

Supporting Information for

Transition-Metal-Free Electrochemical Oxidative C(sp₂)-H

Trifluoromethylation of Hydrazones

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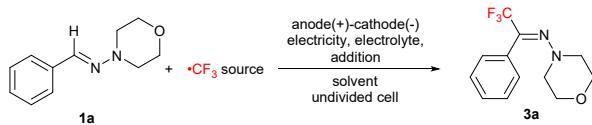
Table for Contents

General information	1
Experimental Section.....	3
Characterization of the products	4
Copies of NMR spectra	14

General information

All reactions were carried out in dried sealed Schlenk tubes with magnetic stirring. All anhydrous and oxygen-free environments were performed under argon atmosphere in oven-dried glassware using Schlenk techniques. All the chemicals were obtained commercially and used without any prior purification. All products were isolated by short chromatography on a silica gel (200-300 mesh) column using hexane and ethyl acetate. ¹H, ¹³C and ¹⁹F NMR spectra were recorded on a Bruker Advance 400 spectrometer at ambient temperature with CDCl₃ as solvent and tetramethylsilane (TMS) as the internal standard. Analytical thin layer chromatography (TLC) was performed on Merck precoated TLC (silica gel 60 F254) plates.

Optimized reaction conditions:



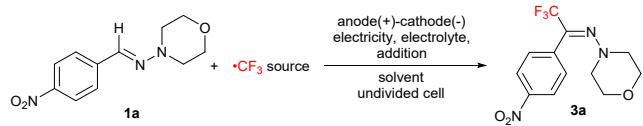
Entry	electrode	electricity	Solvent	•CF ₃ source	electrolyte	addition	atmosphere	Temperature(°C)	Yield(%) ^b
1	C(+)-Pt(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	32
2	C(+)-C(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	45
3	C(+)-Ni(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	32
4 ^[c]	C(+)-Ni(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	37
5	Pt(+)- Pt(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	37
6	RVC(+)-Pt(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	40
7	RVC(+)-Ni(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	37
8	C(+)- Cu(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	20
9	C(+)-Fe(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	40
10	RVC(+)- RVC(-)	8mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	air	50	42
)
11 ^[d]	C(+)-C(-)	3mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	34
12 ^[d]	C(+)-C(-)	5mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	47
13	C(+)-C(-)	12mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	36
14	C(+)-C(-)	0mA	MeCN	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	N.D.
15	C(+)-C(-)	8mA	HFIP	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	35
16	C(+)-C(-)	8mA	DMF	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	Trace
17	C(+)-C(-)	8mA	MeCN: H ₂ O	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	33
			= 1:1						
18	C(+)-C(-)	8mA	MeCN: H ₂ O	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	35
			= 3:1						
19	C(+)-C(-)	8mA	MeCN:	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	trace
			MeOH= 1:1						
20	C(+)-C(-)	8mA	acetone	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	trace
21	C(+)-C(-)	8mA	DCE	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	35
22	C(+)-C(-)	8mA	HFIP: DCE	CF ₃ SO ₂ Na	"Bu ₄ NBF ₄	none	Air	50	27
			= 1:1						

23	C(+)·C(-)	8mA	TFA: H ₂ O =10:1	CF ₃ SO ₂ Na	ⁿ Bu ₄ NBF ₄	none	Air	50	15
24 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	none	Air	50	71
25 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NPF ₆	none	Air	50	65
26	C(+)·C(-)	8mA	MeCN	Togni reagent	LiClO ₄	none	Air	50	22
27	C(+)·C(-)	8mA	MeCN	Togni reagent	NH ₄ BF ₄	none	Air	50	42
28 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	Et ₃ NBr	none	Air	50	60
29	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaHCO ₃	Air	50	72
30	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOH	Air	50	42
31 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	Air	50	80
32	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	KOH	Air	50	30
33	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	HOAc	Air	50	34
34 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	85
35 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	r.t.	64
36 ^[c]	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	70°C	82

^[a]Reaction conditions : 1 (0.3 mmol) , 3 (1.5 eq.), electrolyte (0.3 eq.) , solvent (4 mL) ,constant current electricity, stirred, 2 h.

^[b]Isolated yield. ^[c]Nickel foam. ^[d]4 h. ^[e]1 h.

Optimized reaction conditions: substrates with para-electron-withdrawing groups



Entry	electrode	electricity	Solvent	•CF ₃ source	electrolyte	addition	atmosphere	Temperature(°C)	Yield(%) ^[c]
1	C(+)·C(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	48
2	C(+)·Pt(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	42
3	C(+)·Ni(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	40
4 ^[c]	C(+)·Ni(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	42
5	RVC(+)- Pt(-)	8mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	42
6	C(+)·C(-)	10mA	MeCN	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	48
7	C(+)·C(-)	8mA	Acetone	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	trace
8	C(+)·C(-)	8mA	Toluene	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	N.R.
9	C(+)·C(-)	8mA	DMF	Togni reagent	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	30
^{[b]10}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	ⁿ Bu ₄ NBF ₄	NaOAc	N ₂	50	87
^{[b]11}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	KF	NaOAc	N ₂	50	25
^{[b]12}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	ⁿ Bu ₄ NPF ₆	NaOAc	N ₂	50	84
^{[b]13}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	LiClO ₄	NaOAc	N ₂	50	42
^{[b]14}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	ⁿ Bu ₄ NBF ₄	None	N ₂	50	65
^{[b]15}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	ⁿ Bu ₄ NBF ₄	NaOH	N ₂	50	66
^{[b]16}	C(+)·C(-)	8mA	MeCN	CF ₃ SO ₂ Na	ⁿ Bu ₄ NBF ₄	NaHCO ₃	N ₂	50	75

^[a]Reaction conditions : 1 (0.3 mmol) , 3 (1.5 eq.), electrolyte (0.3 eq.) , solvent (4 mL) ,constant current electricity, stirred, 1 h. ^{[b]2h}

^[c]Isolated yield.

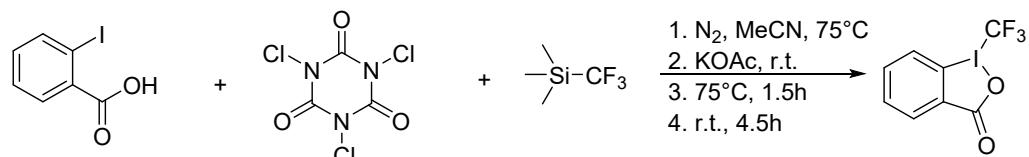
Experimental Section

General procedure for the synthesis of compounds 1



A mixture of hydrazine (2.4 mmol), aldehyde (2.0 mmol) and anhydrous MgSO_4 (2.0 mmol) in CH_2Cl_2 (10 mL) was stirred at room temperature in 6h. After filtration of MgSO_4 , CH_2Cl_2 was removed under reduced pressure and the mixture was subjected to recrystallize to give the desired product 1 with almost quantitative yields.

General procedure for the synthesis of Togni reagent (2b)



Add ortho-iodobenzoic acid (3.47 g, 14 mmol, 1.0 eq) into a 100mL three-neck flask in nitrogen atmosphere, add 30mL acetonitrile, heat to 75°C, then add trichloroisocyanuric acid (0.974 g, 4.74 mmol, 0.3 eq) in 5 minutes with a constant pressure drop funnel. After cool to room temperature, add dried potassium acetate (2.75 g, 28 mmol, 2.0 eq) at once. The reaction system was cooled to room temperature after 1.5 hours of reaction at 75°C. And then, TMSCF_3 (2.90 mL, 19.6 mmol, 1.4 eq) was added at once for 4.5h in room temperature. After concentration, the product crystallizes and is washed with cold acetonitrile to obtain white solid Togni reagent.

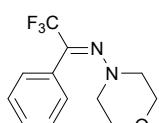
Photo



Cyclic voltammetry studies

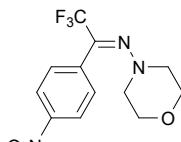
The cyclic voltammograms experiments were conducted in a Schlenk tube that contained the substance dissolved in a 0.1 M solution of tetrabutylammonium hexafluorophosphate in acetonitrile. A glassy carbon electrode working electrode, a platinum wire counter electrode and an Ag/AgCl reference electrode were used. The reference electrode was stored in saturated potassium chloride solution for activation before use. The relevant parameters were controlled by an electrochemical workstation CHI600E.

Characterization of the products



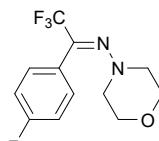
(E)-2,2,2-trifluoro-N-morpholino-1-phenylethan-1-imine (3a)

Colorless solid; m.p.= 40.1-41.0°C. ^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.28 (m, 5H), 3.88 – 3.37 (m, 4H), 3.20 – 2.55 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.35 (q, $^2J_{\text{C},\text{F}} = 33.84\text{Hz}$), 131.73, 129.95, 128.95, 128.60, 121.37 (q, $^1J_{\text{C},\text{F}} = 275.73\text{Hz}$), 66.22, 54.37. ^{19}F NMR (376 MHz, CDCl_3) δ -66.75. Spectroscopic data are in accordance with those described in the literature.^[1]



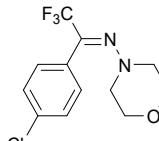
(E)-2,2,2-trifluoro-N-morpholino-1-(4-nitrophenyl)ethan-1-imine (3b)

Yellow solid; m.p.= 161.8-163.3 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.55 – 7.91 (m, 2H), 7.76 – 7.45 (m, 2H), 4.02 – 3.42 (m, 4H), 3.18 – 2.70 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 148.47, 138.28, 132.54 (q, $^2J_{\text{C},\text{F}} = 34.34\text{ Hz}$), 129.97, 121.04(q, $^1J_{\text{C},\text{F}} = 275.73\text{ Hz}$), 124.16, 66.07, 54.53. ^{19}F NMR (376 MHz, CDCl_3) δ -79.87. Spectroscopic data are in accordance with those described in the literature.^[2]



(E)-2,2,2-trifluoro-1-(4-fluorophenyl)-N-morpholinoethan-1-imine (3c)

Yellow solid; m.p.= 119.3- 121.4 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.51 – 7.36 (m, 2H), 7.17 – 7.05 (m, 2H), 3.71 – 3.55 (m, 4H), 3.08 – 2.86 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 163.39 (d, $^1J_{\text{C},\text{F}} = 252.53\text{Hz}$), 135.76 (q, $^2J_{\text{C},\text{F}} = 33.33\text{Hz}$), 130.73 (d, $^3J_{\text{C},\text{F}} = 9.09\text{Hz}$), 127.57 (d, $^4J_{\text{C},\text{F}} = 4.04\text{Hz}$), 121.25(q, $^1J_{\text{C},\text{F}} = 275.73\text{Hz}$), 116.27 (d, $^2J_{\text{C},\text{F}} = 22.22\text{Hz}$), 66.19, 54.36. ^{19}F NMR (376 MHz, CDCl_3) δ -66.86, -109.46. Spectroscopic data are in accordance with those described in the literature.^[3]



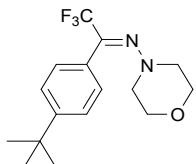
(E)-1-(4-chlorophenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3d)

Yellow solid; m.p.= 45.5- 47.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.49 – 7.32 (m, 4H), 3.72 – 3.51 (m, 4H), 3.14 – 2.86 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.17, 135.02 (q, $^2J_{\text{C},\text{F}} = 35.35\text{Hz}$), 130.03, 129.35, 121.21(q, $^1J_{\text{C},\text{F}} = 275.73\text{Hz}$), 66.17, 54.39. ^{19}F NMR (376 MHz, CDCl_3) δ -66.23. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{12}\text{H}_{12}\text{ClF}_3\text{N}_2\text{O}$ (293.0663), found 293.0668.



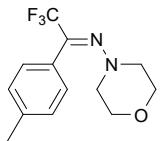
(E)-1-(4-bromophenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3e)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, $J = 8.0\text{ Hz}$, 2H), 7.56 (d, $J = 8.0\text{ Hz}$, 2H), 3.63 (t, 4H), 3.00 (t, $J = 4.9\text{ Hz}$, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 134.92 (q, $^2J_{\text{C},\text{F}} = 33.33\text{Hz}$), 132.31, 130.53, 130.25, 124.43, 121.16 (q, $^1J_{\text{C},\text{F}} = 275.73\text{Hz}$), 66.15, 54.39. ^{19}F NMR (376 MHz, CDCl_3) δ -66.65. Spectroscopic data are in accordance with those described in the literature.^[1]



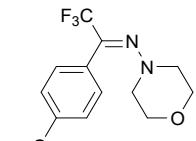
(E)-1-(4-(tert-butyl)phenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3f)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.46 – 7.38 (m, 2H), 7.39 – 7.31 (m, 2H), 3.88 – 3.39 (m, 4H), 3.33 – 2.76 (m, 4H), 1.33 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 153.31, 136.99(q, $^2J_{\text{C},\text{F}} = 32.32\text{Hz}$) 128.51, 128.24, 125.82, 120.06(q, $^1J_{\text{C},\text{F}} = 275.73\text{Hz}$), 66.25, 54.36, 34.98, 31.32. ^{19}F NMR (376 MHz, CDCl_3) δ -66.71. Spectroscopic data are in accordance with those described in the literature.^[2]



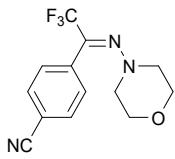
(E)-2,2,2-trifluoro-N-morpholino-1-(p-tolyl)ethan-1-imine (3g)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.32 (d, $J = 8.0\text{ Hz}$, 2H), 7.22 (d, $J = 8.0\text{ Hz}$, 2H), 3.71 – 3.54 (m, 4H), 3.08 – 2.92 (m, 4H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.16, 136.93 (q, $^2J_{\text{C},\text{F}} = 33.33\text{Hz}$), 129.64, 128.64, 128.43, 121.40 (q, $^1J_{\text{C},\text{F}} = 275.73\text{ Hz}$), 66.26, 54.33, 21.57. ^{19}F NMR (376 MHz, CDCl_3) δ -66.81. Spectroscopic data are in accordance with those described in the literature.^[4]



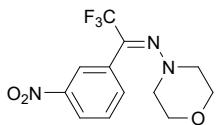
(E)-2,2,2-trifluoro-1-(4-methoxyphenyl)-N-morpholinoethan-1-imine (3h)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.39 (d, $J = 8.7\text{ Hz}$, 2H), 6.98 – 6.84 (m, 2H), 3.84 (s, 3H), 3.71 – 3.51 (m, 4H), 3.21 – 2.77 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 160.69, 137.31(q, $^2J_{\text{C},\text{F}} = 33.33\text{Hz}$), 129.98, 123.46, 121.39(q, $^1J_{\text{C},\text{F}} = 276.74\text{ Hz}$), 114.38, 66.28, 55.41, 54.30. ^{19}F NMR (376 MHz, CDCl_3) δ -66.81. Spectroscopic data are in accordance with those described in the literature. ^[1]



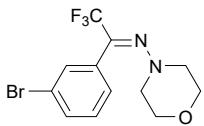
(E)-4-(2,2,2-trifluoro-1-(morpholinoimino)ethyl)benzonitrile (3i)

Yellow solid; m.p.= 149.4- 150.8 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.85 – 7.65 (m, 2H), 7.64 – 7.47 (m, 2H), 3.78 – 3.45 (m, 4H), 3.21 – 2.72 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.46, 133.09 (q, $^2J_{\text{C},\text{F}}= 33.33\text{Hz}$), 132.69, 129.61, 121.09 (q, $^1J_{\text{C},\text{F}}= 275.73\text{Hz}$), 117.96, 114.02, 66.07, 54.55. ^{19}F NMR (376 MHz, CDCl_3) δ -66.22. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{13}\text{H}_{12}\text{F}_3\text{N}_3\text{O}$ (283.0934), found 283.1005.



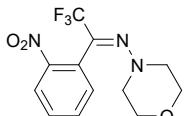
(E)-1-(3-bromophenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3j)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.17 (dd, $J= 8.1, 1.4\text{ Hz}$, 1H), 7.73 (td, $J= 7.5, 1.4\text{ Hz}$, 1H), 7.65 (td, $J= 7.8, 1.6\text{ Hz}$, 1H), 7.49 (dd, $J= 7.5, 1.6\text{ Hz}$, 1H), 3.76 – 3.42 (m, 4H), 3.18 – 2.87 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 147.87, 134.04, 131.53(q, $^2J_{\text{C},\text{F}}= 33.3\text{Hz}$), 131.38, 131.29, 127.28, 125.20, 120.89(q, $^1J_{\text{C},\text{F}}= 275.73\text{Hz}$), 66.24, 53.86. ^{19}F NMR (376 MHz, CDCl_3) δ -66.48. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{12}\text{H}_{12}\text{F}_3\text{N}_3\text{O}_3$ (303.0836), found 303.0909.



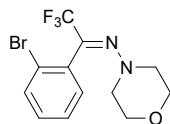
(E)-1-(3-bromophenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3k)

White solid; m.p.= 49.0- 50.2°C. ^1H NMR (400 MHz, CDCl_3) δ 7.63 – 7.50 (m, 2H), 7.46 – 7.27 (m, 2H), 3.83 – 3.45 (m, 4H), 3.19 – 2.83 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 133.89 (q, $^2J_{\text{C},\text{F}}= 33.3\text{Hz}$), 133.68, 133.13, 131.56, 130.49, 127.35, 121.21 (q, $^1J_{\text{C},\text{F}}= 275.73\text{Hz}$), 123.05, 66.17, 54.45. ^{19}F NMR (376 MHz, CDCl_3) δ -66.54. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{12}\text{H}_{12}\text{BrF}_3\text{N}_2\text{O}$ (336.0088), found 336.0061.



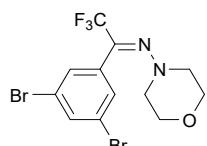
(E)-2,2,2-trifluoro-N-morpholino-1-(2-nitrophenyl)ethan-1-imine (3l)

Yellow solid; m.p.= 99.6- 101.3°C. ^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, $J= 8.1\text{ Hz}$, 2H), 7.57 (d, $J= 8.1\text{ Hz}$, 2H), 3.83 – 3.48 (m, 4H), 3.16 – 2.79 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 147.94, 134.01, 131.58 (q, $^1J_{\text{C},\text{F}}= 35.35\text{ Hz}$), 131.40, 131.28, 127.32, 125.20, 120.90 (q, $^1J_{\text{C},\text{F}}= 275.73\text{ Hz}$), 66.25, 53.89. ^{19}F NMR (376 MHz, CDCl_3) δ -66.48. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{12}\text{H}_{12}\text{F}_3\text{N}_3\text{O}_3$ (303.0836), found 304.0860.



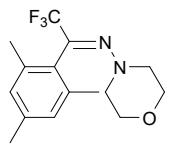
(E)-1-(2-bromophenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3m)

White solid; m.p.= 76.5- 77.2°C. ^1H NMR (400 MHz, CDCl_3) δ 7.63 (dd, 1H), 7.42 – 7.27 (m, 3H), 3.79 – 3.48 (m, 4H), 3.22 – 2.95 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 134.09, 133.06, 131.32, 131.29, 129.20 (q, $^2J_{\text{C},\text{F}}= 35.35\text{Hz}$), 127.57, 124.36, 121.50(q, $^1J_{\text{C},\text{F}}= 276.74\text{Hz}$), 66.57, 53.66. ^{19}F NMR (376 MHz, CDCl_3) δ -66.03. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{12}\text{H}_{12}\text{BrF}_3\text{N}_2\text{O}$ (336.0088), found 336.0108.



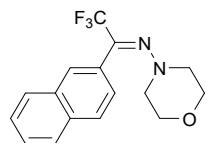
(E)-1-(3,5-dibromophenyl)-2,2,2-trifluoro-N-morpholinoethan-1-imine (3n)

White solid; m.p.= 143.0- 143.7°C. ^1H NMR (600 MHz, CDCl_3) δ 7.73 (t, $J = 1.8\text{ Hz}$, 1H), 7.52 (d, $J = 1.8\text{ Hz}$, 2H), 3.73 – 3.61 (m, 4H), 3.11 – 2.99 (m, 4H). ^{13}C NMR (150 MHz, CDCl_3) δ 135.68, 134.98, 131.46 (q, $^2J_{\text{C},\text{F}}= 34.73\text{Hz}$), 130.35, 121.06 (q, $^1J_{\text{C},\text{F}}= 274.82\text{Hz}$), 123.58, 66.09, 54.47. ^{19}F NMR (565 MHz, CDCl_3) δ -66.27. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{12}\text{H}_{11}\text{Br}_2\text{F}_3\text{N}_2\text{O}$ (414.9263), found 416.9268.



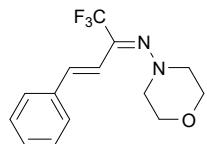
(E)-2,2,2-trifluoro-1-mesityl-N-morpholinoethan-1-imine (3o)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 6.94 – 6.82 (m, 2H), 3.73 – 3.44 (m, 4H), 3.27 – 2.86 (m, 4H), 2.39 (s, 1H), 2.29 (s, 3H), 2.23 (s, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 139.62, 137.36, 133.48 (q, $^2J_{\text{C},\text{F}}= 34.34\text{Hz}$), 129.69, 128.95, 128.64, 121.81(q, $^1J_{\text{C},\text{F}}= 274.82\text{Hz}$), 66.76, 53.37, 21.26. ^{19}F NMR (376 MHz, CDCl_3) δ -65.69. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{15}\text{H}_{19}\text{F}_3\text{N}_2\text{O}$ (301.1522), found 301.1524.



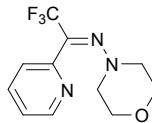
(E)-2,2,2-trifluoro-N-morpholino-1-(naphthalen-2-yl)ethan-1-imine (3p)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.20 – 7.73 (m, 4H), 7.73 – 7.43 (m, 3H), 3.87 – 3.42 (m, 4H), 3.28 – 2.71 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 136.46 (q, $^2J_{\text{C},\text{F}}= 33.33\text{Hz}$), 133.64, 133.00, 129.00, 128.72, 128.60, 128.37, 127.98, 127.63, 127.07, 125.35, 121.51(q, $^1J_{\text{C},\text{F}}= 275.73\text{Hz}$), 66.24, 54.48. ^{19}F NMR (376 MHz, CDCl_3) δ -66.39. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{16}\text{H}_{15}\text{F}_3\text{N}_2\text{O}$ (309.1209), found 308.1215.



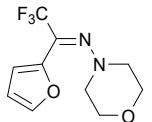
(2E,3E)-1,1,1-trifluoro-N-morpholino-4-phenylbut-3-en-2-imine (3q)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.43 – 7.31 (m, 5H), 7.05 (d, $J = 9.3$ Hz, 1H), 7.02 – 6.96 (m, 1H), 3.82 – 3.72 (m, 4H), 3.09 – 2.99 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 132.17, 131.78 (q, $^3J_{\text{C},\text{F}} = 5.05$ Hz), 131.52, 130.64 (q, $^2J_{\text{C},\text{F}} = 30.30$ Hz), 129.96, 128.94, 128.73, 123.95 (q, $^1J_{\text{C},\text{F}} = 272.7$ Hz), 66.23, 51.11. ^{19}F NMR (376 MHz, CDCl_3) δ -64.96. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{14}\text{H}_{15}\text{F}_3\text{N}_2\text{O}$ (285.1138), found 285.1140.



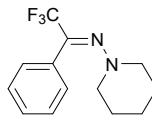
(E)-2,2,2-trifluoro-N-morpholino-1-(pyridin-3-yl)ethan-1-imine (3r)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 8.71 (ddd, $J = 4.9, 1.8, 1.0$ Hz, 1H), 7.76 (td, $J = 7.8, 1.8$ Hz, 1H), 7.49 (d, $J = 7.8$ Hz, 1H), 7.32 (ddd, $J = 7.7, 4.9, 1.2$ Hz, 1H), 3.77 – 3.48 (m, 4H), 3.13 – 2.85 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 151.08, 150.14, 136.80, 132.74 (q, $^2J_{\text{C},\text{F}} = 33.33$ Hz), 124.84, 124.28, 121.51 (q, $^1J_{\text{C},\text{F}} = 275.73$ Hz), 66.16, 54.59. ^{19}F NMR (376 MHz, CDCl_3) δ -65.49. Spectroscopic data are in accordance with those described in the literature.^[4]



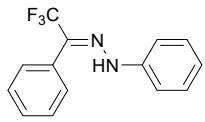
(E)-2,2,2-trifluoro-1-(furan-2-yl)-N-morpholinoethan-1-imine (3s)

White solid; m.p.= 97.3- 98.6 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.40 (s, 1H), 6.79 (d, $J = 3.5$ Hz, 1H), 6.54 (d, $J = 3.5$ Hz, 1H), 3.95 – 3.68 (m, 4H), 3.34 – 2.84 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 154.35, 141.08 (q, $^2J_{\text{C},\text{F}} = 42.42$ Hz), 124.56, 119.25 (q, $^1J_{\text{C},\text{F}} = 267.65$ Hz), 113.37 (q, $^3J_{\text{C},\text{F}} = 3.03$ Hz), 107.28, 66.35, 51.38. ^{19}F NMR (376 MHz, CDCl_3) δ -63.80. Spectroscopic data are in accordance with those described in the literature.^[1]



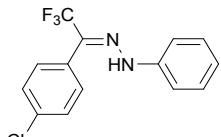
(E)-2,2,2-trifluoro-1-phenyl-N-(piperidin-1-yl)ethan-1-imine (3t)

Yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.59 – 7.29 (m, 5H), 3.18 – 2.80 (m, 4H), 1.60 – 1.38 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 132.63, 132.60 (q, $^2J_{\text{C},\text{F}} = 32.32$ Hz), 132.12, 129.47, 128.74, 128.73, 121.87 (q, $^1J_{\text{C},\text{F}} = 275.73$ Hz), 55.01, 25.04, 24.01. ^{19}F NMR (376 MHz, CDCl_3) δ -66.02. Spectroscopic data are in accordance with those described in the literature.^[3]



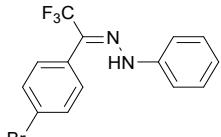
(E)-1-phenyl-2-(2,2,2-trifluoro-1-phenylethylidene)hydrazine (3u)

Brown oil. ^1H NMR (400 MHz, CDCl_3) δ 7.80 (s, 1H), 7.62 – 7.54 (m, 3H), 7.45 – 7.41 (m, 2H), 7.31 – 7.26 (m, 2H), 7.10 – 7.03 (m, 2H), 7.00 – 6.89 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.06, 131.74 (q, $^2J_{\text{C},\text{F}} = 35.35$ Hz), 130.68, 129.99, 129.46, 129.12, 127.23, 122.03, 121.61 (q, $^1J_{\text{C},\text{F}} = 273.71$ Hz), 113.74. ^{19}F NMR (376 MHz, CDCl_3) δ -66.35. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{15}\text{H}_{12}\text{F}_3\text{N}_2$ (265.0947), found 265.0950.



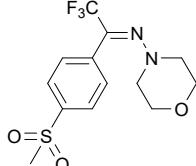
**(E)-1-(1-(4-chlorophenyl)-2,2,2-trifluoroethylidene)-2-phenylhydrazine
(3v)**

Brown oil. ^1H NMR (400 MHz, CDCl_3) δ 7.77 (s, 1H), 7.64 – 7.50 (m, 3H), 7.41 (dd, $J = 7.5, 2.0$ Hz, 2H), 7.25 – 7.17 (m, 2H), 7.04 – 6.95 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.70, 132.49 (q, $^2J_{\text{C},\text{F}} = 35.35$ Hz), 130.85, 130.06, 129.41, 129.03, 126.98, 126.82, 121.45 (q, $^1J_{\text{C},\text{F}} = 273.71$ Hz), 114.92. ^{19}F NMR (376 MHz, CDCl_3) δ -66.54. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{14}\text{H}_{10}\text{ClF}_3\text{N}_2$ (299.0557), found 299.0560.



**(E)-1-(1-(4-bromophenyl)-2,2,2-trifluoroethylidene)-2-phenylhydrazine
(3w)**

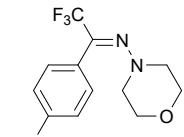
Brown oil. ^1H NMR (400 MHz, CDCl_3) δ 7.77 (s, 1H), 7.63 – 7.52 (m, 3H), 7.47 – 7.32 (m, 4H), 6.99 – 6.91 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 142.19, 132.65 (q, $^2J_{\text{C},\text{F}} = 35.35$ Hz), 132.31, 130.86, 130.06, 129.02, 126.97, 121.45 (q, $^1J_{\text{C},\text{F}} = 273.71$ Hz), 115.36, 114.15. ^{19}F NMR (376 MHz, CDCl_3) δ -66.55. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{14}\text{H}_{10}\text{BrF}_3\text{N}_2$ (343.0052), found 292.0059.



(E)-2,2,2-trifluoro-1-(4-(methylsulfonyl)phenyl)-N-morpholinoethan-1-

imine

Yellow solid; m.p.= 133.6- 135.0 °C. ^1H NMR (400 MHz, CDCl_3) δ 8.23 – 7.79 (m, 2H), 7.74 – 7.49 (m, 2H), 3.69 – 3.54 (m, 4H), 3.09 (s, 3H), 3.03 – 2.95 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.92, 137.49, 132.51(q, $^2J_{\text{C},\text{F}} = 33.33$ Hz), 129.91, 128.01, 121.16(q, $^1J_{\text{C},\text{F}} = 275.73$ Hz), 66.05, 54.53, 44.44. ^{19}F NMR (376 MHz, CDCl_3) δ -66.15. HRMS (ESI) m/z [M+H] $^+$ Calculated for $\text{C}_{13}\text{H}_{15}\text{F}_3\text{N}_2\text{O}_3\text{S}$ (337.0828), found 337.0835.

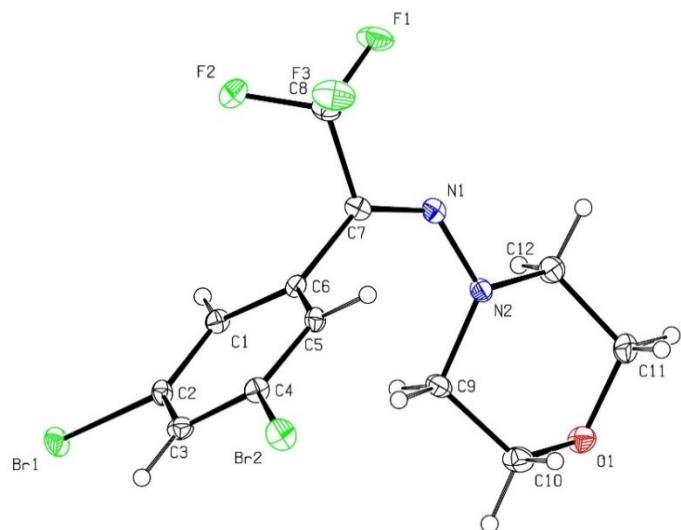
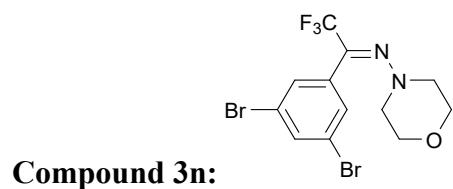


(E)-2,2,2-trifluoro-N-morpholino-1-(4-(trifluoromethyl)phenyl)ethan-1-imine

White solid; m.p.= 92.6- 93.6°C. ^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, $J = 8.0$ Hz, 2H), 7.57 (d, $J = 8.1$ Hz, 2H), 3.73 – 3.46 (m, 4H), 3.14 – 2.72 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 135.55, 133.96(q, $^2J_{\text{C},\text{F}} = 33.33$ Hz), 132.05(q, $^2J_{\text{C},\text{F}} = 32.32$ Hz), 129.28, 123.73(q, $^1J_{\text{C},\text{F}} = 273.71$ Hz), 121.22(q, $^1J_{\text{C},\text{F}} = 275.73$ Hz), 125.98(q, $^3J_{\text{C},\text{F}} = 3.535$ Hz), 66.13, 54.50. ^{19}F NMR (376 MHz, CDCl_3) δ -63.05, -66.47.

Spectroscopic data are in accordance with those described in the literature.^[4]

X. Crystal Data and Structure Refinement for 3n



CCDC number: 2178804

Table 1 Crystal data and structure refinement for 3n.

Identification code	3n
Empirical formula	$\text{C}_{12}\text{H}_{11}\text{Br}_2\text{F}_3\text{N}_2\text{O}$
Formula weight	416.05
Temperature/K	169.0
Crystal system	monoclinic
Space group	$\text{P}2_1/\text{c}$
a/Å	11.9638(4)
b/Å	7.6176(2)
c/Å	16.0976(6)
$\alpha/^\circ$	90
$\beta/^\circ$	111.5740(10)
$\gamma/^\circ$	90
Volume/Å ³	1364.28(8)
Z	4
$\rho_{\text{calc}}/\text{g/cm}^3$	2.026
μ/mm^{-1}	5.973
F(000)	808.0

Crystal size/mm ³	0.49 × 0.32 × 0.18
Radiation	MoKα ($\lambda = 0.71073$)
2Θ range for data collection/°	5.326 to 54.322
Index ranges	-15 ≤ h ≤ 15, -9 ≤ k ≤ 8, -20 ≤ l ≤ 20
Reflections collected	9355
Independent reflections	3005 [$R_{\text{int}} = 0.0580$, $R_{\text{sigma}} = 0.0644$]
Data/restraints/parameters	3005/0/181
Goodness-of-fit on F^2	1.050
Final R indexes [I>=2σ (I)]	$R_1 = 0.0494$, $wR_2 = 0.1232$
Final R indexes [all data]	$R_1 = 0.0567$, $wR_2 = 0.1276$
Largest diff. peak/hole / e Å ⁻³	1.15/-1.99

Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters (Å²×10³) for 3n. U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
Br1	2940.1 (4)	9498.4 (5)	810.4 (3)	24.80 (16)
Br2	6175.9 (4)	9705.7 (5)	4396.4 (3)	27.41 (16)
F1	2329 (2)	2187 (3)	3418.0 (19)	35.6 (6)
F2	3063 (2)	3195 (3)	2484.1 (18)	34.2 (6)
F3	4087 (2)	3348 (3)	3884.9 (19)	37.4 (6)
O1	-564 (2)	9381 (3)	3875 (2)	24.9 (6)
N1	1565 (3)	5300 (4)	3573 (2)	17.6 (6)
N2	1013 (3)	6730 (4)	3731 (2)	18.2 (6)
C1	2782 (3)	7338 (4)	2195 (2)	17.8 (7)
C2	3427 (3)	8679 (5)	2000 (2)	17.6 (7)
C3	4429 (3)	9416 (5)	2646 (3)	20.5 (8)
C4	4781 (3)	8773 (5)	3509 (3)	18.5 (7)
C5	4147 (3)	7454 (5)	3736 (2)	17.1 (7)
C6	3136 (3)	6753 (4)	3076 (2)	15.7 (7)
C7	2454 (3)	5296 (4)	3314 (3)	18.4 (8)
C8	2967 (3)	3500 (5)	3277 (3)	23.8 (8)
C9	1001 (3)	8466 (5)	3338 (3)	23.0 (8)
C10	571 (4)	9812 (5)	3847 (3)	27.2 (9)
C11	-486 (4)	7734 (5)	4313 (3)	27.0 (9)
C12	-132 (3)	6293 (5)	3819 (3)	25.0 (8)

Table 3 Anisotropic Displacement Parameters (Å²×10³) for 3n. The Anisotropic

displacement factor exponent takes the form: $-2\pi^2[\mathbf{h}^2\mathbf{a}^{*2}\mathbf{U}_{11}+2\mathbf{hka}^{*}\mathbf{b}^{*}\mathbf{U}_{12}+\dots]$.

Atom	U₁₁	U₂₂	U₃₃	U₂₃	U₁₃	U₁₂
Br1	27.4 (2)	29.6 (2)	15.5 (3)	6.96 (14)	5.65 (19)	5.27 (14)
Br2	22.8 (2)	32.5 (2)	20.8 (3)	-3.96 (15)	0.83 (19)	-10.77 (15)
F1	33.5 (13)	18.2 (10)	61.1 (19)	4.3 (11)	24.5 (13)	-3.8 (9)
F2	44.4 (14)	28.5 (12)	36.5 (16)	-1.9 (11)	23.0 (13)	7.5 (11)
F3	23.3 (12)	30.6 (12)	45.3 (17)	9.1 (12)	-2.8 (12)	6.8 (10)
O1	20.6 (14)	23.4 (13)	33.2 (18)	-0.9 (12)	12.7 (13)	1.1 (10)
N1	16.8 (15)	20.6 (14)	11.1 (17)	0.6 (11)	0.1 (13)	-1.9 (11)
N2	18.6 (15)	18.6 (13)	18.6 (17)	2.1 (12)	8.2 (13)	0.1 (11)
C1	17.8 (17)	19.1 (15)	12.5 (18)	-1.3 (14)	0.9 (15)	0.7 (13)
C2	16.1 (16)	22.7 (16)	13.9 (19)	2.8 (14)	5.4 (15)	3.9 (13)
C3	18.7 (18)	16.9 (16)	27 (2)	-2.0 (14)	10.0 (18)	-1.0 (13)
C4	15.7 (16)	21.1 (16)	14.6 (19)	-1.8 (14)	0.7 (15)	-0.8 (13)
C5	17.0 (16)	20.6 (16)	13.1 (18)	0.2 (14)	5.0 (15)	0.7 (13)
C6	15.2 (16)	16.7 (15)	16.0 (19)	-3.0 (13)	6.6 (15)	-0.5 (13)
C7	14.3 (17)	18.8 (16)	17 (2)	1.0 (14)	0.5 (15)	-2.6 (12)
C8	19.6 (18)	20.5 (17)	31 (2)	2.3 (16)	8.9 (17)	-2.7 (14)
C9	25.2 (19)	17.9 (16)	29 (2)	5.7 (15)	14.0 (18)	1.5 (14)
C10	21.8 (19)	21.2 (17)	42 (3)	0.5 (17)	15 (2)	-2.3 (15)
C11	29 (2)	28.9 (19)	30 (2)	-1.5 (17)	18.4 (19)	-2.0 (16)
C12	24.0 (19)	23.7 (18)	31 (2)	-2.4 (16)	14.3 (18)	-3.8 (15)

Table 4 Bond Lengths for 3n.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Br1	C2	1.891 (4)	C1	C2	1.385 (5)
Br2	C4	1.892 (4)	C1	C6	1.395 (5)
F1	C8	1.327 (4)	C2	C3	1.385 (5)
F2	C8	1.342 (5)	C3	C4	1.383 (5)
F3	C8	1.343 (5)	C4	C5	1.386 (5)
O1	C10	1.414 (4)	C5	C6	1.391 (5)
O1	C11	1.425 (5)	C6	C7	1.508 (5)
N1	N2	1.346 (4)	C7	C8	1.510 (5)
N1	C7	1.278 (5)	C9	C10	1.515 (5)
N2	C9	1.464 (4)	C11	C12	1.505 (5)
N2	C12	1.465 (4)			

Table 5 Bond Angles for 3n.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C10	O1	C11	109.3 (3)	C5	C6	C1	120.5 (3)
C7	N1	N2	126.1 (3)	C5	C6	C7	119.2 (3)
N1	N2	C9	125.2 (3)	N1	C7	C6	132.4 (3)
N1	N2	C12	112.1 (3)	N1	C7	C8	114.5 (3)
C9	N2	C12	112.7 (3)	C6	C7	C8	113.1 (3)
C2	C1	C6	118.7 (3)	F1	C8	F2	106.7 (3)
C1	C2	Br1	118.9 (3)	F1	C8	F3	107.0 (3)
C1	C2	C3	122.0 (3)	F1	C8	C7	114.1 (3)
C3	C2	Br1	119.1 (3)	F2	C8	F3	105.5 (3)
C4	C3	C2	118.1 (3)	F2	C8	C7	111.8 (3)
C3	C4	Br2	118.9 (3)	F3	C8	C7	111.1 (3)
C3	C4	C5	121.8 (3)	N2	C9	C10	109.2 (3)
C5	C4	Br2	119.3 (3)	O1	C10	C9	112.4 (3)
C4	C5	C6	118.9 (3)	O1	C11	C12	111.1 (3)
C1	C6	C7	120.2 (3)	N2	C12	C11	109.9 (3)

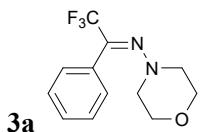
Table 6 Torsion Angles for 3n.

A	B	C	D	Angle/°	A	B	C	D	Angle/°
Br1	C2	C3	C4	-178.8 (3)	C2	C3	C4	C5	-1.3 (5)
Br2	C4	C5	C6	-178.8 (3)	C3	C4	C5	C6	0.6 (5)
O1	C11	C12	N2	57.1 (5)	C4	C5	C6	C1	1.5 (5)
N1	N2	C9	C10	-165.8 (4)	C4	C5	C6	C7	178.9 (3)
N1	N2	C12	C11	159.4 (3)	C5	C6	C7	N1	91.3 (5)
N1	C7	C8	F1	7.5 (5)	C5	C6	C7	C8	-85.4 (4)
N1	C7	C8	F2	128.8 (4)	C6	C1	C2	Br1	-179.2 (2)
N1	C7	C8	F3	-113.6 (4)	C6	C1	C2	C3	1.9 (5)
N2	N1	C7	C6	-2.3 (7)	C6	C7	C8	F1	-175.2 (3)
N2	N1	C7	C8	174.3 (3)	C6	C7	C8	F2	-53.9 (4)
N2	C9	C10	O1	-55.7 (5)	C6	C7	C8	F3	63.7 (4)
C1	C2	C3	C4	0.0 (5)	C7	N1	N2	C9	24.2 (6)
C1	C6	C7	N1	-91.3 (5)	C7	N1	N2	C12	166.9 (4)
C1	C6	C7	C8	92.1 (4)	C9	N2	C12	C11	-53.0 (4)
C2	C1	C6	C5	-2.7 (5)	C10	O1	C11	C12	-60.9 (4)
C2	C1	C6	C7	179.9 (3)	C11	O1	C10	C9	60.6 (5)
C2	C3	C4	Br2	178.0 (3)	C12	N2	C9	C10	51.6 (4)

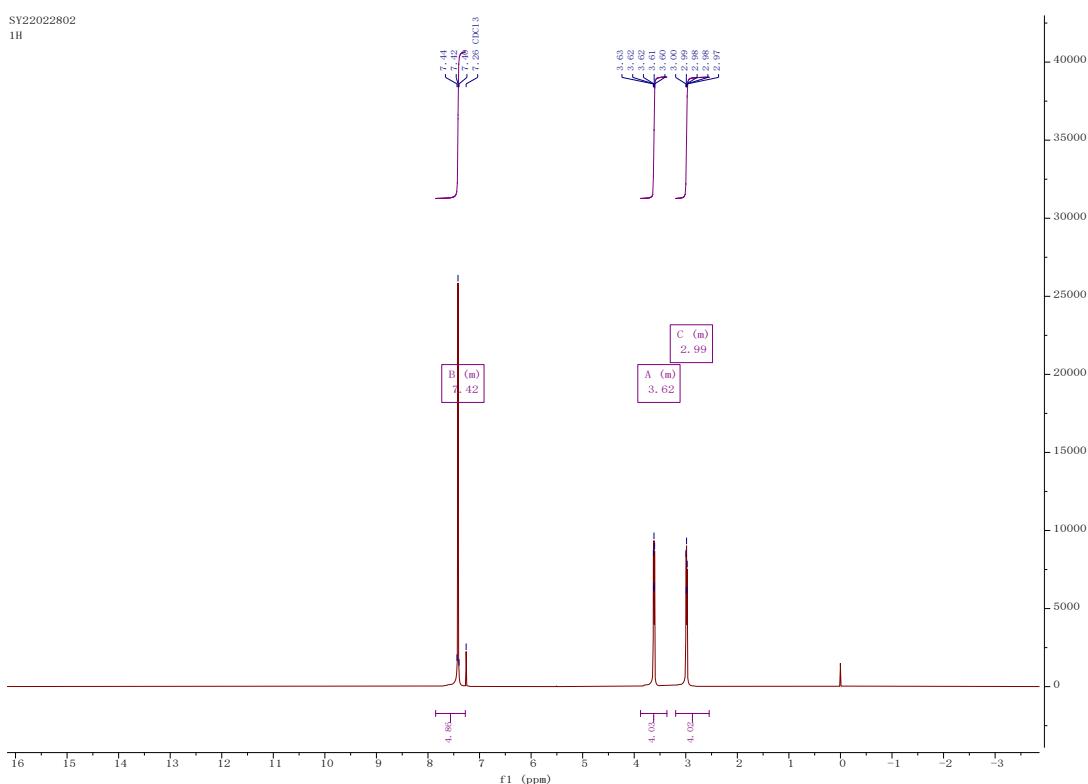
Table 7 Hydrogen Atom Coordinates ($\text{\AA} \times 10^4$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$) for 3n.

Atom	x	y	z	U(eq)
H1	2111.25	6825.53	1738.3	21
H3	4861.99	10336.67	2502.05	25
H5	4399.39	7037.27	4333.14	20
H9A	459.04	8457.17	2702.26	28
H9B	1819.71	8772.32	3369.1	28
H10A	1165.67	9900.2	4464.61	33
H10B	519.91	10973.63	3559.37	33
H11A	-1273.61	7449.74	4350.75	32
H11B	114.98	7814.92	4928.97	32
H12A	-51.28	5173.22	4148.31	30
H12B	-763.24	6143.62	3219.27	30

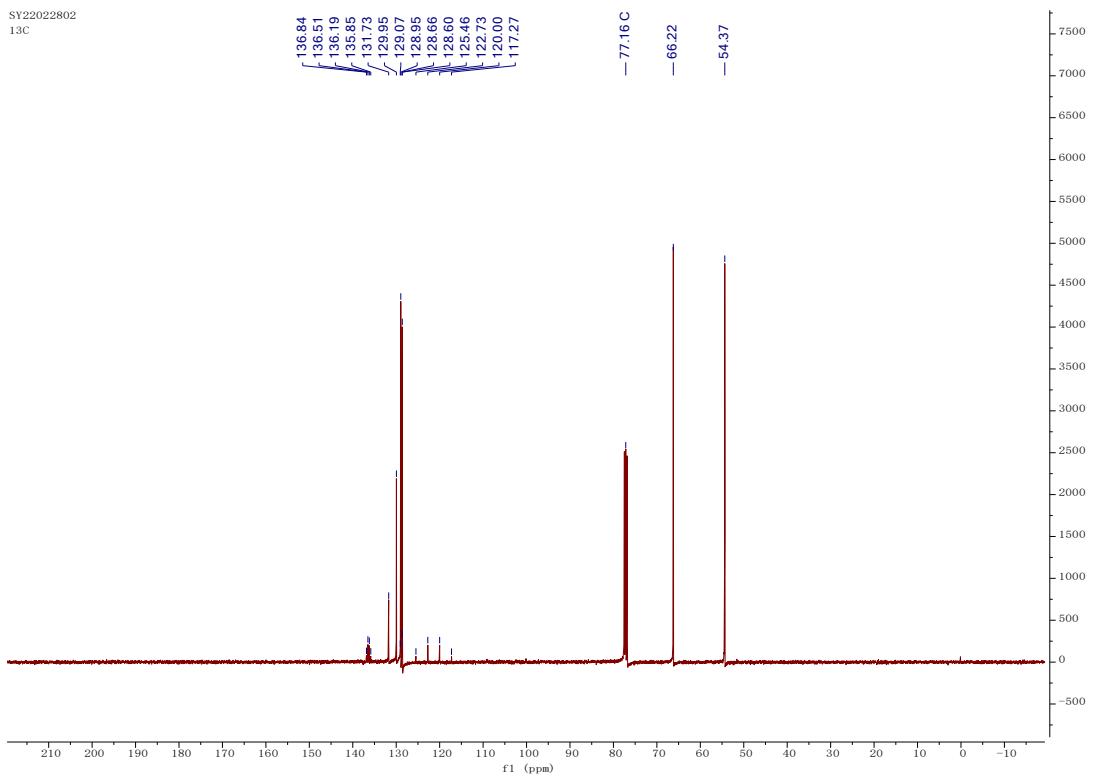
Copies of NMR spectra:¹H-, ¹³C- and ¹⁹F-NMR spectra



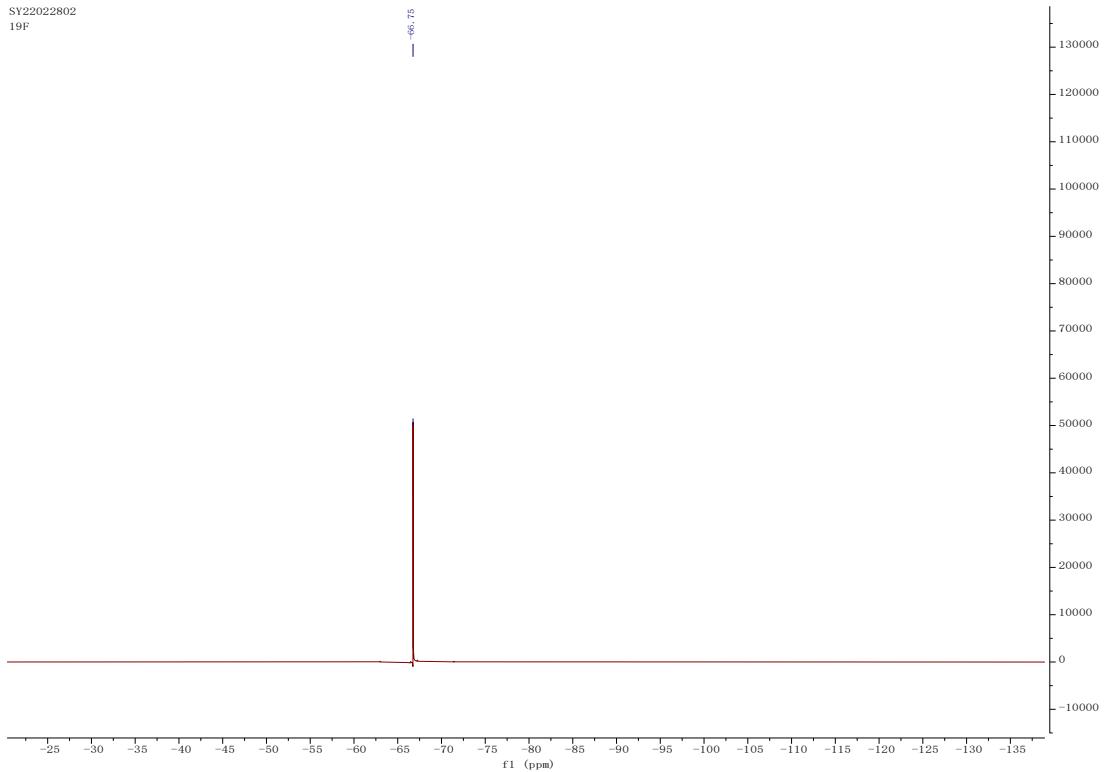
¹H NMR

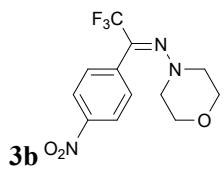


¹³C NMR



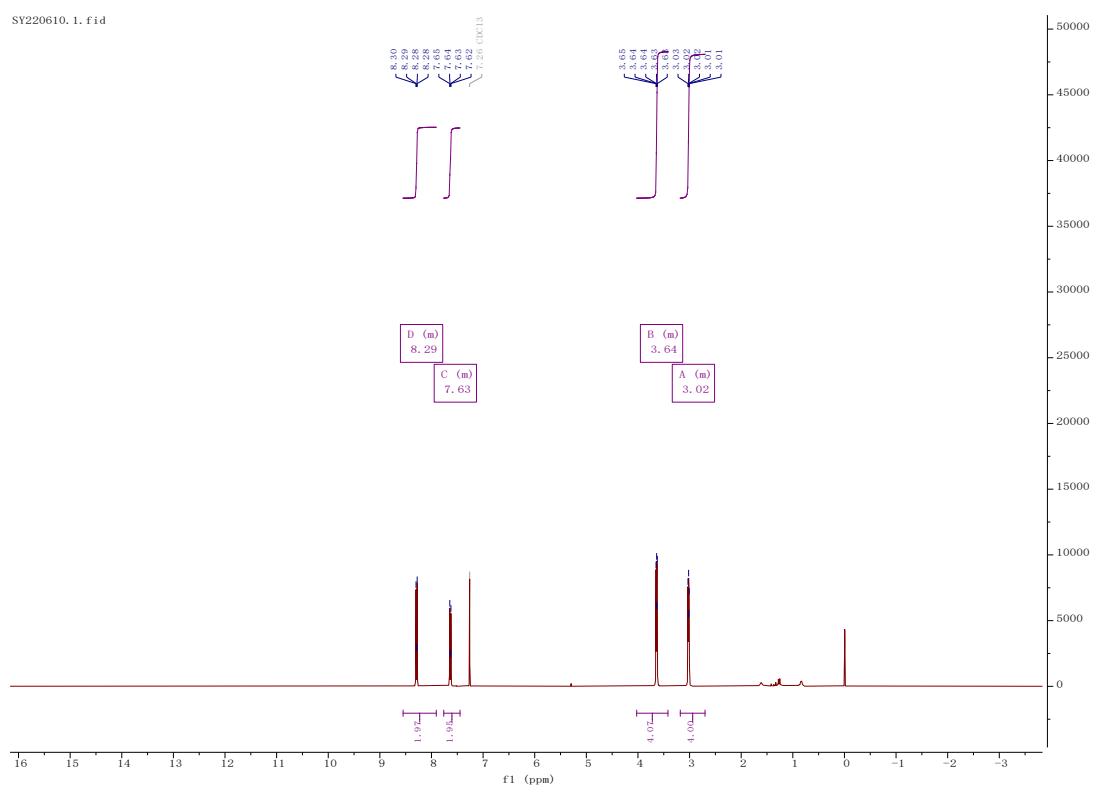
¹⁹F NMR



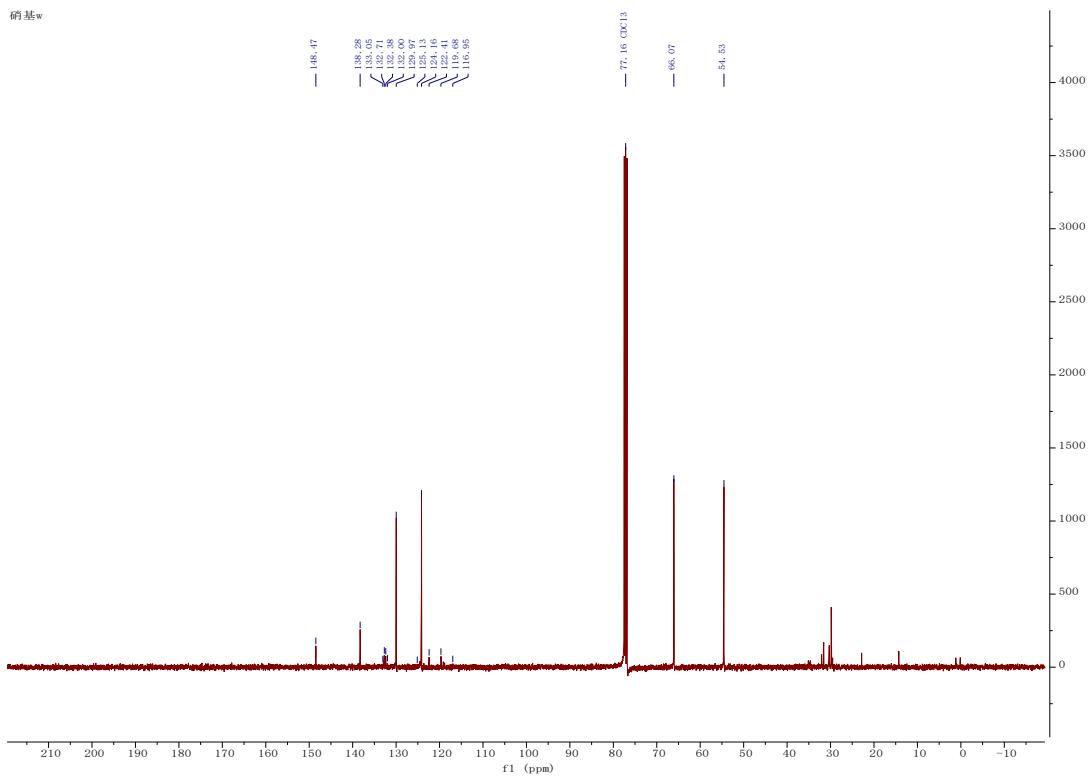


¹H NMR

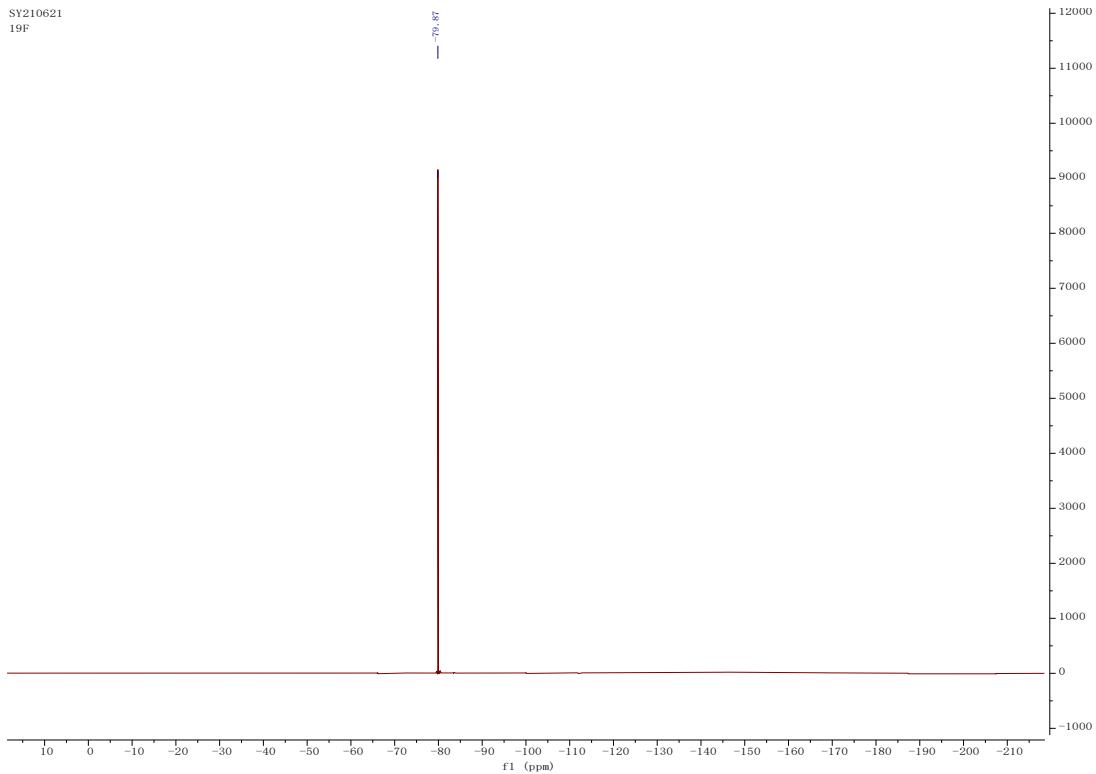
SY220610.1. fid

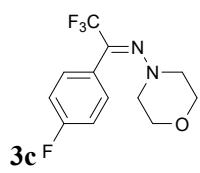


¹³C NMR



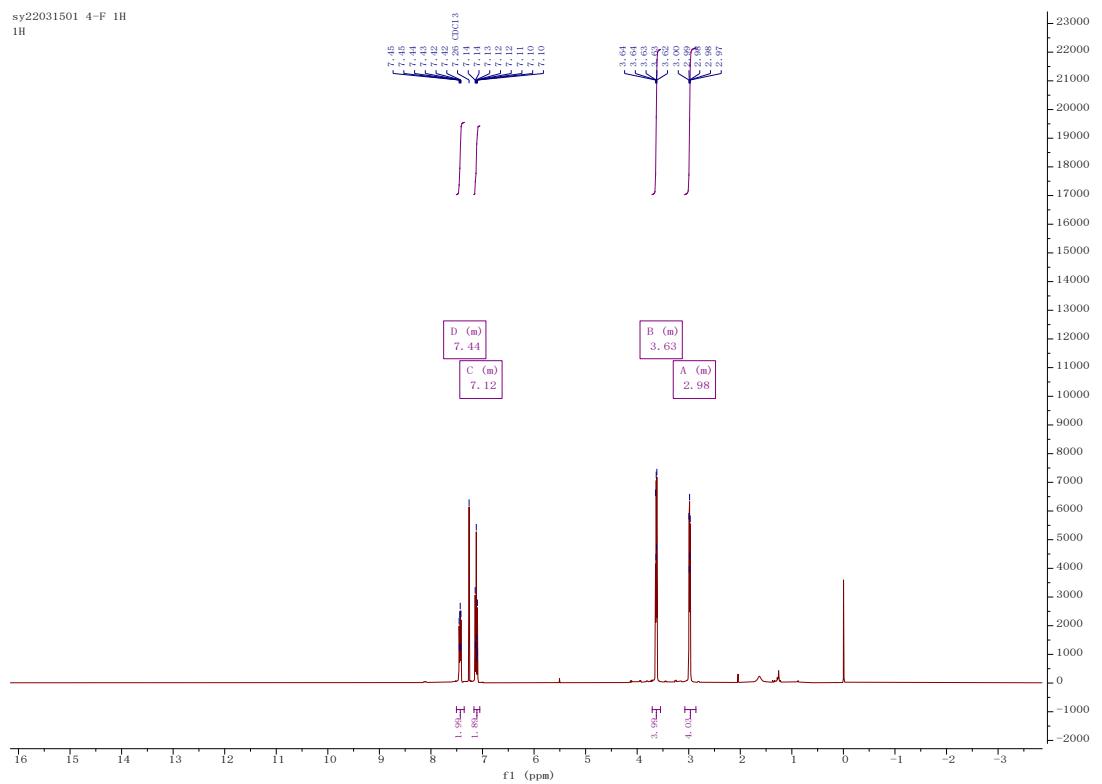
¹⁹F NMR





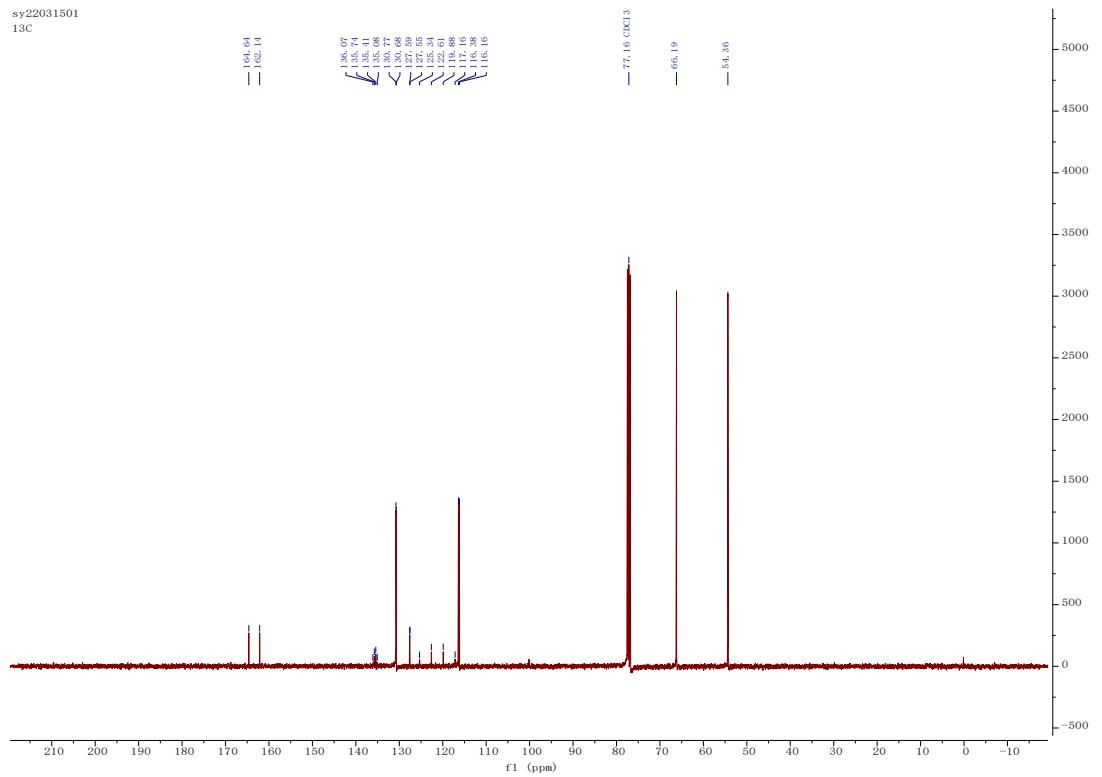
¹H NMR

sy22031501 4-F 1H
1H



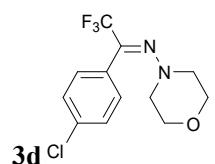
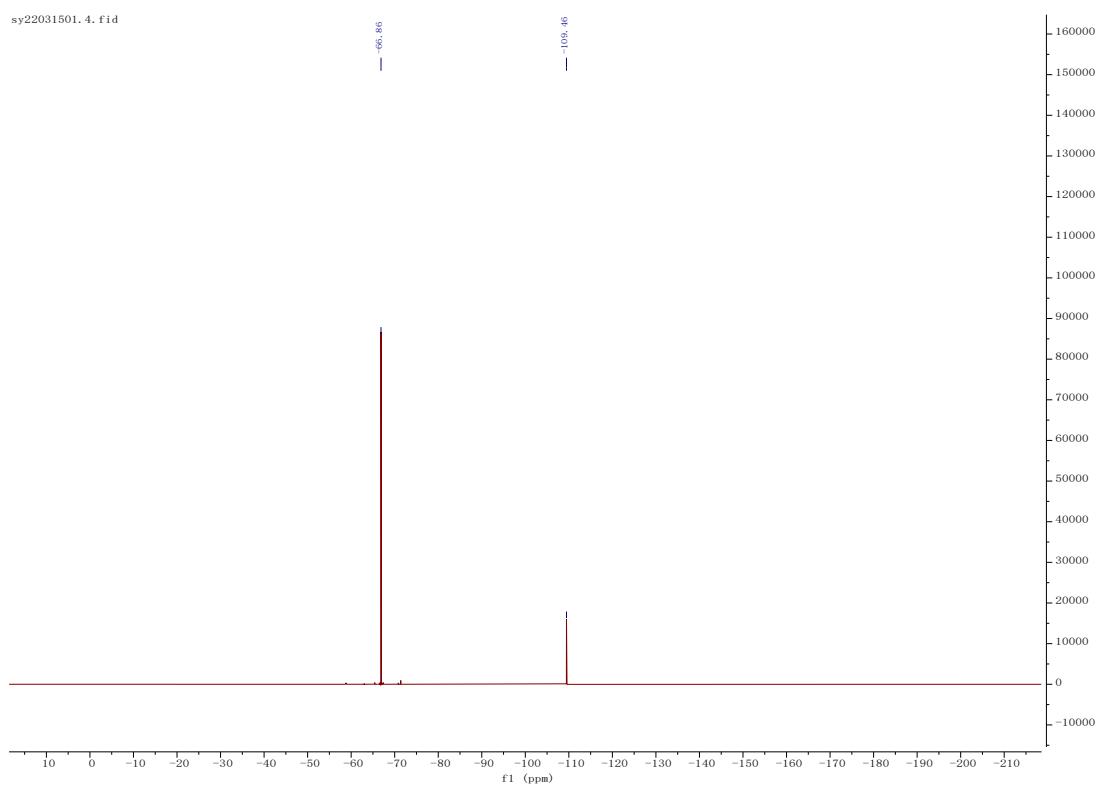
¹³C NMR

sy22031501
13C

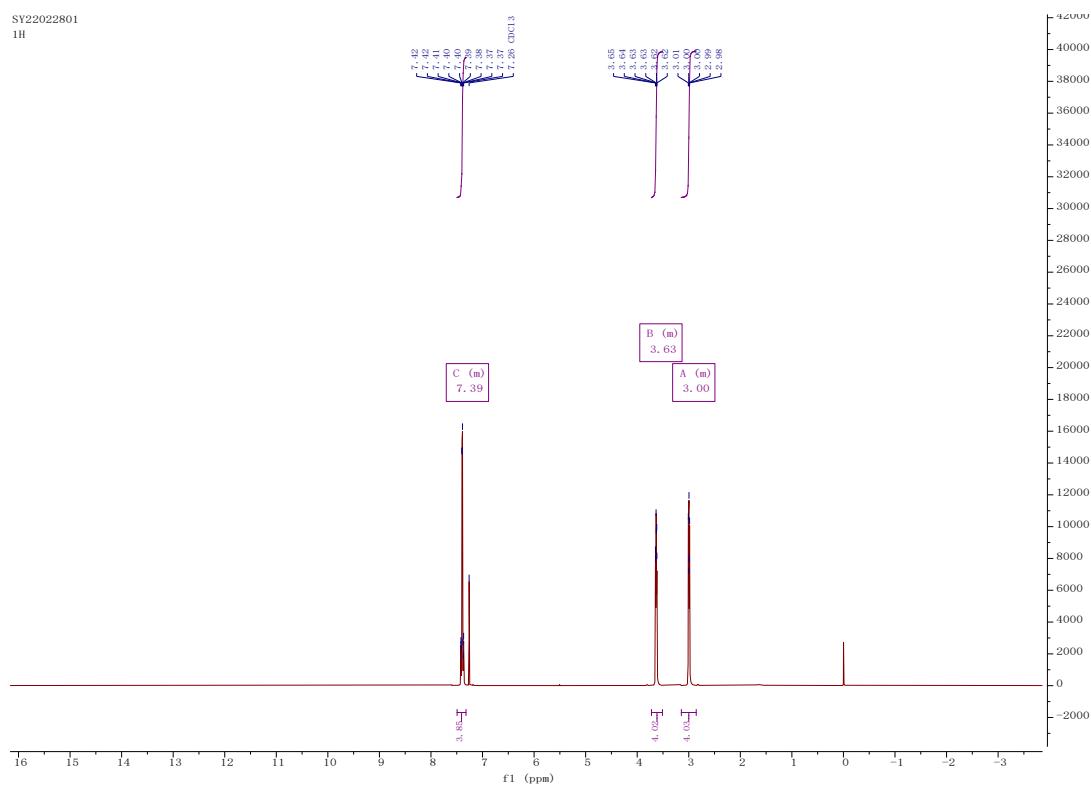


¹⁹F NMR

sy22031501.4.fid

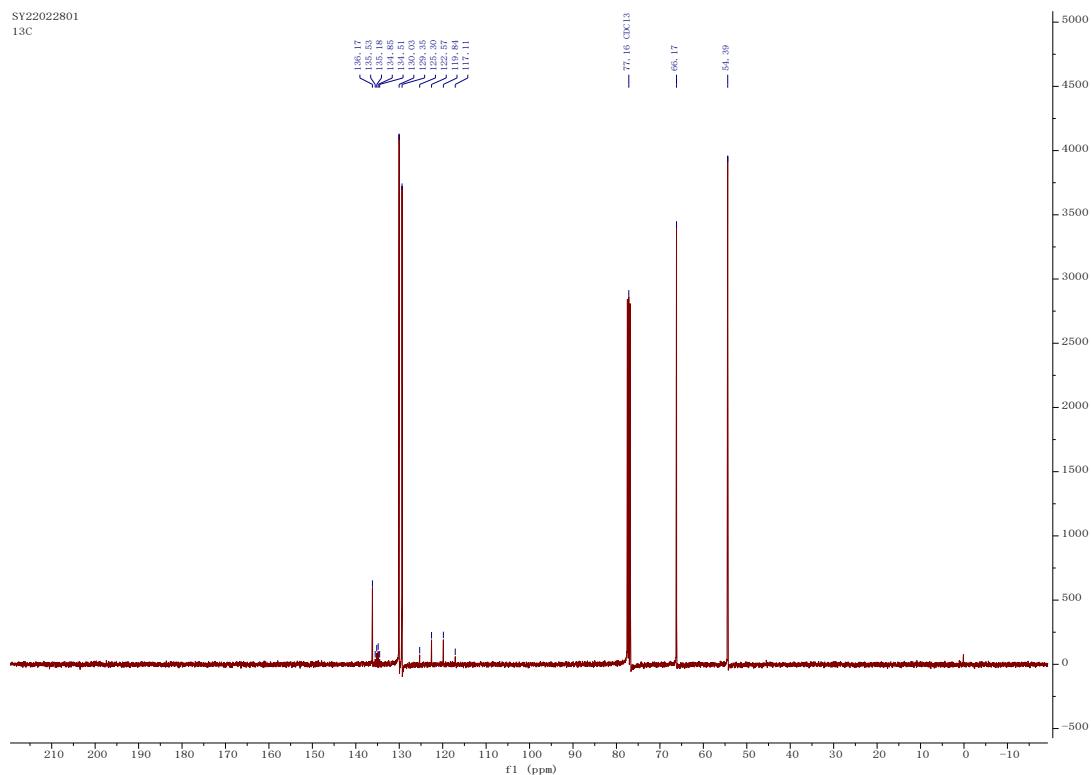


¹H NMR

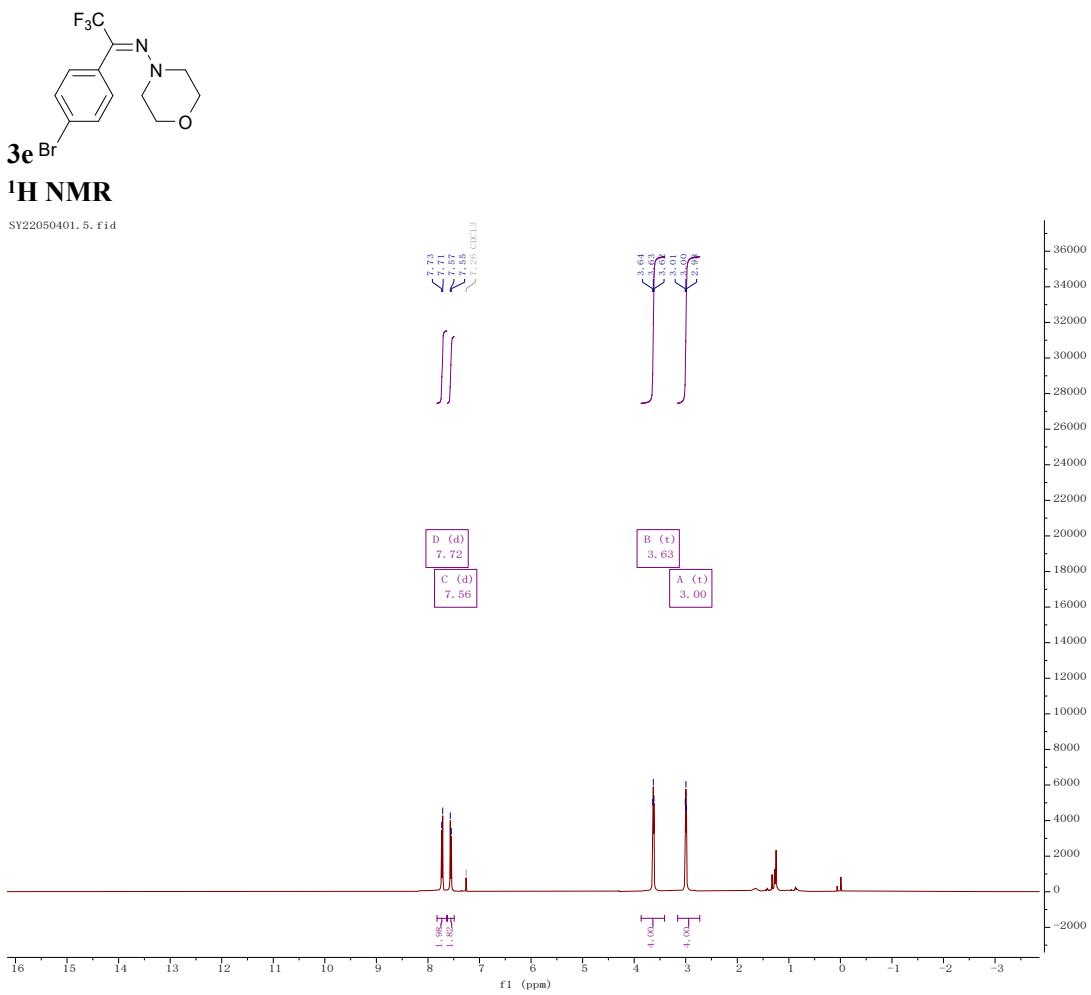
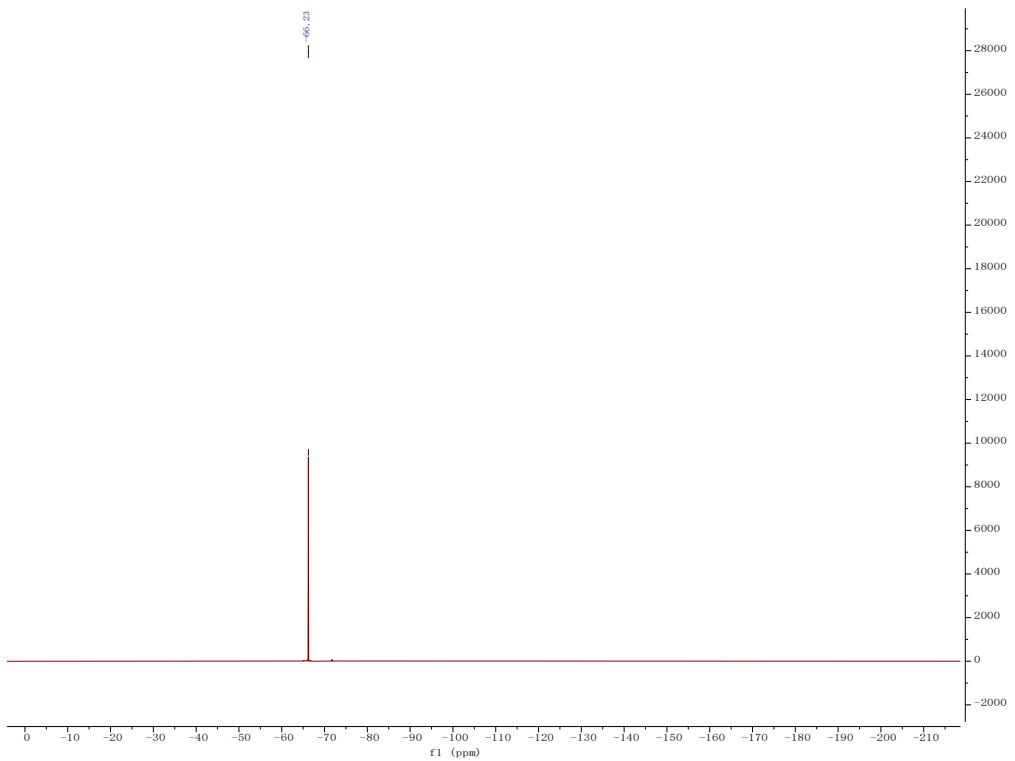


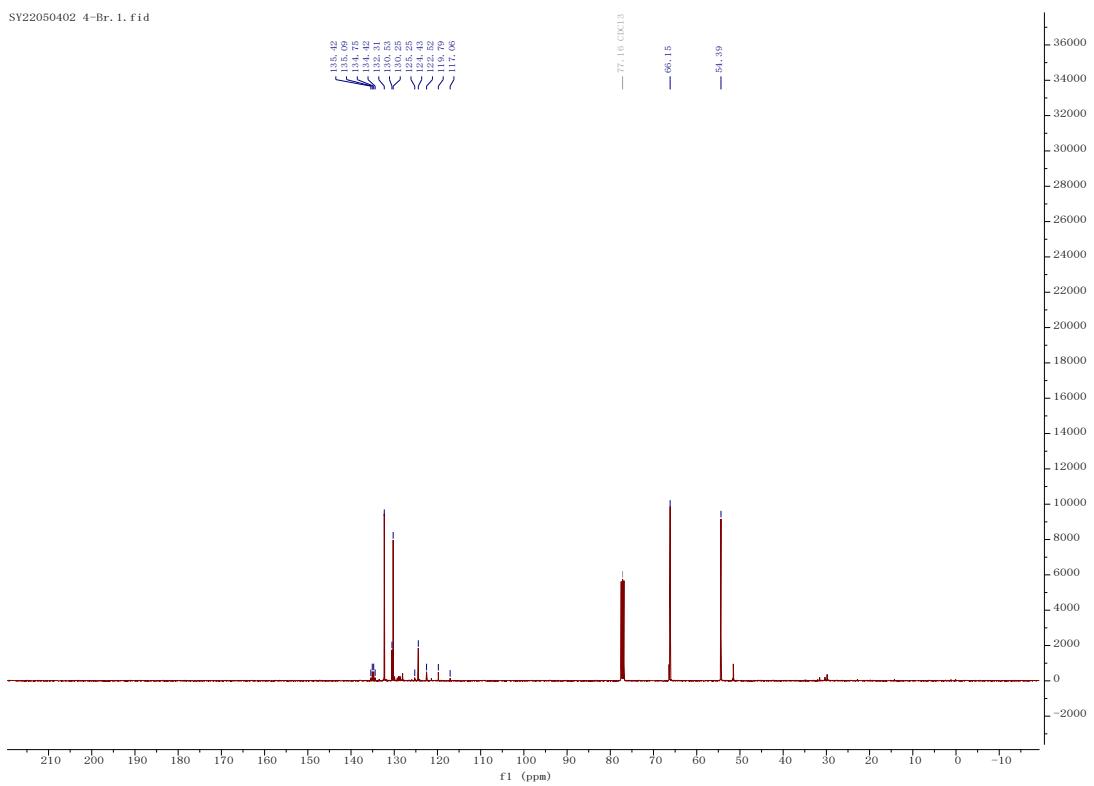
¹³C NMR

SY22022801
13C

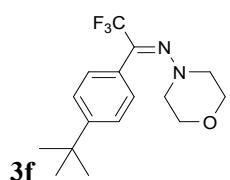
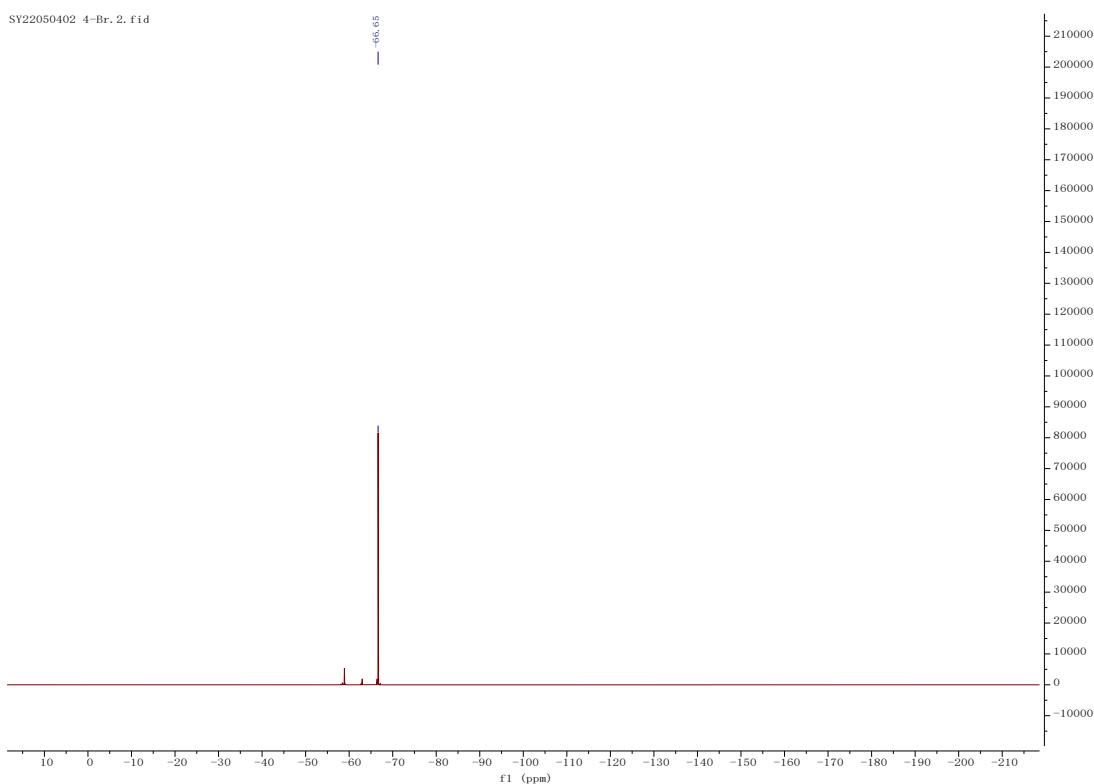


¹⁹F NMR



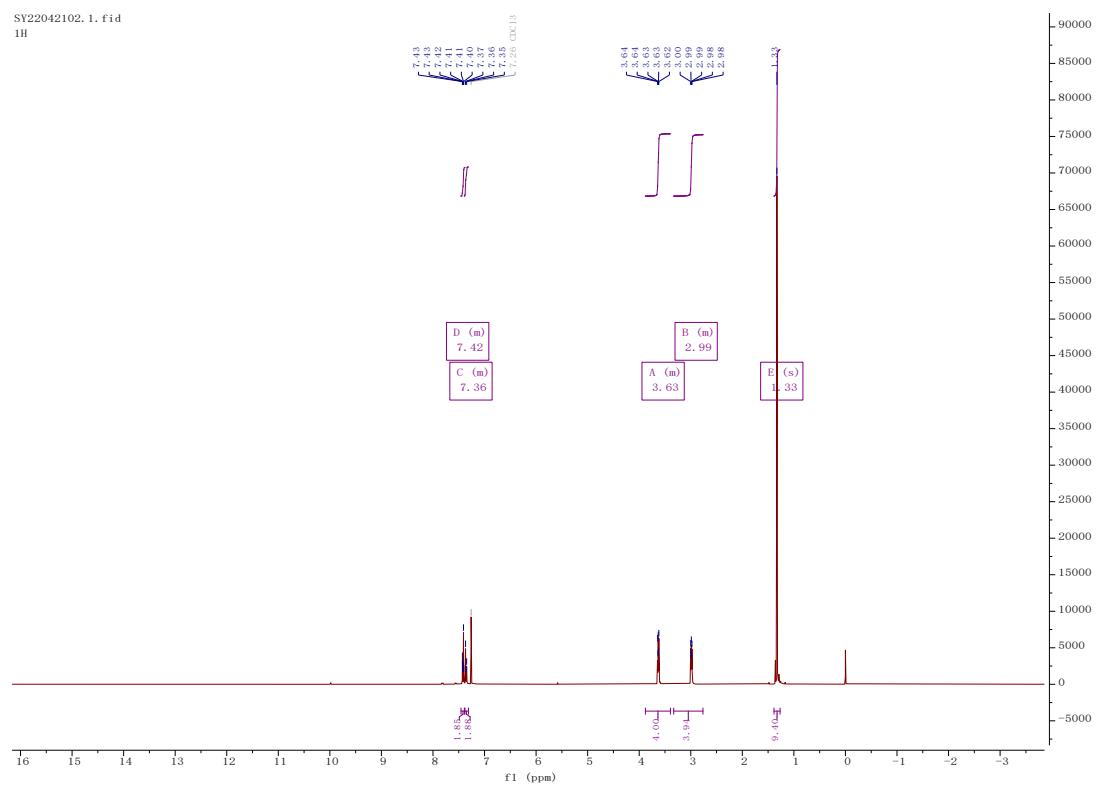


¹⁹F NMR



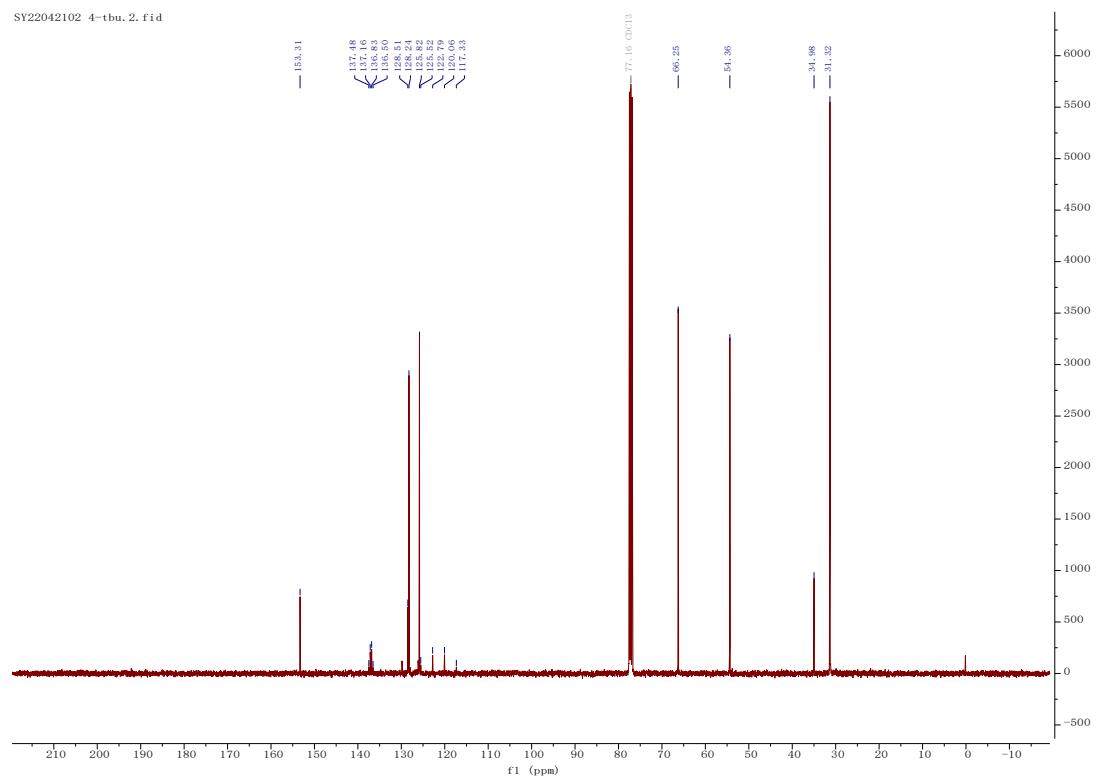
¹H NMR

SY22042102, 1, fid
1H



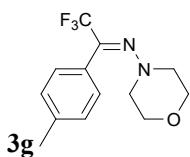
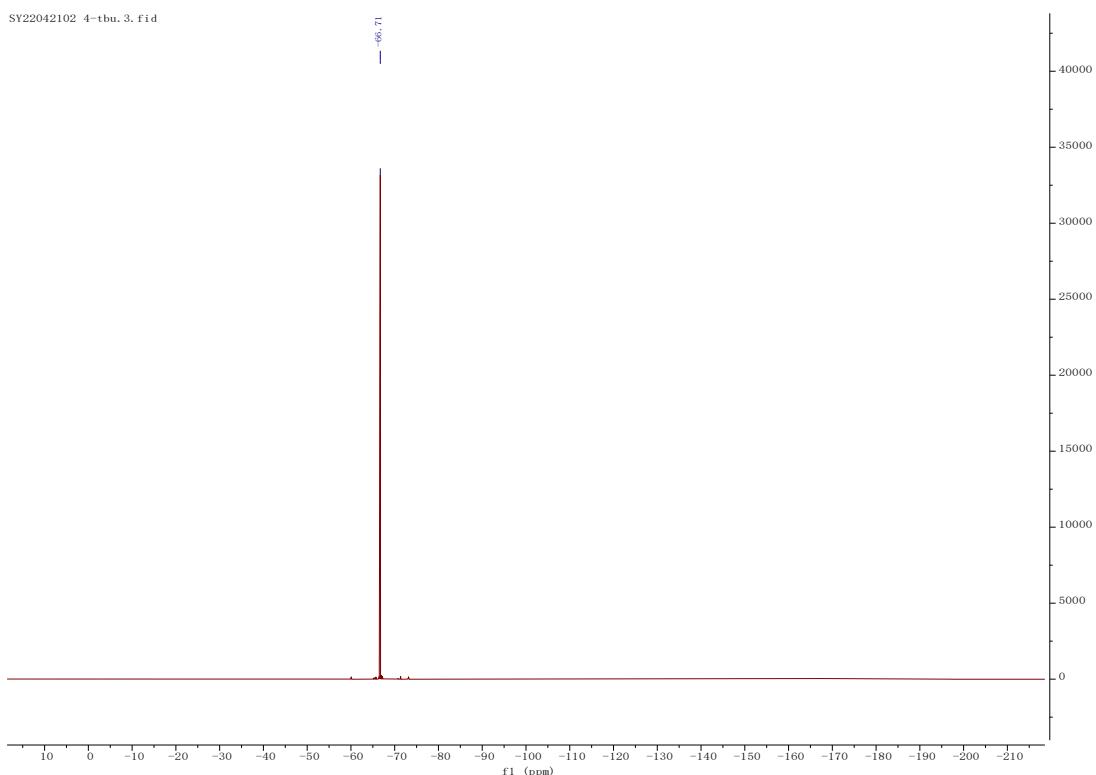
¹³C NMR

SY22042102 4-tbu. 2, fid



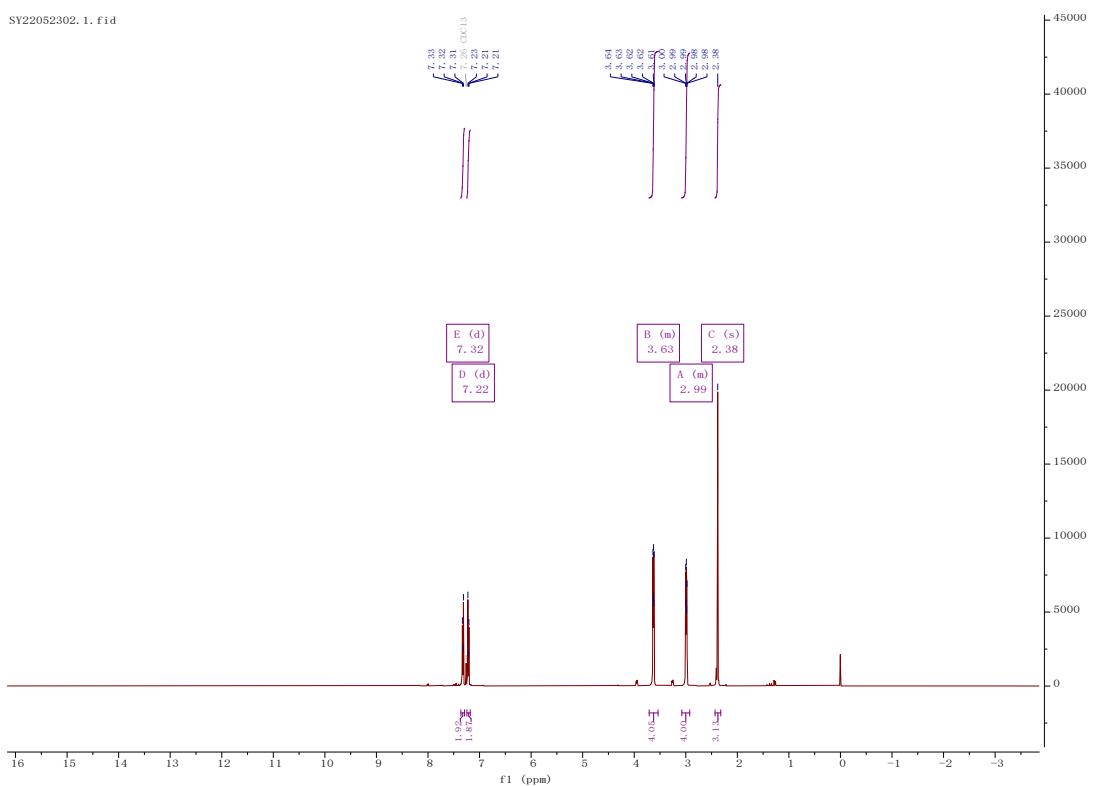
¹⁹F NMR

SY22042102 4-tbu_3.fid



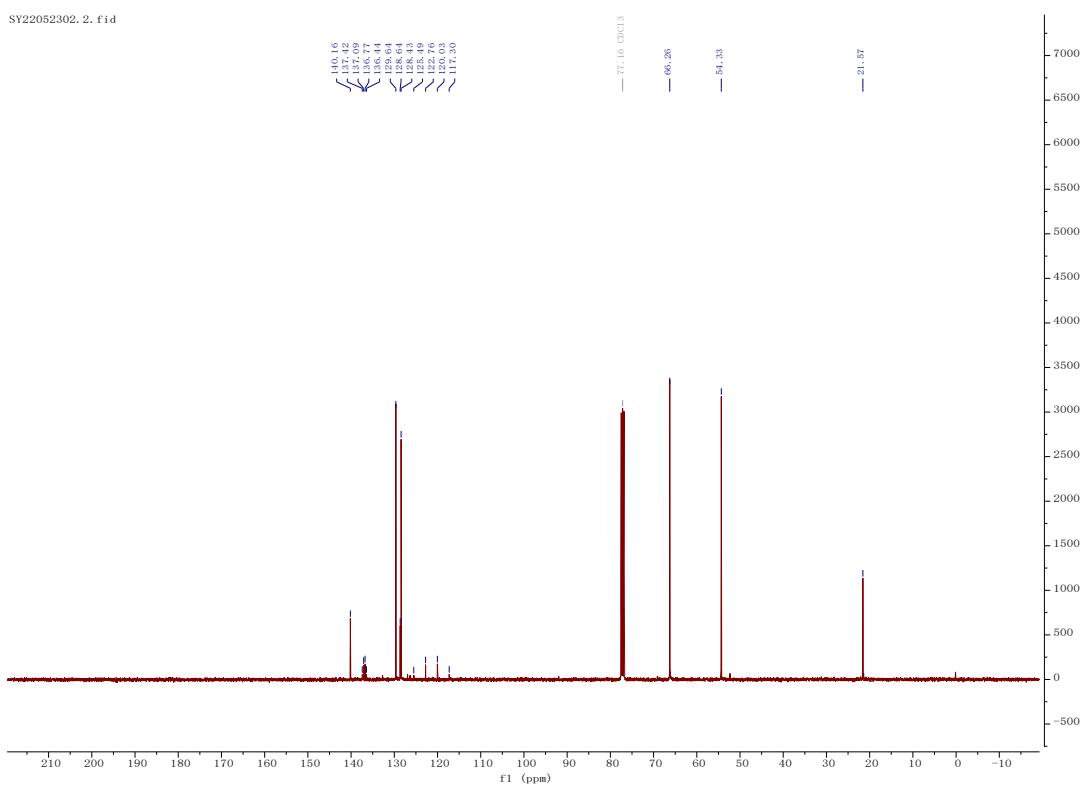
¹H NMR

SY22052302_1.fid



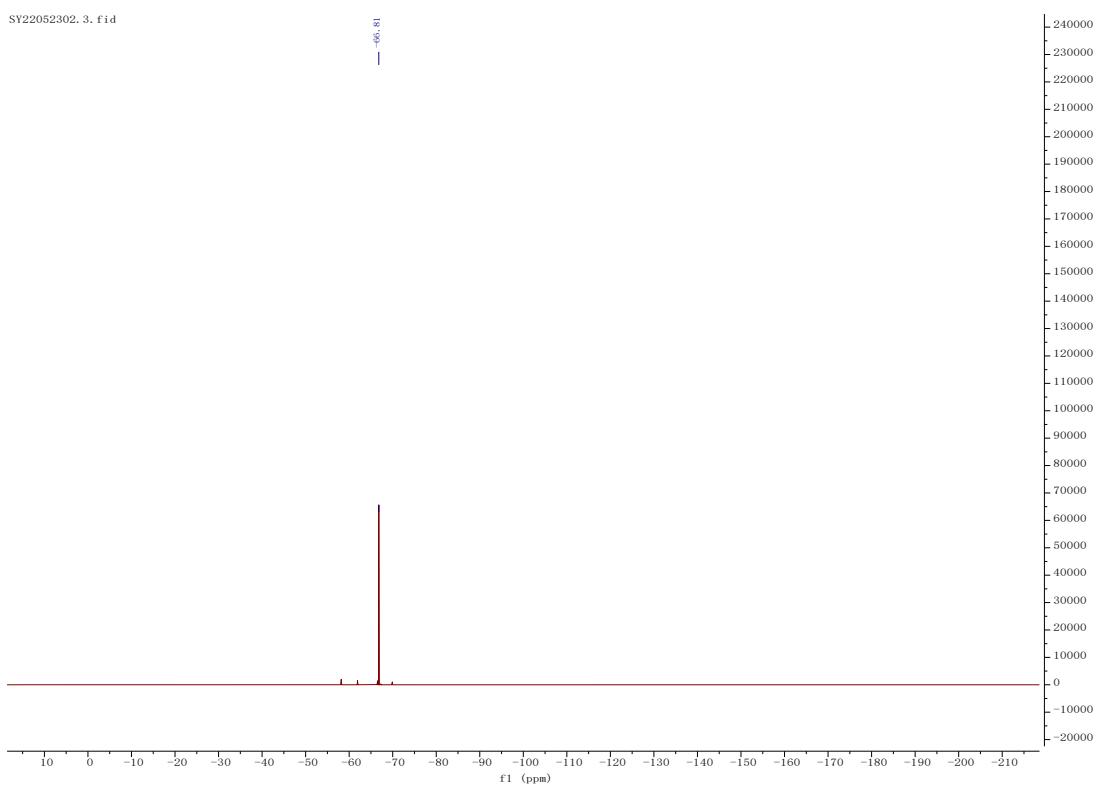
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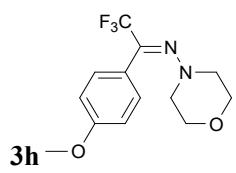
SY22052302, 2, fid



¹⁹F NMR

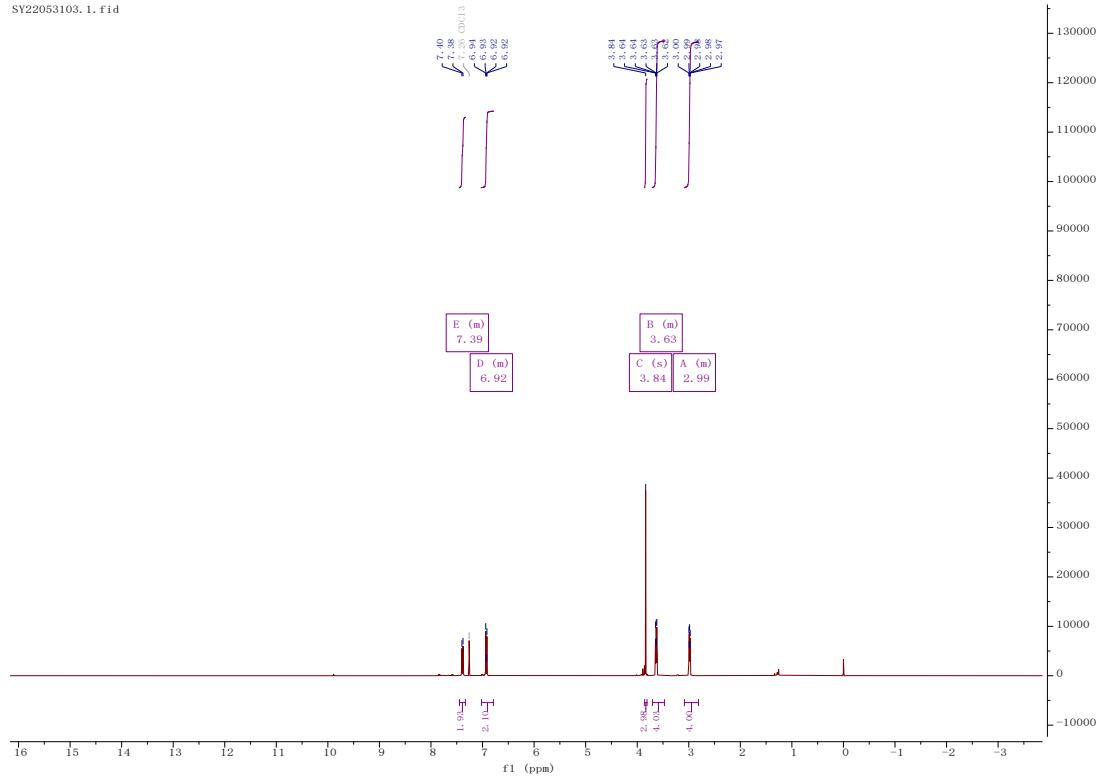
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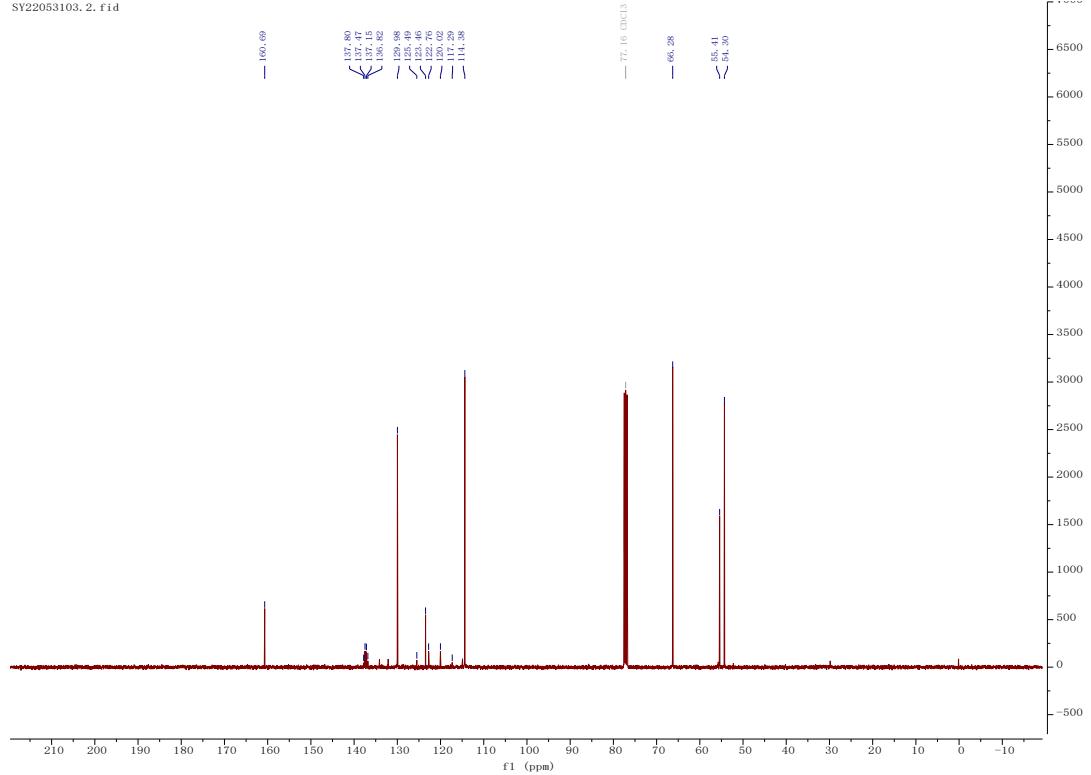
¹H NMR

SY22053103, 1, fid

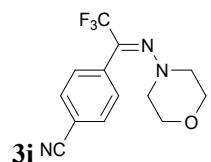
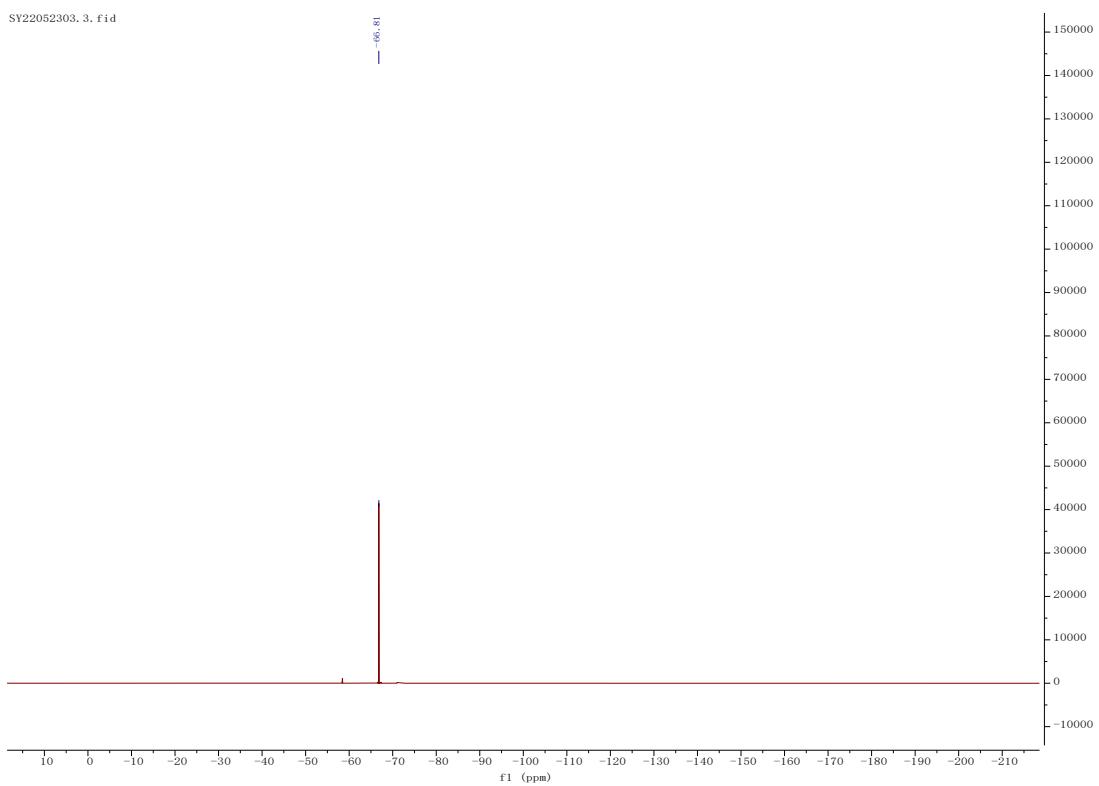


¹³C NMR

SY22053103. 2. fid

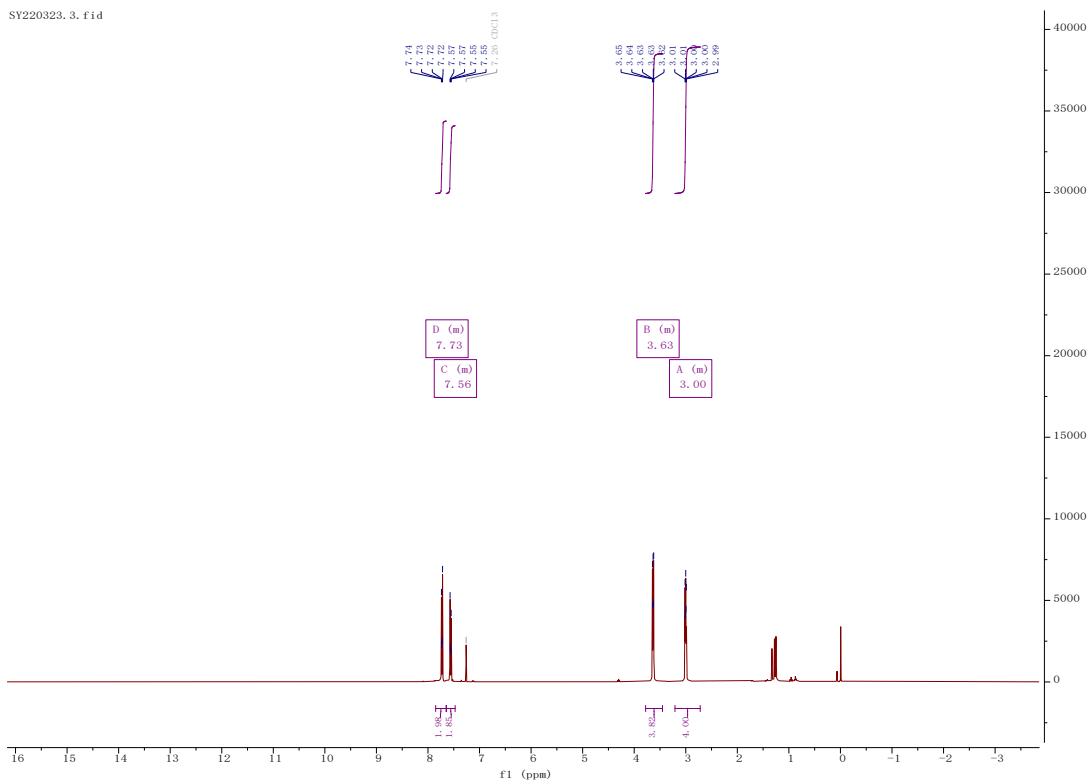


¹⁹F NMR



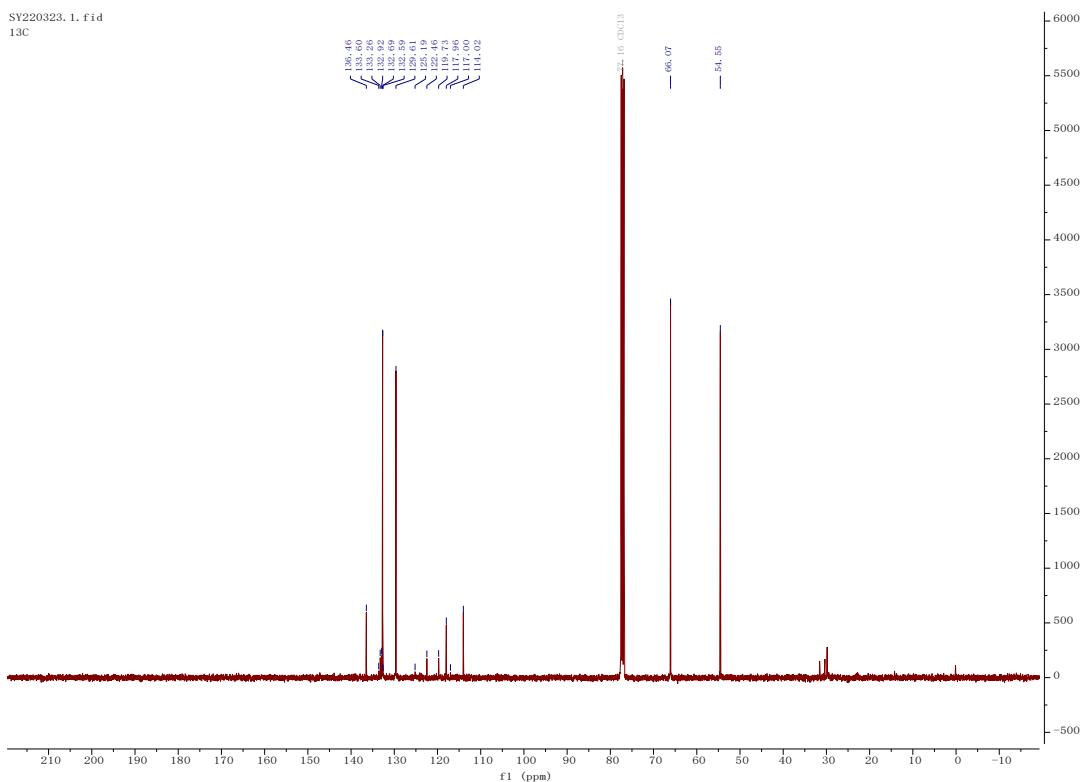
¹H NMR

SY220323. 3. fid



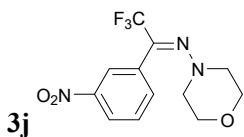
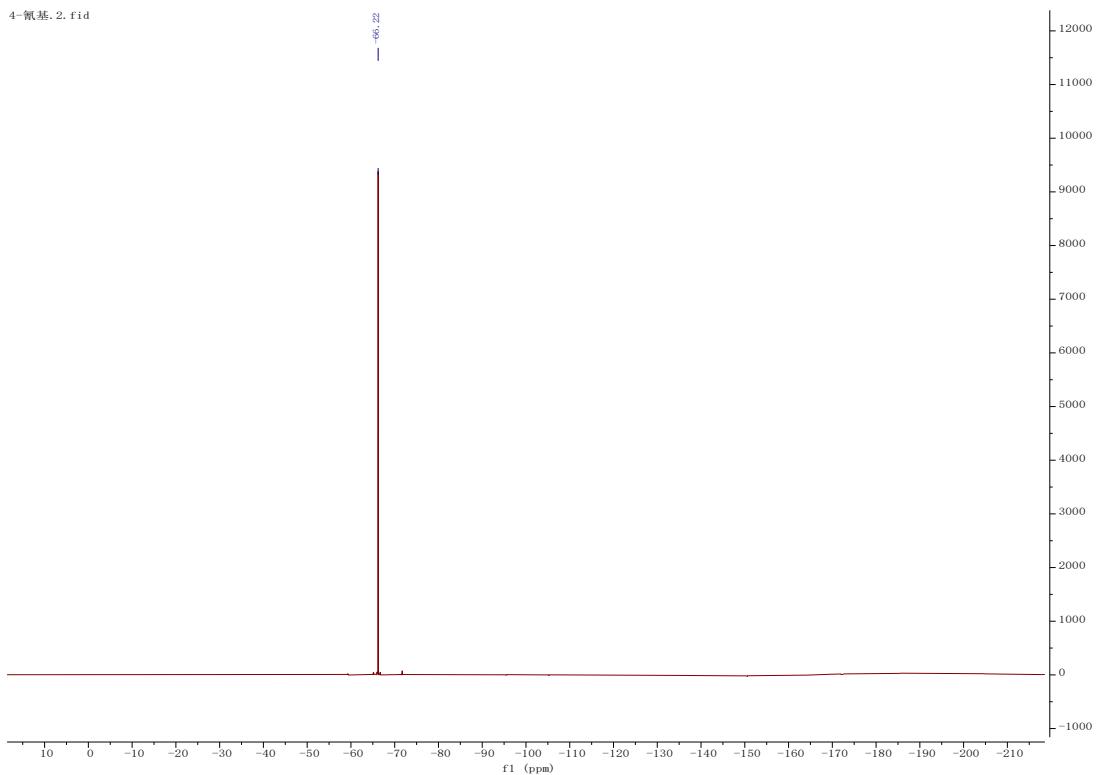
¹³C NMR

SY220323. 1. fid
13C



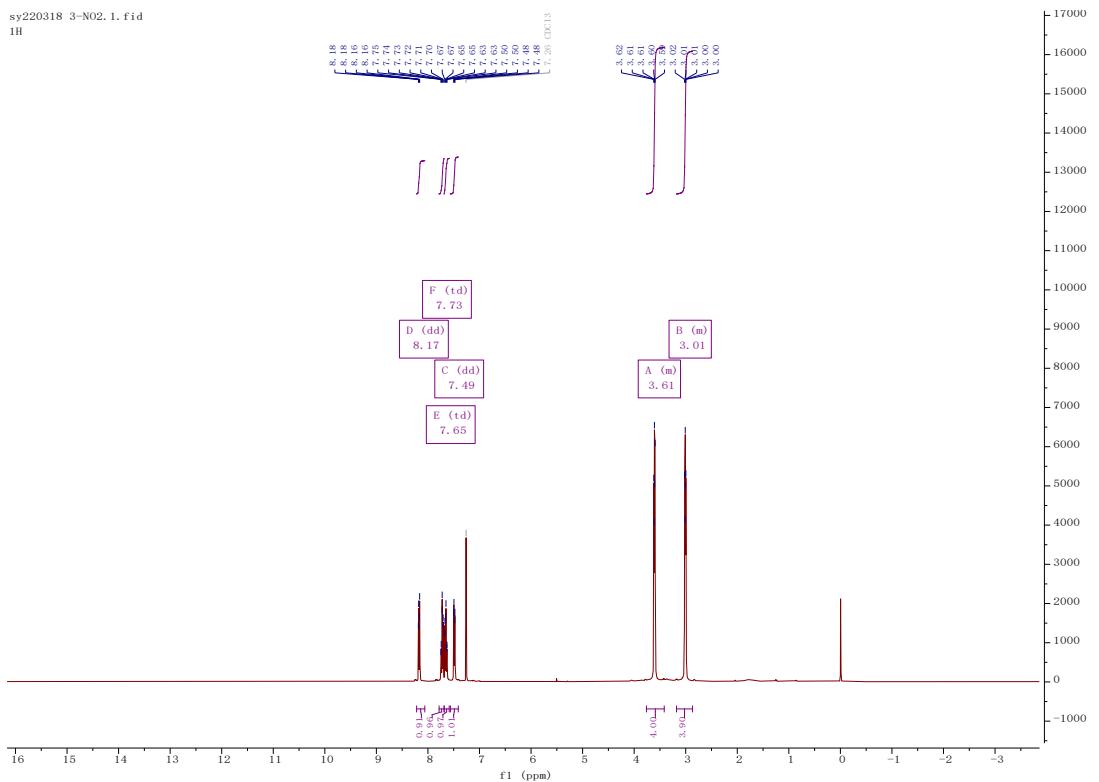
¹⁹F NMR

4-氟基, 2, fid



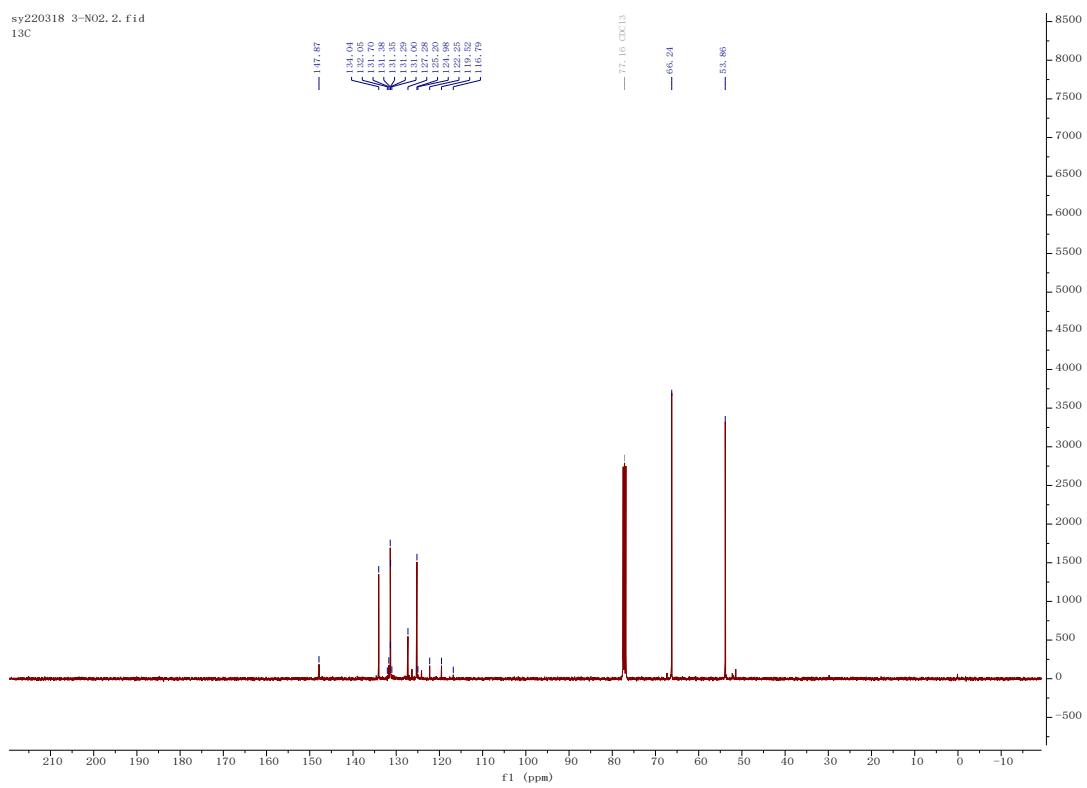
¹H NMR

sy220318 3-N02, 1, fid
1H



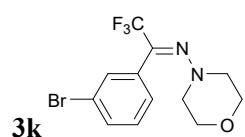
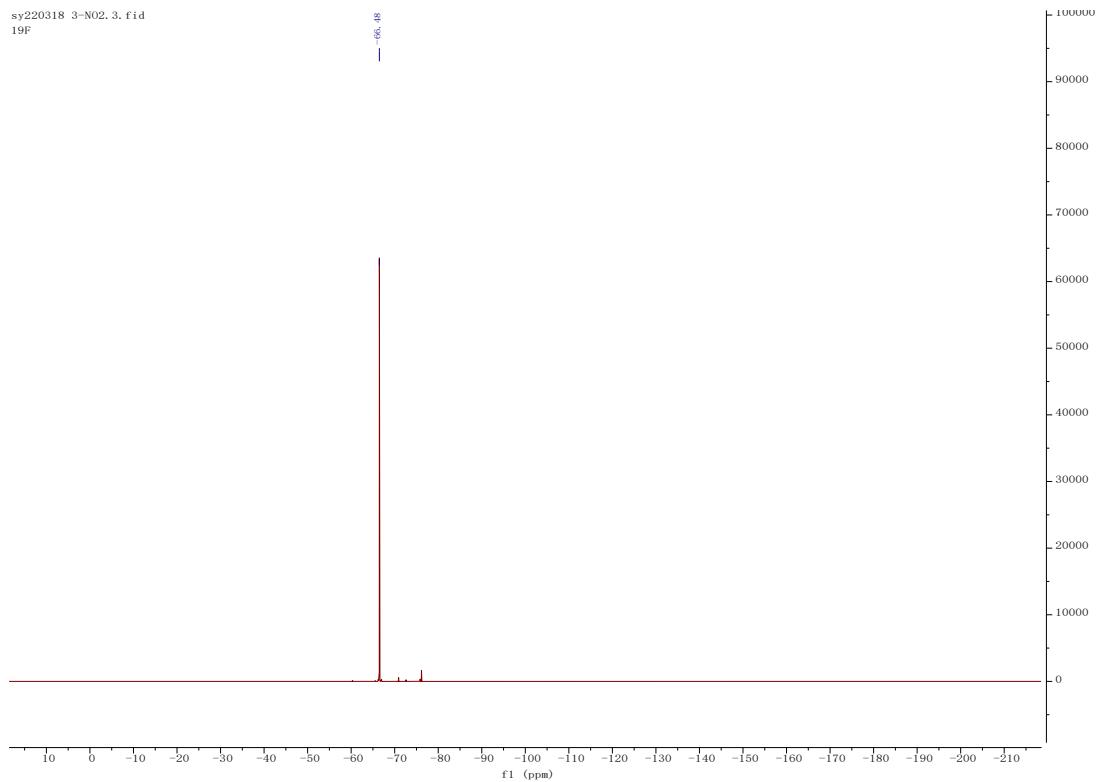
¹³C NMR

sy220318 3-N02, 2. fid
13C



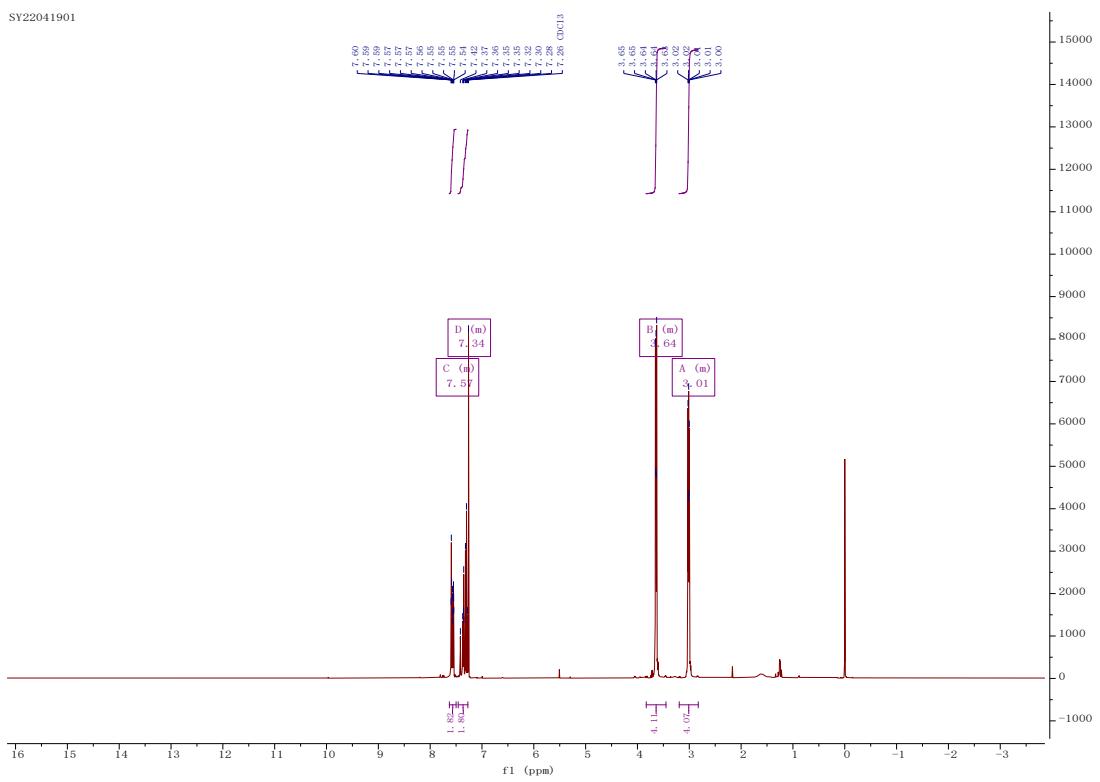
¹⁹F NMR

sy220318 3-N02, 3. fid
19F



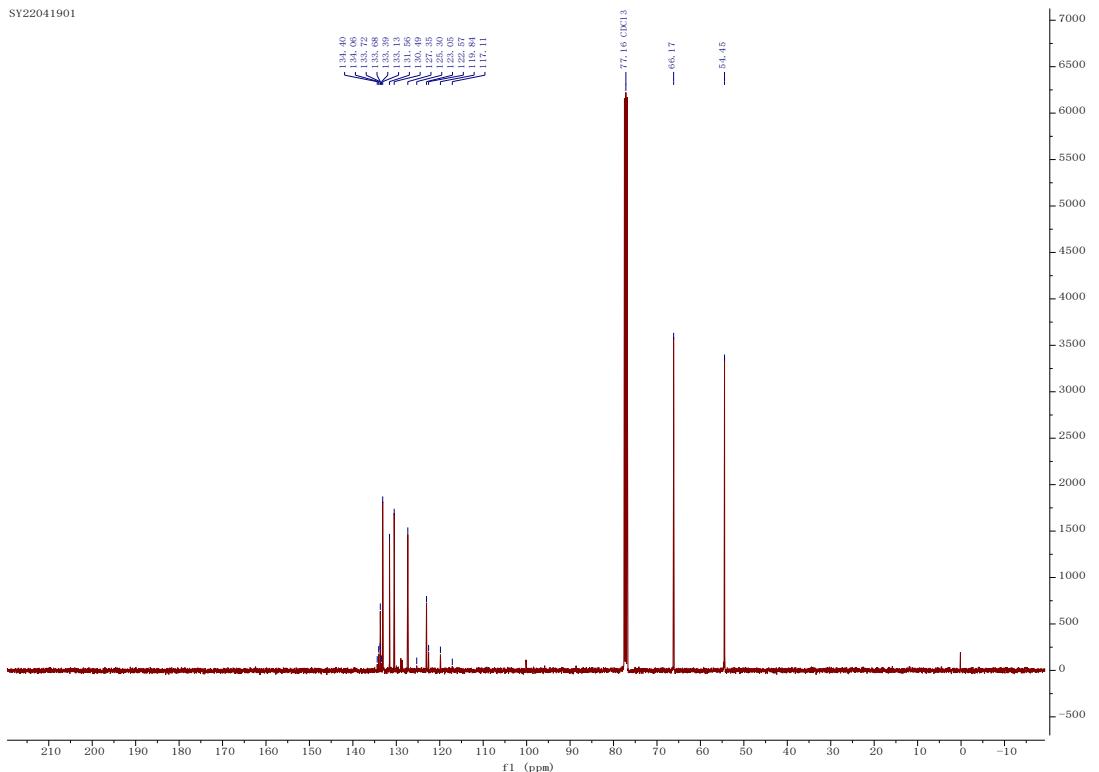
¹H NMR

SY22041901

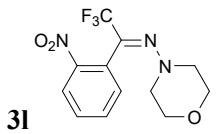
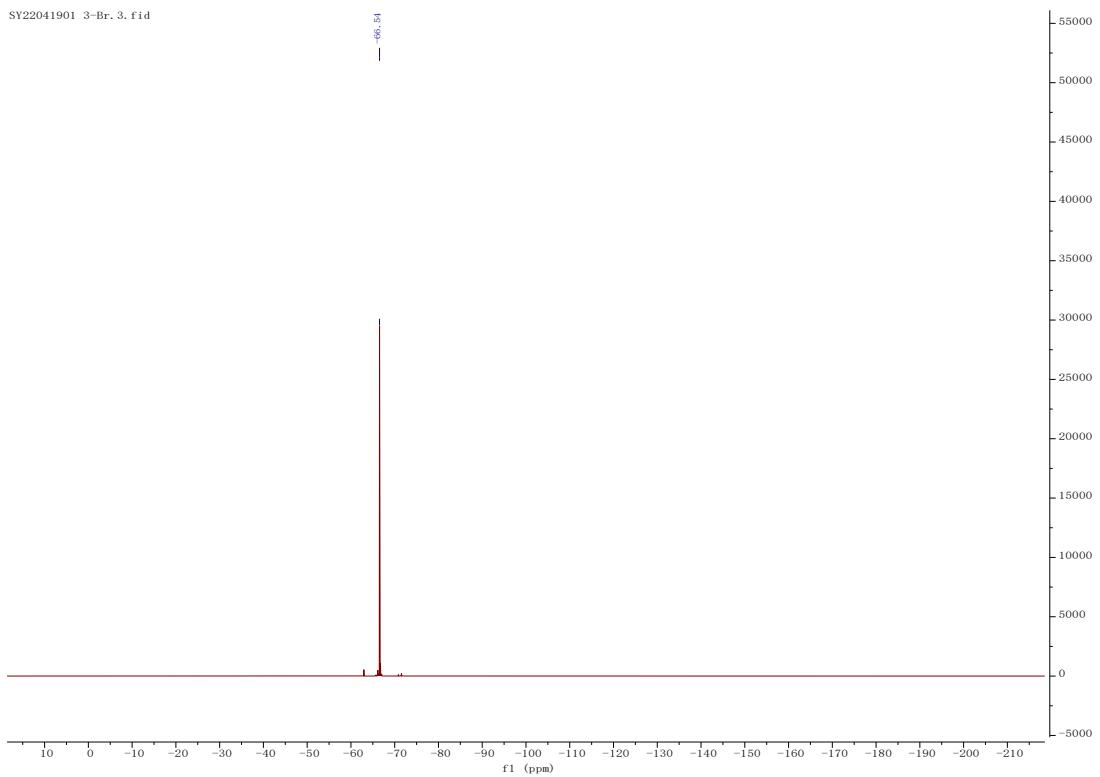


¹³C NMR

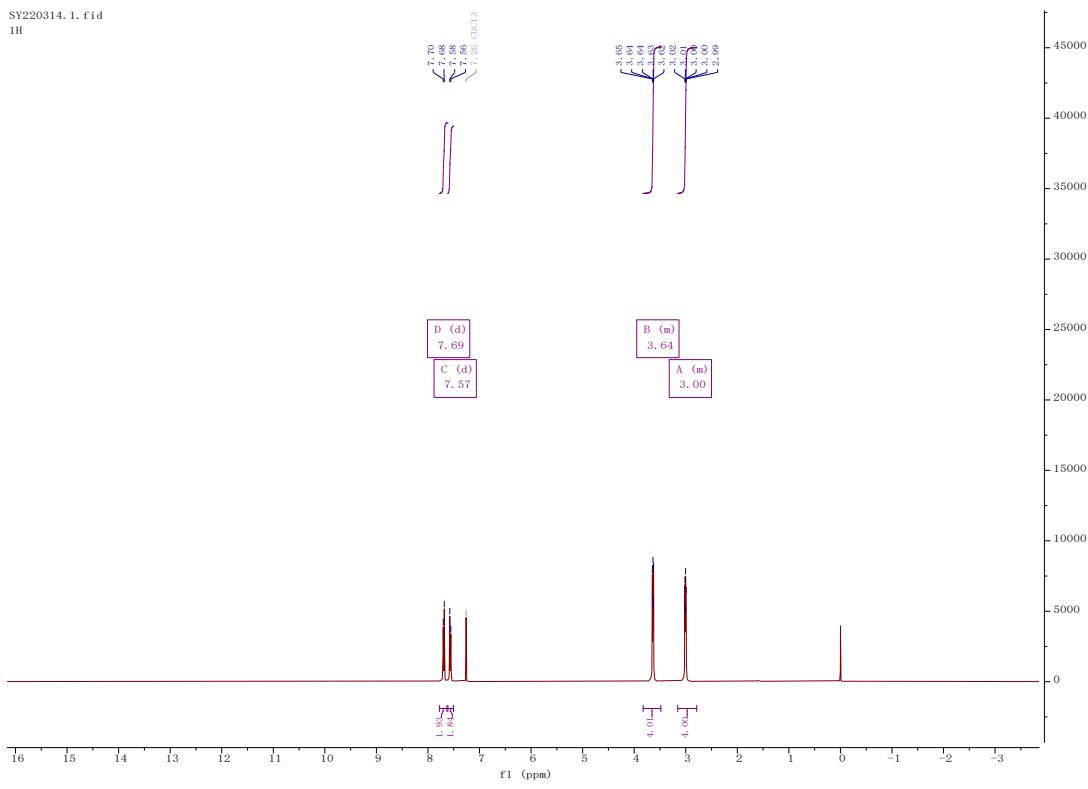
SY22041901



¹⁹F NMR

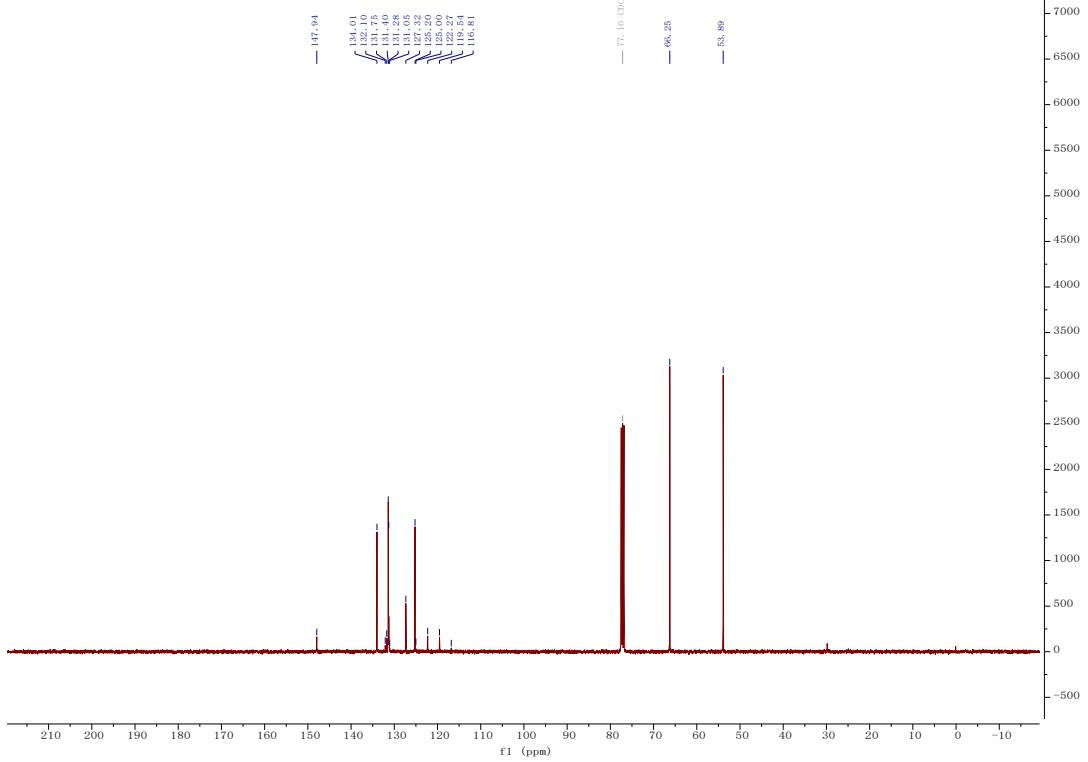


¹H NMR

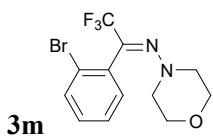
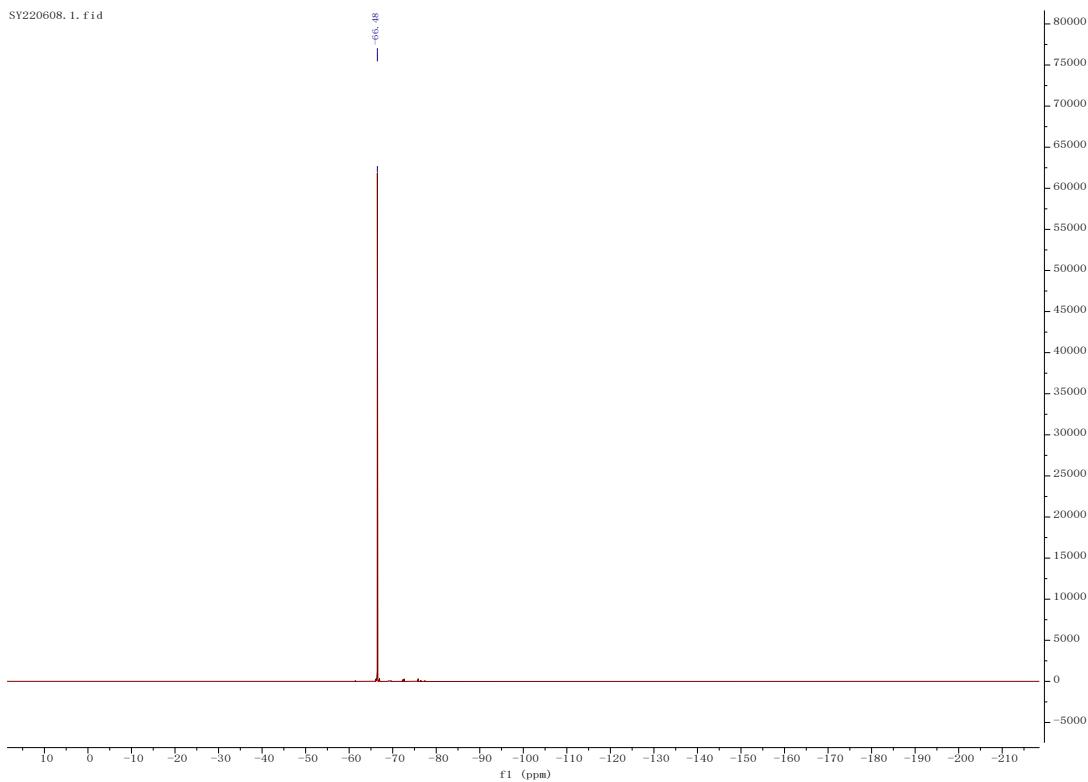


¹³C NMR

SY220608. 2. fid

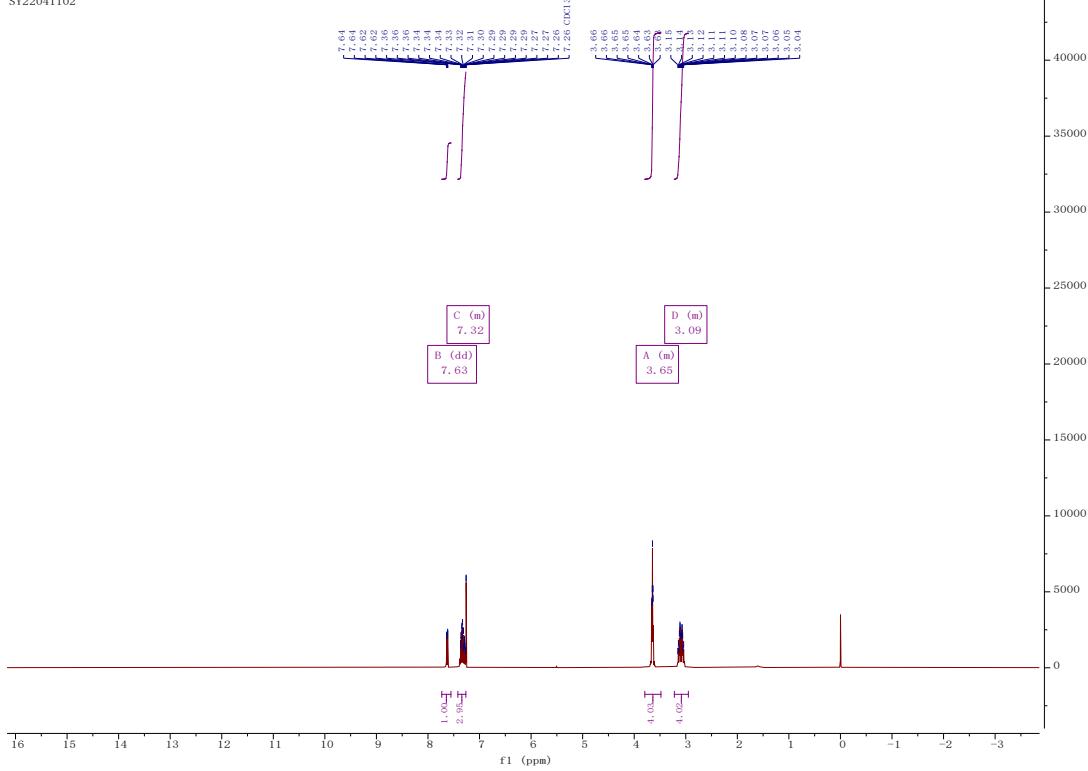


¹⁹F NMR



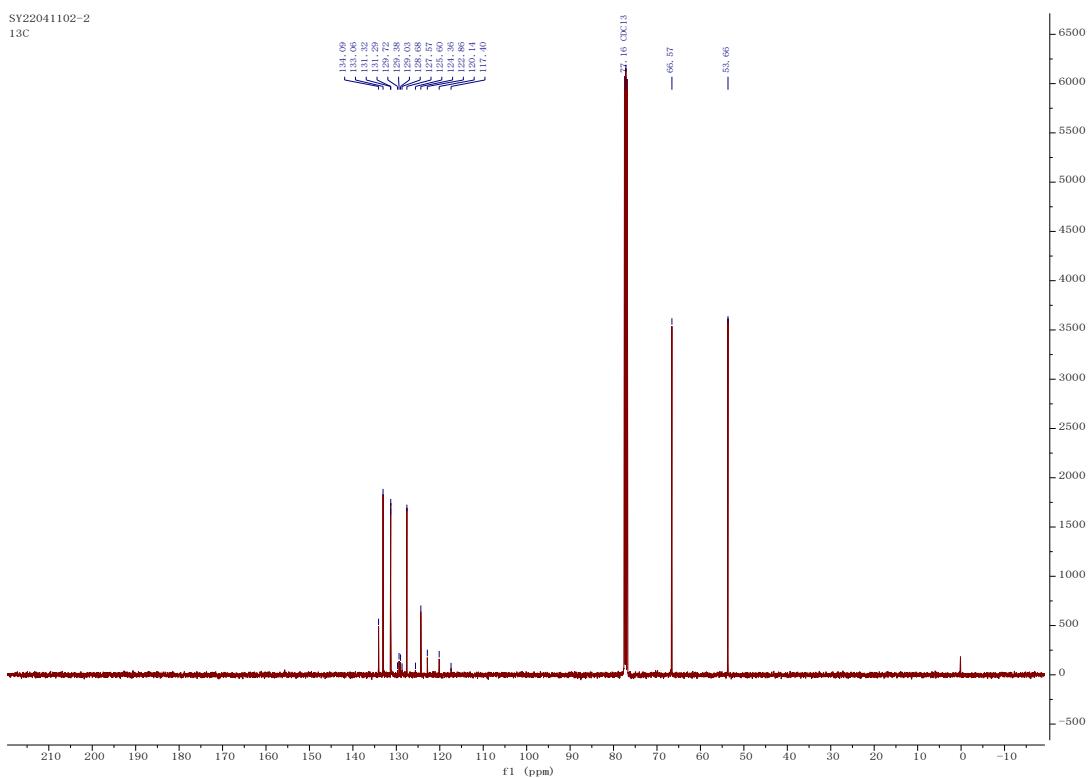
¹H NMR

SY22041102



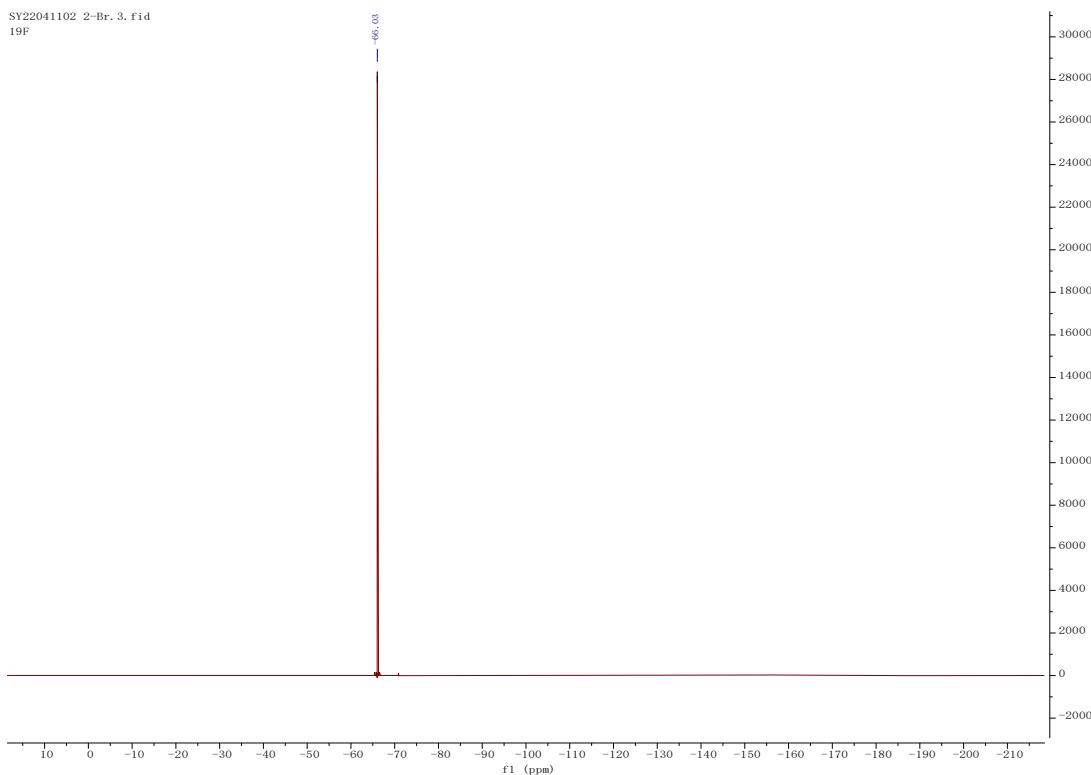
¹³C NMR

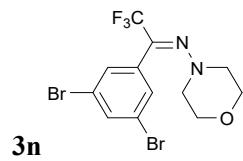
SY22041102-2
13C



¹⁹F NMR

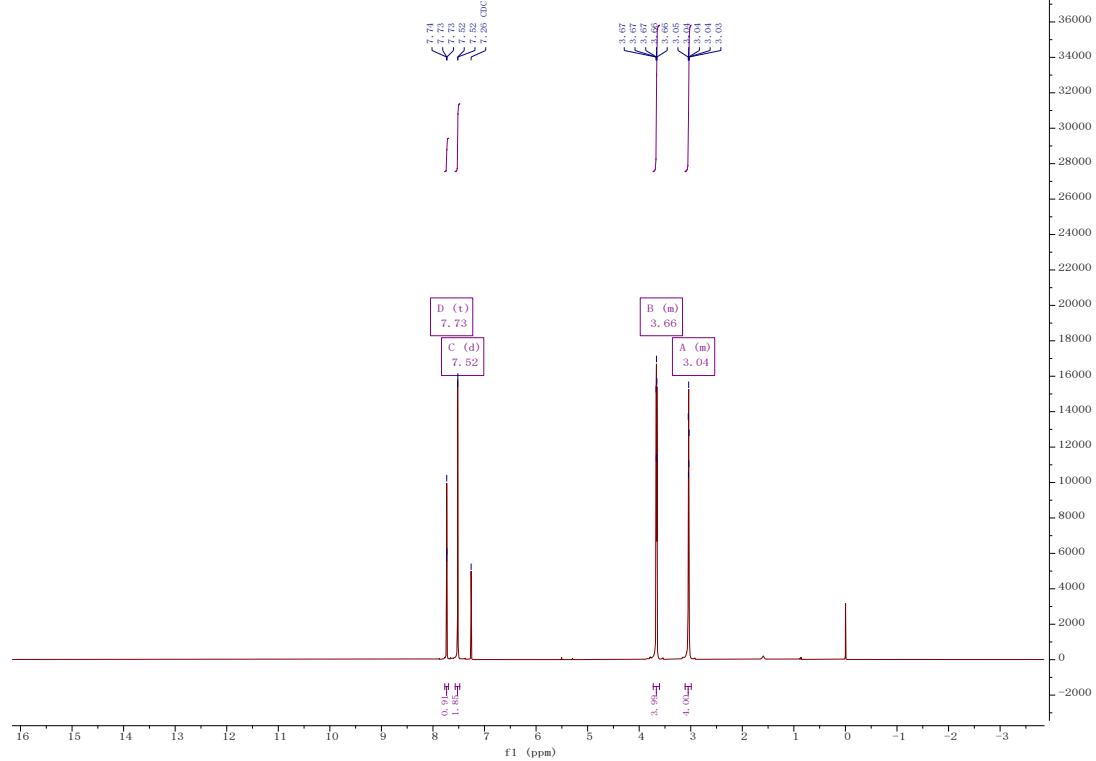
SY22041102 2-Br. 3. fid
19F





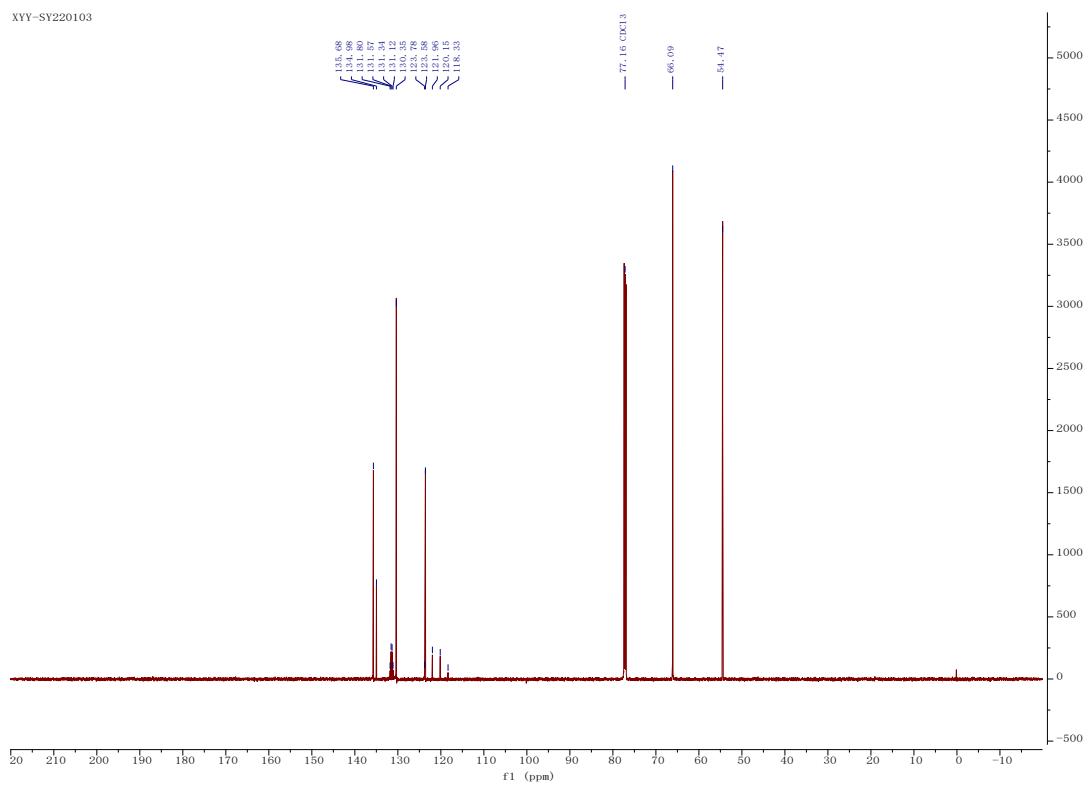
¹H NMR

XYY-SY220103



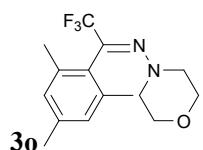
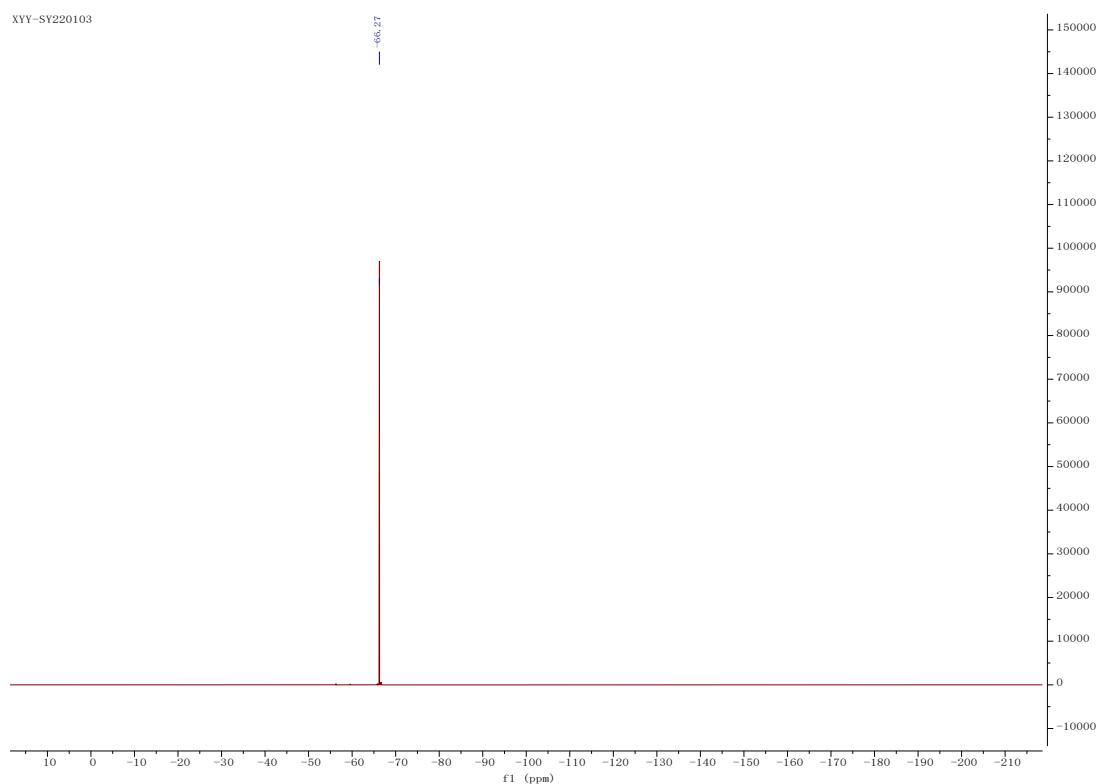
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XYX-SY220103



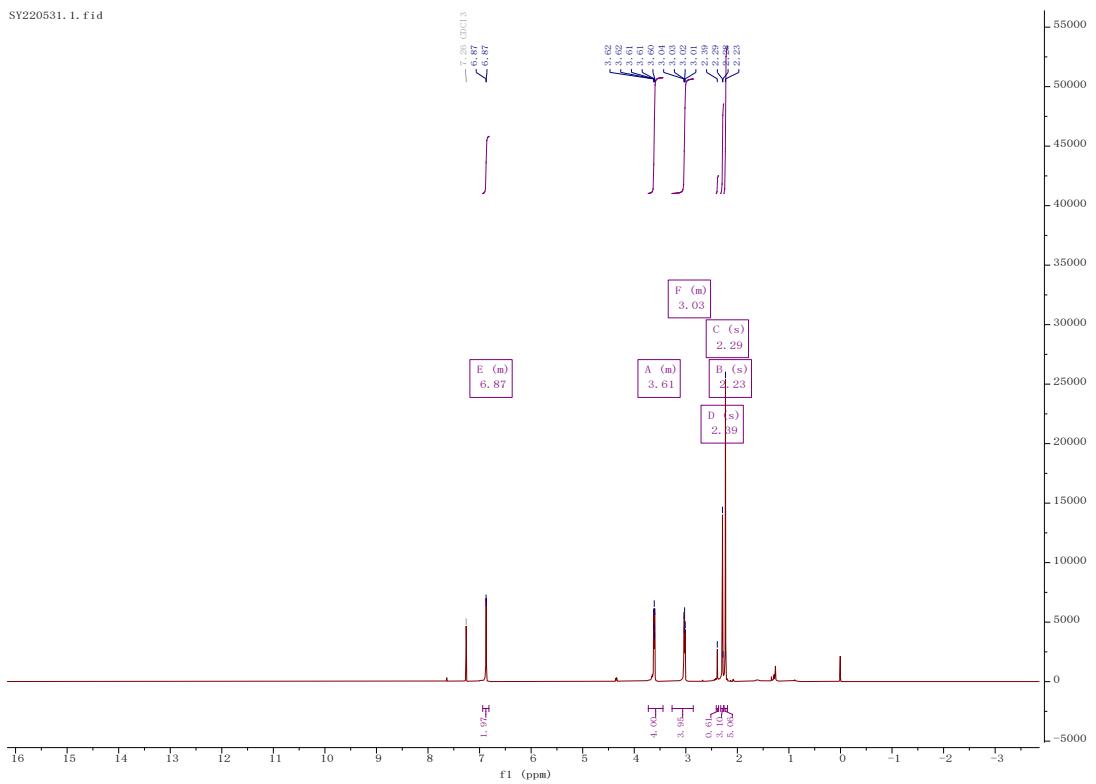
¹⁹F NMR

XYX-SY220103



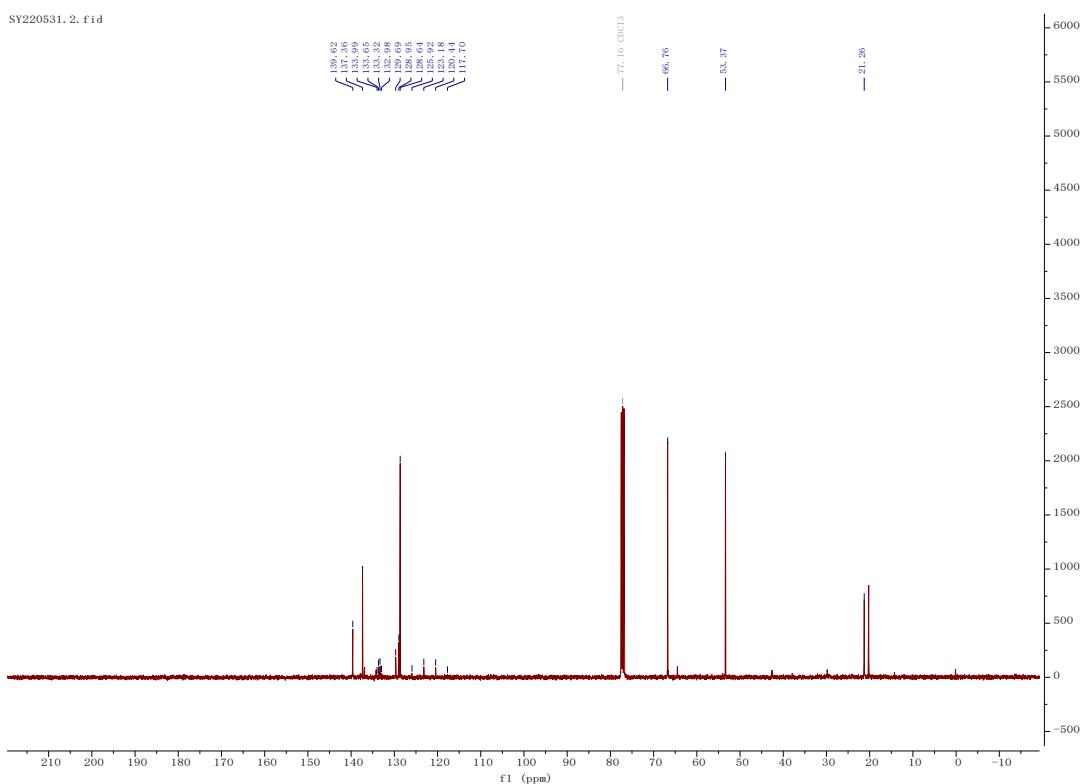
¹H NMR

SY220531. 1. fid



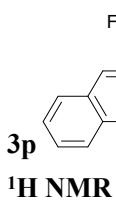
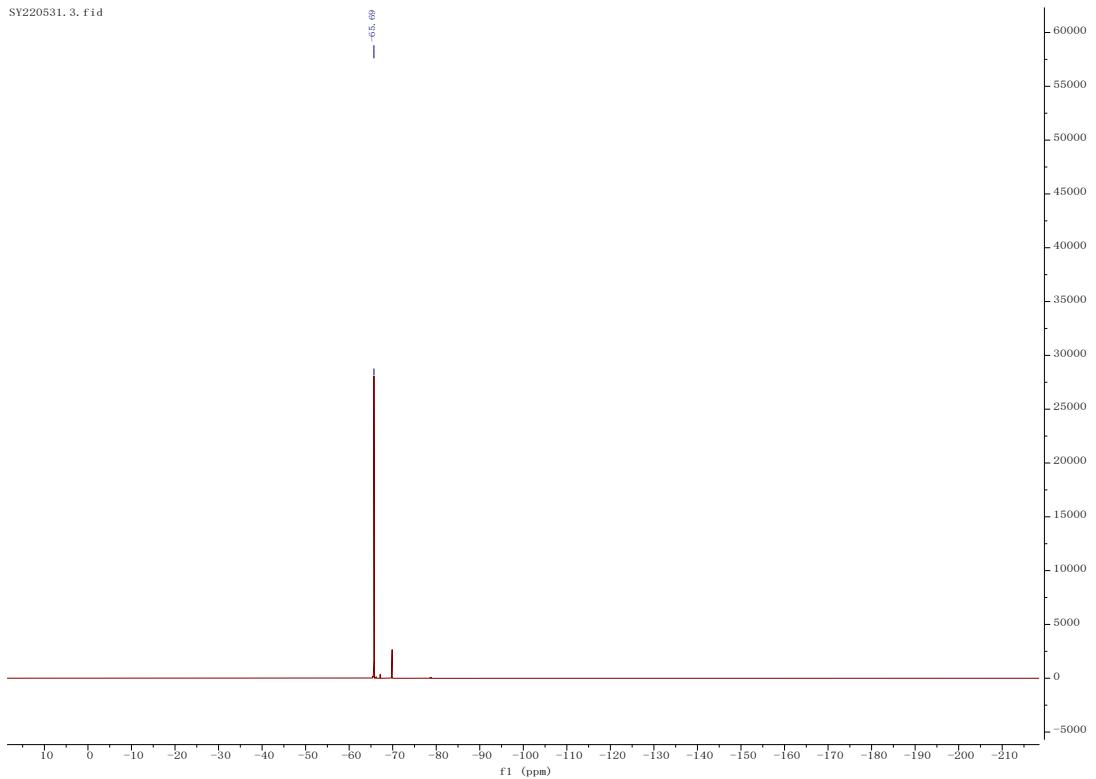
^{13}C NMR

SY220531. 2. fid



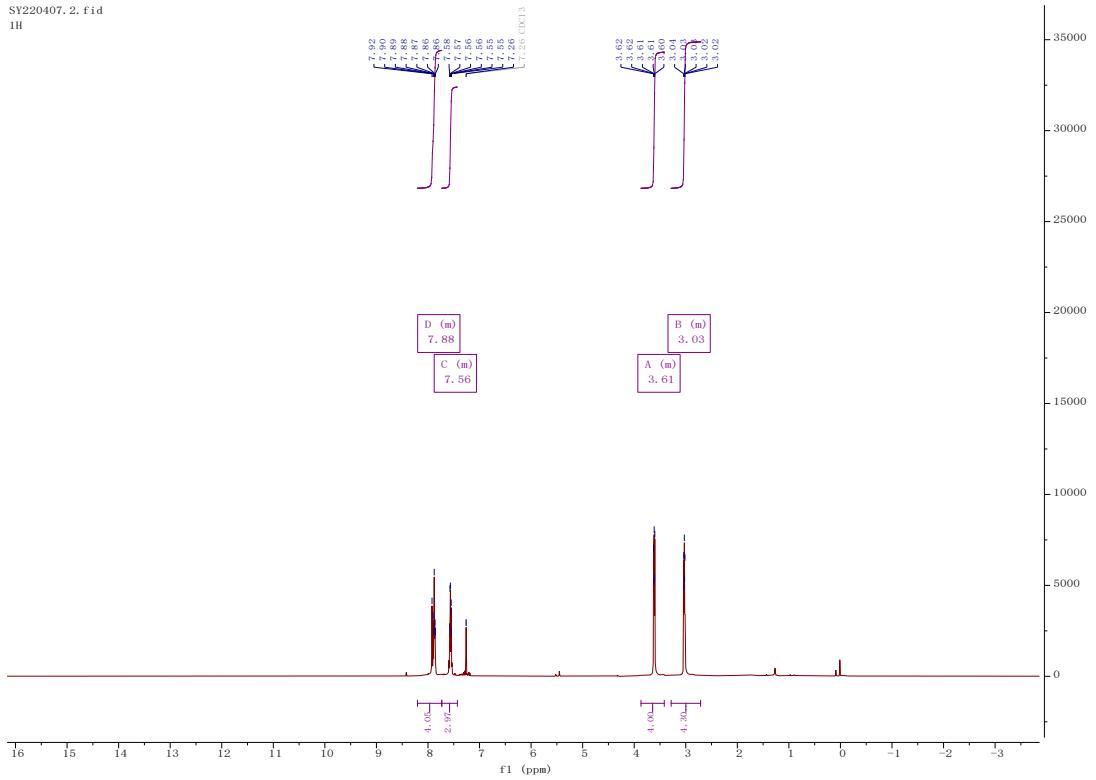
^{19}F NMR

SY220531. 3. fid



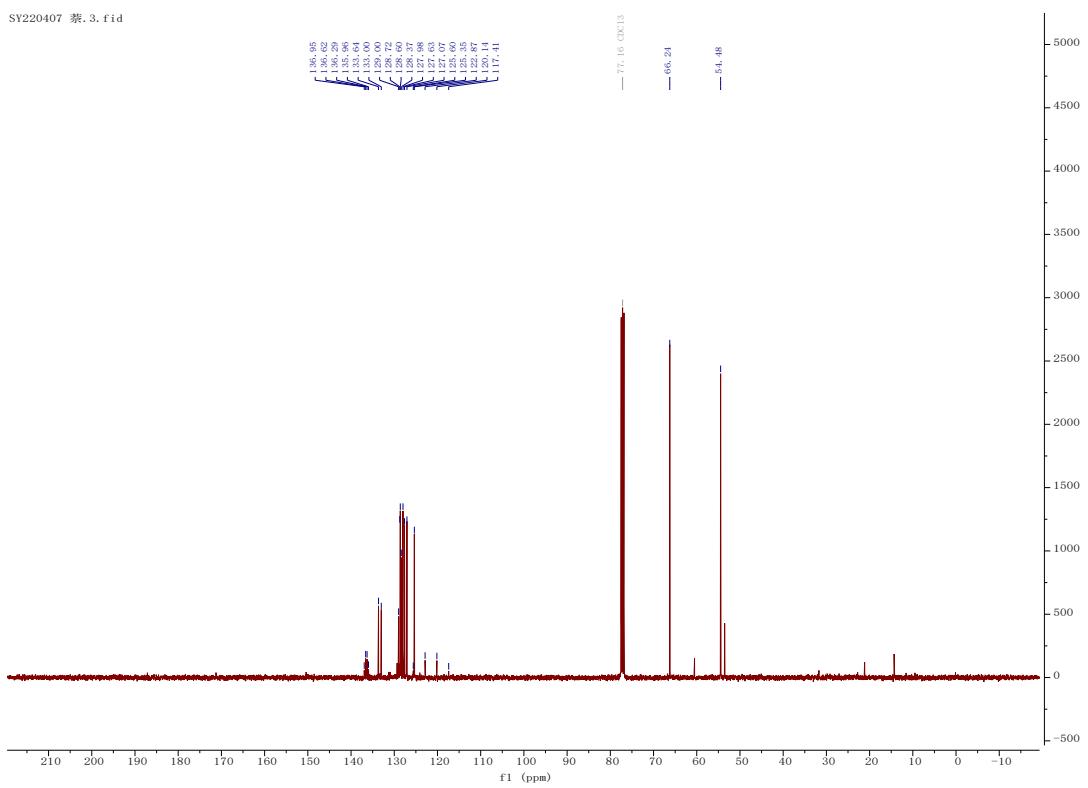
¹H NMR

SY220407. 2. fid
1H



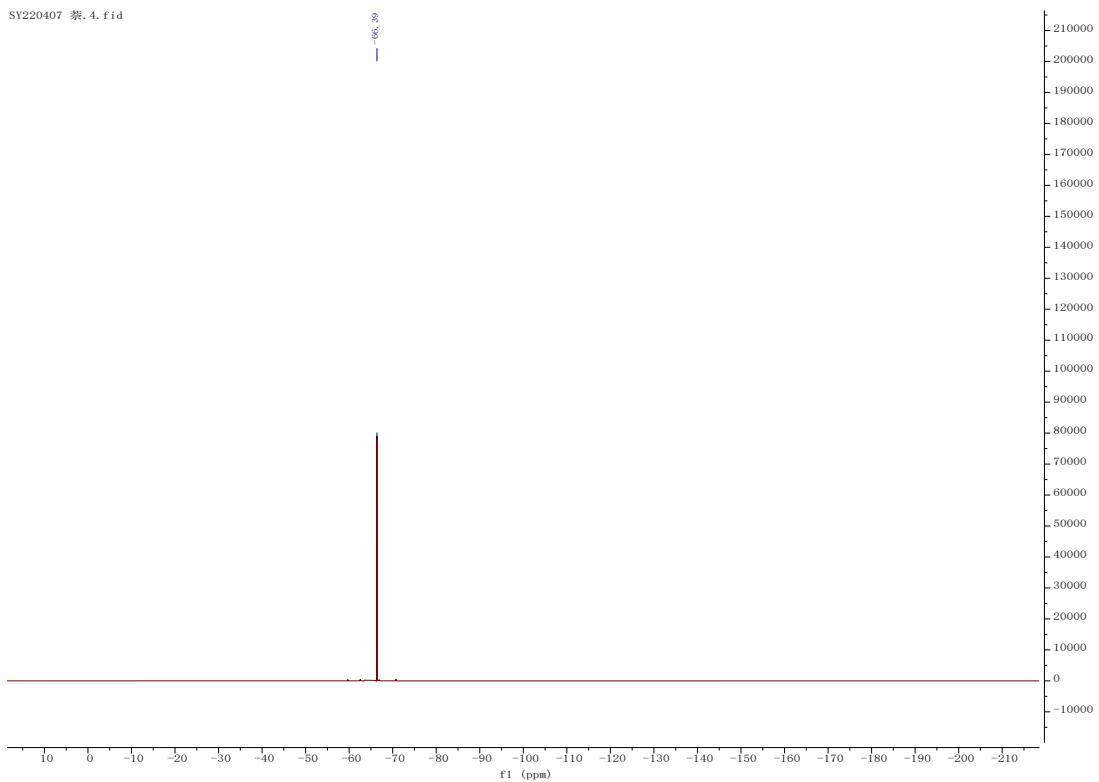
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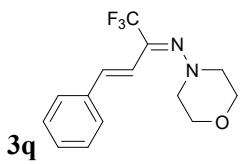
SY220407 蔡. 3. fid



¹⁹F NMR

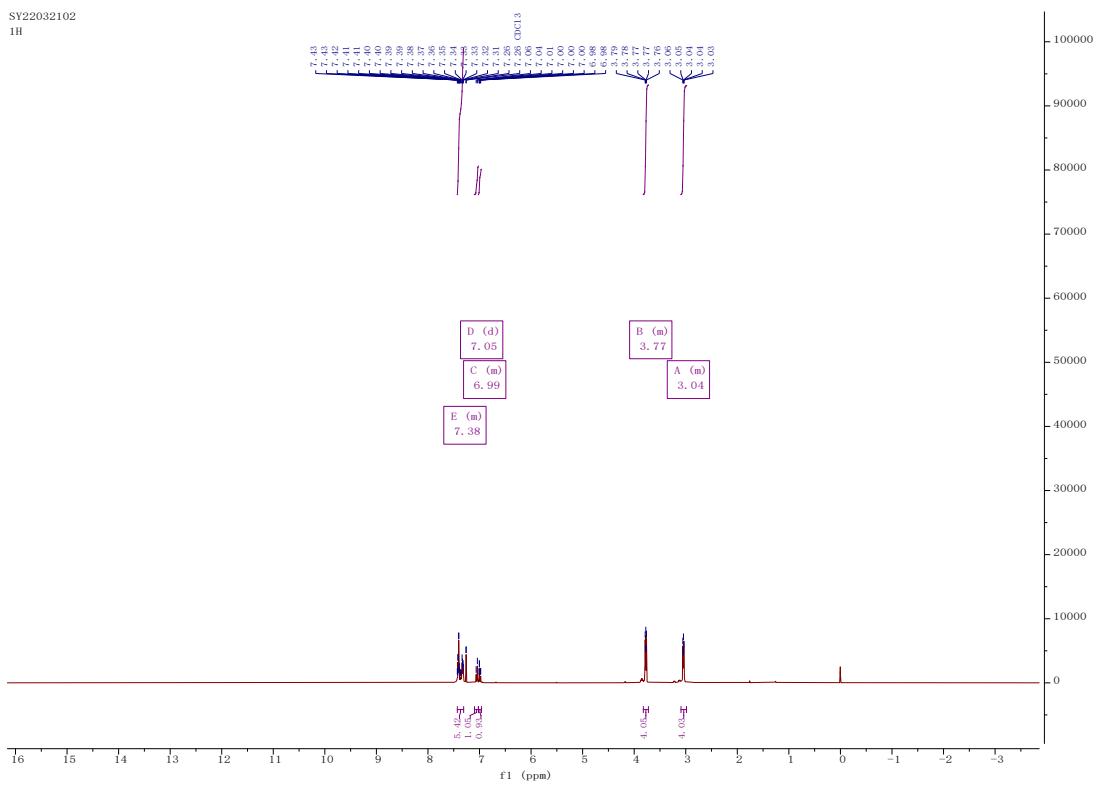
SY220407 蔡. 4. fid





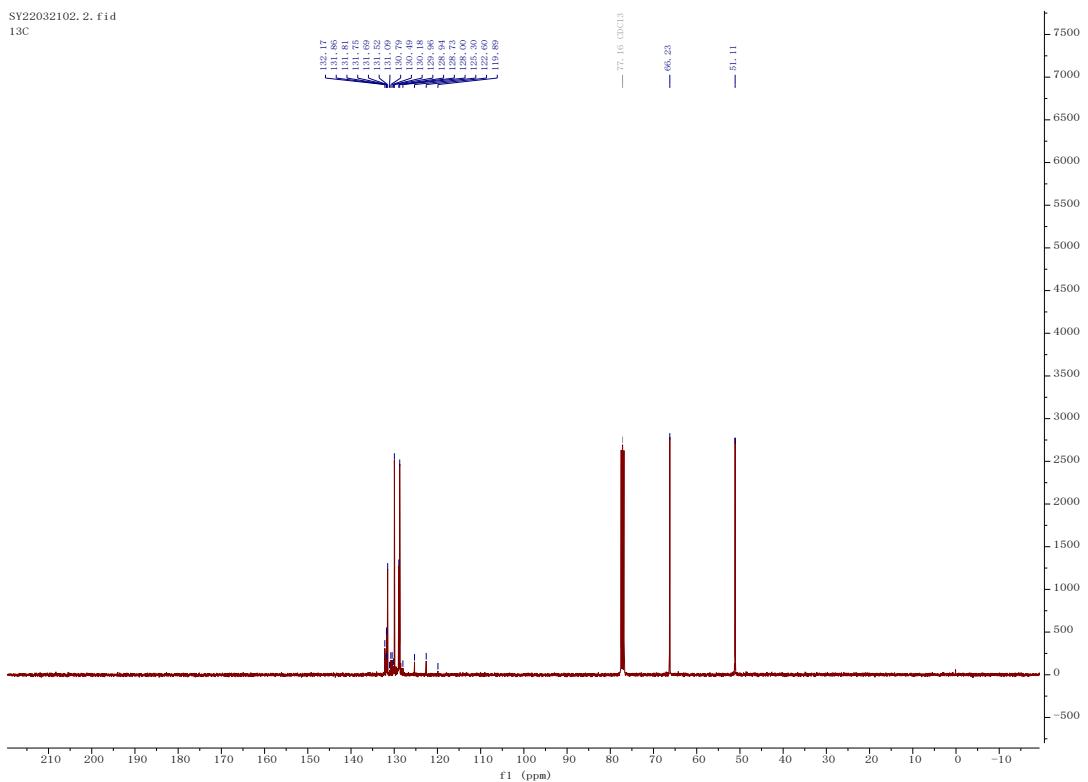
¹H NMR

SY22032102
1H



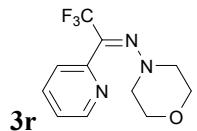
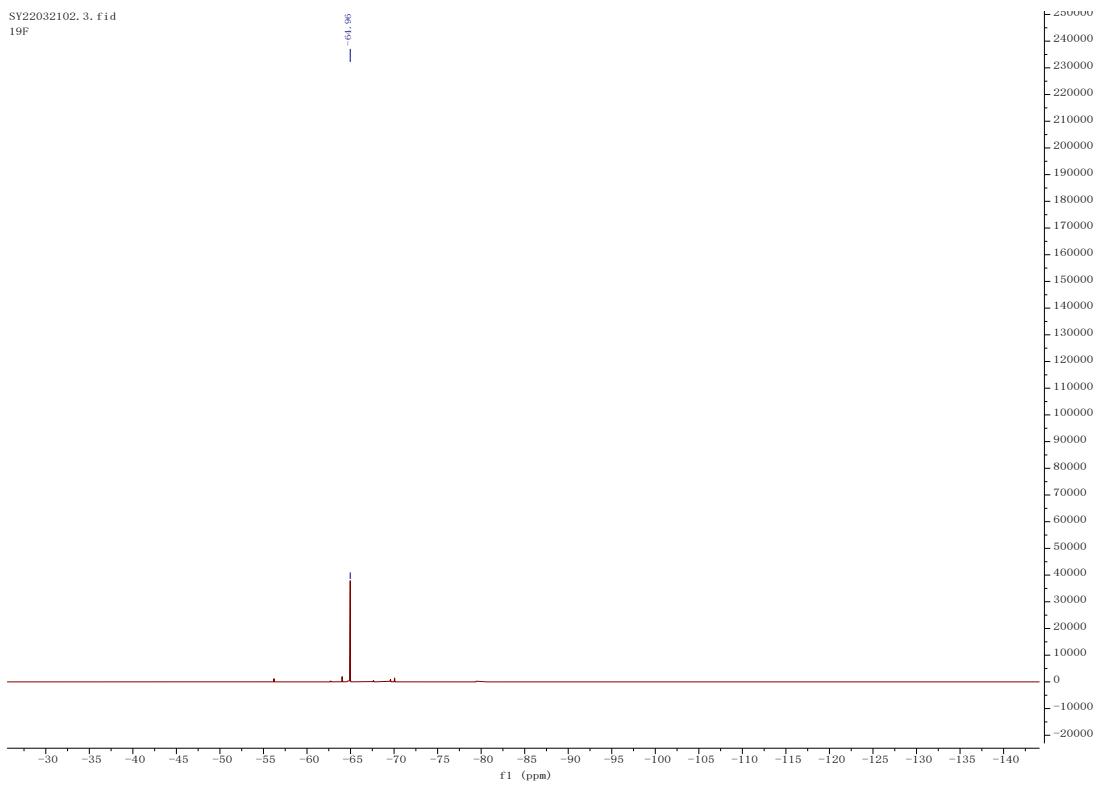
¹³C NMR

SY22032102, 2, fid
13C



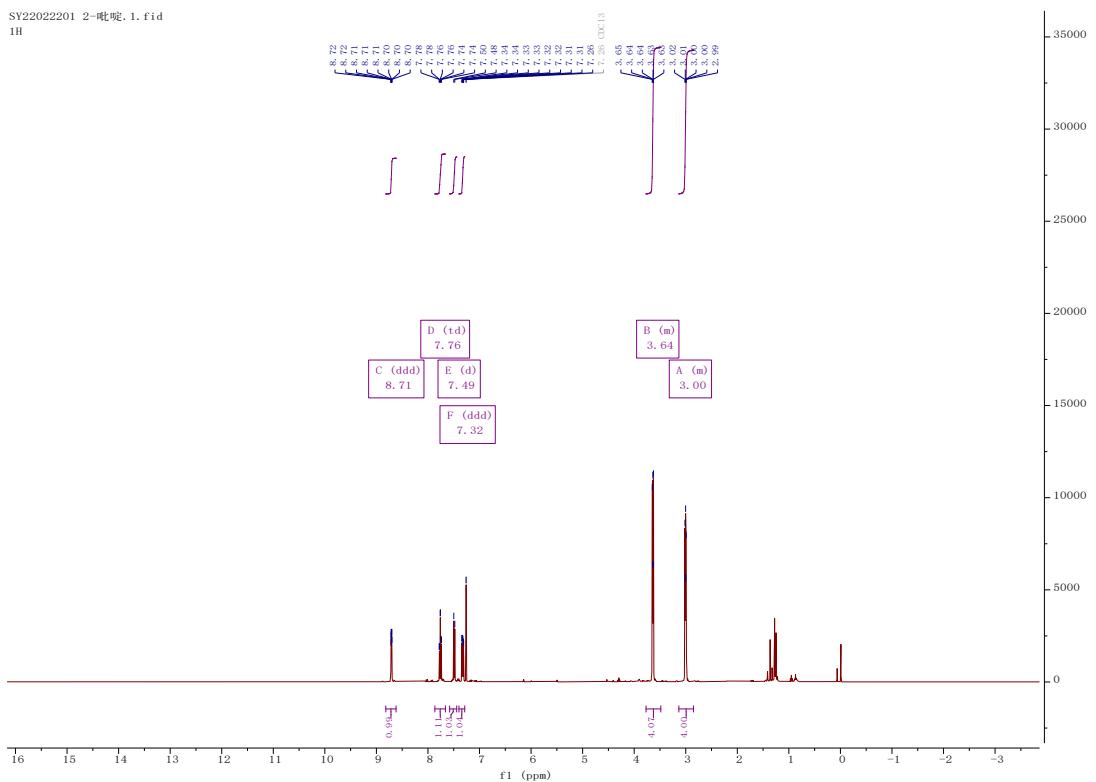
¹⁹F NMR

SY22032102, 3, fid
19F

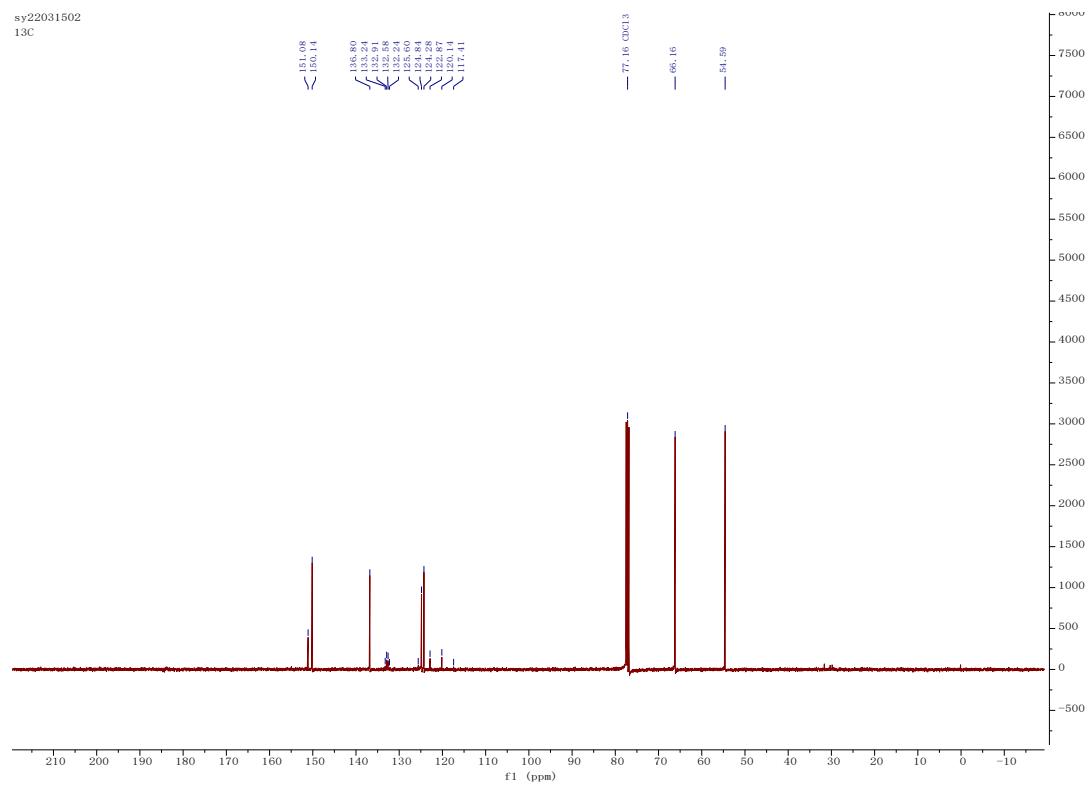


¹H NMR

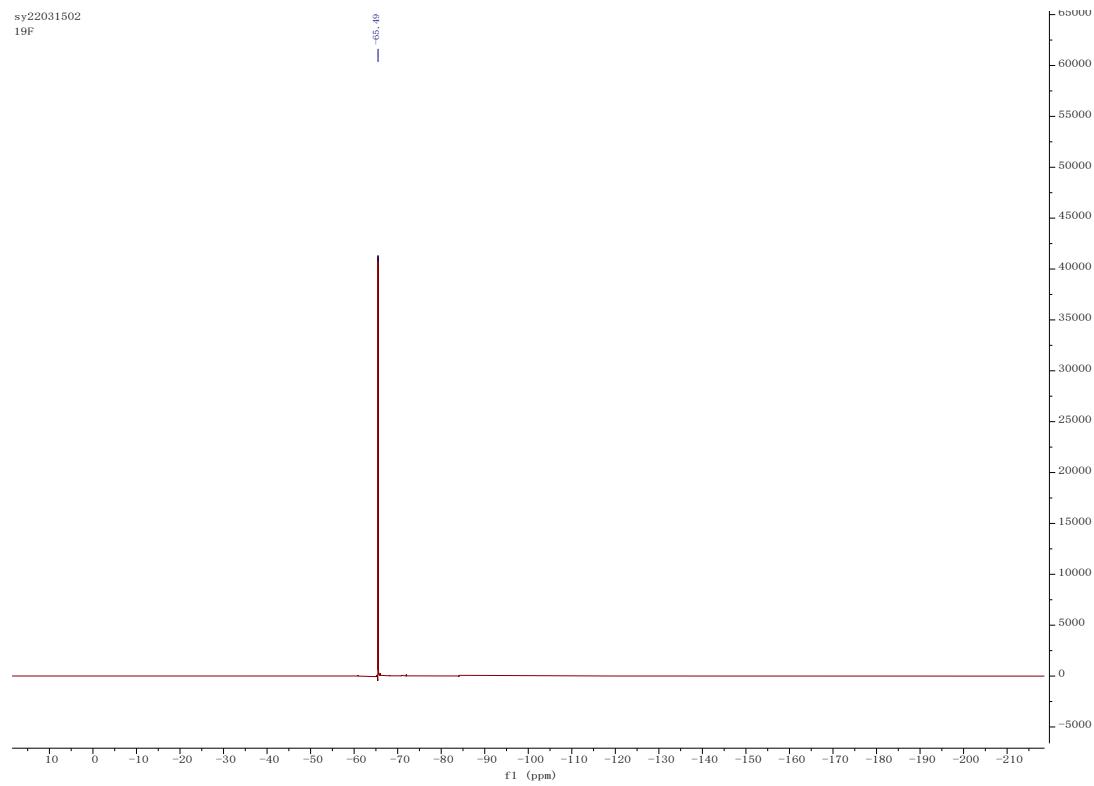
SY22022201 2-吡啶, 1, fid
1H

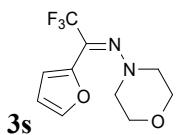


¹³C NMR



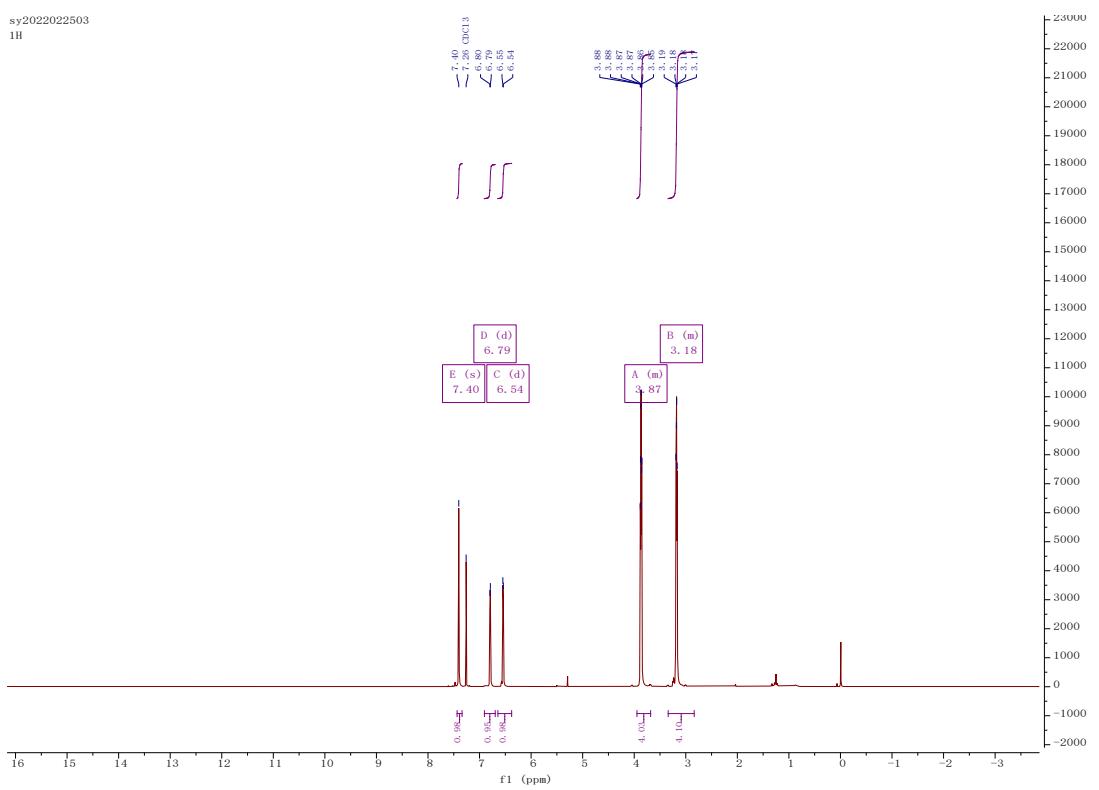
¹⁹F NMR





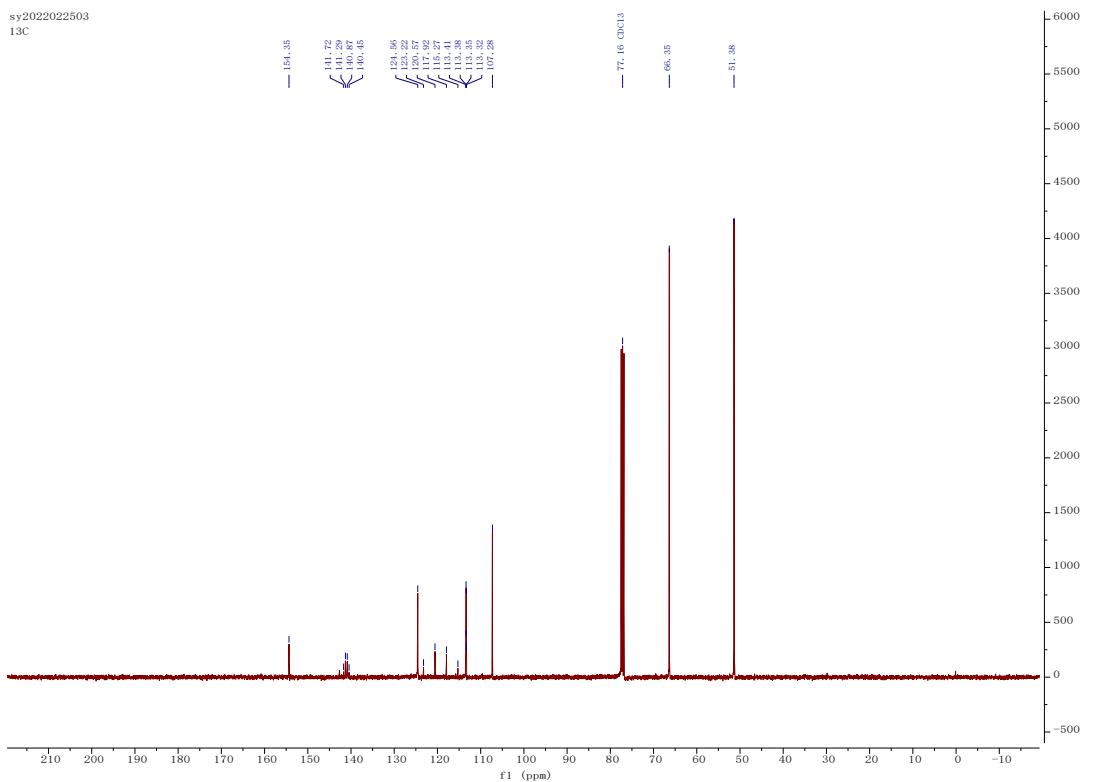
¹H NMR

sy2022022503
1H



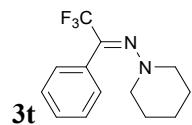
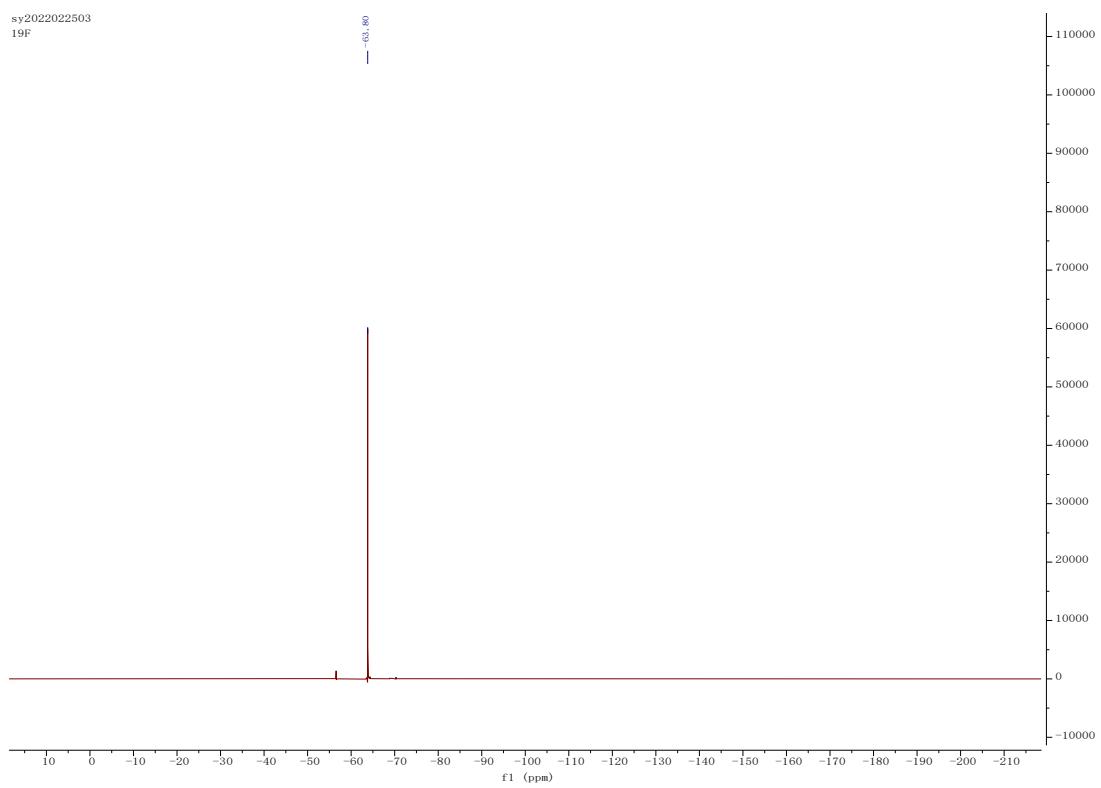
¹³C NMR

sy2022022503
13C



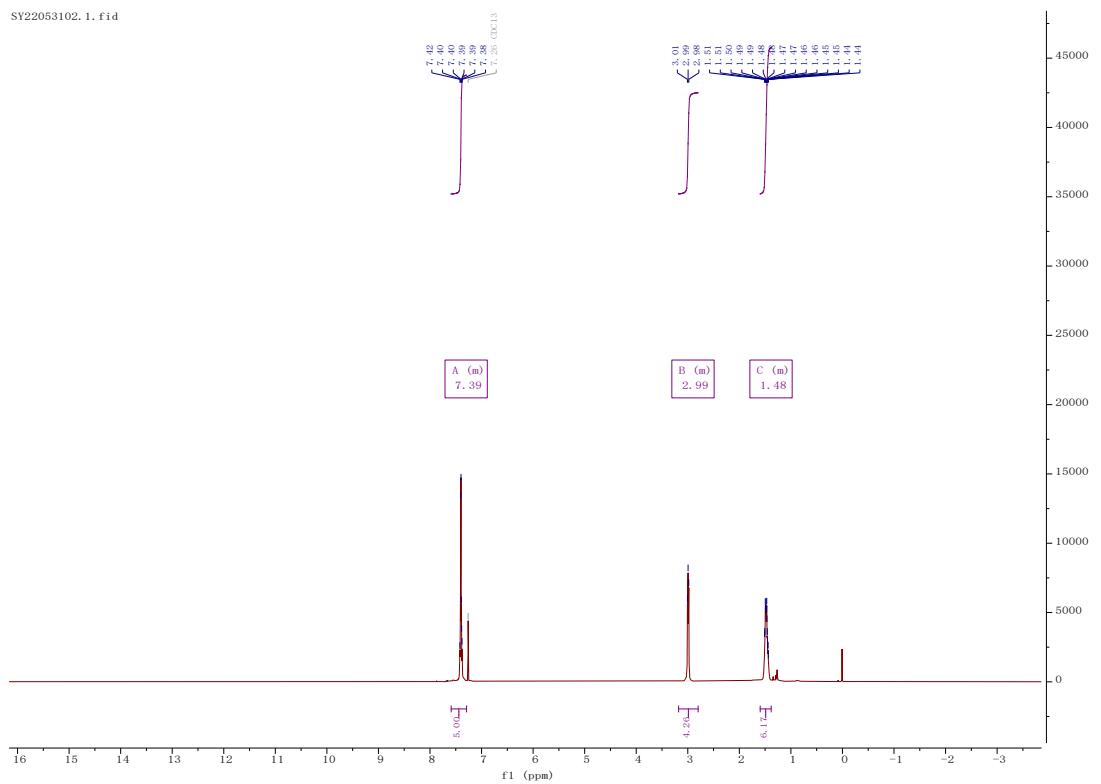
¹⁹F NMR

sy2022022503
19F



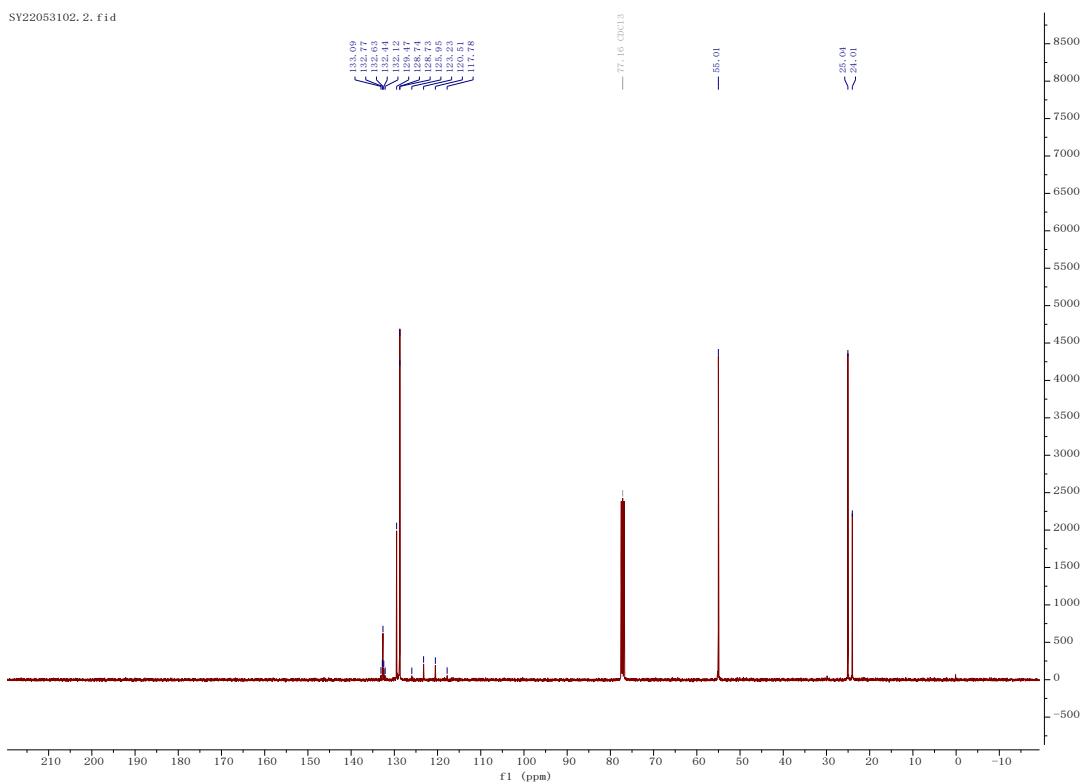
¹H NMR

SY22053102, 1, fid



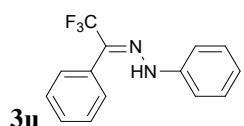
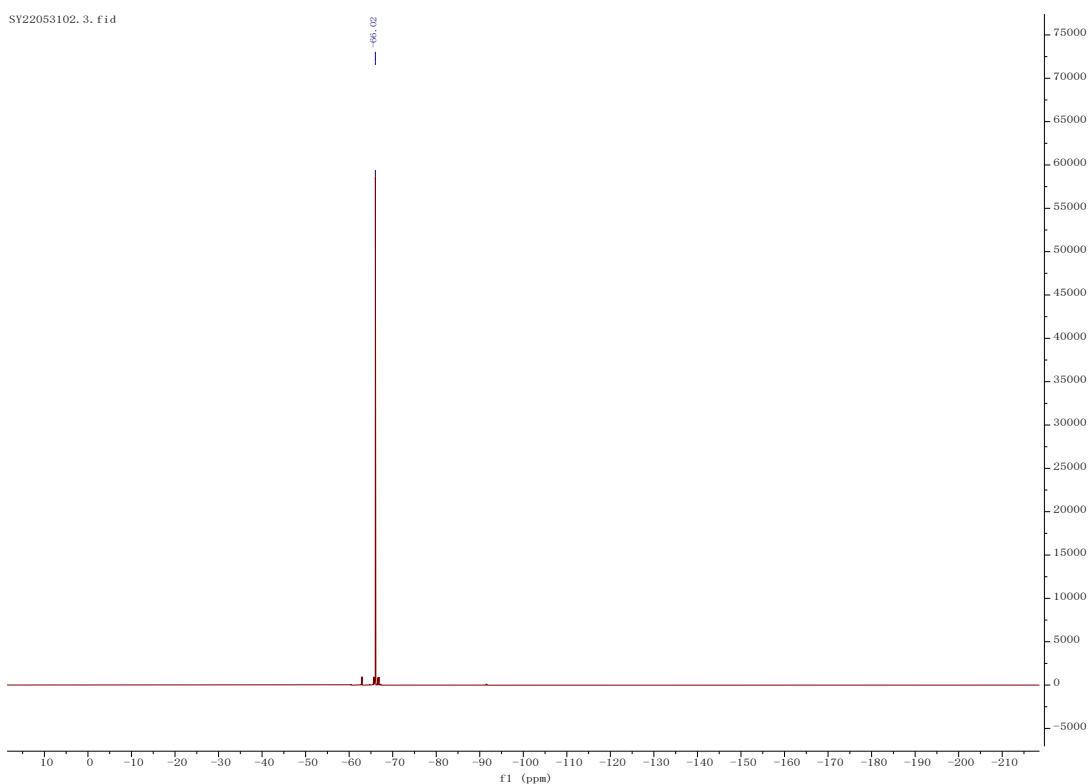
¹³C NMR

SY22053102. 2. fid



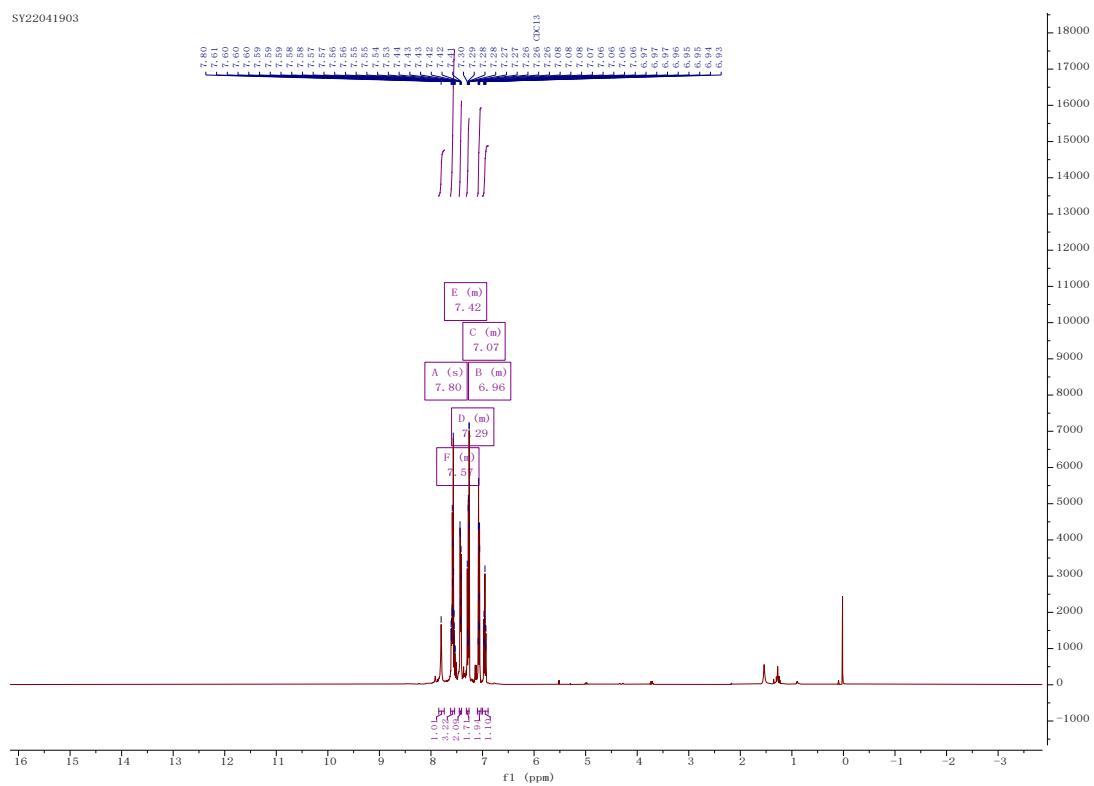
¹⁹F NMR

SY22053102. 3. fid



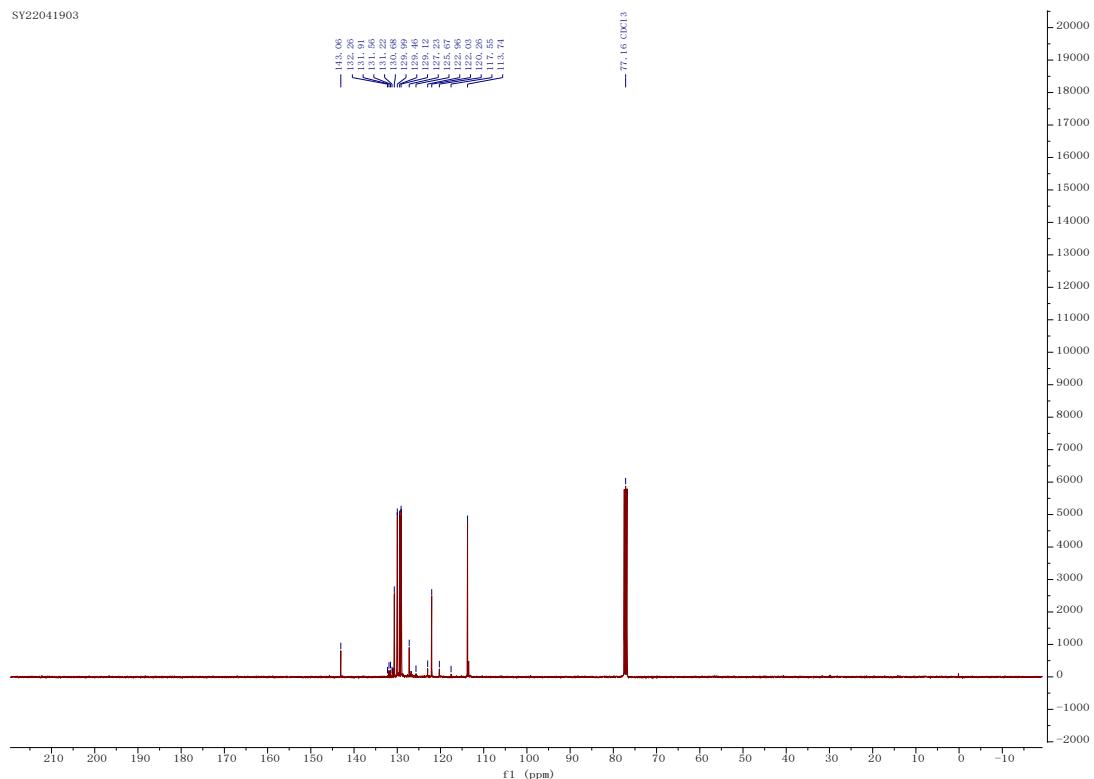
¹H NMR

SY22041903



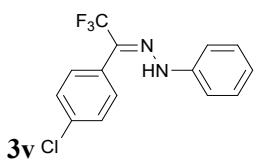
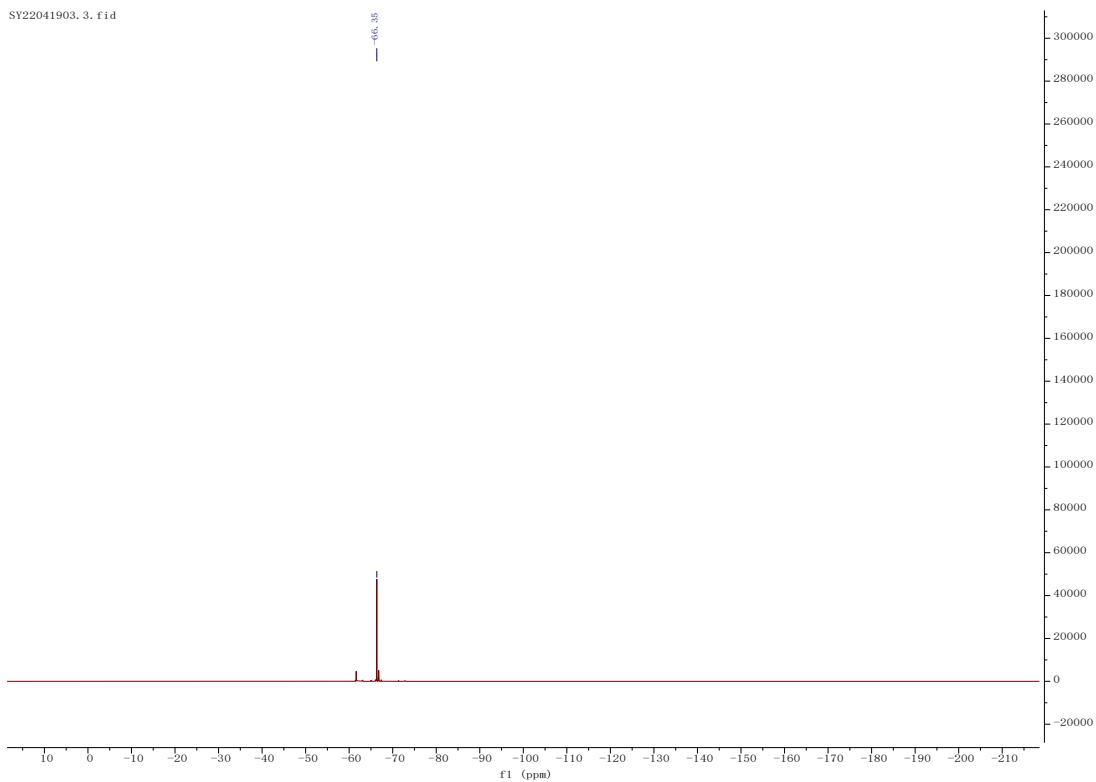
¹³C NMR

SY22041903



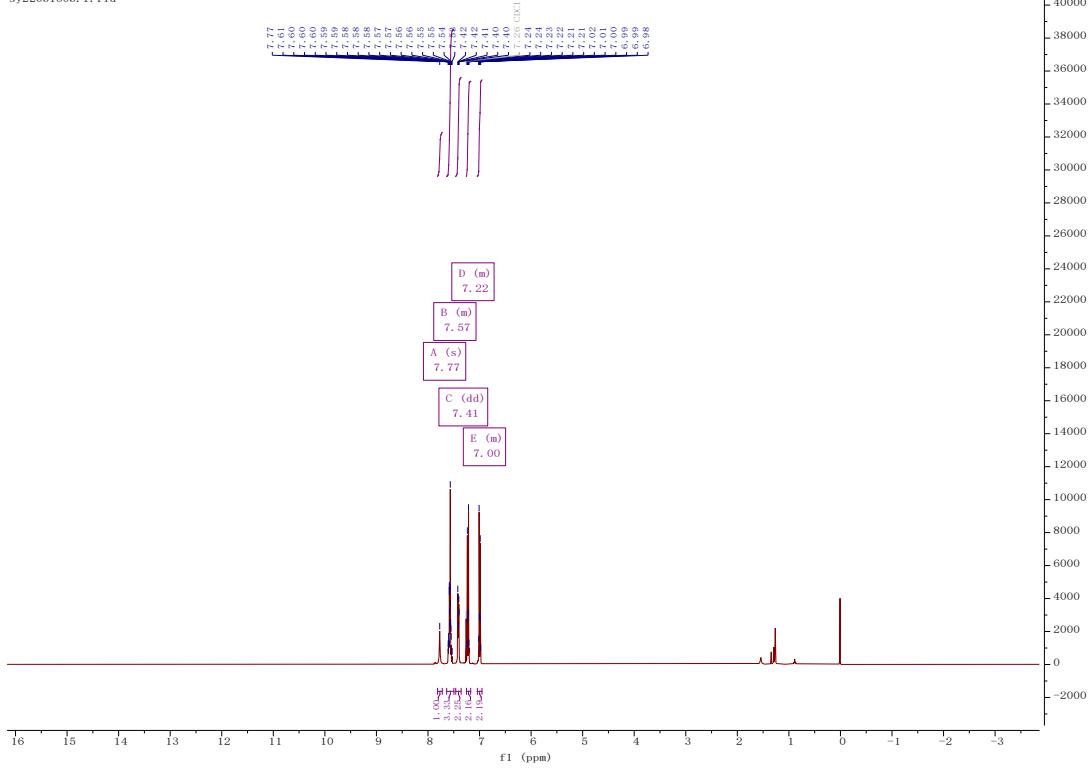
¹⁹F NMR

SY22041903, 3, fid



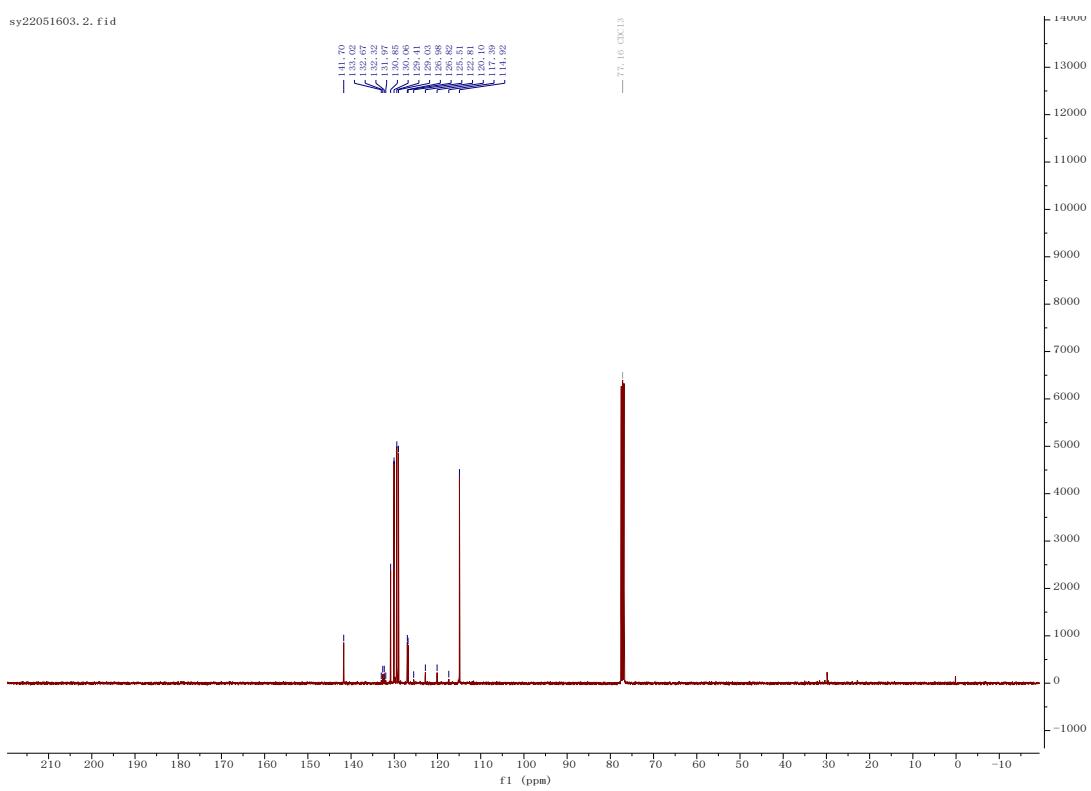
¹H NMR

sy22051603, 1, fid



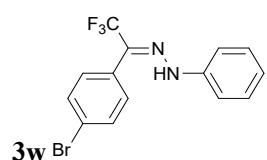
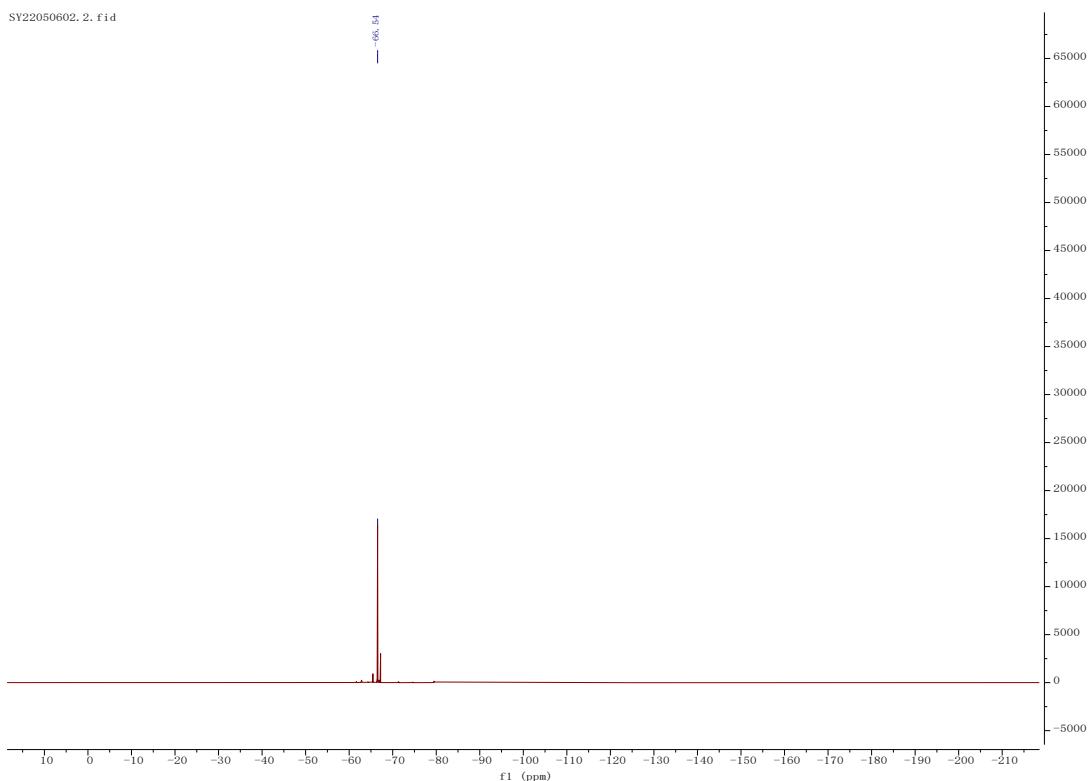
¹³C NMR

SY22051603, 2, fid



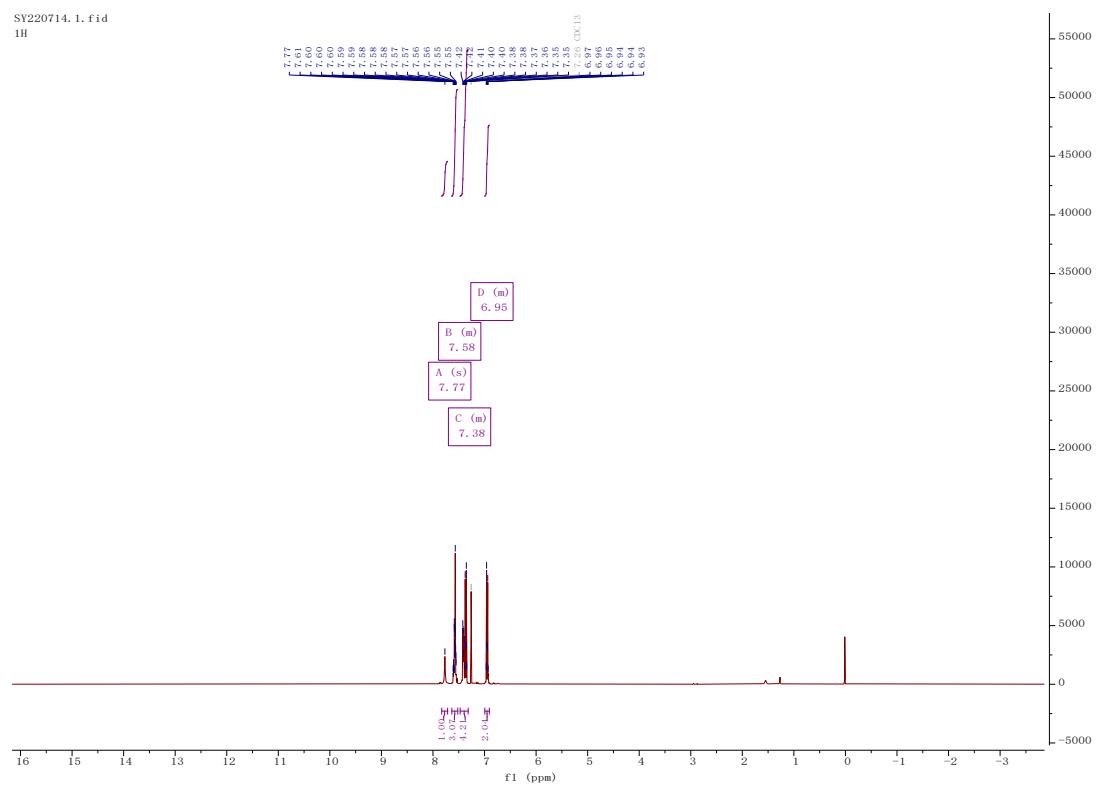
¹⁹F NMR

SY22050602, 2, fid



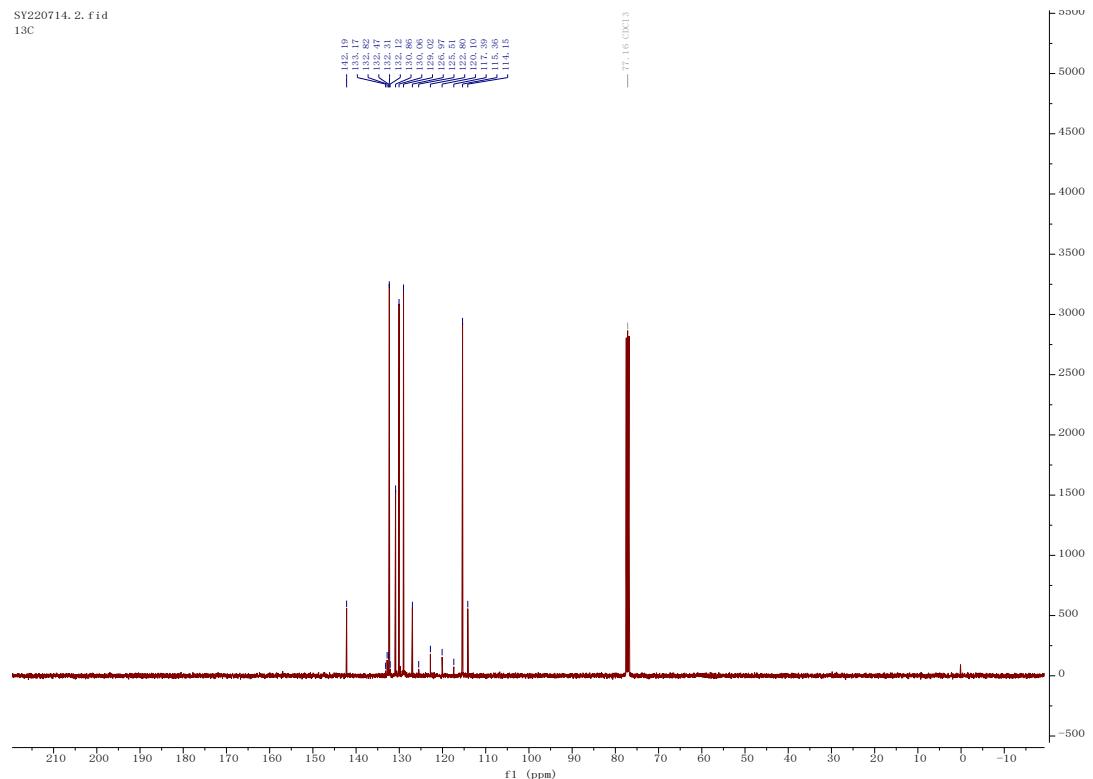
¹H NMR

SY220714. 1. fid
1H



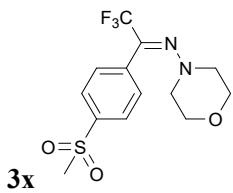
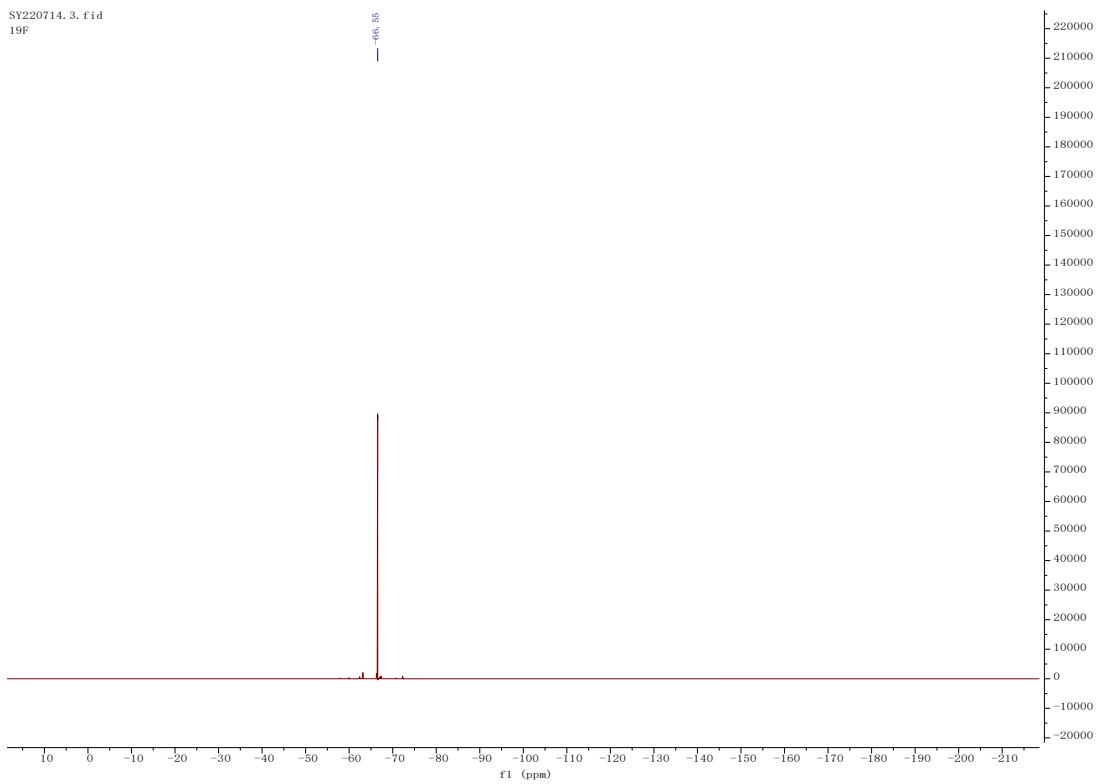
¹³C NMR

SY220714. 2. fid
13C



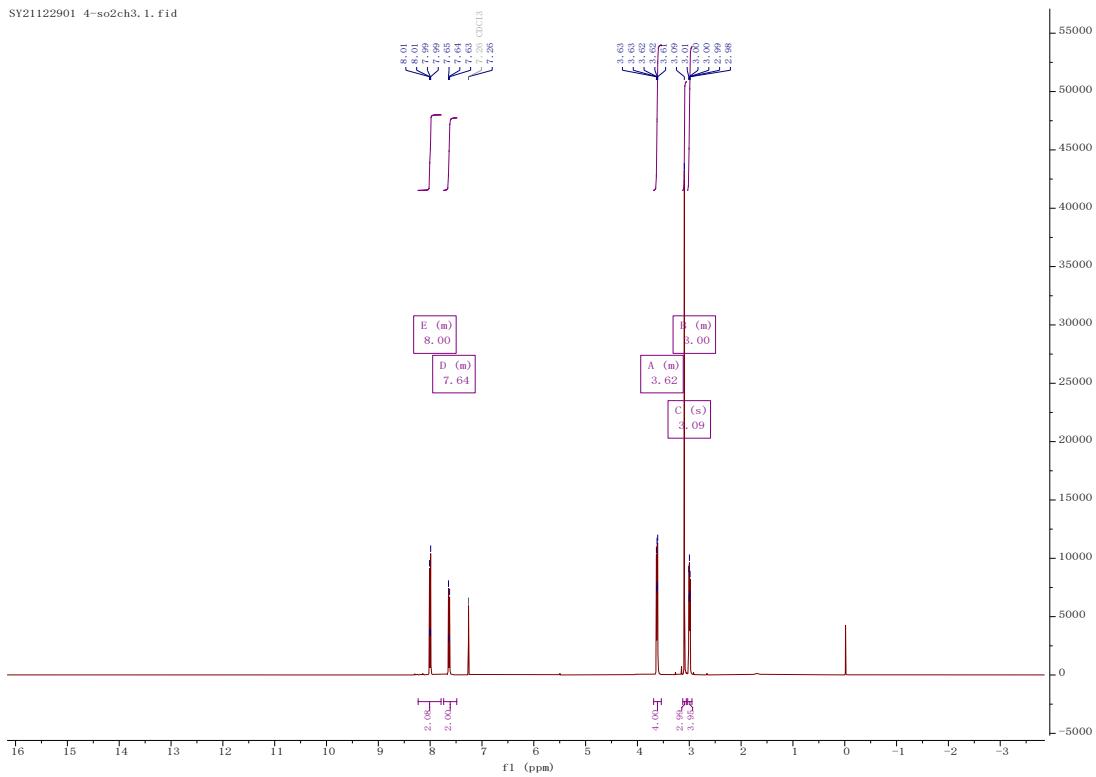
¹⁹F NMR

SY220714. 3. fid
19F



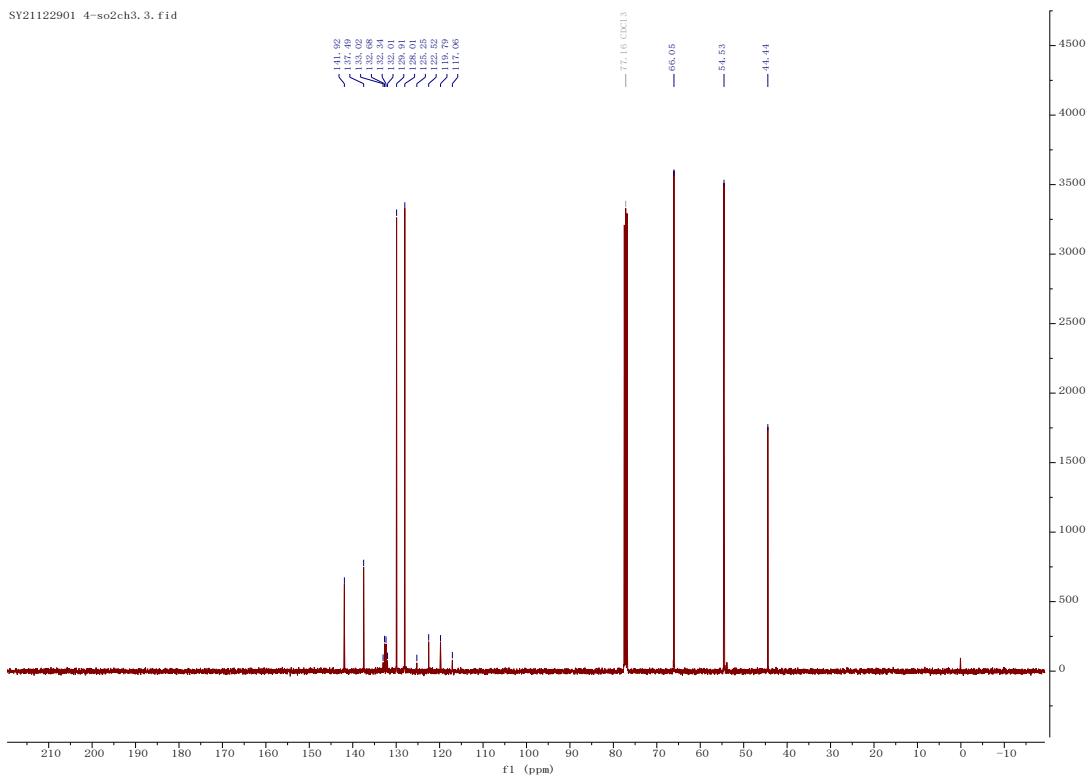
¹H NMR

SY21122901 4-so2ch3, 1, fid



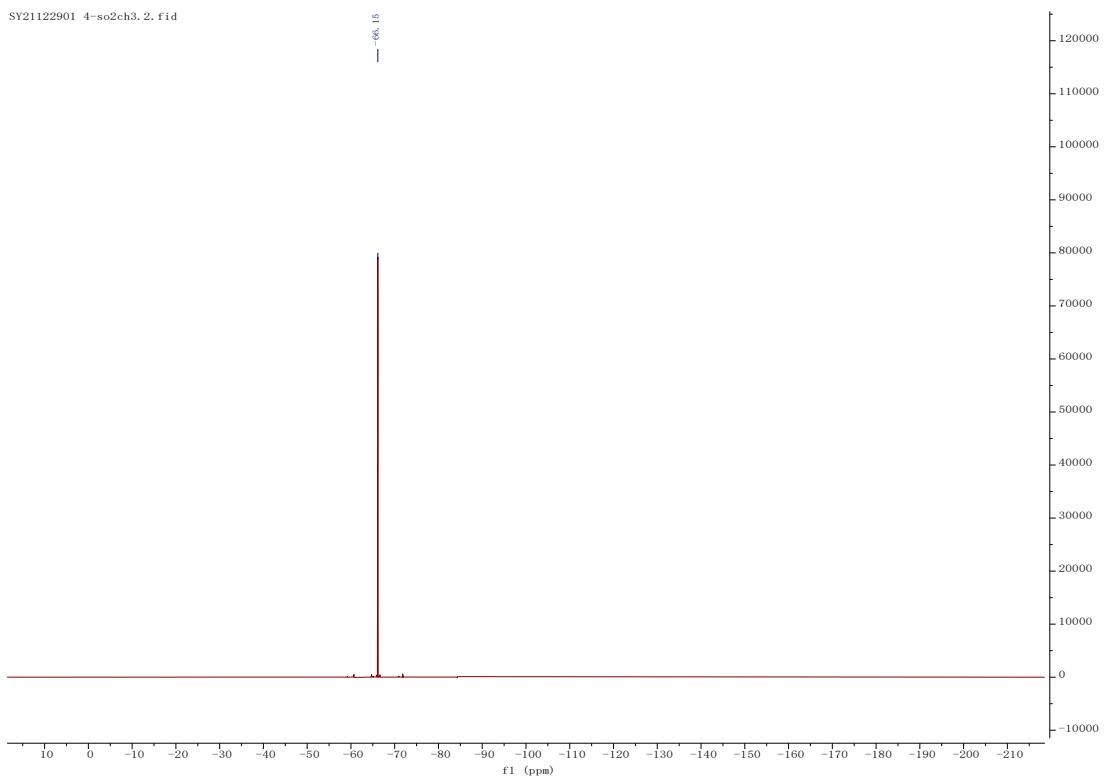
¹³C NMR

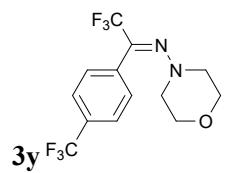
SY21122901 4-so2ch3. 3. fid



¹⁹F NMR

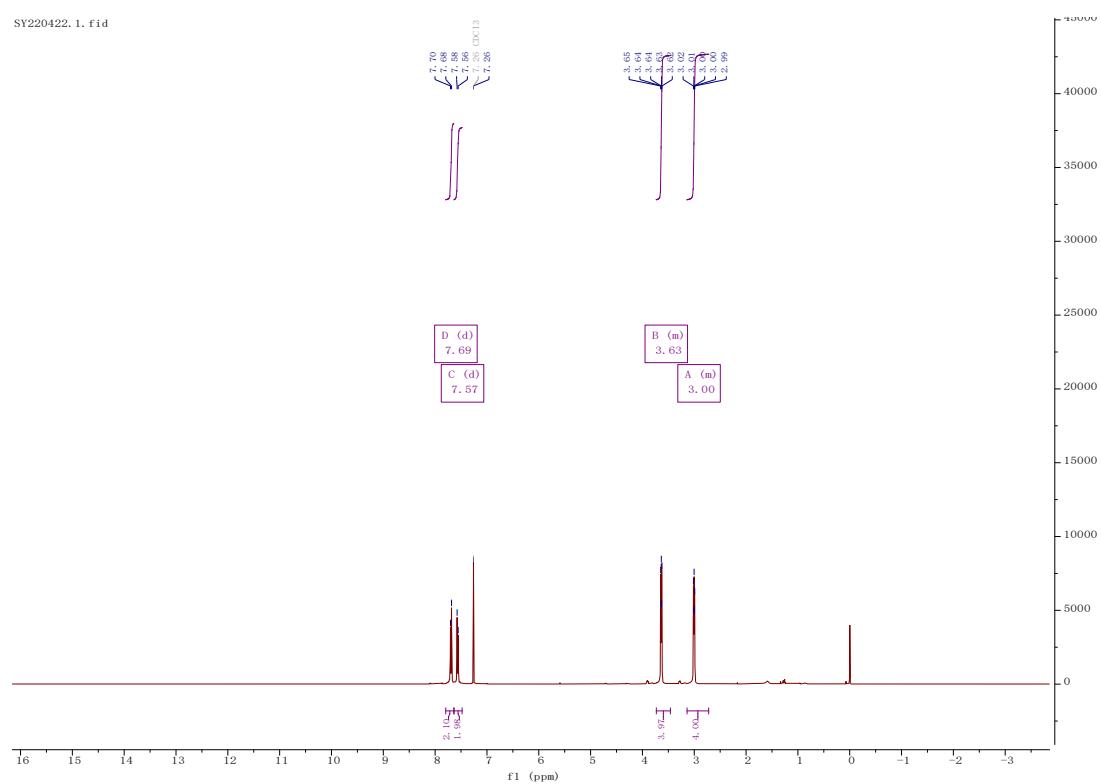
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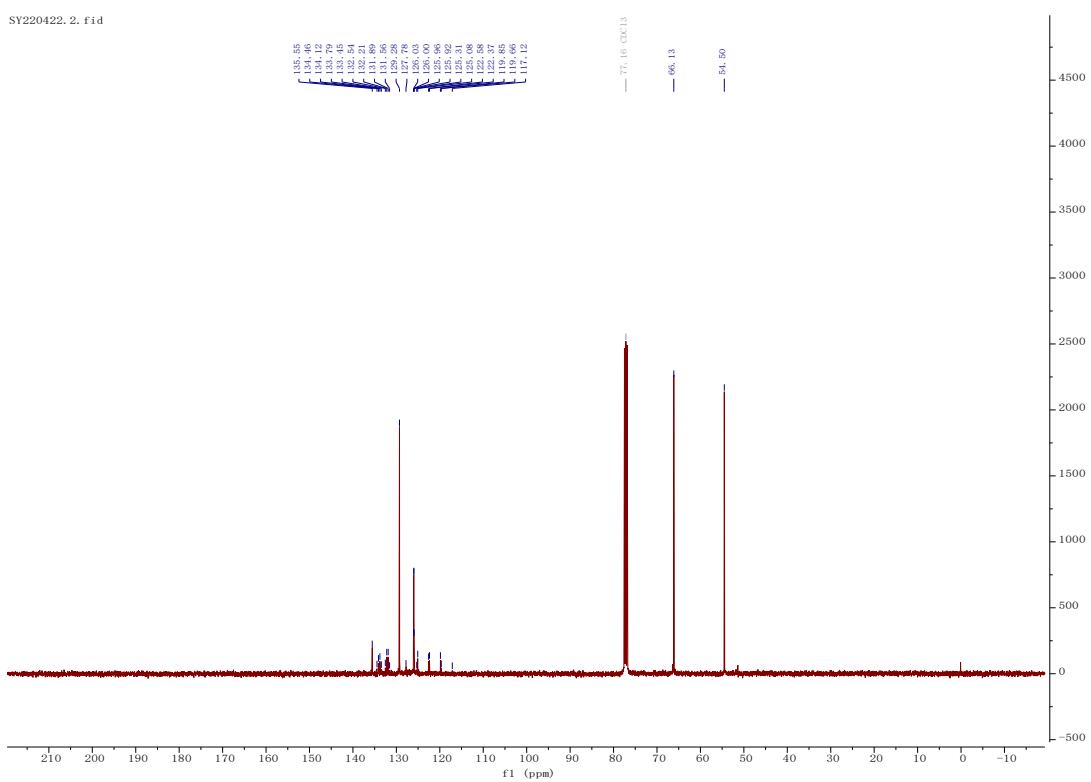
¹H NMR

SY220422, 1. fid



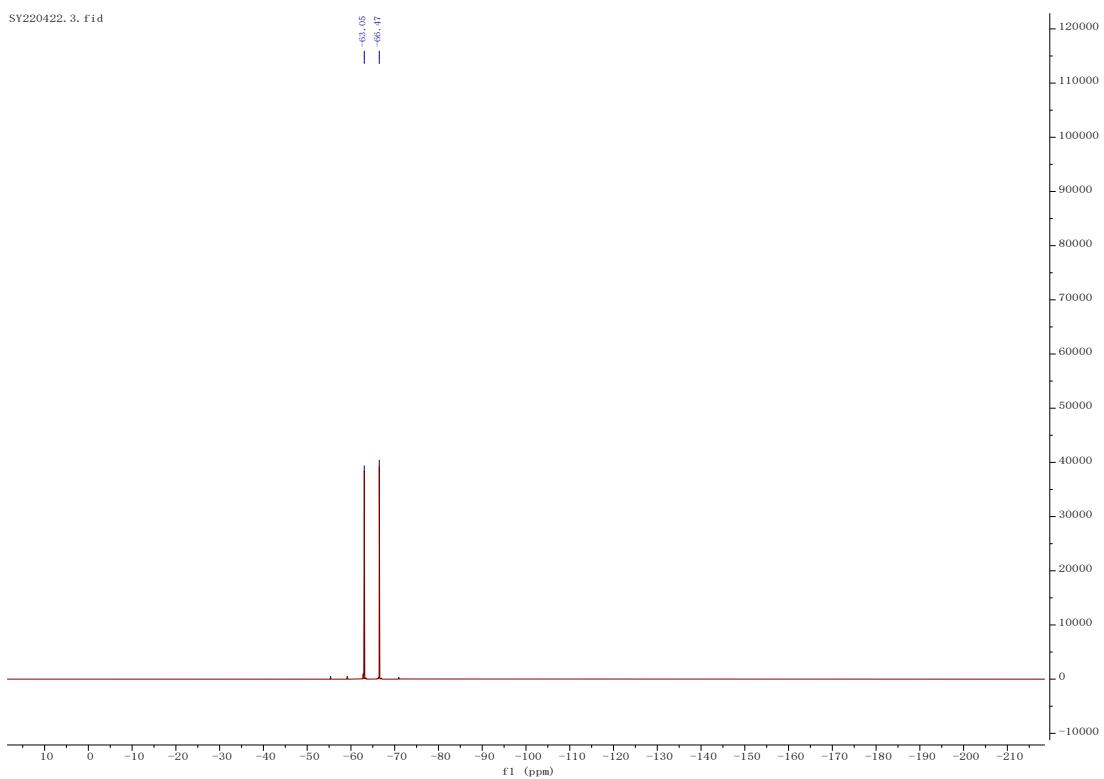
¹³C NMR

SY220422. 2. fid



^{19}F NMR

SY220422. 3. fid



Reference

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