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Supporting information

for

Sodium nitrite-promoted aerobic oxidative coupling of aryl methyl ketones with ammonium under metal-free conditions: A facile access to polysubstitution imidazoles

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General Experimental Information

Reagents and solvents: Commercially available reagents were used without any further purification. All organic solvents were also of reagent grade quality without any further purification.

Chromatography: Flash column chromatography was performed using silicycle silica gel (200-300 mesh).

Analytical thin-layer chromatography (TLC) was performed on 0.2 mm coated silica gel plates (HSGF 254) and visualized using a UV lamp (254 nm or 365 nm).

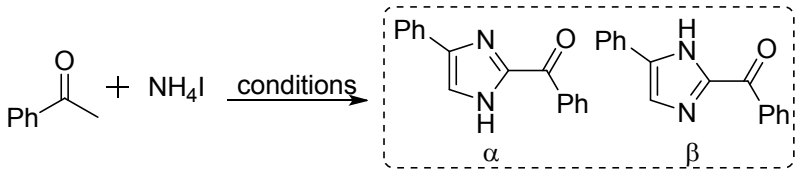
Nuclear Magnetic Resonance Spectroscopy:

^{1}H NMR was recorded on magnet system 400'54 ascend purchased from Bruker Biospin AG. ^{1}H NMR spectra chemical shifts (δ) are reported in parts per million (ppm) referenced to TMS (0 ppm).

^{13}C NMR spectra chemical shifts (δ) are reported in parts per million (ppm) were referenced to carbon resonances in the NMR solvent.

ESI-MS spectra were recorded on Agilent Q-TOF 6520.

Table S1. The Equiv. of $NaNO_2$ Screening[a]

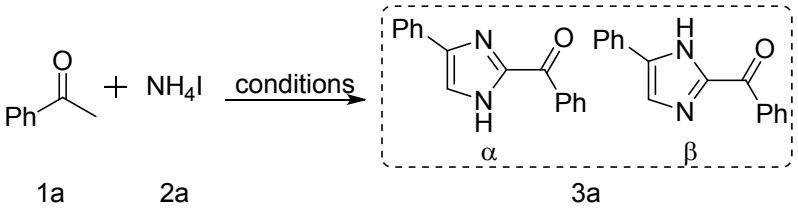


1a 2a 3a

Entry	Equiv. of $NaNO_2$ (mol %)	Yield[b] (%)
1	50	92
2	40	93
3	30	86
4	20	52
5	10	36
6[c]	100	9
7[c]	400	66

[a]Reaction conditions: 1a (1 mmol), 2a (2 mmol), $NaNO_2$, morpholine (0.2 mmol), AcOH (2 mL), 90℃, O_2 (O_2 balloon), 48h. [b]Isolated yield. [c]Under N_2.

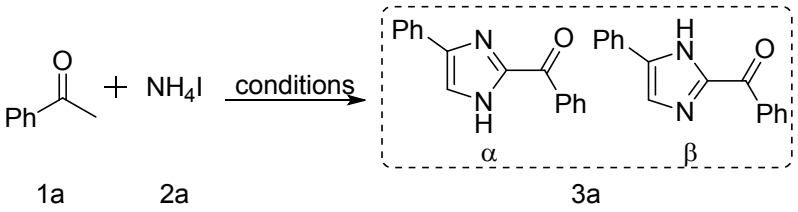
Table S2. The Temperature Screening[a]



Entry	T (℃)	Yield[b] (%)
1	90	93
2	80	94
3	70	85
4	60	63
5	50	42

[a]Reaction conditions: 1a (1 mmol), 2a (2 mmol), $NaNO_2$ (0.4 mmol), morpholine (0.2 mmol), AcOH (2 mL), T (℃), O_2 (O_2 balloon), 48h. [b]Isolated yield.

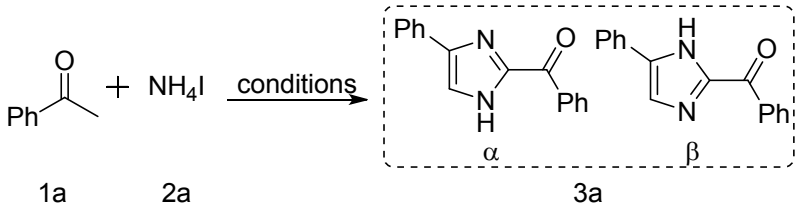
Table S3. The Equiv. of Morpholine Screening[a]



Entry	Equiv. of morpholine	Yield[b] (%)
1	0.2	94
2	0.15	93
3	0.1	84

[a]Reaction conditions: 1a (1 mmol), 2a (2 mmol), $NaNO_2$ (0.4 mmol), morpholine, AcOH (2 mL), T (80℃), O_2 (O_2 balloon), 48h. [b]Isolated yield.

Table S4. The Equiv. of Amine (2a) Screening[a]



Entry	Equiv. of **2a**	Yield[b] (%)
1	2	93
2	1.8	94
3	1.5	92
4	1.2	81

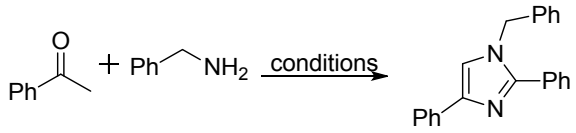
[a]Reaction conditions: 1a (1 mmol), 2a, $NaNO_2$ (0.4 mmol), morpholine (0.15 mmol), AcOH (2 mL), T (80℃), O_2 (O_2 balloon), 48h. [b]Isolated yield.

General Experimental Details for the Synthesis of (4 or 5)-Aryl-2-Benzoyl-Imidazole Analogues

The specific ketone (1 mmol, 1.0 eq), ammonium iodide (1.5 mmol, 1.5 eq) and morpholine (0.15 mmol, 0.15 eq) were dissolved in AcOH (2 mL), then sodium nitrite (0.4 mmol, 0.4 eq) was added to the reaction mixture and stirred for about 48h in a preheated oil batch at 80℃ under O_2 atmosphere (O_2 balloon). After the TLC revealed that the full conversion of corresponding ketone was completed, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated $Na_2S_2O_3$ solution (30 mL) and water (30 mL). The separated organic layer was dried over by anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexane/dichloromethane/ethyl acetate to afford the desired product.

Table S5. Screening of the Direct Oxidative Synthesis of

1,2,4-Trisubstituted Imidazoles from Aryl Methyl Ketones and Benzylamine[a]



1a 2b 4a

Entry	Solvent (v/v)	Additive1	Additive2	Yield[b] (%)
1[c]	AcOH	KI		NO
2	dioxane	KI		45
3	PhCl	KI		8
4	PhCN	KI		55
5	DMSO	KI		5
6	DMF	KI		trace
7	DMC	KI		3
8	AcOH	KI		NO
9	PhCN	KI	H_2SO_4	trace
10	PhCN	KI	TFA	23
11	PhCN	KI	AcOH	trace
12	PhCN	KI	$AlCl_3$	trace
13	PhCN	KI	$FeCl_3$	trace
14	PhCN	KI	Pyridine	52
15	PhCN	KI	TEA	61
16	PhCN	KI	CH_3COONa	19
17[d]	PhCN	KI	TEA	26
18	PhCN	TBAI	TEA	67
19	PhCN	I_2	TEA	trace
20[e]	PhCN	TBAI	TEA	69
21[f]	PhCN	TBAI	TEA	65
22[g]	PhCN	TBAI	TEA	59

[a]Reaction conditions: 1a (1 mmol), 2b (3 mmol), $NaNO_2$ (0.4 mmol), additive 1 (0.15 mmol), additive 2 (0.2 mmol), solvent (2 mL), T (80℃), O_2 (O_2 balloon), 48h. [b]Isolated yield.

[c]Reaction conditions: 1a (1 mmol), 2b (3 mmol), $NaNO_2$ (0.4 mmol), morpholine (0.15 mmol), KI (0.15 mmol), AcOH (2 mL), T (80℃), O_2 (O_2 balloon), 48h. [d]In the presence of morpholine (0.15 mmol). [e]0.5 mmol TEA was used. [f]1 mmol TEA was used. [g]1.5 mmol TEA was used.

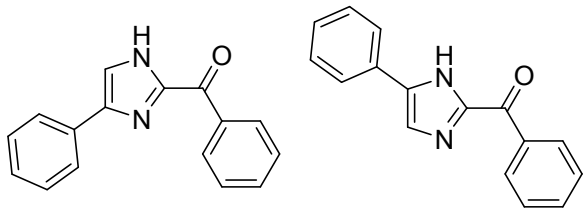
Initially, the reaction was carried out under the standard conditions of the coupling of 1a and 2a with the KI added. However, it was failed to give the desired product (entry 1). Then, the detailed screenings of solvents and additives were performed. Among various solvents examined, PhCN turned out to be the best choice (entries .2-8). In contrast to the coupling of 1a and 2a, AcOH was not effective (entry 8). The reaction was improved mostly with 61% isolated yield obtained when TEA was added (entry 15). Other additives 2 exhibited inferior reactivity (entries 9-14, 16). However, the addition of morpholine decreased the yield (entries 15, 17). Some other iodine salts were examined and TBAI was the most effective (entries 15, 18, 19). The loading amounts of TEA were screened (entries 18, 20-22). It was found that the reaction was not affect by the amounts of TEA remarkably. Based on the screening of the reaction parameters above, the optimal reaction conditions were obtained: 1a (1 mmol), 2b (3 mmol), $NaNO_2$ (0.4 mmol), TBAI (0.15 mmol), TEA (0.2 mmol) in PhCN (2 mL) at 80℃ under O_2 (O_2 balloon) for 48h.

General Experimental Details for the Synthesis of 1,2,4-

Trisubstituted Imidazoles

The specific ketone (1 mmol, 1.0 eq), benzylamine (3 mmol, 3 eq), TBAI (0.15 mmol, 0.15 eq), TEA (0.2 mmol, 0.2 eq) were dissolved in PhCN (2 mL), then sodium nitrite (0.4 mmol, 0.4 eq) was added to the reaction mixture and stirred for about 48h in a preheated oil batch at 80℃ under O_2 atmosphere (O_2 balloon). After the TLC revealed that the full conversion of corresponding ketone was completed, the reaction mixture was cooled and diluted with ethyl acetate (30 mL). The crude product was washed with saturated $Na_2S_2O_3$ solution (30 mL) and water (30 mL). The separated organic layer was dried over by anhydrous Na_2SO_4 and filtered. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel using hexane/ethyl acetate to afford the desired product.

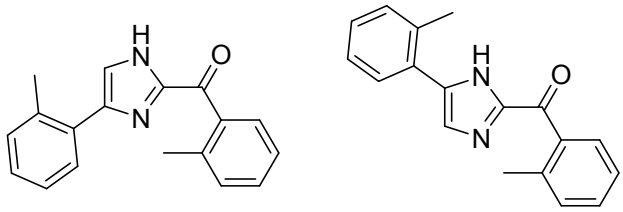
Analytical Data for Compounds 3 and 4



3a

Phenyl-(5-phenyl-1*H*-imidazol-2-yl)ketone (α) and Phenyl-(4-phenyl-1*H*-imidazol-2-yl)ketone (β)

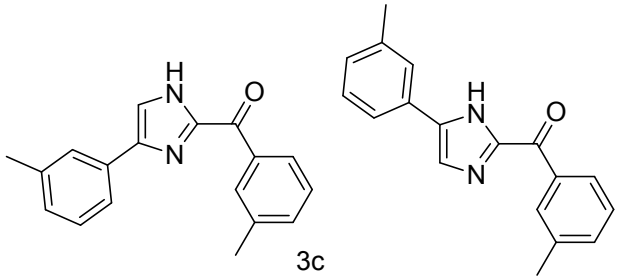
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 50:60:3); white solid (114 mg, 92% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.80 (s, 0.23H), 13.63 (s, 1H), 8.60-8.58 (m, 2H), 8.48–8.44 (m, 1H), 8.09 (d, *J* = 2.5 Hz, 1H), 7.99–7.92 (m, 2.23H), 7.79 (d, *J* = 1.6 Hz, 0.23H), 7.70 (qt, *J* = 9.1, 1.3 Hz, 1.23H), 7.64-7.55 (m, 2.46H), 7.50-7.40 (m, 2.46H), 7.37 (tt, *J* = 8.1, 2,4 Hz, 0.23H), 7.29 (tt, *J* = 8.0, 2.8 Hz, 1H); HRMS (TOF) m/z $[M + H]^+$ Calcd for $C_{16}H_{12}N_2O$ 249.1022 found 249.1020.



3b

2-Methylphenyl-[5-(2-methylphenyl)-1*H*-imidazol-2-yl]ketone (α) and 2-Methylphenyl-[4-(2-methylphenyl)-1*H*-imidazol-2-yl]ketone (β)

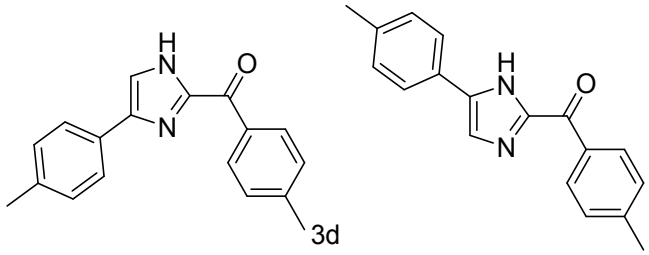
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 50:60:3); yellow solid (123 mg, 89% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.66 (s, 1H), 13.62 (s, 0.35H), 7.89 (dd, *J* = 3.6, 1.2 Hz, 1H), 7.79 (dd, *J* = 3.6, 1.2 Hz, 0.35H), 7.77 (d, *J* = 2.5 Hz, 1H), 7.67–7.63 (m, 1H), 7.56-7.53 (m, 0.35H), 7.49–7.43 (m, 1H), 7.38–7.28 (m, 4.05H), 7.26–7.16 (m, 3.05H), 2.45 (s, 3H), 2.41 (s, 1.05H), 2.40 (s, 3H), 2.38 (s, 1.05H); HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{18}H_{16}N_2O$ 277.1335 found 277.1341.



3c

3-Methylphenyl-[5-(3-methylphenyl)-1*H*-imidazol-2-yl]ketone (α) and 3-Methylphenyl-[4-(3-methylphenyl)-1*H*-imidazol-2-yl]ketone (β)

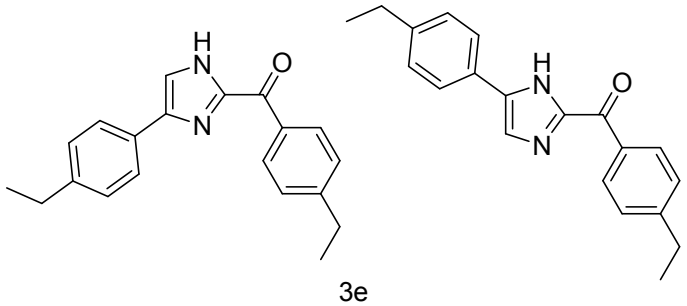
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 50:60:3); yellow solid (126 mg, 91% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.72 (s, 0.3H), 13.58 (s, 1H), 8.50-8.44 (m, 1H), 8.31-8.27 (m, 1.3H), 8.24 (s, 0.3H), 8.03 (d, *J* = 2.2 Hz, 1H), 7.82 (s, 0.3H), 7.78-7.70 (m, 2.6H), 7.52 – 7.41 (m, 2.6H), 7.36-7.26 (m, 1.3H), 7.16 (d, *J* = 7.5 Hz, 0.3H), 7.09 (d, *J* = 7.5 Hz, 1H), 2.43 (s, 3H), 2.41 (s, 0.9H), 2.35 (s, 3.9H); ^{13}C NMR (101 MHz, DMSO) δ 181.04, 180.90, 145.71, 144.66, 142.95, 138.10, 137.65, 137.53, 137.40, 136.21, 135.98, 135.73, 133.64, 133.58, 133.45, 130.72, 128.90, 128.78, 128.51, 128.17(d, *J* = 0.9 Hz), 128.04, 127.91, 127.78, 126.23, 125.40, 122.81, 122.07, 118.45, 21.11, 21.00, 20.96; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{18}H_{16}N_2O$ 277.1335 found 277.1340.



3d

4-Methylphenyl-[5-(4-methylphenyl)-1*H*-imidazol-2-yl]ketone (α) and 4-Methylphenyl-[4-(4-methylphenyl)-1*H*-imidazol-2-yl]ketone (β)

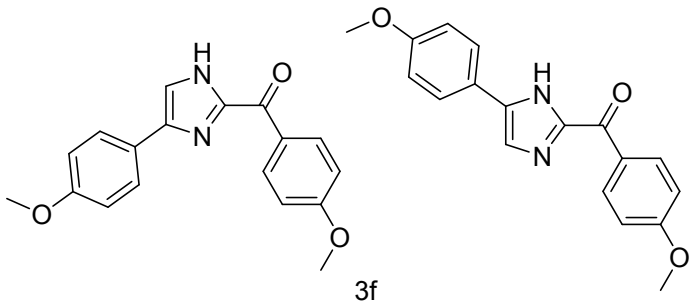
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 50:60:3); yellow solid (119 mg, 86% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.66 (s, 0.3H), 13.52 (s, 1H), 8.52 (d, *J* = 8.2 Hz, 2H), 8.41 (d, *J* = 8.2 Hz, 0.6H), 7.99 (d, *J* = 2.4 Hz, 1H), 7.85 (d, *J* = 8.2 Hz, 0.6H), 7.82 (d, *J* = 8.1 Hz, 2H), 7.72 (d, *J* = 1.5 Hz, 0.3H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.1 Hz, 0.6H), 7.27 (d, *J* = 8.0 Hz, 0.6H), 7.23 (d, *J* = 7.9 Hz, 2H), 2.43 (s, 3H), 2.41 (s, 0.9H), 2.34 (s, 0.9H), 2.33 (s, 3H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{18}H_{16}N_2O$ 277.1335 found 277.1334.



3e

4-Ethylphenyl-[5-(4-ethylphenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-Ethylphenyl-[4-(4-ethylphenyl)-1*H*-imidazol-2-yl]ketone (β)

Purified by column chromatography (dichloromethane/hexane/ethyl acetate 50:60:3); yellow solid (135 mg, 89% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.68 (s, 0.3H), 13.53 (s, 1H), 8.53 (d, *J* = 8.2 Hz, 2H), 8.41 (d, *J* = 8.2 Hz, 0.6H), 8.00 (s, 1H), 7.88 (d, *J* = 8.1 Hz, 0.6H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.73 (s, 0.3 H), 7.45 (d, *J* = 8.2 Hz, 2H), 7.41 (d, *J* = 8.2 Hz, 0.6H), 7.30 (d, *J* = 8.2 Hz, 0.6H), 7.26 (d, *J* = 8.0 Hz, 2H), 2.77-2.68 (m, 2.6H), 2.68-2.59 (m, 2.6H), 1.29-1.18 (m, 7.8H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{20}H_{20}N_2O$ 305.1648 found 305.1649.

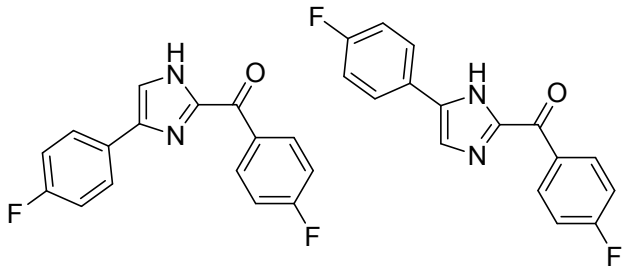


3f

4-Methoxyphenyl-[5-(4-methoxyphenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-Methoxyphenyl-[4-(4-methoxyphenyl)-1*H*-imidazol-2-yl]ketone (β)

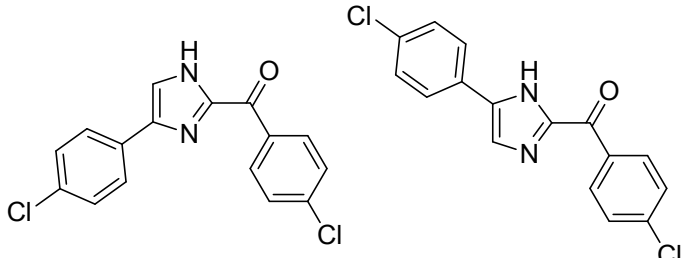
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 40:60:3); white

solid (101 mg, 66% yield); ^{1}H NMR (500 MHz, DMSO) δ 13.50 (s, 0.29H), 13.38 (s, 1H), 8.68 (d, *J* = 5.2 Hz, 2H), 8.57 (s, 0.58H), 7.92-7.82 (m, 3.87H), 7.13 (d, *J* = 7.6 Hz, 2.58H), 7.00 (d, *J* = 8.1 Hz, 2.58H), 3.88 (s, 3.87H), 3.79 (s, 3.87H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{18}H_{16}N_2O_3$ 309.1234 found 309.1269.



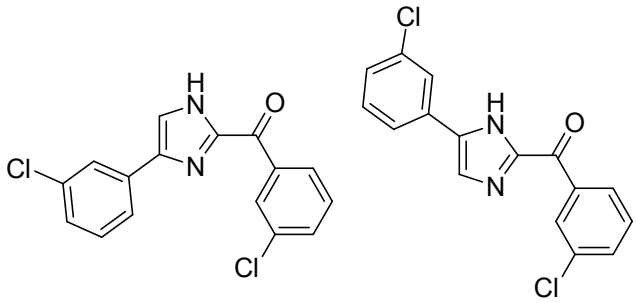
3g

4-Fluorophenyl-[5-(4-fluorophenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-Fluorophenyl-[4-(4-fluorophenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 20:20:1); yellow solid (112 mg, 79% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.82 (s, 0.19H), 13.65 (s, 1H), 8.72 (dd, *J* = 8.8, 5.8 Hz, 2H), 8.59 (t, *J* = 6.8 Hz 0.38H), 8.08 (s, 1H), 7.97 (dd, *J* = 8.6, 5.6 Hz, 2.38H), 7.78 (s, 0.19H), 7.44 (t, *J* = 8.7 Hz, 2.38H), 7.35–7.23 (m, 2.38H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{16}H_{10}N_2OF_2$ 285.0834 found 285.0832.



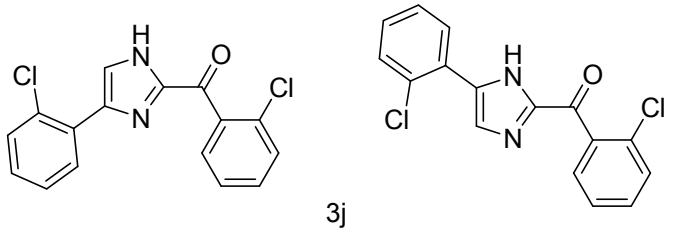
3h

4-Chlorophenyl-[5-(4-chlorophenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-Chlorophenyl-[4-(4-chlorophenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 20:20:1); yellow solid (138 mg, 87% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.91 (s, 0.15H), 13.73 (s, 1H), 8.61 (t, *J* = 1.6 Hz, 1H), 8.59 (t, *J* = 2.1 Hz, 1H), 8.51 (t, *J* = 2.8 Hz, 0.15H), 8.49 (t, *J* = 2.0 Hz, 0.15H), 8.16 (d, *J* = 2.4 Hz, 1H), 8.01 (s, 0.16H), 7.99 (s, 0.16H), 7.96 (t, *J* = 2.0 Hz, 1H), 7.94 (t, *J* = 1.6 Hz, 1H), 7.84 (s, 0.15H), 7.69 (s, 1H), 7.68-7.65 (m, 1.15 H), 7.64 (s, 0.15H), 7.54 (s, 0.15H), 7.52 (s, 0.15H), 7.50-7.48 (m, 1H), 7.48-7.46 (m, 1H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{16}H_{10}N_2OCl_2$ 317.0243 found 317.0279.



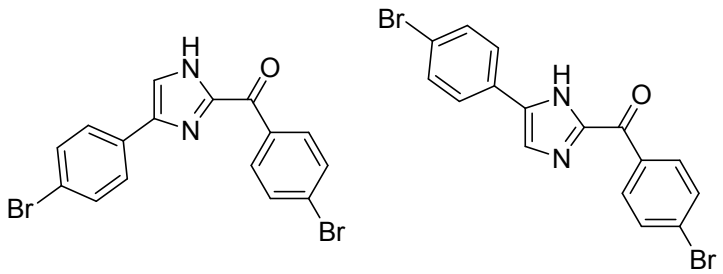
3i

3-Chlorophenyl-[5-(3-chlorophenyl)-1*H*-imidazol-2-yl]ketone (α) and
3-Chlorophenyl-[4-(3-chlorophenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 20:20:1); yellow solid (134 mg, 85% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.97 (s, 0.18H), 13.80 (s, 1H), 8.56 (t, *J* = 2.0 Hz, 1H), 8.53 (dt, *J* = 7.6, 1.2 Hz, 1.18H), 8.38 (d, *J* = 7.9 Hz, 0.18H), 8.25-8.20 (m, 1H), 8.16-8.12 (m, 0.18H), 7.98–7.87 (m, 2.36H), 7.80–7.73 (m, 1.18H), 7.69–7.60 (m, 1.18H), 7.51–7.39 (m, 1.36H), 7.37-7.31 (m, 1H); ^{13}C NMR (101 MHz, DMSO) δ 179.15, 145.67, 144.38, 141.50, 137.75, 137.54, 135.64, 134.50, 133.85, 133.58, 133.07, 132.94, 132.79, 132.63, 130.76, 130.60, 130.40, 130.21, 130.13, 129.99, 129.26, 129.00, 128.05, 126.94, 125.31, 124.38, 124.27, 123.35, 119.99; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{16}H_{10}N_2OCl_2$ 317.0243 found 317.0281.



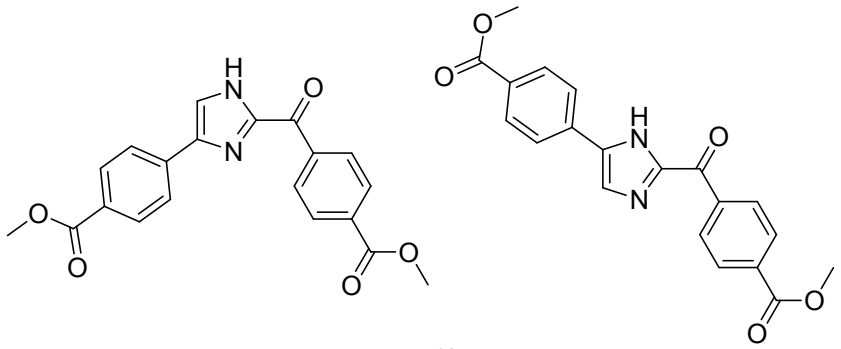
3j

2-Chlorophenyl-[5-(2-chlorophenyl)-1*H*-imidazol-2-yl]ketone (α) and
2-Chlorophenyl-[4-(2-chlorophenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 20:20:1); white solid (141 mg, 89% yield); ^{1}H NMR (400 MHz, DMSO) δ 14.00 (s, 1.14H), 8.09 (s, 1H), 7.87 (d, *J* = 7.4 Hz, 1H), 7.78 (d, *J* = 7.4 Hz, 1.28H), 7.59 (q, *J* = 7.9 Hz, 2.70H), 7.50 (t, *J* = 7.2 Hz, 2.28H), 7.37 (t, *J* = 7.3 Hz, 1H), 7.31 (t, *J* = 7.3 Hz, 1H); ^{13}C NMR (101 MHz, DMSO) δ 183.59, 144.33, 140.23, 137.50, 135.86, 132.40, 132.09, 131.11, 131.07, 130.85, 130.59, 130.43, 130.32, 129.22, 127.77, 127.23, 122.98; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{16}H_{10}N_2OCl_2$ 317.0243 found 317.0255.



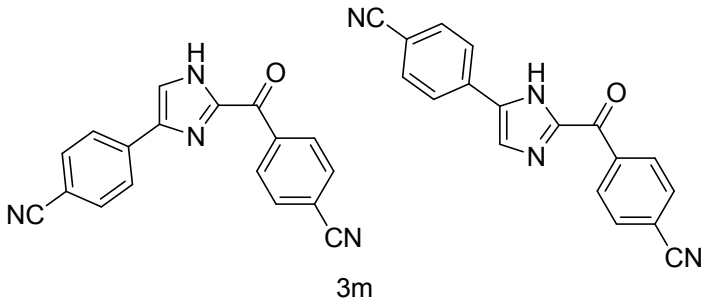
3k

4-Bromophenyl-[5-(4-bromophenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-Bromophenyl-[4-(4-bromophenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 20:20:1); brown solid (163 mg, 81% yield); ^{1}H NMR (400 MHz, DMSO) δ 13.92 (s, 0.15H), 13.75 (s, 1H), 8.52 (d, *J* = 8.6 Hz, 2H), 8.42 (d, *J* = 8.5 Hz, 0.3H), 8.17 (s, 1H), 7.93 (d, *J* = 8.3 Hz, 0.3H), 7.89 (d, *J* = 8.4 Hz, 2H), 7.85-7.77 (m, 2.45H), 7.67 (d, *J* = 7.6 Hz, 0.3H), 7.62 (d, *J* = 8.1 Hz, 2H); HRMS (TOF) m/z [M - H]$^-$ Calcd for $C_{16}H_{10}N_2OBr_2$ 402.9087 found 402.9078.



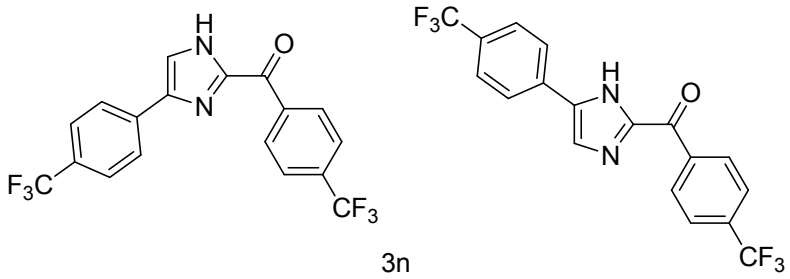
3l

4-(Methoxycarbonyl)phenyl-[5-(4-(methoxycarbonyl)phenyl)-1*H*-imidazol-2-yl]ketone (α)
4-(Methoxycarbonyl)phenyl-[4-(4-(methoxycarbonyl)phenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 8:5:1); yellow solid (142 mg, 78% yield); ^{1}H NMR (400 MHz, DMSO) δ 14.10 (s, 0.13H), 13.89 (s, 1H), 8.62 (d, *J* = 8.1 Hz, 2H), 8.54 (d, *J* = 8.2 Hz, 0.26H), 8.30 (s, 1H), 8.19–7.98 (m, 6.91H), 3.89 (d, *J* = 22.4 Hz, 6.78H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{20}H_{16}N_2O_5$ 365.1132 found 365.1126.



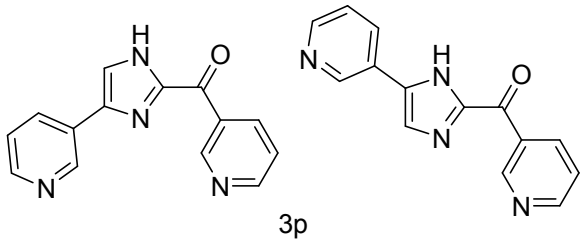
3m

4-Cyanophenyl-[5-(4-cyanophenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-Cyanophenyl-[4-(4-cyanophenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 15:20:1); yellow solid (118 mg, 79% yield); ^{1}H NMR (400 MHz, DMSO) δ 14.17 (s, 0.1H), 13.97 (s, 1H), 8.66 (d, *J* = 8.3 Hz, 2H), 8.55 (d, *J* = 8.2 Hz, 0.2H), 8.48 (d, *J* = 8.2 Hz, 0.1H), 8.37 (s, 1H), 8.19 (d, *J* = 8.5 Hz, 0.2H), 8.14–8.01 (m, 4.2H), 7.94 (d, *J* = 8.2 Hz, 0.2H), 7.90 (d, *J* = 8.3 Hz, 2H); HRMS (TOF) m/z [M - H]$^-$ Calcd for $C_{18}H_{10}N_4O$ 297.0782 found 297.0751.



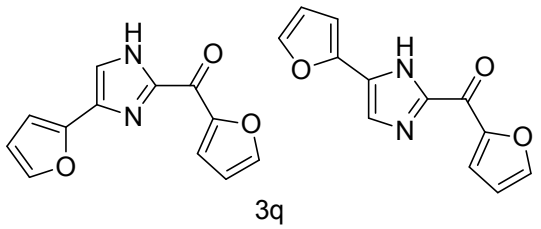
3n

4-(Trifluoromethyl)phenyl-[5-(4-(trifluoromethyl)phenyl)-1*H*-imidazol-2-yl]ketone (α) and
4-(Trifluoromethyl)phenyl-[4-(4-(trifluoromethyl)phenyl)-1*H*-imidazol-2-yl]ketone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 10:12:1); yellow solid (159 mg, 83% yield); ^{1}H NMR (400 MHz, DMSO) δ 14.16 (s, 0.11H), 13.93 (s, 1H), 8.70 (d, *J* = 8.1 Hz, 2H), 8.62 (d, *J* = 6.0 Hz, 0.22H), 8.33 (s, 1H), 8.25-8.10 m, 2.22H), 7.99 (d, *J* = 8.3 Hz, 2.33H), 7.86-7.75 (m, 2.22 H); HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{18}H_{10}N_2OF_6$ 385.0770 found 385.0767.



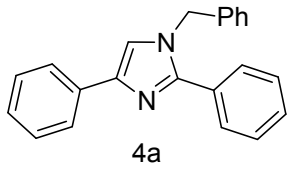
3p

3-Pyridinyl-[5-(3-pyridinyl)-1*H*-imidazol-2-yl]-methanone (α) and
3-Pyridinyl-[4-(3-pyridinyl)-1*H*-imidazol-2-yl]-methanone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 9:12:1); yellow solid (103 mg, 82% yield); ^{1}H NMR (400 MHz, DMSO) δ 14.14 (s, 0.15H), 13.91 (s, 1H), 9.70-9.550 (m, 1H), 9.47–9.24 (m, 0.15H), 9.16 (s, 1H), 9.11-9.01 (m, 0.15H), 8.95–8.68 (m, 2.30H), 8.62-8.45 (m, 1.15H), 8.29 (s, 2H), 8.10-7.90 (m, 0.3H), 7.70-7.61 (m, 1H), 7.59-7.57 (m, 0.3H), 7.56-7.43 (m, 1H); HRMS (TOF) m/z [M + Na]$^+$ Calcd for $C_{14}H_{10}N_4O$ 273.0747 found 273.0748.



3q

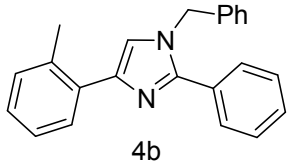
2-Furanyl-[5-(2-furanyl)-1*H*-imidazol-2-yl]methanone (α) and
2-Furanyl-[4-(2-furanyl)-1*H*-imidazol-2-yl]methanone (β)
Purified by column chromatography (dichloromethane/hexane/ethyl acetate 50:60:3); yellow solid (96 mg, 84% yield); ^{1}H NMR (400 MHz, DMSO) δ 8.23 (dd, *J* = 1.6, 0.6 Hz, 1H), 8.09 (dd, *J* = 3.6, 0.6 Hz, 1H), 7.98 (dd, *J* = 2.0, 0.8 Hz, 1H), 7.86 (s, 1H), 7.15 (d, *J* = 3.5 Hz, 1H), 6.87 (dd, *J* = 3.6, 1.7 Hz, 1H), 6.76 (dd, *J* = 3.5, 1.8 Hz, 1H); HRMS (TOF) m/z [M + Na]$^+$ Calcd for $C_{12}H_8N_2O_3$ 251.0427 found 251.0440. It is noteworthy that active hydrogen disappeared.



4a

Benzyl-2,4-diphenyl-1*H*-imidazole (4a)

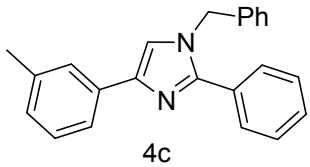
Purified by column chromatography (hexane/ethyl acetate 30:1); yellow solid (214 mg, 69% yield); ^{1}H NMR (400 MHz, DMSO) δ 7.86-7.81 (m, 3H), 7.65–7.60 (m, 2H), 7.49–7.42 (m, 3H), 7.40–7.30 (m, 4H), 7.29–7.19 (m, 2H), 7.10 (d, *J* = 7.2 Hz, 2H), 5.35 (s, 2H); ^{13}C NMR (101 MHz, DMSO) δ 147.71, 140.43, 137.97, 134.70, 130.97, 129.26, 129.21, 129.06, 129.00, 128.87, 128.05, 126.97, 126.92, 124.79, 119.25, 50.37; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{22}H_{18}N_2$ 311.1543 found 311.1545.



4b

1-Benzyl-4-(2-methylphenyl)-2-phenyl-1*H*-imidazole (4b)

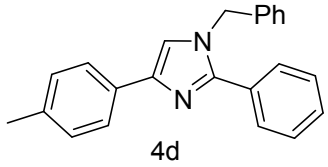
Purified by column chromatography (hexane/ethyl acetate 30:1); yellow solid (237 mg, 73% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.93 (d, *J* = 7.8 Hz, 1H), 7.67–7.64 (m, 3H), 7.50–7.44 (m, 3H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.32-7.24 (m, 3H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.12 (d, *J* = 7.4 Hz, 2H), 5.43 (s, 2H), 2.52 (s, 3H); ^{13}C NMR (126 MHz, DMSO) δ 146.26, 139.24, 137.57, 134.07, 133.30, 130.65, 130.49, 128.66, 128.56, 128.46, 128.27, 127.64, 127.43, 126.35, 126.26, 125.63, 121.16, 49.76, 21.66; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{23}H_{20}N_2$ 325.1699 found 325.1705.



4c

1-Benzyl-4-(3-methylphenyl)-2-phenyl-1*H*-imidazole (4c)

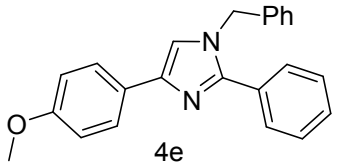
Purified by column chromatography (hexane/ethyl acetate 30:1); yellow solid (230 mg, 71% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.78 (s, 1H), 7.67 (s, 1H), 7.64–7.60 (m, 3H), 7.48-7.42 (m, 3H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.26 (dd, *J* = 16.6, 7.6 Hz, 2H), 7.10 (d, *J* = 7.3 Hz, 2H), 7.03 (d, *J* = 7.5 Hz, 1H), 5.34 (s, 2H), 2.33 (s, 3H); ^{13}C NMR (126 MHz, DMSO) δ 147.09, 139.99, 137.41, 134.08, 130.47, 128.68, 128.60, 128.47, 128.32, 127.49, 127.03, 126.47, 124.86, 121.45, 118.57, 49.84, 21.03; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{23}H_{20}N_2$ 325.1699 found 325.1712.



4d

1-Benzyl-4-(4-methylphenyl)-2-phenyl-1*H*-imidazole (4d)

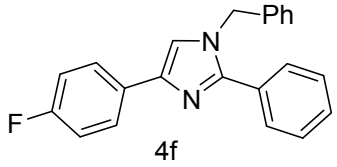
Purified by column chromatography (hexane/ethyl acetate 30:1); yellow solid (223 mg, 69% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.75–7.70 (m, 3H), 7.62 (d, *J* = 7.5 Hz, 2H), 7.49-7.40 (m, 3H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 7.5 Hz, 2H), 5.33 (s, 2H), 2.30 (s, 3H); ^{13}C NMR (126 MHz, DMSO) δ 146.99, 140.01, 137.43, 135.40, 131.44, 130.51, 128.99, 128.68, 128.57, 128.46, 128.30, 127.48, 126.46, 124.22, 118.09, 49.81, 20.70; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{23}H_{20}N_2$ 325.1699 found 325.1704.



4e

1-Benzyl-4-(4-methoxyphenyl)-2-phenyl-1*H*-imidazole (4e)

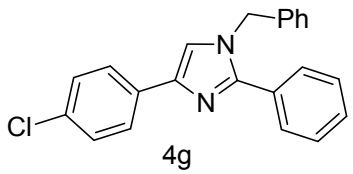
Purified by column chromatography (hexane/ethyl acetate 30:1); yellow solid (221 mg, 65% yield); ^{1}H NMR (400 MHz, DMSO) δ 7.75 (dt, *J* = 8.8, 2.0 Hz, 2H), 7.70 (s, 1H), 7.61 (dd, *J* = 7.8, 1.7 Hz, 2H), 7.48–7.39 (m, 3H), 7.33 (t, *J* = 7.3 Hz, 2H), 7.26 (tt, *J* = 7.2, 2.8 Hz, 1H), 7.09 (d, *J* = 7.1 Hz, 2H), 6.95 (dt, *J* = 8.8, 2.4 Hz, 2H), 5.33 (s, 2H), 3.76 (s, 3H); ^{13}C NMR (101 MHz, DMSO) δ 158.05, 146.92, 139.93, 137.55, 130.55, 128.74, 128.60, 128.53, 128.33, 127.52, 126.96, 126.46, 125.56, 117.50, 113.91, 55.01, 49.80; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{23}H_{20}N_2O$ 341.1648 found 341.1661.



4f

1-Benzyl-4-(4-fluorophenyl)-2-phenyl-1*H*-imidazole (4f)

Purified by column chromatography (hexane/ethyl acetate 25:1); yellow solid (207 mg, 63% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.85 (dd, *J* = 8.6, 5.7 Hz, 2H), 7.80 (s, 1H), 7.62 (dd, *J* = 7.7, 1.4 Hz, 2H), 7.48–7.41 (m, 3H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.19 (t, *J* = 8.9 Hz, 2H), 7.10 (d, *J* = 7.3 Hz, 2H), 5.34 (s, 2H); ^{13}C NMR (126 MHz, DMSO) δ 161.95, 160.02, 147.22, 139.05 , 137.34, 130.76, 130.73, 130.37, 128.69, 128.49, 128.33, 127.50, 126.45, 126.11, 126.04, 118.43, 115.31, 115.14, 49.85; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{22}H_{17}N_2F$ 329.1449 found 329.1460.

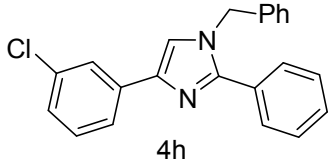


4g

1-Benzyl-4-(4-chlorophenyl)-2-phenyl-1*H*-imidazole (4g)

Purified by column chromatography (hexane/ethyl acetate 25:1); yellow solid (234 mg, 68% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.87 (s, 1H), 7.84 (d, *J* = 8.5 Hz, 2H), 7.61 (dd, *J* = 7.5, 1.6

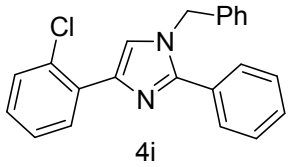
Hz, 2H), 7.48-7.40 (m, 5H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.10 (d, *J* = 7.4 Hz, 2H), 5.34 (s, 2H); ^{13}C NMR (126 MHz, DMSO) δ 147.37, 138.74, 137.26, 133.10, 130.64, 130.28, 128.74, 128.71, 128.50, 128.44, 128.35, 127.53, 126.49, 125.91, 119.14, 49.90; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{22}H_{17}N_2Cl$ 345.1153 found 345.1153.



4h

1-Benzyl-4-(3-chlorophenyl)-2-phenyl-1*H*-imidazole (4h)

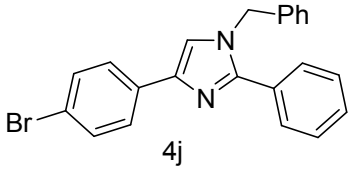
Purified by column chromatography (hexane/ethyl acetate 25:1); yellow solid (244 mg, 71% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.94 (s, 1H), 7.89 (t, *J* = 1.6 Hz, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.62 (dd, *J* = 7.6, 1.8 Hz, 2H), 7.48-7.43 (m, 3H), 7.39 (t, *J* = 7.9 Hz, 1H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.29–7.24 (m, 2H), 7.10 (d, *J* = 7.3 Hz, 2H), 5.34 (s, 2H); ^{13}C NMR (126 MHz, DMSO) δ 147.64, 138.61, 137.36, 136.54, 133.59, 130.51, 130.39, 128.96, 128.88, 128.68, 128.56, 127.73, 126.69, 126.19, 123.98, 122.90, 119.86, 50.13; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{22}H_{17}N_2Cl$ 345.1153 found 345.1154.



4i

1-Benzyl-4-(2-chlorophenyl)-2-phenyl-1*H*-imidazole (4i)

Purified by column chromatography (hexane/ethyl acetate 25:1); white solid (230 mg, 67% yield); ^{1}H NMR (400 MHz, DMSO) δ 8.24 (dd, *J* = 7.9, 1.7 Hz, 1H), 8.04 (s, 1H), 7.66–7.62 (m, 2H), 7.50–7.43 (m, 4H), 7.40 (td, *J* = 7.8, 1.2 Hz, 1H), 7.35–7.30 (m, 2H), 7.28–7.23 (m, 2H), 7.07 (d, *J* = 7.1 Hz, 2H), 5.43 (s, 2H); ^{13}C NMR (101 MHz, DMSO) δ 146.57, 137.43, 135.90, 132.19, 130.20, 130.14, 129.60, 129.16, 128.87, 128.76, 128.58, 128.43, 127.72, 127.56, 127.14, 126.47, 122.64, 49.88; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{22}H_{17}N_2Cl$ 345.1153 found 345.1155.

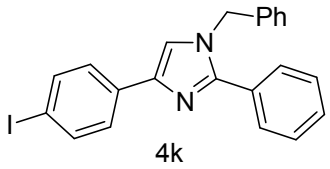


4j

1-Benzyl-4-(4-bromophenyl)-2-phenyl-1*H*-imidazole (4j)

Purified by column chromatography (hexane/ethyl acetate 25:1); yellow solid (275 mg, 71% yield); ^{1}H NMR (400 MHz, DMSO) δ 7.90 (s, 1H), 7.78 (dt, J=8.4, 2.0 Hz, 2H), 7.65–7.58 (m, 2H), 7.56 (m, dt, J=8.4, 2.0 Hz, 2H), 7.49–7.40 (m, 3H), 7.34 (tt, J=7.6, 1.2 Hz, 2H), 7.27 (tt, J=7.2, 2.8 Hz,1H), 7.12–7.06 (m, 2H), 5.35 (s, 2H); ^{13}C NMR (101 MHz, DMSO) δ 147.42, 138.79, 137.31, 133.47, 131.40, 130.28, 128.81, 128.77, 128.57, 128.39, 127.59, 126.52, 126.28, 119.28, 119.15, 49.93; HRMS (TOF) m/z [M + H]$^+$ Calcd for $C_{22}H_{17}N_2Br$ 389.0648 found

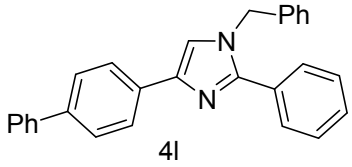
389.0682.



4k

1-Benzyl-4-(4-iodophenyl)-2-phenyl-1H-imidazole (4k)

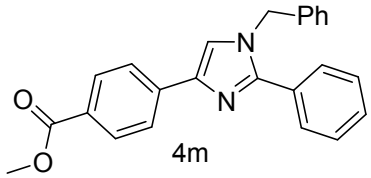
Purified by column chromatography (hexane/ethyl acetate 25:1); yellow solid (270 mg, 62% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.87 (s, 1H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.66–7.60 (m, 4H), 7.48–7.42 (m, 3H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.09 (d, *J* = 7.4 Hz, 2H), 5.34 (s, 2H); ^{13}C NMR (126 MHz, DMSO) δ 147.36, 138.87, 137.24, 137.17, 133.78, 130.26, 128.74, 128.70, 128.49, 128.35, 127.53, 126.50, 126.41, 119.18, 91.58, 49.91; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{22}H_{17}N_2I$ 437.0509 found 437.0507.



4l

4-[1,1'-Biphenyl]-4-yl-2-phenyl-1-(phenylmethyl)-1*H*-imidazole (4l)

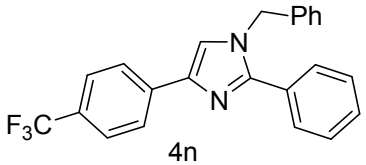
Purified by column chromatography (hexane/ethyl acetate 30:1); white solid (235 mg, 61% yield); ^{1}H NMR (400 MHz, DMSO) δ 7.92 (s, 1H), 7.91 (s, 2H), 7.70 (dd, *J* = 7.8, 2.8 Hz, 4H), 7.66–7.61 (m, 2H), 7.50-7.43 (m, 5H), 7.38–7.32 (m, 3H), 7.28 (dt, *J* = 8.5, 2.8 Hz, 1H), 7.11 (d, *J* = 7.2 Hz, 2H), 5.37 (s, 2H); ^{13}C NMR (101 MHz, DMSO) δ 147.87, 140.40, 140.09, 138.51, 138.01, 133.93, 130.98, 129.46, 129.33, 129.31, 129.13, 128.94, 128.13, 127.77, 127.30, 127.06, 126.88, 125.38, 119.62, 50.47; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{28}H_{22}N_2$ 387.1856 found 387.1864.



4m

1-Benzyl-4-(4-(methoxycarbonyl)phenyl)-2-phenyl-1*H*-imidazole (4m)

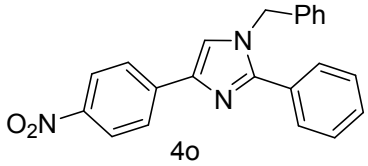
Purified by column chromatography (hexane/ethyl acetate 20:1); yellow solid (250 mg, 68% yield); ^{1}H NMR (400 MHz, DMSO) δ 8.03 (s, 1H), 7.96 (s, 4H), 7.65–7.60 (m, 2H), 7.50–7.44 (m, 3H), 7.34 (tt, *J* = 7.6, 1.2 Hz, 2H), 7.27 (tt, *J* = 7.2, 2.8 Hz, 1H), 7.10 (d, *J* = 7.1 Hz, 2H), 5.37 (s, 2H), 3.85 (s, 3H); ^{13}C NMR (101 MHz, DMSO) δ 166.60, 148.28, 139.32, 139.25, 137.73, 130.67, 130.14, 129.42, 129.29, 129.10, 128.93, 128.13, 127.65, 127.05, 124.70, 121.21, 52.44, 50.51; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{24}H_{20}N_2O_2$ 369.1598 found 369.1603.



4n

1-Benzyl-4-(4-(trifluoromethyl)phenyl)-2-phenyl-1*H*-imidazole (4n)

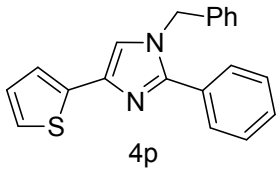
Purified by column chromatography (hexane/ethyl acetate 20:1); yellow solid (246 mg, 65% yield); ^{1}H NMR (400 MHz, DMSO) δ 8.04 (t, *J* = 4.0 Hz, 3H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.66–7.59 (m, 2H), 7.51–7.43 (m, 3H), 7.34 (t, *J* = 7.3 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 1H), 7.10 (d, *J* = 7.2 Hz, 2H), 5.37 (s, 2H); ^{13}C NMR (101 MHz, DMSO) δ 147.78, 138.43, 138.17, 137.22, 130.18, 128.92, 128.78, 128.59, 128.45, 127.62, 126.66, 126.55, 126.34, 125.84, 125.47 (q, *J* = 3.8 Hz), 124.63, 123.14, 120.52, 50.01; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{23}H_{17}N_2F_3$ 379.1417 found 379.1445.



4o

1-benzyl-4-(4-nitrophenyl)-2-phenyl-1H-imidazole (4o)

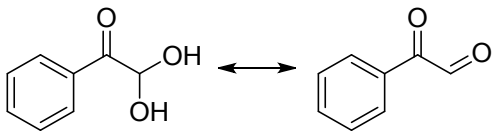
Purified by column chromatography (hexane/ethyl acetate 20:1); yellow solid (209 mg, 59% yield); ^{1}H NMR (500 MHz, DMSO) δ 8.24 (d, *J* = 8.9 Hz, 2H), 8.15 (s, 1H), 8.07 (d, *J* = 8.9 Hz, 2H), 7.65 – 7.61 (m, 2H), 7.50 – 7.45 (m, 3H), 7.34 (t, *J* = 7.4 Hz, 2H), 7.28 (t, *J* = 7.3 Hz, 1H), 7.11 (d, *J* = 7.3 Hz, 2H), 5.38 (s, 2H). ^{13}C NMR (126 MHz, DMSO) δ 148.24, 145.42, 140.81, 137.85, 136.99, 129.95, 129.02, 128.75, 128.56, 128.45, 127.63, 126.57, 124.75, 124.07, 121.95, 50.09; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{22}H_{17}N_3O_2$ 356.1394 found 356.1400.



4p

1-Benzyl-4-thienyl-2-phenyl-1*H*-imidazole (4p)

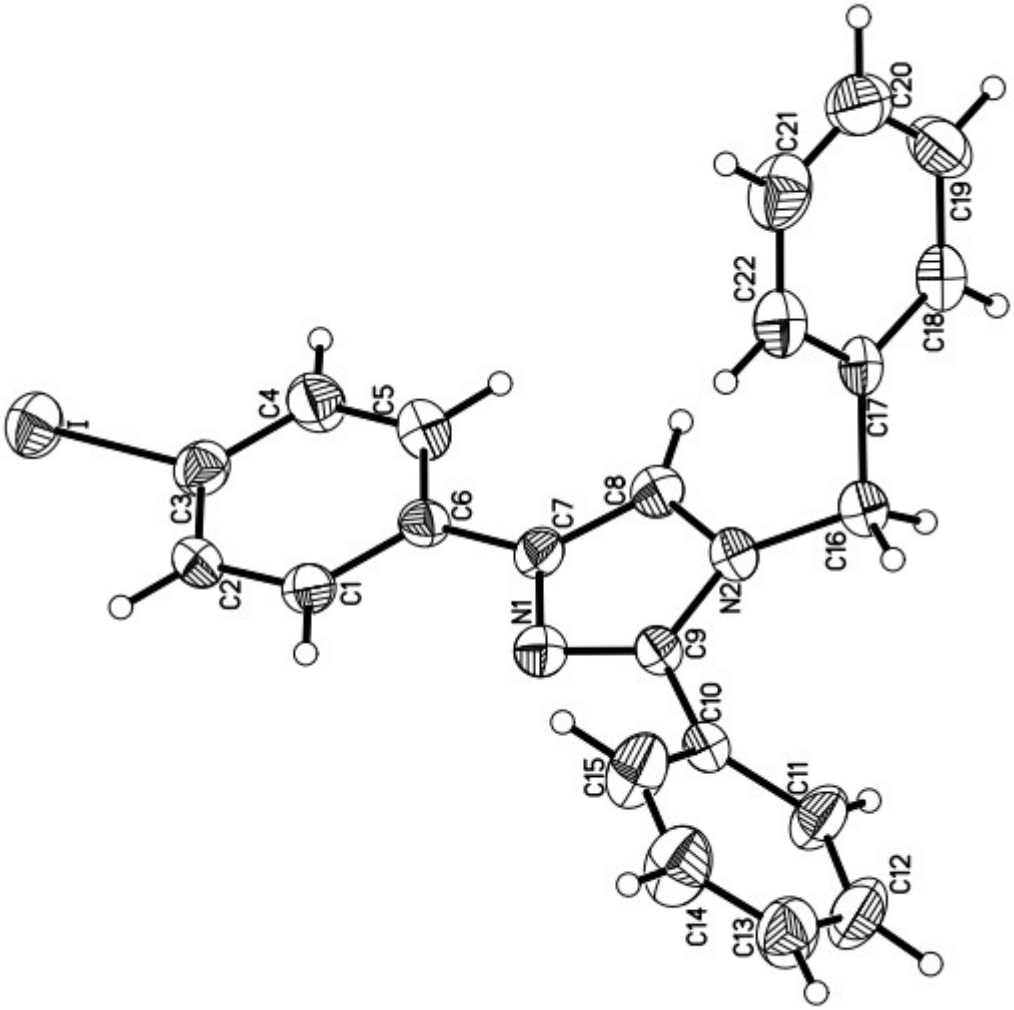
Purified by column chromatography (hexane/ethyl acetate 30:1); yellow solid (215 mg, 68% yield); ^{1}H NMR (500 MHz, DMSO) δ 7.68 (s, 1H), 7.59 (dd, *J* = 7.6, 1.8 Hz, 2H), 7.48–7.42 (m, 3H), 7.39–7.30 (m, 4H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.09 (d, *J* = 7.3 Hz, 2H), 7.05 (dd, *J* = 5.0, 3.6 Hz, 1H), 5.33 (s, 2H); ^{13}C NMR (126 MHz, DMSO) δ 147.11, 138.24, 137.41, 135.66, 130.23, 128.93, 128.86, 128.68, 128.54, 127.75, 127.71, 126.66, 123.58, 121.64, 117.83, 49.98; HRMS (TOF) m/z [M + H]$^{+}$ Calcd for $C_{20}H_{16}N_2S$ 317.1107 found 317.1111.



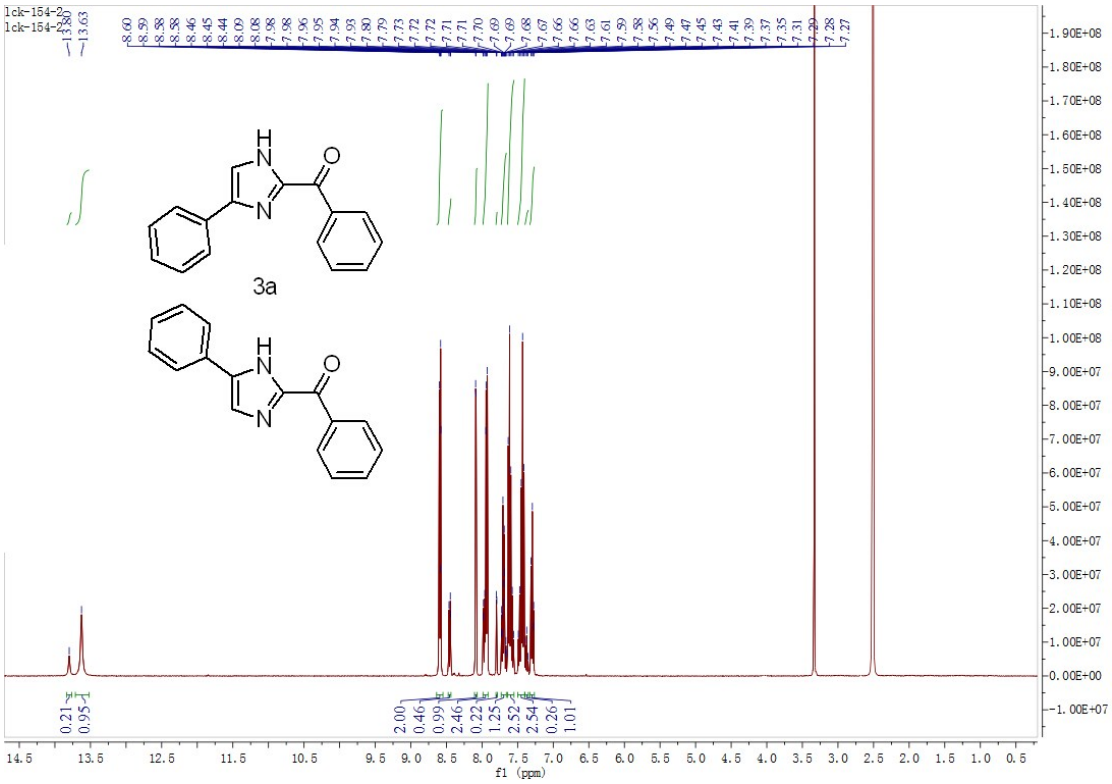
Ⅳ a Ⅳ b

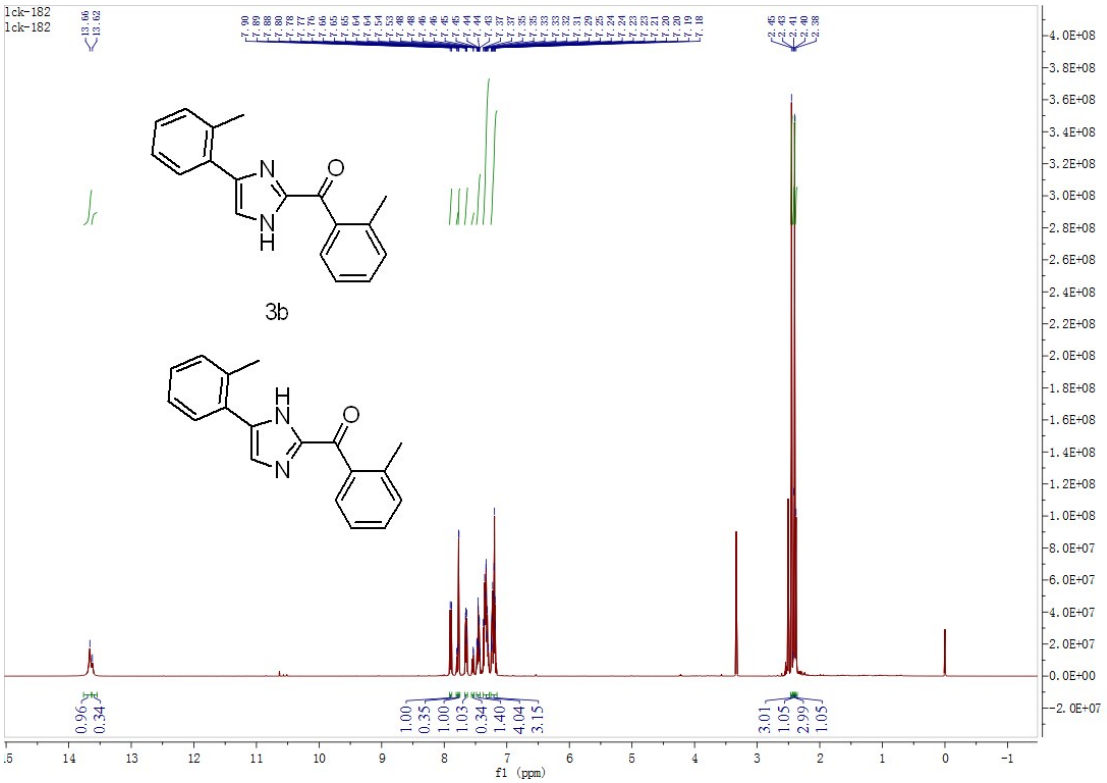
Purified by column chromatography (hexane/ethyl acetate 6:1); yellow solid.
^{1}H NMR (500 MHz, DMSO) δ 9.59 (s, 0.4H), 8.11 (d, *J* = 7.8 Hz, 2.8H), 7.76 (t, *J* = 7.4 Hz, 0.4H), 7.64 (dt, *J* = 15.5, 7.6 Hz, 1.8H), 7.55 (t, *J* = 7.7 Hz, 2H), 6.73 (d, *J* = 6.8 Hz, 2H), 5.72 (s, 1H).HRMS (TOF) m/z [M - H]$^-$ Calcd for $C_8H_8O_3$ 151.0401 found 151.0399.

The Picture of the Single-Crystal for 4k



^{1}H NMR and ^{13}C NMR Spectra of those Compounds

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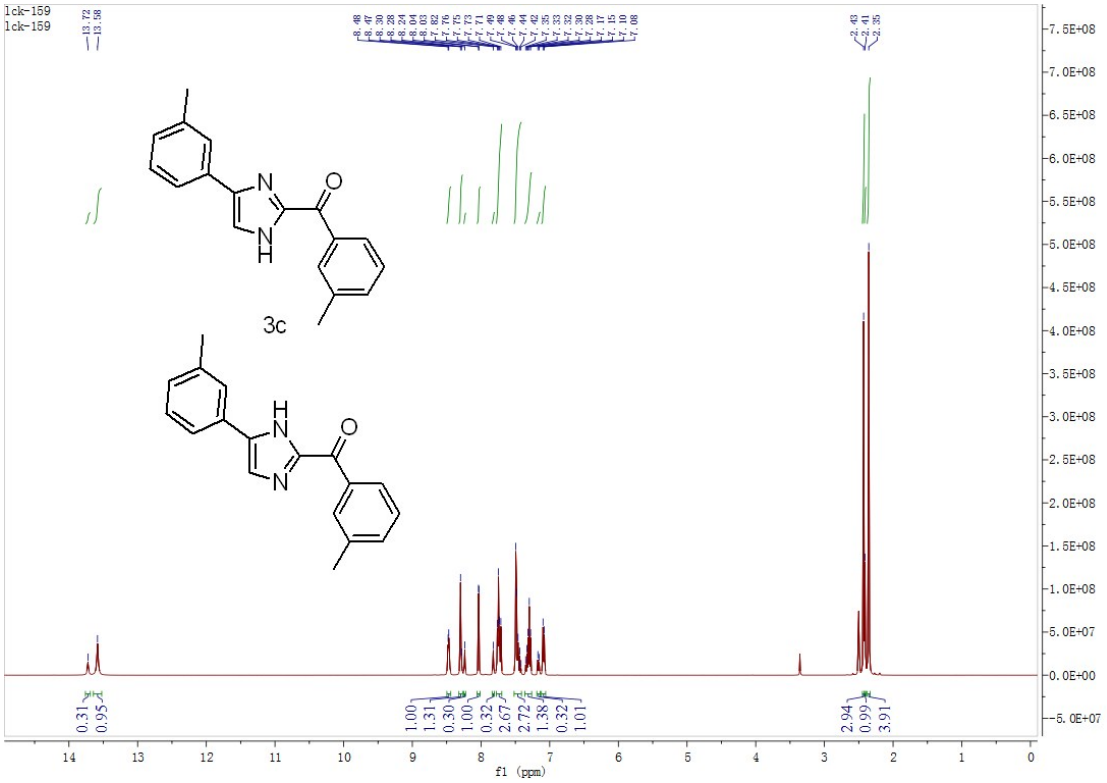
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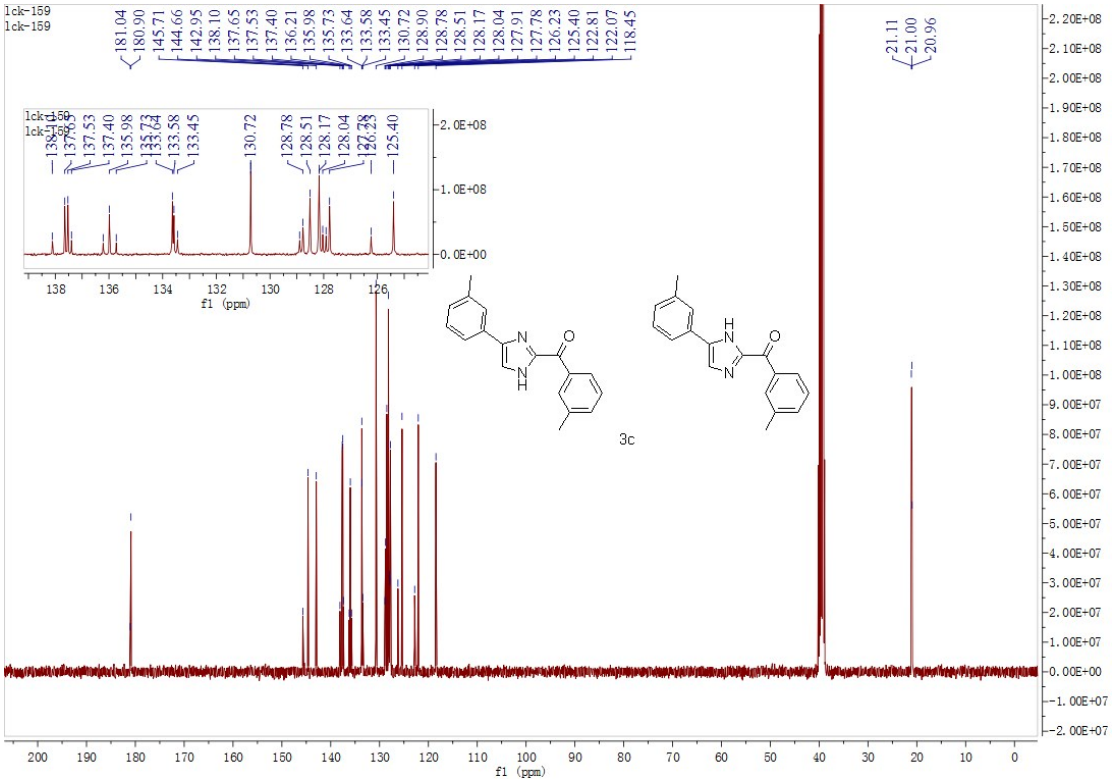
lck-159
3c
f1 (ppm)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lck-159
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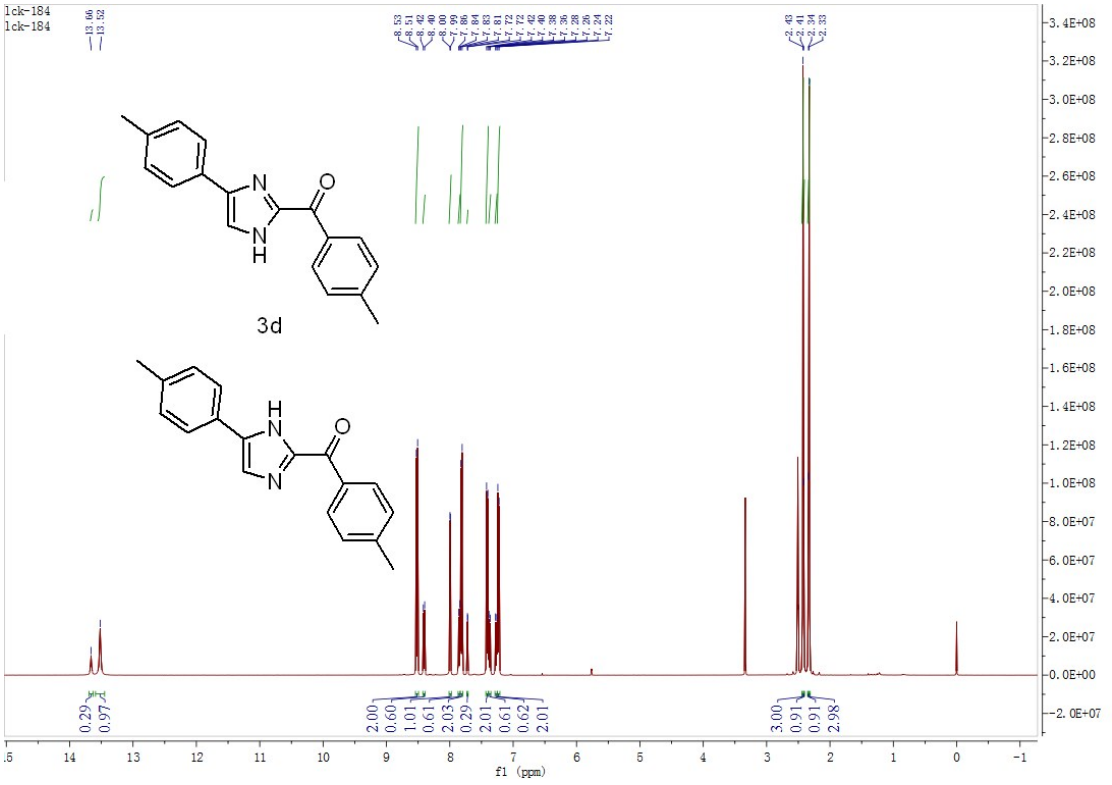
21.00
20.96
f1 (ppm)
3c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lck-184
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7.72
7.42
7.40
7.38
7.36
7.26
7.24
7.22
2.43
2.41
2.34
2.33
0.29
0.97
2.00
0.60
1.01
0.61
2.03
0.29
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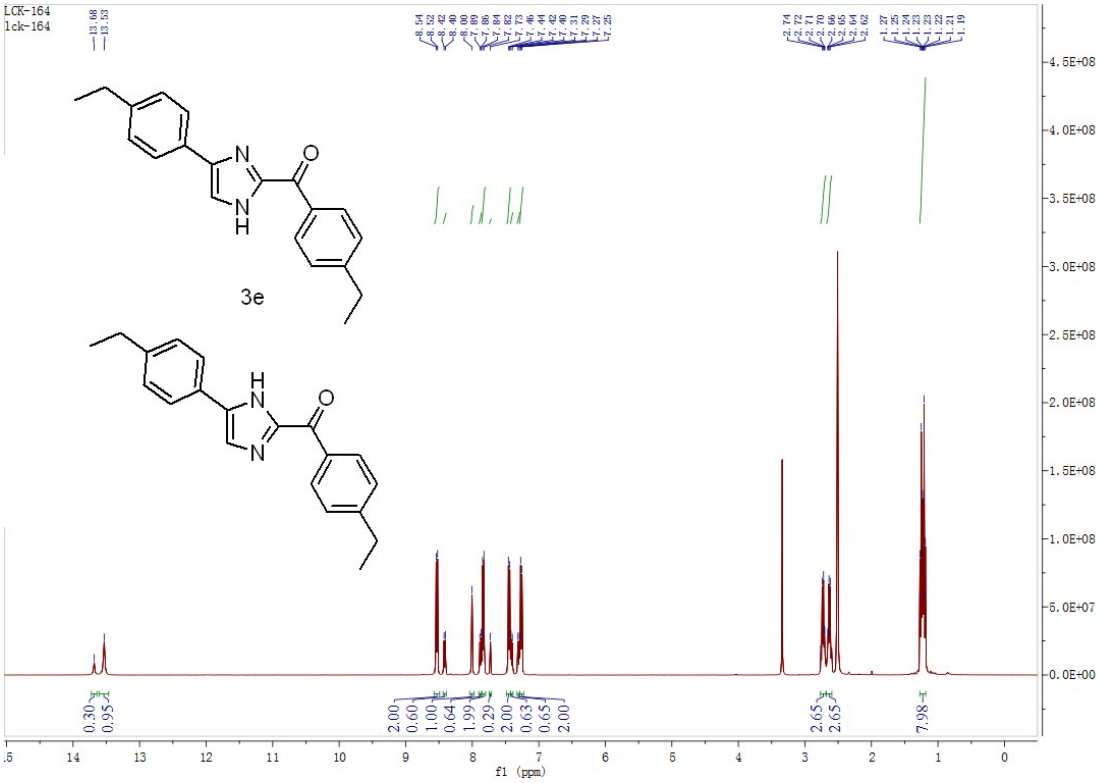
2.01
3.00
0.91
0.91
2.98
f1 (ppm)
3d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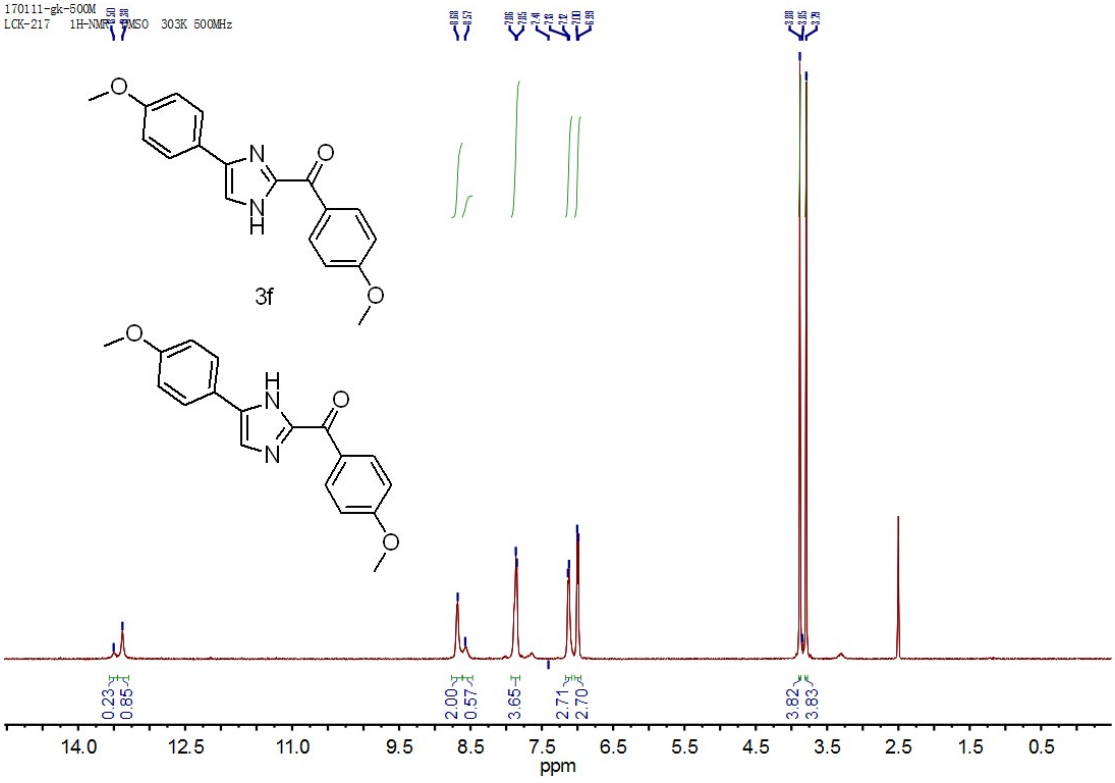
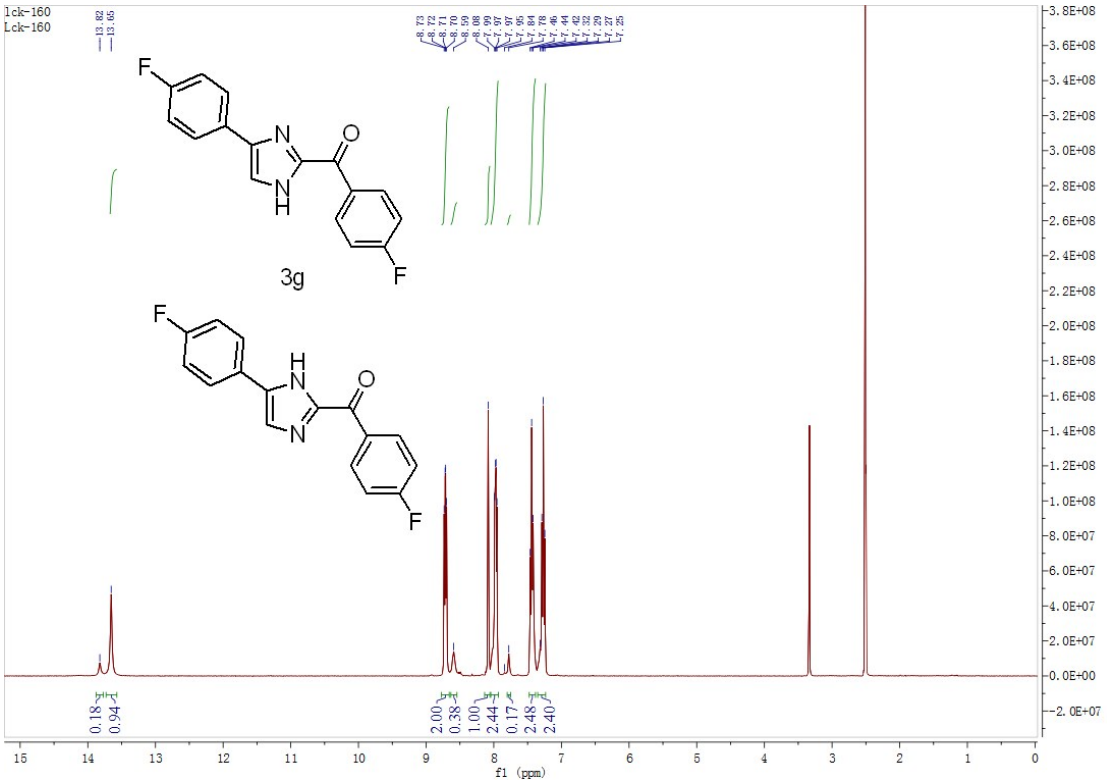
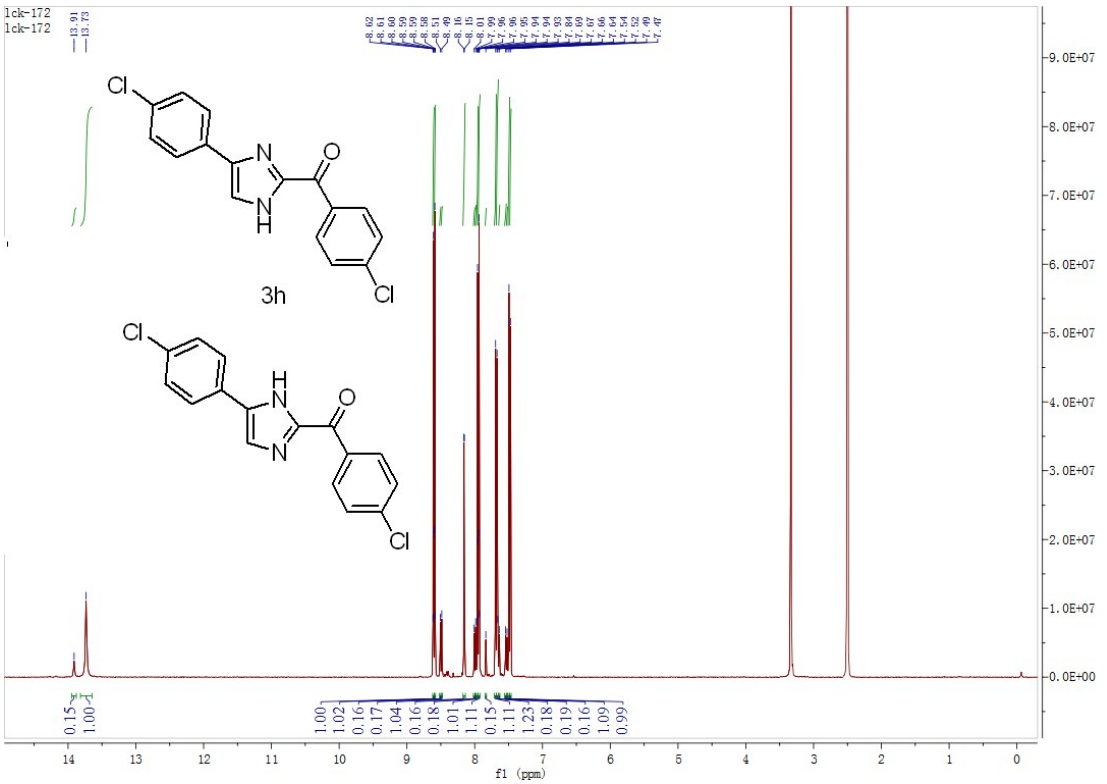
lck-164
3e
f1 (ppm)


170111-gk-500M
LCK-217 1H-NMR DMSO 303K 500MHz
3f
ppm


lck-160
Lck-160
F
N
O
N
H
F
3g
F
H
N
O
N
F
f1 (ppm)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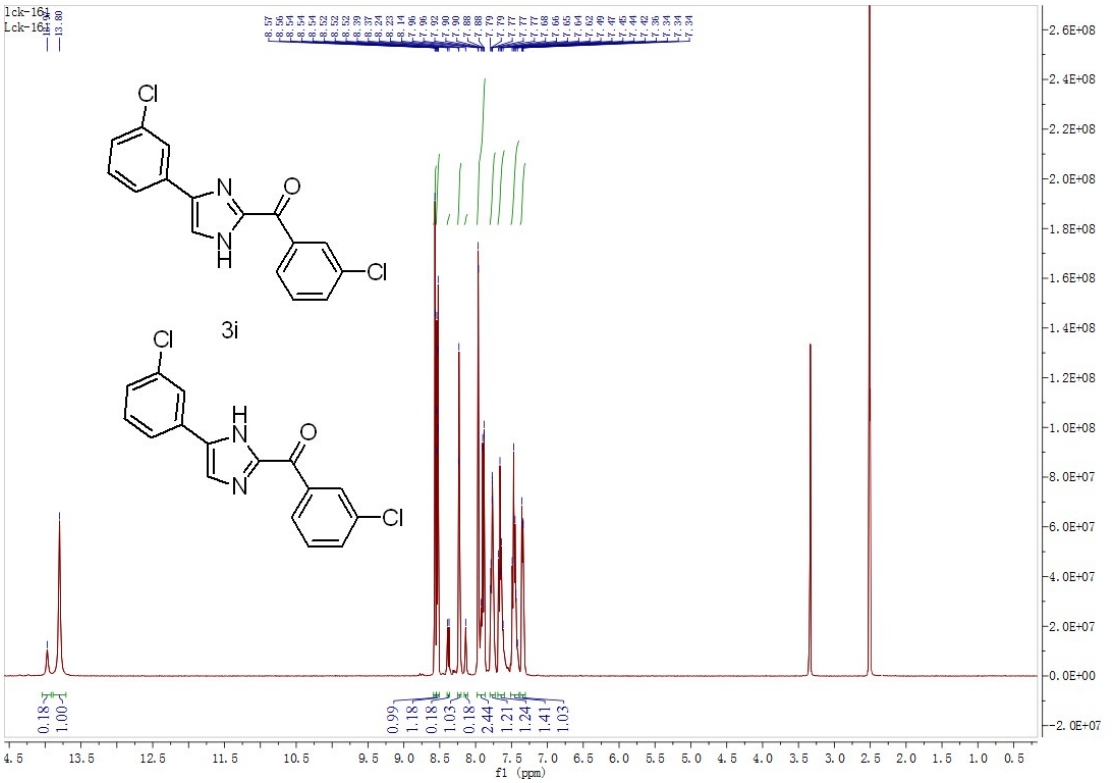
N
H
Cl
3h
Cl
H
N
O
N
Cl
f1 (ppm)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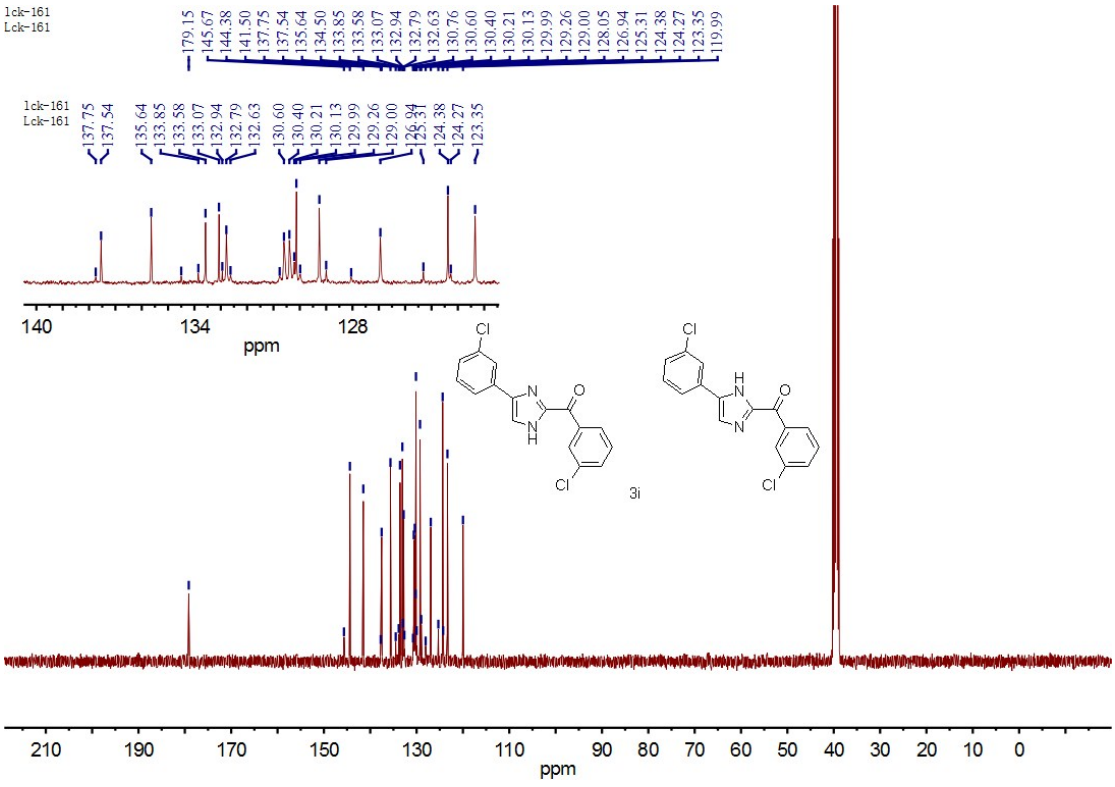
Cl
3i
Cl
H
N
O
N
Cl
f1 (ppm)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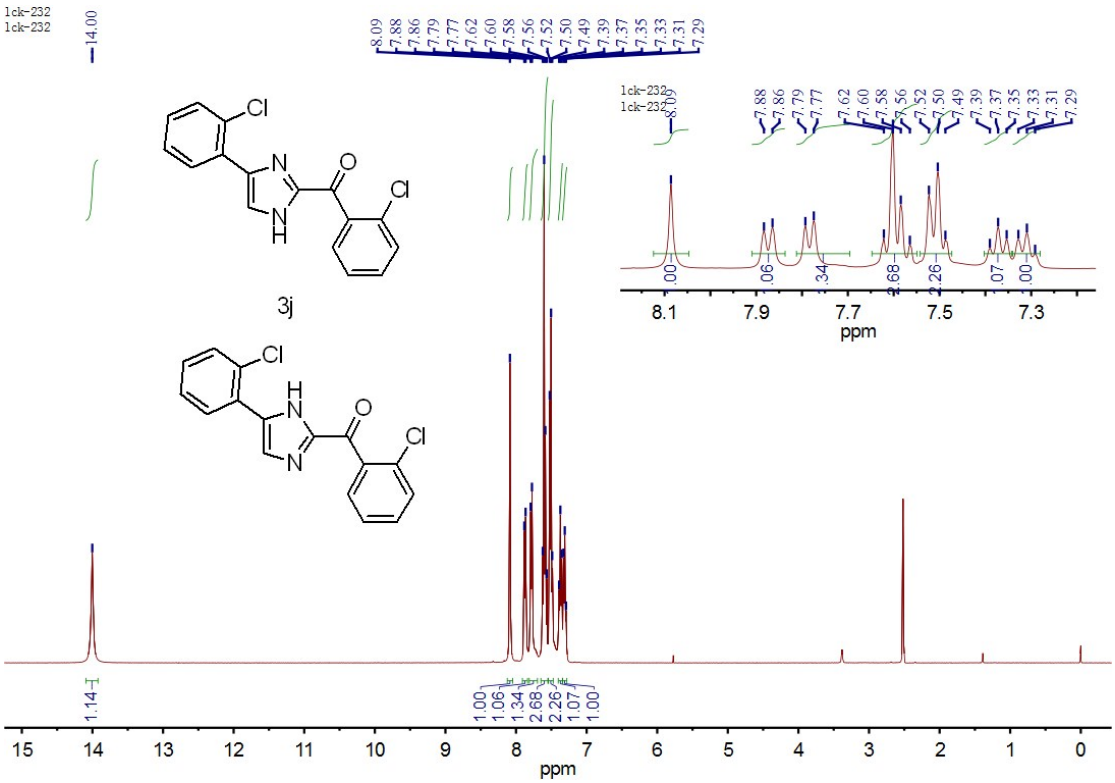
H
Cl
3i
Cl
H
N
O
N
Cl
ppm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7.49
7.39
7.37
7.35
7.33
7.31
7.29
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Cl
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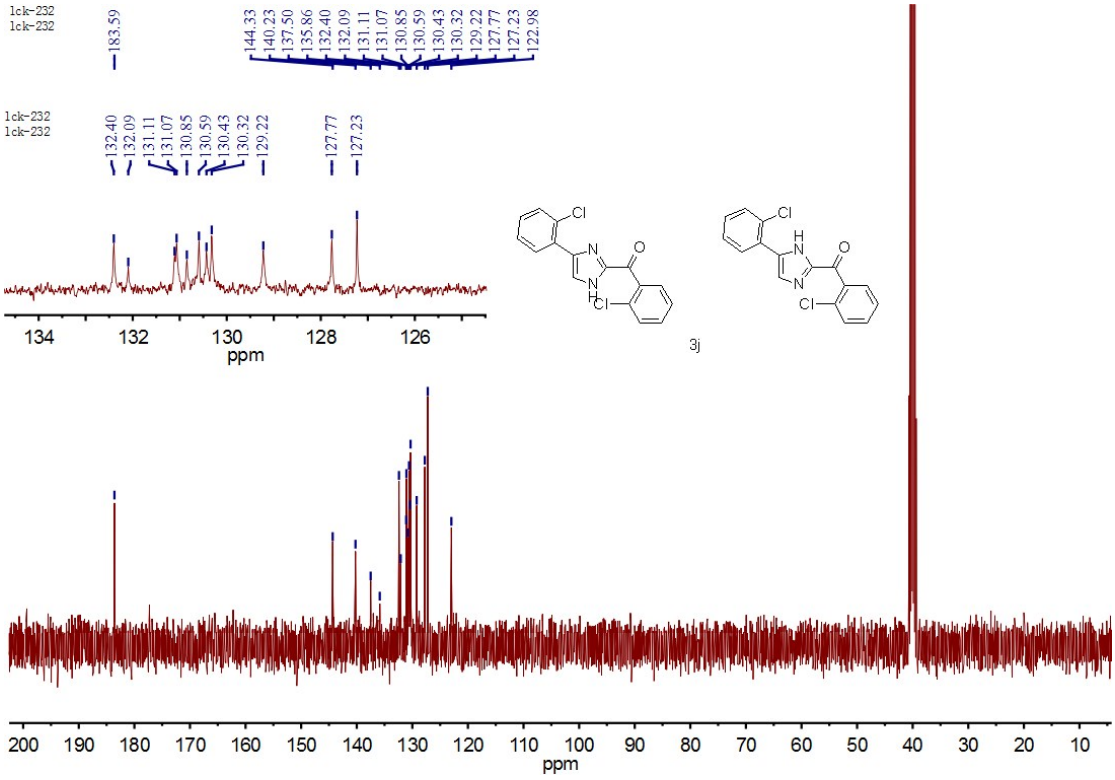
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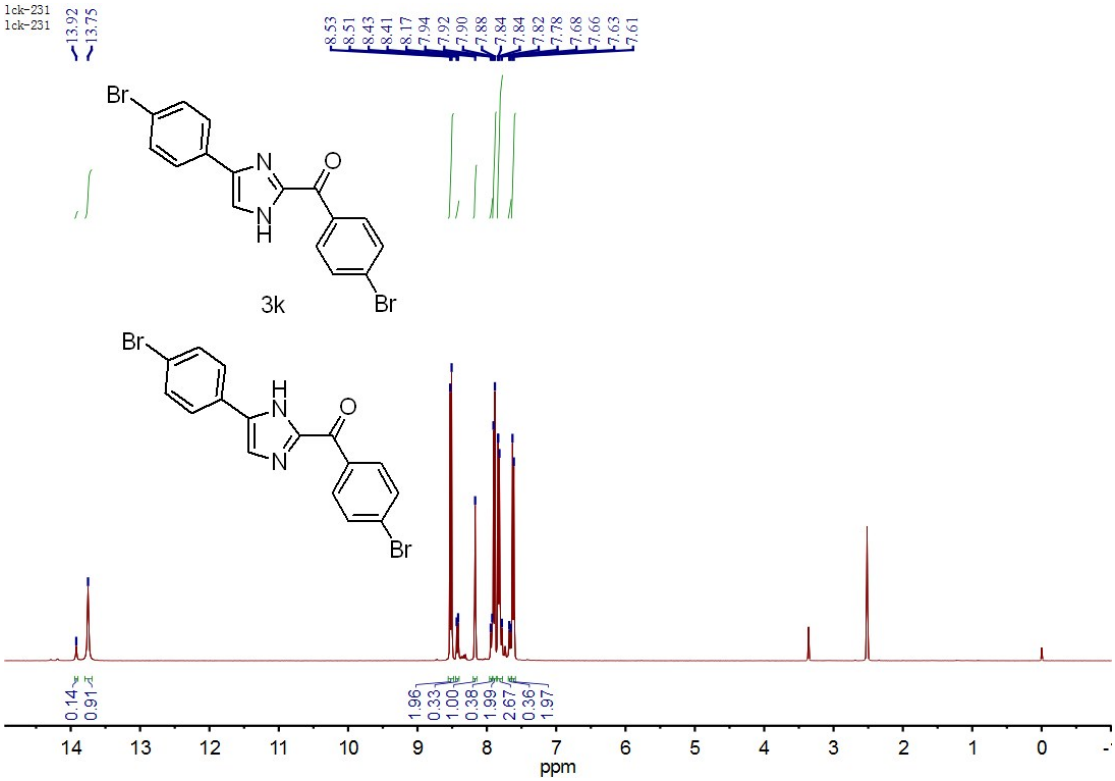
ppm
3j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7.66
7.63
7.61
Br
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O
N
H
Br
3k
Br
H
N
O
N
Br
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0.91
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0.33
1.00
0.38
1.99
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6
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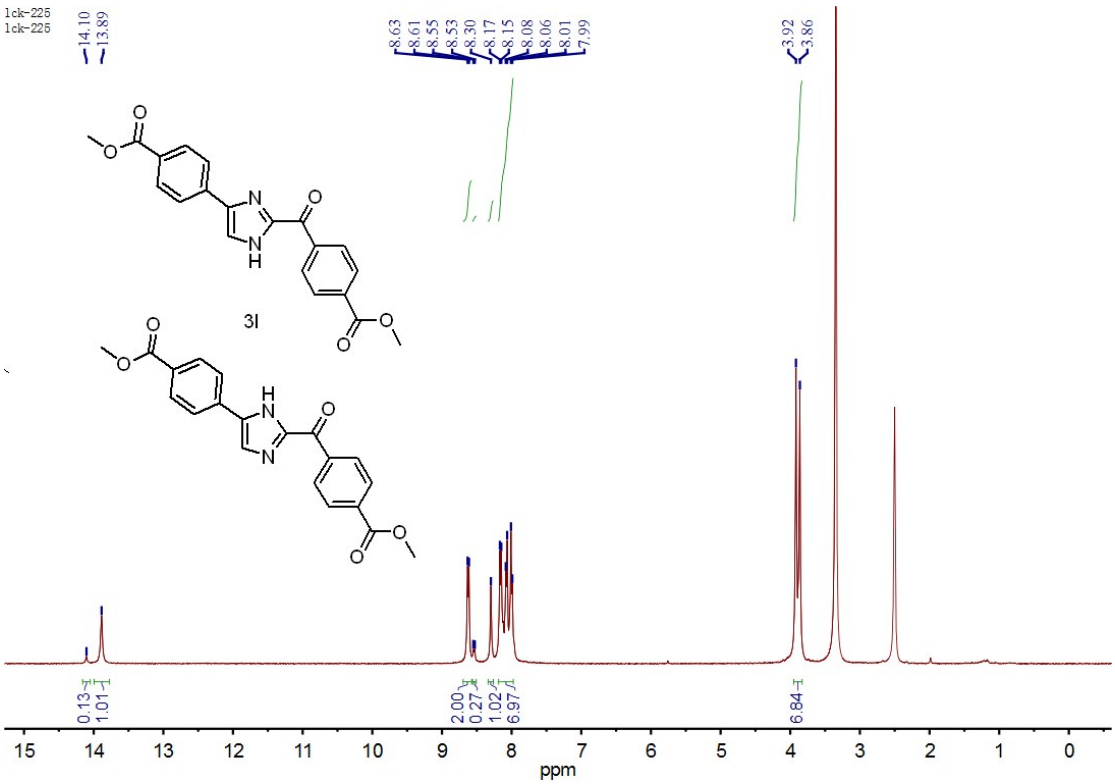
0
ppm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8.08
8.06
8.01
7.99
3.92
3.86
O
O
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O
N
H
O
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3l
O
O
H
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2.00
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1.02
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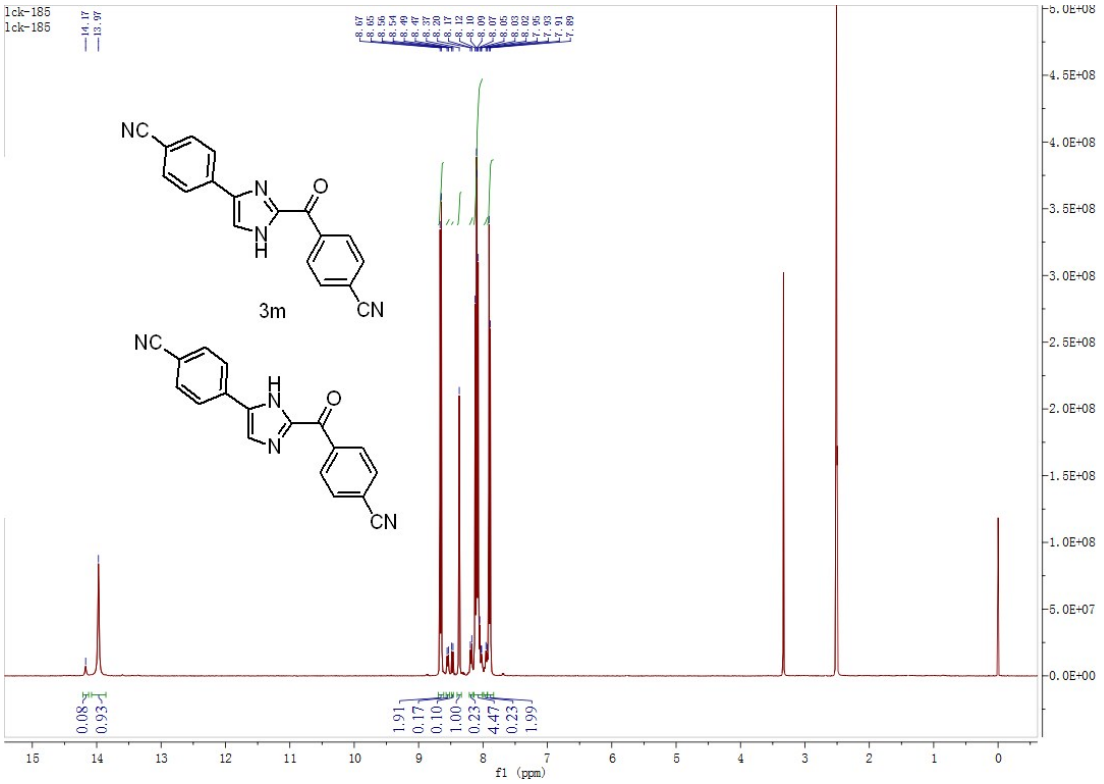
ppm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lck-185
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3m
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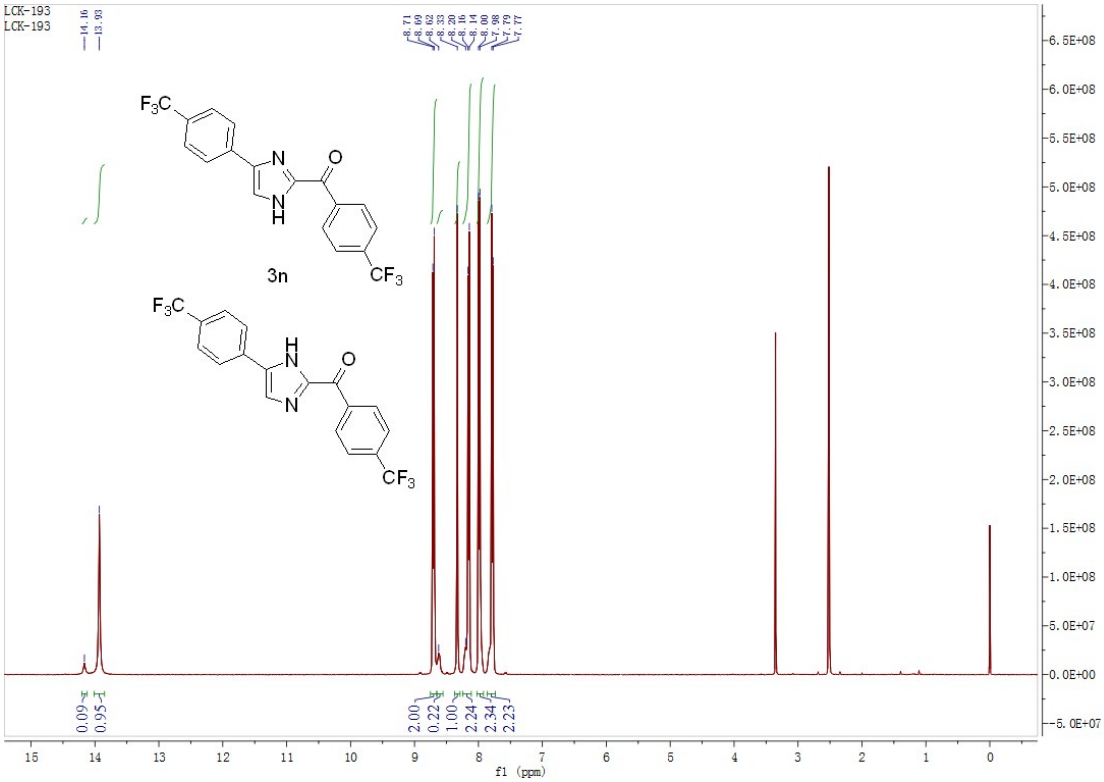
H
N
O
CN
f1 (ppm)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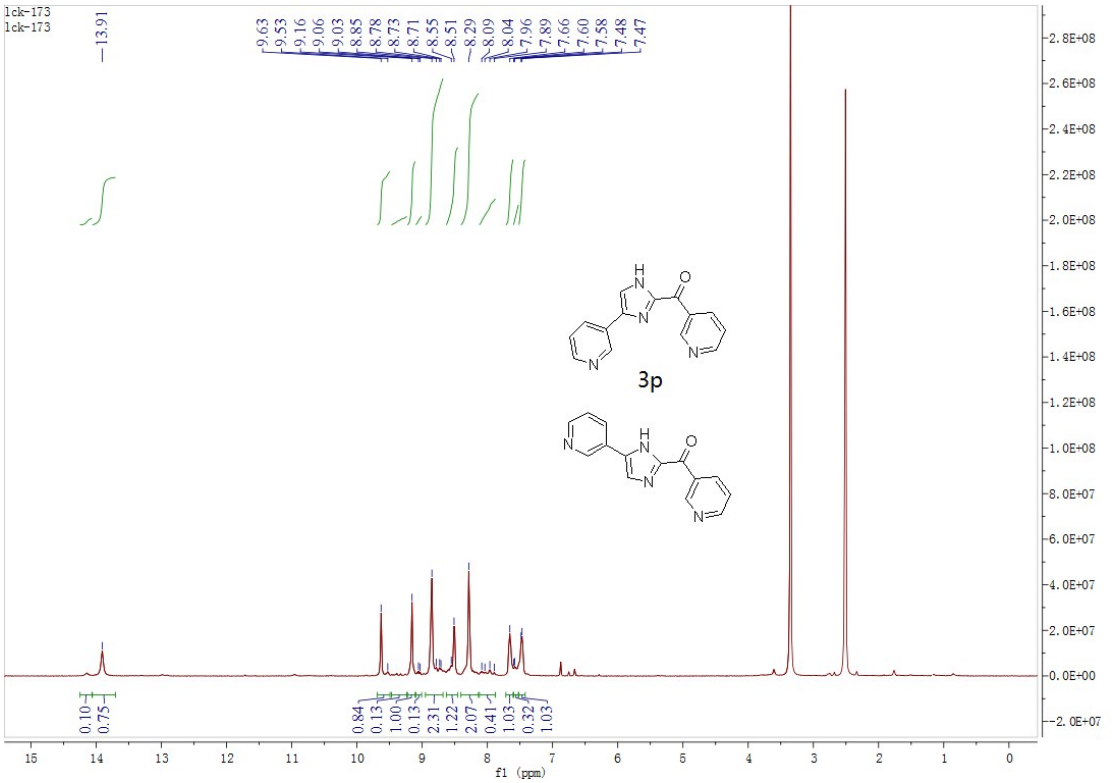
f1 (ppm)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8.51
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8.04
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7.60
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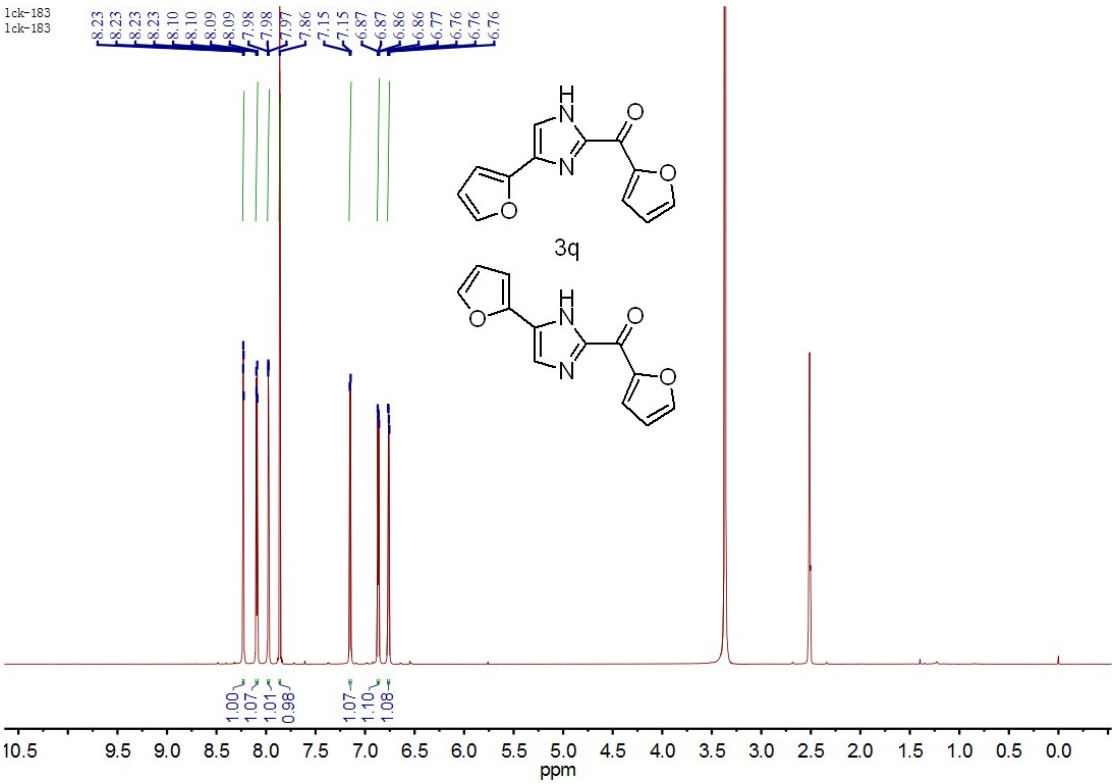
7.47
3p
f1 (ppm)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6.76
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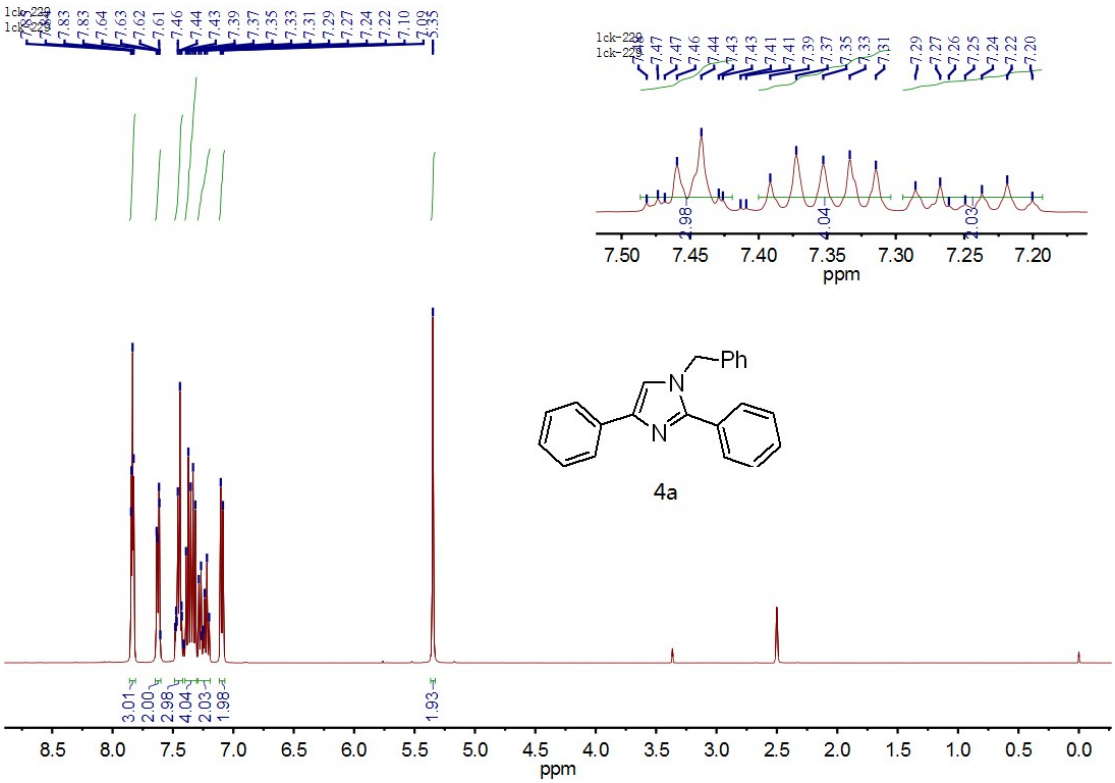
6.76
3q
ppm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1.98
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2.0
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1.0
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Ph
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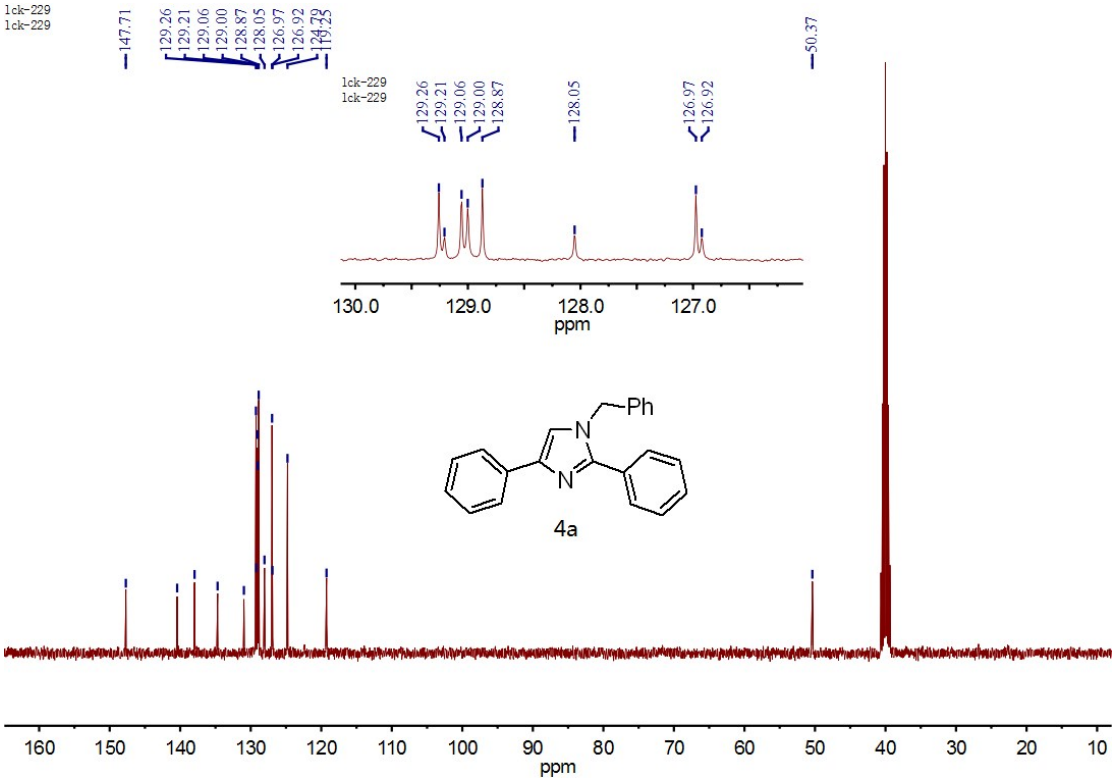
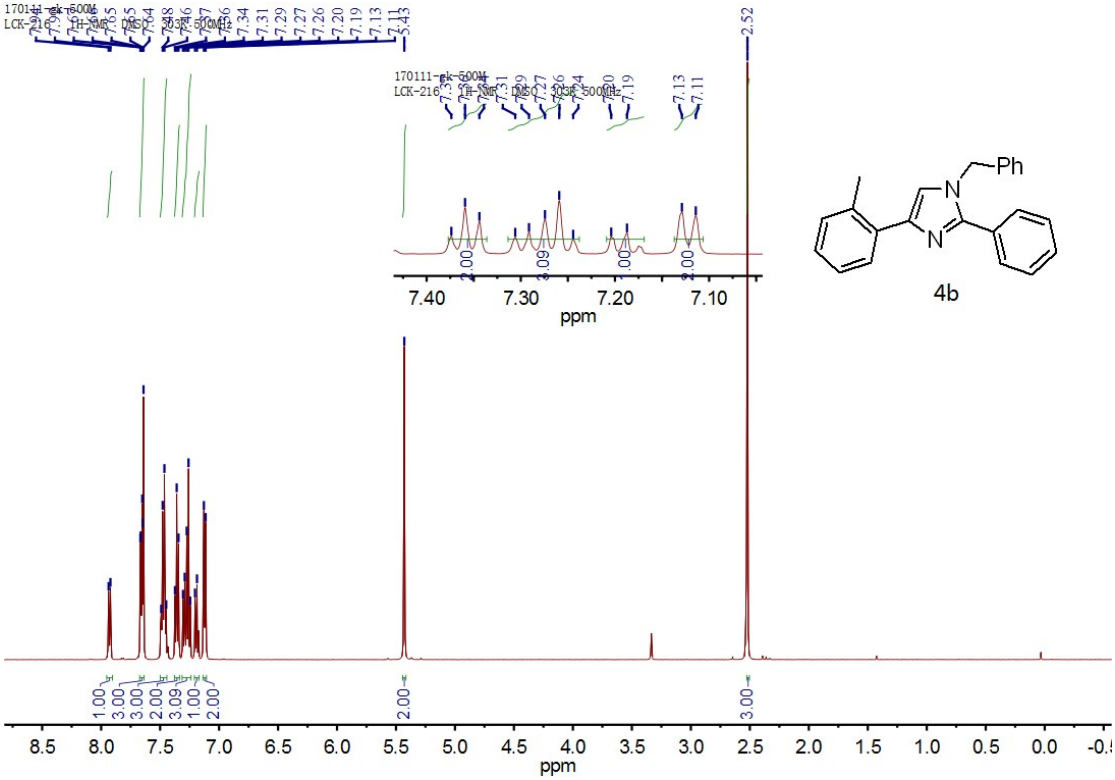
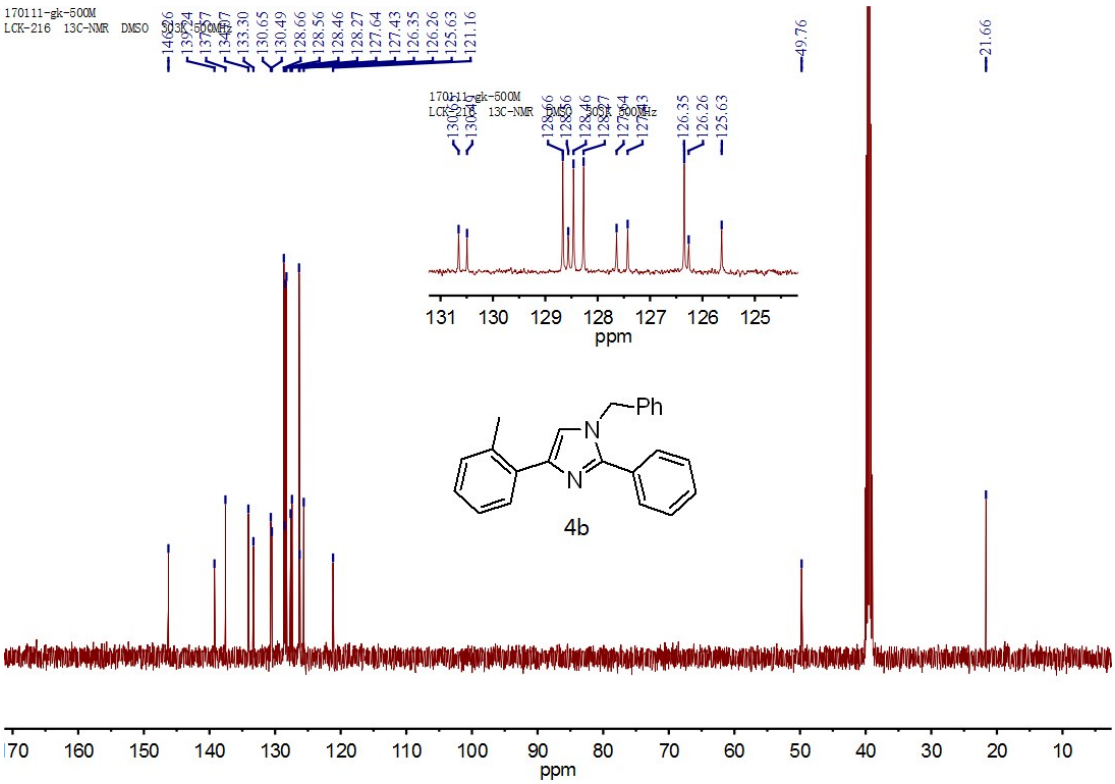
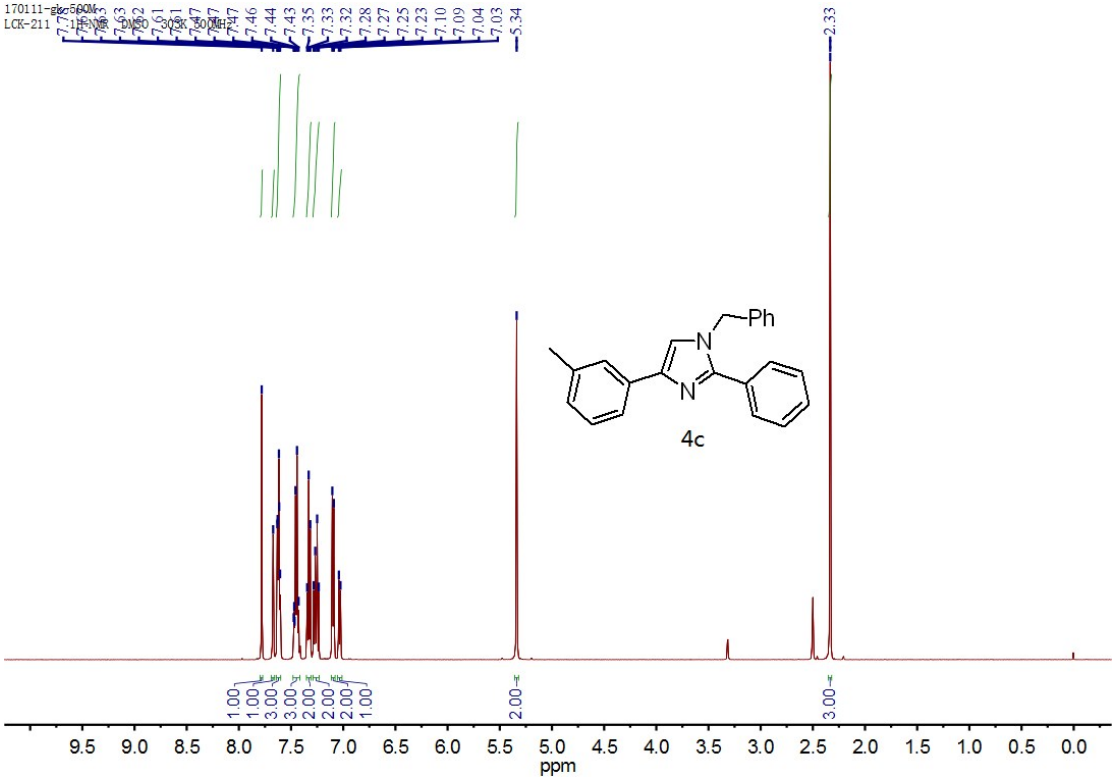
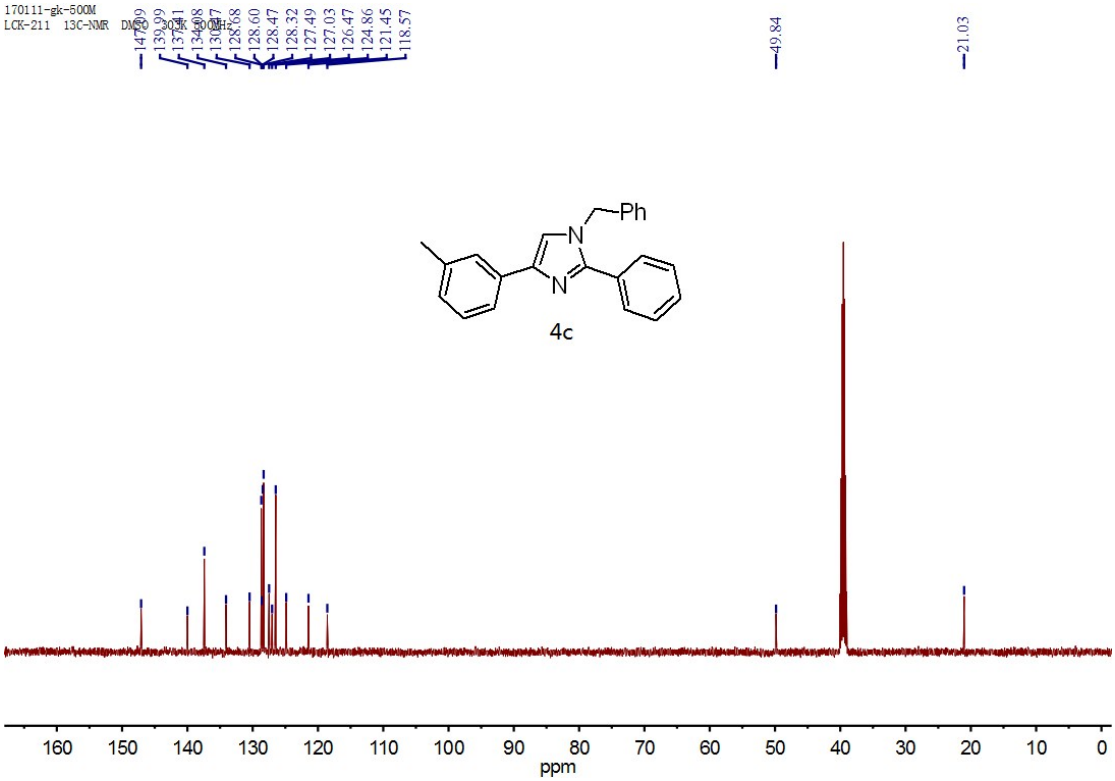
N
4a

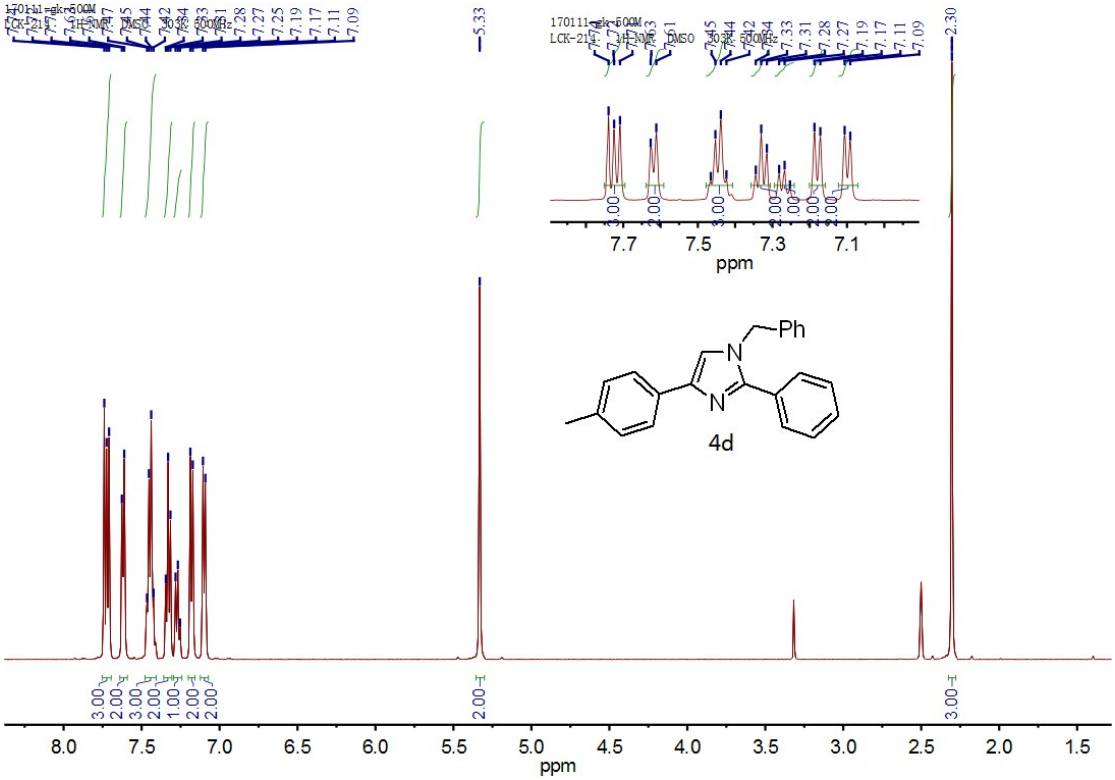

1ck-229
1ck-229
147.71
129.26
129.21
129.06
129.00
128.87
128.05
126.97
126.92
124.29
119.25
50.37
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
130.0
129.0
128.0
127.0
ppm
Ph
N
N
4a

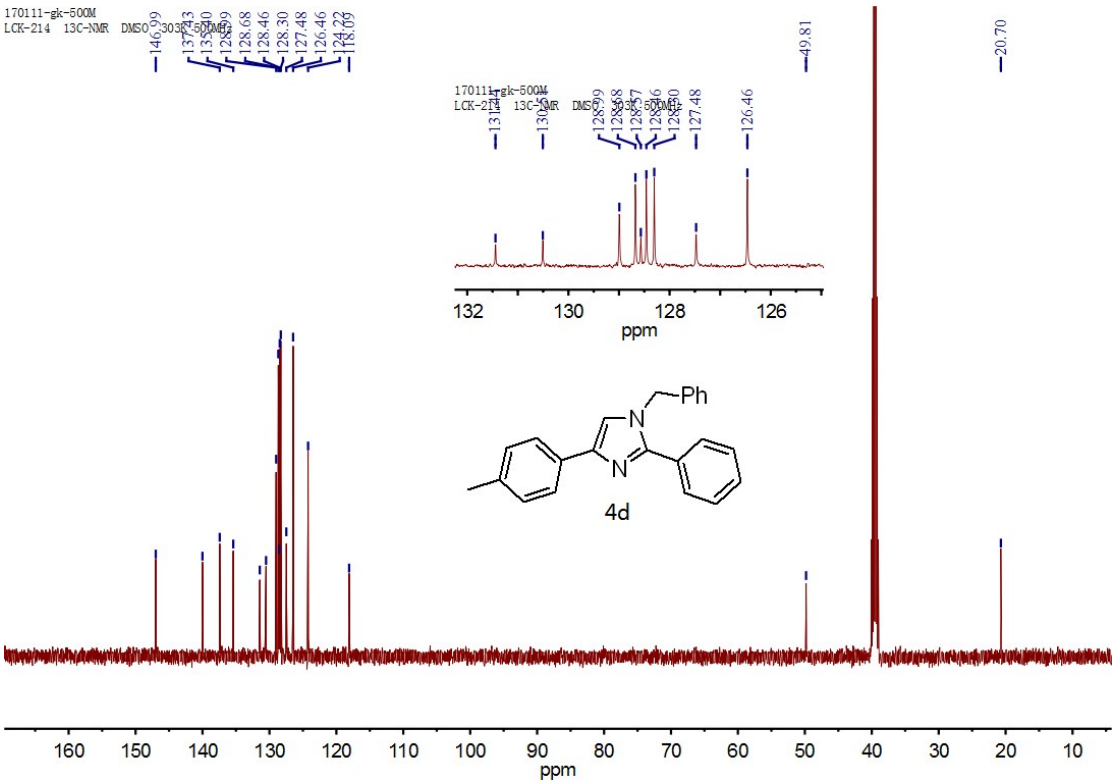

Ph
N
N
4b
ppm

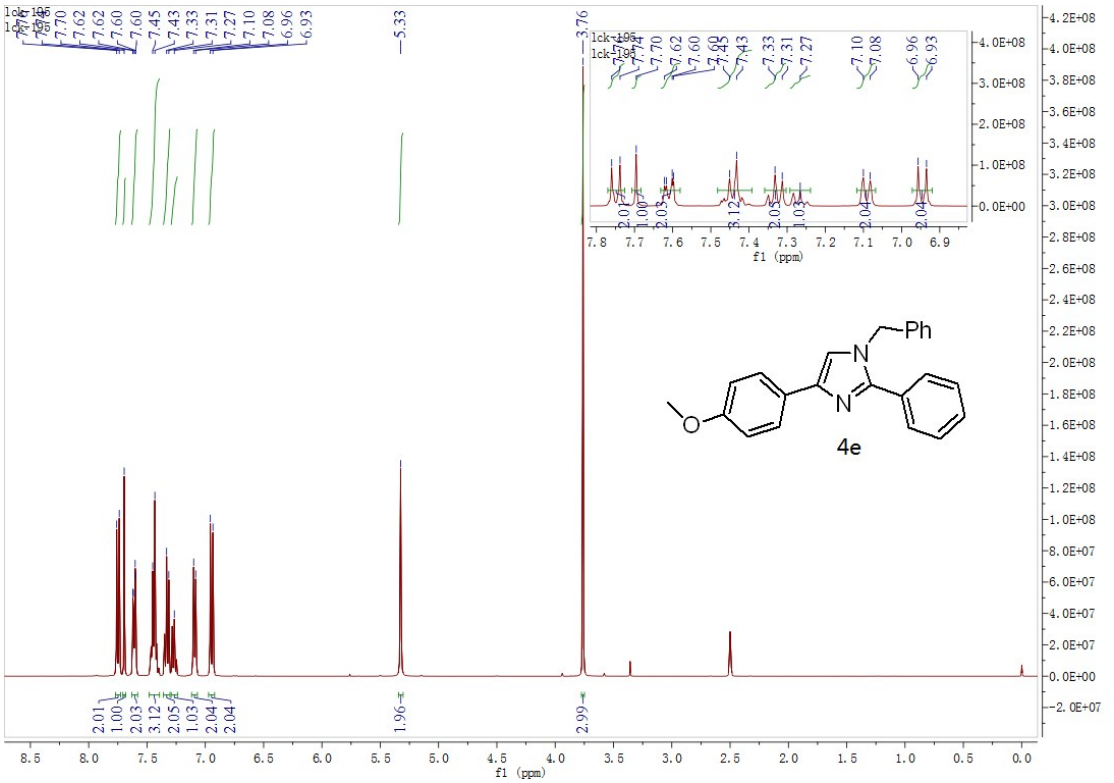

Ph
N
N
4b
ppm

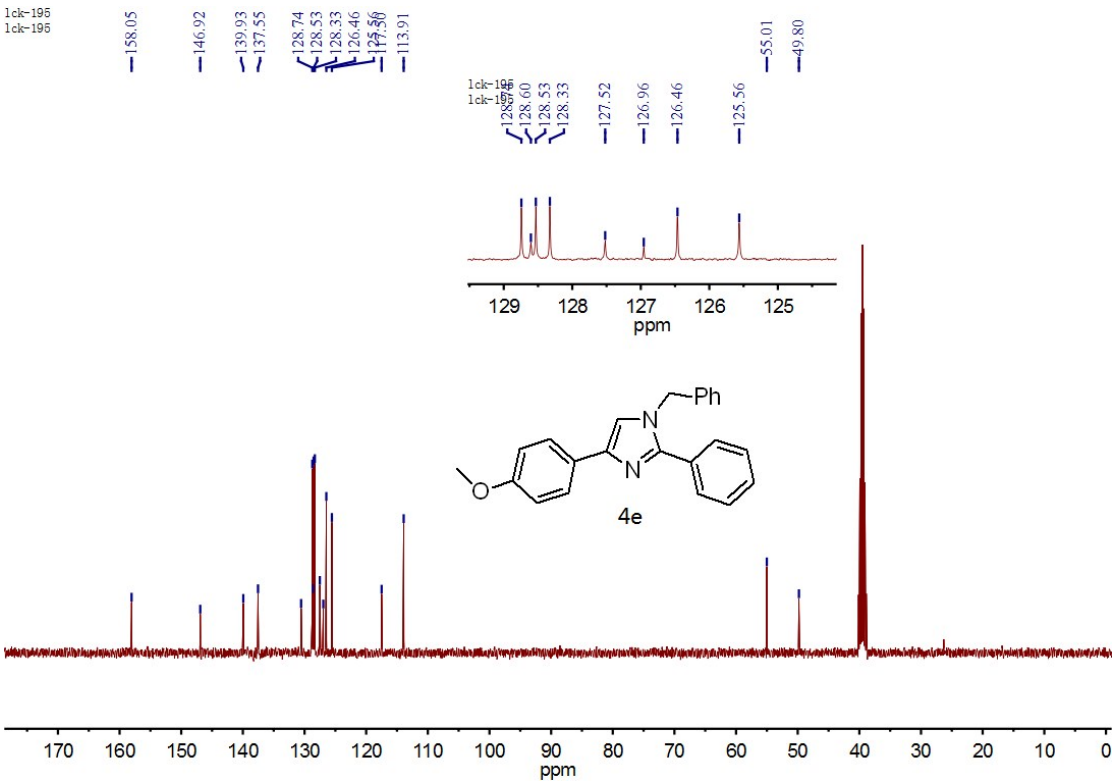

170111-gk-500M
LCK-211
2.33
5.34
Ph
N
N
4c
1.00
1.00
3.00
2.00
2.00
1.00
2.00
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm

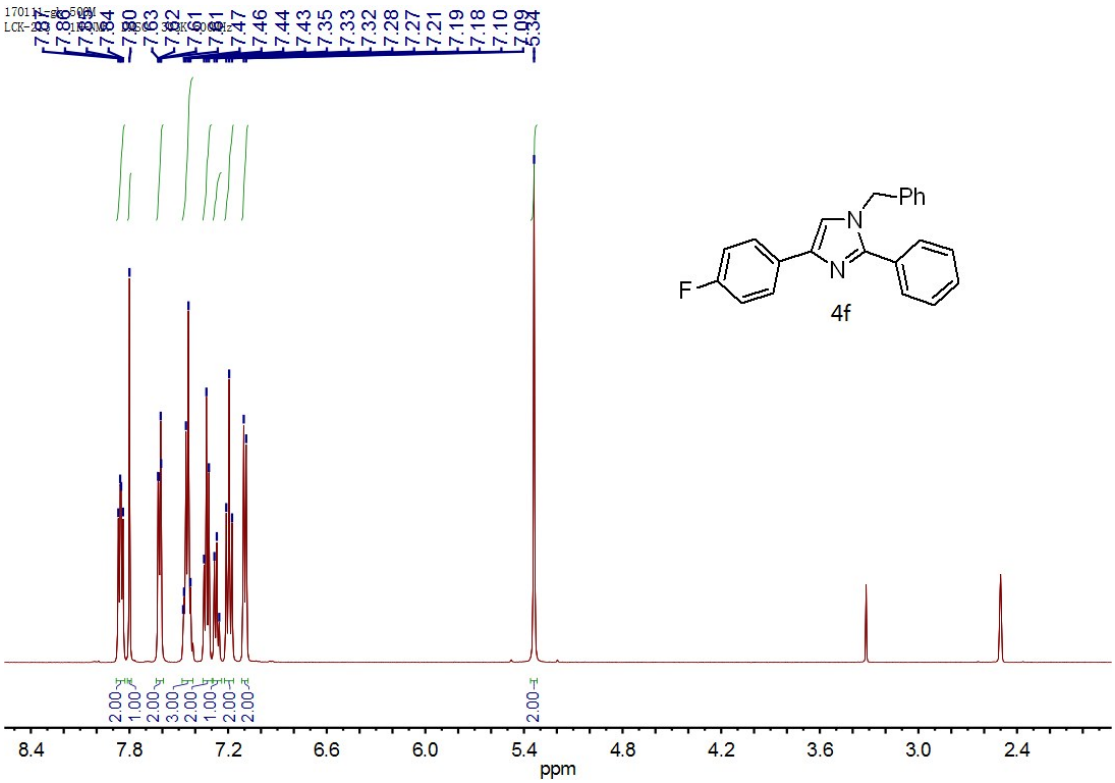

170111-gk-500M
LCK-211 13C-NMR DMSO 303K 500MH
147.09
139.99
137.41
134.08
130.47
128.68
128.60
128.47
128.32
127.49
127.03
126.47
124.86
121.45
118.57
49.84
21.03
Ph
N
N
4c
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

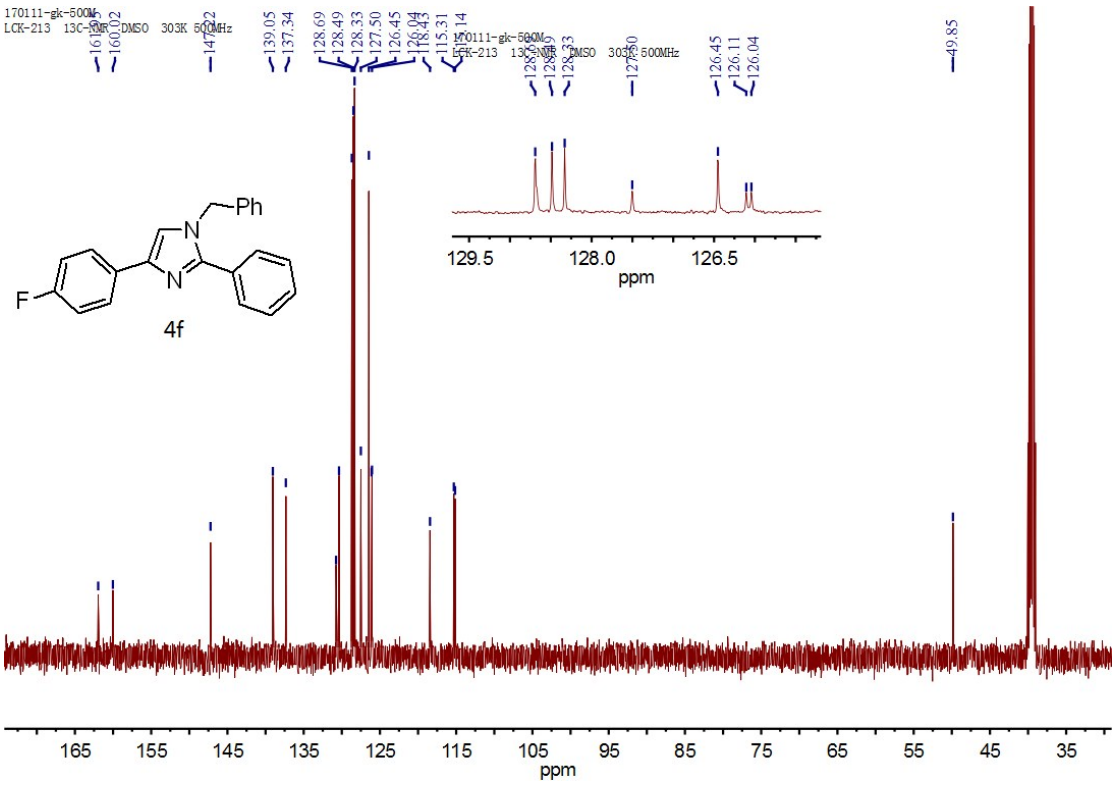

170111-gk-500M
LCK-214 1H-NMR DMSO 303K 500MHz
5.33
2.30
7.7
7.5
7.3
7.1
ppm
Ph
N
N
4d
3.00
2.00
3.00
2.00
1.00
2.00
2.00
2.00
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm

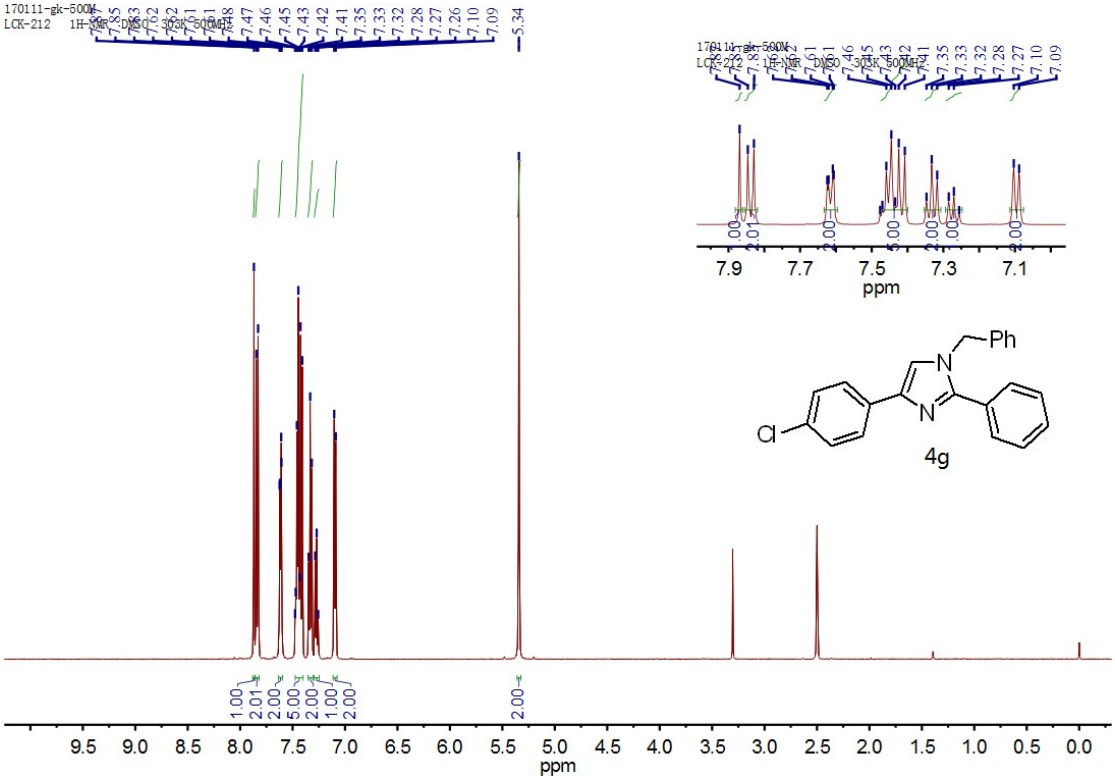

170111-gk-500M
LCK-214 13C-NMR DMSO 303K 500MHz
140.99
137.43
135.40
128.99
128.68
128.46
128.30
127.48
126.46
124.22
118.09
49.81
20.70
132
130
128
126
ppm
Ph
N
N
4d
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

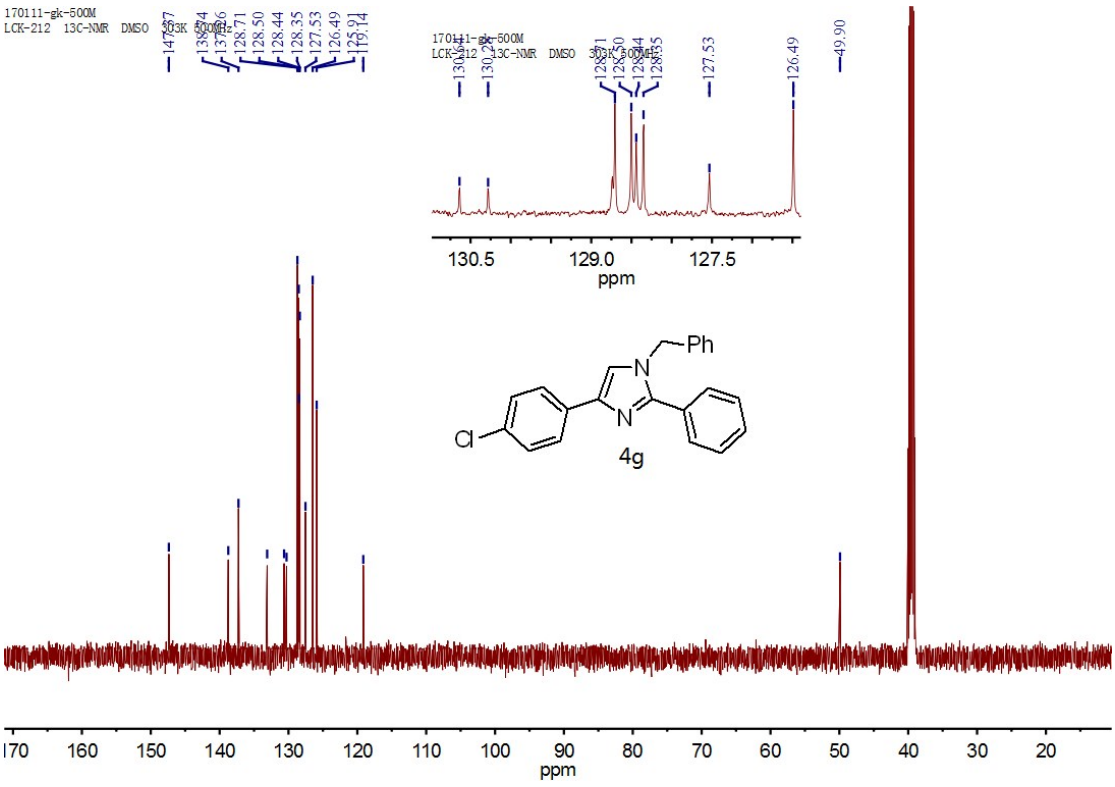

Ph
N
N
O
4e
f1 (ppm)

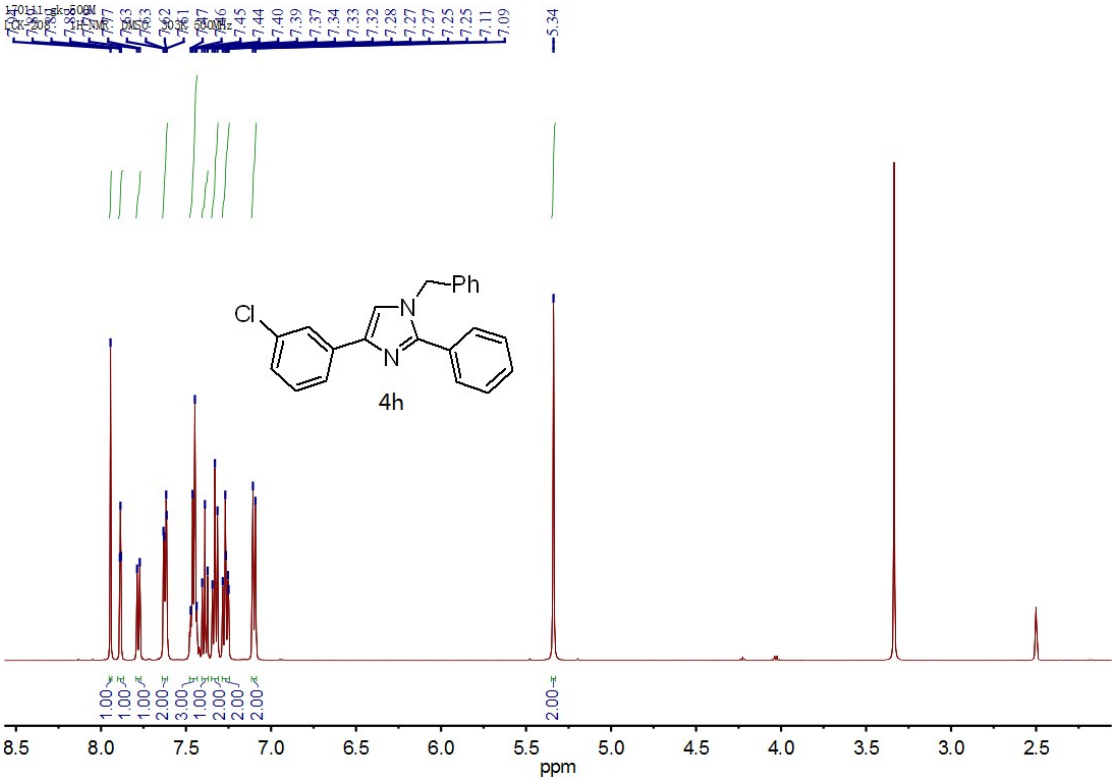

Ph
N
N
O
4e
ppm

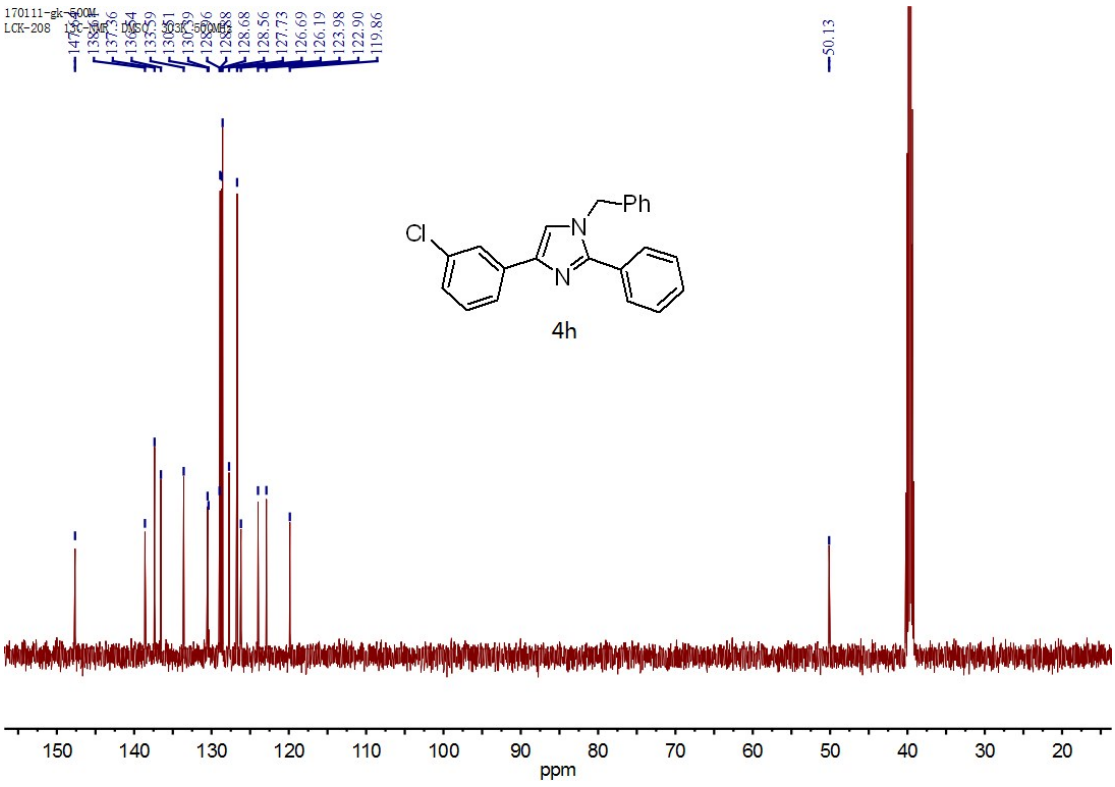

Ph
N
N
F
4f
ppm

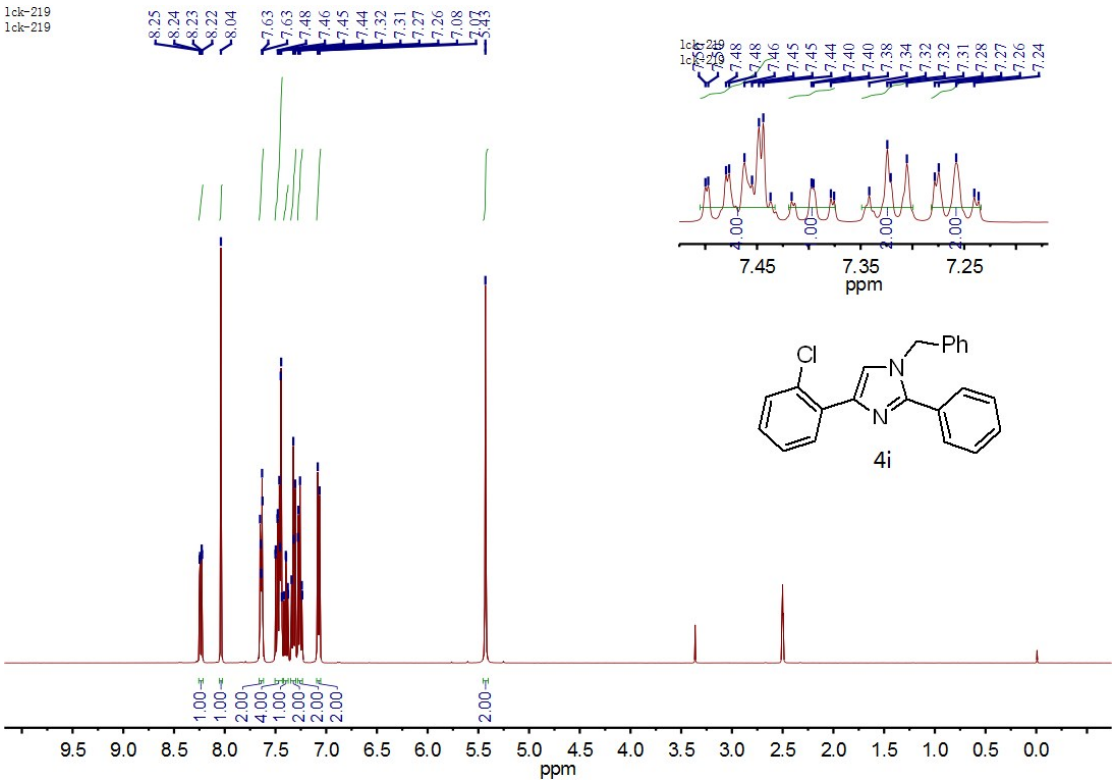

Ph
N
N
F
4f
ppm

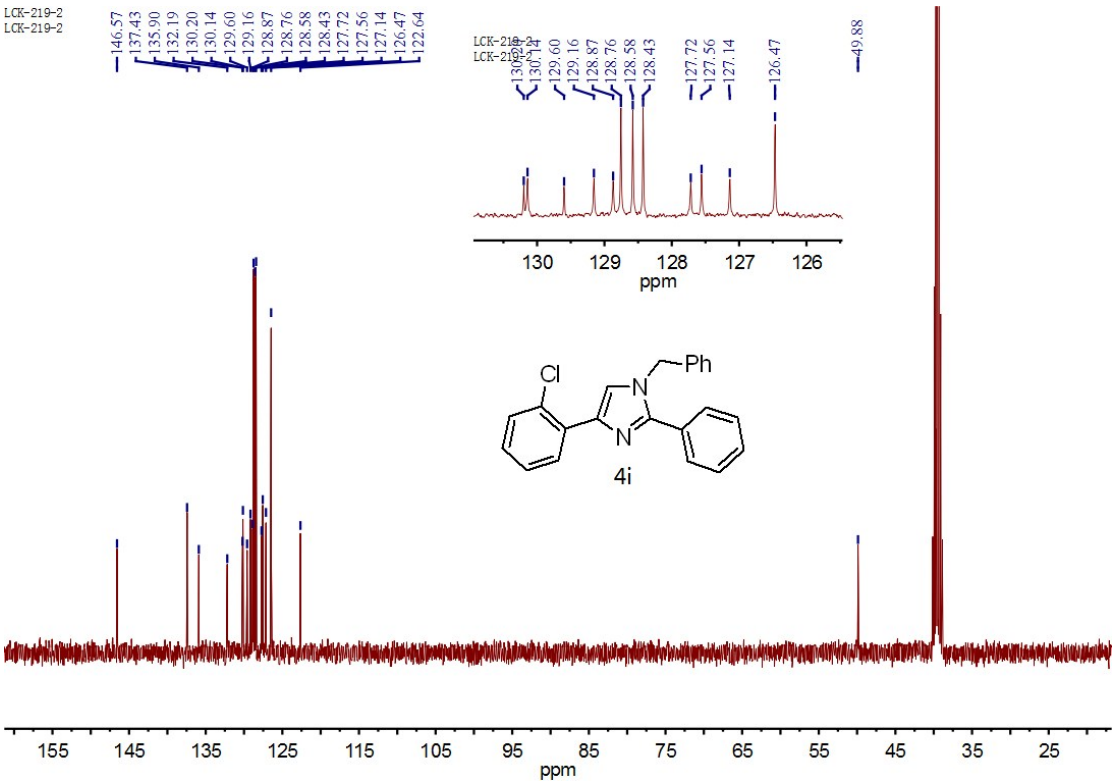

170111-gk-500M
LCK-212 1H-NMR DMSO 303K 500MHz
5.34
1.00
2.01
2.00
5.00
2.00
1.00
2.00
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm
7.9 7.7 7.5 7.3 7.1
ppm
Ph
N
N
Cl
4g

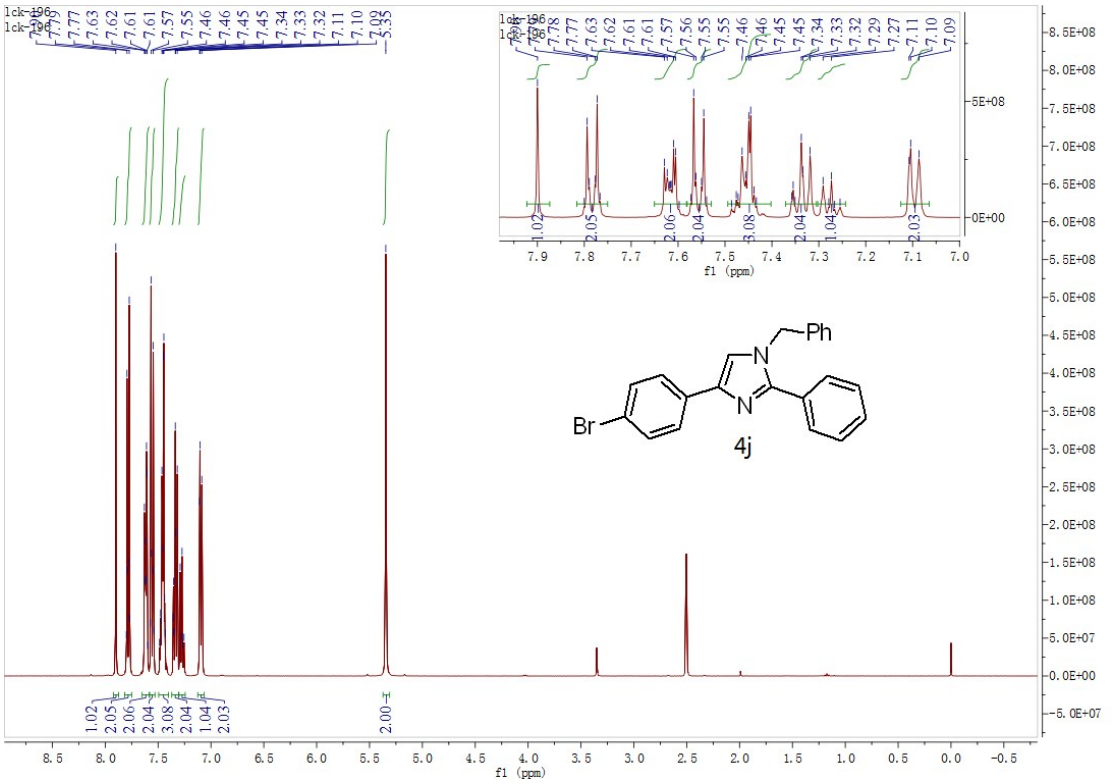

170111-gk-500M
LCK-212 13C-NMR DMSO 303K 500MHz
147.87
139.24
137.26
128.71
128.50
128.44
128.35
127.53
126.49
125.91
119.14
49.90
130.5 129.0 127.5
ppm
Ph
N
N
Cl
4g
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
ppm

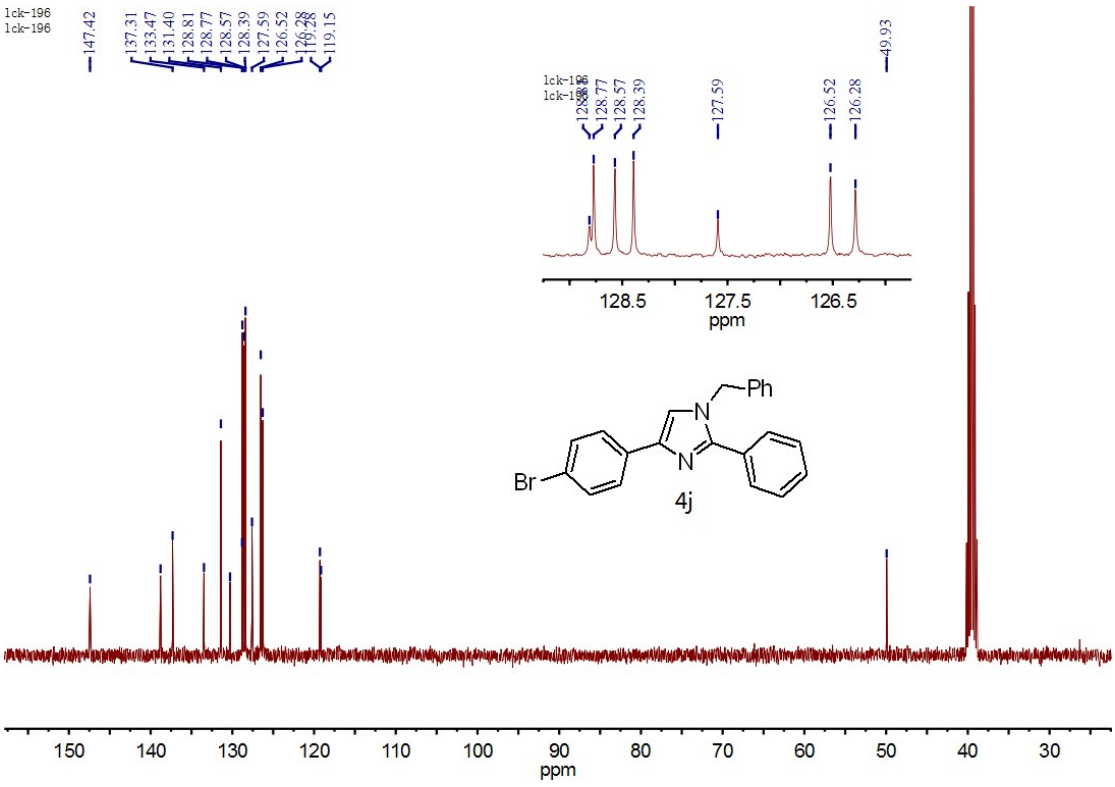

Ph
N
Cl
N
4h
ppm

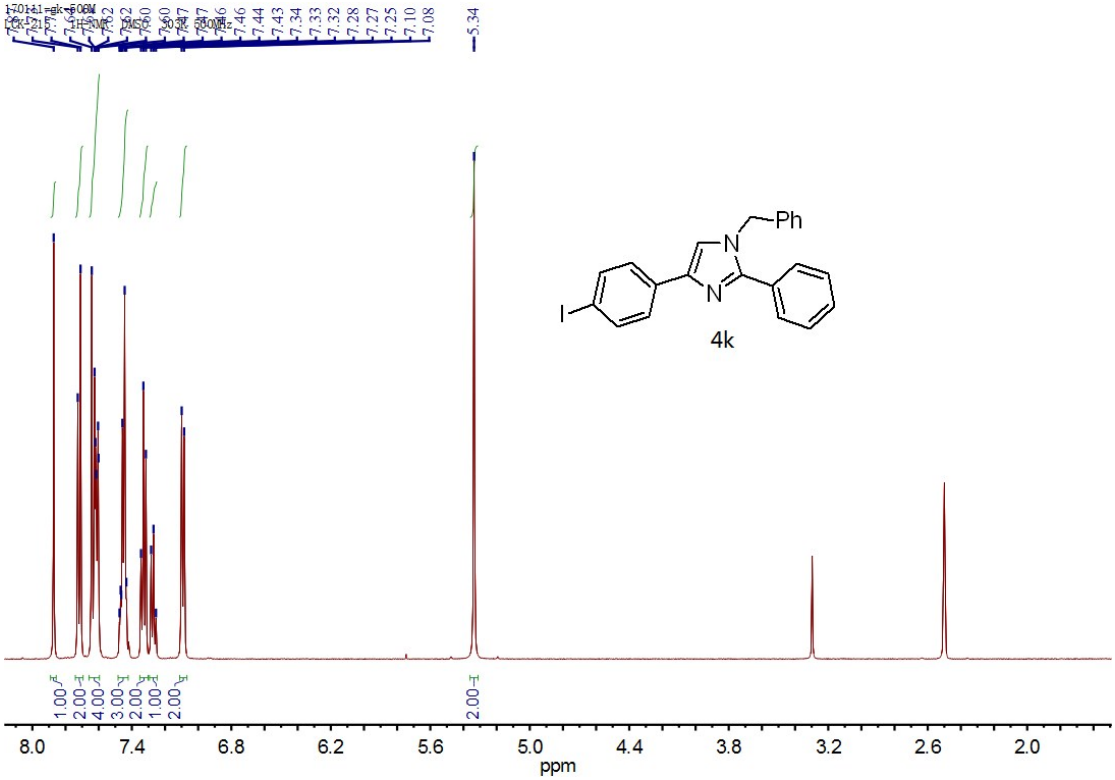

Ph
N
Cl
N
4h
ppm

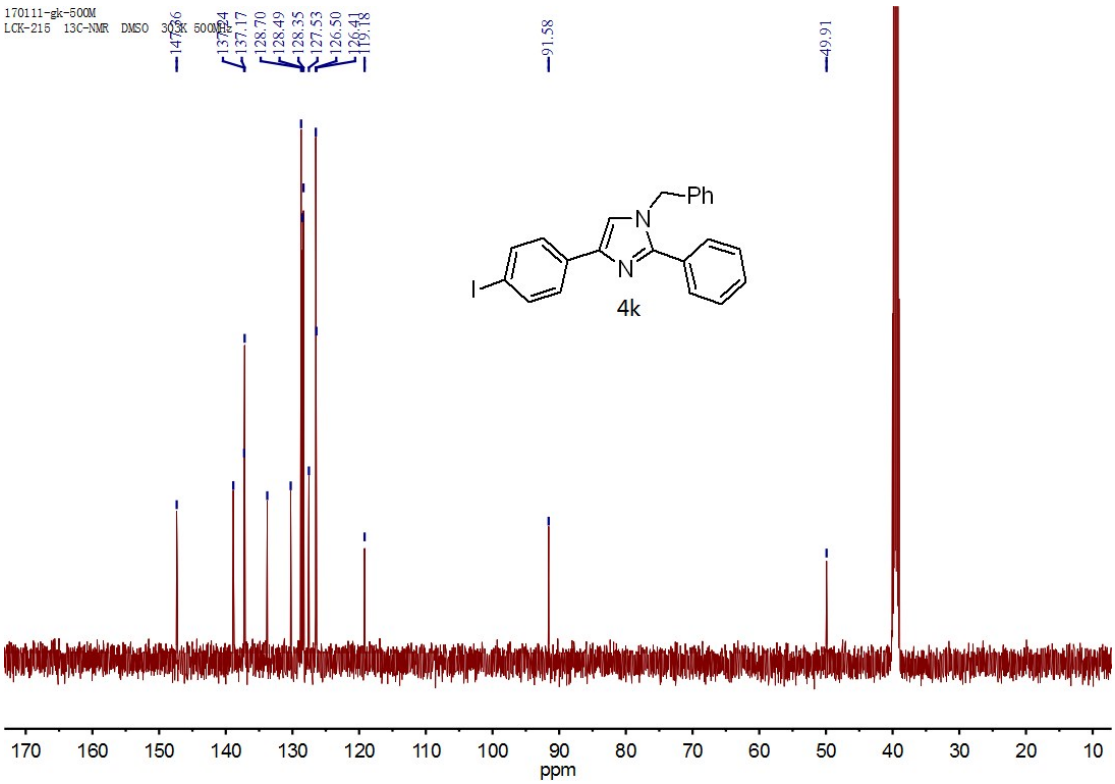

1ck-219
1ck-219
8.25
8.24
8.23
8.22
8.04
7.63
7.63
7.48
7.46
7.45
7.44
7.32
7.31
7.27
7.26
7.08
5.43
7.48
7.46
7.45
7.45
7.44
7.40
7.40
7.38
7.34
7.32
7.32
7.31
7.28
7.27
7.26
7.24
4.00
2.00
2.00
2.00
7.45
7.35
7.25
ppm
Cl
N
Ph
N
4i
1.00
1.00
2.00
4.00
1.00
2.00
2.00
2.00
2.00
9.5
9.0
8.5
8.0
7.5
7.0
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
ppm

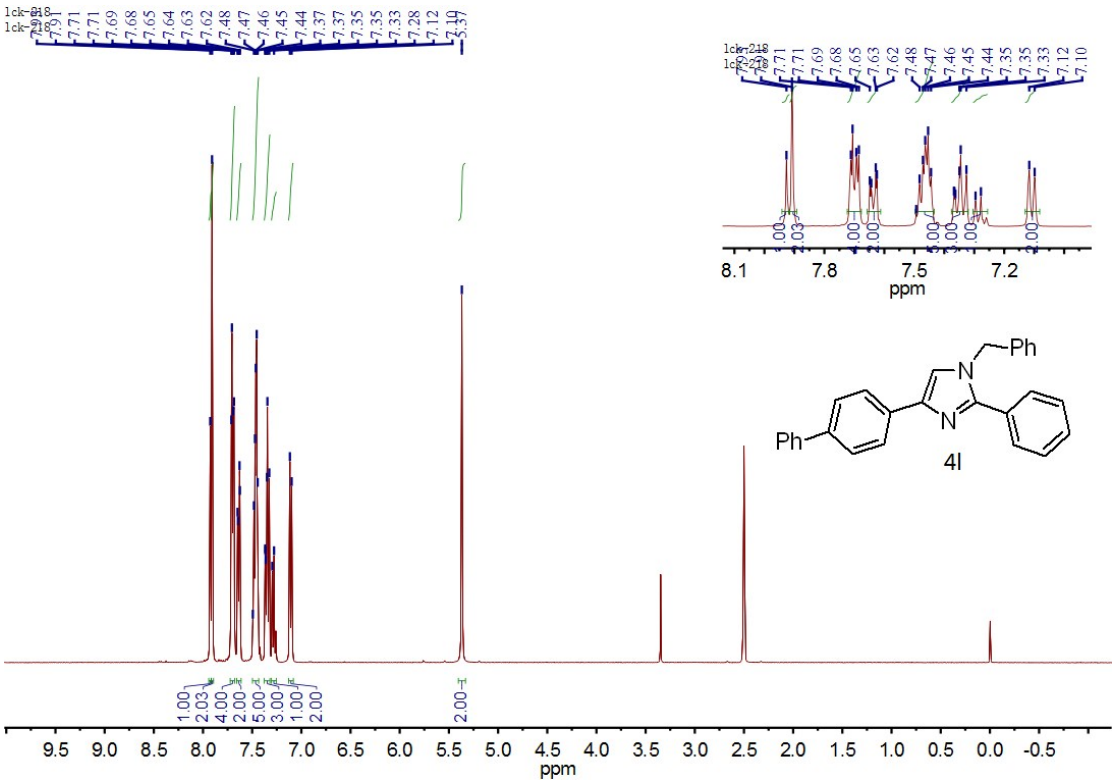

LCK-219-2
LCK-219-2
146.57
137.43
135.90
132.19
130.20
130.14
129.60
129.16
128.87
128.76
128.58
128.43
127.72
127.56
127.14
126.47
122.64
LCK-219-2
LCK-219-2
130.20
130.14
129.60
129.16
128.87
128.76
128.58
128.43
127.72
127.56
127.14
126.47
49.88
130
129
128
127
126
ppm
Cl
N
Ph
N
4i
155
145
135
125
115
105
95
85
75
65
55
45
35
25
ppm

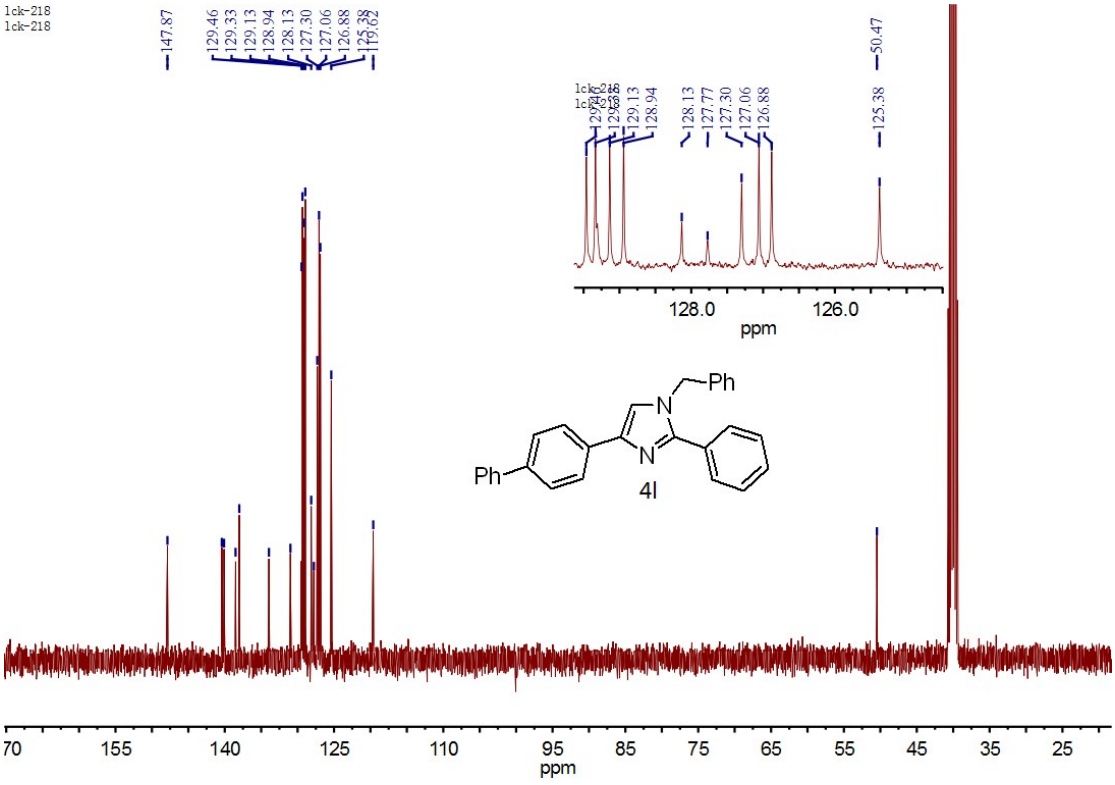

Ph
N
N
Br
4j
f1 (ppm)

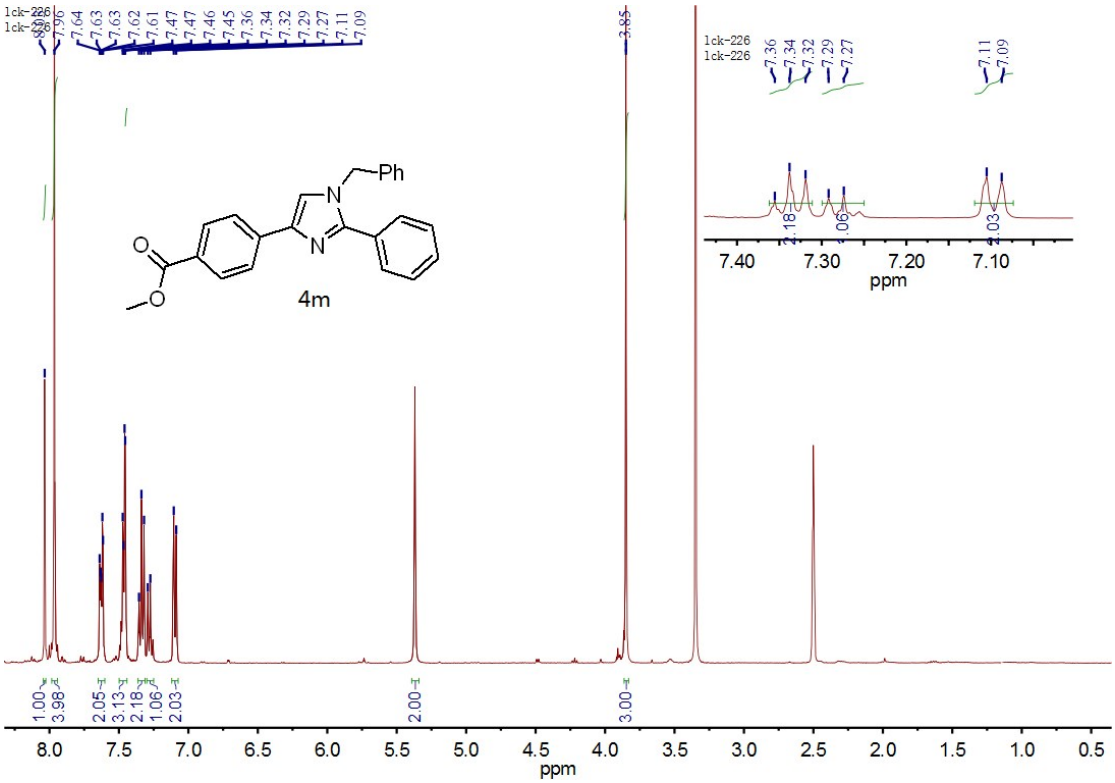

Ph
N
N
Br
4j
ppm

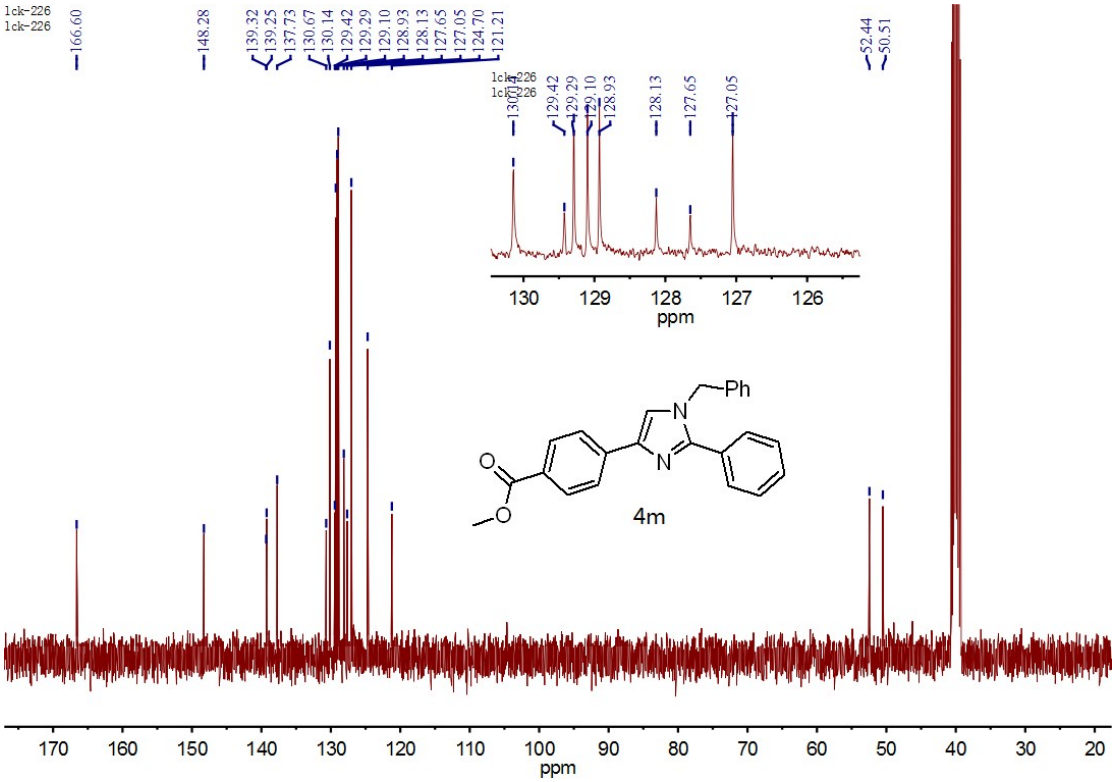

Ph
N
N
I
4k
ppm

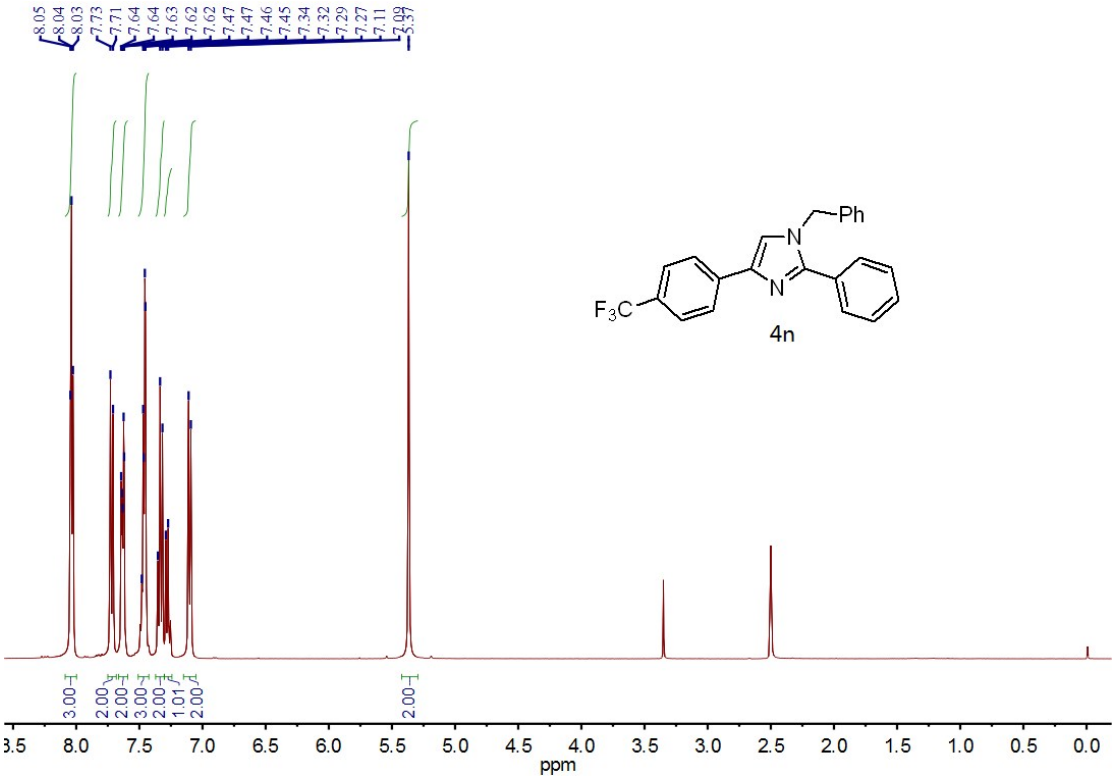

Ph
N
N
I
4k
ppm

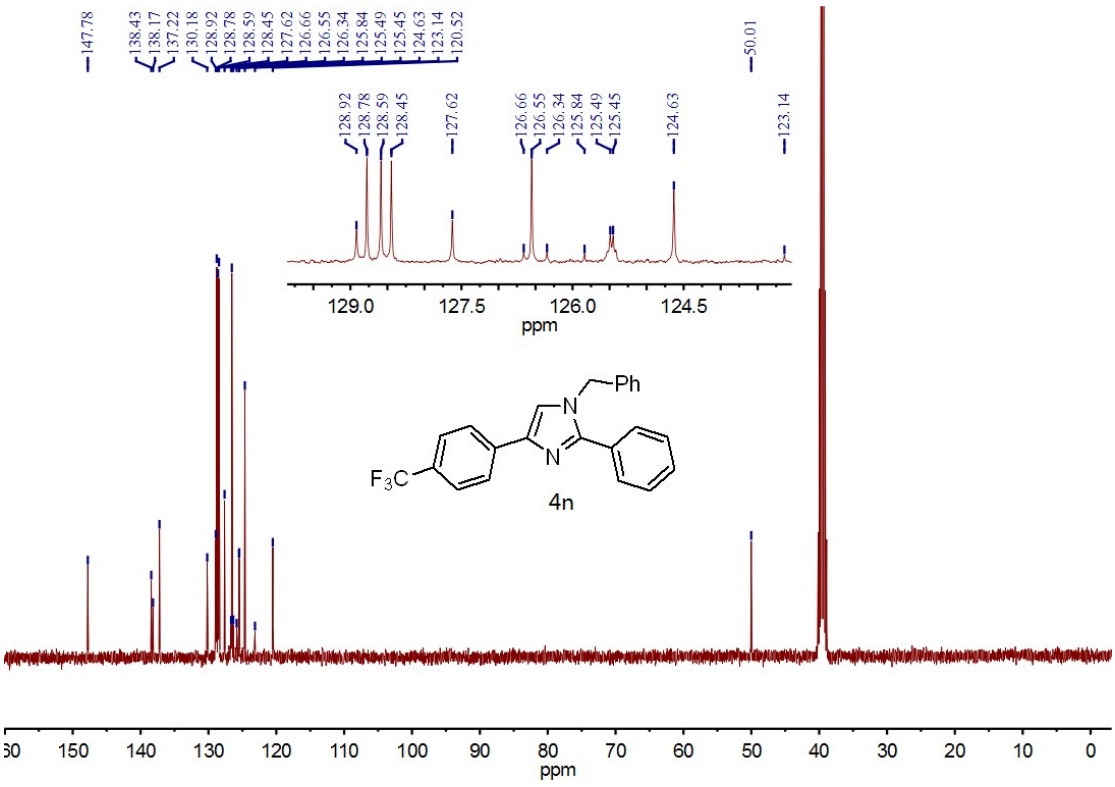

1ck-218
1ck-218
7.91
7.71
7.71
7.69
7.68
7.65
7.64
7.63
7.62
7.48
7.47
7.46
7.45
7.44
7.37
7.37
7.35
7.35
7.33
7.28
7.12
7.10
5.37
1.00
2.03
4.00
2.00
5.00
3.00
1.00
2.00
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
ppm
8.1 7.8 7.5 7.2
ppm
Ph
N
N
Ph
4l

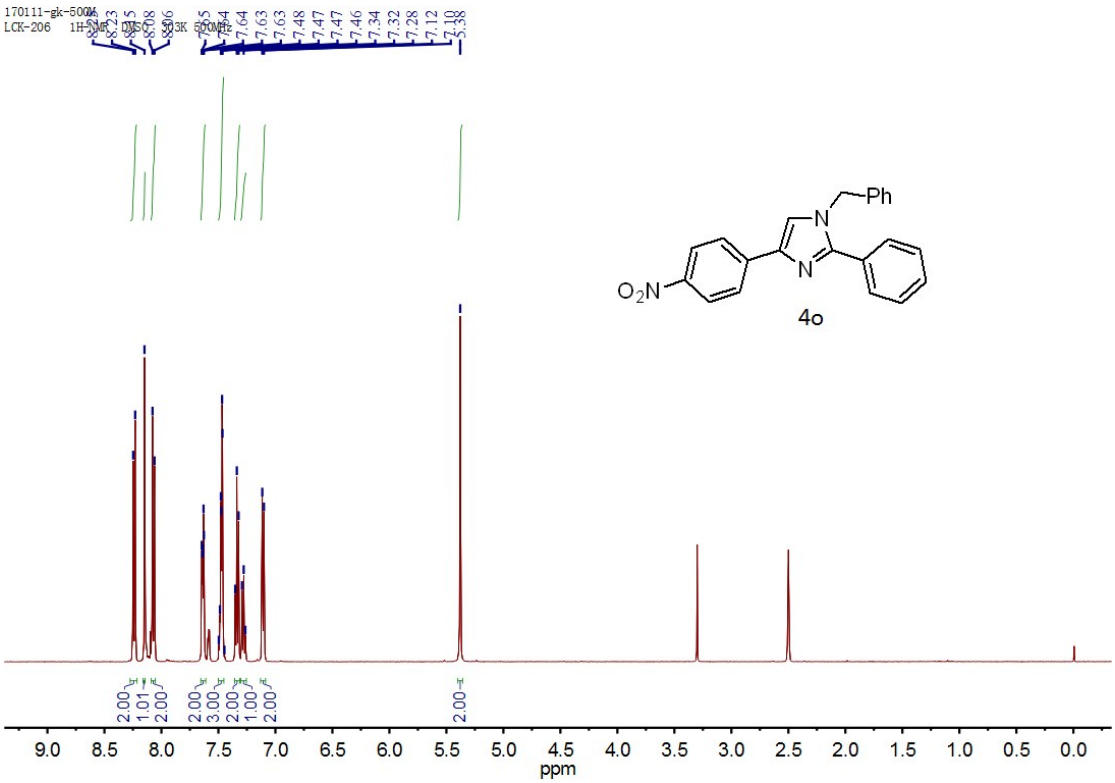

1ck-218
1ck-218
147.87
129.46
129.33
129.13
128.94
128.13
127.30
127.06
126.88
125.38
50.47
70 155 140 125 110 95 85 75 65 55 45 35 25
ppm
128.0 126.0
ppm
Ph
N
N
Ph
4l

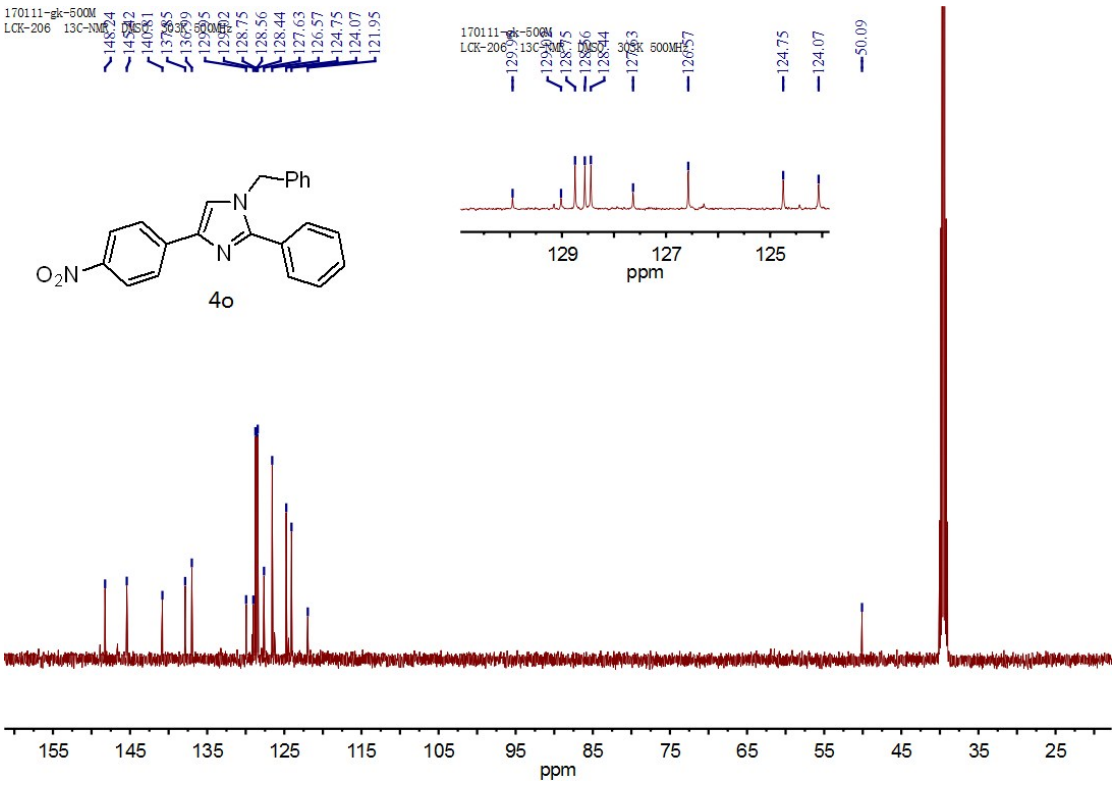

1ck-226
1ck-226
8.01
7.96
7.64
7.63
7.63
7.62
7.61
7.47
7.47
7.46
7.45
7.36
7.34
7.32
7.29
7.27
7.11
7.09
3.85
Ph
N
N
O
O
4m
7.36
7.34
7.32
7.29
7.27
7.11
7.09
2.18
1.06
2.03
7.40
7.30
7.20
7.10
ppm
1.00
3.98
2.05
3.13
2.18
1.06
2.03
2.00
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

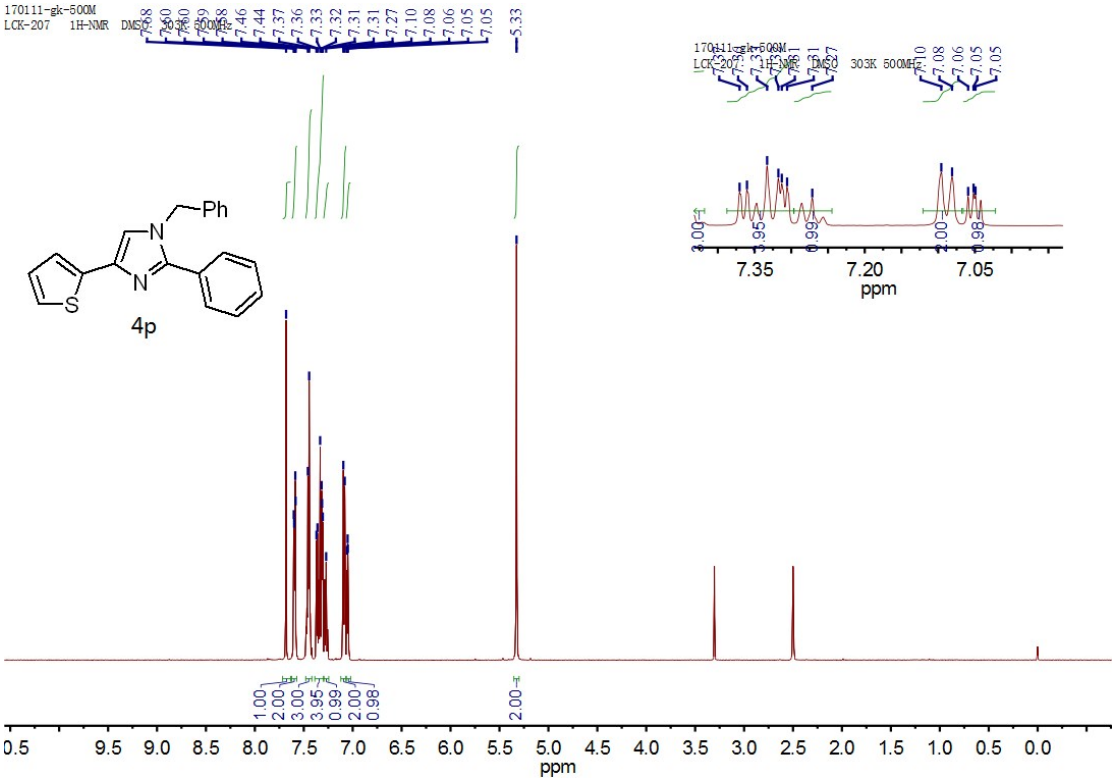

1ck-226
1ck-226
166.60
148.28
139.32
139.25
137.73
130.67
130.14
129.42
129.29
129.10
128.93
128.13
127.65
127.05
124.70
121.21
52.44
50.51
130.67
129.42
129.29
129.10
128.93
128.13
127.65
127.05
130
129
128
127
126
ppm
Ph
N
N
O
O
4m
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

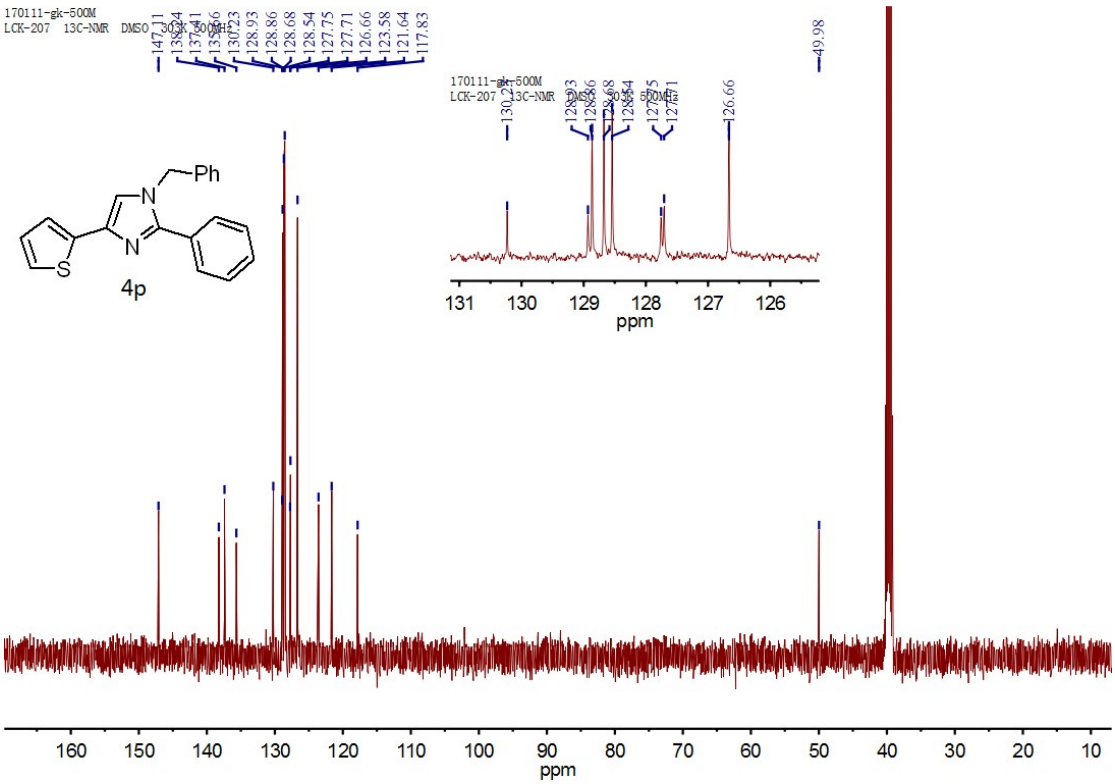

8.05
8.04
8.03
7.73
7.71
7.64
7.64
7.63
7.62
7.62
7.47
7.47
7.46
7.45
7.34
7.32
7.29
7.27
7.11
5.37
3.00
2.00
2.00
3.00
2.00
1.01
2.00
2.00
Ph
N
N
F3C
4n
ppm

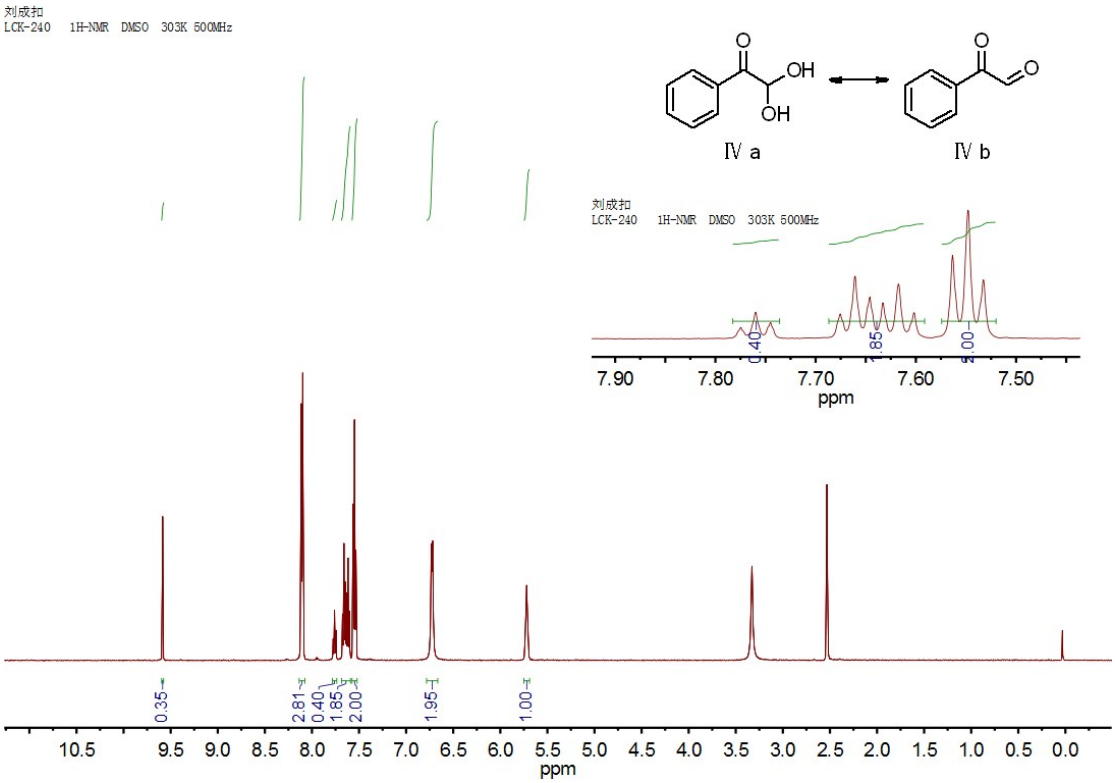

147.78
138.43
138.17
137.22
130.18
128.92
128.78
128.59
128.45
127.62
126.66
126.55
126.34
125.84
125.49
125.45
124.63
123.14
120.52
50.01
129.0
127.5
126.0
124.5
ppm
Ph
N
N
F3C
4n
ppm


Ph
N
N
O_2N
4o
ppm


Ph
N
N
O_2N
4o
129
127
125
ppm
155
145
135
125
115
105
95
85
75
65
55
45
35
25
ppm


170111-gk-500M
LCK-207 1H-NMR DMSO 303K 500MHz
Ph
N
N
S
4p
ppm


170111-gk-500M
LCK-207 13C-NMR DMSO 303K 500MHz
Ph
N
N
S
4p
ppm


刘成扣
LCK-240 1H-NMR DMSO 303K 500MHz
OH
OH
O
O
Ⅳ a
Ⅳ b
刘成扣
LCK-240 1H-NMR DMSO 303K 500MHz
0.40
1.85
2.00
7.90
7.80
7.70
7.60
7.50
ppm
0.35
2.81
0.40
1.85
2.00
1.95
1.00
10.5
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
ppm