

Supporting Information

Synthesis of α -Keto Imides through Copper-Catalyzed Oxidation of *N*-Sulfonyl Ynamides

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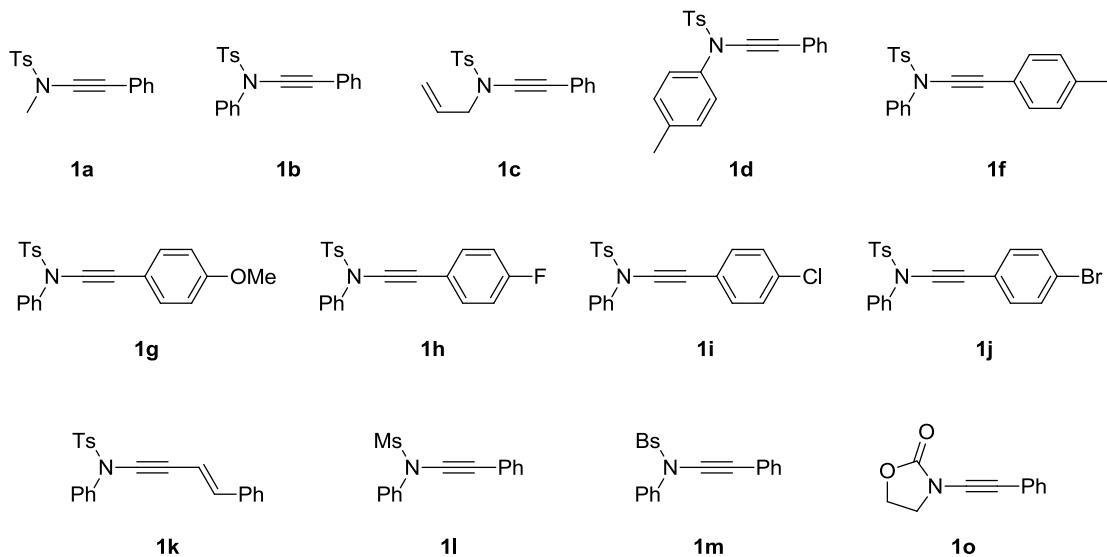
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General Information. Ethyl acetate (ACS grade), hexanes (ACS grade), methanol (ACS grade) and anhydrous DCE (ACS grade) were obtained commercially and used without further purification. Toluene and tetrahydrofuran were purified according to standard methods unless otherwise noted. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed over silica gel (300-400 mesh). Infrared spectra were recorded on a Nicolet AVATER FTIR330 spectrometer as thin film and are reported in reciprocal centimeter (cm^{-1}). Mass spectra were recorded with Micromass QTOF₂ Quadrupole/Time-of-Flight Tandem mass spectrometer using electron spray ionization.

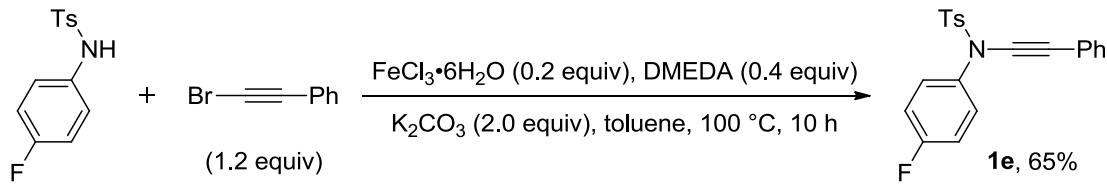
^1H NMR spectra were recorded on a Bruker AV-400 spectrometer and a Bruker AV-500 spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data is being reported as (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration).

^{13}C NMR spectra were recorded on a Bruker AV-400 spectrometer and a Bruker AV-500 spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard.

The data of the following ynamides **1a-d**, **1f-m** and **1o** were reported in our previous work.¹⁻³

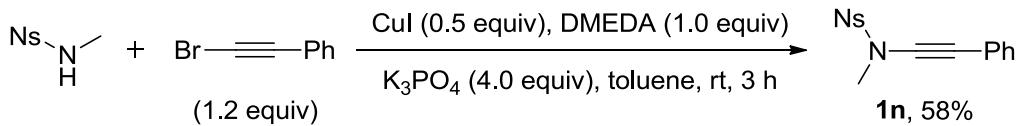


N-(4-fluorophenyl)-4-methyl-*N*-(phenylethyynyl)benzenesulfonamide (**1e**)

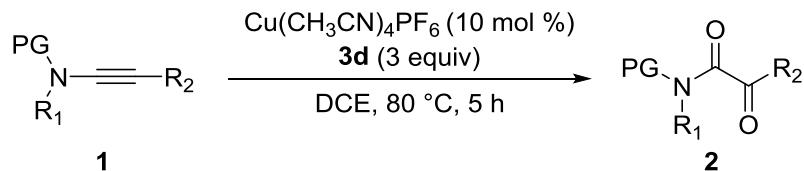


Compound **1e** was prepared in 65% yield (237.3 mg) according to the above known procedure.⁴ Pale yellow oil. ¹H NMR (500 MHz, CDCl₃) δ 7.61 (d, *J* = 8.5 Hz, 2H), 7.38 – 7.26 (m, 9H), 7.01 – 6.95 (m, 2H), 2.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 162.3 (d, *J* = 248.0 Hz), 145.0, 138.8, 133.5 (d, *J* = 8.0 Hz), 132.9, 129.5, 129.1, 128.3, 128.2, 126.2, 118.6, 115.5 (d, *J* = 22.0 Hz), 82.6, 69.3, 21.6; IR (neat): 3067, 2923, 2243, 1598, 1509, 1374, 1176, 837, 692, 581; HRESIMS Calcd for [C₂₁H₁₆FNNaO₂S]⁺ (M + Na⁺) 388.0778, found 388.0779.

N-methyl-2-nitro-*N*-(phenylethyynyl)benzenesulfonamide (**1n**)



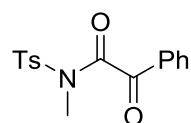
Compound **1n** was prepared in 58% yield (183.3 mg) according to the above known procedure.¹ Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 8.14 (dd, *J* = 1.2 Hz, *J* = 7.2 Hz, 1H), 7.80 – 7.63 (m, 3H), 7.39 – 7.31 (m, 2H), 7.30 – 7.22 (m, 3H), 3.36 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 147.7, 134.8, 131.7, 131.3, 131.2, 129.5, 128.1, 128.0, 124.2, 121.8, 82.1, 70.0, 39.5; IR (neat): 3021, 2933, 2237, 1360, 1163, 960, 783, 757, 693, 551, 516; HRESIMS Calcd for [C₁₅H₁₂N₂NaO₄S]⁺ (M + Na⁺) 339.0410, found 339.0412.



General procedure for the synthesis of α-keto imides **2**:

Cu(CH₃CN)₄PF₆ (0.02 mmol, 7.5 mg) was added to a mixture of the ynamide **1** (0.20 mmol) and 8-methylquinoline *N*-oxide **3d** (95.5 mg, 0.60 mmol) in DCE (4.0 mL) at room temperature. Then, the reaction mixture was stirred at 80 °C and the progress of the reaction was monitored by TLC. The reaction typically took 5 h. Upon completion, the mixture was concentrated and the residue was purified by chromatography on silica gel (eluent: hexanes/ethyl acetate) to afford the desired α-keto imide **2**.

N-methyl-2-oxo-2-phenyl-*N*-tosylacetamide (**2a**)

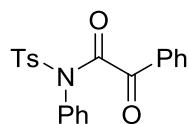


2a

Compound **2a** was prepared in 86% yield (81.9 mg) according to the general procedure (Table 2, entry 1). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.94 (d, *J*

$= 7.2$ Hz, 2H), 7.88 (d, $J = 8.0$ Hz, 2H), 7.67 – 7.63 (m, 1H), 7.57 – 7.50 (m, 2H), 7.39 (d, $J = 8.0$ Hz, 2H), 3.24 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 188.0, 167.2, 145.9, 134.4, 133.4, 132.7, 130.1, 129.6, 128.8, 128.3, 30.7, 21.7; IR (neat): 2917, 2849, 1681(s), 1372, 1232, 1166, 947, 717, 664, 594; HRESIMS Calcd for $[\text{C}_{16}\text{H}_{15}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 340.0614, found 340.0615.

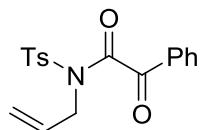
2-oxo-*N*,2-diphenyl-*N*-tosylacetamide (2b)



2b

Compound **2b** was prepared in 89% yield (101.3 mg) according to the general procedure (Table 2, entry 2). Pale yellow solid (mp 152–154 °C). ^1H NMR (500 MHz, CDCl_3) δ 7.93 (d, $J = 7.5$ Hz, 2H), 7.75 (d, $J = 8.0$ Hz, 2H), 7.66 – 7.62 (m, 1H), 7.54 – 7.49 (m, 2H), 7.43 – 7.32 (m, 5H), 7.13 (d, $J = 7.5$ Hz, 2H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.6, 166.7, 145.9, 134.6, 134.1, 133.5, 132.7, 130.6, 130.2, 129.7, 129.6, 129.4, 129.0, 128.9, 21.7; IR (neat): 2924, 2853, 1682(s), 1372, 1230, 1175, 1088, 695, 665, 600; HRESIMS Calcd for $[\text{C}_{21}\text{H}_{17}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 402.0770, found 402.0773.

N-allyl-2-oxo-2-phenyl-*N*-tosylacetamide (2c)

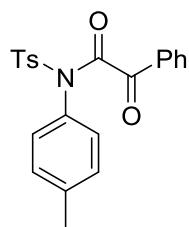


2c

Compound **2c** was prepared in 74% yield (76.2 mg) according to the general procedure (Table 2, entry 3). Pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 8.00 – 7.85 (m, 4H), 7.68 – 7.60 (m, 1H), 7.58 – 7.48 (m, 2H), 7.41 – 7.33 (m, 2H), 5.83 –

5.67 (m, 1H), 5.25 (d, $J = 17.0$ Hz, 1H), 5.16 (d, $J = 10.0$ Hz, 1H), 4.35 (d, $J = 5.5$ Hz, 2H), 2.46 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 187.7, 166.9, 145.8, 134.4(2), 134.3(9), 132.8, 130.6, 130.0, 129.7, 128.8, 128.6, 119.5, 47.2, 21.7; IR (neat): 3039, 2923, 1682(s), 1596, 1372, 1153, 821, 791, 716, 589; HRESIMS Calcd for $[\text{C}_{18}\text{H}_{17}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 366.0770, found 366.0772.

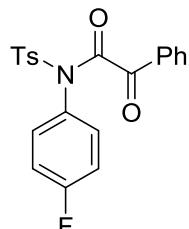
2-oxo-2-phenyl-*N*-(*p*-tolyl)-*N*-tosylacetamide (2d)



2d

Compound **2d** was prepared in 91% yield (107.4 mg) according to the general procedure (Table 2, entry 4). Pale yellow solid (mp 164–166 °C). ^1H NMR (400 MHz, CDCl_3) δ 7.91 (d, $J = 7.6$ Hz, 2H), 7.76 (d, $J = 8.0$ Hz, 2H), 7.64 – 7.59 (m, 1H), 7.52 – 7.46 (m, 2H), 7.33 (d, $J = 8.0$ Hz, 2H), 7.15 (d, $J = 8.0$ Hz, 2H), 7.00 (d, $J = 8.0$ Hz, 2H), 2.45 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.6, 166.7, 145.7, 140.5, 134.5, 134.1, 132.7, 130.7, 130.2, 130.1, 129.7, 129.5, 129.0, 128.8, 21.7, 21.2; IR (neat): 3066, 2923, 1682(s), 1596, 1371, 1172, 957, 715, 655, 590; HRESIMS Calcd for $[\text{C}_{22}\text{H}_{19}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 416.0927, found 416.0926.

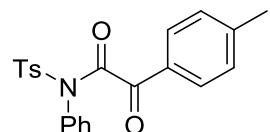
***N*-(4-fluorophenyl)-2-oxo-2-phenyl-*N*-tosylacetamide (2e)**



2e

Compound **2e** was prepared in 83% yield (99.0 mg) according to the general procedure (Table 2, entry 5). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, J = 7.2 Hz, 2H), 7.74 (d, J = 8.0 Hz, 2H), 7.67 – 7.62 (m, 1H), 7.56 – 7.49 (m, 2H), 7.38 – 7.30 (m, 3H), 7.17 – 7.12 (m, 1H), 6.93 – 6.87 (m, 2H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.3, 166.5, 162.6 (d, J = 248.0 Hz), 146.2, 134.7, 133.8, 132.5, 130.4 (d, J = 9.0 Hz), 129.8, 129.6, 129.0, 126.4 (d, J = 3.0 Hz), 118.2, 118.0, 117.5 (d, J = 20.0 Hz), 21.7; IR (neat): 3069, 2924, 1682(s), 1597, 1373, 1174, 958, 714, 656, 590; HRESIMS Calcd for $[\text{C}_{21}\text{H}_{16}\text{FNNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 420.0676, found 420.0679.

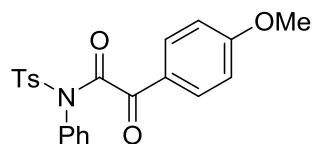
2-oxo-*N*-phenyl-2-(*p*-tolyl)-*N*-tosylacetamide (2f)



2f

Compound **2f** was prepared in 87% yield (102.7 mg) according to the general procedure (Table 2, entry 6). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, J = 7.2 Hz, 2H), 7.76 (d, J = 7.6 Hz, 2H), 7.44 – 7.26 (m, 7H), 7.14 – 7.11 (m, 2H), 2.45 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.2, 166.7, 145.8, 145.7, 134.1, 133.5, 130.5, 130.2, 130.1, 129.7, 129.6, 129.3, 129.0, 21.8, 21.7; IR (neat): 3066, 2923, 1678(s), 1605, 1372, 1177, 1033, 694, 669, 591; HRESIMS Calcd for $[\text{C}_{22}\text{H}_{19}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 416.0927, found 416.0925.

2-(4-methoxyphenyl)-2-oxo-*N*-phenyl-*N*-tosylacetamide (2g)



2g

Compound **2g** was prepared in 82% yield (100.7 mg) according to the general procedure (Table 2, entry 7). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.88 (d, J = 8.8 Hz, 2H), 7.78 (d, J = 8.4 Hz, 2H), 7.40 – 7.32 (m, 5H), 7.13 (d, J = 6.8 Hz, 2H), 6.98 – 6.93 (m, 2H), 3.87 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 186.2, 166.8, 164.7, 145.7, 134.2, 133.6, 132.0, 130.5, 130.1, 129.7, 129.3, 129.0, 125.6, 114.3, 55.6, 21.7; IR (neat): 3068, 2935, 1668(s), 1598, 1371, 1172, 1030, 816, 695, 667; HRESIMS Calcd for $[\text{C}_{22}\text{H}_{19}\text{NNaO}_5\text{S}]^+$ ($\text{M} + \text{Na}^+$) 432.0876, found 432.0876.

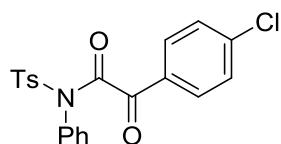
2-(4-fluorophenyl)-2-oxo-N-phenyl-N-tosylacetamide (2h)



2h

Compound **2h** was prepared in 85% yield (101.3 mg) according to the general procedure (Table 2, entry 8). Pale yellow solid (mp 159–161 °C). ^1H NMR (400 MHz, CDCl_3) δ 7.99 – 7.94 (m, 2H), 7.73 (d, J = 8.4 Hz, 2H), 7.44 – 7.32 (m, 5H), 7.23 – 7.17 (m, 2H), 7.12 (d, J = 7.6 Hz, 2H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 186.0, 166.5 (d, J = 256.0 Hz), 166.4, 146.0, 133.9, 133.4, 132.4 (d, J = 10.0 Hz), 130.5, 130.2, 129.8, 129.5, 129.2, 129.0, 116.3 (d, J = 23.0 Hz), 21.7; IR (neat): 3070, 2925, 1682(s), 1598, 1372, 1237, 1033, 957, 669, 590; HRESIMS Calcd for $[\text{C}_{21}\text{H}_{16}\text{FNNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 420.0676, found 420.0675.

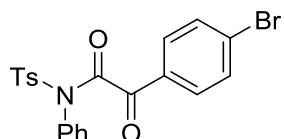
2-(4-chlorophenyl)-2-oxo-N-phenyl-N-tosylacetamide (2i)



2i

Compound **2i** was prepared in 82% yield (101.8 mg) according to the general procedure (Table 2, entry 9). Pale yellow solid (mp 157–159 °C). ¹H NMR (400 MHz, CDCl₃) δ 7.88 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 7.44 – 7.31 (m, 5H), 7.12 (d, *J* = 7.2 Hz, 2H), 2.45 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 186.5, 166.3, 146.0, 133.8, 133.3, 132.3, 131.5, 130.9, 130.4, 130.2, 130.0, 129.8, 129.5, 129.0, 21.7; IR (neat): 3068, 2927, 1676(s), 1590, 1372, 1173, 1034, 759, 666, 579; HRESIMS Calcd for [C₂₁H₁₆ClNNaO₄S]⁺ (M + Na⁺) 436.0381, found 436.0380.

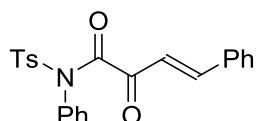
2-(4-bromophenyl)-2-oxo-N-phenyl-N-tosylacetamide (2j)



2j

Compound **2j** was prepared in 79% yield (108.5 mg) according to the general procedure (Table 2, entry 10). Pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.80 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.44 – 7.31 (m, 5H), 7.11 (d, *J* = 7.2 Hz, 2H), 2.45 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 186.5, 166.3, 146.0, 133.9, 133.4, 132.4, 131.6, 130.9, 130.5, 130.3, 130.1, 129.8, 129.5, 129.0, 21.7; IR (neat): 3066, 2924, 1682(s), 1588, 1372, 1174, 957, 753, 665, 578; HRESIMS Calcd for [C₂₁H₁₆BrNNaO₄S]⁺ (M + Na⁺) 479.9876, found 479.9877.

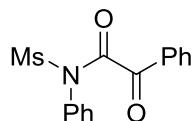
(E)-2-oxo-N,4-diphenyl-N-tosylbut-3-enamide (2k)



2k

Compound **2k** was prepared in 80% yield (97.3 mg) according to the general procedure (Table 2, entry 11). Orange oil. ^1H NMR (400 MHz, CDCl_3) δ 7.80 (d, $J=8.4$ Hz, 2H), 7.61 (d, $J=16.4$ Hz, 1H), 7.58 – 7.54 (m, 2H), 7.48 – 7.38 (m, 6H), 7.35 (d, $J=8.0$ Hz, 2H), 7.18 – 7.14 (m, 2H), 6.83 (d, $J=16.4$ Hz, 1H), 2.48 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.0, 166.4, 148.5, 145.8, 134.3, 133.8(0), 133.7(5), 131.6, 130.5, 130.2, 129.7, 129.4, 129.1(1), 129.0(6), 128.9, 122.4, 21.8; IR (neat): 3063, 2924, 1681(s), 1596, 1372, 1174, 1088, 814, 694, 584; HRESIMS Calcd for $[\text{C}_{24}\text{H}_{20}\text{NaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 428.0927, found 428.0928.

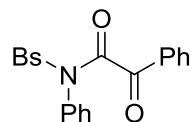
N-(methylsulfonyl)-2-oxo-N,2-diphenylacetamide (2l)



2l

Compound **2l** was prepared in 86% yield (78.3 mg) according to the general procedure (Table 2, entry 12). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.94 (d, $J=7.2$ Hz, 2H), 7.67 – 7.61 (m, 1H), 7.52 (d, $J=8.0$ Hz, 2H), 7.49 – 7.45 (m, 3H), 7.39 – 7.35 (m, 2H), 3.40 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 187.9, 167.1, 134.9, 133.5, 132.3, 130.4, 130.0, 129.8 (4), 129.7 (8), 128.9, 41.2; IR (neat): 2930, 1693(s), 1596, 1369, 1180, 973, 769, 715, 694, 597; HRESIMS Calcd for $[\text{C}_{15}\text{H}_{13}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 326.0457, found 326.0454.

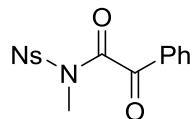
N-((4-bromophenyl)sulfonyl)-2-oxo-N,2-diphenylacetamide (2m)



2m

Compound **2m** was prepared in 80% yield (106.6 mg) according to the general procedure (Table 2, entry 13). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.92 (d, $J = 7.6$ Hz, 2H), 7.76 – 7.60 (m, 5H), 7.54 – 7.49 (m, 2H), 7.45 – 7.36 (m, 3H), 7.14 (d, $J = 7.2$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.4, 166.4, 136.0, 134.8, 133.1, 132.5, 132.4, 130.4(4), 130.4(2), 130.3(9), 130.1, 129.6, 129.0; IR (neat): 3091, 2924, 1695(s), 1376, 1177, 1009, 747, 610, 598, 557; HRESIMS Calcd for $[\text{C}_{20}\text{H}_{14}\text{BrNNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 465.9719, found 465.9723.

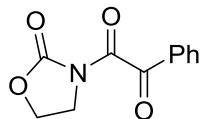
N-methyl-N-((2-nitrophenyl)sulfonyl)-2-oxo-2-phenylacetamide (2n)



2n

Compound **2n** was prepared in 93% yield (97.2 mg) according to the general procedure (Table 2, entry 14). Pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 8.31 – 8.22 (m, 1H), 7.91 (d, $J = 7.5$ Hz, 2H), 7.85 – 7.79 (m, 3H), 7.69 – 7.64 (m, 1H), 7.55 – 7.49 (m, 2H), 3.41 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.6, 166.8, 148.4, 135.3, 135.1, 132.5, 132.2, 132.0, 131.0, 129.8, 129.0, 124.7, 32.4; IR (neat): 3065, 2924, 1789(s), 1696(s), 1396, 1239, 1124, 1032, 722, 656; HRESIMS Calcd for $[\text{C}_{15}\text{H}_{12}\text{N}_2\text{NaO}_6\text{S}]^+$ ($\text{M} + \text{Na}^+$) 371.0308, found 371.0304.

1-(2-oxooxazolidin-3-yl)-2-phenylethane-1,2-dione (2o)

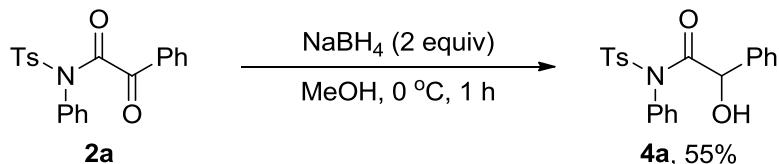


2o

Compound **2o** was prepared in 66% yield (43.4 mg) according to the general procedure (Table 2, entry 15). Pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.91 –

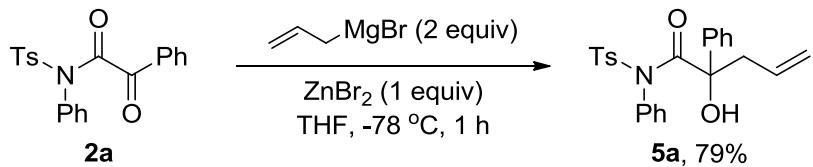
7.86 (m, 2H), 7.68 – 7.62 (m, 1H), 7.55 – 7.48 (m, 2H), 4.60 (t, J = 8.0 Hz, 2H), 4.17 (t, J = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 187.8, 166.5, 153.1, 134.8, 132.4, 129.3, 129.0, 64.0, 40.9; IR (neat): 3099, 2924, 1686(s), 1545, 1377, 1229, 945, 734, 716, 604; HRESIMS Calcd for $[\text{C}_{11}\text{H}_9\text{NNaO}_4]^+$ ($\text{M} + \text{Na}^+$) 242.0424, found 242.0425.

2-hydroxy-N,2-diphenyl-N-tosylacetamide (4a)



Compound **4a** was prepared in 55% yield (41.9 mg, 0.2 mmol scale) according to the known procedure (Scheme 2, entry 1).⁵ Pale yellow oil. ^1H NMR (500 MHz, CDCl_3) δ 7.91 (d, J = 8.0 Hz, 2H), 7.47 – 7.40 (m, 1H), 7.38 (d, J = 8.0 Hz, 2H), 7.33 – 7.23 (m, 3H), 7.20 – 7.15 (m, 2H), 6.90 – 6.76 (m, 2H), 6.73 (d, J = 7.5 Hz, 2H), 4.79 (s, 1H), 3.78 (s, 1H), 2.49 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.0, 145.5, 137.2, 135.4, 133.9, 130.2, 129.5, 129.3, 129.2, 128.8, 128.7, 127.5, 73.3, 21.7; IR (neat): 3477, 3066, 2925, 1702(s), 1489, 1365, 1172, 1087, 696, 569, 548; HRESIMS Calcd for $[\text{C}_{21}\text{H}_{19}\text{NNaO}_4\text{S}]^+$ ($\text{M} + \text{Na}^+$) 404.0927, found 404.0929.

2-hydroxy-N,2-diphenyl-N-tosylpent-4-enamide (5a)



Compound **5a** was prepared in 79% yield (66.6 mg, 0.2 mmol scale) according to the known procedure (Scheme 2, entry 2).⁶ Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, J = 8.4 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 7.29 – 7.23 (m, 1H), 7.19 – 7.13 (m, 1H), 7.11 – 7.03 (m, 4H), 6.84 – 6.79 (m, 2H), 6.67 (d, J = 7.2 Hz, 2H), 5.69 – 5.57 (m, 1H), 5.15 – 5.07 (m, 2H), 3.30 (s, 1H), 2.90 (dd, J = 6.8 Hz, J = 14.0 Hz, 1H), 2.50 (dd, J = 7.6 Hz, J = 14.0 Hz, 1H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 173.4, 144.9, 140.7, 135.6, 134.3, 131.9, 131.3, 129.3(2), 129.3(0), 129.2, 128.2,

128.1, 127.6, 124.5, 120.7, 79.4, 44.7, 21.6; IR (neat): 3505, 3069, 2922, 1697(s), 1596, 1490, 1363, 1169, 1088, 696, 579, 551; HRESIMS Calcd for $[C_{24}H_{23}NNaO_4S]^+$ ($M + Na^+$) 444.1240, found 444.1243.

References:

1. Pan, F.; Li, X.-L.; Chen, X.-M.; Shu, C.; Ruan, P.-P.; Shen, C.-H.; Lu, X.; Ye, L.-W. *ACS Catal.* **2016**, *6*, 6055.
2. Zhou, A.-H.; He, Q.; Shu, C.; Yu, Y.-F.; Liu, S.; Zhao, T.; Zhang, W.; Lu, X.; Ye, L.-W. *Chem. Sci.* **2015**, *6*, 1265.
3. Li, L.; Shu, C.; Zhou, B.; Yu, Y.-F.; Xiao, X.-Y.; Ye, L.-W. *Chem. Sci.* **2014**, *5*, 4057.
4. Yao, B.; Liang, Z.; Niu, T.; Zhang, Y. *J. Org. Chem.* **2009**, *74*, 4630.
5. Ylijoki, K. E. O.; Kündig, E. P. *Chem. Commun.* **2011**, *47*, 10608.
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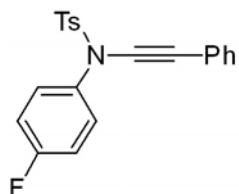
7.618
7.601

7.323
7.311

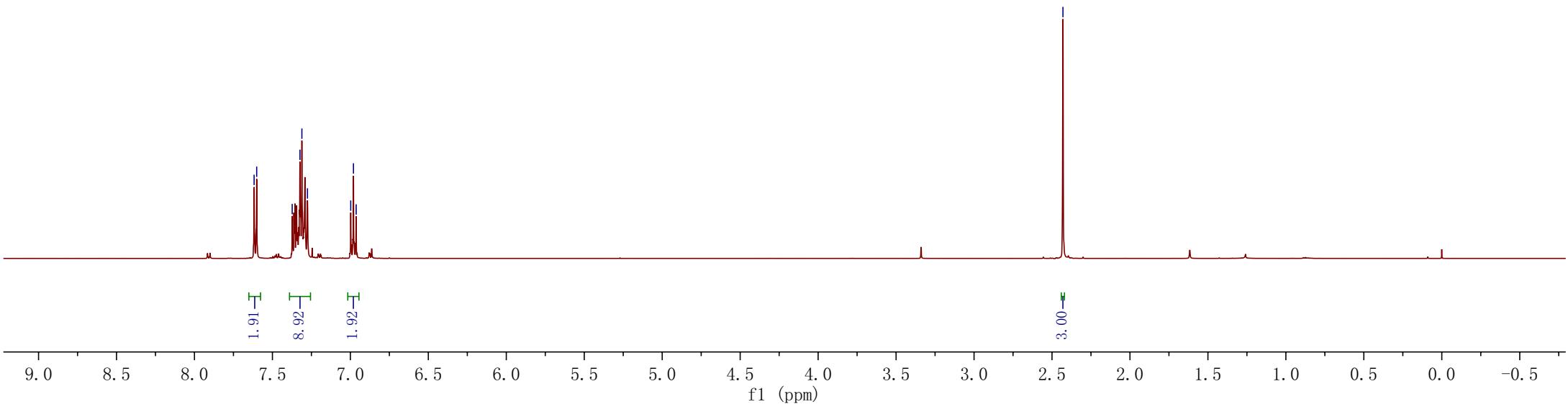
7.275
6.998

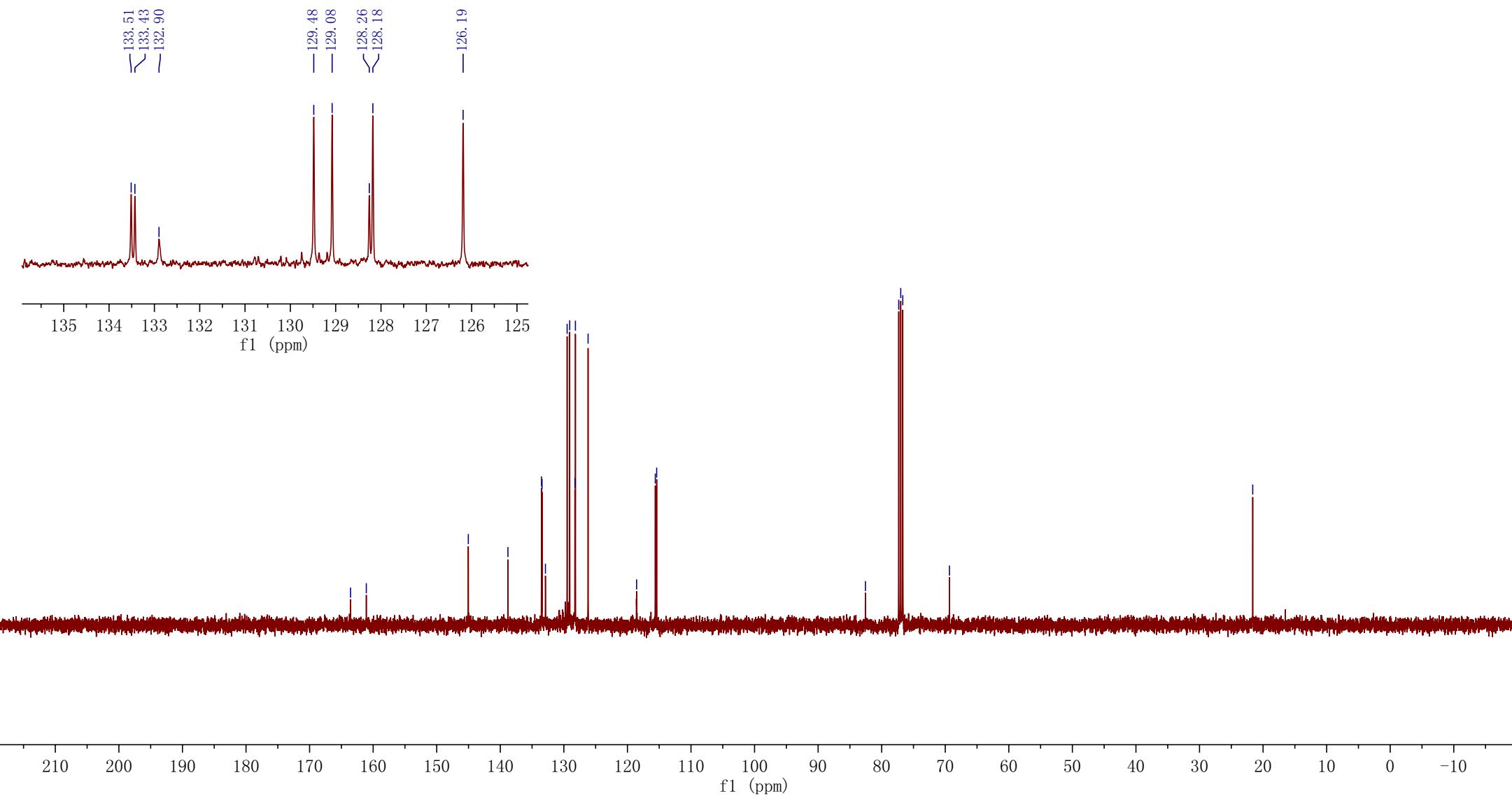
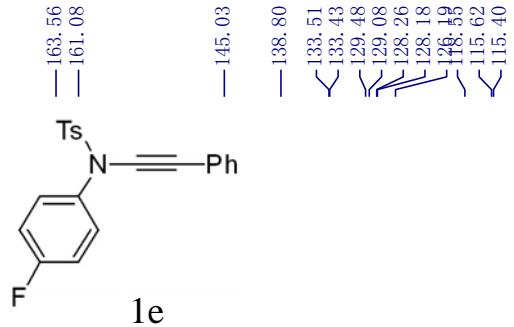
6.980
6.963

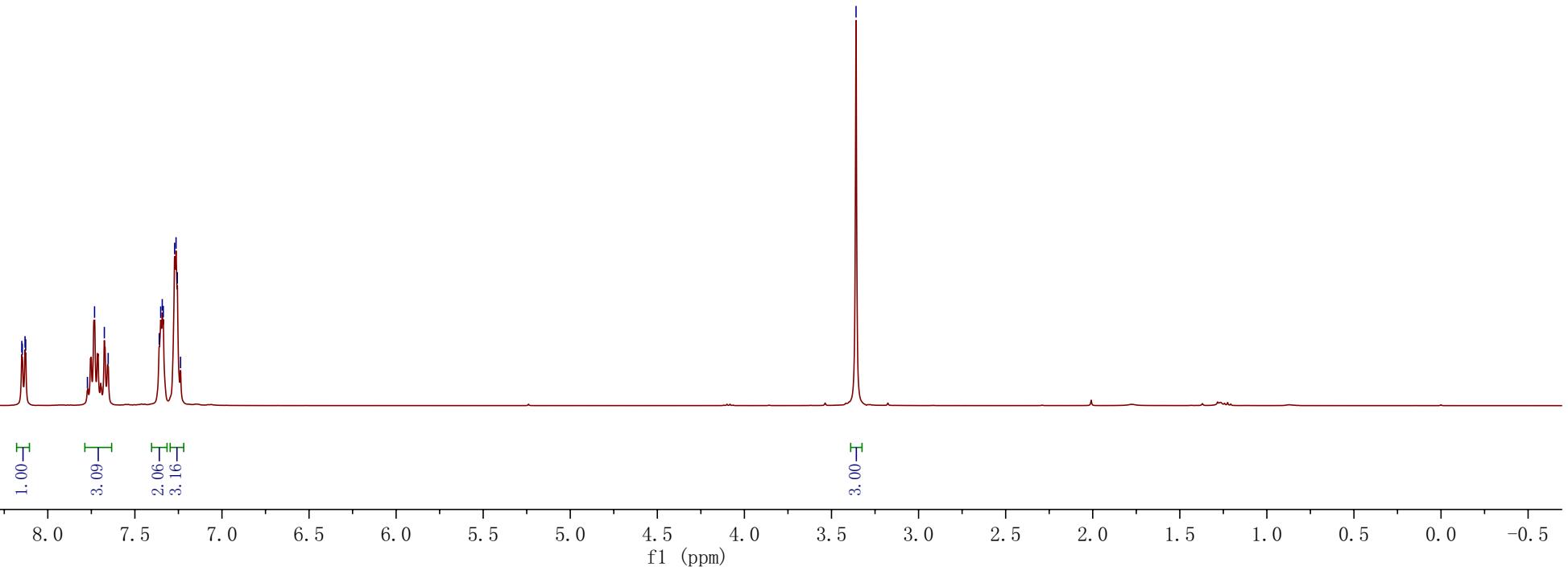
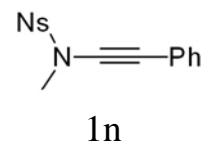
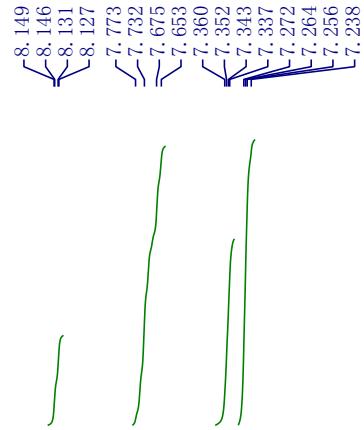
—2.430

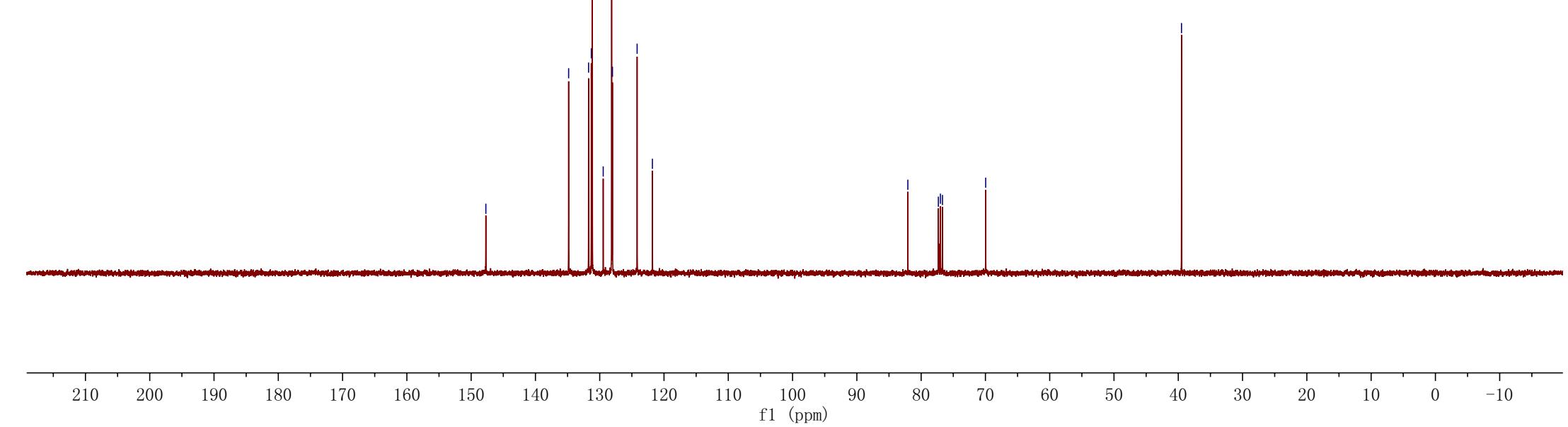
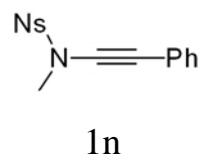


1e

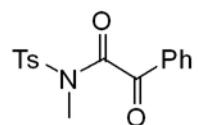
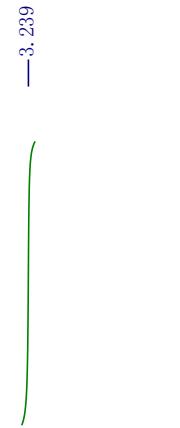
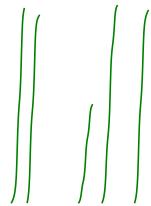




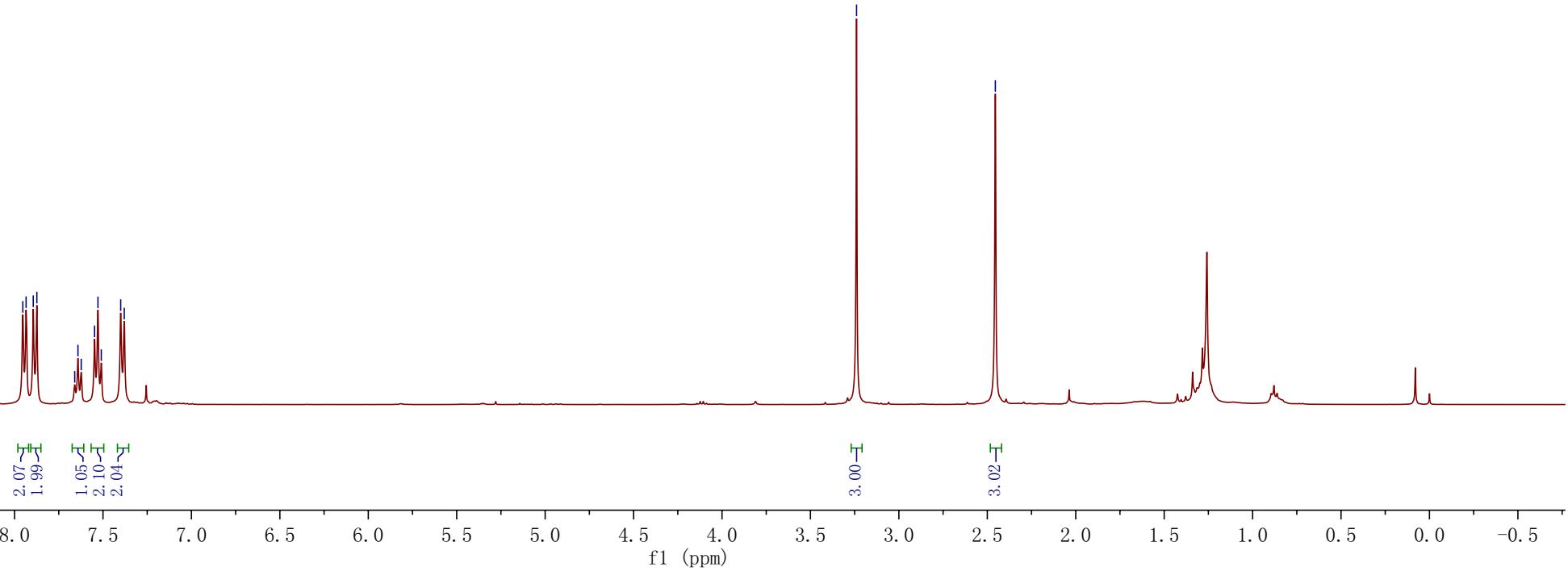


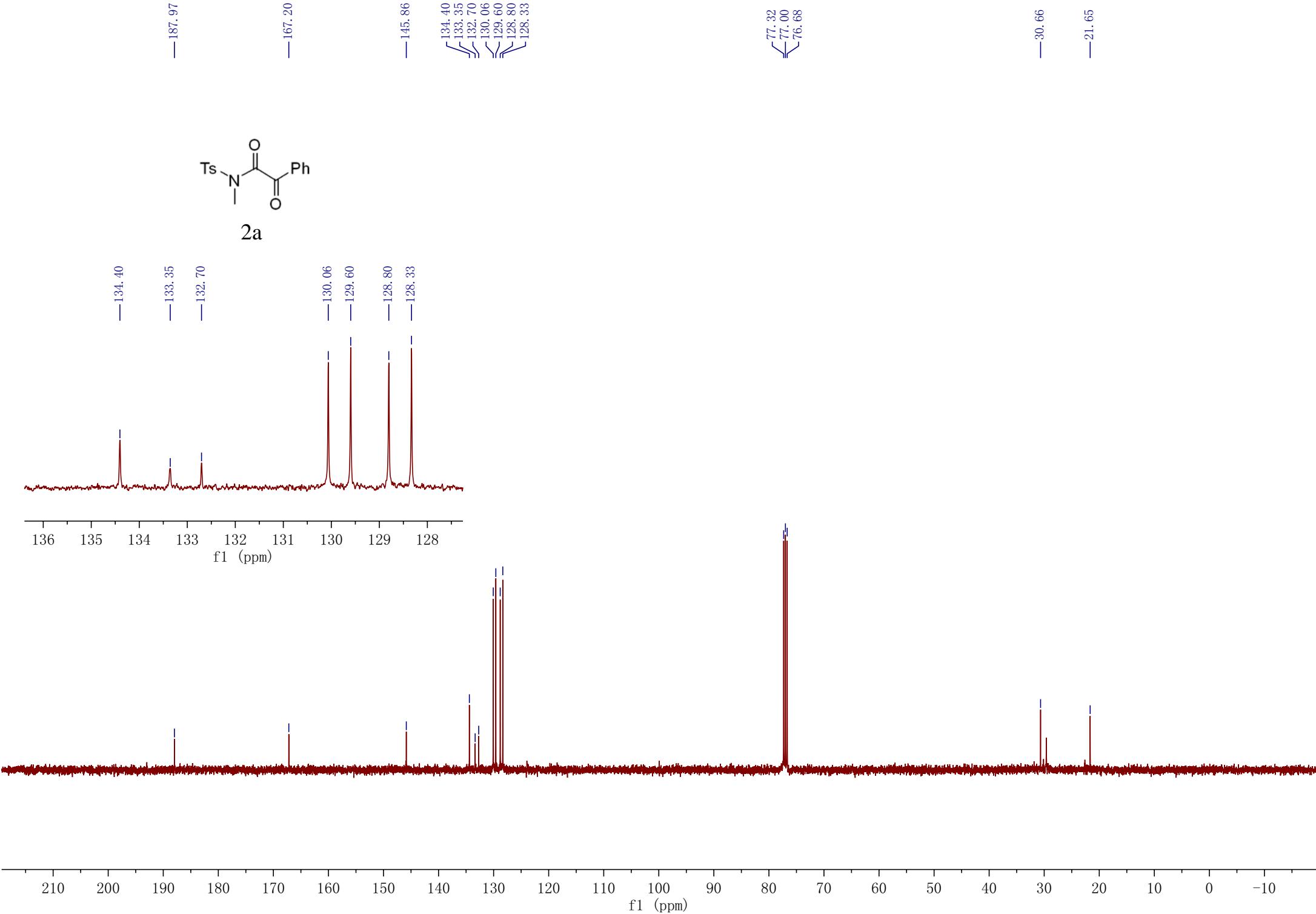


7.953
7.935
7.894
7.874
7.660
7.642
7.623
7.548
7.529
7.510
7.400
7.380



2a



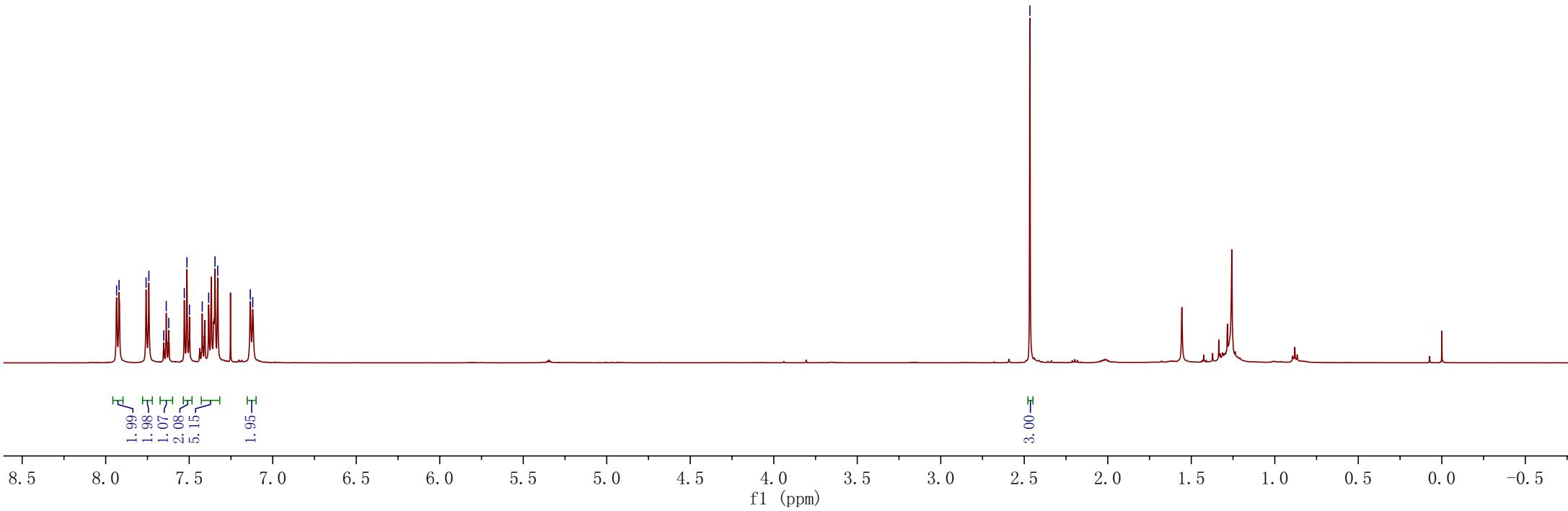


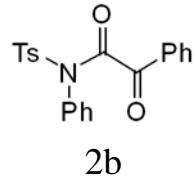
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7.920
7.758
7.742
7.652
7.637
7.623
7.529
7.514
7.498
7.422
7.384
7.346
7.329
7.134
7.119

2.466



2b





—134.58

—134.13

—133.50

—132.72

—166.67

—187.55

—145.85

—134.58

—134.13

—133.50

—132.72

—130.55

—130.17

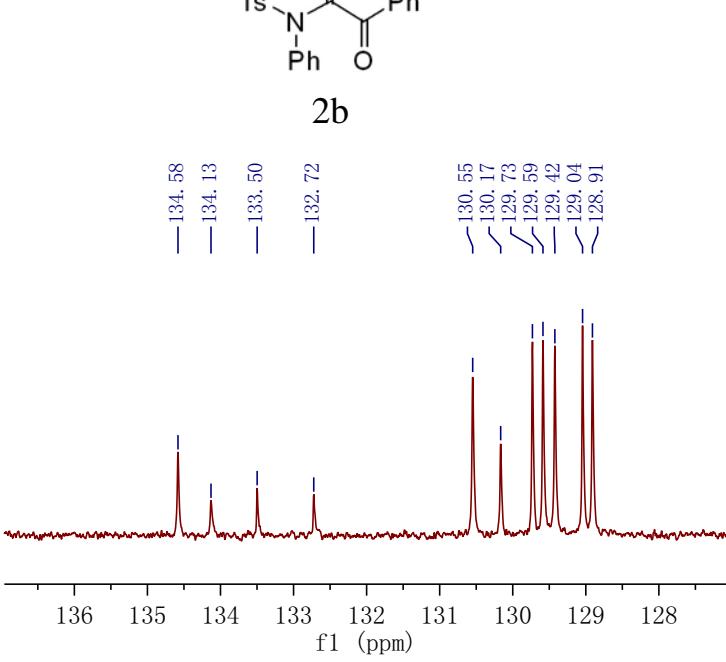
—129.73

—129.59

—129.42

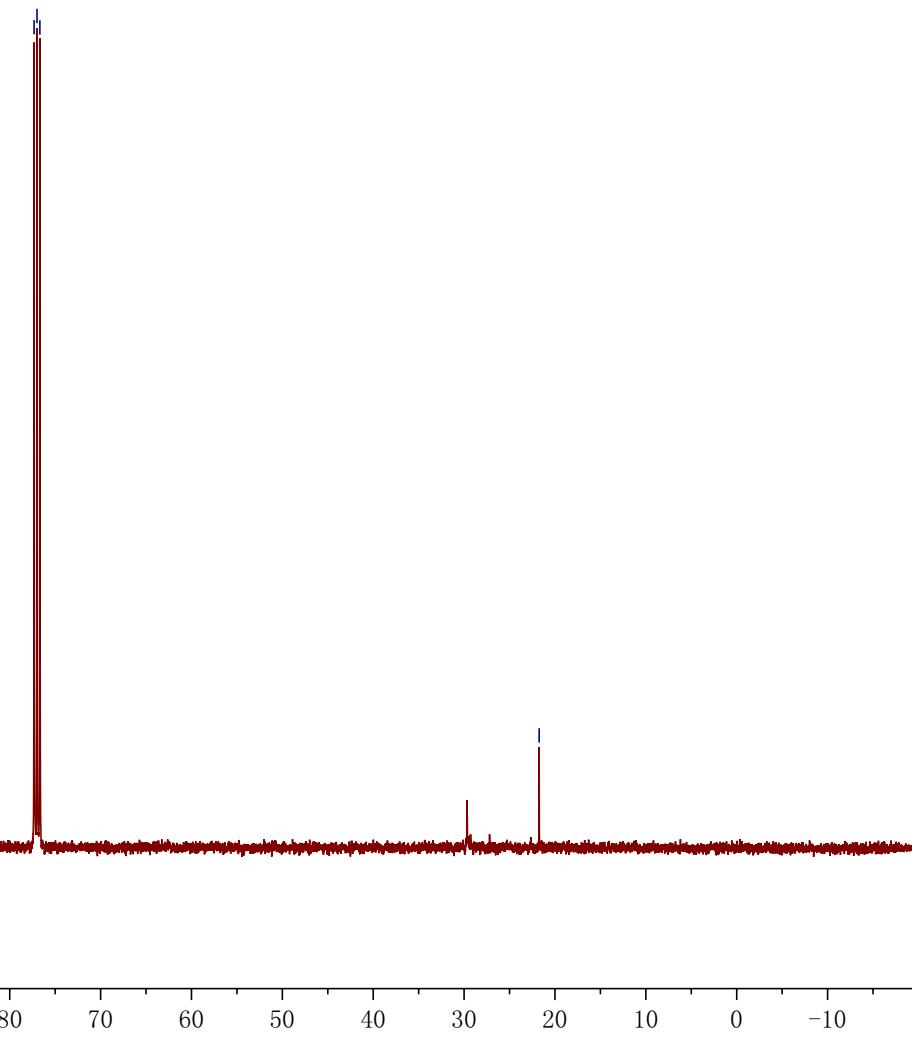
—129.04

—128.91



f1 (ppm)

—21.74



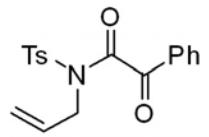
f1 (ppm)

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7.923
7.905
7.890
7.657
7.643
7.629
7.544
7.530
7.515
7.385
7.370
7.258

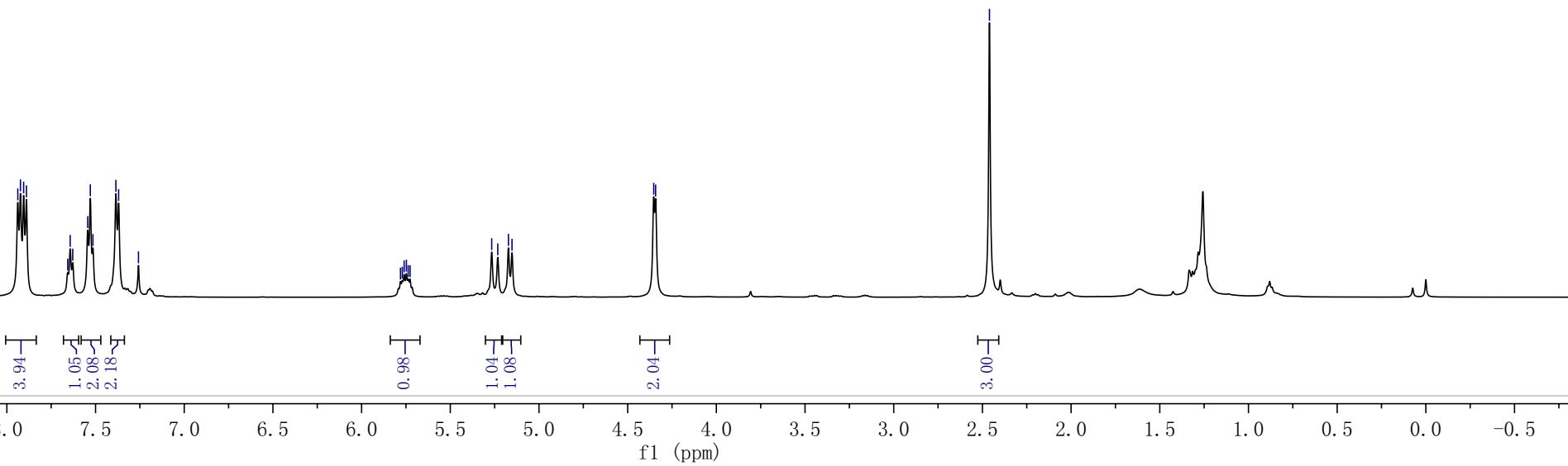
5.781
5.770
5.760
5.747
5.737
5.727
5.266
5.232
5.172
5.152

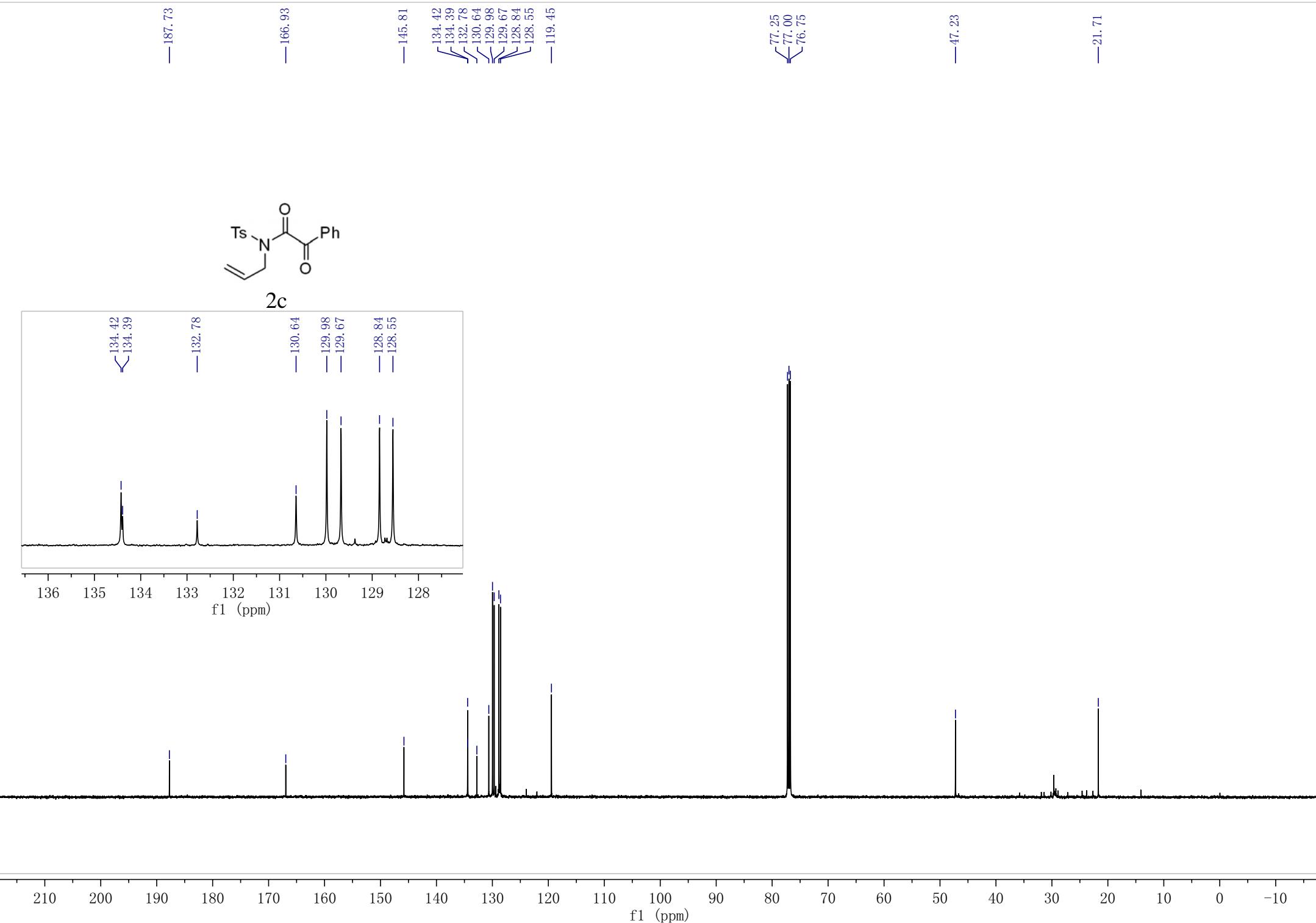
4.353
4.342

-2.460



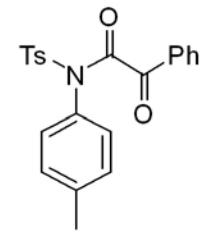
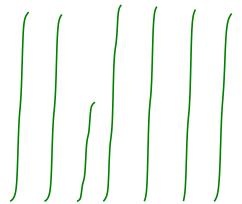
2c





7.923
7.904
7.769
7.749
7.635
7.617
7.598
7.512
7.493
7.474
7.336
7.316
7.163
7.143
7.009
6.989

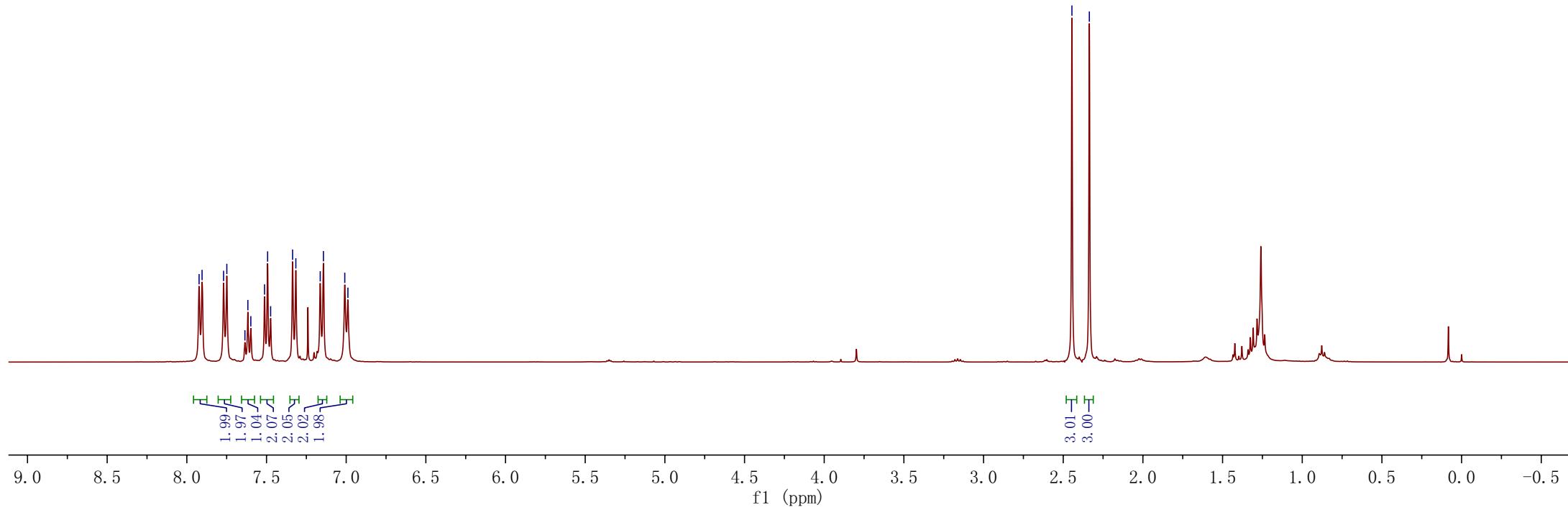
—2.445
—2.336

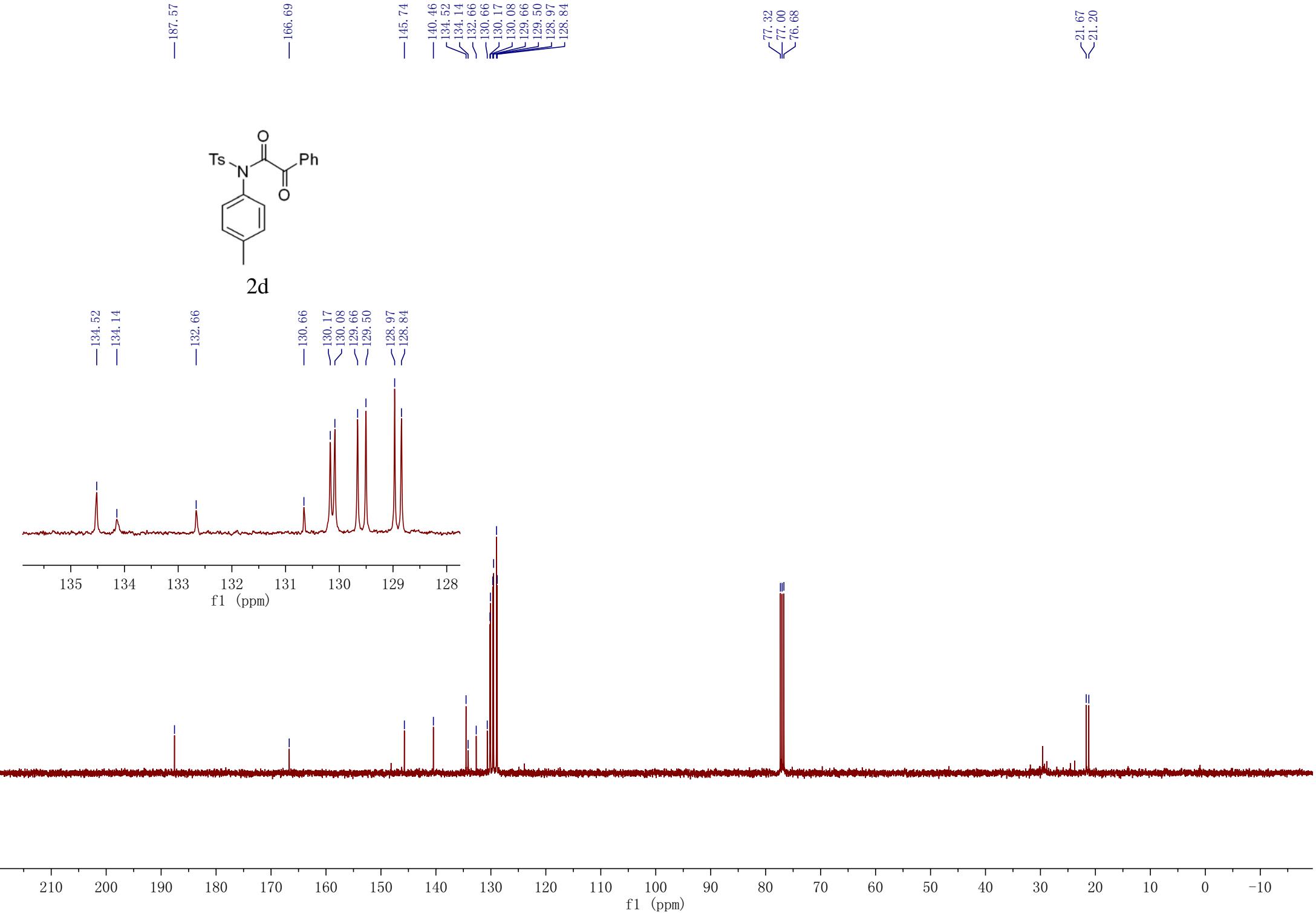


2d

1.99
1.97
1.04
2.07
2.05
2.02
1.98

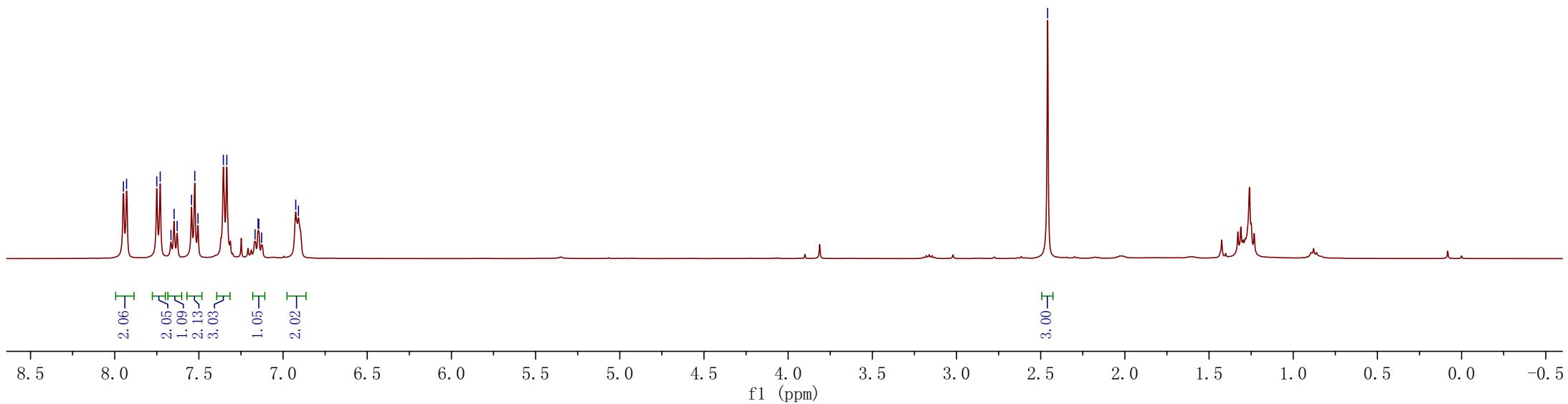
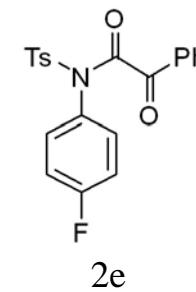
3.01
3.00

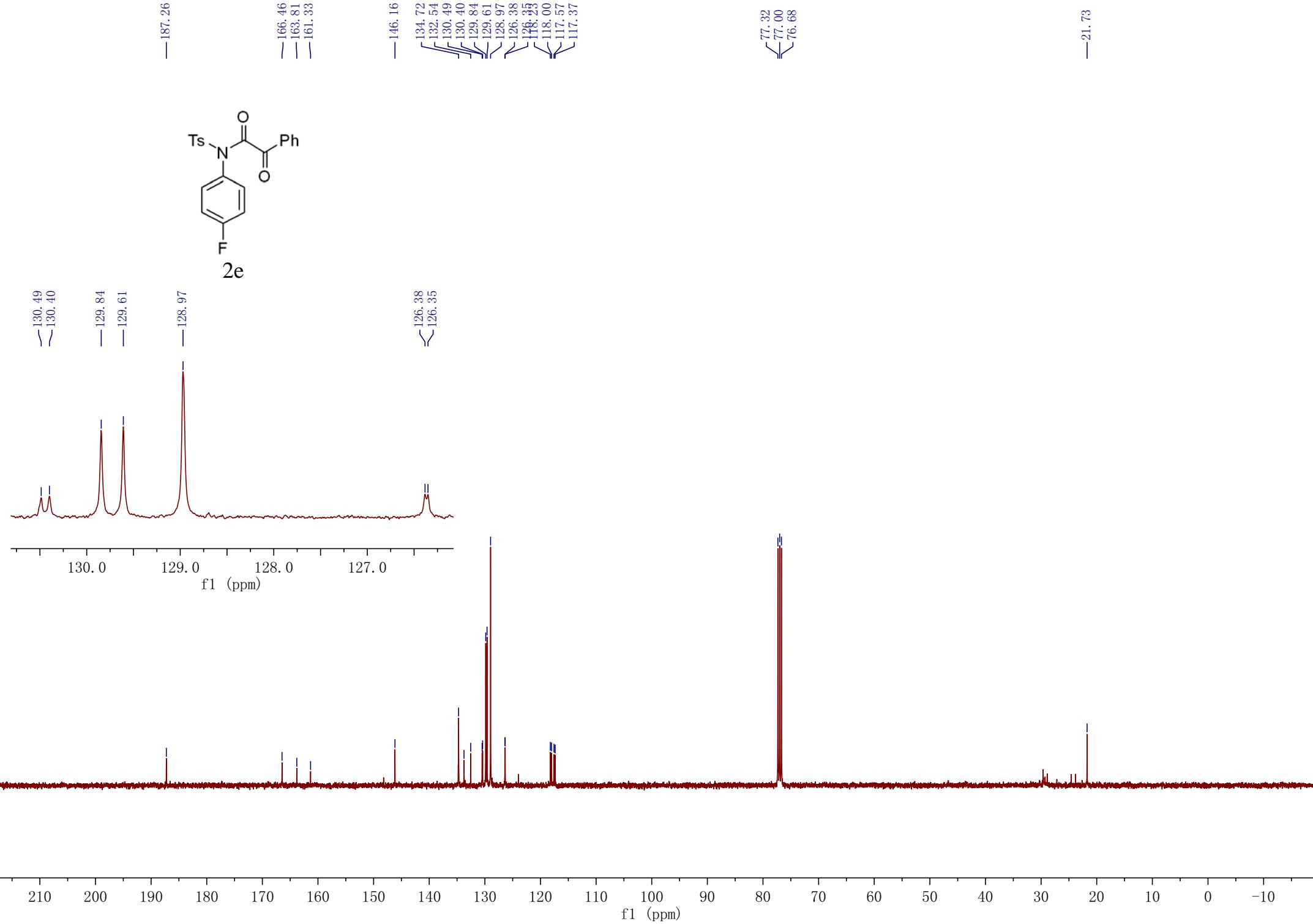




7.948
7.930
7.750
7.730
7.730
7.666
7.648
7.629
7.544
7.524
7.505
7.355
7.334
7.166
7.148
7.144
7.128
6.925
<6.909

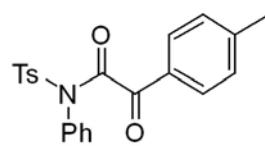
—2.459



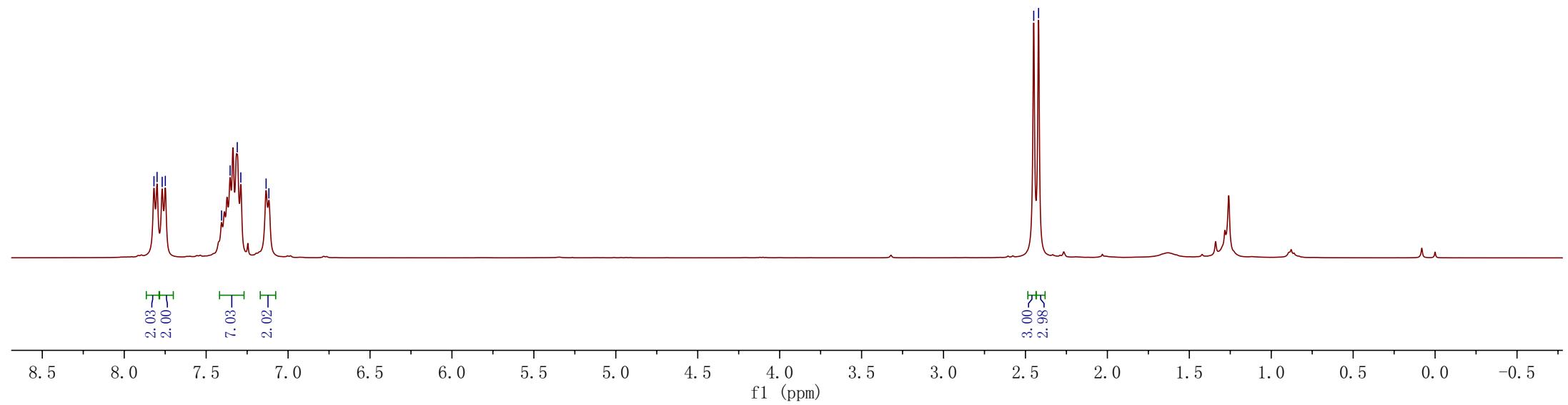


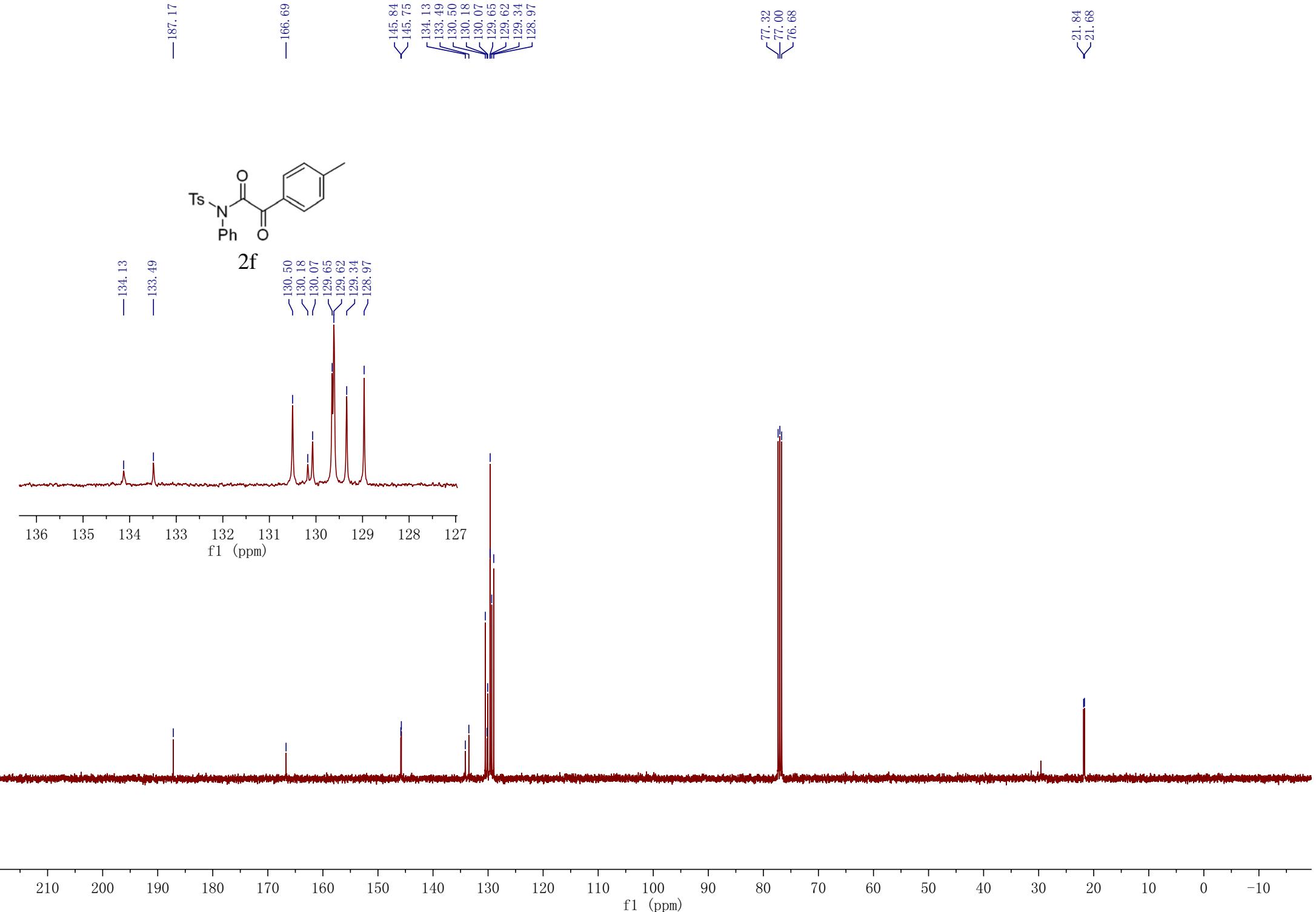
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7.800
7.768
7.749
7.406
7.354
7.310
7.289
7.134
7.117

2.449
2.420



2f

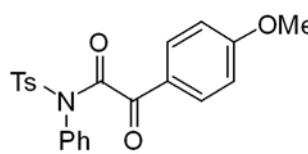




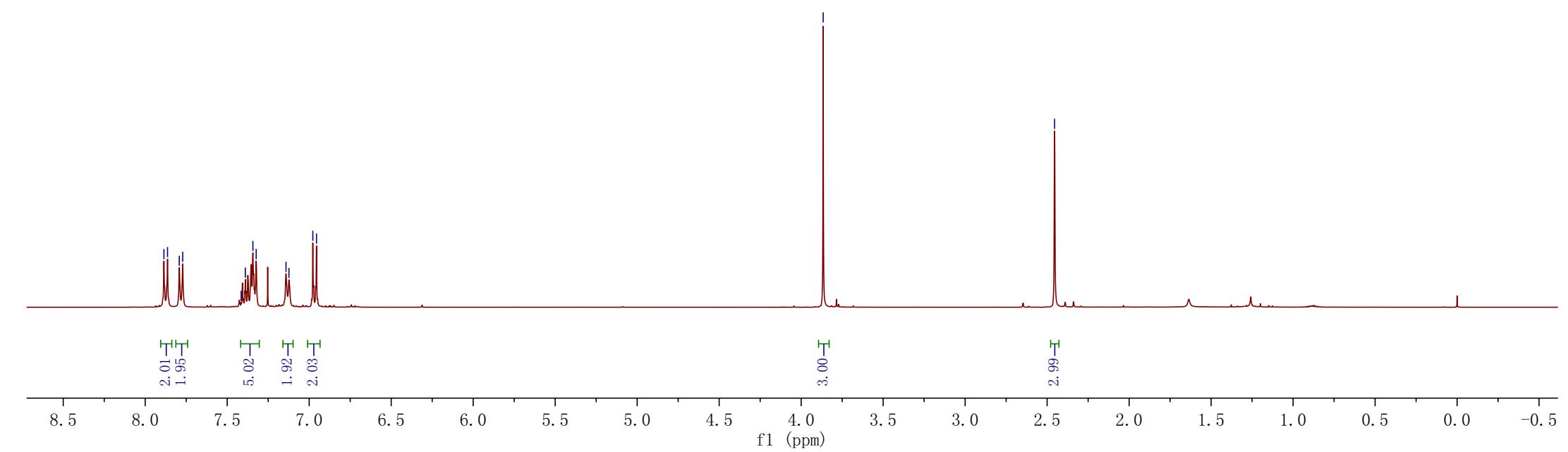
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7.864
7.792
7.771
7.415
7.389
7.343
7.323
7.141
7.124
6.978
6.956

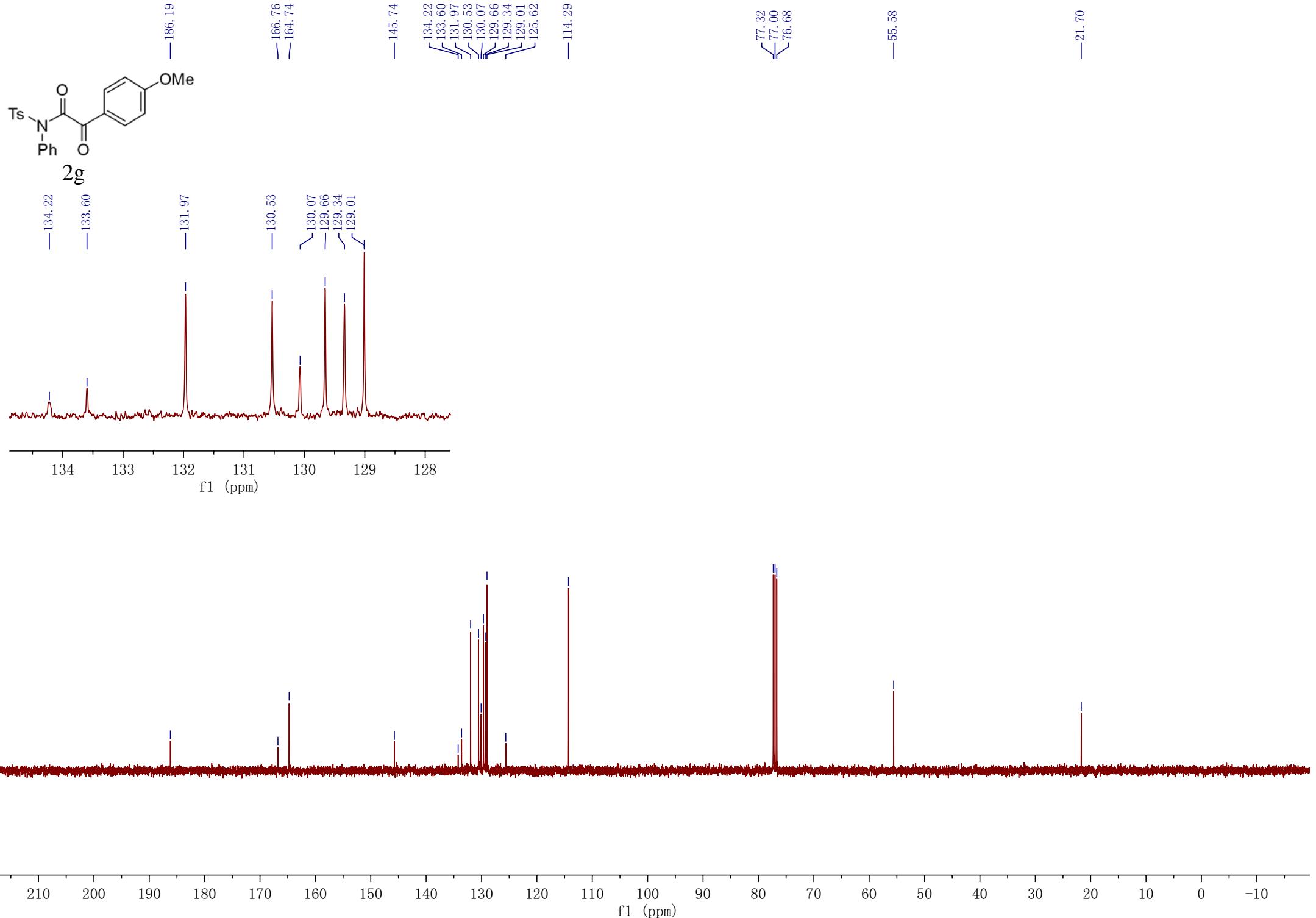
—3.866

—2.455



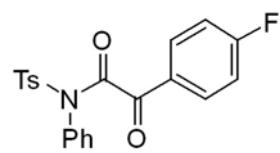
2g



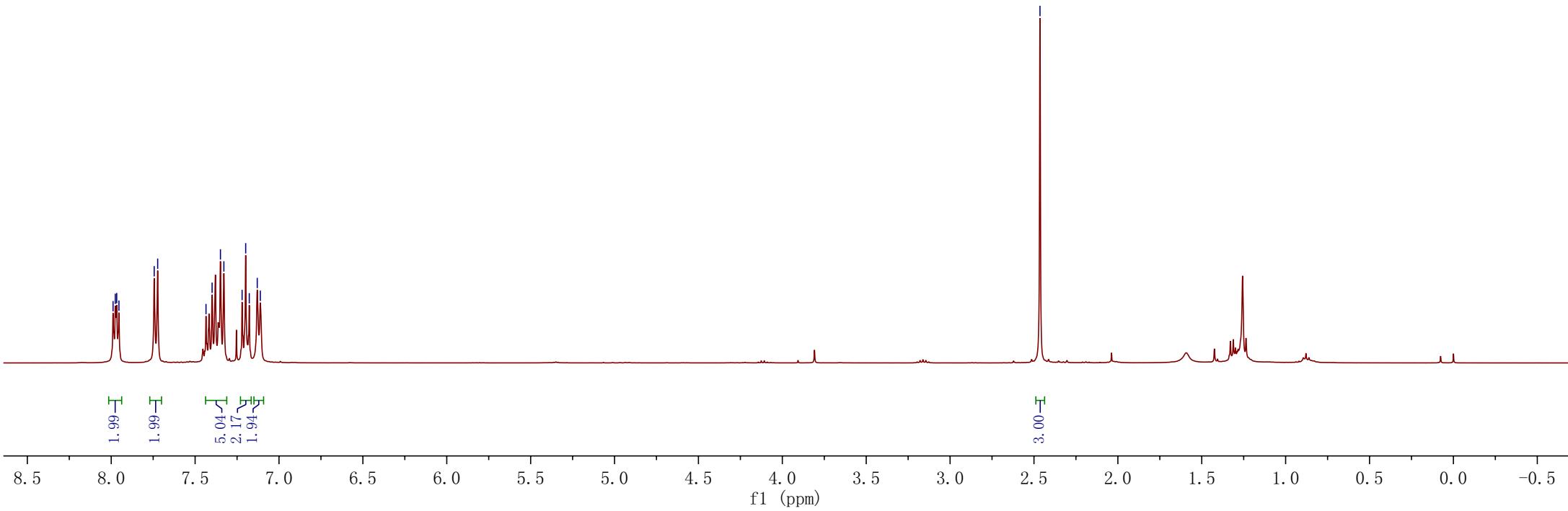


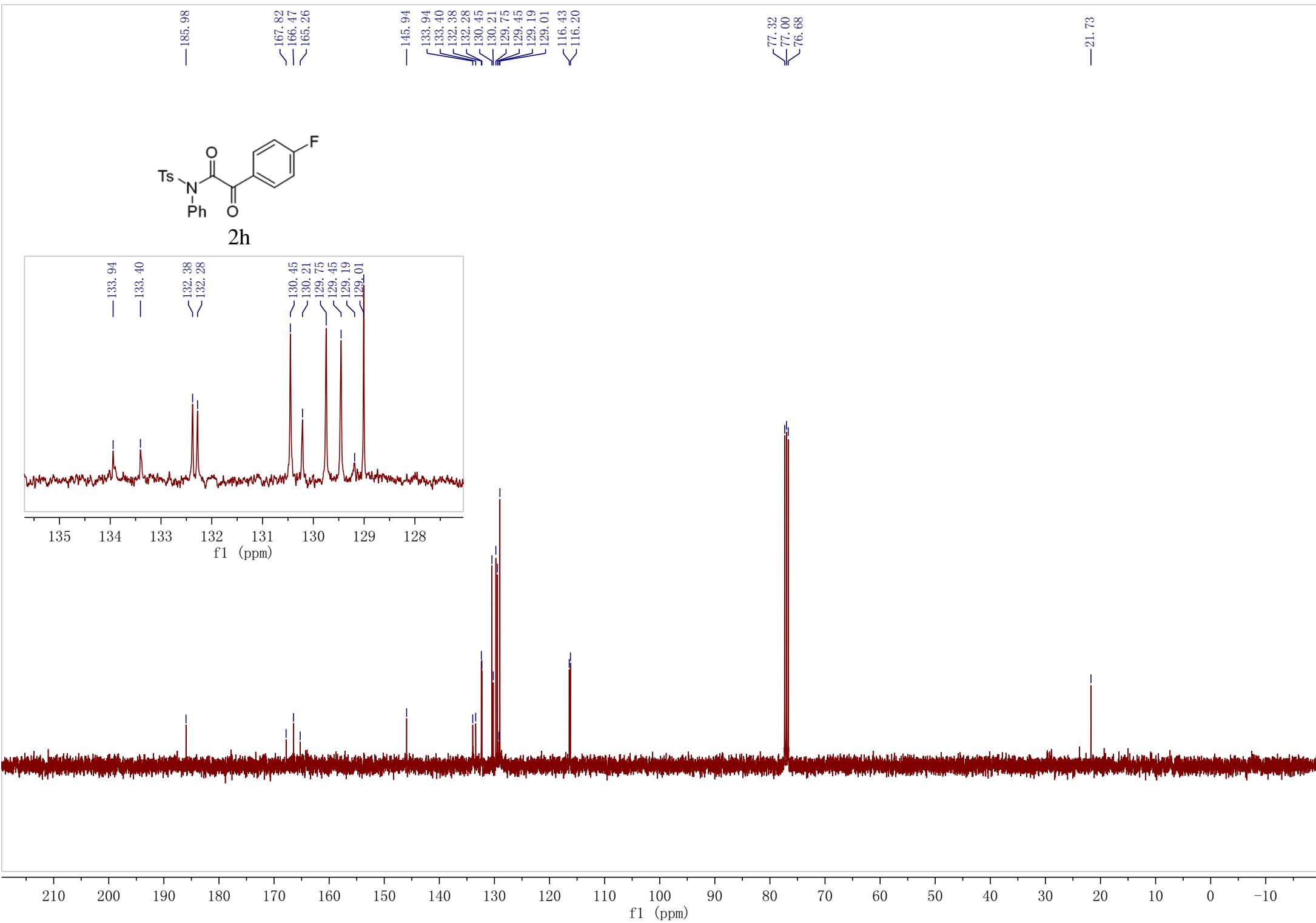
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7.967
7.954
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7.723
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7.399
7.349
7.329
7.220
7.199
7.177
7.130
7.111

—2.464



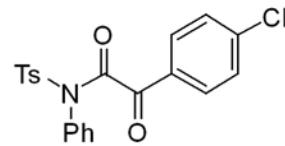
2h





~7.892
~7.871
~7.730
~7.709
~7.506
~7.485
~7.340
~7.320
~7.108

-2.453



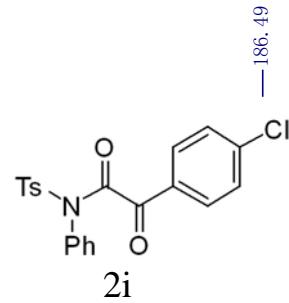
2i

2.01
1.98
2.03
4.99
1.97

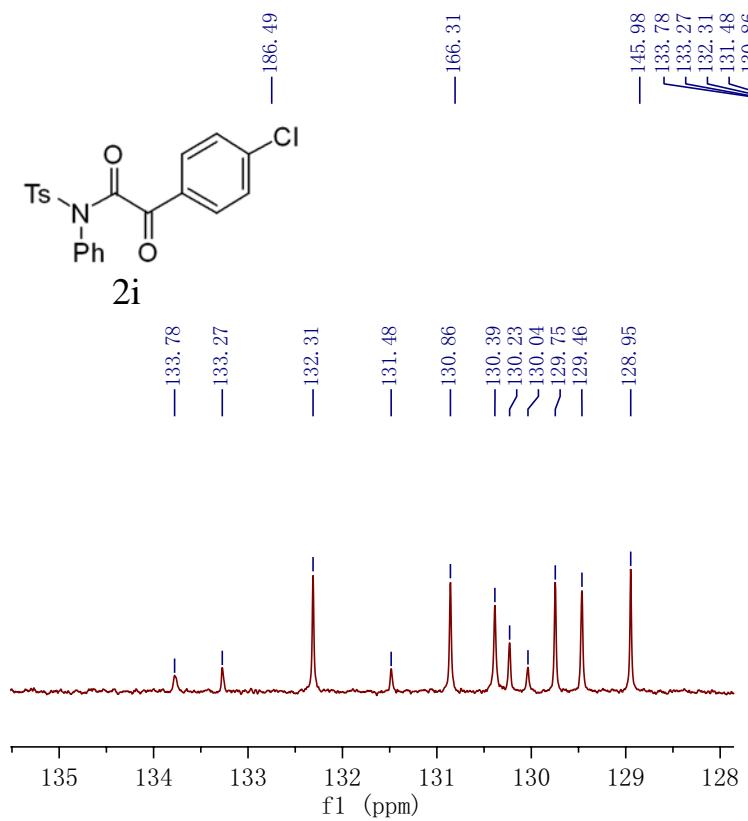
3.00

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)



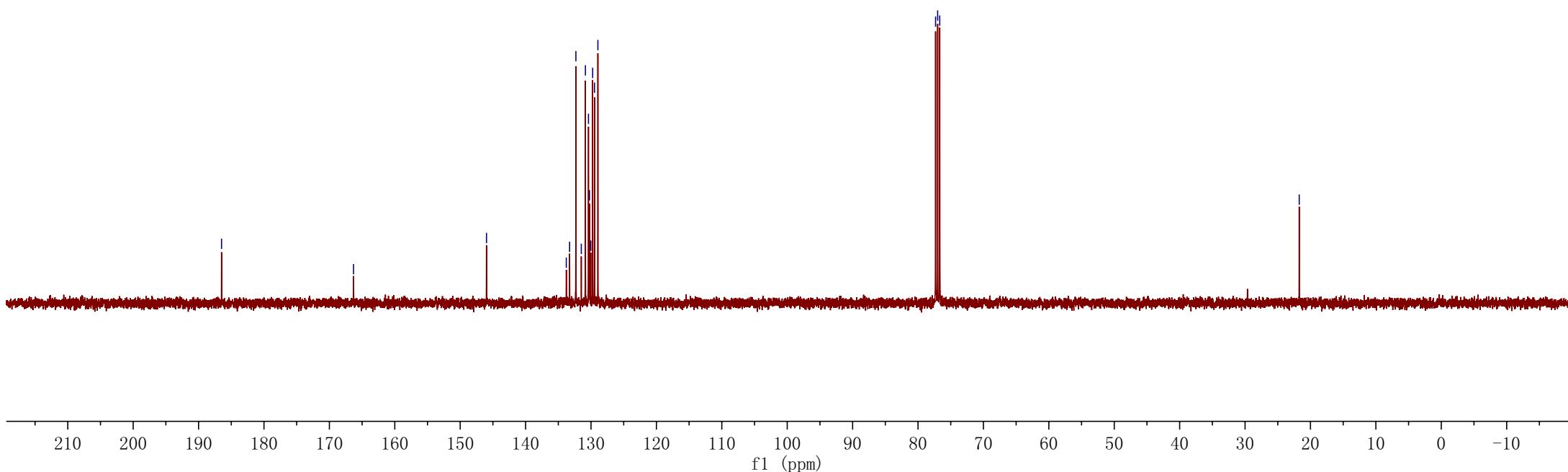
—133.78
 —133.27
 —132.31
 —131.48
 —130.86
 —130.39
 —130.23
 —130.04
 —129.75
 —129.46
 —186.49



—145.98
 —143.78
 —143.27
 —143.31
 —143.48
 —143.86
 —143.39
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 —142.95
 —145.32
 —145.00
 —146.68

—77.32
 —77.00
 —76.68

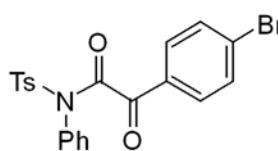
—21.72



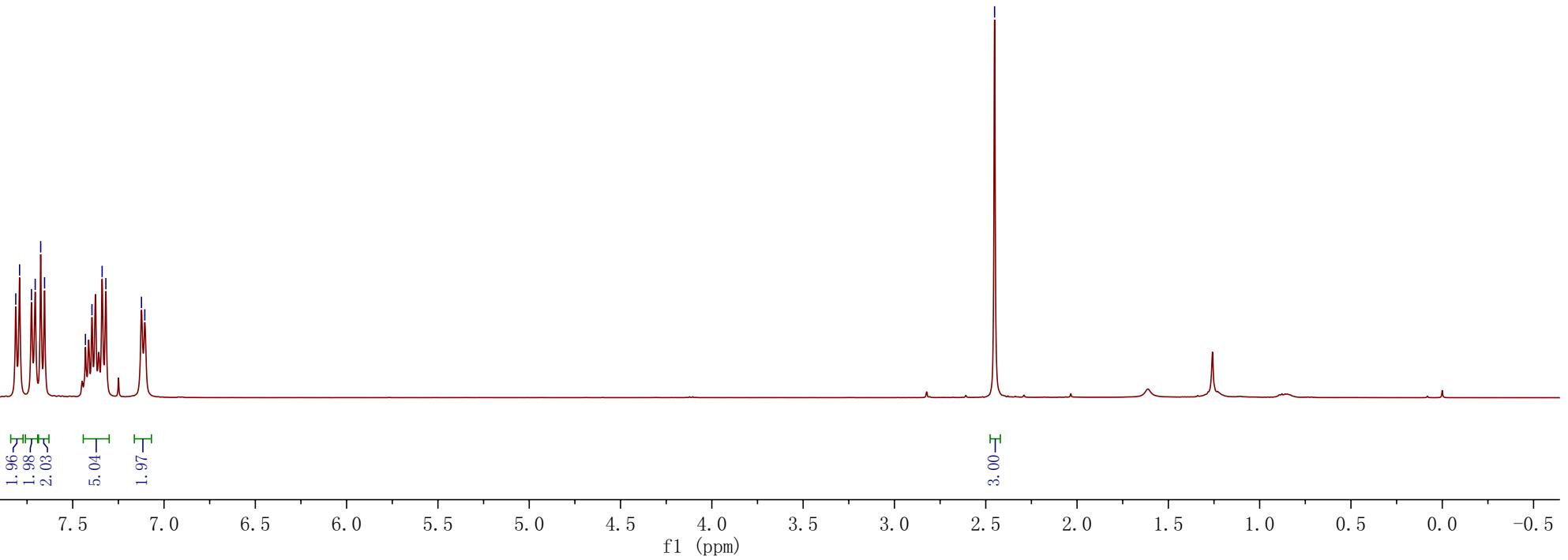
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7.705
7.675
7.654

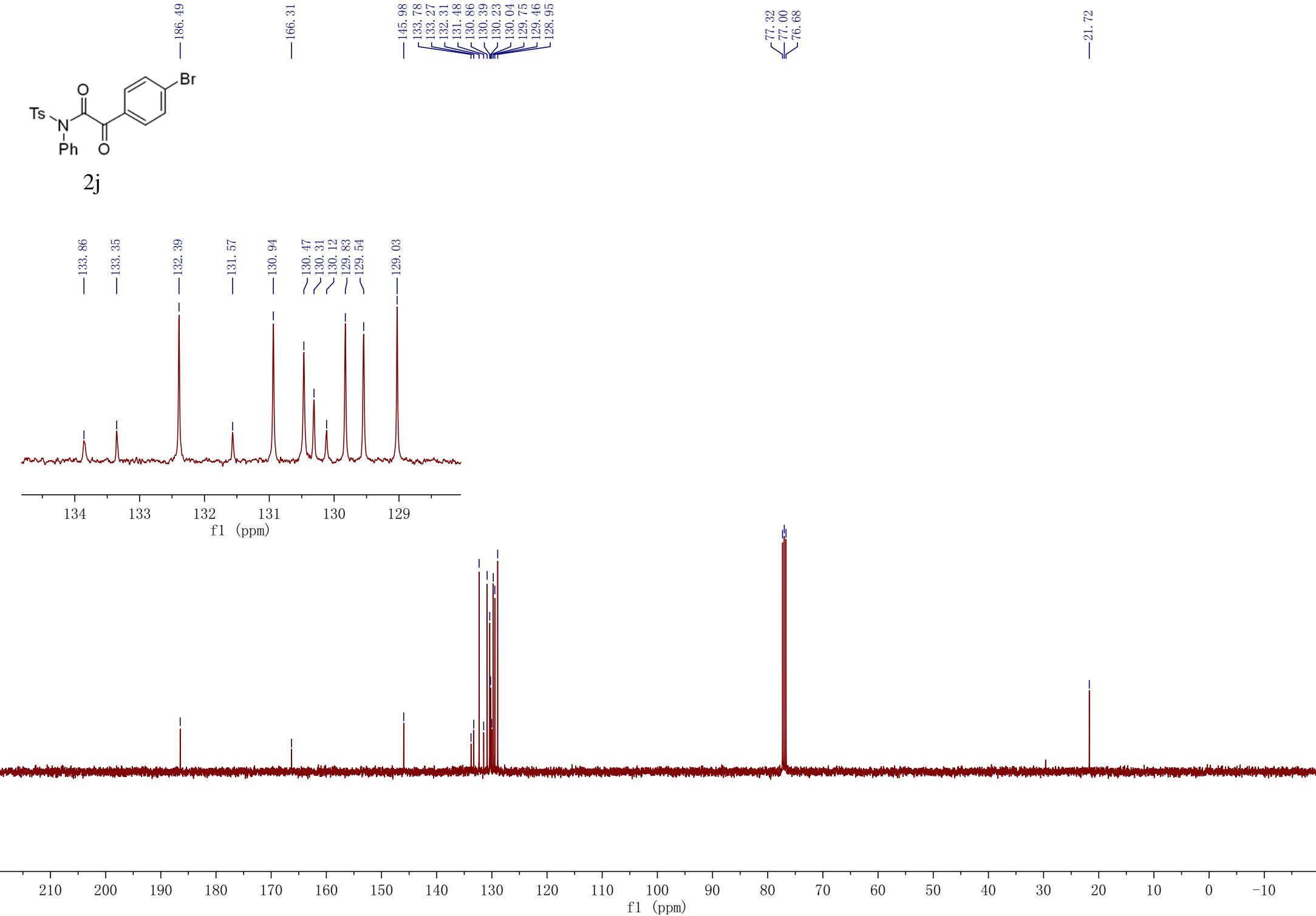
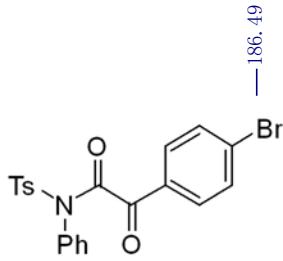
7.395
7.339
7.319
7.123
7.105

— 2.452



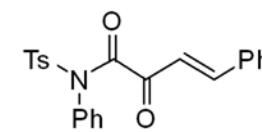
2j



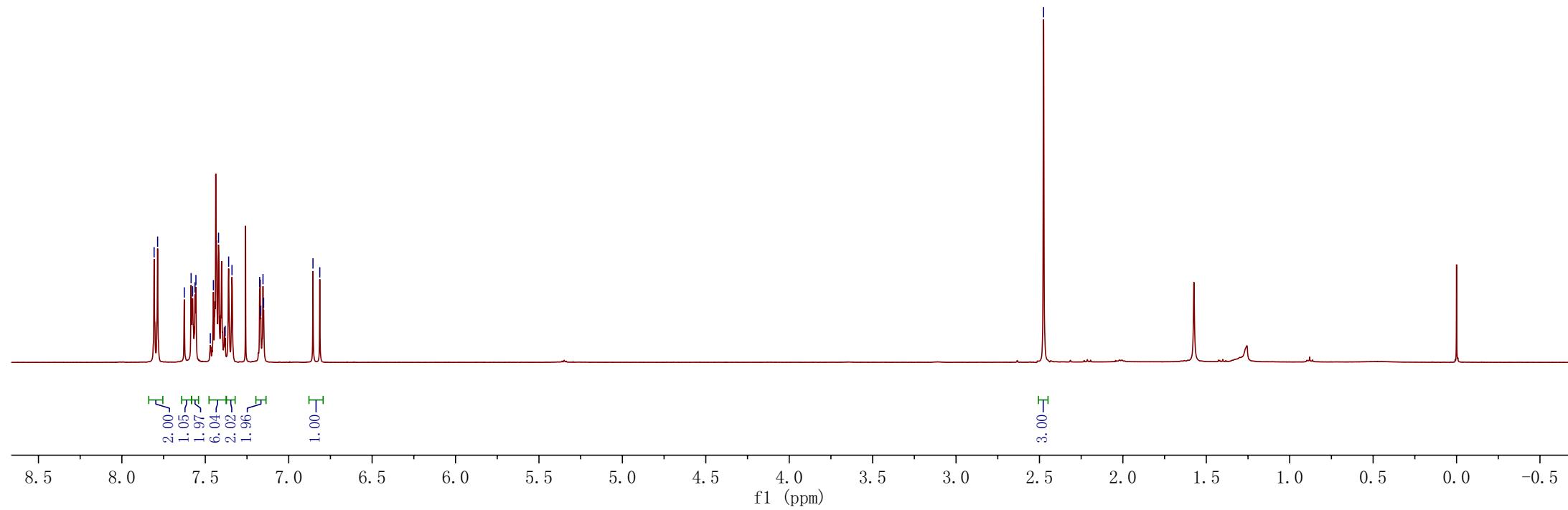


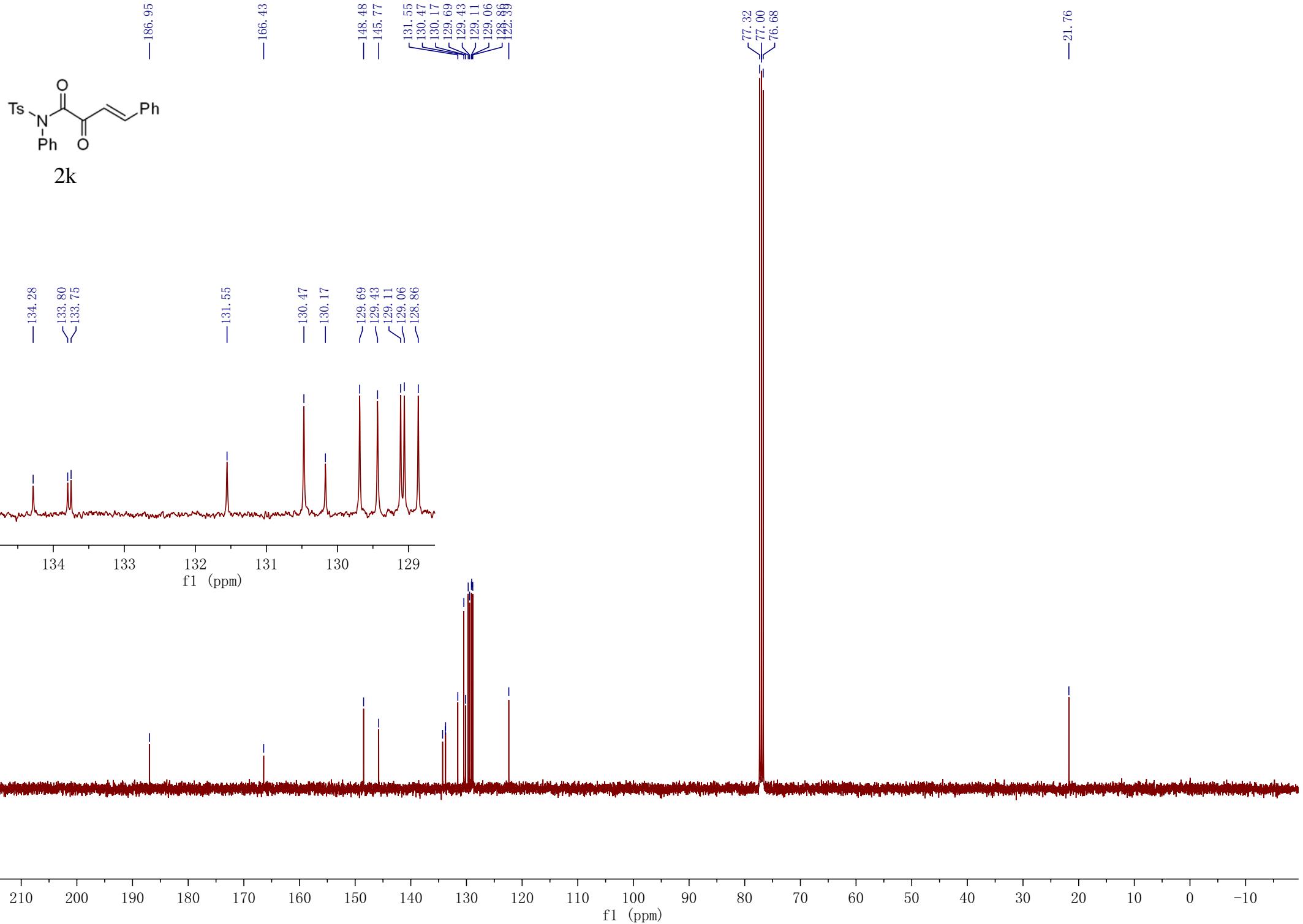
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7.786
7.626
7.585
7.576
7.561
7.556
7.470
7.451
7.421
7.381
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7.174
7.167
7.154
7.150
6.855
~6.814

—2.475



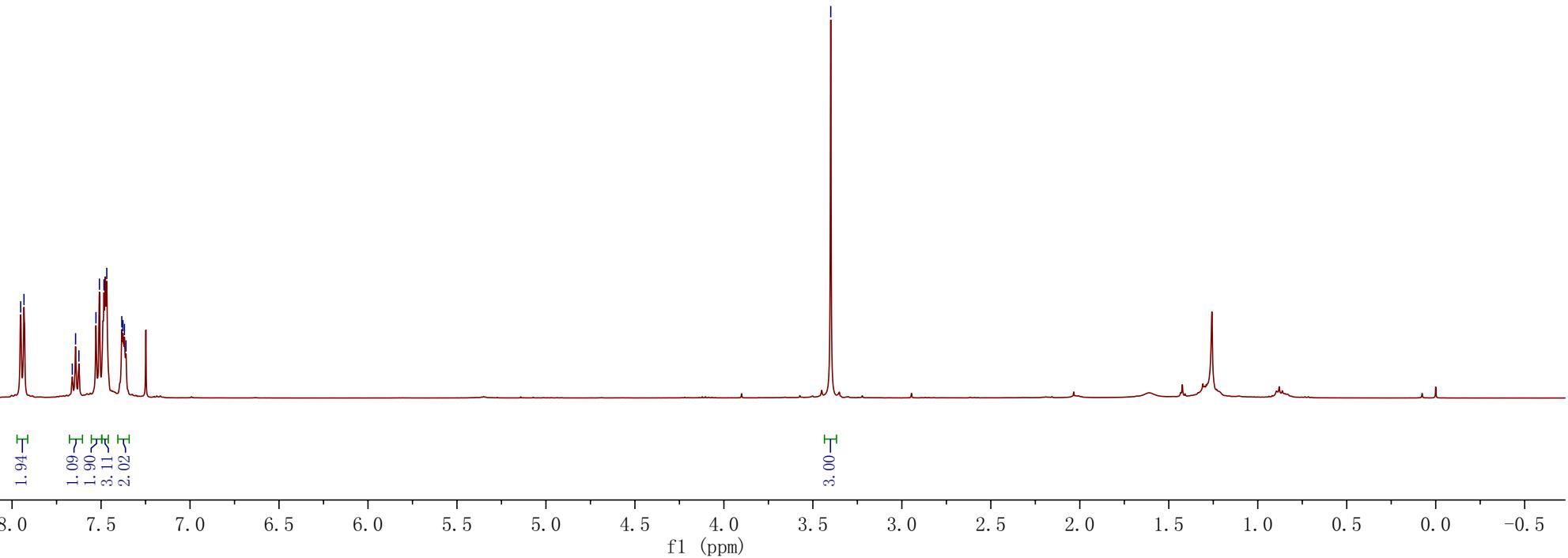
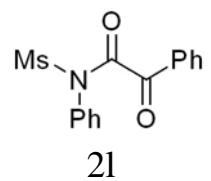
2k

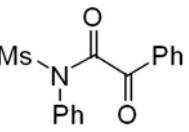




7.951
7.933
7.662
7.643
7.625
7.625
7.529
7.509
7.484
7.468
7.383
7.377
7.369
7.360

—3.400





21

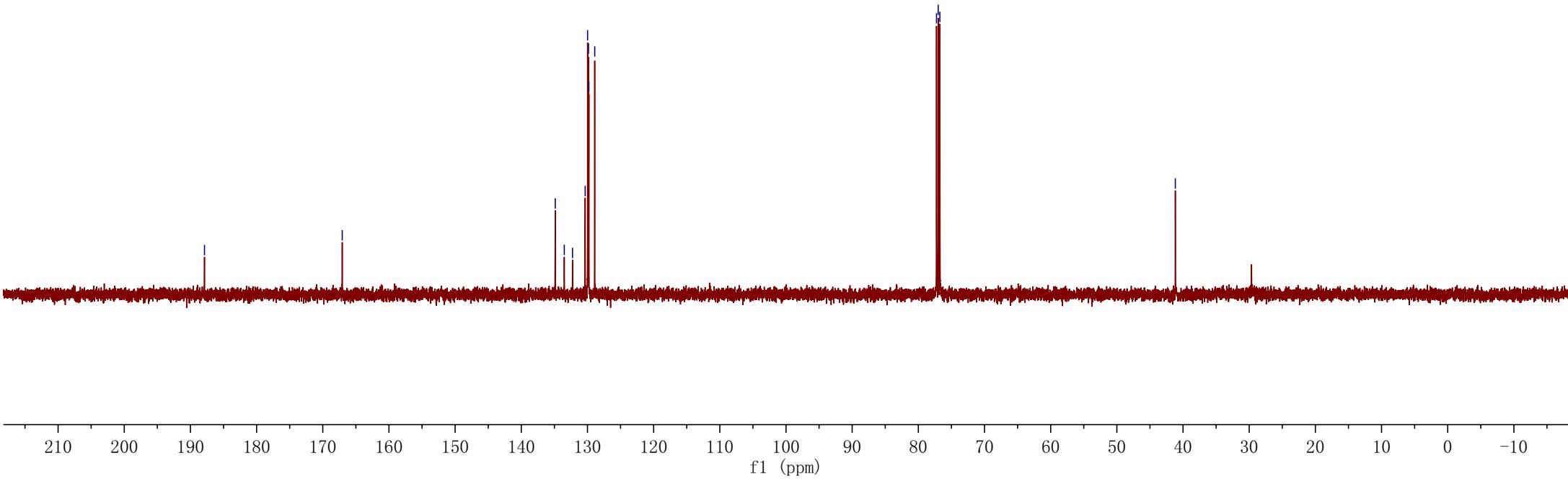
—187.87

—167.06

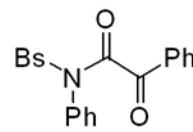
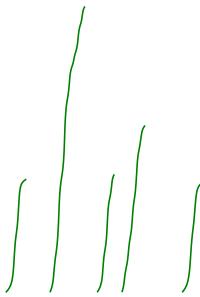
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133.50
132.26
130.35
129.98
129.84
129.78
128.90

77.25
77.00
76.75

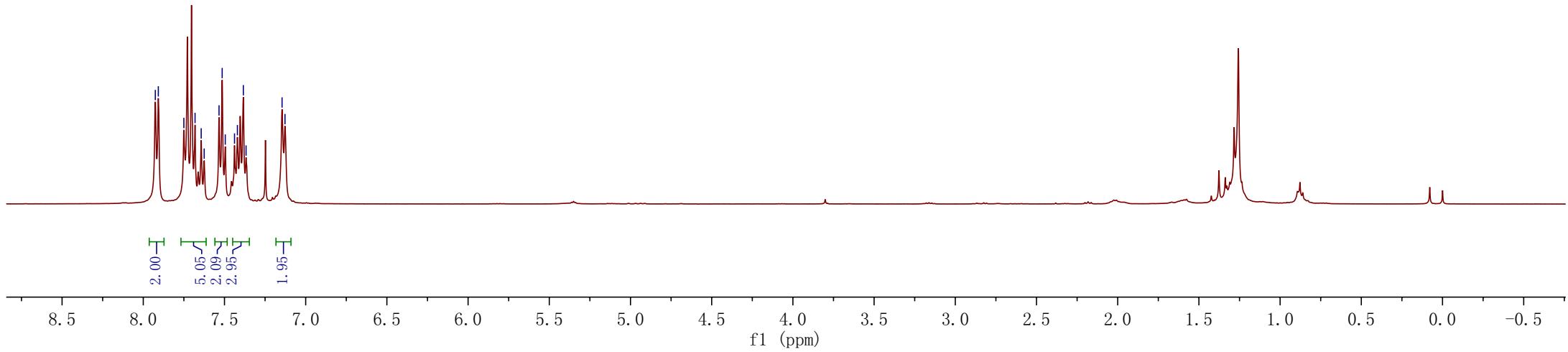
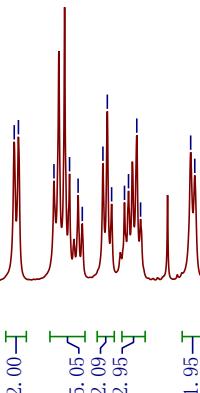
—41.16



7.926
7.907
7.750
7.681
7.643
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7.514
7.495
7.438
7.420
7.384
7.367
7.145
7.127



2m

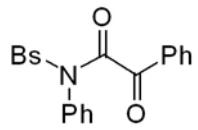


— 187.38

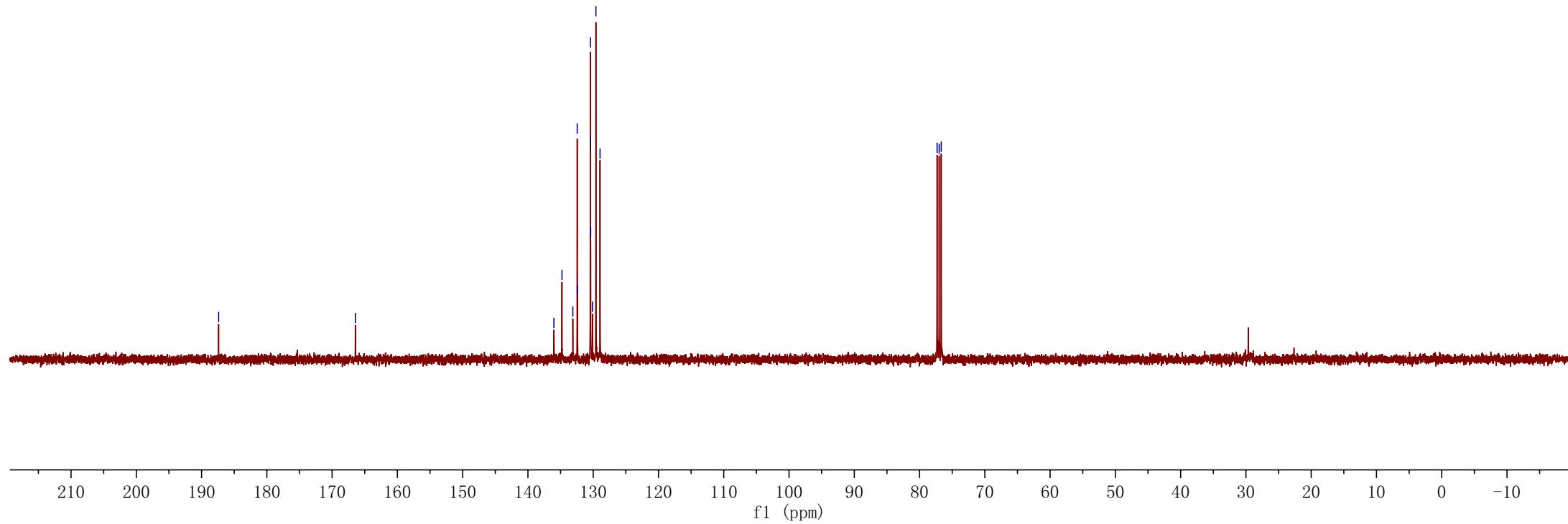
— 166.44

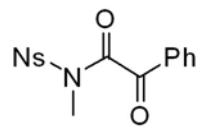
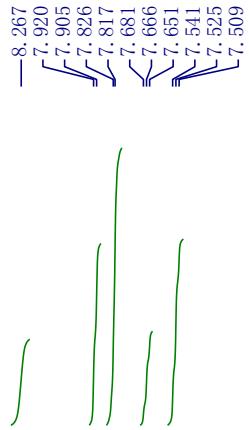
136.02
134.78
133.12
132.45
132.42
130.44
130.42
130.39
130.10
129.59
128.95

77.32
77.00
76.68

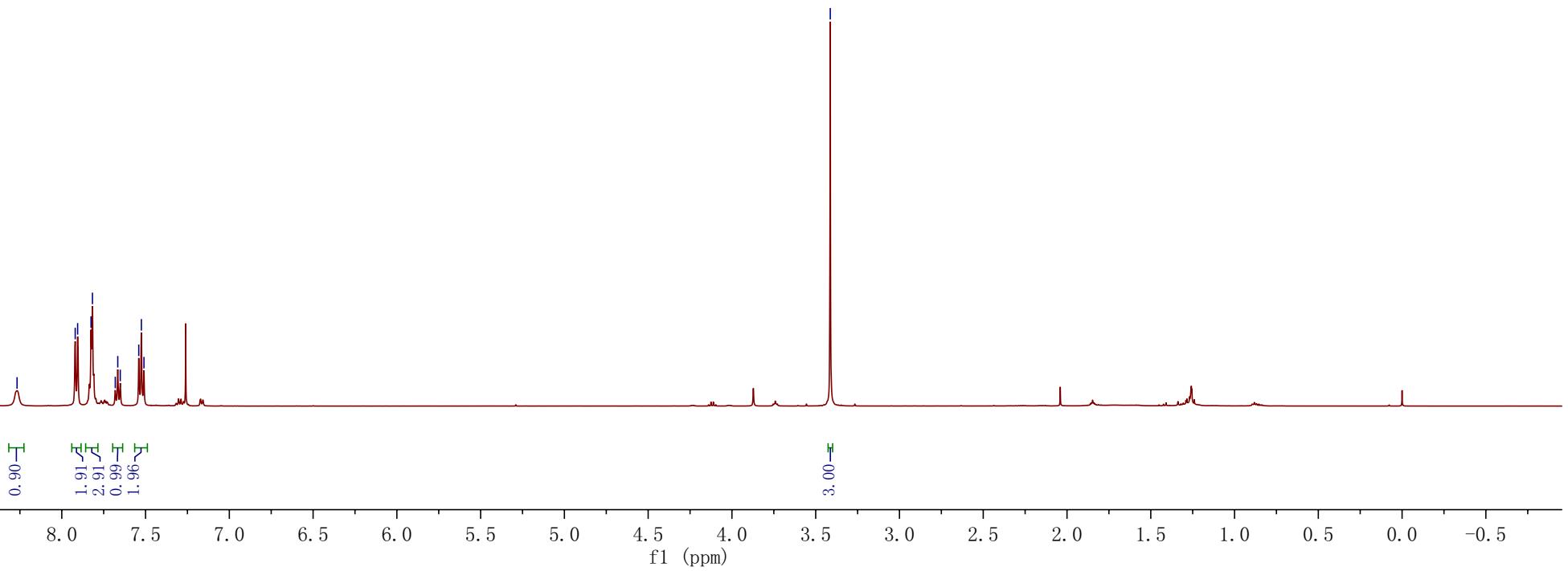


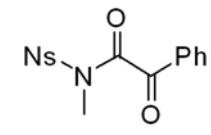
2m





2n





2n

— 187.56
— 166.83
— 148.42

— 77.32
— 77.00
— 76.68

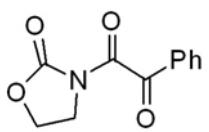
— 32.37

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

7.894
7.876
7.872
7.672
7.651
7.635
7.629
7.537
7.517
7.498

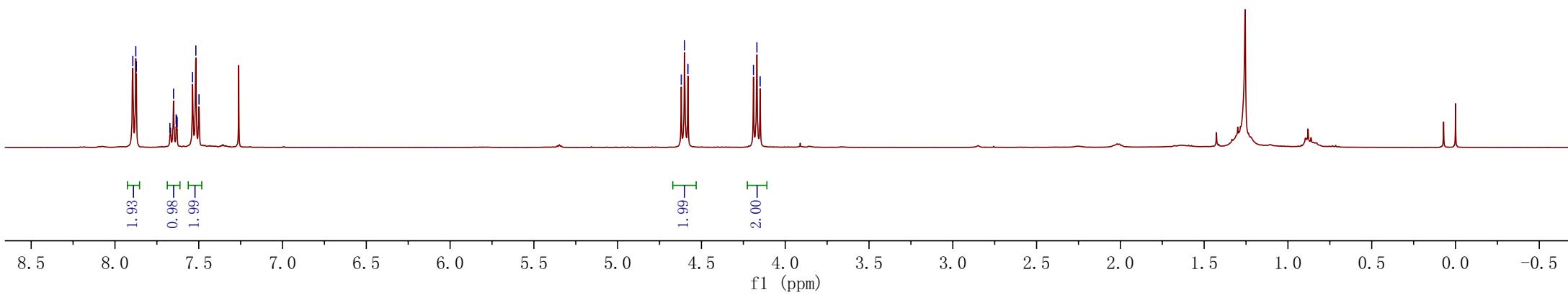
4.621
4.601
4.581
4.190
4.169
4.150

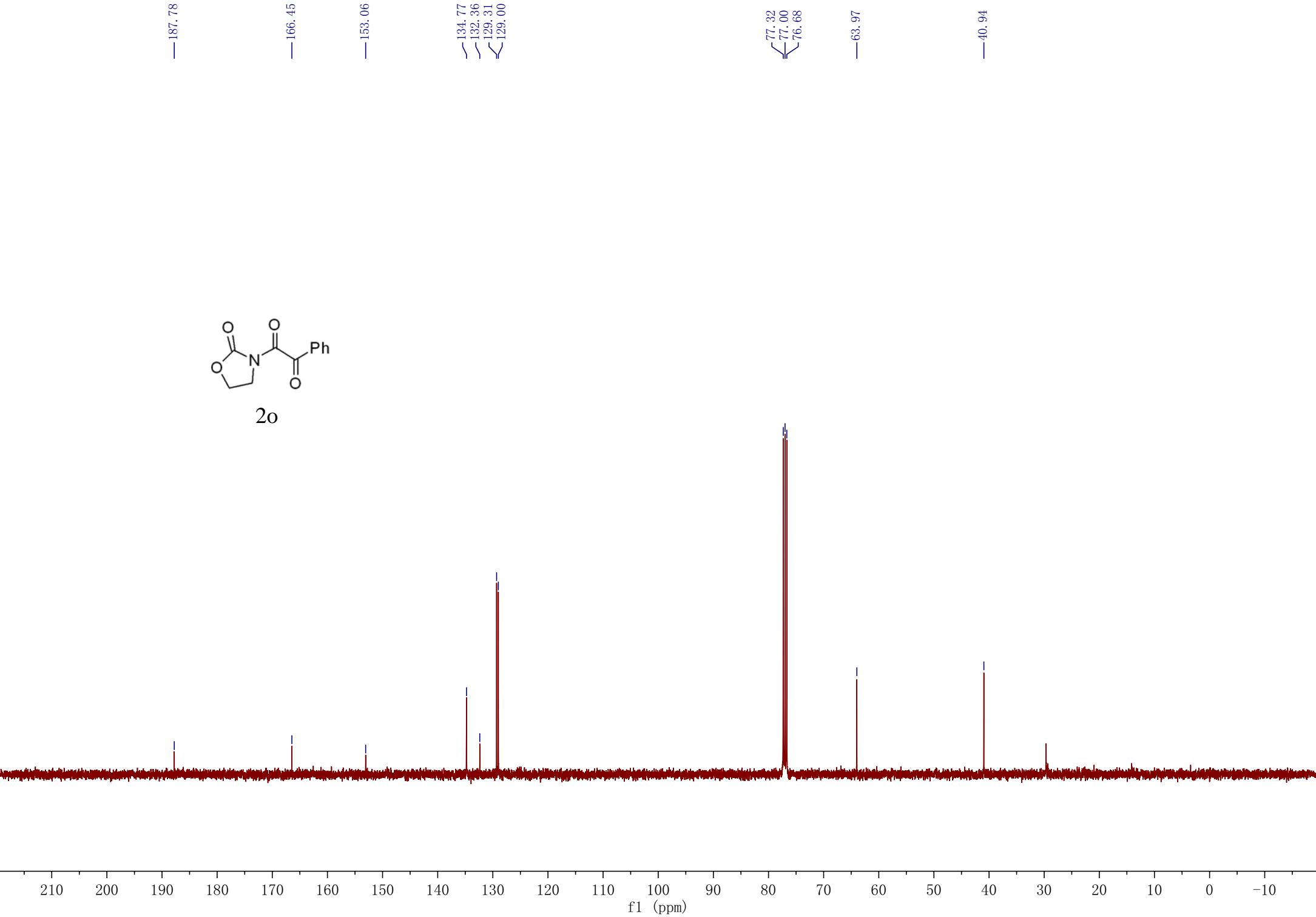


2o

1.93
0.98
1.99

1.99
2.00



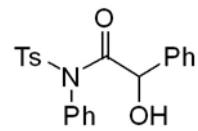


<7.922
<7.906
7.384
7.368
7.284
7.269
7.259
7.254
7.191
7.176
6.826
6.742
<6.727

—4.785

—3.775

—2.493



4a

1.93
1.08
2.03
3.31
1.99
1.80
1.93

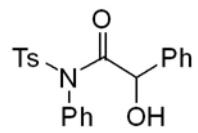
0.95

0.94

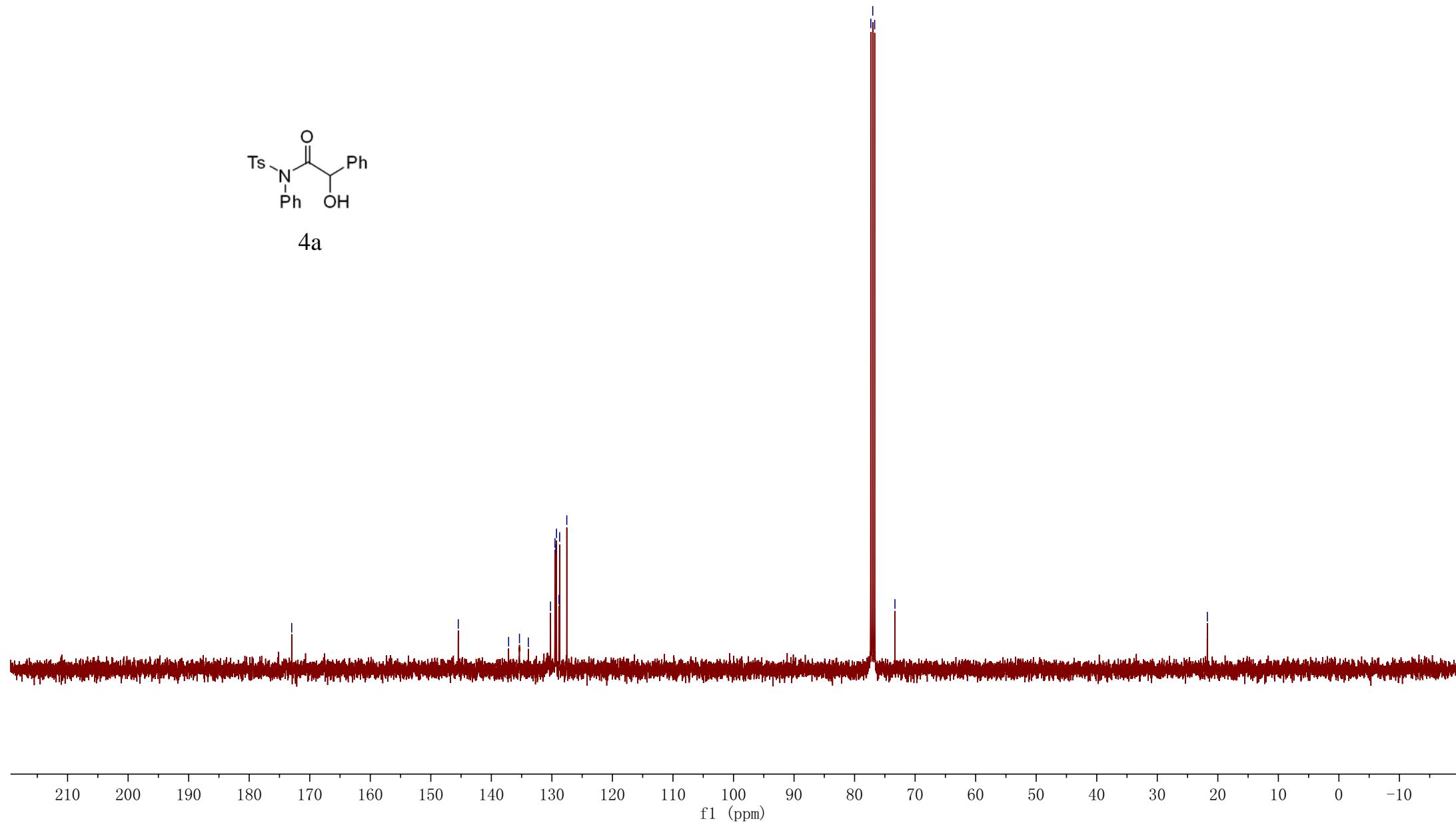
3.00

8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

f1 (ppm)



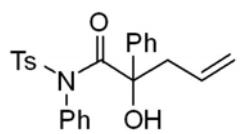
4a



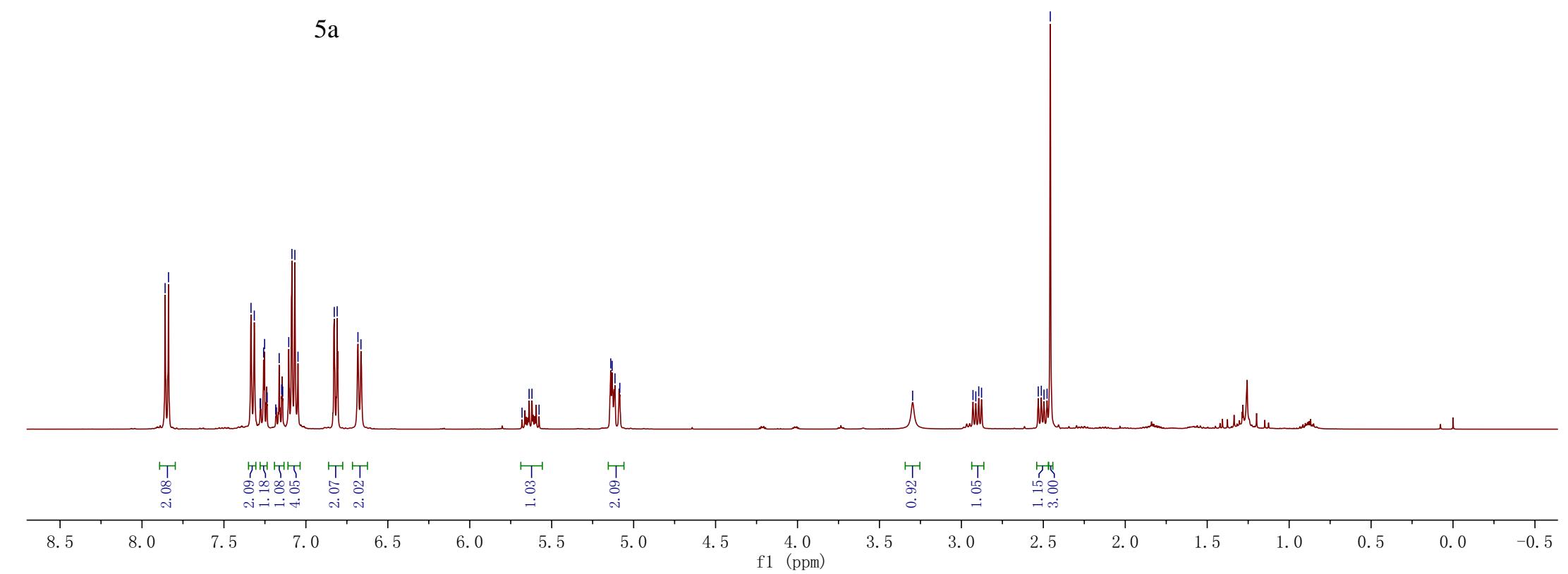
<7.859
<7.838
7.335
7.315
7.258
7.252
7.105
7.085
7.067
6.849
6.809
6.682
6.664

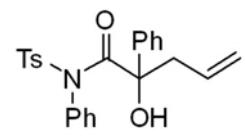
5.681
5.638
5.620
5.577
5.140
5.131
5.114
5.084

-3.298
2.929
2.912
2.894
2.877
2.531
2.496
2.477
2.458



5a





5a

