Electronic Supplementary Material (ESI) for ChemComm.

Supplementary information

Iron catalyzed deconstructive alkylation through chlorine radical induced C-C single bond cleavage under visible light

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1. General Information

Reagents were purchased at Energy Chemical, Bidepharm, Aladdin, TCI, and Adamas-Beta and used without further purification unless otherwise stated. Anhydrous $FeCl_3$ and TBACl (Tetrabutyl ammonium chloride) were stored and weighed in the glovebox. NMR spectra were recorded on Bruker AVANCE III 400 and AVANCE III 600 instruments. ^1H NMR chemical shifts are referenced to the residual hydrogen signals of the deuterated solvent $CDCl_3$ (7.26 ppm) or DMSO-d6 (2.50 ppm and for ^{13}C). The ^{13}C NMR chemical shifts are referenced to the ^{13}C signals of the deuterated solvent $CDCl_3$ (77.16 ppm) or DMSO-d6 (39.52 ppm). Abbreviations used in the description of NMR data are listed as follows: s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, p = pentet, m = multiplet, br = broad. HRMS were recorded on Orbitrap LC/MS (Q Exactive). GC-MS analyses were carried out on Agilent 5977B and Agilent GC/MSD 8890 GC system. TLC was performed using commercially prepared 100-400 mesh silica gel plates (GF254), and visualization was effected at shortwave UV light (254 nm). Product purification was accomplished by flash chromatography using 300-400 mesh silica gel.

The LED light was assembled using the 450 nm chips purchased from GuangHong Chips. The emission spectrum of the LED light is shown below (**Figure S1**).

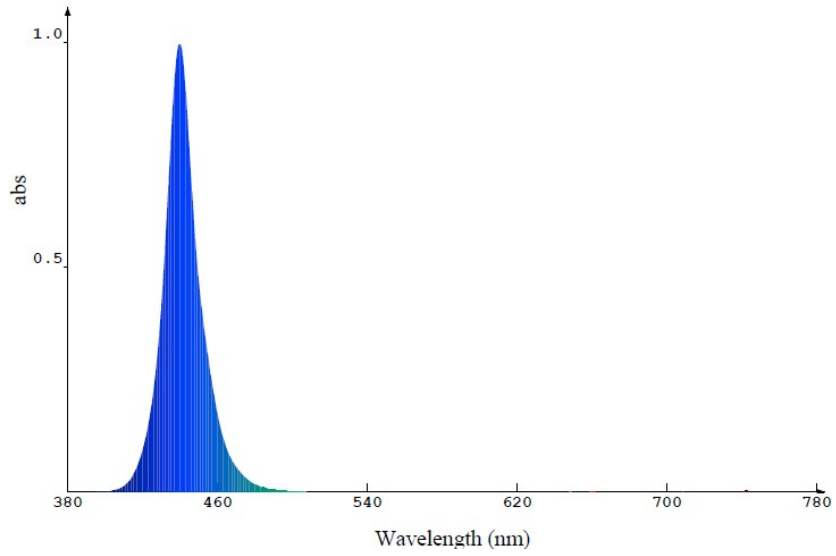


Figure S1. The spectrum of the LED light

2. Application experiments

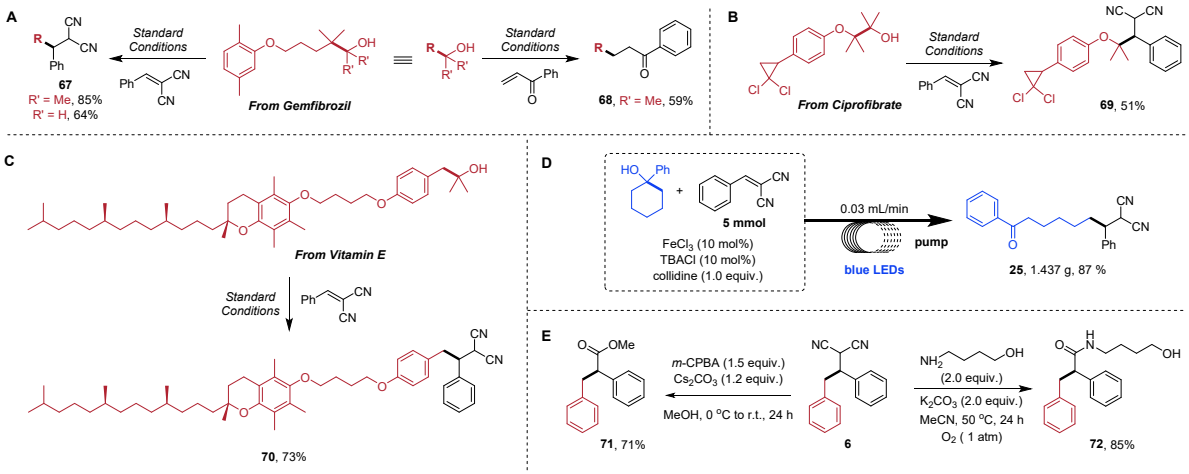


Figure S2. Application experiments. **A**, **B**, **C**, Late-stage functionalization. **D**, Gram scale reaction. **E**, Transformation of malononitrile group.

3. Experimental Section

3.1 Optimization of Reaction Conditions[a]

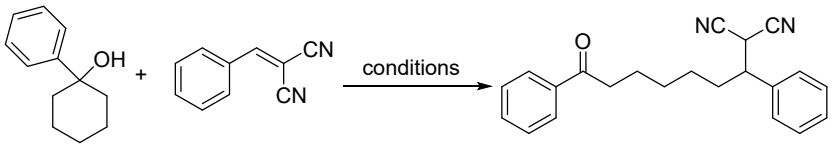


Table S1. Optimization for this study

entry	catalyst	additive	base	light	solvent	yield (%)[b]
1	$FeCl_3$ (10 mol%)	none	2,4,6-collidine	450nm (50W*2)	MeCN	0
2	$FeCl_3$ (10 mol%)	TBACl (5 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	14
3	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	83
4	$FeCl_3$ (10 mol%)	TBACl (20 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	80
5	$FeCl_3$ (10 mol%)	TBACl (30 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	69
6	$FeCl_3$ (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	40
7	$FeCl_3$ (5 mol%)	TBACl (5 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	63
8	$FeCl_3$ (1 mol%)	TBACl (1 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	28
9	$Fe_2(SO_4)_3$ (5 mol%)	None	2,4,6-collidine	450nm (50W*2)	MeCN	0
10	$Fe_2(SO_4)_3$ (5 mol%)	TBACl (40 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	trace

11	$Fe_2(SO_4)_3$ (5 mol%)	TBACl (50 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	trace
12	$Fe(NO_3)_3.9H_2O$ (10 mol%)	None	2,4,6-collidine	450nm (50W*2)	MeCN	0
13	$Fe(NO_3)_3.9H_2O$ (10 mol%)	TBACl (40 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	56
14	$Fe(NO_3)_3.9H_2O$ (10 mol%)	TBACl (50 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	63
15	$Fe(OAc)_2OH$ (10 mol%)	None	2,4,6-collidine	450nm (50W*2)	MeCN	0
16	$Fe(OAc)_2OH$ (10 mol%)	TBACl (40 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	11
17	$Fe(OAc)_2OH$ (10 mol%)	TBACl (50 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	13
18	$FeBr_3$ (10 mol%)	None	2,4,6-collidine	450nm (50W*2)	MeCN	0
19	$FeBr_3$ (10 mol%)	TBACl (40 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	45
20	$FeBr_3$ (10 mol%)	TBACl (50 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	47
21	$Fe(OTf)_3$ (10 mol%)	None	2,4,6-collidine	450nm (50W*2)	MeCN	0
22	$Fe(OTf)_3$ (10 mol%)	TBACl (40 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	26
23	$Fe(OTf)_3$ (10 mol%)	TBACl (50 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	21
24	**PC1** (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	0
25	**PC2** (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	0
26	**PC3** (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	0
27	**PC4** (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	0
28	**PC5** (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	0
29	$CeCl_3$ (5 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	0
30[e]	$FeCl_3$ (10 mol%)	LiCl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	12
31[e]	$FeCl_3$ (10 mol%)	NaCl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	19
32[e]	$FeCl_3$ (10 mol%)	NH_4Cl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	13
33[e]	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	67
34	$FeCl_3$ (10 mol%)	TEACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	78
35	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	81
36	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	none	450nm (50W*2)	MeCN	0
37	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,6-lutidine	450nm (50W*2)	MeCN	62
38	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	pyridine	450nm (50W*2)	MeCN	9
39	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	Et_3N	450nm (50W*2)	MeCN	0
40	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	K_2CO_3	450nm (50W*2)	MeCN	0
41	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	$NaHCO_3$	450nm (50W*2)	MeCN	15

42	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine (0.5 equiv.)	450nm (50W*2)	MeCN	67
43	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine (2.0 equiv.)	450nm (50W*2)	MeCN	80
44	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	None	MeCN	0
45	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	450nm (50W)	MeCN	64
46	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	450nm (24W)	MeCN	57
47	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	400nm (24W)	MeCN	0
48	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	365nm (24W)	MeCN	0
49	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	450nm (50W*2)	DCE	50
50	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	450nm (50W*2)	DCM	55
51[d]	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	74
52[e]	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	94
53[e]	$FeCl_3$ (10 mol%)	TBPCl (10 mol%)	2,4,6-collidine	450nm (50W*2)	MeCN	91
54[f]	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	450nm (24W)	MeCN	61
55[f]	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	white (24W)	MeCN	26
56[f]	$FeCl_3$ (10 mol%)	TBACl (10 mol%)	2,4,6-collidine	sunlight	MeCN	15

[a]Reaction conditions: alcohol (0.4 mmol), alkene (0.2 mmol), catalyst, additive, base (0.2 mmol, 1.0 equiv.), and solvent (2 mL, 0.1 M) were irradiated with LEDs under N_2 for 12 h.

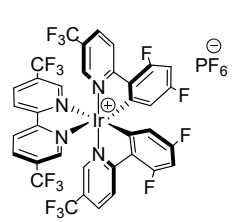
[b]Determined by ^1H NMR analysis using 1,3,5-Trimethoxybenzene as an internal standard.

[c]alcohol (0.8 mmol), alkene (0.4 mmol), catalyst, additive, base (0.4 mmol, 1.0 equiv.), and solvent (4 mL, 0.1 M) were irradiated with LEDs under N_2 for 12 h.

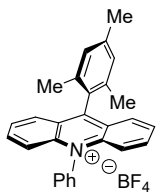
[d]alcohol (0.2 mmol), alkene (0.4 mmol), catalyst, additive, base (0.2 mmol, 1.0 equiv.), and solvent (2 mL, 0.1 M) were irradiated with LEDs under N_2 for 12 h.

[e]48 h, isolated yield.

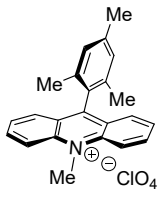
[f]without stirring.



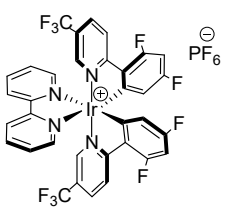
PC1



PC2



PC3



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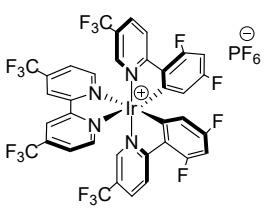
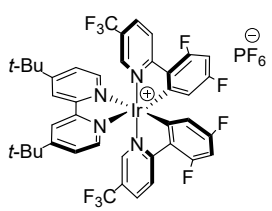
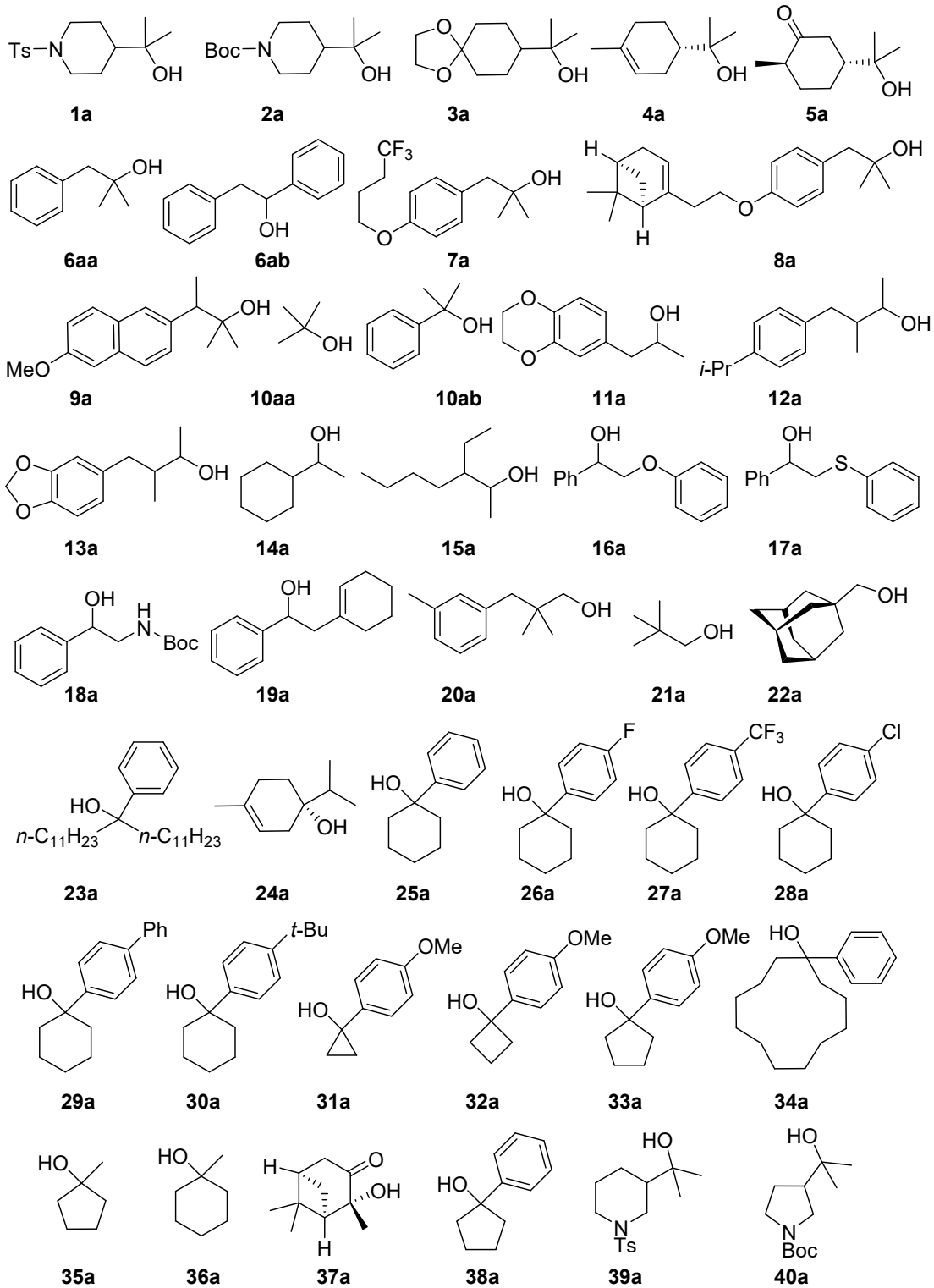


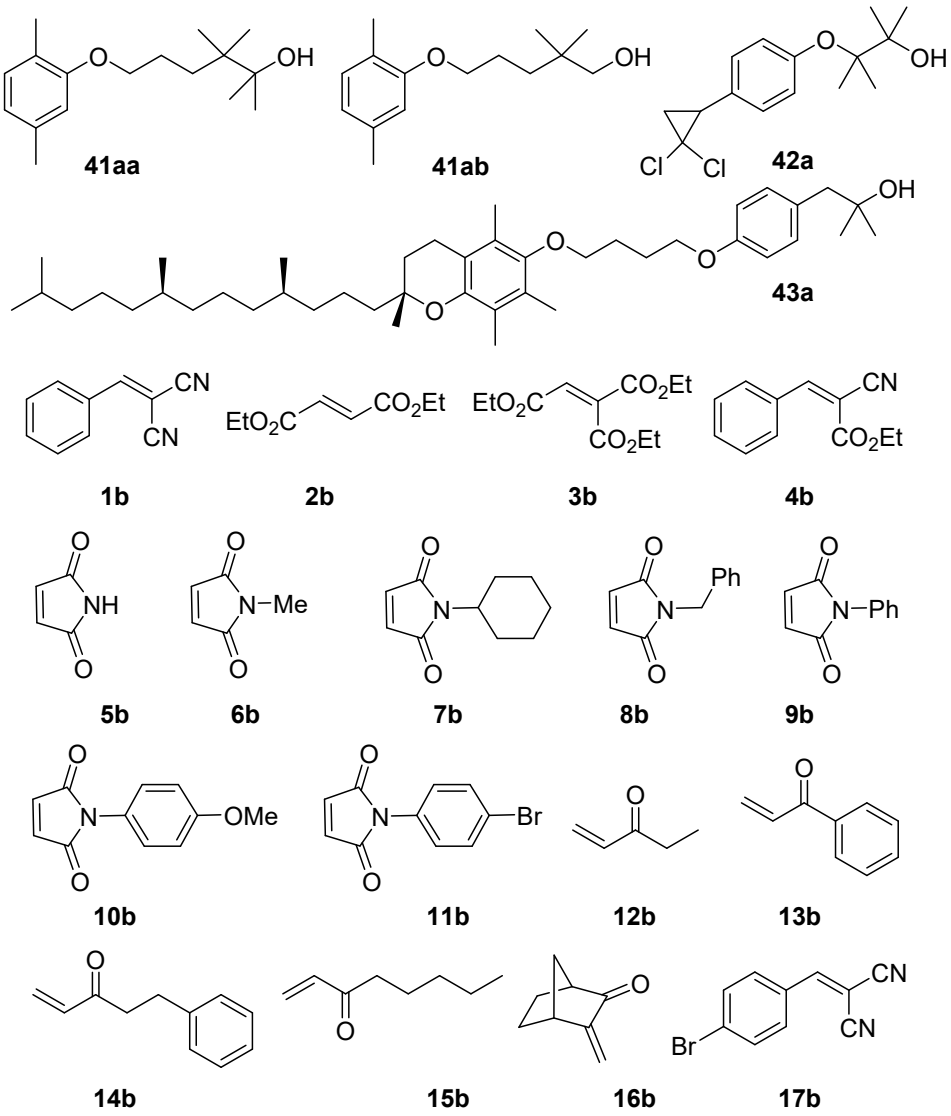
PC5



PC6

3.2 Preparation of Starting Materials





16a, **17a**, **23a**, **25a**, **26a**, **27a**, **28a**, **29a**, **30a**, **31a**, **32a**, **33a**, **34a** were synthesized and used in our laboratory. For details about the procedures and spectral data, please see our previous work.[1] **4a**, **6aa**, **6ab**, **10aa**, **10ab**, **14a**, **18a**, **20a**, **21a**, **22a**, **24a**, **35a**, **36a**, **37a**, **1b**, **2b**, **3b**, **4b**, **5b**, **6b**, **7b**, **8b**, **9b**, **10b**, **11b**, **12b**, **13b**, **15b**, **16b** were purchased and used without further purification. **1a**, **2a**, **3a**, **5a**, **7a**, **8a**, **9a**, **11a**, **12a**, **13a**, **15a**, **19a**, **38a**, **39a**, **40a**, **41aa**, **41ab**, **42a**, **43a**, **14b**, **17b** were synthesized and the procedures are shown below:

General procedure for the methylation of carboxylic acids:

A 250 mL round bottom flask was charged with a stir bar. To a solution of relevant carboxylic acids (0.25 M in DMF) was added potassium carbonate (1.0 equiv.) and iodomethane (1.2 equiv.). The reaction mixture was stirred for 16 hours at room temperature, then extracted with ethyl acetate. The combined organic layers were washed with brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The crude product was purified via column chromatography to

afford the desired esters for the next step.

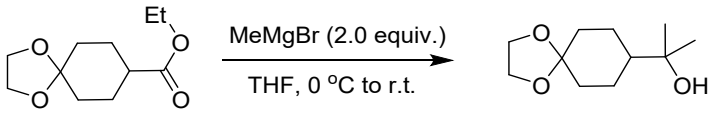
General procedure for synthesis of starting materials with Grignard solution:

A flame-dried round bottom flask was charged with a stir bar and degassed. To a solution of relevant esters, ketones, and aldehydes (0.2 M in anyhydrous THF) was added commercial grignard solution (l M in THF, 2.0 equiv.) dropwise at 0 °C. Then the reaction mixture was stirred at room temperature. Upon completion, the reaction mixture was quenched by slow addition of ice water. The aqueous layer was extracted with ethyl acetate and combined organic layers were washed with brine, dried over anhydrous sodium sulfate. Concentrated and purified by silica gel flash column chromatography to obtain the pure alcohols.

1a was synthesized according to a reported procedure.[2] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.64 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 7.8 Hz, 2H), 3.92 – 3.82 (m, 2H), 2.43 (s, 3H), 2.22 – 2.12 (m, 2H), 1.84 – 1.74 (m, 2H), 1.67 – 1.62 (m, 1H), 1.49 – 1.36 (m, 2H), 1.25 – 1.19 (m, 1H), 1.14 (s, 6H).

2a was synthesized according to a reported procedure.[2] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.23 – 4.11 (m, 2H), 2.66 – 2.56 (m, 2H), 1.76 – 1.66 (m, 2H), 1.49 – 1.33 (m, 11H), 1.24 – 1.09 (m, 8H).

3a was synthesized according to the following procedures:

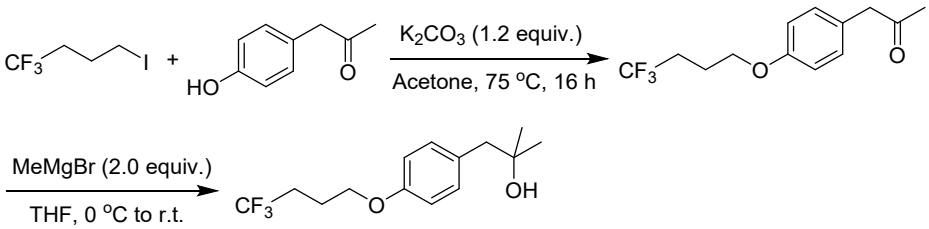


3a

Following the general procedure with ethyl 1,4-dioxaspiro[4.5]decane-8-carboxylate (2.14 g, 10 mmol, 1.0 equiv) to obtain **3a** as a colorless oil (1.62 g, 81%). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 3.94 (s, 4H), 1.85 – 1.77 (m, 4H), 1.62 – 1.50 (m, 3H), 1.39 – 1.31 (m, 3H), 1.19 (s, 6H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 108.9, 72.7, 64.3, 64.3, 48.1, 34.9, 27.1, 24.9. **HRMS (ESI)**: calculated for $C_{11}H_{20}O_3$ $[M+Na]^+$: 223.1305; found: 223.1301.

5a was synthesized according to a reported procedure.[3] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 2.50 – 2.40 (m, 1H), 2.35 – 2.21 (m, 1H), 2.18 – 1.87 (m, 4H), 1.77 – 1.60 (m, 1H), 1.54 – 1.38 (m, 1H), 1.17 – 1.09 (m, 6H), 0.99 – 0.86 (m, 3H).

7a was synthesized according to the following procedures:

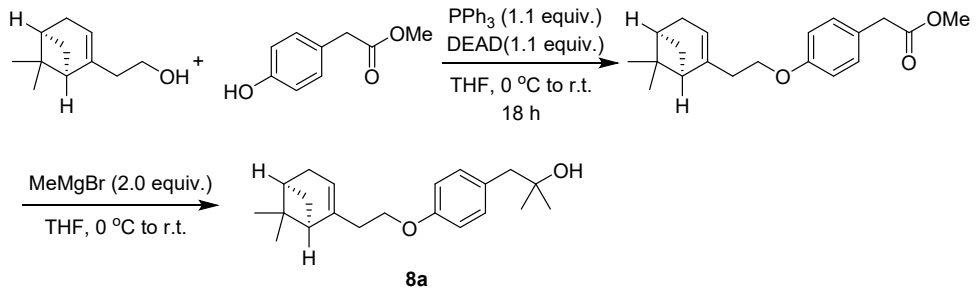


7a

For step one, 1,1,1-trifluoro-4-iodobutane (8.57 g, 36 mmol, 1.2 equiv.), 1-(4-hydroxyphenyl)propan-2-one (4.51 g, 30 mmol, 1.0 equiv.), and potassium carbonate (4.98 g, 36 mmol, 1.2 equiv.) were dissolved in acetone (80 mL). The reaction mixture was stirred at 75 °C for 16 h. Then the reaction mixture was cooled to room temperature, filtered through a thin pad of silica gel, and purified by flash column chromatography to obtain the relevant ketone as a colorless oil (6.32 g, 81 %). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.11 (d, *J* = 8.6 Hz, 2H), 6.85 (d, *J* = 8.6 Hz, 2H), 4.00 (t, *J* = 6.0 Hz, 2H), 3.63 (s, 2H), 2.36 – 2.26 (m, 2H), 2.14 (s, 3H), 2.07 – 2.01 (m, 2H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 206.9, 157.8, 130.6, 127.3 (q, *J* = 276.1 Hz), 126.8, 114.8, 66.1, 50.2, 30.8 (q, *J* = 29.1 Hz), 29.3, 22.3 (q, *J* = 2.9 Hz). **^{19}F NMR (377 MHz, Chloroform-*d*)** δ -66.32.

For step two, to a solution of 1-(4-(4,4,4-trifluorobutoxy)phenyl)propan-2-one (2.6 g, 10 mmol) in THF (50 mL) was added commercial methylmagnesium bromide solution (20 mL, 20 mmol, 2.0 equiv.) dropwise at 0 °C. Then following the general procedure to obtain **7a** as a colorless oil (1.60 g, 58%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.13 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 8.3 Hz, 2H), 4.00 (t, *J* = 6.0 Hz, 2H), 2.70 (s, 2H), 2.39 – 2.24 (m, 2H), 2.08 – 2.00 (m, 2H), 1.21 (s, 6H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 157.5, 131.6, 130.4, 127.3 (q, *J* = 275.9 Hz), 114.4, 70.9, 66.2, 49.0, 30.9 (q, *J* = 29.0 Hz), 29.3, 22.4 (q, *J* = 3.1 Hz). **^{19}F NMR (377 MHz, Chloroform-*d*)** δ -66.31. **HRMS (ESI)**: calculated for $C_{14}H_{19}F_3O_2$ $[M+Na]^+$: 299.1229; found: 299.1224.

8a was synthesized according to the following procedures:



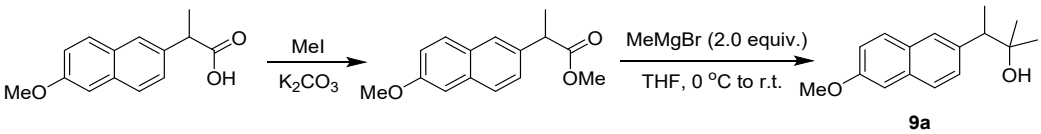
8a

For step one, following the typical Mitsunobu reaction procedure, a flame-dried 100 mL round

bottom flask was charged with a stir bar and degassed. 2-((1R,5S)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)ethan-1-ol (930 mg, 5.6 mmol), methyl 2-(4-hydroxyphenyl)acetate (930 mg, 5.6 mmol, 1.0 equiv.), and PPh_3 (1.63 g, 6.2 mmol, 1.1 equiv.) were dissolved in THF (20 mL). Then DEAD (6.2 mmol, 1.1 equiv., 40% in toluene) solution was added dropwise to the reaction mixture at 0 °C. The reaction mixture was stirred at room temperature for 18 h, concentrated under vacuum and purified by silica gel flash column chromatography to obtain the relevant ester as a colorless oil (757 mg, 43 %) with good purity and used in the next step directly. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.17 (d, *J* = 8.3 Hz, 2H), 6.83 (d, *J* = 8.4 Hz, 2H), 5.37 – 5.31 (m, 1H), 3.95 (t, *J* = 7.0 Hz, 2H), 3.68 (s, 3H), 3.55 (s, 2H), 2.50 – 2.40 (m, 2H), 2.40 – 2.34 (m, 1H), 2.31 – 2.16 (m, 2H), 2.12 – 2.06 (m, 2H), 1.27 (s, 3H), 1.18 (d, *J* = 8.5 Hz, 1H), 0.83 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 172.5, 158.2, 144.6, 130.3, 126.0, 118.7, 114.8, 66.5, 52.1, 46.0, 40.9, 40.4, 38.2, 36.7, 31.8, 31.5, 26.4, 21.3.

For step two, to a solution of relevant ester (1.57 g, 5 mmol, 1.0 equiv.) prepared in step one in THF (25 mL) was added commercial methylmagnesium bromide solution (10 mL, 10 mmol, 2.0 equiv.) dropwise at 0 °C. Then following the general procedure to obtain **8a** as a colorless oil (1.13 g, 72%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.10 (d, *J* = 8.2 Hz, 2H), 6.83 (d, *J* = 8.2 Hz, 2H), 5.37 – 5.29 (m, 1H), 3.95 (t, *J* = 7.1 Hz, 2H), 2.69 (s, 2H), 2.52 – 2.33 (m, 3H), 2.32 – 2.16 (m, 2H), 2.13 – 2.05 (m, 2H), 1.49 – 1.40 (m, 2H), 1.28 (s, 3H), 1.20 (s, 6H), 0.84 (s, 3H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 157.8, 144.7, 131.4, 129.7, 118.6, 114.4, 70.9, 66.5, 48.9, 46.0, 40.8, 38.2, 36.7, 31.8, 31.5, 29.2, 26.4, 21.3. **HRMS (APCI)**: calculated for $C_{21}H_{30}O_2$ $[M+Na]^+$: 337.2138; found: 337.2132.

9a was synthesized according to the following procedures:

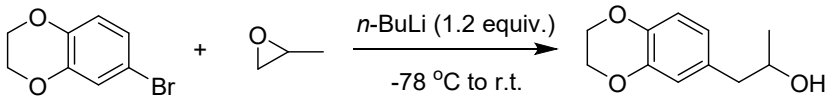


9a

The step one was prepared according to a reported procedure.[4] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.71 – 7.62 (m, 3H), 7.38 (d, *J* = 8.3 Hz, 1H), 7.16 – 7.05 (m, 2H), 3.89 – 3.79 (m, 4H), 3.63 (s, 3H), 1.56 (d, *J* = 6.9 Hz, 3H). For step two, following the general procedure with methyl 2-(6-methoxynaphthalen-2-yl)propanoate (2.44 g, 10 mmol) to obtain **9a** as a colorless oil (2.03 g, 83%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.72 – 7.65 (m, 2H), 7.63 – 7.59 (m, 1H), 7.40 –

7.35 (m, 1H), 7.17 – 7.10 (m, 2H), 3.91 (s, 3H), 2.93 (q, *J* = 7.2 Hz, 1H), 1.41 (d, *J* = 7.2 Hz, 3H), 1.21 (s, 6H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 157.5, 138.7, 133.5, 129.3, 128.9, 128.2, 127.4, 126.5, 118.9, 105.6, 73.0, 55.4, 50.4, 28.3, 27.1, 16.1. **HRMS (ESI)**: calculated for $C_{16}H_{20}O_2$ $[M+Na]^+$: 267.1356; found: 267.1351.

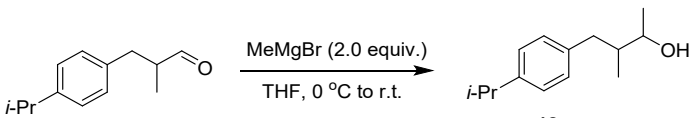
11a was synthesized according to the following procedures:



11a

A flame-dried 100 mL Schlenk tube was charged with a stir bar and degassed. To a solution of 6-bromo-2,3-dihydrobenzo[b][1,4]dioxine (2.15 g, 10 mmol, 1.0 equiv.) in THF (50 mL) was added commercial *n*-BuLi solution (12 mmol, 1.2 equiv.) dropwise at -78 °C. Then 2-methyloxirane (1.16 g, 20 mmol, 2.0 equiv.) was added to the mixture dropwise at -78 °C. The reaction mixture was warmed and stirred at room temperature for 18 h, concentrated under vacuum and purified by silica gel flash column chromatography to obtain **11a** as a colorless oil (621 mg, 32 %). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.82 – 6.79 (m, 1H), 6.74 – 6.71 (m, 1H), 6.69 – 6.65 (m, 1H), 4.24 (s, 4H), 4.00 – 3.91 (m, 1H), 2.72 – 2.64 (m, 1H), 2.61 – 2.52 (m, 1H), 1.23 (d, *J* = 6.2 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 143.6, 142.3, 131.8, 122.4, 118.1, 117.4, 69.0, 64.5, 64.4, 45.1, 22.8. **HRMS (APCI)**: calculated for $C_{11}H_{14}O_3$ $[M-OH]^+$: 177.0905; found: 177.0906.

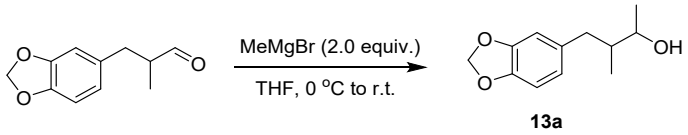
12a was synthesized according to the following procedures:



12a

Following the general procedure with 3-(4-isopropylphenyl)-2-methylpropanal (1.90 g, 10 mmol, 1.0 equiv) to obtain **12a** as a colorless oil (1.50 g, 73%). The dr ratio is 1.3:1, determined by ^{1}H NMR. The product gives two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.14 – 7.05 (m, 9.19H), 3.77 – 3.70 (m, 1.00H), 3.69 – 3.61 (m, 1.27H), 2.90 – 2.76 (m, 4.57H), 2.38 – 2.16 (m, 4.54H), 1.84 – 1.70 (m, 2.31H), 1.25 – 1.20 (m, 13.11H), 1.18 – 1.14 (m, 6.83H), 0.84 (d, *J* = 6.8 Hz, 2.94H), 0.82 (d, *J* = 6.8 Hz, 3.81H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 146.2, 138.5, 138.3, 129.1, 126.3, 126.3, 71.2, 70.3, 42.3, 41.8, 38.9, 38.8, 33.7, 24.1, 20.4, 19.6, 14.6, 13.7. **HRMS (ESI)**: calculated for $C_{14}H_{22}O$ $[M+Na]^+$: 229.1563; found: 229.1559.

13a was synthesized according to the following procedures:



13a

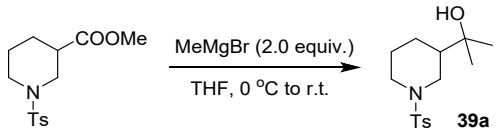
Following the general procedure with 3-(benzo[d][1,3]dioxol-5-yl) -2-methylpropanal (1.92 g, 10 mmol, 1.0 equiv) to obtain **13a** as a colorless oil (1.62 g, 78%). The dr ratio is 1.2:1, determined by ^{1}H NMR. The product gives two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.75 – 6.70 (m, 2.09H), 6.69 – 6.65 (m, 2.08H), 6.64 – 6.58 (m, 2.14H), 5.91 (s, 4.32H), 3.78 – 3.71 (m, 1.00H), 3.71 – 3.63 (m, 1.16H), 2.82 – 2.69 (m, 2.21H), 2.36 – 2.23 (m, 2.23H), 1.80 – 1.66 (m, 2.67H), 1.23 – 1.14 (m, 6.61H), 0.85 (d, *J* = 6.8 Hz, 3.01H), 0.82 (d, *J* = 6.8 Hz, 3.50H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 147.6, 147.6, 145.7, 135.1, 134.9, 122.0, 122.0, 109.6, 109.6, 108.1, 108.1, 100.8, 71.4, 70.3, 42.5, 42.0, 39.2, 38.9, 20.7, 19.9, 14.7, 13.6. **HRMS (ESI)**: calculated for $C_{12}H_{16}O_3$ $[M+Na]^+$: 231.0992; found: 231.0988.

15a was synthesized according to a reported procedure.[5] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 3.87 – 3.80 (m, 1H), 1.43 – 1.20 (m, 9H), 1.17 – 1.11 (m, 3H), 0.95 – 0.86 (m, 6H).

19a was synthesized according to a reported procedure.[6] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.38 – 7.30 (m, 4H), 7.28 – 7.22 (m, 1H), 5.61 – 5.55 (m, 1H), 4.77 – 4.71 (m, 1H), 2.38 – 2.22 (m, 3H), 2.10 – 1.99 (m, 3H), 1.96 – 1.86 (m, 1H), 1.68 – 1.53 (m, 4H).

38a was synthesized according to a reported procedure.[7] **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.52 – 7.47 (m, 2H), 7.34 (t, *J* = 7.7 Hz, 2H), 7.27 – 7.23 (m, 1H), 2.05 – 1.95 (m, 6H), 1.88 – 1.80 (m, 2H).

39a was synthesized according to the following procedures:

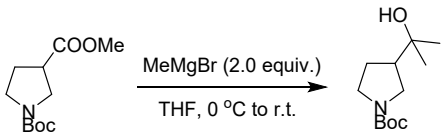


39a

Following the general procedure with methyl 1-tosylpiperidine-3-carboxylate (2.97 g, 10 mmol, 1.0 equiv) to obtain **39a** as a white solid (1.55 g, 52%).**^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.62 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 7.1 Hz, 2H), 4.00 – 3.91 (m, 1H), 3.79 – 3.69 (m, 1H), 2.39 (s, 3H), 2.15 – 1.94 (m, 3H), 1.86 – 1.70 (m, 2H), 1.67 – 1.49 (m, 2H), 1.16 (s, 3H), 1.10 (s, 3H),

1.03 – 0.88 (m, 1H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 143.4, 133.4, 129.7, 127.7, 71.7, 47.7, 46.6, 46.6, 28.2, 26.7, 25.1, 25.0, 21.6. **HRMS (ESI)**: calculated for $C_{15}H_{23}NO_3S$ $[M+Na]^+$: 320.1291; found: 320.1285.

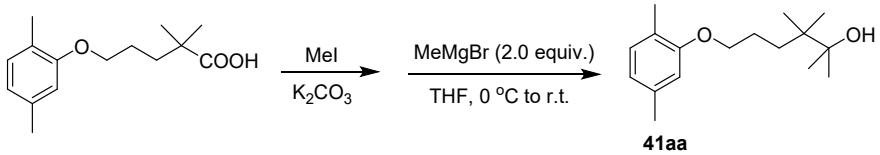
40a was synthesized according to the following procedures:



40a

Following the general procedure with 1-(tert-butyl) 3-methyl pyrrolidine-1,3-dicarboxylate (2.29 g, 10 mmol, 1.0 equiv) to obtain **40a** as a white solid (1.58 g, 69%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 3.59 – 3.36 (m, 2H), 3.28 – 3.18 (m, 1H), 3.17 – 3.08 (m, 1H), 2.28 – 2.13 (m, 1H), 1.91 – 1.67 (m, 2H), 1.44 (s, 9H), 1.22 (s, 6H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 154.8, 79.2, 70.6, 70.5, 49.8, 49.0, 46.9, 46.7, 46.3, 45.9, 28.7, 28.5, 28.2, 28.2, 26.5, 25.9. **HRMS (ESI)**: calculated for $C_{12}H_{23}NO_3$ $[M+Na]^+$: 252.1570; found: 252.1565.

41aa was synthesized according to the following procedures:

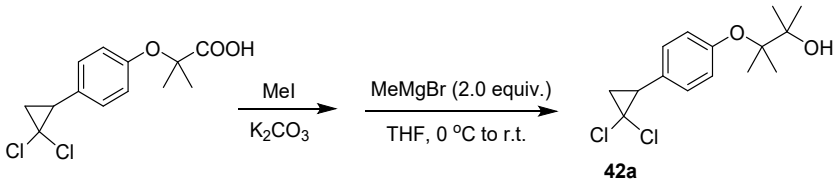


41aa

The step one was prepared according to a reported procedure.[8] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.03 (d, *J* = 7.4 Hz, 1H), 6.68 (d, *J* = 7.5 Hz, 1H), 6.64 (s, 1H), 3.97 – 3.90 (m, 2H), 3.69 (s, 3H), 2.34 (s, 3H), 2.21 (s, 3H), 1.79 – 1.72 (m, 4H), 1.26 (s, 6H). For step two, following the general procedure with methyl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (2.64 g, 10 mmol) to obtain **41aa** as a colorless oil (1.37 g, 52%).**^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.02 (d, *J* = 7.3 Hz, 1H), 6.70 – 6.63 (m, 2H), 3.95 (t, *J* = 6.3 Hz, 2H), 2.33 (s, 3H), 2.20 (s, 3H), 1.87 – 1.77 (m, 2H), 1.58 – 1.49 (m, 2H), 1.23 (s, 6H), 0.96 (s, 6H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 157.2, 136.5, 130.4, 123.7, 120.7, 112.1, 75.7, 68.8, 39.8, 33.3, 25.5, 25.1, 21.6, 21.5, 15.9. **HRMS (ESI)**: calculated for $C_{17}H_{28}O_2$ $[M+Na]^+$: 287.1982; found: 287.1977.

41ab was synthesized according to a reported procedure.[9] **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.05 (d, *J* = 7.4 Hz, 1H), 6.72 – 6.66 (m, 2H), 3.96 (t, *J* = 6.5 Hz, 2H), 3.39 (s, 2H), 2.36 (s, 3H), 2.24 (s, 3H), 1.86 – 1.76 (m, 2H), 1.49 – 1.43 (m, 2H), 0.97 (s, 6H).

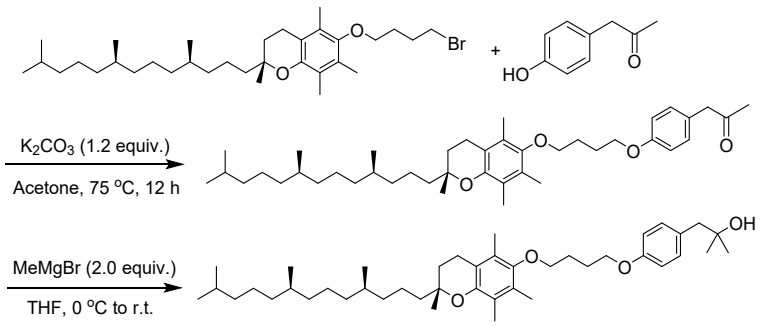
42a was synthesized according to the following procedures:



42a

The step one was prepared according to a reported procedure.[10] **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.11 (d, *J* = 8.6 Hz, 2H), 6.80 (d, *J* = 8.6 Hz, 2H), 3.76 (s, 3H), 2.85 – 2.80 (m, 1H), 1.95 – 1.91 (m, 1H), 1.77 (t, *J* = 7.9 Hz, 1H), 1.59 (s, 6H). For step two, following the general procedure with methyl 2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methylpropanoate (3.03 g, 10 mmol) to obtain **42a** as a colorless oil (879 mg, 29%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.16 (d, *J* = 8.4 Hz, 2H), 6.96 (d, *J* = 8.6 Hz, 2H), 2.91 – 2.83 (m, 1H), 2.76 (s, 1H), 2.00 – 1.93 (m, 1H), 1.84 – 1.78 (m, 1H), 1.33 (s, 6H), 1.24 (s, 6H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 154.3, 130.1, 129.6, 124.2, 85.8, 75.2, 60.9, 35.1, 26.0, 24.8, 21.6.

43a was synthesized according to the following procedures:



43a

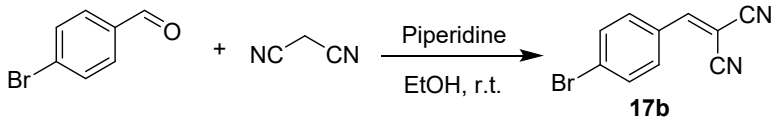
For step one, (R)-6-(4-bromobutoxy)-2,5,7,8-tetramethyl-2-((4R,8R)-4,8,12-trimethyltridecyl)chromane (5.66 g, 10 mmol, 1.0 equiv.), potassium carbonate (1.66 g, 12 mmol, 1.2 equiv.), and 1-(4-hydroxyphenyl)propan-2-one (1.80 g, 12 mmol, 1.2 equiv.) were dissolved in acetone (25 mL). The reaction mixture was stirred at 75 °C for 12 h. Then the reaction mixture was cooled to room temperature, filtered through a thin pad of silica gel, and purified by flash column chromatography to obtain the relevant ketone as a colorless oil (1.84 g, 29 %). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.09 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 4.03 (t, *J* = 6.1 Hz, 2H), 3.70 (t, *J* = 6.0 Hz, 2H), 3.60 (s, 2H), 2.56 (t, *J* = 6.7 Hz, 2H), 2.17 (s, 3H), 2.14 – 2.10 (m, 6H), 2.08 (s, 3H), 2.05 – 1.92 (m, 4H), 1.85 – 1.71 (m, 2H), 1.60 – 1.48 (m, 3H), 1.47 – 1.34 (m, 4H), 1.33 – 1.03 (m, 17H), 0.90 – 0.81 (m, 12H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 207.0, 158.4, 148.4, 147.9, 130.5, 127.9, 126.3, 125.9, 123.0, 117.6, 115.0, 74.9, 72.6, 68.0, 50.3, 40.2,

39.5, 37.6, 37.6, 37.6, 37.4, 32.9, 32.8, 31.5, 29.2, 28.1, 27.2, 26.5, 24.9, 24.6, 24.0, 22.9, 22.8, 21.2, 20.8, 19.9, 19.8, 12.9, 12.1, 11.9. **HRMS (ESI)**: calculated for $C_{42}H_{66}O_4$ $[M+Na]^+$: 657.4853; found: 657.4840.

For step two, to a solution of 1-(4-(4-(((R)-2,5,7,8-tetramethyl-2-((4R,8R) -4,8,12-trimethyltridecyl)chroman-6-yl)oxy)butoxy)phenyl)propan-2-one (3.18 g, 5 mmol) in THF (25 mL) was added commercial methylmagnesium bromide solution (10 mL, 10 mmol, 2.0 equiv.) dropwise at 0 °C. Then following the general procedure to obtain **43a** as a colorless oil (1.99 g, 61%). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.12 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 4.04 (t, *J* = 6.2 Hz, 2H), 3.71 (t, *J* = 6.2 Hz, 2H), 2.70 (s, 2H), 2.57 (t, *J* = 6.7 Hz, 2H), 2.17 (s, 3H), 2.13 (s, 3H), 2.08 (s, 3H), 2.06 – 2.00 (m, 2H), 1.99 – 1.94 (m, 2H), 1.84 – 1.72 (m, 2H), 1.57 – 1.49 (m, 3H), 1.45 – 1.19 (m, 22H), 1.16 – 1.03 (m, 6H), 0.89 – 0.82 (m, 12H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 158.0, 148.4, 147.9, 131.5, 129.8, 127.9, 125.9, 123.0, 117.7, 114.4, 74.9, 72.7, 70.9, 68.0, 49.0, 40.2, 39.5, 37.7, 37.6, 37.5, 37.4, 33.0, 32.9, 31.5, 29.2, 28.1, 27.2, 26.5, 25.0, 24.6, 24.0, 22.9, 22.8, 21.2, 20.8, 19.9, 19.8, 12.9, 12.1, 11.9. **HRMS (ESI)**: calculated for $C_{43}H_{70}O_4$ $[M+Na]^+$: 673.5166; found: 673.5152.

14b was synthesized according to a reported procedure.[11] **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.31 – 7.26 (m, 2H), 7.23 – 7.17 (m, 3H), 6.39 – 6.32 (m, 1H), 6.21 (d, *J* = 17.7 Hz, 1H), 5.83 (d, *J* = 10.7 Hz, 1H), 2.97 – 2.89 (m, 4H).

17b was synthesized according to the knoevenagel condensation reaction.

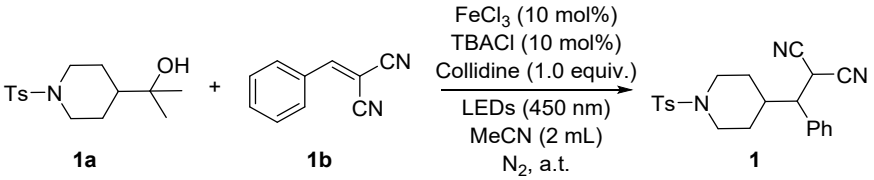


17b

A 100 mL Schlenk tube was charged with a stir bar. To a solution of 4-bromobenzaldehyde (1.85 g, 10 mmol, 1.0 equiv.), malononitrile (660 mg, 10 mmol, 1.0 equiv.) in EtOH (10 mL) was added piperidine (100 μL). The reaction mixture was warmed and stirred at room temperature for 6 h, purified by silica gel flash column chromatography to obtain **16b** as a light yellow solid (1.63 g, 70 %).**^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.79 – 7.76 (m, 2H), 7.73 – 7.67 (m, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 158.6, 133.2, 131.9, 130.1, 129.8, 113.6, 112.5, 83.7.

3.3 Synthesis of Products

General procedure:



$FeCl_3$ (97.3 mg, 0.6 mmol) and TBACl (166.8 mg, 0.6 mmol) were dissolved in MeCN (60 mL) with vigorous agitation in the glovebox. A 25 mL Schlenk tube with screw-cap was added relevant michael acceptor (0.2 mmol, 1.0 equiv., if solid), alcohol (0.4 mmol, 2.0 equiv., if solid). The Schlenk tube was then transferred into the glovebox, where $FeCl_3$/TBACl solution (2 mL) was added. Then, collidine (26 μL, 0.2 mmol, 1.0 equiv.), michael acceptor (0.2 mmol, 1.0 equiv., if liquid), and alcohol (0.4 mmol, 2.0 equiv, if liquid) were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two blue LED lamps and stirred at ambient temperature. After 12-72 hours, the reaction mixture was concentrated and then purified by flash column chromatography to obtain the product.

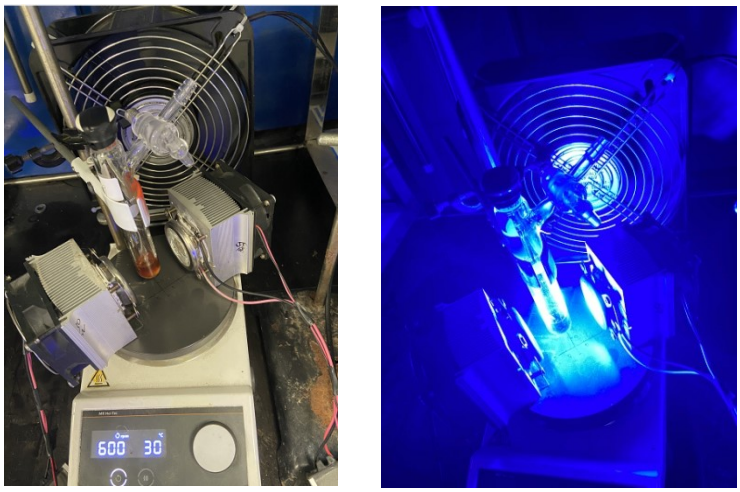
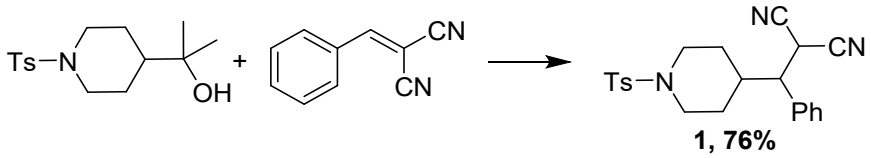


Figure S3. The reaction setup using Schlenk tube

Compound 1

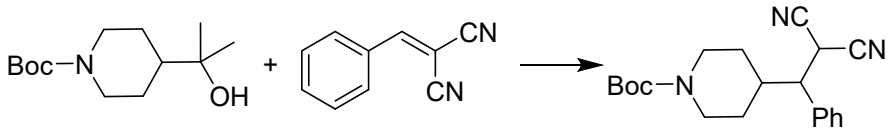


2-(phenyl(1-tosylpiperidin-4-yl)methyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and

benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was concentrated and then purified by flash column chromatography (silica gel, DCM) to afford the titled compound as a colorless oil (59.8 mg, 76%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.61 (d, *J* = 8.2 Hz, 2H), 7.43 – 7.36 (m, 3H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.30 – 7.24 (m, 2H), 4.16 (d, *J* = 5.3 Hz, 1H), 3.96 – 3.87 (m, 1H), 3.77 – 3.69 (m, 1H), 2.96 – 2.87 (m, 1H), 2.45 (s, 3H), 2.33 – 2.24 (m, 1H), 2.17 – 2.07 (m, 1H), 1.98 – 1.87 (m, 2H), 1.55 – 1.41 (m, 2H), 1.32 – 1.23 (m, 1H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 144.0, 135.7, 132.5, 129.9, 129.5, 129.3, 128.2, 127.8, 111.7, 51.2, 46.2, 46.0, 37.5, 29.8, 29.3, 27.2, 21.7. **HRMS (ESI)**: calculated for $C_{22}H_{23}N_3O_2S$ $[M+Na]^+$: 416.1403; found: 416.1395.

Compound 2

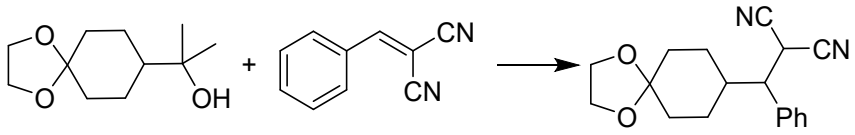


2, 70%

tert-butyl 4-(2,2-dicyano-1-phenylethyl)piperidine-1-carboxylate

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, Upon completion, the reaction mixture was concentrated and then purified by flash column chromatography (silica gel, 200:1 DCM/MeOH) to afford the titled compound as a colorless oil (47.5 mg, 70%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.47 – 7.27 (m, 5H), 4.34 – 3.90 (m, 3H), 2.95 – 2.86 (m, 1H), 2.85 – 2.71 (m, 1H), 2.70 – 2.54 (m, 1H), 2.24 – 2.10 (m, 1H), 1.93 – 1.81 (m, 1H), 1.43 (s, 9H), 1.33 – 1.23 (m, 2H), 1.10 – 0.96 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 154.6, 136.0, 129.5, 129.2, 128.3, 111.9, 111.8, 79.9, 51.8, 43.5, 38.2, 30.4, 28.5, 27.2. **HRMS (ESI)**: calculated for $C_{20}H_{25}N_3O_2$ $[M-H]^-$: 338.1874; found: 338.1871.

Compound 3



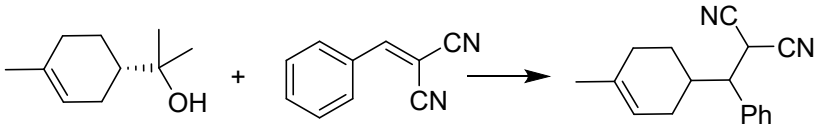
3, 76%

2-(phenyl(1,4-dioxaspiro[4.5]decan-8-yl)methyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and

benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was concentrated and then purified by flash column chromatography (silica gel, 200:1 DCM/MeOH) to afford the titled compound as a colorless oil (45.0 mg, 76%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.45 – 7.29 (m, 5H), 4.17 (d, *J* = 5.1 Hz, 1H), 4.00 – 3.86 (m, 4H), 2.95 – 2.87 (m, 1H), 2.13 – 2.01 (m, 1H), 1.98 – 1.83 (m, 2H), 1.72 – 1.62 (m, 2H), 1.54 – 1.41 (m, 3H), 1.27 – 1.15 (m, 1H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 136.8, 129.4, 129.1, 128.2, 112.1, 111.9, 108.1, 64.5, 64.4, 51.8, 38.1, 34.1, 28.5, 28.2, 27.7. **HRMS (ESI)**: calculated for $C_{18}H_{20}N_2O_2$ [M-H]$^-$: 295.1452; found: 295.1449.

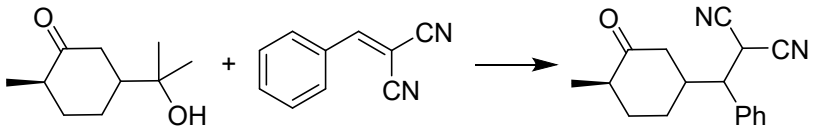
Compound 4



4, 61%, d.r.=1:1 **2-((4-methylcyclohex-3-en-1-yl)(phenyl)methyl)malononitrile**

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with dichloromethane. The solvent was removed and purified by flash column chromatography (silica gel, 40:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (30.5 mg, 61%). The dr ratio is 1:1, determined by ^{1}H NMR. The product gives two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.47 – 7.28 (m, 10H), 5.42 – 5.34 (m, 1H), 5.27 – 5.19 (m, 1H), 4.23 (d, *J* = 5.2 Hz, 1H), 4.15 (d, *J* = 5.0 Hz, 1H), 3.01 – 2.91 (m, 2H), 2.35 – 2.10 (m, 4H), 2.07 – 1.76 (m, 6H), 1.65 (s, 6H), 1.58 – 1.50 (m, 2H), 1.47 – 1.37 (m, 1H), 1.21 – 1.09 (m, 1H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 136.8, 136.7, 135.0, 133.7, 129.3, 129.2, 129.0, 128.9, 128.4, 128.4, 119.4, 118.4, 112.2, 112.0, 111.9, 51.5, 51.1, 35.4, 30.2, 29.8, 29.7, 29.5, 27.6, 27.5, 27.1, 26.8, 23.4, 23.3. **HRMS (ESI)**: calculated for $C_{17}H_{18}N_2$ [M-H]$^-$: 249.1397; found: 249.1398.

Compound 5

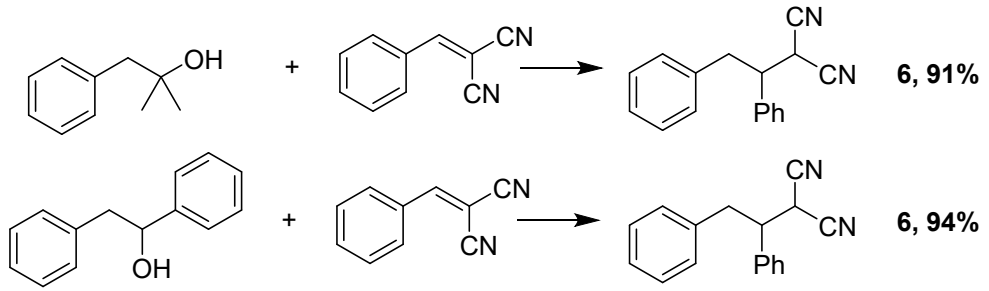


5, 72%, d.r.=1.2:1

2-(((4R)-4-methyl-3-oxocyclohexyl)(phenyl)methyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was concentrated and then purified by flash column chromatography (silica gel, $CHCl_3$) to afford the titled compound as a colorless oil (38.4 mg, 72%). The dr ratio is 1.2:1, determined by ^{1}H NMR. The small polar diastereoisomer: **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.45 – 7.39 (m, 3H), 7.33 – 7.27 (m, 2H), 4.19 (d, *J* = 5.5 Hz, 1H), 3.09 – 3.02 (m, 1H), 2.57 – 2.46 (m, 1H), 2.38 – 2.27 (m, 1H), 2.26 – 2.13 (m, 3H), 1.96 – 1.86 (m, 1H), 1.64 – 1.47 (m, 2H), 1.03 (d, *J* = 6.4 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 210.0, 135.3, 129.7, 129.5, 128.1, 111.7, 52.1, 45.2, 44.6, 41.4, 33.8, 30.3, 27.5, 14.2. The large polar diastereoisomer: **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.47 – 7.39 (m, 3H), 7.34 – 7.28 (m, 2H), 4.13 (d, *J* = 6.3 Hz, 1H), 3.09 – 3.03 (m, 1H), 2.66 – 2.59 (m, 1H), 2.56 – 2.45 (m, 1H), 2.36 – 2.25 (m, 1H), 2.15 – 2.01 (m, 2H), 1.77 – 1.68 (m, 1H), 1.40 – 1.30 (m, 2H), 1.03 (d, *J* = 6.5 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 209.6, 135.2, 129.6, 129.4, 128.4, 111.7, 111.5, 52.0, 46.1, 44.8, 41.3, 33.6, 28.9, 27.2, 14.3. **HRMS (ESI)**: calculated for $C_{17}H_{18}N_2O$ $[M-H]^-$: 265.1346; found: 265.1343.

Compound 6

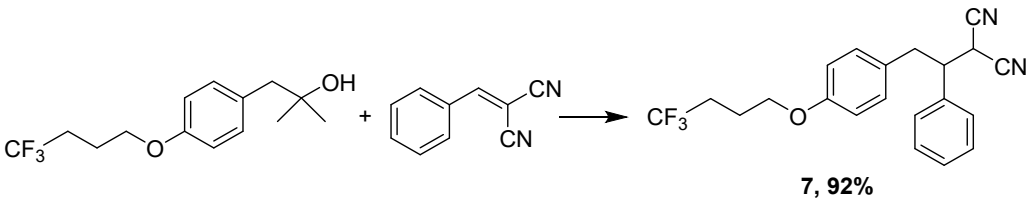


2-(1,2-diphenylethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a pale yellow oil (46.3 mg, 94%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.44 – 7.22 (m, 8H), 7.21 – 7.13 (m, 2H), 3.84 (d, *J* = 5.2 Hz, 1H), 3.49 – 3.41 (m, 1H), 3.31 – 3.20 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 136.7, 136.5, 129.3, 129.2, 129.2, 129.0, 128.1,

127.7, 112.2, 111.6, 48.4, 38.6, 28.6. **HRMS (ESI)**: calculated for $C_{17}H_{14}N_2$ [M-H]$^-$: 245.1084; found: 245.1083.

Compound 7

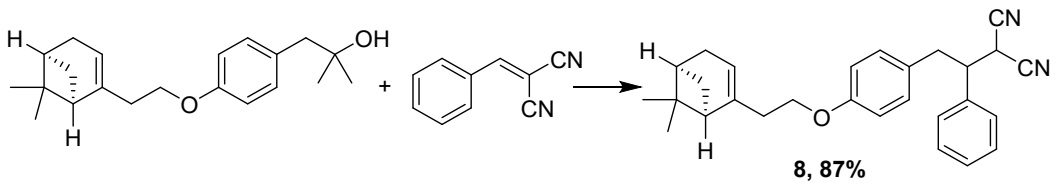


7, 92%

2-(1-phenyl-2-(4-(4,4,4-trifluorobutoxy)phenyl)ethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a pale yellow oil (68.5 mg, 92%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.45 – 7.36 (m, 5H), 7.10 (d, *J* = 8.5 Hz, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 3.99 (t, *J* = 6.0 Hz, 2H), 3.85 (d, *J* = 5.1 Hz, 1H), 3.44 – 3.37 (m, 1H), 3.24 – 3.15 (m, 2H), 2.38 – 2.24 (m, 2H), 2.09 – 2.00 (m, 2H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 158.1, 136.5, 130.2, (130.0, 126.3, 124.5 *are the peaks from CF_3 group, and one of the peaks overlapped with the 128.14 ppm peak*), 129.3, 129.2, 129.0, 128.1, 115.1, 112.2, 111.6, 66.1, 48.6, 37.8, 30.8 (q, *J* = 29.1 Hz), 28.5, 22.3 (q, *J* = 3.0 Hz). **^{19}F NMR (377 MHz, Chloroform-*d*)** δ -66.27. **HRMS (ESI)**: calculated for $C_{21}H_{19}F_3N_2O$ [M-H]$^-$: 371.1377; found: 371.1376.

Compound 8

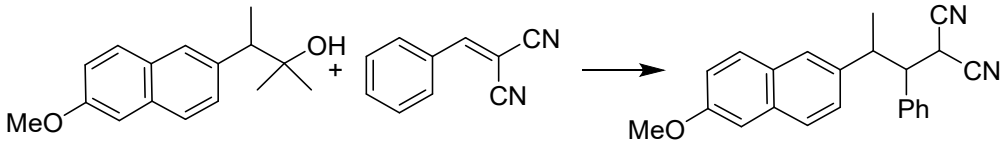


8, 87%

2-(2-(4-(2-((1R,5S)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)ethoxy)phenyl)-1-phenylethyl)malononitrile Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a pale yellow oil (71.4 mg, 87%). **^{1}H NMR (400 MHz,**

Chloroform-*d*) δ 7.45 – 7.36 (m, 5H), 7.09 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 5.38 – 5.31 (m, 1H), 3.94 (t, *J* = 7.0 Hz, 2H), 3.85 (d, *J* = 5.1 Hz, 1H), 3.44 – 3.36 (m, 1H), 3.22 – 3.15 (m, 2H), 2.47 – 2.34 (m, 3H), 2.32 – 2.17 (m, 2H), 2.10 (d, *J* = 5.2 Hz, 2H), 1.28 (s, 3H), 1.18 (d, *J* = 8.5 Hz, 1H), 0.83 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 158.5, 144.5, 136.6, 130.0, 129.3, 129.1, 128.4, 128.2, 118.8, 115.2, 112.3, 111.6, 66.5, 48.6, 46.0, 40.8, 38.2, 37.8, 36.6, 31.8, 31.5, 28.5, 26.4, 21.3. **HRMS (APCI)**: calculated for $C_{28}H_{30}N_2O$ $[M+H]^+$: 411.2431; found: 411.2420.

Compound 9

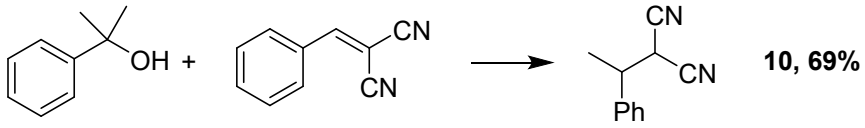


9, 77%, d.r.=1:1

2-(2-(6-methoxynaphthalen-2-yl)-1-phenylpropyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a pale yellow oil (52.4 mg, 77%). The dr ratio is 1:1, determined by ^{1}H NMR. The small polar diastereoisomer: **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.85 – 7.72 (m, 3H), 7.59 – 7.40 (m, 6H), 7.24 – 7.15 (m, 2H), 3.94 (s, 3H), 3.63 (d, *J* = 4.1 Hz, 1H), 3.59 – 3.49 (m, 1H), 3.34 – 3.26 (m, 1H), 1.21 (d, *J* = 6.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 158.2, 137.3, 135.7, 134.3, 129.4, 129.4, 129.3, 129.2, 128.7, 128.7, 126.5, 124.7, 119.8, 112.3, 111.7, 105.8, 55.5, 53.7, 42.2, 28.9, 20.8. The large polar diastereoisomer: **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.62 – 7.53 (m, 2H), 7.39 – 7.31 (m, 1H), 7.28 – 7.18 (m, 3H), 7.15 – 7.09 (m, 1H), 7.07 – 6.90 (m, 4H), 4.09 (d, *J* = 7.9 Hz, 1H), 3.88 (s, 3H), 3.70 – 3.61 (m, 1H), 3.51 (t, *J* = 7.8 Hz, 1H), 1.49 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 157.9, 135.7, 134.9, 133.8, 129.3, 129.0, 128.8, 128.7, 128.7, 127.1, 127.0, 126.5, 119.3, 112.7, 112.1, 105.6, 77.4, 55.4, 52.7, 41.6, 27.7, 20.4. **HRMS (ESI)**: calculated for $C_{23}H_{20}N_2O$ $[M+Na]^+$: 363.1468; found: 363.1460.

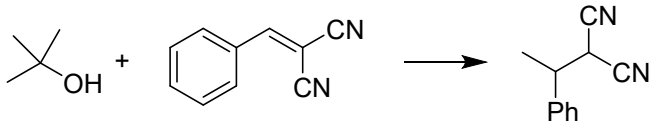
Compound 10



10, 69%

2-(1-phenylethyl)malononitrile

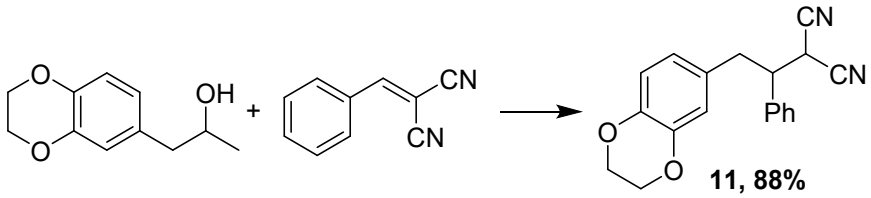
Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 40:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (23.5 mg, 69%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.43 – 7.31 (m, 5H), 3.85 (d, *J* = 6.2 Hz, 1H), 3.49 – 3.40 (m, 1H), 1.64 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 138.2, 129.3, 128.9, 127.3, 112.0, 111.8, 41.2, 31.2, 17.8. **HRMS (ESI)**: calculated for $C_{11}H_{10}N_2$ $[M-H]^-$: 169.0771; found: 169.0764.



10, 62%

Prepared on 0.2 mmol scale following the general procedure with *tert*-butanol (2 mmol, 10 equiv.) and benzylidenemalononitrile (30.8 mg, 0.2 mmol). Upon completion, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 40:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (21.1 mg, 62%).

Compound 11



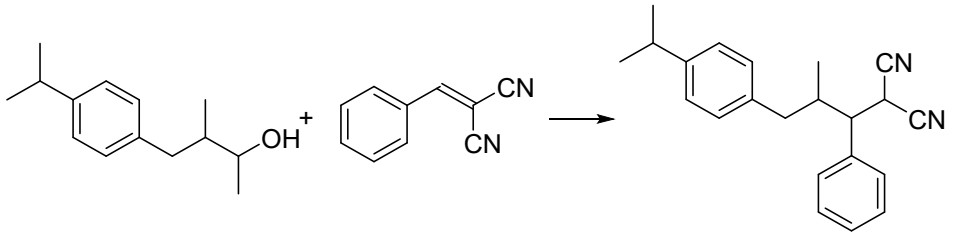
11, 88%

2-(2-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-1-phenylethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (53.6 mg, 88%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.44 –

7.36 (m, 5H), 6.83 – 6.63 (m, 3H), 4.23 (s, 4H), 3.87 (d, *J* = 5.0 Hz, 1H), 3.43 – 3.35 (m, 1H), 3.14 (d, *J* = 7.9 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.0, 143.1, 136.6, 129.8, 129.3, 129.1, 128.1, 121.9, 117.9, 117.6, 112.3, 111.5, 64.4, 64.4, 48.4, 37.8, 28.5. **HRMS (ESI)**: calculated for $C_{19}H_{16}N_2O_2$ [M-H]$^-$: 303.1139; found: 303.1136.

Compound 12

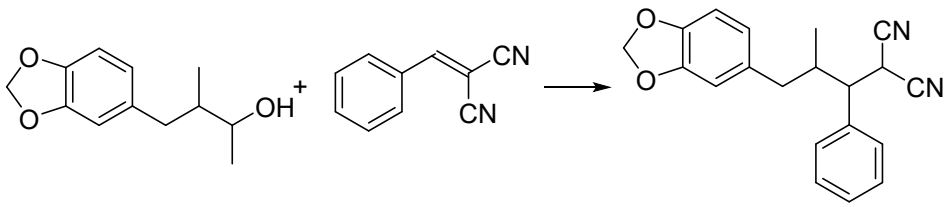


12, 73%, d.r.=1.2:1

2-(3-(4-isopropylphenyl)-2-methyl-1-phenylpropyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 3:2 Petroleum ether/DCM) to afford the titled compound as a colorless oil (46.2 mg, 73%). The dr ratio is 1.2:1, determined by ^{1}H NMR. The isolated product gives 1.4:1 dr ratio and two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.51 – 7.37 (m, 10.41H), 7.29 – 7.27 (m, 1.17H), 7.22 – 7.17 (m, 2.07H), 7.15 – 7.10 (m, 4.90H), 6.98 – 6.93 (m, 2.92H), 4.23 (d, *J* = 7.6 Hz, 0.99H), 4.18 (d, *J* = 5.4 Hz, 1.40H), 3.15 – 3.10 (m, 1.02H), 3.01 – 2.96 (m, 1.44H), 2.95 – 2.82 (m, 2.61H), 2.81 – 2.75 (m, 1.04H), 2.67 – 2.61 (m, 1.50H), 2.59 – 2.50 (m, 2.28H), 2.39 – 2.32 (m, 1.08H), 2.14 – 2.07 (m, 1.54H), 1.26 (d, *J* = 6.9 Hz, 6.94H), 1.23 (d, *J* = 6.9 Hz, 8.67H), 1.06 (d, *J* = 6.6 Hz, 4.23H), 0.80 (d, *J* = 6.7 Hz, 3.02H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 147.4, 147.2, 136.6, 136.4, 136.1, 135.8, 129.5, 129.2, 129.2, 129.2, 129.1, 129.0, 128.7, 128.6, 126.9, 126.6, 112.2, 112.1, 111.8, 52.2, 51.2, 40.8, 39.7, 37.1, 36.9, 33.9, 33.8, 28.1, 27.7, 24.2, 24.2, 17.4, 16.4. **HRMS (ESI)**: calculated for $C_{22}H_{24}N_2$ [M-H]$^-$: 315.1867; found: 315.1864.

Compound 13

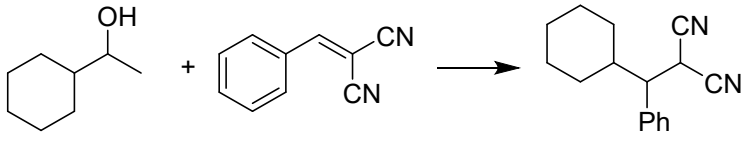


13, 66%, d.r.=1.1:1

2-(3-(benzo[d][1,3]dioxol-5-yl)-2-methyl-1-phenylpropyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 1:2 Petroleum ether/DCM) to afford the titled compound as a colorless oil (42.0 mg, 66%). The dr ratio is 1.1:1, determined by ^{1}H NMR. The isolated product gives 1.4:1 dr ratio and two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.50 – 7.39 (m, 9.65H), 7.30 – 7.27 (m, 1.04H), 7.27 – 7.25 (m, 1.81H), 6.79 – 6.75 (m, 1.04H), 6.73 – 6.67 (m, 2.38H), 6.66 – 6.60 (m, 1.09H), 6.53 – 6.44 (m, 2.72H), 5.96 (s, 2.00H), 5.92 (s, 2.72H), 4.22 (d, *J* = 7.5 Hz, 1.00H), 4.18 (d, *J* = 5.3 Hz, 1.36H), 3.15 – 3.09 (m, 1.00H), 2.99 – 2.93 (m, 1.37H), 2.78 – 2.71 (m, 1.03H), 2.62 – 2.56 (m, 1.41H), 2.54 – 2.41 (m, 2.44H), 2.33 – 2.25 (m, 1.05H), 2.08 – 2.00 (m, 1.48H), 1.06 (d, *J* = 6.6 Hz, 4.08H), 0.79 (d, *J* = 6.7 Hz, 3.00H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.0, 147.8, 146.4, 146.2, 136.6, 135.8, 132.9, 132.6, 129.6, 129.3, 129.2, 129.0, 128.6, 128.5, 122.2, 122.1, 112.2, 112.1, 112.1, 111.8, 109.5, 109.4, 108.5, 108.3, 101.1, 101.1, 52.1, 51.2, 41.0, 39.9, 37.3, 37.1, 28.1, 27.7, 17.3, 16.3. **HRMS (ESI)**: calculated for $C_{20}H_{18}N_2O_2$ [M-H]$^-$: 317.1296; found: 317.1291.

Compound 14[12]



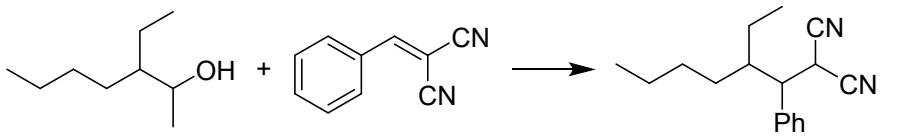
14, 83%

2-(cyclohexyl(phenyl)methyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and

purified by flash column chromatography (silica gel, 25:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (39.6 mg, 83%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.43 – 7.29 (m, 5H), 4.19 (d, *J* = 5.5 Hz, 1H), 2.91 – 2.84 (m, 1H), 2.06 – 1.81 (m, 3H), 1.72 – 1.61 (m, 2H), 1.50 – 1.32 (m, 2H), 1.23 – 1.02 (m, 3H), 0.87 – 0.77 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 136.8, 129.3, 128.9, 128.4, 112.3, 112.1, 52.5, 39.4, 31.3, 30.7, 27.3, 26.0, 26.0, 25.9.

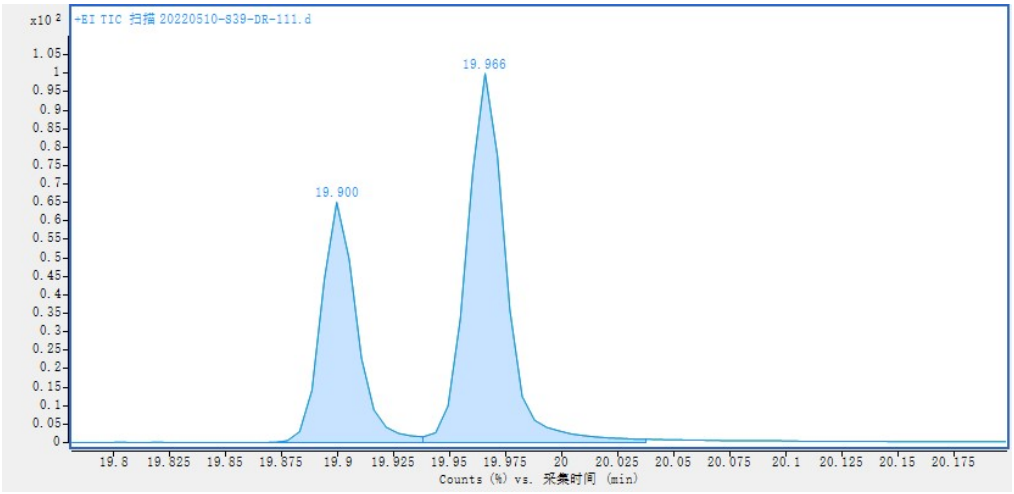
Compound 15



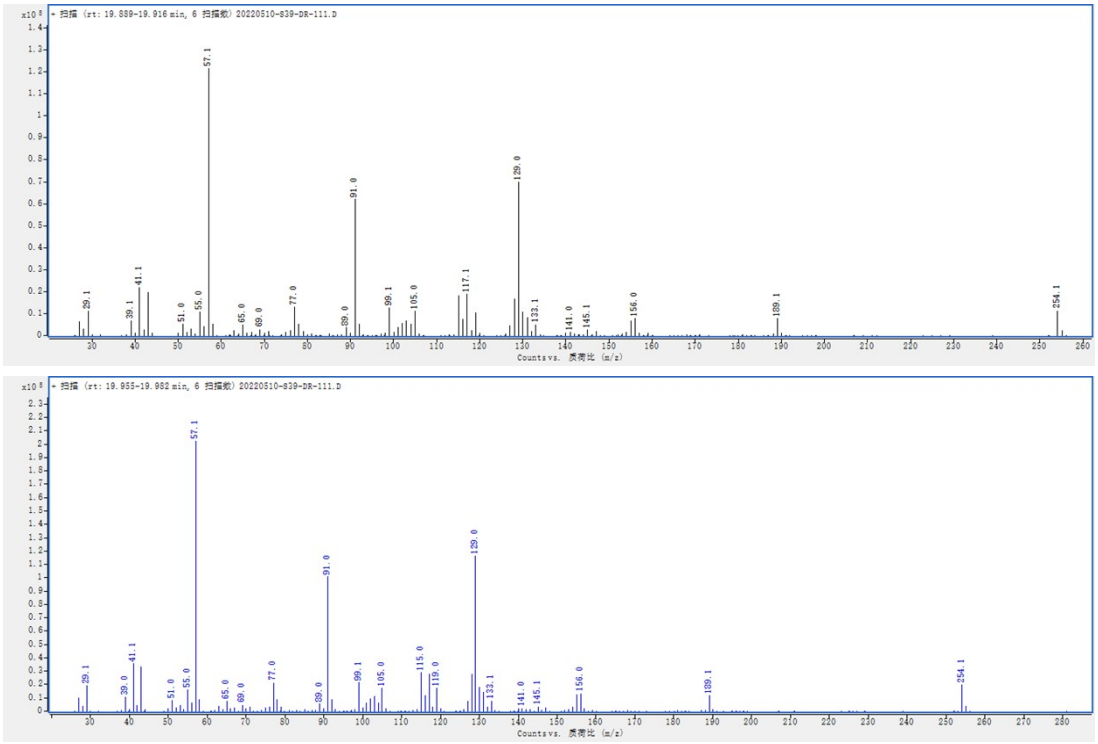
15, 81%, d.r.=1.7:1

2-(2-ethyl-1-phenylhexyl)malononitrile

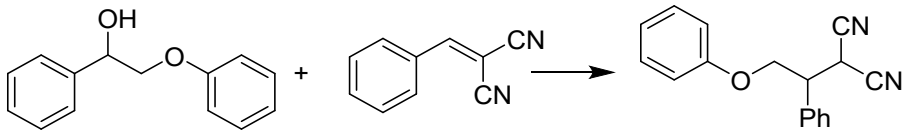
Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a colorless oil (39.6 mg, 83%). The dr ratio is 1.7:1, determined by GC-MS. The product gives two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.45 – 7.30 (m, 5H), 4.15 (t, *J* = 6.2 Hz, 1H), 3.14 – 3.04 (m, 1H), 2.18 – 2.08 (m, 1H), 1.60 – 1.31 (m, 5H), 1.24 – 1.05 (m, 3H), 1.00 – 0.92 (m, 3H), 0.81 – 0.74 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 136.7, 136.6, 129.2, 128.9, 128.5, 128.5, 112.3, 112.1, 49.2, 49.0, 40.0, 40.0, 29.0, 28.7, 28.1, 27.9, 27.8, 27.8, 23.1, 22.8, 22.2, 22.0, 14.2, 14.0, 9.9, 9.7. **HRMS (ESI)**: calculated for $C_{17}H_{22}N_2$ [M-H]$^-$: 253.1710; found: 253.1707.



Peak	Center X	Area	Area %	Area Sum %	Height	Height % (Norm)
1	19.9	1340246	59.02	37.11	1214364	65.04
2	19.966	2270904	100	62.89	1865346	99.9



Compound 16

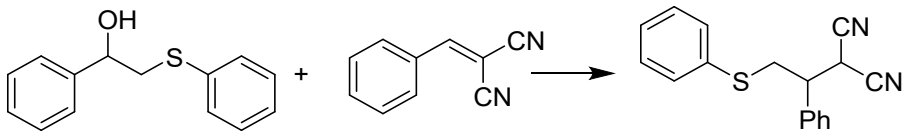


16, 68%

2-(2-phenoxy-1-phenylethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 1:1 Petroleum ether/DCM) to afford the titled compound as a pale yellow oil (35.7 mg, 68%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.48 – 7.40 (m, 5H), 7.36 – 7.29 (m, 2H), 7.03 (t, *J* = 7.4 Hz, 1H), 6.98 – 6.92 (m, 2H), 4.53 (d, *J* = 5.7 Hz, 1H), 4.44 – 4.34 (m, 2H), 3.74 – 3.66 (m, 1H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 157.6, 134.0, 129.9, 129.7, 129.6, 128.3, 122.3, 114.8, 112.0, 111.5, 67.3, 46.2, 26.6. **HRMS (ESI)**: calculated for $C_{17}H_{14}N_2O$ [M-H]$^-$: 261.1033; found: 261.1036.

Compound S17

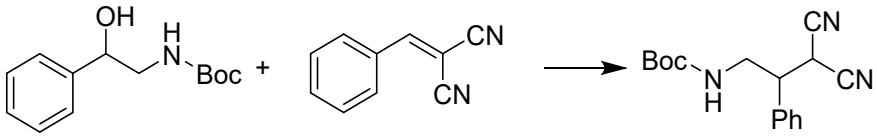


17, 93%

2-(1-phenyl-2-(phenylthio)ethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a pale yellow oil (51.8 mg, 93%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.46 – 7.26 (m, 10H), 4.57 (d, *J* = 4.8 Hz, 1H), 3.51 – 3.36 (m, 2H), 3.33 – 3.26 (m, 1H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 135.6, 133.2, 130.9, 129.7, 129.5, 129.4, 128.0, 127.9, 112.0, 111.2, 45.7, 36.6, 28.1. **HRMS (ESI)**: calculated for $C_{17}H_{14}N_2S$ [M-H]$^-$: 277.0805; found: 277.0808.

Compound 18

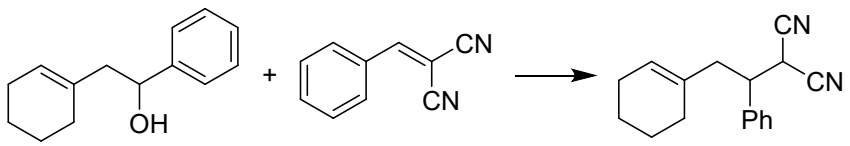


18, 81%

tert-butyl (3,3-dicyano-2-phenylpropyl)carbamate

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the solvent was removed and purified by flash column chromatography (silica gel, DCM) to afford the titled compound as a light yellow oil (46.2 mg, 81%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.46 – 7.29 (m, 5H), 4.84 (s, 1H), 4.21 (d, *J* = 4.8 Hz, 1H), 3.76 – 3.64 (m, 1H), 3.63 – 3.49 (m, 2H), 1.45 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.2, 135.0, 129.5, 129.4, 128.1, 112.0, 111.6, 80.7, 46.3, 43.0, 28.4, 27.3. **HRMS (ESI)**: calculated for $C_{16}H_{19}N_3O_2$ [M+Na]$^+$: 308.1370; found: 308.1361.

Compound 19

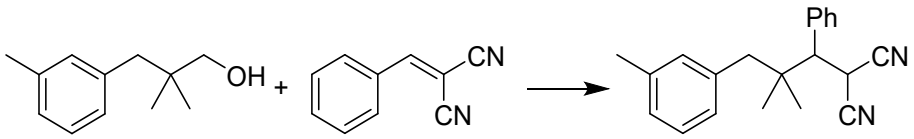


19, 75%

2-(2-(cyclohex-1-en-1-yl)-1-phenylethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a colorless oil (37.6 mg, 75%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.43 – 7.33 (m, 5H), 5.67 – 5.62 (m, 1H), 4.02 (d, *J* = 4.7 Hz, 1H), 3.39 – 3.31 (m, 1H), 2.76 – 2.66 (m, 1H), 2.54 – 2.45 (m, 1H), 2.05 – 1.81 (m, 4H), 1.67 – 1.51 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 137.1, 132.9, 129.2, 128.9, 128.1, 127.1, 112.5, 111.7, 44.2, 41.0, 28.8, 28.1, 25.3, 22.8, 22.1. **HRMS (ESI)**: calculated for $C_{17}H_{18}N_2$ [M-H]$^-$: 249.1397; found: 249.1393.

Compound 20

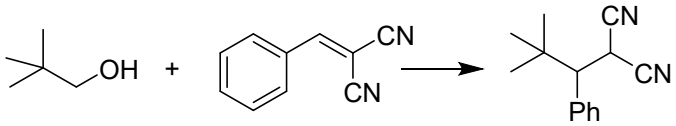


20, 72%

2-(2,2-dimethyl-1-phenyl-3-(m-tolyl)propyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the solvent was removed and purified by flash column chromatography (silica gel, 1:2 Petroleum ether/DCM) to afford the titled compound as a colorless oil (43.5 mg, 72%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.49 – 7.36 (m, 5H), 7.21 – 7.15 (m, 1H), 7.10 – 7.03 (m, 1H), 6.93 – 6.84 (m, 2H), 4.25 (d, *J* = 5.1 Hz, 1H), 3.11 (d, *J* = 5.1 Hz, 1H), 2.67 – 2.55 (m, 2H), 2.33 (s, 3H), 1.13 (s, 3H), 0.97 (s, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 137.8, 136.9, 136.0, 131.7, 130.0, 128.9, 128.9, 128.1, 127.9, 127.5, 113.4, 113.1, 56.2, 47.1, 38.6, 25.3, 25.1, 21.5. **HRMS (ESI)**: calculated for $C_{21}H_{22}N_2$ [M-H]$^-$: 301.1710; found: 301.1706.

Compound 21[13]



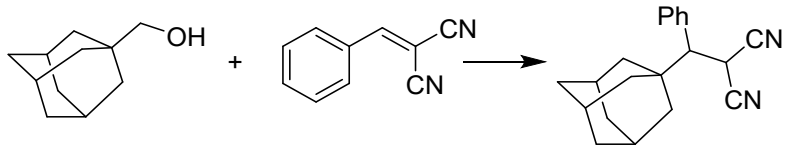
21, 45%

2-(2,2-dimethyl-1-phenylpropyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was

filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 100:1 Petroleum ether/ EtOAc) to afford the titled compound as a colorless oil (19.1 mg, 45%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.44 – 7.36 (m, 5H), 4.22 (d, *J* = 5.7 Hz, 1H), 3.01 (d, *J* = 5.7 Hz, 1H), 1.10 (s, 9H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 136.4, 129.4, 128.8, 128.7, 113.3, 113.3, 56.9, 35.0, 28.6, 25.2.

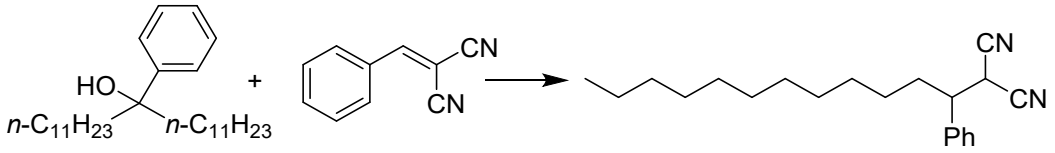
Compound 22



22, 58% **2-(((3r,5r,7r)-adamantan-1-yl)(phenyl)methyl)malononitrile**

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 100:1 Petroleum ether/ EtOAc) to afford the titled compound as a colorless oil (33.7 mg, 58%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.45 – 7.31 (m, 5H), 4.25 (d, *J* = 5.4 Hz, 1H), 2.80 (d, *J* = 5.4 Hz, 1H), 2.08 – 1.99 (m, 3H), 1.75 – 1.56 (m, 12H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 135.4, 129.8, 128.7, 128.7, 113.6, 113.4, 58.2, 40.5, 36.7, 36.5, 28.5, 23.9. **HRMS (ESI)**: calculated for $C_{20}H_{22}N_2$ [M-H]$^-$: 289.1710; found: 289.1712.

Compound 23

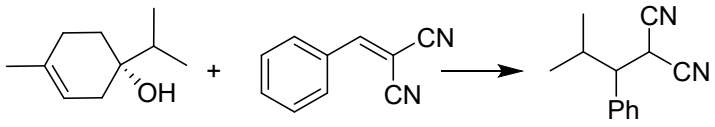


23, 75% **2-(1-phenyldodecyl)malononitrile**

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 50:1 Petroleum ether/EtOAc) to afford the

titled compound as a colorless oil (46.6 mg, 75%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.44 – 7.34 (m, 3H), 7.33 – 7.27 (m, 2H), 3.88 (d, *J* = 6.3 Hz, 1H), 3.23 – 3.15 (m, 1H), 2.03 – 1.93 (m, 2H), 1.35 – 1.16 (m, 18H), 0.87 (t, *J* = 6.9 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 137.0, 129.4, 128.9, 127.9, 112.1, 46.7, 32.2, 32.0, 30.4, 29.7, 29.6, 29.6, 29.4, 29.4, 29.2, 27.1, 22.8, 14.2. **HRMS (ESI)**: calculated for $C_{21}H_{30}N_2$ [M-H]$^-$: 309.2336; found: 309.2337.

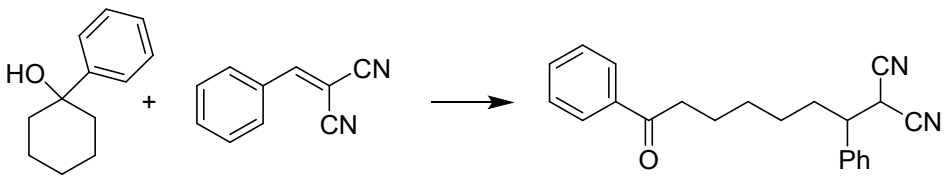
Compound 24



24, 90% **2-(2-methyl-1-phenylpropyl)malononitrile**

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 40:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (35.7 mg, 90%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.51 – 7.28 (m, 5H), 4.16 (d, *J* = 5.7 Hz, 1H), 2.89 – 2.78 (m, 1H), 2.46 – 2.32 (m, 1H), 1.14 (d, *J* = 6.6 Hz, 3H), 0.83 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 136.7, 129.3, 128.9, 128.4, 112.3, 112.0, 53.6, 30.4, 27.9, 21.1, 20.5. **HRMS (ESI)**: calculated for $C_{13}H_{14}N_2$ [M-H]$^-$: 197.1084; found: 197.1080.

Compound 25

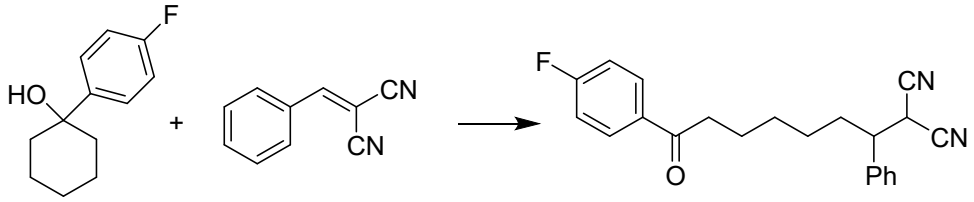


25, 94% **2-(7-oxo-1,7-diphenylheptyl)malononitrile**

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 10:1 Petroleum ether/EtOAc) to afford the

titled compound as a colorless oil (62.1 mg, 94%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.97 – 7.88 (m, 2H), 7.58 – 7.53 (m, 1H), 7.48 – 7.35 (m, 5H), 7.34 – 7.27 (m, 2H), 3.93 (d, *J* = 6.3 Hz, 1H), 3.26 – 3.16 (m, 1H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.07 – 1.95 (m, 2H), 1.76 – 1.62 (m, 2H), 1.46 – 1.34 (m, 2H), 1.33 – 1.23 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 200.2, 137.0, 136.8, 133.1, 129.4, 129.0, 128.7, 128.1, 127.9, 112.1, 46.6, 38.3, 32.0, 30.3, 28.8, 26.9, 23.9. **HRMS (ESI)**: calculated for $C_{22}H_{22}N_2O$ [M+Na]$^+$: 353.1624; found: 353.1619.

Compound 26

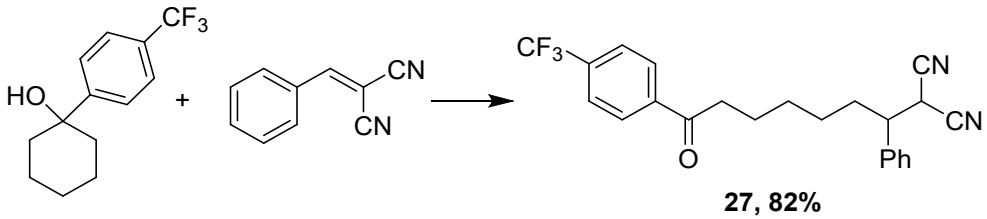


26, 87%

2-(7-(4-fluorophenyl)-7-oxo-1-phenylheptyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (60.6 mg, 87%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.98 – 7.91 (m, 2H), 7.44 – 7.34 (m, 3H), 7.33 – 7.28 (m, 2H), 7.16 – 7.08 (m, 2H), 3.89 (d, *J* = 6.3 Hz, 1H), 3.24 – 3.17 (m, 1H), 2.89 (t, *J* = 7.2 Hz, 2H), 2.06 – 1.97 (m, 2H), 1.77 – 1.63 (m, 2H), 1.45 – 1.35 (m, 2H), 1.32 – 1.24 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 198.5, 167.1, 164.6, 136.8, 133.5, 133.5, 130.8, 130.7, 129.5, 129.1, 127.9, 115.9, 115.7, 112.0, 112.0, 46.7, 38.2, 32.1, 30.4, 28.9, 27.0, 23.9. **^{19}F NMR (377 MHz, Chloroform-*d*)** δ -105.40 – -105.48 (m). **HRMS (ESI)**: calculated for $C_{22}H_{21}FN_2O$ [M+Na]$^+$: 371.1530; found: 371.1522.

Compound 27

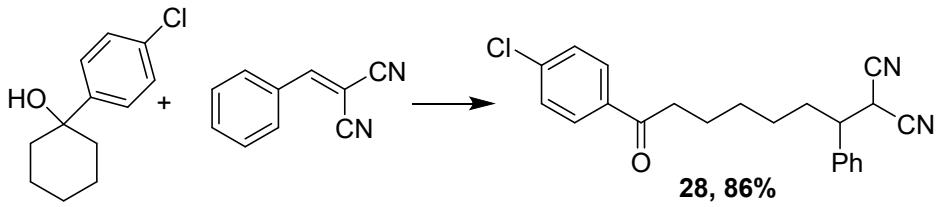


27, 82%

2-(7-oxo-1-phenyl-7-(4-(trifluoromethyl)phenyl)heptyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 10:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (65.3 mg, 82%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.02 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.45 – 7.34 (m, 3H), 7.34 – 7.27 (m, 2H), 3.90 (d, *J* = 6.3 Hz, 1H), 3.26 – 3.17 (m, 1H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.08 – 1.98 (m, 2H), 1.78 – 1.64 (m, 2H), 1.46 – 1.25 (m, 4H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 199.1, 139.7, 136.8, 134.5 (q, *J* = 32.7 Hz), 129.5, 129.1, 128.4, 127.9, 125.8 (q, *J* = 3.6 Hz), 123.7 (d, *J* = 272.7 Hz), 112.0, 112.0, 46.7, 38.6, 32.1, 30.4, 28.8, 27.0, 23.7. **^{19}F NMR (377 MHz, Chloroform-*d*)** δ -63.10. **HRMS (ESI)**: calculated for $C_{23}H_{21}F_3N_2O$ [M+Na]$^+$: 421.1498; found: 421.1490.

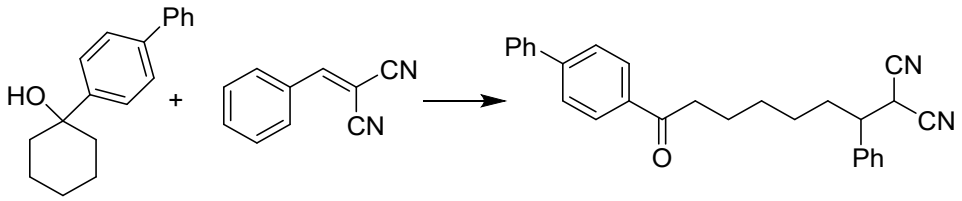
Compound 28



2-(7-(4-chlorophenyl)-7-oxo-1-phenylheptyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 10:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (62.8 mg, 86%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.89 – 7.83 (m, 2H), 7.47 – 7.35 (m, 5H), 7.33 – 7.27 (m, 2H), 3.89 (d, *J* = 6.3 Hz, 1H), 3.26 – 3.16 (m, 1H), 2.88 (t, *J* = 7.2 Hz, 2H), 2.07 – 1.97 (m, 2H), 1.75 – 1.63 (m, 2H), 1.44 – 1.34 (m, 2H), 1.30 – 1.25 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 198.9, 139.6, 136.8, 135.4, 129.5, 129.5, 129.1, 129.0, 127.9, 112.0, 112.0, 46.7, 38.3, 32.1, 30.4, 28.8, 27.0, 23.8. **HRMS (ESI)**: calculated for $C_{22}H_{21}ClN_2O$ [M+Na]$^+$: 387.1235; found: 387.1227.

Compound 29

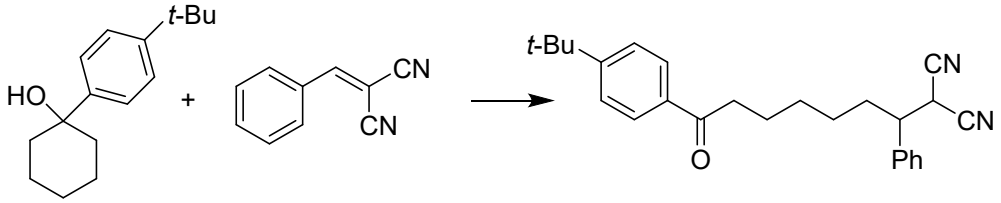


29, 92%

2-(7-([1,1'-biphenyl]-4-yl)-7-oxo-1-phenylheptyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 10:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (74.8 mg, 92%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.02 – 7.96 (m, 2H), 7.70 – 7.60 (m, 4H), 7.50 – 7.44 (m, 2H), 7.43 – 7.28 (m, 6H), 3.89 (d, *J* = 6.3 Hz, 1H), 3.26 – 3.17 (m, 1H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.07 – 1.97 (m, 2H), 1.79 – 1.65 (m, 2H), 1.47 – 1.35 (m, 2H), 1.34 – 1.25 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 199.8, 145.8, 139.9, 136.8, 135.8, 129.4, 129.1, 129.0, 128.7, 128.4, 127.9, 127.4, 112.0, 46.7, 38.4, 32.1, 30.3, 28.9, 26.9, 24.0. **HRMS (ESI**): calculated for $C_{28}H_{26}N_2O$ $[M+Na]^+$: 429.1937; found: 429.1929.

Compound 30



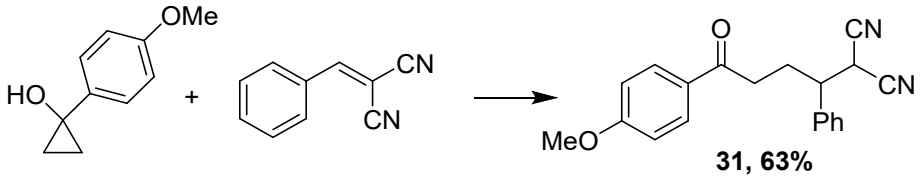
30, 89%

2-(7-(4-(tert-butyl)phenyl)-7-oxo-1-phenylheptyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a light yellow oil (68.8 mg, 89%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.90 – 7.83 (m, 2H), 7.50 – 7.44 (m, 2H), 7.43 – 7.33 (m, 3H), 7.33 – 7.27 (m, 2H), 3.90 (d, *J* = 6.3 Hz, 1H), 3.25 – 3.16 (m, 1H), 2.89 (t, *J* = 7.2 Hz, 2H), 2.07 – 1.96 (m, 2H), 1.75 – 1.59 (m, 2H), 1.44 – 1.22 (m, 13H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 199.9, 156.8, 136.8, 134.5, 129.4, 129.0,

128.1, 127.9, 125.6, 112.0, 46.6, 38.2, 35.2, 32.1, 31.2, 30.3, 28.9, 27.0, 24.0. **HRMS (ESI)**: calculated for $C_{26}H_{30}N_2O$ $[M+Na]^+$: 409.2250; found: 409.2240.

Compound 31

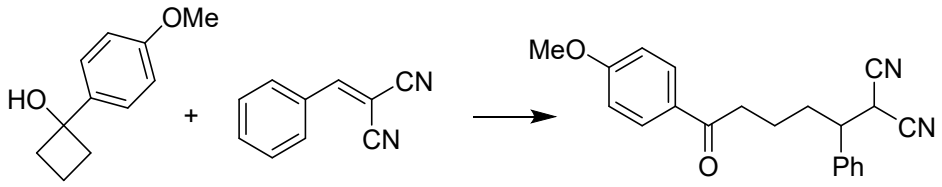


31, 63%

2-(4-(4-methoxyphenyl)-4-oxo-1-phenylbutyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a grey solid (40.1 mg, 63%). **^{1}H NMR (400 MHz, DMSO-d_6)** δ 7.81 (d, J = 8.8 Hz, 2H), 7.47 – 7.33 (m, 5H), 6.99 (d, J = 8.8 Hz, 2H), 5.44 – 5.18 (m, 1H), 3.81 (s, 3H), 3.57 – 3.46 (m, 1H), 2.99 – 2.88 (m, 1H), 2.82 – 2.71 (m, 1H), 2.32 – 2.22 (m, 1H), 2.21 – 2.10 (m, 1H). **^{13}C NMR (101 MHz, DMSO-*d*6)** δ 197.5, 163.6, 138.0, 130.5, 129.7, 129.4, 128.8, 128.5, 114.3, 114.1, 113.7, 56.0, 44.6, 35.3, 29.6, 27.0. **^{1}H NMR (600 MHz, Acetone-d_6)** δ 7.87 (d, J = 8.8 Hz, 2H), 7.55 – 7.41 (m, 4H), 7.41 – 7.34 (m, 1H), 6.98 (d, J = 8.8 Hz, 2H), 4.93 (d, J = 7.0 Hz, 0.5H), 3.86 (s, 3H), 3.65 – 3.59 (m, 1H), 3.05 – 2.99 (m, 1H), 2.94 – 2.88 (m, 1H), 2.51 – 2.45 (m, 1H), 2.39 – 2.32 (m, 1H). **^{13}C NMR (151 MHz, Acetone-*d*6)** δ 197.3, 164.5, 138.4, 130.9, 130.7, 129.9, 129.4, 129.2, 114.5, 114.0, 113.8, 55.9, 46.0, 45.9, 35.8, 30.5, 27.6, 27.5. **HRMS (ESI)**: calculated for $C_{20}H_{18}N_2O_2$ $[M-H]^-$: 317.1296; found: 317.1296.

Compound 32



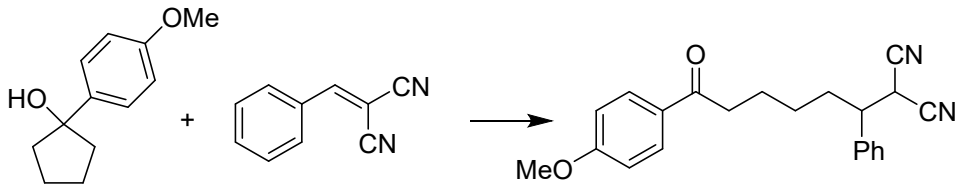
32, 96%

2-(5-(4-methoxyphenyl)-5-oxo-1-phenylpentyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was

filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 10:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (63.8 mg, 96%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.91 – 7.81 (m, 2H), 7.43 – 7.30 (m, 5H), 6.95 – 6.86 (m, 2H), 4.00 (d, *J* = 6.2 Hz, 1H), 3.84 (s, 3H), 3.29 – 3.21 (m, 1H), 3.00 – 2.84 (m, 2H), 2.12 – 2.01 (m, 2H), 1.70 – 1.59 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 197.8, 163.6, 136.5, 130.2, 129.7, 129.3, 129.0, 128.0, 113.8, 112.1, 112.0, 55.5, 46.5, 37.3, 31.6, 30.2, 21.6. **HRMS (ESI)**: calculated for $C_{21}H_{20}N_2O_2$ $[M+Na]^+$: 355.1417; found: 355.1410.

Compound 33

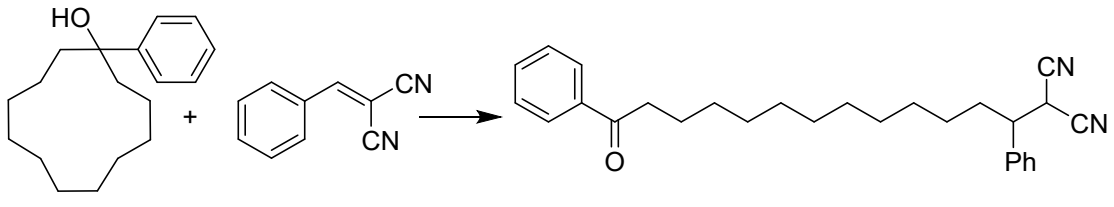


33, 89%

2-(6-(4-methoxyphenyl)-6-oxo-1-phenylhexyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 6:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (61.7 mg, 89%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.94 – 7.84 (m, 2H), 7.44 – 7.26 (m, 5H), 6.98 – 6.84 (m, 2H), 3.98 (d, *J* = 6.3 Hz, 1H), 3.84 (s, 3H), 3.28 – 3.18 (m, 1H), 2.86 (t, *J* = 7.2 Hz, 2H), 2.10 – 1.97 (m, 2H), 1.83 – 1.65 (m, 2H), 1.38 – 1.22 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 198.40, 163.49, 136.70, 130.28, 129.92, 129.32, 128.91, 127.90, 113.78, 112.09, 112.07, 55.51, 46.27, 37.67, 32.04, 30.15, 26.66, 23.74. **HRMS (ESI)**: calculated for $C_{22}H_{22}N_2O_2$ $[M+Na]^+$: 369.1574; found: 369.1569.

Compound 34

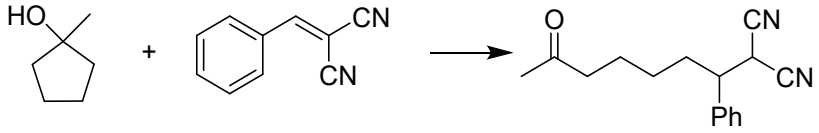


34, 90%

2-(13-oxo-1,13-diphenyltridecyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 3:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (74.6 mg, 90%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.93 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.36 (m, 5H), 7.34 – 7.29 (m, 2H), 3.90 (d, *J* = 6.3 Hz, 1H), 3.23 – 3.16 (m, 1H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.03 – 1.94 (m, 2H), 1.77 – 1.68 (m, 2H), 1.42 – 1.15 (m, 16H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 200.6, 137.1, 136.9, 132.9, 129.3, 128.8, 128.6, 128.1, 127.8, 112.0, 46.6, 38.6, 32.1, 30.3, 29.4, 29.4, 29.3, 29.2, 29.1, 27.0, 24.4. **HRMS (ESI)**: calculated for $C_{28}H_{34}N_2O$ [M+Na]$^+$: 437.2563; found: 437.2556.

Compound 35



35, 93%

2-(6-oxo-1-phenylheptyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (47.3 mg, 93%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.44 – 7.34 (m, 3H), 7.34 – 7.28 (m, 2H), 3.97 (d, *J* = 6.3 Hz, 1H), 3.26 – 3.15 (m, 1H), 2.38 (t, *J* = 7.5 Hz, 2H), 2.08 (s, 3H), 2.03 – 1.95 (m, 2H), 1.66 – 1.50 (m, 2H), 1.28 – 1.16 (m, 2H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 208.6, 136.6, 129.3, 128.9, 127.9, 112.1, 46.2, 43.1, 31.9, 30.2, 29.9, 26.4, 23.0. **HRMS (ESI)**: calculated for $C_{16}H_{18}N_2O$ [M+Na]$^+$: 277.1311; found: 277.1308.

Compound 36

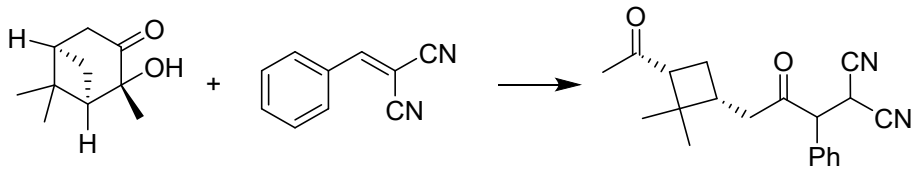
36, 90%

2-(7-oxo-1-

phenyloctyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (48.3 mg, 90%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.43 – 7.34 (m, 3H), 7.33 – 7.28 (m, 2H), 3.94 (d, *J* = 6.4 Hz, 1H), 3.23 – 3.16 (m, 1H), 2.37 (t, *J* = 7.3 Hz, 2H), 2.10 (s, 3H), 2.03 – 1.94 (m, 2H), 1.56 – 1.45 (m, 2H), 1.35 – 1.18 (m, 4H). **^{13}C NMR (101 MHz, Chloroform-*d*)** δ 208.9, 136.7, 129.3, 128.9, 127.8, 112.0, 46.4, 43.3, 31.9, 30.2, 29.9, 28.6, 26.8, 23.3. **HRMS (ESI)**: calculated for $C_{17}H_{20}N_2O$ [M+Na]$^+$: 291.1468; found: 291.1464.

Compound 37

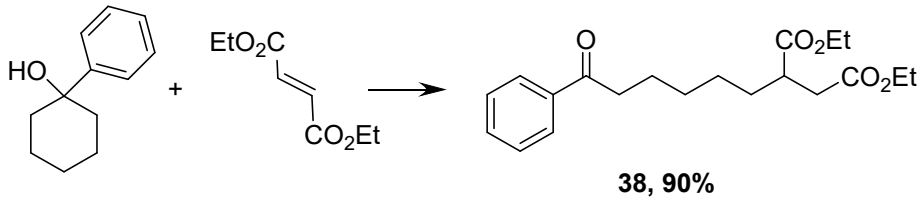


37, 86%

2-(3-(3-acetyl-2,2-dimethylcyclobutyl)-2-oxo-1-phenylpropyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, The solvent was removed and purified by flash column chromatography (silica gel, DCM) to afford the titled compound as a colorless oil (55.4 mg, 86%). The dr ratio is 1:1, determined by ^{1}H NMR. The product gives two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.51 – 7.42 (m, 6H), 7.27 – 7.21 (m, 4H), 4.41 – 4.34 (m, 2H), 4.28 – 4.21 (m, 2H), 2.91 – 2.83 (m, 2H), 2.49 – 2.33 (m, 6H), 2.05 – 1.97 (m, 6H), 1.94 – 1.87 (m, 2H), 1.82 – 1.68 (m, 2H), 1.34 (s, 3H), 1.28 (s, 3H), 0.75 (s, 3H), 0.56 (s, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 207.5, 207.4, 203.4, 203.2, 131.1, 130.9, 130.3, 130.3, 130.2, 130.2, 128.7, 128.6, 112.1, 112.0, 111.4, 58.7, 58.6, 54.2, 54.0, 43.5, 43.2, 42.2, 42.1, 36.9, 36.8, 30.4, 30.3, 25.6, 25.5, 22.8, 22.6, 17.8, 17.2. **HRMS (ESI)**: calculated for $C_{20}H_{22}N_2O_2$ [M+Na]$^+$: 345.1574; found: 345.1565.

Compound 38

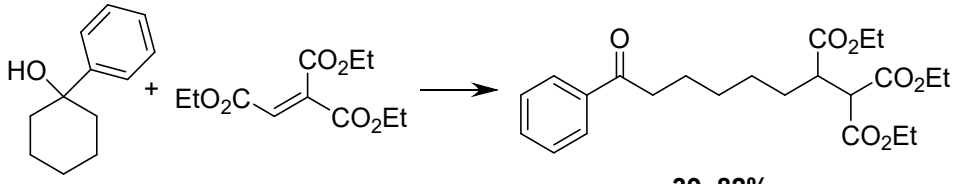


38, 90%

diethyl 2-(6-oxo-6-phenylhexyl)succinate

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 1:2 Petroleum ether/DCM) to afford the titled compound as a colorless oil (62.7 mg, 90%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.98 – 7.92 (m, 2H), 7.58 – 7.53 (m, 1H), 7.49 – 7.43 (m, 2H), 4.18 – 4.10 (m, 4H), 2.96 (t, *J* = 7.3 Hz, 2H), 2.86 – 2.77 (m, 1H), 2.74 – 2.65 (m, 1H), 2.45 – 2.38 (m, 1H), 1.76 – 1.71 (m, 2H), 1.69 – 1.61 (m, 1H), 1.58 – 1.48 (m, 1H), 1.43 – 1.33 (m, 4H), 1.28 – 1.22 (m, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.4, 175.1, 172.1, 137.1, 133.1, 128.7, 128.1, 60.7, 60.7, 41.4, 38.5, 36.3, 31.9, 29.2, 26.9, 24.2, 14.3, 14.3. **HRMS (APCI)**: calculated for $C_{20}H_{28}O_5$ $[M+H]^+$: 349.2009; found: 349.1997.

Compound 39



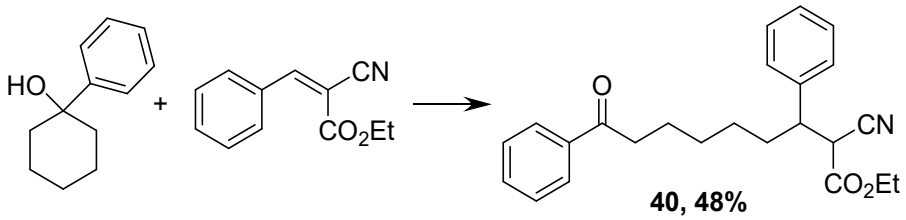
39, 82%

triethyl 8-oxo-8-phenyloctane-1,1,2-tricarboxylate

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a colorless oil (68.9 mg, 82%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.98 – 7.92 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.42 (m, 2H), 4.24 – 4.13 (m, 6H), 3.76 – 3.70 (m, 1H), 3.15 – 3.07 (m, 1H), 2.95 (t, *J* = 7.3 Hz, 2H), 1.76 – 1.68 (m, 2H), 1.64 – 1.53 (m, 2H), 1.47 – 1.31 (m, 4H), 1.29 – 1.22 (m, 9H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.3, 173.5, 168.0, 168.0, 137.0, 132.9, 128.6, 128.0, 61.7, 61.7, 60.8, 53.9, 44.3, 38.4, 29.9, 29.0, 26.3, 24.0, 14.2, 14.1, 14.0. **HRMS (APCI)**:

calculated for $C_{23}H_{32}O_7$ [M+H]$^+$: 421.2221; found: 421.2207.

Compound 40

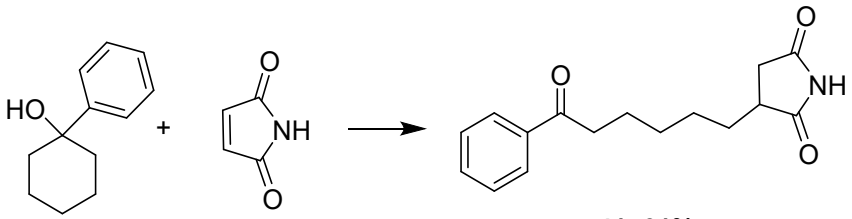


40, 48%

ethyl 2-cyano-9-oxo-3,9-diphenylnonanoate

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 10:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (36.2 mg, 48%). The dr ratio is 1:1, determined by ^{1}H NMR. The product gives two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.95 – 7.88 (m, 4H), 7.57 – 7.51 (m, 2H), 7.48 – 7.41 (m, 4H), 7.35 – 7.23 (m, 10H), 4.16 – 4.04 (m, 4H), 3.78 (d, *J* = 6.3 Hz, 1H), 3.63 (d, *J* = 6.9 Hz, 1H), 3.35 – 3.27 (m, 2H), 2.94 – 2.86 (m, 4H), 2.00 – 1.79 (m, 4H), 1.70 – 1.60 (m, 4H), 1.44 – 1.19 (m, 8H), 1.17 – 1.08 (m, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.3, 200.3, 165.5, 165.4, 139.2, 138.5, 137.1, 133.0, 133.0, 129.0, 128.8, 128.7, 128.6, 128.2, 128.1, 128.1, 127.9, 127.9, 116.0, 115.6, 62.8, 62.7, 46.0, 45.8, 45.2, 44.6, 38.4, 38.4, 33.3, 32.1, 29.0, 29.0, 27.1, 27.0, 24.0, 24.0, 13.9, 13.9. **HRMS (APCI)**: calculated for $C_{24}H_{27}NO_3$ [M+H]$^+$: 378.2064; found: 378.2055.

Compound 41



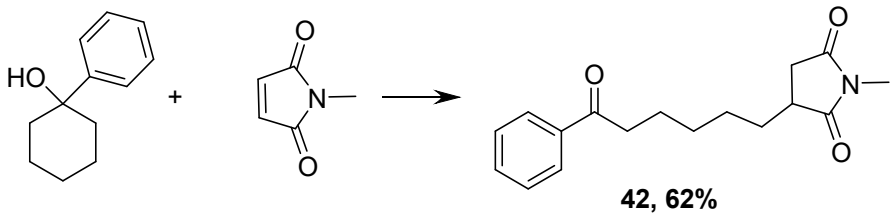
41, 61%

3-(6-oxo-6-phenylhexyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by

flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (33.3 mg, 61%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.77 (s, 1H), 7.98 – 7.92 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 2.98 (t, *J* = 7.2 Hz, 2H), 2.92 – 2.81 (m, 2H), 2.48 – 2.37 (m, 1H), 1.95 – 1.88 (m, 1H), 1.82 – 1.71 (m, 2H), 1.63 – 1.52 (m, 1H), 1.48 – 1.39 (m, 4H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.4, 180.8, 177.4, 137.0, 133.1, 128.7, 128.1, 41.3, 38.4, 35.6, 31.0, 29.0, 26.7, 24.0. **HRMS (ESI)**: calculated for $C_{16}H_{19}NO_3$ $[M-H]^-$: 272.1292; found: 272.1292.

Compound 42

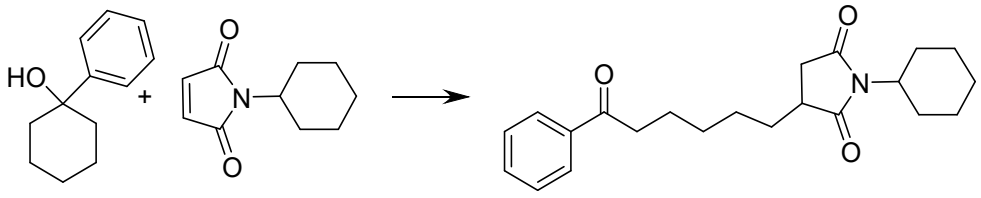


42, 62%

1-methyl-3-(6-oxo-6-phenylhexyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 3:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (35.6 mg, 62%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.91 (m, 2H), 7.59 – 7.52 (m, 1H), 7.50 – 7.42 (m, 2H), 3.02 – 2.93 (m, 5H), 2.88 – 2.75 (m, 2H), 2.42 – 2.33 (m, 1H), 1.98 – 1.88 (m, 1H), 1.80 – 1.72 (m, 2H), 1.58 – 1.38 (m, 5H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.3, 180.2, 176.9, 137.0, 133.1, 128.7, 128.1, 40.0, 38.4, 34.5, 31.3, 29.1, 26.8, 24.9, 24.0. **HRMS (APCI)**: calculated for $C_{17}H_{21}NO_3$ $[M+H]^+$: 288.1594; found: 288.1587.

Compound 43



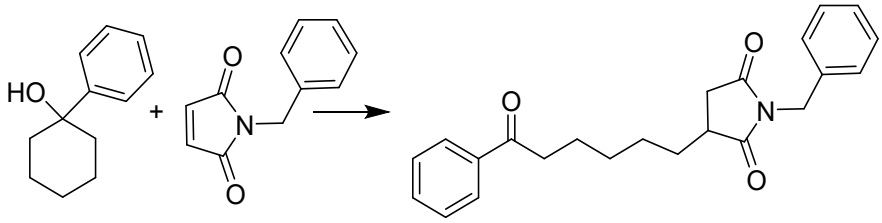
43, 71%

1-cyclohexyl-3-(6-oxo-6-phenylhexyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered

through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 7:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (50.5 mg, 71%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.91 (m, 2H), 7.59 – 7.53 (m, 1H), 7.51 – 7.43 (m, 2H), 4.01 – 3.89 (m, 1H), 2.97 (t, *J* = 7.2 Hz, 2H), 2.82 – 2.66 (m, 2H), 2.39 – 2.25 (m, 1H), 2.20 – 2.07 (m, 2H), 1.93 – 1.72 (m, 5H), 1.69 – 1.63 (m, 1H), 1.59 – 1.53 (m, 2H), 1.46 – 1.14 (m, 8H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.3, 180.2, 176.9, 137.1, 133.1, 128.7, 128.1, 51.7, 39.5, 38.4, 34.4, 31.4, 29.1, 28.9, 26.6, 26.0, 25.1, 24.1. **HRMS (APCI)**: calculated for $C_{22}H_{29}NO_3$ $[M+H]^+$: 356.2220; found: 356.2212.

Compound 44

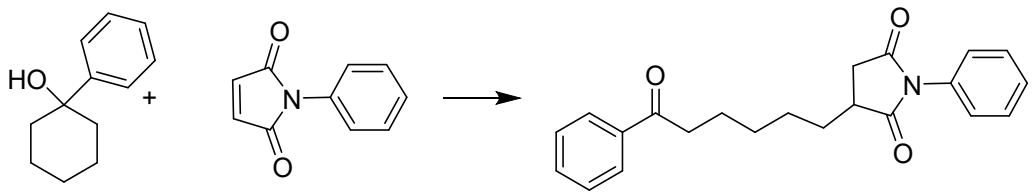


44, 65%

1-benzyl-3-(6-oxo-6-phenylhexyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (47.2 mg, 65%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.97 – 7.91 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 7.38 – 7.33 (m, 2H), 7.31 – 7.23 (m, 3H), 4.63 (s, 2H), 2.94 (t, *J* = 7.3 Hz, 2H), 2.87 – 2.75 (m, 2H), 2.40 – 2.33 (m, 1H), 1.94 – 1.84 (m, 1H), 1.78 – 1.68 (m, 2H), 1.57 – 1.47 (m, 1H), 1.45 – 1.32 (m, 4H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.2, 179.7, 176.4, 137.0, 135.9, 133.1, 128.8, 128.7, 128.7, 128.1, 128.0, 42.4, 39.9, 38.4, 34.4, 31.2, 29.0, 26.6, 24.0. **HRMS (ESI)**: calculated for $C_{23}H_{25}NO_3$ $[M+Na]^+$: 386.1727; found: 386.1716.

Compound 45

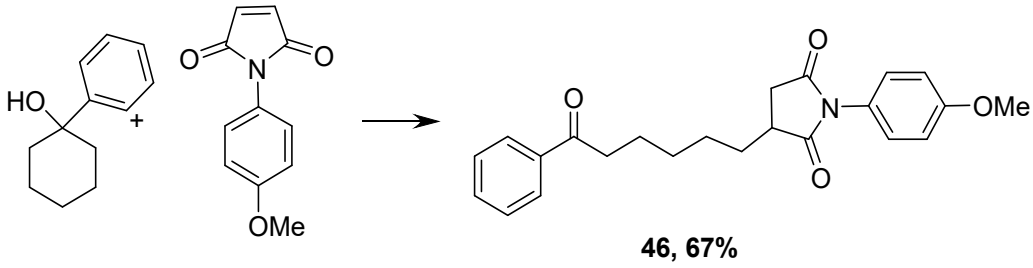


45, 80%

3-(6-oxo-6-phenylhexyl)-1-phenylpyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with dichloromethane. The solvent was removed and purified by flash column chromatography (silica gel, 3:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (55.9 mg, 80%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.98 – 7.92 (m, 2H), 7.59 – 7.52 (m, 1H), 7.50 – 7.42 (m, 4H), 7.41 – 7.35 (m, 1H), 7.30 – 7.24 (m, 2H), 3.05 – 2.92 (m, 4H), 2.59 – 2.51 (m, 1H), 2.07 – 1.96 (m, 1H), 1.82 – 1.74 (m, 2H), 1.71 – 1.62 (m, 1H), 1.55 – 1.39 (m, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.2, 179.0, 175.7, 137.1, 133.1, 132.0, 129.2, 128.7, 128.6, 128.1, 126.5, 40.0, 38.4, 34.7, 31.4, 29.1, 26.7, 24.0. **HRMS (APCI)**: calculated for $C_{22}H_{23}NO_3$ $[M+H]^+$: 350.1751; found: 350.1738.

Compound 46



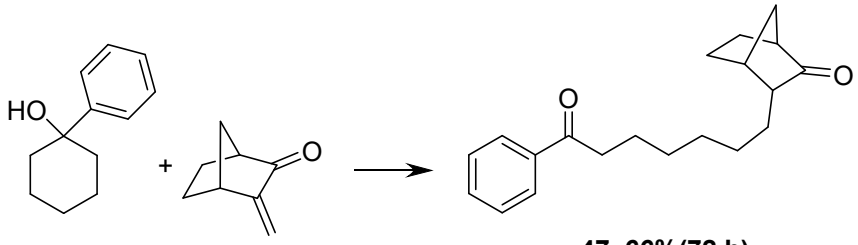
46, 67%

1-(4-methoxyphenyl)-3-(6-oxo-6-phenylhexyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 1:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (50.8 mg, 67%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.91 (m, 2H), 7.60 – 7.53 (m, 1H), 7.51 – 7.41 (m, 2H), 7.18 (d, *J* = 8.9 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 3.82 (s, 3H), 3.05 – 2.92 (m, 4H), 2.60 – 2.49 (m, 1H), 2.08 – 1.96 (m, 1H), 1.84 – 1.74 (m, 2H),

1.70 – 1.63 (m, 1H), 1.55 – 1.41 (m, 4H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.2, 179.3, 176.0, 159.6, 137.1, 133.1, 128.7, 128.1, 127.8, 124.6, 114.6, 55.6, 40.0, 38.4, 34.6, 31.5, 29.1, 26.7, 24.1. **HRMS (APCI)**: calculated for $C_{23}H_{25}NO_4$ $[M+H]^+$: 380.1856; found: 380.1848.

Compound 47

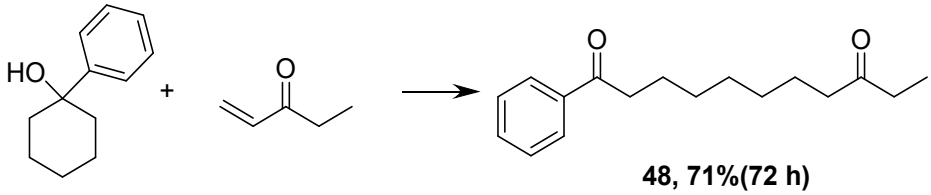


47, 66%(72 h)

(1S,3R,4R)-3-(7-oxo-7-phenylheptyl)bicyclo[2.2.1]heptan-2-one

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 50:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (39.4 mg, 66%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.96 (d, *J* = 7.8 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 2.65 – 2.56 (m, 2H), 2.02 – 1.95 (m, 1H), 1.84 – 1.63 (m, 6H), 1.60 – 1.57 (m, 2H), 1.45 – 1.31 (m, 7H), 1.24 – 1.13 (m, 1H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 220.4, 200.6, 137.2, 133.0, 128.7, 128.2, 53.7, 50.7, 38.7, 38.3, 37.2, 29.5, 29.3, 28.0, 26.4, 25.5, 24.4, 21.3. **HRMS (APCI)**: calculated for $C_{20}H_{26}O_2$ $[M+H]^+$: 299.2006; found: 299.1998.

Compound 48



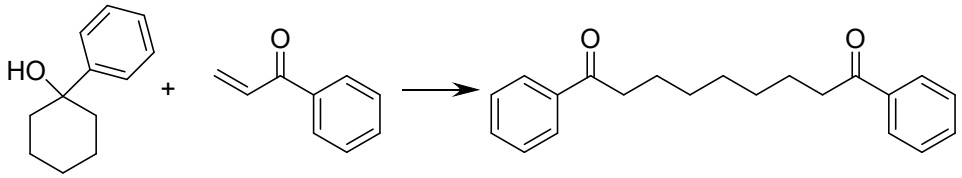
48, 71%(72 h)

1-phenylundecane-1,9-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 60:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (37.0 mg, 71%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.93

(m, 2H), 7.59 – 7.53 (m, 1H), 7.50 – 7.44 (m, 2H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.46 – 2.36 (m, 4H), 1.78 – 1.68 (m, 2H), 1.58 – 1.52 (m, 2H), 1.41 – 1.28 (m, 6H), 1.05 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 212.1, 200.7, 137.2, 133.0, 128.7, 128.2, 42.5, 38.7, 36.0, 29.4, 29.3, 29.2, 24.4, 24.0, 8.0. **HRMS (APCI)**: calculated for $C_{17}H_{24}O_2$ $[M+H]^+$: 261.1849; found: 261.1843.

Compound 49[14]

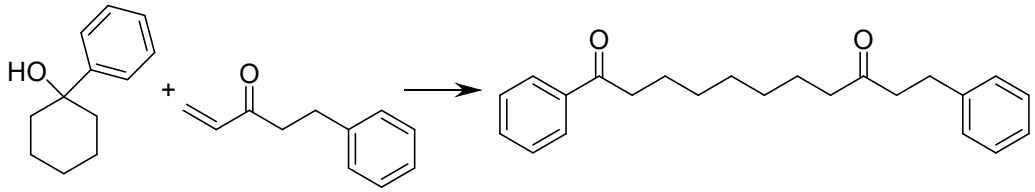


49, 86%(72 h)

1,9-diphenylnonane-1,9-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 60:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (53.0 mg, 86%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.92 (m, 4H), 7.59 – 7.53 (m, 2H), 7.49 – 7.42 (m, 4H), 2.97 (t, *J* = 7.4 Hz, 4H), 1.78 – 1.70 (m, 4H), 1.44 – 1.37 (m, 6H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.7, 137.2, 133.0, 128.7, 128.2, 38.7, 29.5, 29.4, 24.4.

Compound 50



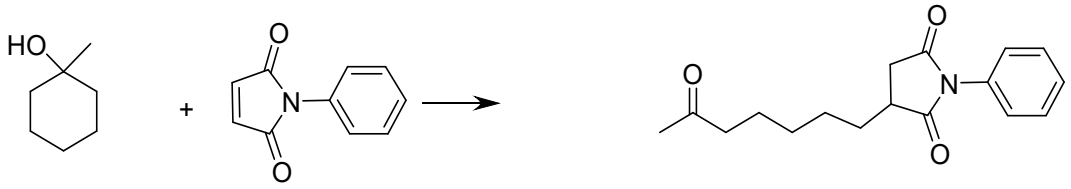
50, 75%(72 h)

1,11-diphenylundecane-1,9-dione

Prepared following the general procedure with 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 70:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (50.5 mg, 75%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.95 (d, *J* =

7.9 Hz, 2H), 7.59 – 7.52 (m, 1H), 7.50 – 7.42 (m, 2H), 7.31 – 7.24 (m, 2H), 7.23 – 7.13 (m, 3H), 2.95 (t, *J* = 7.4 Hz, 2H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 2.37 (t, *J* = 7.4 Hz, 2H), 1.77 – 1.67 (m, 2H), 1.60 – 1.51 (m, 2H), 1.40 – 1.24 (m, 6H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 210.4, 200.6, 141.3, 137.2, 133.0, 128.7, 128.6, 128.4, 128.2, 126.2, 44.4, 43.1, 38.7, 29.9, 29.4, 29.3, 29.2, 24.4, 23.8. **HRMS (APCI)**: calculated for $C_{20}H_{26}O_2$ $[M+H]^+$: 337.2162; found: 337.2153.

Compound 51

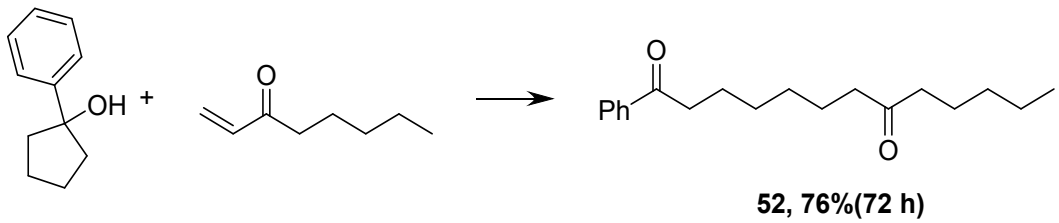


51, 72%

3-(6-oxoheptyl)-1-phenylpyrrolidine-2,5-dione

Prepared following the general procedure with 1-methylcyclohexan-1-ol (45.7 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (41.4 mg, 72%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.48 (t, *J* = 7.6 Hz, 2H), 7.42 – 7.37 (m, 1H), 7.30 – 7.25 (m, 2H), 3.06 – 2.92 (m, 2H), 2.61 – 2.52 (m, 1H), 2.45 (t, *J* = 7.3 Hz, 2H), 2.14 (s, 3H), 2.05 – 1.95 (m, 1H), 1.69 – 1.58 (m, 3H), 1.50 – 1.41 (m, 2H), 1.40 – 1.33 (m, 2H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 209.0, 179.0, 175.7, 132.0, 129.3, 128.7, 126.5, 43.6, 40.1, 34.7, 31.5, 30.1, 29.0, 26.7, 23.5. **HRMS (APCI)**: calculated for $C_{17}H_{21}NO_3$ $[M+H]^+$: 288.1594; found: 288.1588.

Compound 52



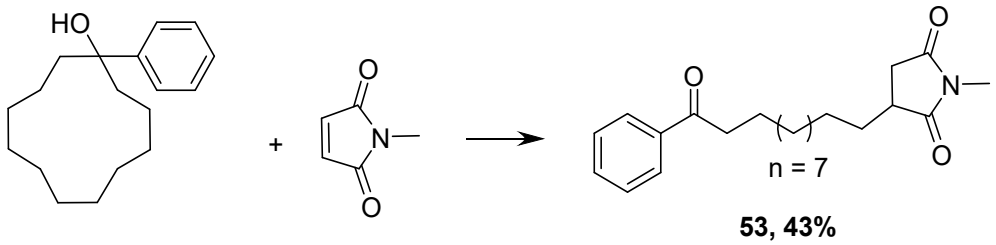
52, 76%(72 h)

1-phenyltridecane-1,8-dione

Prepared following the general procedure with 1-phenylcyclopentan-1-ol (64.9 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 72 h irradiation, the reaction mixture was filtered

through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 60:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (43.8 mg, 76%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.01 – 7.92 (m, 2H), 7.59 – 7.52 (m, 1H), 7.50 – 7.43 (m, 2H), 2.96 (t, *J* = 7.4 Hz, 2H), 2.43 – 2.34 (m, 4H), 1.79 – 1.69 (m, 2H), 1.62 – 1.54 (m, 4H), 1.42 – 1.24 (m, 8H), 0.89 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 211.7, 200.6, 137.2, 133.0, 128.7, 128.2, 43.0, 42.8, 38.6, 31.6, 29.3, 29.2, 24.3, 23.8, 23.7, 22.6, 14.1. **HRMS (APCI)**: calculated for $C_{19}H_{28}O_2$ $[M+H]^+$: 289.2162; found: 289.2155.

Compound 53

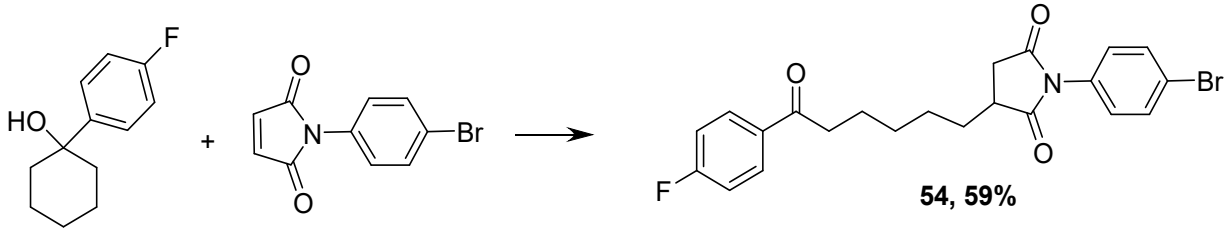


53, 43%

1-methyl-3-(12-oxo-12-phenyldodecyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-phenylcyclododecan-1-ol (104 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 3:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (32.0 mg, 43%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.00 – 7.93 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.01 – 2.93 (m, 5H), 2.87 – 2.74 (m, 2H), 2.44 – 2.33 (m, 1H), 1.96 – 1.86 (m, 1H), 1.78 – 1.69 (m, 2H), 1.54 – 1.44 (m, 1H), 1.41 – 1.25 (m, 16H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.7, 180.3, 177.0, 137.2, 133.0, 128.7, 128.2, 40.1, 38.8, 34.5, 31.5, 29.6, 29.6, 29.6, 29.6, 29.5, 29.5, 29.4, 26.9, 24.9, 24.5. **HRMS (APCI)**: calculated for $C_{23}H_{33}NO_3$ $[M+H]^+$: 372.2533; found: 372.2526.

Compound 54

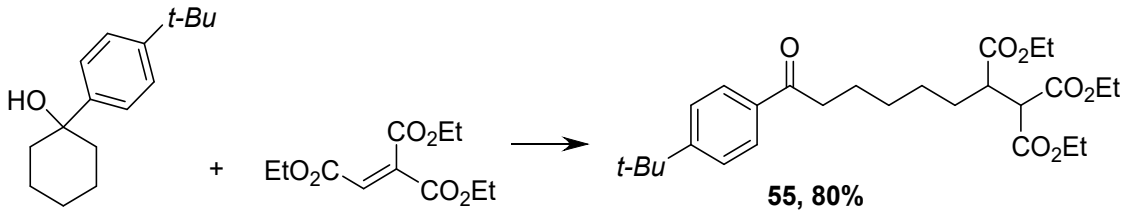


54, 59%

1-(4-bromophenyl)-3-(6-(4-fluorophenyl)-6-oxohexyl)pyrrolidine-2,5-dione

Prepared following the general procedure with 1-(4-fluorophenyl)cyclohexan-1-ol (77.7 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 3:1 Petroleum ether/EtOAc) to afford the titled compound as a white solid (52.7 mg, 59%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.03 – 7.95 (m, 2H), 7.64 – 7.57 (m, 2H), 7.22 – 7.09 (m, 4H), 3.07 – 2.92 (m, 4H), 2.62 – 2.51 (m, 1H), 2.08 – 1.96 (m, 1H), 1.83 – 1.73 (m, 2H), 1.72 – 1.62 (m, 1H), 1.55 – 1.41 (m, 4H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 178.6, 175.3, 165.8 (d, *J* = 254.7 Hz), 133.5 (d, *J* = 3.1 Hz), 132.5, 131.0, 130.8 (d, *J* = 9.3 Hz), 128.0, 122.5, 115.8 (d, *J* = 21.8 Hz), 40.1, 38.3, 34.7, 31.5, 29.1, 26.8, 24.0. **^{19}F NMR (377 MHz, Chloroform-*d*)** δ -105.41. **HRMS (APCI)**: calculated for $C_{22}H_{21}BrFNO_3$ $[M+H]^+$: 446.0762; found: 446.0752.

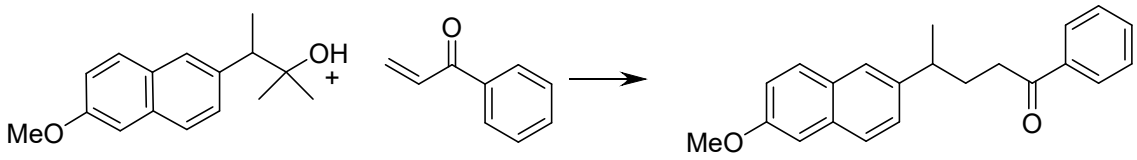
Compound 55



triethyl 8-(4-(tert-butyl)phenyl)-8-oxooctane-1,1,2-tricarboxylate

Prepared following the general procedure with 1-(4-(tert-butyl)phenyl)cyclohexan-1-ol (92.9 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a white solid (76.3 mg, 80%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.89 (d, *J* = 8.5 Hz, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 4.25 – 4.13 (m, 6H), 3.73 (d, *J* = 10.5 Hz, 1H), 3.15 – 3.07 (m, 1H), 2.93 (t, *J* = 7.3 Hz, 2H), 1.77 – 1.67 (m, 2H), 1.64 – 1.53 (m, 2H), 1.34 (s, 13H), 1.30 – 1.22 (m, 9H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.0, 173.6, 168.1, 168.1, 156.7, 134.5, 128.1, 125.6, 61.7, 60.9, 54.0, 44.4, 38.3, 35.1, 31.2, 30.0, 29.1, 26.4, 24.2, 14.2, 14.1, 14.1. **HRMS (APCI)**: calculated for $C_{27}H_{40}O_7$ $[M+H]^+$: 477.2847; found: 477.2832.

Compound 56[15]

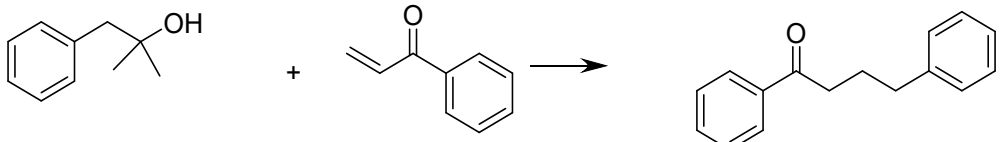


56, 67%(72 h)

4-(6-methoxynaphthalen-2-yl)-1-phenylpentan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 25:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (42.7 mg, 67%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.81 (d, *J* = 7.5 Hz, 2H), 7.72 – 7.64 (m, 2H), 7.54 (s, 1H), 7.48 (t, *J* = 7.4 Hz, 1H), 7.39 – 7.30 (m, 3H), 7.12 (d, *J* = 8.5 Hz, 2H), 3.90 (s, 3H), 3.00 – 2.77 (m, 3H), 2.21 – 1.99 (m, 2H), 1.37 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.5, 157.4, 141.7, 137.0, 133.4, 133.0, 129.2, 128.6, 128.1, 127.2, 126.2, 125.4, 118.8, 105.7, 55.4, 39.6, 36.9, 32.5, 22.9.

Compound 57[16]

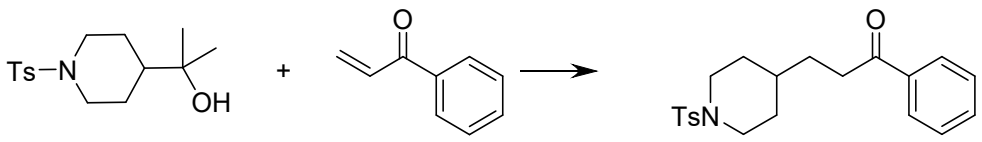


57, 46%(72 h)

1,4-diphenylbutan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 25:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (20.6 mg, 46%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.92 (d, *J* = 7.7 Hz, 2H), 7.55 (t, *J* = 7.1 Hz, 1H), 7.44 (t, *J* = 7.3 Hz, 2H), 7.32 – 7.25 (m, 2H), 7.24 – 7.17 (m, 3H), 2.98 (t, *J* = 7.2 Hz, 2H), 2.72 (t, *J* = 7.4 Hz, 2H), 2.14 – 2.02 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.3, 141.8, 137.1, 133.1, 128.7, 128.6, 128.5, 128.1, 126.1, 77.4, 37.8, 35.3, 25.8.

Compound 58

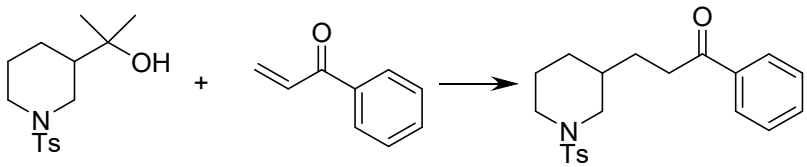


58, 64%(72 h)

1-phenyl-3-(1-tosylpiperidin-4-yl)propan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (47.6 mg, 64%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.92 (d, *J* = 7.6 Hz, 2H), 7.63 (d, *J* = 7.7 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.4 Hz, 2H), 7.31 (d, *J* = 7.7 Hz, 2H), 3.81 – 3.71 (m, 2H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 2.27 – 2.16 (m, 2H), 1.80 – 1.73 (m, 2H), 1.70 – 1.63 (m, 2H), 1.39 – 1.24 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.0, 143.5, 136.8, 133.2, 133.1, 129.7, 128.7, 128.1, 127.8, 77.4, 46.4, 35.4, 34.6, 31.4, 30.2, 21.6. **HRMS (APCI)**: calculated for $C_{21}H_{25}NO_3S$ $[M+H]^+$: 372.1628; found: 372.1620.

Compound 59



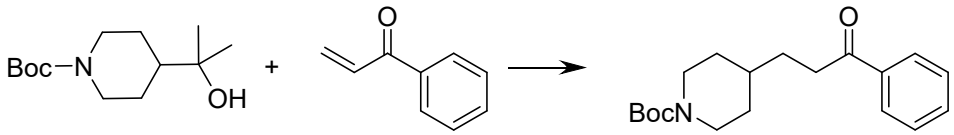
59, 72%(72 h)

1-phenyl-3-(1-tosylpiperidin-3-yl)propan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (53.5 mg, 72%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.96 (d, *J* = 7.7 Hz, 2H), 7.63 (d, *J* = 7.7 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.4 Hz, 2H), 7.32 (d, *J* = 7.8 Hz, 2H), 3.68 – 3.55 (m, 2H), 3.01 (t, *J* = 7.2 Hz, 2H), 2.43 (s, 3H), 2.36 – 2.27 (m, 1H), 2.10 – 2.00 (m, 1H), 1.84 – 1.55 (m, 6H), 0.98 – 0.85 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.8, 143.5, 136.8, 133.2, 133.2, 129.7, 128.8, 128.1, 127.8, 77.4, 51.6, 46.8, 35.9, 35.4, 30.2,

27.8, 24.4, 21.6. **HRMS (APCI)**: calculated for $C_{21}H_{25}NO_3S$ $[M+H]^+$: 372.1628; found: 372.1619.

Compound 60[15]

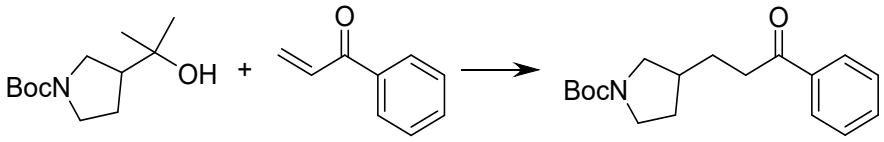


60, 58%(72 h)

tert-butyl 4-(3-oxo-3-phenylpropyl)piperidine-1-carboxylate

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (36.8 mg, 58%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.96 (d, *J* = 7.9 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 4.20 – 4.00 (m, 2H), 3.01 (t, *J* = 7.5 Hz, 2H), 2.74 – 2.61 (m, 2H), 1.76 – 1.66 (m, 4H), 1.46 (s, 10H), 1.20 – 1.09 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 200.3, 155.0, 137.0, 133.1, 128.7, 128.1, 79.4, 44.1, 35.7, 30.8, 28.6.

Compound 61

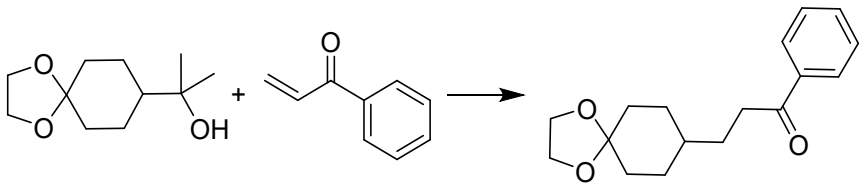


61, 51%(72 h)

tert-butyl (S)-3-(3-oxo-3-phenylpropyl)pyrrolidine-1-carboxylate

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (30.9 mg, 51%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.99 – 7.92 (m, 2H), 7.57 (t, *J* = 6.9 Hz, 1H), 7.47 (t, *J* = 7.1 Hz, 2H), 3.64 – 3.40 (m, 2H), 3.32 – 3.20 (m, 1H), 3.05 – 2.86 (m, 3H), 2.26 – 2.14 (m, 1H), 2.08 – 1.98 (m, 1H), 1.90 – 1.76 (m, 2H), 1.63 – 1.51 (m, 1H), 1.46 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 199.8, 154.7, 136.9, 133.3, 128.8, 128.1, 79.2, 51.7, 51.1, 45.8, 45.5, 38.8, 37.9, 37.1, 32.0, 31.2, 28.7, 27.6. **HRMS (ESI)**: calculated for $C_{18}H_{25}NO_3$ $[M+Na]^+$: 326.1727; found: 326.1719.

Compound 62

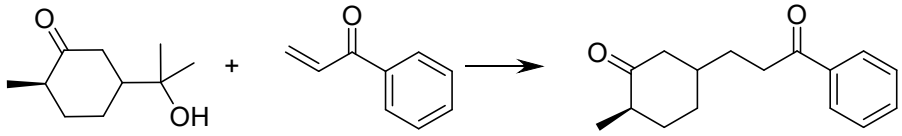


62, 63%(72 h)

1-phenyl-3-(1,4-dioxaspiro[4.5]decan-8-yl)propan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 20:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (34.6 mg, 63%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 3.94 (s, 4H), 3.02 – 2.96 (m, 2H), 1.81 – 1.73 (m, 4H), 1.72 – 1.66 (m, 2H), 1.59 – 1.49 (m, 2H), 1.41 – 1.23 (m, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.6, 137.1, 133.0, 128.7, 128.2, 109.1, 64.4, 64.3, 36.4, 36.1, 34.6, 30.7, 30.2. **HRMS (APCI)**: calculated for $C_{17}H_{22}O_3$ $[M+H]^+$: 275.1642; found: 275.1635.

Compound 63



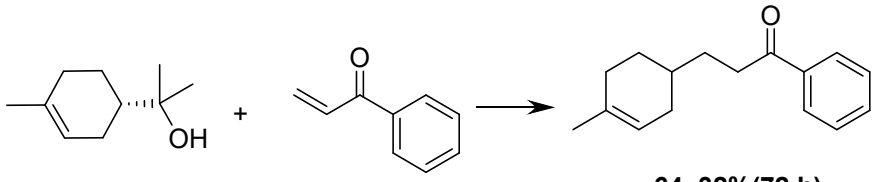
63, 51%(72 h)
d.r.=1.2:1

2-methyl-5-(3-oxo-3-phenylpropyl)cyclohexan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (24.9 mg, 51%). The isolated product gives 1.2:1 dr ratio and two sets of NMR signals, owing to the presence of diastereoisomers. **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 8.05 – 7.93 (m, 4.29H), 7.61 – 7.54 (m, 2.21H), 7.53 – 7.44 (m, 4.16H), 3.07 – 2.91 (m, 4.30H), 2.52 – 2.43 (m, 3.04H), 2.40 – 2.34 (m, 1.18H), 2.32 – 2.27 (m, 1.00H), 2.17 – 2.02 (m, 3.53H), 1.99 – 1.91 (m, 2.43H), 1.90 – 1.80 (m, 3.04H), 1.79 – 1.73 (m, 2.69H), 1.72 –

1.64 (m, 2.70H), 1.50 – 1.43 (m, 1.25H), 1.39 – 1.31 (m, 1.31H), 1.08 (d, *J* = 6.7 Hz, 2.97H), 1.02 (d, *J* = 6.3 Hz, 3.46H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 213.9, 212.6, 199.8, 199.8, 136.9, 133.2, 133.2, 128.7, 128.1, 128.1, 48.2, 45.5, 44.9, 44.7, 40.2, 37.7, 36.1, 35.9, 34.9, 32.2, 31.4, 31.1, 28.3, 28.3, 15.5, 14.4. **HRMS (APCI)**: calculated for $C_{16}H_{20}O_2$ $[M+H]^+$: 245.1536; found: 245.1530.

Compound 64

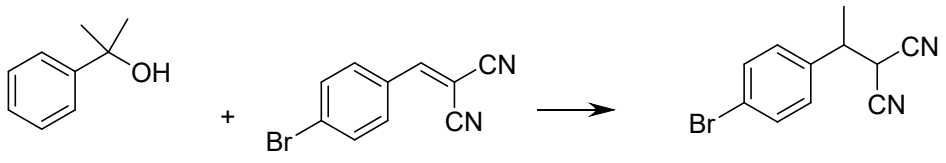


64, 32%(72 h)

3-(4-methylcyclohex-3-en-1-yl)-1-phenylpropan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 30:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (14.6 mg, 32%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.96 (d, *J* = 7.4 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 5.39 – 5.33 (m, 1H), 3.06 – 2.95 (m, 2H), 2.18 – 2.07 (m, 1H), 2.06 – 1.87 (m, 2H), 1.84 – 1.76 (m, 1H), 1.74 – 1.68 (m, 2H), 1.64 (s, 3H), 1.58 – 1.51 (m, 1H), 1.37 – 1.19 (m, 2H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 200.8, 137.2, 134.2, 133.0, 128.7, 128.2, 120.4, 36.4, 33.3, 32.0, 30.9, 30.2, 29.3, 23.7. **HRMS (APCI)**: calculated for $C_{16}H_{20}O$ $[M+H]^+$: 229.1587; found: 229.1579.

Compound 65[17]



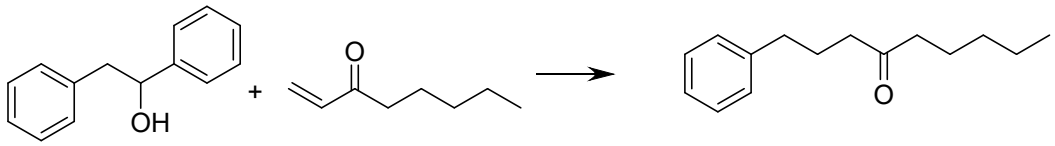
65, 51%

2-(1-(4-bromophenyl)ethyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 2-(4-bromobenzylidene)malononitrile (46.6 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and

purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a light yellow oil (25.4 mg, 51%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.55 (d, *J* = 8.3 Hz, 2H), 7.29 – 7.19 (m, 3H), 3.83 (d, *J* = 6.0 Hz, 1H), 3.48 – 3.39 (m, 1H), 1.64 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 137.1, 132.6, 129.1, 123.2, 111.7, 111.5, 40.8, 31.2, 17.9.

Compound 66[16]

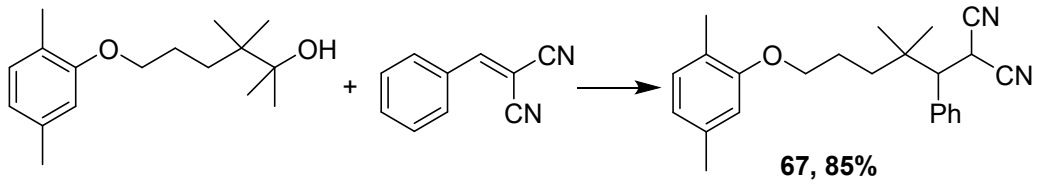


66, 87%(72 h)

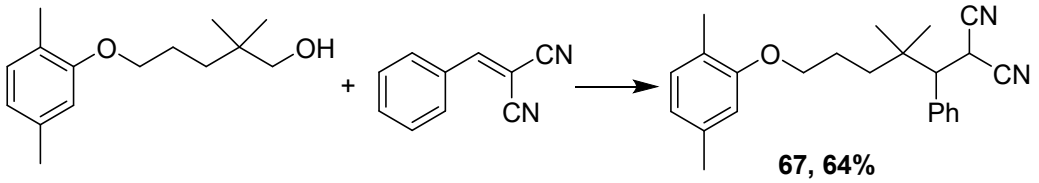
1-phenylnonan-4-one

Prepared following the general procedure with 1,2-diphenylethan-1-ol (79.3 mg, 0.4 mmol, 2.0 equiv.) and relevant michael acceptor. After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 60:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (38.0 mg, 87%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.31 – 7.25 (m, 2H), 7.23 – 7.13 (m, 3H), 2.62 (t, *J* = 7.6 Hz, 2H), 2.44 – 2.33 (m, 4H), 1.95 – 1.86 (m, 2H), 1.59 – 1.50 (m, 2H), 1.33 – 1.22 (m, 4H), 0.88 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 211.3, 141.8, 128.6, 128.5, 126.0, 43.0, 42.0, 35.3, 31.6, 25.4, 23.7, 22.6, 14.1.

Compound 67



67, 85%



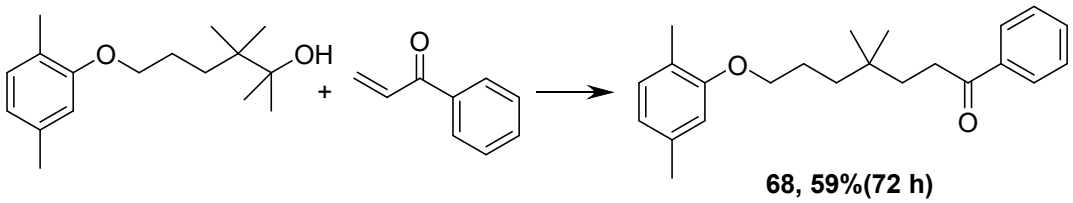
67, 64%

2-(5-(2,5-dimethylphenoxy)-2,2-dimethyl-1-phenylpentyl)malononitrile

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and

purified by flash column chromatography (silica gel, 8:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (61.3 mg, 85%), (46.1 mg, 64%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.46 – 7.33 (m, 5H), 7.00 (d, *J* = 7.5 Hz, 1H), 6.67 (d, *J* = 7.5 Hz, 1H), 4.23 (d, *J* = 5.5 Hz, 1H), 3.95 – 3.85 (m, 2H), 3.10 (d, *J* = 5.5 Hz, 1H), 2.30 (s, 3H), 2.14 (s, 3H), 1.85 – 1.73 (m, 2H), 1.61 – 1.47 (m, 2H), 1.18 (s, 3H), 1.06 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.8, 136.7, 135.9, 130.5, 129.6, 128.9, 128.8, 123.6, 121.0, 113.4, 113.1, 112.0, 67.6, 55.2, 37.5, 37.3, 25.4, 25.3, 24.9, 24.0, 21.5, 15.9. **HRMS (ESI)**: calculated for $C_{24}H_{28}N_2O$ $[M+Na]^+$: 383.2094; found: 383.2084.

Compound 68

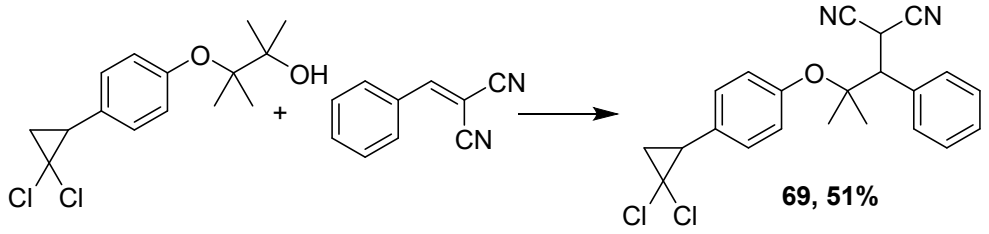


68, 59%(72 h)

7-(2,5-dimethylphenoxy)-4,4-dimethyl-1-phenylheptan-1-one

Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and 1-phenylprop-2-en-1-one (26.4 mg, 0.2 mmol). After 72 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 40:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (39.9 mg, 59%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.96 (d, *J* = 7.4 Hz, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 2H), 6.99 (d, *J* = 7.4 Hz, 1H), 6.68 – 6.59 (m, 2H), 3.93 (t, *J* = 6.3 Hz, 2H), 2.98 – 2.89 (m, 2H), 2.30 (s, 3H), 2.17 (s, 3H), 1.83 – 1.73 (m, 2H), 1.72 – 1.63 (m, 2H), 1.47 – 1.39 (m, 2H), 0.97 (s, 6H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 201.0, 157.1, 137.2, 136.6, 133.0, 130.4, 128.7, 128.2, 123.6, 120.7, 112.1, 68.5, 38.1, 35.9, 33.8, 32.5, 27.2, 24.4, 21.5, 16.0. **HRMS (APCI)**: calculated for $C_{23}H_{30}O_2$ $[M+H]^+$: 339.2319; found: 339.2311.

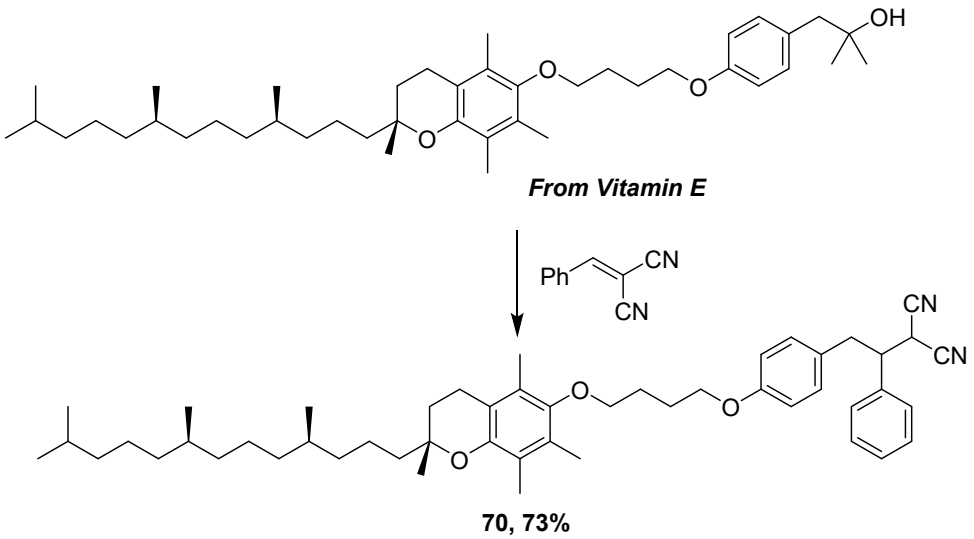
Compound 69



69, 51%

2-(2-(4-(2,2-dichlorocyclopropyl)phenoxy)-2-methyl-1-phenylpropyl)malononitrile Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM to afford the titled compound as a light yellow oil (40.7 mg, 51%). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.57 – 7.52 (m, 2H), 7.43 (d, *J* = 5.6 Hz, 3H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 4.77 (d, *J* = 6.0 Hz, 1H), 3.41 (d, *J* = 6.0 Hz, 1H), 2.91 – 2.83 (m, 1H), 2.00 – 1.95 (m, 1H), 1.81 (t, *J* = 7.9 Hz, 1H), 1.40 (s, 3H), 1.25 (s, 3H). **^{13}C NMR (151 MHz, Chloroform-*d*)** δ 152.9, 135.3, 131.1, 129.9, 129.8, 129.3, 129.2, 124.1, 113.5, 113.1, 81.5, 60.8, 57.3, 35.0, 27.1, 26.1, 24.9, 24.4. **HRMS (APCI)**: calculated for $C_{22}H_{20}Cl_2N_2O$ $[M+H]^+$: 399.1026; found: 399.1020.

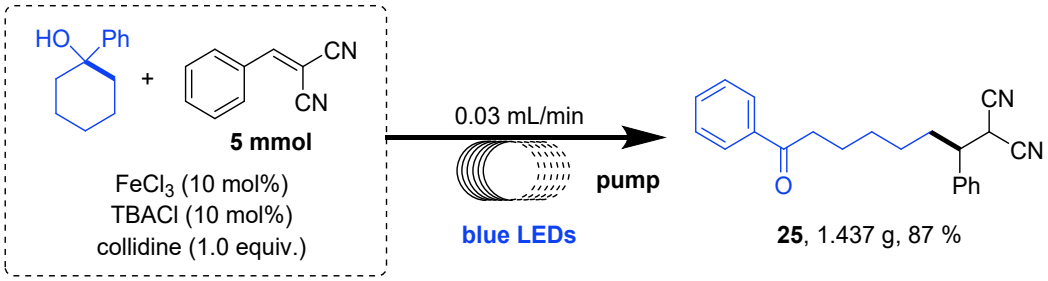
Compound 70



Prepared on 0.2 mmol scale following the general procedure with relevant alcohol and benzylidenemalononitrile (30.8 mg, 0.2 mmol). After 48 h irradiation, the reaction mixture was filtered through a short silica column and washed with DCM. The solvent was removed and purified by flash column chromatography (silica gel, 2:1 Petroleum ether/DCM) to afford the titled compound as a light yellow oil (109.1 mg, 73%). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.44 – 7.36 (m, 5H), 7.09 (d, *J* = 8.4 Hz, 2H), 6.86 (d, *J* = 8.4 Hz, 2H), 4.03 (t, *J* = 6.2 Hz, 2H), 3.86 (d, *J* = 5.1 Hz, 1H), 3.71 (t, *J* = 6.2 Hz, 2H), 3.43 – 3.38 (m, 1H), 3.24 – 3.16 (m, 2H), 2.57 (t, *J* = 6.7 Hz, 2H), 2.17 (s, 3H), 2.13 (s, 3H), 2.08 (s, 3H), 2.06 – 2.00 (m, 2H), 1.99 – 1.93 (m, 2H),

1.84 – 1.78 (m, 1H), 1.78 – 1.72 (m, 1H), 1.55 – 1.49 (m, 2H), 1.47 – 1.03 (m, 22H), 0.88 – 0.83 (m, 12H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 158.7, 148.4, 147.9, 136.7, 130.1, 129.3, 129.1, 128.5, 128.2, 127.9, 125.9, 123.0, 117.7, 115.3, 112.3, 111.6, 74.9, 72.6, 68.1, 48.7, 40.2, 39.5, 37.9, 37.6, 37.6, 37.6, 37.4, 32.9, 32.9, 31.5, 28.5, 28.1, 27.2, 26.5, 24.9, 24.6, 24.0, 22.9, 22.8, 21.2, 20.8, 19.9, 19.8, 12.9, 12.1, 11.9. **HRMS (APCI)**: calculated for $C_{50}H_{70}N_2O_3$ $[M+H]^+$: 747.5459; found: 747.5434.

3.4 Gram-Scale and Large-Scale Reaction



A 100 mL heart shaped bottle flask equipped with a magnetic stirbar was charged with 1-phenylcyclohexan-1-ol (1763 mg, 10 mmol, 2.0 equiv.) and benzylidenemalononitrile (771 mg, 5 mmol, 1.0 equiv.). The flask was then transferred into the glovebox, where $FeCl_3$/TBACl solution (50 mL) from the general procedure was added. Then, collidine (650 µL, 5 mmol, 1.0 equiv.) was added via syringe under nitrogen atmosphere outside the glovebox. As shown in **Figure S4**, a peristaltic pump was filled with the reaction mixture and attached to the flow apparatus made with a condenser. The flow apparatus itself was cooled by Flowing water as well as additional cooling fans, and equipped with six 50 W 450nm blue LEDs. The flow apparatus itself was set up with flow rate = 0.03 mL/min, after approximately 28 h the reacted red brown solution was collected. Excess solvent was removed under reduced pressure followed by purification by flash column chromatography (silica gel, 10:1 Petroleum ether/ EtOAc) to obtain the crude product contains a very small amount of collidine. Then purified through a short silica column and washed with DCM to obtain the pure product (1.437 g, 87%).

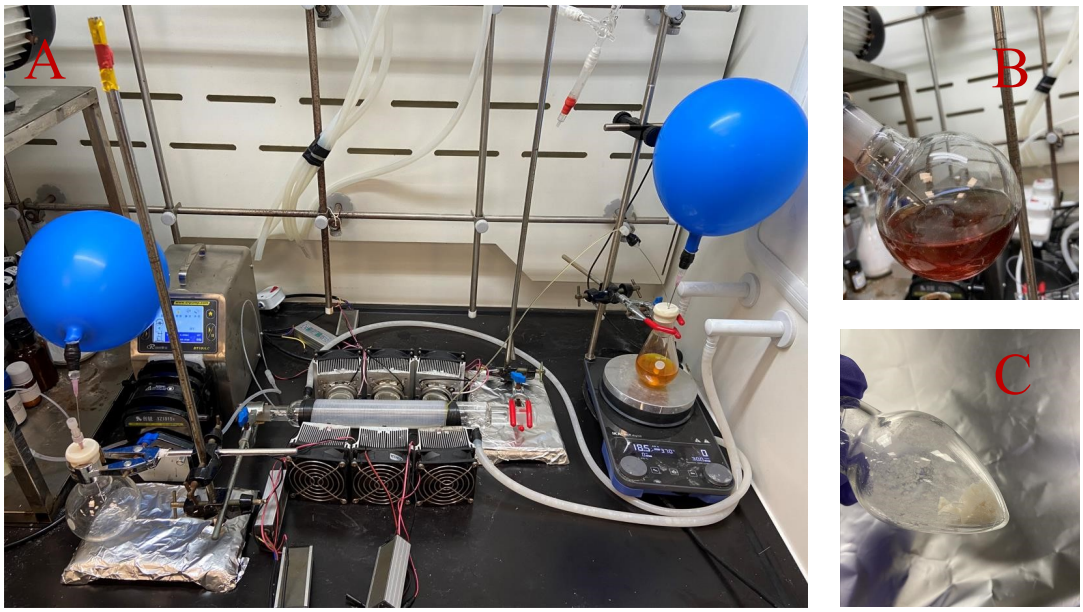
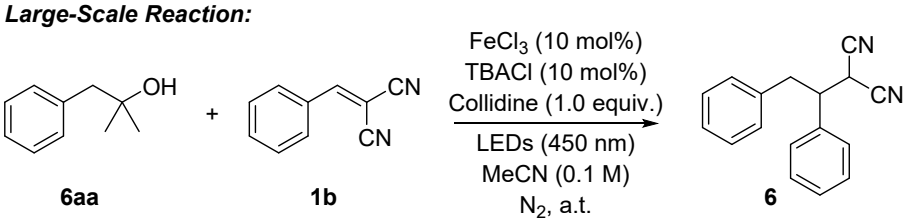


Figure S4. The gram-scale reaction. A, The continuous-flow setup. **B**, The mixture after irradiation. **C**, Compound **25** purified by column chromatography.

Large-Scale Reaction:



6aa **1b** **6**

For the large scale experiment, A 100 mL Schlenk tube equipped with a magnetic stirbar was charged with 2-methyl-1-phenylpropan-2-ol (601 mg, 4 mmol, 2.0 equiv.) and benzylidenemalononitrile (308 mg, 2 mmol, 1.0 equiv.). The flask was then transferred into the glovebox, where $FeCl_3$/TBACl solution (20 mL) from the general procedure was added. Then, collidine (260 μL, 2 mmol, 1.0 equiv.) was added via syringe under nitrogen atmosphere outside the glovebox. As shown in **Figure S5**, the resulting mixture was irradiated by two 50W blue LED lamps and stirred at ambient temperature. After 5 days, the reaction mixture was concentrated and then purified by flash column chromatography to obtain the product **6** as a light yellow oil (369 mg, 75%).

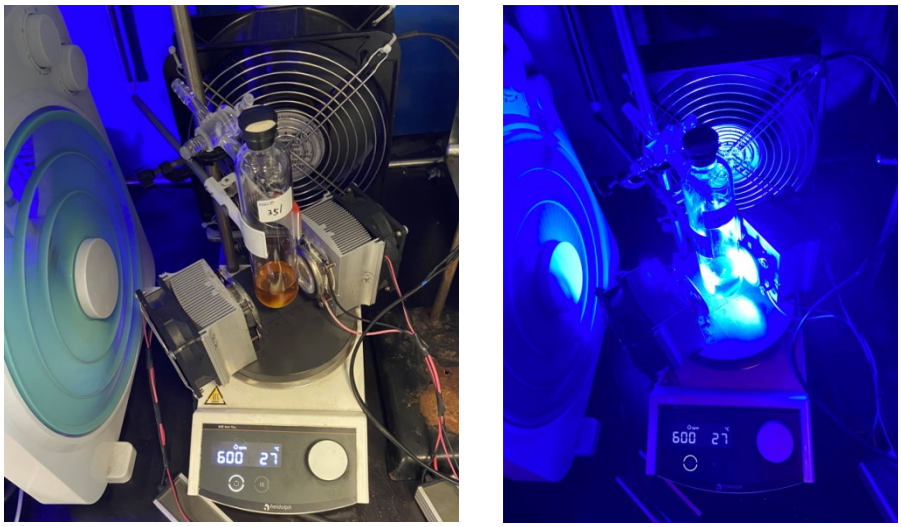
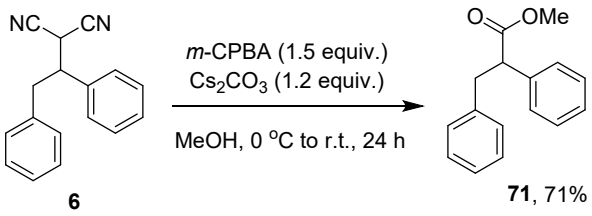


Figure S5. The large-scale reaction setup

3.5 Transformation of Product

Compound 71[18]

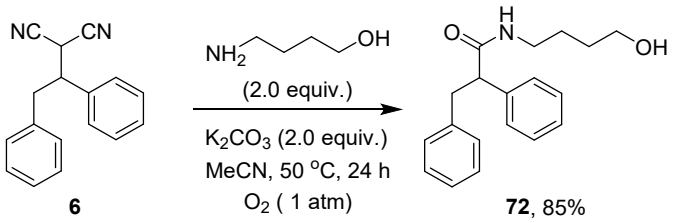


6 **71**, 71%

methyl 2,3-diphenylpropanoate

Under N_2 atmosphere, a flame-dried 25 mL schlenk flask was charged with compound **6** (49.3 mg, 0.2 mmol, 1.0 equiv.) and anhydrous cesium carbonate (78.2 mg, 0.24 mmol, 1.2 equiv.). Then *m*-CPBA (51.8 mg, 0.3 mmol, 1.5 equiv.) dissolved in 2 mL anhydrous MeOH was added dropwise into the mixture at 0 ºC. The reaction was stirred at room temperature for 24 h. Upon completion, The solvent was removed and purified by flash column chromatography (silica gel, 20:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (34.1 mg, 71%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.33 – 7.28 (m, 4H), 7.27 – 7.14 (m, 4H), 7.11 (d, *J* = 7.0 Hz, 2H), 3.89 – 3.82 (m, 1H), 3.59 (s, 3H), 3.46 – 3.38 (m, 1H), 3.06 – 2.97 (m, 1H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 174.0, 139.2, 138.8, 129.0, 128.8, 128.5, 128.1, 127.5, 126.5, 53.7, 52.1, 39.9, 19.3.

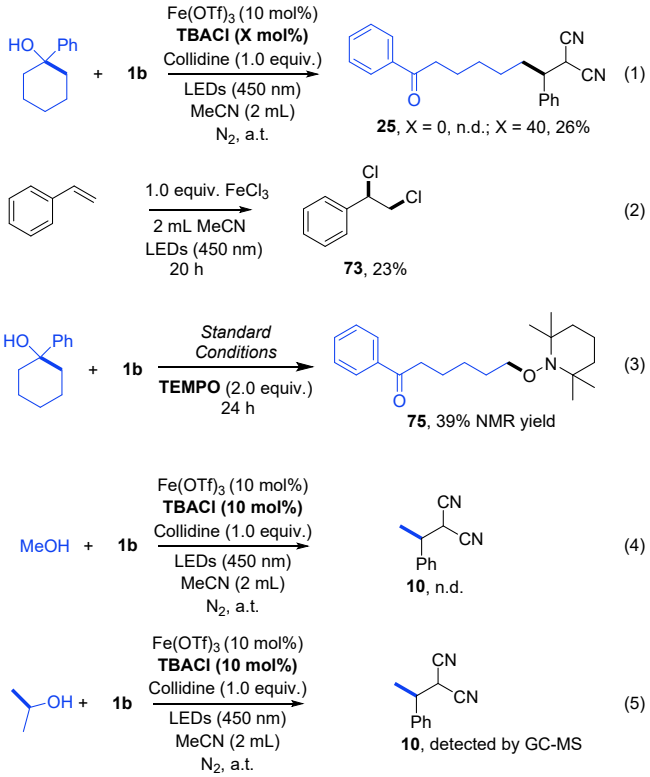
Compound 72[19]



N-(4-hydroxybutyl)-2,3-diphenylpropanamide

Under O_2 balloon atmosphere, a flame-dried 25 mL schlenk flask was charged with compound **6** (49.3 mg, 0.2 mmol, 1.0 equiv.), anhydrous potassium carbonate (55.3 mg, 0.4 mmol, 2.0 equiv.) and 4-amino-1-butanol (35.7 mg, 0.4 mmol, 2.0 equiv.) Then anhydrous MeCN was added as solvent and stirred at 50 °C for 24 h. Upon completion, The solvent was removed and purified by flash column chromatography (silica gel, 1:2 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (50.6 mg, 85%). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.38 – 7.07 (m, 10H), 5.67 (s, 1H), 3.59 – 3.45 (m, 4H), 3.26 – 3.17 (m, 1H), 3.16 – 3.06 (m, 1H), 3.02 – 2.93 (m, 1H), 1.46 – 1.31 (m, 4H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 172.9, 139.9, 139.9, 129.2, 128.8, 128.4, 128.1, 127.4, 126.3, 62.4, 55.8, 39.8, 39.4, 29.6, 26.1.

4. Mechanistic Studies.



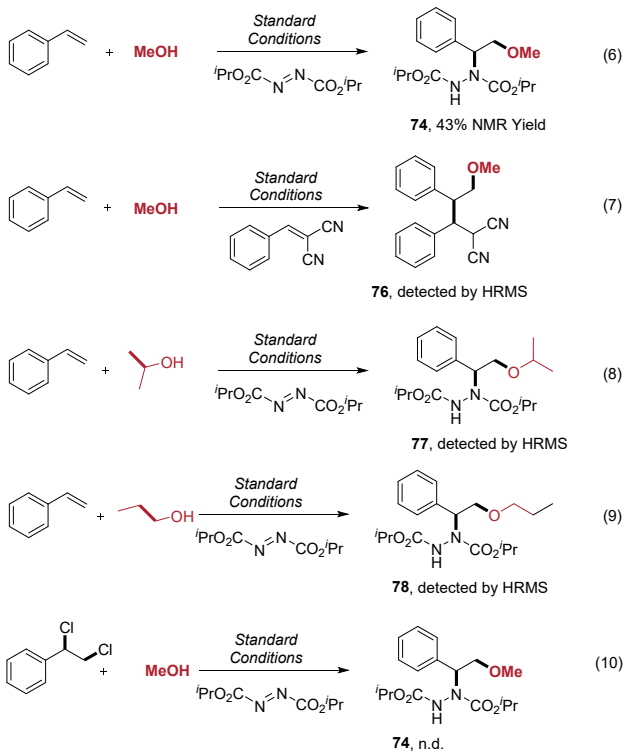
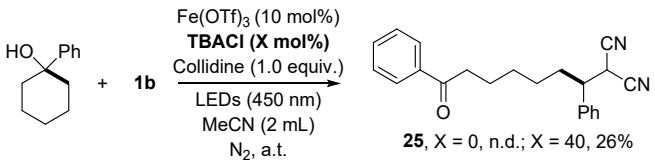


Figure S6. Mechanistic study.

4.1 Chlorine Source Control Experiment

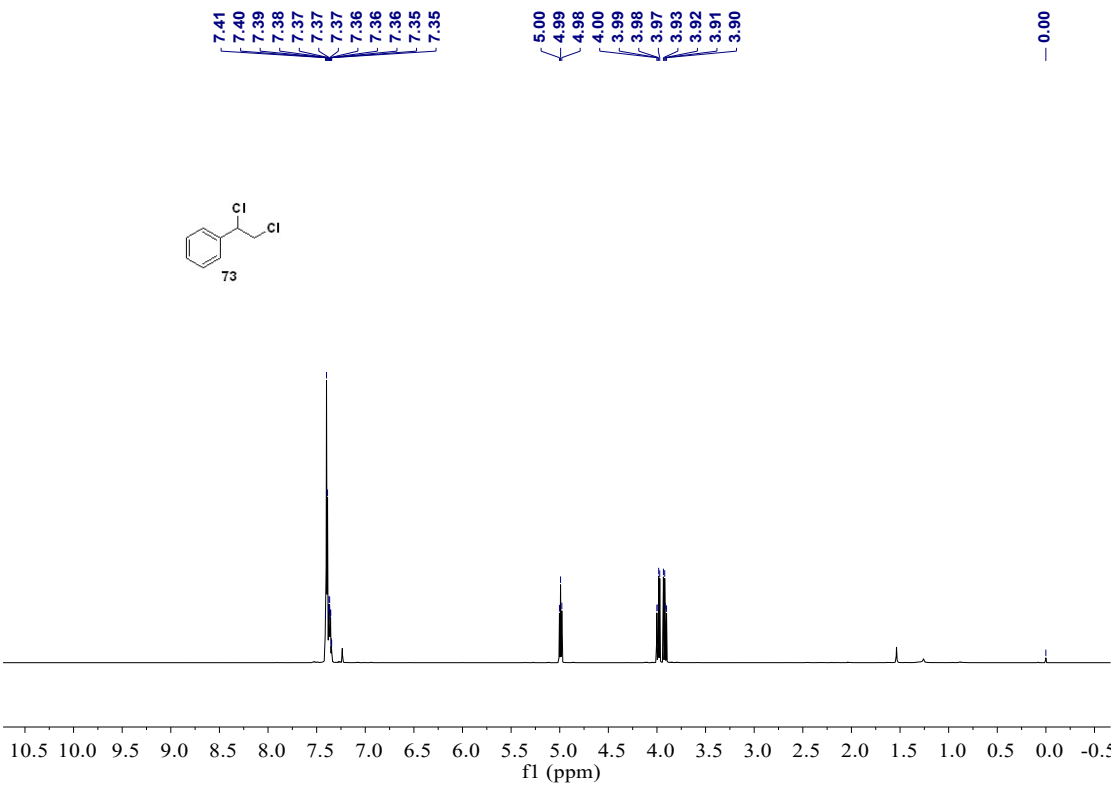


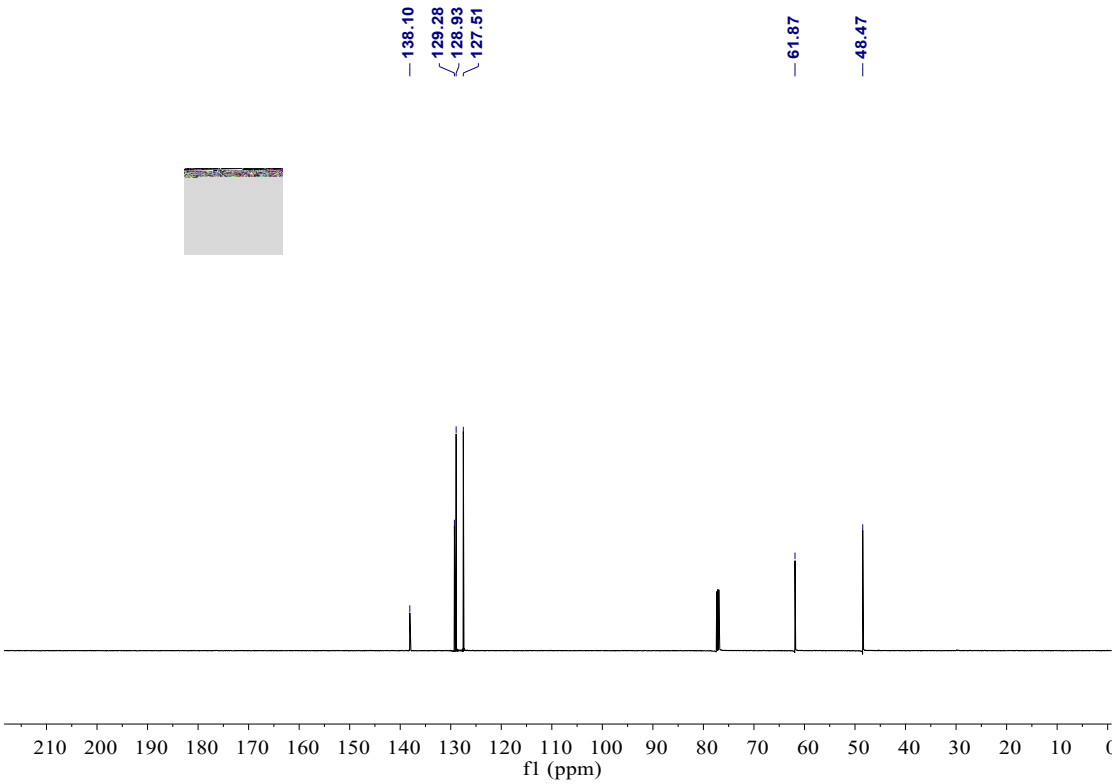
A 25 mL Schlenk tube with screw-cap was added 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.) and benzylidenemalononitrile (30.8 mg, 0.2 mmol, 1.0 equiv.). The Schlenk tube was then transferred into the glovebox, where $Fe(OTf)_3$ (4.1 mg, 0.02 mmol, 0.1 equiv.) and TBACl (X mol%) were added. Then, collidine (26 μL, 0.2 mmol, 1.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps. After 48 hours, the yield of **25** was determined by ^1H NMR analysis using 1,3,5-trimethoxybenzene as an internal standard. In the absence of chlorine source, **25** was not detected. The NMR yield of **25** was 26% when TBACl (22.2 mg, 0.08 mmol, 0.4 equiv.) was added.

4.2 Chlorine Radical Trapping Reaction

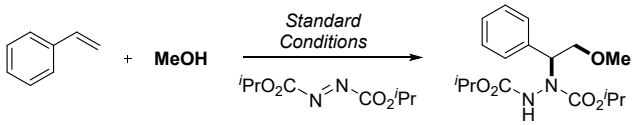
73, 23%

A 25 mL Schlenk tube with screw-cap was added $FeCl_3$ (32.4 mg, 0.2 mmol, 1.0 equiv.) in the glovebox. Then, styrene (23 μL, 0.2 mmol, 1.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps for 20 h. Upon completion, the solvent was removed and purified by flash column chromatography (silica gel, 40:1 Petroleum ether/EtOAc) to afford the titled compound as a colorless oil (8.1 mg, 23%). **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.45 – 7.35 (m, 5H), 5.01 (t, *J* = 7.3 Hz, 1H), 4.03 – 3.99 (m, 1H), 3.96 – 3.92 (m, 1H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 138.1, 129.3, 128.9, 127.5, 61.9, 48.5. Spectra are consistent with reported literature values.[20]



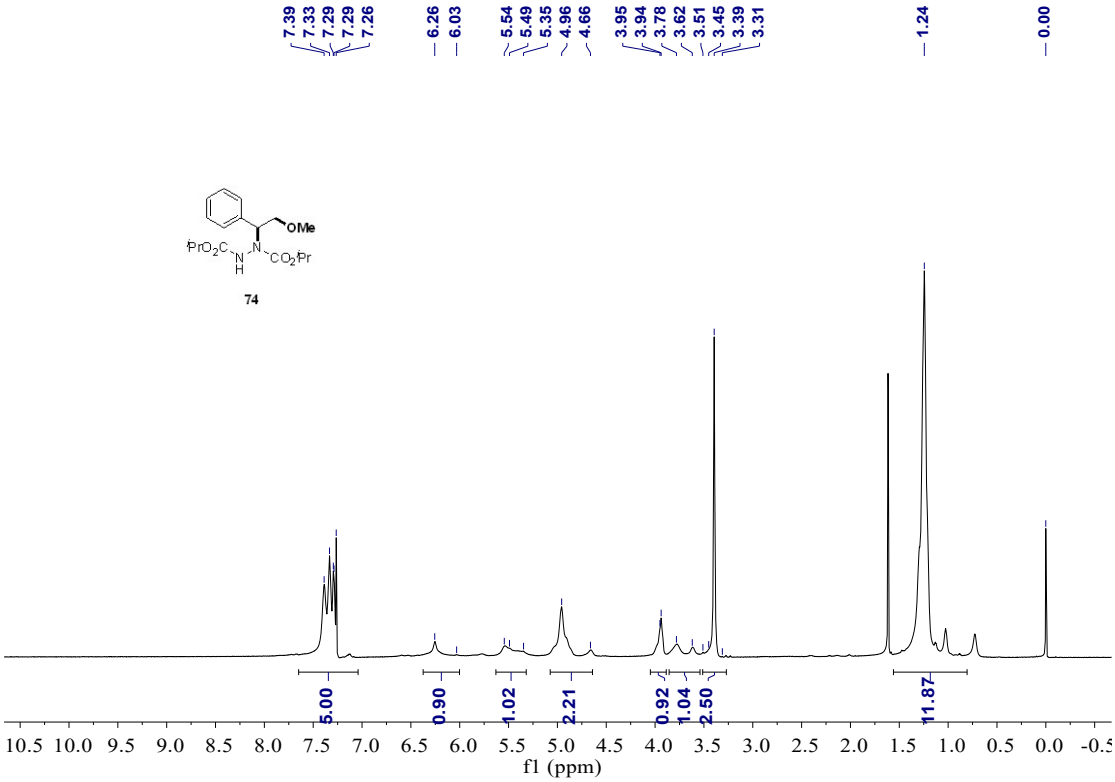


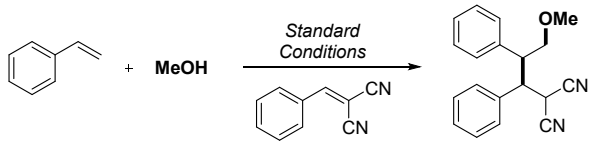
4.3 Alkoxy Radical Trapping Reaction



74, 43% NMR Yield

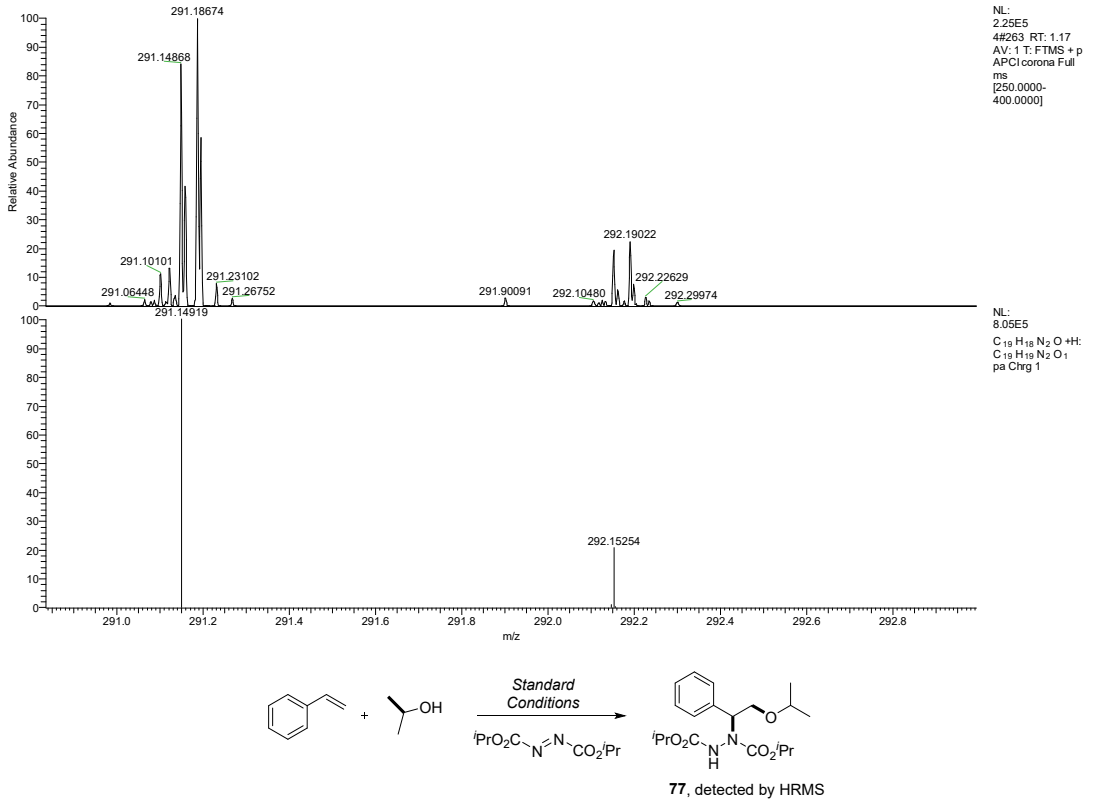
A 25 mL Schlenk tube with screw-cap was transferred into the glovebox, where $FeCl_3$ (3.2 mg, 0.02 mmol, 0.1 equiv.) and TBACl (5.6 mg, 0.02 mmol, 0.1 equiv.) were added. Then, diisopropyl azodicarboxylate (40.4 mg, 0.2 mmol, 1.0 equiv.), styrene (92 µL, 0.8 mmol, 4.0 equiv.), collidine (26 µL, 0.2 mmol, 1.0 equiv.), anhydrous MeOH (16 µL, 0.4 mmol, 2.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps. After 12 hours, the yield of **74** was 43% determined by ^{1}H NMR analysis using 3,4,5-Trichloropyridine as an internal standard. For NMR analysis, **74** was purified by flash column chromatography (silica gel, 5:1 Petroleum ether/EtOAc) as a colorless oil. **^{1}H NMR (600 MHz, Chloroform-*d*)** δ 7.65 – 7.04 (m, 5H), 6.38 – 6.00 (m, 1H), 5.63 – 5.32 (m, 1H), 5.07 – 4.64 (m, 2H), 4.05 – 3.89 (m, 1H), 3.86 – 3.54 (m, 1H), 3.39 (s, 3H), 1.24 (s, 12H). Spectra are consistent with reported literature values.[21]





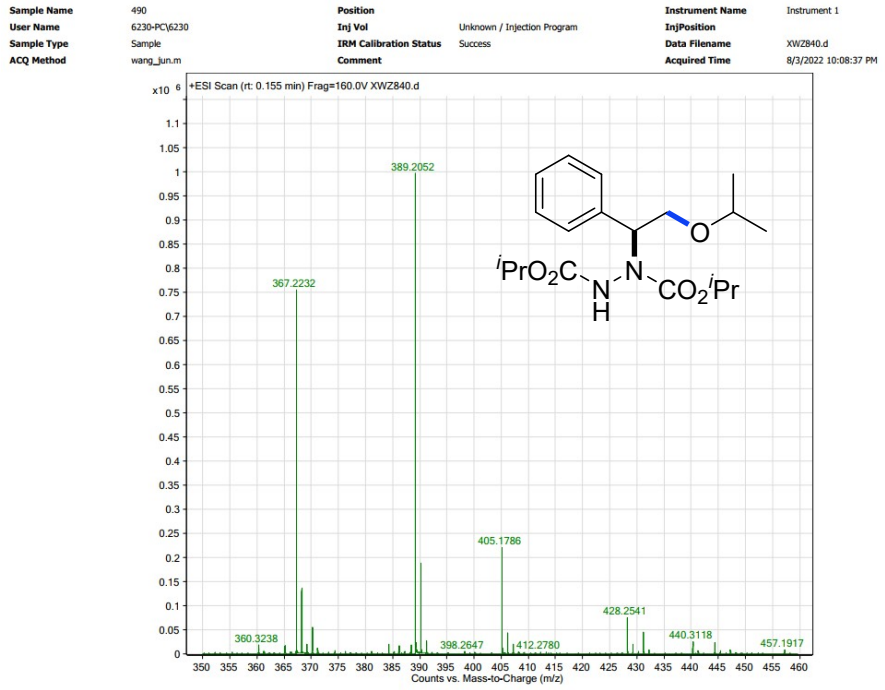
76, detected by HRMS

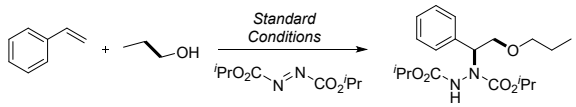
A 25 mL Schlenk tube with screw-cap was transferred into the glovebox, where $FeCl_3$ (3.2 mg, 0.02 mmol, 0.1 equiv.) and TBACl (5.6 mg, 0.02 mmol, 0.1 equiv.) were added. Then, benzylidenemalononitrile (30.8 mg, 0.2 mmol, 1.0 equiv.), styrene (92 μL, 0.8 mmol, 4.0 equiv.), collidine (26 μL, 0.2 mmol, 1.0 equiv.), anhydrous MeOH (16 μL, 0.4 mmol, 2.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps. After 12 hours, **76** was detected by HRMS. **HRMS (APCI)**: calculated for $C_{19}H_{19}N_2O$ $[M+H]^+$: 291.1492; found: 291.1487.



77, detected by HRMS

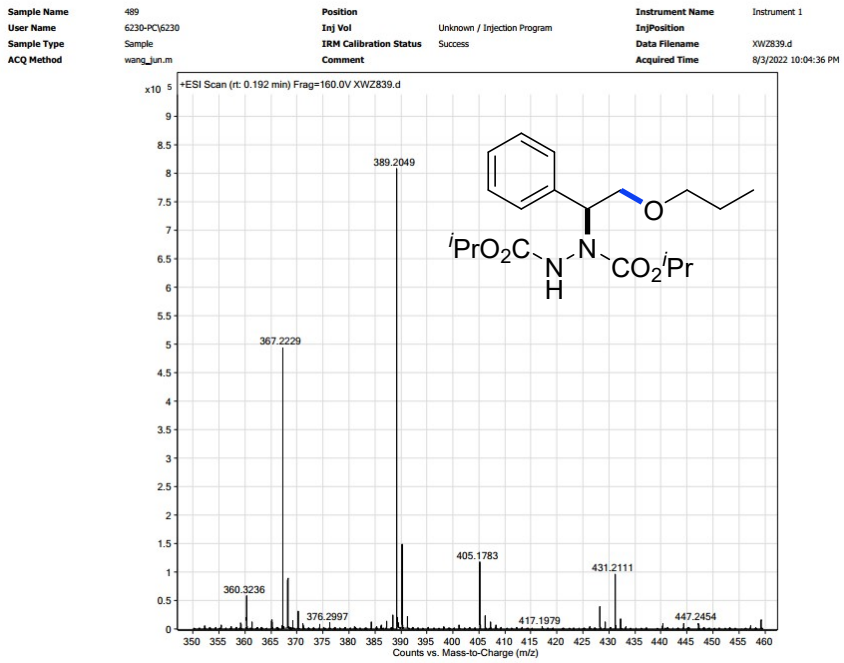
A 25 mL Schlenk tube with screw-cap was transferred into the glovebox, where $FeCl_3$ (3.2 mg, 0.02 mmol, 0.1 equiv.) and TBACl (5.6 mg, 0.02 mmol, 0.1 equiv.) were added. Then, diisopropyl azodicarboxylate (40.4 mg, 0.2 mmol, 1.0 equiv.), styrene (92 μL, 0.8 mmol, 4.0 equiv.), collidine (26 μL, 0.2 mmol, 1.0 equiv.), isopropanol (0.4 mmol, 2.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps. After 12 hours, **77** was detected by HRMS. **HRMS (ESI)**: calculated for $C_{19}H_{30}N_2O_5$ $[M+H]^+$: 367.2227; found: 367.2232. calculated for $C_{19}H_{30}N_2O_5$ $[M+Na]^+$: 389.2047; found: 389.2052.



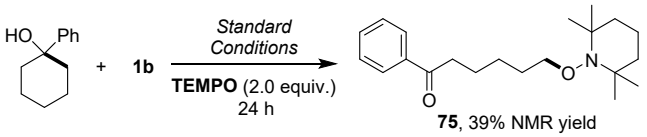


78, detected by HRMS

A 25 mL Schlenk tube with screw-cap was transferred into the glovebox, where $FeCl_3$ (3.2 mg, 0.02 mmol, 0.1 equiv.) and TBACl (5.6 mg, 0.02 mmol, 0.1 equiv.) were added. Then, diisopropyl azodicarboxylate (40.4 mg, 0.2 mmol, 1.0 equiv.), styrene (92 μL, 0.8 mmol, 4.0 equiv.), collidine (26 μL, 0.2 mmol, 1.0 equiv.), *n*-propanol (0.4 mmol, 2.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps. After 12 hours, **78** was detected by HRMS. **HRMS (ESI)**: calculated for $C_{19}H_{30}N_2O_5$ $[M+H]^+$: 367.2227; found: 367.2229. calculated for $C_{19}H_{30}N_2O_5$ $[M+Na]^+$: 389.2047; found: 389.2049.

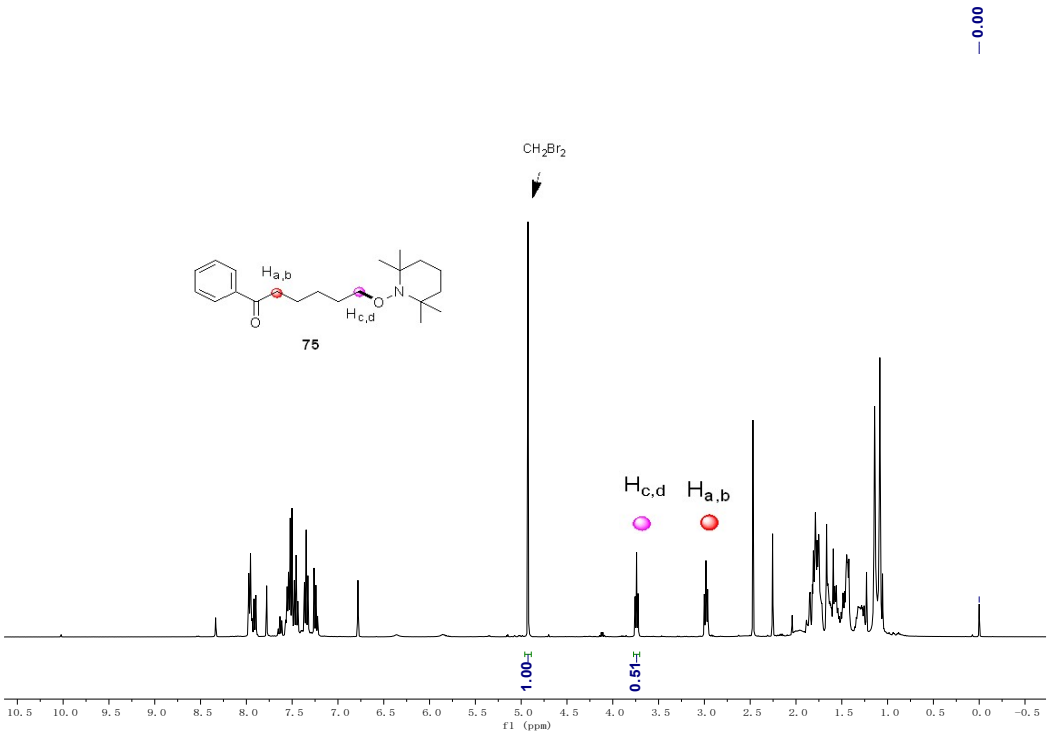


4.4 Radical-Captured Experiment



75, 39% NMR yield

A 25 mL Schlenk tube with screw-cap was added 1-phenylcyclohexan-1-ol (70.5 mg, 0.4 mmol, 2.0 equiv.), 2,2,6,6-tetramethylpiperidinooxy (TEMPO) (62.5 mg, 0.4 mmol, 2.0 equiv.) and benzylidenemalononitrile (30.8 mg, 0.2 mmol, 1.0 equiv.). The tube was then transferred into the glovebox, where $FeCl_3$ (3.2 mg, 0.02 mmol, 0.1 equiv.) and TBACl (5.6 mg, 0.02 mmol, 0.1 equiv.) were added. Then, collidine (26 μL, 0.2 mmol, 1.0 equiv.) and 2 mL anhydrous MeCN were added via syringe under nitrogen atmosphere outside the glovebox. The resulting mixture was irradiated by two 50W blue LED lamps. After 24 hours, the NMR yield of **75** was 39% determined by ^{1}H NMR analysis using dibromomethane (53.6 mg) as an internal standard. Spectra are consistent with reported literature values.[1]



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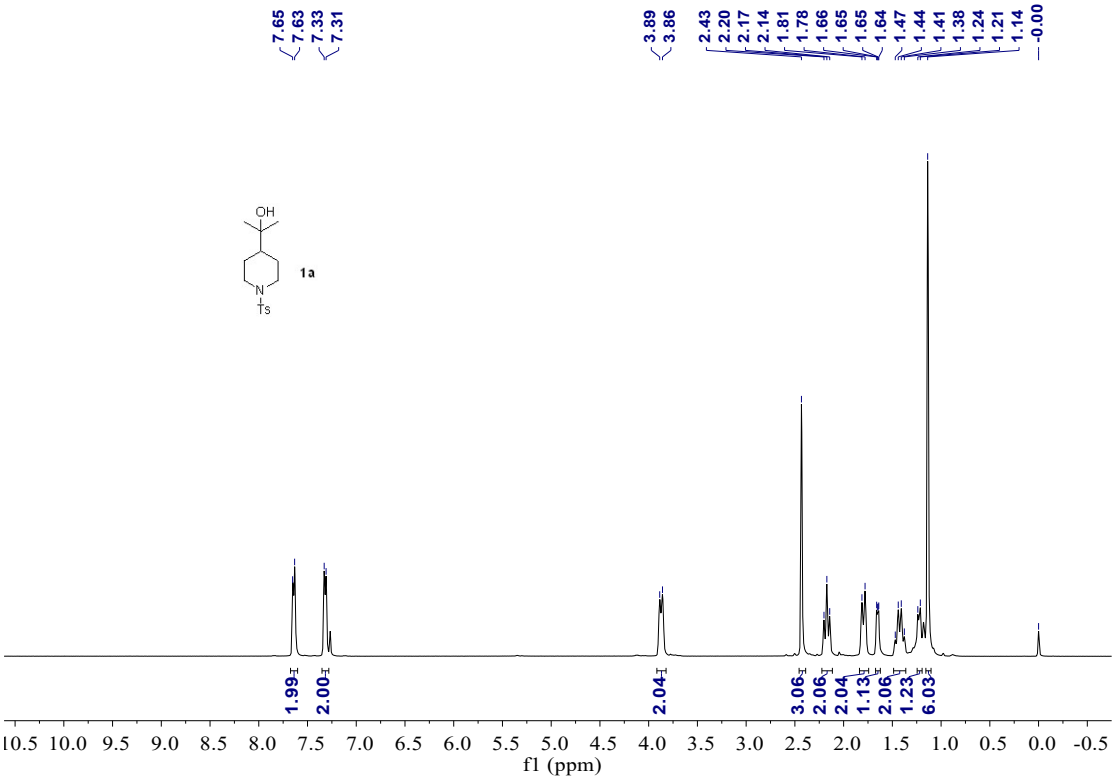
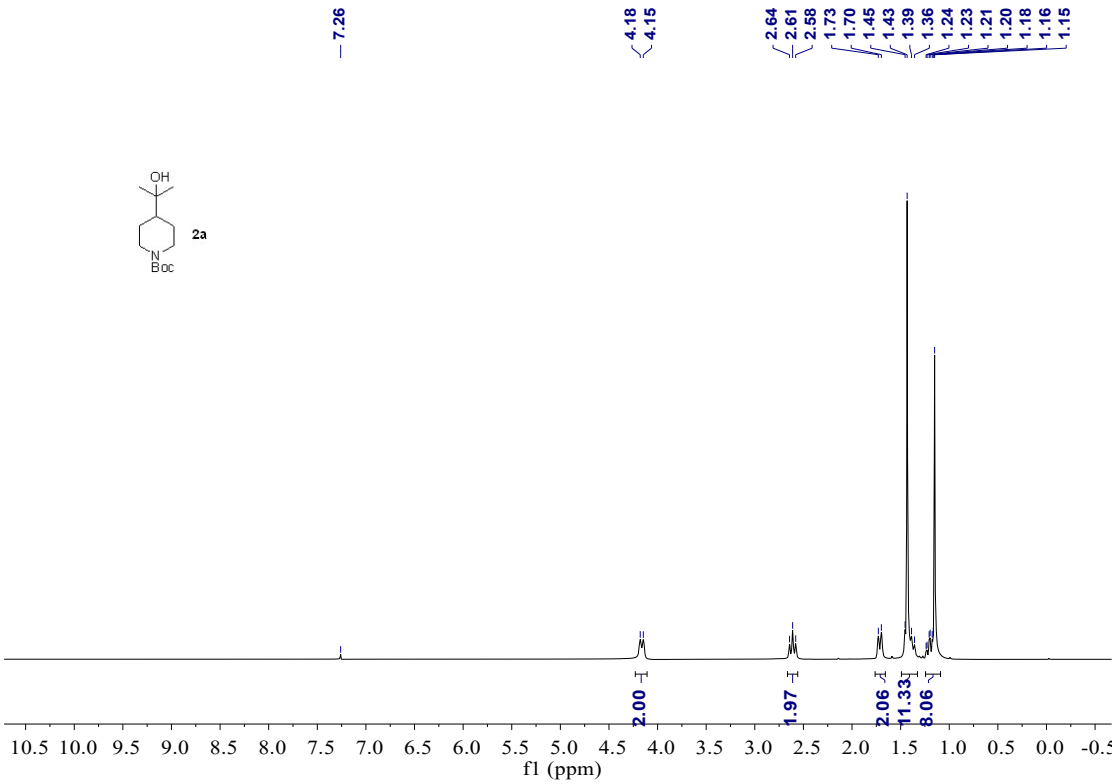
6. NMR Spectra of Products

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2.58
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1.70
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1.43
1.39
1.36
1.24
1.23
1.21
1.20
1.18
1.16
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N
Boc
2a
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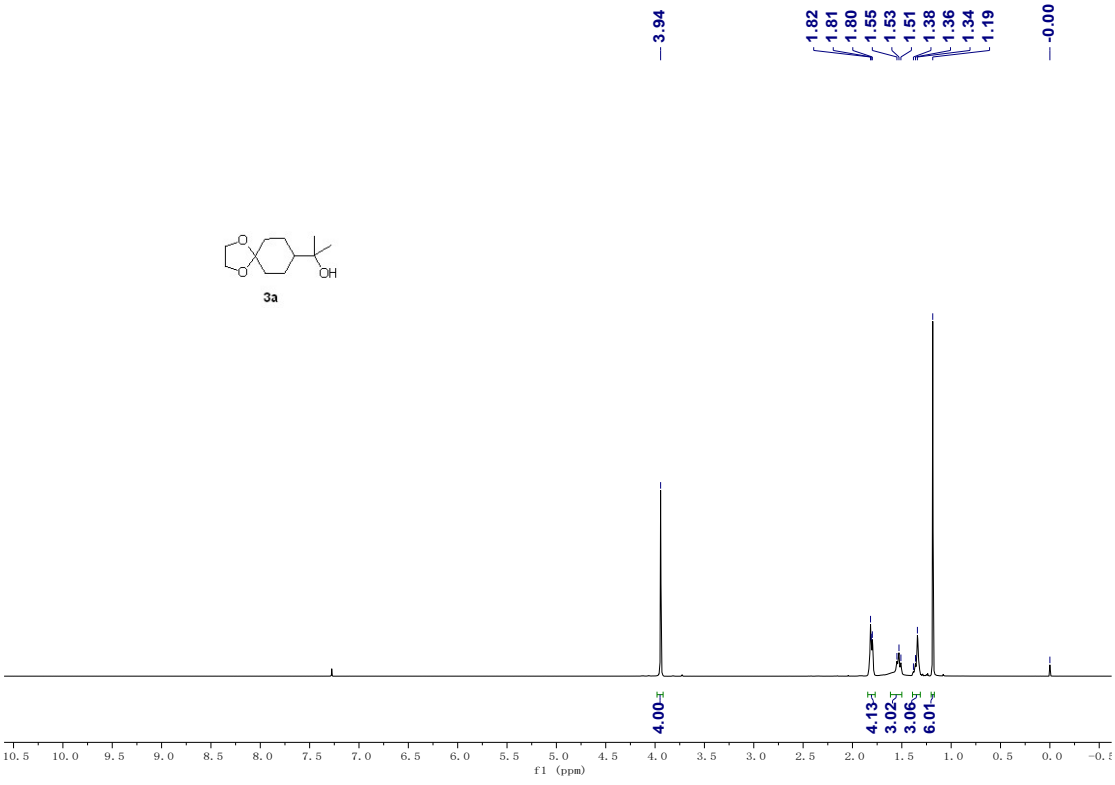
2.06
11.33
8.06
f1 (ppm)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1.53
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O
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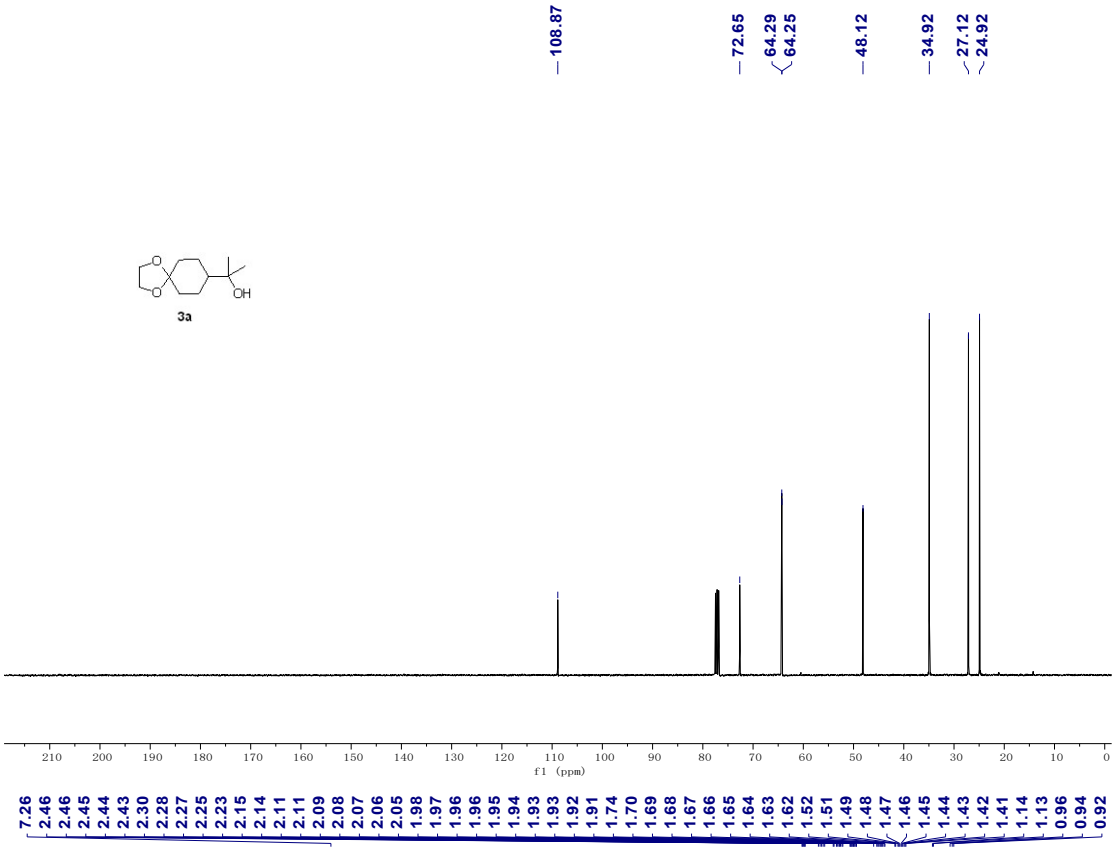
OH
3a
4.00
4.13
3.02
3.06
6.01
f1 (ppm)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27.12
24.92
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3a
f1 (ppm)
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2.46
2.46
2.45
2.44
2.43
2.30
2.28
2.27
2.25
2.23
2.15
2.14
2.11
2.11
2.09
2.08
2.07
2.06
2.05
1.98
1.97
1.96
1.96
1.95
1.94
1.93
1.93
1.92
1.91
1.74
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.62
1.52
1.51
1.49
1.48
1.47
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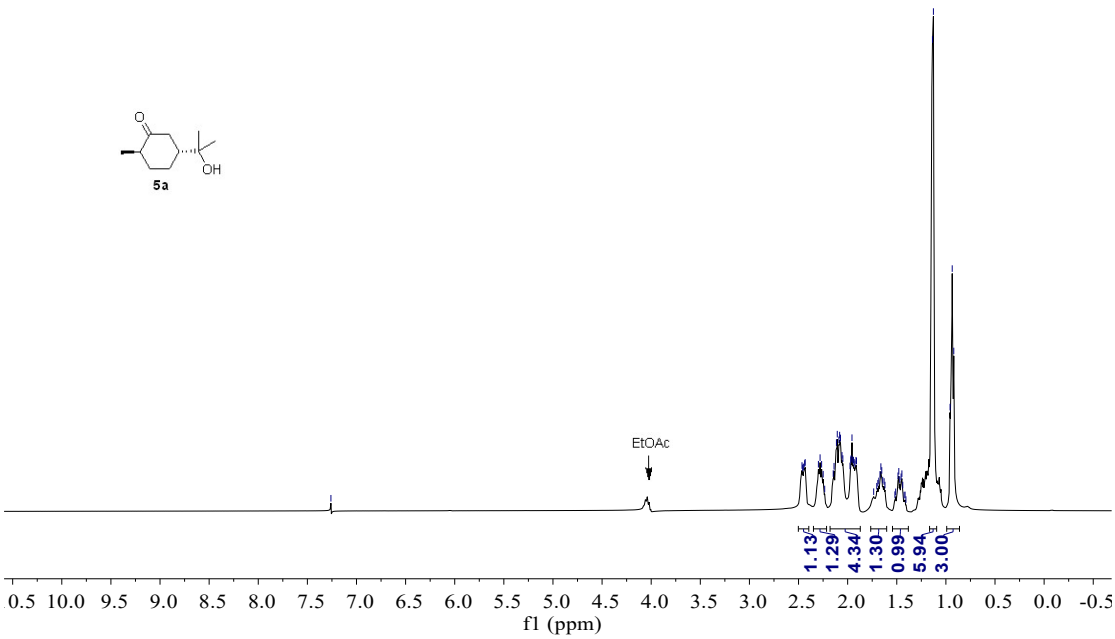
1.45
1.44
1.43
1.42
1.41
1.14
1.13
0.96
0.94
0.92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1.30
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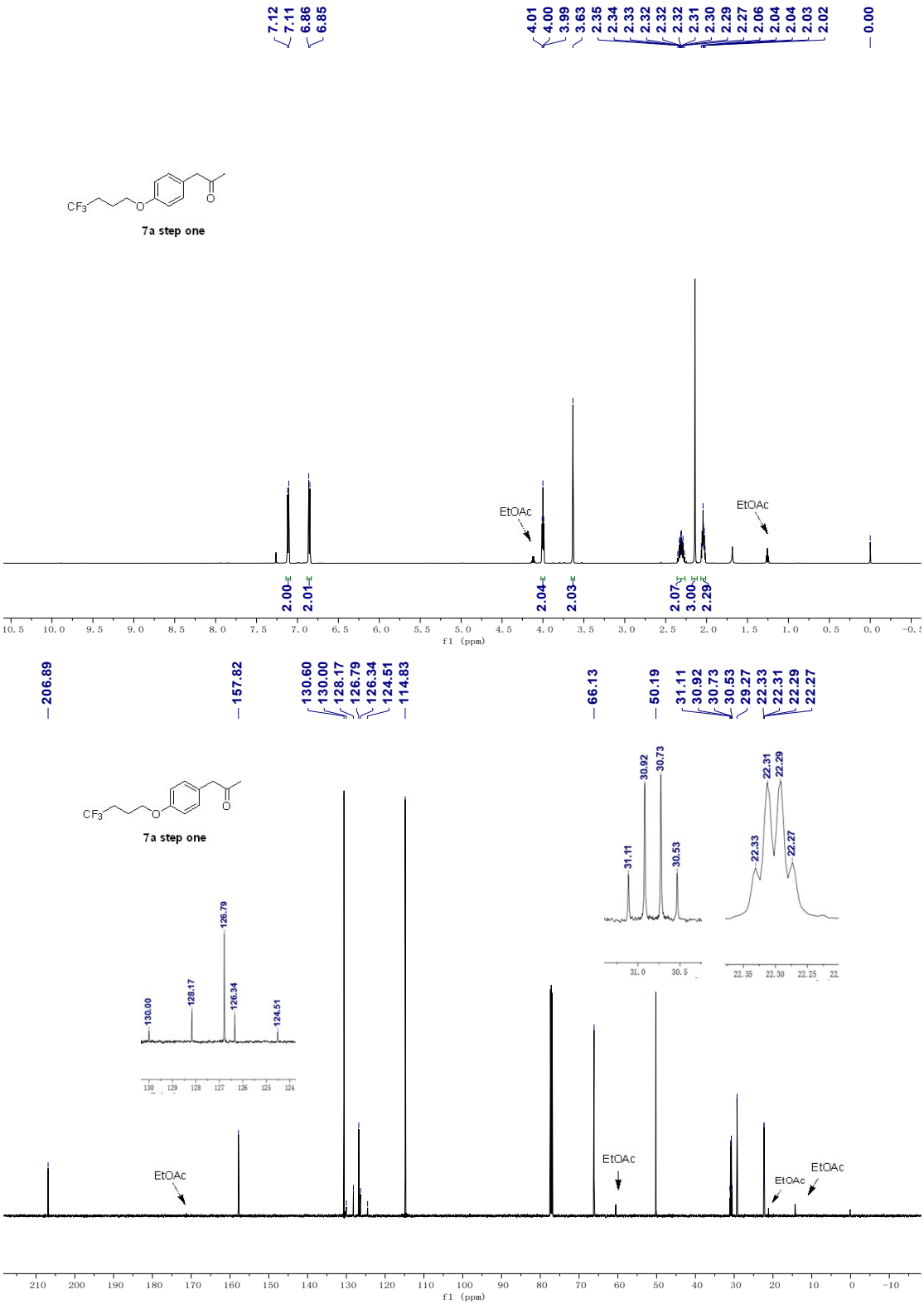
f1 (ppm)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2.34
2.33
2.32
2.32
2.32
2.31
2.30
2.29
2.27
2.06
2.04
2.04
2.03
2.02
0.00
CF3
O
O
7a step one
EtOAc
EtOAc
2.00
2.01
2.04
2.03
2.07
3.00
2.29
f1 (ppm)
206.89
157.82
130.60
130.00
128.17
126.79
126.34
124.51
114.83
66.13
50.19
31.11
30.92
30.73
30.53
29.27
22.33
22.31
22.29
22.27
CF3
O
O
7a step one
126.79
128.17
126.34
130.00
124.51
30.92
30.73
31.11
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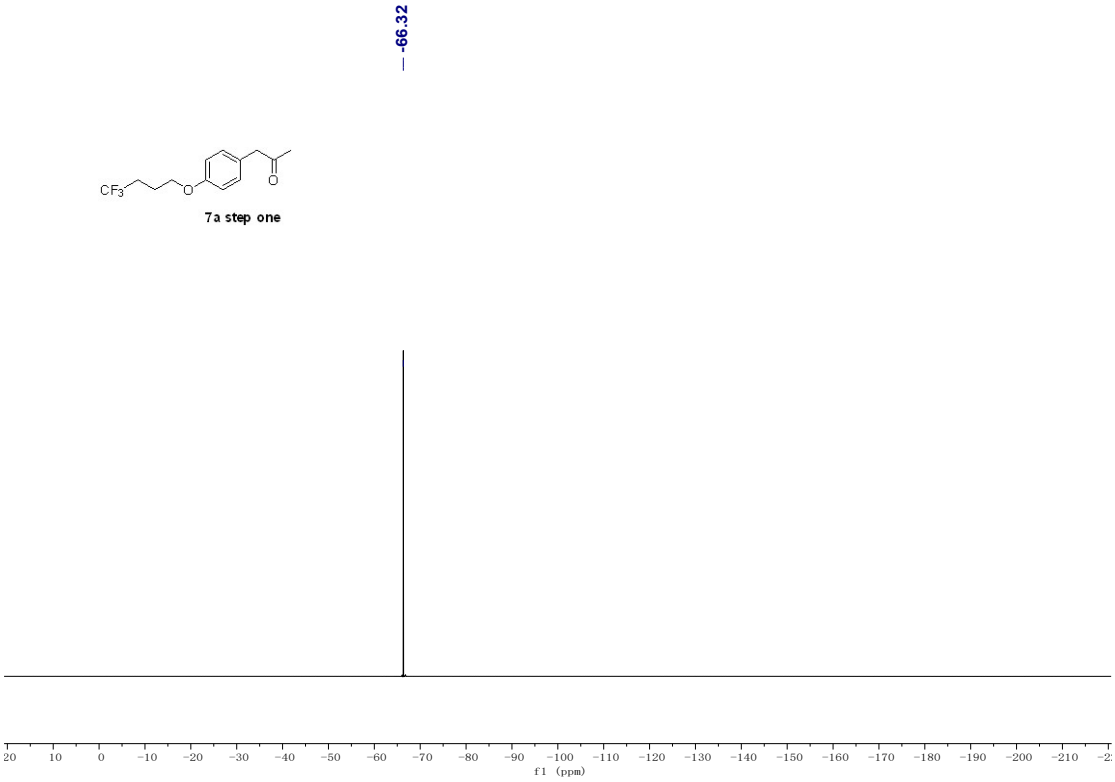
22.31
22.29
22.33
22.27
EtOAc
EtOAc
EtOAc
EtOAc
f1 (ppm)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O
7a step one
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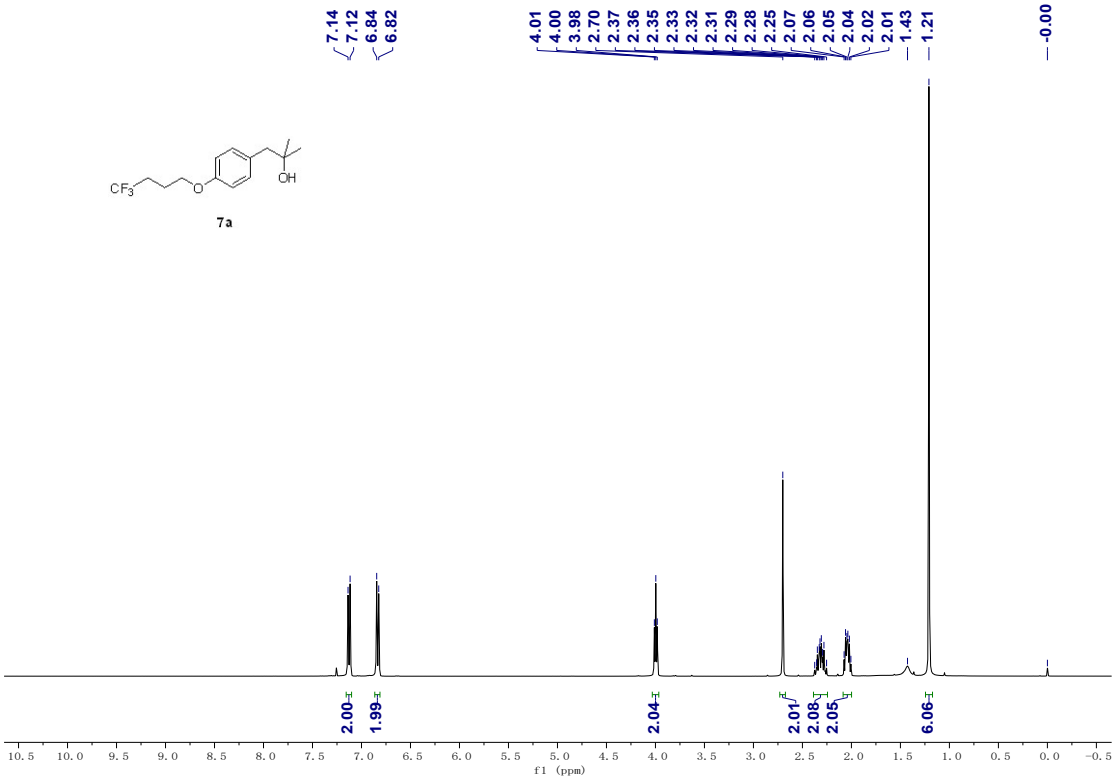
f1 (ppm)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2.35
2.33
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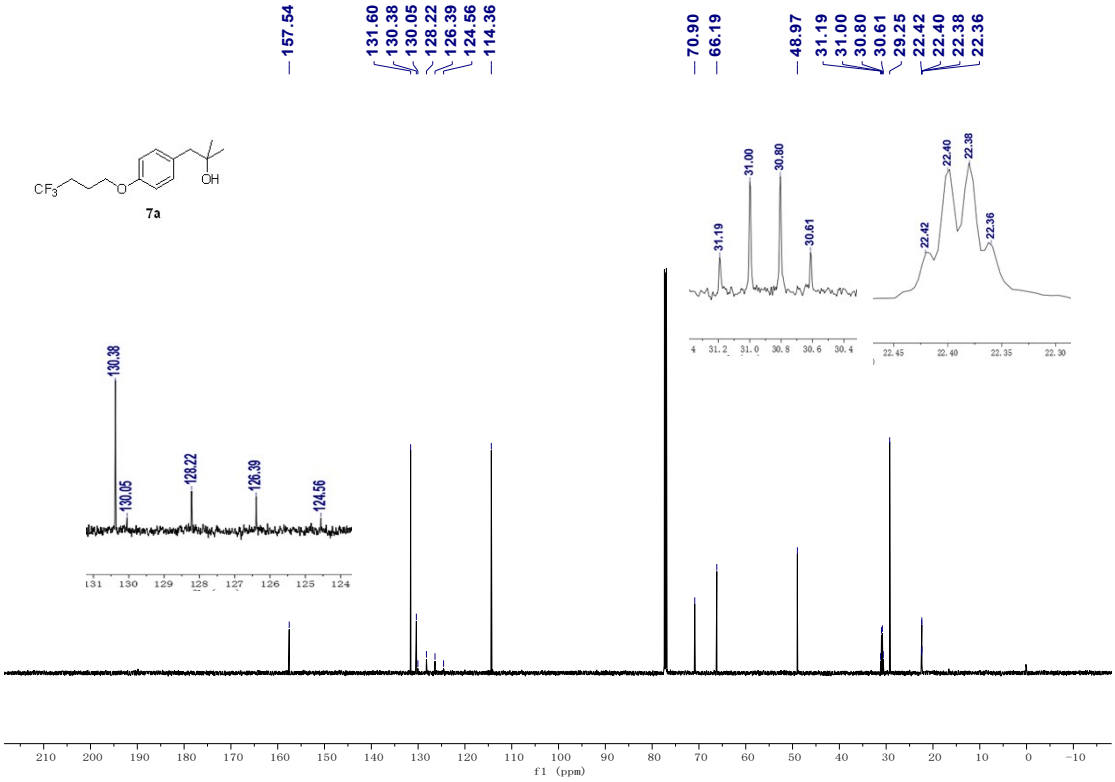
2.31
2.29
2.28
2.25
2.07
2.06
2.05
2.04
2.02
2.01
1.43
1.21
-0.00
CF3
O
OH
7a
2.00
1.99
2.04
2.01
2.08
2.05
6.06
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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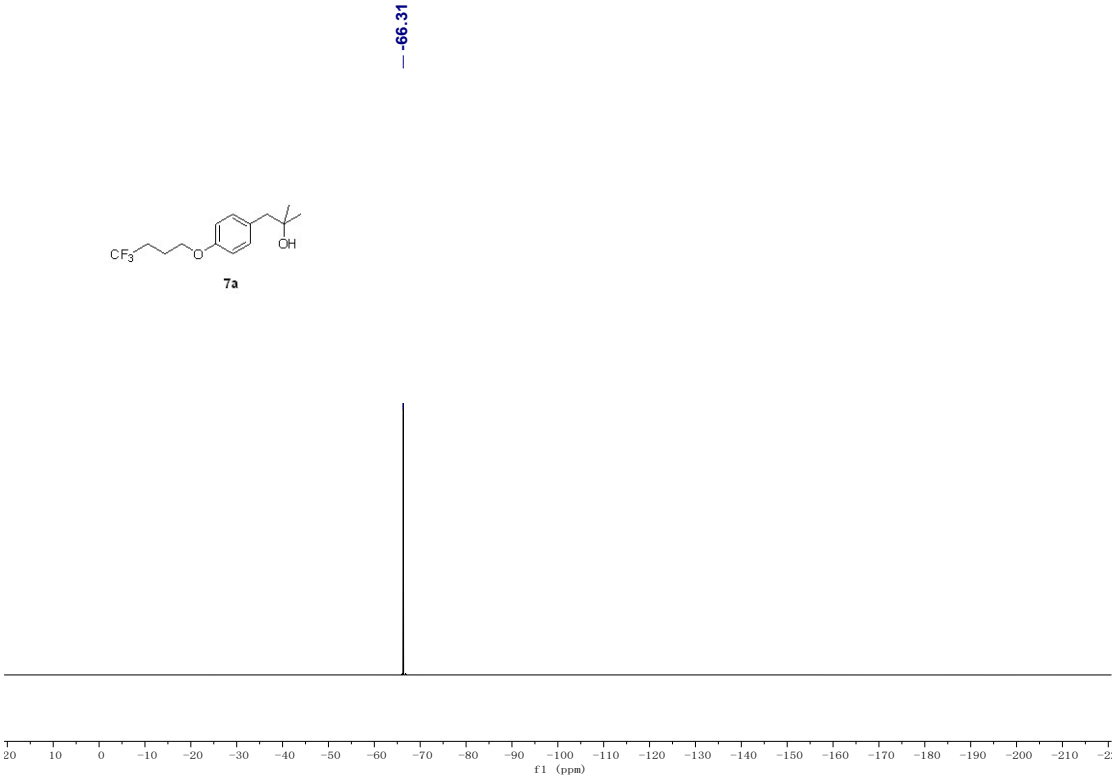
22.40
22.38
22.36
CF3
O
OH
7a
f1 (ppm)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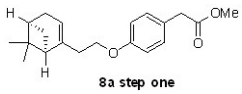

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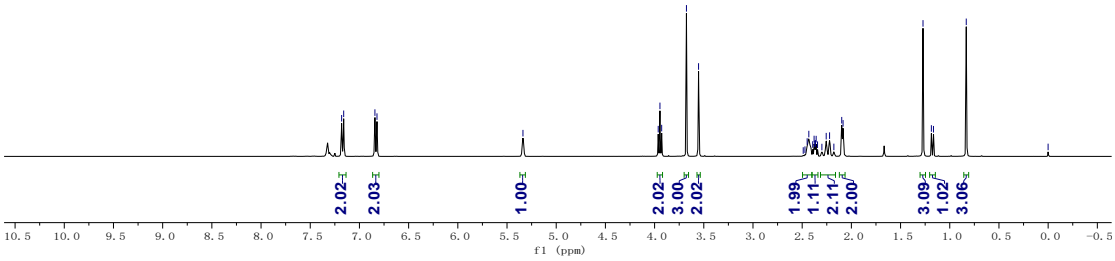

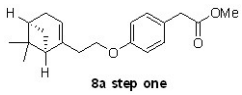

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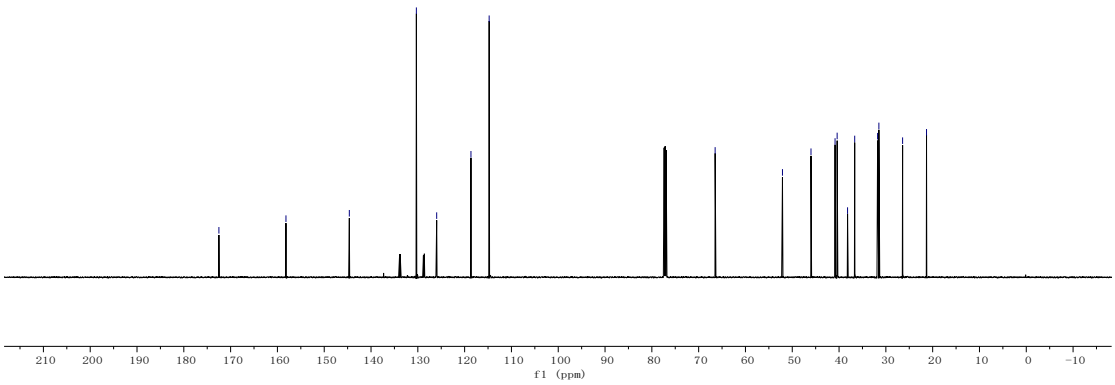

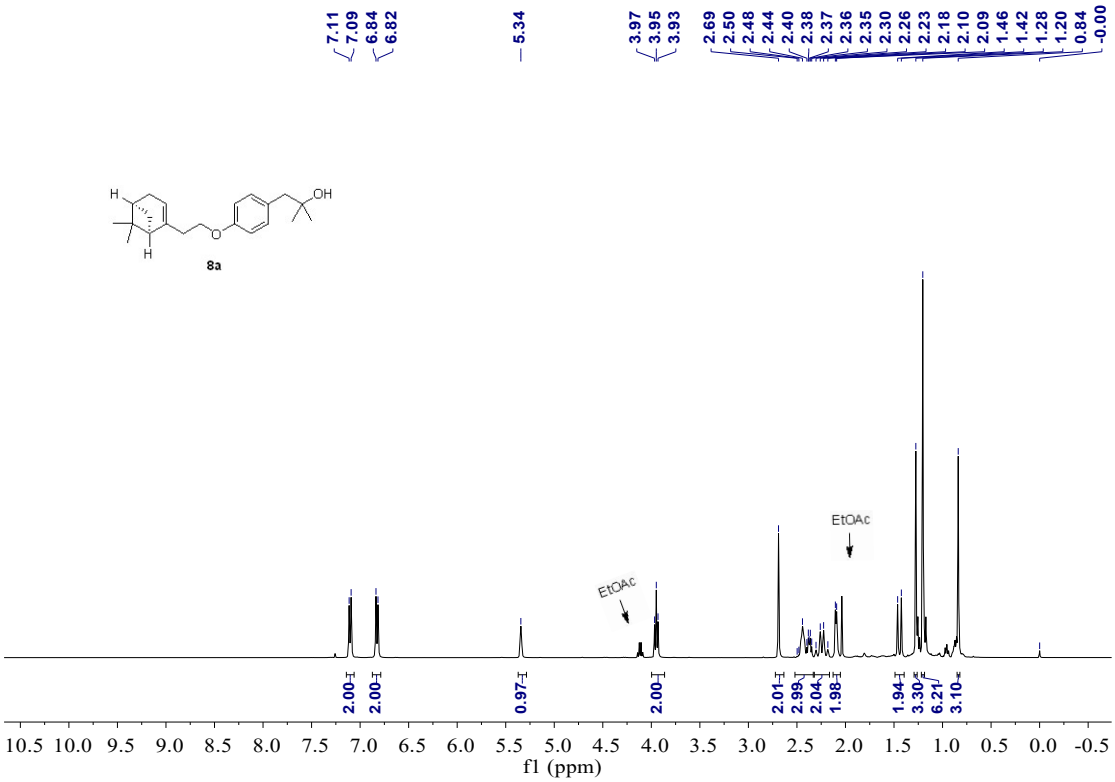
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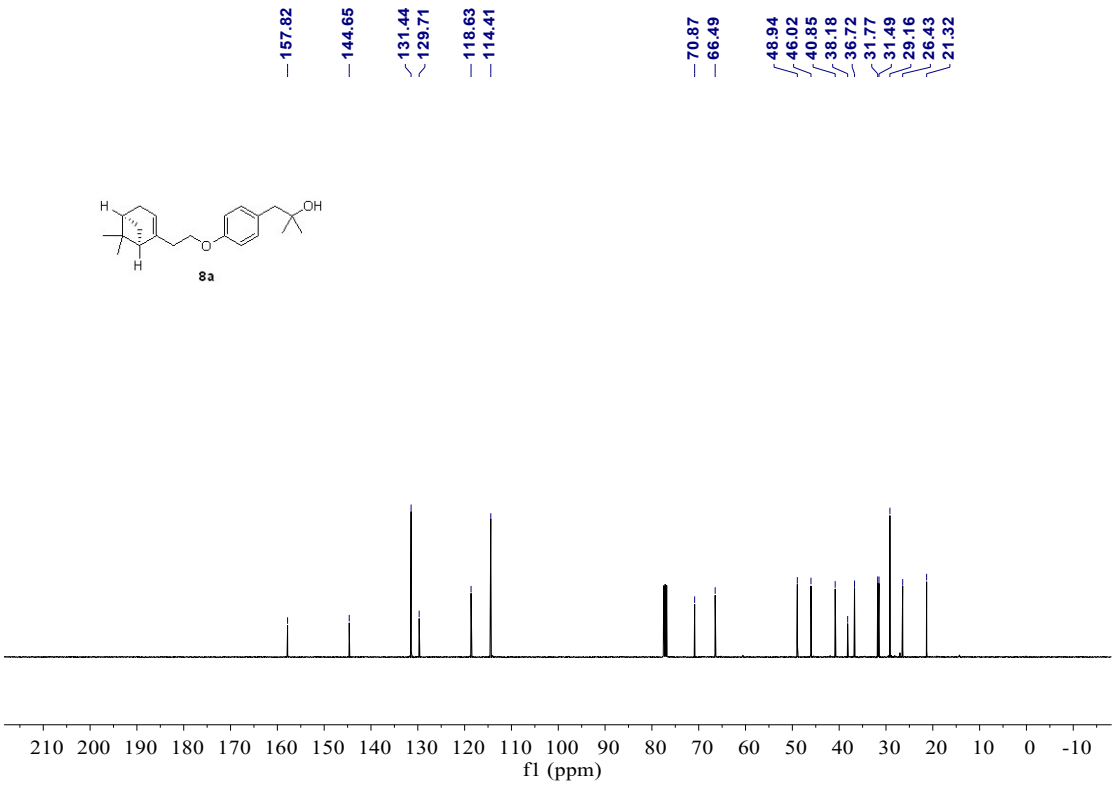
2.40
2.38
2.37
2.36
2.35
2.30
2.26
2.23
2.18
2.10
2.09
1.46
1.42
1.28
1.20
0.84
-0.00
OH
H
H
O
8a
EtOAc
EtOAc
2.00
2.00
0.97
2.00
2.01
2.99
2.04
1.98
1.94
3.30
6.21
3.10
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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46.02
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36.72
31.77
31.49
29.16
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OH
H
H
O
8a
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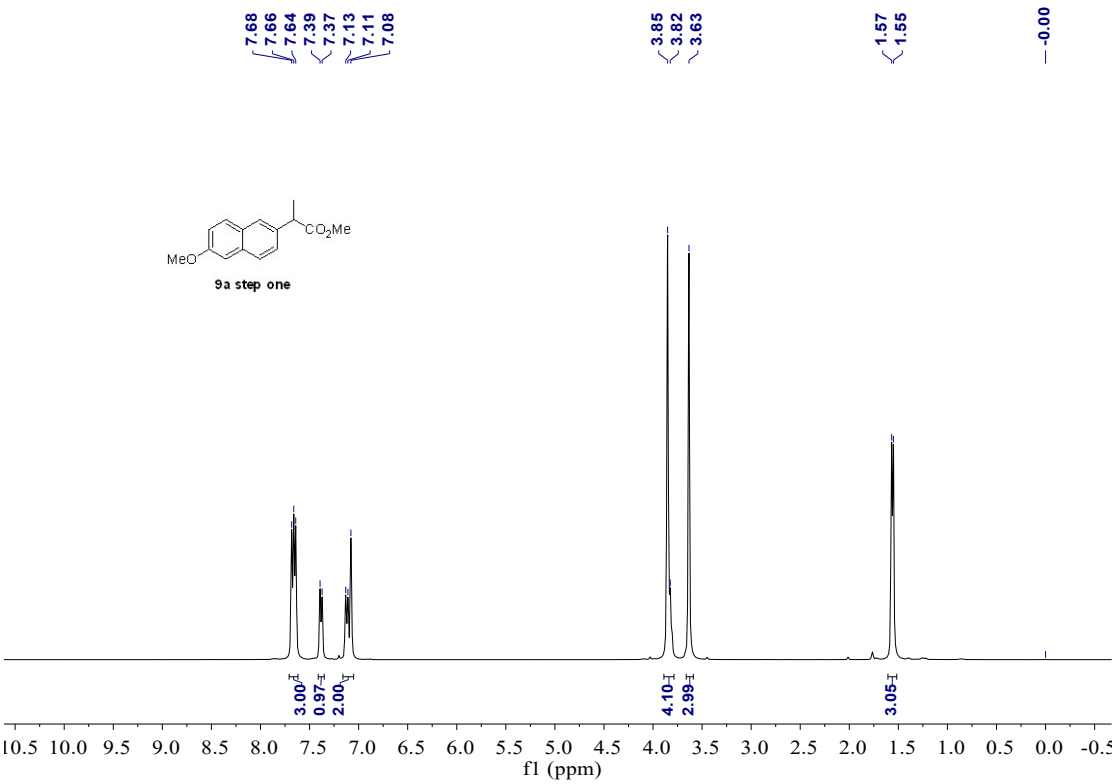
f1 (ppm)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7.08
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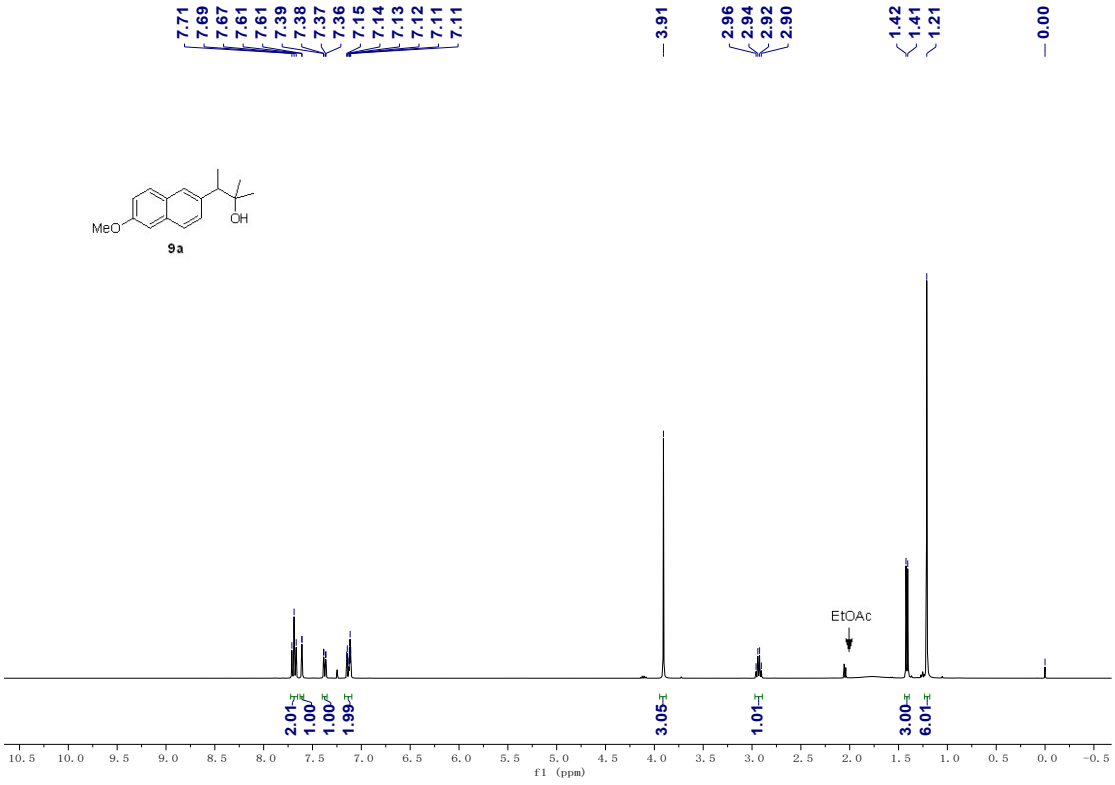
3.82
3.63
1.57
1.55
-0.00
CO2Me
MeO
9a step one
3.00
0.97
2.00
4.10
2.99
3.05
f1 (ppm)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7.14
7.13
7.12
7.11
7.11
3.91
2.96
2.94
2.92
2.90
1.42
1.41
1.21
0.00
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OH
9a
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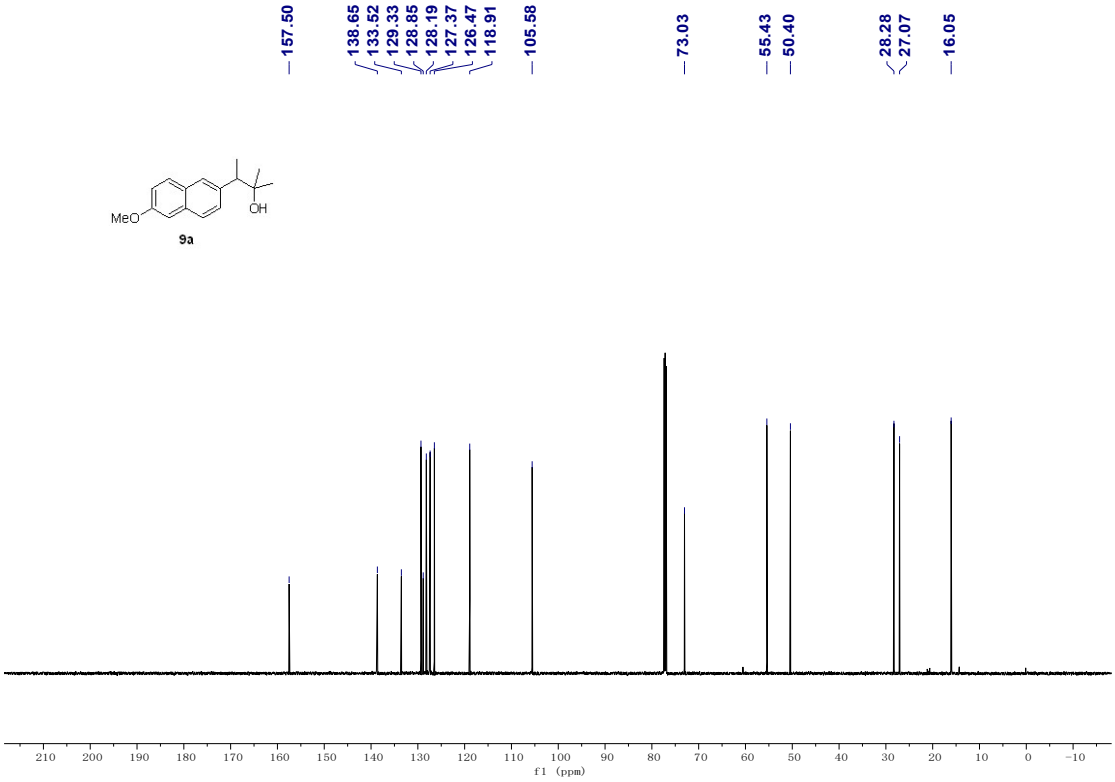
1.00
1.00
1.99
3.05
1.01
3.00
6.01
f1 (ppm)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50.40
28.28
27.07
16.05
MeO
OH
9a
f1 (ppm)
6.81
6.79
6.72
6.72
6.68
6.68
6.66
6.66
4.24
4.00
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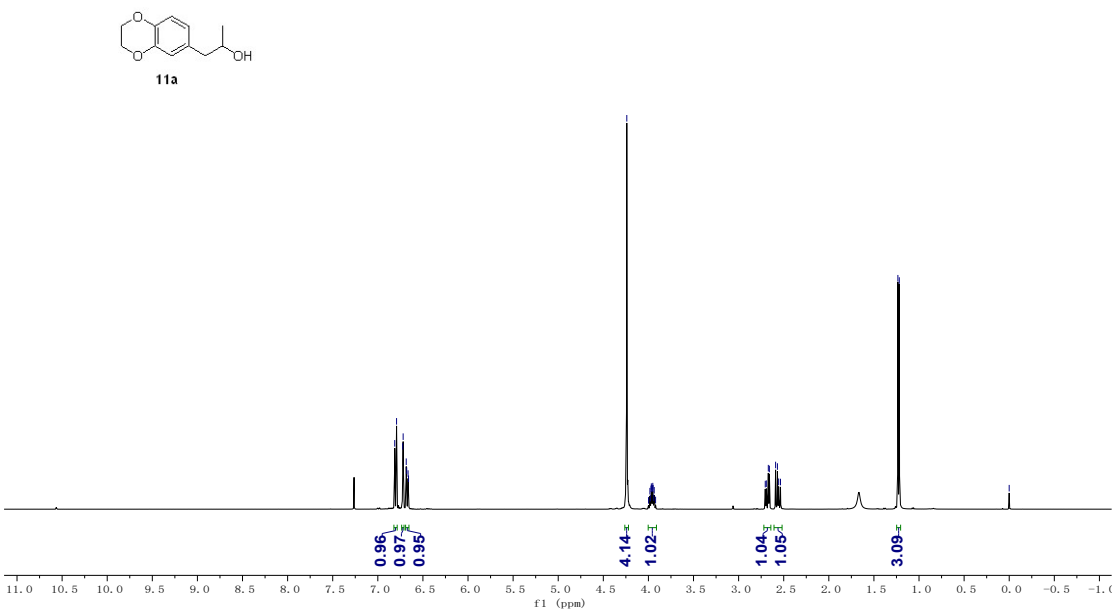
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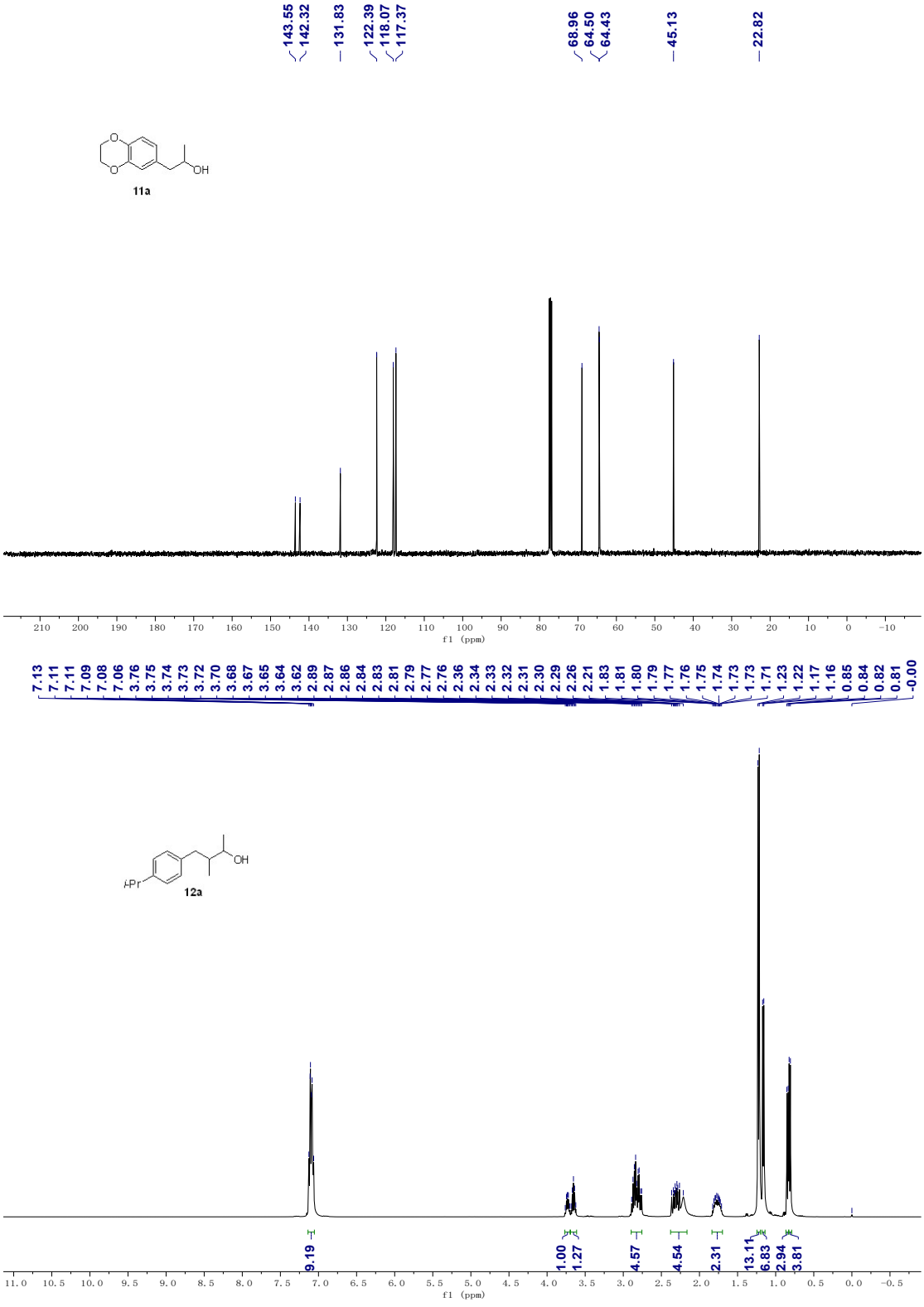
f1 (ppm)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OH
11a
f1 (ppm)
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7.11
7.11
7.09
7.08
7.06
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3.72
3.70
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3.62
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2.84
2.83
2.81
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2.76
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2.32
2.31
2.30
2.29
2.26
2.21
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1.80
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1.75
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1.73
1.71
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0.82
0.81
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OH
iPr
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13.11
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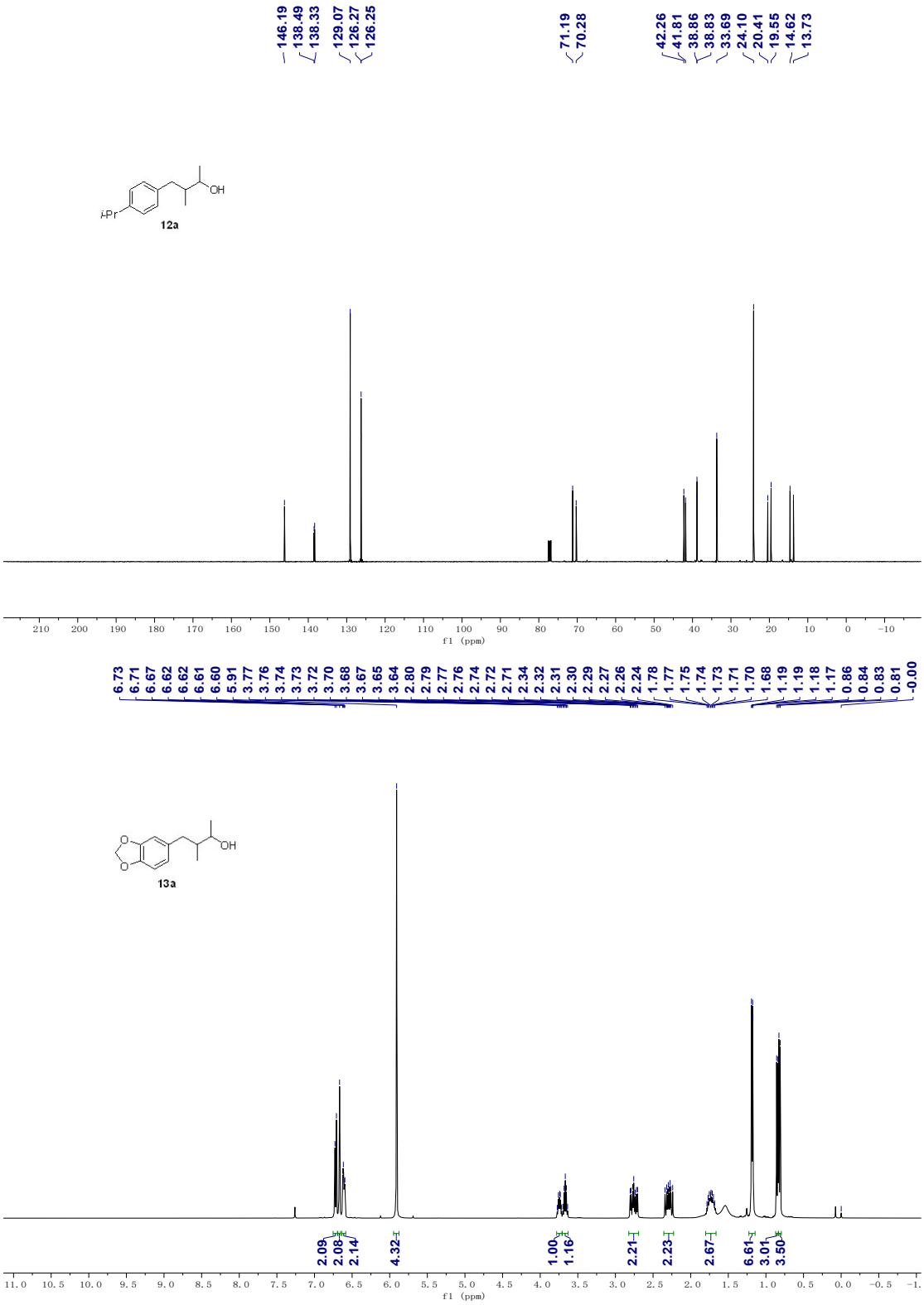
3.81
f1 (ppm)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24.10
20.41
19.55
14.62
13.73
i-Pr
OH
12a
f1 (ppm)
6.73
6.71
6.67
6.62
6.62
6.61
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5.91
3.77
3.76
3.74
3.73
3.72
3.70
3.68
3.67
3.65
3.64
2.80
2.79
2.77
2.76
2.74
2.72
2.71
2.34
2.32
2.31
2.30
2.29
2.27
2.26
2.24
1.78
1.77
1.75
1.74
1.73
1.71
1.70
1.68
1.19
1.19
1.18
1.17
0.86
0.84
0.83
0.81
-0.00
OH
13a
2.09
2.08
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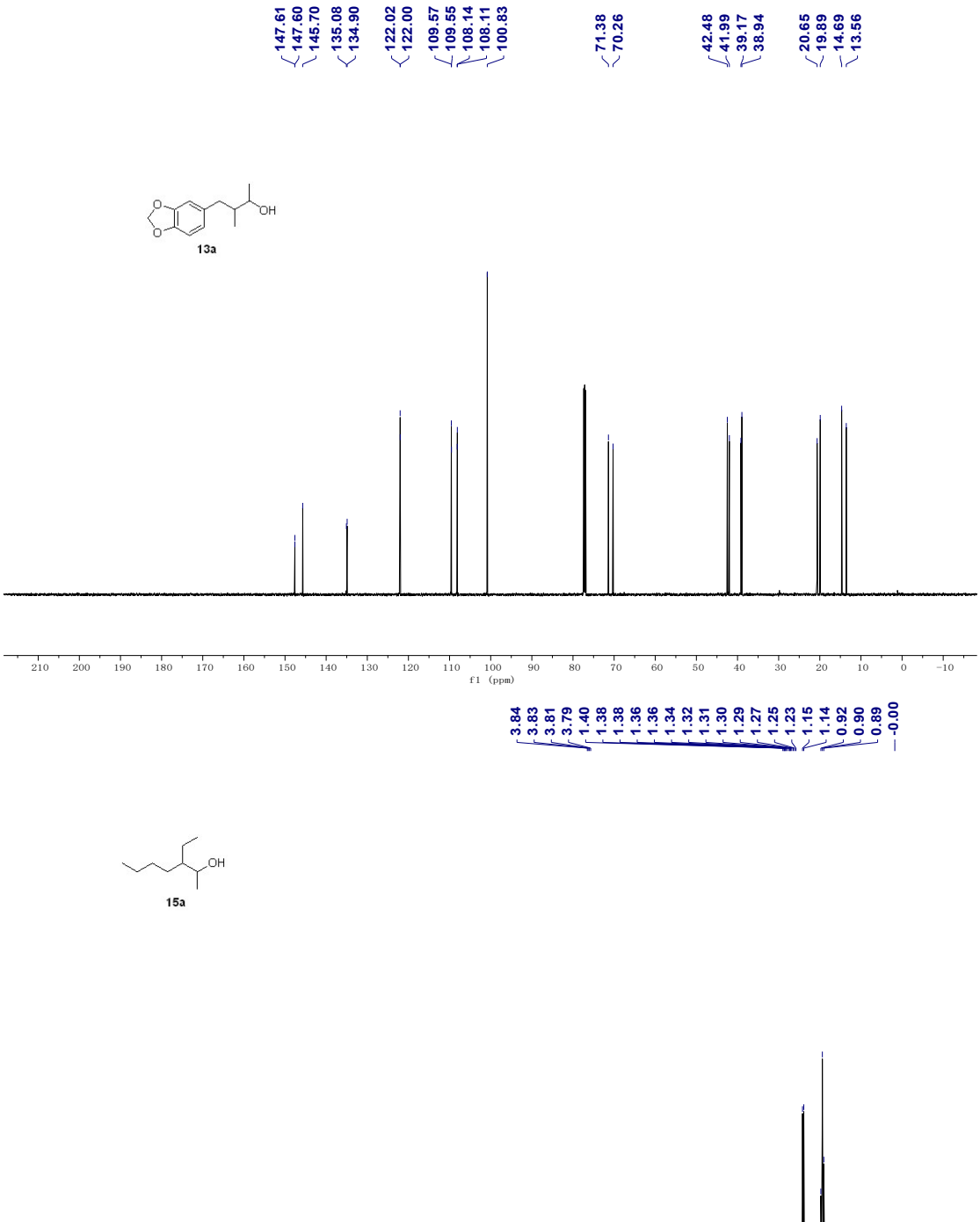
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108.14
108.11
100.83
71.38
70.26
42.48
41.99
39.17
38.94
20.65
19.89
14.69
13.56
OH
13a
f1 (ppm)
3.84
3.83
3.81
3.79
1.40
1.38
1.38
1.36
1.36
1.34
1.32
1.31
1.30
1.29
1.27
1.25
1.23
1.15
1.14
0.92
0.90
0.89
-0.00
OH
15a
1.00
9.03
3.10
6.07
f1 (ppm)


7.37
7.35
7.35
7.33
7.31
7.27
7.27
7.26
7.25
7.24
7.23
5.58
4.76
4.75
4.73
4.72
2.35
2.32
2.30
2.28
2.26
2.25
2.08
2.03
2.03
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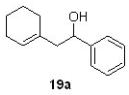
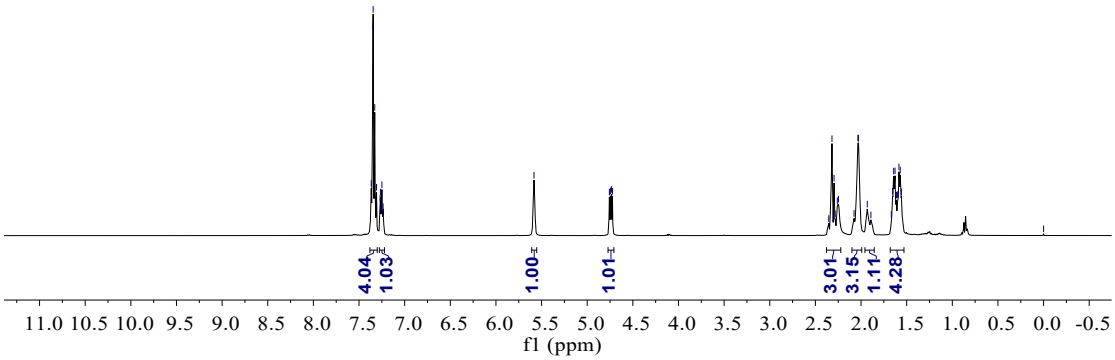

OH

19a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3.01
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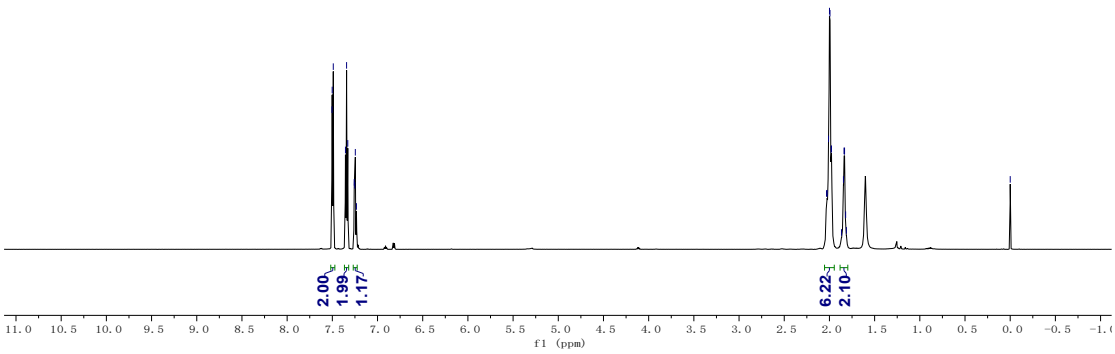
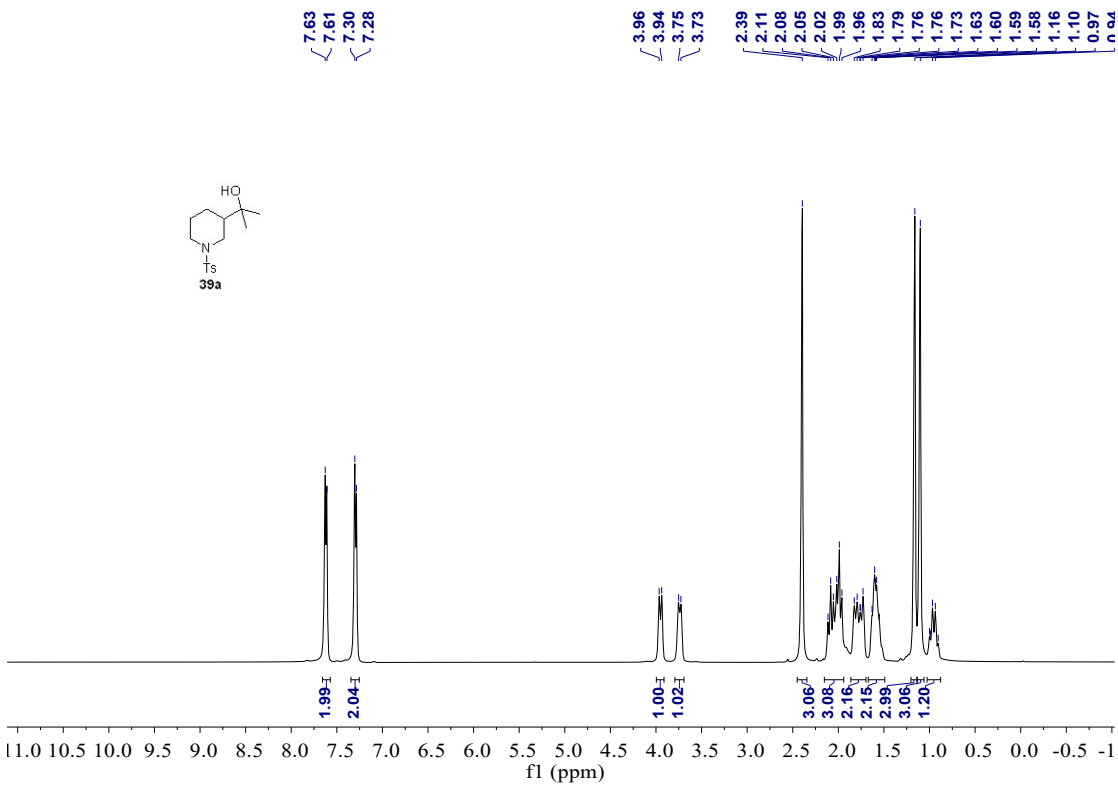
4.28
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

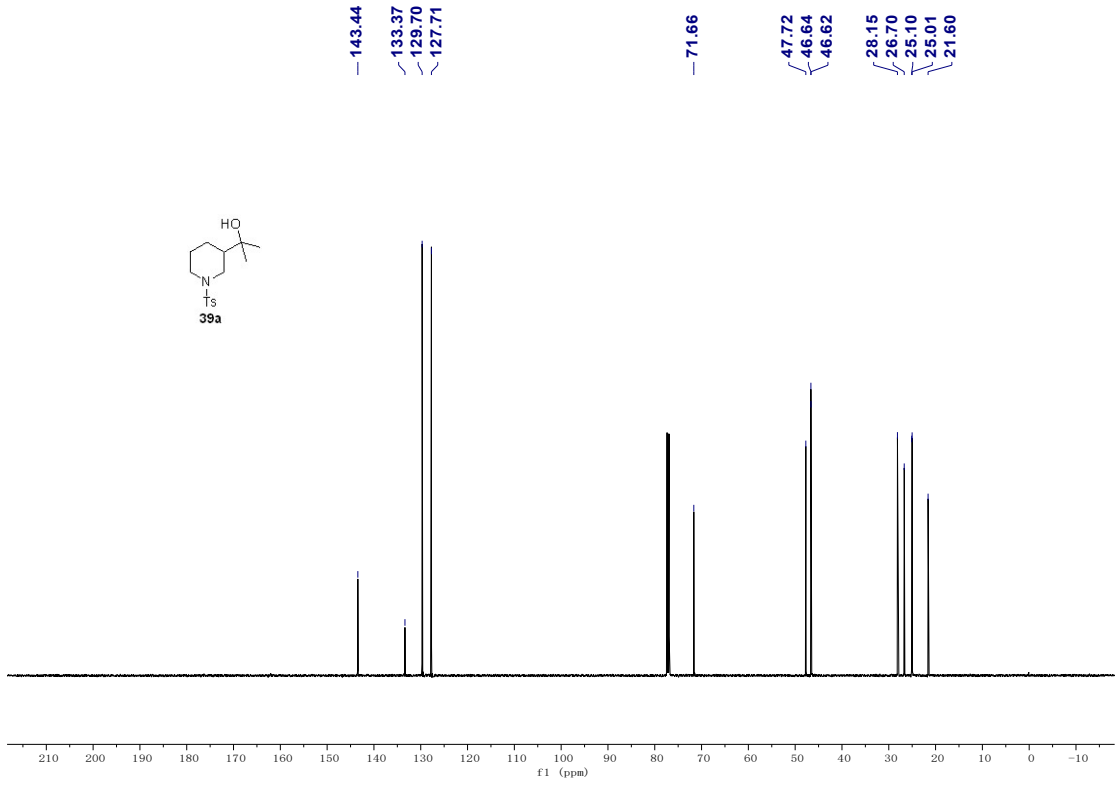

7.50
7.50
7.49
7.35
7.34
7.33
7.26
7.25
7.25
7.23
2.03
2.02
2.01
2.00
1.99
1.98
1.98
1.87
1.85
1.84
1.84
1.83
1.82
1.81
-0.00

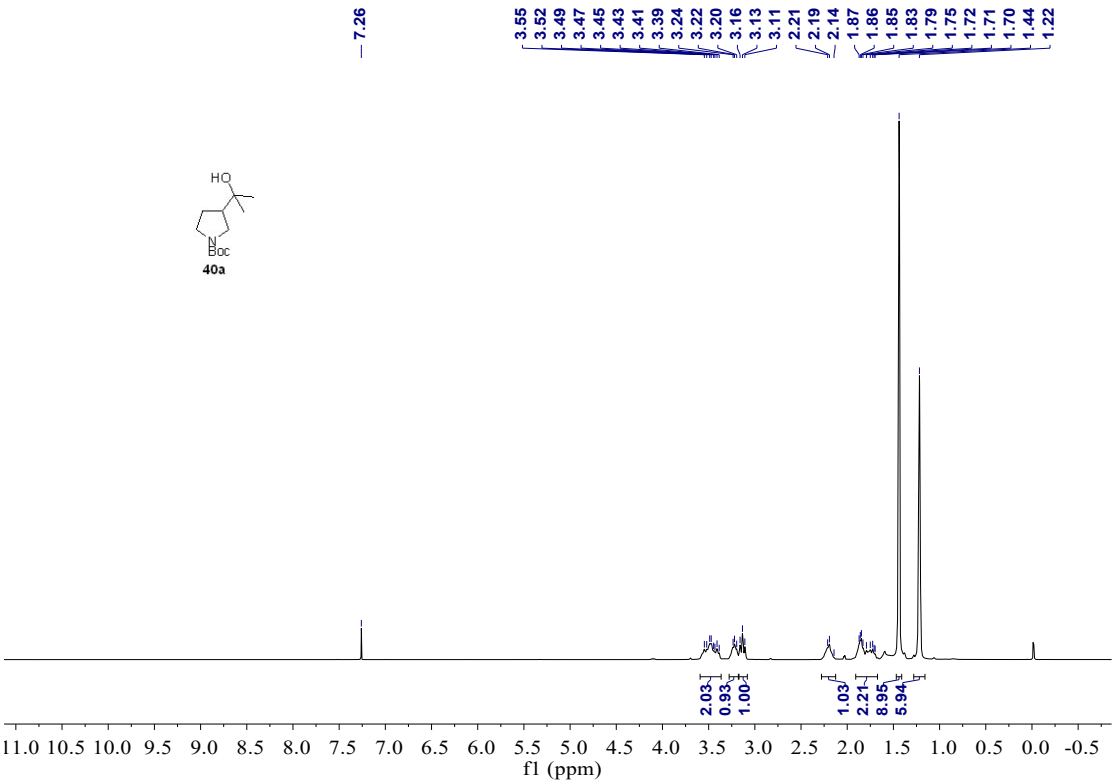

HO

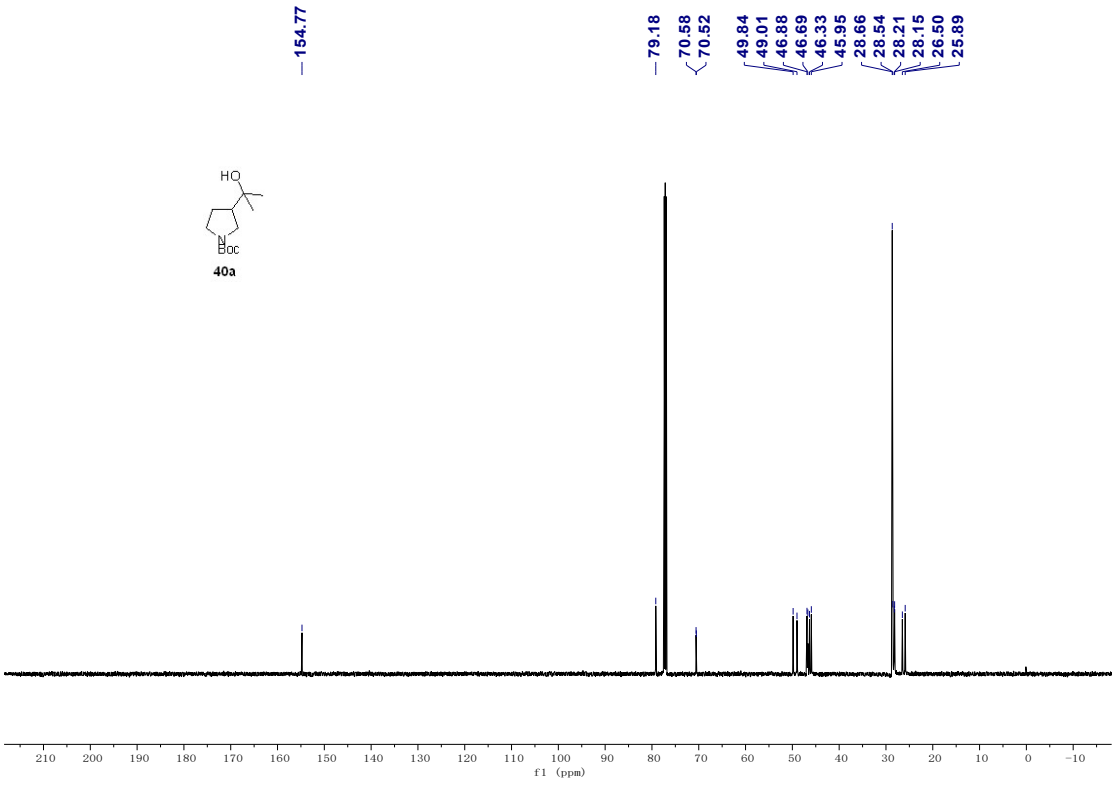
38a

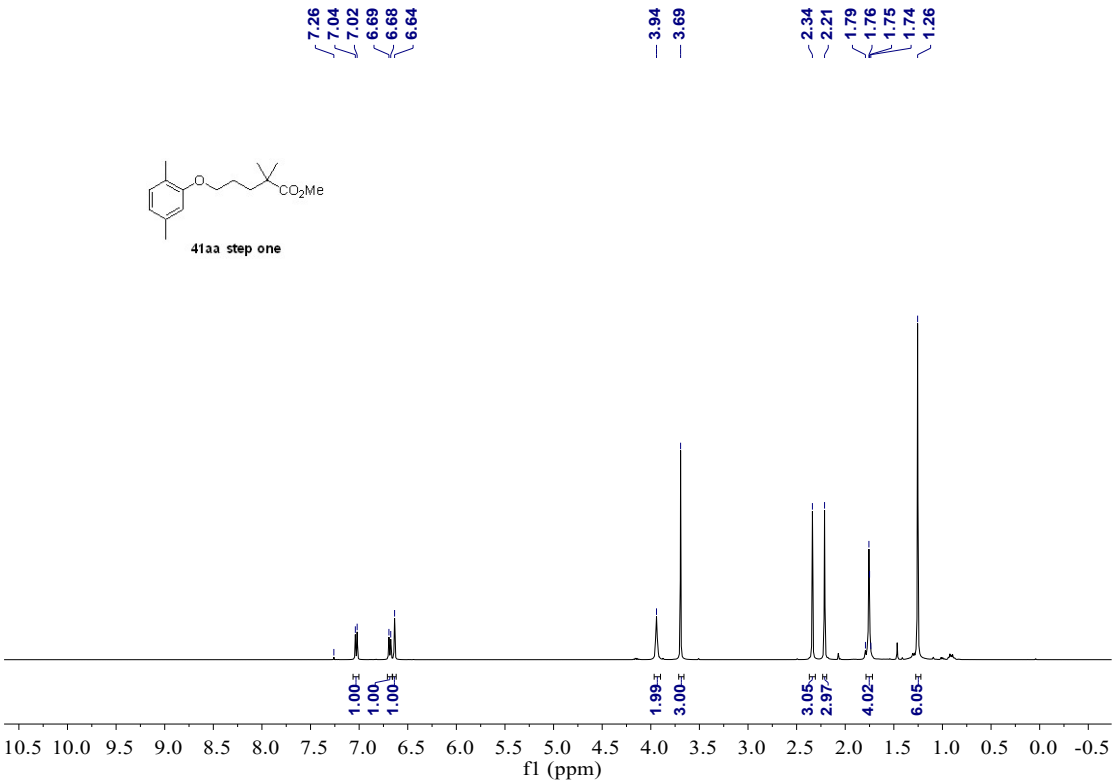

2.00
1.99
1.17
6.22
2.10
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

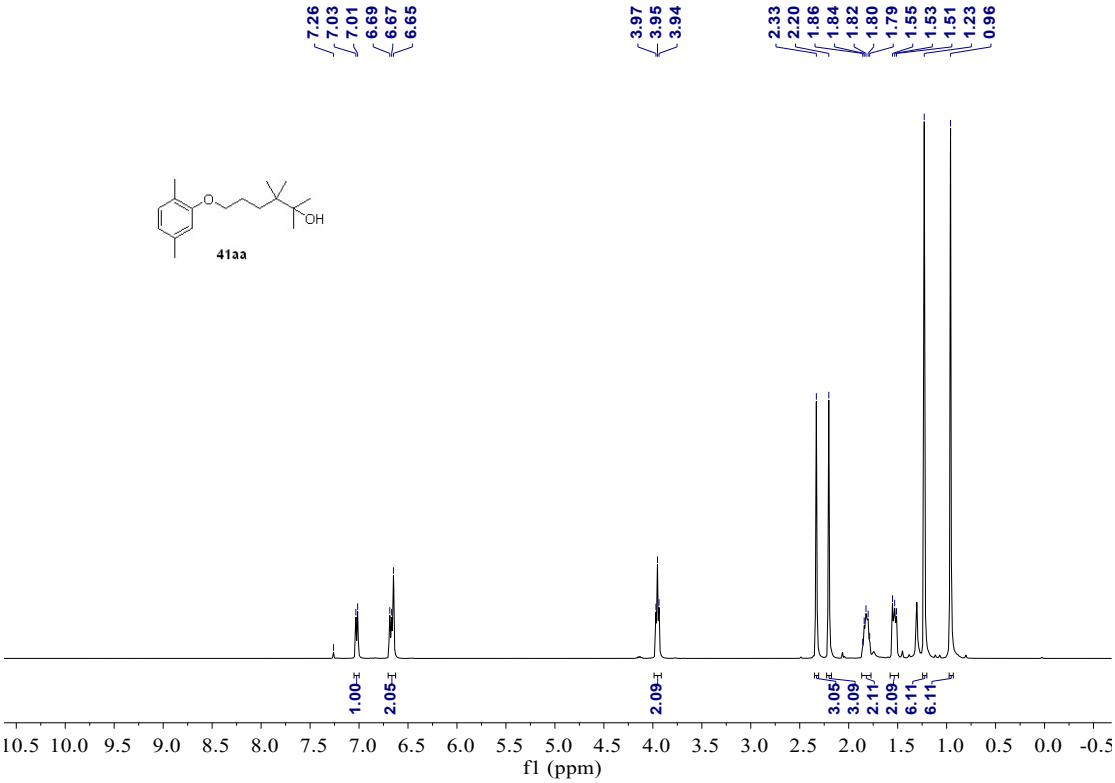

7.63
7.61
7.30
7.28
3.96
3.94
3.75
3.73
2.39
2.11
2.08
2.05
2.02
1.99
1.96
1.83
1.79
1.76
1.76
1.73
1.63
1.60
1.59
1.58
1.16
1.10
0.97
HO
N
Ts
39a
1.99
2.04
1.00
1.02
3.06
3.08
2.15
2.15
2.99
3.06
1.20
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

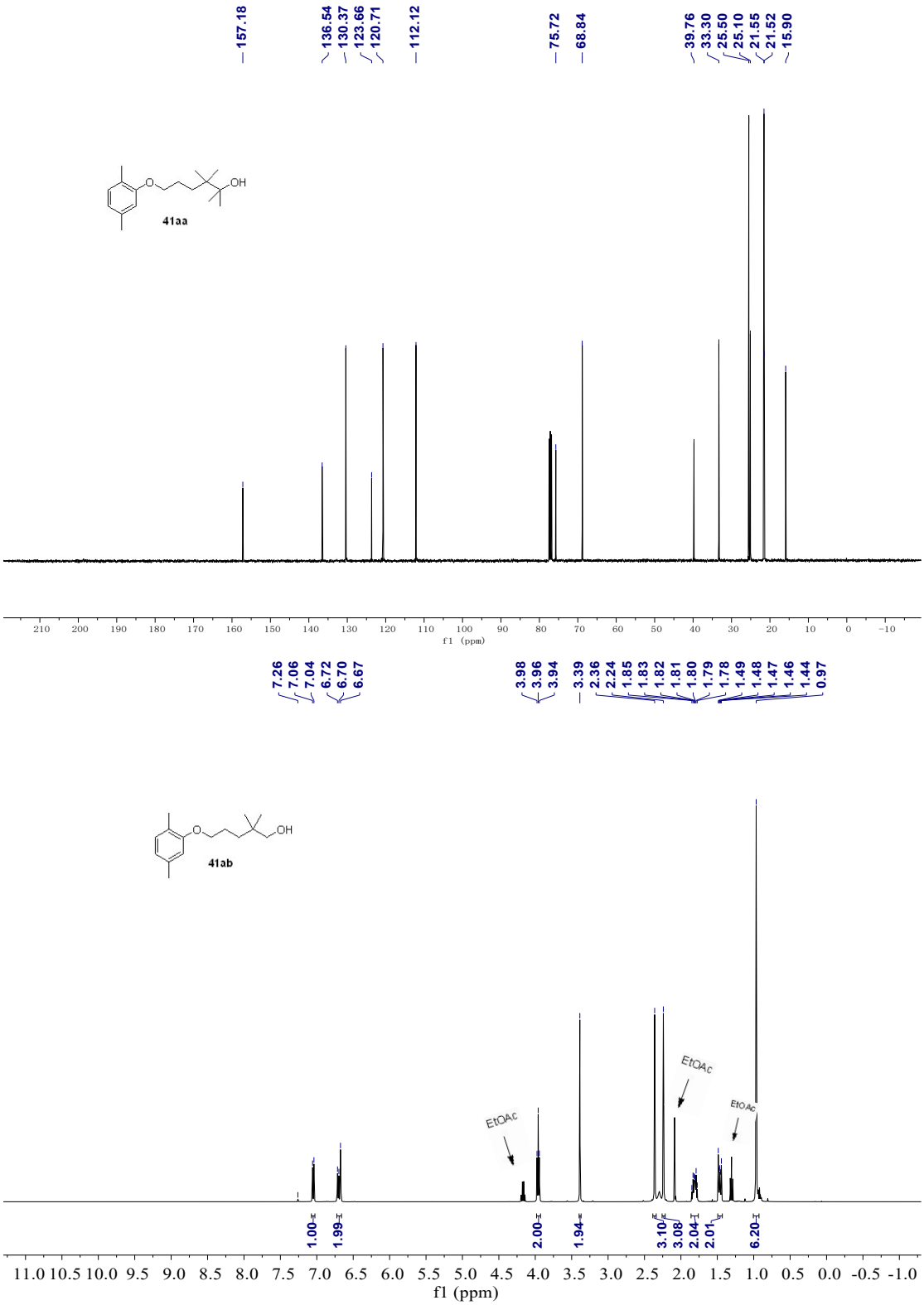

143.44
133.37
129.70
127.71
71.66
47.72
46.64
46.62
28.15
26.70
25.10
25.01
21.60
HO
N
Ts
39a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

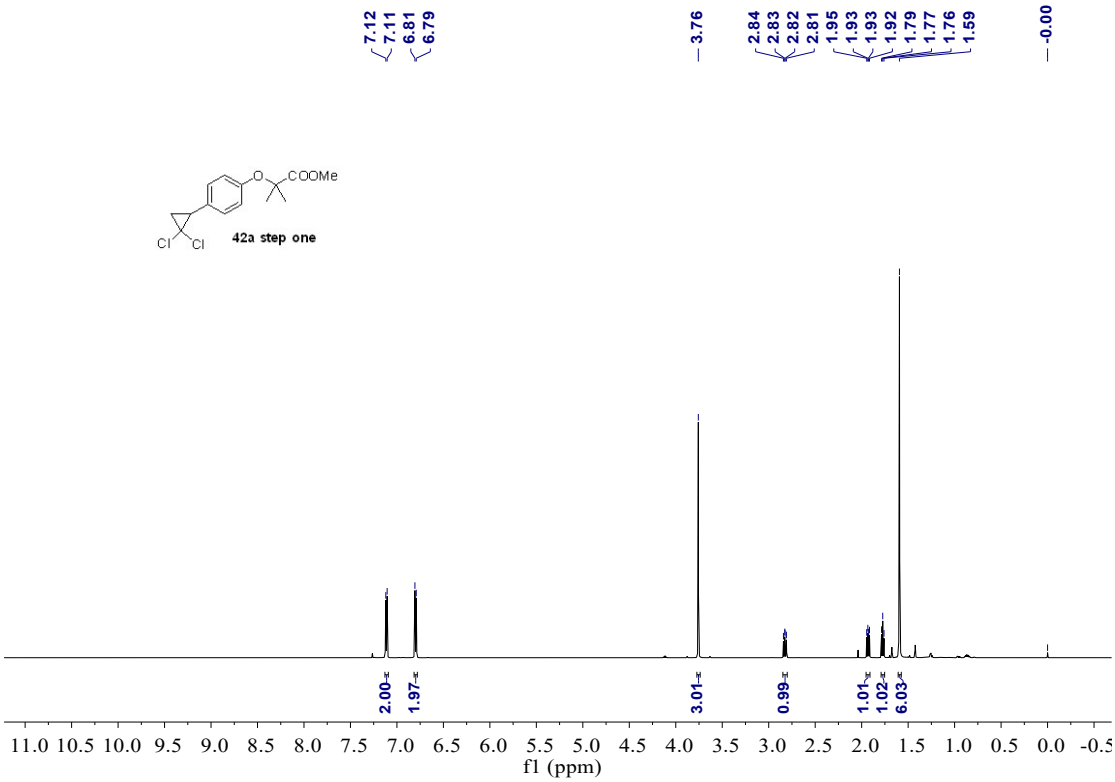

7.26
3.55
3.52
3.49
3.47
3.45
3.43
3.41
3.39
3.24
3.22
3.20
3.16
3.13
3.11
2.21
2.19
2.14
1.87
1.86
1.85
1.83
1.79
1.75
1.72
1.71
1.70
1.44
1.22
HO
N
Boc
40a
2.03
0.93
1.00
1.03
2.21
8.95
5.94
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

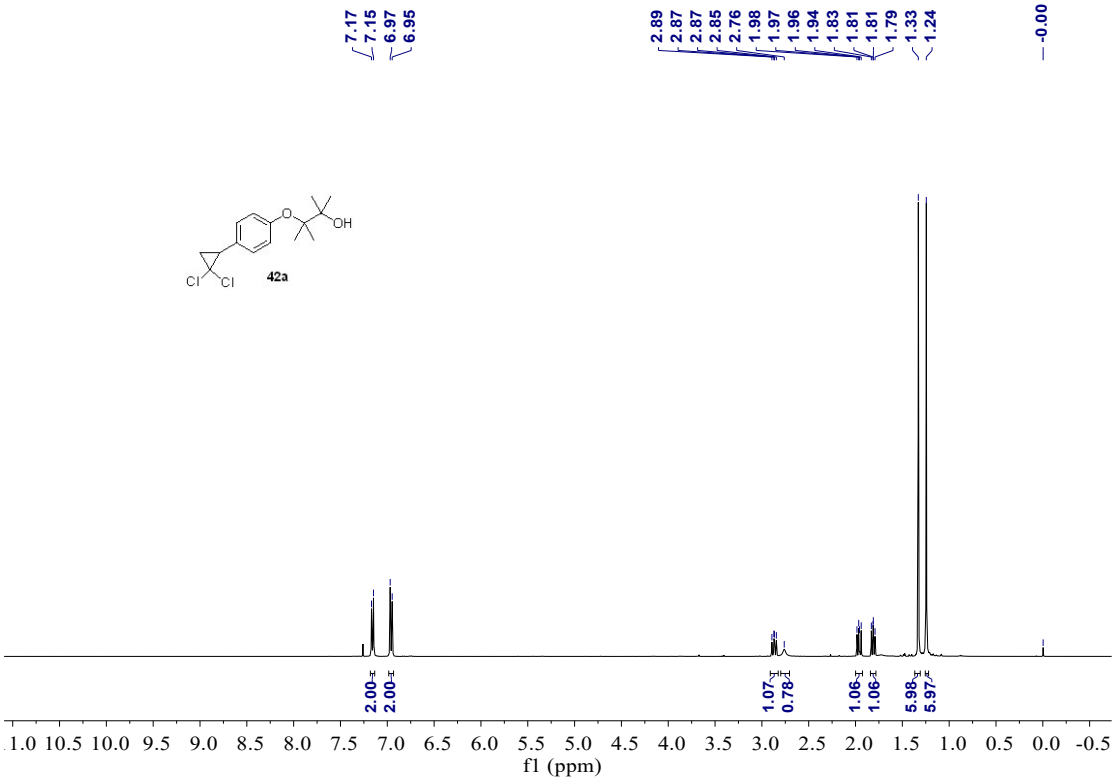

154.77
79.18
70.58
70.52
49.84
49.01
46.88
46.69
46.33
45.95
28.66
28.54
28.21
28.15
26.50
25.89
HO
N
Boc
40a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

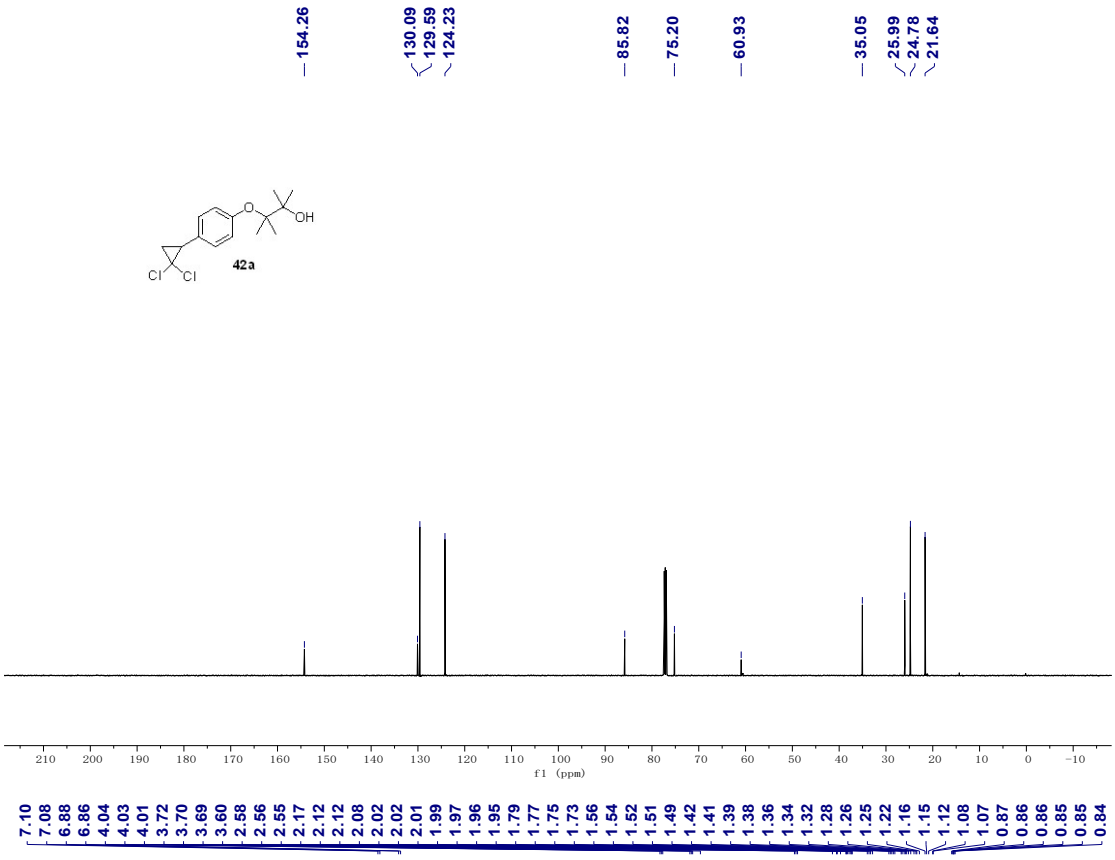

7.26
7.04
7.02
6.69
6.68
6.64
3.94
3.69
2.34
2.21
1.79
1.76
1.75
1.74
1.26
CO2Me
41aa step one
1.00
1.00
1.00
1.99
3.00
3.05
2.97
4.02
6.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

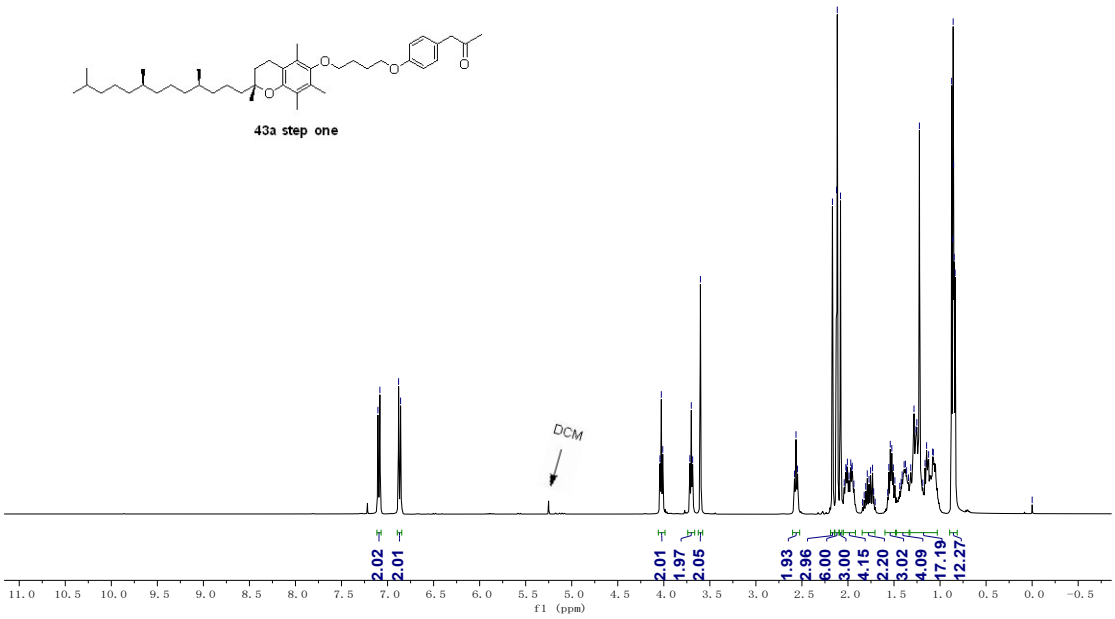

7.26
7.03
7.01
6.69
6.67
6.65
3.97
3.95
3.94
2.33
2.20
1.86
1.84
1.82
1.80
1.79
1.55
1.53
1.51
1.23
0.96
OH
41aa
1.00
2.05
2.09
3.05
3.09
2.11
2.09
6.11
6.11
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

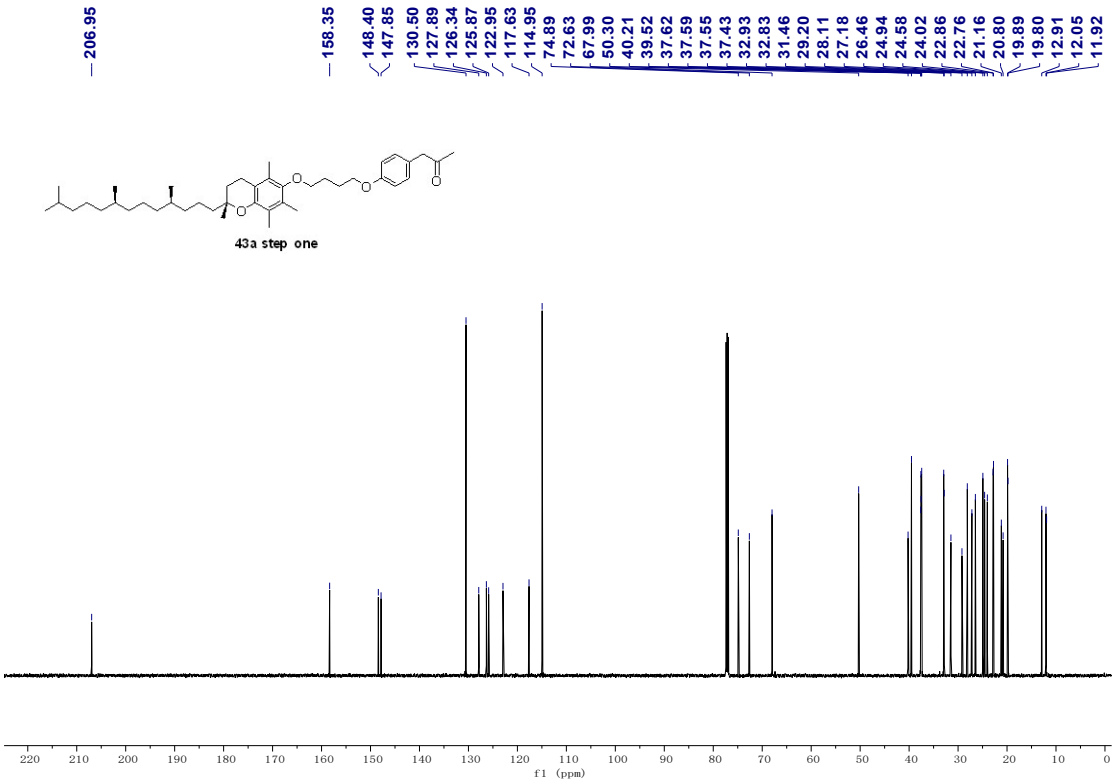

157.18
136.54
130.37
123.66
120.71
112.12
75.72
68.84
39.76
33.30
25.50
25.10
21.55
21.52
15.90
OH
41aa
f1 (ppm)
7.26
7.06
7.04
6.72
6.70
6.67
3.98
3.96
3.94
3.39
2.36
2.24
1.85
1.83
1.82
1.81
1.80
1.79
1.78
1.49
1.48
1.47
1.46
1.44
0.97
OH
41ab
EtOAc
EtOAc
EtOAc
1.00
1.99
2.00
1.94
3.10
3.08
2.04
2.01
6.20
f1 (ppm)

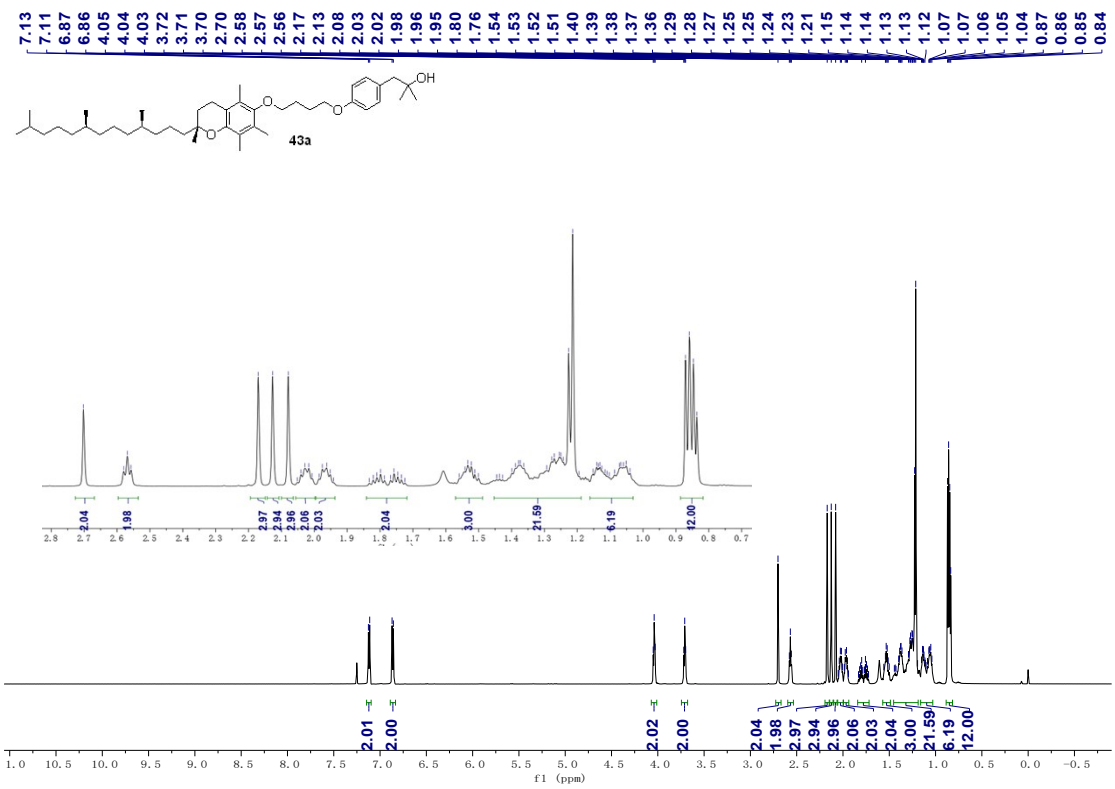

7.12
7.11
6.81
6.79
3.76
2.84
2.83
2.82
2.81
1.95
1.93
1.93
1.92
1.79
1.77
1.76
1.59
-0.00
COOMe
Cl
Cl
42a step one
2.00
1.97
3.01
0.99
1.01
1.02
6.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

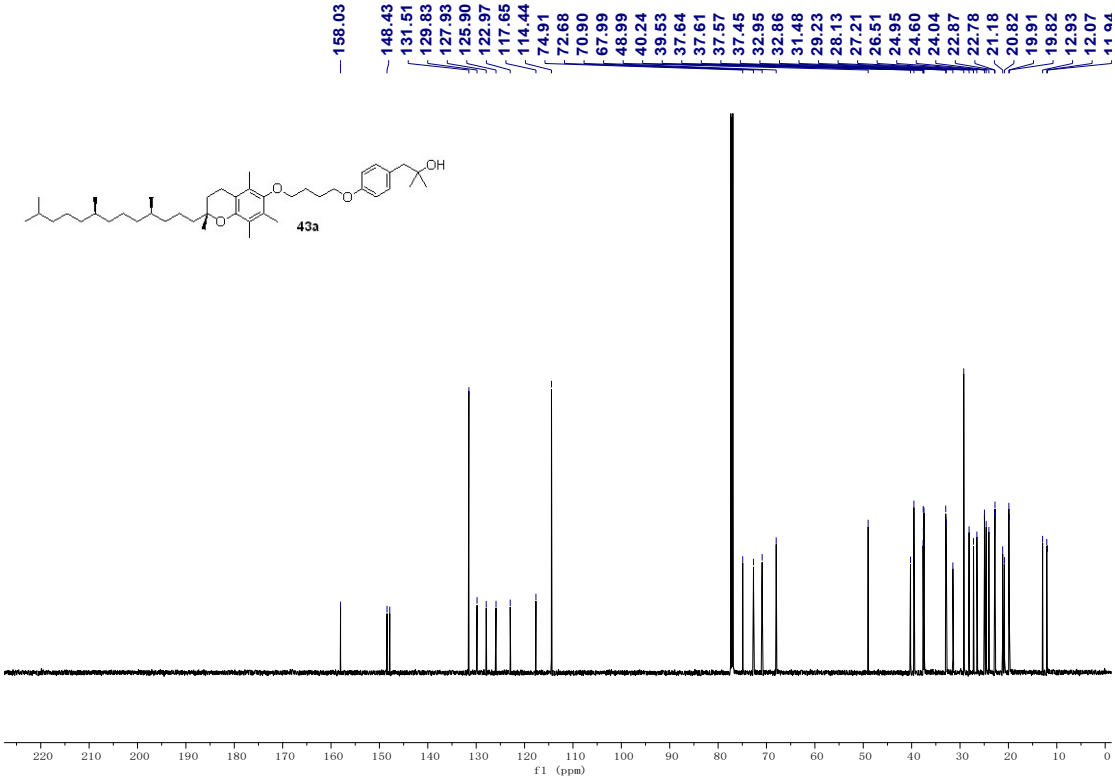

7.17
7.15
6.97
6.95
2.89
2.87
2.87
2.85
2.76
1.98
1.97
1.96
1.94
1.83
1.81
1.81
1.79
1.33
1.24
-0.00
OH
Cl
Cl
42a
2.00
2.00
1.07
0.78
1.06
1.06
5.98
5.97
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

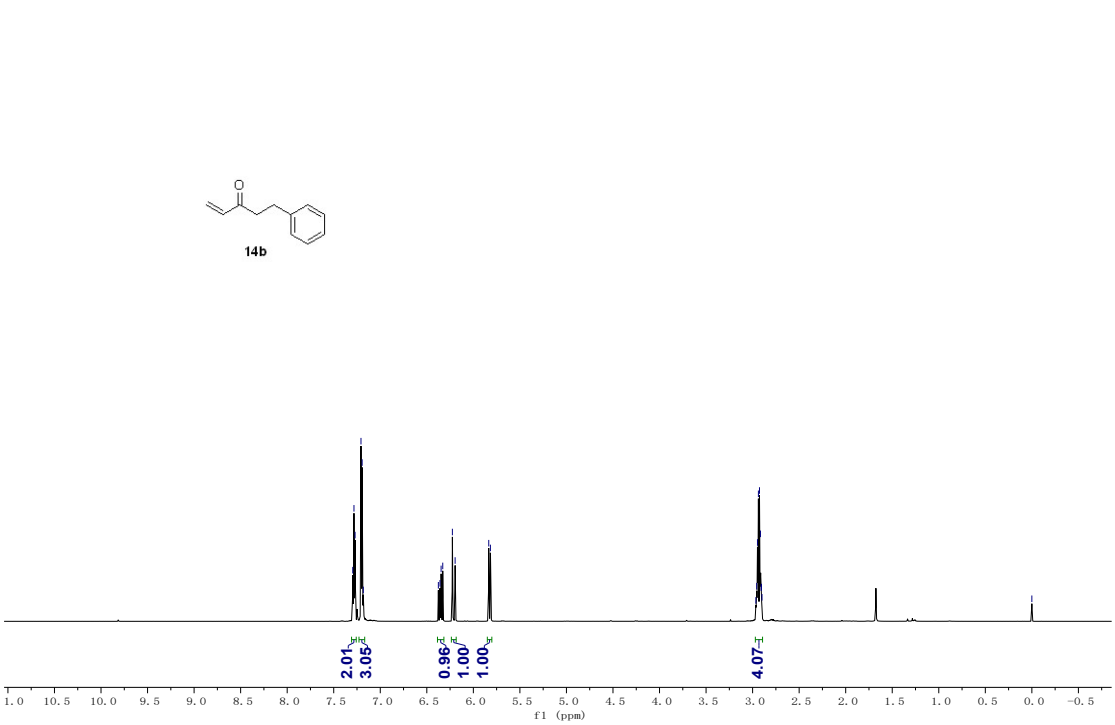

154.26
130.09
129.59
124.23
85.82
75.20
60.93
35.05
25.99
24.78
21.64
OH
Cl
Cl
42a
f1 (ppm)
7.10
7.08
6.88
6.86
4.04
4.03
4.01
3.72
3.70
3.69
3.60
2.58
2.56
2.55
2.17
2.12
2.12
2.08
2.02
2.02
2.01
1.99
1.97
1.96
1.95
1.79
1.77
1.75
1.73
1.56
1.54
1.52
1.51
1.49
1.42
1.41
1.39
1.38
1.36
1.34
1.32
1.28
1.26
1.25
1.22
1.16
1.15
1.12
1.08
1.07
0.87
0.86
0.86
0.85
0.85
0.84

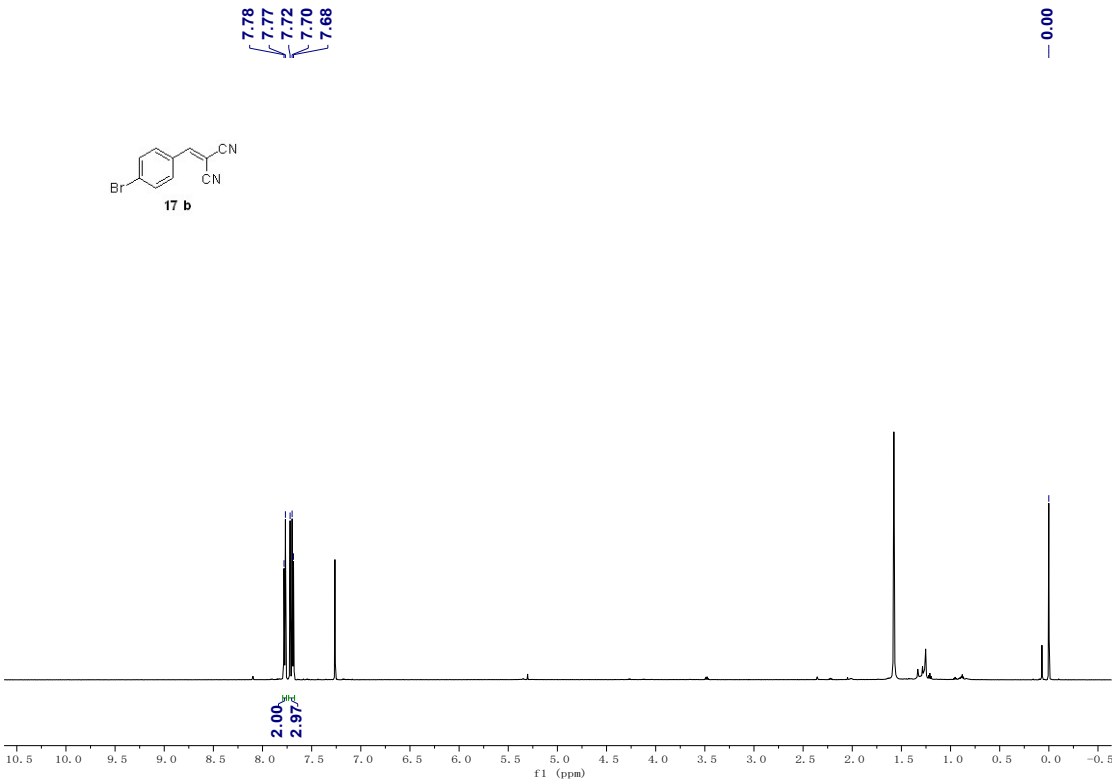

O
O
O
43a step one
DCM
2.02
2.01
2.01
1.97
2.05
1.93
2.96
6.00
3.00
4.15
2.20
3.02
4.09
17.19
12.27
f1 (ppm)

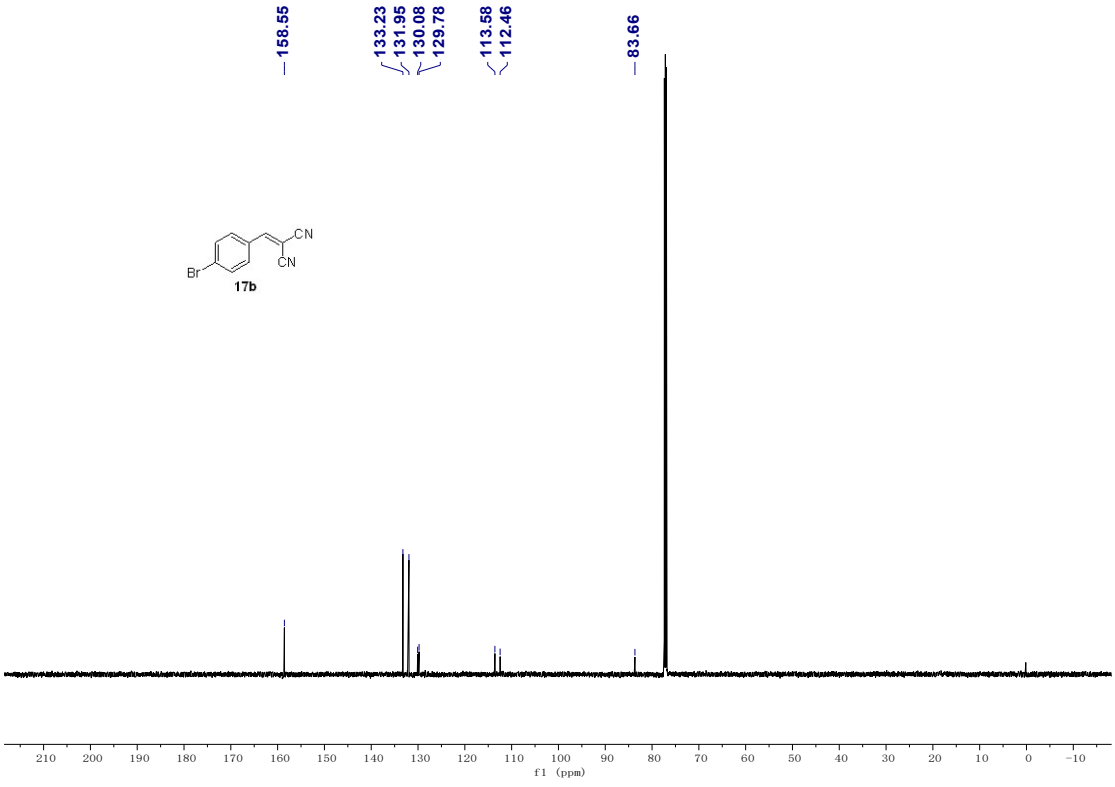

206.95
158.35
148.40
147.85
130.50
127.89
126.34
125.87
122.95
117.63
114.95
74.89
72.63
67.99
50.30
40.21
39.52
37.62
37.59
37.55
37.43
32.93
32.83
31.46
29.20
28.11
27.18
26.46
24.94
24.58
24.02
22.86
22.76
21.16
20.80
19.89
19.80
12.91
12.05
11.92
43a step one
f1 (ppm)

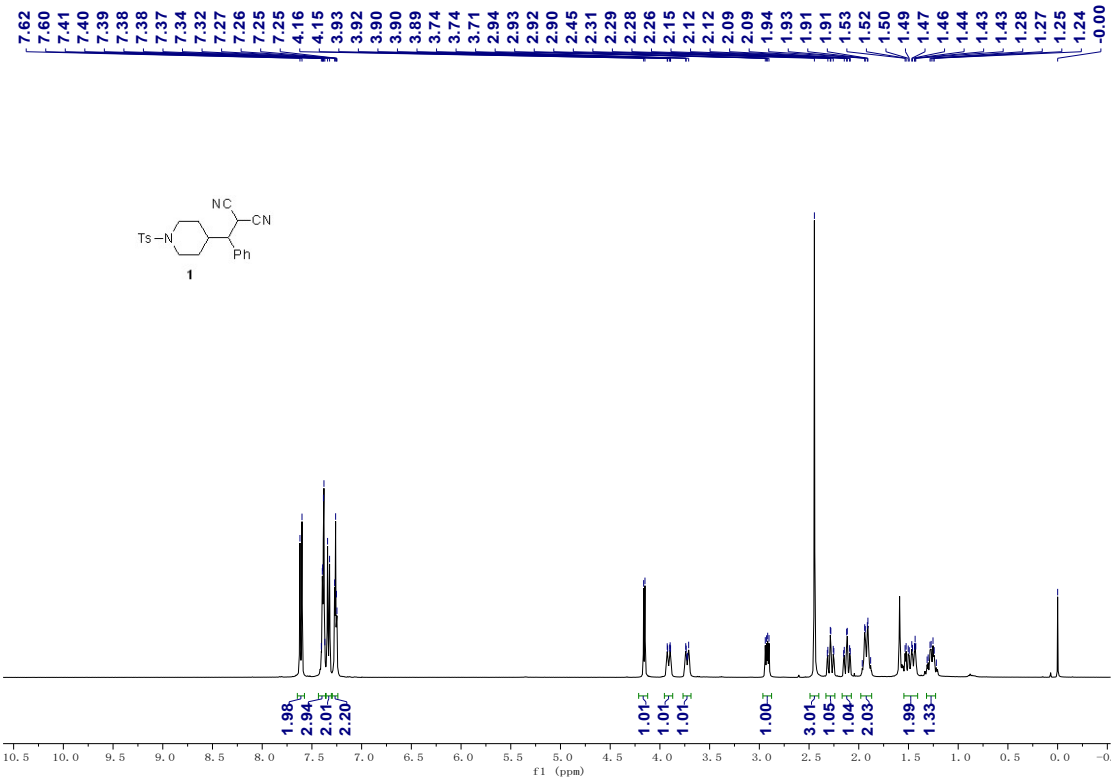

7.13
7.11
6.87
6.86
4.05
4.04
4.03
3.72
3.71
3.70
2.70
2.58
2.57
2.56
2.17
2.13
2.08
2.03
2.02
1.98
1.96
1.95
1.80
1.76
1.54
1.53
1.52
1.51
1.40
1.39
1.38
1.37
1.36
1.29
1.28
1.27
1.25
1.25
1.24
1.23
1.21
1.15
1.14
1.14
1.13
1.13
1.12
1.07
1.07
1.06
1.05
1.04
0.87
0.86
0.85
0.84
OH
43a
2.04
1.98
2.97
2.94
2.96
2.06
2.03
2.04
3.00
21.59
6.19
12.00
2.01
2.00
2.02
2.00
2.04
1.98
2.97
2.94
2.96
2.06
2.03
2.04
3.00
21.59
6.19
12.00
f1 (ppm)

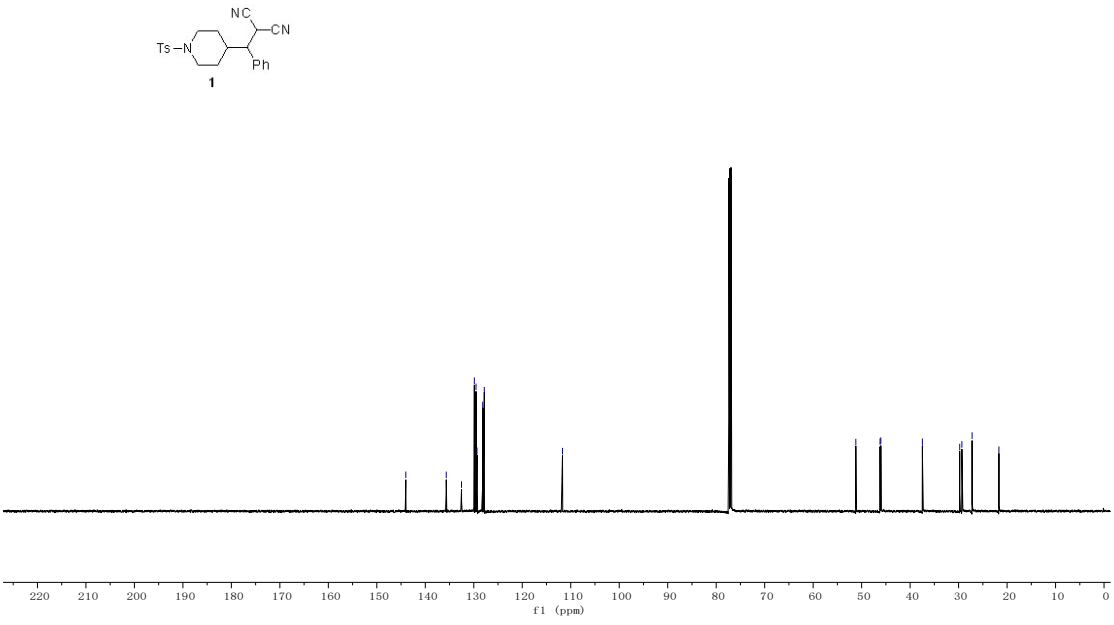

158.03
148.43
131.51
129.83
127.93
125.90
122.97
117.65
114.44
74.91
72.68
70.90
67.99
48.99
40.24
39.53
37.64
37.61
37.57
37.45
32.95
32.86
31.48
29.23
28.13
27.21
26.51
24.95
24.60
24.04
22.87
22.78
21.18
20.82
19.91
19.82
12.93
12.07
OH
43a
f1 (ppm)

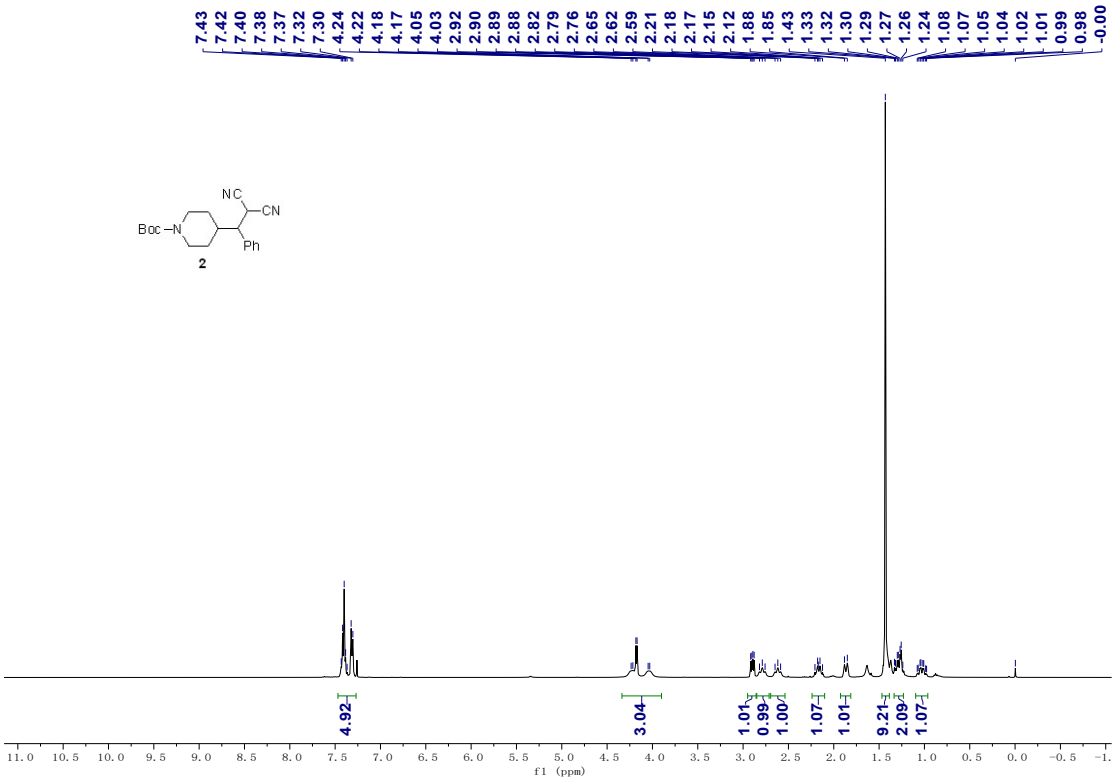

7.30
7.28
7.27
7.21
7.19
7.18
6.38
6.36
6.35
6.33
6.23
6.20
5.83
5.82
2.96
2.96
2.95
2.94
2.93
2.92
2.91
2.90
2.90
0.00
O
14b
2.01
3.05
0.96
1.00
1.00
4.07
f1 (ppm)

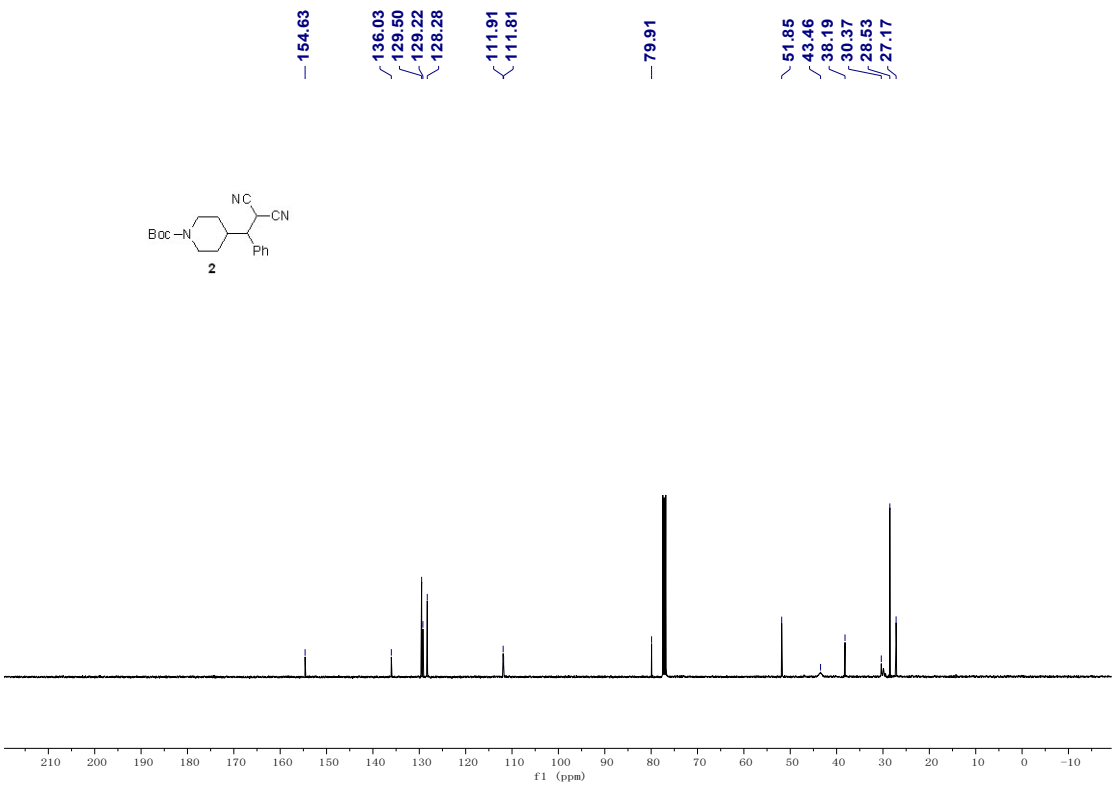

7.78
7.77
7.72
7.70
7.68
0.00
CN
CN
Br
17 b
2.00
2.97
f1 (ppm)

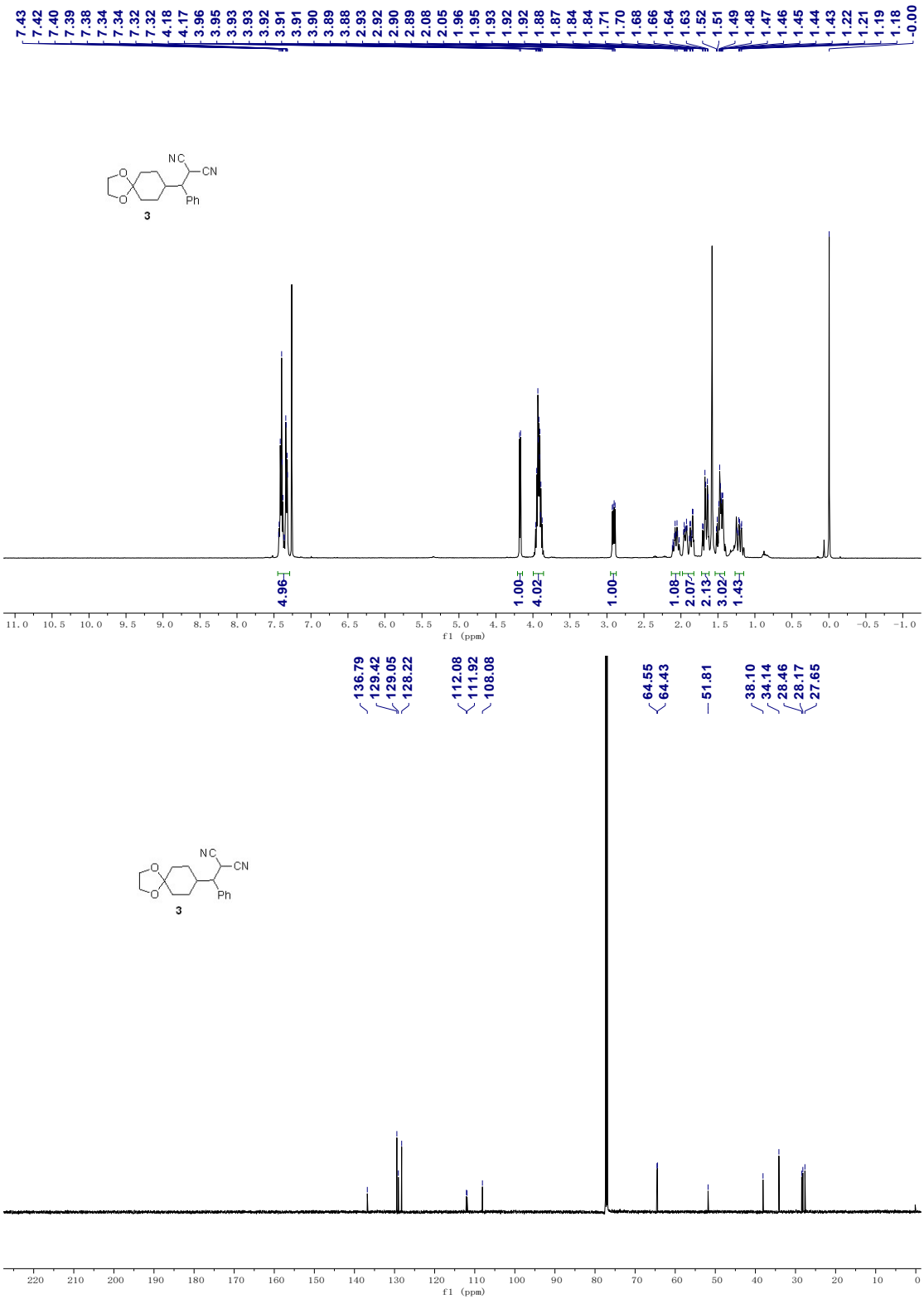

158.55
133.23
131.95
130.08
129.78
113.58
112.46
83.66
CN
CN
Br
17b
f1 (ppm)

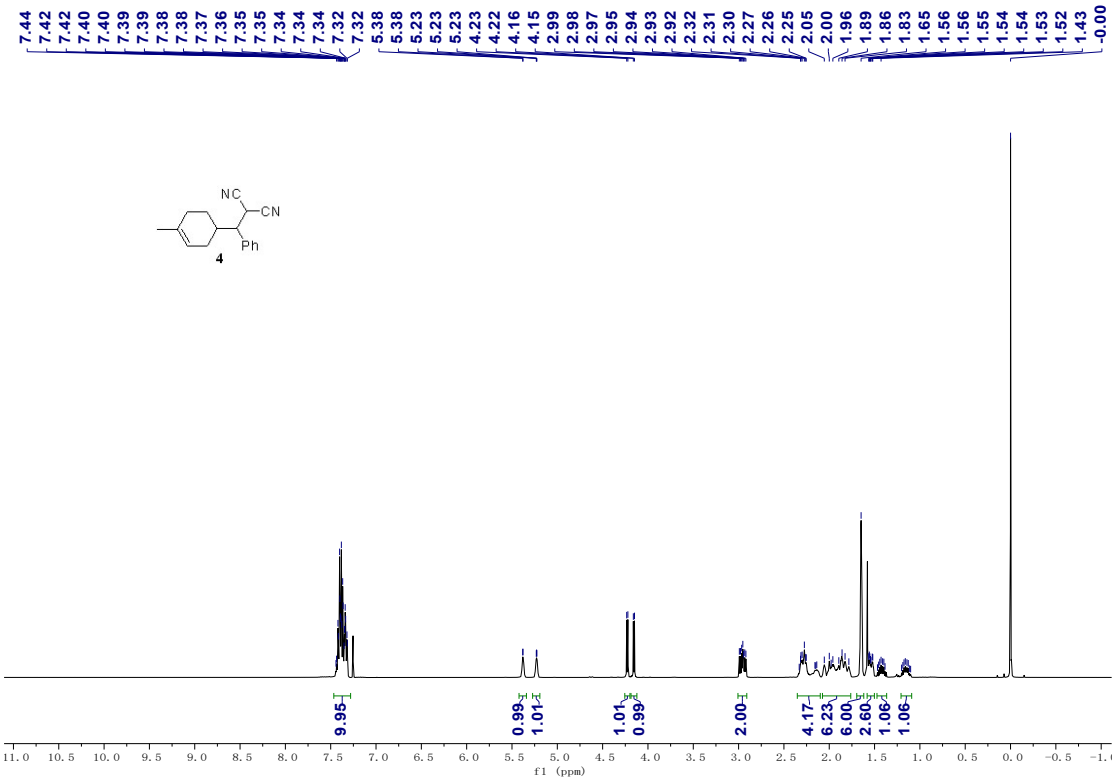

7.62 7.60 7.41 7.40 7.39 7.38 7.38 7.37 7.34 7.32 7.27 7.26 7.25 7.25 4.16 4.15 3.93 3.92 3.90 3.90 3.89 3.74 3.74 3.71 2.94 2.93 2.92 2.90 2.45 2.31 2.29 2.28 2.26 2.15 2.12 2.12 2.09 2.09 1.94 1.93 1.91 1.91 1.53 1.52 1.50 1.49 1.47 1.46 1.44 1.43 1.43 1.28 1.27 1.25 1.24 -0.00
NC
CN
Ts–N
Ph
1
1.98 2.94 2.01 2.20 1.01 1.01 1.01 1.00 3.01 1.05 1.04 2.03 1.99 1.33
f1 (ppm)

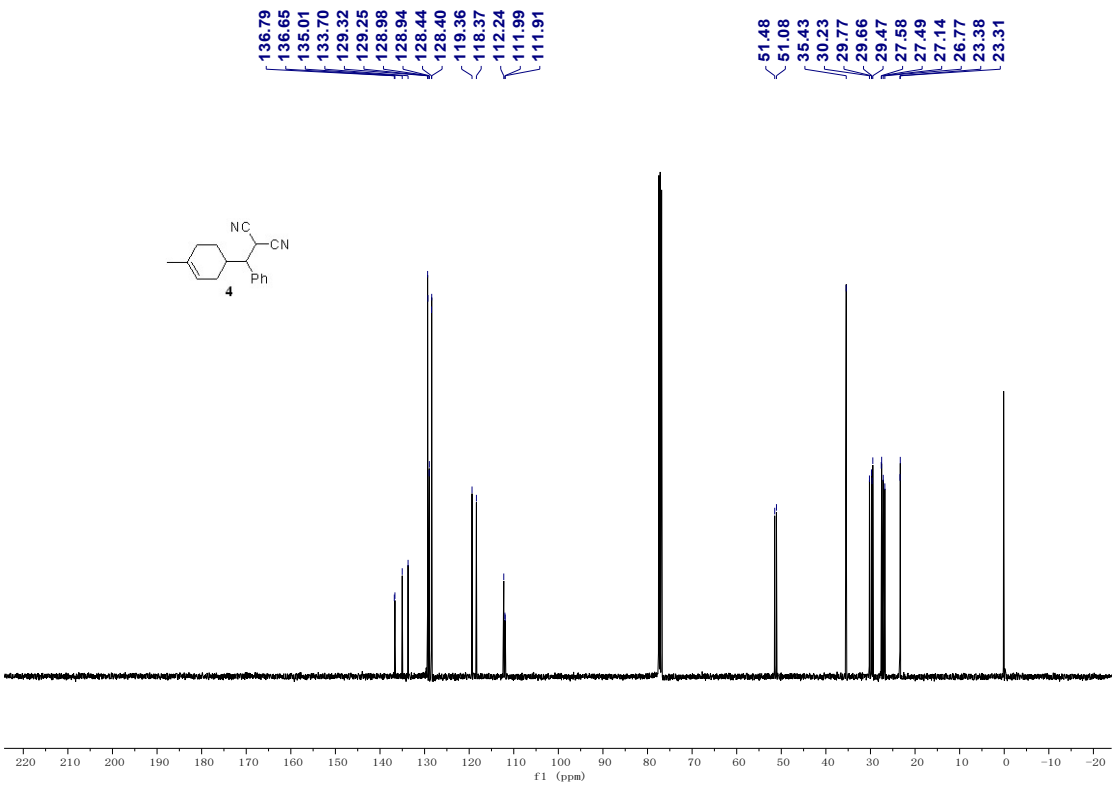

144.02 135.68 132.53 129.89 129.54 129.34 128.16 127.83 111.71
51.20 46.20 46.04 37.47 29.77 29.32 27.22 21.68
NC
CN
Ts–N
Ph
1
f1 (ppm)

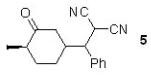

7.43 7.42 7.40 7.38 7.37 7.32 7.30 4.24 4.22 4.18 4.17 4.05 4.03 2.92 2.90 2.89 2.88 2.82 2.79 2.76 2.65 2.62 2.59 2.21 2.18 2.17 2.15 2.12 1.88 1.85 1.43 1.33 1.32 1.30 1.29 1.27 1.26 1.24 1.08 1.07 1.05 1.04 1.02 1.01 0.99 0.98 -0.00
NC
CN
Boc–N
Ph
2
4.92 3.04 1.01 0.99 1.00 1.07 1.01 9.21 2.09 1.07
f1 (ppm)


154.63 136.03 129.50 129.22 128.28 111.91 111.81 79.91 51.85 43.46 38.19 30.37 28.53 27.17
NC
CN
Boc–N
Ph
2
f1 (ppm)

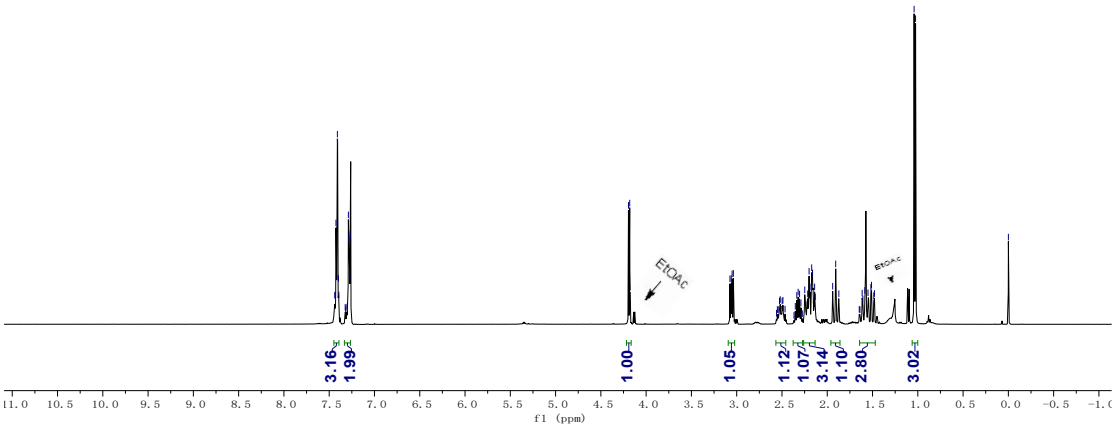

7.43 7.42 7.40 7.39 7.38 7.34 7.34 7.32 7.32 4.18 4.17 3.96 3.95 3.93 3.93 3.92 3.91 3.91 3.90 3.89 3.88 2.93 2.92 2.90 2.89 2.08 2.05 1.96 1.95 1.93 1.92 1.92 1.88 1.87 1.84 1.84 1.71 1.70 1.68 1.66 1.64 1.63 1.52 1.51 1.49 1.48 1.47 1.46 1.45 1.44 1.43 1.22 1.21 1.19 1.18 -0.00
NC
CN
Ph
3
4.96
1.00
4.02
1.00
1.08
2.07
2.13
3.02
1.43
f1 (ppm)
136.79
129.42
129.05
128.22
112.08
111.92
108.08
64.55
64.43
51.81
38.10
34.14
28.46
28.17
27.65
NC
CN
Ph
3
f1 (ppm)

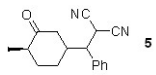

7.44 7.42 7.42 7.40 7.40 7.39 7.39 7.38 7.38 7.37 7.36 7.35 7.35 7.34 7.34 7.34 7.32 7.32
5.38 5.38 5.23 5.23 5.23 4.23 4.22 4.16 4.15 2.99 2.98 2.97 2.95 2.94 2.93 2.92 2.32 2.31 2.30 2.27 2.26 2.25 2.05 2.00 1.96 1.89 1.86 1.83 1.65 1.56 1.56 1.55 1.54 1.54 1.53 1.52 1.43 -0.00
NC
CN
Ph
4
9.95
0.99
1.01
1.01
0.99
2.00
4.17
6.23
6.00
2.60
1.06
1.06
f1 (ppm)


136.79 136.65 135.01 133.70 129.32 129.25 128.98 128.94 128.44 128.40 119.36 118.37 112.24 111.99 111.91
51.48 51.08 35.43 30.23 29.77 29.66 29.47 27.58 27.49 27.14 26.77 23.38 23.31
NC
CN
Ph
4
f1 (ppm)

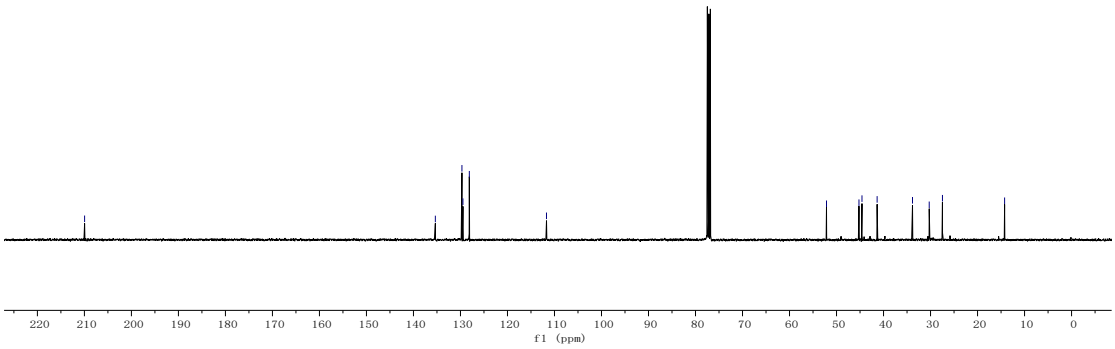


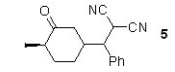
The small polar diastereoisomer



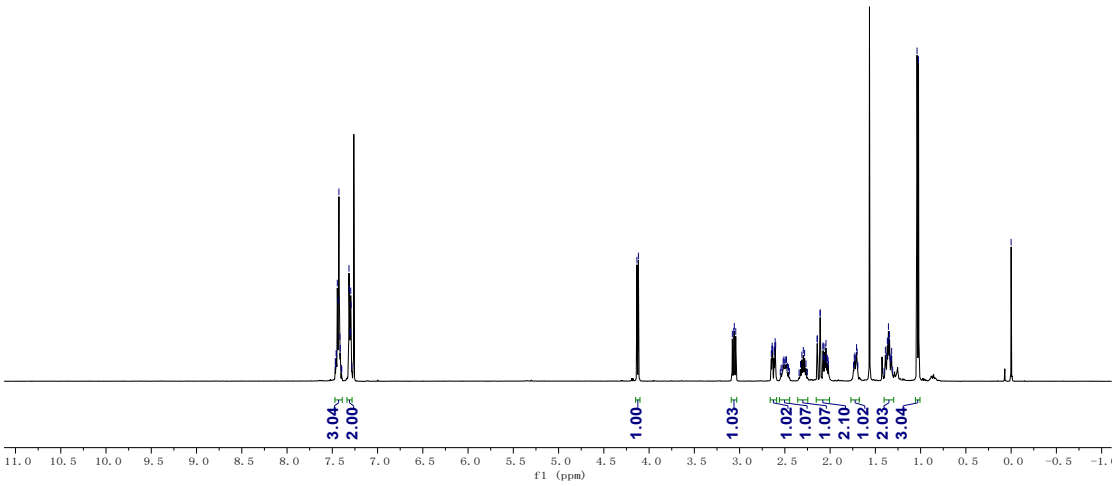


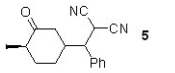
The small polar diastereoisomer



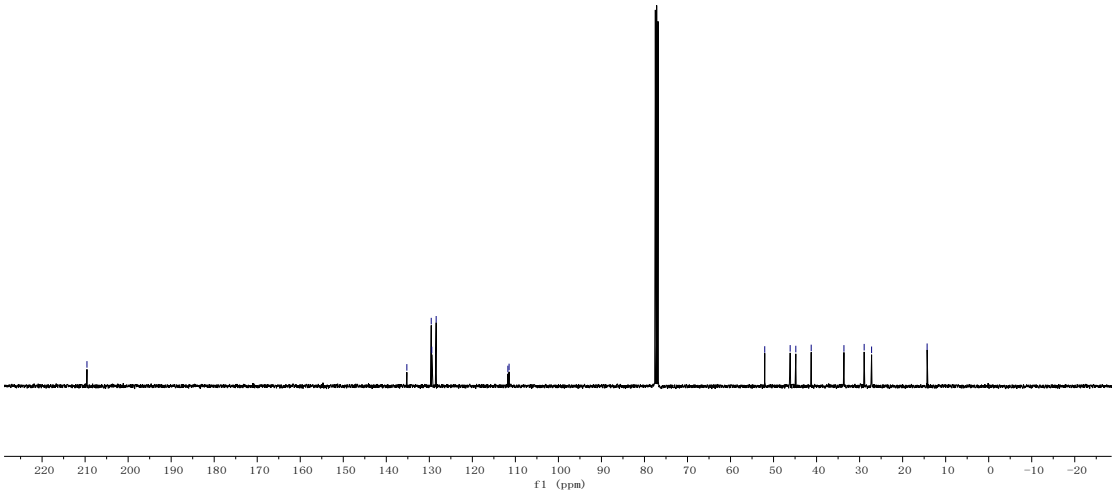


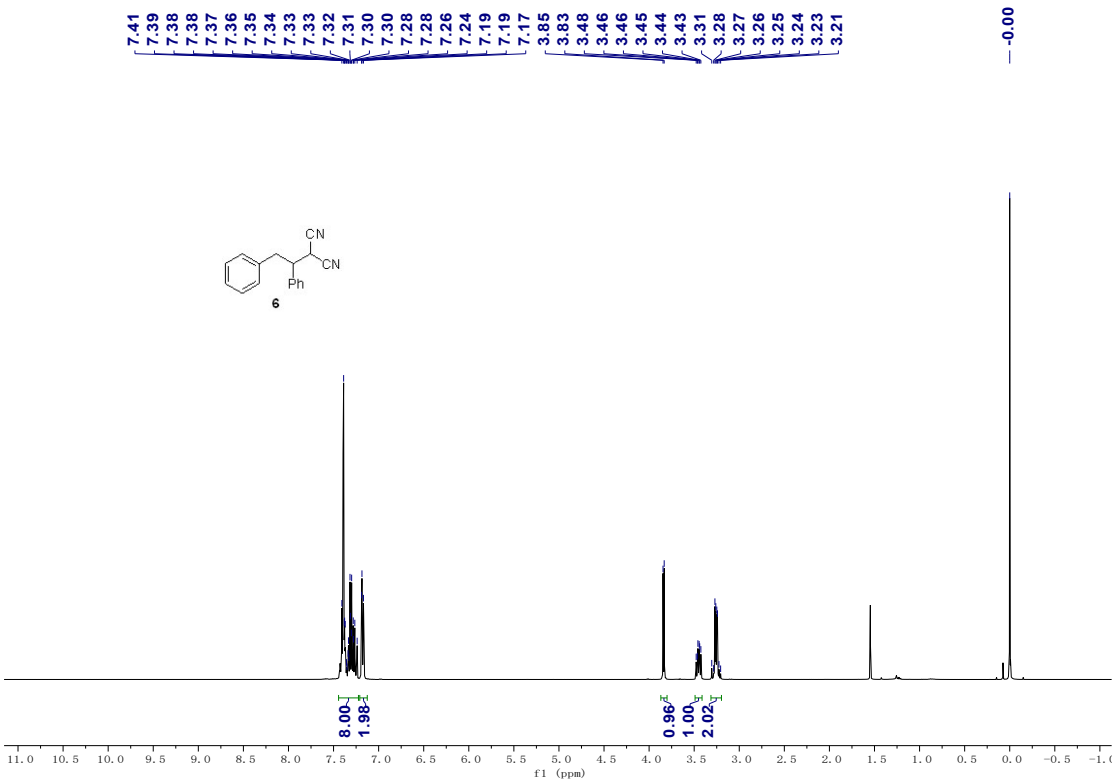
The large polar diastereoisomer

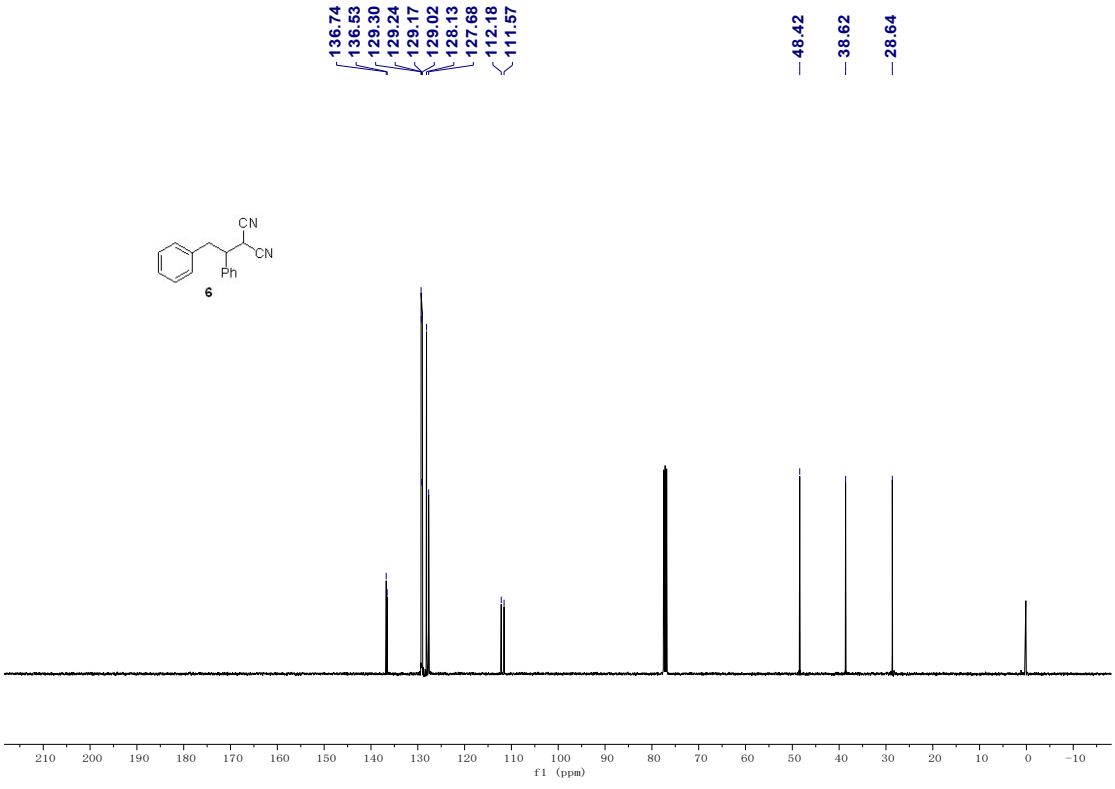


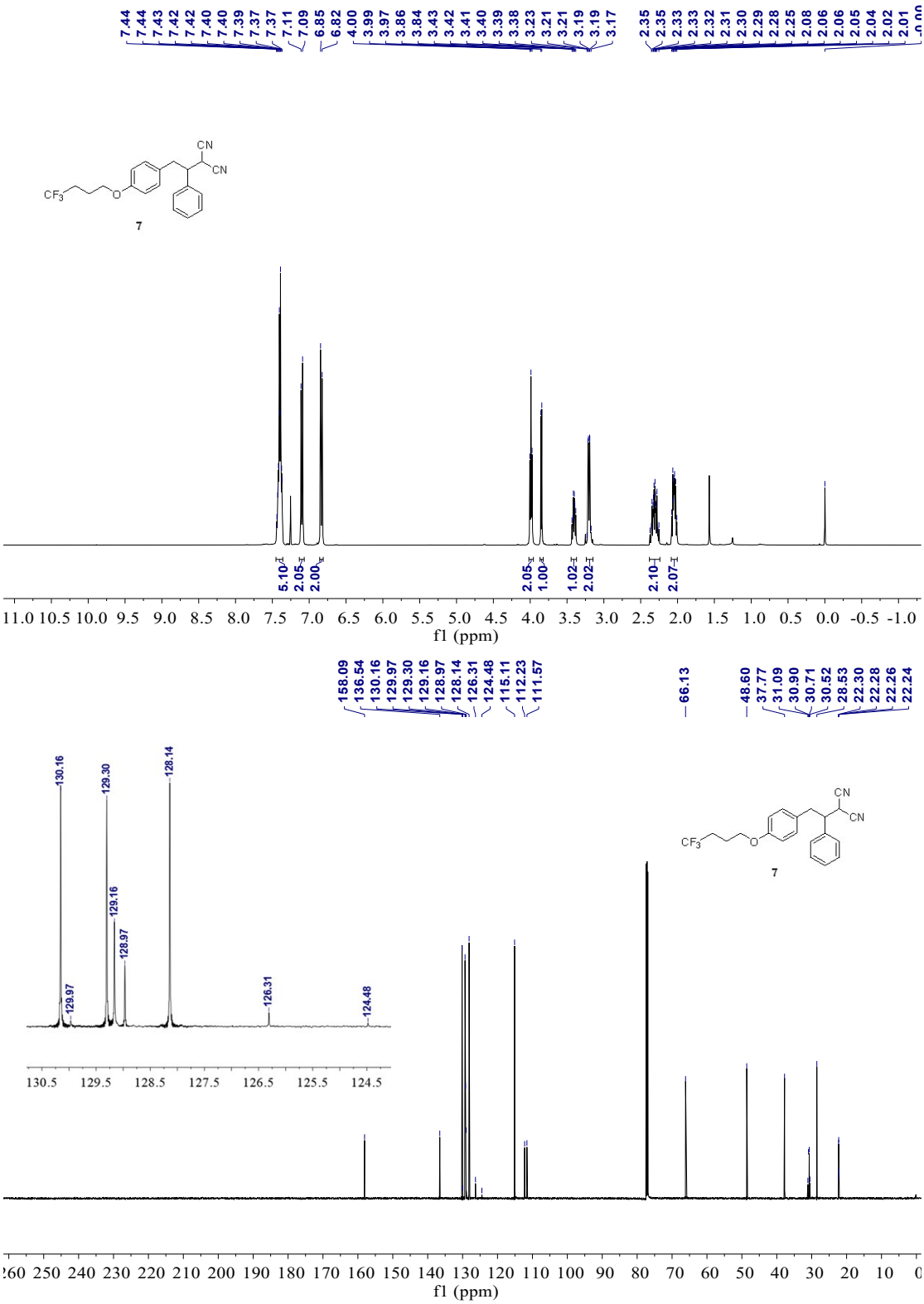


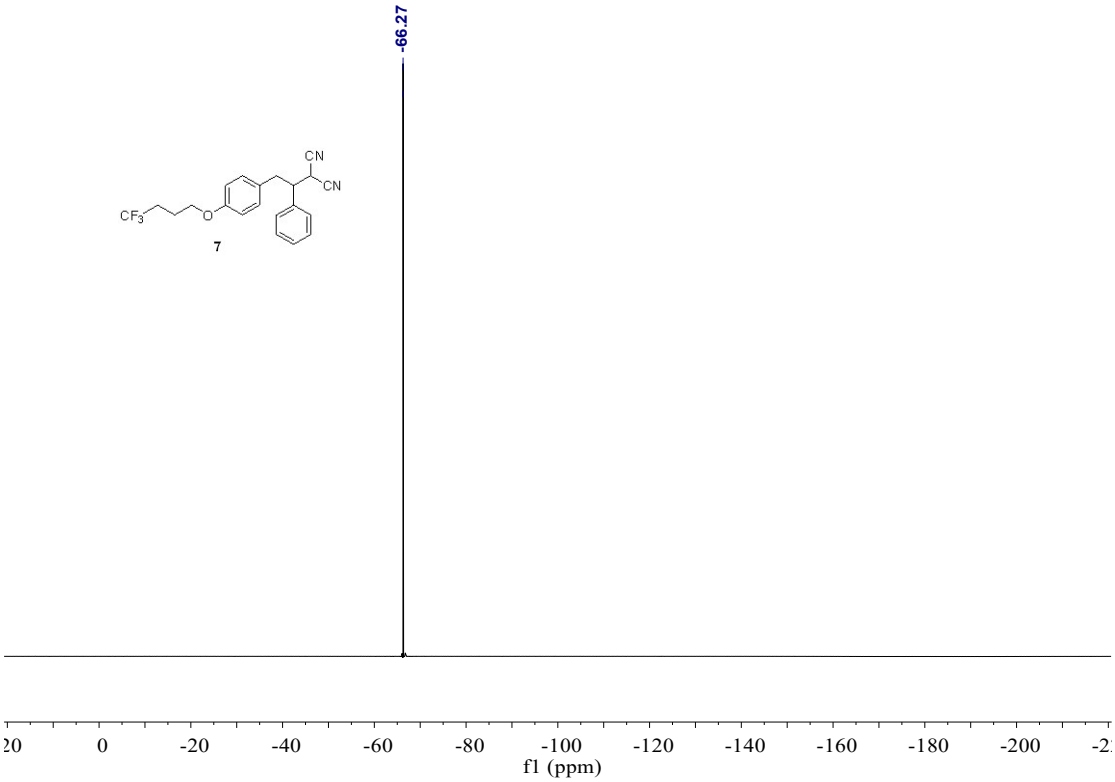
The large polar diastereoisomer

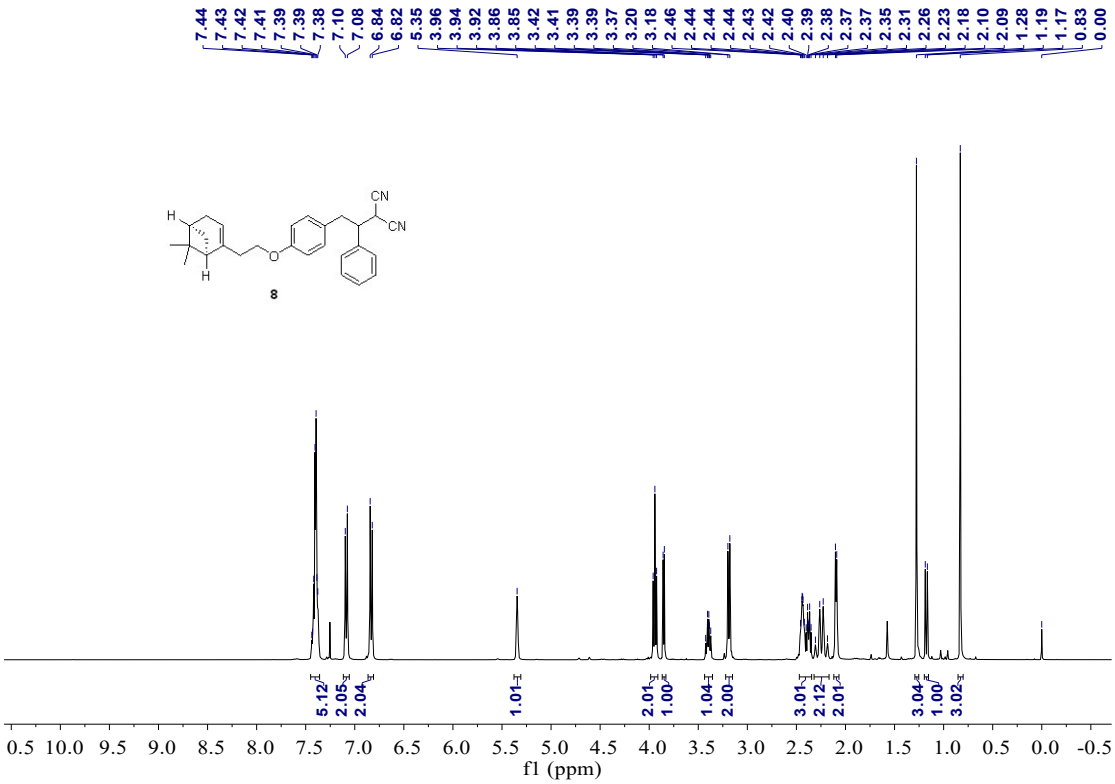


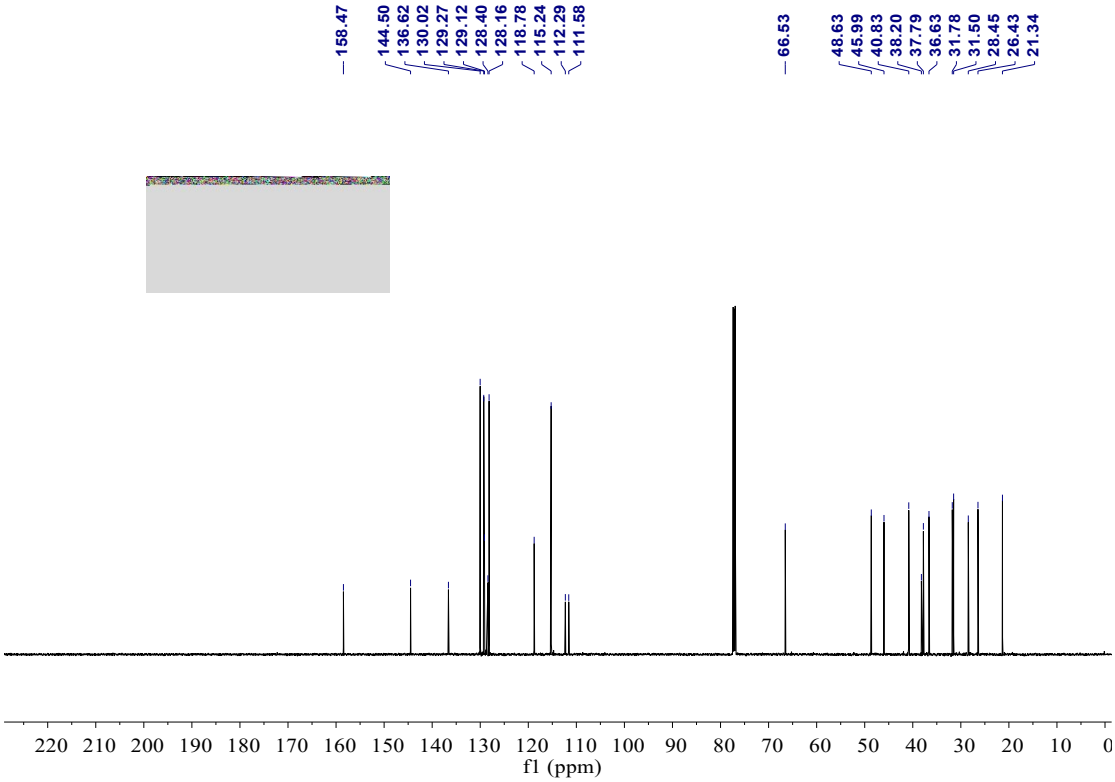

7.41
7.39
7.38
7.38
7.37
7.36
7.35
7.34
7.33
7.33
7.32
7.31
7.30
7.30
7.28
7.28
7.26
7.24
7.19
7.19
7.17
3.85
3.83
3.48
3.46
3.46
3.45
3.44
3.43
3.31
3.28
3.27
3.26
3.25
3.24
3.23
3.21
-0.00
CN
CN
Ph
6
8.00
1.98
0.96
1.00
2.02
f1 (ppm)

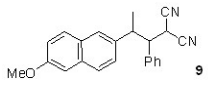

136.74
136.53
129.30
129.24
129.17
129.02
128.13
127.68
112.18
111.57
48.42
38.62
28.64
CN
CN
Ph
6
f1 (ppm)


7.44 7.44 7.43 7.42 7.42 7.40 7.40 7.39 7.37 7.37 7.11 7.09 6.85 6.82 4.00 3.99 3.97 3.86 3.84 3.43 3.42 3.41 3.40 3.39 3.38 3.23 3.21 3.21 3.19 3.19 3.17 2.35 2.35 2.33 2.33 2.32 2.31 2.30 2.29 2.28 2.25 2.08 2.06 2.06 2.05 2.04 2.02 2.01
CN
CN
CF3
O
7
5.10 2.05 2.00 2.05 1.00 1.02 2.02 2.10 2.07
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 -0.5 -1.0
f1 (ppm)
158.09 136.54 130.16 129.97 129.30 129.16 128.97 128.14 126.31 124.48 115.11 112.23 111.57
66.13
48.60 37.77 31.09 30.90 30.71 30.52 28.53 22.30 22.28 22.26 22.24
130.16 129.97 129.30 129.16 128.97 128.14 126.31 124.48
130.5 129.5 128.5 127.5 126.5 125.5 124.5
260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-66.27
CN
CN
CF3
O
7
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
f1 (ppm)

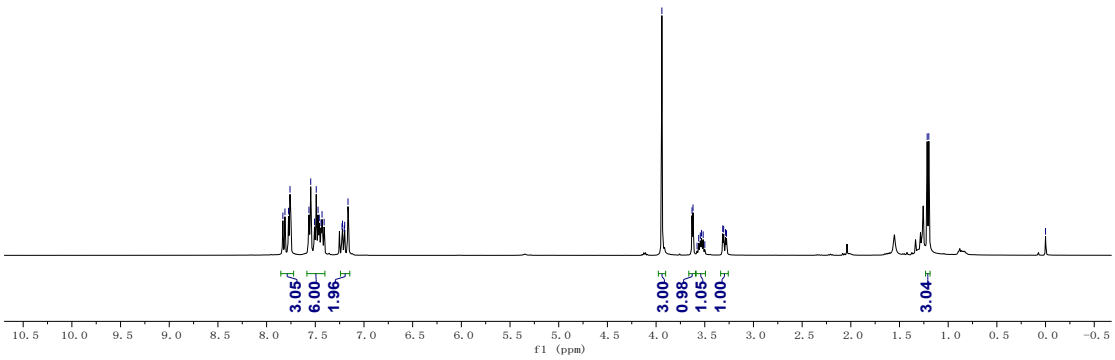

7.44
7.43
7.42
7.41
7.39
7.39
7.38
7.10
7.08
6.84
6.82
5.35
3.96
3.94
3.92
3.86
3.85
3.42
3.41
3.39
3.39
3.37
3.20
3.18
2.46
2.44
2.44
2.44
2.43
2.42
2.40
2.39
2.38
2.37
2.37
2.35
2.31
2.26
2.23
2.18
2.10
2.09
1.28
1.19
1.17
0.83
0.00
H
H
O
CN
CN
8
5.12
2.05
2.04
1.01
2.01
1.00
1.04
2.00
3.01
2.12
2.01
3.04
1.00
3.02
0.5
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
-0.5
f1 (ppm)

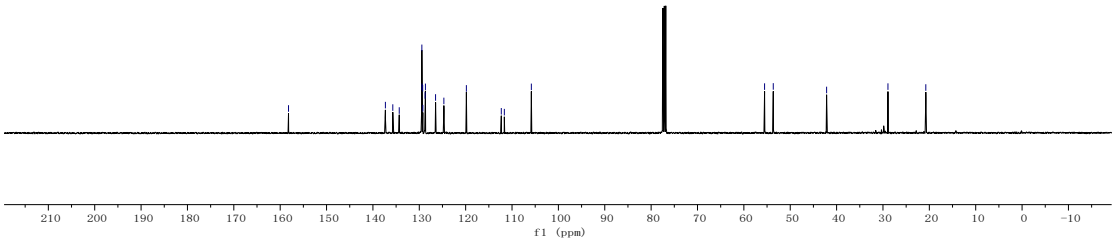


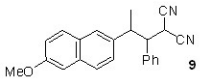


9

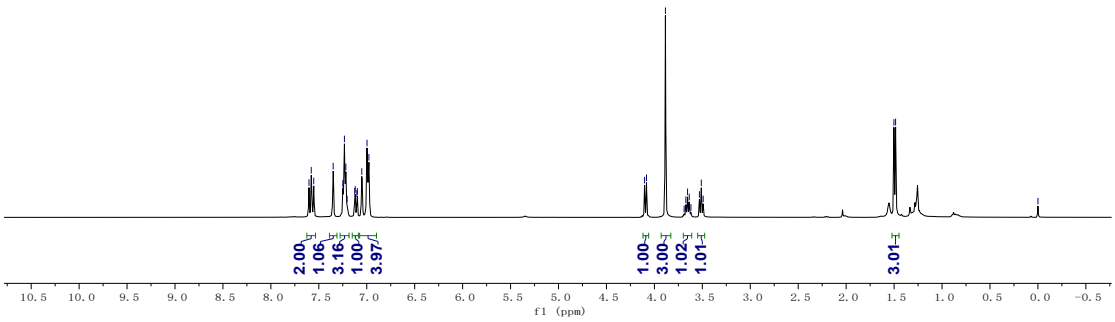
The small polar diastereoisomer

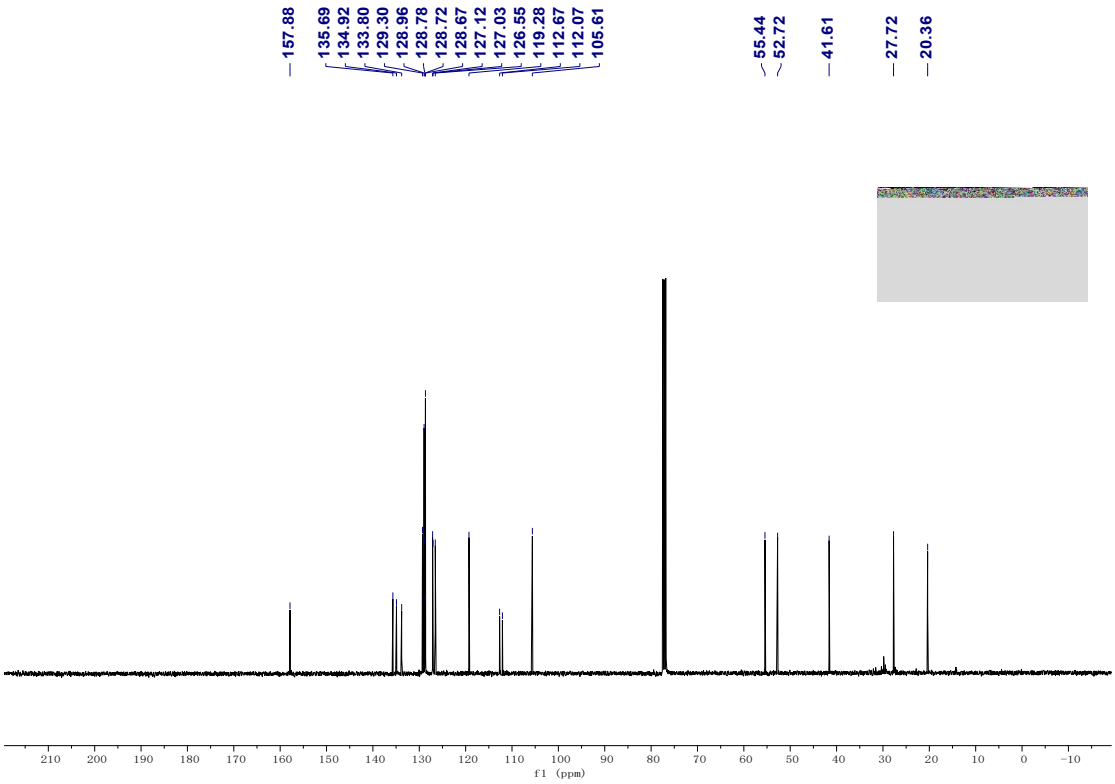


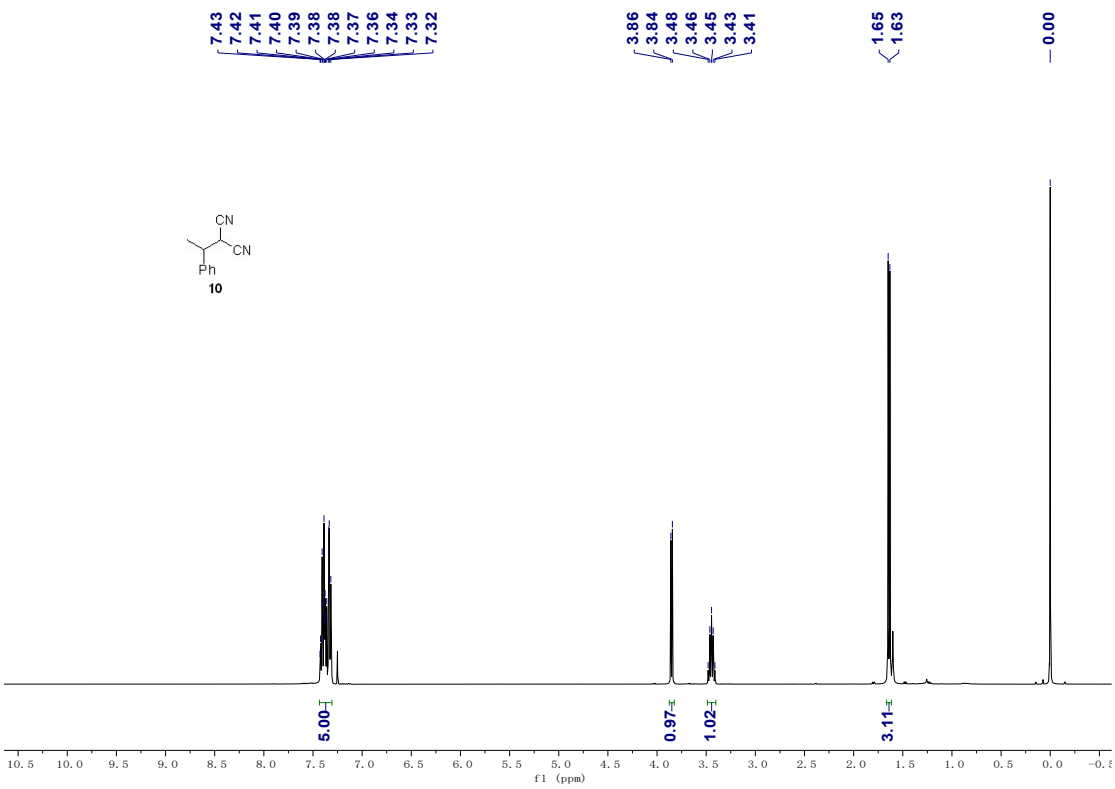




The large polar diastereoisomer




157.88
135.69
134.92
133.80
129.30
128.96
128.78
128.72
128.67
127.12
127.03
126.55
119.28
112.67
112.07
105.61
55.44
52.72
41.61
27.72
20.36
f1 (ppm)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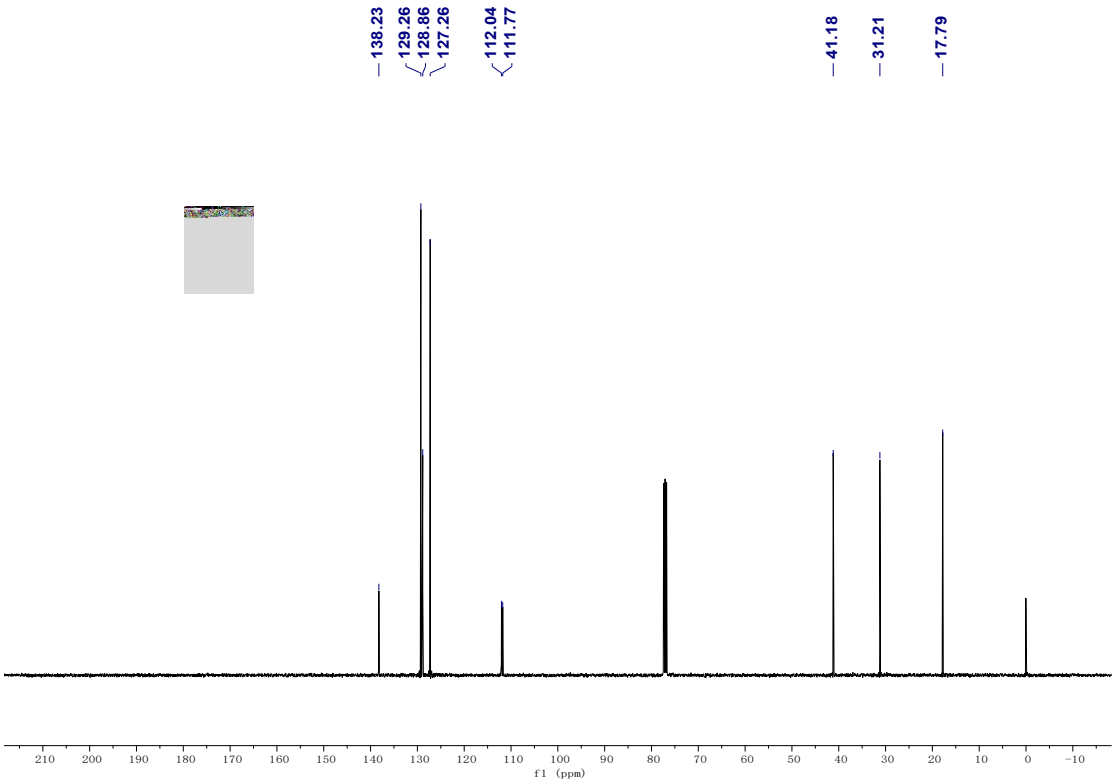
7.42
7.41
7.40
7.39
7.38
7.38
7.37
7.36
7.34
7.33
7.32
3.86
3.84
3.48
3.46
3.45
3.43
3.41
1.65
1.63
0.00
CN
CN
Ph
10
5.00
0.97
1.02
3.11
f1 (ppm)


138.23
129.26
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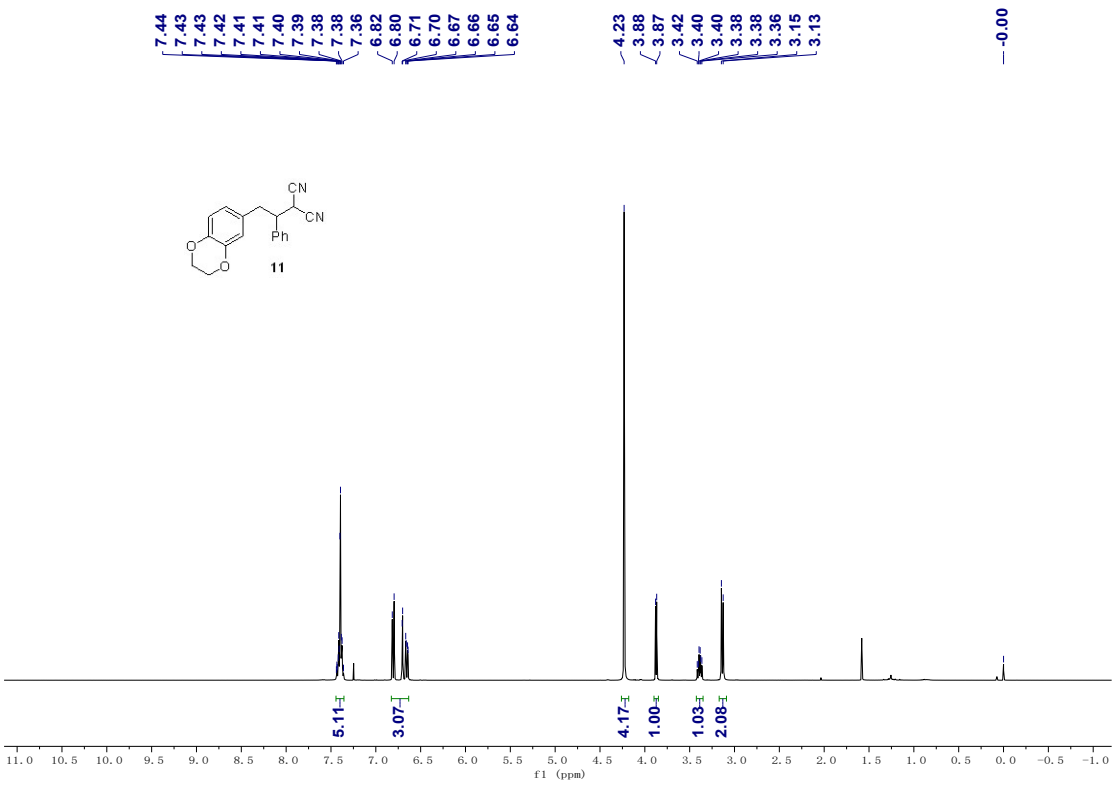
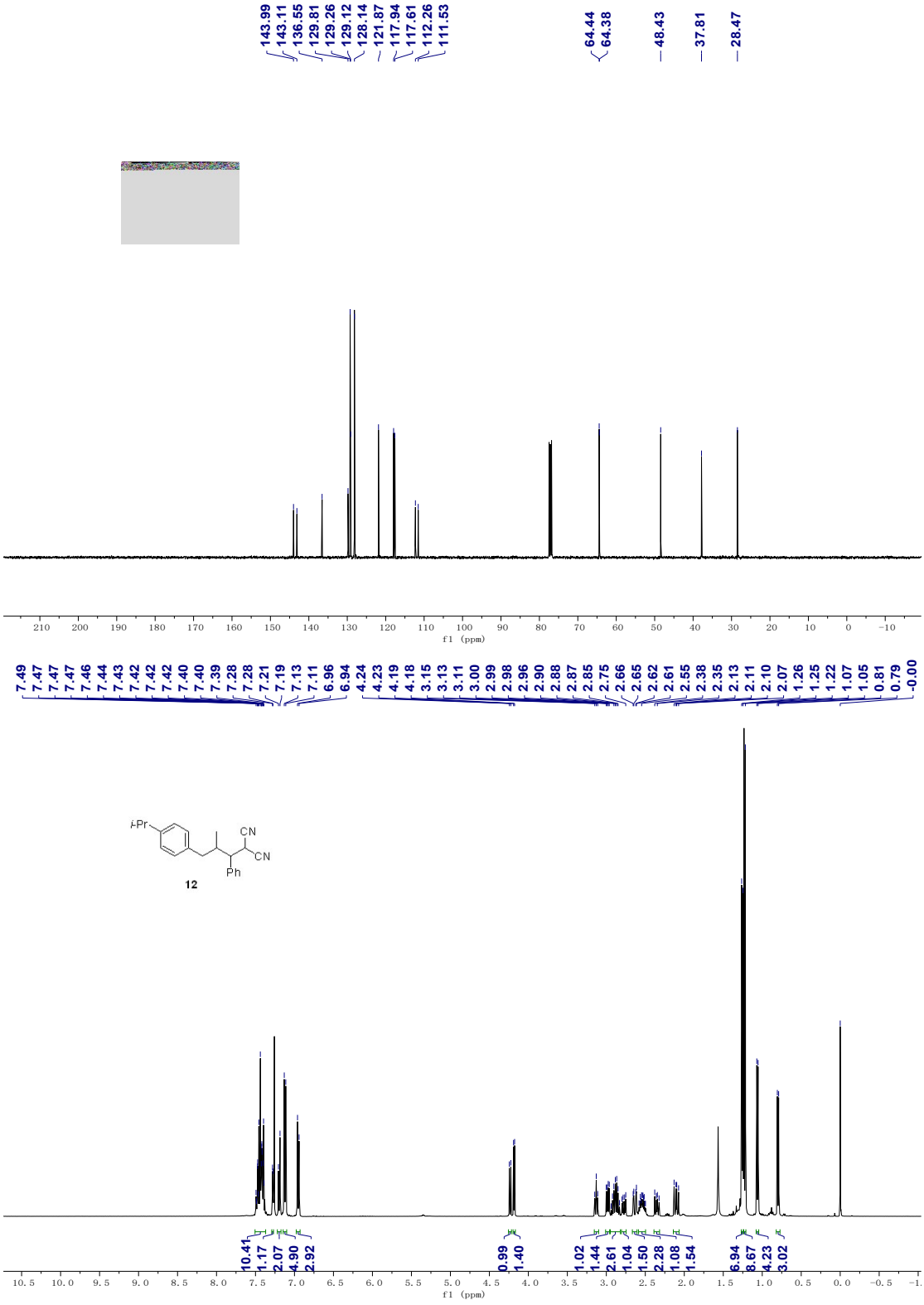
127.26
112.04
111.77
41.18
31.21
17.79
f1 (ppm)


7.44
7.43
7.43
7.42
7.41
7.41
7.40
7.39
7.38
7.38
7.36
6.82
6.80
6.71
6.70
6.67
6.66
6.65
6.64
4.23
3.88
3.87
3.42
3.40
3.40
3.38
3.38
3.36
3.15
3.13
-0.00
CN
CN
Ph
O
O
11
5.11
3.07
4.17
1.00
1.03
2.08
f1 (ppm)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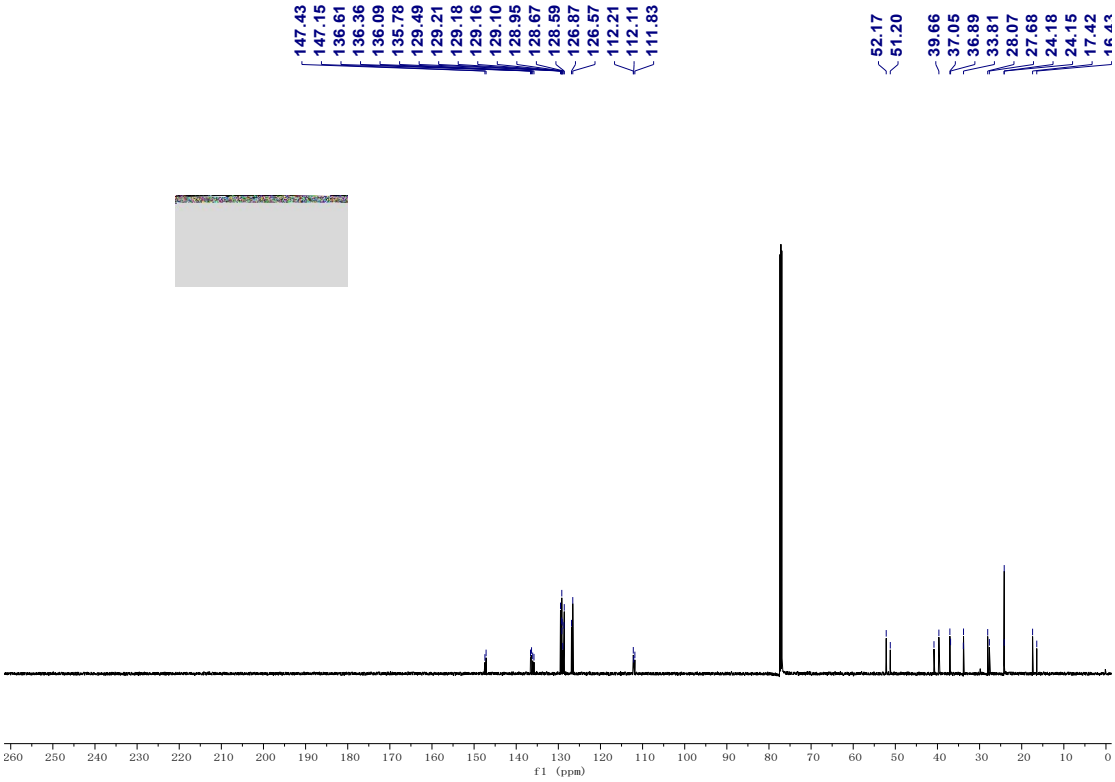
129.26
129.12
128.14
121.87
117.94
117.61
112.26
111.53
64.44
64.38
48.43
37.81
28.47
f1 (ppm)
7.49
7.47
7.47
7.47
7.46
7.44
7.43
7.42
7.42
7.42
7.40
7.40
7.39
7.28
7.28
7.21
7.19
7.13
7.11
6.96
6.94
4.24
4.23
4.19
4.18
3.15
3.13
3.11
3.00
2.99
2.98
2.96
2.90
2.88
2.87
2.85
2.75
2.66
2.65
2.62
2.61
2.55
2.38
2.35
2.13
2.11
2.10
2.07
1.26
1.25
1.22
1.07
1.05
0.81
0.79
-0.00
i-Pr
CN
CN
Ph
12
10.41
1.17
2.07
4.90
2.92
0.99
1.40
1.02
1.44
2.61
1.04
1.50
2.28
1.08
1.54
6.94
8.67
4.23
3.02
f1 (ppm)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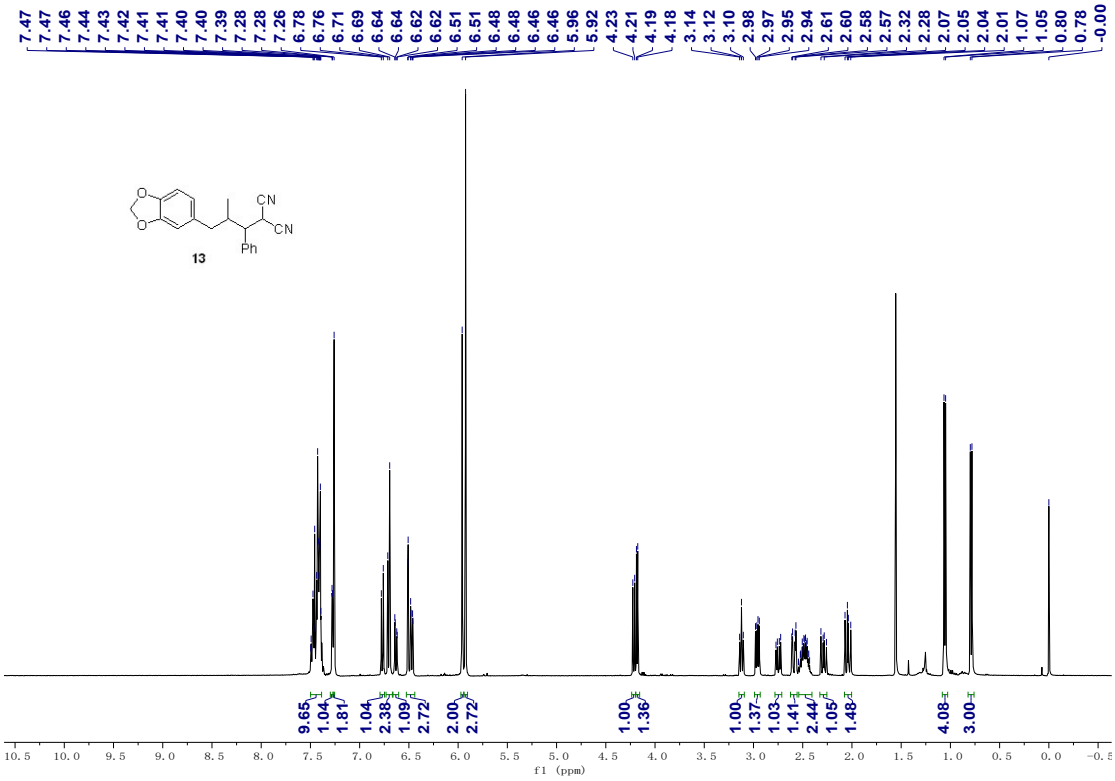
129.21
129.18
129.16
129.10
128.95
128.67
128.59
126.87
126.57
112.21
112.11
111.83
52.17
51.20
39.66
37.05
36.89
33.81
28.07
27.68
24.18
24.15
17.42
f1 (ppm)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7.47
7.46
7.44
7.43
7.42
7.41
7.41
7.40
7.40
7.39
7.28
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7.26
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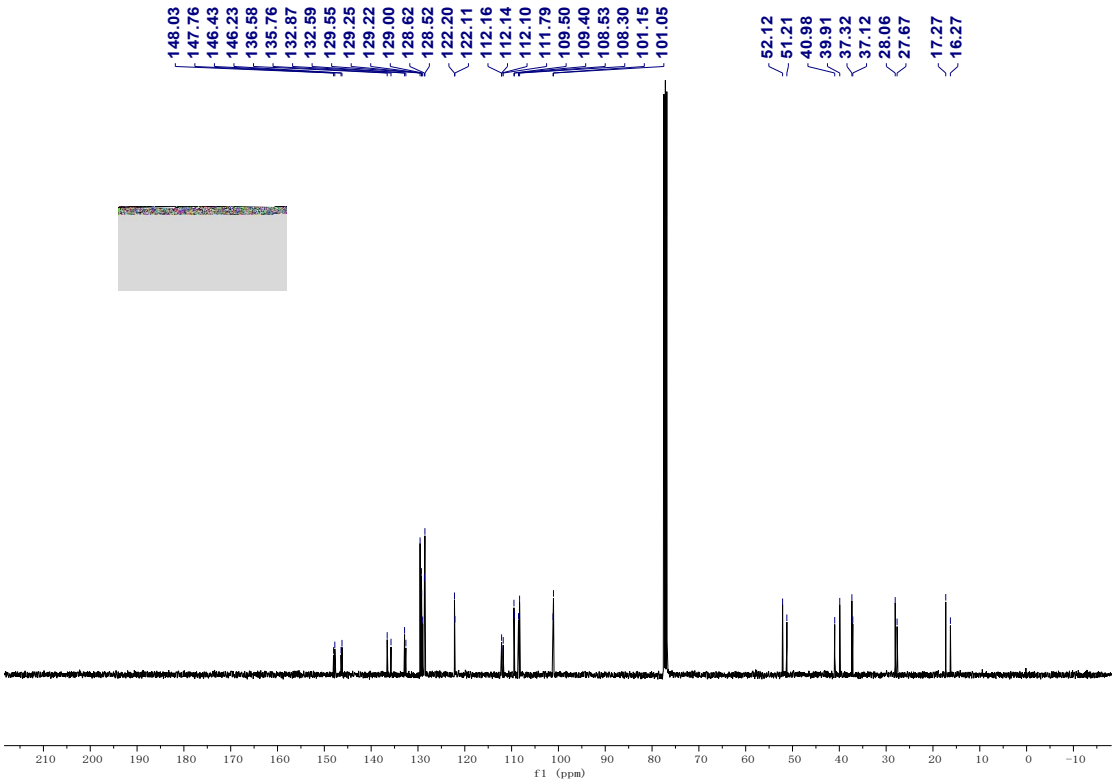
6.69
6.64
6.64
6.62
6.62
6.51
6.51
6.48
6.48
6.46
6.46
5.96
5.92
4.23
4.21
4.19
4.18
3.14
3.12
3.10
2.98
2.97
2.95
2.94
2.61
2.60
2.58
2.57
2.32
2.28
2.07
2.05
2.04
2.01
1.07
1.05
0.80
0.78
-0.00
CN
CN
Ph
13
9.65
1.04
1.81
1.04
2.38
1.09
2.72
2.00
2.72
1.00
1.36
1.00
1.37
1.03
1.41
2.44
1.05
1.48
4.08
3.00
f1 (ppm)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146.23
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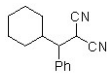
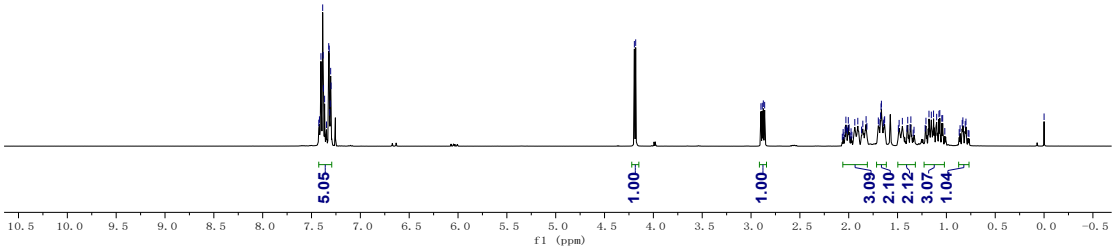
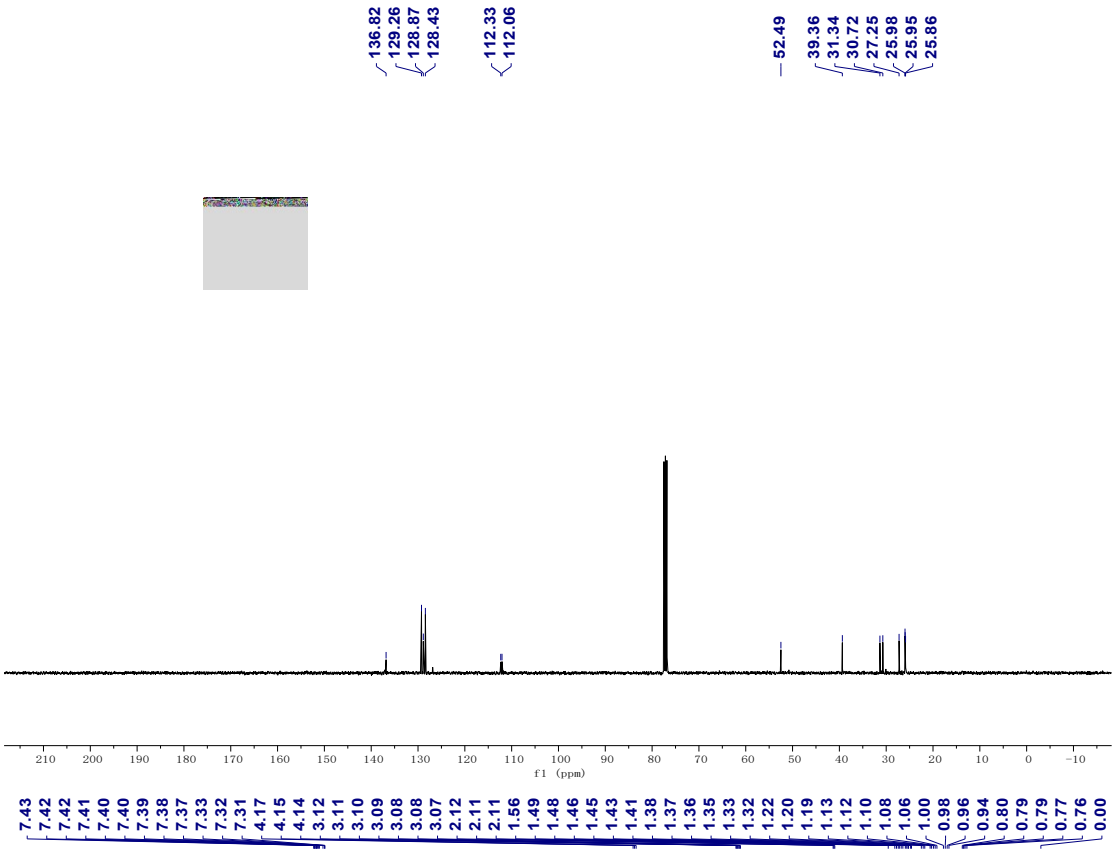
132.59
129.55
129.25
129.22
129.00
128.62
128.52
122.20
122.11
112.16
112.14
112.10
111.79
109.50
109.40
108.53
108.30
101.15
101.05
52.12
51.21
40.98
39.91
37.32
37.12
28.06
27.67
17.27
16.27
f1 (ppm)


7.43
7.42
7.40
7.39
7.38
7.38
7.37
7.35
7.32
7.32
7.31
7.31
7.30
7.30
4.19
4.18
2.90
2.89
2.87
2.86
2.03
2.00
2.00
1.94
1.91
1.86
1.82
1.81
1.70
1.69
1.67
1.67
1.67
1.64
1.64
1.63
1.48
1.45
1.40
1.37
1.36
1.22
1.21
1.18
1.15
1.13
1.10
1.08
1.07
1.05
1.04
0.84
0.83
0.81
0.80
-0.00


CN
CN
Ph

14


5.05
1.00
1.00
3.09
2.10
2.12
3.07
1.04
f1 (ppm)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128.43
112.33
112.06
52.49
39.36
31.34
30.72
27.25
25.98
25.95
25.86
f1 (ppm)

7.43
7.42
7.42
7.41
7.40
7.40
7.39
7.38
7.37
7.33
7.32
7.31
4.17
4.15
4.14
3.12
3.11
3.10
3.09
3.08
3.08
3.07
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2.11
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1.48
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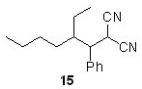
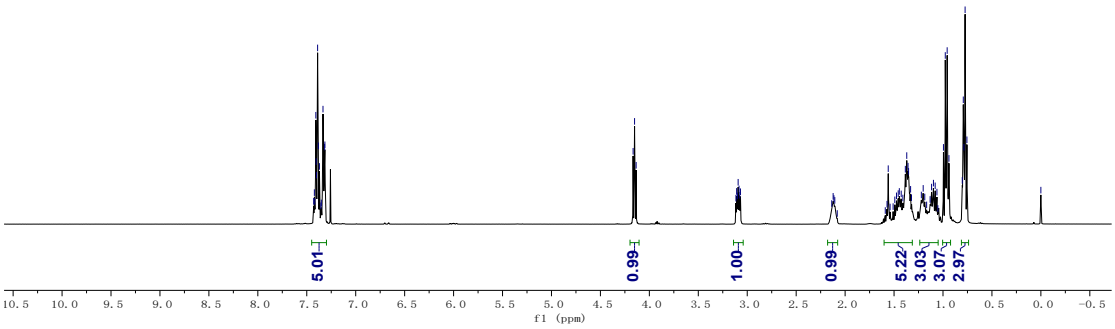
1.22
1.20
1.19
1.13
1.12
1.10
1.08
1.06
1.00
0.98
0.96
0.94
0.80
0.79
0.79
0.77
0.76
0.00


CN
CN
Ph

15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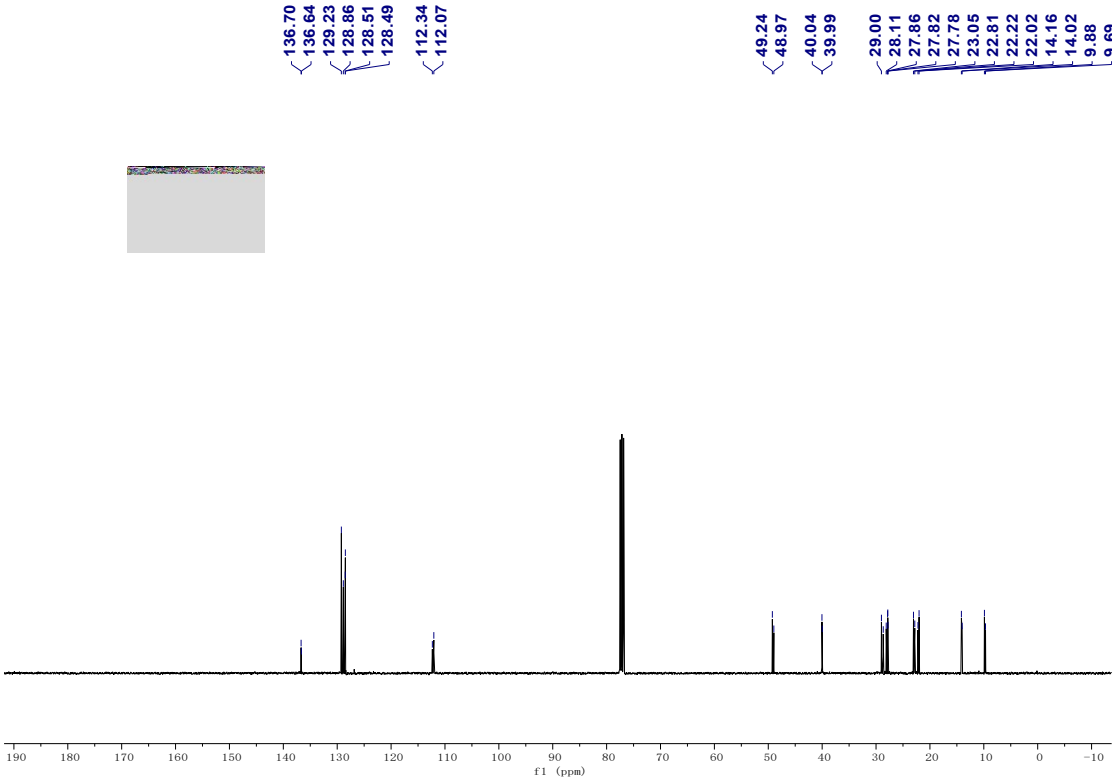
3.07
2.97
f1 (ppm)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129.23
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128.51
128.49
112.34
112.07
49.24
48.97
40.04
39.99
29.00
28.11
27.86
27.82
27.78
23.05
22.81
22.22
22.02
14.16
14.02
9.88
f1 (ppm)
7.46
7.45
7.43
7.42
7.35
7.34
7.34
7.32
7.32
7.30
7.05
7.03
7.01
6.96
6.96
6.94
4.54
4.52
4.43
4.41
4.39
4.38
4.37
4.36
4.34
3.72
3.71
3.70
3.70
3.69
3.69
3.68
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CN
CN
Ph
16
5.04
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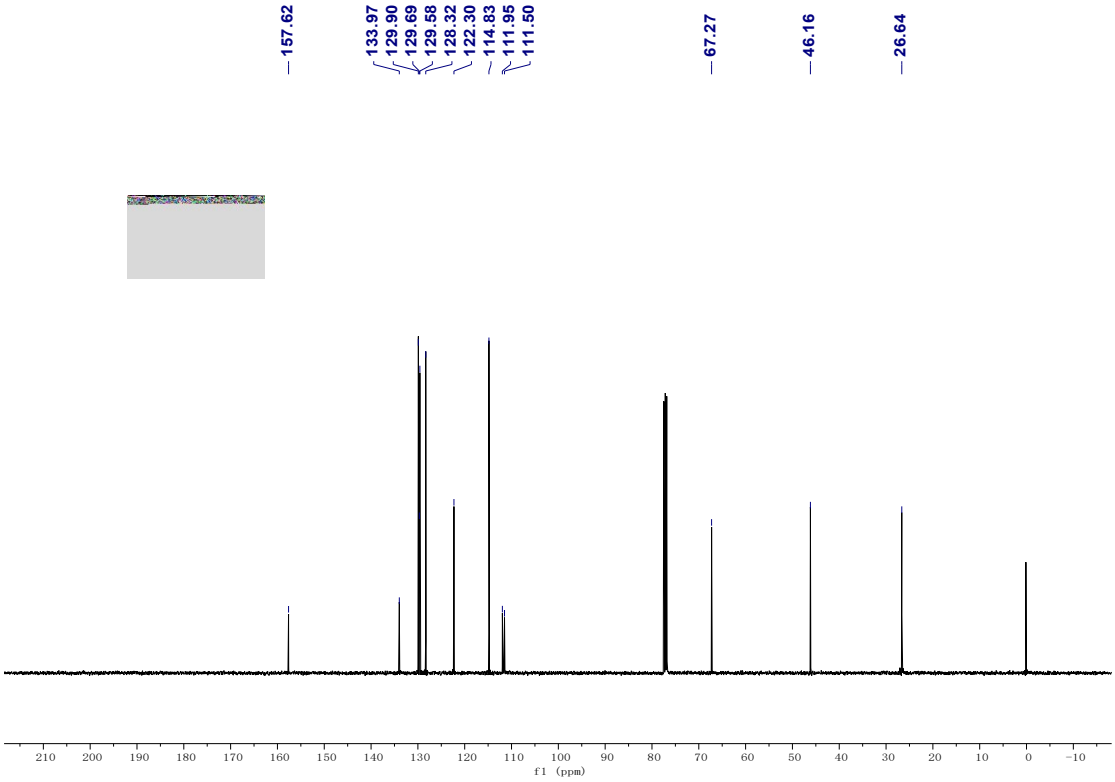
2.02
0.99
2.09
1.05
f1 (ppm)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129.69
129.58
128.32
122.30
114.83
111.95
111.50
67.27
46.16
26.64
f1 (ppm)


7.41
7.41
7.40
7.40
7.39
7.38
7.36
7.36
7.36
7.34
7.32
7.31
7.31
7.30
7.30
7.29
7.27
4.57
4.56
3.50
3.49
3.47
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3.29
3.29
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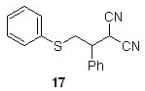
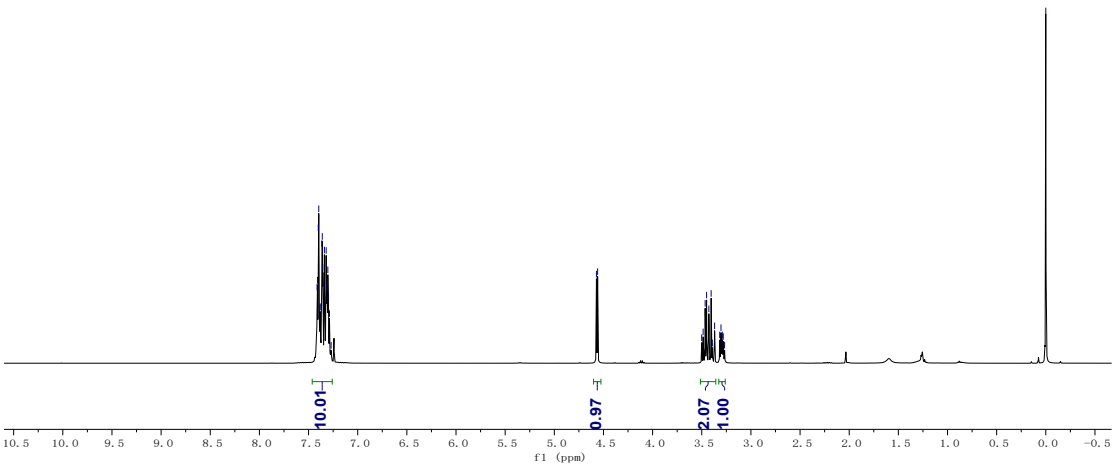
3.27
0.00


CN
CN
S
Ph

17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1.00
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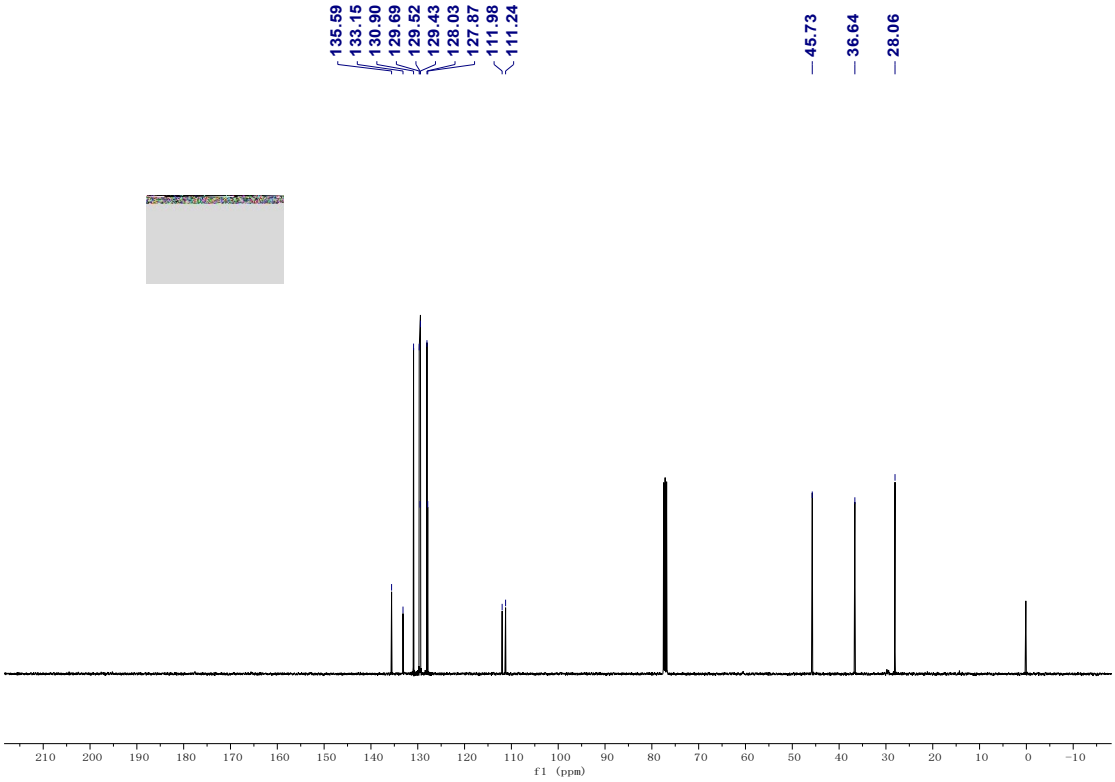
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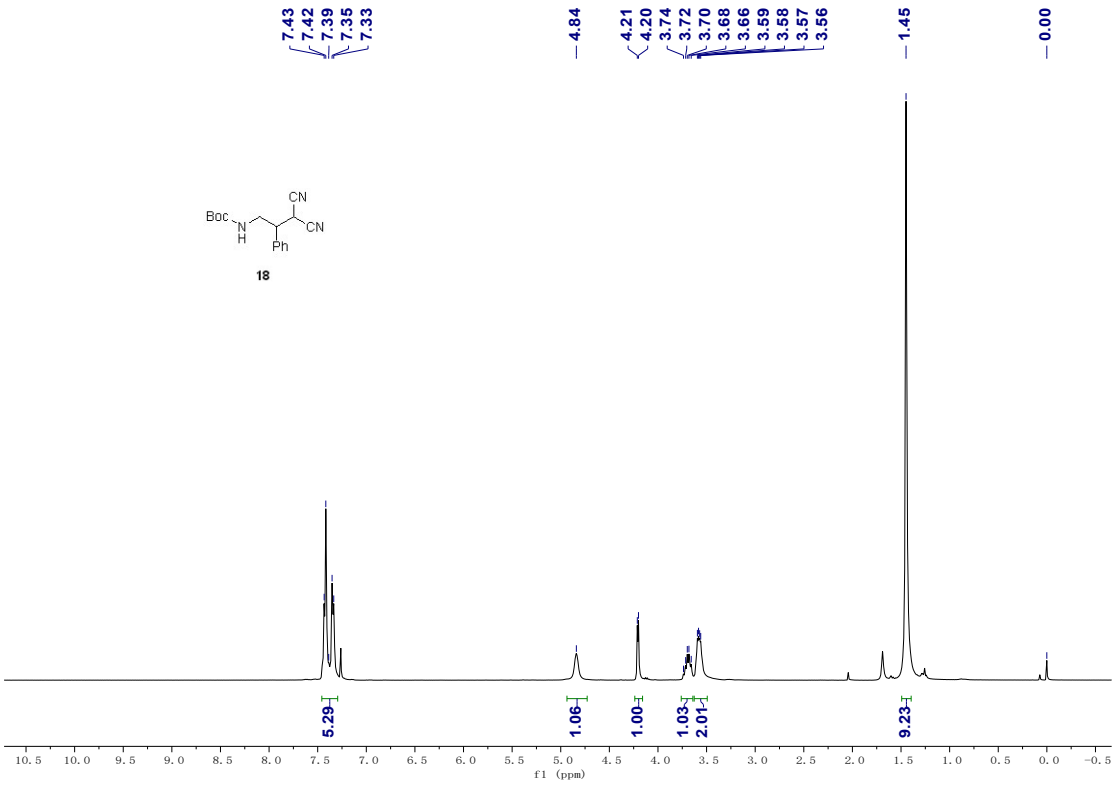
127.87
111.98
111.24
45.73
36.64
28.06
f1 (ppm)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4.20
3.74
3.72
3.70
3.68
3.66
3.59
3.58
3.57
3.56
1.45
0.00
CN
Boc
N
H
CN
Ph
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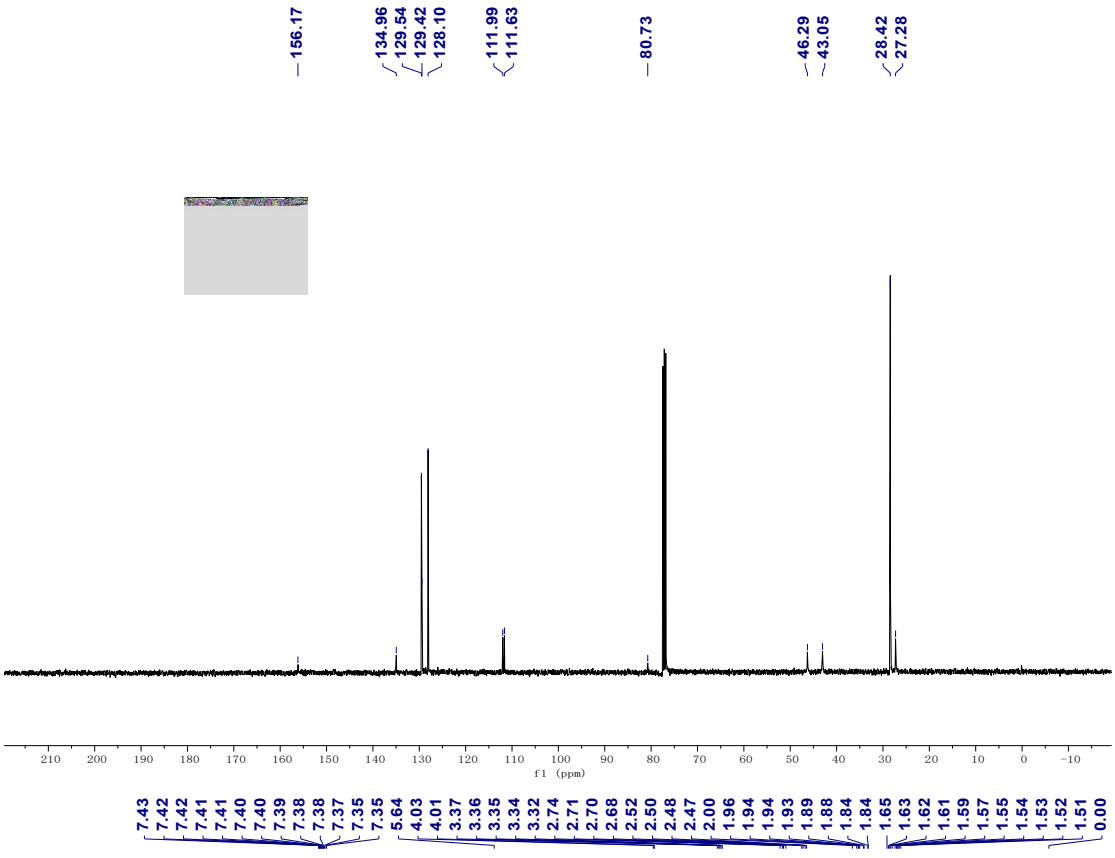
5.29
1.06
1.00
1.03
2.01
9.23
f1 (ppm)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134.96
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111.99
111.63
80.73
46.29
43.05
28.42
27.28
f1 (ppm)
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.37
7.35
7.35
5.64
4.03
4.01
3.37
3.36
3.35
3.34
3.32
2.74
2.71
2.70
2.68
2.52
2.50
2.48
2.47
2.00
1.96
1.94
1.94
1.93
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1.88
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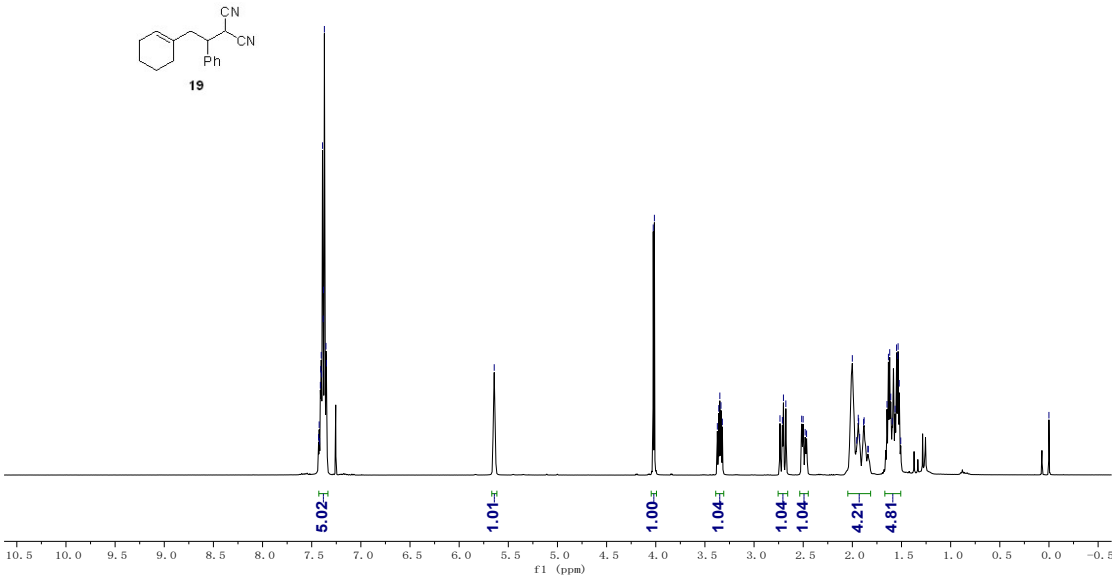
1.55
1.54
1.53
1.52
1.51
0.00


CN
CN
Ph
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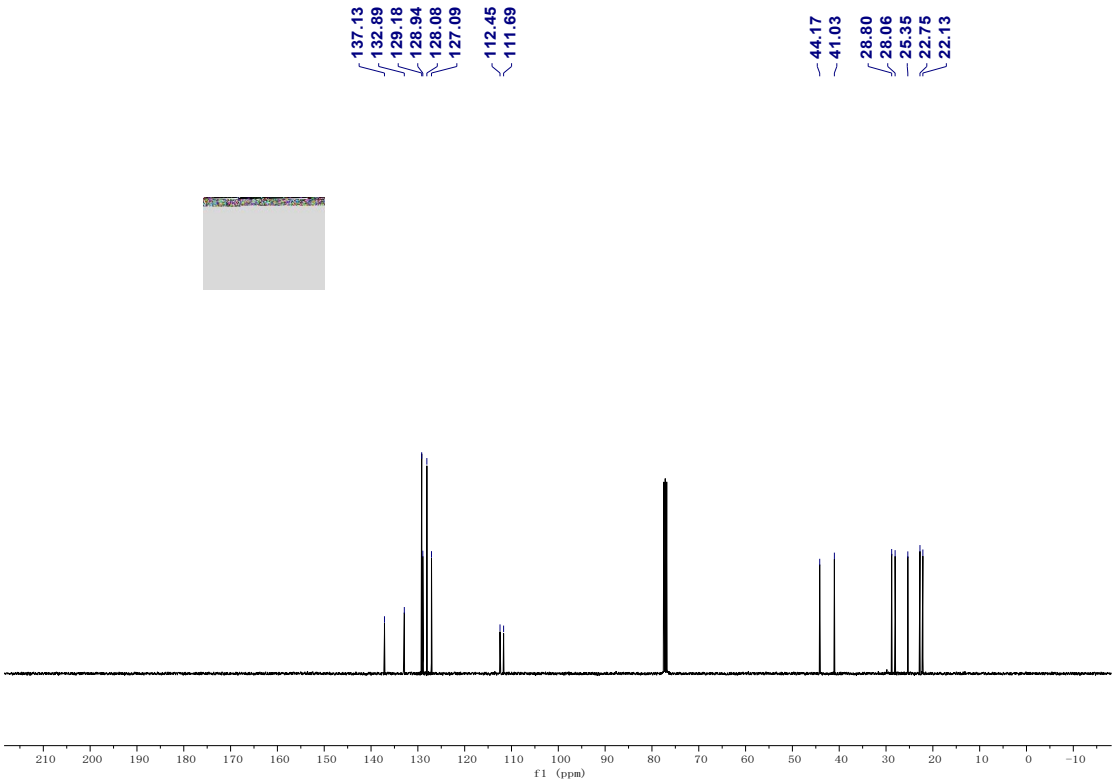
1.00
1.04
1.04
1.04
4.21
4.81
f1 (ppm)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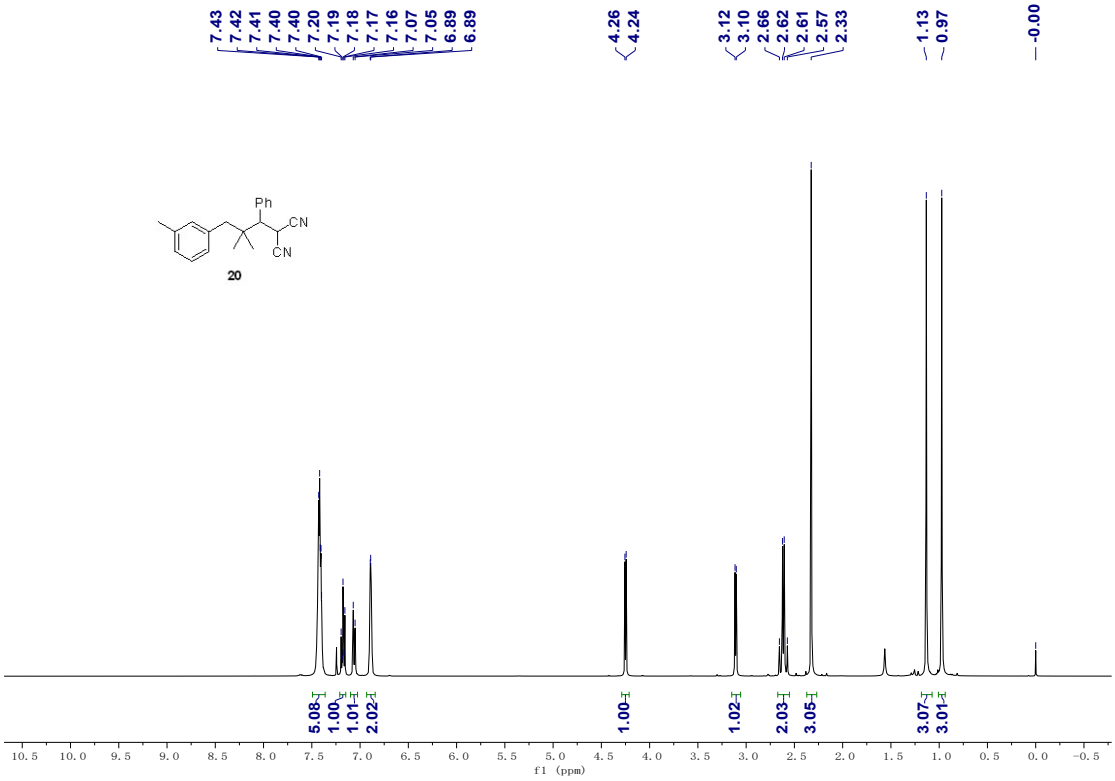
132.89
129.18
128.94
128.08
127.09
112.45
111.69
44.17
41.03
28.80
28.06
25.35
22.75
22.13
f1 (ppm)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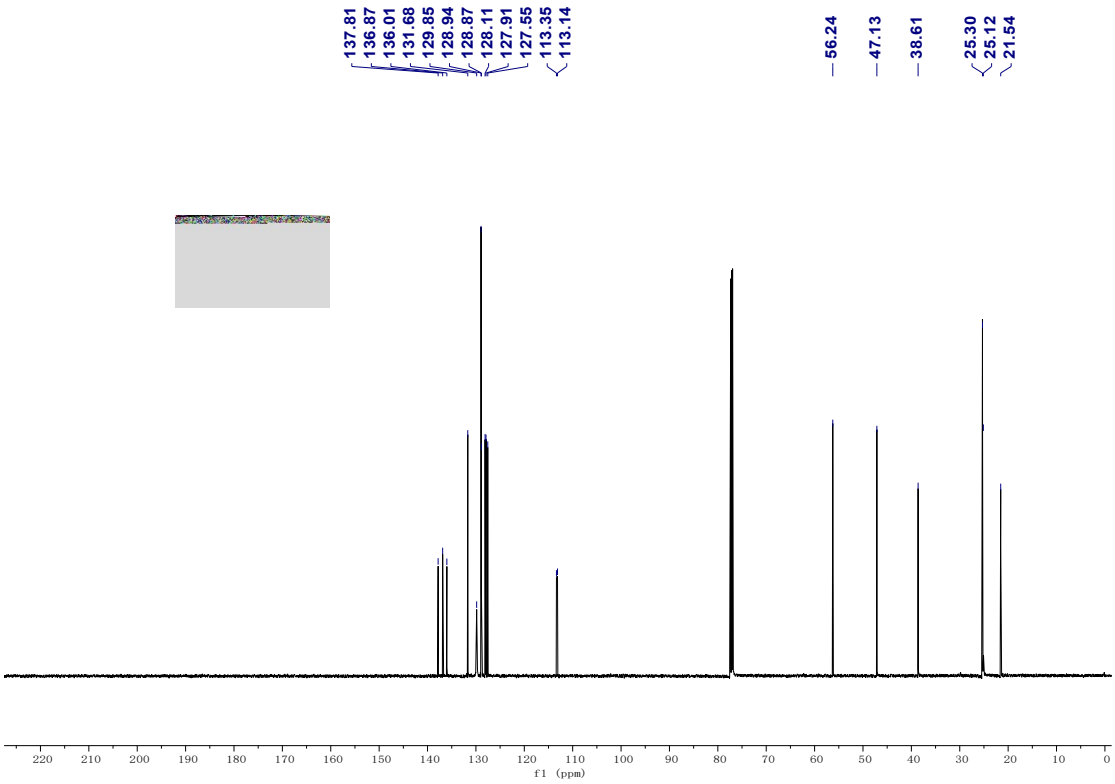
7.40
7.20
7.19
7.18
7.17
7.16
7.07
7.05
6.89
6.89
4.26
4.24
3.12
3.10
2.66
2.62
2.61
2.57
2.33
1.13
0.97
-0.00
Ph
CN
CN
20
5.08
1.00
1.01
2.02
1.00
1.02
2.03
3.05
3.07
3.01
f1 (ppm)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136.01
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129.85
128.94
128.87
128.11
127.91
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113.35
113.14
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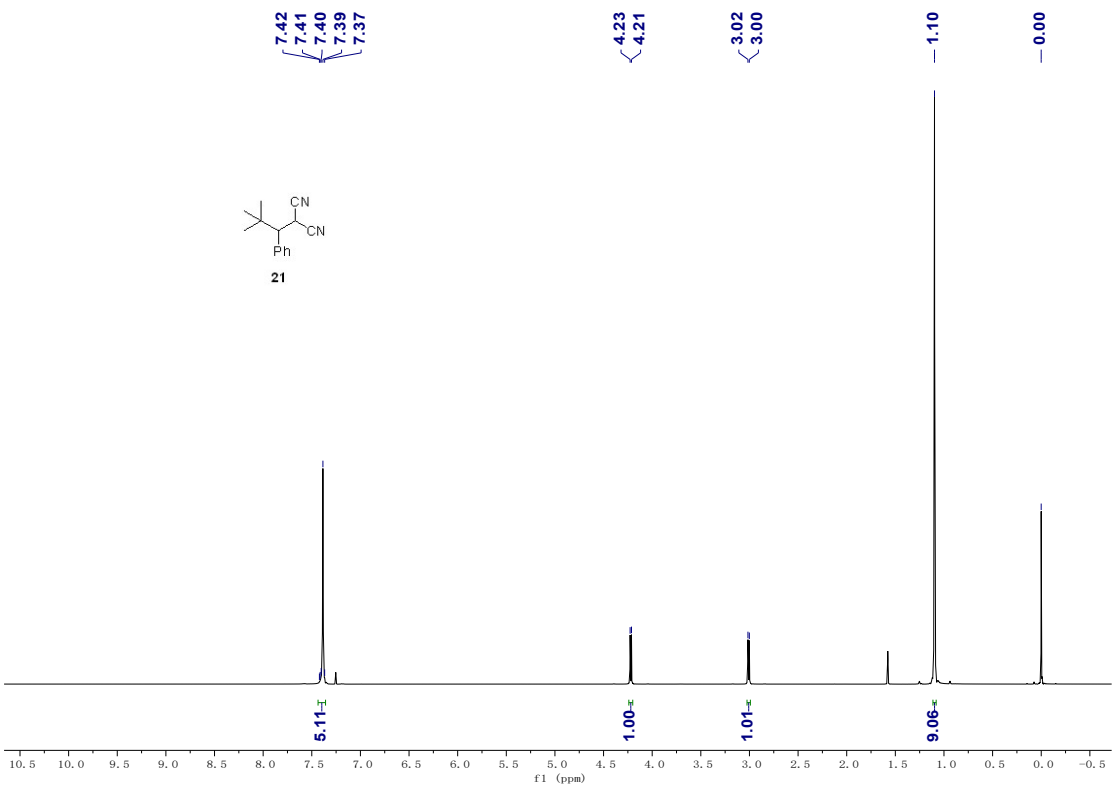
38.61
25.30
25.12
21.54
f1 (ppm)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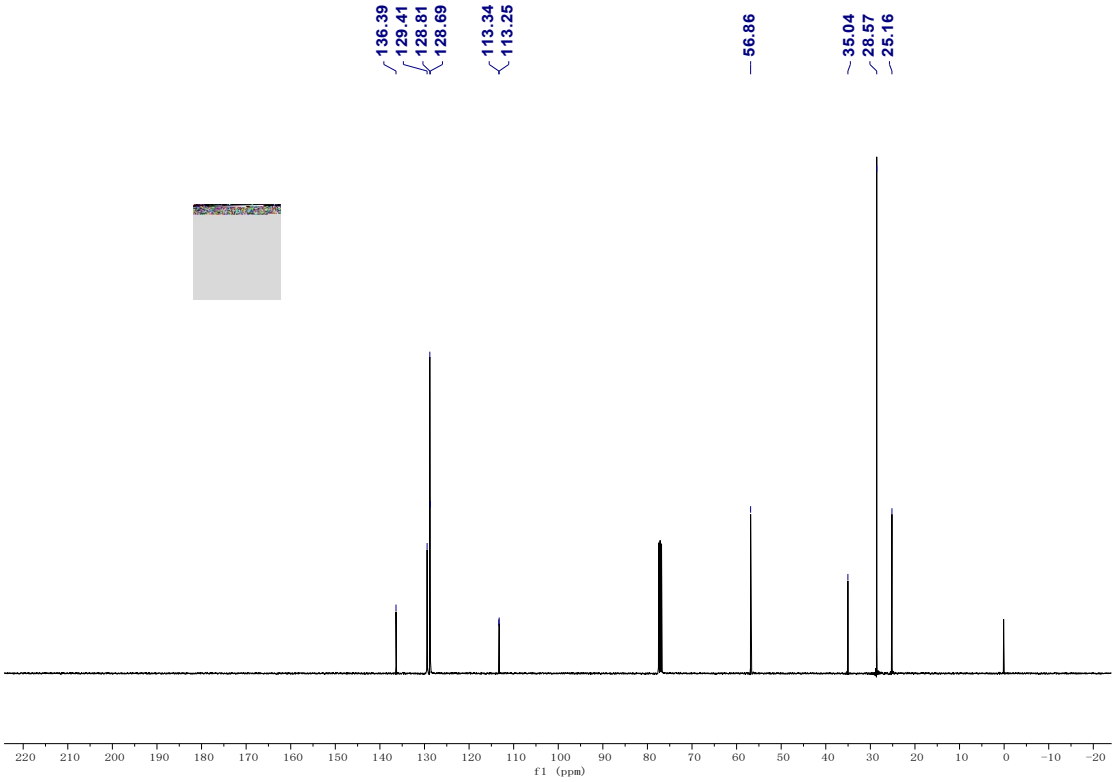
7.41
7.40
7.39
7.37
4.23
4.21
3.02
3.00
1.10
0.00
CN
CN
Ph
21
5.11
1.00
1.01
9.06
f1 (ppm)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113.25
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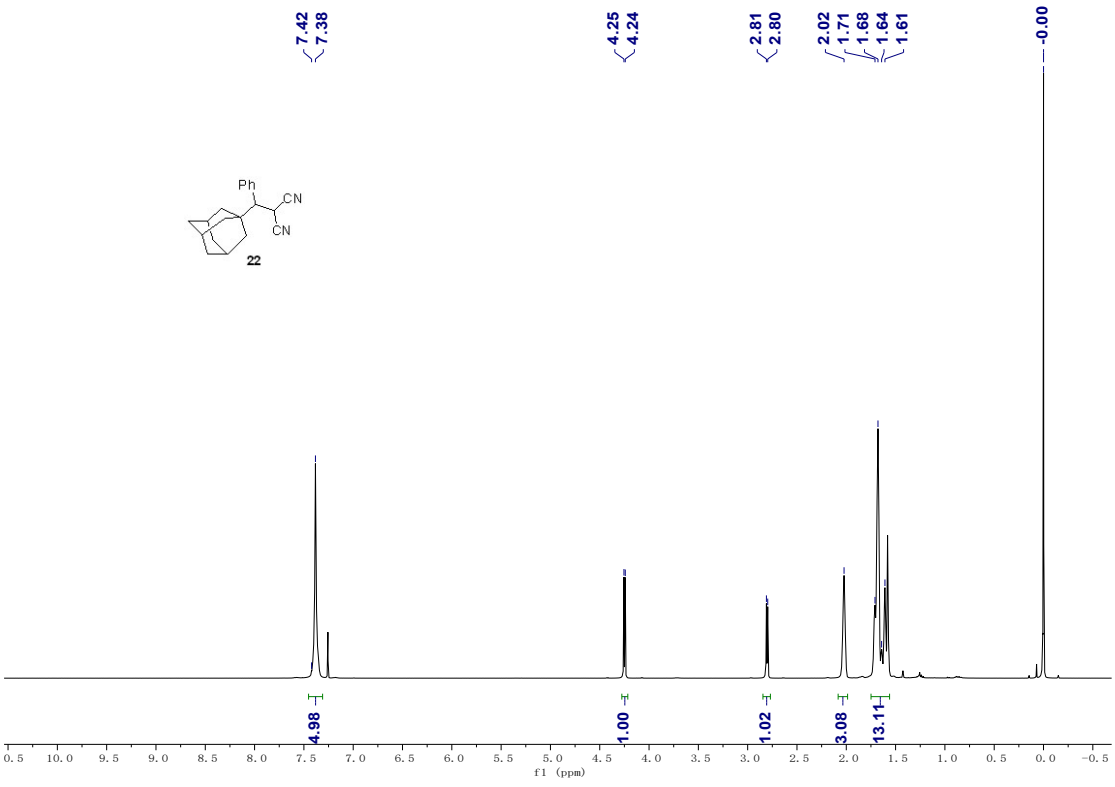
35.04
28.57
25.16
f1 (ppm)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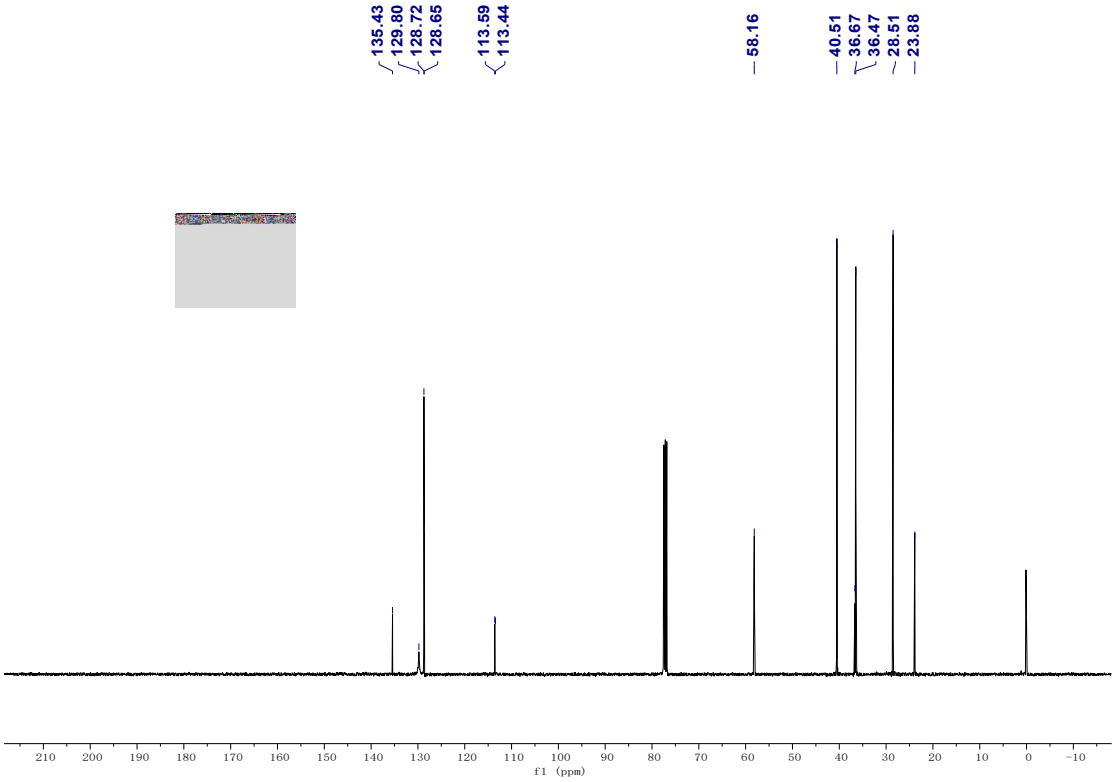
4.25
4.24
2.81
2.80
2.02
1.71
1.68
1.64
1.61
-0.00
Ph
CN
CN
22
4.98
1.00
1.02
3.08
13.11
f1 (ppm)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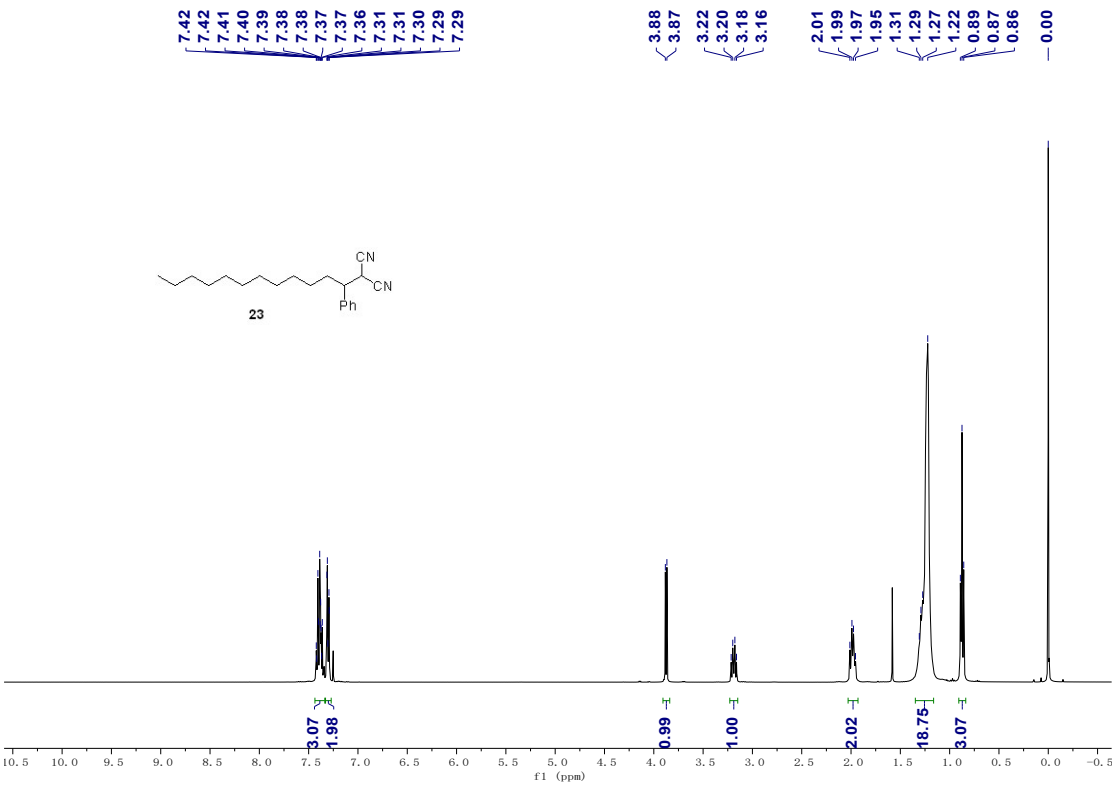
113.44
58.16
40.51
36.67
36.47
28.51
23.88
f1 (ppm)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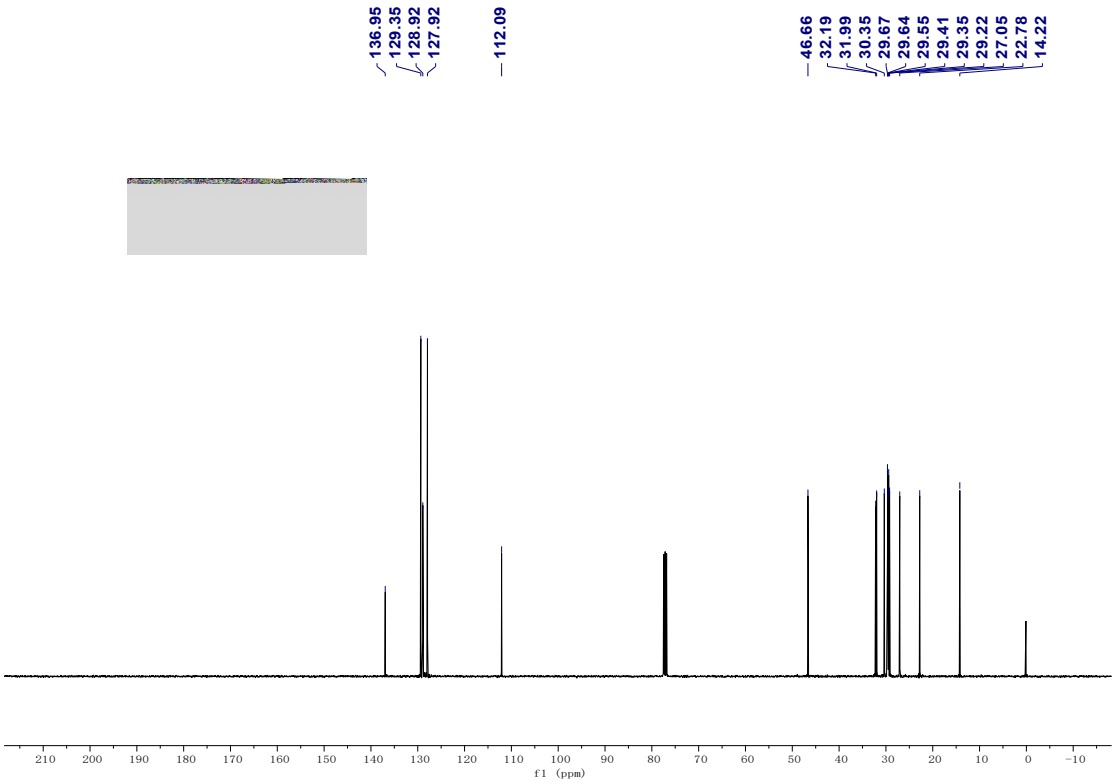
7.37
7.37
7.36
7.31
7.31
7.30
7.29
7.29
3.88
3.87
3.22
3.20
3.18
3.16
2.01
1.99
1.97
1.95
1.31
1.29
1.27
1.22
0.89
0.87
0.86
0.00
CN
CN
Ph
23
3.07
1.98
0.99
1.00
2.02
18.75
3.07
f1 (ppm)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127.92
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46.66
32.19
31.99
30.35
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29.64
29.55
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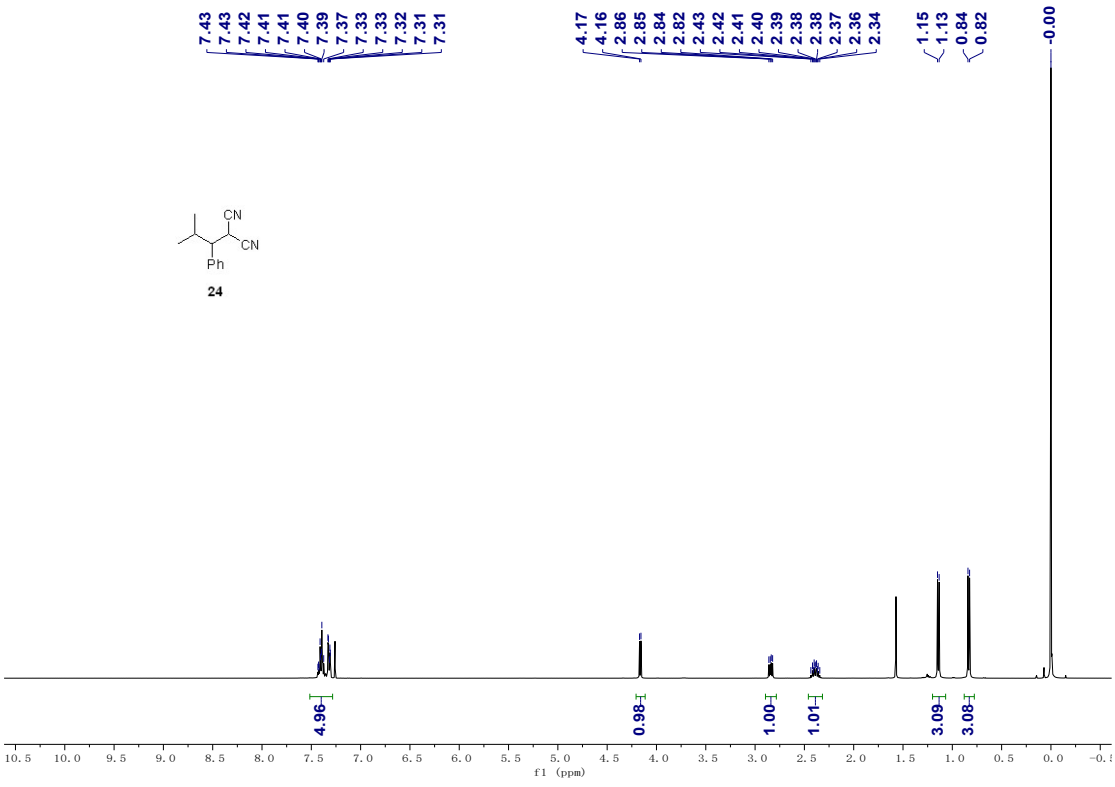
29.22
27.05
22.78
14.22
f1 (ppm)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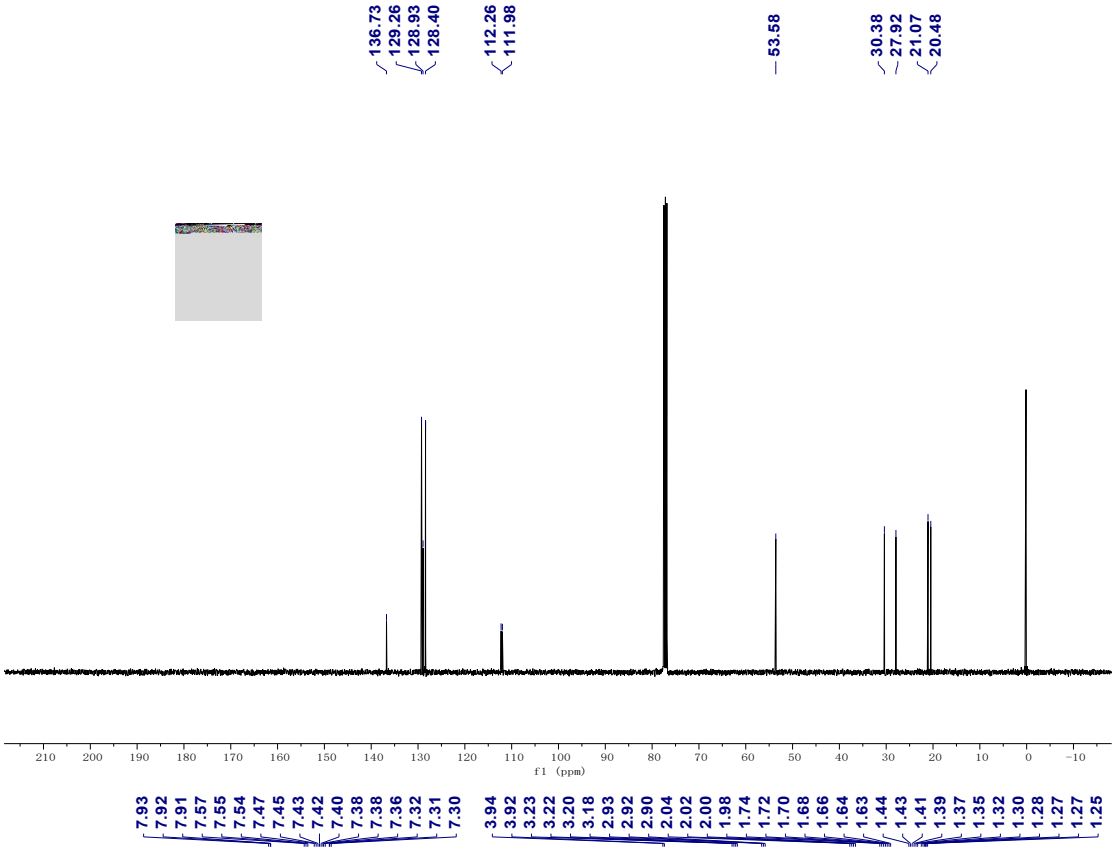
7.41
7.40
7.39
7.37
7.33
7.33
7.32
7.31
7.31
4.17
4.16
2.86
2.85
2.84
2.82
2.43
2.42
2.41
2.40
2.39
2.38
2.38
2.37
2.36
2.34
1.15
1.13
0.84
0.82
-0.00
CN
CN
Ph
24
4.96
0.98
1.00
1.01
3.09
3.08
f1 (ppm)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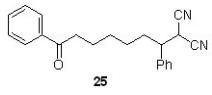
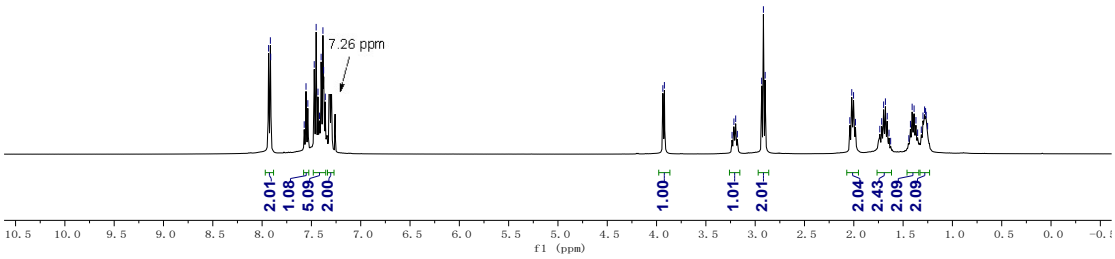
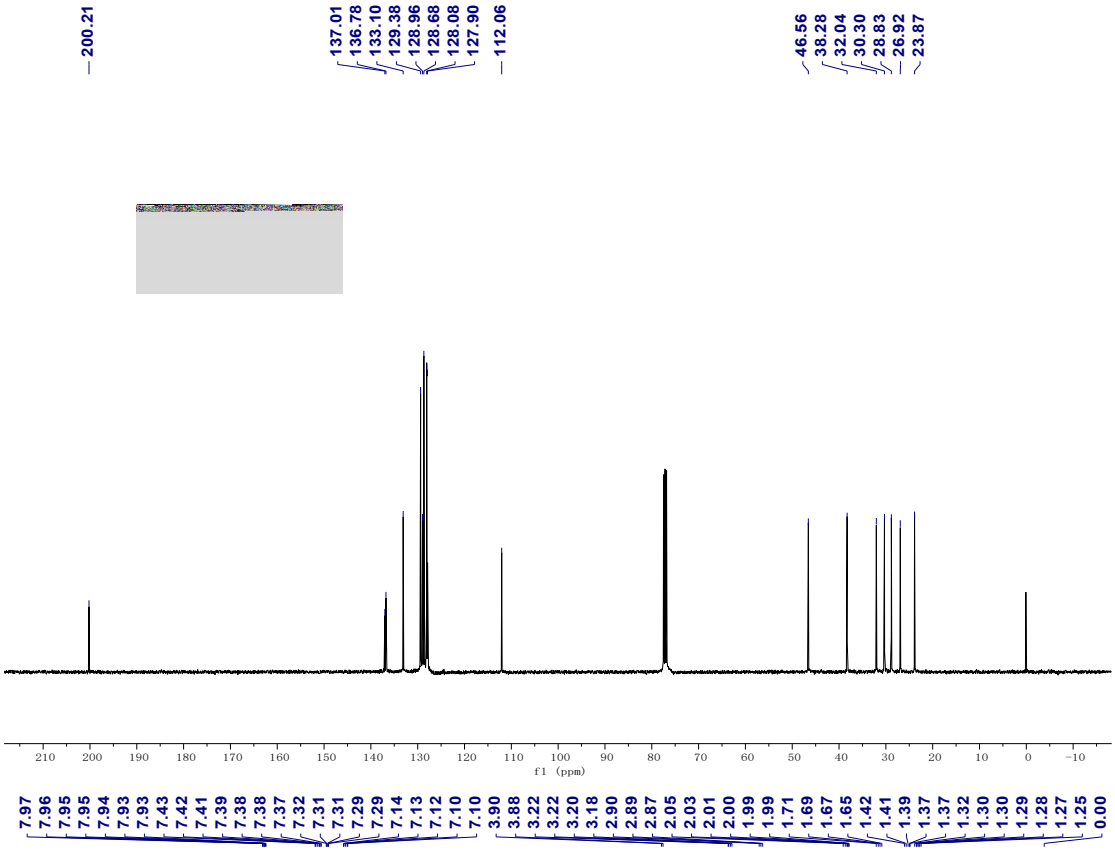
111.98
53.58
30.38
27.92
21.07
20.48
f1 (ppm)
7.93
7.92
7.91
7.57
7.55
7.54
7.47
7.45
7.43
7.42
7.40
7.38
7.38
7.36
7.32
7.31
7.30
3.94
3.92
3.23
3.22
3.20
3.18
2.93
2.92
2.90
2.04
2.02
2.00
1.98
1.74
1.72
1.70
1.68
1.66
1.64
1.63
1.44
1.43
1.41
1.39
1.37
1.35
1.32
1.30
1.28
1.27
1.27
1.25


CN
CN
Ph
O

25


7.26 ppm
2.01
1.08
5.09
2.00
1.00
1.01
2.01
2.04
2.43
2.09
2.05
f1 (ppm)


200.21
137.01
136.78
133.10
129.38
128.96
128.68
128.08
127.90
112.06
46.56
38.28
32.04
30.30
28.83
26.92
23.87
f1 (ppm)

7.97
7.96
7.95
7.95
7.94
7.93
7.93
7.43
7.42
7.41
7.39
7.38
7.38
7.37
7.32
7.31
7.31
7.29
7.29
7.14
7.13
7.12
7.10
7.10
3.90
3.88
3.22
3.22
3.20
3.18
2.90
2.89
2.87
2.05
2.03
2.01
2.00
1.99
1.99
1.71
1.69
1.67
1.65
1.42
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1.30
1.29
1.28
1.27
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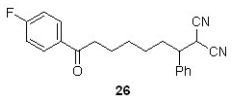
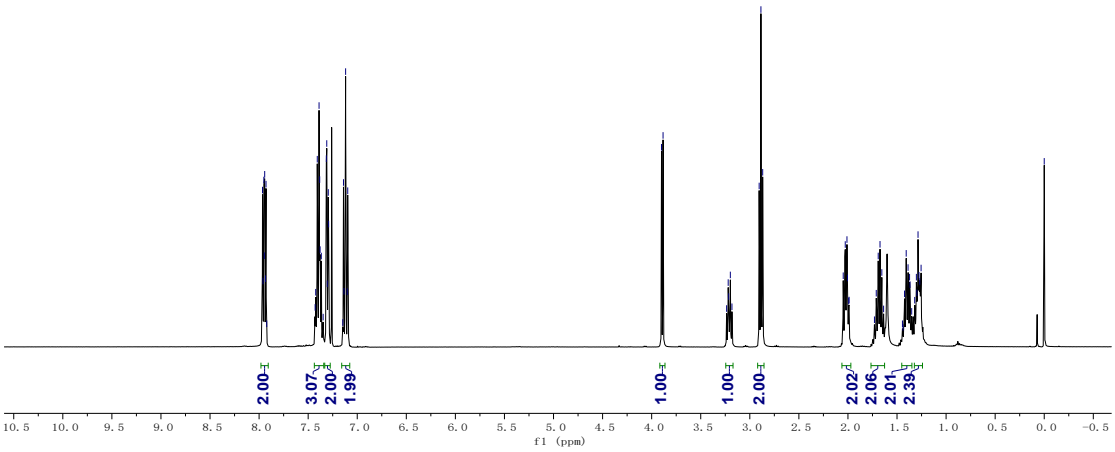
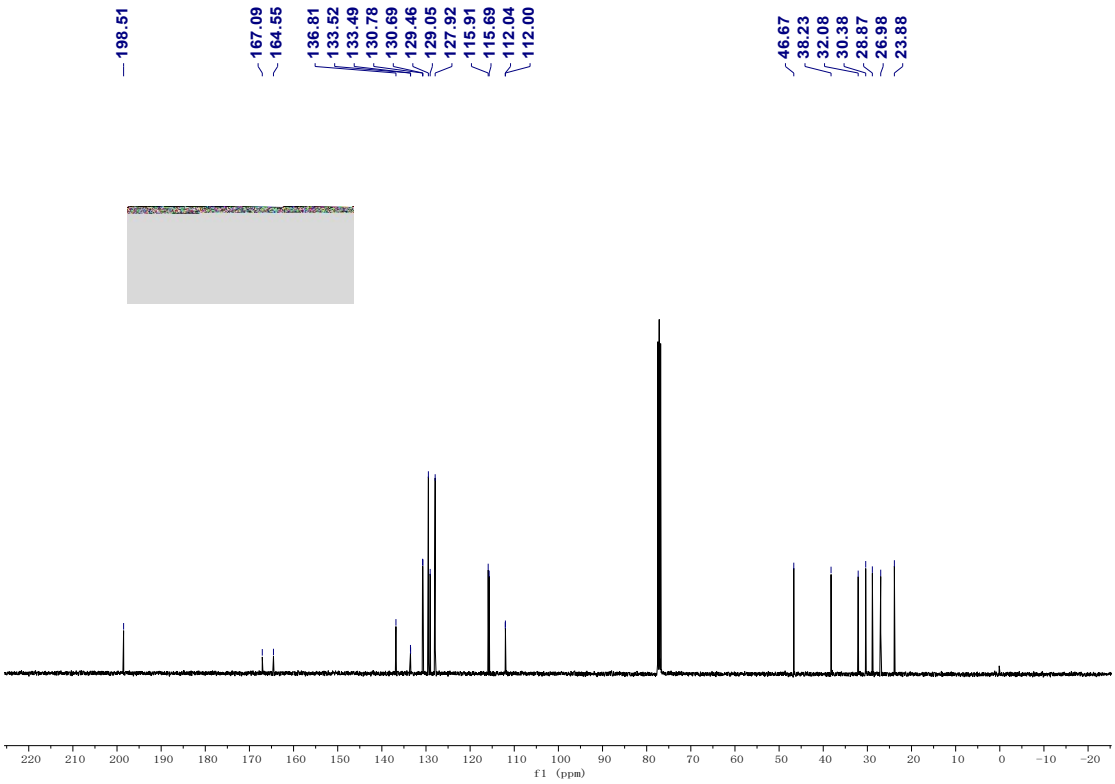

F
CN
CN
O
Ph

26


2.00
3.07
2.00
1.99
1.00
1.00
2.00
2.02
2.06
2.01
2.39
f1 (ppm)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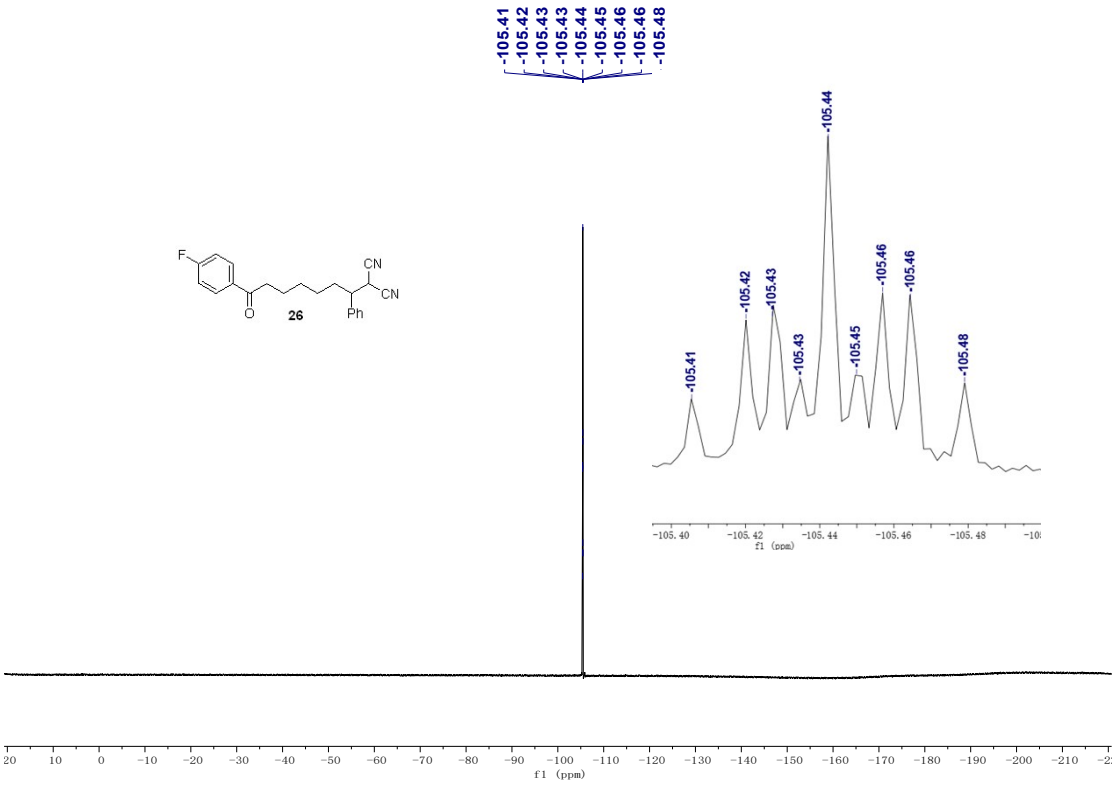
133.52
133.49
130.78
130.69
129.46
129.05
127.92
115.91
115.69
112.04
112.00
46.67
38.23
32.08
30.38
28.87
26.98
23.88
f1 (ppm)


-105.41
-105.42
-105.43
-105.43
-105.44
-105.45
-105.46
-105.46
-105.48
F
CN
CN
O
26
Ph
-105.44
-105.42
-105.43
-105.43
-105.46
-105.45
-105.46
-105.48
-105.41
f1 (ppm)


8.03
8.01
7.73
7.71
7.43
7.42
7.42
7.41
7.40
7.39
7.38
7.38
7.37
7.32
7.31
7.31
7.30
7.29
3.90
3.89
3.24
3.22
3.22
3.20
3.19
2.96
2.94
2.92
2.05
2.04
2.03
2.02
2.01
2.00
1.99
1.75
1.73
1.71
1.69
1.68
1.66
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1.46
1.44
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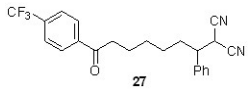
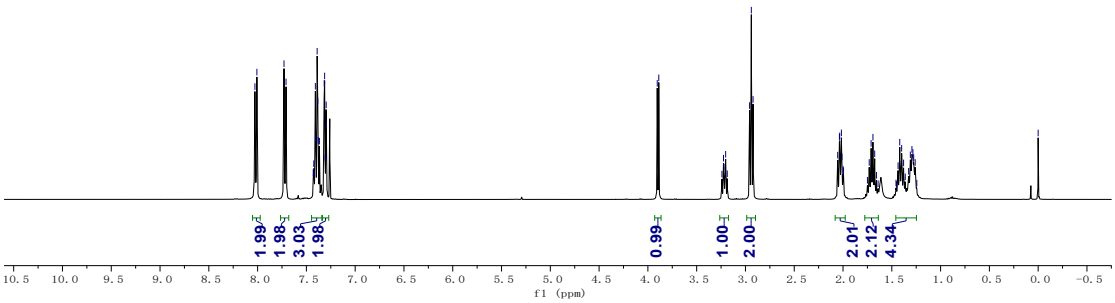
1.31
1.29
1.28
1.28
1.26
1.24
0.00

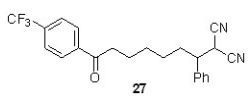

CF3
CN
CN
O
Ph

27


1.99
1.98
3.03
1.98
0.99
1.00
2.00
2.01
2.12
4.34
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 −0.5
f1 (ppm)

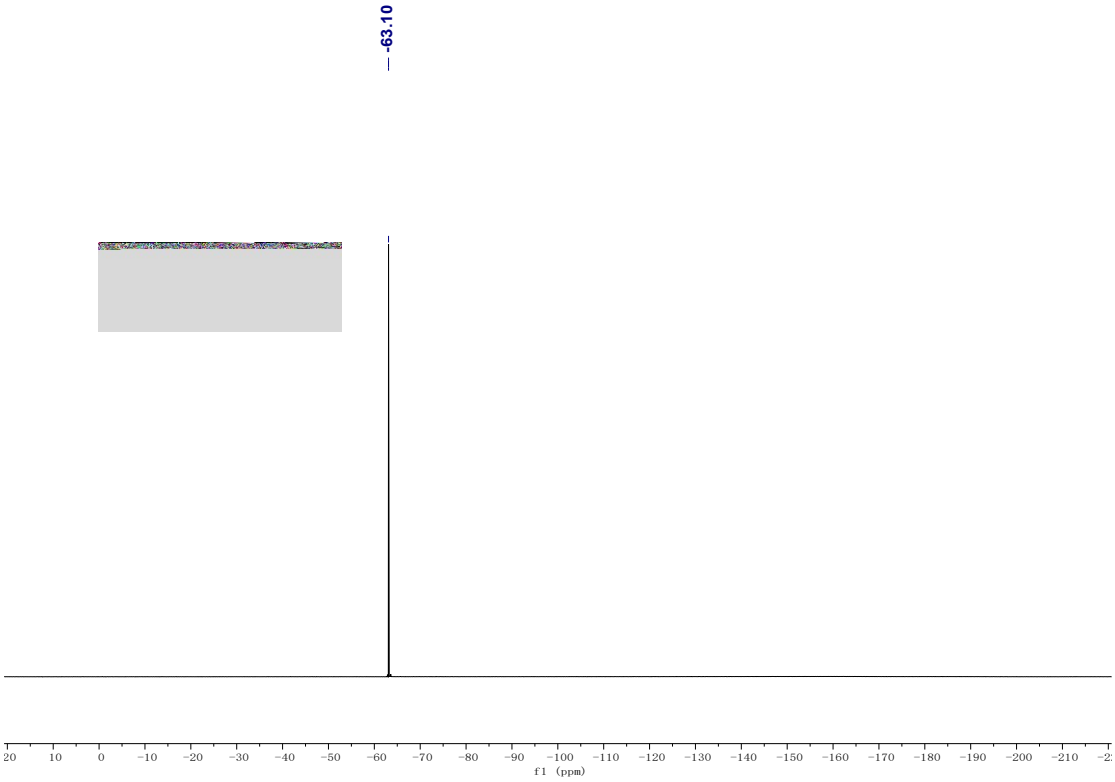

199.08
139.68
136.78
134.94
134.61
134.29
133.97
129.46
129.07
128.44
127.91
125.87
125.83
125.79
125.76
125.08
122.37
112.04
112.00


46.66
38.61
32.05
30.38
28.81
26.96
23.67


CF3
CN
CN
O
Ph

27

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)


-63.10
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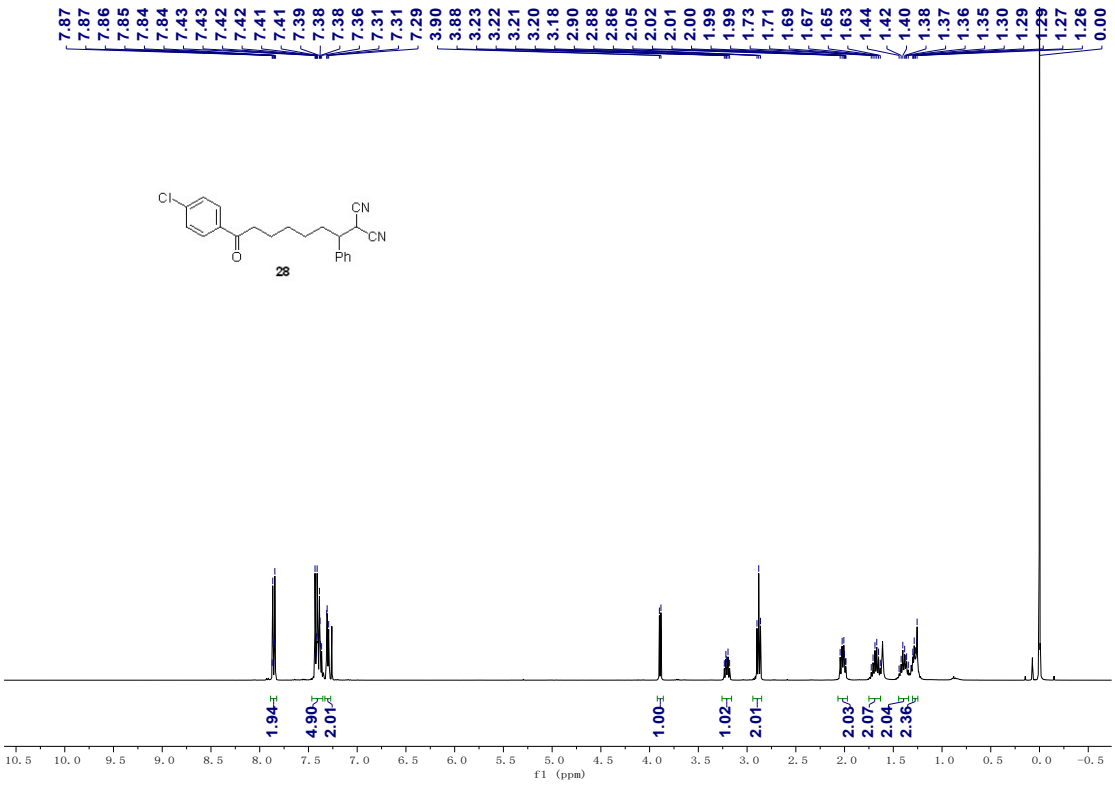
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7.43
7.43
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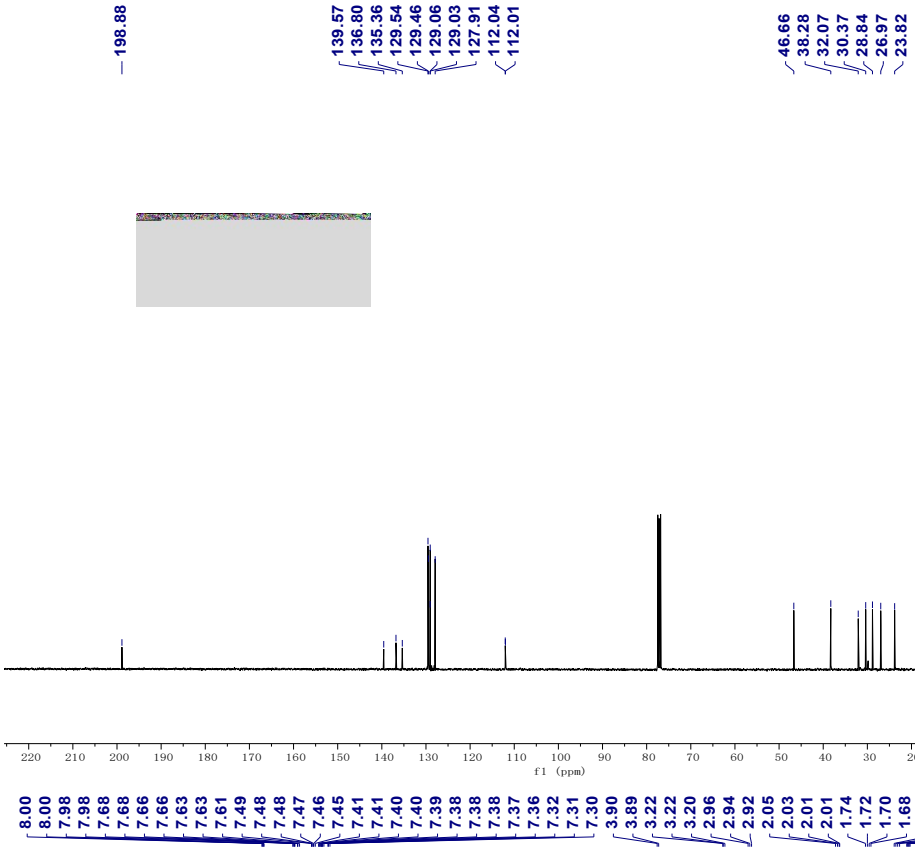
7.41
7.41
7.39
7.38
7.38
7.36
7.31
7.31
7.29
3.90
3.88
3.23
3.22
3.21
3.20
3.18
2.90
2.88
2.86
2.05
2.02
2.01
2.00
1.99
1.99
1.73
1.71
1.69
1.67
1.65
1.63
1.44
1.42
1.40
1.38
1.37
1.36
1.35
1.30
1.29
1.29
1.27
1.26
0.00
Cl
O
CN
CN
Ph
28
1.94
4.90
2.01
1.00
1.02
2.01
2.03
2.07
2.04
2.36
f1 (ppm)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139.57
136.80
135.36
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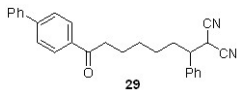
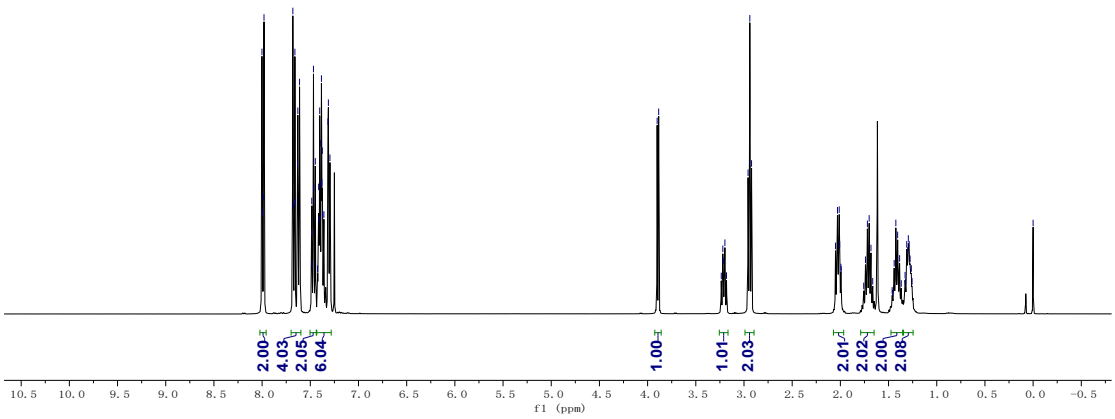
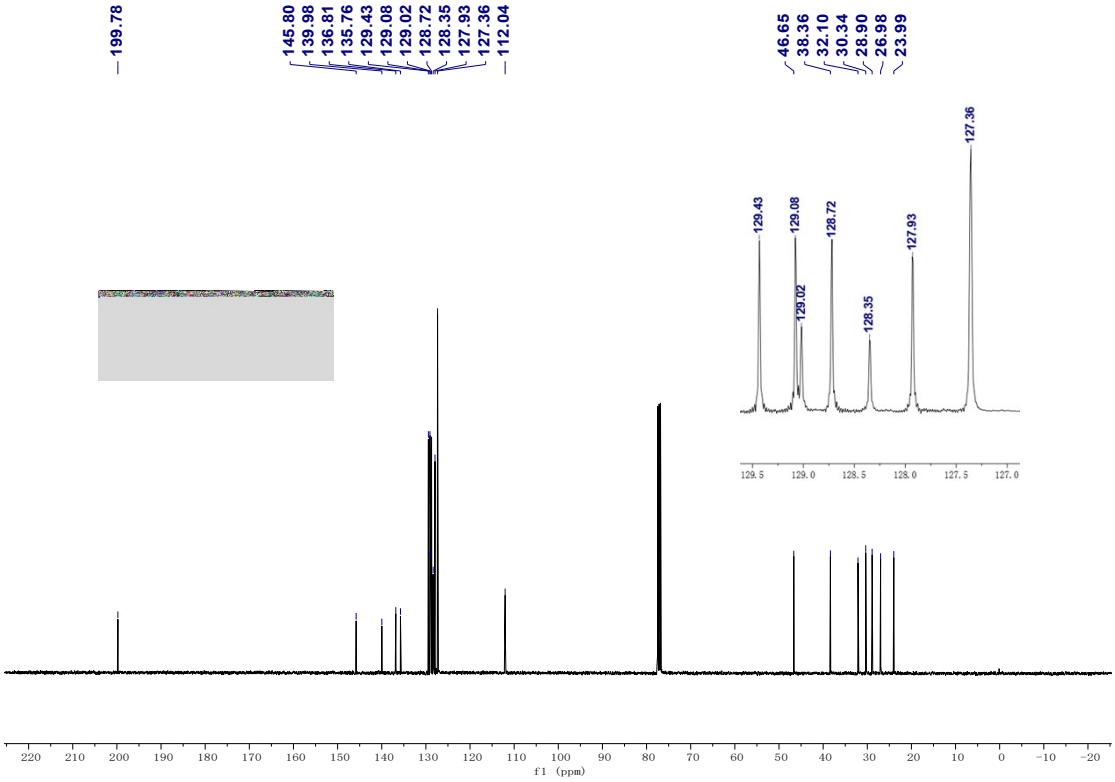
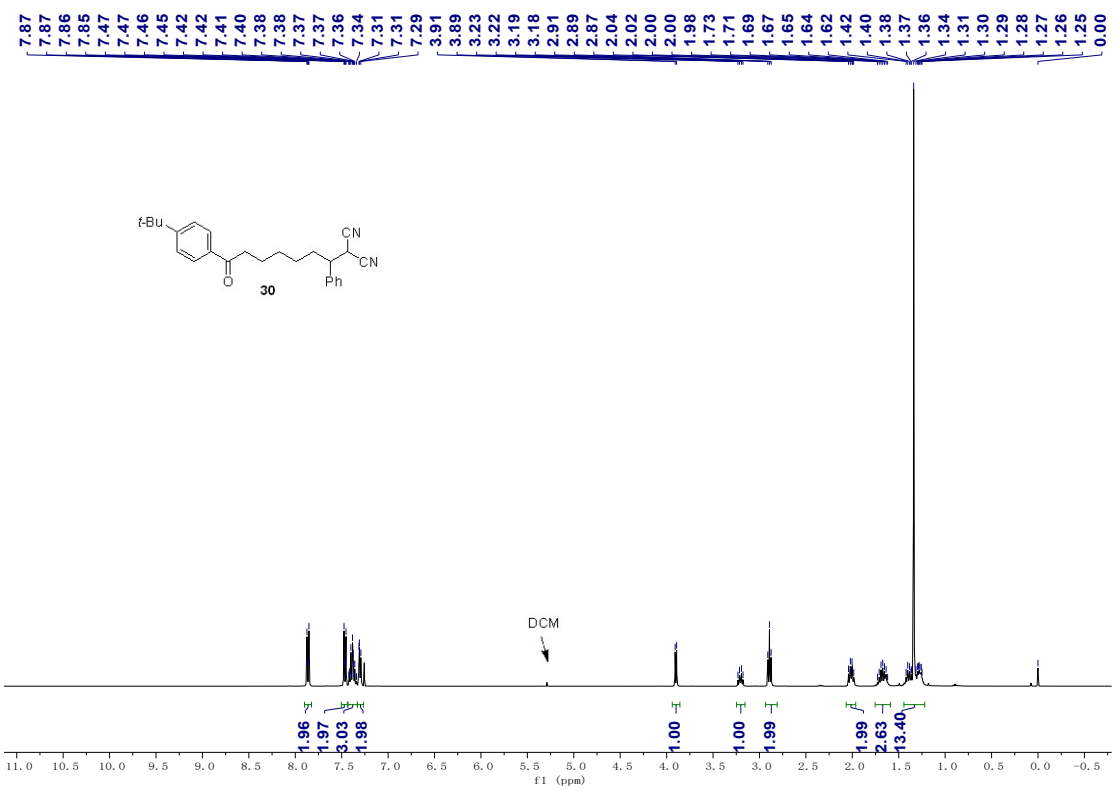
129.46
129.06
129.03
127.91
112.04
112.01
46.66
38.28
32.07
30.37
28.84
26.97
23.82
f1 (ppm)
8.00
8.00
7.98
7.98
7.68
7.68
7.66
7.66
7.63
7.63
7.61
7.49
7.48
7.48
7.47
7.46
7.45
7.41
7.41
7.40
7.40
7.39
7.38
7.38
7.38
7.37
7.36
7.32
7.31
7.30
3.90
3.89
3.22
3.22
3.20
2.96
2.94
2.92
2.05
2.03
2.01
2.01
1.74
1.72
1.70
1.68
1.44
1.42
1.41
1.39
1.31
1.30
1.29
1.29
1.28
0.00

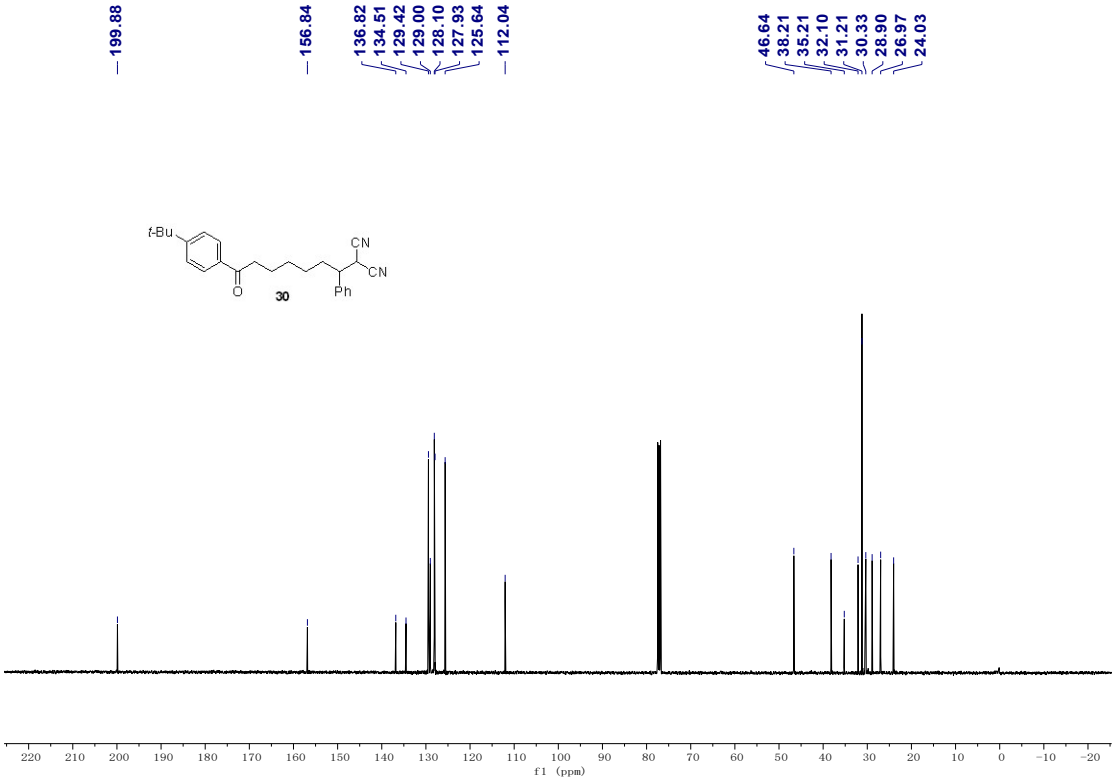

Ph
CN
CN
O
Ph

29


2.00
4.03
2.05
6.04
1.00
1.01
2.03
2.01
2.02
2.00
2.08
f1 (ppm)


199.78
145.80
139.98
136.81
135.76
129.43
129.08
129.02
128.72
128.35
127.93
127.36
112.04
46.65
38.36
32.10
30.34
28.90
26.98
23.99
f1 (ppm)


t-Bu
CN
CN
Ph
O
30
DCM
f1 (ppm)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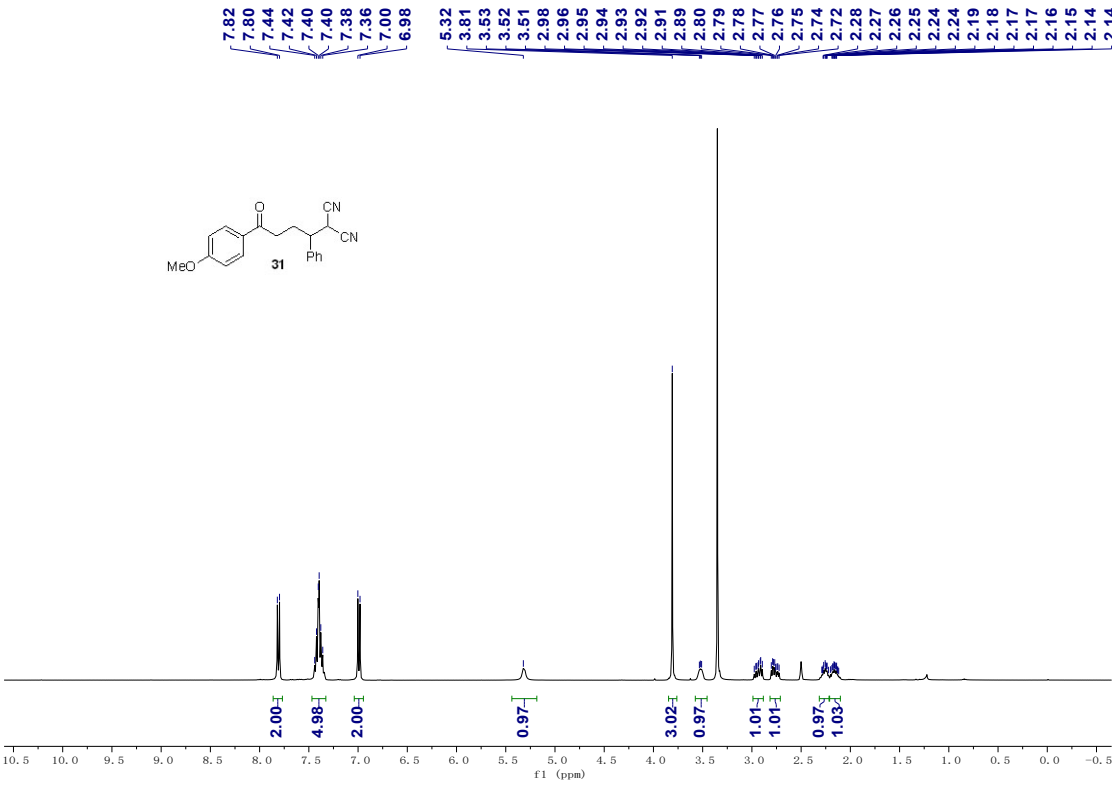
129.00
128.10
127.93
125.64
112.04
46.64
38.21
35.21
32.10
31.21
30.33
28.90
26.97
24.03
t-Bu
CN
CN
O
Ph
30
f1 (ppm)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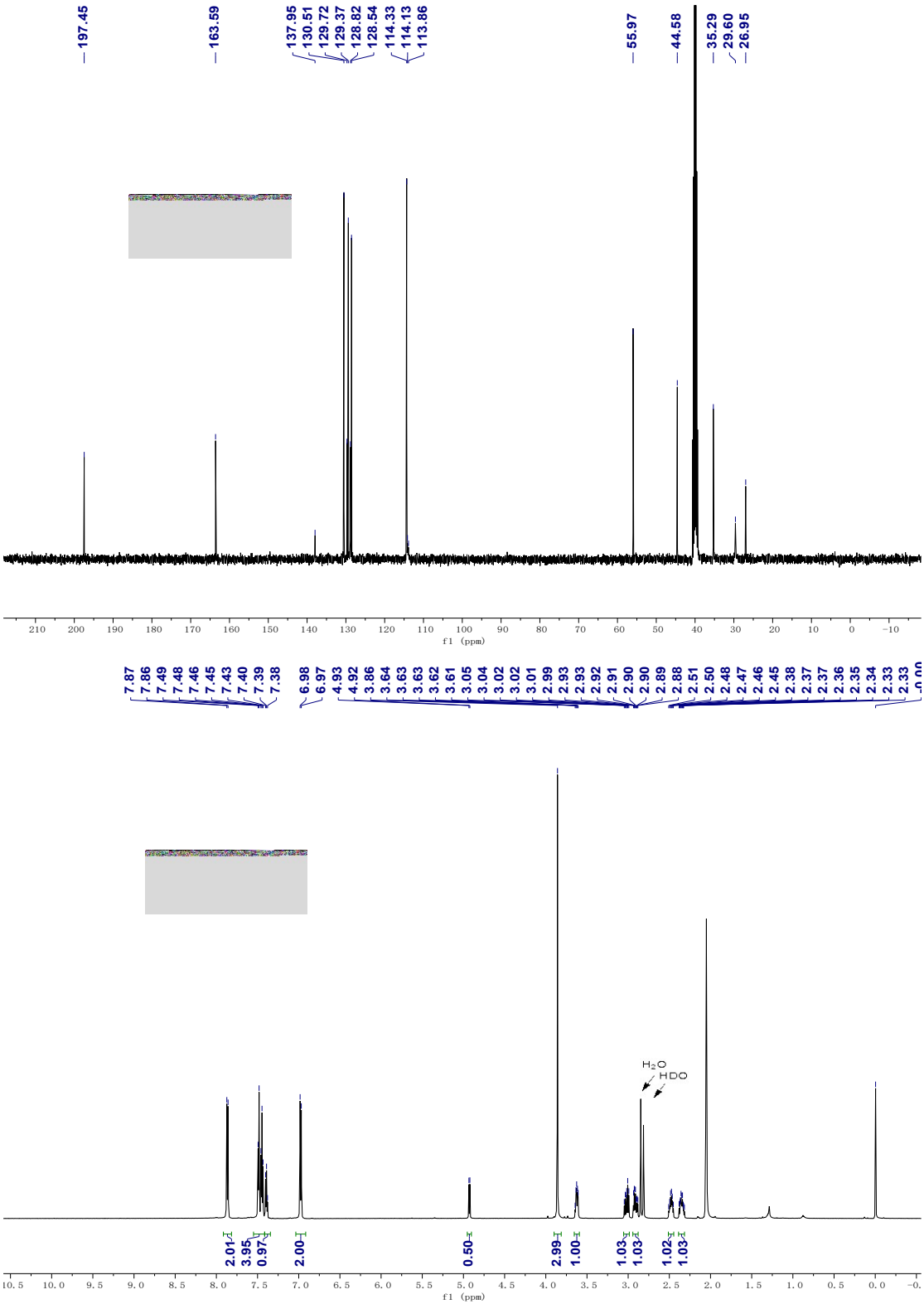
7.80
7.44
7.42
7.40
7.40
7.38
7.36
7.00
6.98
5.32
3.81
3.53
3.52
3.51
2.98
2.96
2.95
2.94
2.93
2.92
2.91
2.89
2.80
2.79
2.78
2.77
2.76
2.75
2.74
2.72
2.28
2.27
2.26
2.25
2.24
2.24
2.19
2.18
2.17
2.17
2.16
2.15
2.14
O
CN
CN
MeO
Ph
31
2.00
4.98
2.00
0.97
3.02
0.97
1.01
1.01
0.97
1.03
f1 (ppm)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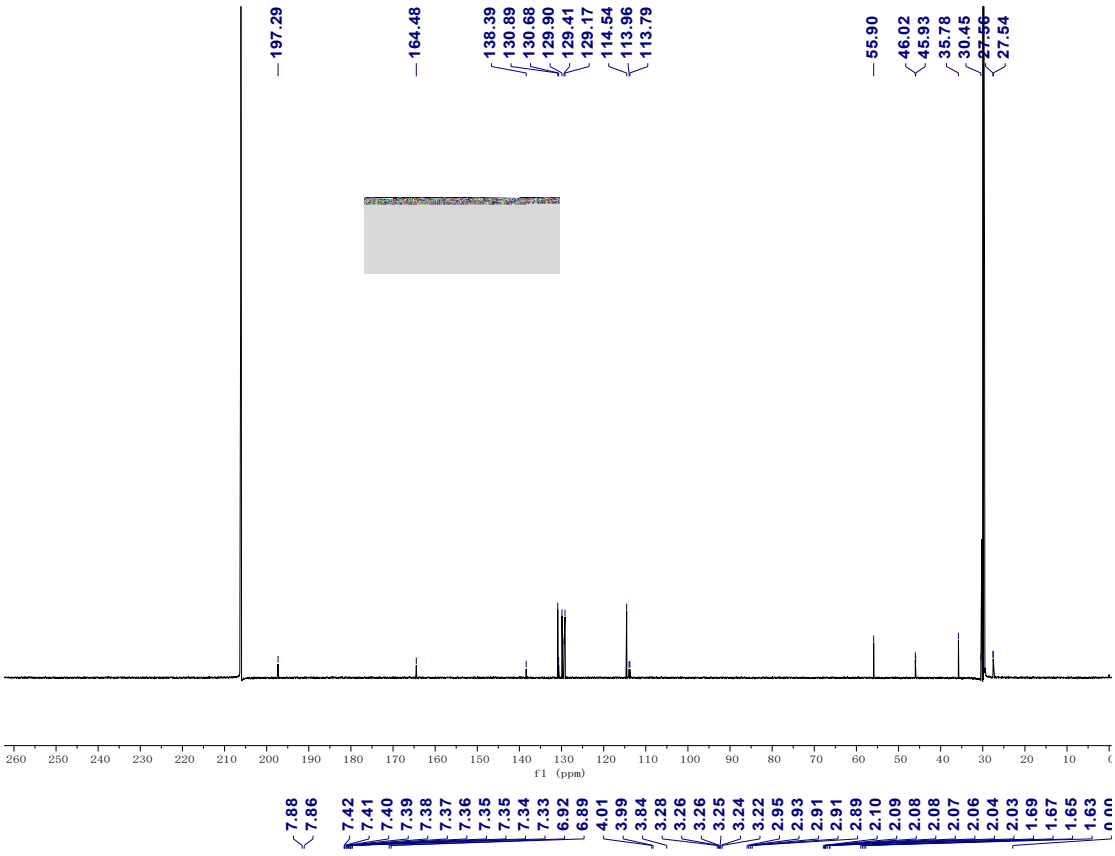
129.37
128.82
128.54
114.33
114.13
113.86
55.97
44.58
35.29
29.60
26.95
f1 (ppm)
7.87
7.86
7.49
7.48
7.46
7.45
7.43
7.40
7.39
7.38
6.98
6.97
4.93
4.92
3.86
3.64
3.63
3.63
3.62
3.61
3.05
3.04
3.02
3.02
3.01
2.99
2.93
2.93
2.92
2.91
2.90
2.90
2.89
2.88
2.51
2.50
2.48
2.47
2.46
2.45
2.38
2.37
2.37
2.36
2.35
2.34
2.33
2.33
H2O
HDO
2.01
3.95
0.97
2.00
0.50
2.99
1.00
1.03
1.03
1.02
1.03
f1 (ppm)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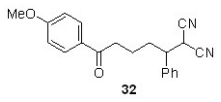
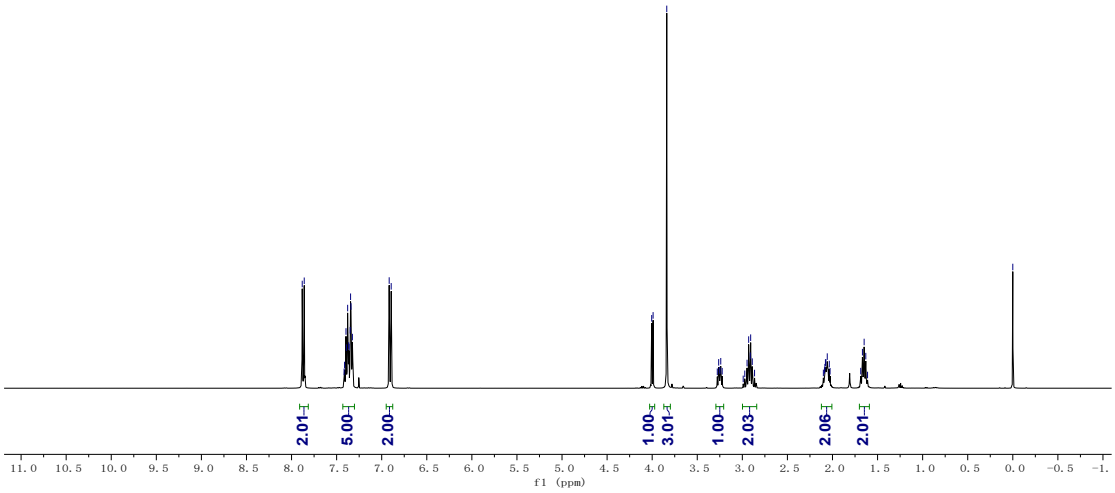
129.90
129.41
129.17
114.54
113.96
113.79
55.90
46.02
45.93
35.78
30.45
27.56
27.54
f1 (ppm)
7.88
7.86
7.42
7.41
7.40
7.39
7.38
7.37
7.36
7.35
7.35
7.34
7.33
6.92
6.89
4.01
3.99
3.84
3.28
3.26
3.26
3.25
3.24
3.22
2.95
2.93
2.91
2.91
2.89
2.10
2.09
2.08
2.08
2.07
2.06
2.04
2.03
1.69
1.67
1.65
1.63


MeO
CN
CN
O
Ph

32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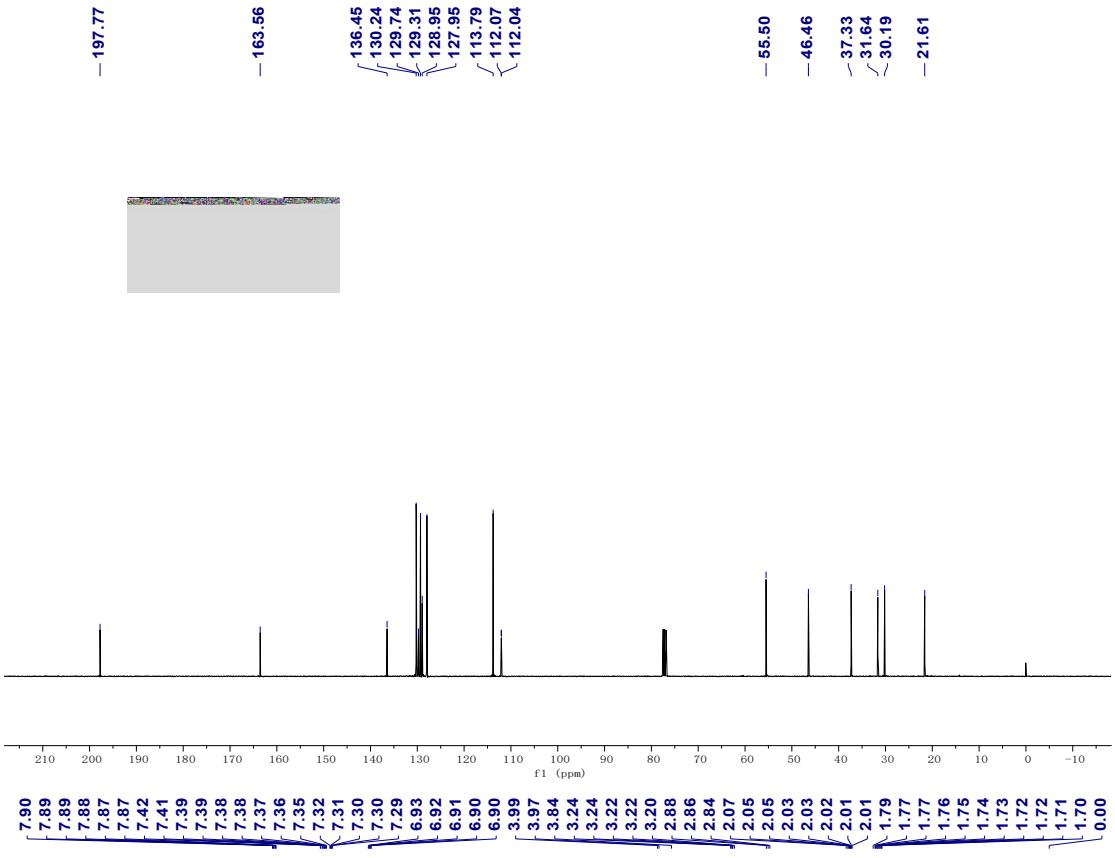
5.00
2.00
1.00
3.01
1.00
2.03
2.06
2.01
f1 (ppm)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130.24
129.74
129.31
128.95
127.95
113.79
112.07
112.04
55.50
46.46
37.33
31.64
30.19
21.61
f1 (ppm)
7.90
7.89
7.89
7.88
7.87
7.87
7.42
7.41
7.39
7.39
7.38
7.38
7.37
7.36
7.35
7.32
7.31
7.30
7.30
7.29
6.93
6.92
6.91
6.90
6.90
3.99
3.97
3.84
3.24
3.24
3.22
3.22
3.20
2.88
2.86
2.84
2.07
2.05
2.05
2.03
2.03
2.02
2.01
2.01
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1.77
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1.73
1.72
1.72
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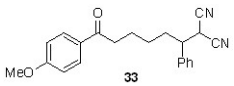
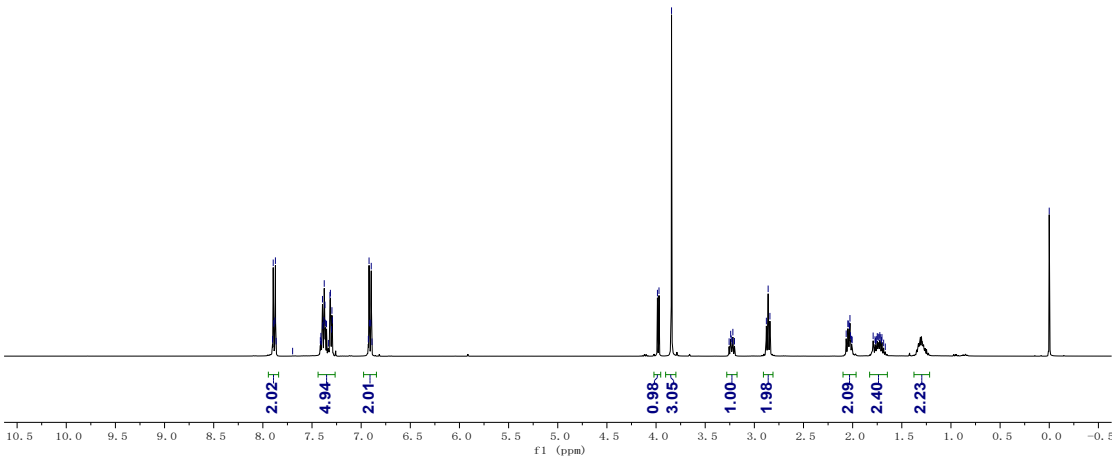
1.70
0.00


O
CN
CN
MeO
Ph

33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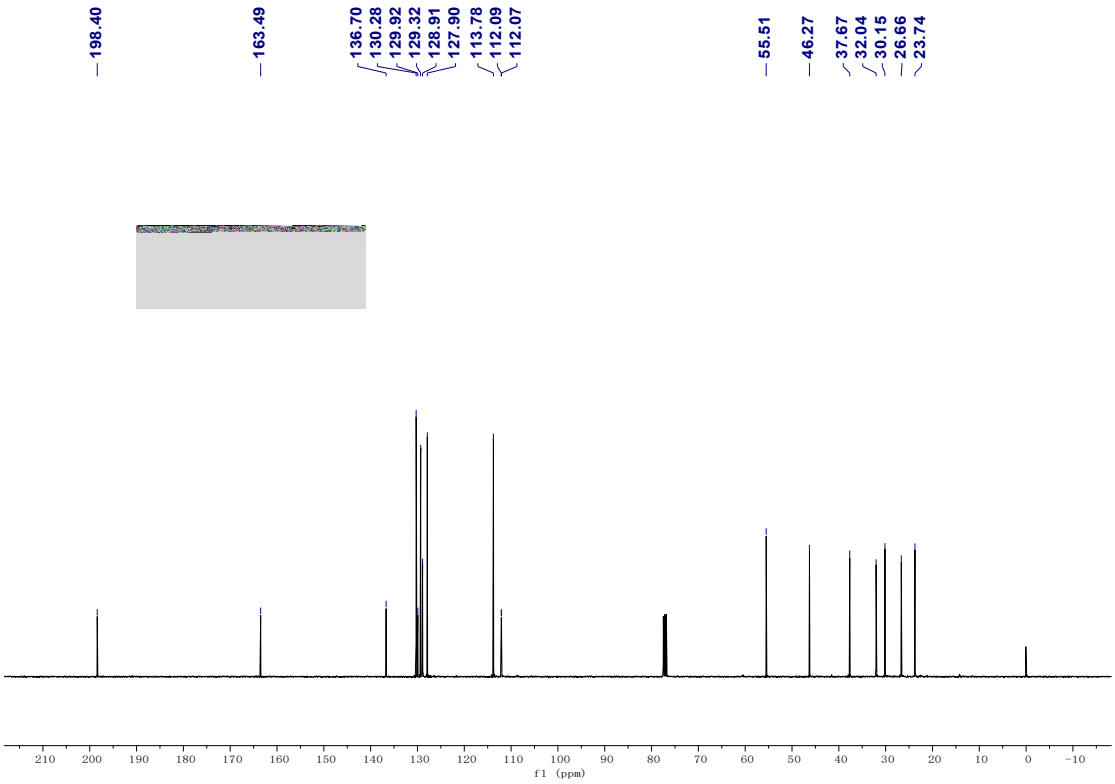
2.01
0.98
3.05
1.00
1.98
2.09
2.40
2.23
f1 (ppm)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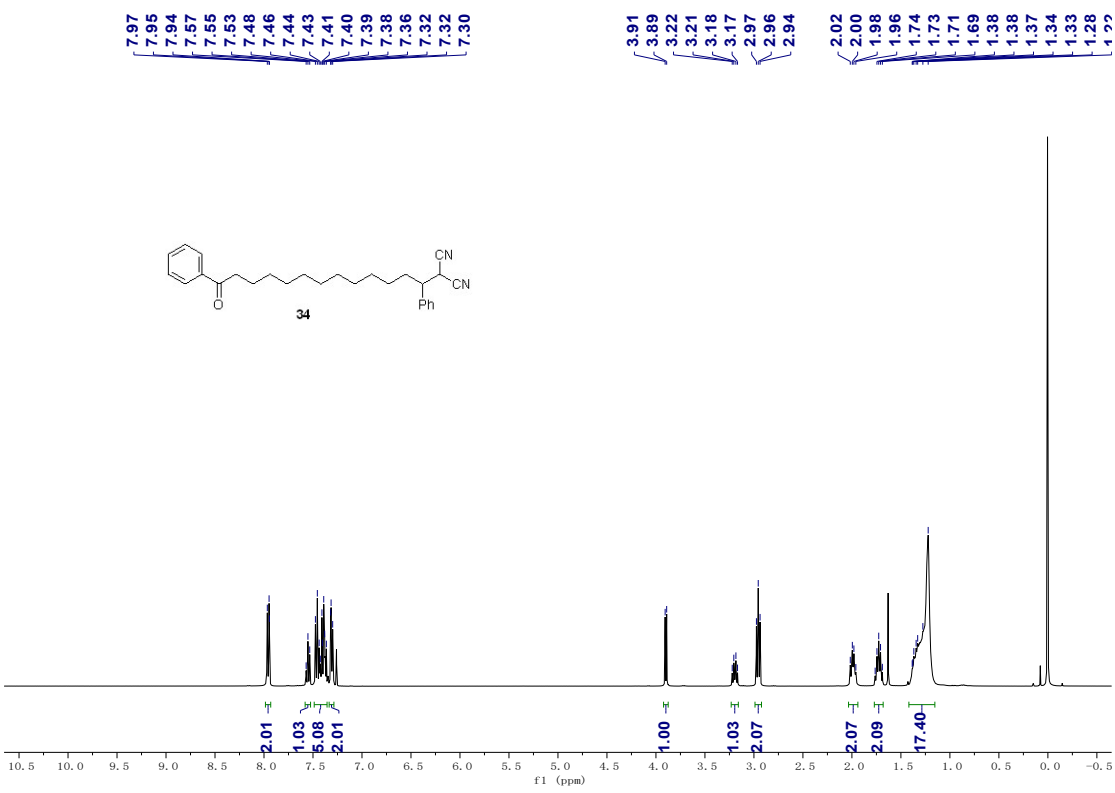
112.07
55.51
46.27
37.67
32.04
30.15
26.66
23.74
f1 (ppm)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7.53
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7.46
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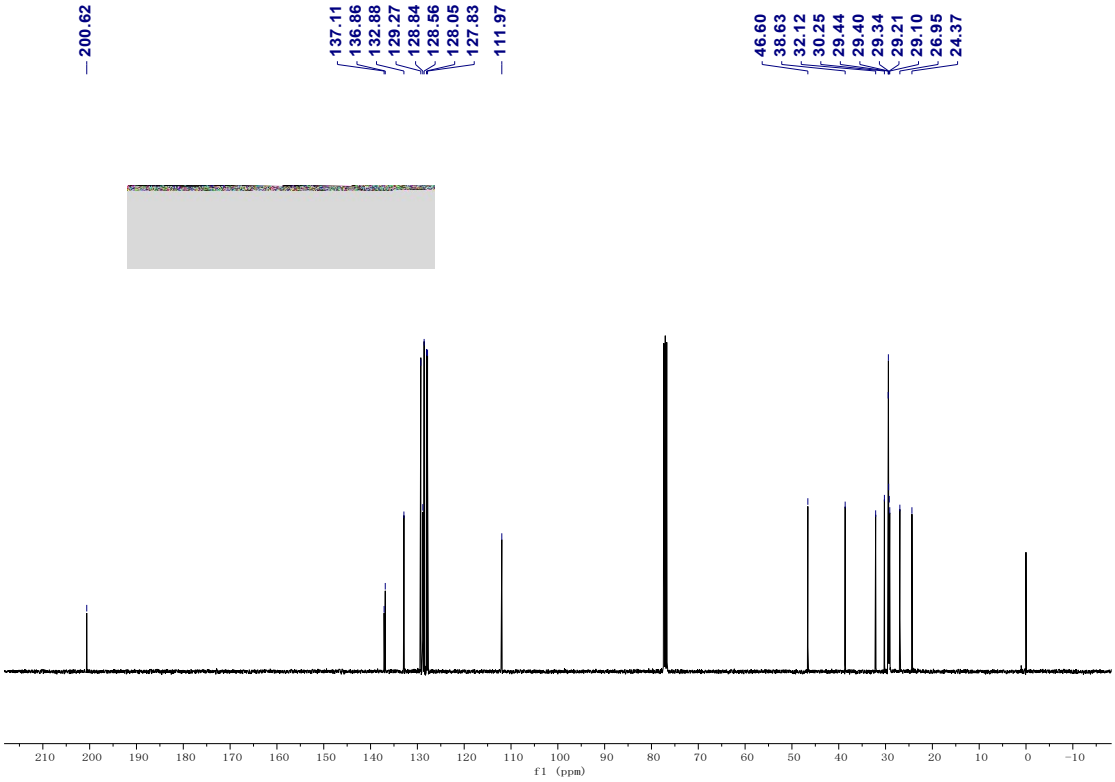
7.43
7.41
7.40
7.39
7.38
7.36
7.32
7.30
3.91
3.89
3.22
3.21
3.18
3.17
2.97
2.96
2.94
2.02
2.00
1.98
1.96
1.74
1.73
1.71
1.69
1.38
1.38
1.37
1.34
1.33
1.28
CN
CN
Ph
O
34
2.01
1.03
5.08
2.01
1.00
1.03
2.07
2.07
2.09
17.40
f1 (ppm)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137.11
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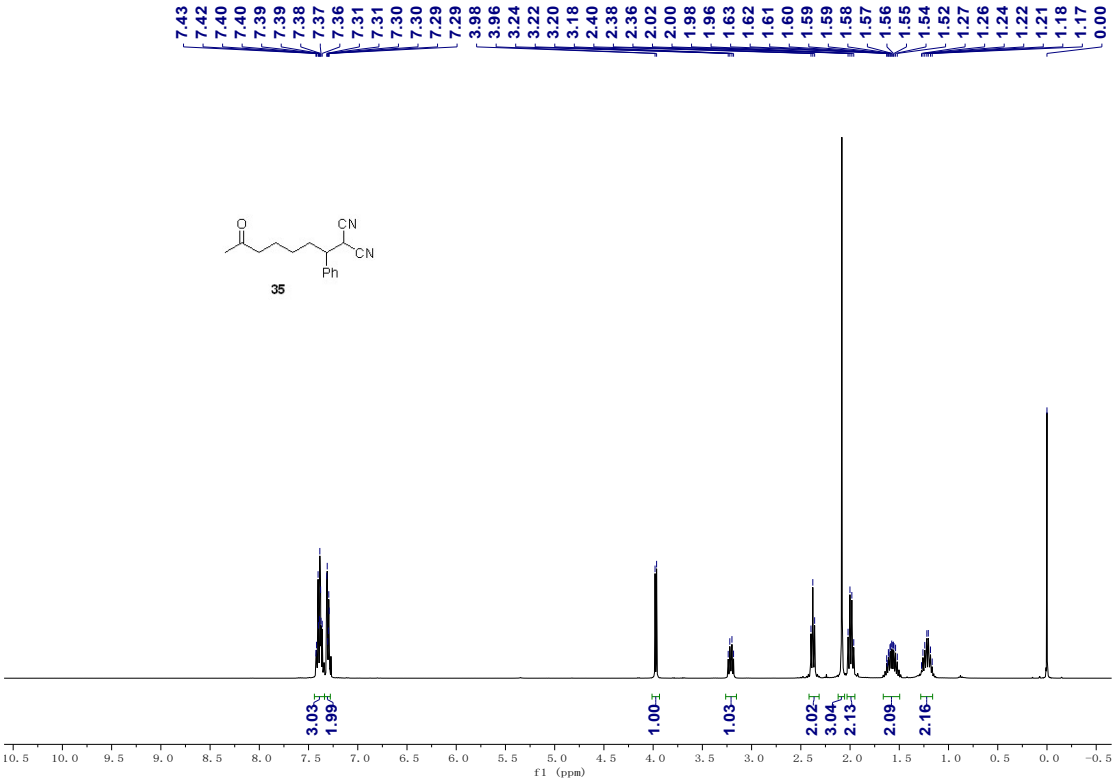
132.88
129.27
128.84
128.56
128.05
127.83
111.97
46.60
38.63
32.12
30.25
29.44
29.40
29.34
29.21
29.10
26.95
24.37
f1 (ppm)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7.38
7.37
7.36
7.31
7.31
7.30
7.30
7.29
7.29
3.98
3.96
3.24
3.22
3.20
3.18
2.40
2.38
2.36
2.02
2.00
1.98
1.96
1.63
1.62
1.61
1.60
1.59
1.59
1.58
1.57
1.56
1.55
1.54
1.52
1.27
1.26
1.24
1.22
1.21
1.18
1.17
0.00
O
CN
CN
Ph
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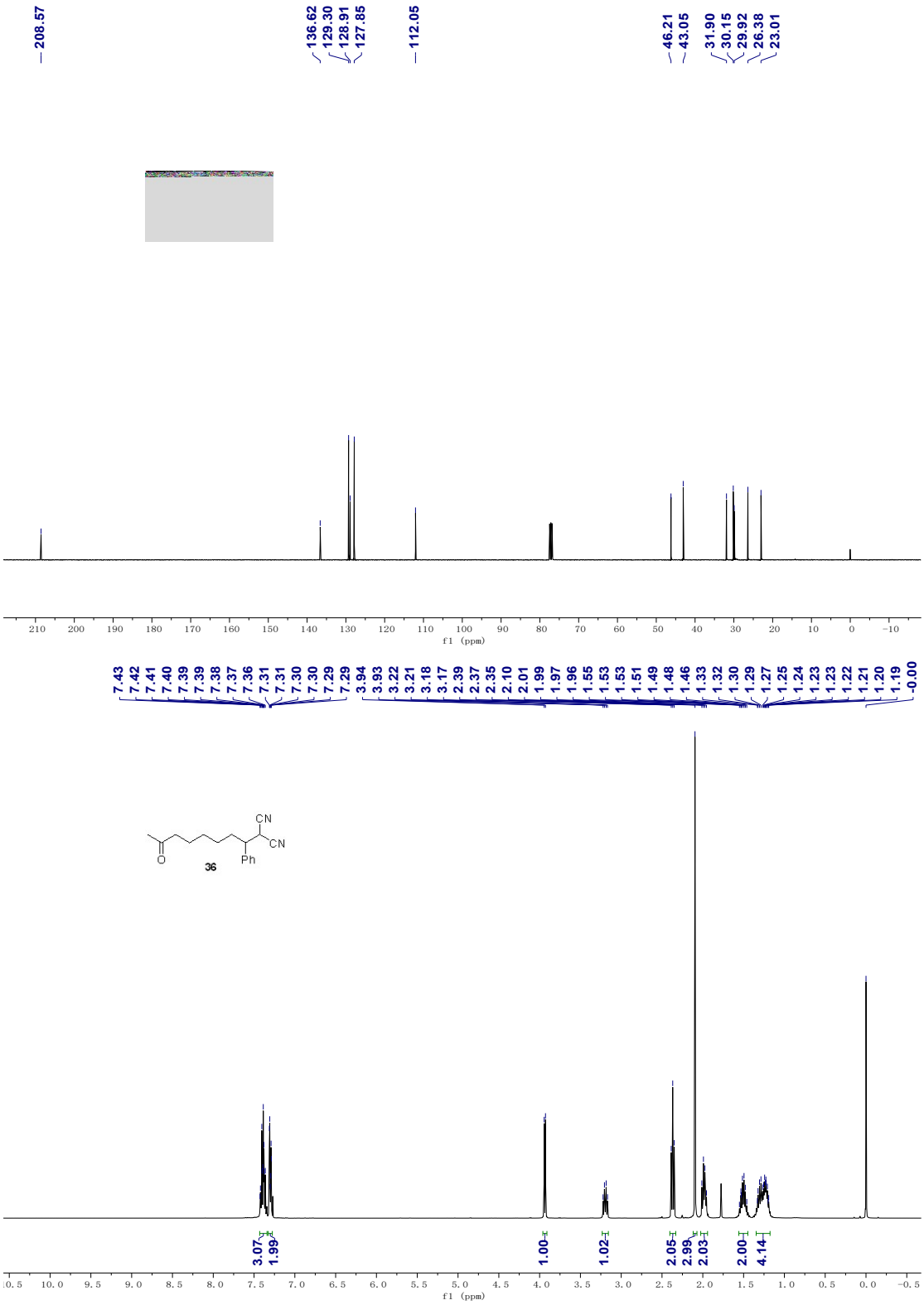
1.03
2.02
3.04
2.13
2.09
2.16
f1 (ppm)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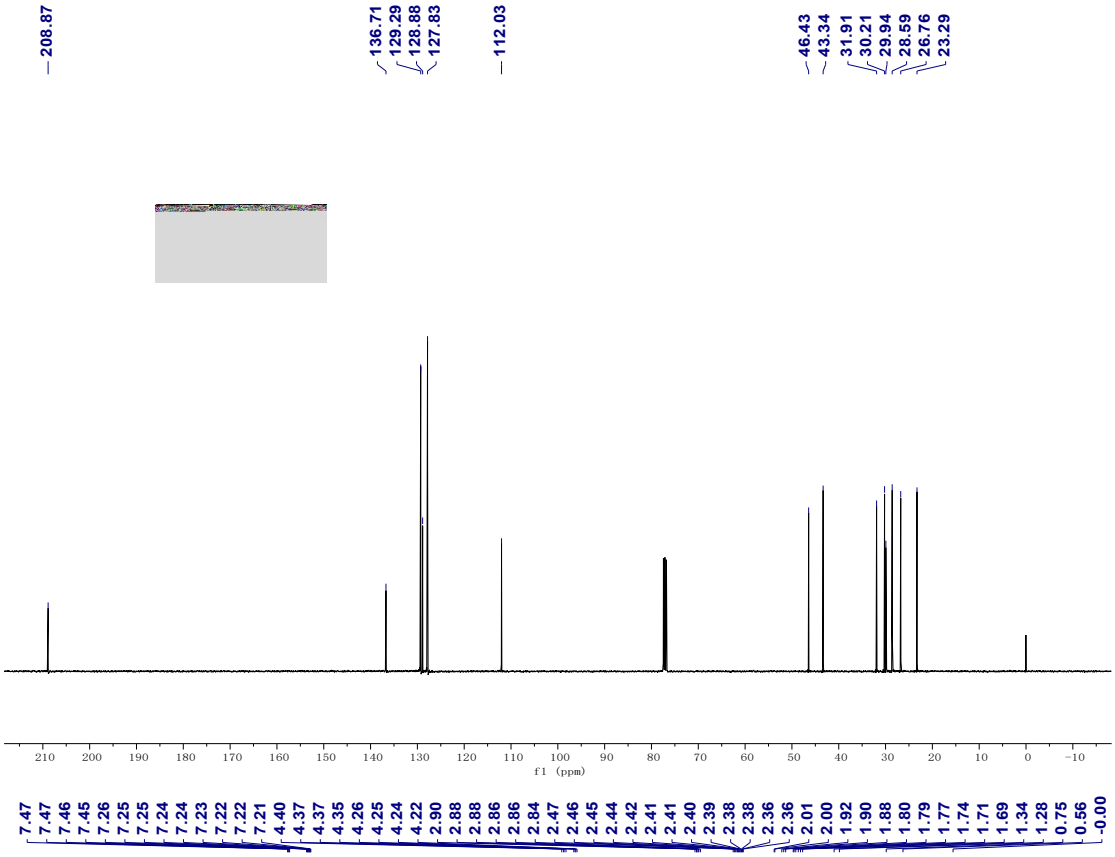
30.15
29.92
26.38
23.01
f1 (ppm)
7.43
7.42
7.41
7.40
7.39
7.39
7.38
7.37
7.36
7.31
7.31
7.30
7.30
7.29
7.29
3.94
3.93
3.22
3.21
3.18
3.17
2.39
2.37
2.35
2.10
2.01
1.99
1.97
1.96
1.55
1.53
1.53
1.51
1.49
1.48
1.46
1.33
1.32
1.30
1.29
1.27
1.25
1.24
1.23
1.23
1.22
1.21
1.20
1.19
-0.00
CN
CN
O
Ph
36
3.07
1.99
1.00
1.02
2.05
2.95
2.03
2.00
4.14
f1 (ppm)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127.83
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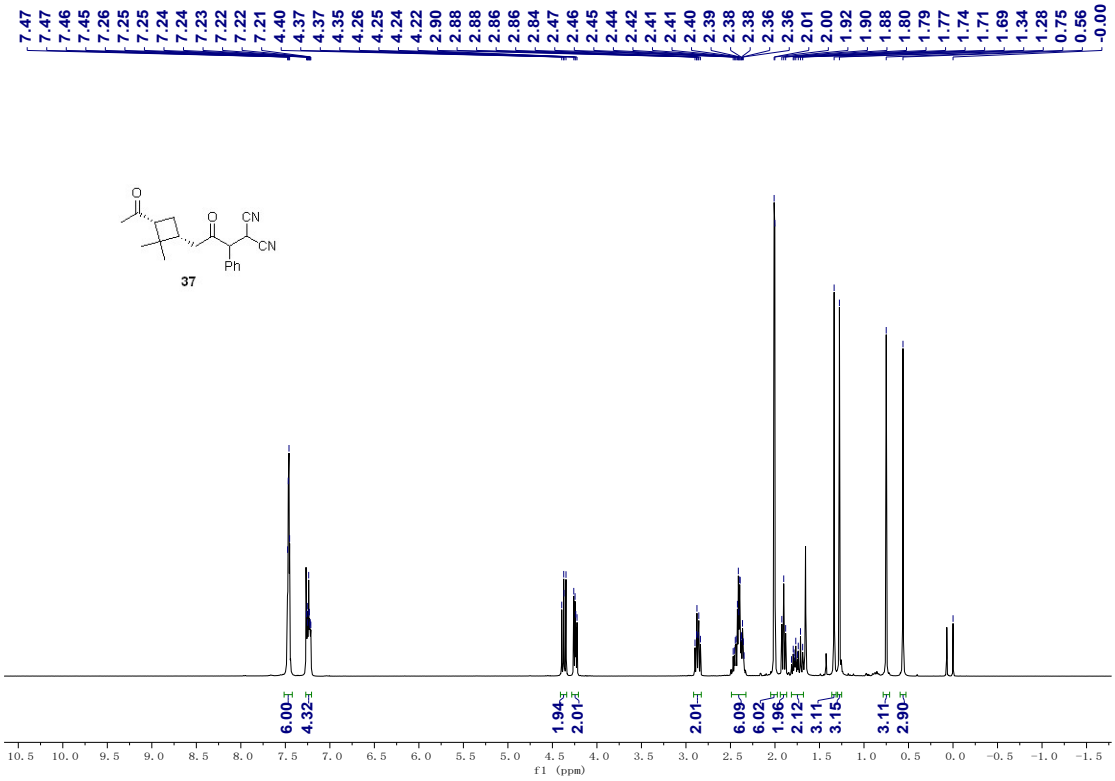
46.43
43.34
31.91
30.21
29.94
28.59
26.76
23.29
f1 (ppm)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7.25
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7.24
7.24
7.23
7.22
7.22
7.21
4.40
4.37
4.37
4.35
4.26
4.25
4.24
4.22
2.90
2.88
2.88
2.86
2.86
2.84
2.47
2.46
2.45
2.44
2.42
2.41
2.41
2.40
2.39
2.38
2.38
2.36
2.36
2.01
2.00
1.92
1.90
1.88
1.80
1.79
1.77
1.74
1.71
1.69
1.34
1.28
0.75
0.56
-0.00

O
O
CN
CN
Ph
37
6.00
4.32
1.94
2.01
2.01
6.09
6.02
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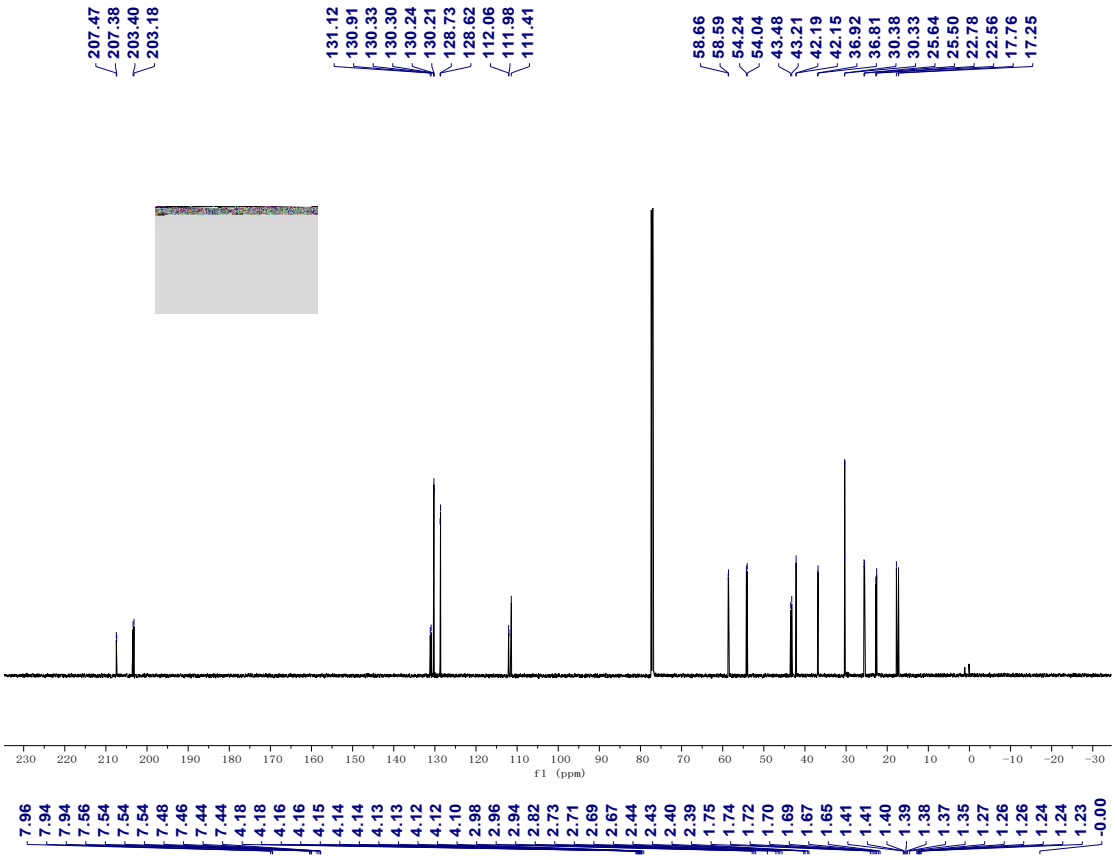
2.12
3.11
3.15
3.11
2.90
f1 (ppm)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130.91
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130.30
130.24
130.21
128.73
128.62
112.06
111.98
111.41
58.66
58.59
54.24
54.04
43.48
43.21
42.19
42.15
36.92
36.81
30.38
30.33
25.64
25.50
22.78
22.56
17.76
17.25
f1 (ppm)

7.96
7.94
7.94
7.56
7.54
7.54
7.54
7.48
7.46
7.44
7.44
4.18
4.18
4.16
4.16
4.15
4.14
4.14
4.13
4.13
4.12
4.12
4.10
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2.82
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1.26
1.26
1.24
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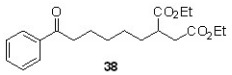
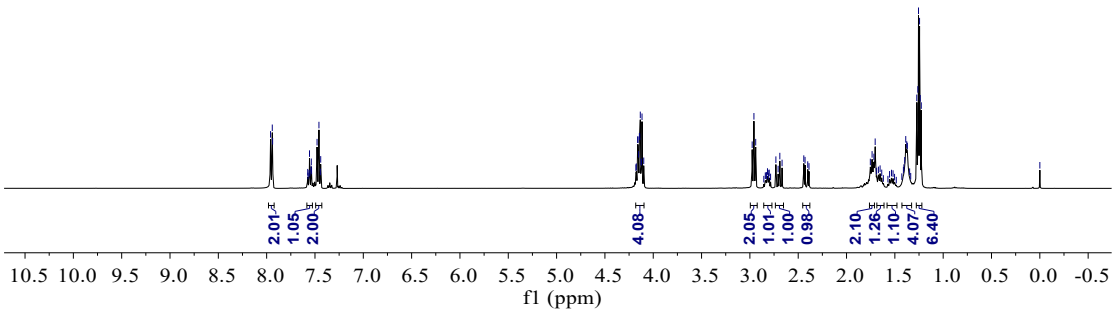
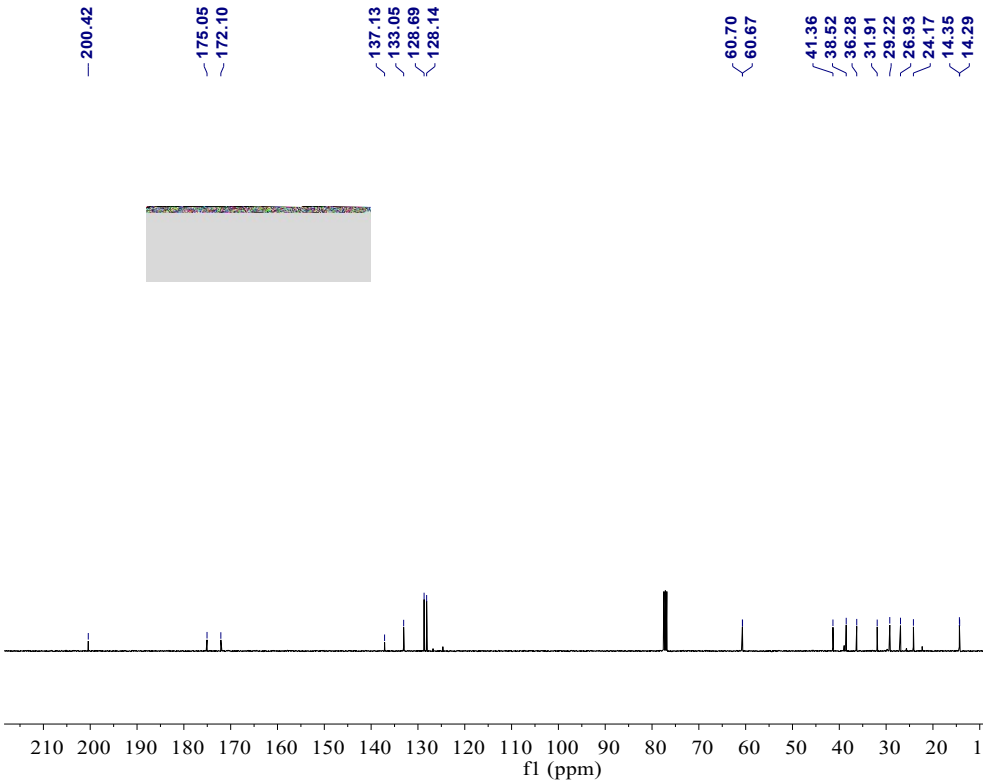
1.23
-0.00


O
CO2Et
CO2Et

38


2.01
1.05
2.00
4.08
2.05
1.01
1.00
0.98
2.10
1.26
1.10
4.07
6.40
f1 (ppm)


200.42
175.05
172.10
137.13
133.05
128.69
128.14
60.70
60.67
41.36
38.52
36.28
31.91
29.22
26.93
24.17
14.35
14.29
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.96
7.96
7.94
7.94
7.93
7.57
7.55
7.54
7.54
7.53
7.48
7.47
7.46
7.44
4.24
4.22
4.21
4.19
4.19
4.17
4.17
4.16
4.15
4.14
3.75
3.72
3.13
3.11
3.11
2.97
2.95
2.93
1.74
1.72
1.70
1.60
1.60
1.58
1.42
1.41
1.40
1.39
1.38
1.37
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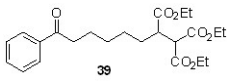
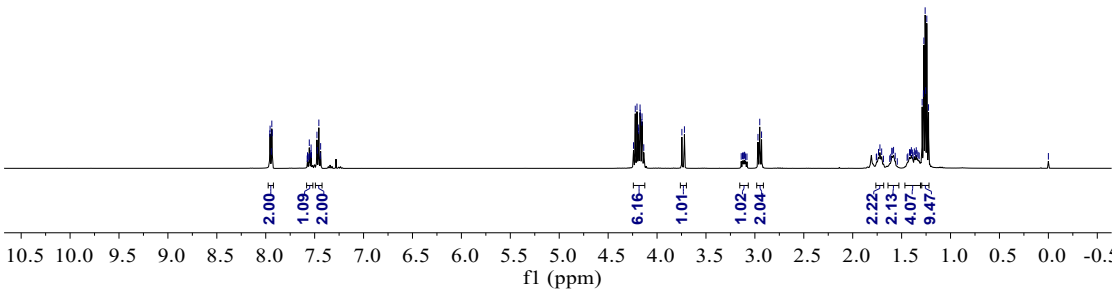
1.28
1.27
1.26
1.25
1.24
1.22


O
CO2Et
CO2Et
CO2Et
39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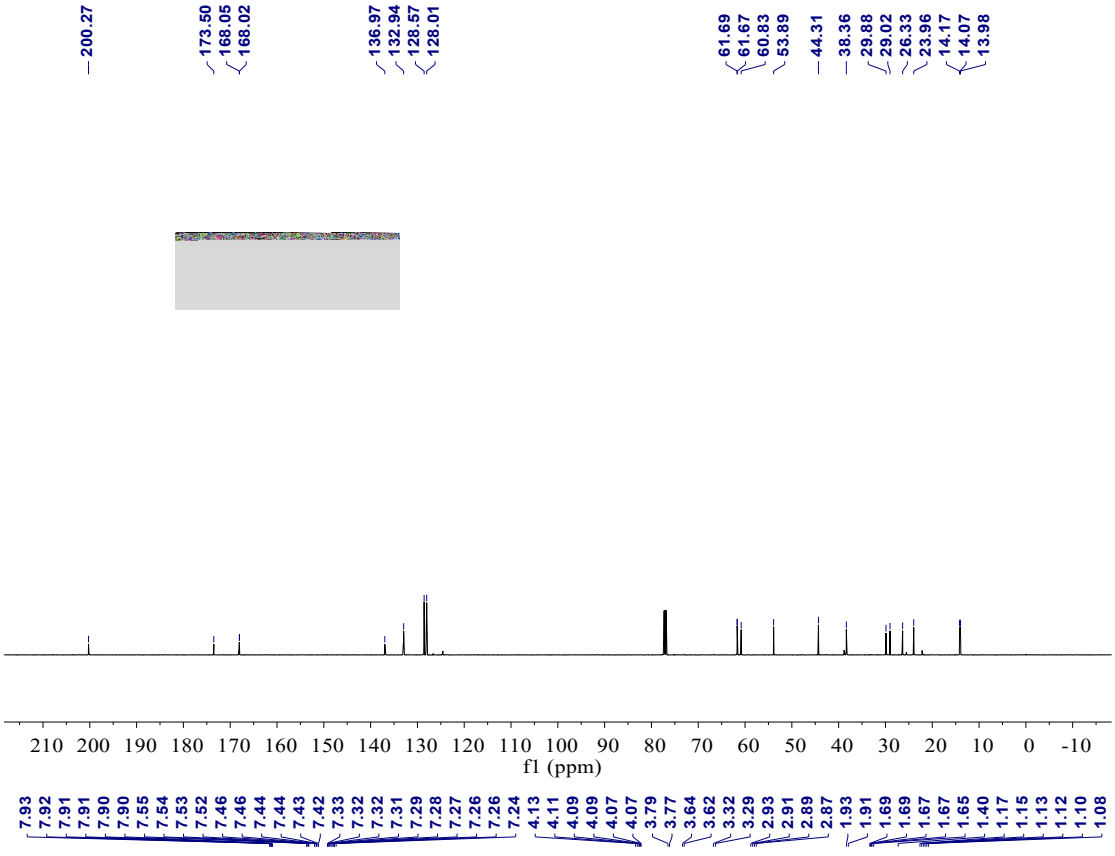
1.09
2.00
6.16
1.01
1.02
2.04
2.22
2.13
4.07
9.47
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.57
128.01
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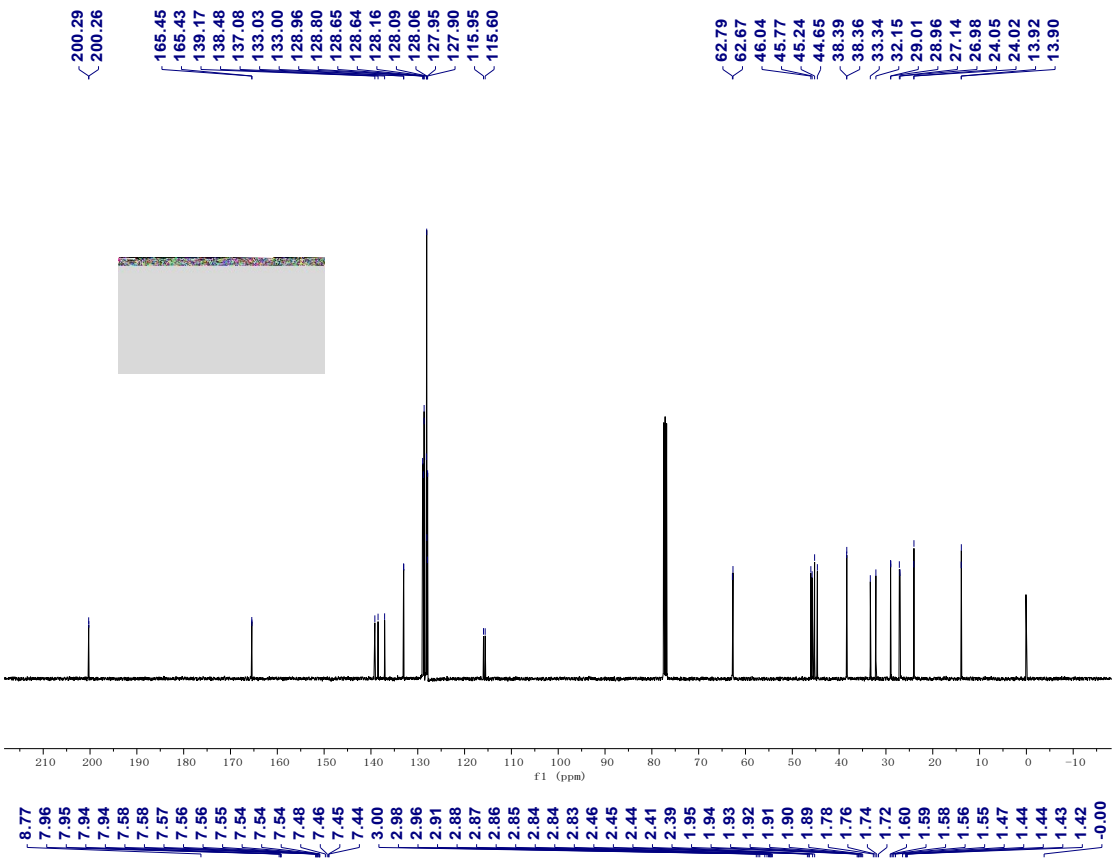
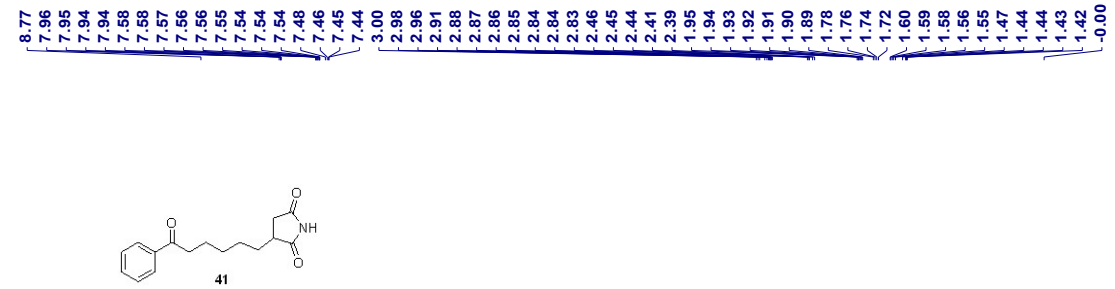
61.67
60.83
53.89
44.31
38.36
29.88
29.02
26.33
23.96
14.17
14.07
13.98
f1 (ppm)
7.93
7.92
7.91
7.91
7.90
7.90
7.55
7.54
7.53
7.52
7.46
7.46
7.44
7.44
7.43
7.42
7.33
7.32
7.32
7.31
7.29
7.28
7.27
7.26
7.26
7.24
4.13
4.11
4.09
4.09
4.07
4.07
3.79
3.77
3.64
3.62
3.32
3.29
2.93
2.91
2.89
2.87
1.93
1.91
1.69
1.69
1.67
1.67
1.65
1.40
1.17
1.15
1.13
1.12
1.10
1.08
O
CN
CO2Et
40
4.02
2.02
4.08
10.46
4.06
1.00
1.01
2.03
4.06
4.17
4.19
8.32
6.22
f1 (ppm)


200.29
200.26
165.45
165.43
139.17
138.48
137.08
133.03
133.00
128.96
128.80
128.65
128.64
128.16
128.09
128.06
127.95
127.90
115.95
115.60
62.79
62.67
46.04
45.77
45.24
44.65
38.39
38.36
33.34
32.15
29.01
28.96
27.14
26.98
24.05
24.02
13.92
13.90
f1 (ppm)


8.77
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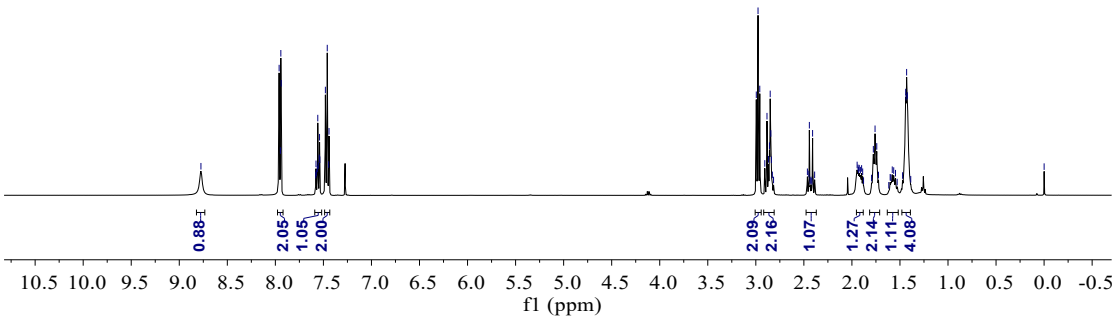
7.95
7.94
7.94
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.54
7.54
7.48
7.46
7.45
7.44
3.00
2.98
2.96
2.91
2.88
2.87
2.86
2.85
2.84
2.84
2.83
2.46
2.45
2.44
2.41
2.39
1.95
1.94
1.93
1.92
1.91
1.90
1.89
1.78
1.76
1.74
1.72
1.60
1.59
1.58
1.56
1.55
1.47
1.44
1.44
1.43
1.42
-0.00

O
NH
O
O

41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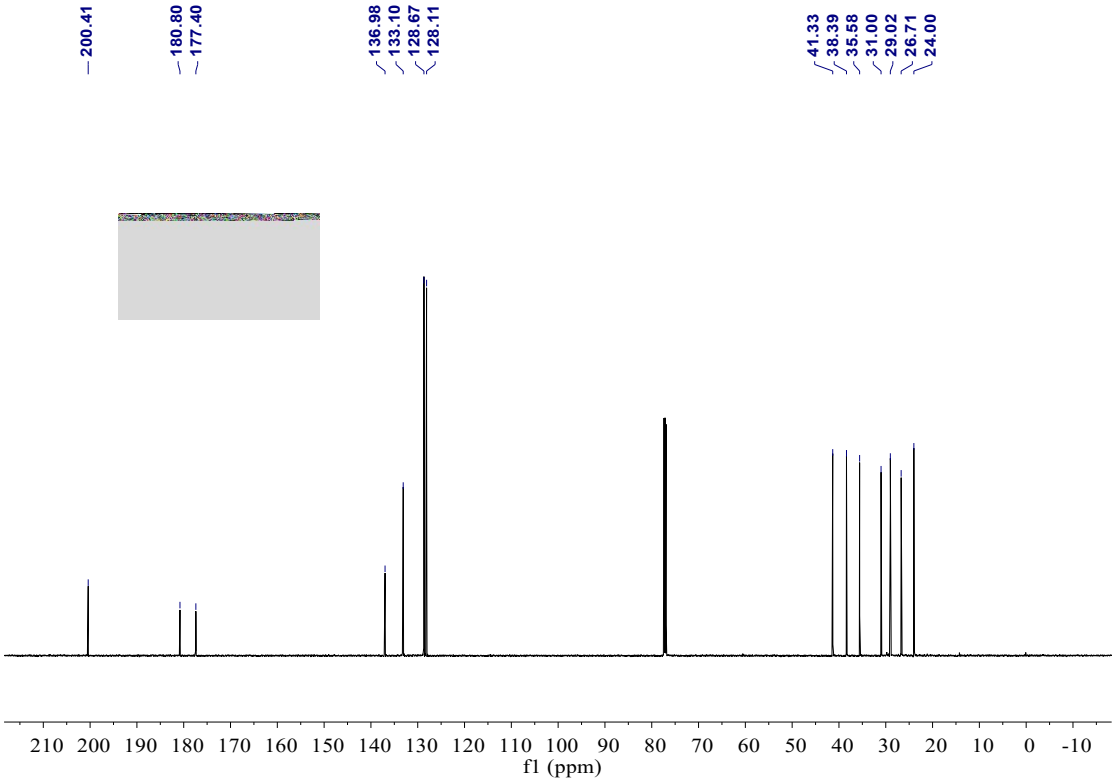
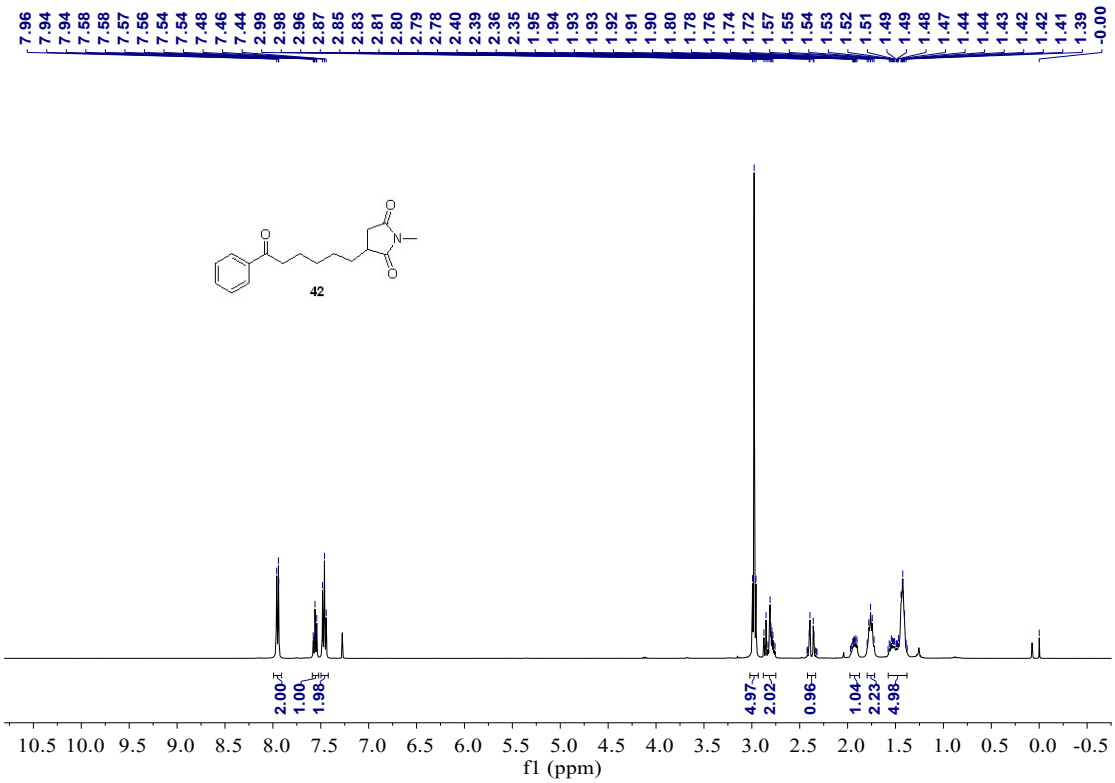
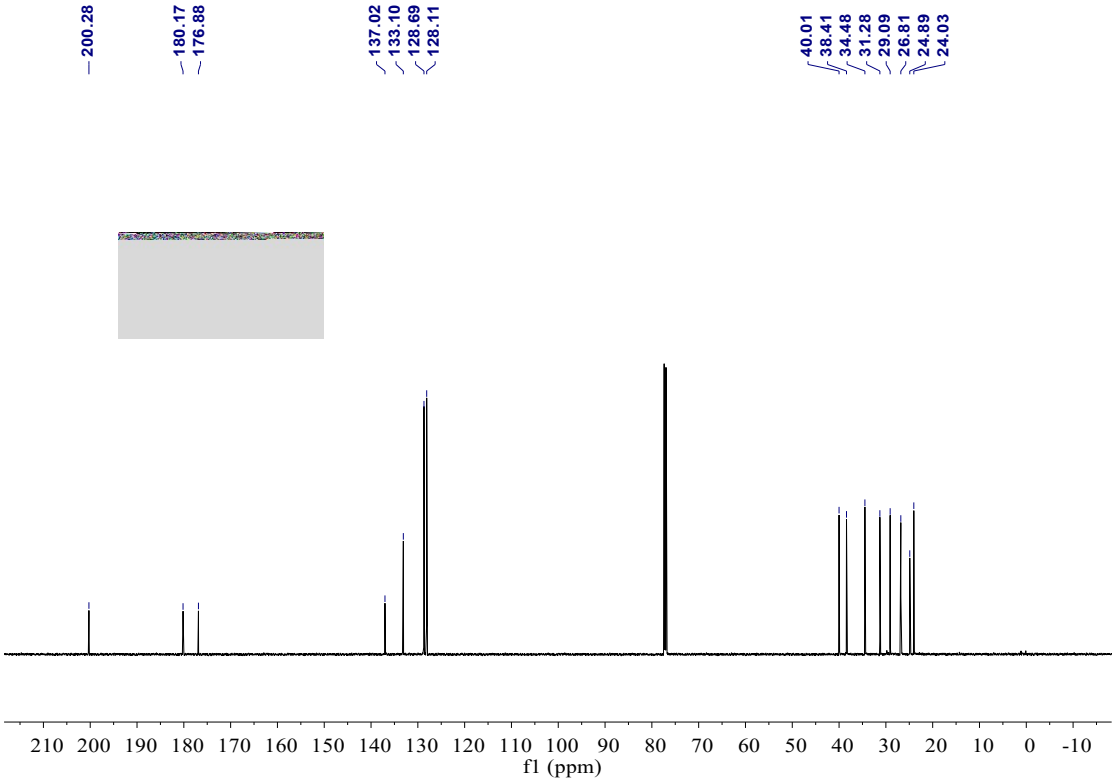
2.09
2.16
1.07
1.27
2.14
1.11
4.08
f1 (ppm)


200.41
180.80
177.40
136.98
133.10
128.67
128.11
41.33
38.39
35.58
31.00
29.02
26.71
24.00
f1 (ppm)


7.96
7.94
7.94
7.58
7.58
7.57
7.56
7.54
7.54
7.48
7.46
7.44
2.99
2.98
2.96
2.87
2.85
2.83
2.81
2.80
2.79
2.78
2.40
2.39
2.36
2.35
1.95
1.94
1.93
1.93
1.92
1.91
1.90
1.80
1.78
1.76
1.74
1.72
1.57
1.55
1.54
1.53
1.52
1.51
1.49
1.49
1.48
1.47
1.44
1.44
1.43
1.42
1.42
1.41
1.39
-0.00
O
O
N
O
42
2.00
1.00
1.98
4.97
2.02
0.96
1.04
2.23
4.98
f1 (ppm)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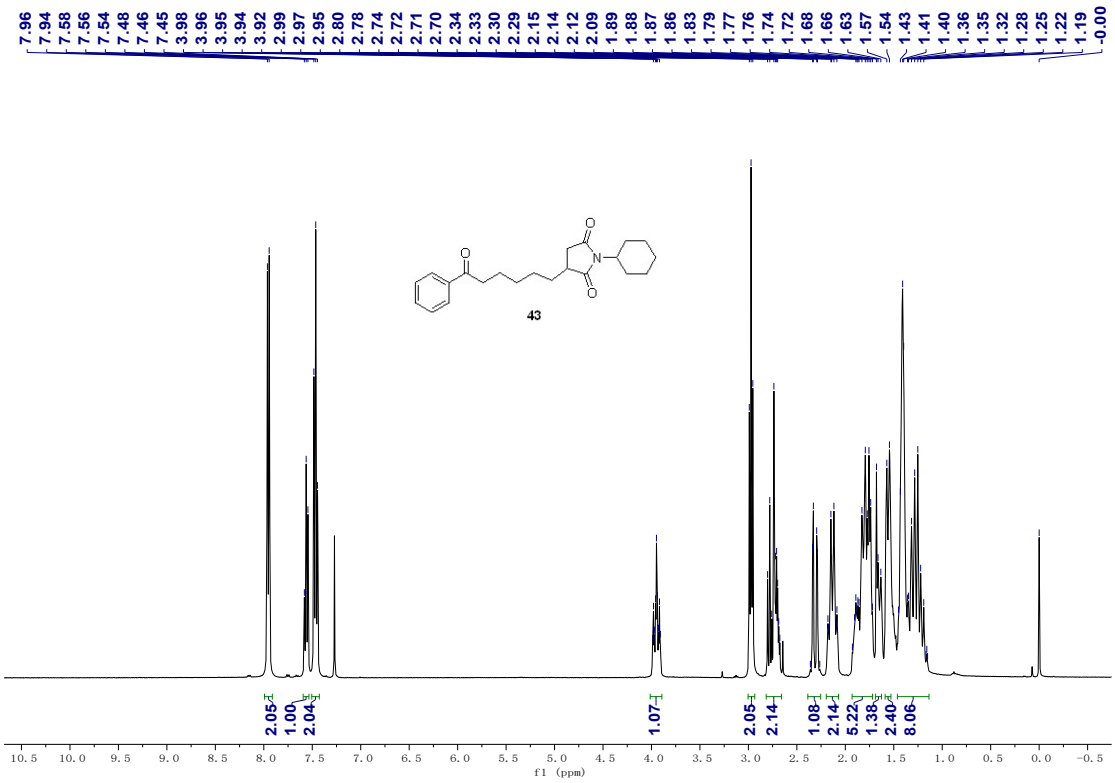
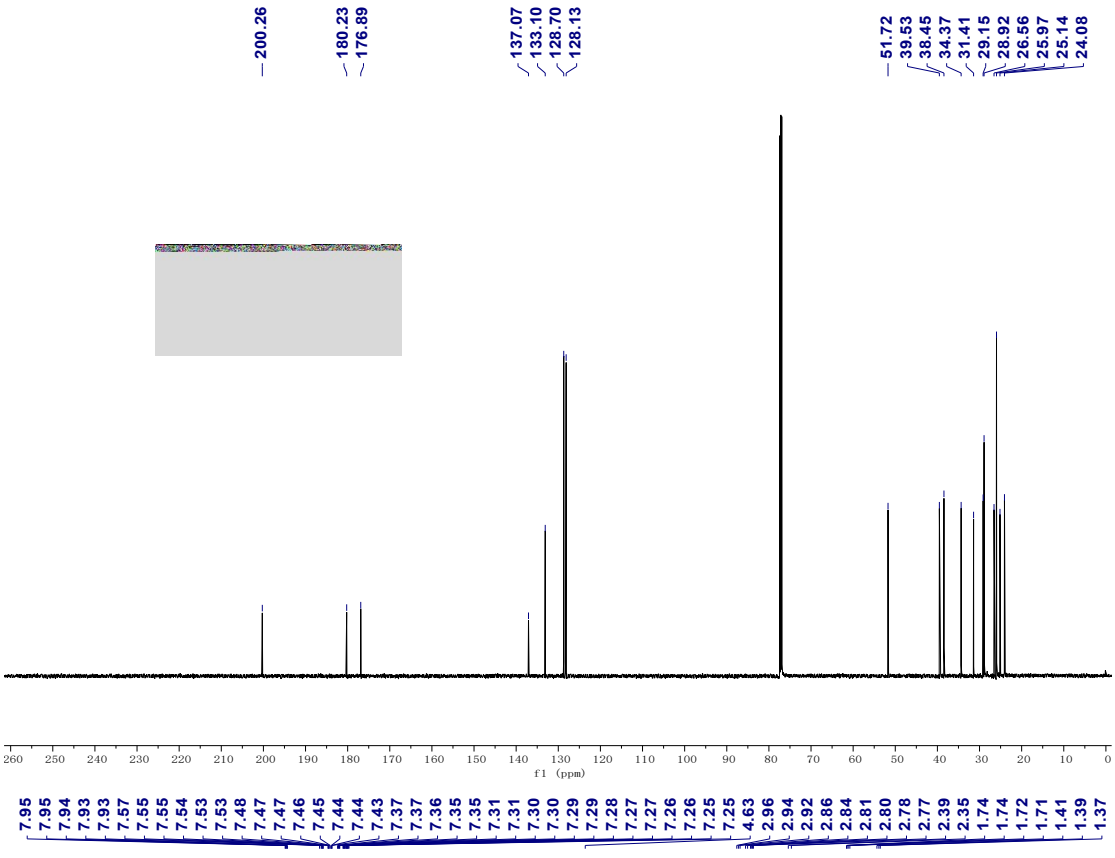
180.17
176.88
137.02
133.10
128.69
128.11
40.01
38.41
34.48
31.28
29.09
26.81
24.89
24.03
f1 (ppm)


7.96
7.94
7.58
7.56
7.54
7.48
7.46
7.45
3.98
3.96
3.95
3.94
3.92
2.99
2.97
2.95
2.80
2.78
2.74
2.72
2.71
2.70
2.34
2.33
2.30
2.29
2.15
2.14
2.12
2.09
1.89
1.88
1.87
1.86
1.83
1.79
1.77
1.76
1.74
1.72
1.68
1.66
1.63
1.57
1.54
1.43
1.41
1.40
1.36
1.35
1.32
1.28
1.25
1.22
1.19
-0.00
43
2.05
1.00
2.04
1.07
2.05
2.14
1.08
2.14
5.22
1.38
2.40
8.06
f1 (ppm)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137.07
133.10
128.70
128.13
51.72
39.53
38.45
34.37
31.41
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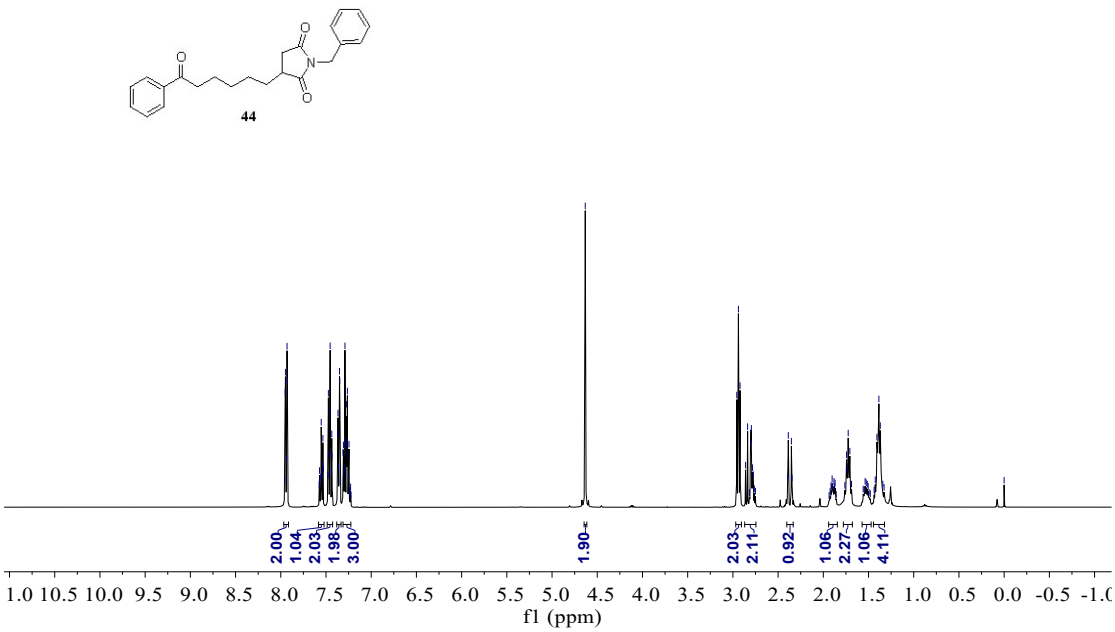
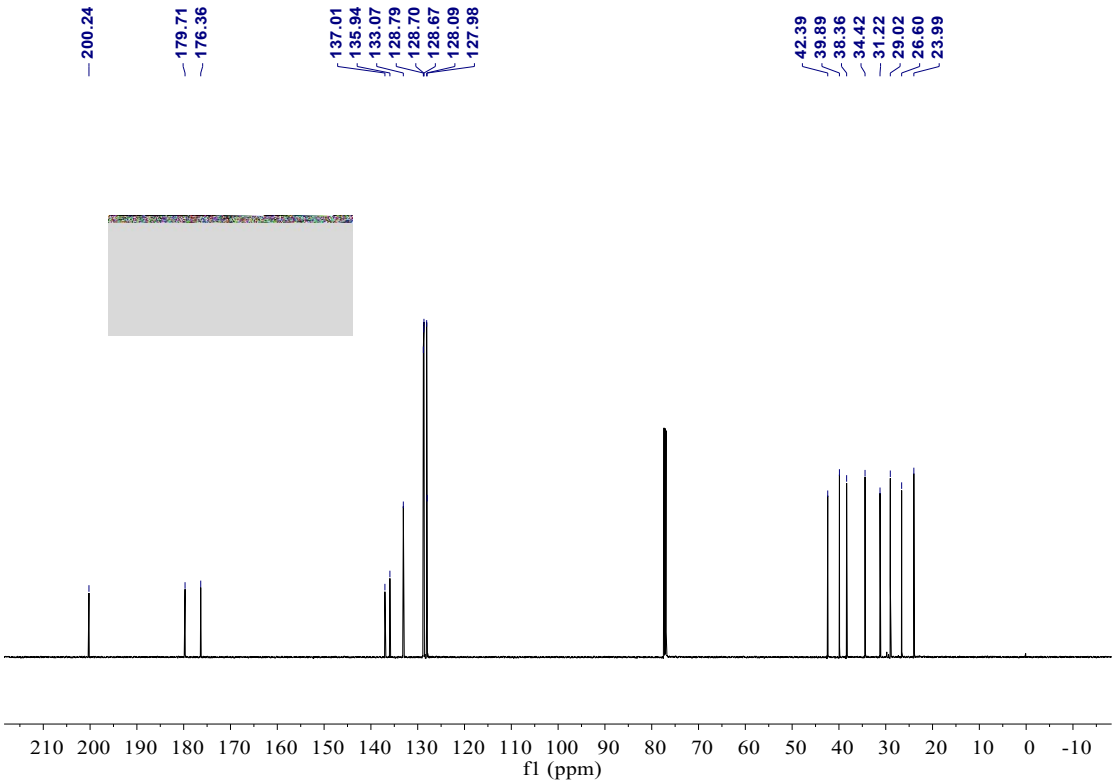
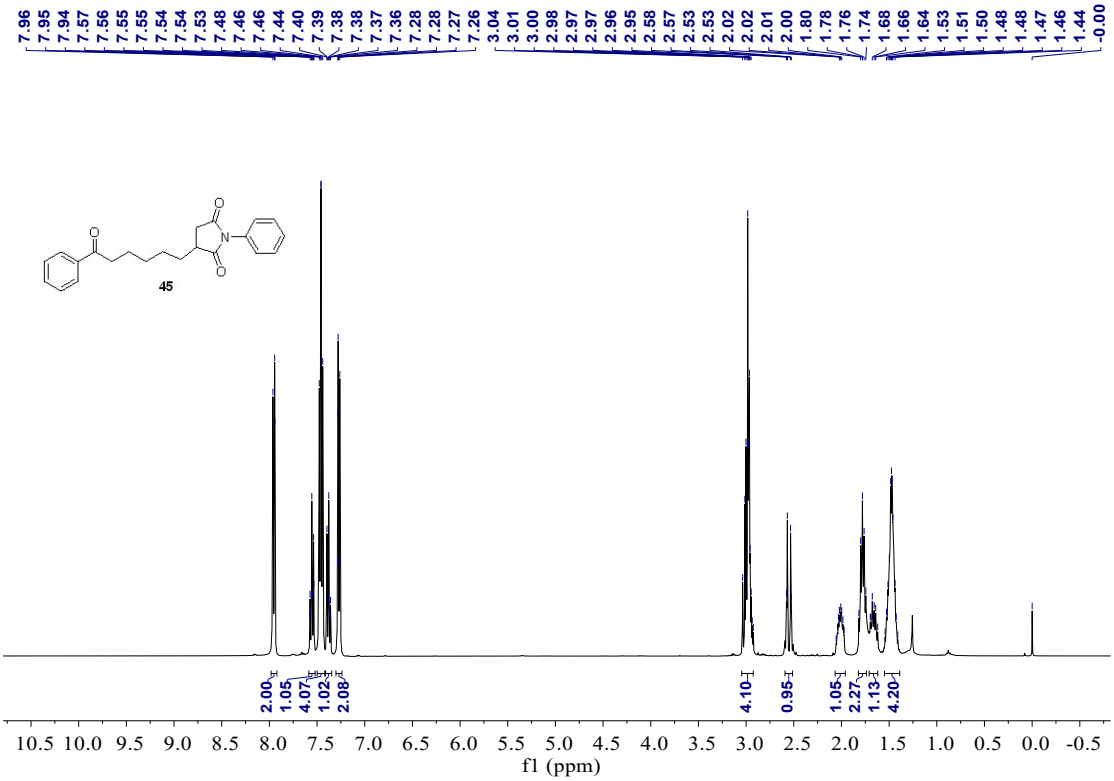
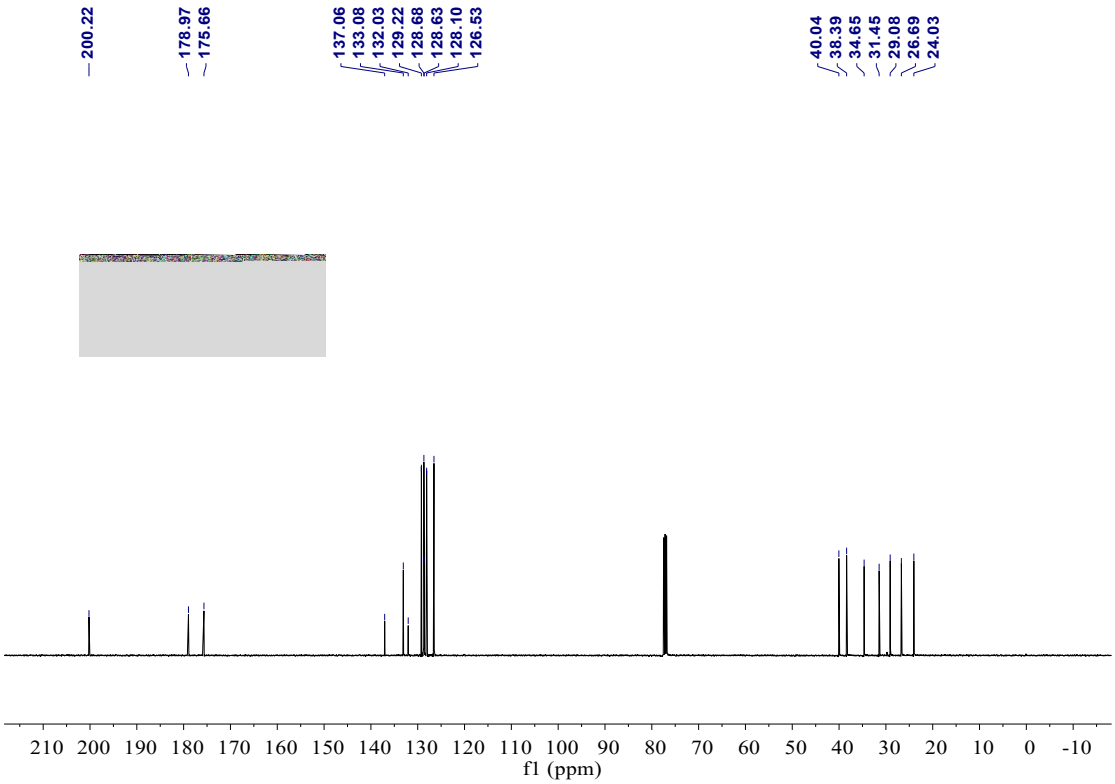
28.92
26.56
25.97
25.14
24.08
f1 (ppm)


44
2.00
1.04
2.03
1.98
3.00
1.90
2.03
2.11
0.92
1.06
2.27
1.06
4.11
f1 (ppm)


200.24
179.71
176.36
137.01
135.94
133.07
128.79
128.70
128.67
128.09
127.98
42.39
39.89
38.36
34.42
31.22
29.02
26.60
23.99
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.96
7.95
7.94
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.48
7.46
7.46
7.44
7.40
7.39
7.38
7.38
7.37
7.36
7.28
7.28
7.27
7.26
3.04
3.01
3.00
2.98
2.97
2.97
2.96
2.95
2.58
2.57
2.53
2.53
2.02
2.02
2.01
2.00
1.80
1.78
1.76
1.74
1.68
1.66
1.64
1.53
1.51
1.50
1.48
1.48
1.47
1.46
1.44
-0.00
O
N
O
O
45
2.00
1.05
4.07
1.02
2.08
4.10
0.95
1.05
2.27
1.13
4.20
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 -0.5
f1 (ppm)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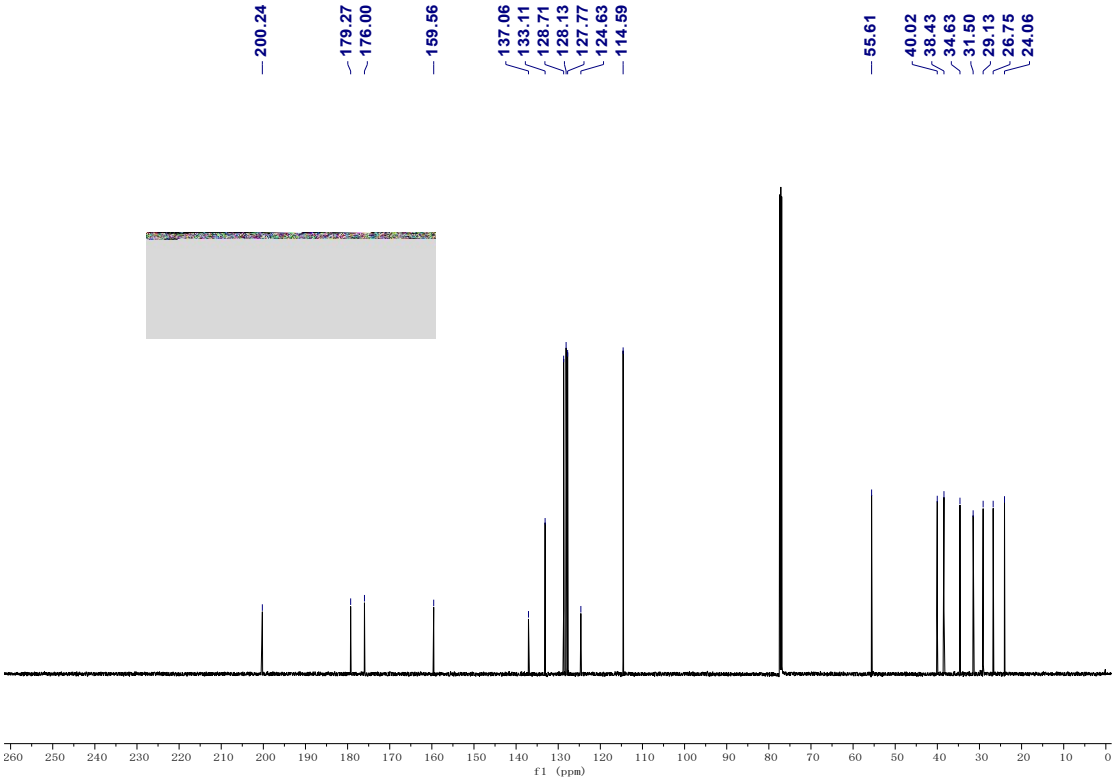
175.66
137.06
133.08
132.03
129.22
128.68
128.63
128.10
126.53
40.04
38.39
34.65
31.45
29.08
26.69
24.03
f1 (ppm)
7.97
7.95
7.95
7.58
7.56
7.55
7.48
7.47
7.45
7.19
7.17
6.98
6.96
3.82
3.03
3.01
2.99
2.97
2.95
2.94
2.93
2.92
2.59
2.57
2.56
2.53
2.52
2.50
2.06
2.04
2.03
2.02
2.02
2.01
2.00
1.99
1.82
1.80
1.78
1.76
1.75
1.69
1.69
1.67
1.66
1.64
1.62
1.53
1.51
1.48
1.48
1.47
1.41
-0.00
OMe
46
2.00
1.00
2.03
2.00
2.04
3.01
4.04
1.03
1.08
2.01
1.30
4.08
f1 (ppm)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176.00
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137.06
133.11
128.71
128.13
127.77
124.63
114.59
55.61
40.02
38.43
34.63
31.50
29.13
26.75
24.06
f1 (ppm)


7.97
7.95
7.58
7.56
7.54
7.48
7.46
7.44
2.98
2.97
2.95
2.60
2.59
2.01
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1.97
1.96
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1.80
1.78
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1.75
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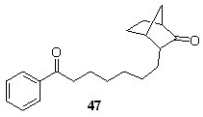
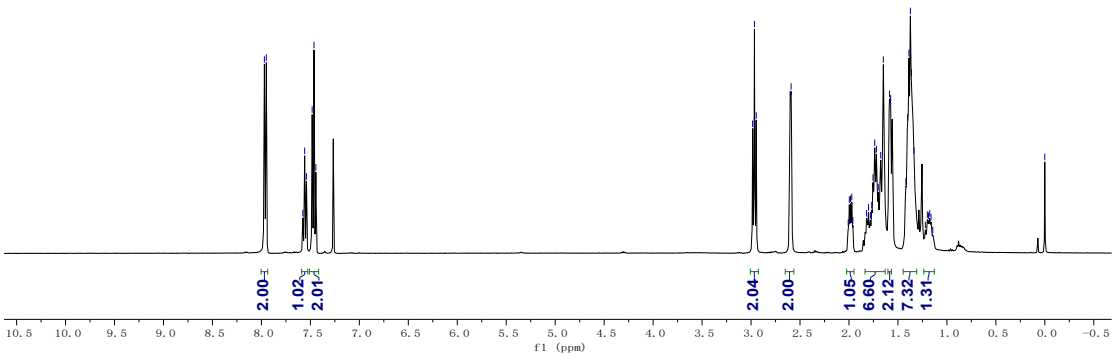
1.70
1.68
1.65
1.59
1.57
1.42
1.40
1.39
1.37
1.36
1.34
1.22
1.20
1.19
1.18
1.16
1.14
0.00


O
O
47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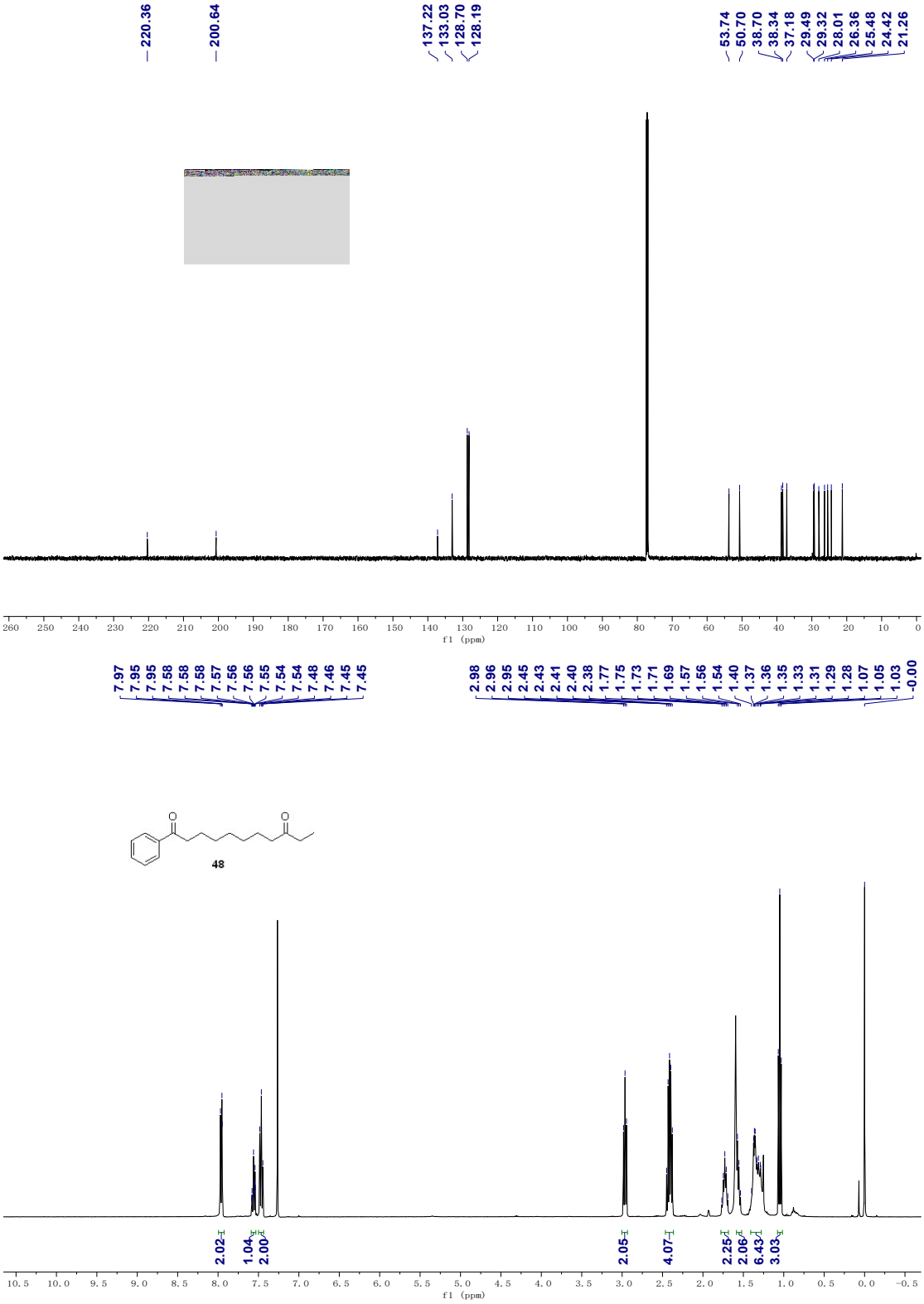
1.02
2.01
2.04
2.00
1.05
6.60
2.12
7.32
1.31
f1 (ppm)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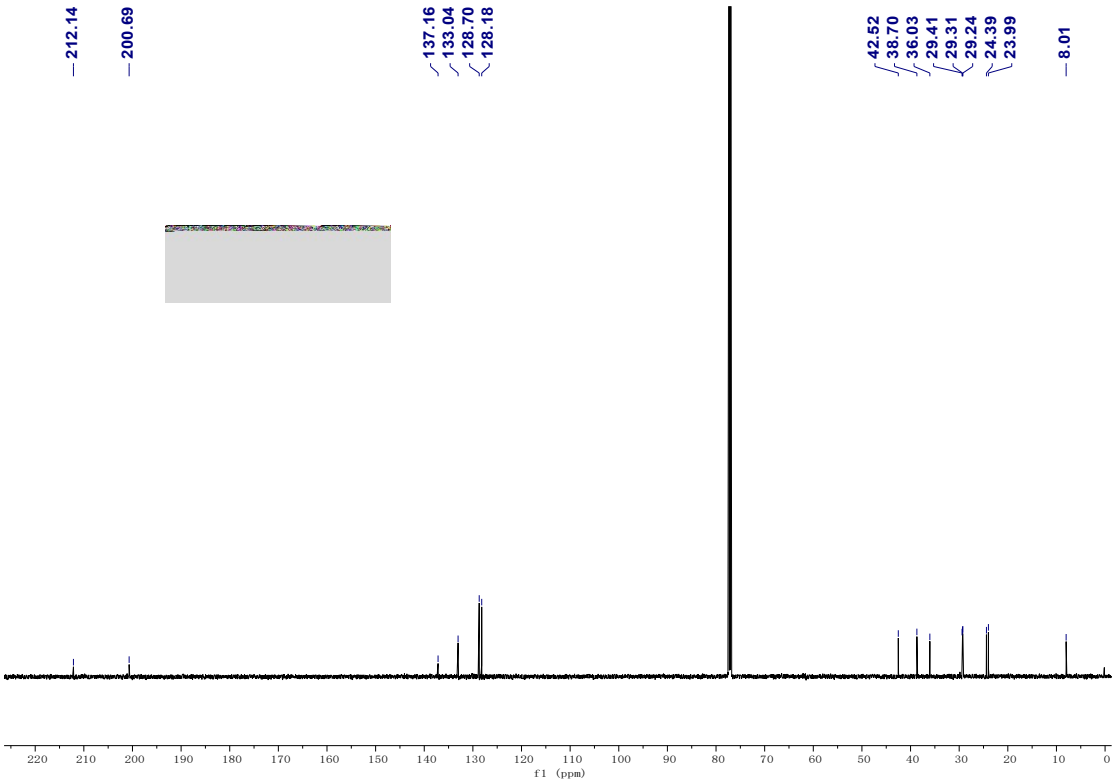
128.19
53.74
50.70
38.70
38.34
37.18
29.49
29.32
28.01
26.36
25.48
24.42
21.26
f1 (ppm)
7.97
7.95
7.95
7.58
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.54
7.48
7.46
7.45
7.45
2.98
2.96
2.95
2.45
2.43
2.41
2.40
2.38
1.77
1.75
1.73
1.71
1.69
1.57
1.56
1.54
1.40
1.37
1.36
1.35
1.33
1.31
1.29
1.28
1.07
1.05
1.03
-0.00
O
O
48
2.02
1.04
2.00
2.05
4.07
2.25
2.06
6.43
3.03
f1 (ppm)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128.18
42.52
38.70
36.03
29.41
29.31
29.24
24.39
23.99
8.01
f1 (ppm)


7.97
7.96
7.95
7.95
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.54
7.54
7.48
7.46
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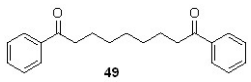
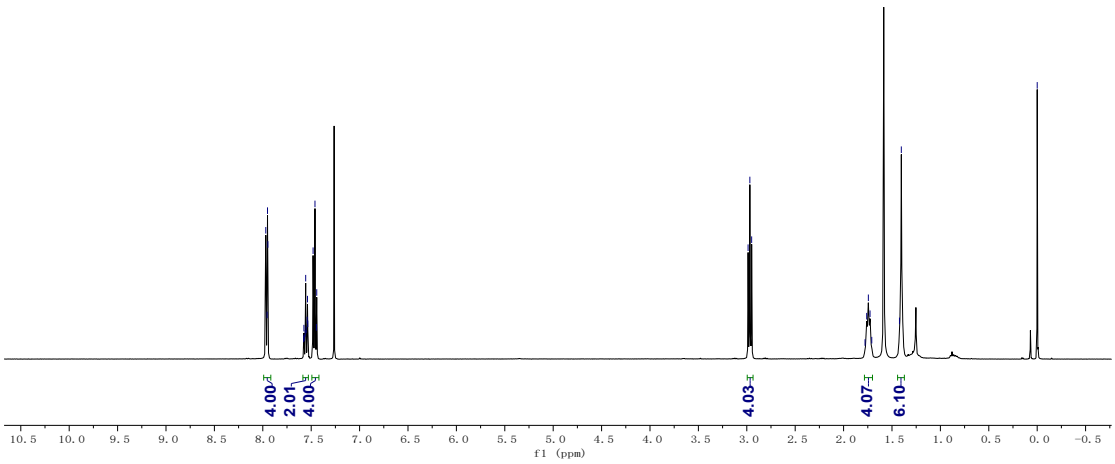
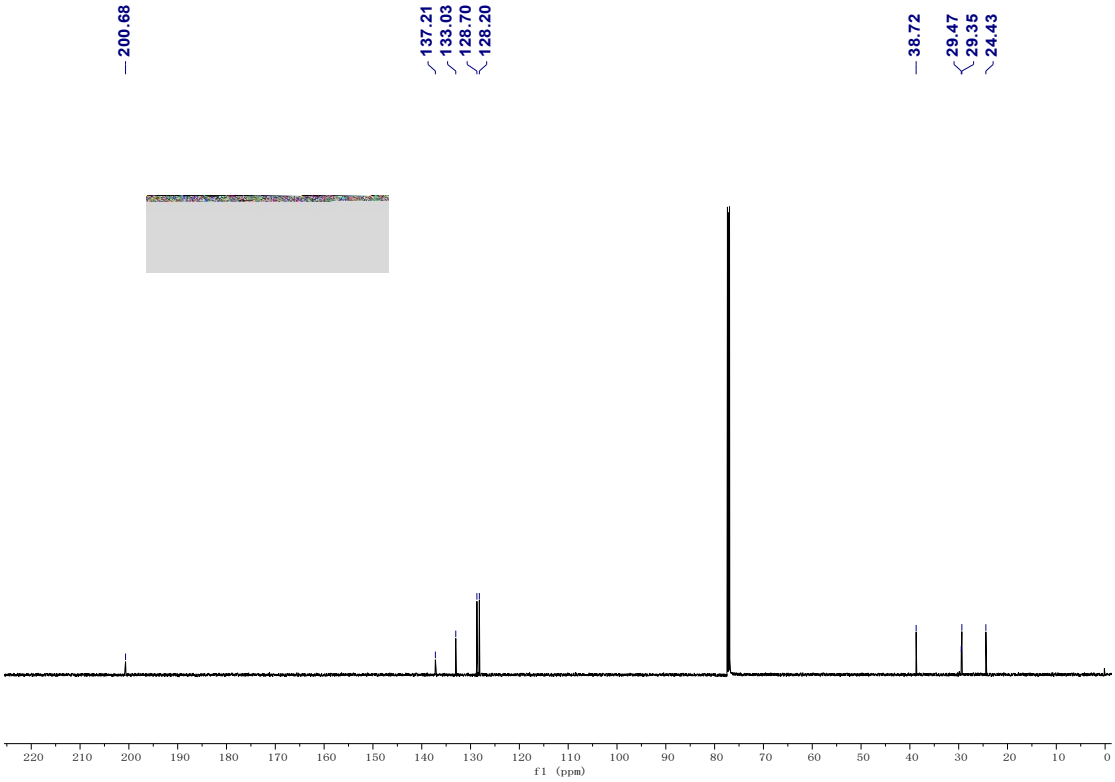
1.73
1.71
1.42
1.40
-0.00

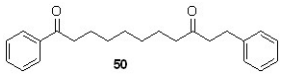

O
O

49


4.00
2.01
4.00
4.03
4.07
6.10
f1 (ppm)


200.68
137.21
133.03
128.70
128.20
38.72
29.47
29.35
24.43
f1 (ppm)


7.96
7.94
7.57
7.55
7.54
7.48
7.46
7.44
7.29
7.27
7.26
7.20
7.19
7.17
2.97
2.95
2.93
2.91
2.89
2.87
2.74
2.72
2.70
2.39
2.37
2.36
1.76
1.74
1.72
1.70
1.68
1.59
1.57
1.55
1.54
1.52
1.35
1.34
1.33
1.30
1.29
1.26
0.00


O
O
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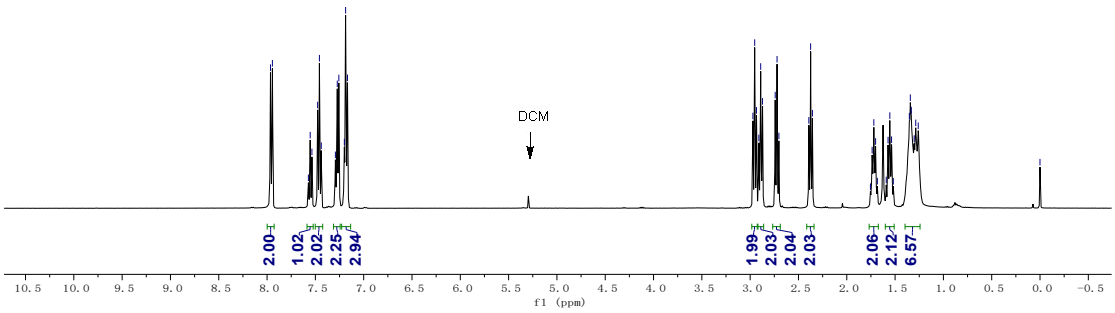
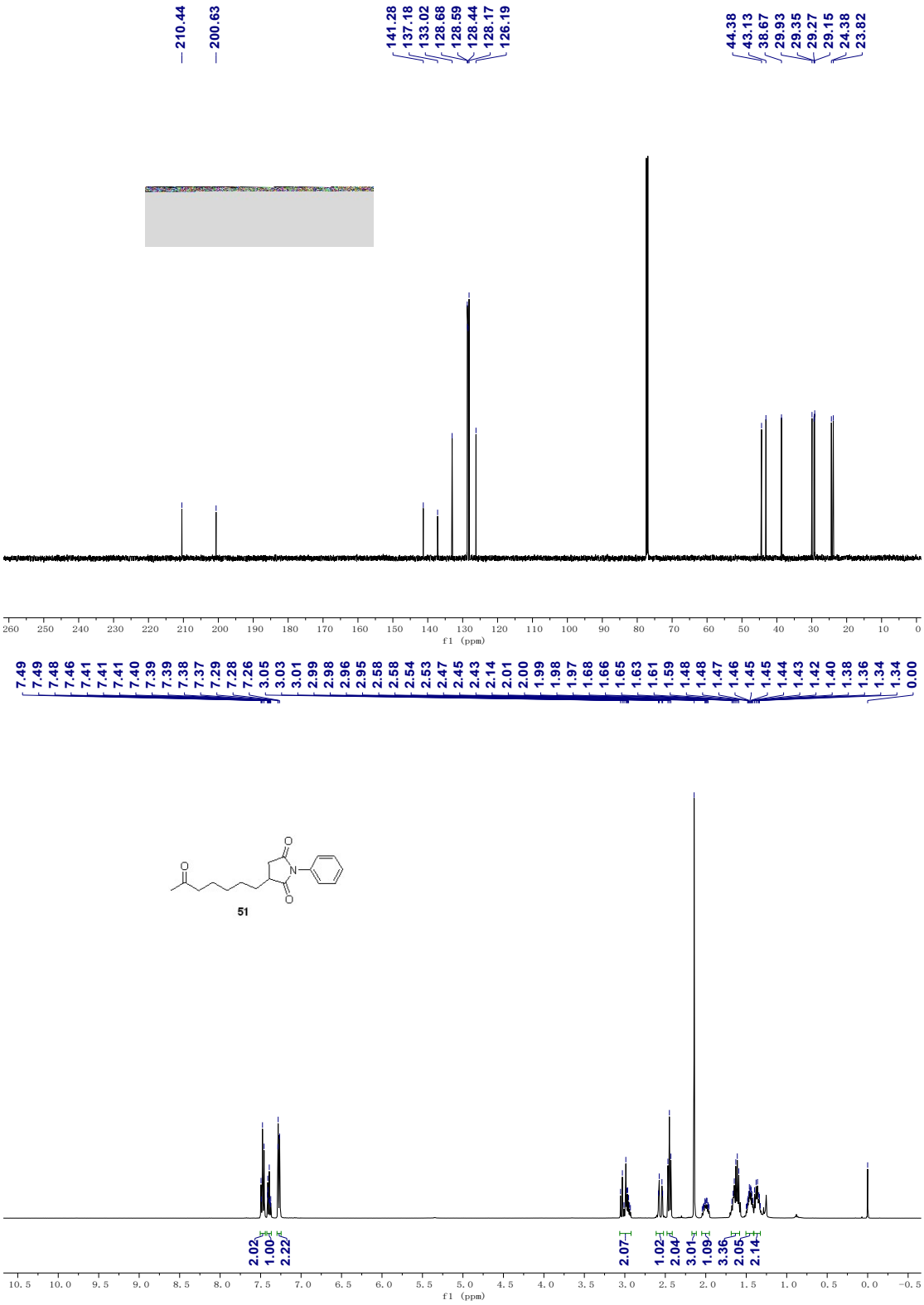
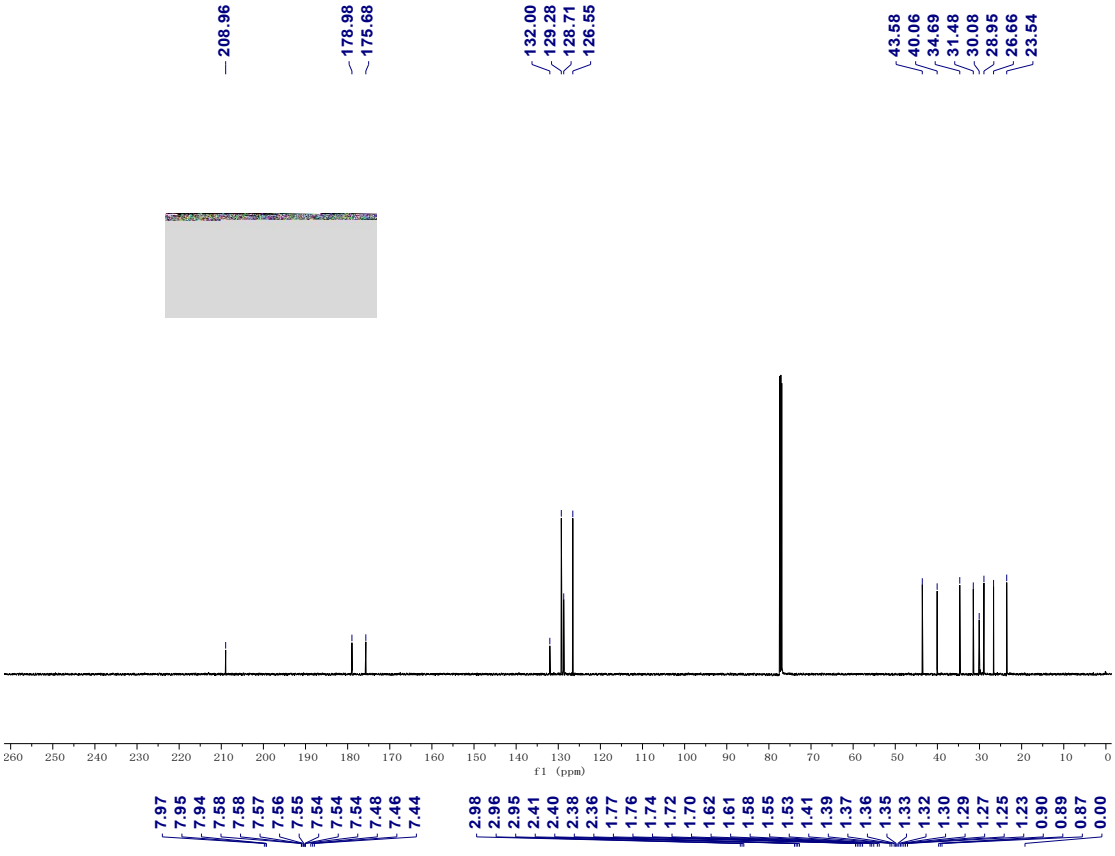

DCM
2.00
1.02
2.02
2.25
2.94
1.99
2.03
2.04
2.03
2.06
2.12
6.57
f1 (ppm)


210.44
200.63
141.28
137.18
133.02
128.68
128.59
128.44
128.17
126.19
44.38
43.13
38.67
29.93
29.35
29.27
29.15
24.38
23.82
f1 (ppm)
7.49
7.49
7.48
7.46
7.41
7.41
7.41
7.40
7.39
7.39
7.38
7.37
7.29
7.28
7.26
3.05
3.03
3.01
2.99
2.98
2.96
2.95
2.58
2.58
2.54
2.53
2.47
2.45
2.43
2.14
2.01
2.00
1.99
1.98
1.97
1.68
1.66
1.65
1.63
1.61
1.59
1.48
1.48
1.47
1.46
1.45
1.45
1.44
1.43
1.42
1.40
1.38
1.36
1.34
1.34
0.00
51
2.02
1.00
2.22
2.07
1.02
2.04
3.01
1.09
3.36
2.05
2.14
f1 (ppm)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178.98
175.68
132.00
129.28
128.71
126.55
43.58
40.06
34.69
31.48
30.08
28.95
26.66
23.54
f1 (ppm)
7.97
7.95
7.94
7.58
7.58
7.57
7.56
7.55
7.54
7.54
7.54
7.48
7.46
7.44
2.98
2.96
2.95
2.41
2.40
2.38
2.36
1.77
1.76
1.74
1.72
1.70
1.62
1.61
1.58
1.55
1.53
1.41
1.39
1.37
1.36
1.35
1.33
1.32
1.30
1.29
1.27
1.25
1.23
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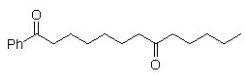
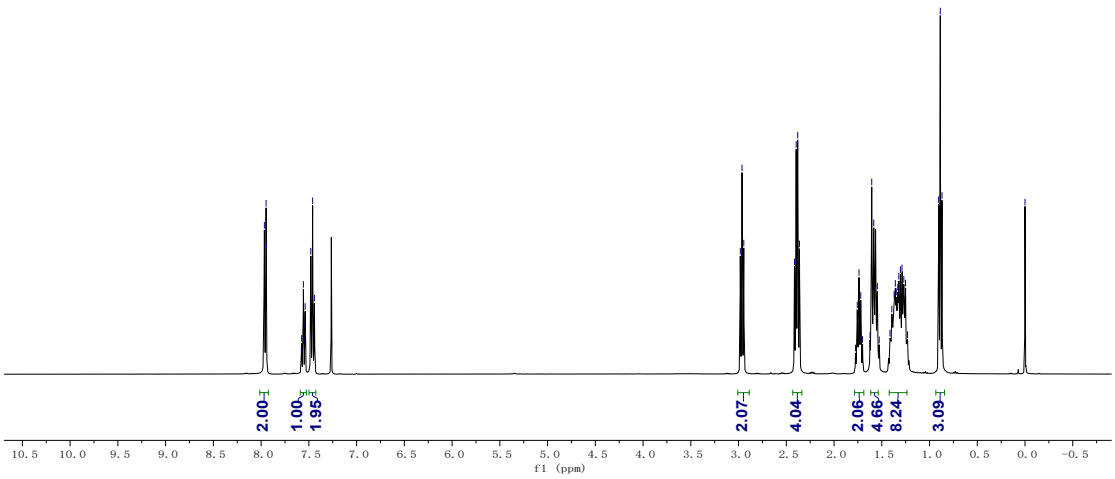
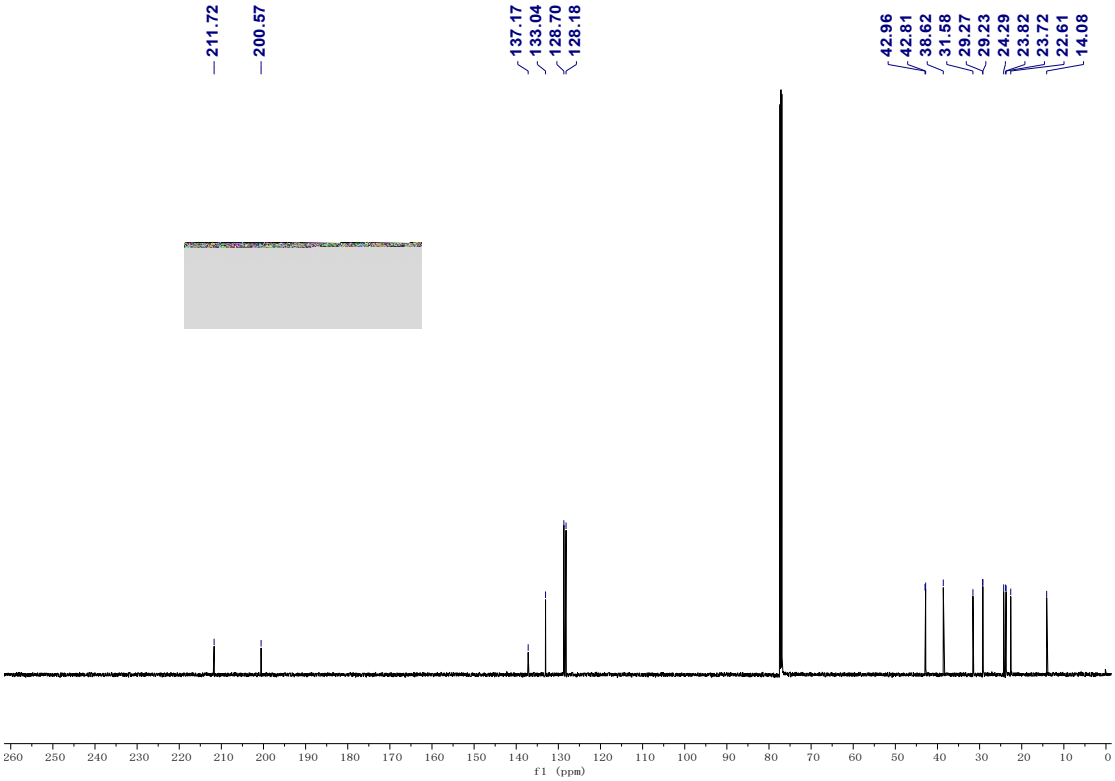
0.87
0.00

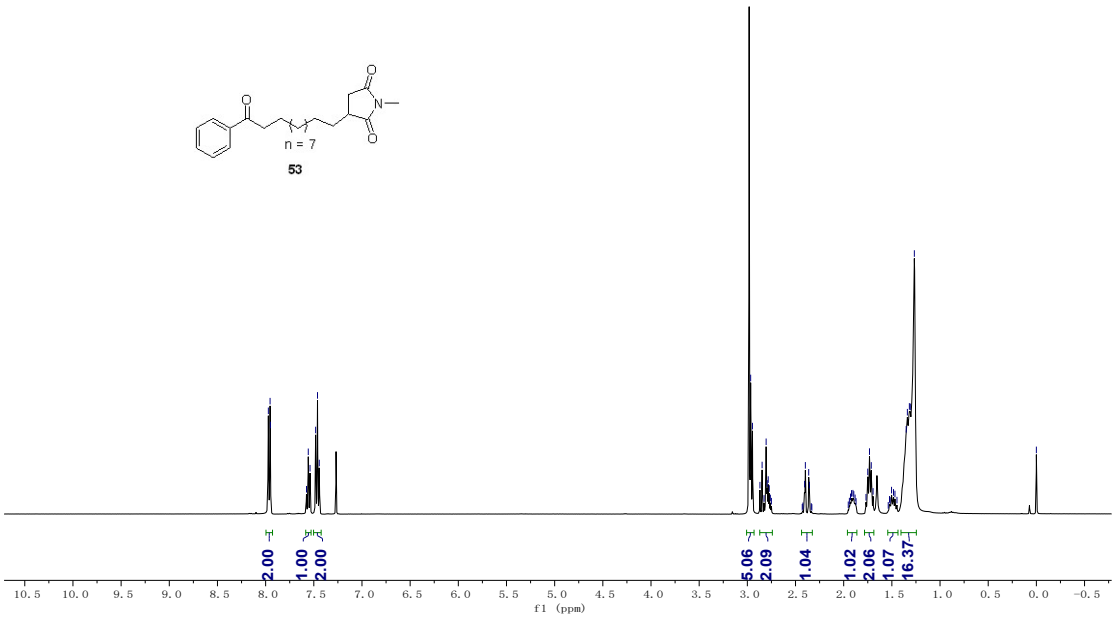

Ph
O
O

52


2.00
1.00
1.95
2.07
4.04
2.06
4.68
8.24
3.09
f1 (ppm)


211.72
200.57
137.17
133.04
128.70
128.18
42.96
42.81
38.62
31.58
29.27
29.23
24.29
23.82
23.72
22.61
14.08
f1 (ppm)


7.97
7.95
7.95
7.58
7.56
7.54
7.48
7.46
7.44
2.97
2.95
2.87
2.85
2.83
2.81
2.79
2.78
2.77
2.76
2.75
2.43
2.42
2.41
2.40
2.36
2.36
2.34
1.95
1.94
1.93
1.92
1.90
1.89
1.88
1.87
1.77
1.75
1.73
1.71
1.70
1.54
1.52
1.52
1.50
1.48
1.47
1.45
1.35
1.34
1.32
1.31
0.07
0.00


n = 7
53
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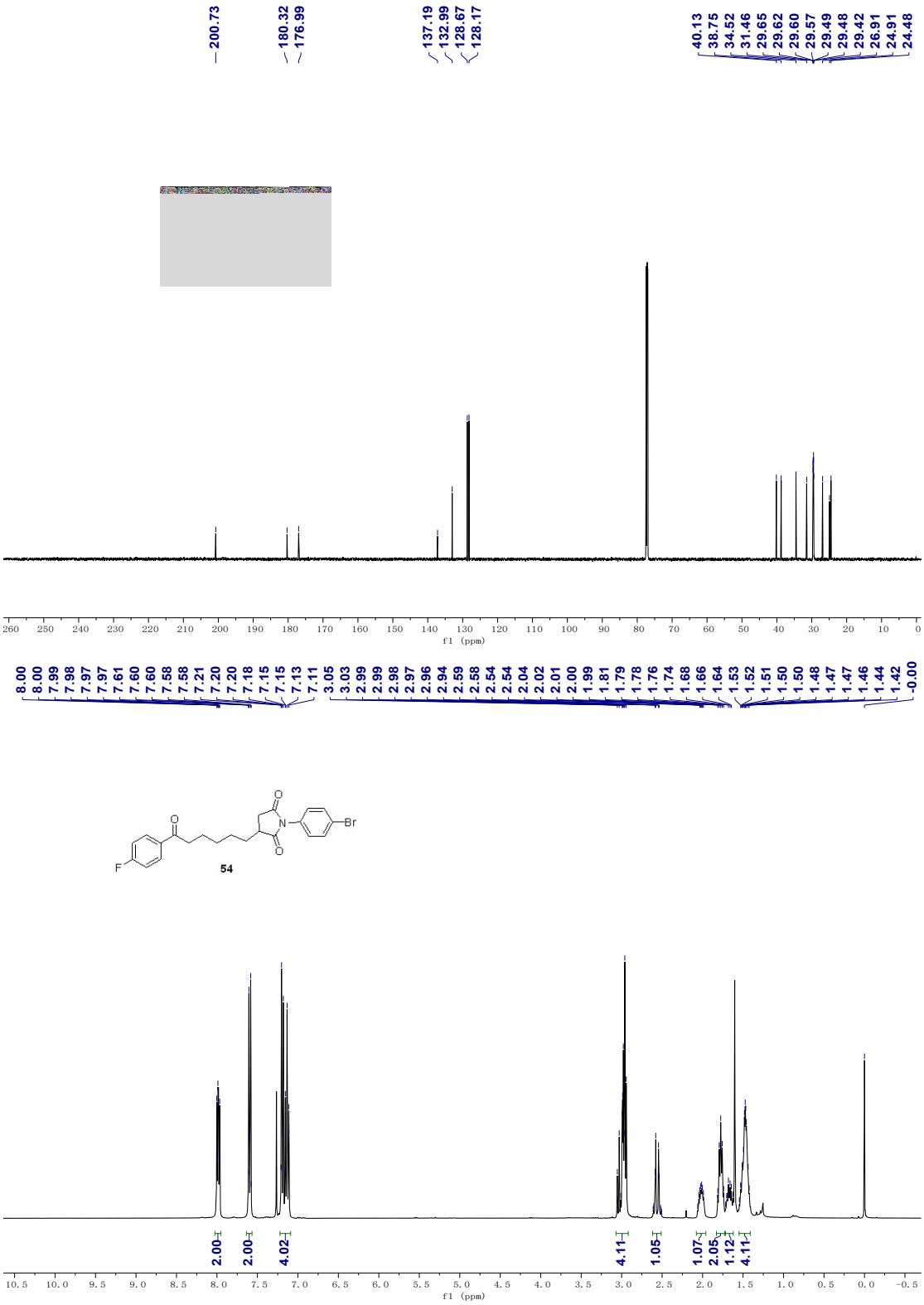
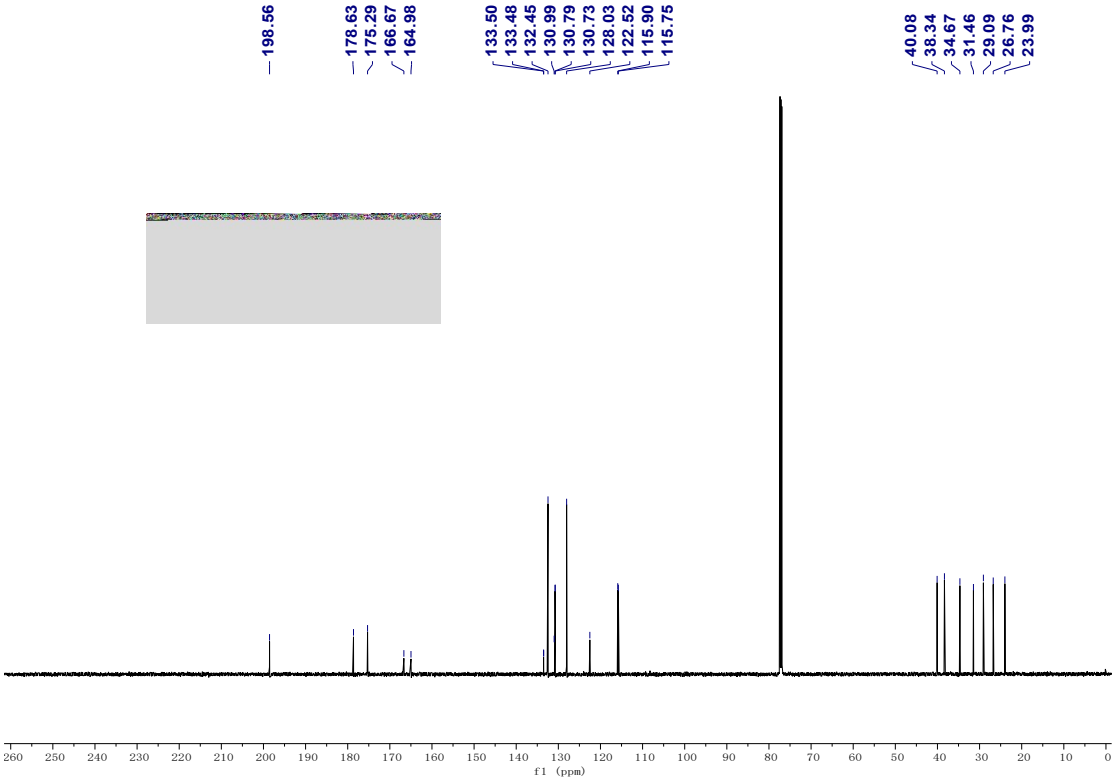
1.00
2.00
5.06
2.09
1.04
1.02
2.06
1.07
16.37
f1 (ppm)


200.73
180.32
176.99
137.19
132.99
128.67
128.17
40.13
38.75
34.52
31.46
29.65
29.62
29.60
29.57
29.49
29.48
29.42
26.91
24.91
24.48
f1 (ppm)
8.00
8.00
7.99
7.98
7.97
7.97
7.61
7.60
7.60
7.58
7.58
7.21
7.20
7.20
7.18
7.15
7.15
7.13
7.11
3.05
3.03
2.99
2.99
2.98
2.97
2.96
2.94
2.59
2.58
2.54
2.54
2.04
2.02
2.01
2.00
1.99
1.81
1.79
1.78
1.76
1.74
1.68
1.66
1.64
1.53
1.52
1.51
1.50
1.50
1.48
1.47
1.47
1.46
1.44
1.42
-0.00
O
O
N
Br
O
F
54
2.00
2.00
4.02
4.11
1.05
1.07
2.05
1.12
4.11
f1 (ppm)


198.56
178.63
175.29
166.67
164.98
133.50
133.48
132.45
130.99
130.79
130.73
128.03
122.52
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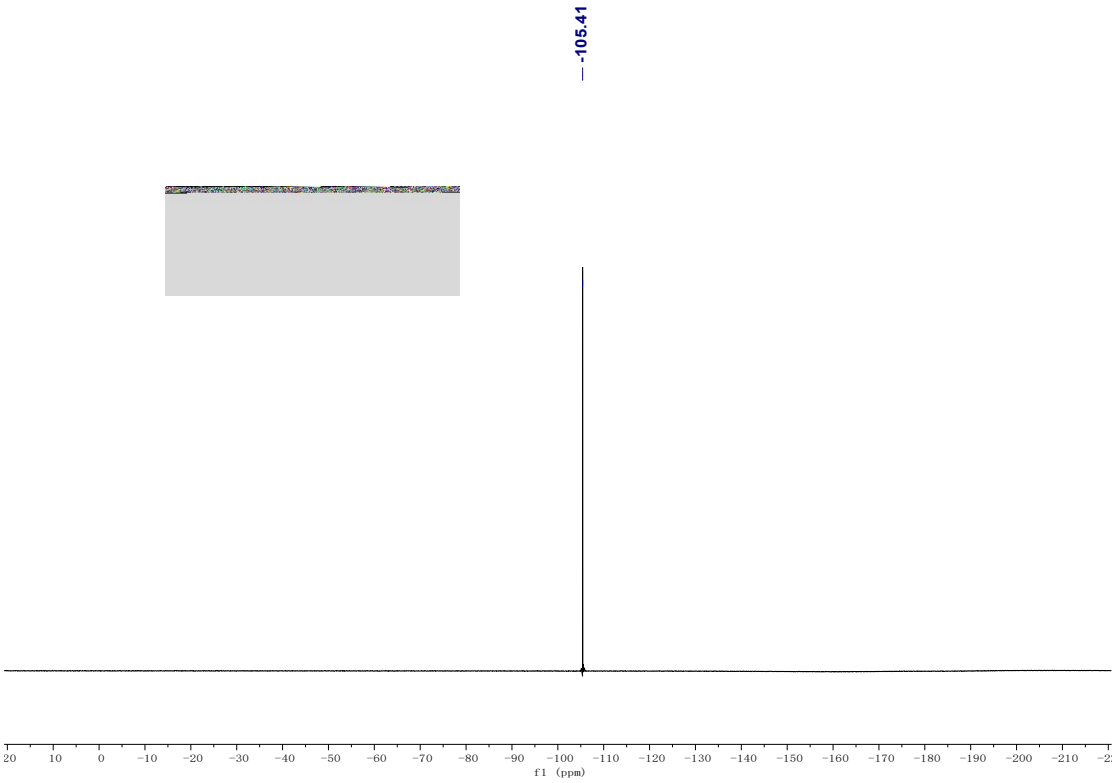
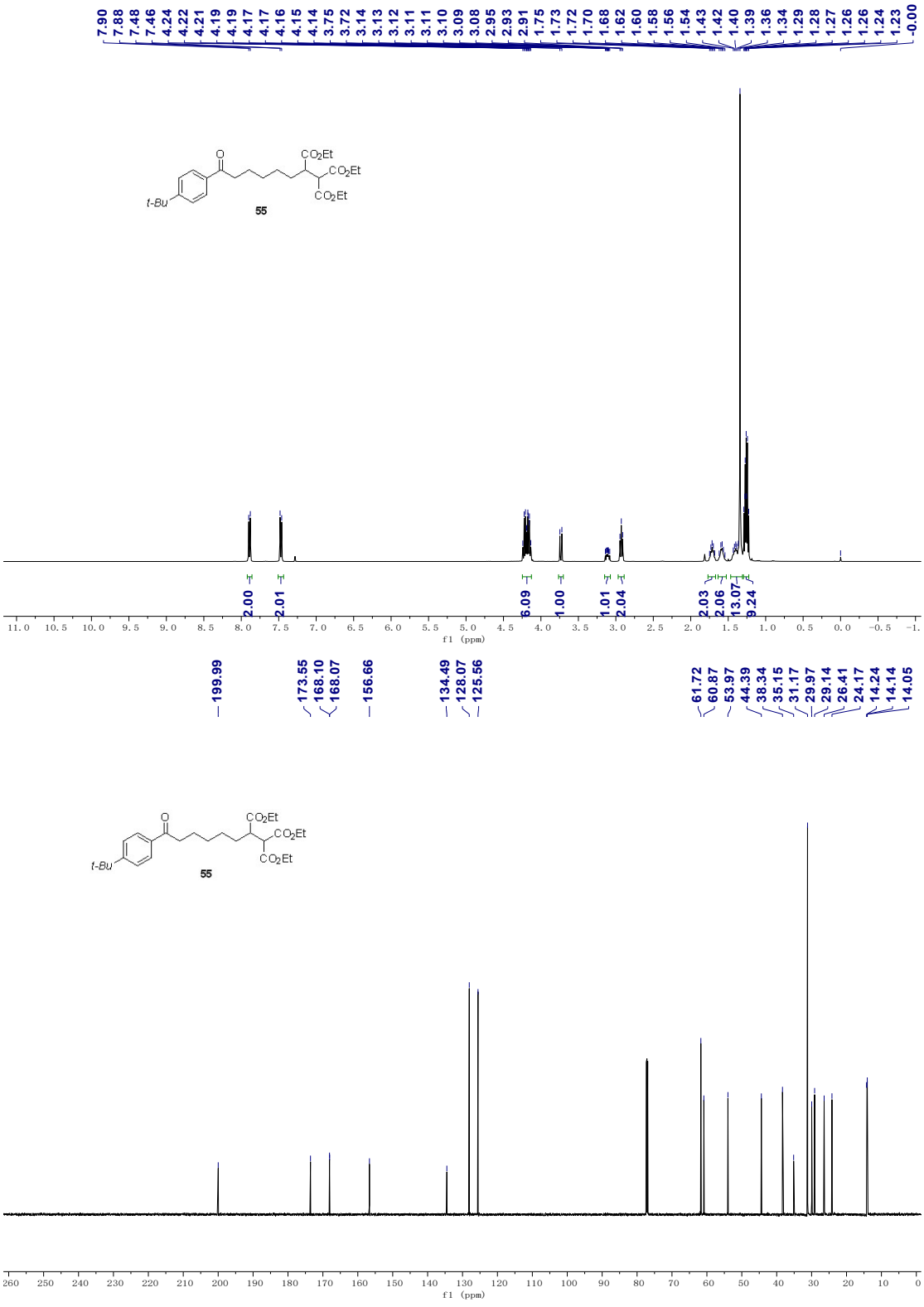
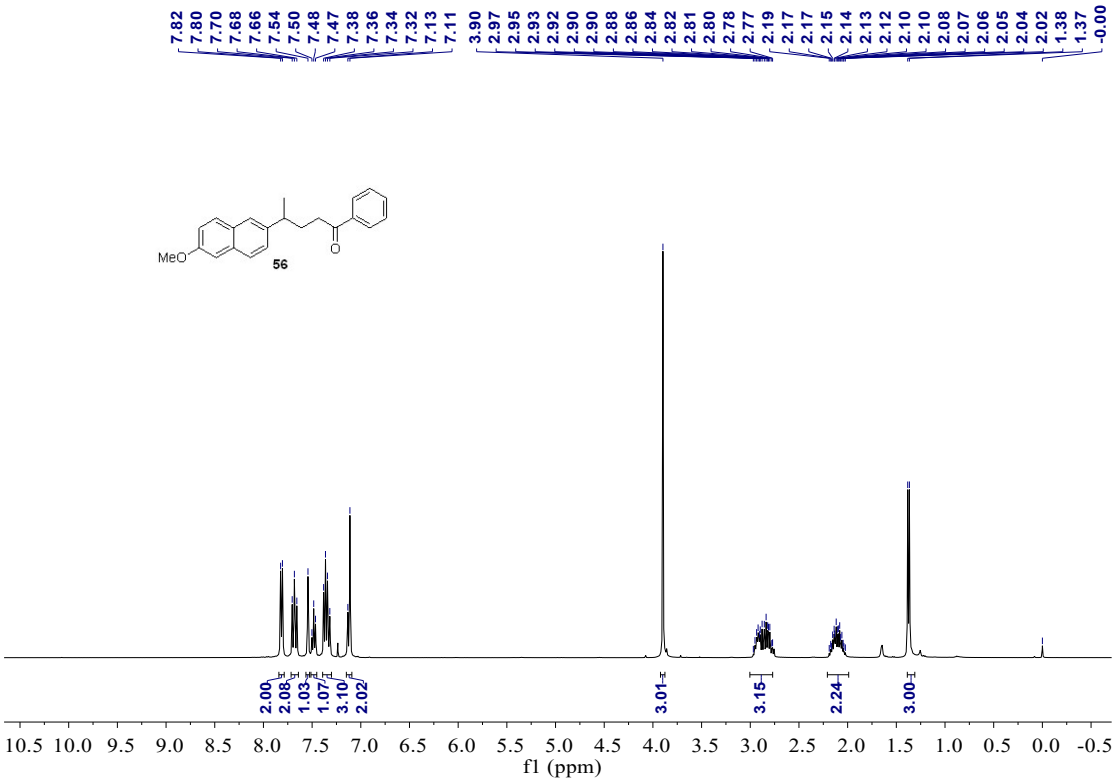
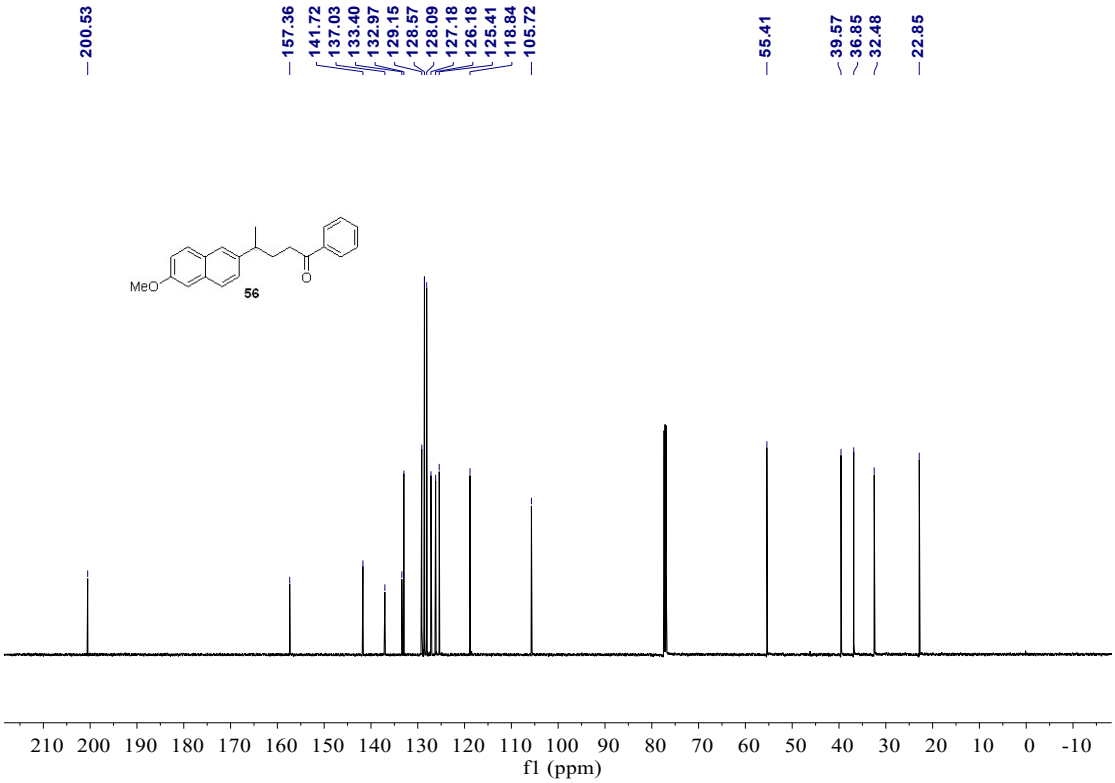
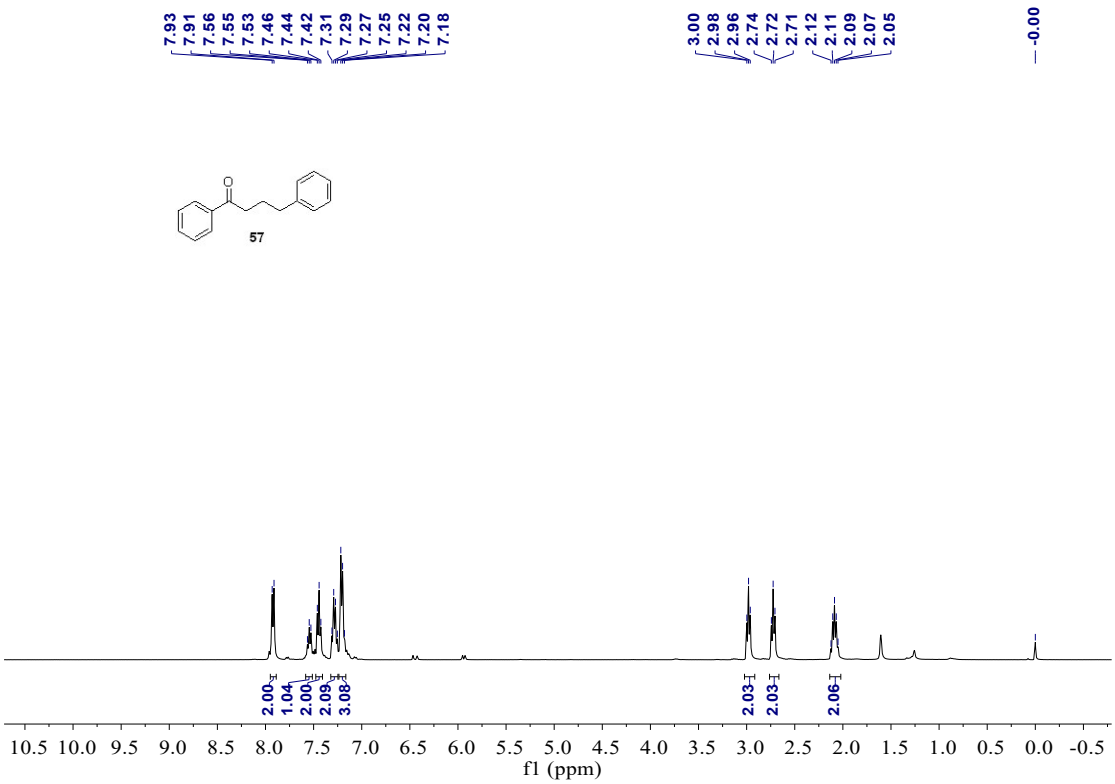
34.67
31.46
29.09
26.76
23.99
f1 (ppm)

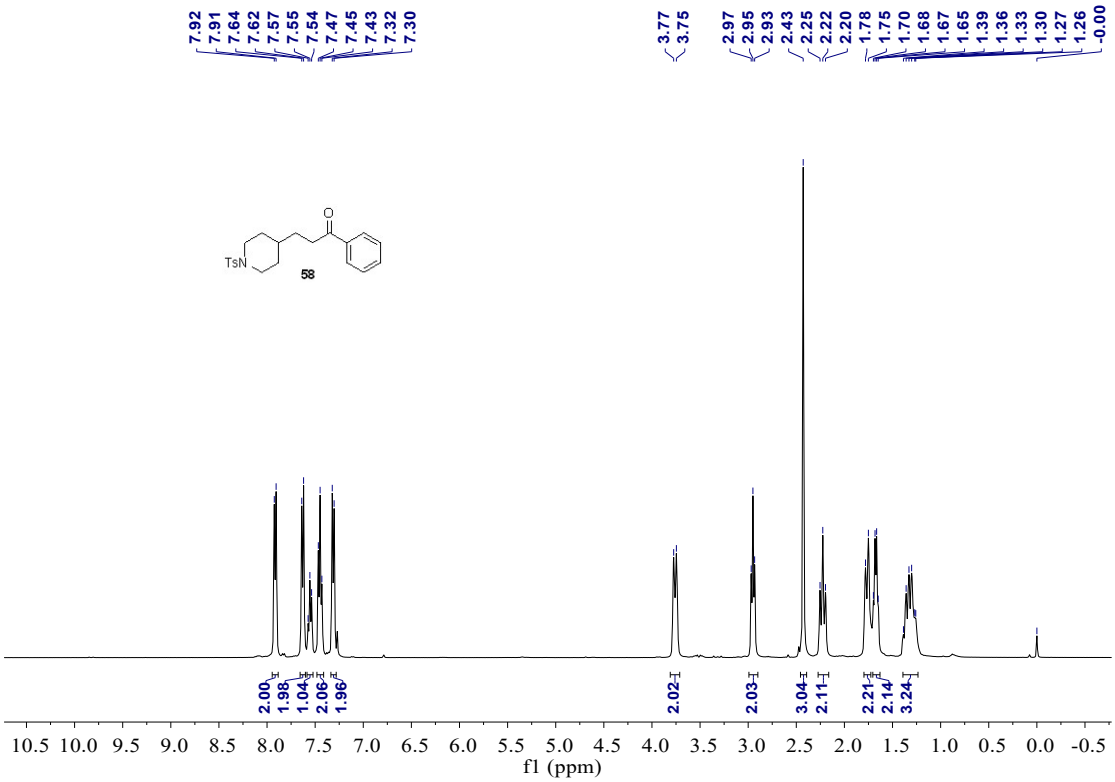

-105.41
f1 (ppm)

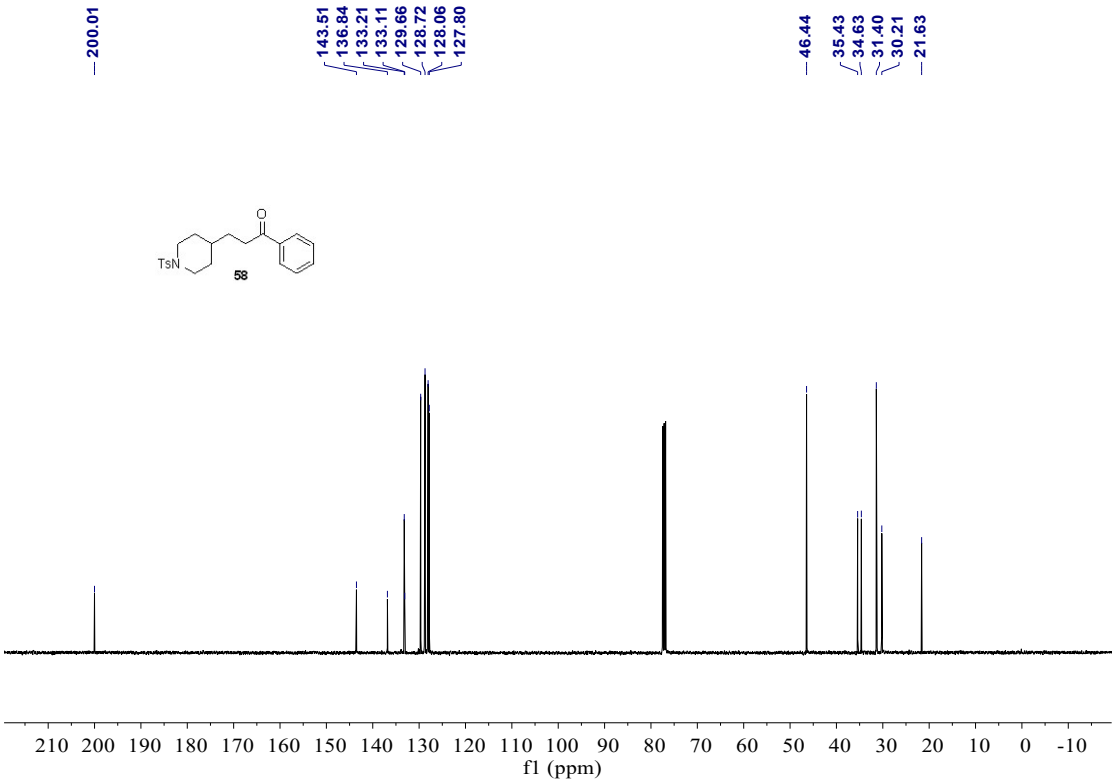

7.90 7.88 7.48 7.46 4.24 4.22 4.21 4.19 4.19 4.17 4.17 4.16 4.15 4.14 3.75 3.72 3.14 3.13 3.12 3.11 3.11 3.10 3.09 3.08 2.95 2.93 2.91 1.75 1.73 1.72 1.70 1.68 1.62 1.60 1.58 1.56 1.54 1.43 1.42 1.40 1.39 1.36 1.34 1.29 1.28 1.27 1.26 1.26 1.24 1.23 -0.00
O
CO2Et
CO2Et
CO2Et
t-Bu
55
2.00 2.01 6.09 1.00 1.01 2.04 2.03 2.06 13.07 9.24
f1 (ppm)
199.99 173.55 168.10 168.07 156.66 134.49 128.07 125.56 61.72 60.87 53.97 44.39 38.34 35.15 31.17 29.97 29.14 26.41 24.17 14.24 14.14 14.05
O
CO2Et
CO2Et
CO2Et
t-Bu
55
f1 (ppm)

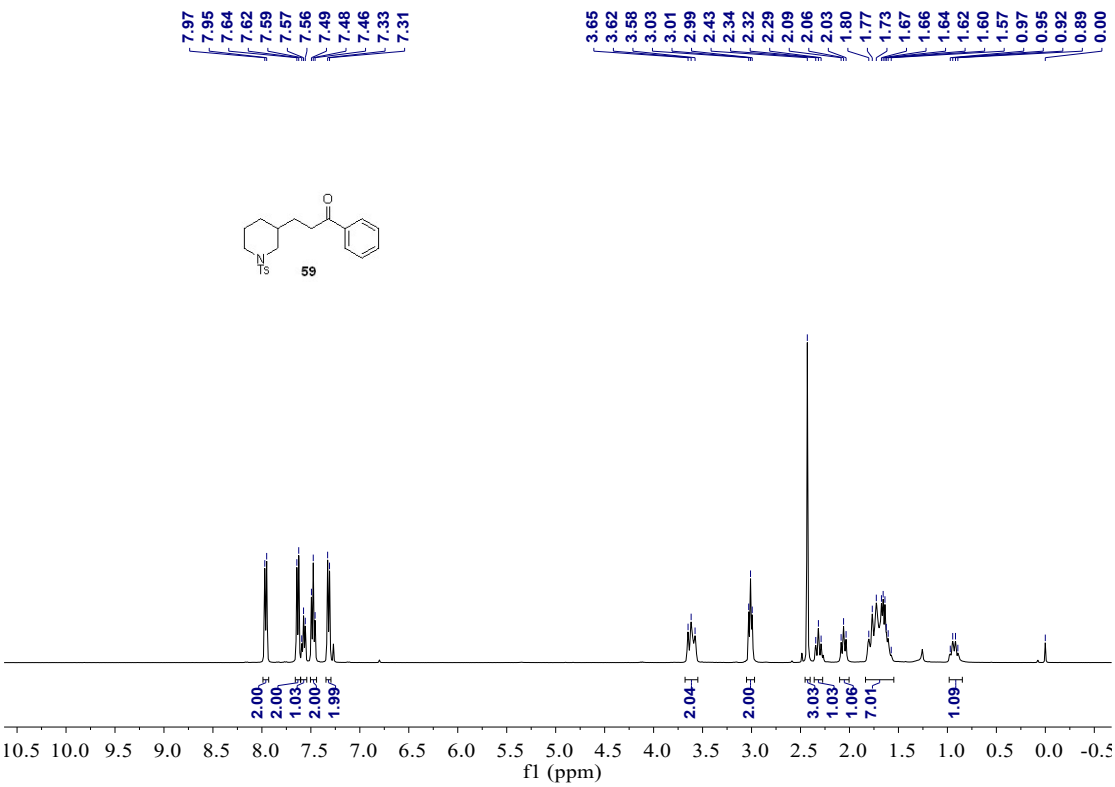

7.82 7.80 7.70 7.68 7.66 7.54 7.50 7.48 7.47 7.38 7.36 7.34 7.32 7.13 7.11
3.90 2.97 2.95 2.93 2.92 2.90 2.90 2.88 2.86 2.84 2.82 2.81 2.80 2.78 2.77 2.19 2.17 2.17 2.15 2.14 2.13 2.12 2.10 2.10 2.08 2.07 2.06 2.05 2.04 2.02 1.38 1.37 -0.00
MeO
56
2.00 2.05 1.03 1.07 3.10 2.02
3.01 3.15 2.24 3.00
10.5 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 -0.5
f1 (ppm)

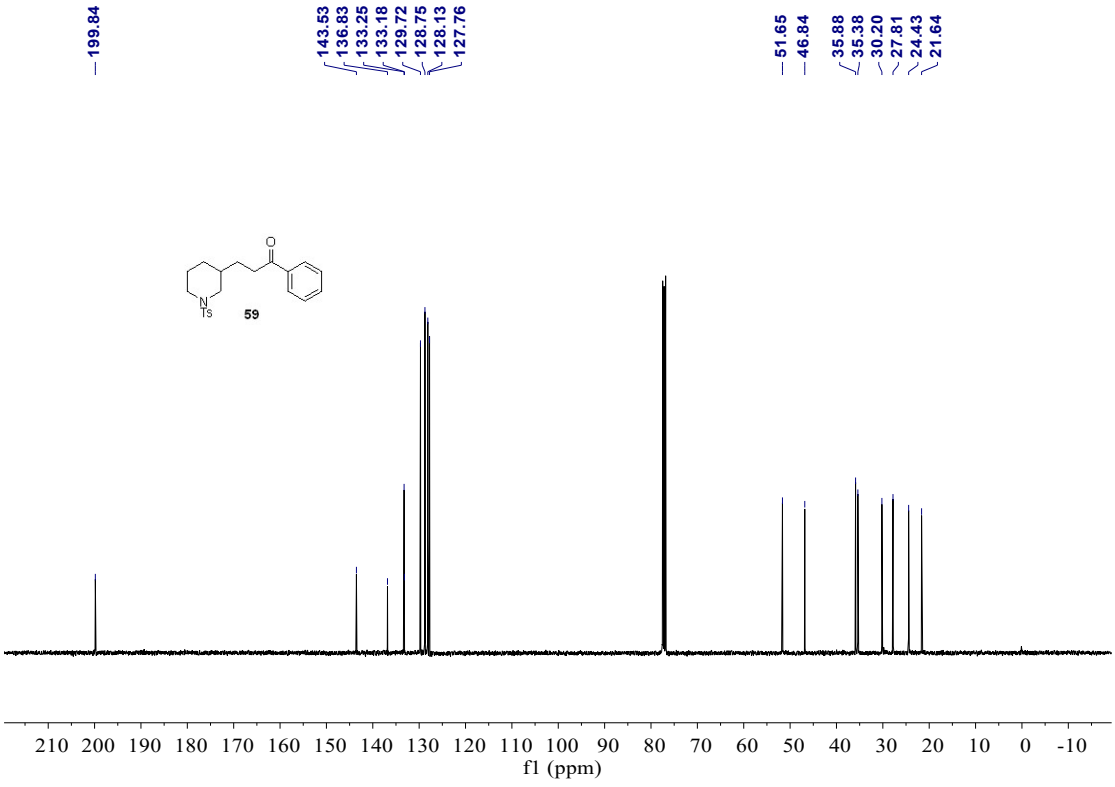

200.53
157.36 141.72 137.03 133.40 132.97 129.15 128.57 128.09 127.18 126.18 125.41 118.84 105.72
55.41
39.57 36.85 32.48
22.85
MeO
56
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.93
7.91
7.56
7.55
7.53
7.46
7.44
7.42
7.31
7.29
7.27
7.25
7.22
7.20
7.18
3.00
2.98
2.96
2.74
2.72
2.71
2.12
2.11
2.09
2.07
2.05
-0.00
57
2.00
1.04
2.00
2.09
3.08
2.03
2.03
2.06
f1 (ppm)
200.26
141.81
137.11
133.08
128.69
128.65
128.53
128.14
126.08
37.82
35.33
25.83
57
f1 (ppm)


7.92
7.91
7.64
7.62
7.57
7.55
7.54
7.47
7.45
7.43
7.32
7.30
3.77
3.75
2.97
2.95
2.93
2.43
2.25
2.22
2.20
1.78
1.75
1.70
1.68
1.67
1.65
1.39
1.36
1.33
1.30
1.27
1.26
-0.00
TsN
58
2.00
1.98
1.04
2.06
1.96
2.02
2.03
3.04
2.11
2.21
2.14
3.24
f1 (ppm)


200.01
143.51
136.84
133.21
133.11
129.66
128.72
128.06
127.80
46.44
35.43
34.63
31.40
30.21
21.63
TsN
58
f1 (ppm)


7.97
7.95
7.64
7.62
7.59
7.57
7.56
7.49
7.48
7.46
7.33
7.31
3.65
3.62
3.58
3.03
3.01
2.99
2.43
2.34
2.32
2.29
2.09
2.06
2.03
1.80
1.77
1.73
1.67
1.66
1.64
1.62
1.60
1.57
0.97
0.95
0.92
0.89
0.00
O
N
Ts
59
2.00
2.00
1.03
2.00
1.99
2.04
2.00
3.03
1.03
1.06
7.01
1.09
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 -0.5
f1 (ppm)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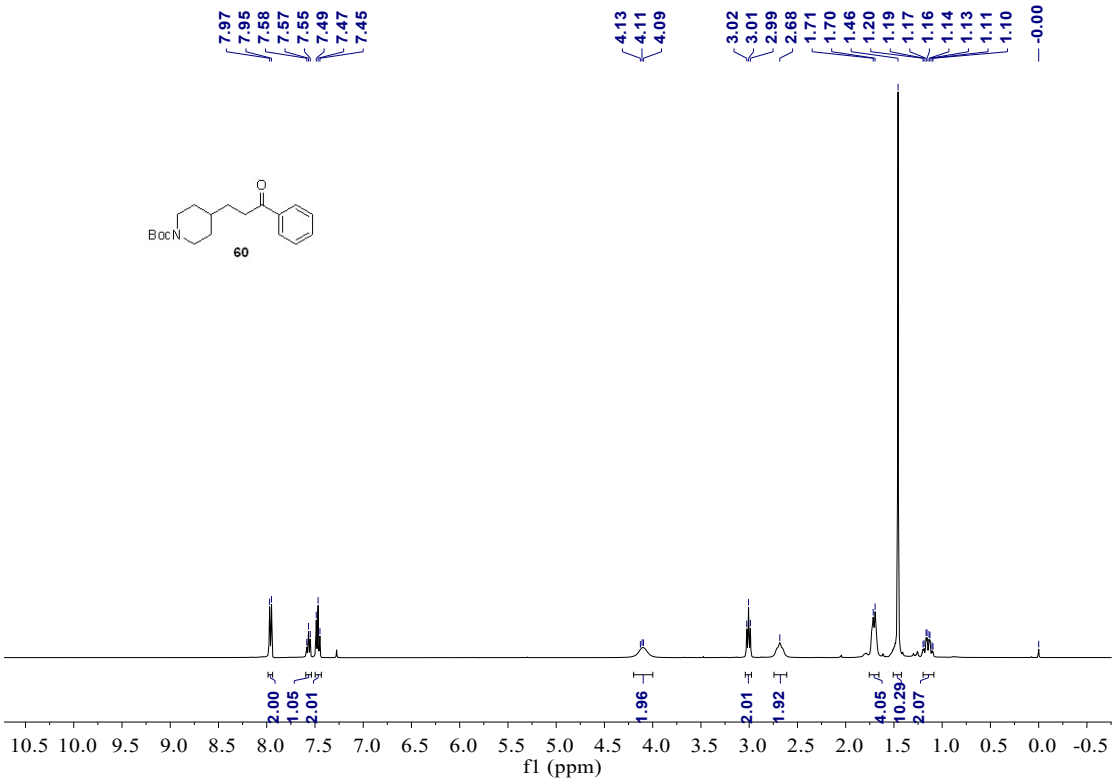
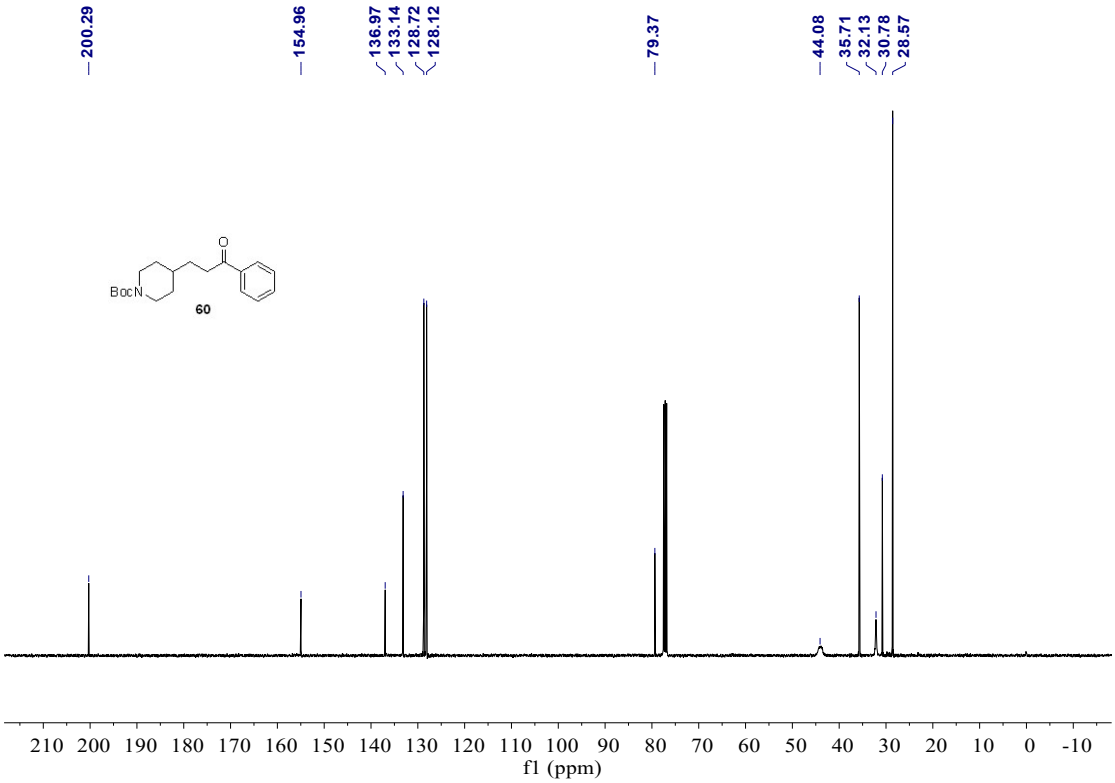
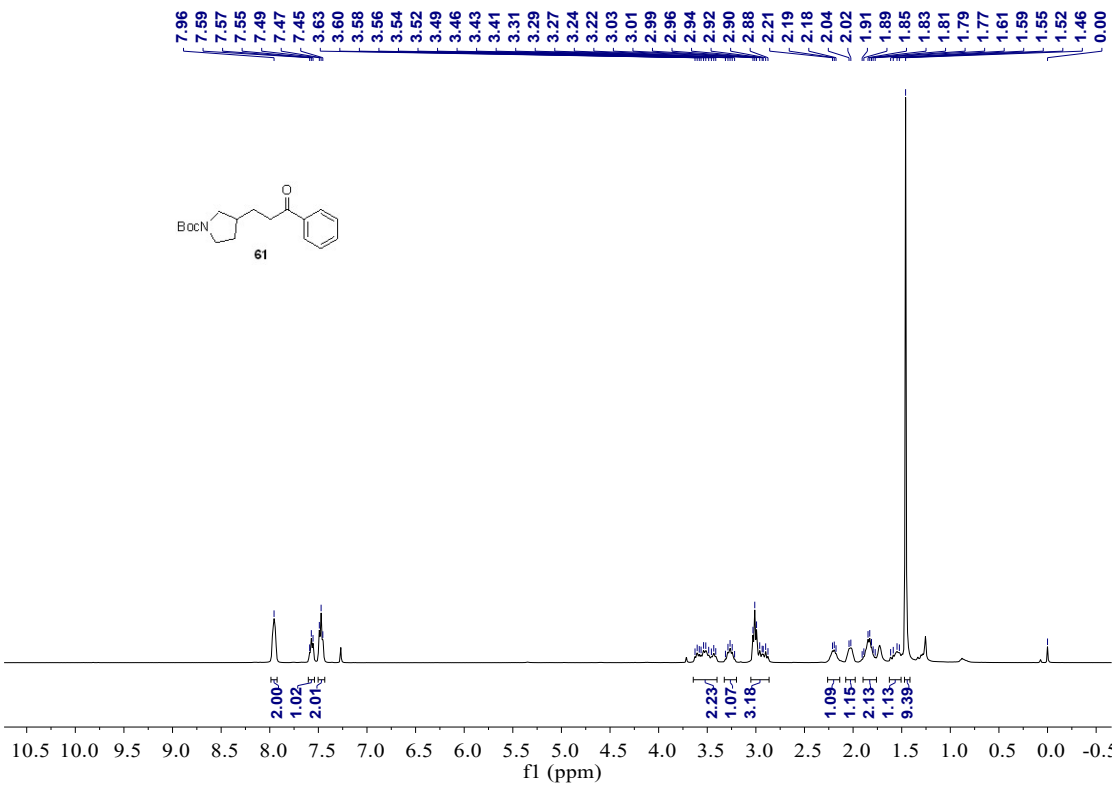
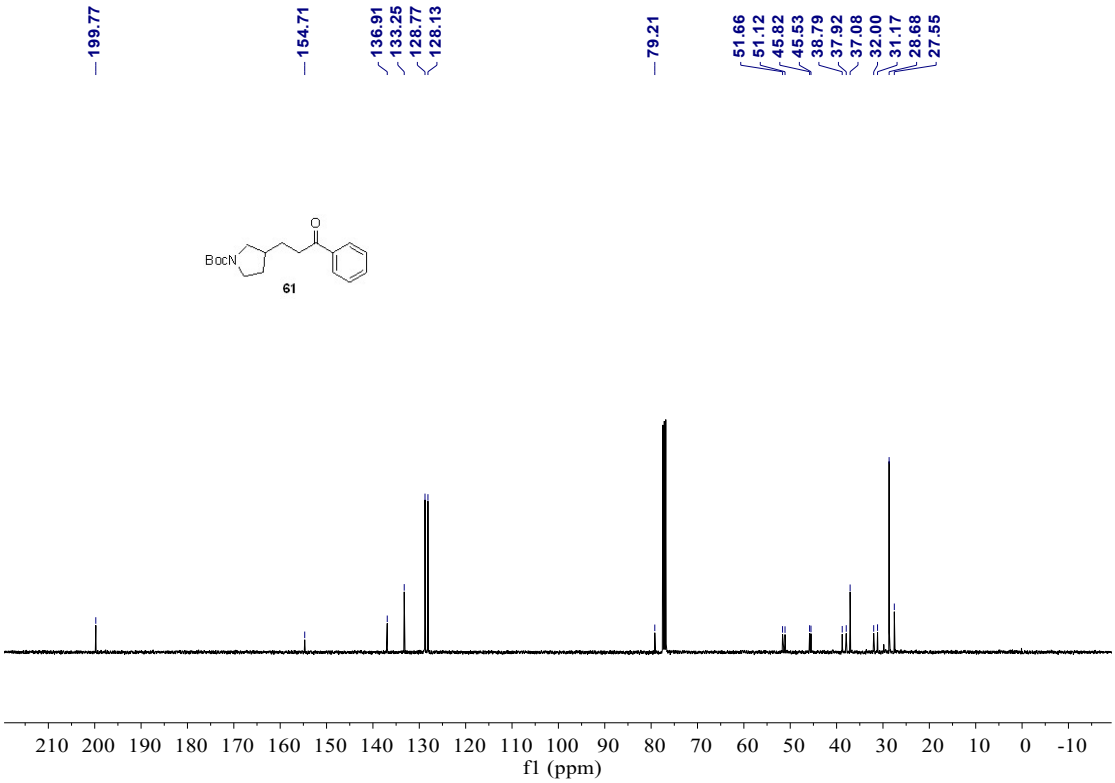
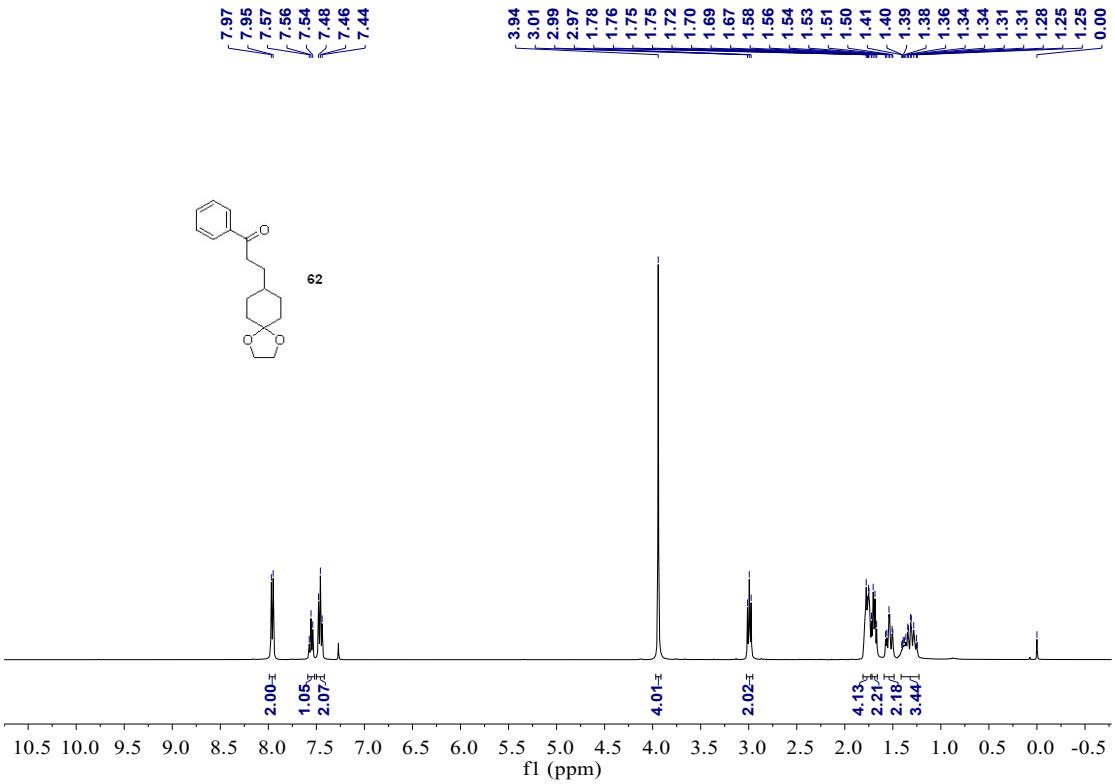
143.53
136.83
133.25
133.18
129.72
128.75
128.13
127.76
51.65
46.84
35.88
35.38
30.20
27.81
24.43
21.64
O
N
Ts
59
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

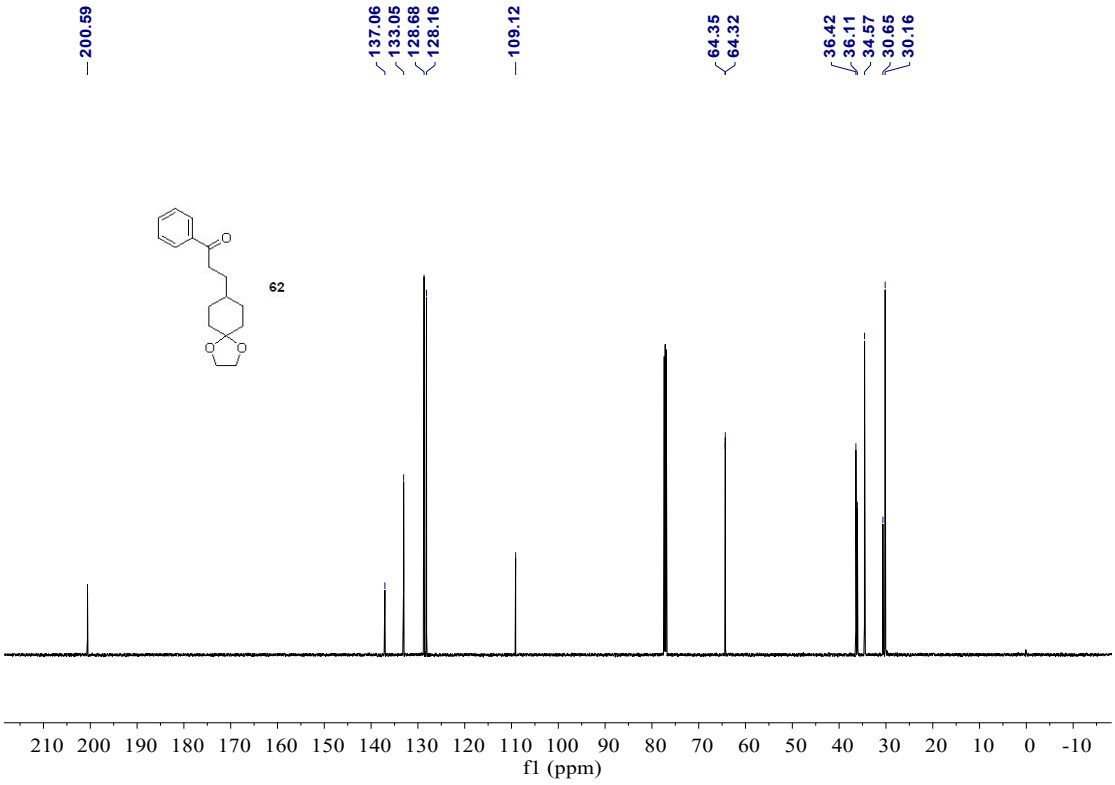

7.97
7.95
7.58
7.57
7.55
7.49
7.47
7.45
4.13
4.11
4.09
3.02
3.01
2.99
2.68
1.71
1.70
1.46
1.20
1.19
1.17
1.16
1.14
1.13
1.11
1.10
-0.00
BocN
60
2.00
1.05
2.01
1.96
2.01
1.92
4.05
10.29
2.07
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

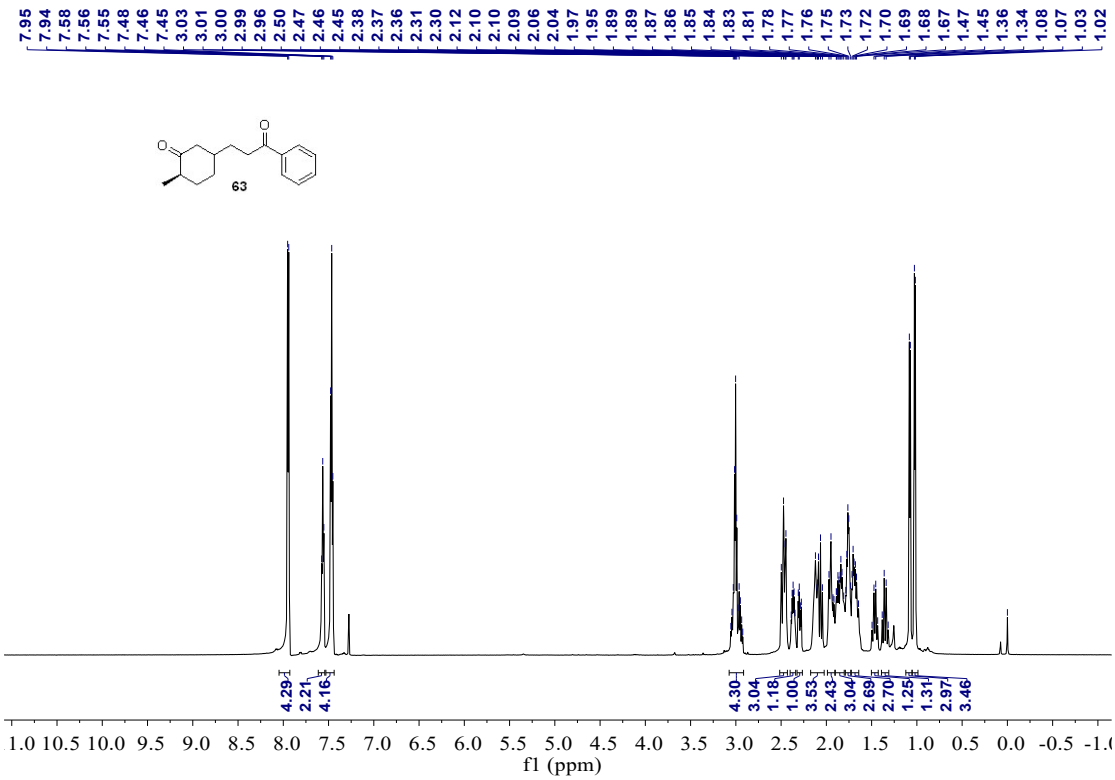

200.29
154.96
136.97
133.14
128.72
128.12
79.37
44.08
35.71
32.13
30.78
28.57
BocN
60
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

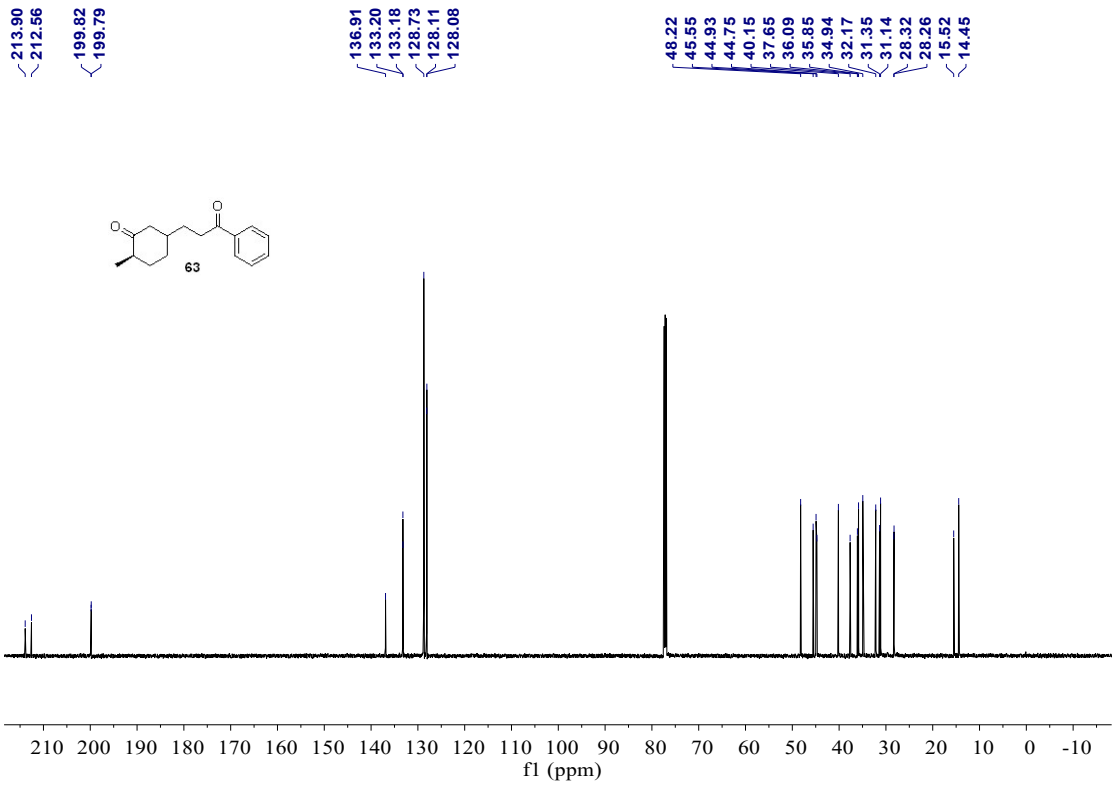

7.96 7.59 7.57 7.55 7.49 7.47 7.45 3.63 3.60 3.58 3.56 3.54 3.52 3.49 3.46 3.43 3.41 3.31 3.29 3.27 3.24 3.22 3.03 3.01 2.99 2.96 2.94 2.92 2.90 2.88 2.21 2.19 2.18 2.04 2.02 1.91 1.89 1.85 1.83 1.81 1.79 1.77 1.61 1.59 1.55 1.52 1.46 0.00
BocN
61
2.00 1.02 2.01 2.23 1.07 3.18 1.09 1.15 2.13 1.13 9.39
f1 (ppm)

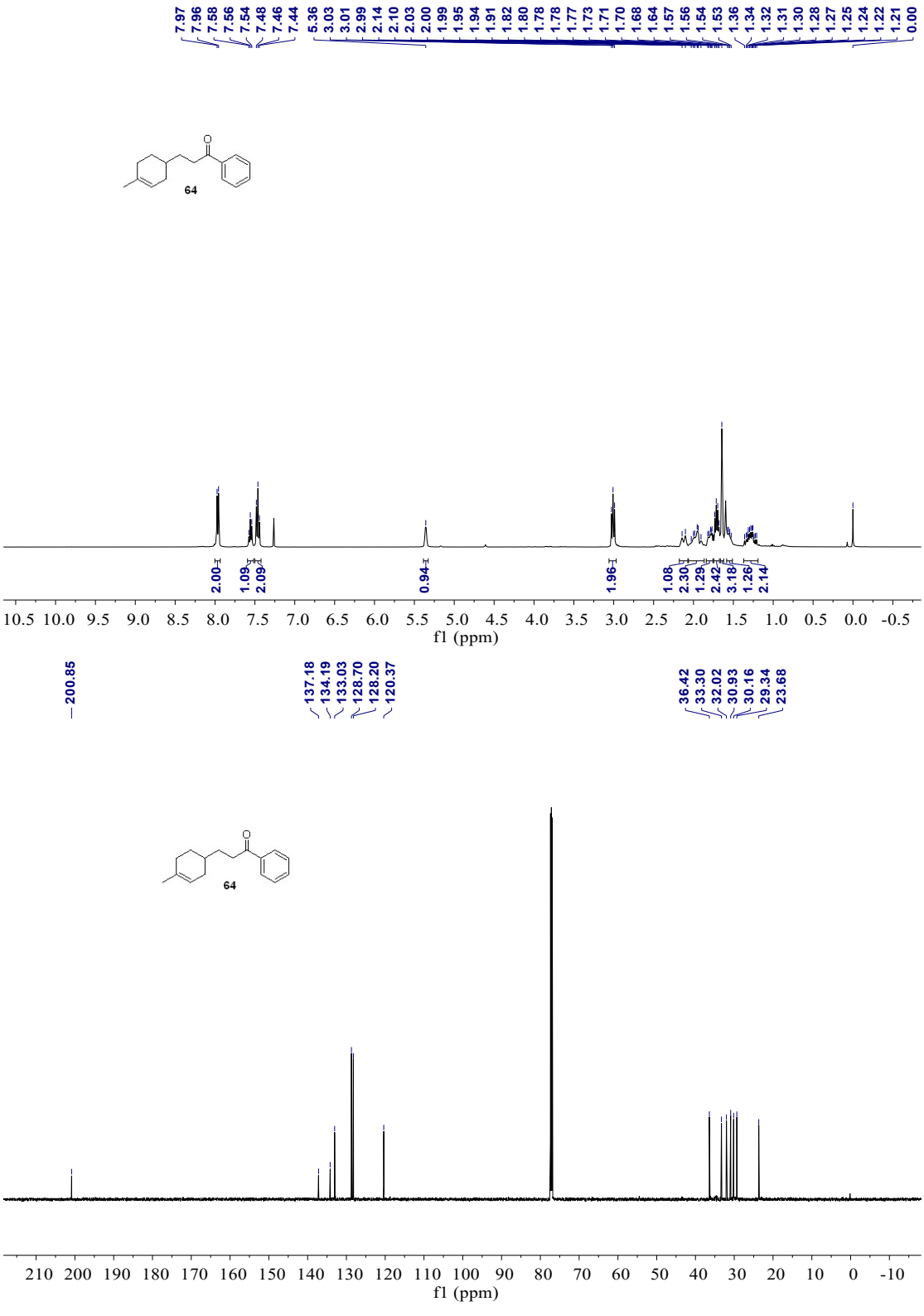

199.77 154.71 136.91 133.25 128.77 128.13 79.21 51.66 51.12 45.82 45.53 38.79 37.92 37.08 32.00 31.17 28.68 27.55
BocN
61
f1 (ppm)

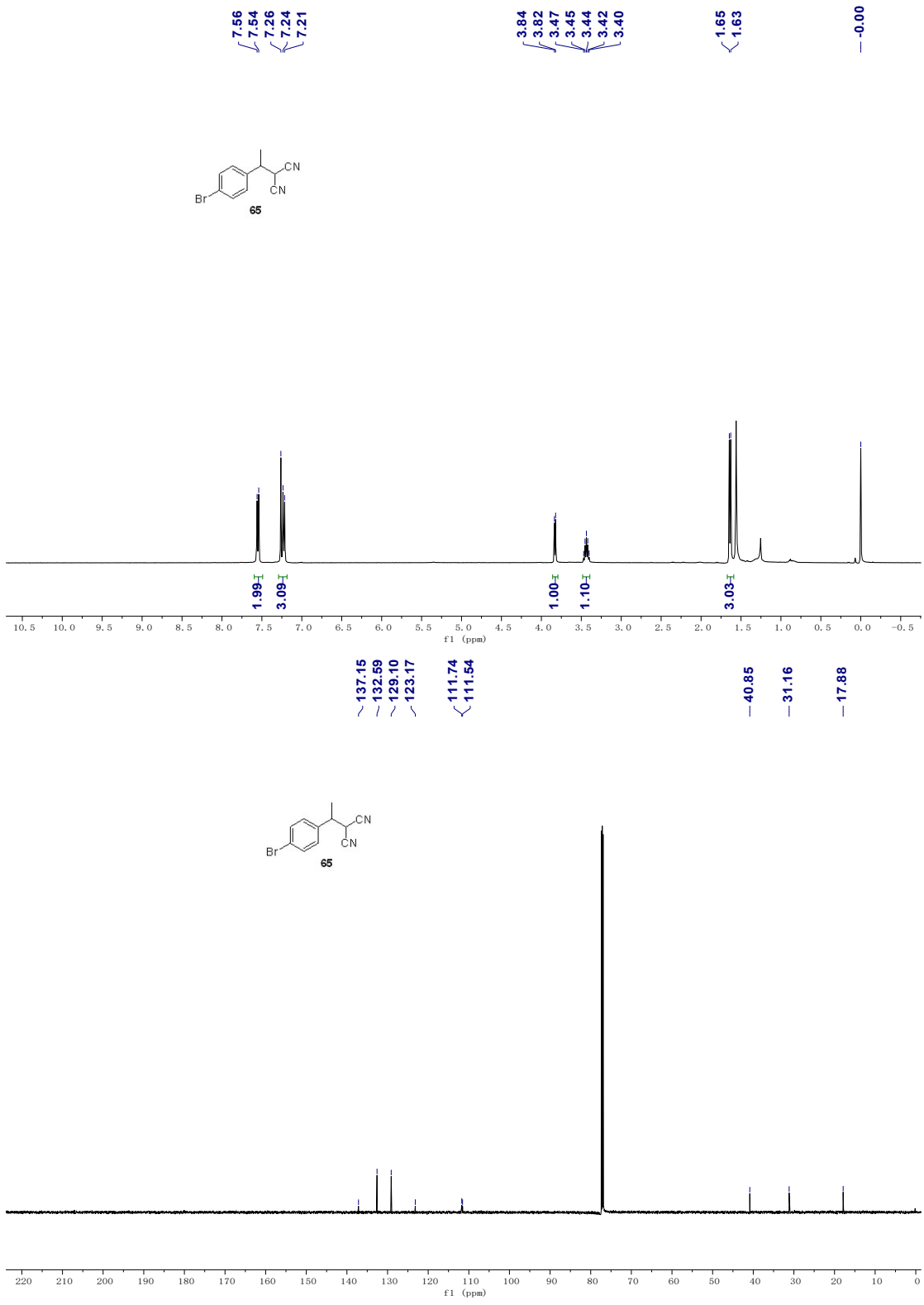

7.97
7.95
7.57
7.56
7.54
7.48
7.46
7.44
3.94
3.01
2.99
2.97
1.78
1.76
1.75
1.75
1.72
1.70
1.69
1.67
1.58
1.56
1.54
1.53
1.51
1.50
1.41
1.40
1.39
1.38
1.36
1.34
1.34
1.31
1.28
1.25
1.25
0.00
62
2.00
1.05
2.07
4.01
2.02
4.13
2.21
2.18
3.44
f1 (ppm)


200.59
137.06
133.05
128.68
128.16
109.12
64.35
64.32
36.42
36.11
34.57
30.65
30.16
62
f1 (ppm)

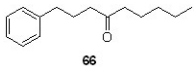

7.95 7.94 7.58 7.56 7.55 7.48 7.46 7.45 3.03 3.01 3.00 2.99 2.96 2.50 2.47 2.46 2.45 2.38 2.37 2.36 2.31 2.30 2.12 2.10 2.10 2.09 2.06 2.04 1.97 1.95 1.89 1.89 1.87 1.86 1.85 1.84 1.83 1.81 1.78 1.77 1.76 1.75 1.73 1.72 1.70 1.69 1.68 1.67 1.47 1.45 1.36 1.34 1.08 1.07 1.03 1.02
63
4.29 2.21 4.16 4.30 3.04 1.16 1.00 3.53 2.43 3.04 2.69 2.70 1.25 1.31 2.97 3.46
f1 (ppm)


213.90 212.56 199.82 199.79 136.91 133.20 133.18 128.73 128.11 128.08 48.22 45.55 44.93 44.75 40.15 37.65 36.09 35.85 34.94 32.17 31.35 31.14 28.32 28.26 15.52 14.45
63
f1 (ppm)

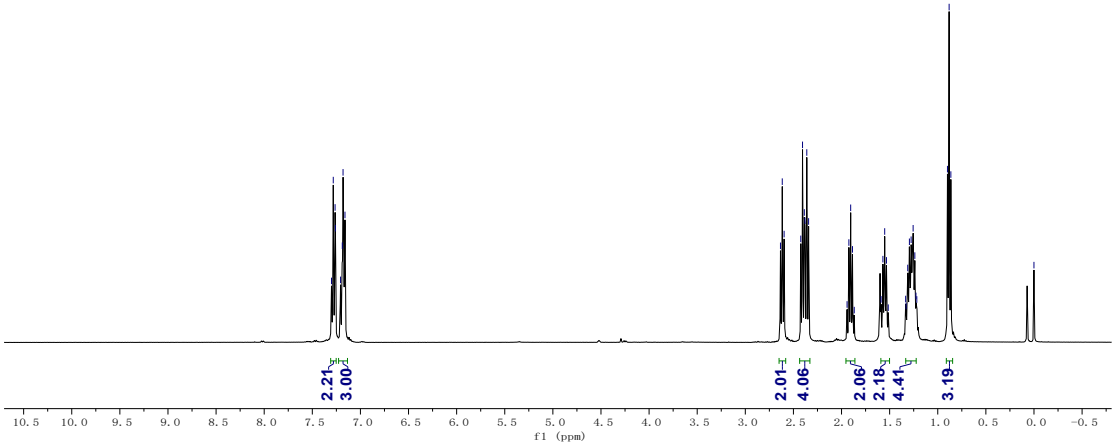

7.97 7.96 7.58 7.56 7.54 7.48 7.46 7.44 5.36 3.03 3.01 2.99 2.14 2.10 2.03 2.00 1.99 1.95 1.94 1.91 1.82 1.80 1.78 1.78 1.77 1.73 1.71 1.70 1.68 1.64 1.57 1.56 1.54 1.53 1.36 1.34 1.32 1.31 1.30 1.28 1.27 1.25 1.24 1.22 1.21 0.00
64
2.00 1.09 2.09 0.94 1.96 1.08 2.30 1.29 2.42 3.18 1.26 2.14
10.5 10.0 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 -0.5
f1 (ppm)
200.85 137.18 134.19 133.03 128.70 128.20 120.37 36.42 33.30 32.02 30.93 30.16 29.34 23.68
64
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.56
7.54
7.26
7.24
7.21
3.84
3.82
3.47
3.45
3.44
3.42
3.40
1.65
1.63
0.00
CN
CN
Br
65
1.99
3.09
1.00
1.10
3.03
f1 (ppm)
137.15
132.59
129.10
123.17
111.74
111.54
40.85
31.16
17.88
CN
CN
Br
65
f1 (ppm)

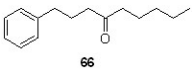

7.30
7.28
7.26
7.26
7.21
7.19
7.18
7.16
2.63
2.62
2.60
2.42
2.41
2.39
2.38
2.36
2.34
1.94
1.92
1.90
1.89
1.87
1.59
1.57
1.55
1.53
1.51
1.33
1.33
1.31
1.29
1.28
1.26
1.24
1.23
1.22
0.90
0.88
0.86
0.00


O

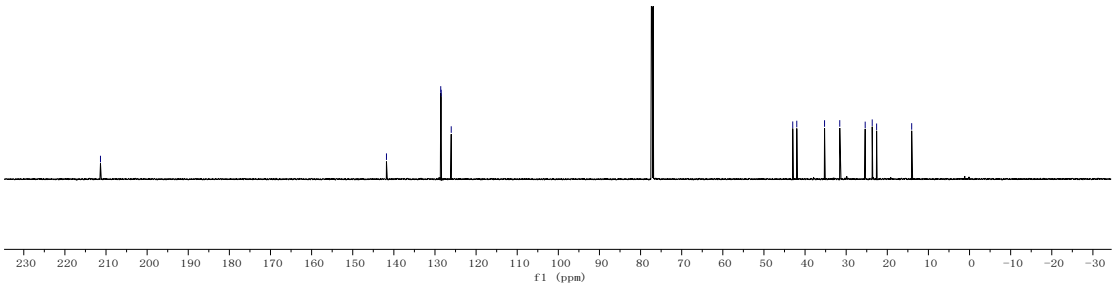
66

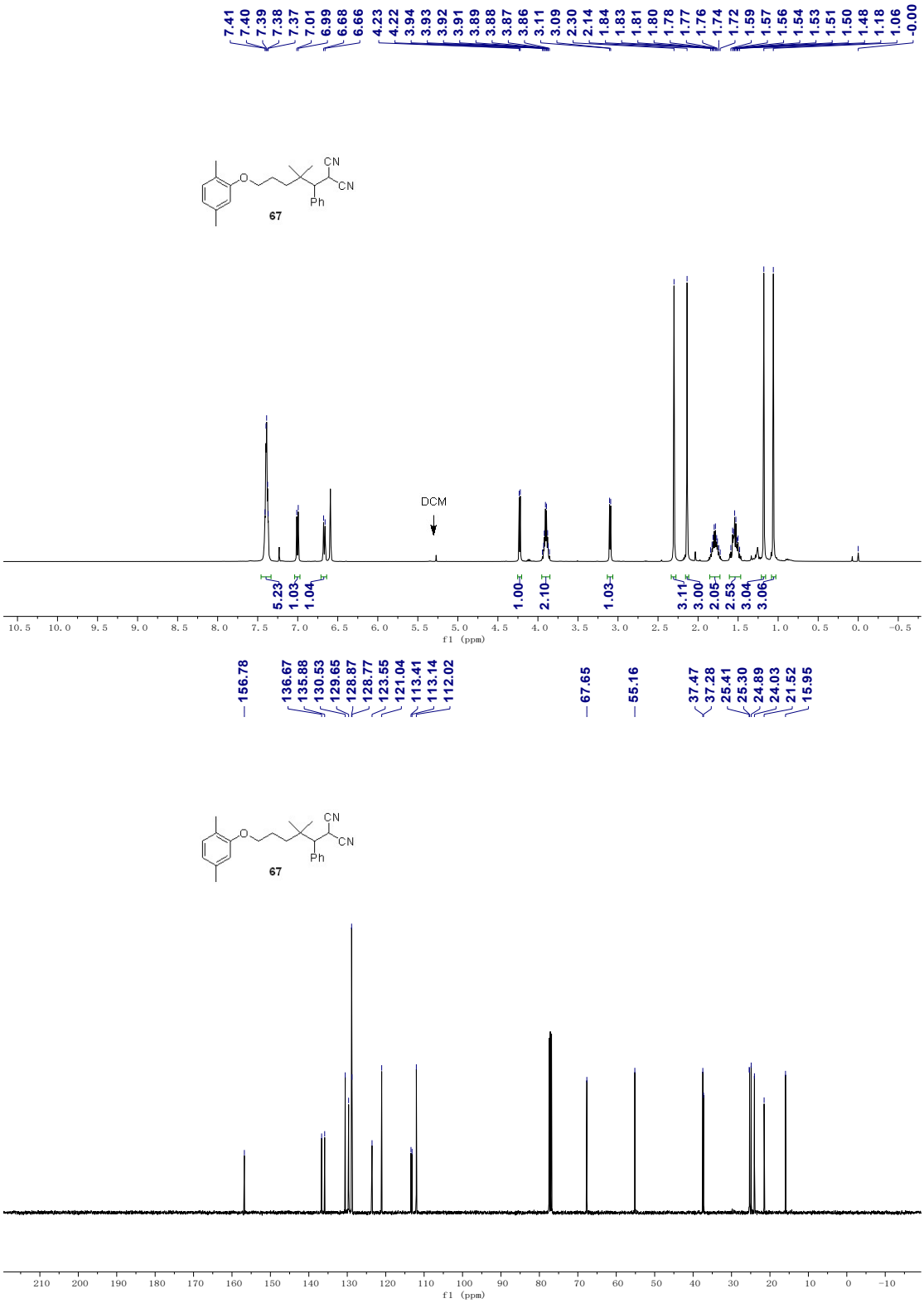

2.21
3.00
2.01
4.06
2.06
2.18
4.41
3.19
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

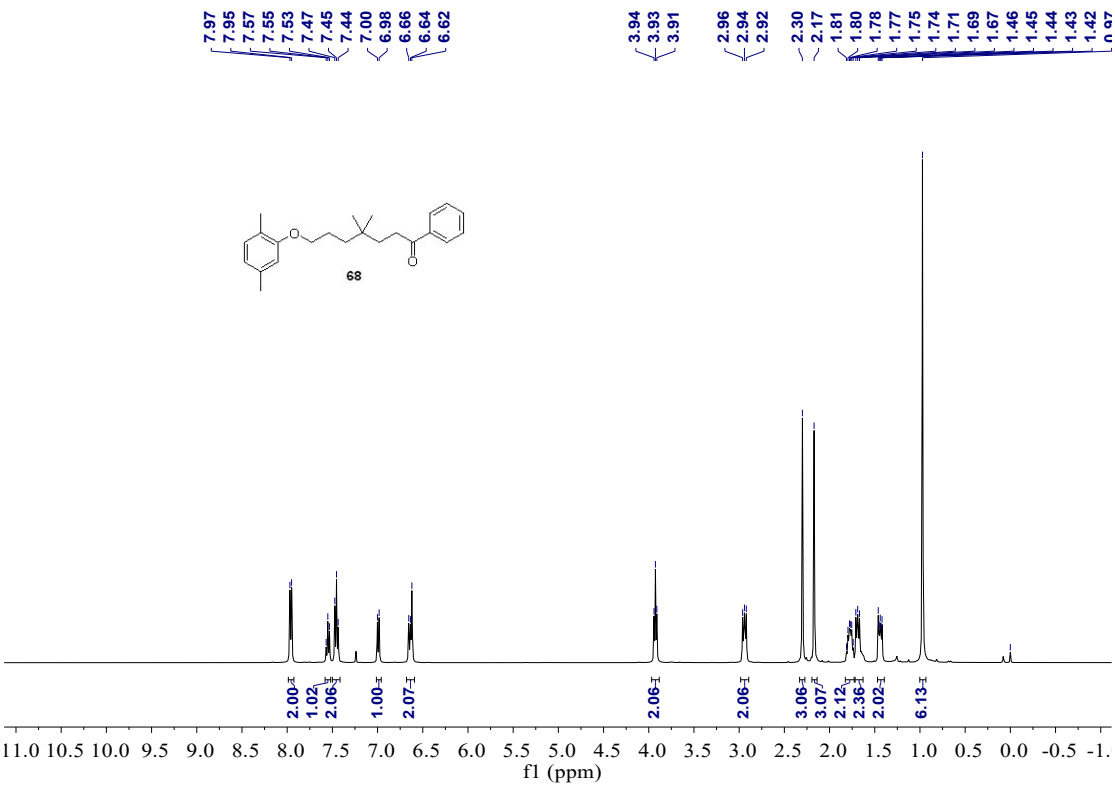

211.35
141.80
128.60
128.49
126.05
43.00
42.01
35.26
31.56
25.38
23.67
22.60
14.07

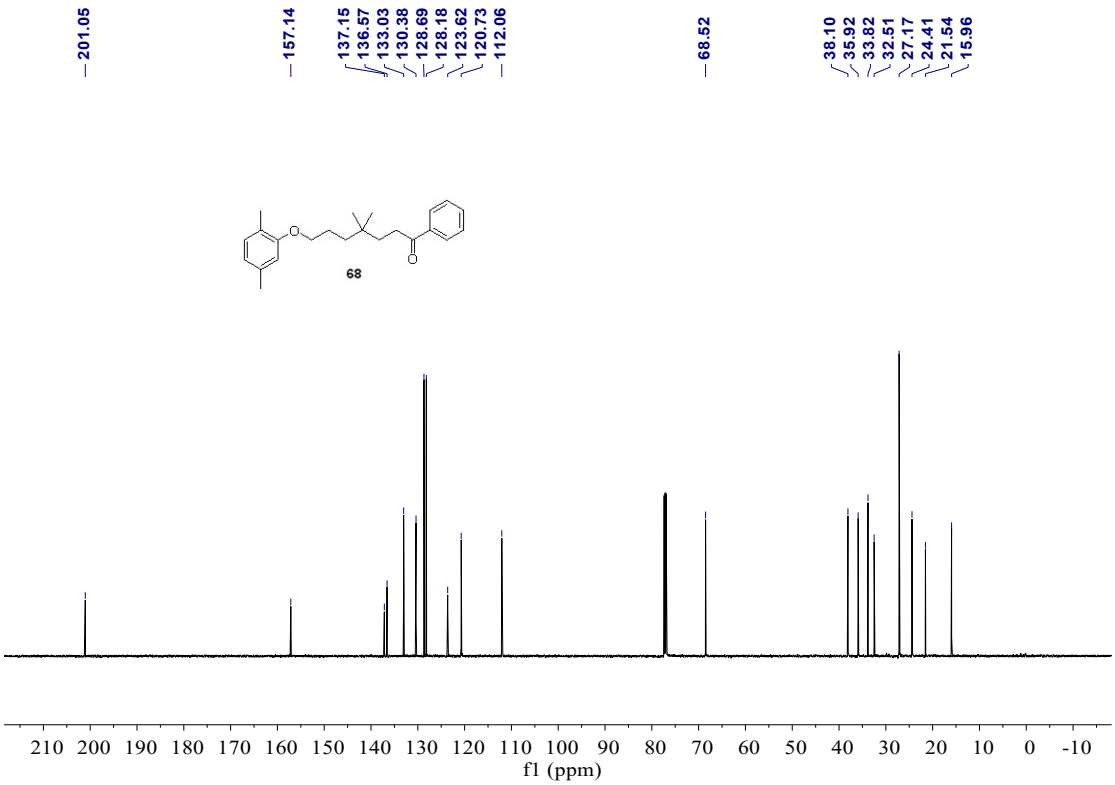

O

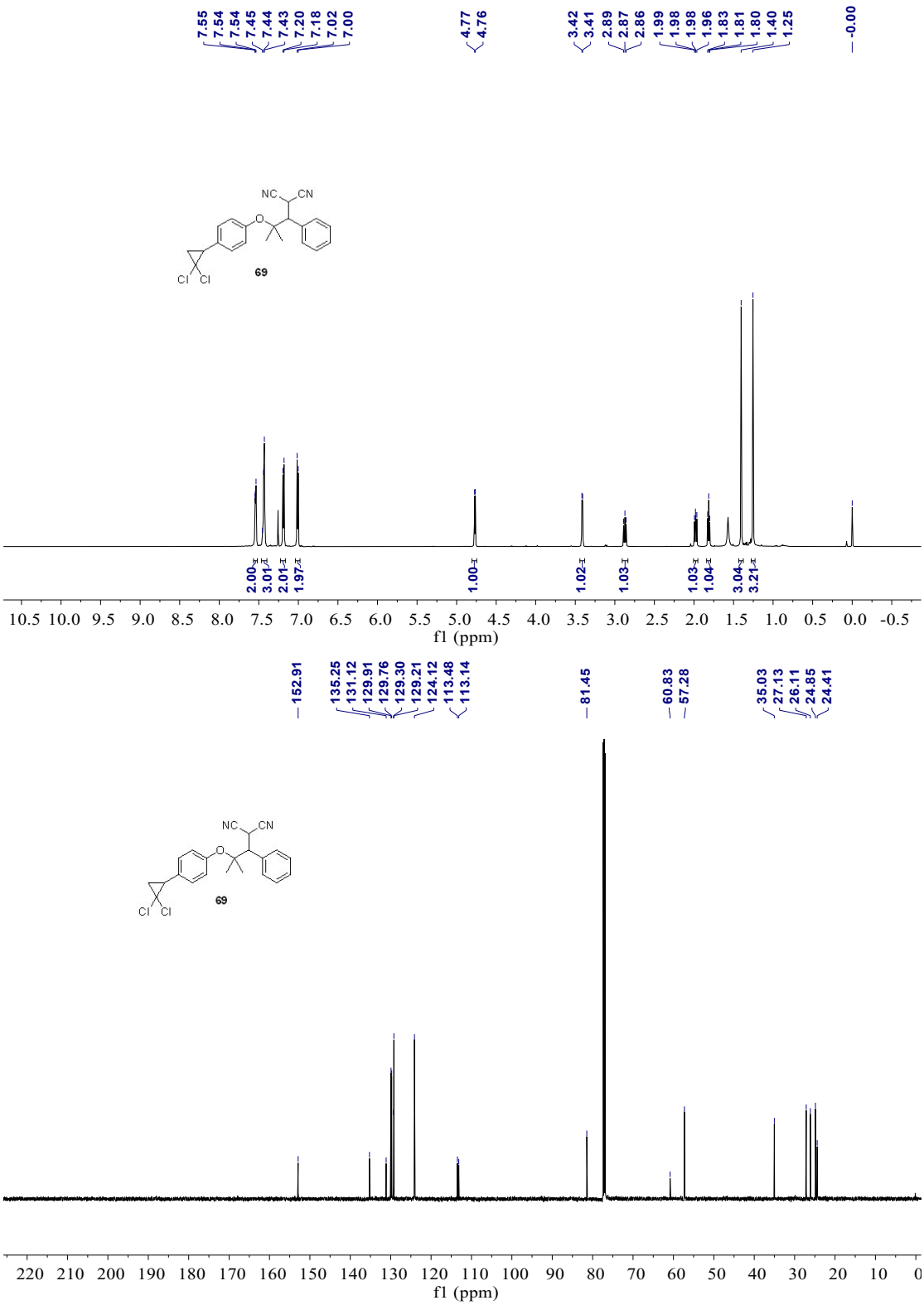
66

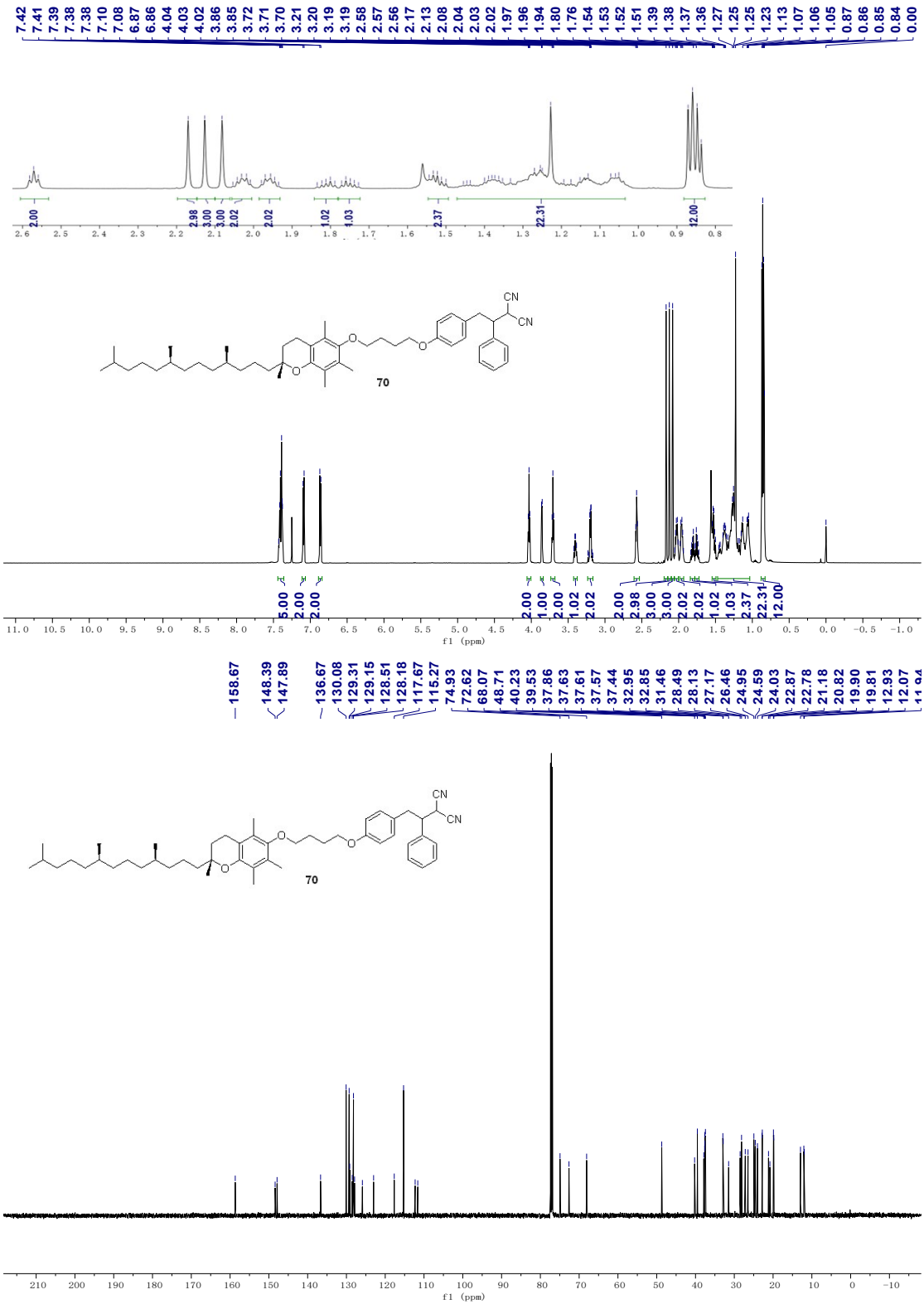

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30
f1 (ppm)

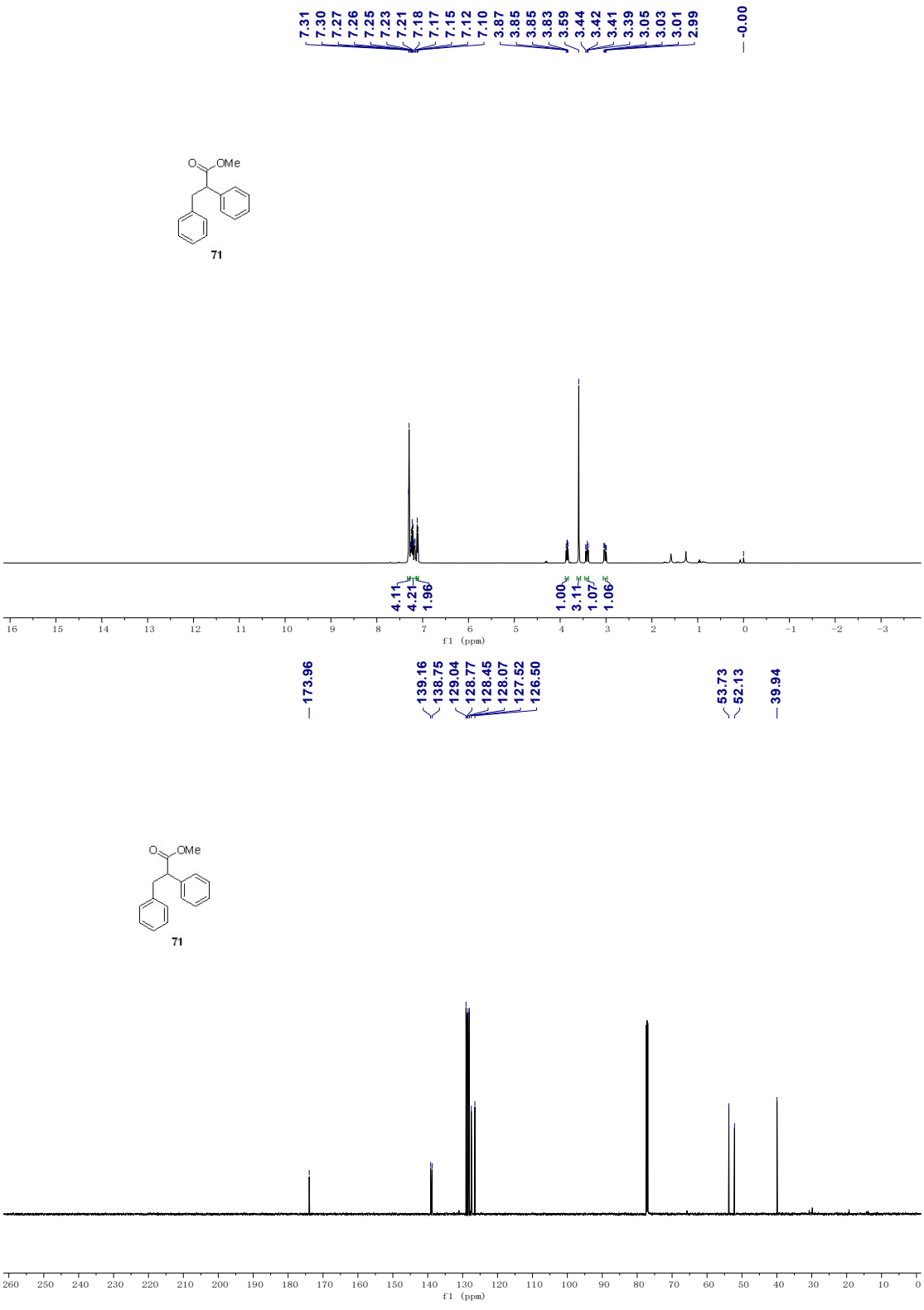

7.41 7.40 7.39 7.38 7.37 7.01 6.99 6.68 6.66 4.23 4.22 3.94 3.93 3.92 3.91 3.89 3.88 3.87 3.86 3.11 3.09 2.30 2.14 1.84 1.83 1.81 1.80 1.78 1.77 1.76 1.74 1.72 1.59 1.57 1.56 1.54 1.53 1.51 1.50 1.48 1.18 1.06 -0.00
CN
CN
Ph
O
67
DCM
5.23 1.03 1.04 1.00 2.10 1.03 3.11 3.00 2.05 2.53 3.04 3.06
f1 (ppm)
156.78 136.67 135.88 130.53 129.65 128.87 128.77 123.55 121.04 113.41 113.14 112.02 67.65 55.16 37.47 37.28 25.41 25.30 24.89 24.03 21.52 15.95
f1 (ppm)

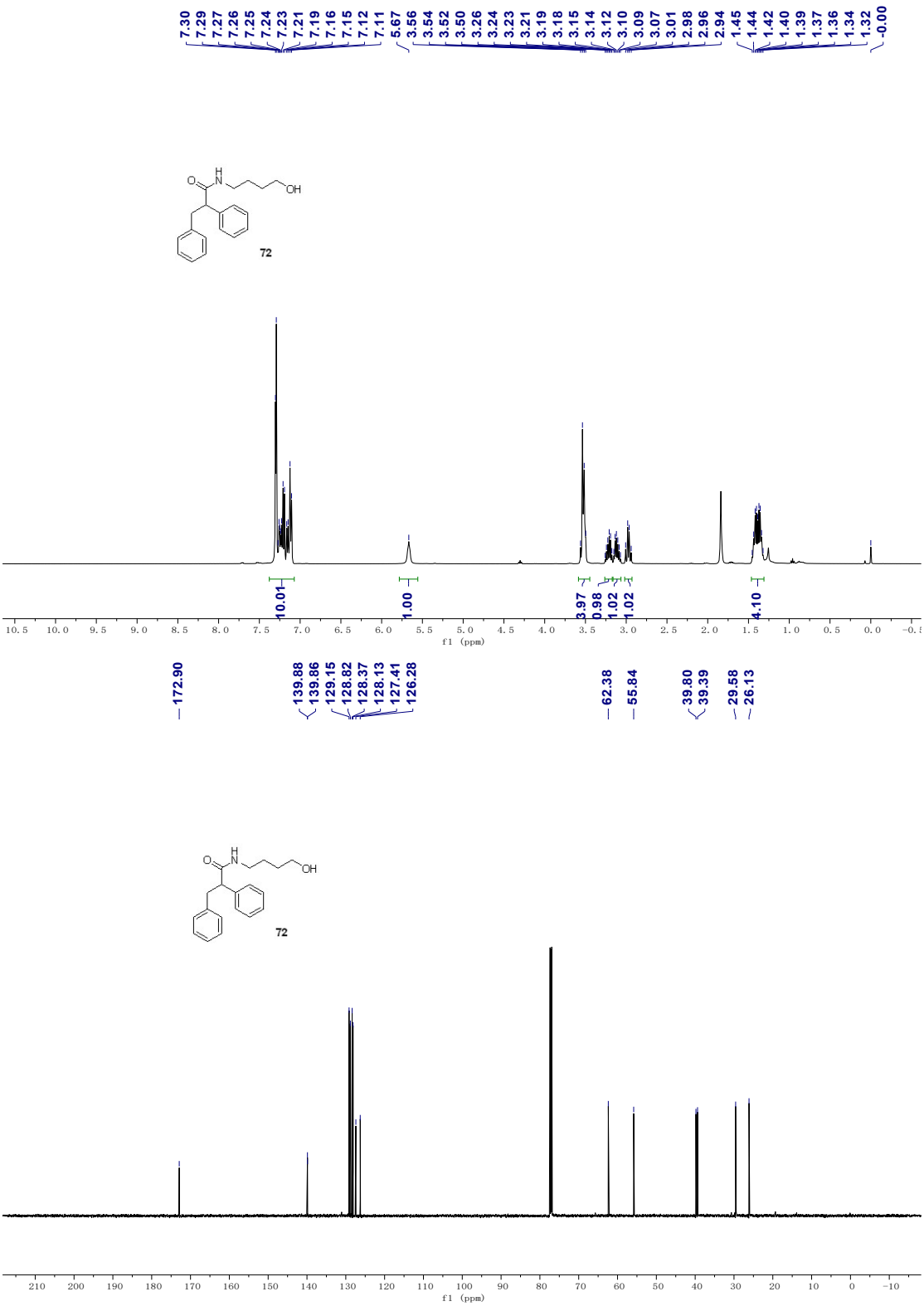

7.97
7.95
7.57
7.55
7.53
7.47
7.45
7.44
7.00
6.98
6.66
6.64
6.62
3.94
3.93
3.91
2.96
2.94
2.92
2.30
2.17
1.81
1.80
1.78
1.77
1.75
1.74
1.71
1.69
1.67
1.46
1.45
1.44
1.43
1.42
68
2.00
1.02
2.06
1.00
2.07
2.06
2.06
3.06
3.07
2.12
2.36
2.02
6.13
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.
f1 (ppm)


201.05
157.14
137.15
136.57
133.03
130.38
128.69
128.18
123.62
120.73
112.06
68.52
38.10
35.92
33.82
32.51
27.17
24.41
21.54
15.96
68
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.55
7.54
7.54
7.45
7.44
7.43
7.20
7.18
7.02
7.00
4.77
4.76
3.42
3.41
2.89
2.87
2.86
1.99
1.98
1.98
1.96
1.83
1.81
1.80
1.40
1.25
-0.00
NC
CN
O
Cl
Cl
69
2.00
3.01
2.01
1.97
1.00
1.02
1.03
1.03
1.04
3.04
3.21
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 -0.5
f1 (ppm)
152.91
135.25
131.12
129.91
129.76
129.30
129.21
124.12
113.48
113.14
81.45
60.83
57.28
35.03
27.13
26.11
24.85
24.41
NC
CN
O
Cl
Cl
69
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.42 7.41 7.39 7.38 7.38 7.10 7.08 6.87 6.86 4.04 4.03 4.02 3.86 3.85 3.72 3.71 3.70 3.21 3.20 3.19 3.19 2.58 2.57 2.56 2.17 2.13 2.08 2.04 2.03 2.02 1.97 1.96 1.94 1.80 1.76 1.54 1.53 1.52 1.51 1.39 1.38 1.37 1.36 1.27 1.25 1.25 1.23 1.13 1.07 1.06 1.05 0.87 0.86 0.85 0.84 0.00
2.00 2.98 3.00 3.00 2.02 2.02 1.02 1.03 2.37 22.31 12.00
CN
CN
O
O
70
5.00 2.00 2.00 2.00 1.00 2.00 1.02 2.02 2.00 2.98 3.00 3.00 2.02 2.02 1.02 1.03 2.37 22.31 12.00
f1 (ppm)
158.67 148.39 147.89 136.67 130.08 129.31 129.15 128.51 128.18 117.67 115.27 74.93 72.62 68.07 48.71 40.23 39.53 37.86 37.63 37.61 37.57 37.44 32.95 32.85 31.46 28.49 28.13 27.17 26.46 24.95 24.59 24.03 22.87 22.78 21.18 20.82 19.90 19.81 12.93 12.07
CN
CN
O
O
70
f1 (ppm)


7.31
7.30
7.27
7.26
7.25
7.23
7.21
7.18
7.17
7.15
7.12
7.10
3.87
3.85
3.85
3.83
3.59
3.44
3.42
3.41
3.39
3.05
3.03
3.01
2.99
-0.00
OMe
71
4.11
4.21
1.96
1.00
3.11
1.07
1.06
f1 (ppm)
173.96
139.16
138.75
129.04
128.77
128.45
128.07
127.52
126.50
53.73
52.13
39.94
OMe
71
f1 (ppm)


7.30
7.29
7.27
7.26
7.25
7.24
7.23
7.21
7.19
7.16
7.15
7.12
7.11
5.67
3.56
3.54
3.52
3.50
3.26
3.24
3.23
3.21
3.19
3.18
3.15
3.14
3.12
3.10
3.09
3.07
3.01
2.98
2.96
2.94
1.45
1.44
1.42
1.40
1.39
1.37
1.36
1.34
1.32
-0.00
72
10.01
1.00
3.97
0.98
1.02
1.02
4.10
f1 (ppm)
172.90
139.88
139.86
129.15
128.82
128.37
128.13
127.41
126.28
62.38
55.84
39.80
39.39
29.58
26.13
72
f1 (ppm)