Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting information

A Photoredox/Nickel Dual-Catalytic Strategy for Benzylic C-H Alkoxylation

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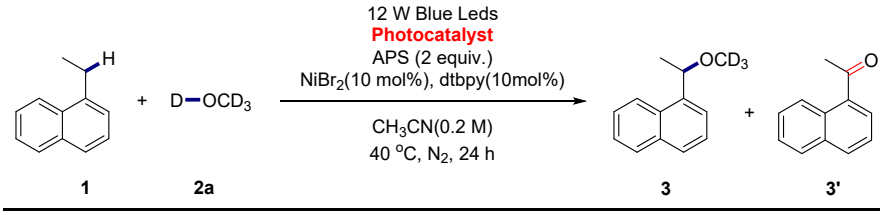
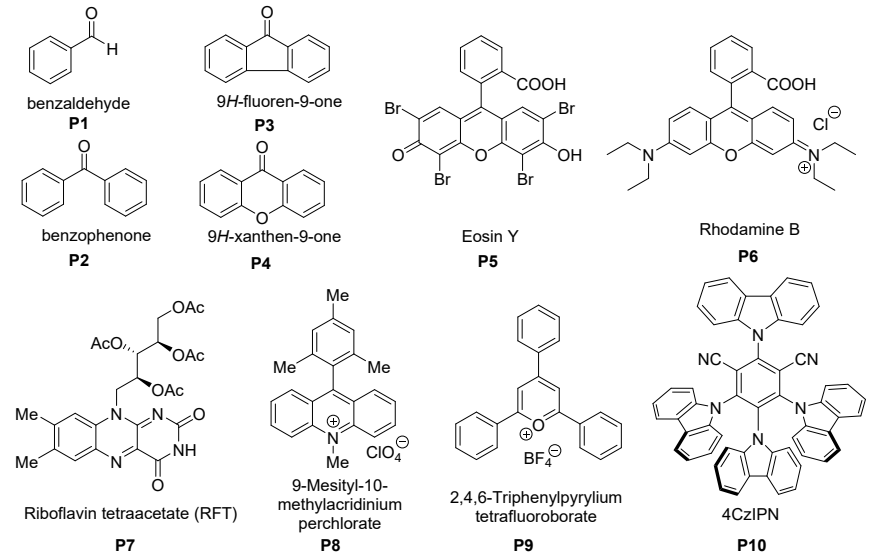
1. The Optimization of Photoredox/Nickel Dual-Catalyzed Benzylic C-H Alkoxylation.

Table S1. The optimization of photocatalyst. [a]



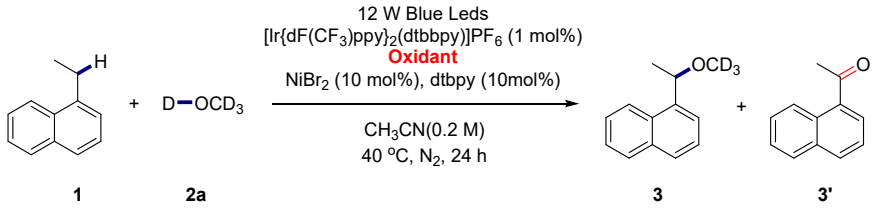


Entry	Photocatalyst	yield(3) [b]	yield(3') [c]
1	**P1** (100 mol%)	16%	-
2	**P2** (20 mol%)	22%	4%
3	**P3** (20 mol%)	14%	3%
4	**P4** (20 mol%)	trace	-
5	**P5** (5 mol%)	trace	-
6	**P6** (5 mol%)	30%	5%
7	**P7** (5 mol%)	16%	-
8	**P8** (5 mol%)	32%	5%
9	**P9** (5 mol%)	trace	-
10	**P10** (5 mol%)	22%	5%
11	**P11** (1 mol%)	trace	-
12	**P12** (1 mol%)	trace	-
13	**P13** (1 mol%)	43%	6%
14	**P14** (1 mol%)	32%	4%
15	**P13** (0.5 mol%)	33%	5%

16	**P13** (2 mol%)	43%	8%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), photocatalyst (1-100 mol%), APS (1.0 mmol), $NiBr_2$ (10 %mmol), dtbpy (10 %mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] Yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as an internal standard.

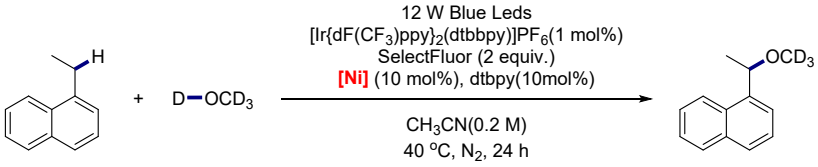
Table S2. The optimization of the oxidant. [a]



Entry	Oxidants	yield(3) [b]	yield(3') [c]
1	APS(2.0 equiv.)	43%	6%
2	$Na_2S_2O_8$(2.0 equiv.)	n.d.[d]	9%
3	$K_2S_2O_8$(2.0 equiv.)	n.d.[d]	7%
4	Selectfluor(2.0 equiv.)	54%	-
5	NFSI(2.0 equiv.)	46%	5%
6	$Et_3N \cdot 3HF$(2.0 equiv.)	n.d.[d]	-
7	NBS(2.0 equiv.)	trace	7%
8	NCS(2.0 equiv.)	35%	6%
9	PIDA(2.0 equiv.)	trace	6%
10	PIFA(2.0 equiv.)	20%	7%
11	mCPBA(2.0 equiv.)	n.d.[d]	15%
12	Oxane(2.0 equiv.)	n.d.[d]	20%
13 [e]	Selectfluor(1.0 equiv.)	24%	-
14 [f]	Selectfluor(1.5 equiv.)	44%	-
15 [g]	Selectfluor(2.5 equiv.)	46%	-
15 [h]	Selectfluor(3.0 equiv.)	45%	-

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), [Ir{dF(CF3)ppy}2(dtbbpy)]PF_6 (1 mol%), $NiBr_2$ (10 %mmol), dtbpy (10 %mmol), oxidant (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] Yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as an internal standard; [d] not detected; [e] 0.5 mmol Selectfluor was used; [f] 0.75 mmol Selectfluor was used; [g] 1.25 mmol Selectfluor was used; [h] 1.5 mmol Selectfluor was used.

Table S3. The optimization of the nickel salts. [a]

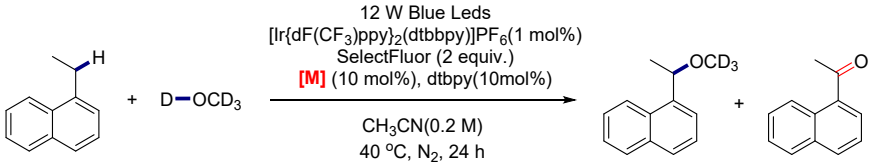


Entry	[Ni]	yield(3) [b]
1	$NiBr_2$	54%

2	$NiBr_2$ • dme	40%
3	$NiCl_2$ • dme	31%
4	$Ni(OAc)_2$ • $4H_2O$	32%
5	$Ni(CF_3SO_3)_2$	11%
6	$Ni(acac)_2$	60%
7	$Ni(cod)_2$	40%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), [Ir{dF(CF$_3$)ppy}$_2$(dtbbpy)]PF$_6$ (1 mol%), [Ni] (10 %mmol), dtbpy (10 %mmol), Selectfluor (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported.

Table S4. The optimization of other metal salts. [a]

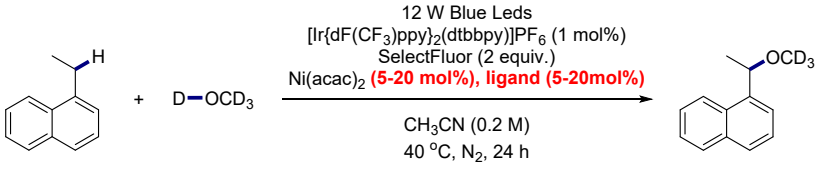


1 **2a** **3** **3'**

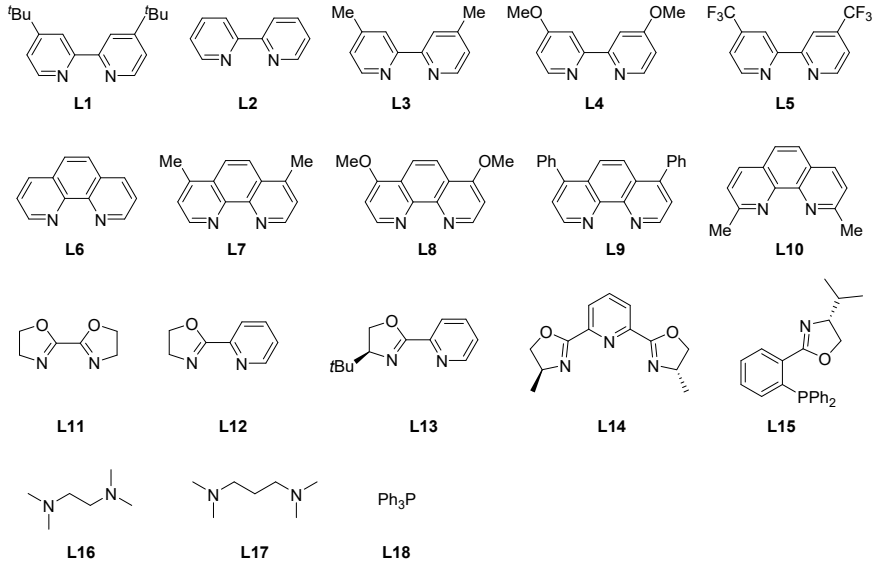
Entry	[M]	yield(3) [b]	yield(3') [c]
1	$Ni(acac)_2$	60%	-
2	$CuBr_2$	46%	4%
3	$Cu(acac)_2$	40%	3%
4	CuCl	51%	4%
5	$FeCl_3$ • $4H_2O$	n.d.[d]	5%
6	$CoCl_2$	n.d.[d]	-
7	$MnCl_2$	n.d.[d]	7%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), [Ir{dF(CF$_3$)ppy}$_2$(dtbbpy)]PF$_6$ (1 mol%), [M] (10 %mmol), dtbpy (10 %mmol), Selectfluor (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] Yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard; [d] not detected.

Table S5. The optimization of the ligand. [a]



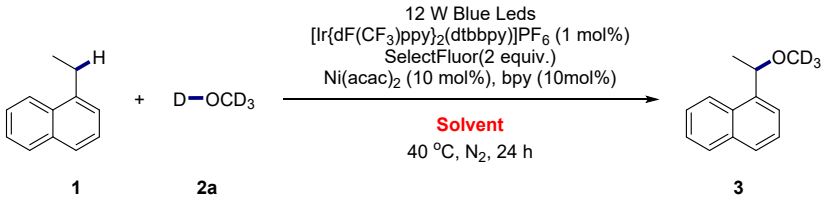
1 **2a** **3**



Entry	Ligands	yield(3) [b]
1	**L1**(10 mol%)	60%
2	**L2**(10 mol%)	81%
3	**L3**(10 mol%)	73%
4	**L4**(10 mol%)	34%
5	**L5**(10 mol%)	40%
6	**L6**(10 mol%)	67%
7	**L7**(10 mol%)	56%
8	**L8**(10 mol%)	28%
9	**L9**(10 mol%)	trace
10	**L10**(10 mol%)	trace
11	**L11**(10 mol%)	67%
12	**L12**(10 mol%)	33%
13	**L13**(10 mol%)	21%
14	**L14**(10 mol%)	21%
15	**L15**(10 mol%)	71%
16	**L16**(10 mol%)	40%
17	**L17**(10 mol%)	27%
18	**L18**(10 mol%)	54%
19 [c]	**L2**(5 mol%)	69%
20 [d]	**L2**(20 mol%)	75%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), [Ir{dF(CF_3)ppy}$_2$(dtbbpy)]PF_6 (1 mol%), Ni(acac)$_2$ (10 %mmol), ligand (10 %mmol), Selectfluor (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] 0.025 mmol Ni(acac)$_2$ and 0.025 mmol bpy was used; [d] 0.1 mmol Ni(acac)$_2$ and 0.1 mmol bpy was used.

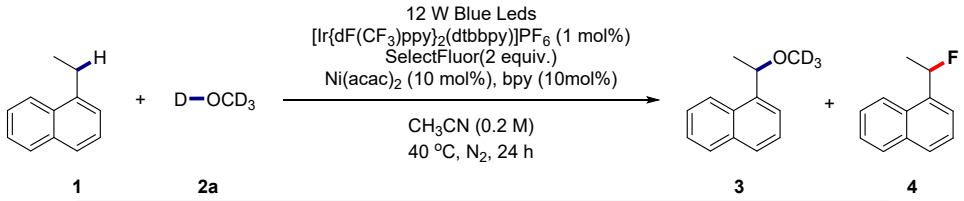
Table S6. The optimization of the solvent. [a]



Entry	Solvents	yield(3) [b]
1	CH_3CN(0.2 M)	81%
2	DCE(0.2 M)	n.d.[c]
3	DCM(0.2 M)	27%
4	DMF(0.2 M)	n.d.[c]
5	DMA(0.2 M)	trace
6	DMSO(0.2 M)	n.d.[c]
7	EtOAc(0.2 M)	n.d.[c]
8	HFIP(0.2 M)	n.d.[c]
9	DCM:HFIP=4:1(0.2 M)	n.d [c]
10 [d]	CH_3CN(0.1 M)	61%
11 [e]	CH_3CN(0.4 M)	71%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), [Ir{dF(CF_3)ppy}$_2$(dtbbpy)]PF_6 (1 mol%), $Ni(acac)_2$ (10 %mmol), bpy (10 %mmol), Selectfluor (1.0 mmol), solvent (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] not detected; [d] 5 ml CH_3CN was used; [e] 1.25 mmol CH_3CN was used.

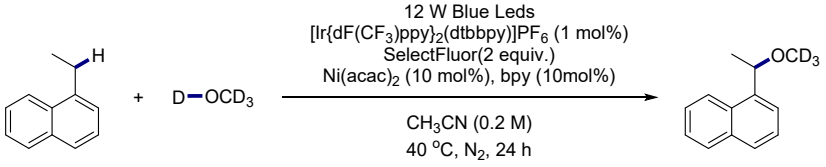
Table S7. The optimization of the amount of d^4-MeOH. [a]



Entry	d^4-MeOH	yield(3) [b]	yield(4) [c]
1	1 equiv.	40%	7%
2	2 equiv.	45%	5%
3	3 equiv.	70%	2%
4	4 equiv.	76%	-
5	5 equiv.	81%	-
6	8 equiv.	78%	-
7	10 equiv.	75%	-

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (0.5-5 mmol), [Ir{dF(CF_3)ppy}$_2$(dtbbpy)]PF_6 (1 mol%), $Ni(acac)_2$ (10 %mmol), bpy (10 %mmol), Selectfluor (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] Yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as an internal standard.

Table S8. Control Experiments. [a]



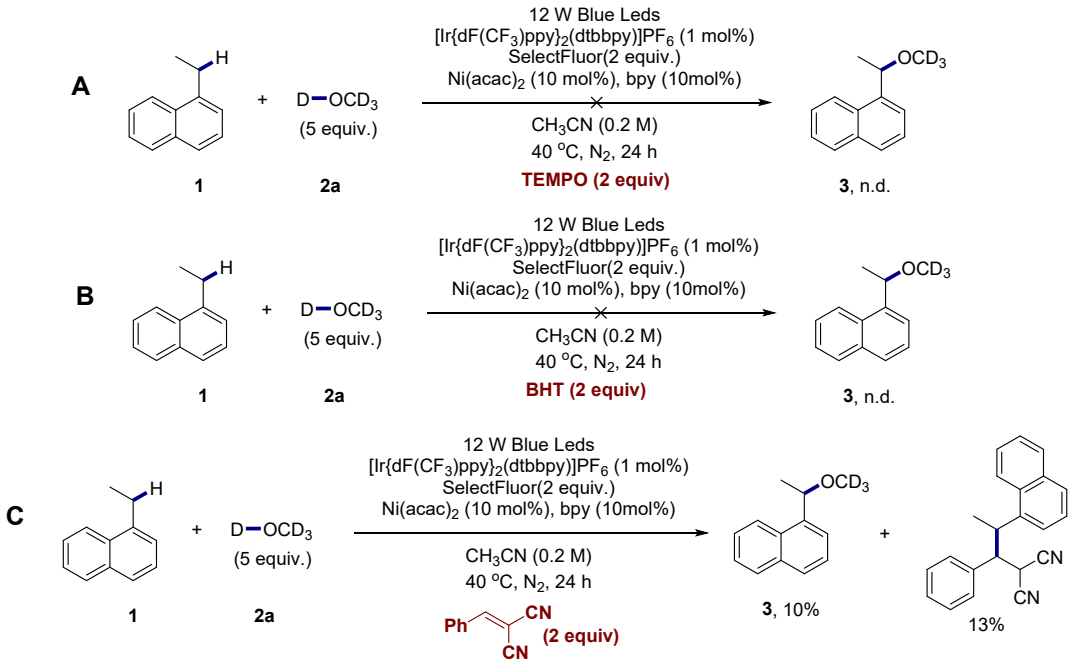
1 **2a** **3**

Entry	Conditions	yield(3) [b]
1	Standard	81%
2	No photocatalyst	14%
3	In darkness	n.d.[c]
4	No Selectfluor	n.d.[c]
5	No $Ni(acac)_2$	n.d.[c]
6	No bpy	67%
7	Under air	30%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** (2.5 mmol), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (1 mol%), $Ni(acac)_2$ (10 %mmol), bpy (10 %mmol), Selectfluor (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, 24 h, unless otherwise noted; [b] Isolated yields were reported; [c] not detected.

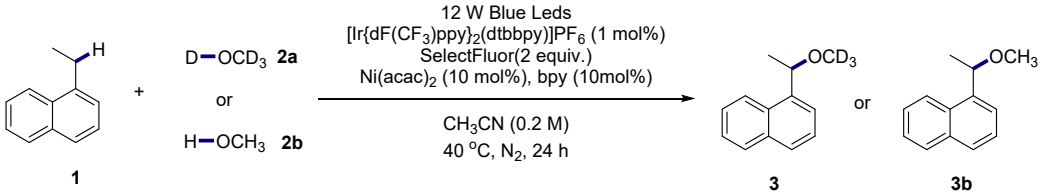
2. The Study of the Mechanism of Photoredox/Nickel Dual-Catalyzed Benzylic C-H Alkoxylation

Scheme S1. The radical quenching and trapping experiments.



Notes: The reaction was completely inhibited by the presence of TEMPO and BHT, further suggesting the radical based mechanism (Scheme S1A, S1B). However, only the use of 2-benzylidenemalononitrile as additive is able to produce the radial trapping product accompanied with the formation of little target product **3** (Scheme S1C), the isolated yield of radical adduct product to benzylidenemalononitrile is 13%, and the radical adduct product is evaluated by ^{1}H-NMR and ^{13}C-NMR.

Scheme S2. The preliminary kinetic study of alkoxylation with MeOH and d^4-MeOH [a]



Entry	Time (h)	Yield of 3 (%) [b, c]	Yield of 3b (%) [b, d]
1	2	36%	42%
2	4	44%	49%
3	8	56%	61%

4	16	72%	76%
5	20	75%	79%
6	24	81%	82%

[a] Conditions employed 12 W Blue Leds, **1** (0.5 mmol), **2a** or **2b** (2.5 mmol), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (1 mol%), $Ni(acac)_2$ (10 %mmol), bpy (10 %mmol), Selectfluor (1.0 mmol), CH_3CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2, 40 °C, unless otherwise noted; [b] Yields were determined by ^1H NMR using 1,3,5-trimethoxybenzene as an internal standard; [c] performed with d^4-MeOH (2.5 mmol); [d] performed with MeOH (2.5 mmol).

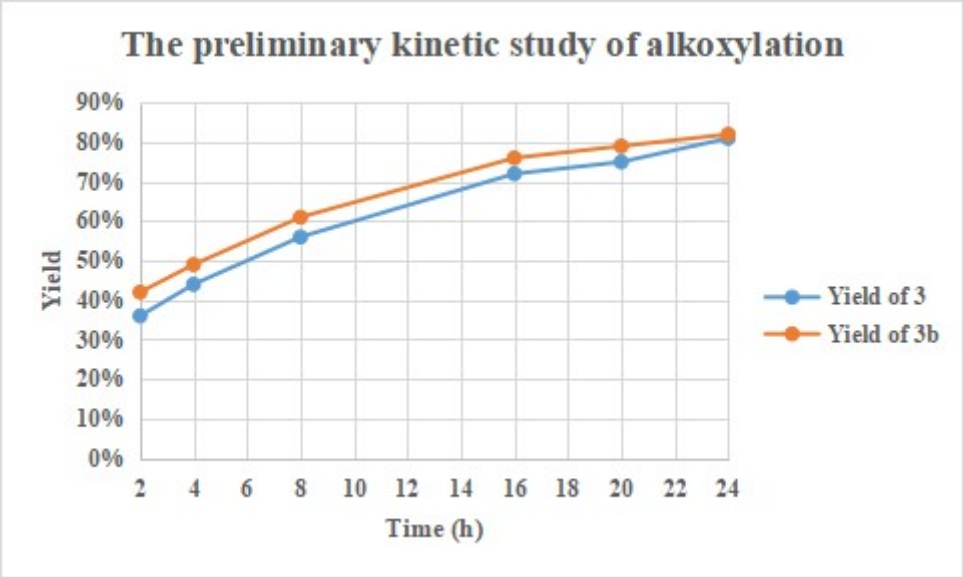
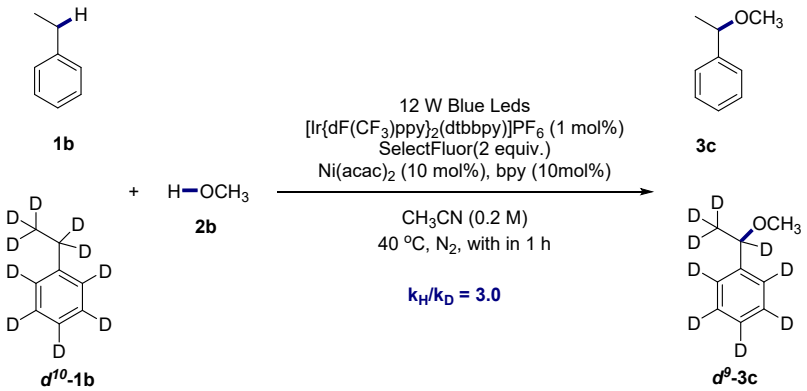
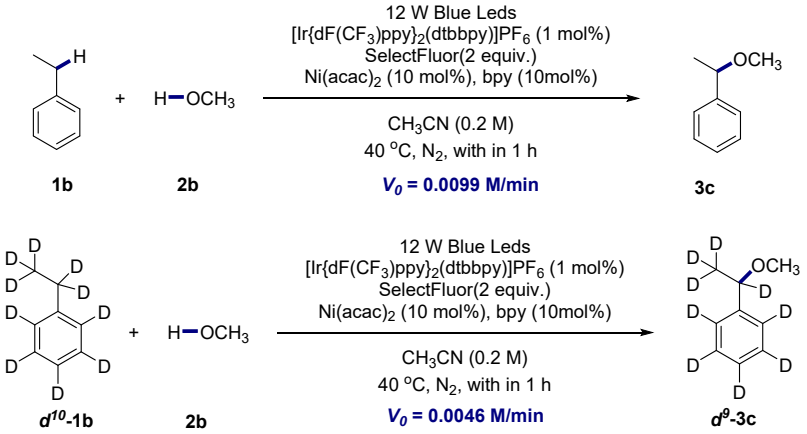


Fig. S1 The preliminary kinetic study of alkoxylation with MeOH and d^4-MeOH

Scheme S3. The kinetic isotopic effect determination from intermolecular competition reactions employing ethylbenzene and d^{10}-ethylbenzene as coupling partner [a]



Scheme S4. The kinetic isotopic effect determination from two parallel reactions employing ethylbenzene and d^{10}-ethylbenzene as coupling partner [a]



Entry	Time(min)	NMR Yield (%) [b]	[Product](M)	V_0
ethylbenzene	10	5	0.01	0.0099M/min
	20	11	0.022	
	30	15	0.03	
	40	21	0.042	
	50	25	0.05	
	60	30	0.06	

Entry	Time(min)	NMR Yield (%)	[Product](M)	V_0
d^{10}-ethylbenzene	10	2	0.004	0.0046M/min
	20	5	0.01	
	30	7	0.014	
	40	9	0.018	
	50	11	0.022	
	60	14	0.028	

[a] Conditions employed 12 W Blue Leds, ethylbenzene (or d^{10}-ethylbenzene) (0.5 mmol), **2b** (2.5 mmol), [Ir{dF(CF$_3$)ppy}$_2$(dtbbpy)]PF$_6$ (1 mol%), Ni(acac)$_2$ (10 %mmol), bpy (10 %mmol), Selectfluor (1.0 mmol), CH$_3$CN (2.5 mL), the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N$_2$, 40 °C; [b] Analyzed by NMR spectroscopy for the formation of product with 1,3,5-trimethoxybenzene as an internal standard.

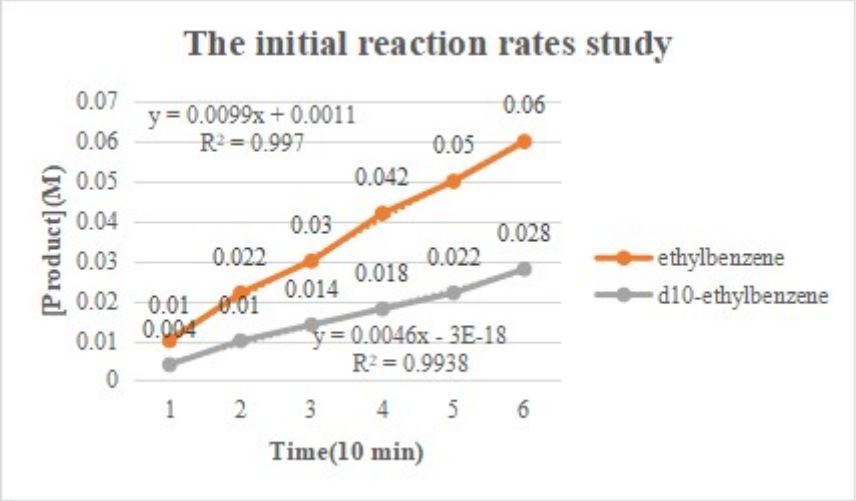
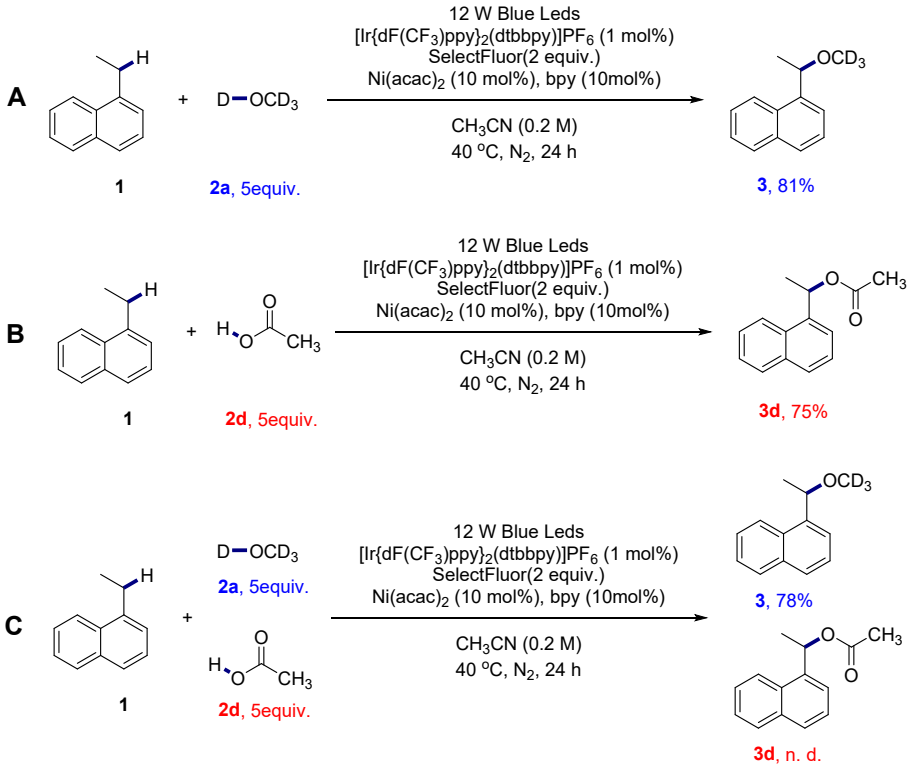


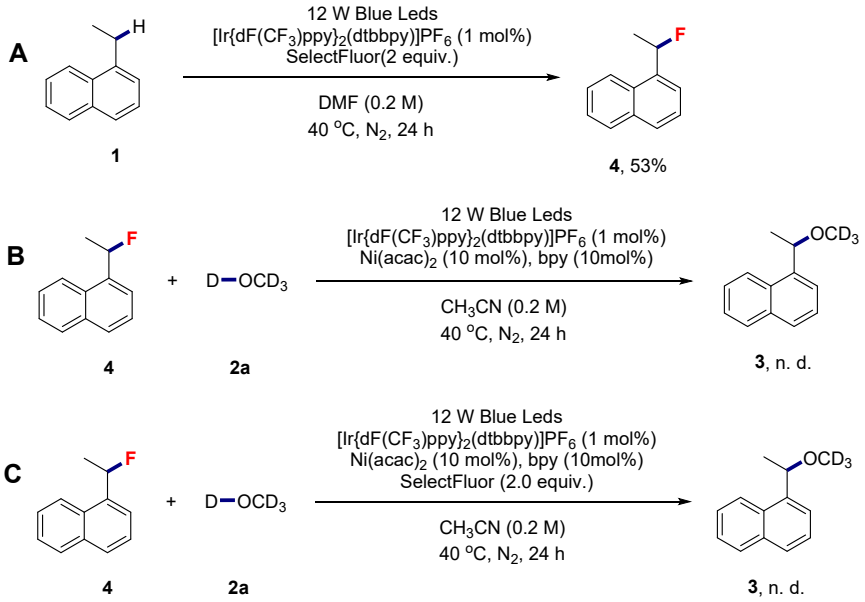
Fig. S2 The initial rate study of ethylbenzene and d^{10}-ethylbenzene based coupling reaction with MeOH

Scheme S5. Competition experiments in 1-methylnaphthalene etherification and esterification



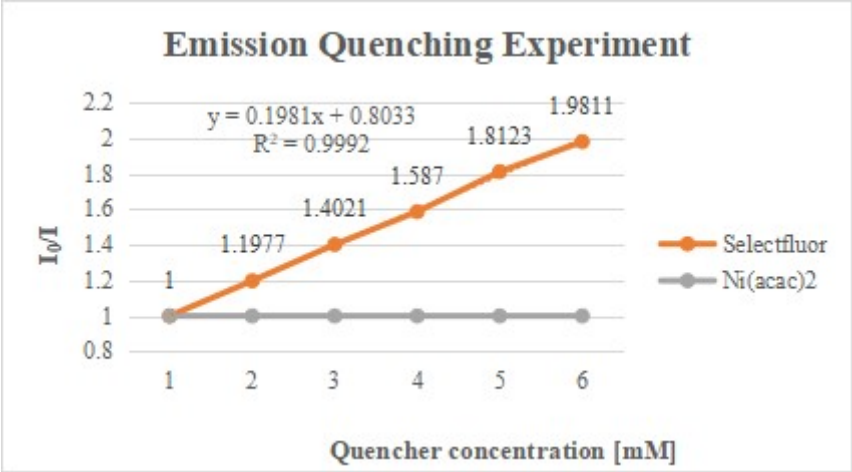
Notes： The competition experiments of d^4-MeOH **2a** and CH_3COOH **2d** with 1-methylnaphthalene. This result further indicates that dialkoxylated Ni(II) species is more likely to intercept benzyl radical than diacetyloxyNi(II) species.

Scheme S6. Competition experiments in 1-methylnaphthalene etherification and fluorination



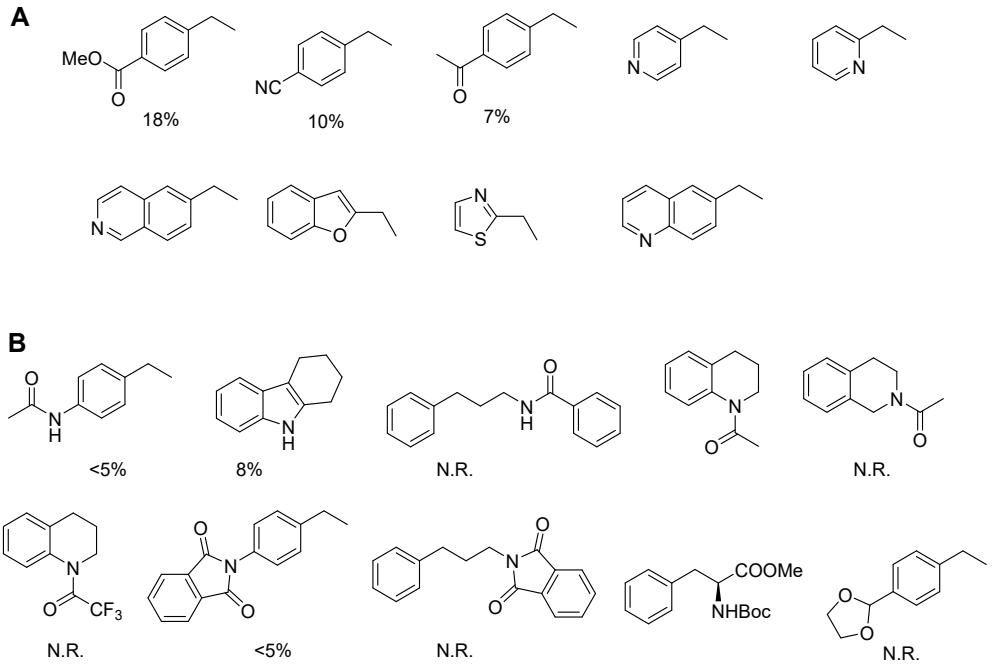
Notes： This result indicates that benzyl radical is more likely to be intercepted by dialkoxylated Ni(II) species than Selectfluor, and fluorination product **4** is not the intermediate of benzylic C-H alkoxylation.

Scheme S7. Emission Quenching Experiment



Emission intensities were recorded using a FluoroMax-4 (Horiba Scientific) fluorescence spectrophotometer. All $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ solutions were excited at 380 nm and the emission intensity was collected at 470 nm. In a typical experiment, to a 1×10^{-6} M solution of $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ in CH_3CN was added the appropriate amount of a quencher in a screw-top quartz cuvette.

Scheme S8. Unsuccessful substrates in benzylic C-H alkoxylation



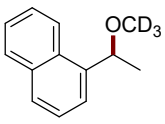
Notes： In our research, significant amount of fluorination products were observed with electron deficient aromatic substrates(Scheme S8A). Based on previous research, benzylic C-H derivatives with pyridine, quinoline, acetyl and ester are more likely to react directly with Selecfluor rather than alkoxylation.[1-4] Substrates with N-H groups, amide, imide and acetal suffered from low conversion, possibly reflecting inhibition by quenching (after hydrolysis) of reactive radicals(Scheme S8B).

3. Experiment Procedures and Product Characterization

Commercial reagents and solvents were used as received, unless otherwise stated. Organic solution was concentrated under reduced pressure on a IKA rotary evaporator using an alcohol bath. Analytical thin layer chromatography (TLC) was performed on 0.25 mm silica gel plates (Qingdao Haiyang Chemical China), and the compounds were visualized with a UV light at 254 nm. Further visualization was achieved by staining with $KMnO_4$. Flash chromatography was performed on silica gel 200-300 mesh (purchased from Qingdao Haiyang Chemical China) with commercial solvents (purchased from Energy Chemical® and Adamas-beta®). The ^1H and ^{13}C NMR spectra were recorded on a Bruker AVANCE III 400 Spectrometer (400 and 100 MHz for ^1H and ^{13}C NMR, respectively), Bruker AVANCE III 500 Spectrometer (500 and 125 MHz for ^1H and ^{13}C NMR, respectively), Bruker Ascend 600 Spectrometer (600 and 150 MHz for ^1H and ^{13}C NMR, respectively) and are internally referenced to residual solvent signals (note: $CDCl_3$ referenced at 7.26 and 77.00 ppm in ^1H and ^{13}C NMR, respectively). Multiplicities were given as s (singlet), d (doublet), t (triplet), dd (double of doublet), quin (quintet) and m (multiplets). Coupling constants were reported in Hertz (Hz). Data for ^{13}C NMR are reported in terms of chemical shift. High-resolution mass spectrometry (HRMS) was recorded on Waters LCT Premier XE spectrometer.

General Procedure :

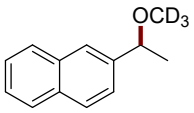
To a 5 mL vial equipped with a Teflon septum and magnetic stir bar were added the corresponding benzylic substrate (if solid, 0.5 mmol, 1.0 equiv.), the corresponding alcohol (if solid, 2.5 mmol, 5.0 equiv.), Selectfluor (1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (0.05 mmol, 10mol%), bpy (0.05 mmol, 10mol%), and $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (0.005 mmol, 1 mol%). The vial was sealed and placed under N_2 atmosphere, then CH_3CN (2.5 mL, 0.2 M), benzylic substrate (if liquid, 0.5 mmol, 1.0 equiv.), alcohol (if liquid, 2.5 mmol, 5.0 equiv.) were added into the vial via injection through the cap, the reaction mixture was degassed via freeze pump thaw (× 3 times) and refilled with N_2. The sealed vial was placed in 12 W Blue Leds and irradiated for 24 hours. When the reaction finished, the reaction mixture was diluted with saturated $NaHCO_3$ aqueous solution, extracted with ethyl acetate (3 × 20 mL), the combined organic extracts were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo. Purification of the crude product by flash chromatography on silica gel using the indicated solvent system afforded the desired product.



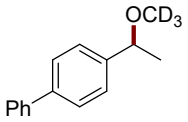
1-(1-(*d*^{3}-methoxy)ethyl)naphthalene (3):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (77 mg, 81 % yield).

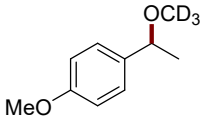
^{1}H NMR (500 MHz, $CDCl_3$) δ 8.18 (d, *J* = 8.1 Hz, 1H), 7.89 (d, *J* = 7.6 Hz, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.57 (d, *J* = 7.0 Hz, 1H), 7.54 - 7.44 (m, 3H), 5.07 (q, *J* = 6.5 Hz, 1H), 1.62 (d, *J* = 6.6 Hz, 3H).
^{13}C NMR (150 MHz, $CDCl_3$) δ 139.1, 133.9, 130.8, 128.9, 127.78, 125.8, 125.5, 125.4, 123.3, 123.2, 77.2, 55.7(quin, J = 21.0 Hz), 23.2.
HRMS (ESI) Calcd. for $C_{13}H_{12}D_3O$ [$(M+H)^+$] 190.1311, found 190.1315.



2-(1-(*d*^{3}-methoxy)ethyl)naphthalene (5):
According to the general procedure, 2-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (76 mg, 80 % yield).
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 - 7.81 (m, 3H), 7.74 (s, 1H), 7.51 - 7.43 (m, 3H), 4.47 (q, *J* = 6.5 Hz, 1H), 1.52 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (150 MHz, $CDCl_3$) δ 140.9, 133.3, 133.0, 128.4, 127.8, 127.7, 126.1, 125.7, 125.1, 124.1, 79.7, 23.9.
HRMS (ESI) Calcd. for $C_{13}H_{12}D_3O$ [$(M+H)^+$] 190.1311, found 190.1317.



4-(1-(*d*^{3}-methoxy)ethyl)-1,1'-biphenyl (6):
According to the general procedure, 4-ethyl-1,1'-biphenyl (91 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (88 mg, 82 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 - 7.46 (m, 4H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.32 - 7.22 (m, 3H), 4.26 (q, *J* = 6.5 Hz, 1H), 1.39 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (150 MHz, $CDCl_3$) δ 142.6, 140.9, 140.4, 128.7, 127.2, 127.0, 126.6, 79.2, 55.6 (quin, *J* = 21.5 Hz), 23.8.
HRMS (ESI) Calcd. for $C_{15}H_{14}D_3O$ [$(M+H)^+$] 216.1468, found 216.1473.



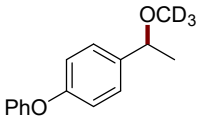
1-methoxy-4-(1-(*d*^{3}-methoxy)ethyl)benzene (7):

According to the general procedure, 1-ethyl-4-methoxybenzene (68 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (63 mg, 75 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 8.4 Hz, 2H), 6.82 (d, *J* = 8.4 Hz, 2H), 4.18 (q, *J* = 6.4 Hz, 1H), 3.74 (s, 3H), 1.35 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 159.0, 135.6, 127.4, 113.8, 79.0, 55.3, 23.8.

HRMS (ESI) Calcd. for $C_{10}H_{12}D_3O_2$ $[(M+H)^+]$ 170.1260, found 170.1265.



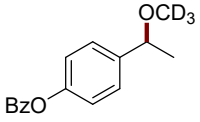
1-(1-(*d*^{3}-methoxy)ethyl)-4-phenoxybenzene (8):

According to the general procedure, 1-ethyl-4-phenoxybenzene (99 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (5 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (90 mg, 78 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 7.9 Hz, 2H), 7.22 - 7.18 (m, 2H), 7.02 (t, *J* = 7.4 Hz, 1H), 6.93 (dd, *J* = 11.5, 8.3 Hz, 4H), 4.21 (q, *J* = 6.4 Hz, 1H), 1.36 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 156.6, 138.3, 129.7, 127.6, 123.2, 118.9, 118.7, 79.0, 23.7.

HRMS (ESI) Calcd. for $C_{15}H_{14}D_3O_2$ $[(M+H)^+]$ 232.1432, found 232.1434.



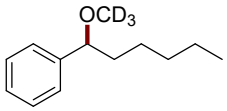
4-(1-(*d*^{3}-methoxy)ethyl)phenyl benzoate (9):

According to the general procedure, 4-ethylphenyl benzoate [5] (113 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (100 mg, 77 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.1 Hz, 2H), 7.61 - 7.52 (m, 1H), 7.43 (dd, *J* = 11.2, 4.1 Hz, 2H), 7.34 - 7.25 (m, 2H), 7.16 - 7.06 (m, 2H), 4.25 (q, *J* = 6.4 Hz, 1H), 1.37 (d, *J* = 6.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 150.1, 141.2, 133.6, 130.1, 129.5, 128.5, 127.2, 121.6, 79.0, 23.9.

HRMS (ESI) Calcd. for $C_{16}H_{14}D_3O_3$ [$(M+H)^+$] 260.1366, found 260.1369.



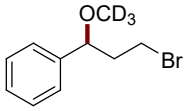
(1-(*d*^{3}-methoxy)hexyl)benzene (10):

According to the general procedure, hexylbenzene (81 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (70 mg, 72 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 - 7.24 (m, 2H), 7.21 (d, *J* = 7.1 Hz, 3H), 4.00 (t, *J* = 6.6 Hz, 1H), 1.80 - 1.66 (m, 1H), 1.59 - 1.52 (m, 1H), 1.37 - 1.28 (m, 1H), 1.25 - 1.13 (m, 6H), 0.78 (t, *J* = 6.1 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 142.6, 128.3, 127.4, 126.7, 84.0, 55.7 (quin, *J* = 21.3 Hz), 38.2, 31.8, 25.5, 22.6, 14.0.

HRMS (ESI) Calcd. for $C_{13}H_{18}D_3O$ [$(M+H)^+$] 196.1781, found 196.1790.



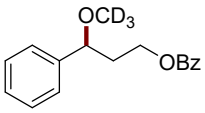
(3-bromo-1-(*d*^{3}-methoxy)propyl)benzene (11):

According to the general procedure, (3-bromopropyl)benzene (100 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (65 mg, 56 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 - 7.27 (m, 2H), 7.26 - 7.20 (m, 3H), 4.28 (dd, *J* = 8.2, 4.9 Hz, 1H), 3.49 (dd, *J* = 15.4, 8.5 Hz, 1H), 3.37 - 3.23 (m, 1H), 2.30 - 2.19 (m, 1H), 2.08 - 1.97 (m, 1H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 141.1, 128.6, 127.9, 126.6 81.3, 56.0 (quin, *J* = 21.5 Hz), 41.1 30.3.

HRMS (ESI) Calcd. for $C_{10}H_{11}D_3BrO$ [$(M+H)^+$] 232.0416, found 232.0424.



3-(*d*^{3}-methoxy)-3-phenylpropyl benzoate (12):

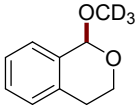
According to the general procedure, 3-phenylpropyl benzoate [6] (120 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was

subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (89 mg, 65 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.5 Hz, 2H), 7.48 (t, *J* = 7.4 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.32 - 7.14 (m, 5H), 4.42 - 4.33 (m, 1H), 4.30 - 4.20 (m, 2H), 2.24 - 2.13 (m, 1H), 2.07 - 1.96 (m, 1H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 166.5, 141.5, 132.9, 130.4, 129.6, 128.6, 128.4, 127.9, 126.6, 80.7, 62.0, 55.9 (quin, *J* = 21.4 Hz), 37.30.

HRMS (ESI) Calcd. for $C_{17}H_{16}D_3O_3$ [$(M+H)^+$] 274.1522, found 274.1528.



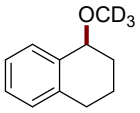
1-(*d*^{3}-methoxy)isochromane (13):

According to the general procedure, isochromane (67 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (71 mg, 85 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 - 7.11 (m, 3H), 7.05 (d, *J* = 7.2 Hz, 1H), 5.38 (s, 1H), 4.11 - 4.00 (m, 1H), 3.89 - 3.79 (m, 1H), 3.03 - 2.90 (m, 1H), 2.60 - 2.52 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 134.1, 134.0, 128.5, 128.2, 127.4, 126.4, 97.7, 57.8, 28.0.

HRMS (ESI) Calcd. for $C_{10}H_{10}D_3O_2$ [$(M+H)^+$] 168.1104, found 168.1108.



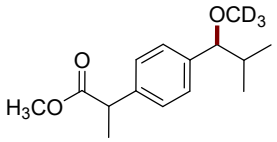
1-(*d*^{3}-methoxy)-1,2,3,4-tetrahydronaphthalene (14):

According to the general procedure, 1,2,3,4-tetrahydronaphthalene (66 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (58 mg, 70 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 - 7.24 (m, 1H), 7.15 - 7.06 (m, 2H), 7.06 - 6.98 (m, 1H), 4.24 (t, *J* = 4.6 Hz, 1H), 2.80 - 2.70 (m, 1H), 2.70 - 2.57 (m, 1H), 2.00 - 1.87 (m, 2H), 1.86 - 1.74 (m, 1H), 1.73 - 1.58 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 136.6, 129.3, 129.0, 127.5, 125.7, 29.1, 27.4, 18.7.

HRMS (ESI) Calcd. for $C_{11}H_{12}D_3O$ [$(M+H)^+$] 166.1311, found 166.1317.



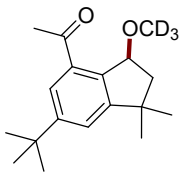
methyl 2-(4-(1-(*d*^{3}-methoxy)-2-methylpropyl)phenyl)propanoate (15):

According to the general procedure, methyl 2-(4-isobutylphenyl)propanoate [7] (110 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (89 mg, 70 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.19 (d, *J* = 7.9 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 3.66 (dd, *J* = 6.7, 4.7 Hz, 2H), 3.60 (s, 3H), 1.87 -1.76 (m, 1H), 1.43 (d, *J* = 7.2 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 3H), 0.66 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 175.2, 140.0, 139.4, 127.7, 127.1, 89.3, 56.2 (quin, *J* = 21.2 Hz), 52.0, 45.1, 34.7, 19.0, 18.9, 18.6.

HRMS (ESI) Calcd. for $C_{15}H_{20}D_3O_3$ $[(M+H)^+]$ 254.1835, found 254.1837.



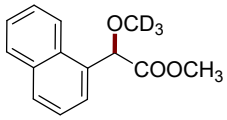
1-(6-(*tert*-butyl)-3-(*d*^{3}-methoxy)-1,1-dimethyl-2,3-dihydro-1*H*-inden-4-yl)ethan-1-one (16):

According to the general procedure, 1-(6-(*tert*-butyl)-1,1-dimethyl-2,3-dihydro- 1*H*-inden-4-yl)ethan-1-one (122 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (100 mg, 79 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 1.7 Hz, 1H), 7.28 (d, *J* = 1.7 Hz, 1H), 5.14 (dd, *J* = 5.5, 3.3 Hz, 1H), 2.55 (s, 3H), 2.01 (dd, *J* = 4.3, 2.8 Hz, 2H), 1.28 (s, 3H), 1.27 (s, 9H), 1.23 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 200.8, 154.2, 152.6, 137.8, 135.2, 125.0, 123.2, 81.5), 46.1, 42.6, 34.9, 31.4, 31.2, 29.7, 28.7.

HRMS (ESI) Calcd. for $C_{18}H_{24}D_3O_2$ $[(M+H)^+]$ 278.2199, found 278.2202.



methyl 2-(*d*^{3}-methoxy)-2-(naphthalen-1-yl)acetate (17):

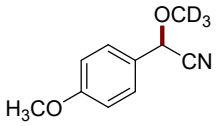
According to the general procedure, methyl 2-(naphthalen-1-yl)acetate [7] (100 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.),

$Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (99 mg, 85 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, J = 8.3 Hz, 1H), 7.89 (t, J = 7.5 Hz, 2H), 7.64 (d, J = 7.0 Hz, 1H), 7.61 - 7.47 (m, 3H), 5.42 (s, 1H), 3.71 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 133.9, 132.0, 131.0, 129.5, 128.7, 126.8, 126.6, 125.9, 125.2, 123.9, 81.03, 52.3.

HRMS (ESI) Calcd. for $C_{14}H_{12}D_3O_3$ $[(M+H)^+]$ 234.1209, found 234.1216.



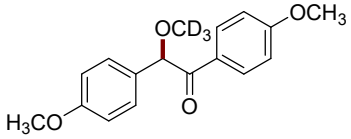
2-(*d*^{3}-methoxy)-2-(4-methoxyphenyl)acetonitrile (18):

According to the general procedure, 2-(4-methoxyphenyl)acetonitrile (74 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (74 mg, 82 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, J = 8.7 Hz, 2H), 6.94 (d, J = 8.7 Hz, 2H), 5.14 (s, 1H), 3.83 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 160.8, 128.9, 125.4, 117.2, 114.4, 71.8, 55.4.

HRMS (ESI) Calcd. for $C_{10}H_9D_3NO_2$ $[(M+H)^+]$ 181.1056, found 181.1063.



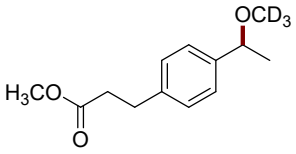
2-(*d*^{3}-methoxy)-1,2-*bis*(4-methoxyphenyl)ethan-1-one (19):

According to the general procedure, 1,2-*bis*(4-methoxyphenyl)ethan-1-one (128 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (20 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (106 mg, 73 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, J = 8.9 Hz, 2H), 7.36 (d, J = 8.7 Hz, 2H), 6.86 (dd, J = 8.9, 2.4 Hz, 4H), 5.44 (s, 1H), 3.82 (s, 3H), 3.76 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 195.6, 163.5, 159.7, 131.34, 129.0, 128.5, 128.0, 114.3, 113.7, 85.7, 55.4, 55.3.

HRMS (ESI) Calcd. for $C_{17}H_{16}D_3O_4$ [$(M+H)^+$] 290.1472, found 290.1478.



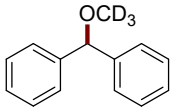
methyl 3-(4-(1-(*d*^{3}-methoxy)ethyl)phenyl)propanoate (20):

According to the general procedure, methyl 3-(4-ethylphenyl)propanoate [7] (96 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (87 mg, 77 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 4.27 (q, *J* = 6.4 Hz, 1H), 3.68 (s, 3H), 2.95 (t, *J* = 7.8 Hz, 2H), 2.64 (t, *J* = 7.9 Hz, 2H), 1.42 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 141.5, 139.7, 128.3, 126.3, 79.2, 51.6, 35.6, 30.6), 23.7.

HRMS (ESI) Calcd. for $C_{13}H_{16}D_3O_3$ [$(M+H)^+$] 226.1522, found 226.1526.



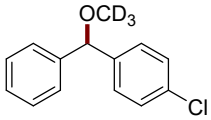
((*d*^{3}-methoxy)methylene)dibenzene (21):

According to the general procedure, diphenylmethane (84 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (84 mg, 84 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (q, *J* = 7.7 Hz, 8H), 7.16 (t, *J* = 6.7 Hz, 2H), 5.16 (s, 1H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 142.1, 128.4, 127.5, 126.9, 85.4, 56.2 (quin, *J* = 21.3 Hz).

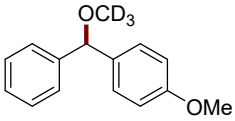
HRMS (ESI) Calcd. for $C_{14}H_{12}D_3O$ [$(M+H)^+$] 202.1311, found 202.1317.



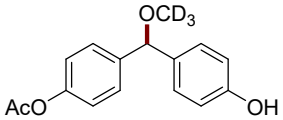
1-chloro-4-((*d*^{3}-methoxy)(phenyl)methyl)benzene (22):

According to the general procedure, 1-benzyl-4-chlorobenzene (101 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (93 mg, 79 % yield).

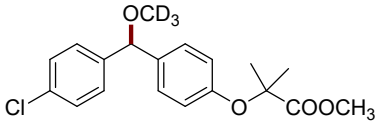
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 - 7.29 (m, 4H), 7.29 - 7.22 (m, 5H), 5.20 (s, 1H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 141.6, 140.7, 133.1, 128.5, 128.2, 127.7, 126.8, 84.6, 56.1 (quin, J = 21.9 Hz).
HRMS (ESI) Calcd. for $C_{14}H_{11}D_3ClO$ [$(M+H)^+$] 236.0921, found 236.0928.



1-methoxy-4-((*d*^{3}-methoxy)(phenyl)methyl)benzene (23):
According to the general procedure, 1-benzyl-4-methoxybenzene (99 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (95 mg, 82 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 - 7.29 (m, 4H), 7.25 (dd, J = 5.8, 2.8 Hz, 3H), 6.85 (d, J = 8.7 Hz, 2H), 5.20 (s, 1H), 3.78 (s, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 142.3, 134.3, 128.3, 128.2, 127.3, 126.8, 113.8, 84.9, 55.2.
HRMS (ESI) Calcd. for $C_{15}H_{14}D_3O_2$ [$(M+H)^+$] 232.1412, found 232.1421.



4-((4-hydroxyphenyl)(*d*^{3}-methoxy)methyl)phenyl acetate (24):
According to the general procedure, 4-(4-hydroxybenzyl)phenyl acetate [8] (121 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (20 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (98 mg, 71 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, J = 8.5 Hz, 2H), 7.15 (d, J = 8.5 Hz, 2H), 7.03 (d, J = 8.6 Hz, 2H), 6.72 (d, J = 8.6 Hz, 2H), 5.45 (s, 1H), 5.18 (s, 1H), 2.28 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.7, 155.2, 149.8, 139.8, 133.7, 129.9, 128.5, 127.9, 121.4, 115.3, 84.3, 21.1.
HRMS (ESI) Calcd. for $C_{16}H_{14}D_3O_4$ [$(M+H)^+$] 276.1310, found 276.1315.



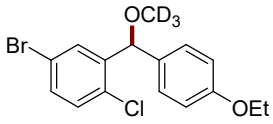
methyl 2-(4-((4-chlorophenyl)(*d*^{3}-methoxy)methyl)phenoxy)-2-methylpropanoate (25):
According to the general procedure, methyl 2-(4-(4-chlorobenzyl)phenoxy)-2- methylpropanoate [9] (159 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0

mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (100 mg, 57 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, J = 4.1 Hz, 2H), 7.19 (dd, J = 9.3, 4.5 Hz, 2H), 7.13 (d, J = 8.6 Hz, 2H), 6.70 (d, J = 8.5 Hz, 2H), 5.11 (s, 1H), 3.67 (s, 3H), 1.50 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.8, 154.6, 142.0, 135.8, 128.5, 128.3, 128.2, 127.9, 127.4, 126.9, 118.8, 84.8, 79.0, 52.4, 25.3.

HRMS (ESI) Calcd. for $C_{19}H_{19}D_3ClO_4$ $[(M+H)^+]$ 352.1390, found 352.1401.



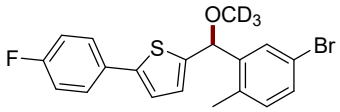
4-bromo-1-chloro-2-((4-ethoxyphenyl)(*d*^{3}-methoxy)methyl)benzene (26):

According to the general procedure, 4-bromo-1-chloro-2-(4-ethoxybenzyl)benzene (163 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (170 mg, 95 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 2.4 Hz, 1H), 7.24 (dd, J = 8.5, 2.4 Hz, 1H), 7.21 - 7.15 (m, 2H), 7.10 (d, J = 8.5 Hz, 1H), 6.77 (d, J = 8.7 Hz, 2H), 5.44 (s, 1H), 3.93 (q, J = 7.0 Hz, 2H), 1.32 (t, J = 7.0 Hz, 3H).

^{13}C NMR (150 MHz, $CDCl_3$) δ 158.6, 142.0, 131.7, 131.5, 131.4, 130.9, 130.6, 128.7, 121.0, 114.3, 80.7, 63.4, 56.1 (quin, J = 21.4 Hz), 14.8.

HRMS (ESI) Calcd. for $C_{16}H_{14}D_3BrClO_2$ $[(M+H)^+]$ 358.0289, found 358.0297.

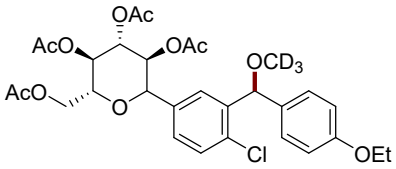


2-((5-bromo-2-methylphenyl)(*d*^{3}-methoxy)methyl)-5-(4-fluorophenyl)thiophene (27):

According to the general procedure, 2-(5-bromo-2-methylbenzyl)-5-(4-fluorophenyl) thiophene (181 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a white solid (181 mg, 92 % yield).

^1H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, J = 2.0 Hz, 1H), 7.55 -7.46 (m, 2H), 7.36 (dd, J = 8.1, 2.1 Hz, 1H), 7.09 - 6.99 (m, 4H), 6.76 (d, J = 3.6 Hz, 1H), 5.53 (s, 1H), 2.25 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ 163.3, 161.3, 143.8, 143.7, 141.2, 134.5, 132.2, 130.8, 130.5, 129.0, 127.4, 127.3, 126.8, 122.3, 120.1, 115.8, 115.7, 77.8, 56.3 (quin, J = 21.4 Hz), 18.7.
HRMS (ESI) Calcd. for $C_{19}H_{14}D_3BrFOS$ [$(M+H)^+$] 394.0356, found 394.0359.



(2*R*,3*R*,4*R*,5*S*)-2-(acetoxymethyl)-6-(4-chloro-3-((4-ethoxyphenyl)(d^3-methoxy)methyl)phenyl)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate (28):

According to the general procedure, (2*R*,3*R*,4*R*,5*S*)-2-(acetoxymethyl)-6-(4-chloro- 3-(4-ethoxybenzyl)phenyl)tetrahydro-2*H*-pyran-3,4,5-triyl triacetate (289 mg, 0.5 mmol, 1.0 equiv.), d^4-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a white solid (268 mg, 88 % yield).

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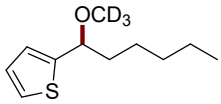
^{1}H NMR (400 MHz, $CDCl_3$, **mixture**) δ 7.74 (d, J = 8.9 Hz, 2H), 7.43 (s, 2H), 7.29 (d, J = 9.6 Hz, 2H), 6.93 (d, J = 8.9 Hz, 2H), 6.23 (6.21) (s, 1H), 5.35 - 5.30 (m, 1H), 5.25 - 5.17 (m, 1H), 5.10 - 5.03 (m, 1H), 4.43 - 4.36 (m, 1H), 4.30 - 4.25 (m, 1H), 4.19 - 4.09(m, 2H), 3.86 - 3.83 (m, 1H), 3.42 - 3.31 (m, 1H), 2.10 - 2.05 (m, 6H), 2.02 - 1.96 (1.87 - 1.83) (m, 6H), 1.47 - 1.41 (m, 3H).
^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 193.2, 170.6, 170.2, 169.4, 168.8, 163.7, 144.6, 144.4, 139.0, 135.3, 132.5, 131.5, 131.4, 130.1, 130.0, 129.6, 129.5, 129.0, 128.8, 127.7, 127.6, 114.4, 79.2, 79.0, 76.2, 74.0, 73.9, 72.6, 72.5, 68.5, 63.9, 62.2, 20.7, 20.6, 20.4, 14.6.
HRMS (ESI) Calcd. for $C_{30}H_{33}D_3ClO_{11}$ [$(M+H)^+$] 610.2134, found 610.2139.

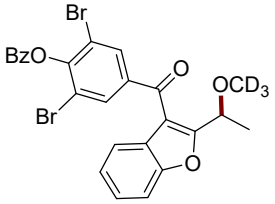


2-(1-(d^3-methoxy)hexyl)thiophene (29):

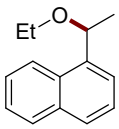
According to the general procedure, 2-hexylthiophene (84 mg, 0.5 mmol, 1.0 equiv.), d^4-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (71 mg, 71 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, J = 5.1 Hz, 1H), 6.89 (d, J = 4.9 Hz, 2H), 4.28 (t, J = 6.8 Hz, 1H), 1.84 (dd, J = 18.4, 10.8 Hz, 1H), 1.67 (dd, J = 12.3, 6.1 Hz, 1H), 1.39 - 1.29 (m, 1H), 1.21 (s, 7H), 0.80 (t, J = 5.7 Hz, 3H).

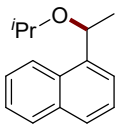
^{13}C NMR (150 MHz, $CDCl_3$) δ 146.4, 126.3, 125.2, 124.8, 79.4, 55.7 (quin, J = 21.1 Hz), 38.3, 31.6, 25.5, 22.6, 14.1.
HRMS (ESI) Calcd. for $C_{11}H_{16}D_3OS$ [$(M+H)^+$] 202.1345, found 202.1348.



2,6-dibromo-4-(2-(1-(*d*^{3}-methoxy)ethyl)benzofuran-3-carbonyl)phenyl benzoate (30):
According to the general procedure, 2,6-dibromo-4-(2-ethylbenzofuran-3-carbonyl) phenyl benzoate [5] (264 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (20 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (106 mg, 73 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.33 - 8.23 (m, 2H), 8.12 (s, 2H), 7.71 (t, J = 7.5 Hz, 1H), 7.58 (dd, J = 15.4, 7.8 Hz, 3H), 7.45 - 7.35 (m, 2H), 7.30 (dd, J = 11.1, 4.0 Hz, 1H), 4.76 (q, J = 6.5 Hz, 1H), 1.64 (d, J = 6.6 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 187.5, 162.9, 162.6, 154.0, 150.2, 138.5, 134.4, 133.2, 130.6, 128.8, 127.9, 125.7, 125.6, 124.2, 121.3, 118.6, 117.3, 111.9, 71.1, 19.3.
HRMS (ESI) Calcd. for $C_{25}H_{16}D_3Br_2O_5$ [$(M+H)^+$] 561.9762, found 561.9767.



1-(1-ethoxyethyl)naphthalene (31):
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), EtOH (115 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (75 mg, 75 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 7.7 Hz, 1H), 7.91 - 7.84 (m, 1H), 7.76 (d, J = 8.1 Hz, 1H), 7.58 (d, J = 6.9 Hz, 1H), 7.52 - 7.41 (m, 3H), 5.16 (q, J = 6.5 Hz, 1H), 3.44 (q, J = 7.0 Hz, 2H), 1.61 (d, J = 6.5 Hz, 3H), 1.23 (t, J = 7.0 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 134.0, 130.9, 128.9, 127.7, 125.8, 125.6, 125.4, 123.4, 123.2, 75.5, 64.1, 23.6, 15.6.
HRMS (ESI) Calcd. for $C_{14}H_{17}O$ [$(M+H)^+$] 201.1279, found 201.1283.



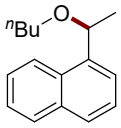
1-(1-isopropoxyethyl)naphthalene (32):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), isopropyl alcohol (150 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (66 mg, 62 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, J = 7.9 Hz, 1H), 7.91 - 7.83 (m, 1H), 7.76 (d, J = 8.2 Hz, 1H), 7.61 (d, J = 7.0 Hz, 1H), 7.53 - 7.44 (m, 3H), 5.28 (q, J = 6.5 Hz, 1H), 3.58 - 3.49 (m, 1H), 1.58 (d, J = 6.6 Hz, 3H), 1.20 (d, J = 6.0 Hz, 3H), 1.14 (d, J = 6.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 133.9, 130.8, 128.9, 127.6 125.7, 125.5, 125.3, 123.5, 123.4, 72.3, 68.8, 24.2, 23.4, 21.4.

HRMS (ESI) Calcd. for $C_{15}H_{19}O$ $[(M+H)^+]$ 215.1436, found 215.1441.



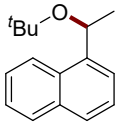
1-(1-butoxyethyl)naphthalene (33):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), 1-butanol (185 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (81 mg, 71 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, J = 7.6 Hz, 1H), 7.90 - 7.82 (m, 1H), 7.76 (d, J = 8.1 Hz, 1H), 7.57 (d, J = 7.0 Hz, 1H), 7.51 - 7.44 (m 3H), 5.13 (q, J = 6.5 Hz, 1H), 3.41 - 3.34 (m, 2H), 1.60 (d, J = 6.6 Hz, 3H), 1.57 (dd, J = 8.3, 4.7 Hz, 2H), 1.38 (dd, J = 14.7, 7.3 Hz, 2H), 0.88 (t, J = 7.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 133.9, 130.8, 128.8, 127.6, 125.7, 125.5, 125.4, 123.4, 123.2, 75.6, 68.7, 32.2, 23.5, 19.4, 13.9.

HRMS (ESI) Calcd. for $C_{16}H_{21}O$ $[(M+H)^+]$ 229.1592, found 229.1599.



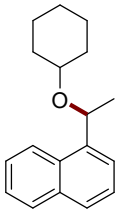
1-(1-(*tert*-butoxy)ethyl)naphthalene (34):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), *tert*-butanol (185 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (78 mg, 68 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, J = 8.1 Hz, 1H), 7.88 - 7.82 (m, 1H), 7.72 (t, J = 8.5 Hz, 2H), 7.54 - 7.42 (m, 3H), 5.38 (q, J = 6.5 Hz, 1H), 1.51 (d, J = 6.5 Hz, 3H), 1.18 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 143.3, 133.7, 129.8, 128.9, 126.89, 125.6, 125.1, 123.3,123.1, 74.3, 67.0, 28.4, 26.0.

HRMS (ESI) Calcd. for $C_{16}H_{21}O$ $[(M+H)^+]$ 229.1592, found 229.1601.



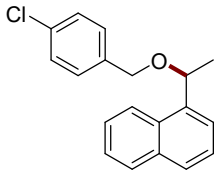
1-(1-(cyclohexyloxy)ethyl)naphthalene (35):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), cyclohexanol (250 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (76 mg, 60 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, J = 7.7 Hz, 1H), 7.90 - 7.83 (m, 1H), 7.76 (d, J = 8.2 Hz, 1H), 7.63 (d, J = 7.0 Hz, 1H), 7.48 (dt, J = 11.8, 4.8 Hz, 3H), 5.34 (q, J = 6.5 Hz, 1H), 3.26 - 3.17 (m, 1H), 2.03 (d, J = 10.3 Hz, 1H), 1.80 (d, J = 10.3 Hz, 1H), 1.74 - 1.62 (m, 2H), 1.58 (d, J = 6.6 Hz, 3H), 1.48 (dd, J = 10.9, 4.8 Hz, 1H), 1.41 - 1.31 (m, 2H), 1.17 - 1.05 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.7, 133.9, 130.79, 128.9, 127.45, 125.7, 125.6, 125.3, 123.4, 75.1, 71.8, 33.5, 31.8, 25.8, 24.4, 24.3, 24.2.

HRMS (ESI) Calcd. for $C_{18}H_{23}O$ $[(M+H)^+]$ 255.1749, found 255.1753.



1-(1-((4-chlorobenzyl)oxy)ethyl)naphthalene (36):

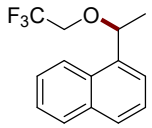
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (4-chlorophenyl)methanol (356 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%),

[Ir{dF(CF$_3$)ppy}$_2$(dtbbpy)]PF$_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH$_3$CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (121 mg, 82 % yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.22 - 8.13 (m, 1H), 7.92 - 7.85 (m, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.61 (d, *J* = 7.0 Hz, 1H), 7.48 (dd, *J* = 9.0, 6.1 Hz, 3H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 7.7 Hz, 2H), 5.22 (q, *J* = 6.5 Hz, 1H), 4.47 (d, *J* = 12.0 Hz, 1H), 4.31 (d, *J* = 12.0 Hz, 1H), 1.65 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 139.0, 137.1, 134.0, 133.2, 130.80, 129.0, 128.9, 128.5, 128.0, 125.9, 125.6, 125.5, 123.6, 123.3, 75.3, 69.7, 23.5.

HRMS (ESI) Calcd. for C$_{19}$H$_{18}$ClO [(M+H)$^+$] 297.1046, found 297.1051.



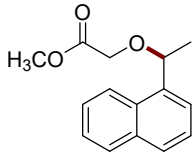
1-(1-(2,2,2-trifluoroethoxy)ethyl)naphthalene (37):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), 2,2,2-trifluoroethan-1-ol (250 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), Ni(acac)$_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), [Ir{dF(CF$_3$)ppy}$_2$(dtbbpy)]PF$_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH$_3$CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (petroleum ether) to provide the title compound as a colourless oil (83 mg, 65 % yield).

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.14 (d, *J* = 7.6 Hz, 1H), 7.89 (dd, *J* = 6.9, 2.4 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.61 - 7.42 (m, 4H), 5.33 (q, *J* = 6.5 Hz, 1H), 3.86 - 3.60 (m, 2H), 1.69 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, CDCl$_3$) δ 137.3, 130.6, 129.0, 128.5, 126.3, 125.7, 125.5, 123.7, 123.0, 77.6, 66.1, 65.8, 29.7, 23.2.

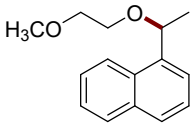
HRMS (ESI) Calcd. for C$_{14}$H$_{14}$F$_3$O [(M+H)$^+$] 255.0997, found 255.1003.



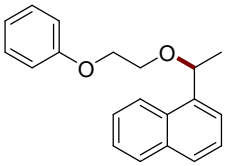
methyl 2-(1-(naphthalen-1-yl)ethoxy)acetate (38):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), methyl 2-hydroxyacetate (225 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), Ni(acac)$_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), [Ir{dF(CF$_3$)ppy}$_2$(dtbbpy)]PF$_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH$_3$CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (102 mg, 84 % yield).

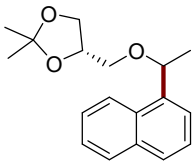
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 7.6 Hz, 1H), 7.88 (dd, *J* = 6.8, 2.5 Hz, 1H), 7.79 (d, *J* = 8.1 Hz, 1H), 7.58 (d, *J* = 6.9 Hz, 1H), 7.54 - 7.43 (m, 3H), 5.33 (q, *J* = 6.5 Hz, 1H), 4.10 (d, *J* = 16.4 Hz, 1H), 3.94 (d, *J* = 16.4 Hz, 1H), 3.72 (s, 3H), 1.70 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 171.0, 137.9, 133.9, 130.8, 128.9, 128.2, 126.1, 125.6, 125.5, 123.6, 123.2, 76.3, 65.8, 51.8, 23.3.
HRMS (ESI) Calcd. for $C_{15}H_{17}O_3$ [$(M+H)^+$] 245.1178 , found 245.1184.



1-(1-(2-methoxyethoxy)ethyl)naphthalene (39):
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), 2-methoxyethan-1-ol (190 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (104 mg, 90 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 1H), 7.72 - 7.64 (m, 1H), 7.58 (d, *J* = 8.2 Hz, 1H), 7.41 (d, *J* = 7.0 Hz, 1H), 7.36 - 7.25 (m, 3H), 5.02 (q, *J* = 6.5 Hz, 1H), 3.43 - 3.29 (m, 4H), 3.20 (s, 3H), 1.46 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2, 133.9, 130.8, 128.8, 127.7, 125.8, 125.5, 125.4, 123.4, 123.3, 76.1, 72.1, 67.9, 59.0, 23.5.
HRMS (ESI) Calcd. for $C_{15}H_{19}O_2$ [$(M+H)^+$] 231.1385, found 231.1391.



1-(1-(2-phenoxyethoxy)ethyl)naphthalene (40):
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), 2-phenoxyethan-1-ol (345 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (126 mg, 86 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 - 8.17 (m, 1H), 7.86 (dd, *J* = 6.9, 2.5 Hz, 1H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.62 (d, *J* = 6.9 Hz, 1H), 7.56 - 7.41 (m, 3H), 7.31 - 7.23 (m, 2H), 6.97 - 6.84 (m, 3H), 5.27 (q, *J* = 6.5 Hz, 1H), 4.17 - 4.06 (m, 2H), 3.79 - 3.67 (m, 2H), 1.65 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 139.0, 133.9, 130.8, 129.4, 128.9, 127.9, 125.9, 125.5, 123.6, 123.4, 120.7, 114.6, 76.3, 67.4, 67.1, 23.4.
HRMS (ESI) Calcd. for $C_{20}H_{21}O_2$ [$(M+H)^+$] 293.1542, found 293.1553.



(4*S*)-2,2-dimethyl-4-((1-(naphthalen-1-yl)ethoxy)methyl)-1,3-dioxolane (41):

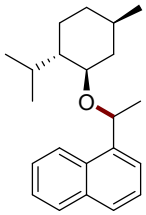
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (*S*)-(2,2-dimethyl-1,3-dioxolan-4-yl)methanol (330 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (123 mg, 86 % yield).

（1:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.21 - 8.13 (m, 1H), 7.90 - 7.83 (m, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.58 - 7.51 (m, 1H), 7.53 - 7.42 (m, 3H), 5.22 - 5.16 (m, 1H), 4.38 - 4.23 (m, 1H), 4.10 - 3.98 (m, 1H), 3.82 - 3.76(3.65 - 3.58) (m, 1H), 3.52 - 3.45 (m, 1H), 3.40 - 3.33 (m, 1H), 1.67 - 1.58 (m, 3H), 1.42 - 1.31 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 139.0, 138.9, 133.9, 130.7, 128.9, 128.0, 127.9, 125.9, 125.5, 123.6, 123.5, 123.4, 123.3, 109.4, 109.3, 76.5, 75.0, 74.8, 70.1, 69.7, 67.0, 66.8, 26.7, 25.4, 23.3.

HRMS (ESI) Calcd. for $C_{18}H_{23}O_3$ $[(M+H)^+]$ 287.1647, found 287.1653.



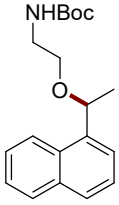
1-(1-(((1*R*,2*S*,5*R*)-2-isopropyl-5-methylcyclohexyl)oxy)ethyl)naphthalene (42):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), DL-menthol (391 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (2 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (87 mg, 56 % yield).

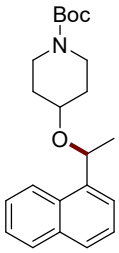
（1.1:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture 1**) δ 8.07 (d, *J* = 8.1 Hz, 1H), 7.83 - 7.75 (m, 1H), 7.67 (d, *J* = 8.2 Hz, 1H), 7.60 (d, *J* = 7.0 Hz, 1H), 7.46 - 7.36 (m, 3H), 5.17 (q, *J* = 6.4 Hz, 1H), 3.26 - 3.15 (m, 1H), 2.43 - 2.32 (m 1H), 1.78 - 1.66 (m, 1H), 1.60 - 1.52 (m, 2H), 1.49 (d, *J* = 6.5 Hz, 4H), 1.31 - 1.18 (m, 2H), 0.91 (d, *J* = 7.1 Hz, 4H), 0.83 (d, *J* = 7.0 Hz, 3H), 0.79 - 0.70 (m, 2H), 0.67 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture 1**) δ 141.6, 133.8, 130.3, 128.8, 127.4, 125.6, 125.5, 125.2, 123.7, 123.5, 78.9, 74.2, 49.1, 42.0, 34.5, 31.6, 25.5, 23.6, 23.1, 22.2, 21.3, 16.2.
HRMS (ESI) Calcd. for $C_{22}H_{31}O$ [$(M+H)^+$] 311.2375, found 311.2381.
^{1}H NMR (400 MHz, $CDCl_3$, **mixture 2**) δ 8.05 (d, J = 8.0 Hz, 1H), 7.73 - 7.65 (m, 1H), 7.57 (d, J = 8.1 Hz, 1H), 7.44 (d, J = 6.7 Hz, 1H), 7.38 - 7.21 (m, 3H), 5.21 (q, J = 6.6 Hz, 1H), 2.88 - 2.76 (m, 1H), 2.25 - 2.17 (m, 1H), 2.11 (d, J = 11.0 Hz, 1H), 1.42 (d, J = 6.6 Hz, 4H), 1.41 - 1.32 (m, 2H), 1.13 - 1.01 (m, 3H), 0.74 (d, J = 6.5 Hz, 3H), 0.68 (d, J = 7.0 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$, **mixture 2**) δ 140.2, 133.8, 130.9, 128.8, 127.6, 125.6, 125.4, 125.3, 124.4, 123.5, 75.9, 70.8, 48.7, 40.6, 34.5, 31.4, 24.9, 24.6, 22.7, 22.5, 21.3, 15.4.
HRMS (ESI) Calcd. for $C_{22}H_{31}O$ [$(M+H)^+$] 311.2375, found 311.2383.



***tert*-butyl (2-(1-(naphthalen-1-yl)ethoxy)ethyl)carbamate (43):**
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), *tert*-butyl (2-hydroxyethyl)carbamate (403 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (20 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (121 mg, 77 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 7.9 Hz, 1H), 7.91 - 7.84 (m, 1H), 7.77 (d, J = 8.1 Hz, 1H), 7.56 - 7.42 (m, 4H), 5.14 (q, J = 6.5 Hz, 1H), 4.93 (s, 1H), 3.43 (dd, J = 11.4, 6.6 Hz, 2H), 3.33 - 3.23 (m, 2H), 1.62 (d, J = 6.5 Hz, 3H), 1.43 (s, 9H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 138.9, 133.9, 130.7, 128.9, 128.0, 125.9, 125.5, 125.4, 123.5, 123.3, 76.1, 67.7, 28.4, 27.5.
HRMS (ESI) Calcd. for $C_{19}H_{26}NO_3$ [$(M+H)^+$] 316.1913, found 316.1917.



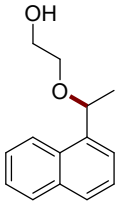
***tert*-butyl 4-(1-(naphthalen-1-yl)ethoxy)piperidine-1-carboxylate (44)：**
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), *tert*-butyl 4-hydroxypiperidine-1-carboxylate (503 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general

procedure and purified by flash chromatography (20 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (135 mg, 76 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 7.7 Hz, 1H), 7.90 - 7.83 (m, 1H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.59 (d, *J* = 6.9 Hz, 1H), 7.54 - 7.41 (m, 3H), 5.31 (q, *J* = 6.5 Hz, 1H), 3.77 (d, *J* = 21.4 Hz, 2H), 3.49 - 3.37 (m, 1H), 3.04 - 2.86 (m, 2H), 1.87 (s, 1H), 1.67 (s, 1H), 1.59 (d, *J* = 6.5 Hz, 4H), 1.54 (dd, *J* = 8.6, 4.2 Hz, 1H), 1.44 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 154.8, 140.0, 133.9, 130.6, 128.9, 127.7, 125.8, 125.5, 125.4, 123.5, 123.3, 79.3, 72.5, 72.1, 28.4, 24.1.

HRMS (ESI) Calcd. for $C_{22}H_{30}NO_3$ [$(M+H)^+$] 356.2221, found 356.2229.



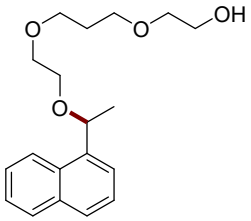
2-(1-(naphthalen-1-yl)ethoxy)ethan-1-ol (45):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), ethylene glycol (155 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (95 mg, 88 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.9 Hz, 1H), 7.92 - 7.83 (m, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.56 (d, *J* = 6.9 Hz, 1H), 7.53 - 7.41 (m, 3H), 5.20 (q, *J* = 6.5 Hz, 1H), 3.78 - 3.68 (m, 2H), 3.55 - 3.46 (m, 2H), 2.13 (br, 1H), 1.65 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.9, 133.9, 130.7, 128.9, 128.0, 125.9, 125.5, 123.4, 123.2, 76.2, 69.8, 62.1, 23.2.

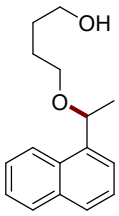
HRMS (ESI) Calcd. for $C_{14}H_{17}O_2$ [$(M+H)^+$] 217.1229, found 217.1240.



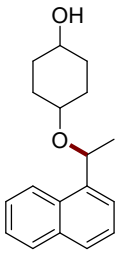
2-(3-(2-(1-(naphthalen-1-yl)ethoxy)ethoxy)propoxy)ethan-1-ol (46):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), triethylene glycol (411 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % dichloromethane/methanol) to provide the title compound as a colourless oil (137 mg, 86 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.18 (d, *J* = 7.6 Hz, 1H), 7.90 - 7.83 (m, 1H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.58 (d, *J* = 7.0 Hz, 1H), 7.52 - 7.41 (m, 3H), 5.20 (q, *J* = 6.5 Hz, 1H), 3.78 - 3.51 (m, 13H), 2.68 (s, 1H), 1.63 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 133.9, 130.7, 128.8, 127.8, 125.8, 125.5, 125.4, 123.4, 123.3, 76.1, 72.5, 70.7, 70.6, 70.3, 67.9, 61.7, 23.4.
HRMS (ESI) Calcd. for $C_{19}H_{27}O_4$ [$(M+H)^+$] 319.1909, found 319.1917.



4-(1-(naphthalen-1-yl)ethoxy)butan-1-ol (47):
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), butane-1,4-diol (225 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (92 mg, 75 % yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.8 Hz, 1H), 7.82 - 7.76 (m, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.48 (d, *J* = 6.7 Hz, 1H), 7.45 - 7.35 (m, 3H), 5.09 (q, *J* = 6.5 Hz, 1H), 3.56 (t, *J* = 5.7 Hz, 2H), 3.40 - 3.27 (m, 2H), 2.17 (s, 1H), 1.67 - 1.56 (m, 4H), 1.54 (d, *J* = 6.6 Hz, 3H).
^{13}C NMR (100 MHz, $CDCl_3$) δ 139.2, 133.9, 130.7, 128.9, 127.8, 125.8, 125.5, 125.4, 123.3, 123.2, 75.9, 68.7, 62.7, 30.2, 26.9, 23.3.
HRMS (ESI) Calcd. for $C_{16}H_{21}O_2$ [$(M+H)^+$] 245.1542, found 245.1549.



4-(1-(naphthalen-1-yl)ethoxy)cyclohexan-1-ol (48):
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), cyclohexane-1,4-diol (290 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (97 mg, 72 % yield).
（1.2:1 dr）

^{1}H NMR (400 MHz, $CDCl_3$, **mixture 1**) δ 8.14 (d, *J* = 7.7 Hz, 1H), 7.84 - 7.76 (m, 1H), 7.69 (d, *J* = 8.1 Hz, 1H), 7.55 (d, *J* = 6.9 Hz, 1H), 7.46 - 7.35 (m, 3H), 5.21 (q, *J* = 6.5 Hz, 1H), 3.67 - 3.61 (m, 1H), 3.34 - 3.25 (m, 1H), 2.01 - 1.81 (m, 2H), 1.77 - 1.61 (m, 4H), 1.52 (d, *J* = 6.5 Hz, 4H), 1.44 - 1.33 (m, 2H).

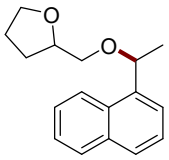
^{13}C NMR (100 MHz, $CDCl_3$, **mixture 1**) δ 140.4, 133.9, 130.7, 128.9, 127.6, 125.7, 125.5, 125.4, 123.5, 77.2, 72.1, 30.7, 30.5, 28.8, 26.7, 24.2.

HRMS (ESI) Calcd. for $C_{18}H_{23}O_2$ [$(M+H)^+$] 271.1698, found 271.1703.

^{1}H NMR (400 MHz, $CDCl_3$, **mixture 2**) δ 8.15 - 8.09 (m, 1H), 7.81 (dd, *J* = 6.7, 2.8 Hz, 1H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.53 (d, *J* = 6.7 Hz, 1H), 7.46 - 7.37 (m, 3H), 5.23 (q, *J* = 6.6 Hz, 1H), 3.64 - 3.53 (m, 1H), 3.25 - 3.15 (m, 1H), 2.10 - 2.03 (m, 1H), 1.96 - 1.77 (m, 4H), 1.50 (d, *J* = 6.5 Hz, 4H), 1.42 - 1.30 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture 2**) δ 140.3, 130.7, 128.9, 127.6, 125.8, 125.4, 123.4, 77.2, 74.3, 72.6, 69.8, 33.0, 32.7, 29.3, 29.0, 24.2.

HRMS (ESI) Calcd. for $C_{18}H_{23}O_2$ [$(M+H)^+$] 271.1698, found 271.1707.



2-((1-(naphthalen-1-yl)ethoxy)methyl)tetrahydrofuran (49):

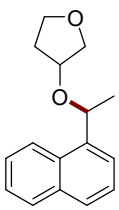
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (tetrahydrofuran-2-yl)methanol (255 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (109 mg, 85 % yield).

（1:1 dr）

^{1}H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.27 - 8.13 (m, 1H), 7.91 - 7.83 (m, 1H), 7.76 (d, *J* = 8.1 Hz, 1H), 7.59 (dd, *J* = 12.8, 7.0 Hz, 1H), 7.55 - 7.41 (m, 3H), 5.26 - 5.15 (m, 1H), 4.14 - 4.04 (m, 1H), 3.92 - 3.82 (m, 1H), 3.77 (dd, *J* = 14.8, 6.9 Hz, 1H), 3.46 - 3.32 (m, 2H), 1.96 - 1.77 (m, 3H), 1.63 (t, *J* = 6.4 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 139.4, 139.3, 133.9, 130.8, 128.8, 127.75, 127.7, 125.8, 125.5, 125.4, 123.6, 123.5, 123.3, 78.2, 77.8, 76.5, 76.0, 71.7, 71.3, 68.3, 28.3, 28.0, 25.6, 25.5, 23.5.

HRMS (ESI) Calcd. for $C_{17}H_{21}O_2$ [$(M+H)^+$] 257.1542, found 257.1549.



3-(1-(naphthalen-1-yl)ethoxy)tetrahydrofuran (50):

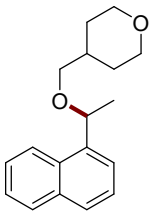
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), tetrahydrofuran-3-ol (220 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (89 mg, 74 % yield).

（1:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.23 - 8.17 (m, 1H), 7.92 - 7.84 (m, 1H), 7.81 - 7.77 (m, 1H), 7.62 - 7.41 (m, 4H), 5.25 - 5.11 (m, 1H), 4.12 - 4.03 (m, 1H), 4.02 - 3.91 (m, 1H), 4.02 - 3.91(3.75 - 3.62) (m, 1H), 3.85 - 3.75 (m, 1H), 3.75 - 3.62 (m, 1H), 2.18 - 1.80 (m, 2H), 1.57 - 1.49 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 139.4, 139.3, 133.9, 130.7, 128.9, 127.9, 125.9, 125.5, 123.8, 123.7, 123.3, 77.3, 77.0, 74.3, 74.0, 73.5, 72.7, 67.1, 33.4, 32.4, 23.9.

HRMS (ESI) Calcd. for $C_{16}H_{19}O_2$ $[(M+H)^+]$ 243.1385, found 243.1391.



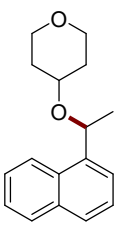
4-((1-(naphthalen-1-yl)ethoxy)methyl)tetrahydro-2*H*-pyran (51):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (tetrahydro-2*H*-pyran-4-yl)methanol (290 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (112 mg, 83 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.21 - 8.14 (m, 1H), 7.87 (dd, J = 6.6, 2.8 Hz, 1H), 7.77 (d, J = 8.1 Hz, 1H), 7.55 (d, J = 6.8 Hz, 1H), 7.52 - 7.42 (m, 3H), 5.11 (q, J = 6.5 Hz, 1H), 3.95 (dd, J = 11.2, 3.1 Hz, 2H), 3.39 (t, J = 11.2 Hz, 2H), 3.26 - 3.18 (m, 2H), 1.95 - 1.81 (m, 1H), 1.68 (dd, J = 21.4, 8.1 Hz, 2H), 1.59 (t, J = 7.0 Hz, 3H), 1.37 - 1.22 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6, 133.9, 130.8, 128.9, 127.7, 125.8, 125.5, 125.4, 123.4, 123.3, 76.0, 73.9, 67.7, 35.7, 30.1, 30.0, 23.4.

HRMS (ESI) Calcd. for $C_{18}H_{23}O_2$ $[(M+H)^+]$ 271.1698, found 271.1701.



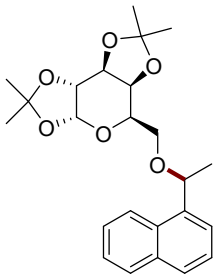
4-(1-(naphthalen-1-yl)ethoxy)tetrahydro-2*H*-pyran (52):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), tetrahydro-2H-pyran-4-ol (255 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (90 mg, 70 % yield).

^1H NMR (400 MHz, $CDCl_3$) δ 8.22 - 8.17 (m, 1H), 7.86 (dd, *J* = 7.0, 2.4 Hz, 1H), 7.76 (d, *J* = 8.2 Hz, 1H), 7.60 (d, *J* = 6.9 Hz, 1H), 7.54 - 7.42 (m, 3H), 5.33 (q, *J* = 6.5 Hz, 1H), 3.97 - 3.91 (m, 1H), 3.90 - 3.84 (m, 1H), 3.50 - 3.41 (m, 1H), 3.36 - 3.22 (m, 2H), 2.00 - 1.91 (m, 1H), 1.79 - 1.62 (m, 3H), 1.60 (d, *J* = 6.5 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0, 133.9, 130.6, 128.9 127.7, 125.8 125.5, 125.4, 123.5, 123.3, 72.1, 71.5, 65.9, 65.7, 33.5, 32.0, 24.1.

HRMS (ESI) Calcd. for $C_{17}H_{21}O_2$ $[(M+H)^+]$ 257.1542, found 257.1544.



(3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyl-5-((1-(naphthalen-1-yl)ethoxy)methyl)tetrahydro-5*H*-*bis*([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran (53):

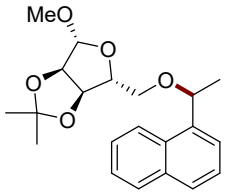
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), ((3a*R*,5*R*,5a*S*,8a*S*,8b*R*)-2,2,7,7-tetramethyltetrahydro-5*H*-*bis*([1,3]dioxolo)[4,5-*b*:4',5'-*d*]pyran-5-yl)methanol (651 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (170 mg, 82 % yield).

（1.1:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.22 - 8.17 (m, 1H), 7.86 - 7.84 (m, 1H), 7.76 - 7.74 (m, 1H), 7.59 - 7.57 (m, 1H), 7.51 - 7.37 (m, 3H), 5.56 - 5.49 (m, 1H), 5.30 - 5.20 (m, 1H), 4.60 - 4.54 (m, 1H), 4.35 - 4.16 (m, 2H), 4.03 - 3.96 (m, 1H), 3.60 - 3.59 (d, *J* = 6.7 Hz, 2H), 1.64 - 1.62 (m, 3H), 1.58 (1.49) (s, 3H), 1.40 (1.37) (s, 3H), 1.34 - 1.27 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 139.1, 133.9, 130.8, 128.8, 128.7, 127.8, 125.7, 125.5, 125.4, 125.3, 123.6, 123.5, 123.4, 109.1, 108.5, 108.4, 96.3, 76.3, 75.9, 71.1, 71.0, 70.7, 70.6, 67.5, 67.3, 67.0, 66.5, 26.1, 26.0, 24.9, 24.4, 24.3, 23.13.

HRMS (ESI) Calcd. for $C_{24}H_{31}O_6$ $[(M+H)^+]$ 415.2121, found 415.2125.



(3a*R*,4*R*,6*R*,6a*R*)-4-methoxy-2,2-dimethyl-6-((1-(naphthalen-1-yl)ethoxy)methyl)tetrahydrofuro[3,4-*d*][1,3]dioxole (54):

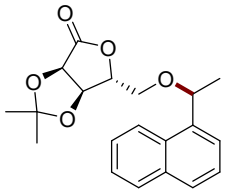
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), ((3a*R*,4*R*,6*R*,6a*R*)-6-methoxy-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)methanol (511 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (149 mg, 83 % yield).

（1.08:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.18 - 8.16 (m, 1H), 7.90 - 7.83 (m, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 7.58 - 7.55 (m, 1H), 7.53 - 7.42 (m, 3H), 5.21 - 5.11 (m, 1H), 4.94 (4.92) (s, 1H), 4.76 (4.60) (d, *J* = 6.0 Hz, 1H), 4.57 (4.49) (d, *J* = 6.0 Hz, 1H), 4.44 - 4.32 (m, 1H), 3.50 - 3.29 (m, 2H), 3.26 (3.18) (s, 3H), 1.64 - 1.61 (m, 3H), 1.49 (s, 3H), 1.34 (1.30) (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 139.1, 134.0, 130.7, 128.9, 128.0, 127.9, 125.9, 125.6, 125.5, 123.5, 123.4, 112.4, 109.3, 85.6, 85.5, 85.3, 85.2, 82.4, 82.2, 76.5, 69.9, 69.7, 54.9, 54.8, 26.6, 26.5, 25.1, 23.5, 23.4.

HRMS (ESI) Calcd. for $C_{21}H_{27}O_5$ $[(M+H)^+]$ 359.1858, found 359.1866.



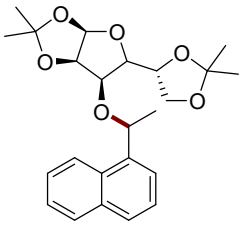
(3a*R*,6*R*,6a*R*)-2,2-dimethyl-6-((1-(naphthalen-1-yl)ethoxy)methyl)dihydrofuro[3,4-*d*][1,3]dioxol-4(3a*H*)-one (55):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (3a*R*,6*R*,6a*R*)-6-(hydroxymethyl)-2,2-dimethyldihydrofuro[3,4-*d*][1,3]dioxol-4(3a*H*)-one (470 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (144 mg, 84 % yield).

（1.2:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture 1**) δ 8.13 - 8.07 (m, 1H), 7.89 (dd, *J* = 6.6, 2.8 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.55 - 7.41 (m, 4H), 5.11 (q, *J* = 6.5 Hz, 1H), 4.95 (d, *J* = 5.5 Hz, 1H), 4.73 (d, *J* = 5.5 Hz, 1H), 4.59 (t, *J* = 1.8 Hz, 1H), 3.65 - 3.53 (m, 2H), 1.60 (d, *J* = 6.6 Hz, 3H), 1.48 (s, 3H), 1.39 (s, 3H).

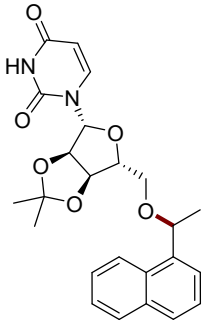
^{13}C NMR (100 MHz, $CDCl_3$, **mixture 1**) δ 174.4, 137.9, 134.0, 130.4, 129.1, 128.4, 126.1, 125.8, 125.4, 123.6, 123.0, 113.1, 81.2, 78.4, 78.1, 75.8, 68.1, 26.8, 25.6, 23.0.
HRMS (ESI) Calcd. for $C_{20}H_{23}O_5$ [$(M+H)^+$] 343.1545, found 343.1553.
^{1}H NMR (400 MHz, $CDCl_3$, **mixture 2**) δ 8.02 (d, *J* = 8.1 Hz, 1H), 7.91 - 7.84 (m, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.54 - 7.42 (m, 4H), 5.22 (q, *J* = 6.5 Hz, 1H), 4.90 (d, *J* = 5.5 Hz, 1H), 4.79 (d, *J* = 5.5 Hz, 1H), 4.60 (t, *J* = 1.9 Hz, 1H), 3.67 (dd, *J* = 10.5, 2.3 Hz, 1H), 3.51 (dd, *J* = 10.5, 1.7 Hz, 1H), 1.60 (d, *J* = 6.5 Hz, 3H), 1.48 (s, 3H), 1.41 (s, 3H).
^{13}C NMR (100 MHz, $CDCl_3$, **mixture 2**) δ 174.4, 137.2, 133.9, 130.5, 129.0, 128.3, 126.2, 125.6, 123.4, 122.8, 113.2, 81.0, 78.5, 76.3, 75.8, 67.2, 26.8, 25.7, 22.9.
HRMS (ESI) Calcd. for $C_{20}H_{23}O_5$ [$(M+H)^+$] 343.1545, found 343.1549.



(3a*R*,6*R*,6a*R*)-5-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyl-6-(1-(naphthalen-1-yl)ethoxy)tetrahydrofuro[2,3-*d*][1,3]dioxole (56):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (3a*R*,6*R*,6a*R*)-5-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-2,2-dimethyltetrahydrofuro[2,3-*d*][1,3]dioxol-6-ol (651 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (20 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (114 mg, 55 % yield).
（1.1:1 dr）
^{1}H NMR (400 MHz, $CDCl_3$, **mixture 1**) δ 8.21 (d, *J* = 8.0 Hz, 1H), 7.92 - 7.85 (m, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.68 (d, *J* = 6.9 Hz, 1H), 7.56 - 7.44 (m, 3H), 5.66 (d, *J* = 3.9 Hz, 1H), 5.45 (q, *J* = 6.5 Hz, 1H), 4.57 (t, *J* = 4.3 Hz, 1H), 4.29 - 4.17 (m, 2H), 3.84 (t, *J* = 7.6 Hz, 1H), 3.78 - 3.71 (m, 1H), 3.60 (dd, *J* = 8.3, 4.7 Hz, 1H), 1.70 - 1.60 (m, 6H), 1.40 (s, 3H), 1.31 (s, 3H), 1.26 (s, 3H).
^{13}C NMR (100 MHz, $CDCl_3$, **mixture 1**) δ 138.3, 133.9, 130.7, 129.0, 128.2, 126.0, 125.6, 124.3, 123.1, 112.9, 109.4, 104.3, 78.4, 77.8, 76.2, 75.1, 74.2, 26.9, 26.1, 25.1, 23.9.
HRMS (ESI) Calcd. for $C_{24}H_{31}O_6$ [$(M+H)^+$] 415.2121 , found 415.2129.
^{1}H NMR (400 MHz, $CDCl_3$, **mixture 2**) δ 8.22 - 8.16 (m, 1H), 7.88 (dd, *J* = 6.7, 2.6 Hz, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.75 (d, *J* = 6.9 Hz, 1H), 7.50 (td, *J* = 7.2, 4.3 Hz, 3H), 5.61 (q, *J* = 6.5 Hz, 1H), 5.49 (d, *J* = 3.5 Hz, 1H), 4.27 (td, *J* = 6.6, 4.5 Hz, 1H), 4.16 (dd, *J* = 8.7, 4.3 Hz, 1H), 4.11 (t, *J* = 3.9 Hz, 1H), 4.08 (dd, *J* = 8.3, 6.3 Hz, 1H), 4.05 - 3.99 (m, 1H), 3.75 (dd, *J* = 8.7, 4.3 Hz, 1H), 1.66 (d, *J* = 6.5 Hz, 3H), 1.60 (s, 3H), 1.56 (s, 3H), 1.41 (s, 3H), 1.24 (s, 3H).
^{13}C NMR (100 MHz, $CDCl_3$, **mixture 2**) δ 139.4, 133.8, 130.8, 128.8, 128.1, 125.9, 125.6, 125.4, 124.0, 123.44, 112.8, 109.7, 103.1, 79.2, 78.7, 78.3, 77.2, 76.4, 75.2, 65.8, 27.0, 26.6, 26.2, 24.9, 23.3.

HRMS (ESI) Calcd. for $C_{24}H_{31}O_6$ [$(M+H)^+$] 415.2121 , found 415.2123.



1-((3a*R*,4*R*,6*R*,6a*R*)-2,2-dimethyl-6-((1-(naphthalen-1-yl)ethoxy)methyl)tetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)pyrimidine-2,4(1*H*,3*H*)-dione (57):

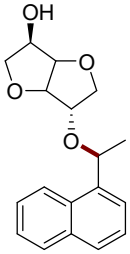
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), 1-((3a*R*,4*R*,6*R*,6a*R*)-6-(hydroxymethyl)-2,2-dimethyltetrahydrofuro[3,4-*d*][1,3]dioxol-4-yl)pyrimidine-2,4(1*H*,3*H*)-dione (711 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a white solid (160 mg, 73 % yield).

（1:1 dr）

^1H NMR (400 MHz, $CDCl_3$, **mixture**) δ 9.26 (9.11) (s, 1H), 8.22 - 8.03 (m, 1H), 7.92 - 7.83 (m, 1H), 7.81 - 7.77 (m, 1H), 7.73 - 7.39 (m, 5H), 5.94 (5.90) (d, *J* = 2.6 Hz, 1H), 5.71 - 5.68 (5.27 - 5.23) (m, 1H), 5.20 - 5.10 (m, 1H), 4.87 - 4.68 (m, 2H), 4.43 - 4.34 (m, 1H), 3.81 - 3.48 (m, 2H), 1.67 - 1.63 (m, 3H), 1.58 (1.57) (s, 3H), 1.35 (1.32) (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 163.3, 163.1, 150.1, 150.0, 141.1, 140.8, 138.1, 137.9, 134.0, 133.9, 130.5, 129.1, 128.4, 128.3, 126.1, 125.9, 125.7, 125.4, 125.3, 123.9, 123.3, 123.2, 123.0, 114.3, 114.0, 102.0, 93.1, 92.0, 85.9, 85.6, 85.2, 84.8, 81.1, 80.8, 77.2, 76.3, 68.9, 68.5, 27.2, 25.4, 25.3, 22.9, 22.1.

HRMS (ESI) Calcd. for $C_{24}H_{27}N_2O_6$ [$(M+H)^+$] 439.1869, found 439.1877.



(3*R*,6*S*)-6-(1-(naphthalen-1-yl)ethoxy)hexahydrofuro[3,2-*b*]furan-3-ol (58):

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), isosorbide (365 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash

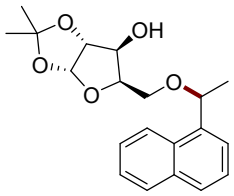
chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (107 mg, 71 % yield).

（1.1:1 dr）

^{1}H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.17 - 8.13 (m, 1H), 7.92 - 7.83 (m, 1H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.62 - 7.37 (m, 4H), 5.33 - 5.19 (m, 1H), 4.71 - 4.69 (4.62 - 4.59) (m, 1H), 4.62 - 4.59 (4.39 - 4.37) (m, 1H), 4.31 - 4.20 (m, 1H), 4.20 - 4.17 (4.09 - 4.06) (m, 1H), 4.01 - 3.94 (m, 1H), 3.86 - 3.70 (m, 2H), 3.49 - 3.40 (m, 1H), 2.62 (br, 1H), 1.66 - 1.58 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 138.7, 134.0, 130.7, 130.5, 129.0, 128.3, 128.2, 126.1, 125.6, 125.5, 123.9, 123.8, 123.4, 123.3, 86.6, 86.2, 82.1, 81.8, 75.3, 74.8, 74.2, 73.5, 73.4, 73.3, 72.3, 72.2, 23.7.

HRMS (ESI) Calcd. for $C_{18}H_{21}O_4$ [$(M+H)^+$] 301.1440, found 301.1444.



(3a*R*,5*R*,6*S*,6a*R*)-2,2-dimethyl-5-((1-(naphthalen-1-yl)ethoxy)methyl)tetrahydrofuro[2,3-*d*][1,3]dioxol-6-ol (59):

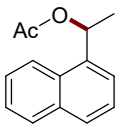
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), (3a*R*,5*R*,6*S*,6a*R*)-5-(hydroxymethyl)-2,2-dimethyltetrahydrofuro[2,3-*d*][1,3]dioxol-6-ol (475 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (50 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (132 mg, 77 % yield).

（1.13:1 dr）

^{1}H NMR (400 MHz, $CDCl_3$, **mixture**) δ 8.18 - 8.07 (m, 1H), 7.88 (7.86) (s, H), 7.80 - 7.78 (m, 1H), 7.62 - 7.41 (m, 4H), 6.01 (5.95) (d, *J* = 3.7 Hz, 1H), 5.35 (5.24) (q, *J* = 6.5 Hz, 1H), 4.54 (4.49) (d, *J* = 3.7 Hz, 1H), 4.33 - 4.27 (m, 1H), 4.24 (4.16) (dd, *J* = 6.8, 3.5 Hz, 1H), 4.04 (3.49) (br, 1H), 3.93 - 3.76 (m, 2H), 1.66 - 1.63 (m, 3H), 1.47 (1.45) (s, 3H), 1.32 (1.31) (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$, **mixture**) δ 138.1, 137.9, 134.0, 130.7, 130.6, 129.1, 129.0, 128.2, 126.2, 126.1, 125.7, 125.6, 123.6, 123.5, 123.1, 123.0, 111.6, 104.9, 85.4, 78.1, 77.3, 77.0, 76.5, 67.0, 66.8, 26.8, 26.7, 26.2, 23.2.

HRMS (ESI) Calcd. for $C_{20}H_{25}O_5$ [$(M+H)^+$] 345.1702, found 345.1707.



1-(naphthalen-1-yl)ethyl acetate:

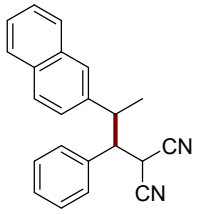
According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), acetic acid (150 mg, 2.5 mmol, 5.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg,

0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (5 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (80 mg, 75 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.3 Hz, 1H), 7.87 (d, *J* = 7.7 Hz, 1H), 7.80 (d, *J* = 8.2 Hz, 1H), 7.60 (d, *J* = 7.1 Hz, 1H), 7.56 - 7.42 (m, 3H), 6.65 (q, *J* = 6.6 Hz, 1H), 2.12 (s, 3H), 1.70 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 137.4, 133.8, 130.2, 128.9, 128.4, 126.3, 125.6, 125.3, 123.2, 123.1, 69.4, 21.7, 21.3.

HRMS (ESI) Calcd. for $C_{14}H_{15}O_2$ [$(M+H)^+$] 215.1072, found 215.1074.



2-(2-(naphthalen-2-yl)-1-phenylpropyl)malononitrile:

According to the general procedure, 1-ethylnaphthalene (78 mg, 0.5 mmol, 1.0 equiv.), *d*^{4}-MeOH (90 mg, 2.5 mmol, 5.0 equiv.), 2-benzylidenemalononitrile (154 mg, 1.0 mmol, 2.0 equiv.), Selectfluor (354 mg, 1.0 mmol, 2.0 equiv.), $Ni(acac)_2$ (12.9 mg, 0.05 mmol, 10 mol%), bpy (7.8 mg, 0.05 mmol, 10 mol%), $[Ir\{dF(CF_3)ppy\}_2(dtbbpy)]PF_6$ (5.6 mg, 0.005 mmol, 1 mol%) and 2.5 mL CH_3CN were used. After 24 hours, the reaction mixture was subjected to the workup protocol outlined in the general procedure and purified by flash chromatography (10 % ethyl acetate/petroleum ether) to provide the title compound as a colourless oil (20 mg, 13 % yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.6 Hz, 1H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.47 (t, *J* = 7.2 Hz, 1H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.23 (t, *J* = 7.7 Hz, 1H), 7.12 (d, *J* = 6.0 Hz, 3H), 7.06 (dd, *J* = 10.5, 5.2 Hz, 3H), 4.49 - 4.30 (m, 1H), 4.24 (d, *J* = 4.9 Hz, 1H), 3.76 - 3.61 (m, 1H), 1.53 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 136.1, 133.9, 131.0, 129.1, 128.6, 128.5, 128.2, 127.4, 126.4, 125.6, 125.1, 122.2, 112.2, 111.9, 51.9, 29.7, 28.0, 20.9.

HRMS (ESI) Calcd. for $C_{22}H_{19}N_2$ [$(M+H)^+$] 311.1543, found 311.1551.

4. References

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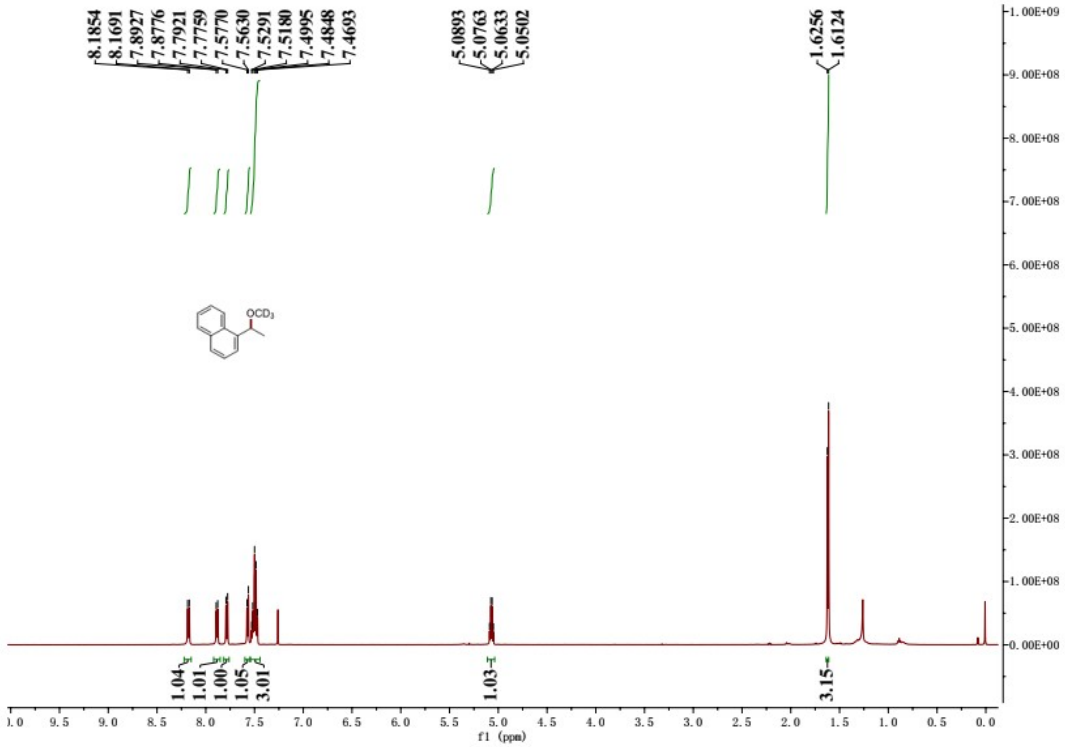
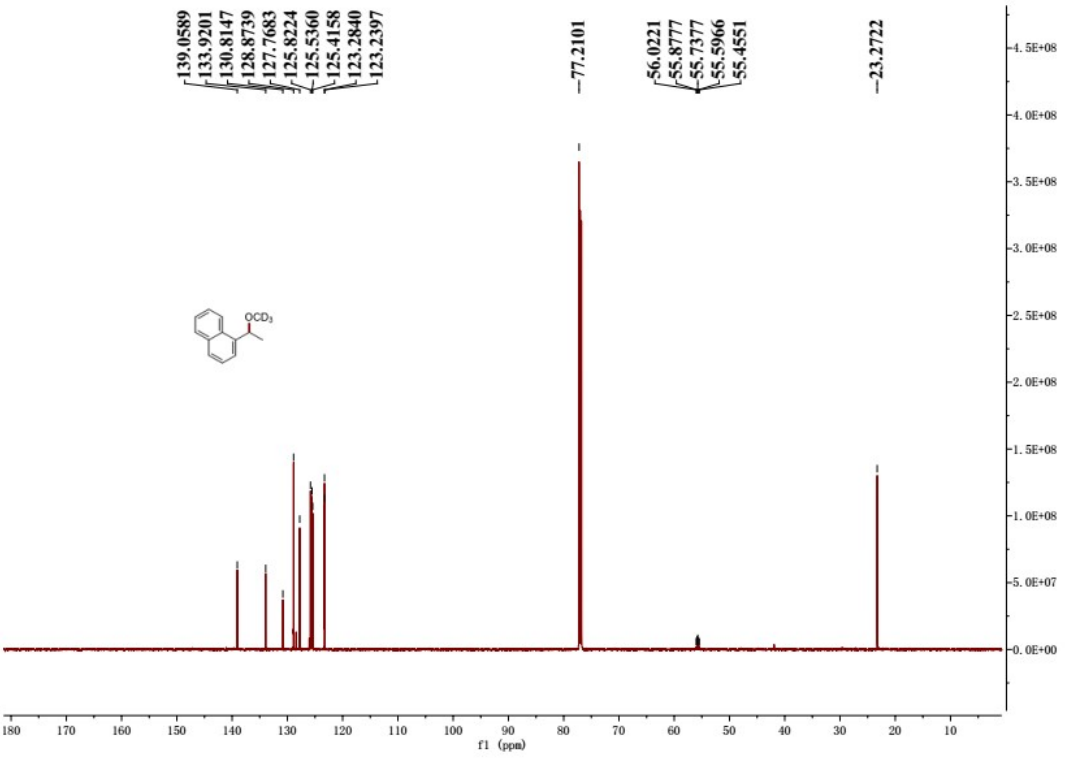
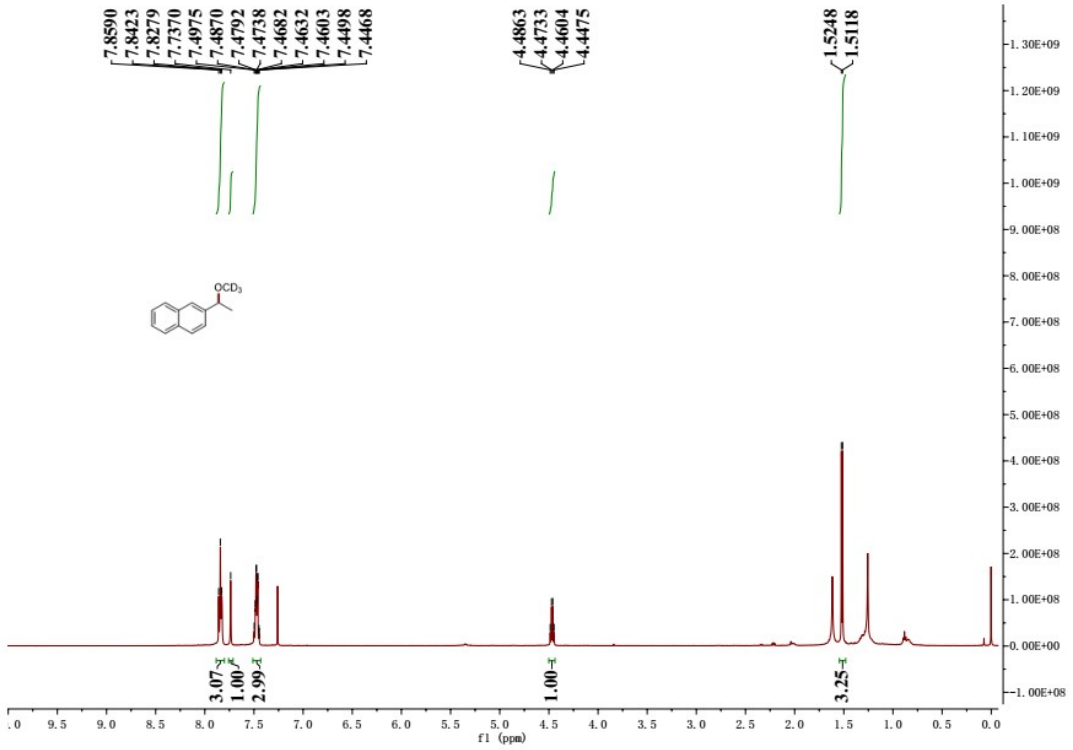
5. Spectral Data for Products



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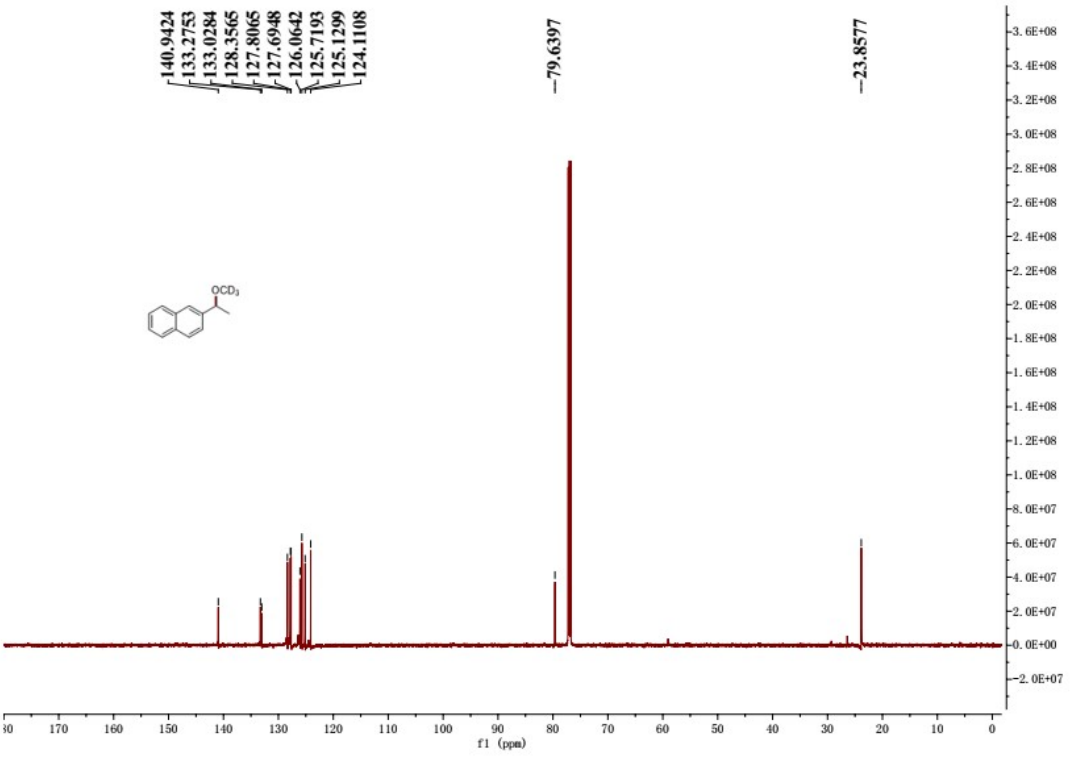
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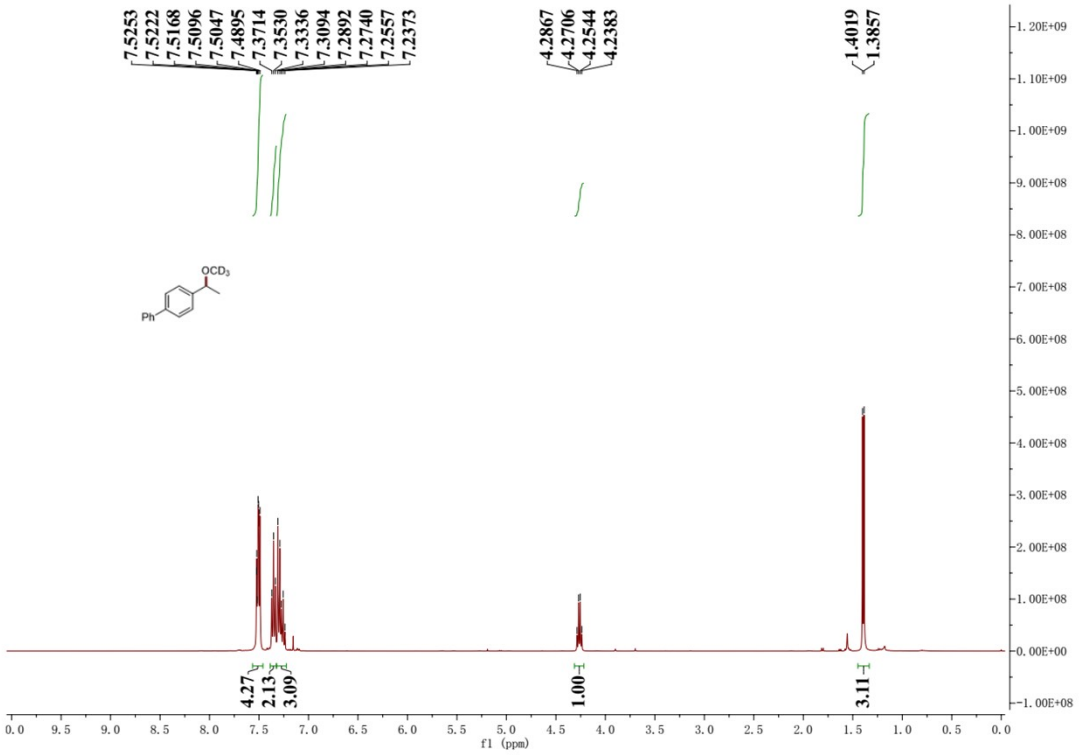
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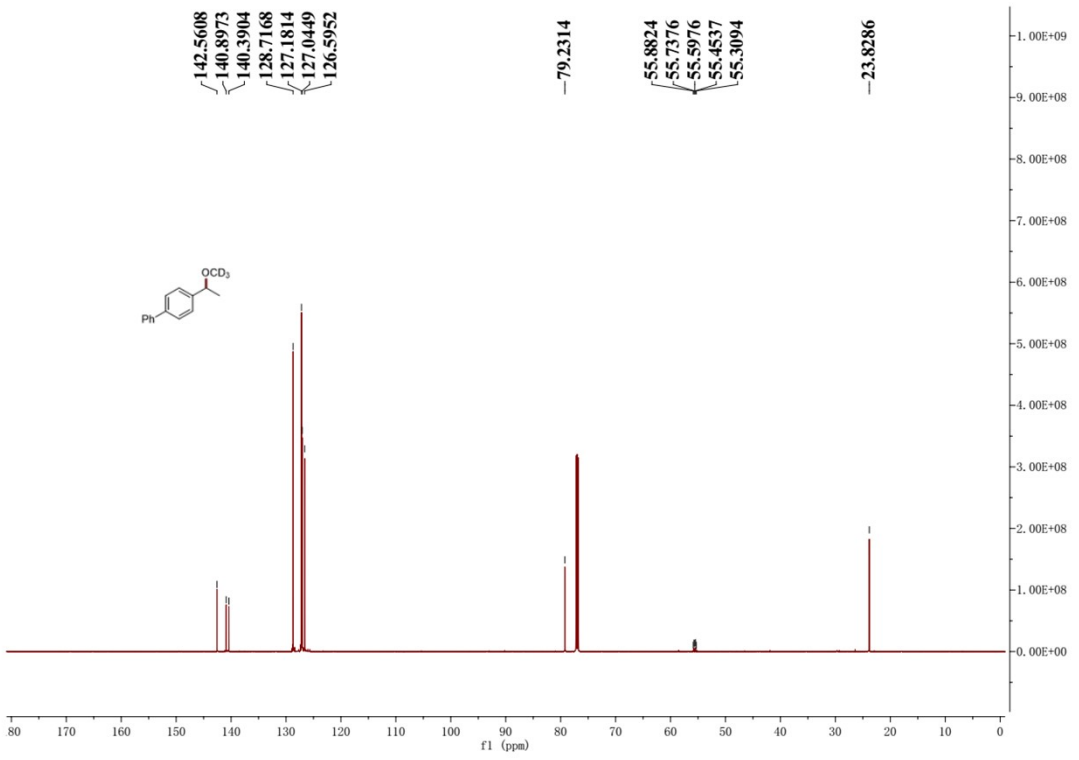
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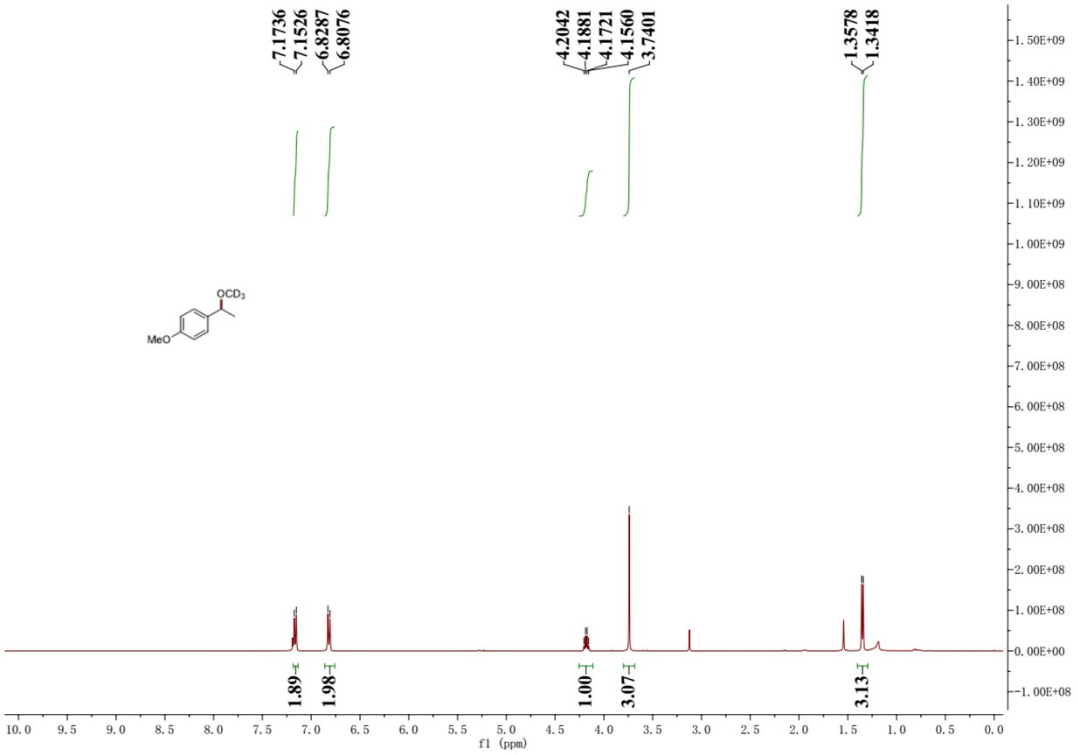
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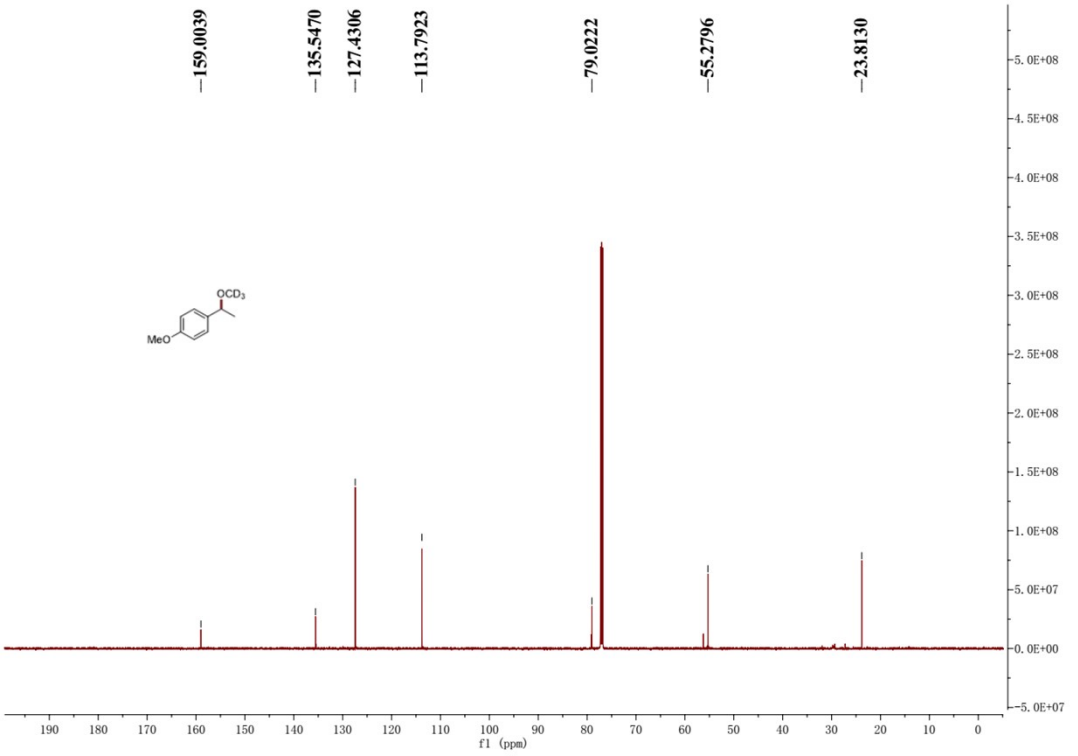
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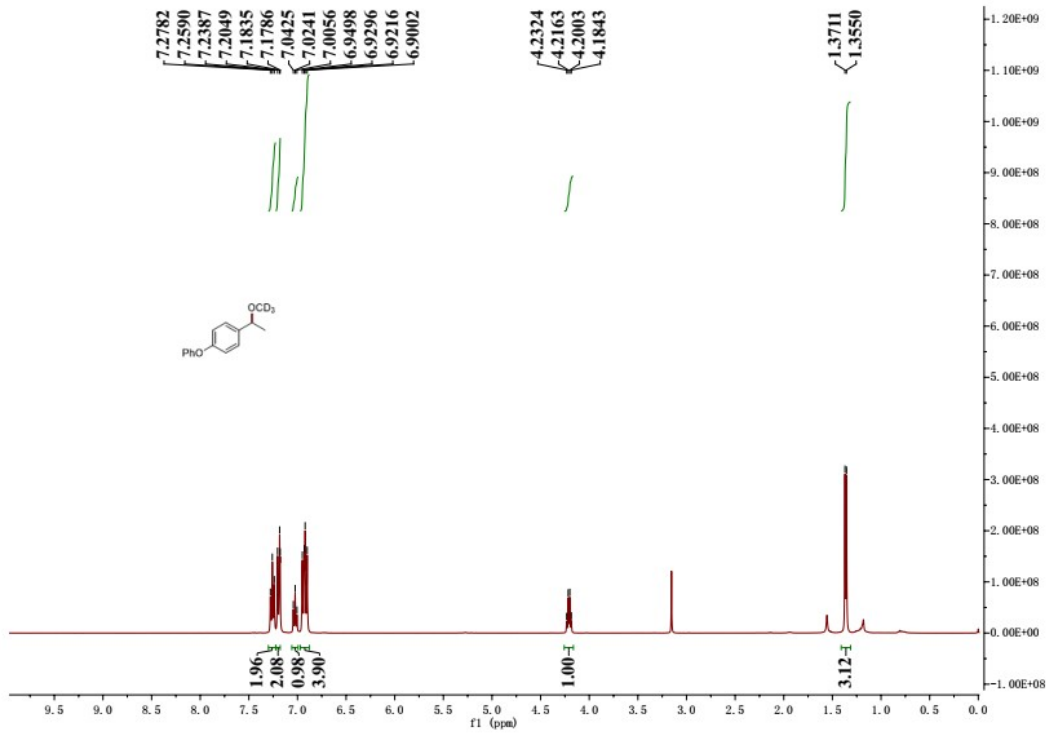
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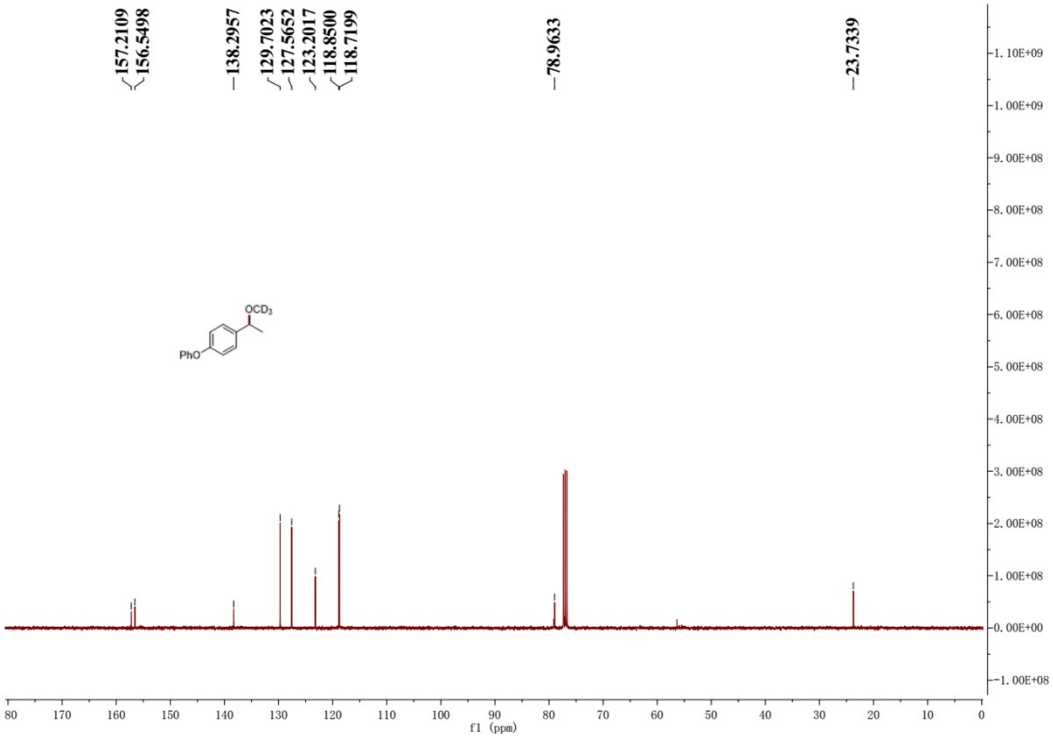
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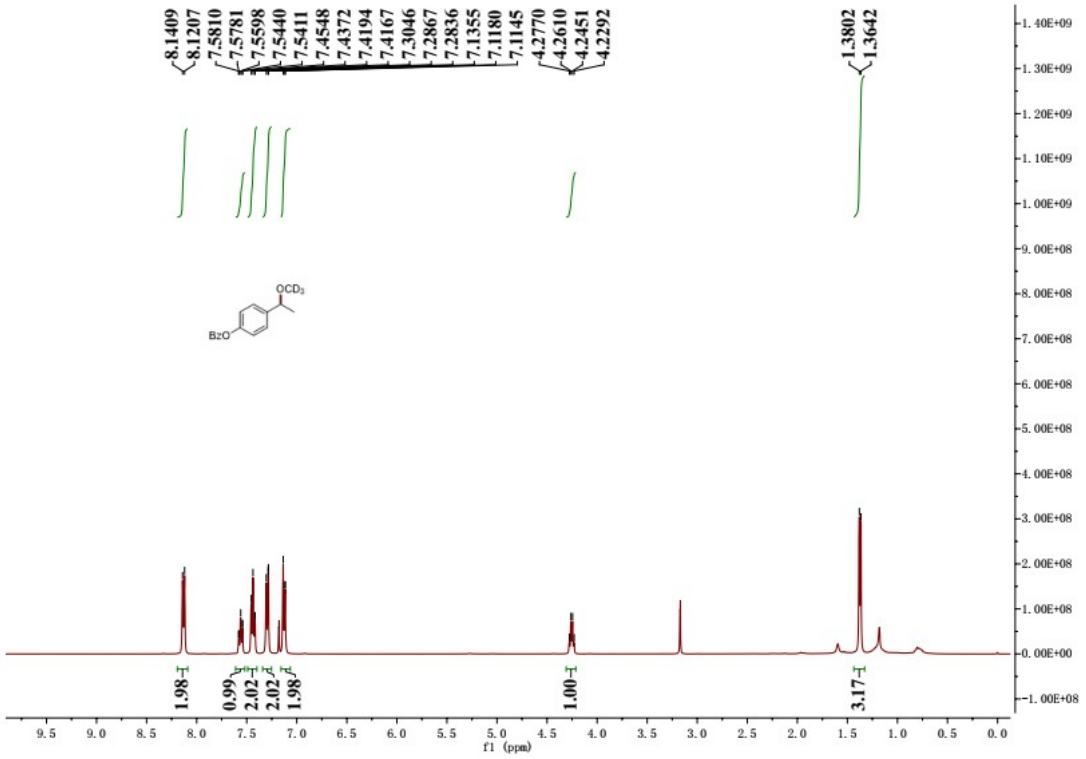
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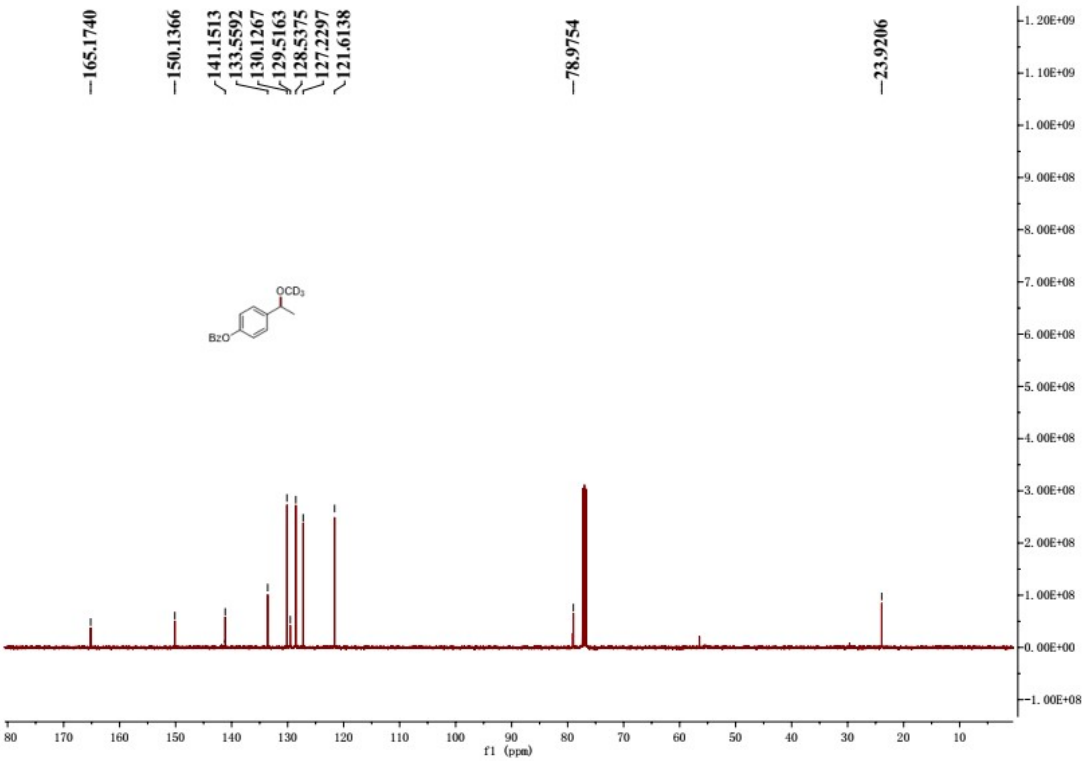
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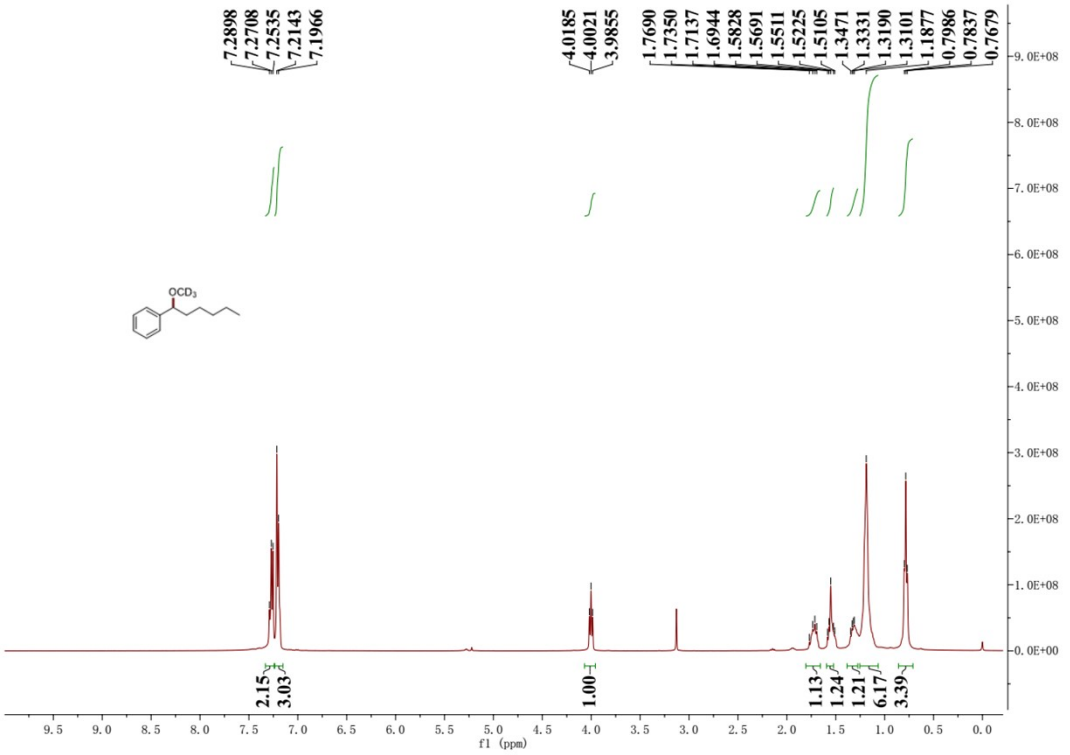
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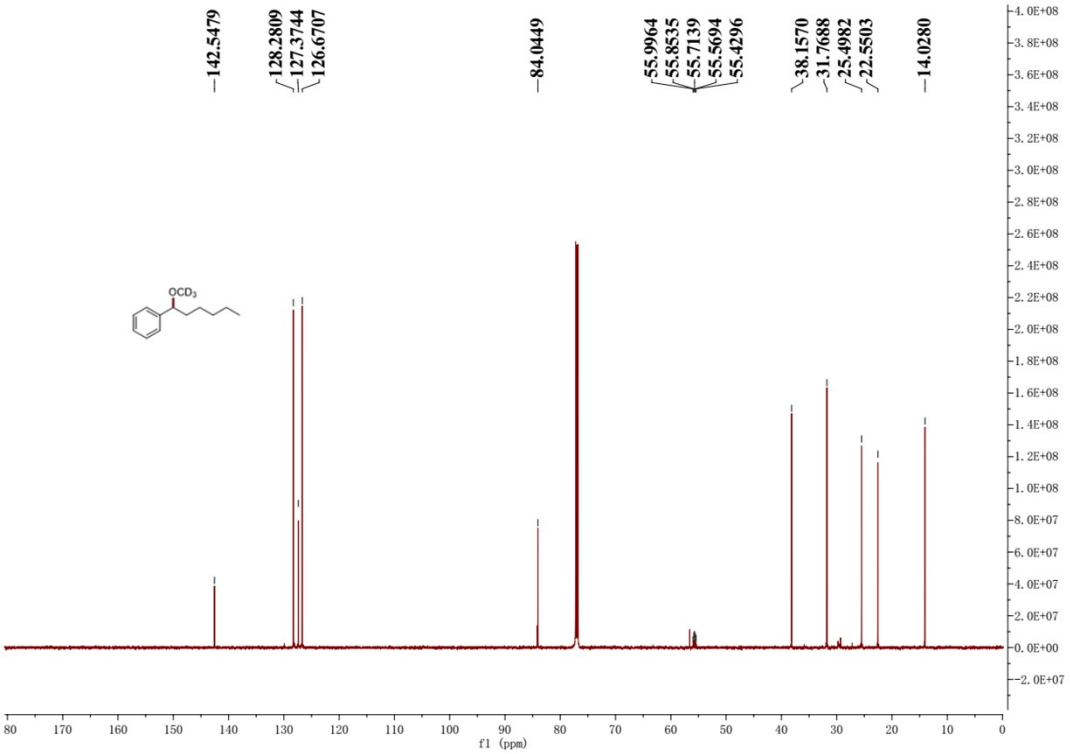
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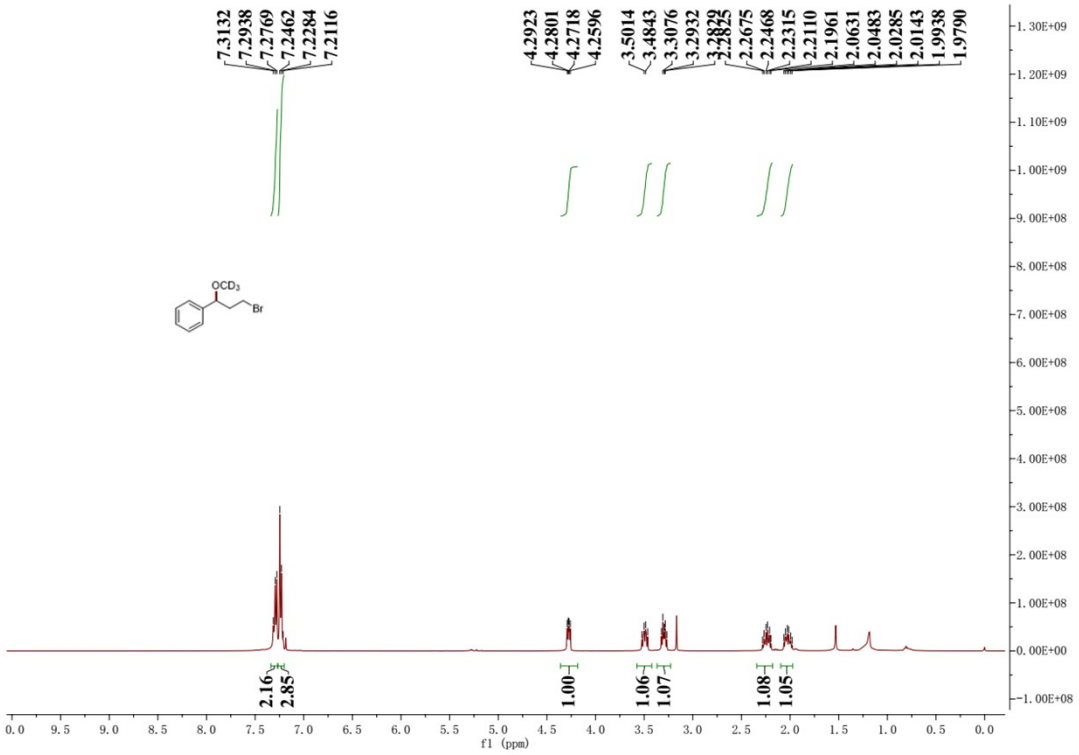
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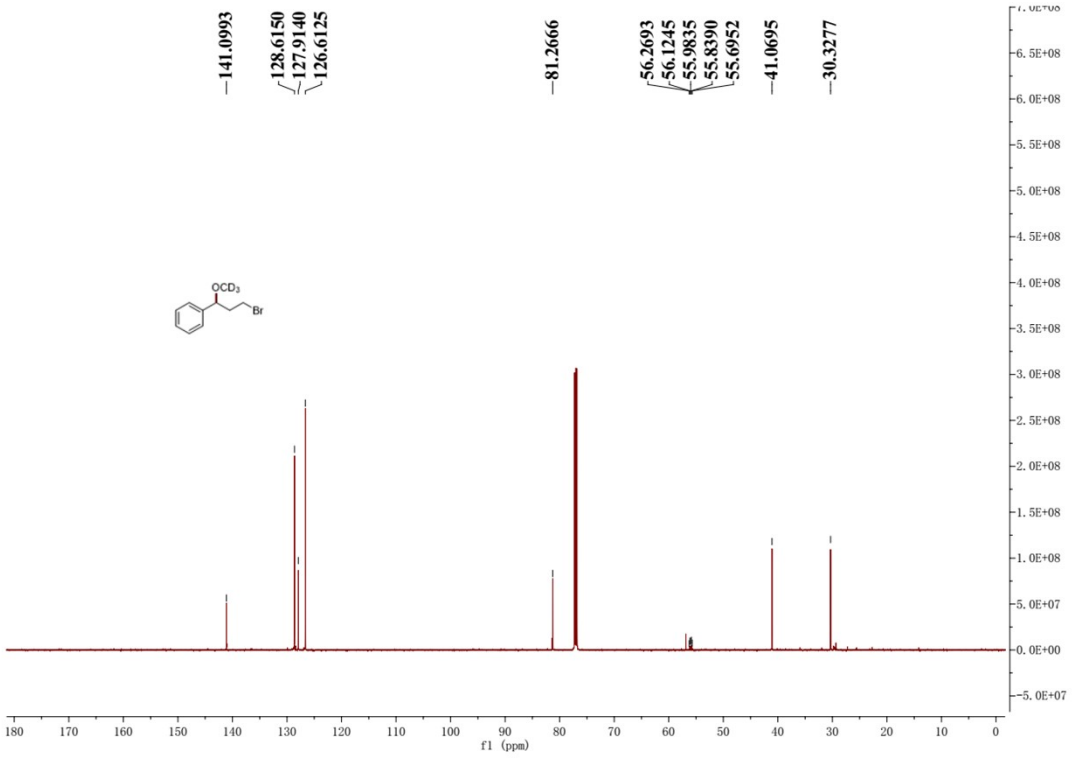
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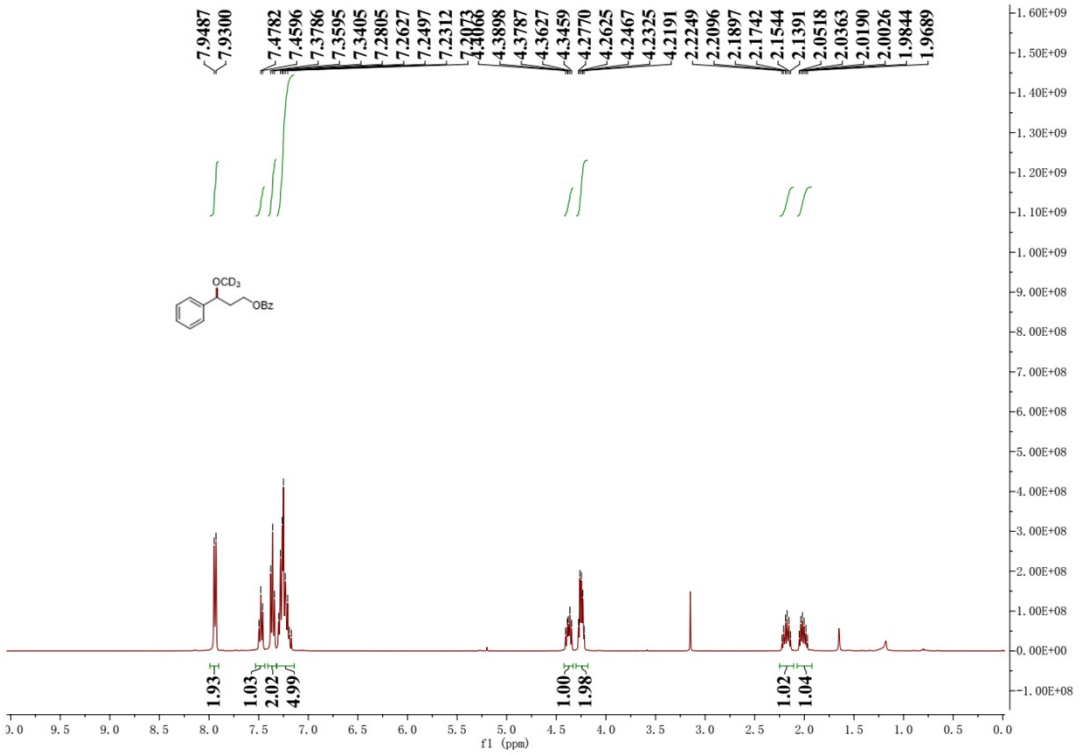
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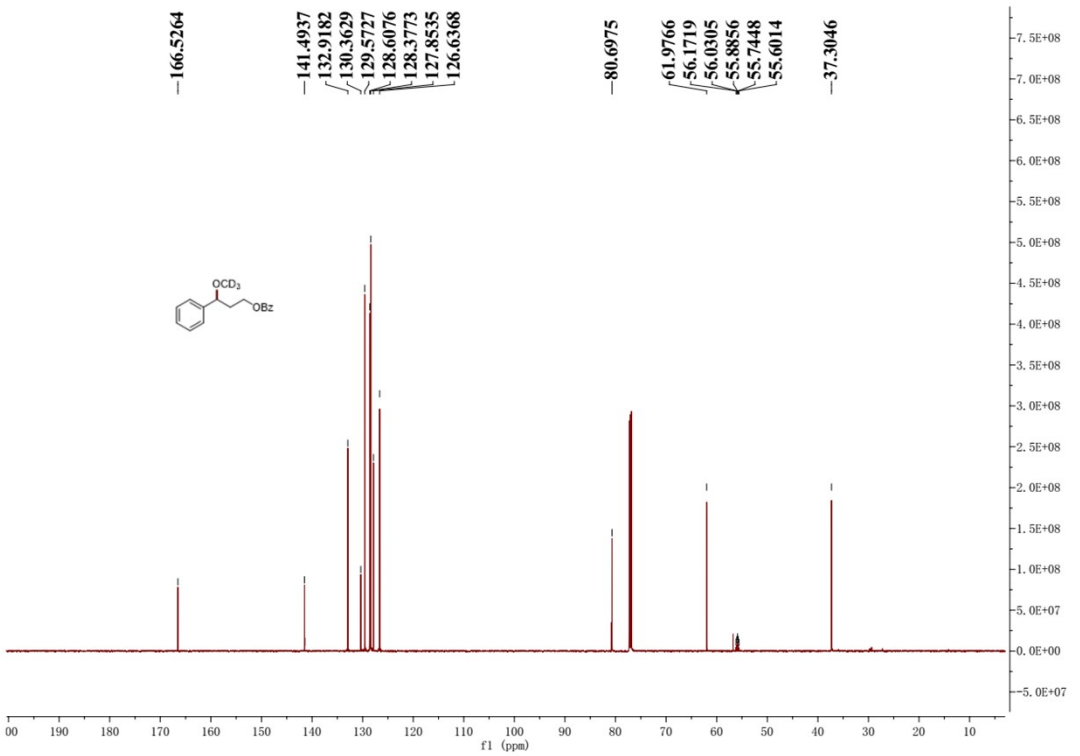
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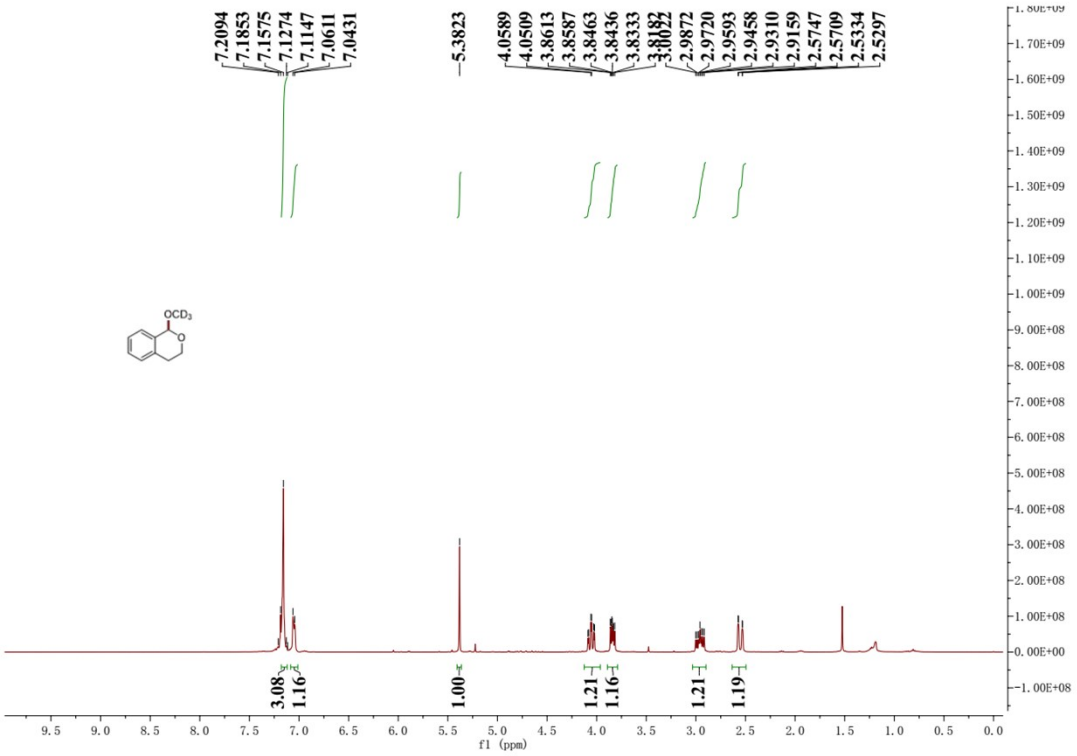
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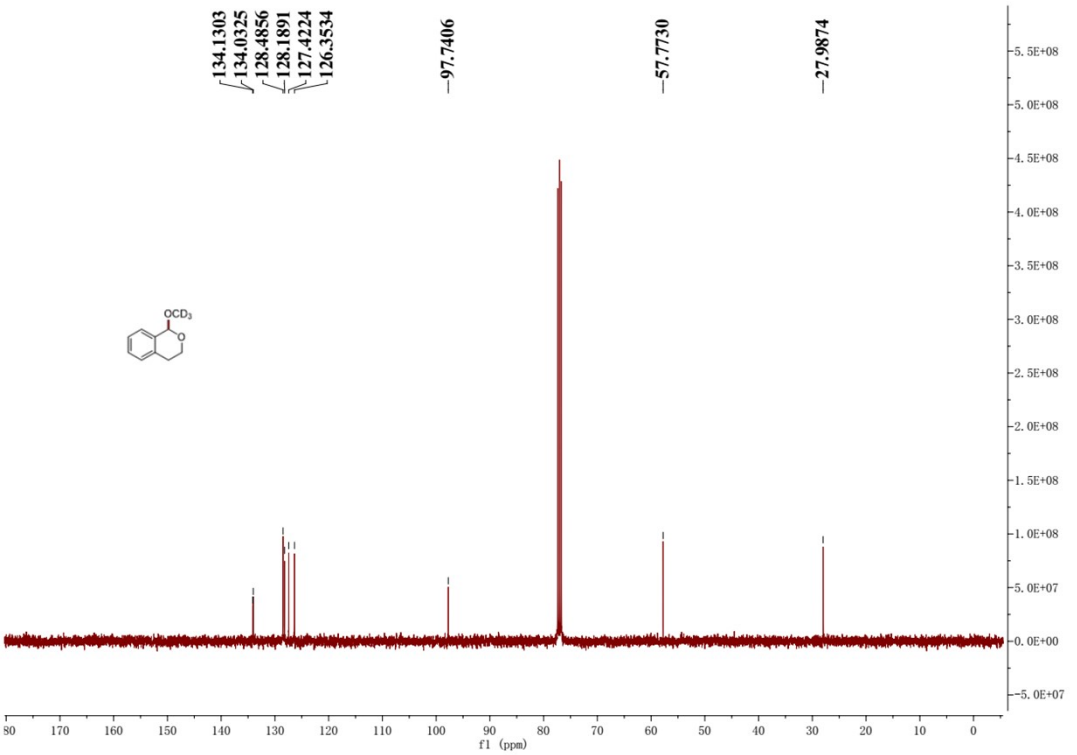
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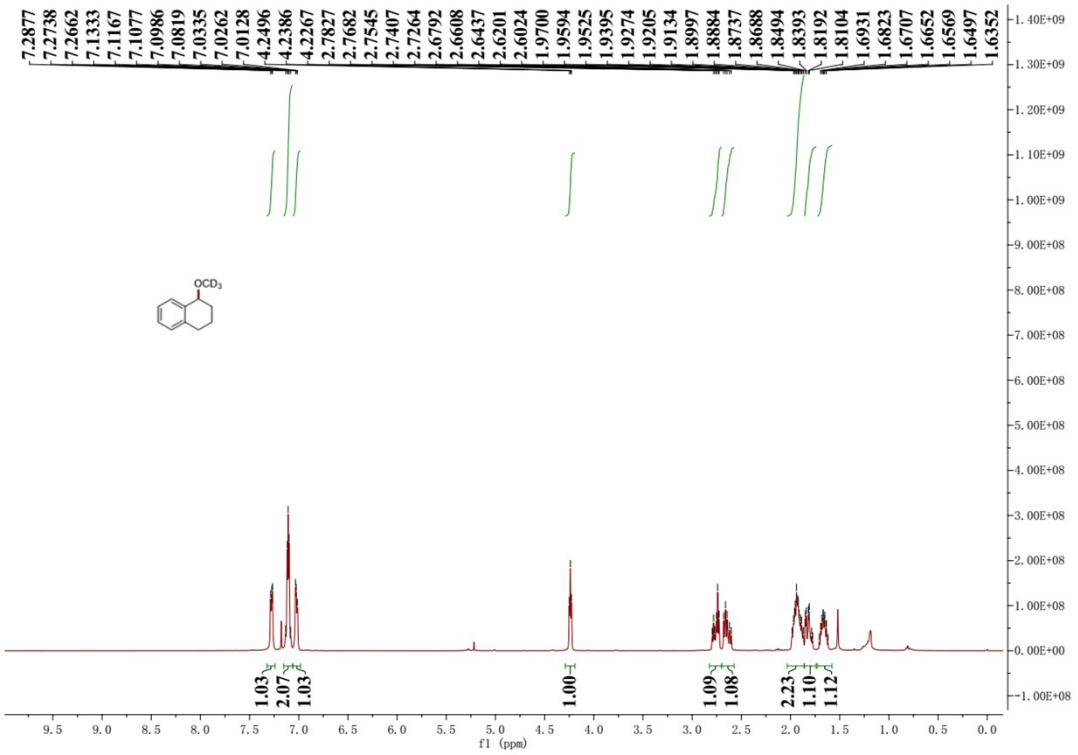
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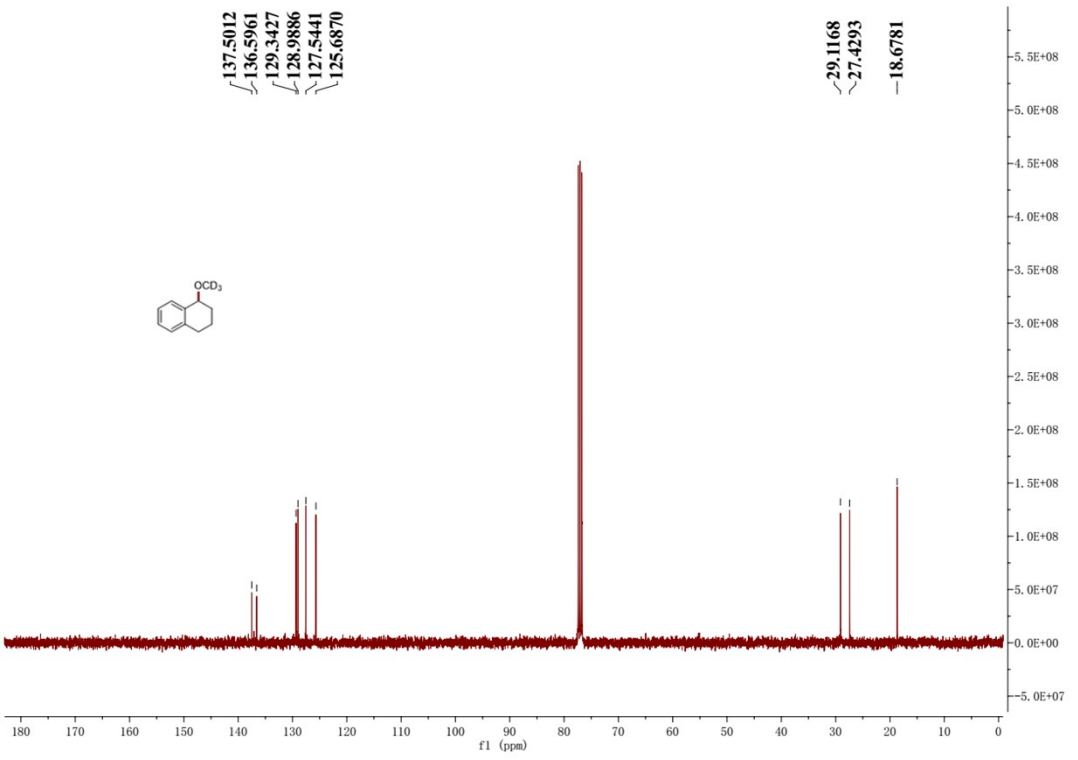
1.12
f1 (ppm)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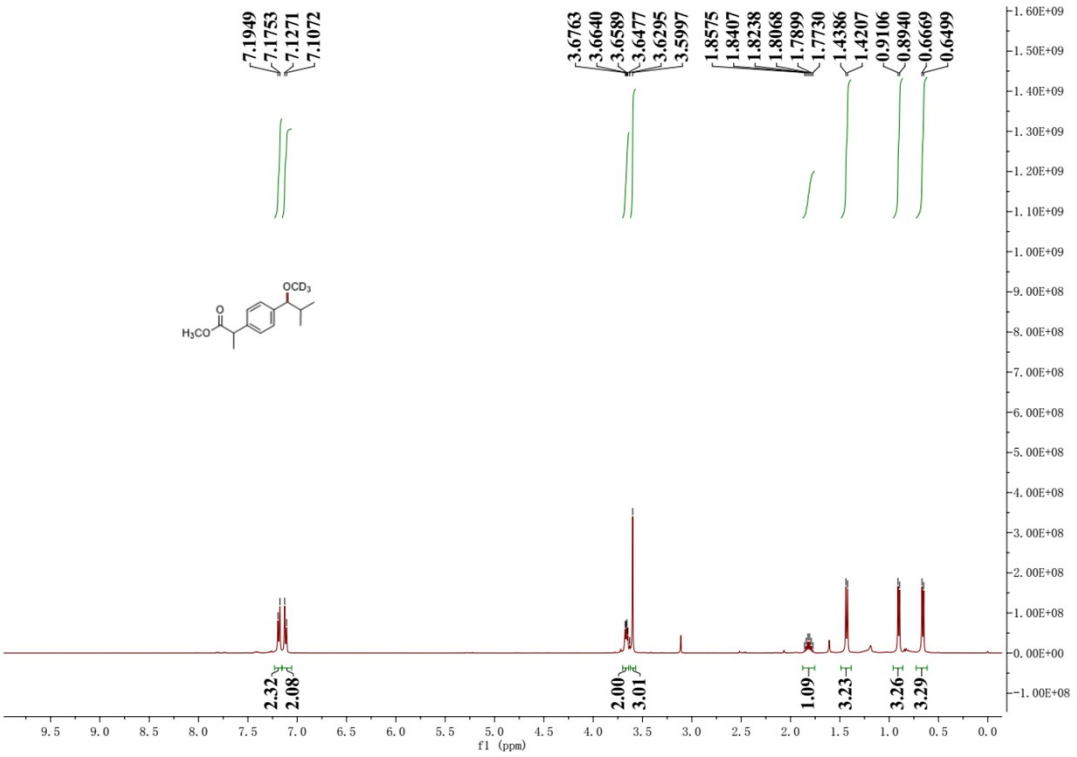
127.5441
125.6870
29.1168
27.4293
18.6781
OCD3
f1 (ppm)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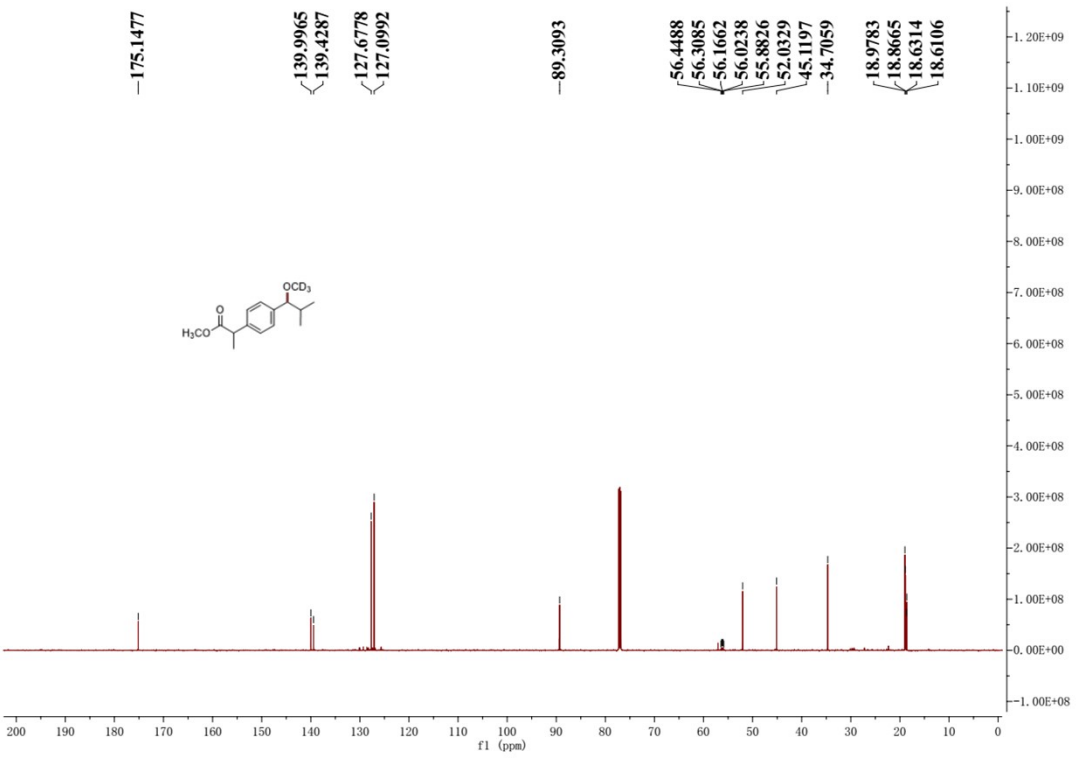
7.1753
7.1271
7.1072
3.6763
3.6640
3.6589
3.6477
3.6295
3.5997
1.8575
1.8407
1.8238
1.8068
1.7899
1.7730
1.4386
1.4207
0.9106
0.8940
0.6669
0.6499
OCD3
H3CO
2.32
2.08
2.00
3.01
1.09
3.23
3.26
3.29
f1 (ppm)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139.4287
127.6778
127.0992
89.3093
56.4488
56.3085
56.1662
56.0238
55.8826
52.0329
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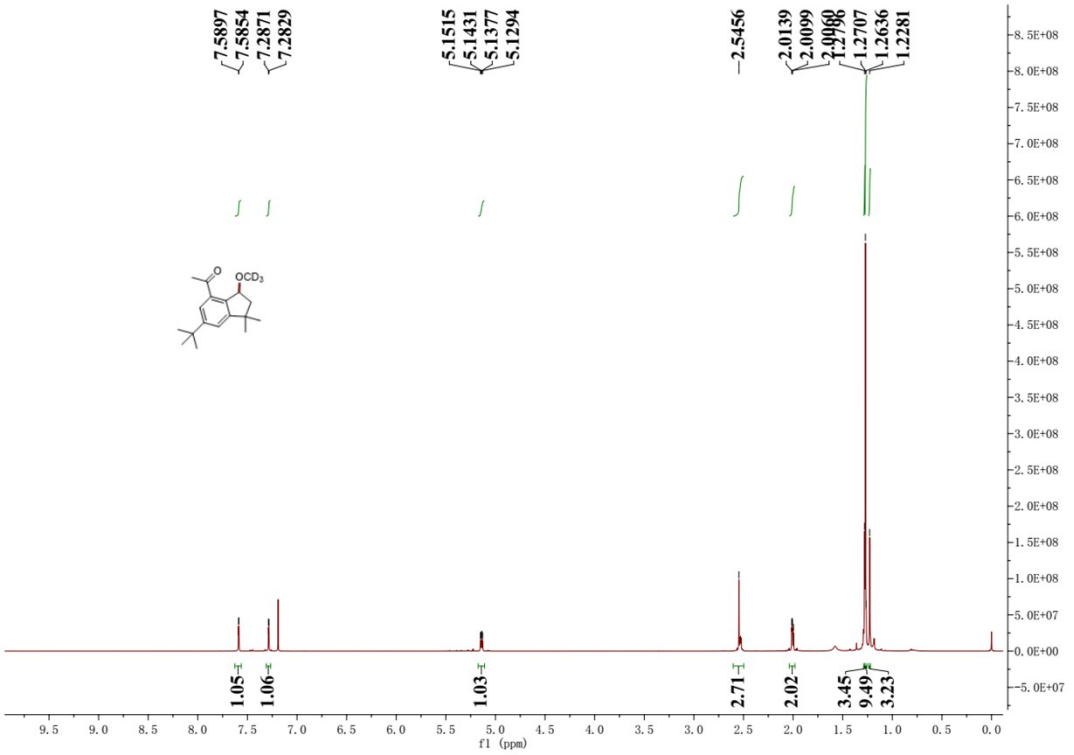
34.7059
18.9783
18.8665
18.6314
18.6106
OCD3
H3CO
f1 (ppm)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7.2871
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5.1431
5.1377
5.1294
2.5456
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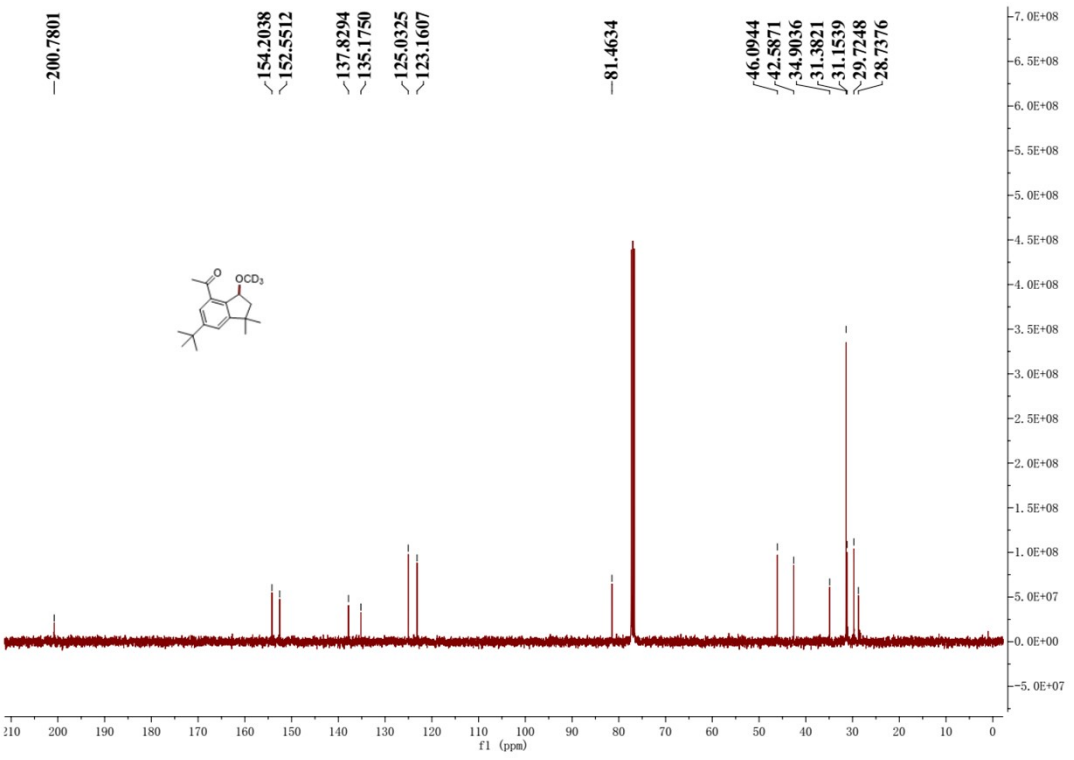
2.0099
1.2707
1.2636
1.2281
OCD3
1.05
1.06
1.03
2.71
2.02
3.45
9.49
3.23
f1 (ppm)


200.7801
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152.5512
137.8294
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125.0325
123.1607
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42.5871
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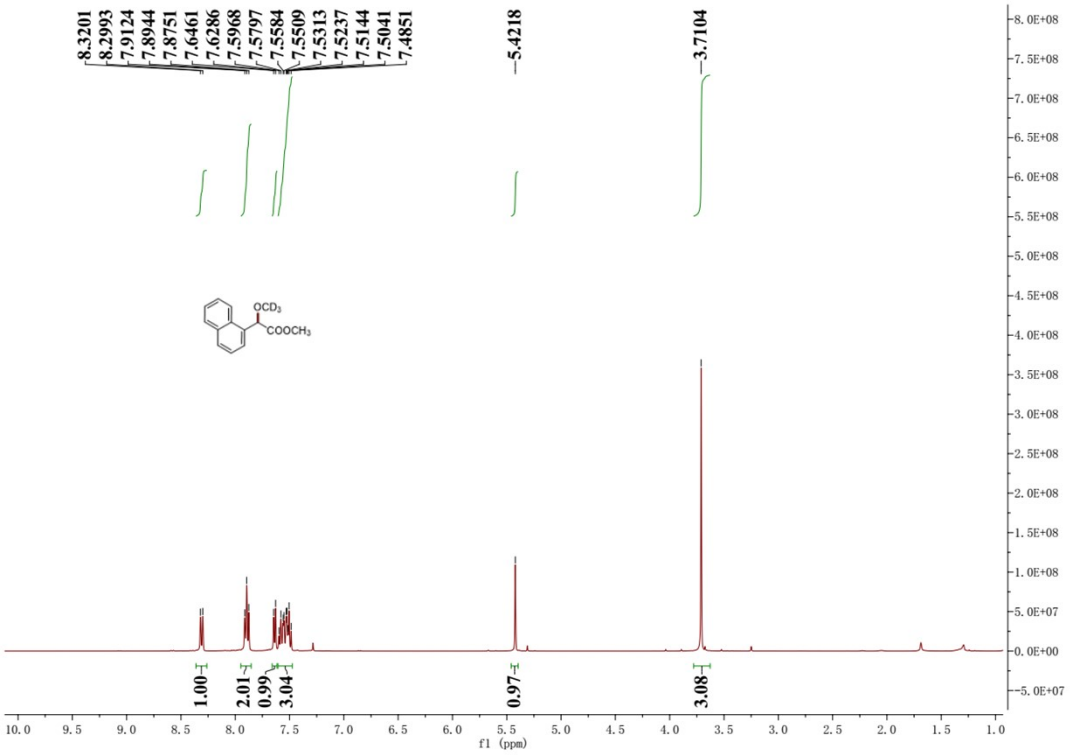
31.3821
31.1539
29.7248
28.7376
OCD3
f1 (ppm)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8.2993
7.9124
7.8944
7.8751
7.6461
7.6286
7.5968
7.5797
7.5584
7.5509
7.5313
7.5237
7.5144
7.5041
7.4851
5.4218
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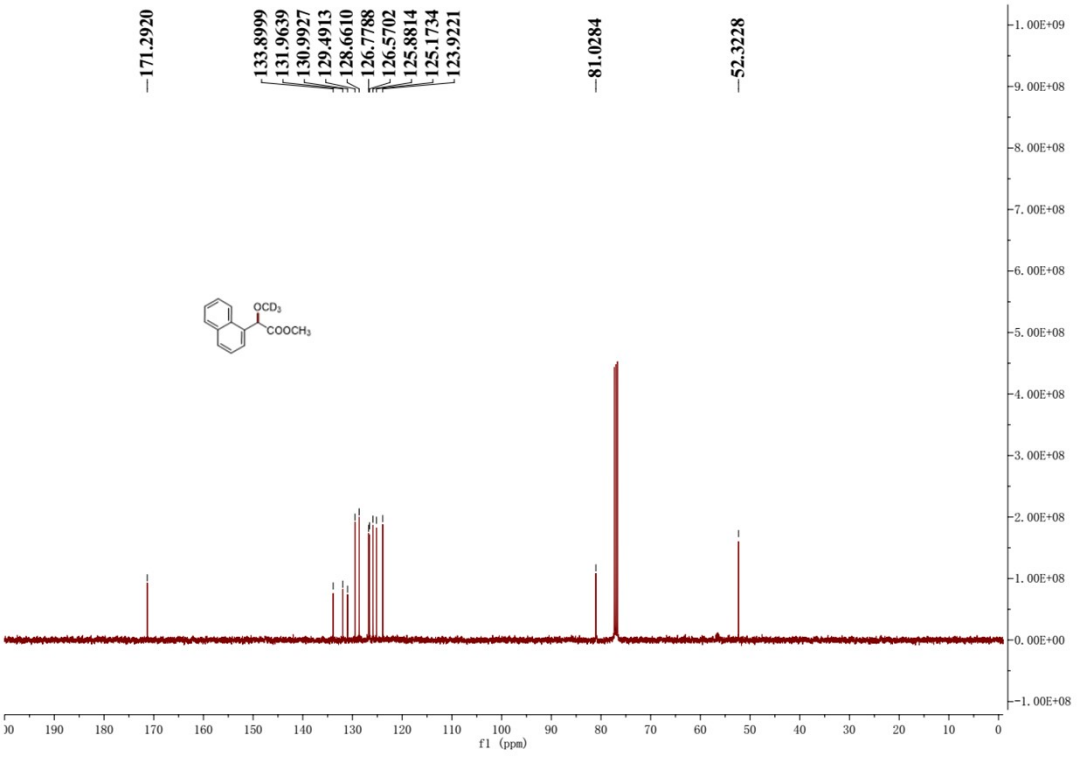
OCD3
COOCH3
1.00
2.01
0.99
3.04
0.97
3.08
f1 (ppm)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130.9927
129.4913
128.6610
126.7788
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123.9221
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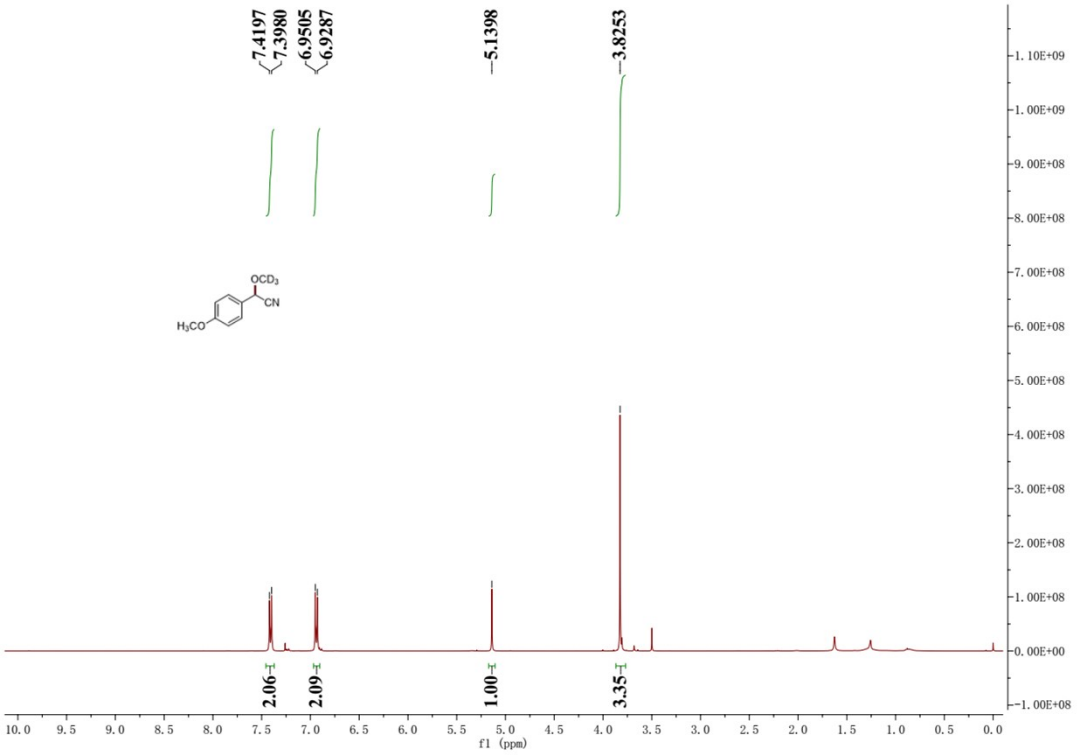
52.3228
OCD3
COOCH3
f1 (ppm)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6.9505
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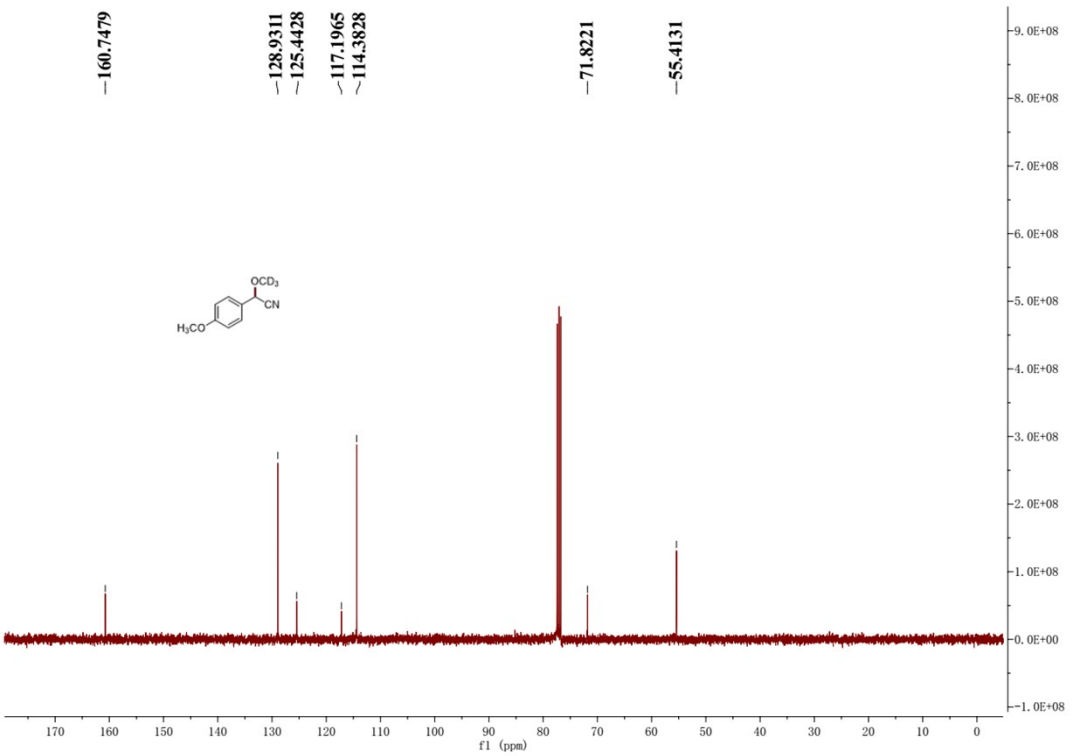
OCD3
CN
H3CO
2.06
2.09
1.00
3.35
f1 (ppm)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128.9311
125.4428
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114.3828
71.8221
55.4131
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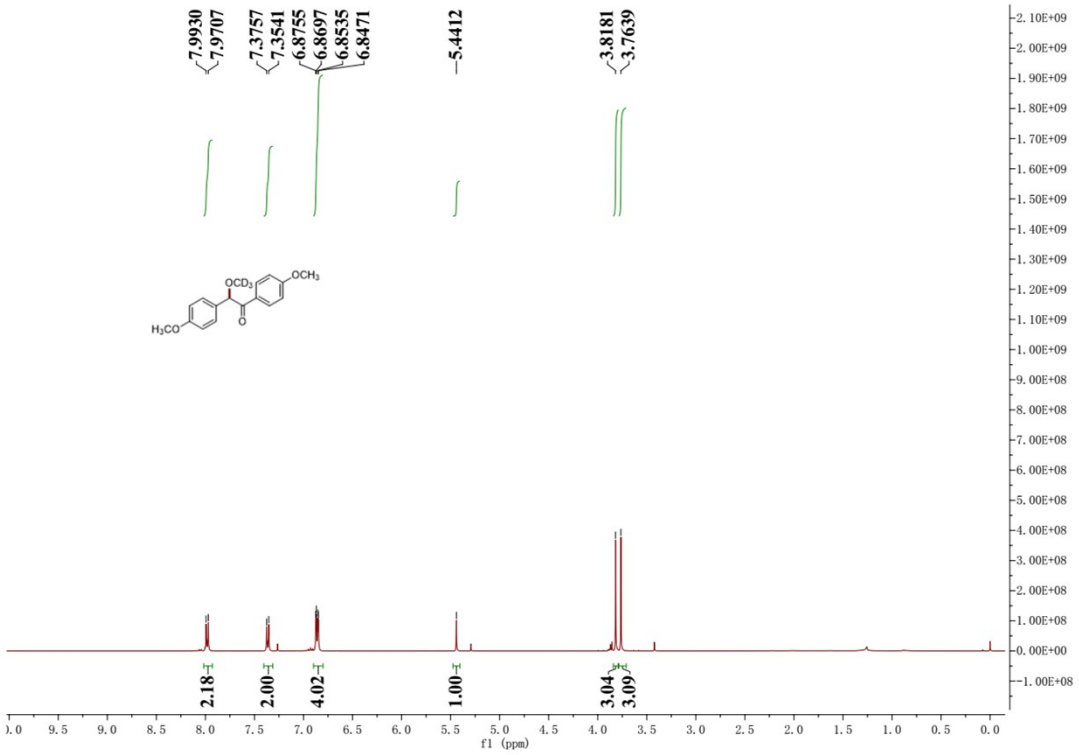
CN
H3CO
f1 (ppm)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7.3757
7.3541
6.8755
6.8697
6.8535
6.8471
5.4412
3.8181
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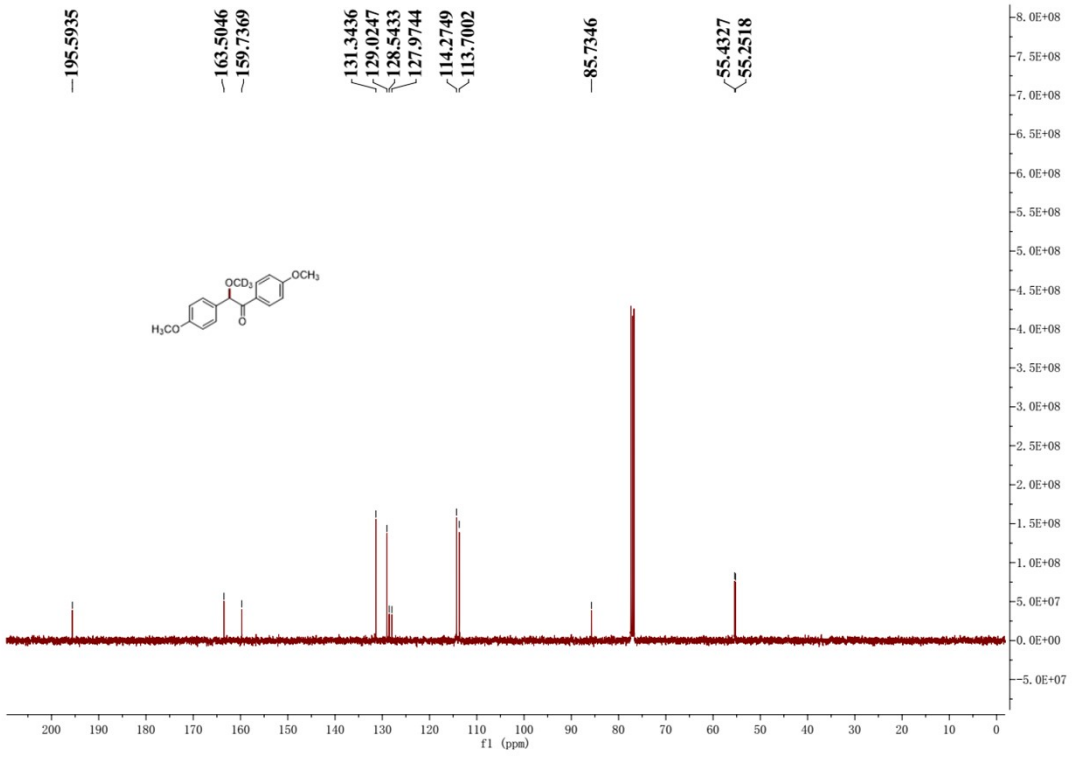
OCD3
OCH3
H3CO
O
2.18
2.00
4.02
1.00
3.04
3.09
f1 (ppm)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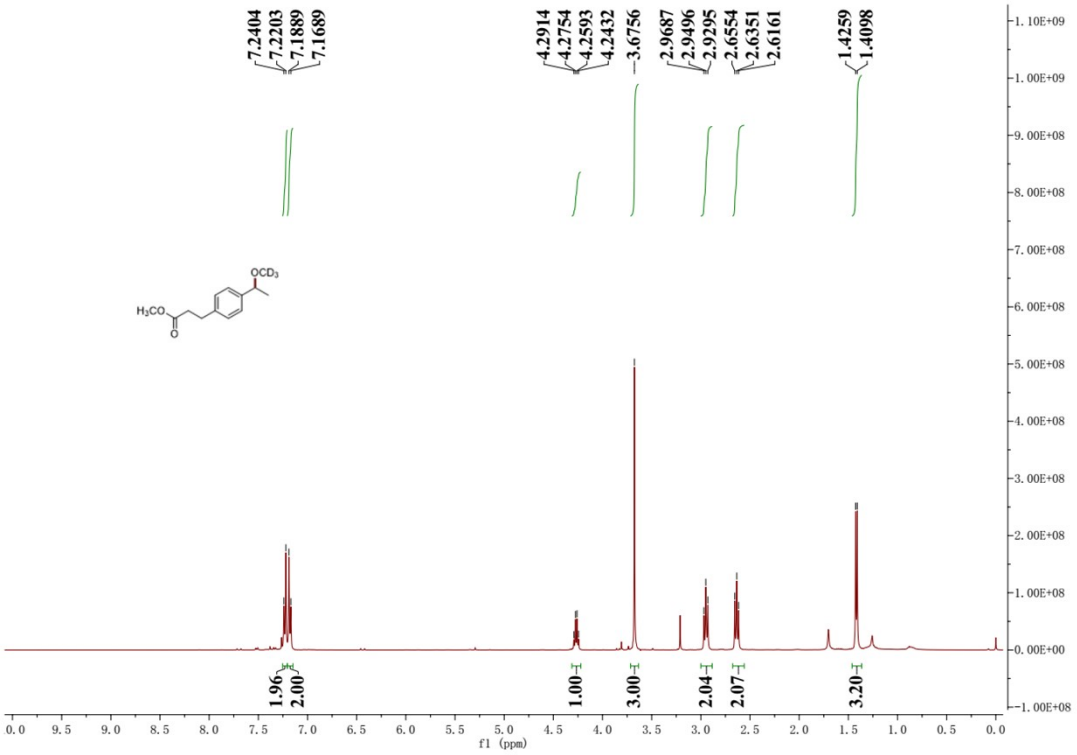
163.5046
159.7369
131.3436
129.0247
128.5433
127.9744
114.2749
113.7002
85.7346
55.4327
55.2518
OCD3
OCH3
H3CO
O
f1 (ppm)


7.2404
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7.1889
7.1689
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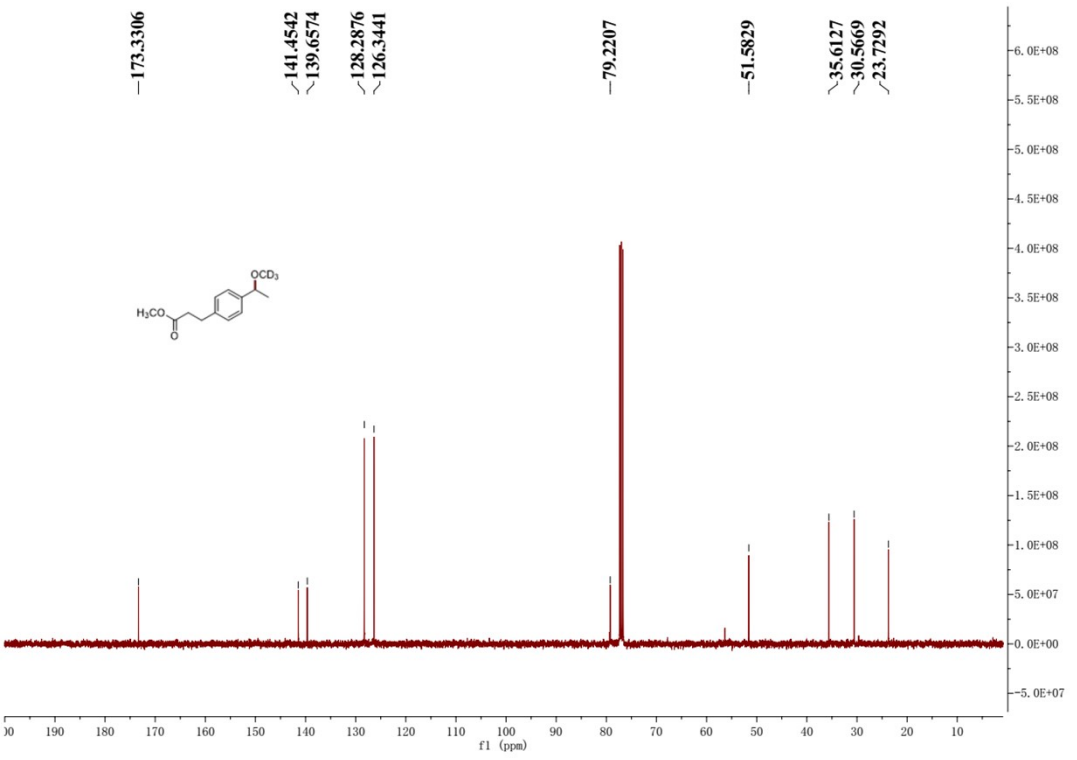
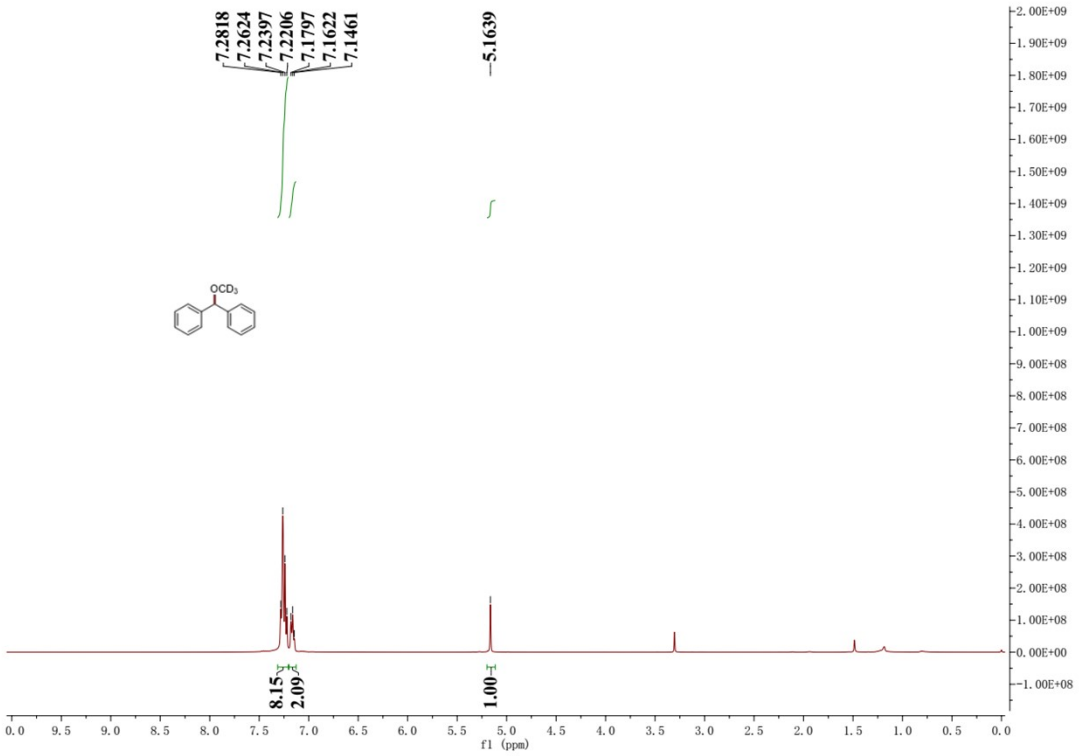
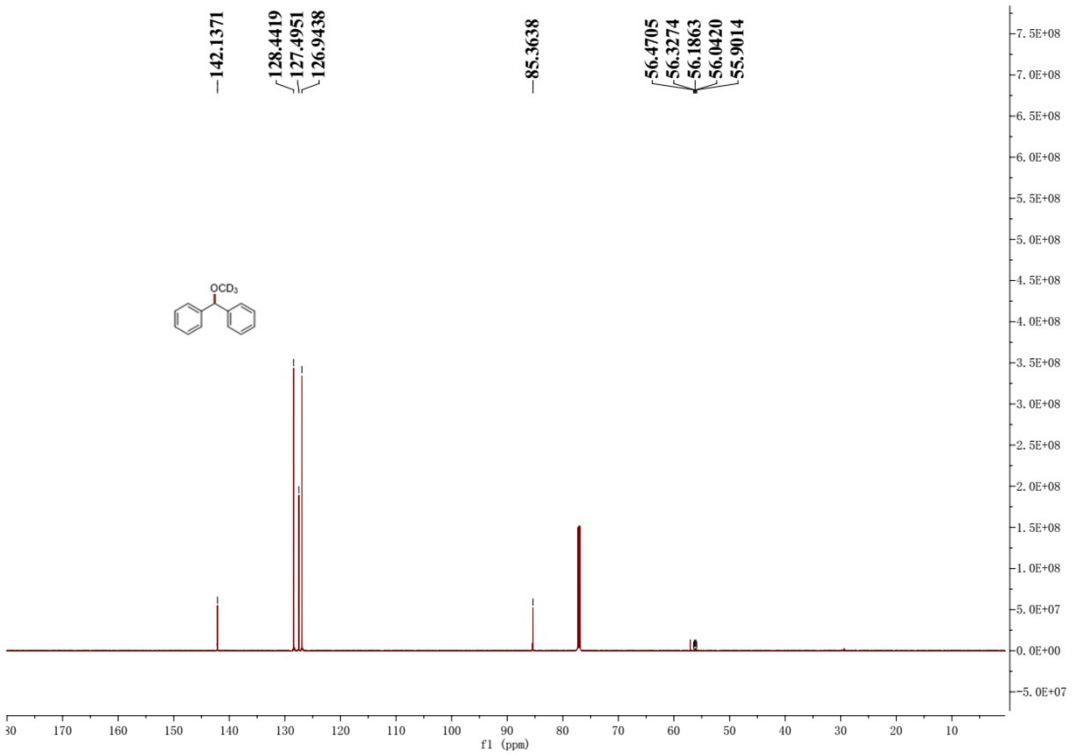
4.2754
4.2593
4.2432
3.6756
2.9687
2.9496
2.9295
2.6554
2.6351
2.6161
1.4259
1.4098
OCD3
H3CO
O
1.96
2.00
1.00
3.00
2.04
2.07
3.20
f1 (ppm)


173.3306
141.4542
139.6574
128.2876
126.3441
79.2207
51.5829
35.6127
30.5669
23.7292
OCD3
H3CO
O
f1 (ppm)


7.2818
7.2624
7.2397
7.2206
7.1797
7.1622
7.1461
5.1639
OCD3
8.15
2.09
1.00
f1 (ppm)


142.1371
128.4419
127.4951
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85.3638
56.4705
56.3274
56.1863
56.0420
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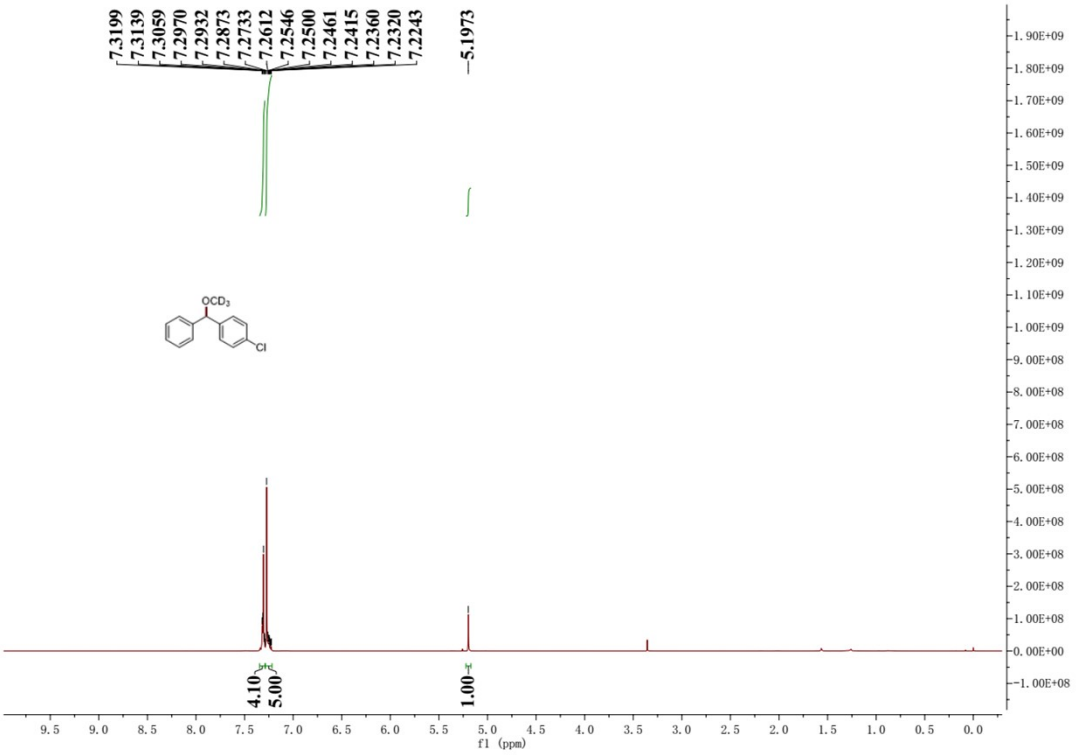
OCD3
f1 (ppm)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7.3139
7.3059
7.2970
7.2932
7.2873
7.2733
7.2612
7.2546
7.2500
7.2461
7.2415
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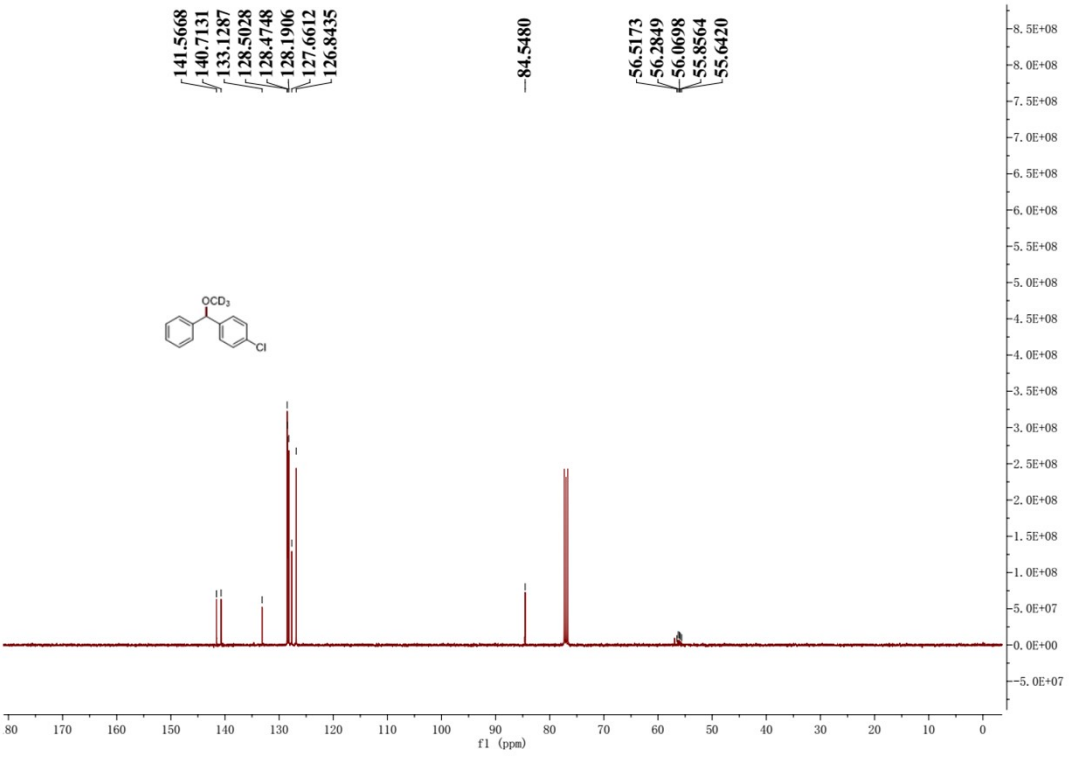
7.2320
7.2243
5.1973
OCD3
Cl
4.10
5.00
1.00
f1 (ppm)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133.1287
128.5028
128.4748
128.1906
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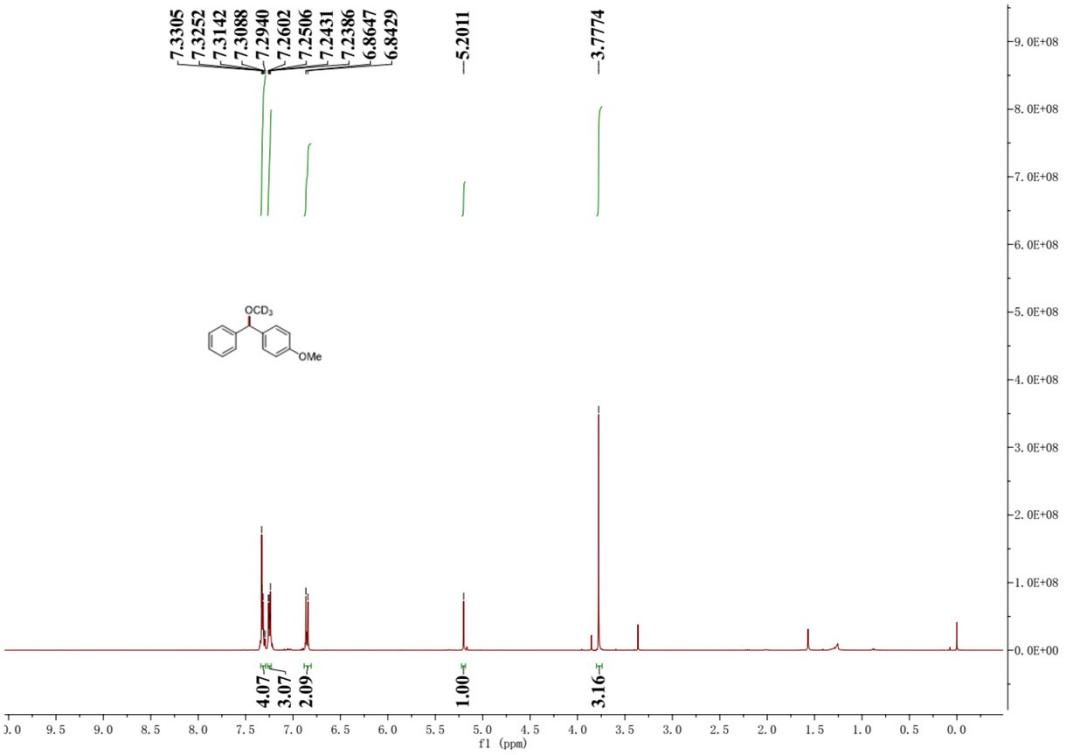
56.5173
56.2849
56.0698
55.8564
55.6420
OCD3
Cl
f1 (ppm)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7.3252
7.3142
7.3088
7.2940
7.2602
7.2506
7.2431
7.2386
6.8647
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3.7774
OCD3
OMe
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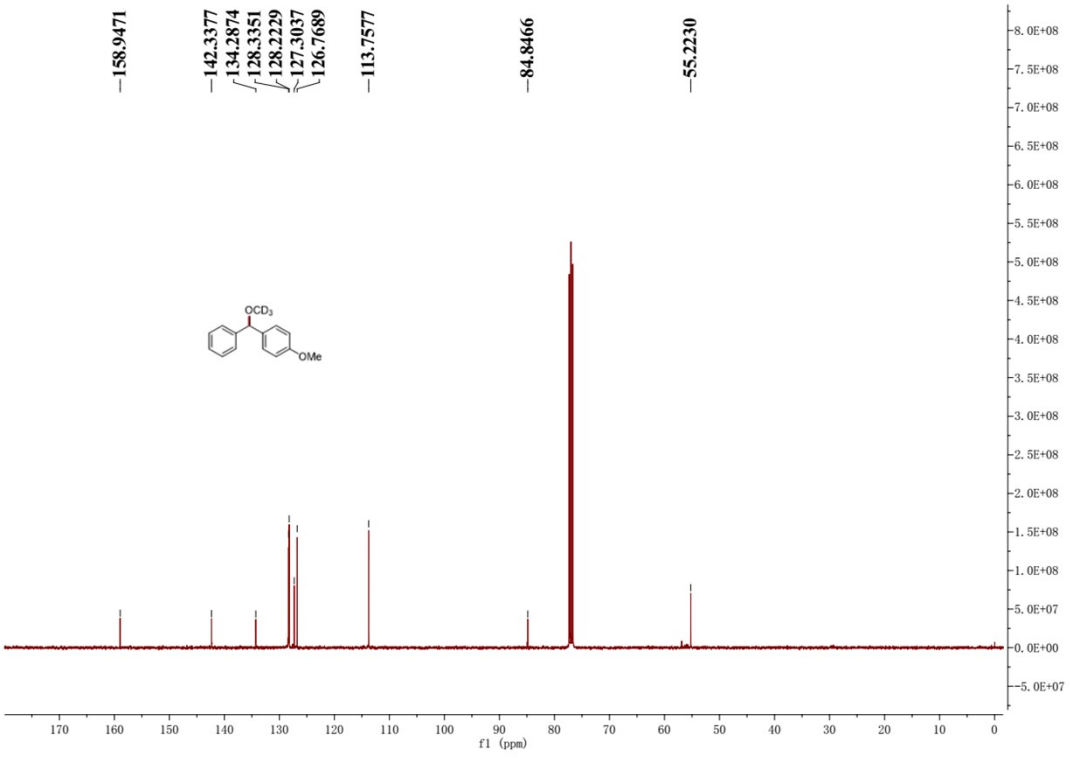
3.07
2.09
1.00
3.16
f1 (ppm)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127.3037
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113.7577
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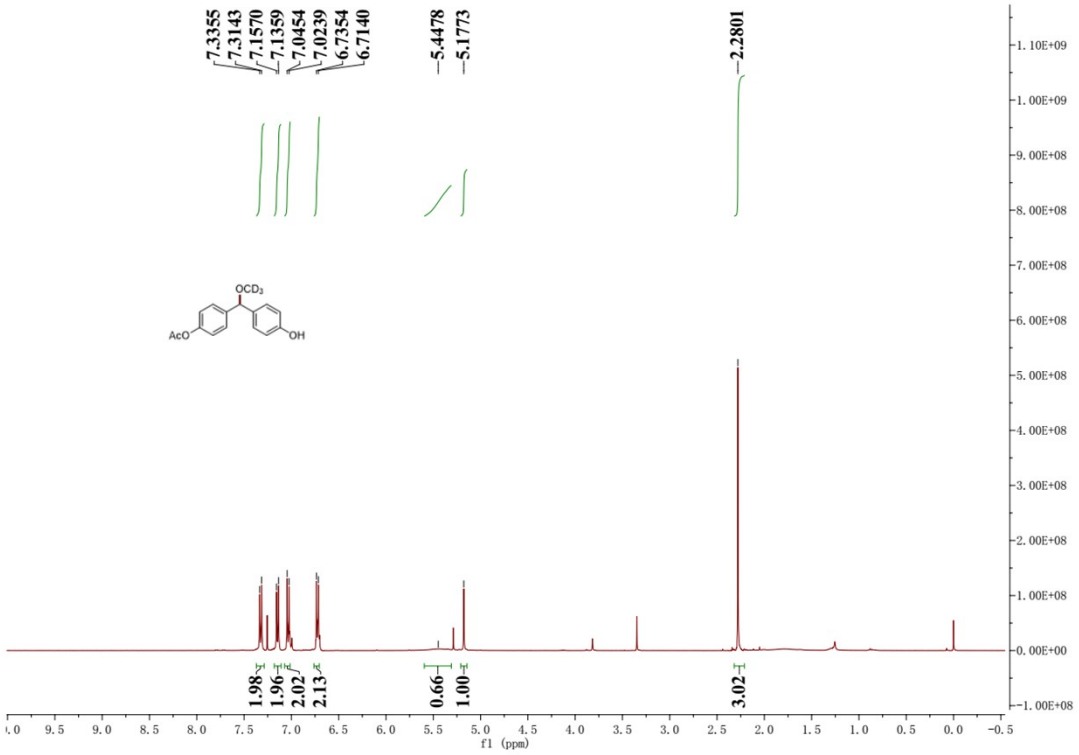
55.2230
OCD3
OMe
f1 (ppm)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7.1570
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7.0239
6.7354
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5.4478
5.1773
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AcO
OH
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1.96
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2.13
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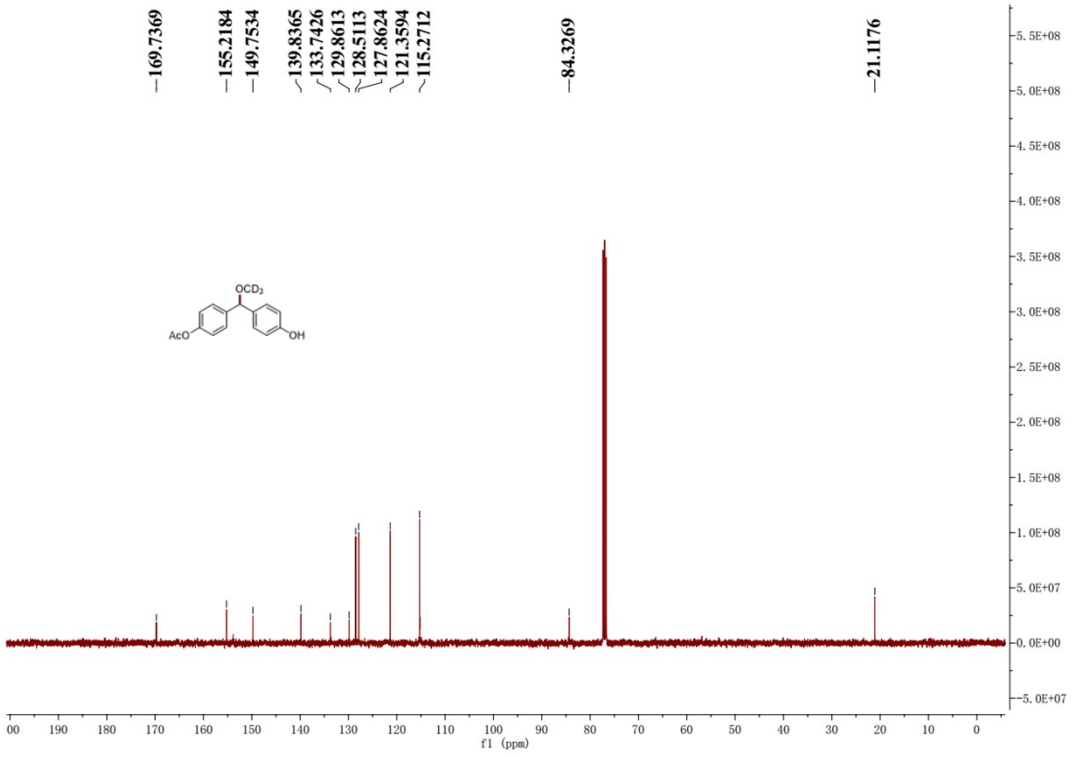
1.00
3.02
f1 (ppm)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121.3594
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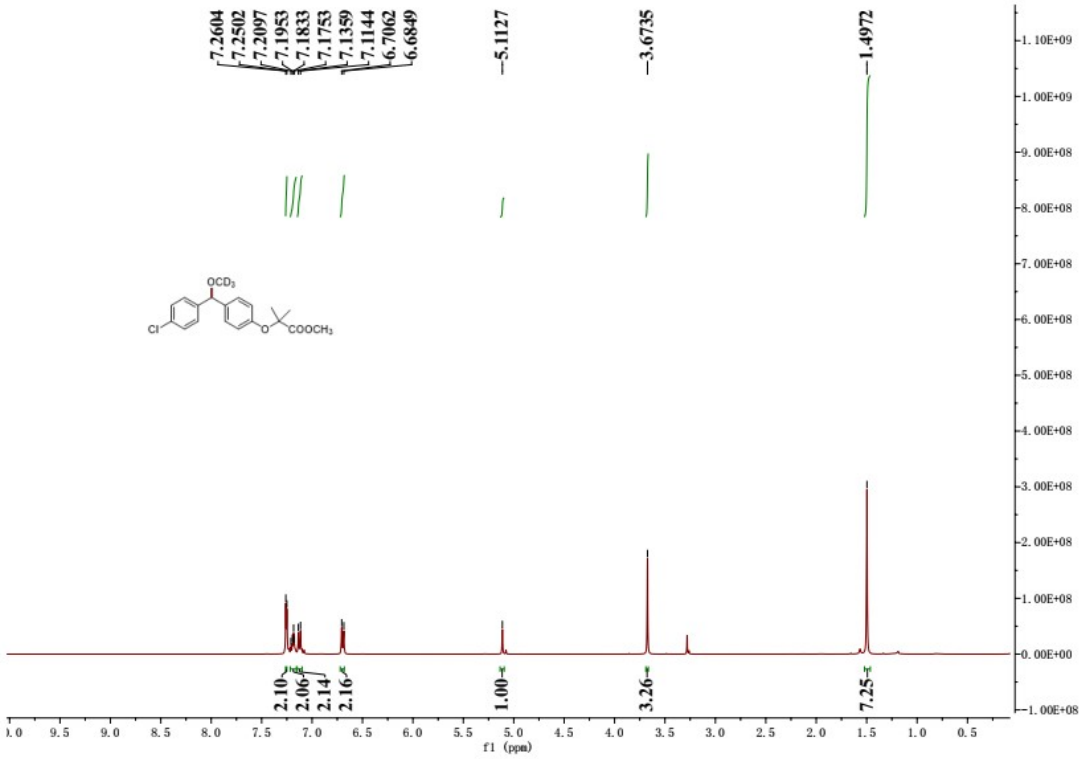
21.1176
OCD3
AcO
OH
f1 (ppm)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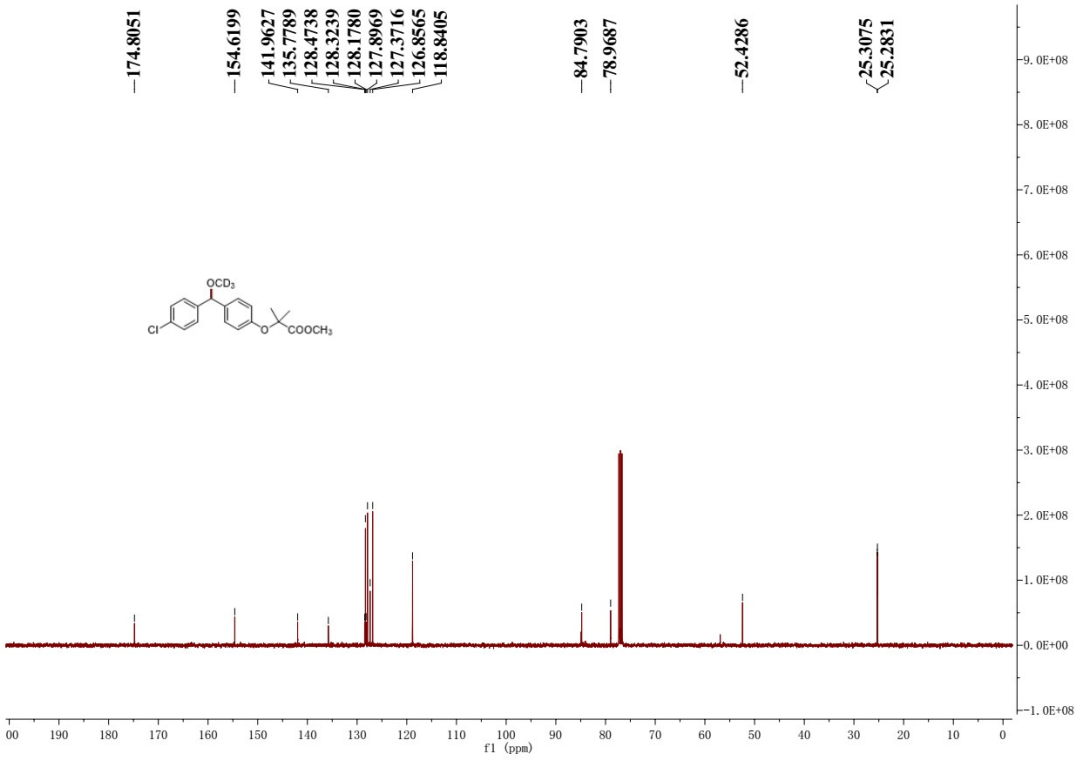
7.2097
7.1953
7.1833
7.1753
7.1359
7.1144
6.7062
6.6849
5.1127
3.6735
1.4972
OCD3
Cl
O
COOCH3
2.10
2.06
2.14
2.16
1.00
3.26
7.25
f1 (ppm)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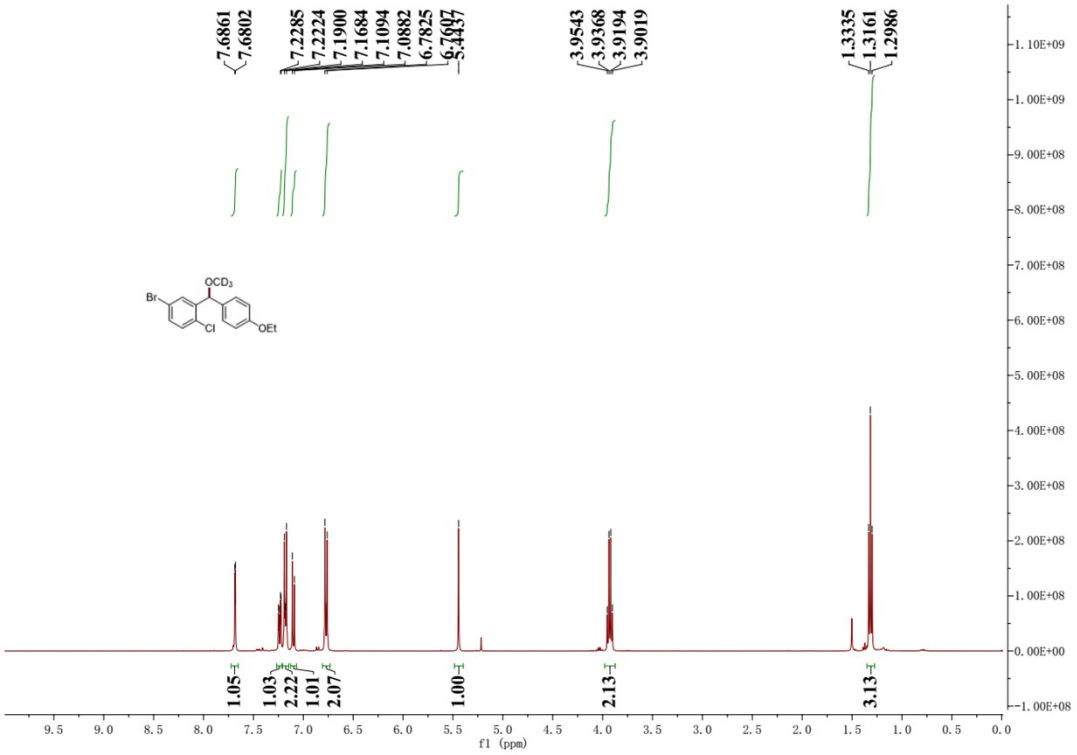
78.9687
52.4286
25.3075
25.2831
OCD3
Cl
O
COOCH3
f1 (ppm)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7.1094
7.0882
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3.9368
3.9194
3.9019
1.3335
1.3161
1.2986
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Cl
OEt
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1.03
2.22
1.01
2.07
1.00
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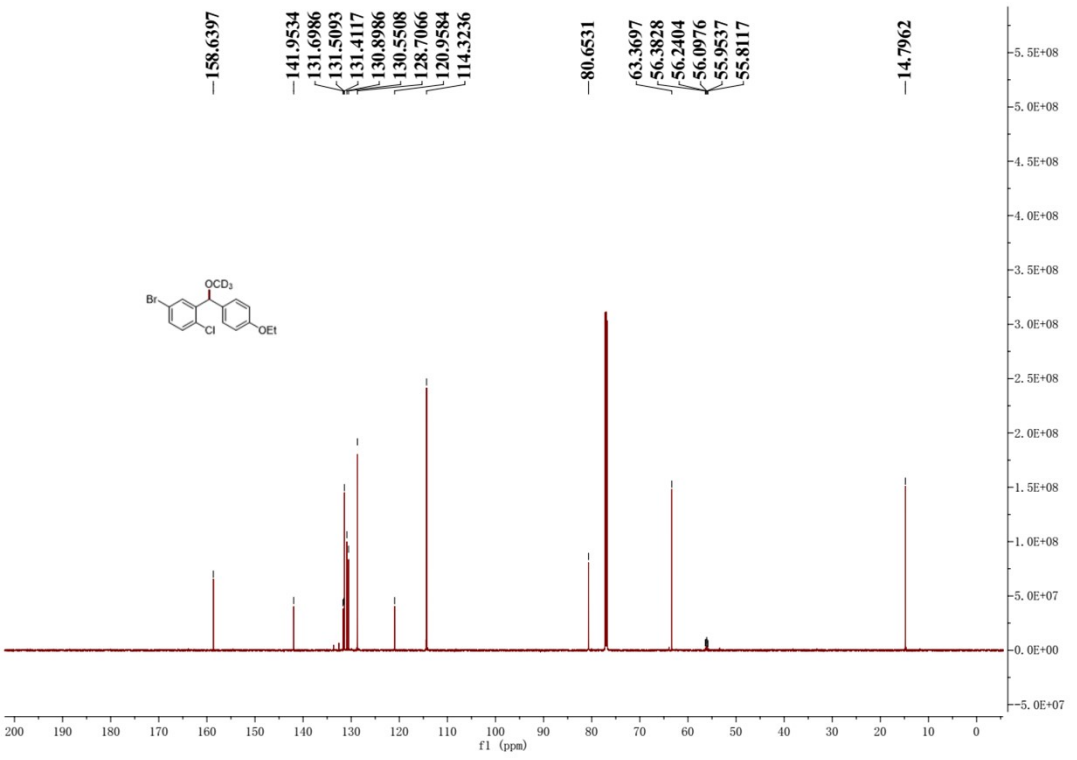
3.13
f1 (ppm)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63.3697
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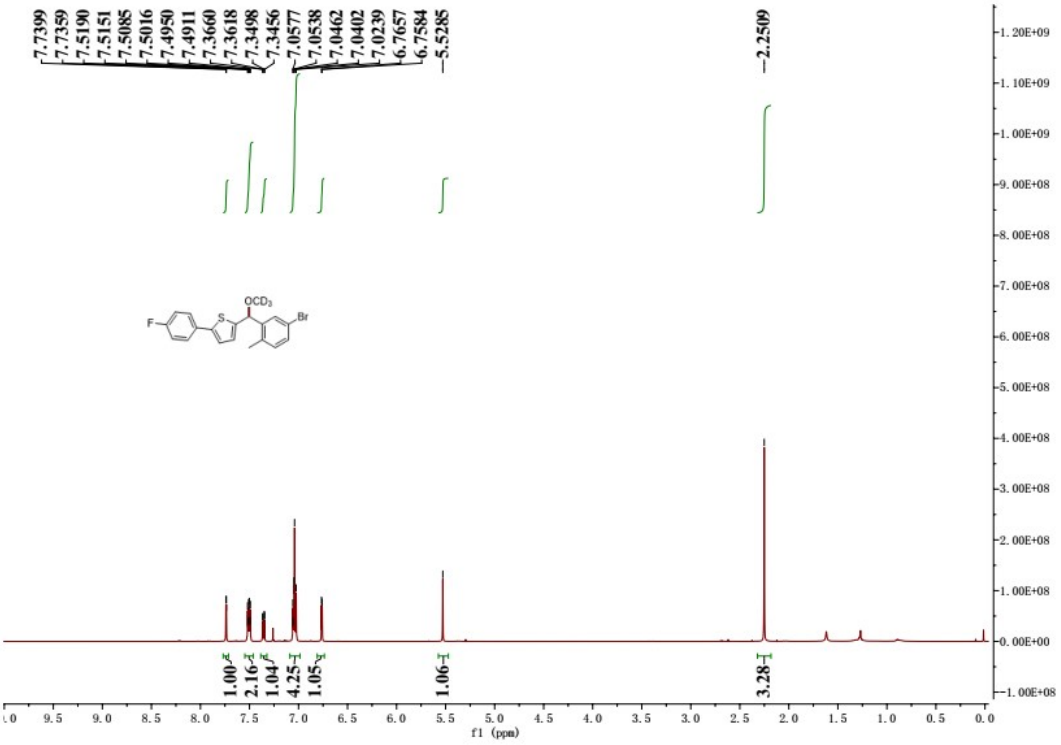
Br
Cl
OEt
f1 (ppm)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7.3456
7.0577
7.0538
7.0462
7.0402
7.0239
6.7657
6.7584
5.5285
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S
Br
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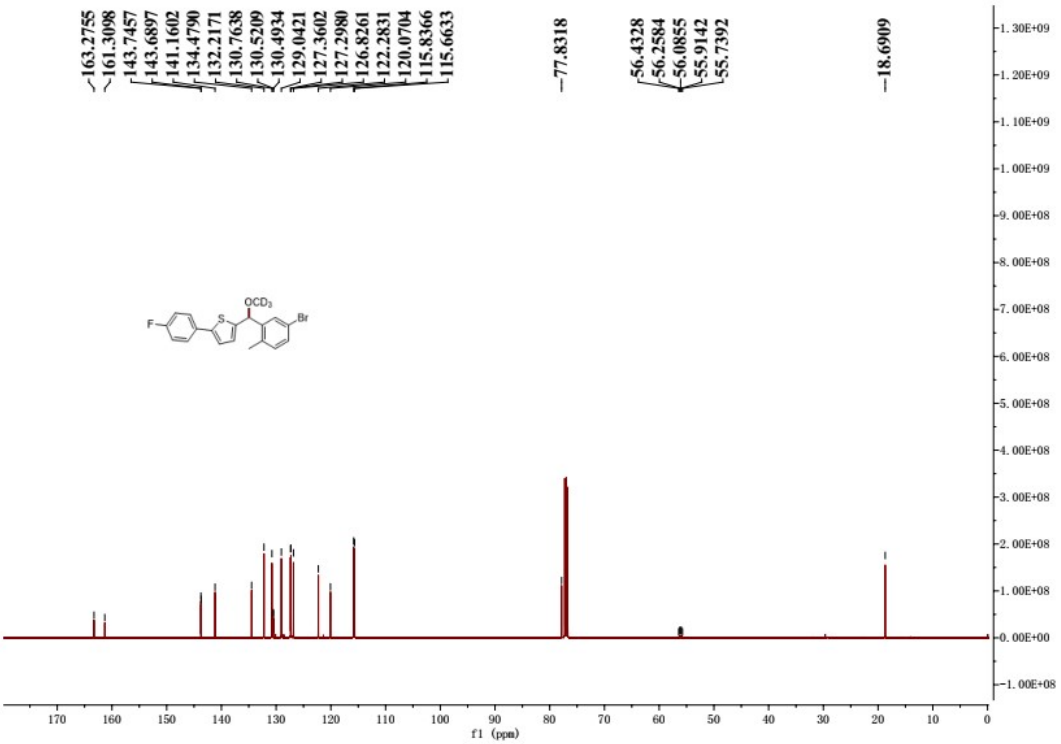
4.25
1.05
1.06
3.28
f1 (ppm)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127.2980
126.8261
122.2831
120.0704
115.8366
115.6633
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56.0855
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55.7392
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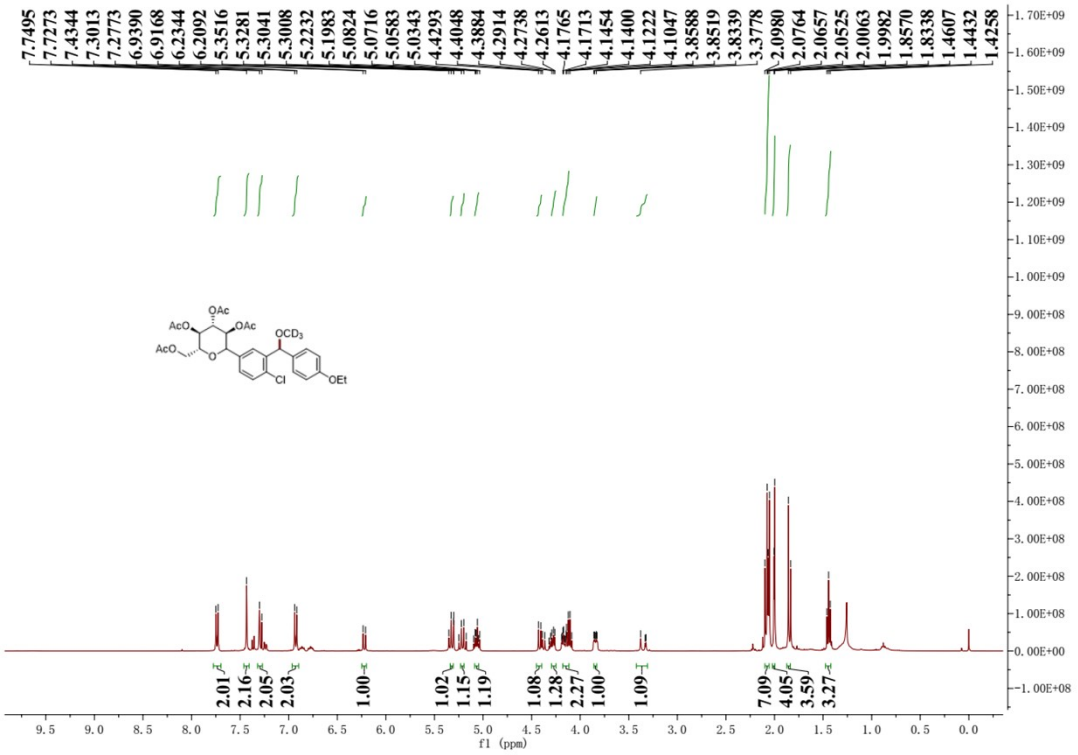
OCD3
F
S
Br
f1 (ppm)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5.3281
5.3041
5.3008
5.2232
5.1983
5.0824
5.0716
5.0583
5.0343
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4.2738
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3.8519
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1.8570
1.8338
1.4607
1.4432
1.4258
OAc
AcO
OAc
AcO
OCD3
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OEt
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2.16
2.05
2.03
1.00
1.02
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1.19
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1.28
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7.09
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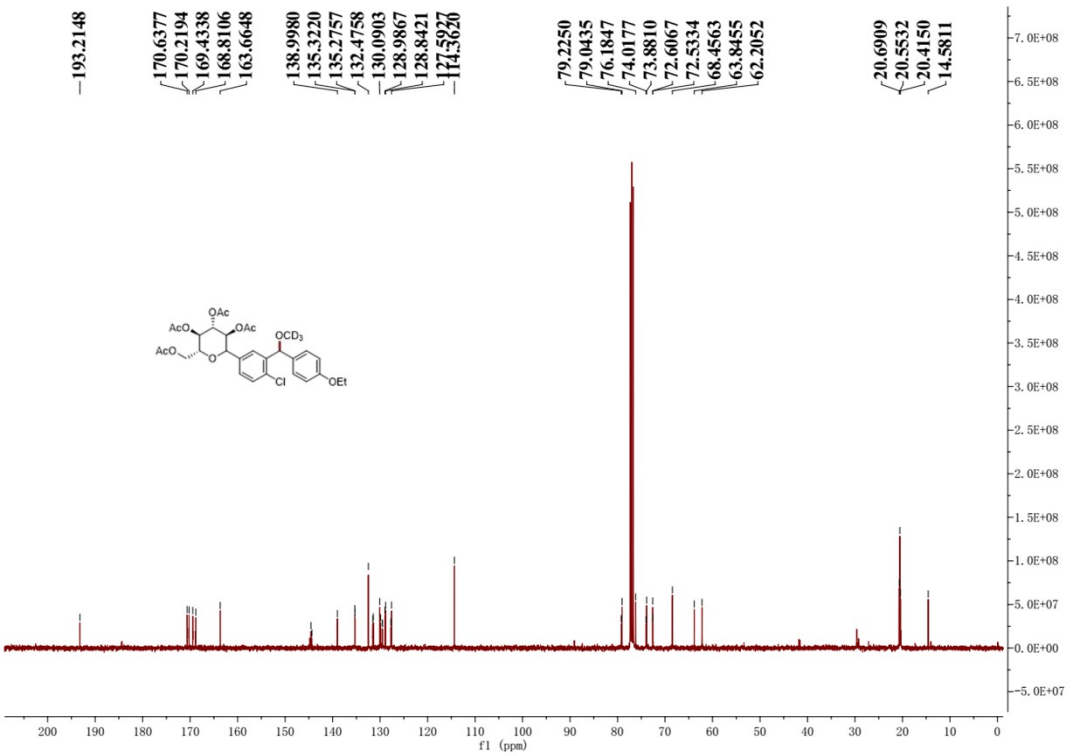
3.27
f1 (ppm)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128.9867
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127.5927
114.3620
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76.1847
74.0177
73.8810
72.6067
72.5334
68.4563
63.8455
62.2052
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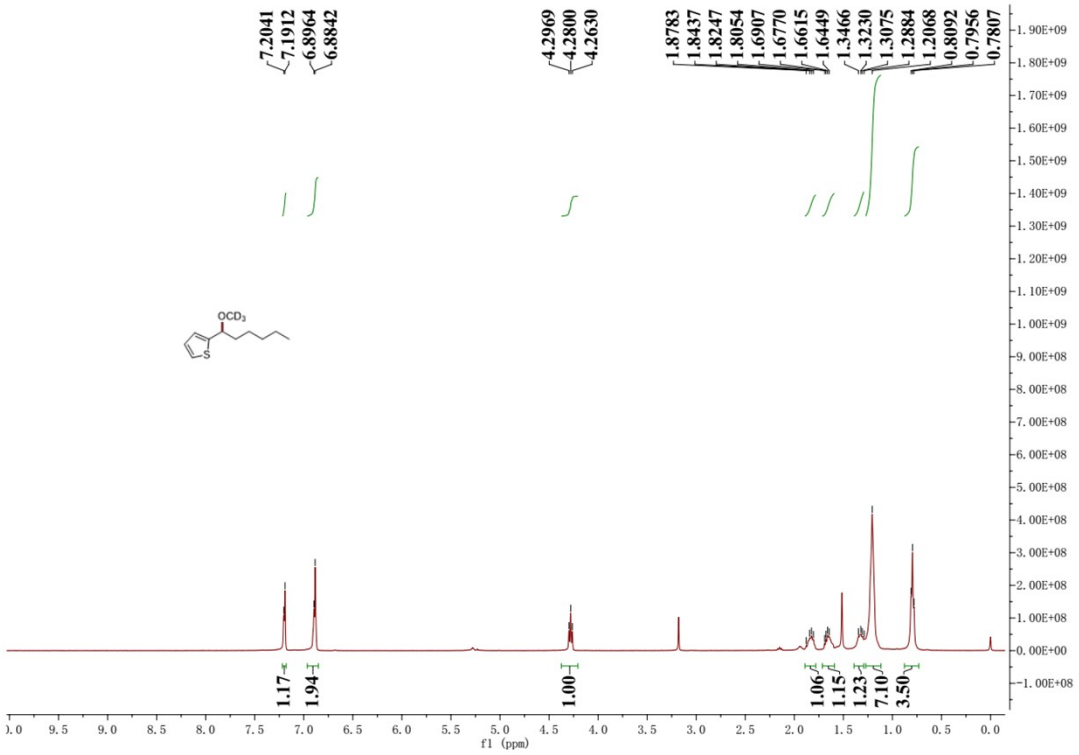
OAc
AcO
OAc
AcO
OCD3
Cl
OEt
f1 (ppm)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1.8054
1.6907
1.6770
1.6615
1.6449
1.3466
1.3230
1.3075
1.2884
1.2068
0.8092
0.7956
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1.94
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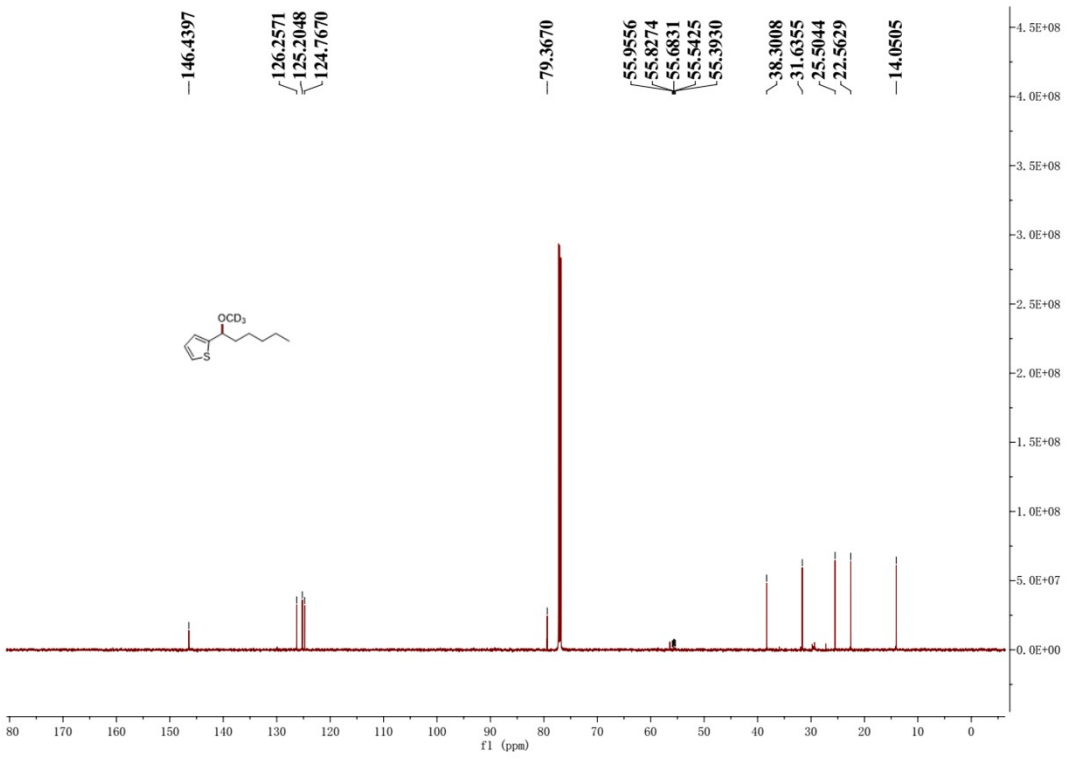
1.06
1.15
1.23
7.10
3.50
f1 (ppm)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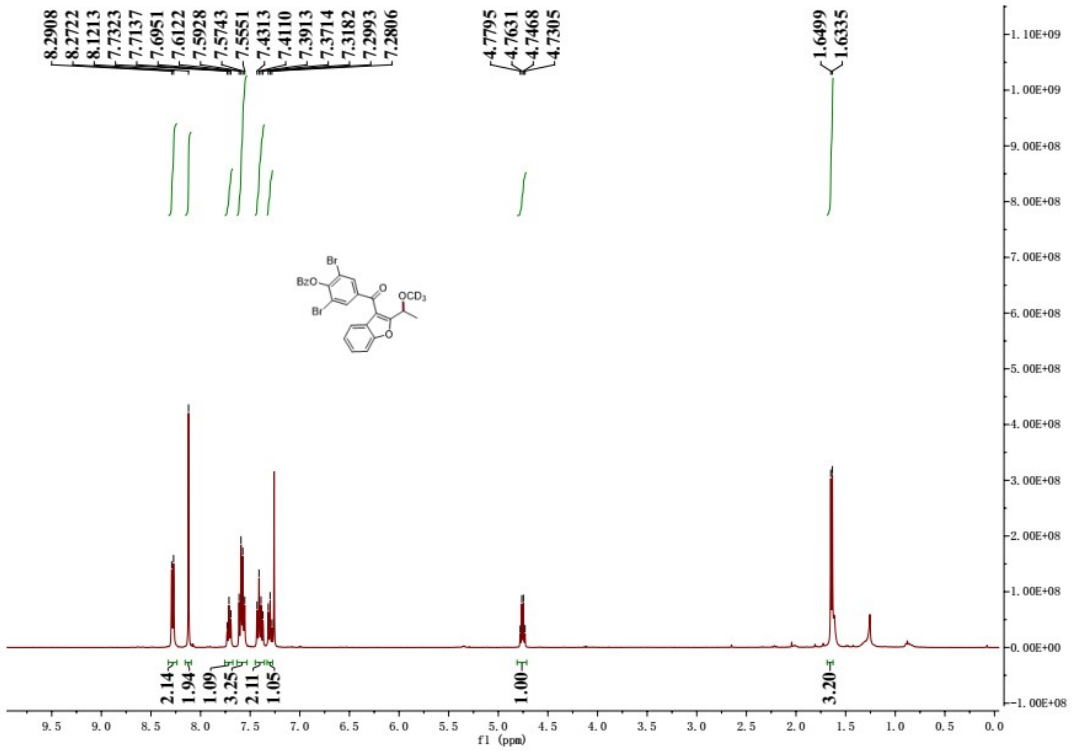
31.6355
25.5044
22.5629
14.0505
OCD3
f1 (ppm)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7.4110
7.3913
7.3714
7.3182
7.2993
7.2806
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4.7631
4.7468
4.7305
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1.6335
Br
BzO
Br
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1.09
3.25
2.11
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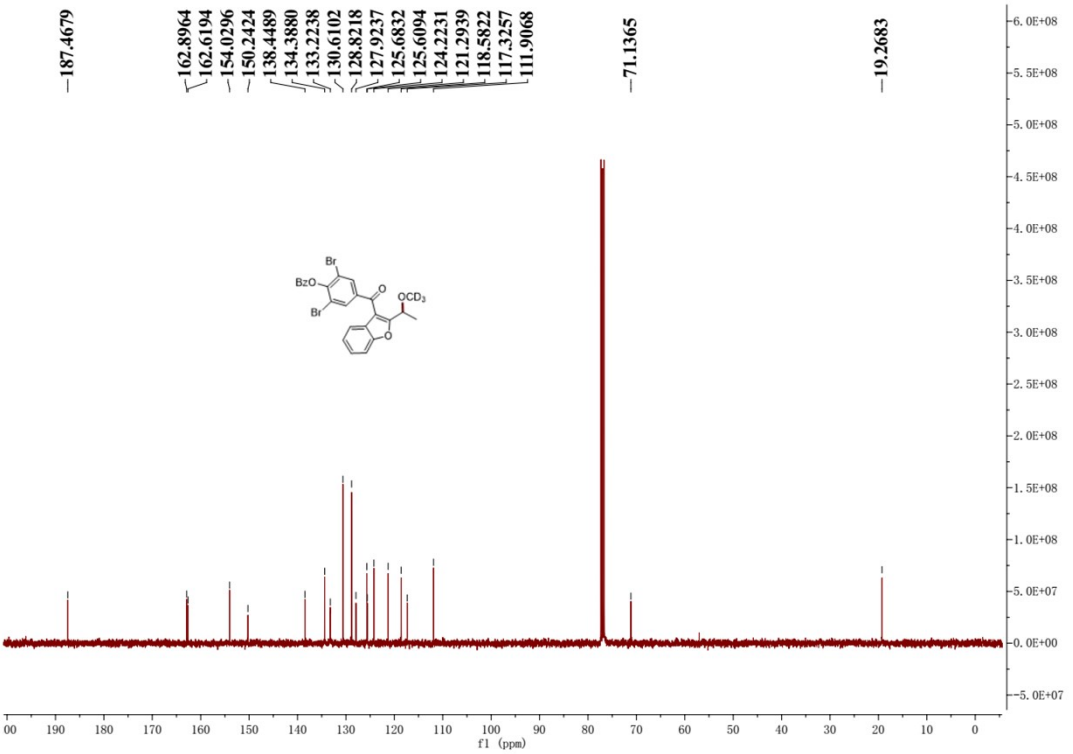
3.20
f1 (ppm)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124.2231
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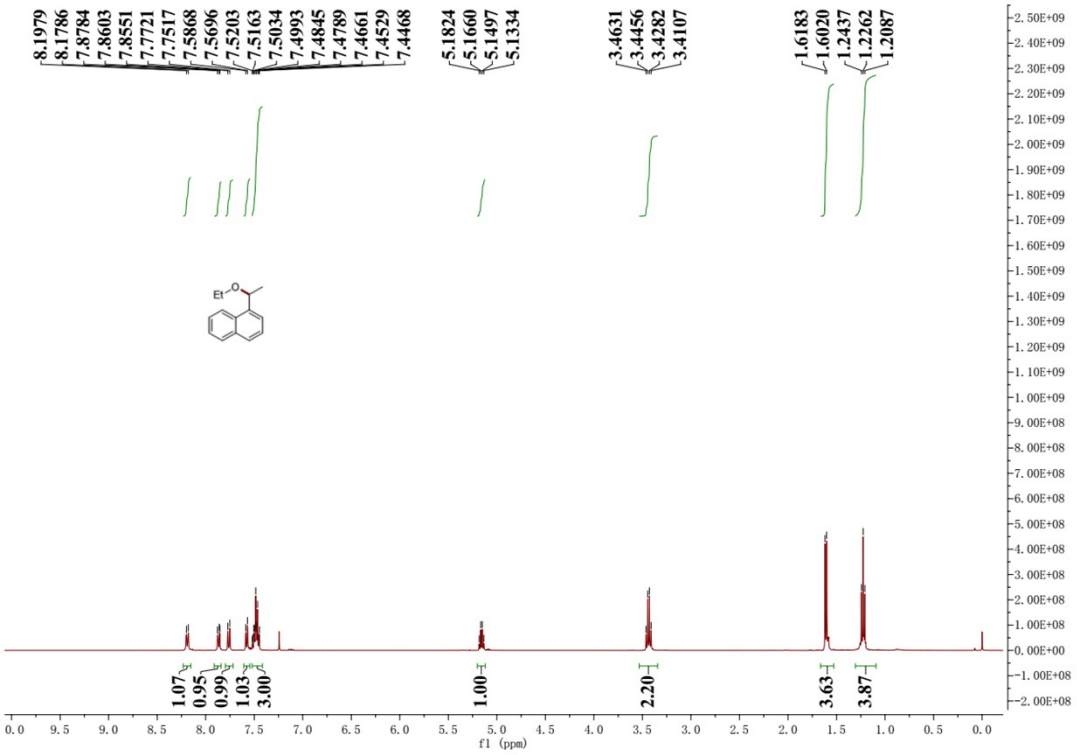
Br
BzO
Br
O
OCD3
f1 (ppm)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1.2437
1.2262
1.2087
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3.00
1.00
2.20
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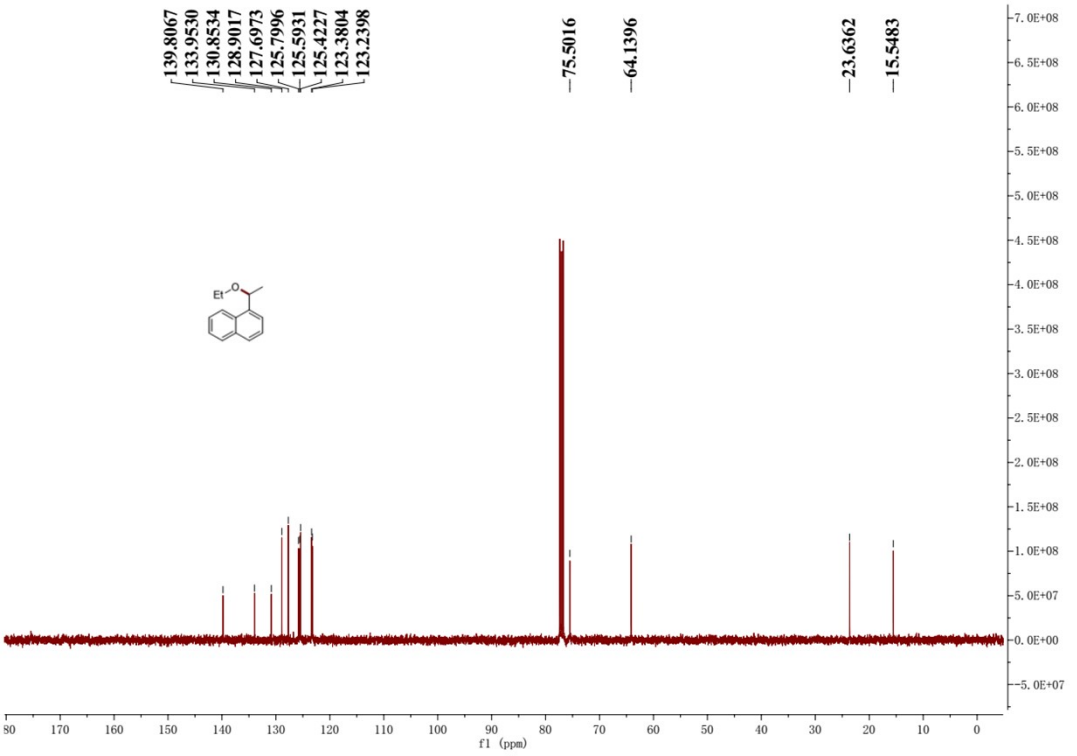
3.87
f1 (ppm)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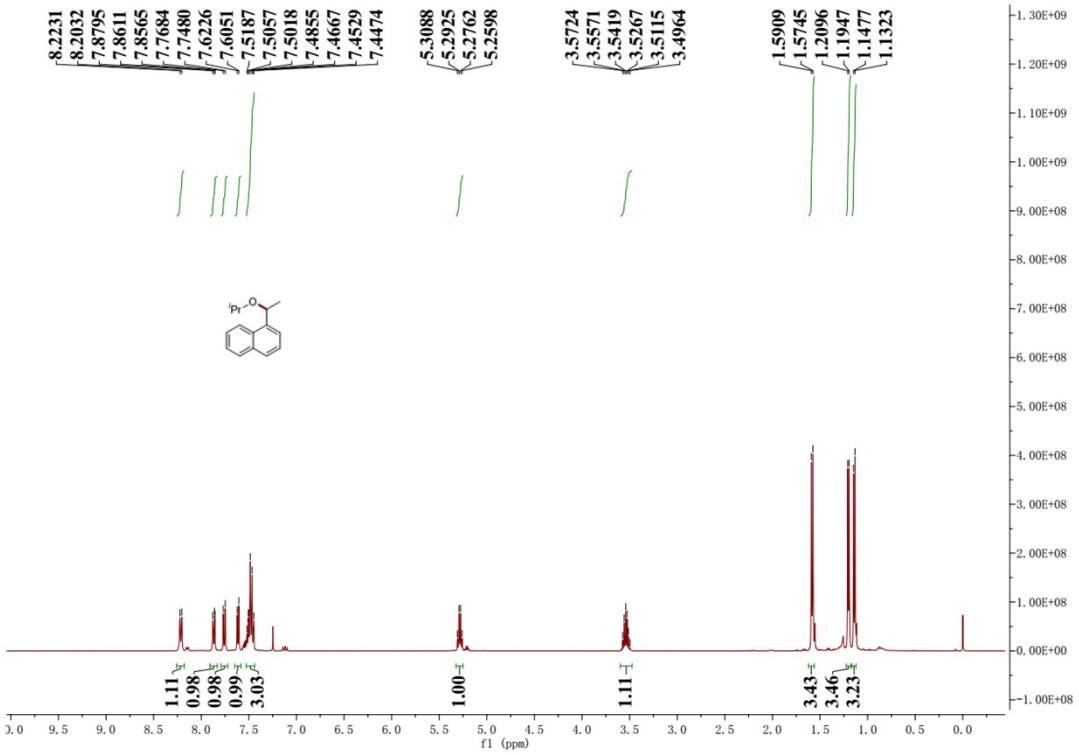
Et
O
f1 (ppm)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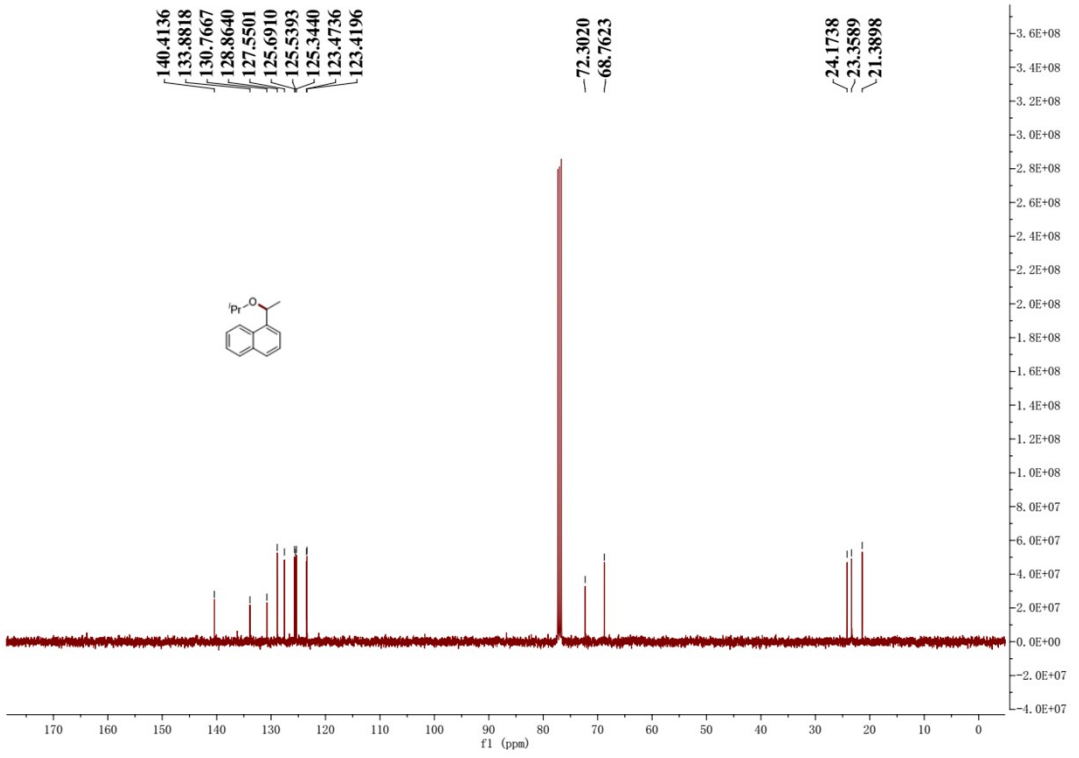
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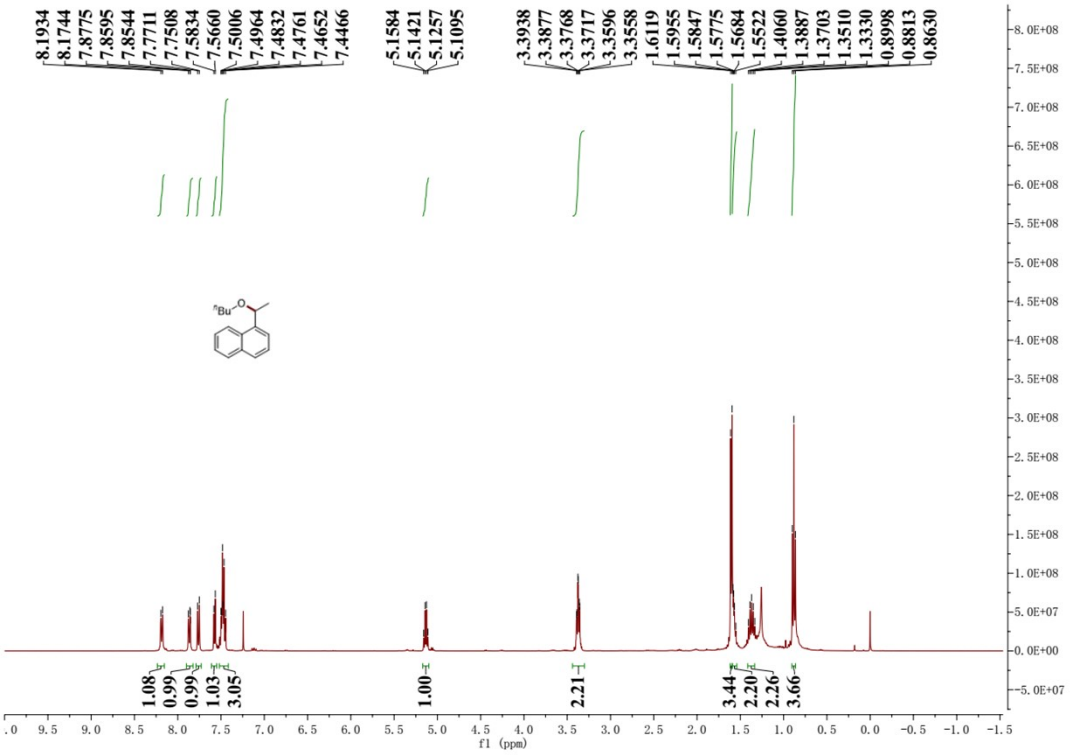
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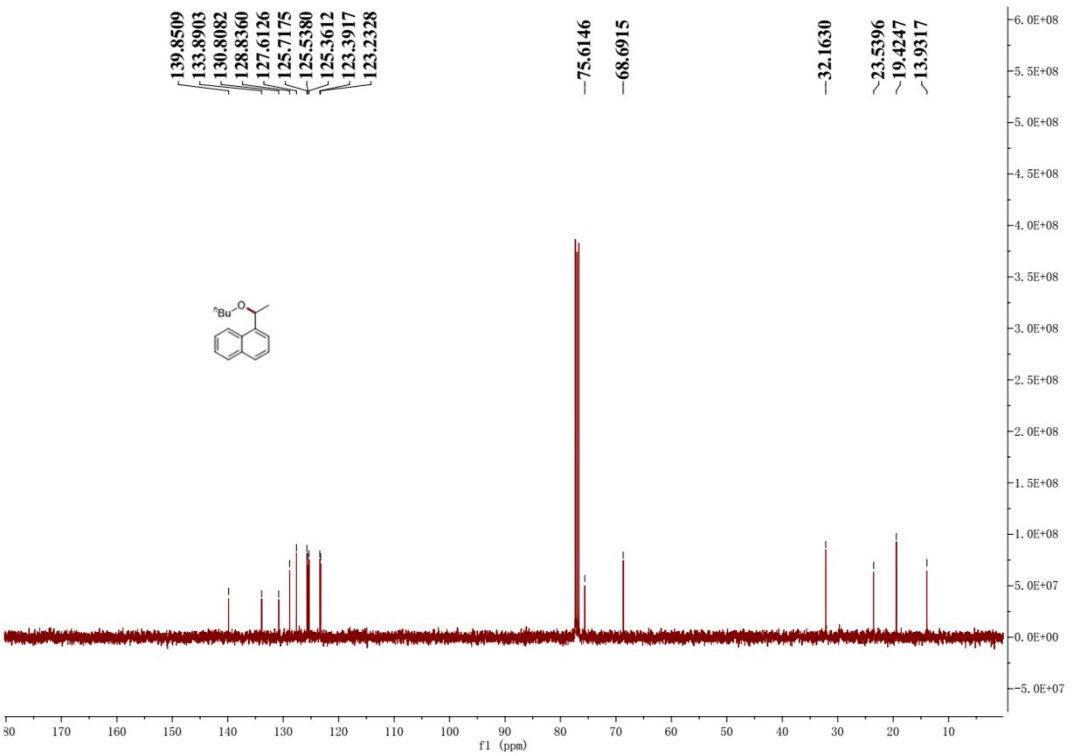
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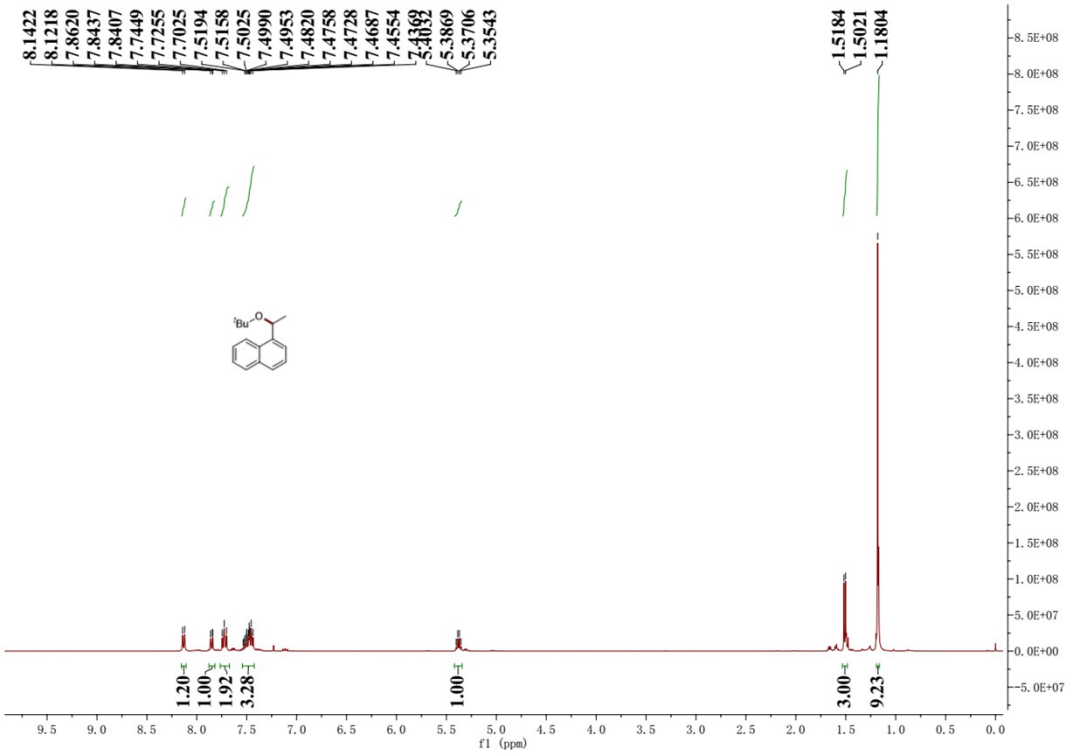
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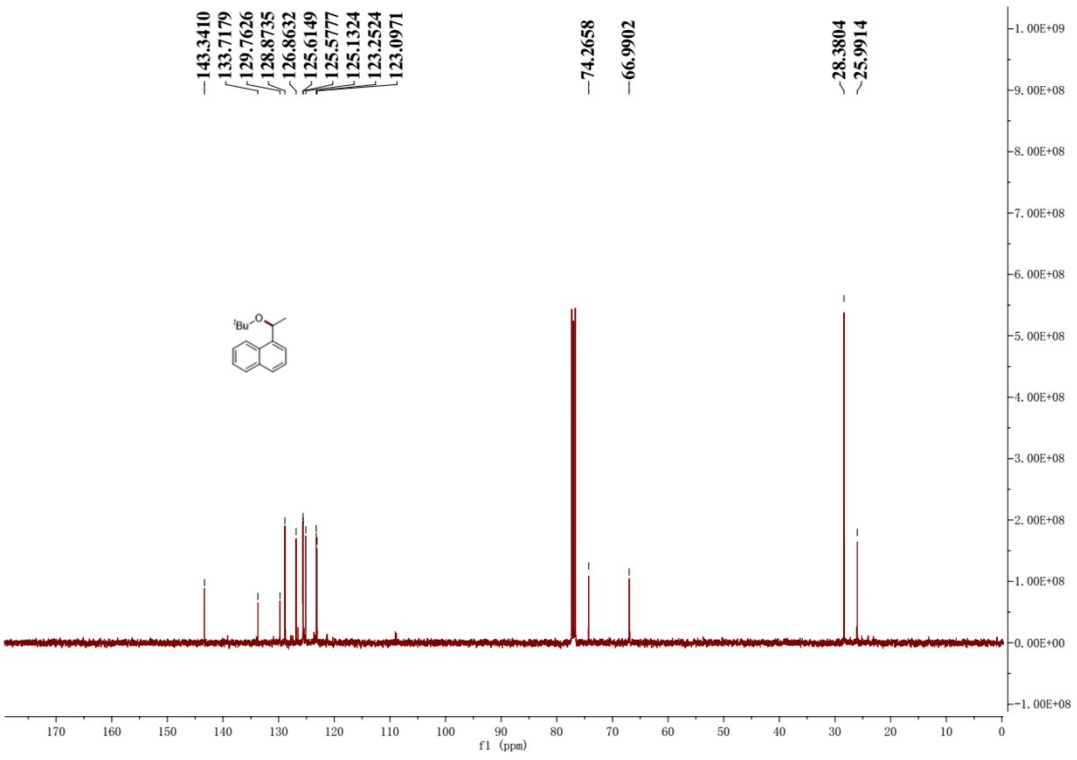
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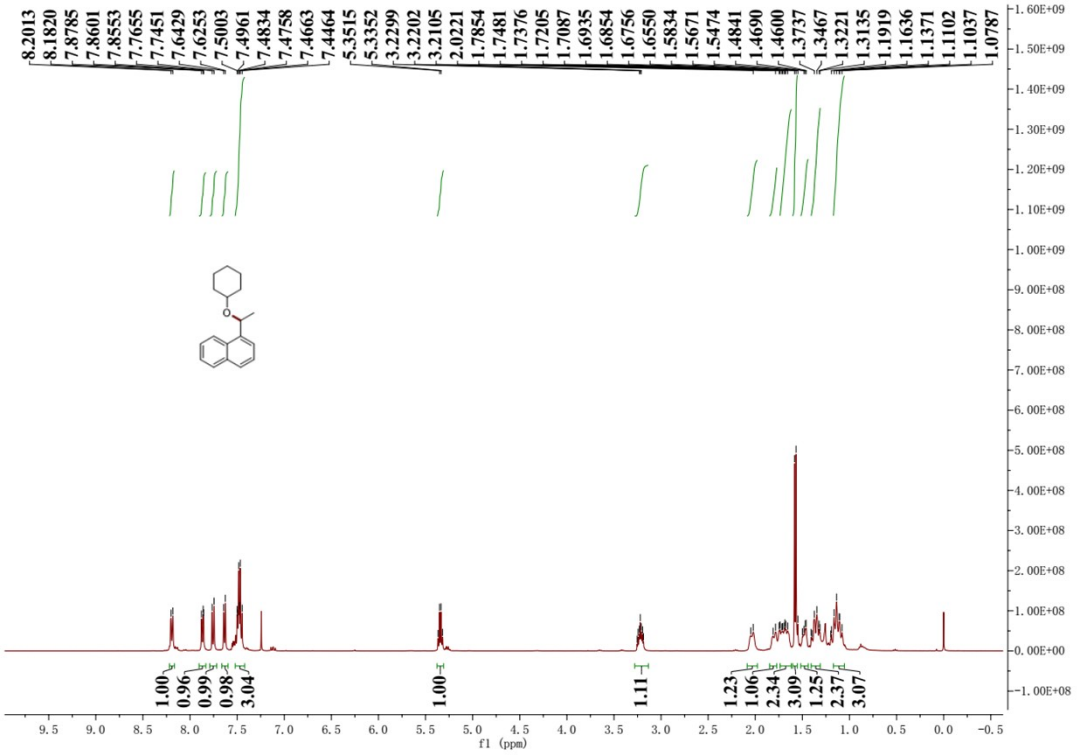
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1.1636
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1.00
0.96
0.99
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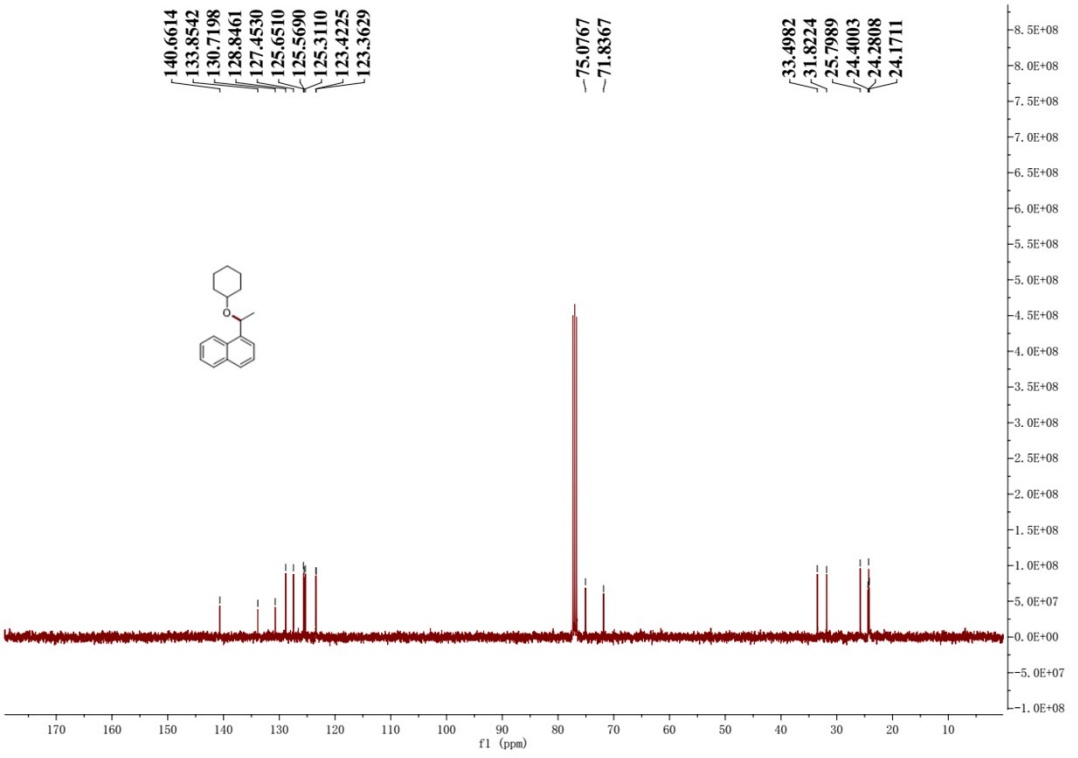
1.06
2.34
3.09
1.25
2.37
3.07
f1 (ppm)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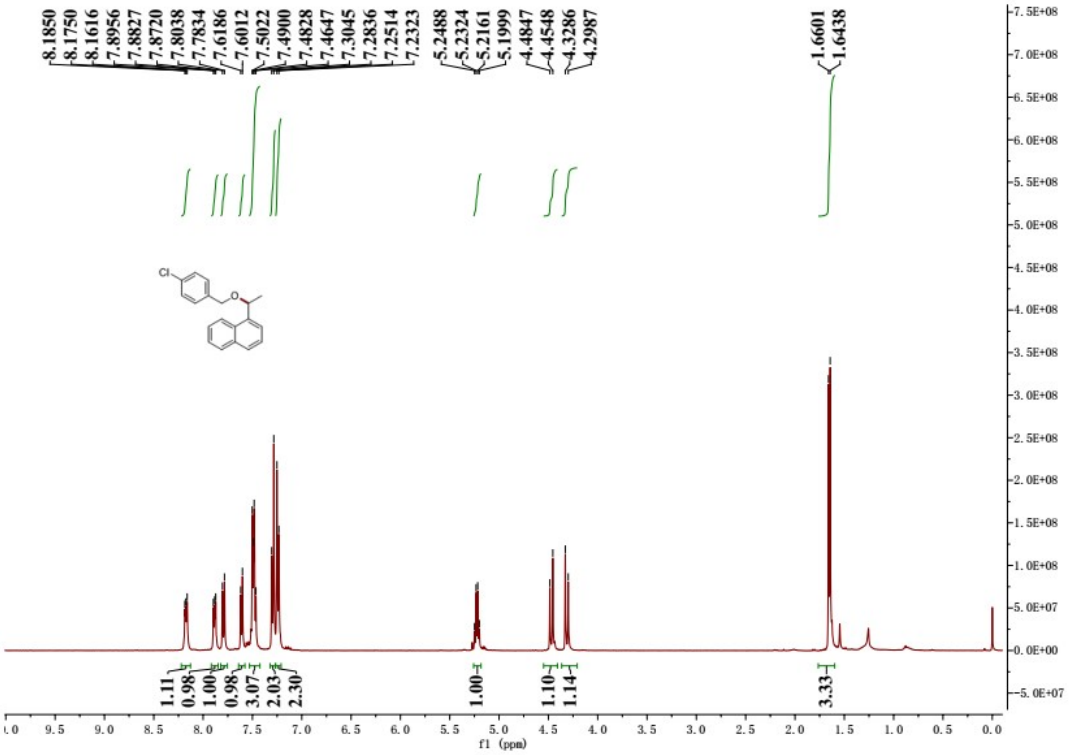
f1 (ppm)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Cl
O
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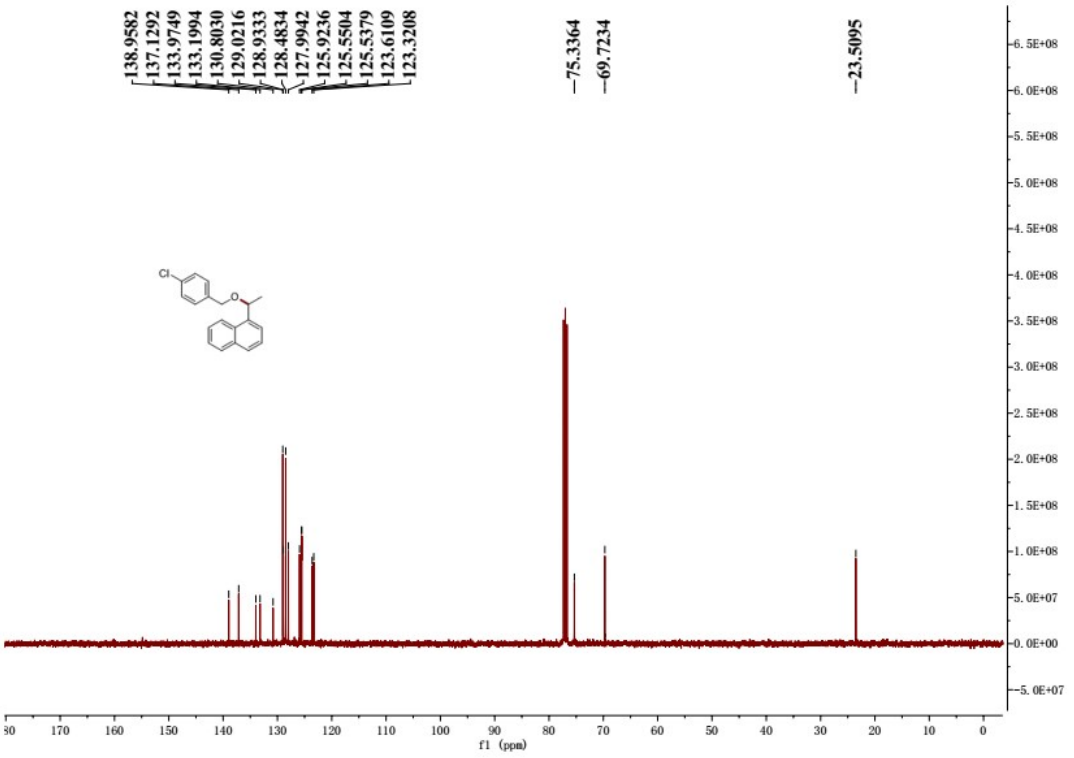
3.33
f1 (ppm)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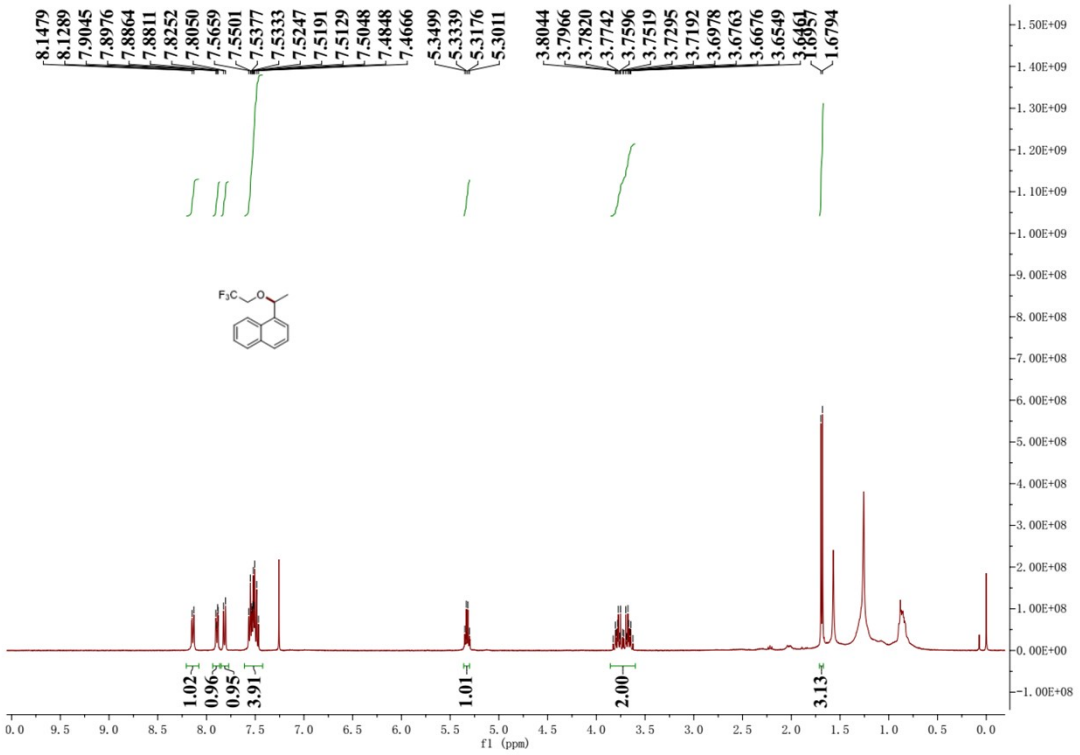
Cl
O
f1 (ppm)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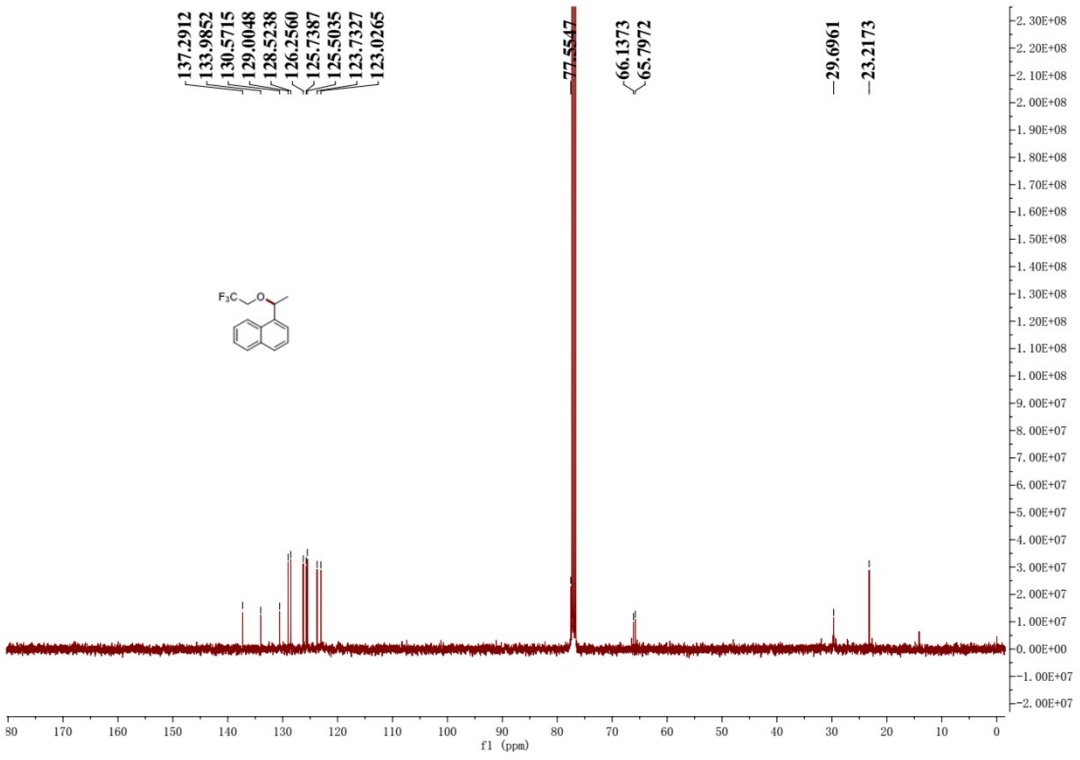
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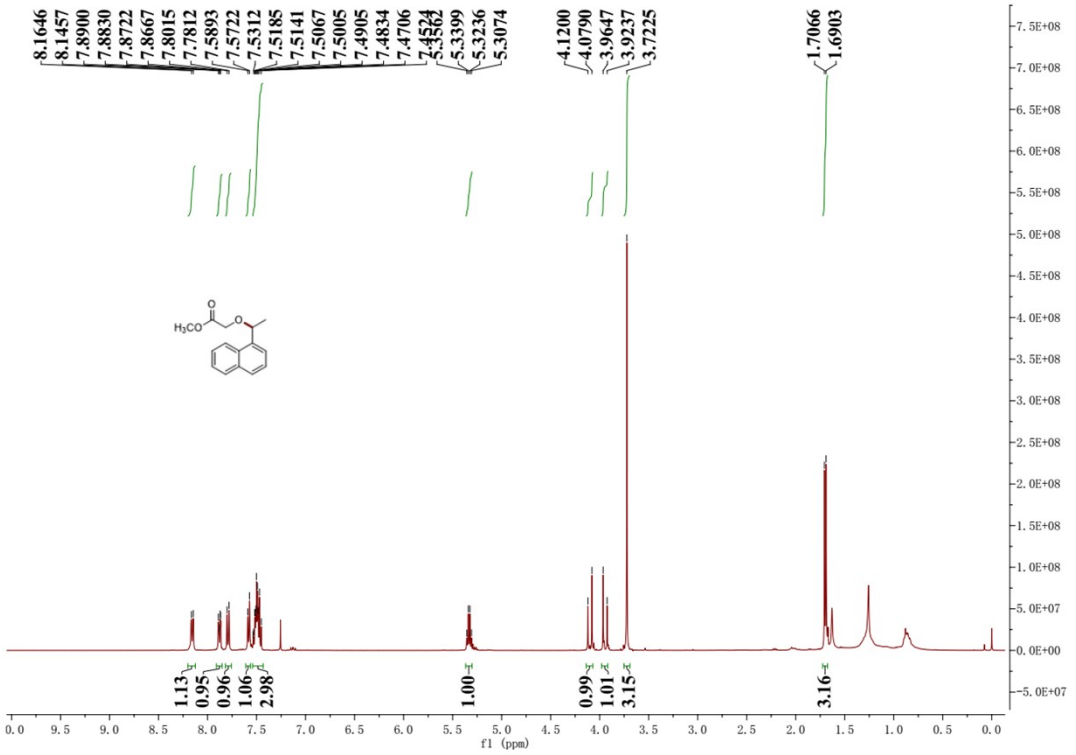
f1 (ppm)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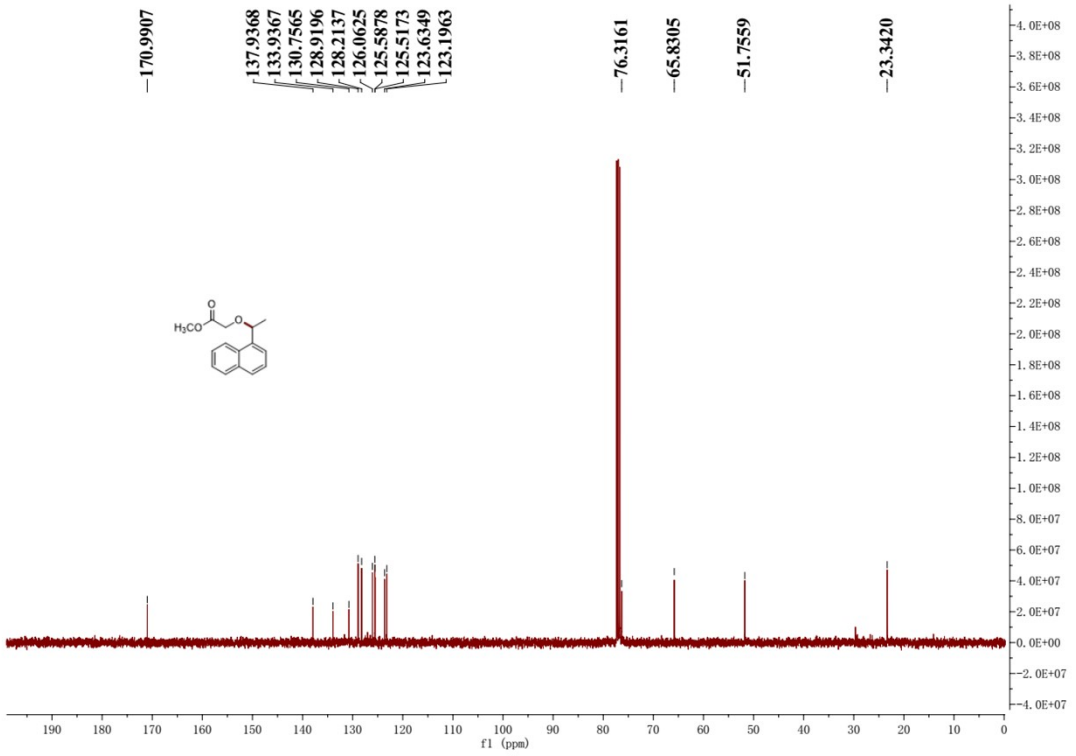
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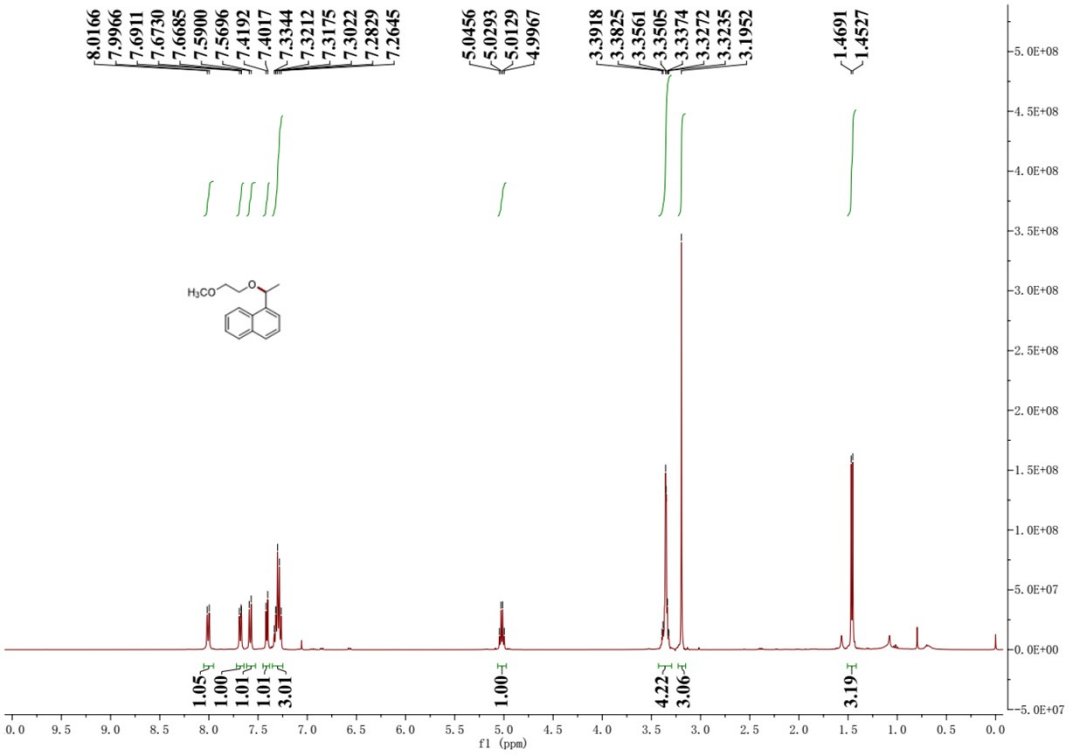
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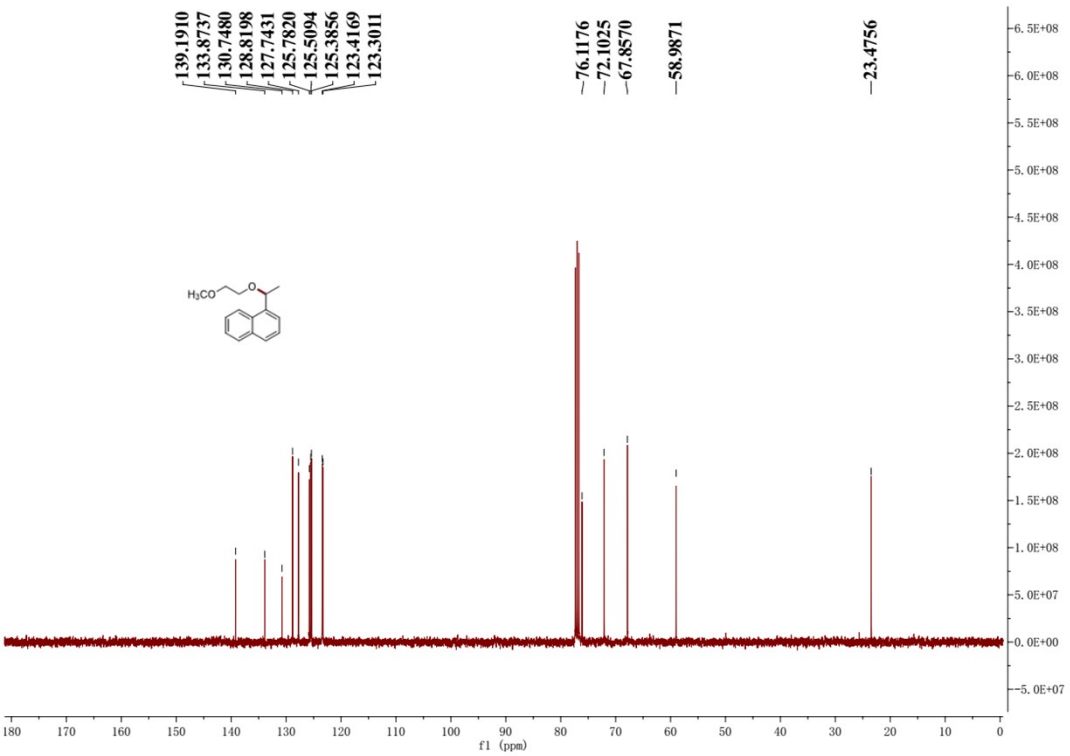
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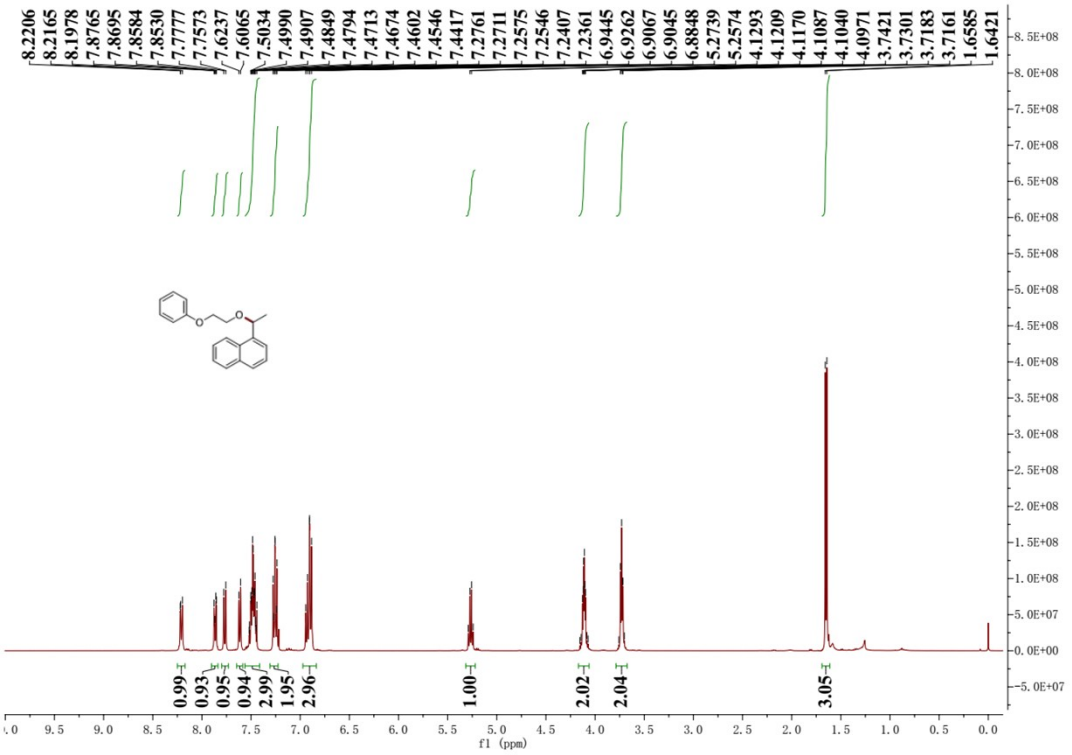
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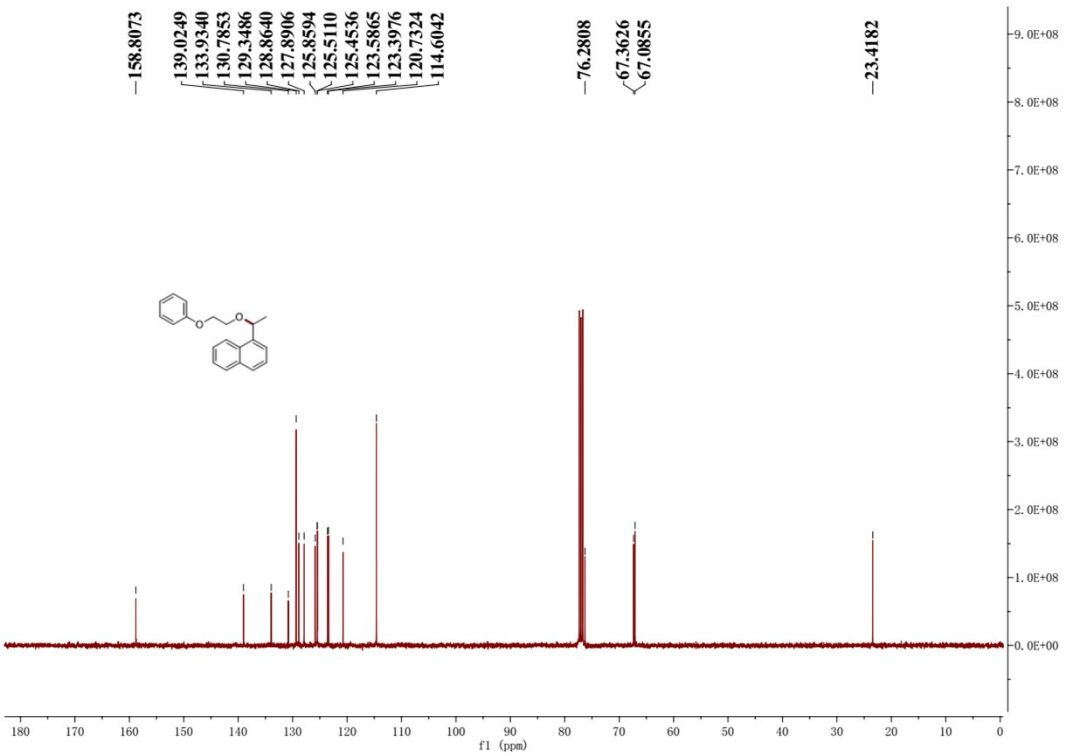
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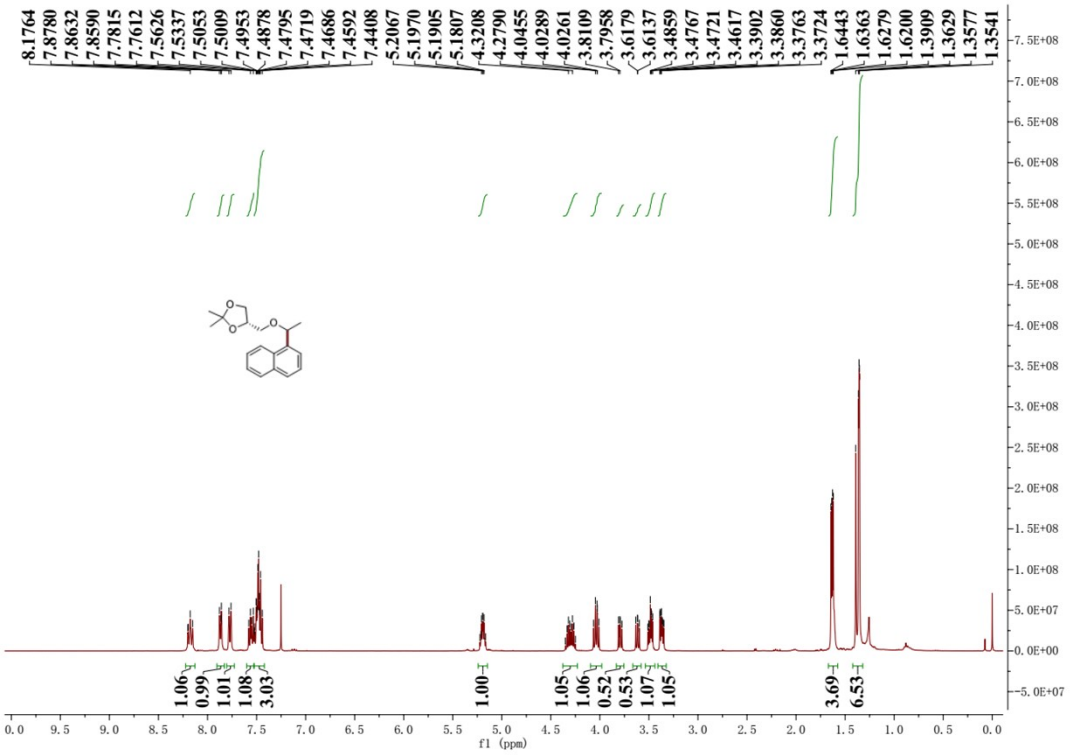
23.4182
f1 (ppm)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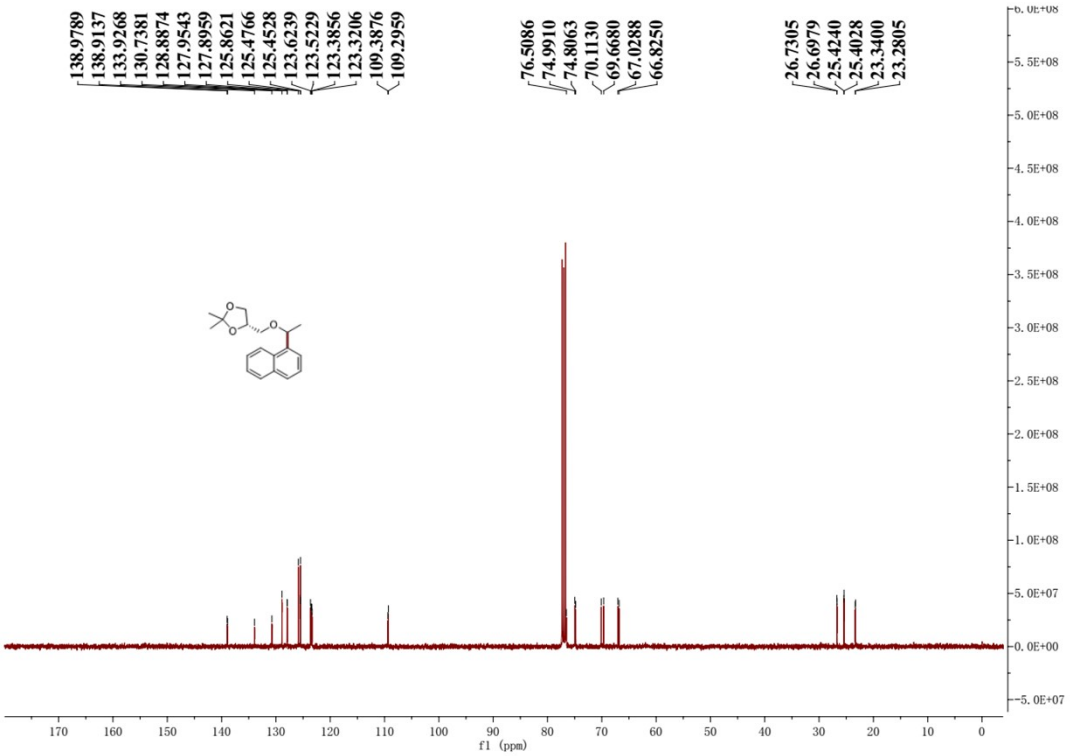
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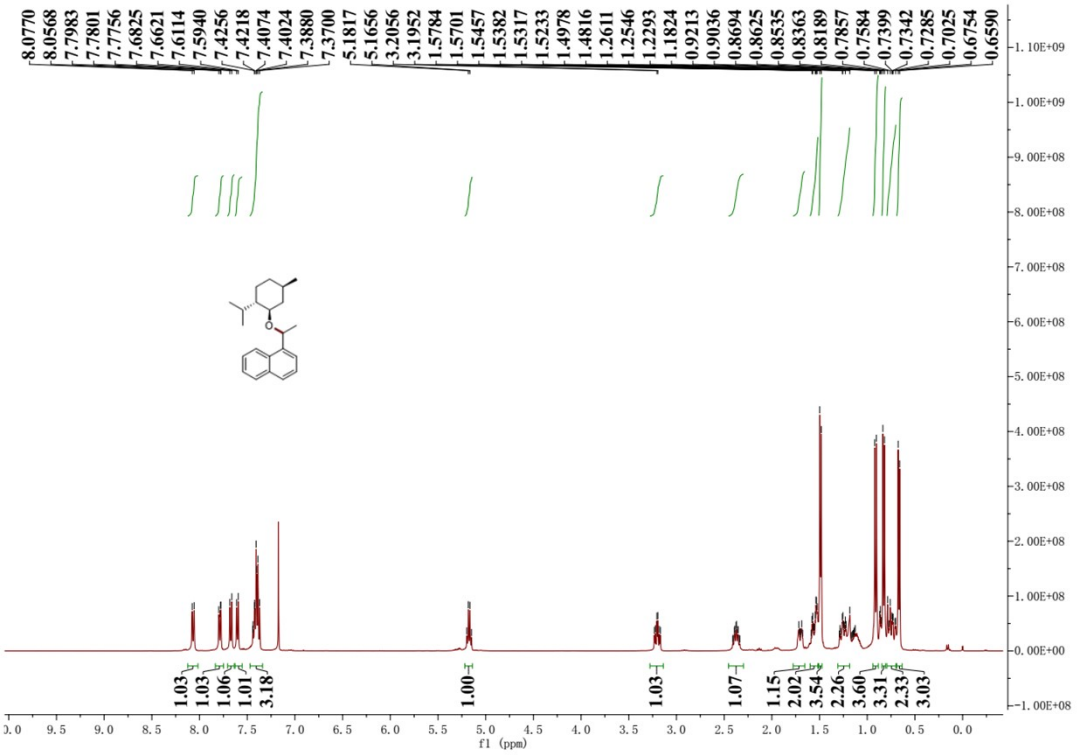
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2.33
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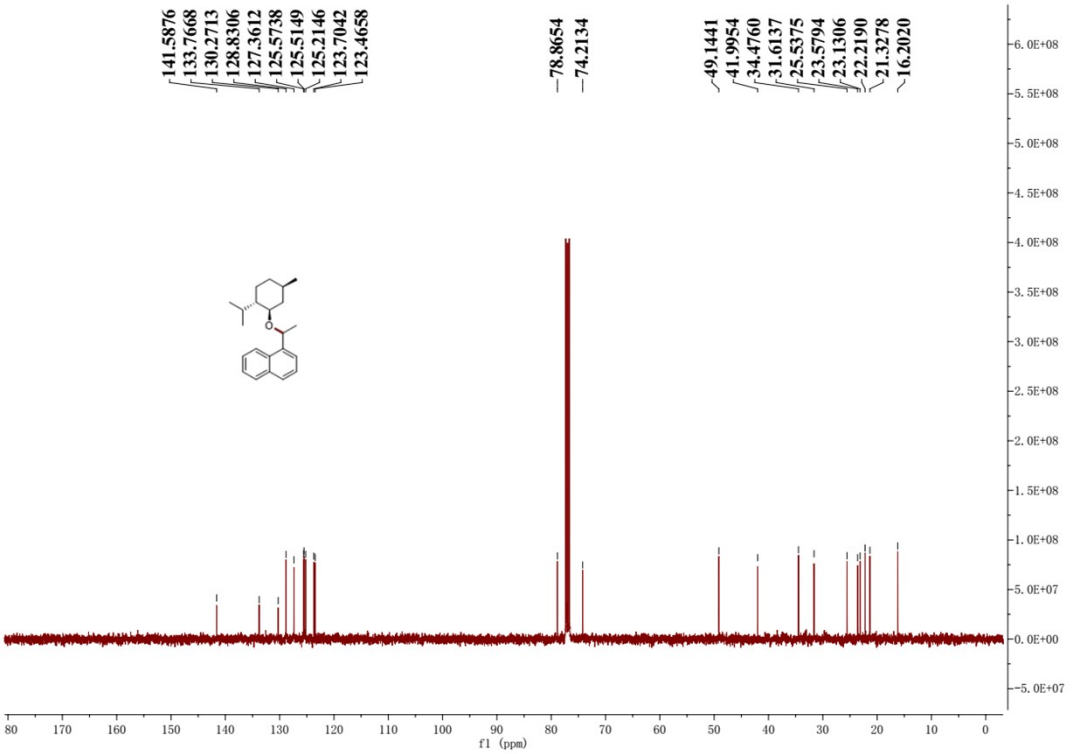
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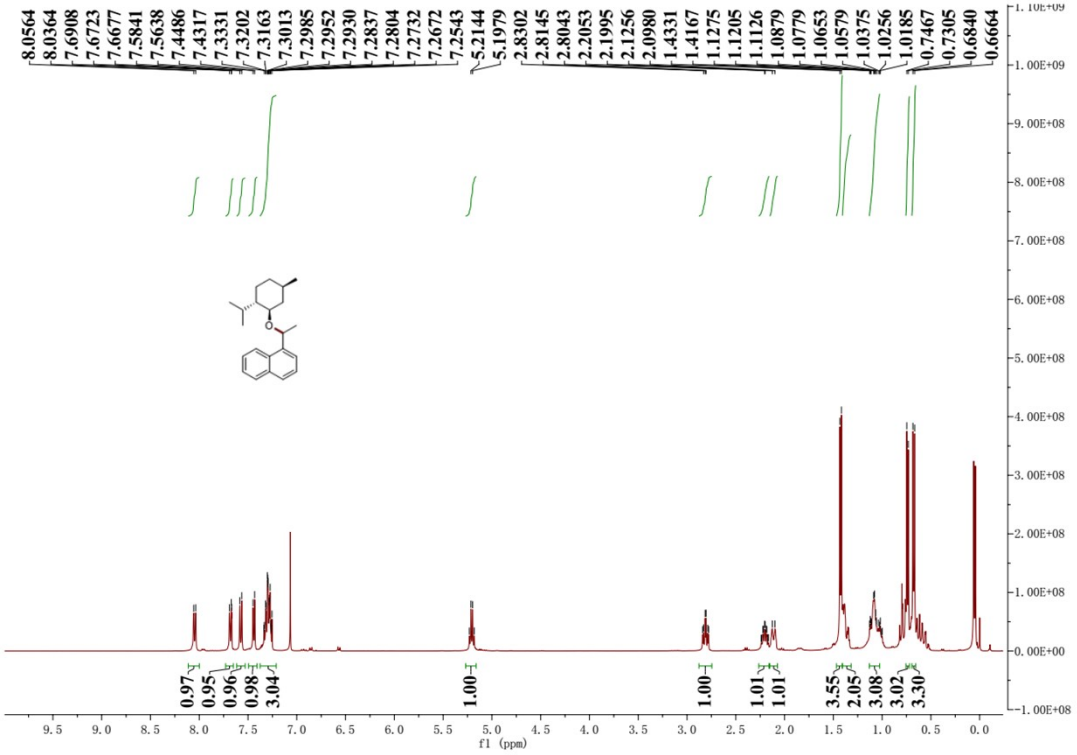
16.2020
f1 (ppm)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1.1275
1.1205
1.1126
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0.7305
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1.01
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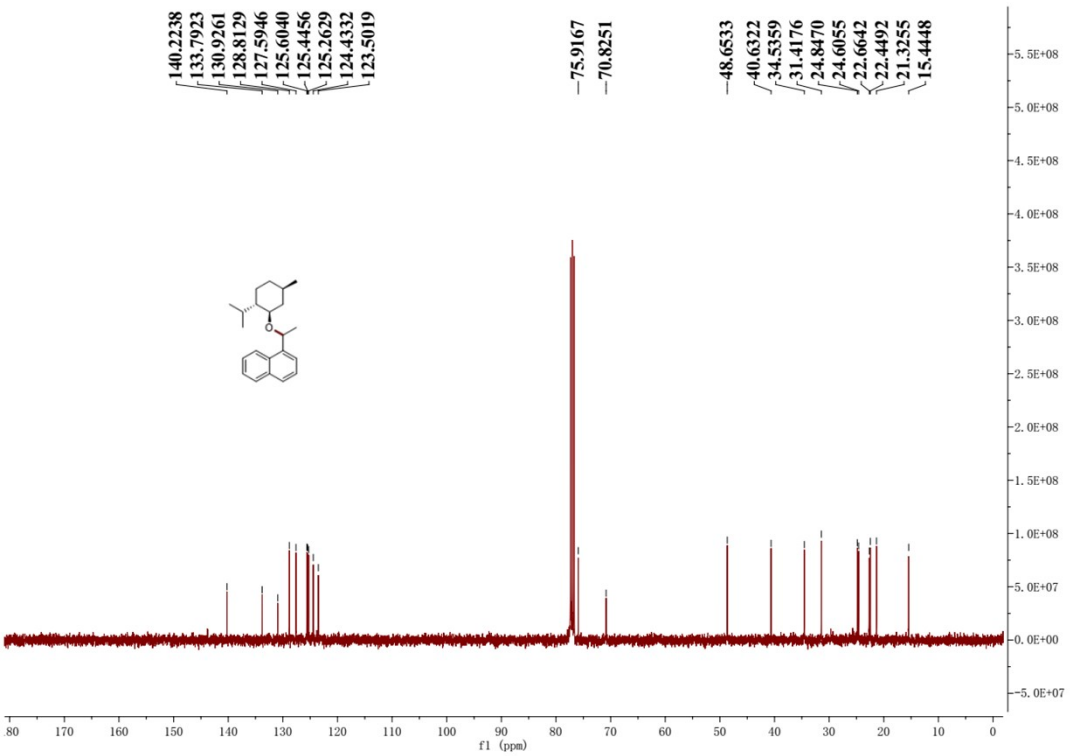
f1 (ppm)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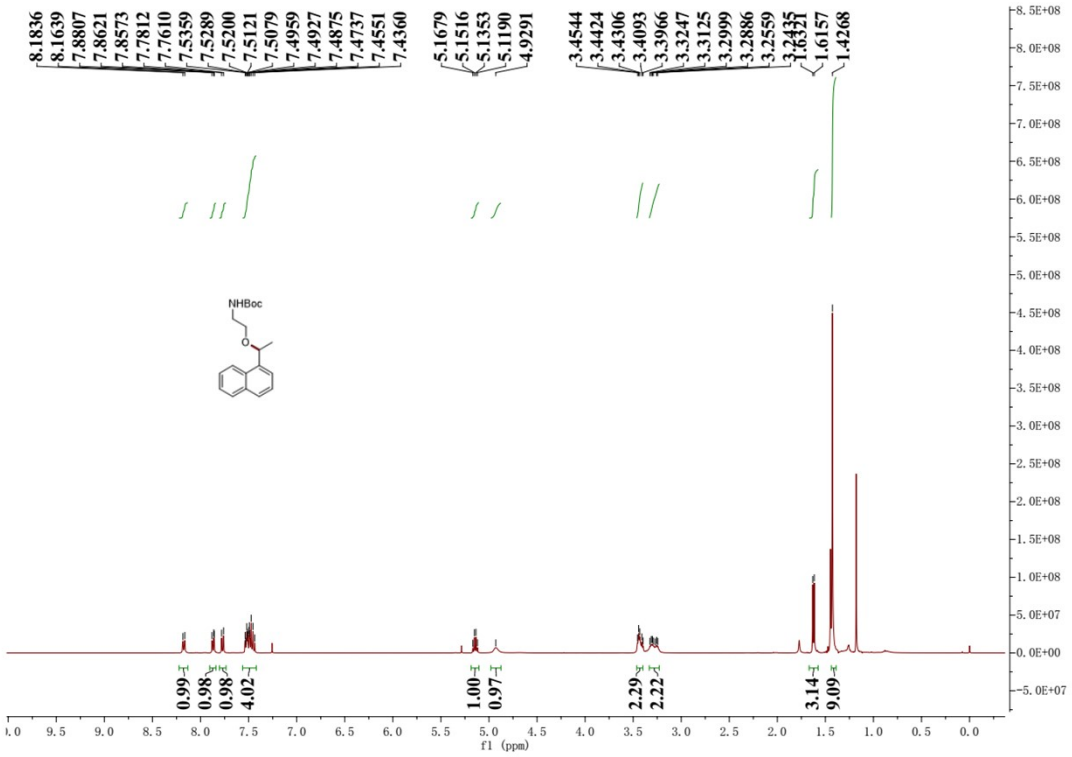
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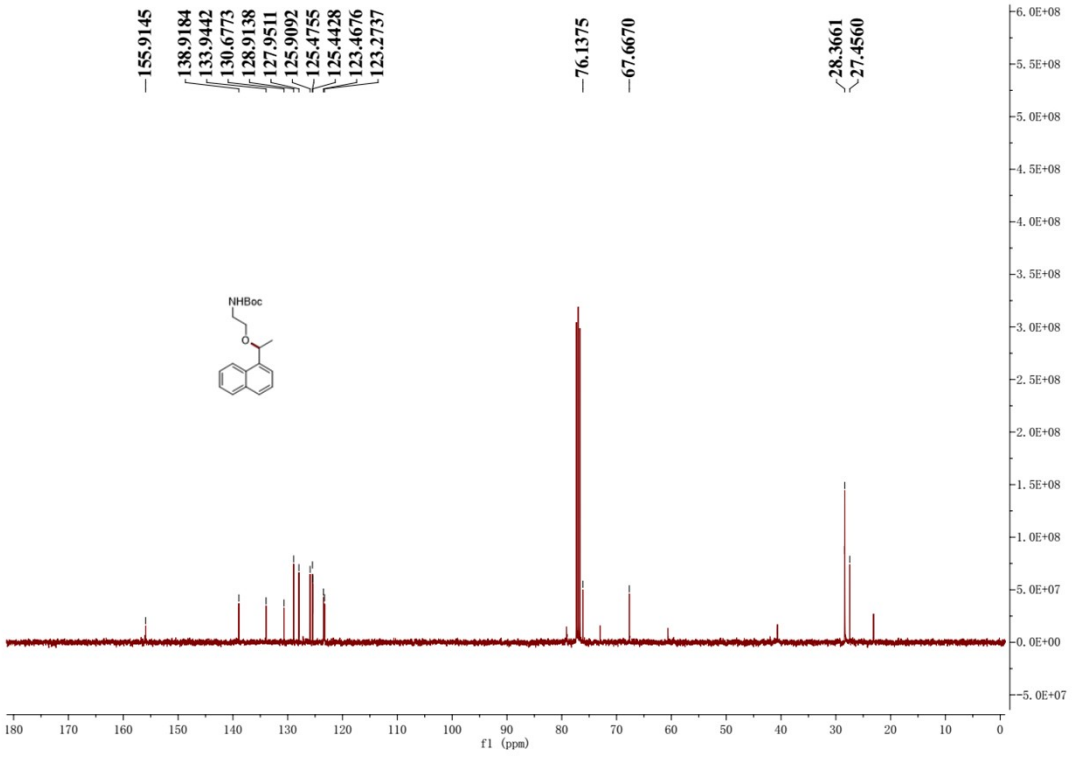
f1 (ppm)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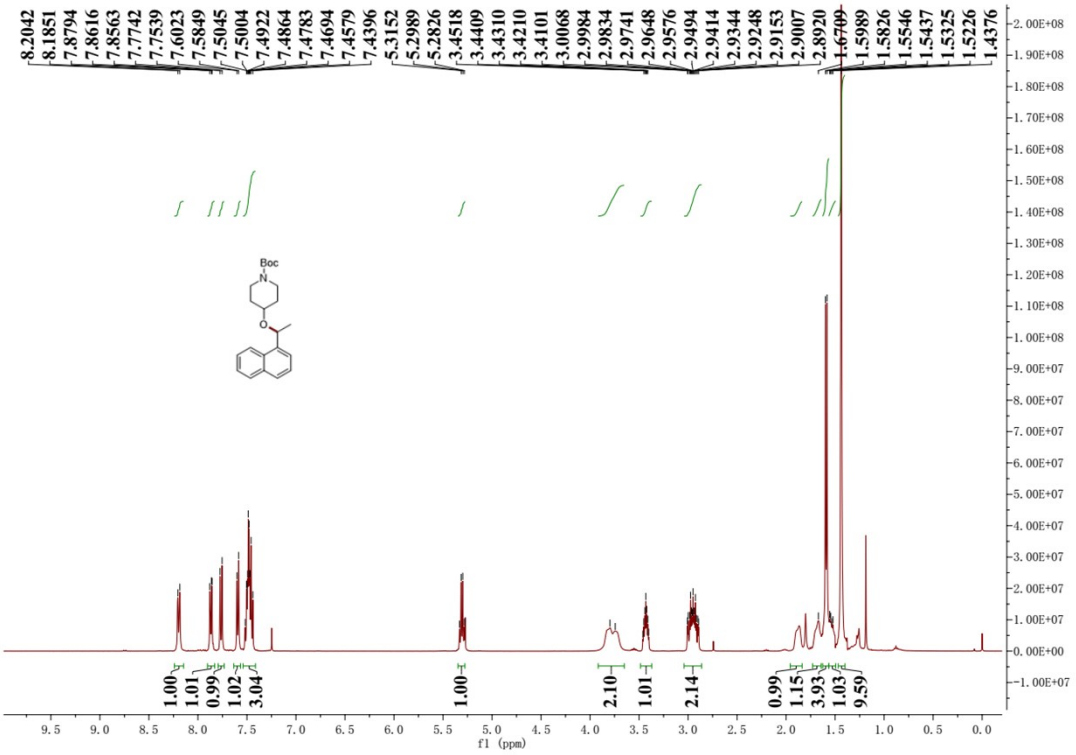
NHBoc
f1 (ppm)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7.4694
7.4579
7.4396
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3.4518
3.4409
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3.4210
3.4101
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Boc
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0.99
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1.01
2.14
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3.93
1.03
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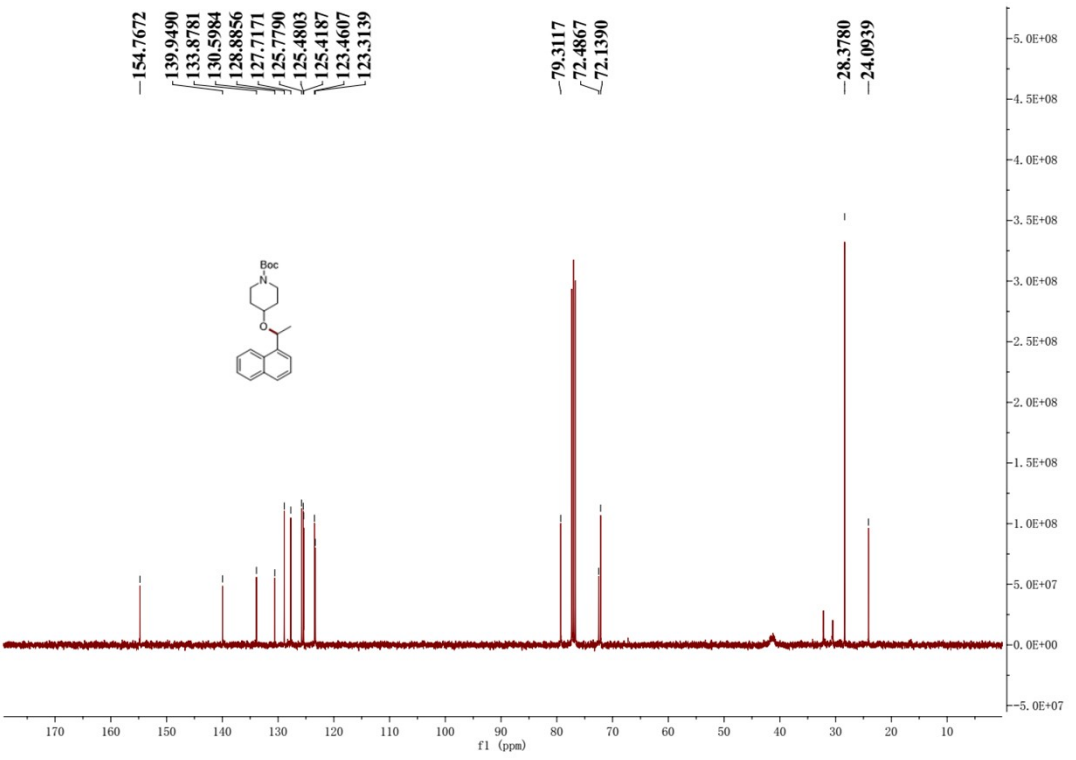
f1 (ppm)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28.3780
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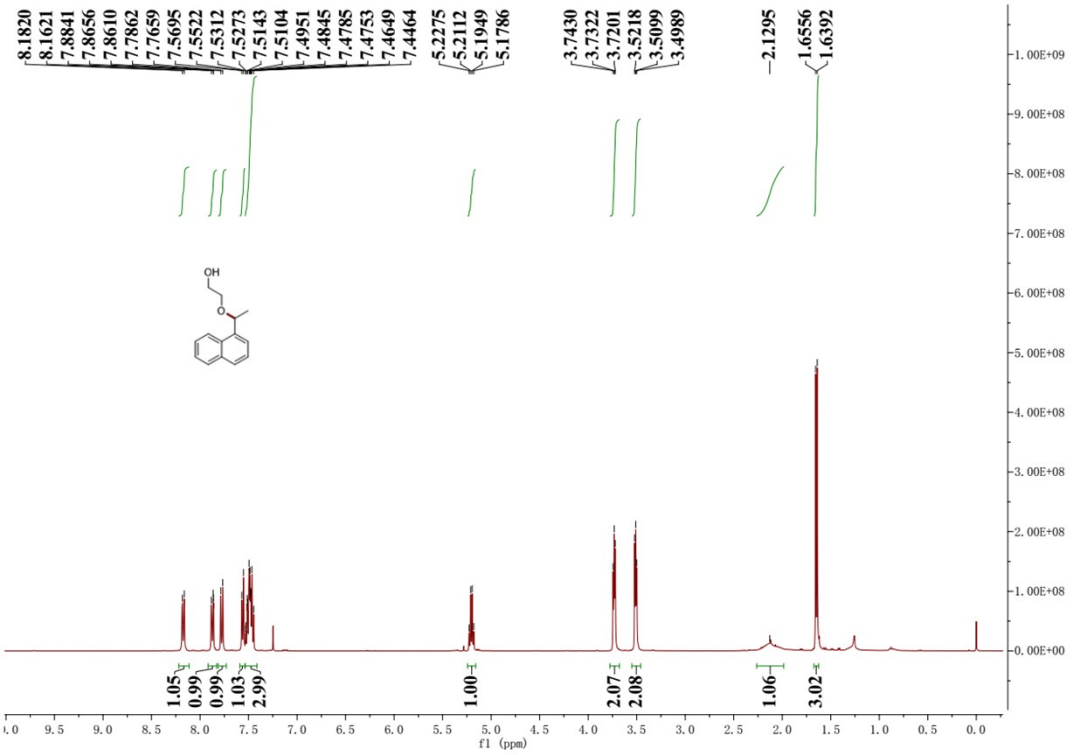
Boc
f1 (ppm)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7.5143
7.5104
7.4951
7.4845
7.4785
7.4753
7.4649
7.4464
5.2275
5.2112
5.1949
5.1786
3.7430
3.7322
3.7201
3.5218
3.5099
3.4989
2.1295
1.6556
1.6392
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0.99
1.03
2.99
1.00
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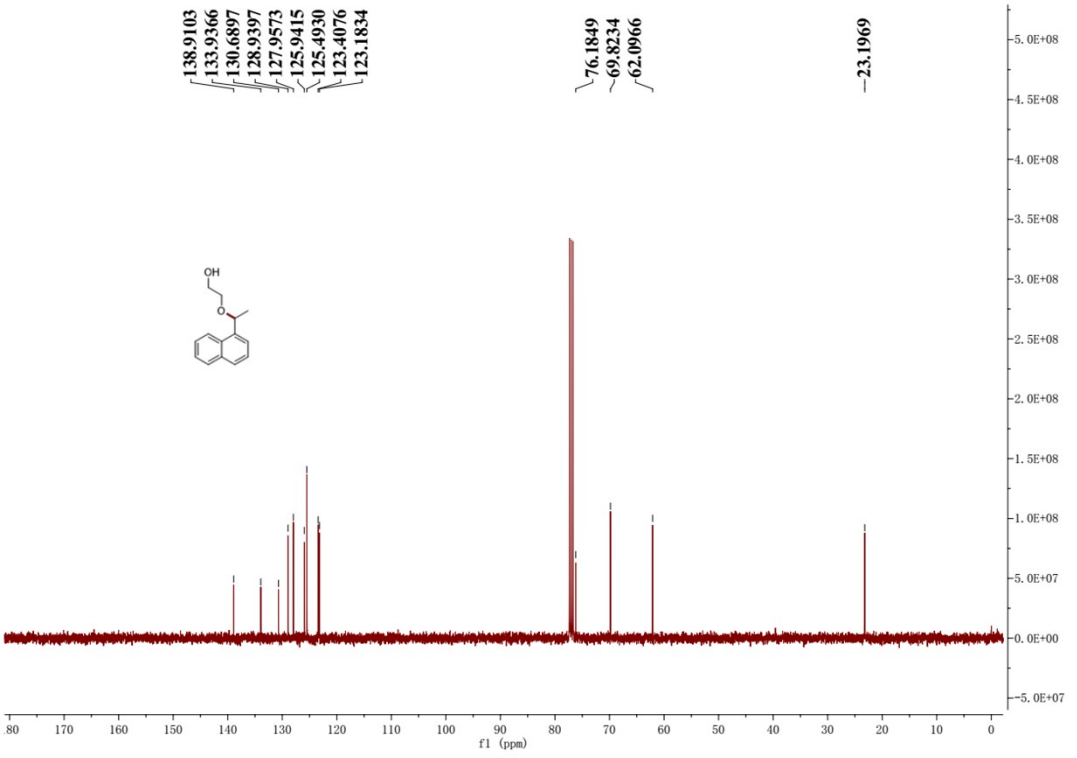
1.06
3.02
f1 (ppm)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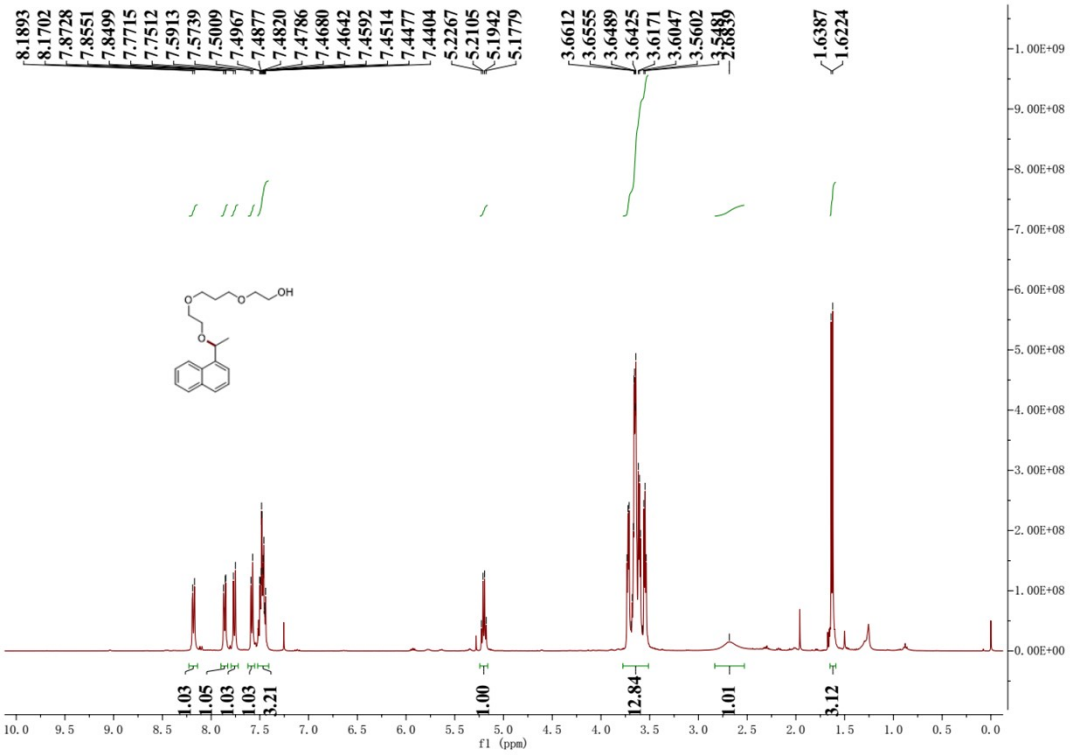
23.1969
OH
O
f1 (ppm)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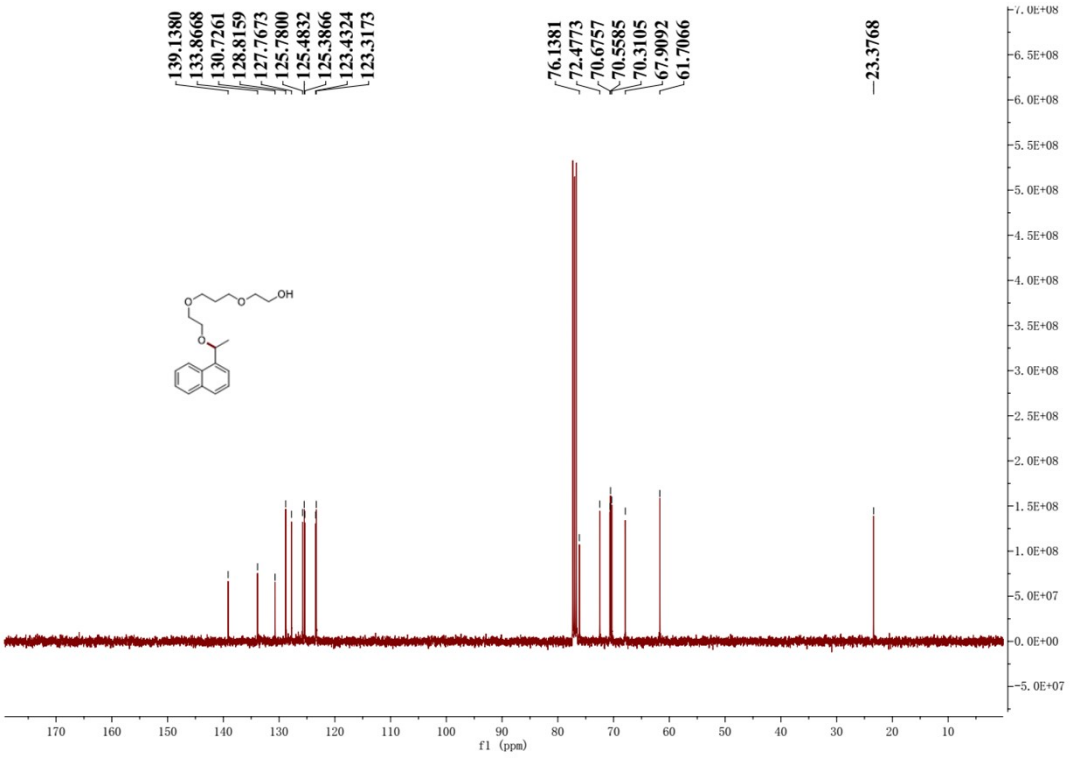
3.12
f1 (ppm)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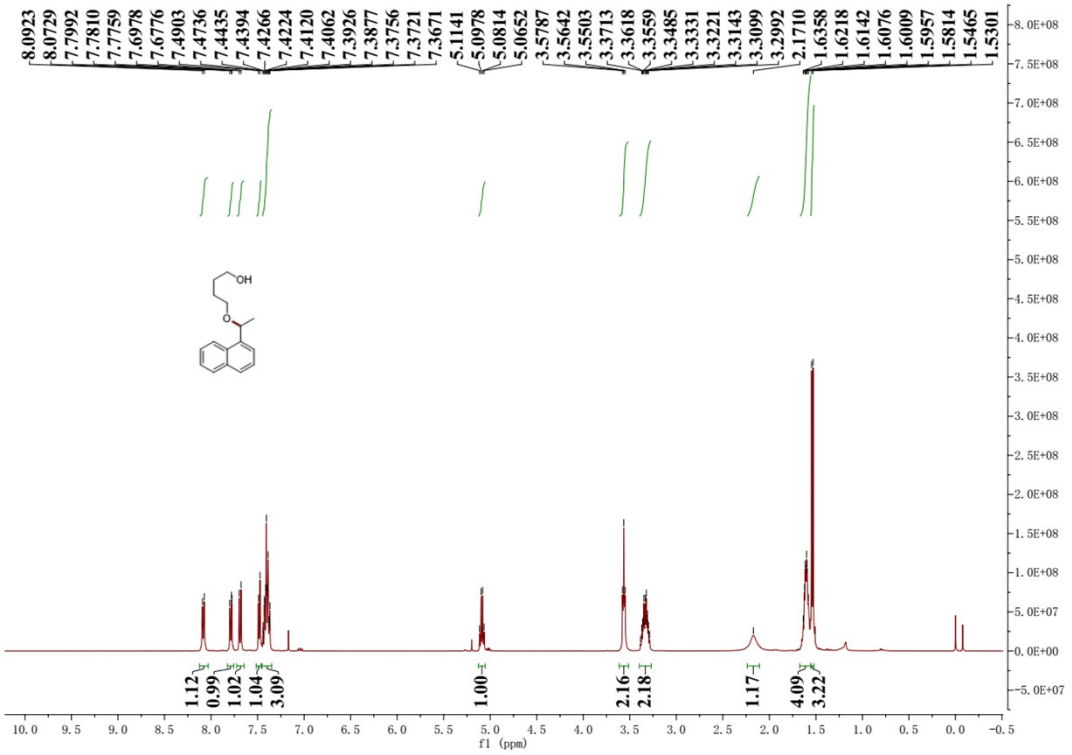
OH
f1 (ppm)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3.3221
3.3143
3.3099
3.2992
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OH
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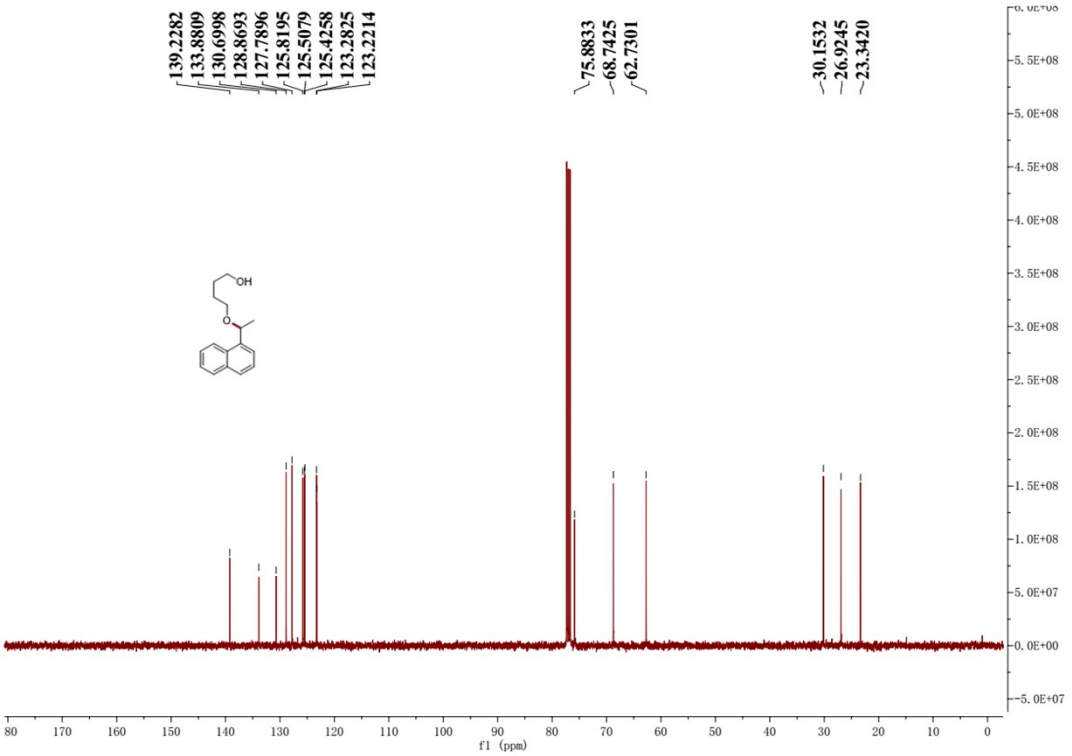
f1 (ppm)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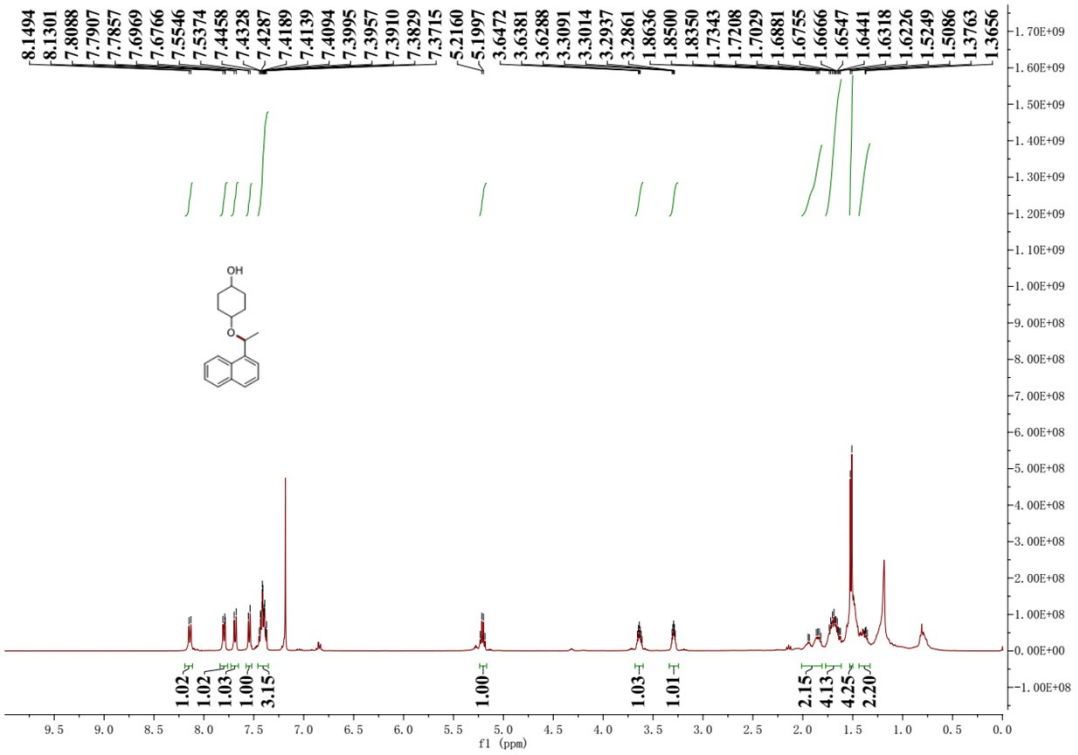
OH
f1 (ppm)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OH
O
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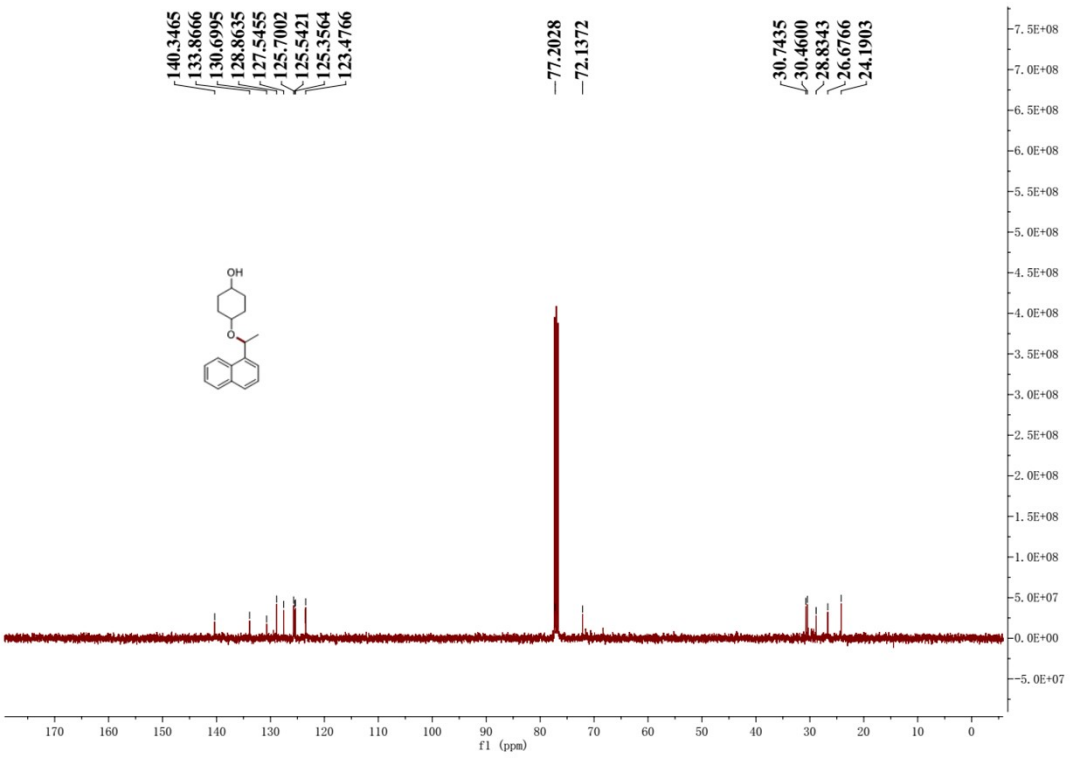
2.20
f1 (ppm)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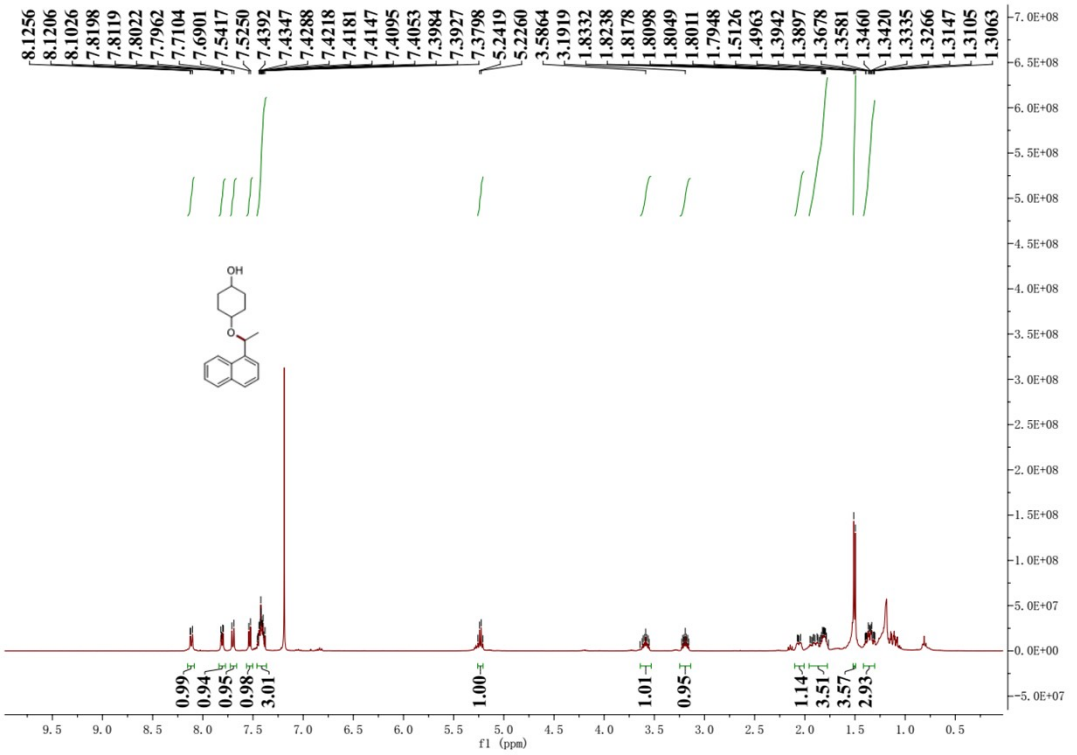
O
f1 (ppm)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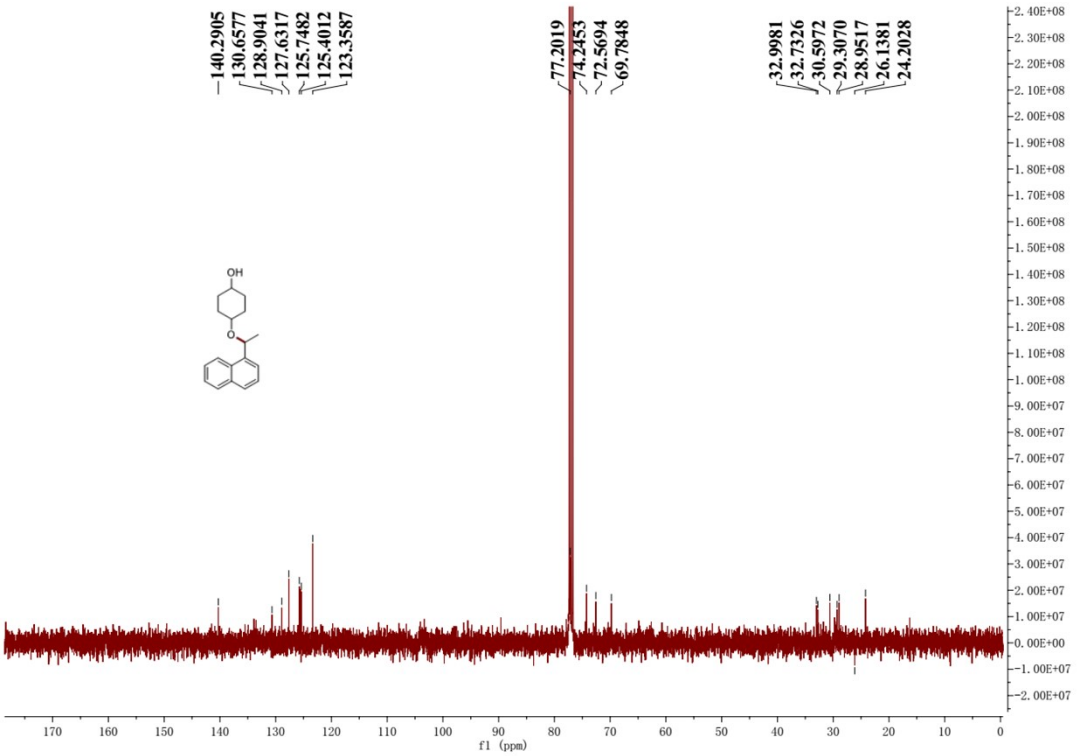
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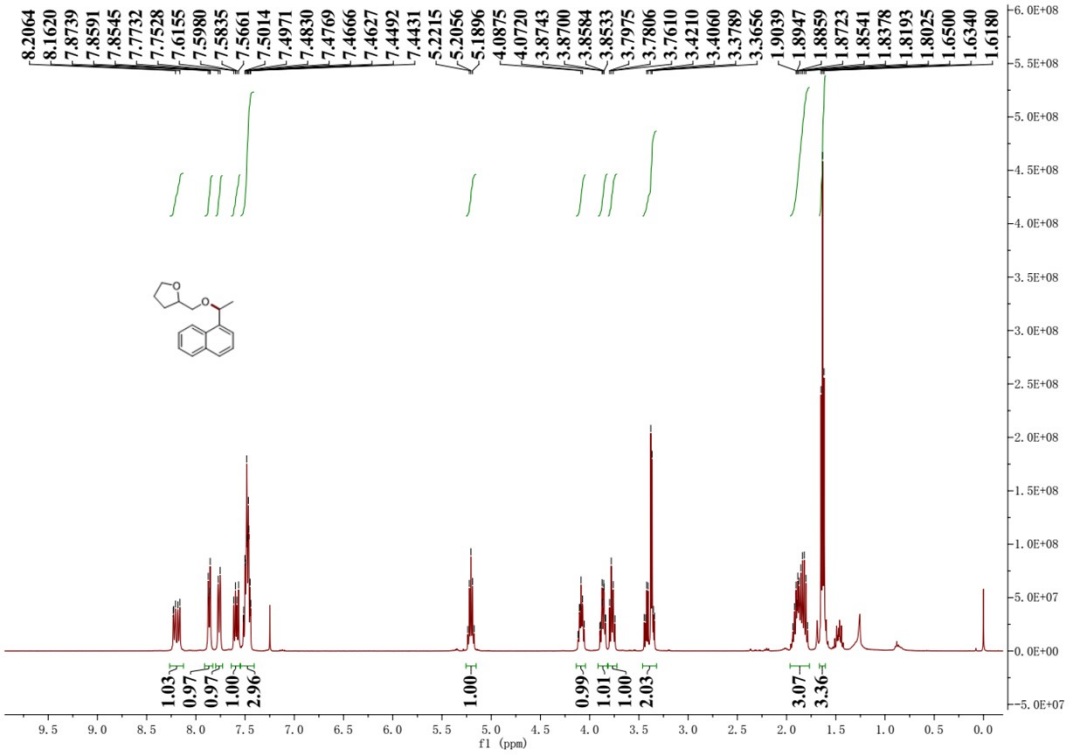
OH
O
f1 (ppm)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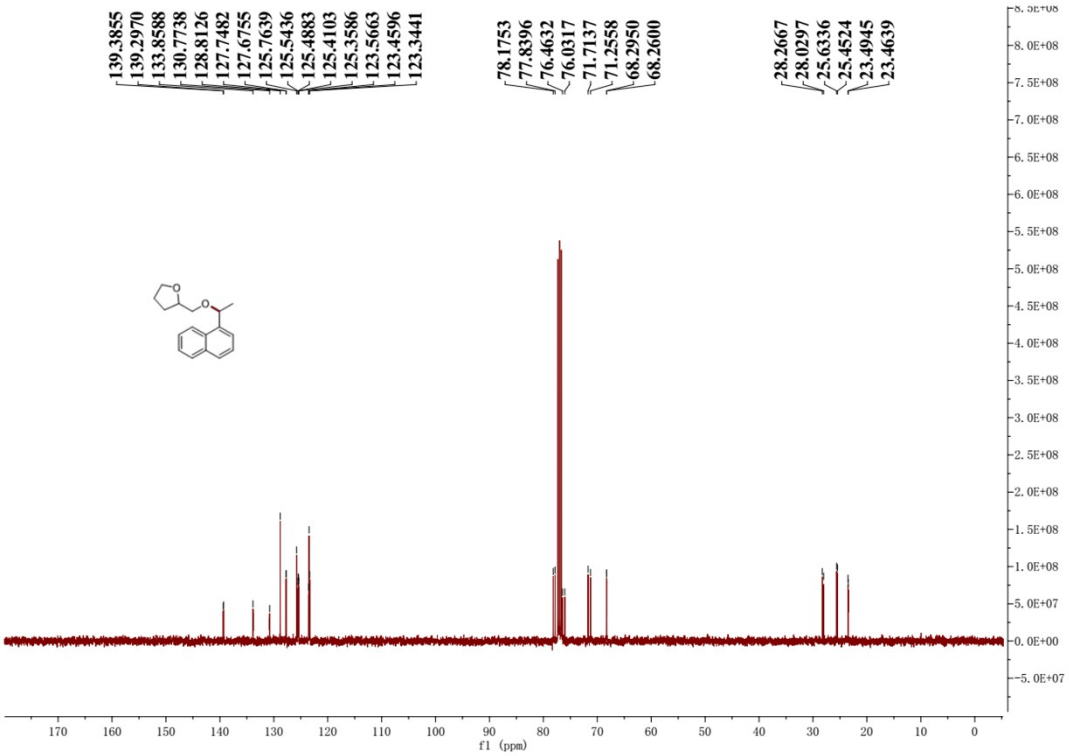
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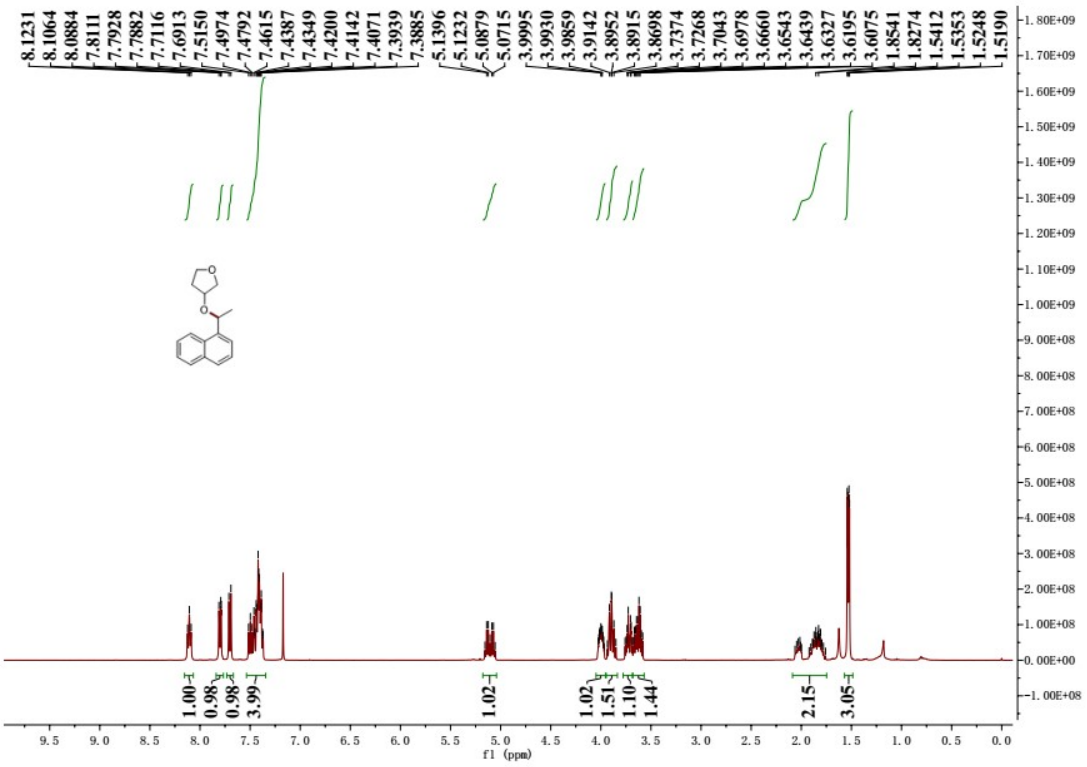
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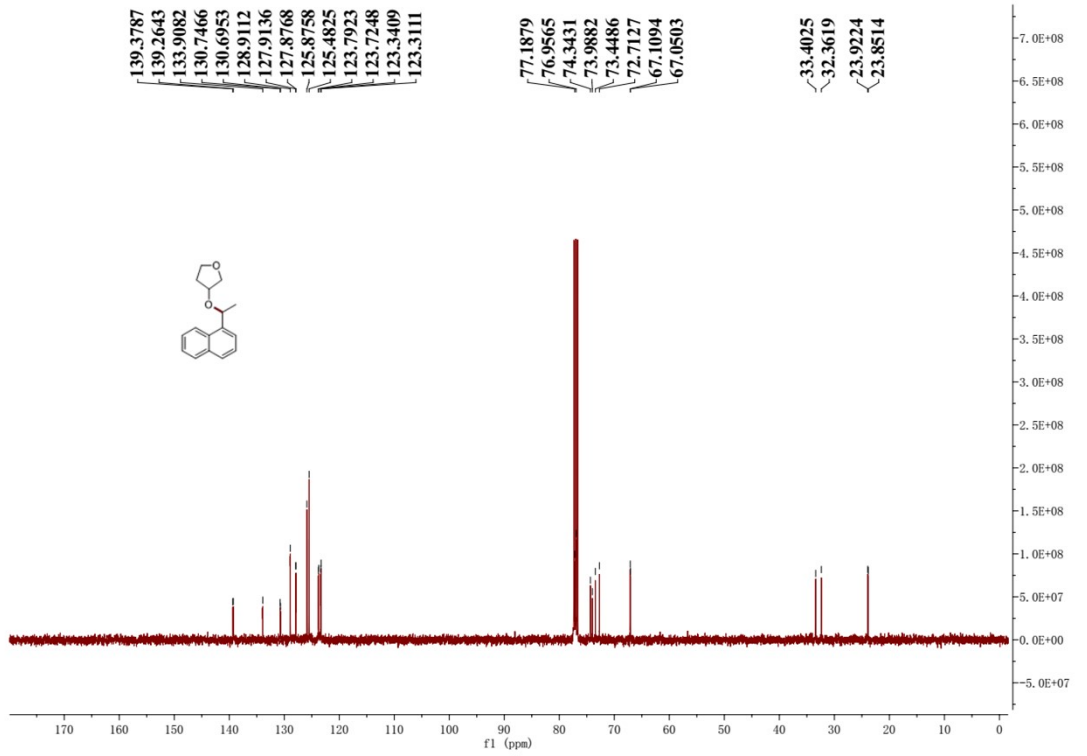
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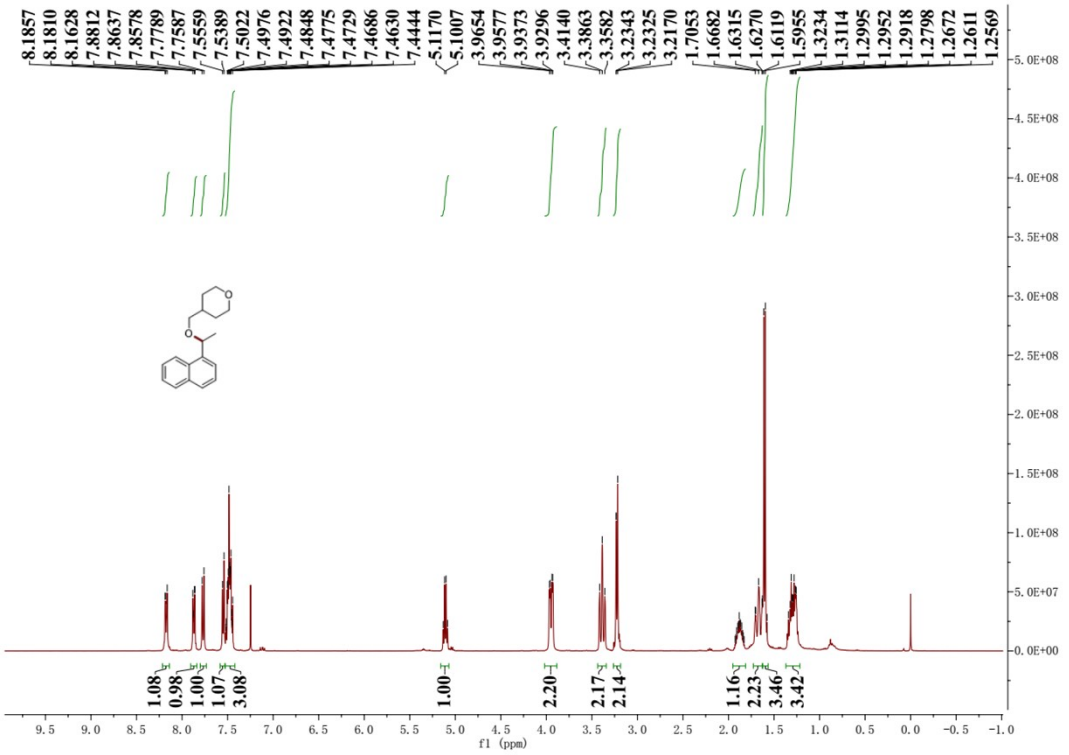
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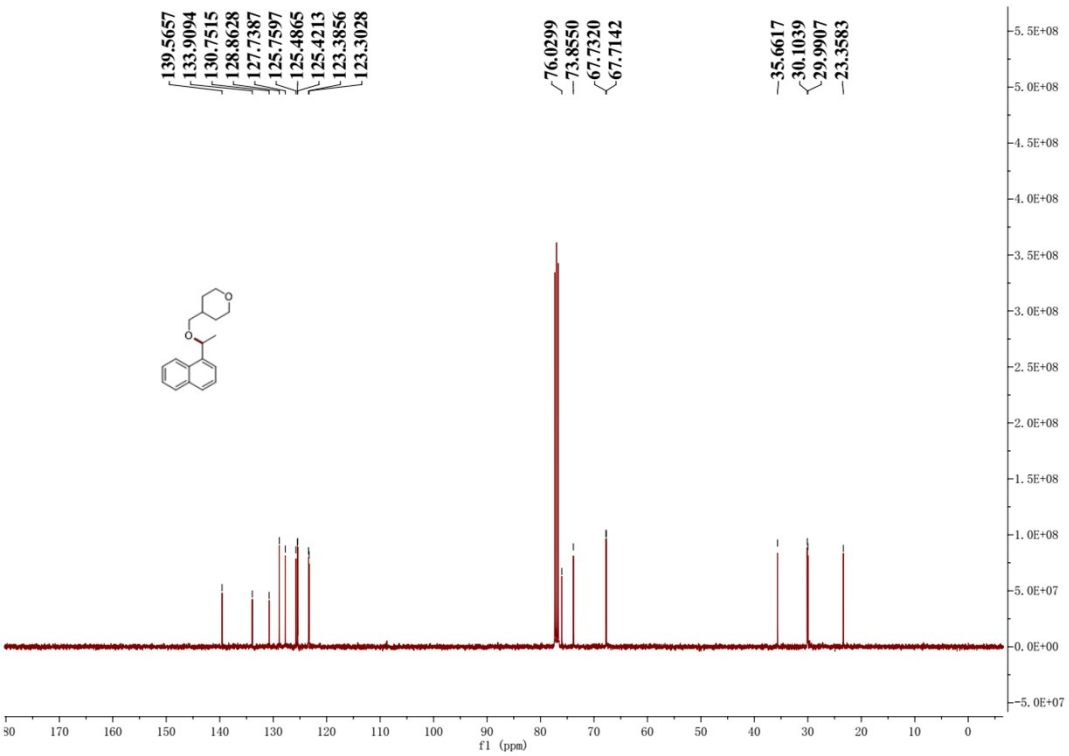
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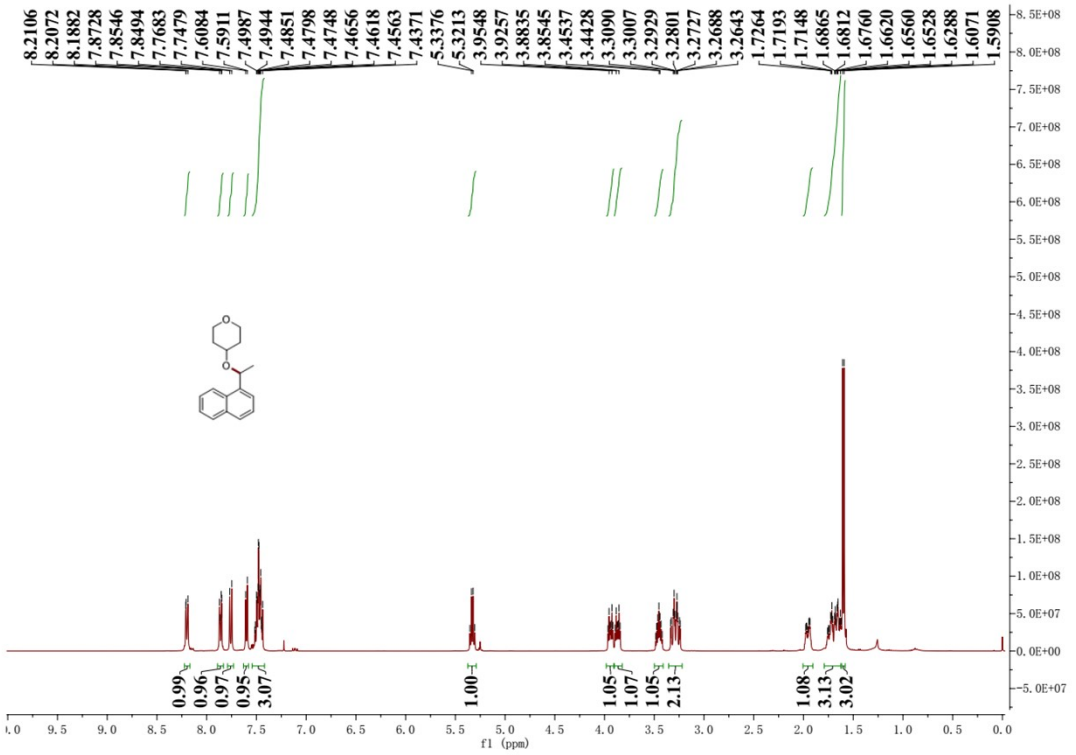
f1 (ppm)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5.3213
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1.07
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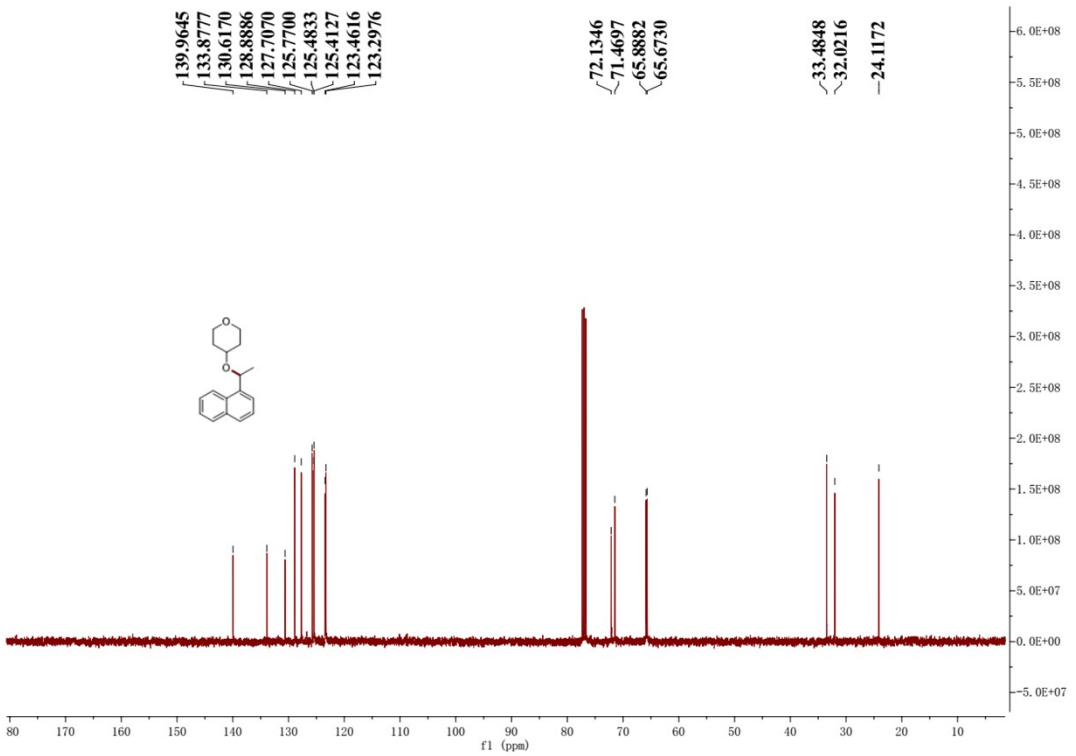
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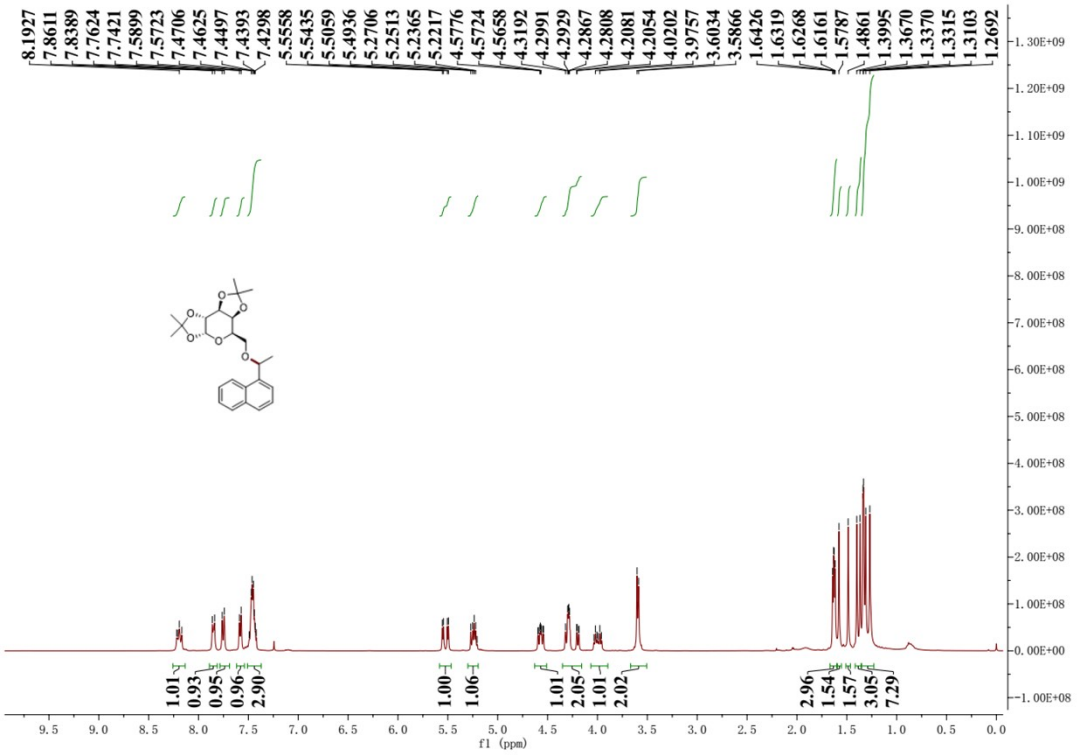
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4.2054
4.0202
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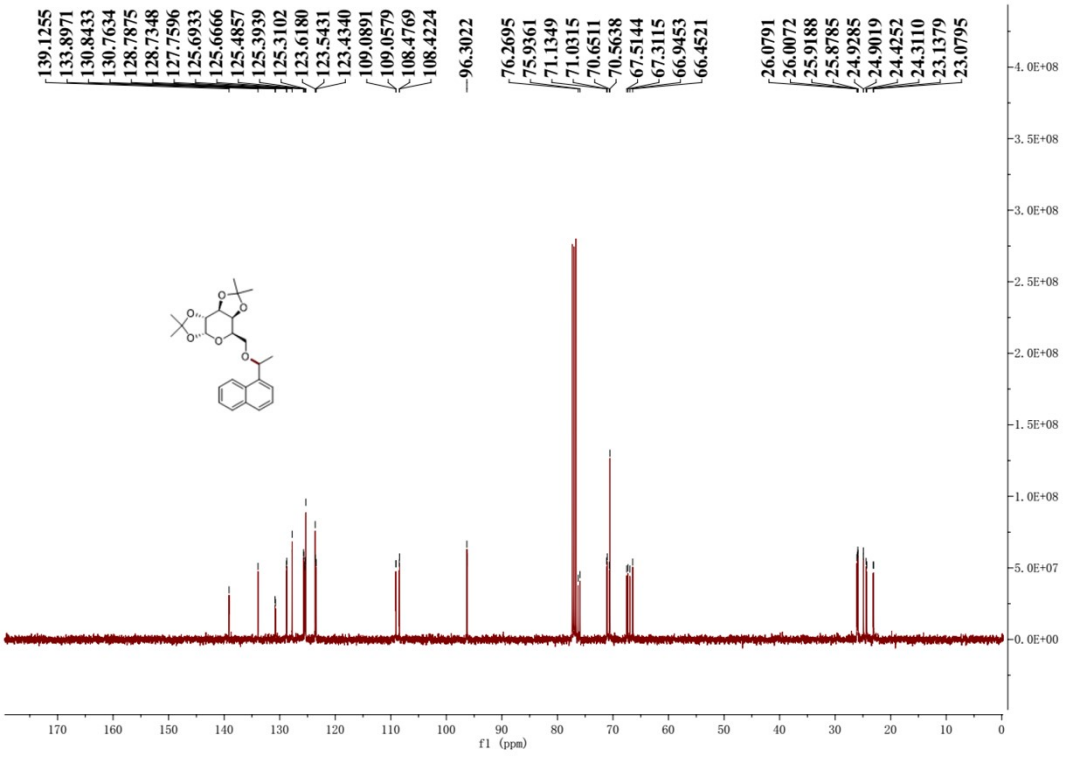
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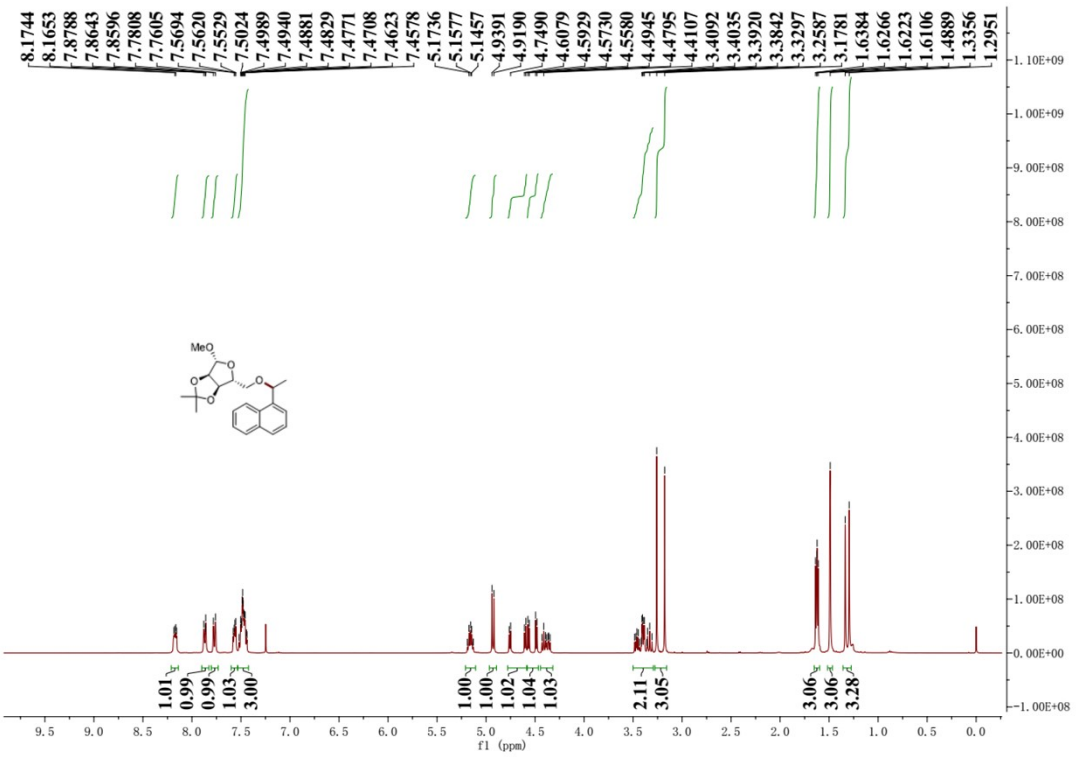
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1.6223
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1.2951
MeO
1.01
0.99
0.99
1.03
3.00
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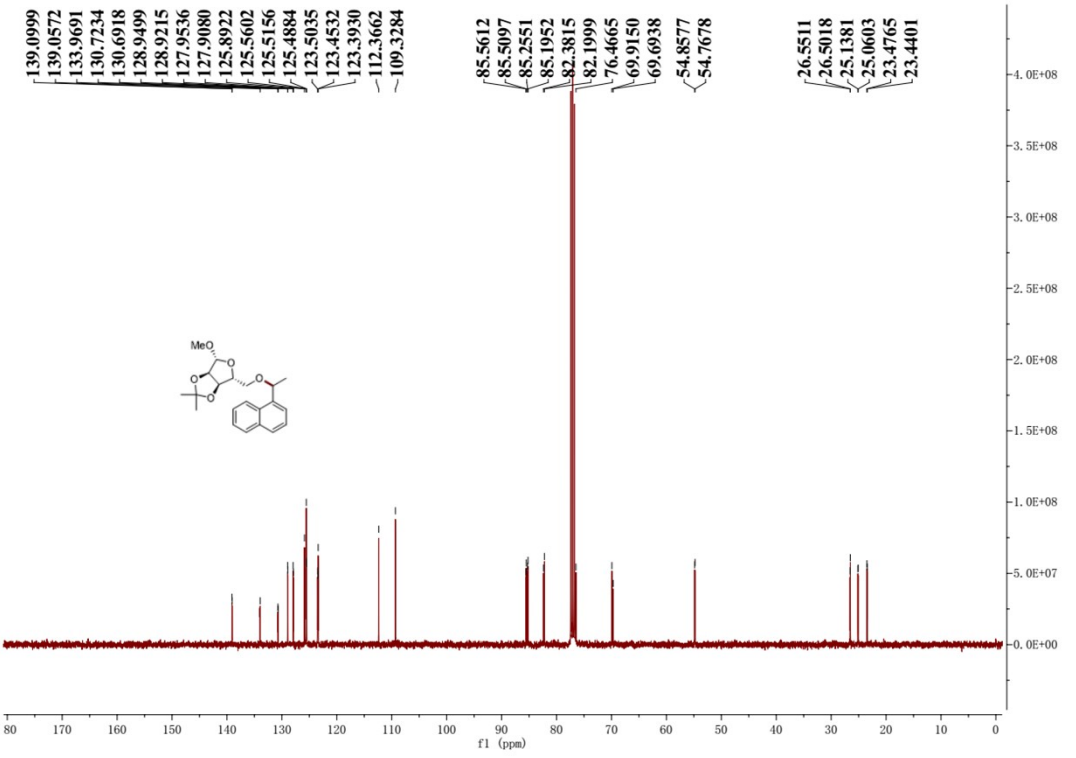
1.02
1.04
1.03
2.11
3.05
3.06
3.06
3.28
f1 (ppm)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85.5097
85.2551
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82.3815
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76.4665
69.9150
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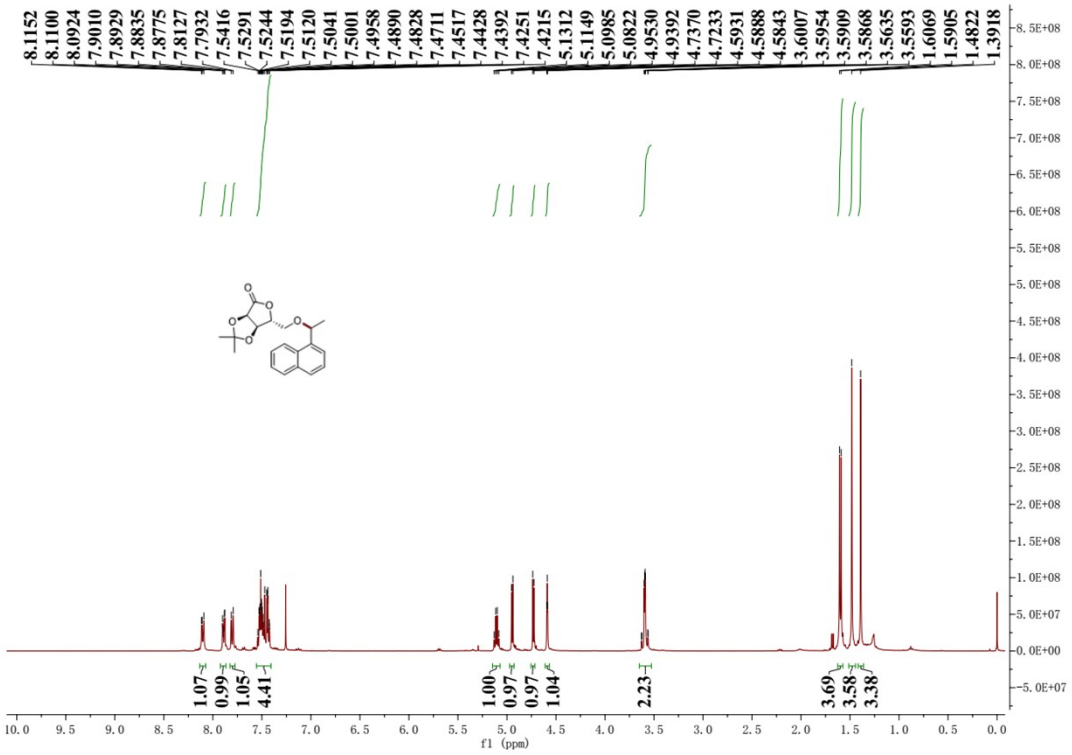
MeO
f1 (ppm)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7.4828
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7.4517
7.4428
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7.4251
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1.07
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1.04
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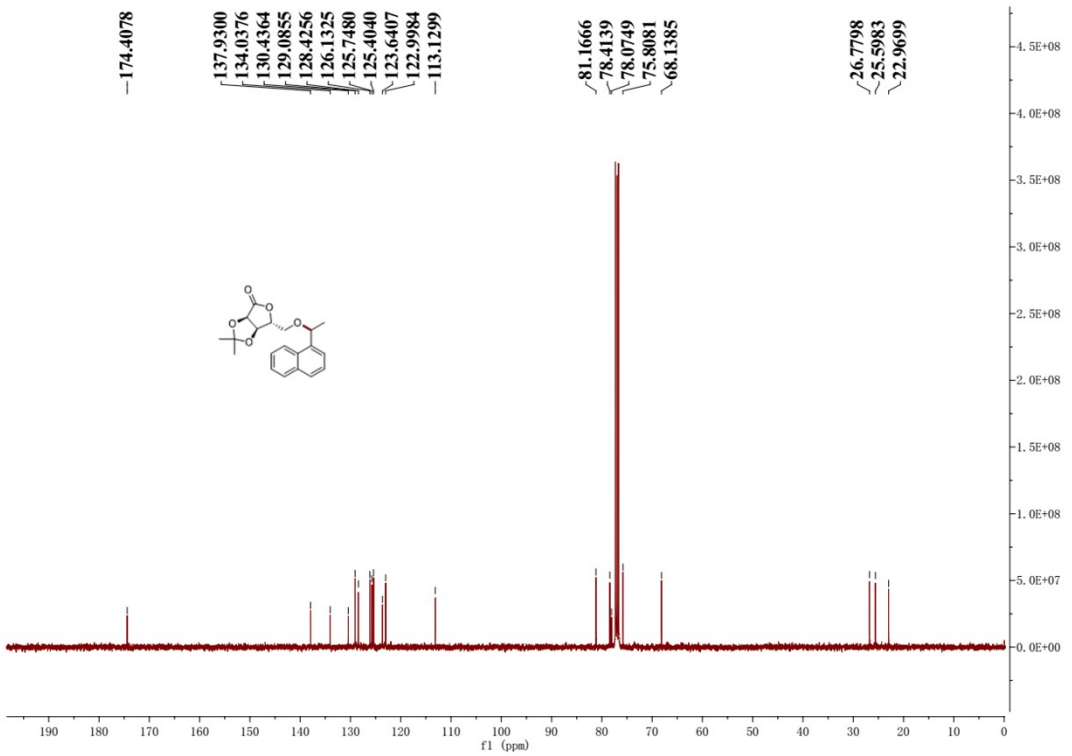
3.38
f1 (ppm)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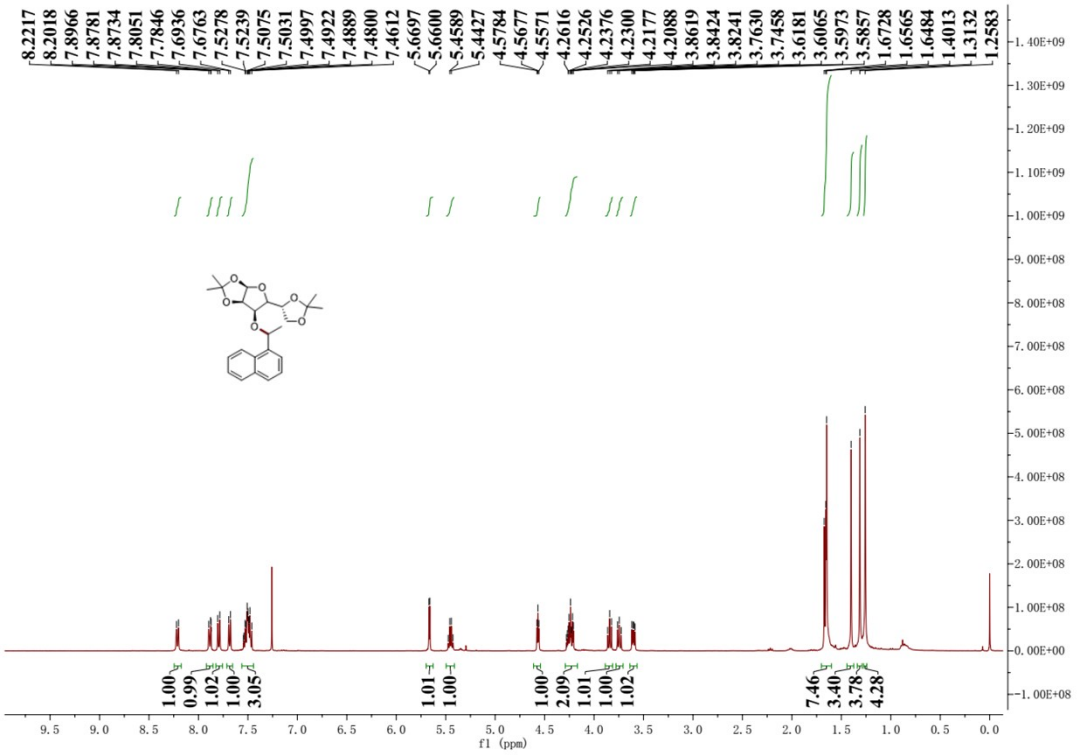
22.9699
f1 (ppm)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1.2583
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3.05
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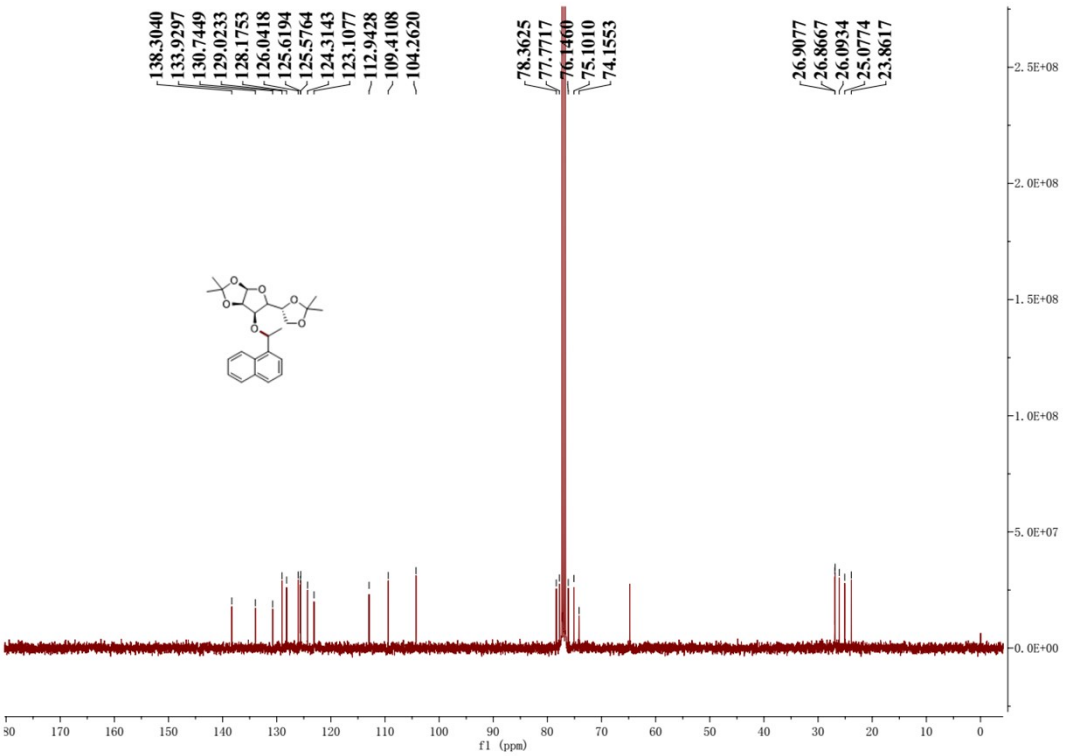
f1 (ppm)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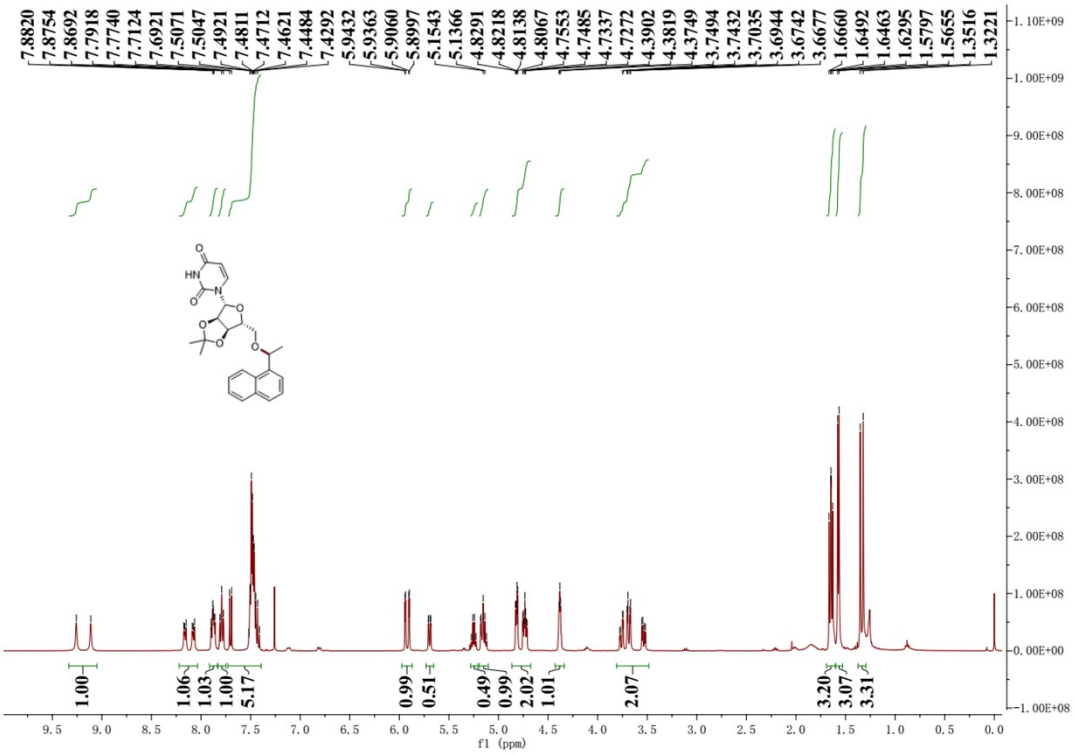
23.8617
f1 (ppm)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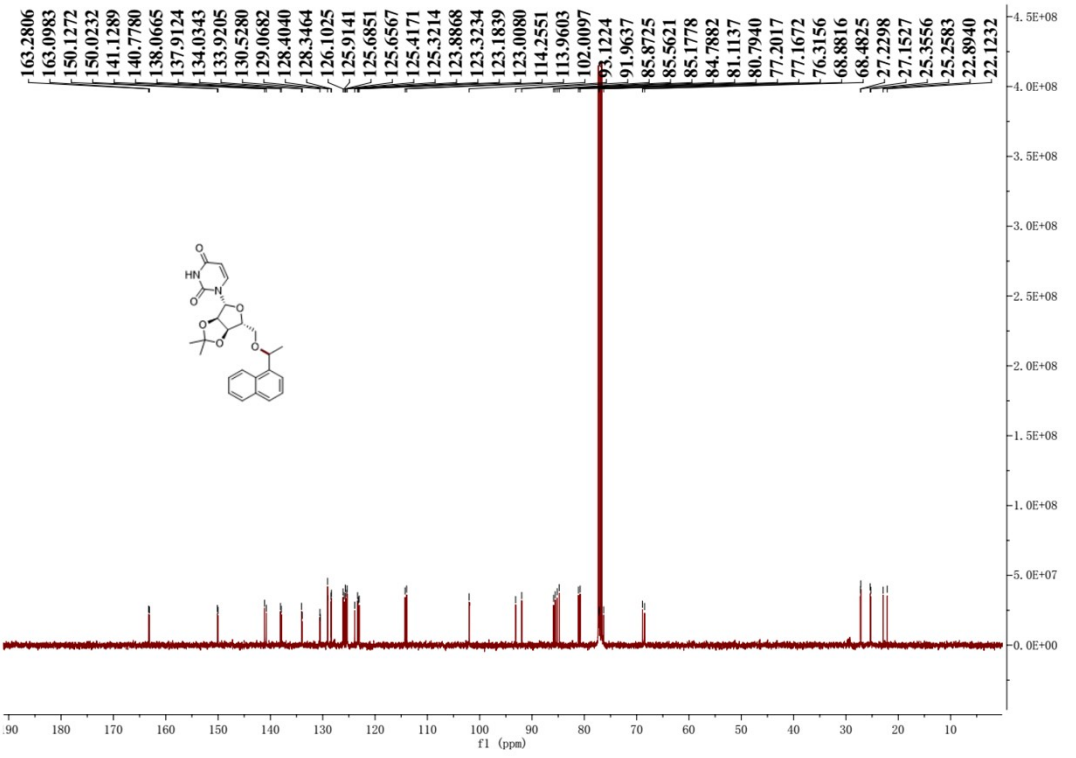
3.31
f1 (ppm)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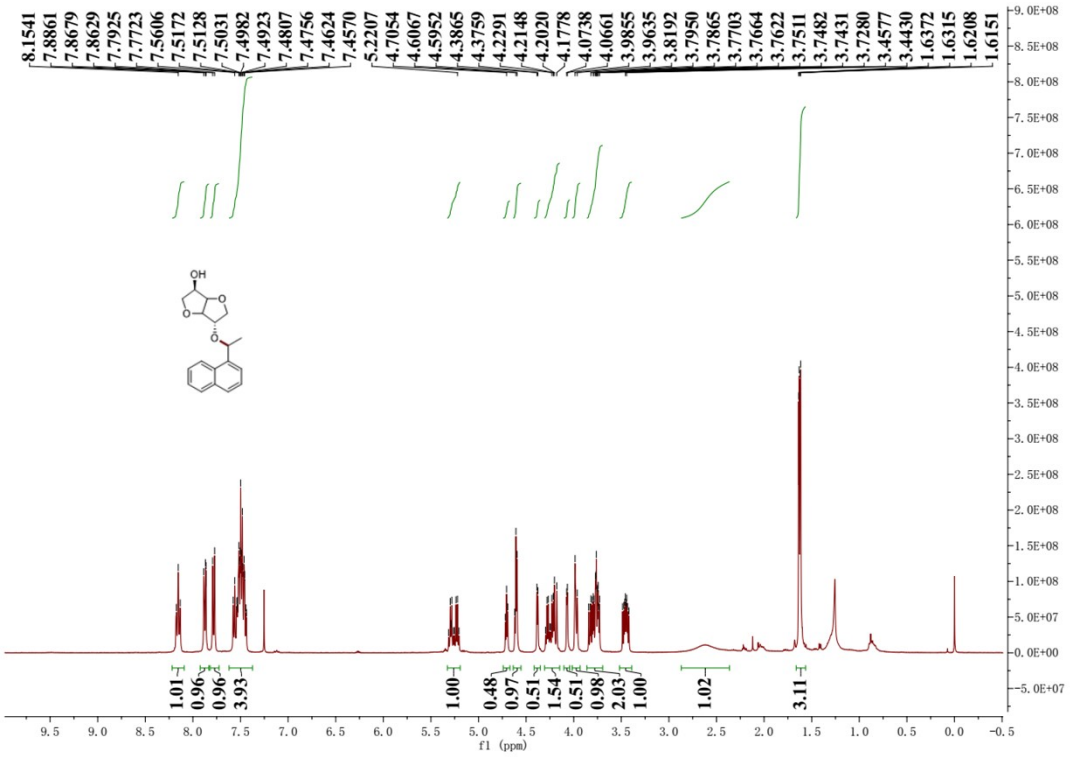
f1 (ppm)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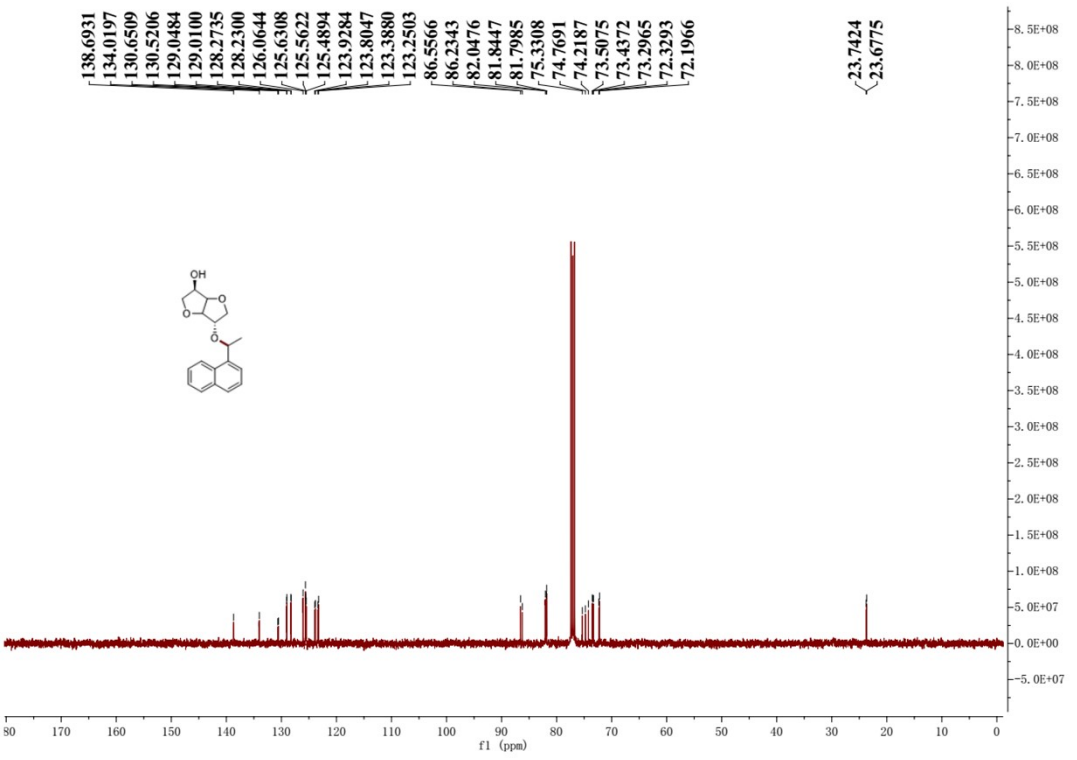
3.11
f1 (ppm)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23.7424
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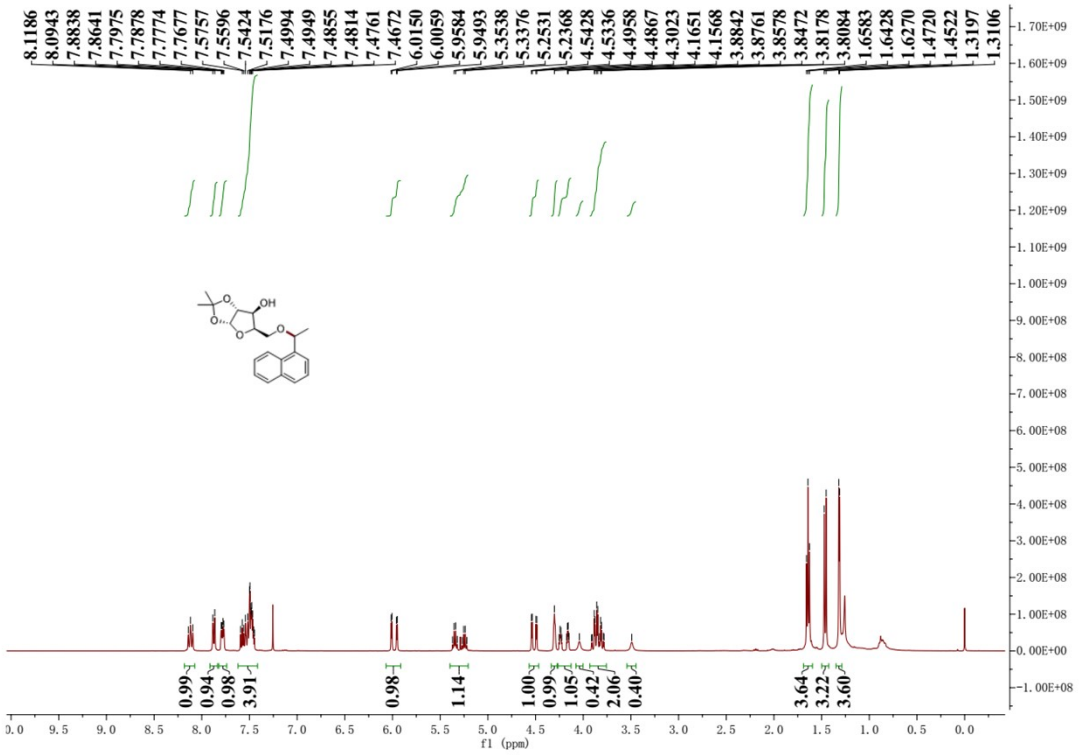
f1 (ppm)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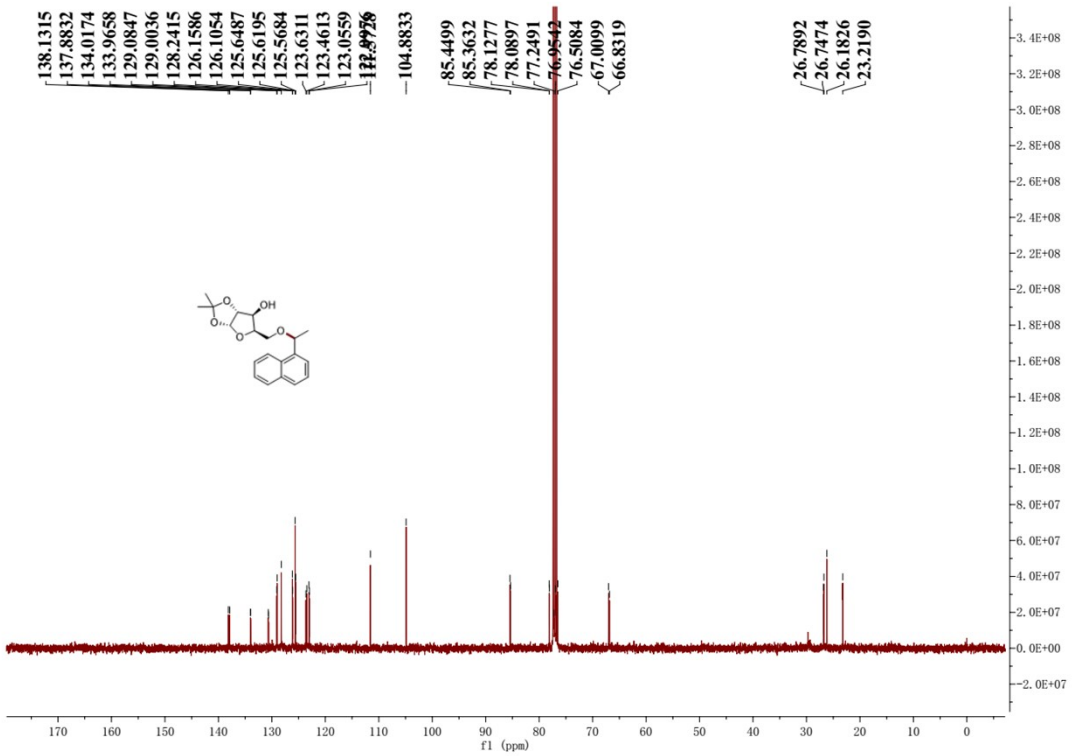
3.22
3.60
f1 (ppm)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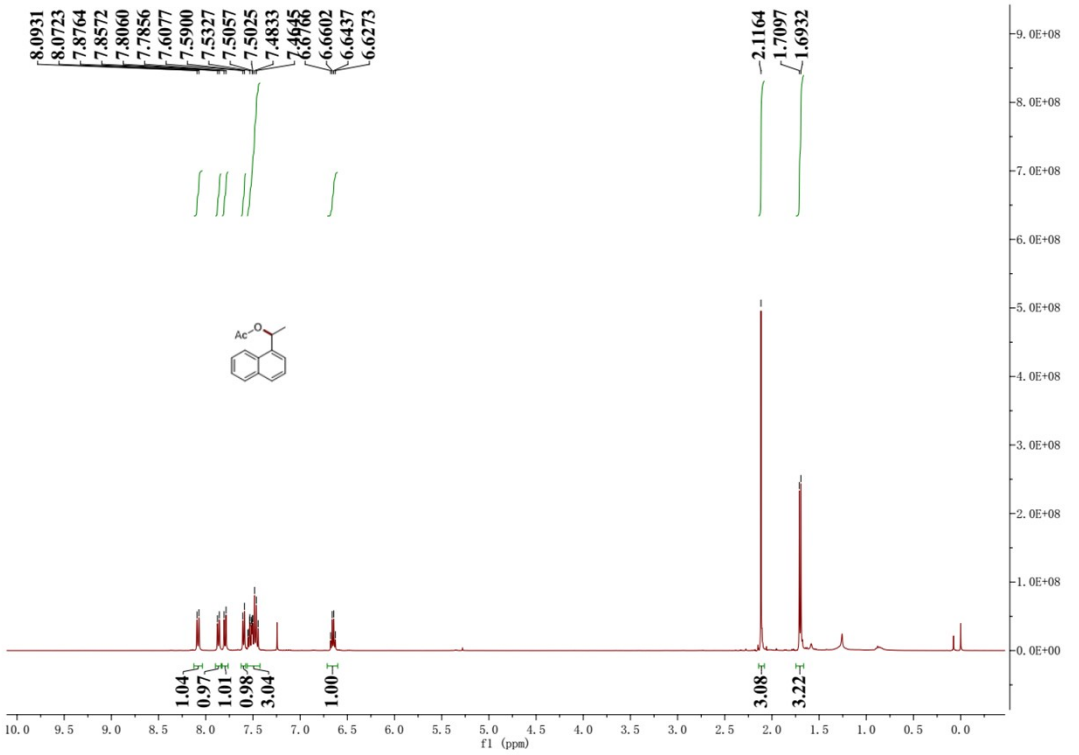
f1 (ppm)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7.4833
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6.6437
6.6273
2.1164
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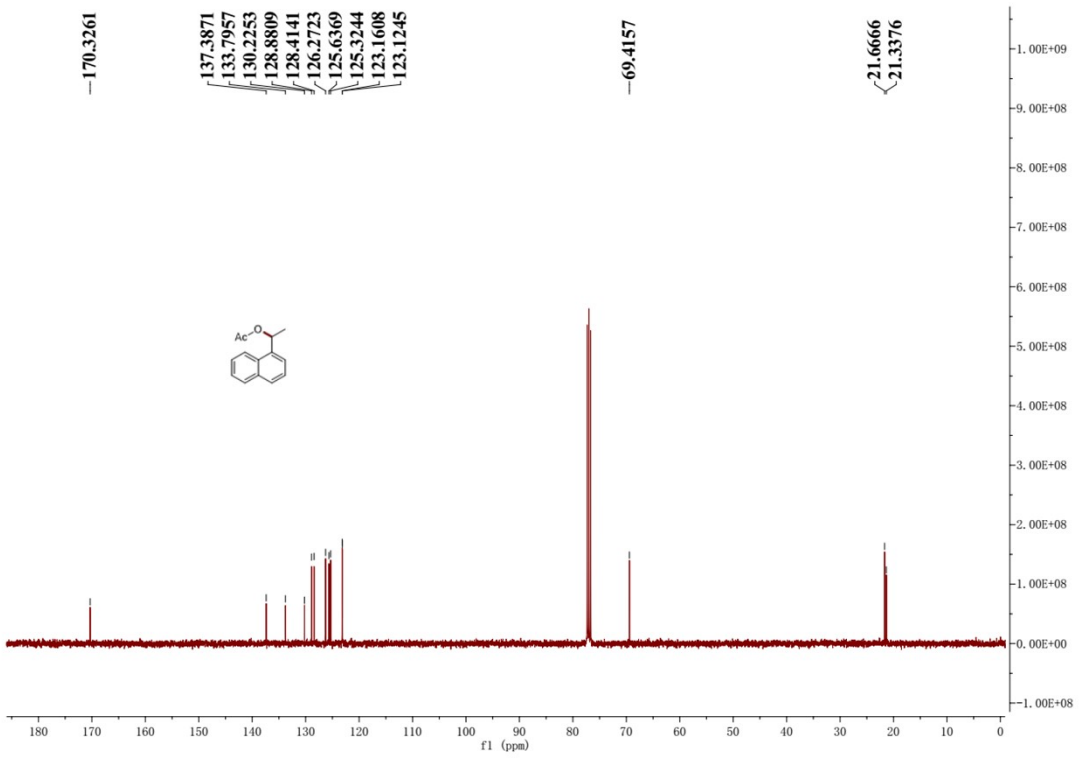
3.22
f1 (ppm)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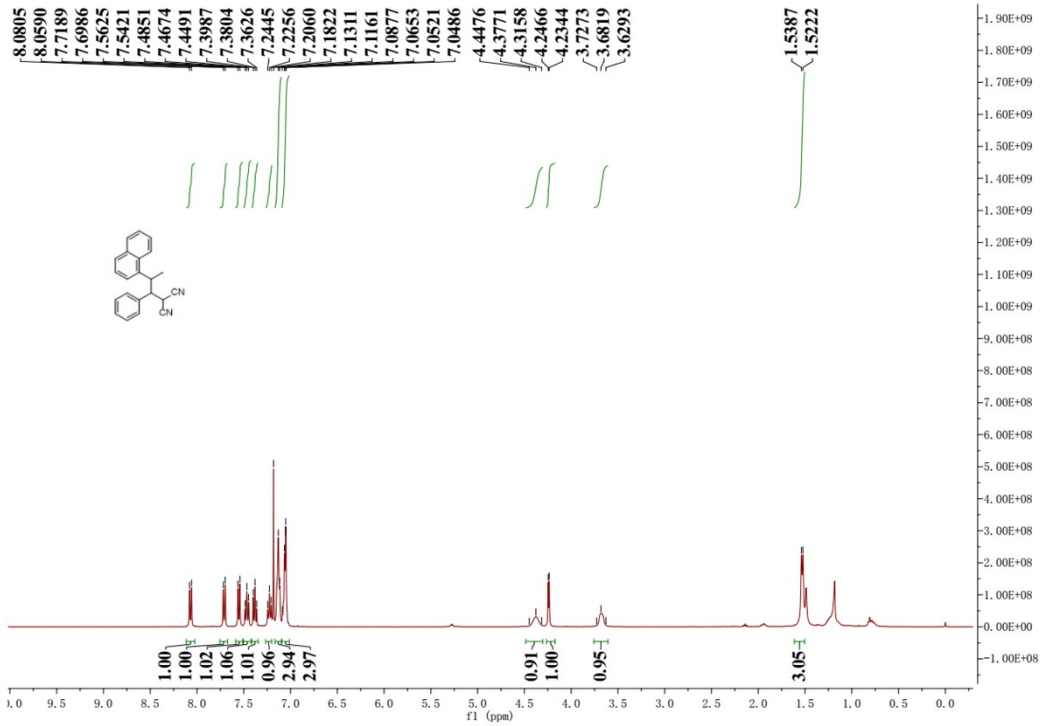
f1 (ppm)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7.2445
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3.6819
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1.5222
CN
CN
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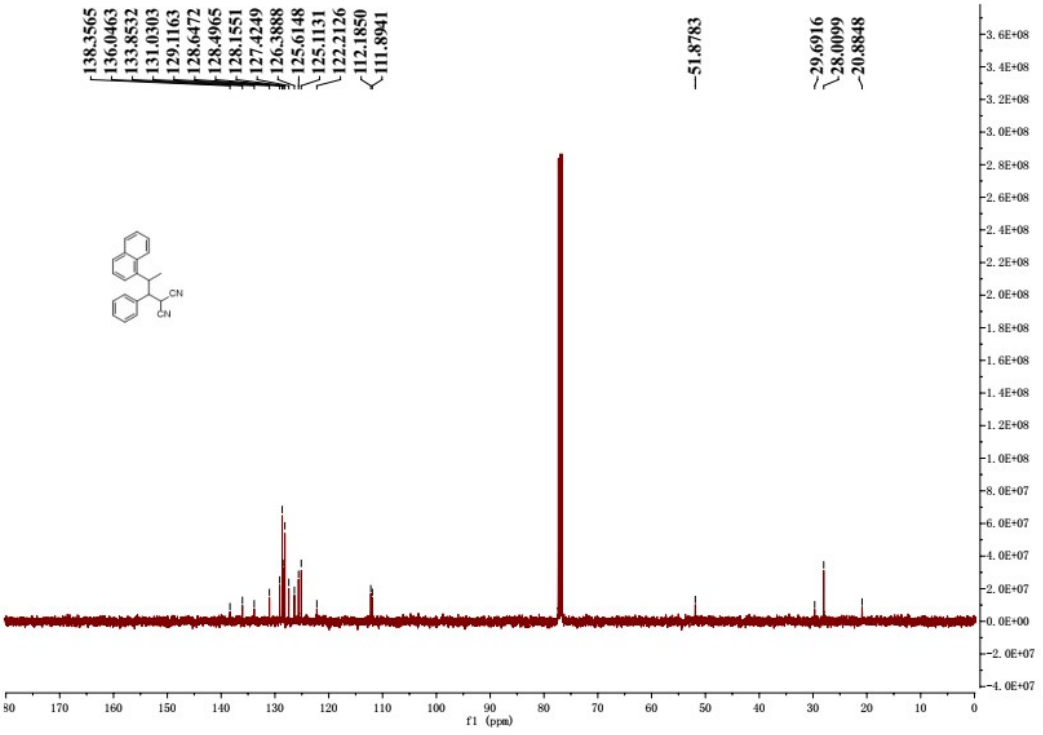
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28.0099
20.8848
CN
CN
f1 (ppm)