Supplementary Information

Convergent and Divergent Synthesis of Dihydroisoquinoline-1,4-diones Enabled by a Photocatalytic Skeleton-Editing [4+2] Strategy

Hai-Wu Du,[1] Jun-Song Jia,[2] Xiao-Yi Chen,[1] Zhang-Yin Yuan,[1,2] Jia-Ni Lin,[1] Yu-Long Li,[2,*] Qiong Yu,[1,*] and Wei Shu[1,2,*]

[1]*Shenzhen Key Laboratory of Small Molecule Drug Discovery and Synthesis, Shenzhen Grubbs Institute, Guangming Advanced Research Institute, Department of Chemistry, and Guangdong Provincial Key Laboratory of Catalysis, Southern University of Science and Technology, Shenzhen 518055, Guangdong, P. R. China*

[2]*College of Chemistry and Environmental Engineering, Innovation Center for Chenguang High Performance Fluorine Material, and Key Laboratory of Green Catalysis of Higher Education Institutes of Sichuan, Sichuan University of Science and Engineering, Zigong 643000, Sichuan, P. R. China*

Table of Contents

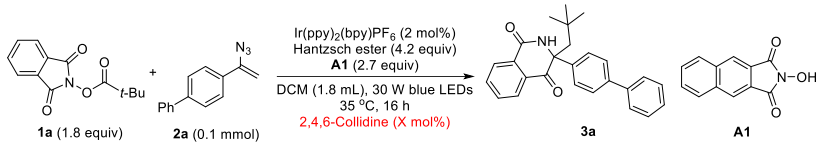
I. General Information

General remarks: ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ (unless otherwise noted) on a Bruker AVANCE 600 MHz or a Bruker AVANCE 400 MHz spectrometer. Chemical shifts in ^{1}H NMR spectra were reported in parts per million (ppm) on the δ scale from an internal standard of TMS (0.00 ppm). Data for ^{1}H NMR were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad), coupling constant in Herts (Hz) and integration. Data for ^{13}C NMR spectra were reported in terms of chemical shift in ppm from the central peak of $CDCl_3$ (77.16 ppm). High-resolution electrospray ionization and electronic impact mass spectrometry were performed on a Thermo Scientific Q Exactive mass spectrometer (mass analyzer type: Orbitrap).

Materials and methods: Unless otherwise noted, all reactions and substrates preparation were conducted in flame-dried glassware under a nitrogen atmosphere using anhydrous solvent re-distilled according to Purification of Laboratory Chemicals (Fifth Edition). Commercially available reagents were used without further purification. Thin layer chromatography (TLC) was performed on Jiangyou TLC silica gel plates HSG F254 and visualized by UV light or staining with anisaldehyde or potassium permanganate. Flash column chromatography was performed over silica gel (300-400 mesh). Photochemical reactions were carried out under irradiation from 30 W blue LED (composed of 30 LED units each with 1.0 W).

II. Reaction Condition Optimization

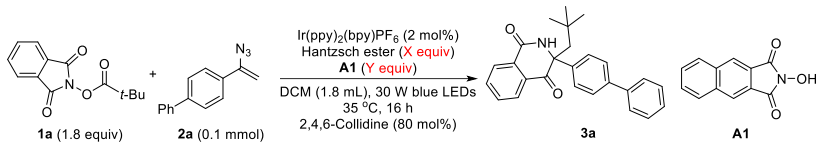
Table S1. Investigation of the amount of 2,4,6-collidine for two-component reaction.[a]



Entry	2,4,6-Collidine (X mol%)	Conv. of **2a** (%)	Yield of **3a** (%)
1	0	>95	43
2	10	>95	42
3	20	>95	50
4	40	>95	57
5	80	>95	62
6	100	>95	52
7	150	>95	37
8	200	>95	33

[a] Reaction conditions: A mixture of **1a** (0.18 mmol), **2a** (0.10 mmol), **A1** (0.27 mmol), $Ir(ppy)_2(bpy)PF_6$ (0.002 mmol), Hantzsch ester (0.42 mmol) and 2,4,6-collidine (X mol%) in DCM (1.8 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 16 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard.

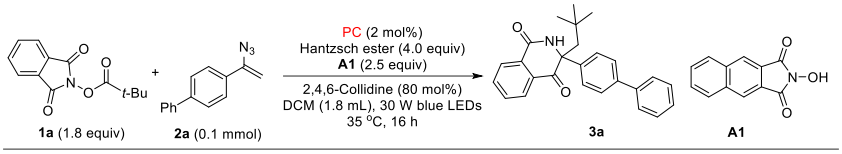
Table S2. Investigation of the amount of **A1** and Hantzsch ester for two-component reaction.[a]



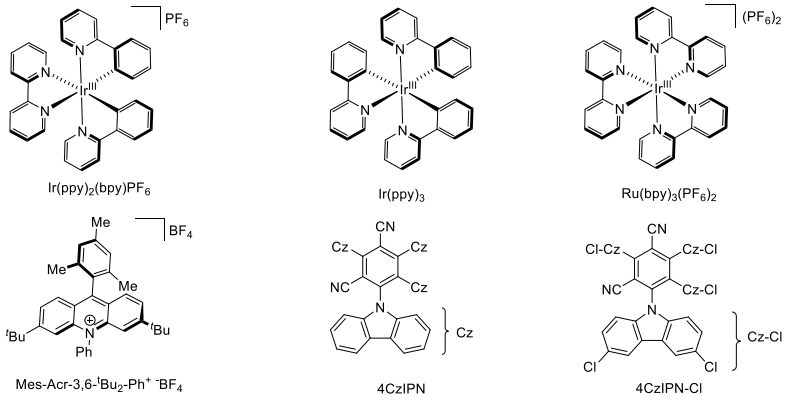
Entry	(X equiv)	(Y equiv)	Conv. of **2a** (%)	Yield of **3a** (%)
1	4.2	2.2	>95	55
2	4.2	2.5	>95	67
3	4.2	3.0	>95	60
4	4.2	3.5	>95	47
5	3.5	2.5	>95	58
6	3.8	2.5	>95	62
7	4.0	2.5	>95	68 (63)
8	4.5	2.5	>95	60

[a] Reaction conditions: A mixture of **1a** (0.18 mmol), **2a** (0.10 mmol), **A1** (Y equiv), $Ir(ppy)_2(bpy)PF_6$ (0.002 mmol), Hantzsch ester (X equiv) and 2,4,6-collidine (0.08 mmol) in DCM (1.8 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 16 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard. Isolated yield after flash chromatography is shown in the parentheses.

Table S3. Investigation of the PC for two-component reaction.[a]

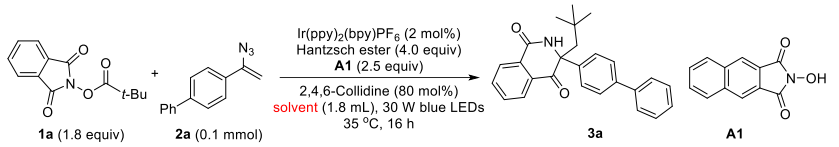


Entry	PC	Conv. of **2a** (%)	Yield of **3a** (%)
1	$Ir(ppy)_2(bpy)PF_6$	>95	68 (63)
2	4CzIPN	>95	56
3	4CzIPN-Cl	>95	52
4	$Ir(ppy)_3$	>95	trace
5	$Ru(bpy)_3(PF_6)_2$	>95	34
6	Mes-Acr-3,6-^{t}Bu$_2$-Ph$^+$ $^-$BF$_4$	>95	trace



[a] Reaction conditions: A mixture of **1a** (0.18 mmol), **2a** (0.10 mmol), **A1** (0.25 mmol), PC (0.002 mmol), Hantzsch ester (0.40 mmol) and 2,4,6-collidine (0.08 mmol) in DCM (1.8 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 16 h. Yield was determined by ^{1}H NMR of the crude mixture using mesitylene as internal standard. Isolated yield after flash chromatography is shown in the parentheses.

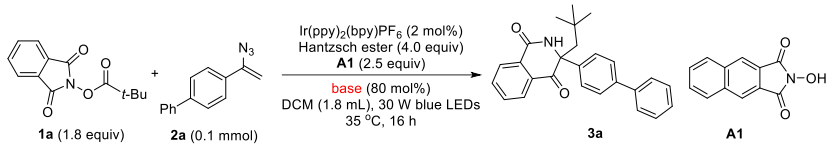
Table S4. Investigation of the solvent for two-component reaction.[a]



Entry	Solvent	Conv. of **2a** (%)	Yield of **3a** (%)
1	DCE	>95	41
2	1,1,2-TCE	>95	35
3	PhCl	>95	44
4	MeCN	>95	22
5	THF	>95	ND

[a] Reaction conditions: A mixture of **1a** (0.18 mmol), **2a** (0.10 mmol), **A1** (0.25 mmol), $Ir(ppy)_2(bpy)PF_6$ (0.002 mmol), Hantzsch ester (0.40 mmol) and 2,4,6-collidine (0.08 mmol) in solvent (1.8 mL) was irradiated with 30 W blue LEDs at room temperature under N_2 for 16 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard. ND = not detected.

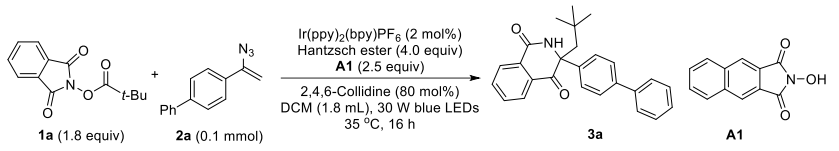
Table S5. Investigation of the base for two-component reaction.[a]



Entry	Base	Conv. of **2a** (%)	Yield of **3a** (%)
1	2,6-lutidine	>95	48
2	DABCO	>95	56
3	DMAP	>95	59
4	K_2CO_3	>95	47
5	Cs_2CO_3	>95	12
6	K_2HPO_4	>95	58

[a] Reaction conditions: A mixture of **1a** (0.18 mmol), **2a** (0.10 mmol), **A1** (0.25 mmol), $Ir(ppy)_2(bpy)PF_6$ (0.002 mmol), Hantzsch ester (0.40 mmol) and base (0.08 mmol) in DCM (1.8 mL) was irradiated with 30 W blue LEDs at room temperature under N_2 for 16 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard.

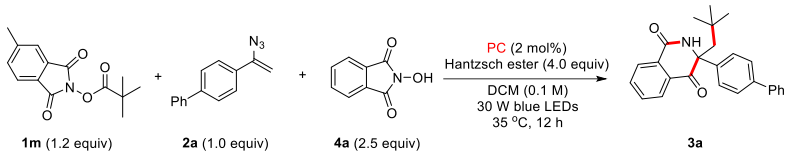
Table S6. Control experiment for two-component reaction.[a]



Entry	variation from standard conditions	Conv. of **2a** (%)	Yield of **3a** (%)
1	w/o $Ir(ppy)_2(bpy)PF_6$	>95	ND
2	w/o Light	<5	ND
3	w/o Hantzsch	>95	ND
4	w/o **A1**	>95	11
5	Air	>95	31

[a] Reaction conditions: A mixture of **1a** (0.18 mmol), **2a** (0.10 mmol), **A1** (0.25 mmol), $Ir(ppy)_2(bpy)PF_6$ (0.002 mmol), Hantzsch ester (0.40 mmol) and 2,4,6-collidine (0.08 mmol) in DCM (1.8 mL) was irradiated with 30 W blue LEDs at room temperature under N_2 for 16 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard. ND = not detected.

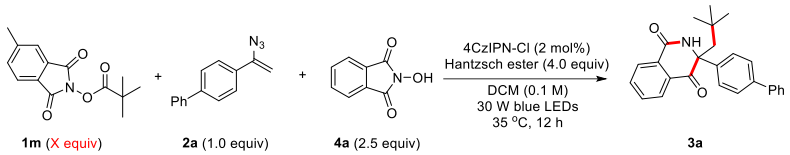
Table S7. Investigation of photocatalyst for three-component reaction.[a]



Entry	PC	Conv. of **2a** (%)	Yield of **3a** (%)
1	**4CzIPN-Cl**	**>95**	**58**
2	$Ir(ppy)_2(bpy)PF_6$	>95	45
3	4CzIPN	>95	50

[a] Reaction conditions: A mixture of **1m** (0.12 mmol), **2a** (0.10 mmol), **4a** (0.25 mmol), PC (0.002 mmol), Hantzsch ester (0.40 mmol) in DCM (1.0 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 12 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard.

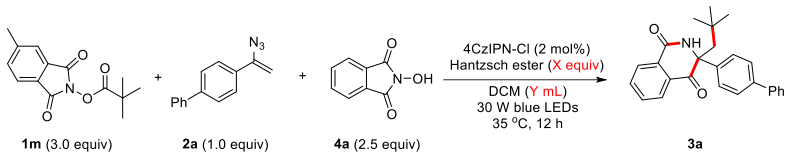
Table S8. Investigation of amount of **1m** for three-component reaction.[a]



Entry	**1m** (X equiv)	Conv. of **2a** (%)	Yield of **3a** (%)
1	1.2	>95	58
2	1.5	>95	61
3	1.8	>95	64
4	2.0	>95	65
5	2.2	>95	65
6	2.4	>95	65
7	2.6	>95	68
8	2.8	>95	69
9	3.0	>95	72
10	3.2	>95	69
11	3.5	>95	61

[a] Reaction conditions: A mixture of **1m** (X equiv), **2a** (0.10 mmol), **4a** (0.25 mmol), 4CzIPN-Cl (0.002 mmol), Hantzsch ester (0.40 mmol) in DCM (1.0 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 12 h. Yield was determined by ^{1}H NMR of the crude mixture using mesitylene as internal standard.

Table S9. Investigation of amount of Hantzsch ester and concentration for three-component reaction.[a]

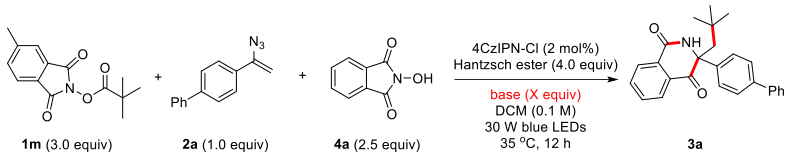


1m (3.0 equiv) **2a** (1.0 equiv) **4a** (2.5 equiv) **3a**

Entry	HE (X equiv)	DCM (Y mL)	Conv. of **2a** (%)	Yield of **3a** (%)
1	3.5	1.0	>95	40
2	3.8	1.0	>95	44
3	4.2	1.0	>95	62
4	4.5	1.0	>95	53
5	4.0	0.8	>95	66
6	4.0	1.2	>95	66
7	4.0	1.5	>95	56

[a] Reaction conditions: A mixture of **1m** (0.30 mmol), **2a** (0.10 mmol), **4a** (0.25 mmol), 4CzIPN-Cl (0.002 mmol), Hantzsch ester (X equiv) in DCM (Y mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 12 h. Yield was determined by ^{1}H NMR of the crude mixture using mesitylene as internal standard.

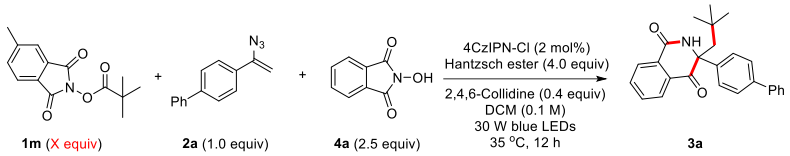
Table S10. Investigation of base for three-component reaction.[a]



Entry	base (X equiv)	Conv. of **2a** (%)	Yield of **3a** (%)
1	DMAP (1.0 equiv)	>95	38
2	DABCO (1.0 equiv)	>95	45
3	2,4,6-collidine (1.0 equiv)	>95	61
4	2,6-lutidine (1.0 equiv)	>95	49
5	Li_2CO_3 (1.0 equiv)	>95	61
6	K_2HPO_4 (1.0 equiv)	>95	31
7	$KHCO_3$ (1.0 equiv)	>95	44
8	NaOAc (1.0 equiv)	>95	24
9	2,4,6-collidine (0.8 equiv)	>95	60
10	2,4,6-collidine (0.6 equiv)	>95	61
11	2,4,6-collidine (0.4 equiv)	>95	61
12	2,4,6-collidine (0.2 equiv)	>95	62

[a] Reaction conditions: A mixture of **1m** (0.30 mmol), **2a** (0.10 mmol), **4a** (0.25 mmol), 4CzIPN-Cl (0.002 mmol), Hantzsch ester (0.4 mmol) and base (X equiv) in DCM (1.0 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 12 h. Yield was determined by ^1H NMR of the crude mixture using mesitylene as internal standard.

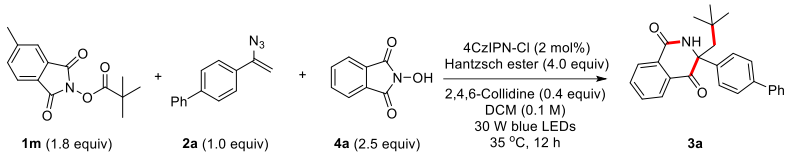
Table S11. Investigation of amount of **1m** for three-component reaction.[a]



Entry	**1m** (X equiv)	Conv. of **2a** (%)	Yield of **3a** (%)
1	3.2	>95	51
2	2.8	>95	59
3	2.6	>95	59
4	2.4	>95	75
5	2.2	>95	76
6	2.0	>95	78
7	1.8	>95	80 (78)
8	1.6	>95	72
9	1.4	>95	66

[a] Reaction conditions: A mixture of **1m** (X equiv), **2a** (0.10 mmol), **4a** (0.25 mmol), 4CzIPN-Cl (0.002 mmol), Hantzsch ester (0.4 mmol) and 2,4,6-collidine (0.04 mmol) in DCM (1.0 mL) was irradiated with 30 W blue LEDs at 35 ºC under N_2 for 12 h. Yield was determined by ^{1}H NMR of the crude mixture using mesitylene as internal standard. Isolated yield after flash chromatography is shown in the parentheses.

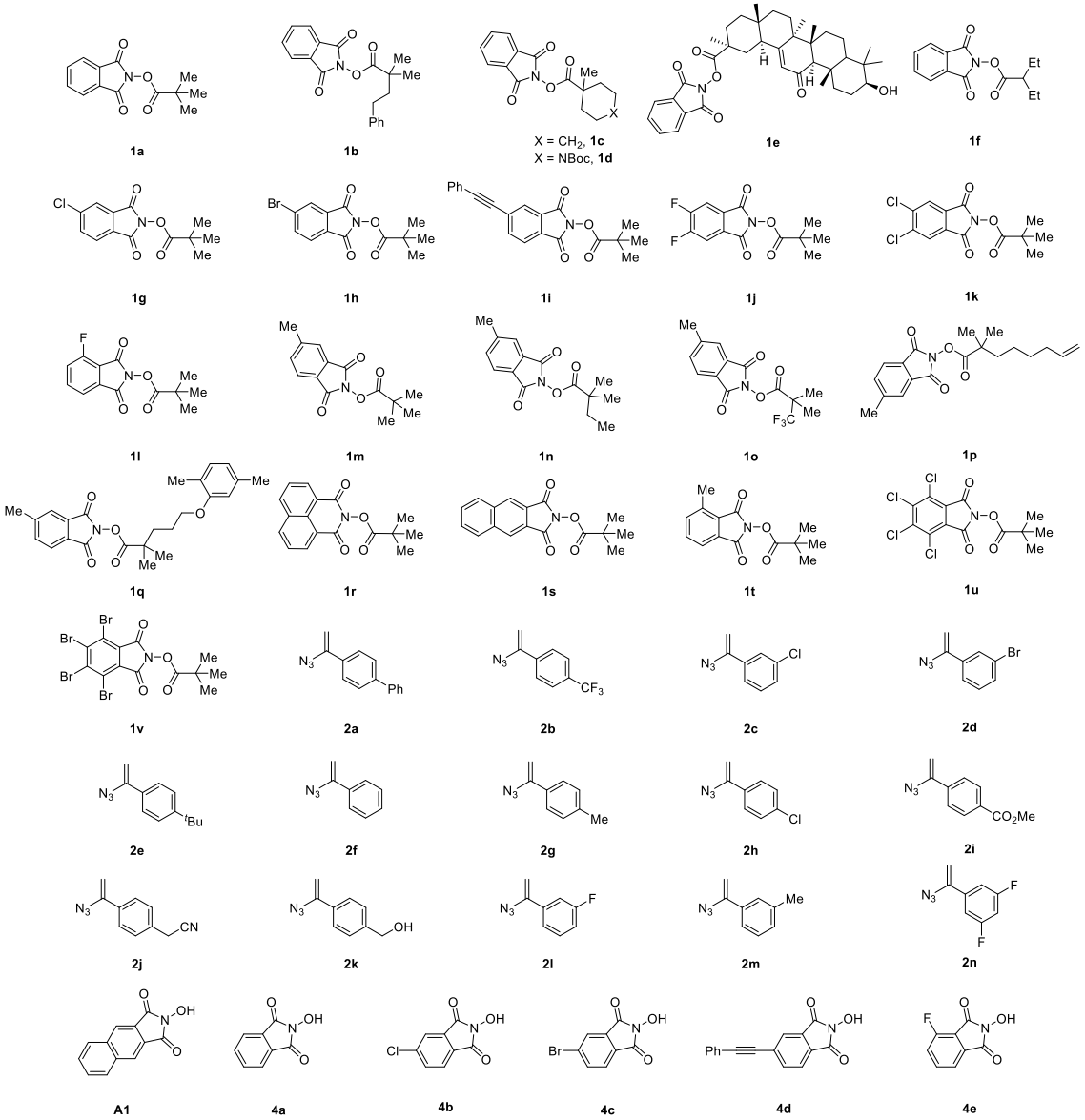
Table S12. Control experiment for three-component reaction.[a]



Entry	variation from standard conditions	Conv. of **2a** (%)	Yield of **3a** (%)
1	w/o PC	55	ND
2	w/o Hantzsch ester	>95	ND
3	w/o light	<5	ND
4	w/o 2,4,6-collidine	>95	56
5	w/o **4a**	>95	ND

[a] Reaction conditions: a mixture of **1m** (0.18 mmol), **2a** (0.10 mmol), **4a** (0.25 mmol), 4CzIPN-Cl (0.002 mmol), Hantzsch ester (0.4 mmol) and 2,4,6-collidine (0.04 mmol) in DCM (1.0 mL) was irradiated with 30 W blue LEDs at 35 °C under N_2 for 12 h. Yield was determined by ^{1}H NMR of the crude mixture using mesitylene as internal standard. Isolated yield after flash chromatography is shown in the parentheses. ND = not detected. ND = not detected.

III. Substrate Source



Synthesis and characterization of substrates

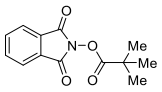
4a, **4f** were purchased and used directly without further purification.

The following substrates are known compounds:

1a[1], **1b**[2], **1c**[3], **1d**[4], **1e**[5], **1f**[1], **1t**[6], **2a**[1], **2b**[1], **2c**[1], **2d**[1], **2e**[1], **2f**[1], **2g**[1], **2h**[7], **2i**[7], **2j**[8], **2k**[1], **2l**[1], **2m**[7], **2n**[9], **A1**[10], **4b**[11], **4c**[10], **4e**[12].

These compounds were prepared according to literature: **1g**[1], **1h**[1], **1i**[1], **1j**[1], **1k**[1], **1l**[1], **1m**[1], **1n**[1], **1o**[1], **1p**[1], **1q**[1], **1r**[1], **1s**[1], **1u**[1], **1v**[1], **4d**[10].

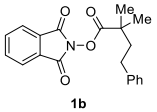
1,3-Dioxoisoindolin-2-yl pivalate (1a)



1a

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.78 (dd, *J* = 5.4, 3.1 Hz, 2H), 1.43 (s, 9H).

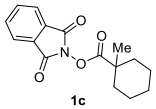
1,3-Dioxoisoindolin-2-yl 2,2-dimethyl-4-phenylbutanoate (1b)



1b

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.92 – 7.87 (m, 2H), 7.82 – 7.76 (m, 2H), 7.33 – 7.26 (m, 4H), 7.22 – 7.17 (m, 1H), 2.80 – 2.74 (m, 2H), 2.06 – 2.00 (m, 2H), 1.47 (s, 6H).

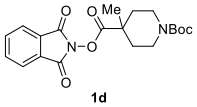
1,3-Dioxoisoindolin-2-yl 1-methylcyclohexane-1-carboxylate (1c)



1c

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.90 – 7.86 (m, 2H), 7.80 – 7.76 (m, 2H), 2.24 (d, *J* = 13.2 Hz, 2H), 1.70 – 1.62 (m, 3H), 1.61 – 1.55 (m, 2H), 1.43 (s, 3H), 1.42 – 1.35 (m, 2H), 1.31 – 1.25 (m, 1H).

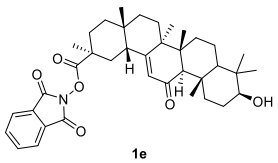
1-(*tert*-Butyl) 4-(1,3-dioxoisoindolin-2-yl) 4-methylpiperidine-1,4-dicarboxylate (1d)



1d

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.90 – 7.87 (m, 2H), 7.81 – 7.78 (m, 2H), 4.05 – 3.85 (m, 2H), 3.18 – 3.08 (m, 2H), 2.28 – 2.22 (m, 2H), 1.56 – 1.50 (m, 2H), 1.48 (s, 3H), 1.46 (s, 9H).

1,3-Dioxoisoindolin-2-yl (2*S*,4a*S*,6a*S*,6b*R*,10*S*,12a*S*,12b*R*,14b*S*)-10-hydroxy-2,4*a*,6*a*,6*b*,9,9,12*a*-heptamethyl-13-oxo-1,2,3,4,4*a*,5,6,6*a*,6*b*,7,8,8*a*,9,10,11,12,12*a*,12*b*,13,14*b*-icosahydropicene-2-carboxylate (1e)

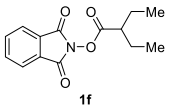


1e

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.79 (dd, *J* = 5.5, 3.1 Hz, 2H), 5.76 (s, 1H), 3.22 (dd, *J* = 10.8, 5.5 Hz, 1H), 2.79 (dt, *J* = 13.4, 3.6 Hz, 1H), 2.50 – 2.40 (m, 1H), 2.33 (s, 1H), 2.18 – 1.99 (m, 4H), 1.92 – 1.74 (m, 3H), 1.70 – 1.63 (m, 3H), 1.55 – 1.44 (m, 4H), 1.44 (s, 3H), 1.38 (s, 3H), 1.25 – 1.18 (m, 2H), 1.14 (s, 3H), 1.13 (s, 3H), 1.11 – 1.03 (m, 2H), 1.00 (s, 3H), 0.91 (s, 3H), 0.80 (s, 3H),

0.73 – 0.67 (m, 1H).

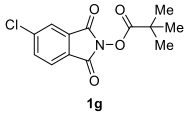
1,3-Dioxoisoindolin-2-yl 2-ethylbutanoate (1f)



1f

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.90 – 7.84 (m, 2H), 7.81 – 7.75 (m, 2H), 2.64 – 2.56 (m, 1H), 1.85 – 1.75 (m, 2H), 1.76 – 1.66 (m, 2H), 1.07 (t, *J* = 7.5 Hz, 6H).

5-Chloro-1,3-dioxoisoindolin-2-yl pivalate (1g)



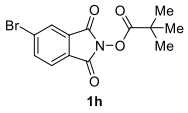
1g

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 1.8 Hz, 1H)，7.82 (d, *J* = 8.0 Hz, 1H), 7.74 (dd, *J* = 8.0, 1.8 Hz, 1H), 1.43 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 161.3, 161.0, 141.6, 134.9, 130.8, 127.2, 125.3, 124.5, 38.6, 27.1.

HR-MS (ESI-TOF) calcd for $C_{13}H_{12}ClNNaO_4^+$ $[M+Na]^+$ 304.0347; found 304.0348.

5-Bromo-1,3-dioxoisoindolin-2-yl pivalate (1h)



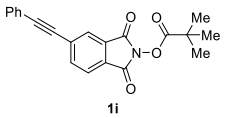
1h

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 1.7 Hz, 1H), 7.92 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.74 (d, *J* = 8.0 Hz, 1H), 1.43 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 161.5, 160.9, 137.8, 130.7, 129.8, 127.6, 127.3, 125.4, 38.6, 27.1.

HR-MS (ESI-TOF) calcd for $C_{13}H_{12}BrNNaO_4^+$ $[M+Na]^+$ 347.9842; found 347.9847.

1,3-Dioxo-5-(phenylethynyl)isoindolin-2-yl pivalate (1i)



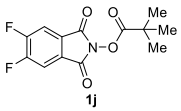
1i

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (s, 1H), 7.91 – 7.82 (m, 2H), 7.59 – 7.54 (m, 2H), 7.43 – 7.36 (m, 3H), 1.44 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 161.7, 161.6, 137.5, 132.0, 130.5, 129.50, 129.45, 128.7, 127.7, 126.7, 124.0, 122.1, 94.8, 87.6, 38.6, 27.2.

HR-MS (ESI-TOF) calcd for $C_{21}H_{17}NNaO_4^+$ $[M+Na]^+$ 370.1050; found 370.1050.

5,6-Difluoro-1,3-dioxoisoindolin-2-yl pivalate (1j)



1j

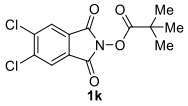
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (t, *J* = 7.3 Hz, 2H), 1.42 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 160.4, 154.9 (dd, *J* = 262.6, 14.9 Hz), 126.2 (t, *J* = 5.8 Hz), 114.3 (dd, *J* = 14.9, 7.7 Hz), 38.6, 27.1.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -123.93 (t, *J* = 7.3 Hz, 2F).

HR-MS (ESI-TOF) calcd for $C_{13}H_{11}F_2NNaO_4^+$ $[M+Na]^+$ 306.0548; found 306.0552.

5,6-Dichloro-1,3-dioxoisoindolin-2-yl pivalate (1k)



1k

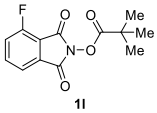
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (s, 2H), 1.43 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.2, 160.4, 139.9, 128.2, 126.2, 38.6, 27.1.

^{13}C Quantitative-NMR (151 MHz, Chloroform-*d*) δ 174.3 (1C), 160.4 (2C), 139.9 (2C), 128.2 (2C), 126.2 (2C), 38.6 (1C), 27.1 (3C).

HR-MS (ESI-TOF) calcd for $C_{13}H_{11}Cl_2NNaO_4^+$ $[M+Na]^+$ 337.9957; found 337.9959.

4-Fluoro-1,3-dioxoisoindolin-2-yl pivalate (1l)



1l

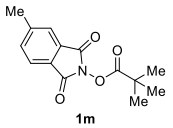
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 (ddd, *J* = 8.4, 7.4, 4.4 Hz, 1H), 7.71 (dd, *J* = 7.4, 0.8 Hz, 1H), 7.45 (td, *J* = 8.4, 0.8 Hz, 1H), 1.43 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.3, 161.1 (d, *J* = 3.3 Hz), 158.9 (d, *J* = 0.9 Hz), 157.7 (d, *J* = 267.2 Hz), 137.5 (d, *J* = 7.9 Hz), 131.0 (d, *J* = 1.6 Hz), 123.3 (d, *J* = 19.7 Hz), 120.2 (d, *J* = 3.7 Hz), 115.1 (d, *J* = 12.8 Hz), 38.5, 27.1.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -111.05 (dd, *J* = 8.7, 4.4 Hz, 1F).

HR-MS (ESI-TOF) calcd for $C_{13}H_{12}FNNaO_4^+$ $[M+Na]^+$ 288.0643; found 288.0642.

5-Methyl-1,3-dioxoisoindolin-2-yl pivalate (1m)



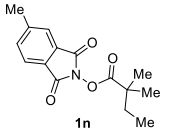
1m

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.76 (d, *J* = 7.6 Hz, 1H), 7.68 (s, 1H), 7.56 (d, *J* = 7.7 Hz, 1H), 2.53 (s, 3H), 1.43 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 174.6, 162.5, 162.4, 146.3, 135.3, 129.5, 126.5, 124.5, 124.0, 38.5, 27.2, 22.3.

HR-MS (ESI-TOF) calcd for $C_{14}H_{15}NNaO_4^+$ $[M+Na]^+$ 284.0893; found 284.0891.

5-Methyl-1,3-dioxoisoindolin-2-yl 2,2-dimethylbutanoate (1n)



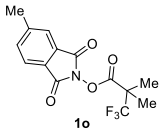
1n

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.75 (d, *J* = 7.6 Hz, 1H), 7.67 (s, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 2.52 (s, 3H), 1.78 (q, *J* = 7.4 Hz, 2H), 1.38 (s, 6H), 1.04 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 174.1, 162.6, 162.4, 146.2, 135.3, 129.5, 126.5, 124.5, 123.9, 42.6, 33.7, 24.8, 22.3, 9.2.

HR-MS (ESI-TOF) calcd for $C_{15}H_{17}NNaO_4^+$ $[M+Na]^+$ 298.1050; found 298.1049.

5-Methyl-1,3-dioxoisoindolin-2-yl 3,3,3-trifluoro-2,2-dimethylpropanoate (1o)



1o

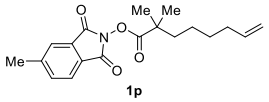
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.77 (d, *J* = 7.6 Hz, 1H), 7.70 (s, 1H), 7.59 (d, *J* = 7.2 Hz, 1H), 2.54 (s, 3H), 1.66 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 167.3, 162.8, 161.7, 146.6, 135.6, 129.2, 126.3, 125.6 (q, *J* = 283.8 Hz), 124.7, 124.2, 48.4 (q, *J* = 27.7 Hz), 22.3, 19.8 (q, *J* = 2.3 Hz).

^{19}F NMR (376 MHz, Chloroform-*d*) δ -74.94 (s, 3F).

HR-MS (ESI-TOF) calcd for $C_{14}H_{12}F_3NNaO_4^+$ $[M+Na]^+$ 338.0611; found 338.0610.

5-Methyl-1,3-dioxoisoindolin-2-yl 2,2-dimethyloct-7-enoate (1p)



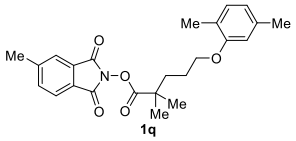
1p

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 7.6 Hz, 1H), 7.67 (s, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 5.89 – 5.77 (m, 1H), 5.05 – 4.88 (m, 2H), 2.52 (s, 3H), 2.14 – 2.06 (m, 2H), 1.75 – 1.67 (m, 2H), 1.49 – 1.42 (m, 4H), 1.38 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.0, 162.4, 162.3, 146.2, 138.9, 135.2, 129.4, 126.4, 124.4, 123.9, 114.5, 42.2, 40.6, 33.6, 29.3, 25.2, 24.3, 22.2.

HR-MS (ESI-TOF) calcd for $C_{19}H_{23}NNaO_4^+$ $[M+Na]^+$ 352.1519; found 352.1517.

5-Methyl-1,3-dioxoisoindolin-2-yl 5-(2,5-dimethylphenoxy)-2,2-dimethylpentanoate (1q)



1q

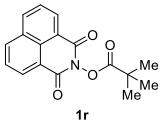
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.76 (d, *J* = 7.7 Hz, 1H), 7.68 (s, 1H), 7.57 (d, *J* = 7.7 Hz, 1H), 7.00 (d, *J* = 7.5 Hz, 1H), 6.68 – 6.63 (m, 2H), 4.01 (t, *J* = 3.4 Hz, 2H), 2.53 (s, 3H), 2.31 (s, 3H), 2.19 (s, 3H), 1.95 (d, *J* = 2.8 Hz, 4H), 1.44 (s, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 173.9, 162.5, 162.4, 157.1, 146.3, 136.6, 135.3, 130.4, 129.4, 126.5, 124.5, 123.9, 123.7, 120.8, 112.1, 67.9, 42.1, 37.5, 25.2, 25.1, 22.3, 21.5, 15.9.

^{13}C Quantitative-NMR (151 MHz, Chloroform-*d*) δ 173.9 (1C), 162.5 (1C), 162.4 (1C), 157.1 (1C), 146.3 (1C), 136.6 (1C), 135.3 (1C), 130.4 (1C), 129.4 (1C), 126.4 (1C), 124.5 (1C), 123.9 (1C), 123.7 (1C), 120.8 (1C), 112.1(1C), 67.9 (1C), 42.1 (1C), 37.5 (1C), 25.2 (2C), 25.1 (1C), 22.3 (1C), 21.5 (1C), 15.9 (1C).

HR-MS (ESI-TOF) calcd for $C_{24}H_{27}NNaO_5^+$ $[M+Na]^+$ 432.1781; found 432.1782.

1,3-Dioxo-1*H*-benzo[*de*]isoquinolin-2(3*H*)-yl pivalate (1r)



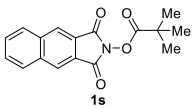
1r

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.61 (dd, *J* = 7.2, 1.2 Hz, 2H), 8.27 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.78 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 2H), 1.51 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 159.8, 135.1, 132.0, 131.9, 127.7, 127.2, 122.5, 38.7, 27.2.

HR-MS (ESI-TOF) calcd for $C_{17}H_{15}NNaO_4^+$ $[M+Na]^+$ 320.0893; found 320.0894.

1,3-Dioxo-1,3-dihydro-2*H*-benzo[*f*]isoindol-2-yl pivalate (1s)



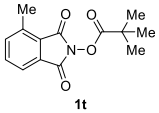
1s

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.38 (s, 2H), 8.10 – 8.04 (m, 2H), 7.76 – 7.71 (m, 2H), 1.46 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.4, 161.8, 135.6, 130.5, 129.8, 125.8, 124.7, 38.6, 27.2.

HR-MS (ESI-TOF) calcd for $C_{17}H_{15}NNaO_4^+$ $[M+Na]^+$ 320.0893; found 320.0891.

4-Methyl-1,3-dioxoisoindolin-2-yl pivalate (1t)



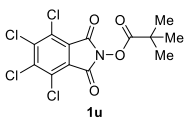
1t

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 (d, *J* = 7.6 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 7.6 Hz, 1H), 2.69 (s, 3H), 1.43 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.6, 163.1, 162.3, 138.8, 137.2, 134.3, 129.6, 125.9, 121.7, 38.6, 27.2, 17.8.

HR-MS (ESI-TOF) calcd for $C_{14}H_{15}NNaO_4^+$ $[M+Na]^+$ 284.0893; found 284.0895.

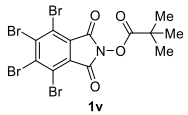
4,5,6,7-Tetrachloro-1,3-dioxoisoindolin-2-yl pivalate (1u)



1u

^{1}H NMR (400 MHz, Chloroform-*d*) δ 1.43 (s, 9H).

4,5,6,7-Tetrabromo-1,3-dioxoisoindolin-2-yl pivalate (1v)



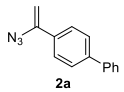
1v

^{1}H NMR (400 MHz, Chloroform-*d*) δ 1.43 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 174.0, 158.3, 138.6, 128.0, 122.1, 38.6, 27.1.

HR-MS (ESI-TOF) calcd for $C_{13}H_9Br_4NNaO_4^+$ $[M+Na]^+$ 581.7157; found 581.7163.

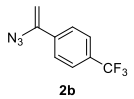
4-(1-Azidovinyl)-1,1'-biphenyl (2a)



2a

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.66 – 7.62 (m, 2H), 7.62 – 7.58 (m, 4H), 7.47 – 7.42 (m, 2H), 7.37 (td, *J* = 7.2, 1.2 Hz, 1H), 5.49 (d, *J* = 2.5 Hz, 1H), 4.99 (d, *J* = 2.5 Hz, 1H).

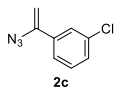
1-(1-Azidovinyl)-4-(trifluoromethyl)benzene (2b)



2b

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.68 (d, *J* = 8.2 Hz, 2H), 7.61 (d, *J* = 8.2 Hz, 2H), 5.54 (d, *J* = 2.7 Hz, 1H), 5.07 (d, *J* = 2.7 Hz, 1H).

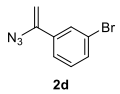
1-(1-Azidovinyl)-3-chlorobenzene (2c)



2c

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.52 (s, 1H), 7.41 (d, *J* = 7.6 Hz, 1H), 7.30 – 7.24 (m, 2H), 5.44 – 5.42 (m, 1H), 4.97 – 4.95 (m, 1H).

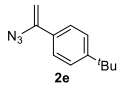
1-(1-Azidovinyl)-3-bromobenzene (2d)



2d

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (t, *J* = 1.9 Hz, 1H), 7.48 (tdd, *J* = 7.9, 1.9, 1.0 Hz, 2H), 7.22 (t, *J* = 7.9 Hz, 1H), 5.46 (d, *J* = 2.7 Hz, 1H), 4.99 (d, *J* = 2.7 Hz, 1H).

1-(1-Azidovinyl)-4-(*tert*-butyl)benzene (2e)



2e

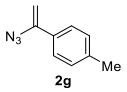
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 – 7.51 (m, 2H), 7.46 – 7.39 (m, 2H), 5.44 (d, *J* = 2.3 Hz, 1H), 4.96 (d, *J* = 2.3 Hz, 1H), 1.37 (s, 9H).

(1-Azidovinyl)benzene (2f)

2f

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 – 7.55 (m, 2H), 7.43 – 7.33 (m, 3H), 5.45 (d, *J* = 2.4 Hz, 1H), 4.97 (d, *J* = 2.4 Hz, 1H).

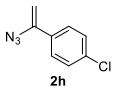
1-(1-Azidovinyl)-4-methylbenzene (2g)



2g

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.53 – 7.47 (m, 2H), 7.22 – 7.18 (m, 2H), 5.43 (d, *J* = 2.3 Hz, 1H), 4.95 (d, *J* = 2.3 Hz, 1H), 2.40 (s, 3H).

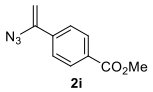
1-(1-Azidovinyl)-4-chlorobenzene (2h)



2h

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.52 – 7.47 (m, 2H), 7.35 – 7.31 (m, 2H), 5.44 – 5.43 (m, 1H), 5.00 – 4.96 (m, 1H).

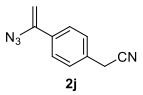
Methyl 4-(1-azidovinyl)benzoate (2i)



2i

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.98 (m, 2H), 7.66 – 7.59 (m, 2H), 5.57 (d, *J* = 2.7 Hz, 1H), 5.07 (d, *J* = 2.7 Hz, 1H), 3.92 (s, 3H).

2-(4-(1-Azidovinyl)phenyl)acetonitrile (2j)



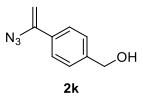
2j

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 5.46 (d, *J* = 2.6 Hz, 1H), 4.99 (d, *J* = 2.6 Hz, 1H), 3.76 (s, 2H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 144.4, 134.3, 130.9, 128.2, 126.4, 117.6, 98.5, 23.5.

HR-MS (ESI-TOF) calcd for $C_{10}H_7N_4^-$ $[M-H]^-$ 183.0676; found 183.0669.

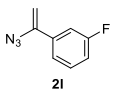
(4-(1-azidovinyl)phenyl)methanol (2k)



2k

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 5.43 (d, *J* = 2.4 Hz, 1H), 4.96 (d, *J* = 2.4 Hz, 1H), 4.70 (d, *J* = 4.0 Hz, 2H).

1-(1-Azidovinyl)-3-fluorobenzene (2l)



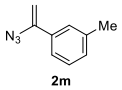
2l

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 – 7.24 (m, 3H), 7.04 (tdd, *J* = 8.1, 2.6, 1.4

Hz, 1H), 5.47 (d, *J* = 2.6 Hz, 1H), 5.00 (d, *J* = 2.6 Hz, 1H).

^{19}F NMR (376 MHz, Chloroform-*d)* δ -112.76 – -112.87 (m,1F).

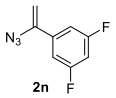
1-(1-Azidovinyl)-3-methylbenzene (2m)



2m

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 – 7.37 (m, 2H), 7.29 (t, *J* = 3.8 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 5.44 (d, *J* = 2.4 Hz, 1H), 4.97 (d, *J* = 2.4 Hz, 1H), 2.40 (s, 3H).

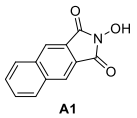
1-(1-Azidovinyl)-3,5-difluorobenzene (2n)



2n

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.13 – 7.06 (m, 2H), 6.79 (tt, *J* = 8.7, 2.3 Hz, 1H), 5.49 (d, *J* = 2.9 Hz, 1H), 5.04 (d, *J* = 2.9 Hz, 1H).

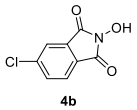
2-Hydroxy-1*H*-benzo[*f*]isoindole-1,3(2*H*)-dione (A1)



A1

^{1}H NMR (600 MHz, DMSO-d$_6$) δ 10.97 (brs, 1H), 8.47 (s, 2H), 8.27 – 8.22 (m, 2H), 7.79 – 7.75 (m, 2H).

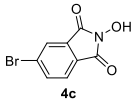
5-Chloro-2-hydroxyisoindoline-1,3-dione (4b)



4b

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.94 (brs, 1H), 7.94 – 7.79 (m, 3H).

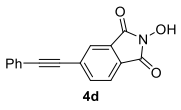
5-Bromo-2-hydroxyisoindoline-1,3-dione (4c)



4c

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.92 (brs, 1H), 8.05 – 8.01 (m, 2H), 7.76 (d, *J* = 8.3 Hz, 1H).

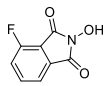
2-Hydroxy-5-(phenylethynyl)isoindoline-1,3-dione (4d)



4d

^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.91 (brs, 1H), 7.99 – 7.94 (m, 2H), 7.89 – 7.84 (m, 1H), 7.66 – 7.60 (m, 2H), 7.50 – 7.45 (m, 3H).

4-Fluoro-2-hydroxyisoindoline-1,3-dione (4e)

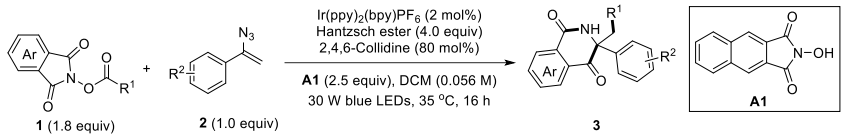


4e

^{1}H NMR (400 MHz, DMSO-d_6) δ 10.91 (brs, 1H), 7.90 – 7.84 (m, 1H), 7.70 – 7.62 (m, 2H).

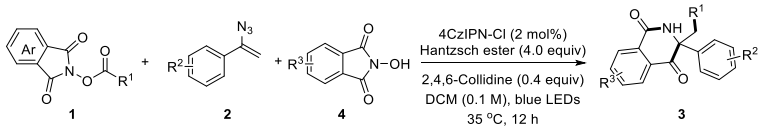
IV. General Procedures

Typical Procedure A



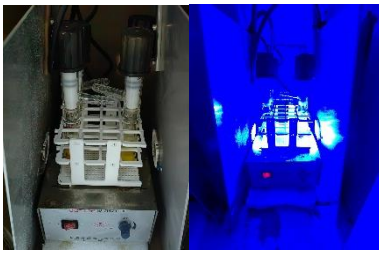
To a Schlenk tube were added **1** (0.18 mmol, 1.8 equiv), **2** (if solid) (0.10 mmol, 1.0 equiv), **A1** (53.4 mg, 0.25 mmol, 2.5 equiv), $Ir(ppy)_2(bpy)PF_6$ (1.6 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.8 mL), 2,4,6-collidine (9.7 mg, 0.08 mmol, 80 mol%) and **2** (if liquid) (0.10 mmol, 1.0 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring until TLC indicated the complete consumption of **2** (typically 16 h). The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography to afford the pure product (**3a**-**3p**).

Typical Procedure B



To a Schlenk tube were added **1** (0.18 mmol, 1.8 equiv), **2** (if solid) (0.10 mmol, 1.0 equiv), **4** (0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%) and **2** (if liquid) (0.10 mmol, 1.0 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring until TLC indicated the complete consumption of **2** (typically 12 h). The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product (**3**).

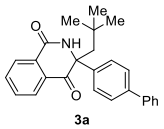
Reaction Setup



The reaction was carried out using the above setup with two 30W of blue LEDs (LED DRIVER YJ-30W, λ = 460-470 nm). There is 2.5 cm distance between the reactor and LEDs at 35 °C, which is monitored by a thermometer.

V. Characterization of Products

3-([1,1'-Biphenyl]-4-yl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3a)



3a

Following typical procedure A, the reaction of **1a** (44.5 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3a** as white solid (24.1 mg, 63% yield).

Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3a** as white solid (30.0 mg, 78% yield).

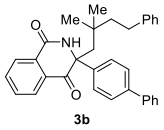
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.32 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.03 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.79 (td, *J* = 7.6, 1.3 Hz, 1H), 7.68 (td, *J* = 7.6, 1.3 Hz, 1H), 7.61 – 7.55 (m, 2H), 7.55 – 7.47 (m, 4H), 7.43 – 7.37 (m, 2H), 7.36 – 7.29 (m, 1H), 6.58 (brs, 1H), 3.13 (d, *J* = 14.9 Hz, 1H), 1.88 (d, *J* = 14.9 Hz, 1H), 1.01 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.5, 162.7, 141.3, 141.1, 140.2, 135.0, 133.3, 131.1, 131.0, 128.9, 128.5, 127.70, 127.68, 127.4, 127.2, 126.1, 70.9, 52.8, 32.0, 31.7.

^{13}C Quantitative-NMR (151 MHz, Chloroform-*d*) δ 192.4 (1C), 162.6 (1C), 141.3 (1C), 141.2 (1C), 140.2 (1C), 135.0 (1C), 133.4 (1C), 131.1 (1C), 131.0 (1C), 128.9 (2C), 128.5 (1C), 127.72 (1C), 127.69 (2C), 127.4 (1C), 127.2 (2C), 126.0 (2C), 70.9 (1C), 52.8 (1C), 32.0 (1C), 31.7 (3C).

HR-MS (ESI-TOF) calcd for $C_{26}H_{25}NNaO_2^+$ $[M+Na]^+$ 406.1778 found 406.1780.

3-([1,1'-Biphenyl]-4-yl)-3-(2,2-dimethyl-4-phenylbutyl)-2,3-dihydroisoquinoline-1,4-dione (3b)



3b

Following typical procedure A, the reaction of **1b** (60.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3b** as white solid (24.1 mg, 51% yield).

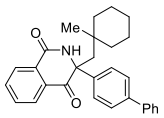
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.32 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.03 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.79 (td, *J* = 7.6, 1.4 Hz, 1H), 7.68 (td, *J* = 7.6, 1.4 Hz, 1H), 7.61 – 7.56 (m, 2H), 7.55 – 7.49 (m, 4H), 7.44 – 7.38 (m, 2H), 7.36 – 7.31 (m, 1H), 7.28 – 7.22 (m, 2H), 7.19 – 7.12 (m, 3H), 6.57 (brs, 1H), 3.19 (d, *J* = 14.9 Hz, 1H), 2.68 – 2.59 (m, 2H), 1.97 (d, *J* = 14.9 Hz, 1H), 1.66 – 1.59 (m, 2H), 1.13 (s, 3H), 1.00 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.4, 162.7, 142.8, 141.3, 141.2, 140.2, 135.1, 133.4, 131.0, 130.9, 129.0, 128.5, 128.4, 127.7, 127.4, 127.2, 126.1, 125.8, 70.8, 50.9, 47.1, 34.6, 30.7, 29.4, 28.4.

^{13}C Quantitative-NMR (151 MHz, Chloroform-*d*) δ 192.4 (1C), 162.6 (1C), 142.8 (1C), 141.3 (1C), 141.2 (1C), 140.2 (1C), 135.1 (1C), 133.4 (1C), 131.0 (1C), 130.9 (1C), 129.0 (2C), 128.5 (3C), 128.4 (2C), 127.7 (3C), 127.4 (1C), 127.2 (2C), 126.0 (2C), 125.8 (1C), 70.8 (1C), 50.9 (1C), 47.1 (1C), 34.6 (1C), 30.7 (1C), 29.4 (1C), 28.4 (1C).

HR-MS (ESI-TOF) calcd for $C_{33}H_{31}NNaO_2^+$ $[M+Na]^+$ 496.2247; found 496.2249.

3-([1,1'-Biphenyl]-4-yl)-3-((1-methylcyclohexyl)methyl)-2,3-dihydroisoquinoline-1,4-dione (3c)



3c

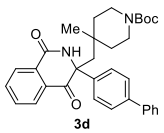
Following typical procedure A, the reaction of **1c** (51.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3c** as white solid (22.2 mg, 52% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.32 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.04 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.78 (td, *J* = 7.6, 1.4 Hz, 1H), 7.68 (td, *J* = 7.6, 1.4 Hz, 1H), 7.63 – 7.57 (m, 2H), 7.55 – 7.47 (m, 4H), 7.44 – 7.36 (m, 2H), 7.36 – 7.28 (m, 1H), 6.84 (brs, 1H), 3.06 (d, *J* = 14.9 Hz, 1H), 1.96 (d, *J* = 14.9 Hz, 1H), 1.53 – 1.60 (m, 1H), 1.48 – 1.35 (m, 6H), 1.31 – 1.19 (m, 3H), 0.96 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.5, 162.6, 141.5, 141.1, 140.2, 135.0, 133.4, 131.0, 130.9, 128.9, 128.5, 127.71, 127.68, 127.4, 127.2, 126.1, 70.8, 53.3, 40.0, 39.7, 34.6, 26.2, 24.9, 22.1, 21.9.

HR-MS (ESI-TOF) calcd for $C_{29}H_{29}NNaO_2^+$ $[M+Na]^+$ 446.2091; found 446.2090.

***tert*-Butyl 4-((3-([1,1'-biphenyl]-4-yl)-1,4-dioxo-1,2,3,4-tetrahydroisoquinolin-3-yl)methyl)-4-methylpiperidine-1-carboxylate (3d)**



3d

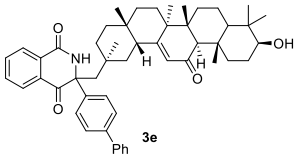
Following typical procedure A, the reaction of **1d** (69.8 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3d** as white solid (26.0 mg, 50% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.31 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.80 (td, *J* = 7.6, 1.4 Hz, 1H), 7.69 (td, *J* = 7.6, 1.4 Hz, 1H), 7.61 – 7.56 (m, 2H), 7.55 – 7.47 (m, 4H), 7.43 – 7.37 (m, 2H), 7.36 – 7.29 (m, 1H), 7.00 (brs, 1H), 3.79 – 3.64 (m, 2H), 3.11 (d, *J* = 14.9 Hz, 1H), 3.05 – 2.94 (m, 2H), 1.94 (d, *J* = 14.9 Hz, 1H), 1.65 – 1.51 (m, 3H) 1.42 (s, 9H), 1.29 – 1.23 (m, 1H), 1.02 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.3, 162.6, 155.0, 141.4, 141.1, 140.1, 135.2, 133.6, 130.9, 130.8, 129.0, 128.6, 127.8, 127.5, 127.2, 126.0, 79.6, 70.6, 53.0, 38.8, 38.6, 33.1, 28.6, 23.2.

HR-MS (ESI-TOF) calcd for $C_{33}H_{36}N_2NaO_4^+$ $[M+Na]^+$ 547.2567; found 547.2569.

3-([1,1'-Biphenyl]-4-yl)-3-(((2*S*,4*aR*,6*aS*,6*bR*,10*S*,12*aS*,12*bR*,14*bS*)-10-hydroxy-2,4*a*,6*a*,6*b*,9,9,12*a*-heptamethyl-13-oxo-1,2,3,4,4*a*,5,6,6*a*,6*b*,7,8,8*a*,9,10,11,12,12*a*,12*b*,13,14*b*-icosahydropicen-2-yl)methyl)-2,3-dihydroisoquinoline-1,4-dione (3e)



3e

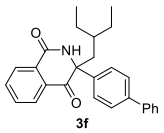
Following typical procedure A, the reaction of **1e** (110.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3e** as white solid (32.2 mg, 44% yield, dr = 1:1:1:2).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.31 (dd, *J* = 7.8, 1.2 Hz, 1H), 8.03 (ddd, *J* = 7.8, 5.7, 1.2 Hz, 1H), 7.81 – 7.75 (m, 1H), 7.68 (dtd, *J* = 12.4, 7.6, 1.3 Hz, 1H), 7.60 – 7.56 (m, 2H), 7.53 – 7.46 (m, 4H), 7.39 (td, *J* = 7.8, 2.7 Hz, 2H), 7.34 – 7.30 (m, 1H), 6.84 (brs, 1H), 5.47 (s, 1H), 3.22 (dt, *J* = 10.5, 4.7 Hz, 1H), 3.07 (d, *J* = 14.9 Hz, 1H), 2.78 – 2.74 (m, 1H), 2.25 (s, 1H), 2.15 – 2.08 (m, 1H), 2.03 – 1.93 (m, 1H), 1.85 (d, *J* = 14.9 Hz, 1H), 1.67 – 1.53 (m, 4H), 1.47 – 1.35 (m, 3H), 1.29 – 1.18 (m, 4H), 1.11 (d, *J* = 2.1 Hz, 6H), 1.08 (s, 6H), 1.00 (s, 3H), 0.97 (s, 3H), 0.96 – 0.85 (m, 3H), 0.81 (s, 6H), 0.70 – 0.61 (m, 1H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 200.2, 192.2, 169.6, 162.6, 141.3, 141.1, 140.1, 135.2, 133.4, 130.9, 129.0, 128.6, 128.5, 128.3, 127.7, 127.4, 127.1, 126.0, 78.9, 70.6, 61.9, 55.3, 55.0, 47.1, 45.6, 44.6, 43.4, 39.2, 37.3, 35.9, 35.5, 34.4, 33.6, 32.8, 32.3, 28.7, 28.2, 27.4, 26.42, 26.36, 23.7, 23.2, 22.5, 18.8, 17.6, 16.4, 15.7.

HR-MS (ESI-TOF) calcd for $C_{51}H_{61}NNaO_4^+$ $[M+Na]^+$ 774.4493; found 774.4496.

3-([1,1'-Biphenyl]-4-yl)-3-(2-ethylbutyl)-2,3-dihydroisoquinoline-1,4-dione (3f)



3f

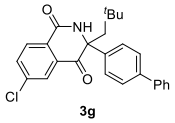
Following typical procedure A, the reaction of **1f** (47.0 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol)，4CzIPN (1.6 mg, 0.002 mmol), DIPEA (10.4 mg, 0.08 mmol) in DCM (1.0 mL) for 16 h afforded **3f** as white solid (8.0 mg, 20% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 7.8 Hz, 1H), 8.01 (d, *J* = 7.8 Hz, 1H), 7.78 (t, *J* = 7.6 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.61 – 7.56 (m, 2H), 7.55 – 7.49 (m, 4H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 1H), 6.63 (brs, 1H), 2.79 (dd, *J* = 14.6, 7.0 Hz, 1H), 2.00 (dd, *J* = 14.6, 5.0 Hz, 1H), 1.56 – 1.48 (m, 1H), 1.36 – 1.30 (m, 2H), 1.25 – 1.19 (m, 2H), 0.86 – 0.78 (m, 6H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.8, 163.2, 141.2, 140.5, 140.2, 135.1, 133.4, 131.12, 131.10, 129.0, 128.5, 127.74, 127.69, 127.3, 127.2, 126.2, 70.7, 43.9, 36.8, 26.2, 26.0, 10.7, 10.2.

HR-MS (ESI-TOF) calcd for $C_{27}H_{28}NO_2^+$ $[M+H]^+$ 398.2115; found 398.2114.

3-([1,1'-Biphenyl]-4-yl)-6-chloro-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3g)



3g

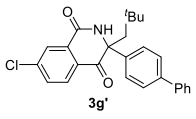
Following typical procedure A, the reaction of **1g** (50.6 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3g** and **3g'** as white solid (28.8 mg, 69% yield, rr = 1:1).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.26 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 2.2 Hz, 1H), 7.72 (dd, *J* = 8.4, 2.2 Hz, 1H), 7.58 – 7.48 (m, 6H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.37 – 7.31 (m, 1H), 6.74 (brs, 1H), 3.10 (d, *J* = 14.9 Hz, 1H), 1.87 (d, *J* = 14.9 Hz, 1H), 1.00 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.4, 161.9, 141.4, 140.7, 140.3, 140.1, 135.1, 132.1, 130.4, 129.3, 129.0, 127.8, 127.3, 127.2, 126.0, 71.3, 52.6, 31.9, 31.7.

HR-MS (ESI-TOF) calcd for $C_{26}H_{25}ClNO_2^+$ $[M+H]^+$ 418.1568; found 418.1569.

3-([1,1'-Biphenyl]-4-yl)-7-chloro-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3g')

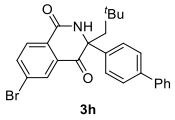


3g'

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 2.1 Hz, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.62 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.59 – 7.48 (m, 6H), 7.44 – 7.37 (m, 2H), 7.37 – 7.30 (m, 1H), 6.77 (brs, 1H), 3.11 (d, *J* = 14.9 Hz, 1H), 1.90 (d, *J* = 14.9 Hz, 1H), 1.01 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 191.4, 161.5, 142.1, 141.4, 140.9, 140.1, 133.7, 132.5, 129.24, 129.19, 129.0, 128.6, 127.8, 127.2, 126.0, 71.0, 52.6, 32.0, 31.7.
HR-MS (ESI-TOF) calcd for $C_{26}H_{25}ClNO_2^+$ $[M+H]^+$ 418.1568; found 418.1570.

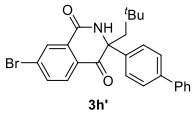
3-([1,1'-Biphenyl]-4-yl)-6-bromo-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3h)



3h

Following typical procedure A, the reaction of **1h** (58.5 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3h** and **3h'** as white solid (33.2 mg, 72% yield, rr = 1:1).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.18 (d, *J* = 8.3 Hz, 1H), 8.14 (d, *J* = 2.0 Hz, 1H), 7.89 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.57 – 7.48 (m, 6H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 1H), 6.55 (brs, 1H), 3.11 (d, *J* = 14.9 Hz, 1H), 1.89 (d, *J* = 14.9 Hz, 1H), 1.01 (s, 9H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.2, 161.9, 141.5, 140.7, 140.1, 138.1, 132.1, 130.4, 130.3, 129.7, 129.0, 128.7, 127.84, 127.82, 127.2, 126.0, 71.3, 52.6, 32.0, 31.7.
HR-MS (ESI-TOF) calcd for $C_{26}H_{24}BrNNaO_2^+$ $[M+Na]^+$ 484.0883; found 484.0884.

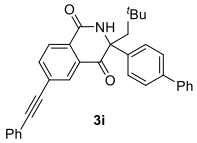
3-([1,1'-Biphenyl]-4-yl)-7-bromo-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3h')



3h'

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.47 (d, *J* = 2.0 Hz, 1H), 7.88 (d, *J* = 8.3 Hz, 1H), 7.80 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.58 – 7.48 (m, 6H), 7.44 – 7.38 (m, 2H), 7.37 – 7.31 (m, 1H), 6.58 (brs, 1H), 3.11 (d, *J* = 14.9 Hz, 1H), 1.89 (d, *J* = 14.9 Hz, 1H), 1.01 (s, 9H).
^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.6, 161.3, 141.4, 140.8, 140.1, 136.7, 132.3, 131.6, 130.8, 129.6, 129.1, 129.0, 127.8, 127.2, 126.0, 71.0, 52.6, 32.0, 31.7.
HR-MS (ESI-TOF) calcd for $C_{26}H_{24}BrNNaO_2^+$ $[M+Na]^+$ 484.0883; found 484.0884.

3-([1,1'-Biphenyl]-4-yl)-3-neopentyl-6-(phenylethynyl)-2,3-dihydroisoquinoline-1,4-dione (3i)



3i

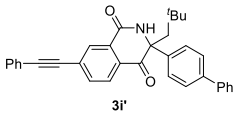
Following typical procedure A, the reaction of **1i** (62.5 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3i** and **3i'** as white solid (32.0 mg, 66% yield, rr = 1:1).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 8.0 Hz, 1H), 8.16 (d, *J* = 1.8 Hz, 1H), 7.88 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.60 – 7.56 (m, 2H), 7.55 – 7.48 (m, 6H), 7.43 – 7.38

(m, 2H), 7.39 – 7.30 (m, 4H), 6.66 (brs, 1H), 3.13 (d, *J* = 14.9 Hz, 1H), 1.90 (d, *J* = 14.9 Hz, 1H), 1.03 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.8, 162.2, 141.3, 141.0, 140.2, 137.4, 132.0, 131.0, 130.4, 129.9, 129.2, 128.98, 128.95, 128.7, 128.6, 127.78, 127.76, 127.2, 126.0, 122.4, 93.8, 87.7, 71.1, 52.6, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{34}H_{30}NO_2^+$ $[M+H]^+$ 484.2271; found 484.2271.

3-([1,1'-Biphenyl]-4-yl)-3-neopentyl-7-(phenylethynyl)-2,3-dihydroisoquinoline-1,4-dione (3i')



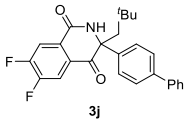
3i'

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.46 (s, 1H), 8.02 (d, *J* = 8.0 Hz, 1H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.64 – 7.60 (m, 2H), 7.58 – 7.49 (m, 6H), 7.43 – 7.35 (m, 5H), 7.33 (t, *J* = 7.4 Hz, 1H), 7.20 (brs, 1H), 3.14 (d, *J* = 14.8 Hz, 1H), 1.96 (d, *J* = 14.8 Hz, 1H), 1.04 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.8, 162.2, 141.2, 141.2, 140.2, 135.8, 132.0, 131.5, 131.2, 130.6, 129.8, 129.3, 128.9, 128.6, 127.7, 127.5, 127.2, 126.1, 122.4, 94.9, 88.1, 70.9, 52.8, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{34}H_{29}NNaO_2^+$ $[M+Na]^+$ 506.2091; found 506.2092.

3-([1,1'-Biphenyl]-4-yl)-6,7-difluoro-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3j)



3j

Following typical procedure A, the reaction of **1j** (50.9 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3j** as white solid (28.9 mg, 69% yield).

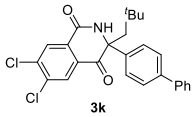
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.10 (dd, *J* = 9.9, 7.3 Hz, 1H), 7.81 (dd, *J* = 9.6, 7.4 Hz, 1H), 7.57 – 7.49 (m, 6H), 7.45 – 7.37 (m, 2H), 7.37 – 7.32 (m, 1H), 6.73 (brs, 1H), 3.10 (d, *J* = 14.9 Hz, 1H), 1.90 (d, *J* = 14.9 Hz, 1H), 1.01 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.4, 160.8, 154.9 (dd, *J* = 261.1, 13.2 Hz), 153.9 (dd, *J* = 259.5, 13.5 Hz), 141.5, 140.6, 140.0, 129.3 (dd, *J* = 6.7, 3.5 Hz), 129.0, 128.8 (dd, *J* = 5.5, 3.6 Hz), 127.9, 127.2, 125.9, 118.0 (d, *J* = 19.7 Hz), 116.7 (d, *J* = 18.9 Hz), 71.3, 52.7, 32.0, 31.7.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -124.7 (ddd, *J* = 20.9, 10.0, 7.4 Hz, 1F), -127.7 (ddd, *J* = 20.8, 9.7, 7.2 Hz,1F).

HR-MS (ESI-TOF) calcd for $C_{26}H_{24}F_2NO_2^+$ $[M+H]^+$ 420.1770; found 420.1769.

3-([1,1'-Biphenyl]-4-yl)-6,7-dichloro-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3k)



3k

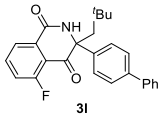
Following typical procedure A, the reaction of **1k** (56.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3k** as white solid (27.5 mg, 61% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.38 (s, 1H), 8.08 (s, 1H), 7.56 – 7.49 (m, 6H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 6.88 (brs, 1H), 3.10 (d, *J* = 14.9 Hz, 1H), 1.91 (d, *J* = 14.9 Hz, 1H), 1.01 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 190.6, 161.0, 141.5, 140.5, 140.3, 140.0, 138.7, 130.7, 130.3, 130.0, 129.4, 129.0, 127.88, 127.85, 127.2, 125.9, 71.4, 52.6, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{26}H_{24}Cl_2NO_2^+$ $[M+H]^+$ 452.1179; found 452.1180.

3-([1,1'-Biphenyl]-4-yl)-5-fluoro-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3l)



3l

Following typical procedure A, the reaction of **1l** (47.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3l** and **3l'** as white solid (18.0 mg, 45% yield, rr = 1:1).

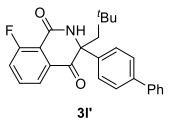
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 7.8 Hz, 1H), 7.67 – 7.60 (m, 1H), 7.60 – 7.55 (m, 2H), 7.55 – 7.49 (m, 4H), 7.49 – 7.42 (m, 1H), 7.43 – 7.38 (m, 2H), 7.36 – 7.30 (m, 1H), 6.44 (brs, 1H), 3.05 (d, *J* = 14.9 Hz, 1H), 1.88 (d, *J* = 14.9 Hz, 1H), 1.02 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.5 (d, *J* = 3.4 Hz), 161.9 (d, *J* = 266.6 Hz), 160.2 (d, *J* = 3.5 Hz), 141.4, 140.7, 140.1, 134.7 (d, *J* = 9.5 Hz), 132.8, 129.0, 127.80, 127.78, 127.2, 126.0, 123.9 (d, *J* = 5.6 Hz), 123.8 (d, *J* = 13.0 Hz), 118.4 (d, *J* = 4.4 Hz), 70.8, 52.6, 31.9, 31.8.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -109.86 – -109.92 (m, 1F).

HR-MS (ESI-TOF) calcd for $C_{26}H_{25}FNO_2^+$ $[M+H]^+$ 402.1864; found 402.1862.

3-([1,1'-Biphenyl]-4-yl)-8-fluoro-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3l')



3l'

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.15 (d, *J* = 7.8 Hz, 1H), 7.73 (td, *J* = 8.0, 4.4 Hz, 1H), 7.57 – 7.49 (m, 6H), 7.43 – 7.38 (m, 2H), 7.38 – 7.31 (m, 2H), 6.68 (brs, 1H), 3.07 (d, *J* = 14.9 Hz, 1H), 1.87 (d, *J* = 14.9 Hz, 1H), 1.03 (s, 9H).

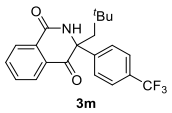
^{13}C NMR (151 MHz, Chloroform-*d*) δ 189.3 (d, *J* = 1.3 Hz), 161.6 (d, *J* = 3.2 Hz), 161.3 (d, *J* = 269.3 Hz), 141. 4, 140.6, 140.1, 136.3 (d, *J* = 9.8 Hz), 132.7, 129.0, 127.81,

127.79, 127.2, 126.0, 124.7 (d, J = 3.7 Hz), 122.0 (d, J = 21.4 Hz), 119.2 (d, J = 5.4 Hz), 71.2, 52.0, 31.9, 31.8.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -110.87 – -110.92 (m, 1F)

HR-MS (ESI-TOF) calcd for $C_{26}H_{25}FNO_2^+$ $[M+H]^+$ 402.1864; found 402.1862.

3-Neopentyl-3-(4-(trifluoromethyl)phenyl)-2,3-dihydroisoquinoline-1,4-dione (3m)



3m

Following typical procedure A, the reaction of **1a** (44.5 mg, 0.18 mmol), **2b** (21.3 mg, 0.10 mmol) for 16 h afforded **3m** as white solid (19.0 mg, 51% yield).

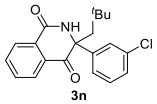
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (d, J = 7.8 Hz, 1H), 8.01 (d, J = 7.8 Hz, 1H), 7.79 (tt, J = 7.6, 1.4 Hz, 1H), 7.72 – 7.64 (m, 3H), 7.58 – 7.53 (m, 2H), 7.10 (brs, 1H), 3.07 (d, J = 14.8 Hz, 1H), 1.87 (d, J = 14.8 Hz, 1H), 1.00 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.1, 162.6, 146.3, 135.3, 133.6, 131.0, 130.8, 130.5 (q, J = 32.7 Hz), 128.6, 127.4, 126.1, 126.0 (q, J = 3.7 Hz), 123.9 (q, J = 272.4 Hz), 70.9, 52.9, 32.1, 31.7.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.73 (s, 3F).

HR-MS (ESI-TOF) calcd for $C_{21}H_{21}F_3NO_2^+$ $[M+H]^+$ 376.1519; found 376.1521.

3-(3-Chlorophenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3n)



3n

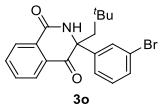
Following typical procedure A, the reaction of **1a** (44.5 mg, 0.18 mmol), **2c** (17.9 mg, 0.10 mmol) for 16 h afforded **3n** as white solid (17.7 mg, 52% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, J = 7.8, 1.3 Hz, 1H), 8.02 (dd, J = 7.8, 1.3 Hz, 1H), 7.79 (td, J = 7.6, 1.4 Hz, 1H), 7.69 (td, J = 7.6, 1.4 Hz, 1H), 7.54 – 7.51 (m, 1H) , 7.45 – 7.38 (m, 1H), 7.25 – 7.19 (m, 2H), 6.74 (brs, 1H), 3.03 (d, J = 14.8 Hz, 1H), 1.83 (d, J = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.0, 162.4, 144.4, 135.2, 135.1, 133.5, 130.9, 130.8, 130.2, 128.6, 128.5, 127.4, 126.0, 123.8, 70.6, 52.9, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{20}H_{20}ClNNaO_2^+$ $[M+Na]^+$ 364.1075; found 364.1075.

3-(3-Bromophenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3o)



3o

Following typical procedure A, the reaction of **1a** (44.5 mg, 0.18 mmol), **2d** (22.3 mg, 0.10 mmol) for 16 h afforded **3o** as white solid (20.0 mg, 52% yield).

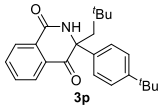
^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, J = 7.8, 1.3 Hz, 1H), 8.02 (dd, J = 7.8,

1.3 Hz, 1H), 7.80 (td, *J* = 7.6, 1.4 Hz, 1H), 7.72 – 7.65 (m, 2H), 7.46 (ddd, *J* = 8.1, 1.9, 1.0 Hz, 1H), 7.38 (ddd, *J* = 8.1, 1.9, 1.0 Hz, 1H), 7.17 (t, *J* = 8.0 Hz, 1H), 6.65 (brs, 1H), 3.02 (d, *J* = 14.8 Hz, 1H), 1.82 (d, *J* = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.0, 162.3, 144.6, 135.2, 133.5, 131.4, 130.9, 130.8, 130.5, 128.8, 128.6, 127.4, 124.2, 123.3, 70.6, 53.0, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{20}H_{20}BrNNaO_2^+$ $[M+Na]^+$ 408.0570; found 408.0572.

3-(4-(*tert*-Butyl)phenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3p)



3p

Following typical procedure A, the reaction of **1a** (44.5 mg, 0.18 mmol), **2e** (20.1 mg, 0.10 mmol), HOAc (4.8 mg, 0.08 mmol) for 16 h afforded **3p** as white solid (13.1 mg, 36% yield).

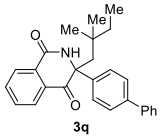
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2e** (20.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3p** as white solid (15.2 mg, 42% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 7.8 Hz, 1H), 8.01 (d, *J* = 7.8 Hz, 1H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.47 – 7.42 (m, 2H), 7.32 – 7.28 (m, 2H), 6.92 (brs, 1H), 3.10 (d, *J* = 14.8 Hz, 1H), 1.84 (d, *J* = 14.8 Hz, 1H), 1.24 (s, 9H), 0.99 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.6, 162.6, 151.2, 139.4, 134.9, 133.2, 131.2, 131.0, 128.5, 127.3, 126.0, 125.2, 70.8, 52.8, 34.5, 31.9, 31.7, 31.3.

HR-MS (ESI-TOF) calcd for $C_{24}H_{29}NNaO_2^+$ $[M+Na]^+$ 386.2091; found 386.2093.

3-([1,1'-Biphenyl]-4-yl)-3-(2,2-dimethylbutyl)-2,3-dihydroisoquinoline-1,4-dione (3q)



3q

Following typical procedure B, the reaction of **1n** (49.5 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3q** as white solid (27.0 mg, 68% yield).

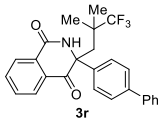
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.32 (d, *J* = 7.8 Hz, 1H), 8.04 (d, *J* = 7.8 Hz, 1H), 7.78 (t, *J* = 7.6 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.60 (d, *J* = 8.7 Hz, 2H), 7.55 – 7.49 (m, 4H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.35 – 7.30 (m, 1H), 6.80 (brs, 1H), 3.09 (d, *J* = 15.0 Hz, 1H), 1.91 (d, *J* = 15.0 Hz, 1H), 1.36 (q, *J* = 7.4 Hz, 2H), 1.01 (s, 3H), 0.91 (s, 3H), 0.88 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.5, 162.6, 141.4, 141.2, 140.2, 135.0, 133.4,

131.1, 131.0, 128.9, 128.5, 127.71, 126.69, 127.4, 127.2, 126.1, 70.8, 50.7, 37.1, 34.5, 28.9, 27.9, 8.6.

HR-MS (ESI-TOF) calcd for $C_{27}H_{27}NNaO_2^+$ $[M+Na]^+$ 420.1934; found 420.1938.

3-([1,1'-Biphenyl]-4-yl)-3-(3,3,3-trifluoro-2,2-dimethylpropyl)-2,3-dihydroisoquinoline-1,4-dione (3r)



3r

Following typical procedure B, the reaction of **1o** (56.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3r** as white solid (21.9 mg, 50% yield).

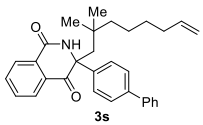
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.31 (d, *J* = 7.8 Hz, 1H), 8.05 (d, *J* = 7.8 Hz, 1H), 7.80 (t, *J* = 7.6 Hz, 1H), 7.70 (t, *J* = 7.6 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* =7.8 Hz, 2H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 6.77 (brs, 1H), 3.41 (d, *J* = 15.3 Hz, 1H), 2.11 (d, *J* = 15.3 Hz, 1H), 1.32 (s, 3H), 1.13 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.7, 162.8, 141.7, 140.2, 140.0, 135.4, 133.7, 130.8, 130.7, 129.04 (q, *J* = 283.3 Hz), 129.00, 128.7, 128.0, 127.9, 127.6, 127.2, 126.0, 69.9, 43.8, 41.2 (q, *J* = 24.5 Hz), 22.8 (q, *J* = 2.0 Hz), 21.9 (q, *J* = 2.0 Hz).

^{19}F NMR (376 MHz, Chloroform-*d*) δ -79.12 (s, 3F).

HR-MS (ESI-TOF) calcd for $C_{26}H_{22}F_3NNaO_2^+$ $[M+Na]^+$ 460.1495; found 460.1496.

3-([1,1'-Biphenyl]-4-yl)-3-(2,2-dimethyloct-7-en-1-yl)-2,3-dihydroisoquinoline-1,4-dione (3s)



3s

Following typical procedure B, the reaction of **1p** (59.3 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3s** as white solid (27.1 mg, 60% yield).

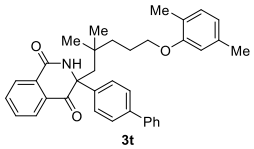
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.32 (d, *J* = 7.8 Hz, 1H), 8.03 (d, *J* = 7.8 Hz, 1H), 7.79 (t, *J* = 7.6 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.59 (d, *J* = 8.2 Hz, 2H), 7.55 – 7.47 (m, 4H), 7.40 (t, *J* = 7.4 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 1H), 6.53 (brs, 1H), 5.78 (ddt, *J* = 17.0, 10.2, 6.7 Hz, 1H), 4.98 (d, *J* = 17.0, Hz, 1H), 4.93 (d, *J* = 10.2 Hz, 1H), 3.09 (d, *J* = 14.9 Hz, 1H), 2.03 (q, *J* = 7.5 Hz, 2H), 1.90 (d, *J* = 14.9 Hz, 1H), 1.34 – 1.27 (m, 6H), 1.02 (s, 3H), 0.91 (s, 3H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.3, 162.4, 141.2, 141.1, 140.1, 138.9, 134.9, 133.3, 130.9, 130.8, 128.8, 128.4, 127.61, 127.59, 127.3, 127.1, 125.9, 114.4, 70.7, 50.9,

44.7, 34.3, 33.7, 29.6, 29.3, 28.4, 23.5.

HR-MS (ESI-TOF) calcd for $C_{31}H_{33}NNaO_2^+$ $[M+Na]^+$ 474.2404; found 474.2406.

3-([1,1'-Biphenyl]-4-yl)-3-(5-(2,5-dimethylphenoxy)-2,2-dimethylpentyl)-2,3-dihydroisoquinoline-1,4-dione (3t)



3t

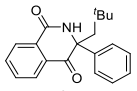
Following typical procedure B, the reaction of **1q** (73.7 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3t** as white solid (31.3 mg, 59% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.32 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.05 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.76 (td, *J* = 7.6, 1.4 Hz, 1H), 7.70 – 7.61 (m, 3H), 7.56 – 7.48 (m, 4H), 7.44 – 7.38 (m, 2H), 7.37 – 7.30 (m, 1H), 7.24 (s, 1H), 7.00 (d, *J* = 7.5 Hz, 1H), 6.66 (t, *J* = 7.5, Hz, 1H), 6.58 (brs, 1H), 3.89 (t, *J* = 6.4 Hz, 2H), 3.17 (d, *J* = 14.9 Hz, 1H), 2.29 (s, 3H), 2.16 (s, 3H), 2.00 (d, *J* = 14.9 Hz, 1H), 1.90 – 1.81 (m, 2H), 1.56 – 1.47 (m, 2H), 1.12 (s, 3H), 0.99 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.5, 162.8, 157.1, 141.4, 141.2, 140.2, 136.5, 135.0, 133.4, 131.1, 130.9, 130.4, 128.9, 128.5, 127.7, 127.4, 127.2, 126.1, 123.7, 120.8, 112.1, 70.8, 68.4, 50.8, 41.3, 34.3, 28.3, 27.0, 24.5, 21.5, 15.9.

HR-MS (ESI-TOF) calcd for $C_{36}H_{37}NNaO_3^+$ $[M+Na]^+$ 554.2666; found 554.2669.

3-Neopentyl-3-phenyl-2,3-dihydroisoquinoline-1,4-dione (3u)



3u

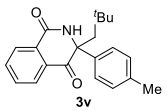
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2f** (14.5 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3u** as white solid (15.4 mg, 50% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.00 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.77 (td, *J* = 7.6, 1.4 Hz, 1H), 7.66 (td, *J* = 7.6, 1.4 Hz, 1H), 7.55 – 7.49 (m, 2H), 7.33 – 7.27 (m, 2H), 7.26 – 7.21 (m, 1H), 6.52 (brs, 1H), 3.09 (d, *J* = 14.8 Hz, 1H), 1.84 (d, *J* = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.4, 162.6, 142.3, 135.0, 133.4, 131.0, 130.9, 129.0, 128.5, 128.3, 127.4, 125.5, 71.0, 52.7, 31.9, 31.7.

HR-MS (ESI-TOF) calcd for $C_{20}H_{21}NNaO_2^+$ $[M+Na]^+$ 330.1465; found 330.1465.

3-Neopentyl-3-(*p*-tolyl)-2,3-dihydroisoquinoline-1,4-dione (3v)



3v

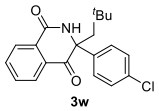
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2g** (15.9 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3v** as white solid (14.0 mg, 44% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.00 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.76 (td, *J* = 7.6, 1.4 Hz, 1H), 7.66 (td, *J* = 7.6, 1.4 Hz, 1H), 7.43 – 7.34 (m, 2H), 7.13 – 7.08 (m, 2H), 6.50 (brs, 1H), 3.07 (d, *J* = 14.8 Hz, 1H), 2.27 (s, 3H), 1.81 (d, *J* = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 192.5, 162.6, 139.4, 138.2, 134.9, 133.3, 131.02, 130.95, 129.7, 128.4, 127.4, 125.4, 70.8, 52.6, 31.9, 31.7, 21.0.

HR-MS (ESI-TOF) calcd for $C_{21}H_{23}NNaO_2^+$ $[M+Na]^+$ 344.1621; found 344.1622.

3-(4-Chlorophenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3w)



3w

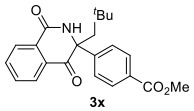
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2h** (17.9 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3w** as white solid (22.9 mg, 67% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.01 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.79 (td, *J* = 7.6, 1.4 Hz, 1H), 7.69 (td, *J* = 7.6, 1.4 Hz, 1H), 7.51 – 7.44 (m, 2H), 7.29 – 7.25 (m, 2H), 6.79 (s, 1H), 3.04 (d, *J* = 14.8 Hz, 1H), 1.84 (d, *J* = 14.8 Hz, 1H), 1.00 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.2, 162.5, 140.9, 135.2, 134.5, 133.5, 131.0, 130.8, 129.1, 128.6, 127.4, 127.1, 70.6, 52.7, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{20}H_{21}ClNO_2^+$ $[M+H]^+$ 342.1255; found 342.1256.

Methyl 4-(3-neopentyl-1,4-dioxo-1,2,3,4-tetrahydroisoquinolin-3-yl)benzoate (3x)



3x

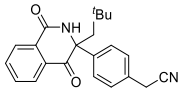
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2i** (20.3 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3x** as white solid (17.2 mg, 47% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 7.8 Hz, 1H), 8.00 (d, *J* = 7.8 Hz, 1H), 7.95 (d, *J* = 8.3 Hz, 2H), 7.79 (t, *J* = 7.6 Hz, 1H), 7.67 (t, *J* = 7.6 Hz, 1H), 7.61 (d, *J* = 8.3 Hz, 2H), 6.68 (brs, 1H), 3.87 (s, 3H), 3.07 (d, *J* = 14.8 Hz, 1H), 1.87 (d, *J* = 14.8 Hz, 1H), 1.00 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.0, 166.6, 162.5, 147.1, 135.2, 133.5, 131.0, 130.8, 130.2, 130.1, 128.6, 127.4, 125.7, 71.1, 52.6, 52.4, 32.0, 31.7.

HR-MS (ESI-TOF) calcd for $C_{22}H_{24}NO_4^+$ $[M+H]^+$ 366.1700; found 366.1690.

2-(4-(3-Neopentyl-1,4-dioxo-1,2,3,4-tetrahydroisoquinolin-3-yl)phenyl)acetonitrile (3y)



3y

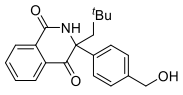
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2j** (18.4 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3y** as white solid (18.7 mg, 54% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.00 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.79 (td, *J* = 7.6, 1.4 Hz, 1H), 7.68 (td, *J* = 7.6, 1.4 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.29 – 7.25 (m, 2H), 6.76 (brs, 1H), 3.68 (s, 2H), 3.06 (d, *J* = 14.8 Hz, 1H), 1.84 (d, *J* = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.3, 162.5, 142.5, 135.2, 133.5, 131.0, 130.8, 130.1, 128.6, 128.6, 127.4, 126.5, 117.5, 70.8, 52.7, 32.0, 31.7, 23.2.

HR-MS (ESI-TOF) calcd for $C_{22}H_{22}N_2NaO_2^+$ $[M+Na]^+$ 369.1573; found 369.1574.

3-(4-(Hydroxymethyl)phenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3z)



3z

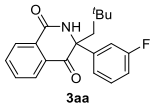
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2k** (17.5 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3z** as white solid (17.8 mg, 53% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.00 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.78 (td, *J* = 7.6, 1.4 Hz, 1H), 7.67 (td, *J* = 7.6, 1.4 Hz, 1H), 7.53 – 7.48 (m, 2H), 7.32 – 7.28 (m, 2H), 6.54 (s, 1H), 4.63 (s, 2H), 3.08 (d, *J* = 14.8 Hz, 1H), 1.83 (d, *J* = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.4, 162.8, 141.5, 141.2, 135.0, 133.4, 131.0, 130.9, 128.5, 127.5, 127.3, 125.8, 70.8, 64.5, 52.7, 31.9, 31.7.

HR-MS (ESI-TOF) calcd for $C_{21}H_{24}NO_3^+$ $[M+H]^+$ 338.1751; found 338.1744.

3-(3-Fluorophenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3aa)



3aa

Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2l** (16.3 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3aa** as white solid (18.5 mg, 57% yield).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.02 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.79 (td, *J* = 7.6, 1.4 Hz, 1H), 7.68 (td, *J* = 7.6, 1.4 Hz, 1H), 7.33 – 7.28

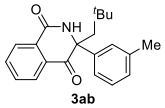
(m, 1H), 7.28 – 7.26 (m, 1H), 7.26 – 7.22 (m, 1H), 6.93 (tt, *J* = 8.3, 1.3 Hz, 1H), 6.80 (brs, 1H), 3.03 (d, *J* = 14.8 Hz, 1H), 1.86 (d, *J* = 14.8 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.1, 163.1 (d, *J* = 247.2 Hz), 162.4, 145.0 (d, *J* = 6.5 Hz), 135.2, 133.5, 131.0, 130.8, 130.4 (d, *J* = 8.2 Hz), 128.6, 127.4, 121.1 (d, *J* = 2.8 Hz), 115.3 (d, *J* = 21.2 Hz), 113.2 (d, *J* = 23.5 Hz), 70.7 (d, *J* = 1.6 Hz), 52.8, 32.0, 31.7.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -111.11 – -111.25 (m, 1F).

HR-MS (ESI-TOF) calcd for $C_{20}H_{21}FNO_2^+$ $[M+H]^+$ 326.1551; found 326.1551.

3-Neopentyl-3-(*m*-tolyl)-2,3-dihydroisoquinoline-1,4-dione (3ab)



3ab

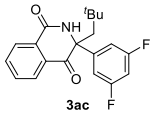
Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2m** (15.9 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3ab** as white solid (13.2 mg, 41% yield).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 7.7 Hz, 1H), 8.01 (d, *J* = 7.7 Hz, 1H), 7.77 (t, *J* = 7.6 Hz, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.33 – 7.29 (m, 2H), 7.18 (t, *J* = 8.0 Hz, 1H), 7.05 (d, *J* = 7.5 Hz, 1H), 6.65 (brs, 1H), 3.06 (d, *J* = 14.9 Hz, 1H), 2.30 (s, 3H), 1.78 (d, *J* = 14.9 Hz, 1H), 0.97 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.5, 162.6, 142.3, 138.8, 134.9, 133.3, 131.1, 131.0, 129.0, 128.9, 128.5, 127.4, 126.1, 122.6, 71.0, 52.8, 31.9, 31.7, 21.8.

HR-MS (ESI-TOF) calcd for $C_{21}H_{24}NO_2^+$ $[M+H]^+$ 322.1802; found 322.1802.

3-(3,5-Difluorophenyl)-3-neopentyl-2,3-dihydroisoquinoline-1,4-dione (3ac)



3ac

Following typical procedure B, the reaction of **1m** (47.0 mg, 0.18 mmol), **2n** (18.1 mg, 0.10 mmol), **4a** (40.8 mg, 0.25 mmol) for 12 h afforded **3ac** as white solid (22.3 mg, 65% yield).

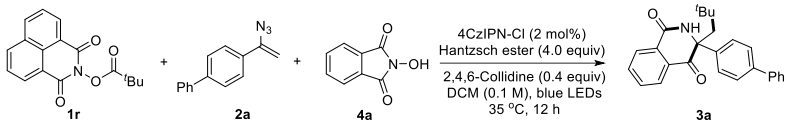
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.31 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.03 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.81 (td, *J* = 7.6, 1.3 Hz, 1H), 7.71 (td, *J* = 7.6, 1.3 Hz, 1H), 7.10 – 7.05 (m, 2H), 6.69 (tt, *J* = 8.4, 2.3 Hz, 1H), 6.63 (brs, 1H), 2.98 (d, *J* = 14.9 Hz, 1H), 1.83 (d, *J* = 14.9 Hz, 1H), 0.99 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 191.8, 163.24 (d, *J* = 249.6 Hz), 163.15 (d, *J* = 249.6 Hz). 162.5, 146.7 (t, *J* = 7.6 Hz), 135.3, 133.5, 131.0, 130.8, 128.6, 127.4, 109.2 (d, *J* = 6.0 Hz), 109.1 (d, *J* = 6.0 Hz), 103.7 (t, *J* = 25.6 Hz). 70.5 (t, *J* = 2.2 Hz), 52.8 (d, *J* = 13.7 Hz), 32.0, 31.6.

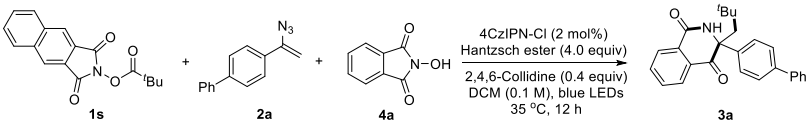
^{19}F NMR (376 MHz, Chloroform-*d*) δ -107.68 – -107.79 (m, 2F).

HR-MS (ESI-TOF) calcd for $C_{20}H_{20}F_2NO_2^+$ $[M+H]^+$ 344.1457; found 344.1458.

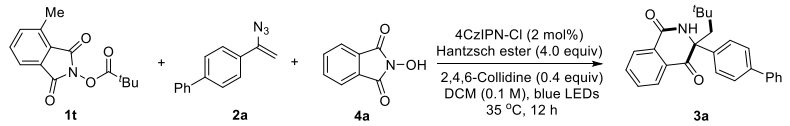
Convergent synthesis of 3a from different NHPIs



To a Schlenk tube were added **1r** (53.5 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4a** (32.6 mg, 0.20 mmol, 2.0 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3a** in 51% (19.5 mg) yield.

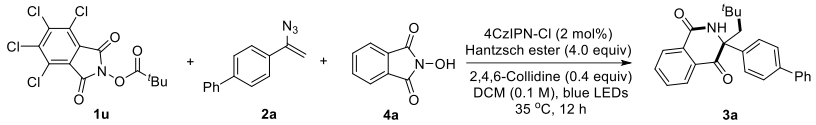


To a Schlenk tube were added **1s** (53.5 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3a** in 69% (26.4 mg) yield.



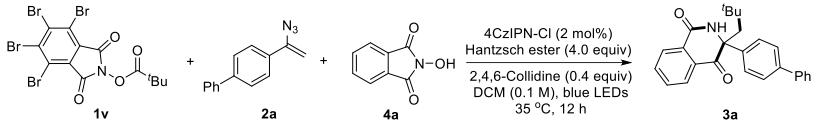
To a Schlenk tube were added **1t** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%). Then the tube was sealed and exposed

to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3a** in 66% (25.3 mg) yield.



1u 2a 4a 3a

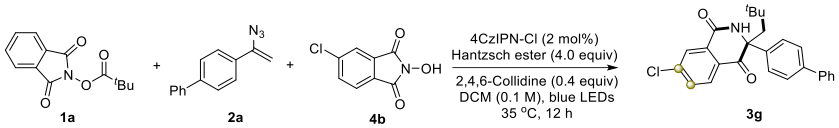
To a Schlenk tube were added **1u** (45.9 mg, 0.12 mmol, 1.2 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3a** in 52% (19.9 mg) yield.



1v 2a 4a 3a

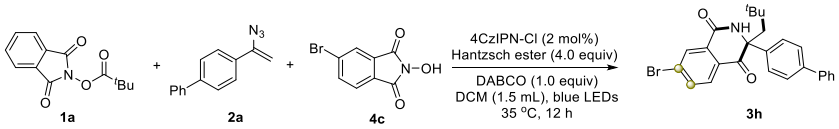
To a Schlenk tube were added **1v** (89.4 mg, 0.16 mmol, 1.6 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3a** in 45% (17.3 mg) yield.

Divergent synthesis of 3 from 1a



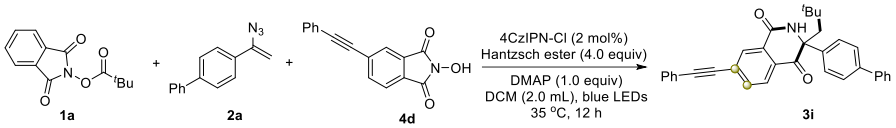
1a 2a 4b 3g

To a Schlenk tube were added **1a** (29.6 mg, 0.12 mmol, 1.2 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4b** (23.6 mg, 0.12 mmol, 1.2 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) 2,4,6-collidine (4.8 μL, 0.04 mmol, 40 mol%). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3g** in 45% (18.8 mg, rr = 1:1) yield.



1a 2a 4c 3h

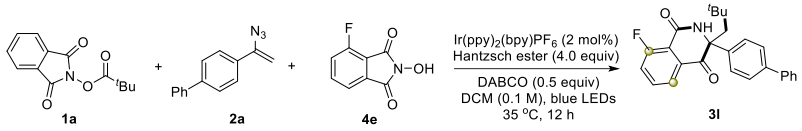
To a Schlenk tube were added **1a** (44.5 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4c** (43.2 mg, 0.18 mmol, 1.8 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv), DABCO (11.2 mg, 0.1 mmol, 1.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.5 mL). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3h** in 46% (21.2 mg, rr = 1:1) yield.



1a 2a 4d 3i

To a Schlenk tube were added **1a** (44.5 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4d** (47.3 mg, 0.18 mmol, 1.8 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv), DMAP (12.1 mg, 0.1 mmol, 1.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (2.0 mL). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL).

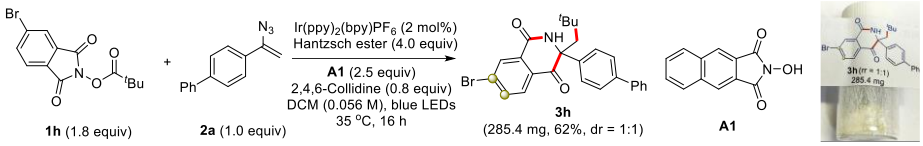
The organic phases were dried over anhydrous sodium sulfate, filtered, and evacuated under vacuum. The residue was purified directly by column chromatography to afford the pure product **3i** in 48% (23.2 mg, rr = 1:1) yield.



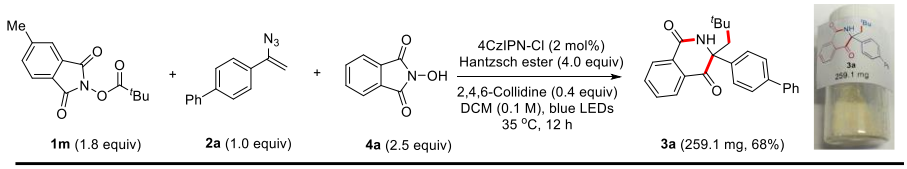
To a Schlenk tube were added **1a** (44.5 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.10 mmol, 1.0 equiv), **4e** (32.6 mg, 0.18 mmol, 1.8 equiv), $Ir(ppy)_2(bpy)PF_6$ (1.6 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv), DABCO (5.6 mg, 0.05 mmol, 50 mol%). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3l** in 48% (19.2 mg, rr = 1:1) yield.

VI. Mechanistic Study

Scale-up reaction

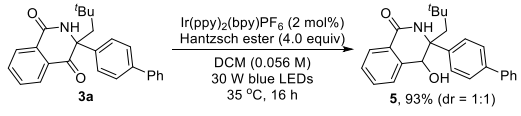


To a Schlenk tube were added **1h** (0.59 g, 1.8 mmol, 1.8 equiv), **2a** (0.22 g, 1.0 mmol, 1.0 equiv), **A1** (0.44 g, 2.5 mmol, 2.5 equiv), $Ir(ppy)_2(bpy)PF_6$ (16.0 mg, 0.02 mmol, 2.0 mol%), Hantzsch ester (1.01 g, 4.0 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (18.0 mL) and 2,4,6-collidine (96.0 μL, 0.8 mmol, 0.8 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (20.0 mL) and extracted with DCM (3 × 10.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3h** (285.4 mg, 62%, dr = 1:1).



To a Schlenk tube were added **1m** (0.47 g, 1.8 mmol, 1.8 equiv), **2a** (0.22 g, 1.0 mmol, 1.0 equiv), **4a** (0.41 g, 2.5 mmol, 2.5 equiv), 4CzIPN-Cl (21.0 mg, 0.02 mmol, 2.0 mol%), Hantzsch ester (1.01 g, 4.0 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles, followed by the addition of DCM (10.0 mL) and 2,4,6-collidine (48.0 μL, 0.4 mmol, 0.4 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with stirring 12 h. The reaction was diluted with H_2O (20.0 mL) and extracted with DCM (3 × 10.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **3a** (259.1 mg, 68%).

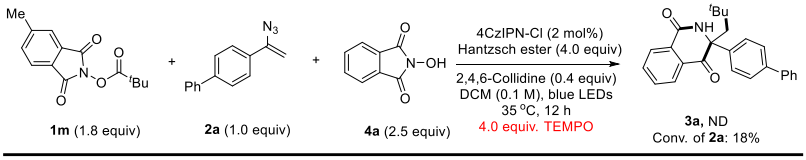
Selective reduction of 3a



To a Schlenk tube were added **3a** (38.3 mg, 0.10 mmol, 1.0 equiv), $Ir(ppy)_2(bpy)PF_6$

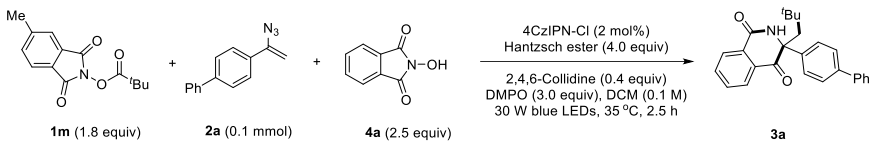
(1.6 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.40 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.8 mL). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C with 16 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **5** (35.8 mg, 93% yield). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.14 (d, *J* = 7.6 Hz, 1H), 7.64 (d, *J* = 8.1 Hz, 2H), 7.62 – 7.58 (m, 4H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.49 – 7.41 (m, 4H), 7.35 (t, *J* = 7.4 Hz, 1H), 6.48 (brs, 1H), 4.77 (s, 1H), 2.23 (d, *J* = 14.9 Hz, 1H), 2.19 (brs, 1H), 1.85 (d, *J* = 14.9 Hz, 1H), 0.78 (s, 9H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 165.1, 140.6, 140.1, 139.0, 138.9, 133.2, 129.05, 128.96, 128.3, 127.9, 127.7, 127.5, 127.1, 127.0, 126.7, 75.0, 65.5, 49.1, 32.0, 31.8. HR-MS (ESI-TOF) calcd for $C_{26}H_{28}NO_2^+$ $[M+H]^+$ 386.2115 found 386.2102.

Radical intermediate quench reactions



To a Schlenk tube were added **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), TEMPO (62.4 mg, 0.40 mmol, 4.0 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The conversion of **2a** (18%) and yield of **3a** (0%) were determined by ^{1}H NMR of the crude reaction mixture using mesitylene as internal standard.

EPR Experiment



To a Schlenk tube were added **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), DMPO (33.9 mg, 0.30 mmol,

3.0 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 2.5 h, then detected the EPR signal.

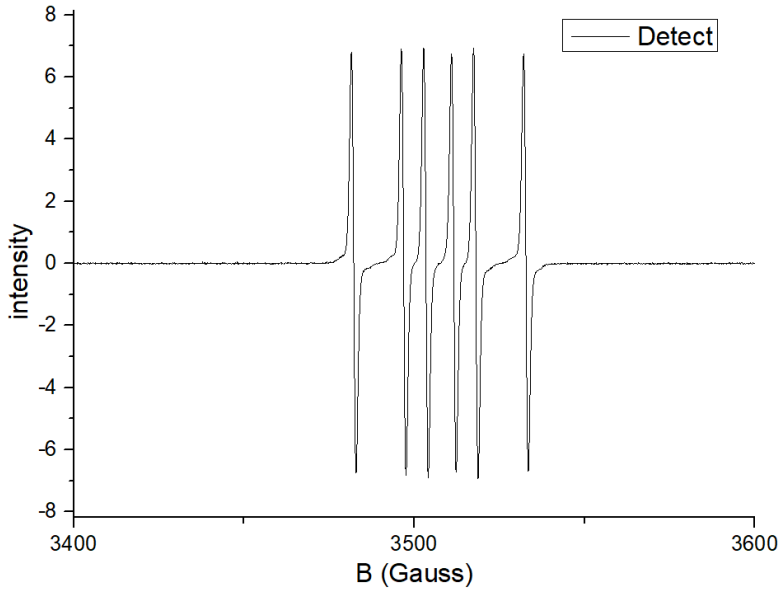
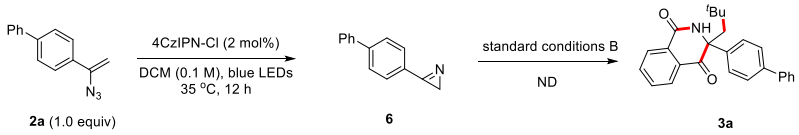


Figure S1. EPR spectra of the reaction mixture.

Excluding 2*H*-azirine as an intermediate

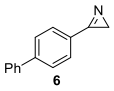


To a Schlenk tube were added **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 12 h. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The combined organic phases were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified directly by column chromatography to afford the pure product **6**.

Then, to a Schlenk tube were added **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **6** (19.3 mg, 0.1 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv). Then the tube was sealed

and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 12 h. The crude reaction mixture was analyzed by ^{1}H NMR spectroscopy and desired product **3a** was not detected.

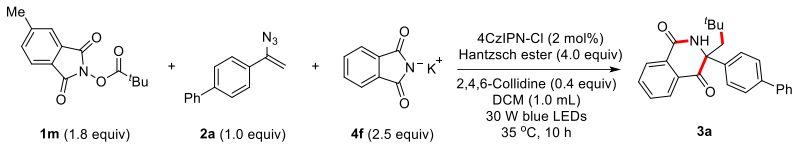
3-([1,1'-biphenyl]-4-yl)-2*H*-azirine (6)[1]



6

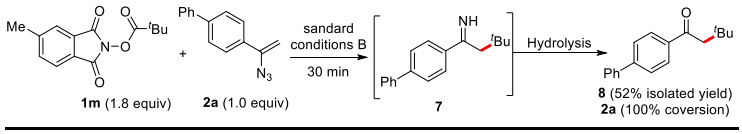
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.00 – 7.97 (m, 2H), 7.81 – 7.78 (m, 2H), 7.67 – 7.65 (m, 2H), 7.51 – 7.47 (m, 2H), 7.44 – 7.40 (m, 1H), 1.82 (s, 2H).

Excluding phthalimide anion as intermediate for the reaction



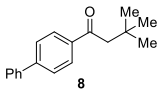
To a Schlenk tube were added **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), **4f** (46.3 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 10 h. The crude reaction mixture was analyzed by ^{1}H NMR spectroscopy and the desired product **3a** was not detected.

Identifying ketimine as an intermediate



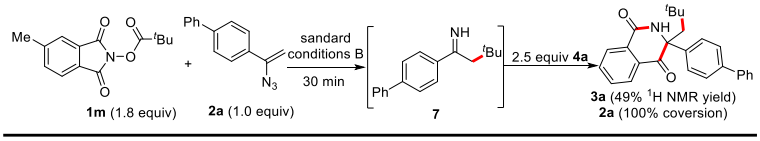
To a Schlenk tube were added **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 30 minutes. The reaction was diluted with H_2O (5.0 mL) and extracted with DCM (3 × 5.0 mL). The organic phases were dried over Na_2SO_4 and concentrated under reduced pressure after filtration. The residue was purified directly by column chromatography to afford the pure product **8** (13.0 mg) in 52% yield.

1-([1,1'-Biphenyl]-4-yl)-3,3-dimethylbutan-1-one (8)[1]



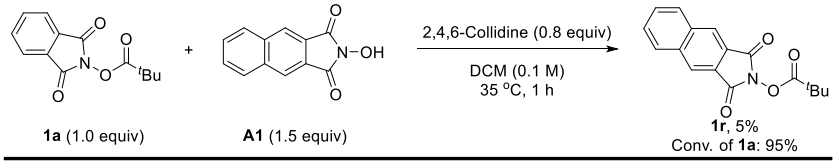
8

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 – 7.99 (m, 2H), 7.70 – 7.61 (m, 4H), 7.50 – 7.44 (m, 2H), 7.42 – 7.37 (m, 1H), 2.89 (s, 2H), 1.09 (s, 9H).



To a Schlenk tube were added **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv). The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv). Then the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 30 minutes. Then **4a** (40.7 mg, 0.25 mmol, 2.5 equiv) was added under N_2, and the tube was sealed and exposed to blue LEDs (30 W LED light bulbs 2.5 cm away from the vial) at 35 °C for 12 h. The reaction mixture was diluted with ethyl acetate and poured into a separatory funnel, washed with brine. The combined organic layers were dried over Na_2SO_4 and concentrated under reduced pressure after filtration. The conversion of **2a** (100%) and yield of **3a** (49%) were determined by ^{1}H NMR of the crude reaction mixture using mesitylene as internal standard.

Cross-over experiment



To a Schlenk tube were added **1a** (24.7 mg, 0.10 mmol, 1.0 equiv), **A1** (32.0 mg, 0.15 mmol, 1.5 equiv), The tube was evacuated and back-filled with nitrogen (3 cycles), followed by the addition of DCM (1.0 mL) and 2,4,6-collidine (9.6 μL, 0.08 mmol, 0.8 equiv). Then the tube was sealed and stirred at 35 °C for 1 h. The reaction mixture was concentrated under reduced pressure. The yield of **1r** (5%) and conv. of **1a** (95%) was determined by ^{1}H NMR of the crude reaction mixture using mesitylene as internal standard.

Quantum yield measurement

Blue LED (λmax = 440 nm) was used for measurement of quantum yield.

Determination of the light intensity at 440 nm

The photon flux of the blue LED light was determined by standard ferrioxalate actinometry,[13-15] the photon flux of the LED (λmax = 440 nm) was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (2.21 g) in H_2SO_4 (30 mL of a 0.05 M solution). A buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (50 mg) and sodium acetate (11.25g) in H_2SO_4 (50 mL of a 0.5 M solution). Both solutions were stored in the dark. To determine the photon flux of the LED, the ferrioxalate solution (2.0 mL) was placed in a cuvette and irradiated for 45 seconds at λmax = 440 nm. After irradiation, the phenanthroline solution (0.35 mL) was added to the cuvette and the mixture was allowed to stand in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A nonirradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated using eq 1.

$$\text{Mol of } Fe^{2+} = \frac{v \cdot \Delta_{A510nm}}{l \cdot \varepsilon} = \frac{(0.00235\ L) \cdot (1.655)}{(1.0\ cm) \cdot 11100 \frac{L}{mol} \cdot cm} = 3.50 \times 10^{-7}\ mol \qquad (1)$$

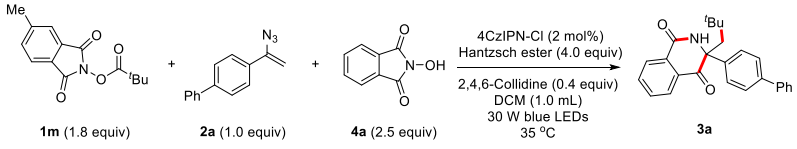
V is the total volume (0.00235 L) of the solution after addition of phenanthroline, ΔA (1.425) is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions (**Figure S2**), l is the path length (1.00 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 $Lmol^{-1}\ cm^{-1}$). The photon flux can be calculated using eq 2.

$$\text{Photon flux} = \frac{mol\ of Fe^{2+}}{\emptyset \cdot t \cdot f} = \frac{3.50 \times 10^{-7}}{(1.01) \cdot (45\ s) \cdot (0.996)} = 7.73 \times 10^{-9}\ einstein/s \qquad (2)$$

Where Φ is the quantum yield for the ferrioxalate actinometer (1.01 at λ = 440 nm), t is the irradiation time (45 s), and f is the fraction of light absorbed at 440 nm by the ferrioxalate actinometer. This value is calculated using eq 3 where A_{440} nm is the absorbance of the ferrioxalate solution at 440 nm.

$$f = 1 - 10^{-A_{440nm}} = 1 - 10^{-2.354} = 0.996 \qquad (3)$$

The photon flux was thus calculated to be 7.73×10^{-9} einstein/s



A cuvette sealed with a rubber stopper was charged with **1m** (47.0 mg, 0.18 mmol, 1.8 equiv), **2a** (22.1 mg, 0.1 mmol, 1.0 equiv), **4a** (40.7 mg, 0.25 mmol, 2.5 equiv), 4CzIPN-Cl (2.1 mg, 0.002 mmol, 2.0 mol%), Hantzsch ester (101.3 mg, 0.4 mmol, 4.0 equiv), 2,4,6-collidine (4.8 μL, 0.04 mmol, 0.4 equiv) in 1 mL DCM under argon atmosphere. The sample was stirred and irradiated (λ = 440 nm) for 1800 s (0.5 h) at room temperature. After irradiation, the solvent was removed. The yield of product

formed was determined as 23% yield (2.3×10^{-5} mol of **3a**) by crude ^{1}H NMR based on a mesitylene standard. The reaction quantum yield (Φ) was determined using eq 5 where the photon flux is 7.73×10^{-9} einstein/s (determined by actinometry as described above), t is the reaction time (1800 s) and f is the fraction of incident light absorbed by the photosensitizer, 4CzIPN-Cl, determined using eq 4. An absorption spectrum of the catalyst (1.25×10^{-4} M) gave an absorbance value of 0.752 at 440 nm (**Figure S3**), indicating that the fraction of light absorbed by the photocatalyst (f) is 0.823.

$$f = 1 - 10^{-A_{440nm}} = 1 - 10^{-0.752} = 0.823 \qquad (4)$$

$$\emptyset = \frac{mol\ of\ product}{flux \cdot t \cdot f} = \frac{2.30\times10^{-5}\ mol}{7.73\times10^{-9}\ einstein/s \times 1800s \times 0.823} = 2.01 \qquad (5)$$

The reaction quantum yield (Φ) was calculated to be 2.01.

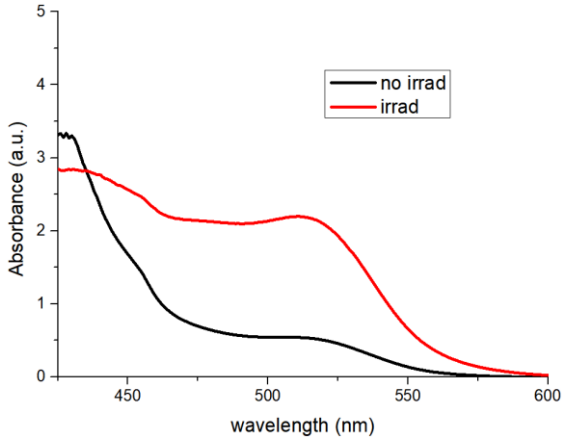


Figure S2. The irradiation experiment and non-irradiation experiment absorption spectra.

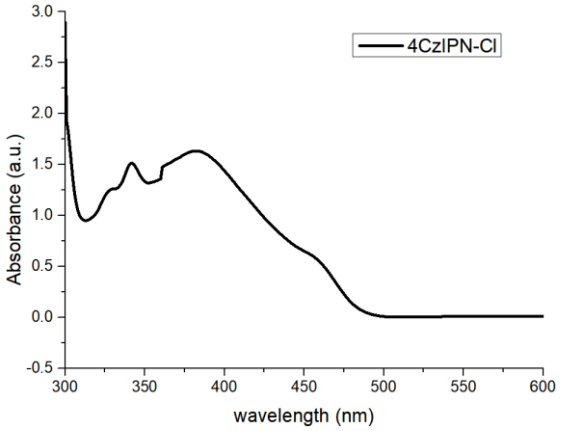
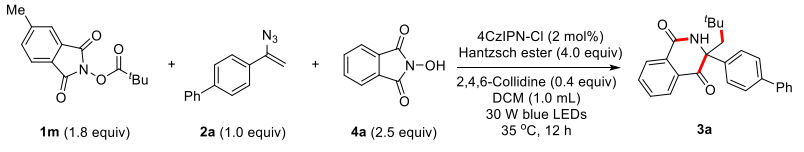


Figure S3. Absorption spectra of 4CzIPN-Cl.

The luminescence quenching experiments



Emission intensities were recorded using FS-5 fluorescence spectrometer (Edinburgh instrument) for all experiments. All 4CzIPN-Cl solutions were excited at 454 nm and the emission intensity was collected at 425-750 nm. In a typical experiment, the DCM as solution of 4CzIPN-Cl (0.5 μM) was added the appropriate amount of quencher in a 4.5 cm quartz cuvette. After degassing with nitrogen for 10 min, the emission spectra of the samples were collected. The results showed that Hantzsch could quench the photoexcited 4CzIPN-Cl effectively, while **1m**, **2a**, **4a** and 2,4,6-collidine were less effective. The emission intensity at 542 nm was observed.

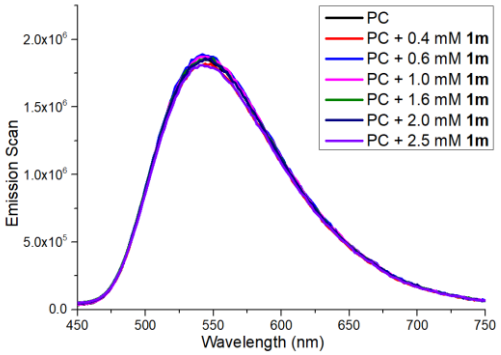


Figure S4. The luminescence quenching of **1m**.

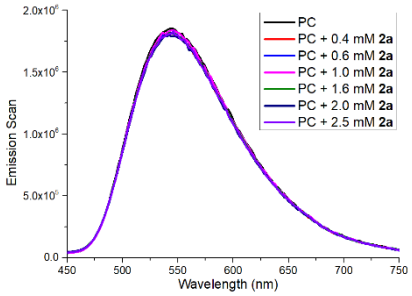


Figure S5. The luminescence quenching of **2a**.

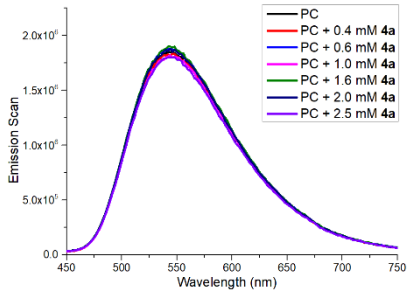


Figure S6. The luminescence quenching of **4a**.

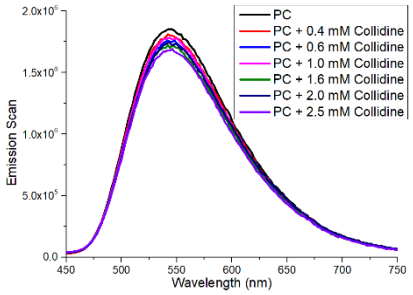


Figure S7. The luminescence quenching of 2,4,6-collidine.

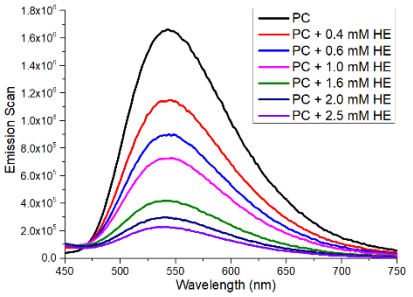


Figure S8. The luminescence quenching of Hantzsch ester.

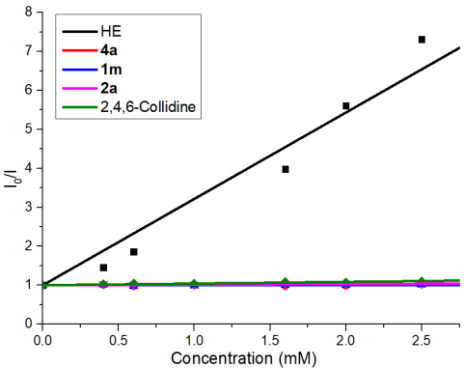


Figure S9. The lines of luminescence quenching experiments.

The Cyclic Voltammetry Experiments

Cyclic voltammetry was carried out in a gas-tight glass cell with CHI 660Epotentiostat in a nitrogen atmosphere. A glassy carbon disk electrode (diameter is 1.0 mm.PTFE shroud) was used as a working electrode. A platinum foil was used as a counter electrode.Ag/AgNO; electrode was used as a reference electrode, which was calibrated with Fc/Fc* redoxcouple, and a scan rate of 1 V/s. Samples were prepared with 0.05 mmol of substrate in 10.0 mL of 0.1 M tetrabutylammonium hexafluorophosphate in dry, degassed acetonitrile.

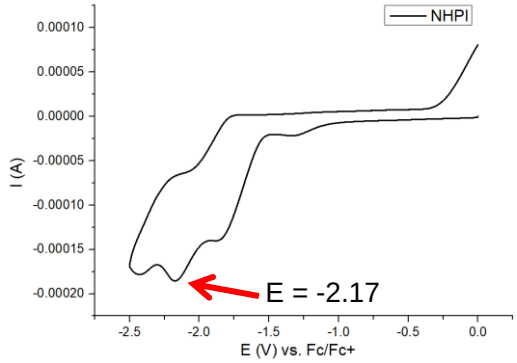


Figure S10. Cyclic voltammogram of NHPI (**4a**).

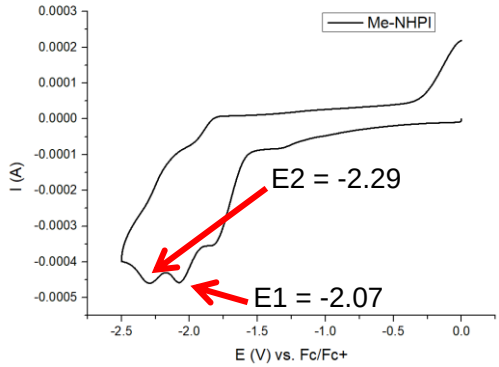
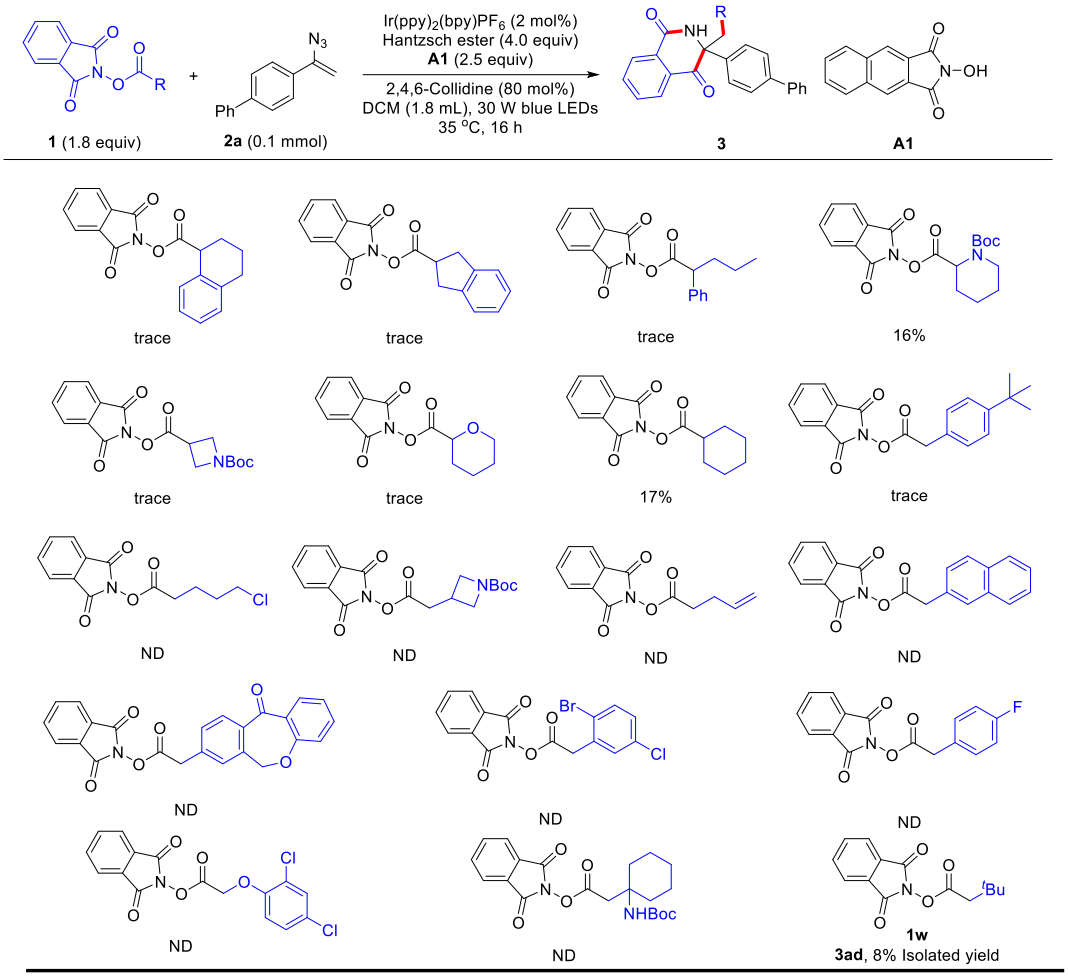


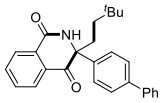
Figure S11. Cyclic voltammogram of Me-NHPI.

VII. Unsuccessful Substrates



Yield was determined by ^{1}H NMR of the crude mixture using mesitylene internal standard.

3-([1,1'-biphenyl]-4-yl)-3-(3,3-dimethylbutyl)-2,3-dihydroisoquinoline-1,4-dione (3ad)

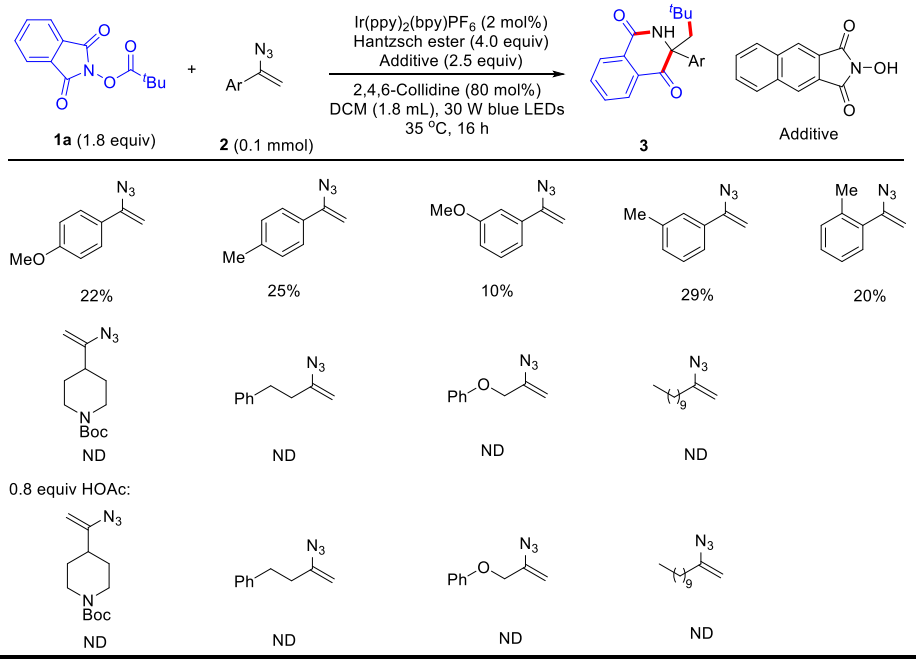


Following typical procedure A, the reaction of **1w** (47.0 mg, 0.18 mmol), **2a** (22.1 mg, 0.10 mmol) for 16 h afforded **3ad** as white solid (3.0 mg, 8% yield).

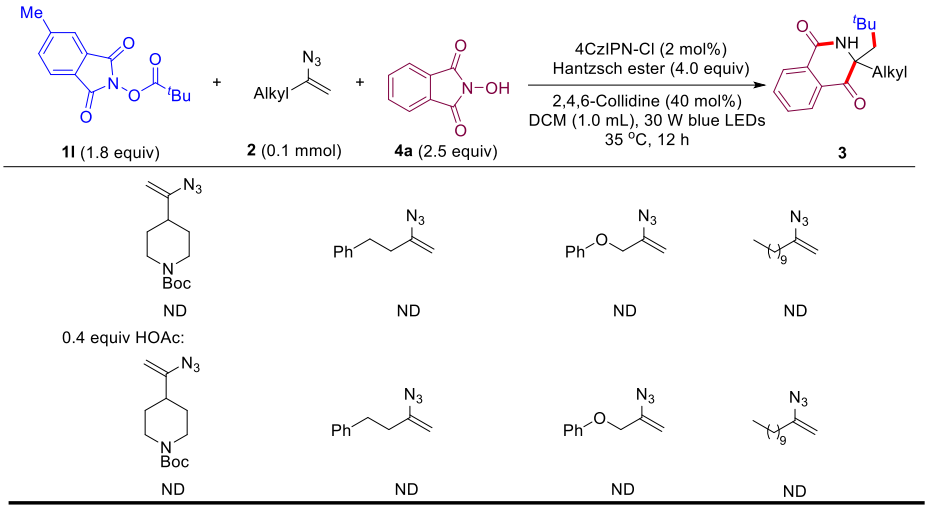
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 7.8 Hz, 1H), 8.00 (d, *J* = 7.5 Hz, 1H), 7.78 (t, *J* = 7.5 Hz, 1H), 7.68 (t, *J* = 7.3 Hz, 1H), 7.60 – 7.50 (m, 6H), 7.41 (t, *J* = 7.2 Hz, 2H), 7.36 – 7.31 (m, 1H), 6.54 (brs, 1H), 2.63 (tt, *J* = 13.3, 3.2 Hz, 1H), 2.07 (tt, *J* = 13.3, 3.2 Hz, 1H), 1.34 – 1.27 (m, 2H), 0.91 (s, 9H).

^{13}C NMR (151 MHz, Chloroform-*d*) δ 193.1, 163.4, 141.3, 140.2, 139.4, 135.1, 133.4, 131.3, 131.2, 129.0, 128.5, 127.8, 127.20, 127.18, 126.3, 70.8, 37.8, 35.8, 30.4, 29.4.

HR-MS (ESI-TOF) calcd for $C_{27}H_{28}NO_2^+$ $[M+H]^+$ 398.2115; found 398.2113.



Yield was determined by ^{1}H NMR of the crude mixture using mesitylene internal standard.



Yield was determined by ^{1}H NMR of the crude mixture using mesitylene internal standard.

VIII. X-Ray Diffraction Data

X-Ray Diffraction Data of 3a (CCDC 2361327)

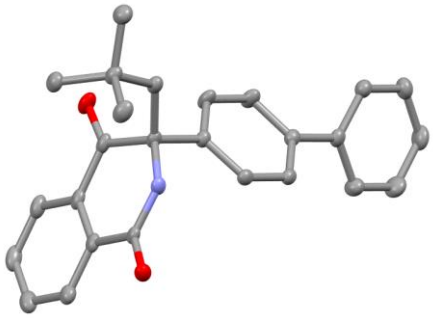


Table S13. Crystal data and structure refinement for **3a** (dhw512602_0m)

Identification code	dhw512602_0m
Empirical formula	$C_{26}H_{25}NO_2$
Formula weight	383.47
Temperature/K	100.0(2)
Crystal system	triclinic
Space group	P-1
a/Å	13.8148(7)
b/Å	17.2854(8)
c/Å	19.6680(9)
α/°	79.694(2)
β/°	74.868(2)
γ/°	68.162(2)
Volume/Å^3	4190.8(4)
Z	8
ρ_{calc}g/cm^3	1.216
μ/mm^{-1}	0.598
F(000)	1632.0
Crystal size/mm^3	0.03 × 0.02 × 0.02
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	4.674 to 144.762
Index ranges	-17 ≤ h ≤ 17, -21 ≤ k ≤ 21, -24 ≤ l ≤ 22
Reflections collected	79252
Independent reflections	16346 [R_{int} = 0.0603, R_{sigma} = 0.0467]
Data/restraints/parameters	16346/0/1057

Goodness-of-fit on F^2	1.047
Final R indexes [I>=2σ (I)]	R_1 = 0.0581, wR_2 = 0.1555
Final R indexes [all data]	R_1 = 0.0764, wR_2 = 0.1726
Largest diff. peak/hole / e $Å^{-3}$	1.17/-0.50

X-Ray Diffraction Data of 3h (CCDC 2361326)

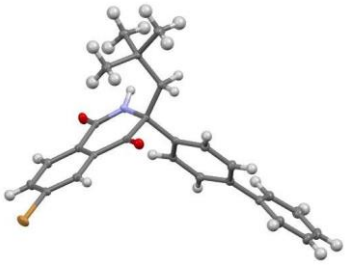


Table S14. Crystal data and structure refinement for **3h** (DHW202403215Q_0m)

Identification code	DHW202403215Q_0m
Empirical formula	$C_{26}H_{24}BrNO_2$
Formula weight	462.37
Temperature/K	100.0
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	12.1999(2)
b/Å	8.1197(2)
c/Å	22.7110(5)
α/°	90
β/°	103.9270(10)
γ/°	90
Volume/$Å^3$	2183.61(8)
Z	4
$\rho_{calc}g/cm^3$	1.406
μ/mm^{-1}	2.736
F(000)	952.0
Crystal size/mm^3	0.03 × 0.02 × 0.02
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	7.466 to 145.394
Index ranges	$-15 \le h \le 15$, $-8 \le k \le 9$, $-27 \le l \le 28$

Reflections collected	27244
Independent reflections	4281 [R_{int} = 0.0209, R_{sigma} = 0.0131]
Data/restraints/parameters	4281/0/274
Goodness-of-fit on F^2	1.070
Final R indexes [I>=2σ (I)]	R_1 = 0.0231, wR_2 = 0.0601
Final R indexes [all data]	R_1 = 0.0235, wR_2 = 0.0603
Largest diff. peak/hole / e $Å^{-3}$	0.37/-0.47

IX. References

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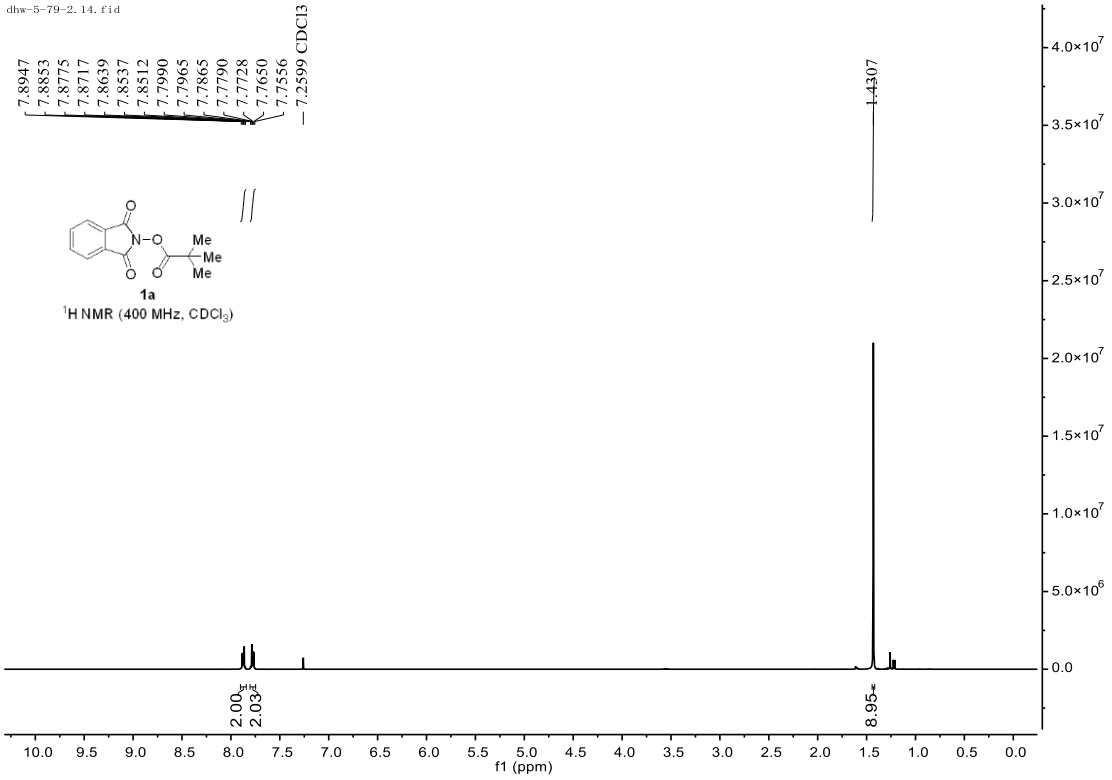
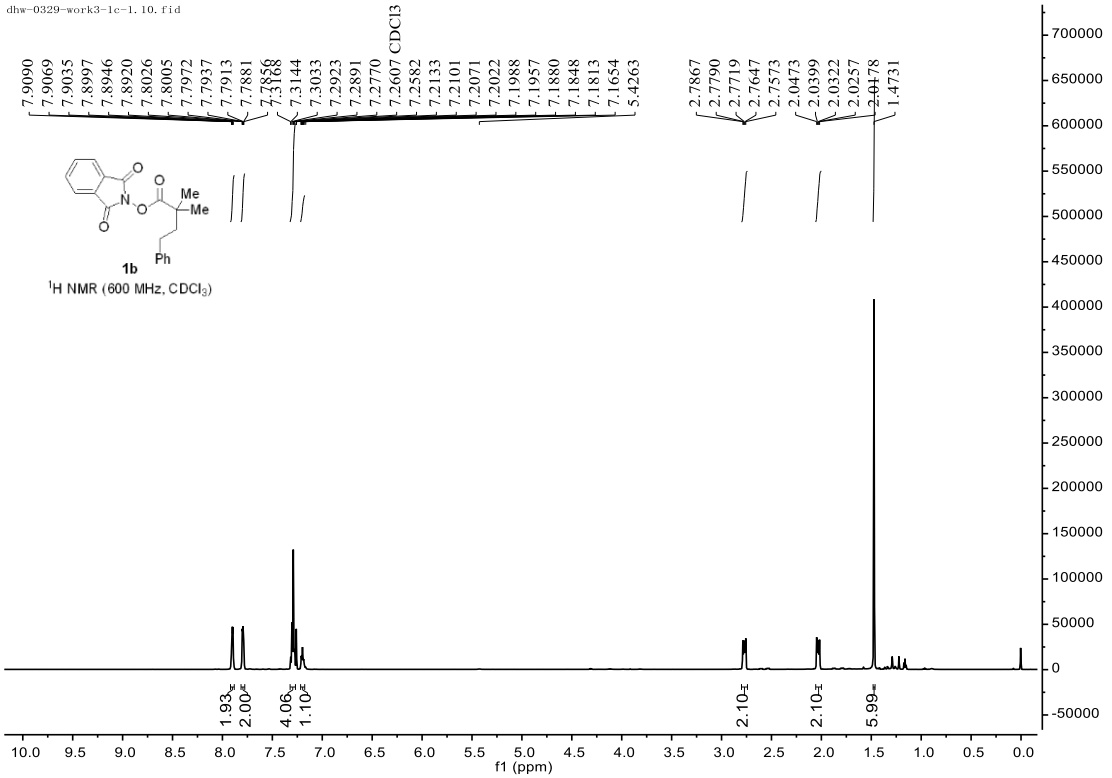
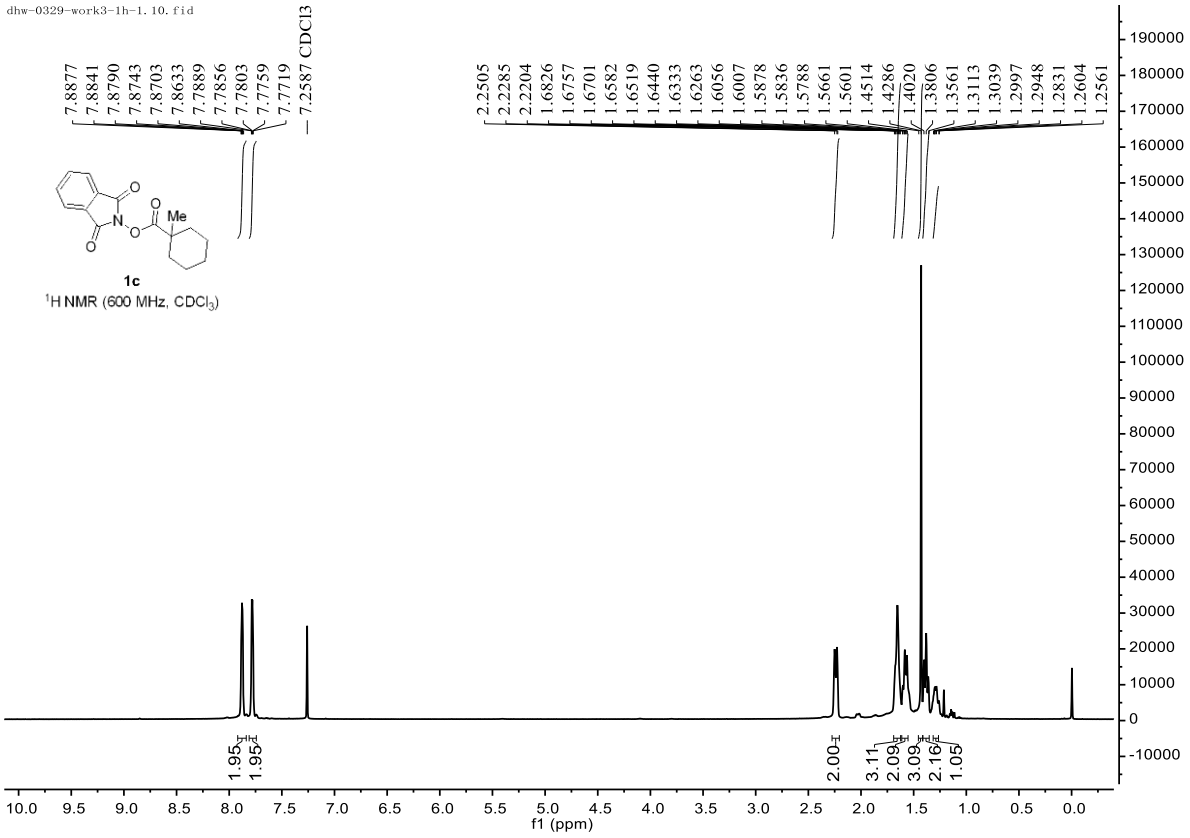
X. ^{1}H、^{13}C and ^{19}F Spectra of Compounds



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7.8743
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7.7856
7.7803
7.7759
7.7719
7.2587 CDCl3
2.2505
2.2285
2.2204
1.6826
1.6757
1.6701
1.6582
1.6519
1.6440
1.6333
1.6263
1.6056
1.6007
1.5878
1.5836
1.5788
1.5661
1.5601
1.4514
1.4286
1.4020
1.3806
1.3561
1.3113
1.3039
1.2997
1.2948
1.2831
1.2604
1.2561
Me
1c
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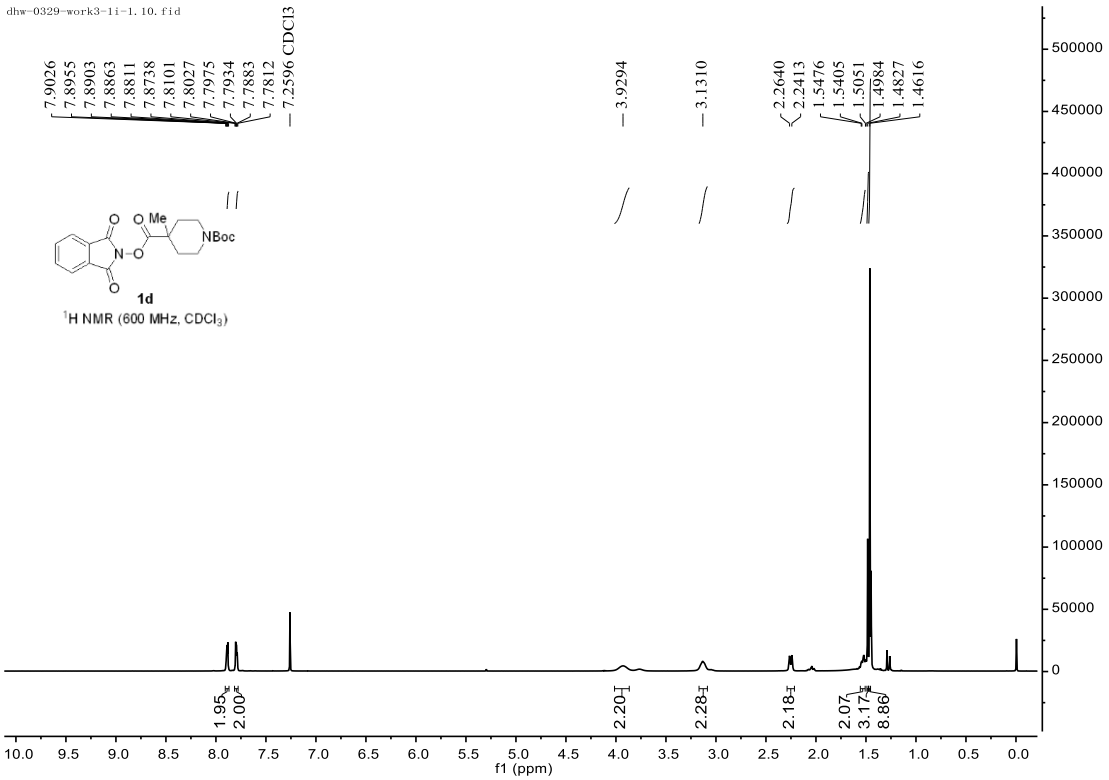
1.95
2.00
3.11
2.09
3.09
2.16
1.05
f1 (ppm)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7.7934
7.7883
7.7812
7.2596 CDCl3
3.9294
3.1310
2.2640
2.2413
1.5476
1.5405
1.5051
1.4984
1.4827
1.4616
Me
NBoc
1d
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8.86
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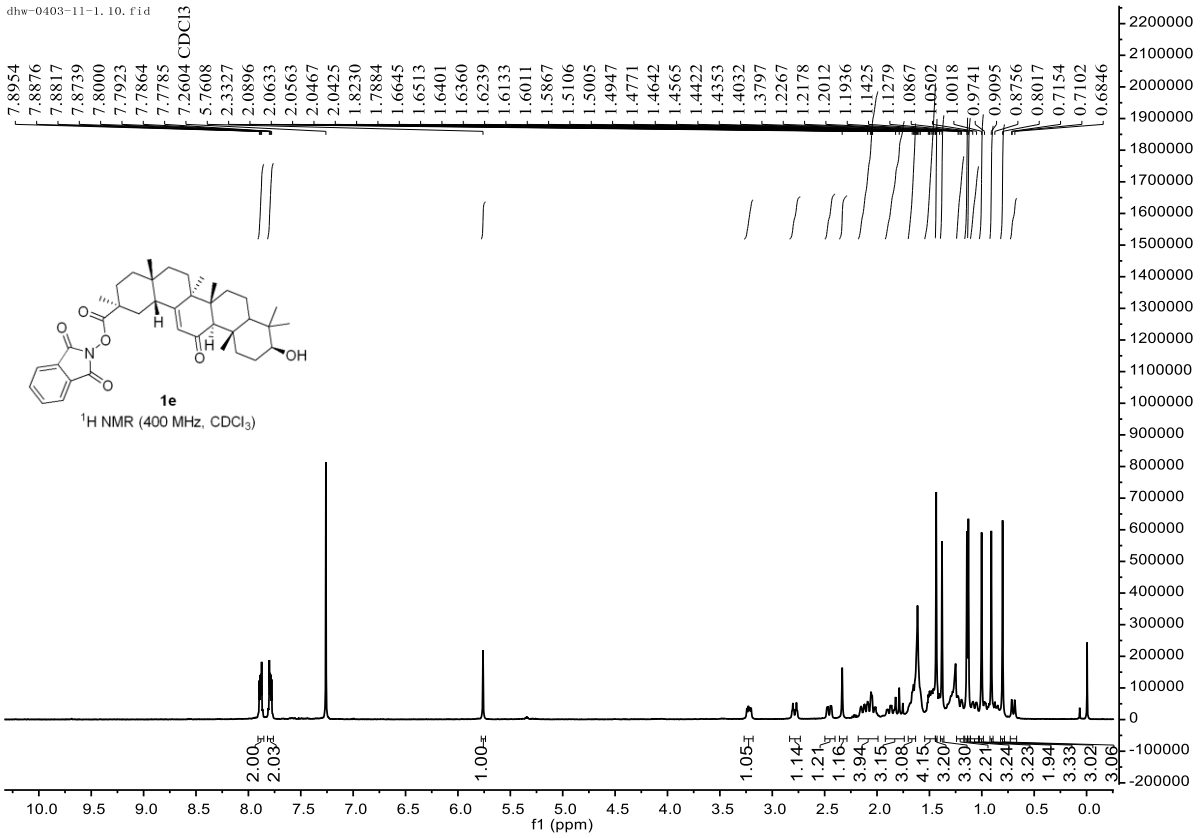
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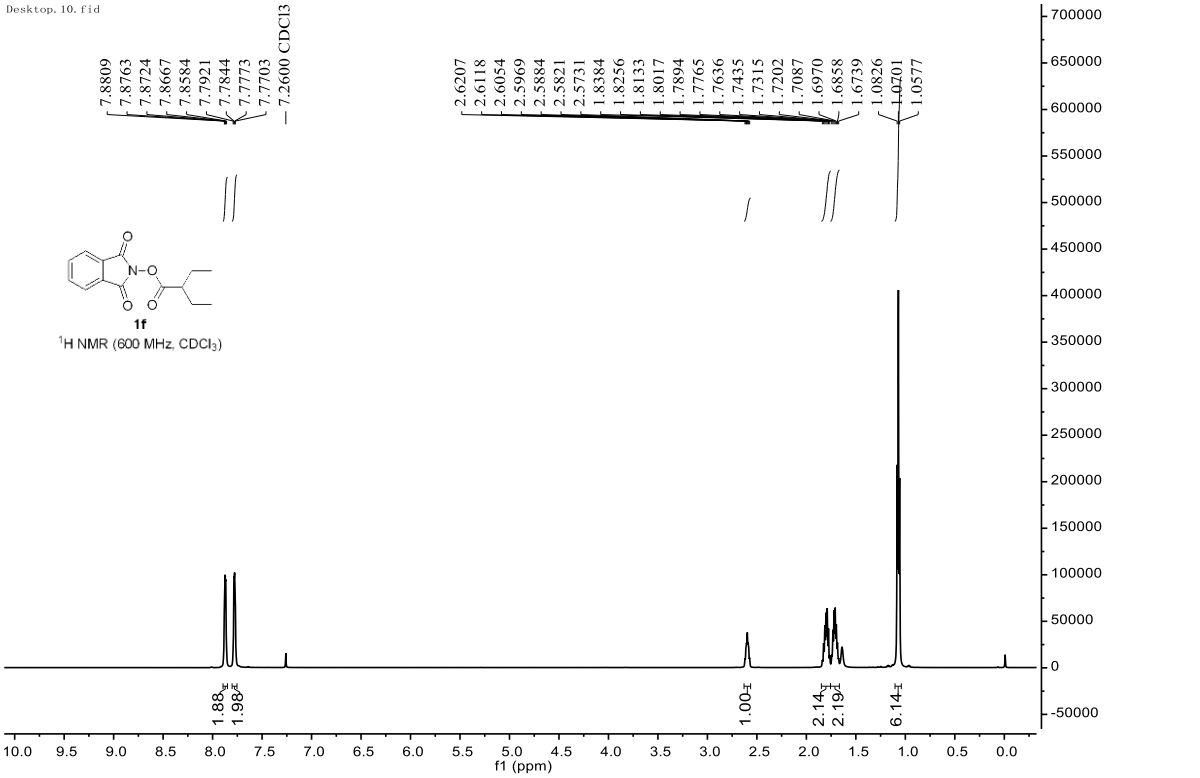
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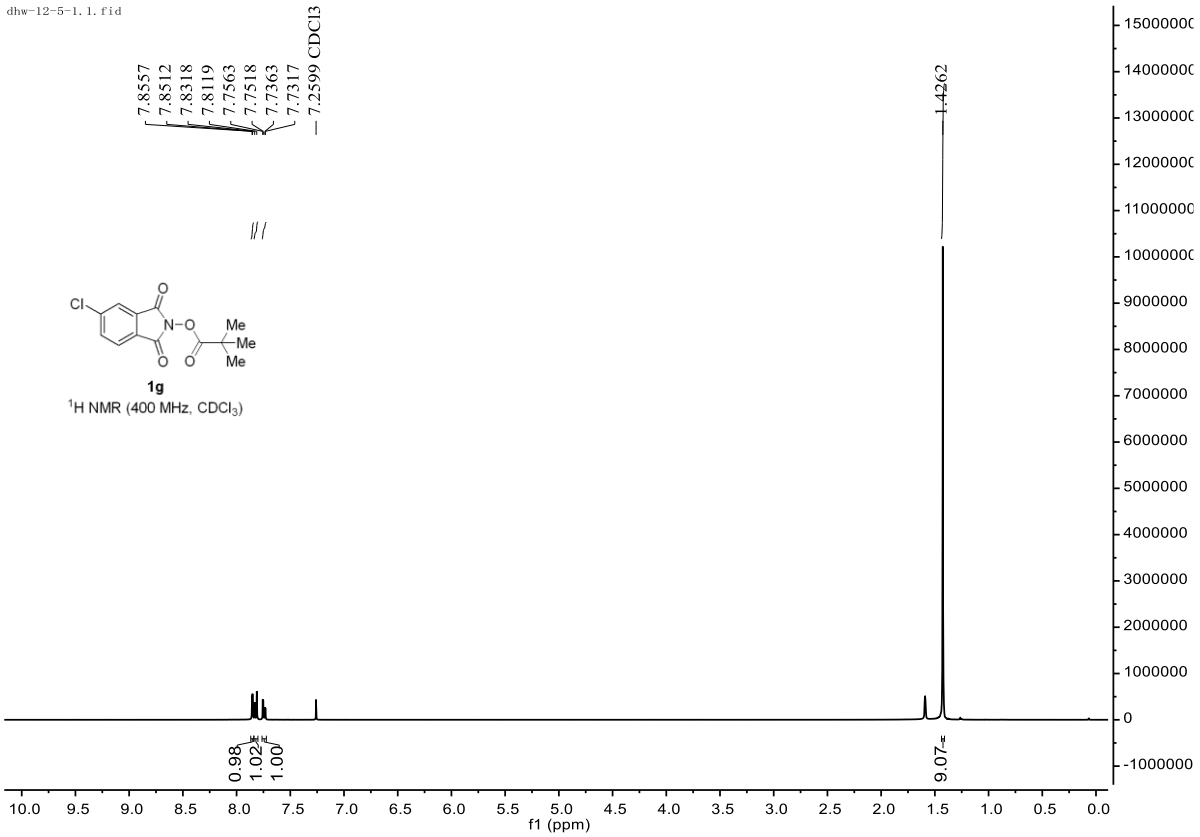
1f
1H NMR (600 MHz, CDCl3)
f1 (ppm)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7.8119
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7.7317
7.2599 CDCl3
1.4262
Cl
O
N–O
O
Me
Me
Me
1g
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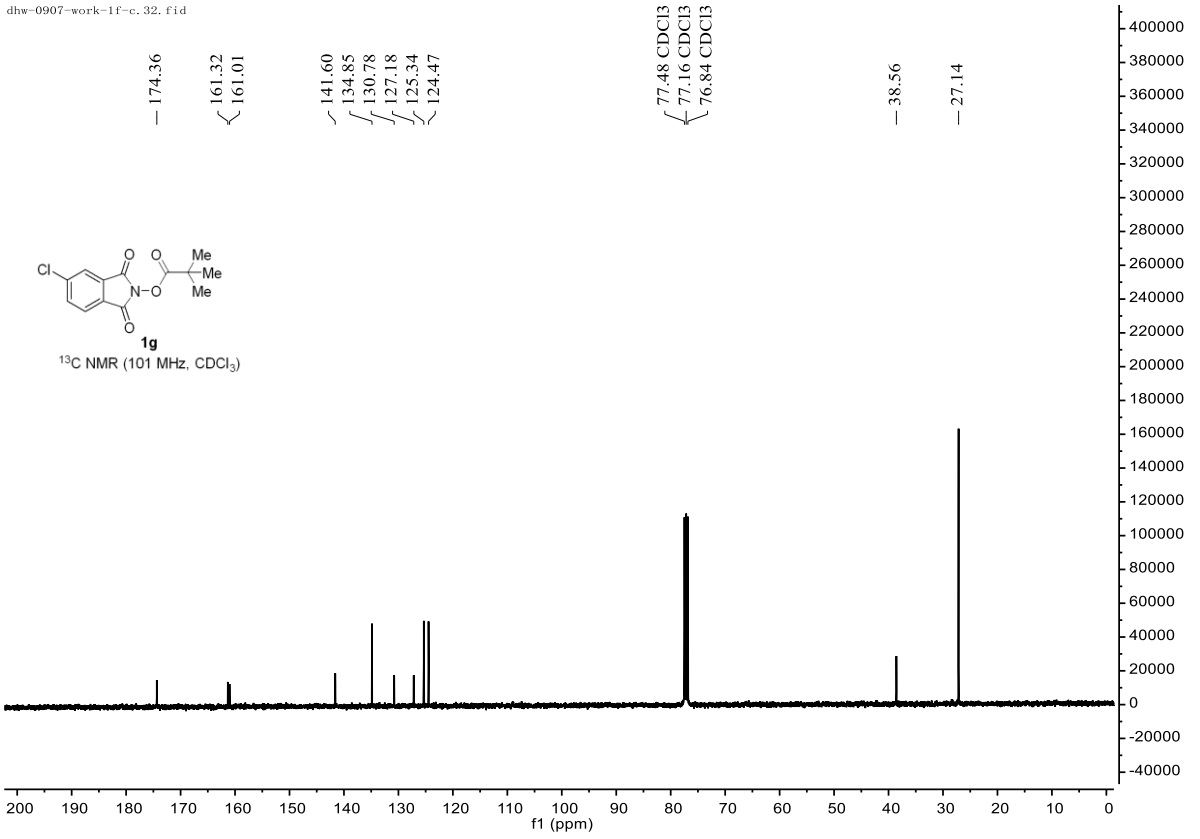
1.02
1.00
9.07
f1 (ppm)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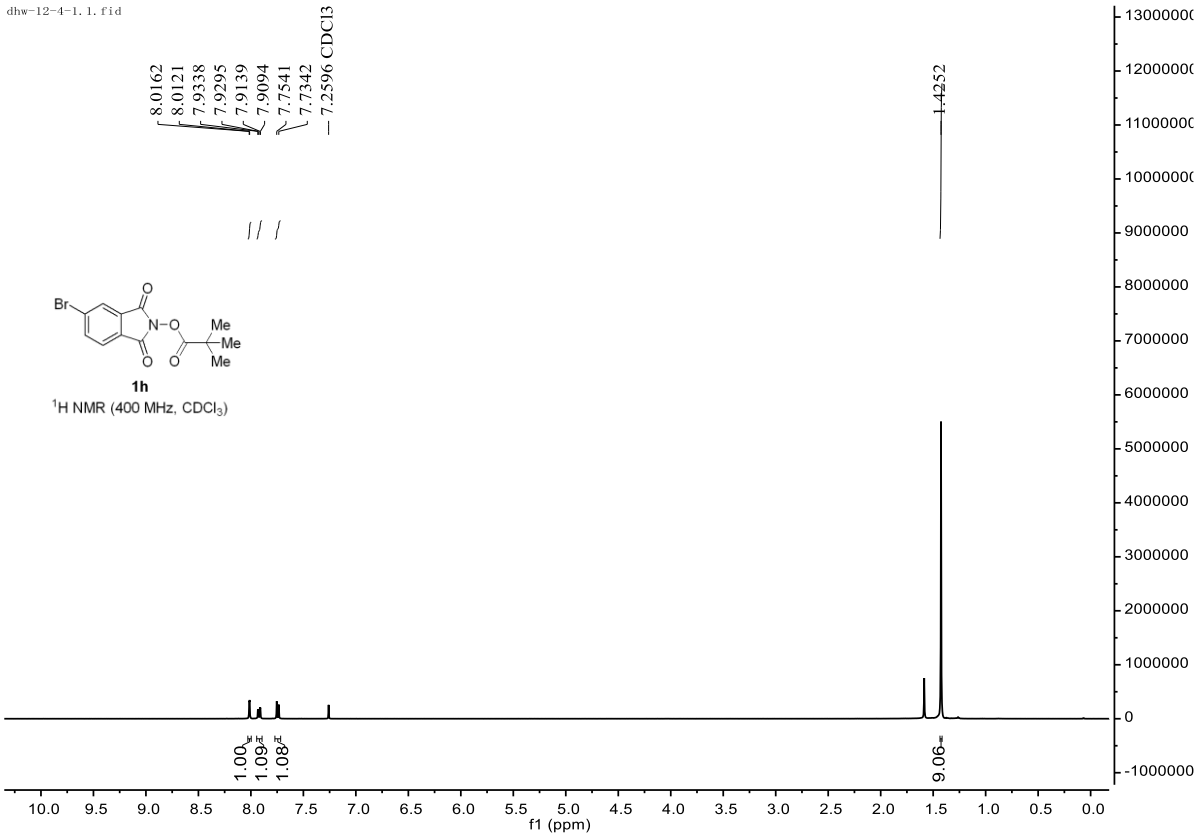
124.47
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
38.56
27.14
Cl
O
N–O
O
Me
Me
Me
1g
13C NMR (101 MHz, CDCl3)
f1 (ppm)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7.7342
7.2596 CDCl3
1.4252
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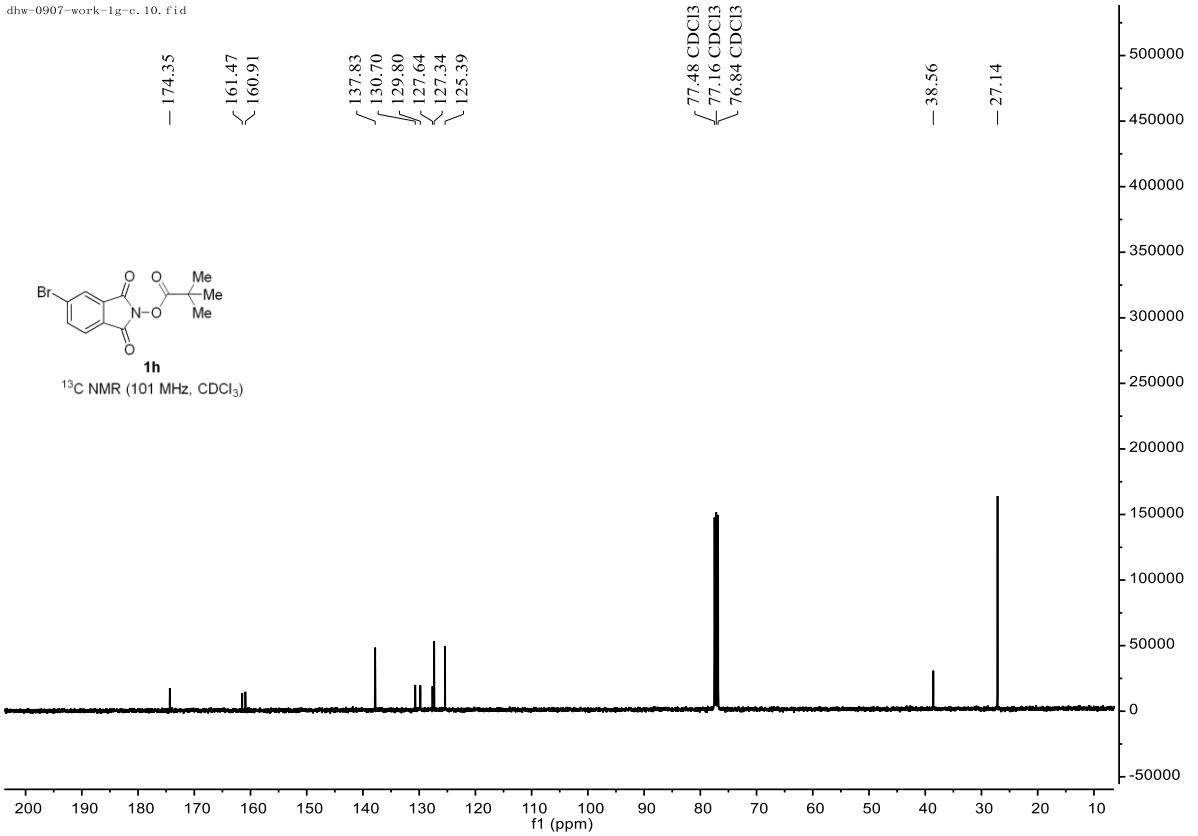
N-O
Me
Me
Me
1h
1H NMR (400 MHz, CDCl3)
1.00
1.09
1.08
9.06
f1 (ppm)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77.48 CDCl3
77.16 CDCl3
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38.56
27.14
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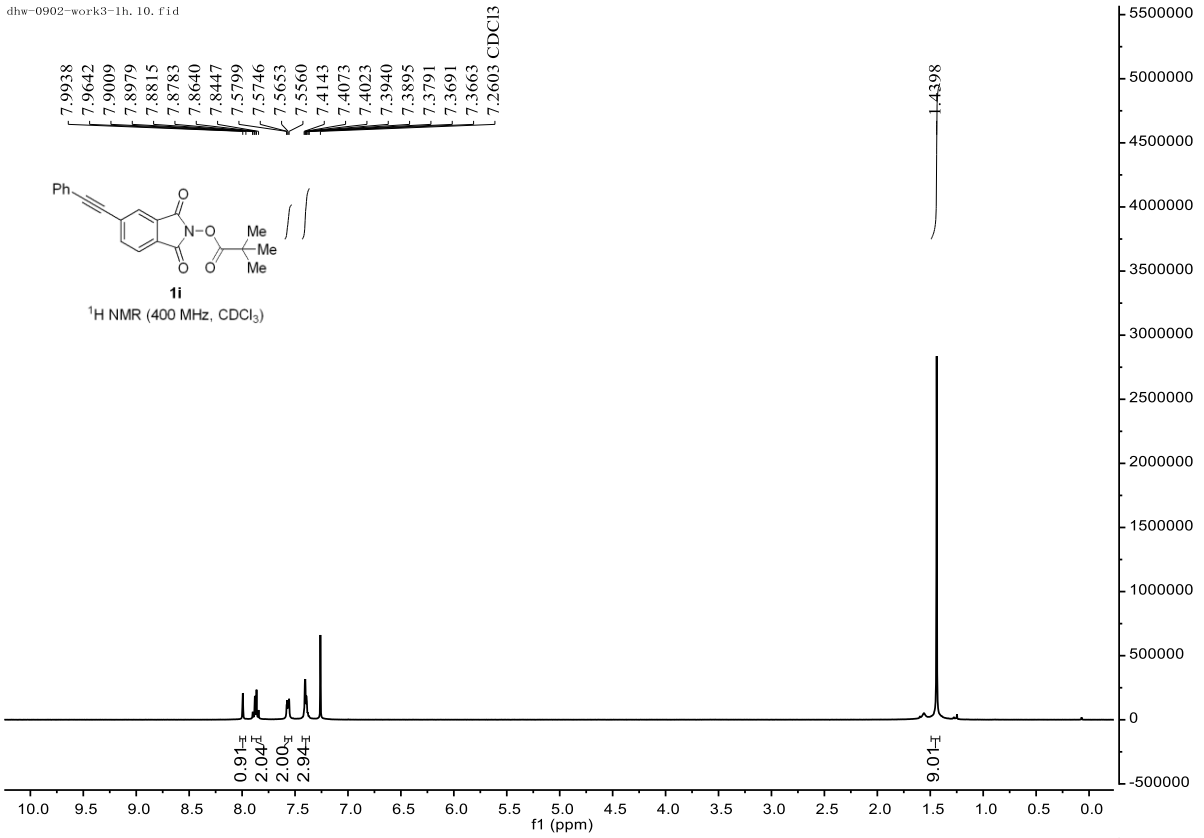
N-O
Me
Me
Me
1h
13C NMR (101 MHz, CDCl3)
f1 (ppm)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7.3691
7.3663
7.2603 CDCl3
1.4398
Ph
O
N-O
Me
Me
Me
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1i
1H NMR (400 MHz, CDCl3)
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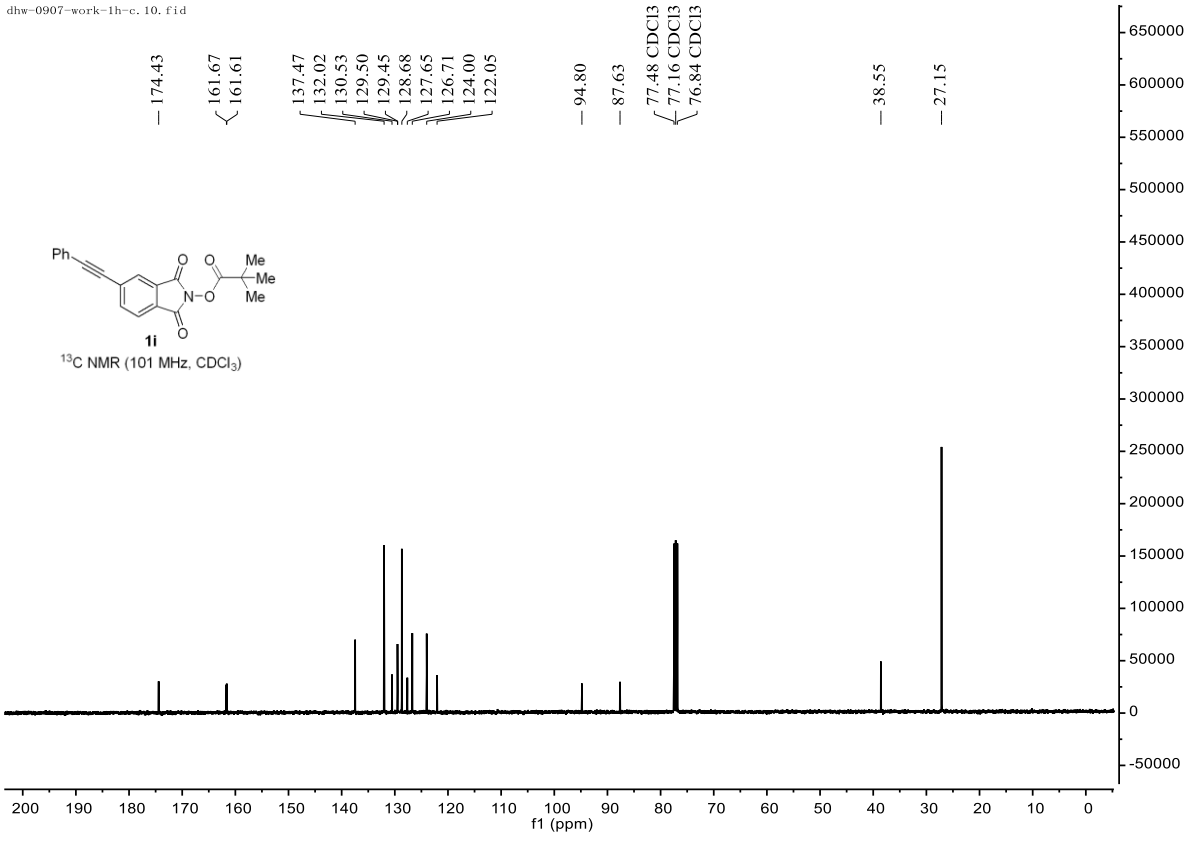
2.00
2.94
9.01
f1 (ppm)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122.05
94.80
87.63
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
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Ph
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Me
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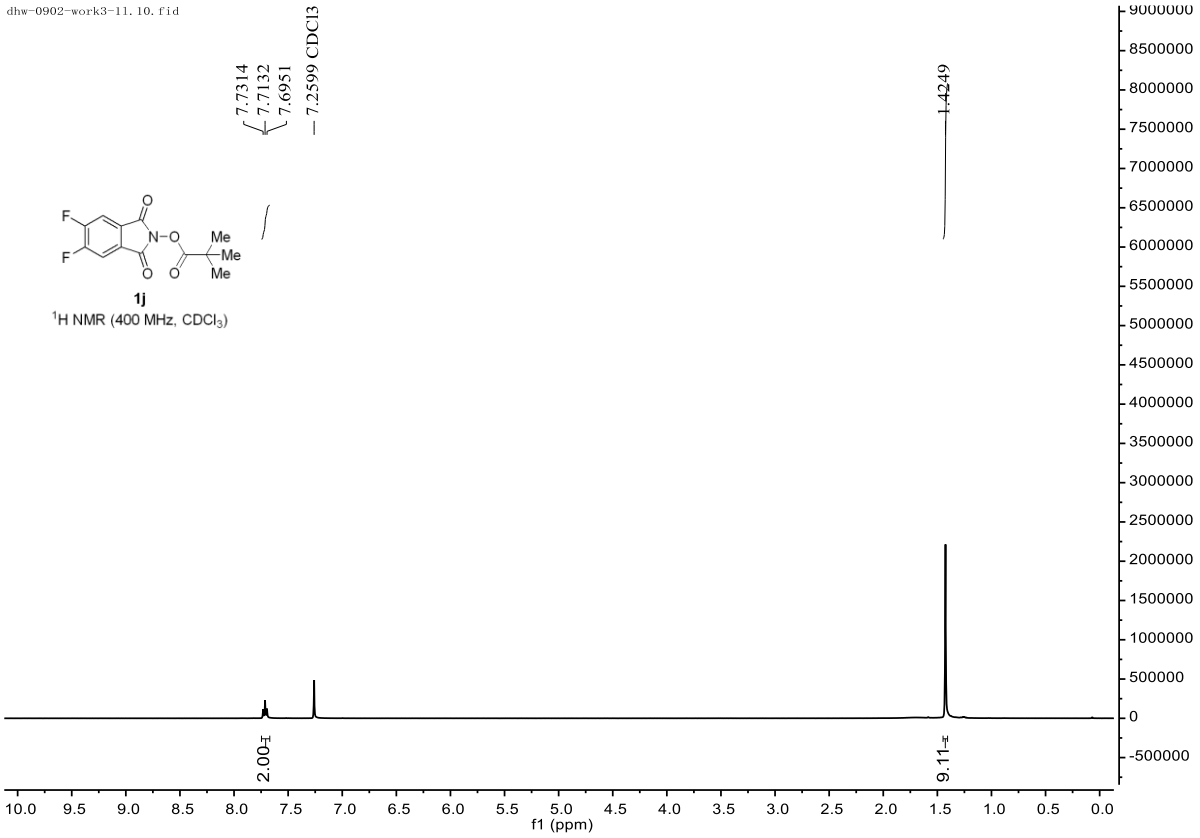
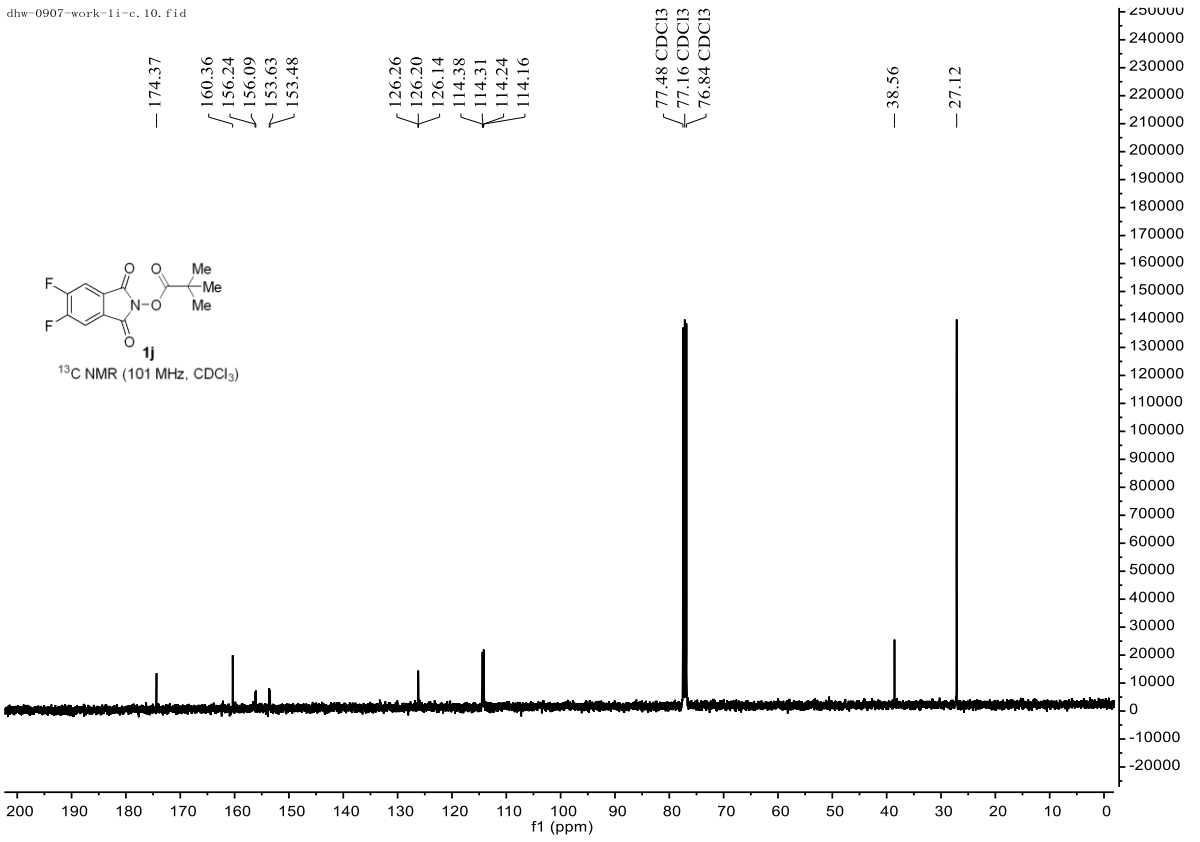
O
1i
13C NMR (101 MHz, CDCl3)
f1 (ppm)



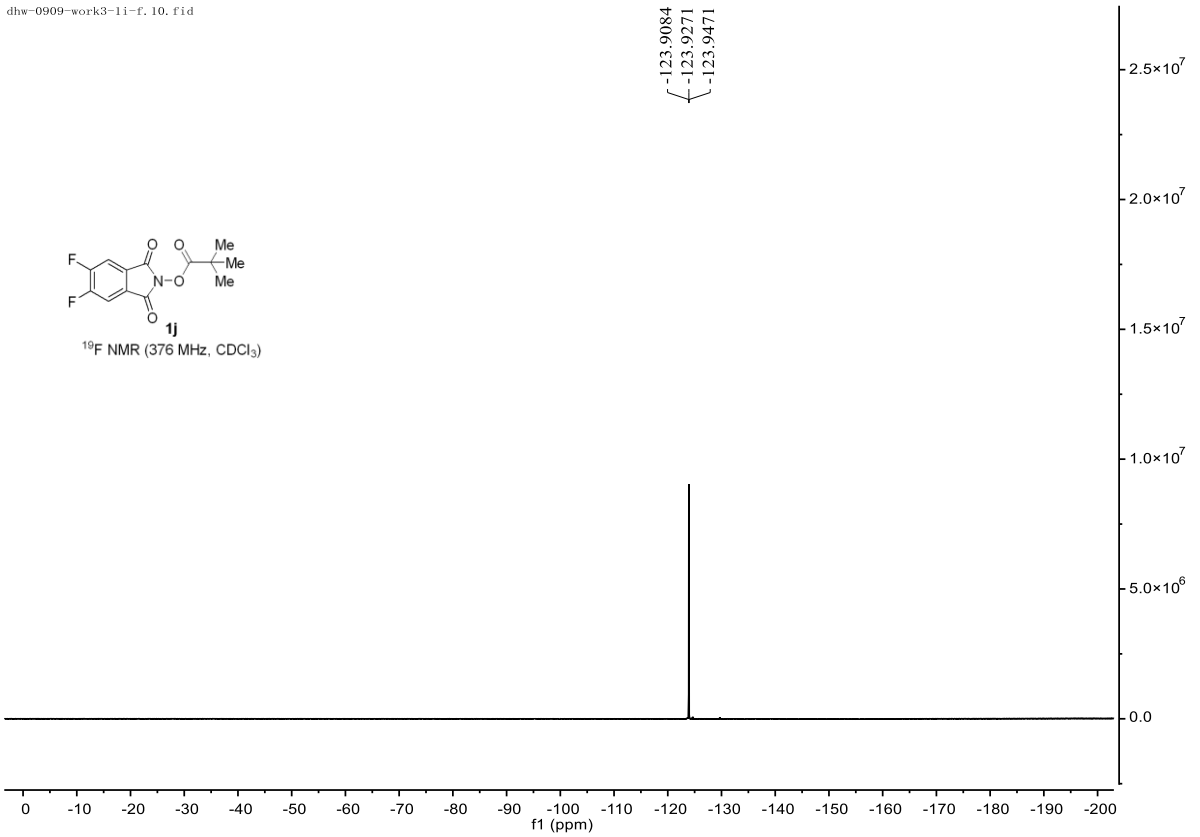
1j

^{1}H NMR (400 MHz, $CDCl_3$)



1j

^{13}C NMR (101 MHz, $CDCl_3$)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-123.9271
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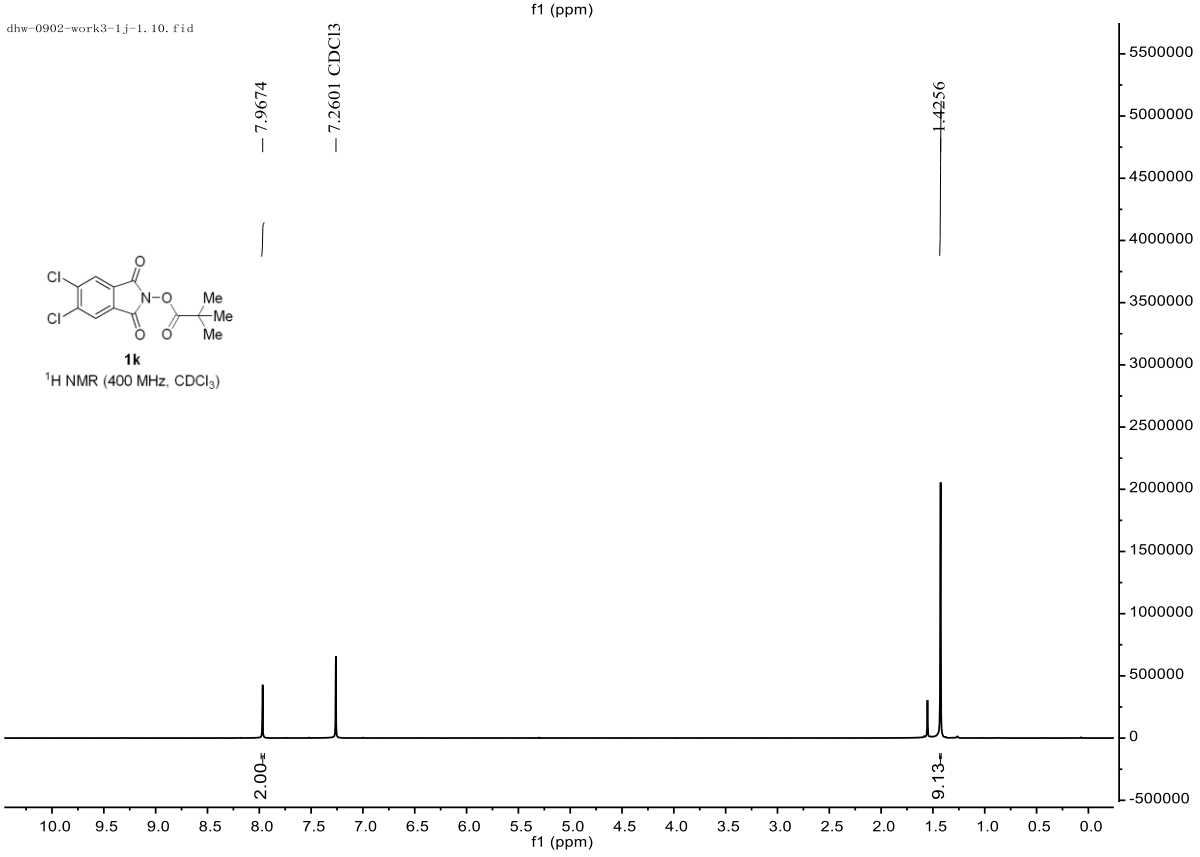
F
O
O
N
O
O
Me
Me
Me
O
1j
19F NMR (376 MHz, CDCl3)
2.5×10^7
2.0×10^7
1.5×10^7
1.0×10^7
5.0×10^6
0.0
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)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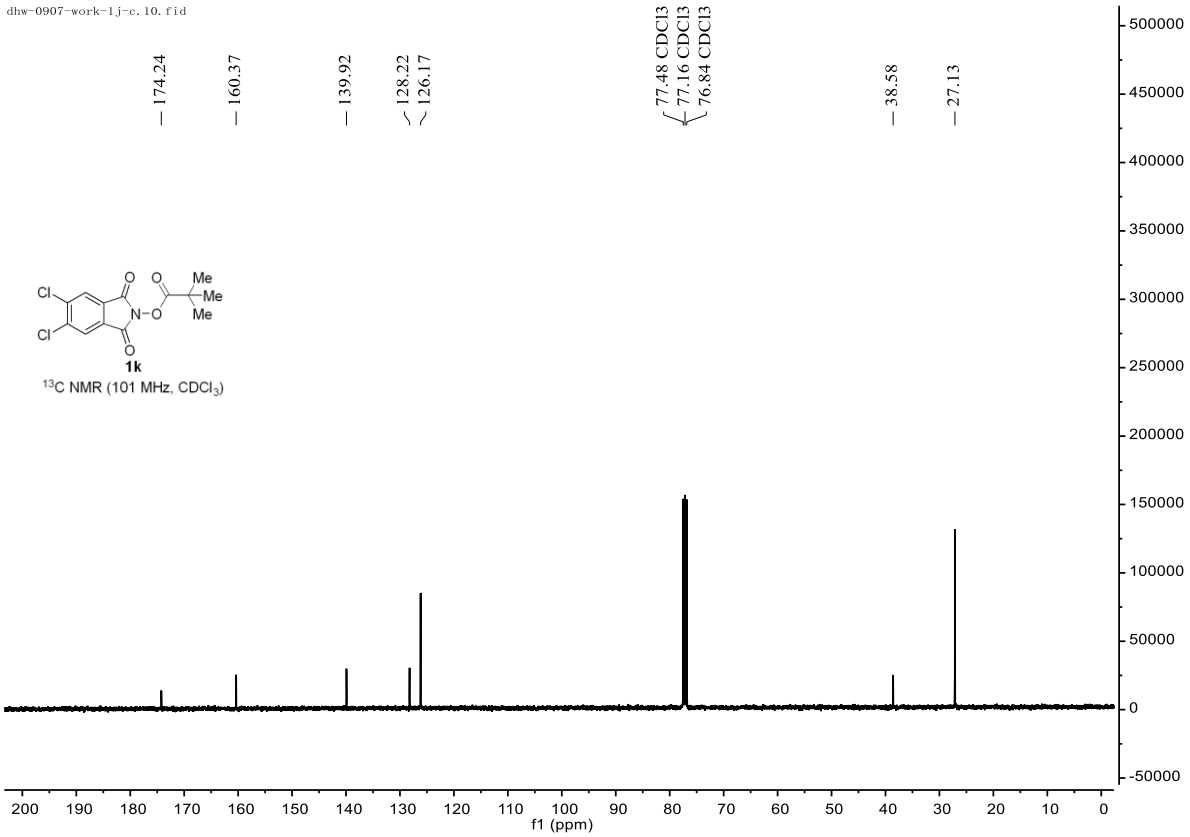
7.2601 CDCl3
1.4256
Cl
Cl
O
O
N
O
O
Me
Me
Me
O
1k
1H NMR (400 MHz, CDCl3)
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
2.00
9.13
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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126.17
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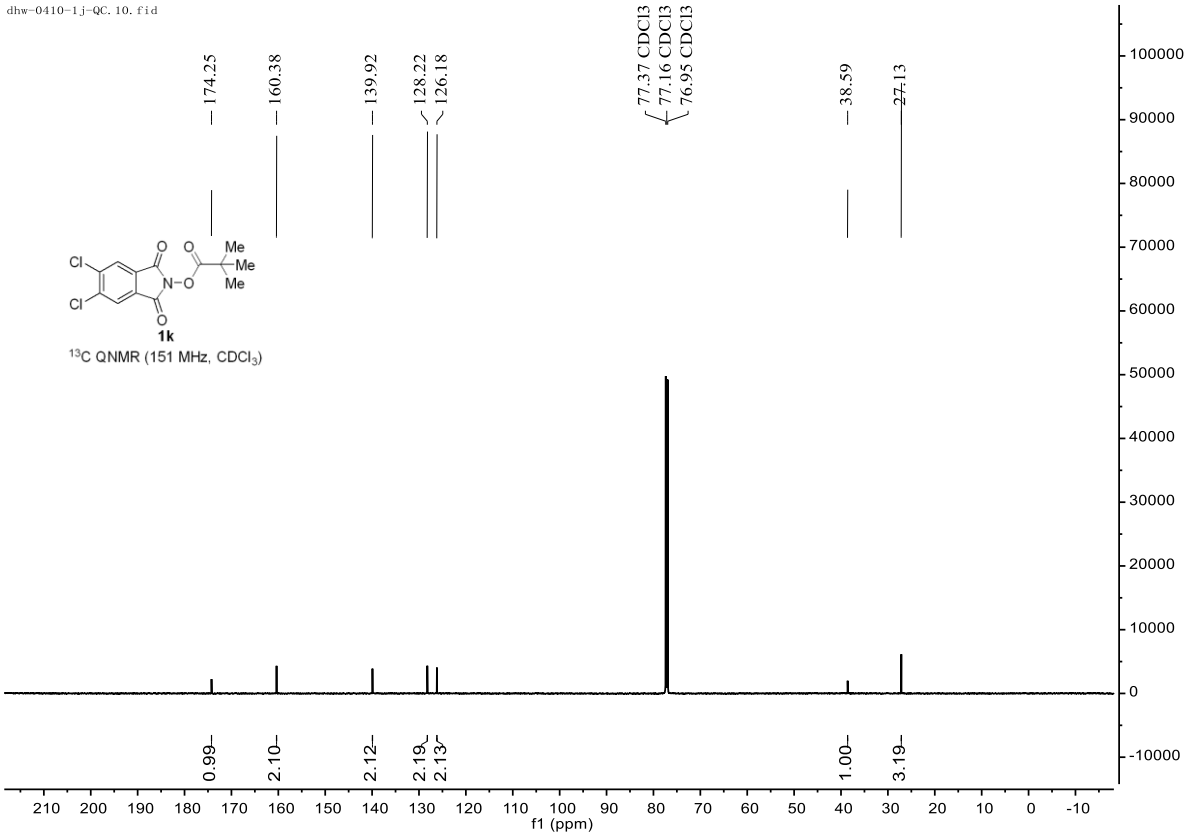
77.16 CDCl3
76.84 CDCl3
38.58
27.13
Cl
Cl
O
O
N
O
O
Me
Me
Me
1k
13C NMR (101 MHz, CDCl3)
f1 (ppm)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128.22
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76.95 CDCl3
38.59
27.13
0.99
2.10
2.12
2.19
2.13
1.00
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Me
Me
1k
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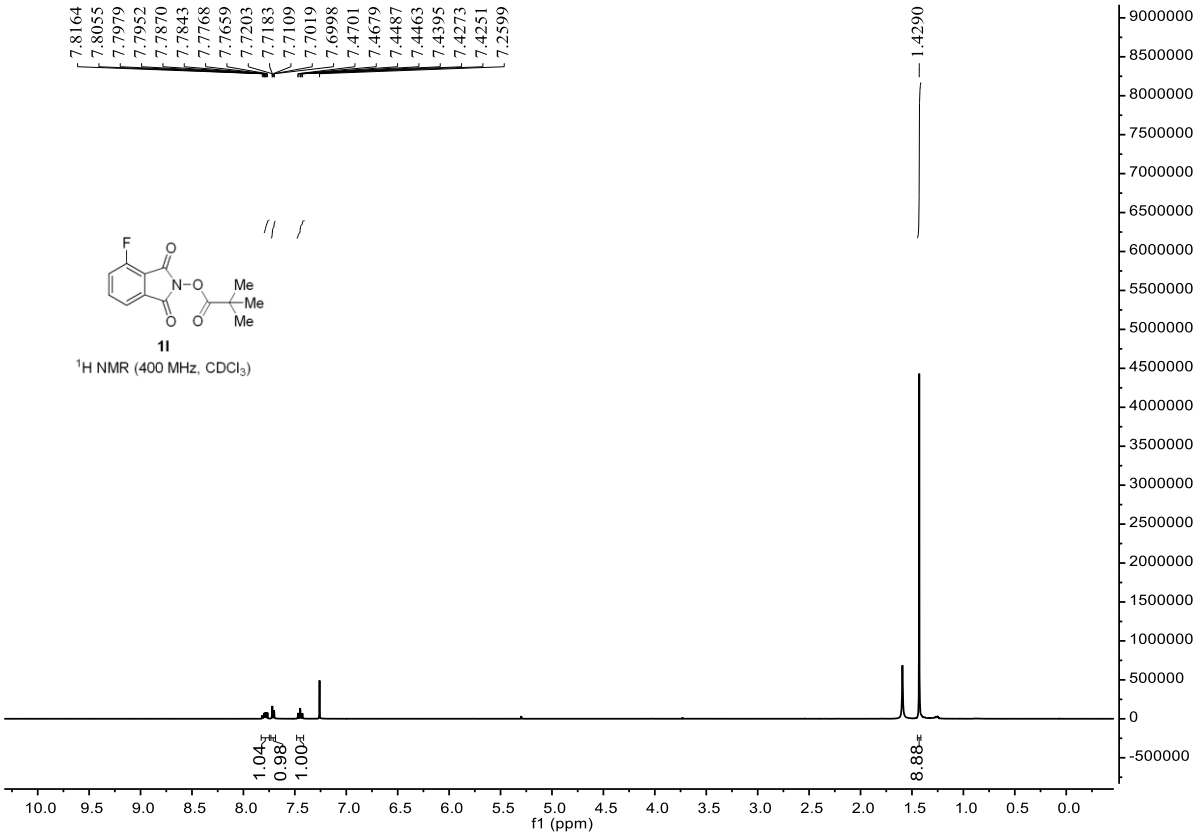
f1 (ppm)



1l

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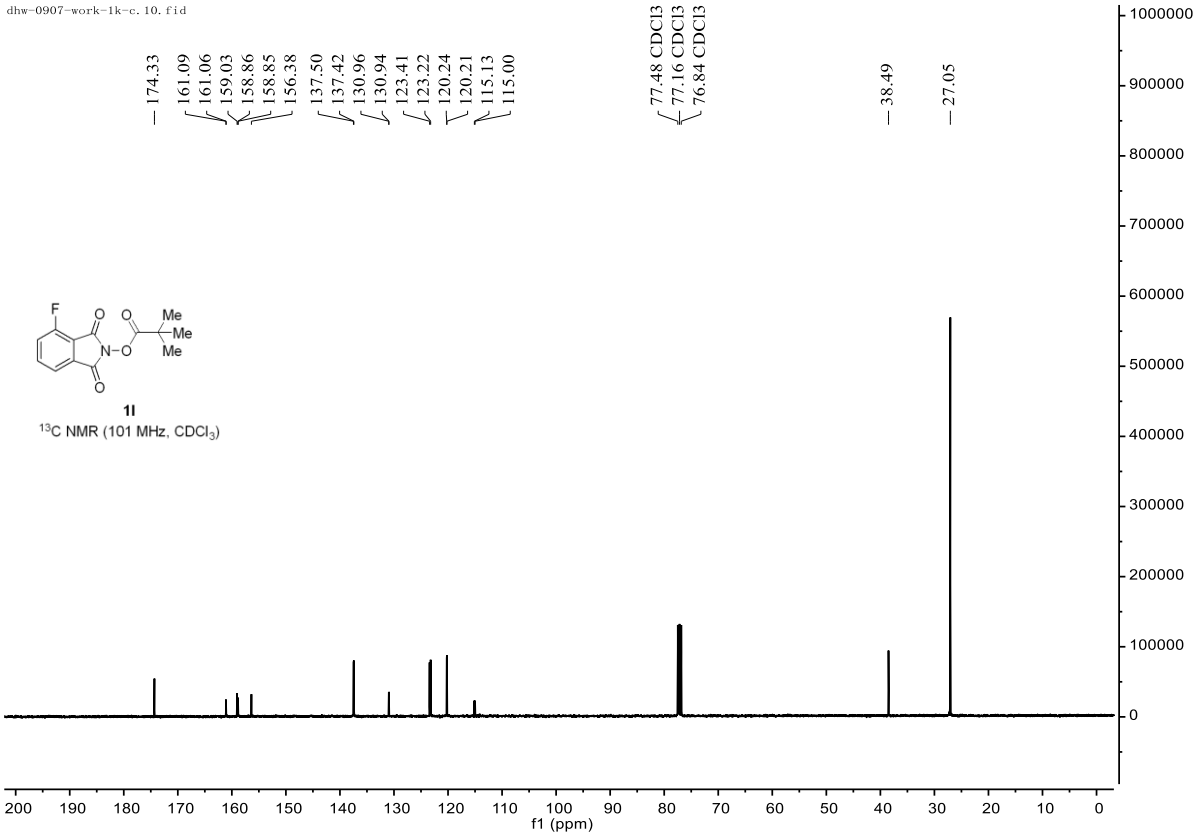

1l

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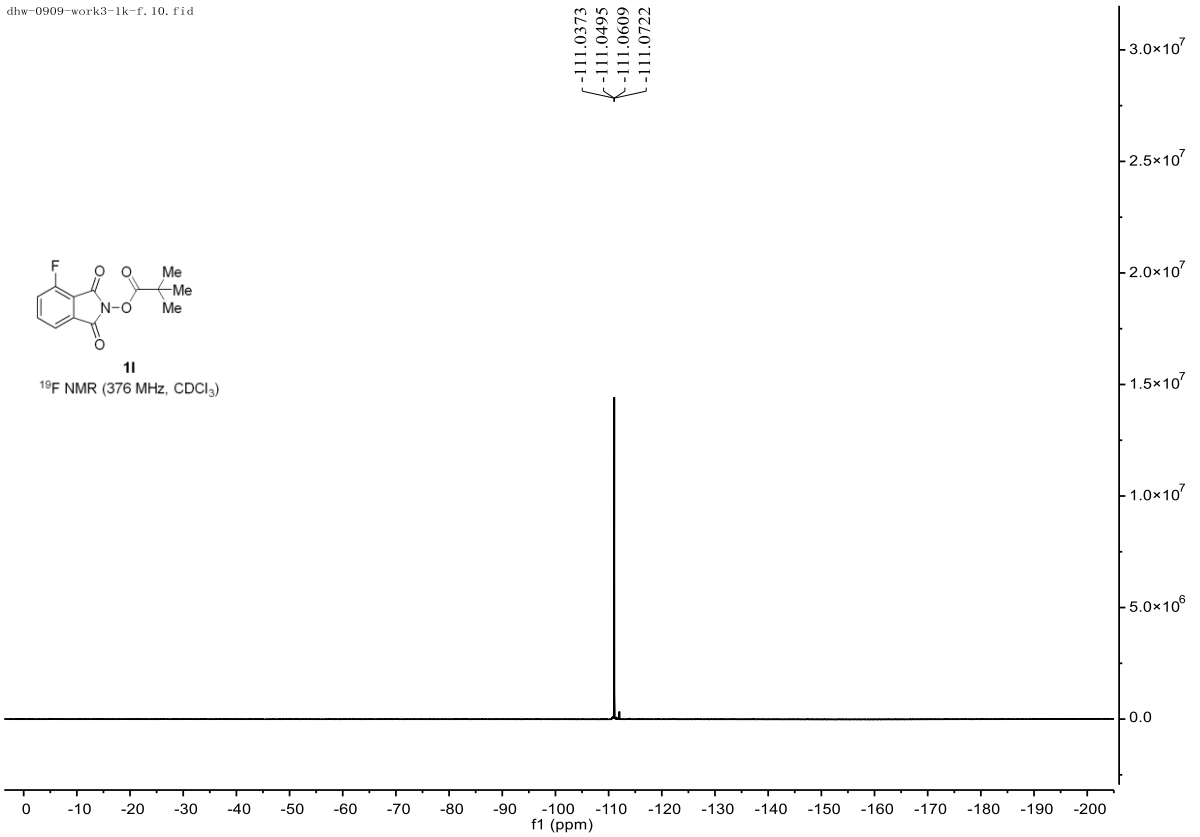
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Me
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O
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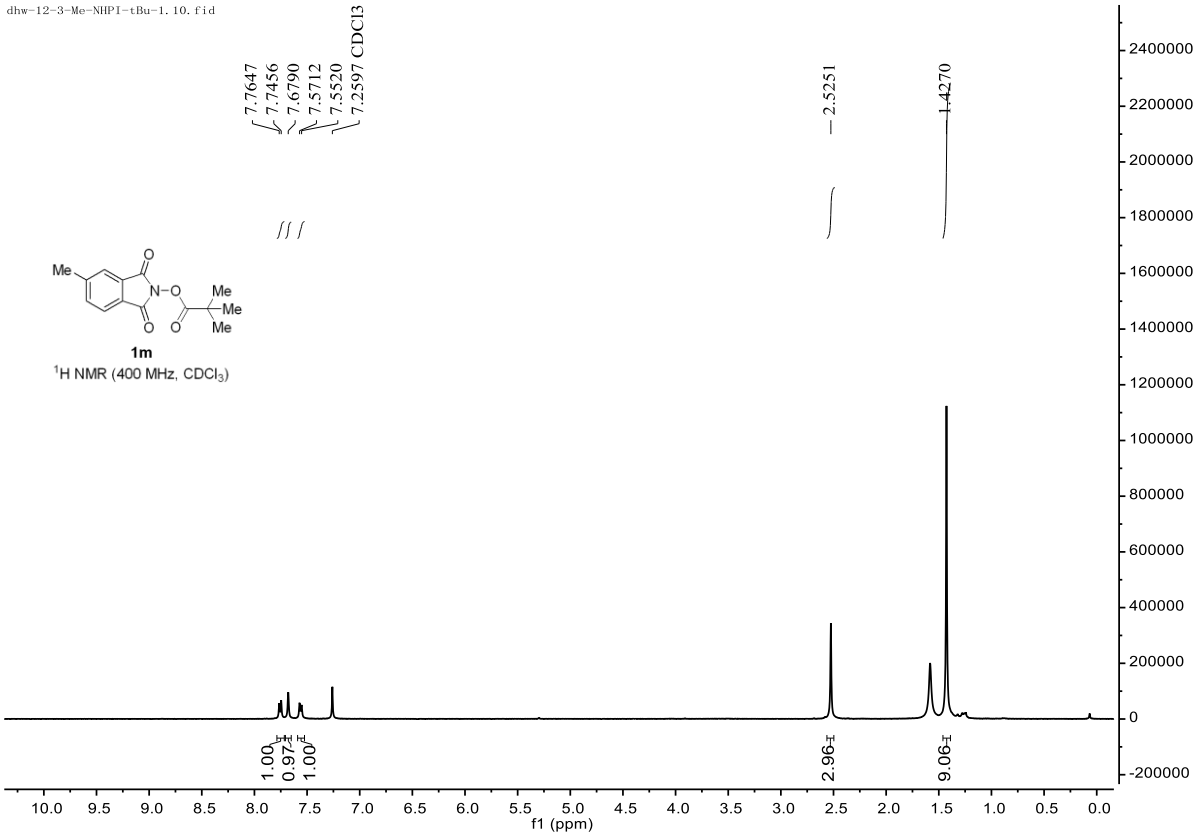
1l
19F NMR (376 MHz, CDCl3)
f1 (ppm)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1.4270
Me
O
N
O
Me
Me
Me
O
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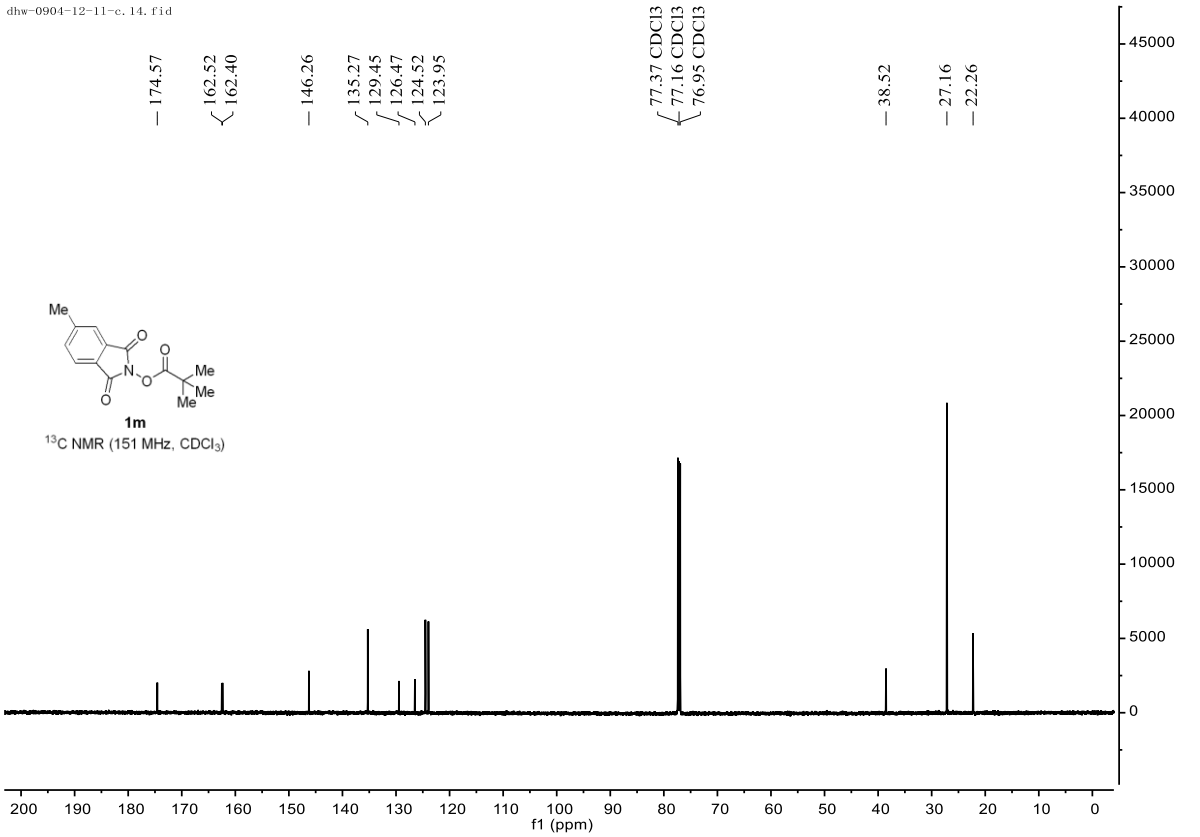
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123.95
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77.16 CDCl3
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38.52
27.16
22.26
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Me
Me
1m
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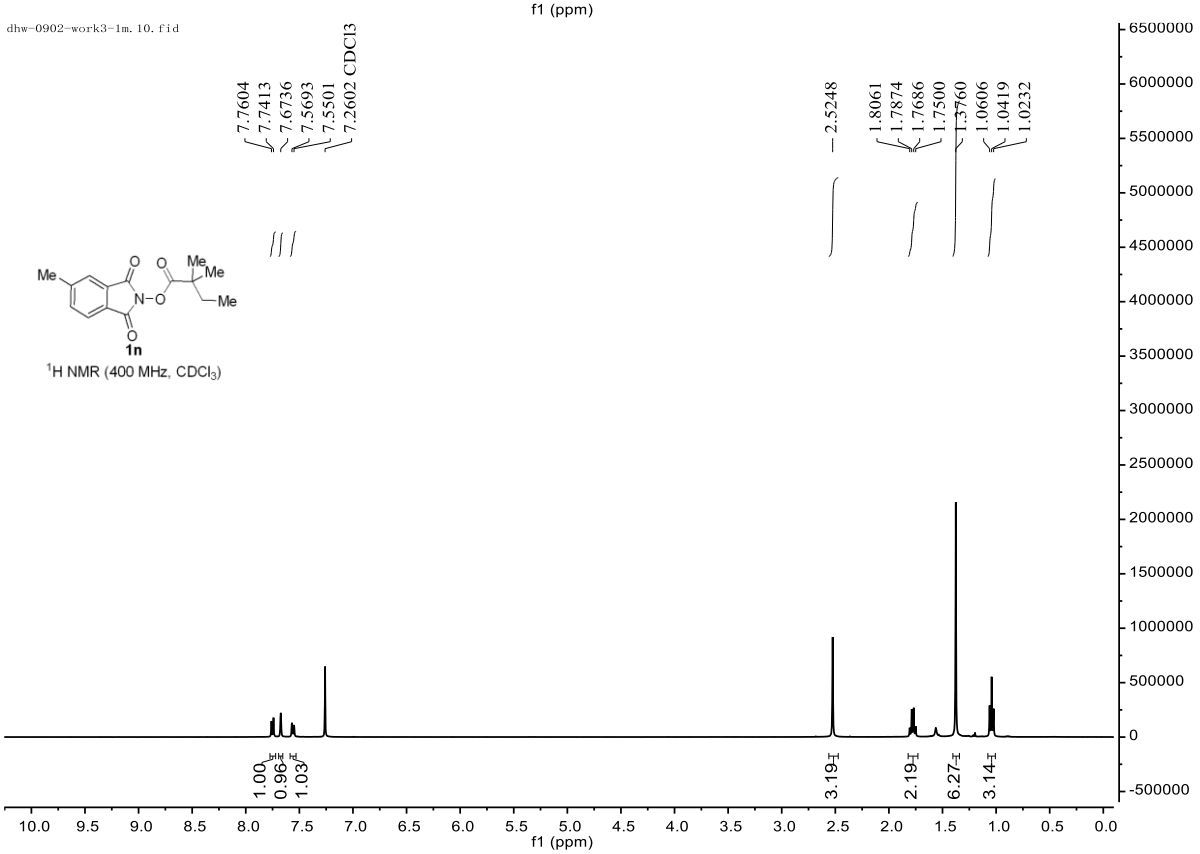
f1 (ppm)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1.7686
1.7500
1.3760
1.0606
1.0419
1.0232
Me
Me
Me
Me
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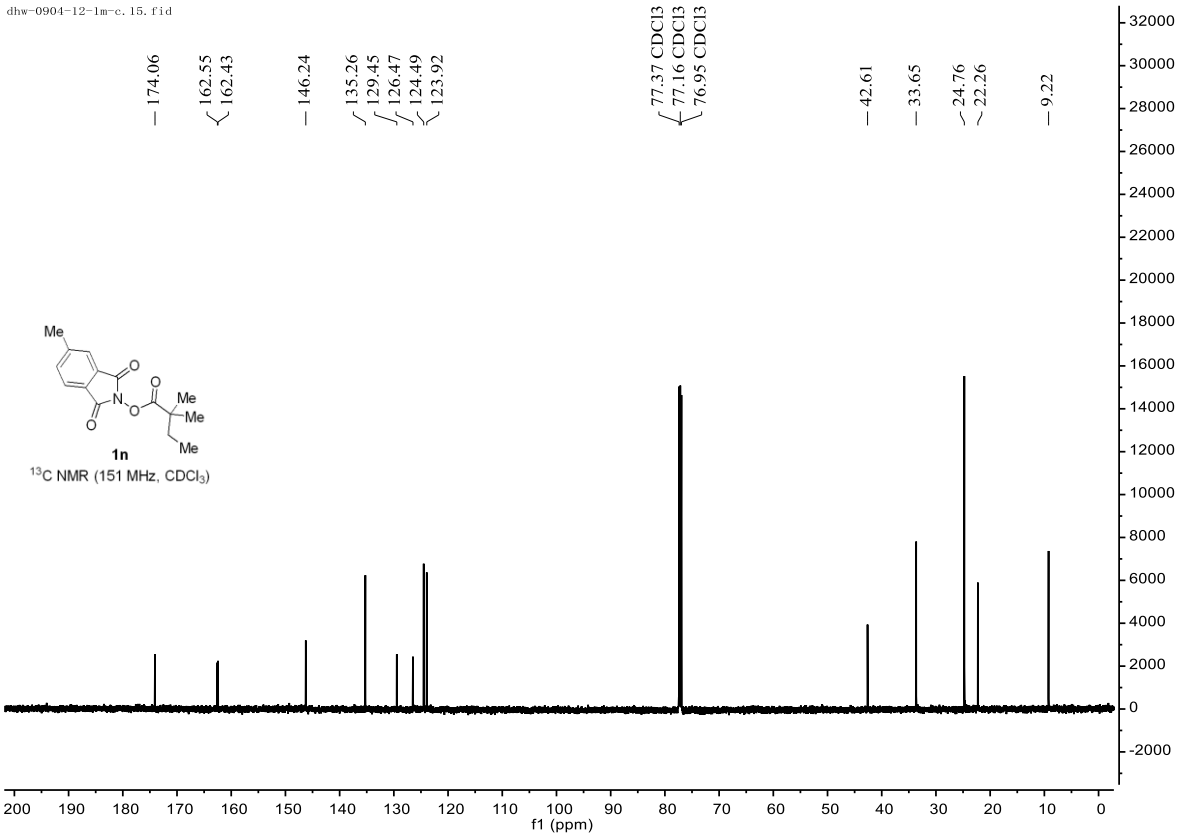
3.19
2.19
6.27
3.14
f1 (ppm)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77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
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22.26
9.22
Me
Me
Me
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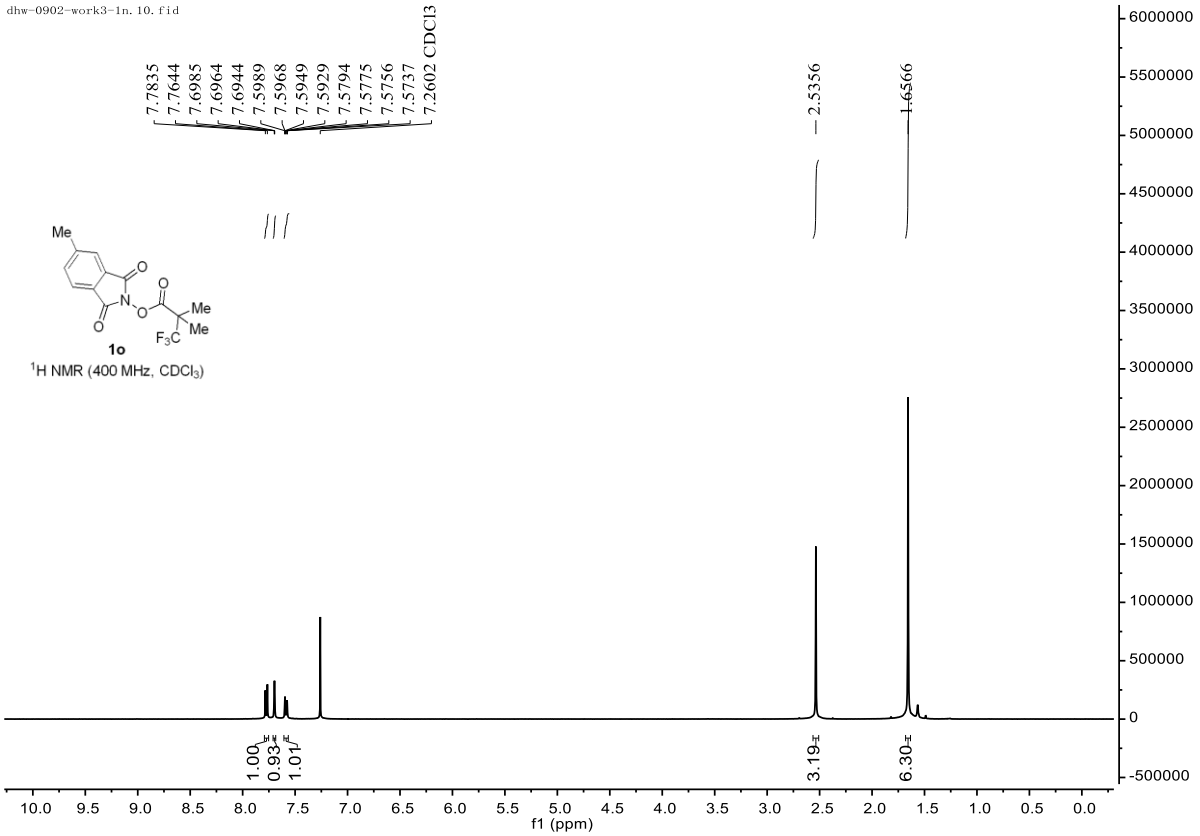
1n
13C NMR (151 MHz, CDCl3)
f1 (ppm)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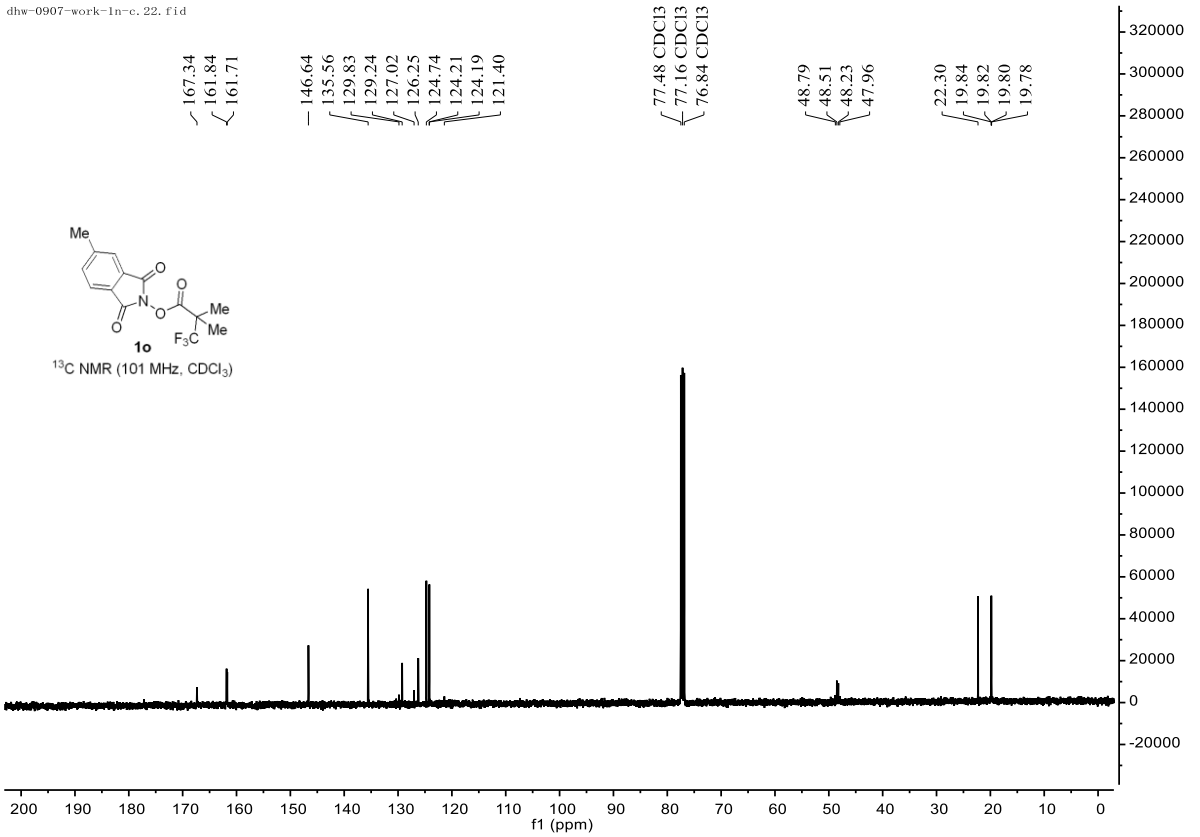
7.5794
7.5775
7.5756
7.5737
7.2602 CDCl3
2.5356
1.6566
Me
Me
Me
F3C
1o
1H NMR (400 MHz, CDCl3)
1.00
0.93
1.01
3.19
6.30
f1 (ppm)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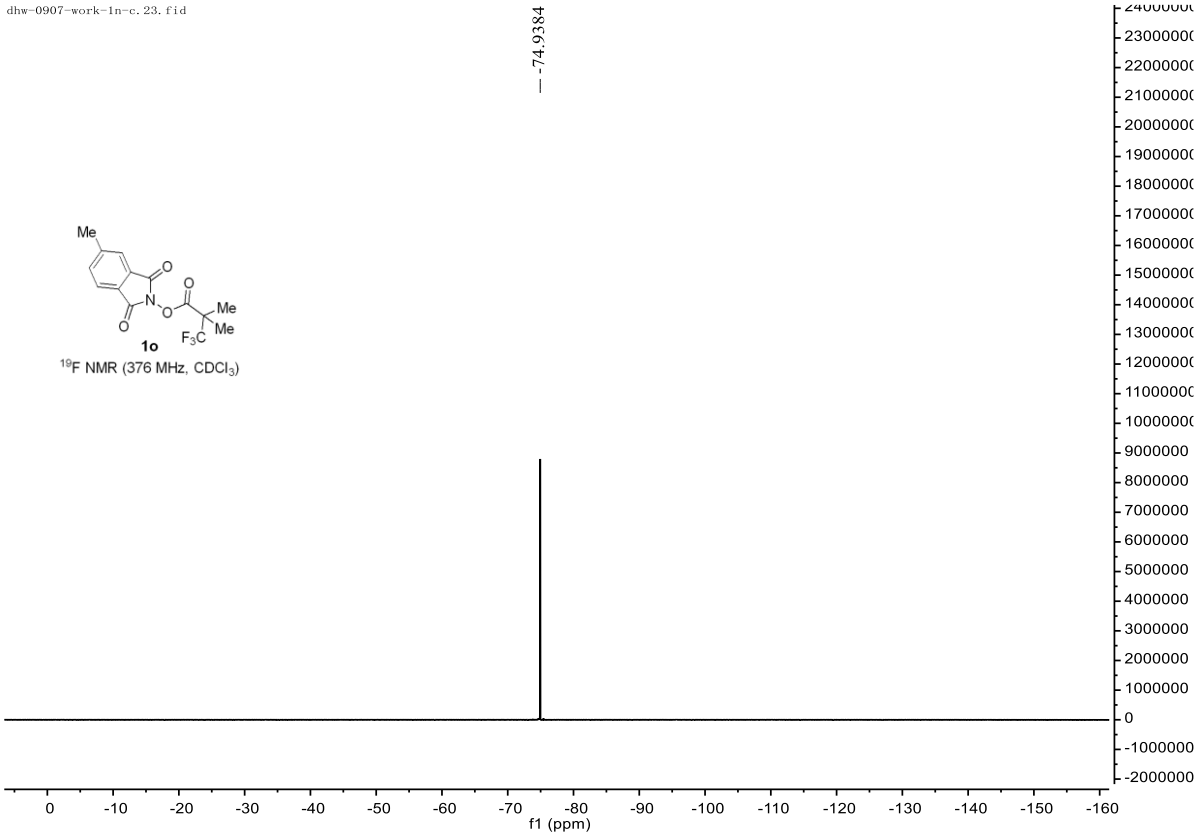
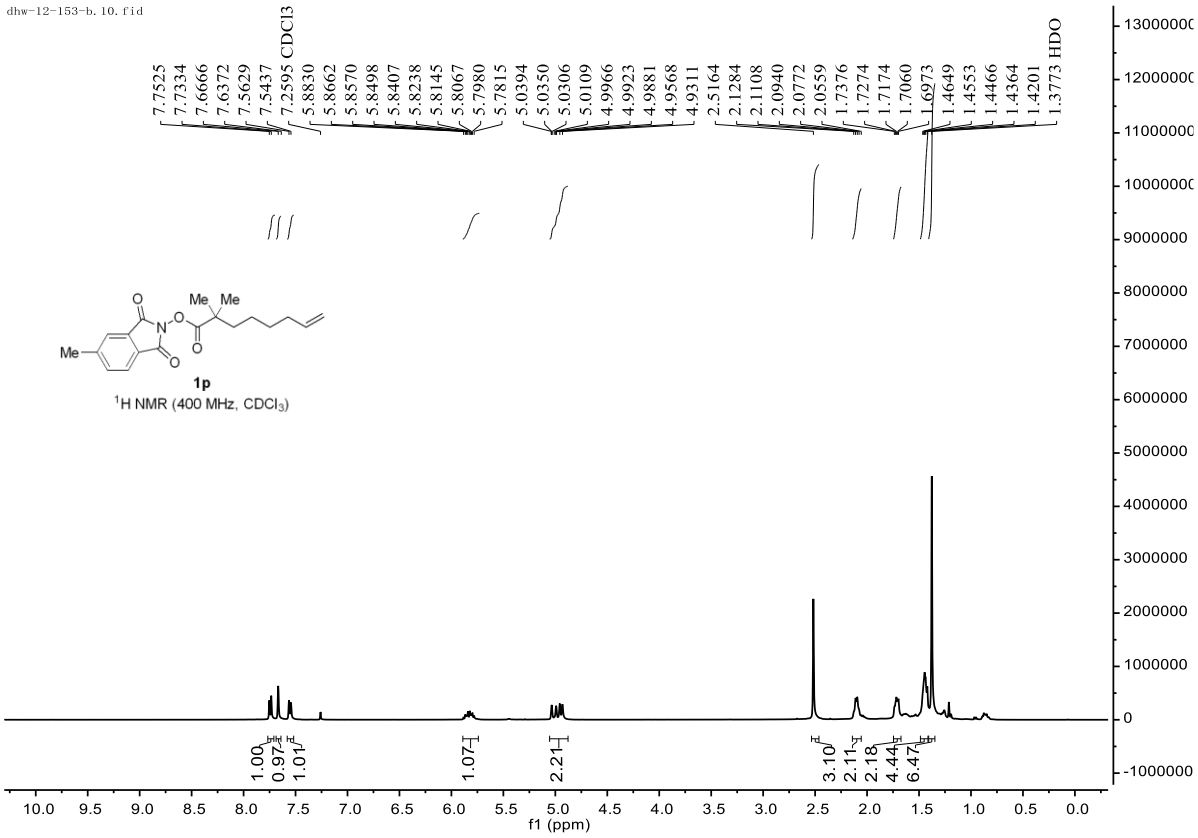
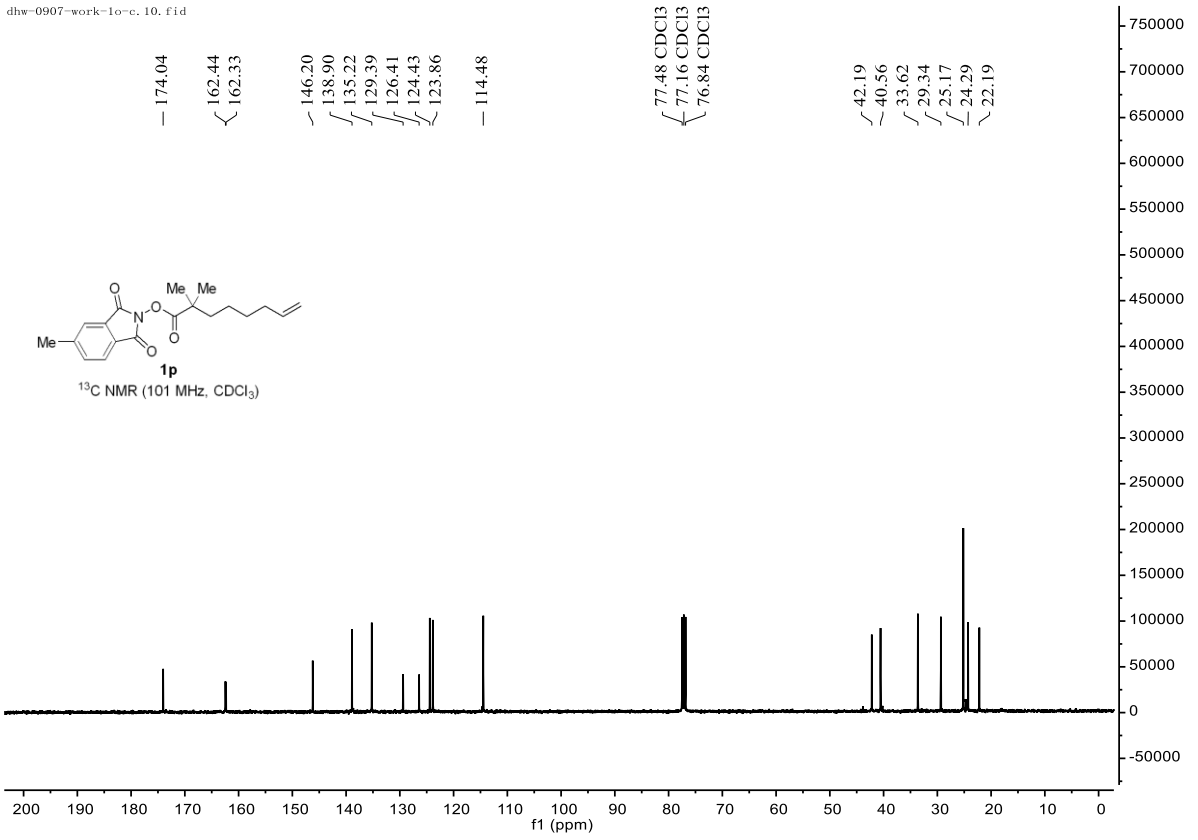
167.34
161.84
161.71
146.64
135.56
129.83
129.24
127.02
126.25
124.74
124.21
124.19
121.40
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
48.79
48.51
48.23
47.96
22.30
19.84
19.82
19.80
19.78
Me
O
O
N
O
O
Me
Me
F3C
1o
13C NMR (101 MHz, CDCl3)
f1 (ppm)


dhw-0907-work-1n-c.23.fid
-74.9384
Me
O
O
N
O
O
Me
Me
F3C
1o
19F NMR (376 MHz, CDCl3)
f1 (ppm)



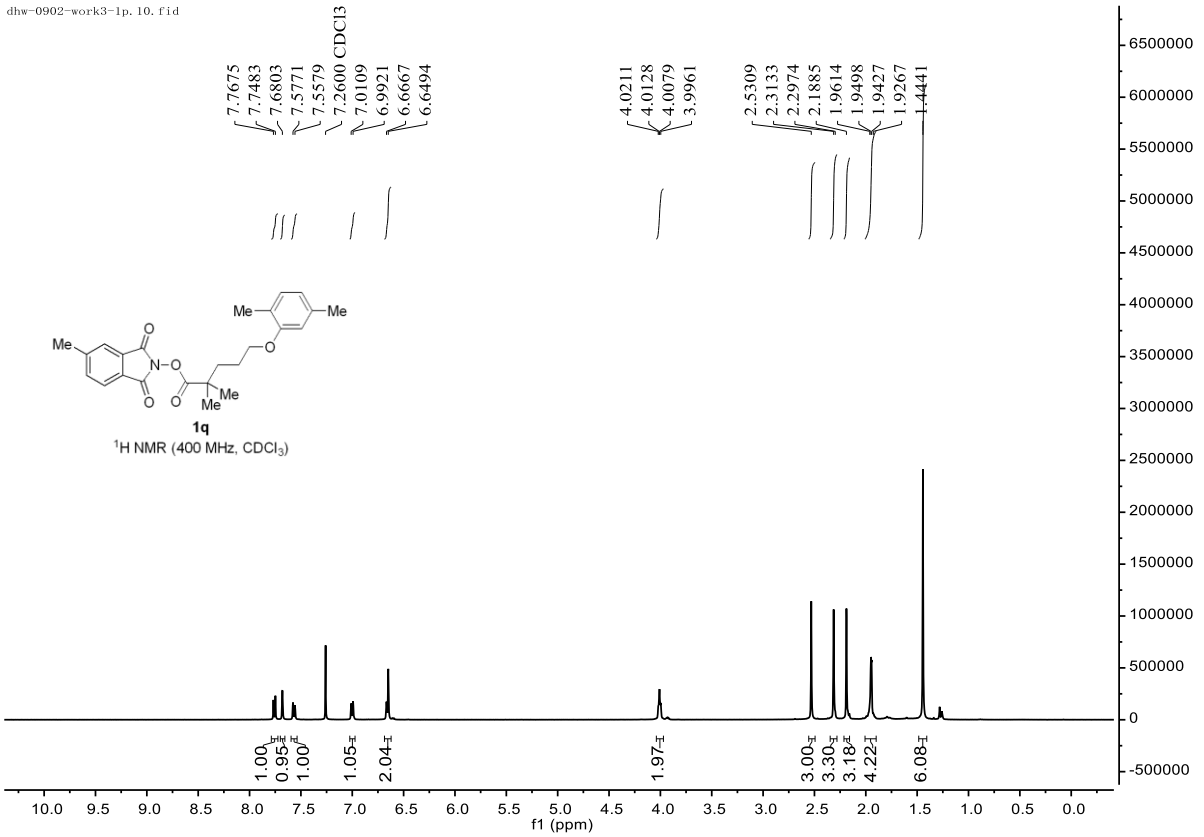
1p

^{1}H NMR (400 MHz, $CDCl_3$)



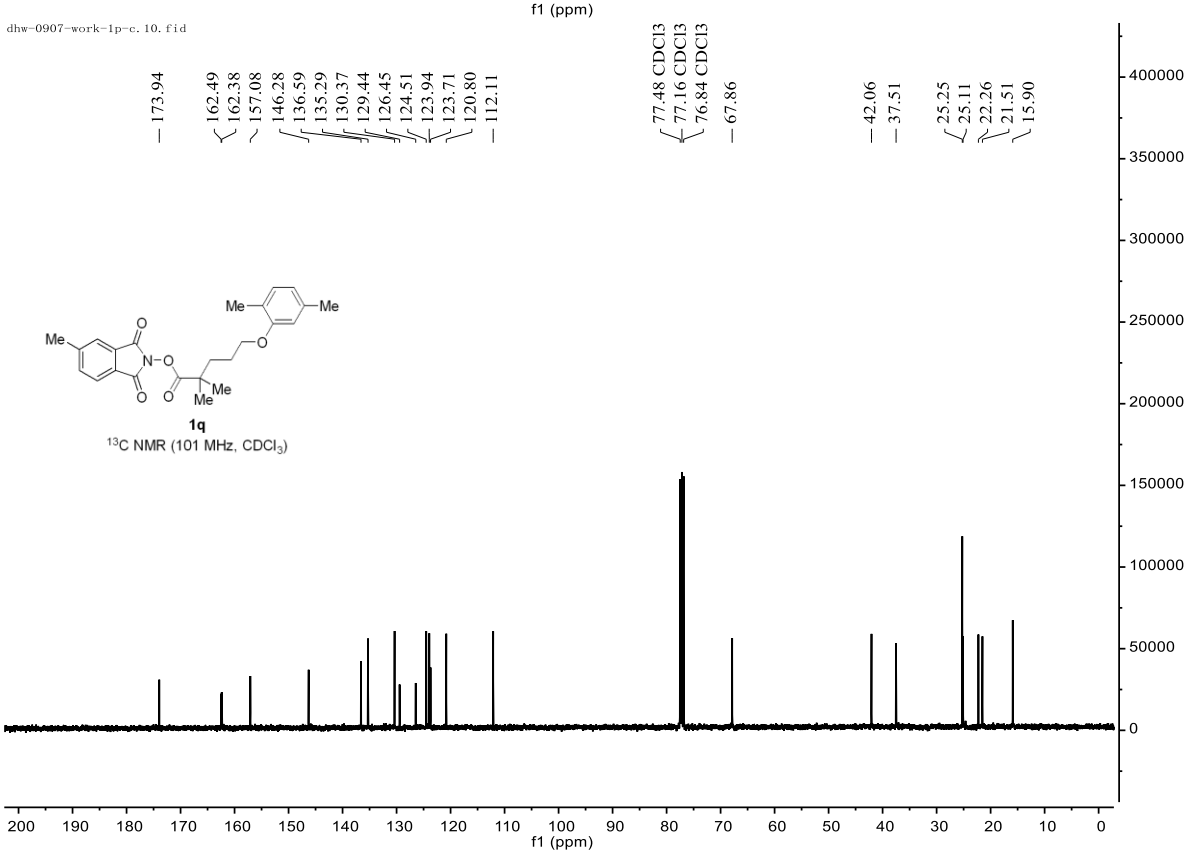
1p

^{13}C NMR (101 MHz, $CDCl_3$)



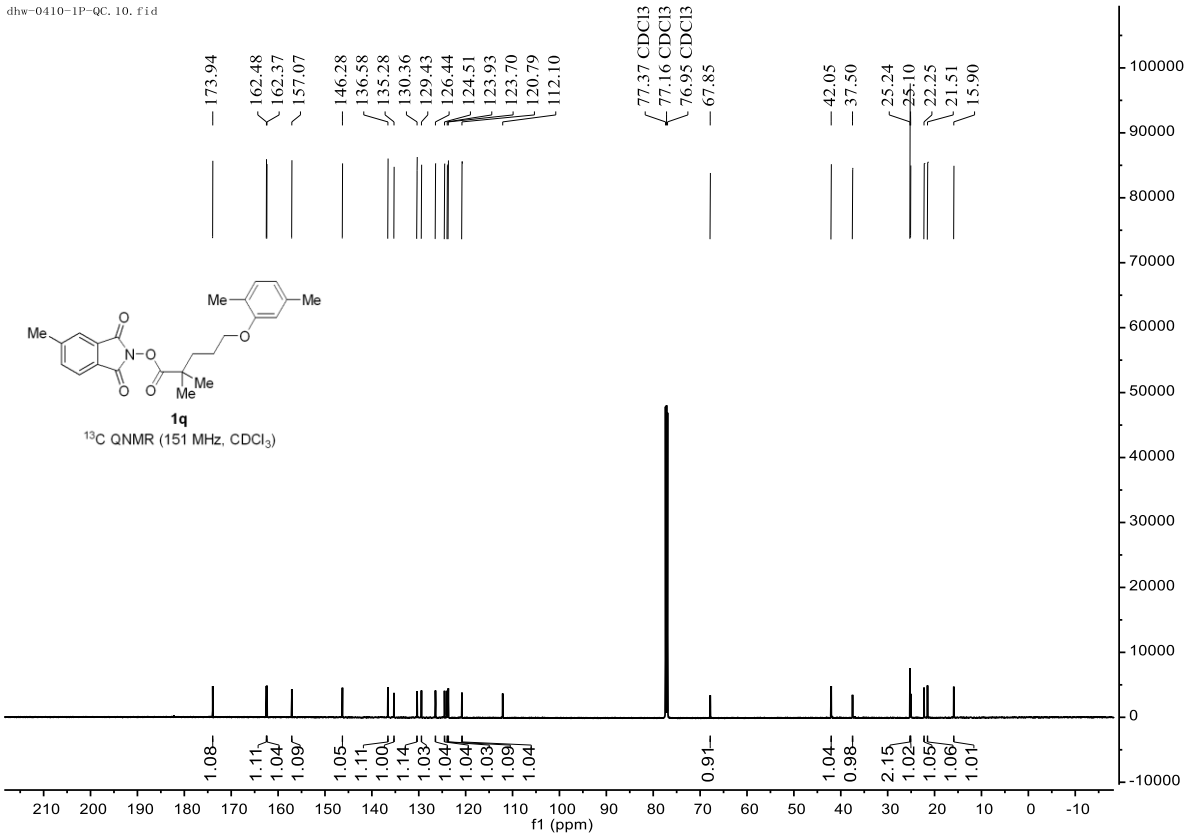
1q

^{1}H NMR (400 MHz, $CDCl_3$)



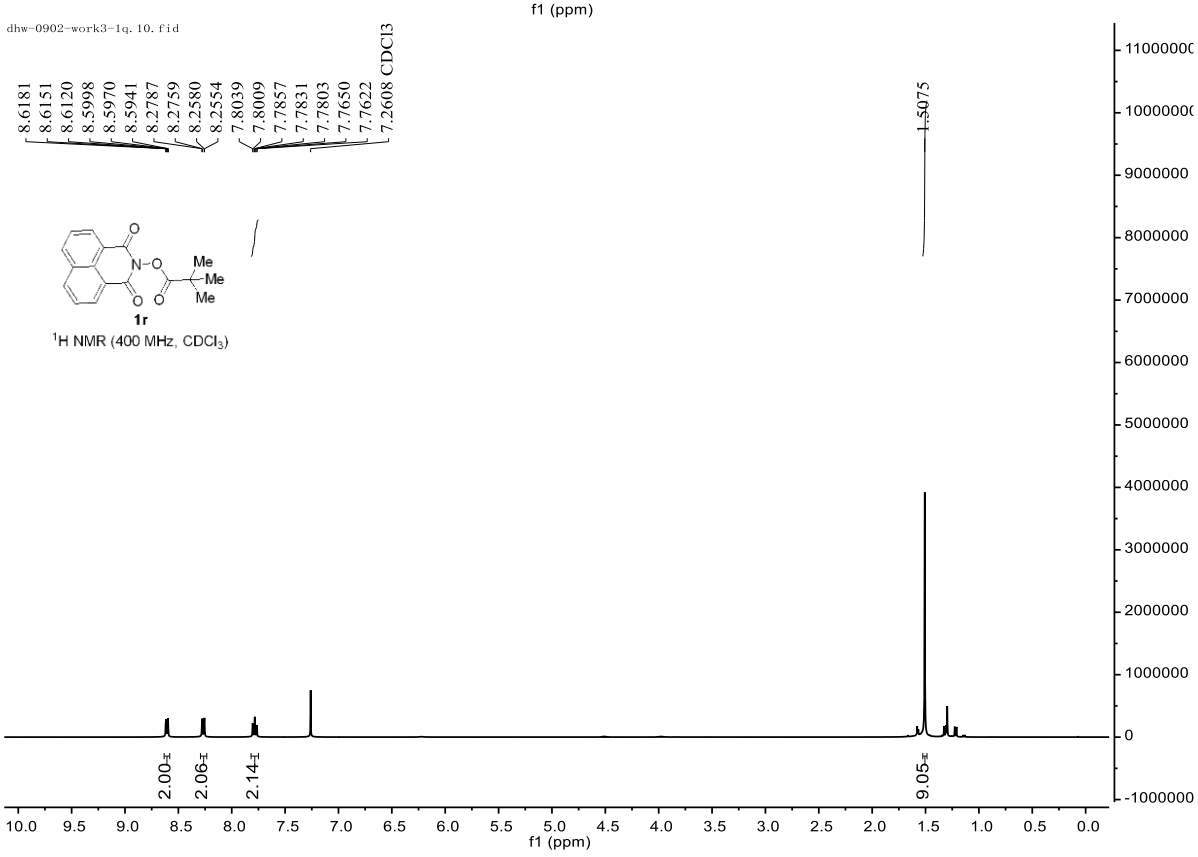
1q

^{13}C NMR (101 MHz, $CDCl_3$)



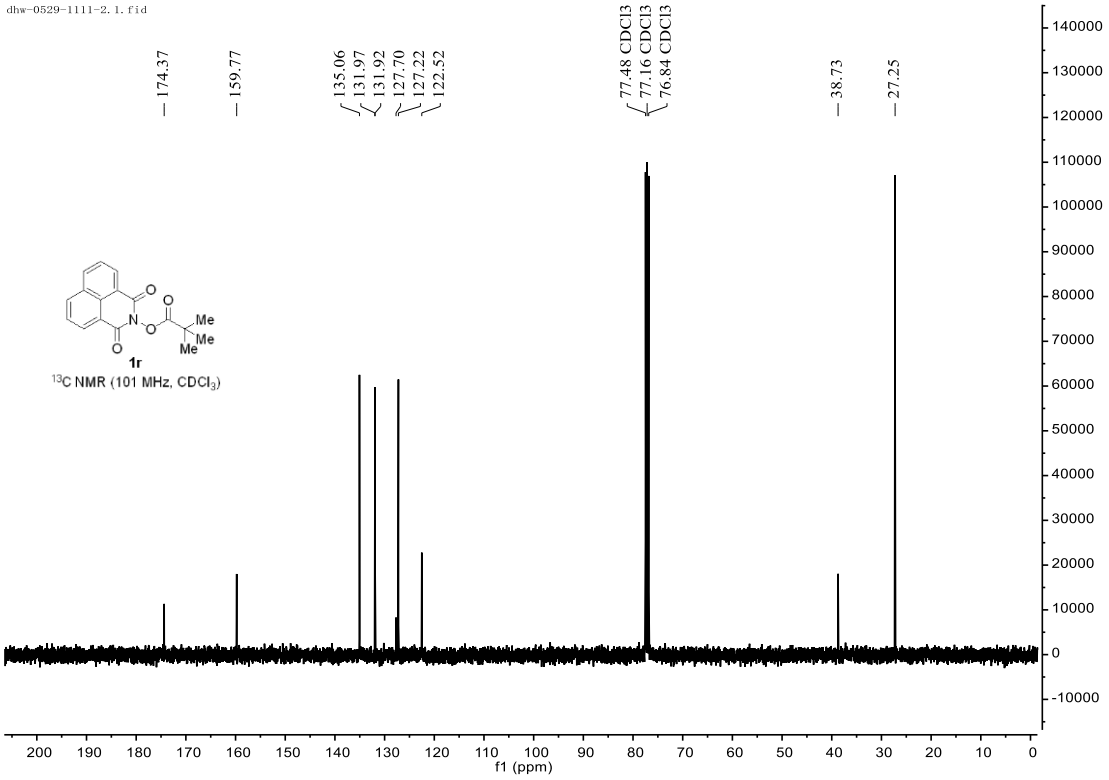
1q

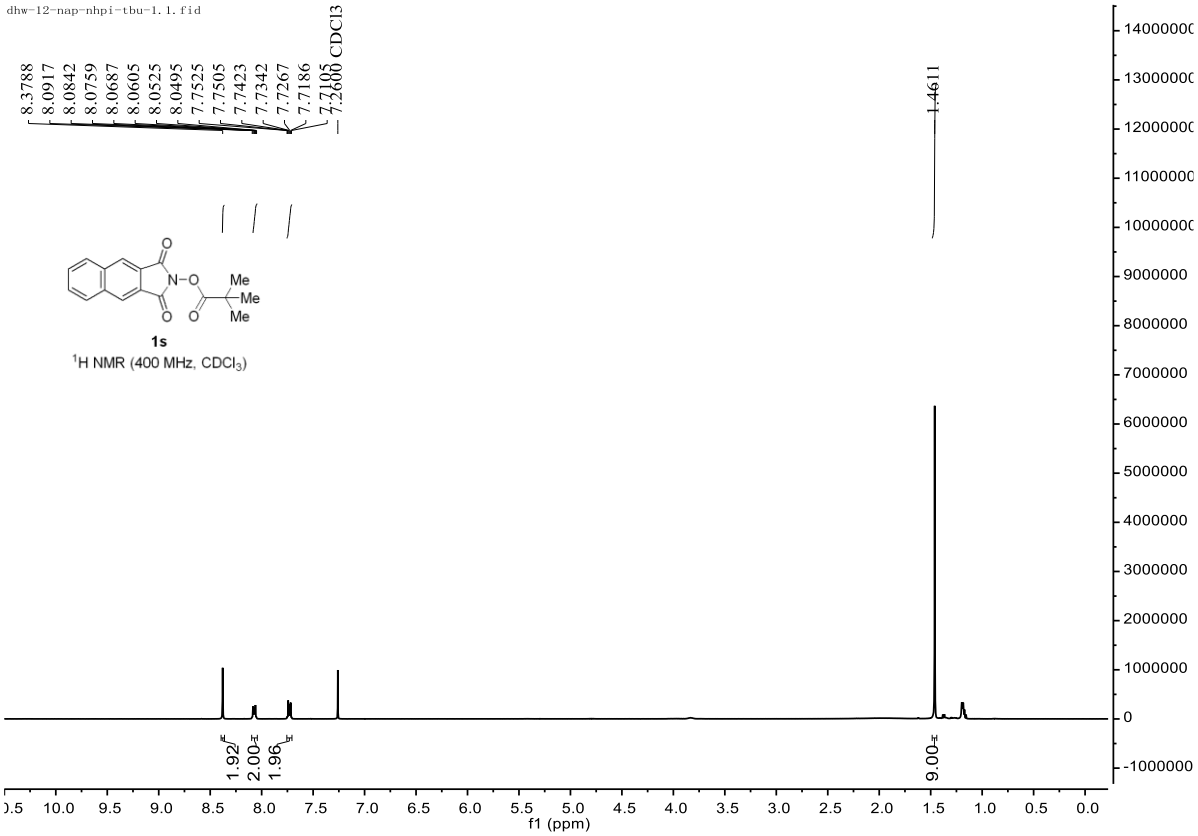
^{13}C QNMR (151 MHz, $CDCl_3$)



1r

^{1}H NMR (400 MHz, $CDCl_3$)


dhw-0529-1111-2.1.fid
174.37
159.77
135.06
131.97
131.92
127.70
127.22
122.52
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
38.73
27.25
1r
13C NMR (101 MHz, CDCl3)
f1 (ppm)


dhw-12-nap-nhpi-tbu-1.1.fid
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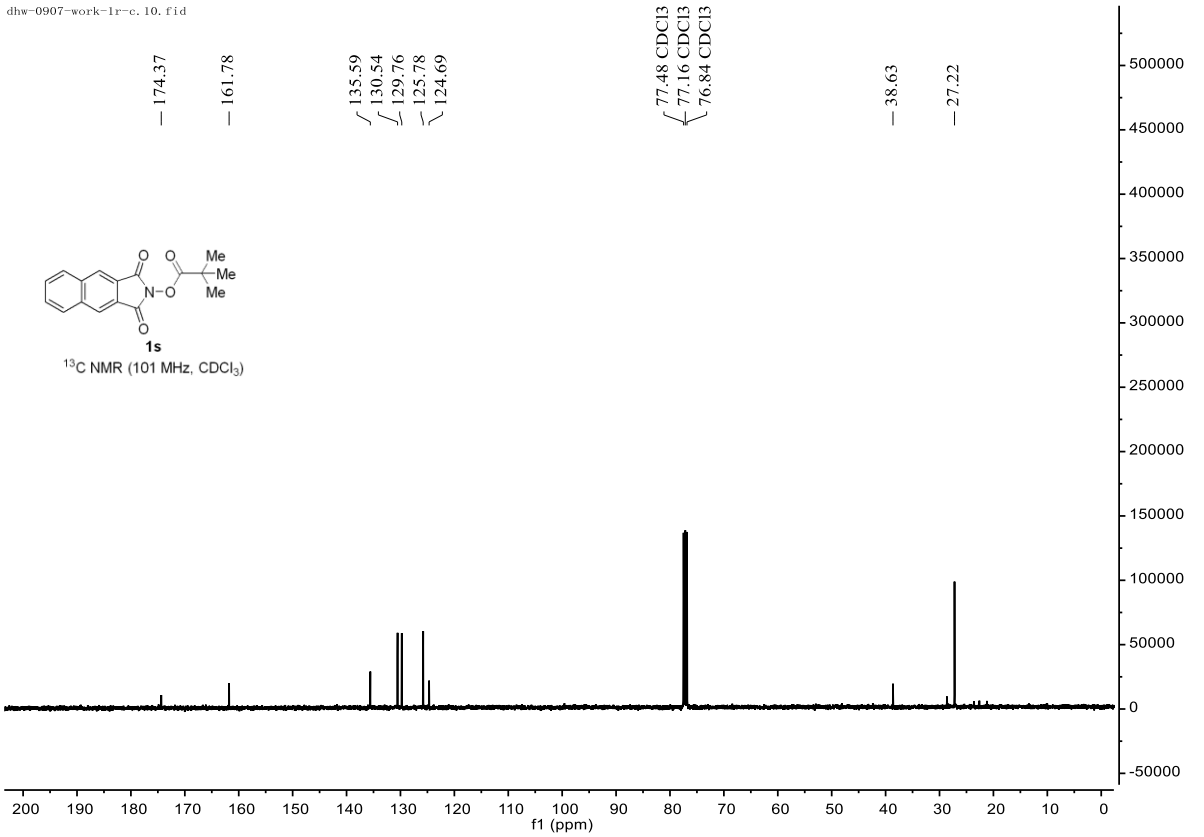
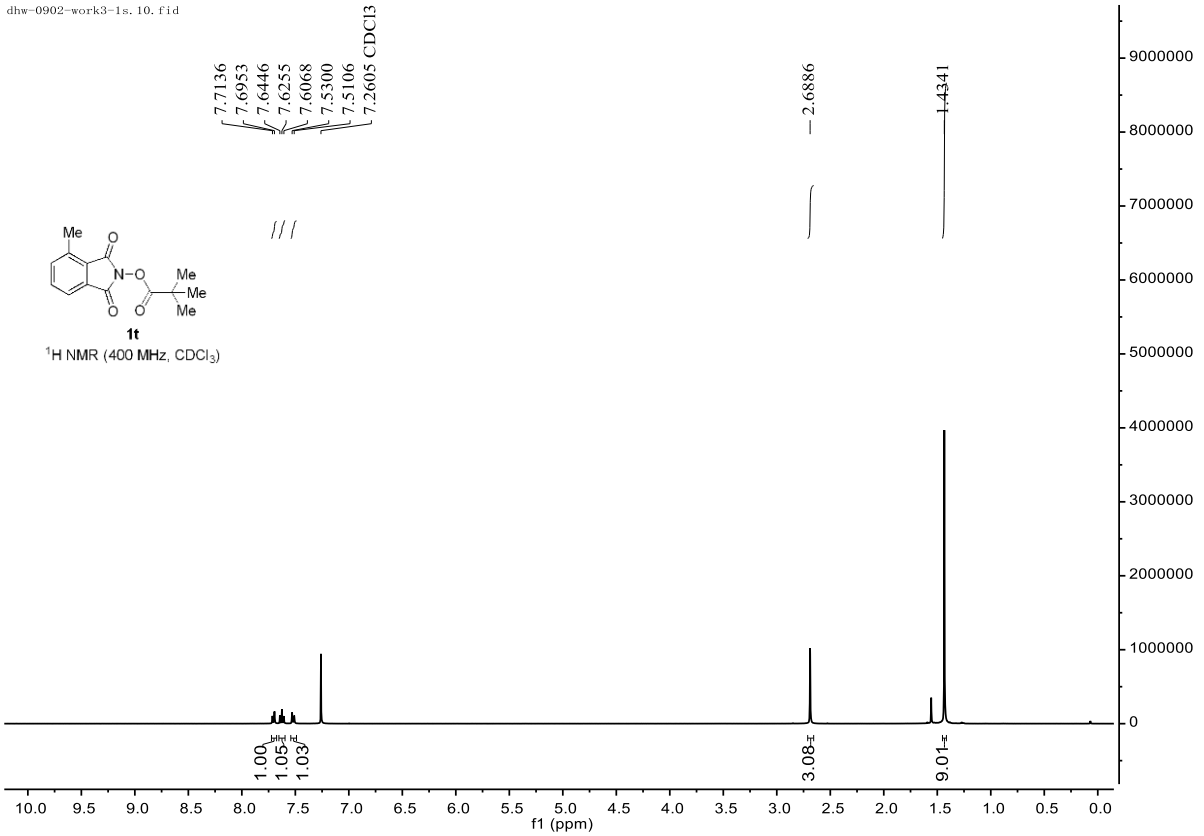
8.0917
8.0842
8.0759
8.0687
8.0605
8.0525
8.0495
7.7525
7.7505
7.7423
7.7342
7.7267
7.7186
7.7105
7.2600 CDCl3
1.4611
1s
1H NMR (400 MHz, CDCl3)
1.92
2.00
1.96
9.00
f1 (ppm)


dhw-0907-work-1r-c.10.fid
174.37
161.78
135.59
130.54
129.76
125.78
124.69
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
38.63
27.22
1s
13C NMR (101 MHz, CDCl3)
f1 (ppm)


dhw-0902-work3-1s.10.fid
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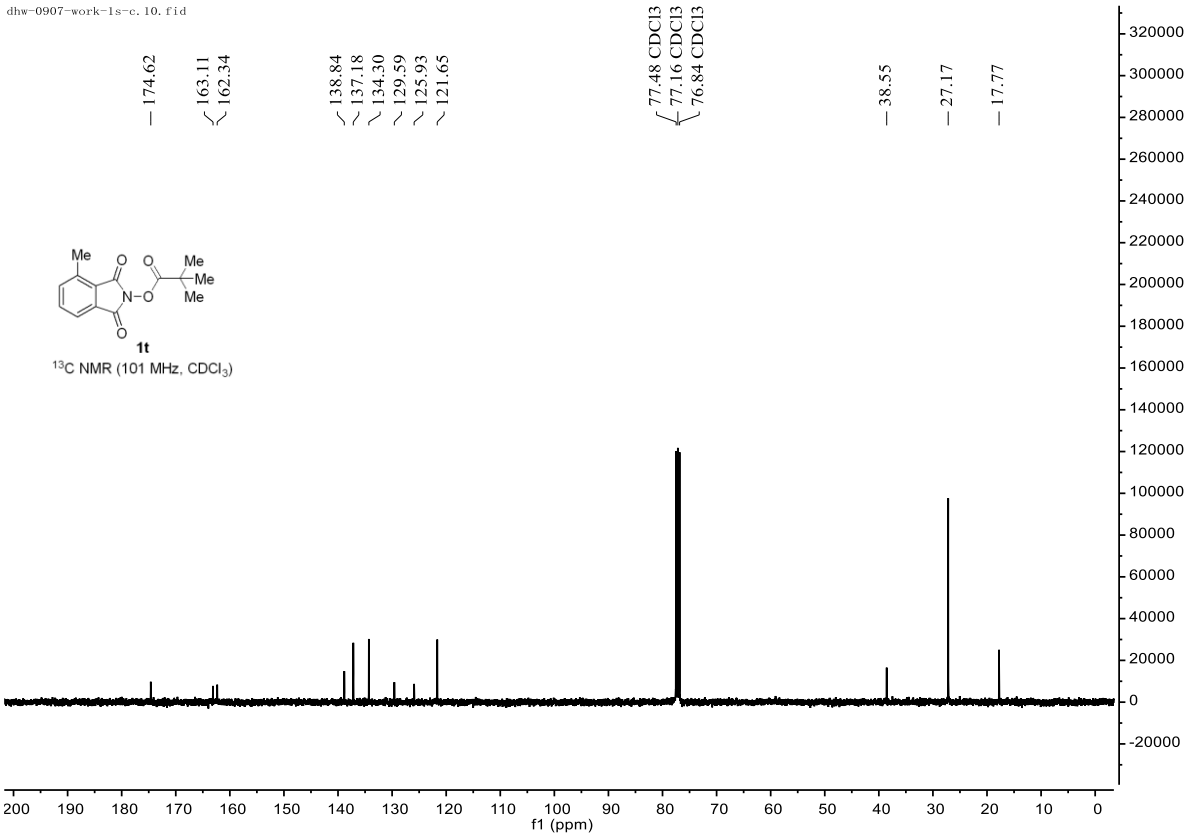
7.6953
7.6446
7.6255
7.6068
7.5300
7.5106
7.2605 CDCl3
2.6886
1.4341
1.00
1.05
1.03
3.08
9.01
1t
1H NMR (400 MHz, CDCl3)
f1 (ppm)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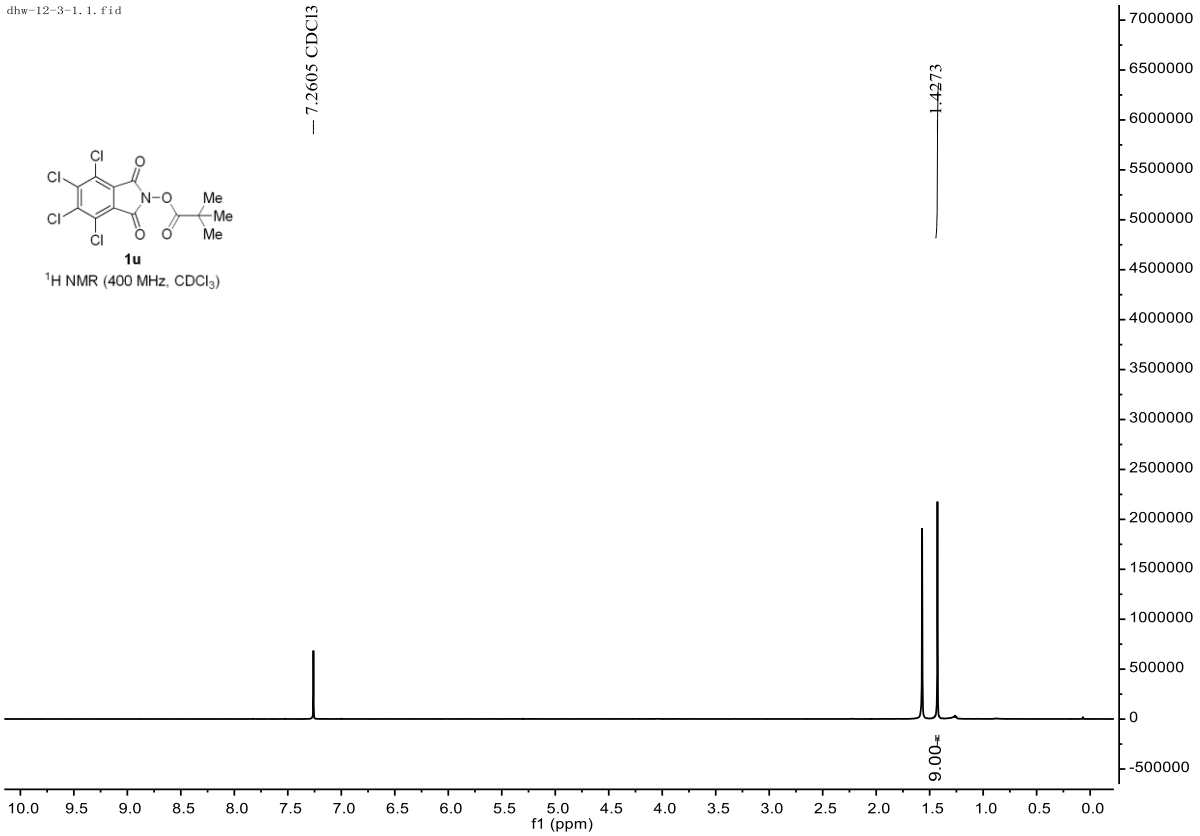
174.62
163.11
162.34
138.84
137.18
134.30
129.59
125.93
121.65
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
38.55
27.17
17.77
1t
13C NMR (101 MHz, CDCl3)
f1 (ppm)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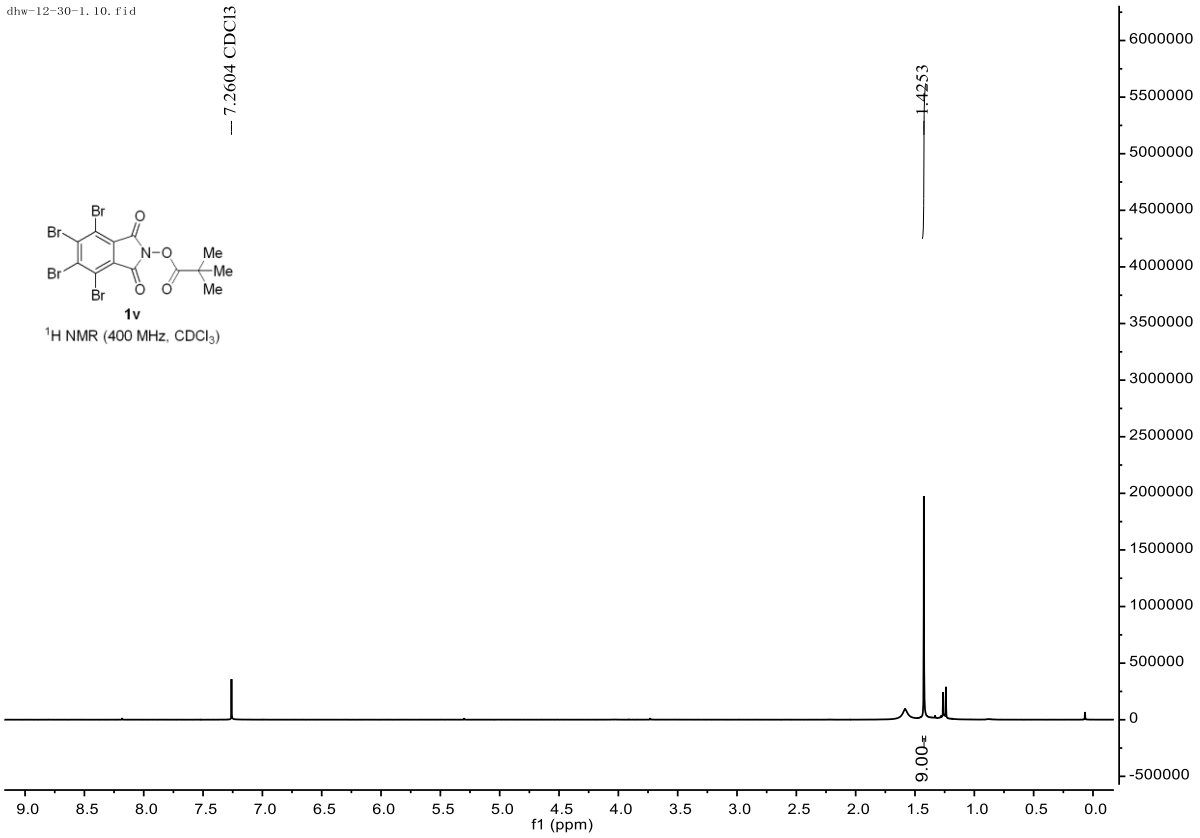
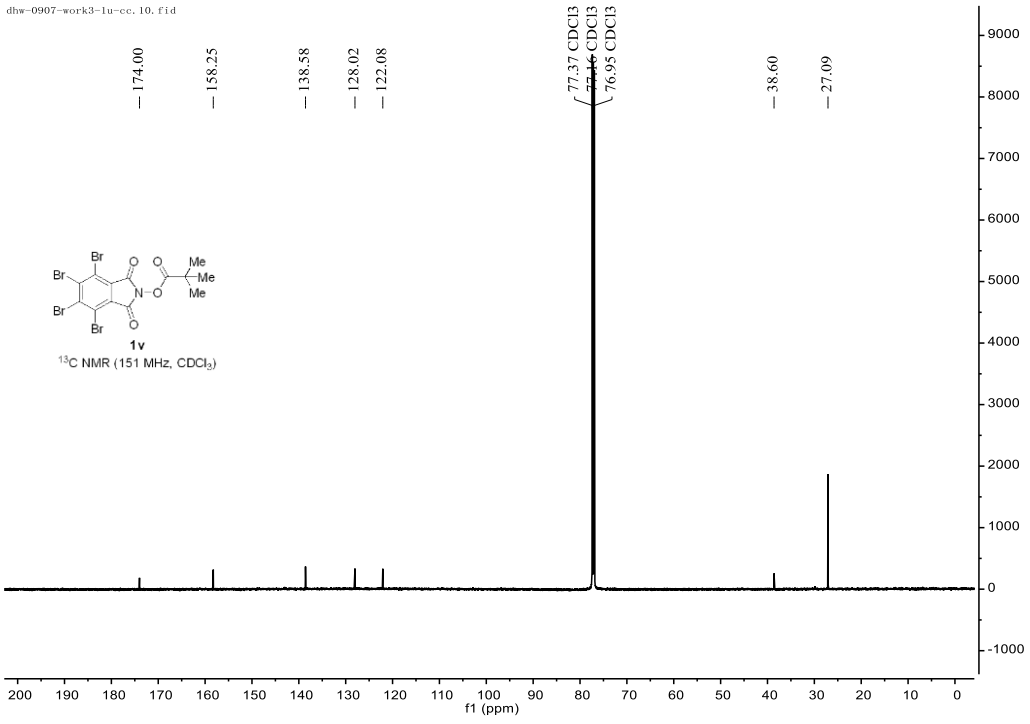
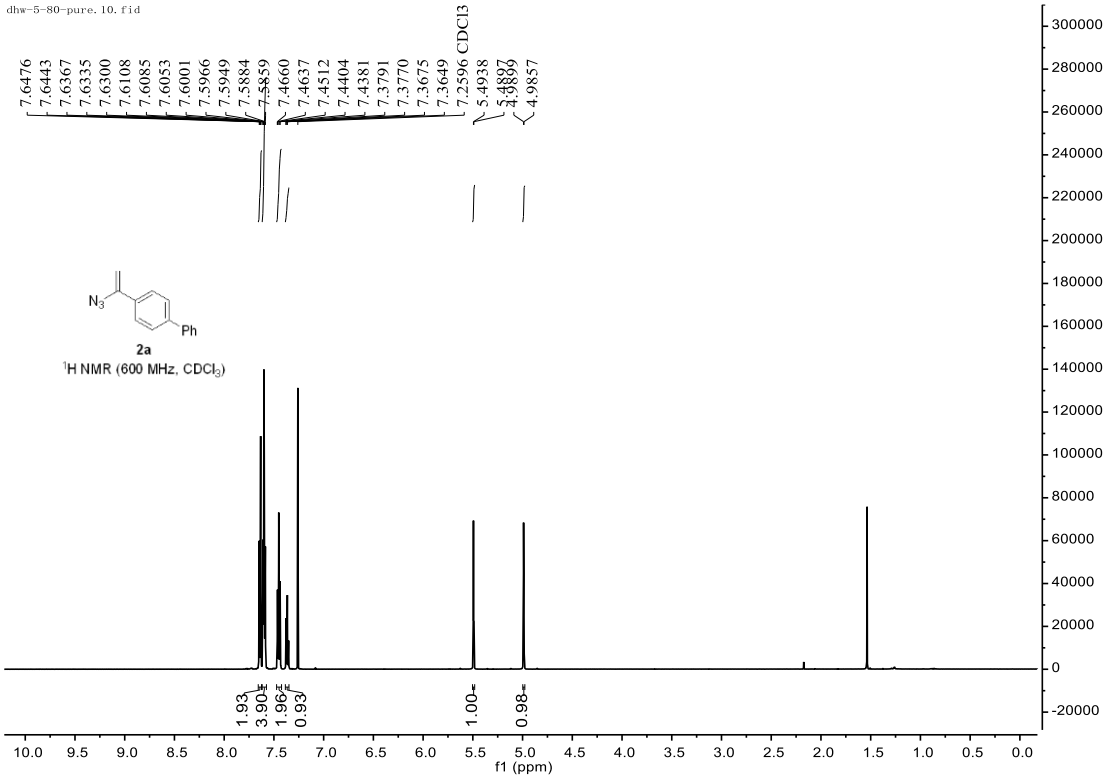
1.4273
9.00
1u
1H NMR (400 MHz, CDCl3)
f1 (ppm)


dhw-12-30-1.10.fid
7.2604 CDCl3
1.4253
9.00
f1 (ppm)
1v
^{1}H NMR (400 MHz, CDCl$_3$)


dhw-0907-work3-1u-cc.10.fid
174.00
158.25
138.58
128.02
122.08
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
38.60
27.09
f1 (ppm)
1v
^{13}C NMR (151 MHz, CDCl$_3$)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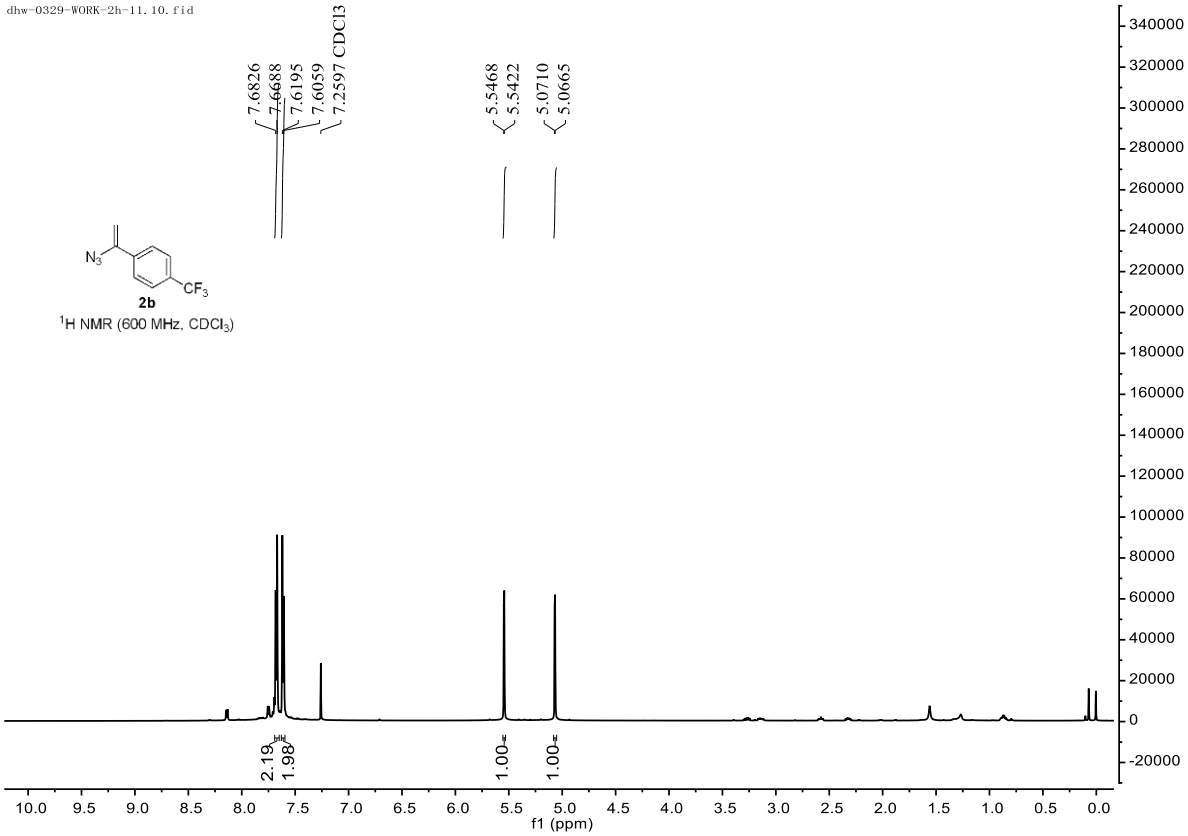
7.6443
7.6367
7.6335
7.6300
7.6108
7.6085
7.6053
7.6001
7.5966
7.5949
7.5884
7.5859
7.4660
7.4637
7.4512
7.4404
7.4381
7.3791
7.3770
7.3675
7.3649
7.2596 CDCl3
5.4938
5.4887
4.9889
4.9857
N3
Ph
2a
1H NMR (600 MHz, CDCl3)
1.93
3.90
1.96
0.93
1.00
0.98
f1 (ppm)


dhw-0329-WORK-2h-11.10.fid
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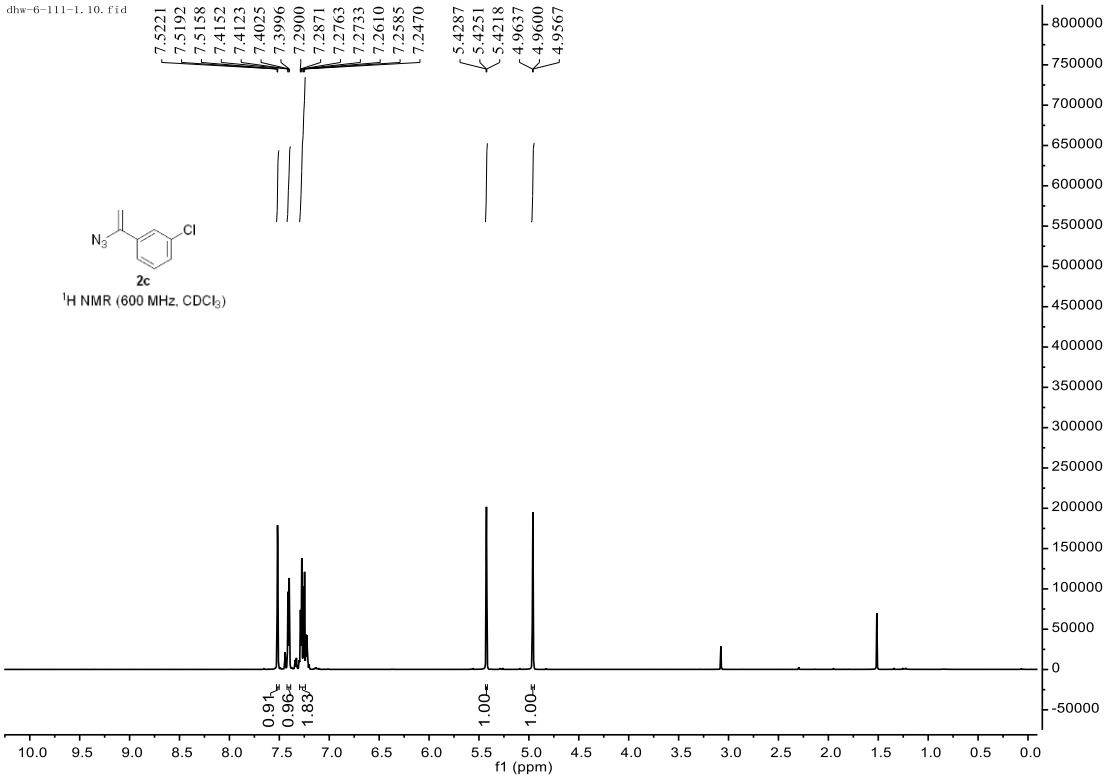
7.6688
7.6195
7.6059
7.2597 CDCl3
5.5468
5.5422
5.0710
5.0665
N3
CF3
2b
1H NMR (600 MHz, CDCl3)
2.19
1.98
1.00
1.00
f1 (ppm)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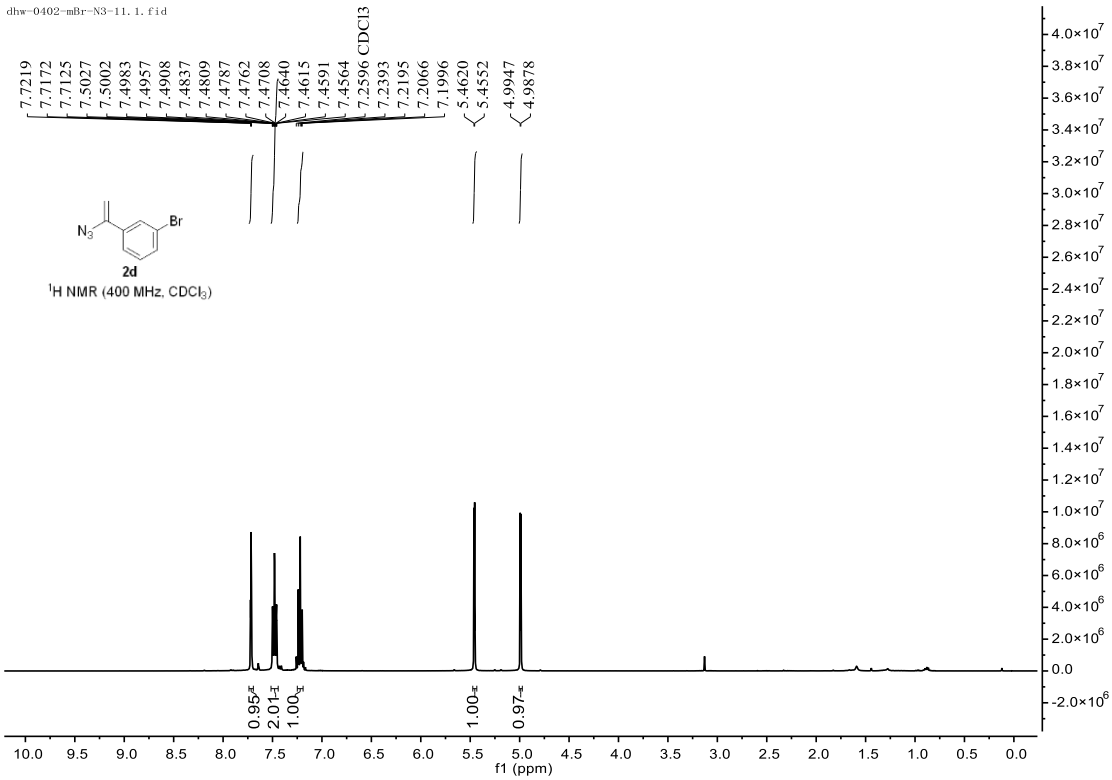
7.5192
7.5158
7.4152
7.4123
7.4025
7.3996
7.2900
7.2871
7.2763
7.2733
7.2610
7.2585
7.2470
5.4287
5.4251
5.4218
4.9637
4.9600
4.9567
N3
Cl
2c
1H NMR (600 MHz, CDCl3)
0.91
0.96
1.83
1.00
1.00
f1 (ppm)


dhw-0402-mBr-N3-11.1.fid
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7.7172
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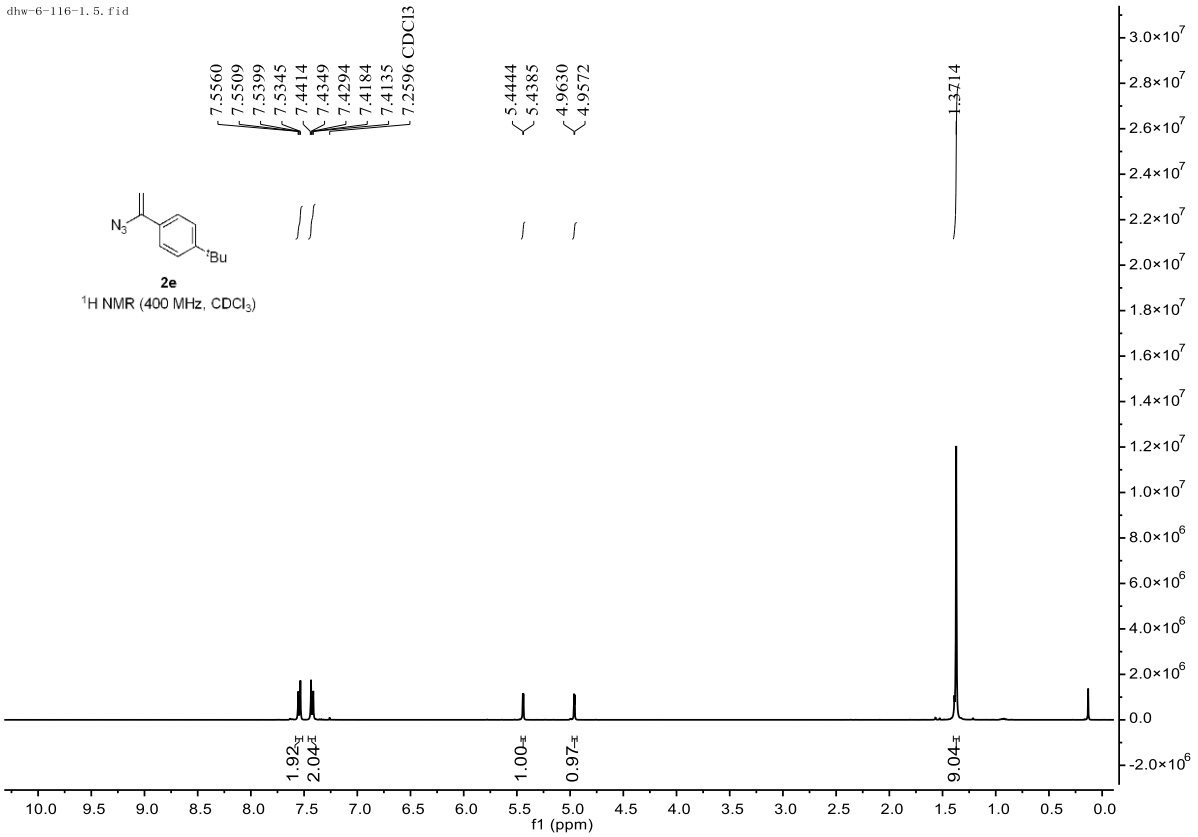
7.5027
7.5002
7.4983
7.4957
7.4908
7.4837
7.4809
7.4787
7.4762
7.4708
7.4640
7.4615
7.4591
7.4564
7.2596 CDCl3
7.2393
7.2195
7.2066
7.1996
5.4620
5.4552
4.9947
4.9878
N3
Br
2d
1H NMR (400 MHz, CDCl3)
0.95
2.01
1.00
1.00
0.97
f1 (ppm)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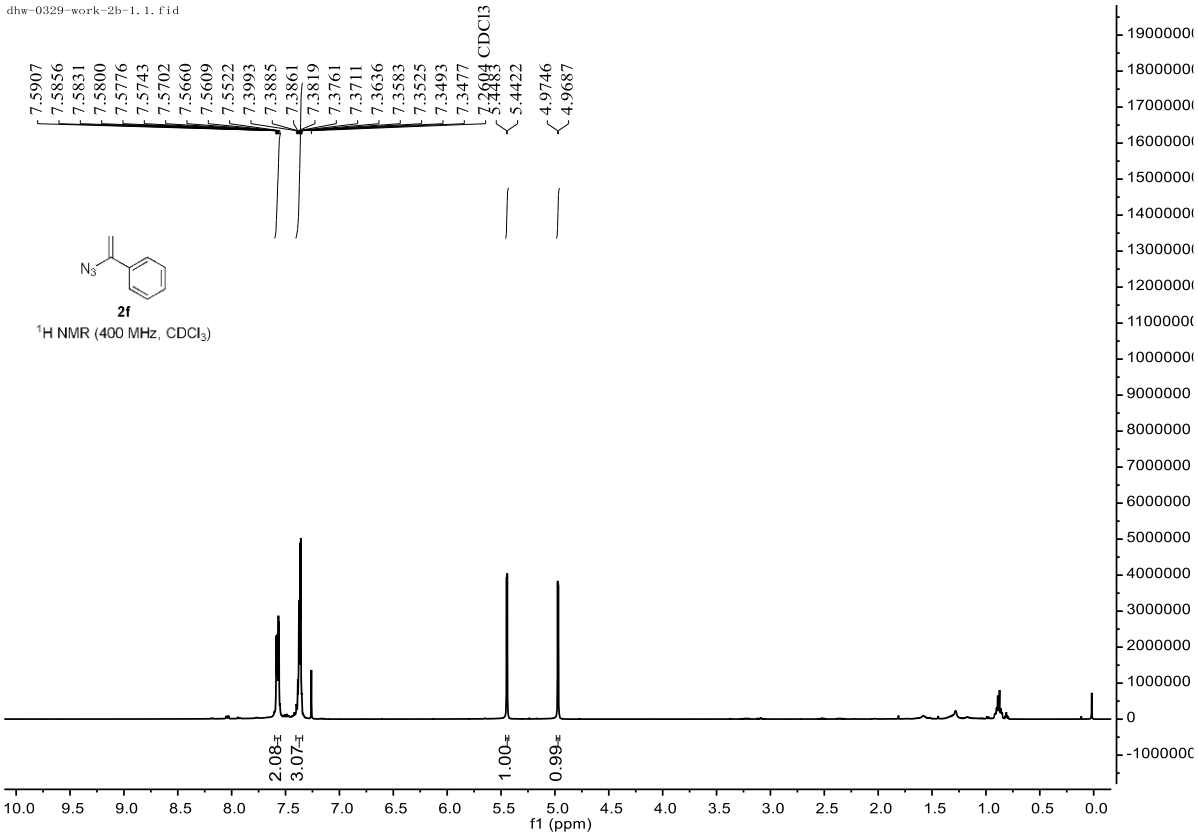
7.5509
7.5399
7.5345
7.4414
7.4349
7.4294
7.4184
7.4135
7.2596 CDCl3
5.4444
5.4385
4.9630
4.9572
1.3714
N3
tBu
2e
1H NMR (400 MHz, CDCl3)
1.92
2.04
1.00
0.97
9.04
f1 (ppm)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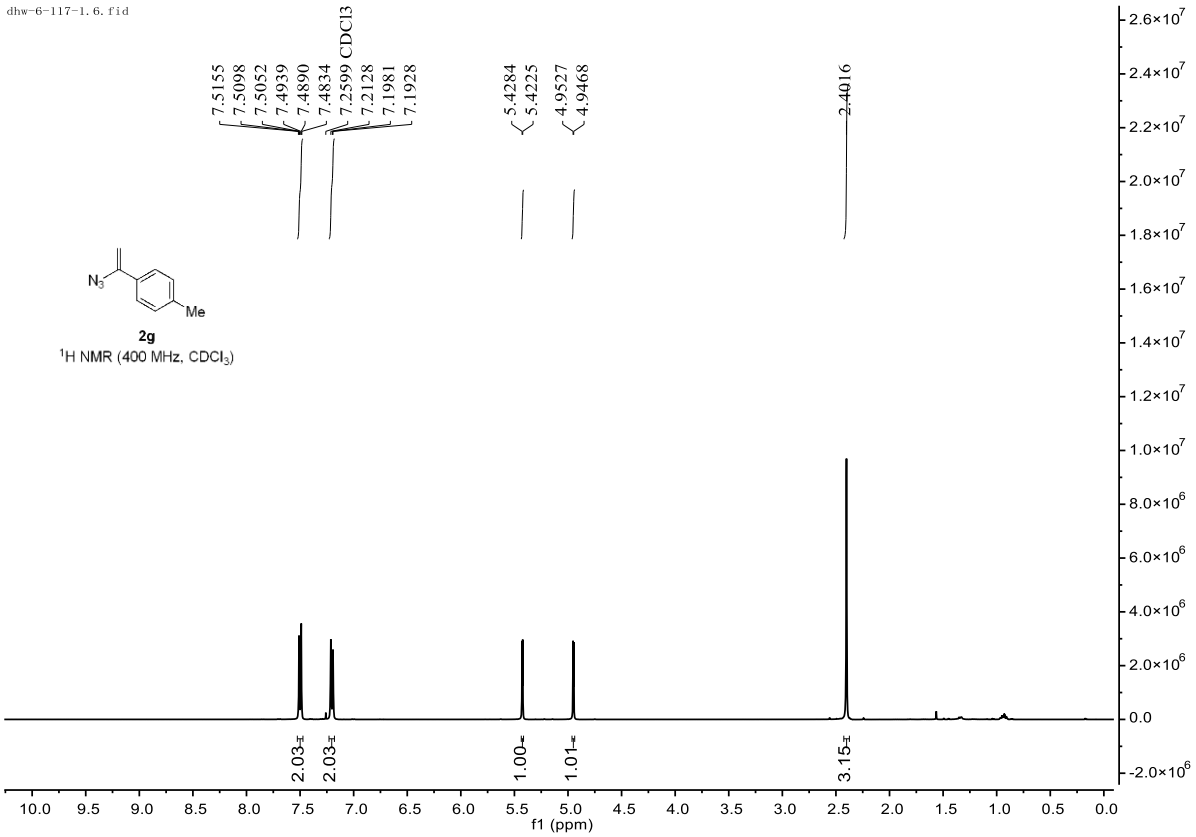
7.5776
7.5743
7.5702
7.5660
7.5609
7.5522
7.3993
7.3885
7.3861
7.3819
7.3761
7.3711
7.3636
7.3583
7.3525
7.3493
7.3477
7.2604 CDCl3
5.4483
5.4422
4.9746
4.9687
N3
2f
1H NMR (400 MHz, CDCl3)
2.08
3.07
1.00
0.99
f1 (ppm)


dhw-6-117-1.6.fid
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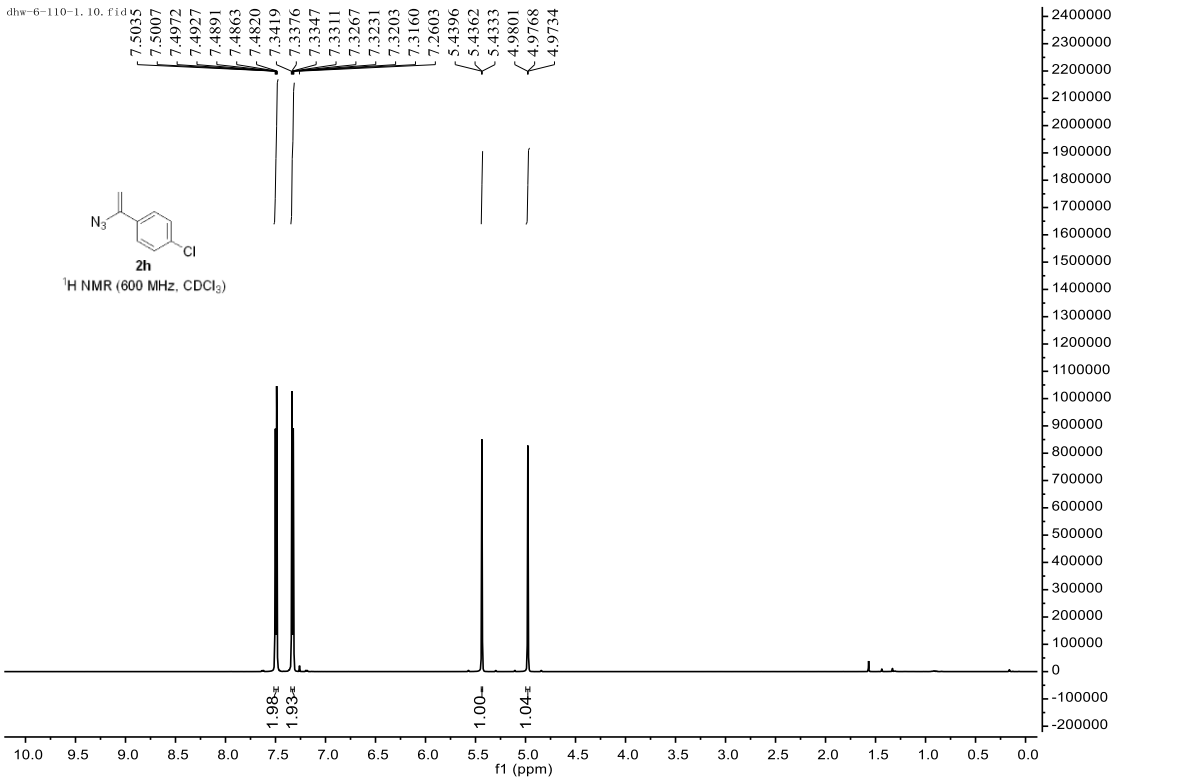
7.5052
7.4939
7.4890
7.4834
7.2599 CDCl3
7.2128
7.1981
7.1928
5.4284
5.4225
4.9527
4.9468
2.4016
N3
Me
2g
1H NMR (400 MHz, CDCl3)
2.03
2.03
1.00
1.01
3.15
f1 (ppm)


dhw-6-110-1.10.fid
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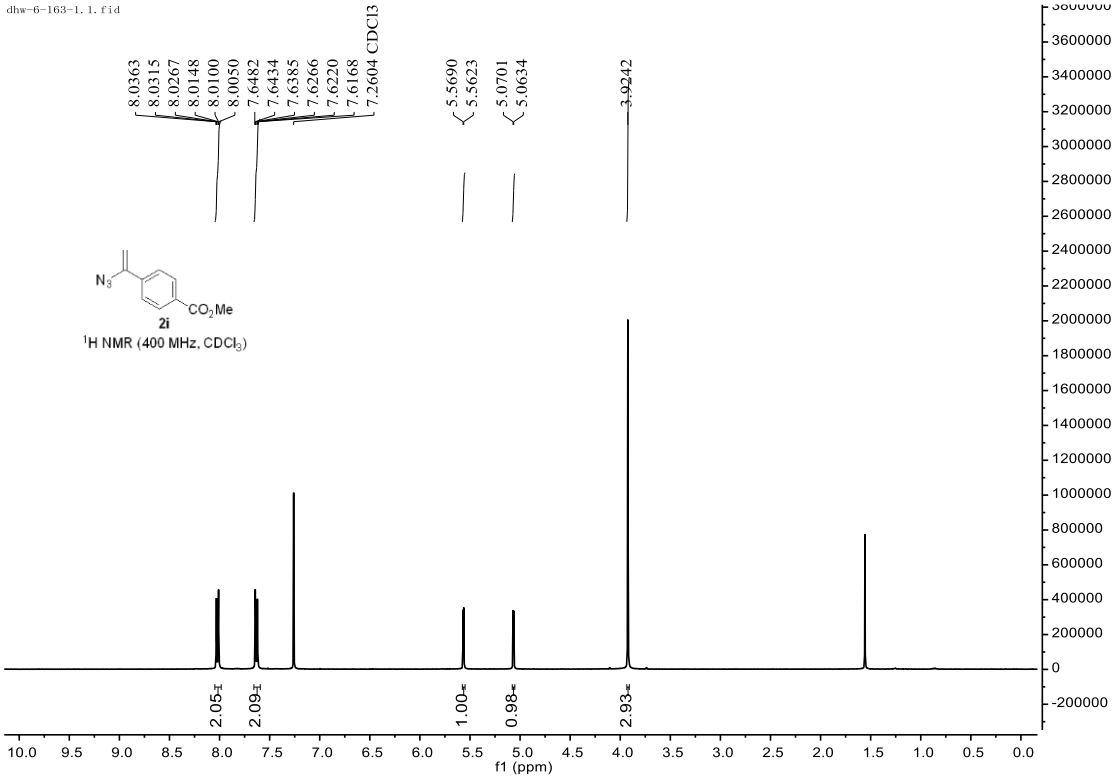
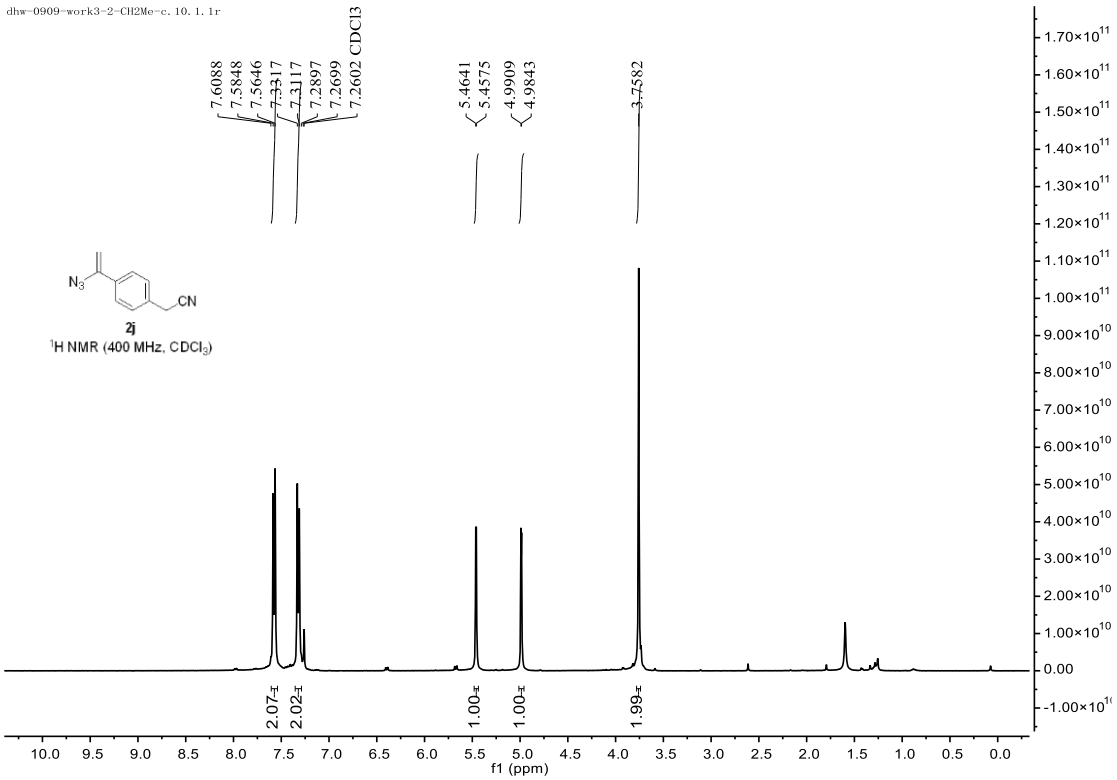
7.4927
7.4891
7.4863
7.4820
7.3419
7.3376
7.3347
7.3311
7.3267
7.3231
7.3203
7.3160
7.2603
5.4396
5.4362
5.4333
4.9801
4.9768
4.9734
N3
Cl
2h
1H NMR (600 MHz, CDCl3)
1.98
1.93
1.00
1.04
f1 (ppm)



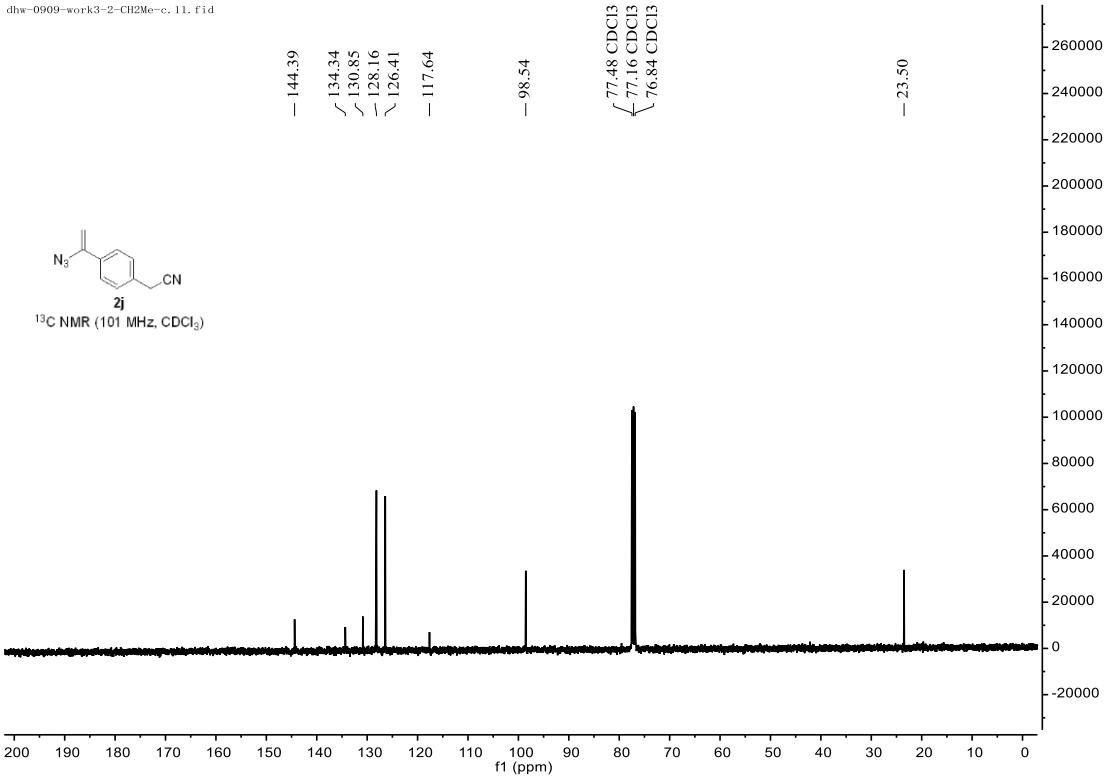
2i

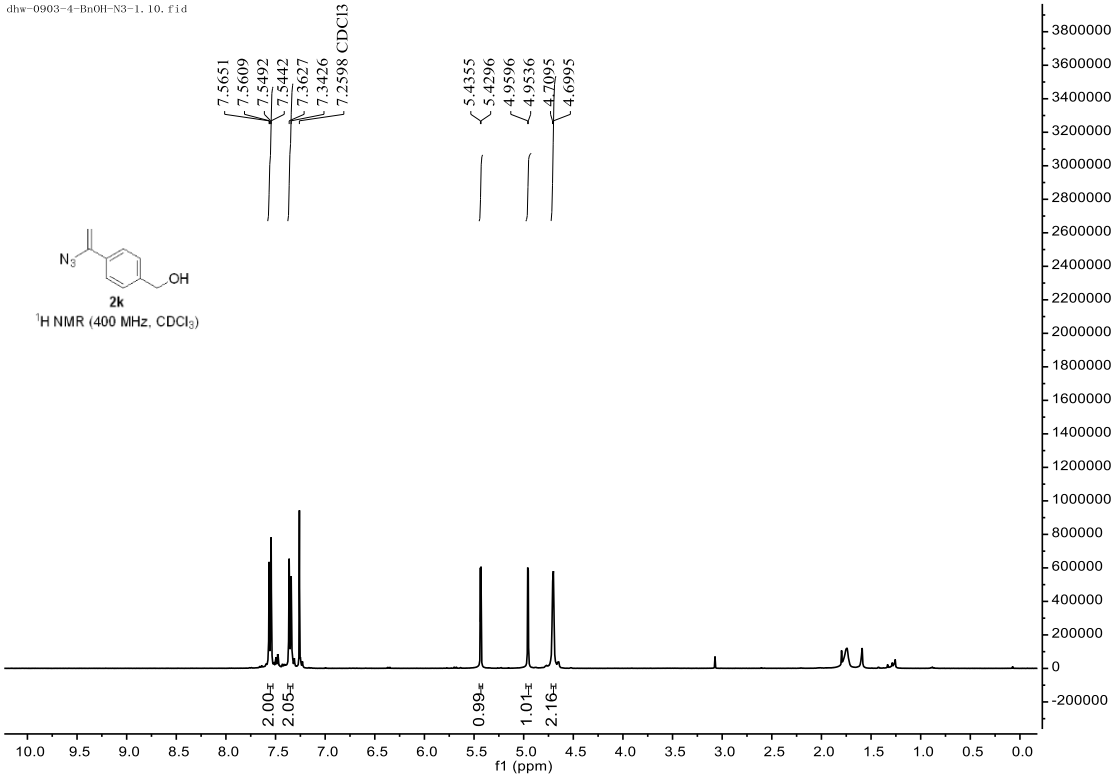
^{1}H NMR (400 MHz, $CDCl_3$)



2j

^{1}H NMR (400 MHz, $CDCl_3$)


dhw-0909-work3-2-CH2Me-c.11.fid
144.39
134.34
130.85
128.16
126.41
117.64
98.54
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
23.50
N3
CN
2j
13C NMR (101 MHz, CDCl3)
f1 (ppm)


dhw-0903-4-BnOH-N3-1.10.fid
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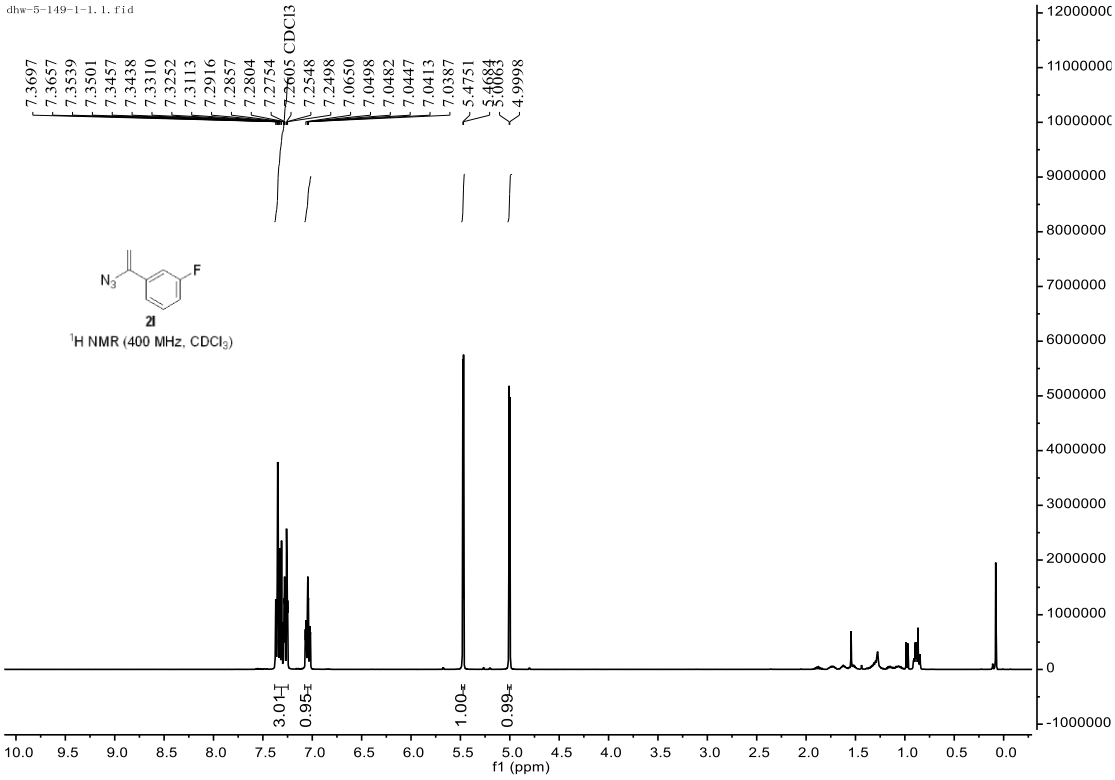
7.5492
7.5442
7.3627
7.3426
7.2598 CDCl3
5.4355
5.4296
4.9596
4.9536
4.7095
4.6995
N3
OH
2k
1H NMR (400 MHz, CDCl3)
2.00
2.05
0.99
1.01
2.16
f1 (ppm)


dhw-5-149-1-1.1.fid
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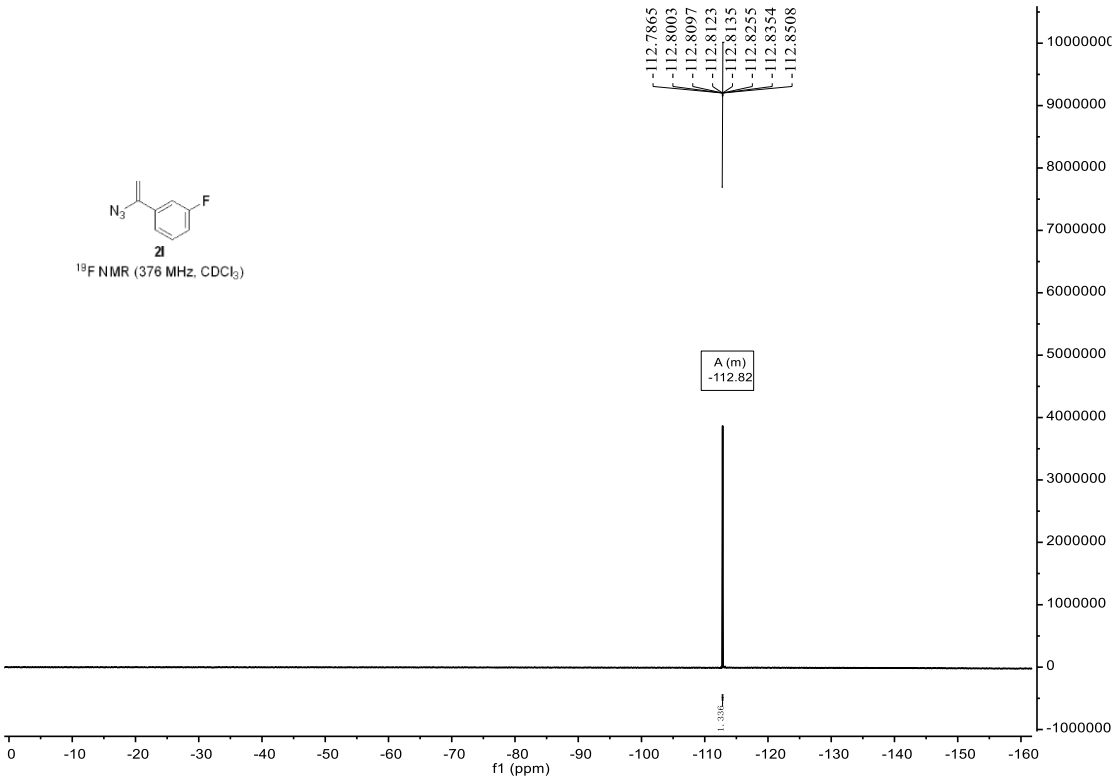
7.3657
7.3539
7.3501
7.3457
7.3438
7.3310
7.3252
7.3113
7.2916
7.2857
7.2804
7.2754
7.2605 CDCl3
7.2548
7.2498
7.0650
7.0498
7.0482
7.0447
7.0413
7.0387
5.4751
5.4684
5.0063
4.9998
N3
F
2l
1H NMR (400 MHz, CDCl3)
3.01
0.95
1.00
0.99
f1 (ppm)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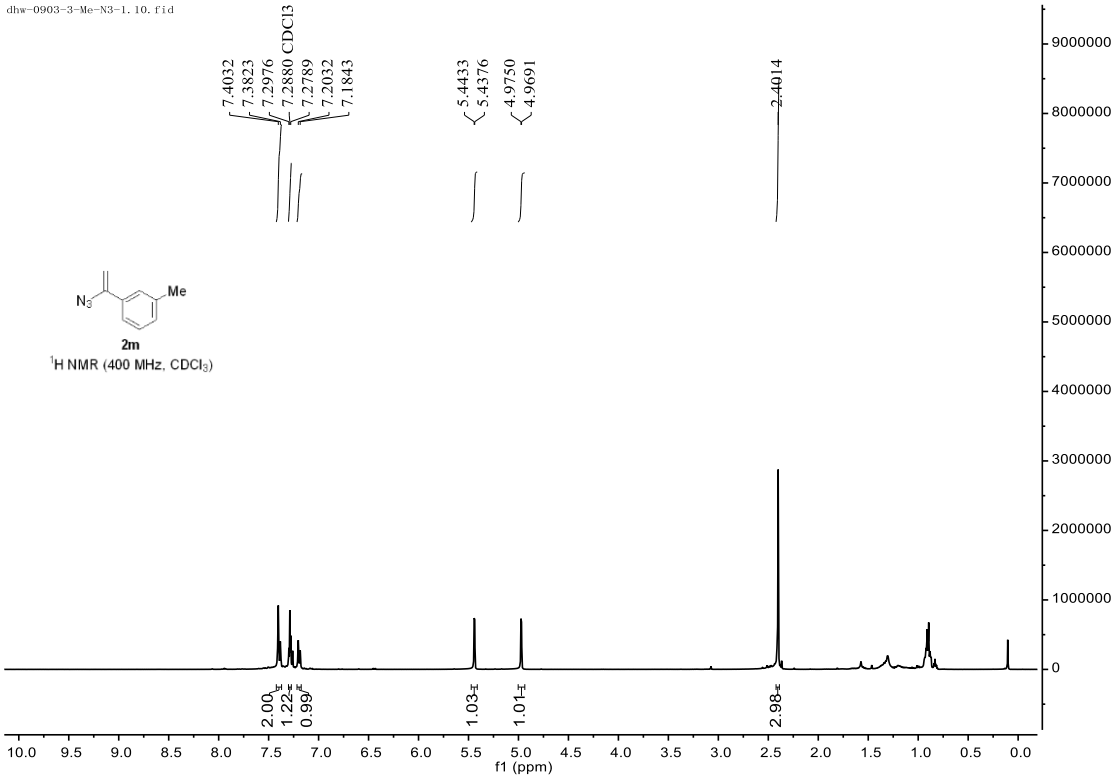
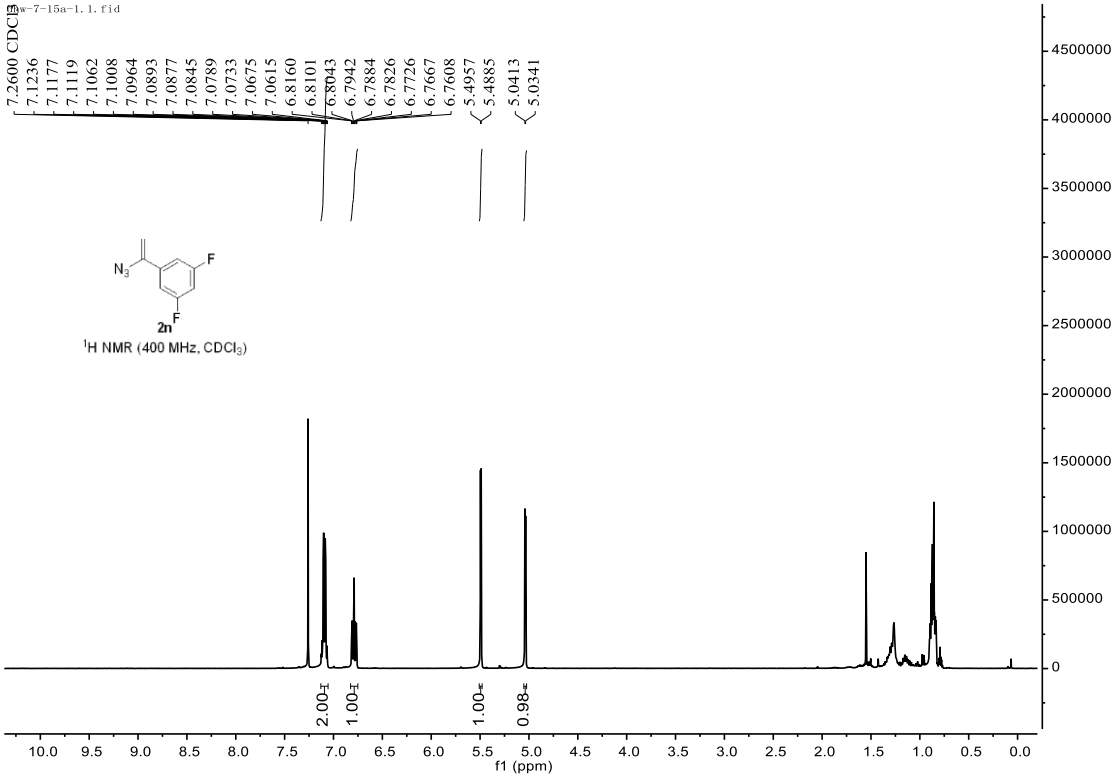
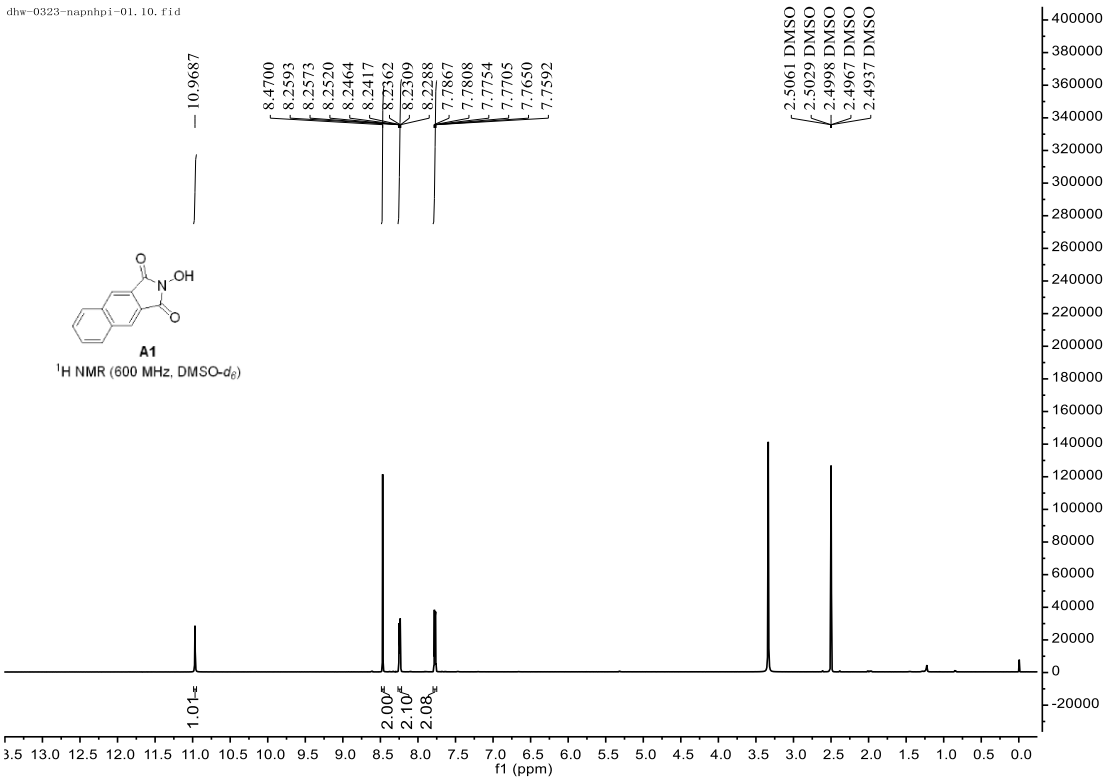
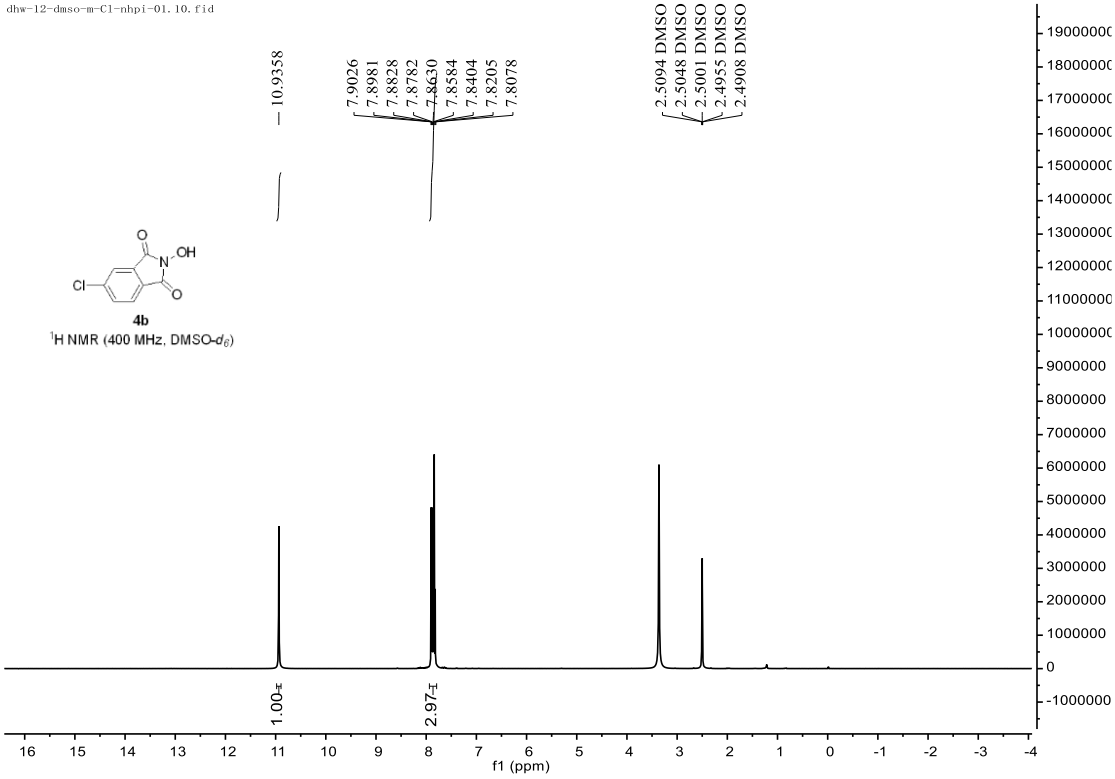
-112.8003
-112.8097
-112.8123
-112.8135
-112.8255
-112.8354
-112.8508
N3
F
2l
19F NMR (376 MHz, CDCl3)
A (m)
-112.82
1.336
f1 (ppm)


dhw-0903-3-Me-N3-1.10.fid
7.4032
7.3823
7.2976
7.2880 CDCl3
7.2789
7.2032
7.1843
5.4433
5.4376
4.9750
4.9691
2.4014
Me
N3
2m
1H NMR (400 MHz, CDCl3)
2.00
1.22
0.99
1.03
1.01
2.98
f1 (ppm)


dhw-7-15a-1.1.fid
7.2600 CDCl3
7.1236
7.1177
7.1119
7.1062
7.1008
7.0964
7.0893
7.0877
7.0845
7.0789
7.0733
7.0675
7.0615
6.8160
6.8101
6.8043
6.7942
6.7884
6.7826
6.7726
6.7667
6.7608
5.4957
5.4885
5.0413
5.0341
N3
F
F
2n
1H NMR (400 MHz, CDCl3)
2.00
1.00
1.00
0.98
f1 (ppm)


dhw-0323-napnhpi-01.10.fid
10.9687
8.4700
8.2593
8.2573
8.2520
8.2464
8.2417
8.2362
8.2309
8.2288
7.7867
7.7808
7.7754
7.7705
7.7650
7.7592
2.5061 DMSO
2.5029 DMSO
2.4998 DMSO
2.4967 DMSO
2.4937 DMSO
A1
1H NMR (600 MHz, DMSO-d6)
1.01
2.00
2.10
2.08
f1 (ppm)


dhw-12-dmso-m-Cl-nhpi-01.10.fid
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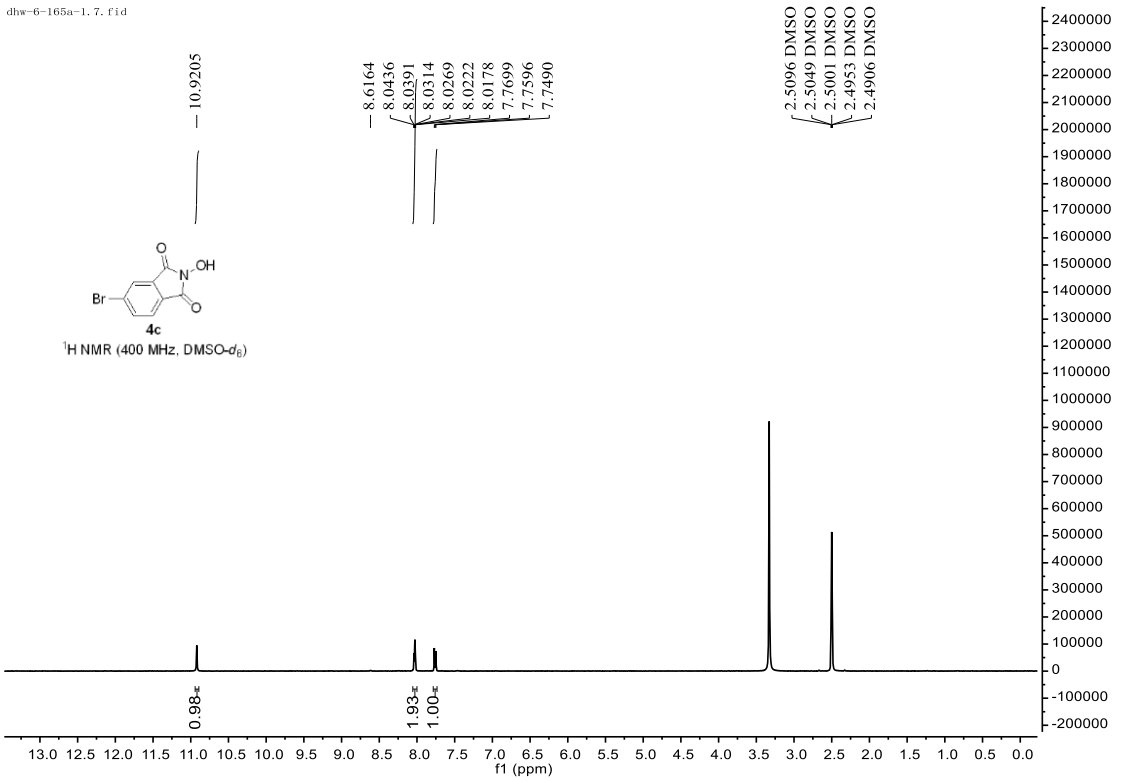
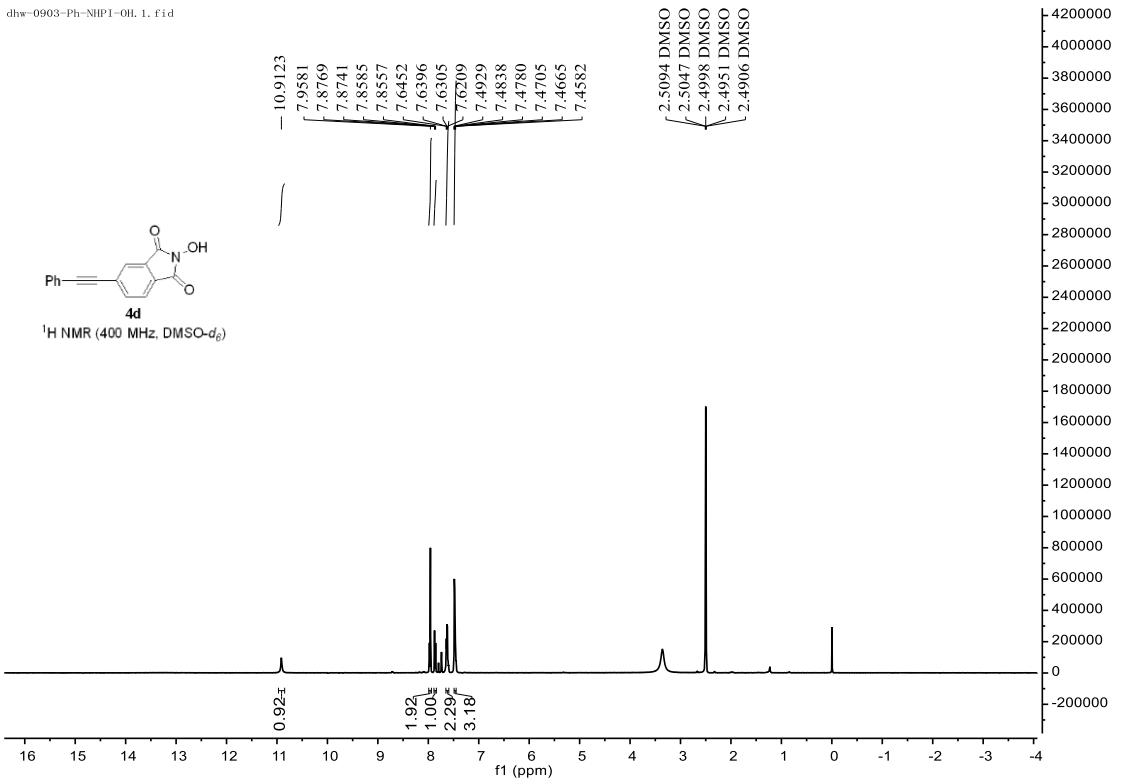
7.9026
7.8981
7.8828
7.8782
7.8630
7.8584
7.8404
7.8205
7.8078
2.5094 DMSO
2.5048 DMSO
2.5001 DMSO
2.4955 DMSO
2.4908 DMSO
4b
1H NMR (400 MHz, DMSO-d6)
1.00
2.97
f1 (ppm)



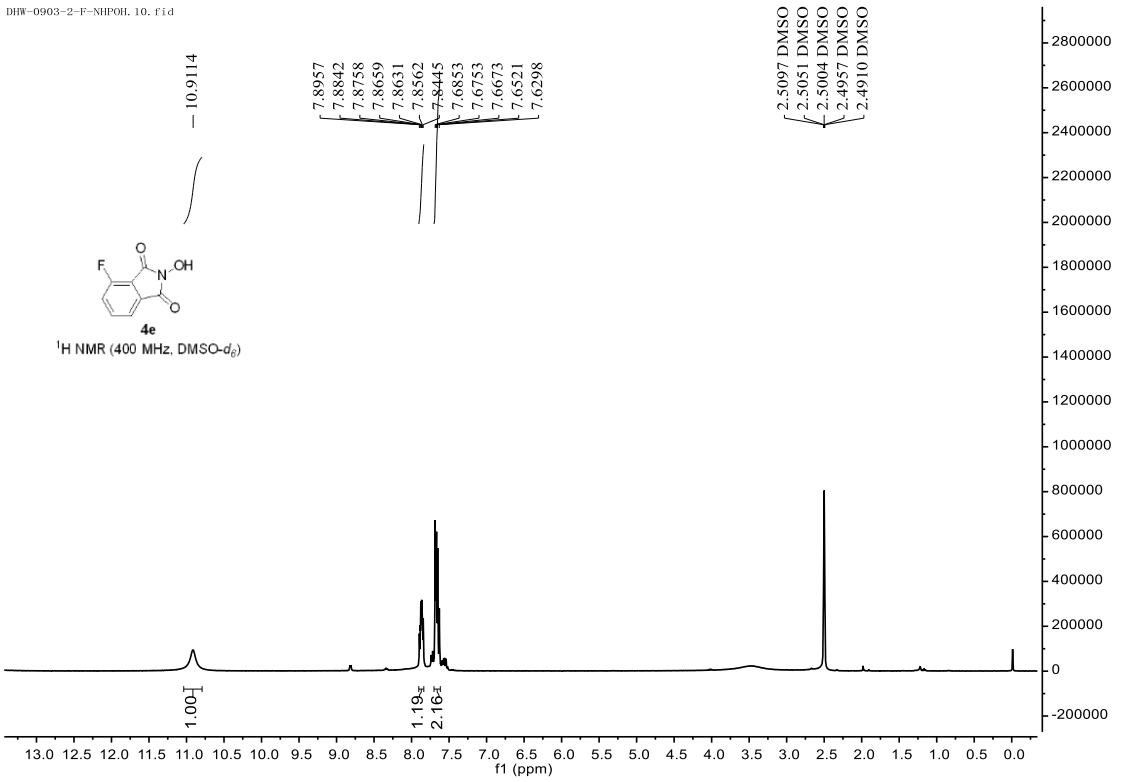
4c

^{1}H NMR (400 MHz, DMSO-d_6)



4d

^{1}H NMR (400 MHz, DMSO-d_6)


DHW-0903-2-F-NHPOH.10.fid
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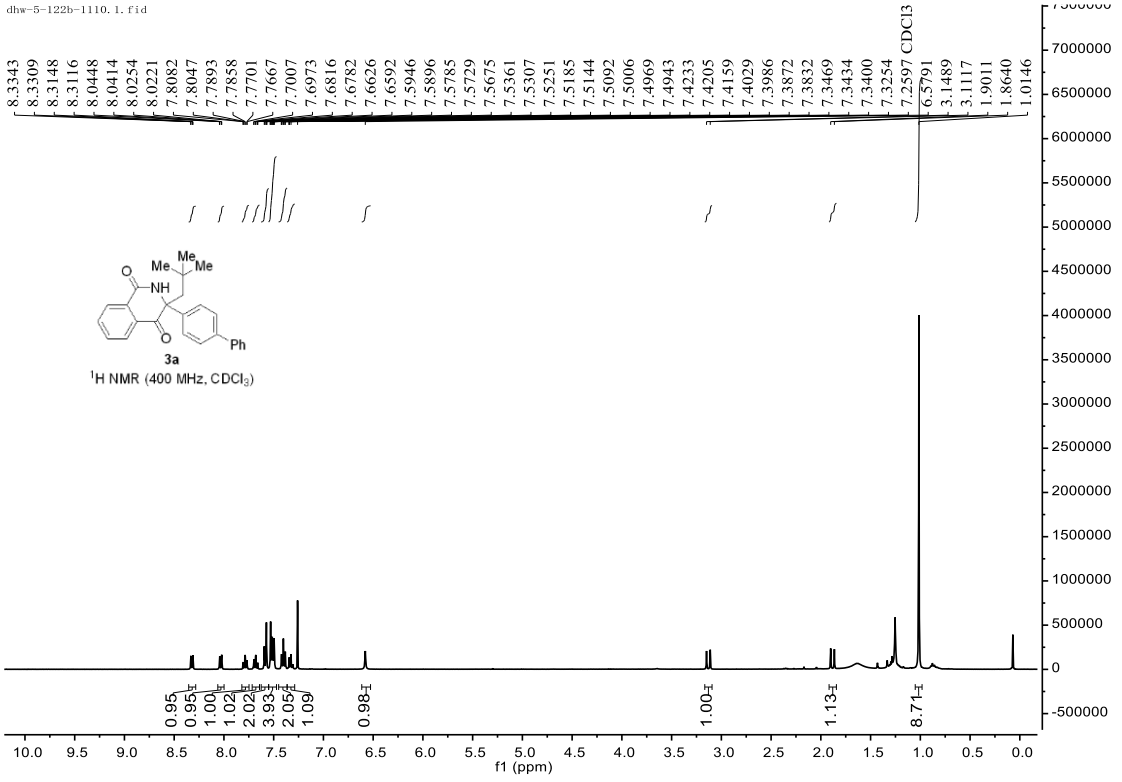
7.8957
7.8842
7.8758
7.8659
7.8631
7.8562
7.8445
7.6853
7.6753
7.6673
7.6521
7.6298
2.5097 DMSO
2.5051 DMSO
2.5004 DMSO
2.4957 DMSO
2.4910 DMSO
F
N
OH
O
O
4e
1H NMR (400 MHz, DMSO-d6)
1.00
1.19
2.16
f1 (ppm)


dhw-5-122b-1110.1.fid
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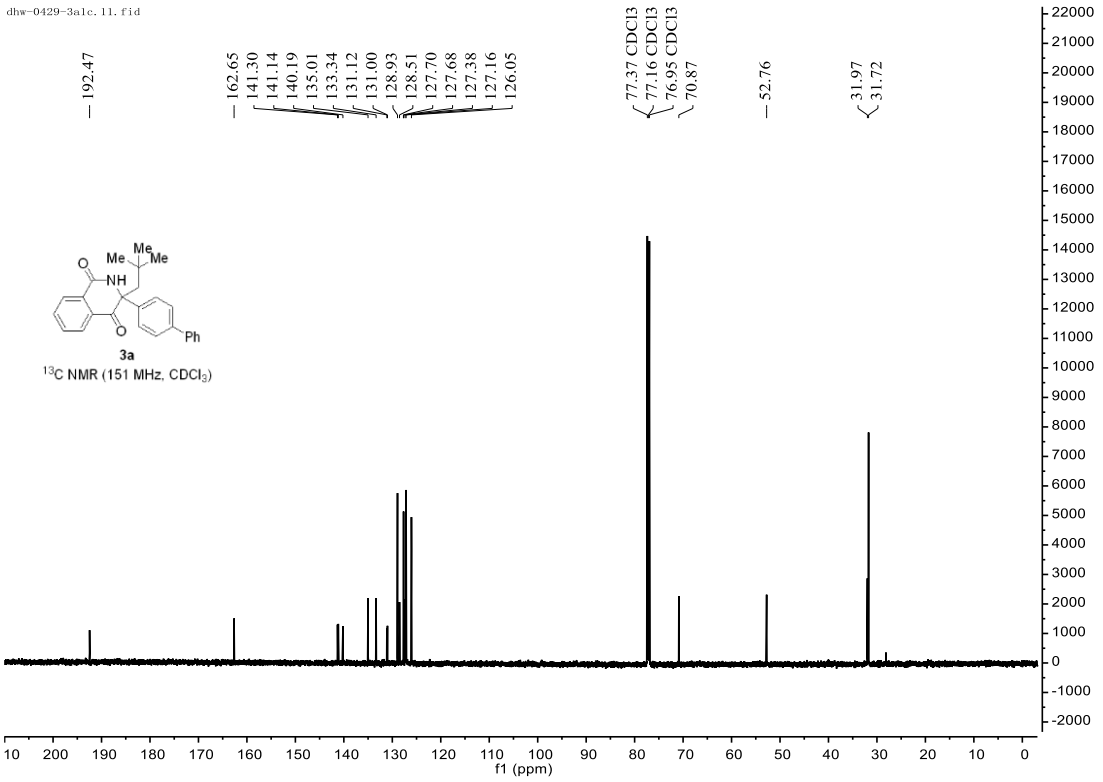
8.3309
8.3148
8.3116
8.0448
8.0414
8.0254
8.0221
7.8082
7.8047
7.7893
7.7858
7.7701
7.7667
7.7007
7.6973
7.6816
7.6782
7.6626
7.6592
7.5946
7.5896
7.5785
7.5729
7.5675
7.5361
7.5307
7.5251
7.5185
7.5144
7.5092
7.5006
7.4969
7.4943
7.4233
7.4205
7.4159
7.4029
7.3986
7.3872
7.3832
7.3469
7.3434
7.3400
7.3254
7.2597 CDCl3
6.5791
3.1489
3.1117
1.9011
1.8640
1.0146
Me
Me
Me
NH
O
O
Ph
3a
1H NMR (400 MHz, CDCl3)
0.95
0.95
1.00
1.02
2.02
3.93
2.05
1.09
0.98
1.00
1.13
8.71
f1 (ppm)


dhw-0429-3a1c.11.fid
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162.65
141.30
141.14
140.19
135.01
133.34
131.12
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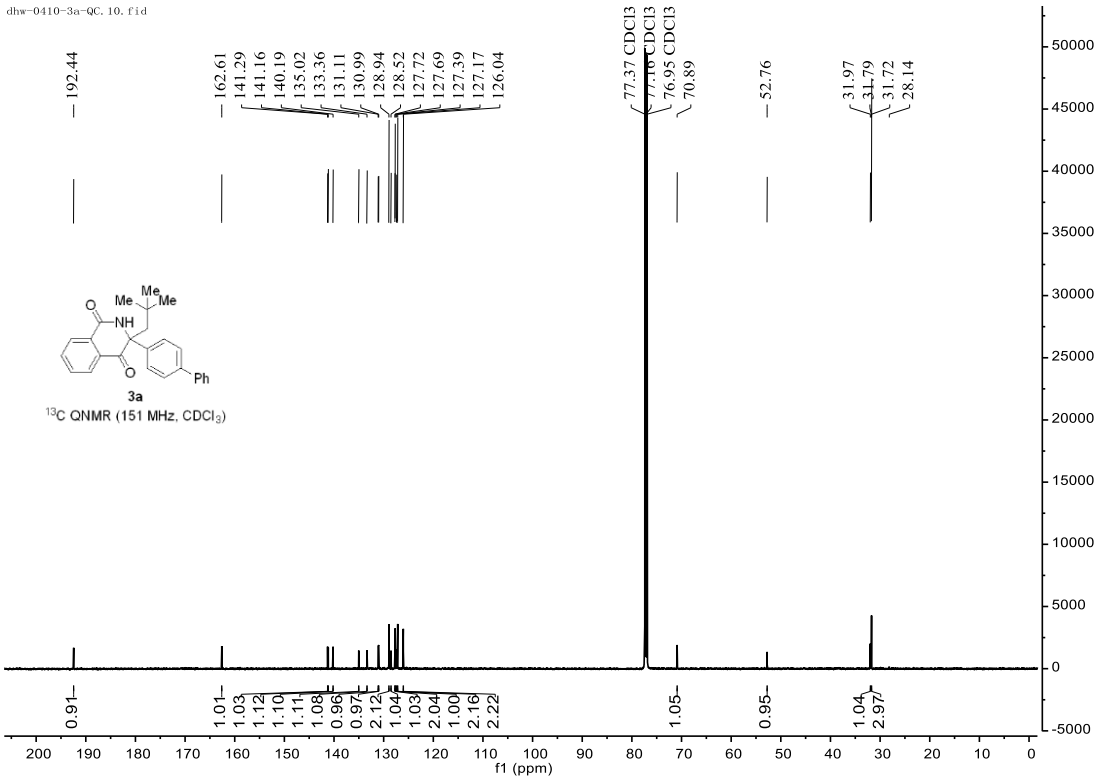
128.93
128.51
127.70
127.68
127.38
127.16
126.05
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.87
52.76
31.97
31.72
Me
Me
Me
O
NH
O
Ph
3a
13C NMR (151 MHz, CDCl3)
f1 (ppm)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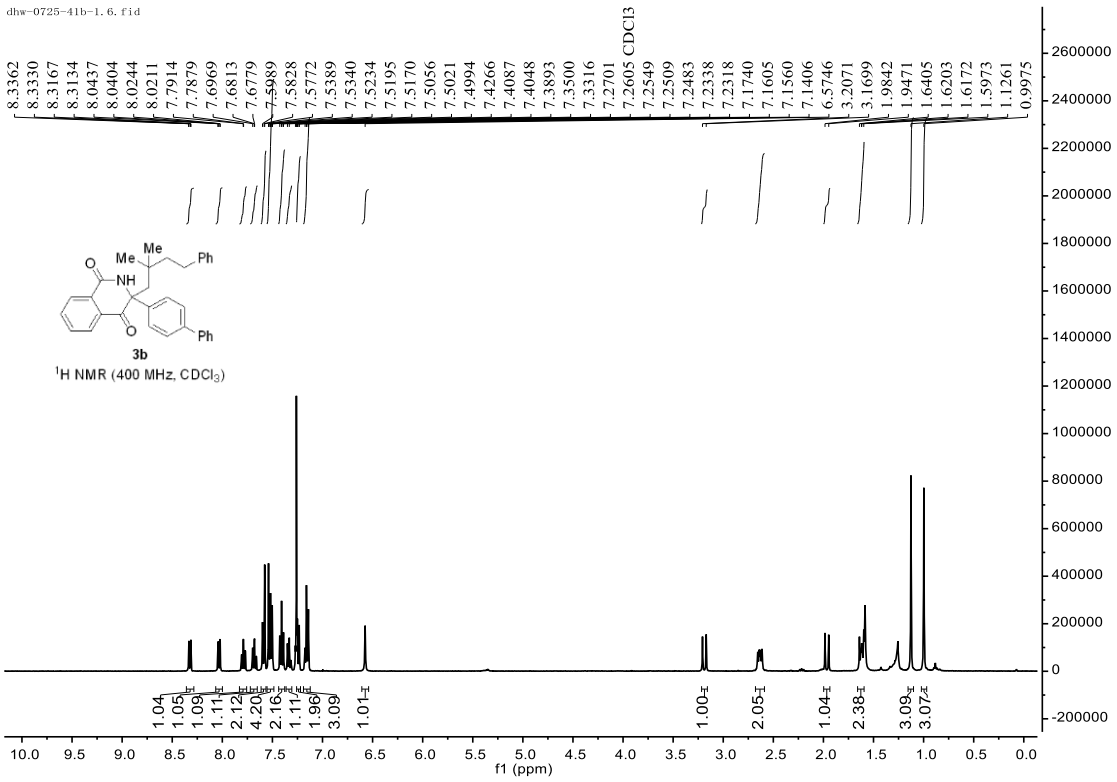
141.29
141.16
140.19
135.02
133.36
131.11
130.99
128.94
128.52
127.72
127.69
127.39
127.17
126.04
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.89
52.76
31.97
31.79
31.72
28.14
0.91
1.01
1.03
1.12
1.10
1.11
1.08
0.96
0.97
2.12
1.04
1.03
2.04
1.00
2.16
2.22
1.05
0.95
1.04
2.97
Me
Me
Me
O
NH
O
Ph
3a
13C QNMR (151 MHz, CDCl3)
f1 (ppm)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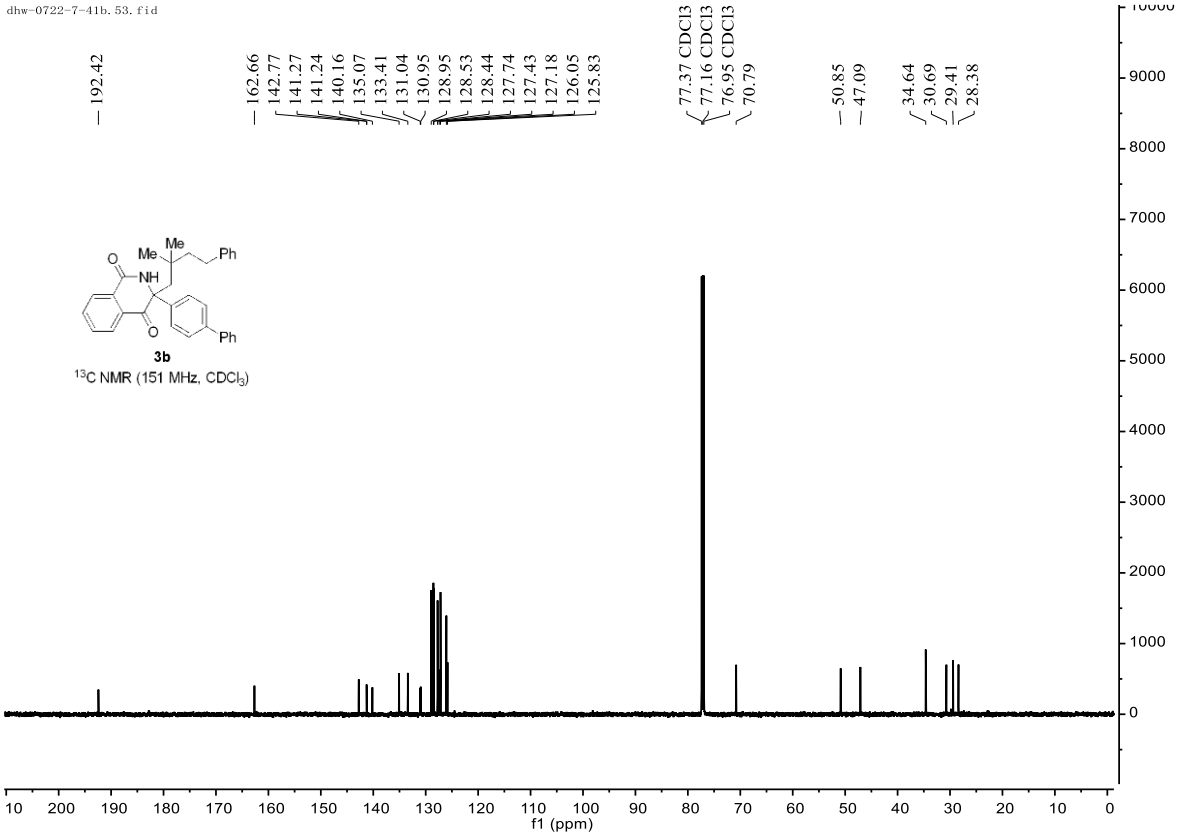
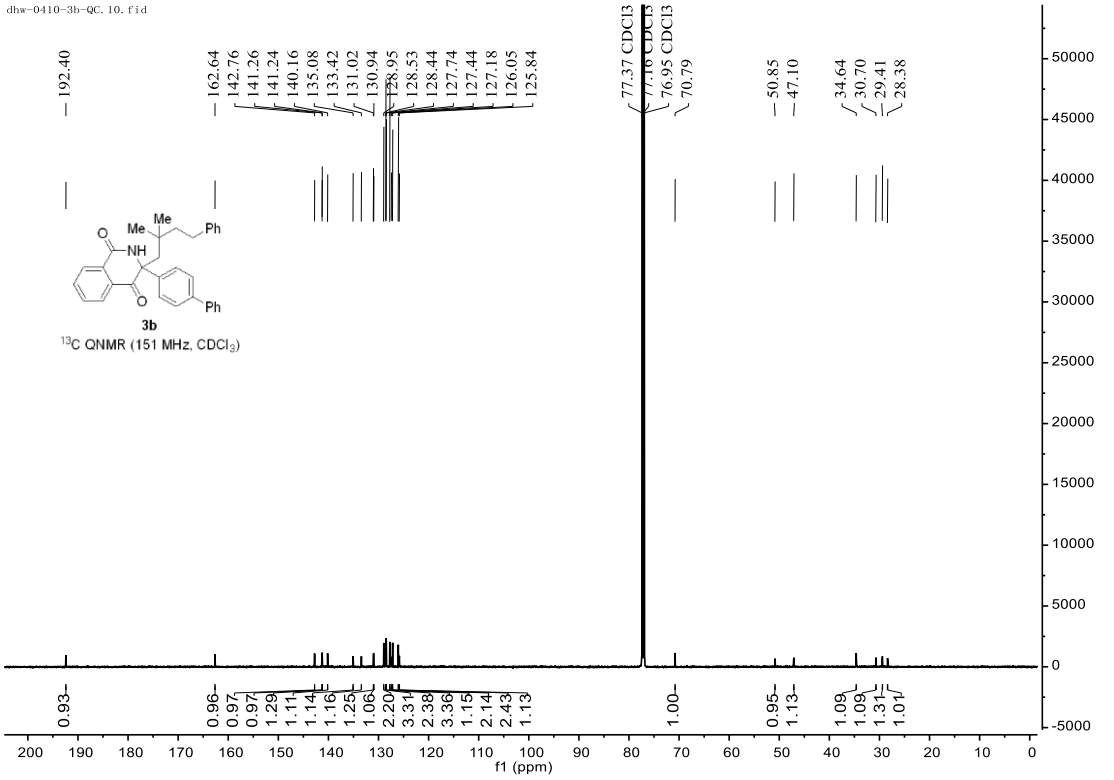
8.3330
8.3167
8.3134
8.0437
8.0404
8.0244
8.0211
7.7914
7.7879
7.6969
7.6813
7.6779
7.5989
7.5828
7.5772
7.5389
7.5340
7.5234
7.5195
7.5170
7.5056
7.5021
7.4994
7.4266
7.4087
7.4048
7.3893
7.3500
7.3316
7.2701
7.2605 CDCl3
7.2549
7.2509
7.2483
7.2338
7.2318
7.1740
7.1605
7.1560
7.1406
6.5746
3.2071
3.1699
1.9842
1.9471
1.6405
1.6203
1.6172
1.5973
1.1261
0.9975
Me
Ph
NH
O
Ph
3b
1H NMR (400 MHz, CDCl3)
1.04
1.05
1.09
1.11
2.12
4.20
2.16
1.11
1.96
3.09
1.01
1.00
2.05
1.04
2.38
3.09
3.07
f1 (ppm)


dhw-0722-7-41b.53.fid
192.42
162.66
142.77
141.27
141.24
140.16
135.07
133.41
131.04
130.95
128.95
128.53
128.44
127.74
127.43
127.18
126.05
125.83
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.79
50.85
47.09
34.64
30.69
29.41
28.38
Me
Ph
NH
O
Ph
3b
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0410-3b-QC.10.fid
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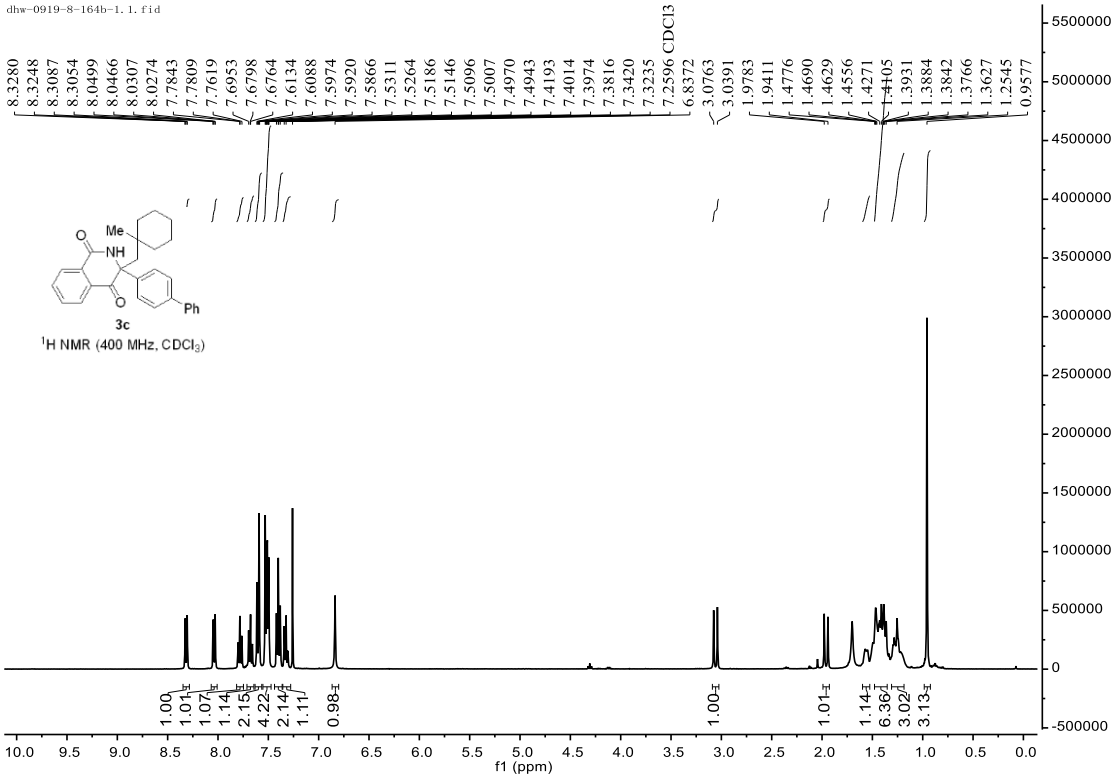
142.76
141.26
141.24
140.16
135.08
133.42
131.02
130.94
128.95
128.53
128.44
127.74
127.44
127.18
126.05
125.84
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.79
50.85
47.10
34.64
30.70
29.41
28.38
0.93
0.96
0.97
0.97
1.29
1.11
1.14
1.16
1.25
1.06
2.20
3.31
2.38
3.36
1.15
2.14
2.43
1.13
1.00
0.95
1.13
1.09
1.09
1.31
1.01
f1 (ppm)
Me
Me
Ph
NH
O
O
Ph
3b
13C QNMR (151 MHz, CDCl3)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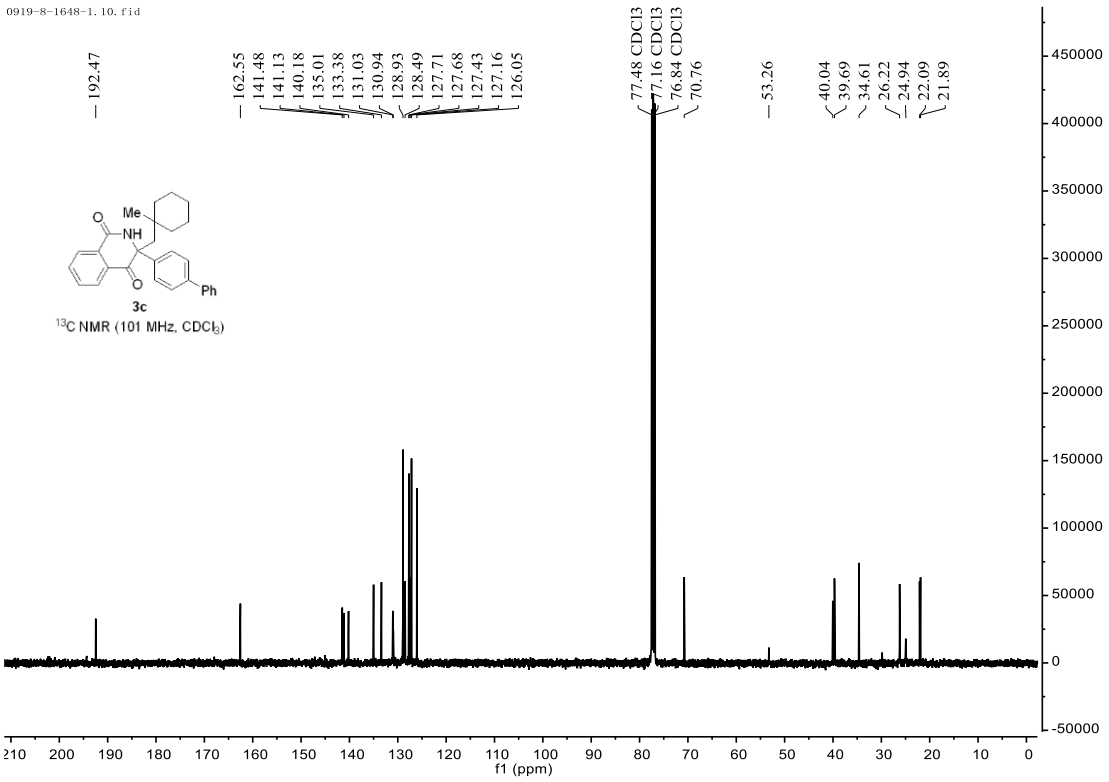
8.3248
8.3087
8.3054
8.0499
8.0466
8.0307
8.0274
7.7843
7.7809
7.7619
7.6953
7.6798
7.6764
7.6134
7.6088
7.5974
7.5920
7.5866
7.5311
7.5264
7.5186
7.5146
7.5096
7.5007
7.4970
7.4943
7.4193
7.4014
7.3974
7.3816
7.3420
7.3235
7.2596 CDCl3
6.8372
3.0763
3.0391
1.9783
1.9411
1.4776
1.4690
1.4629
1.4556
1.4271
1.4105
1.3931
1.3884
1.3842
1.3766
1.3627
1.2545
0.9577
1.00
1.01
1.07
1.14
2.15
4.22
2.14
1.11
0.98
1.00
1.01
1.14
6.36
3.02
3.13
f1 (ppm)
Me
NH
O
O
Ph
3c
1H NMR (400 MHz, CDCl3)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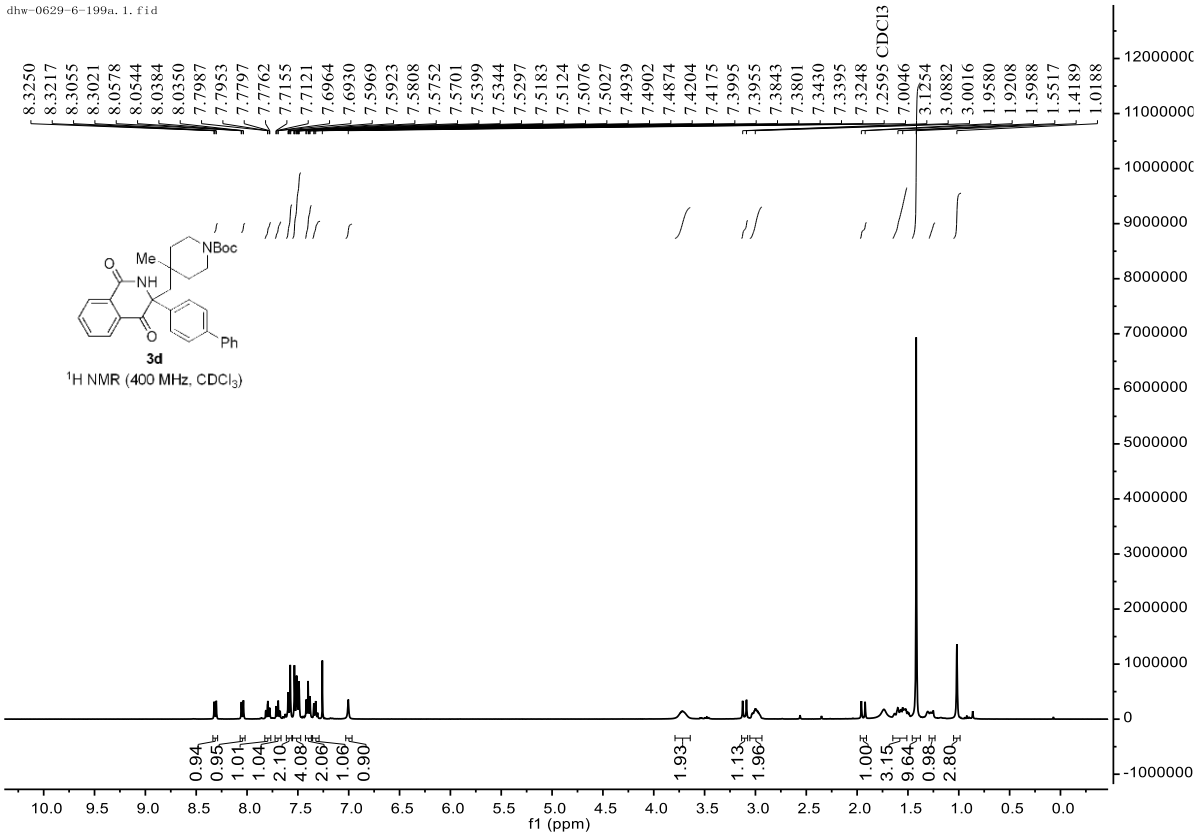
162.55
141.48
141.13
140.18
135.01
133.38
131.03
130.94
128.93
128.49
127.71
127.68
127.43
127.16
126.05
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.76
53.26
40.04
39.69
34.61
26.22
24.94
22.09
21.89
Me
NH
O
O
Ph
3c
13C NMR (101 MHz, CDCl3)
f1 (ppm)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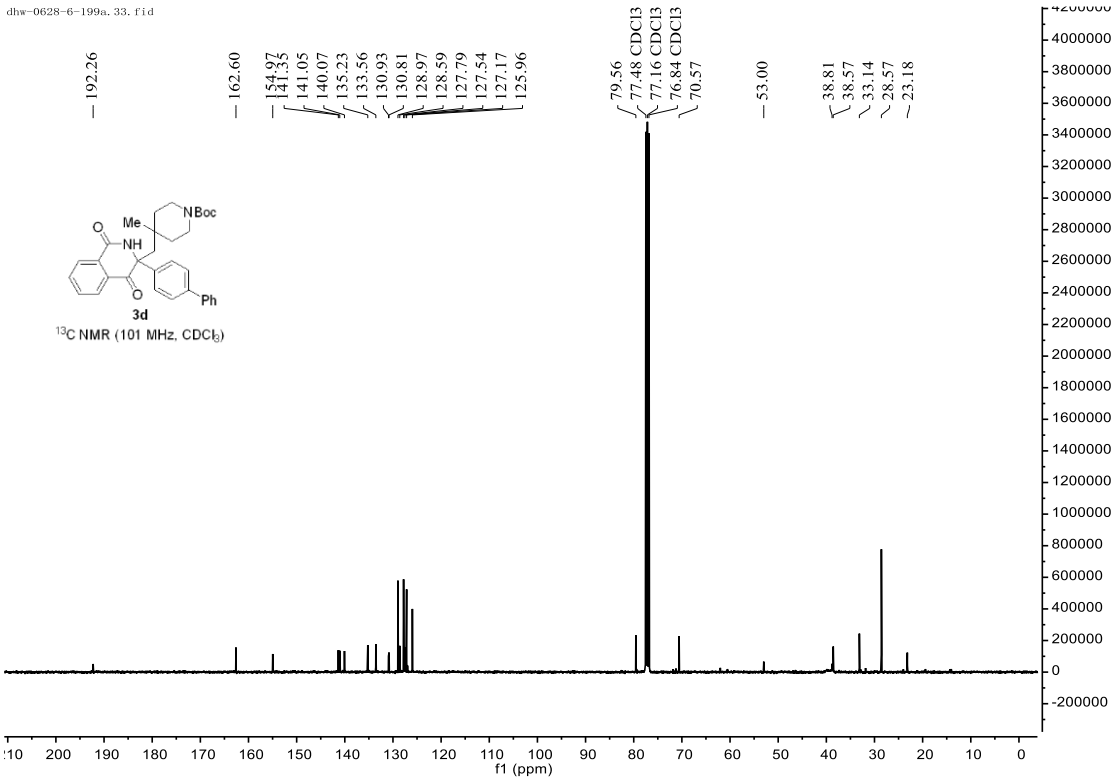
8.3021
8.0578
8.0544
8.0384
8.0350
7.7987
7.7953
7.7797
7.7762
7.7155
7.7121
7.6964
7.6930
7.5969
7.5923
7.5808
7.5752
7.5701
7.5399
7.5344
7.5297
7.5183
7.5124
7.5076
7.5027
7.4939
7.4902
7.4874
7.4204
7.4175
7.3995
7.3955
7.3843
7.3801
7.3430
7.3395
7.3248
7.2595 CDCl3
7.0046
3.1254
3.0882
3.0016
1.9580
1.9208
1.5988
1.5517
1.4189
1.0188
NBoc
Me
NH
O
O
Ph
3d
1H NMR (400 MHz, CDCl3)
0.94
0.95
1.01
1.04
2.10
4.08
2.06
1.06
0.90
1.93
1.13
1.96
1.00
3.15
9.64
0.98
2.80
f1 (ppm)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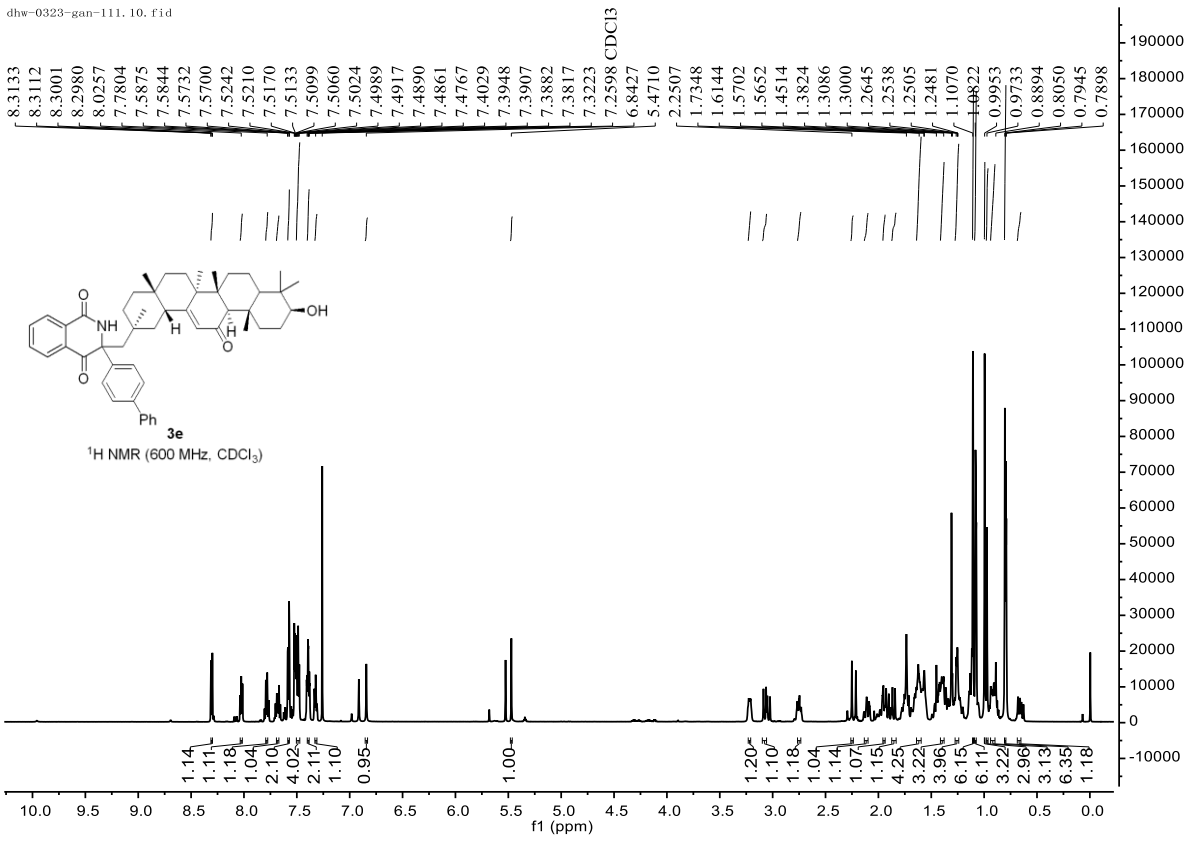
154.97
141.35
141.05
140.07
135.23
133.56
130.93
130.81
128.97
128.59
127.79
127.54
127.17
125.96
79.56
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.57
53.00
38.81
38.57
33.14
28.57
23.18
NBoc
Me
NH
O
O
Ph
3d
13C NMR (101 MHz, CDCl3)
f1 (ppm)


dhw-0323-gan-111.10.fid
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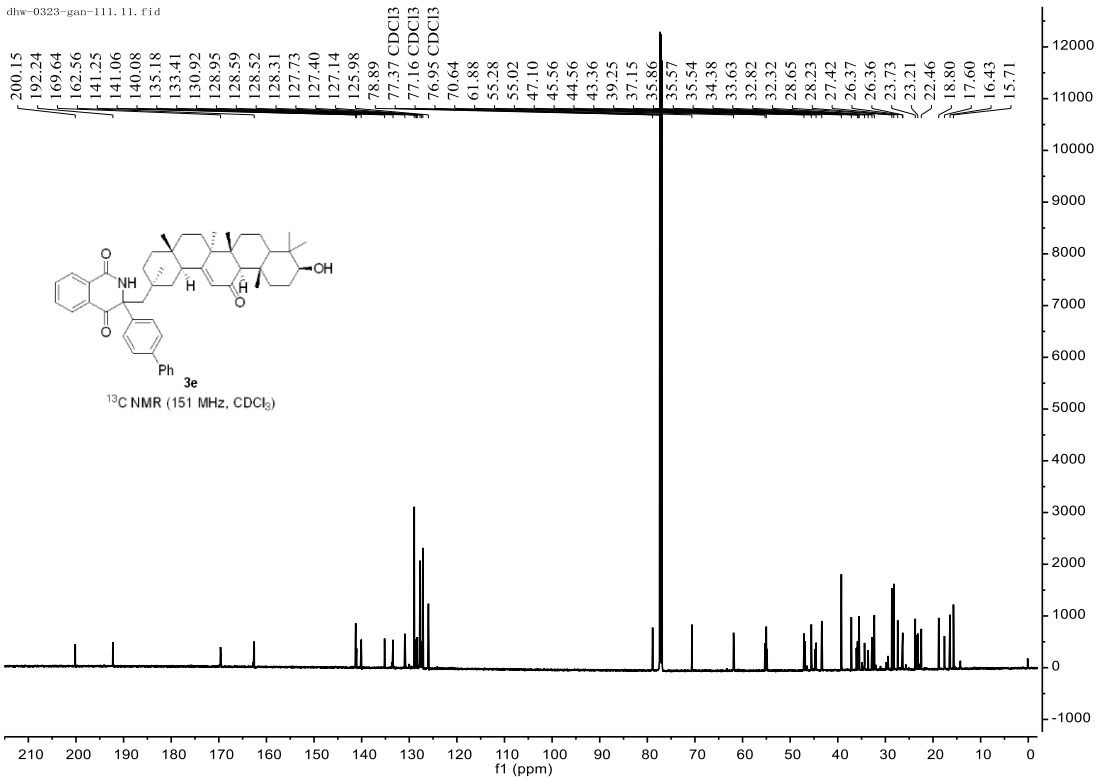
8.3112
8.3001
8.2980
8.0257
7.7804
7.5875
7.5844
7.5732
7.5700
7.5242
7.5210
7.5170
7.5133
7.5099
7.5060
7.5024
7.4989
7.4917
7.4890
7.4861
7.4767
7.4029
7.3948
7.3907
7.3882
7.3817
7.3223
7.2598 CDCl3
6.8427
5.4710
2.2507
1.7348
1.6144
1.5702
1.5652
1.4514
1.3824
1.3086
1.3000
1.2645
1.2538
1.2505
1.2481
1.1070
0.9953
0.9733
0.8894
0.8050
0.7945
0.7898
NH
H
H
O
O
OH
Ph
3e
1H NMR (600 MHz, CDCl3)
1.14
1.11
1.18
1.04
2.10
4.02
2.11
1.10
0.95
1.00
1.20
1.10
1.18
1.04
1.14
1.07
1.15
4.25
3.22
3.96
6.15
6.11
3.22
2.96
3.13
6.35
1.18
f1 (ppm)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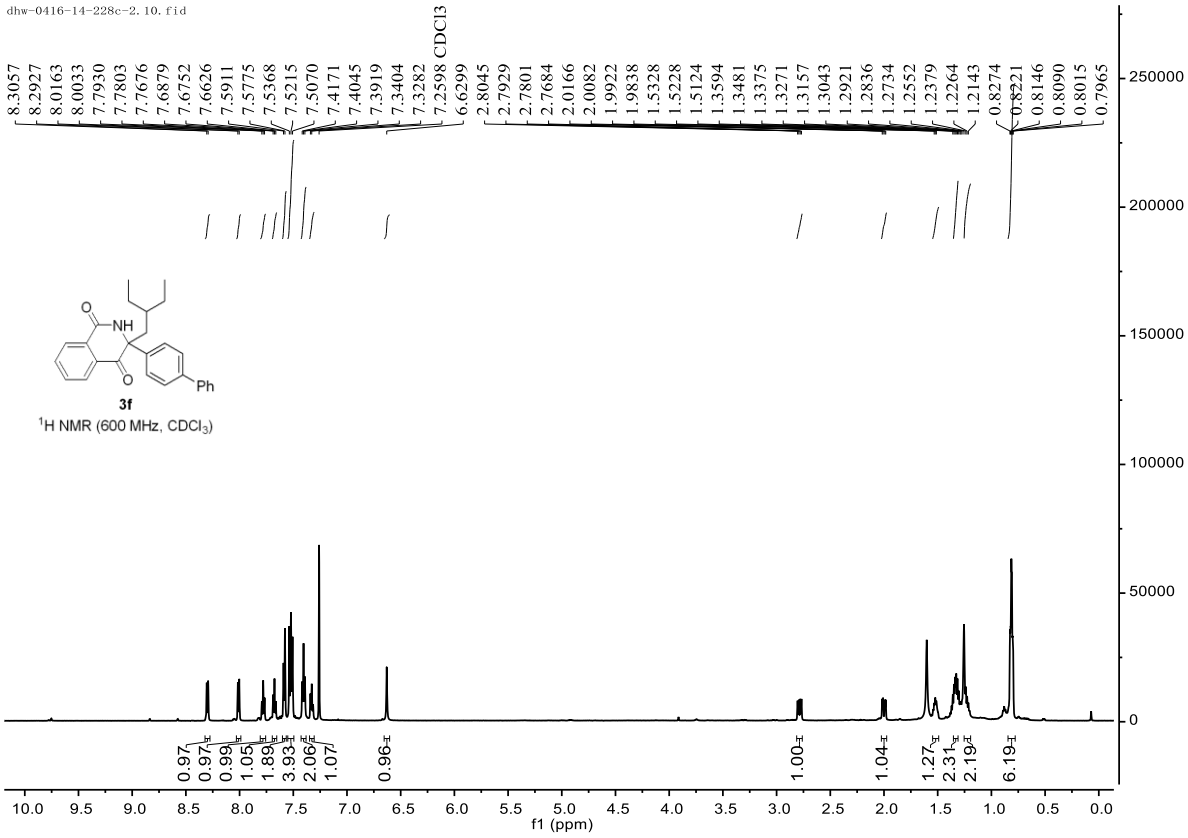
192.24
169.64
162.56
141.25
141.06
140.08
135.18
133.41
130.92
128.95
128.59
128.52
128.31
127.73
127.40
127.14
125.98
78.89
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.64
61.88
55.28
55.02
47.10
45.56
44.56
43.36
39.25
37.15
35.86
35.57
35.54
34.38
33.63
32.82
32.32
28.65
28.23
27.42
26.37
26.36
23.73
23.21
22.46
18.80
17.60
16.43
15.71
OH
NH
O
Ph
3e
13C NMR (151 MHz, CDCl3)
f1 (ppm)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8.2927
8.0163
8.0033
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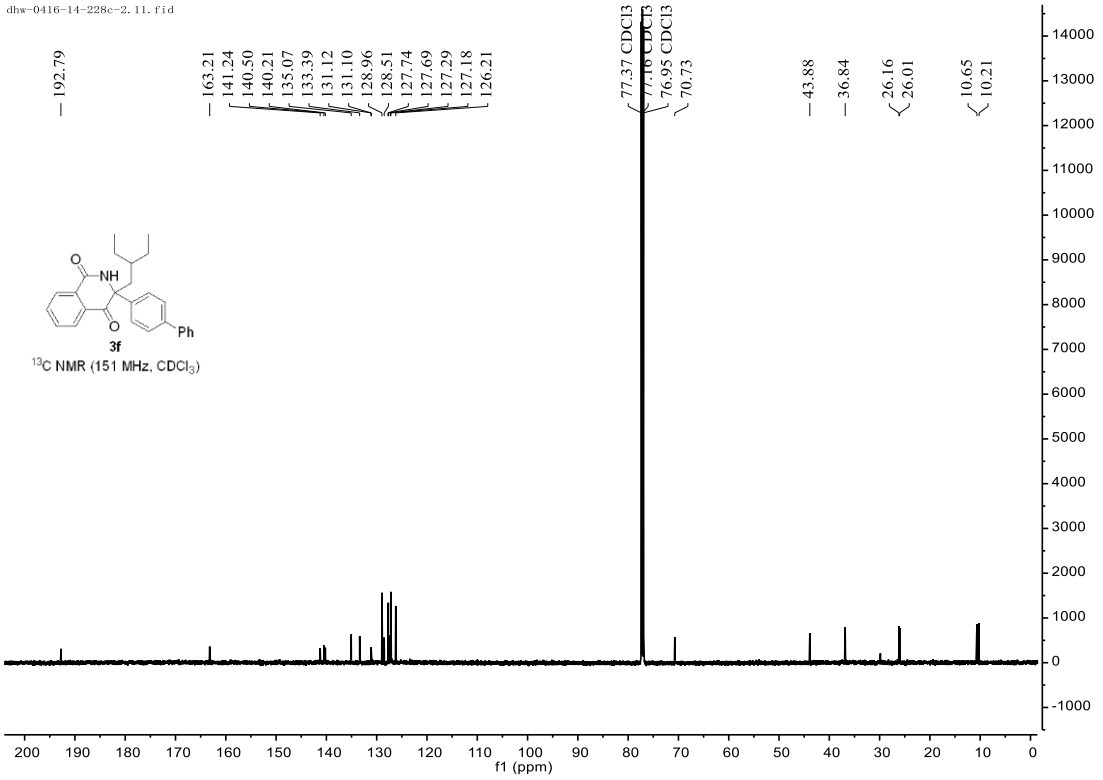
7.7803
7.7676
7.6879
7.6752
7.6626
7.5911
7.5775
7.5368
7.5215
7.5070
7.4171
7.4045
7.3919
7.3404
7.3282
7.2598 CDCl3
6.6299
2.8045
2.7929
2.7801
2.7684
2.0166
2.0082
1.9922
1.9838
1.5328
1.5228
1.5124
1.3594
1.3481
1.3375
1.3271
1.3157
1.3043
1.2921
1.2836
1.2734
1.2552
1.2379
1.2264
1.2143
0.8274
0.8221
0.8146
0.8090
0.8015
0.7965
NH
O
Ph
3f
1H NMR (600 MHz, CDCl3)
0.97
0.97
0.99
1.05
1.89
3.93
2.06
1.07
0.96
1.00
1.04
1.27
2.31
2.19
6.19
f1 (ppm)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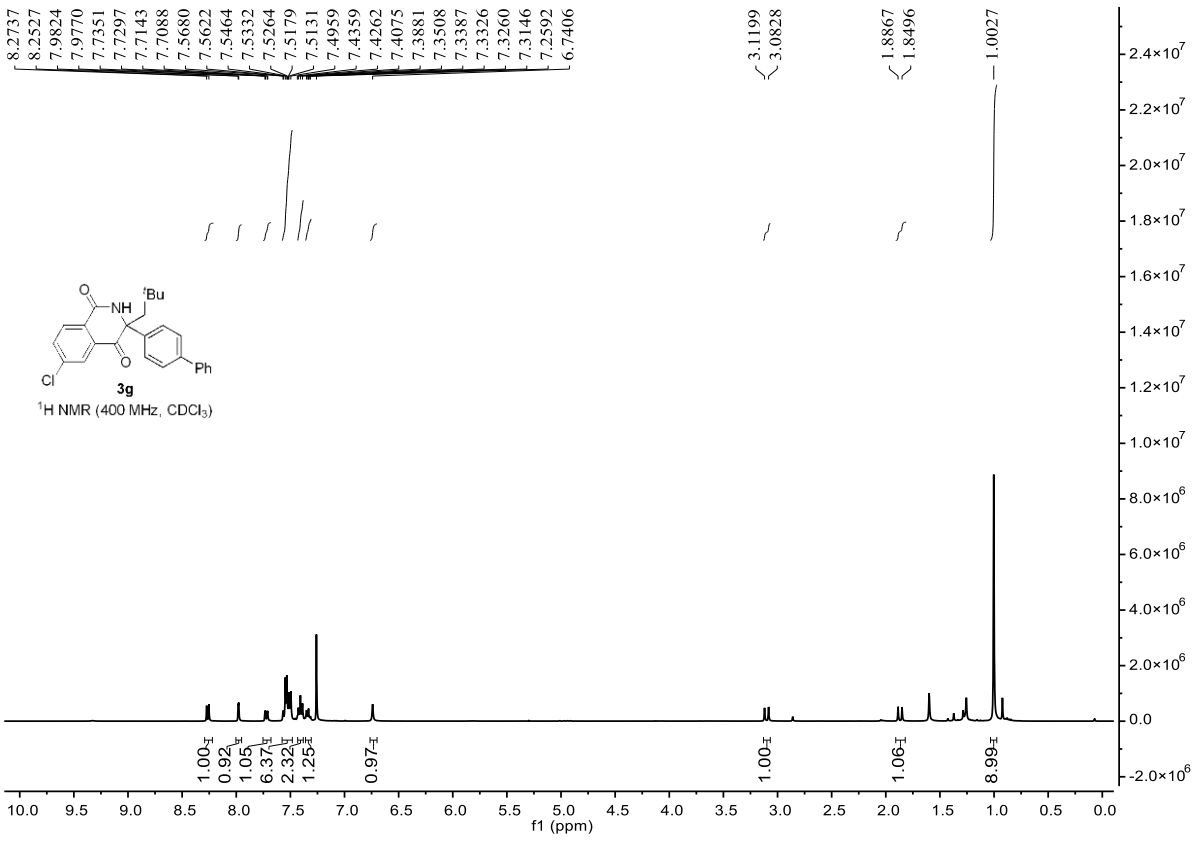
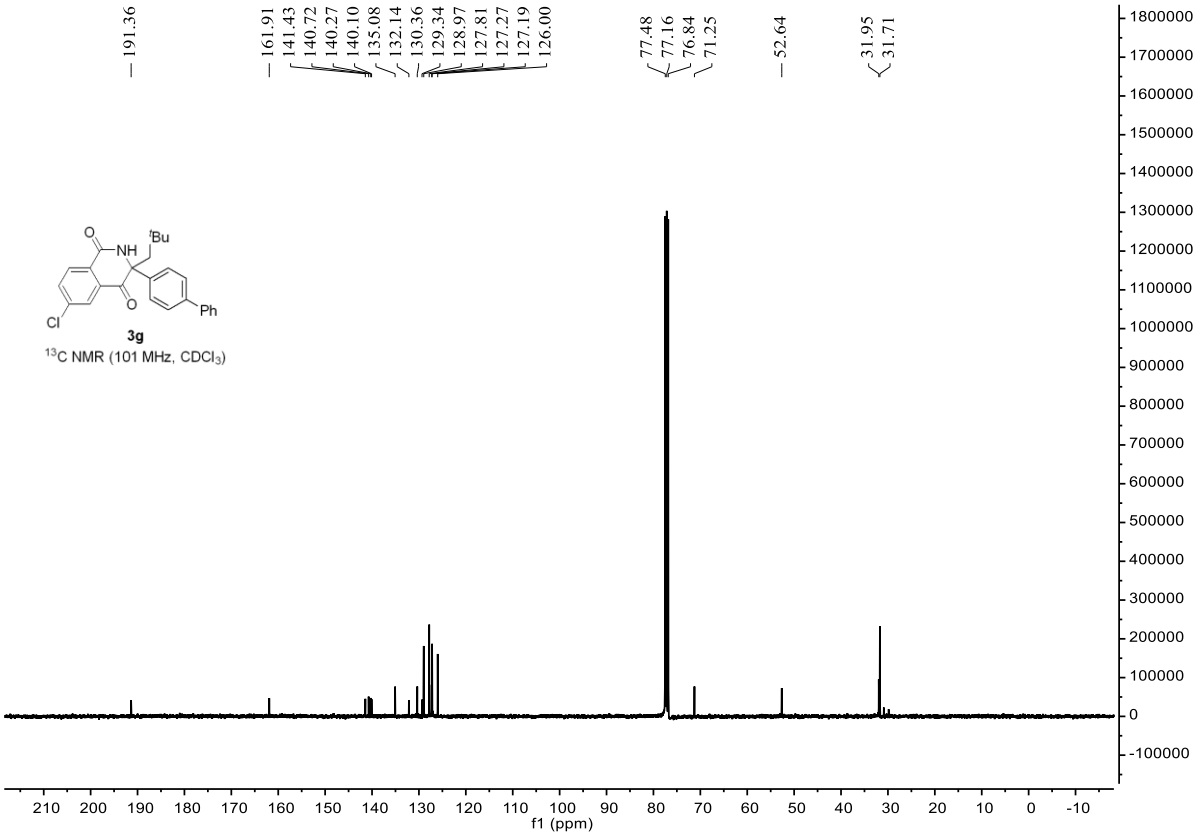
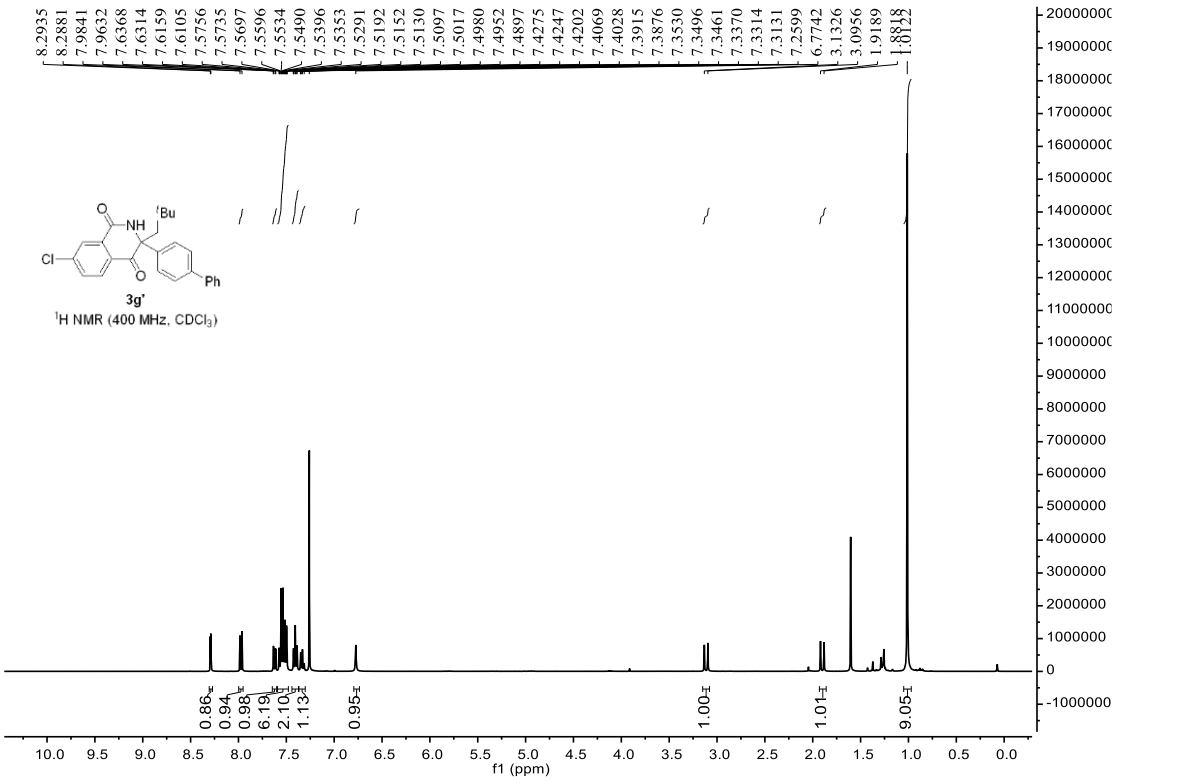
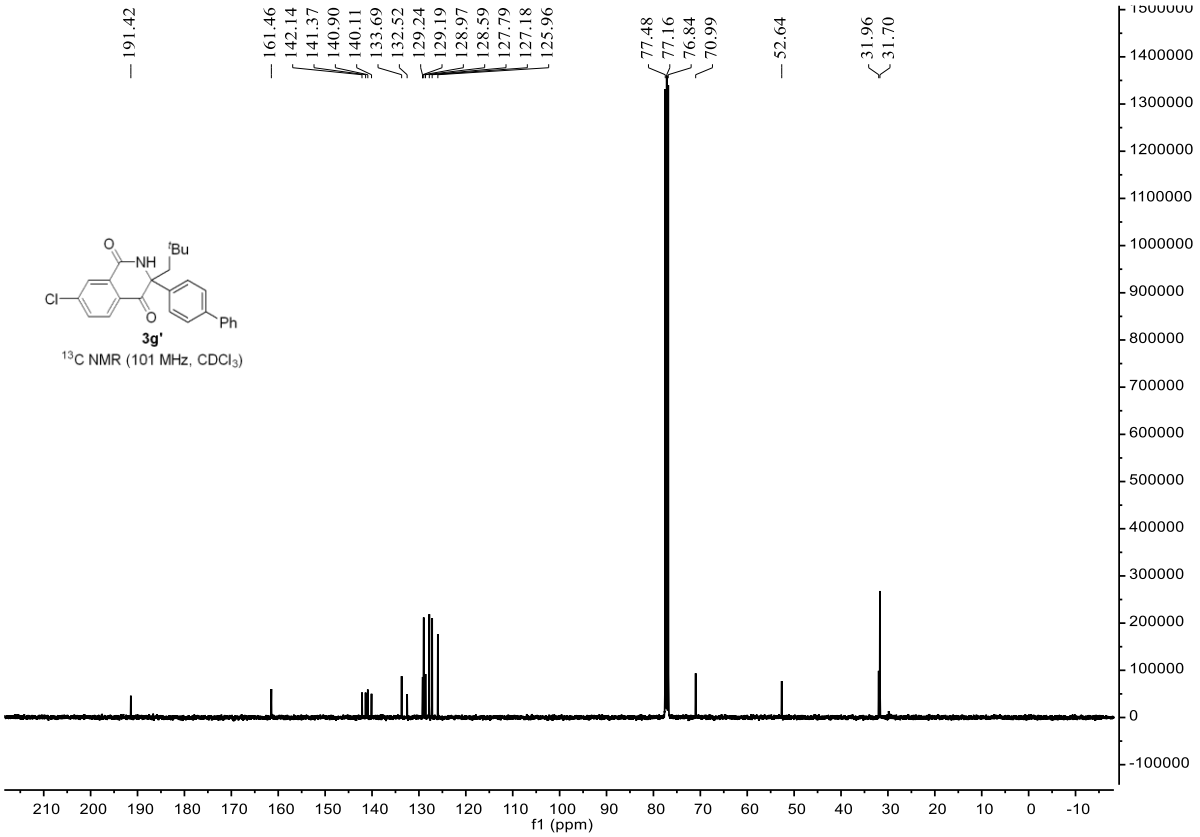
163.21
141.24
140.50
140.21
135.07
133.39
131.12
131.10
128.96
128.51
127.74
127.69
127.29
127.18
126.21
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.73
43.88
36.84
26.16
26.01
10.65
10.21
NH
O
O
Ph
3f
13C NMR (151 MHz, CDCl3)
f1 (ppm)


8.2737
8.2527
7.9824
7.9770
7.7351
7.7297
7.7143
7.7088
7.5680
7.5622
7.5464
7.5332
7.5264
7.5179
7.5131
7.4959
7.4359
7.4262
7.4075
7.3881
7.3508
7.3387
7.3326
7.3260
7.3146
7.2592
6.7406
3.1199
3.0828
1.8867
1.8496
1.0027
NH
tBu
O
O
Cl
Ph
3g
1H NMR (400 MHz, CDCl3)
1.00
0.92
1.05
6.37
2.32
1.25
0.97
1.00
1.06
8.99
f1 (ppm)

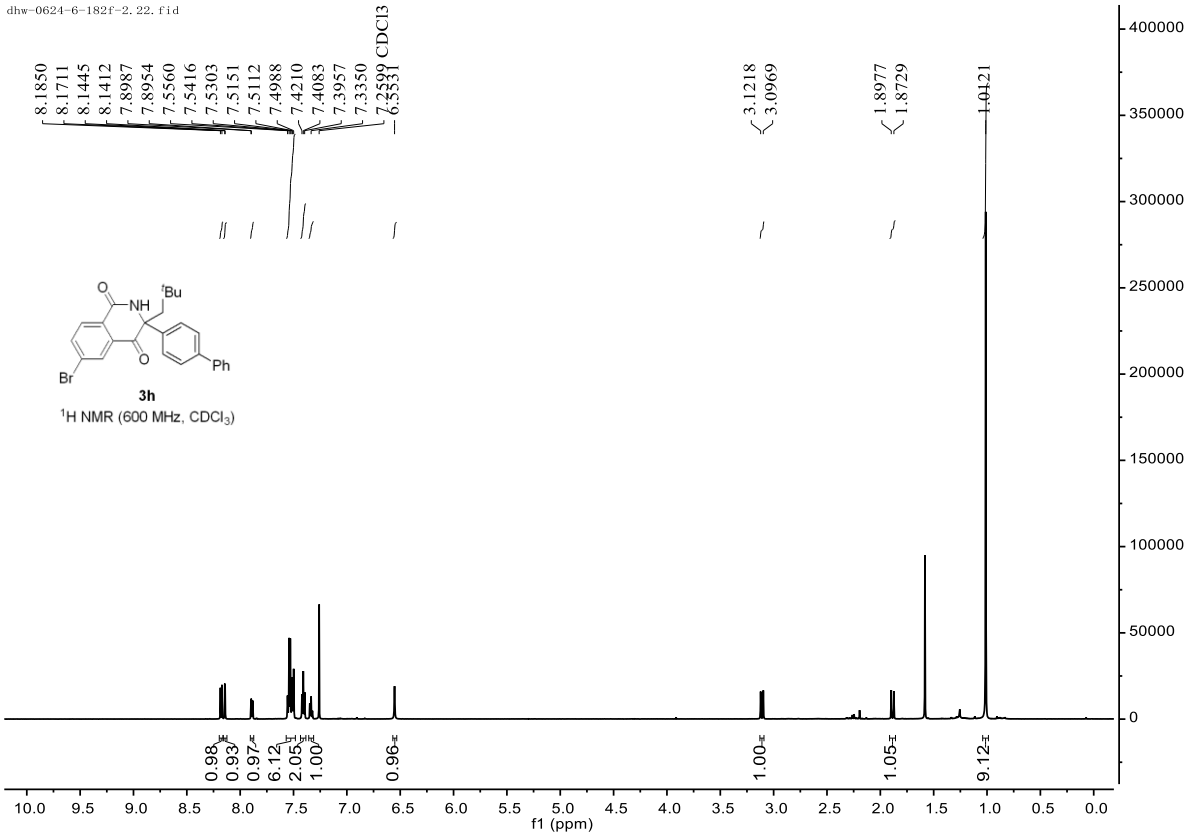

191.36
161.91
141.43
140.72
140.27
140.10
135.08
132.14
130.36
129.34
128.97
127.81
127.27
127.19
126.00
77.48
77.16
76.84
71.25
52.64
31.95
31.71
NH
tBu
O
O
Cl
Ph
3g
13C NMR (101 MHz, CDCl3)
f1 (ppm)


8.2935
8.2881
7.9841
7.9632
7.6368
7.6314
7.6159
7.6105
7.5756
7.5735
7.5697
7.5596
7.5534
7.5490
7.5396
7.5353
7.5291
7.5192
7.5152
7.5130
7.5097
7.5017
7.4980
7.4952
7.4897
7.4275
7.4247
7.4202
7.4069
7.4028
7.3915
7.3876
7.3530
7.3496
7.3461
7.3370
7.3314
7.3131
7.2599
6.7742
3.1326
3.0956
1.9189
1.8818
1.0122
NH
tBu
O
O
Cl
Ph
3g'
1H NMR (400 MHz, CDCl3)
0.86
0.94
0.98
6.19
2.10
1.13
0.95
1.00
1.01
9.05
f1 (ppm)



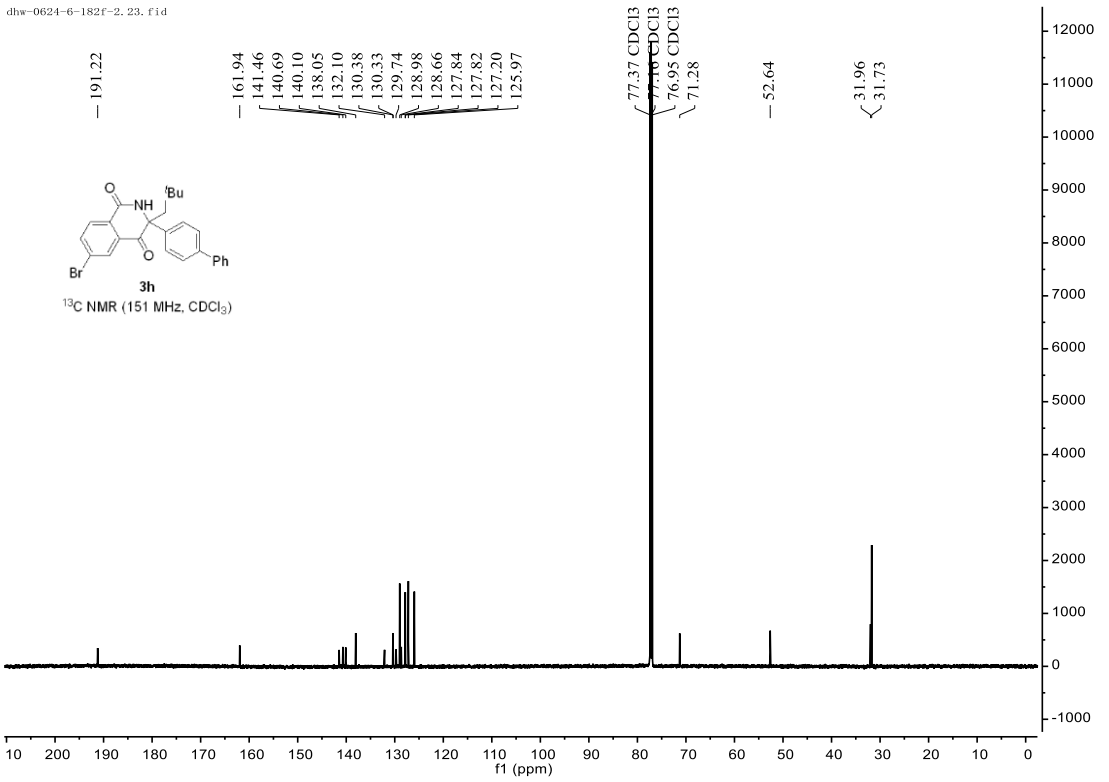
3g'

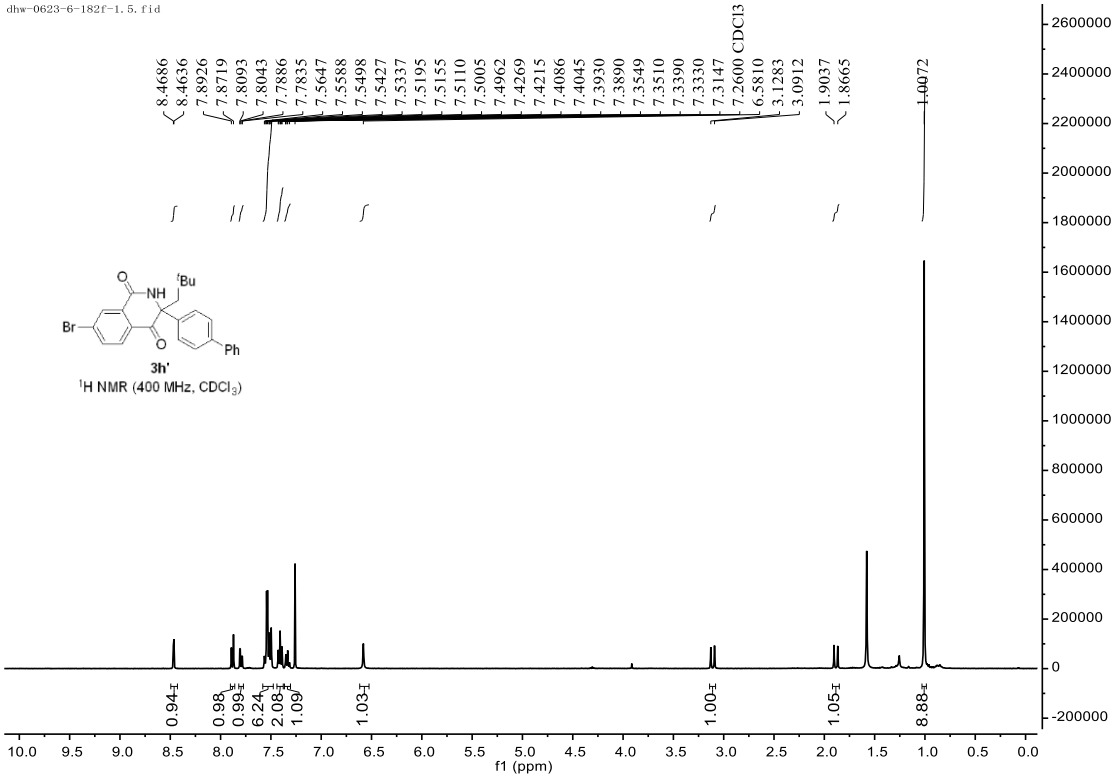
^{13}C NMR (101 MHz, $CDCl_3$)



3h

^{1}H NMR (600 MHz, $CDCl_3$)


dhw-0624-6-182f-2.23.fid
191.22
161.94
141.46
140.69
140.10
138.05
132.10
130.38
130.33
129.74
128.98
128.66
127.84
127.82
127.20
125.97
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.28
52.64
31.96
31.73
NH
tBu
O
Br
Ph
3h
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0623-6-182f-1.5.fid
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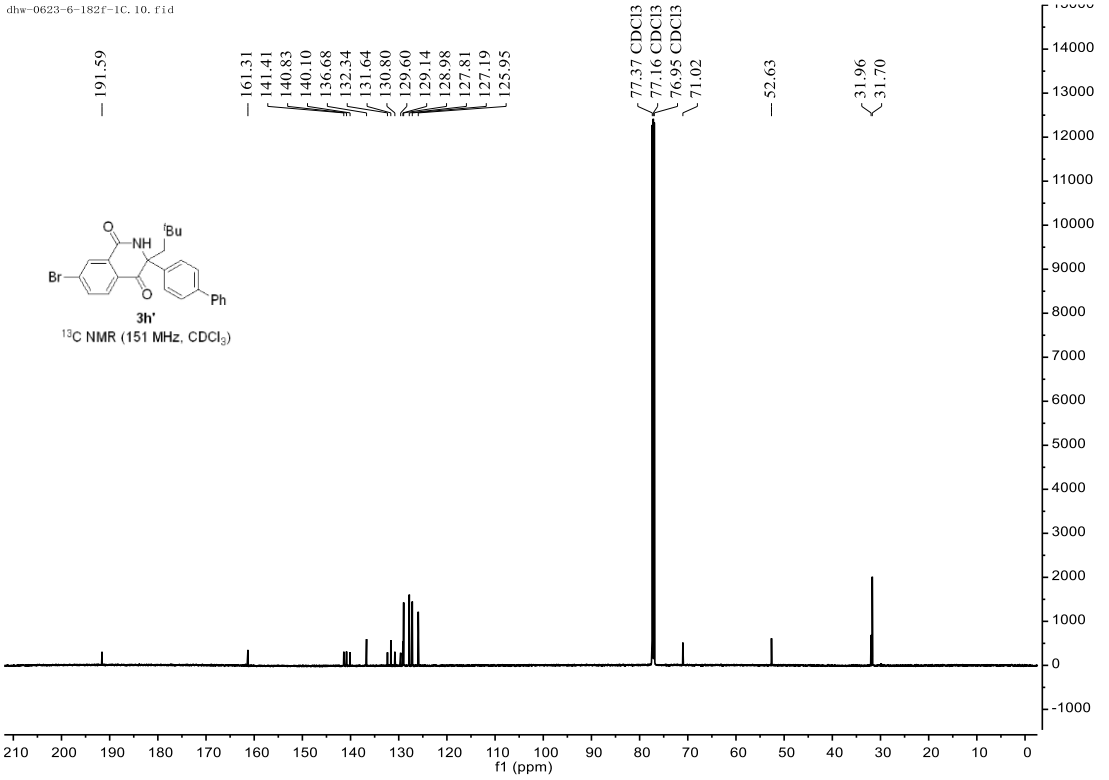
8.4636
7.8926
7.8719
7.8093
7.8043
7.7886
7.7835
7.5647
7.5588
7.5498
7.5427
7.5337
7.5195
7.5155
7.5110
7.5005
7.4962
7.4269
7.4215
7.4086
7.4045
7.3930
7.3890
7.3549
7.3510
7.3390
7.3330
7.3147
7.2600 CDCl3
6.5810
3.1283
3.0912
1.9037
1.8665
1.0072
0.94
0.98
0.99
6.24
2.08
1.09
1.03
1.00
1.05
8.88
NH
tBu
Br
O
Ph
3h'
1H NMR (400 MHz, CDCl3)
f1 (ppm)


dhw-0623-6-182f-1C.10.fid
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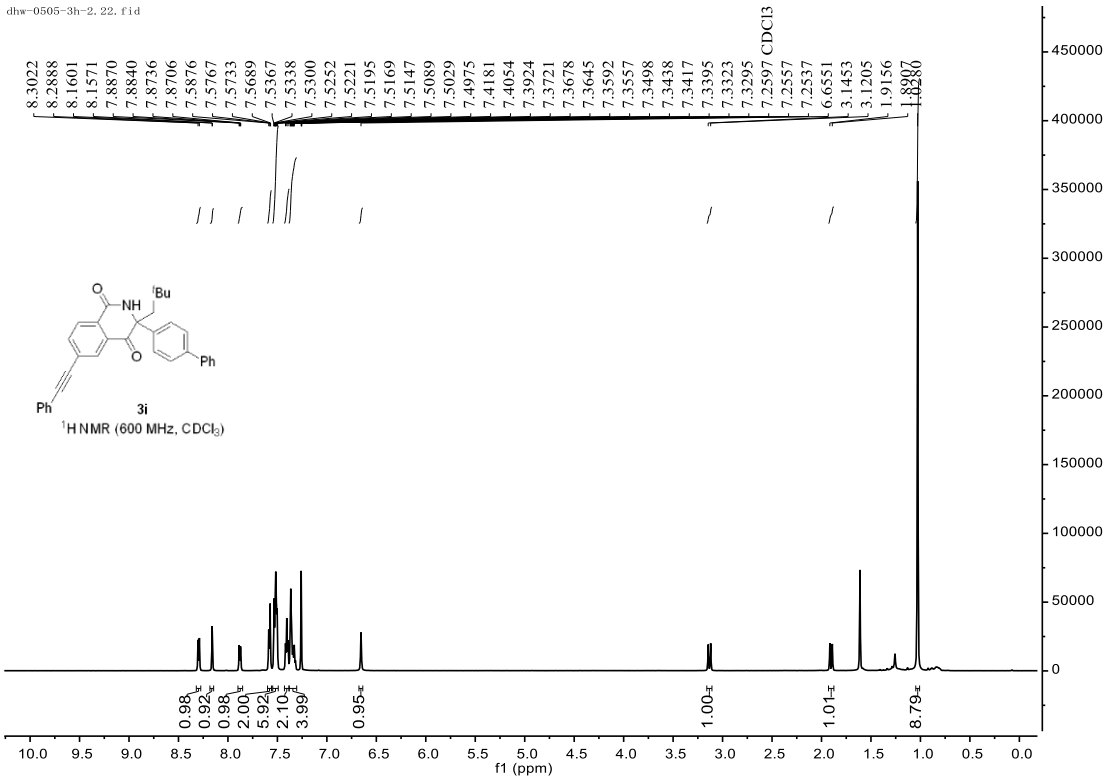
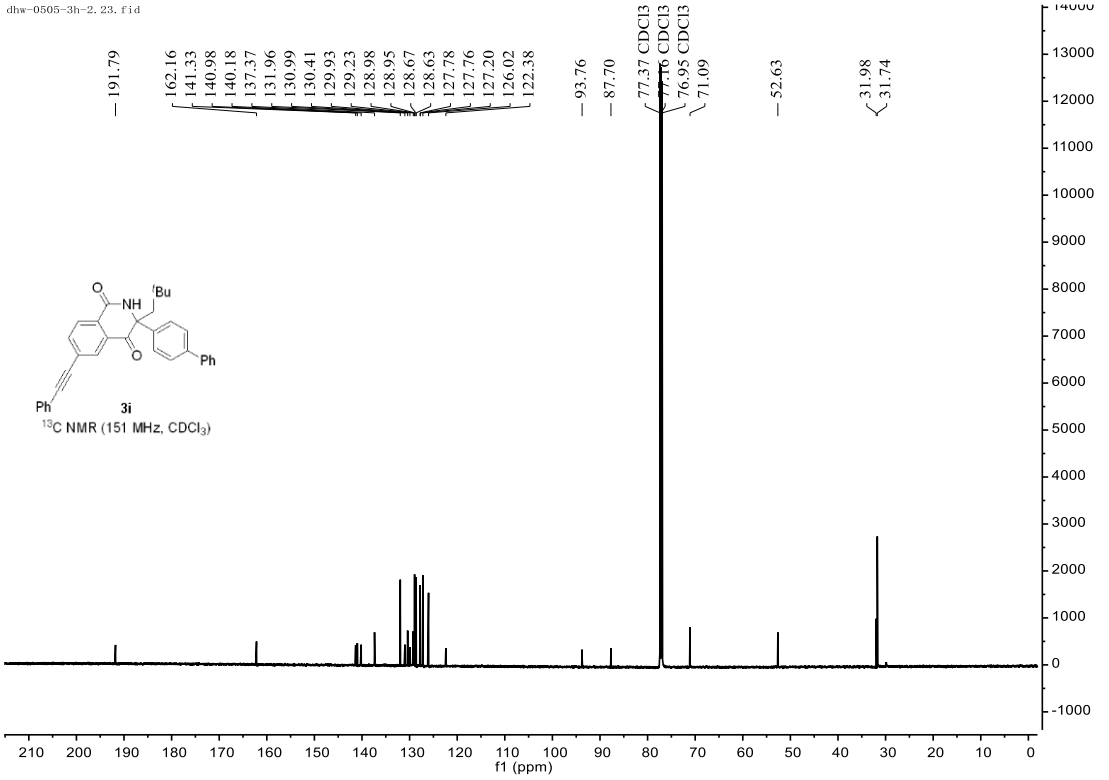
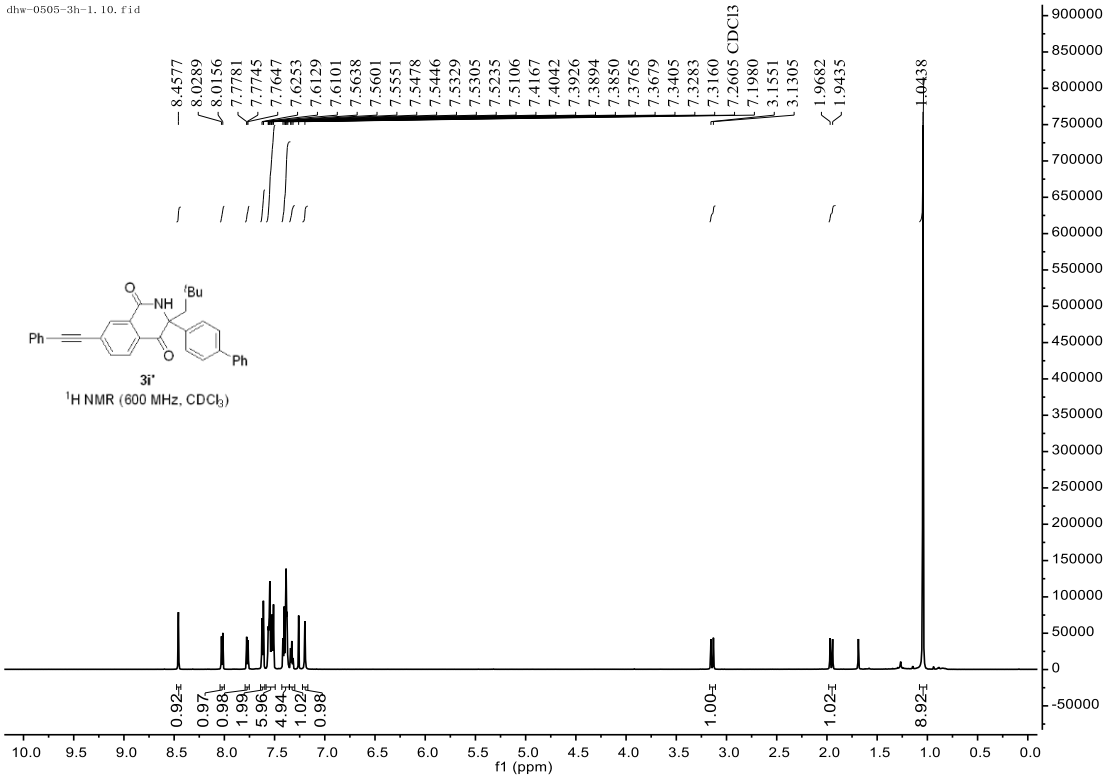
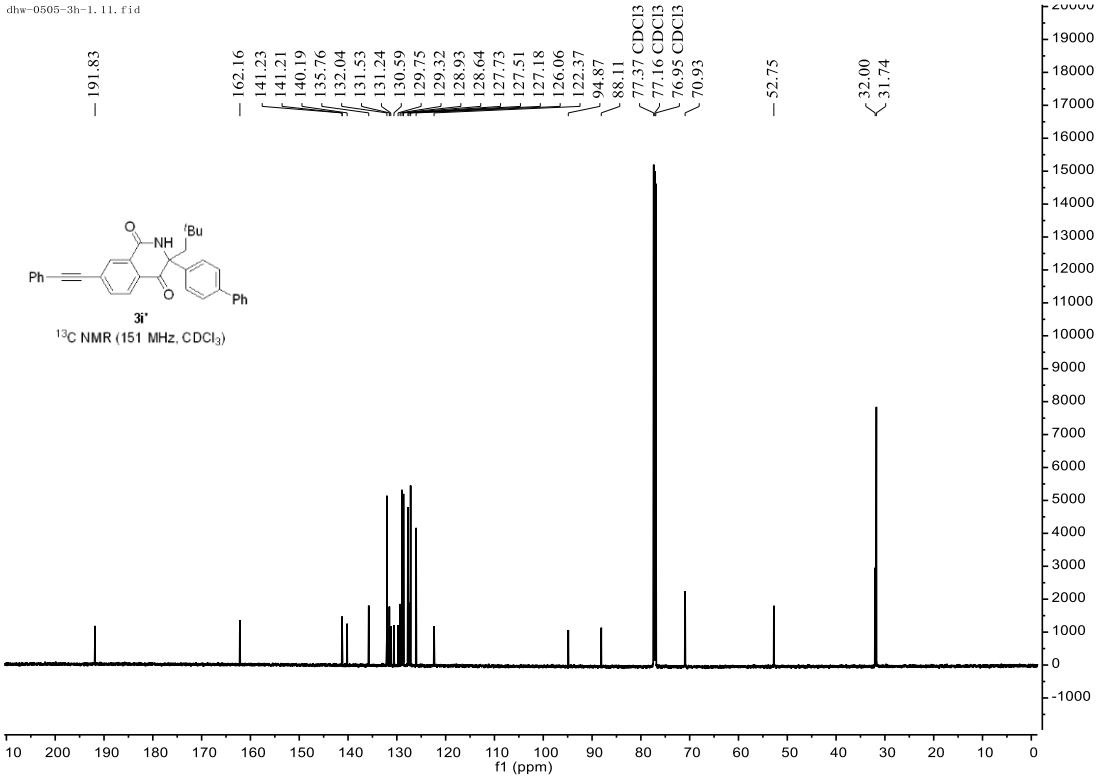
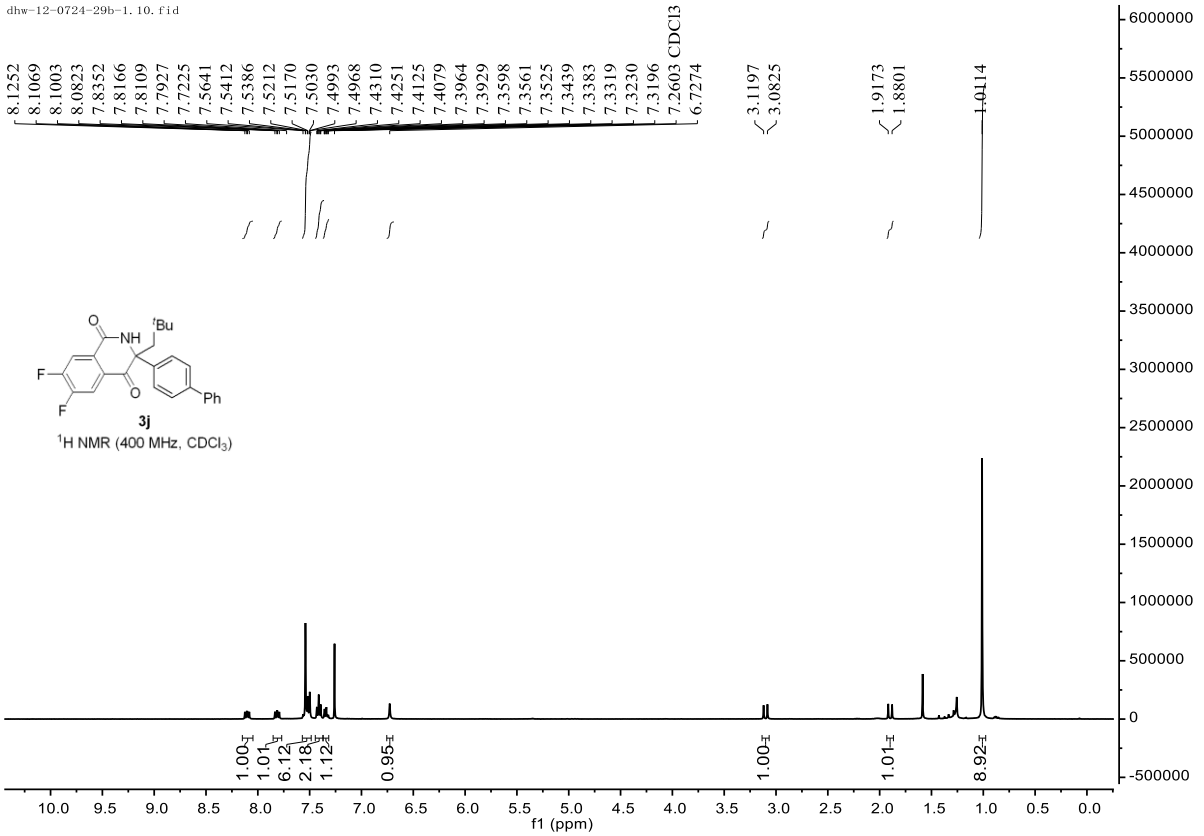
161.31
141.41
140.83
140.10
136.68
132.34
131.64
130.80
129.60
129.14
128.98
127.81
127.19
125.95
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.02
52.63
31.96
31.70
Br
NH
tBu
O
O
Ph
3h'
13C NMR (151 MHz, CDCl3)
f1 (ppm)

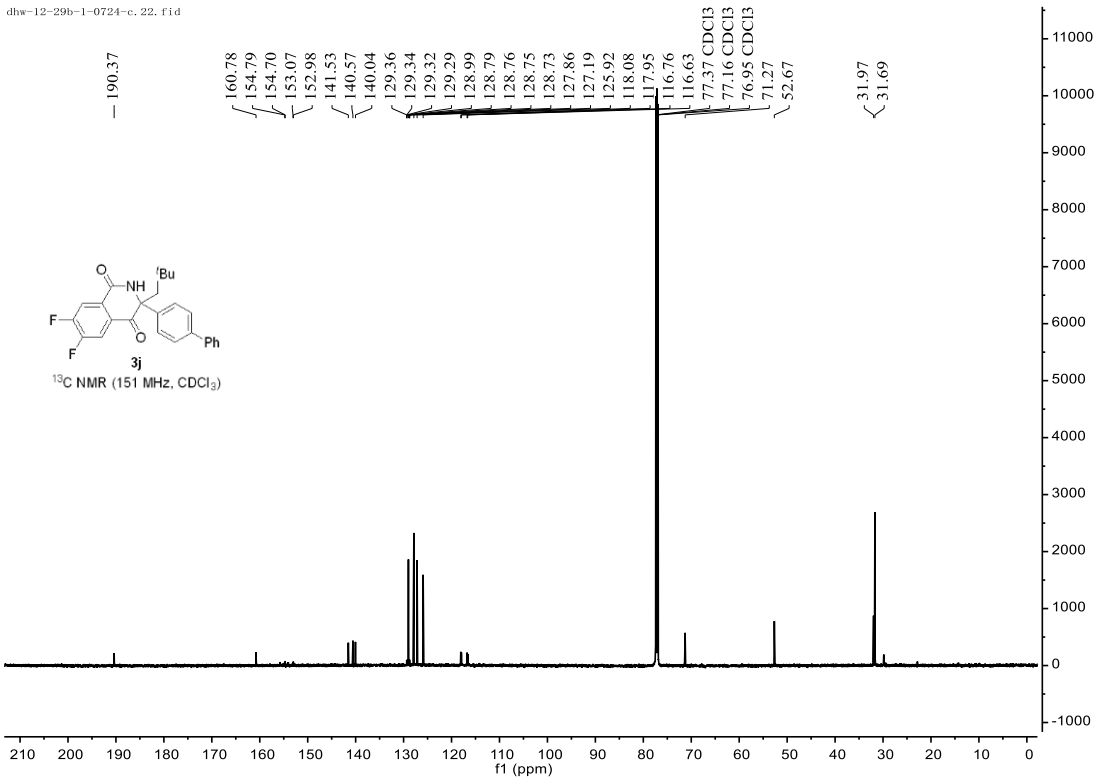

dhw-0505-3h-2.22.fid
8.3022
8.2888
8.1601
8.1571
7.8870
7.8840
7.8736
7.8706
7.5876
7.5767
7.5733
7.5689
7.5367
7.5338
7.5300
7.5252
7.5221
7.5195
7.5169
7.5147
7.5089
7.5029
7.4975
7.4181
7.4054
7.3924
7.3721
7.3678
7.3645
7.3592
7.3557
7.3498
7.3438
7.3417
7.3395
7.3323
7.3295
7.2597 CDCl3
7.2557
7.2537
6.6551
3.1453
3.1205
1.9156
1.8907
1.8280
NH
tBu
O
O
Ph
Ph
3i
1H NMR (600 MHz, CDCl3)
0.98
0.92
0.98
2.00
5.92
2.10
3.99
0.95
1.00
1.01
8.79
f1 (ppm)

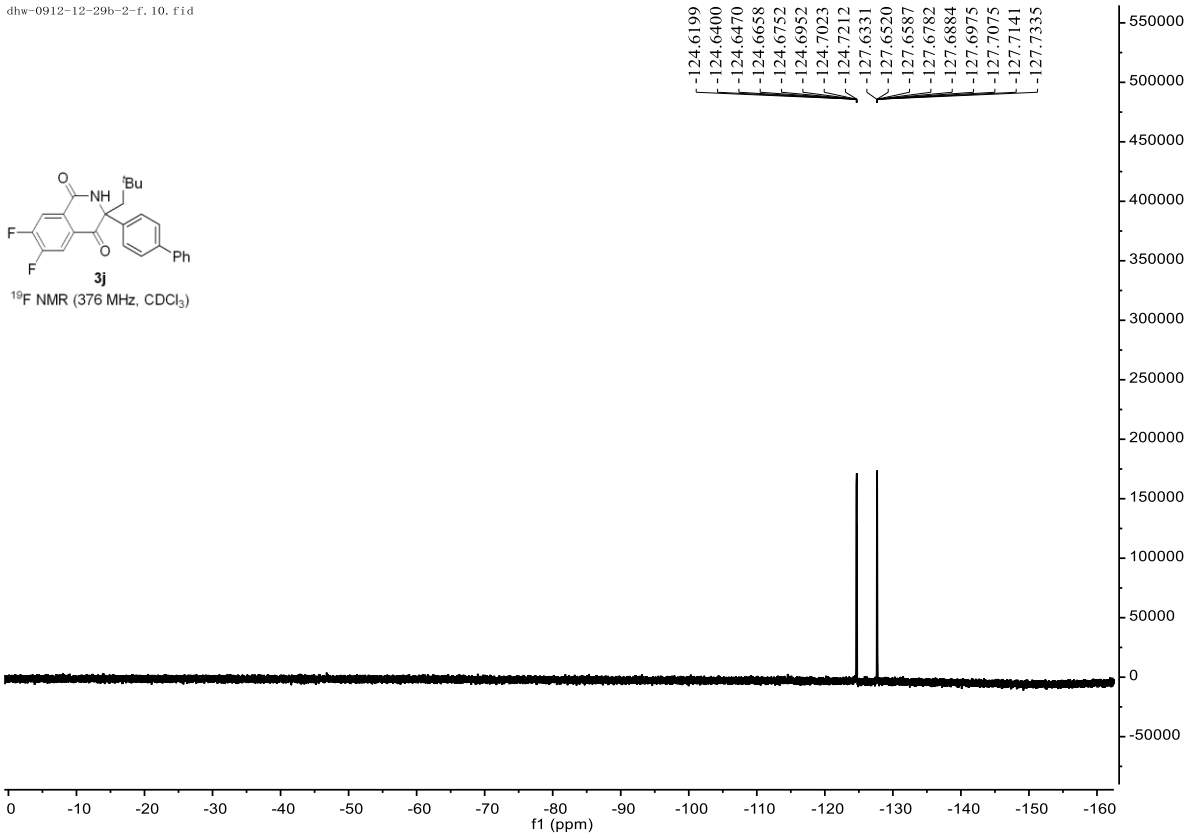

dhw-0505-3h-2.23.fid
191.79
162.16
141.33
140.98
140.18
137.37
131.96
130.99
130.41
129.93
129.23
128.98
128.95
128.67
128.63
127.78
127.76
127.20
126.02
122.38
93.76
87.70
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.09
52.63
31.98
31.74
NH
tBu
O
Ph
Ph
3i
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0505-3h-1.10.fid
8.4577
8.0289
8.0156
7.7781
7.7745
7.7647
7.6253
7.6129
7.6101
7.5638
7.5601
7.5551
7.5478
7.5446
7.5329
7.5305
7.5235
7.5106
7.4167
7.4042
7.3926
7.3894
7.3850
7.3765
7.3679
7.3405
7.3283
7.3160
7.2605 CDCl3
7.1980
3.1551
3.1305
1.9682
1.9435
1.0438
0.92
0.97
0.98
1.99
5.96
4.94
1.02
0.98
1.00
1.02
8.92
NH
tBu
O
Ph
Ph
3i'
1H NMR (600 MHz, CDCl3)
f1 (ppm)


dhw-0505-3h-1.11.fid
191.83
162.16
141.23
141.21
140.19
135.76
132.04
131.53
131.24
130.59
129.75
129.32
128.93
128.64
127.73
127.51
127.18
126.06
122.37
94.87
88.11
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.93
52.75
32.00
31.74
NH
tBu
Ph
Ph
3i'
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-12-0724-29b-1.10.fid
8.1252
8.1069
8.1003
8.0823
7.8352
7.8166
7.8109
7.7927
7.7225
7.5641
7.5412
7.5386
7.5212
7.5170
7.5030
7.4993
7.4968
7.4310
7.4251
7.4125
7.4079
7.3964
7.3929
7.3598
7.3561
7.3525
7.3439
7.3383
7.3319
7.3230
7.3196
7.2603 CDCl3
6.7274
3.1197
3.0825
1.9173
1.8801
1.0114
NH
tBu
F
F
O
Ph
3j
1H NMR (400 MHz, CDCl3)
1.00
1.01
6.12
2.18
1.12
0.95
1.00
1.01
8.92
f1 (ppm)


dhw-12-29b-1-0724-c.22.fid
190.37
160.78
154.79
154.70
153.07
152.98
141.53
140.57
140.04
129.36
129.34
129.32
129.29
128.99
128.79
128.76
128.75
128.73
127.86
127.19
125.92
118.08
117.95
116.76
116.63
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.27
52.67
31.97
31.69
tBu
NH
O
F
F
Ph
3j
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0912-12-29b-2-f.10.fid
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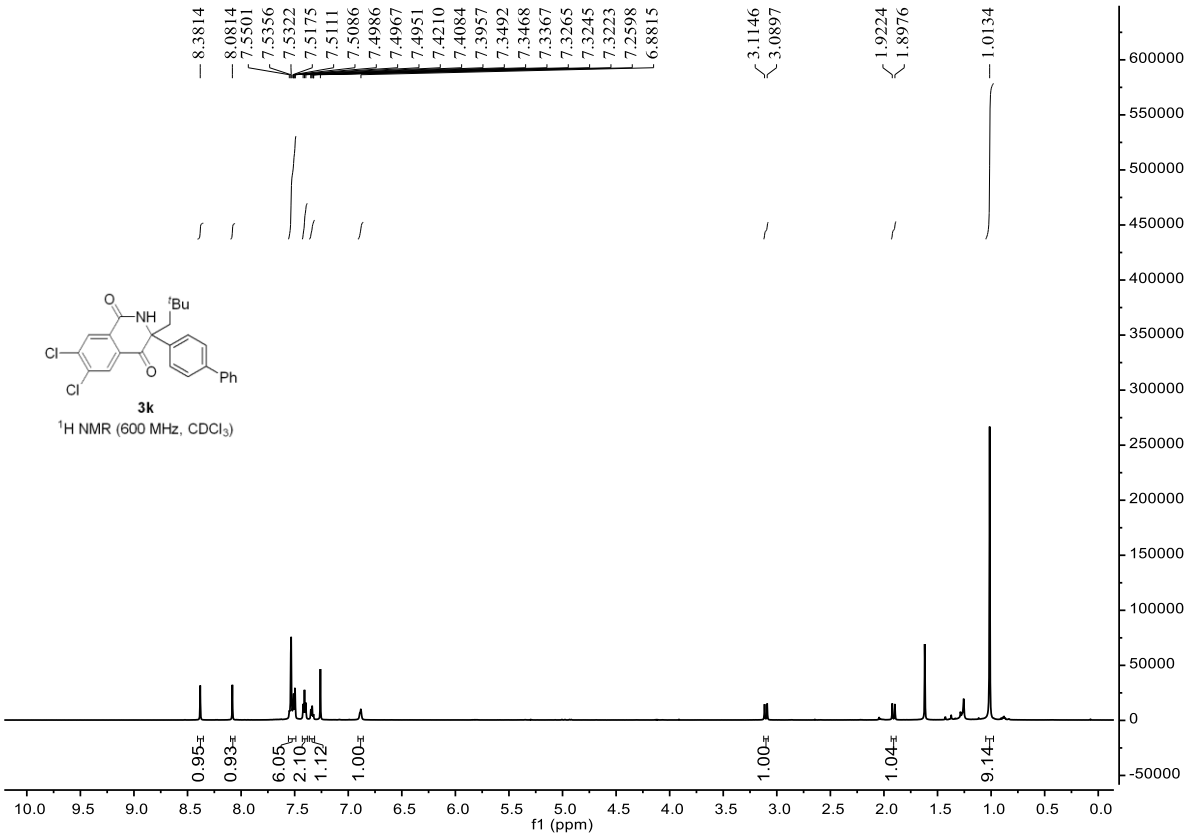
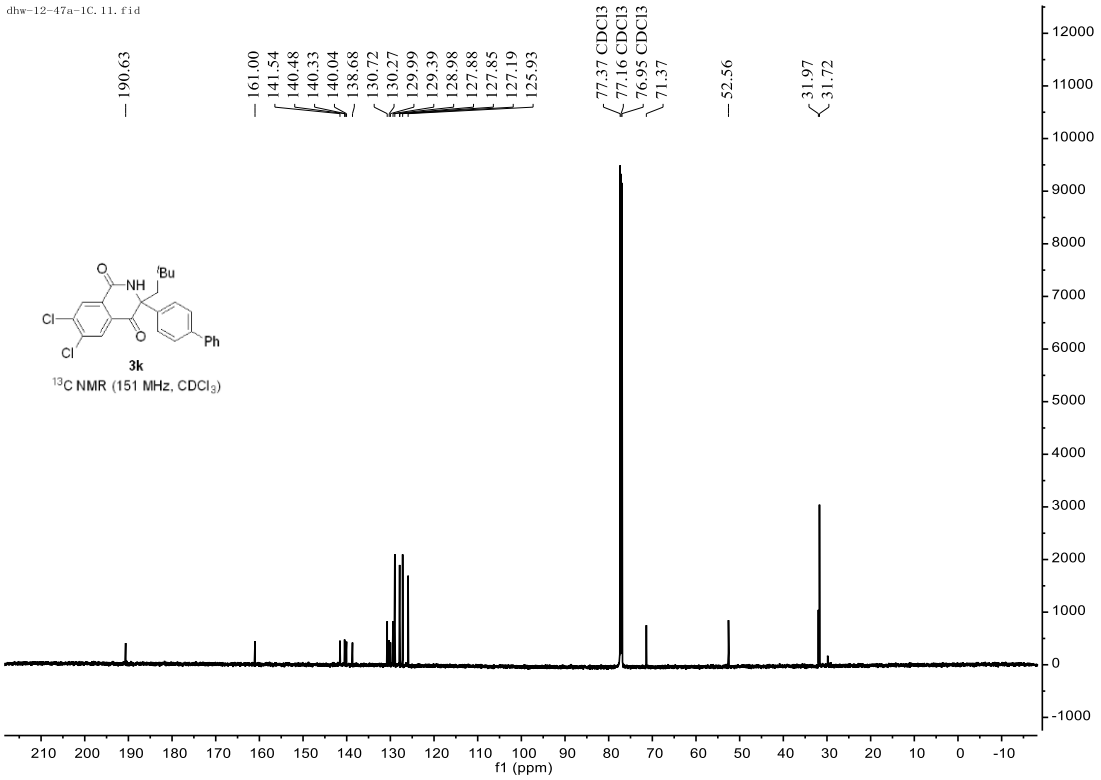
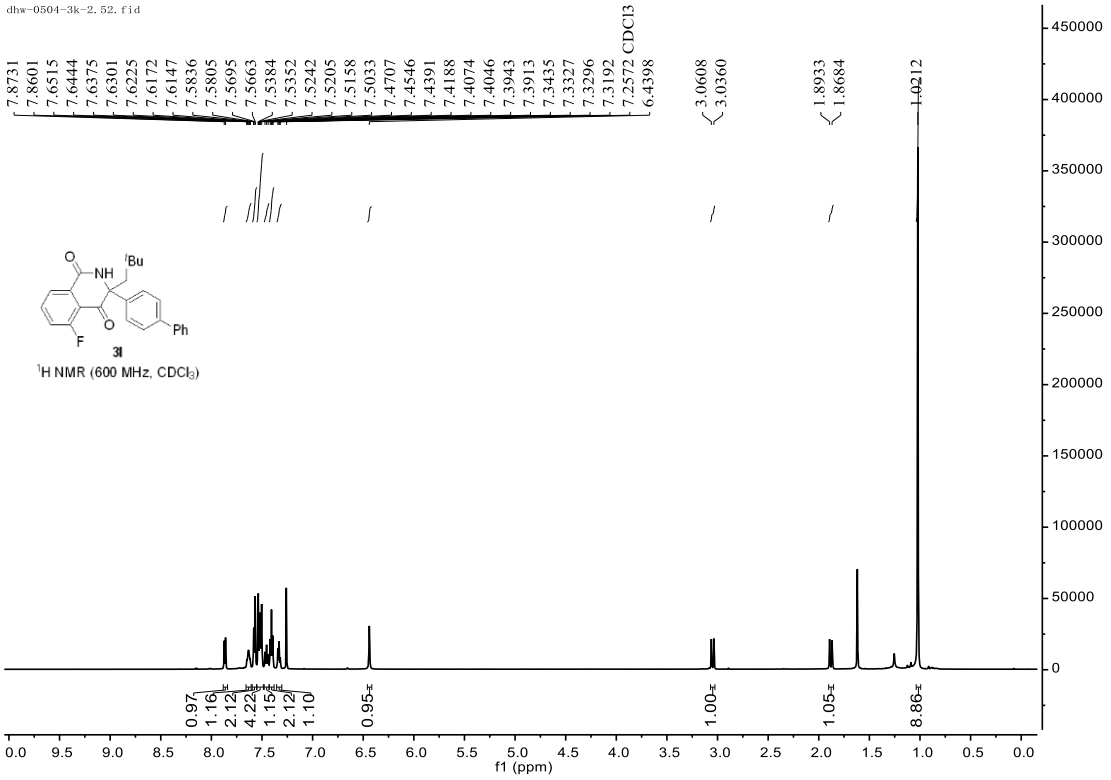
-124.6400
-124.6470
-124.6658
-124.6752
-124.6952
-124.7023
-124.7212
-127.6331
-127.6520
-127.6587
-127.6782
-127.6884
-127.6975
-127.7075
-127.7141
-127.7335
tBu
NH
O
F
F
Ph
3j
19F NMR (376 MHz, CDCl3)
f1 (ppm)

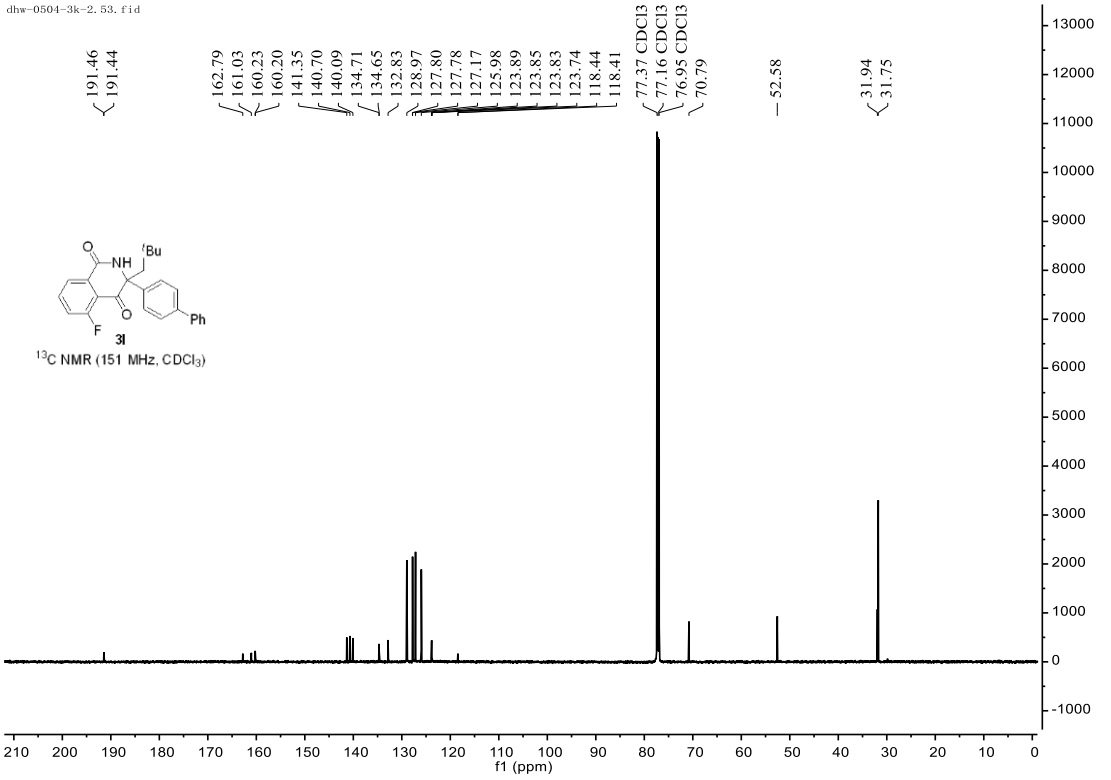

8.3814
8.0814
7.5501
7.5356
7.5322
7.5175
7.5111
7.5086
7.4986
7.4967
7.4951
7.4210
7.4084
7.3957
7.3492
7.3468
7.3367
7.3265
7.3245
7.3223
7.2598
6.8815
3.1146
3.0897
1.9224
1.8976
1.0134
0.95
0.93
6.05
2.10
1.12
1.00
1.00
1.04
9.14
f1 (ppm)

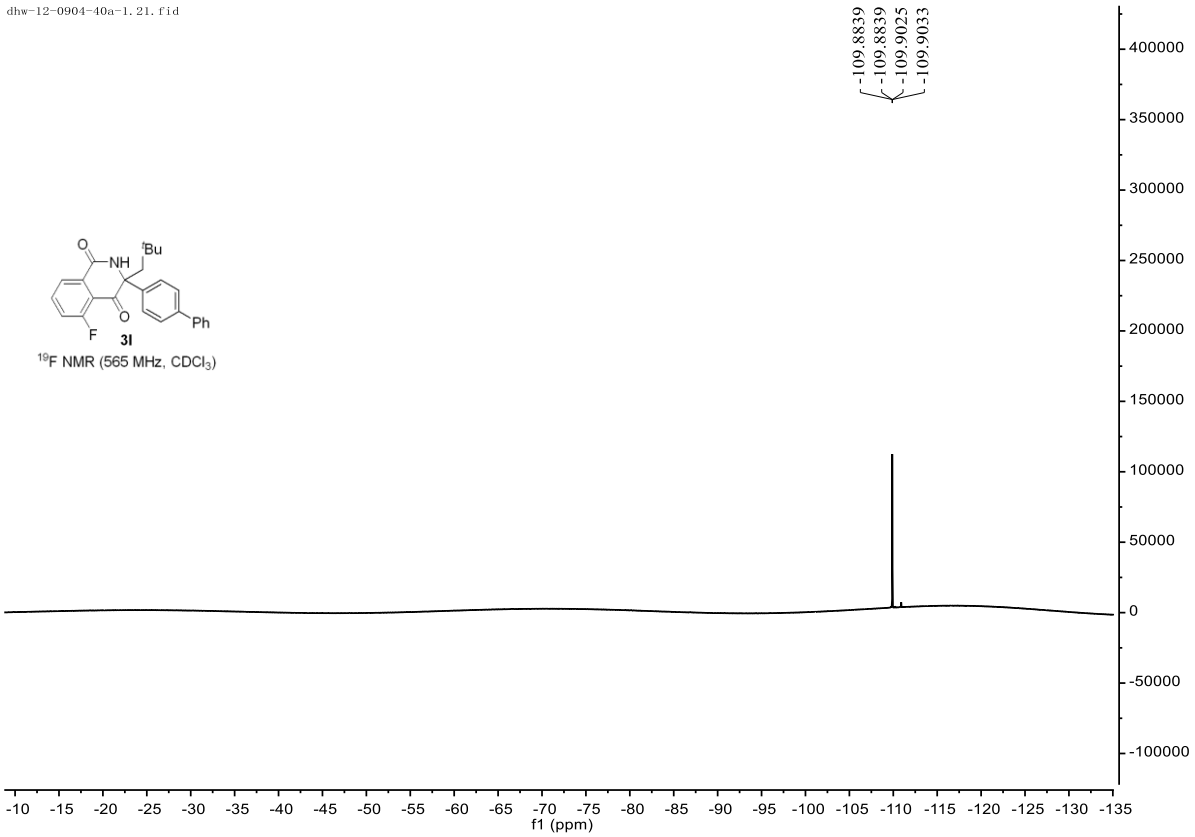
3k
1H NMR (600 MHz, CDCl3)

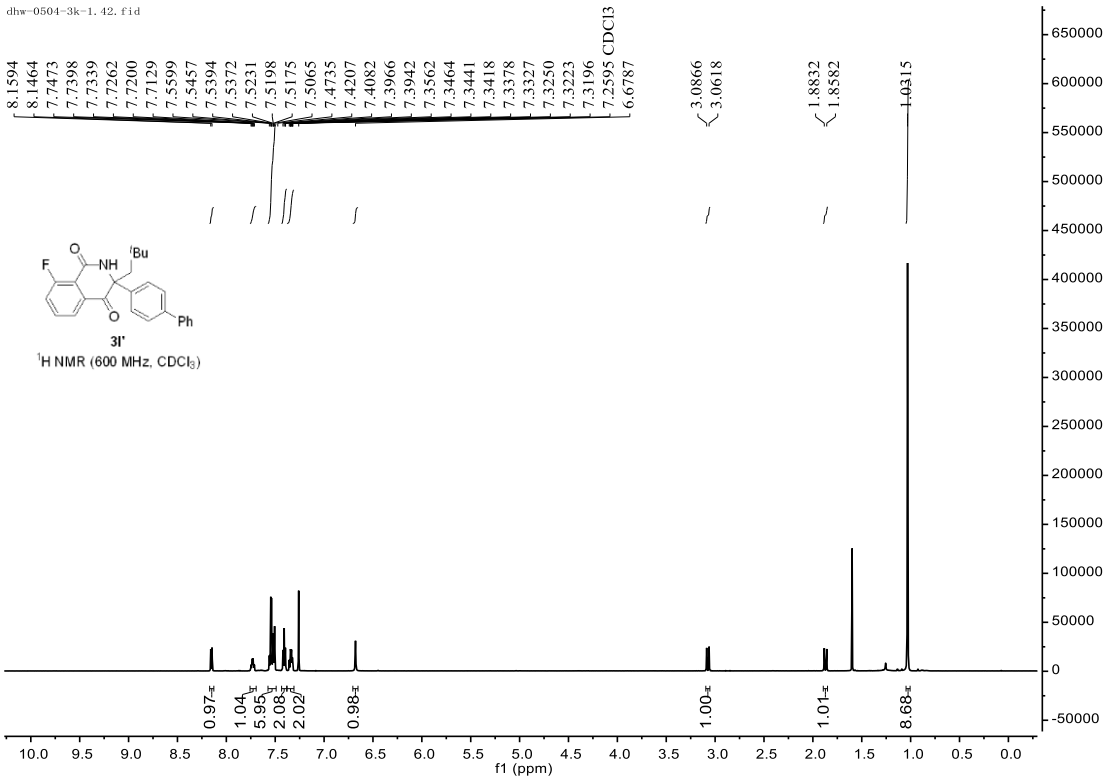

dhw-12-47a-1C.11.fid
190.63
161.00
141.54
140.48
140.33
140.04
138.68
130.72
130.27
129.99
129.39
128.98
127.88
127.85
127.19
125.93
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.37
52.56
31.97
31.72
f1 (ppm)

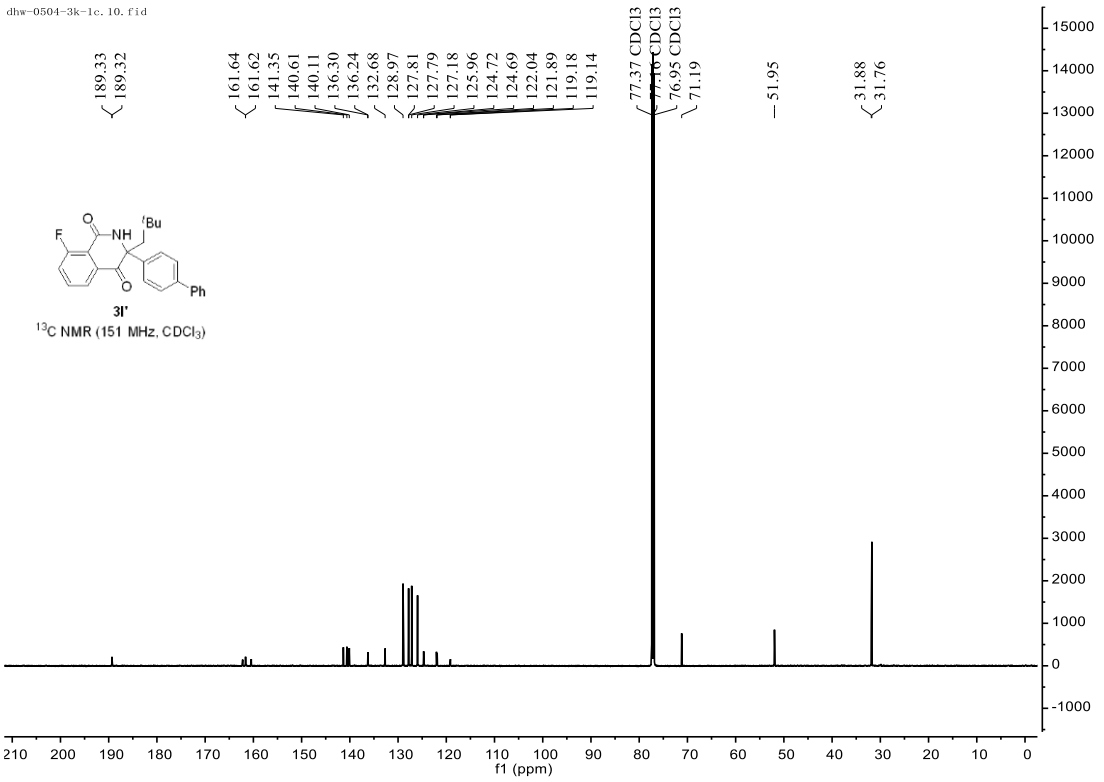
3k
13C NMR (151 MHz, CDCl3)

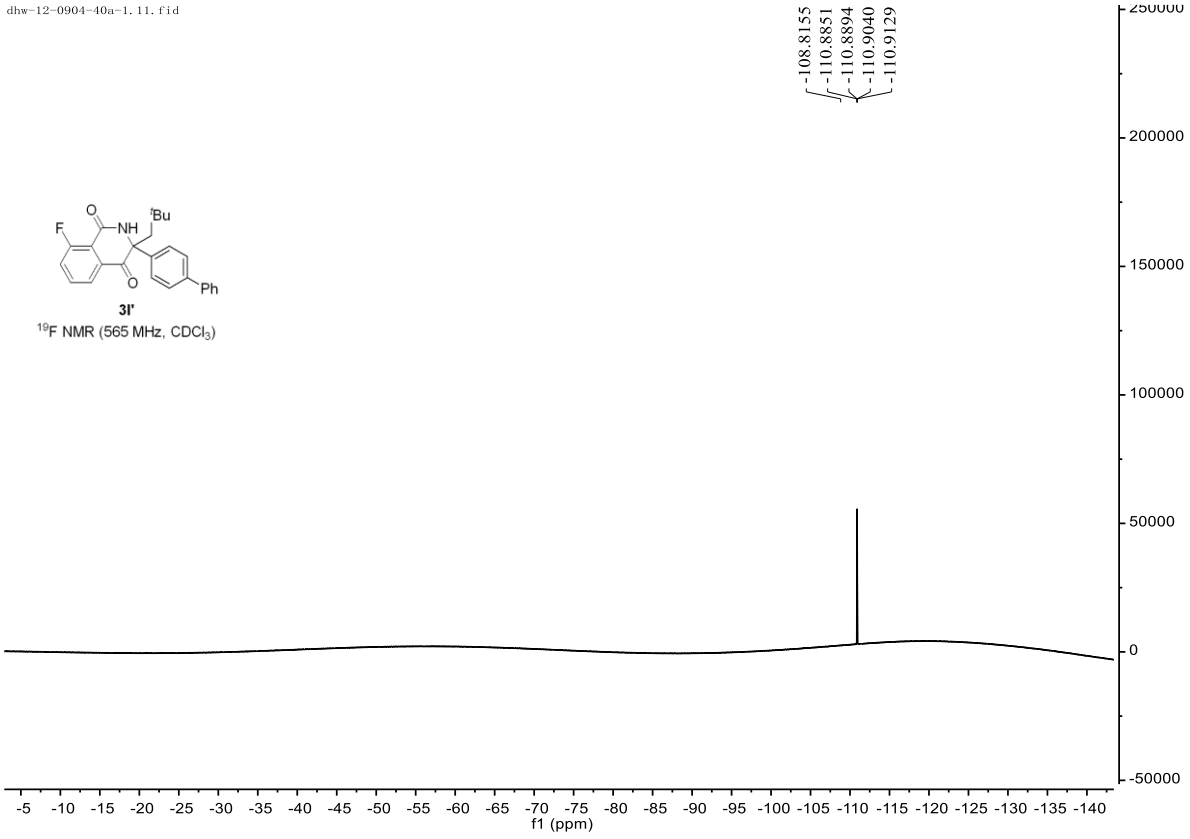

dhw-0504-3k-2.52.fid
7.8731
7.8601
7.6515
7.6444
7.6375
7.6301
7.6225
7.6172
7.6147
7.5836
7.5805
7.5695
7.5663
7.5384
7.5352
7.5242
7.5205
7.5158
7.5033
7.4707
7.4546
7.4391
7.4188
7.4074
7.4046
7.3943
7.3913
7.3435
7.3327
7.3296
7.3192
7.2572 CDCl3
6.4398
3.0608
3.0360
1.8933
1.8684
1.0212
tBu
NH
O
O
F
Ph
3l
1H NMR (600 MHz, CDCl3)
0.97
1.16
2.12
4.22
1.15
2.12
1.10
0.95
1.00
1.05
8.86
f1 (ppm)

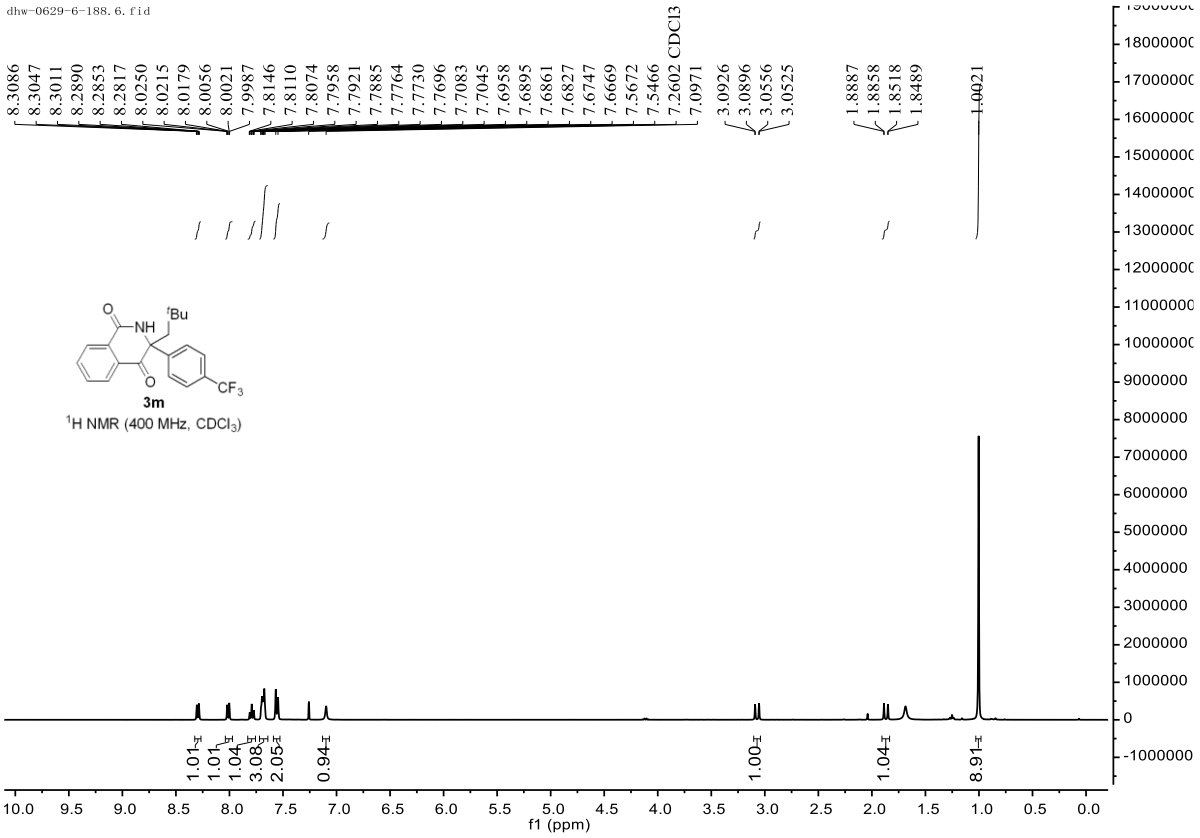

dhw-0504-3k-2.53.fid
191.46
191.44
162.79
161.03
160.23
160.20
141.35
140.70
140.09
134.71
134.65
132.83
128.97
127.80
127.78
127.17
125.98
123.89
123.85
123.83
123.74
118.44
118.41
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.79
52.58
31.94
31.75
tBu
NH
O
O
F
Ph
3l
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-12-0904-40a-1.21.fid
-109.8839
-109.8839
-109.9025
-109.9033
3l
19F NMR (565 MHz, CDCl3)
f1 (ppm)


dhw-0504-3k-1.42.fid
8.1594
8.1464
7.7473
7.7398
7.7339
7.7262
7.7200
7.7129
7.5599
7.5457
7.5394
7.5372
7.5231
7.5198
7.5175
7.5065
7.4735
7.4207
7.4082
7.3966
7.3942
7.3562
7.3464
7.3441
7.3418
7.3378
7.3327
7.3250
7.3223
7.3196
7.2595 CDCl3
6.6787
3.0866
3.0618
1.8832
1.8582
1.0315
3l'
1H NMR (600 MHz, CDCl3)
0.97
1.04
5.95
2.08
2.02
0.98
1.00
1.01
8.68
f1 (ppm)


dhw-0504-3k-1c.10.fid
189.33
189.32
161.64
161.62
141.35
140.61
140.11
136.30
136.24
132.68
128.97
127.81
127.79
127.18
125.96
124.72
124.69
122.04
121.89
119.18
119.14
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.19
51.95
31.88
31.76
F
O
NH
tBu
O
Ph
3l'
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-12-0904-40a-1.11.fid
-108.8155
-110.8851
-110.8894
-110.9040
-110.9129
F
O
NH
tBu
O
Ph
3l'
19F NMR (565 MHz, CDCl3)
f1 (ppm)


dhw-0629-6-188.6.fid
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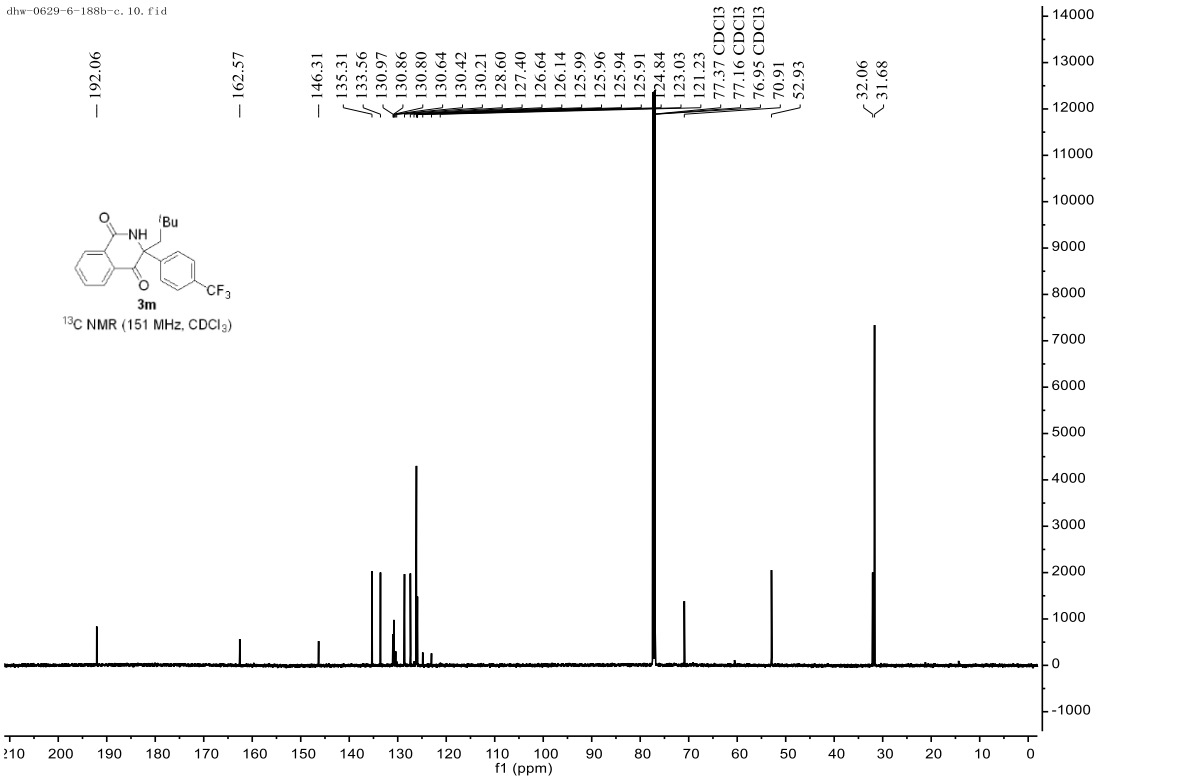
8.3047
8.3011
8.2890
8.2853
8.2817
8.0250
8.0215
8.0179
8.0056
8.0021
7.9987
7.8146
7.8110
7.8074
7.7958
7.7921
7.7885
7.7764
7.7730
7.7696
7.7083
7.7045
7.6958
7.6895
7.6861
7.6827
7.6747
7.6669
7.5672
7.5466
7.2602 CDCl3
7.0971
3.0926
3.0896
3.0556
3.0525
1.8887
1.8858
1.8518
1.8489
1.0021
tBu
NH
O
O
CF3
3m
1H NMR (400 MHz, CDCl3)
1.01
1.01
1.04
3.08
2.05
0.94
1.00
1.04
8.91
f1 (ppm)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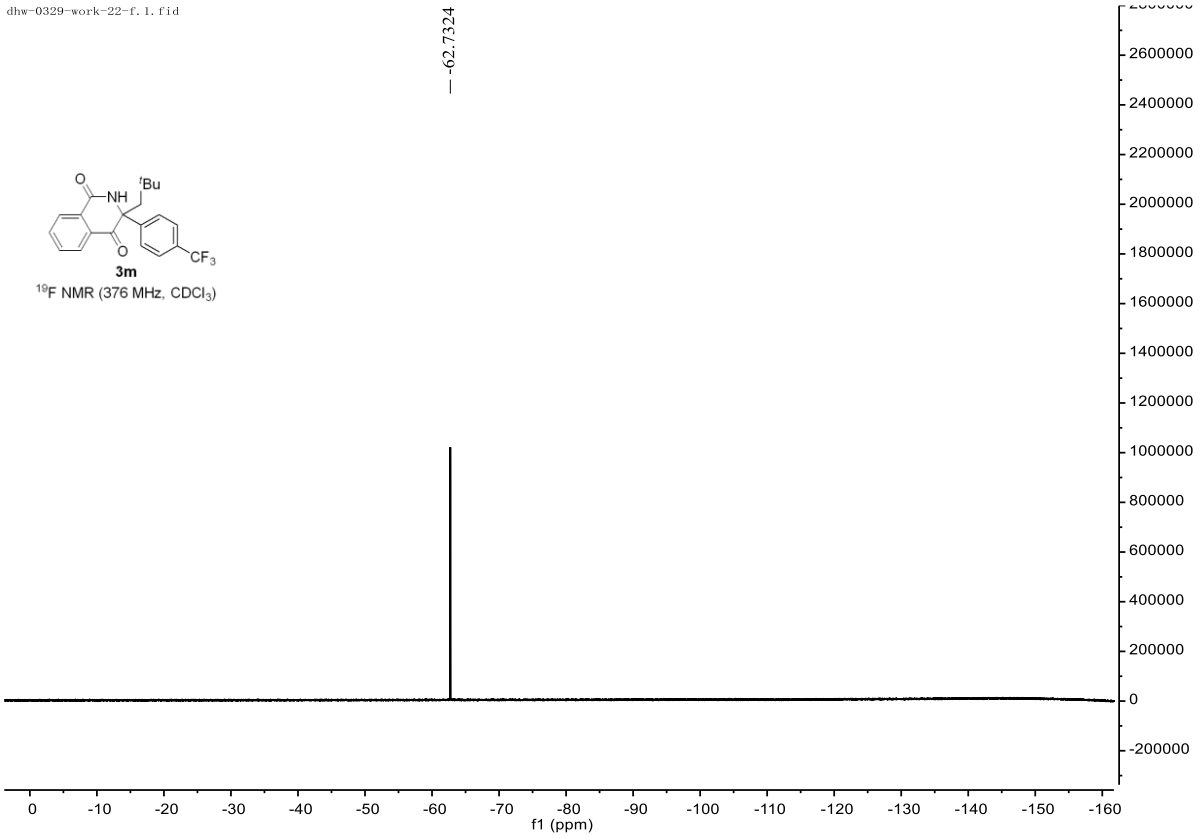
192.06
162.57
146.31
135.31
133.56
130.97
130.86
130.80
130.64
130.42
130.21
128.60
127.40
126.64
126.14
125.99
125.96
125.94
125.91
124.84
123.03
121.23
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.91
52.93
32.06
31.68
tBu
NH
O
O
CF3
3m
13C NMR (151 MHz, CDCl3)
f1 (ppm)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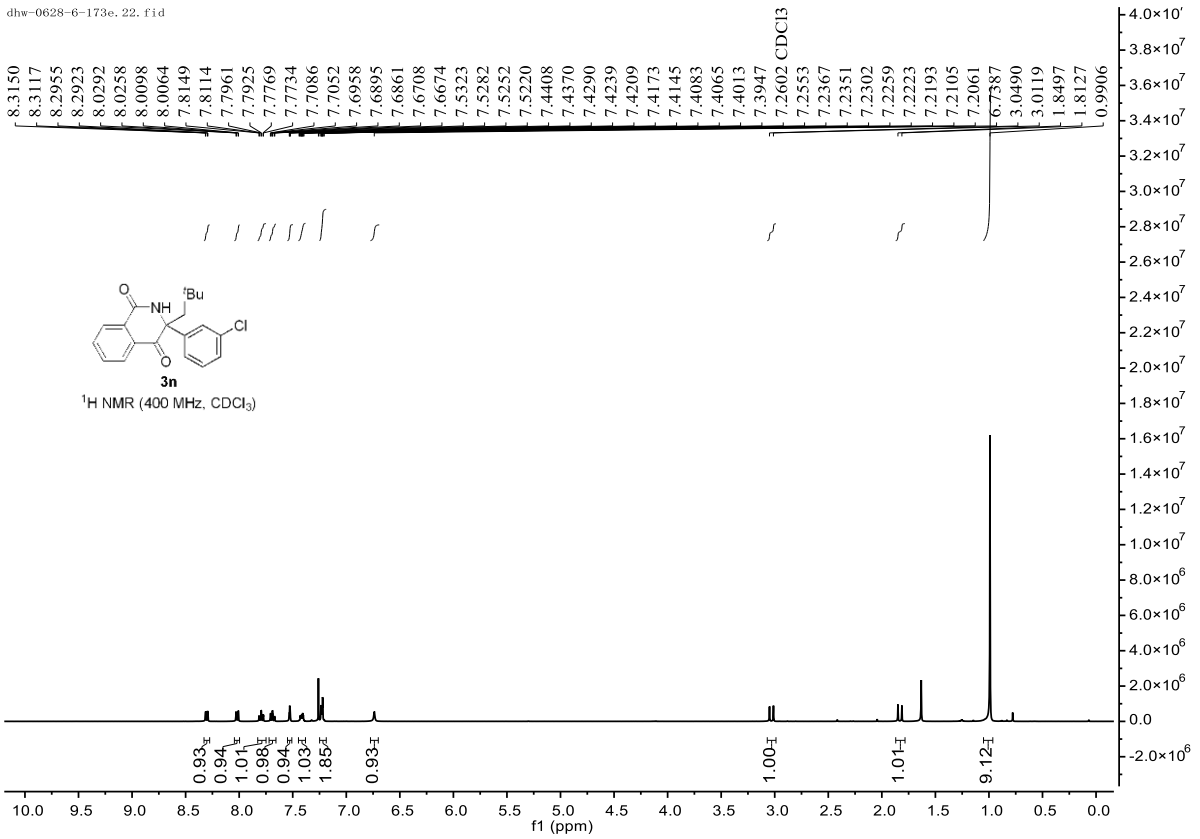
-62.7324
3m
19F NMR (376 MHz, CDCl3)
f1 (ppm)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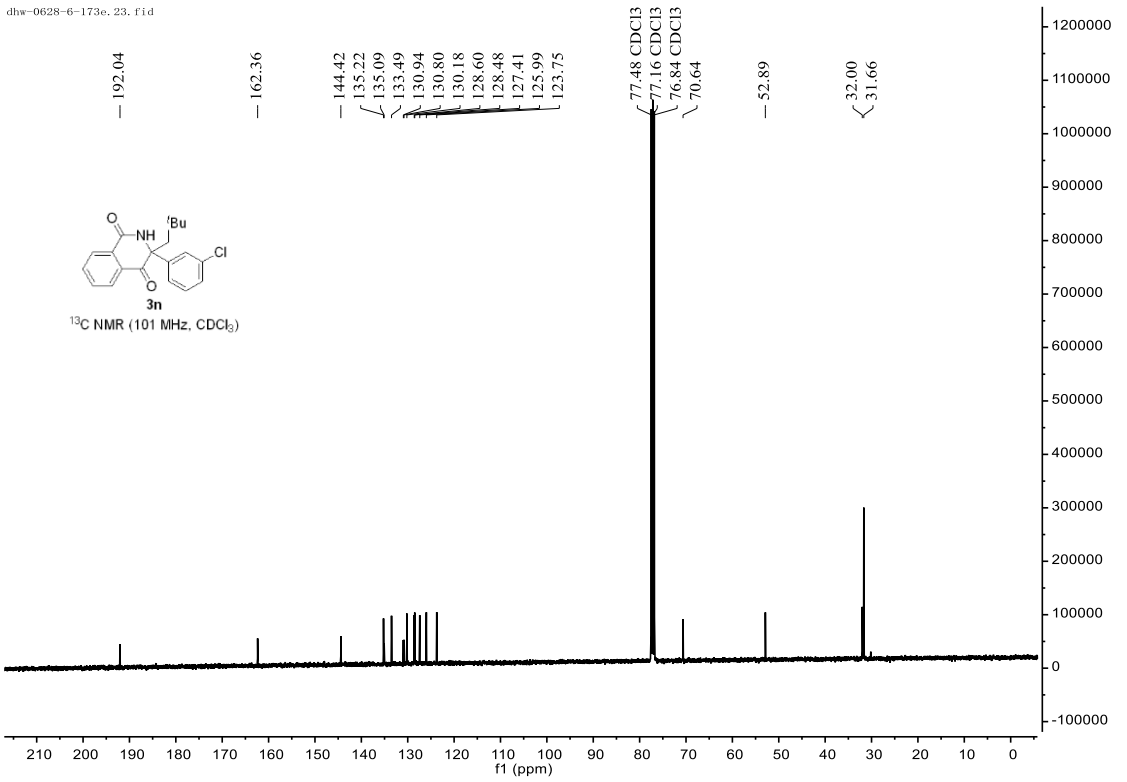
CDCl3
3n
1H NMR (400 MHz, CDCl3)
0.93
0.94
1.01
0.98
0.94
1.03
1.85
0.93
1.00
1.01
9.12
f1 (ppm)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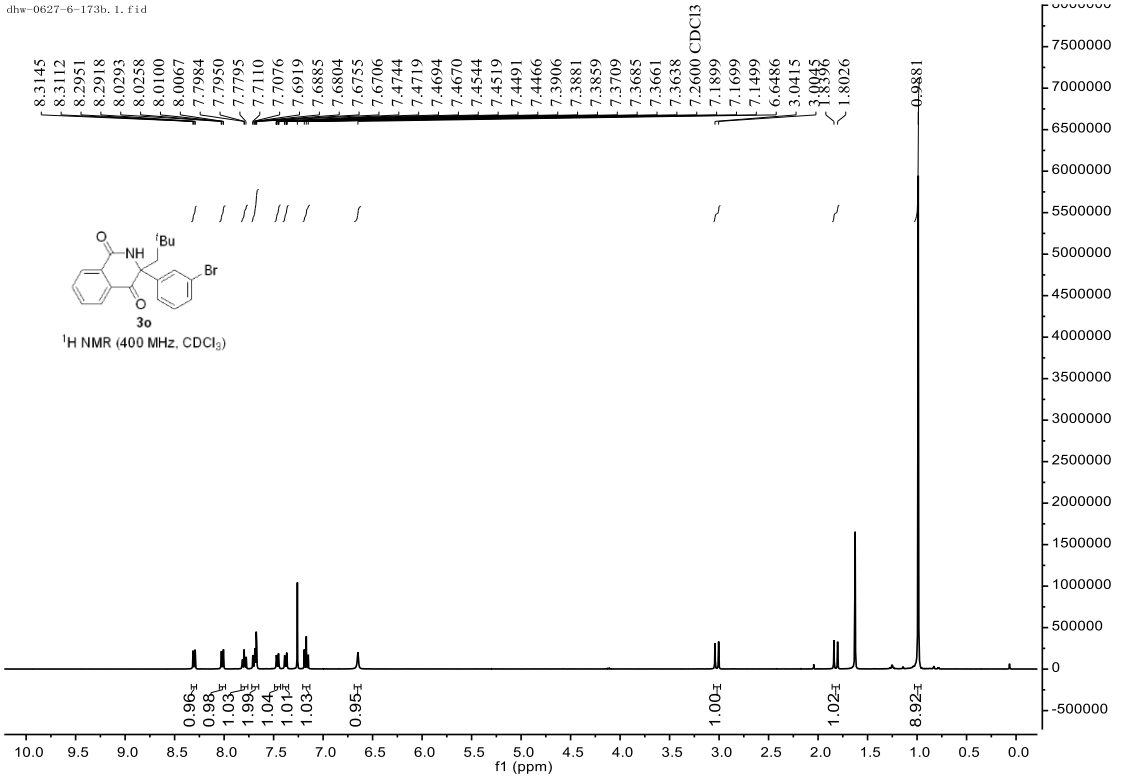
144.42
135.22
135.09
133.49
130.94
130.80
130.18
128.60
128.48
127.41
125.99
123.75
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.64
52.89
32.00
31.66
tBu
NH
Cl
O
3n
13C NMR (101 MHz, CDCl3)
f1 (ppm)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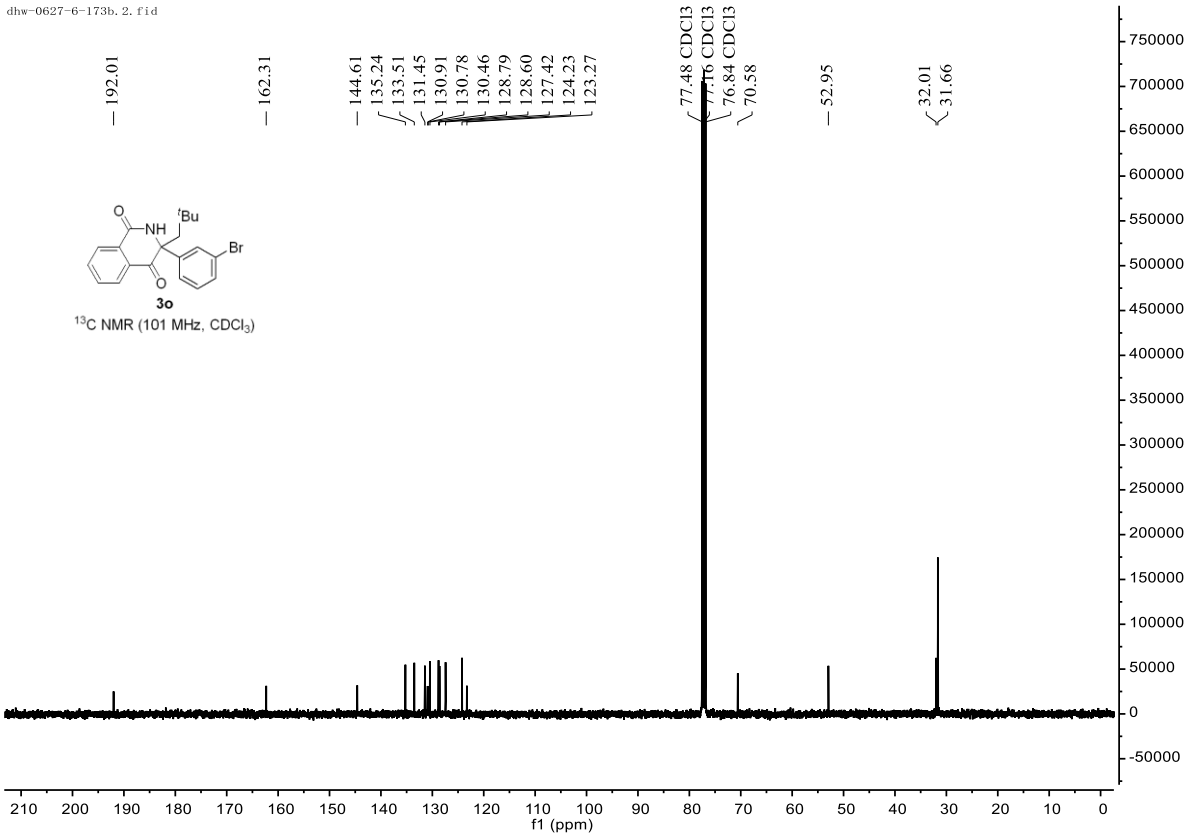
8.3112
8.2951
8.2918
8.0293
8.0258
8.0100
8.0067
7.7984
7.7950
7.7795
7.7110
7.7076
7.6919
7.6885
7.6804
7.6755
7.6706
7.4744
7.4719
7.4694
7.4670
7.4544
7.4519
7.4491
7.4466
7.3906
7.3881
7.3859
7.3709
7.3685
7.3661
7.3638
7.2600 CDCl3
7.1899
7.1699
7.1499
6.6486
3.0415
3.0045
1.8026
0.9881
tBu
NH
Br
O
3o
1H NMR (400 MHz, CDCl3)
0.96
0.98
1.03
1.99
1.04
1.01
1.03
0.95
1.00
1.02
8.92
f1 (ppm)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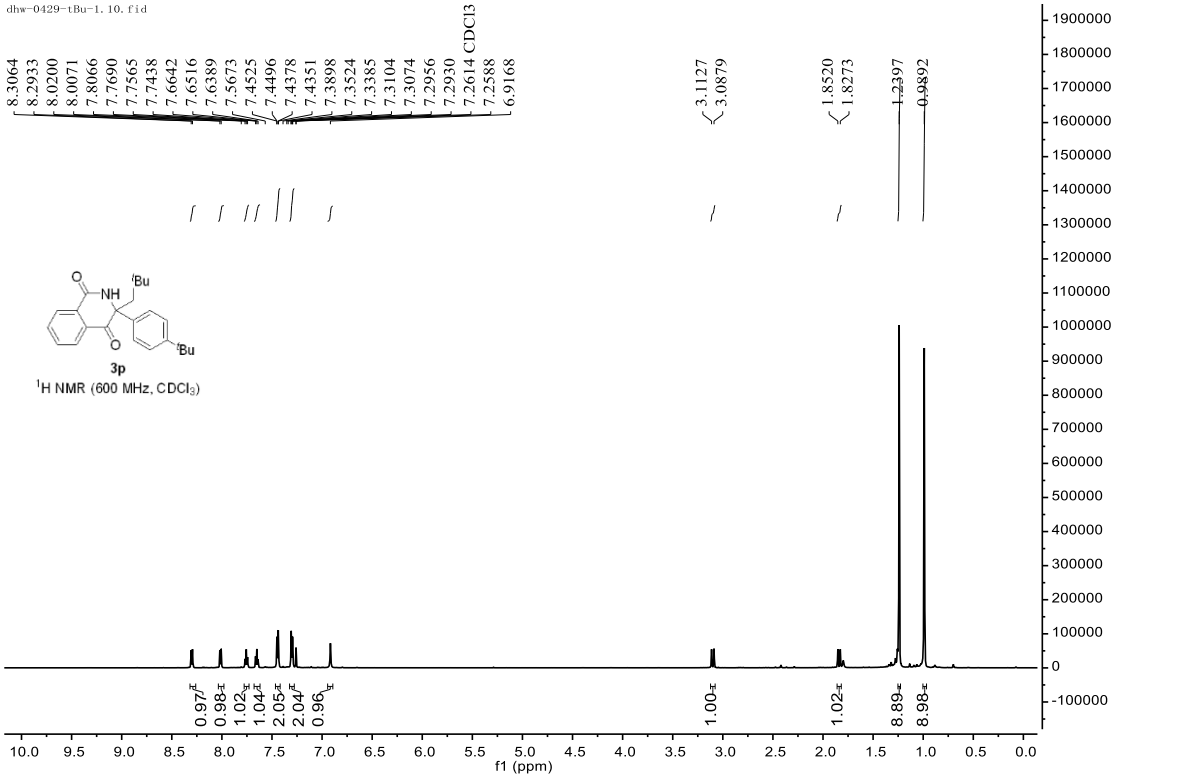
144.61
135.24
133.51
131.45
130.91
130.78
130.46
128.79
128.60
127.42
124.23
123.27
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.58
52.95
32.01
31.66
tBu
NH
Br
3o
13C NMR (101 MHz, CDCl3)
f1 (ppm)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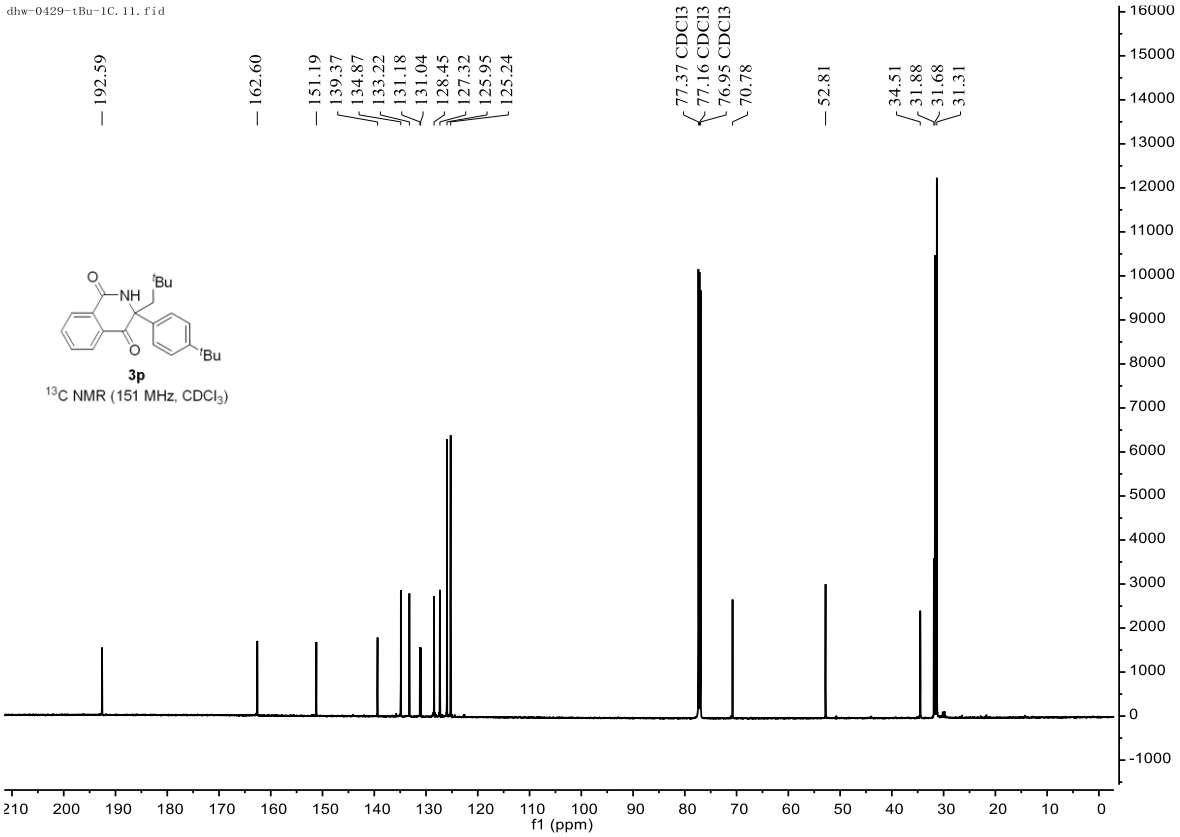
8.2933
8.0200
8.0071
7.8066
7.7690
7.7565
7.7438
7.6642
7.6516
7.6389
7.5673
7.4525
7.4496
7.4378
7.4351
7.3898
7.3524
7.3385
7.3104
7.3074
7.2956
7.2930
7.2614 CDCl3
7.2588
6.9168
3.1127
3.0879
1.8520
1.8273
1.2397
0.9892
0.97
0.98
1.02
1.04
2.05
2.04
0.96
1.00
1.02
8.89
8.98
tBu
NH
tBu
3p
1H NMR (600 MHz, CDCl3)
f1 (ppm)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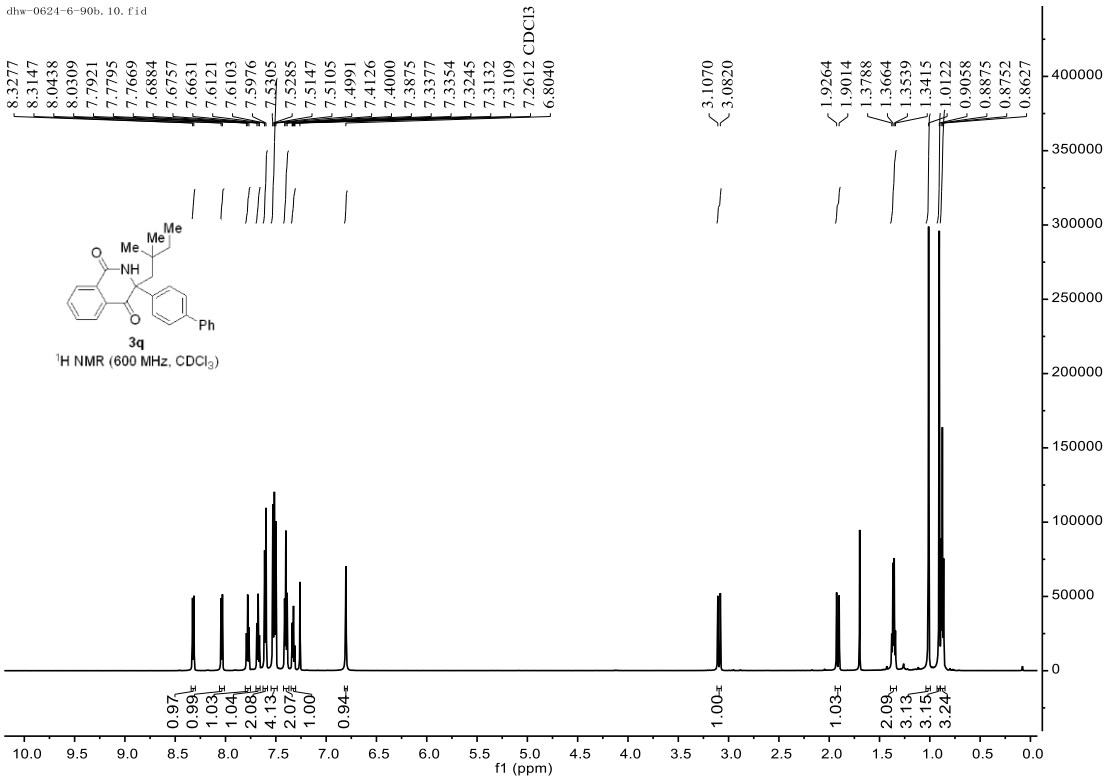
162.60
151.19
139.37
134.87
133.22
131.18
131.04
128.45
127.32
125.95
125.24
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.78
52.81
34.51
31.88
31.68
31.31
tBu
NH
O
tBu
3p
13C NMR (151 MHz, CDCl3)
f1 (ppm)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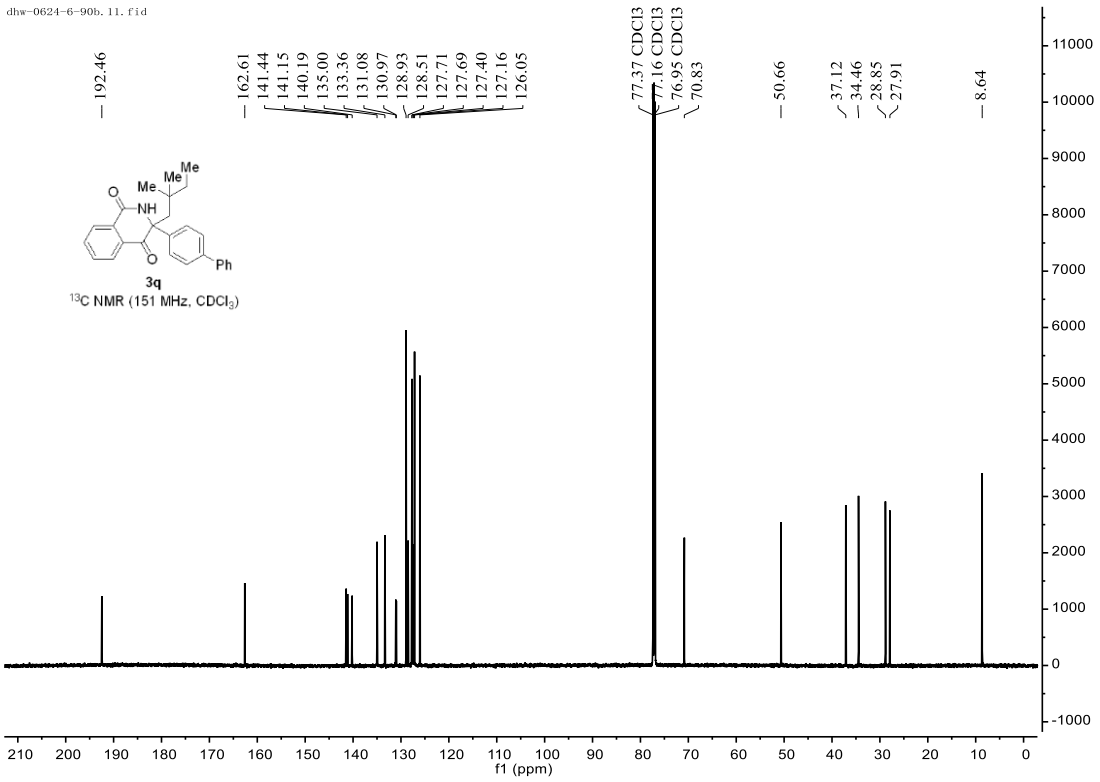
8.3147
8.0438
8.0309
7.7921
7.7795
7.7669
7.6884
7.6757
7.6631
7.6121
7.6103
7.5976
7.5305
7.5285
7.5147
7.5105
7.4991
7.4126
7.4000
7.3875
7.3377
7.3354
7.3245
7.3132
7.3109
7.2612 CDCl3
6.8040
3.1070
3.0820
1.9264
1.9014
1.3788
1.3664
1.3539
1.3415
1.0122
0.9058
0.8875
0.8752
0.8627
Me
Me
Me
NH
O
O
Ph
3q
1H NMR (600 MHz, CDCl3)
0.97
0.99
1.03
1.04
2.08
4.13
2.07
1.00
0.94
1.00
1.03
2.09
3.13
3.15
3.24
f1 (ppm)


dhw-0624-6-90b.11.fid
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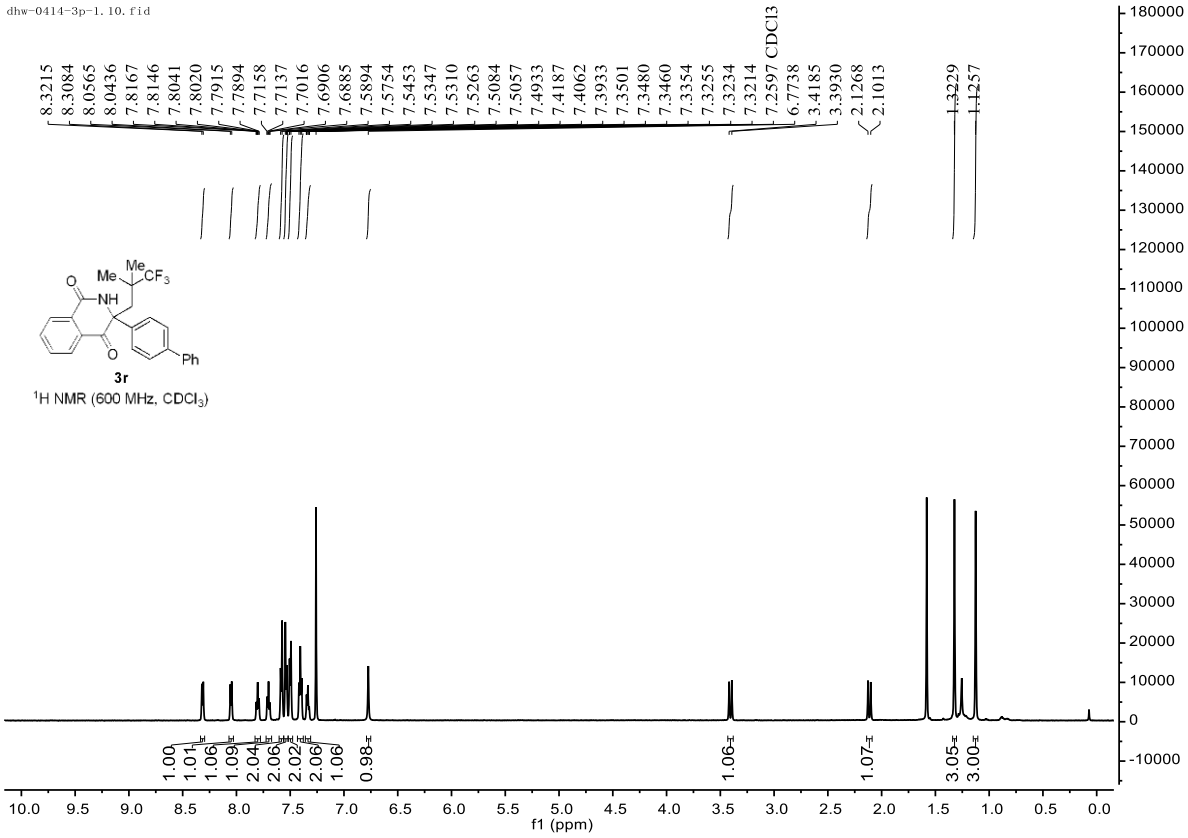
162.61
141.44
141.15
140.19
135.00
133.36
131.08
130.97
128.93
128.51
127.71
127.69
127.40
127.16
126.05
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.83
50.66
37.12
34.46
28.85
27.91
8.64
Me
Me
NH
O
O
Ph
3q
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0414-3p-1.10.fid
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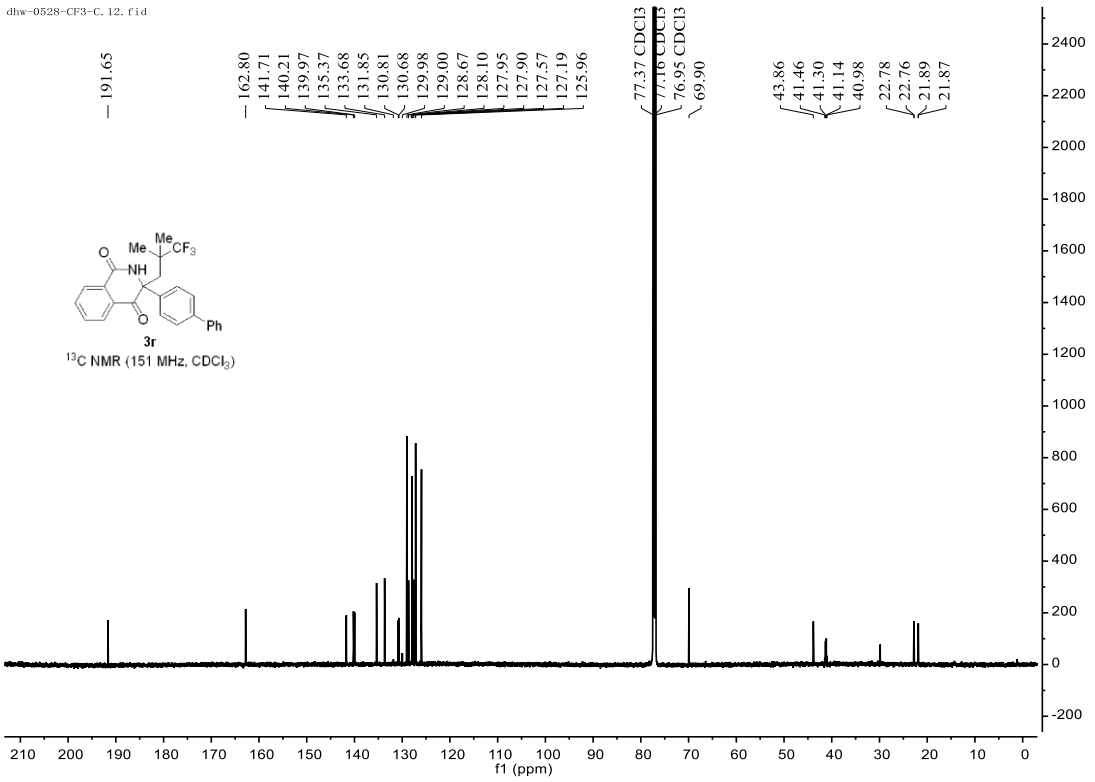
8.3084
8.0565
8.0436
7.8167
7.8146
7.8041
7.8020
7.7915
7.7894
7.7158
7.7137
7.7016
7.6906
7.6885
7.5894
7.5754
7.5453
7.5347
7.5310
7.5263
7.5084
7.5057
7.4933
7.4187
7.4062
7.3933
7.3501
7.3480
7.3460
7.3354
7.3255
7.3234
7.3214
7.2597 CDCl3
6.7738
3.4185
3.3930
2.1268
2.1013
1.3229
1.1257
Me
CF3
NH
O
O
Ph
3r
1H NMR (600 MHz, CDCl3)
1.00
1.01
1.06
1.09
2.04
2.06
2.02
2.06
1.06
0.98
1.06
1.07
3.05
3.00
f1 (ppm)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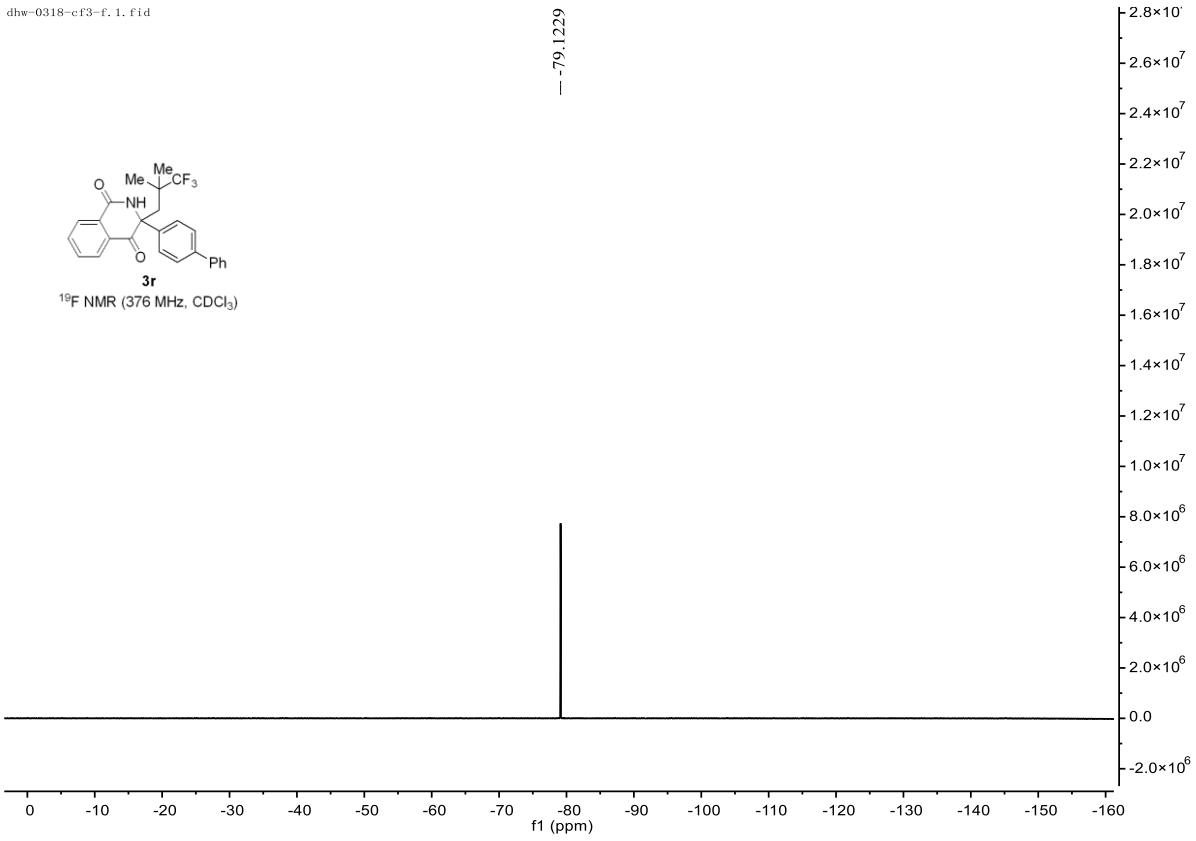
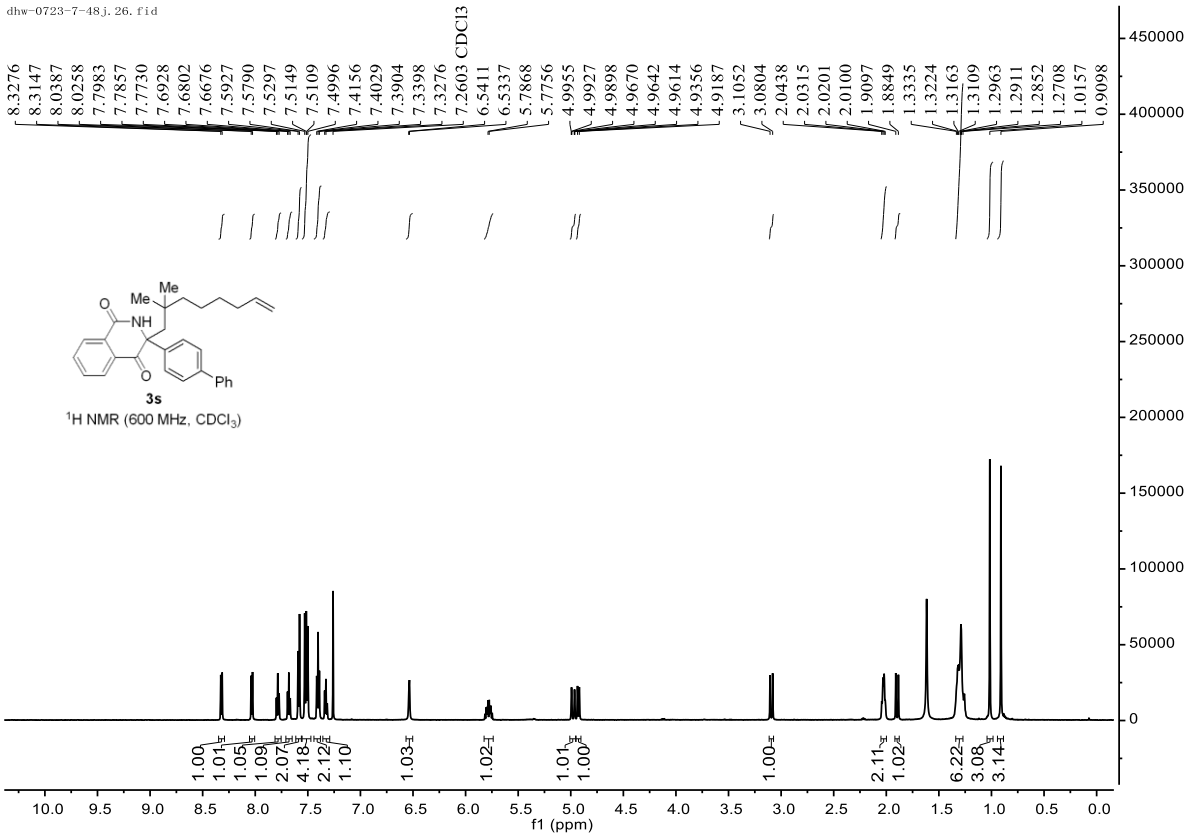
162.80
141.71
140.21
139.97
135.37
133.68
131.85
130.81
130.68
129.98
129.00
128.67
128.10
127.95
127.90
127.57
127.19
125.96
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
69.90
43.86
41.46
41.30
41.14
40.98
22.78
22.76
21.89
21.87
Me
CF3
NH
O
Ph
3r
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0318-cf3-f.1.fid
-79.1229
Me
CF3
NH
O
Ph
3r
19F NMR (376 MHz, CDCl3)
f1 (ppm)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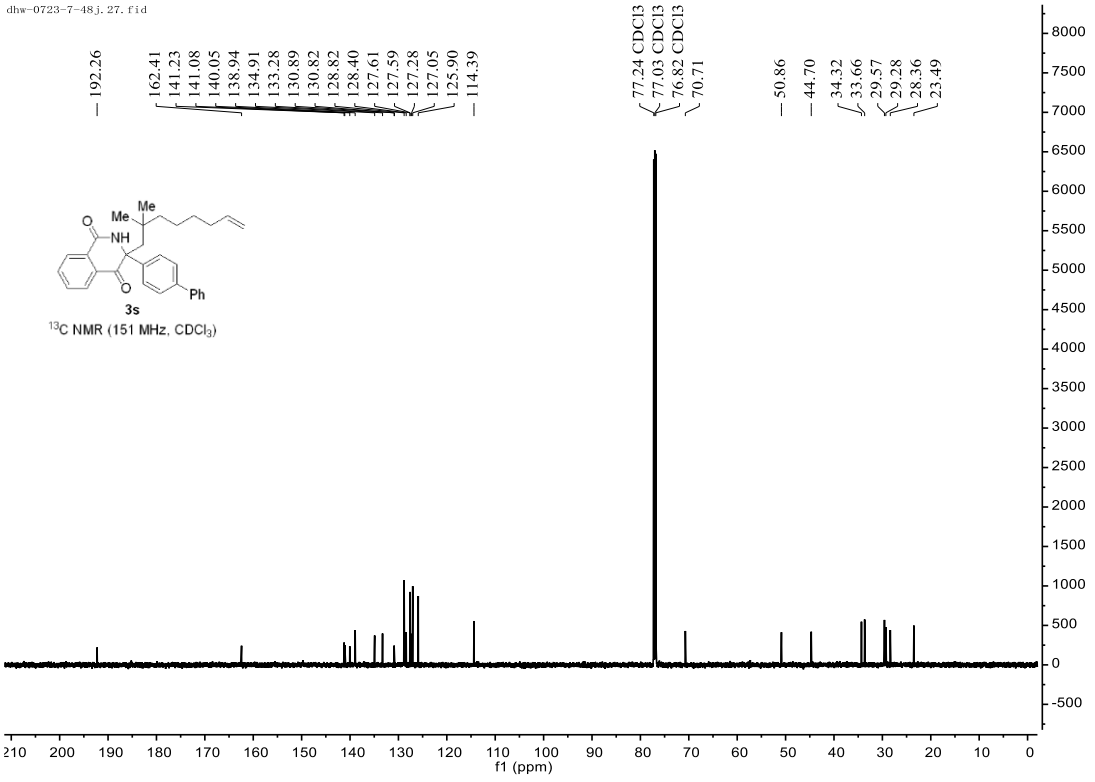
8.0387
8.0258
7.7983
7.7857
7.7730
7.6928
7.6802
7.6676
7.5927
7.5790
7.5297
7.5149
7.5109
7.4996
7.4156
7.4029
7.3904
7.3398
7.3276
7.2603 CDCl3
6.5411
6.5337
5.7868
5.7756
4.9955
4.9927
4.9898
4.9670
4.9642
4.9614
4.9356
4.9187
3.1052
3.0804
2.0438
2.0315
2.0201
2.0100
1.9097
1.8849
1.3335
1.3224
1.3163
1.3109
1.2963
1.2911
1.2852
1.2708
1.0157
0.9098
Me
NH
O
O
Ph
3s
1H NMR (600 MHz, CDCl3)
1.00
1.01
1.05
1.09
2.07
4.18
2.12
1.10
1.03
1.02
1.01
1.00
1.00
2.11
1.02
6.22
3.08
3.14
f1 (ppm)


dhw-0723-7-48j.27.fid
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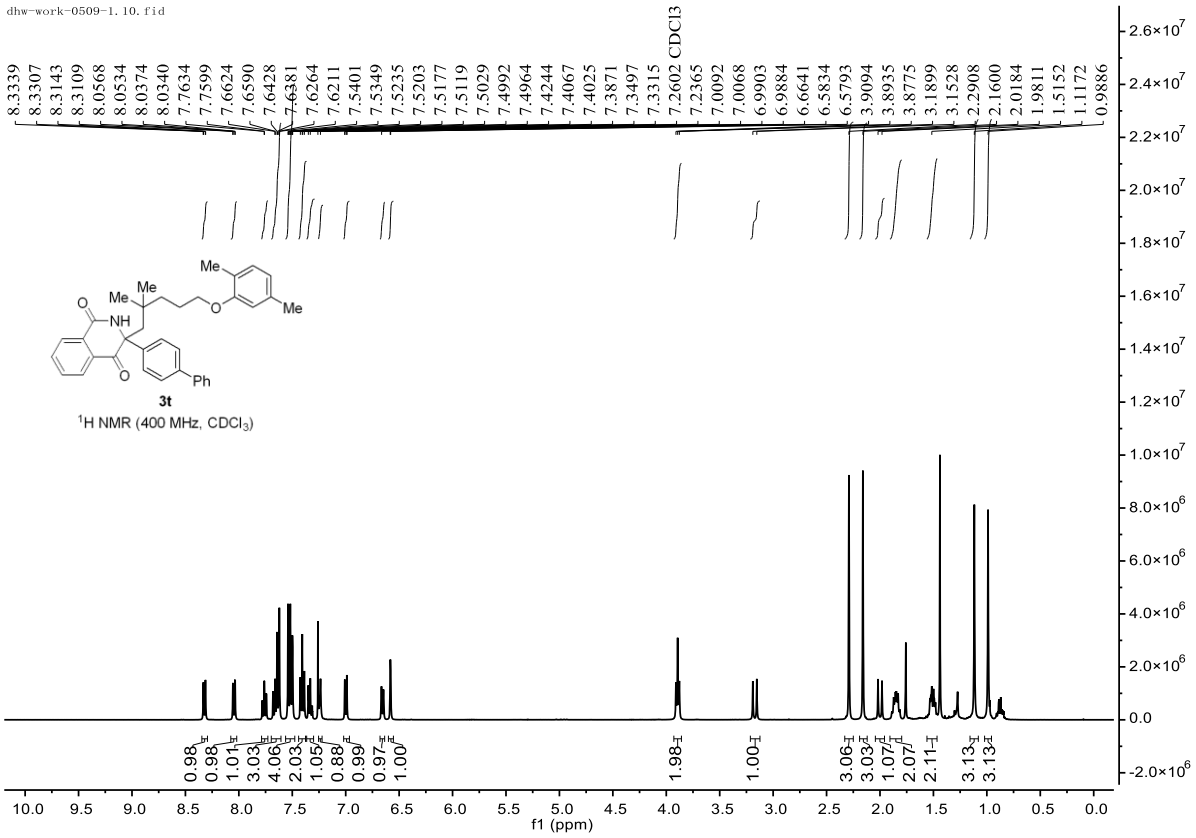
141.23
141.08
140.05
138.94
134.91
133.28
130.89
130.82
128.82
128.40
127.61
127.59
127.28
127.05
125.90
114.39
77.24 CDCl3
77.03 CDCl3
76.82 CDCl3
70.71
50.86
44.70
34.32
33.66
29.57
29.28
28.36
23.49
Me
NH
O
O
Ph
3s
13C NMR (151 MHz, CDCl3)
f1 (ppm)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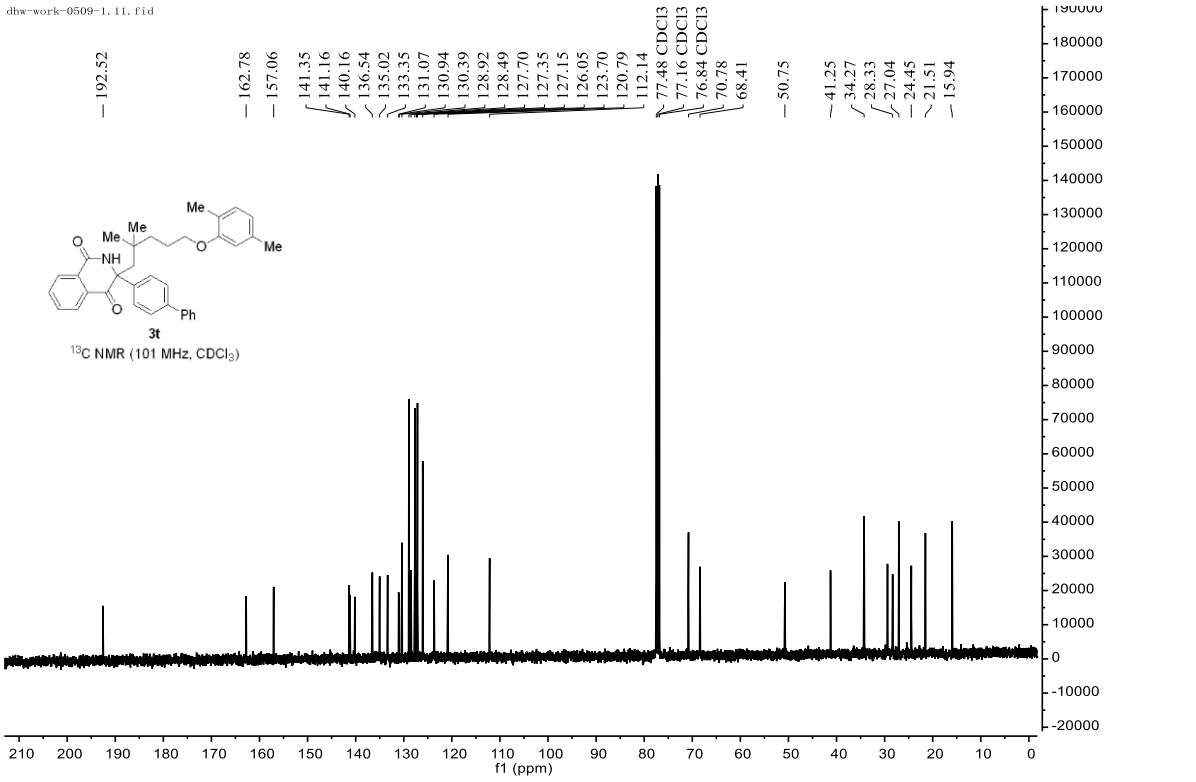
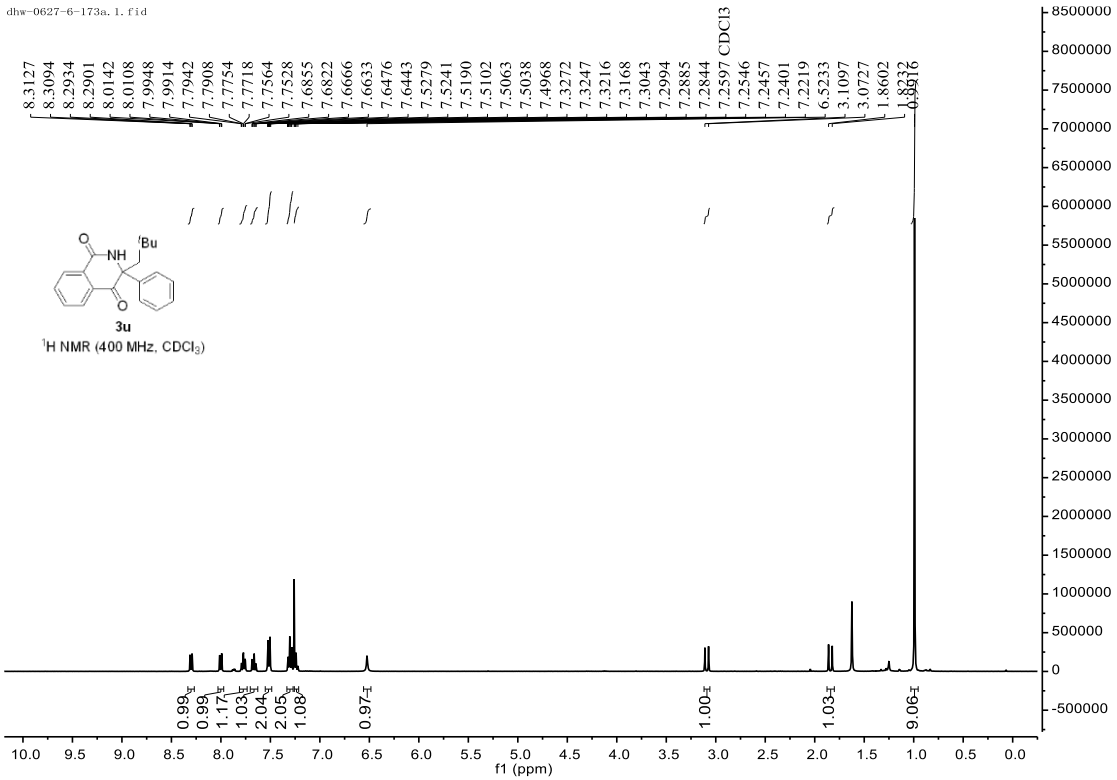
3t
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)


dhw-work-0509-1.11.fid
3t
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)


dhw-0627-6-173a.1.fid
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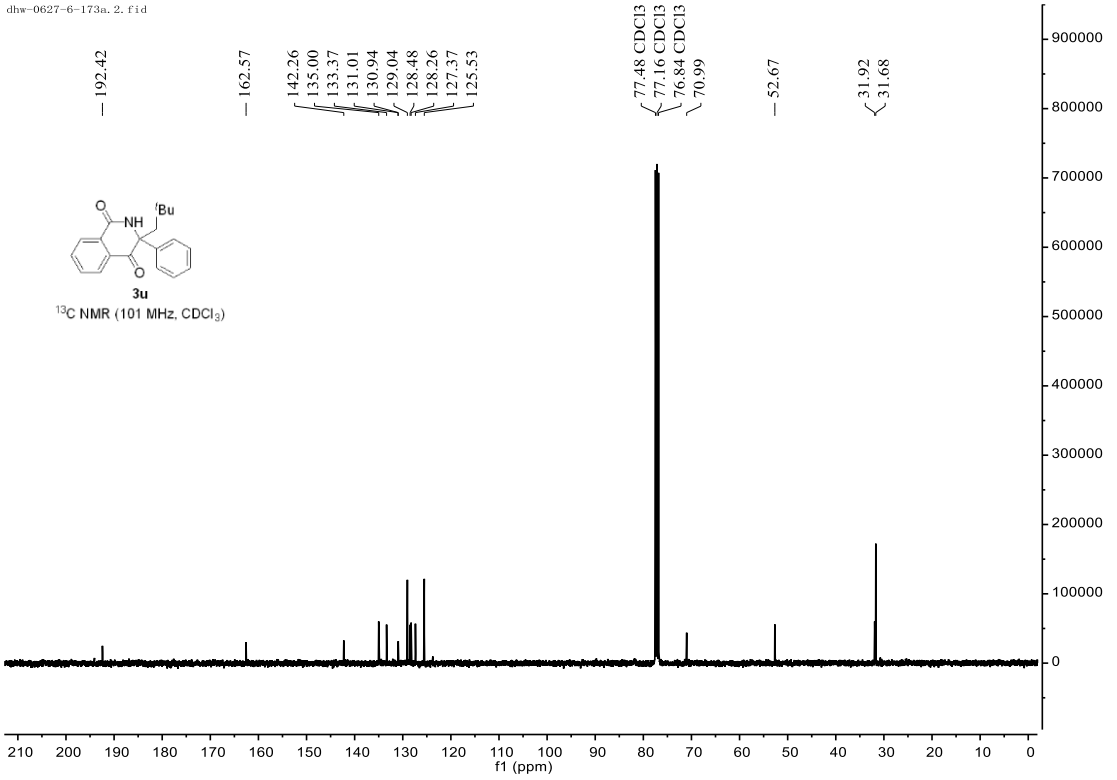
8.3094
8.2934
8.2901
8.0142
8.0108
7.9948
7.9914
7.7942
7.7908
7.7754
7.7718
7.7564
7.7528
7.6855
7.6822
7.6666
7.6633
7.6476
7.6443
7.5279
7.5241
7.5190
7.5102
7.5063
7.5038
7.4968
7.3272
7.3247
7.3216
7.3168
7.3043
7.2994
7.2885
7.2844
7.2597 CDCl3
7.2546
7.2457
7.2401
7.2219
6.5233
3.1097
3.0727
1.8602
0.9316
tBu
NH
O
O
3u
1H NMR (400 MHz, CDCl3)
0.99
0.99
1.17
1.03
2.04
2.05
1.08
0.97
1.00
1.03
9.06
f1 (ppm)


dhw-0627-6-173a.2.fid
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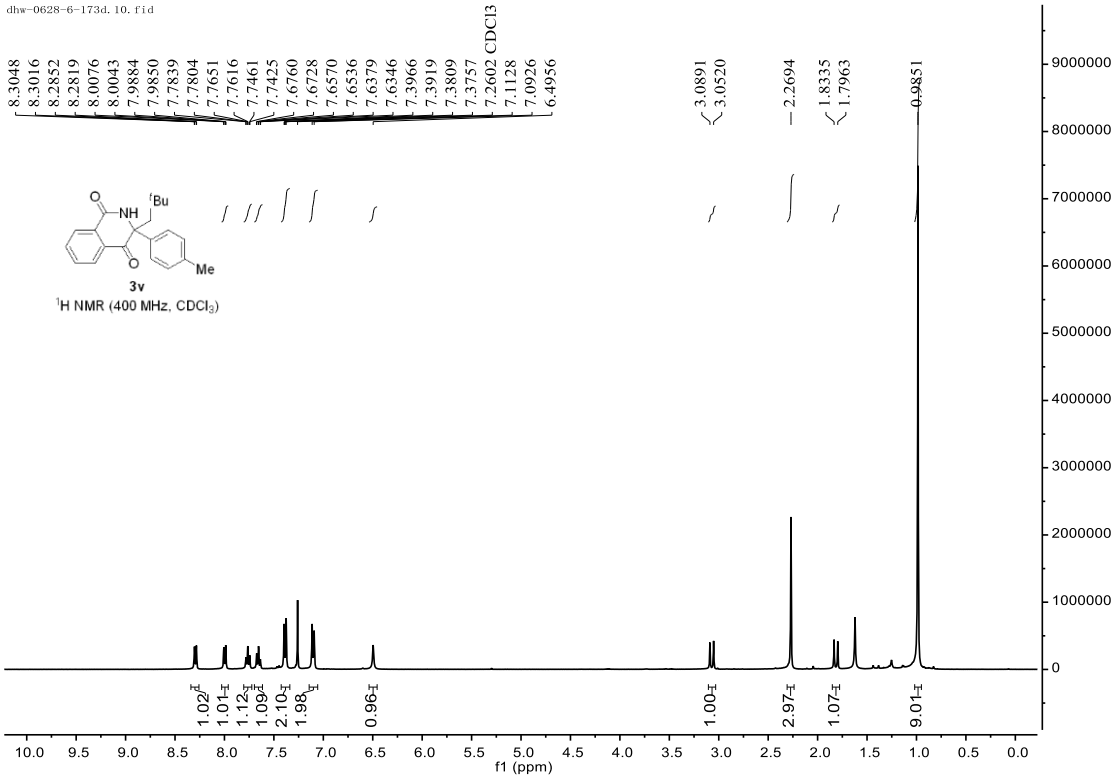
162.57
142.26
135.00
133.37
131.01
130.94
129.04
128.48
128.26
127.37
125.53
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.99
52.67
31.92
31.68
tBu
NH
O
O
3u
13C NMR (101 MHz, CDCl3)
f1 (ppm)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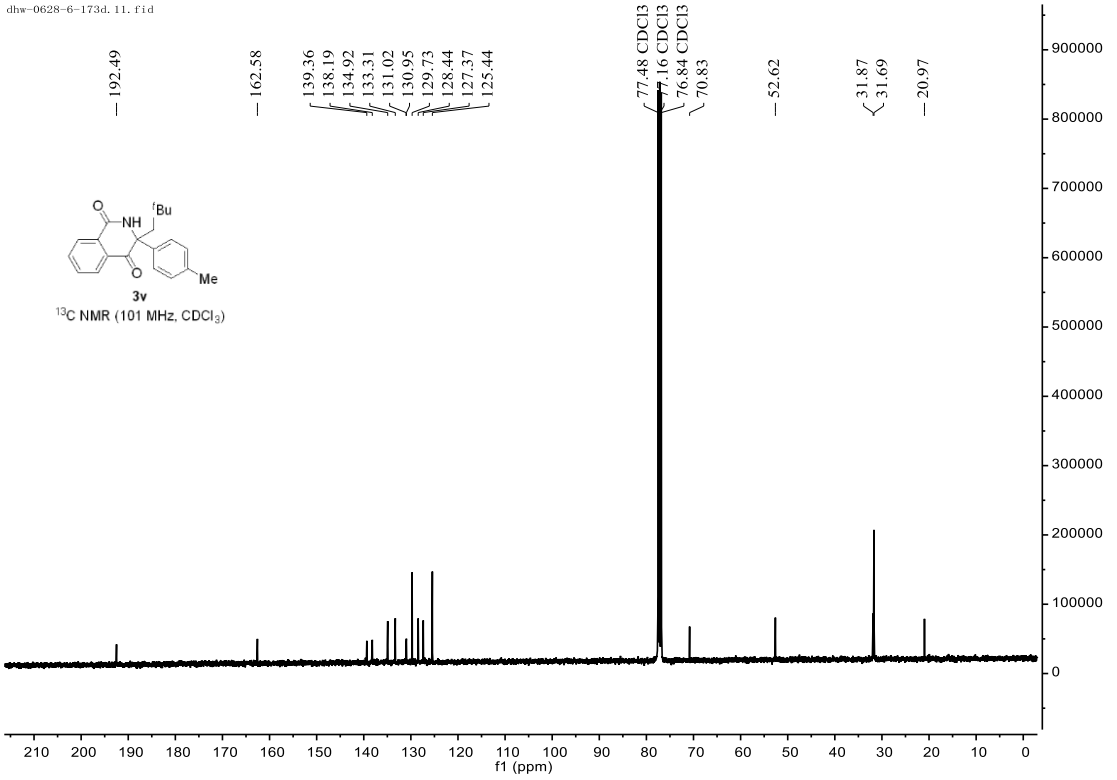
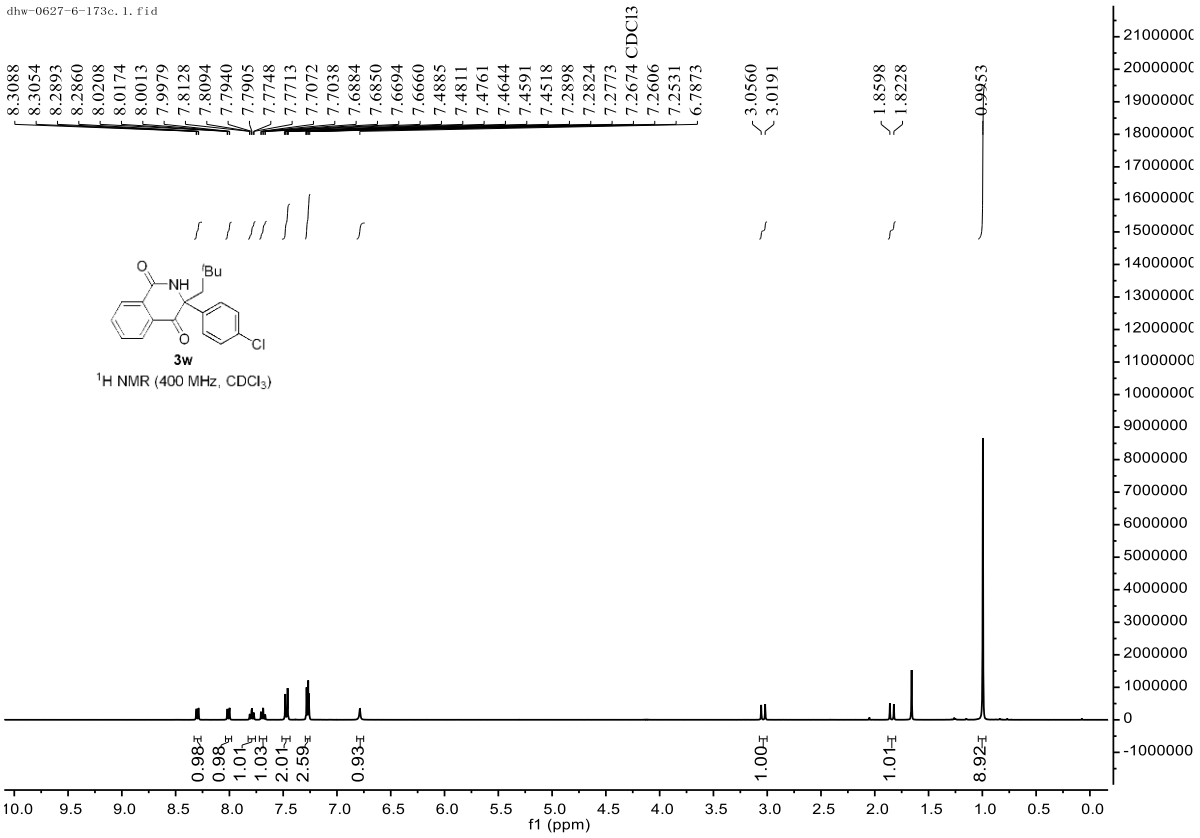
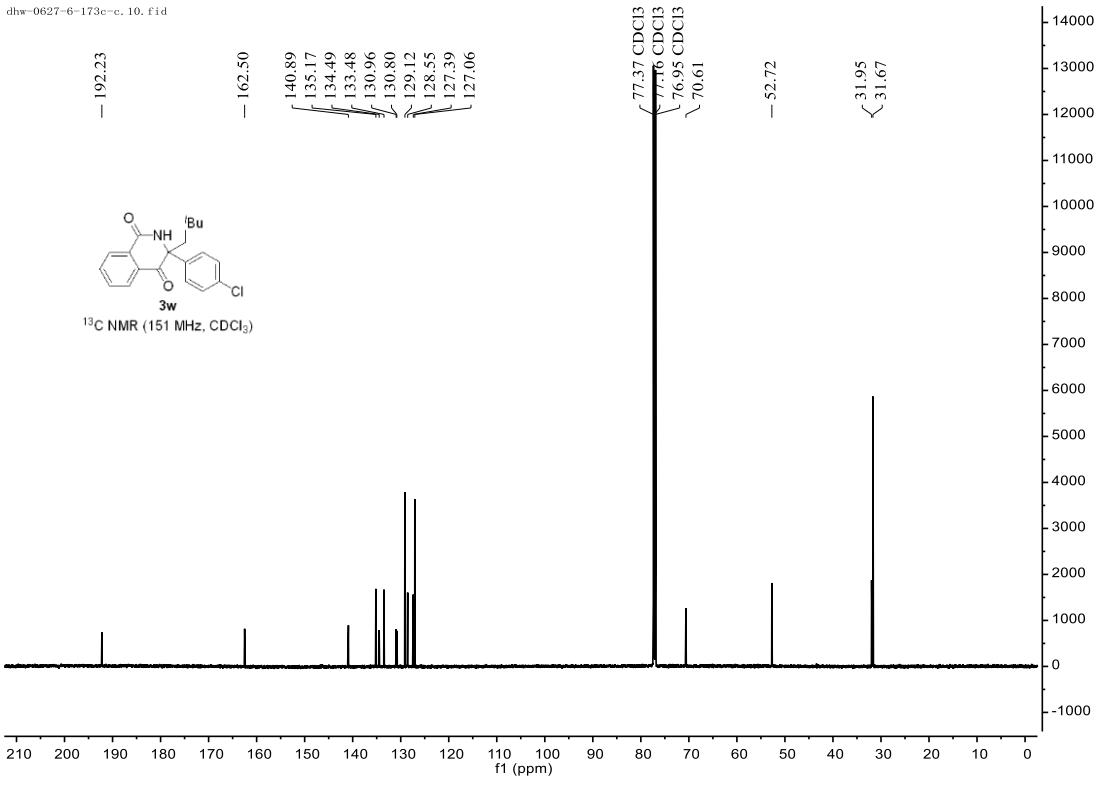
8.2852
8.2819
8.0076
8.0043
7.9884
7.9850
7.7839
7.7804
7.7651
7.7616
7.7461
7.7425
7.6760
7.6728
7.6570
7.6536
7.6379
7.6346
7.3966
7.3919
7.3809
7.3757
7.2602 CDCl3
7.1128
7.0926
6.4956
3.0891
3.0520
2.2694
1.8335
1.7963
0.9851
1.02
1.01
1.12
1.09
2.10
1.98
0.96
1.00
2.97
1.07
9.01
f1 (ppm)
3v
1H NMR (400 MHz, CDCl3)


dhw-0628-6-173d.11.fid
192.49
162.58
139.36
138.19
134.92
133.31
131.02
130.95
129.73
128.44
127.37
125.44
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
70.83
52.62
31.87
31.69
20.97
f1 (ppm)
3v
13C NMR (101 MHz, CDCl3)



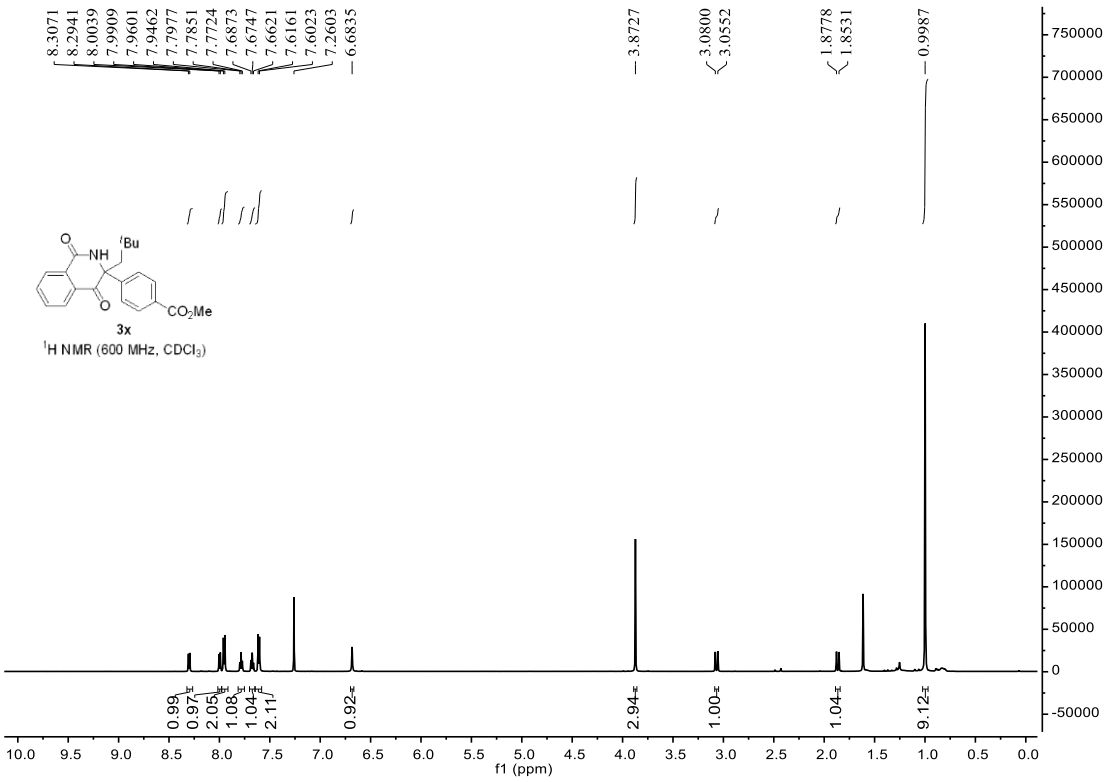
3w

^{1}H NMR (400 MHz, $CDCl_3$)



3w

^{13}C NMR (151 MHz, $CDCl_3$)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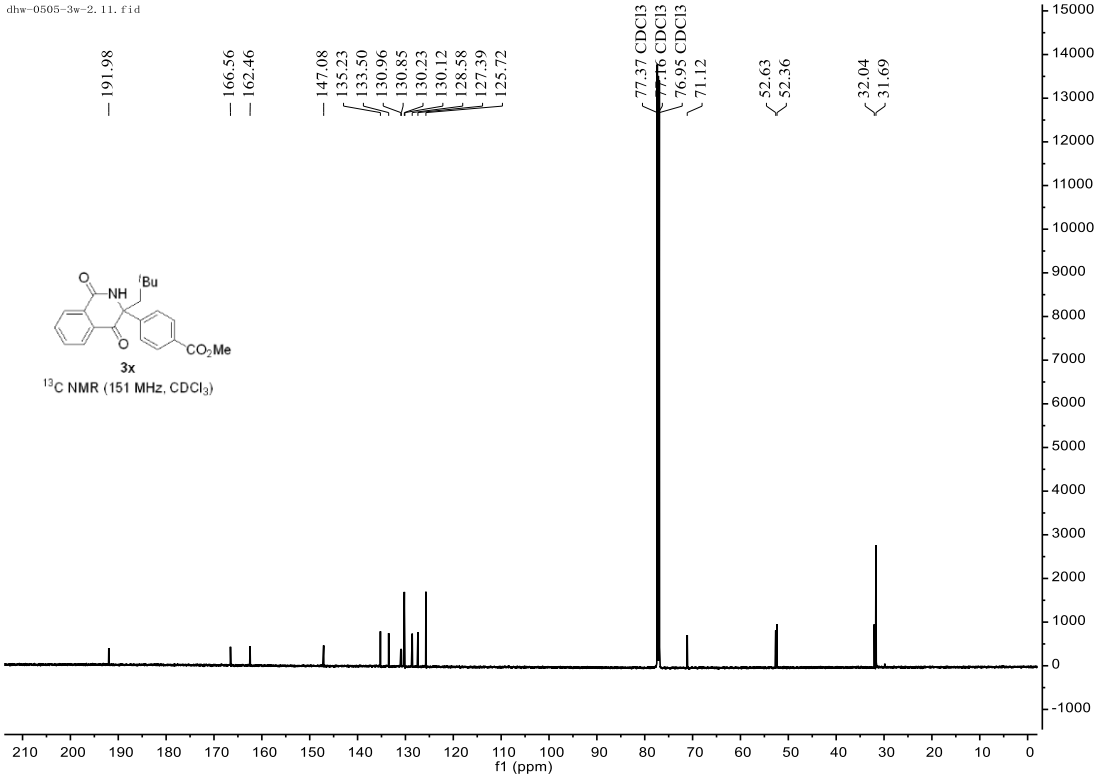
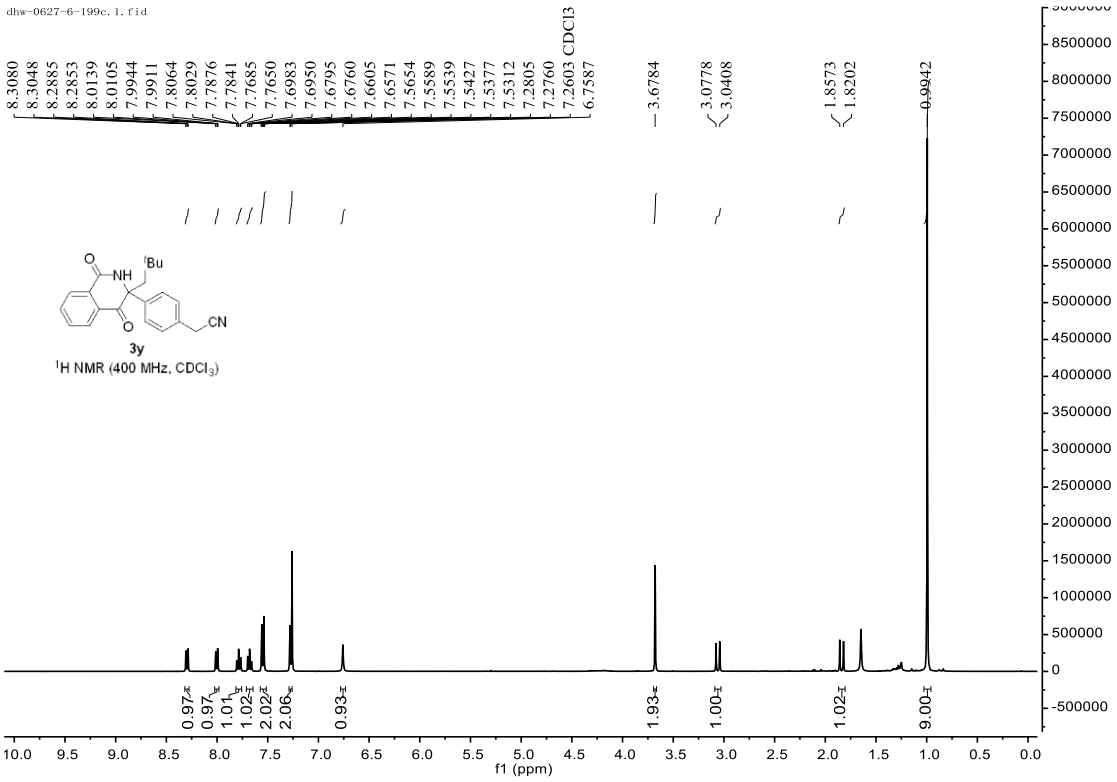
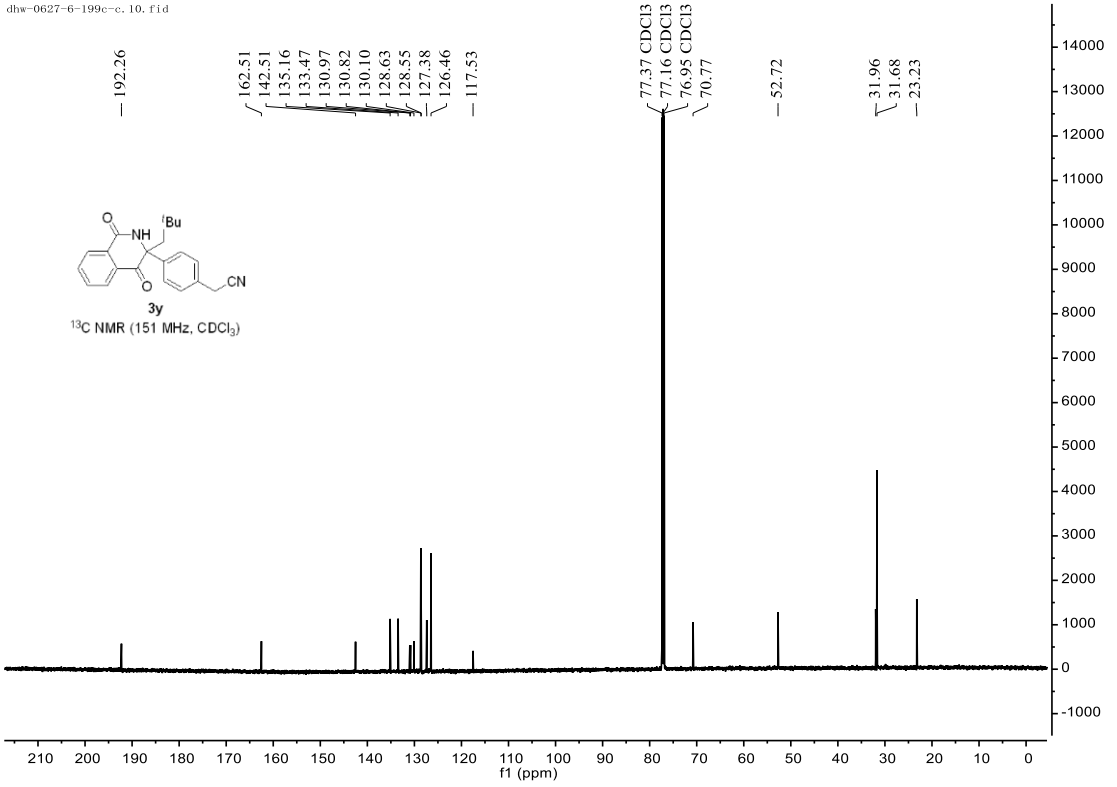
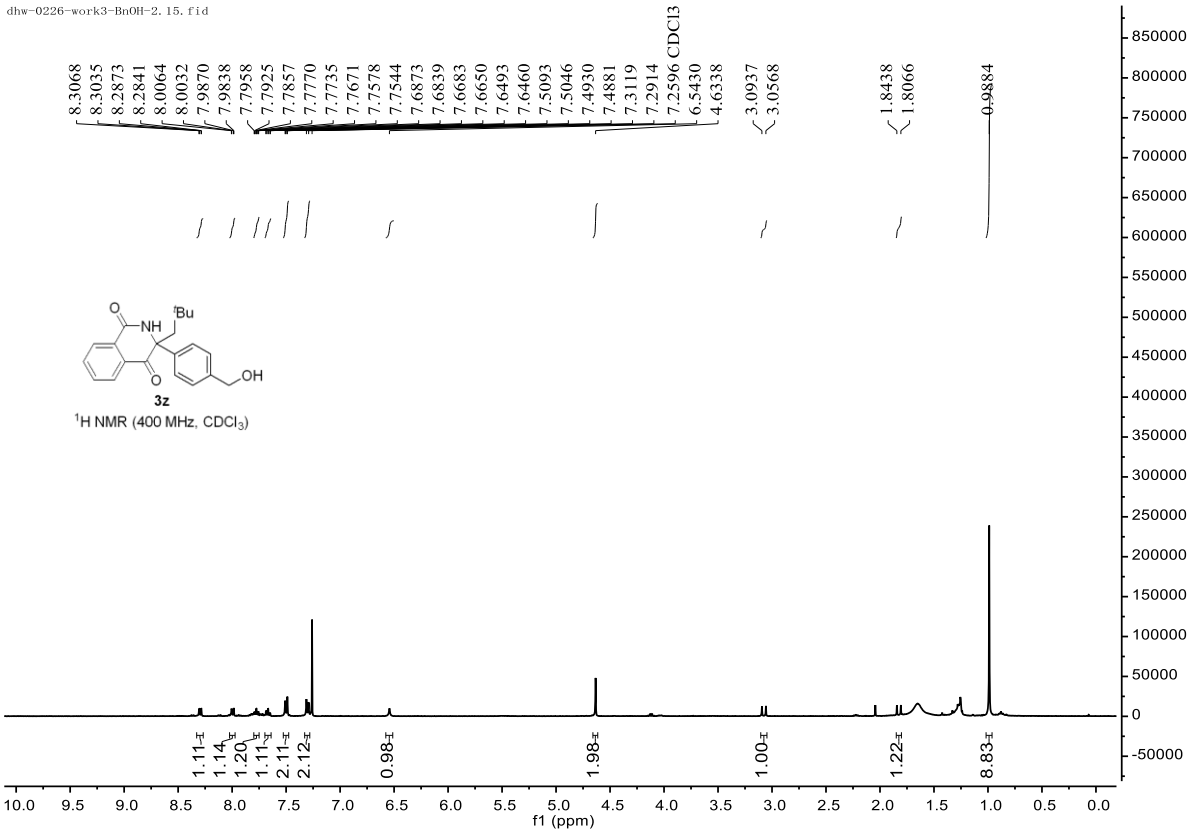
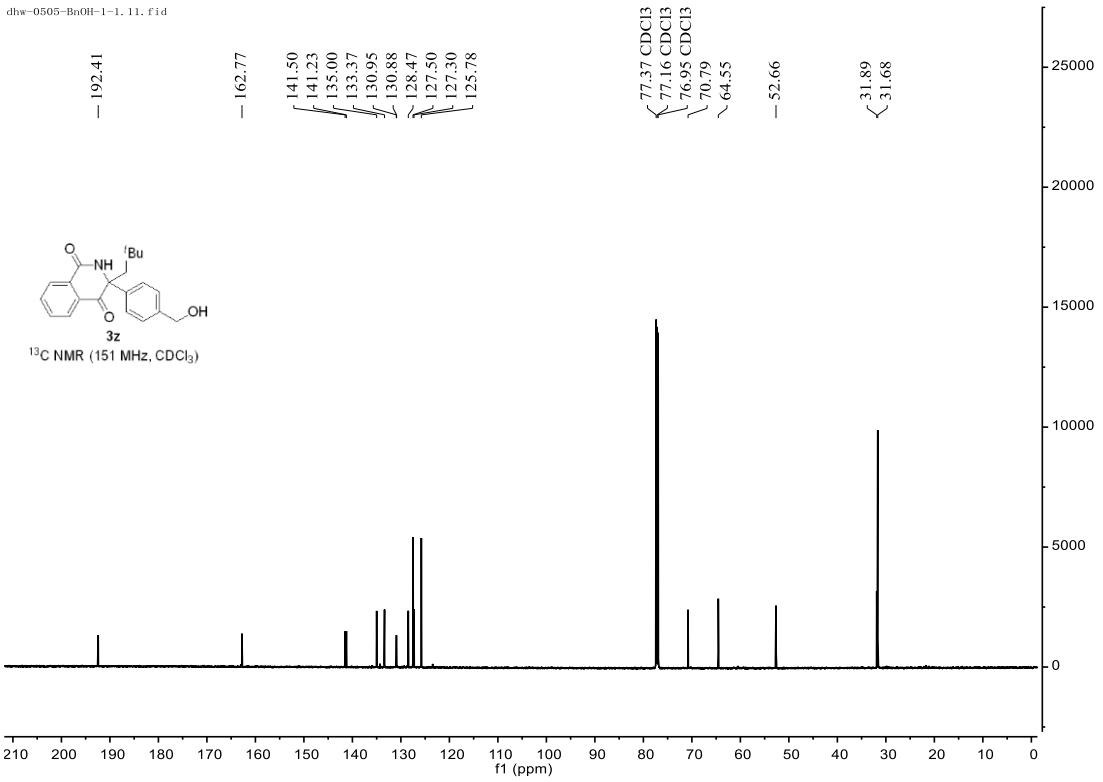
8.2941
8.0039
7.9909
7.9601
7.9462
7.7977
7.7851
7.7724
7.6873
7.6747
7.6621
7.6161
7.6023
7.2603
6.6835
3.8727
3.0800
3.0552
1.8778
1.8531
0.9987
tBu
NH
O
O
CO2Me
3x
1H NMR (600 MHz, CDCl3)
0.99
0.97
2.05
1.08
1.04
2.11
0.92
2.94
1.00
1.04
9.12
f1 (ppm)

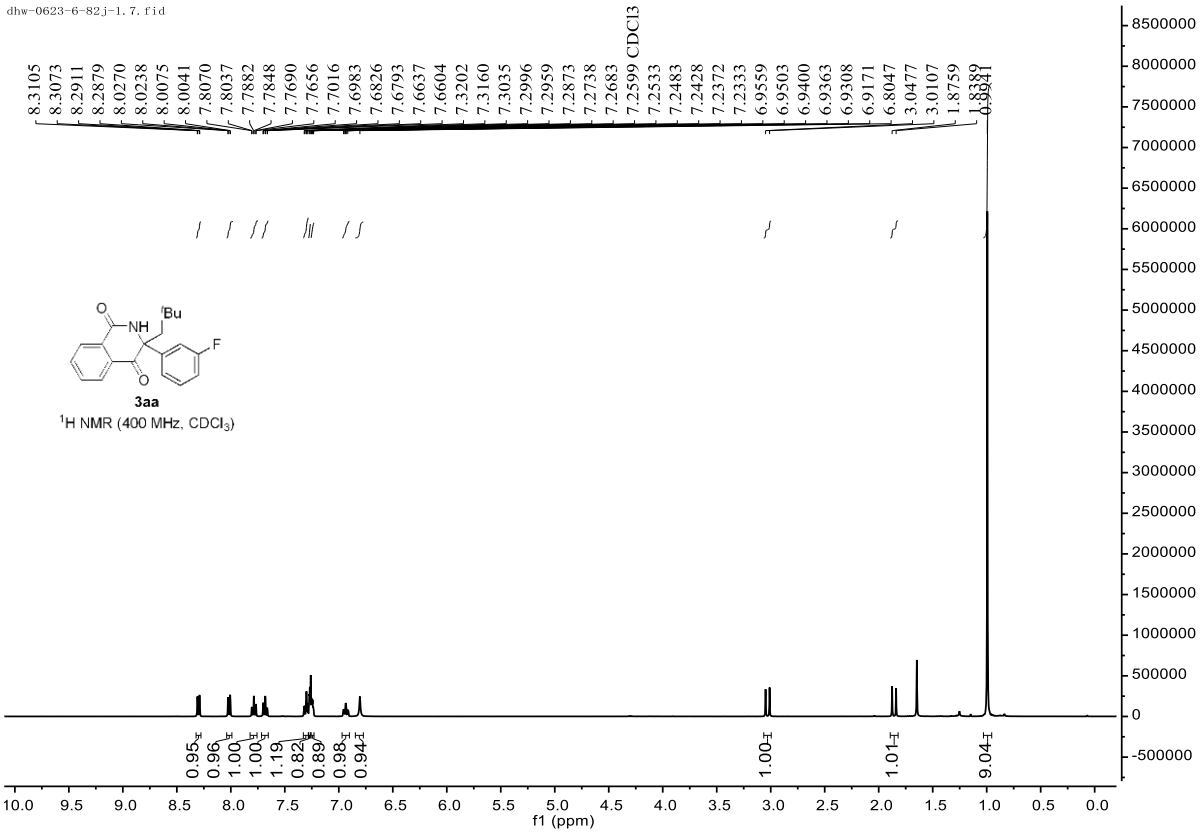

dhw-0505-3w-2.11.fid
191.98
166.56
162.46
147.08
135.23
133.50
130.96
130.85
130.23
130.12
128.58
127.39
125.72
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
71.12
52.63
52.36
32.04
31.69
tBu
NH
O
O
CO2Me
3x
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0627-6-199c.1.fid
8.3080
8.3048
8.2885
8.2853
8.0139
8.0105
7.9944
7.9911
7.8064
7.8029
7.7876
7.7841
7.7685
7.7650
7.6983
7.6950
7.6795
7.6760
7.6605
7.6571
7.5654
7.5589
7.5539
7.5427
7.5377
7.5312
7.2805
7.2760
7.2603 CDCl3
6.7587
3.6784
3.0778
3.0408
1.8573
1.8202
0.9942
0.97
0.97
1.01
1.02
2.02
2.06
0.93
1.93
1.00
1.02
9.00
f1 (ppm)
3y
1H NMR (400 MHz, CDCl3)


dhw-0627-6-199c-c.10.fid
192.26
162.51
142.51
135.16
133.47
130.97
130.82
130.10
128.63
128.55
127.38
126.46
117.53
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.77
52.72
31.96
31.68
23.23
f1 (ppm)
3y
13C NMR (151 MHz, CDCl3)

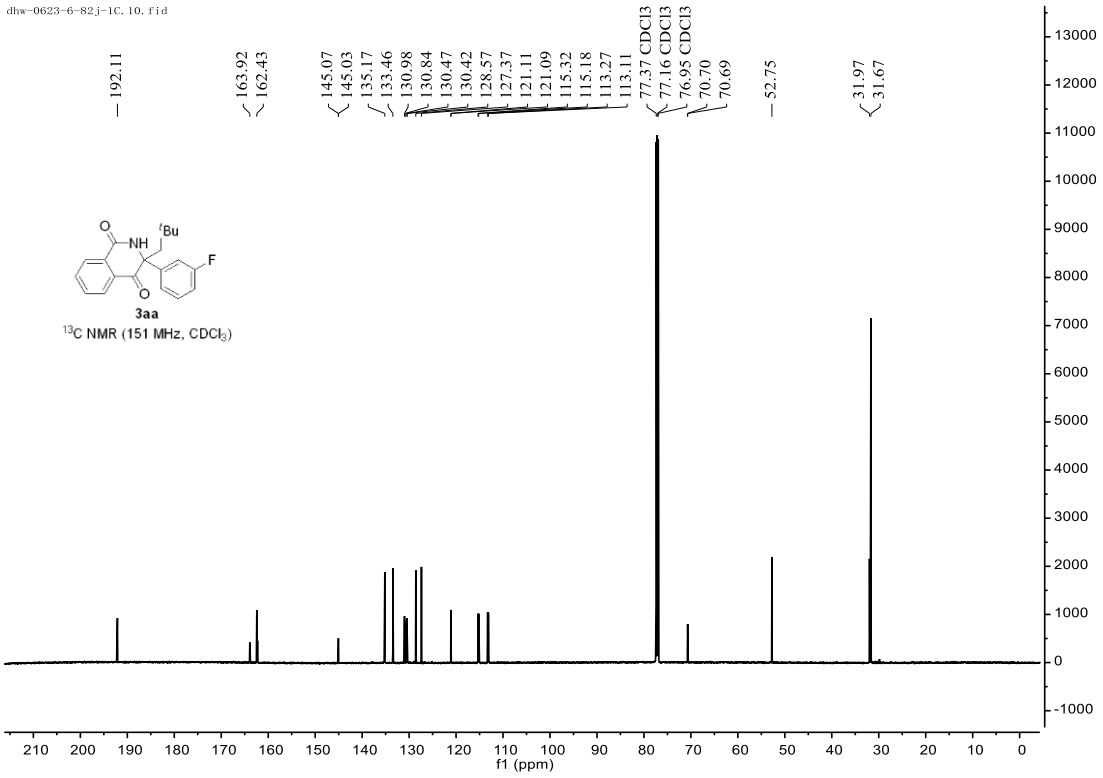

dhw-0226-work3-BnOH-2.15.fid
8.3068
8.3035
8.2873
8.2841
8.0064
8.0032
7.9870
7.9838
7.7958
7.7925
7.7857
7.7770
7.7735
7.7671
7.7578
7.7544
7.6873
7.6839
7.6683
7.6650
7.6493
7.6460
7.5093
7.5046
7.4930
7.4881
7.3119
7.2914
7.2596 CDCl3
6.5430
4.6338
3.0937
3.0568
1.8438
1.8066
0.9884
tBu
NH
O
OH
3z
1H NMR (400 MHz, CDCl3)
1.11
1.14
1.20
1.11
2.11
2.12
0.98
1.98
1.00
1.22
8.83
f1 (ppm)


dhw-0505-BnOH-1-1.11.fid
192.41
162.77
141.50
141.23
135.00
133.37
130.95
130.88
128.47
127.50
127.30
125.78
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.79
64.55
52.66
31.89
31.68
tBu
NH
O
OH
3z
13C NMR (151 MHz, CDCl3)
f1 (ppm)



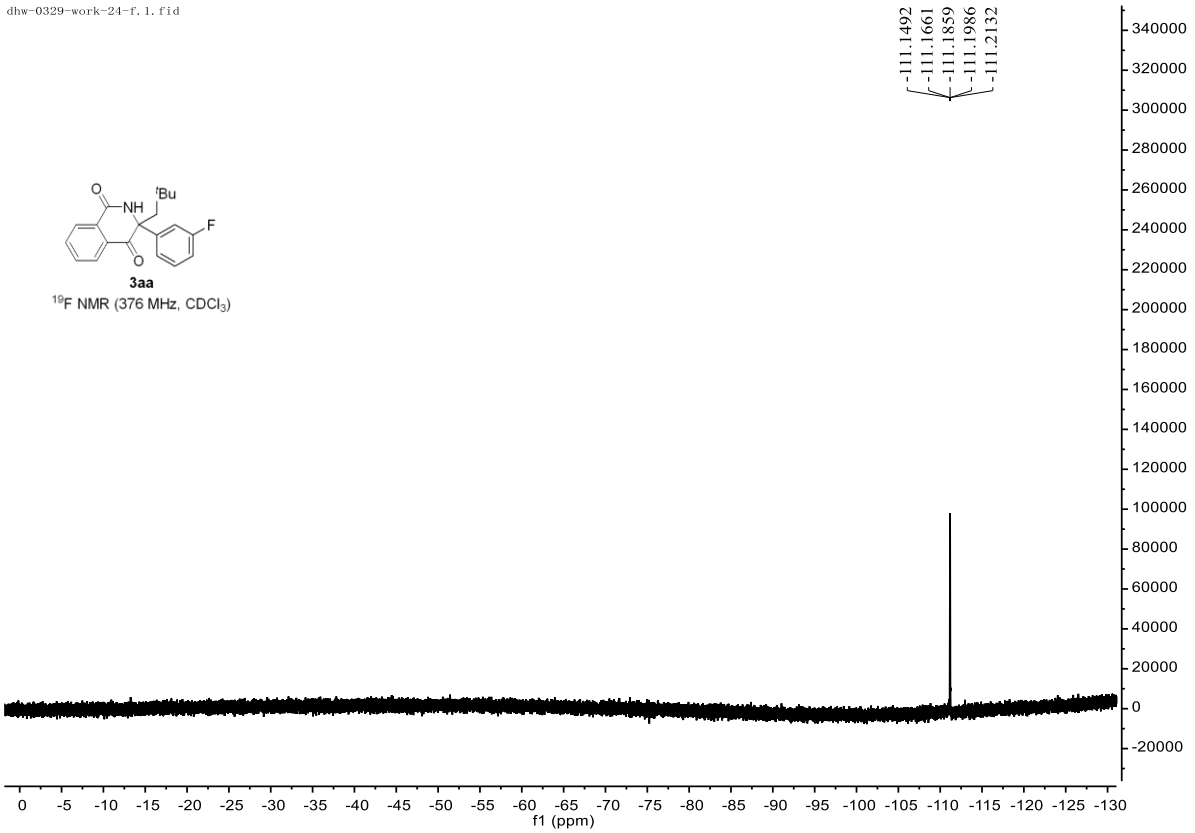
3aa

^{1}H NMR (400 MHz, $CDCl_3$)



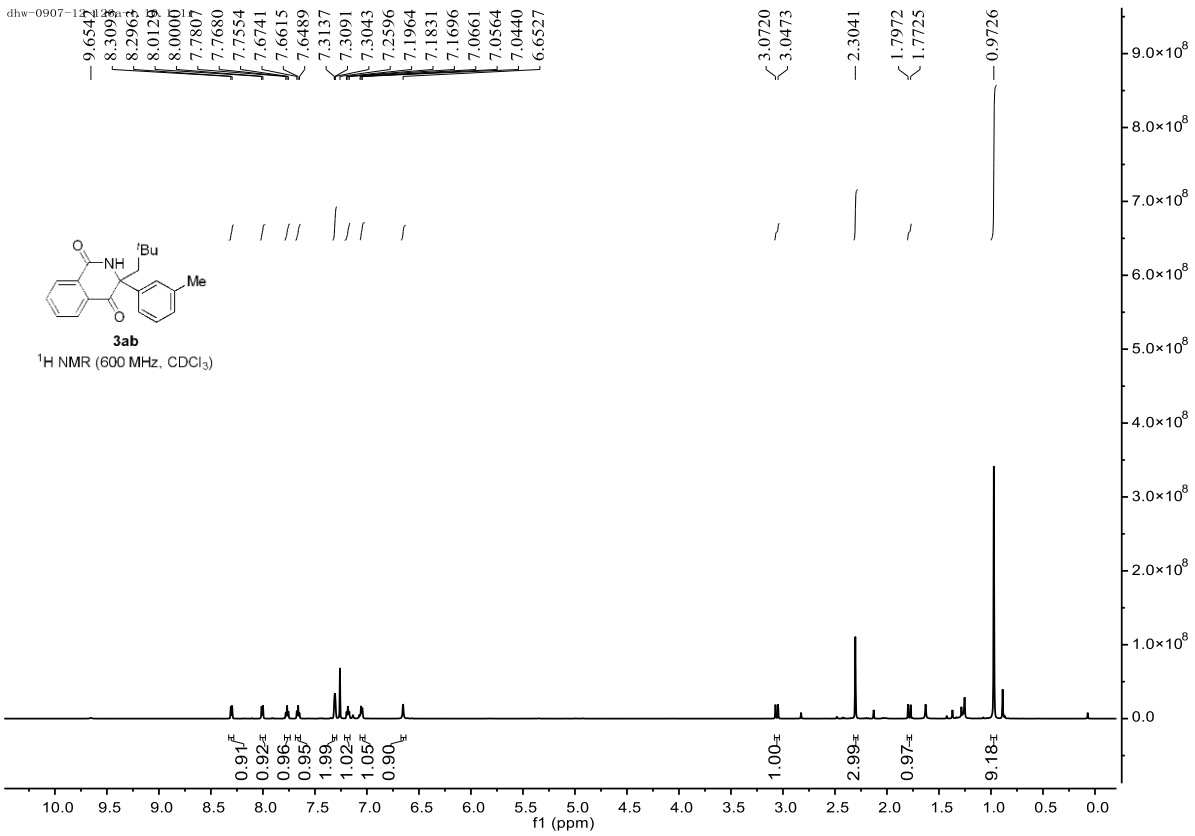
3aa

^{13}C NMR (151 MHz, $CDCl_3$)



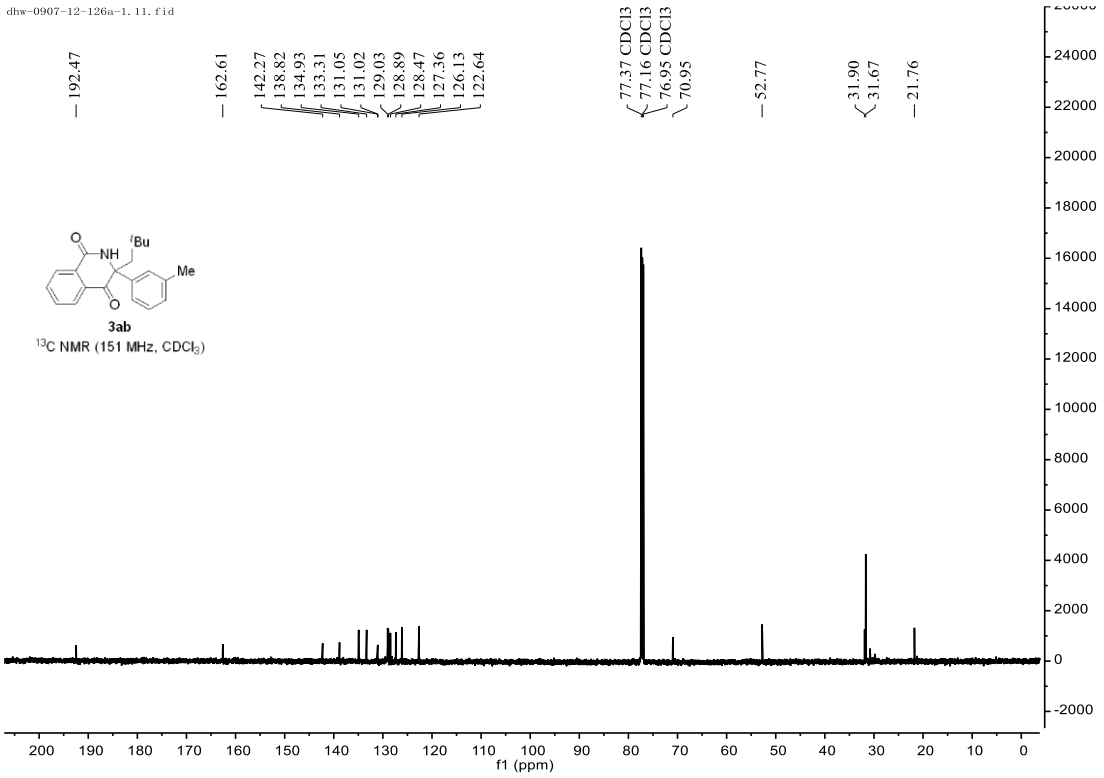
3aa

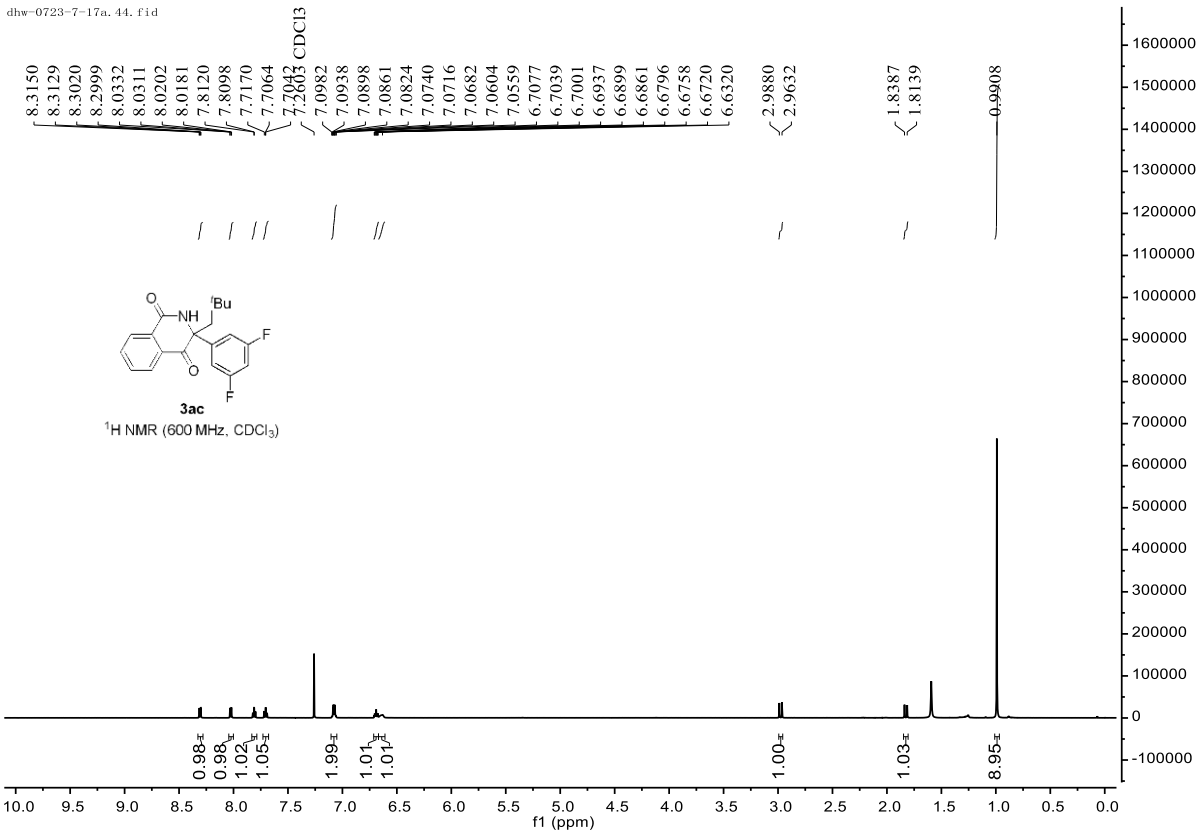
^{19}F NMR (376 MHz, $CDCl_3$)

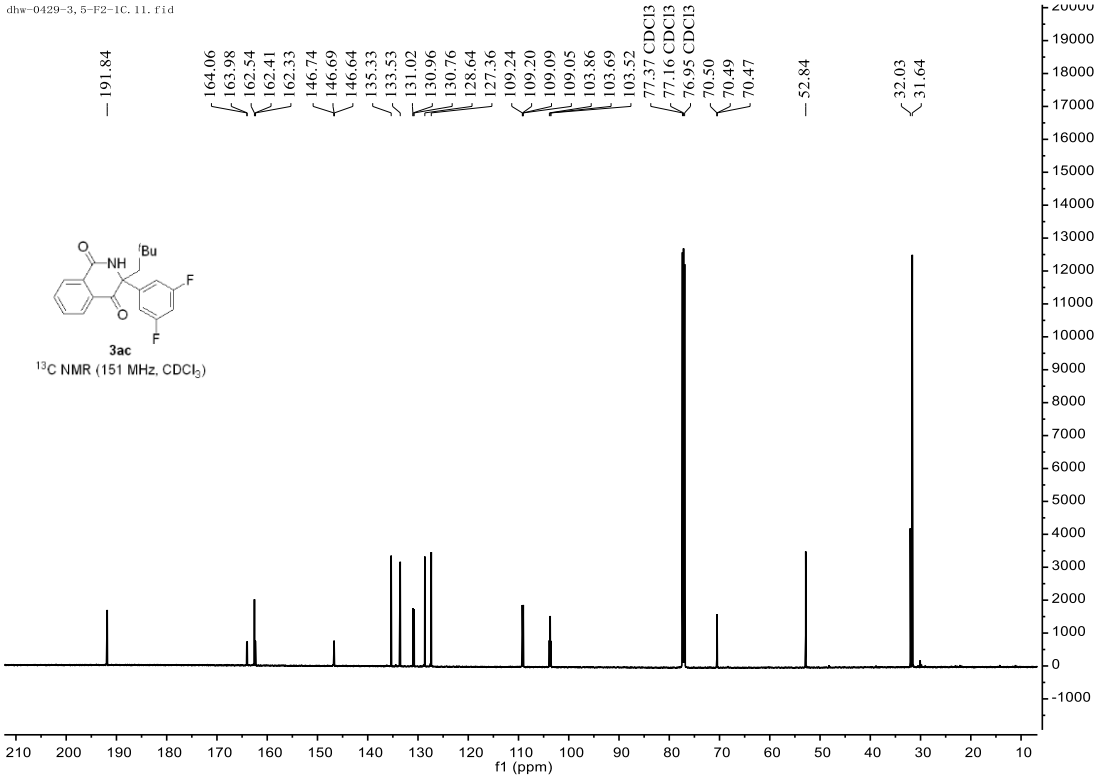


3ab

^{1}H NMR (600 MHz, $CDCl_3$)

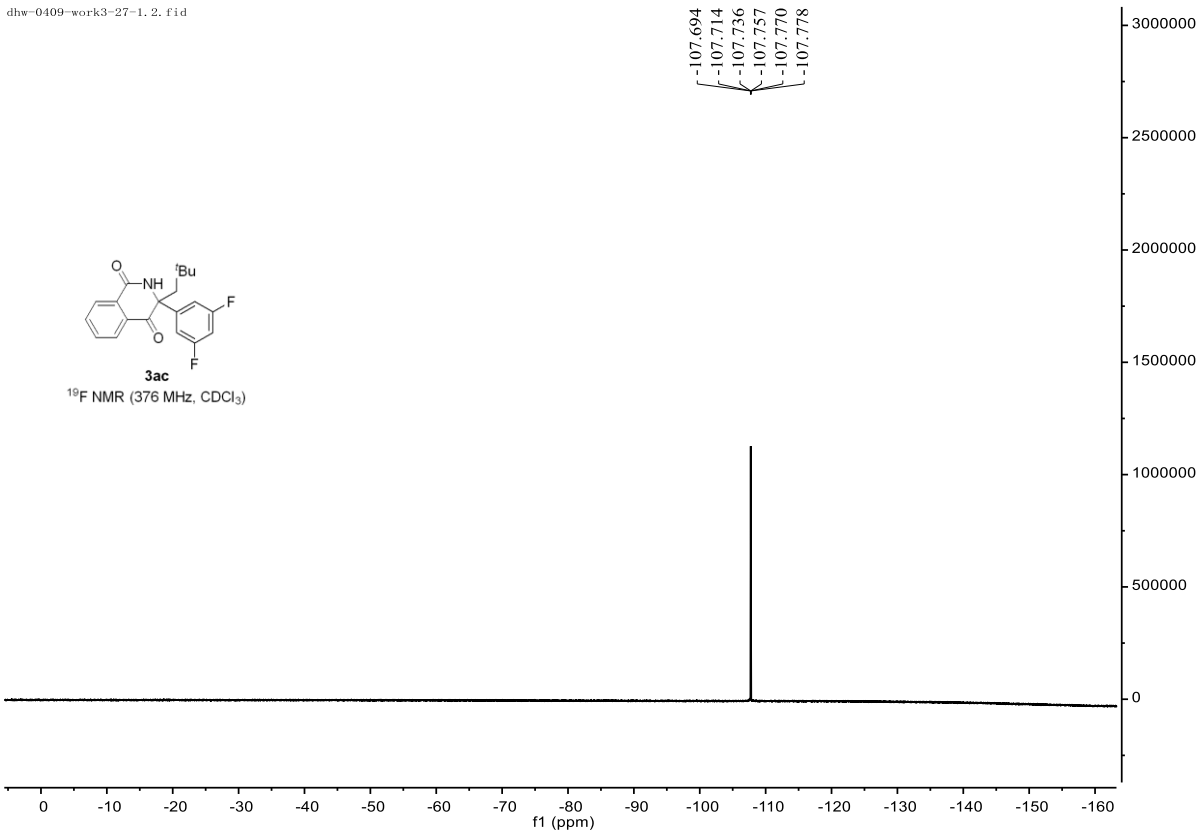

dhw-0907-12-126a-1.11.fid
192.47
162.61
142.27
138.82
134.93
133.31
131.05
131.02
129.03
128.89
128.47
127.36
126.13
122.64
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.95
52.77
31.90
31.67
21.76
tBu
NH
Me
3ab
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-0723-7-17a.44.fid
8.3150
8.3129
8.3020
8.2999
8.0332
8.0311
8.0202
8.0181
7.8120
7.8098
7.7170
7.7064
7.7042
7.2603 CDCl3
7.0982
7.0938
7.0898
7.0861
7.0824
7.0740
7.0716
7.0682
7.0604
7.0559
6.7077
6.7039
6.7001
6.6937
6.6899
6.6861
6.6796
6.6758
6.6720
6.6320
2.9880
2.9632
1.8387
1.8139
0.9908
tBu
NH
F
F
3ac
1H NMR (600 MHz, CDCl3)
0.98
0.98
1.02
1.05
1.99
1.01
1.01
1.00
1.03
8.95
f1 (ppm)



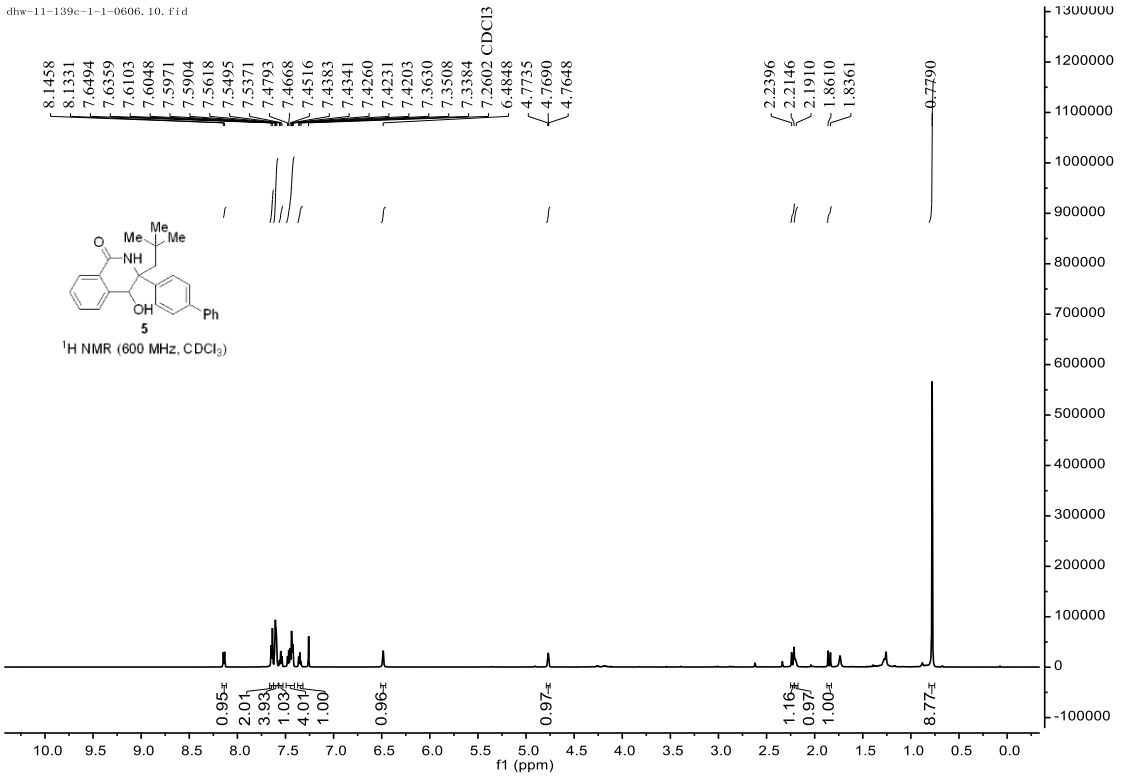
3ac

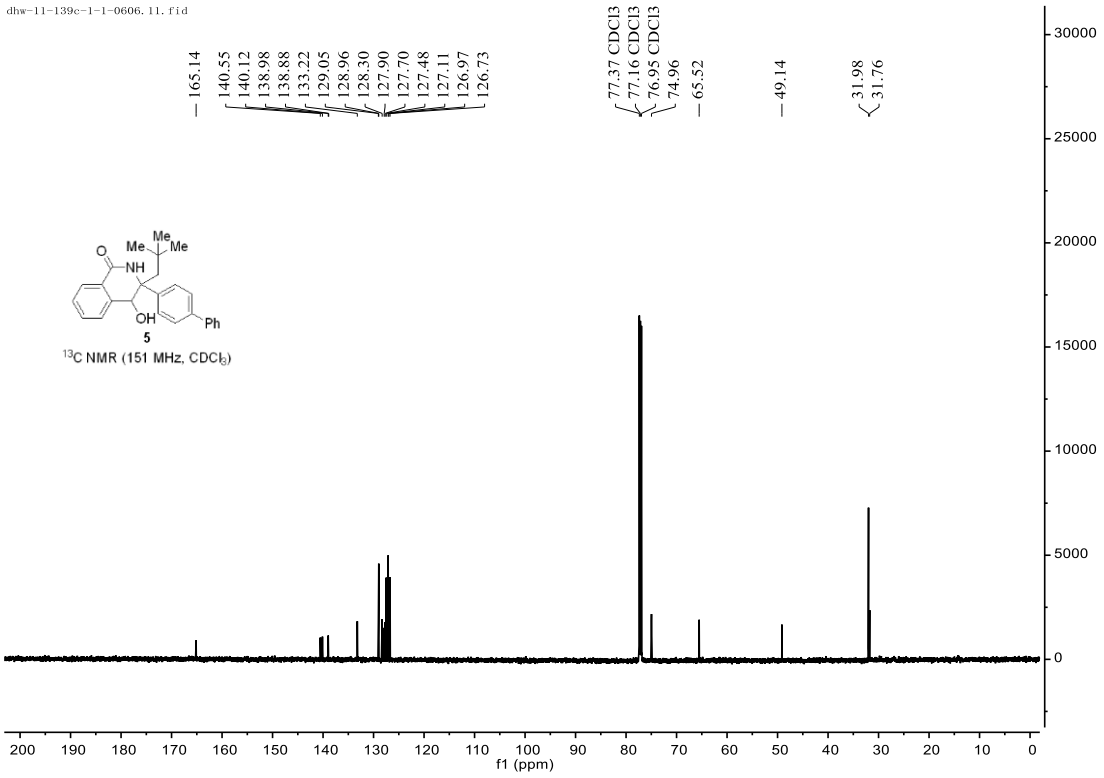
^{13}C NMR (151 MHz, $CDCl_3$)

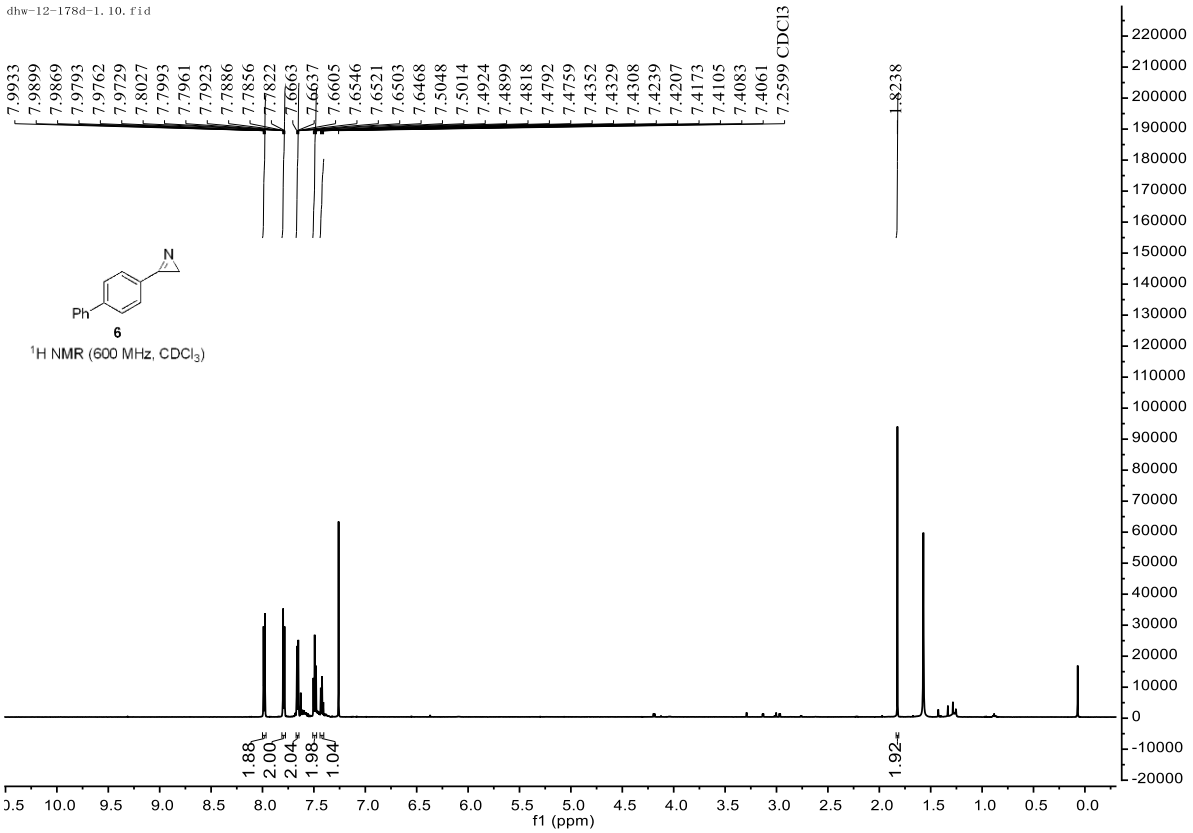


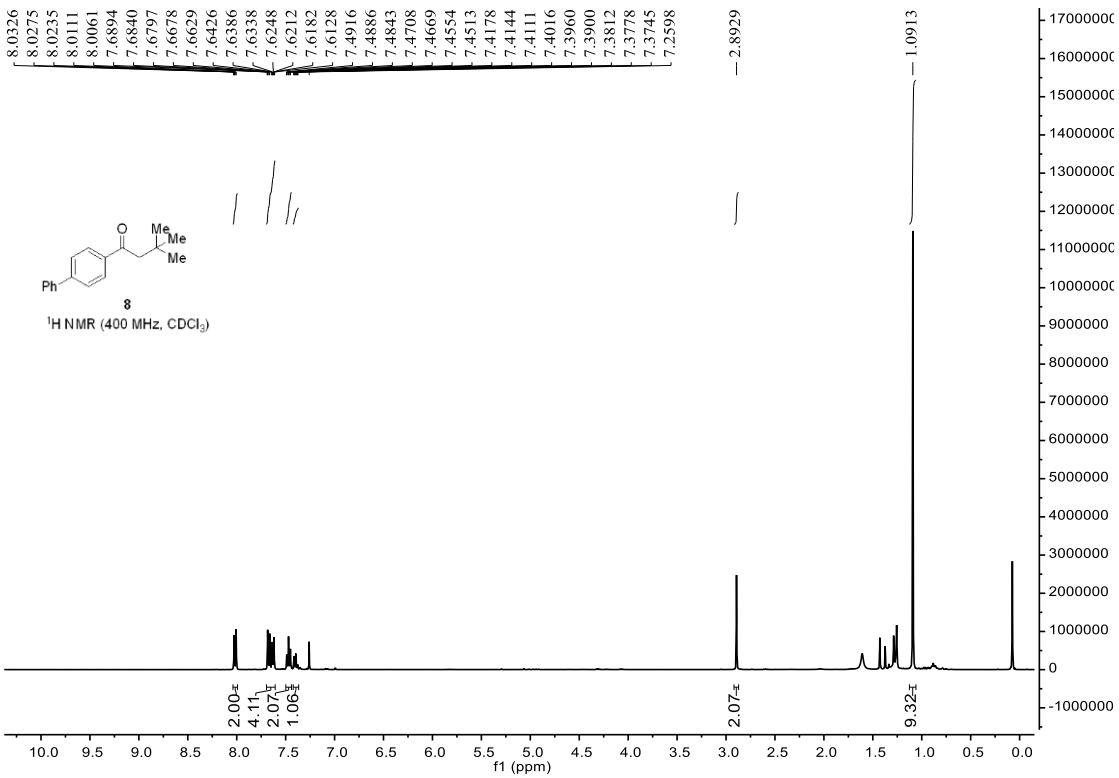
3ac

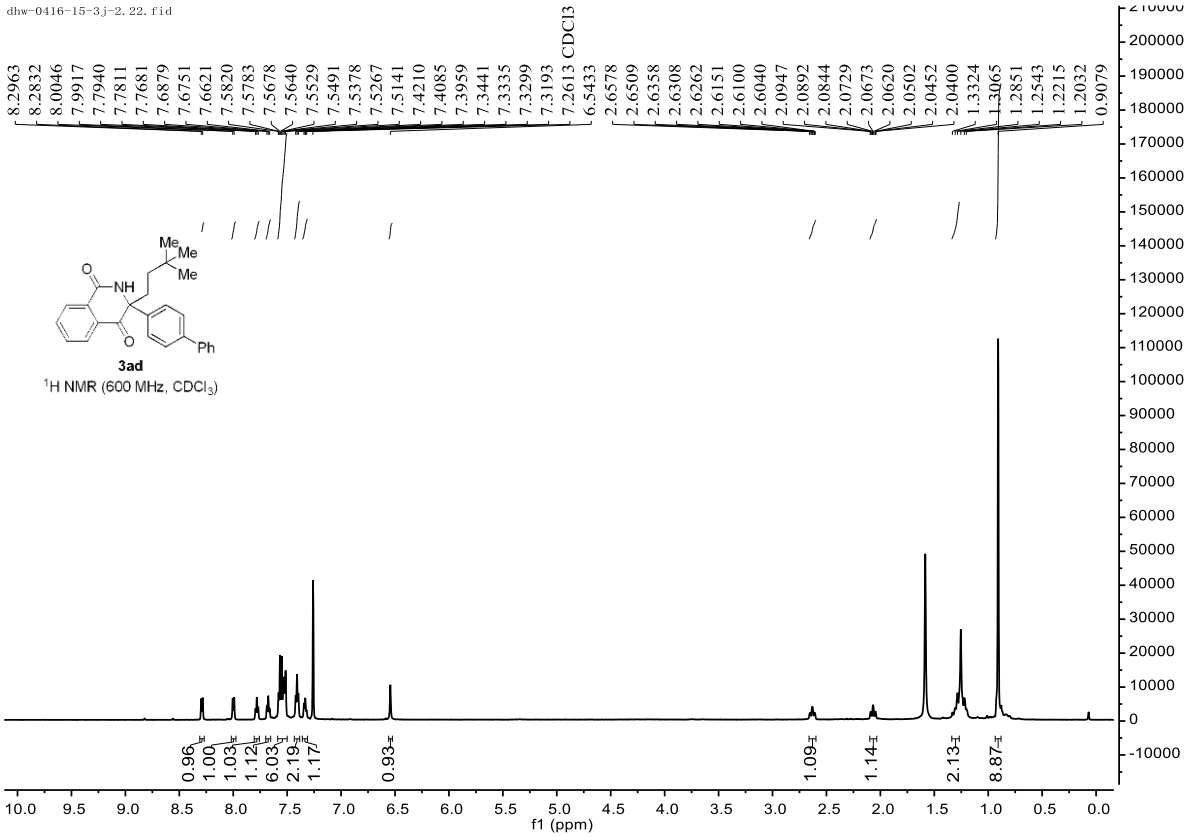
^{19}F NMR (376 MHz, $CDCl_3$)


dhw-11-139c-1-1-0606.10.fid
8.1458
8.1331
7.6494
7.6359
7.6103
7.6048
7.5971
7.5904
7.5618
7.5495
7.5371
7.4793
7.4668
7.4516
7.4383
7.4341
7.4260
7.4231
7.4203
7.3630
7.3508
7.3384
7.2602 CDCl3
6.4848
4.7735
4.7690
4.7648
2.2396
2.2146
2.1910
1.8610
1.8361
0.7790
Me
Me
Me
O
NH
OH
Ph
5
1H NMR (600 MHz, CDCl3)
0.95
2.01
3.93
1.03
4.01
1.00
0.96
0.97
1.16
0.97
1.00
8.77
f1 (ppm)


dhw-11-139c-1-1-0606.11.fid
165.14
140.55
140.12
138.98
138.88
133.22
129.05
128.96
128.30
127.90
127.70
127.48
127.11
126.97
126.73
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
74.96
65.52
49.14
31.98
31.76
Me
Me
Me
O
NH
OH
Ph
5
13C NMR (151 MHz, CDCl3)
f1 (ppm)


dhw-12-178d-1.10.fid
7.9933
7.9899
7.9869
7.9793
7.9762
7.9729
7.8027
7.7993
7.7961
7.7923
7.7886
7.7856
7.7822
7.7663
7.7637
7.6605
7.6546
7.6521
7.6503
7.6468
7.5048
7.5014
7.4924
7.4899
7.4818
7.4792
7.4759
7.4352
7.4329
7.4308
7.4239
7.4207
7.4173
7.4105
7.4083
7.4061
7.2599 CDCl3
1.8238
N
Ph
6
1H NMR (600 MHz, CDCl3)
1.88
2.00
2.04
1.98
1.04
1.92
f1 (ppm)


8.0326
8.0275
8.0235
8.0111
8.0061
7.6894
7.6840
7.6797
7.6678
7.6629
7.6426
7.6386
7.6338
7.6248
7.6212
7.6182
7.6128
7.4916
7.4886
7.4843
7.4708
7.4669
7.4554
7.4513
7.4178
7.4144
7.4111
7.4016
7.3960
7.3900
7.3812
7.3778
7.3745
7.2598
2.8929
1.0913
O
Me
Me
Me
Ph
8
1H NMR (400 MHz, CDCl3)
2.00
4.11
2.07
1.06
2.07
9.32
f1 (ppm)


dhw-0416-15-3j-2.22.fid
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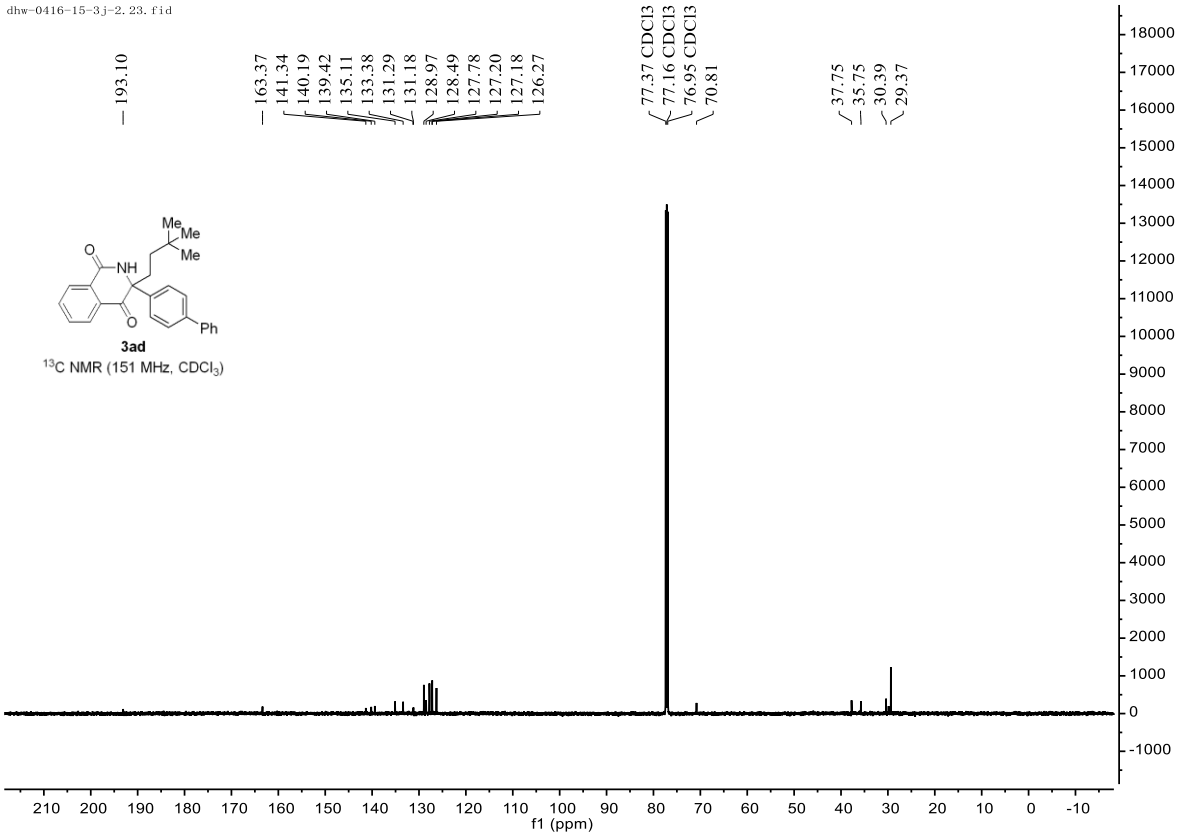
8.2832
8.0046
7.9917
7.7940
7.7811
7.7681
7.6879
7.6751
7.6621
7.5820
7.5783
7.5678
7.5640
7.5529
7.5491
7.5378
7.5267
7.5141
7.4210
7.4085
7.3959
7.3441
7.3335
7.3299
7.3193
7.2613 CDCl3
6.5433
2.6578
2.6509
2.6358
2.6308
2.6262
2.6151
2.6100
2.6040
2.0947
2.0892
2.0844
2.0729
2.0673
2.0620
2.0502
2.0452
2.0400
1.3324
1.3065
1.2851
1.2543
1.2215
1.2032
0.9079
Me
Me
Me
NH
O
O
Ph
3ad
1H NMR (600 MHz, CDCl3)
0.96
1.00
1.03
1.12
6.03
2.19
1.17
0.93
1.09
1.14
2.13
8.87
f1 (ppm)


dhw-0416-15-3j-2.23.fid
193.10
163.37
141.34
140.19
139.42
135.11
133.38
131.29
131.18
128.97
128.49
127.78
127.20
127.18
126.27
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
70.81
37.75
35.75
30.39
29.37
Me
Me
Me
NH
O
O
Ph
3ad
13C NMR (151 MHz, CDCl3)
f1 (ppm)