Electronic Supplementary Information

Catalytic Asymmetric α-Amination of β-Keto Esters and β-Keto Amides with a Chiral *N*,*N*′-Dioxide-Copper(I) Complex

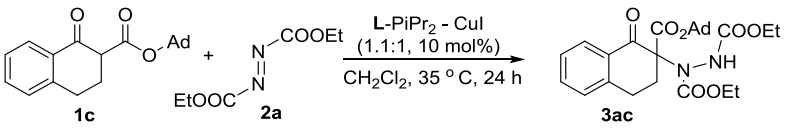
Xiao Xiao, Lili Lin,* Xiangjin Lian, Xiaohua Liu and Xiaoming Feng*

Contents:

1. General remarks

^{1}H NMR spectra were recorded on commercial instruments (400 MHz). Chemical shifts were reported in ppm from tetramethylsilane with the solvent resonance as the internal standard ($CDCl_3$, δ = 7.26). Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C NMR spectra were collected on commercial instruments (100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm from the tetramethylsilane with the solvent resonance as internal standard ($CDCl_3$, δ = 77.16). The enantiomeric excess was determined by HPLC analysis on commercial chiral columns. Optical rotations were measured on a commercial polarimeter and reported as follows: $[\alpha]^T_D$ (c = g/100 mL, solvent). HR-ESIMS spectra were recorded using a commercial apparatus and methanol was used to dissolve the sample. Unless otherwise indicated, reagents obtained from commercial sources were used without further purification. Solvents were dried and distilled prior to use according to the standard methods. Azodicarboxylates were obtained from commercial sources but should be distilled or recrystallized prior to use. Substrates **1** were prepared according to the literature and references therein.[1] The *N*,*N*'-dioxdes were prepared according to the methods reported in the literature.[2] The racemic products of the catalytic asymmetric amination reactions were prepared with the CuI.

2. Typical experimental procedure for the catalytic asymmetric α-Amination reaction

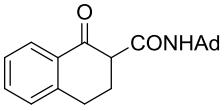


1c **2a** **3ac**

Ad=1-adamantyl

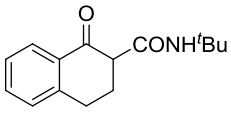
A dry reaction tube was charged with CuI (1.9 mg, 0.01 mmol), **L-PiPr$_2$** (7.2 mg, 0.011 mmol) and substrate **1c** (0.1mmol). Then, CH_2Cl_2 (0.5 mL) was added and the mixture was stirred at 35 ^{o}C for 0.5 h. Next, substrate **2a** (0.12 mmol) was added. The mixture was stirred at 35 ^{o}C for 24 hours. The residue was purified by flash chromatography (petroleum ether/AcOEt, 5:1 to 3:1) on silica gel to afford the products. The enantiomeric excesses (*ee*) were determined by high–performance liquid chromatography (HPLC).

3. Characterization of the substrate 4

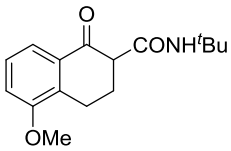


4a (Table 3, entries 1-3): $C_{21}H_{25}NO_2$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 8.01 -8.03 (d, 1H, *J* = 8.0 Hz), 7.47-7.51 (t, 1H, *J* = 8.0 Hz), 7.23-7.33 (m, 2H), 6.72 (s, 1H), 3.28 -3.32 (m, 1H), 2.84-3.18 (m, 2H), 2.39-2.45 (m, 2H), 2.03-2.06 (m, 9H), 1.67-1.71 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 196.9, 166.1, 144.9, 134.1, 131.8, 128.8, 127.7, 126.6, 53.2, 52.0, 41.4, 36.4, 29.4, 27.7, 25.2. ESI-HRMS: calcd for $C_{21}H_{25}NO_2Na^+$, ($[M + Na]^+$) 346.1783, found

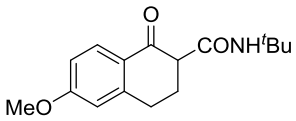
346.1790.



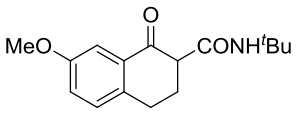
4b (Table 3, entry 4-6): $C_{15}H_{19}NO_2$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 8.02 -8.04 (d, 1H, *J* = 8.0 Hz), 7.48-7.52 (t, 1H, *J* = 7.2 Hz), 7.25-7.34 (m, 2H), 6.91 (s, 1H), 3.28-3.32 (m, 1H), 2.85-2.95 (m, 2H), 2.41-2.46 (m, 2H), 1.37 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 197.1, 166.2, 144.8, 134.2, 134.2, 132.0, 128.8, 127.8, 126.8, 53.2, 51.4, 28.7, 27.8, 25.3. ESI-HRMS: calcd for $C_{15}H_{19}NO_2Na^+$, ($[M + Na]^+$) 268.1313, found 268.1310.



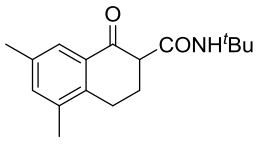
4c (Table 3, entry 7): $C_{16}H_{21}NO_3$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.62-7.64 (m, 1H), 7.25-7.29 (m, 1H), 7.02-7.04 (m, 1H), 6.89 (s, 1H), 3.86 (s, 3H), 3.24-3.27 (m, 1H), 2.82-3.14 (m, 2H), 2.38-2.44 (m, 2H), 1.36 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 197.3, 167.0, 156.6, 133.9, 132.9, 126.9, 119.1, 114.8, 55.6, 53.0, 51.3, 29.6, 28.6, 24.7, 21.3. ESI-HRMS: calcd for $C_{16}H_{20}NO_3^-$, ($[M - H]^-$) 274.1448, found 274.1435.



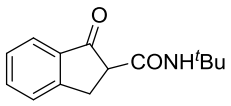
4d (Table 3, entry 8): $C_{16}H_{21}NO_3$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.52-7.72 (m, 1H), 7.00 (s, 1H), 6.81-6.84 (m, 1H), 6.69-7.00 (m, 1H), 4.21-4.23 (m, 1H), 3.86 (s, 3H), 3.24-3.27 (m, 1H), 3.06-3.14 (m, 1H), 2.82-2.86 (m, 1H), 2.38-2.44 (m, 2H), 1.36 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 195.9, 166.6, 164.3, 147.6, 132.5, 130.2, 125.5, 113.3, 112.5, 68.2, 55.5, 51.3, 38.7, 28.8, 25.3, 23.1, 14.2. ESI-HRMS: calcd for $C_{16}H_{20}NO_3^-$, ($[M - H]^-$) 274.1448, found 274.1439.



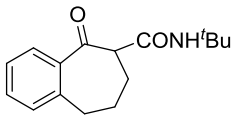
4e (Table 3, entry 9): $C_{16}H_{21}NO_3$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.50-7.51 (m, 1H), 7.16-7.18 (m, 1H), 7.07-7.10 (m, 1H), 6.86 (s, 1H), 3.83 (s, 3H), 3.26-3.29 (m, 1H), 3.04-3.11 (m, 1H), 2.80-2.88 (m, 1H), 2.38-2.44 (m, 2H), 1.37 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 197.2, 166.6, 158.5, 137.7, 132.8, 130.1, 122.7, 109.6, 55.6, 53.3, 51.5, 28.9, 27.1, 25.6. ESI-HRMS: calcd for $C_{16}H_{20}NO_3^-$, ($[M - H]^-$) 274.1448, found 274.1435.



4f (Table 3, entry 10): $C_{17}H_{23}NO_2$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.72-7.50 (m, 1H), 7.50 (s, 1H), 6.96 (s, 1H), 6.67 (s, 1H), 3.95 (s, 3H), 3.91 (s, 3H), 3.23-3.26 (m, 1H), 3.06-3.13 (m, 1H), 2.79-2.87 (m, 1H), 2.39-2.45 (m, 2H), 1.37 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 195.8, 166.6, 154.4, 148.1, 140.3, 131.0, 128.8, 125.1, 110.2, 108.7, 68.2, 56.1, 52.6, 51.4, 38.8, 28.8, 27.6, 25.6, 23.8, 14.0, 11.1. ESI-HRMS: calcd for $C_{17}H_{22}NO_2^-$, ($[M - H]^-$) 272.1656, found 272.1653.

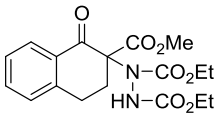


4g (Table 3, entry 11): $C_{14}H_{17}NO_2$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.73-7.75 (d, 1H, *J* = 8.0 Hz), 7.60-7.64 (m, 1H), 7.50-7.52 (d, 1H, *J* = 8.0 Hz), 7.36-7.40 (m, 1H), 7.03 (s, 1H), 3.75-3.80 (m, 1H), 3.49-3.52 (m, 1H), 3.27-3.33 (m, 1H), 1.39 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 203.9, 165.3, 154.5, 135.8, 135.5, 127.6, 126.8, 124.4, 53.6, 51.5, 28.8, 28.6. ESI-HRMS: calcd for $C_{14}H_{16}NO_2^-$, ($[M - H]^-$) 230.1186, found 230.1185.

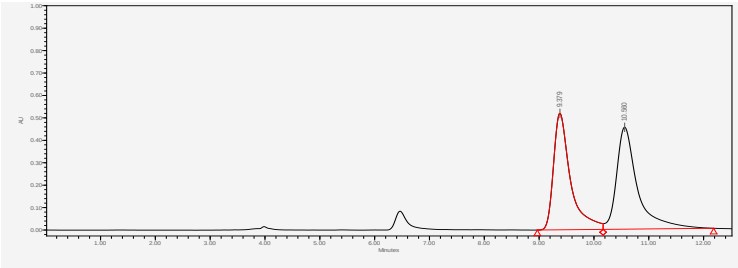


4h (Table 3, entry 12): $C_{16}H_{21}NO_2$ ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.62-7.64 (m, 1H), 7.41-7.43 (m, 1H), 7.28-7.32 (m, 1H, *J* = 8.0 Hz), 7.20-7.22 (d, 1H, *J* = 8.0 Hz), 7.10 (s, 1H), 3.53-3.57 (m, 1H), 2.89-2.92 (m, 2H), 1.90-2.17 (m, 4H), 1.38 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 206.1, 167.5, 141.4, 139.0, 132.4, 129.7, 128.4, 126.6, 53.3, 51.2, 32.5, 29.2, 28.8, 26.9, 24.4. ESI-HRMS: calcd for $C_{16}H_{20}NO_2^-$, ([M - H]$^-$) 258.1499, found 258.1494.

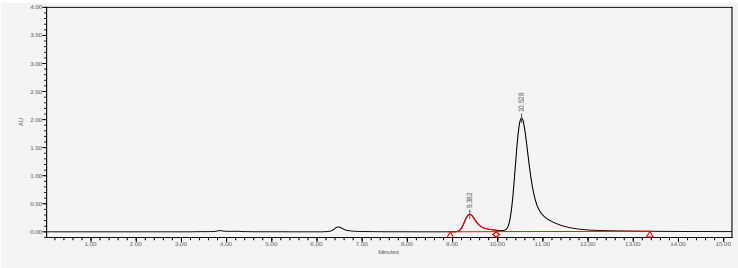
4. Characterization of the products 3 and 5



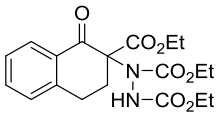
3aa (Table 2, entry 1): $C_{18}H_{22}N_2O_7$ Yellow oil in 94% yield. [α] $^{25.8}_D$ = −16.111 (c = 0.76 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.92 -8.00 (m, 1H), 7.46-7.50 (m, 1H), 7.21-7.30 (m, 2H), 6.48-6.74 (m, 1H), 3.97 -4.25 (m, 4H), 3.80 (s, 3H), 2.72-3.52 (m, 4H), 1.25-1.29(t, 3H, *J* = 7.2 Hz), 1.17-1.11 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 169.3, 156.9, 143.7, 133.8, 133.4, 132.2, 131.7, 128.5, 127.9, 126.7, 75.6, 68.4, 68.4, 25.7, 5, 63.3, 62.0, 52.9, 31.1, 25.7, 14.3. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major = 10.52 min, t_R minor = 9.38 min. ESI-HRMS: calcd for $C_{18}H_{22}N_2O_7Na^+$, ([M + Na] $^+$) 401.1325, found 401.1318.



Peak	Retention Time	Area	% Area
1	9.379	11180998	48.12
2	10.560	12056985	51.88

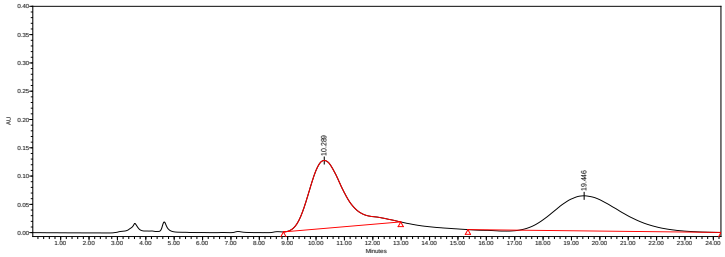


Peak	Retention Time	Area	% Area
1	9.382	6267238	10.23
2	10.528	55016038	89.77

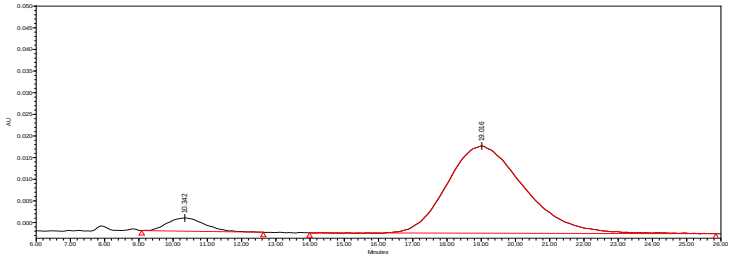


3ba (Table 2, entry 2): $C_{19}H_{24}N_2O_7$ Yellow oil in 92% yield. [α] $^{25.8}_D$ = −13.412 (c = 0.75 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.92 -8.01 (m, 1H), 7.46-7.50 (m, 1H), 7.24-7.30 (m, 2H), 6.37-6.65 (m, 1H), 3.95 -4.31 (m, 6H), 2.68-3.68 (m, 4H), 1.24-1.29 (m, 6H), 1.09-1.18 (m,

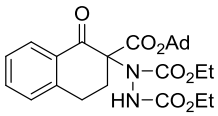
3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 168.6, 157.0, 144.1, 133.8, 133.3, 132.3, 131.7, 128.6, 128.5, 128.1, 127.9, 126.7, 75.5, 63.2, 62.5, 62.0, 31.0, 25.7, 14.4, 13.8. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ASH, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major = 19.01 min, t_R minor = 10.34 min. ESI-HRMS: calcd for $C_{19}H_{24}N_2O_7Na^+$, ([M + Na] $^+$) 415.1481, found 415.1474.



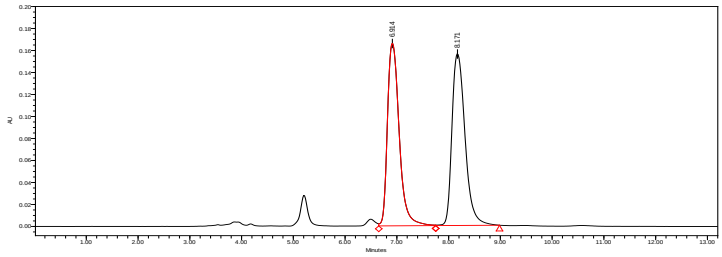
Peak	Retention Time	Area	% Area
1	10.289	10547603	50.85
2	19.446	10194172	49.15



Peak	Retention Time	Area	% Area
1	10.342	234572	6.91
2	19.016	3158755	93.09

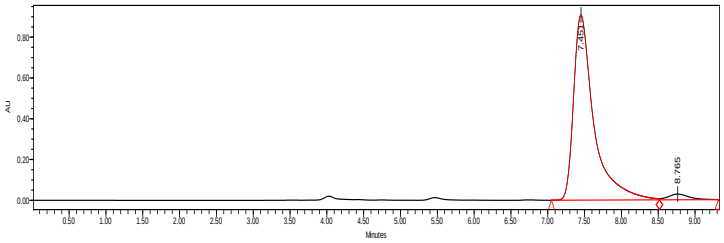


3ca (Table 2, entry 3): $C_{27}H_{34}N_2O_7$ white oil in 96% yield. [α] $^{25.8}_{D}$ = −14.859 (c = 0.90 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.80 -8.01 (m, 1H), 7.44-7.49 (m, 1H), 7.20 -7.32 (m, 2H), 6.24-6.67 (m, 1H), 3.98-4.32 (m, 4H), 2.64-3.42 (m, 4H), 2.09-2.15 (m, 9H), 1.63 (s, 6H), 1.27-1.31 (t, 3H, *J* = 7.2 Hz), 1.09-1.18 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 167.2, 133.6, 133.3, 128.6, 128.2, 127.8, 126.7, 83.8, 83.1, 75.8, 63.4, 62.0, 41.1, 36.3, 31.4, 30.9, 27.0, 25.9, 14.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major = 7.45 min, t_R minor = 8.76 min. ESI-HRMS: calcd for $C_{27}H_{34}N_2O_7Na^+$, ([M + Na] $^+$) 521.2258, found 521.2255.

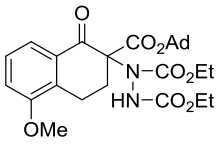


Peak	Retention Time	Area	% Area

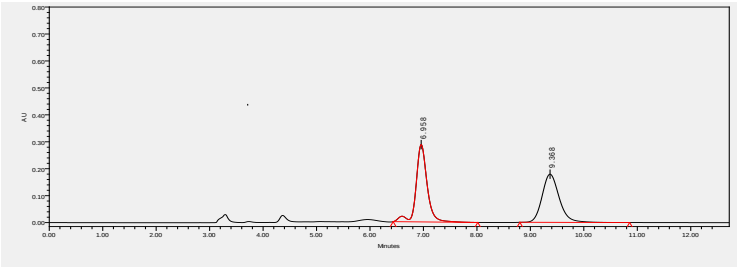
1	6.914	2619738	48.88
2	8.171	2739610	51.12



Peak	Retention Time	Area	% Area
1	7.451	17300947	97.33
2	8.765	474712	2.67



3da (Table 2, entry 4): $C_{28}H_{36}N_2O_8$ Yellow oil in 91% yield. $[\alpha]_D^{25.8}$ = −12.373 (c = 0.96 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.51 -7.61 (m, 1H), 7.23-7.27 (m, 1H), 6.99 -7.01 (d, 1H, *J* = 8 Hz), 6.23-6.61 (m, 1H), 4.00-4.24 (m, 4H), 3.85-3.86 (m, 3H), 2.61 -3.29 (m, 4H), 2.10-2.14 (m, 9H), 1.63(s, 6H), 1.27-1.30 (m, 3H), 1.11-1.18 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 167.2, 156.4, 133.3, 132.7, 127.2, 119.9, 114.3, 114.0, 83.7, 83.0, 75.5, 63.3, 62.0, 55.7, 41.1, 36.2, 30.9, 26.9, 20.2, 19.8, 14.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ASH, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major = 6.84 min, t_R minor = 9.18 min. ESI-HRMS: calcd for $C_{28}H_{36}N_2O_8Na^+$, ($[M + Na]^+$) 551.2364, found 551.2343.



Peak	Retention Time	Area	% Area
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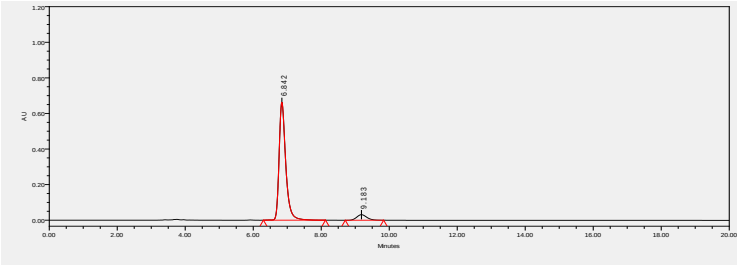
2	9.368	3966497	48.98



Peak	Retention Time	Area	% Area
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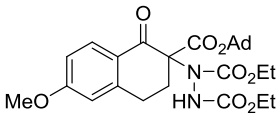
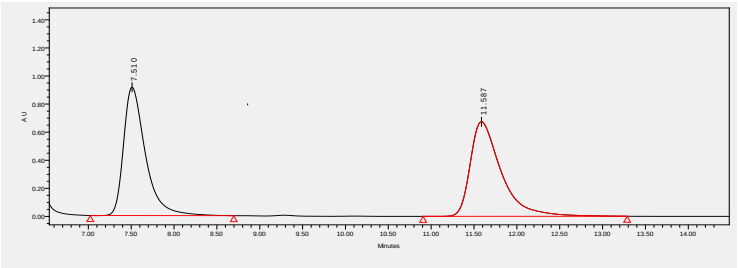
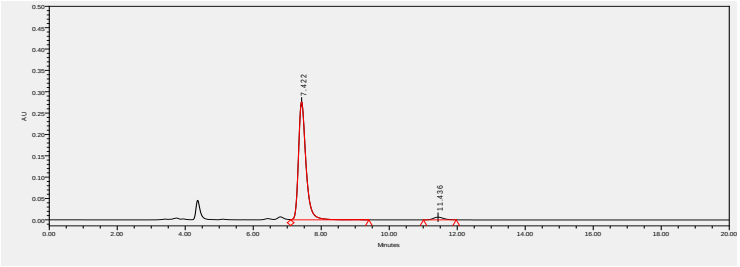
2	9.183	480723	5.07



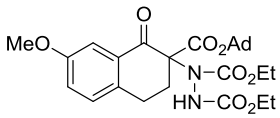
3ea (Table 2, entry 5): $C_{28}H_{36}N_2O_8$ Yellow oil in 98% yield. $[\alpha]_D^{25.8}$ = −16.096 (c = 0.92 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.88 -7.99 (m, 1H), 6.79-6.84 (m, 1H), 6.66 -6.69 (m, 1H), 6.30-6.51 (m, 1H), 3.85-4.24 (m, 4H), 3.85 (s, 3H), 2.62 -3.32 (m, 4H), 2.12-2.15 (m, 9H), 1.64 (s, 6H), 1.27-1.30 (m, 3H), 1.10-1.18 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 190.0, 163.8, 163.6, 130.5, 130.1, 125.9, 125.4, 113.5, 112.1, 83.1, 82.9, 75.6, 63.1, 61.8, 55.5, 41.0, 36.1, 31.3, 30.8, 26.9, 26.0, 20.2, 14.4. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ASH, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =7.42 min, t_R minor = 11.44 min. ESI-HRMS: calcd for $C_{28}H_{36}N_2O_8Na^+$, ([M + Na] $^+$) 551.2364, found 551.2380



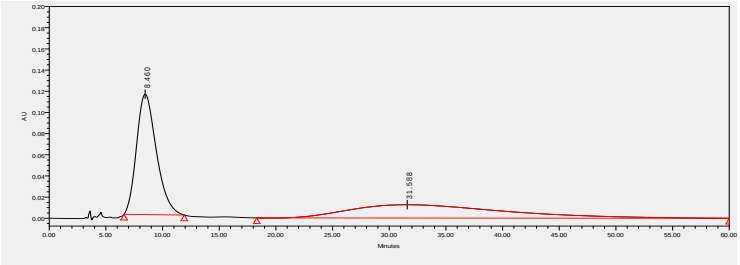
Peak	Retention Time	Area	% Area
1	7.510	16343273	49.23
2	11.587	16856002	50.77



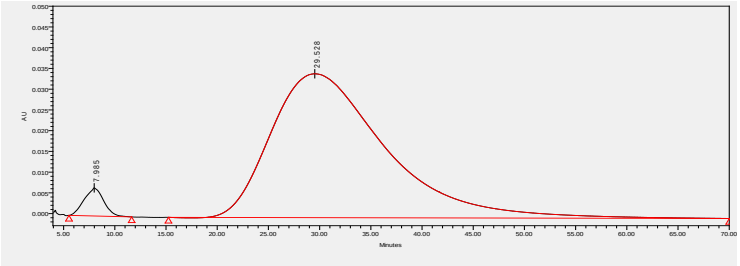
Peak	Retention Time	Area	% Area
1	7.422	4222245	96.95
2	11.436	132787	3.05



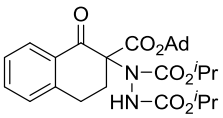
3fa (Table 2, entry 6): $C_{28}H_{36}N_2O_8$ Yellow oil in 95% yield. $[\alpha]_D^{25.8}$ = −21.373 (c = 0.88 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.39-7.49 (m, 1H), 7.11-7.16 (m, 1H), 7.05 -7.07 (m, 1H), 6.23-6.469 (m, 1H), 4.02-4.25 (m, 4H), 3.80-3.83 (m, 3H), 3.85 (s, 3H), 2.64 -3.30 (m, 4H), 2.10-2.15 (m, 9H), 1.63 (s, 6H), 1.27-1.31 (m, 3H), 1.11-1.20 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 158.3, 129.8, 122.2, 122.1, 109.8, 109.5, 83.8, 83.2, 75.6, 63.2, 62.0, 55.5, 41.1, 36.1, 31.8, 30.7, 25.2, 14.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ASH, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =29.53 min, t_R minor = 7.99 min. calcd for $C_{28}H_{36}N_2O_8Na^+$, ([M + Na] $^+$) 551.2364, found 551.2360.



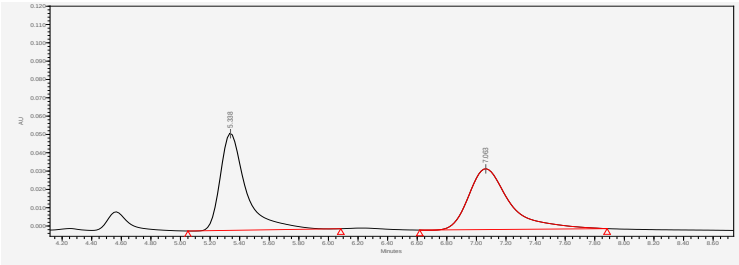
Peak	Retention Time	Area	% Area
1	8.460	13901663	53.75
2	31.588	11960797	46.25



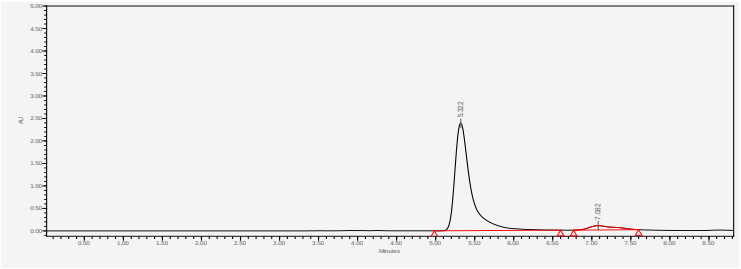
Peak	Retention Time	Area	% Area
1	7.985	896929	3.22
2	29.528	26963286	96.78



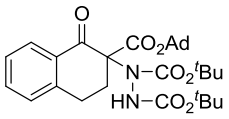
3cb (Table 2, entry 7): $C_{29}H_{38}N_2O_7$ Yellow oil in 95% yield. $[\alpha]_D^{25.8}$ = −17.545 (c = 1.02 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.89-8.00 (m, 1H), 7.45-7.48 (m, 1H), 7.19 -7.29 (m, 2H), 6.17-6.56 (m, 1H), 4.68-5.14 (m, 2H), 2.62 -3.44 (m, 4H), 2.10-2.13 (m, 9H), 1.64 (s, 6H), 1.21-1.39 (m, 6H), 0.79-1.14 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 156.4, 155.2, 133.7, 133.2, 128.5, 128.1, 127.7, 126.7, 126.6, 83.7, 83.0, 75.7, 69.6, 41.2, 41.1, 36.2, 30.1, 29.5, 25.8, 25.7, 22.2, 22.0, 21.9. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ASH, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =5.32 min, t_R minor = 7.08 min. calcd for $C_{29}H_{38}N_2O_7Na^+$, ($[M + Na]^+$) 549.2571, found 549.2587.



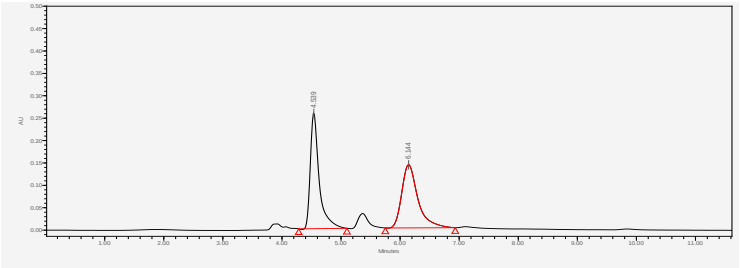
Peak	Retention Time	Area	% Area
1	5.338	642416	50.00
2	7.063	642539	50.00



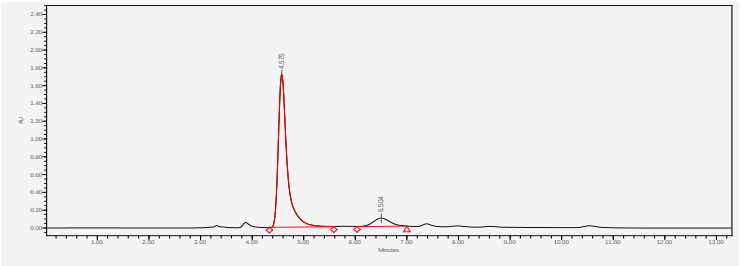
Peak	Retention Time	Area	% Area
1	5.322	33659645	94.74
2	7.082	1868114	5.26



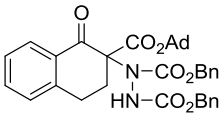
3cc (Table 2, entry 8): $C_{31}H_{42}N_2O_7$ Yellow oil in 76% yield. $[\alpha]_D^{25.8}$ = −11.770 (c = 1.09 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.85-8.01 (m, 1H), 7.20-7.46 (m, 3H), 6.03-6.52 (m, 1H), 2.56 -3.42 (m, 4H), 2.01-2.18 (m, 9H), 1.63-1.66 (m, 6H), 1.39-1.51 (m, 12H), 1.15-1.19 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 156.0, 154.3, 133.6, 129.0, 128.0, 126.7, 83.3, 82.7, 81.0, 40.9, 36.0, 30.7, 28.4, 25.7. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =4.58 min, t_R minor = 6.50 min. calcd for $C_{31}H_{42}N_2O_7Na^+$, ($[M + Na]^+$) 577.2890, found 577.2894.



Peak	Retention Time	Area	% Area
1	4.539	2688141	50.13
2	6.144	2674303	49.87

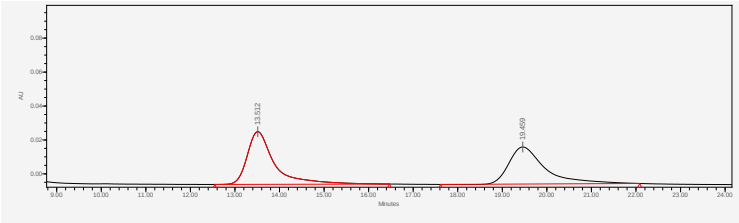


Peak	Retention Time	Area	% Area
1	4.575	20602395	90.85
2	6.504	2074892	9.15

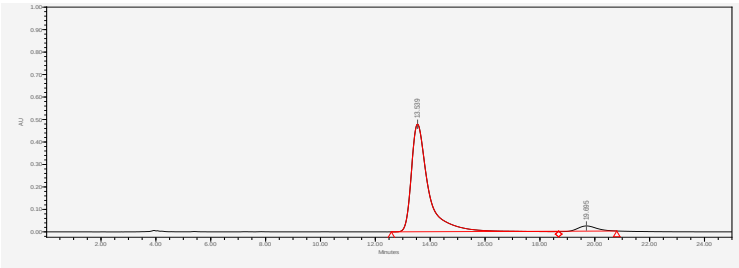


3cd (Table 2, entry 9): $C_{37}H_{38}N_2O_7$ Yellow oil in 98% yield. $[\alpha]_D^{25.8}$ = −27.417 (c = 1.22 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.87-7.88 (m, 1H), 7.01 -7.46 (m, 13H), 6.38-6.84 (m, 1H), 4.59-5.20 (m, 4H), 2.66 -3.30 (m, 4H), 2.00-2.10 (m, 9H), 1.59 (s, 6H). ^{13}C NMR

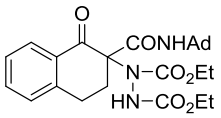
(100 MHz, $CDCl_3$, 25 ^oC): δ = 167.5, 135.6, 133.4, 133.1, 128.6, 128.5, 128.3, 128.1, 128.0, 126.9, 126.7, 126.6, 75.8, 67.9, 67.5, 41.1, 36.0, 31.3, 30.9, 25.9. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ASH, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =13.54 min, t_R minor = 19.70 min. calcd for $C_{37}H_{38}N_2O_7$ Na^+, ([M + Na] $^+$) 645.2577, found 645.2576.



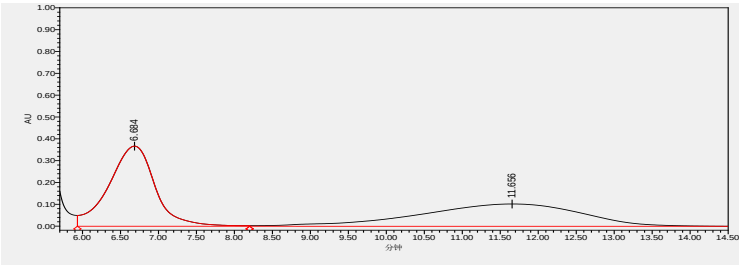
Peak	Retention Time	Area	% Area
1	13.512	1319423	51.49
2	19.459	1242978	48.51



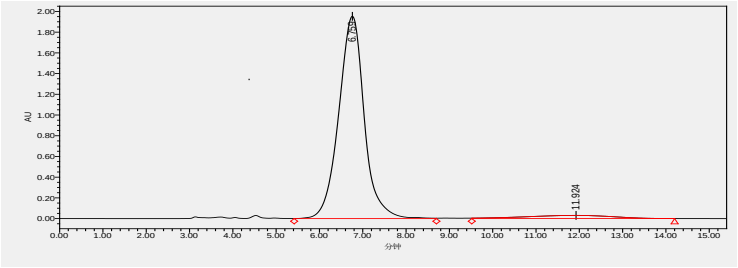
Peak	Retention Time	Area	% Area
1	13.539	20663041	94.96
2	19.695	1097819	5.04



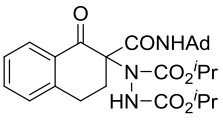
5aa (Table 3, entry 1): $C_{27}H_{35}N_3O_6$ Yellow oil in 95% yield. [α] $^{25.8}_D$ = −84.538 (c = 0.93 in CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$, 25 ^oC, TMS): δ = 7.88-8.02 (m, 1H), 7.17-7.57 (m, 4H), 6.82 (m, 1H), 4.08-4.29 (m, 4H), 2.28-3.25 (m, 4H), 2.01-2.04 (m, 9H), 1.64 (s, 1.64), 1.22-1.33 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^oC): δ = 193.4, 168.3, 166.5, 143.0, 142.0, 133.9, 133.1, 128.4, 126.7, 75.0, 62.9, 51.8, 40.9, 36.4, 29.7, 26.7, 14.4, 14.0. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major = 6.76 min, t_R minor = 11.92 min. calcd for $C_{27}H_{35}N_3O_6$ Na^+, ([M + Na] $^+$) 520.2429, found 520.2424.



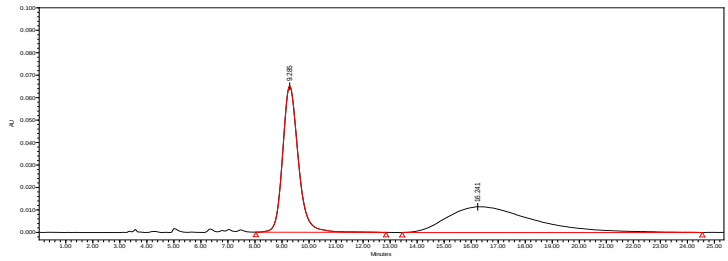
Peak	Retention Time	Area	% Area
1	6.684	15441084	50.50
2	11.656	15133094	49.50



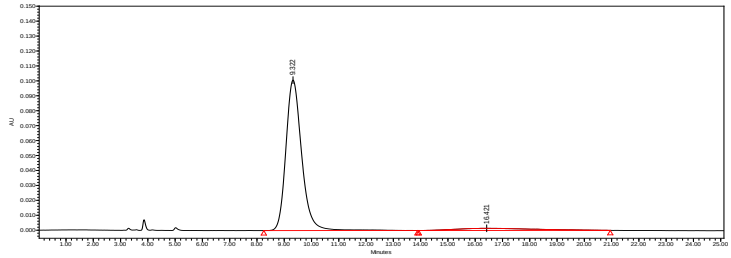
Peak	Retention Time	Area	% Area
1	6.759	79956089	94.91
2	11.924	4290436	5.09



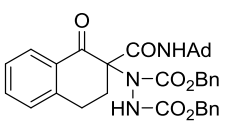
5ab (Table 3, entry 2): $C_{29}H_{39}N_3O_6$ Yellow oil in 90% yield. $[\alpha]_D^{25.8}$ = −89.355 (c = 0.97 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.76-8.01 (m, 1H), 6.74-7.43 (m, 5H), 4.91-5.06 (m, 2H), 2.25 -3.25 (m, 4H), 2.01-2.09 (m, 9H), 1.64-1.66 (m, 6H), 1.16-1.34 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =193.7, 168.3, 159.0, 156.9, 155.5, 143.1, 141.9, 133.8, 128.5, 126.8, 126.5, 83.7, 83.0, 74.9, 72.4, 70.4, 52.4, 51.7, 41.2, 36.5, 29.5, 25.8, 26.5, 22.3, 22.0, 21.9, 21.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 80/20, flow rate 1.0 mL/min, detection at 254 nm): t_R major =9.32 min, t_R minor = 16.42 min. calcd for $C_{29}H_{39}N_3O_6$ Na^+, ($[M + Na]^+$) 549.2571, found 549.2573.



Peak	Retention Time	Area	% Area
1	9.285	2622118	51.07
2	16.241	2512617	48.93

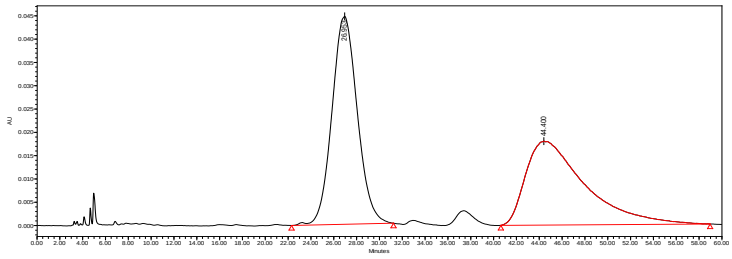


Peak	Retention Time	Area	% Area
1	9.322	4055460	93.35
2	16.421	288903	6.65

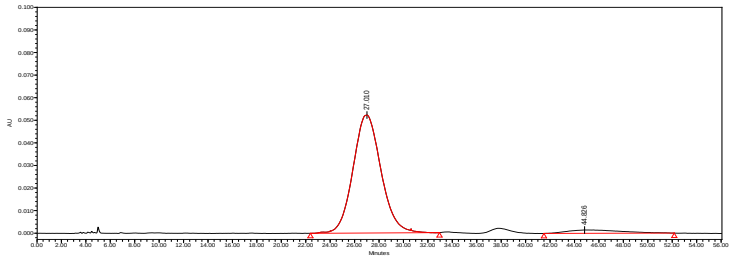


5ad (Table 3, entry 3): $C_{37}H_{39}N_3O_6$ Yellow oil in 96% yield. $[\alpha]_D^{25.8}$ = −74.252 (c = 1.07 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.78-7.99 (m, 1H), 7.04-7.49 (m, 15H), 4.91-5.29 (m, 4H), 2.25 -3.25

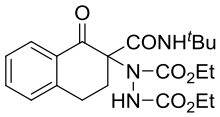
(m, 4H), 1.77-1.97(m, 6H), 1.54-1.62 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =193.4, 167.9, 159.2, 157.2, 155.9, 143.2, 142.0, 135.2, 132.3, 128.7, 128.6, 128.4, 128.2, 128.1, 127.2, 126.8, 75.1, 68.1, 51.8, 40.9, 36.4, 29.4, 26.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ID, hexane/ 2-propanol = 80/20, flow rate 1.0 mL/min, detection at 254 nm): t_R major =27.01 min, t_R minor = 44.83 min. calcd for $C_{37}H_{39}N_3O_6\ Na^+$, ([M + Na] $^+$) 644.2737, found 644.2734.



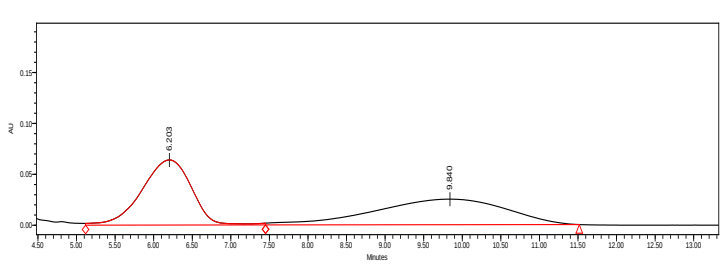
Peak	Retention Time	Area	% Area
1	26.953	6887386	51.47
2	44.400	6492801	48.53



Peak	Retention Time	Area	% Area
1	27.010	8326293	94.82
2	44.826	454648	5.18

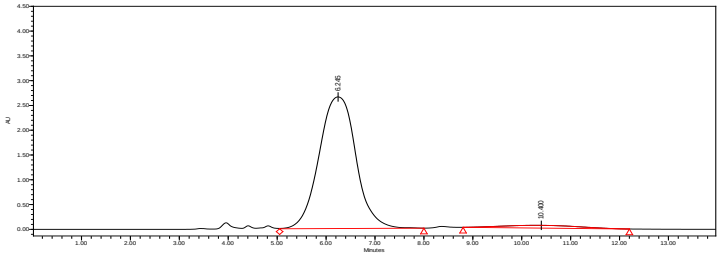


5ba (Table 3, entry 4): $C_{21}H_{29}N_3O_6$ Yellow oil in 96% yield. [α] $^{25.8}_{D}$ = −112.857 (c = 0.75 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.89-8.02 (m, 1H), 7.17-7.71 (m, 4H), 6.61-6.84 (m, 1H), 4.14- 4.28 (m, 4H), 2.29-3.22 (m, 4H), 0.86 -1.75 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =193.6, 168.6, 159.5, 157.4, 143.1, 142.0, 133.8, 133.1, 128.8, 128.3, 127.3, 126.9, 63.7, 62.9, 51.7, 51.2, 33.0, 28.5, 26.6, 14.0. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =6.24 min, t_R minor = 10.40 min. calcd for $C_{21}H_{29}N_3O_6\ Na^+$, ([M + Na] $^+$) 442.1954, found 442.1958.

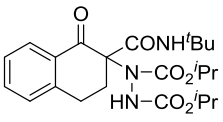


Peak	Retention Time	Area	% Area
1	6.203	2865062	49.02

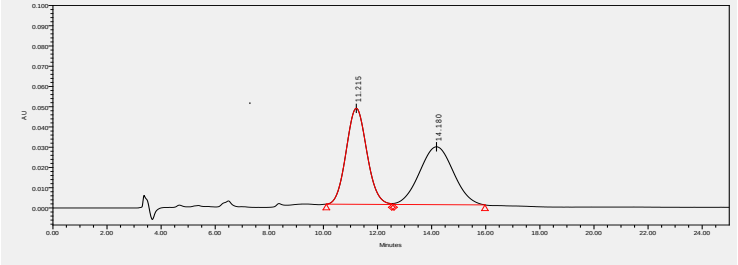
2	9.840	2980033	50.98



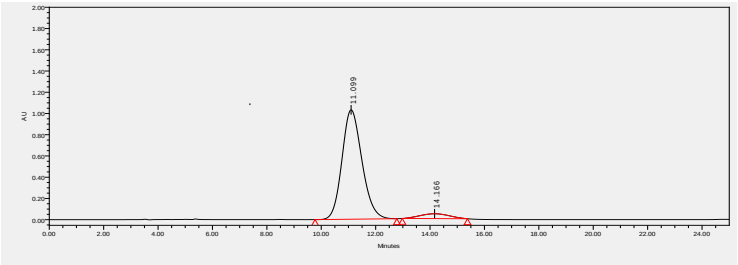
Peak	Retention Time	Area	% Area
1	6.245	139335921	95.85
2	10.400	6040095	4.15



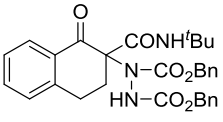
5bb (Table 3, entry 5): $C_{23}H_{33}N_3O_6$ Yellow oil in 90% yield. $[\alpha]^{25.8}_D$ = −104.392 (c = 0.84 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.96-8.01 (m, 1H), 7.16-7.88 (m, 4H), 6.63-6.86 (m, 1H), 4.92-5.02 (m, 2H), 2.27-3.22 (m, 4H), 1.20-1.38 (m, 21H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =193.7, 168.3, 166.9, 158.8, 156.9, 155.4, 143.1, 133.7, 132.5, 128.7, 128.1, 127.1, 127.0, 126.7, 72.5, 70.4, 51.9, 51.1, 33.1, 26.5, 21.8, 21.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ID, hexane/ 2-propanol = 80/20, flow rate 1.0 mL/min, detection at 254 nm): t_R major =11.09 min, t_R minor = 14.16 min. calcd for $C_{23}H_{33}N_3O_6$ Na^+, ($[M + Na]^+$) 470.2262, found 470.2265



Peak	Retention Time	Area	% Area
1	11.215	2522116	50.34
2	14.180	2488502	49.66

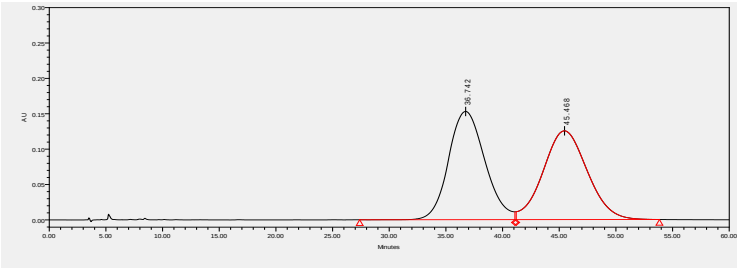


Peak	Retention Time	Area	% Area
1	11.099	52529702	93.85
2	14.166	3439745	6.15

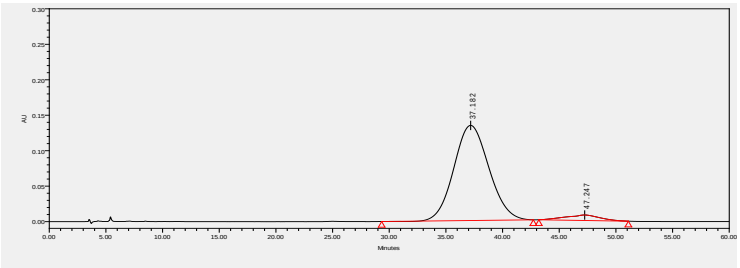


5bd (Table 3, entry 6): $C_{31}H_{33}N_3O_6$ Yellow oil in 94% yield. $[\alpha]^{25.8}_D$ = −97.508 (c = 0.99 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS):

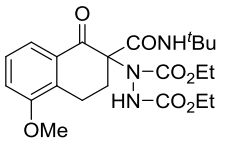
δ = 7.92-8.00 (m, 1H), 6.95-7.86 (m, 15H), 5.01-5.30 (m, 4H), 2.27-3.20 (m, 4H), 1.17-1.30 (m, 9H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ = 166.5, 143.1, 141.8, 135.1, 133.2, 128.7, 128.6, 128.2, 128.1, 127.9, 127.0, 126.8, 68.8, 68.3, 51.7, 51.1, 32.7, 31.2, 28.3, 26.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ID, hexane/ 2-propanol = 80/20, flow rate 1.0 mL/min, detection at 254 nm): t_R major =37.18 min, t_R minor = 47.25 min. calcd for $C_{31}H_{33}N_3O_6$ Na^+, ([M + Na] $^+$) 566.2267, found 566.2277



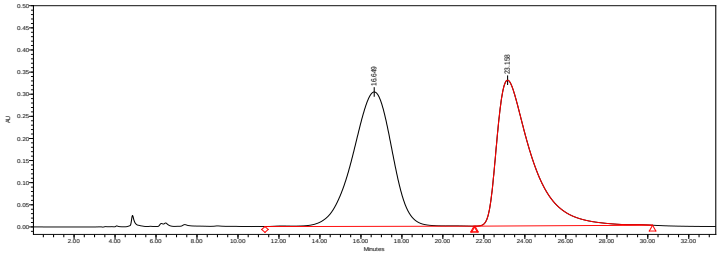
Peak	Retention Time	Area	% Area
1	36.742	34128939	50.14
2	45.468	33939708	49.86



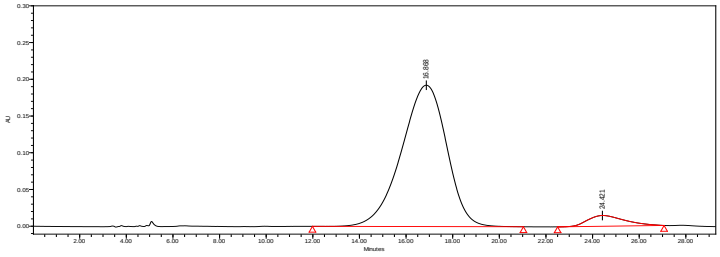
Peak	Retention Time	Area	% Area
1	37.182	28885671	95.18
2	47.247	1461347	4.82



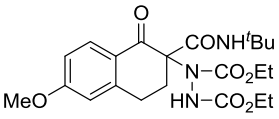
5ca (Table 3, entry 7): $C_{22}H_{31}N_3O_7$ Yellow oil in 90% yield. [α] $^{22.6}_{D}$ = −146.617 (c = 0.81 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.69 (s, 1H), 7.51-7.60 (m, 1H), 7.23-7.34 (m, 2H), 6.96-7.02 (m, 1H), 6.64- 6.81 (m, 1H), 4.13-4.34 (m, 4H), 3.84-3.86 (m, 3H), 2.31-3.19 (m, 4H), 1.20 -1.38 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =193.8, 168.1, 155.8, 130.8, 127.3, 118.7, 113.7, 74.1, 63.4, 55.7, 50.7, 32.1, 28.2, 20.6, 14.3. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ID, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =16.86 min, t_R minor = 24.42 min. calcd for $C_{22}H_{30}N_3O_7^-$, ([M - H]$^-$) 448.2089, found 448.2087.



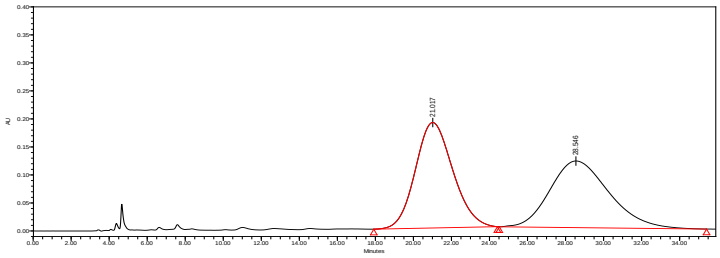
Peak	Retention Time	Area	% Area
1	16.649	40410203	50.93
2	23.158	38936066	49.07



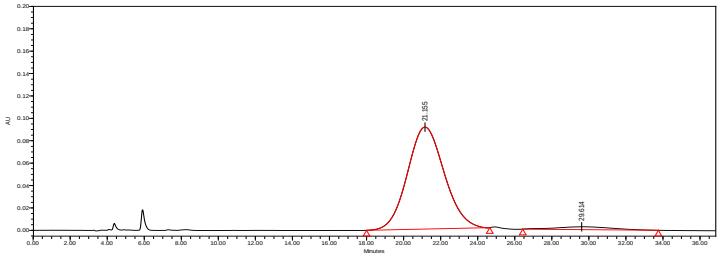
Peak	Retention Time	Area	% Area
1	16.868	26016202	94.67
2	24.421	1463697	5.33



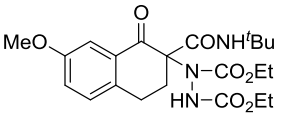
5da (Table 3, entry 8): $C_{22}H_{31}N_3O_7$ Yellow oil in 93% yield. $[\alpha]_D^{22.6}$ = −158.333 (c = 0.84 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.85-8.00 (m, 1H), 7.67 (s, 1H), 6.77-6.90 (m, 2H), 6.64-6.67 (m, 1H), 4.14-4.29 (m, 4H), 3.82-3.85 (m, 3H), 2.26-3.20 (m, 4H), 1.22 -1.36 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =191.9, 164.0, 163.4, 156.0, 145.7, 144.4, 130.6, 129.7, 127.0, 125.7, 113.4, 112.3, 63.4, 62.8, 62.5, 55.5, 51.6, 51.2, 28.4, 26.8, 14.3. Enantiomeric excesses HPLC (DAICEL CHIRALPAK ID, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =21.15 min, t_R minor = 29.61 min. calcd for $C_{22}H_{30}N_3O_7^-$, ([M - H]$^-$) 448.2089, found 448.2085.



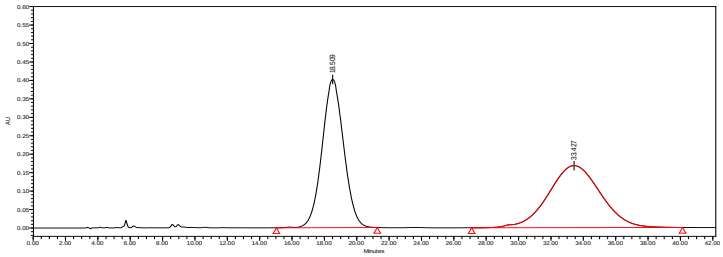
Peak	Retention Time	Area	% Area
1	21.017	25777288	50.09
2	28.546	25687578	49.91



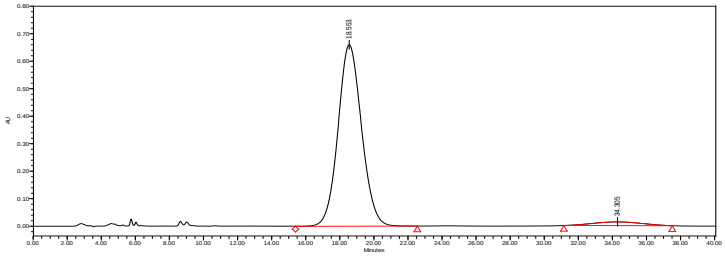
Peak	Retention Time	Area	% Area
1	21.155	12571420	96.08
2	29.614	512278	3.92



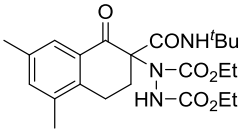
5ea (Table 3, entry 4): $C_{22}H_{31}N_3O_7$ Yellow oil in 92% yield. $[\alpha]_D^{22.6}$ = −172.205 (c = 0.83 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.72 (s, 1H), 7.35-7.49 (m, 1H), 6.79-7.15 (m, 3H), 6.64-6.67 (m, 1H), 4.13-4.31 (m, 4H), 3.76-3.81 (m, 3H), 2.25-3.11 (m, 4H), 1.23 -1.37 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =193.5, 168.5, 159.6, 158.2, 157.2, 156.0, 135.6, 134.3, 129.9, 129.4, 121.5, 109.3, 74.9, 64.1, 63.5, 62.9, 55.4, 51.0, 33.1, 28.2, 25.8, 14.5. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IE, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =18.55 min, t_R minor = 34.30 min. calcd for $C_{22}H_{30}N_3O_7^-$, ([M - H]$^-$) 448.2089, found 448.2079.



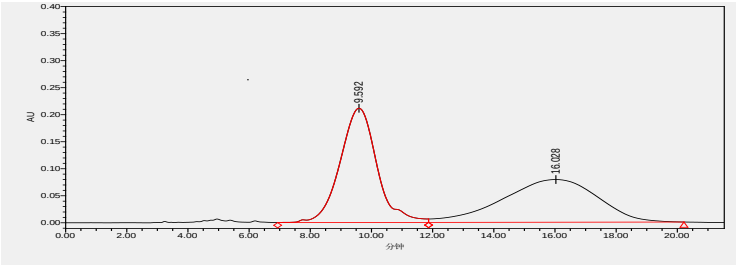
Peak	Retention Time	Area	% Area
1	18.509	37486633	49.85
2	33.427	37711376	50.15



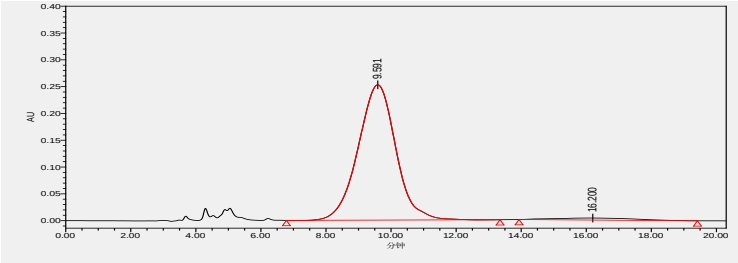
Peak	Retention Time	Area	% Area
1	18.553	63363853	95.92
2	34.305	2694619	4.08



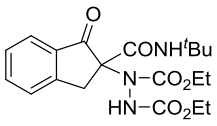
5fa (Table 3, entry 10): $C_{23}H_{33}N_3O_6$ Yellow oil in 89% yield. $[\alpha]_D^{22.6}$ = −134.250 (c = 0.80 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.59-7.69 (m, 2H), 7.13-7.18 (m, 1H), 6.62-6.70 (m, 1H), 6.64-6.67 (m, 1H), 4.14-4.33 (m, 4H), 2.10-3.16 (m, 10H), 1.22 -1.38 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =194.1, 159.6, 135.6, 137.4, 135.5, 125.4, 74.4, 63.0, 50.7, 32.1, 28.2, 21.0, 19.1, 14.3. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 85/15, flow rate 1.0 mL/min, detection at 254 nm): t_R major =9.59 min, t_R minor = 16.20 min. calcd for $C_{23}H_{32}N_3O_6^-$, ([M - H]$^-$) 446.2296, found 446.2286.



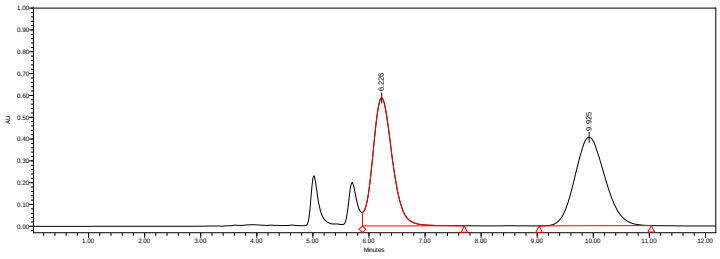
Peak	Retention Time	Area	% Area
1	9.592	17692705	50.80
2	16.028	17135064	49.20



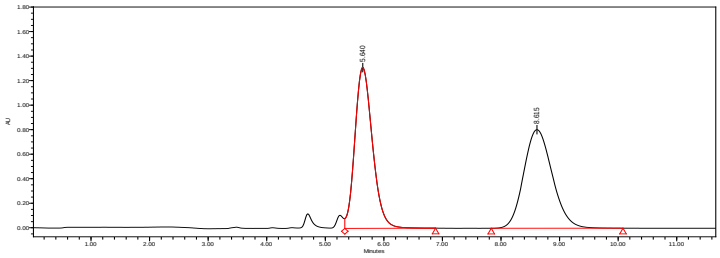
Peak	Retention Time	Area	% Area
1	9.591	20298166	96.90
2	16.200	648571	3.10



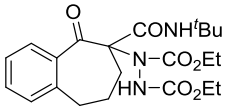
5ga (Table 3, entry 11): $C_{20}H_{27}N_3O_6$ Yellow oil in 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.73-7.76 (m, 1H), 7.59-7.63 (m, 1H), 7.45-7.47 (m, 1H), 7.34-7.38 (m, 1H), 6.65-7.27 (m, 2H), 4.43- 4.48 (m, 1H), 4.13- 4.34 (m, 4H), 3.23-3.28 (m, 1H), 1.21 -1.35 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =155.4, 153.5, 136.2, 133.7, 127.8, 126.4, 125.1, 63.6, 62.8, 51.7, 28.5, 14.5, 14.4, 14.3. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IA, hexane/ 2-propanol = 80/20, flow rate 1.0 mL/min, detection at 254 nm): t_R 1 =5.64 min, t_R 2 = 8.61 min. calcd for $C_{20}H_{26}N_3O_6^-$, ([M - H]$^-$) 404.1827, found 404.1828.



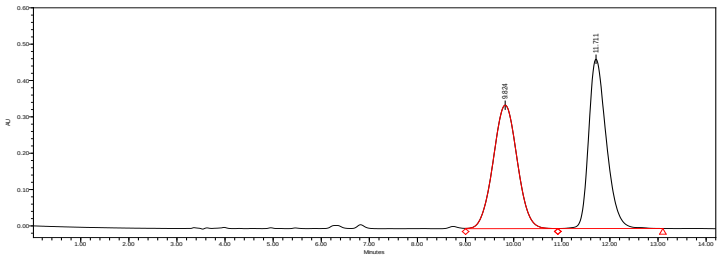
Peak	Retention Time	Area	% Area
1	6.226	14228190	48.51
2	9.925	15101248	51.49



Peak	Retention Time	Area	% Area
1	5.640	27357944	50.12
2	8.615	27232193	49.88



5ha (Table 3, entry 12): $C_{22}H_{31}N_3O_6$ Yellow oil in 87% yield. $[\alpha]_D^{22.6}$ = −18.375 (c = 0.75 in CH_2Cl_2). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C, TMS): δ = 7.71-8.20 (m, 1H), 7.24-7.39 (m, 2H), 7.09-7.27 (m, 2H), 6.79-6.84 (m, 1H), 4.04- 4.31 (m, 4H), 1.84- 3.91 (m, 6H), 0.89 -1.39 (m, 15H). ^{13}C NMR (100 MHz, $CDCl_3$, 25 ^{o}C): δ =170.8, 156.0, 139.9, 137.8, 136.1, 130.3, 128.4, 127.9, 126.6, 125.8, 100.0, 64.3, 63.5, 61.7, 51.5, 50.7, 33.9, 28.5, 23.6, 22.7, 14.3. Enantiomeric excesses HPLC (DAICEL CHIRALPAK IE, hexane/ 2-propanol = 70/30, flow rate 1.0 mL/min, detection at 254 nm): t_R major =9.77 min, t_R minor = 11.71 min. calcd for $C_{22}H_{30}N_3O_6^-$, ([M - H]$^-$) 432.2140, found 432.2156.



Peak	Retention Time	Area	% Area
1	9.824	11828130	49.87
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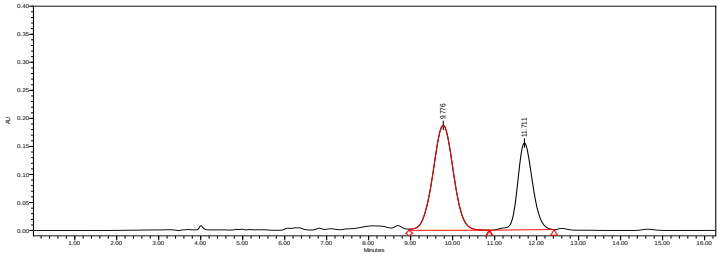


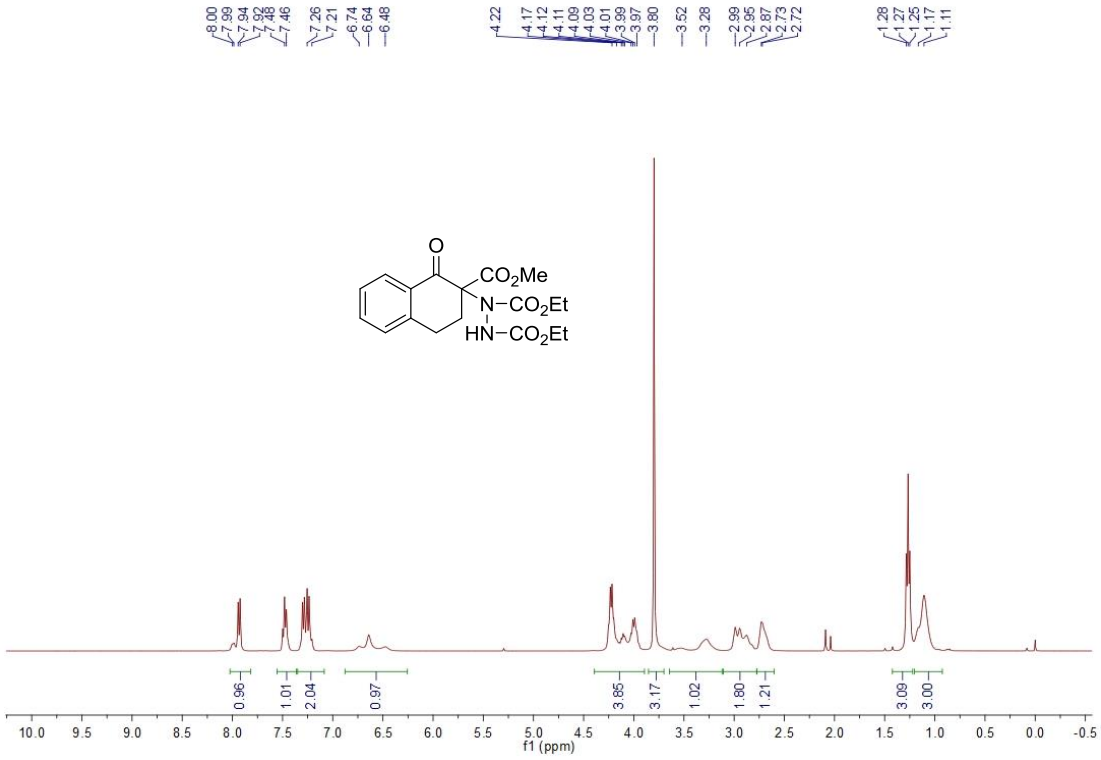
Peak	Retention Time	Area	% Area
1	9.776	6377654	62.50
2	11.711	3826734	37.50

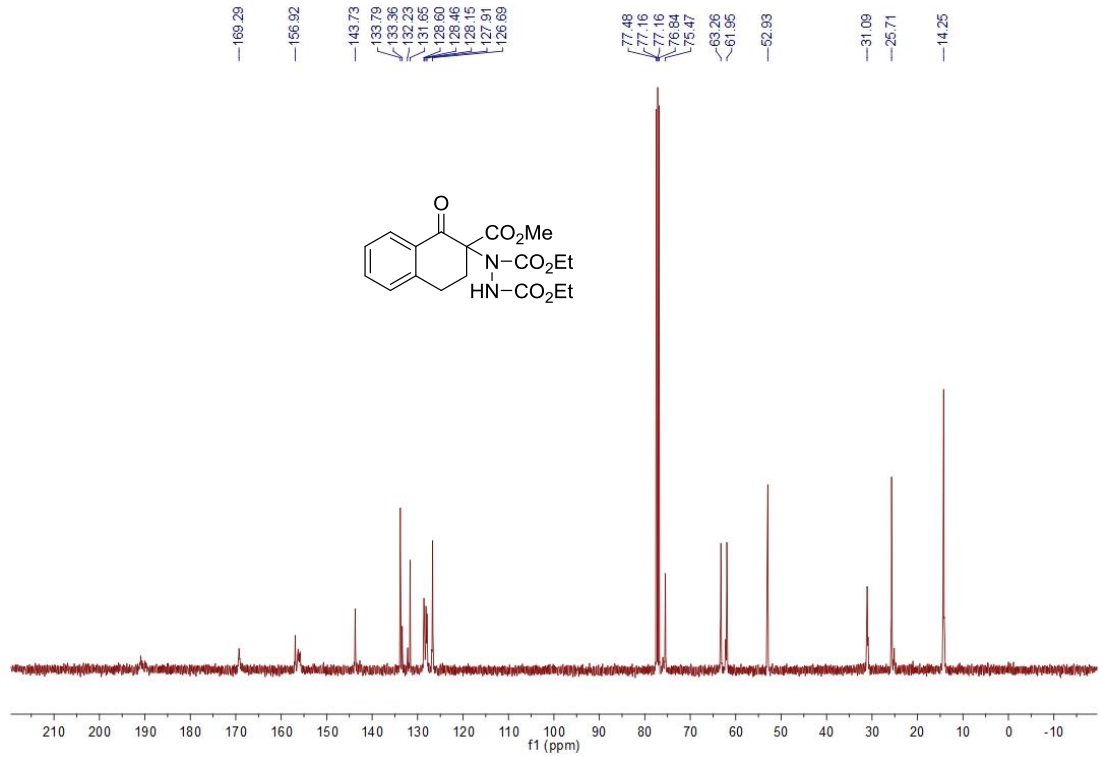
5. References:

1. C. K. Yin, W. D. Cao, L. L. Lin, X. H. Liu, X. M. Feng, *Adv. Synth. Cata.* **2013**, *355*, 1924-1930.

2. (a) Y. H. Wen, X. Huang, J. L. Huang, Y. Xiong, B. Qin, X. M. Feng, Synlett 2005, 2445-2448.
(b) J. L. Huang, J. Wang, X. H. Chen, Y. H. Wen, X. H. Liu, X. M. Feng, Adv. Synth. Catal. **2008**, 350, 287-294.

6. Copy of ^{1}H NMR and ^{13}C NMR spectra for products:

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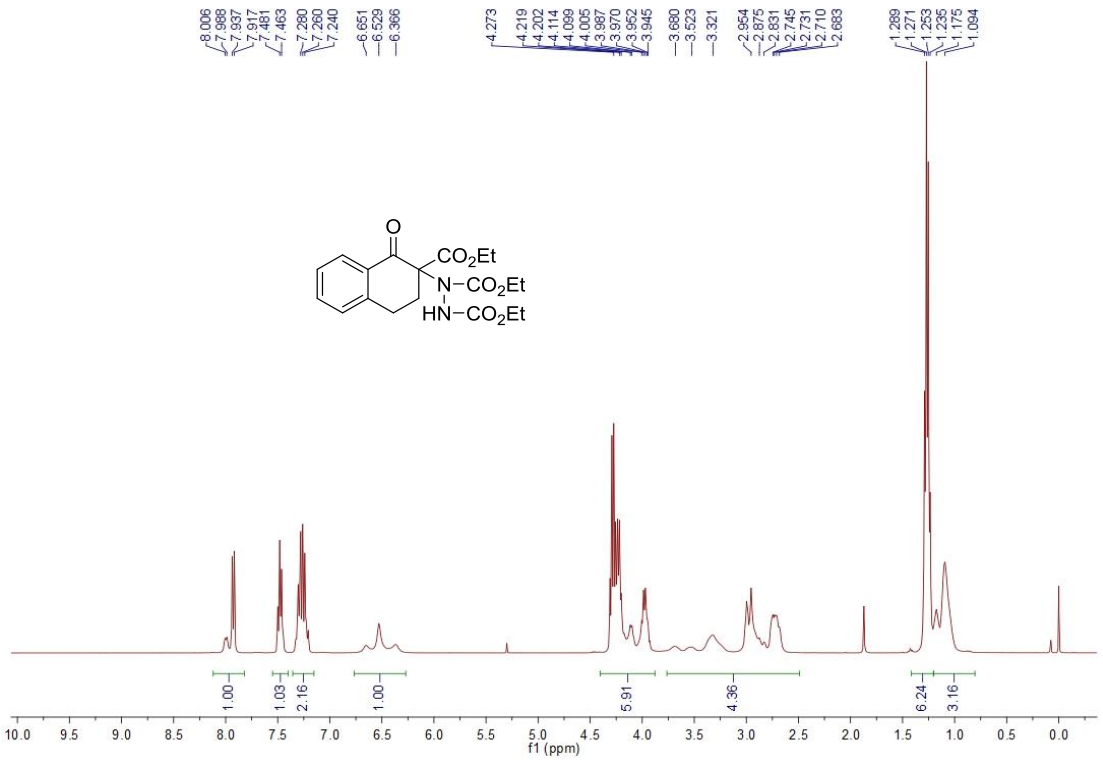
f1 (ppm)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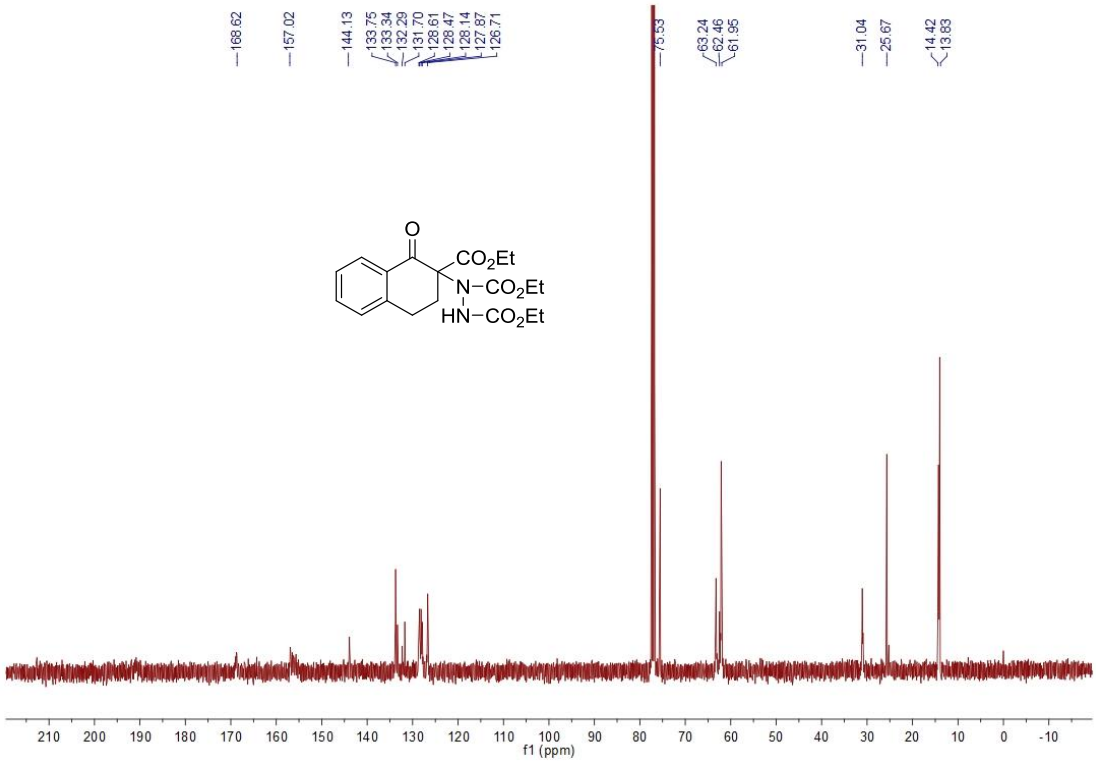
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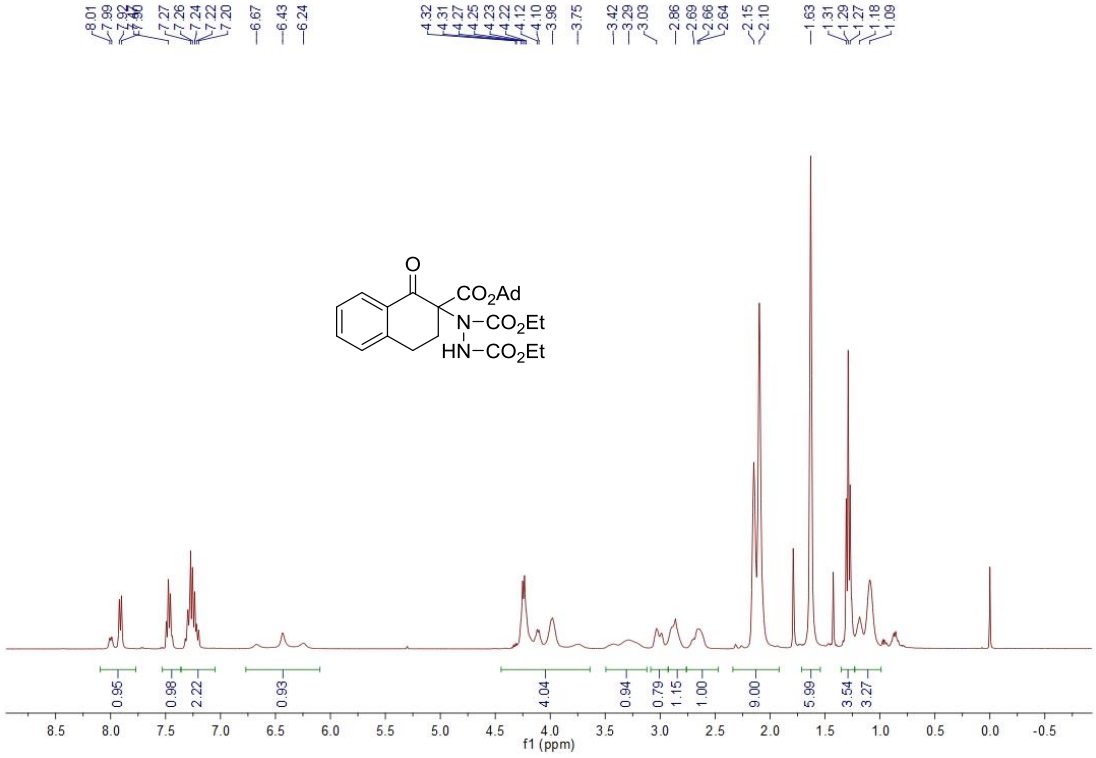
HN−CO2Et
f1 (ppm)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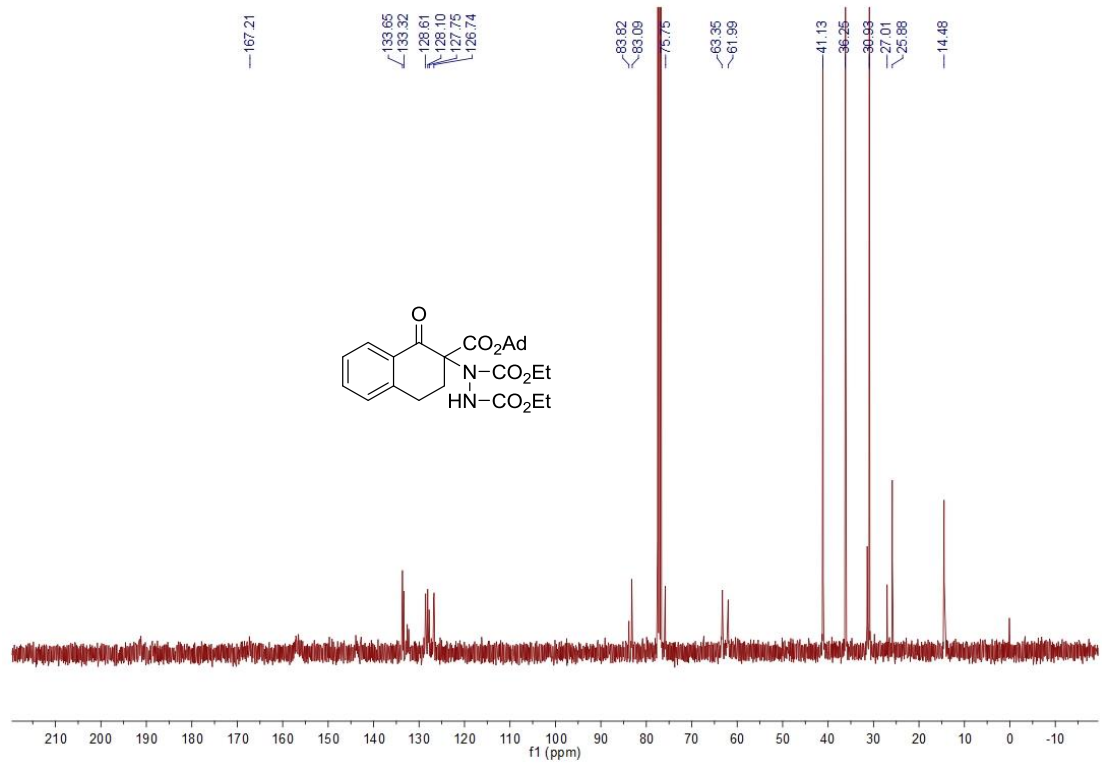
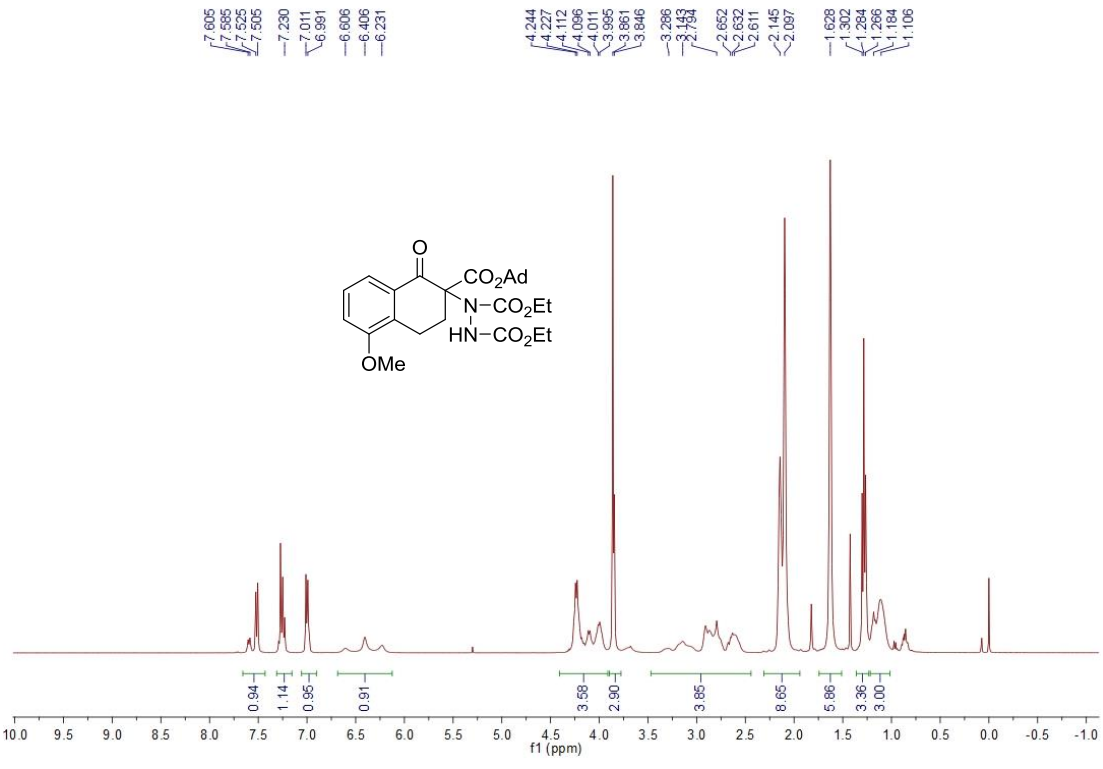
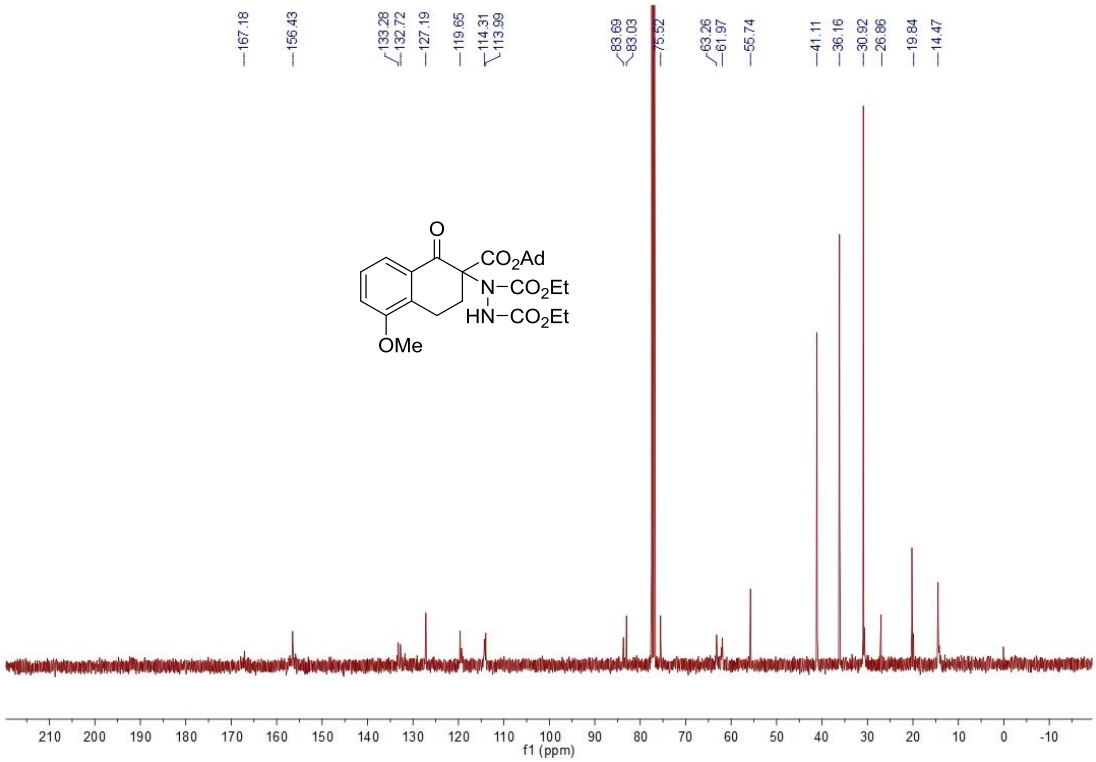
7.99
7.27
7.26
7.24
7.22
7.20
6.67
6.43
6.24
4.32
4.31
4.27
4.25
4.23
4.22
4.12
4.10
3.98
3.75
3.42
3.29
3.03
2.86
2.69
2.66
2.64
2.15
2.10
1.63
1.31
1.29
1.27
1.18
1.09
O
CO2Ad
N−CO2Et
HN−CO2Et
0.95
0.98
2.22
0.93
4.04
0.94
0.79
1.15
1.00
9.00
5.99
3.54
3.27
f1 (ppm)


167.21
133.65
133.32
128.61
128.10
127.75
126.74
83.82
83.09
75.75
63.35
61.99
41.13
36.25
30.93
27.01
25.88
14.48
O
CO2Ad
N−CO2Et
HN−CO2Et
f1 (ppm)


7.605
7.585
7.525
7.505
7.230
7.011
6.991
6.606
6.406
6.231
4.244
4.227
4.112
4.096
4.011
3.995
3.861
3.846
3.286
3.143
2.794
2.652
2.632
2.611
2.145
2.097
1.628
1.302
1.284
1.266
1.184
1.106
O
CO2Ad
N−CO2Et
HN−CO2Et
OMe
0.94
1.14
0.95
0.91
3.58
2.90
3.85
8.65
5.86
3.36
3.00
f1 (ppm)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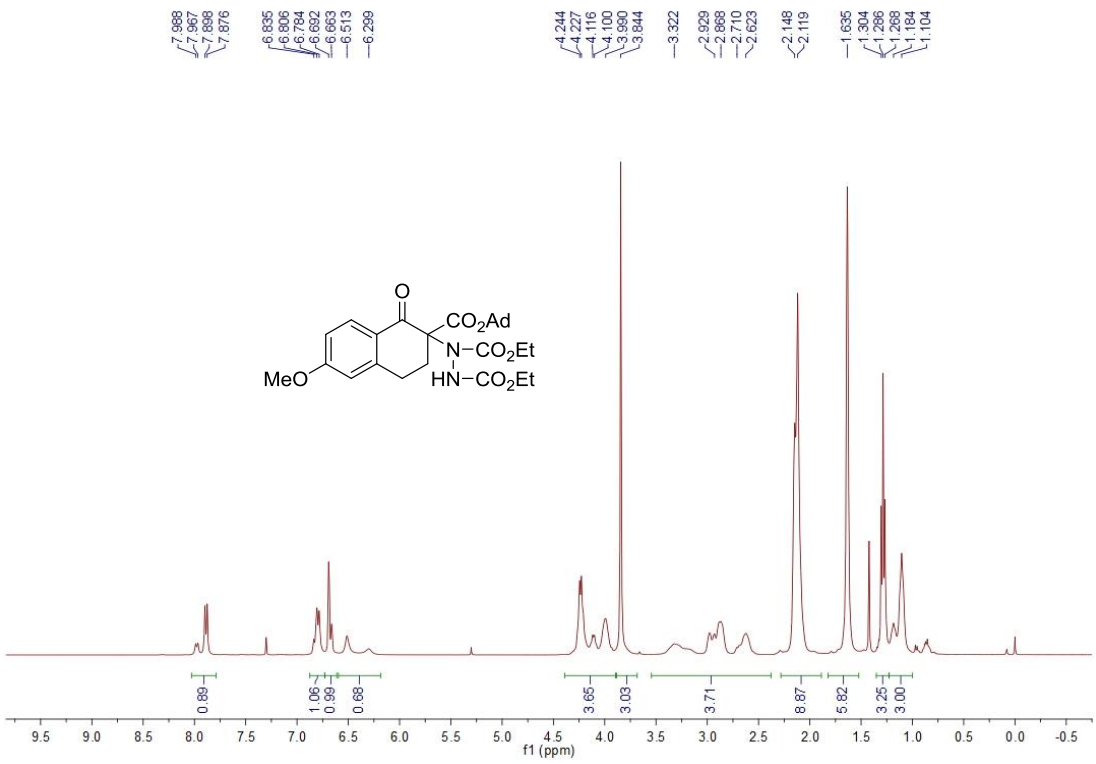
156.43
133.28
132.72
127.19
119.65
114.31
113.99
83.69
83.03
75.52
63.26
61.97
55.74
41.11
36.16
30.92
26.86
19.84
14.47
O
CO2Ad
N−CO2Et
HN−CO2Et
OMe
f1 (ppm)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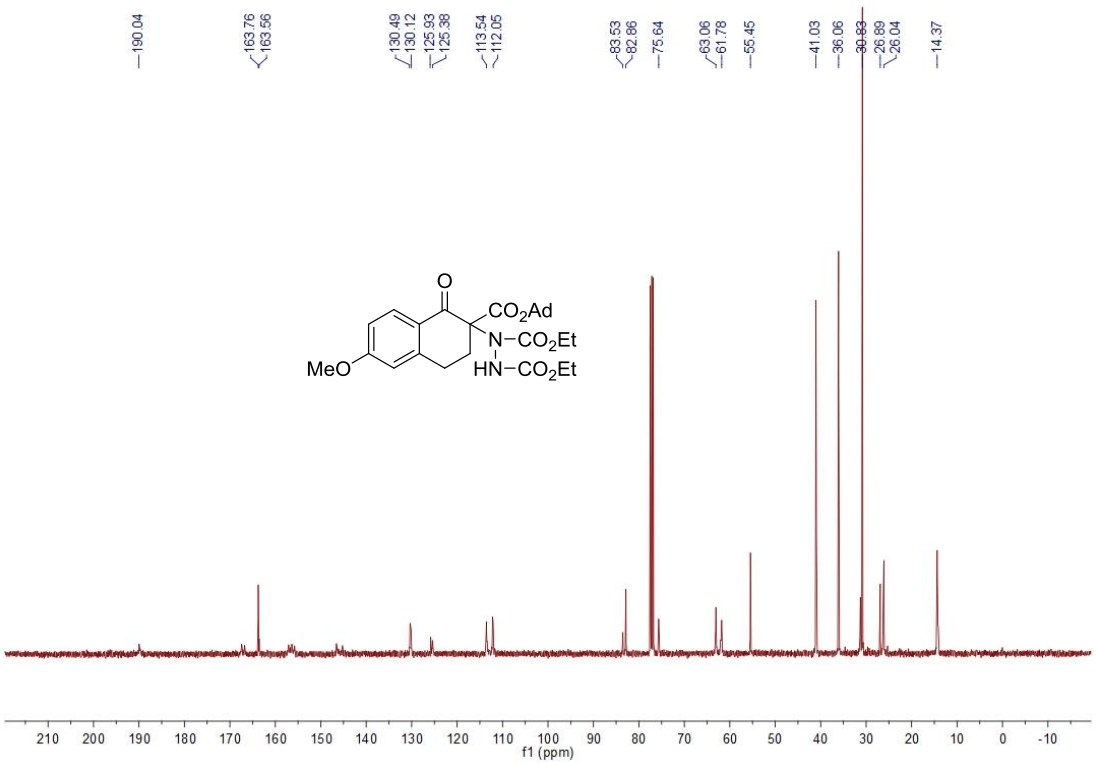
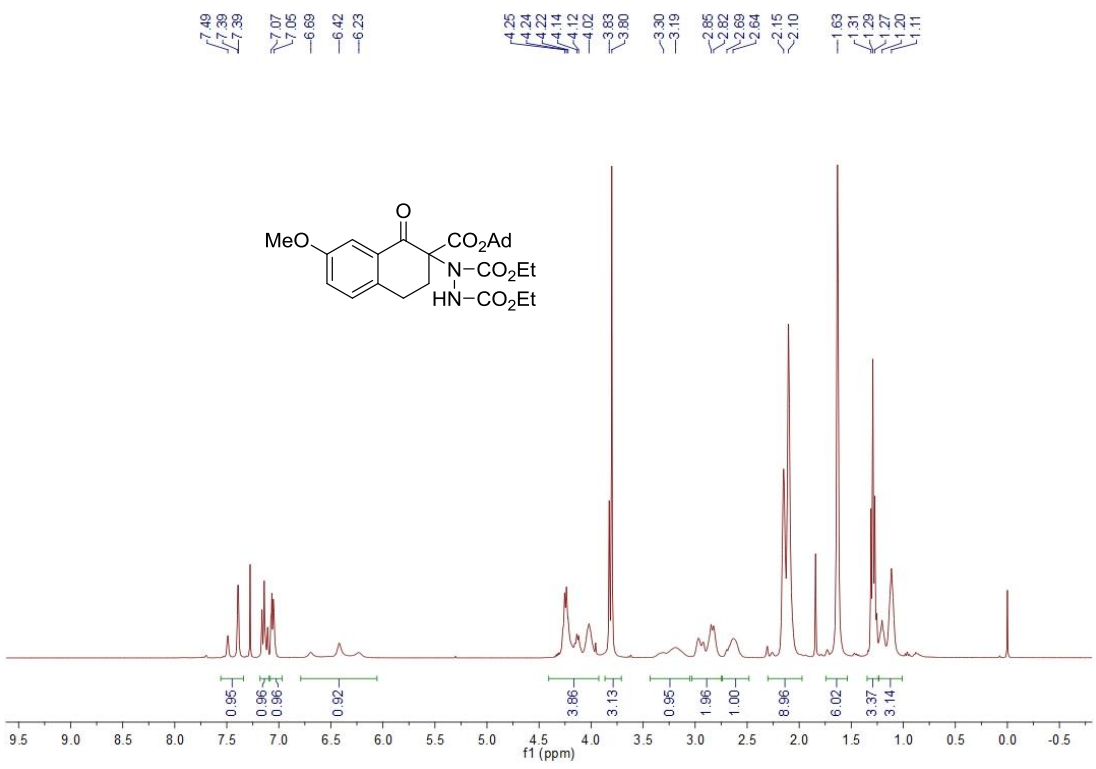
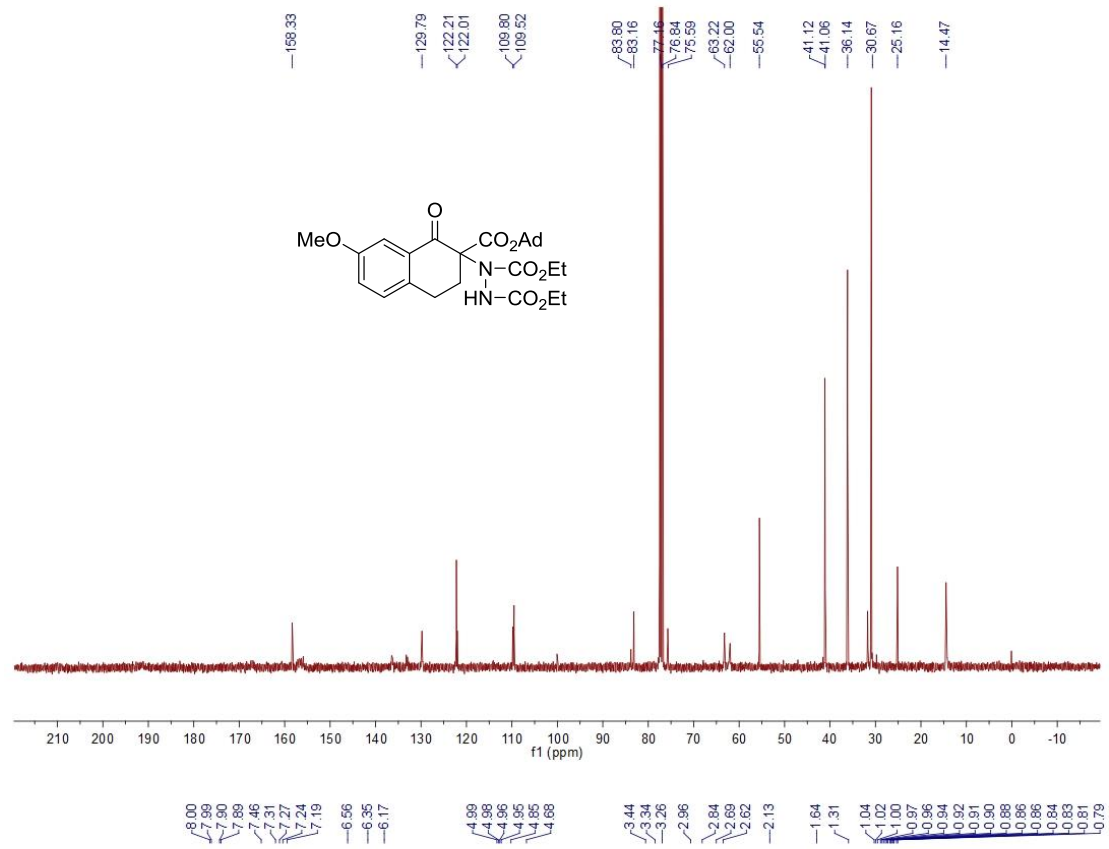
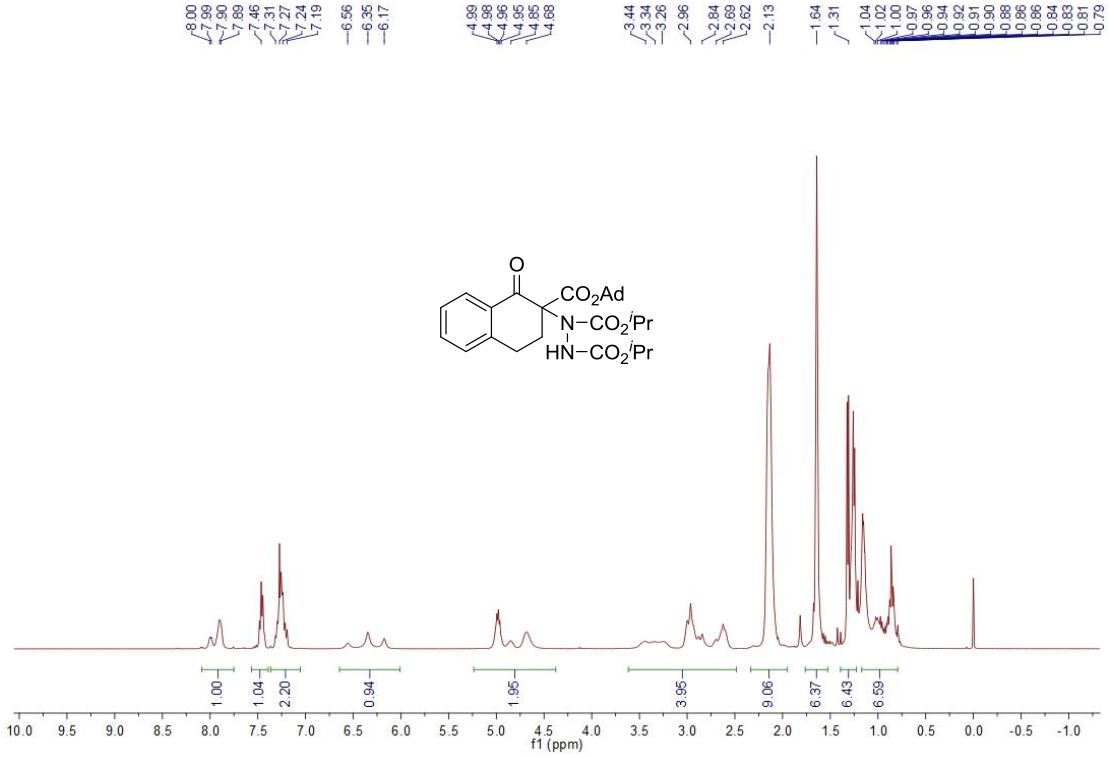
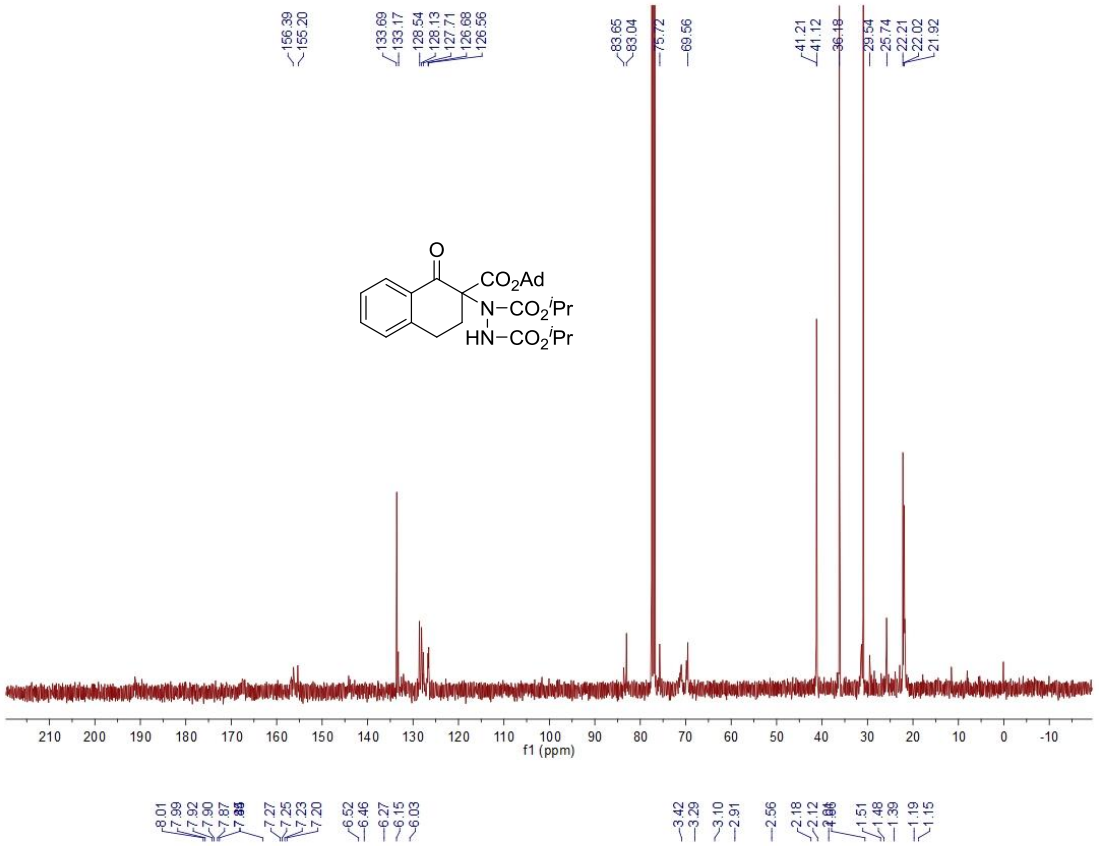
7.967
7.898
7.876
6.835
6.806
6.784
6.692
6.663
6.513
6.299
4.244
4.227
4.116
4.100
3.990
3.844
3.322
2.929
2.868
2.710
2.623
2.148
2.119
1.635
1.304
1.286
1.268
1.184
1.104
O
CO2Ad
N−CO2Et
HN−CO2Et
MeO
0.89
1.06
0.99
0.68
3.65
3.03
3.71
8.87
5.82
3.25
3.00
f1 (ppm)

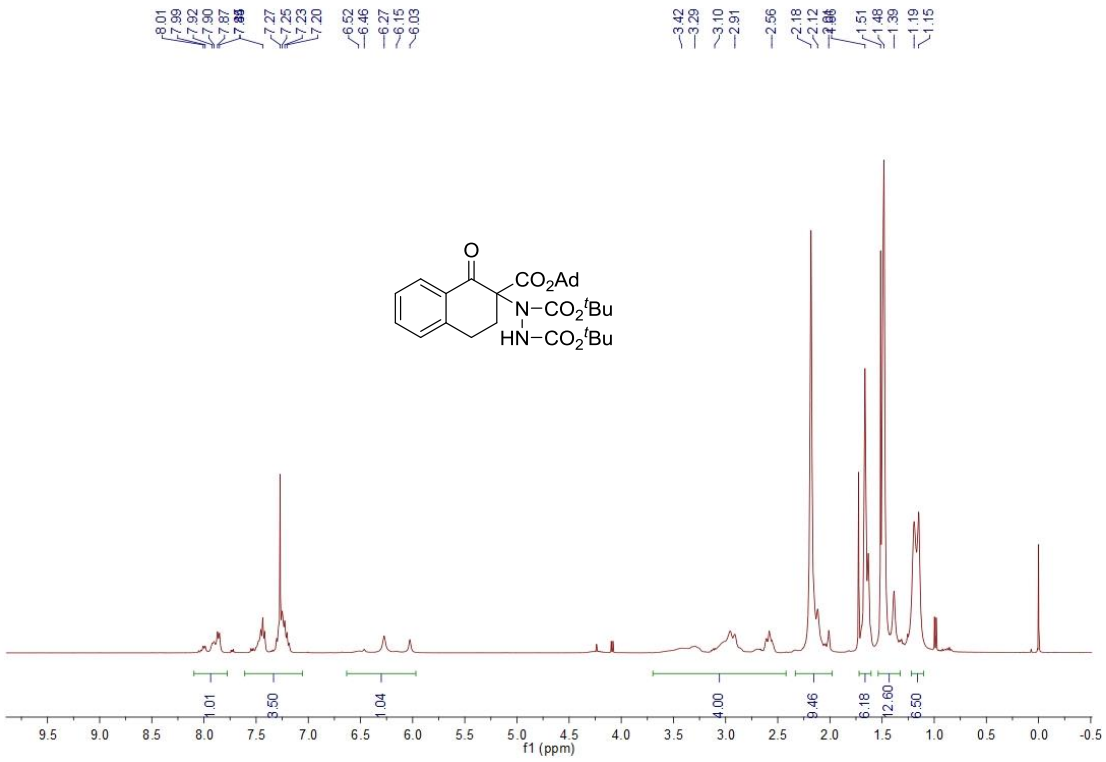

190.04
163.76
163.56
130.49
130.12
125.93
125.38
113.54
112.05
83.53
82.86
75.64
63.06
61.78
55.45
41.03
36.06
30.83
26.89
26.04
14.37
O
CO2Ad
N−CO2Et
HN−CO2Et
MeO
f1 (ppm)

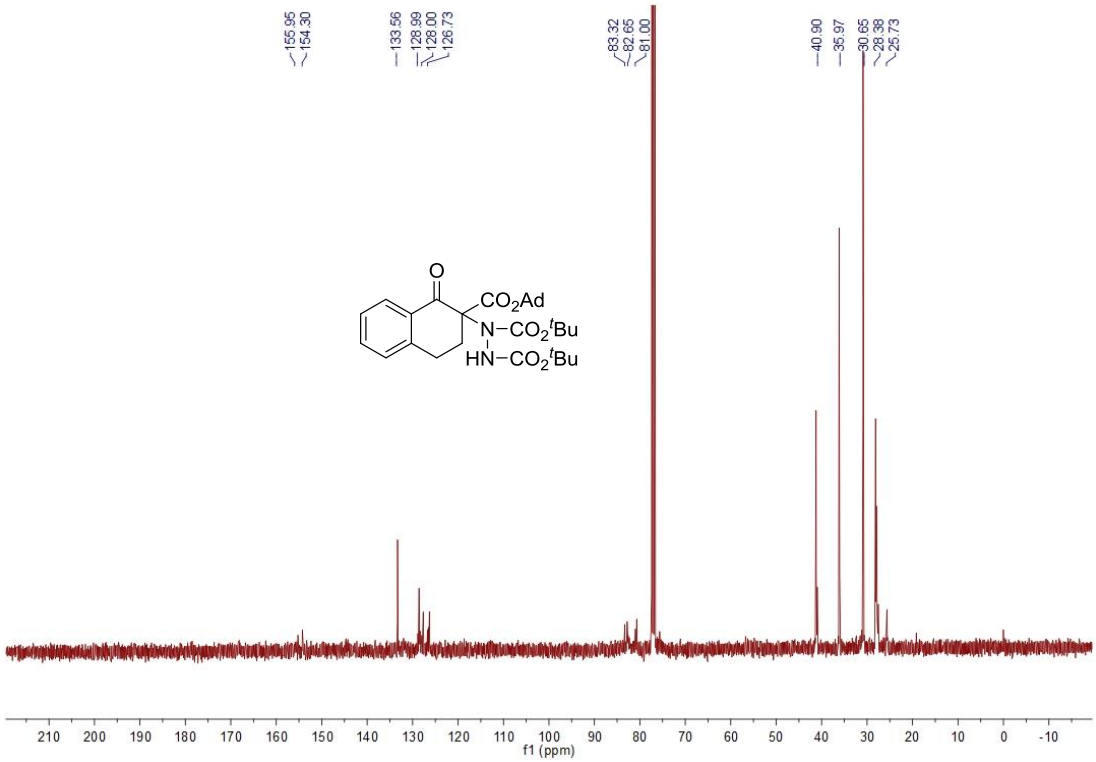

7.49
7.39
7.39
7.07
7.05
6.69
6.42
6.23
4.25
4.24
4.22
4.14
4.12
4.02
3.83
3.80
3.30
3.19
2.85
2.82
2.69
2.64
2.15
2.10
1.63
1.31
1.29
1.27
1.20
1.11
O
MeO
CO2Ad
N−CO2Et
HN−CO2Et
0.95
0.96
0.96
0.92
3.86
3.13
0.95
1.96
1.00
8.96
6.02
3.37
3.14
f1 (ppm)

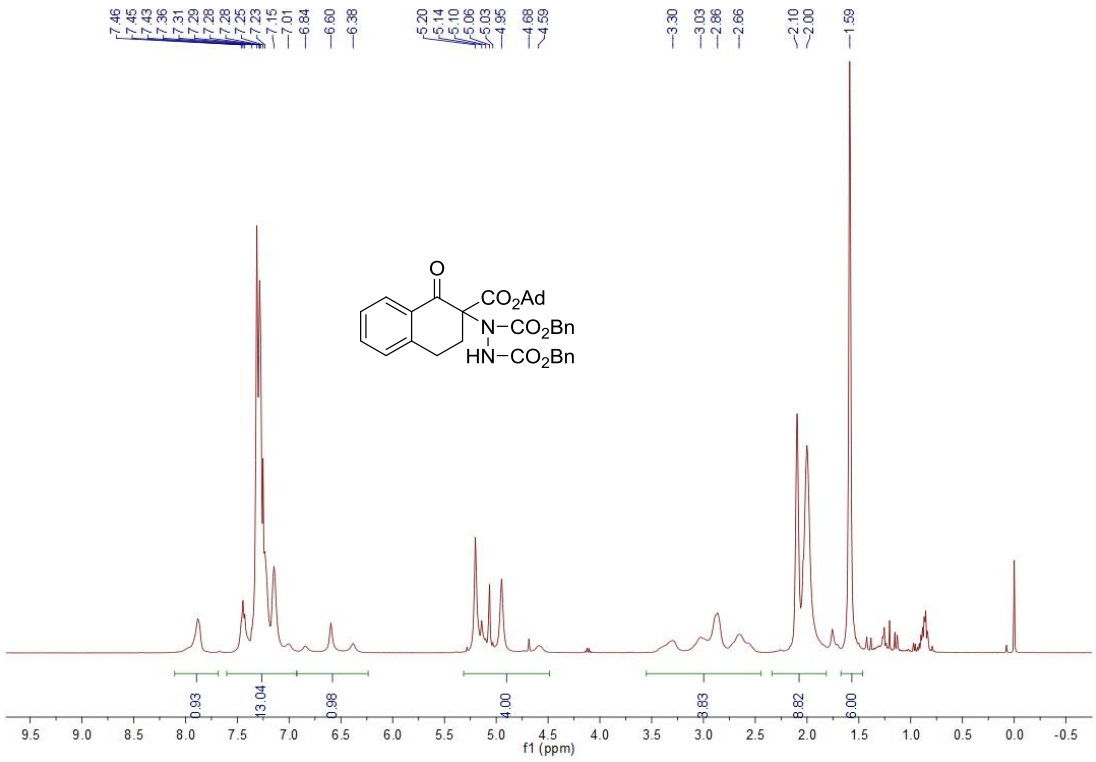

158.33
129.79
122.21
122.01
109.80
109.52
83.80
83.16
77.16
76.84
75.59
63.22
62.00
55.54
41.12
41.06
36.14
30.67
25.16
14.47
MeO
CO2Ad
N−CO2Et
HN−CO2Et
f1 (ppm)

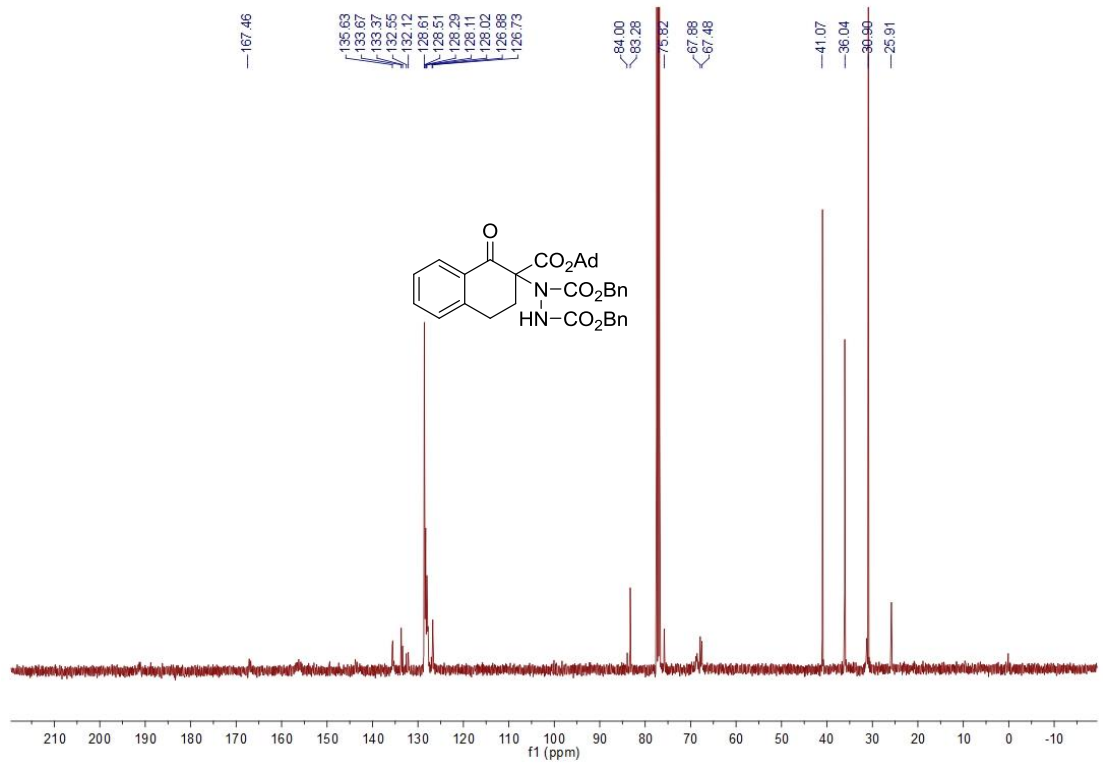

CO2Ad
N−CO2iPr
HN−CO2iPr
1.00
1.04
2.20
0.94
1.95
3.95
9.06
6.37
6.43
6.59
f1 (ppm)


156.39
155.20
133.69
133.17
128.54
128.13
127.71
126.68
126.56
83.65
83.04
75.72
69.56
41.21
41.12
36.18
29.54
25.74
22.21
22.02
21.92
O
CO2Ad
N–CO2iPr
HN–CO2iPr
f1 (ppm)


8.01
7.99
7.92
7.90
7.87
7.27
7.25
7.23
7.20
6.52
6.46
6.27
6.15
6.03
3.42
3.29
3.10
2.91
2.56
2.18
2.12
1.51
1.48
1.39
1.19
1.15
O
CO2Ad
N–CO2tBu
HN–CO2tBu
1.01
3.50
1.04
4.00
9.46
6.18
12.60
6.50
f1 (ppm)


155.95
154.30
133.56
128.99
128.00
126.73
83.32
82.65
81.00
40.90
35.97
30.65
28.38
25.73
O
CO2Ad
N−CO2tBu
HN−CO2tBu
f1 (ppm)


7.46
7.45
7.43
7.36
7.31
7.29
7.28
7.25
7.23
7.15
7.01
6.84
6.60
6.38
5.20
5.14
5.10
5.06
5.03
4.95
4.68
4.59
3.30
3.03
2.86
2.66
2.10
2.00
1.59
O
CO2Ad
N−CO2Bn
HN−CO2Bn
0.93
13.04
0.98
4.00
8.83
8.82
6.00
f1 (ppm)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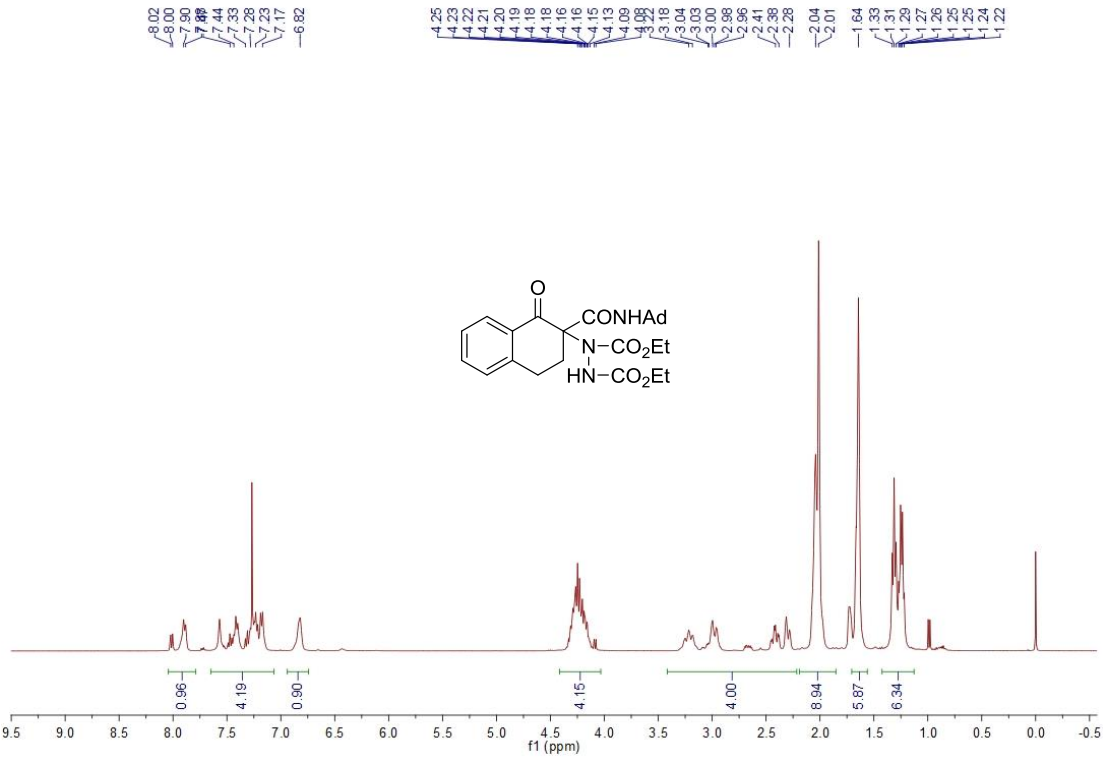
135.63
133.67
133.37
132.55
132.12
128.61
128.51
128.29
128.11
128.02
126.88
126.73
84.00
83.28
75.82
67.88
67.48
41.07
36.04
30.90
25.91
O
CO2Ad
N−CO2Bn
HN−CO2Bn
f1 (ppm)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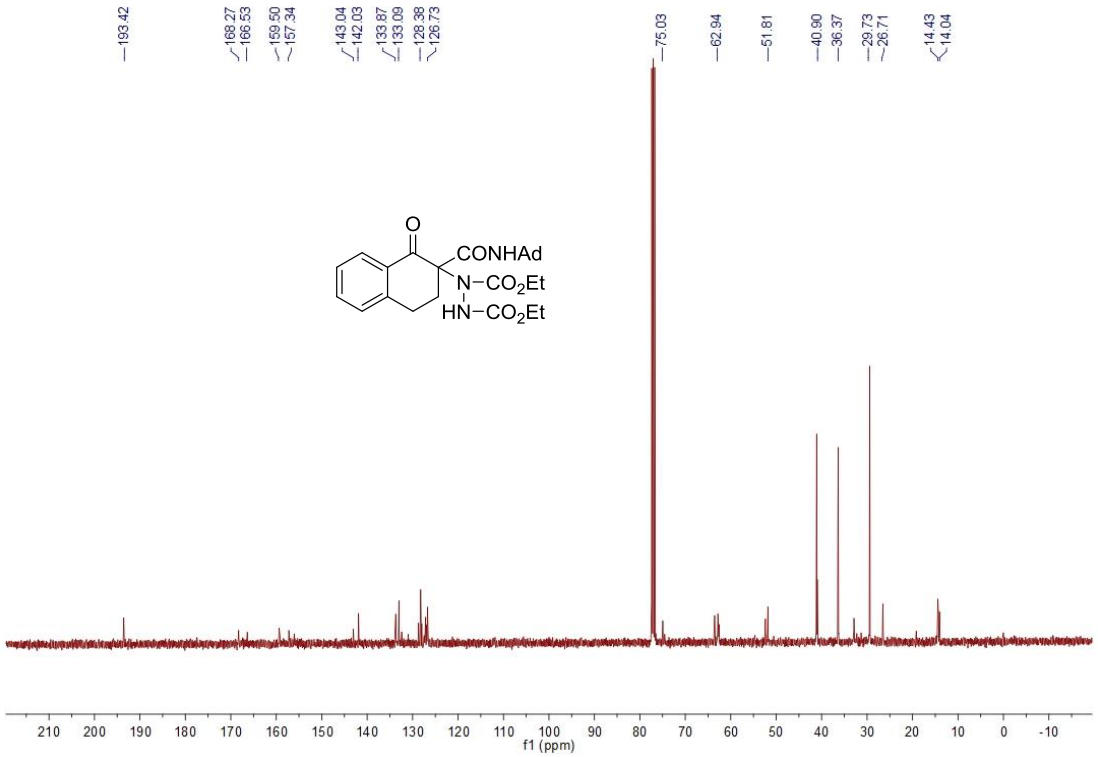
8.00
7.90
7.44
7.33
7.28
7.23
7.17
6.82
4.25
4.23
4.22
4.21
4.20
4.19
4.18
4.16
4.15
4.13
4.09
3.18
3.04
3.03
3.00
2.98
2.96
2.41
2.38
2.28
2.04
2.01
1.64
1.33
1.31
1.29
1.27
1.26
1.25
1.25
1.24
1.22
O
CONHAd
N−CO2Et
HN−CO2Et
0.96
4.19
0.90
4.15
4.00
8.94
5.87
6.34
f1 (ppm)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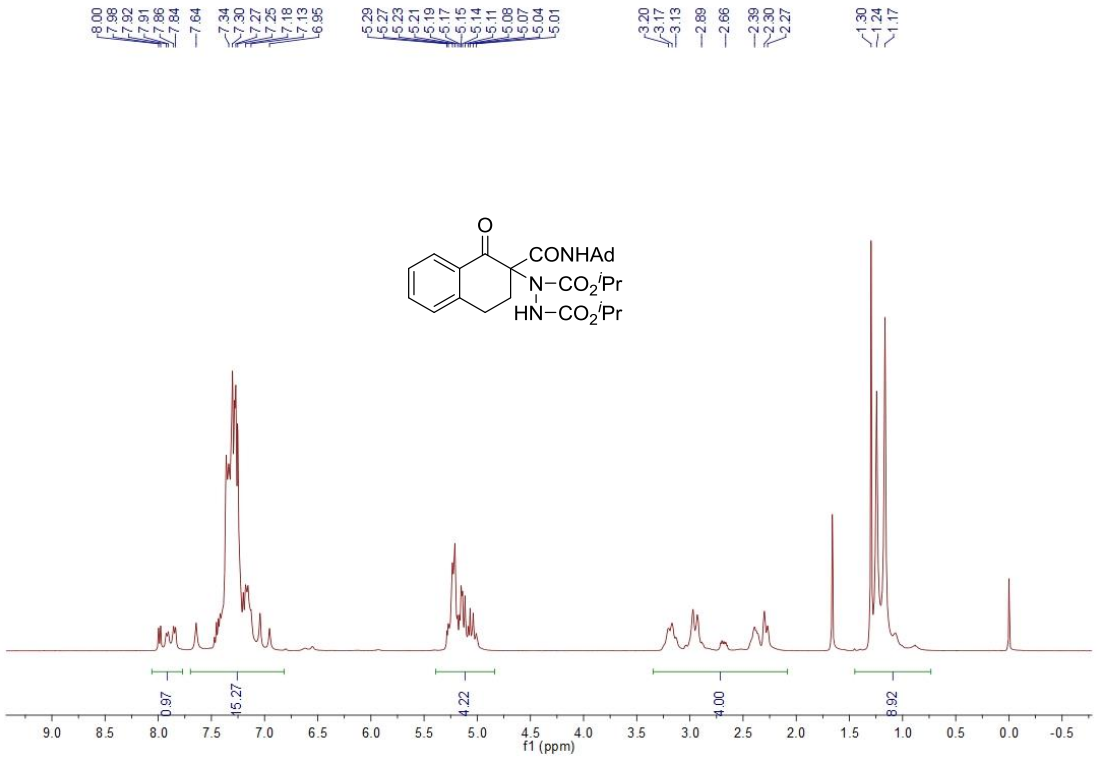
169.27
166.53
159.50
157.34
143.04
142.03
133.87
133.09
128.38
126.73
75.03
62.94
51.81
40.90
36.37
29.73
26.71
14.43
14.04
O
CONHAd
N−CO2Et
HN−CO2Et
f1 (ppm)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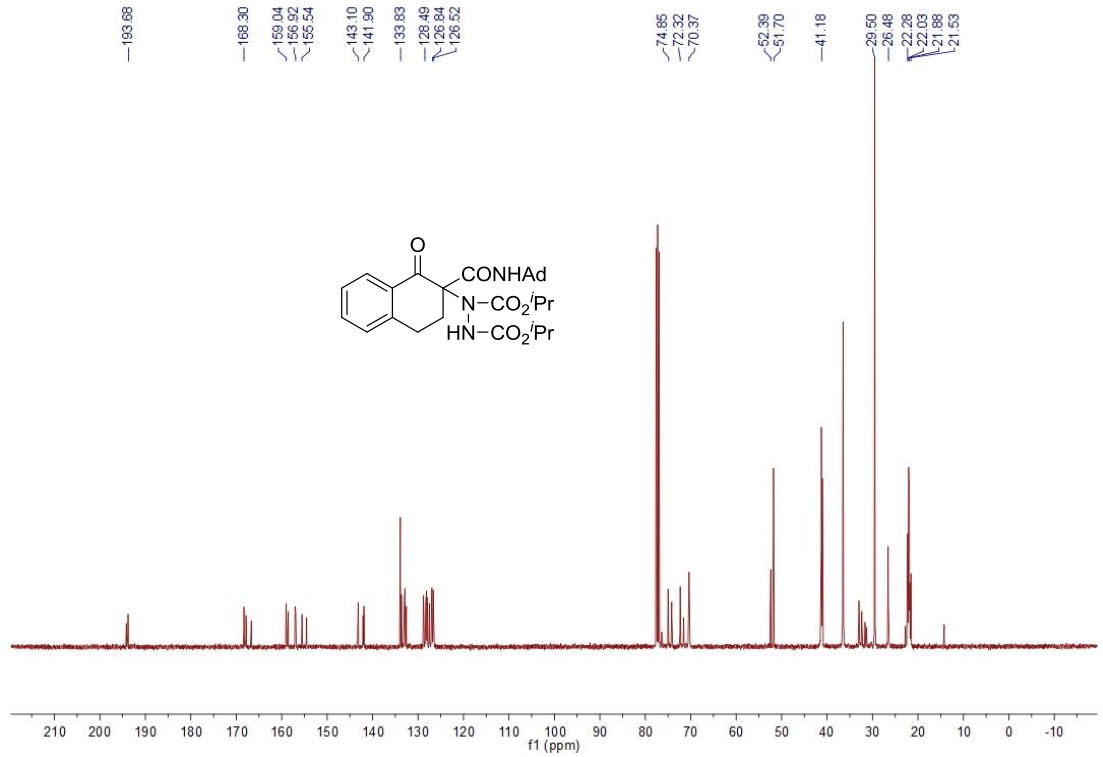
7.98
7.92
7.91
7.86
7.84
7.64
7.34
7.30
7.27
7.25
7.18
7.13
6.95
5.29
5.27
5.23
5.21
5.19
5.17
5.15
5.14
5.11
5.08
5.07
5.04
5.01
3.20
3.17
3.13
2.89
2.66
2.39
2.30
2.27
1.30
1.24
1.17
O
CONHAd
N−CO2iPr
HN−CO2iPr
0.97
15.27
4.22
4.00
8.92
f1 (ppm)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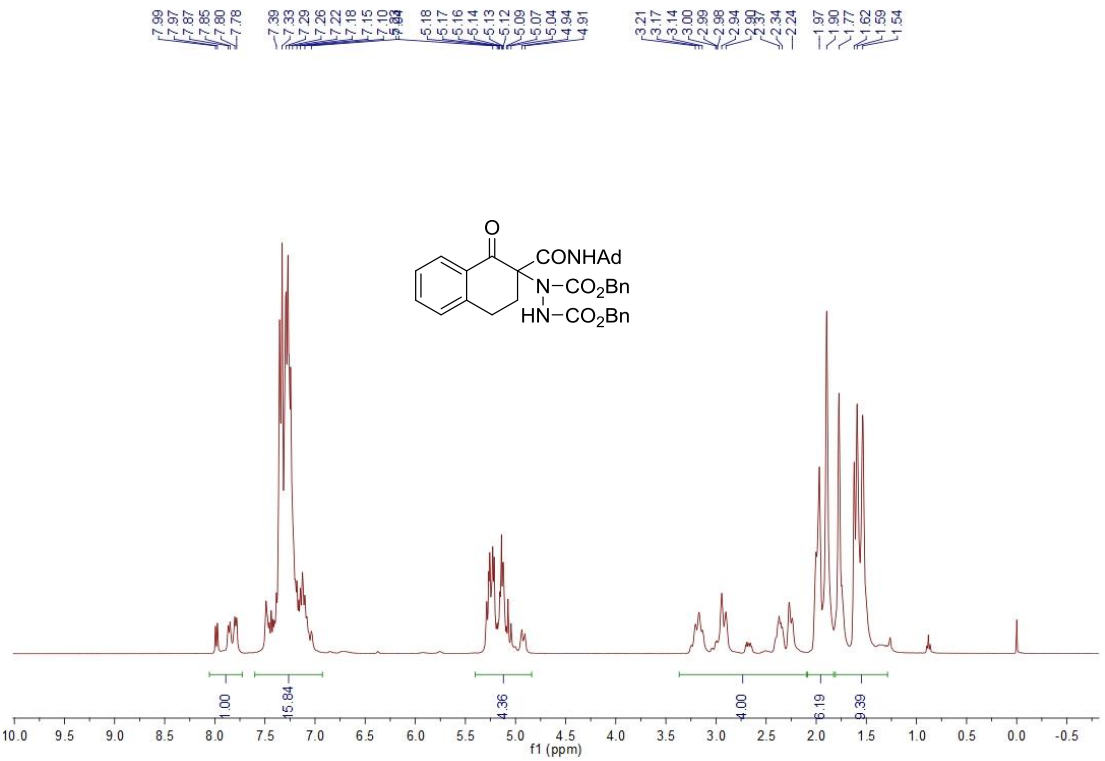
159.04
156.92
155.54
143.10
141.90
133.83
128.49
126.84
126.52
74.85
72.32
70.37
52.39
51.70
41.18
29.50
26.48
22.28
22.03
21.88
21.53
O
CONHAd
N−CO2iPr
HN−CO2iPr
f1 (ppm)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7.87
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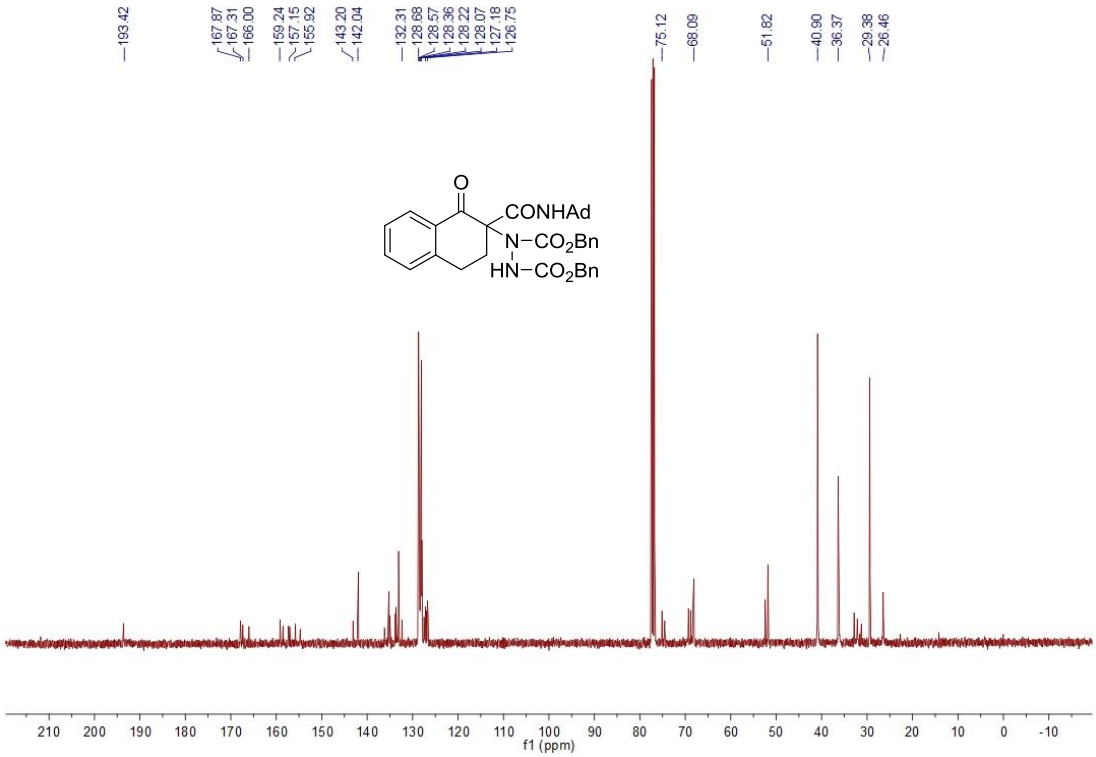
7.78
7.39
7.33
7.29
7.26
7.22
7.18
7.15
5.18
5.17
5.16
5.14
5.13
5.12
5.09
5.07
5.04
4.94
4.91
3.21
3.17
3.14
3.00
2.99
2.98
2.94
2.90
2.37
2.34
2.24
1.97
1.90
1.77
1.62
1.59
1.54
O
CONHAd
N−CO2Bn
HN−CO2Bn
1.00
15.84
4.36
4.00
6.19
9.39
f1 (ppm)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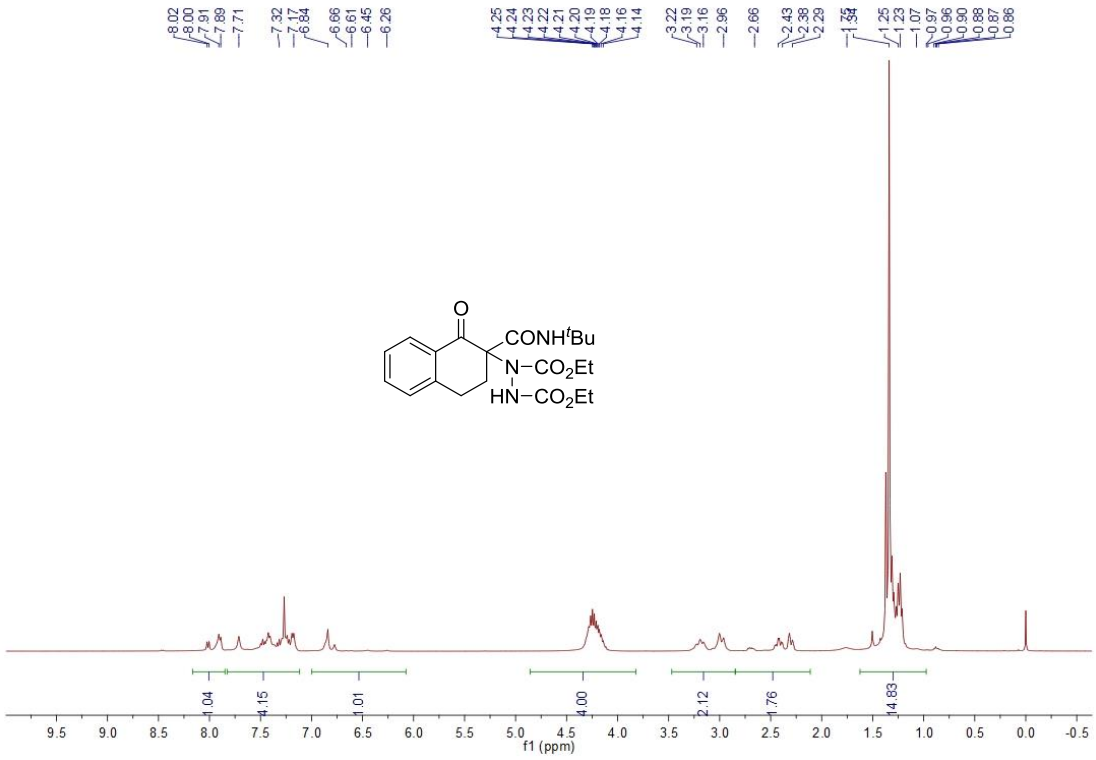
167.87
167.31
166.00
159.24
157.15
155.92
143.20
142.04
132.31
128.68
128.57
128.36
128.22
128.07
127.18
126.75
75.12
68.09
51.82
40.90
36.37
29.38
26.46
O
CONHAd
N−CO2Bn
HN−CO2Bn
f1 (ppm)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8.00
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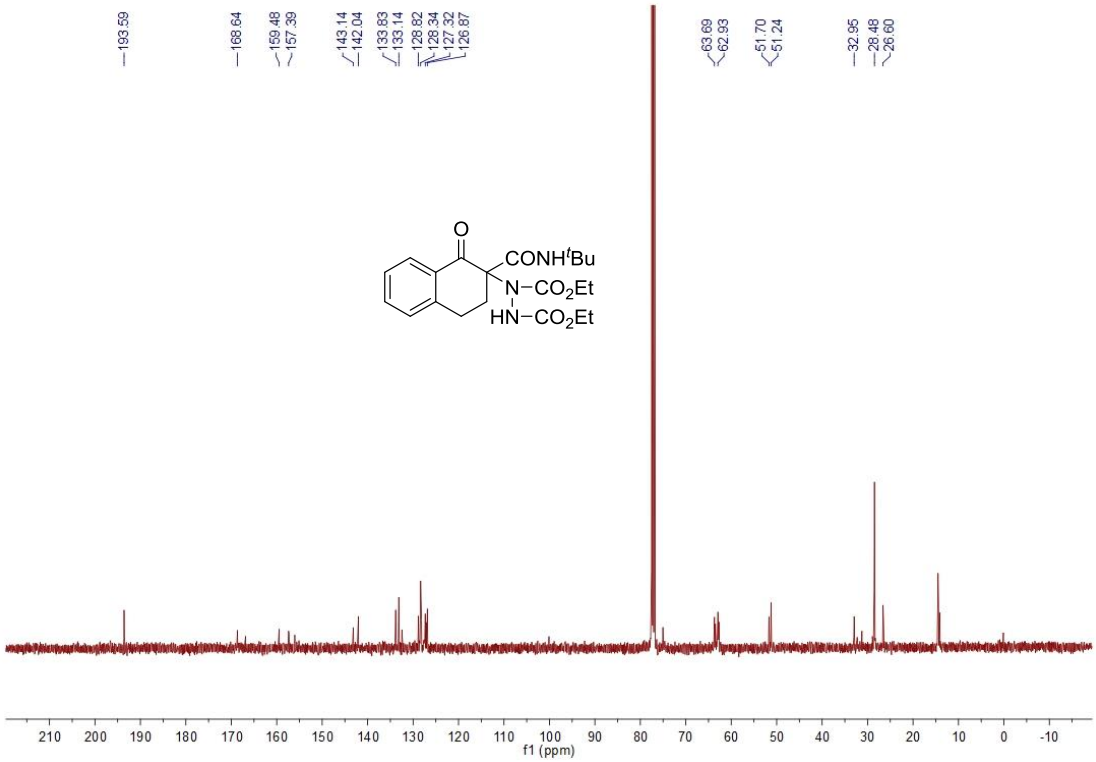
7.32
7.17
6.84
6.66
6.61
6.45
6.26
4.25
4.24
4.23
4.22
4.21
4.20
4.19
4.18
4.16
4.14
3.22
3.19
3.16
2.96
2.66
2.43
2.38
2.29
1.25
1.23
1.07
0.97
0.96
0.90
0.88
0.87
0.86
O
CONHtBu
N−CO2Et
HN−CO2Et
1.04
4.15
1.01
4.00
2.12
1.76
14.83
f1 (ppm)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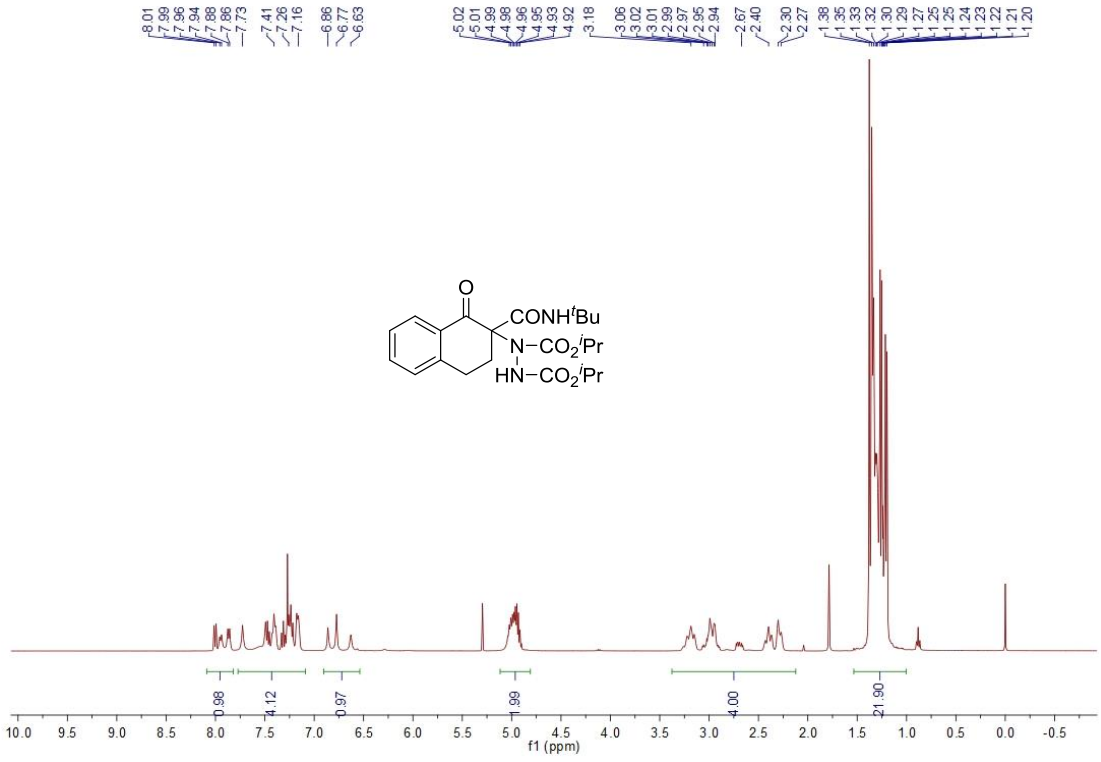
142.04
133.83
133.14
128.82
128.34
127.32
126.87
63.69
62.93
51.70
51.24
32.95
28.48
26.60
O
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
f1 (ppm)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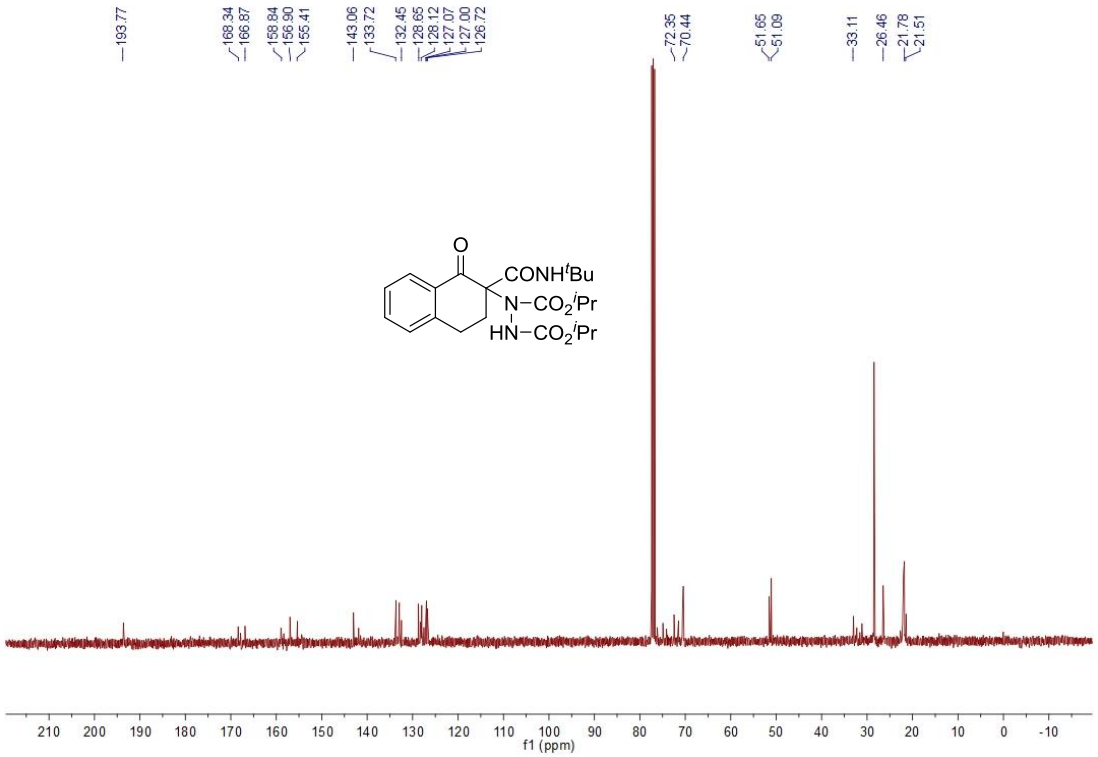
7.94
7.88
7.86
7.73
7.41
7.26
7.16
6.86
6.77
6.63
5.02
5.01
4.99
4.98
4.96
4.95
4.93
4.92
3.18
3.06
3.02
3.01
2.99
2.97
2.95
2.94
2.67
2.40
2.30
2.27
1.38
1.35
1.33
1.32
1.30
1.29
1.27
1.25
1.25
1.24
1.23
1.22
1.21
1.20
O
CONH^{t}Bu
N−CO$_2$^{i}Pr
HN−CO$_2$^{i}Pr
0.98
4.12
0.97
1.99
4.00
21.90
f1 (ppm)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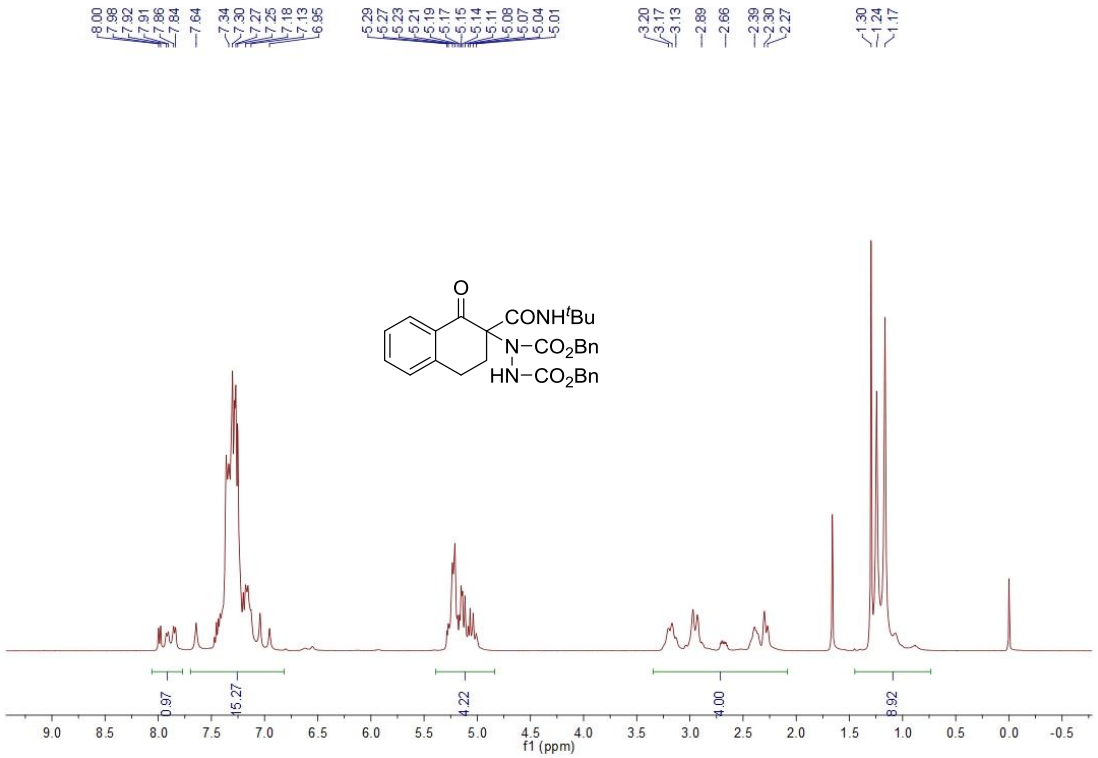
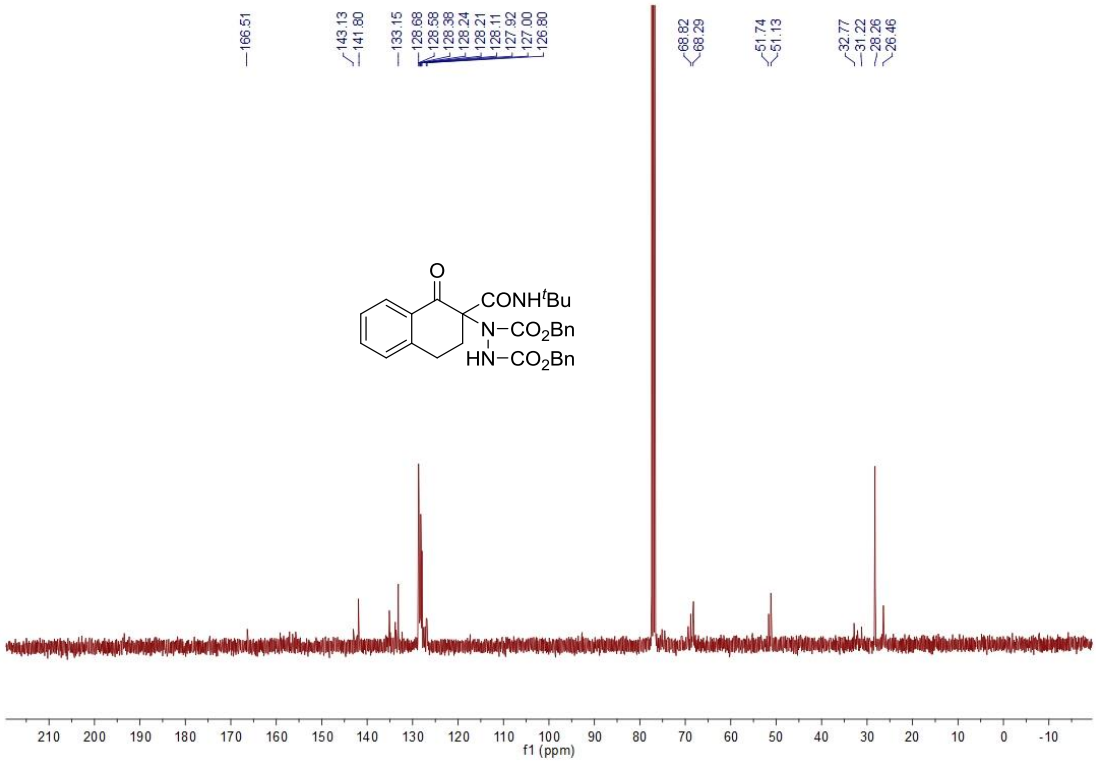
158.84
156.90
155.41
143.06
133.72
132.45
128.65
128.12
127.07
127.00
126.72
72.35
70.44
51.65
51.09
33.11
26.46
21.78
21.51
O
CONHᵗBu
N−CO₂ⁱPr
HN−CO₂ⁱPr
f1 (ppm)


8.00
7.98
7.92
7.91
7.86
7.84
7.64
7.34
7.30
7.27
7.25
7.18
7.13
6.95
5.29
5.27
5.23
5.21
5.19
5.17
5.15
5.14
5.11
5.08
5.07
5.04
5.01
3.20
3.17
3.13
2.89
2.66
2.39
2.30
2.27
1.30
1.24
1.17
O
CONHᵗBu
N−CO₂Bn
HN−CO₂Bn
0.97
15.27
4.22
4.00
8.92
f1 (ppm)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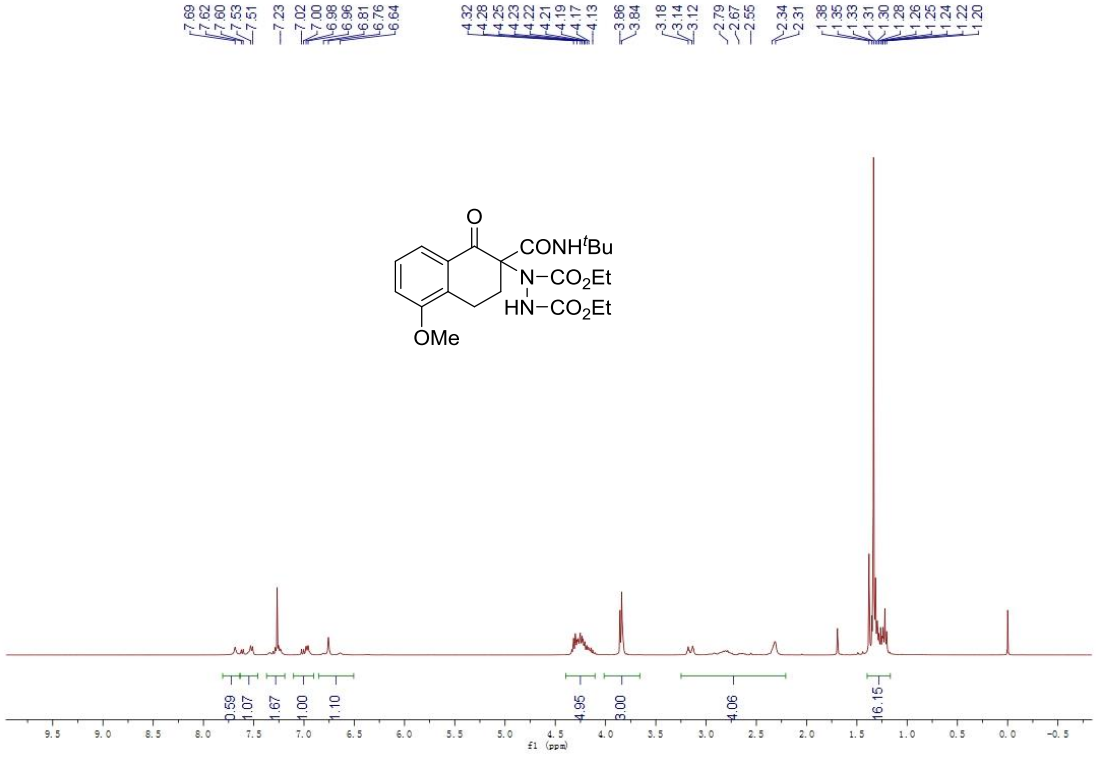
133.15
128.68
128.58
128.38
128.24
128.21
128.11
127.92
127.00
126.80
68.82
68.29
51.74
51.13
32.77
31.22
28.26
26.46
O
CONHtBu
N−CO2Bn
HN−CO2Bn
f1 (ppm)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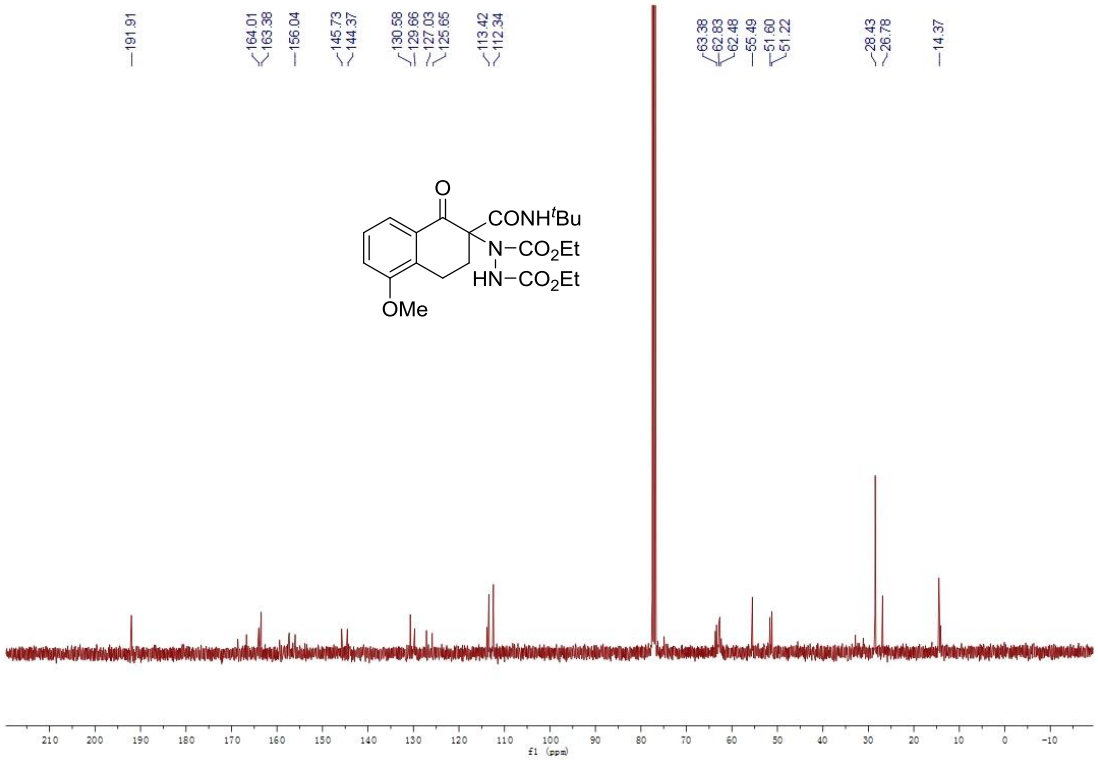
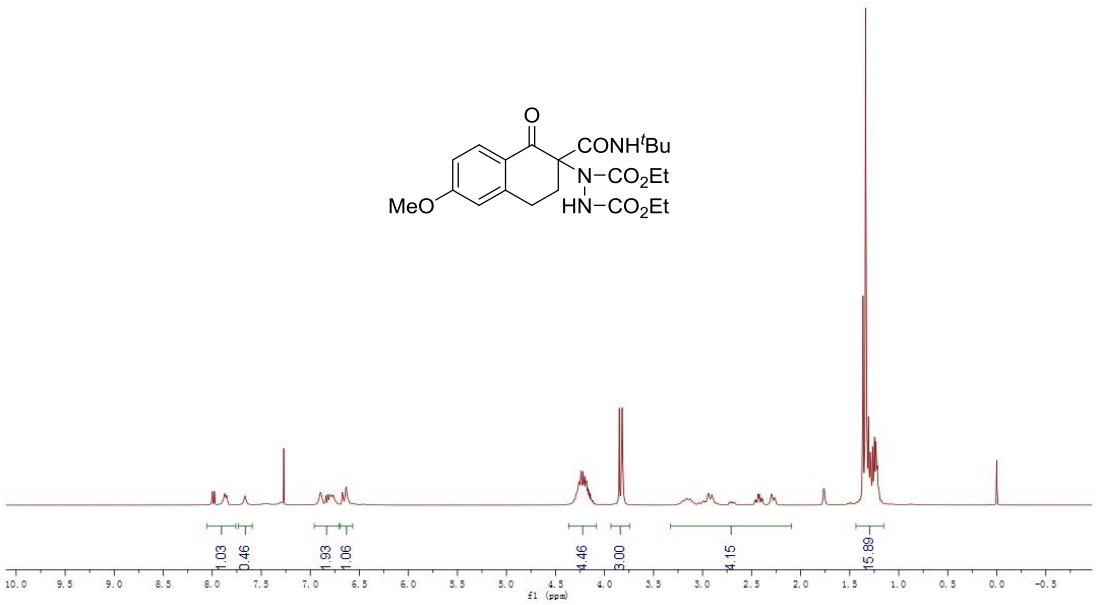
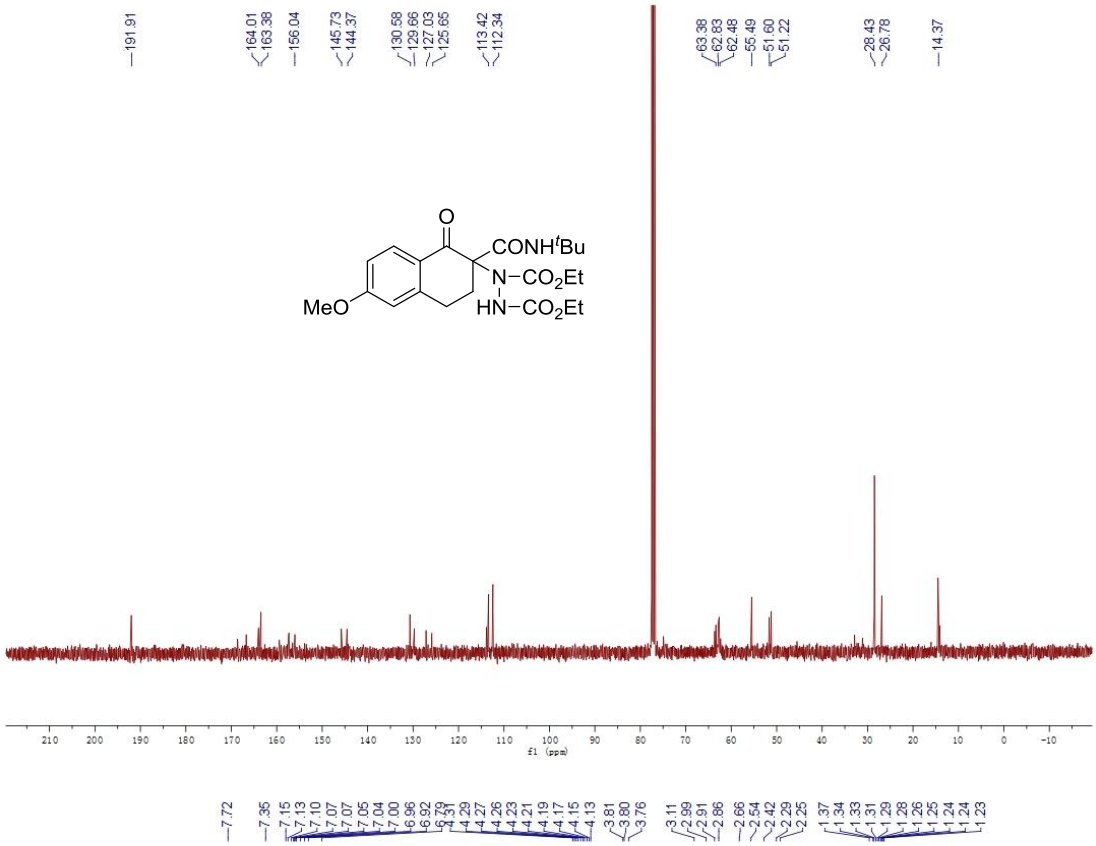
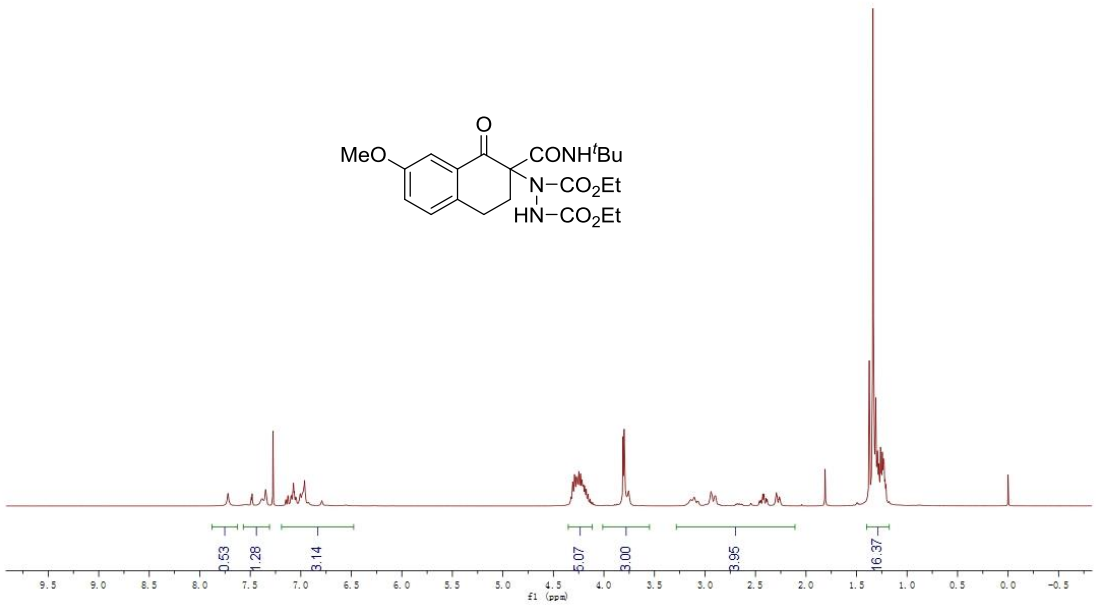
7.62
7.60
7.53
7.51
7.23
7.02
7.00
6.98
6.96
6.81
6.76
6.64
4.32
4.28
4.25
4.23
4.22
4.21
4.19
4.17
4.13
3.86
3.84
3.18
3.14
3.12
2.79
2.67
2.55
2.34
2.31
1.38
1.35
1.33
1.31
1.30
1.28
1.26
1.25
1.24
1.22
1.20
O
CONHtBu
N−CO2Et
HN−CO2Et
OMe
0.59
1.07
1.67
1.00
1.10
4.95
3.00
4.06
16.15
f1 (ppm)

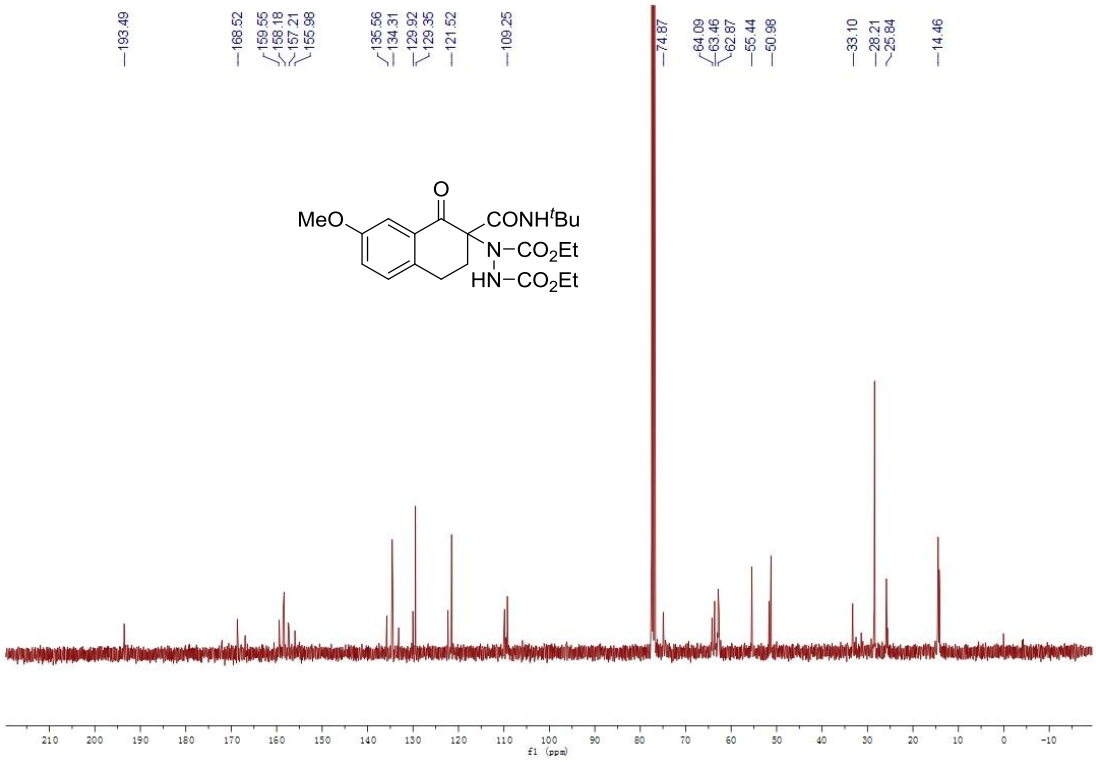

191.91
164.01
163.38
156.04
145.73
144.37
130.58
129.66
127.03
125.65
113.42
112.34
63.38
62.83
62.48
55.49
51.60
51.22
28.43
26.78
14.37
O
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
OMe
f1 (ppm)

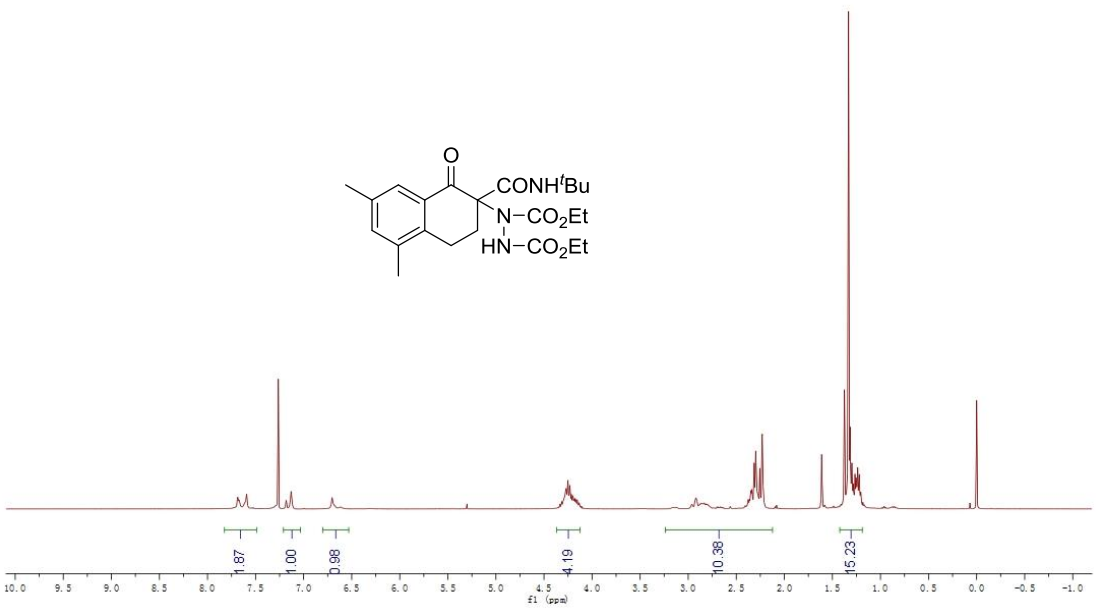

8.00
7.98
7.87
7.85
7.67
6.90
6.85
6.84
6.82
6.82
6.79
6.77
6.67
6.67
6.64
4.24
4.21
4.20
4.19
4.18
4.17
4.16
4.15
4.14
3.85
2.94
2.91
2.73
2.70
2.68
2.42
2.39
2.26
1.36
1.34
1.31
1.29
1.29
1.27
1.25
1.23
1.23
1.22

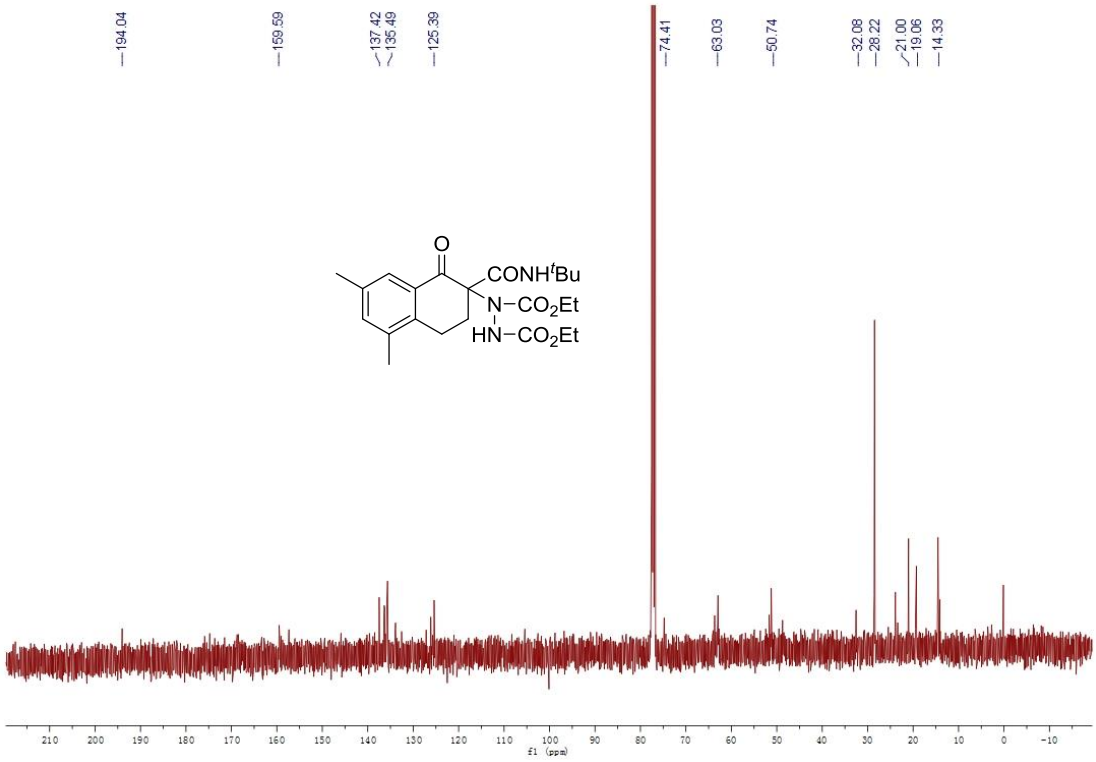

O
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
MeO
1.03
0.46
1.93
1.06
4.46
3.00
4.15
15.89
f1 (ppm)


191.91
164.01
163.38
156.04
145.73
144.37
130.58
129.66
127.03
125.65
113.42
112.34
63.38
62.83
62.48
55.49
51.60
51.22
28.43
26.78
14.37
O
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
MeO
f1 (ppm)

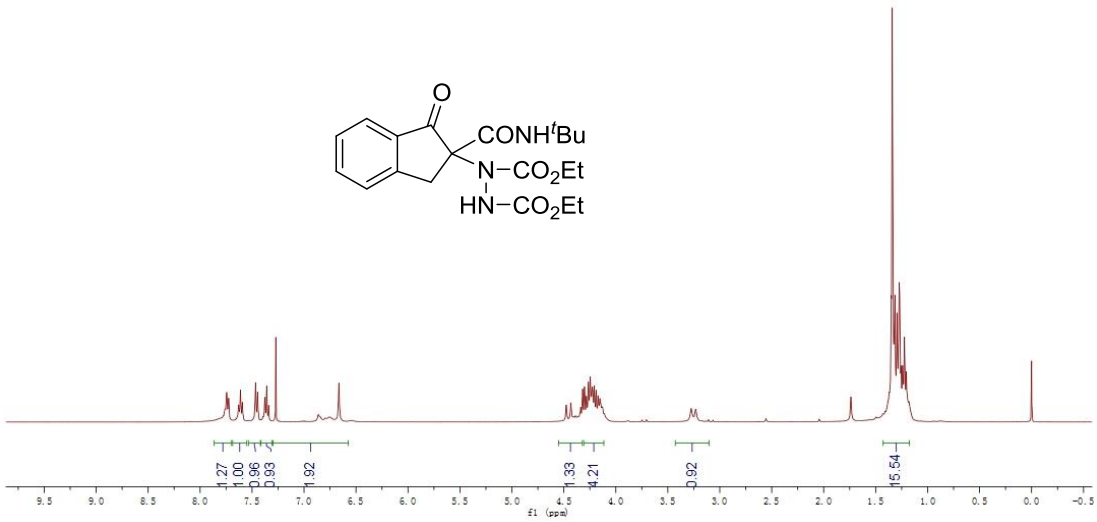

7.72
7.36
7.15
7.13
7.10
7.07
7.07
7.05
7.04
7.00
6.96
6.92
6.79
4.31
4.29
4.27
4.26
4.23
4.21
4.19
4.17
4.15
4.13
3.81
3.80
3.76
3.11
2.99
2.91
2.86
2.66
2.54
2.42
2.29
2.25
1.37
1.34
1.33
1.31
1.29
1.28
1.26
1.25
1.24
1.24
1.23
O
MeO
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
0.53
1.28
3.14
5.07
3.00
3.95
16.37
f1 (ppm)

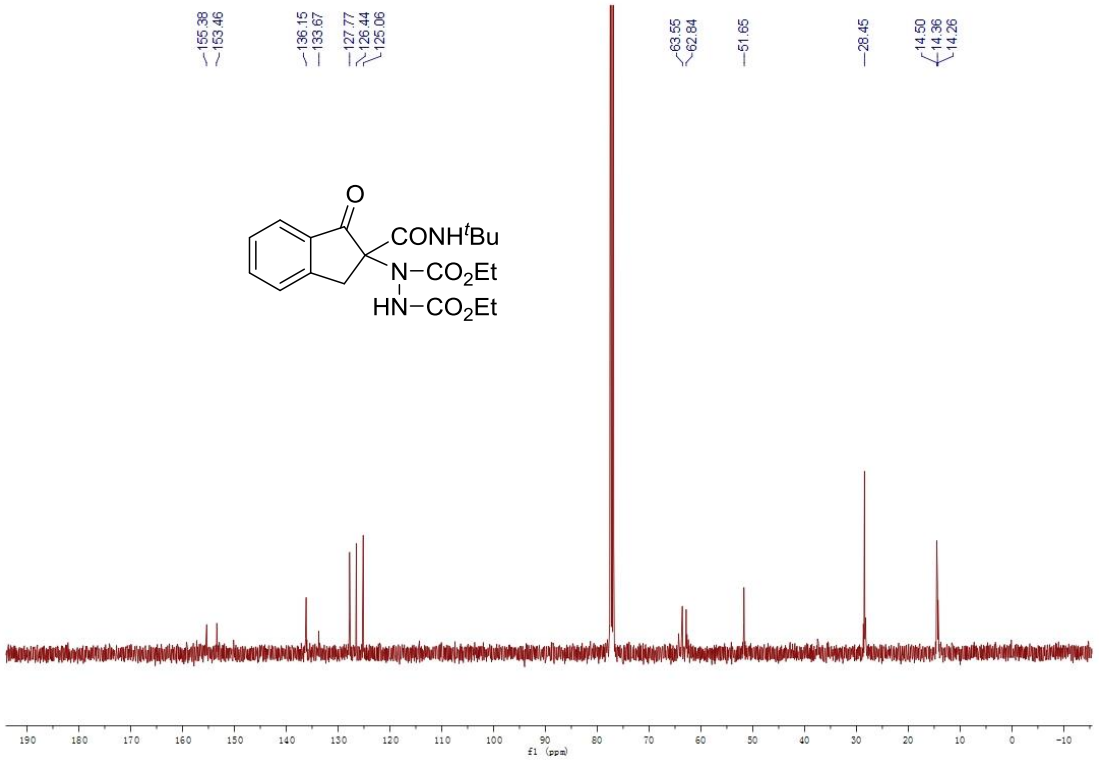

193.49
168.52
159.55
158.18
157.21
155.98
135.56
134.31
129.92
129.35
121.52
109.25
74.87
64.09
63.46
62.87
55.44
50.98
33.10
28.21
25.64
14.46
MeO
O
CONHᵗBu
N−CO₂Et
HN−CO₂Et
f1 (ppm)

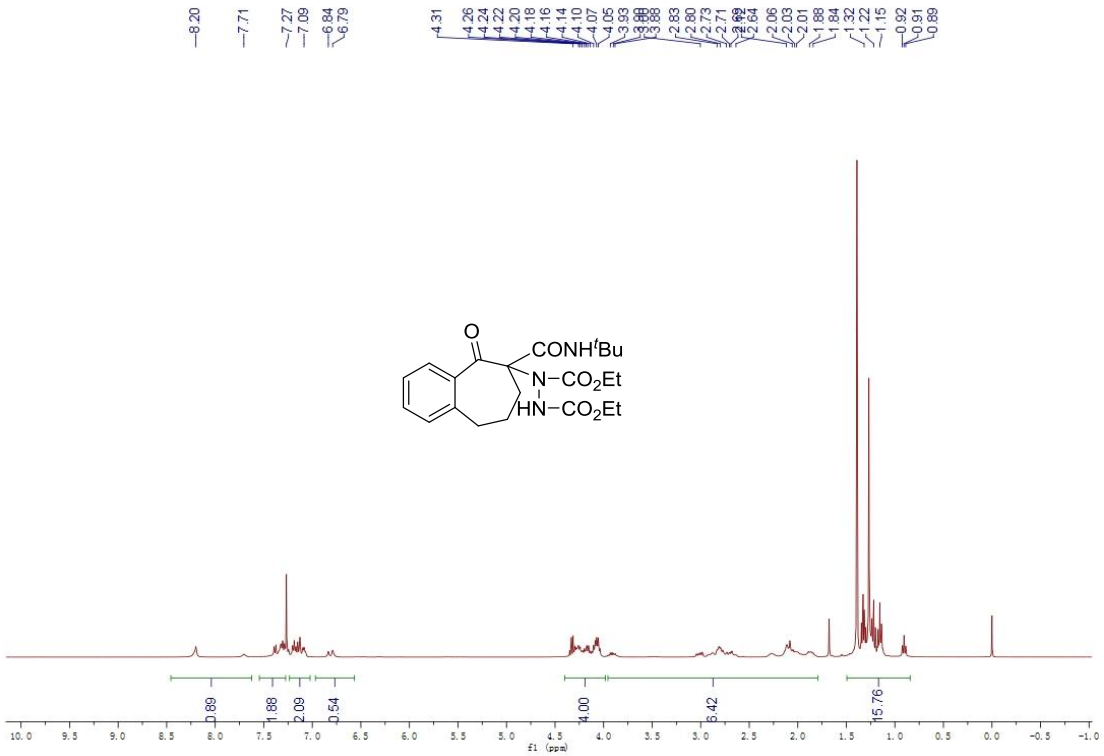

7.69
7.67
7.59
7.18
7.13
6.70
6.62
4.25
4.22
4.21
4.20
4.19
4.18
4.17
4.16
4.15
4.15
4.14
3.16
3.14
3.13
2.92
2.67
2.56
2.36
2.31
2.14
2.05
1.38
1.33
1.31
1.30
1.28
1.27
1.25
1.25
1.24
1.22
O
CONHᵗBu
N−CO₂Et
HN−CO₂Et
1.87
1.00
0.98
4.19
10.38
15.23
f1 (ppm)

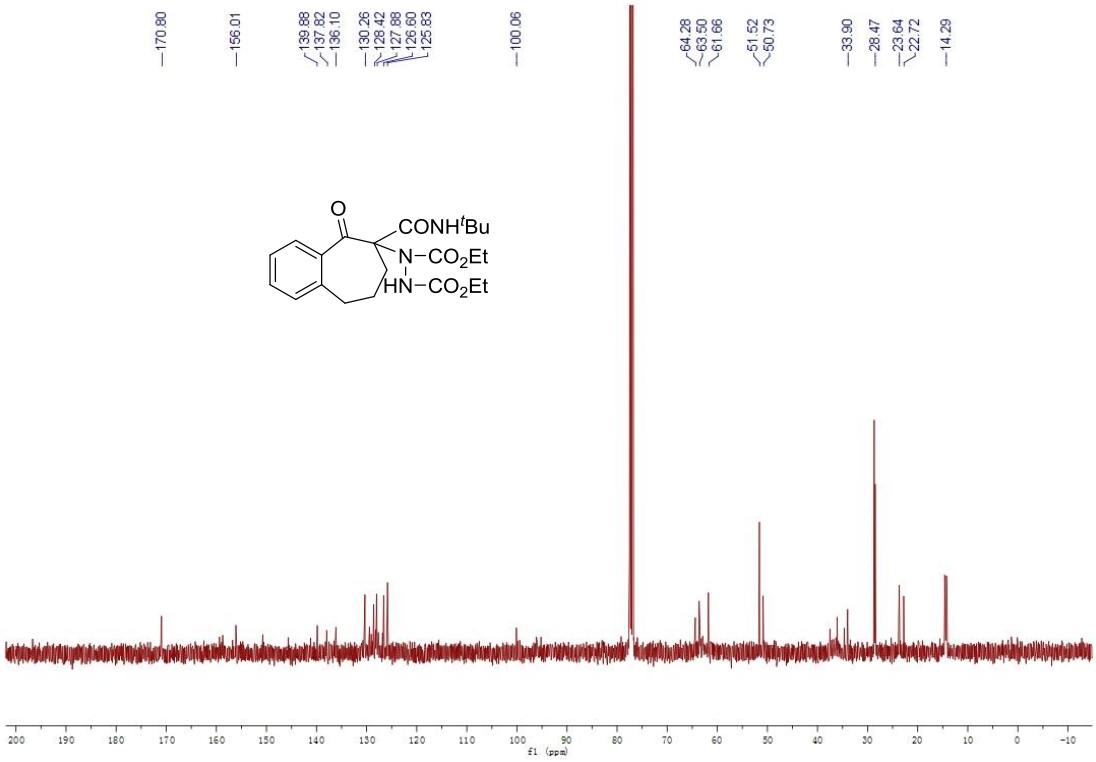

194.04
159.59
137.42
135.49
125.39
74.41
63.03
50.74
32.08
28.22
21.00
19.06
14.33
O
CONHᵗBu
N−CO₂Et
HN−CO₂Et
f1 (ppm)

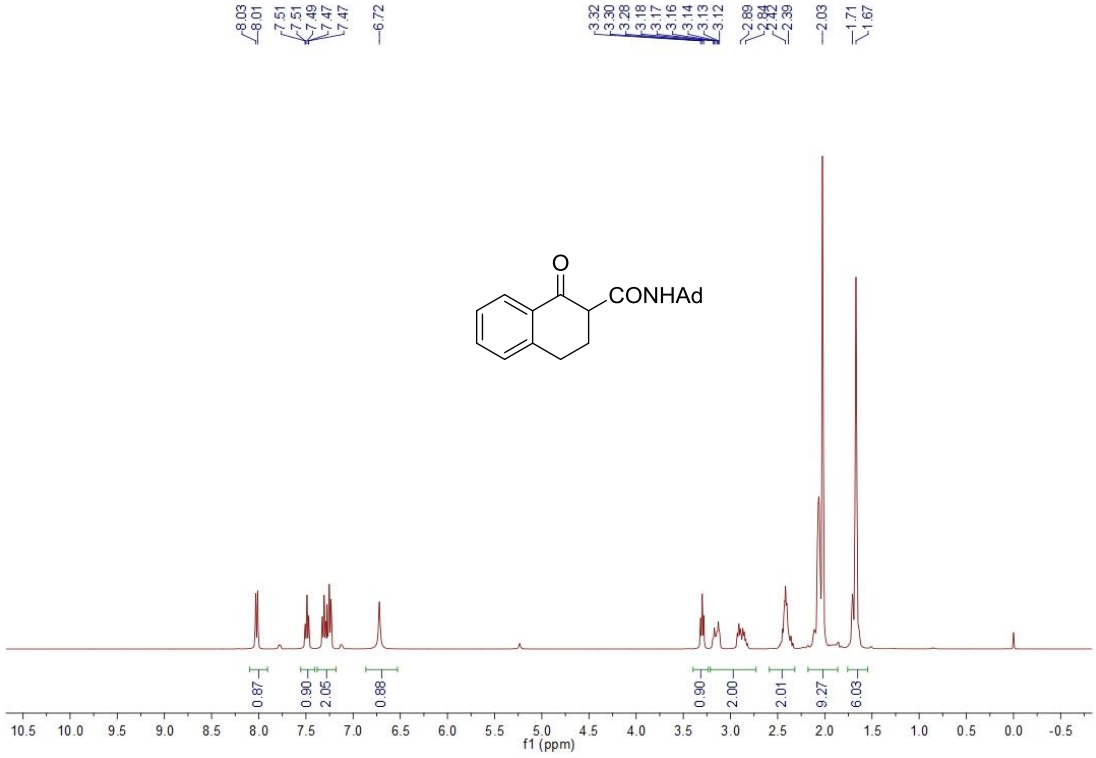

7.760
7.744
7.725
7.630
7.612
7.594
7.466
7.447
7.378
7.359
7.341
7.272
6.862
6.754
6.664
4.302
4.283
4.264
4.246
4.226
4.208
4.190
4.172
4.153
4.145
4.135
4.128
3.275
3.232
1.350
1.341
1.332
1.319
1.314
1.295
1.273
1.270
1.256
1.241
1.223
1.205

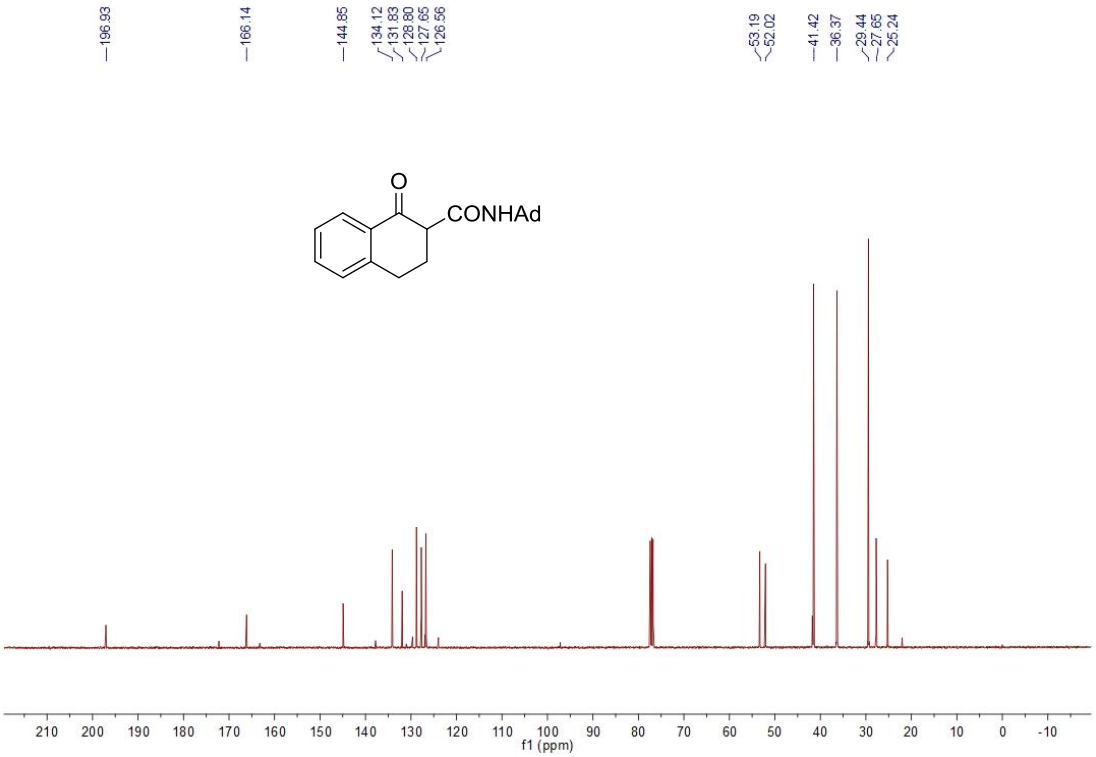

O
CONHᵗBu
N−CO₂Et
HN−CO₂Et
1.27
1.00
0.96
0.93
1.92
1.33
4.21
0.92
15.54
f1 (ppm)

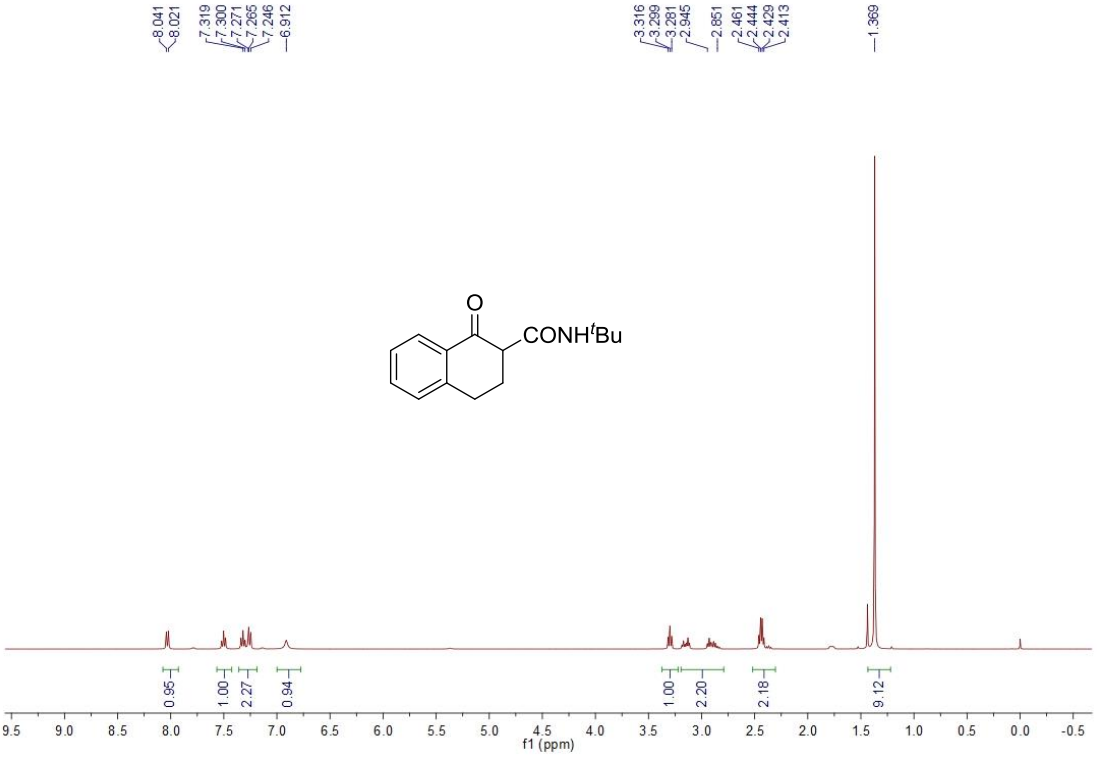

155.38
153.46
136.15
133.67
127.77
126.44
125.06
63.55
62.84
51.65
28.45
14.50
14.36
14.26
O
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
f1 (ppm)

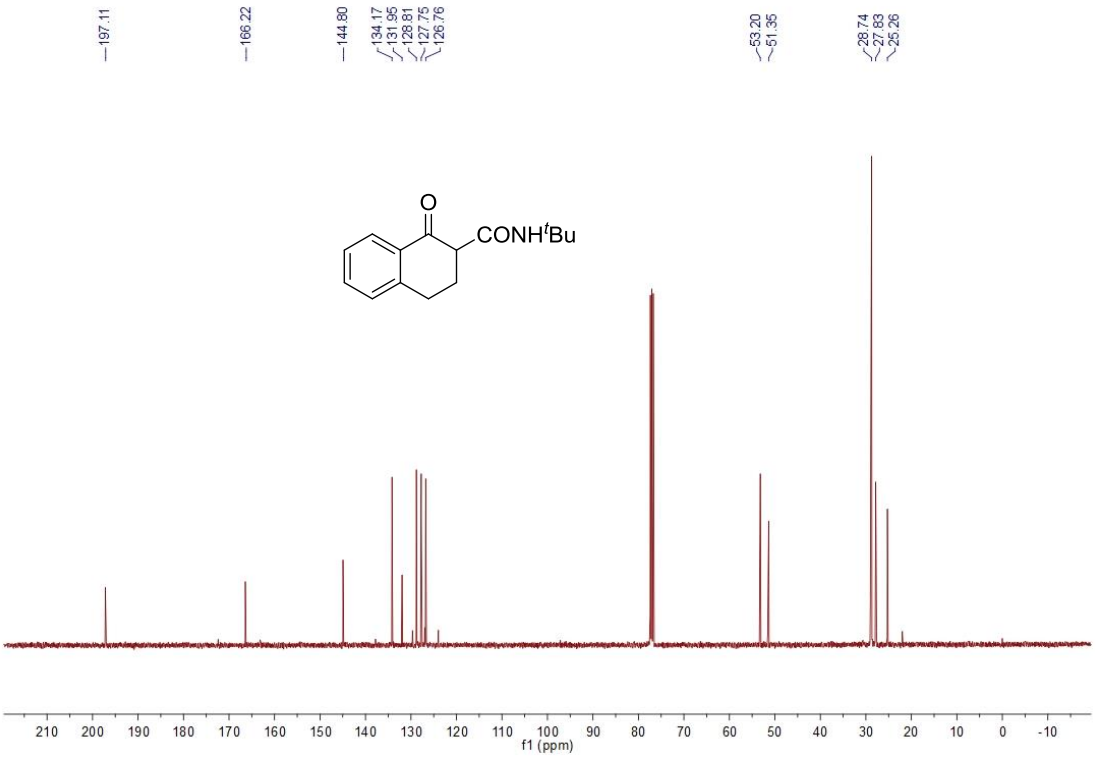

8.20
7.71
7.27
7.09
6.84
6.79
4.31
4.26
4.24
4.22
4.20
4.18
4.16
4.14
4.10
4.07
4.05
2.83
2.80
2.73
2.71
2.64
2.06
2.03
2.01
1.88
1.84
1.32
1.22
1.15
0.92
0.91
0.89
O
CONH^{t}Bu
N−CO$_2$Et
HN−CO$_2$Et
0.89
1.88
2.09
0.54
4.00
5.42
15.76
f1 (ppm)

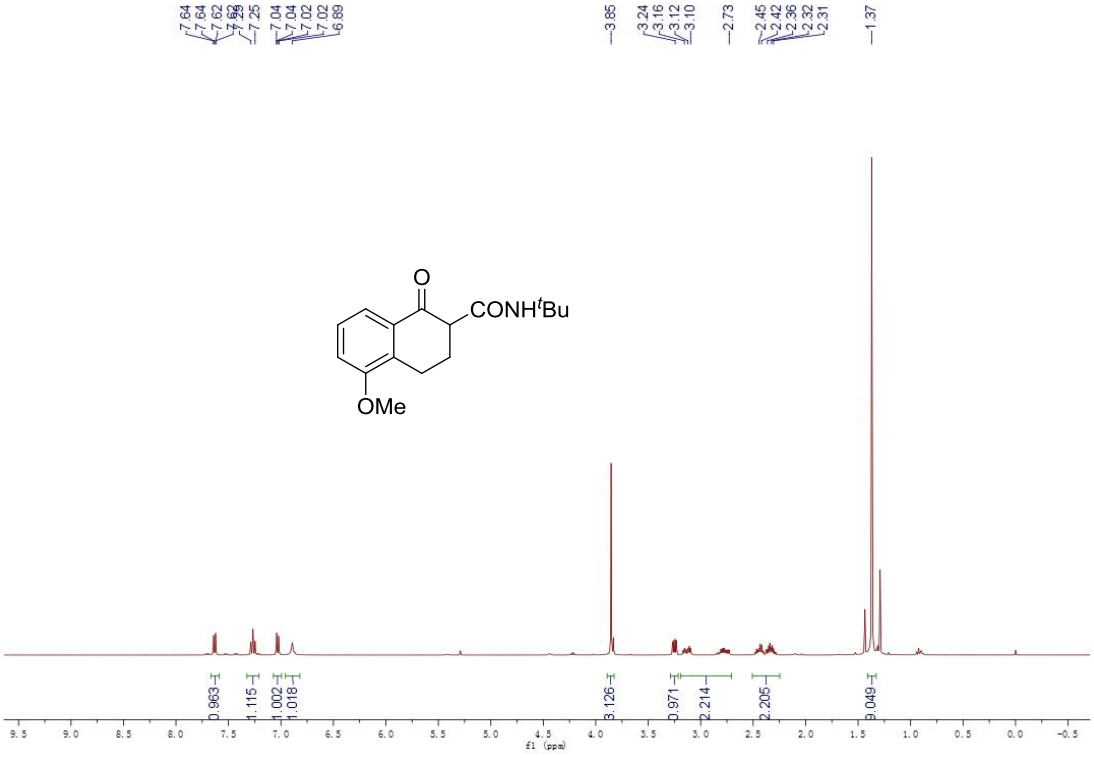

170.80
156.01
139.88
137.82
136.10
130.26
128.42
127.88
126.60
125.83
100.06
64.28
63.50
61.66
51.52
50.73
33.90
28.47
23.64
22.72
14.29
O
CONHtBu
N−CO2Et
HN−CO2Et
f1 (ppm)

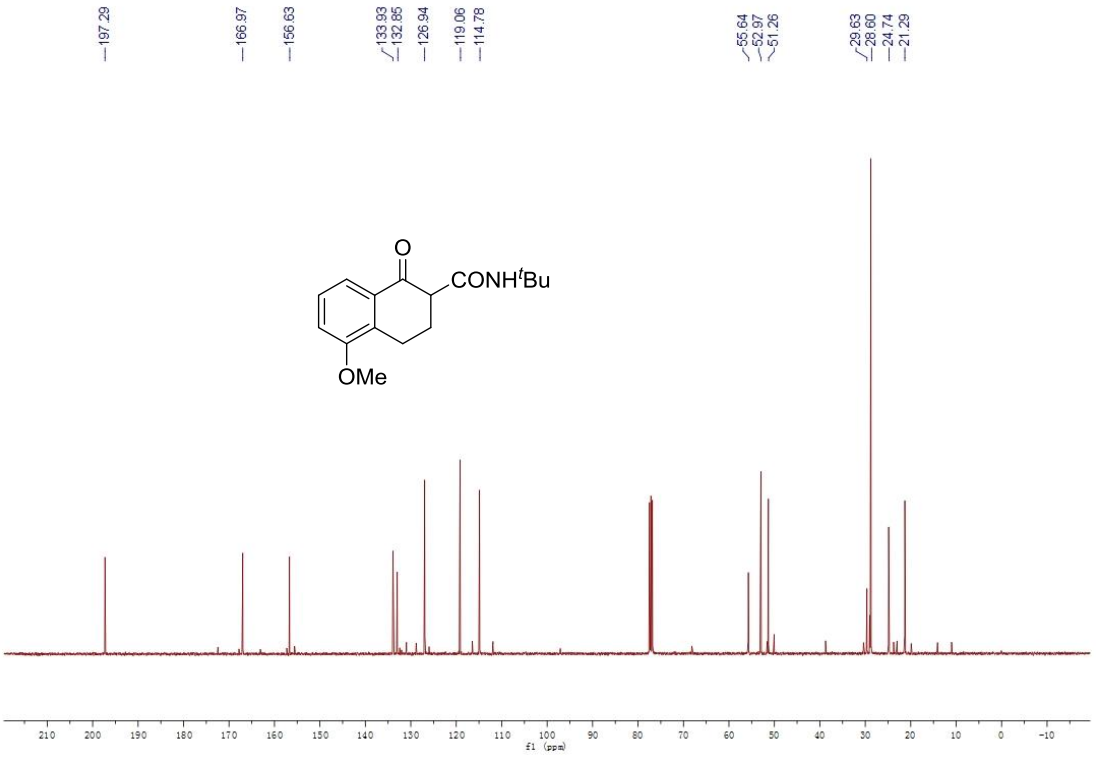

8.03
8.01
7.51
7.51
7.49
7.47
7.47
6.72
3.32
3.30
3.28
3.18
3.17
3.16
3.14
3.13
3.12
2.89
2.84
2.42
2.39
2.03
1.71
1.67
O
CONHAd
0.87
0.90
2.05
0.88
0.90
2.00
2.01
9.27
6.03
f1 (ppm)


196.93
166.14
144.85
134.12
131.83
128.80
127.65
126.56
53.19
52.02
41.42
36.37
29.44
27.65
25.24
O
CONHAd
f1 (ppm)


8.041
8.021
7.319
7.300
7.271
7.265
7.246
6.912
3.316
3.299
3.281
2.945
2.851
2.461
2.444
2.429
2.413
1.369
O
CONH^{t}Bu
0.95
1.00
2.27
0.94
1.00
2.20
2.18
9.12
f1 (ppm)


197.11
166.22
144.80
134.17
131.95
128.81
127.75
126.76
53.20
51.35
28.74
27.83
25.26
O
CONHtBu
f1 (ppm)


7.64
7.64
7.62
7.62
7.25
7.04
7.04
7.02
7.02
6.89
3.85
3.24
3.16
3.12
3.10
2.73
2.45
2.42
2.36
2.32
2.31
1.37
O
CONHtBu
OMe
0.963
1.115
1.002
1.018
3.126
0.971
2.214
2.205
9.049
f1 (ppm)


197.29
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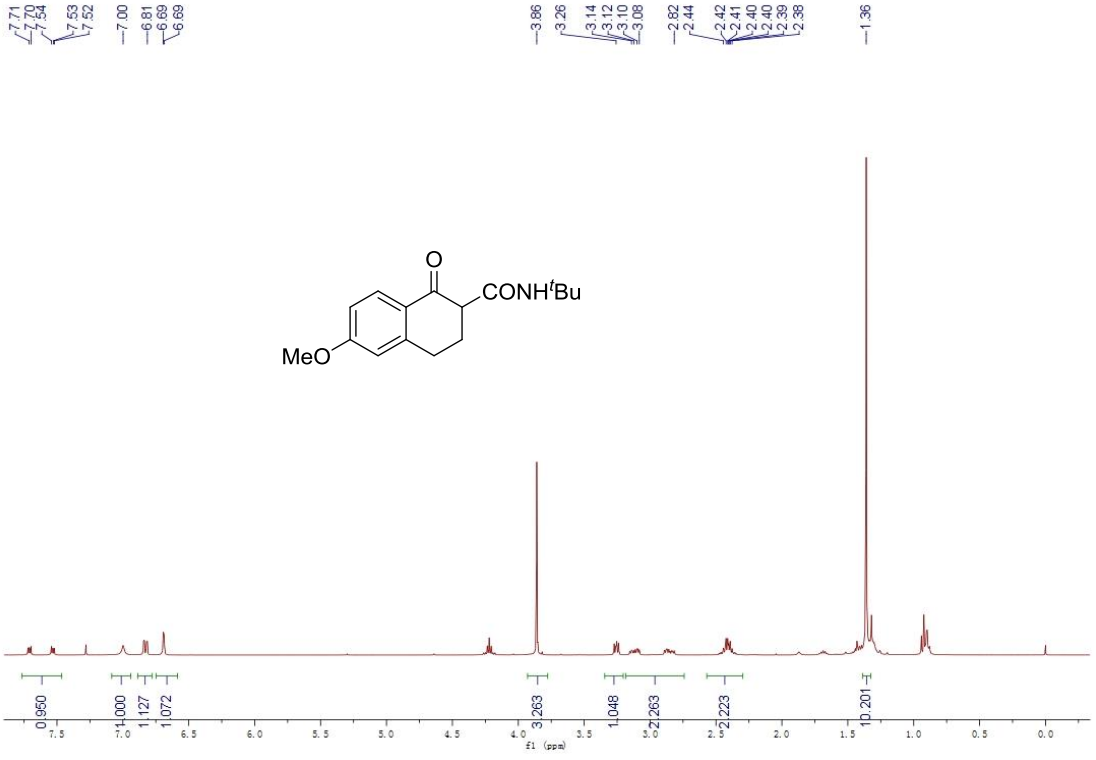
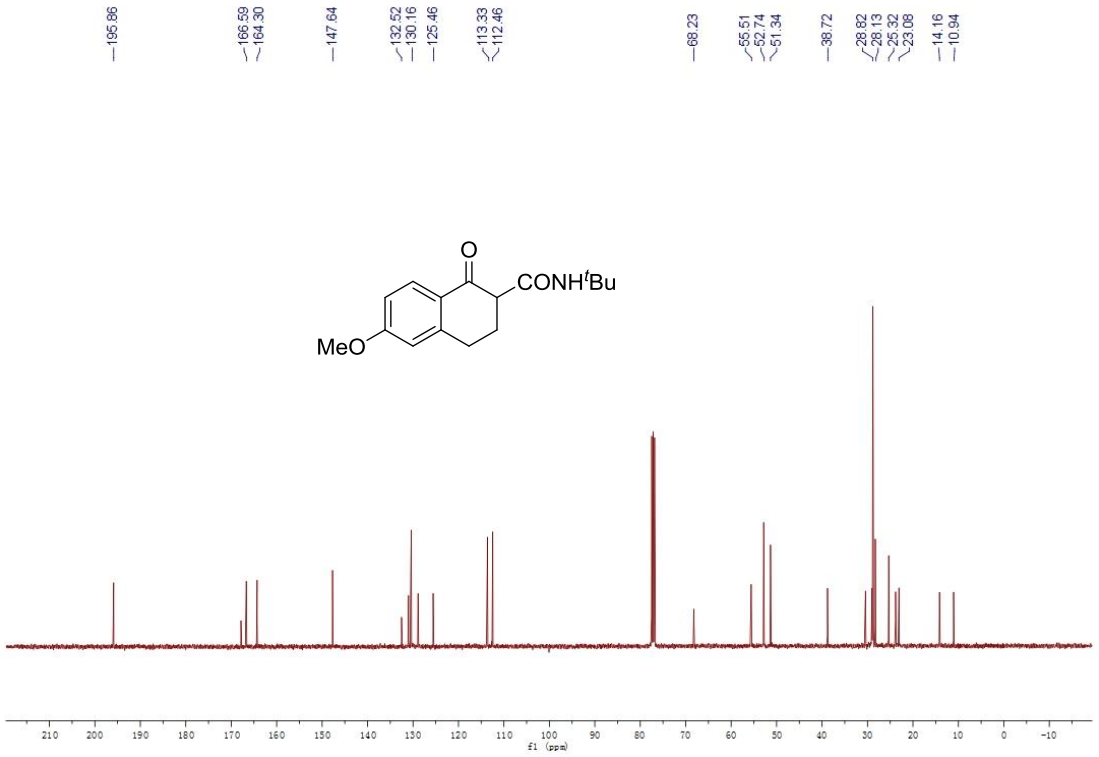
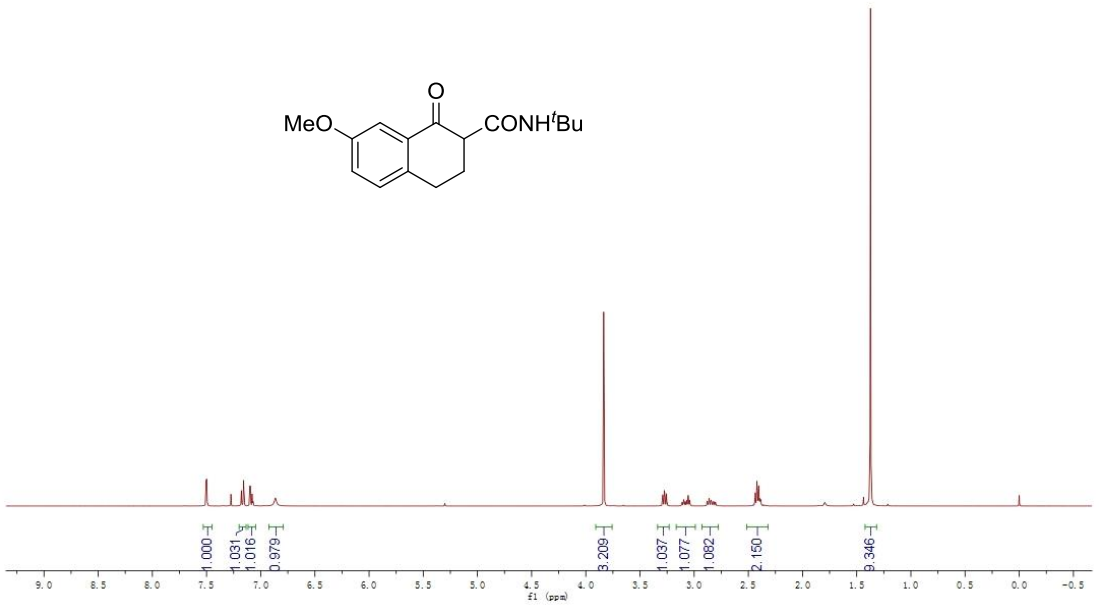
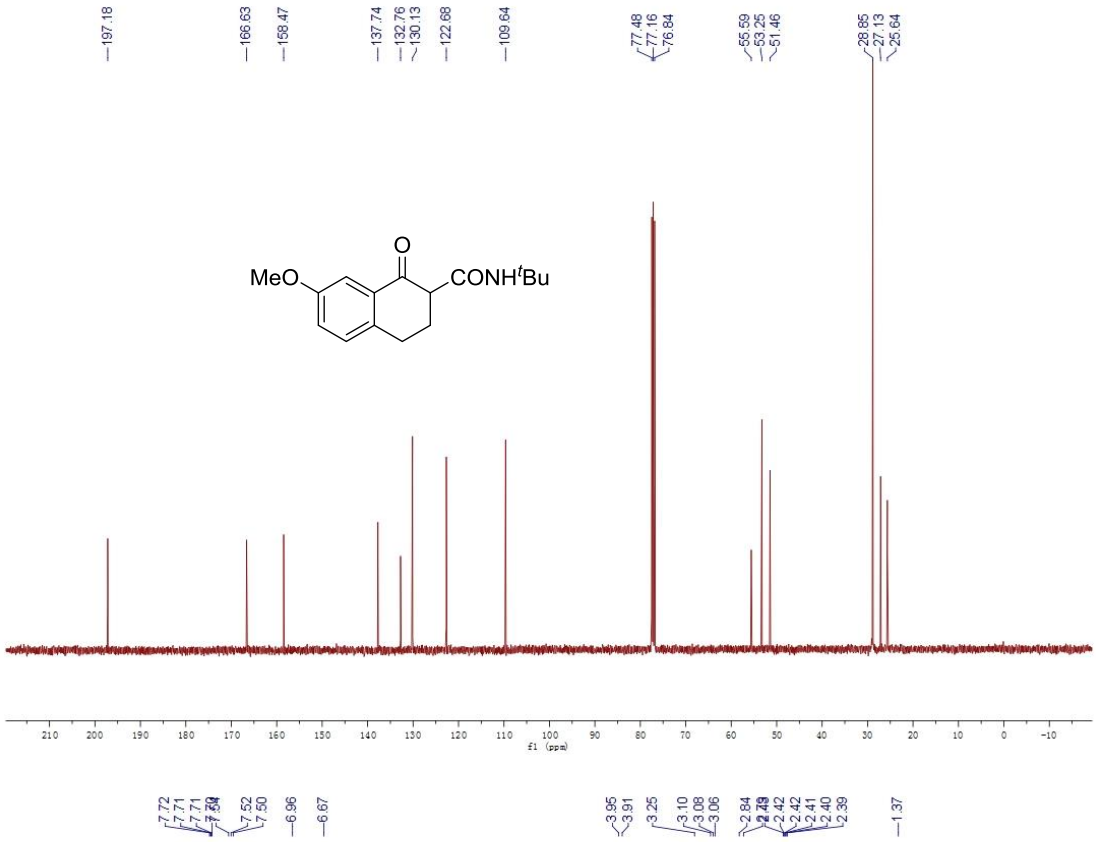
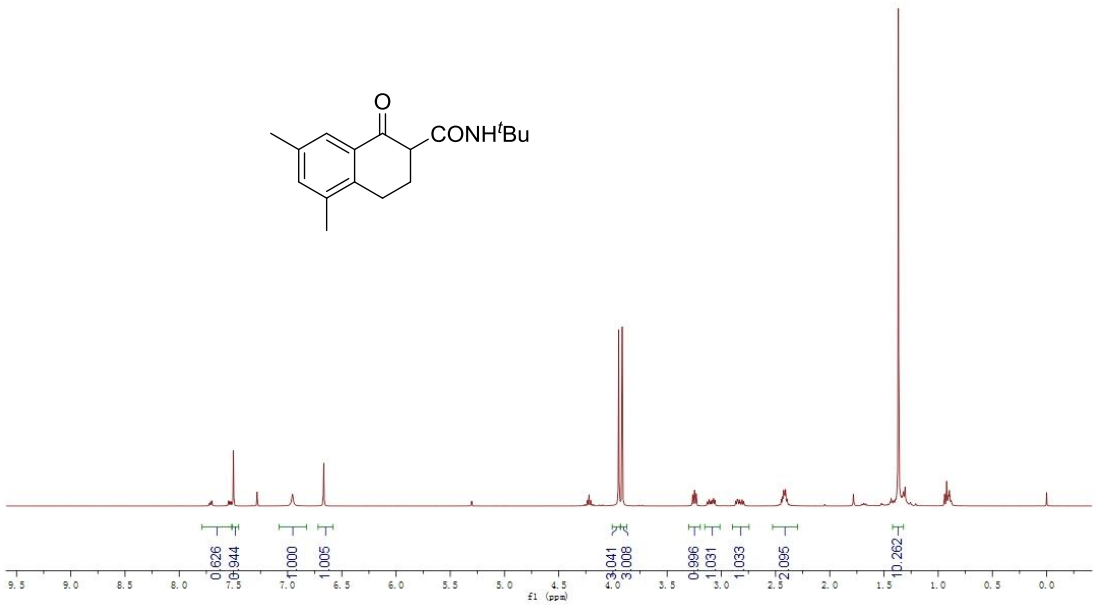
156.63
133.93
132.85
126.94
119.06
114.78
55.64
52.97
51.26
29.63
28.60
24.74
21.29
O
CONHᵗBu
OMe
f1 (ppm)

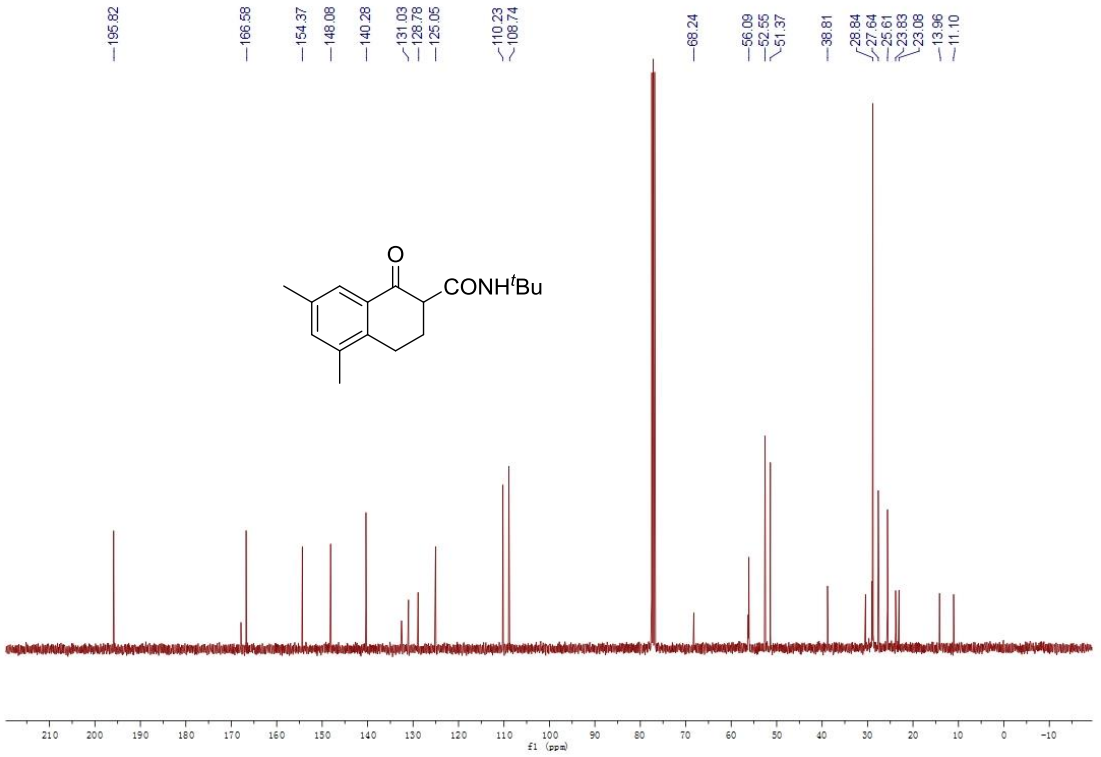

7.71
7.70
7.54
7.53
7.52
7.00
6.81
6.69
6.69
3.86
3.26
3.14
3.12
3.10
3.08
2.82
2.44
2.42
2.41
2.40
2.40
2.39
2.38
1.36
O
CONHᵗBu
MeO
0.950
1.000
1.127
1.072
3.263
1.048
2.263
2.223
10.201
f1 (ppm)

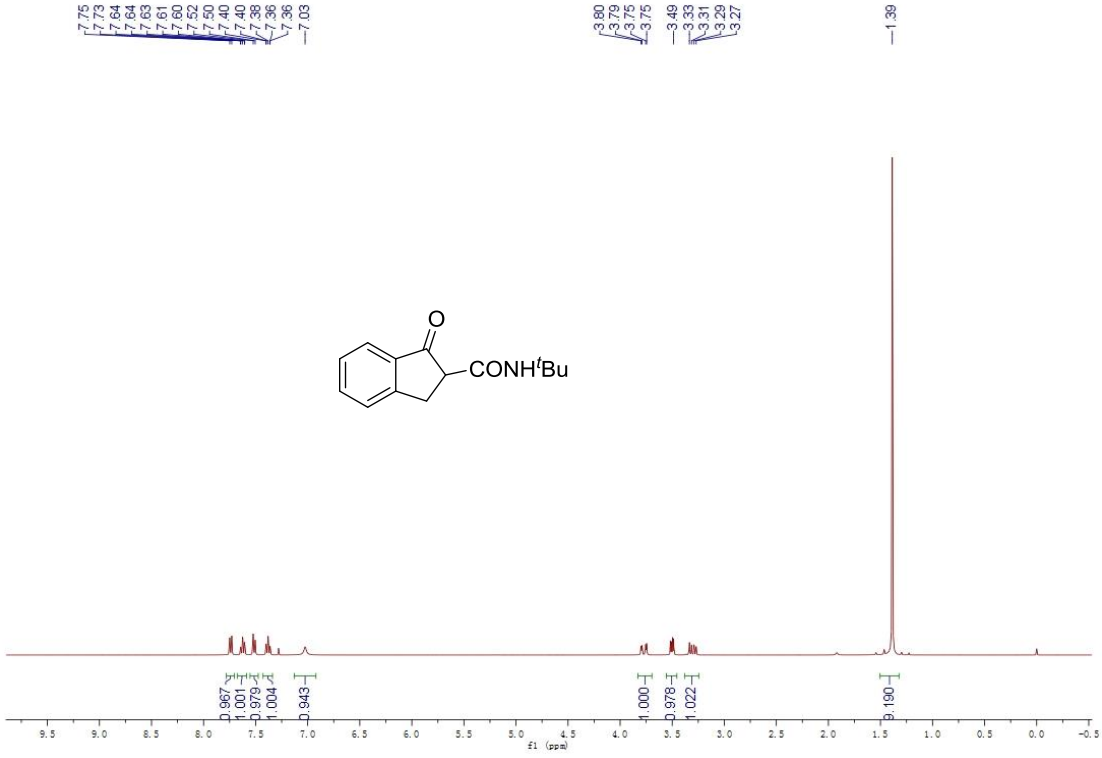

195.86
166.59
164.30
147.64
132.52
130.16
125.46
113.33
112.46
68.23
55.51
52.74
51.34
38.72
28.82
28.13
25.32
23.08
14.16
10.94
O
CONH^{t}Bu
MeO
f1 (ppm)

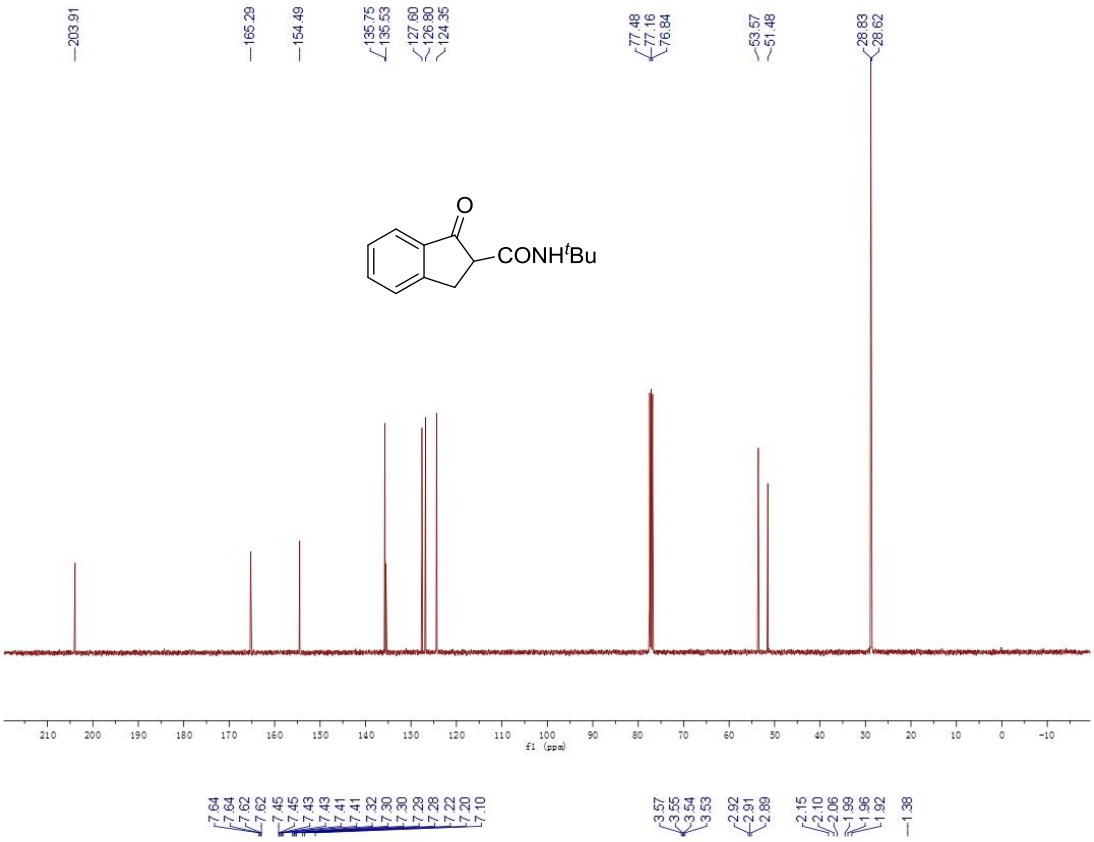

7.50
7.50
7.10
7.07
6.86
3.83
3.29
3.27
3.26
3.04
2.88
2.42
2.40
2.40
2.39
2.38
1.37
O
MeO
CONH^{t}Bu
1.000
1.031
1.016
0.979
3.209
1.037
1.077
1.082
2.150
9.346
f1 (ppm)

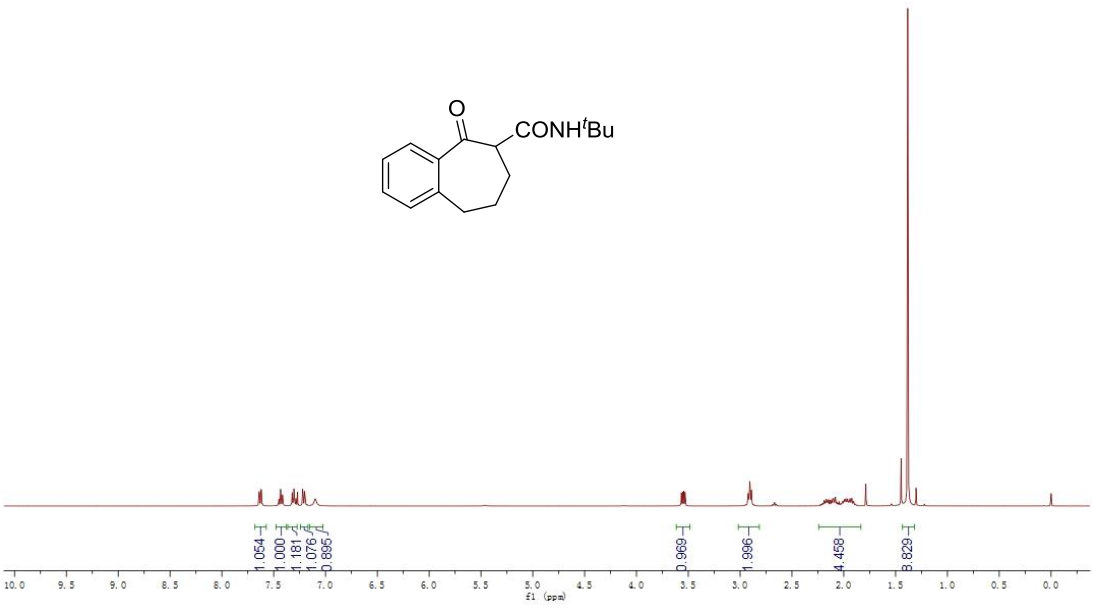

197.18
166.63
158.47
137.74
132.76
130.13
122.68
109.64
77.48
77.16
76.84
55.59
53.25
51.46
28.85
27.13
25.64
MeO
O
CONHtBu
f1 (ppm)
7.72
7.71
7.71
7.52
7.50
6.96
6.67
3.95
3.91
3.25
3.10
3.08
3.06
2.84
2.42
2.42
2.41
2.40
2.39
1.37

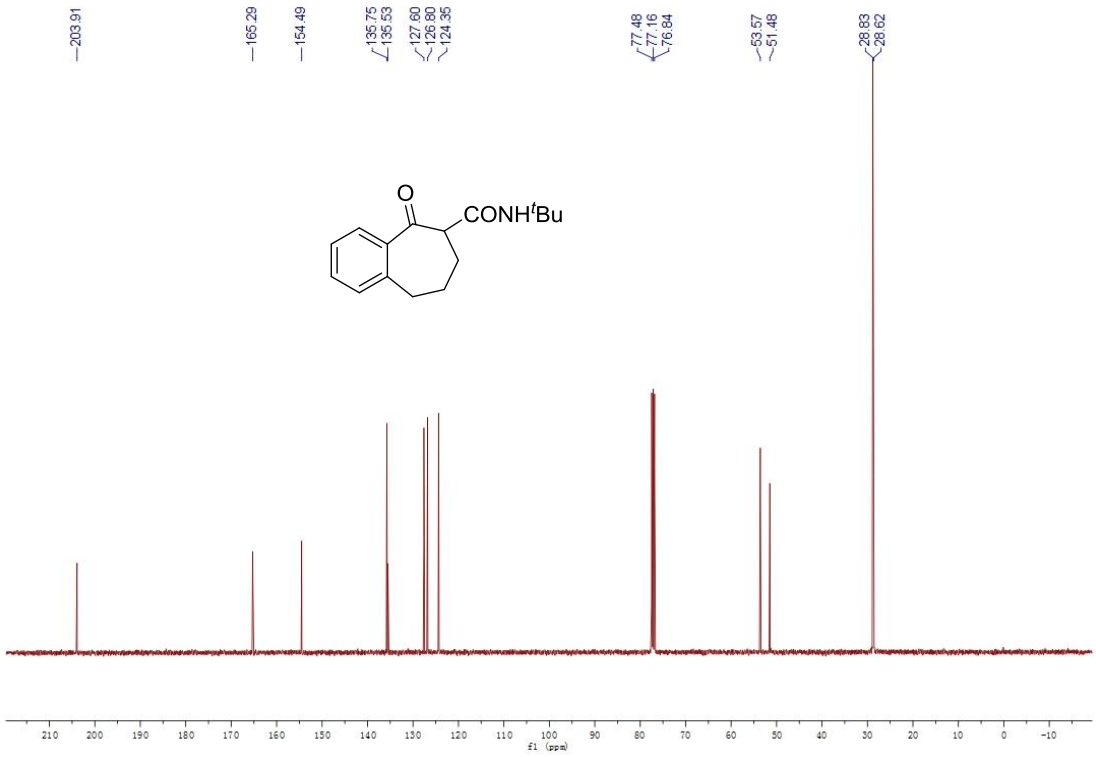

O
CONHtBu
0.626
0.944
1.000
1.005
3.041
3.008
0.996
1.031
1.033
2.095
10.262
f1 (ppm)


195.82
166.58
154.37
148.08
140.28
131.03
128.78
125.05
110.23
108.74
68.24
56.09
52.55
51.37
38.81
28.84
27.64
25.61
23.83
23.08
13.96
11.10
O
CONH^{t}Bu
f1 (ppm)


7.75
7.73
7.64
7.64
7.63
7.61
7.60
7.52
7.50
7.40
7.40
7.38
7.36
7.36
7.03
3.80
3.79
3.75
3.75
3.49
3.33
3.31
3.29
3.27
1.39
O
CONH^{t}Bu
0.967
1.001
0.979
1.004
0.943
1.000
0.978
1.022
3.190
f1 (ppm)


203.91
165.29
154.49
135.75
135.53
127.60
126.80
124.35
77.48
77.16
76.84
53.57
51.48
28.83
28.62
O
CONHtBu
f1 (ppm)
7.64
7.64
7.62
7.62
7.45
7.45
7.43
7.43
7.41
7.41
7.32
7.30
7.30
7.29
7.28
7.22
7.20
7.10
3.57
3.55
3.54
3.53
2.92
2.91
2.89
2.15
2.10
2.06
1.99
1.96
1.92
1.38


O
CONHtBu
1.054
1.000
1.181
1.076
0.895
0.969
1.996
4.458
8.829
f1 (ppm)


203.91
165.29
154.49
135.75
135.53
127.60
126.80
124.35
77.48
77.16
76.84
53.57
51.48
28.83
28.62
O
CONH^{t}Bu
f1 (ppm)