Supporting Information

An Effective Late-Stage Functionalization Tool: Direct Deoxymethylation of Phenols

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Experimental Section

1: General information.

All source materials and reagents were purchased from commercial suppliers and are used without pretreatment unless otherwise indicated. All experiments involving palladium were performed using standard Schlenk techniques under nitrogen unless stated otherwise. All results were detected using thin-layer chromatography (TLC) on commercial silica gel plates. Visualization of the developed plates was performed under UV light (254 nm). Rapid column chromatography was performed on silica gel. Column chromatography was performed with silica gel (300-400 mesh) using various combinations of non-aqueous organic solvents as eluents.

NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on Bruker AVANCE III 400/600 MHz (^{1}HNMR) and 101 MHz (^{13}CNMR) instruments with TMS as the internal standard and the following abbreviations were used to identify the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, dq = doublet of quartets, br = broad and all combinations thereof can be explained by their integral parts. Coupling constant (J) was reported in hertz unit (Hz). The high resolution mass spectra (HRMS) were recorded on an Agilent 6210 LC/TOF spectrometer. High-resolution mass spectrometry analysis was performed on the ThermoFlisher ITQ1100.

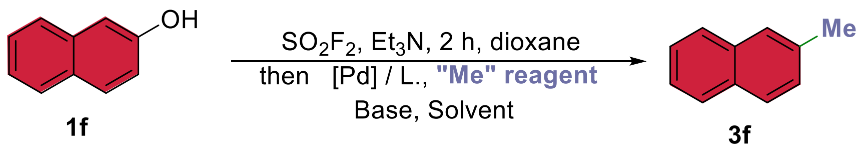
2: Optimization of reaction conditions

General Procedure for Reaction Optimization.

A 25mL Schlenk flask equipped with a stirring bar is filled with Aryl phenols (neat, 0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 2h.[1] Then addition $Pd(OAc)_2$ (typically, 5 mol%), ligand (typically, 6 mol%), base (typically, 0.4 mmol, 2.0 equiv.), “Me” reagent (typically, 0.2 mmol, 1.0 equiv.), solvent (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum as eluent.

1. J. Dong, L. Krasnova, M. G. Finn and K. B. Sharpless, *Angew. Chem., Int. Ed.*, 2014, **53**, 9430-9448.

Table S1. Optimization of the reaction conditions.[a]



Entry	"Me" reagent	Base (equiv.)	Cat. (mol%)	Ligand (mol%)	Solvent	**3f** Yield (%)[b]
1	MeI	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
2	$(CH_3O)_2SO_2$	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
3	$ZnMe_2$	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
4	MeMgBr	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
5	$MeBF_3K$	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	45
6	$MeB(OH)_2$	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	71
7	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	99
8	TMB	KH_2PO_3 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	15
9	TMB	$KHCO_3$ (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	24
10	TMB	Cs_2CO_3 (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	76
11	TMB	t-BuONa (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	56
12	TMB	DBU (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
13	TMB	DIPEA (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
14	TMB	Et_3N (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
15	TMB	DMAP (2.0)	$Pd(OAc)_2$ (5)	DPPE (6)	Dioxane	n.r.
16	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	TNP (6)	Dioxane	15
17	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Cphos (6)	Dioxane	88
18	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	JnhnPhos (6)	Dioxane	93
19	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	S-Phos (6)	Dioxane	97
20	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	99
21	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	tert-Butyl XPhos	Dioxane	97
22	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	THF	18
23	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	DMF	72
24	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	DMSO	33
25	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	CCl_4	n.r.
26	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	EDC	n.r.
27	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	H_2O	n.r.
28	TMB	K_3PO_4 (1.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	79
29	TMB	K_3PO_4 (1.5)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	87

30	TMB	K_3PO_4 (3.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	99
31	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (10)	Ruphos (15)	Dioxane	94
32	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (7)	Ruphos (9)	Dioxane	98
33	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (2)	Ruphos (3)	Dioxane	93
34[c]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (1)	Ruphos (1.5)	Dioxane	98
35[d]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	99
36[e]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	64
37[f]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	n.r.
38[g]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	61
39[h]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	63
40[i]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	n.r.
41[j]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	n.r.
41[k]	TMB	K_3PO_4 (2.0)	$Pd(OAc)_2$ (5)	Ruphos (6)	Dioxane	99

[a] Reaction conditions: **1f** (0.2 mmol), Et_3N (1.0 mmol, 5.0 eq.), dioxane (0.2 M), r.t., 2 h, under SO_2F_2 gas atmosphere. Then addition "Me" reagent (0.2 mmol, 1.0 eq.), $Pd(OAc)_2$, Ligand , Base , Solvent (0.2 M), 120 ºC, 2.0 h; Under nitrogen atmosphere.

[b] Isolated yield.

[c] The time of methylation reaction is 6 h

[d] The time of methylation reaction is 8 h

[e] The time of methylation reaction is 1 h

[f] r.t. Instead of 120 ºC.

[g] 80 ºC Instead of 120 ºC.

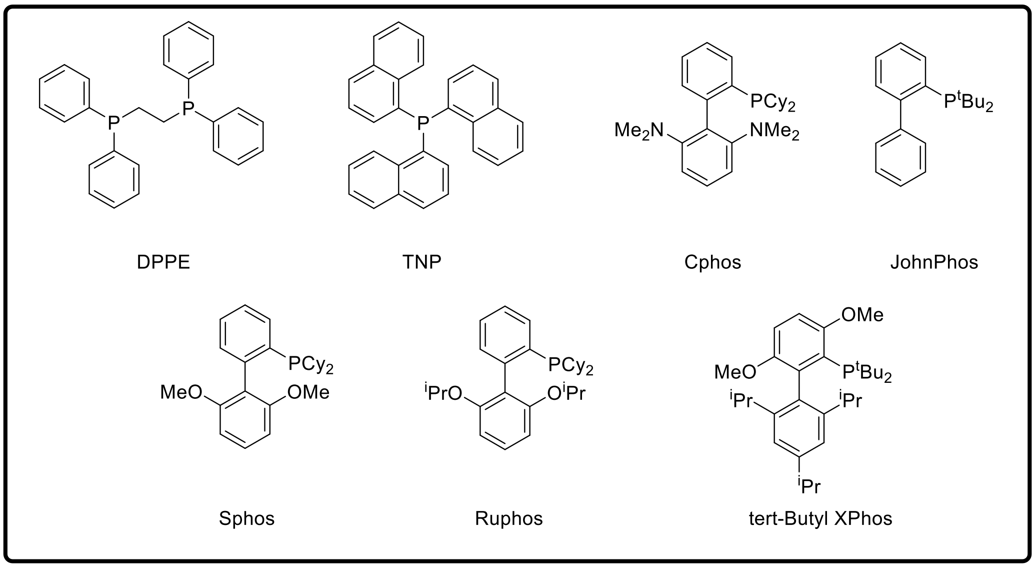
[h] 130ºC Instead of 120 ºC.

[i] air Instead of nitrogen.

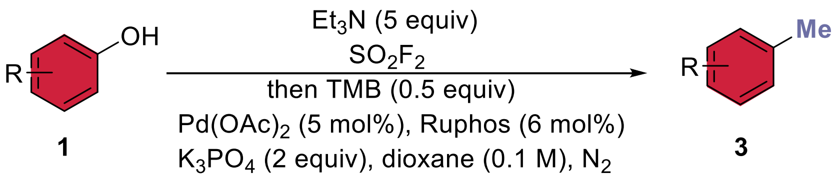
[j] SO_2F_2 gas Instead of nitrogen.

[k] 0.1 mmol of TMB.

TMB = trimethylboroxine, n.r. = no reaction, r.t.= room temperature.

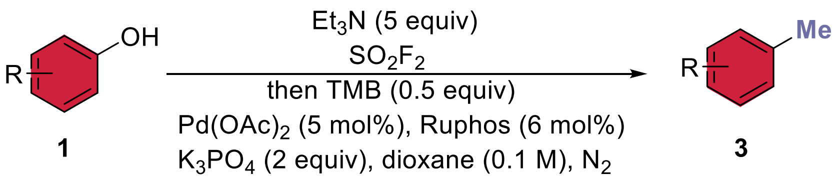


3: Experimental details and characterization of products



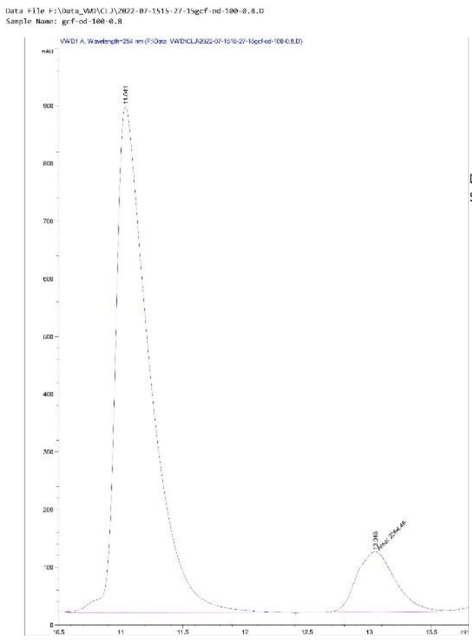
General procedure for the synthesis of Deoxymethylation of Phenols (1a-1w):

A 25mL Schlenk flask equipped with a stirring bar is filled with Aryl phenols (0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 2h. Then addition $Pd(OAc)_2$ (5 mol%), RuPhos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), trimethylboroxine (typically, 0.1 mmol, 0.5 equiv.), dioxane (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 2 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum as eluent to provide the desired products.



General procedure for the synthesis of Deoxymethylation of Phenols in natural molecules (1aa-1ar):

A 25mL Schlenk flask equipped with a stirring bar is filled with Aryl phenols (0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 6h. Then addition $Pd(OAc)_2$ (5 mol%), RuPhos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), trimethylboroxine (typically, 0.1 mmol, 0.5 equiv.), dioxane (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 8 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum as eluent to provide the desired products.



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Area Percent Report

Sorted By : Signal
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Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

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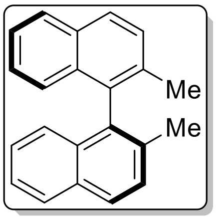
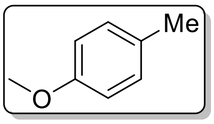


3an



1-methoxy-4-methylbenzene (3a)

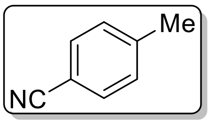
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Yellow oil, 83% yield, 20.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.11 (d, *J* = 8.2 Hz, 2H), 6.86 – 6.80 (m, 2H), 3.81 (s, 3H), 2.31 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.45 , 129.90 , 129.85 , 113.69 , 55.30 , 20.47 .

HRMS (EI-TOF) calcd for $C_8H_{10}O$: 122.0732; Found：122.0733.

NMR spectroscopic data agreed with literature values.[1]



4-methylbenzonitrile (3b)

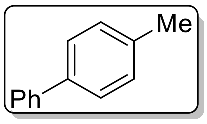
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Colorless oil, 91% yield, 21.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 7.9 Hz, 2H), 2.44 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.73 , 132.07 , 129.86 , 119.20 , 109.30 , 21.87 .

HRMS (EI-TOF) calcd for $C_8H_{10}N$: 117.0578; Found：117.0582.

NMR spectroscopic data agreed with literature values.[2]



4-methyl-1,1'-biphenyl (3c)

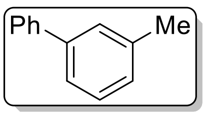
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White solid, 99% yield, 33.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 (d, *J* = 7.6 Hz, 2H), 7.55 (d, *J* = 8.1 Hz, 2H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.45 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.20, 138.39, 137.07, 129.53, 128.76, 127.04, 127.02 , 21.16.

HRMS (EI-TOF) calcd for $C_{13}H_{12}$: 168.0939; Found：168.0940.

NMR spectroscopic data agreed with literature values.[3]



3-methyl-1,1'-biphenyl (3d)

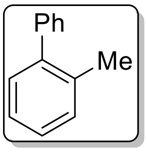
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White oil, 99% yield, 33.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 – 7.68 (m, 2H), 7.58 – 7.49 (m, 4H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.27 (s, 1H), 2.53 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 141.46 , 141.34 , 138.42 , 128.81 , 128.78 , 128.11 , 128.09 , 127.29 , 127.28 , 124.39 , 21.66 .

HRMS (EI-TOF) calcd for $C_{13}H_{12}$: 168.0939; Found：168.0940.

NMR spectroscopic data agreed with literature values.[3]



2-methyl-1,1'-biphenyl (3e)

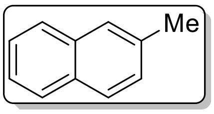
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Colorless oil, 96% yield, 32.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 – 7.47 (m, 2H), 7.42 (t, *J* = 6.6 Hz, 3H), 7.38 – 7.29 (m, 4H), 2.36 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 142.03 , 142.00 , 135.41 , 130.38 , 129.87 , 129.27 , 128.14 , 127.32 , 126.83 , 125.84 , 20.55 .

HRMS (EI-TOF) calcd for $C_{13}H_{12}$: 168.0939; Found：168.0940.

NMR spectroscopic data agreed with literature values.[3]



2-methylnaphthalene (3f)

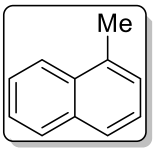
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White solid, 99% yield, 28.2 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 (d, *J* = 8.0 Hz, 1H), 7.95 – 7.89 (m, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.58 (pd, *J* = 6.9, 1.4 Hz, 2H), 7.48 – 7.37 (m, 2H), 2.77 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 134.33, 133.60, 132.67, 128.59, 126.62, 126.44, 125.78, 125.64, 125.60, 124.18, 19.46.

HRMS (EI-TOF) calcd for $C_{11}H_{10}$: 142.0783; Found：142.0788.

NMR spectroscopic data agreed with literature values.[1]



1-Methylnaphthalene (3g)

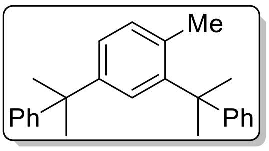
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Colorless oil, 99% yield, 28.2 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 7.8 Hz, 1H), 7.78 (dd, *J* = 8.1, 3.7 Hz, 2H), 7.65 (s, 1H), 7.46 (pd, *J* = 6.9, 1.4 Hz, 2H), 7.35 (dd, *J* = 8.4, 1.6 Hz, 1H), 2.55 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.47 , 133.67 , 131.70 , 128.14 , 127.70 , 127.62 , 127.24 , 126.85 , 125.88 , 124.97 , 21.75.

HRMS (EI-TOF) calcd for $C_{11}H_{10}$: 142.0783; Found：142.0783.

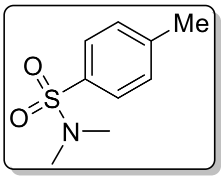
NMR spectroscopic data agreed with literature values.[1]



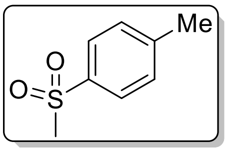
((4-methyl-1,3-phenylene)bis(propane-2,2-diyl))dibenzene (3h)

Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Yellow oil, 61% yield, 40.1 mg.

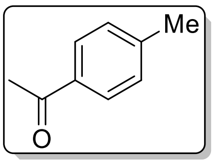
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (s, 1H), 7.40 – 7.21 (m, 10H), 7.11 (d, *J* = 7.9 Hz, 1H), 7.03 (d, *J* = 7.9 Hz, 1H), 1.83 (s, 9H), 1.69 (s, 6H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 151.14 , 151.07 , 147.77 , 146.75 , 134.08 , 132.04 , 128.25 , 128.03 , 126.87 , 125.94 , 125.61 , 125.31 , 124.80 , 124.72 , 43.76 , 42.96 , 30.99 , 30.82 , 21.27 .
HRMS (EI-TOF) calcd for $C_{25}H_{28}$: 328.2191; Found： 328.2198.



_N, N_, 4-trimethylbenzenesulfonamide (3i)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). White solid, 90% yield, 35.9 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 8.2 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 2.70 (s, 6H), 2.45 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.51 , 132.38 , 129.64 , 127.81 , 37.98 , 21.54 .
HRMS (EI-TOF) calcd for $C_9H_{13}NO_2S$: 199.0667; Found： 199.0672.
NMR spectroscopic data agreed with literature values.[4]

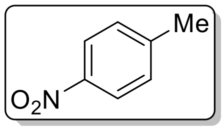


1-methyl-4-(methylsulfonyl)benzene (3j)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). White solid, 99% yield, 33.7 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 3.04 (s, 3H), 2.45 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.70, 137.68, 129.98, 127.36, 44.62, 21.63.
HRMS (EI-TOF) calcd for $C_8H_{10}O_2S$: 170.0402; Found： 170.0405.
NMR spectroscopic data agreed with literature values.[5]



1-(p-tolyl)ethan-1-one (3k)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Colorless oil, 99% yield, 26.5 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 2.60 (s, 3H), 2.44 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 197.93 , 143.91 , 134.71 , 129.26 , 128.46 , 26.57 , 21.67 .
HRMS (EI-TOF) calcd for $C_9H_{10}O$: 134.0732; Found： 134.0740.
NMR spectroscopic data agreed with literature values.[5]

1-methyl-4-nitrobenzene (3l)



Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). White solid, 91% yield, 25.0 mg.

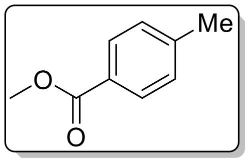
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (d, J = 8.7 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 2.49 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.88 , 129.77 , 123.51 , 21.57 .

HRMS (EI-TOF) calcd for $C_7H_7NO_2$: 137.0477; Found： 137.0478.

NMR spectroscopic data agreed with literature values.[6]

methyl 4-methylbenzoate (3m)



Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Colorless oil, 99% yield, 29.7 mg.

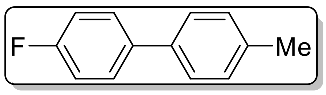
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 3.92 (s, 3H), 2.42 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.20 , 143.57 , 129.61 , 129.09 , 127.43 , 51.96 , 21.66 .

HRMS (EI-TOF) calcd for $C_9H_{10}O_2$: 150.0681; Found： 150.0683.

NMR spectroscopic data agreed with literature values.[5]

4-fluoro-4'-methyl-1,1'-biphenyl (3n)



Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White solid, 98% yield, 36.5 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 – 7.57 (m, 2H), 7.52 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 2H), 7.25 – 7.13 (m, 2H), 2.48 (s, 3H).

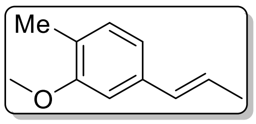
^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.60 , 161.15 , 137.39 (d, *J* = 13.5 Hz), 137.10 , 129.62 , 128.53 (d, *J* = 8.0 Hz), 126.92 , 115.73 , 115.51 , 21.14 .

^{19}F NMR (565 MHz, CDCl3) δ -116.22 .

HRMS (EI-TOF) calcd for $C_{13}H_{11}F$: 186.0845; Found： 186.0849.

NMR spectroscopic data agreed with literature values.[7]

2-methoxy-1-methyl-4-(prop-1-en-1-yl)benzene (3o)



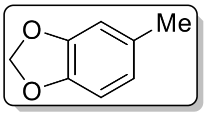
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 30 : 1). Colorless oil, 97% yield, 31.5 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.11 (d, *J* = 7.5 Hz, 1H), 6.96 – 6.81 (m, 2H), 6.44 (d, *J* = 15.9 Hz, 1H), 6.35 – 6.18 (m, 1H), 3.90 (s, 3H), 2.27 (s, 3H), 1.95 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.83 , 136.99 , 131.21 , 130.63 , 125.34 , 124.79 , 118.12 , 107.26 , 55.21 , 18.49 , 16.06 .

HRMS (EI-TOF) calcd for $C_{11}H_{14}O$: 162.1045; Found： 162.1052.

NMR spectroscopic data agreed with literature values.[8]



5-methylbenzo[d][1,3]dioxole (3p)

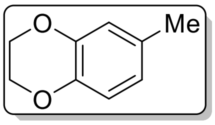
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Colorless oil, 82% yield, 22.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.78 – 6.61 (m, 3H), 5.94 (s, 2H), 2.31 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 147.47 , 145.27 , 131.51 , 121.53 , 109.61 , 108.06 , 100.69 , 21.20 .

HRMS (EI-TOF) calcd for $C_8H_8O_2$: 136.0524; Found： 136.0529.

NMR spectroscopic data agreed with literature values.[9]



6-methyl-2,3-dihydrobenzo[b][1,4]dioxine (3q)

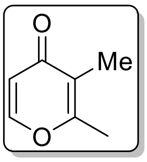
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Colorless oil, 84% yield, 25.2 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.80 (d, *J* = 8.2 Hz, 1H), 6.76 – 6.63 (m, 2H), 4.26 (s, 4H), 2.29 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 143.21 , 141.32 , 131.09 , 121.97 , 117.66 , 116.93 , 64.45 , 64.34 , 20.69 .

HRMS (EI-TOF) calcd for $C_9H_{10}O_2$: 150.0681; Found： 150.0681.

NMR spectroscopic data agreed with literature values.[8]



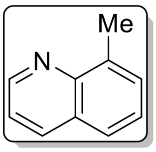
2,3-dimethyl-4*H*-pyran-4-one (3r)

Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 1 : 1). Yellow solid, 83% yield, 20.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (d, *J* = 5.6 Hz, 1H), 6.33 (d, *J* = 5.6 Hz, 1H), 2.32 (s, 3H), 1.98 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.91 , 162.02 , 154.19 , 122.51 , 115.11 , 17.78 , 9.90 .

HRMS (EI-TOF) calcd for $C_7H_8O_2$: 124.0524; Found： 124.0532.

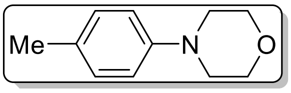


8-methylquinoline (3s)

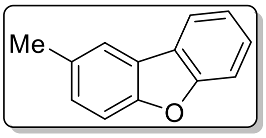
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Yellow oil, 45% yield, 12.9 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.98 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.16 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.69 (d, *J* = 8.2 Hz, 1H), 7.59 (d, *J* = 6.9 Hz, 1H), 7.50 – 7.40 (m, 2H), 2.85 (s, 3H).

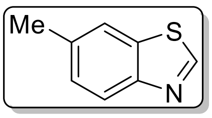
^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.25 , 147.29 , 137.05 , 136.44 , 129.71 , 128.30 , 126.36 , 125.91 , 120.87 , 18.21 .
HRMS (EI-TOF) calcd for $C_{10}H_9N$: 143.0735; Found：143.0745.
NMR spectroscopic data agreed with literature values.[10]



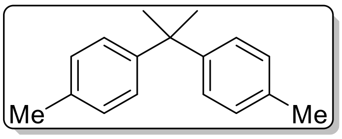
4-(p-tolyl)morpholine (3t)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Colorless oil, 97% yield, 34.4 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.22 (t, *J* = 7.8 Hz, 1H), 6.83 – 6.73 (m, 3H), 3.93 – 3.88 (m, 4H), 3.21 – 3.16 (m, 4H), 2.37 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 151.29 , 138.95 , 129.08 , 121.12 , 116.65 , 112.97 , 66.98 , 49.56 , 21.83 .
HRMS (EI-TOF) calcd for $C_{11}H_{15}NO$: 177.1154; Found：177.1160.
NMR spectroscopic data agreed with literature values.[11]



2-methyldibenzo[b,d]furan (3u)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White solid, 99% yield, 36.1 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.3 Hz, 1H), 7.80 (s, 1H), 7.64 (d, *J* = 8.2 Hz, 1H), 7.57 – 7.47 (m, 2H), 7.40 (t, *J* = 7.0 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 2.58 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 156.54 , 154.61 , 132.23 , 128.29 , 127.00 , 124.33 , 124.26 , 122.59 , 120.71 , 120.62 , 111.71 , 111.21 , 21.42 .
HRMS (EI-TOF) calcd for $C_{13}H_{10}O$: 182.0732; Found：182.0740.
NMR spectroscopic data agreed with literature values.[12]



6-methylbenzo[d]thiazole (3v)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Yellow oil, 99% yield, 29.5 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.92 (s, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 7.75 (s, 1H), 7.34 (d, *J* = 8.4 Hz, 1H), 2.52 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 152.92 , 151.36 , 135.74 , 133.88 , 127.87 , 123.05 , 121.56 , 21.54 .
HRMS (EI-TOF) calcd for C_8H_7NS: 149.0299; Found：149.0305.
NMR spectroscopic data agreed with literature values.[13]



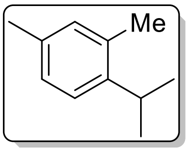
4,4'-(propane-2,2-diyl)bis(methylbenzene) (3w)

Reaction performed according to general procedure; TMB (0.1 mmol instead of 0.5 mmol); purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1).
Colorless oil, 75% yield, 33.7 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31 – 7.16 (m, 8H), 2.44 (s, 6H), 1.79 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 148.02 , 135.06 , 128.80 , 126.78 , 42.35 , 30.97 , 21.03 .

HRMS (EI-TOF) calcd for $C_{17}H_{20}$: 224.1565; Found：224.1571.



1-isopropyl-2,4-dimethylbenzene (3aa)

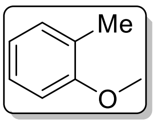
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Colorless oil, 84% yield, 17.8 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.17 (d, *J* = 7.8 Hz, 1H), 7.08 – 6.94 (m, 2H), 3.19 – 3.06 (m, 1H), 2.32 (d, *J* = 8.1 Hz, 6H), 1.24 (d, *J* = 6.9 Hz, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 134.88 , 131.03 , 126.83 , 124.65 , 28.90 , 23.34 , 20.87 , 19.24 .

HRMS (EI-TOF) calcd for $C_{11}H_{16}$: 148.1252; Found：148.1255.

NMR spectroscopic data agreed with literature values.[14]



1-methoxy-2-methylbenzene (3ab)

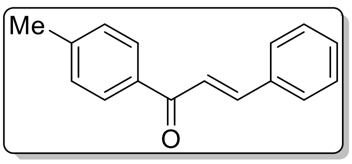
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). Colorless oil, 93% yield, 22.7 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.26 – 7.16 (m, 2H), 6.96 – 6.86 (m, 2H), 3.88 (s, 3H), 2.29 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.75 , 130.64 , 126.83 , 126.62 , 120.29 , 109.91 , 55.25 , 16.26 .

HRMS (EI-TOF) calcd for $C_8H_{10}O$: 122.0732; Found：122.0733.

NMR spectroscopic data agreed with literature values.[5]



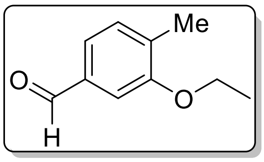
3-phenyl-1-(p-tolyl)prop-2-en-1-one (3ac)

Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Colorless oil, 96% yield, 42.7 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.84 (d, *J* = 15.7 Hz, 1H), 7.66 (dd, *J* = 6.5, 3.0 Hz, 2H), 7.57 (d, *J* = 15.7 Hz, 1H), 7.43 (dd, *J* = 5.0, 1.8 Hz, 3H), 7.32 (d, *J* = 8.0 Hz, 2H), 2.45 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 189.99 , 144.41 , 143.70 , 135.64 , 135.01 , 130.49 , 129.39 , 128.98 , 128.71 , 128.47 , 122.06 , 21.73 .

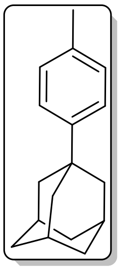
HRMS (EI-TOF) calcd for $C_{16}H_{14}O$: 222.1045; Found：222.1056.
NMR spectroscopic data agreed with literature values.[15]



3-ethoxy-4-methylbenzaldehyde (3ad)

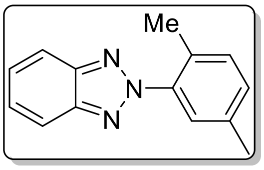
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 30 : 1). Colorless oil, 98% yield, 32.2 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.93 (s, 1H), 7.40 – 7.27 (m, 3H), 4.12 (q, *J* = 7.0 Hz, 2H), 2.31 (s, 3H), 1.47 (t, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.13 , 157.67 , 135.78 , 135.02 , 130.84 , 124.25 , 108.76 , 63.69 , 16.90 , 14.76 .
HRMS (EI-TOF) calcd for $C_{10}H_{12}O_2$: 164.0837; Found：164.0840.
NMR spectroscopic data agreed with literature values.[16]



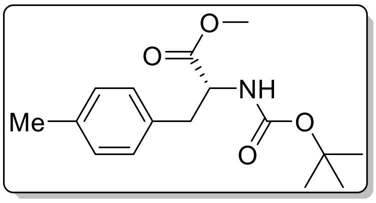
1-(p-tolyl)adamantane (3ae)

Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White solid, 95% yield, 43.0 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.2 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 2.44 (s, 3H), 2.21 (s, 3H), 2.03 (d, *J* = 2.7 Hz, 6H), 1.93 – 1.84 (m, 6H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 148.55 , 134.99 , 128.92 , 124.83 , 43.38 , 36.95 , 35.93 , 29.11 , 21.00 .
HRMS (EI-TOF) calcd for $C_{17}H_{22}$: 226.1722; Found：226.1726.
NMR spectroscopic data agreed with literature values.[17]



2-(2,5-dimethylphenyl)-2*H*-benzo[d][1,2,3]triazole (3af)

Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 20 : 1). White solid, 72% yield, 32.2 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.56 (s, 1H), 7.47 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.25 (d, *J* = 7.9 Hz, 1H), 2.42 (d, *J* = 11.5 Hz, 6H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 144.70 , 140.00 , 136.63 , 131.59 , 130.36 , 130.10 , 126.84 , 126.45 , 118.40 , 20.81 , 18.42 .
HRMS (EI-TOF) calcd for $C_{14}H_{13}N_3$: 223.1109; Found：223.1114.



methyl (*R*)-2-((tert-butoxycarbonyl)amino)-3-(p-tolyl)propanoate (3ag)

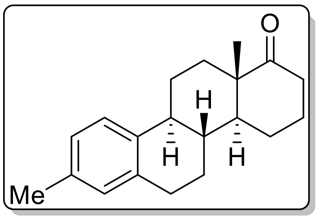
Reaction performed according to general procedure; stirring at room temperature under SO_2F_2 atmosphere for 2h instead of 6h; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). White solid, 55% yield, 32.3 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.11 (d, *J* = 7.8 Hz, 2H), 7.02 (d, *J* = 7.7 Hz, 2H), 5.02 (s, 1H), 4.58 (s, 1H), 3.72 (s, 3H), 3.06 (dd, *J* = 12.3, 5.8 Hz, 2H), 2.33 (s, 3H), 1.44 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.44 , 155.14 , 136.56 , 132.86 , 129.16 , 79.83 , 67.07 , 54.49 , 52.16 , 37.84 , 28.30 , 21.06 .

HRMS (EI-TOF) calcd for $C_{16}H_{23}NO_4$: 293.1627; Found：293.1699.

NMR spectroscopic data agreed with literature values.[18]



(*4aS,4bR,10bS,12aS*)-8,12a-dimethyl-3,4,4a,4b,5,6,10b,11,12,12a-decahydrochrysen-1(2*H*)-one (3ah)

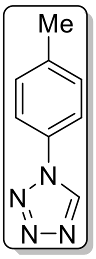
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 5 : 1). White solid, 81% yield, 45.8 mg.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.24 (d, *J* = 7.9 Hz, 1H), 7.02 (d, *J* = 8.0 Hz, 1H), 6.98 (s, 1H), 2.98 – 2.88 (m, 2H), 2.58 – 2.42 (m, 2H), 2.34 (s, 3H), 2.29 (s, 1H), 2.24 – 2.15 (m, 1H), 2.15 – 1.90 (m, 4H), 1.75 – 1.34 (m, 7H), 0.95 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 136.75 , 136.32 , 135.33 , 129.76 , 126.59 , 125.31 , 50.51 , 48.06 , 44.28 , 38.32 , 35.92 , 31.64 , 29.38 , 26.61 , 25.83 , 21.63 , 20.89 , 13.88 .

HRMS (EI-TOF) calcd for $C_{20}H_{26}O$: 282.1984; Found：282.2056.

NMR spectroscopic data agreed with literature values.[8]



1-(p-tolyl)-1*H*-tetrazole (3ai)

Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 3 : 1). Pink solid, 67% yield, 21.5 mg.

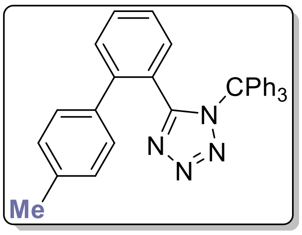
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.99 (s, 1H), 7.60 (d, *J* = 8.5 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 2.47 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 140.49 (d, *J* = 5.7 Hz), 131.47 , 130.70 , 121.14 , 21.22 .

HRMS (EI-TOF) calcd for $C_8H_8N_4$: 160.0749; Found：160.0755.

NMR spectroscopic data agreed with literature values.[19]

5-(4'-methyl-[1,1'-biphenyl]-2-yl)-1-trityl-1*H*-tetrazole (3aj)



Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Wheat solid, 84% yield, 80.4 mg.

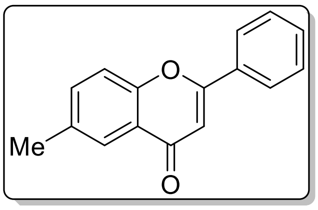
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.5 Hz, 1H), 7.59 – 7.49 (m, 3H), 7.45 – 7.40 (m, 3H), 7.36 (t, *J* = 7.7 Hz, 6H), 7.15 (d, *J* = 7.9 Hz, 2H), 7.06 (d, *J* = 7.9 Hz, 8H), 2.40 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 164.34 , 142.42 , 141.41 , 138.31 , 136.46 , 130.82 , 130.39 (d, *J* = 3.8 Hz), 130.03 , 129.24 , 128.75 , 128.32 , 127.73 , 127.40 , 126.57 , 83.00 , 21.34 .

HRMS (EI-TOF) calcd for $C_{33}H_{26}N_4$: 478.2157; Found： 478.2214.

NMR spectroscopic data agreed with literature values.[20]

6-methyl-2-phenyl-4*H*-chromen-4-one (3ak)



Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 5 : 1). Wheat solid, 91% yield, 43.0 mg.

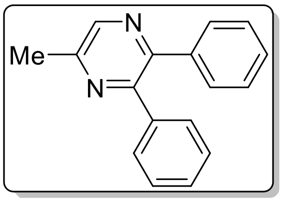
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 (s, 1H), 8.00 – 7.91 (m, 2H), 7.65 – 7.45 (m, 5H), 6.85 (s, 1H), 2.50 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 178.63 , 163.36 , 154.59 , 135.27 , 135.05 , 131.93 , 131.55 , 129.05 , 126.31 , 125.08 , 123.61 , 117.87 , 107.45 , 20.97 .

HRMS (EI-TOF) calcd for $C_{16}H_{12}O_2$: 236.0837; Found： 236.0840.

NMR spectroscopic data agreed with literature values.[21]

5-methyl-2,3-diphenylpyrazine (3al)



Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Yellow solid, 82% yield, 40.4 mg.

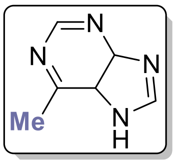
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.51 (s, 1H), 7.46 (ddt, *J* = 7.6, 4.6, 2.5 Hz, 4H), 7.31 (td, *J* = 5.3, 4.8, 1.9 Hz, 6H), 2.68 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 151.62 , 151.22 , 149.76 , 141.91 , 138.79 (d, *J* = 13.2 Hz), 129.68 (d, *J* = 7.8 Hz), 128.62 – 128.14 (m), 21.40 .

HRMS (EI-TOF) calcd for $C_{17}H_{14}O_2$: 246.1157; Found： 246.1206.

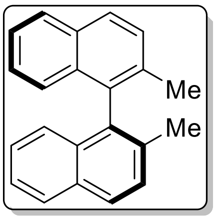
NMR spectroscopic data agreed with literature values.[22]

6-methyl-4,5-dihydro-7*H*-purine (3am)

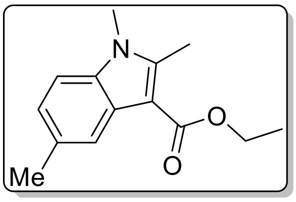


Reaction performed according to general procedure; stirring at room temperature under SO_2F_2 atmosphere for 2h instead of 6h; purified by

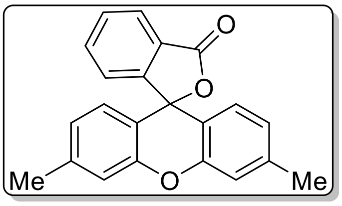
chromatography on silica gel (Petroleum ether : Ethyl acetate = 1 : 1). Wheat solid, 74% yield, 20.2 mg.
^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 13.39 (s, 1H), 8.74 (s, 1H), 8.51 (s, 1H), 3.40 (s, 1H), 2.70 (s, 3H).
HRMS (EI-TOF) calcd for $C_6H_8N_4$: 136.0749; Found：136.0795.
NMR spectroscopic data agreed with literature values.[23]



2,2'-dimethyl-1,1'-binaphthalene (3an)
Reaction performed according to general procedure; TMB (0.1 mmol instead of 0.5 mmol); purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Colorless oil, 67% yield, 37.8 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.96 (m, 4H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.29 (d, *J* = 6.5 Hz, 2H), 7.18 (d, *J* = 8.5 Hz, 2H), 2.15 (s, 6H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 135.24 , 134.39 , 132.87 , 132.33 , 128.85 , 128.06 , 127.56 , 126.22 , 125.75 , 125.02 , 20.17 .
HRMS (EI-TOF) calcd for $C_{22}H_{18}$: 282.1409; Found：282.1469.
NMR spectroscopic data agreed with literature values.[24]

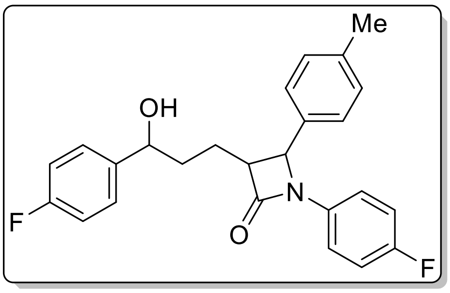


ethyl 1,2,5-trimethyl-1*H*-indole-3-carboxylate (3ao)
Reaction performed according to general procedure; purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Yellow solid, 82% yield, 37.9 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (s, 1H), 7.14 (d, *J* = 8.3 Hz, 1H), 7.07 (d, *J* = 8.3 Hz, 1H), 4.45 (q, *J* = 7.1 Hz, 2H), 3.56 (s, 3H), 2.71 (s, 3H), 2.54 (s, 3H), 1.51 (t, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 166.27 , 145.14 , 134.91 , 130.91 , 126.87 , 123.36 , 121.20 , 108.72 , 103.34 , 59.30 , 29.45 , 21.70 , 14.72 , 11.85 .
HRMS (EI-TOF) calcd for $C_{14}H_{17}NO_2$: 231.1259; Found：232.1332.
NMR spectroscopic data agreed with literature values.[25]



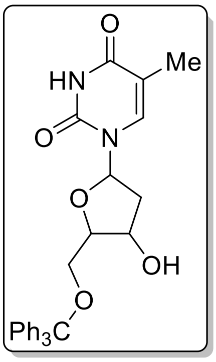
3',6'-dimethyl-3*H*-spiro[isobenzofuran-1,9'-xanthen]-3-one (3ap)
Reaction performed according to general procedure; TMB (0.1 mmol instead of 0.5 mmol); purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 10 : 1). Yellow solid, 90% yield, 59.1 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 (d, *J* = 7.0 Hz, 1H), 7.65 (dt, *J* = 15.2, 7.0 Hz, 2H), 7.16 (d, *J* = 7.2 Hz, 1H), 7.12 (s, 2H), 6.86 (d, *J* = 7.5 Hz, 2H), 6.72 (d, *J* = 8.1 Hz, 2H), 2.40 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.60 , 153.64 , 151.17 , 141.18 , 135.01 , 129.64 , 127.77 , 126.44 , 125.05 , 124.76 , 123.86 , 117.28 , 116.01 , 21.31 .
HRMS (EI-TOF) calcd for $C_{22}H_{16}O_3$: 328.3670; Found：328.2170.
NMR spectroscopic data agreed with literature values.[26]



1-(4-fluorophenyl)-3-(3-(4-fluorophenyl)-3-hydroxypropyl)-4-(p-tolyl)azetidin-2-one (3aq)

Reaction performed according to general procedure; stirring at room temperature under SO_2F_2 atmosphere for 1h instead of 6h; TMB (0.1 mmol instead of 0.5 mmol); purified by chromatography on silica gel (Petroleum ether : Ethyl acetate = 3 : 1). Colorless oil, 75% yield, 61.1 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.29 (dd, *J* = 8.6, 5.5 Hz, 2H), 7.26 – 7.16 (m, 6H), 7.04 – 6.96 (m, 2H), 6.92 (t, *J* = 8.7 Hz, 2H), 4.70 (t, *J* = 5.9 Hz, 1H), 4.61 (d, *J* = 2.2 Hz, 1H), 3.10 – 3.05 (m, 1H), 2.36 (s, 3H), 1.94 (dd, *J* = 20.3, 6.7 Hz, 4H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.30 , 167.77 , 163.34 , 160.90 , 160.20 , 157.78 , 140.27 , 138.54 , 134.46 , 133.89 (d, *J* = 2.5 Hz), 129.94 , 127.38 , 125.83 , 118.43 (d, *J* = 7.8 Hz), 115.91 , 115.69 , 115.36 , 115.14 , 72.94 , 61.31 , 60.46 , 60.29 , 36.64 , 25.03 , 21.16 , 21.02 , 14.18 .
^{19}F NMR (377 MHz, Chloroform-*d*) δ -115.08 , -117.96 .
HRMS (EI-TOF) calcd for $C_{25}H_{25}F_2NO_2$: 407.1697; Found：407.1801.



1-(4-hydroxy-5-((trityloxy)methyl)tetrahydrofuran-2-yl)-5-methylpyrimidine-2,4(1*H*,3*H*)-dione (3ar)

Reaction performed according to general procedure; stirring at room temperature under SO_2F_2 atmosphere for 1h instead of 6h; purified by chromatography on silica gel (Ethyl acetate). Wheat solid, 46% yield, 44.6 mg.
^{1}H NMR (400 MHz, Chloroform-*d*) δ 11.36 (s, 1H), 7.52 (s, 1H), 7.41 (d, *J* = 7.5 Hz, 6H), 7.34 (t, *J* = 7.6 Hz, 6H), 7.26 (t, *J* = 7.1 Hz, 3H), 6.25 (t, *J* = 6.8 Hz, 1H), 5.37 (d, *J* = 4.4 Hz, 1H), 4.43 – 4.32 (m, 1H), 3.92 (d, *J* = 3.4 Hz, 1H), 3.31 – 3.17 (m, 2H), 2.34 – 2.12 (m, 2H), 1.48 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.72 , 150.43 , 143.52 , 135.71 , 128.30 , 128.00 , 127.19 , 109.64 , 86.45 , 85.45 , 83.81 , 70.52 , 64.02 , 54.91 , 11.81 .
HRMS (EI-TOF) calcd for $C_{29}H_{28}N_2O_5$: 484.1998; Found：484.2053.

reference

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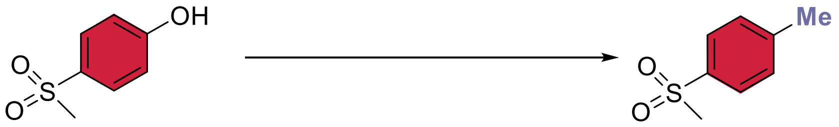
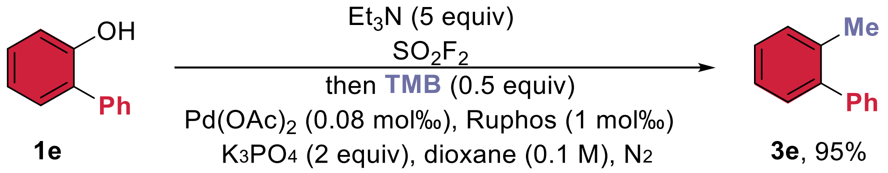
4: Gram-Scale Reaction



1j, 1.032 g **3j**, 0.6 g (88%)

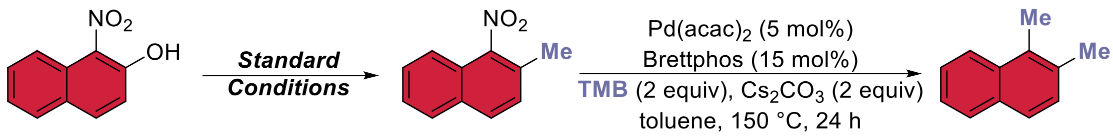
A 250mL Schlenk flask equipped with a stirring bar is filled with 4-(methylsulfonyl)phenol **1j** (4.0 mmol, 1.0 equiv.), Et_3N (20.0 mmol, 5.0 equiv.), dioxane (20 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 12h. Then addition $Pd(OAc)_2$ (5 mol%), RuPhos (6 mol%), K_3PO_4 (8.0 mmol, 2.0 equiv.), trimethylboroxine (2.0 mmol, 0.5 equiv.), dioxane (20 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 8 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum (PE:EA=10:1) as eluent to provide the desired product **3j**. 0.6g, 88% yield.

5: Experiments for parts per million (ppm) level catalysts



A 25mL Schlenk flask equipped with a stirring bar is filled with [1,1'-biphenyl]-2-ol **1e** (0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 2h. Then addition $Pd(OAc)_2$ (0.08 mol‰), RuPhos (1 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), trimethylboroxine (0.1 mmol, 0.5 equiv.), dioxane (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 6 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum (PE:EA=20:1) as eluent to provide the desired product **3e**. 95% yield.

6. Experiments for Iterative Reaction

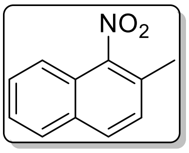


4 5 6

A 25mL Schlenk flask equipped with a stirring bar is filled with 1-nitronaphthalen-2-ol **4** (0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 2h. Then addition $Pd(OAc)_2$ (5 mol%), RuPhos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), trimethylboroxine (0.1 mmol, 0.5 equiv.), dioxane (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 2 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum (PE:EA=10:1) as eluent to provide the desired product **5**. 83% yield.

Next, a 25mL Schlenk flask equipped with a stirring bar is filled with 2-methyl-1-nitronaphthalene **5** (0.2 mmol, 1.0 equiv.), $Pd(acac)_2$ (5 mol%), BrettPhos (15 mol%), Cs_2CO_3 (0.4 mmol, 2.0 equiv.), trimethylboroxine (0.4 mmol, 2.0 equiv.), toluene (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 24 h at 150 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The solvent was removed under vacuum and the residue was purified by a flash column chromatography on silica gel using ethyl acetate and petroleum (PE:EA=20:1) as eluent to provide the desired product **6**.[27] 54% yield.

27. B. Y. Feng, Y. D. Yang and J. S. You, *Chem. Sci.*, 2020, **11**, 6031-6035.



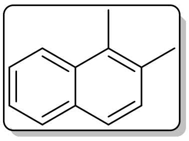
2-methyl-1-nitronaphthalene (5)

Yellow solid, 83% yield, 31.0 mg.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.89 (d, J = 8.3 Hz, 2H), 7.74 (d, J = 8.5 Hz, 1H), 7.62 (t, J = 7.7 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.37 (d, J = 8.4 Hz, 1H), 2.53 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 132.26 , 130.44 , 128.52 , 128.04 (d, J = 14.9 Hz), 127.53 , 126.65 , 124.69 , 121.25 , 17.85 .

HRMS (EI-TOF) calcd for $C_{11}H_9NO_2$: 187.0633; Found： 187.0640.



1,2-dimethylnaphthalene (6)

Colorless oil, 54% yield, 16.8 mg.

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.15 (d, *J* = 8.5 Hz, 1H), 7.91 (d, *J* = 8.1 Hz, 1H), 7.73 (d, *J* = 8.3 Hz, 1H), 7.60 (t, *J* = 7.0 Hz, 1H), 7.53 (t, *J* = 7.9 Hz, 1H), 7.41 (d, *J* = 8.3 Hz, 1H), 2.71 (s, 3H), 2.60 (s, 3H).

^{13}C NMR (101 MHz, DMSO-d_6) δ 133.08 , 132.83 , 132.26 , 131.09 , 128.99 , 128.40 , 125.69 (d, *J* = 1.0 Hz), 124.44 , 123.69 , 20.74 , 14.45 .

HRMS (EI-TOF) calcd for $C_{12}H_{12}$: 156.0939; Found： 156.0944.

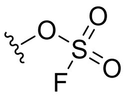
7: Mechanistic Studies

7.1:

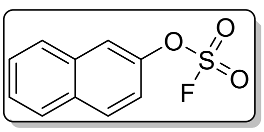
Table S2: Control experiments.[a]

D.

1f **3f**

Entry	X	Deviation	Yield 3f (%)[b]
1	OH	-	99
2	OH	w / o $Pd(OAc)_2$	0
3	OH	w / o Ruphos	0
4	OH	w / o K_3PO_4	28
5[c]	OH	w / BHT	99
6[d]	 (2f)	-	99

[a] Reaction conditions: **1f** (0.2 mmol), Et_3N (1.0 mmol, 5.0 eq.), dioxane (0.2 M), r.t., 2 h, under SO_2F_2 gas atmosphere; then addition $Pd(OAc)_2$ (5 mol%), Ruphos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), TMB (0.1 mmol, 0.5 eq.), dioxane (0.2 M), 120 ºC, 2 h, under nitrogen atmosphere. [b] Isolated yield. [c] After completion of the reaction at room temperature addition Butylated hydroxytoluene (BHT) (0.4 mmol, 2.0 eq.). [d] **2f** (0.2 mmol), $Pd(OAc)_2$ (5 mol%), Ruphos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), TMB (0.1 mmol, 0.5 eq.), dioxane (0.2 M), 120 ºC, 2 h, under nitrogen atmosphere.

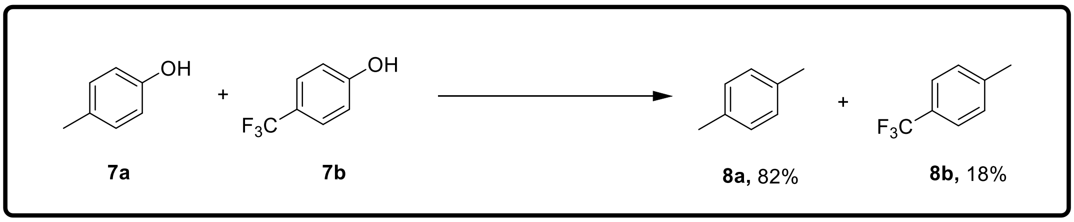


naphthalen-2-yl sulfurofluoridate (2f)

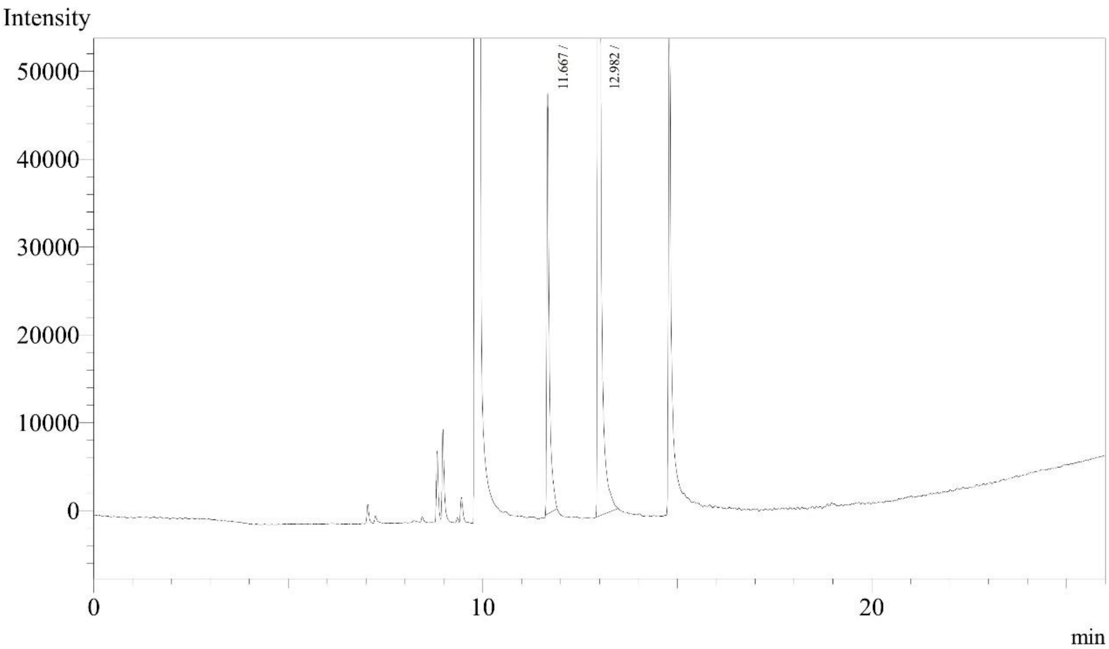
A 25mL Schlenk flask equipped with a stirring bar is filled with Aryl phenols (0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 2h. Yellow solid.

^{19}F NMR (377 MHz, Chloroform-*d*) δ 37.75 .

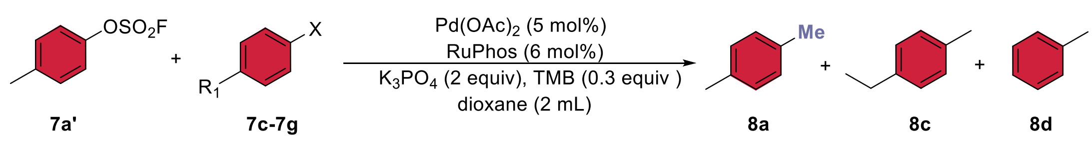
7.2: Competition experiments.



A 25mL Schlenk flask equipped with a stirring bar is filled with p-cresol **7a** (0.2 mmol, 1.0 equiv.), 4-(trifluoromethyl)phenol **7b** (0.2 mmol, 1.0 equiv.), Et_3N (1.0 mmol, 5.0 equiv.), dioxane (1 mL). Under positive pressure of SO_2F_2 gas, five evacuation/backfill cycles are performed under high vacuum to fill the reaction tube and vigorous stirring at room temperature for 2h. Then addition $Pd(OAc)_2$ (5 mol%), RuPhos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), trimethylboroxine (0.67 mmol.), dioxane (1 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 2 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times. GC yield: **8a** (82%); **8b** (18%).



Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	ID#	Cmpd Name
1	11.667	212984	47583	17.807				
2	12.982	983114	235605	82.193				
Total		1196098	283188					

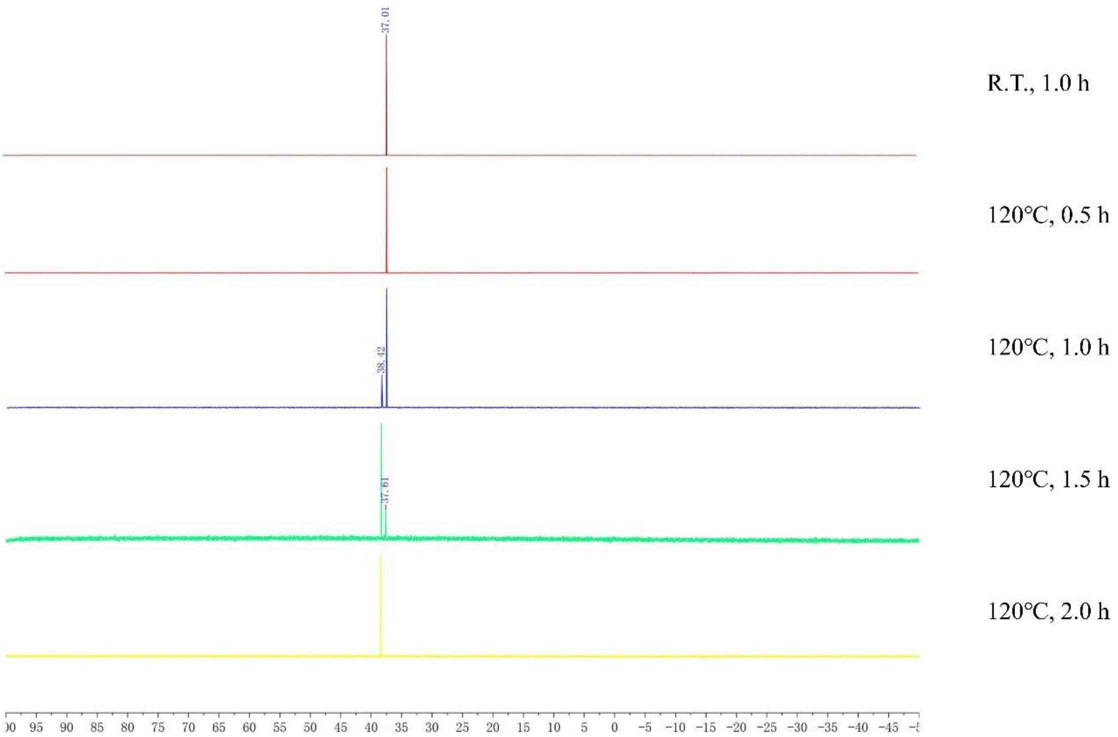


Entry	X	R_1	Yield 8a：8c：8d (%)[b]
1	Br (**7c**)	Et	33:67:0
2	Cl (**7d**)	Et	81:19:0
3	OTf (**7e**)	H	21:0:79
4	OTs (**7f**)	H	17:0:83
5	OMs (**7g**)	H	77:0:23
6[c]	OPFBs (**7h**)	H	47:0:53

[a] Reaction conditions: **7a'** (0.2 mmol), $Pd(OAc)_2$ (5 mol%), Ruphos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), TMB (0.1 mmol, 0.5 eq.), dioxane (0.1 M), 120 ºC, 2 h, under nitrogen atmosphere. [b] Yields determined by high performance liquid chromatography. [c] OPFBs = 2,3,4,5,6-pentafluorobenzenesulfonate.

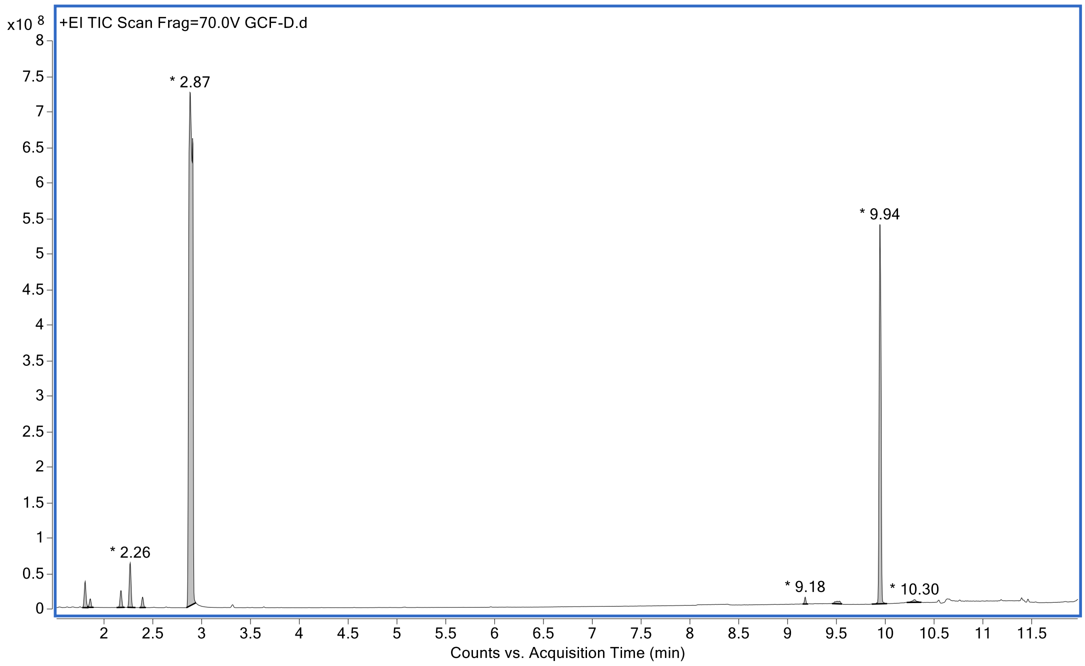
A 25mL Schlenk flask equipped with a stirring bar is filled with **7a'** (0.2 mmol, 1.0 equiv.), **7c** (or **7d-7h**) (0.2 mmol, 1.0 equiv.), $Pd(OAc)_2$ (5 mol%), RuPhos (6 mol%), K_3PO_4 (0.4 mmol, 2.0 equiv.), trimethylboroxine (0.67 mmol.), dioxane (2 mL). Under a positive pressure of nitrogen and five evacuations/backfilling cycles under high vacuum. The mixture was allowed to react for 2 h at 120 ºC. The reaction was quenched by water and the reaction mixture was extracted with EA for three times.

7.3: Kinetic experments.

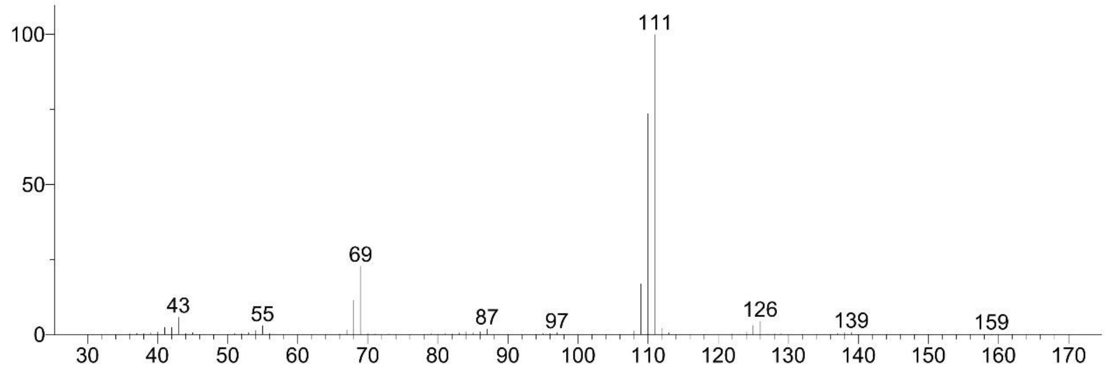


Scheme S1 ^{19}F NMR under different reaction times.

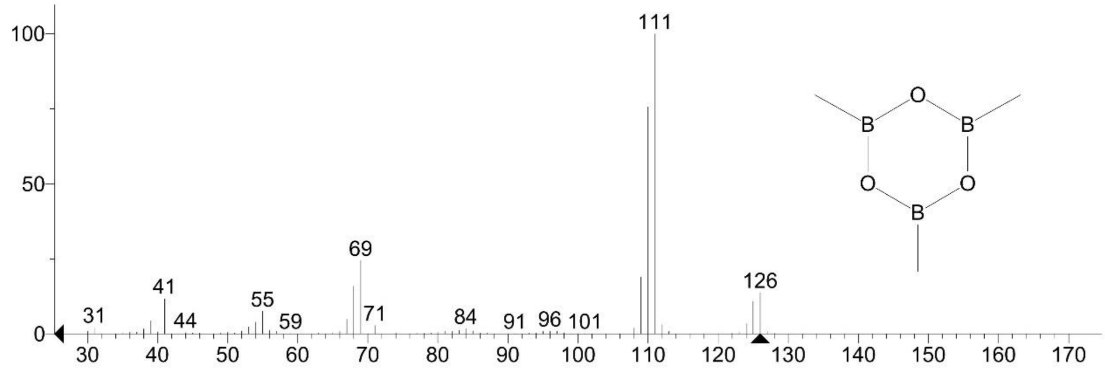
7.4: GC-MS analysis of 3c.



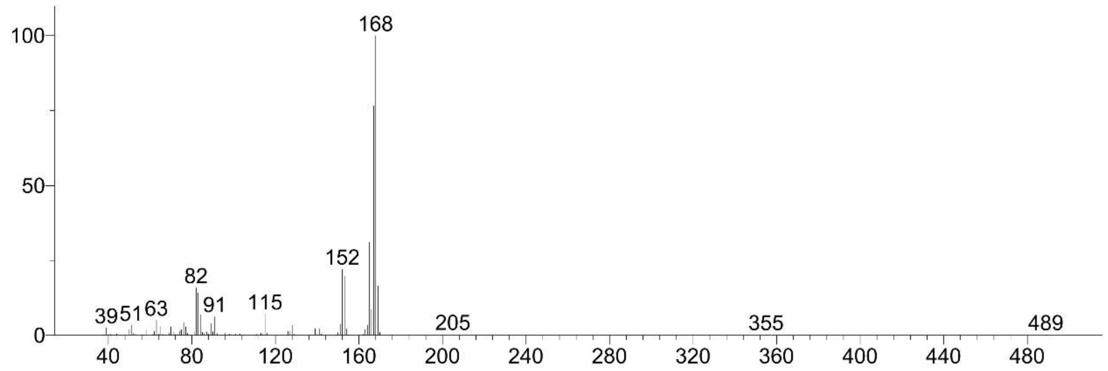
Unknown: +EI Scan (rt: 2.16-2.17 min, 3 scans) Frag=70.0V GCF-D.D Subtract
Compound in Library Factor = 196



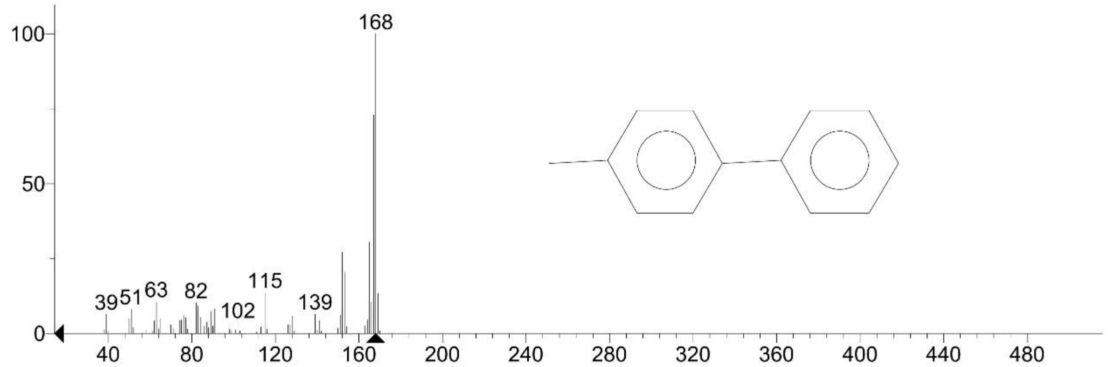
Hit 1 : Trimethylboroxine
C3H9B3O3; MF: 852; RMF: 867; Prob 92.7%; CAS: 823-96-1; Lib: mainlib; ID: 109260.



Unknown: +EI Scan (rt: 9.92-9.93 min, 3 scans) Frag=70.0V GCF-D.D Subtract
Compound in Library Factor = 133

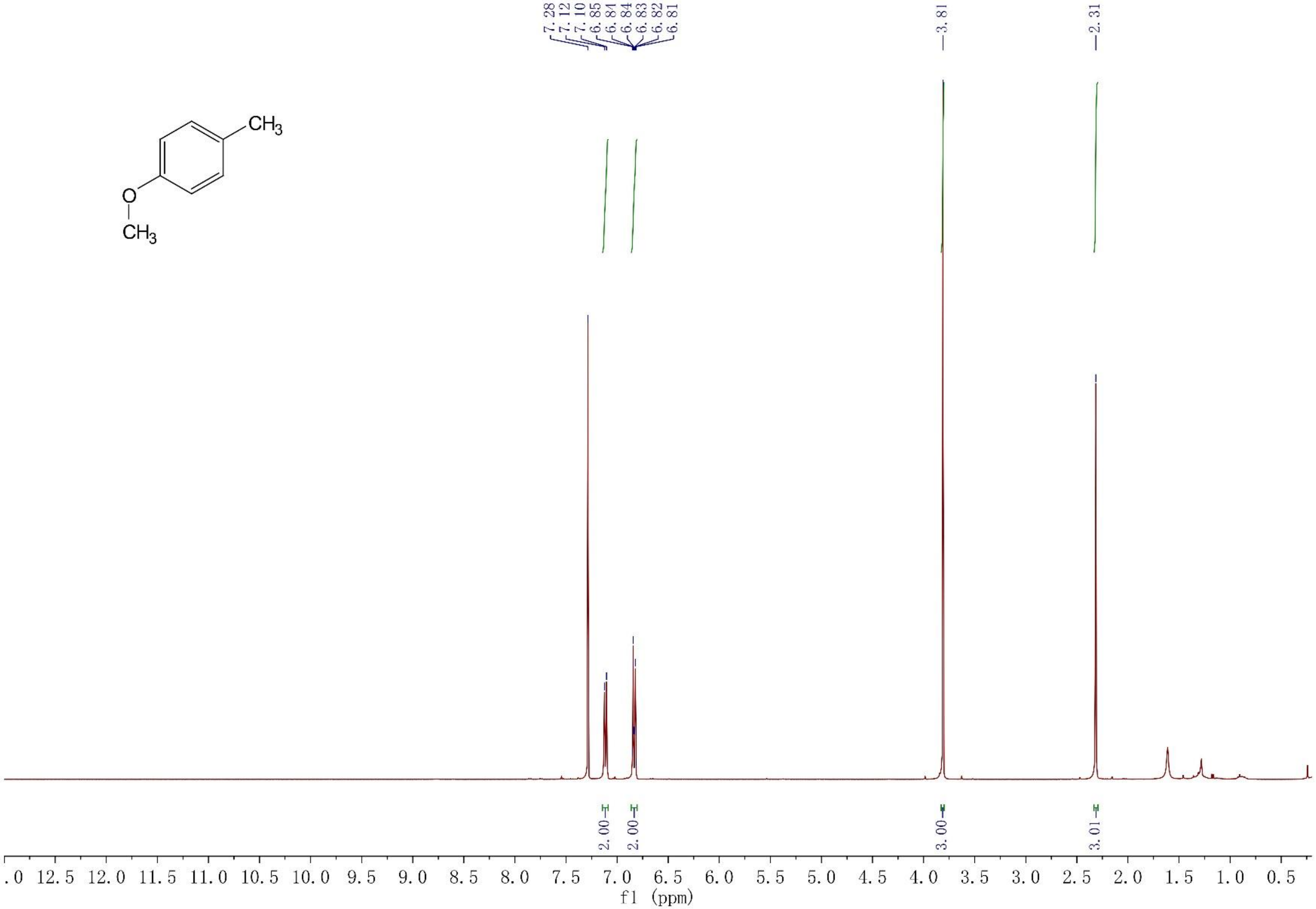


Hit 1 : 1,1'-Biphenyl, 4-methyl-
C13H12; MF: 928; RMF: 934; Prob 31.9%; CAS: 644-08-6; Lib: mainlib; ID: 195914.

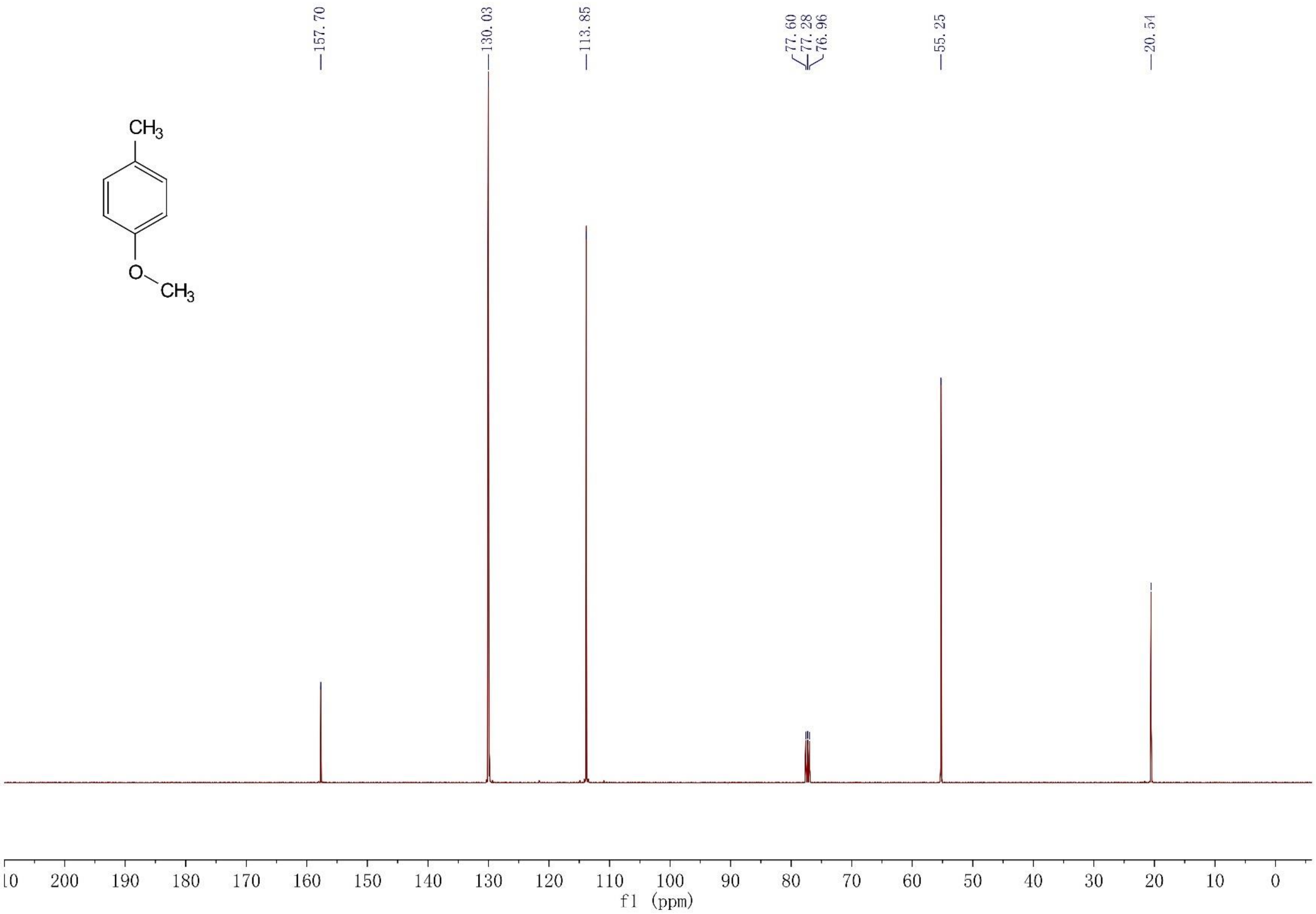


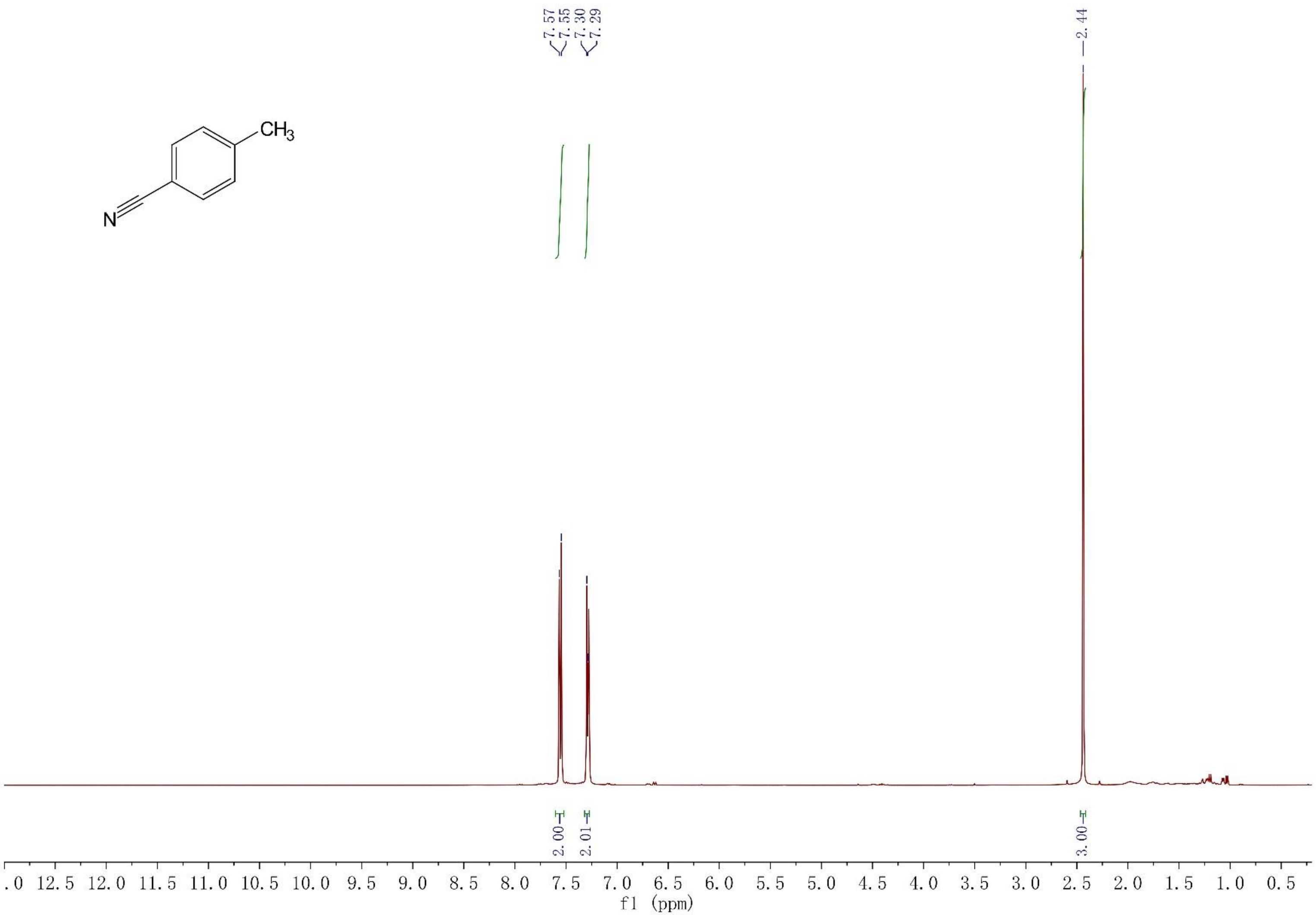
Scheme S2 GC-MS of the 2h reaction system.

8: Copies of ^{1}H, ^{13}C and ^{19}F spectra for products



7.28
7.12
7.10
6.85
6.84
6.84
6.83
6.82
6.81
3.81
2.31
CH3
O
CH3
2.00
2.00
3.00
3.01
.0 12.5 12.0 11.5 11.0 10.5 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
f1 (ppm)


157.70
130.03
113.85
77.60
77.28
76.96
55.25
20.54
CH3
O
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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N
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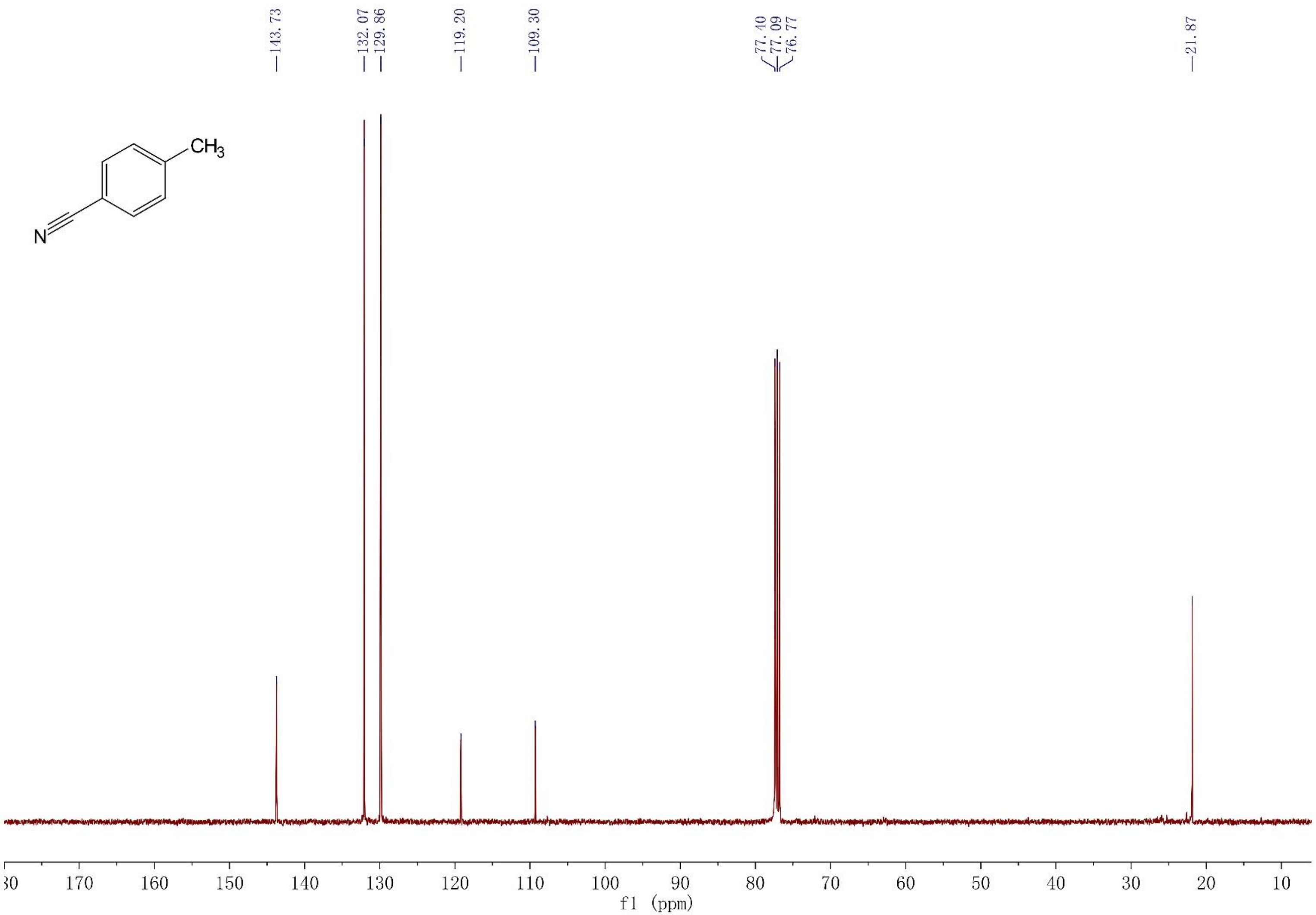
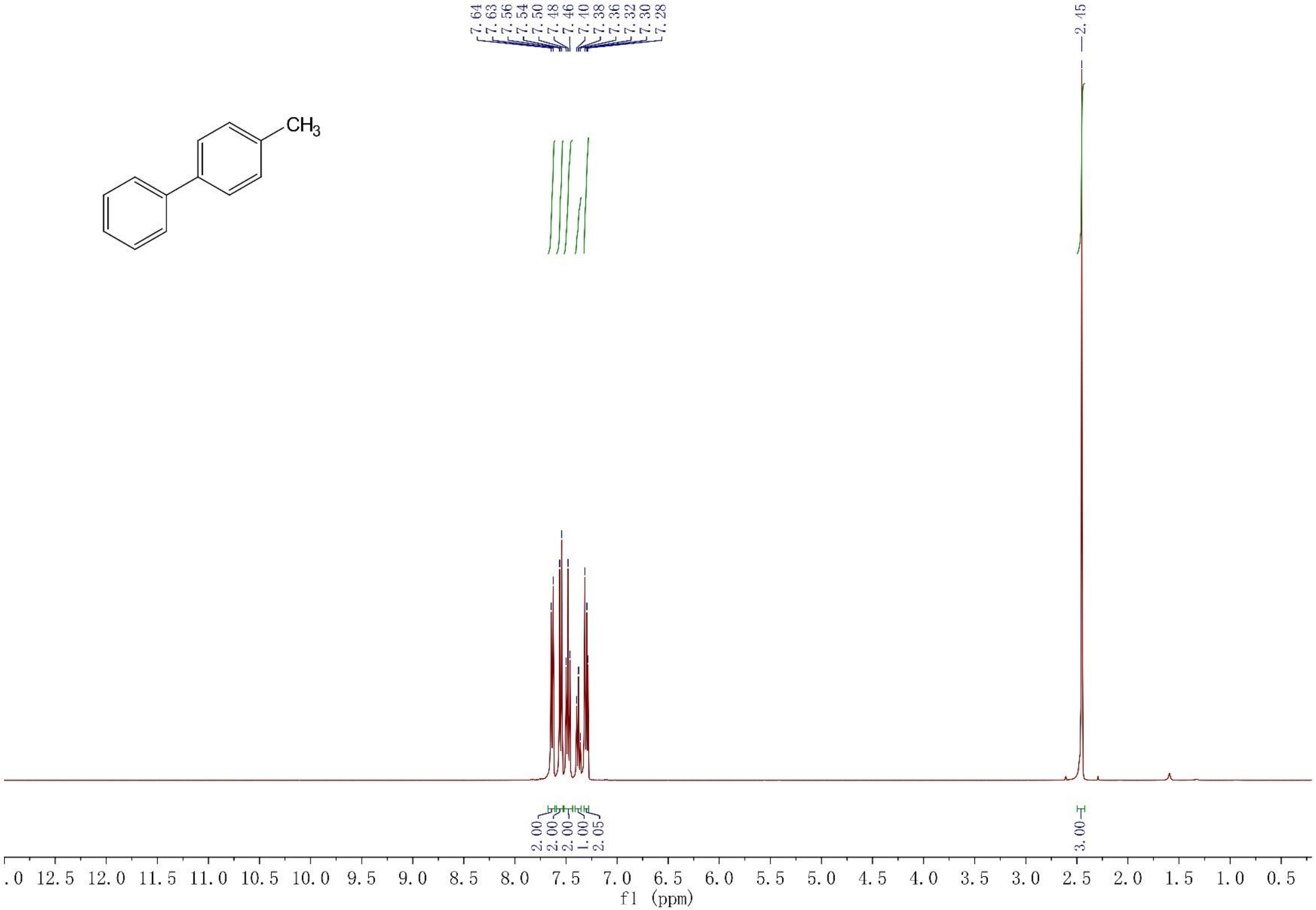
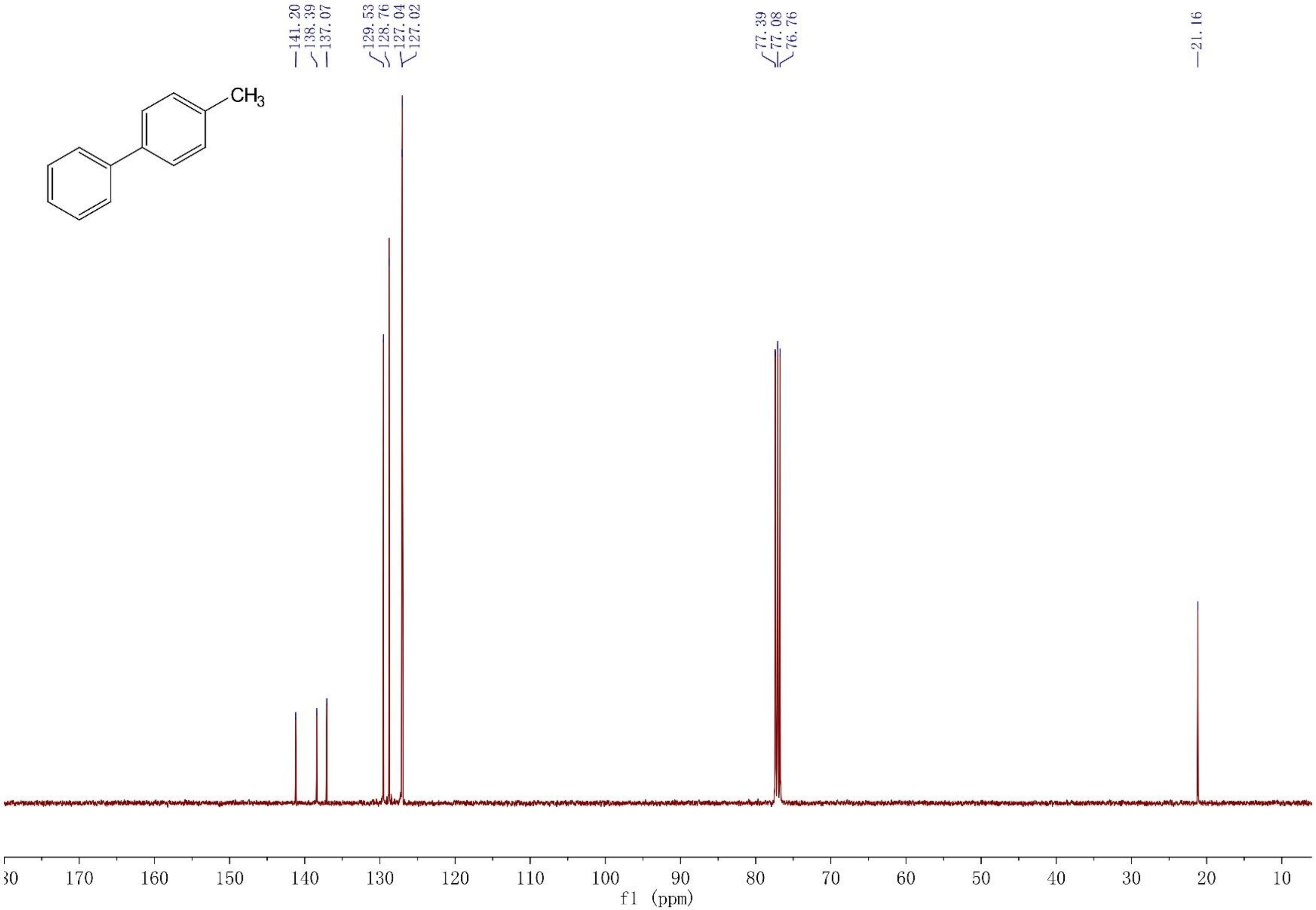
7.55
7.30
7.29
2.44
2.00
2.01
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

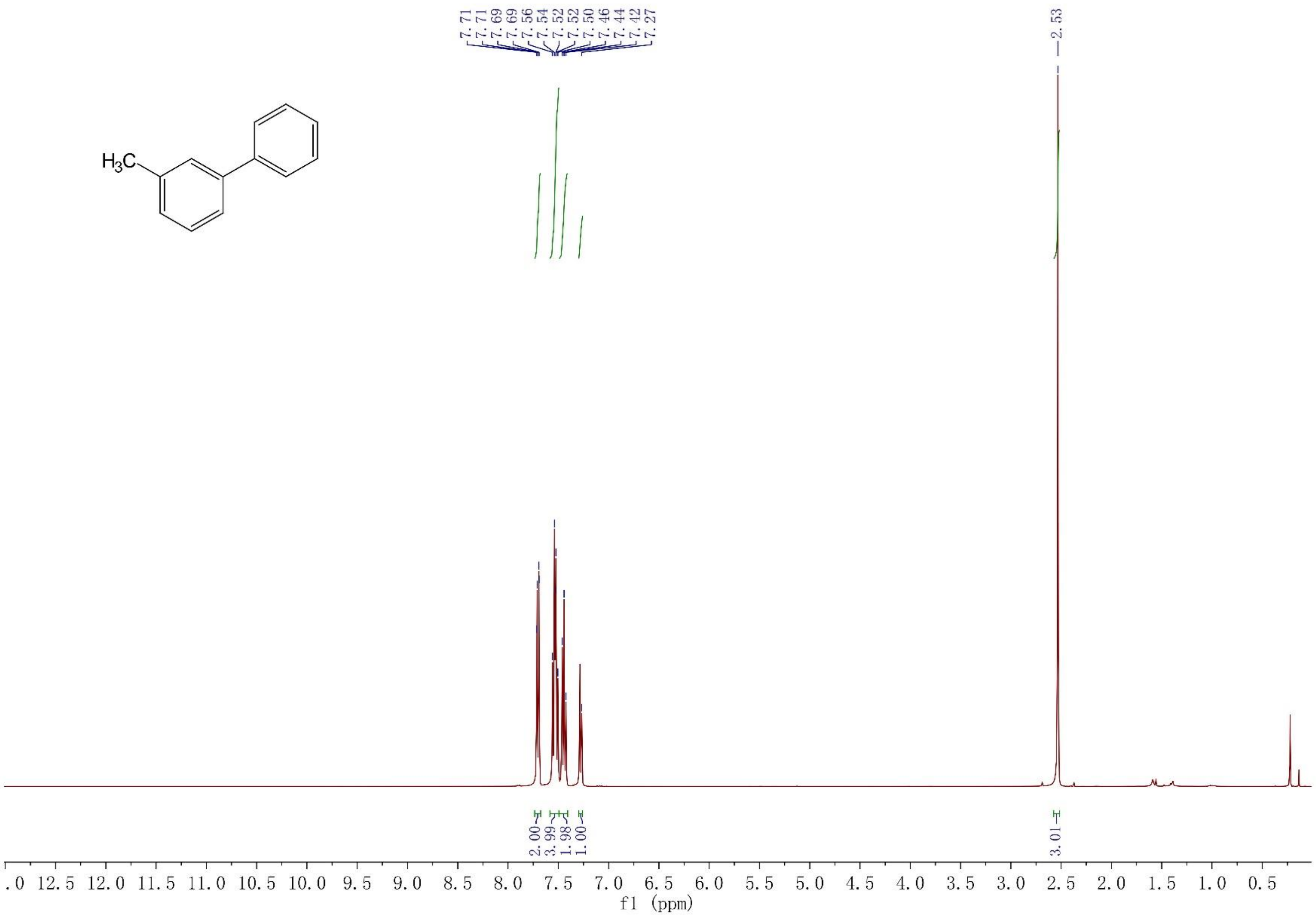

CH3
N
143.73
132.07
129.86
119.20
109.30
77.40
77.09
76.77
21.87
30
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



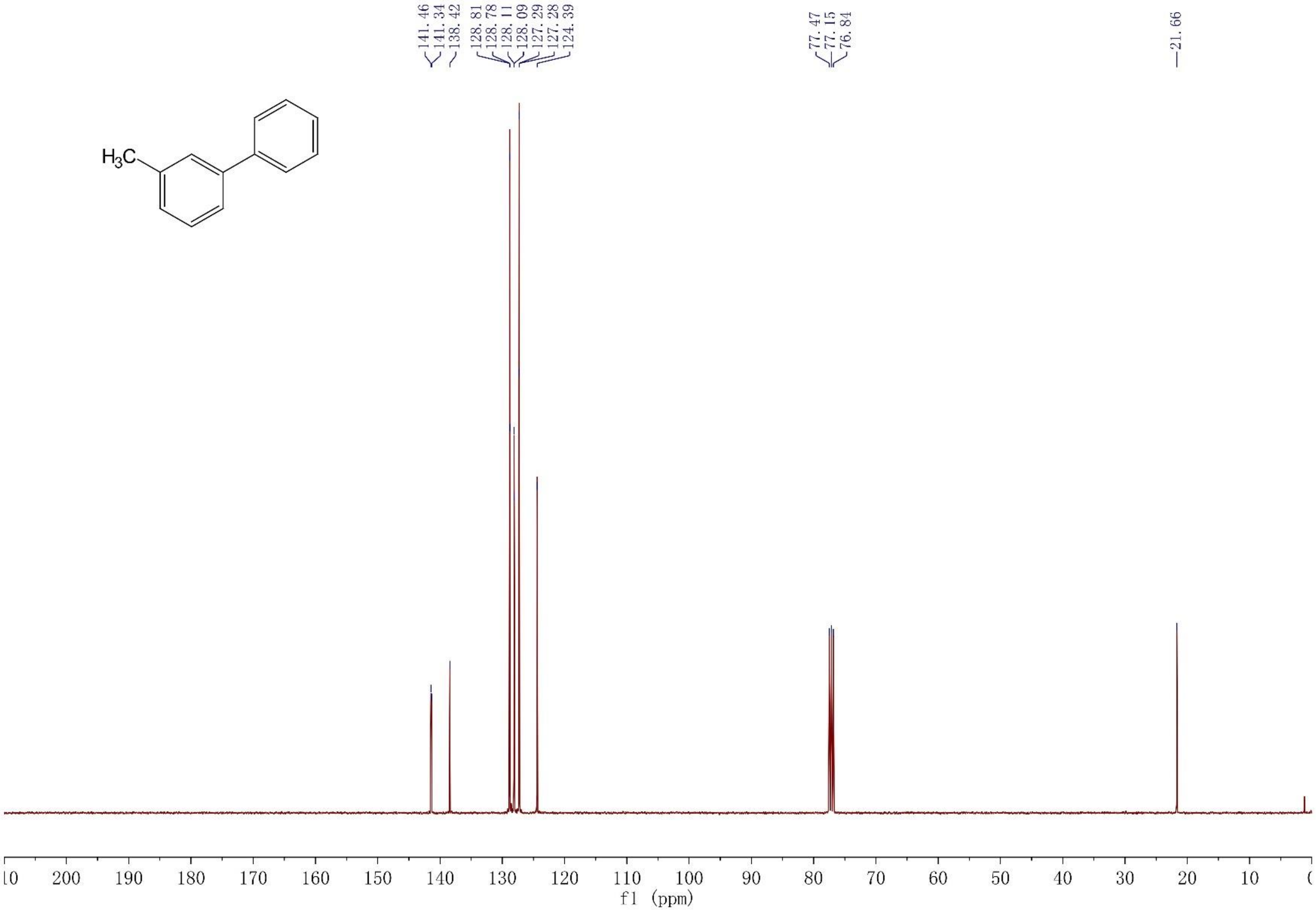
CH3
7.64
7.63
7.56
7.54
7.50
7.48
7.46
7.40
7.38
7.36
7.32
7.30
7.28
2.45
2.00
2.00
2.00
1.00
2.05
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

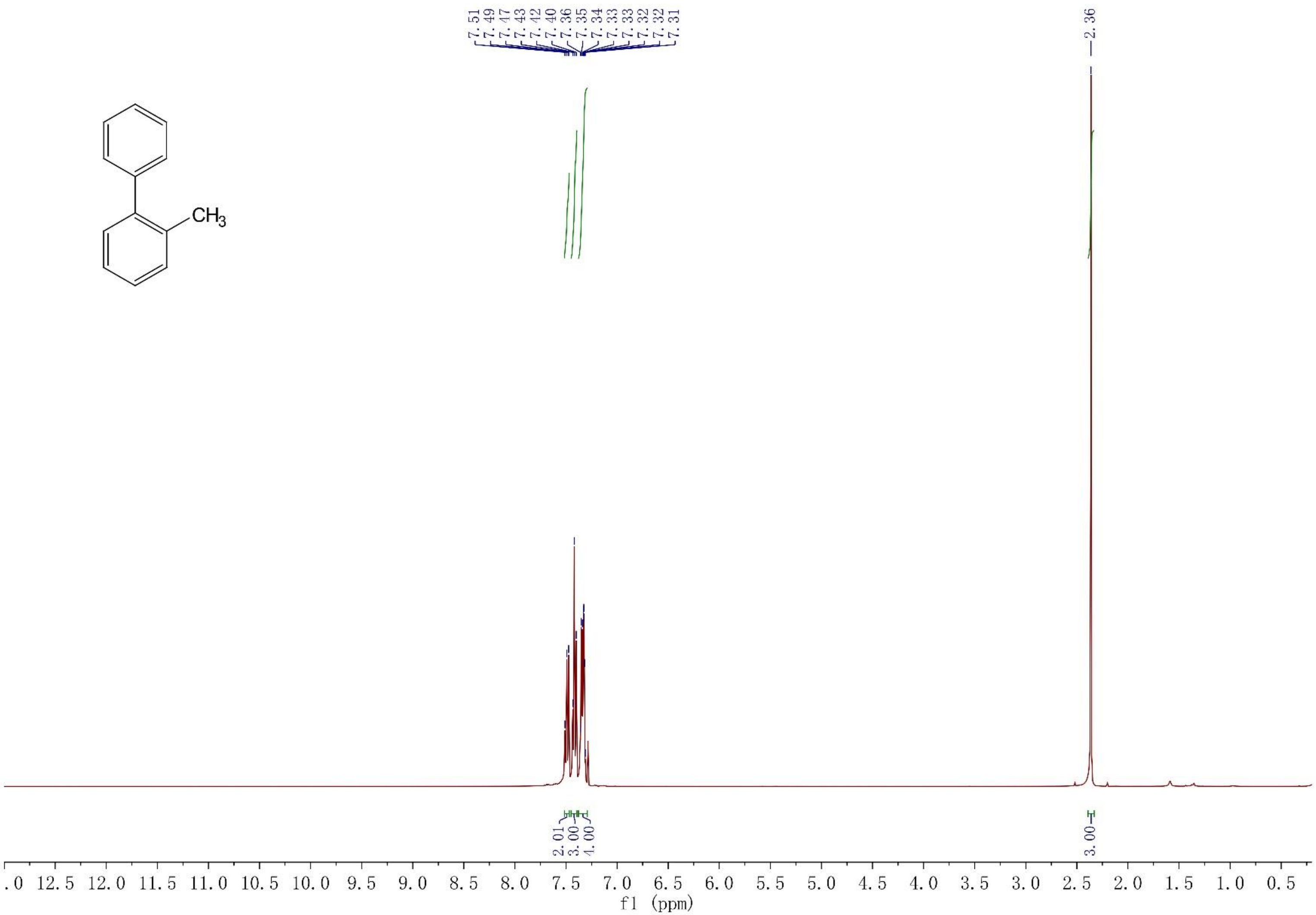


CH3
141.20
138.39
137.07
129.53
128.76
127.04
127.02
77.39
77.08
76.76
21.16
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



H3C
7.71
7.71
7.69
7.69
7.56
7.54
7.52
7.52
7.50
7.46
7.44
7.42
7.27
2.53
2.00
3.99
1.98
1.00
3.01
.0 12.5 12.0 11.5 11.0 10.5 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
f1 (ppm)


141.46
141.34
138.42
128.81
128.78
128.11
128.09
127.29
127.28
124.39
77.47
77.15
76.84
21.66
H_3C
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



CH3
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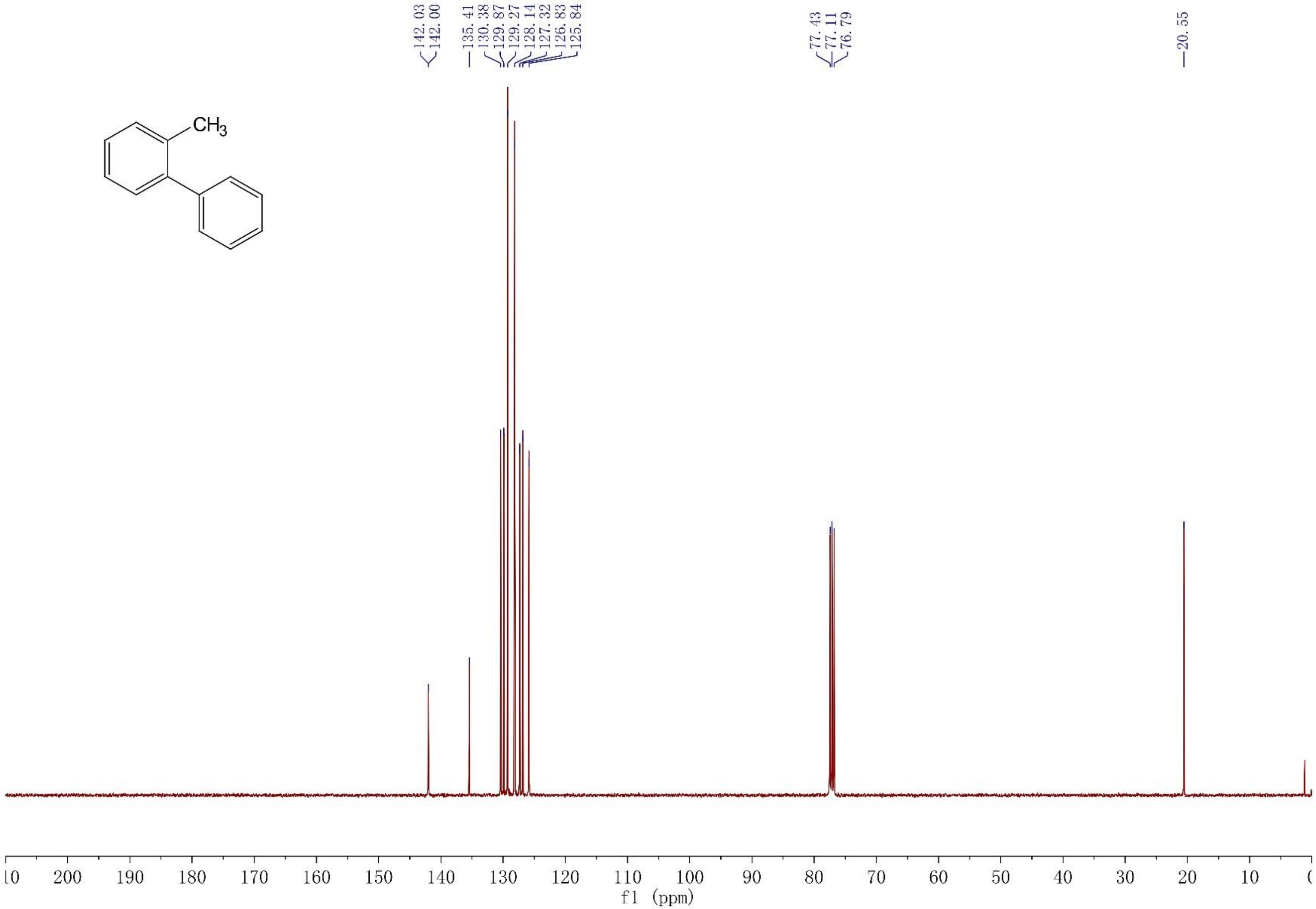
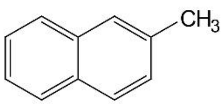
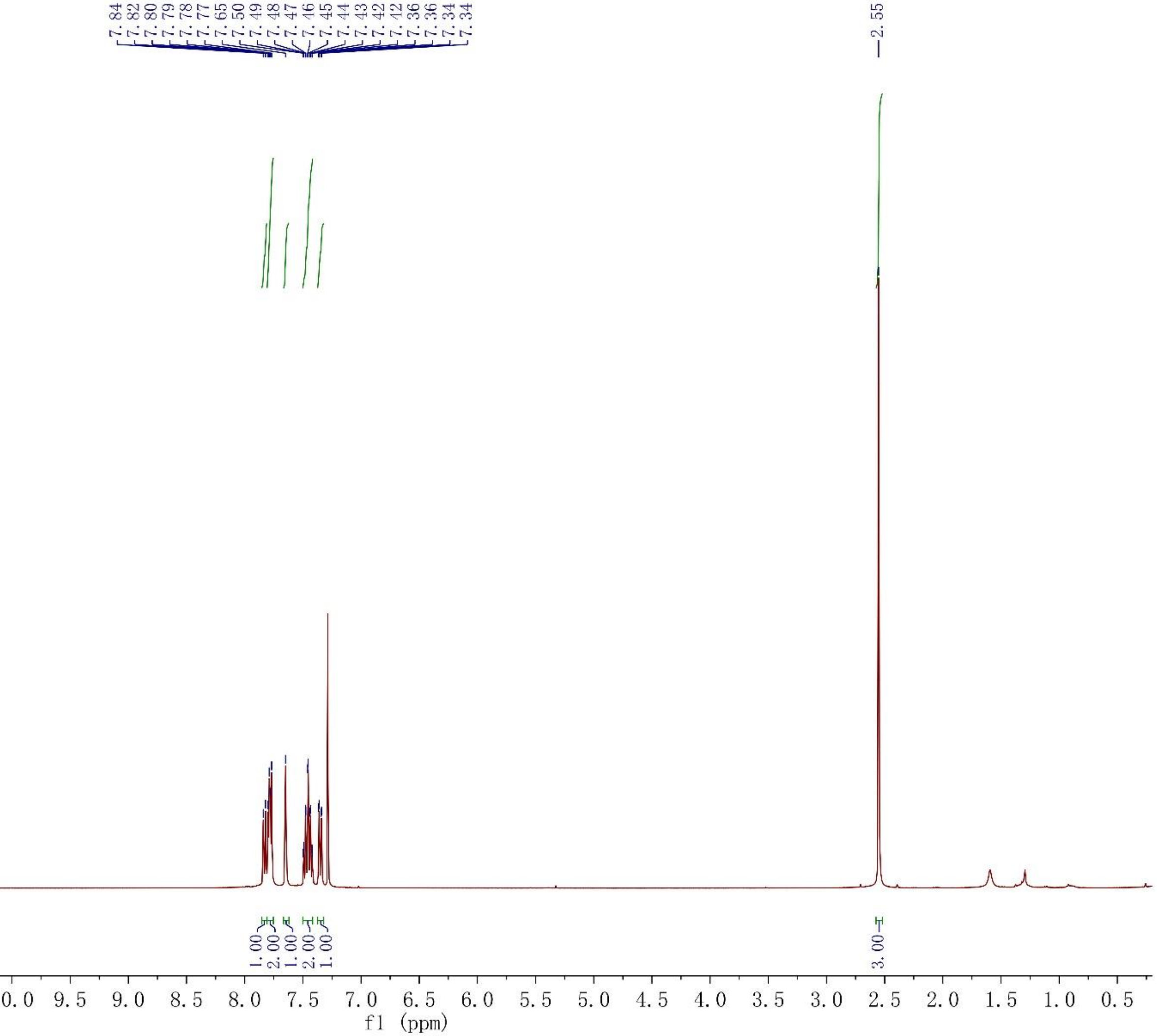
7.49
7.47
7.43
7.42
7.40
7.36
7.35
7.34
7.33
7.33
7.32
7.32
7.31
2.36
2.01
3.00
4.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



CH3
142.03
142.00
135.41
130.38
129.87
129.27
128.14
127.32
126.83
125.84
77.43
77.11
76.79
20.55
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


CH3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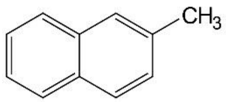
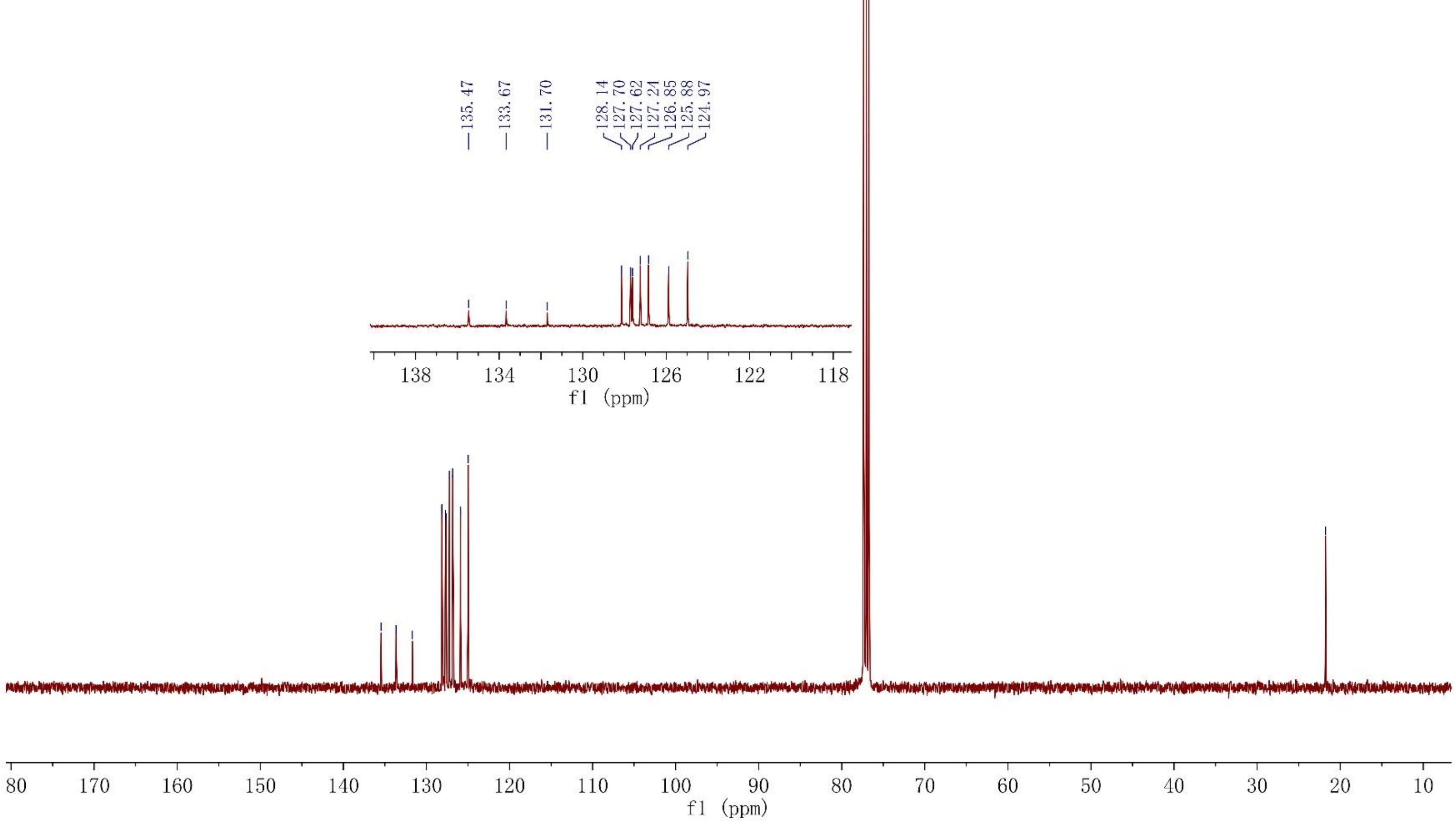
7.79
7.78
7.77
7.65
7.50
7.49
7.48
7.47
7.46
7.45
7.44
7.43
7.42
7.42
7.36
7.36
7.34
7.34
2.55
1.00
2.00
1.00
2.00
1.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


CH3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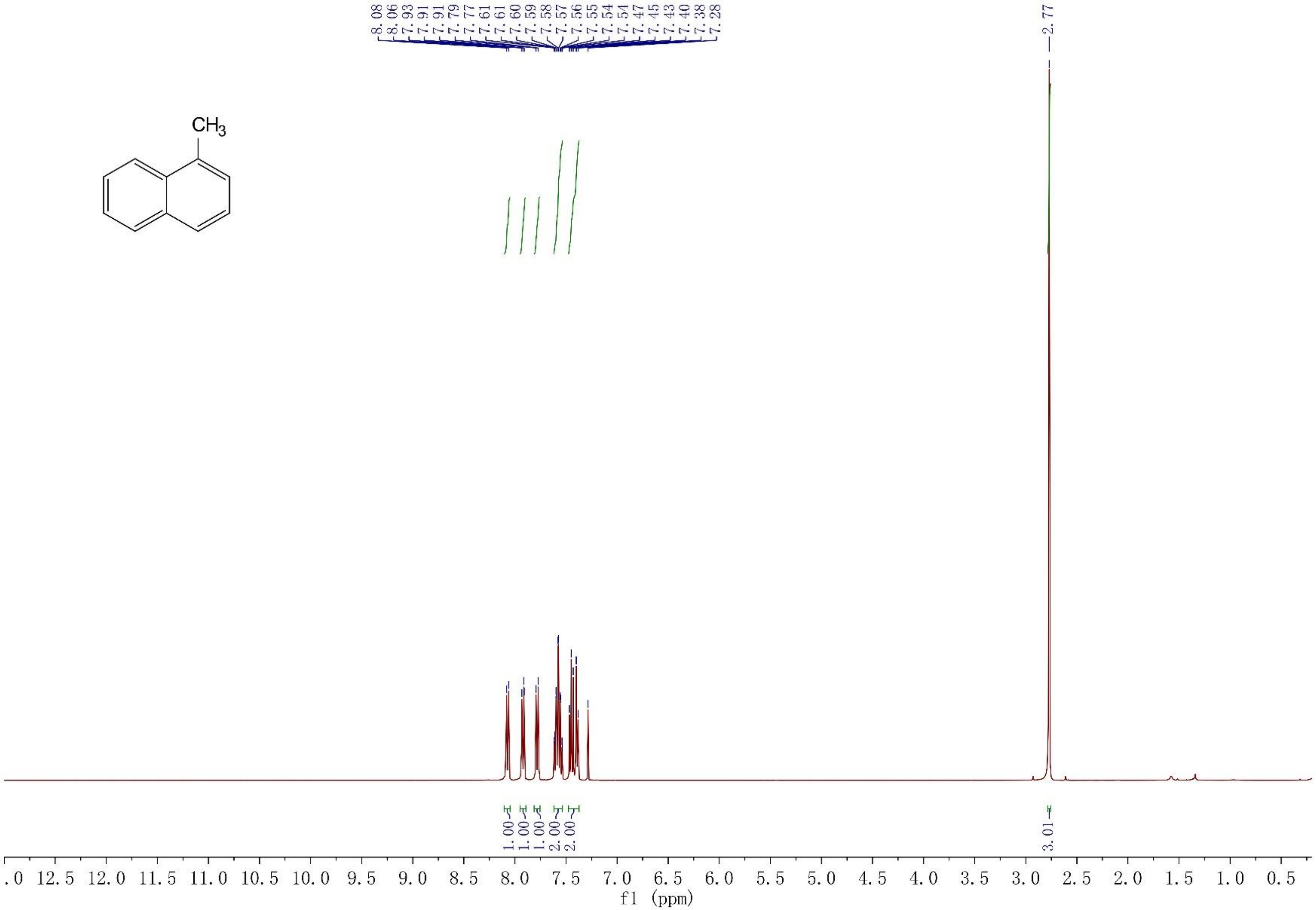
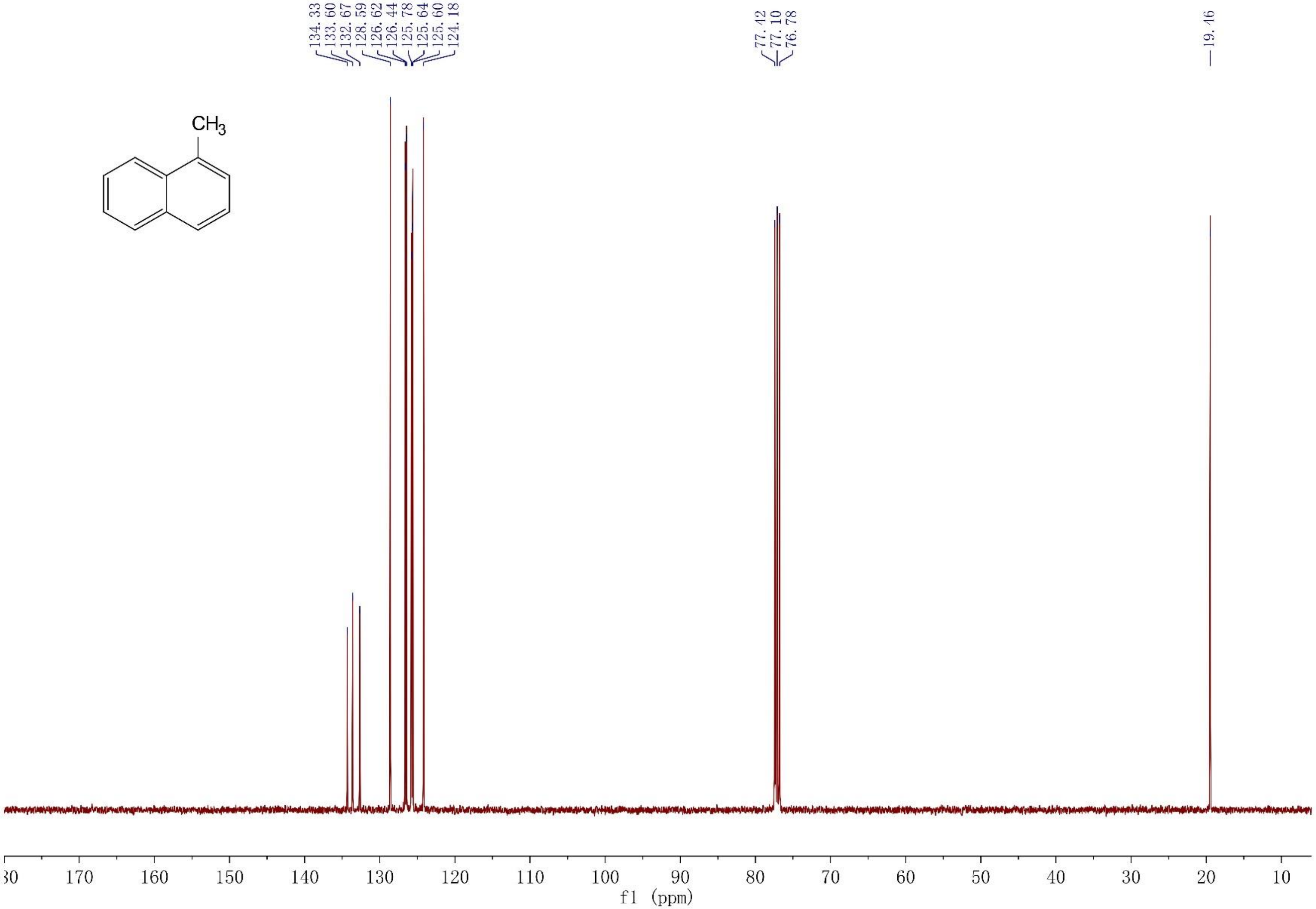
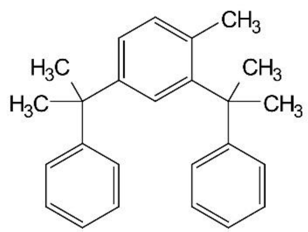
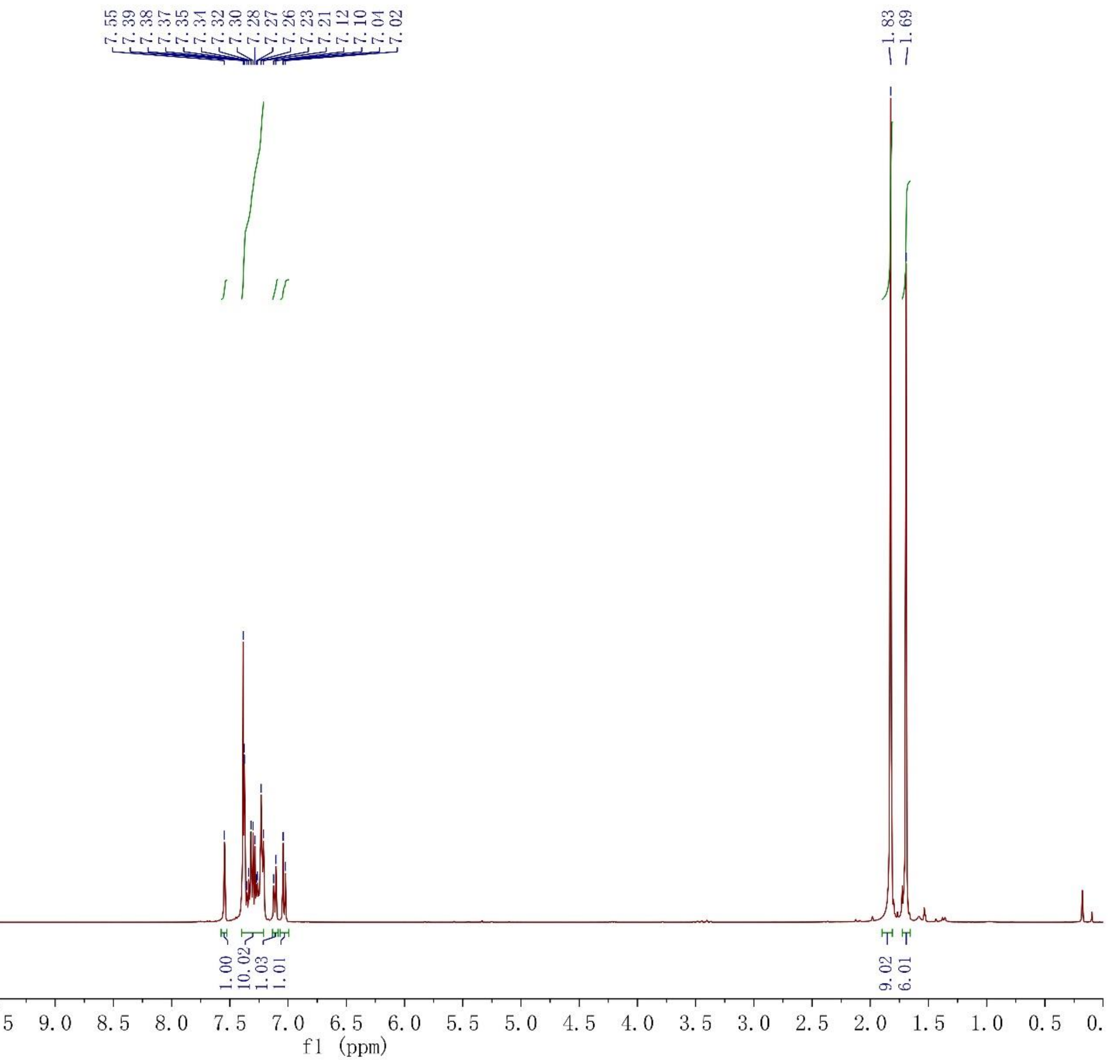
133.67
131.70
128.14
127.70
127.62
127.24
126.85
125.88
124.97
77.37
77.05
76.73
21.75
135.47
133.67
131.70
128.14
127.70
127.62
127.21
126.85
125.88
124.97
138
134
130
126
122
118
f1 (ppm)
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


CH3
8.08
8.06
7.93
7.91
7.91
7.79
7.77
7.61
7.61
7.60
7.59
7.58
7.57
7.56
7.55
7.54
7.54
7.47
7.45
7.43
7.40
7.38
7.28
2.77
1.00
1.00
1.00
2.00
2.00
3.01
.0 12.5 12.0 11.5 11.0 10.5 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
f1 (ppm)



134.33
133.60
132.67
128.59
126.62
126.44
125.78
125.64
125.60
124.18
77.42
77.10
76.78
19.46
CH3
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

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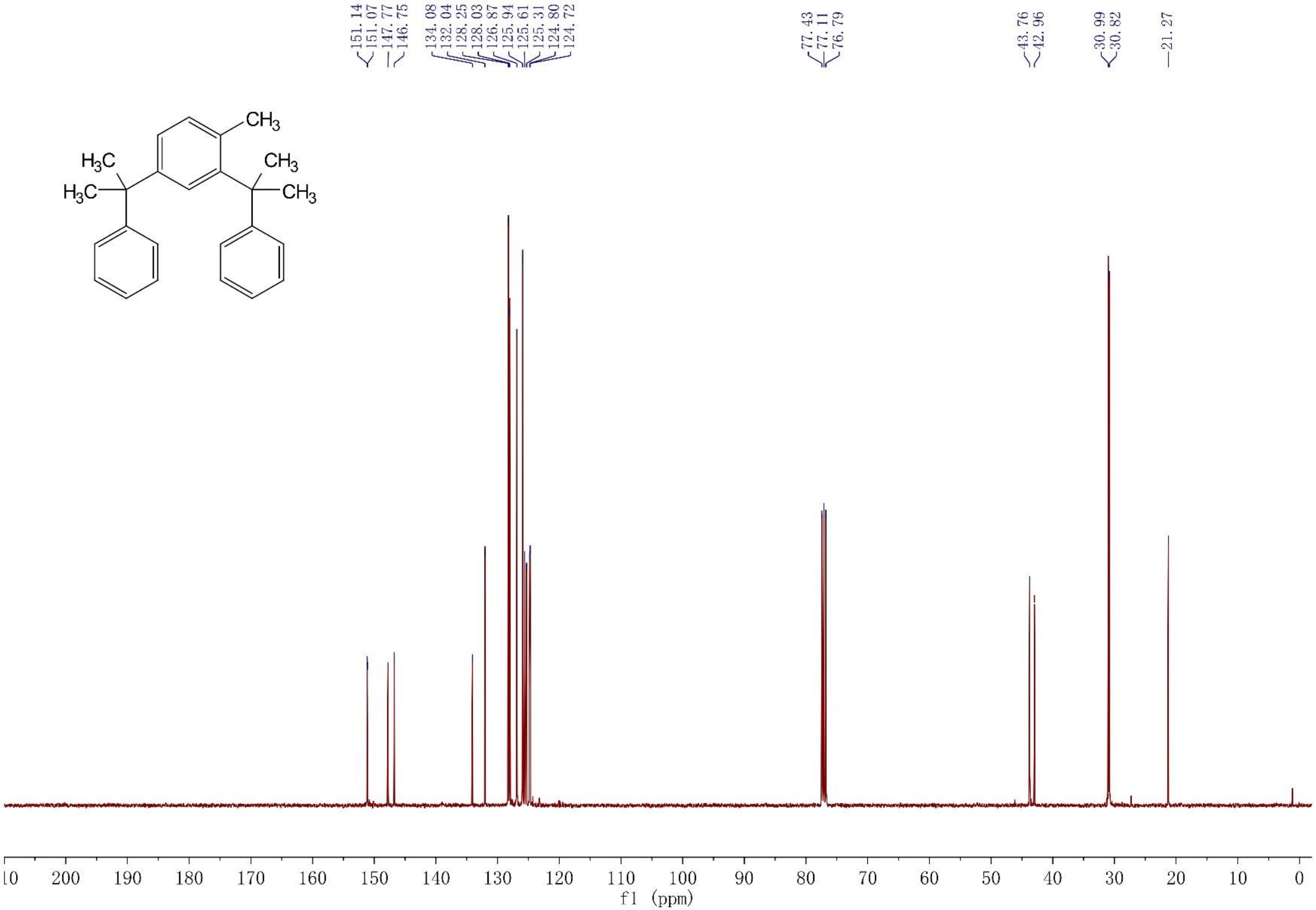
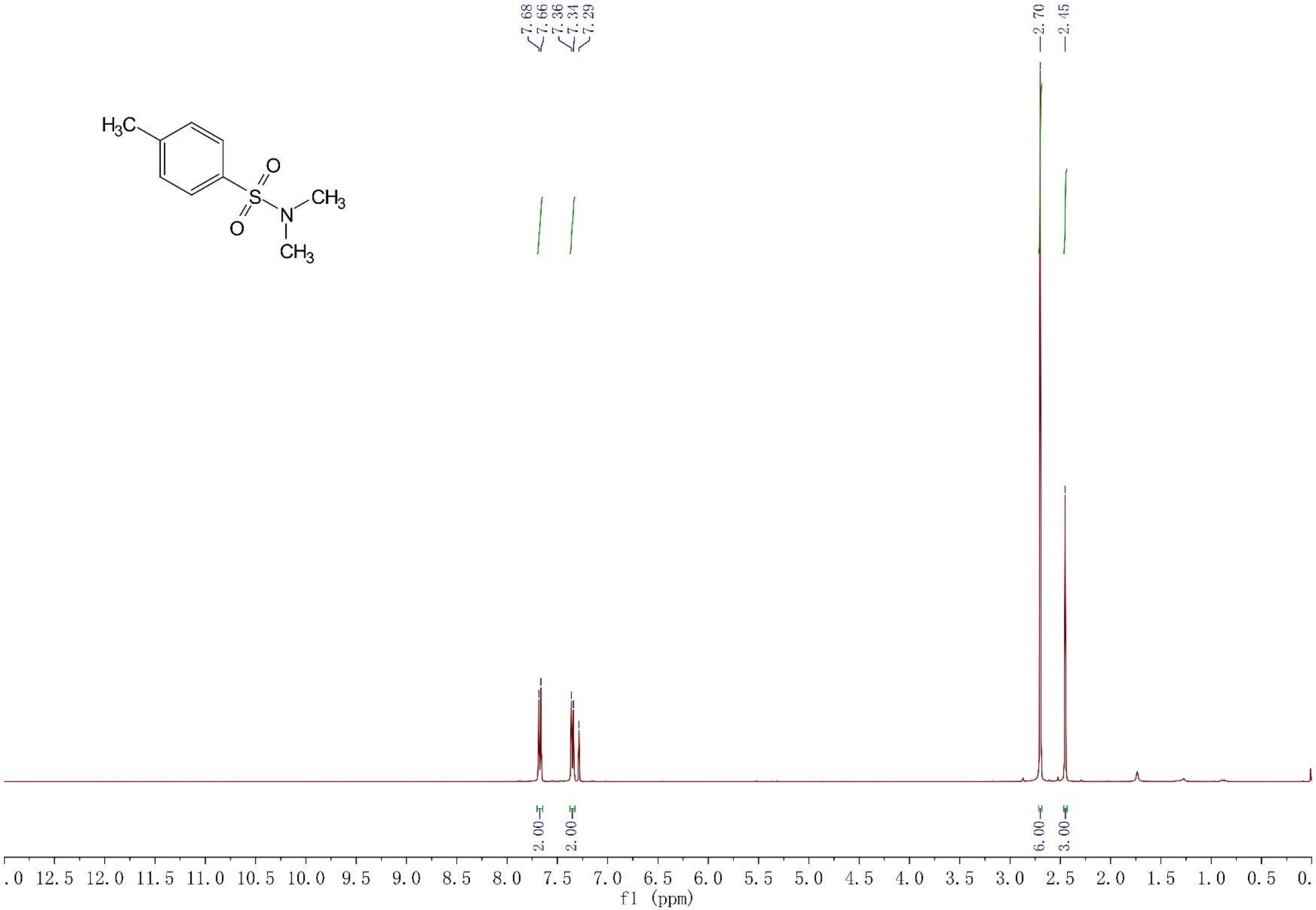
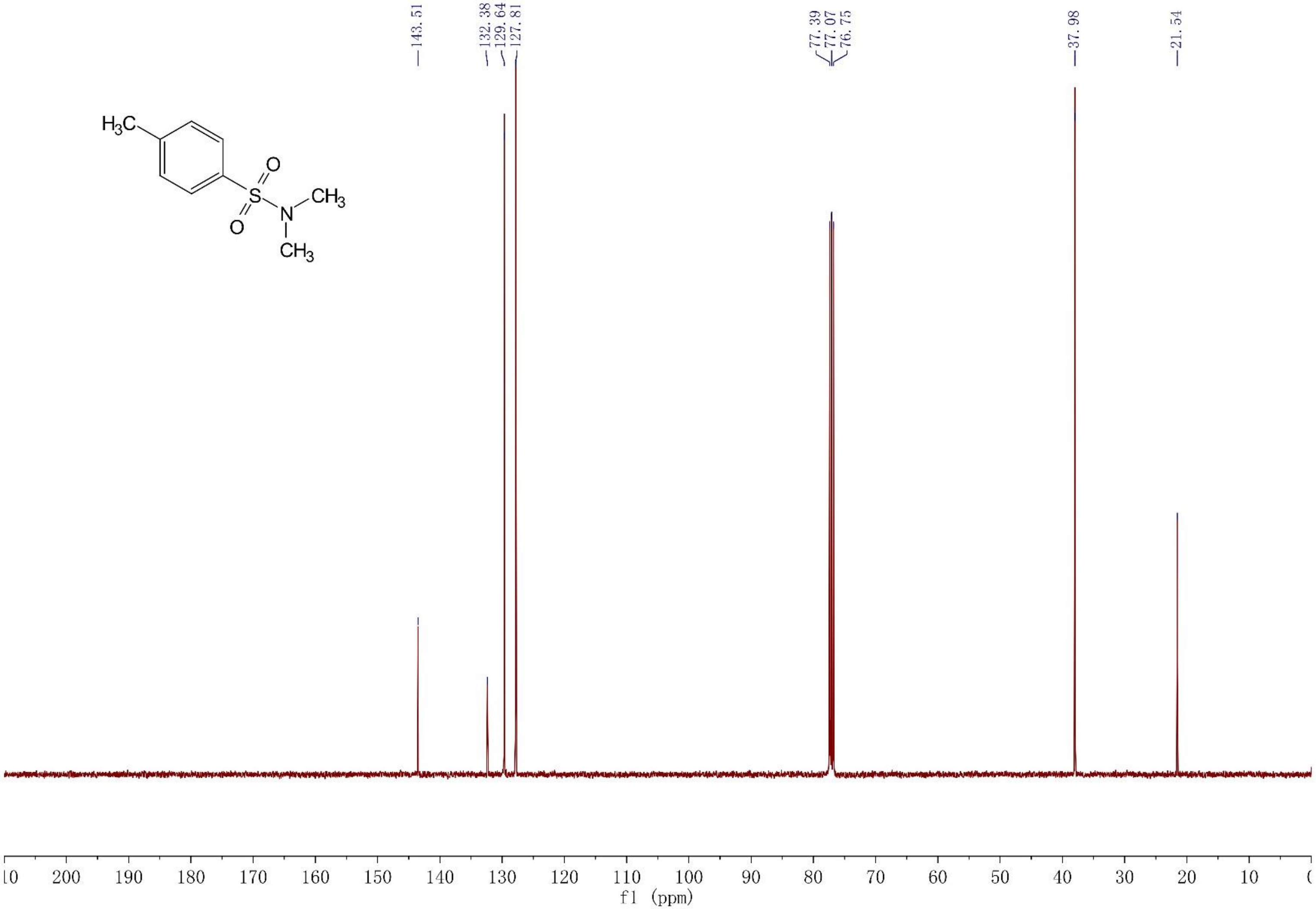
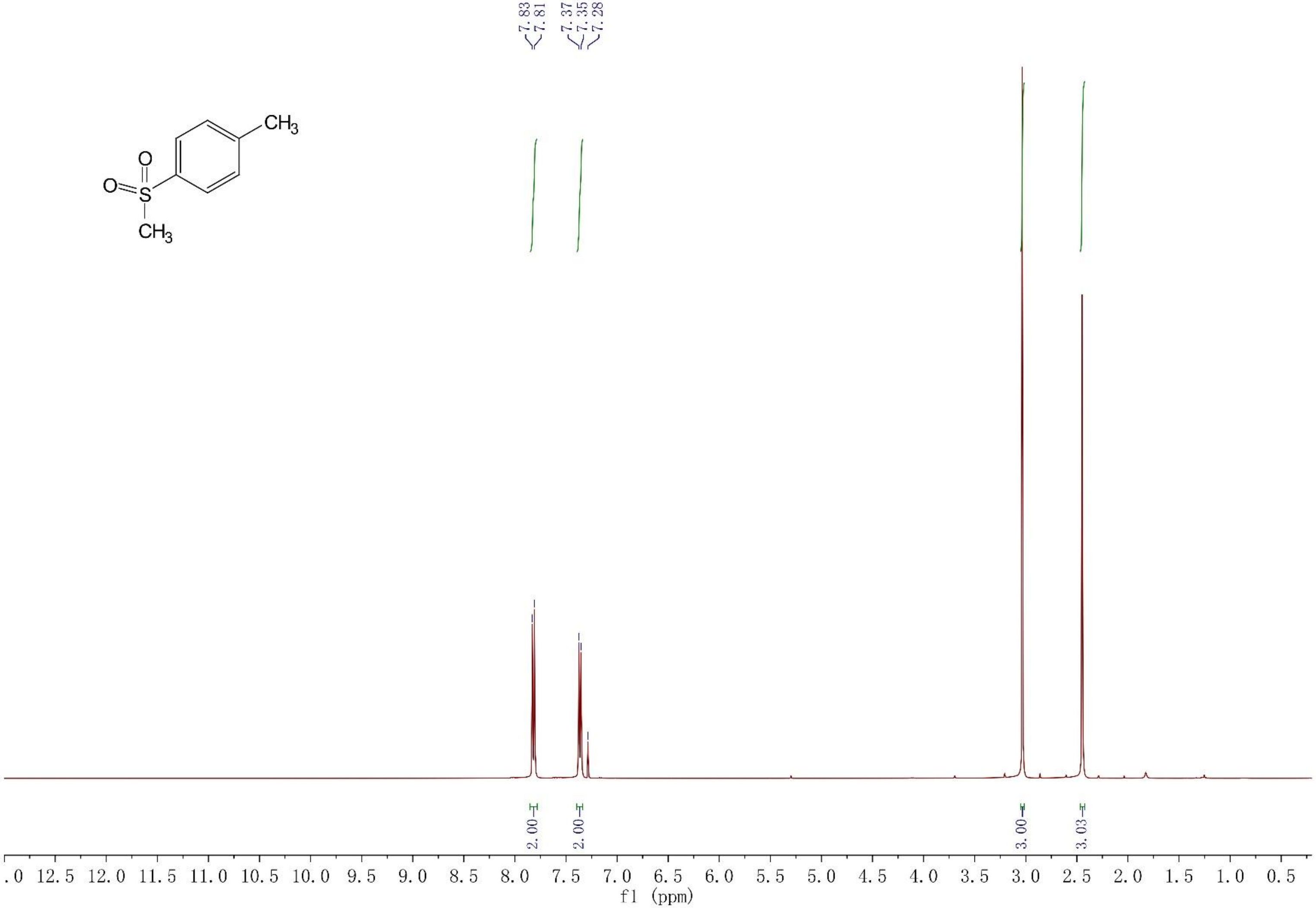
7.39
7.38
7.37
7.35
7.34
7.32
7.30
7.28
7.27
7.26
7.23
7.21
7.12
7.10
7.04
7.02
1.83
1.69
1.00
10.02
1.03
1.01
9.02
6.01
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)

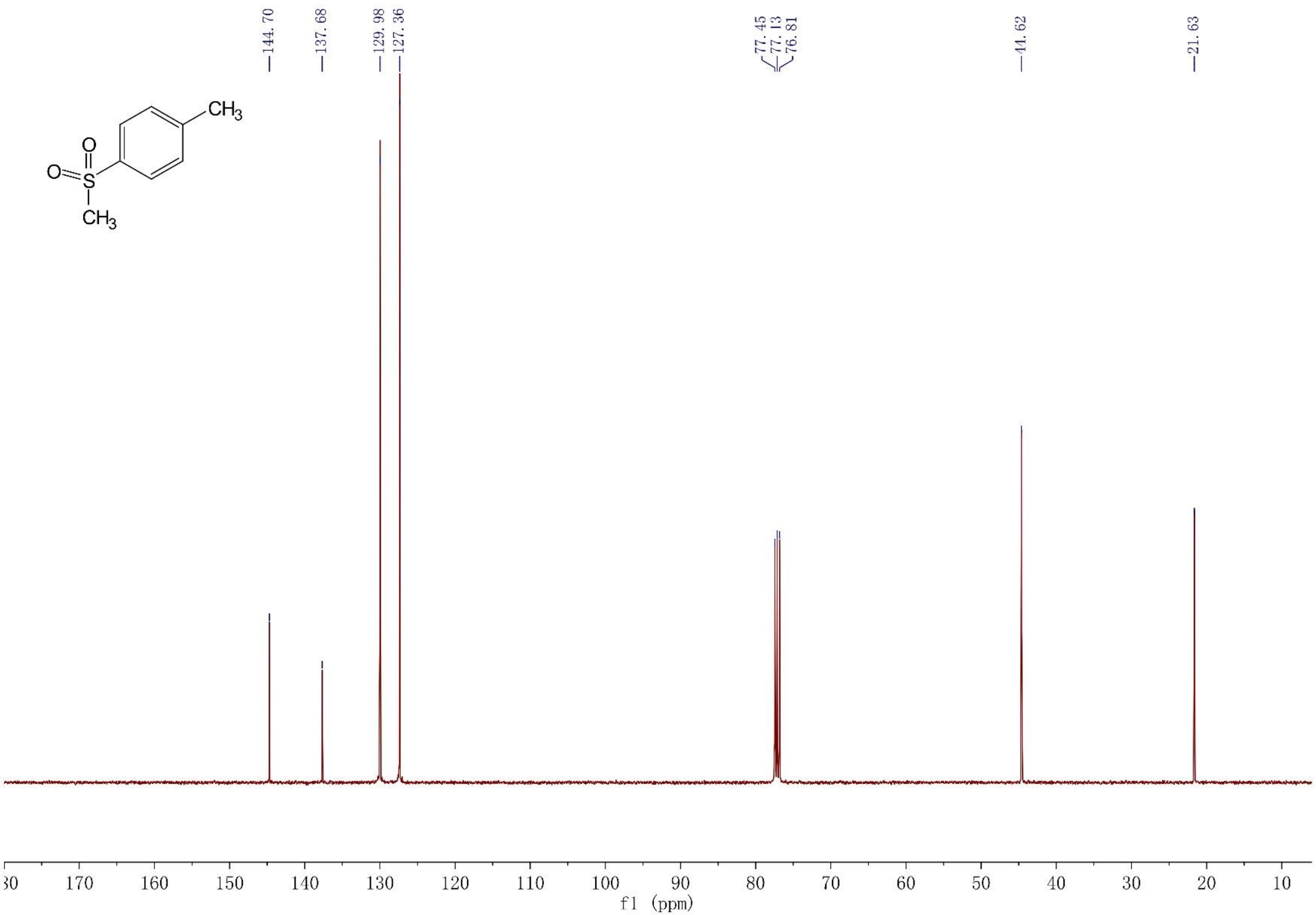

151.14
151.07
147.77
146.75
134.08
132.04
128.25
128.03
126.87
125.94
125.61
125.31
124.80
124.72
77.43
77.11
76.79
43.76
42.96
30.99
30.82
21.27
CH3
H3C
CH3
H3C
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.68
7.66
7.36
7.34
7.29
2.70
2.45
2.00
2.00
6.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 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.
f1 (ppm)


143.51
132.38
129.64
127.81
77.39
77.07
76.75
37.98
21.54
H3C
O
S
N
CH3
O
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



CH3
O
O
S
CH3
7.83
7.81
7.37
7.35
7.28
2.00
2.00
3.00
3.03
.0 12.5 12.0 11.5 11.0 10.5 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
f1 (ppm)



144.70
137.68
129.98
127.36
77.45
77.13
76.81
44.62
21.63
CH3
O
O
S
CH3
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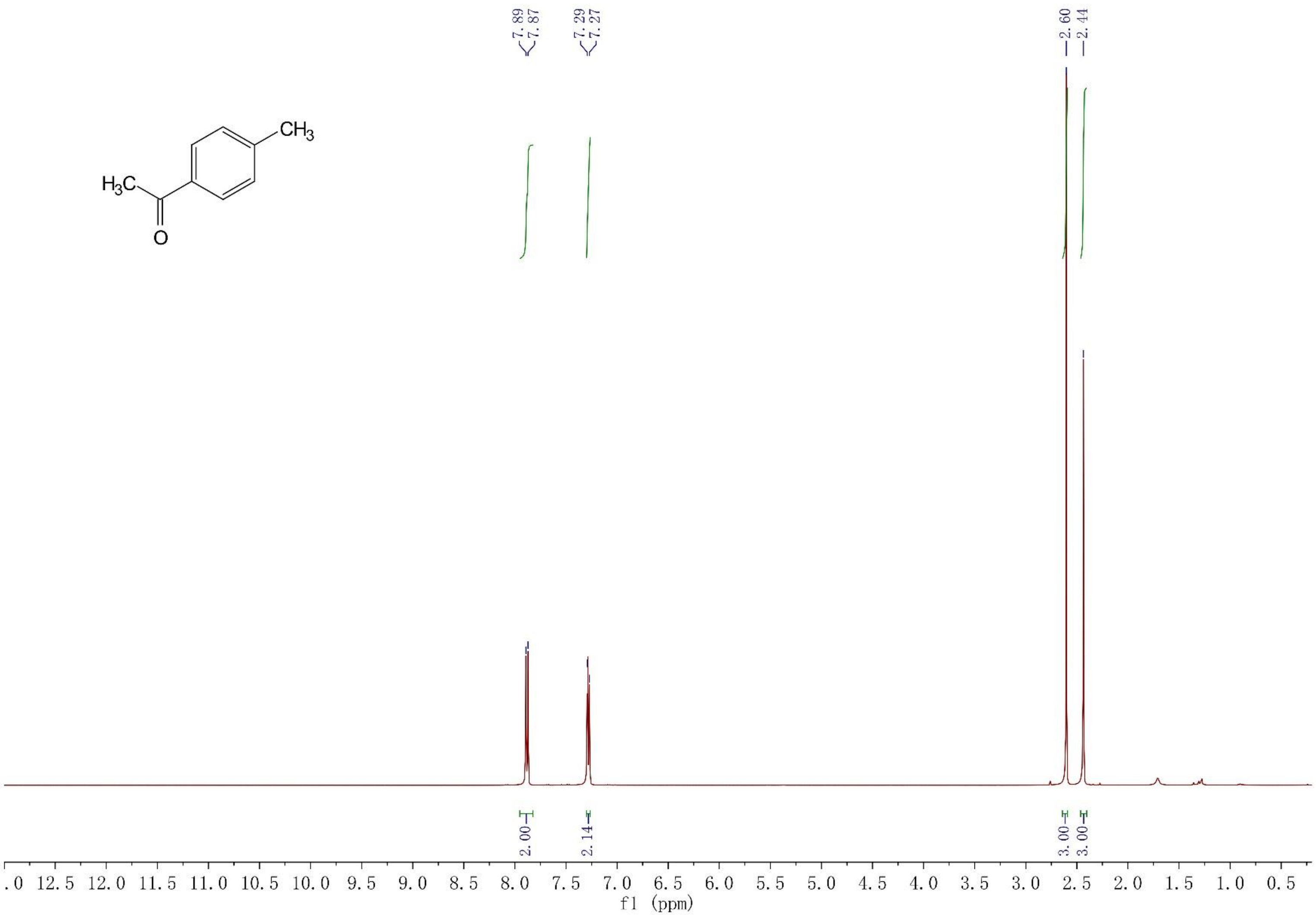
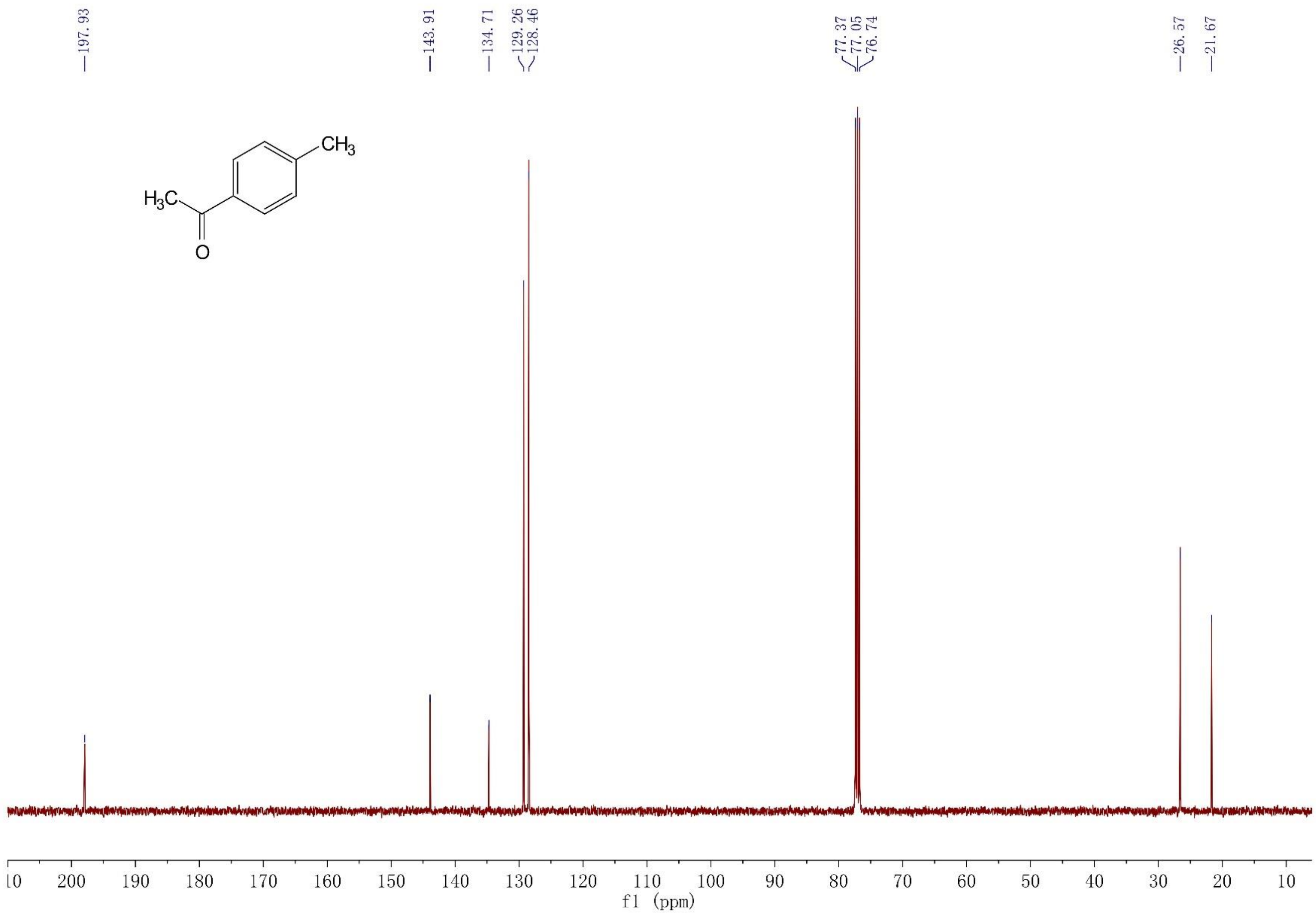
f1 (ppm)



CH3
H3C
O
7.89
7.87
7.29
7.27
2.60
2.44
2.00
2.14
3.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 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
f1 (ppm)



197.93
143.91
134.71
129.26
128.46
77.37
77.05
76.74
26.57
21.67
CH3
H3C
O
10
200
190
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80
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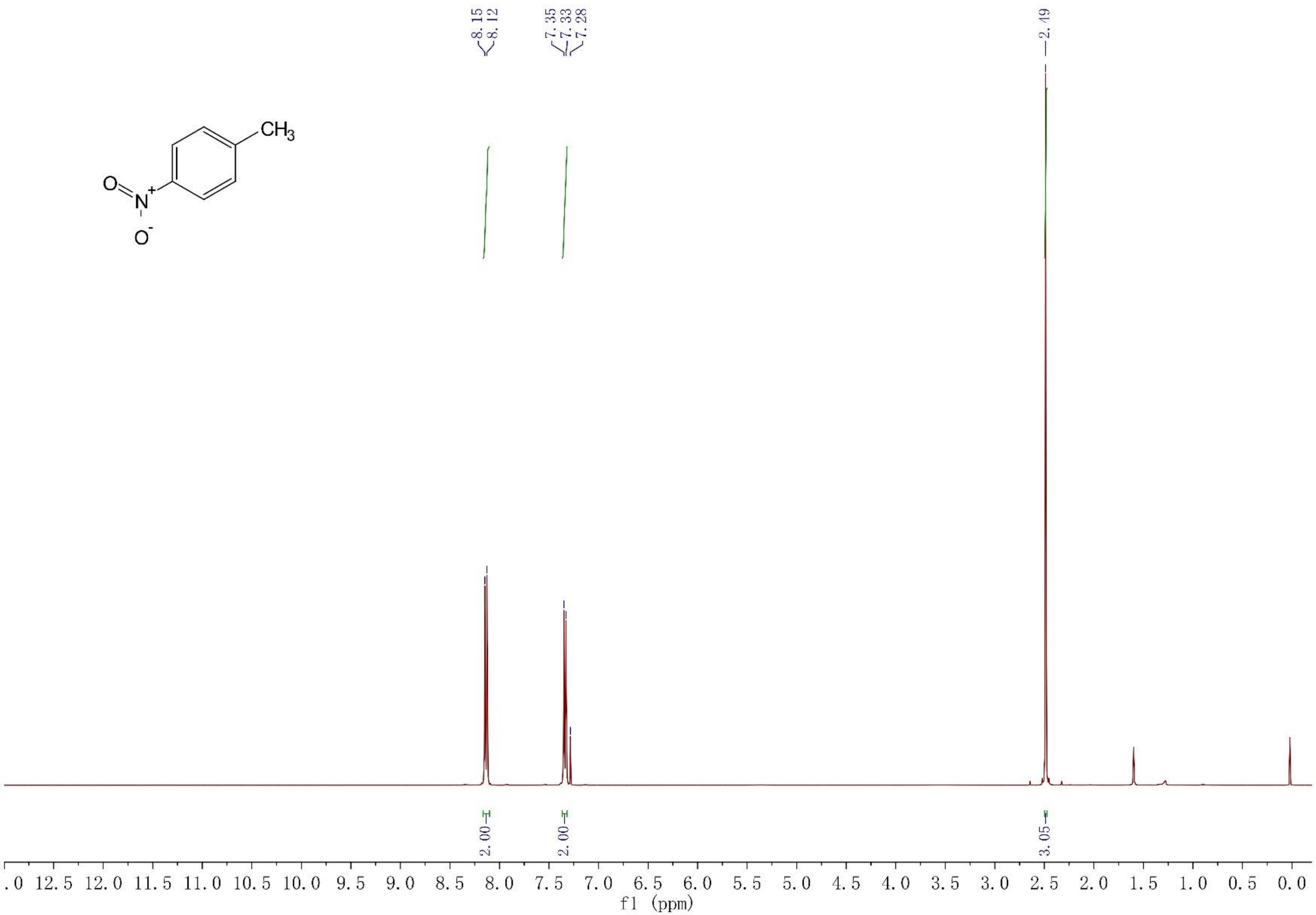
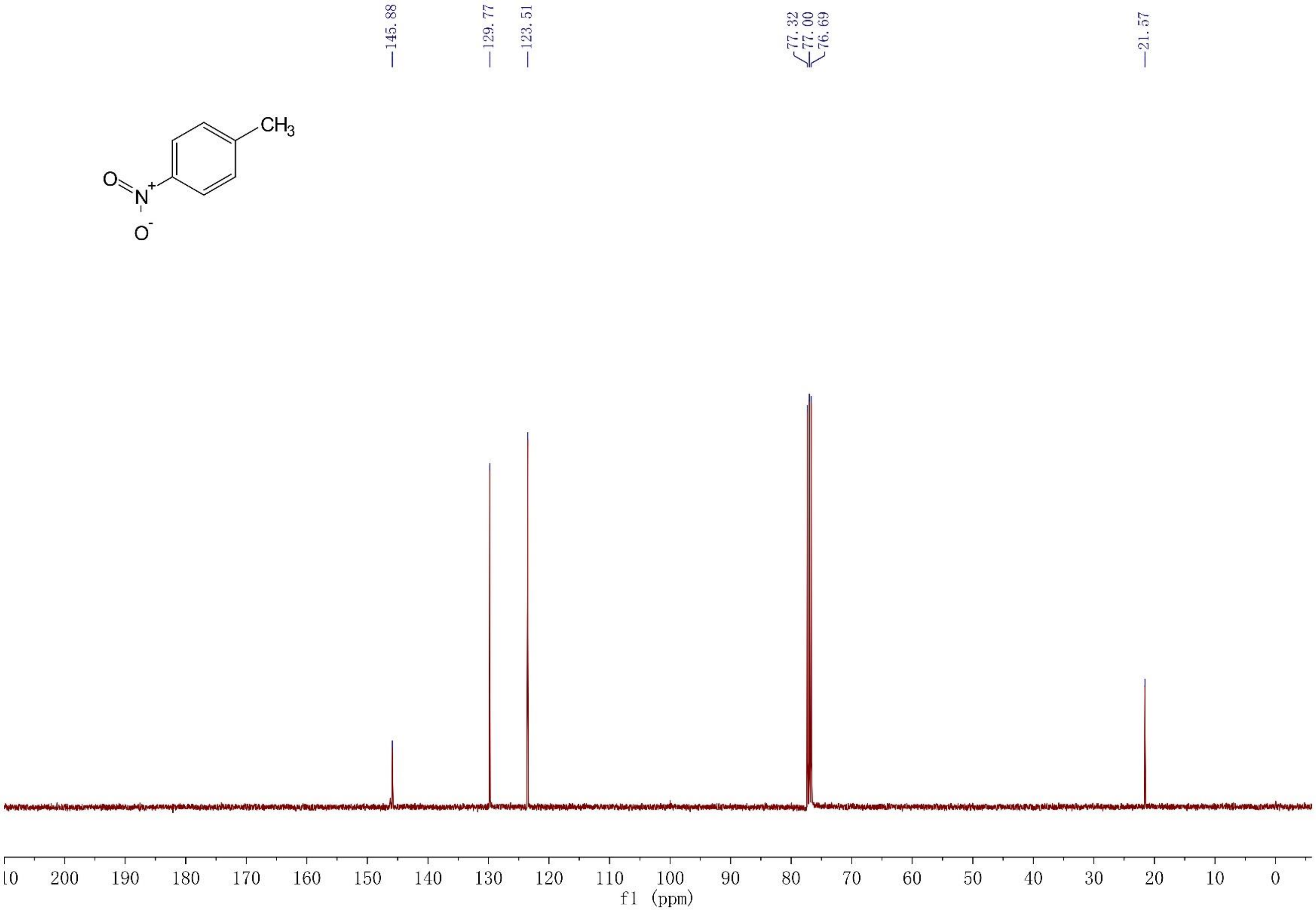
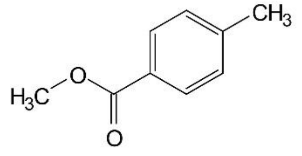
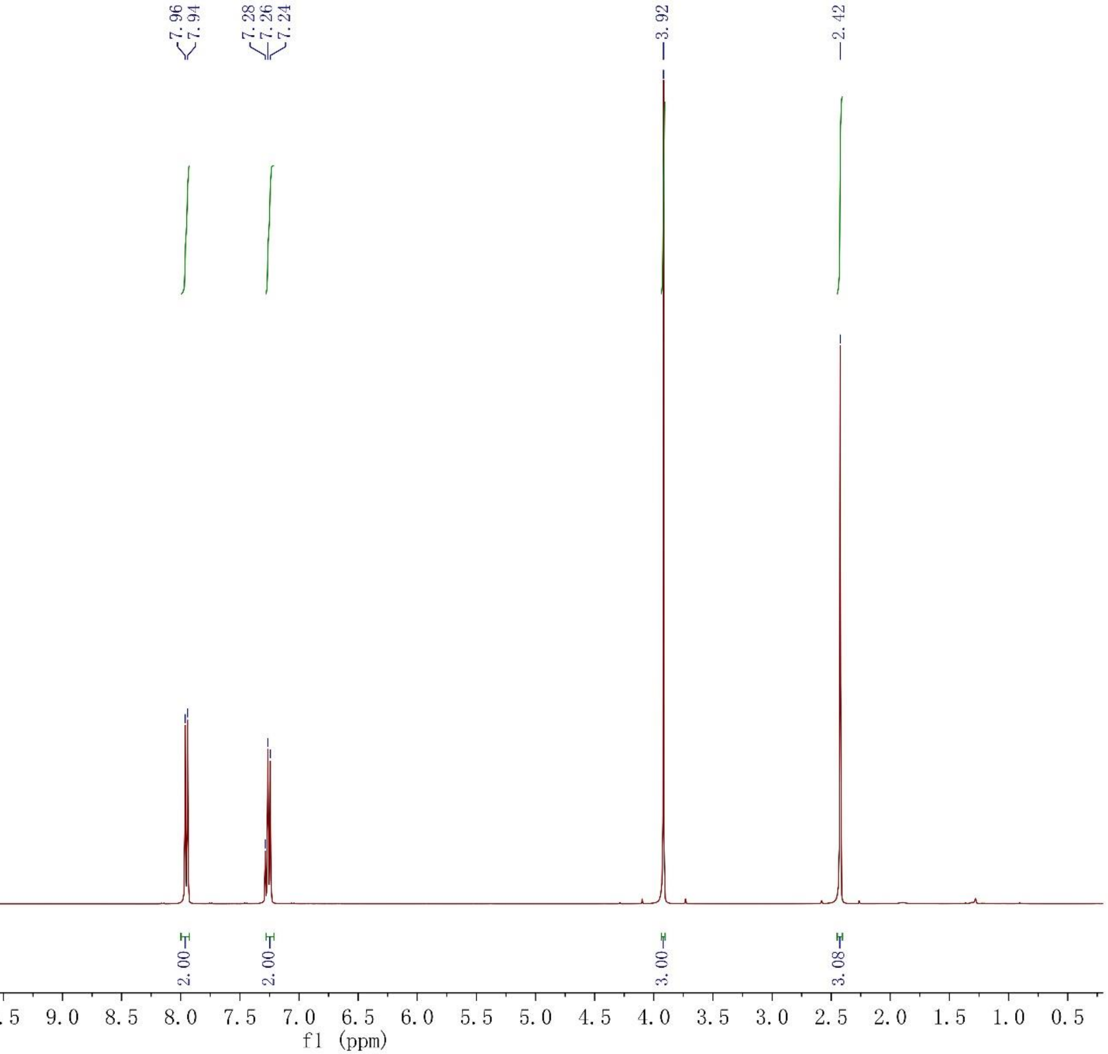
60
50
40
30
20
10
f1 (ppm)



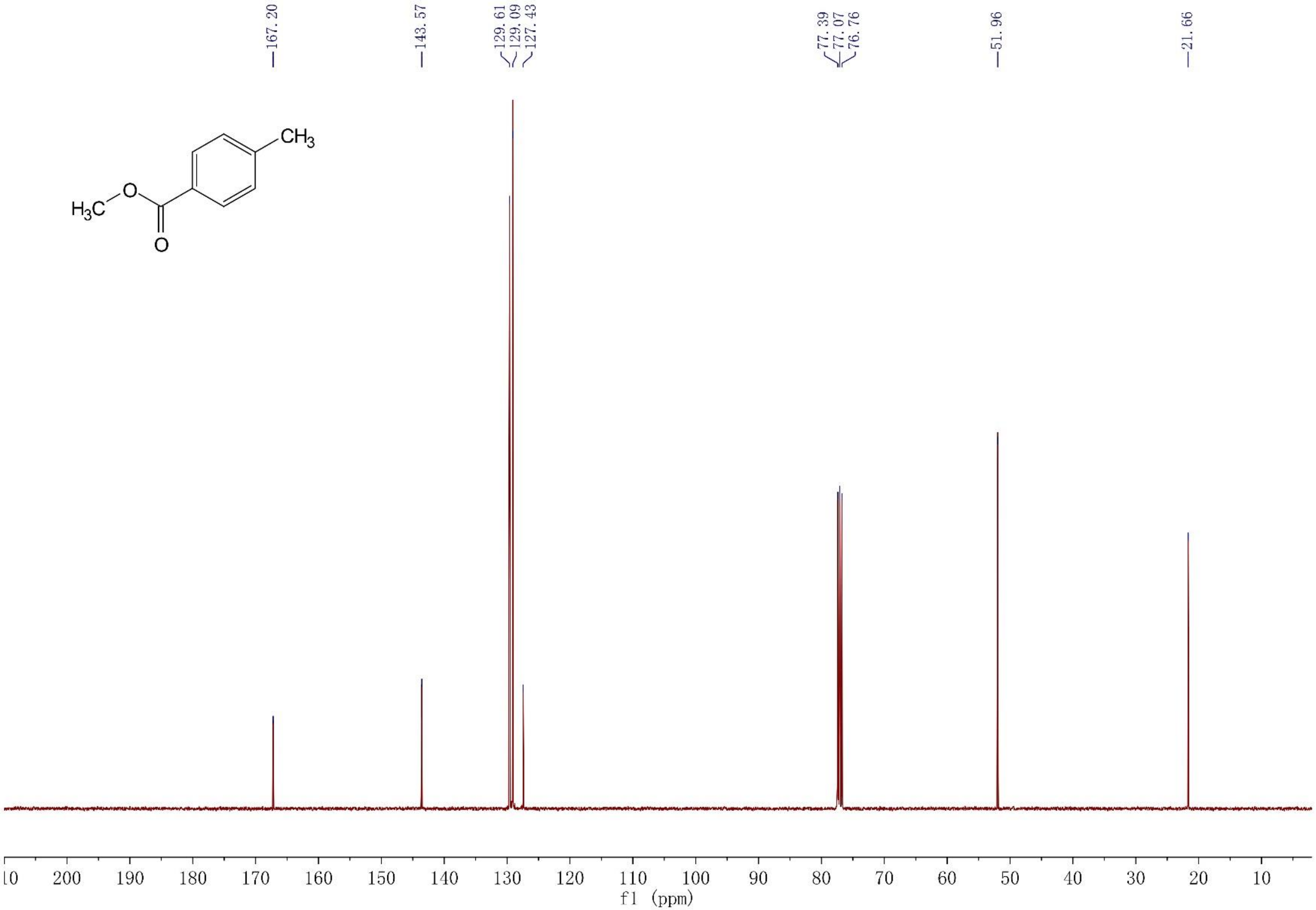
8.15
8.12
7.35
7.33
7.28
2.49
CH3
O
N+
O-
2.00
2.00
3.05
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

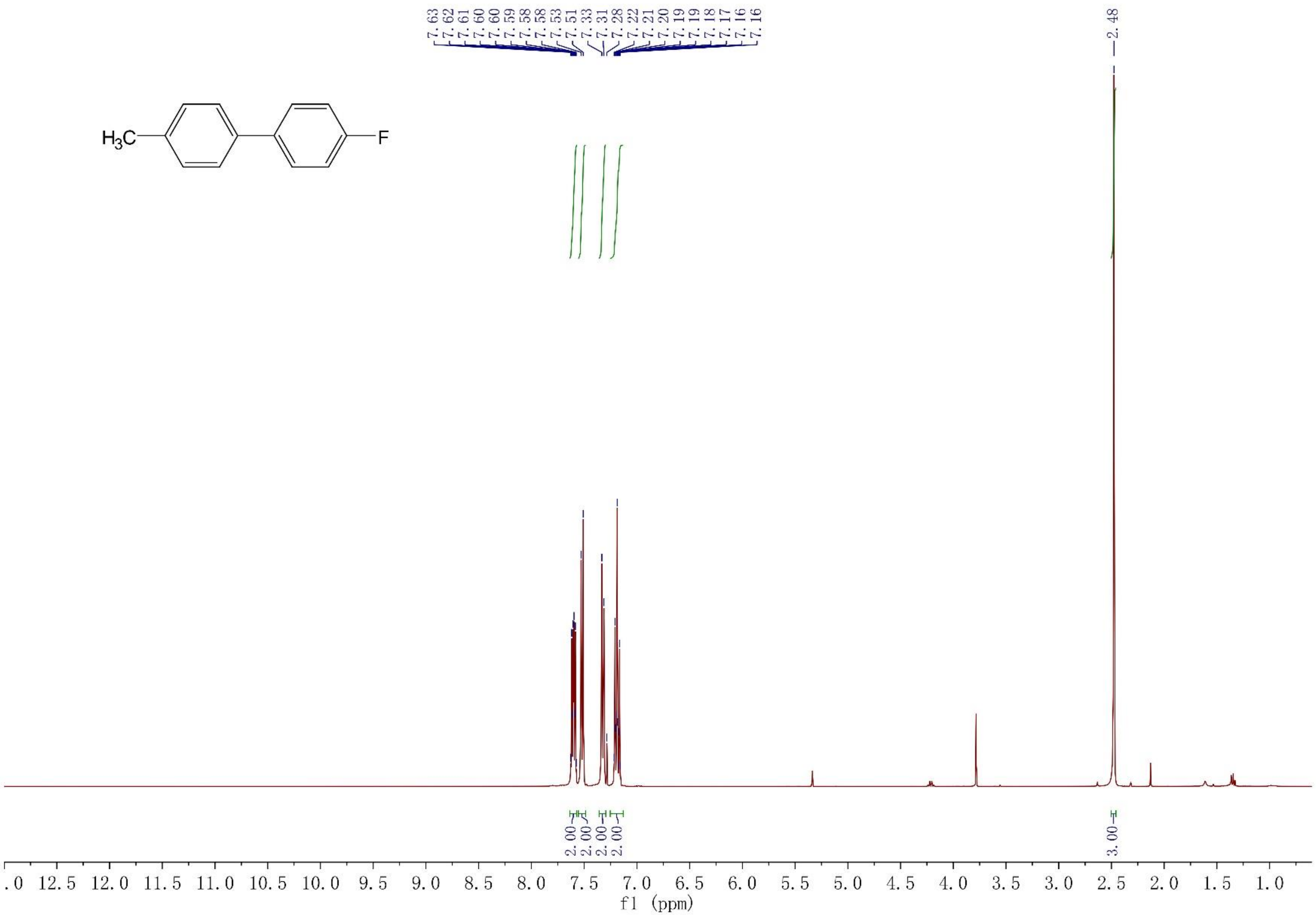

145.88
129.77
123.51
77.32
77.00
76.69
21.57
CH3
O
N+
O-
f1 (ppm)



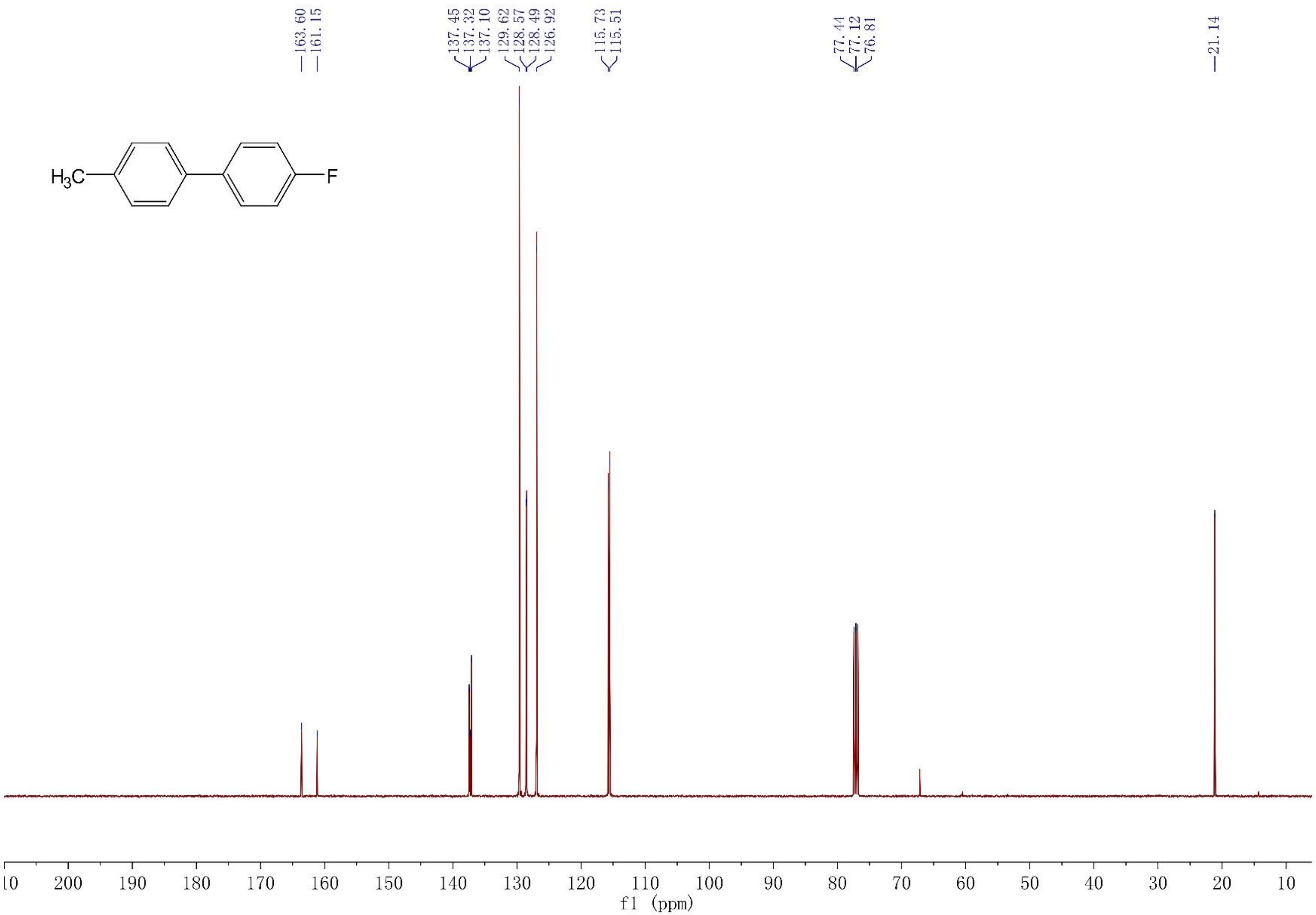


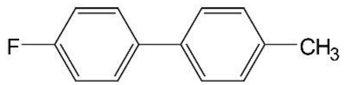
7.96
7.94
7.28
7.26
7.24
3.92
2.42
2.00
2.00
3.00
3.08
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

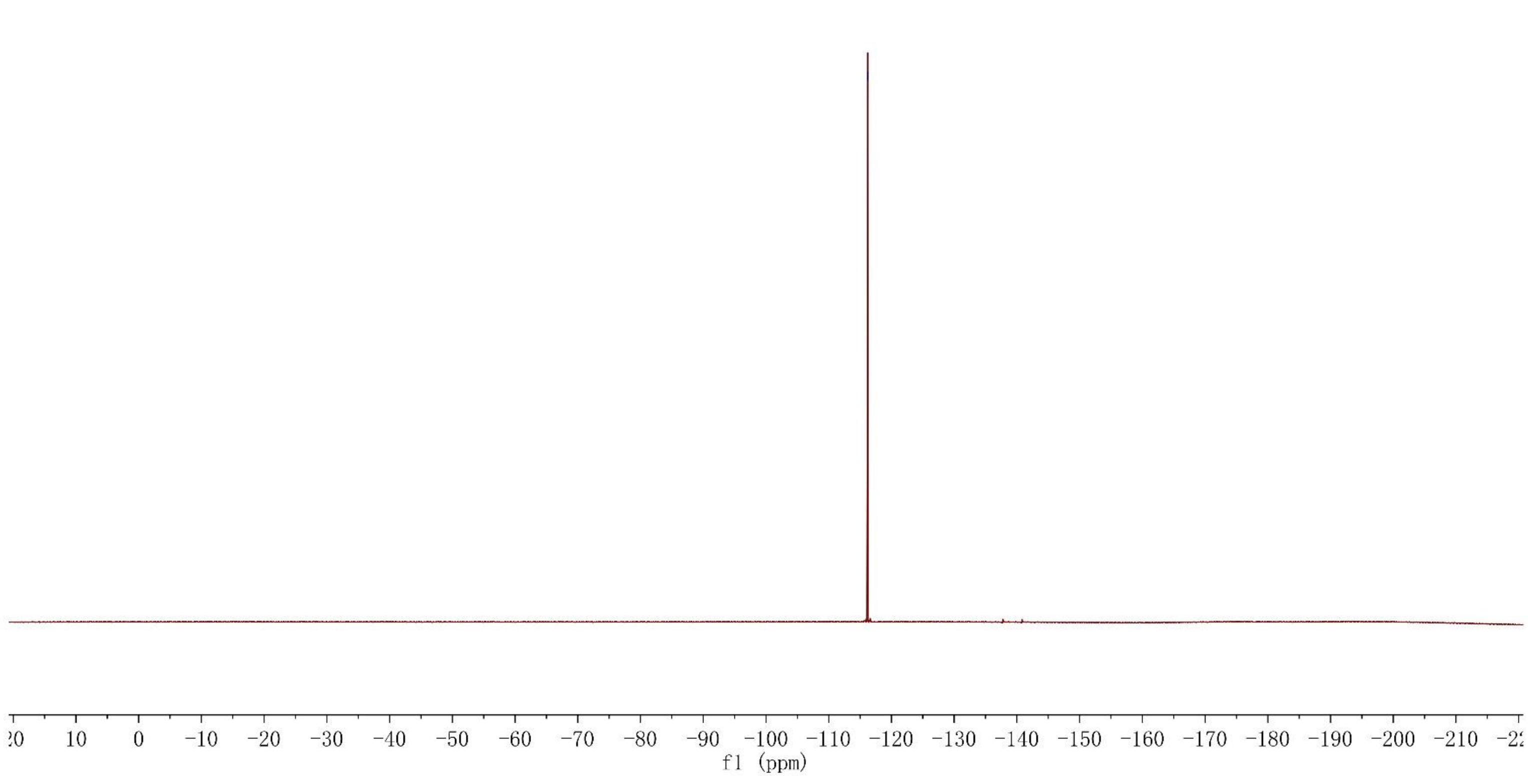

167.20
143.57
129.61
129.09
127.43
77.39
77.07
76.76
51.96
21.66
CH3
H3C
O
O
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



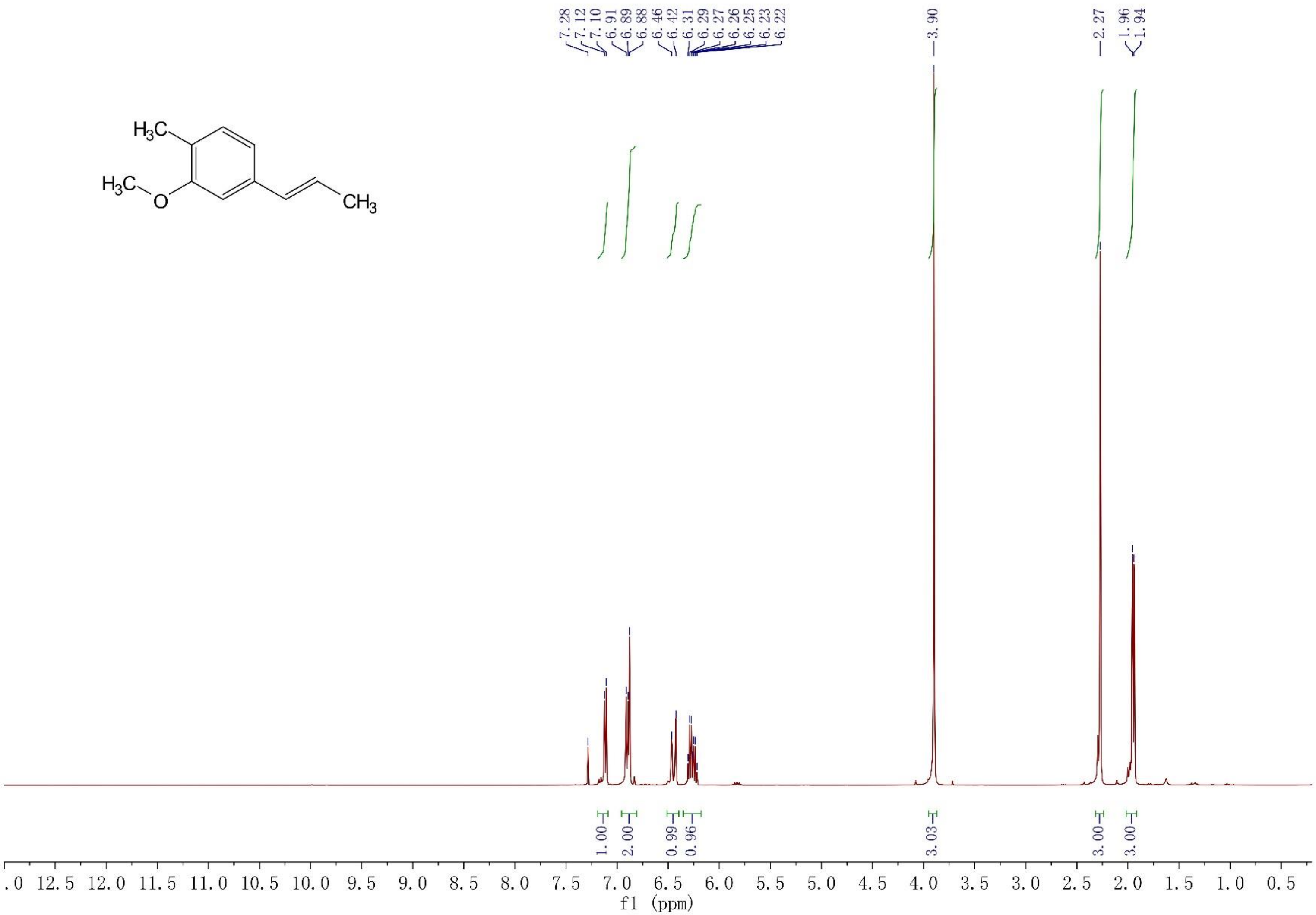
H3C
F
7.63
7.62
7.61
7.60
7.60
7.59
7.58
7.58
7.53
7.51
7.33
7.31
7.28
7.22
7.21
7.20
7.19
7.19
7.18
7.17
7.16
7.16
2.48
2.00
2.00
2.00
2.00
3.00
f1 (ppm)


163.60
161.15
137.45
137.32
137.10
129.62
128.57
128.49
126.92
115.73
115.51
77.44
77.12
76.81
21.14
H3C
F
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

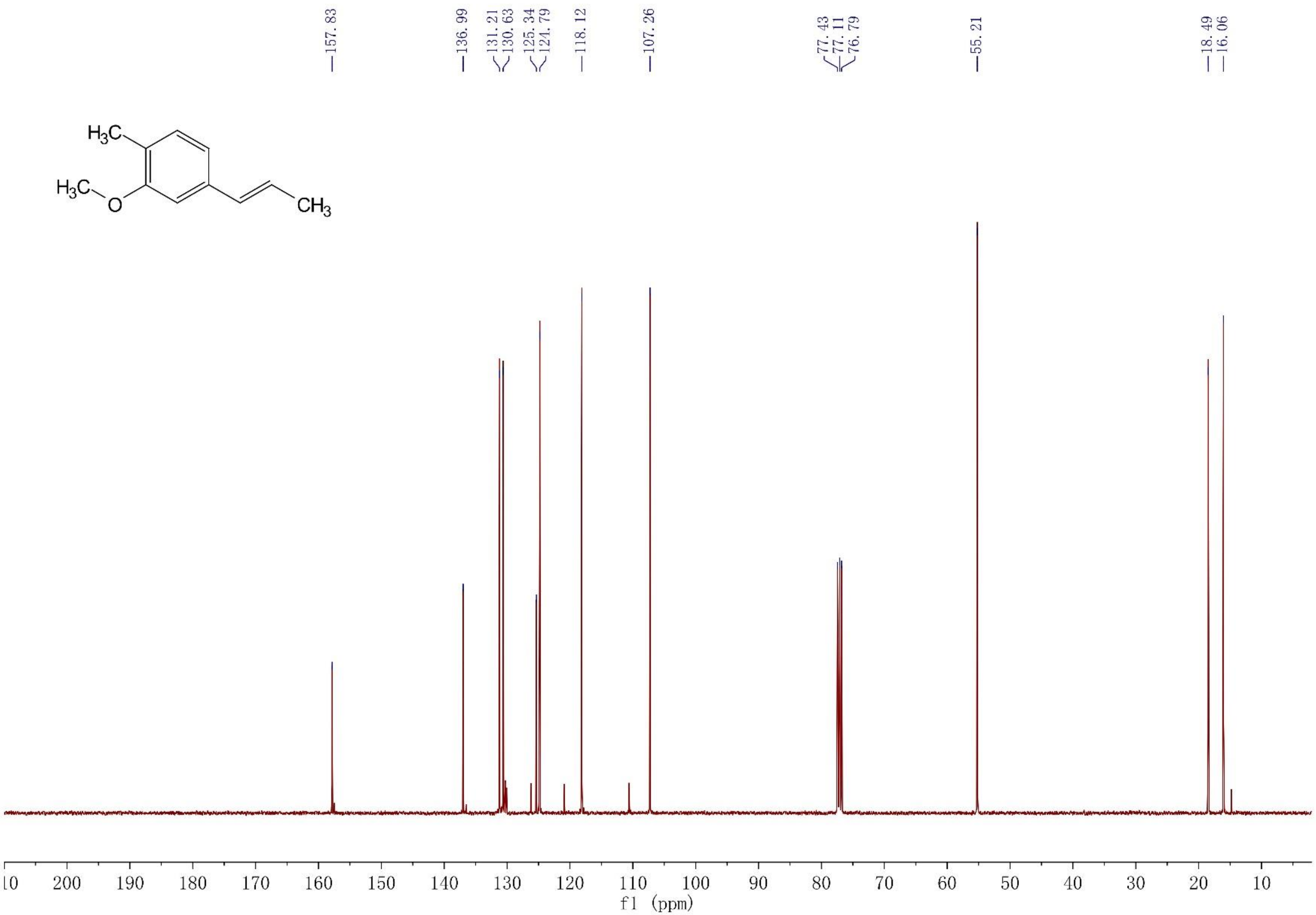




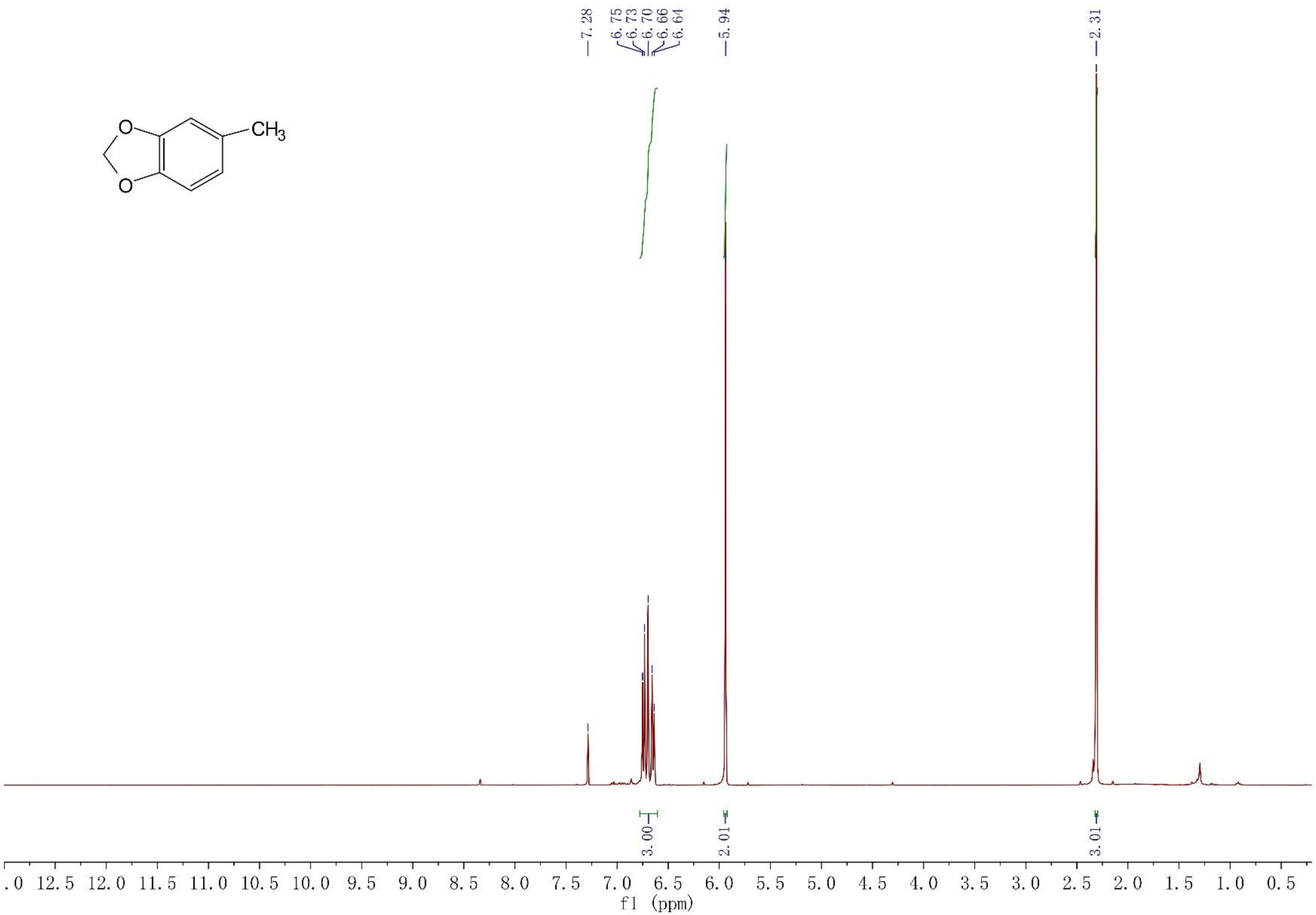
−116.22
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)

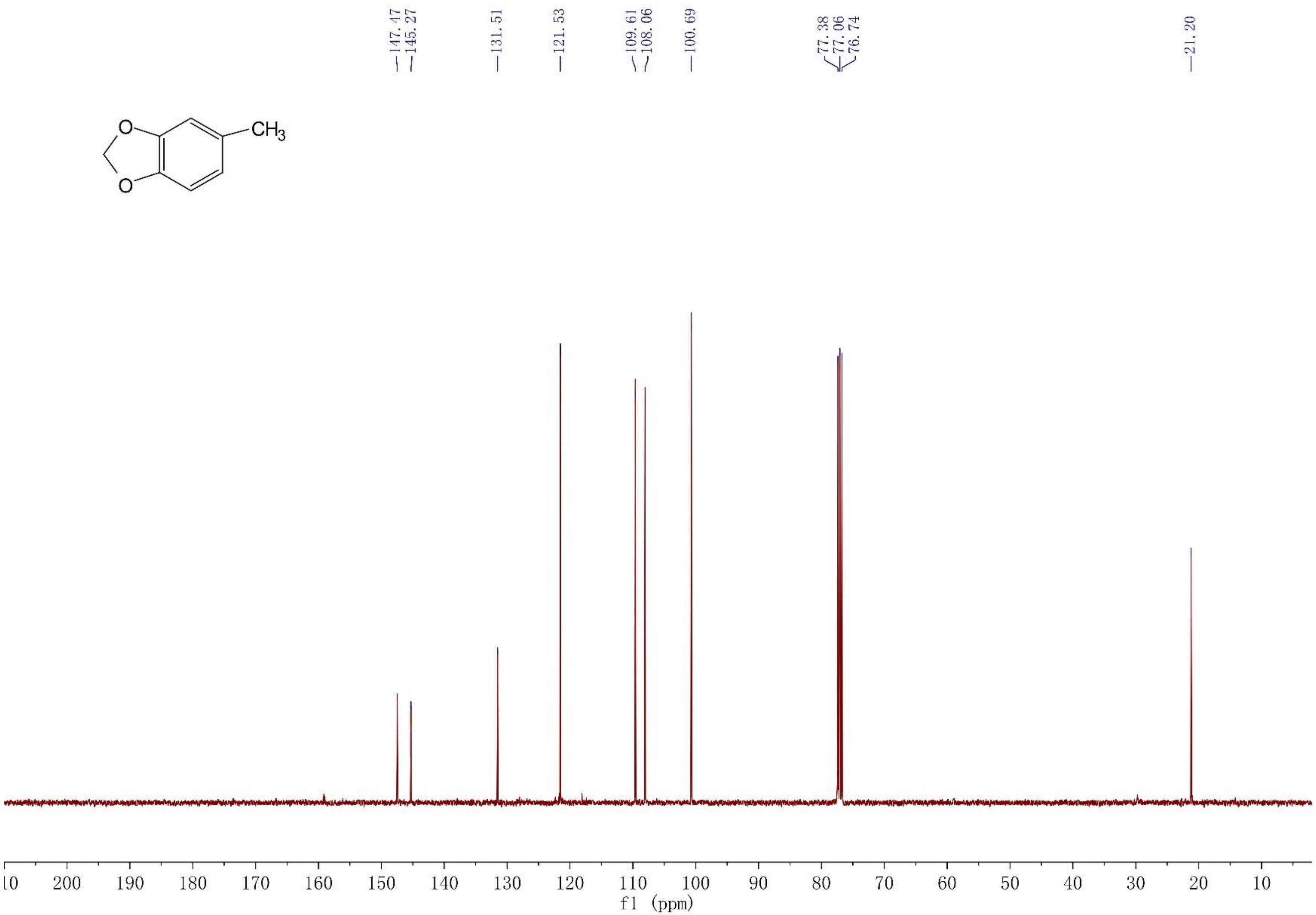


H3C
H3C
O
CH3
7.28
7.12
7.10
6.91
6.89
6.88
6.46
6.42
6.31
6.29
6.27
6.26
6.25
6.23
6.22
3.90
2.27
1.96
1.94
1.00
2.00
0.99
0.96
3.03
3.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

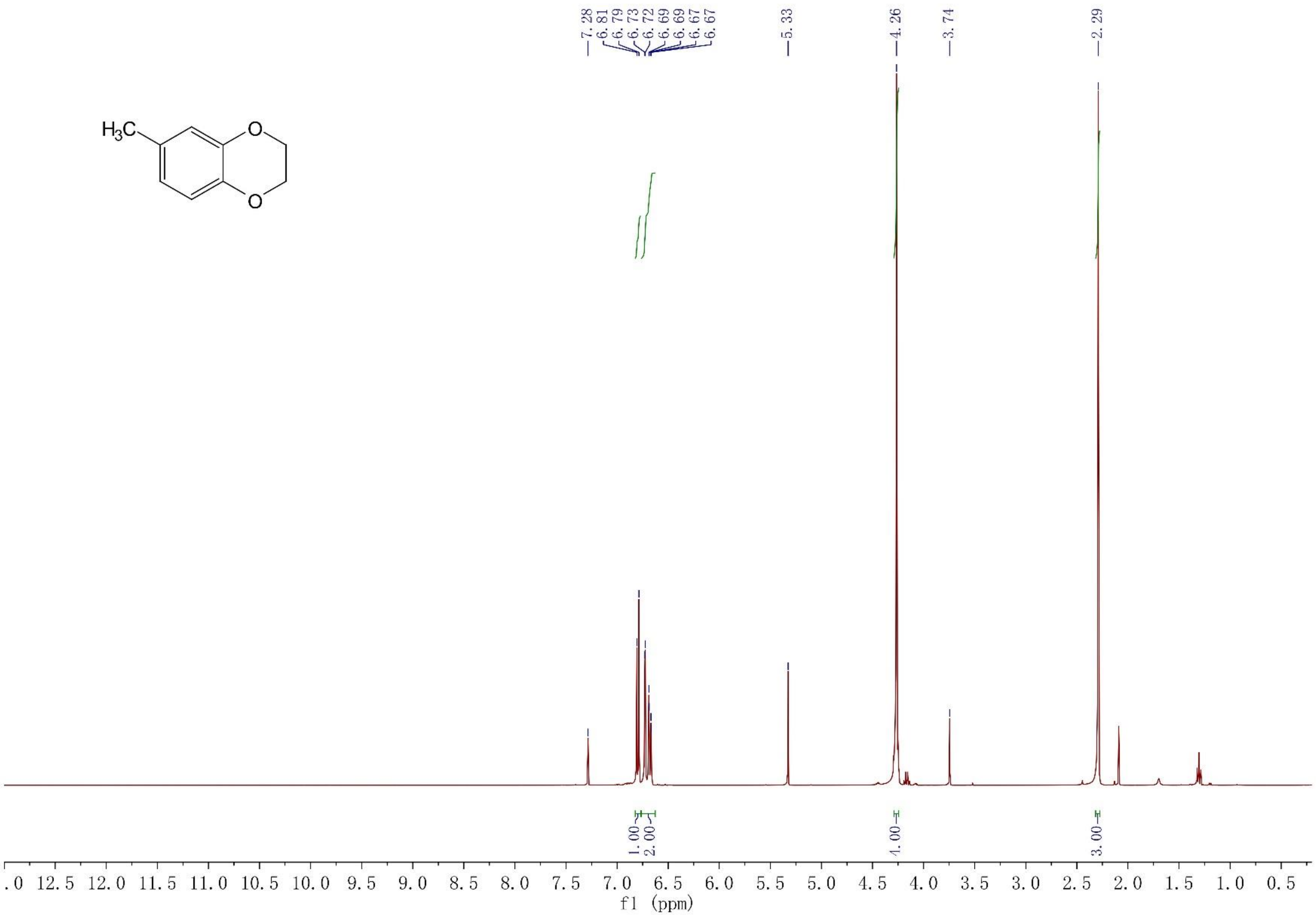


157.83
136.99
131.21
130.63
125.34
124.79
118.12
107.26
77.43
77.11
76.79
55.21
18.49
16.06
H3C
H3C
O
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

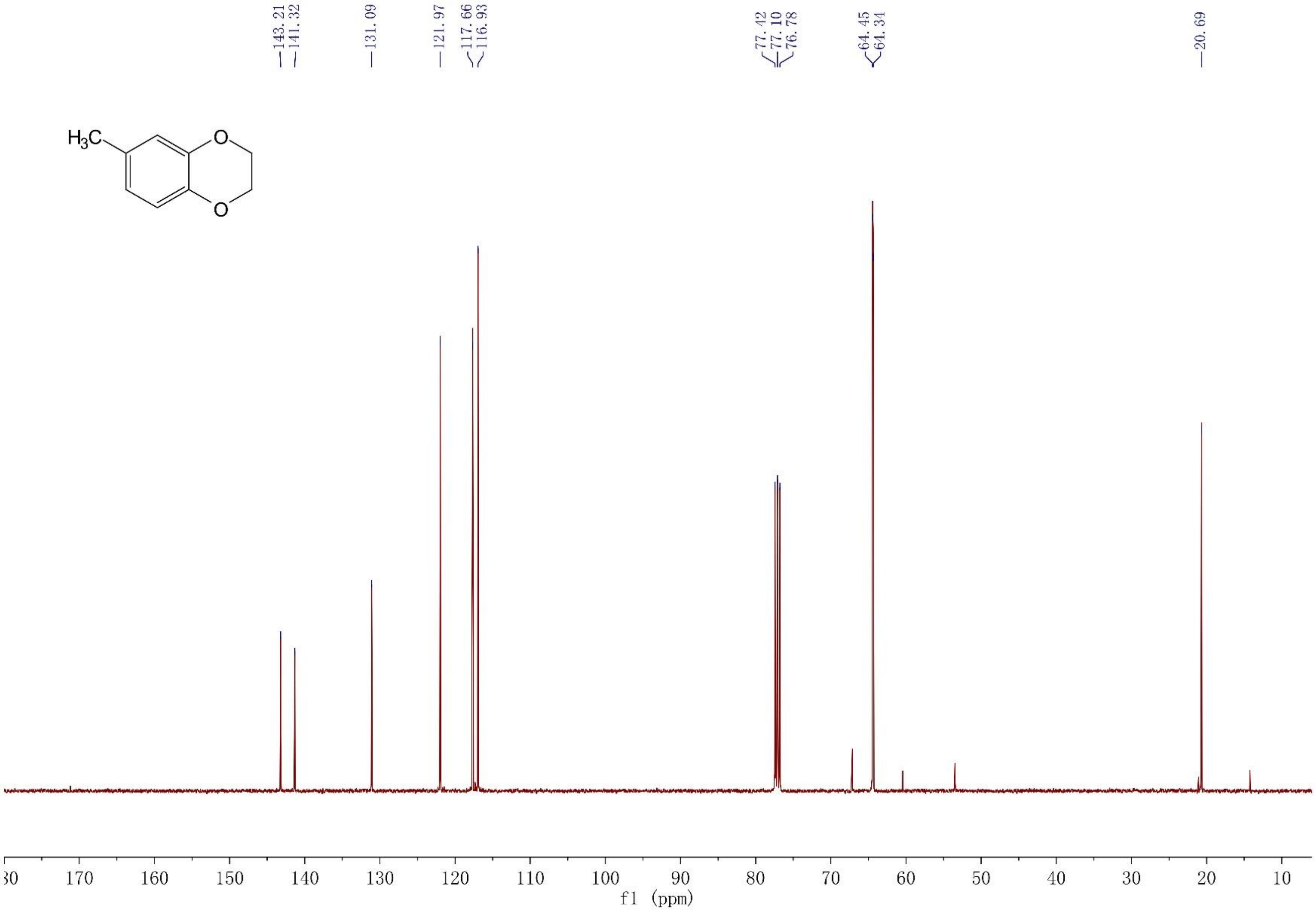

CH3
7.28
6.75
6.73
6.70
6.66
6.64
5.94
2.31
3.00
2.01
3.01
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



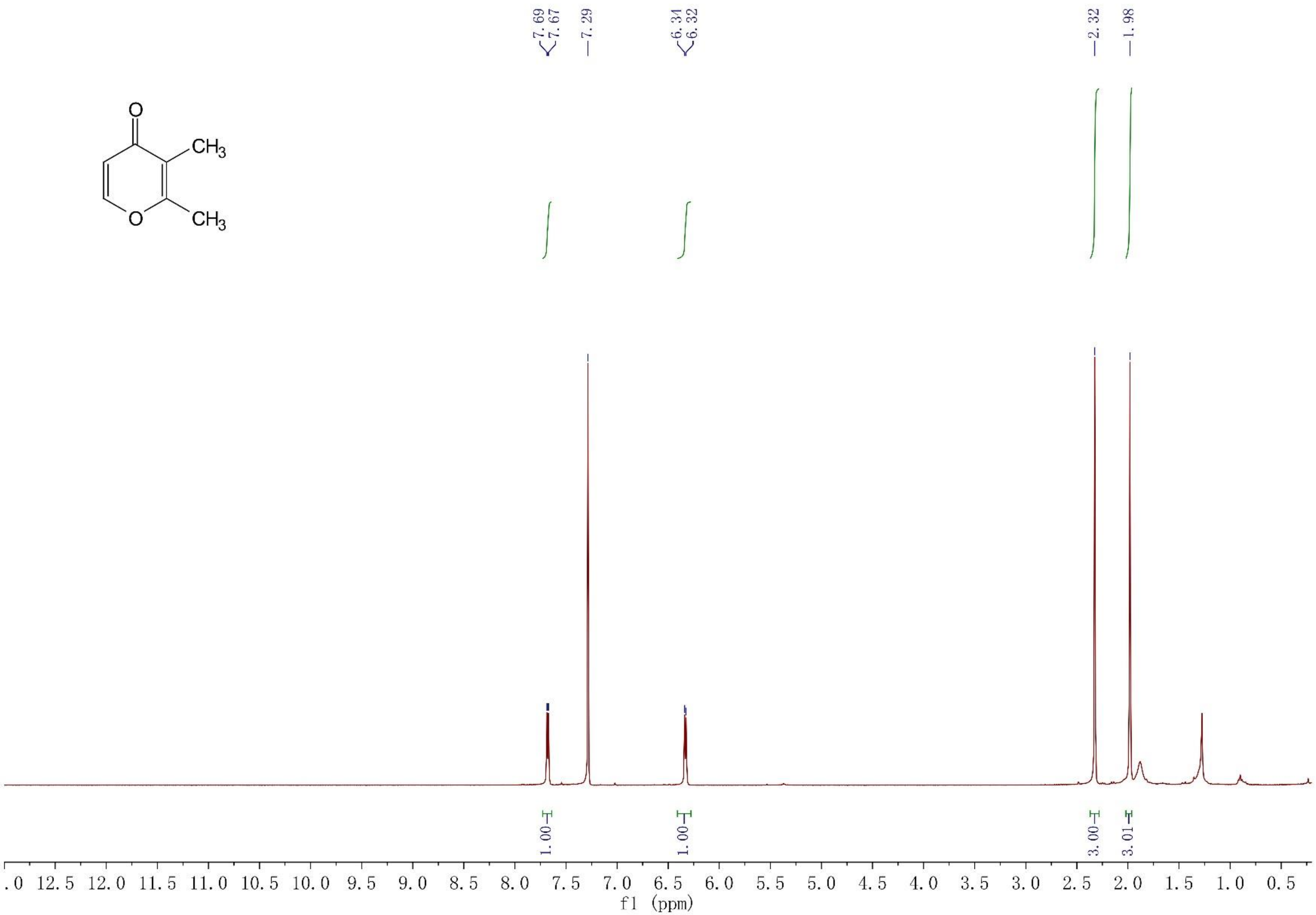
CH3
147.47
145.27
131.51
121.53
109.61
108.06
100.69
77.38
77.06
76.74
21.20
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



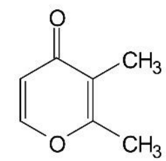
H3C
O
O
7.28
6.81
6.79
6.73
6.72
6.69
6.69
6.67
6.67
5.33
4.26
3.74
2.29
1.00
2.00
4.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



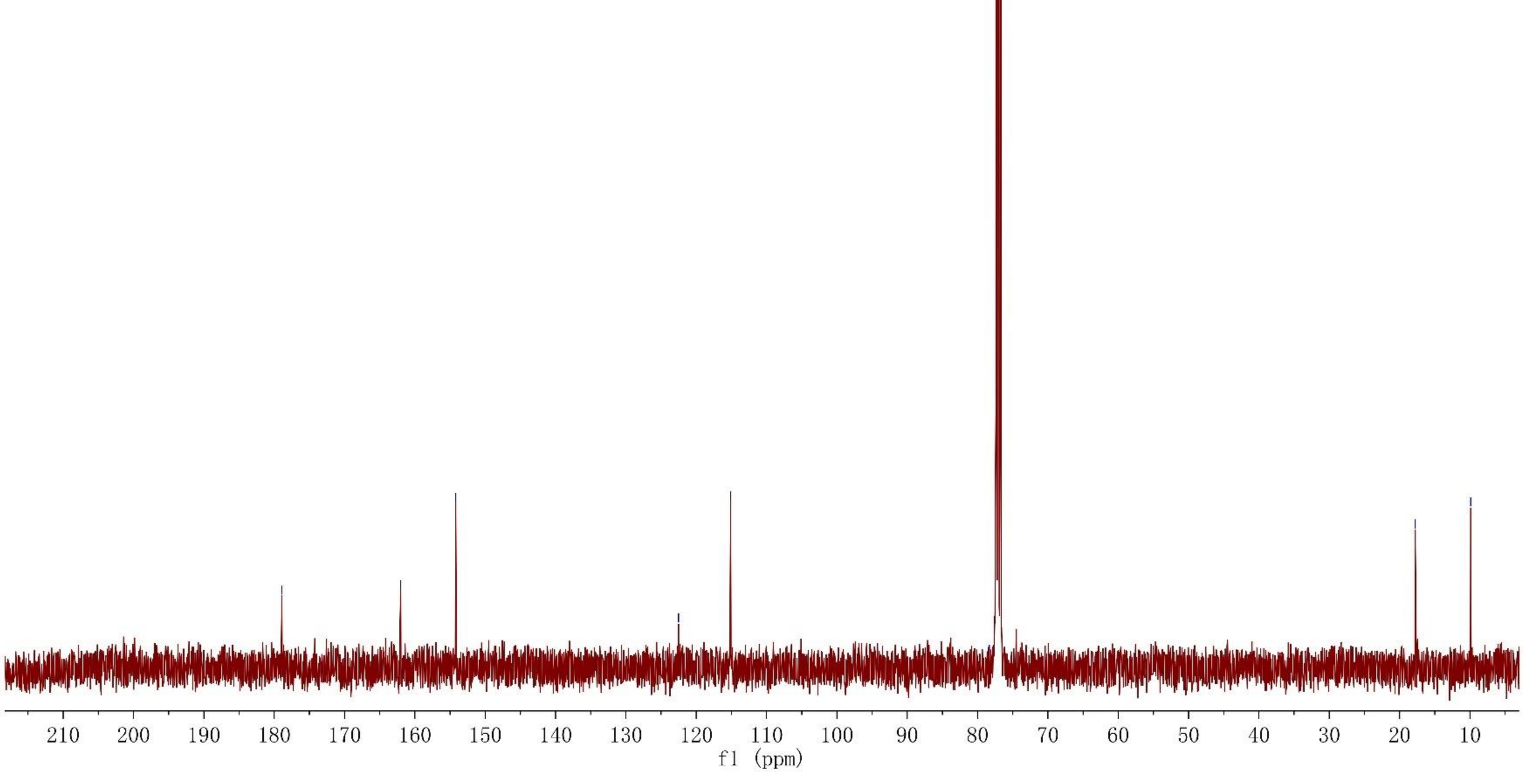
143.21
141.32
131.09
121.97
117.66
116.93
77.42
77.10
76.78
64.45
64.31
20.69
H3C
O
O
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



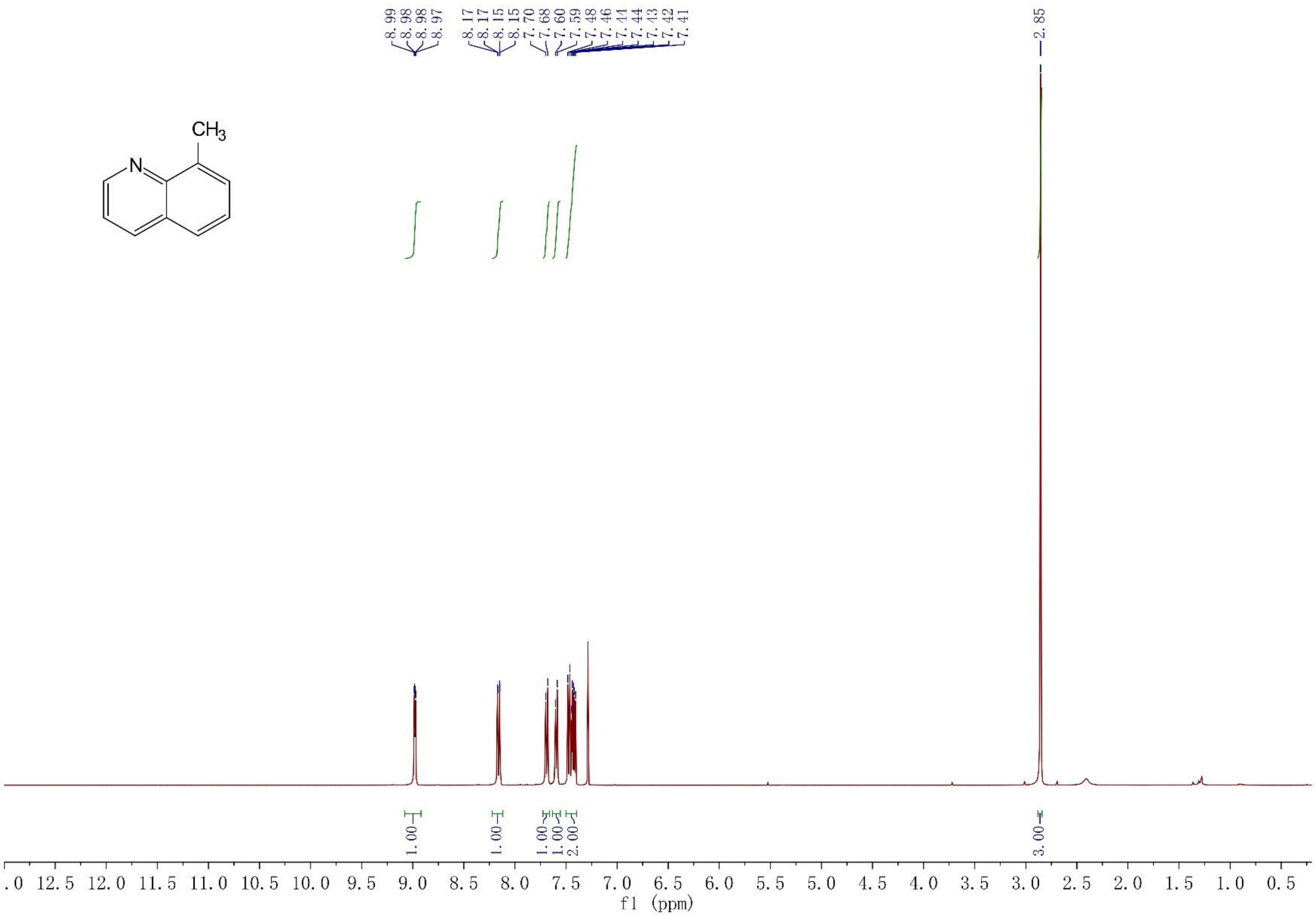
7.69
7.67
7.29
6.34
6.32
2.32
1.98
CH3
CH3
O
O
1.00
1.00
3.00
3.01
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



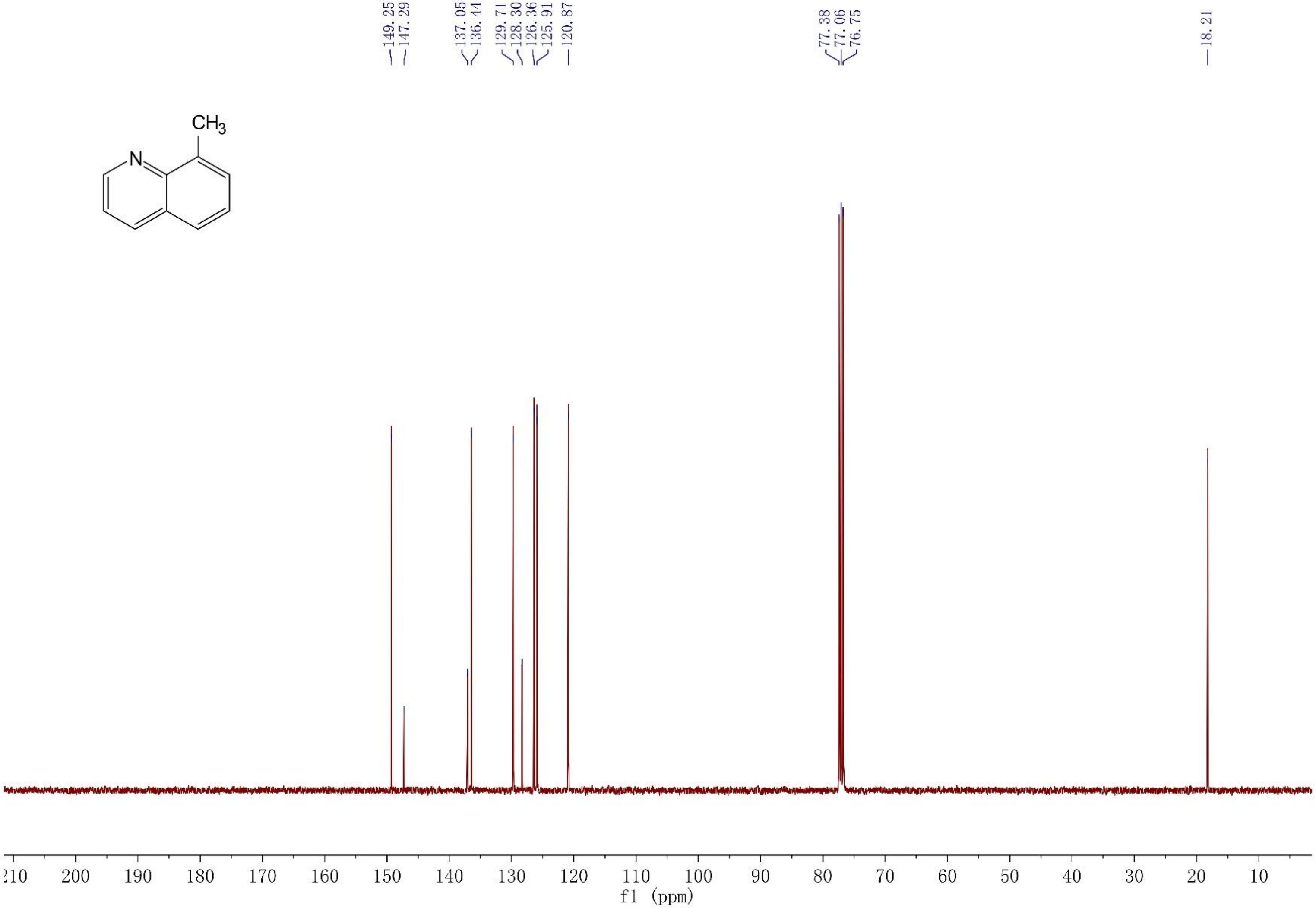
O
CH_3
O
CH_3



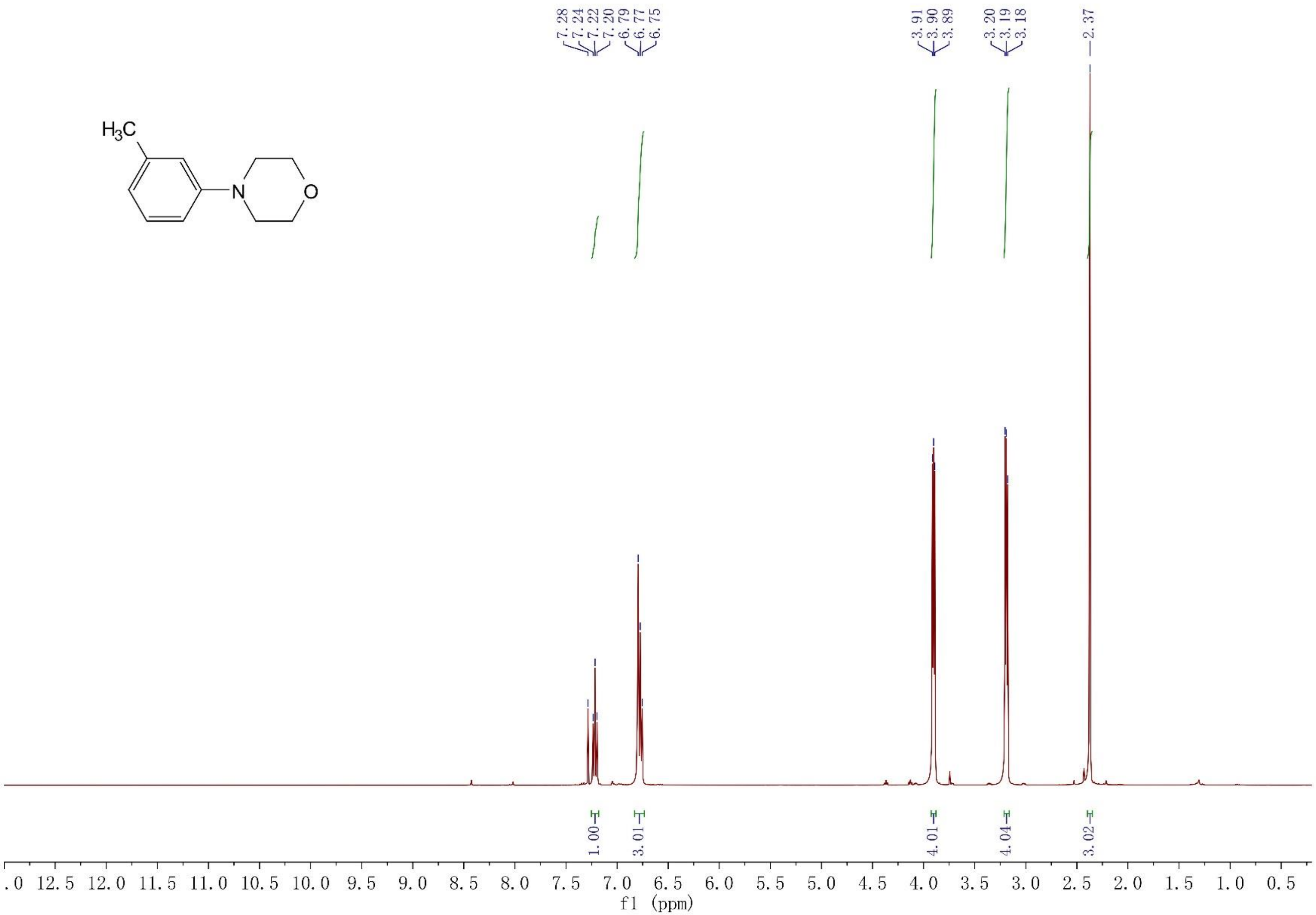
178.91
162.02
154.19
122.51
115.11
78.33
77.35
76.72
17.78
9.90
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



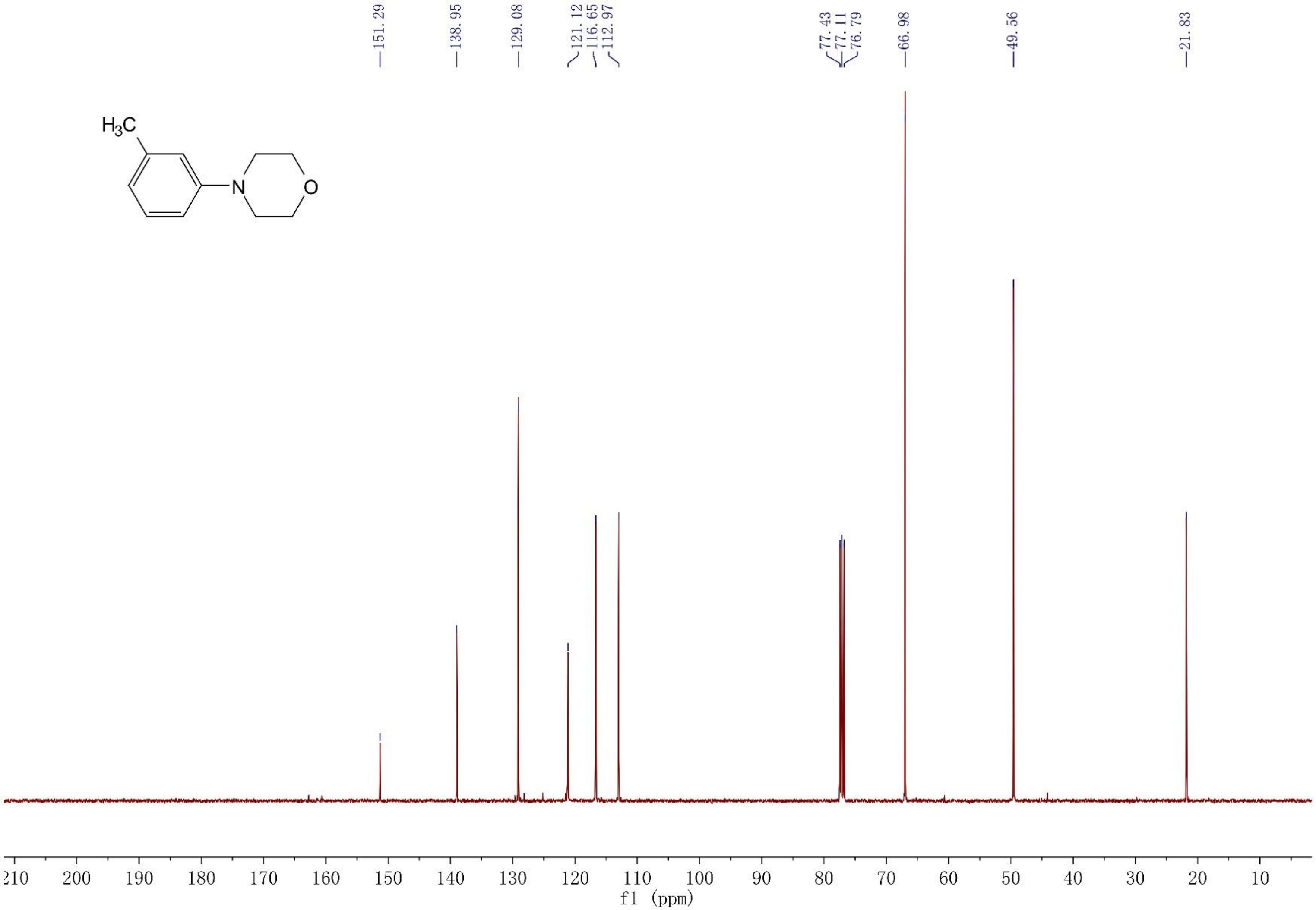
8.99
8.98
8.98
8.97
8.17
8.17
8.15
8.15
7.70
7.68
7.60
7.59
7.48
7.46
7.44
7.44
7.43
7.42
7.41
2.85
CH3
N
1.00
1.00
1.00
1.00
2.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



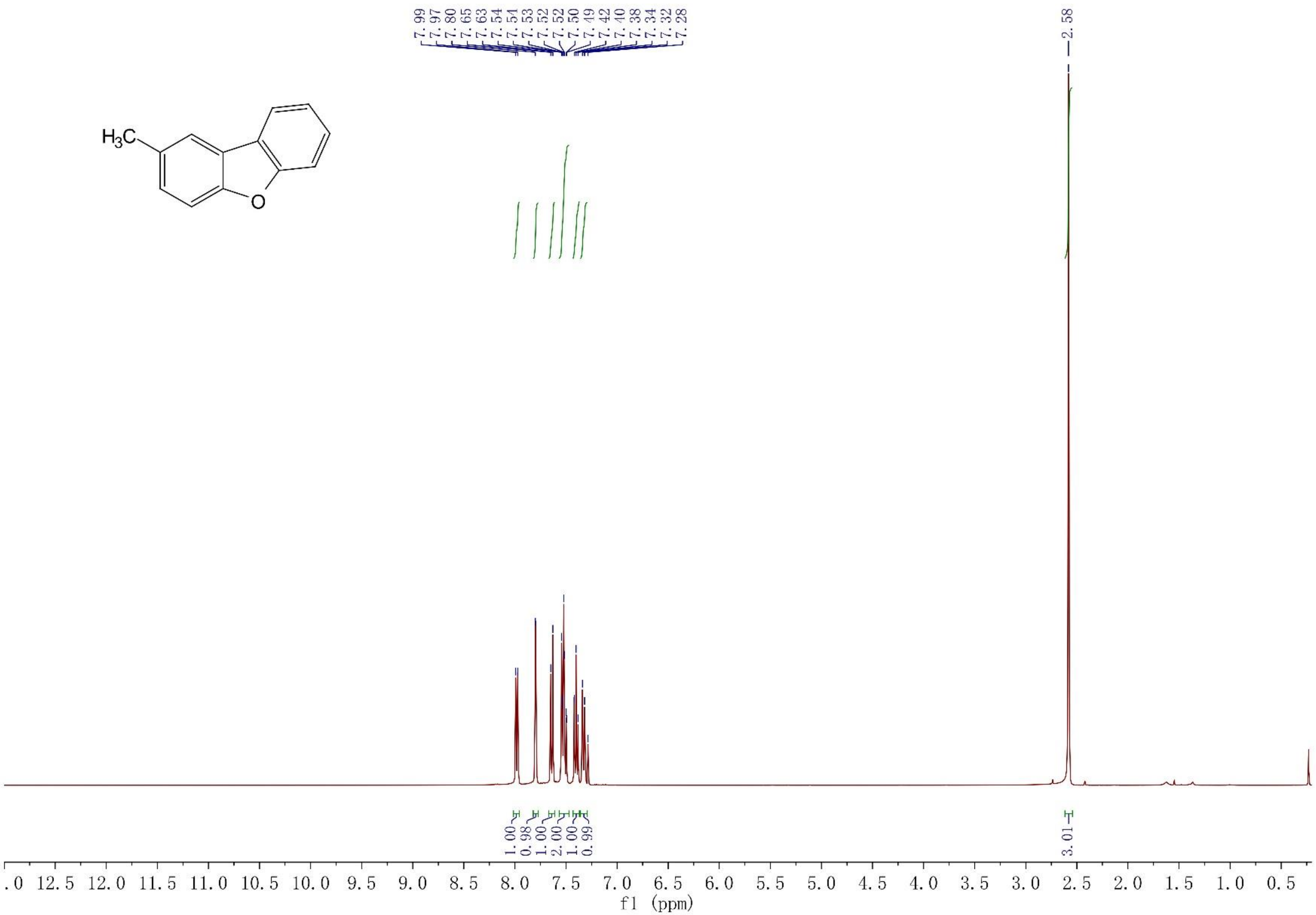
149.25
147.29
137.05
136.44
129.71
128.30
126.36
125.91
120.87
77.38
77.06
76.75
18.21
CH3
N
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

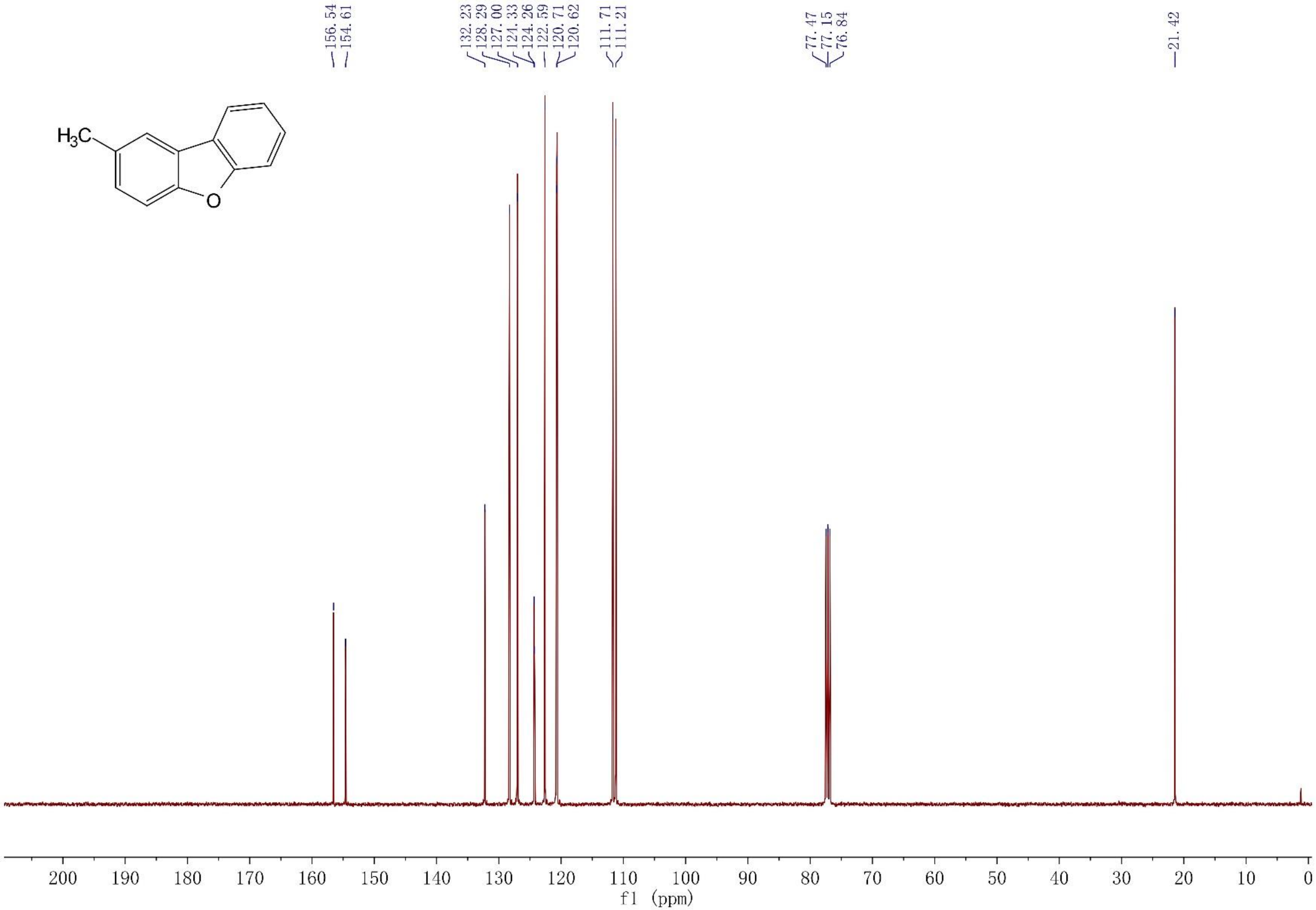


H3C
N
O
7.28
7.24
7.22
7.20
6.79
6.77
6.75
3.91
3.90
3.89
3.20
3.19
3.18
2.37
1.00
3.01
4.01
4.04
3.02
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

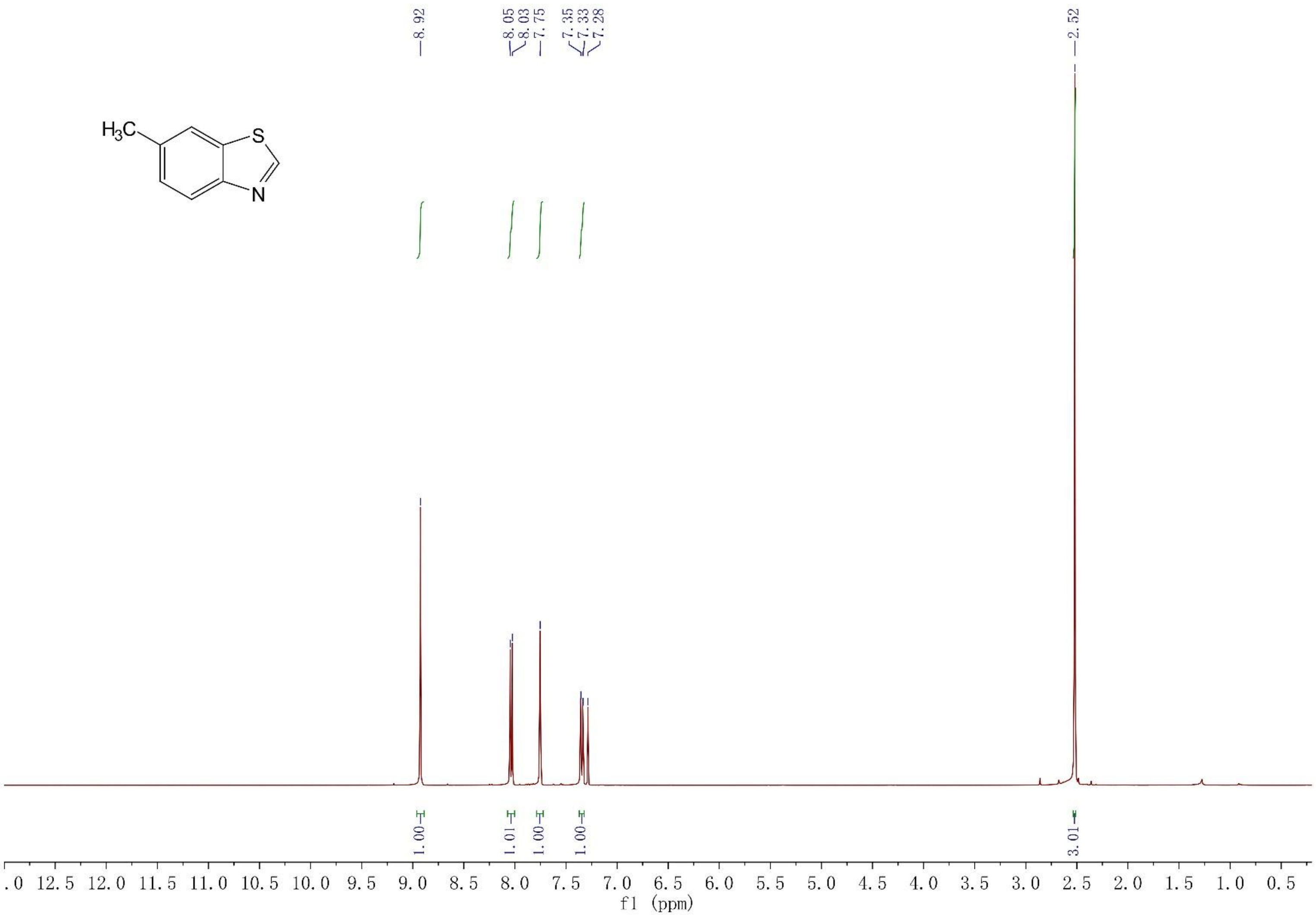


H3C
N
O
151.29
138.95
129.08
121.12
116.65
112.97
77.43
77.11
76.79
66.98
49.56
21.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

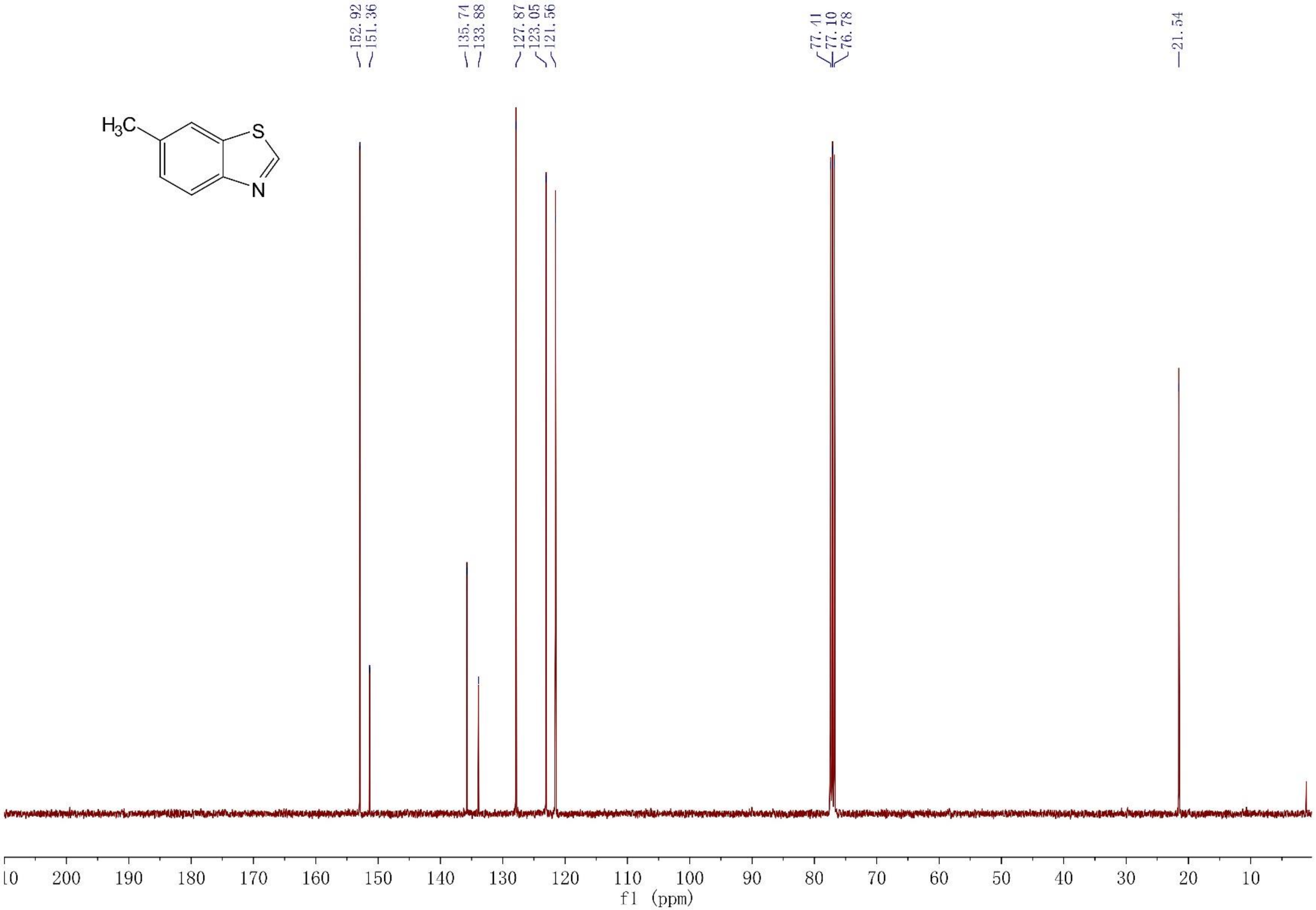

H3C
O
7.99
7.97
7.80
7.65
7.63
7.54
7.54
7.53
7.52
7.52
7.50
7.49
7.42
7.40
7.38
7.34
7.32
7.28
2.58
1.00
0.98
1.00
2.00
1.00
0.99
3.01
f1 (ppm)



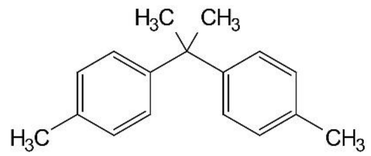
156.54
154.61
132.23
128.29
127.00
124.33
124.26
122.59
120.71
120.62
111.71
111.21
77.47
77.15
76.84
21.42
H_3C
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

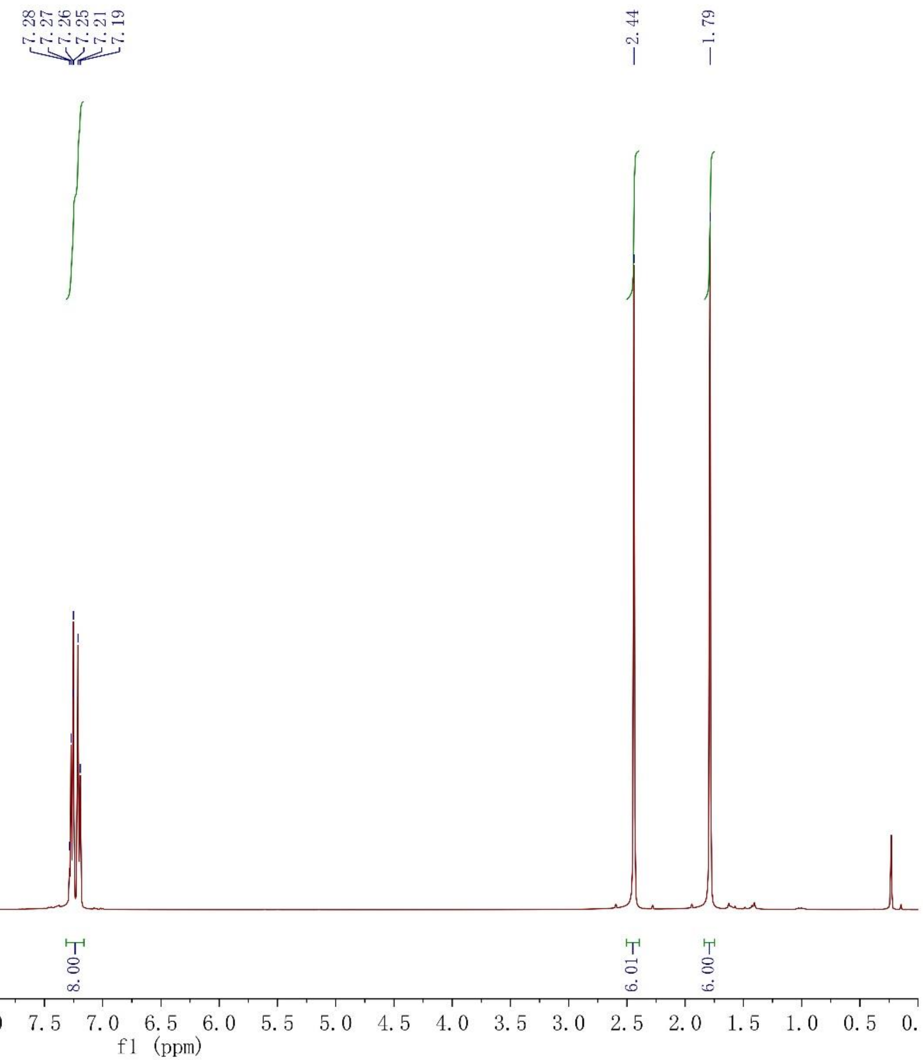


H3C
S
N
8.92
8.05
8.03
7.75
7.35
7.33
7.28
2.52
1.00
1.01
1.00
1.00
3.01
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

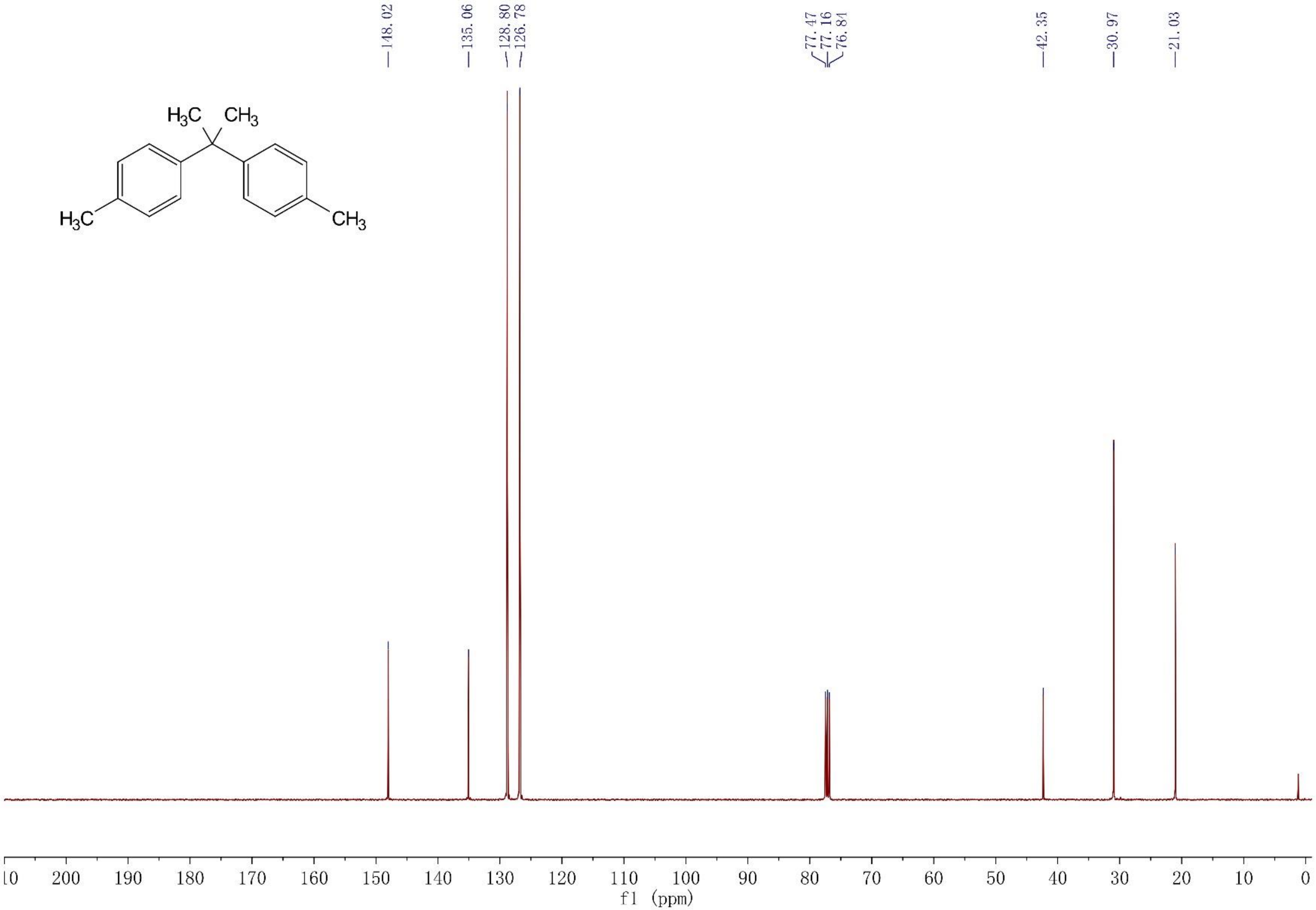


152.92
151.36
135.74
133.88
127.87
123.05
121.56
77.41
77.10
76.78
21.54
H3C
S
N
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

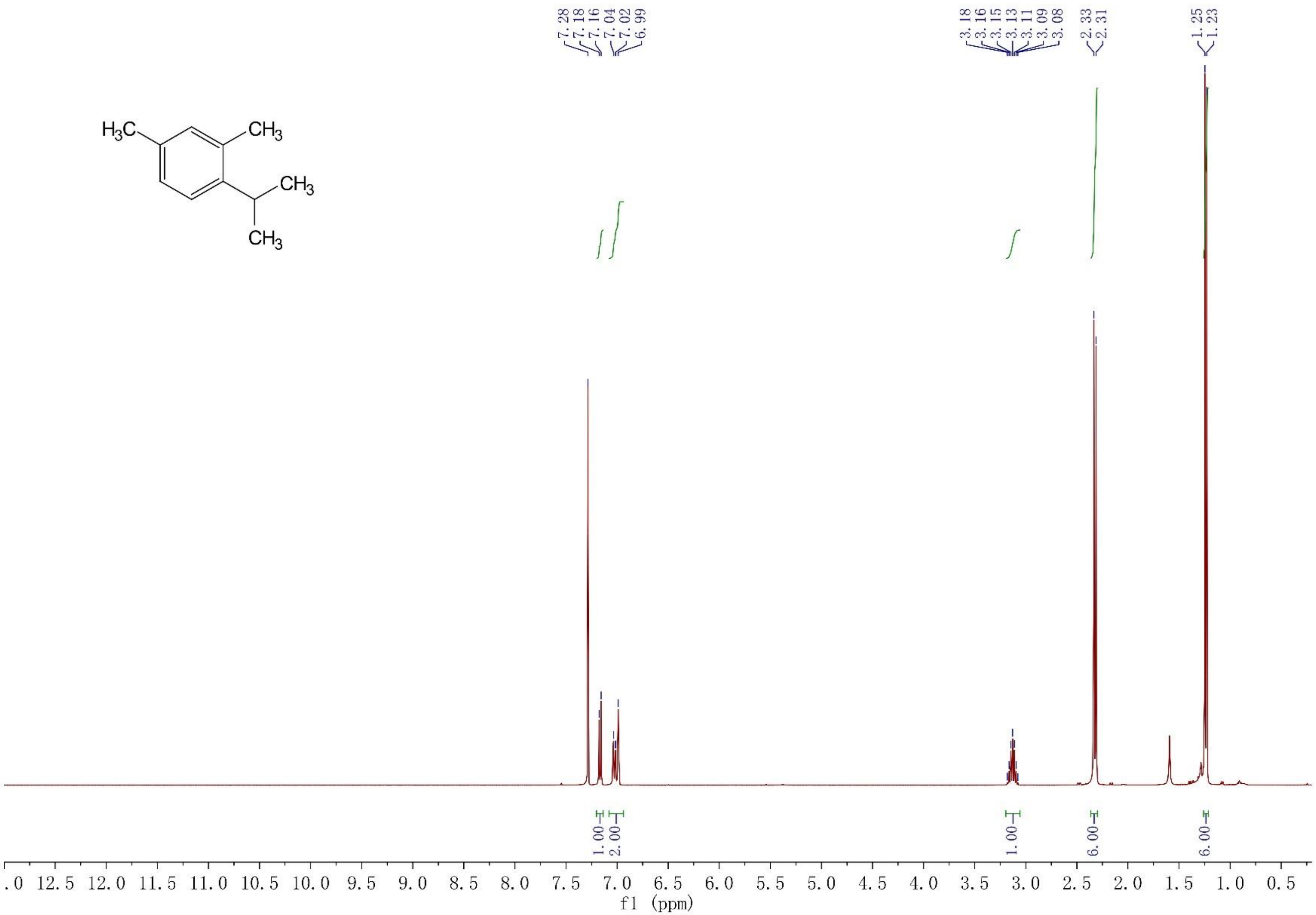




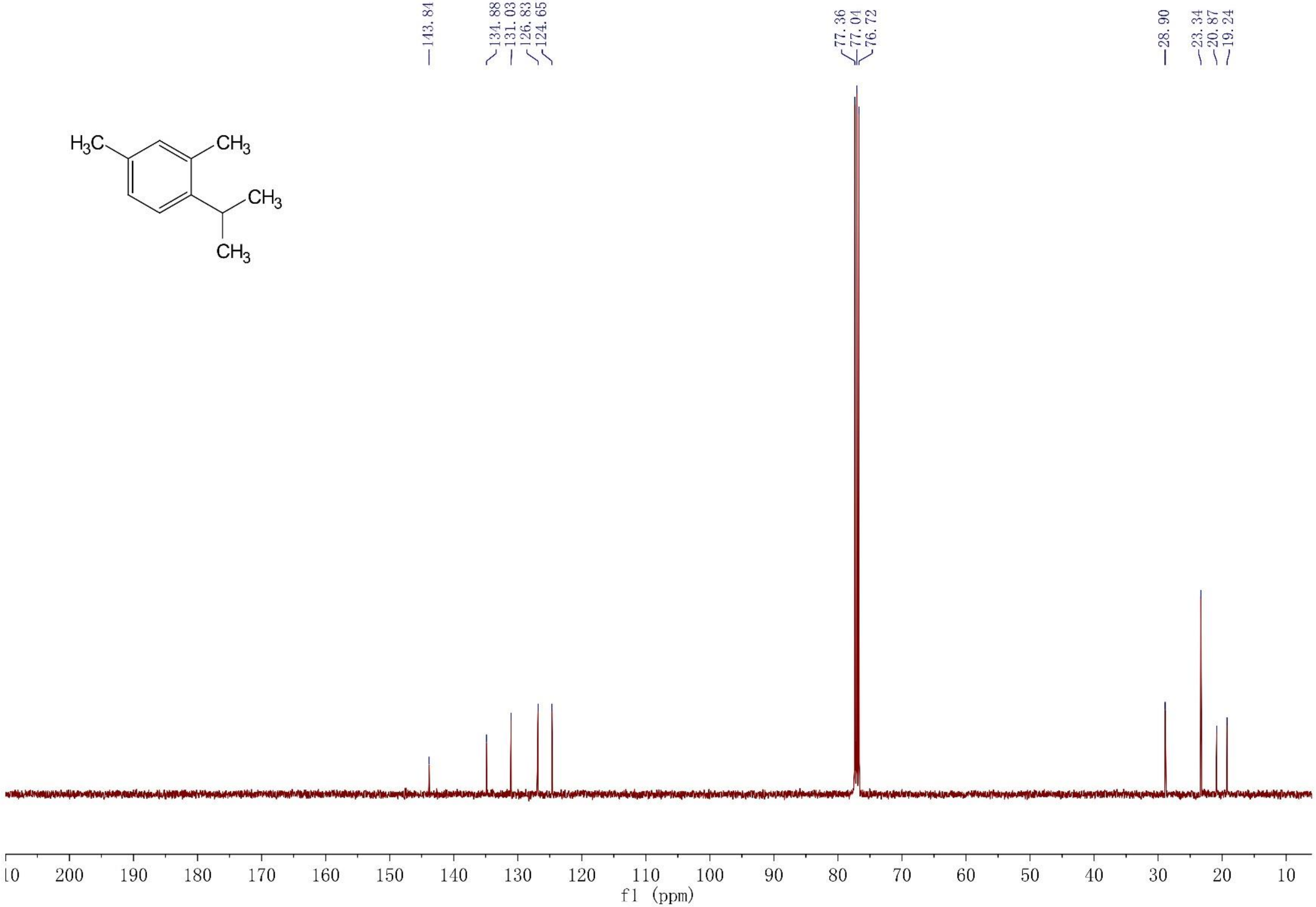
7.28
7.27
7.26
7.25
7.21
7.19
2.44
1.79
8.00
6.01
6.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.
f1 (ppm)



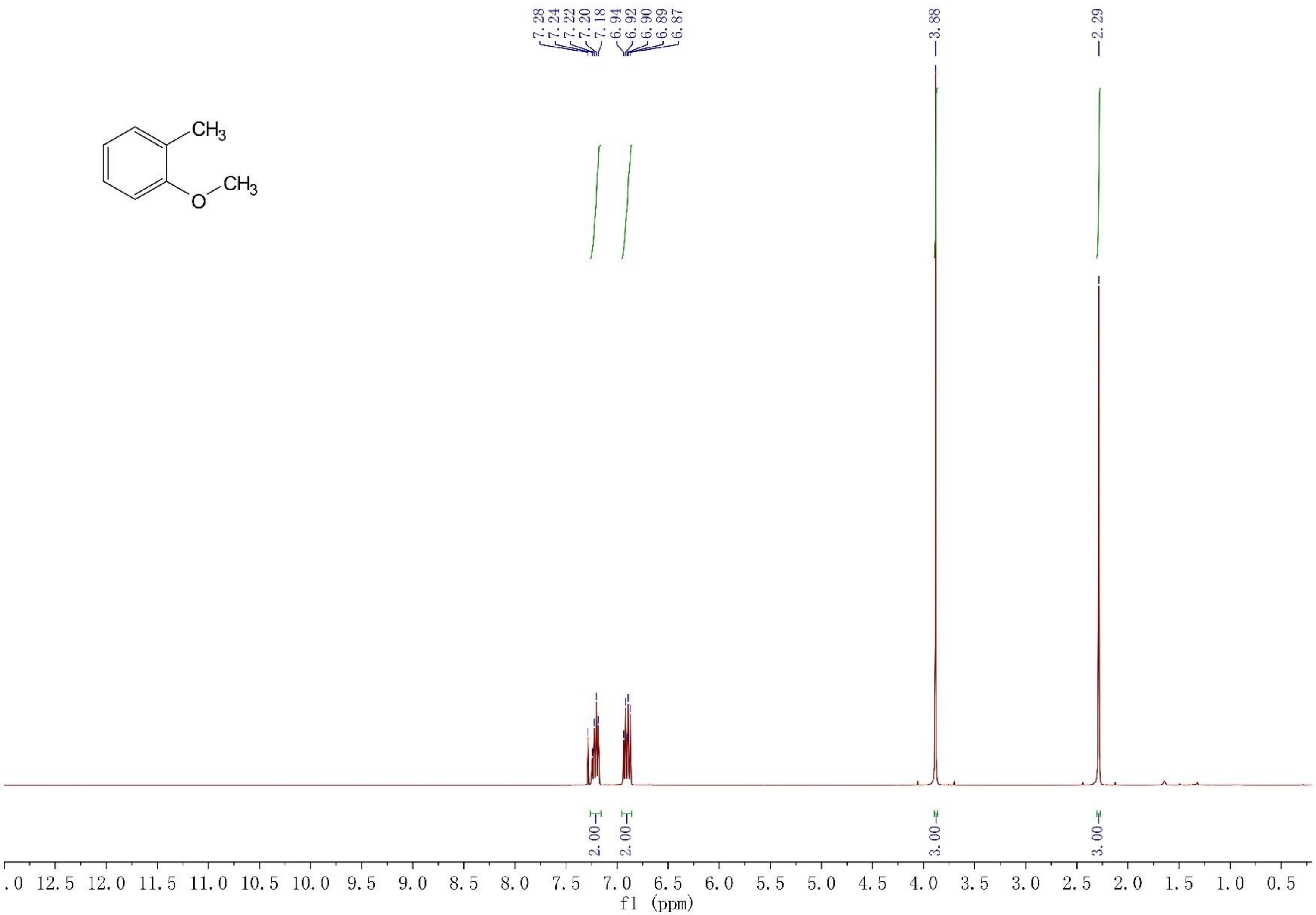
148.02
135.06
128.80
126.78
77.47
77.16
76.84
42.35
30.97
21.03
H_3C CH_3
H_3C
CH_3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



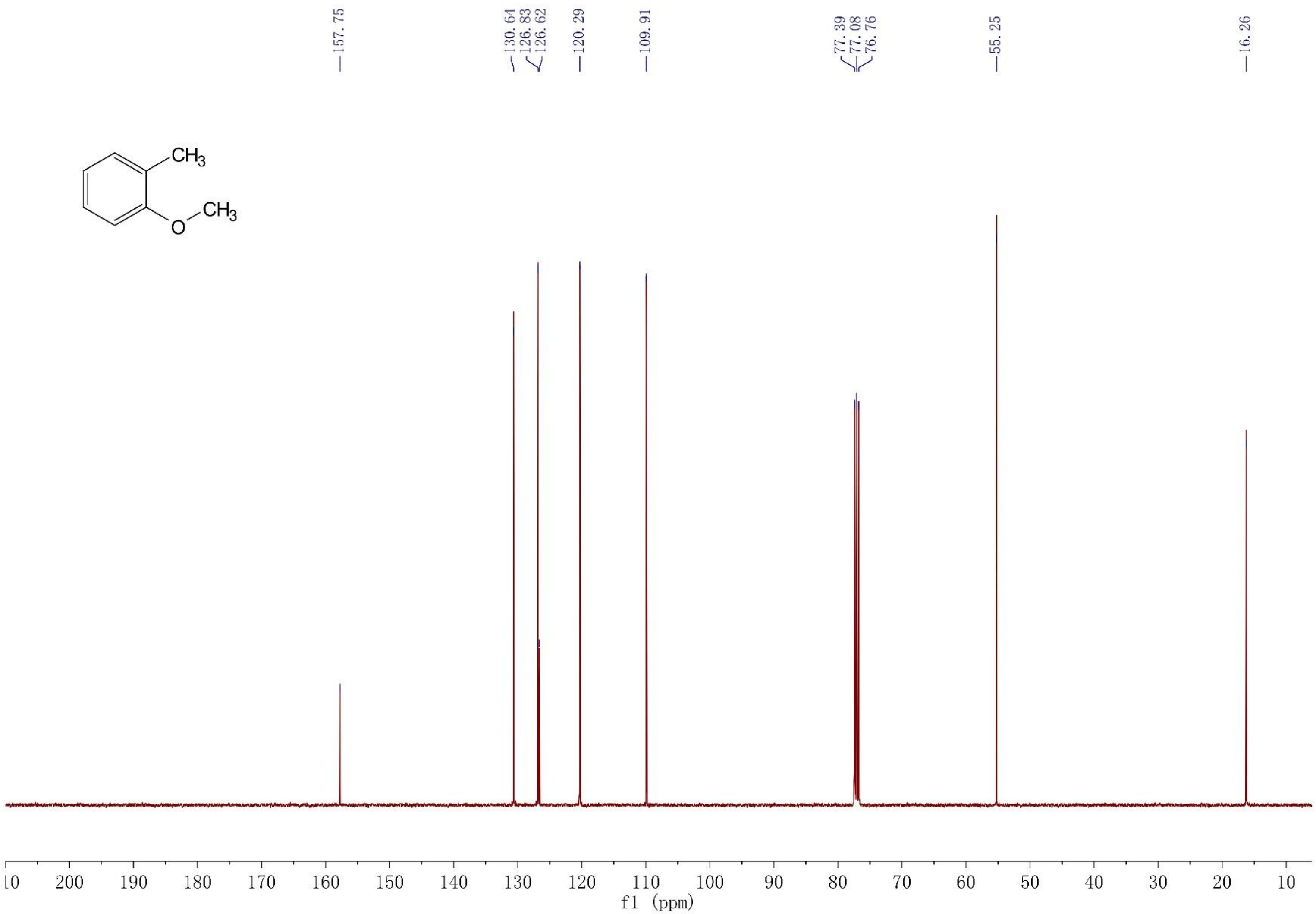
7.28
7.18
7.16
7.04
7.02
6.99
3.18
3.16
3.15
3.13
3.11
3.09
3.08
2.33
2.31
1.25
1.23
1.00
2.00
1.00
6.00
6.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

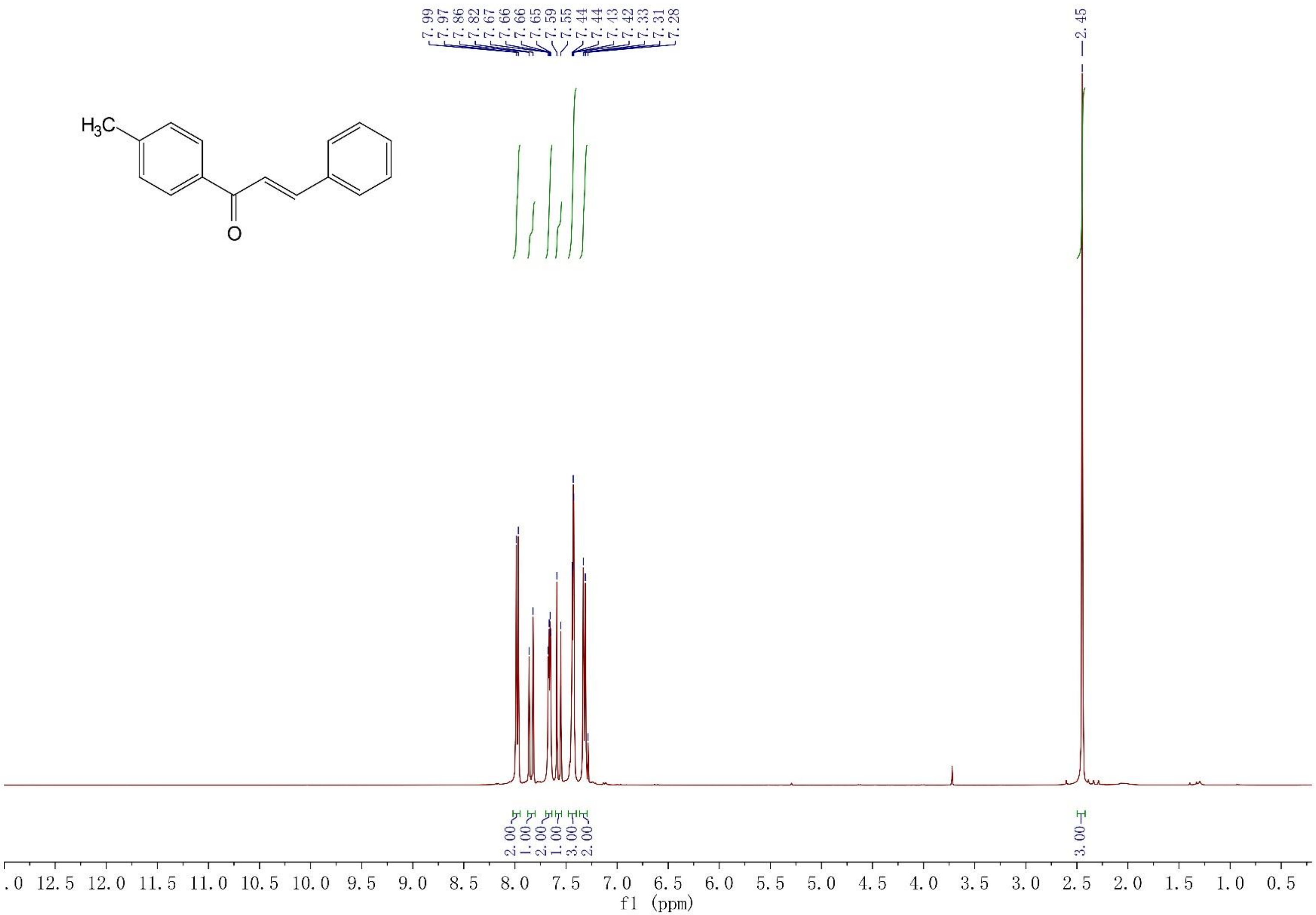


H_3C
CH_3
CH_3
CH_3
143.84
134.88
131.03
126.83
124.65
77.36
77.04
76.72
28.90
23.34
20.87
19.24
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

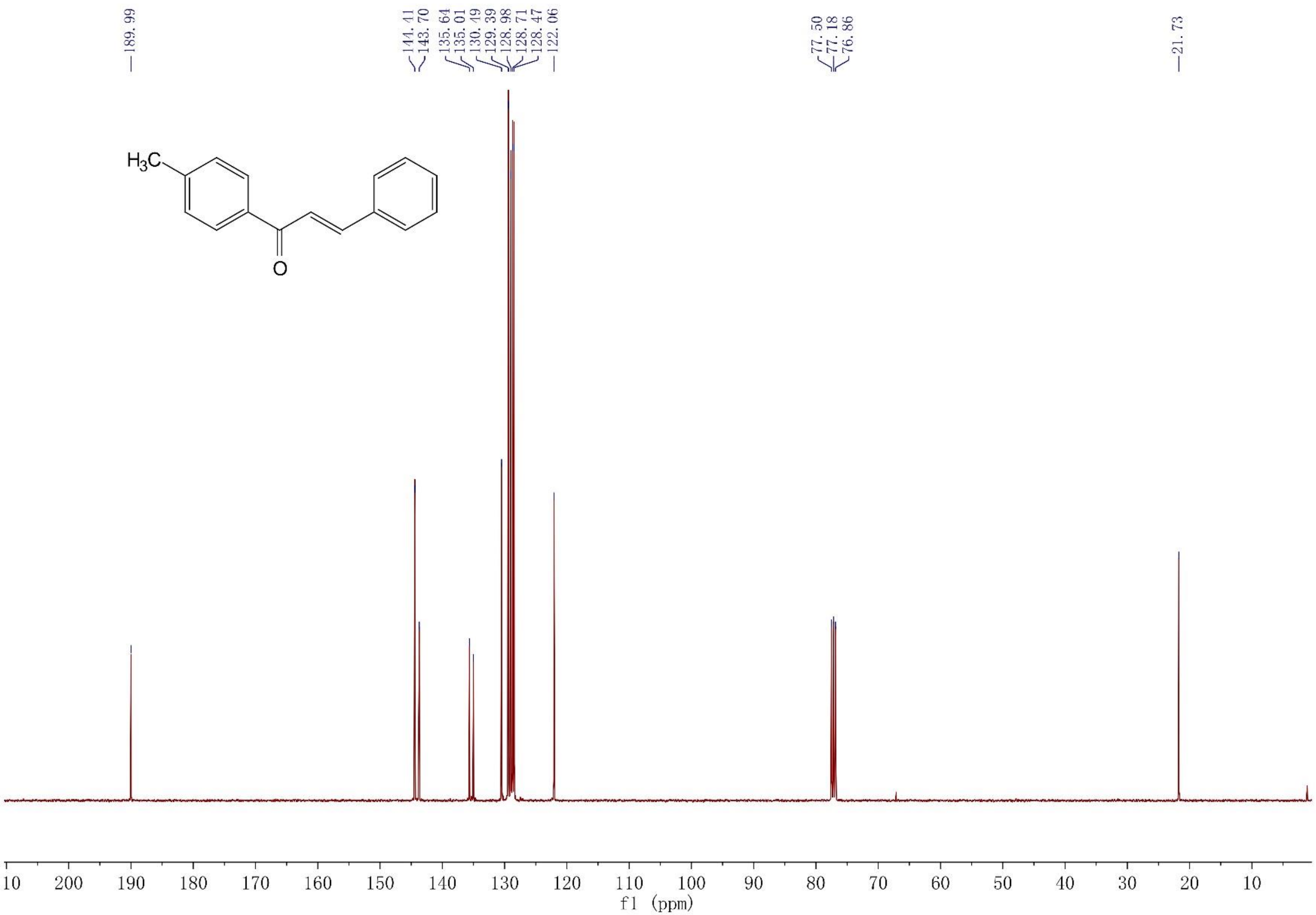


CH3
O
CH3
7.28
7.24
7.22
7.20
7.18
6.94
6.92
6.90
6.89
6.87
3.88
2.29
2.00
2.00
3.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

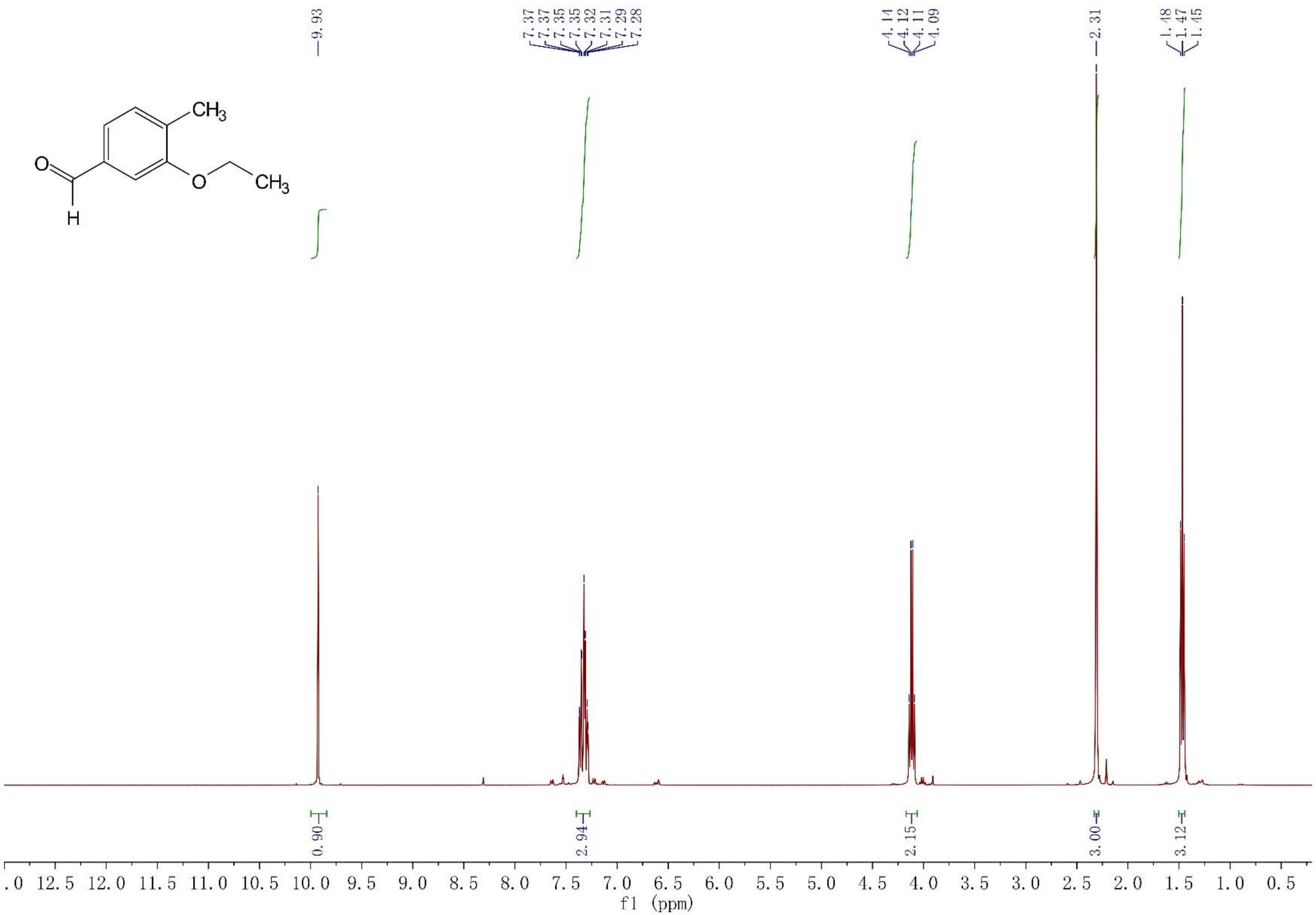

157.75
130.64
126.83
126.62
120.29
109.91
77.39
77.08
76.76
55.25
16.26
CH3
O
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



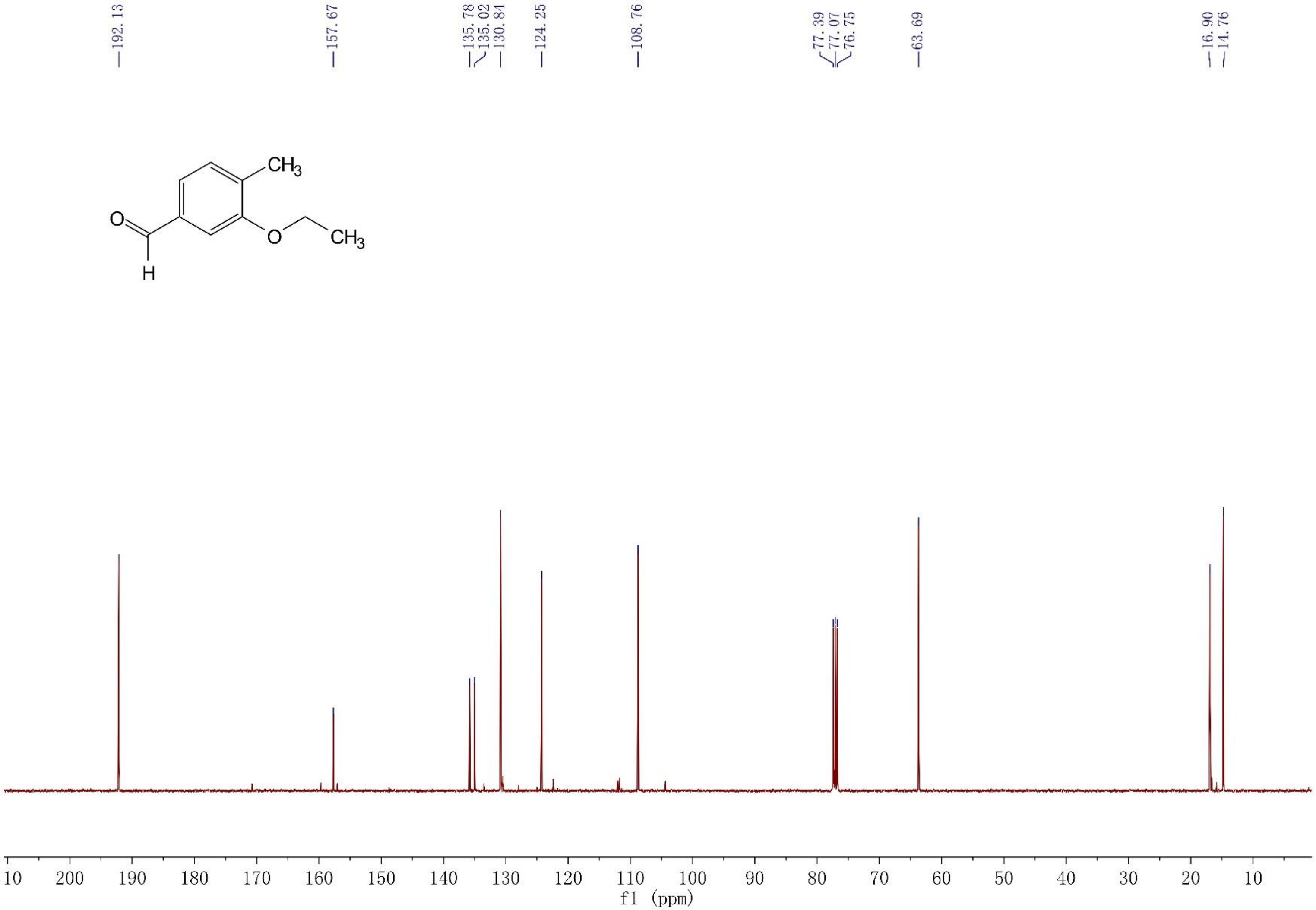
H3C
O
7.99
7.97
7.86
7.82
7.67
7.66
7.66
7.65
7.59
7.55
7.44
7.44
7.43
7.42
7.33
7.31
7.28
2.45
2.00
1.00
2.00
1.00
3.00
2.00
3.00
f1 (ppm)



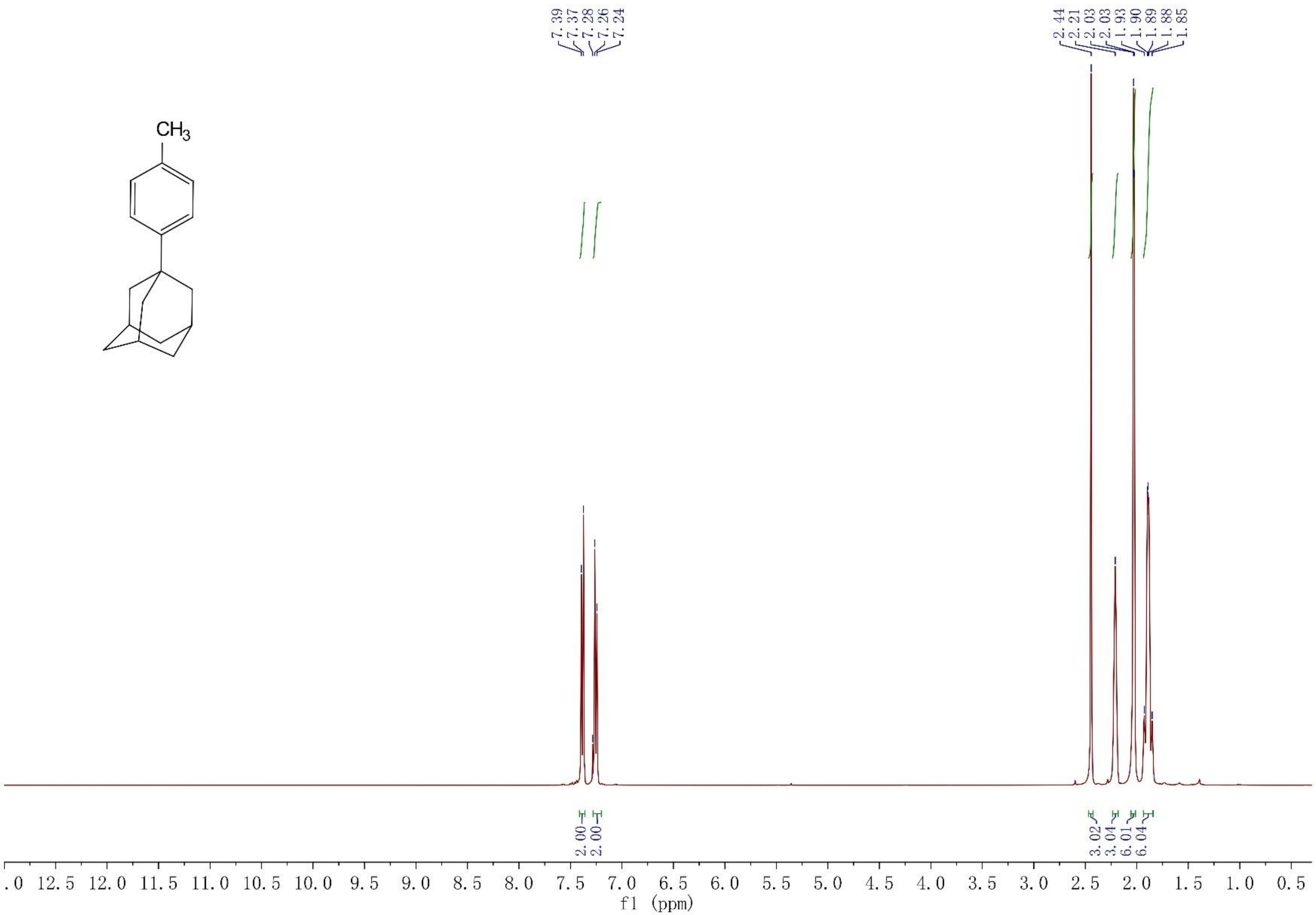
189.99
144.41
143.70
135.64
135.01
130.49
129.39
128.98
128.71
128.47
122.06
77.50
77.18
76.86
21.73
H3C
O
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



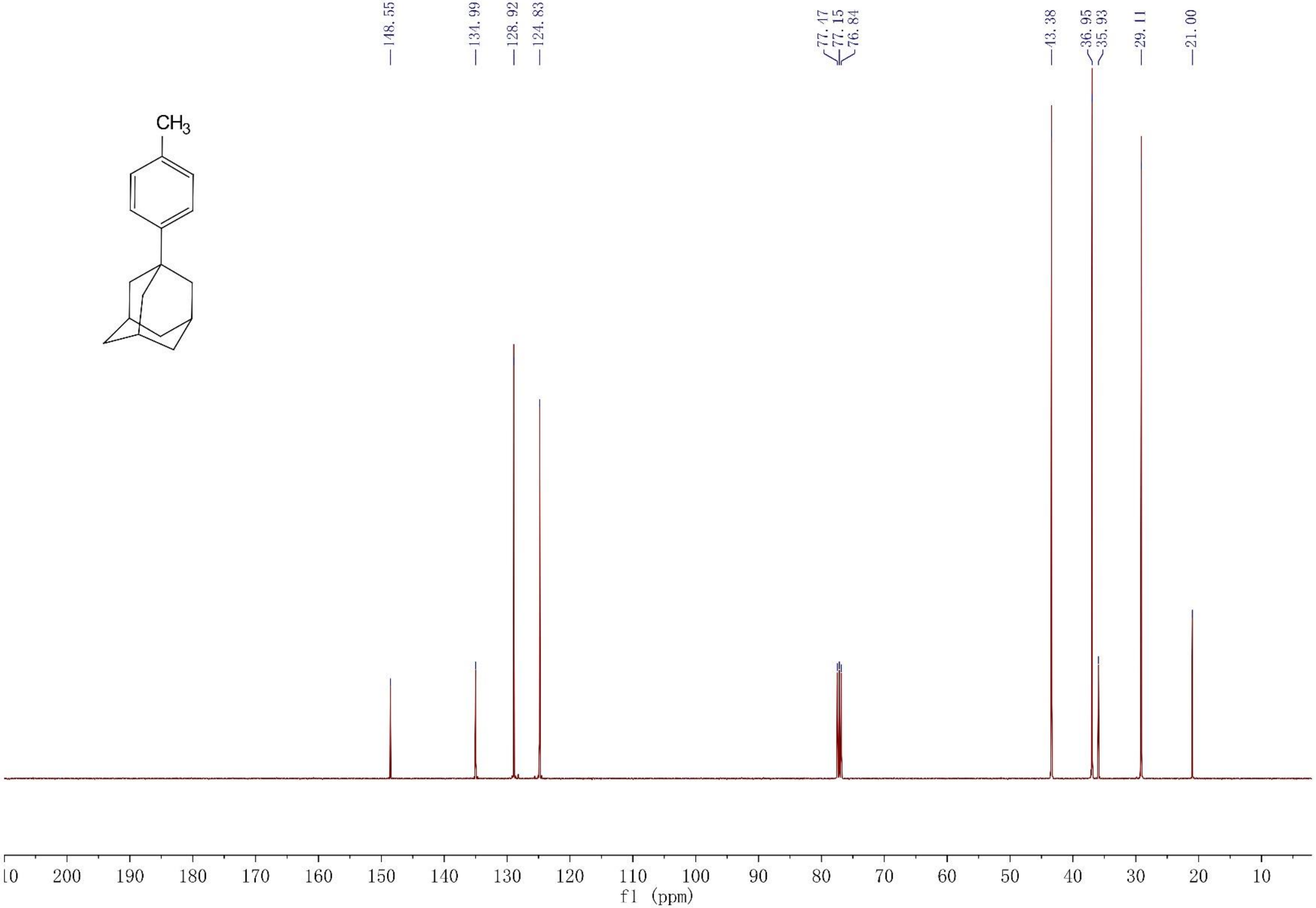
CH3
O
O
CH3
H
9.93
7.37
7.37
7.35
7.35
7.32
7.31
7.29
7.28
4.14
4.12
4.11
4.09
2.31
1.48
1.47
1.45
0.90
2.94
2.15
3.00
3.12
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

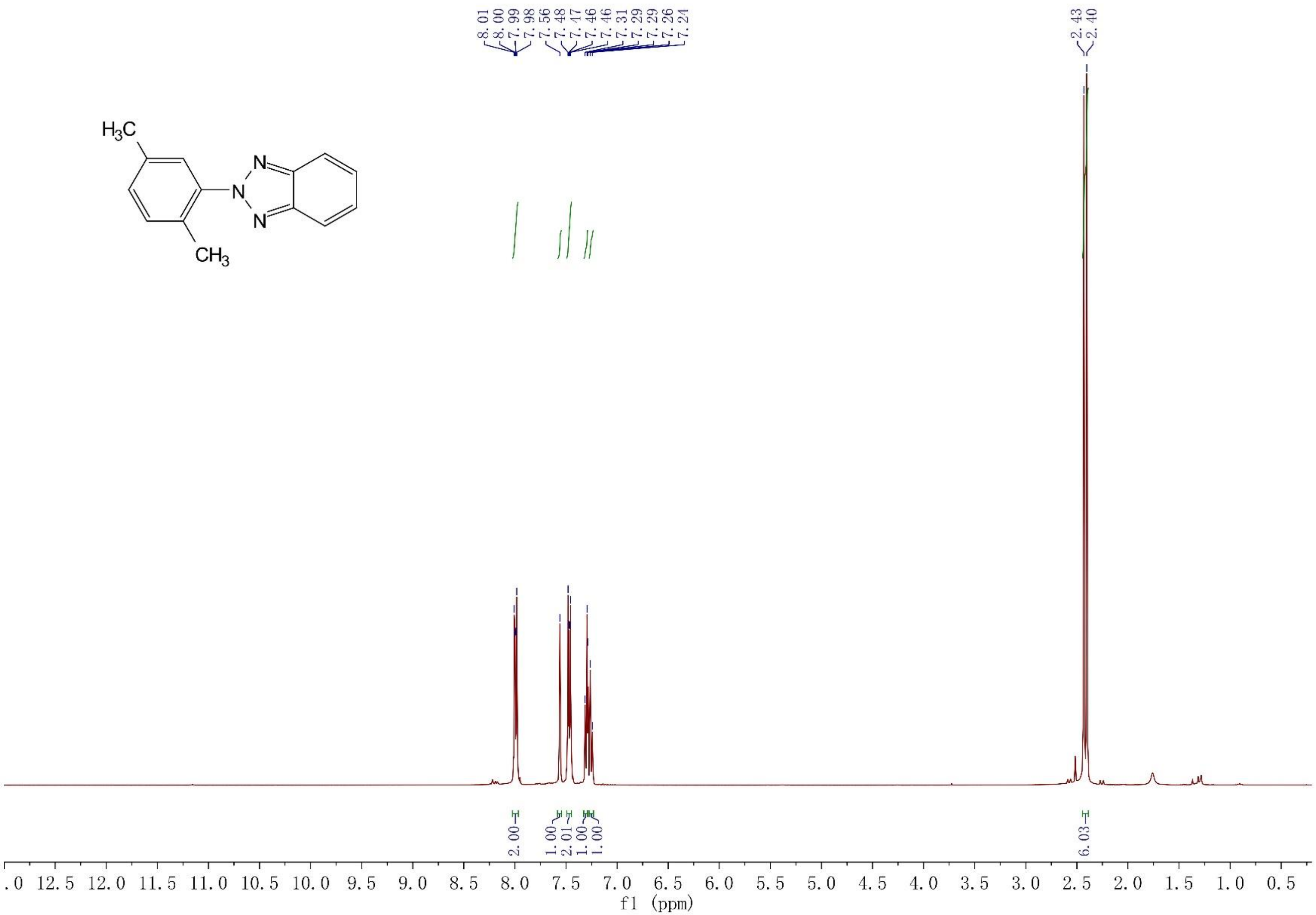


192.13
157.67
135.78
135.02
130.81
124.25
108.76
77.39
77.07
76.75
63.69
16.90
14.76
CH3
O
O
CH3
H
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

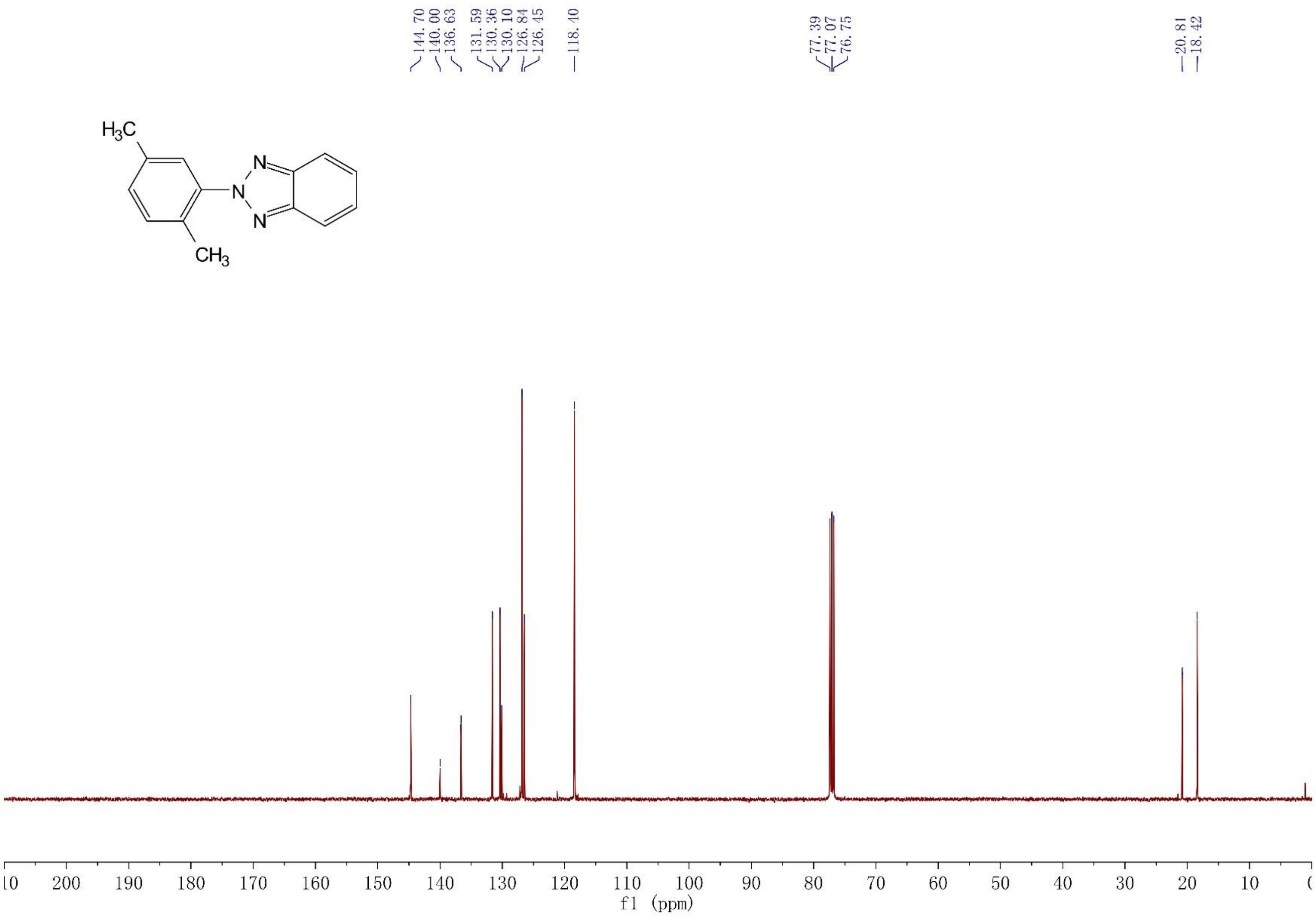


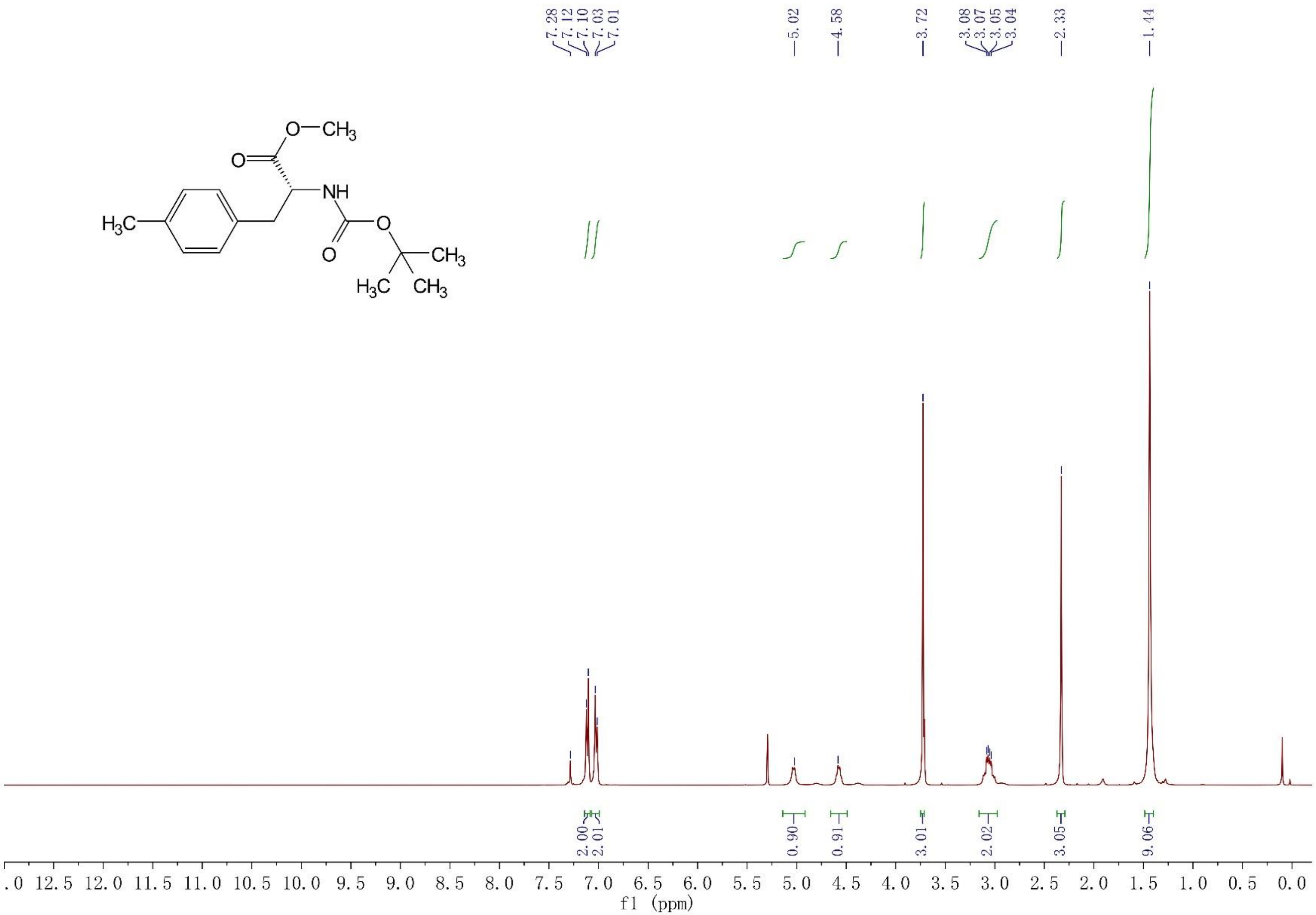
CH3
7.39
7.37
7.28
7.26
7.24
2.44
2.21
2.03
2.03
1.93
1.90
1.89
1.88
1.85
2.00
2.00
3.02
3.04
6.01
6.04
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


CH3
148.55
134.99
128.92
124.83
77.47
77.15
76.84
43.38
36.95
35.93
29.11
21.00
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

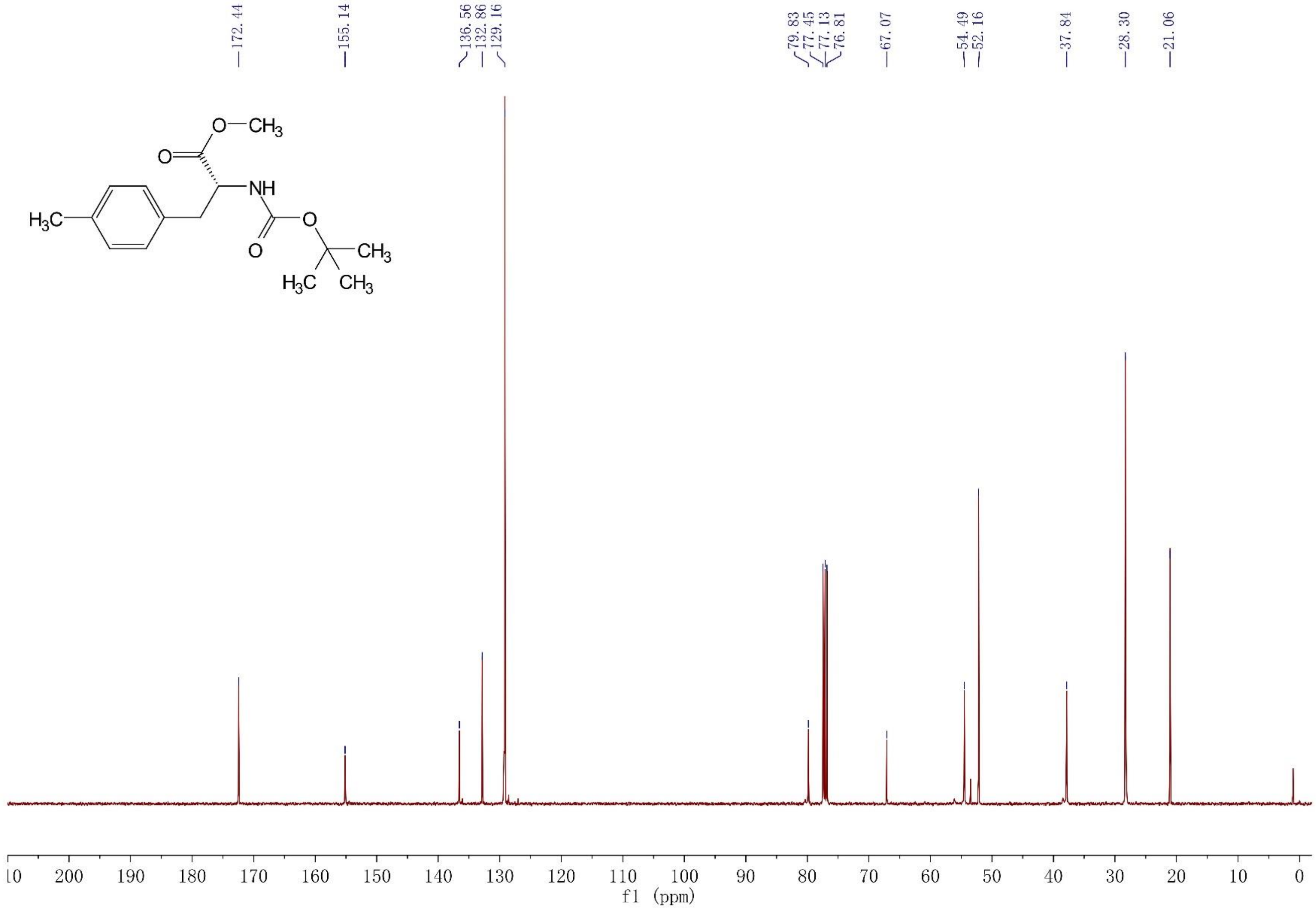


8.01
8.00
7.99
7.98
7.56
7.48
7.47
7.46
7.46
7.31
7.29
7.29
7.26
7.21
2.43
2.40
H3C
N
N
N
CH3
2.00
1.00
2.01
1.00
1.00
6.03
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

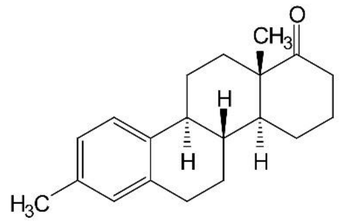

144.70
140.00
136.63
131.59
130.36
130.10
126.84
126.45
118.40
77.39
77.07
76.75
20.81
18.42
H_3C
N
N
N
CH_3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

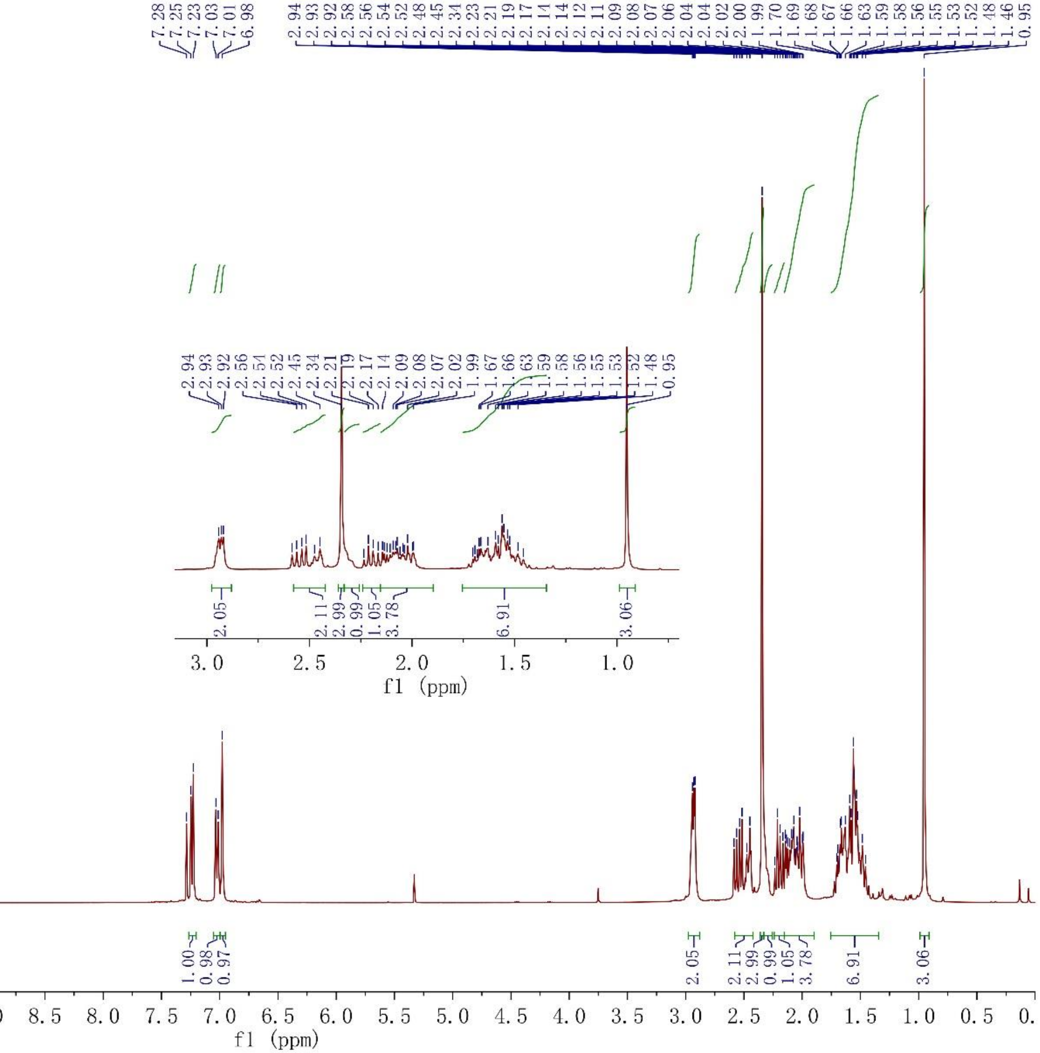


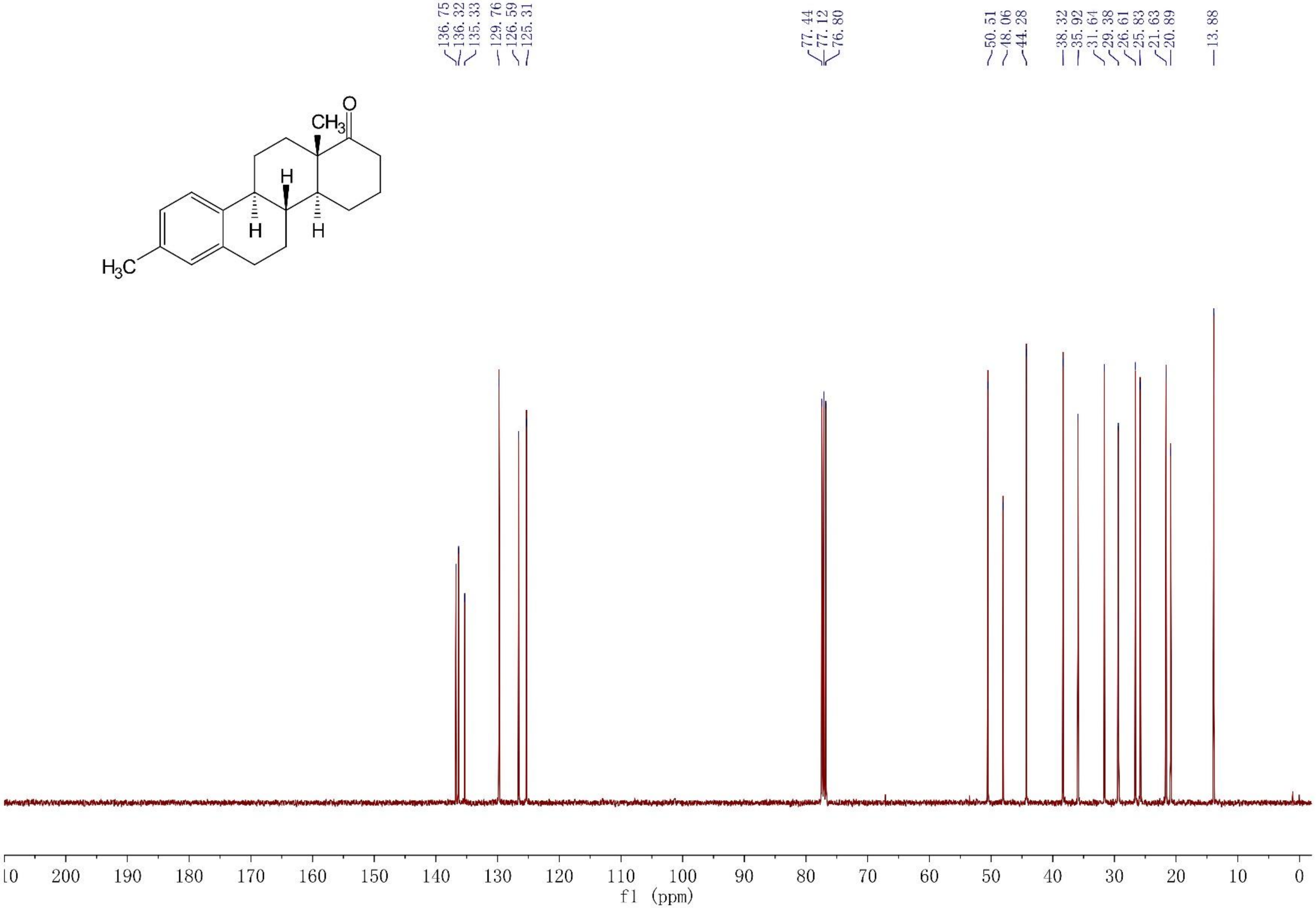
7.28
7.12
7.10
7.03
7.01
5.02
4.58
3.72
3.08
3.07
3.05
3.04
2.33
1.44
O
CH3
O
NH
H3C
O
O
CH3
H3C
CH3
2.00
2.01
0.90
0.91
3.01
2.02
3.05
9.06
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

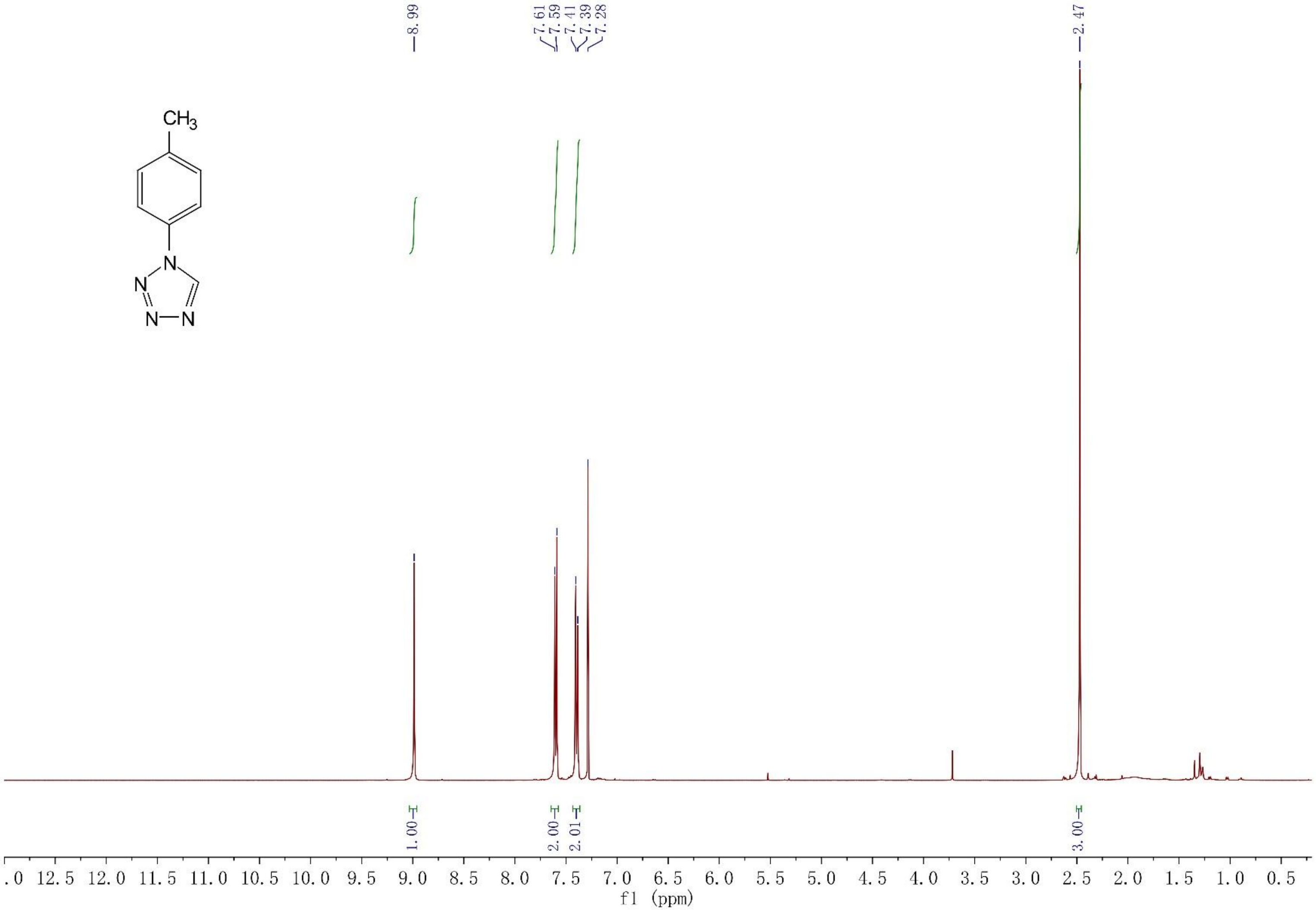


172.44
155.14
136.56
132.86
129.16
79.83
77.45
77.13
76.81
67.07
54.49
52.16
37.81
28.30
21.06
O—CH3
O
NH
H3C
O
O
CH3
H3C
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

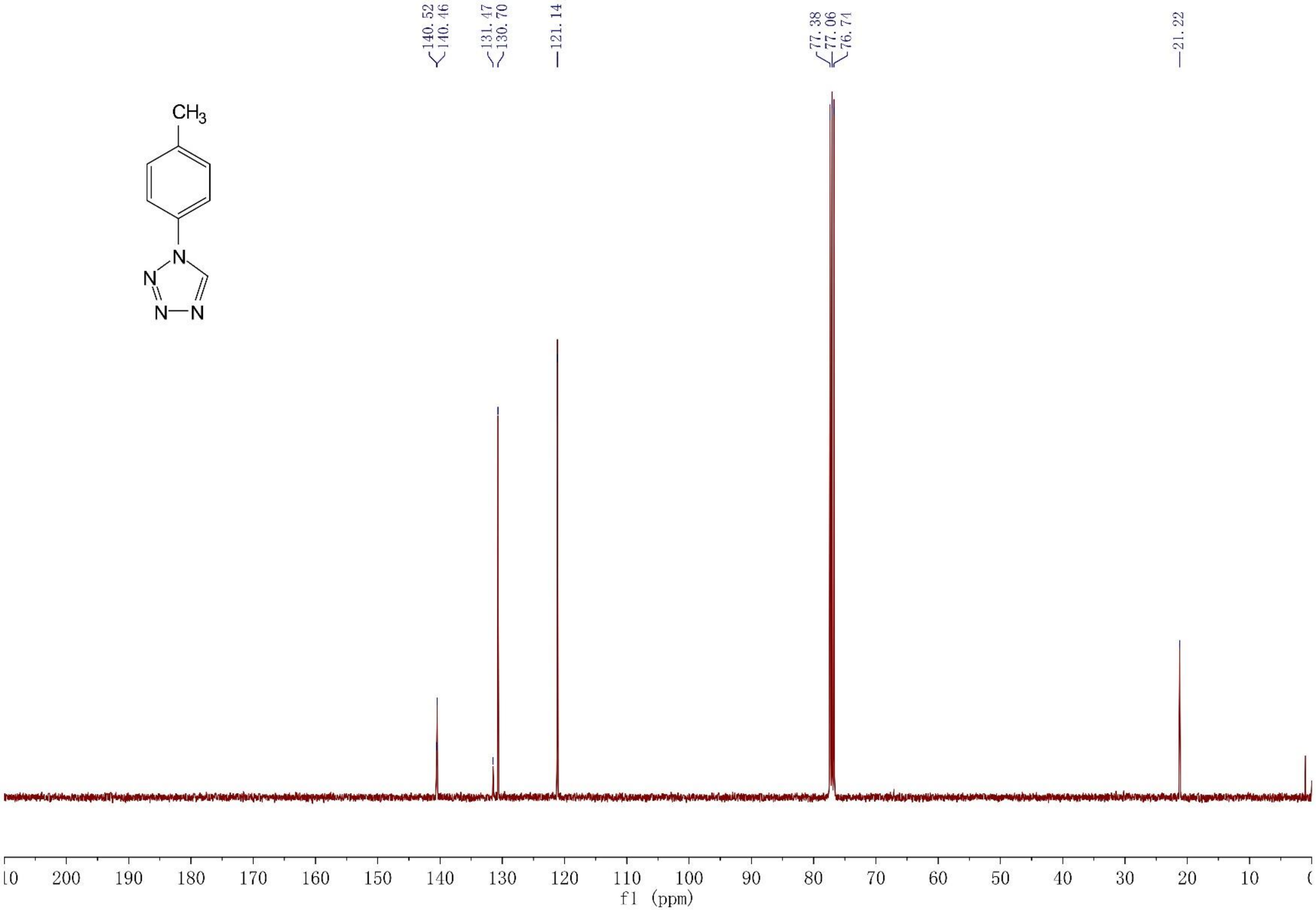

CH3
O
H
H
H
H3C


7.28
7.25
7.23
7.03
7.01
6.98
2.94
2.93
2.92
2.58
2.56
2.54
2.52
2.48
2.45
2.34
2.23
2.21
2.19
2.17
2.14
2.12
2.11
2.09
2.08
2.07
2.06
2.04
2.04
2.02
2.00
1.99
1.70
1.69
1.68
1.67
1.66
1.63
1.59
1.58
1.56
1.55
1.53
1.52
1.48
1.46
0.95
1.00
0.98
0.97
2.05
2.11
2.99
0.99
1.05
3.78
6.91
3.06
f1 (ppm)

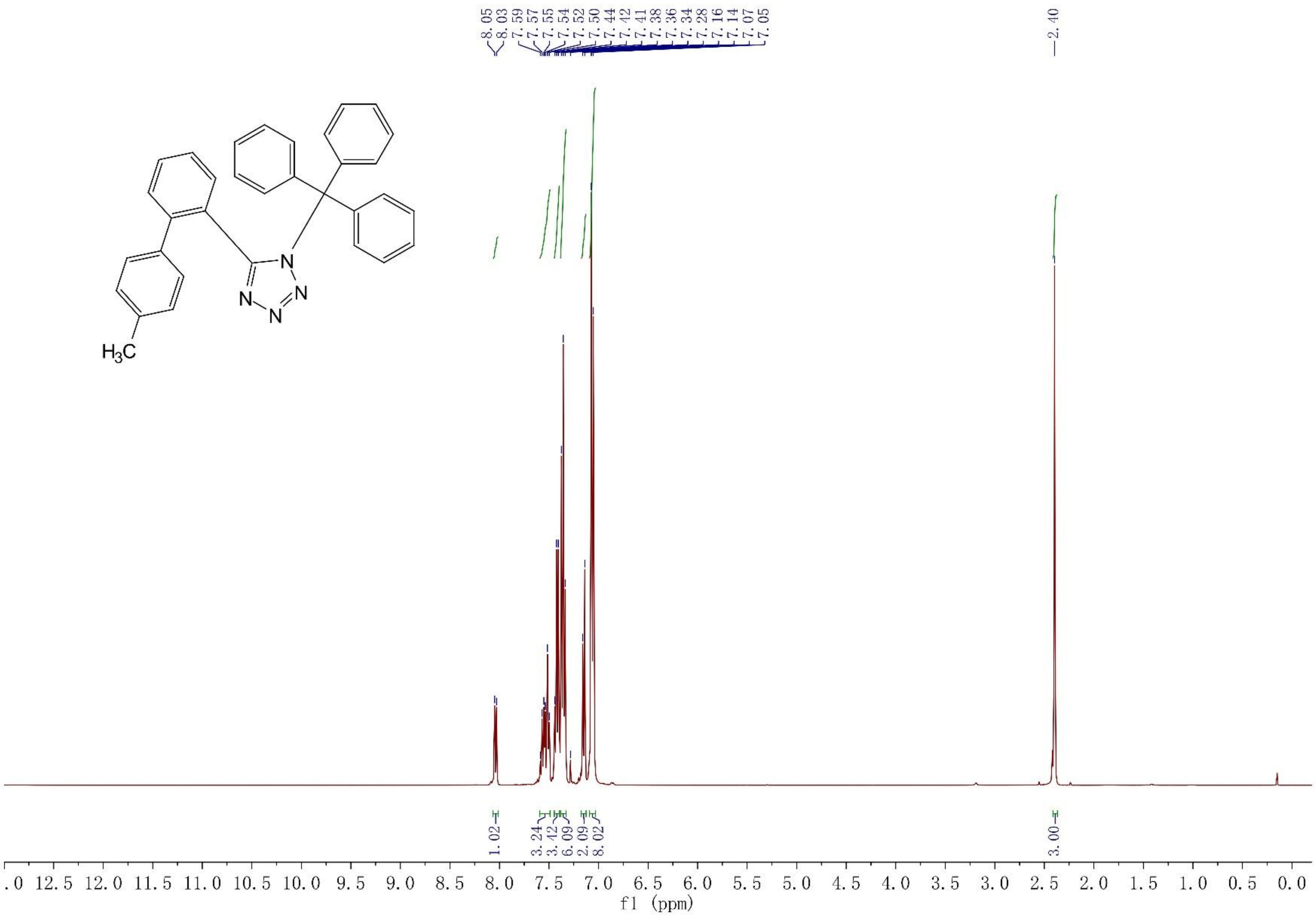

136.75
136.32
135.33
129.76
126.59
125.31
77.44
77.12
76.80
50.51
48.06
44.28
38.32
35.92
31.64
29.38
26.61
25.83
21.63
20.89
13.88
CH3
O
H
H
H
H3C
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



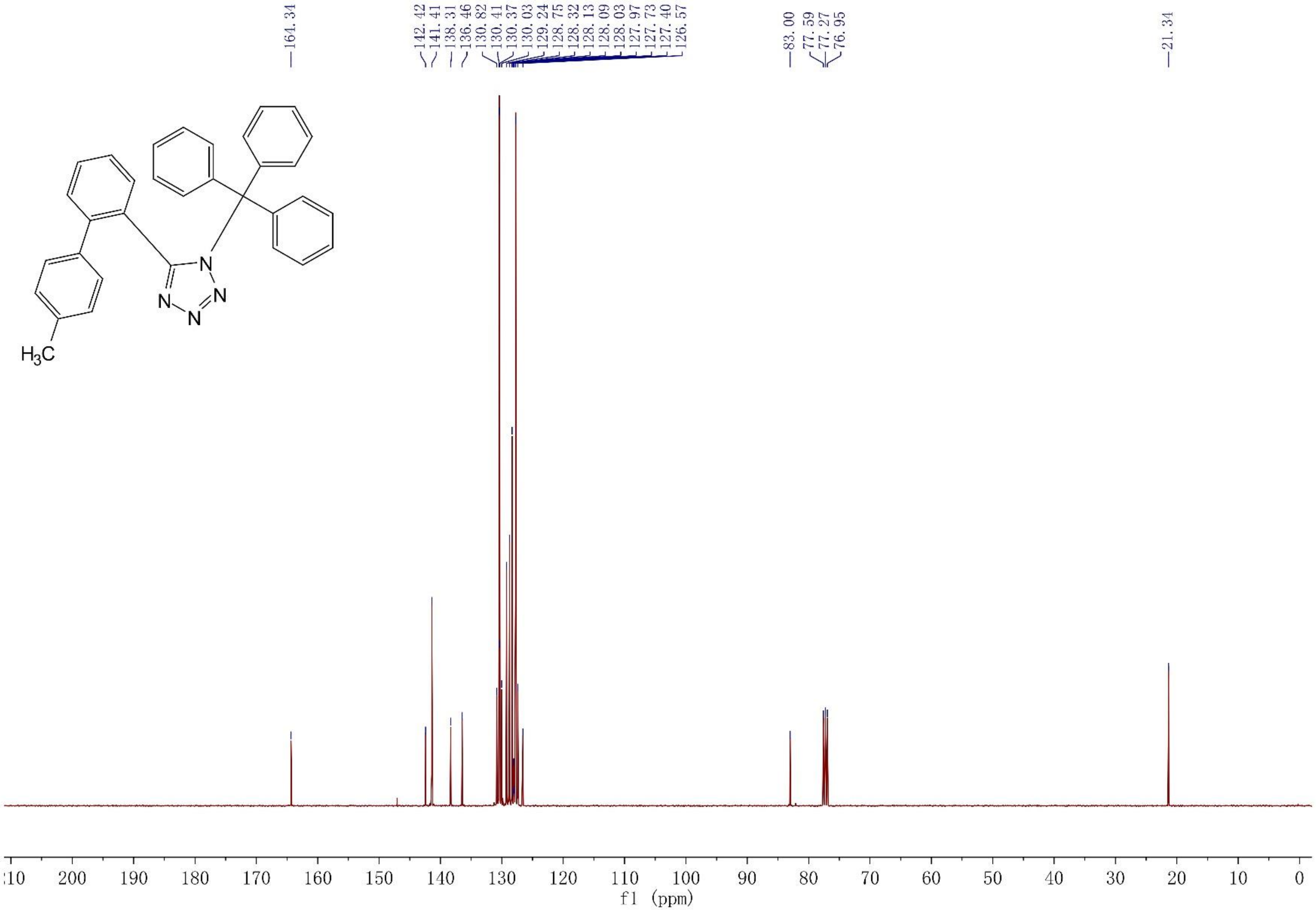
8.99
7.61
7.59
7.41
7.39
7.28
2.47
CH3
N
N
N
N
1.00
2.00
2.01
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



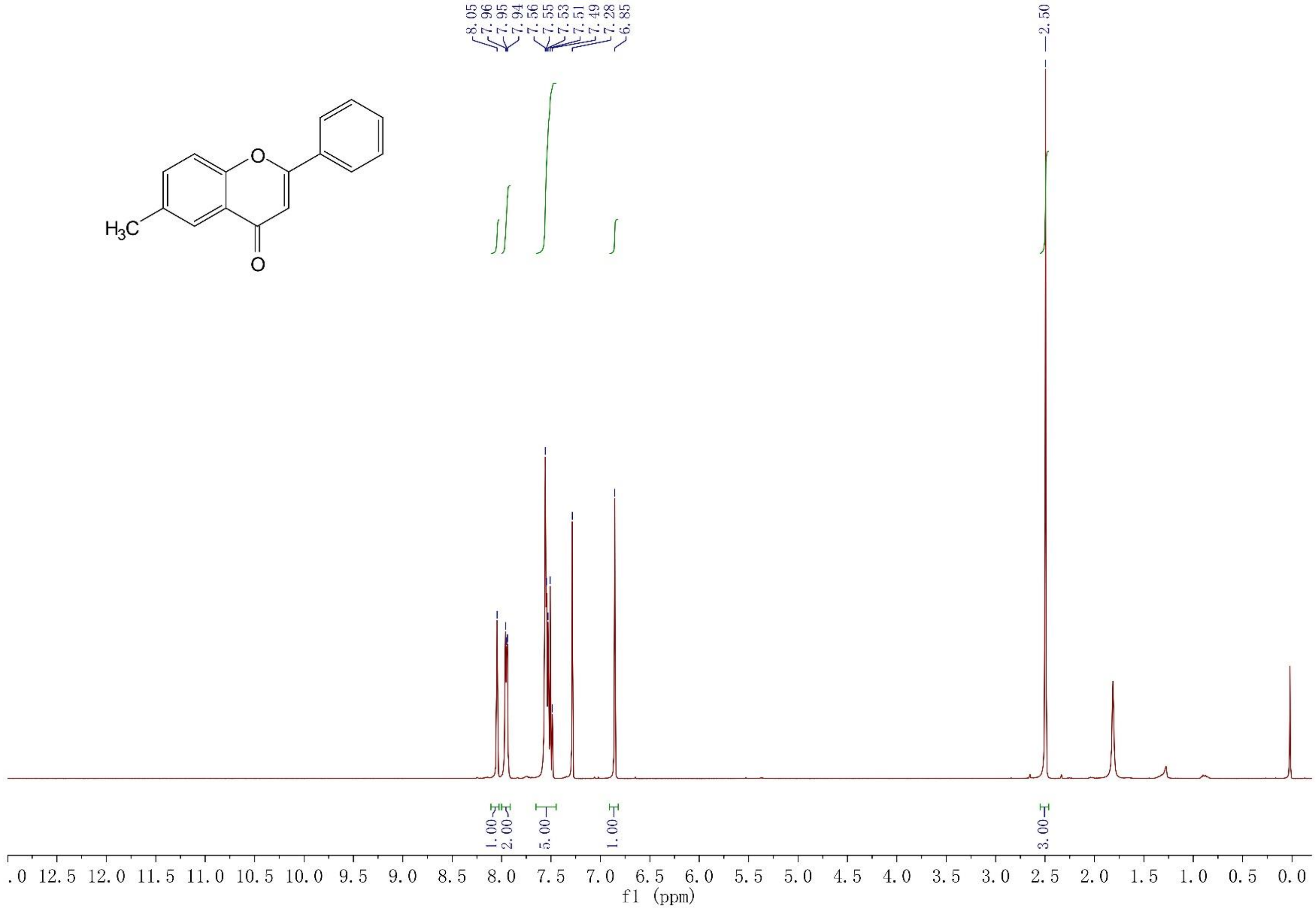
CH3
N
N
N
N
140.52
140.46
131.47
130.70
121.14
77.38
77.06
76.71
21.22
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

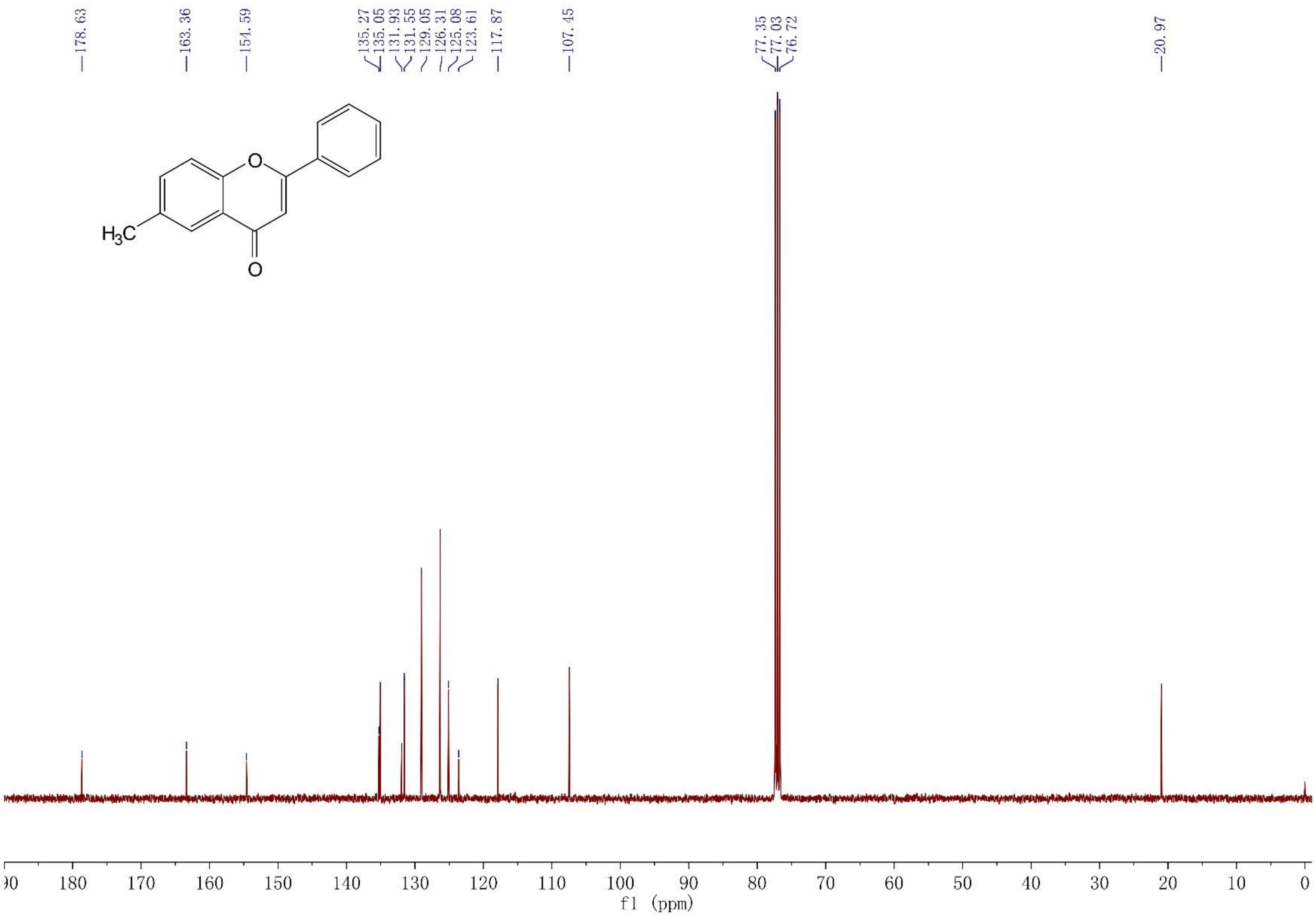


8.05
8.03
7.59
7.57
7.55
7.54
7.52
7.50
7.44
7.42
7.41
7.38
7.36
7.34
7.28
7.16
7.14
7.07
7.05
2.40
N
N
N
N
H3C
1.02
3.24
3.42
6.09
2.09
8.02
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

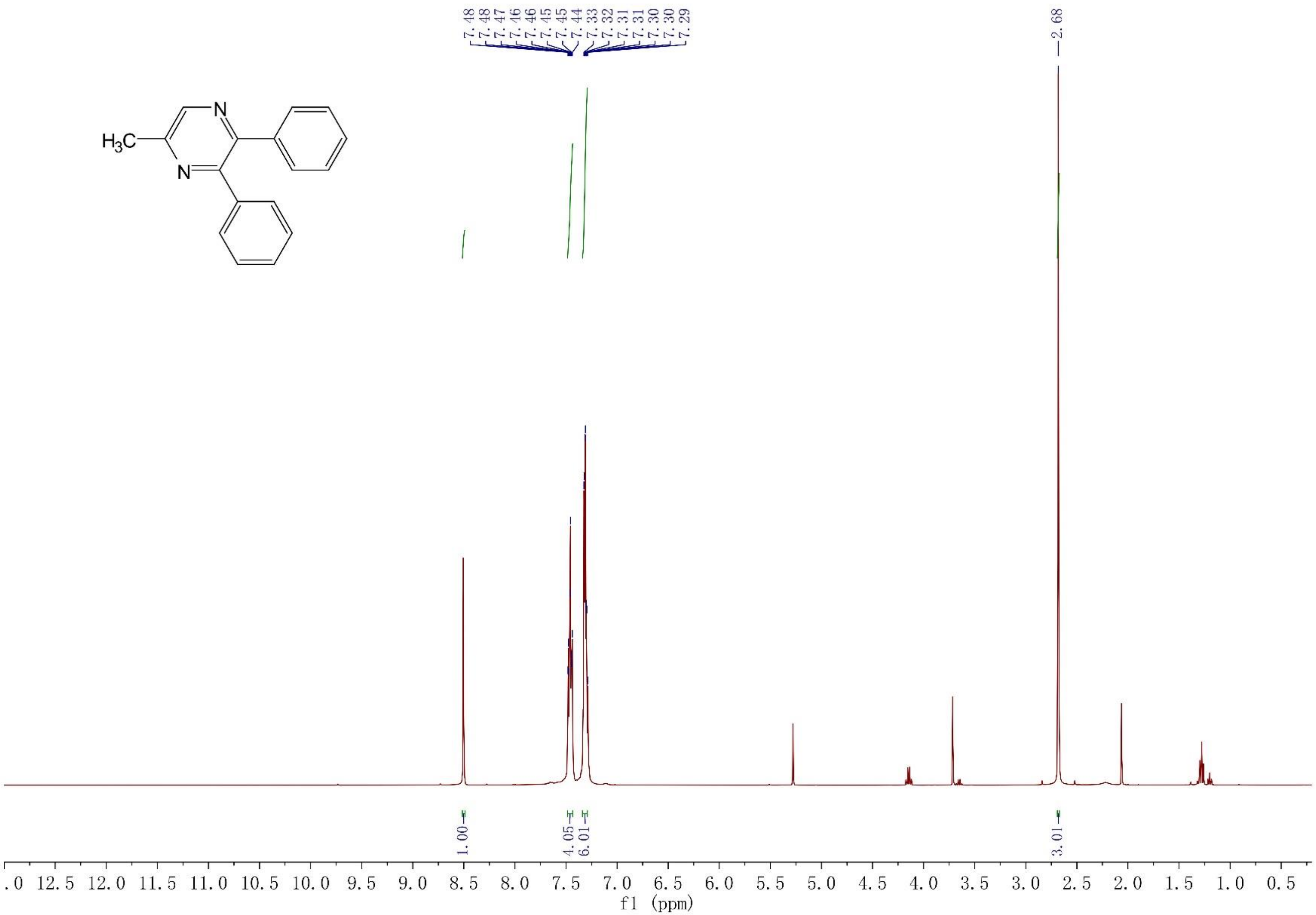


N
N
N
N
H3C
164.34
142.42
141.41
138.31
136.46
130.82
130.41
130.37
130.03
129.24
128.75
128.32
128.13
128.09
128.03
127.97
127.73
127.40
126.57
83.00
77.59
77.27
76.95
21.34
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

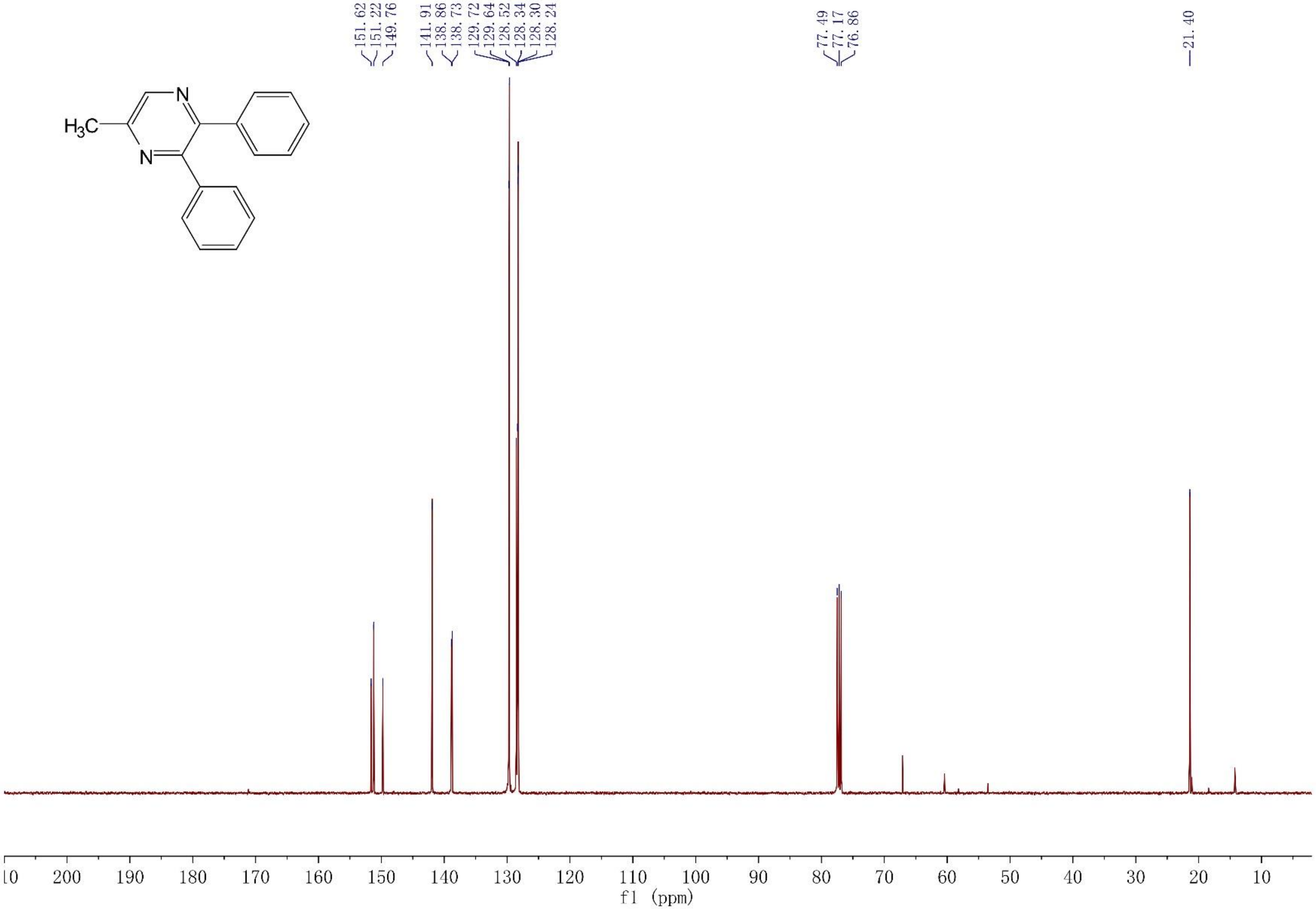

H3C
O
O
8.05
7.96
7.95
7.94
7.56
7.55
7.53
7.51
7.49
7.28
6.85
2.50
1.00
2.00
5.00
1.00
3.00
f1 (ppm)



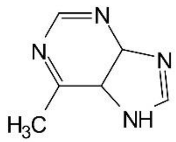
178.63
163.36
154.59
135.27
135.05
131.93
131.55
129.05
126.31
125.08
123.61
117.87
107.45
77.35
77.03
76.72
20.97
H3C
O
O
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

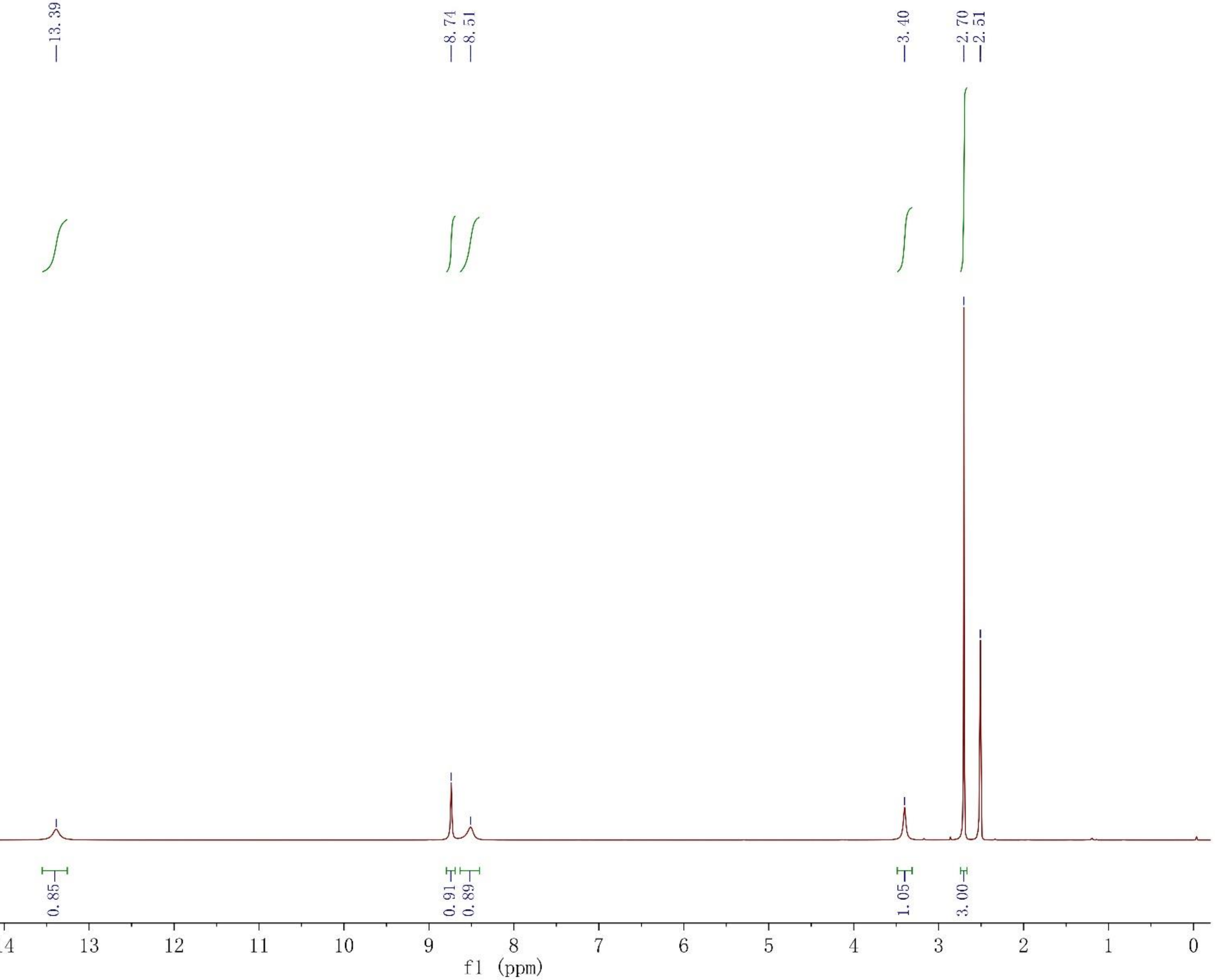


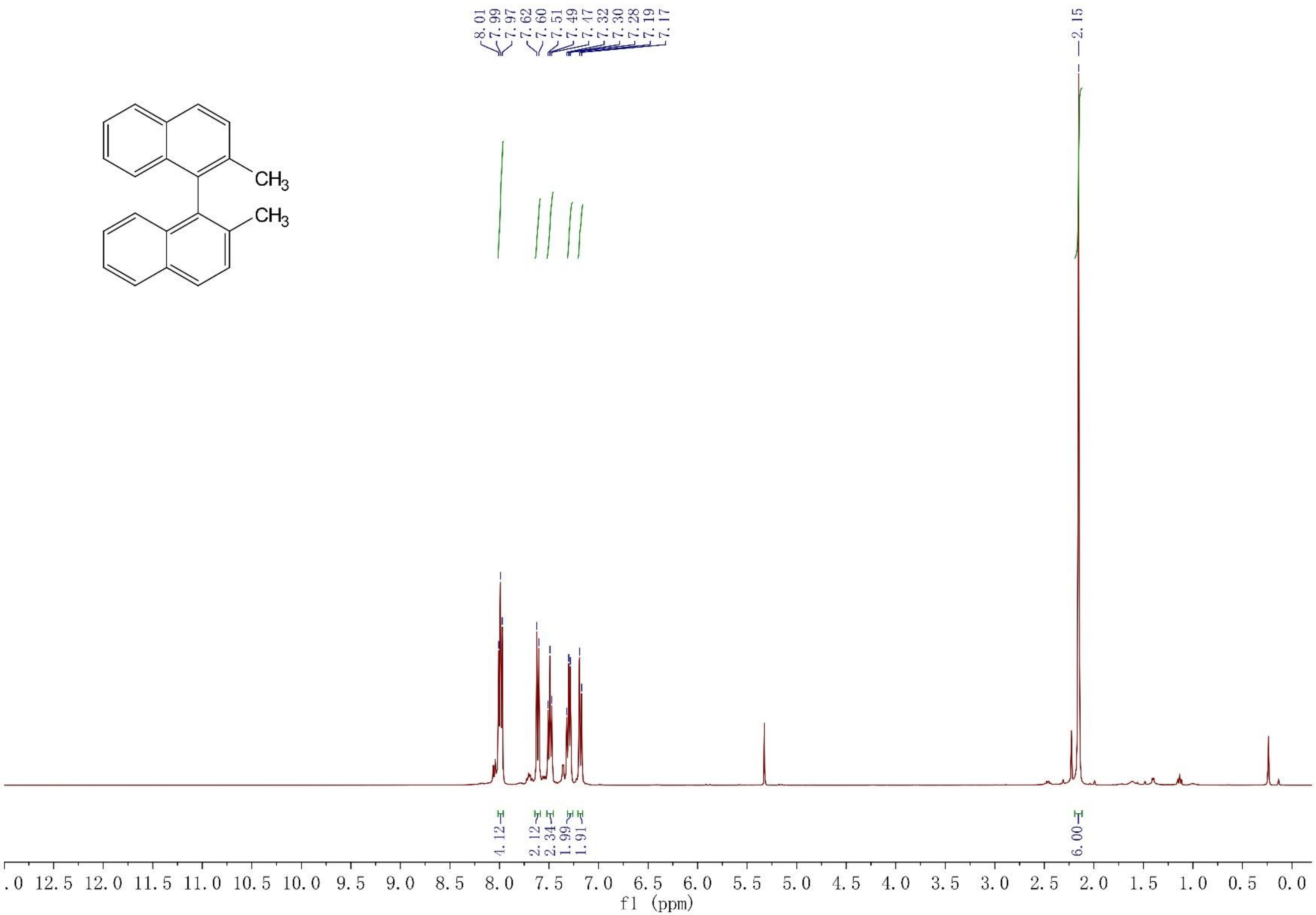
H3C
N
N
7.48
7.48
7.47
7.46
7.46
7.45
7.45
7.44
7.33
7.32
7.31
7.31
7.30
7.30
7.29
2.68
1.00
4.05
6.01
3.01
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)



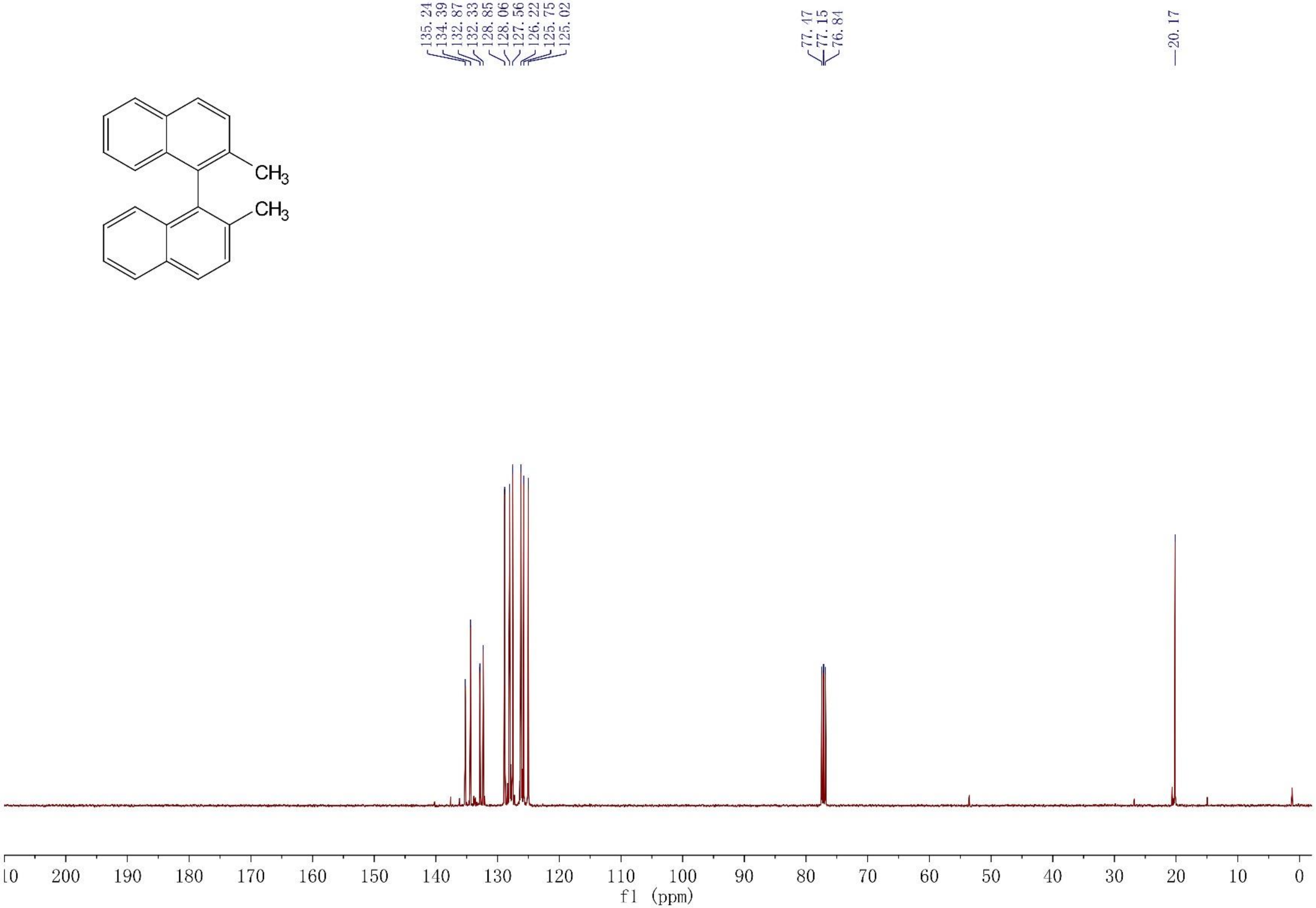
H3C
N
N
151.62
151.22
149.76
141.91
138.86
138.73
129.72
129.64
128.52
128.34
128.30
128.24
77.49
77.17
76.86
21.40
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

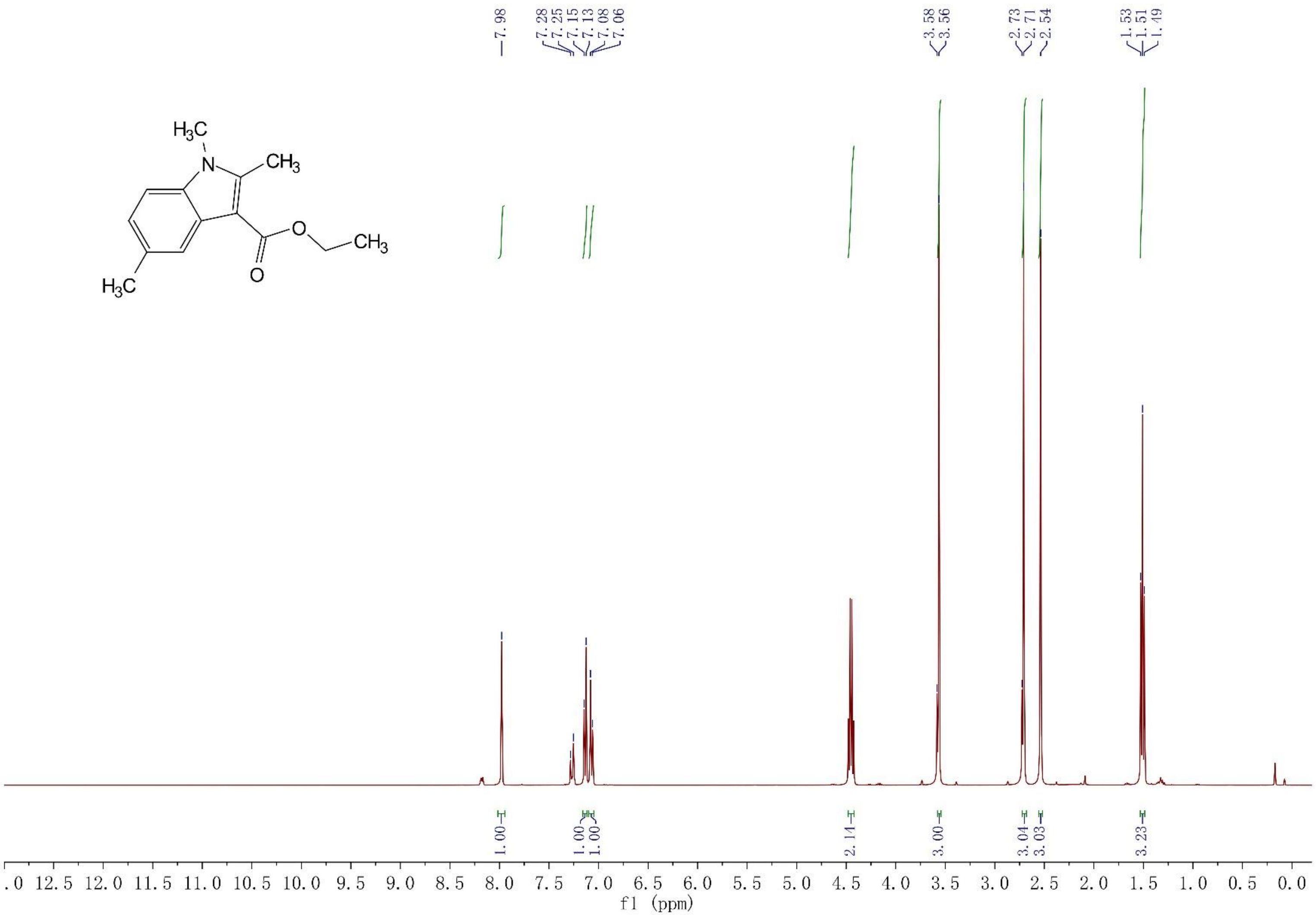

N
N
N
NH
H3C


13.39
8.74
8.51
3.40
2.70
2.51
0.85
0.91
0.89
1.05
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

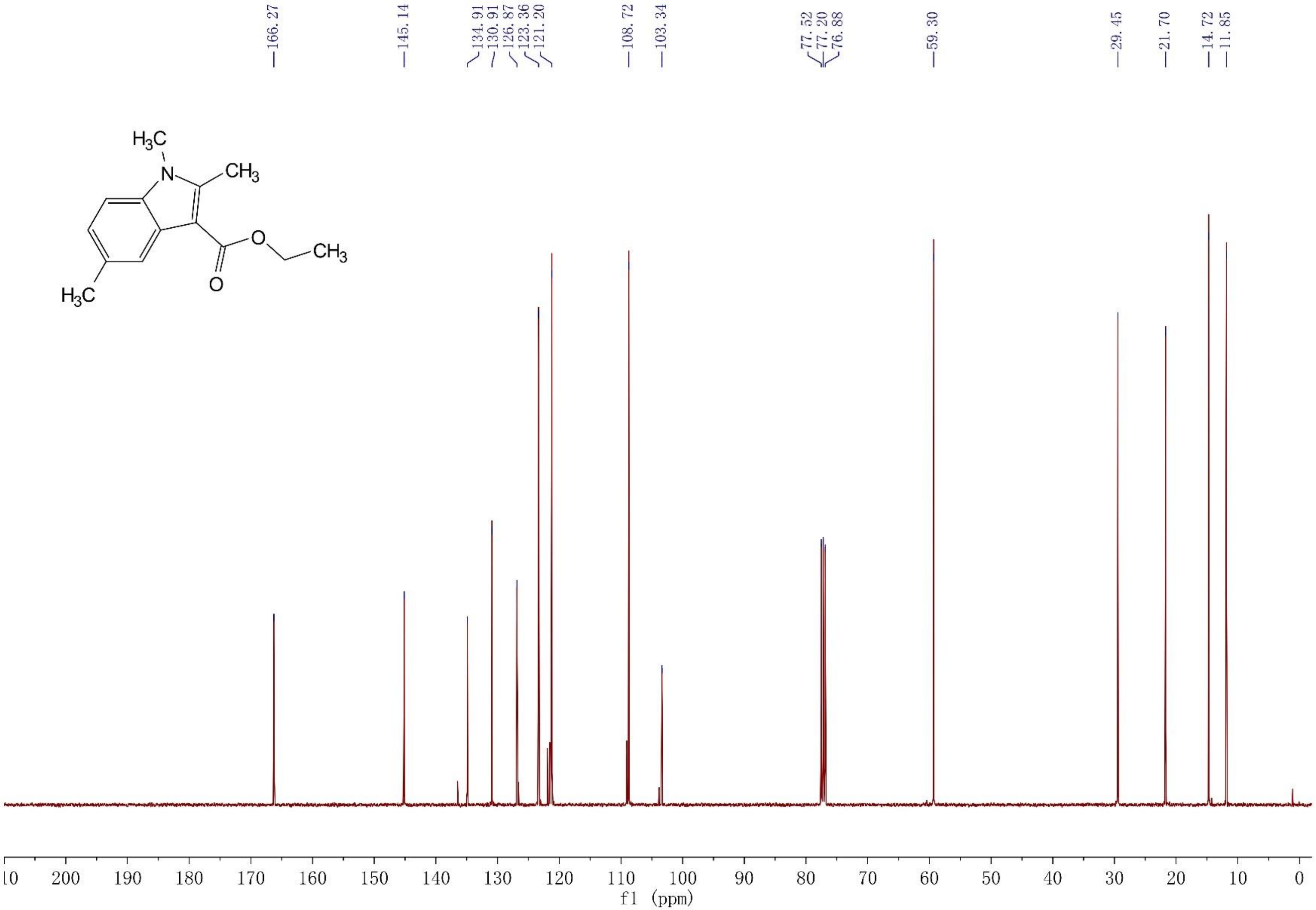


8.01
7.99
7.97
7.62
7.60
7.51
7.49
7.47
7.32
7.30
7.28
7.19
7.17
2.15
CH3
CH3
4.12
2.12
2.34
1.99
1.91
6.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

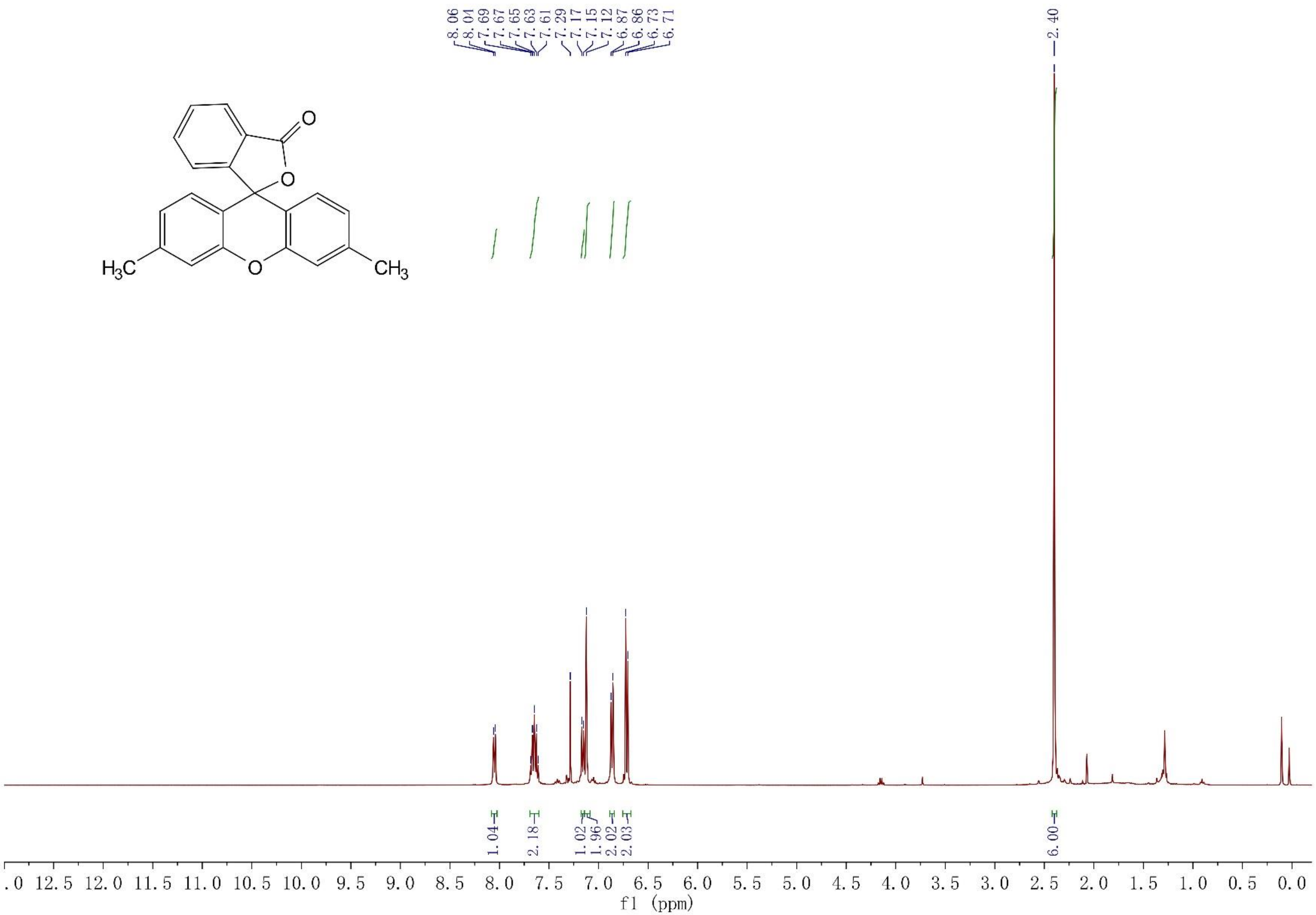

135.24
134.39
132.87
132.33
128.85
128.06
127.56
126.22
125.75
125.02
77.47
77.15
76.84
20.17
CH3
CH3
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



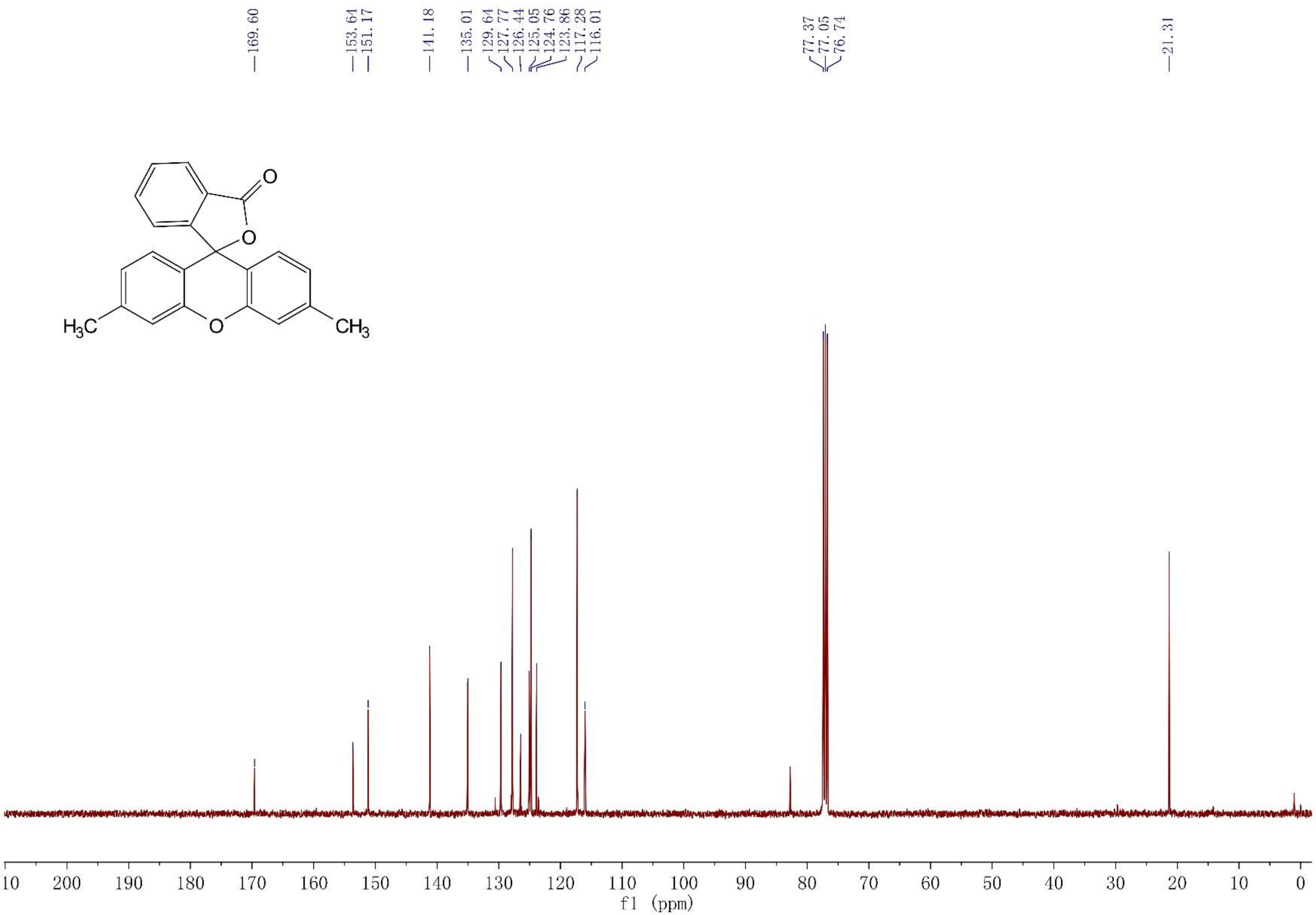
7.98
7.28
7.25
7.15
7.13
7.08
7.06
3.58
3.56
2.73
2.71
2.54
1.53
1.51
1.49
1.00
1.00
1.00
2.14
3.00
3.04
3.03
3.23
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

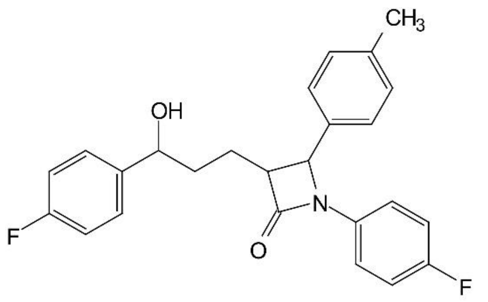


166.27
145.14
134.91
130.91
126.87
123.36
121.20
108.72
103.34
77.52
77.20
76.88
59.30
29.45
21.70
14.72
11.85
f1 (ppm)

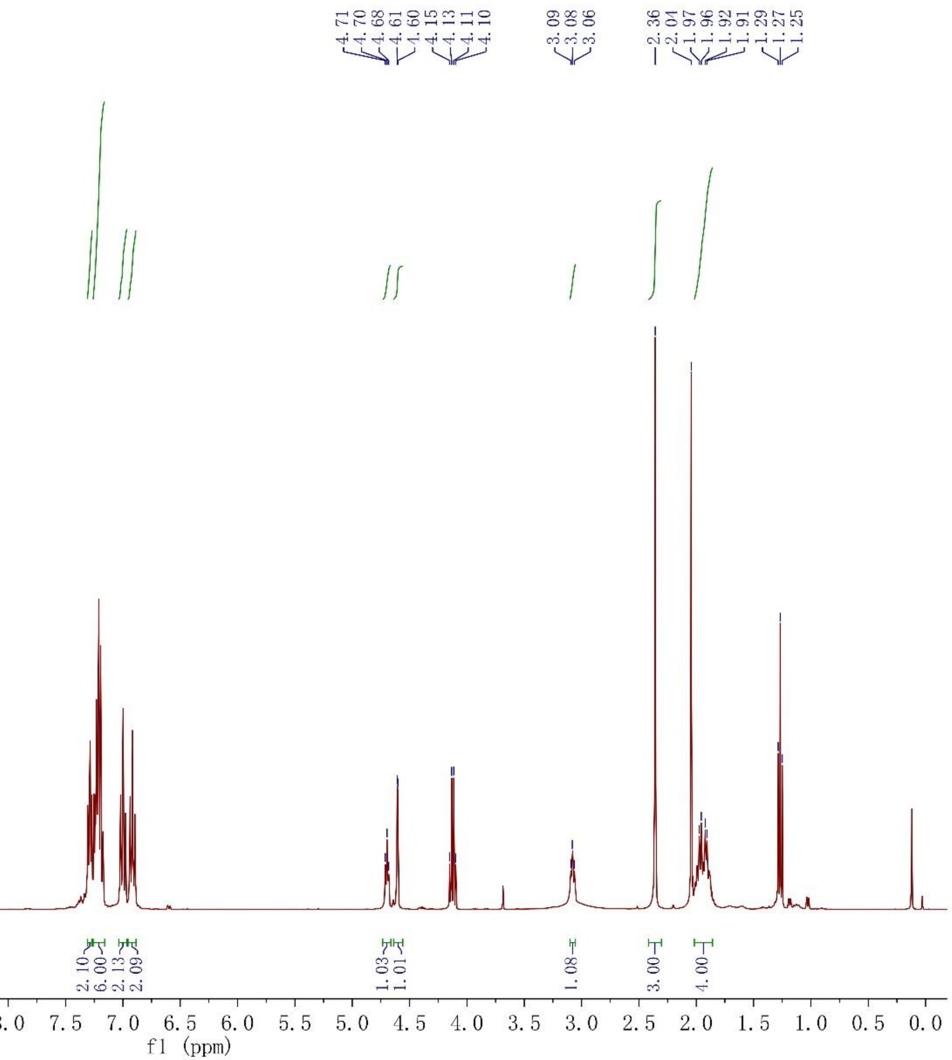


8.06
8.04
7.69
7.67
7.65
7.63
7.61
7.29
7.17
7.15
7.12
6.87
6.86
6.73
6.71
2.40
O
O
O
H3C
CH3
1.04
2.18
1.02
1.96
2.02
2.03
6.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

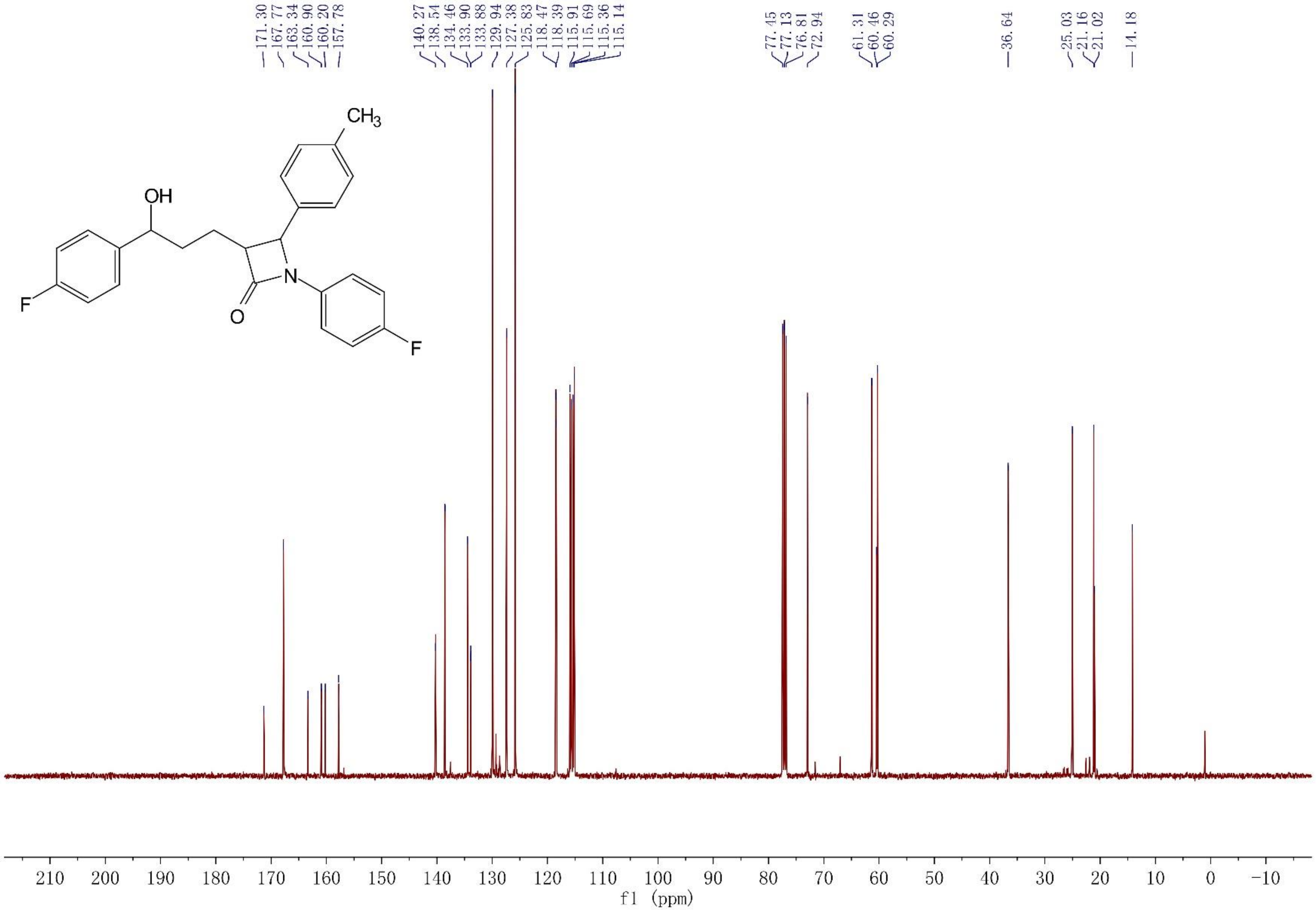

169.60
153.64
151.17
141.18
135.01
129.64
127.77
126.44
125.05
124.76
123.86
117.28
116.01
77.37
77.05
76.74
21.31
O
O
O
H_3C
CH_3
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

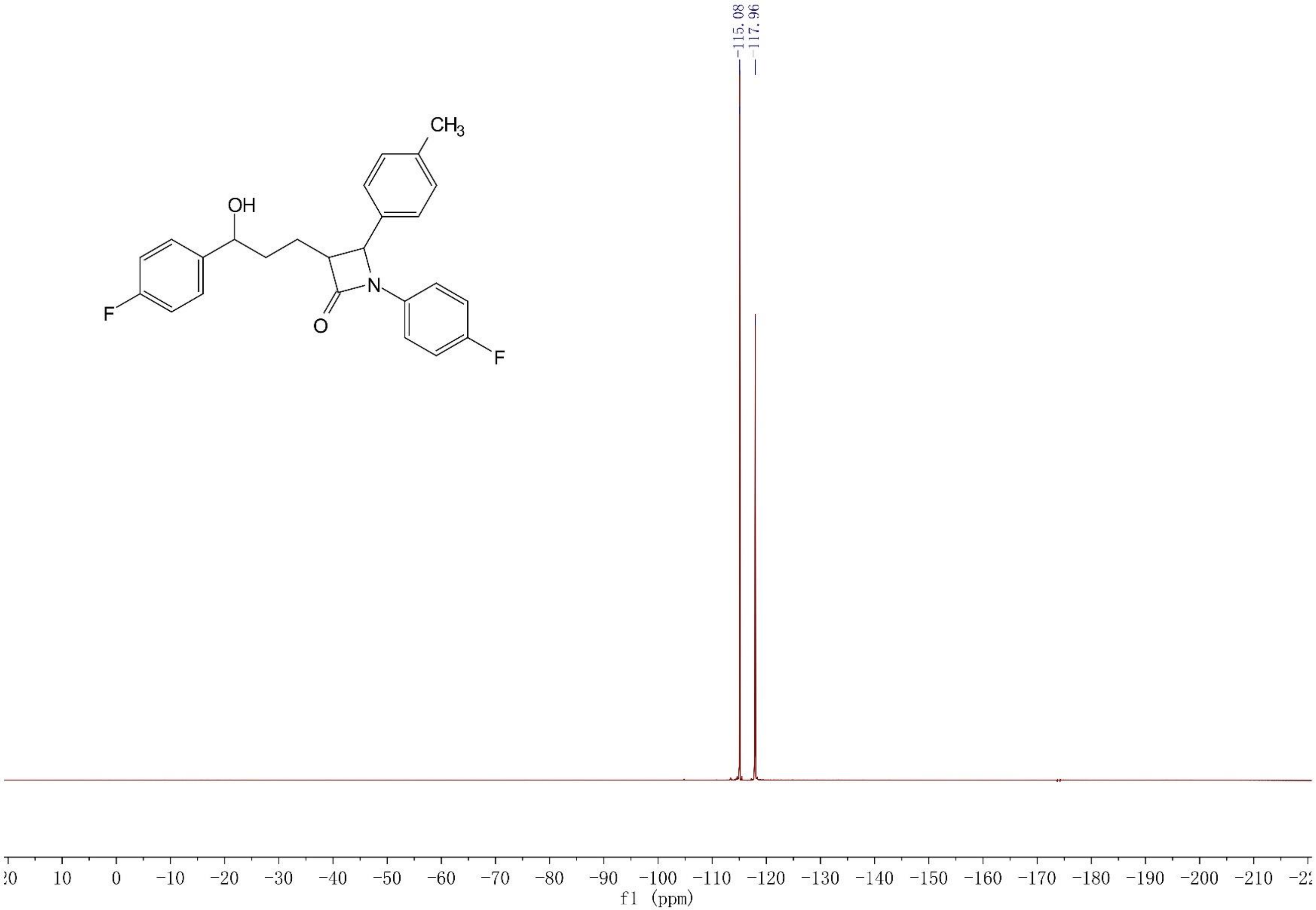


OH
CH3
N
O
F
F

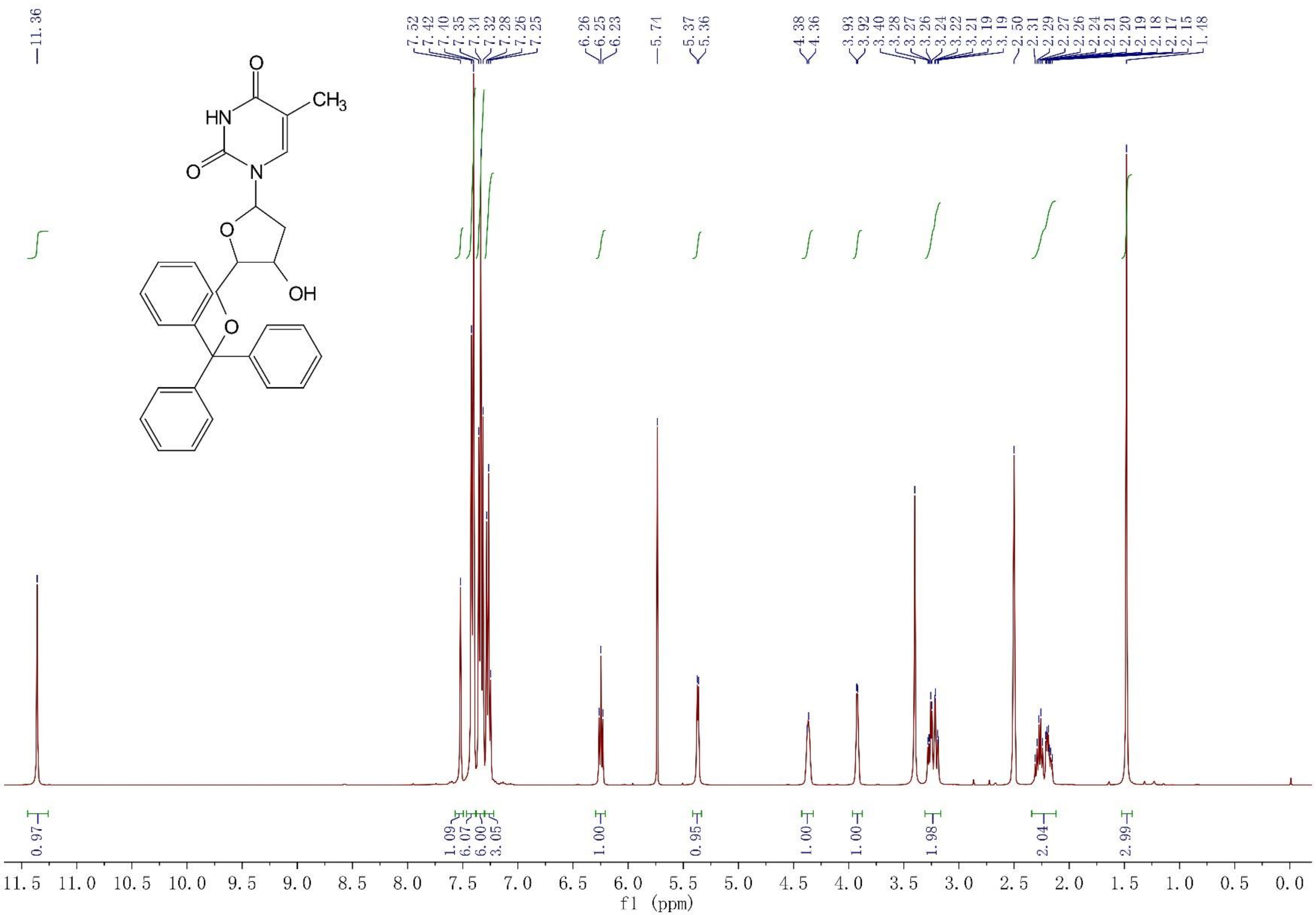


4.71
4.70
4.68
4.61
4.60
4.15
4.13
4.11
4.10
3.09
3.08
3.06
2.36
2.04
1.97
1.96
1.92
1.91
1.29
1.27
1.25
2.10
6.00
2.13
2.09
1.03
1.01
1.08
3.00
4.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

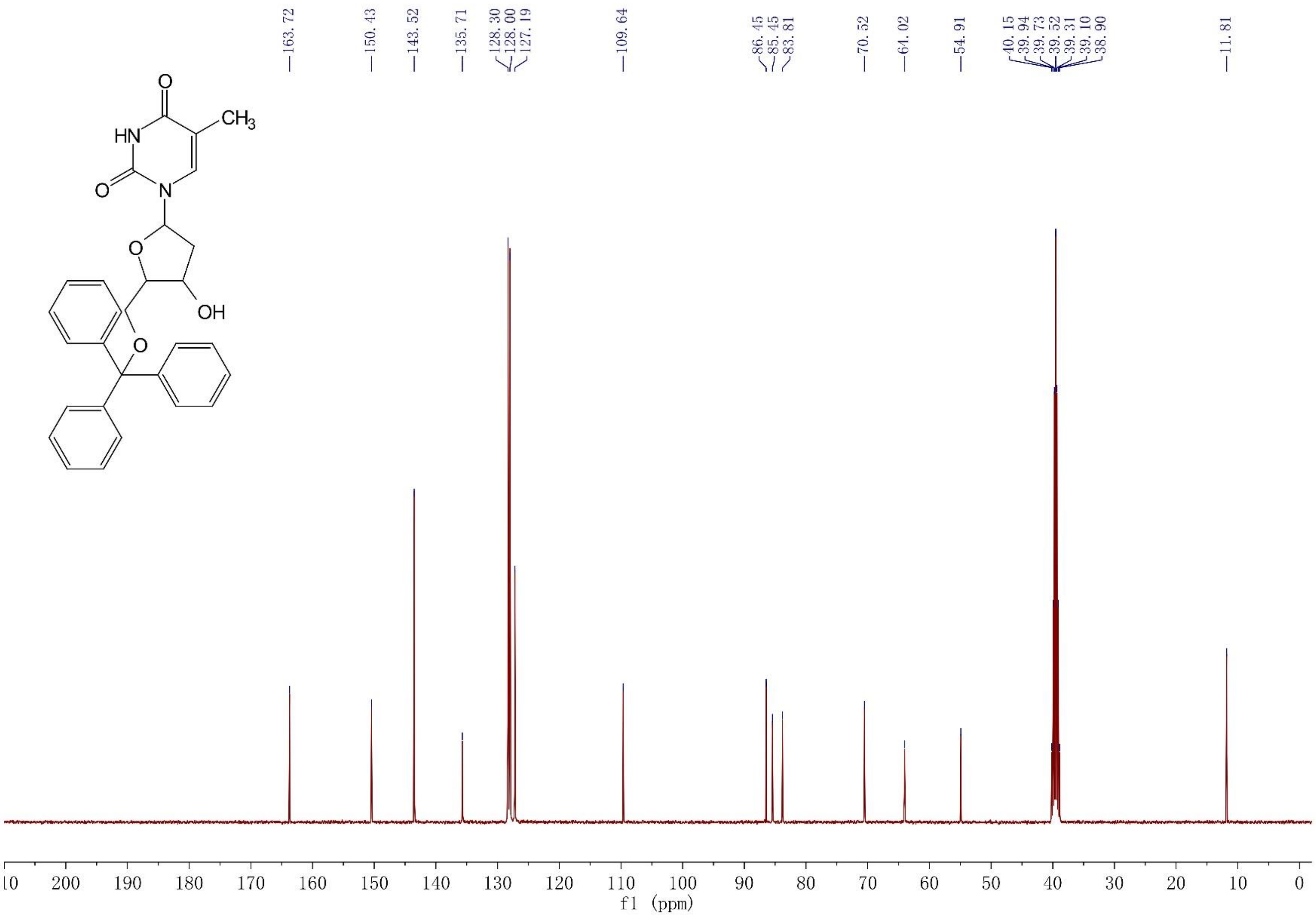

171.30
167.77
163.34
160.90
160.20
157.78
140.27
138.54
134.46
133.90
133.88
129.94
127.38
125.83
118.47
118.39
115.91
115.69
115.36
115.14
77.45
77.13
76.81
72.94
61.31
60.46
60.29
36.64
25.03
21.16
21.02
14.18
CH3
OH
N
O
F
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



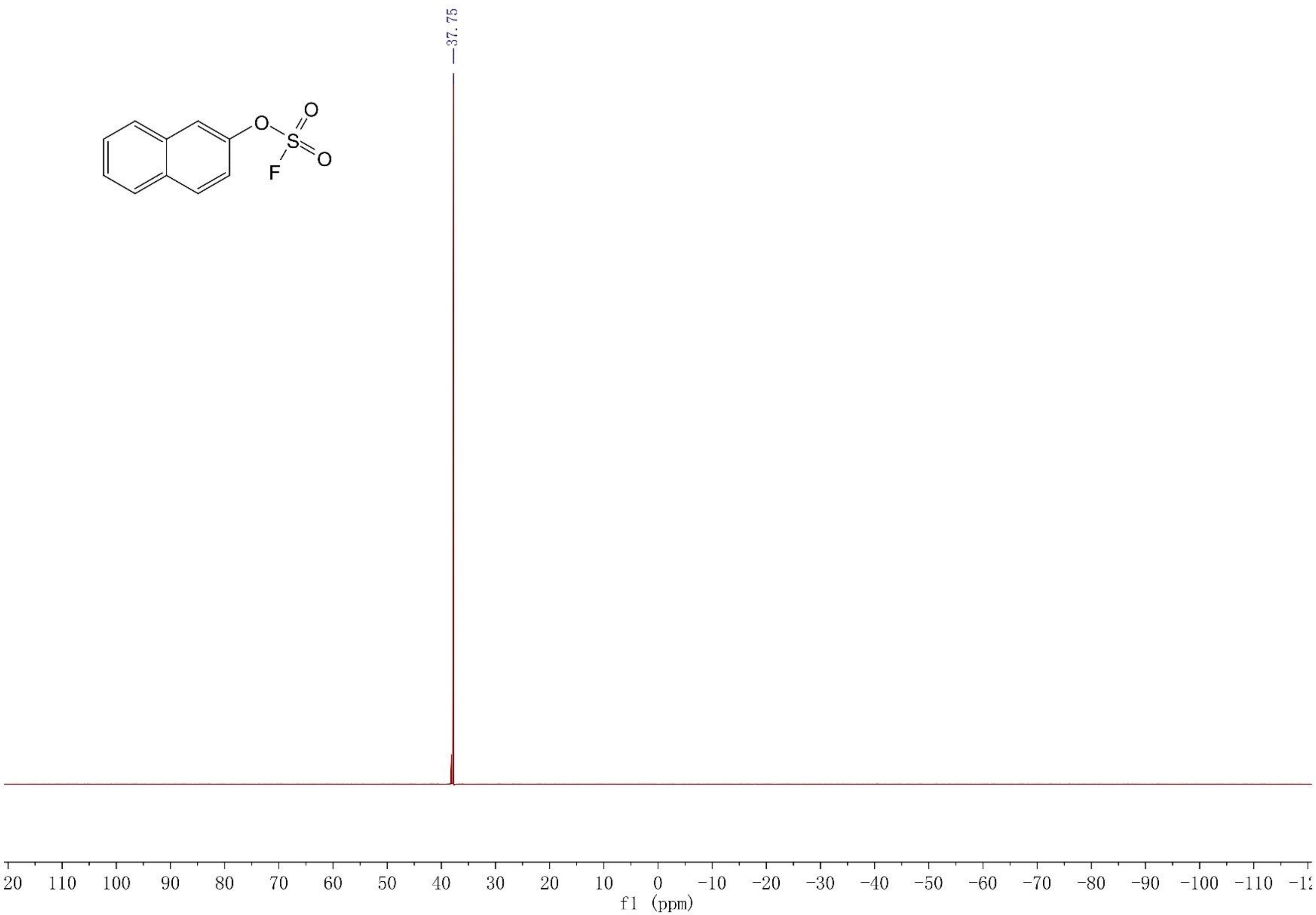
-115.08
-117.96
OH
CH3
N
O
F
F
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)



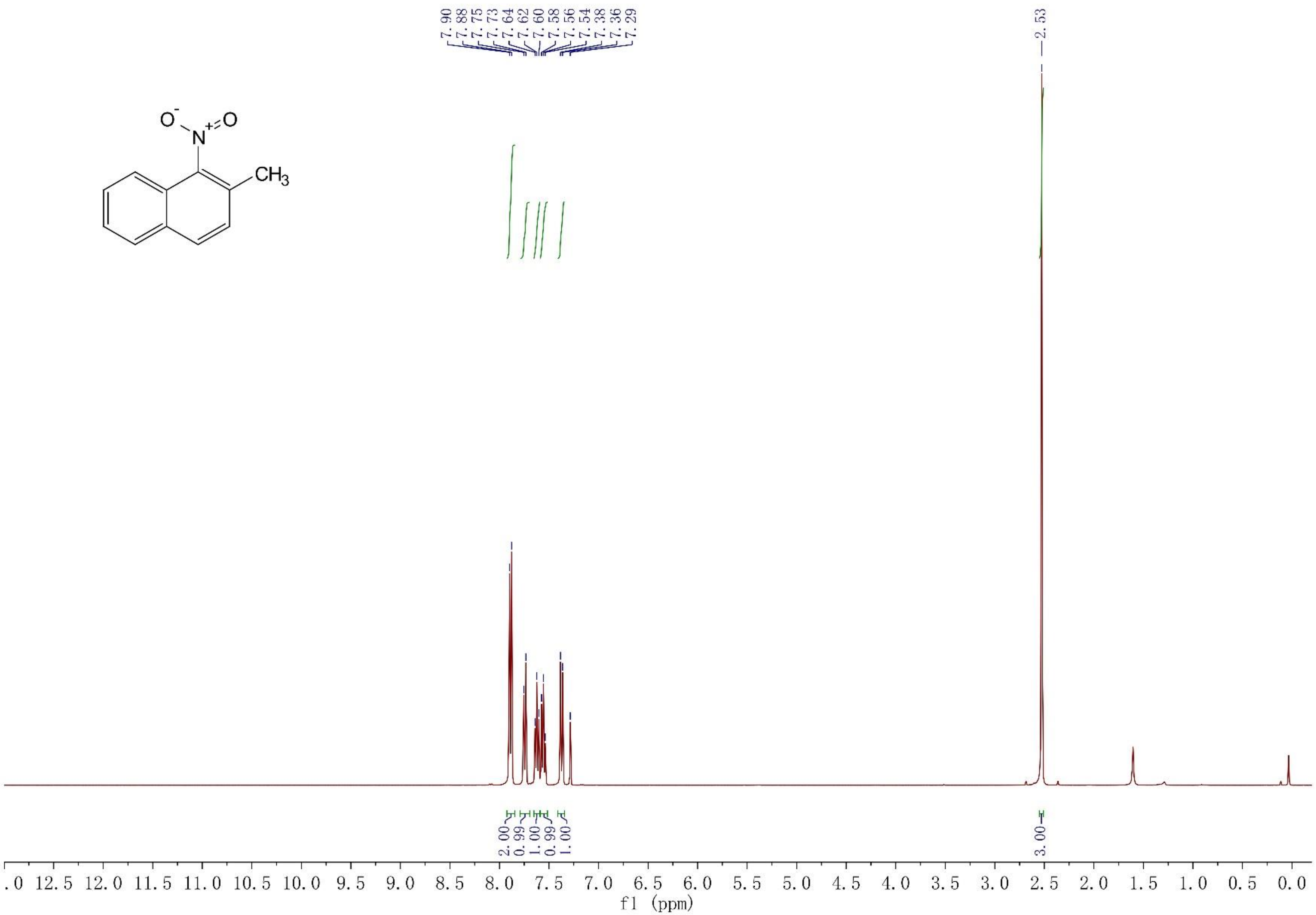
11.36
7.52 7.42 7.40 7.35 7.31 7.32 7.28 7.26 7.25
6.26 6.25 6.23
5.74
5.37 5.36
4.38 4.36
3.93 3.92 3.40 3.28 3.27 3.26 3.24 3.22 3.21 3.19 3.19
2.50 2.31 2.29 2.27 2.26 2.24 2.21 2.20 2.19 2.18 2.17 2.15 1.48
0.97
1.09 6.07 6.00 3.05
1.00
0.95
1.00
1.00
1.98
2.04
2.99
11.5 11.0 10.5 10.0 9.5 9.0 8.5 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)



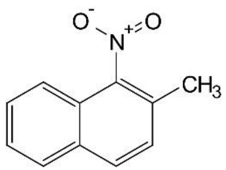
163.72
150.43
143.52
135.71
128.30
128.00
127.19
109.64
86.45
85.45
83.81
70.52
64.02
54.91
40.15
39.94
39.73
39.52
39.31
39.10
38.90
11.81
O
HN
CH3
O
N
O
OH
O
f1 (ppm)

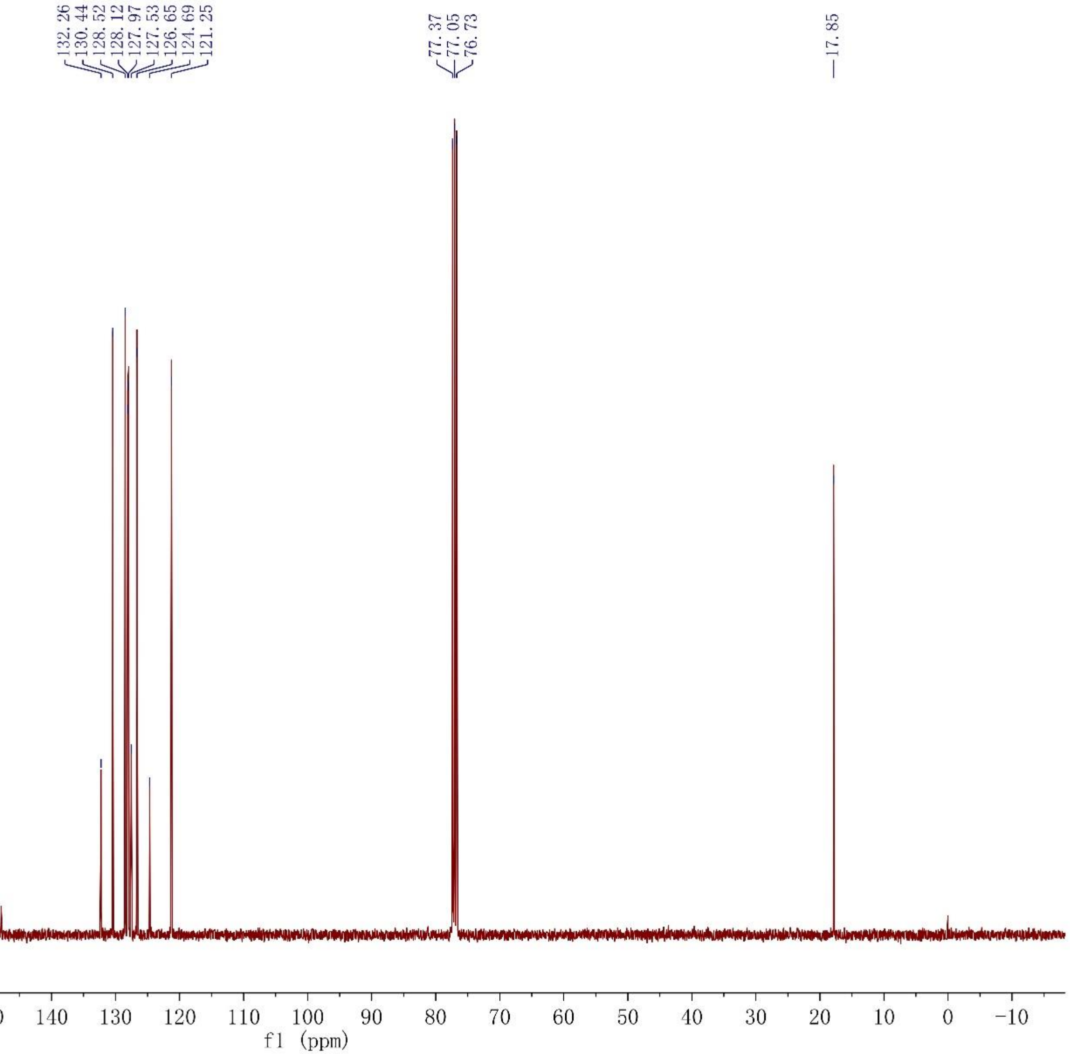


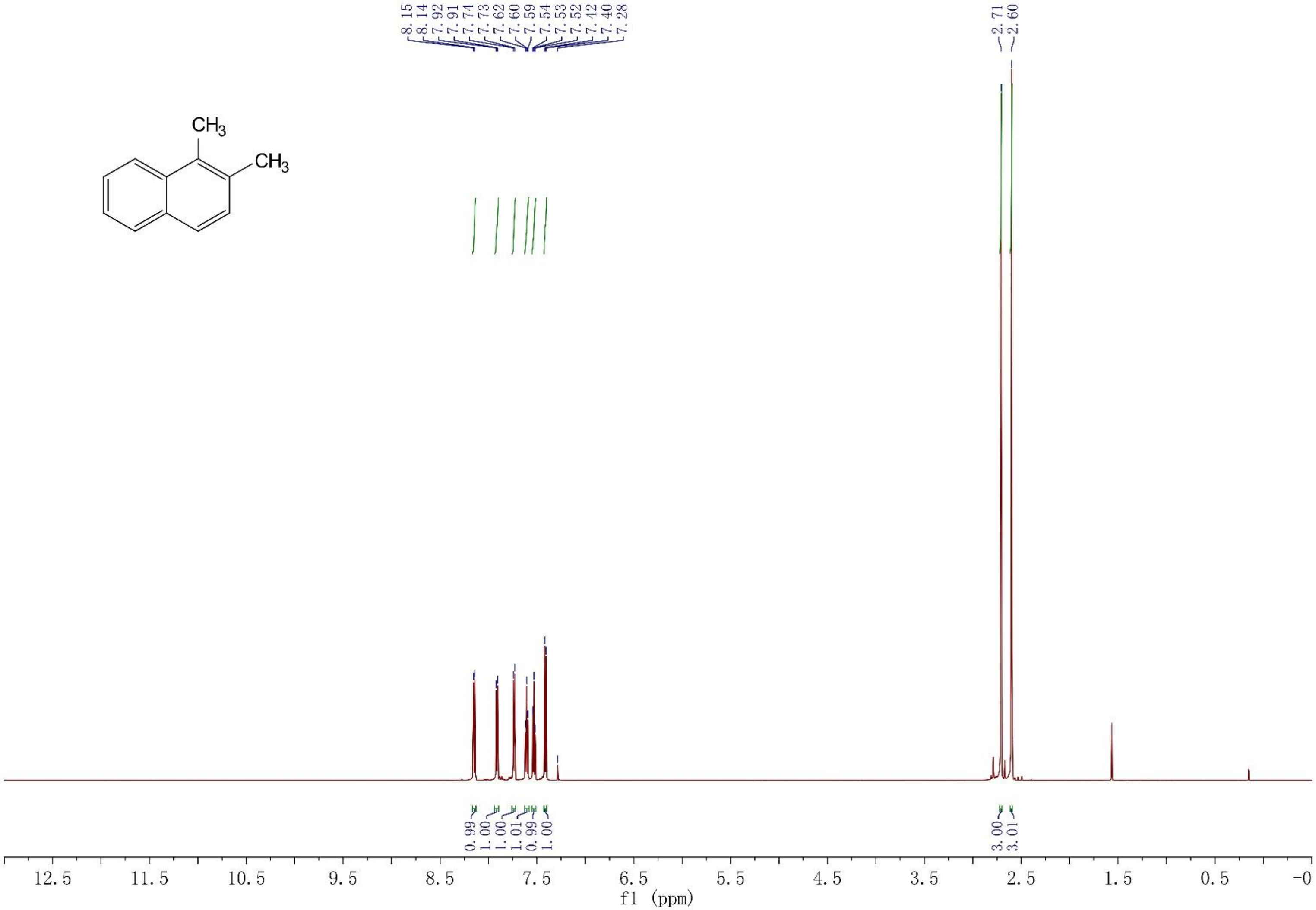
O
S
O
O
F
37.75
20 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110
f1 (ppm)



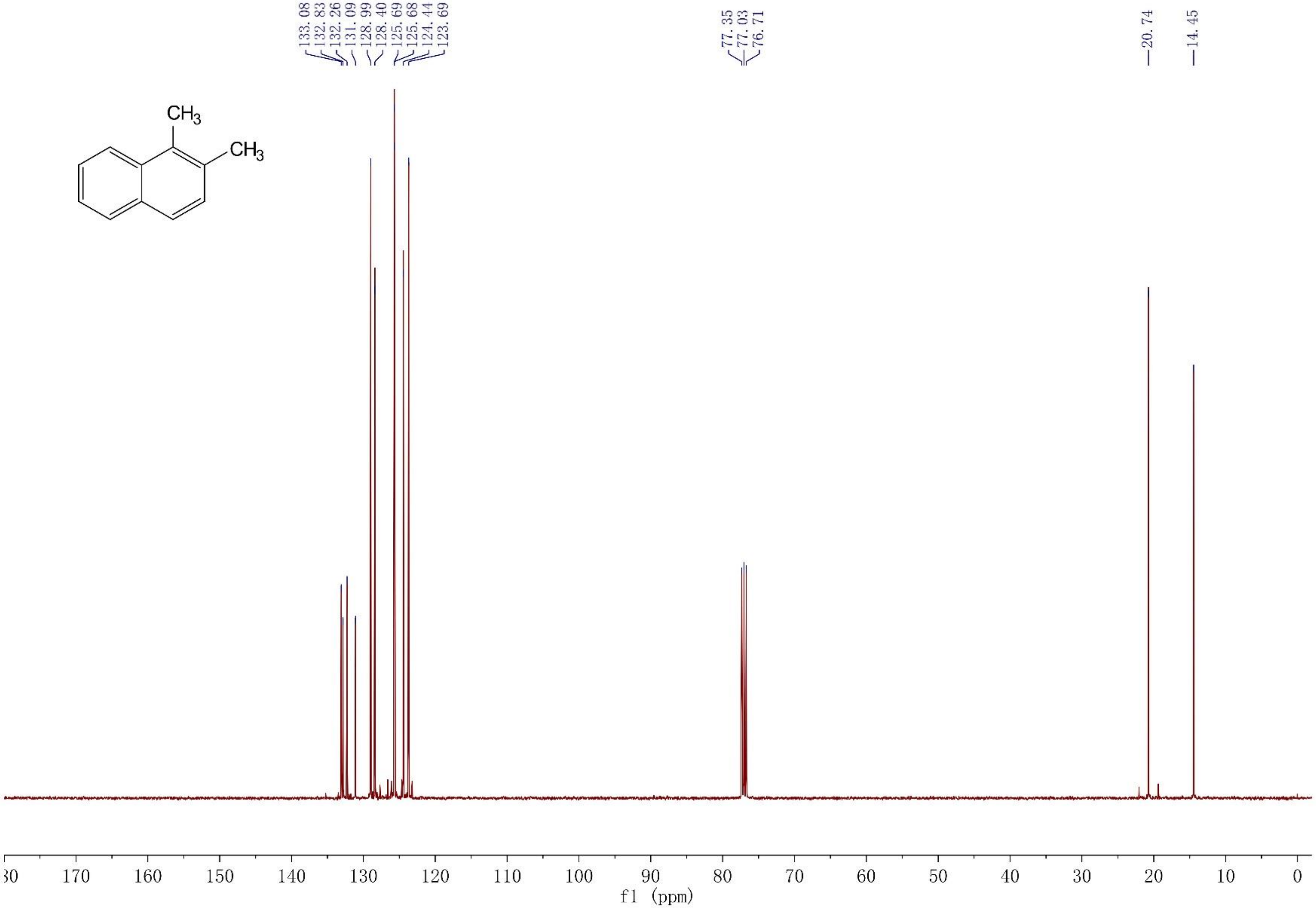
O
N
O
CH3
7.90
7.88
7.75
7.73
7.64
7.62
7.60
7.58
7.56
7.54
7.38
7.36
7.29
2.53
2.00
0.99
1.00
0.99
1.00
3.00
.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


O
N
O
CH3


132.26
130.44
128.52
128.12
127.97
127.53
126.65
124.69
121.25
77.37
77.05
76.73
17.85
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)



8.15
8.14
7.92
7.91
7.74
7.73
7.62
7.60
7.59
7.54
7.53
7.52
7.42
7.40
7.28
2.71
2.60
CH3
CH3
0.99
1.00
1.01
0.99
1.00
3.00
3.01
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0
f1 (ppm)



CH3
CH3
133.08
132.83
132.26
131.09
128.99
128.40
125.69
125.68
124.44
123.69
77.35
77.03
76.71
20.74
14.45
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)