Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

A Photochemical Halogen-Atom Transfer Pathway for the Carboxylation of Alkenes with CO_2

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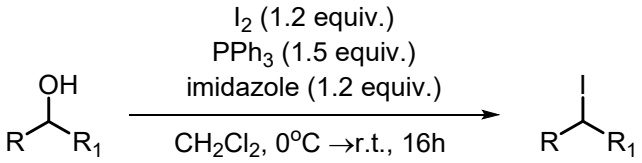
Contents

1. General information

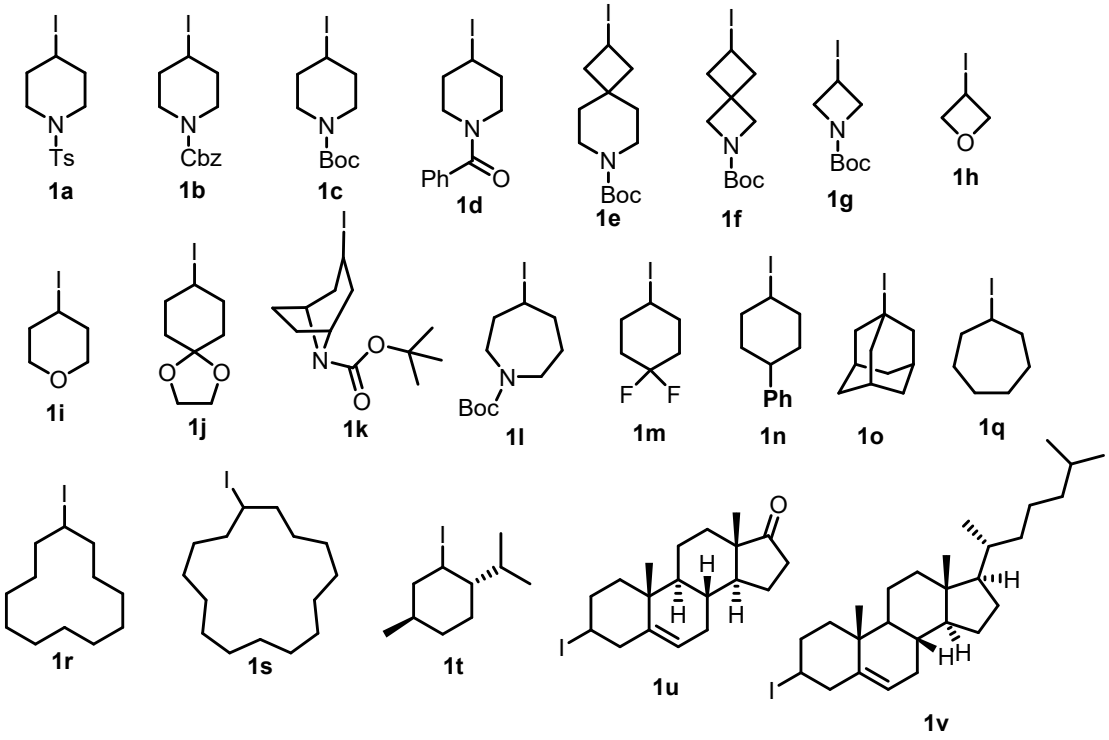
All operations were performed under an argon atmosphere unless otherwise specified. Flash column chromatography was performed over silica gel (200-300 mesh). ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded at ambient temperature using JNM-ECZ500R/S1 (500 MHz) spectrometer or JNM-ECZ600R/S1 (600 MHz) spectrometer. ^{1}H NMR chemical shifts (in ppm) were referenced to $CDCl_3$ (δ = 7.26 ppm), as internal standards. ^{13}C NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.1 ppm). The following abbreviations are used: s = singlet, d = doublet, t = triplet, m = multiplet. GCMS data were obtained on SHIMADZU GCMS-QP2020 NX with EI mode. HRMS data were obtained on Thermo Scientific Orbitrap Elite Mass Spectrometer with an ESI source. Analytical thin-layer chromatography (TLC) was carried out on Merck 60 F254 pre-coated silica gel plate (0.2 mm thickness). Visualization was accomplished by UV light (254 nm), phosphomolybdic acid or $KMnO_4$ staining solutions followed by heating, also by Gas Chromatograph Mass spectrometer analysis (GC-MS). Unless otherwise noted, materials obtained from commercial suppliers were used without further purification.

2. Starting material synthesis



A round-bottom flask equipped with a stirring bar was charged with the alcohol (1.0 equiv.), Ph_3P (1.2 equiv.) and imidazole (1.2 equiv.). The flask was evacuated and refilled with N_2. CH_2Cl_2 (0.1 M) was added, and the reaction was cooled to 0 °C with an ice-water bath. I_2 (1.2 equiv.) was added portion-wise and then the cooling bath

was removed. The reaction was stirred 16 hours at room temperature and then diluted with H_2O. The layers were separated, and the aqueous layer was extracted with CH_2Cl_2 (x 3 times). The combined organic layers were washed with $Na_2S_2O_3$ sat., brine, dried (Na_2SO_4), filtered and evaporated. Purification by flash column chromatography (**1a**-**1t**) on silica gel or recrystallization (**1u, 1v**) from EtOH gave the products. All the spectra date are in agreement with the reports.[1-6]

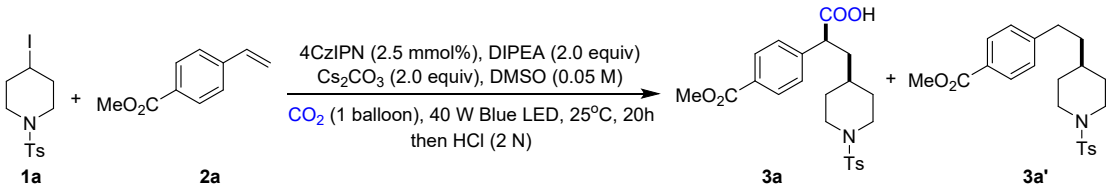


3. General procedure

A dry tube equipped with a stirring bar was charged with the photocatalyst (2.5 mmol%), **1a** (73mg, 0.2 mmol, 1.0 equiv.), **2a** (64.8 mg, 0.4 mmol, 2.0 equiv.), the amine (if solid) (0.4 mmol, 2.0 equiv.) and base (0.4 mmol, 2.0 equiv.). Then evacuated under high vacuum and backfilled with N_2 (x 3). Anhydrous DMSO (4 mL), and DIPEA (0.4 mmol, 2.0 equiv.) was added via syringe under CO_2 atmosphere. Once added, the Schlenk tube was sealed at atmospheric pressure of CO_2 (1 balloon). The reaction was stirred and irradiated with a 456nm Kessil LEDs (1 cm away, with cooling fan to keep the reaction at room temperature and keeping the reaction region located in the

center of LED lamp) for 20 hours. The resulting mixture was diluted with 2 mL EA and quenched by 2 mL 2 N HCl, then stirred for 5 min. The reaction mixture was extracted by EtOAc three times and the combined organic phases were concentrated in vacuo. The residue was purified by silica gel flash column chromatography (PE/EA/AcOH 10/1/0.1%~1/1/0.1%) to give the desired products.

Table S1 Optimization of reaction Conditions.



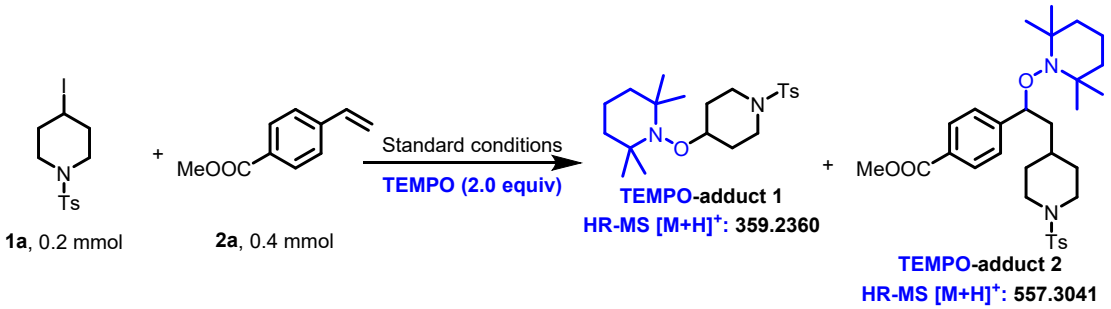
1a **2a** **3a** **3a'**

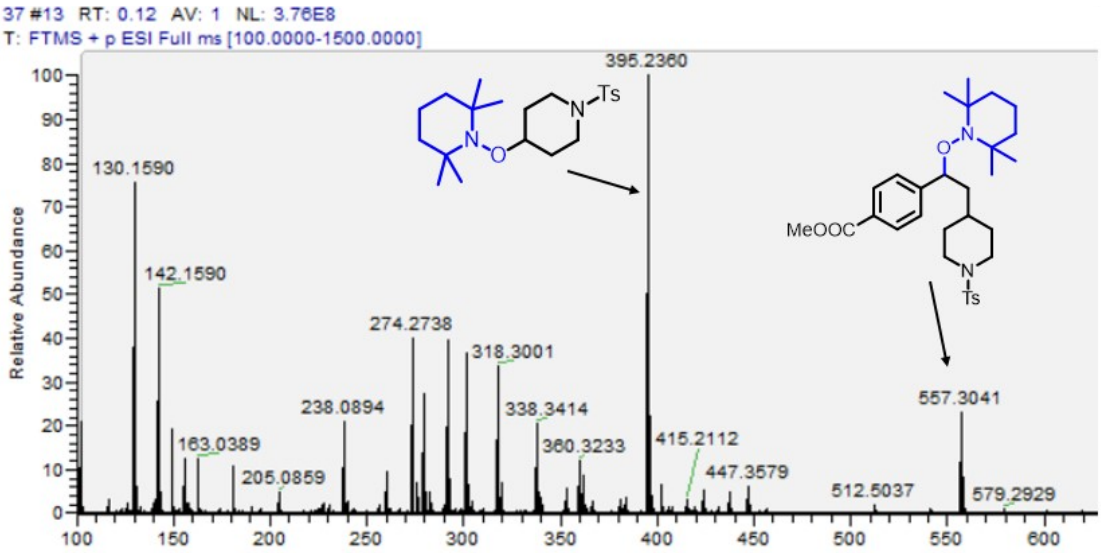
Entry[a]	Variation from standard conditions	Yield of **3a** [%][b]	Yield of **3a'** [%][c]
1	none	90 (82)	4
2	DMA instead of DMSO	86	5
3	MeCN instead of DMSO	<5	45
4	THF instead of DMSO	<5	20
5	DMF instead of DMSO	79	12
6	CsF instead of Cs_2CO_3	82	4
7	K_2CO_3 instead of Cs_2CO_3	78	6

[a] Standard conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), 4CzIPN (2.5 mol%), Cs_2CO_3 (0.4 mmol), DIPEA (0.4 mmol), DMSO (4 mL), CO_2 (1 balloon), 40 W 456 nm blue LED, 25 °C, quenched with 2N HCl (aq.). "w/o" is short for "without". "N.D." is short for "not detected". [b] Yield was determined by ^{1}H-NMR with dibromomethane as an internal standard. The isolated yield is given in parentheses. [c] Yield was determined by GC-MS using 1,3,5-trimethoxybenzene as the internal standard.

4. Mechanism studies

4.1 Radical trapping experiment





4.2 Stern-Volmer emission quenching experiment

Samples for the quenching experiments were prepared in a 4 mL quart cuvette with a cap. 4CzIPN was irradiated at 440 nm and the emission intensity at about 540nm was observed. In a typical, the emission spectrum of a 10^{-5} M solution of 4CzIPN in DMSO and DMF were collected.

DIPEA: A stock solution of DIPEA (10^{-3} M) in DMF was prepared. Then different amounts of this stock solution were added to the 2 mL of 4CzIPN in DMF (10^{-5}).

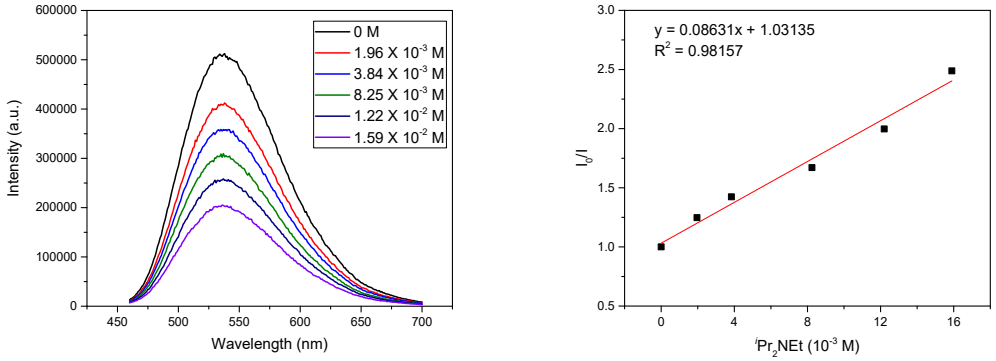


Figure S1. Stern-Volmer fluorescence quenching experiments using 4CzIPN with DIPEA

Alkyl iodide **1a**: A stock solution of **1a** (18.25 mg, 0.05 mmol) in 10 mL DMSO was prepared. Different amounts of this stock solution were added to 2 mL of 4CzIPN in DMSO (10^{-5} M).

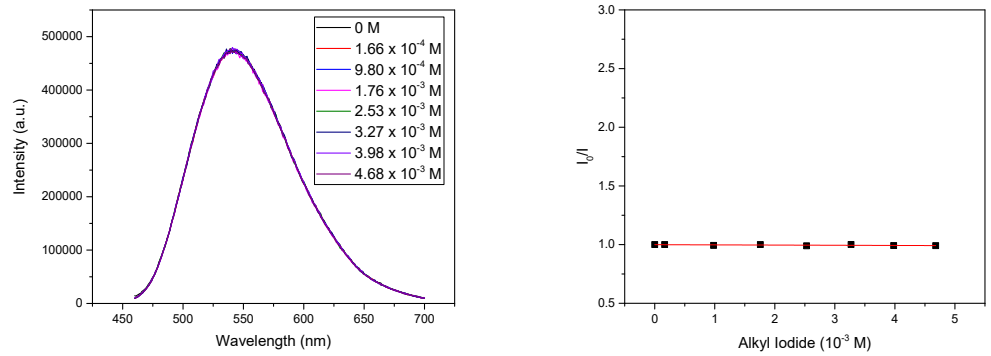


Figure S2. Stern-Volmer fluorescence quenching experiments using 4CzIPN with Alkyl iodide **1a**.

A stock solution of **2a** (8.1 mg, 0.05 mmol) in 10 mL DMSO was prepared. Different amounts of this stock solution were added to 2 mL of 4CzIPN in DMSO (10^{-5} M).

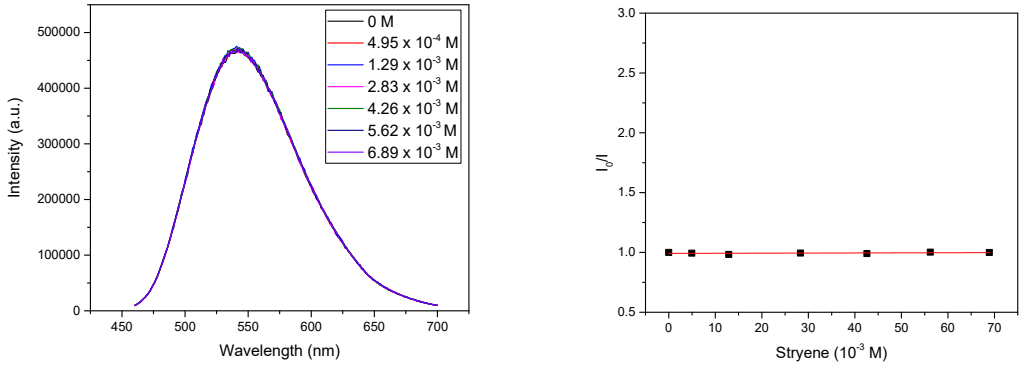
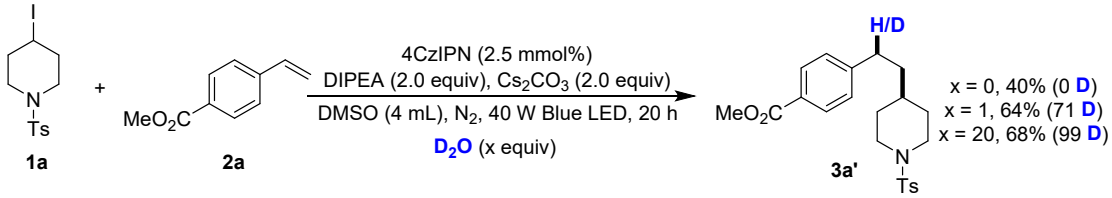
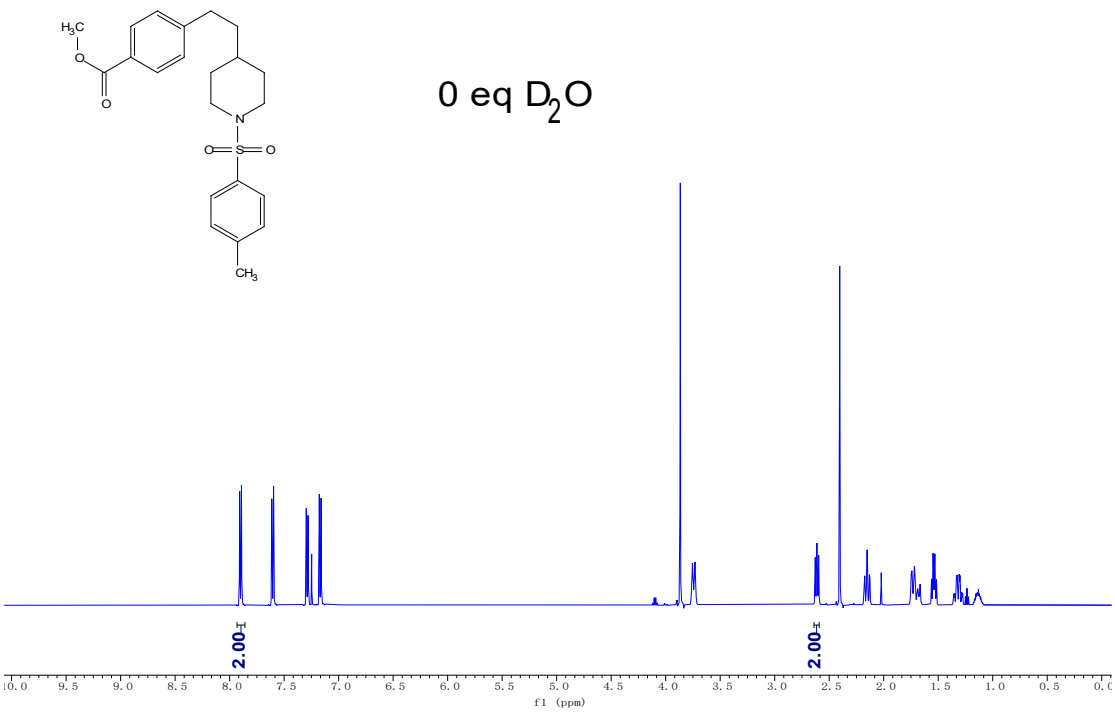


Figure S3. Stern-Volmer fluorescence quenching experiments using 4CzIPN with Stryene.

4.3 Intercepting the carbanion intermediate with D_2O



A dry tube equipped with a stirring bar was charged with the photocatalyst (2.5 mmol%), **1a** (73 mg, 0.2 mmol, 1.0 equiv.), **2a** (64.8 mg, 0.4 mmol, 2.0 equiv.), and Cs_2CO_3 (0.4 mmol, 2.0 equiv.). Anhydrous DMSO (4 mL), D_2O (x equiv.) and DIPEA (0.4 mmol, 2.0 equiv.) was added. Then evacuated under high vacuum and backfilled with N_2 (x 3 times). The reaction was stirred and irradiated with a 456nm Kessil LEDs (1 cm away, with cooling fan to keep the reaction at room temperature and keeping the reaction region located in the center of LED lamp) for 20 hours. The reaction mixture was extracted by EtOAc six times and the combined organic phases were concentrated in vacuo. The residue was purified by recrystallization from EA.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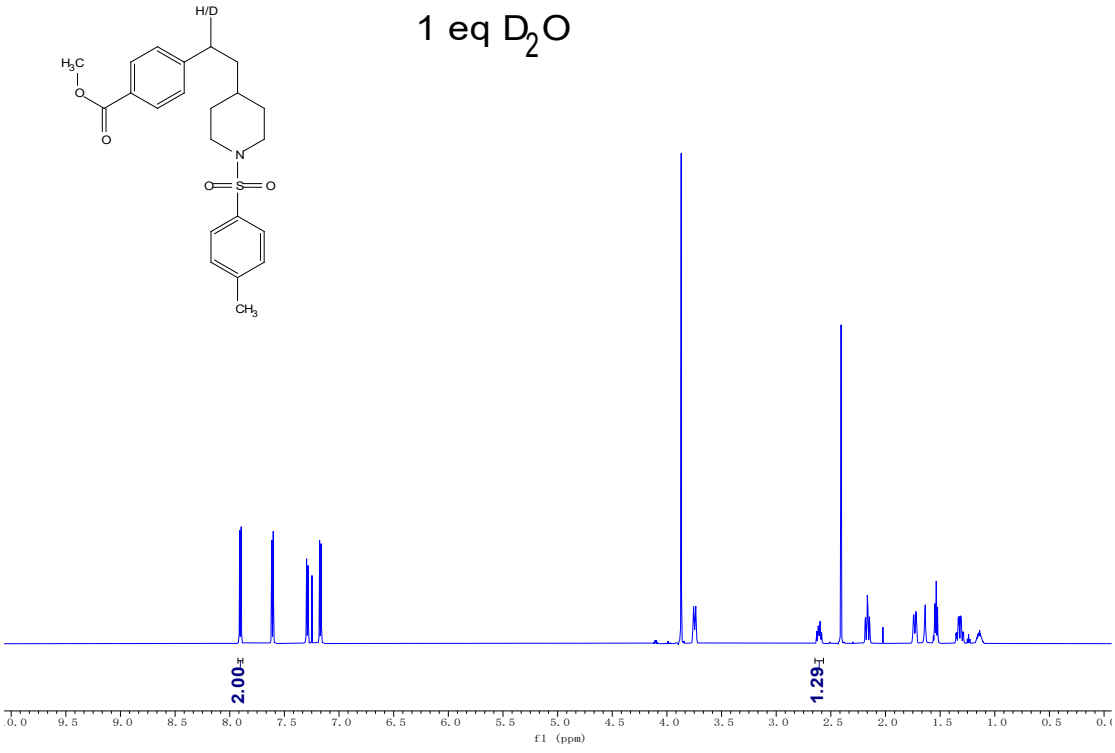
2.00
f1 (ppm)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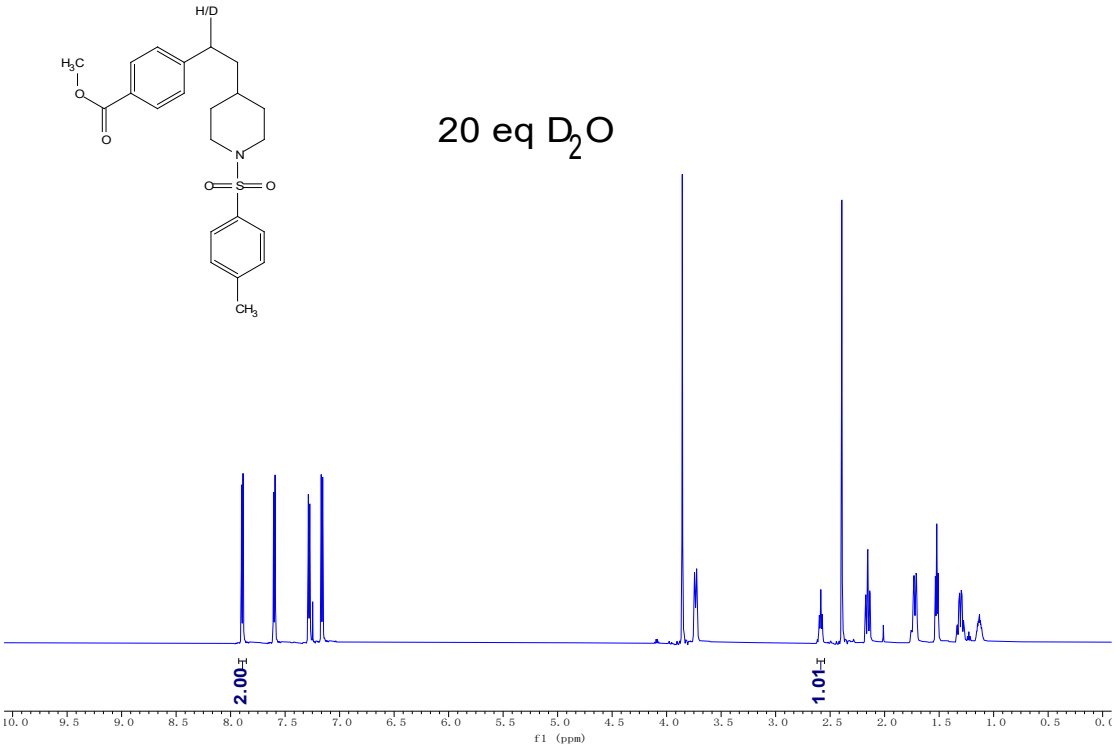
H/D
2.00
1.29
f1 (ppm)



4.4 Defluorinative alkylation of trifluoromethylakene

8 **1a**

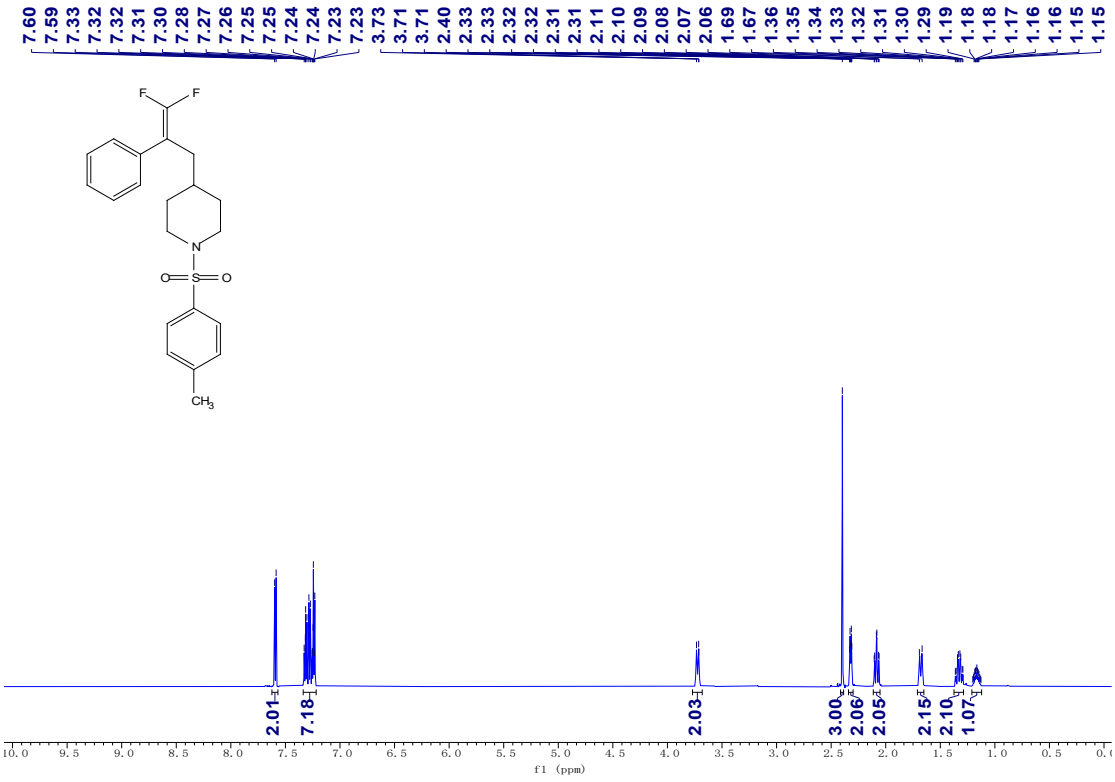
A dry tube equipped with a stirring bar was charged with the photocatalyst (2.5 mmol%), 8 (59 ul, 0.4 mmol, 2.0 equiv.), 1a (73 mg, 0.2 mmol, 1.0 equiv.), and Cs2CO3 (0.4 mmol, 2.0 equiv.). Anhydrous DMSO (4 mL), and DIPEA (0.4 mmol, 2.0 equiv.) was added. Then evacuated under high vacuum and backfilled with N2 (x 3). The reaction was stirred and irradiated with a 456 nm Kessil LEDs (1 cm away, with cooling fan to keep the reaction at room temperature and keeping the reaction region located in the center of LED lamp) for 20 hours. The reaction mixture was extracted by EtOAc six times and the combined organic phases were concentrated in vacuo. The residue was purified by silica gel flash column chromatography.

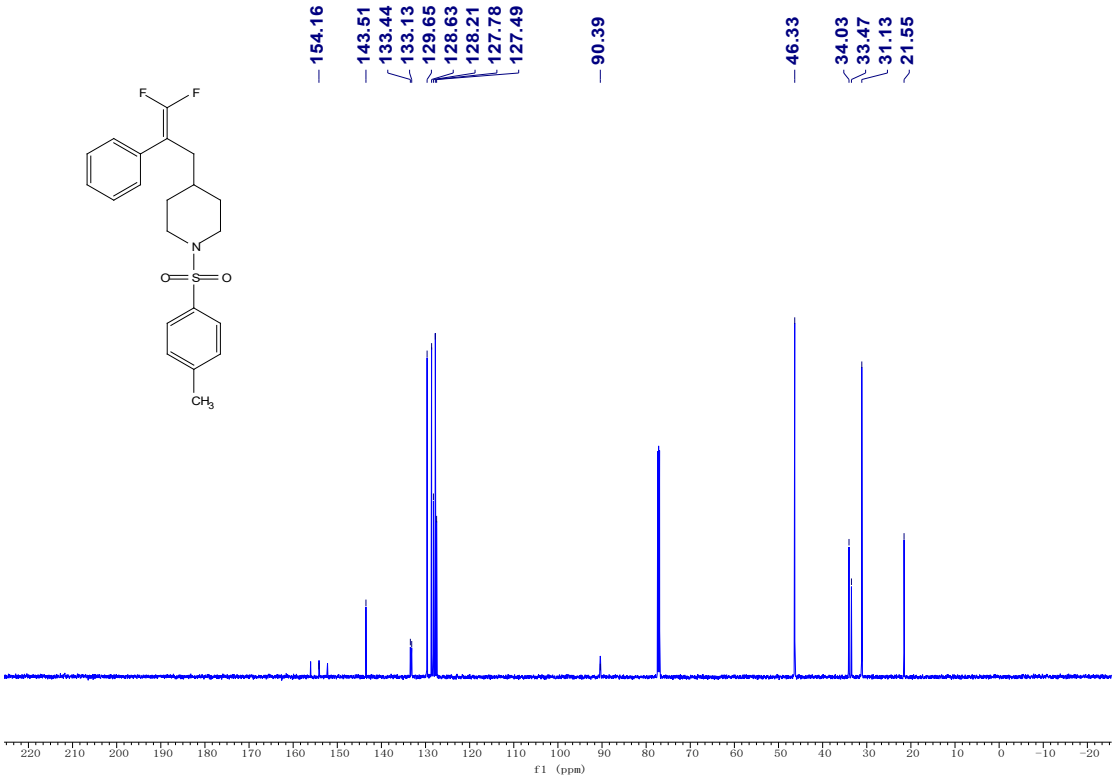
^{1}H NMR (600 MHz, Chloroform-d) δ 7.59 (d, J = 8.3 Hz, 2H), 7.35 – 7.30 (m, 2H), 7.28 (d, J = 8.1 Hz, 2H), 7.26 – 7.22 (m, 3H), 3.72 (d, J = 11.8 Hz, 2H), 2.40 (s, 3H), 2.34 – 2.30 (m, 2H), 2.11 – 2.05 (m, 2H), 1.68 (d, J = 15.0 Hz, 2H), 1.39 – 1.26 (m, 2H), 1.24 – 1.11 (m, 1H).

^{13}C NMR (151 MHz, CHLOROFORM-D) δ 154.16 (dd, J = 291.0, 286.8 Hz), 143.51, 133.44, 133.13, 129.65, 128.63, 128.21, 127.78, 127.49, 90.39, 46.33, 34.03, 33.47, 31.13, 21.55., 90.39 (dd, J = 21.6, 13.6 Hz).

^{19}F NMR (565 MHz, Chloroform-d) δ -90.46 (d, J = 41.3 Hz), -90.78 (d, J = 41.2 Hz).

HRMS (ESI+) $[M+H]^+$ calculated m/z for $[C_{21}H_{24}F_2NO_2S]^+$: 392.1490, found: 392.1483

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133.13
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90.39
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34.03
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31.13
21.55
F
F
N
O
S
O
CH3
f1 (ppm)


-90.46
-90.78
F
F
N
O
S
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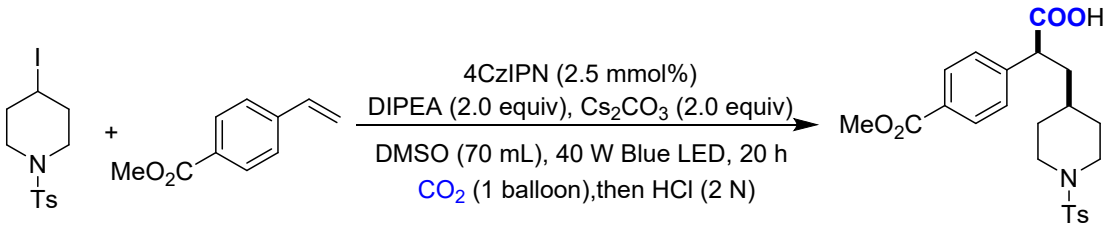
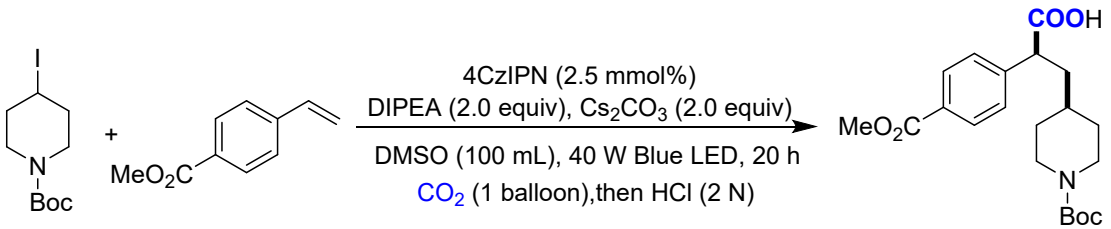
CH3
f1 (ppm)

4.5 Gram-scale synthesis and Product derivatizations



1c, 3.5 mmol **2a**, 7 mmol **3a**, 68%, 1.06g

A dry flask equipped with a stirring bar was charged with the photocatalyst (2.5 mmol%), **1a** (3.5 mmol, 1.0 equiv.), **2a** (7 mmol, 2.0 equiv.) and Cs_2CO_3 (7 mmol, 2.0 equiv.). Then evacuated under high vacuum and backfilled with N_2 (x 3 times). Anhydrous DMSO (70 mL), and DIPEA (7 mmol, 2.0 equiv.) was added via syringe under CO_2 atmosphere. Once added, the flask was sealed at atmospheric pressure of CO_2 (1 balloon). The reaction was stirred and irradiated with 50 W Blue LEDs (1 cm away, with cooling fan to keep the reaction at room temperature and keeping the reaction region located in the center of LED lamp) for 20 hours. The resulting mixture was diluted with 70 mL EA and quenched by 70 mL 2 N HCl, then stirred for 10 min. The reaction mixture was extracted by EtOAc six times and the combined organic phases were concentrated in vacuo. The residue was purified by recrystallization (PE/EtOAc), resulting in the formation of 1.06 g of the final product (**3a**).



1c, 5 mmol **2a**, 10 mmol **3c**, 63%, 1.23g

A dry flask equipped with a stirring bar was charged with the photocatalyst (2.5 mmol%), **1c** (5 mmol, 1.0 equiv.), **2a** (10 mmol, 2.0 equiv.) and Cs_2CO_3 (10 mmol, 2.0 equiv.). Then evacuated under high vacuum and backfilled with N_2 (x 3 times). Anhydrous DMSO (100 mL), and DIPEA (10 mmol, 2.0 equiv.) was added via syringe under CO_2 atmosphere. Once added, the flask was sealed at atmospheric pressure of

CO_2 (1 balloon). The reaction was stirred and irradiated with 40 W Blue LEDs (1 cm away, with cooling fan to keep the reaction at room temperature and keeping the reaction region located in the center of LED lamp) for 20 hours. The resulting mixture was diluted with 100 mL EA and quenched by 70 mL 2 N HCl, then stirred for 10 min. The reaction mixture was extracted by EtOAc six times and the combined organic phases were concentrated in vacuo. The residue was purified by silica gel flash column chromatography.

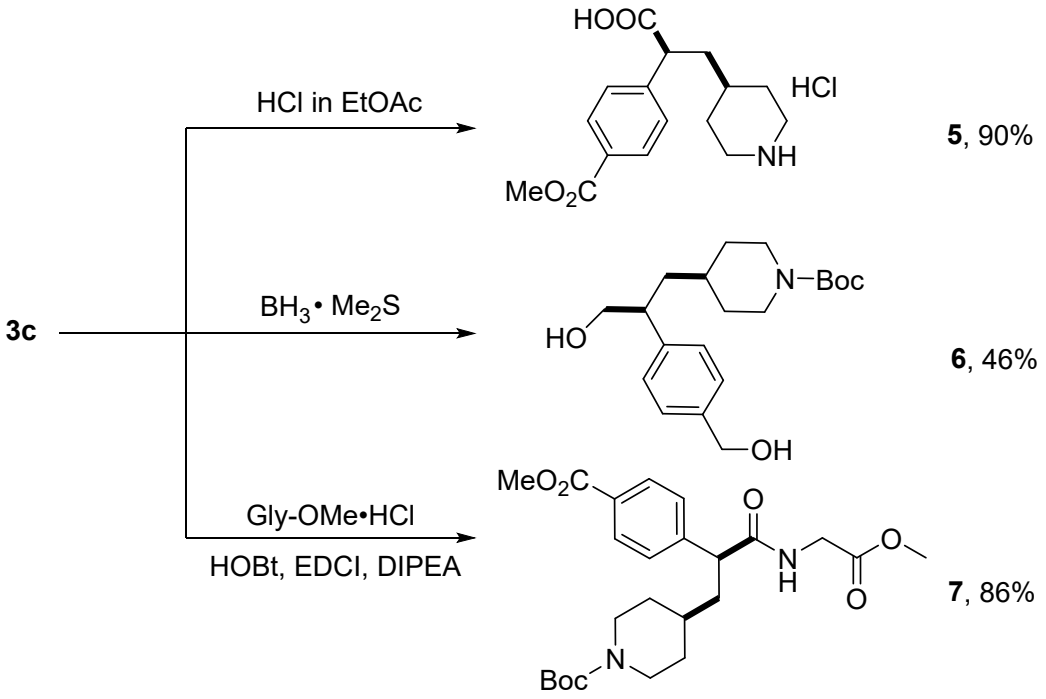
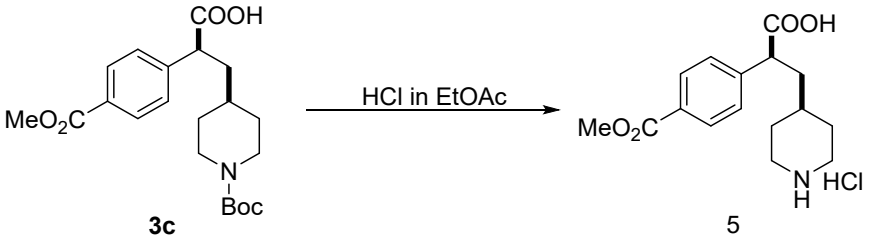


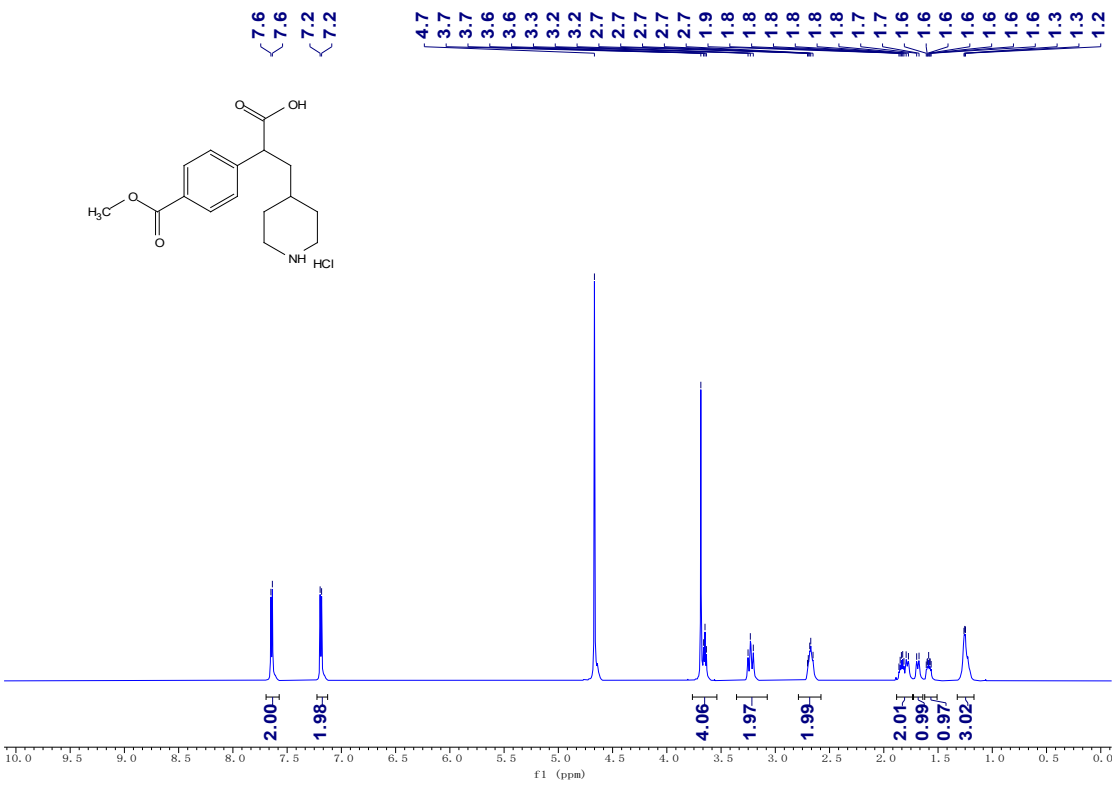
Figure S4. Product derivatizations



A dry tube equipped with a stirring bar was charged with **3c** (1 mmol, 391mg). HCl (2 M in EtOAc, 20 mL) was added via syringe. The reaction was stirred at room temperature for 12h. The mixture was filtered and washed with EtOAc to give the final product as a white solid (295 mg, 0.902 mmol, 90%). ^{1}H NMR (600 MHz, D_2O) δ 7.64

(d, *J* = 8.1 Hz, 1H), 7.19 (d, *J* = 8.1 Hz, 1H), 3.69 (s, 1H), 3.65 (t, *J* = 7.7 Hz, 0H), 3.27 – 3.19 (m, 1H), 2.71 – 2.64 (m, 1H), 1.88 – 1.76 (m, 1H), 1.69 (d, *J* = 13.1 Hz, 1H), 1.62 – 1.55 (m, 1H), 1.34 – 1.16 (m, 1H). ^{13}C NMR (151 MHz, D_2O) δ 177.26, 168.63, 144.22, 129.92, 128.52, 128.21, 52.60, 48.23, 43.85, 43.81, 38.07, 30.98, 28.40, 27.83

HRMS (ESI+) $[M+H]^+$ calculated m/z for $[C_{12}H_{22}NO_4]^+$: 292.1543, found: 292.1534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3.6
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1.3
1.2
O
OH
H3C
O
O
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0.97
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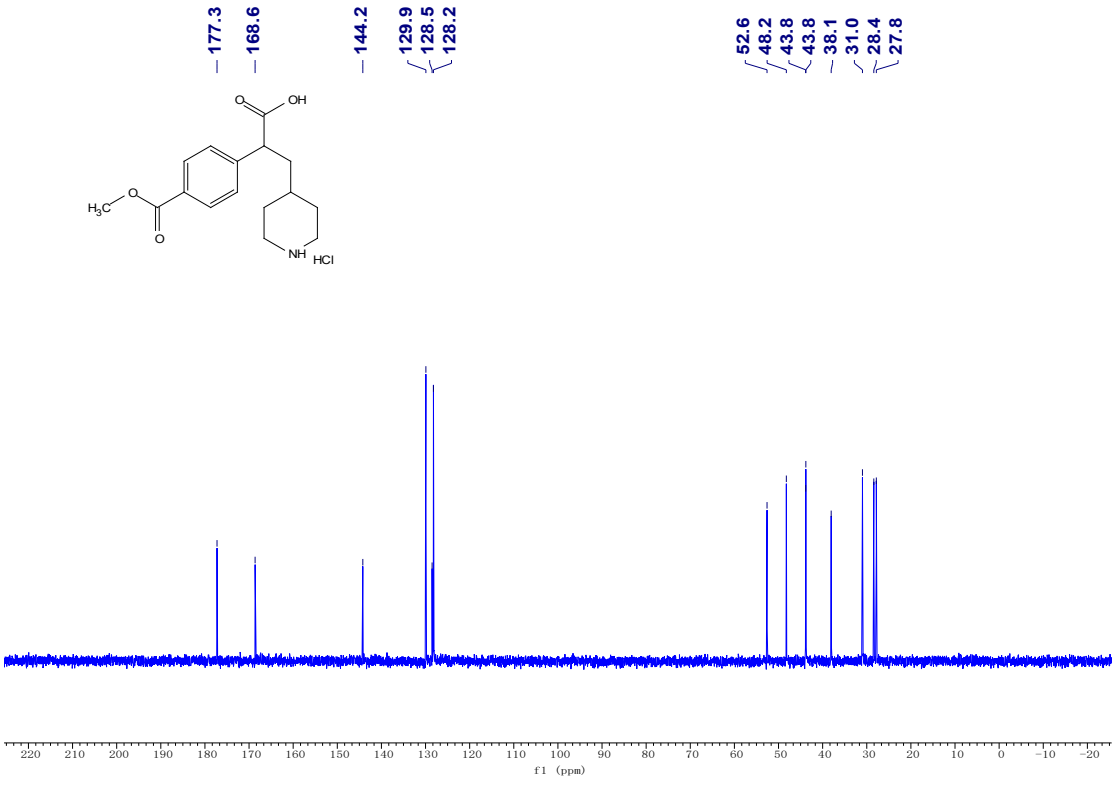
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128.2
52.6
48.2
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H3C
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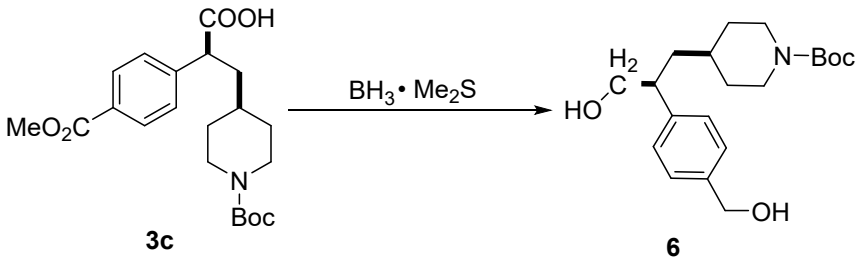
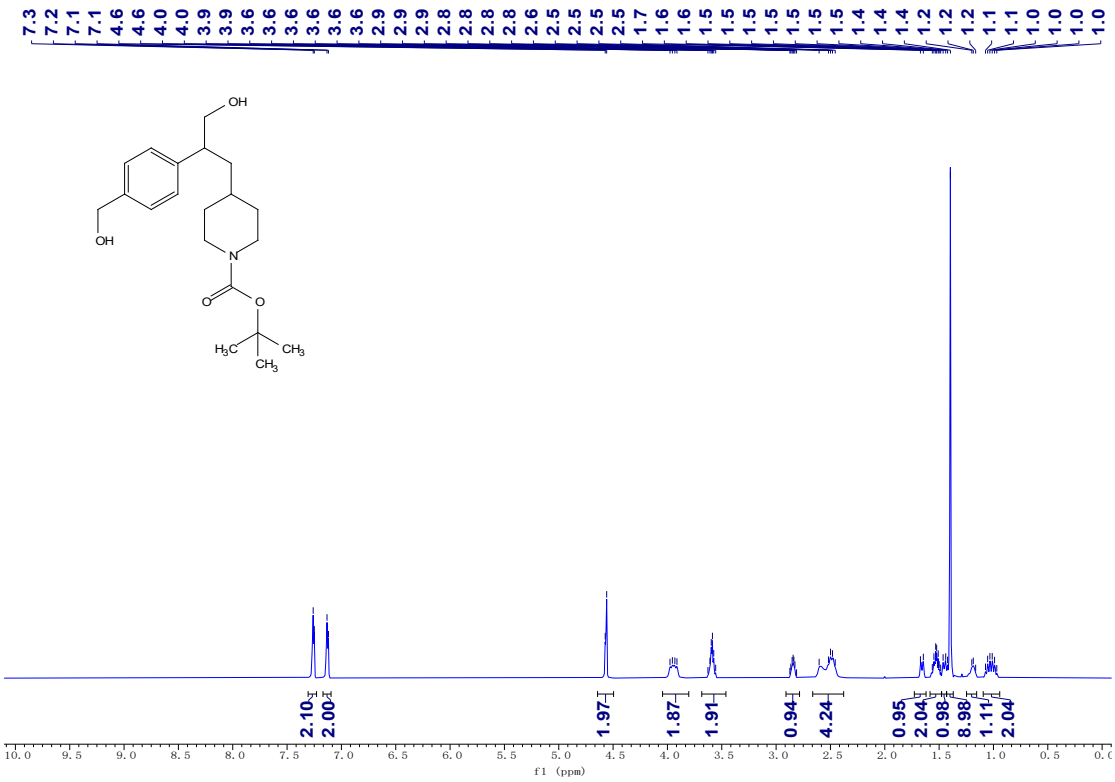
O
NH
HCl
f1 (ppm)



3c **6**

To a 0 ^{o}C solution of **3c** (117mg, 0.3 mmol, 1.0 equiv) in 3.0 mL of THF is slowly added under N_2 the $BH_3{\cdot}Me_2S$ (1.0 mL, 2.0 M in THF). The solution is stirred at room temperature during 22 h more. The solution is then cooled to 0 ºC and 6 mL of water is slowly added. The organic layer is extracted with 6 mL EtOAc for three times, washed with 10 mL of brine, 10 mL of aqueous $NaHCO_3$, 10 mL of H_2O and finally 10 mL of brine. The organic layer is then dried on Na_2SO_4, filtered and evaporated. Final purification by flash chromatography gave the final product as white solid (48.5 mg, 0.138 mmol , 46%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.28 – 7.24 (m, 1H), 7.15 – 7.11 (m, 1H), 4.56 (s, 1H), 3.94 (dd, *J* = 25.9, 13.3 Hz, 2H), 3.59 (q, *J* = 7.2 Hz, 1H), 2.89 – 2.80 (m, 1H), 2.68 – 2.35 (m, 5H), 1.66 (d, *J* = 13.7 Hz, 1H), 1.59 – 1.48 (m, 1H), 1.45 (d, *J* = 14.3 Hz, 1H), 1.40 (s, 4H), 1.25 – 1.13 (m, 1H), 1.11 – 0.93 (m, 1H). ^{13}C NMR (151 MHz, CHLOROFORM-*D*) δ 154.89, 141.47, 139.52, 128.07, 127.38, 79.35, 67.82, 64.66, 45.13, 43.79, 38.66, 33.08, 32.93, 31.42, 28.42.

HRMS (ESI+) $[M+H]^+$ calculated m/z for $[C_{20}H_{32}NO_4]^+$: 350.2326, found: 350.2320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OH
N
O
O
H3C
CH3
CH3
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2.00
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0.94
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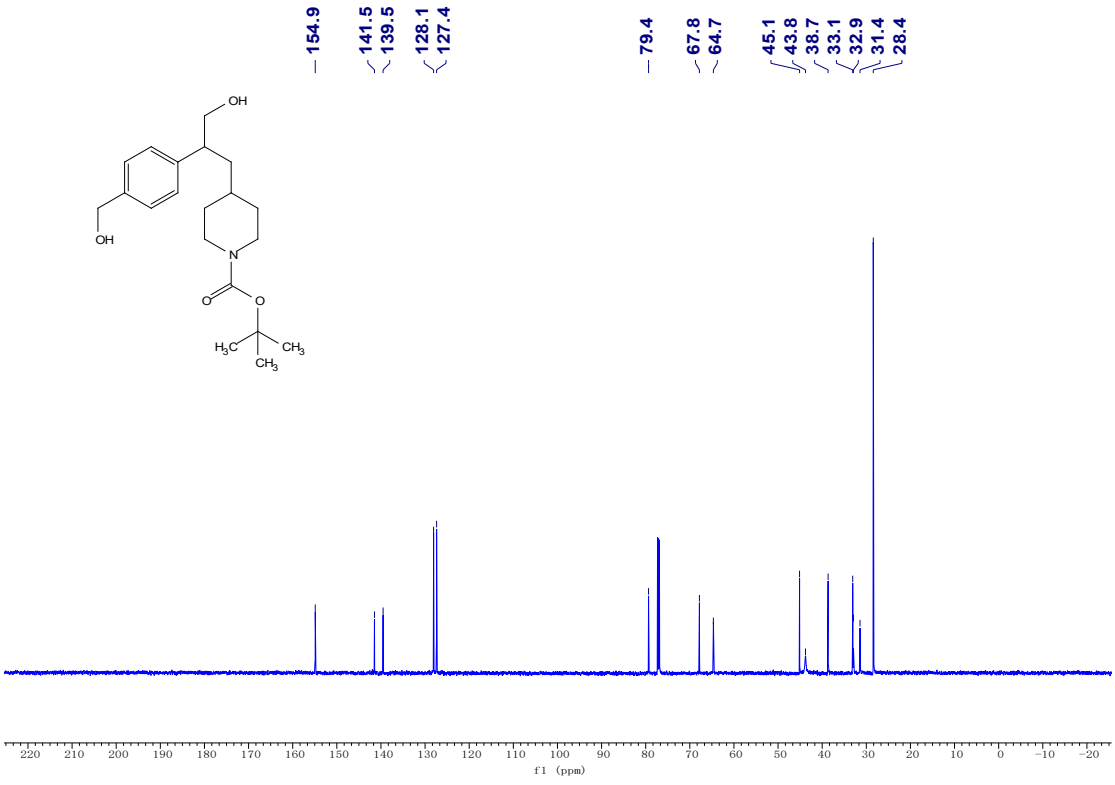
0.95
2.04
0.98
8.98
1.11
2.04
f1 (ppm)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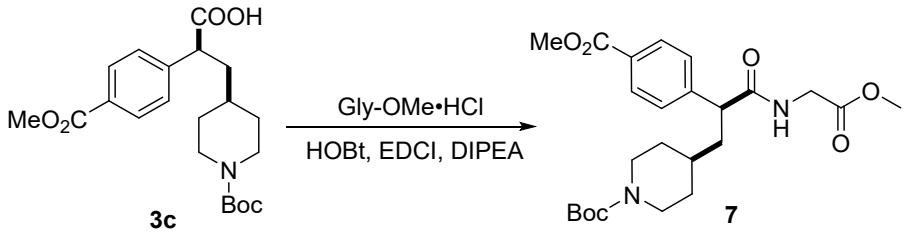
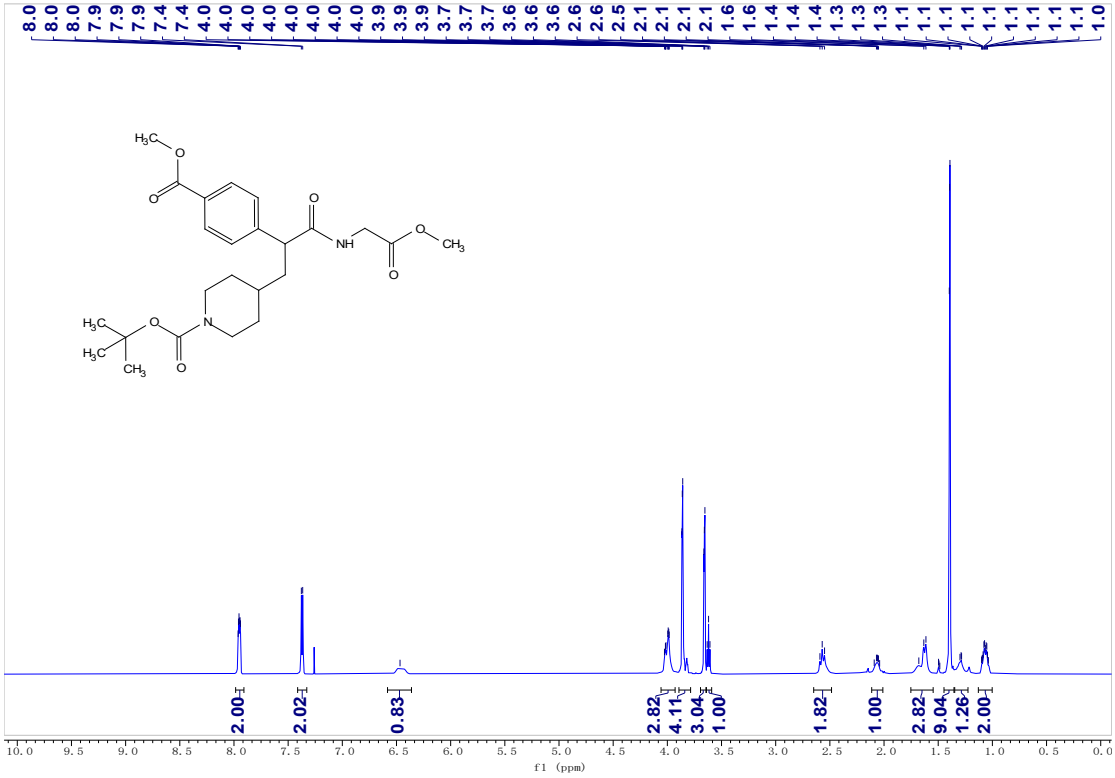
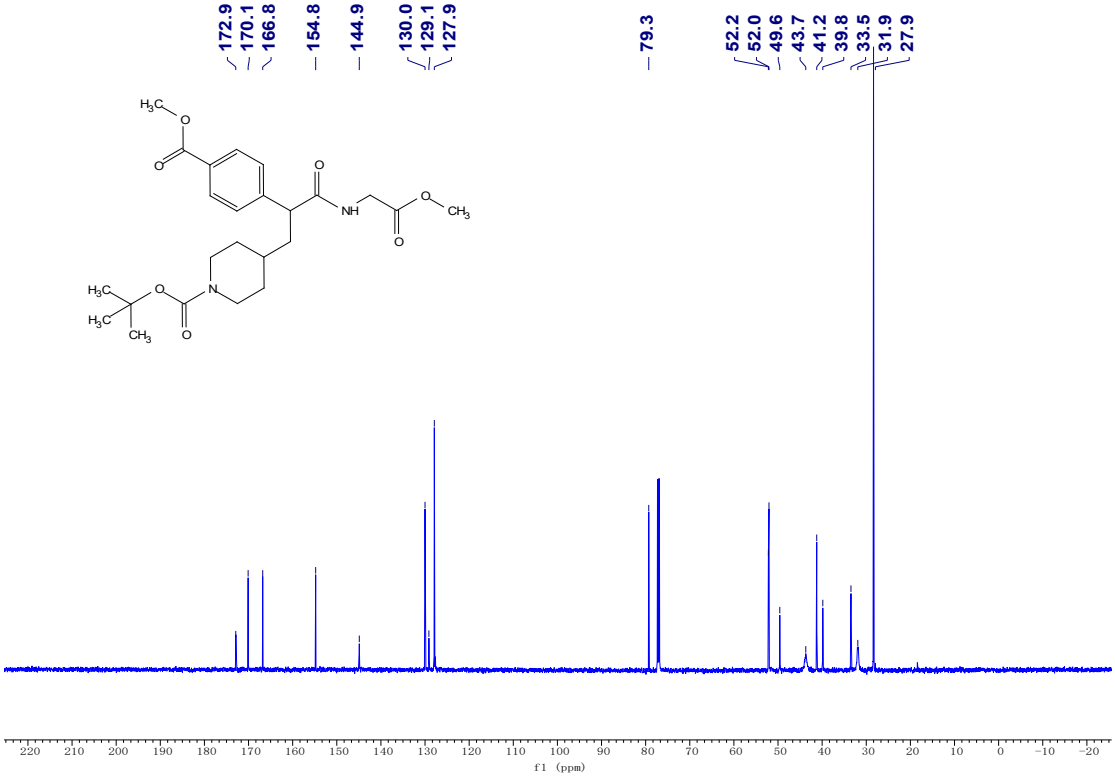
f1 (ppm)

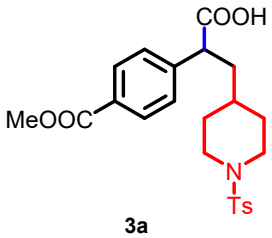


1-(3-Dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDCl: 46 mg, 0.24 mmol, 1.2 equiv) and 1-hydroxybenzotriazole (HOBt: 32.4 mg, 0.24 mmol, 1.2 equiv) S60 were added to solutions of **3c** (78.2 mg, 0.2 mmol, 1.0 equiv), glycine methyl ester hydrochloride (Gly-OMe·HCl: 30.1 mg, 0.24 mmol, 1.2 equiv), and i-Pr_2NEt (38.8 mg, 0.5 mmol, 2.5 equiv) in $CHCl_3$ (2.0 mL) at 0 °C. The mixtures were stirred for 2 h at 0 °C and 10 h at room temperature. The organic layer is evaporated and purified by silica gel flash column chromatography (petroleum ether/EtOAc 5/1) to give the final product as an oil (79.5 mg, 0.172 mmol, 86%). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 – 7.92 (m, 2H), 7.37 (d, *J* = 7.5 Hz, 22H), 6.47 (s, 1H), 4.05 – 3.95 (m, 3H), 3.91 – 3.82 (m, 4H), 3.69 – 3.63 (m, 3H), 3.62 (t, *J* = 7.7 Hz, 1H), 2.56 (d, *J* = 13.0 Hz, 2H), 2.15 – 1.98 (m, 1H), 1.81 – 1.55 (m, 3H), 1.40 – 1.39 (m, 9H), 1.33 – 1.27 (m, 1H), 1.13 – 0.98 (m, 2H).^{13}C NMR (151 MHz, CHLOROFORM-*D*) δ 172.90, 170.09, 166.79, 154.79, 144.90, 130.00, 129.12, 127.90, 79.29, 52.18, 52.05, 49.60, 43.71, 41.23, 39.85, 33.47, 31.91, 27.92. **HRMS (ESI+)** $[M+H]^+$ calculated m/z for $[C_{24}H_{35}N_2O_7]^+$: 463.2430, found:463.2422


8.0 8.0 8.0 7.9 7.9 7.9 7.4 7.4 4.0 4.0 4.0 4.0 4.0 4.0 4.0 3.9 3.9 3.9 3.7 3.7 3.6 3.6 3.6 2.6 2.6 2.5 2.1 2.1 2.1 2.1 1.6 1.6 1.4 1.4 1.4 1.3 1.3 1.1 1.1 1.1 1.1 1.1 1.1 1.1 1.1 1.0
2.00 2.02 0.83 2.82 4.11 3.04 1.00 1.82 1.00 2.82 9.04 1.26 2.00
f1 (ppm)


172.9 170.1 166.8 154.8 144.9 130.0 129.1 127.9 79.3 52.2 52.0 49.6 43.7 41.2 39.8 33.5 31.9 27.9
f1 (ppm)

5. Synthesis and Characterization of Products



3a

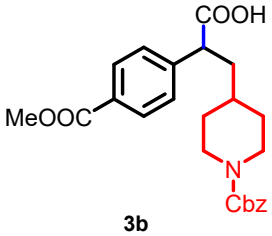
2-(4-(methoxycarbonyl)phenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 73 mg (82%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 7.6 Hz, 1H), 7.32 (d, *J* = 8.3 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 3.88 (s, 1H), 3.75 – 3.69 (m, 2H), 3.66 (t, *J* = 7.8 Hz, 1H), 2.40 (s, 2H), 2.12 – 2.03 (m, 2H), 1.97 (dt, *J* = 14.6, 7.5 Hz, 1H), 1.78 – 1.63 (m, 2H), 1.35 – 1.24 (m, 2H), 1.08 – 0.99 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 178.5, 166.7, 143.5, 143.0, 132.9, 130.1, 129.6, 129.6, 128.1, 127.7, 52.3, 48.4, 46.2, 46.2, 39.0, 32.7, 31.5, 31.0, 21.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{27}NNaO_6S]^+$: 468.1451, found: 468.1449



3b

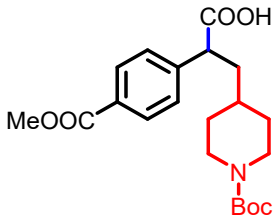
3-(1-((benzyloxy)carbonyl)piperidin-4-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 65 mg (76%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 – 7.95 (m, 2H), 7.40 – 7.36 (m, 2H), 7.36 – 7.25 (m, 5H), 5.09 (s, 2H), 4.22 – 4.05 (m, 2H), 3.90 (s, 3H), 3.73 (t, *J* = 7.8 Hz, 1H), 2.77 – 2.54 (m, 2H), 2.10 – 1.91 (m, 1H), 1.77 – 1.58 (m, 3H), 1.40 – 1.25 (m, 1H), 1.21 – 1.02 (m, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.8, 166.8, 155.4, 143.5, 136.7, 130.1, 129.4, 128.5, 128.1, 128.0, 127.9, 67.2, 52.2, 48.5, 44.0, 43.9, 39.5, 33.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{24}H_{27}NNaO_6]^+$: 448.1731, found: 448.1723



3c

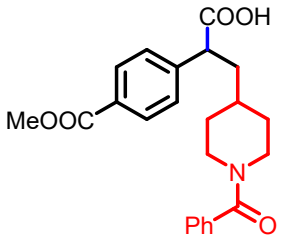
3-(1-(tert-butoxycarbonyl)piperidin-4-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 57 mg (73%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), δ 4.19 – 3.94 (m, 2H), 3.88 (s, 3H), 3.72 (t, *J* = 7.8 Hz, 1H), 2.69 – 2.46 (m, 1H)., 2.07 – 1.96 (m, 1H), 1.71 (m, 1H), 1.66 – 1.56 (m, 2H), 1.41 (s, 9H), 1.34 – 1.25 (m, 1H), 1.13 – 1.03 (m, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 188.6, 178.0, 155.0, 143.7, 130.1, 129.4, 128.2, 79.7, 52.2, 48.6, 39.6, 33.7, 28.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{25}NNaO_5]^+$: 414.1887, found: 414.1879



3d

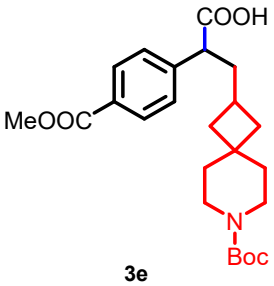
3-(1-benzoylpiperidin-4-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 52 mg (65%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.8 Hz, 2H), 7.40 – 7.31 (m, 7H), 4.65 (d, *J* = 12.8 Hz, 1H), 3.90 (s, 3H), 3.69 (t, *J* = 8.1 Hz, 2H), 2.88 (t, *J* = 13.1 Hz, 1H), 2.71 – 2.59 (m, 1H), 2.09 – 2.00 (m, 1H), 1.84 – 1.76 (m, 1H), 1.75 – 1.68 (m, 1H), 1.67 – 1.55 (m, 1H), 1.48 – 1.38 (m, 1H), 1.22 (s, 1H), 1.17 – 1.04 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 176.9, 170.7, 166.9, 143.7, 135.7, 130.1, 129.7, 129.4, 128.5, 128.1, 126.9, 52.3, 48.6, 47.9, 42.4, 39.5, 33.9.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{25}NNaO_5]^+$: 418.1625, found: 418.1620



3e

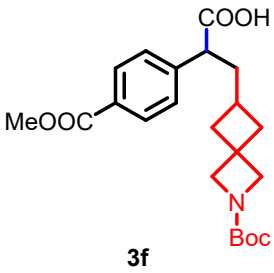
3-(7-(tert-butoxycarbonyl)-7-azaspiro[3.5]nonan-2-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 63 mg (72%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.2 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 3.89 (s, 3H), 3.53 (t, *J* = 7.6 Hz, 1H), 3.24 (d, *J* = 38.6 Hz, 4H), 2.22 – 2.15 (m, 1H), 2.13 – 2.05 (m, 1H), 1.94 – 1.86 (m, 2H), 1.88 – 1.77 (m, 1H), 1.56 – 1.19 (m, 15H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 178.4, 166.9, 155.1, 143.6, 130.0, 129.4, 128.2, 79.5, 52.2, 49.7, 41.0, 37.8, 37.6, 34.1, 26.9.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{24}H_{33}NNaO_6]^+$: 454.2200, found: 454.2192



3f

3-(2-(tert-butoxycarbonyl)-2-azaspiro[3.3]heptan-6-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

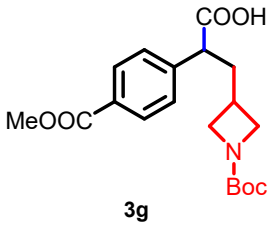
The general procedure A was followed. Colorless oil. Yield: 61 mg (75%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 3.87 (s, 3H), 3.83 – 3.77 (m, 2H), 3.73 – 3.66 (m, 2H), 3.48 (t, *J* = 7.6 Hz, 1H), 2.22 – 2.15

(m, 1H), 2.15 – 2.04 (m, 2H), 2.01 – 1.92 (m, 1H), 1.85 – 1.72 (m, 2H), 1.67 – 1.59 (m, 1H), 1.37 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.0, 166.9, 156.4, 143.8, 129.9, 129.2, 128.1, 79.7, 52.2, 49.7, 39.8, 38.9, 38.6, 34.1, 28.3, 27.8.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{22}H_{29}NNaO_6]^+$: 426.1887, found: 426.1880



3g

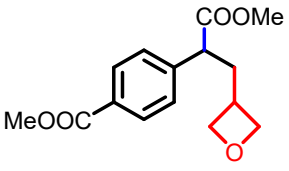
3-(1-(tert-butoxycarbonyl)azetidin-3-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 52 mg (71%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 2H), 3.94 (t, *J* = 8.2 Hz, 1H), 3.89 (s, 3H), 3.85 (t, *J* = 8.2 Hz, 1H), 3.56 (dd, *J* = 8.7, 6.0 Hz, 1H), 3.52 (t, *J* = 7.5 Hz, 1H), 3.43 (t, *J* = 7.2 Hz, 1H), 2.46 – 2.31 (m, 2H), 2.07 (d, *J* = 7.3 Hz, 1H), 1.38 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 176.8, 166.8, 156.5, 143.0, 130.1, 129.6, 128.1, 79.9, 52.3, 49.5, 37.6, 28.4, 27.0.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{19}H_{25}NNaO_6]^+$: 386.1574, found: 386.1568



3h

methyl 4-(1-methoxy-3-(oxetan-3-yl)-1-oxopropan-2-yl)benzoate

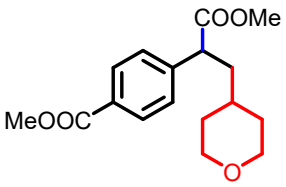
The general procedure A was followed. Colorless oil. Yield: 26 mg (45%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 4.72 – 4.63 (m, 1H), 4.59 – 4.50 (m, 1H), 4.40 – 4.31 (m, 1H), 4.24 – 4.14 (m, 1H), 3.87

(s, 3H), 3.63 (s, 3H), 3.49 (t, *J* = 7.7 Hz, 1H), 2.95 – 2.81 (m, 1H), 2.54 – 2.36 (m, 1H), 2.26 – 2.08 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 173.3, 166.7, 143.4, 130.2, 129.6, 128.0, 52.4, 52.2, 49.6, 37.3, 33.6.

HRMS (ESI+) $[M+H]^+$ calculated m/z for $[C_{15}H_{19}O_5]^+$: 279.1227, found: 279.1224



3i

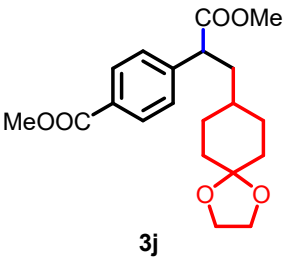
methyl 4-(1-methoxy-1-oxo-3-(tetrahydro-2H-pyran-4-yl)propan-2-yl)benzoate

The general procedure A was followed. Colorless oil. Yield: 34 mg (55%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 3.85 (s, 5H), 3.72 (t, *J* = 7.8 Hz, 1H), 3.61 (s, 3H), 3.29 – 3.18 (m, 2H), 2.09 – 1.92 (m, 1H), 1.74 – 1.65 (m, 1H), 1.54 (dd, *J* = 21.7, 12.9 Hz, 2H), 1.39 – 1.29 (m, 1H), 1.29 – 1.20 (m, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 173.8, 166.7, 144.0, 129.9, 129.2, 127.9, 67.7, 67.6, 52.1, 52.0, 48.2, 40.2, 32.9, 32.6, 32.6.

HRMS (ESI+) $[M+H]^+$ calculated m/z for $[C_{17}H_{23}O_5]^+$: 307.1540, found: 307.1540



3j

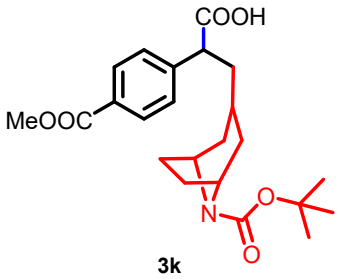
methyl 4-(1-methoxy-1-oxo-3-(1,4-dioxaspiro[4.5]decan-8-yl)propan-2-yl)benzoate

The general procedure A was followed. Colorless oil. Yield: 42 mg (58%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 2H), 3.84 (d, *J* = 5.7 Hz, 7H), 3.68 (t, *J* = 7.8 Hz, 1H), 3.59 (s, 3H), 2.04 – 1.93 (m, 1H), 1.73 – 1.58 (m, 5H), 1.37 (t, *J* = 12.8 Hz, 2H), 1.28 – 1.16 (m, 2H), 1.16 – 1.08 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 173.8, 166.7, 144.3, 129.9, 129.1, 127.9, 108.7, 64.1, 52.1, 52.0, 49.1, 39.7, 34.2, 34.2, 33.9, 30.0, 29.7.

HRMS (ESI+) $[M+H]^+$ calculated m/z for $[C_{20}H_{27}O_6]^+$: 363.1802, found: 363.1801



3k

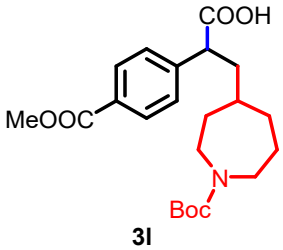
3-(8-(tert-butoxycarbonyl)-8-azabicyclo[3.2.1]octan-3-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. colorless oil. Yield: 72 mg (86%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.1 Hz, 2H), 4.17 (s, 1H), 4.08 (s, 1H), 3.89 (s, 3H), 3.67 (t, *J* = 7.6 Hz, 1H), 1.98 – 1.89 (m, 1H), 1.83 (s, 2H), 1.66 (s, 2H), 1.58 – 1.47 (m, 2H), 1.47 – 1.32 (m, 12H), 1.28 (s, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.6, 166.9, 153.5, 143.8, 130.1, 129.5, 128.1, 79.5, 53.4, 52.2, 48.6, 40.1, 37.7, 28.6, 27.6, 26.3.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{31}NNaO_6]^+$: 440.2044, found: 440.2044



3l

3-(1-(tert-butoxycarbonyl)azepan-4-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

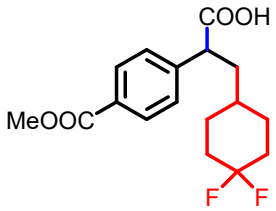
The general procedure A was followed. Colorless oil. Yield: 52 mg (63%) dr = 1:1

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.4 Hz, 2H), 7.37 (d, *J* = 7.5 Hz, 2H), 3.89 (s, 3H), 3.75 – 3.64 (m, 1H), 3.61 – 3.52 (m, 1H), 3.48 – 3.40 (m, 1H), 3.40 – 3.34

(m, 1H), 3.33 – 3.22 (m, 1H), 3.10 – 3.01 (m, 1H), 2.07 – 1.97 (m, 1H), 1.85 – 1.63 (m, 4H), 1.53 – 1.22 (m, 12H), 1.22 – 1.11 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 178.2, 166.9, 155.7, 143.8, 143.8, 130.1, 129.4, 129.3, 128.2, 128.2, 79.4, 52.2, 49.2, 49.2, 46.8, 46.3, 46.31, 44.9, 44.6, 40.3, 40.2, 36.6, 36.4, 34.9, , 34.3, 32.9, 32.4, 28.5, 26.8, 26.7, 26.6.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{22}H_{31}NNaO_6]^+$: 428.2044, found: 440.2044



3m

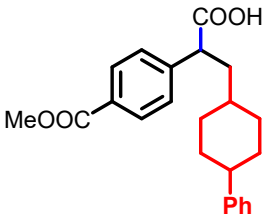
3-(4,4-difluorocyclohexyl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 36 mg (55%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.3 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 3.89 (s, 3H), 3.73 (t, *J* = 7.8 Hz, 1H), 2.14 – 1.93 (m, 3H), 1.88 – 1.68 (m, 3H), 1.67 – 1.51 (m, 2H), 1.32 – 1.19 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.1, 166.8, 143.3, 130.1, 129.5, 128.1, 123.4 (t, J = 241.2, 239.0 Hz), 52.2, 49.0, 38.7, 33.39 (d, *J* = 10.1 Hz), 33.3 (d, *J* = 4.3 Hz), 33.1 (d, *J* = 5.8 Hz), 33.0, 28.9(d, *J* = 9.3 Hz), 28.5 (d, *J* = 9.4 Hz). ^{19}F NMR (565 MHz, CHLOROFORM-*D*) δ -91.7 (d, *J* = 235.3 Hz), -102.0 (d, *J* = 235.3 Hz).

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{17}H_{20}F_2NaO_4]^+$: 349.1222, found: 349.1223



2-(4-(methoxycarbonyl)phenyl)-3-(4-phenylcyclohexyl)propanoic acid

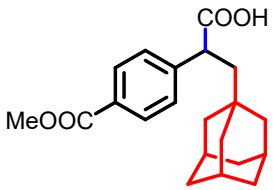
The general procedure A was followed. White solid. Yield: 50 mg (68%), dr = 1.8:1

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.44 (dd, *J* = 8.3, 5.1 Hz, 1H), 7.34 – 7.23 (m, 1H), 7.26 – 7.13 (m, 2H), 3.92 (d, *J* = 1.5 Hz, 1H), 3.82 (t, *J* = 7.8

Hz, 0.35H), 3.73 (t, *J* = 7.8 Hz, 0.62H), δ 2.63 – 2.51 (m, 0.59H), 2.50 – 2.40 (m, 0.34H), 2.34 – 2.20 (m, 0.61H), 2.16 – 1.99 (m, 0.41H), 2.00 – 1.85 (m, 2H), 1.85 – 1.75 (m, 0.39H), 1.76 – 1.56 (m, 5.7H), 1.47 – 1.34 (m, 0.75H), 1.33 – 1.19 (m, 0.51H), 1.17 – 1.06 (m, 0.71H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.5, 166.9, 147.4, 147.0, 143.8, 143.7, 130.1, 129.5, 129.4, 128.4, 128.3, 126.9, 126.8, 126.0, 125.9, 52.2, 49.6, 48.9, 44.4, 43.4, 40.5, 35.2, 34.9, 34.0, 33.9, 33.5, 33.1, 30.5, 30.1, 29.6, 28.8, 28.6.

HRMS (ESI-) $[M-H]^-$ calculated m/z for $[C_{23}H_{25}O_4]^-$: 365.1758, found: 356.1761



3o

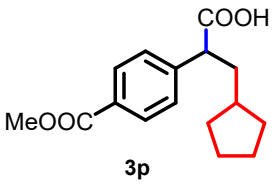
3-(adamantan-1-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic

The general procedure A was followed. White solid. Yield: 43 mg (62%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 8.3 Hz, 1H), 7.39 (d, *J* = 8.3 Hz, 1H), 3.89 (s, 1H), 3.77 (dd, *J* = 8.3, 4.5 Hz, 1H), 2.13 (dd, *J* = 14.2, 8.3 Hz, 1H), 1.91 (s, 2H), 1.66 (d, *J* = 12.2 Hz, 4H), 1.58 (d, *J* = 11.5 Hz, 2H), 1.48 (d, *J* = 14.2 Hz, 2H), 1.41 (d, *J* = 11.9 Hz, 2H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 180.2, 166.9, 145.6, 130.0, 129.1, 128.1, 128.1, 52.2, 47.5, 46.3, 42.34, 36.9, 28.6, 28.6.

HRMS (ESI-) $[M-H]^-$ calculated m/z for $[C_{21}H_{25}O_4]^-$: 341.1758, found: 341.1760



3p

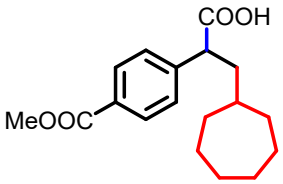
3-cyclopentyl-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 25 mg (44%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 – 7.95 (m, 2H), 7.42 – 7.34 (m, 2H), 7.39 – 7.25 (m, 5H), 5.09 (s, 1H), 4.22 – 4.05 (m, 1H), 3.90 (s, 1H), 2.77 – 2.54 (m, 1H), 2.10 – 1.91 (m, 1H), 1.77 – 1.58 (m, 2H), 1.40 – 1.25 (m, 1H), 1.21 – 1.02 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.5, 166.9, 143.8, 130.0, 129.4, 128.3, 52.2, 50.8, 39.5, 37.8, 32.7, 32.3, 25.1, 25.1.

HRMS (ESI-) $[M-H]^-$ calculated m/z for $[C_{26}H_{19}O_4]^-$: 275.1289, found: 275.1292



3q

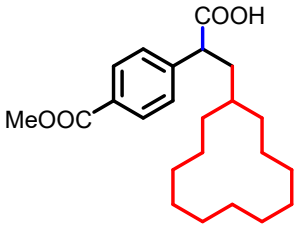
3-cycloheptyl-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 31 mg (51%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.3 Hz, 1H), 7.39 (d, *J* = 8.3 Hz, 3H), 3.90 (s, 1H), 3.72 (t, *J* = 7.8 Hz, 0H), 2.04 – 1.96 (m, 1H), 1.76 – 1.63 (m, 1H), 1.63 – 1.54 (m, 1H), 1.54 – 1.39 (m, 1H), 1.38 – 1.29 (m, 1H), 1.24 – 1.13 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.8, 166.9, 143.9, 130.0, 129.3, 128.3, 52.2, 49.4, 41.0, 36.5, 34.6, 33.9, 28.6, 28.5, 26.1, 26.0.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{18}H_{24}NaO_4]^+$: 327.1567, found: 327.1566



3r

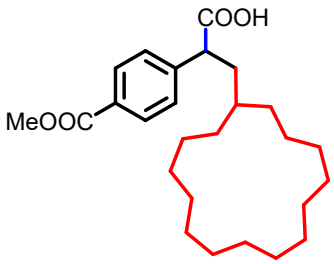
3-cyclododecyl-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 51 mg (67%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.2 Hz, 1H), 7.39 (d, *J* = 8.2 Hz, 1H), 3.73 (t, *J* = 7.8 Hz, 1H), 2.00 – 1.91 (m, 1H), 1.77 – 1.68 (m, 1H), 1.41 – 1.11 (m, 12H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.8, 166.9, 143.9, 130.0, 129.3, 128.3, 52.2, 49.4, 38.2, 31.6, 28.9, 28.8, 24.8, 24.6, 24.1, 23.5, 23.3, 23.1, 22.9, 21.5, 21.3.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{34}NaO_4]^+$: 397.2349, found: 397.2349



3s

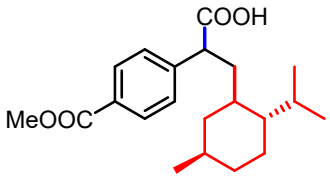
3-cyclopentadecyl-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 58 mg (69%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 3.90 (s, 3H), 3.72 (t, *J* = 7.7 Hz, 1H), 2.09 – 1.89 (m, 1H), 1.85 – 1.67 (m, 1H), 1.33 – 1.20 (m, 29H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 179.7, 166.9, 143.9, 130.0, 129.3, 128.4, 52.2, 49.4, 38.4, 34.0, 32.3, 32.0, 27.6, 27.0, 26.9, 26.8, 26.7, 26.7, 26.6, 26.4, 24.3, 24.2.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{26}H_{40}NaO_4]^+$: 439.2819, found: 439.2821



3t

3-((2S,5R)-2-isopropyl-5-methylcyclohexyl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

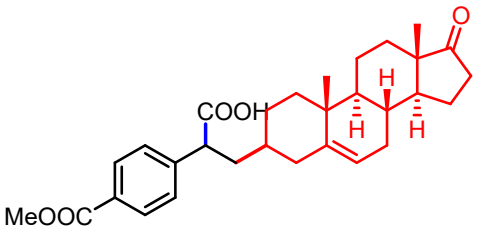
The general procedure A was followed. Colorless oil. Yield: 33 mg (47%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.03 – 7.94 (m, 2H), 7.43 (t, *J* = 8.7 Hz, 2H), 3.90 (s, 3H), 3.81 (dd, *J* = 11.2, 4.0 Hz, 0.6H), 3.72 (dd, *J* = 11.0, 4.3 Hz, 0.29H), 2.56 – 2.47 (m, 0.6H), 2.11 – 2.01 (m, 0.95H), 2.01 – 1.95 (m, 0.33H) 1.92 – 1.86 (m, 0.61H), 1.77 – 1.57 (m, 3H), 1.48 – 1.36 (m, 0.68H), 1.34 – 1.26 (m, 1.69H), 1.24 – 1.17 (m, 0.74H),

1.04 – 0.91 (m, 1.74H), 0.91 – 0.84 (m, 6.83H), 0.81 (d, *J* = 6.5 Hz, 1.72H), 0.74 (d, *J* = 6.9 Hz, 1.92H), 0.59 (q, *J* = 12.0 Hz, 0.61H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.2, 166.9, 144.8, 144.7, 130.1, 129.4, 129.3, 128.1, 128.0, 52.2, 49.4, 48.9, 48.4, 47.6, 41.4, 38.2, 37.7, 37.4, 35.8, 35.2, 33.3, 32.6, 30.0, 29.2, 26.5, 26.0, 24.9, 24.3, 22.8, 21.7, 21.7, 20.7, 15.2.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{21}H_{30}NaO_4]^+$: 369.2036, found: 369.2036



3u

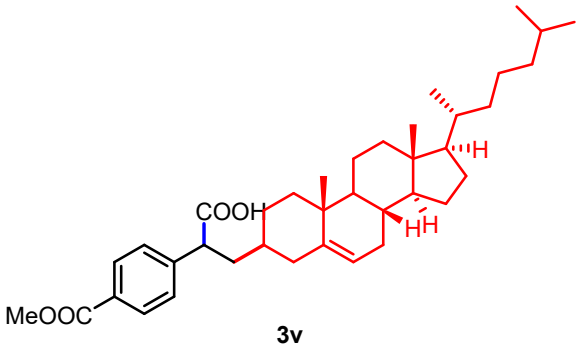
3-((8R,9S,10R,13S,14S)-10,13-dimethyl-17-oxo-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 51 mg (53%). dr = 4:1

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.0 Hz, 2H), 7.41 – 7.36 (m, 2H), 5.31 (d, *J* = 5.1 Hz, 0.73H), 5.24 (d, *J* = 5.1 Hz, 0.18H), 3.90 (s, 3H), 3.62 (t, *J* = 7.7 Hz, 1H), 2.50 – 2.39 (m, 2H), 2.13 – 1.98 (m, 3H), 1.96 – 1.89 (m, 1H), 1.88 – 1.76 (m, 2H), 1.76 – 1.60 (m, 6H), 1.59 – 1.37 (m, 4H), 1.30 – 1.11 (m, 3H), 1.08 – 1.01 (m, 1H), 1.00 (s, 3H), 0.86 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 221.6, 179.2, 166.9, 143.7, 140.1, 130.0, 129.5, 128.3, 121.1, 52.2, 51.9, 50.5, 49.2, 47.6, 37.6, 36.5, 35.9, 34.1, 34.0, 31.5, 30.8, 26.0, 21.9, 20.1, 19.5, 13.6.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{30}H_{38}NaO_5]^+$: 501.2611, found: 501.2614



3v

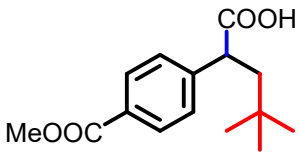
3-((8S,10R,13R,14S,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl)-2-(4-(methoxycarbonyl)phenyl)propanoic acid

The general procedure A was followed. White solid. Yield: 53 mg (46%). dr = 4.8:1

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 7.7 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 5.31 – 5.20 (m, 1H), 3.90 (s, 3H), 3.77 (t, *J* = 7.7 Hz, 0.15H), 3.63 (td, *J* = 7.8, 3.1 Hz, 0.72H), 2.43 (d, *J* = 13.4 Hz, 1H), 2.11 – 1.97 (m, 2H), 1.93 (d, *J* = 14.8 Hz, 1H), 1.87 – 1.60 (m, 5H), 1.59 – 1.49 (m, 4H), 1.49 – 1.30 (m, 7H), 1.29 – 1.19 (m, 2H), 1.19 – 1.01 (m, 7H), 1.00 – 0.95 (m, 5H), 0.94 (s, 1H), 0.91 (d, *J* = 6.5 Hz, 3H), 0.87 (dd, *J* = 6.7, 2.7 Hz, 6H), 0.66 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 179.7, 179.5, 166.9, 143.9, 143.8, 142.4, 139.9, 139.8, 130.0, 130.0, 129.3, 129.3, 128.4, 128.3, 128.2, 121.9, 121.8, 120.0, 56.8, 56.2, 52.2, 50.4, 49.4, 49.2, 42.3, 39.8, 39.6, 37.4, 37.4, 37.2, 36.7, 36.4, 36.3, 35.9, 34.3, 34.1, 34.0, 33.9, 31.9, 31.6, 28.30, 28.1, 26.1, 25.9, 24.3, 23.9, 22.9, 22.6, 20.8, 19.4, 18.8, 11.9.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{38}H_{56}NaO_4]^+$: 599.4071, found: 599.4078



3w

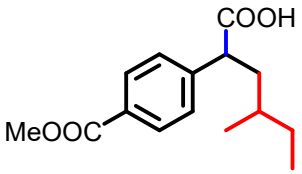
2-(4-(methoxycarbonyl)phenyl)-4,4-dimethylpentanoic acid

The general procedure A was followed. Colorless oil. Yield: 25mg (47%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 3.90 (s, 3H), 3.56 (t, *J* = 7.7 Hz, 1H), 2.13 – 2.04 (m, 1H), 1.83 – 1.74 (m, 1H), 1.57 – 1.48 (m, 1H), 1.23 – 1.14 (m, 1H), 1.11 – 1.02 (m, 1H), 0.85 (t, *J* = 7.0 Hz, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.15, 166.96, 143.79, 130.04, 129.43, 128.26, 52.21, 51.86, 36.57, 31.04, 27.92, 22.51, 22.38.

HRMS (ESI-) $[M-H]^+$ calculated m/z for $[C_{15}H_{19}O_4]^-$: 263.1289, found:263.1283



3x

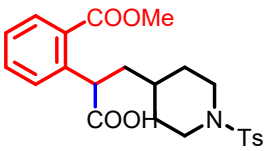
2-(4-(methoxycarbonyl)phenyl)-4-methylhexanoic acid

The general procedure A was followed. Colorless oil. Yield: 24mg (45%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 8.3, 2.7 Hz, 2H), 7.40 (t, *J* = 8.3 Hz, 2H), 3.90 (s, 3H), 3.77 – 3.71 (m, 1H), 2.17 – 2.09 (m, 0.5H), 1.93 – 1.80 (m, 1H), 1.57 – 1.50 (m, 0.5H), 1.45 – 1.28 (m, 2H), 1.22 – 1.11 (m, 1.5H), 0.91 – 0.83 (m, 4.5H), 0.81 (t, *J* = 7.2 Hz, 1.5H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 179.37, 179.16, 166.95, 144.15, 143.66, 130.05, 129.41, 129.38, 128.38, 128.20, 52.21, 49.39, 49.31, 40.24, 39.61, 32.29, 31.87, 29.52, 29.16, 19.07, 18.69, 11.10, 11.01.

HRMS (ESI-) $[M-H]^+$ calculated m/z for $[C_{15}H_{19}O_4]^-$: 263.1289, found:263.1287



4a

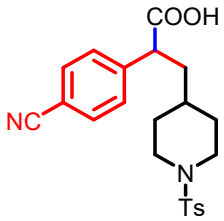
2-(2-(methoxycarbonyl)phenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 65 mg (73%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.84 (d, *J* = 7.8 Hz, 1H), 7.58 (d, *J* = 7.9 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 6.7 Hz, 3H), 4.66 (t, *J* = 7.4 Hz, 1H), 3.87 (s, 3H), 3.69 (d, *J* = 10.7 Hz, 2H), 2.39 (s, 3H), 2.19 – 1.99 (m, 3H), 1.71 (d, *J* = 13.2 Hz, 2H), 1.67 – 1.59 (m, 1H), 1.35 – 1.21 (m, 2H), 1.21 – 1.08 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 178.1, 168.4, 143.4, 139.5, 132.9, 132.5, 130.8, 129.5, 129.4, 128.4, 127.6, 127.3, 52.5, 46.2, 43.7, 38.8, 33.0, 31.3, 31.3, 21.4.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{27}NNaO_6S]^+$: 468.1451, found: 468.1449



4b

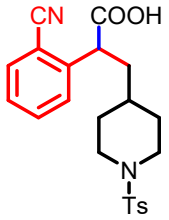
2-(4-cyanophenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 65 mg (78%)

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.2 Hz, 4H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 3.74 (d, *J* = 9.9 Hz, 2H), 3.67 (t, *J* = 7.8 Hz, 1H), 2.41 (s, 3H), 2.15 – 2.05 (m, 2H), 2.03 – 1.95 (m, 1H), 1.75 – 1.65 (m, 3H), 1.36 – 1.27 (m, 2H), 1.10 – 1.00 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.9, 143.7, 143.2, 133.0, 132.7, 129.7, 128.9, 127.8, 118.4, 111.8, 48.4, 46.2, 46.1, 39.1, 32.8, 31.5, 31.1, 21.6.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{22}H_{24}N_2NaO_4S]^+$: 435.1349, found: 435.1348



4c

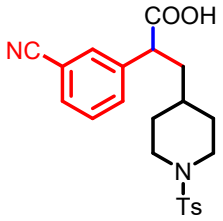
2-(2-cyanophenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 61 mg (74%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.62 – 7.53 (m, 4H), 7.45 (d, *J* = 7.9 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 4.10 (t, *J* = 7.5 Hz, 1H), 3.72 (t, 2H), 2.39 (s, 3H), 2.13 (t, *J* = 11.9 Hz, 2H), 2.08 – 2.00 (m, 1H), 1.80 (d, *J* = 13.1 Hz, 1H), 1.75 – 1.64 (m, 2H), 1.37 – 1.26 (m, 2H), 1.13 – 1.02 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.3, 143.5, 141.8, 133.4, 133.1, 129.6, 128.1, 127.9, 127.6, 117.4, 112.9, 46.2, 46.1, 39.3, 32.9, 31.3, 31.1, 21.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{22}H_{24}N_2NaO_4S]^+$: 435.1349, found: 435.1349



4d

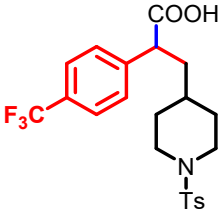
2-(3-cyanophenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 54 mg (65%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.0 Hz, 2H), 7.55 (d, *J* = 9.3 Hz, 2H), 7.50 (d, *J* = 8.1 Hz, 1H), 7.41 (t, *J* = 7.7 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 3.73 (d, *J* = 9.8 Hz, 2H), 3.64 (t, *J* = 7.8 Hz, 1H), 2.41 (s, 3H), 2.16 – 2.06 (m, 2H), 2.03 – 1.96 (m, 1H), 1.74 – 1.65 (m, 3H), 1.36 – 1.27 (m, 2H), 1.11 – 1.00 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.9, 143.6, 139.5, 133.0, 132.7, 131.6, 131.4, 129.7, 129.7, 127.7, 118.4, 113.0, 48.0, 46.2, 46.1, 39.2, 32.8, 31.4, 31.1, 21.6.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{22}H_{24}N_2NaO_4S]^+$: 435.1349, found: 435.1346



4e

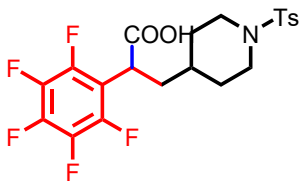
3-(1-tosylpiperidin-4-yl)-2-(4-(trifluoromethyl)phenyl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 61 mg (67%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.2 Hz, 1H), 7.54 (d, *J* = 8.1 Hz, 1H), 7.38 (d, *J* = 8.1 Hz, 1H), 7.28 (d, *J* = 8.1 Hz, 1H), 3.75 – 3.70 (m, 1H), 3.67 (t, *J* = 7.8 Hz, 1H), 2.40 (s, 2H), 2.17 – 2.05 (m, 1H), 1.98 (dt, *J* = 13.9, 7.6 Hz, 1H), 1.75 – 1.67 (m, 1H), 1.37 – 1.26 (m, 1H), 1.07 (m, 1H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 178.4, 143.6, 142.0, 132.9, 130.0 (q, *J* = 32.7 Hz), 129.7, 128.5, 127.7, 125.8 (d, *J* = 4.1 Hz), 124.0 (q, *J* = 272.2 Hz), 48.3, 46.2, 46.1, 39.1, 32.7, 31.4, 31.0, 21.5.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -62.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{22}H_{24}F_3NNaO_4S]^+$: 478.1270, found: 478.1269



4f

2-(perfluorophenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

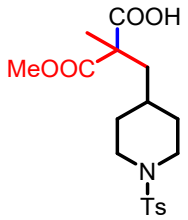
The general procedure A was followed. White Solid. Yield: 69 mg (72%)

^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 4.02 (dd, *J* = 9.6, 5.8 Hz, 1H), 3.78 – 3.67 (m, 2H), 2.39 (s, 3H), 2.16 – 2.04 (m, 3H), 1.82 – 1.70 (m, 2H), 1.62 (d, *J* = 10.5 Hz, 1H), 1.39 – 1.22 (m, 2H), 1.06 – 0.93 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ176.3, 146.0-144.3 (m, 2C), 143.6, 141.6-139.8(m, 1C), 138.6-136.7 (m, 2C), 133.0, 129.7, 127.7, 112.2 (t, *J* = 17.9 Hz), 46.2, 46.1, 37.5, 36.3, 33.0, 31.7, 30.6, 21.5.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -141.4 (dd, *J* = 21.7, 6.5 Hz), -154.2 (t, *J* = 21.0 Hz), -161.1 (td, *J* = 21.3, 6.6 Hz).

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{21}H_{20}F_5NNaO_4S]^+$: 500.0925, found: 500.0922



4g

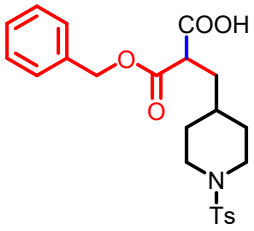
3-methoxy-2-methyl-3-oxo-2-((1-tosylpiperidin-4-yl)methyl)propanoic acid

The general procedure A was followed. White Solid. Yield: 60 mg (77%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 7.9 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 3.70 (s, 3H), 3.68 (d, *J* = 11.7 Hz, 2H), 2.42 (s, 3H), 2.20 – 2.13 (m, 2H), 1.91 – 1.78 (m, 2H), 1.63 (d, *J* = 12.7 Hz, 2H), 1.40 (s, 3H), 1.38 – 1.31 (m, 2H), 1.31 – 1.24 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.3, 173.1, 143.6, 132.8, 129.7, 127.8, 52.8, 52.8, 46.3, 41.5, 32.3, 32.2, 31.7, 20.9.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{18}H_{25}NNaO_6S]^+$: 406.1295, found: 406.1292



4h

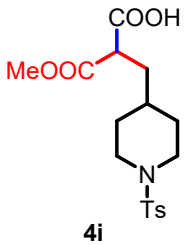
3-(benzyloxy)-3-oxo-2-((1-tosylpiperidin-4-yl)methyl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 58 mg (65%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 6.6 Hz, 2H), 7.36 – 7.18 (m, 7H), 5.28 – 5.03 (m, 2H), 3.69 (d, *J* = 14.4 Hz, 2H), 3.52 – 3.36 (m, 2H), 2.43 (s, 3H), 2.07 (q, *J* = 12.1 Hz, 2H), 1.90 – 1.73 (m, 2H), 1.65 (t, *J* = 18.0 Hz, 2H), 1.32 – 1.21 (m, 2H), 1.12 – 1.00 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 173.2, 169.1, 143.6, 135.2, 132.9, 129.6, 128.6, 128.5, 128.5, 128.4, 127.7, 67.3, 49.0, 46.1, 46.1, 34.8, 32.8, 31.1, 30.8, 21.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{23}H_{27}NNaO_6S]^+$: 468.1451, found: 468.1447



4i

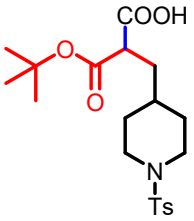
3-methoxy-3-oxo-2-((1-tosylpiperidin-4-yl)methyl)propanoic acid

The general procedure A was followed. White Solid. Yield: 51 mg (68%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 7.8 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 3.75 – 3.65 (m, 5H), 3.40 (t, *J* = 7.6 Hz, 1H), 2.39 (s, 3H), 2.16 (t, *J* = 11.9 Hz, 2H), 1.86 – 1.74 (m, 2H), 1.70 (d, *J* = 12.4 Hz, 2H), 1.32 – 1.11 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 173.9, 169.5, 143.6, 132.9, 129.6, 127.6, 52.7, 48.8, 46.1, 34.7, 32.9, 31.0, 21.5.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{17}H_{23}NNaO_6S]^+$: 392.1138, found: 392.1132



4j

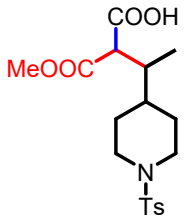
3-(tert-butoxy)-3-oxo-2-((1-tosylpiperidin-4-yl)methyl)propanoic acid

The general procedure A was followed. White Solid. Yield: 59 mg (72%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.58 (d, *J* = 8.3 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 3.66 (d, *J* = 11.4 Hz, 2H), 2.40 (s, 3H), 2.17 (t, *J* = 11.6 Hz, 2H), 1.83 – 1.75 (m, 2H), 1.70 – 1.62 (m, 2H), 1.39 (s, 9H), 1.36 – 1.25 (m, 6H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 177.7, 171.9, 143.5, 132.8, 129.6, 127.7, 82.6, 53.4, 46.3, 41.2, 32.3, 32.2, 31.7, 27.7, 21.5, 20.8.

HRMS (ESI+) $[M-H]^-$ calculated m/z for $[C_{20}H_{28}NO_6S]^-$: 410.1643, found: 410.1637



4k

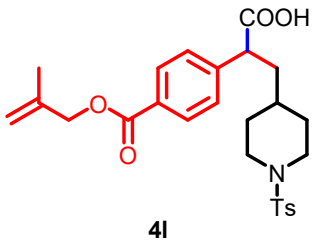
2-(methoxycarbonyl)-3-(1-tosylpiperidin-4-yl)butanoic acid

The general procedure A was followed. Colorless oil. Yield: 37 mg (48%). dr = 1:1

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 3.79 (d, *J* = 11.7 Hz, 2H), 3.68 (s, 3H), 3.42 – 3.33 (m, 1H), 2.39 (s, 3H), 2.19 – 2.03 (m, 3H), 1.72 – 1.55 (m, 2H), 1.51 – 1.42 (m, 1H), 1.38 – 1.17 (m, 2H), 0.94 – 0.84 (m, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 173.0, 172.7, 169.7, 169.2, 143.6, 133.0, 132.9, 129.7, 127.7, 54.6, 54.4, 52.7, 52.5, 46.5, 46.4, 37.8, 37.7, 29.7, 26.8, 26.6, 21.5, 13.1, 13.0.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{18}H_{25}NNaO_6S]^+$: 406.1295, found: 406.1290



4l

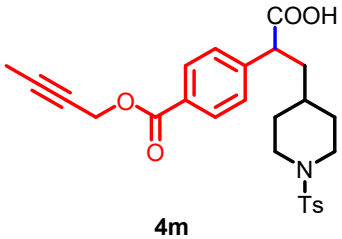
2-(4-(((2-methylallyl)oxy)carbonyl)phenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 72 mg (74%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.99 (d, *J* = 8.3 Hz, 2H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 5.00 (d, *J* = 43.0 Hz, 2H), 4.72 (s, 2H), 3.76 – 3.64 (m, 3H), 2.40 (s, 3H), 2.13 – 2.05 (m, 2H), 2.02 – 1.94 (m, 1H), 1.81 (s, 3H), 1.79 – 1.64 (m, 3H), 1.36 – 1.23 (m, 3H), 1.10 – 1.00 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 178.3, 165.9, 143.6, 143.2, 139.9, 133.0, 130.2, 129.7, 129.7, 128.2, 127.7, 113.1, 68.3, 48.4, 46.2, 46.2, 39.0, 32.7, 31.5, 31.0, 21.5, 19.6.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{26}H_{31}NNaO_6S]^+$: 508.1764, found: 508.1761



4m

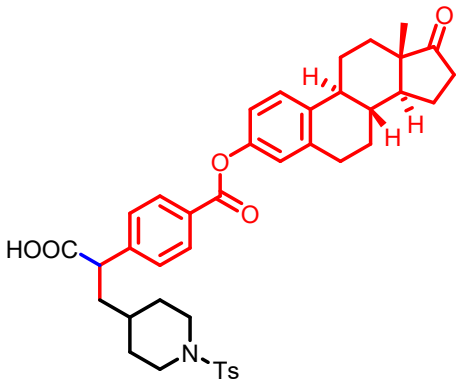
2-(4-((but-2-yn-1-yloxy)carbonyl)phenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. Colorless oil. Yield: 77 mg (79%).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.7 Hz, 2H), 7.57 (d, *J* = 7.8 Hz, 2H), 7.29 (dd, *J* = 26.8, 8.1 Hz, 4H), 5.04 – 4.70 (m, 2H), 3.83 – 3.46 (m, 3H), 2.39 (s, 3H), 2.07 (q, *J* = 13.1 Hz, 2H), 2.01 – 1.91 (m, 1H), 1.89 – 1.81 (m, 3H), 1.78 – 1.61 (m, 3H), 1.35 – 1.23 (m, 2H), 1.10 – 0.95 (m, 1H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 178.6, 165.6, 143.5, 143.2, 132.8, 130.2, 129.6, 129.1, 128.1, 127.6, 83.4, 73.1, 53.4, 48.4, 46.2, 46.1, 38.9, 32.6, 31.4, 31.9, 21.5, 3.6.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{26}H_{29}NNaO_6S]^+$: 506.1608, found: 506.1605



4n

2-(4-((((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl)oxy)carbonyl)phenyl)-3-(1-tosylpiperidin-4-yl)propanoic acid

The general procedure A was followed. White Solid. Yield: 85 mg (62%)

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.10 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.1 Hz, 2H), 7.39 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.5 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 6.93 (dd, *J* = 8.5, 2.6 Hz, 1H), 6.89 (d, *J* = 2.5 Hz, 1H), 3.78 – 3.67 (m, 3H), 2.99 – 2.85 (m, 2H), 2.50 (dd,

J = 19.1, 8.8 Hz, 1H), 2.40 (s, 4H), 2.33 – 2.25 (m, 1H), 2.18 – 1.92 (m, 7H), 1.79 – 1.40 (m, 9H), 1.37 – 1.27 (m, 2H), 1.13 – 1.03 (m, 1H), 0.90 (s, 3H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 221.3, 178.0, 165.0, 148.7, 143.8, 143.6, 138.2, 137.6, 132.9, 130.7, 129.7, 129.1, 128.3, 127.7, 126.5, 121.6, 118.8, 50.4, 48.5, 48.0, 46.2, 46.2, 44.2, 39.0, 38.0, 35.9, 32.8, 31.5, 31.0, 29.5, 26.4, 25.8, 21.6, 21.6, 13.9.

HRMS (ESI+) $[M+Na]^+$ calculated m/z for $[C_{40}H_{45}NNaO_7S]^+$: 706.2809, found: 706.2802

6. Reference

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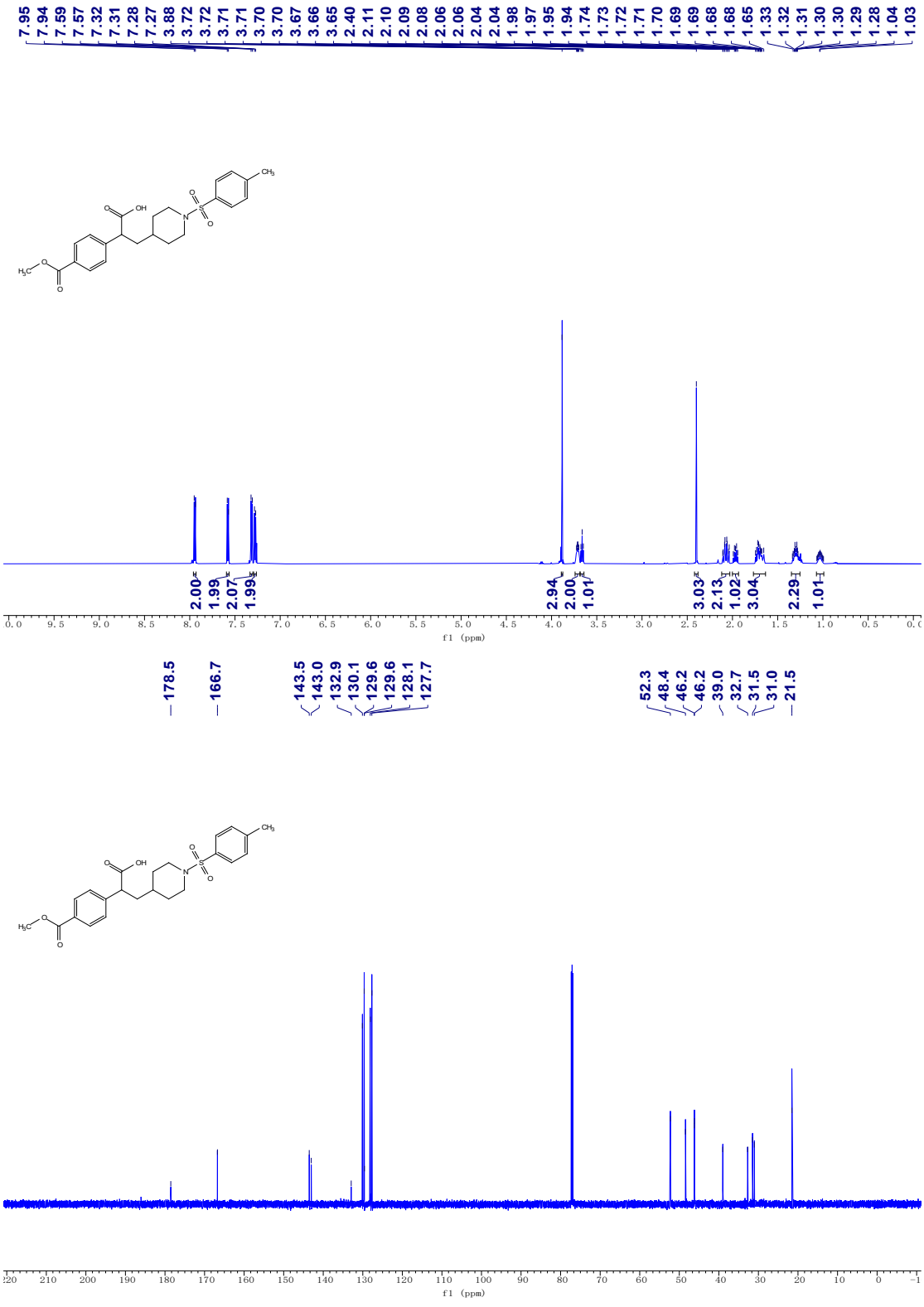
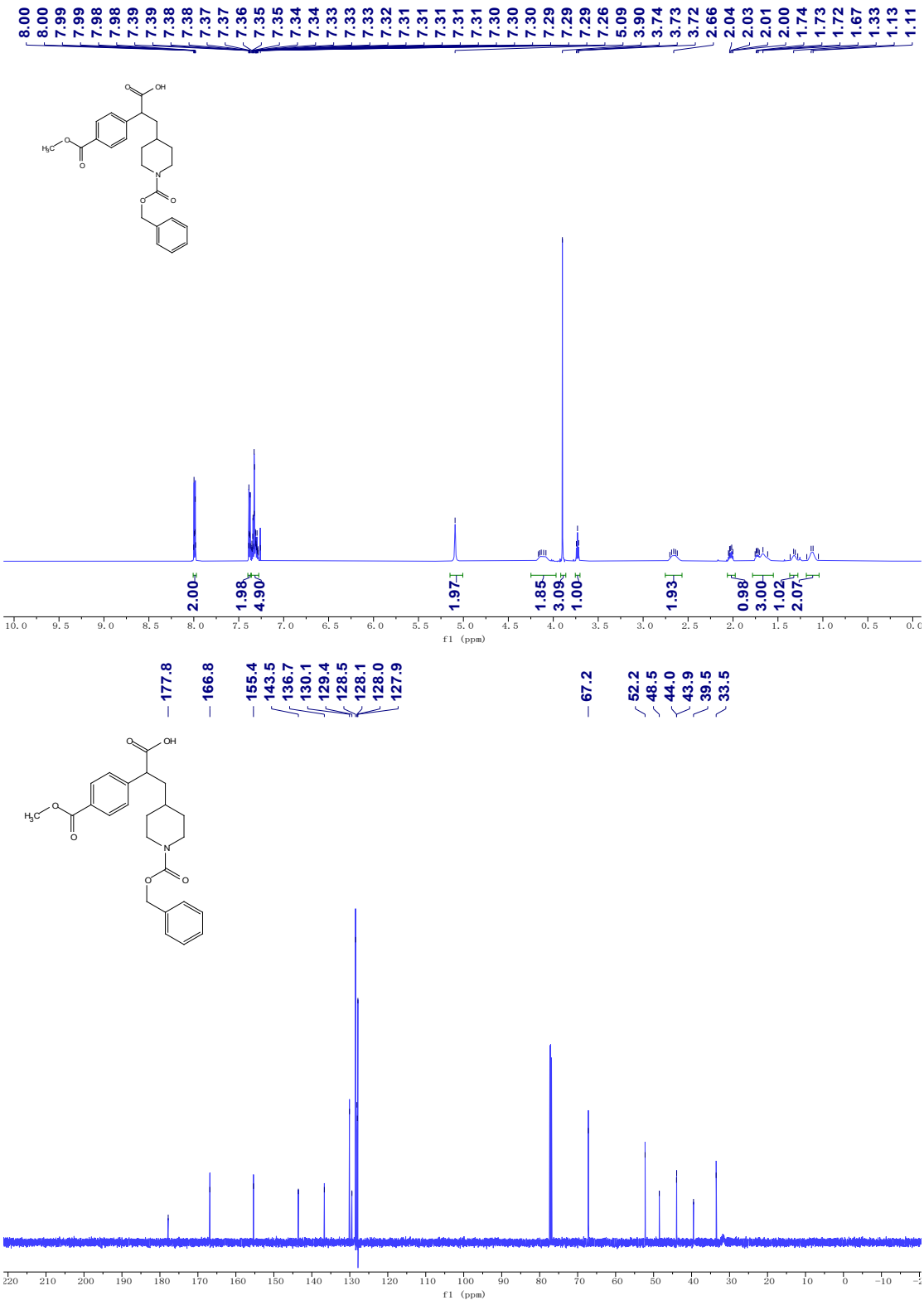
7. NMR Spectra

3a



3b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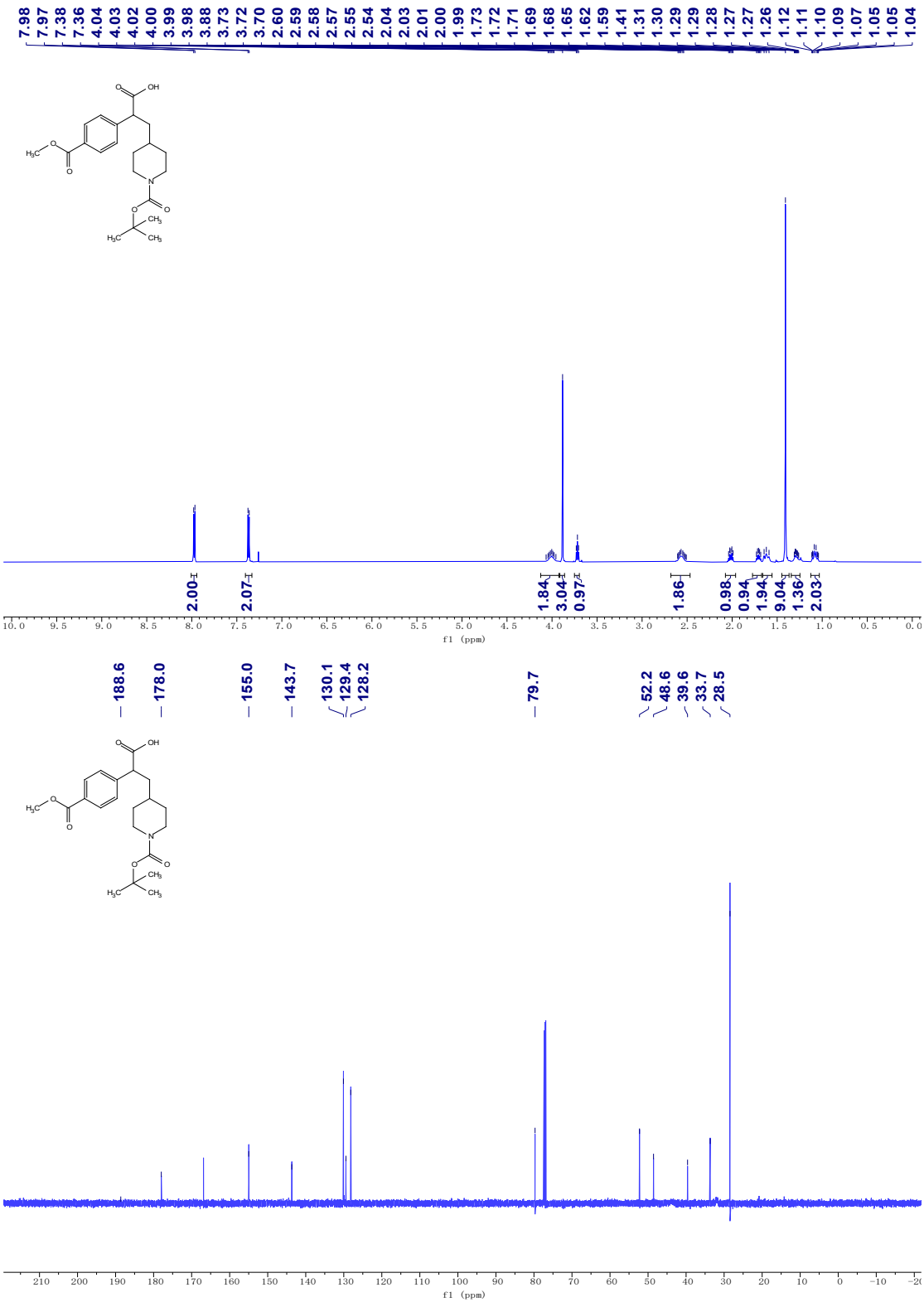
OH
O
N
2.00 1.98 4.90 1.97 1.85 3.09 1.00 1.93 0.98 3.00 1.02 2.07
f1 (ppm)
177.8 166.8 155.4 143.5 136.7 130.1 129.4 128.5 128.1 128.0 127.9 67.2 52.2 48.5 44.0 43.9 39.5 33.5
f1 (ppm)

3c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2.00 2.07 1.84 3.04 0.97 1.86 0.98 0.94 1.94 9.04 1.36 2.03
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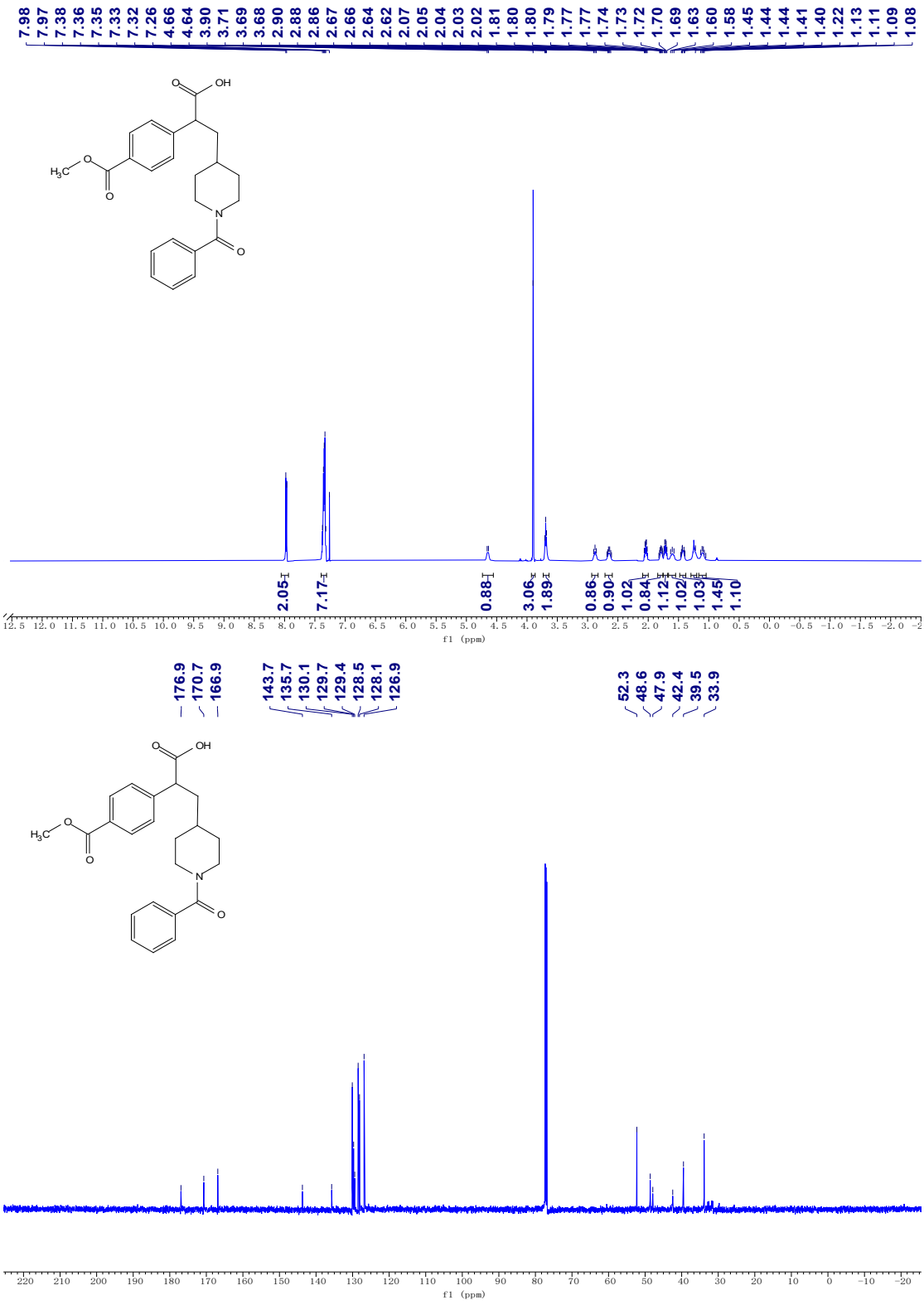
f1 (ppm)

3d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O
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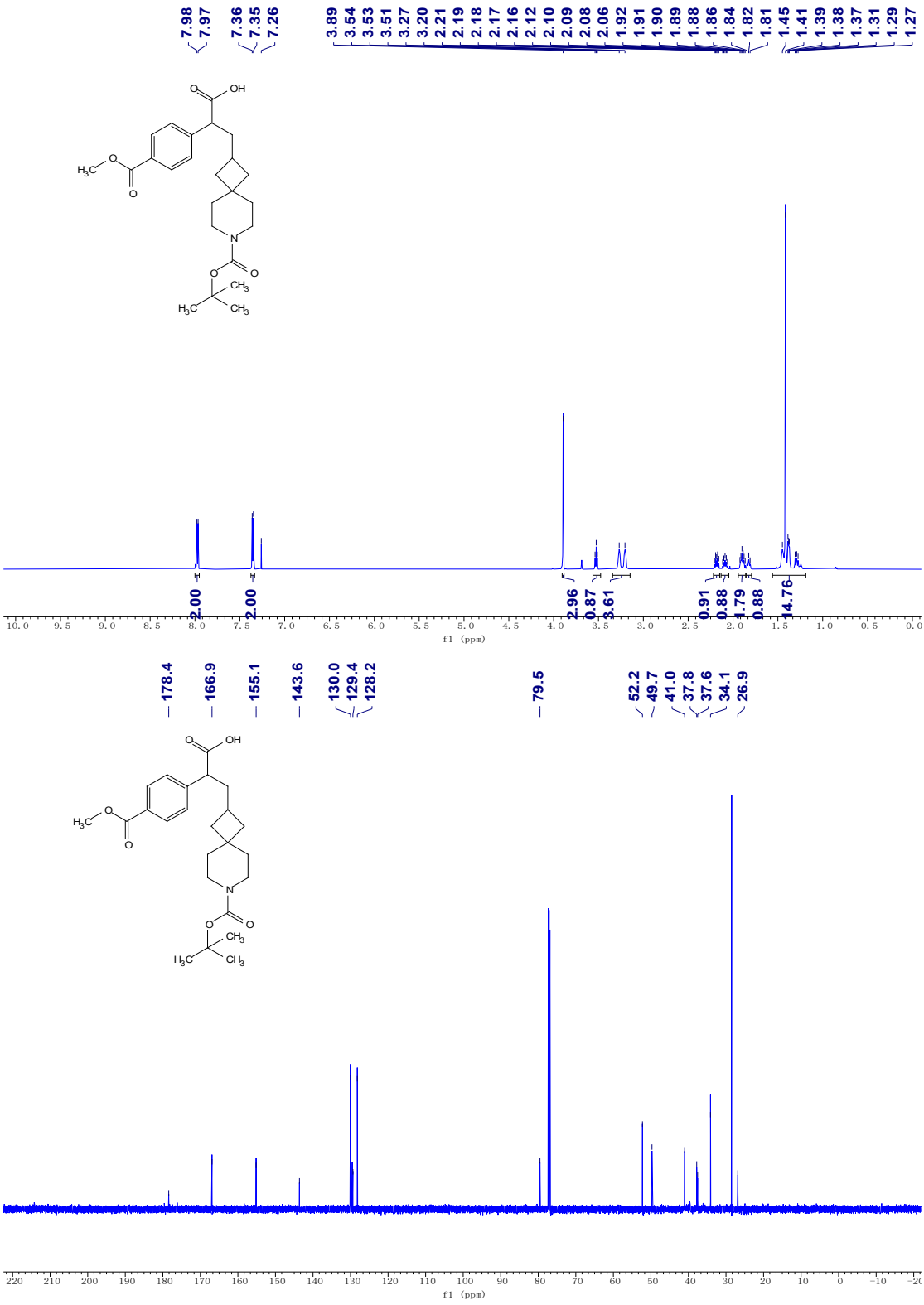
H3C
O
O
N
O
2.05 7.17 0.88 3.06 1.89 0.86 0.90 1.02 0.84 1.12 1.02 1.03 1.45 1.10
f1 (ppm)
176.9 170.7 166.9 143.7 135.7 130.1 129.7 129.4 128.5 128.1 126.9 52.3 48.6 47.9 42.4 39.5 33.9
f1 (ppm)

3e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2.18
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1.29
1.27
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O
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f1 (ppm)
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166.9
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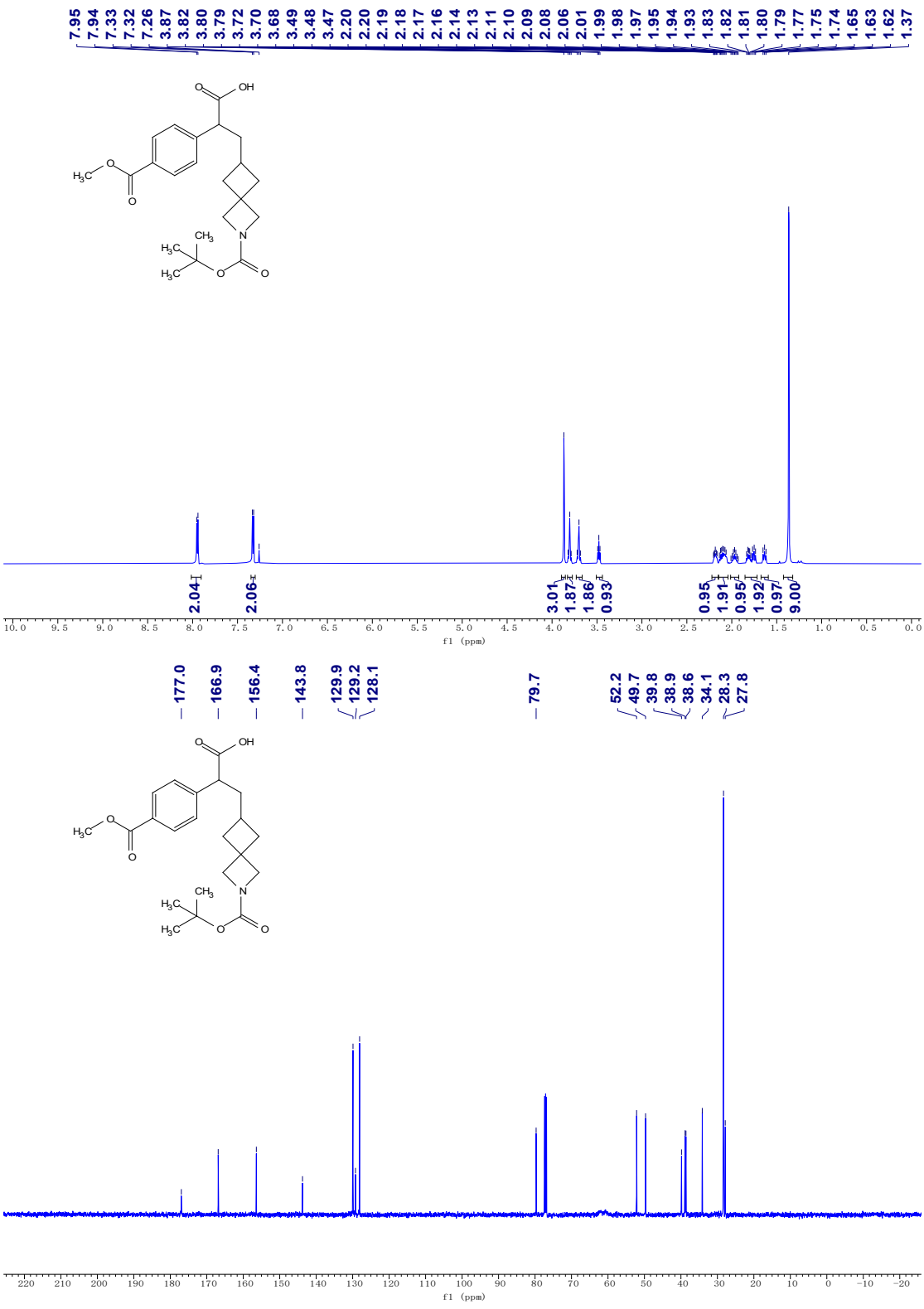
f1 (ppm)

3f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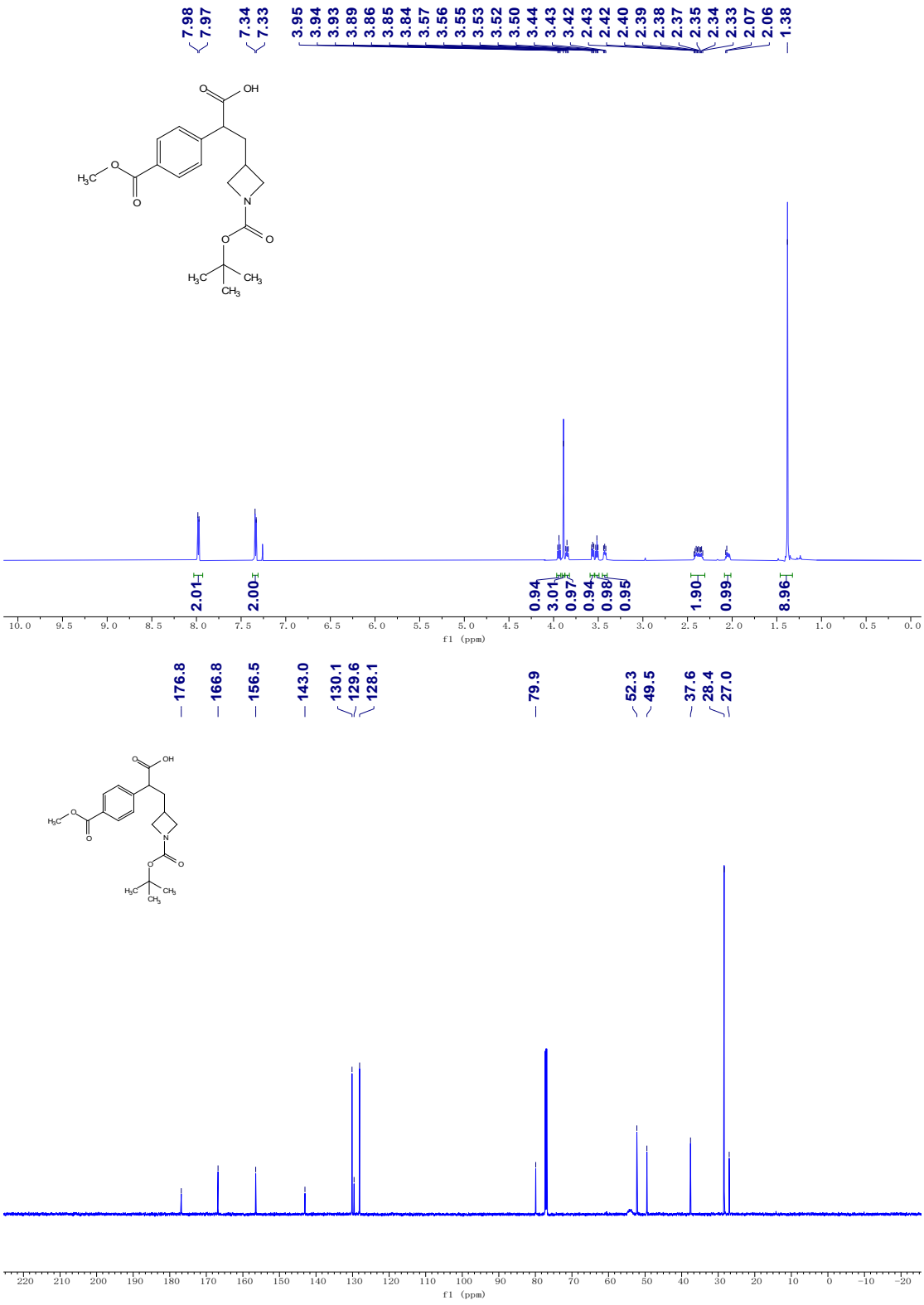
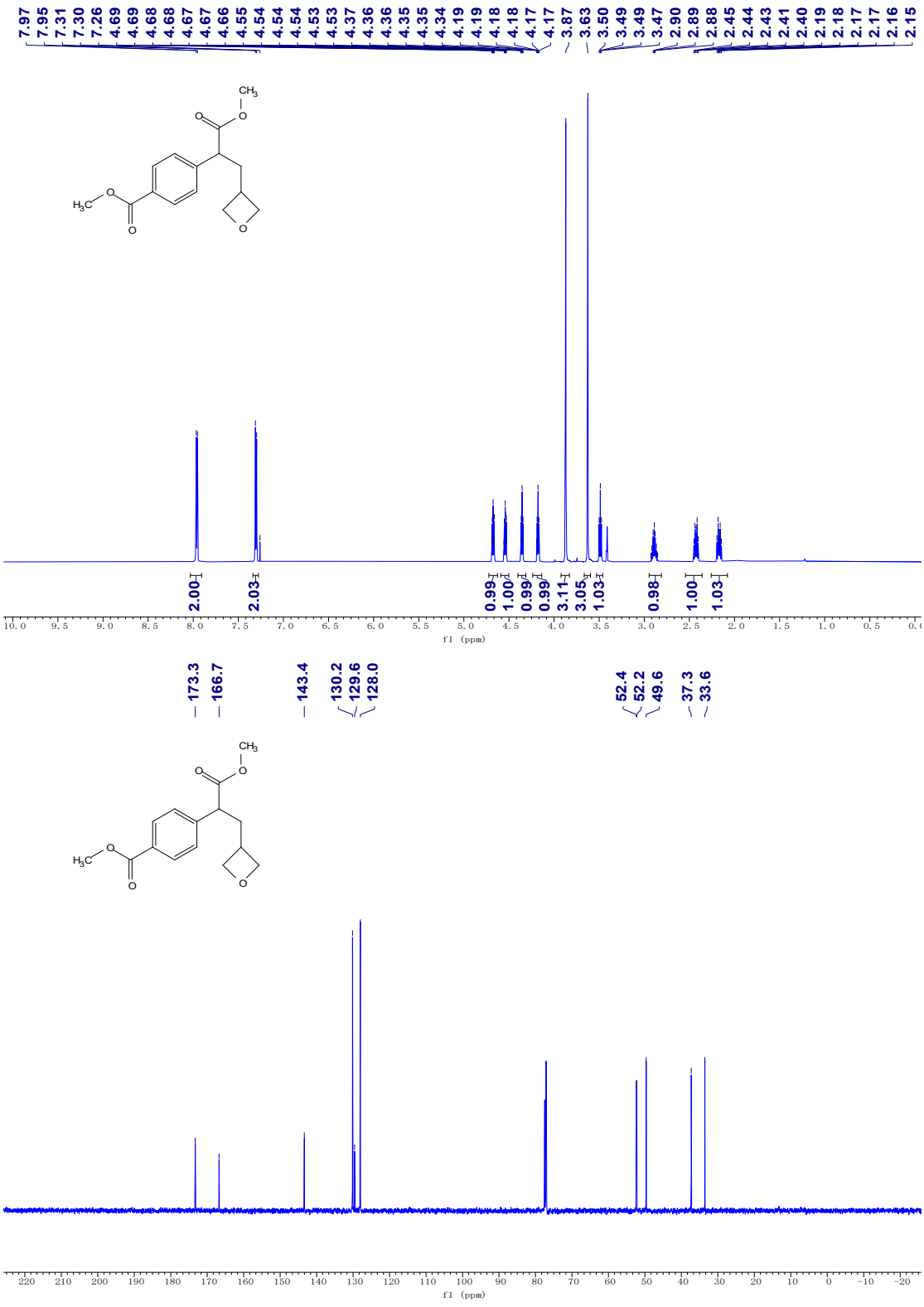
OH
O
H3C
CH3
N
2.04 2.06 3.01 1.87 1.86 0.93 0.95 1.91 0.95 1.92 0.97 9.00
f1 (ppm)
177.0 166.9 156.4 143.8 129.9 129.2 128.1 79.7 52.2 49.7 39.8 38.9 38.6 34.1 28.3 27.8
f1 (ppm)

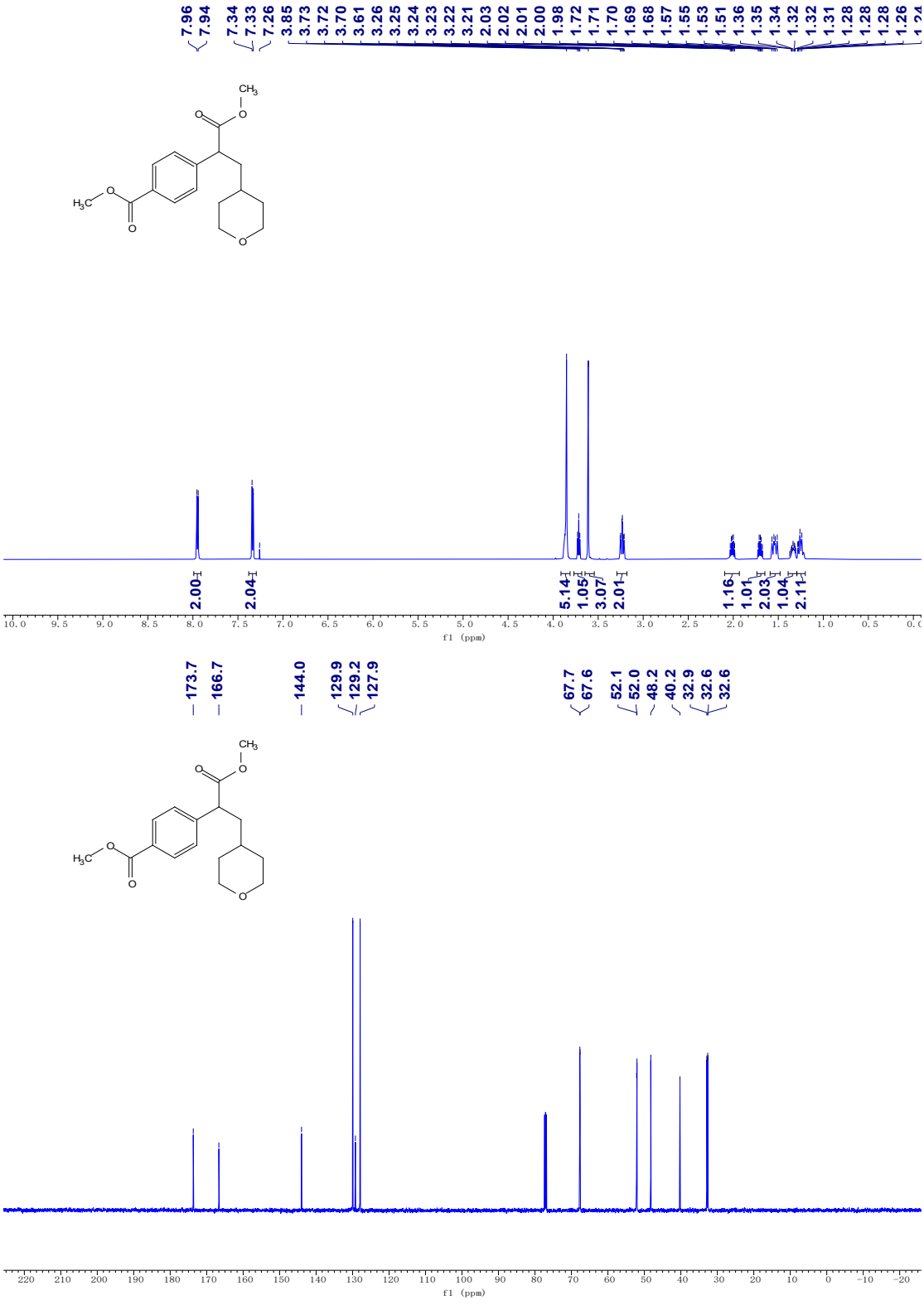
3g


7.98
7.97
7.34
7.33
3.95
3.94
3.93
3.89
3.86
3.85
3.84
3.57
3.56
3.55
3.53
3.52
3.50
3.44
3.43
3.42
2.43
2.42
2.40
2.39
2.38
2.37
2.35
2.34
2.33
2.07
2.06
1.38
OH
O
N
H3C
CH3
2.01
2.00
0.94
3.01
0.97
0.94
0.98
0.95
1.90
0.99
8.96
f1 (ppm)
176.8
166.8
156.5
143.0
130.1
129.6
128.1
79.9
52.3
49.5
37.6
28.4
27.0
f1 (ppm)

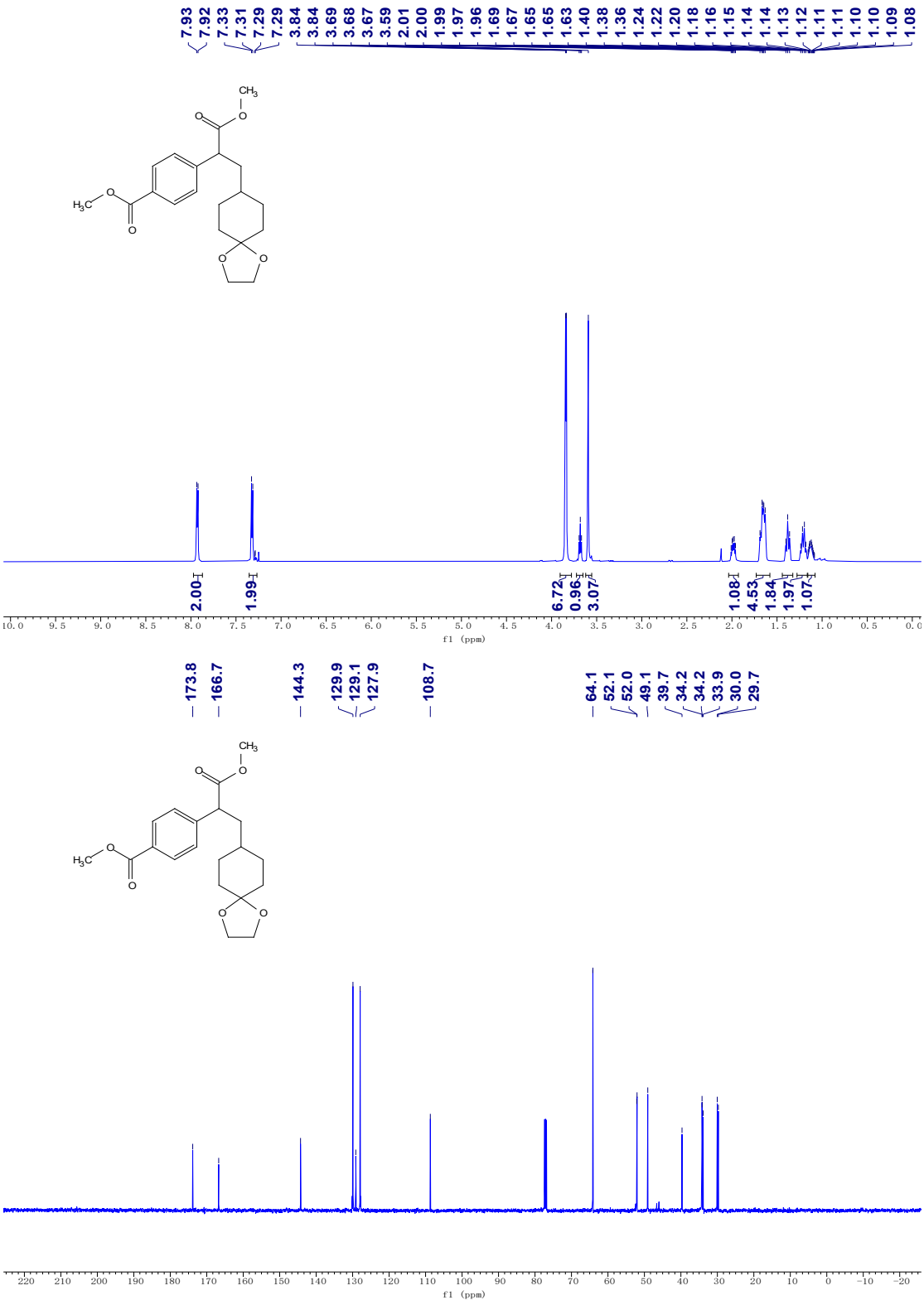
3h


7.97 7.95 7.31 7.30 7.26 4.69 4.69 4.68 4.68 4.67 4.67 4.66 4.55 4.54 4.54 4.54 4.53 4.53 4.37 4.36 4.36 4.35 4.35 4.34 4.19 4.19 4.18 4.18 4.17 4.17 3.87 3.63 3.50 3.49 3.49 3.47 2.90 2.89 2.88 2.45 2.44 2.43 2.41 2.40 2.19 2.18 2.17 2.17 2.16 2.15
2.00 2.03 0.99 1.00 0.99 0.99 3.11 3.05 1.03 0.98 1.00 1.03
f1 (ppm)
173.3 166.7 143.4 130.2 129.6 128.0 52.4 52.2 49.6 37.3 33.6
f1 (ppm)

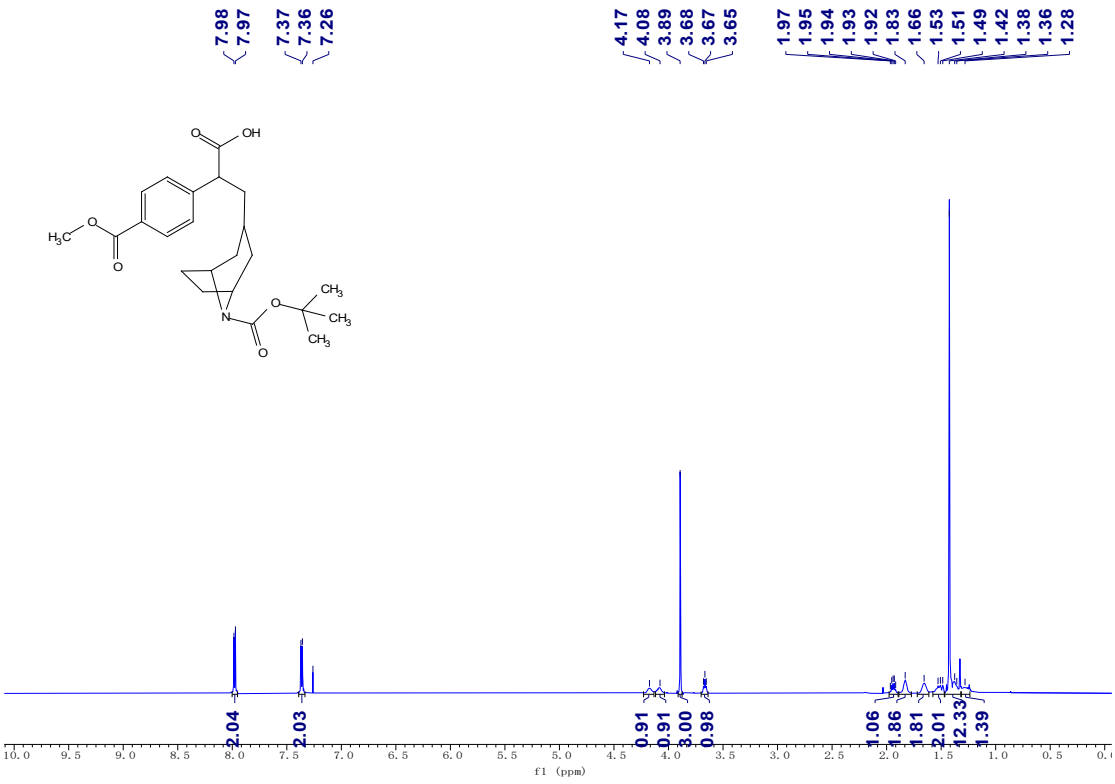
3i

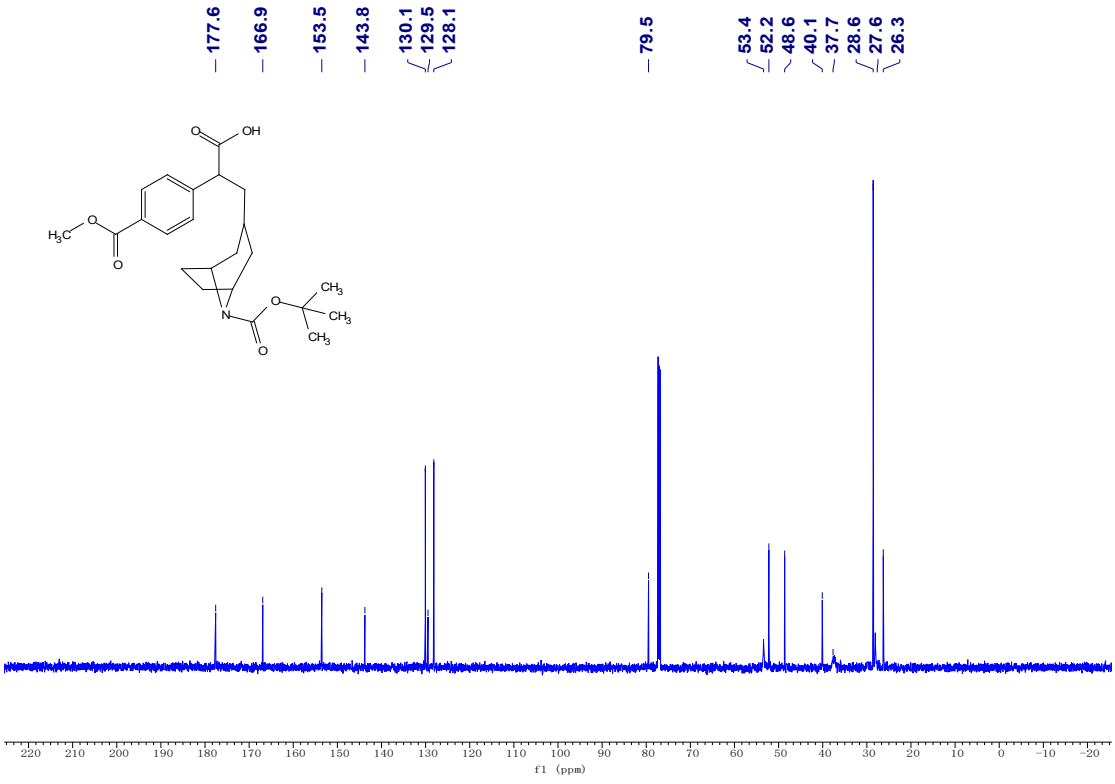

7.96
7.94
7.34
7.33
7.26
3.85
3.73
3.72
3.70
3.61
3.26
3.25
3.24
3.23
3.22
3.21
2.03
2.02
2.01
2.00
1.98
1.72
1.71
1.70
1.69
1.68
1.57
1.55
1.53
1.51
1.36
1.35
1.34
1.32
1.32
1.31
1.28
1.28
1.28
1.26
2.00
2.04
5.14
1.05
3.07
2.01
1.16
1.01
2.03
1.04
2.11
f1 (ppm)
173.7
166.7
144.0
129.9
129.2
127.9
67.7
67.6
52.1
52.0
48.2
40.2
32.9
32.6
32.6
f1 (ppm)

3j

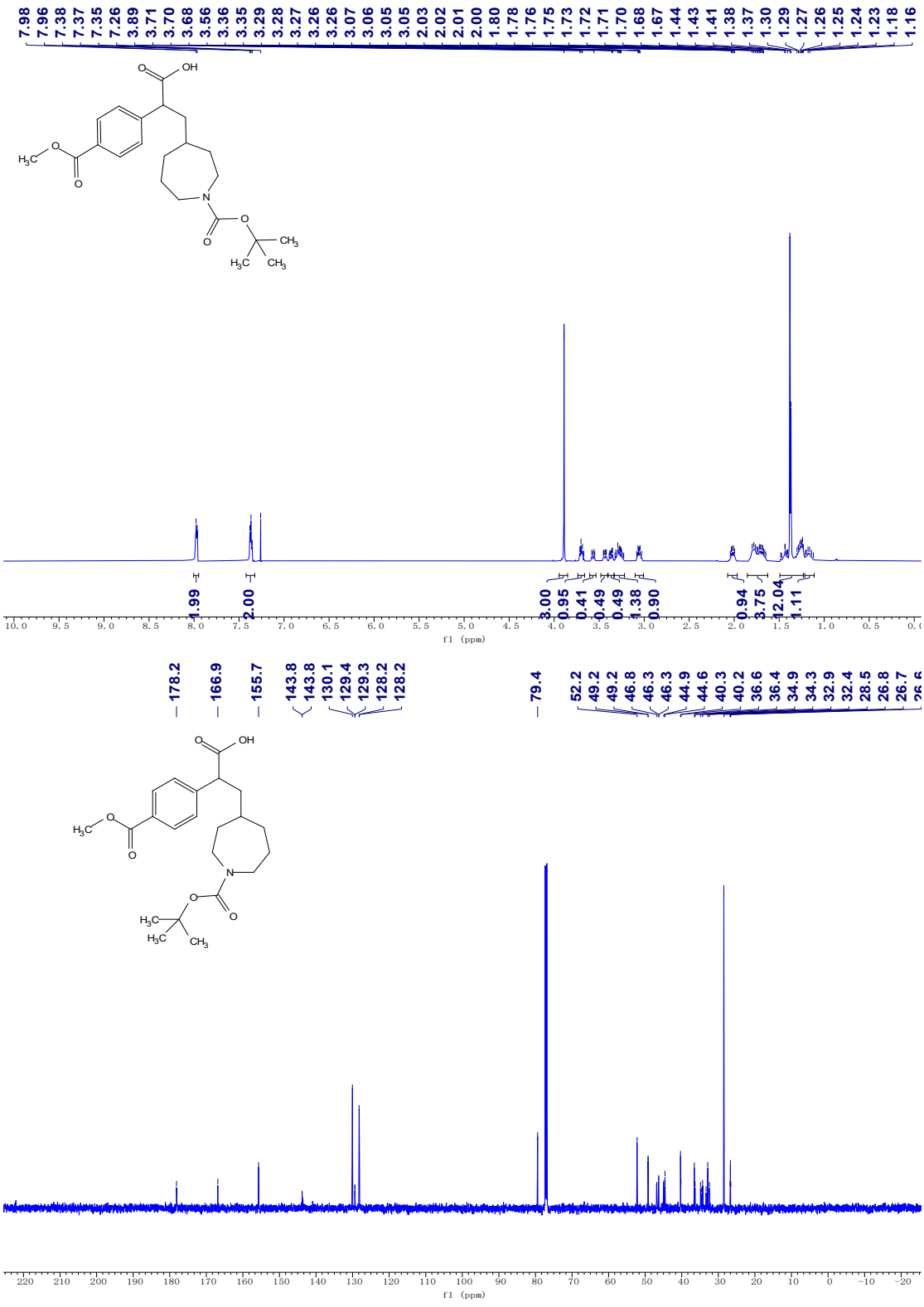

7.93 7.92 7.33 7.31 7.29 7.29 3.84 3.84 3.69 3.68 3.67 3.59 2.01 2.00 1.99 1.97 1.96 1.69 1.67 1.65 1.65 1.63 1.40 1.38 1.36 1.24 1.22 1.20 1.18 1.16 1.15 1.14 1.14 1.13 1.12 1.11 1.11 1.10 1.10 1.09 1.08
CH3
O
O
O
H3C
O
O
O
2.00 1.99 6.72 0.96 3.07 1.08 4.53 1.84 1.97 1.07
f1 (ppm)
173.8 166.7 144.3 129.9 129.1 127.9 108.7 64.1 52.1 52.0 49.1 39.7 34.2 34.2 33.9 30.0 29.7
CH3
O
O
O
H3C
O
O
O
f1 (ppm)

3k

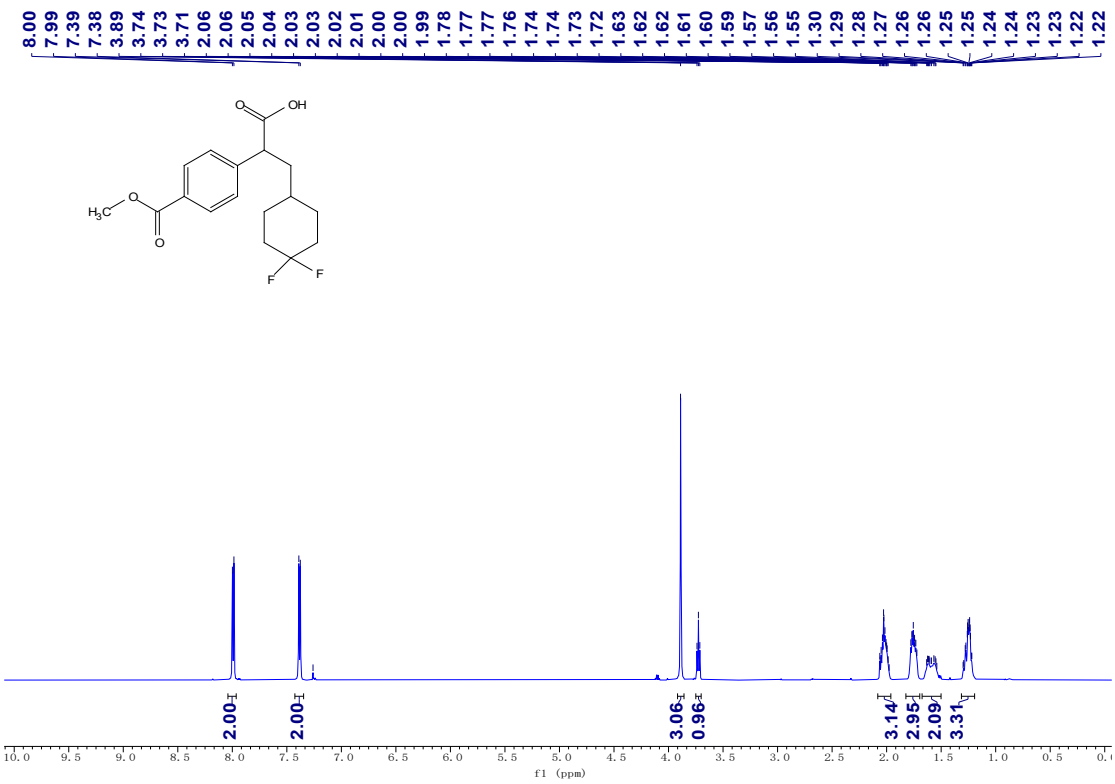

7.98
7.97
7.37
7.36
7.26
4.17
4.08
3.89
3.68
3.67
3.65
1.97
1.95
1.94
1.93
1.92
1.83
1.66
1.53
1.51
1.49
1.42
1.38
1.36
1.28
2.04
2.03
0.91
0.91
3.00
0.98
1.06
1.86
1.81
2.01
12.33
1.39
f1 (ppm)

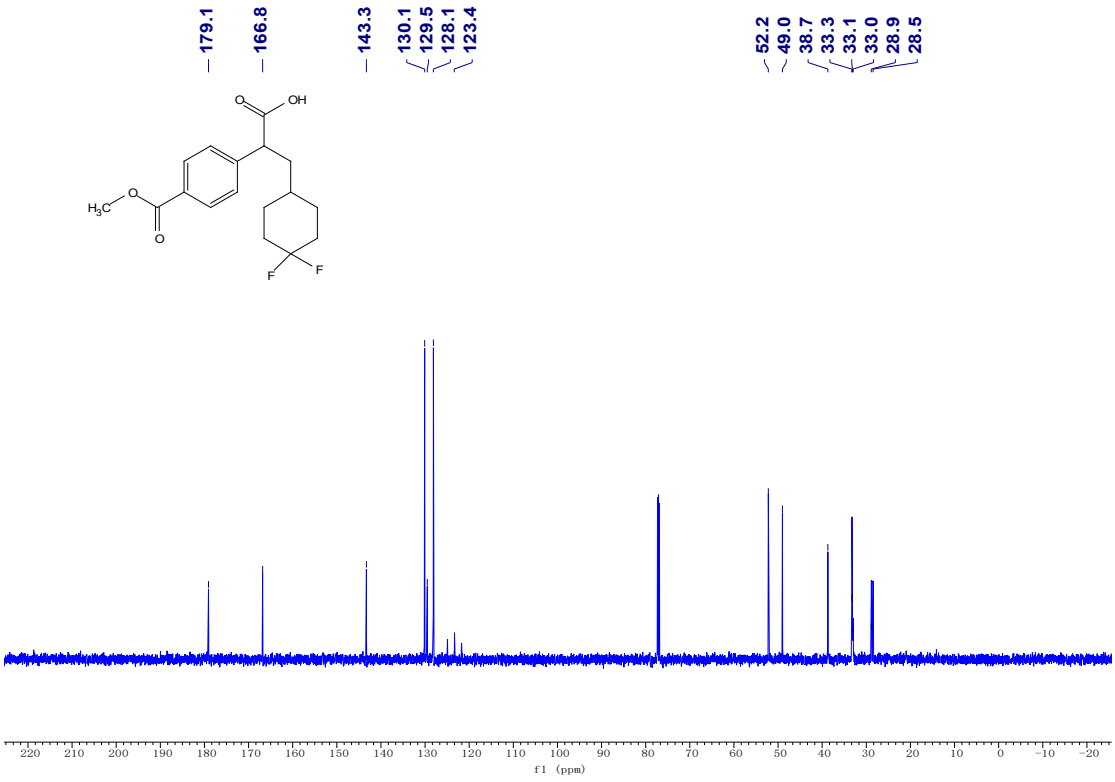

177.6
166.9
153.5
143.8
130.1
129.5
128.1
79.5
53.4
52.2
48.6
40.1
37.7
28.6
27.6
26.3
f1 (ppm)

3l


7.98 7.96 7.38 7.37 7.35 7.26 3.89 3.71 3.70 3.68 3.56 3.36 3.35 3.29 3.28 3.27 3.26 3.26 3.07 3.06 3.05 3.05 2.03 2.02 2.01 2.00 1.80 1.78 1.76 1.75 1.73 1.72 1.71 1.70 1.68 1.67 1.44 1.43 1.41 1.38 1.37 1.30 1.29 1.27 1.26 1.25 1.24 1.23 1.18 1.16
1.99 2.00 3.00 0.95 0.41 0.49 0.49 1.38 0.90 0.94 3.75 12.04 1.11
f1 (ppm)
178.2 166.9 155.7 143.8 143.8 130.1 129.4 129.3 128.2 128.2 79.4 52.2 49.2 49.2 46.8 46.3 46.3 44.9 44.6 40.3 40.2 36.6 36.4 34.9 34.3 32.9 32.4 28.5 26.8 26.7
f1 (ppm)

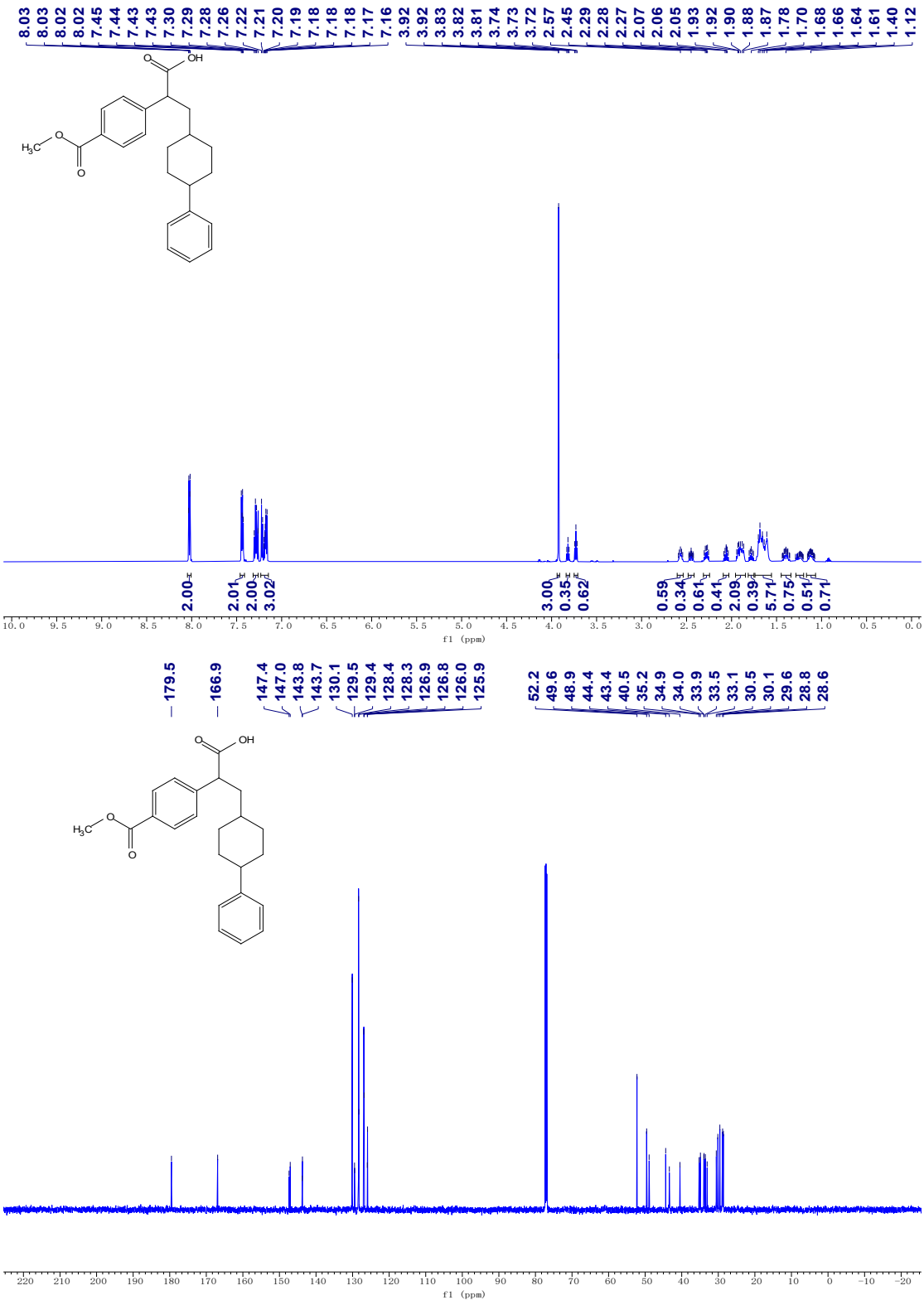
3m


8.00 7.99 7.39 7.38 3.89 3.74 3.73 3.71 2.06 2.06 2.05 2.04 2.03 2.03 2.02 2.01 2.00 2.00 1.99 1.78 1.77 1.77 1.76 1.74 1.74 1.73 1.72 1.63 1.62 1.62 1.61 1.60 1.59 1.57 1.56 1.55 1.30 1.29 1.28 1.27 1.26 1.26 1.25 1.25 1.24 1.24 1.23 1.23 1.22 1.22
O
OH
H3C
O
O
F
F
2.00 2.00 3.06 0.96 3.14 2.95 2.09 3.31
f1 (ppm)

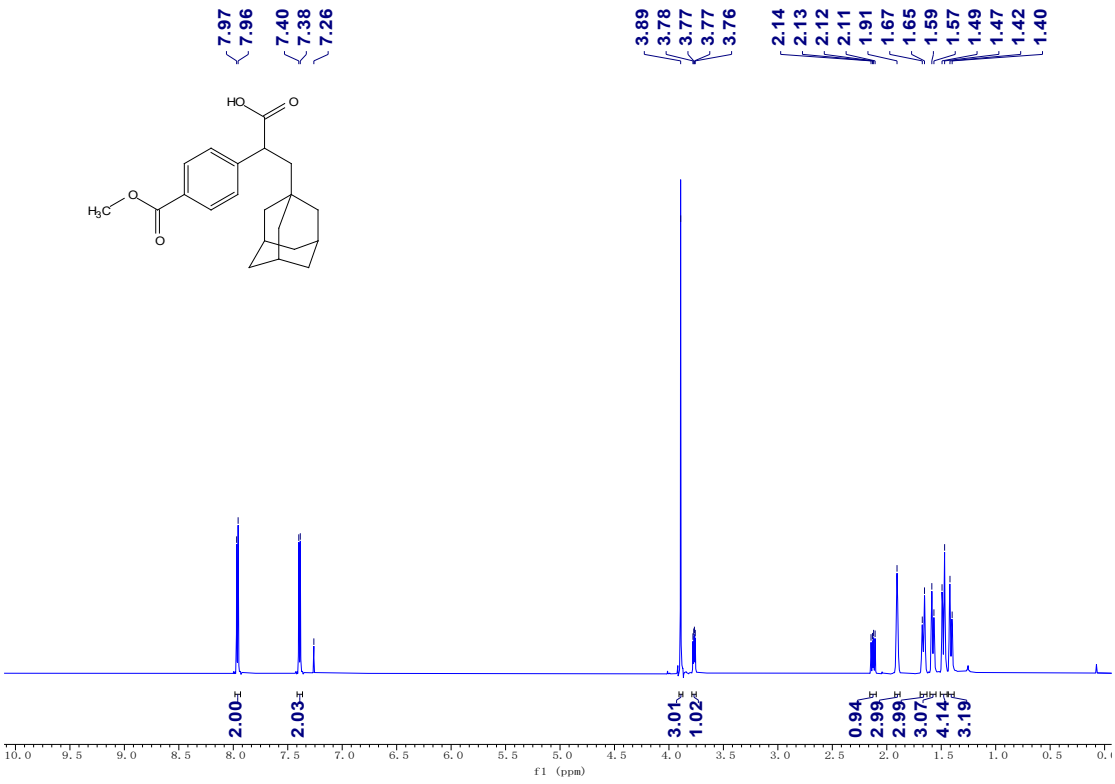

179.1 166.8 143.3 130.1 129.5 128.1 123.4 52.2 49.0 38.7 33.3 33.1 33.0 28.9 28.5
O
OH
H3C
O
O
F
F
f1 (ppm)

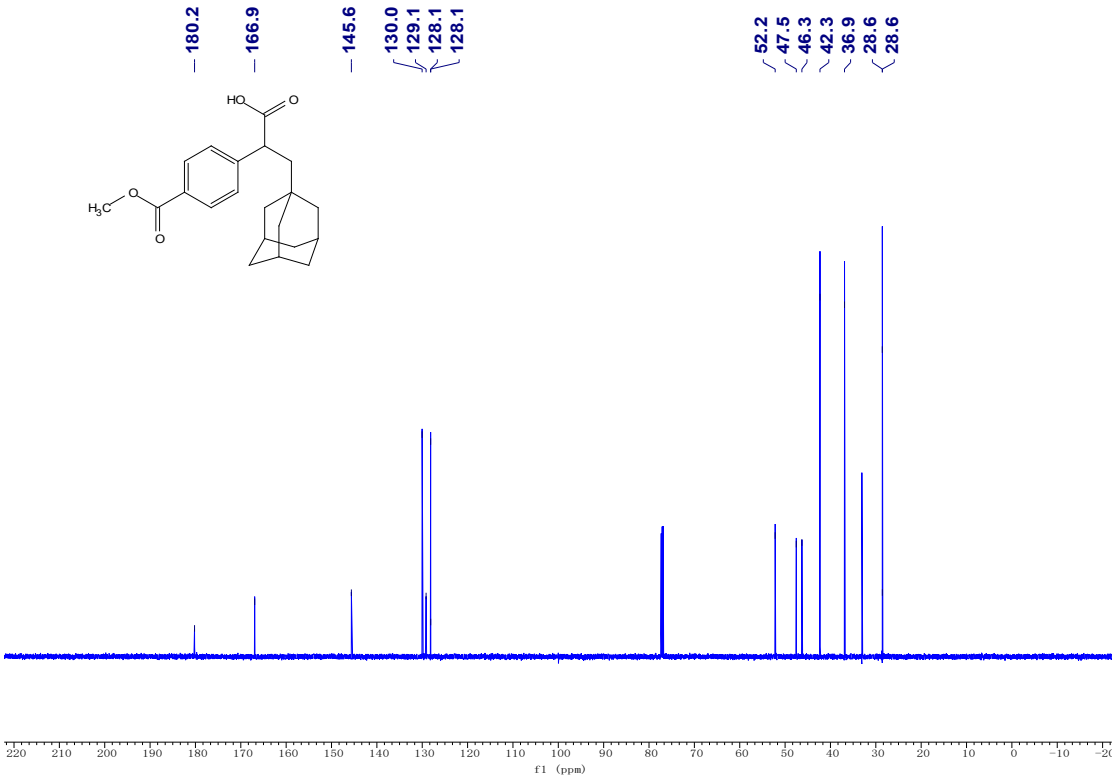

-91.7
-102.0
O
OH
H3C
O
O
F
F
40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240
f1 (ppm)

3n

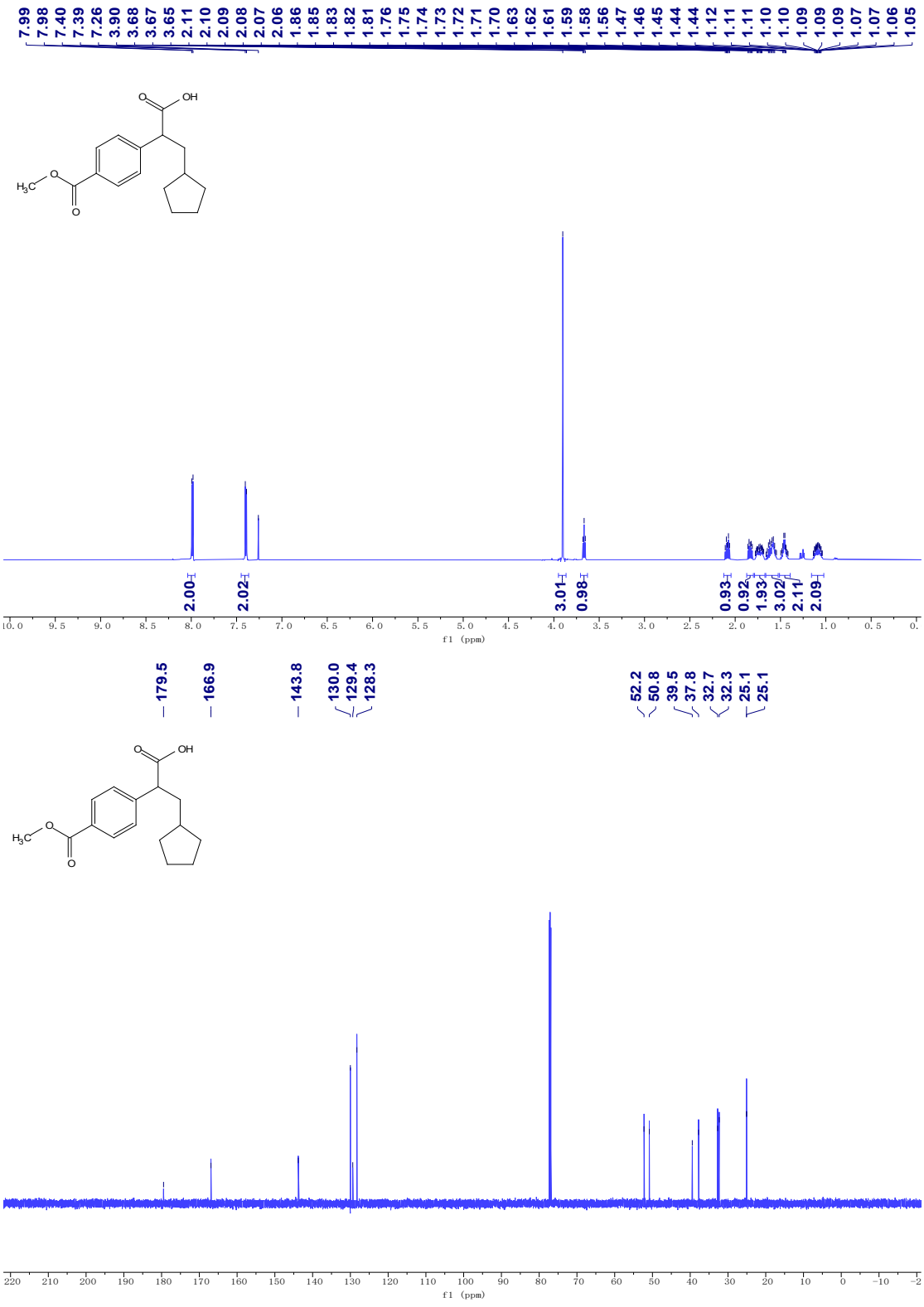

8.03 8.03 8.02 8.02 7.45 7.44 7.43 7.43 7.30 7.29 7.28 7.26 7.22 7.21 7.20 7.19 7.18 7.18 7.17 7.16 3.92 3.92 3.83 3.82 3.81 3.74 3.73 3.72 2.57 2.45 2.29 2.28 2.27 2.07 2.06 2.05 1.93 1.92 1.90 1.88 1.87 1.78 1.70 1.68 1.66 1.64 1.61 1.40 1.12
OH
O
H3C
2.00 2.01 2.00 3.02 3.00 0.35 0.62 0.59 0.34 0.61 0.41 2.09 0.39 5.71 0.75 0.51 0.71
f1 (ppm)
179.5 166.9 147.4 147.0 143.8 143.7 130.1 129.5 129.4 128.4 128.3 126.9 126.8 126.0 125.9 52.2 49.6 48.9 44.4 43.4 40.5 35.2 34.9 34.0 33.9 33.5 33.1 30.5 30.1 29.6 28.8 28.6
f1 (ppm)

3o

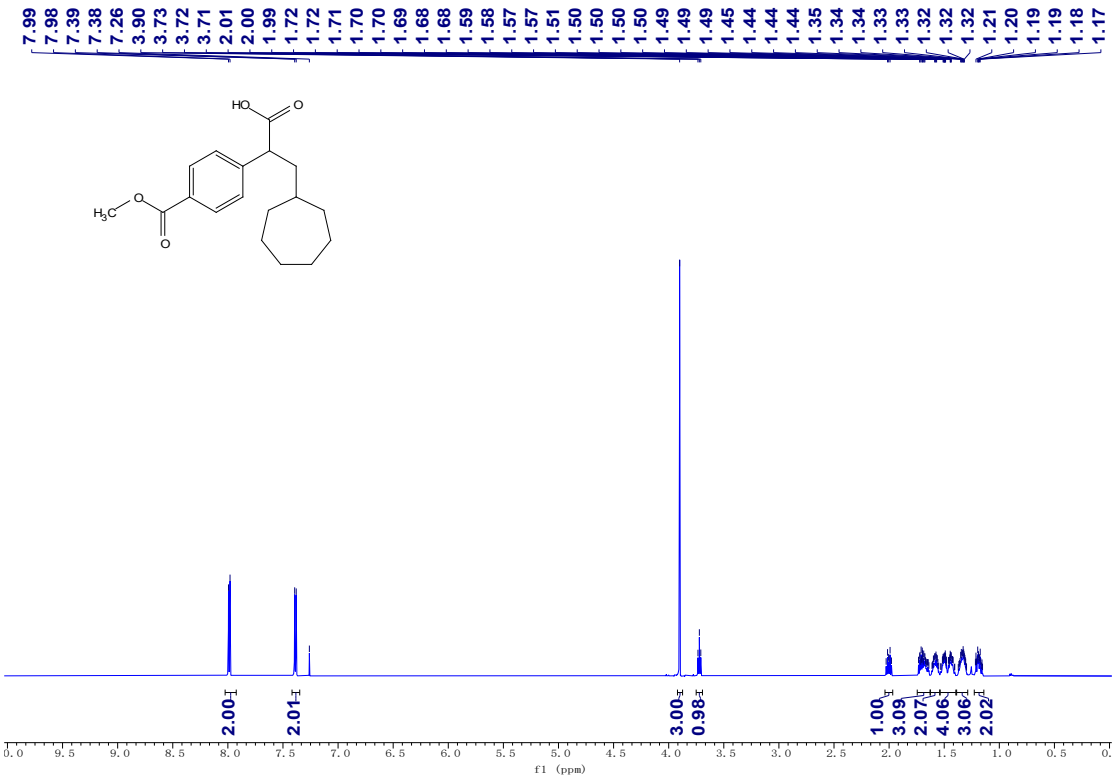

7.97
7.96
7.40
7.38
7.26
3.89
3.78
3.77
3.77
3.76
2.14
2.13
2.12
2.11
1.91
1.67
1.65
1.59
1.57
1.49
1.47
1.42
1.40
HO
O
O
H3C
O
2.00
2.03
3.01
1.02
0.94
2.99
2.99
3.07
4.14
3.19
f1 (ppm)

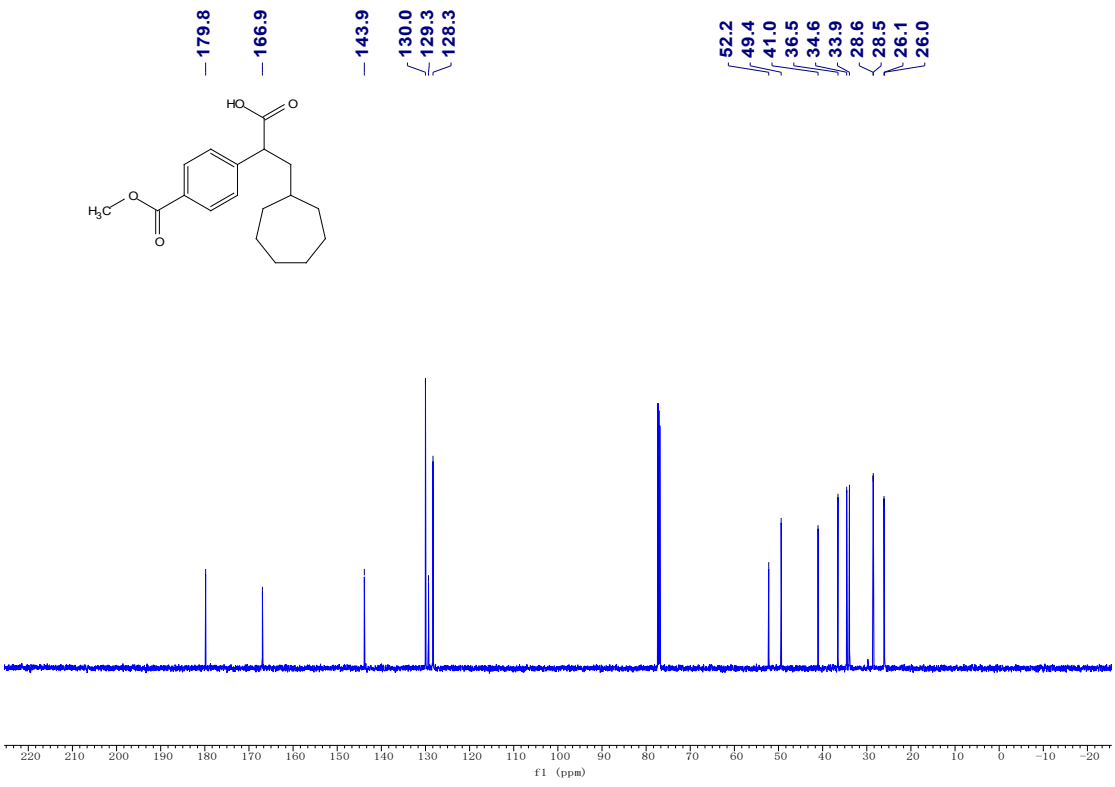

180.2
166.9
145.6
130.0
129.1
128.1
128.1
52.2
47.5
46.3
42.3
36.9
28.6
28.6
HO
O
O
H3C
O
f1 (ppm)

3p

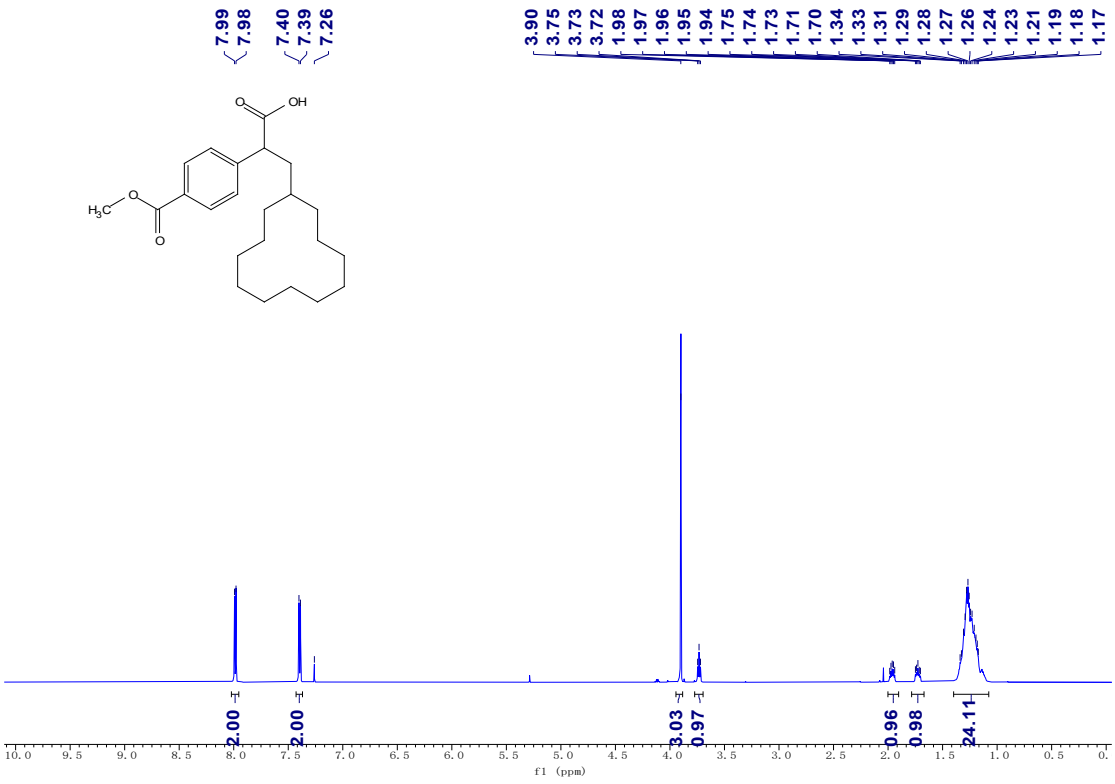

7.99 7.98 7.40 7.39 7.26 3.90 3.68 3.67 3.65 2.11 2.10 2.09 2.08 2.07 2.06 1.86 1.85 1.83 1.82 1.81 1.76 1.75 1.74 1.73 1.72 1.71 1.70 1.63 1.62 1.61 1.59 1.58 1.56 1.47 1.46 1.45 1.44 1.44 1.12 1.11 1.11 1.10 1.10 1.09 1.09 1.09 1.07 1.07 1.06 1.05
O
OH
H3C
O
O
2.00 2.02 3.01 0.98 0.93 0.92 1.93 3.02 2.11 2.09
f1 (ppm)
179.5 166.9 143.8 130.0 129.4 128.3 52.2 50.8 39.5 37.8 32.7 32.3 25.1 25.1
f1 (ppm)

3q


7.99 7.98 7.39 7.38 7.26 3.90 3.73 3.72 3.71 2.01 2.00 1.99 1.72 1.72 1.71 1.70 1.70 1.69 1.68 1.68 1.59 1.58 1.57 1.57 1.51 1.50 1.50 1.50 1.50 1.49 1.49 1.49 1.45 1.44 1.44 1.44 1.35 1.34 1.34 1.33 1.33 1.32 1.32 1.32 1.21 1.20 1.19 1.19 1.18 1.17
HO
O
H3C
O
O
2.00 2.01 3.00 0.98 1.00 3.09 2.07 4.06 3.06 2.02
f1 (ppm)


179.8 166.9 143.9 130.0 129.3 128.3
52.2 49.4 41.0 36.5 34.6 33.9 28.6 28.5 26.1 26.0
HO
O
H3C
O
O
f1 (ppm)

3r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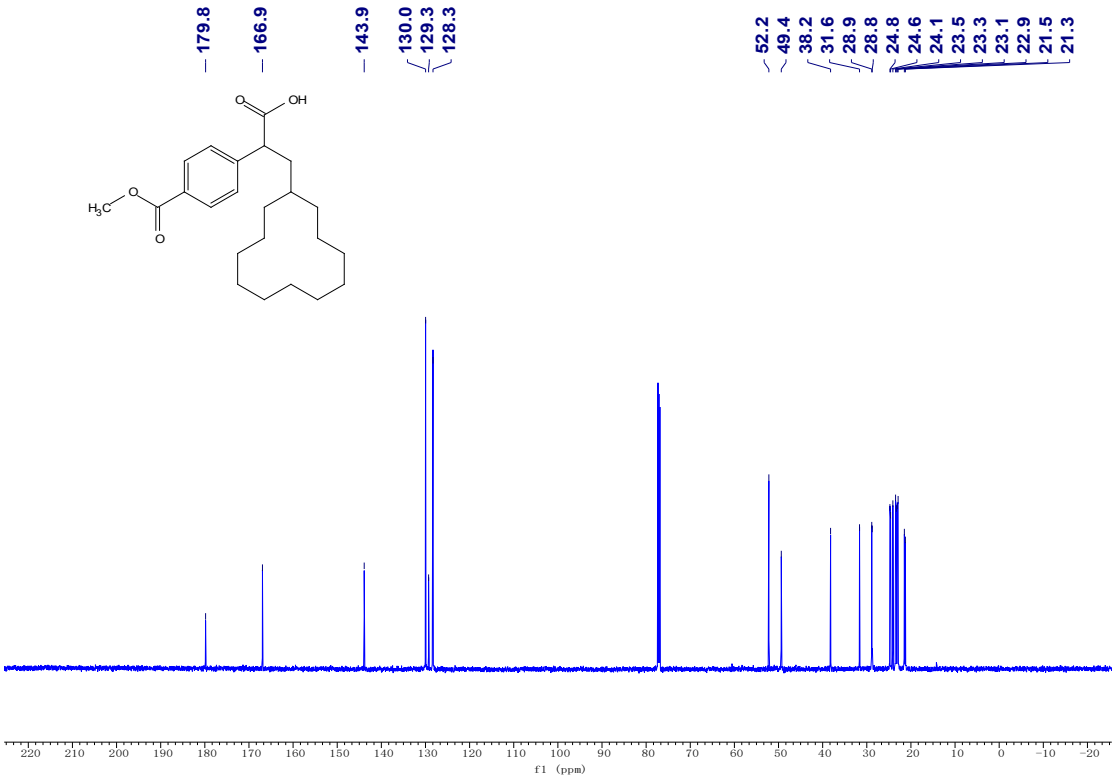
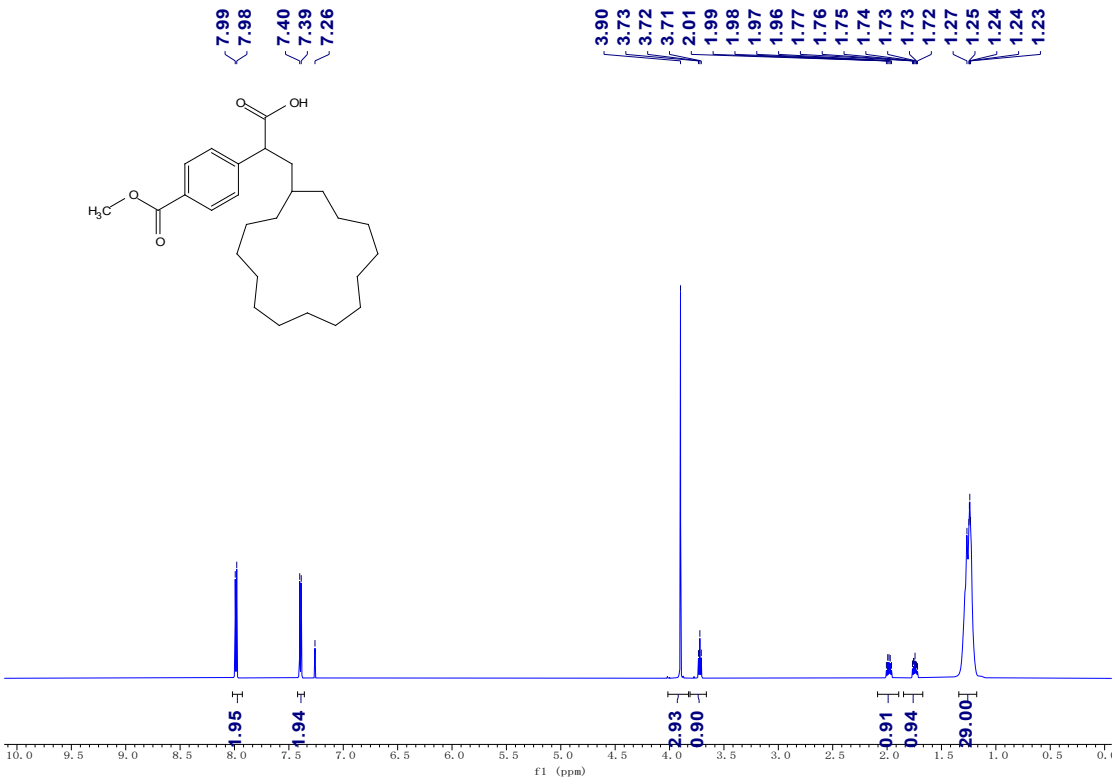
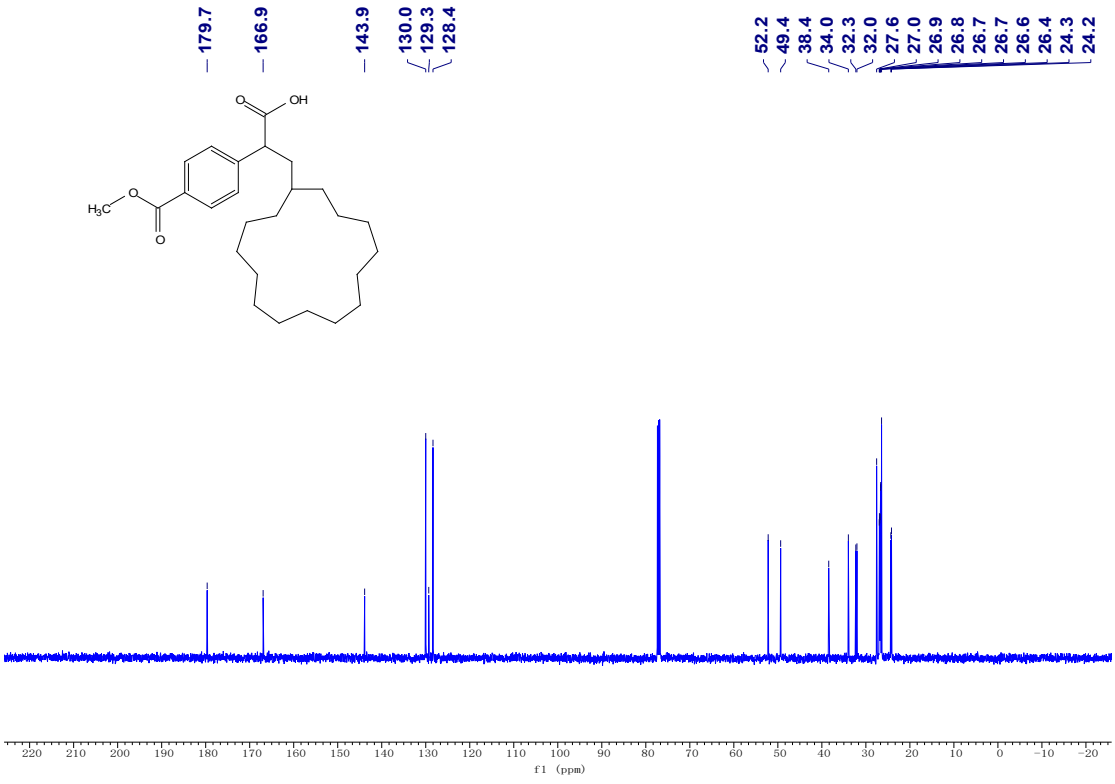
7.98
7.40
7.39
7.26
3.90
3.75
3.73
3.72
1.98
1.97
1.96
1.95
1.94
1.75
1.74
1.73
1.71
1.70
1.34
1.33
1.31
1.29
1.28
1.27
1.26
1.24
1.23
1.21
1.19
1.18
1.17
O
OH
H3C
O
O
2.00
2.00
3.03
0.97
0.96
0.98
24.11
f1 (ppm)

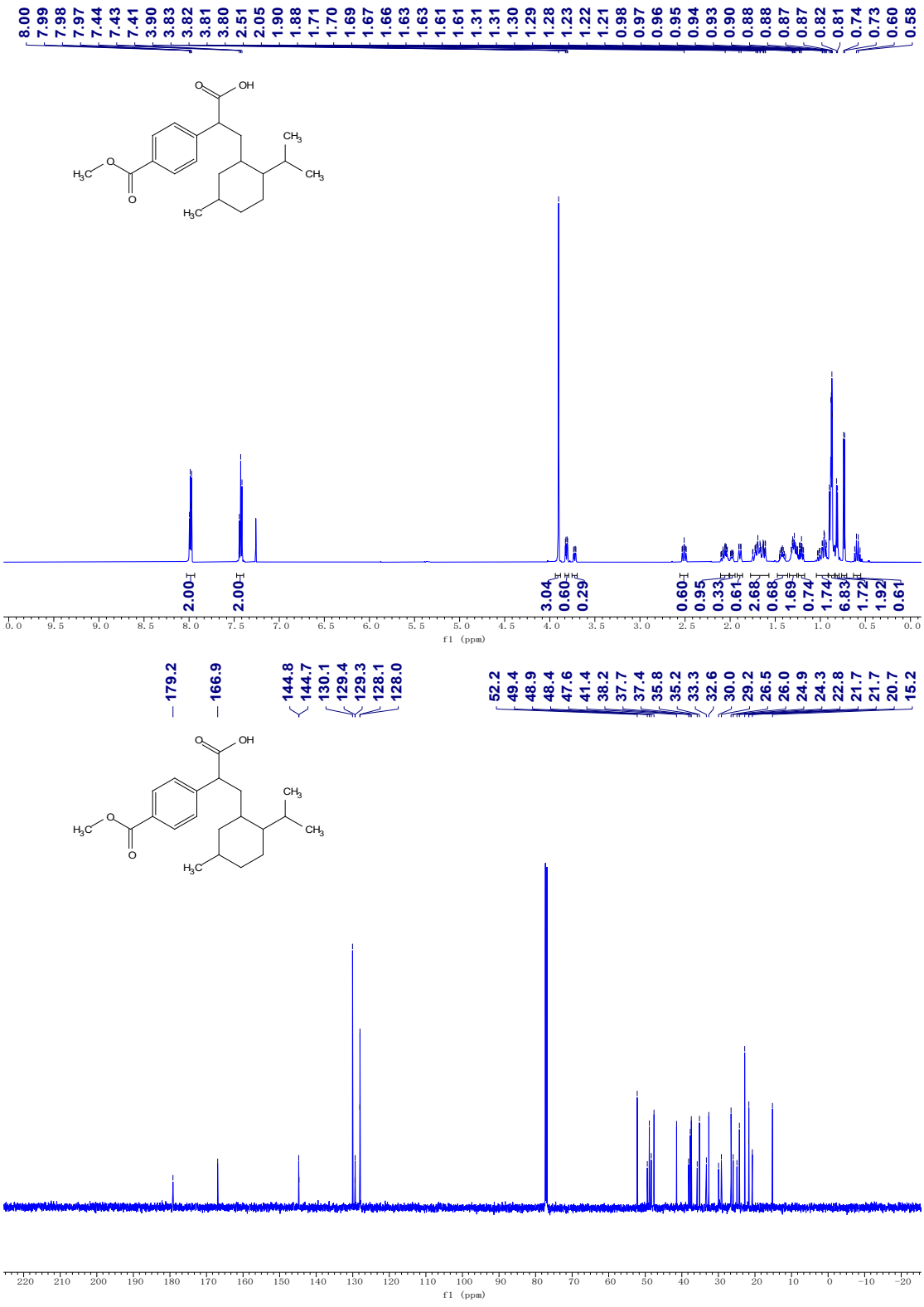

179.8
166.9
143.9
130.0
129.3
128.3
52.2
49.4
38.2
31.6
28.9
28.8
24.8
24.6
24.1
23.5
23.3
23.1
22.9
21.5
21.3
O
OH
H3C
O
O
f1 (ppm)

3s

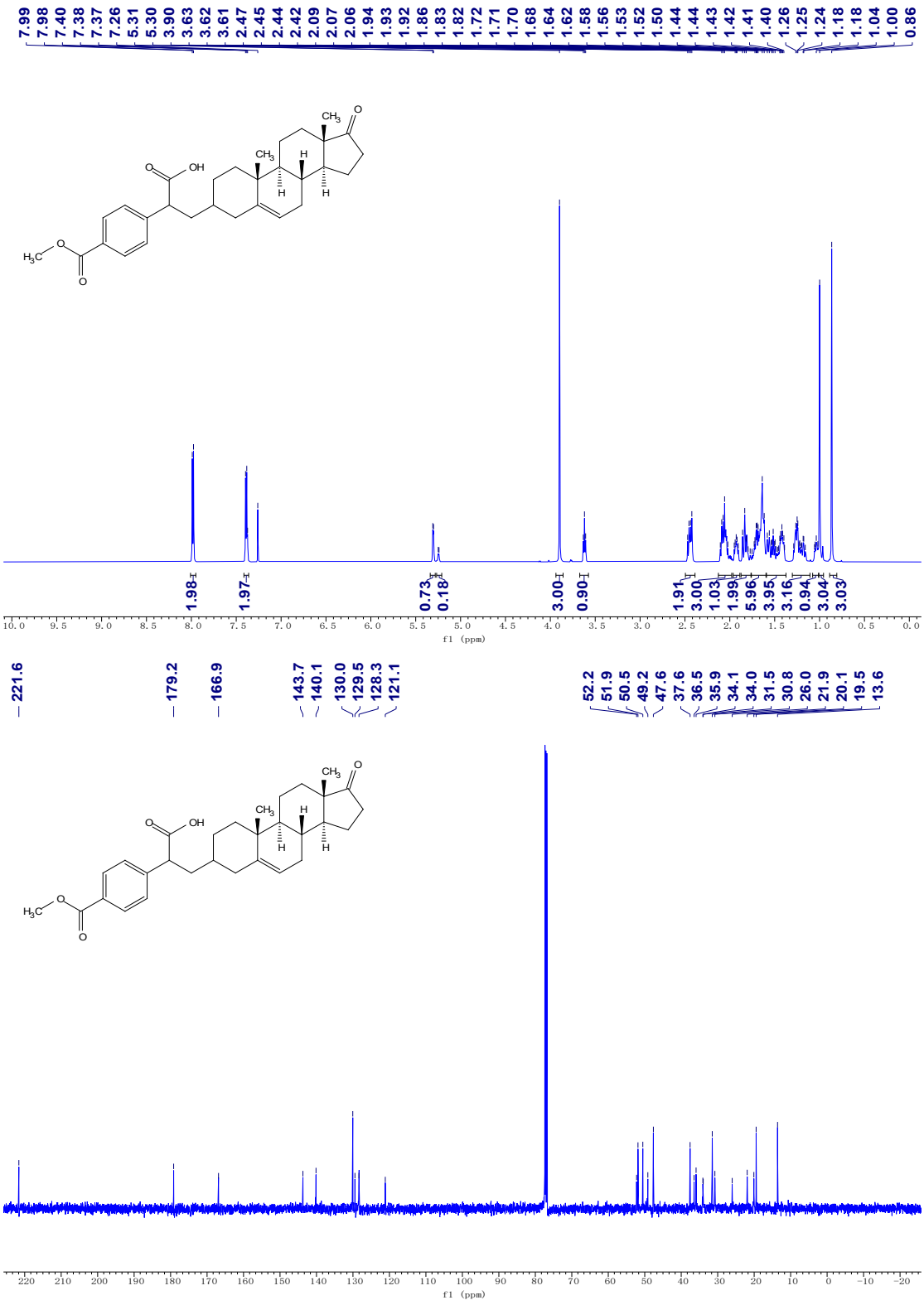

7.99
7.98
7.40
7.39
7.26
3.90
3.73
3.72
3.71
2.01
1.99
1.98
1.97
1.96
1.77
1.76
1.75
1.74
1.73
1.73
1.72
1.27
1.25
1.24
1.24
1.23
O
OH
H3C
O
O
1.95
1.94
2.93
0.90
0.91
0.94
29.00
f1 (ppm)


179.7
166.9
143.9
130.0
129.3
128.4
52.2
49.4
38.4
34.0
32.3
32.0
27.6
27.0
26.9
26.8
26.7
26.7
26.6
26.4
24.3
24.2
O
OH
H3C
O
O
f1 (ppm)

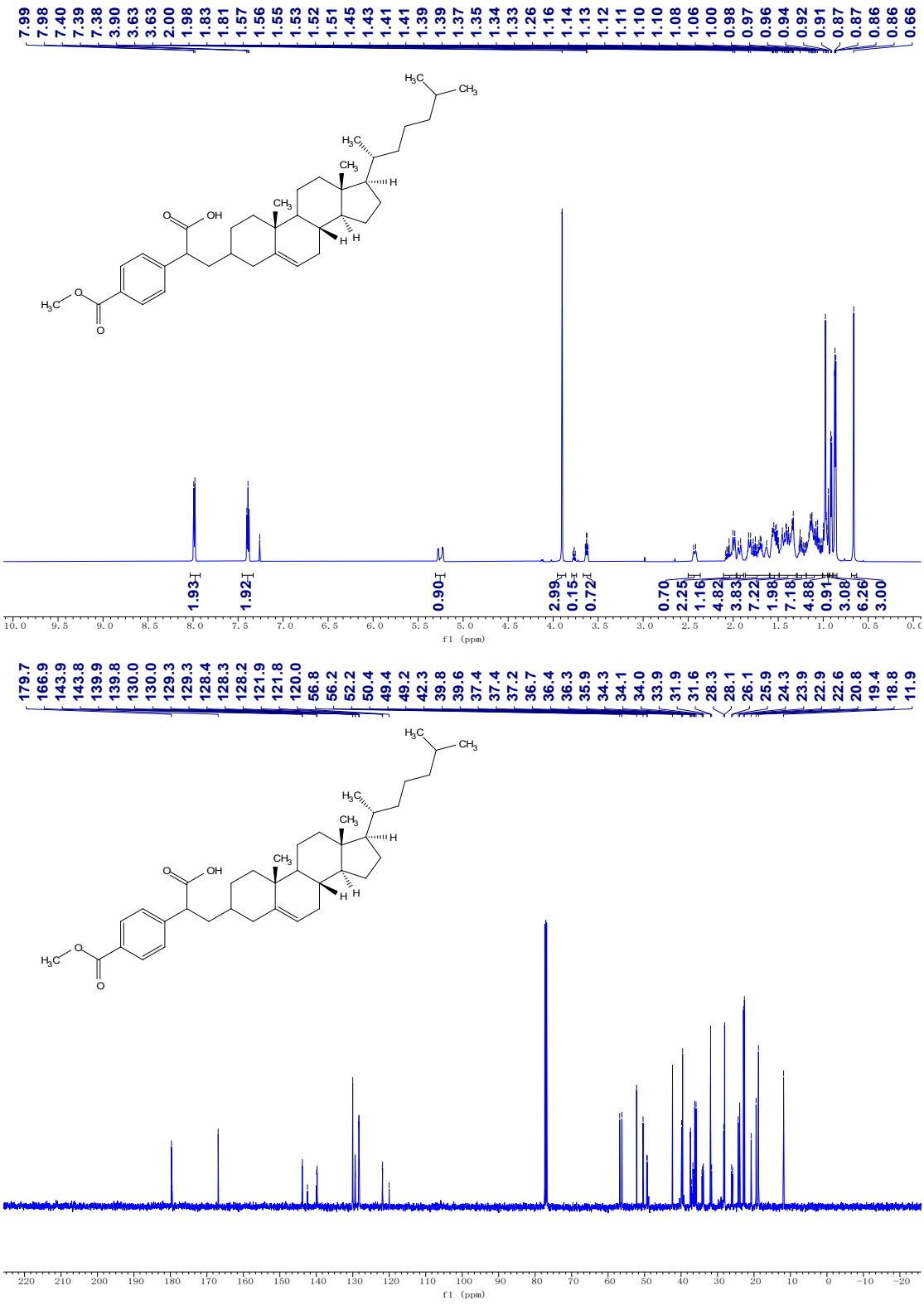
3t


8.00 7.99 7.98 7.97 7.44 7.43 7.41 3.90 3.83 3.82 3.81 3.80 2.51 2.05 1.90 1.88 1.71 1.70 1.69 1.67 1.66 1.63 1.63 1.61 1.61 1.31 1.31 1.30 1.29 1.28 1.23 1.22 1.21 0.98 0.97 0.96 0.95 0.94 0.93 0.90 0.88 0.88 0.87 0.87 0.82 0.81 0.74 0.73 0.60 0.58
2.00 2.00 3.04 0.60 0.29 0.60 0.95 0.33 0.61 2.68 0.68 1.69 0.74 1.74 6.83 1.72 1.92 0.61
f1 (ppm)
179.2 166.9 144.8 144.7 130.1 129.4 129.3 128.1 128.0
52.2 49.4 48.9 48.4 47.6 41.4 38.2 37.7 37.4 35.8 35.2 33.3 32.6 30.0 29.2 26.5 26.0 24.9 24.3 22.8 21.7 21.7 20.7 15.2
f1 (ppm)

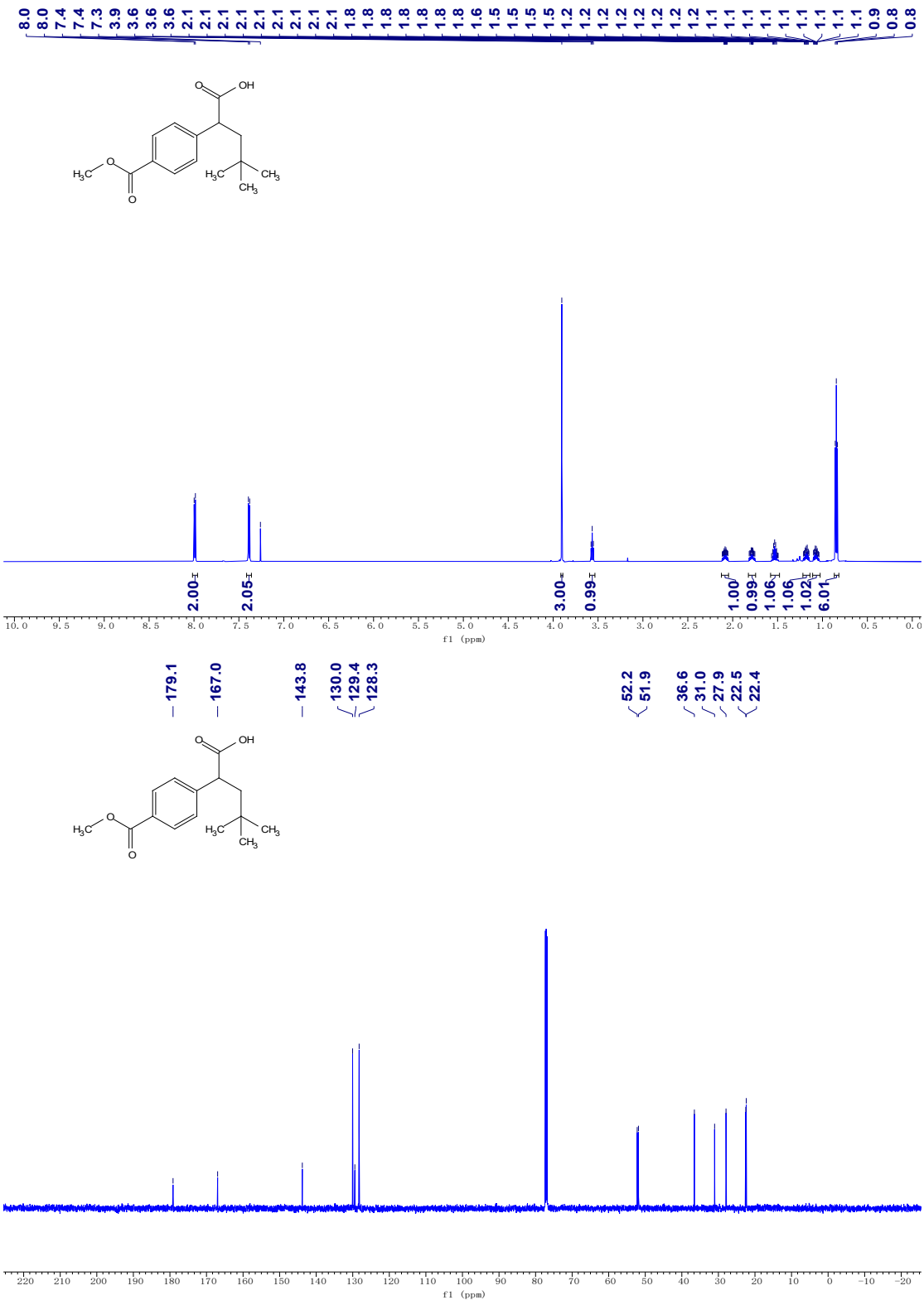
3u


7.99 7.98 7.40 7.38 7.37 7.26 5.31 5.30 3.90 3.63 3.62 3.61 2.47 2.45 2.44 2.42 2.09 2.07 2.06 1.94 1.93 1.92 1.86 1.83 1.82 1.72 1.71 1.70 1.68 1.64 1.62 1.58 1.56 1.53 1.52 1.50 1.44 1.44 1.43 1.42 1.41 1.40 1.26 1.25 1.24 1.18 1.18 1.04 1.00 0.86
CH3
O
OH
H
H
H
H3C
O
O
1.98 1.97 0.73 0.18 3.00 0.90 1.91 3.00 1.03 1.99 5.96 3.95 3.16 0.94 3.04 3.03
f1 (ppm)
221.6 179.2 166.9 143.7 140.1 130.0 129.5 128.3 121.1
52.2 51.9 50.5 49.2 47.6 37.6 36.5 35.9 34.1 34.0 31.5 30.8 26.0 21.9 20.1 19.5 13.6
f1 (ppm)

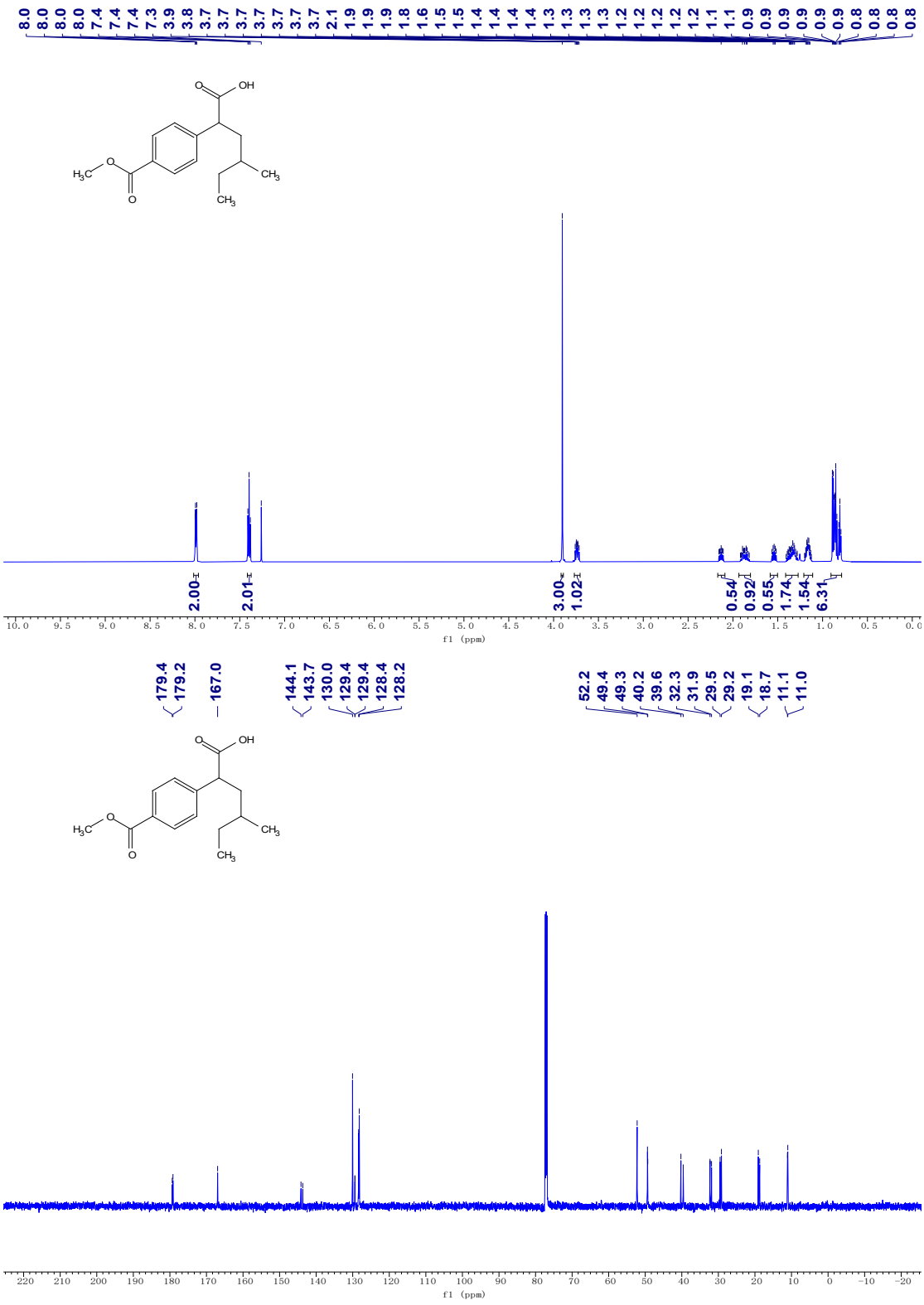
3v


7.99 7.98 7.40 7.39 7.38 3.90 3.63 3.63 2.00 1.98 1.83 1.81 1.57 1.56 1.55 1.53 1.52 1.51 1.45 1.43 1.41 1.41 1.39 1.39 1.37 1.35 1.34 1.33 1.26 1.16 1.14 1.13 1.12 1.11 1.10 1.10 1.08 1.06 1.00 0.98 0.97 0.96 0.94 0.92 0.91 0.87 0.87 0.86 0.86 0.66
1.93 1.92 0.90 2.99 0.15 0.72 0.70 2.25 1.16 4.82 3.83 7.22 1.98 7.18 4.88 0.91 3.08 6.26 3.00
f1 (ppm)
179.7 166.9 143.9 143.8 139.9 139.8 130.0 130.0 129.3 129.3 128.4 128.3 128.2 121.9 121.8 120.0 56.8 56.2 52.2 50.4 49.4 49.2 42.3 39.8 39.6 37.4 37.4 37.2 36.7 36.4 36.3 35.9 34.3 34.1 34.0 33.9 31.9 31.6 28.3 28.1 26.1 25.9 24.3 23.9 22.9 22.6 20.8 19.4 18.8 11.9
f1 (ppm)

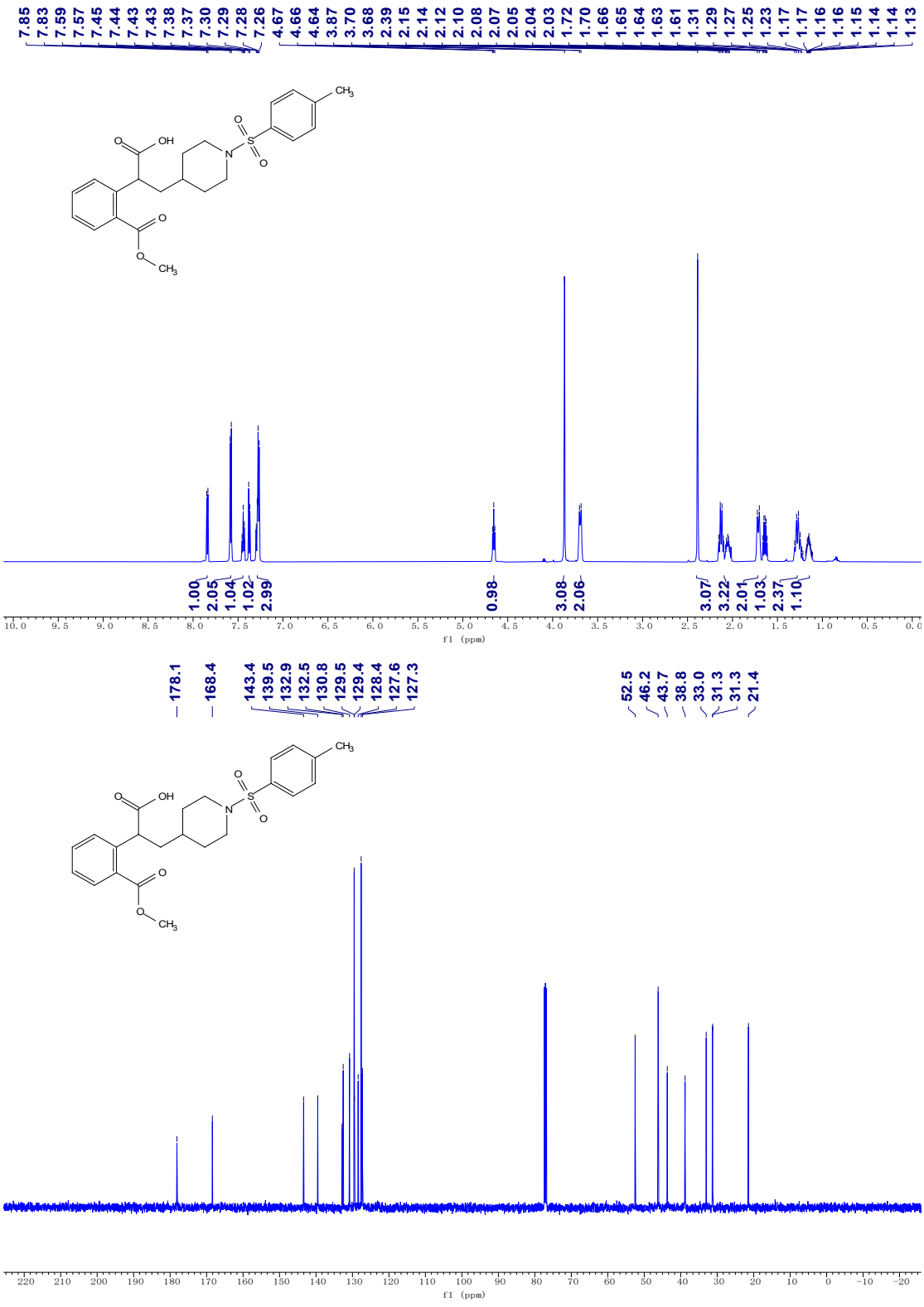
3w


8.0 8.0 7.4 7.4 7.3 3.9 3.6 3.6 3.6 2.1 2.1 2.1 2.1 2.1 2.1 2.1 1.8 1.8 1.8 1.8 1.8 1.8 1.6 1.5 1.5 1.5 1.5 1.2 1.2 1.2 1.2 1.2 1.2 1.2 1.1 1.1 1.1 1.1 1.1 1.1 1.1 1.1 0.9 0.8 0.8
O
OH
H3C
O
O
H3C
CH3
CH3
2.00
2.05
3.00
0.99
1.00
0.99
1.06
1.06
1.02
6.01
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
179.1
167.0
143.8
130.0
129.4
128.3
52.2
51.9
36.6
31.0
27.9
22.5
22.4
O
OH
H3C
O
O
H3C
CH3
CH3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

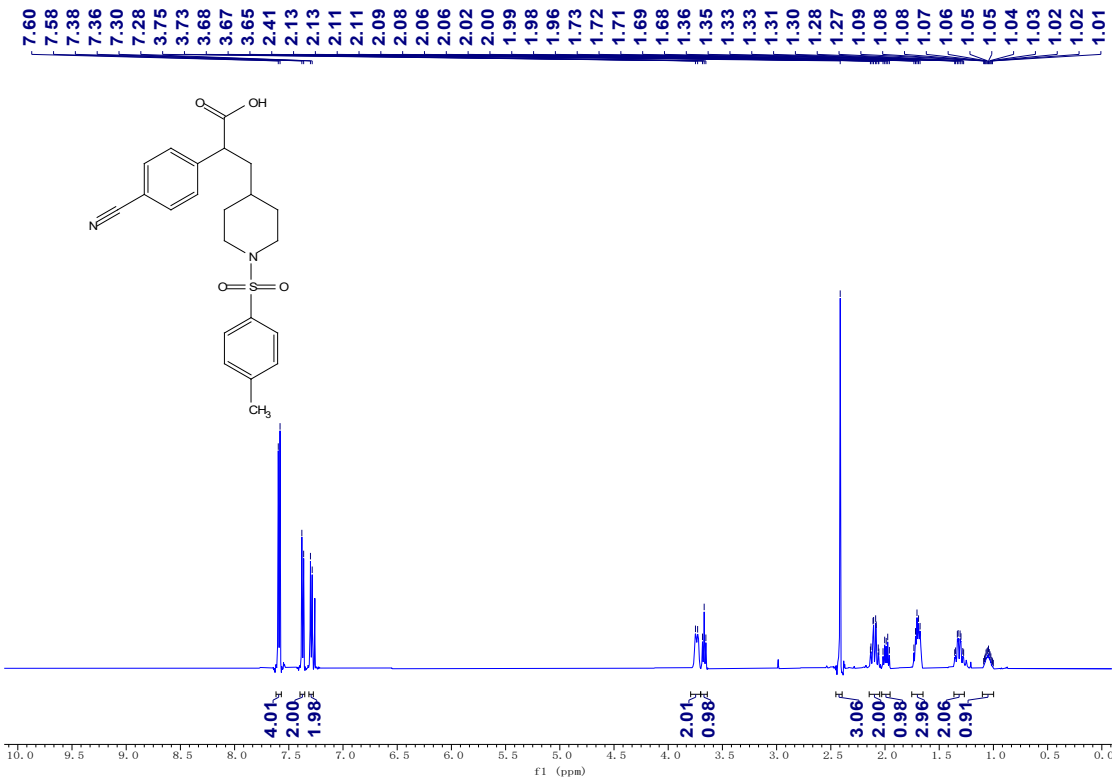
3x

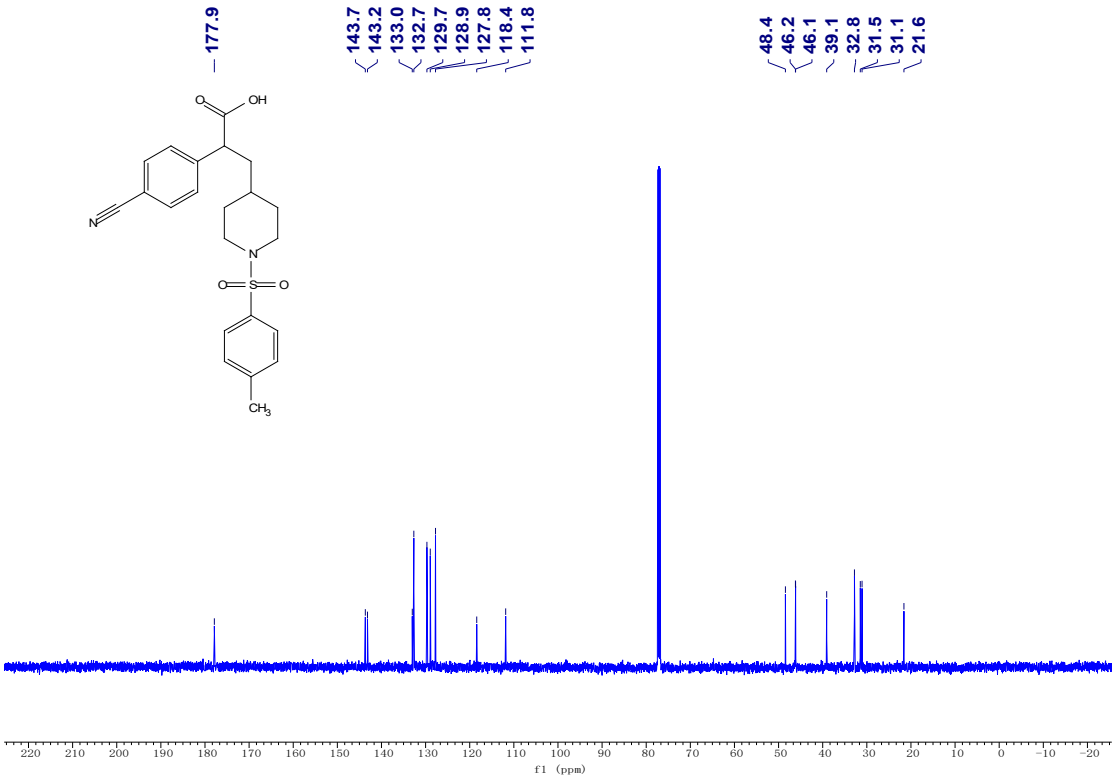

8.0 8.0 8.0 8.0 7.4 7.4 7.4 7.3 3.9 3.8 3.7 3.7 3.7 3.7 3.7 3.7 2.1 1.9 1.9 1.9 1.8 1.6 1.5 1.5 1.4 1.4 1.4 1.4 1.3 1.3 1.3 1.3 1.2 1.2 1.2 1.2 1.1 1.1 0.9 0.9 0.9 0.9 0.9 0.9 0.8 0.8 0.8 0.8
O
OH
H3C
O
O
CH3
CH3
2.00
2.01
3.00
1.02
0.54
0.92
0.55
1.74
1.54
6.31
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
179.4
179.2
167.0
144.1
143.7
130.0
129.4
129.4
128.4
128.2
52.2
49.4
49.3
40.2
39.6
32.3
31.9
29.5
29.2
19.1
18.7
11.1
11.0
O
OH
H3C
O
O
CH3
CH3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

4a

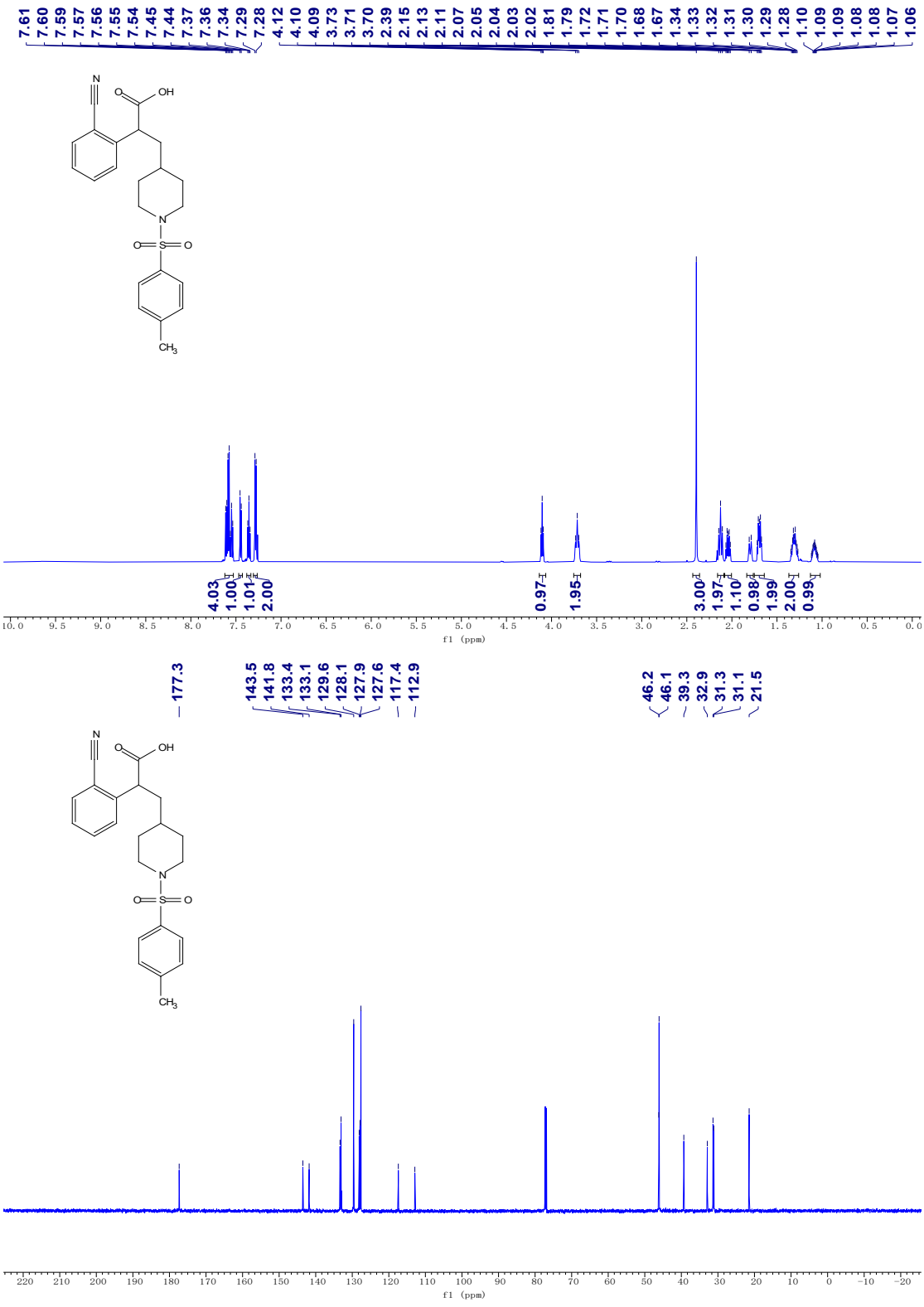

7.85 7.83 7.59 7.57 7.45 7.44 7.43 7.43 7.38 7.37 7.30 7.29 7.28 7.26 4.67 4.66 4.64 3.87 3.70 3.68 2.39 2.15 2.14 2.12 2.10 2.08 2.07 2.05 2.04 2.03 1.72 1.70 1.66 1.65 1.64 1.63 1.61 1.31 1.29 1.27 1.25 1.23 1.17 1.17 1.16 1.16 1.15 1.14 1.14 1.13
1.00 2.05 1.04 1.02 2.99 0.98 3.08 2.06 3.07 3.22 2.01 1.03 2.37 1.10
f1 (ppm)
178.1 168.4 143.4 139.5 132.9 132.5 130.8 129.5 129.4 128.4 127.6 127.3 52.5 46.2 43.7 38.8 33.0 31.3 31.3 21.4
f1 (ppm)

4b

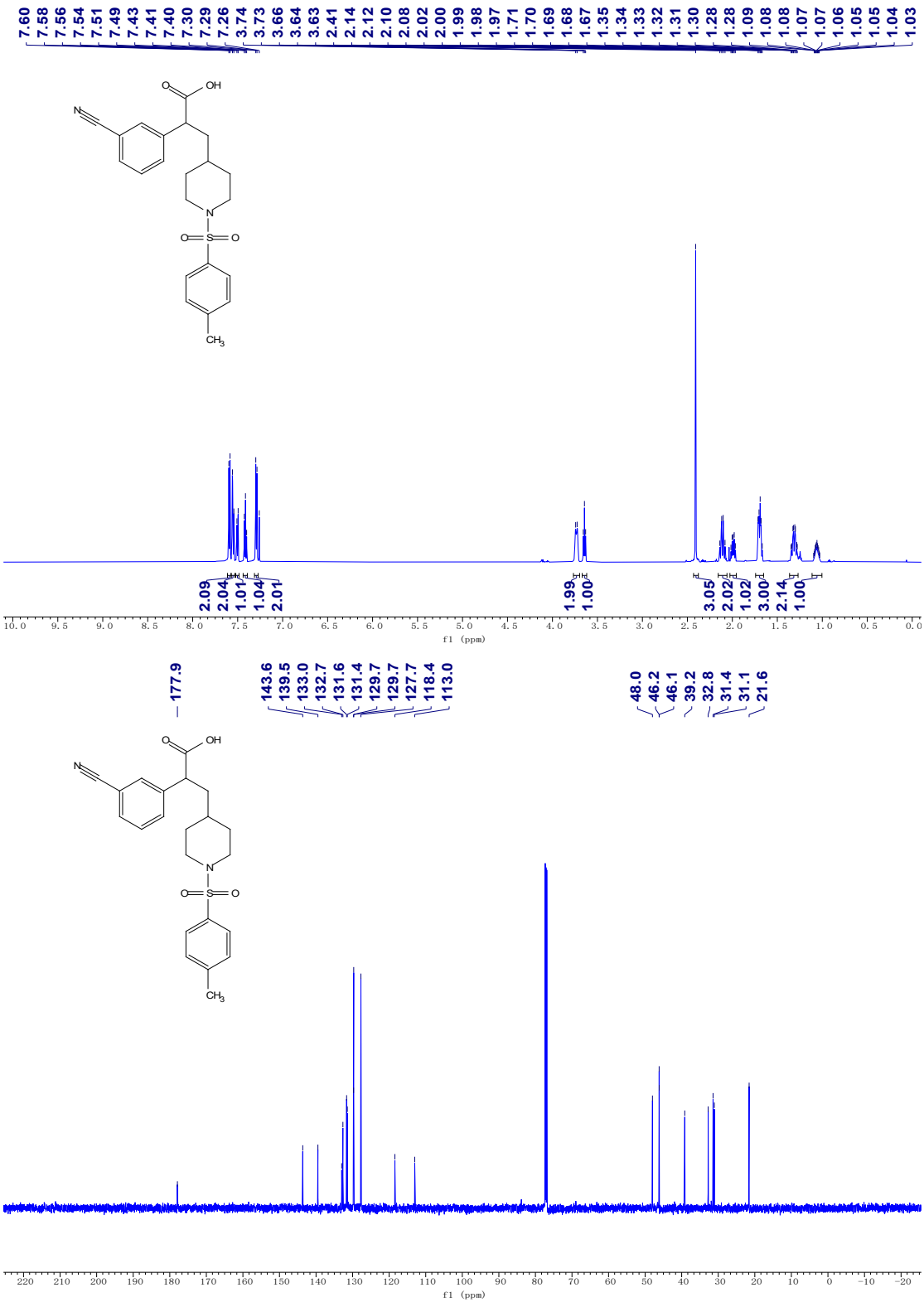

7.60 7.58 7.38 7.36 7.30 7.28 3.75 3.73 3.68 3.67 3.65 2.41 2.13 2.13 2.11 2.11 2.09 2.08 2.06 2.06 2.02 2.00 1.99 1.98 1.96 1.73 1.72 1.71 1.69 1.68 1.36 1.35 1.33 1.33 1.31 1.30 1.28 1.27 1.09 1.08 1.08 1.07 1.06 1.05 1.05 1.04 1.03 1.02 1.02 1.01
O
OH
N
O S O
CH3
4.01 2.00 1.98 2.01 0.98 3.06 2.00 0.98 2.96 2.06 0.91
f1 (ppm)


177.9
143.7 143.2 133.0 132.7 129.7 128.9 127.8 118.4 111.8
48.4 46.2 46.1 39.1 32.8 31.5 31.1 21.6
O
OH
N
O S O
CH3
f1 (ppm)

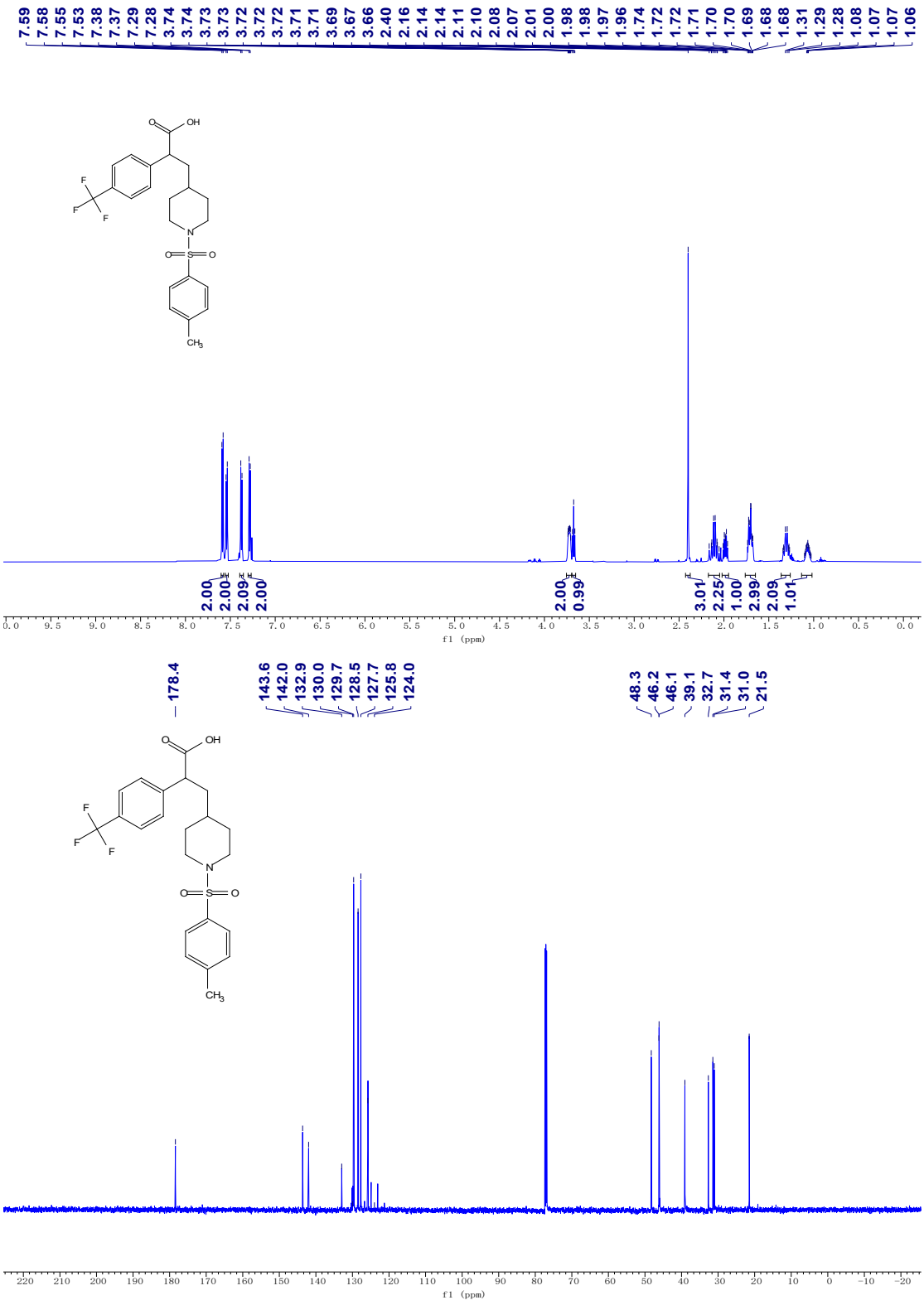
4c


7.61 7.60 7.59 7.57 7.56 7.55 7.54 7.45 7.44 7.37 7.36 7.34 7.29 7.28
4.12 4.10 4.09 3.73 3.71 3.70 2.39 2.15 2.13 2.11 2.07 2.05 2.04 2.03 2.02 1.81 1.79 1.72 1.71 1.70 1.68 1.67 1.34 1.33 1.32 1.31 1.30 1.29 1.28 1.10 1.09 1.09 1.08 1.08 1.07 1.06
N
O
OH
N
O S O
CH3
4.03 1.00 1.01 2.00 0.97 1.95 3.00 1.97 1.10 0.98 1.99 2.00 0.99
f1 (ppm)
177.3 143.5 141.8 133.4 133.1 129.6 128.1 127.9 127.6 117.4 112.9 46.2 46.1 39.3 32.9 31.3 31.1 21.5
f1 (ppm)

4d

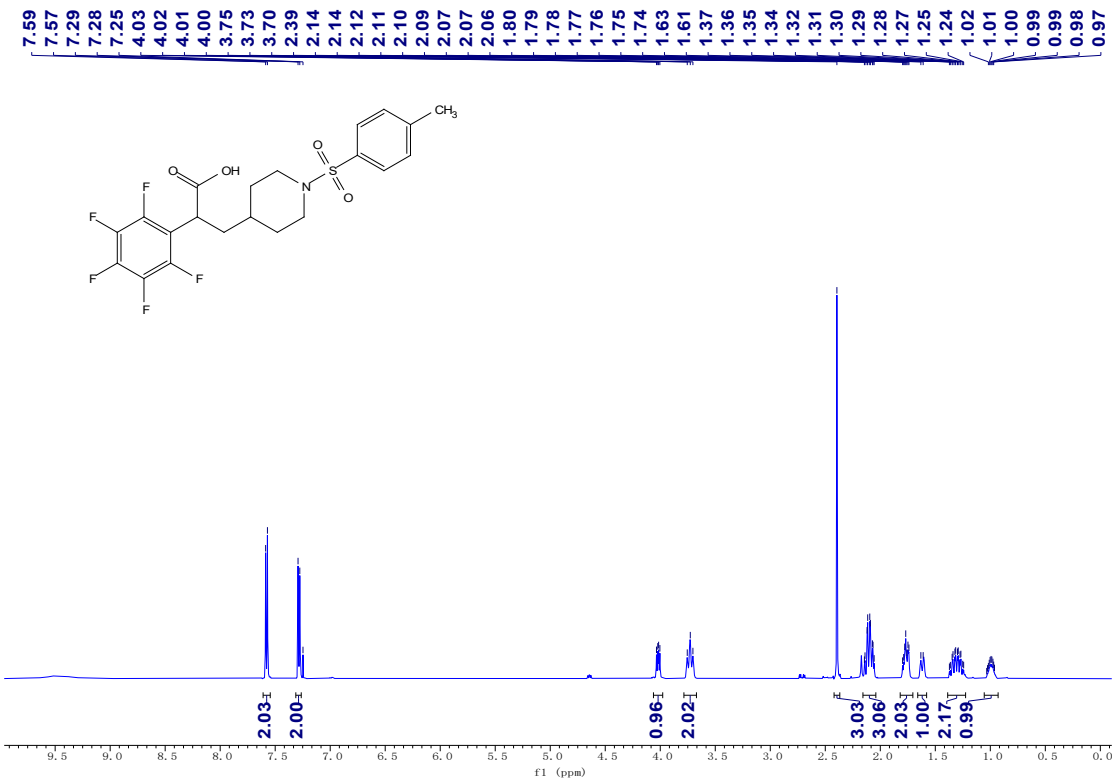

7.60 7.58 7.56 7.54 7.51 7.49 7.43 7.41 7.40 7.30 7.29 7.26 3.74 3.73 3.66 3.64 3.63 2.41 2.14 2.12 2.10 2.08 2.02 2.00 1.99 1.98 1.97 1.71 1.70 1.69 1.68 1.67 1.35 1.34 1.33 1.32 1.31 1.30 1.28 1.28 1.09 1.08 1.08 1.07 1.07 1.06 1.05 1.05 1.04 1.03
O
OH
N
O
S
O
CH3
2.09 2.04 1.01 1.04 2.01 1.99 1.00 3.05 2.02 1.02 3.00 2.14 1.00
f1 (ppm)
177.9
143.6 139.5 133.0 132.7 131.6 131.4 129.7 129.7 127.7 118.4 113.0
48.0 46.2 46.1 39.2 32.8 31.4 31.1 21.6
f1 (ppm)

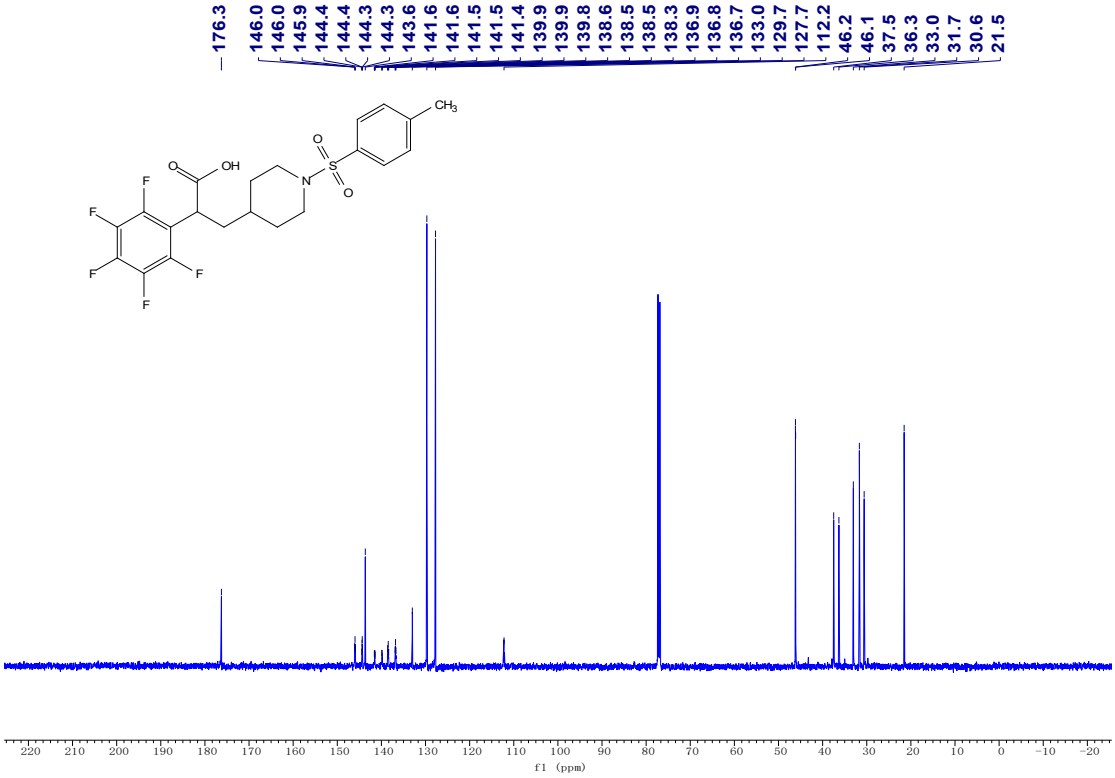
4e

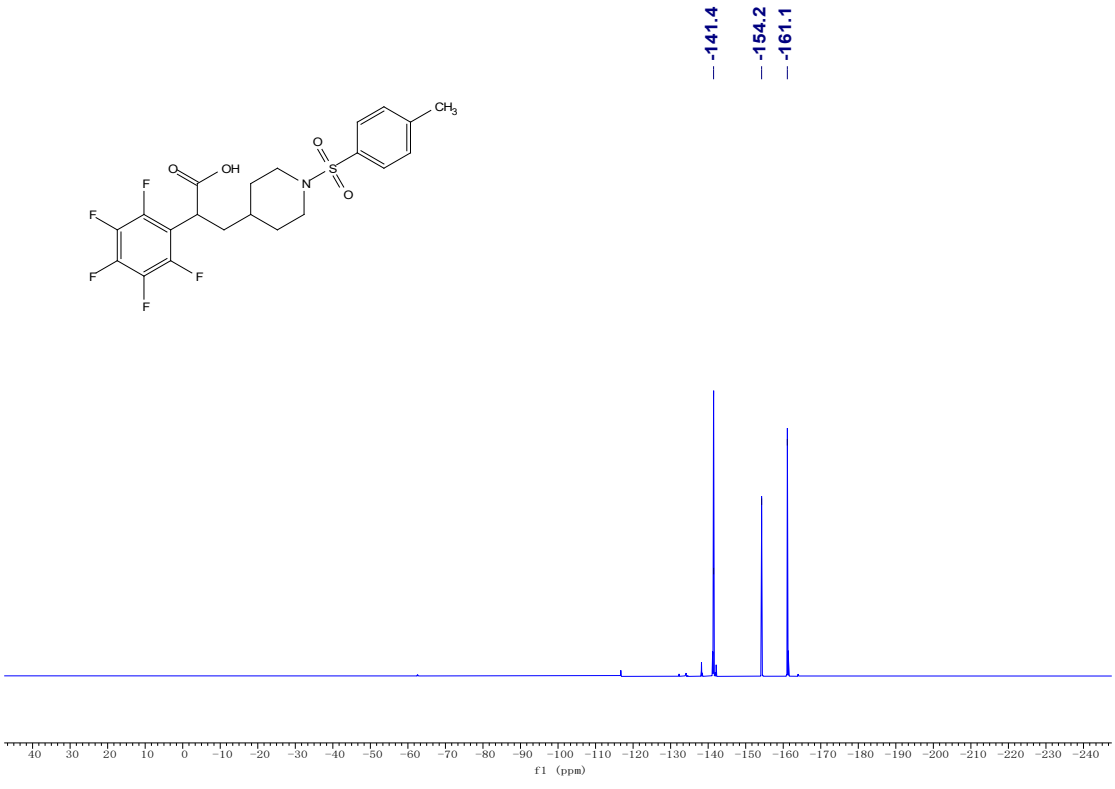

7.59 7.58 7.55 7.53 7.38 7.37 7.29 7.28 3.74 3.74 3.73 3.73 3.72 3.72 3.72 3.71 3.71 3.69 3.67 3.66 2.40 2.16 2.14 2.14 2.11 2.10 2.08 2.07 2.01 2.00 1.98 1.98 1.97 1.96 1.74 1.72 1.72 1.71 1.70 1.70 1.69 1.68 1.68 1.31 1.29 1.28 1.08 1.07 1.07 1.06
OH
F
F
F
N
O=S=O
CH3
2.00 2.00 2.09 2.00 2.00 0.99 3.01 2.25 1.00 2.99 2.09 1.01
f1 (ppm)
178.4
143.6 142.0 132.9 130.0 129.7 128.5 127.7 125.8 124.0
48.3 46.2 46.1 39.1 32.7 31.4 31.0 21.5
f1 (ppm)


-62.5
O
OH
F
F
F
N
O
S
O
CH3
40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240
f1 (ppm)

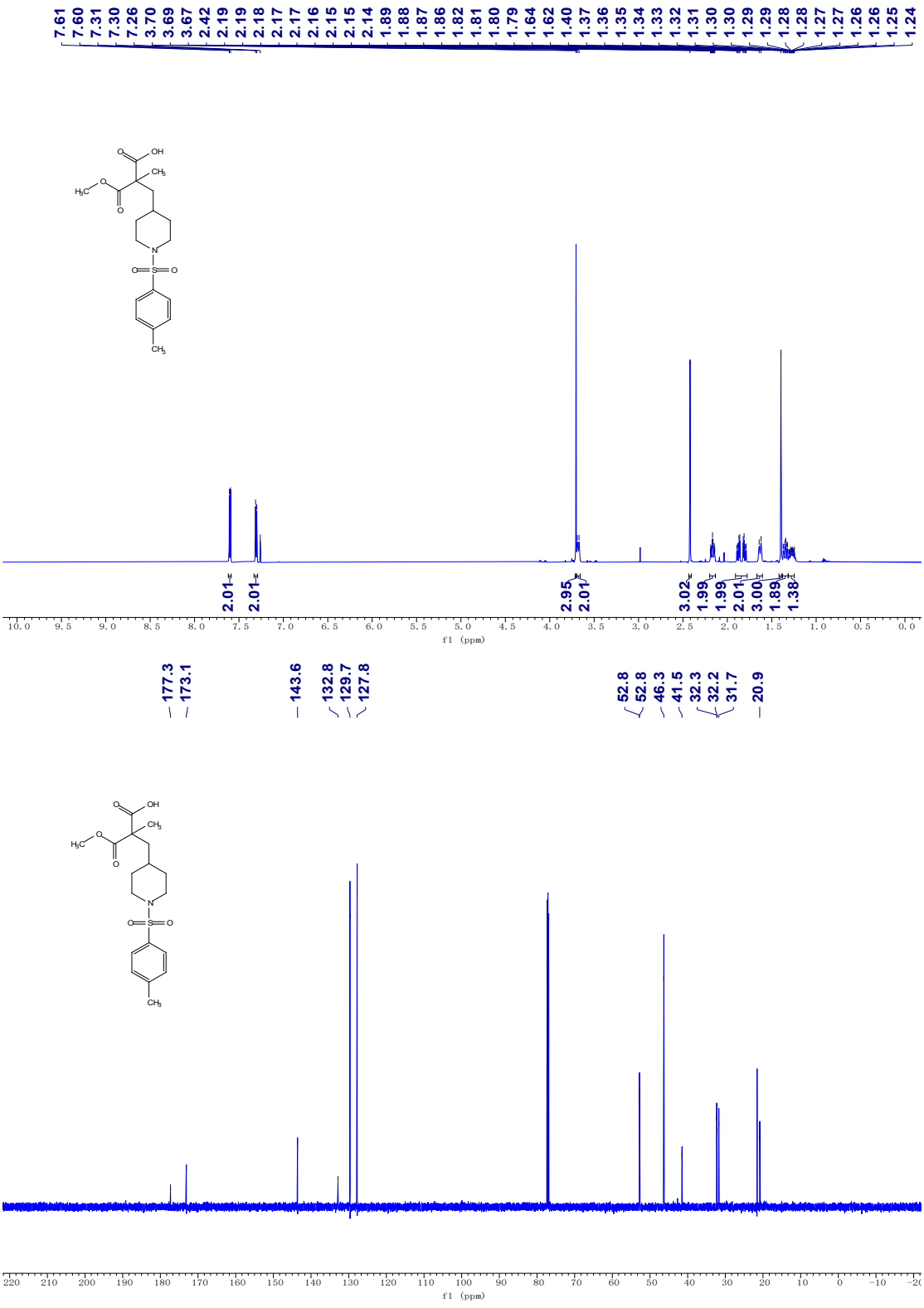
4f


7.59 7.57 7.29 7.28 7.25 4.03 4.02 4.01 4.00 3.75 3.73 3.70 2.39 2.14 2.14 2.12 2.11 2.10 2.09 2.07 2.07 2.06 1.80 1.79 1.78 1.77 1.76 1.75 1.74 1.63 1.61 1.37 1.36 1.35 1.34 1.32 1.31 1.30 1.29 1.28 1.27 1.25 1.24 1.02 1.01 1.00 0.99 0.99 0.98 0.97
CH3
O
OH
F
F
F
F
F
N
S
O
O
2.03 2.00 0.96 2.02 3.03 3.06 2.03 1.00 2.17 0.99
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

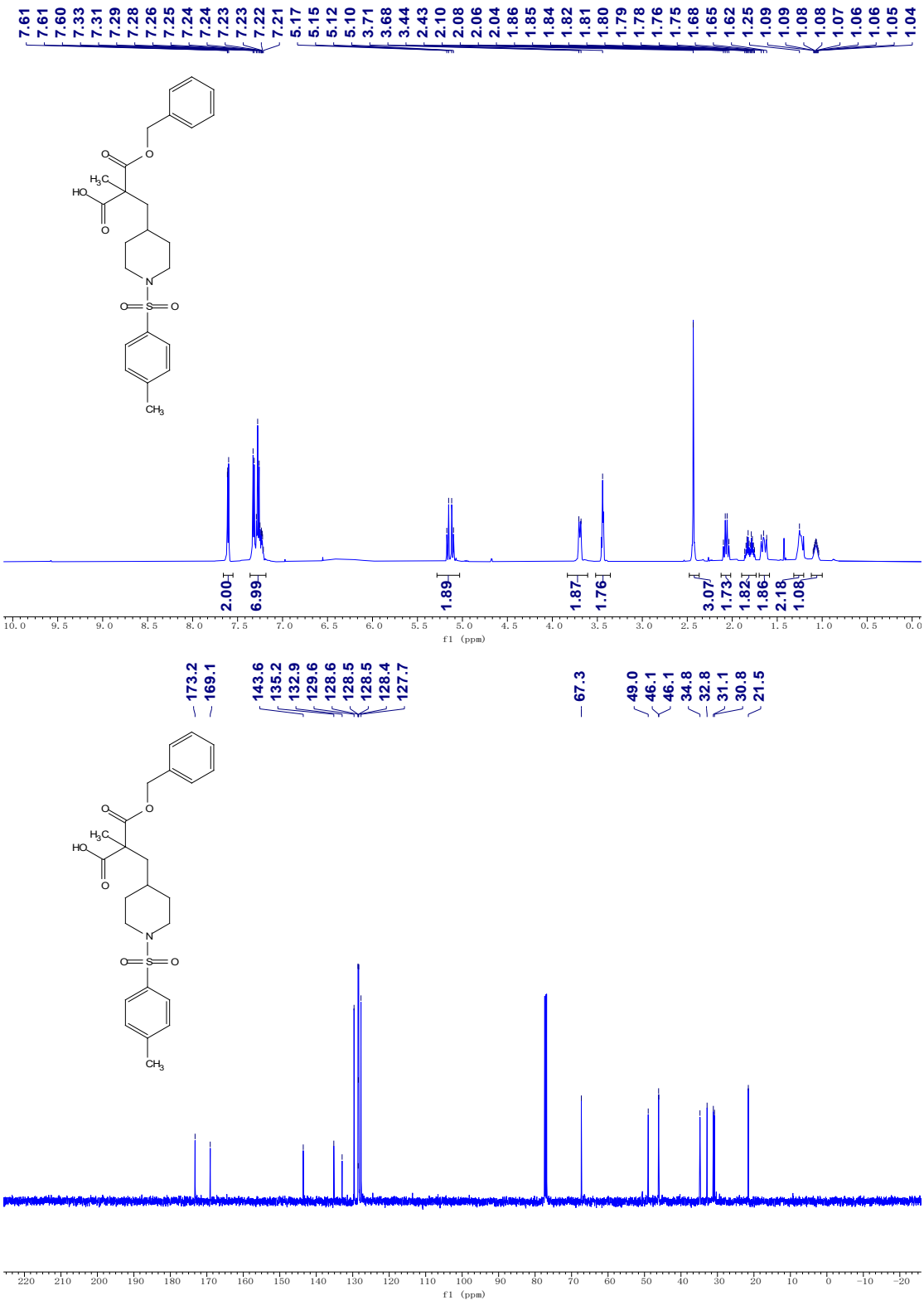

176.3
146.0
146.0
145.9
144.4
144.4
144.3
144.3
143.6
141.6
141.6
141.5
141.5
141.4
139.9
139.9
139.8
138.6
138.5
138.5
138.3
136.9
136.8
136.7
133.0
129.7
127.7
112.2
46.2
46.1
37.5
36.3
33.0
31.7
30.6
21.5
CH3
O
OH
S
N
F
f1 (ppm)


-141.4
-154.2
-161.1
CH3
O
OH
S
N
F
f1 (ppm)

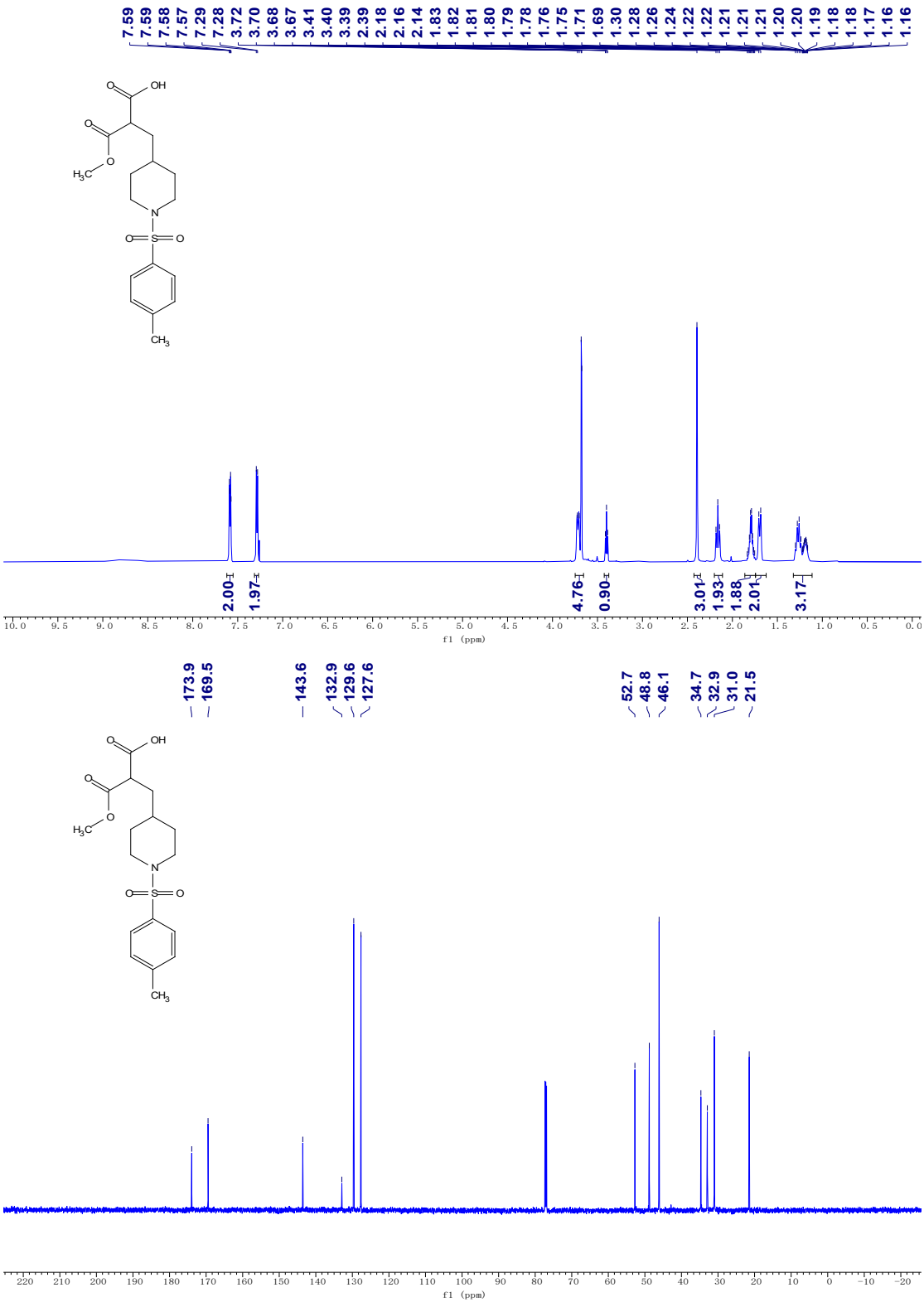
4g


7.61 7.60 7.31 7.30 7.26 3.70 3.69 3.67 2.42 2.19 2.19 2.18 2.17 2.17 2.16 2.15 2.15 2.14 1.89 1.88 1.87 1.86 1.82 1.81 1.80 1.79 1.64 1.62 1.40 1.37 1.36 1.35 1.34 1.33 1.32 1.31 1.30 1.30 1.29 1.29 1.28 1.28 1.27 1.27 1.26 1.26 1.25 1.24
2.01 2.01 2.95 2.01 3.02 1.99 1.99 2.01 3.00 1.89 1.38
f1 (ppm)
177.3 173.1 143.6 132.8 129.7 127.8 52.8 52.8 46.3 41.5 32.3 32.2 31.7 20.9
f1 (ppm)

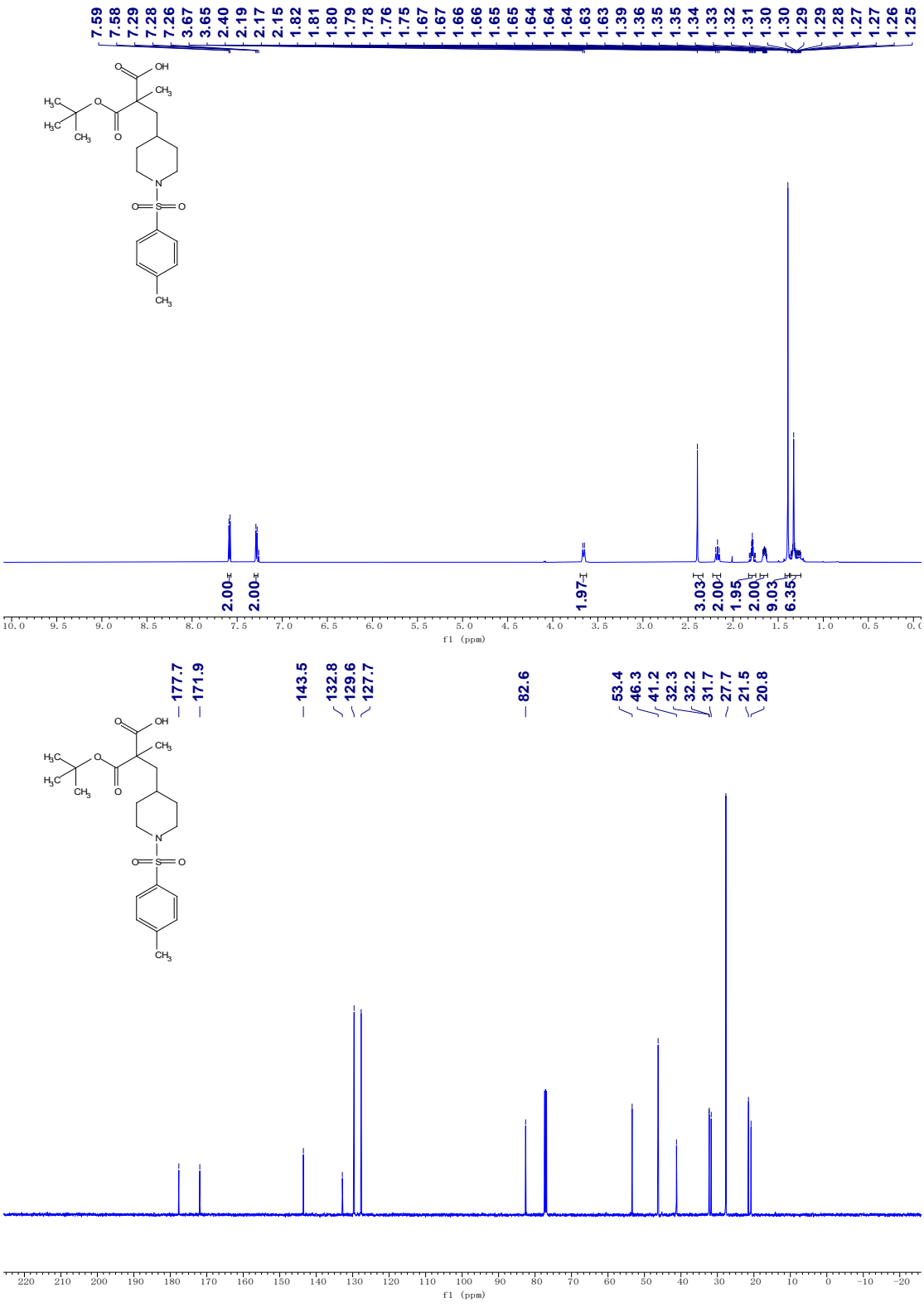
4h


7.61 7.61 7.60 7.33 7.31 7.29 7.28 7.26 7.25 7.24 7.24 7.23 7.23 7.22 7.21 5.17 5.15 5.12 5.10 3.71 3.68 3.44 2.43 2.10 2.08 2.06 2.04 1.86 1.85 1.84 1.82 1.81 1.80 1.79 1.78 1.76 1.75 1.68 1.65 1.62 1.25 1.09 1.09 1.08 1.08 1.07 1.06 1.06 1.05 1.04
2.00 6.99 1.89 1.87 1.76 3.07 1.73 1.82 1.86 2.18 1.08
f1 (ppm)
173.2 169.1 143.6 135.2 132.9 129.6 128.6 128.5 128.5 128.4 127.7 67.3 49.0 46.1 46.1 34.8 32.8 31.1 30.8 21.5
f1 (ppm)

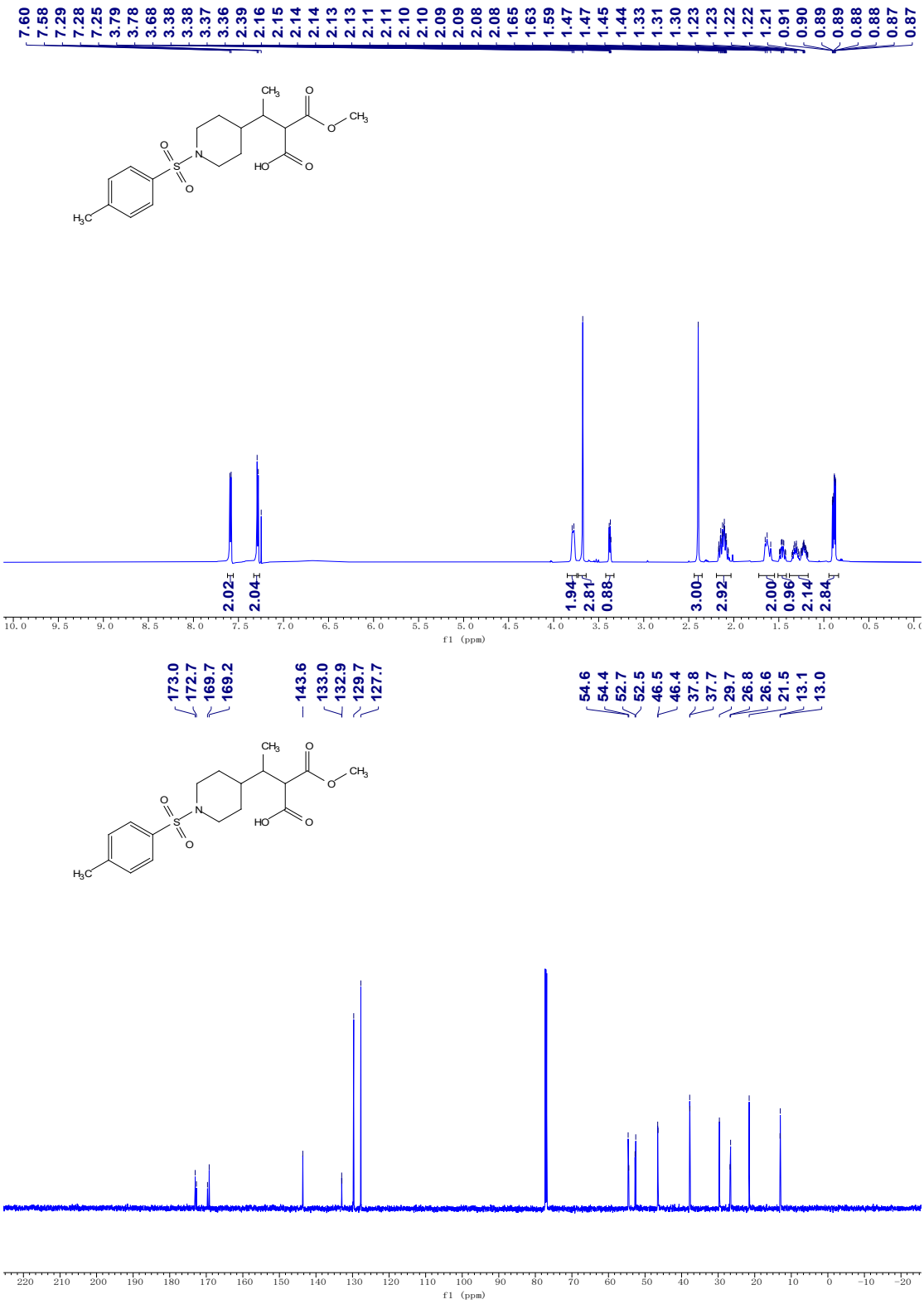
4i


7.59 7.59 7.58 7.57 7.29 7.28 3.72 3.70 3.68 3.67 3.41 3.40 3.39 2.39 2.18 2.16 2.14 1.83 1.82 1.81 1.80 1.79 1.78 1.76 1.75 1.71 1.69 1.30 1.28 1.26 1.24 1.22 1.22 1.21 1.21 1.21 1.20 1.20 1.19 1.18 1.18 1.17 1.16 1.16
2.00 1.97 4.76 0.90 3.01 1.93 1.88 2.01 3.17
f1 (ppm)
173.9 169.5 143.6 132.9 129.6 127.6 52.7 48.8 46.1 34.7 32.9 31.0 21.5
f1 (ppm)

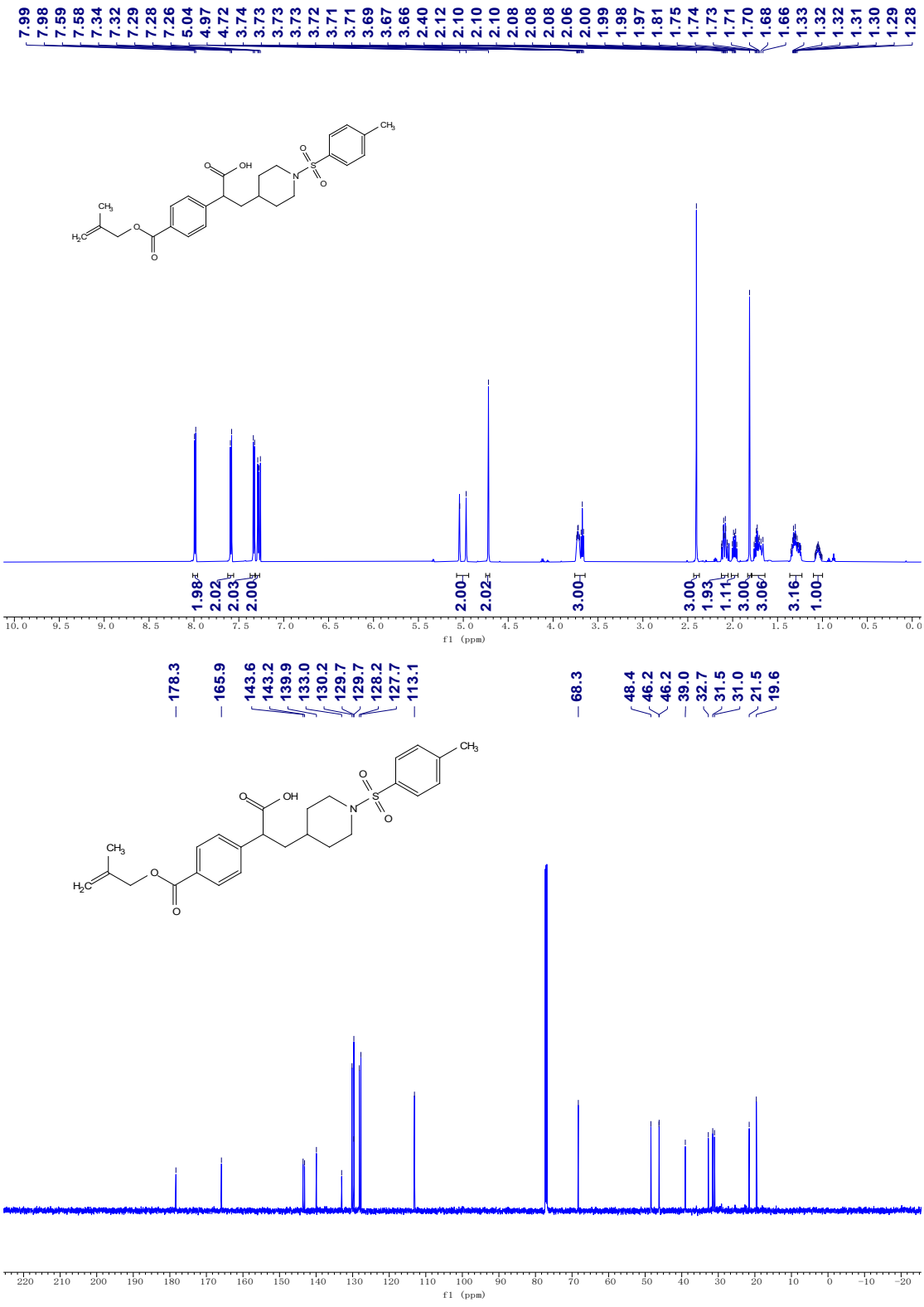
4j


7.59 7.58 7.29 7.28 7.26 3.67 3.65 2.40 2.19 2.17 2.15 1.82 1.81 1.80 1.79 1.78 1.76 1.75 1.67 1.67 1.66 1.66 1.65 1.65 1.64 1.64 1.64 1.63 1.63 1.39 1.36 1.35 1.35 1.34 1.33 1.32 1.31 1.30 1.30 1.29 1.29 1.28 1.27 1.27 1.26 1.25
2.00 2.00 1.97 3.03 2.00 1.95 2.00 9.03 6.35
f1 (ppm)
177.7 171.9 143.5 132.8 129.6 127.7 82.6 53.4 46.3 41.2 32.3 32.2 31.7 27.7 21.5 20.8
f1 (ppm)

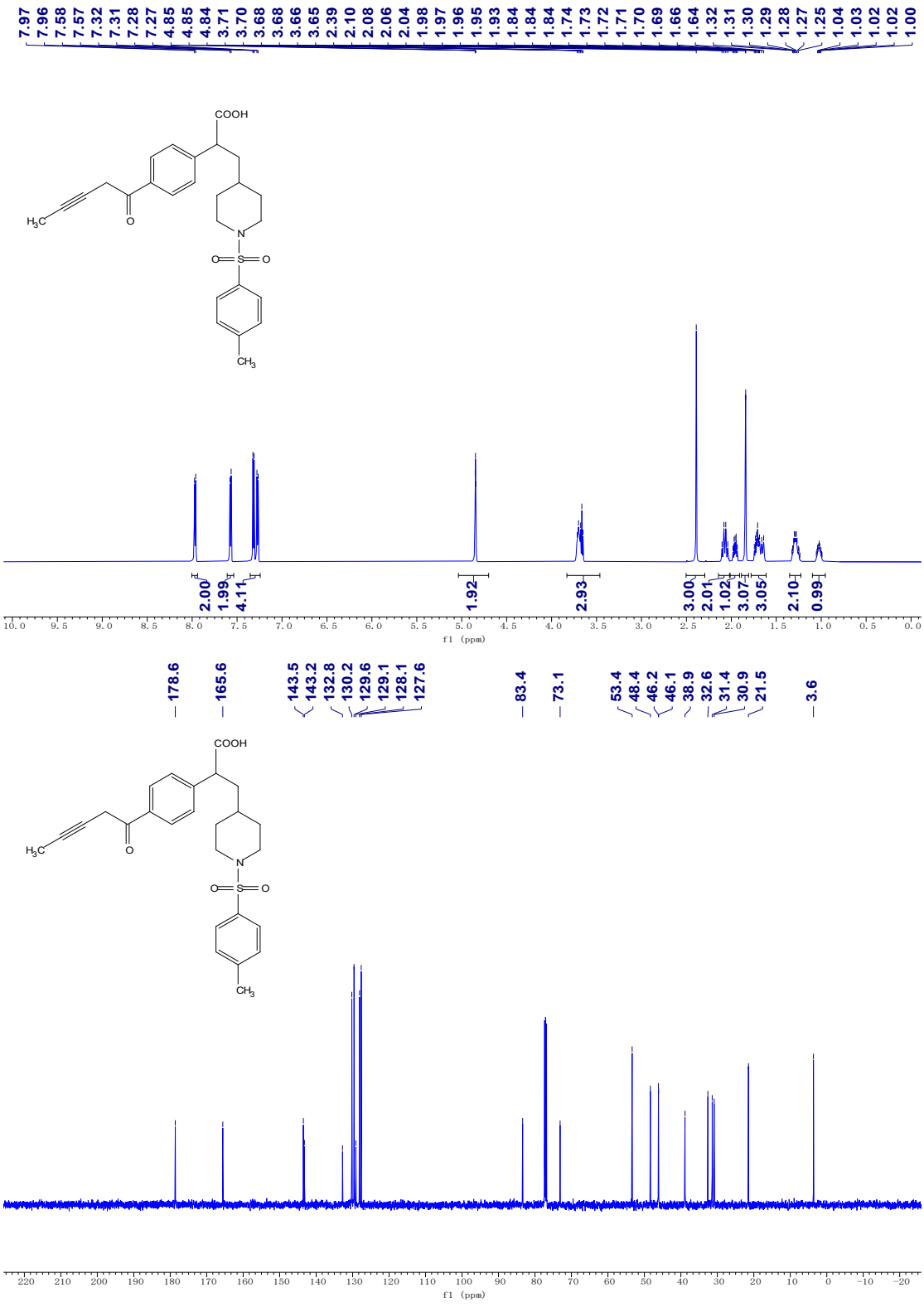
4k


7.60 7.58 7.29 7.28 7.25 3.79 3.78 3.68 3.38 3.38 3.37 3.36 2.39 2.16 2.15 2.14 2.14 2.13 2.13 2.11 2.11 2.10 2.10 2.09 2.09 2.08 2.08 1.65 1.63 1.59 1.47 1.47 1.45 1.44 1.33 1.31 1.30 1.23 1.23 1.22 1.22 1.21 0.91 0.90 0.89 0.89 0.88 0.88 0.87 0.87
2.02 2.04 1.94 2.81 0.88 3.00 2.92 2.00 0.96 2.14 2.84
f1 (ppm)
173.0 172.7 169.7 169.2 143.6 133.0 132.9 129.7 127.7 54.6 54.4 52.7 52.5 46.5 46.4 37.8 37.7 29.7 26.8 26.6 21.5 13.1 13.0
f1 (ppm)

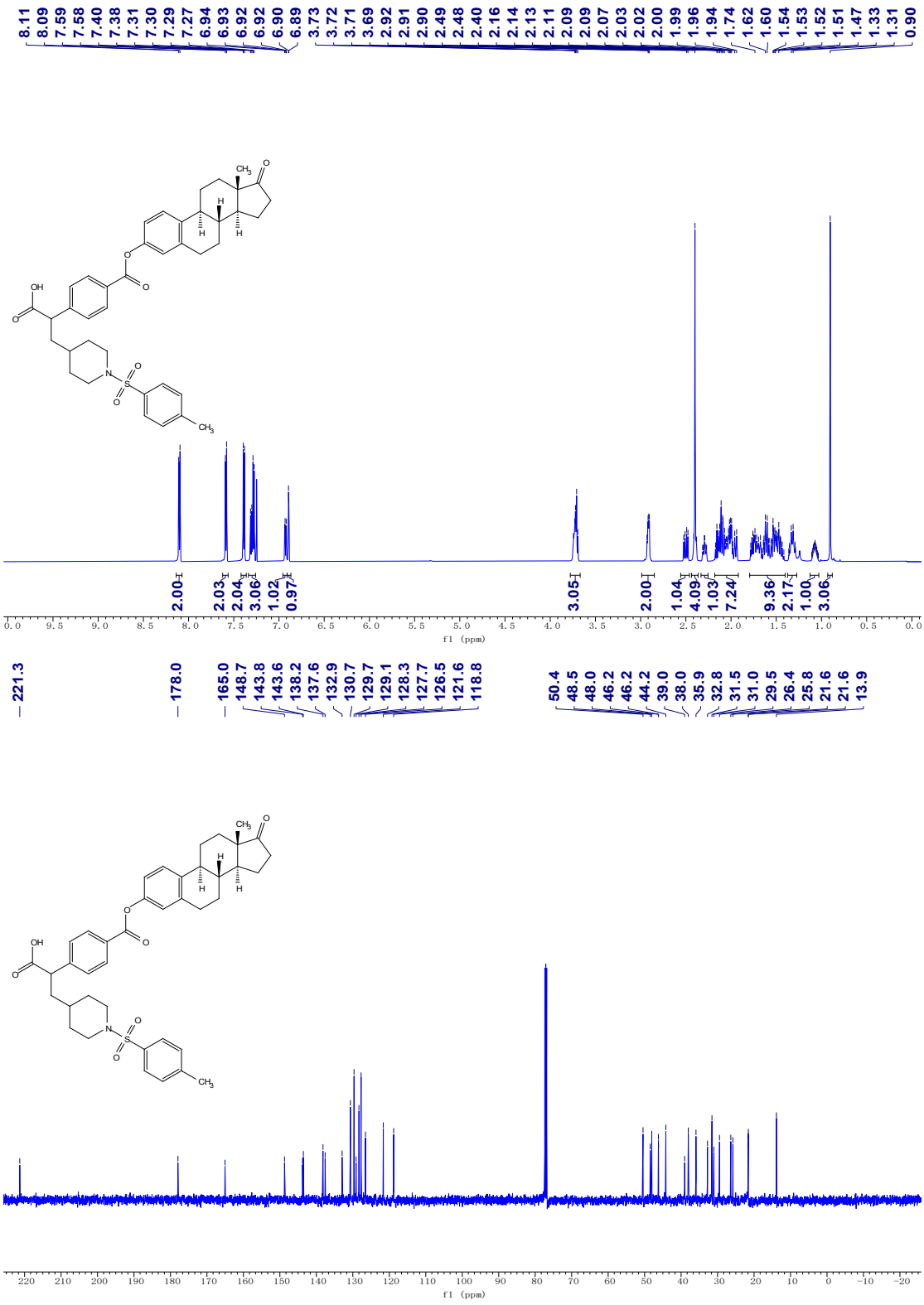
4l


7.99 7.98 7.59 7.58 7.34 7.32 7.29 7.28 7.26 5.04 4.97 4.72 3.74 3.73 3.73 3.73 3.72 3.71 3.71 3.69 3.67 3.66 2.40 2.12 2.10 2.10 2.08 2.08 2.08 2.06 2.00 1.99 1.98 1.97 1.81 1.75 1.74 1.73 1.71 1.70 1.68 1.66 1.33 1.32 1.32 1.31 1.30 1.29 1.28
1.98 2.02 2.03 2.00 2.00 2.02 3.00 3.00 1.93 1.11 3.00 3.06 3.16 1.00
f1 (ppm)
178.3 165.9 143.6 143.2 139.9 133.0 130.2 129.7 129.7 128.2 127.7 113.1 68.3 48.4 46.2 46.2 39.0 32.7 31.5 31.0 21.5 19.6
f1 (ppm)

4m


7.97 7.96 7.58 7.57 7.32 7.31 7.28 7.27 4.85 4.85 4.84 3.71 3.70 3.68 3.68 3.66 3.65 2.39 2.10 2.08 2.06 2.04 1.98 1.97 1.96 1.95 1.93 1.84 1.84 1.84 1.74 1.73 1.72 1.71 1.70 1.69 1.66 1.64 1.32 1.31 1.30 1.29 1.28 1.27 1.25 1.04 1.03 1.02 1.02 1.00
COOH
H3C
O
N
O=S=O
CH3
2.00 1.99 4.11 1.92 2.93 3.00 2.01 1.02 3.07 3.05 2.10 0.99
f1 (ppm)
178.6 165.6 143.5 143.2 132.8 130.2 129.6 129.1 128.1 127.6 83.4 73.1 53.4 48.4 46.2 46.1 38.9 32.6 31.4 30.9 21.5 3.6
COOH
H3C
O
N
O=S=O
CH3
f1 (ppm)

4n


8.11 8.09 7.59 7.58 7.40 7.38 7.31 7.30 7.29 7.27 6.94 6.93 6.92 6.92 6.90 6.89 3.73 3.72 3.71 3.69 2.92 2.91 2.90 2.49 2.48 2.40 2.16 2.14 2.13 2.11 2.09 2.09 2.07 2.03 2.02 2.00 1.99 1.96 1.94 1.74 1.62 1.60 1.54 1.53 1.52 1.51 1.47 1.33 1.31 0.90
2.00 2.03 2.04 3.06 1.02 0.97 3.05 2.00 1.04 4.09 1.03 7.24 9.36 2.17 1.00 3.06
f1 (ppm)
221.3 178.0 165.0 148.7 143.8 143.6 138.2 137.6 132.9 130.7 129.7 129.1 128.3 127.7 126.5 121.6 118.8 50.4 48.5 48.0 46.2 46.2 44.2 39.0 38.0 35.9 32.8 31.5 31.0 29.5 26.4 25.8 21.6 21.6 13.9
f1 (ppm)