}): 3409, 3323, 3026, 2952, 2868, 1659, 1601, 1500, 1386, 1366, 1303, 1259, 1212, 1190, 1057. HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{26}\text{ClFNO} (\text{M}+\text{H})^+$ 362.1681, found 362.1685.

2-(2-bromophenyl)-2,4,4-trimethyl-N-phenylpentanamide (3ka)



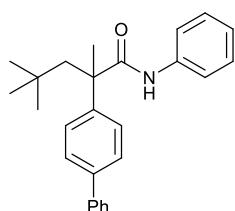
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (71%, 53.0 mg). m.p. 159-161 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.67-7.62 (m, 2H), 7.46-7.42 (m, 1H), 7.37 (d, J = 7.6 Hz, 2H), 7.31-7.26 (m, 2H), 7.25-7.21 (m, 1H), 6.85 (s, 1H), 7.08 (t, J = 7.4 Hz, 1H), 6.77 (s, 1H), 3.78 (s, 1H), 2.88 (d, J = 14.9 Hz, 1H), 2.05 (d, J = 14.9 Hz, 1H), 1.91 (s, 3H), 0.76 (s, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 175.7, 141.6, 138.1, 135.2, 130.1, 129.4, 128.9, 127.8, 125.9, 124.2, 120.3, 53.0, 45.7, 31.9, 31.1, 27.3. IR (cm⁻¹): 3283, 3056, 2982, 2956, 1660, 1596, 1500, 1464, 1438, 1379, 1312, 1252, 1023. HRMS (ESI) *m/z* calcd for C₂₀H₂₅BrNO (M+H)⁺ 374.1114, found 374.1117.

2-(5-bromo-2-methoxyphenyl)-2,4,4-trimethyl-N-phenylpentanamide (3la)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 5/1) gave a light yellow solid (70%, 56.4 mg). m.p. 121-123 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.57 (d, J = 2.4 Hz, 1H), 7.46-7.43 (m, 1H), 7.35 (d, J = 7.7 Hz, 2H), 7.29-7.25 (m, 2H), 7.05 (t, J = 7.3 Hz, 1H), 6.78 (s, 1H), 6.76 (d, J = 8.6 Hz, 1H), 3.72 (s, 3H), 2.48 (d, J = 14.5 Hz, 1H), 1.99 (d, J = 14.5 Hz, 1H), 1.75 (s, 3H), 0.78 (s, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 175.9, 157.2, 138.3, 134.5, 131.7, 131.0, 128.9, 123.8, 119.9, 113.2, 113.1, 55.7, 49.5, 46.8, 31.8, 31.3, 25.6. IR (cm⁻¹): 3412, 3334, 3056, 3026, 2953, 2867, 1682, 1596, 1518, 1499, 1483, 1462, 1436, 1393, 1309, 1288, 1245, 1178, 1129, 1027. HRMS (ESI) *m/z* calcd for C₂₁H₂₇BrNO₂ (M+H)⁺ 404.1220, found 404.1226.

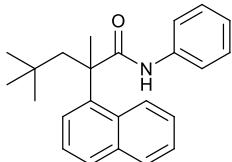
2-([1,1'-biphenyl]-4-yl)-2,4,4-trimethyl-N-phenylpentanamide (3ma)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (55%, 40.8 mg). m.p. 168-170 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.64 (d, J = 8.4 Hz, 4H), 7.56 (d, J = 8.3 Hz, 2H), 7.48 (t, J = 7.4 Hz, 2H), 7.42-7.37 (m, 3H), 7.29 (t, J = 7.6 Hz, 2H),

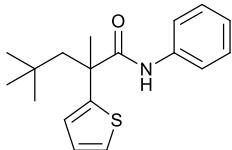
7.09 (t, $J = 7.3$ Hz, 1H), 6.94 (s, 1H), 2.41 (d, $J = 14.7$ Hz, 1H), 2.20 (d, $J = 14.7$ Hz, 1H), 1.84 (s, 3H), 0.96 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.5, 144.2, 140.3, 139.9, 138.0, 128.9, 128.8, 127.5, 127.4, 127.3, 127.0, 124.2, 119.8, 51.7, 50.6, 32.0, 31.8, 25.2. IR (cm^{-1}): 3405, 3346, 3056, 3028, 2952, 2868, 1667, 1597, 1520, 1499, 1486, 1436, 1309, 1242, 1177, 1075, 1006. HRMS (ESI) m/z calcd for $\text{C}_{26}\text{H}_{30}\text{NO} (\text{M}+\text{H})^+$ 372.2322, found 372.2324.

2,4,4-trimethyl-2-(naphthalen-1-yl)-N-phenylpentanamide (3na)



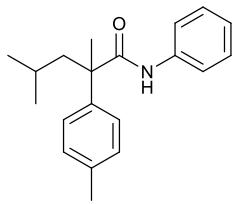
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (73%, 50.4 mg). m.p. 157-159 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 8.05-8.02 (m, 1H), 7.89-7.87 (m, 2H), 7.77 (d, $J = 7.0$ Hz, 1H), 7.54 (t, $J = 7.8$ Hz, 1H), 7.48-7.44 (m, 2H), 7.22-7.16 (m, 4H), 7.03-7.00 (m, 1H), 6.79 (s, 1H), 2.68 (d, $J = 14.9$ Hz, 1H), 2.33 (d, $J = 14.9$ Hz, 1H), 2.04 (s, 3H), 0.62 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 177.7, 137.9, 134.4, 132.4, 129.3, 128.8, 126.4, 125.9, 125.7, 125.4, 125.1, 124.2, 120.2, 51.7, 32.0, 31.1, 27.8. IR (cm^{-1}): 3405, 3330, 3051, 3030, 2952, 2866, 1682, 1596, 1518, 1500, 1483, 1396, 1309, 1241, 1204, 1103, 1029. HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{28}\text{NO} (\text{M}+\text{H})^+$ 346.2165, found 346.2166.

2,4,4-trimethyl-N-phenyl-2-(thiophen-2-yl)pentanamide (3oa)



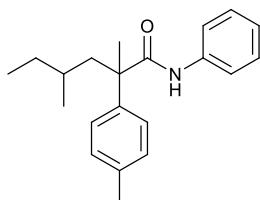
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (61%, 36.7 mg). m.p. 104-105 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.40 (d, $J = 8.3$ Hz, 2H), 7.32-7.27 (m, 4H), 7.11-7.03 (m, 3H), 2.47 (d, $J = 14.6$ Hz, 1H), 2.10 (d, $J = 14.6$ Hz, 1H), 1.88 (s, 3H), 0.98 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 173.9, 150.8, 137.8, 128.9, 127.1, 124.9, 124.8, 124.3, 119.8, 52.2, 50.5, 32.2, 31.4, 25.9. IR (cm^{-1}): 3391, 3058, 3023, 2952, 2869, 1667, 1597, 1521, 1499, 1480, 1437, 1366, 1309, 1241, 1177, 1057. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{24}\text{NOS} (\text{M}+\text{H})^+$ 302.1573, found 302.1575.

2,4-dimethyl-N-phenyl-2-(*p*-tolyl)pentanamide (3ab)



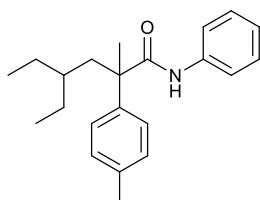
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (61%, 36.0 mg). m.p. 109-110 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.39-7.34 (m, 4H), 7.29-7.25 (m, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.87 (s, 1H), 2.38 (s, 3H), 2.13-2.08 (q, 1H), 2.04-1.99 (q, 1H), 1.67 (s, 3H), 0.94 (d, *J* = 6.6 Hz, 3H), 0.71 (d, *J* = 6.7 Hz, 3H). ¹³C NMR (CDCl₃, 100 MHz): δ 175.7, 141.0, 138.0, 136.9, 129.6, 128.9, 126.9, 124.0, 119.7, 51.3, 47.2, 24.8, 24.7, 24.6, 24.3, 21.0. IR (cm⁻¹): 3296, 3055, 3024, 2952, 2925, 2866, 1658, 1596, 1524, 1498, 1464, 1437, 1313, 1248, 1144, 1021. HRMS (ESI) *m/z* calcd for C₂₀H₂₆NO (M+H)⁺ 296.2009, found 296.2007.

2,4-dimethyl-N-phenyl-2-(p-tolyl)hexanamide (3ac)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (55%, 34.0 mg). m.p. 98-100 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.39-7.25 (m, 6H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.88 (s, 1H), 2.39 (s, 3H), 2.23-2.18 (q, 0.52H), 2.13-2.08 (q, 0.57H), 2.03-1.98 (q, 0.57H), 1.95-1.89 (q, 0.52H), 1.66-1.65 (d, 3H), 1.43-1.36 (m, 1.51H), 1.29-1.06 (m, 2.57H), 0.92 (d, *J* = 6.7 Hz, 1.6H), 0.86 (t, *J* = 7.4 Hz, 1.49H), 0.79 (t, *J* = 7.4vHz, 1.6H), 0.67 (d, *J* = 6.5 Hz, 1.48H). ¹³C NMR (CDCl₃, 100 MHz): δ 175.8, 175.7, 141.2, 140.9, 138.1, 138.0, 137.0, 136.9, 129.5, 128.9, 127.1, 126.9, 124.0, 119.7, 51.4, 51.3, 45.4, 45.3, 31.5, 31.4, 30.8, 30.7, 24.5, 24.3, 21.2, 21.1, 21.0, 11.4, 11.3. IR (cm⁻¹): 3406, 3337, 3056, 3022, 2960, 2925, 2872, 1666, 1597, 1517, 1499, 1460, 1437, 1379, 1310, 1241, 1116, 1019. HRMS (ESI) *m/z* calcd for C₂₁H₂₈NO (M+H)⁺ 310.2165, found 310.2167.

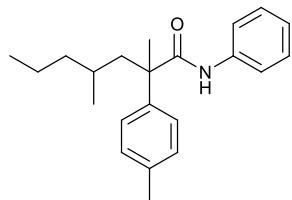
4-ethyl-2-methyl-N-phenyl-2-(p-tolyl)hexanamide (3ad)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (48%, 31.0 mg). m.p. 111-113 °C. ¹H NMR (CDCl₃, 400 MHz): δ 7.37 (d, *J* = 7.7 Hz,

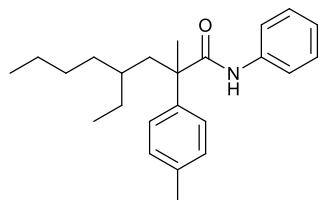
2H), 7.33-7.25 (m, 4H), 7.20 (d, $J = 8.1$ Hz, 2H), 7.07 (t, $J = 7.3$ Hz, 1H), 6.84 (s, 1H), 2.38 (s, 3H), 2.11-2.06 (q, 1H), 2.01-1.96 (q, 1H), 1.64 (s, 3H), 1.37-1.24 (m, 3H), 1.15-1.08 (m, 2H), 0.83(t, $J = 7.1$ Hz, 3H), 0.71 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.8, 141.1, 138.0, 136.9, 129.5, 128.9, 127.0, 124.0, 119.6, 51.4, 42.0, 36.1, 26.8, 26.7, 24.3, 21.0, 10.7, 10.5. IR (cm^{-1}): 3408, 3331, 3020, 2960, 2925, 2873, 1655, 1596, 1515, 1499, 1458, 1436, 1379, 1310, 1241, 1114, 1074, 1019. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{30}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 324.2322, found 324.2324.

2,4-dimethyl-N-phenyl-2-(p-tolyl)heptanamide (3ae)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a yellow solid (60%, 38.7 mg). m.p. 80-82 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.39 (m, 2H), 7.33-7.25 (m, 4H), 7.21 (d, $J = 8.3$ Hz, 2H), 7.09-7.04 (m, 1H), 6.86 (d, $J = 4.0$ Hz, 1H), 2.38 (s, 3H), 2.21-2.07 (m, 1H), 1.99-1.89 (m, 1H), 1.65 (d, $J = 2.1$ Hz, 4H), 1.51-1.43 (m, 1H), 1.35-1.06 (m, 4H), 0.91 (d, $J = 6.6$ Hz, 1.72H), 0.84 (t, $J = 6.9$ Hz, 1.35H), 0.78 (t, $J = 6.8$ Hz, 1.66H), 0.91 (d, $J = 6.6$ Hz, 1.4H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.8, 175.7, 141.2, 140.9, 138.1, 138.0, 136.97, 136.93, 129.5, 128.9, 128.8, 127.1, 126.9, 124.0, 119.7, 51.5, 51.3, 45.8, 45.7, 41.4, 41.3, 28.9, 28.8, 24.5, 24.3, 21.7, 21.6, 21.04, 21.02, 20.1, 19.9, 14.3, 14.2. IR (cm^{-1}): 3406, 3335, 3022, 2956, 2927, 2870, 1667, 1597, 1515, 1499, 1436, 1378, 1310, 1242, 1192, 1136, 1075, 1019. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{30}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 324.2322, found 324.2323.

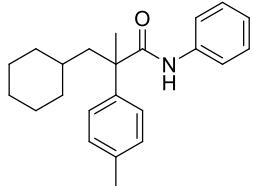
4-ethyl-2-methyl-N-phenyl-2-(p-tolyl)octanamide (3af)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow liquid (51%, 35.8 mg). ^1H NMR (CDCl_3 , 400 MHz): δ 7.36 (d, $J = 7.9$ Hz, 2H), 7.32-7.25 (m, 4H), 7.20 (d, $J = 8.0$ Hz, 2H), 7.08-7.04 (m, 1H), 6.84 (d, $J = 4.0$ Hz, 1H), 2.38 (s, 3H), 2.13-1.94 (m, 2H), 1.63 (s, 3H), 1.34-1.21 (m, 5H), 1.12-1.04 (m, 4H), 0.85-0.77 (m, 4.49H), 0.71 (t, $J = 7.3$ Hz, 1.54H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.9, 175.8, 141.1, 141.0, 138.1, 136.98, 136.96, 129.5, 129.5, 129.4, 128.9, 128.8, 127.1, 127.0, 123.99, 123.98, 119.6, 119.5, 51.5, 51.4, 42.4, 34.7, 34.6, 34.3, 34.1, 28.7, 28.4, 27.4, 27.2, 24.4, 24.2, 23.1, 22.9, 21.02, 21.00, 14.1, 14.0,

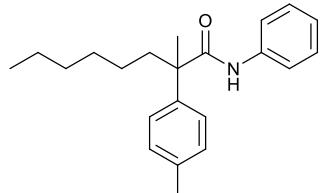
10.7, 10.4. IR (cm^{-1}): 3408, 3340, 3025, 2957, 2927, 2871, 2857, 1666, 1597, 1517, 1499, 1459, 1437, 1378, 1310, 1241, 1116, 1075, 1019. HRMS (ESI) m/z calcd for $\text{C}_{24}\text{H}_{34}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 352.2635, found 352.2638.

3-cyclohexyl-2-methyl-N-phenyl-2-(p-tolyl)propanamide (3ag)²



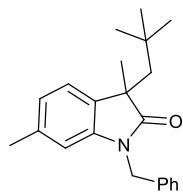
Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a light yellow solid (64%, 43.9 mg). m.p. 240-242 °C. ^1H NMR (CDCl_3 , 400 MHz): δ 7.38-7.31 (m, 4H), 7.30-7.25 (m, 2H), 7.21 (d, $J = 8.1$ Hz, 2H), 7.07 (t, $J = 7.4$ Hz, 1H), 6.87 (s, 1H), 2.38 (s, 3H), 2.09-2.05 (q, 1H), 2.00-1.95 (q, 1H), 1.72-1.69 (m, 1H), 1.65 (s, 3H), 1.59-1.56 (m, 2H), 1.45-0.86 (m, 8H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.7, 141.2, 138.0, 136.9, 129.5, 128.9, 126.9, 124.0, 119.7, 51.2, 45.9, 35.3, 35.2, 34.1, 26.5, 26.4, 26.2, 24.4, 21.0.

2-methyl-N-phenyl-2-(p-tolyl)octanamide (3ah)



Flash column chromatography on silica gel (petroleum ether/ethyl acetate 20/1) gave a yellow liquid (14%, 9 mg). ^1H NMR (CDCl_3 , 400 MHz): δ 7.39-7.36 (m, 2H), 7.31-7.25 (m, 4H), 7.21 (d, $J = 8.0$ Hz, 2H), 7.07 (t, $J = 7.4$ Hz, 1H), 6.83 (s, 1H), 2.38 (s, 3H), 2.11-2.01 (m, 1H), 1.62 (s, 3H), 1.30-1.19 (m, 8H), 0.86 (t, $J = 6.8$ Hz, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 175.6, 140.6, 138.0, 136.9, 129.6, 128.9, 126.9, 124.0, 119.7, 51.2, 38.9, 31.7, 29.9, 24.4, 24.0, 22.7, 21.0, 14.1. IR (cm^{-1}): 3411, 3346, 3020, 2953, 2922, 2852, 1658, 1593, 1497, 1461, 1440, 1372, 1305, 1120, 1069, 1011. HRMS (ESI) m/z calcd for $\text{C}_{22}\text{H}_{30}\text{NO}$ ($\text{M}+\text{H}$) $^+$ 324.2322, found 324.2324.

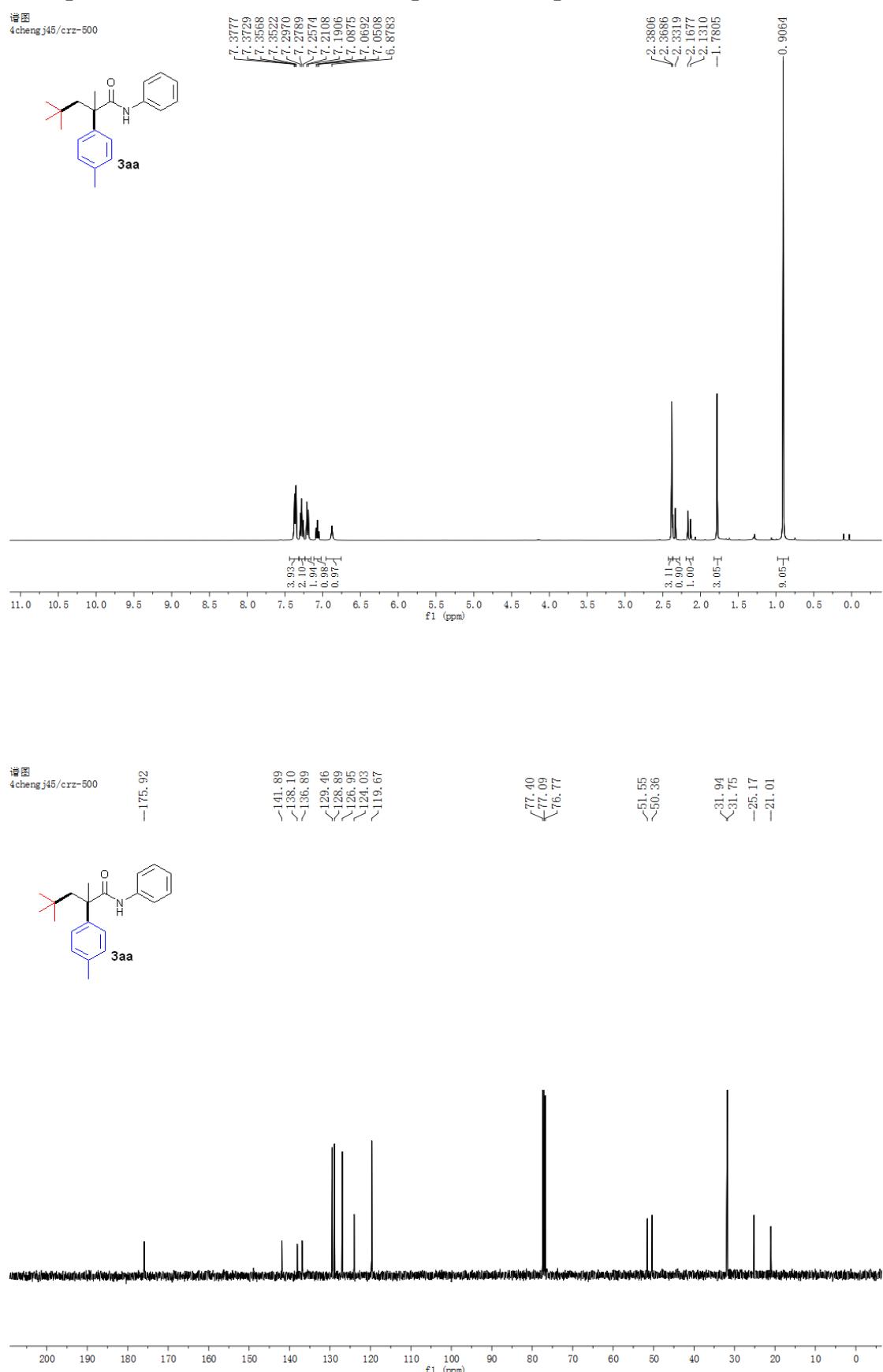
1-benzyl-3,6-dimethyl-3-neopentylindolin-2-one (4)



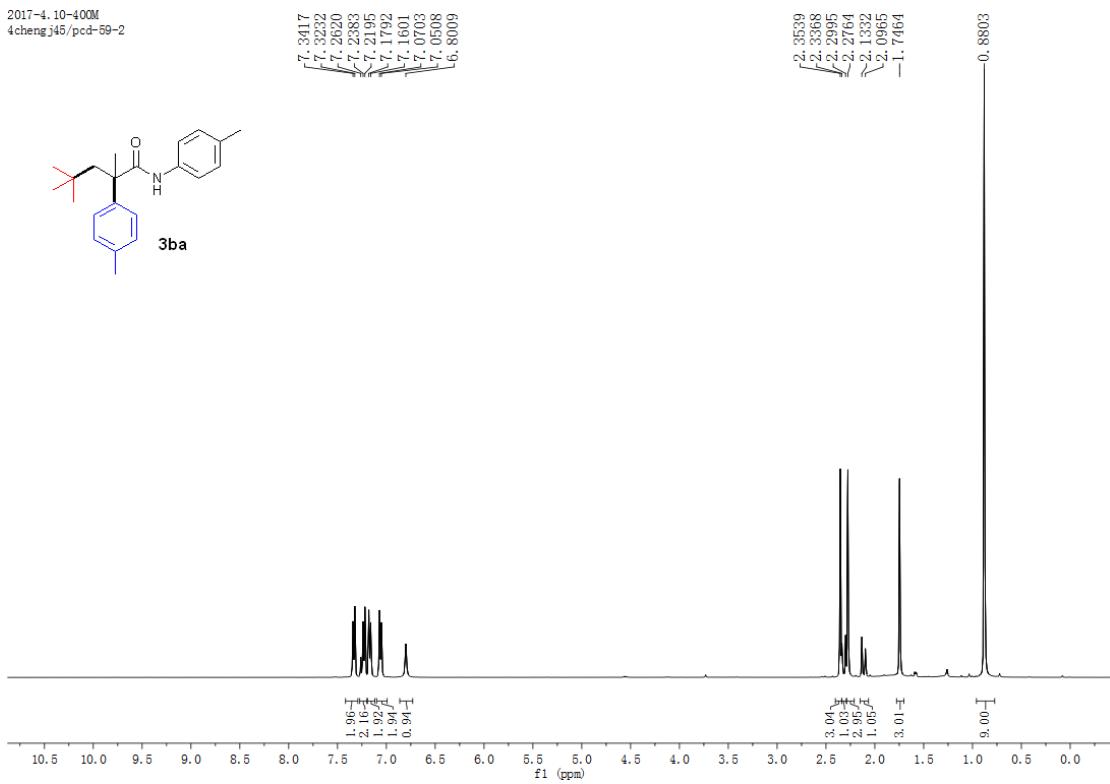
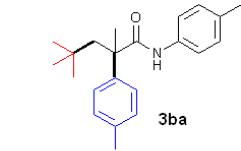
² H. Zhang, C. Pan, N. Jin, Z. Gu, H. Hu and C. Zhu, *Chem. Commun.*, 2015, **51**, 1320.

Flash column chromatography on silica gel (petroleum ether/ethyl acetate 10/1) gave a yellow liquid (51%, 32.7 mg). ¹H NMR (CDCl₃, 400 MHz): δ 7.32-7.22 (m, 5H), 7.08 (d, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.5 Hz, 1H), 6.60 (s, 1H), 5.03 (t, *J* = 15.5 Hz, 1H), 4.77 (t, *J* = 15.5 Hz, 1H), 2.30 (s, 3H), 2.19 (t, *J* = 14.4 Hz, 1H), 1.87 (t, *J* = 14.4 Hz, 1H), 1.32 (s, 3H), 0.64 (s, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 181.5, 142.1, 137.4, 136.3, 131.2, 128.7, 127.6, 127.5, 123.8, 122.6, 109.9, 50.5, 47.3, 43.8, 31.9, 30.9, 29.2, 21.8. IR (cm⁻¹): 3011, 2950, 2852, 1685, 1602, 1517, 1491, 1462, 1352, 1235, 1016. HRMS (ESI) *m/z* calcd for C₂₂H₂₈NO (M+H)⁺ 322.2165, found 322.2169.

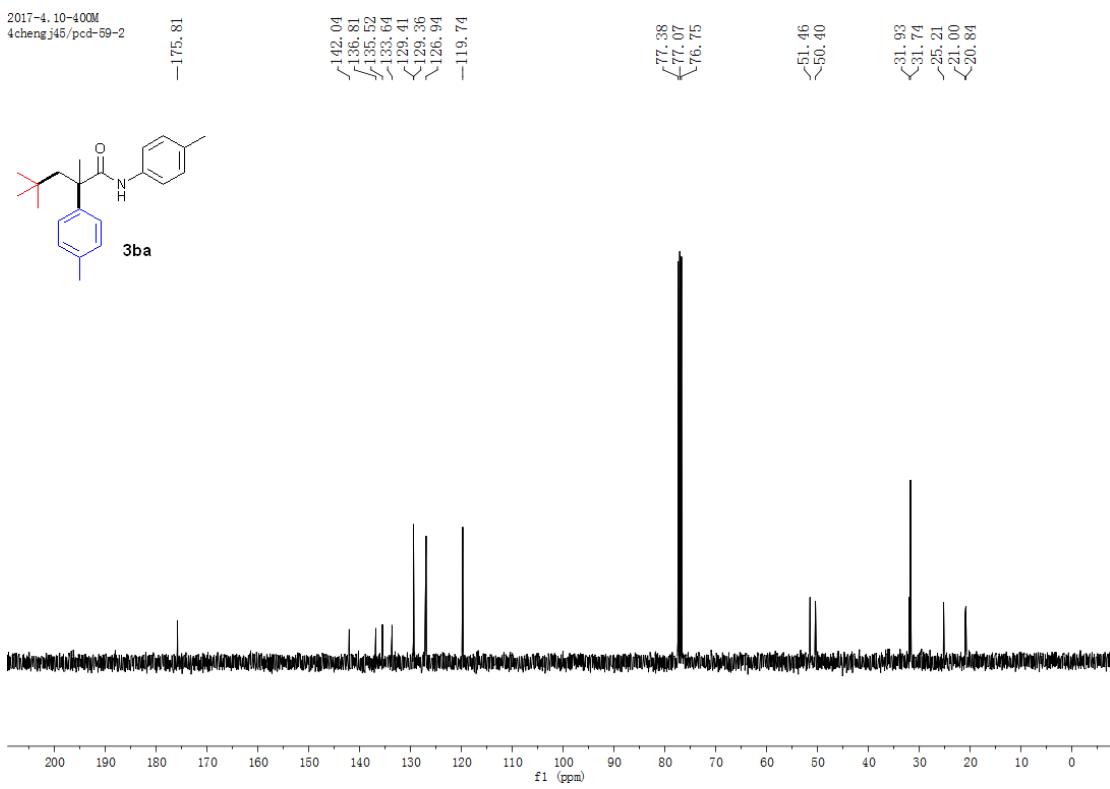
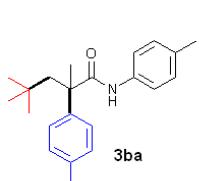
3. Copies of ^1H NMR and ^{13}C NMR spectra of the products

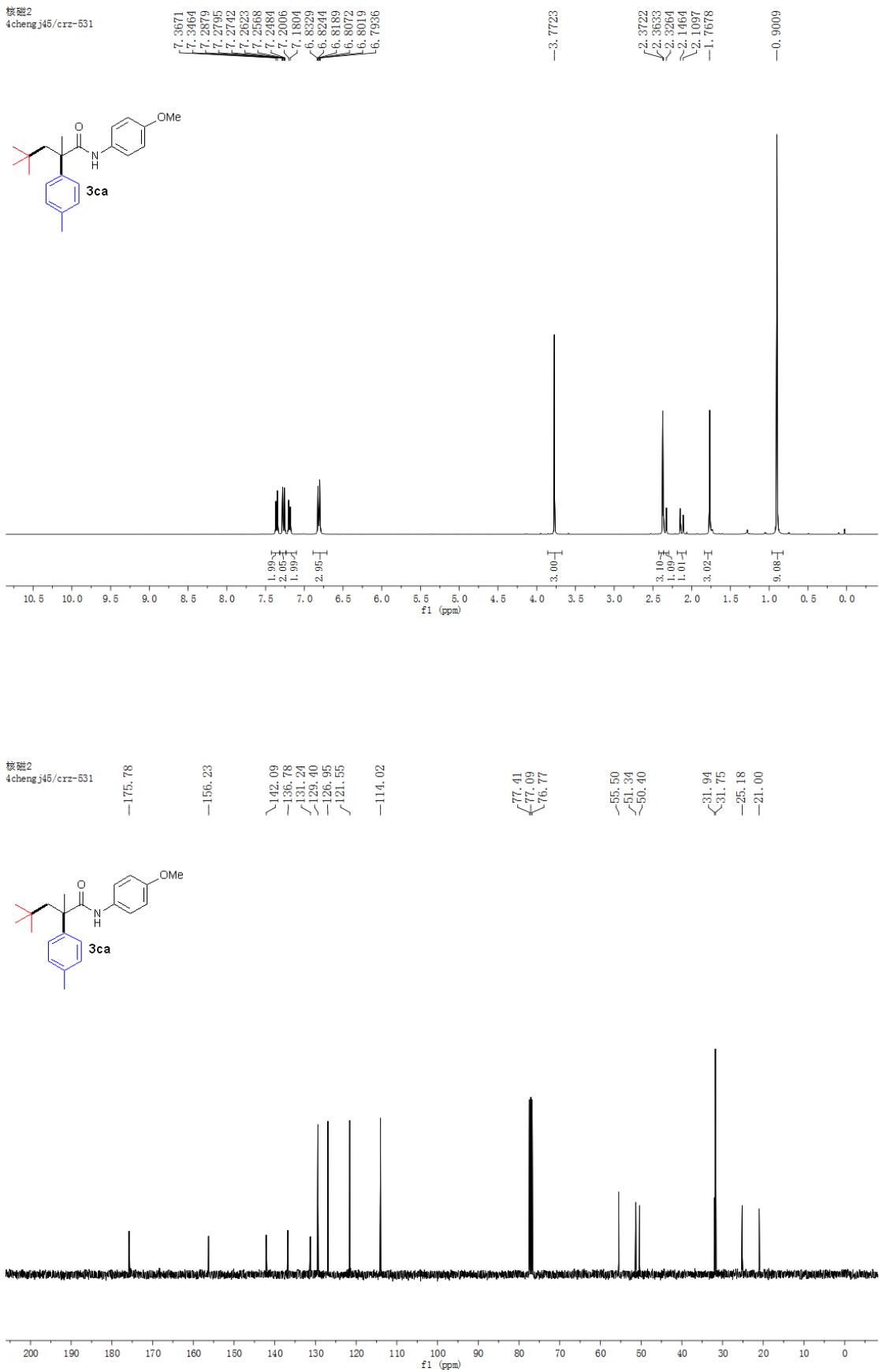


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4chengj45\pcd-59-2

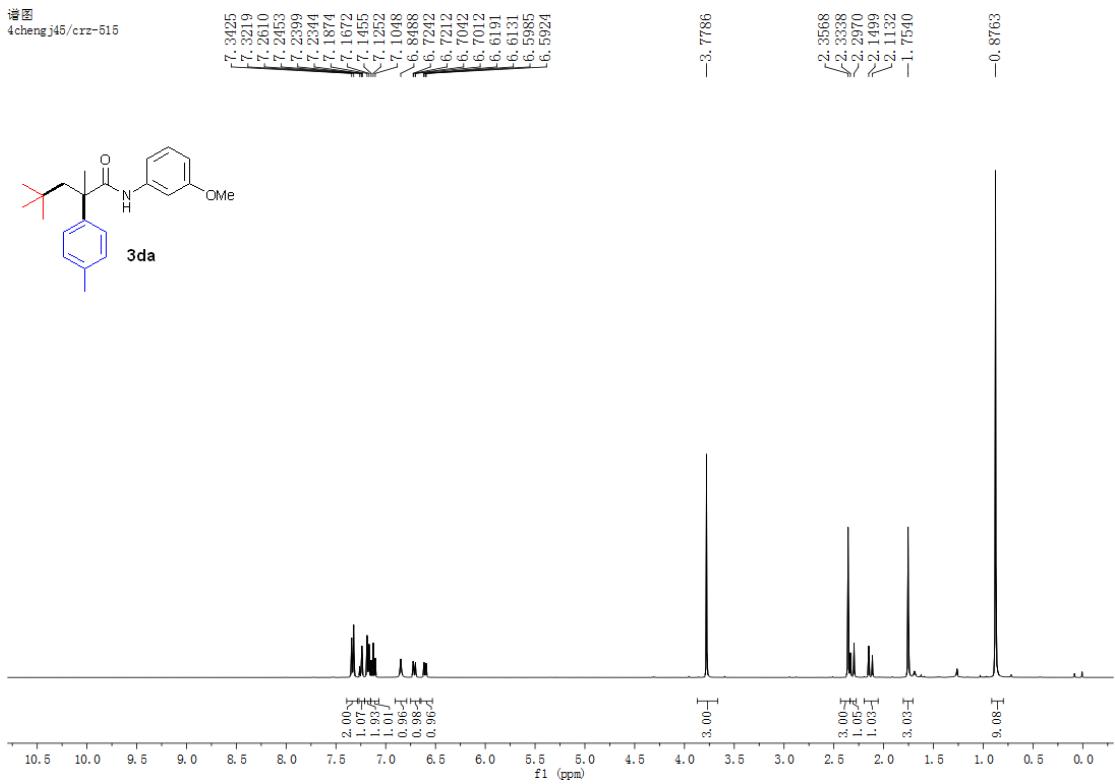


2017-4.10-400M
4chengj45\pcd-59-2

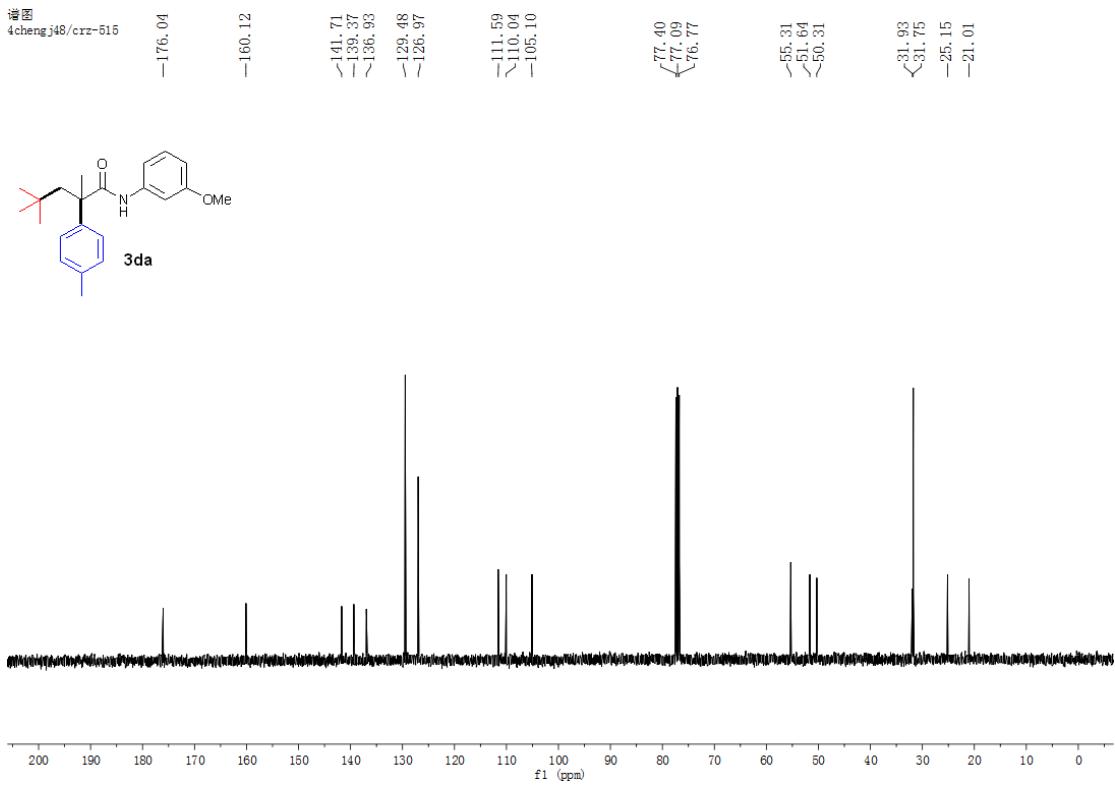




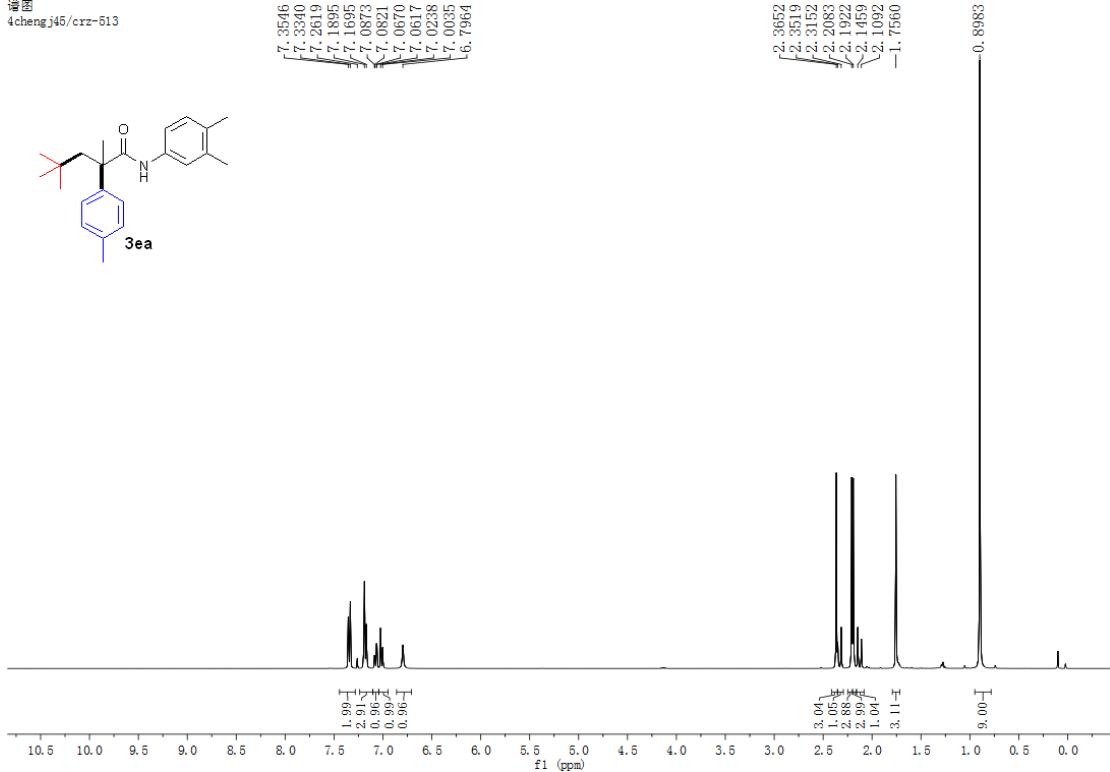
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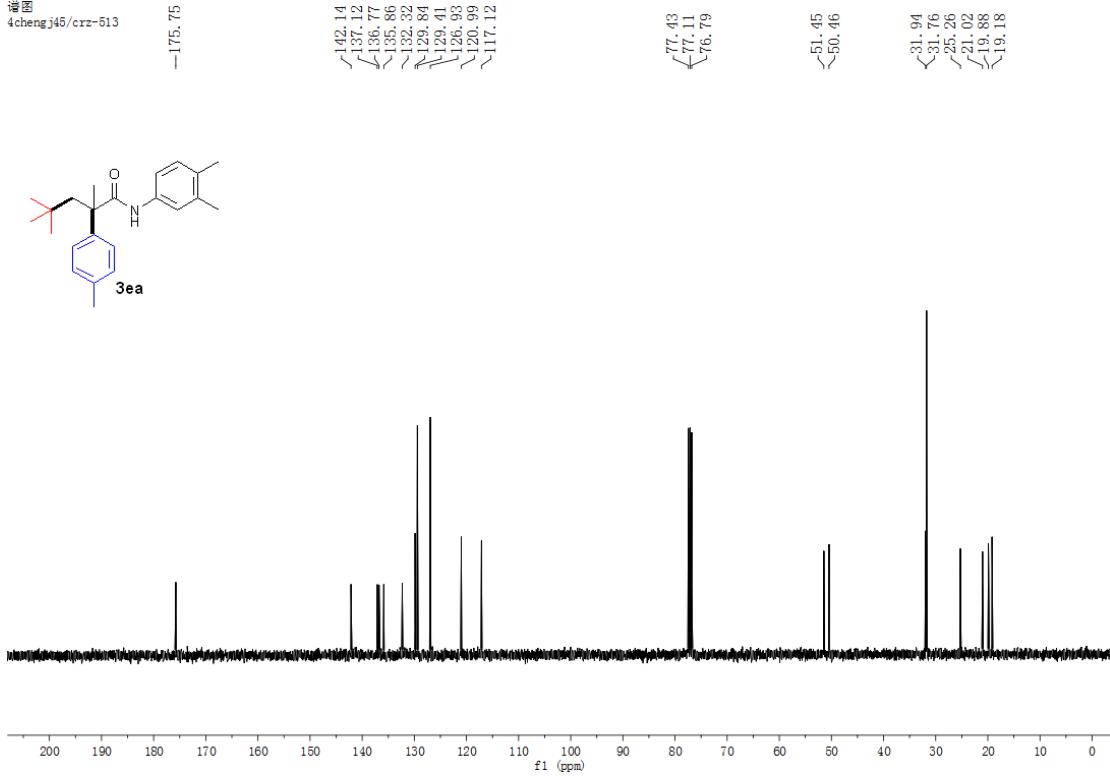
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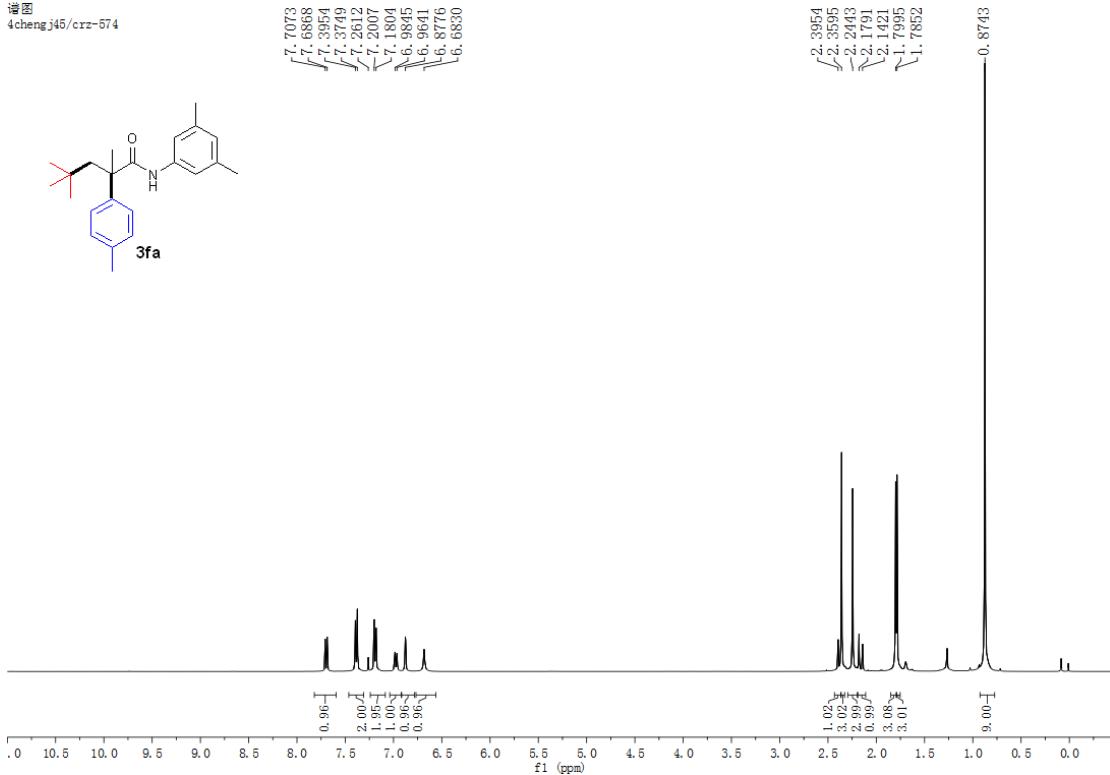
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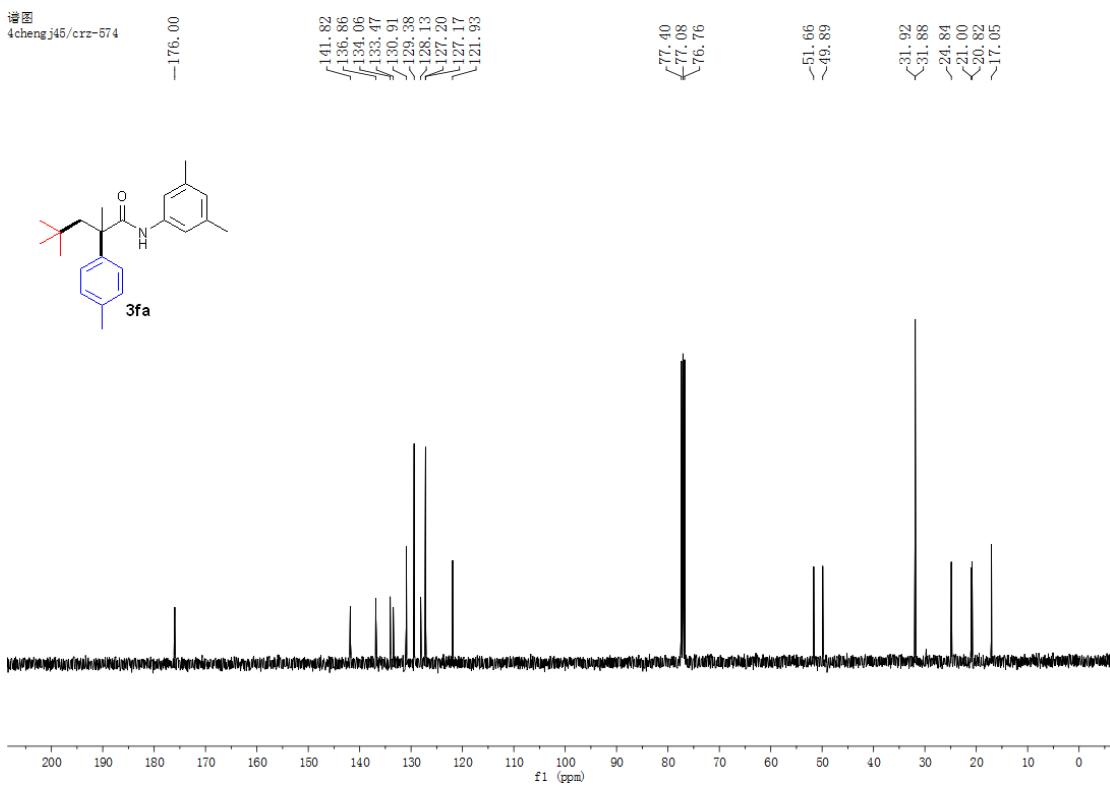
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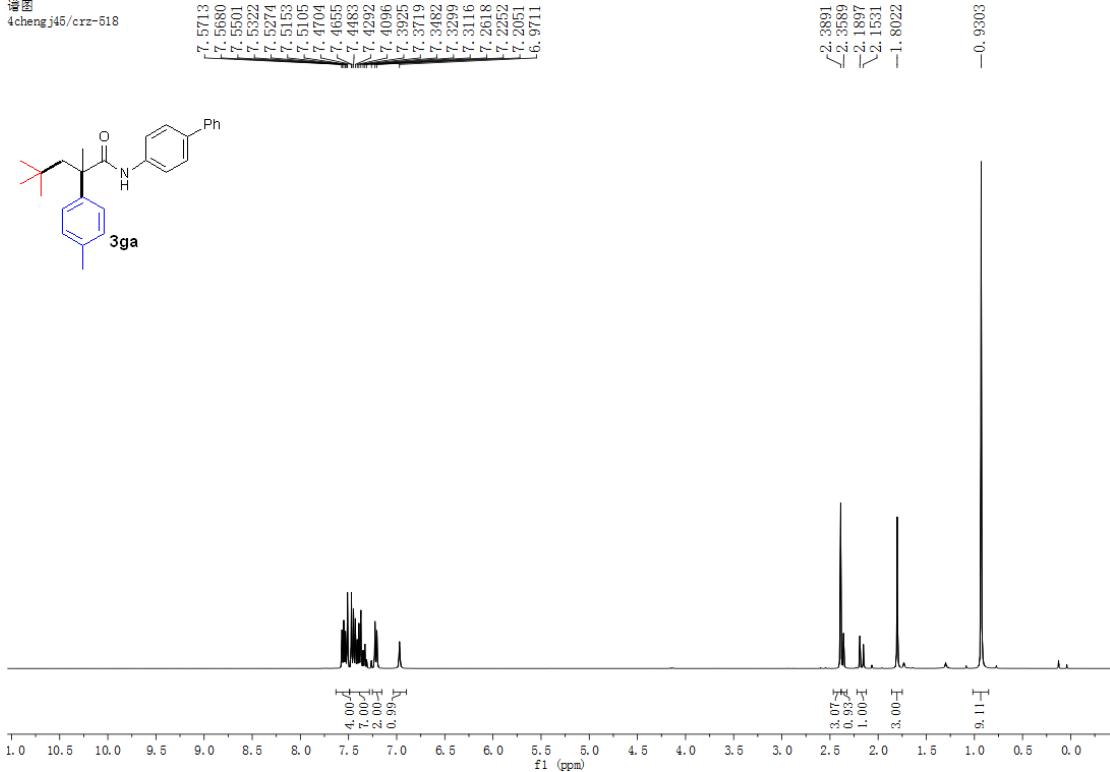
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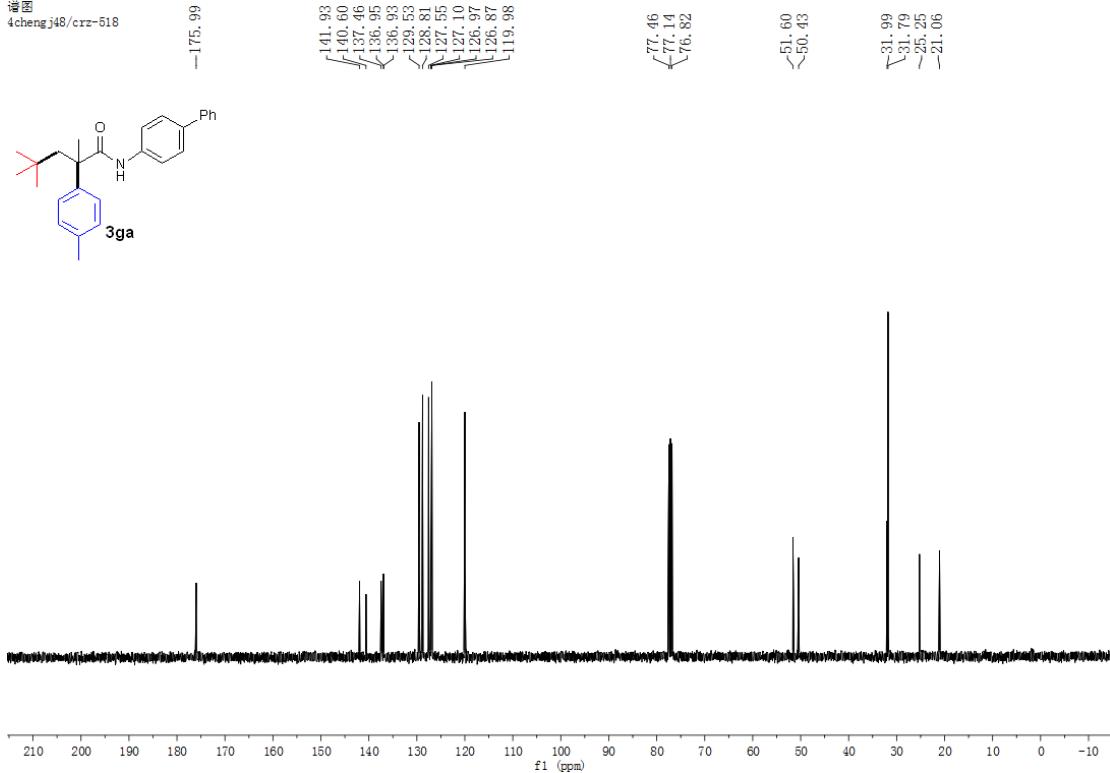
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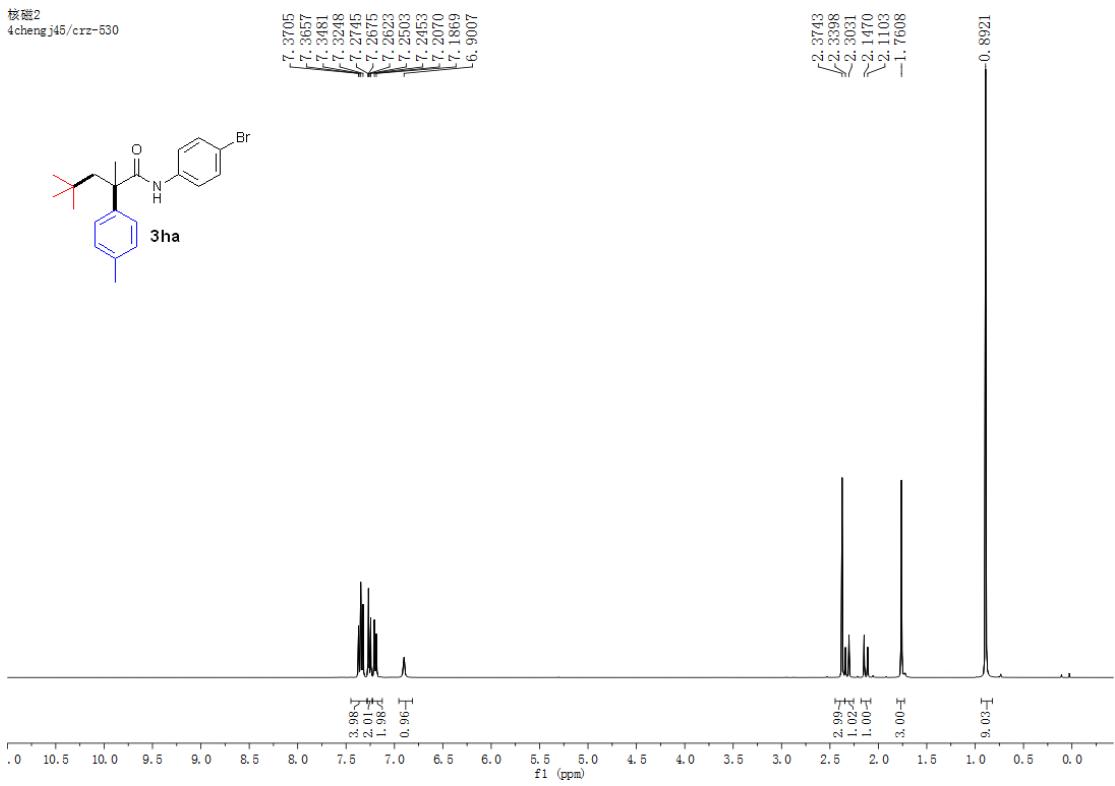
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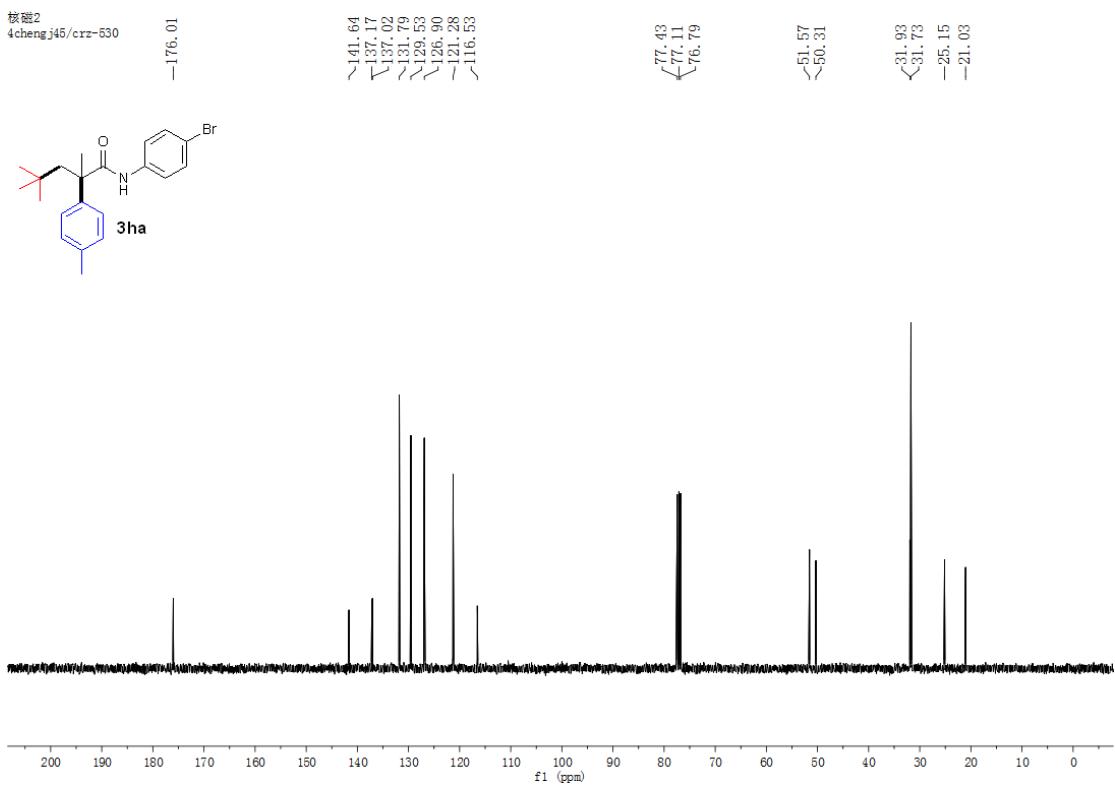
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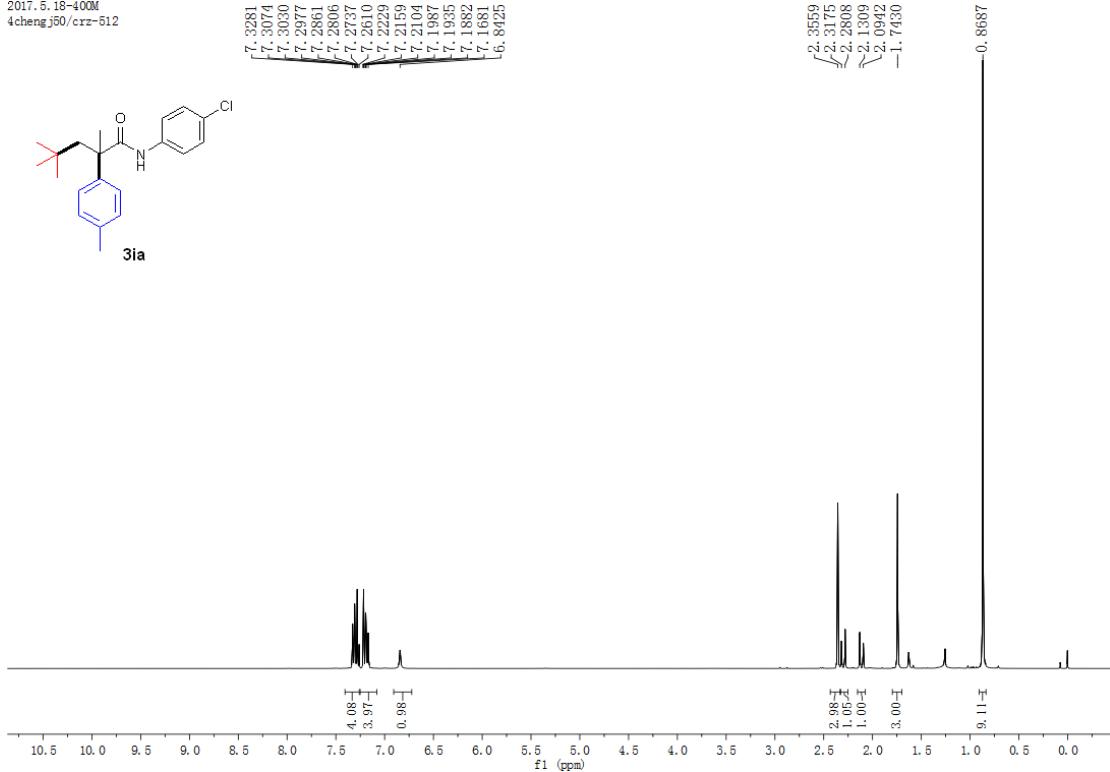
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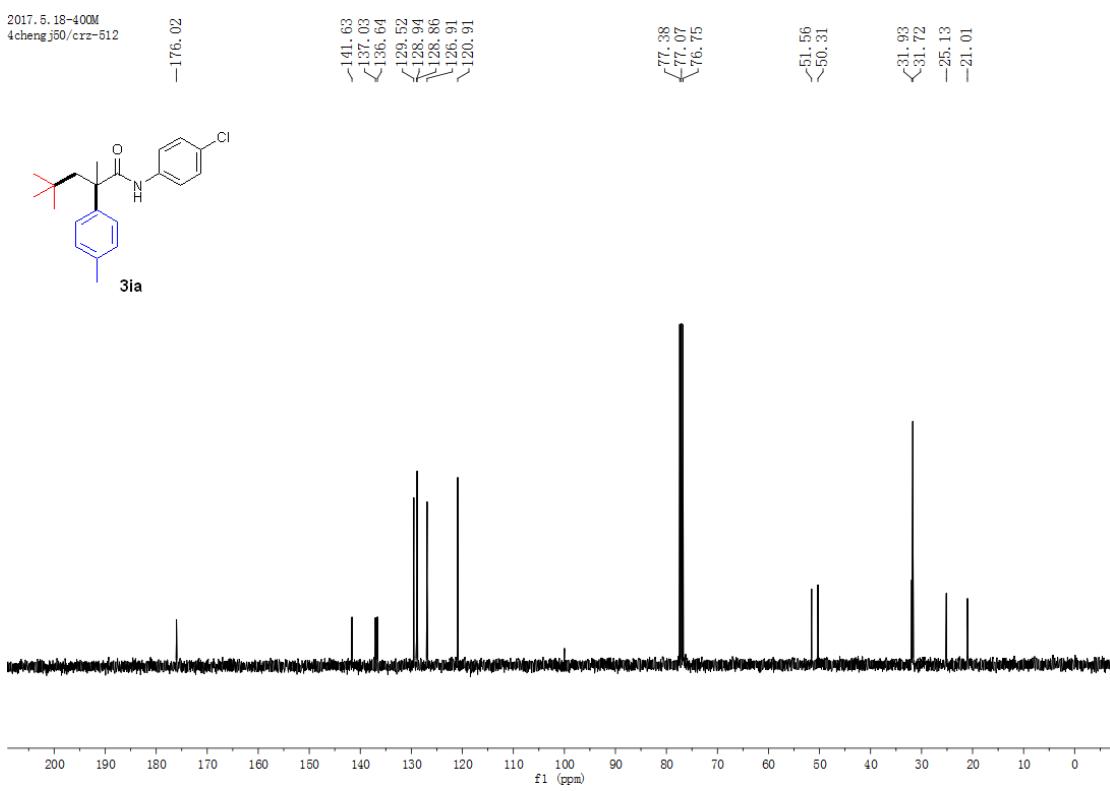
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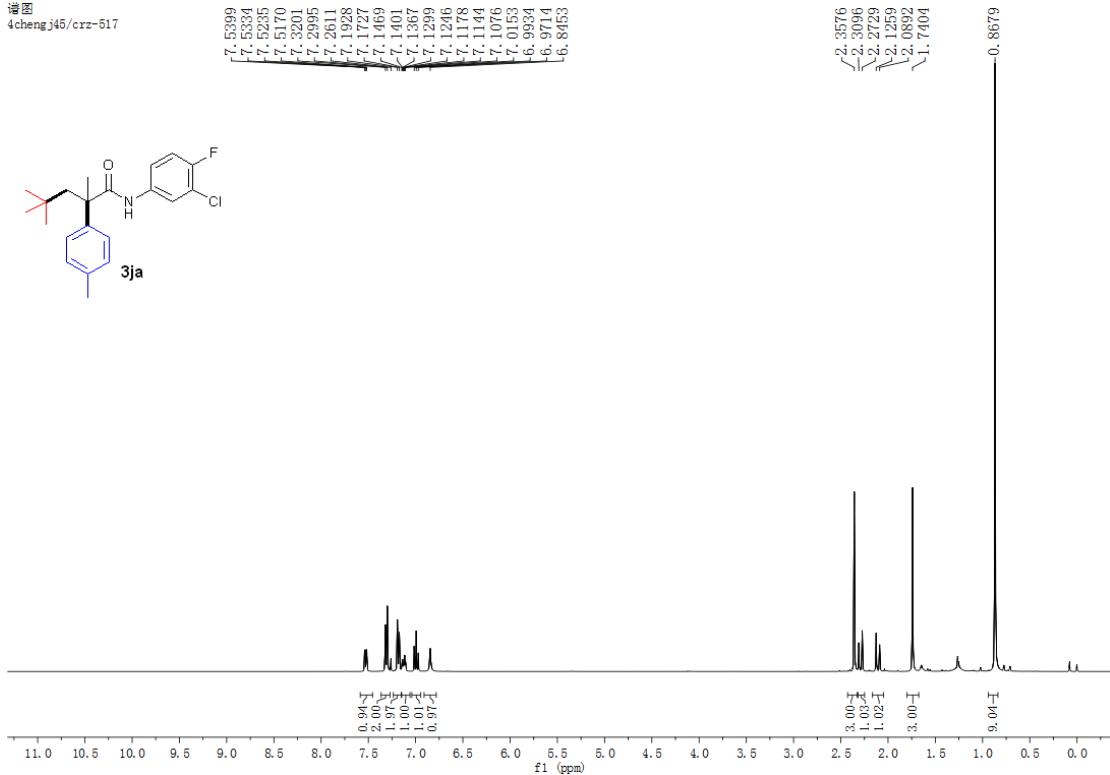
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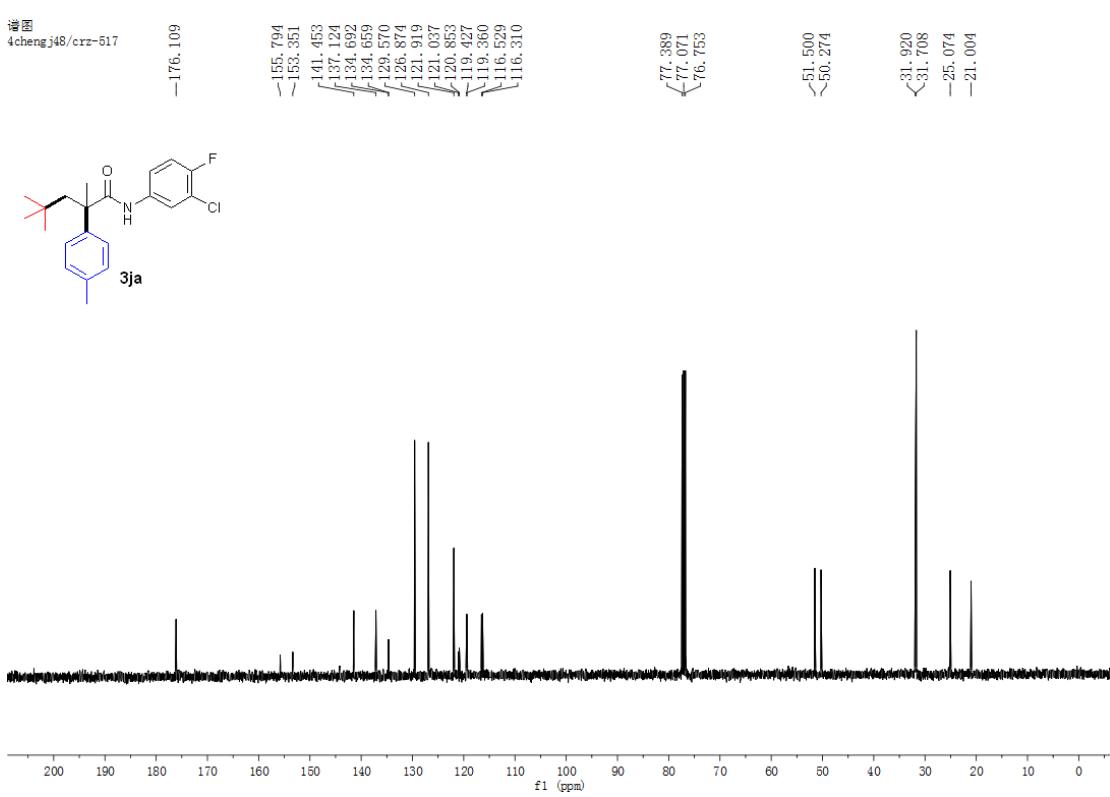
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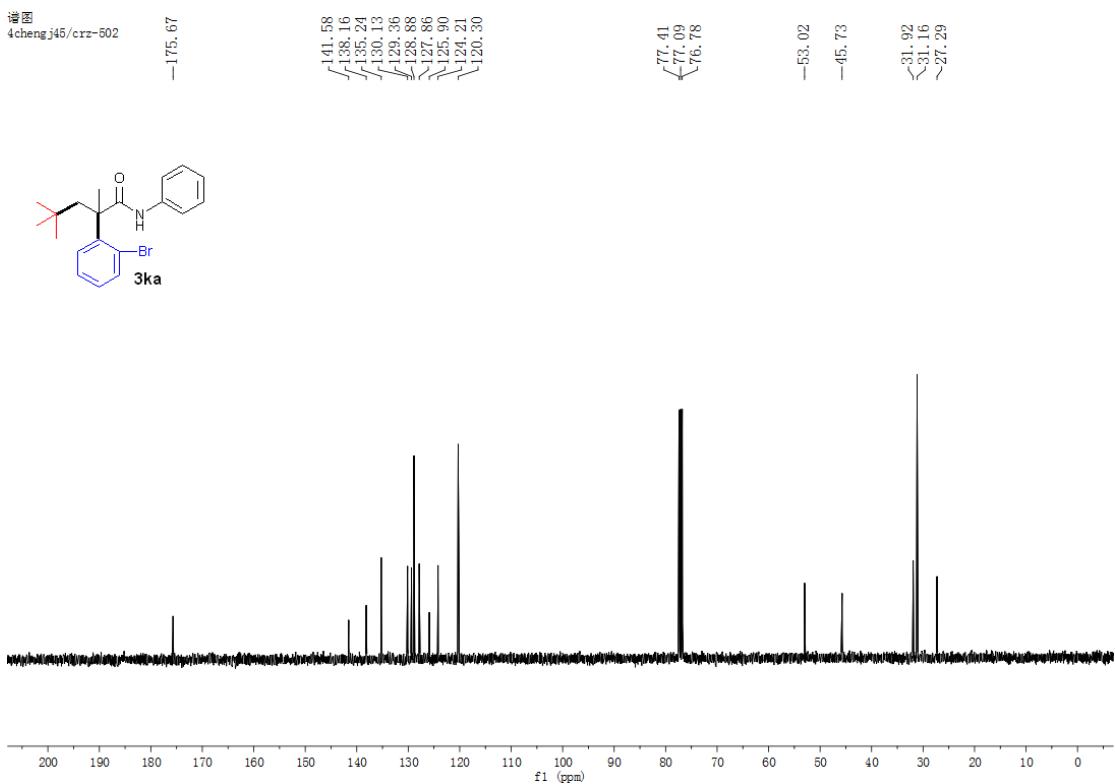
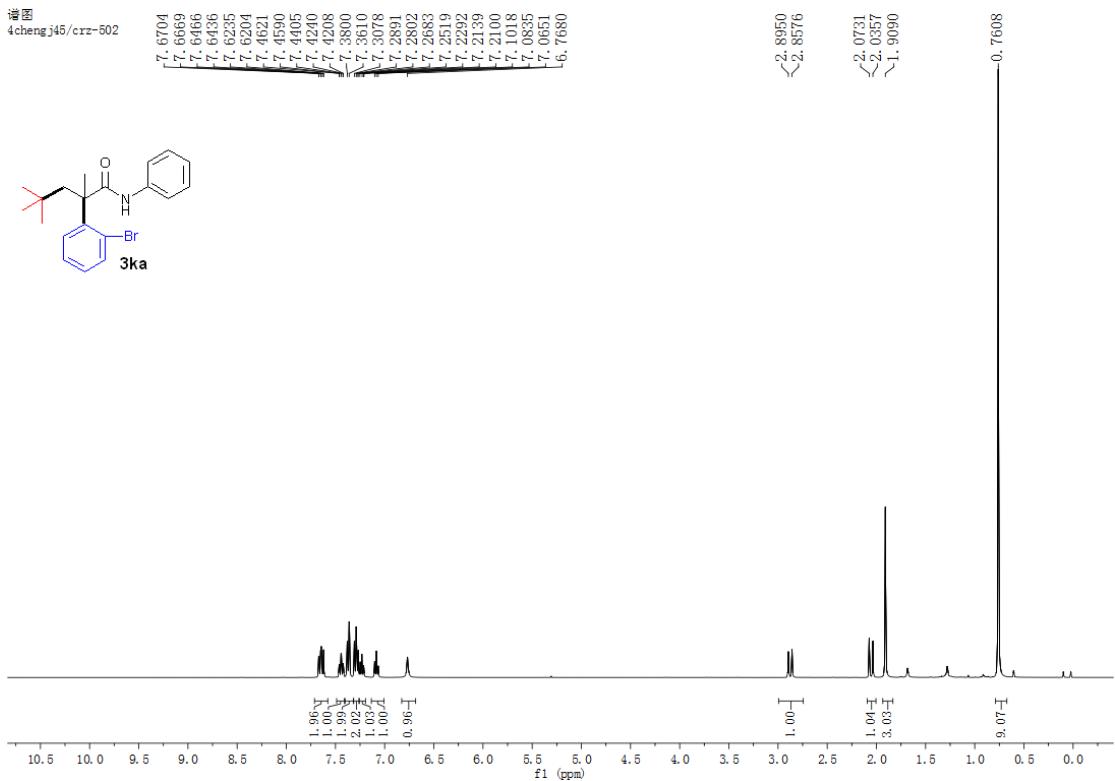


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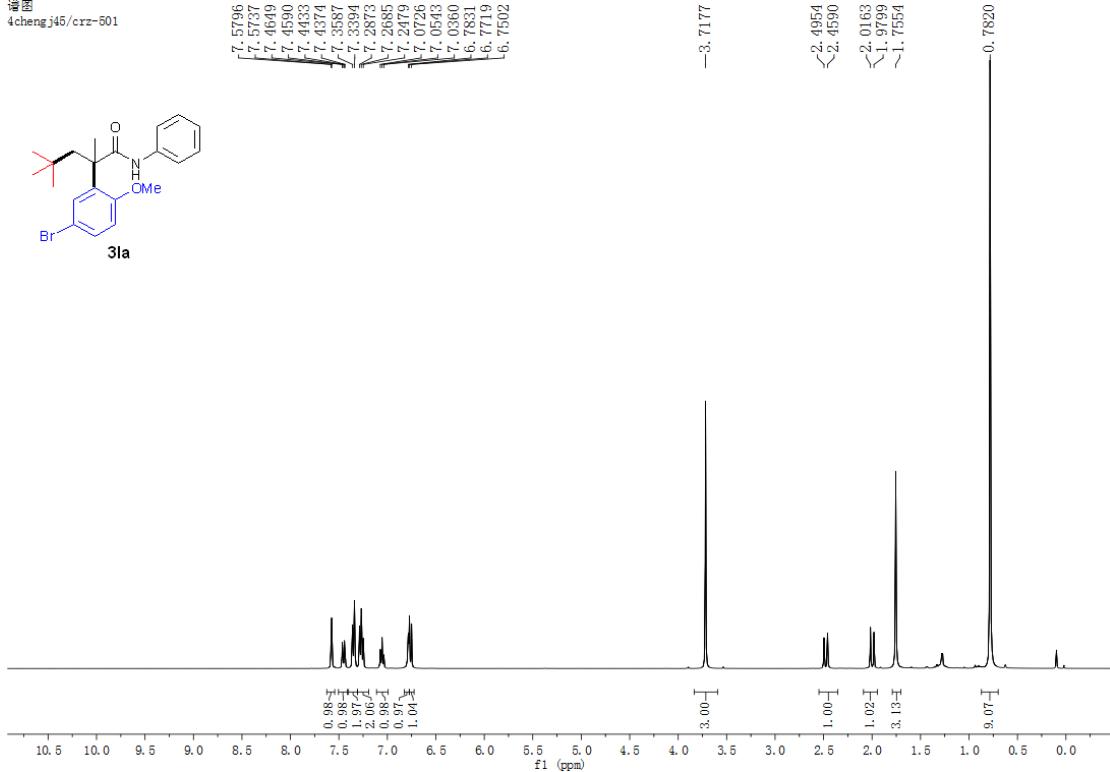


谱图
4chengj48/crz-517

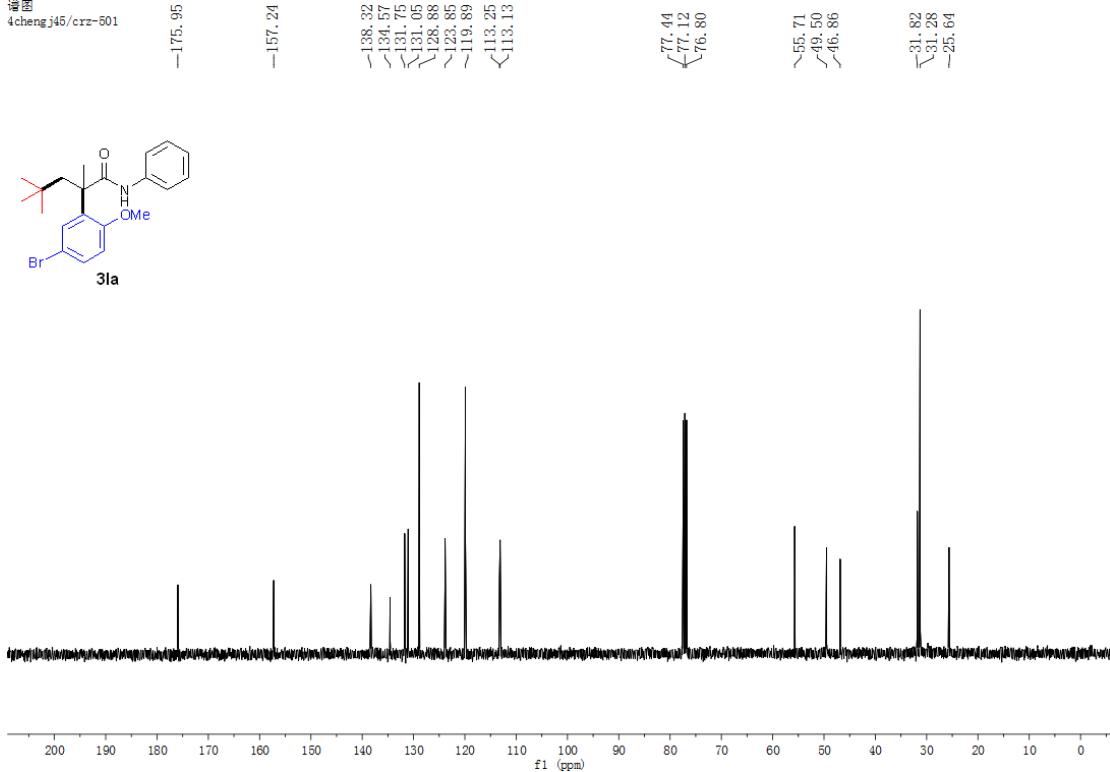


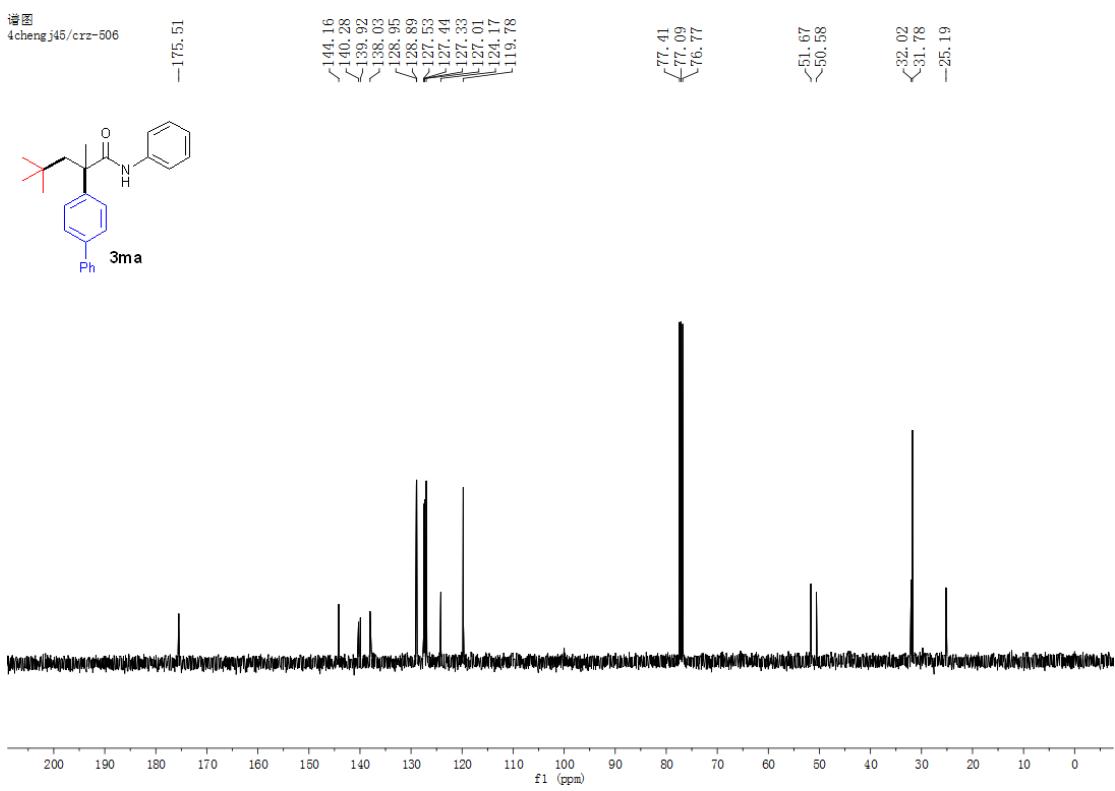
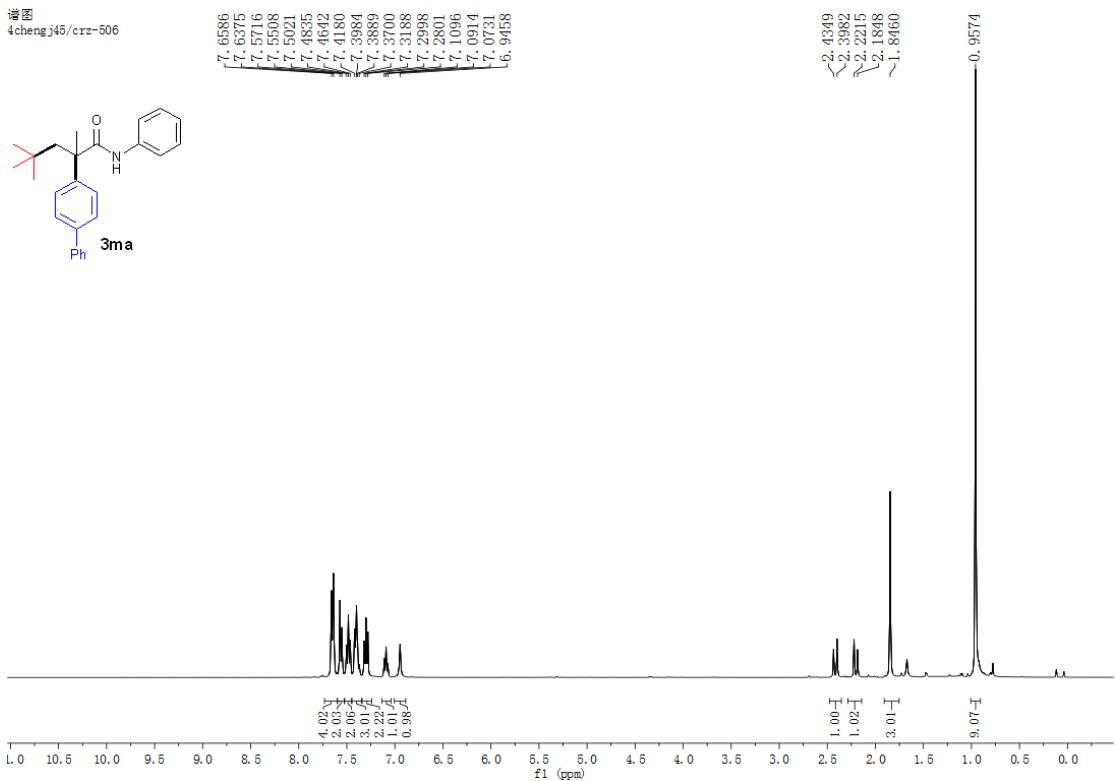


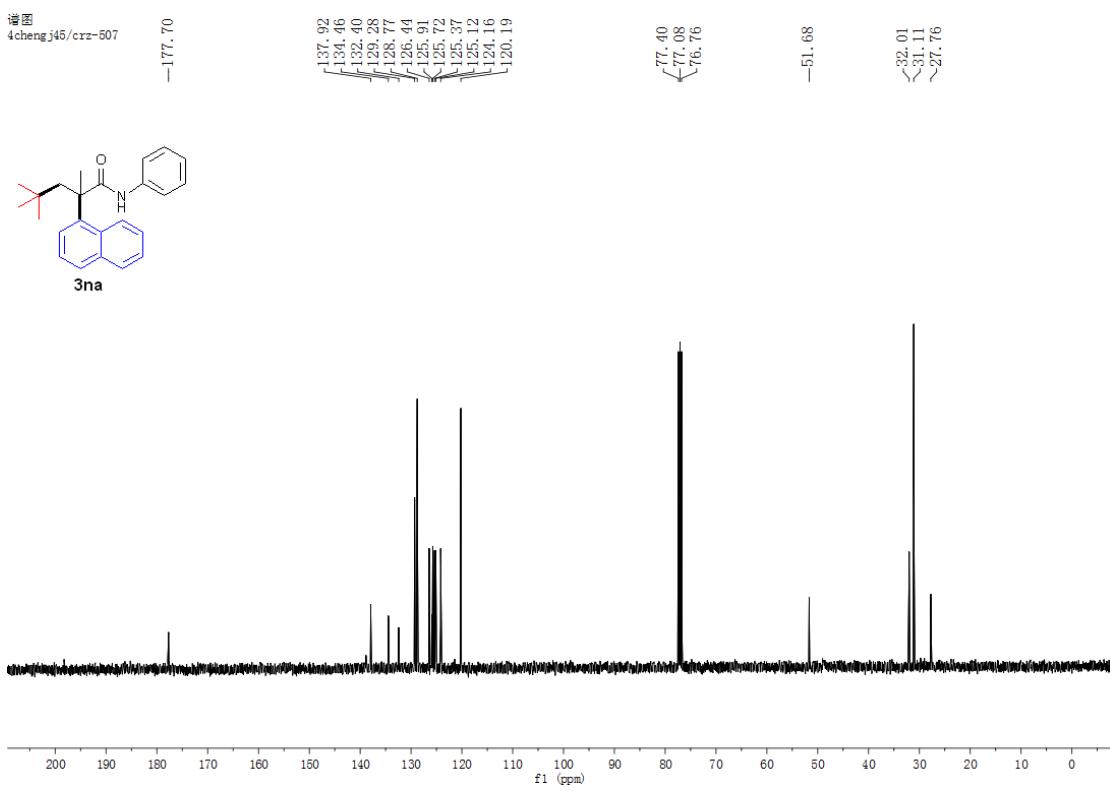
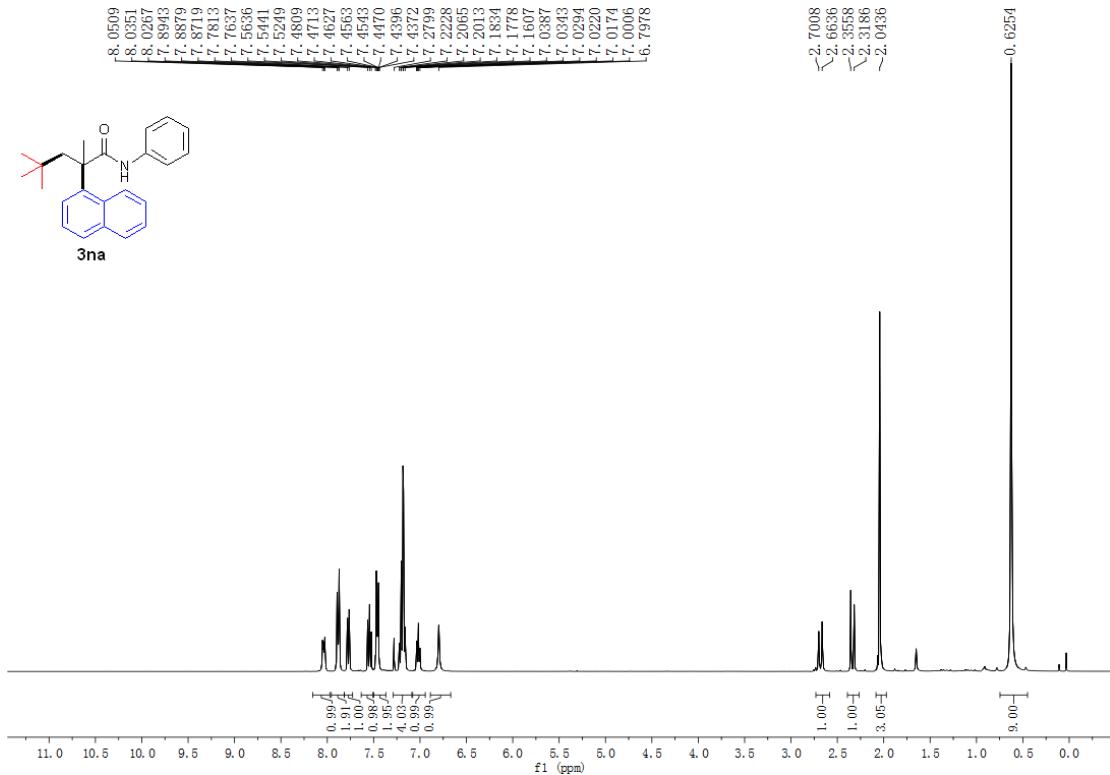
谱图
4chengj45/crz-501



谱图
4chengj45/crz-501

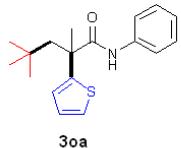




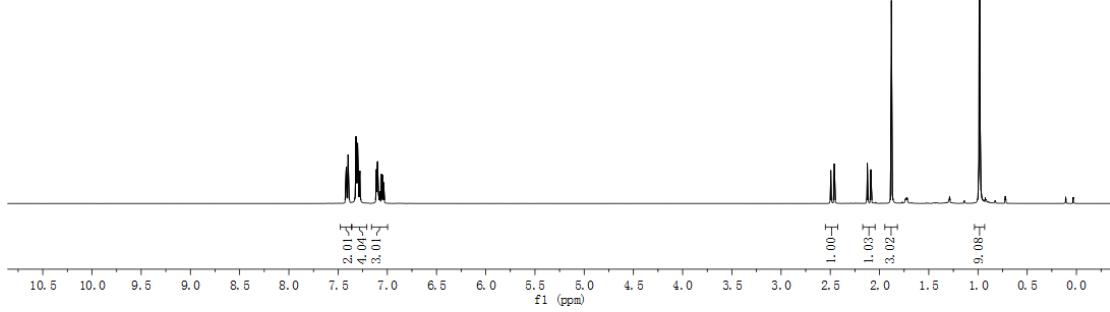


谱图
4chengj45/crz-508

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7.3560
7.3176
7.3145
7.3044
7.3011
7.3004
7.2787
7.1103
7.1076
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7.0842
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7.0580
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7.0562
7.0362

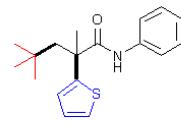


3oa

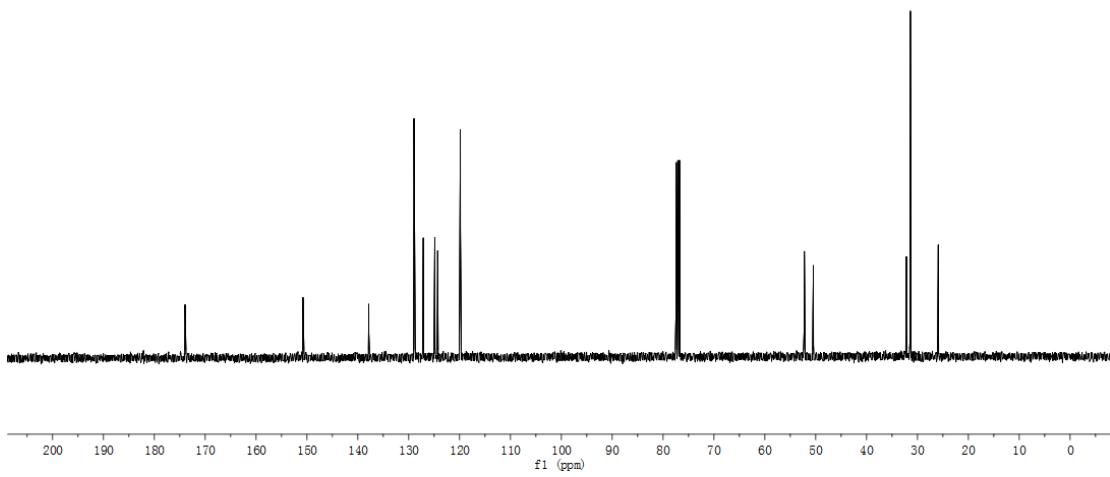


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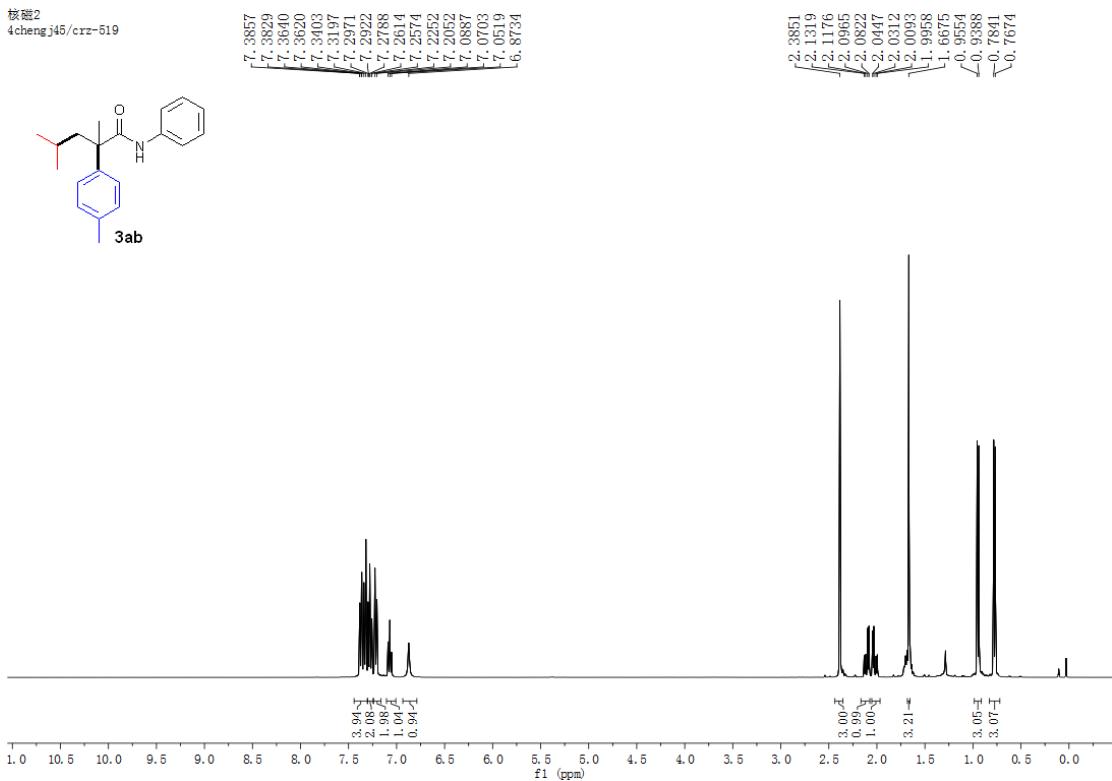
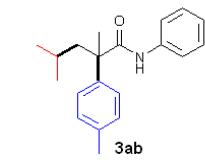
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-119.85



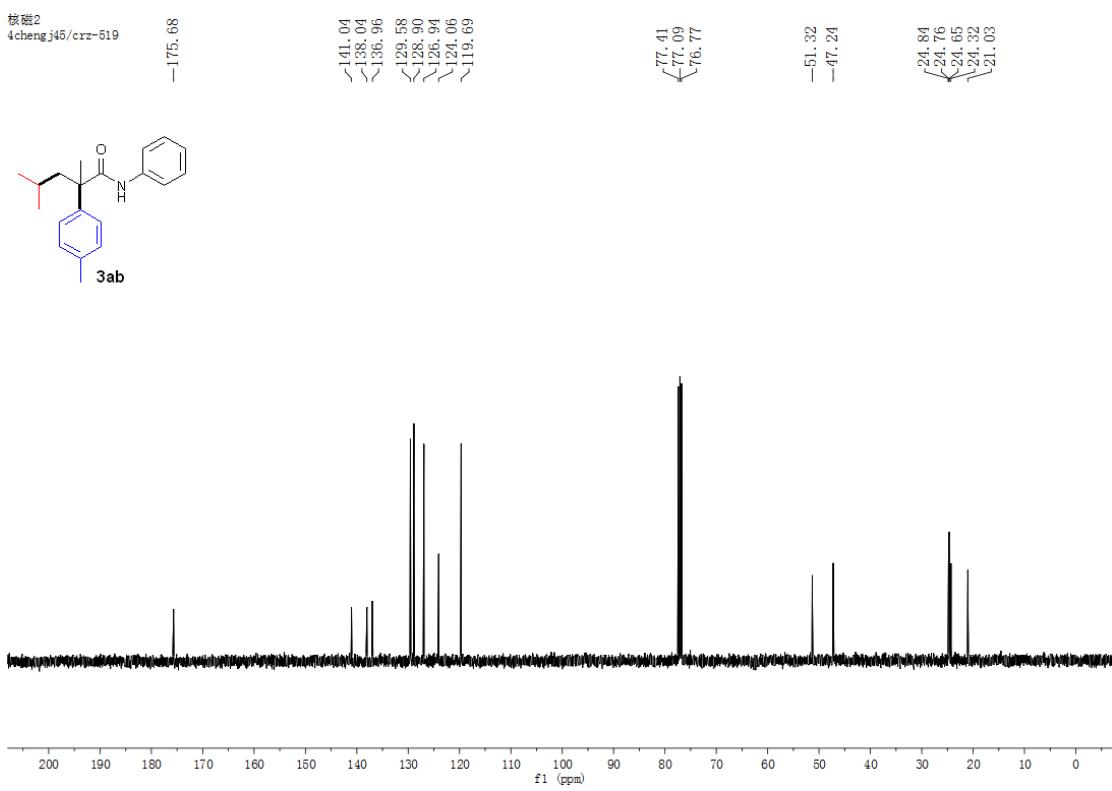
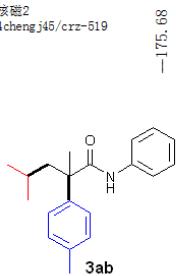
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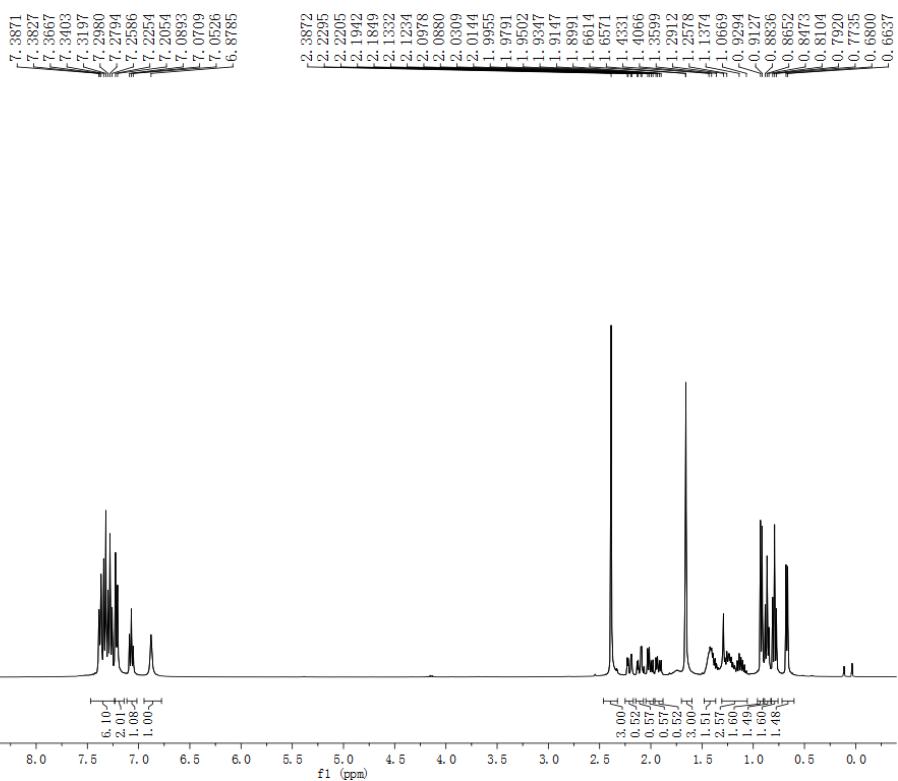
核磁2
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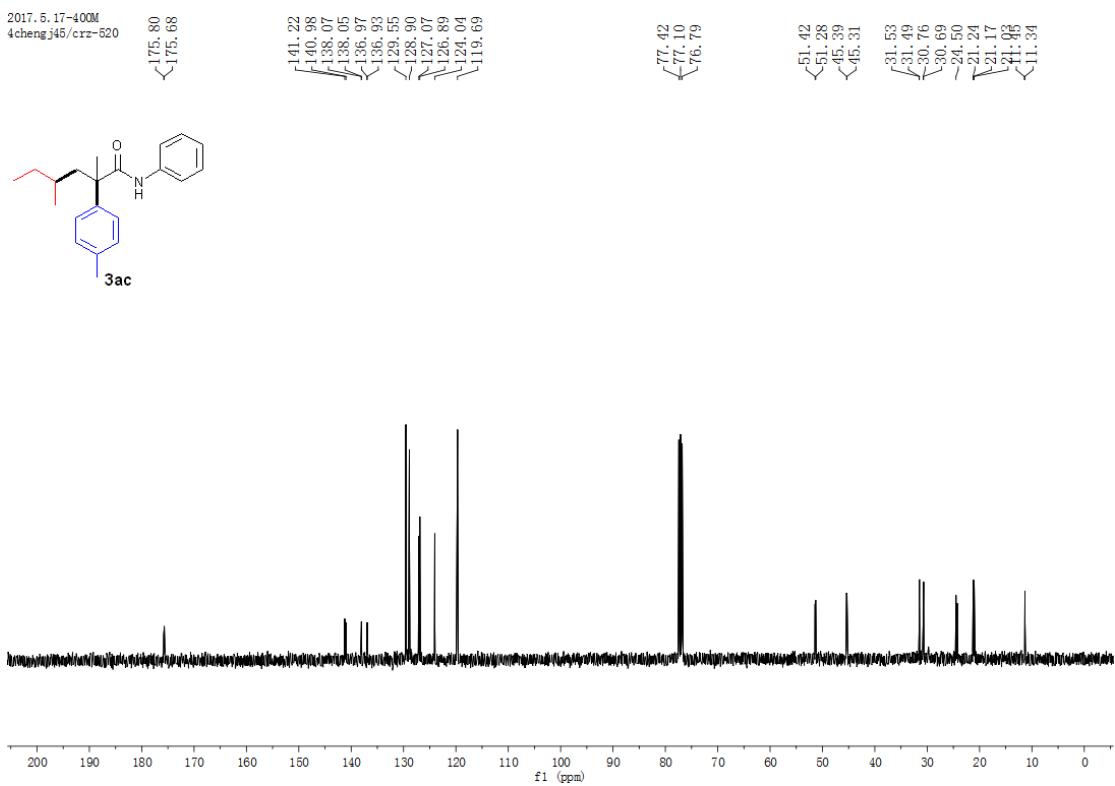
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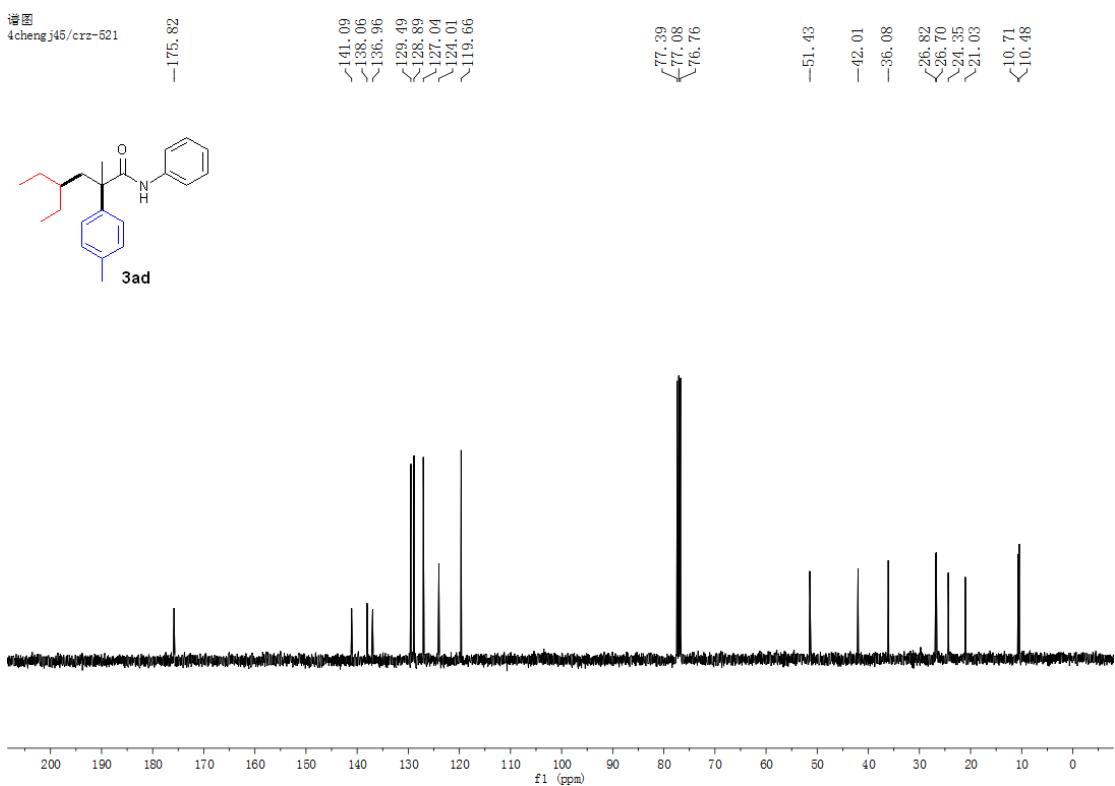
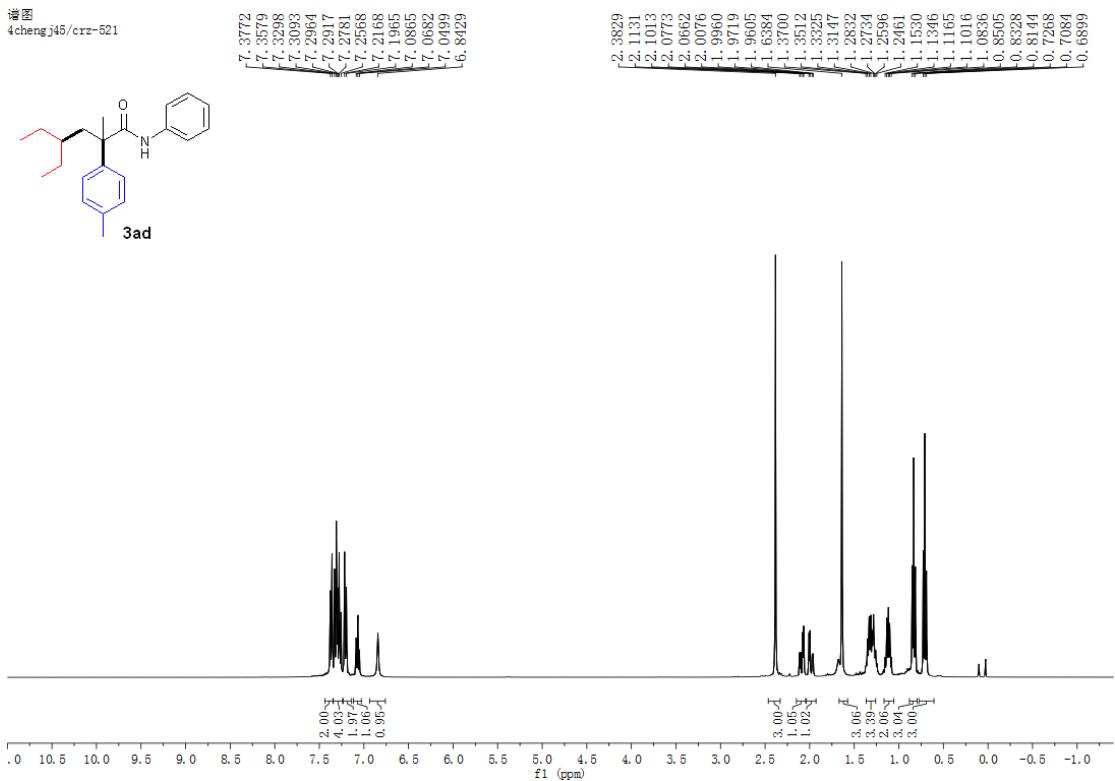


2017.5.17-400M
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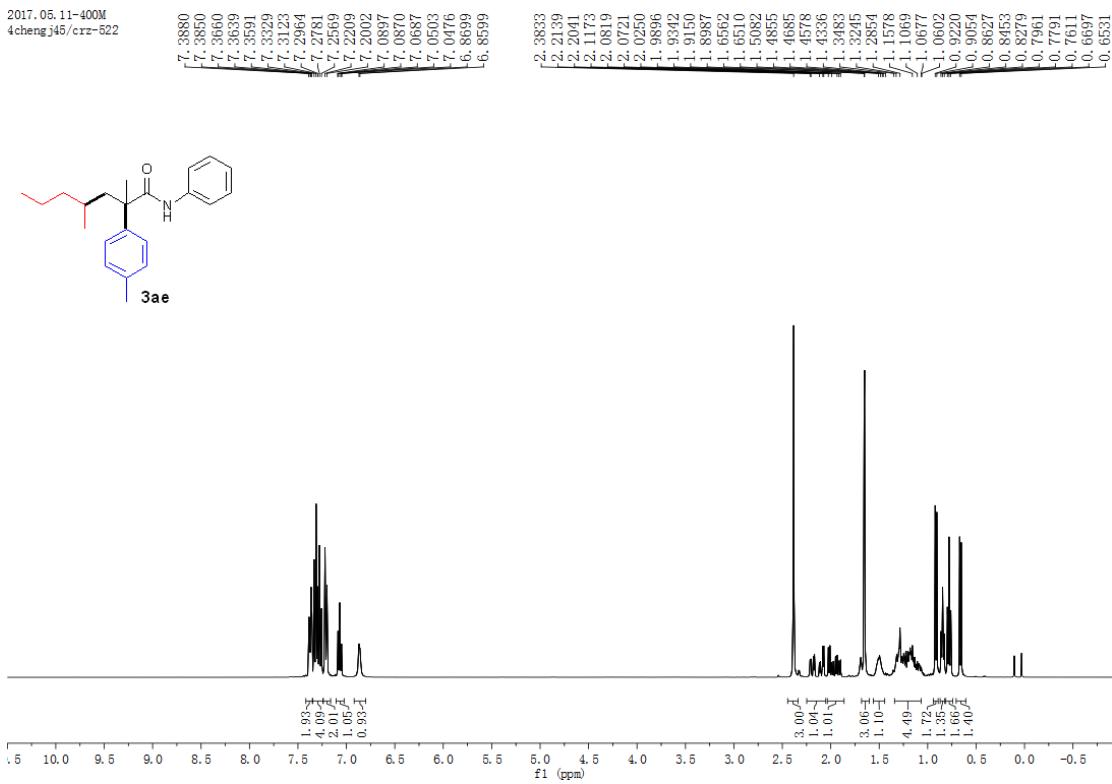
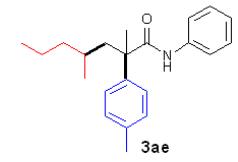


2017.5.17-400M
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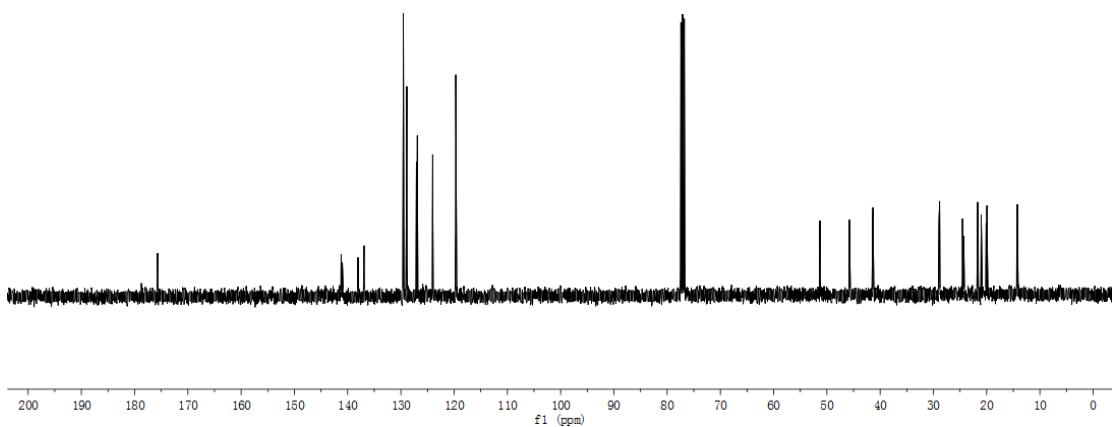
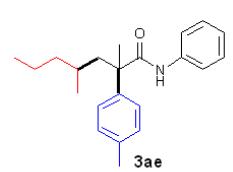




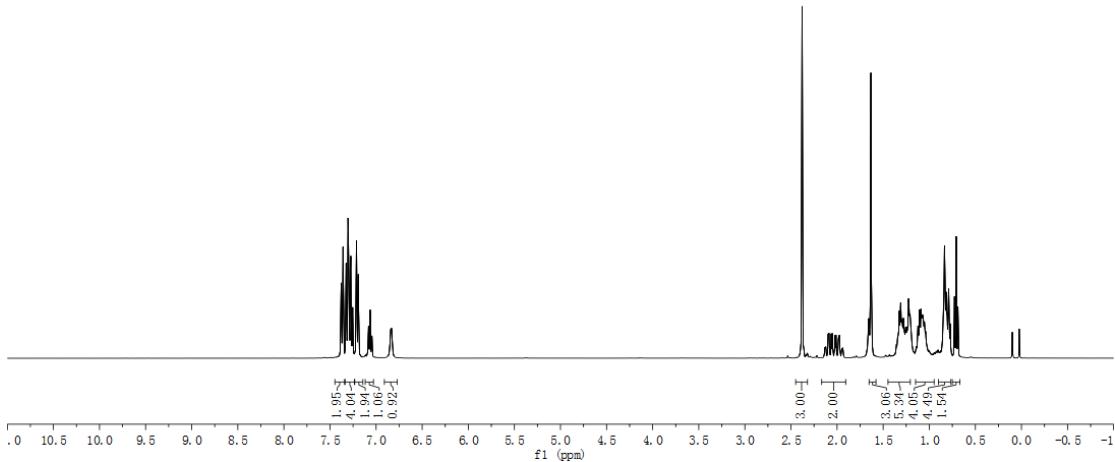
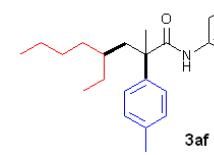
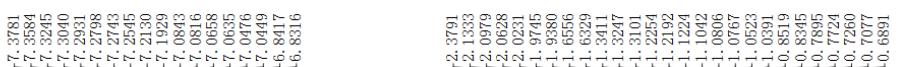
2017.05.11-400M
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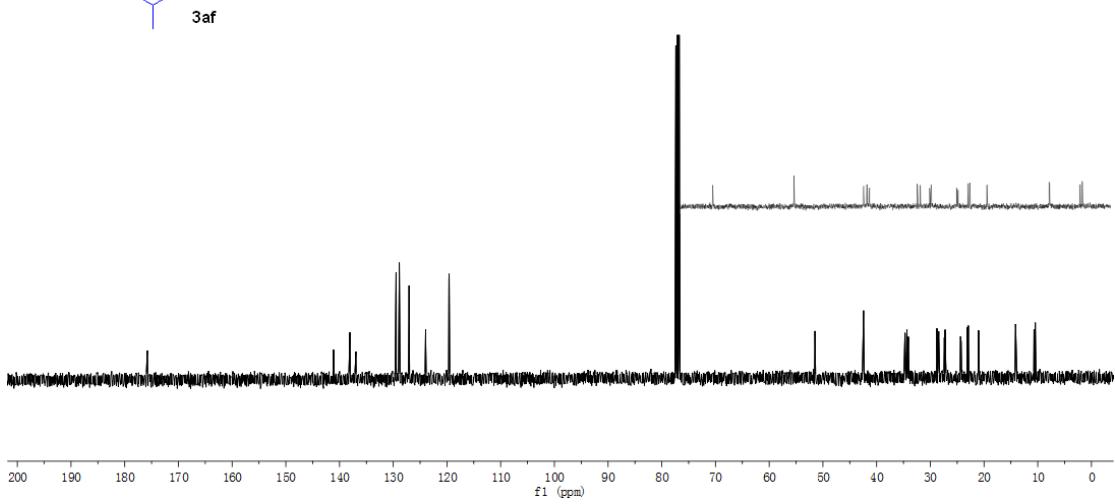
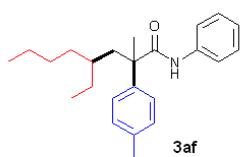
核磁2
4chengj45/crz-522



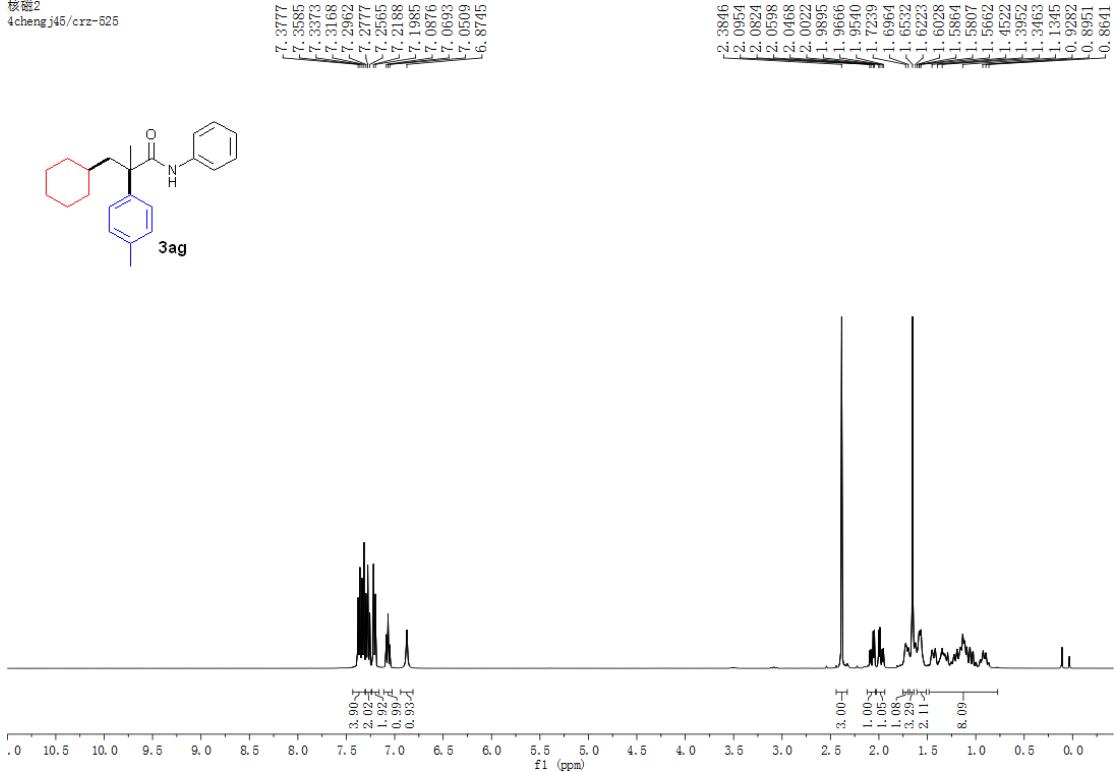
2017.05.11-400M
4chengj45/crz-23



2017.05.11-400M
4chengj45/crz-23



核磁2
4chengj45/crz-525



核磁2
4chengj45/crz-525

