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Supporting information for

A ligand-free strategy for the copper-catalyzed direct alkynylation of trifluoromethyl ketones

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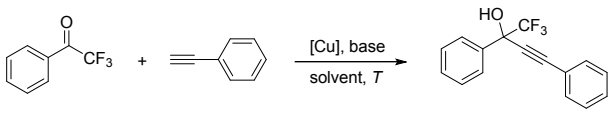
Contents

Table S1 Optimization of alkynylation[a]



Entry	Solvent	*T* (°C)	Base	Catalyst	Yield[b] (%)
1	DMF	70	KO*t*-Bu	CuI	35
2	DMA	70	KO*t*-Bu	CuI	15
3	NMP	70	KO*t*-Bu	CuI	29
4	THF	70	KO*t*-Bu	CuI	4
5	Toluene	70	KO*t*-Bu	CuI	5
6	C_2H_5OH	70	KO*t*-Bu	CuI	3
7	*i*-PrOH	70	KO*t*-Bu	CuI	22
8	Glycerol	70	KO*t*-Bu	CuI	trace
9	H_2O	70	KO*t*-Bu	CuI	trace
10	DMSO	70	KO*t*-Bu	CuI	97
11	DMF	70	K_2CO_3	CuI	78
12	DMA	70	K_2CO_3	CuI	85
13	NMP	70	K_2CO_3	CuI	65
14	THF	70	K_2CO_3	CuI	trace
15	Toluene	70	K_2CO_3	CuI	6
16	C_2H_5OH	70	K_2CO_3	CuI	5
17	*i*-PrOH	70	K_2CO_3	CuI	19
18	Glycerol	70	K_2CO_3	CuI	trace
19	H_2O	70	K_2CO_3	CuI	trace
20	DMSO	50	K_2CO_3	$FeCl_3$	0
21	DMSO	50	K_2CO_3	$FeSO_4$	0
22	DMSO	50	K_2CO_3	$Fe(NO_3)_2$	0
23	DMSO	50	K_2CO_3	$AlCl_3$	0
24	DMSO	50	K_2CO_3	$Al_2(SO_4)_3$	0
25	DMSO	50	K_2CO_3	$MgCl_2$	0
26	DMSO	50	K_2CO_3	BF_3	0
27[c]	DMSO	50	K_2CO_3	CuI	90
28[d]	DMSO	50	K_2CO_3	CuI	96
29[e]	DMSO	50	K_2CO_3	CuI	72

[a] Reaction conditions: 2,2,2-trifluoroacetophenone (0.5 mmol), phenylacetylene (1.0 mmol), Cu source (10 mol%), base (20 mol%) in solvent (1 mL) under N_2, 24 h. [b] Isolated yield. [c] In air. [d] phenylacetylene (1.5 equiv.). [e] phenylacetylene (1.2 equiv.).

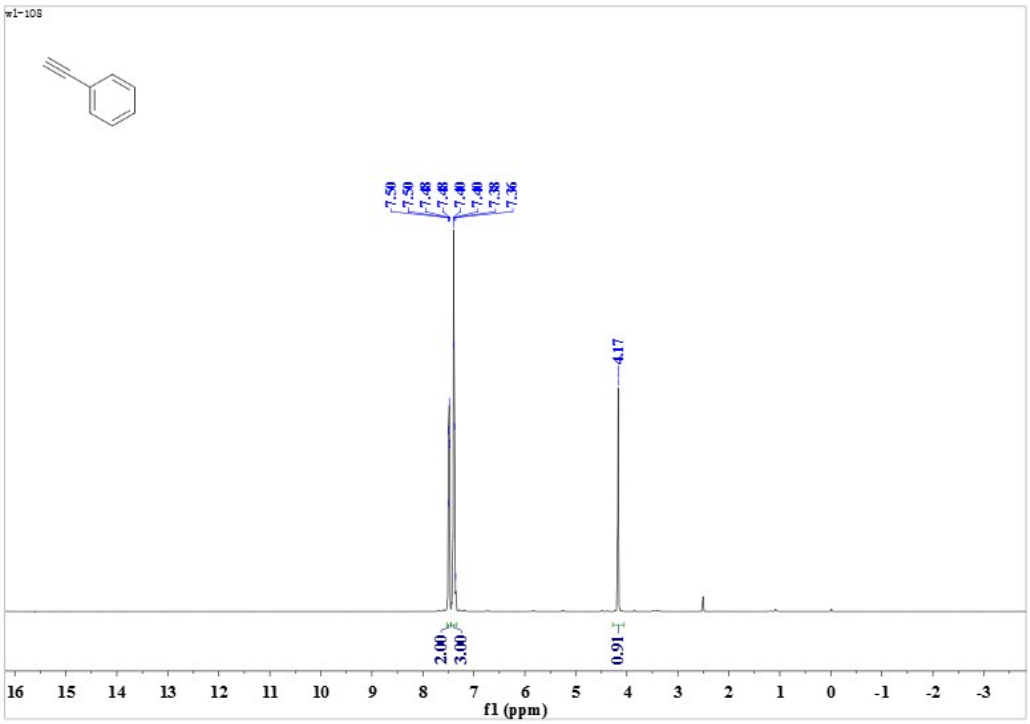


Figure S1. ^{1}H NMR of phenylacetylene in DMSO-d_6.

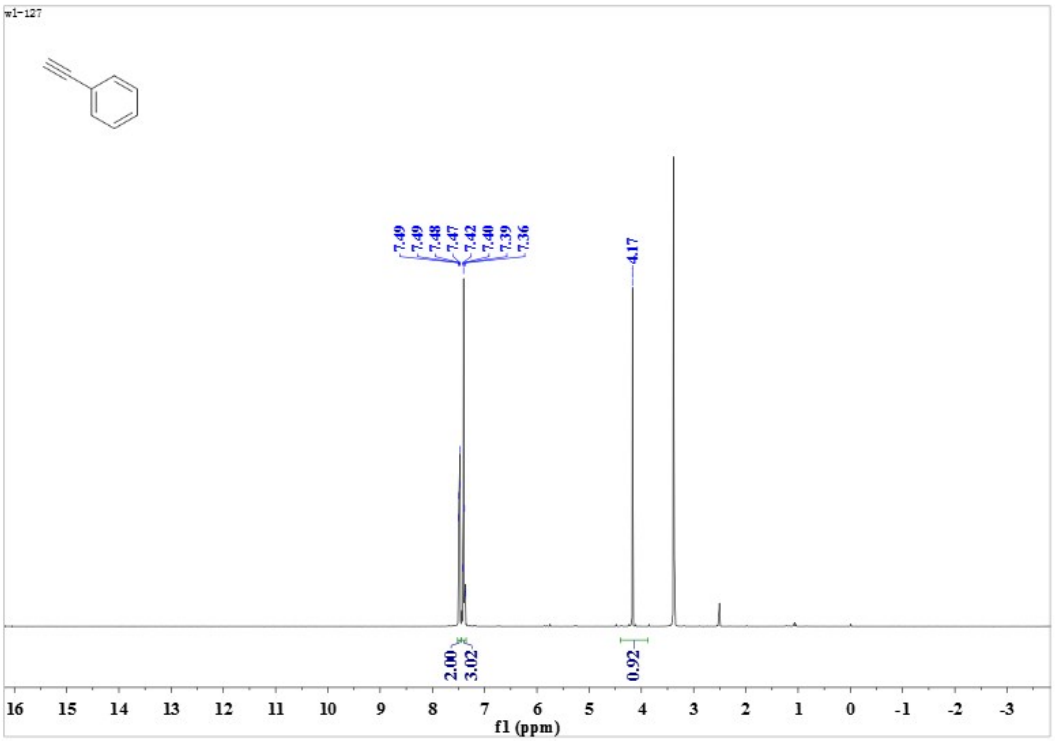


Figure S2. ^{1}H NMR of phenylacetylene in DMSO-d_6. Reaction condition: a mixture of phenylacetylene (1.0 mmol) and K_2CO_3 (1.0 mmol) in 1 mL DMSO-d_6 was allowed to react in Schlenk tubes for 24 h under nitrogen atmosphere.

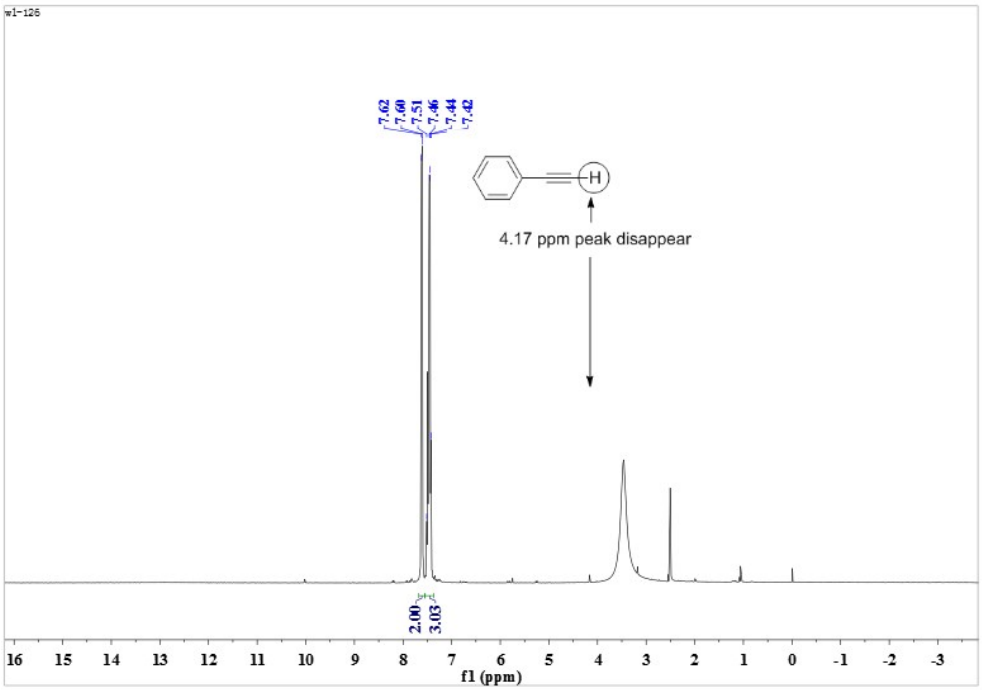


Figure S3. ^{1}H NMR of phenylacetylene in DMSO-d_6. Reaction condition: a mixture of phenylacetylene (1.0 mmol), K_2CO_3 (1.0 mmol), and $Cu(OAc)_2$ (1.0 mmol) in 1 mL DMSO-d_6 was allowed to react in Schlenk tubes for 24 h under nitrogen atmosphere.

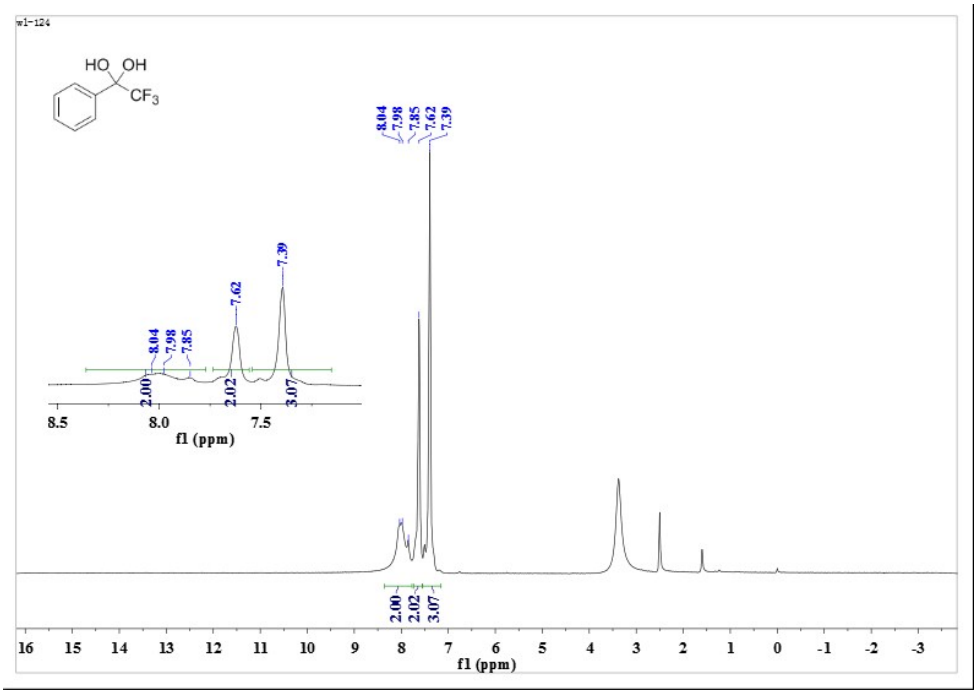


Figure S4. ^{1}H NMR of 2,2,2-trifluoro-1-phenylethane-1,1-diol in DMSO-d_6. Reaction condition: a mixture of 2,2,2-trifluoroacetophenone (1.0 mmol), K_2CO_3 (1.0 mmol), and $Cu(OAc)_2$ (1.0 mmol) in 1 mL DMSO-d_6 was allowed to react in Schlenk tubes for 24 h under nitrogen atmosphere.

Experimental Section

General Experimental Methods

All reactions were carried out in Schlenk tubes under an atmosphere of nitrogen. DMSO (dimethyl sulfoxide) was distilled from 4Å-molecular sieves. Trifluoromethyl ketones and other reagents were purchased from Alfa Aesar, Acros, and Adamas. ^{1}H NMR spectra (400 MHz) and ^{13}C NMR spectra (100 MHz) were recorded on a Varian Inova-400 spectrometer using TMS as internal standard. ^{19}F NMR spectra (376 MHz) were recorded on a Varian Inova-400 spectrometer using CF_3COOH as internal standard. Mass spectroscopy data of the compounds were collected on a Bruker ultrafleXtreme mass spectrometer. All products were isolated by short chromatography on a silica gel (300–400 mesh) column.

General Procedure for Optimization of Reaction Condition

A mixture of 2,2,2-trifluoroacetophenone (0.5 mmol), phenylacetylene (1.0 mmol), Cu source (0.05 mmol), and base (0.1 mmol) in solvent (1 mL) was allowed to react in Schlenk tubes for 24 h under nitrogen atmosphere. After reaction, the reaction mixture was added to brine (15 mL) and extracted three times with dichloromethane (3×15 mL). The solvent was concentrated under vacuum and the product was isolated by short chromatography on a silica gel (300–400 mesh) column.

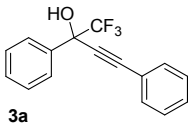
General Procedure for Testing of Scope

A mixture of ketone (0.5 mmol), alkyne (0.75 mmol), $Cu(OAc)_2$ (0.05 mmol), and K_2CO_3 (0.1 mmol) in DMSO (1 mL) was allowed to react in Schlenk tubes at 50 °C or 70 °C for 24 h under nitrogen atmosphere. After reaction, the reaction mixture was added to brine (15 mL) and extracted three times with dichloromethane (3×15 mL). The solvent was concentrated under vacuum and the product was isolated by short chromatography on a silica gel (300–400 mesh) column.

General Procedure for Scaled reaction

A mixture of 2,2,2-trifluoroacetophenone (5 mmol), cyclopropyl acetylene (7.5 mmol), $Cu(OAc)_2$ (0.5 mmol), and K_2CO_3 (1 mmol) in DMSO (10 mL) was allowed to react in Schlenk tubes at 50 °C for 24 h. After reaction, the reaction mixture was added to brine (100 mL) and extracted three times with dichloromethane (3×100 mL). The solvent was concentrated under vacuum and the product was isolated by short chromatography on a silica gel (300–400 mesh) column.

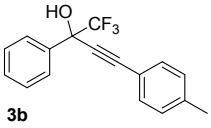
Characterization Data of Products



3a

1,1,1-trifluoro-2,4-diphenylbut-3-yn-2-ol [1]

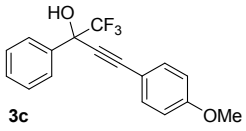
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (133 mg, 96%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.00 (d, J = 4.0 Hz, 1H), 7.84-7.82 (m, 2H), 7.60 (d, J = 3.2 Hz, 2H), 7.52-7.44 (m, 6H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.15, 132.11, 130.05, 129.67, 129.26, 128.60, 127.55,122.83 (t, $J_{C\text{-}F}$ = 284.0 Hz), 119.98, 86.94, 86.15, 72.43 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.86, ppm.



3b

1,1,1-trifluoro-2-phenyl-4-(*p*-tolyl)but-3-yn-2-ol [2]

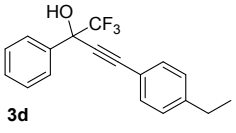
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (133 mg, 91%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.93 (d, J = 2.0 Hz, 1H), 7.81 (d, J = 6.0 Hz, 2H), 7.48 (d, J = 7.2 Hz, 5H), 7.26 (d, J = 7.2 Hz, 2H), 2.34 (s, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 139.98, 137.24, 132.03, 129.88, 129.64, 128.58, 127.54, 122.84 (t, $J_{C\text{-}F}$ = 284.0 Hz), 118.08, 87.10, 85.51, 72.56 (q, $J_{C\text{-}F}$ = 31.0 Hz), 21.44, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.85, ppm.



3c

1,1,1-trifluoro-4-(4-methoxyphenyl)-2-phenylbut-3-yn-2-ol [1]

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a white solid (148 mg, 96%), mp = 94-95 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.85 (s, 1H), 7.77 (d, J = 2.0 Hz, 2H), 7.49 (d, J = 8.4 Hz, 5H), 7.00 (d, J = 2.0 Hz, 2H), 3.80 (s, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 160.57, 137.28, 133.78, 129.63, 128.59, 127.54, 122.84 (t, $J_{C\text{-}F}$ = 284.0 Hz), 114.93, 112.89, 87.07, 84.72, 72.52 (q, $J_{C\text{-}F}$ = 31.0 Hz), 55.75, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.83, ppm.

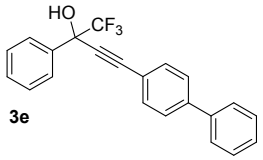


3d

4-(4-ethylphenyl)-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (135 mg, 89%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.94 (s, 1H), 7.81 (d, J = 7.2

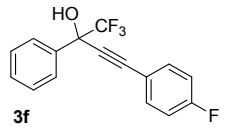
Hz, 2H), 7.51-7.43 (m, 5H), 7.29 (d, J = 8.0 Hz, 2H), 2.64 (q, J = 7.6 Hz, 2H), 1.18 (t, J = 7.6 Hz, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 146.13, 137.23, 132.12, 129.63, 128.69, 128.57, 127.54, 122.83 (t, $J_{C\text{-}F}$ = 284.0 Hz), 118.33, 87.10, 85.51, 72.55 (q, $J_{C\text{-}F}$ = 31.0 Hz), 28.53, 15.59, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.89, ppm; HRMS (MALDI): m/z calcd for $C_{18}H_{15}F_3O$ $[M+H\text{-}H_2O]^+$ 287.1042, found 287.1042.



3e

4-([1,1'-biphenyl]-4-yl)-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol

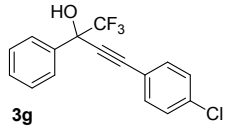
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a white solid (162 mg, 92%), mp = 124-125 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.96 (s, 1H), 7.79-7.76 (m, 4H), 7.72 (d, J = 7.2 Hz, 2H), 7.66 (d, J = 8.4 Hz, 2H), 7.52-7.46 (m, 5H), 7.41 (d, J = 7.2 Hz, 1H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 141.58, 139.34, 137.04, 132.75, 129.76, 129.52, 128.67, 128.55, 127.53, 127.49, 127.17, 122.78 (t, $J_{C\text{-}F}$ = 284.0 Hz), 109.99, 86.77, 86.72, 72.39 (t, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.78, ppm; HRMS (MALDI): m/z calcd for $C_{22}H_{15}F_3O$ $[M+H\text{-}H_2O]^+$ 335.1042, found 335.1042.



3f

1,1,1-trifluoro-4-(4-fluorophenyl)-2-phenylbut-3-yn-2-ol [1]

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (128 mg, 87%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.95 (s, 1H), 7.78 (d, J = 7.2 Hz, 2H), 7.65 (t, J = 7.2 Hz, 2H), 7.51-7.44 (m, 3H), 7.30 (t, J = 8.8 Hz, 2H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 164.20, 161.73, 137.00, 134.66, 134.57, 129.72, 128.63, 127.52, 122.75 (t, $J_{C\text{-}F}$ = 284.0 Hz), 117.49, 117.46, 116.74, 116.51, 85.92, 85.86, 72.52 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.84, -30.90 – -30.97 (m), ppm.

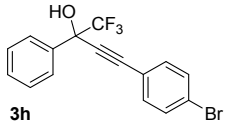


3g

4-(4-chlorophenyl)-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol [2]

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (101 mg, 65%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.02 (s, 1H), 7.80 (d, J = 8.0 Hz, 2H), 7.63-7.60 (m, 2H), 7.54-7.44 (m, 5H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 136.66, 134.71, 133.64, 129.49, 129.25, 128.39, 127.27, 123.90 (d, $J_{C\text{-}F}$ = 285.0 Hz), 119.64, 86.87, 85.53, 72.31 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz,

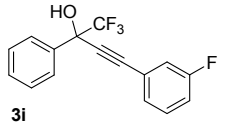
DMSO-d_6): δ -0.82, ppm.



3h

4-(4-bromophenyl)-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol

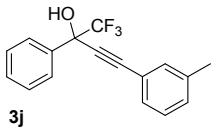
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (137 mg, 77%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.00 (s, 1H), 7.79 (d, J = 7.2 Hz, 2H), 7.67 (d, J = 8.0 Hz, 2H), 7.55-7.44 (m, 5H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 136.87, 134.04, 132.40, 129.76, 128.65, 127.51, 123.68, 122.69 (t, $J_{C\text{-}F}$ = 284.0 Hz), 120.24, 87.24, 85.87, 72.58 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.79, ppm; HRMS (MALDI): m/z calcd for $C_{16}H_{10}BrF_3O$ [M+H-H_2O]$^+$ 336.9834, found 336.9831.



3i

1,1,1-trifluoro-4-(3-fluorophenyl)-2-phenylbut-3-yn-2-ol

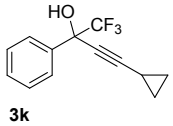
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (93 mg, 63%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.00 (s, 1H), 7.78 (d, J = 7.2 Hz, 2H), 7.54-7.42 (m, 6H), 7.36 (t, J = 8.4 Hz, 1H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.48, 161.04, 136.81, 131.59, 131.51, 129.79, 128.68, 128.61, 128.58, 127.52, 122.95, 122.86, 122.69 (t, $J_{C\text{-}F}$ = 284.0 Hz), 118.87, 118.64, 117.65, 117.44, 86.97, 85.59, 85.55, 72.53 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.78, -33.86 – -33.92 (m), ppm; HRMS (MALDI): m/z calcd for $C_{16}H_{10}F_4O$ [M+H-H_2O]$^+$ 277.0635, found 277.0635.



3j

1,1,1-trifluoro-2-phenyl-4-(*m*-tolyl)but-3-yn-2-ol [2]

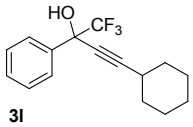
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (142 mg, 97%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.96 (s, 1H), 7.81 (d, J = 7.2 Hz, 2H), 7.52-7.44 (m, 3H), 7.42-7.27 (m, 4H), 2.33(s, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 138.50, 136.92, 132.25, 130.57, 129.42, 128.93, 128.91, 128.35, 127.29, 122.57 (t, $J_{C\text{-}F}$ = 285.0 Hz), 120.70, 86.82, 85.54, 72.30 (q, $J_{C\text{-}F}$ = 31.0 Hz), 20.79, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.86, ppm.



3k

4-cyclopropyl-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol [1]

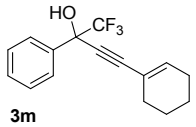
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (80 mg, 67%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.65 (d, J = 8.0 Hz, 2H), 7.54 (s, 1H), 7.45-7.38 (m, 3H), 1.52-1.46 (m, 1H), 0.88 (dd, J = 8.2 Hz, J = 2.8 Hz, 2H), 0.72-0.69 (m, 2H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 138.19, 130.05, 129.03, 128.10, 123.43 (t, $J_{C\text{-}F}$ = 284.0 Hz), 92.09, 72.82, 72.58 (q, $J_{C\text{-}F}$ = 31.0 Hz), 9.19, 9.14, 0.00, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -1.08, ppm.



3l

4-cyclohexyl-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol

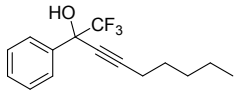
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (86 mg, 61%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.69 (d, J = 6.8 Hz, 2H), 7.54 (s, 1H), 7.46-7.39 (m, 3H), 2.64-2.60 (m, 1H), 1.76 (d, J = 8.4 Hz, 2H), 1.70-1.66 (m, 2H), 1.53-1.43 (m, 3H), 1.38-1.35 (m, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.65, 129.42, 128.38, 127.50, 122.86 (t, $J_{C\text{-}F}$ = 284.0 Hz), 92.01, 77.59, 71.97 (q, $J_{C\text{-}F}$ = 31.0 Hz), 32.02 (d, $J_{C\text{-}F}$ = 3.7 Hz), 28.32, 25.73, 24.29, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -1.25, ppm; HRMS (EI): m/z calcd for $C_{16}H_{17}F_3O$ $[M]^+$ 282.1232, found 282.1234.



3m

4-(cyclohex-1-en-1-yl)-1,1,1-trifluoro-2-phenylbut-3-yn-2-ol

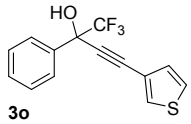
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (116 mg, 83%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.70 (s, 2H), 7.68 (s, 1H), 7.47-7.40 (m, 3H), 6.26-6.24 (m, 1H), 2.12-2.10 (m, 4H), 1.62-1.54 (m, 4H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.71, 137.37, 129.53, 128.48, 127.47, 122.79 (t, $J_{C\text{-}F}$ = 284.0 Hz), 119.24, 88.73, 83.53, 72.35 (q, $J_{C\text{-}F}$ = 31.0 Hz), 28.76, 25.55, 22.06, 21.26, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -1.01, ppm; HRMS (MALDI): m/z calcd for $C_{16}H_{15}F_3O$ $[M+H\text{-}H_2O]^+$ 263.1042, found 263.1044.



3n

1,1,1-trifluoro-2-phenylnon-3-yn-2-ol

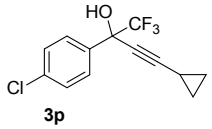
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (96 mg, 71%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.69 (d, J = 7.6 Hz, 2H), 7.56 (s, 1H), 7.45-7.41 (m, 3H), 2.35 (t, J = 7.2 Hz, 2H), 1.56-1.49 (m, 2H), 1.43-1.29 (m, 4H), 0.88 (t, J = 7.2 Hz, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.62, 129.43, 128.35, 127.49, 122.85 (t, $J_{C\text{-}F}$ = 284.0 Hz), 88.54, 77.41, 71.98 (q, $J_{C\text{-}F}$ = 31.0 Hz), 30.75, 27.78, 21.97, 18.22, 14.25, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -1.20, ppm; HRMS (EI): m/z calcd for $C_{15}H_{17}F_3O$ $[M]^+$ 270.1232, found 270.1231.



3o

1,1,1-trifluoro-2-phenyl-4-(thiophen-3-yl)but-3-yn-2-ol

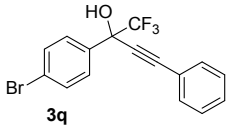
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (120 mg, 85%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.00 (s, 1H), 7.94 (s, 1H), 7.79 (d, J = 6.8 Hz, 2H), 7.67 (s, 1H), 7.49-7.47 (m, 3H), 7.28 (d, J = 4.8 Hz, 1H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.13, 132.05, 130.01, 129.67, 128.60, 127.70, 127.54, 122.79 (t, $J_{C\text{-}F}$ = 284.0 Hz), 85.53, 82.58, 72.59 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.79, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.77, ppm; HRMS (EI): m/z calcd for $C_{14}H_9F_3OS$ $[M]^+$ 282.0326, found 282.0327.



3p

2-(4-chlorophenyl)-4-cyclopropyl-1,1,1-trifluorobut-3-yn-2-ol

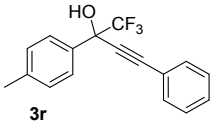
Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (111 mg, 81%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.71 (s, 1H), 7.66 (d, J = 8.4 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 1.53-1.47 (s, 1H), 0.93-0.87 (m, 2H), 0.76-0.69 (m, 2H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 137.26, 135.05, 130.05, 129.18, 123.25 (t, $J_{C\text{-}F}$ = 284.0 Hz), 92.58, 72.33, 72.25, (q, $J_{C\text{-}F}$ = 31.0 Hz), 9.25, 9.20, 0.00, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -1.27, ppm; HRMS (MALDI): m/z calcd for $C_{13}H_{10}ClF_3O$ $[M+H-H_2O]^+$ 257.0339, found 257.0340.



3q

2-(4-bromophenyl)-1,1,1-trifluoro-4-phenylbut-3-yn-2-ol [3]

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (165 mg, 93%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.14 (s, 1H), 7.73 (q, J = 8.8 Hz, 4H), 7.61-7.59 (m, 2H), 7.52-7.44 (m, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 136.56, 132.15, 131.68, 130.19, 129.74, 129.28, 123.36, 122.53 (t, $J_{C\text{-}F}$ = 284.0 Hz), 120.85, 87.25, 85.46, 72.22 (q, $J_{C\text{-}F}$ = 31.0 Hz), ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.98, ppm.

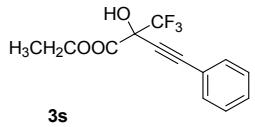


3r

1,1,1-trifluoro-4-phenyl-2-(*p*-tolyl)but-3-yn-2-ol [1]

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (96 mg, 66%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.88 (s, 1H), 7.66 (d, J = 8.0 Hz, 2H), 7.57 (dd, J = 7.2 Hz, J = 2.0 Hz, 2H), 7.50-7.43 (m, 3H), 7.28 (d, J = 8.0 Hz, 2H),

2.34 (s, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 139.17, 134.18, 132.09, 130.05, 129.29, 129.16, 127.42, 122.83 (t, $J_{C\text{-}F}$ = 284.0 Hz), 121.11, 86.74, 86.24, 72.41 (q, $J_{C\text{-}F}$ = 31.0 Hz), 21.08, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ -0.93, ppm.



3s

ethyl 2-hydroxy-4-phenyl-2-(trifluoromethyl)but-3-ynoate [4]

Purification by flash chromatography (petroleum ether/EtOAc = 10:1): a pale yellow oil (131 mg, 96%); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.30 (s, 1H), 7.54-7.44 (m, 5H), 4.34-4.31 (m, 2H), 1.29 (t, J = 7.2 Hz, 3H), ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.18, 132.16, 130.39, 129.30, 122.76 (q, $J_{C\text{-}F}$ = 284.0 Hz), 120.39, 86.71, 82.04, 72.12 (q, $J_{C\text{-}F}$ = 32.0 Hz), 63.47, 14.11, ppm; ^{19}F NMR (376 MHz, DMSO-d_6): δ 1.14, ppm.

Reference

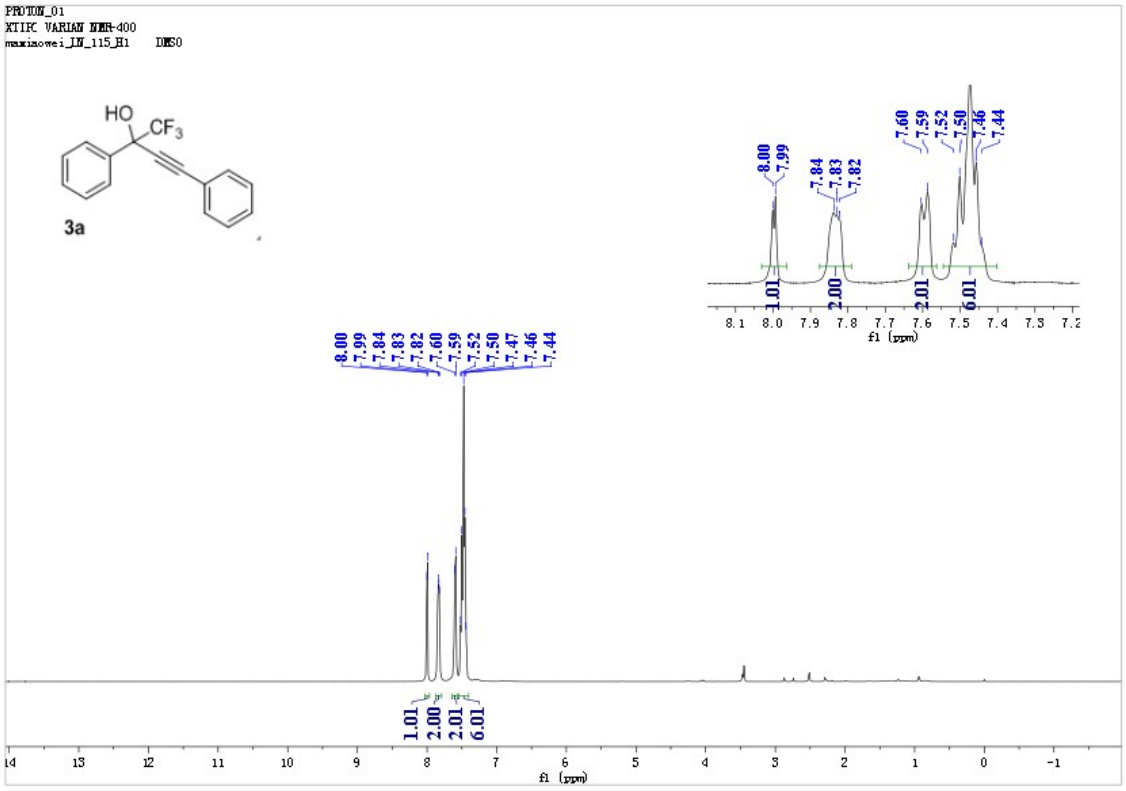
[1] C. A. Correia, D. T. McQuade and P. H. Seeberger, *Adv. Synth. Catal.*, 2013, **355**, 3517–3521.

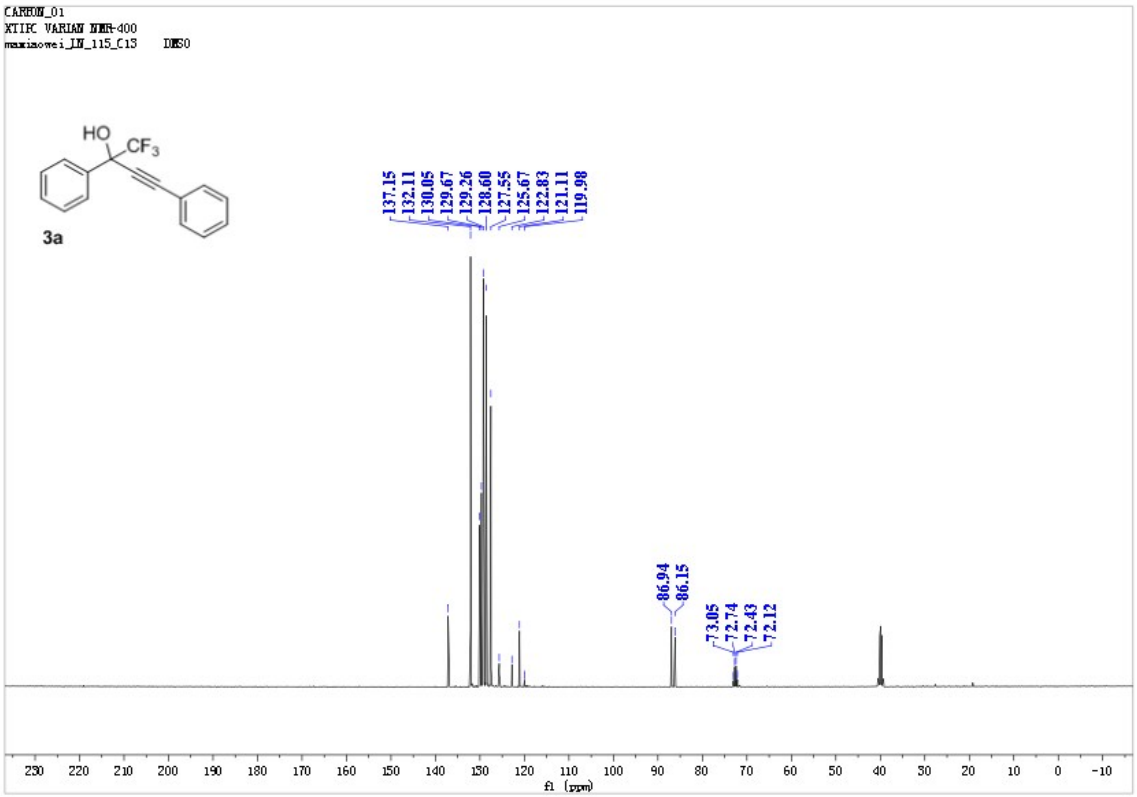
[2] H. Wang, K.-F. Yang, L. Li, Y. Bai, Z.-J. Zheng, W.-Q. Zhang, Z.-W. Gao and L.-W. Xu, *ChemCatChem*, 2014, **6**, 580–591.

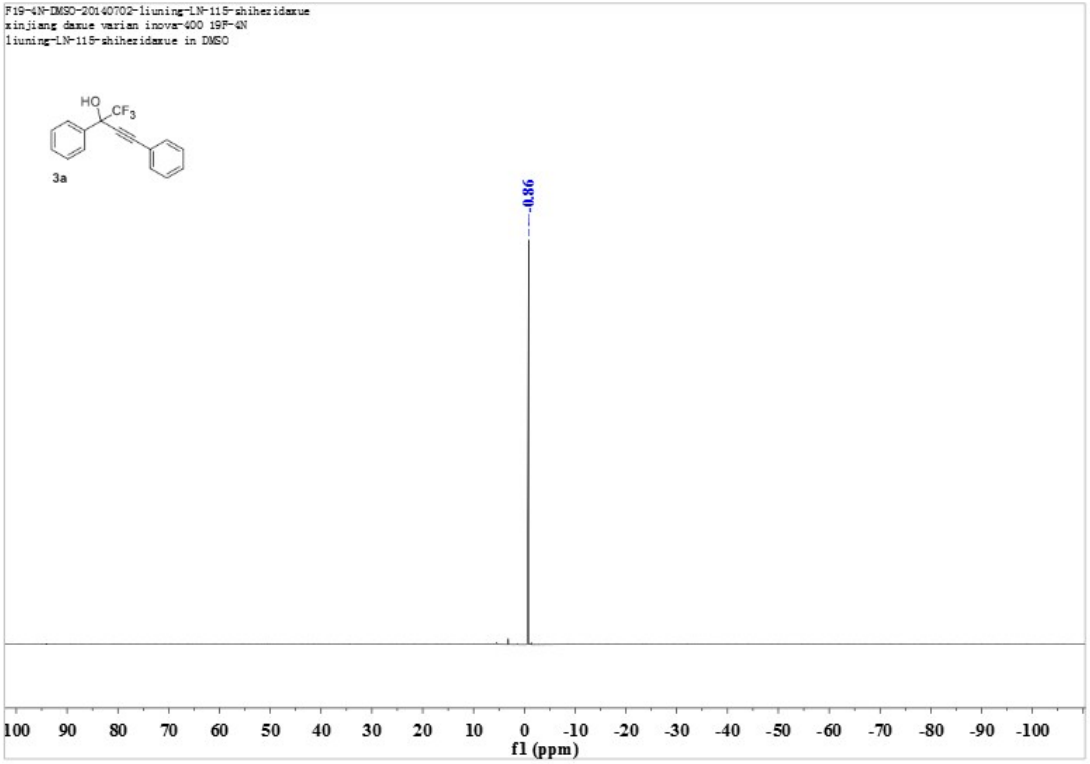
[3] R. Motoki, M. Kanai and M. Shibasaki, *Org. Lett.*, 2007, **9**, 2997–3000.

[4] G.-J. Deng and C.-J. Li, *Synlett*, 2008, 1571-1573.

NMR Spectra for all Products



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HO
CF3
3a
-0.86
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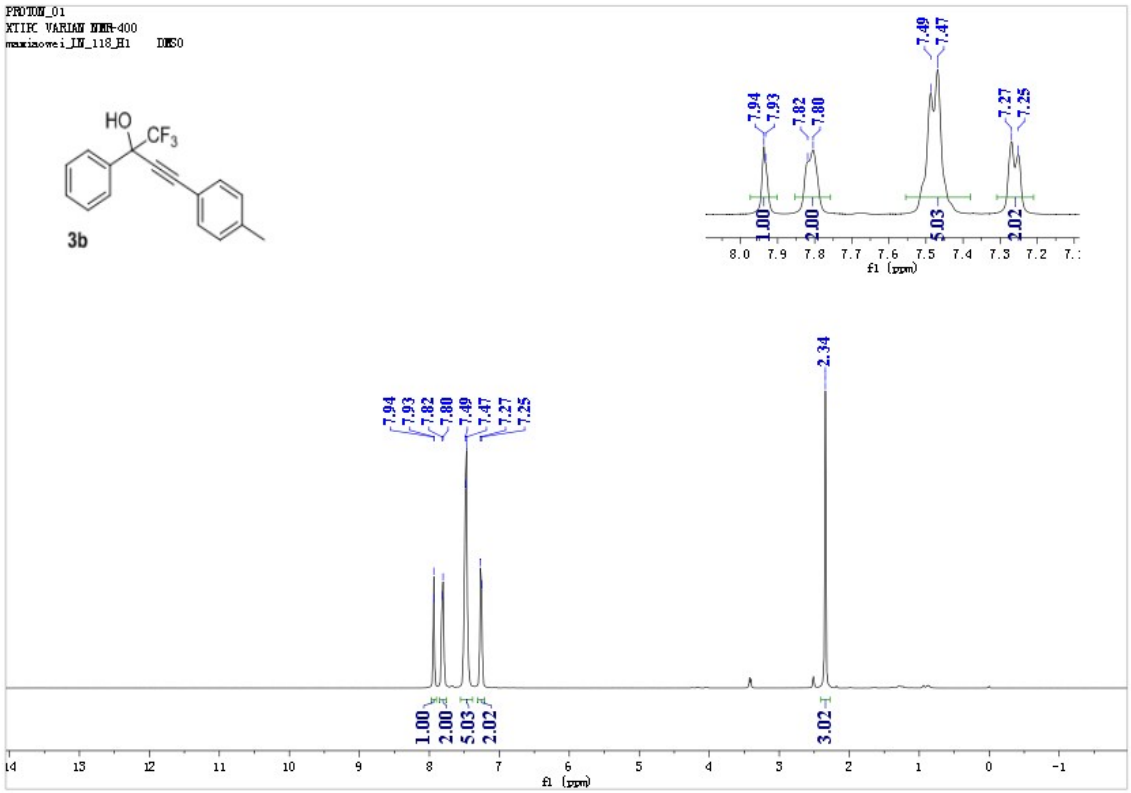
f1 (ppm)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CF3
3b
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7.27
7.25
2.34
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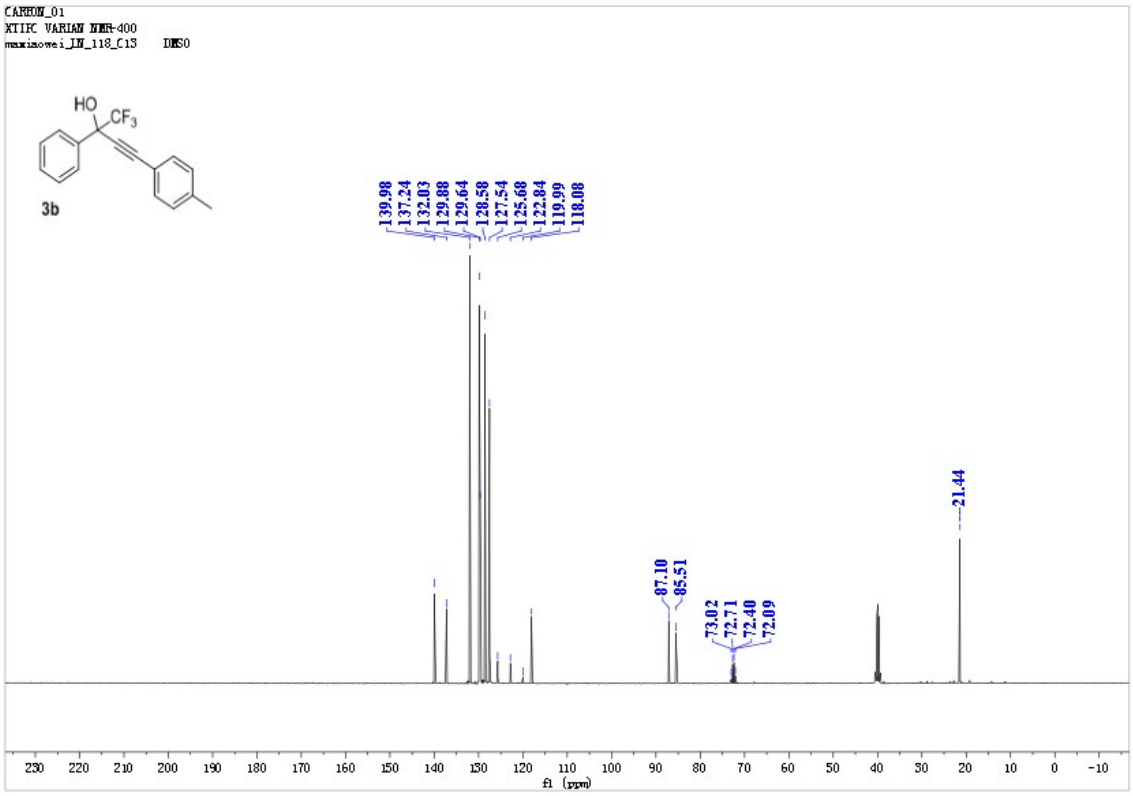
2.00
5.03
2.02
3.02
f1 (ppm)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85.51
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72.40
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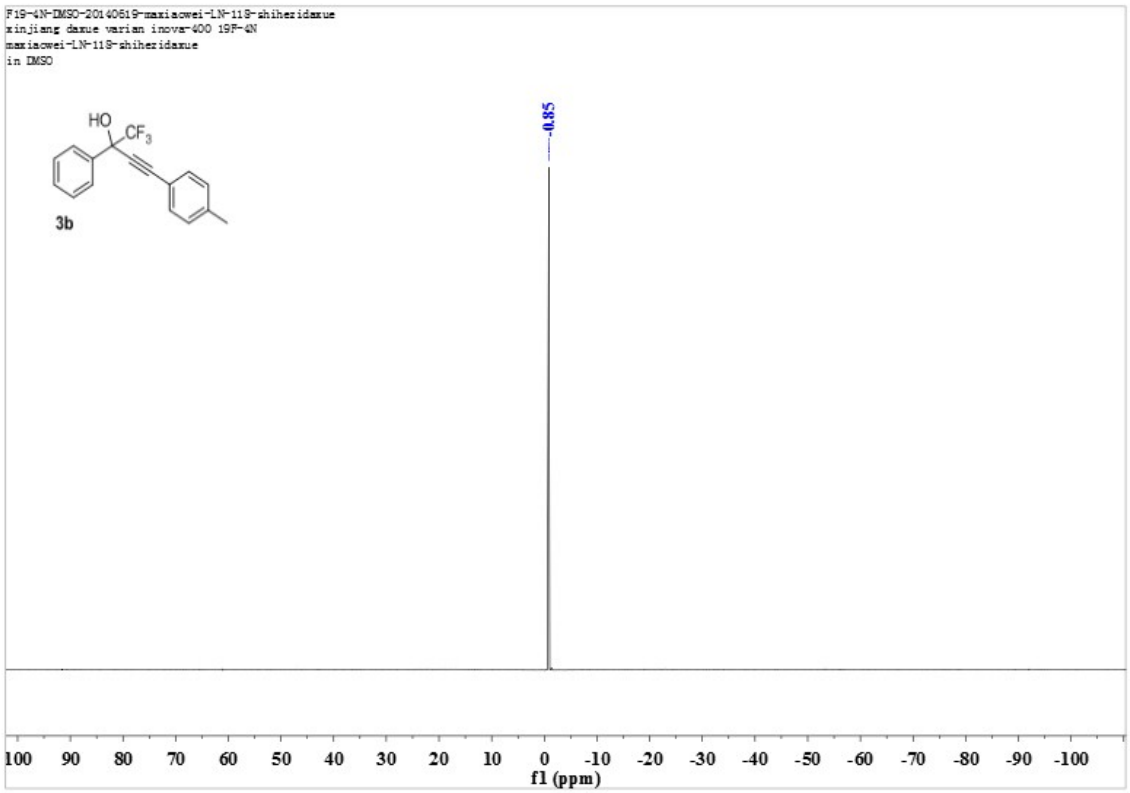
21.44
f1 (ppm)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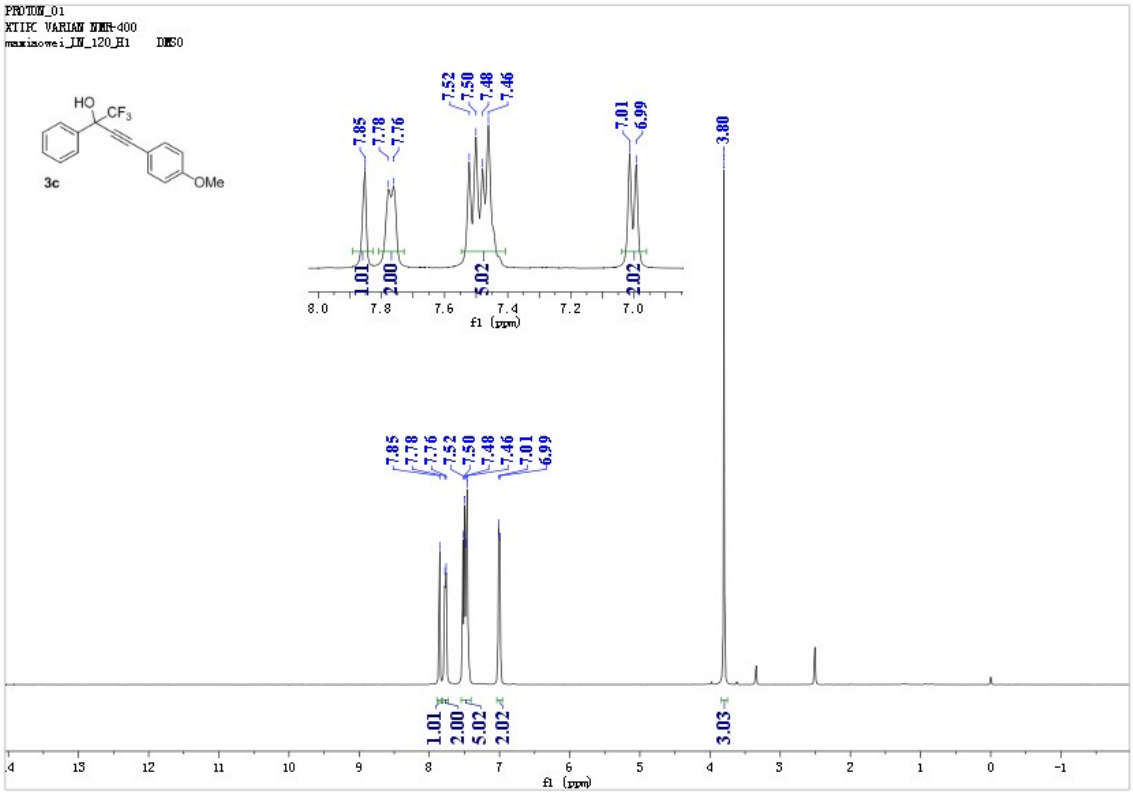
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-118-shiheridaxue
in DMSO
HO
CF3
3b
-0.85
f1 (ppm)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HO
CF3
OMe
3c
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7.76
7.52
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7.48
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7.01
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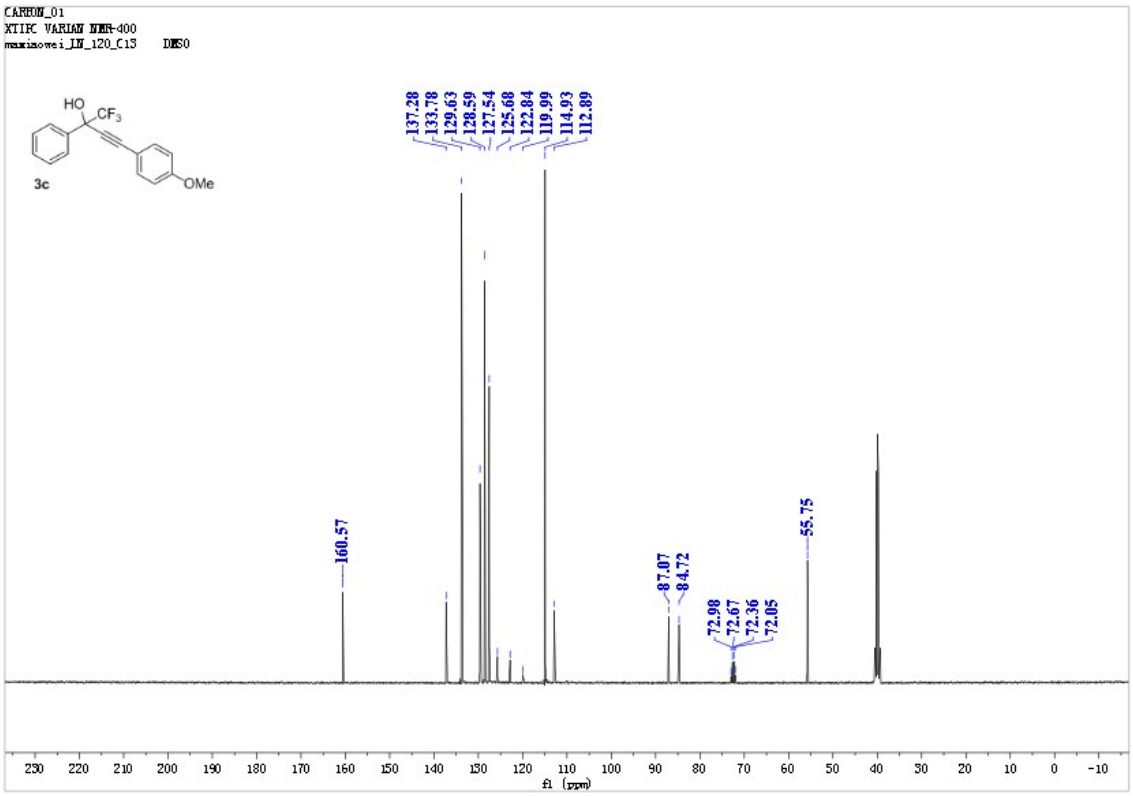
3.80
1.01
2.00
5.02
2.02
3.03
f1 (ppm)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HO
CF3
OMe
3c
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127.54
125.68
122.84
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72.67
72.36
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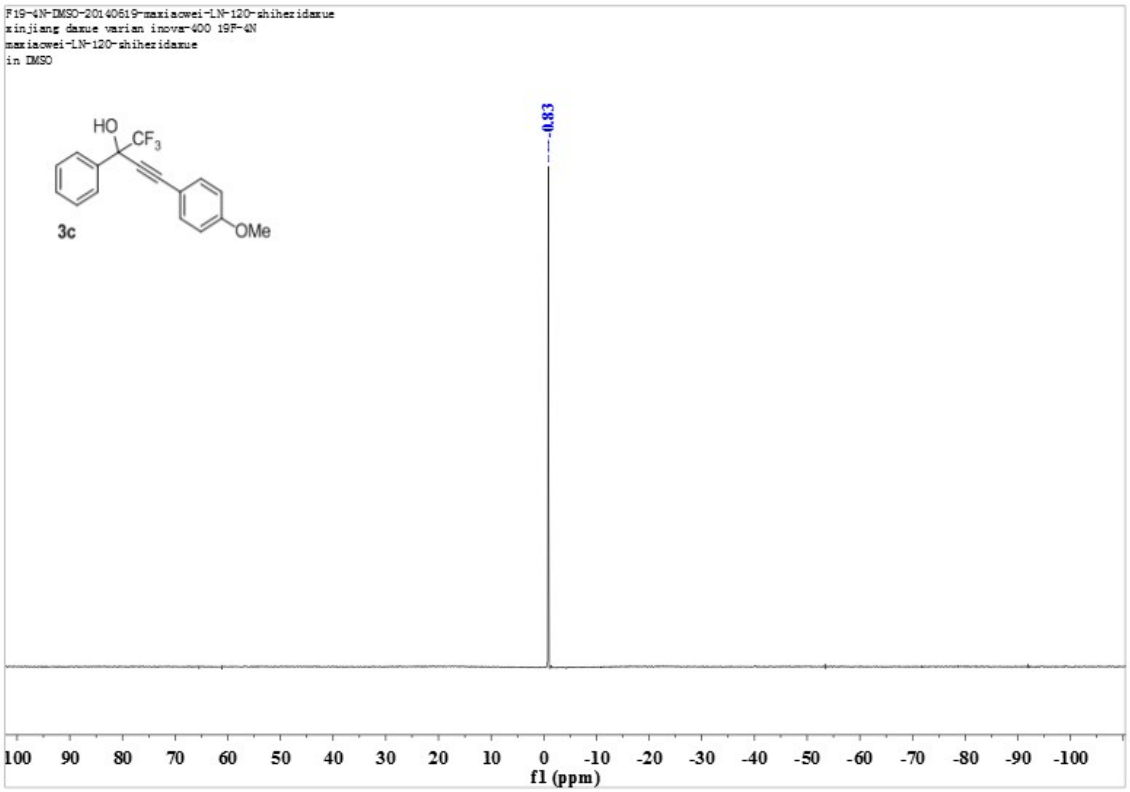
55.75
f1 (ppm)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xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-120-shiheridaxue
in DMSO
HO
CF3
3c
OMe
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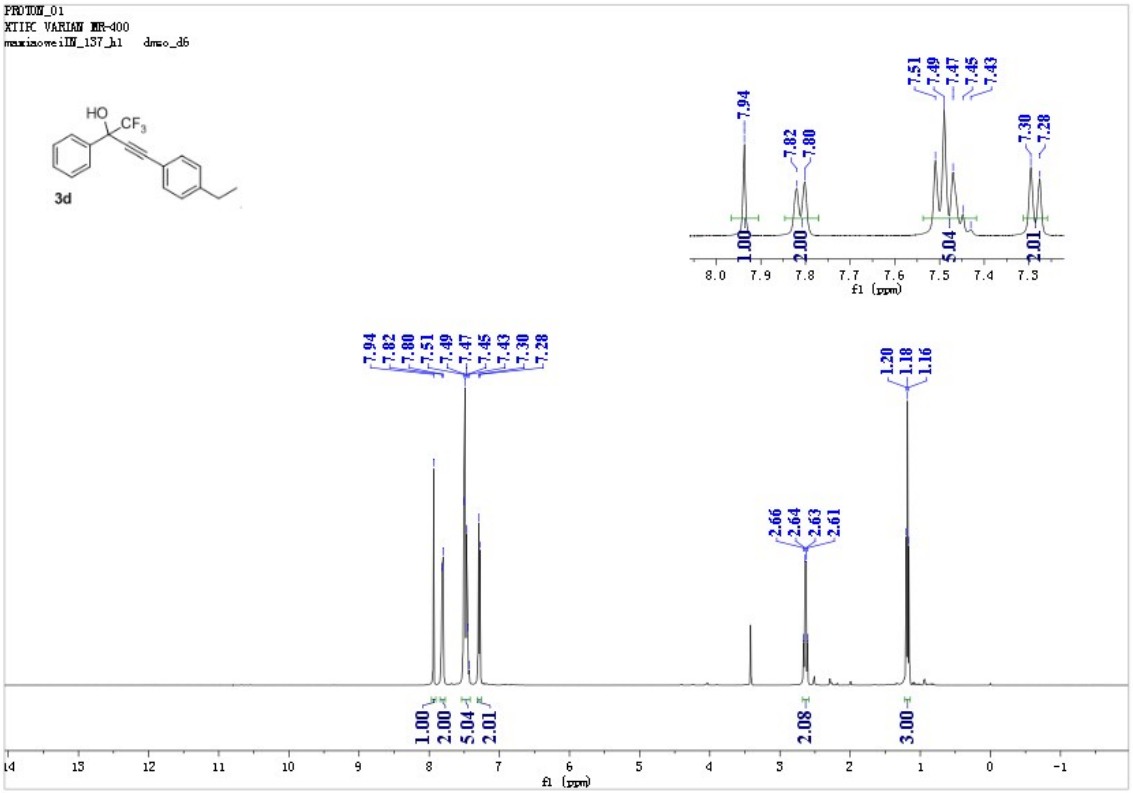
f1 (ppm)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CF3
3d
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7.80
7.51
7.49
7.47
7.45
7.43
7.30
7.28
1.00
2.00
5.04
2.01
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3
f1 (ppm)
1.20
1.18
1.16
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2.64
2.63
2.61
2.08
3.00
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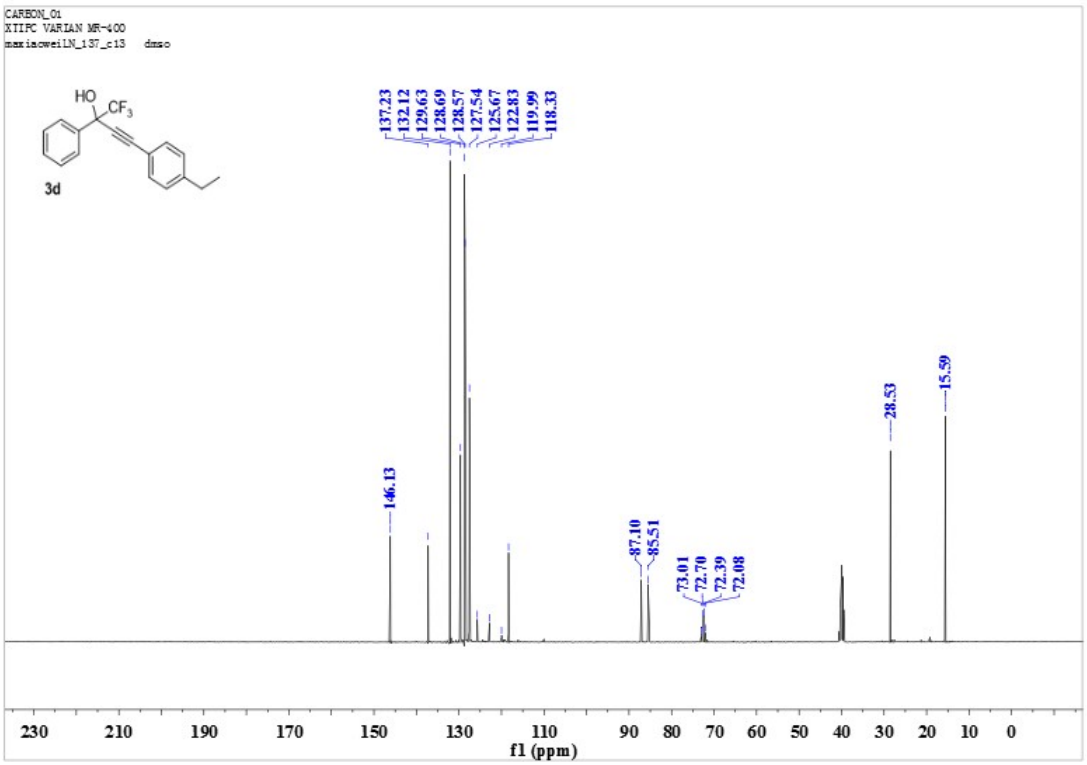
f1 (ppm)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maxiaoweiLN_137_c13 dmso
HO
CF3
3d
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132.12
129.63
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128.57
127.54
125.67
122.83
119.99
118.33
146.13
87.10
85.51
73.01
72.70
72.39
72.08
28.53
15.59
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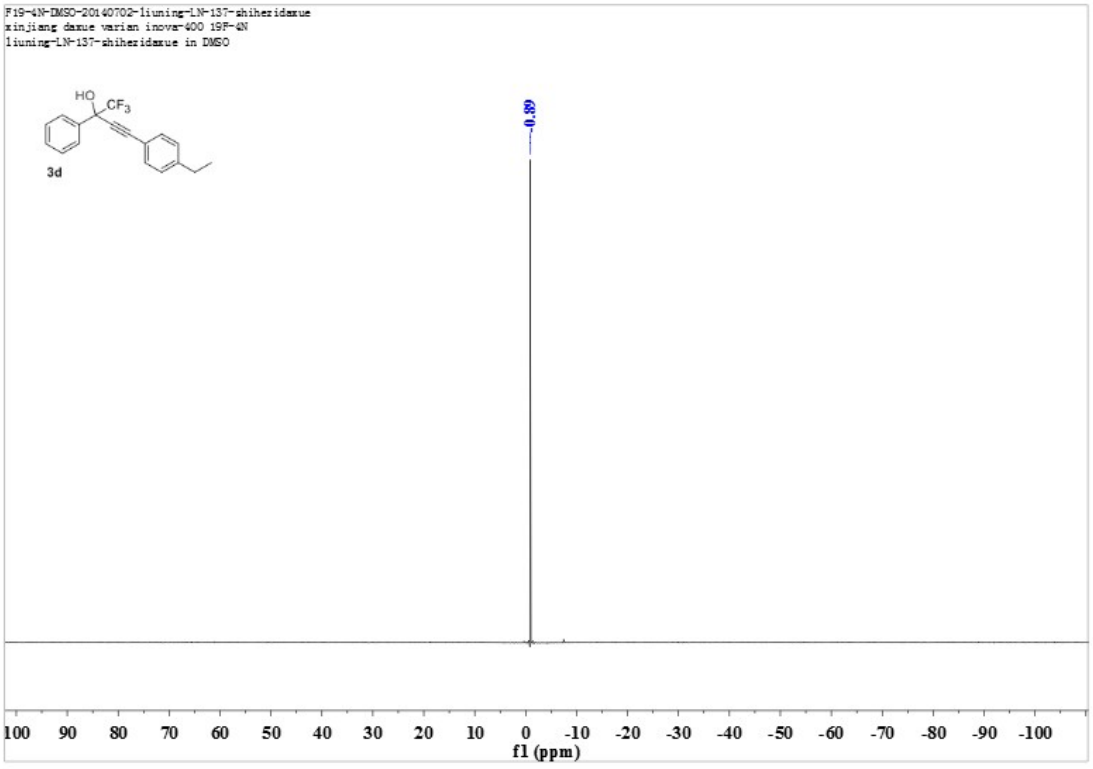
f1 (ppm)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xinjiang daxue varian inova-400 19F-4N
liuning-LN-137-shiheridaxue in DMSO
HO
CF3
3d
-0.89
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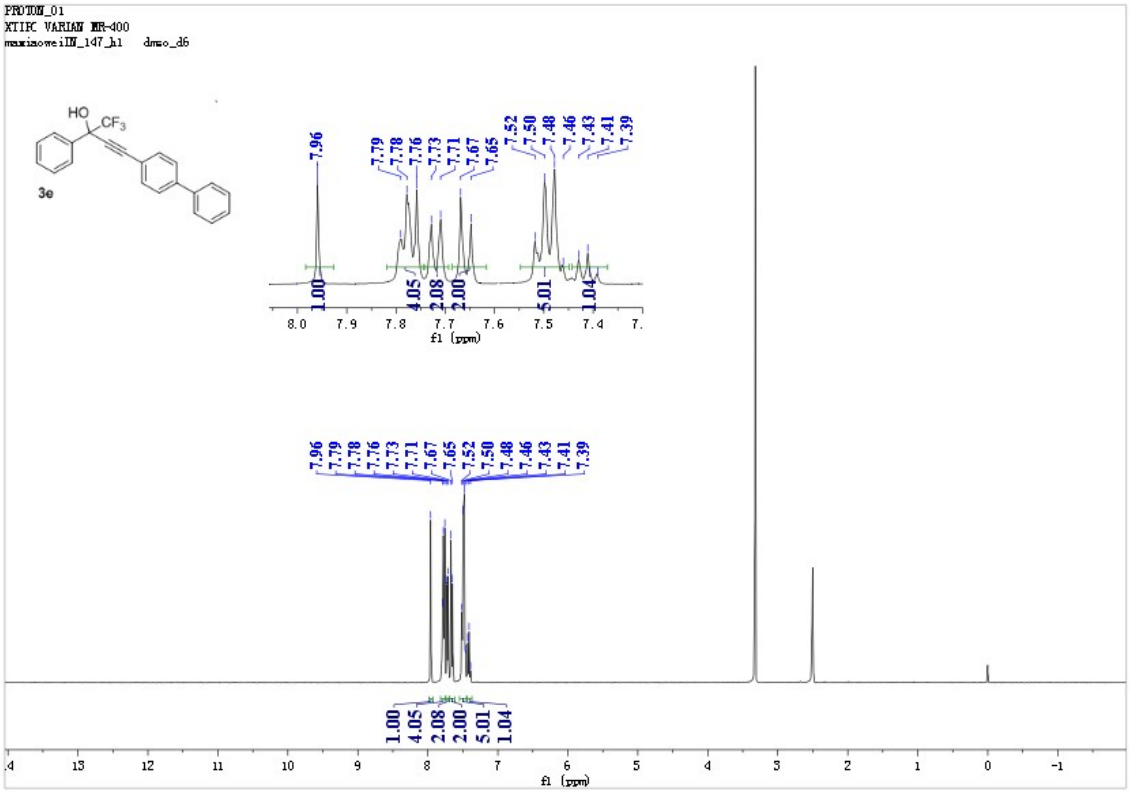
f1 (ppm)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CF3
3e
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2.00
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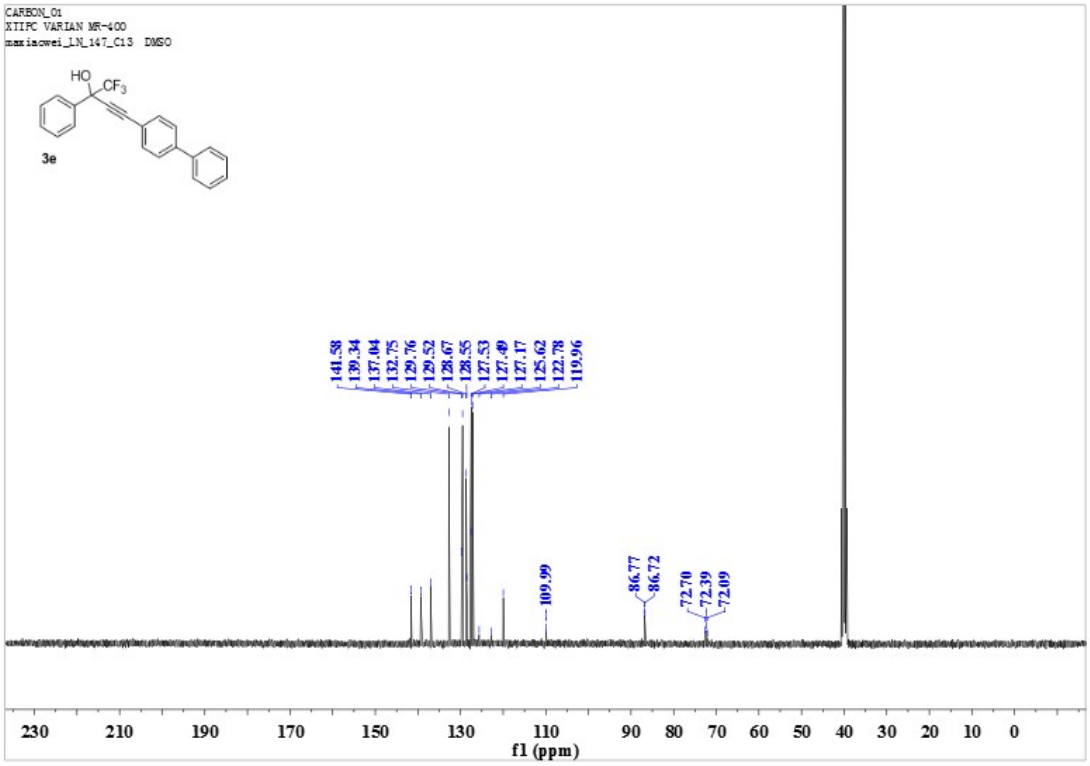
1.04
f1 (ppm)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CF3
3e
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128.55
127.53
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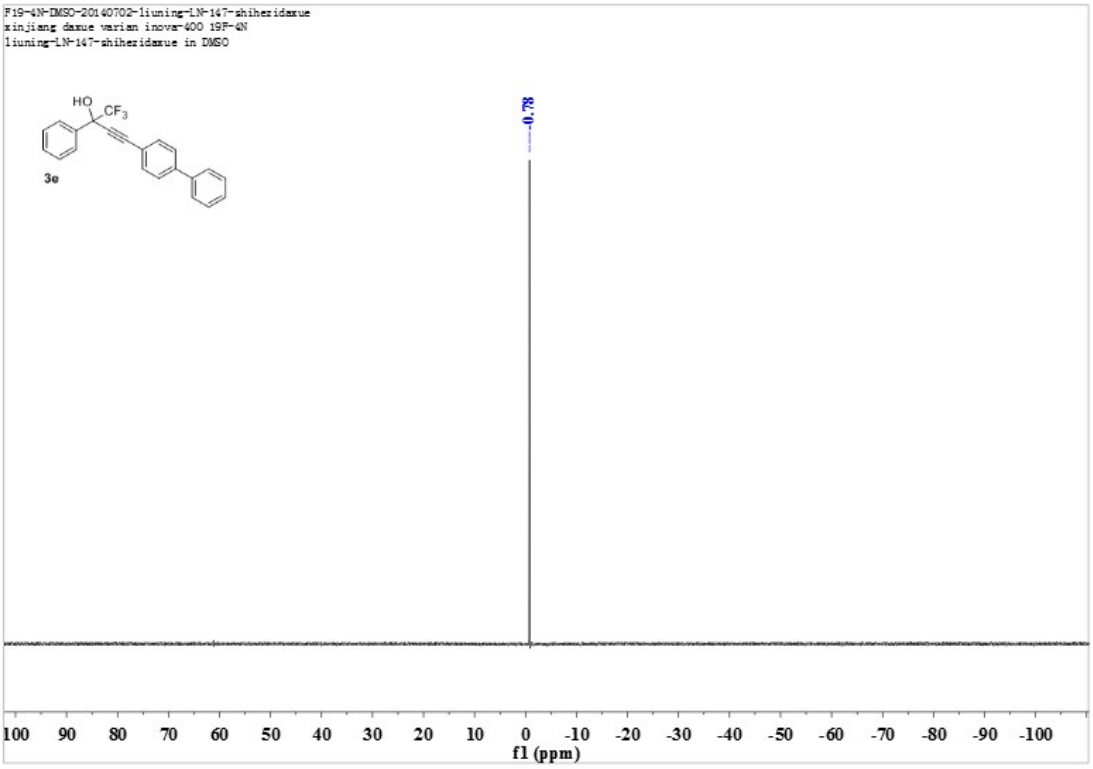
72.09
f1 (ppm)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liuning-LN-147-shiheridaxue in DMSO
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CF3
3e
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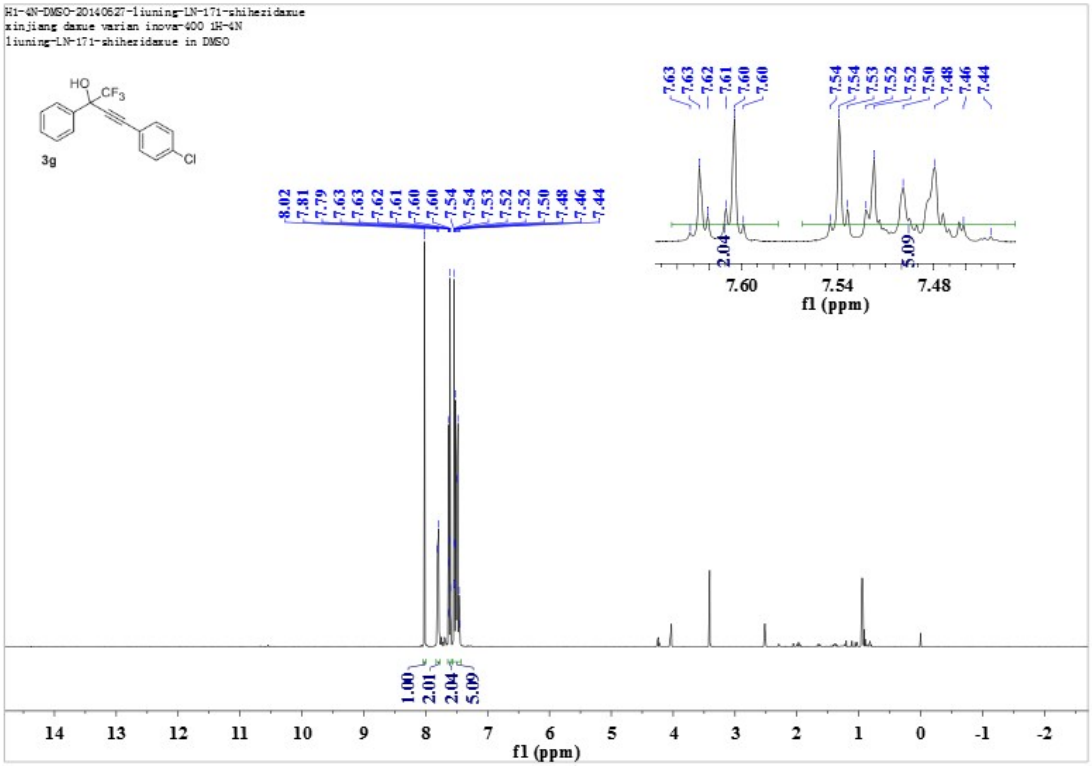
f1 (ppm)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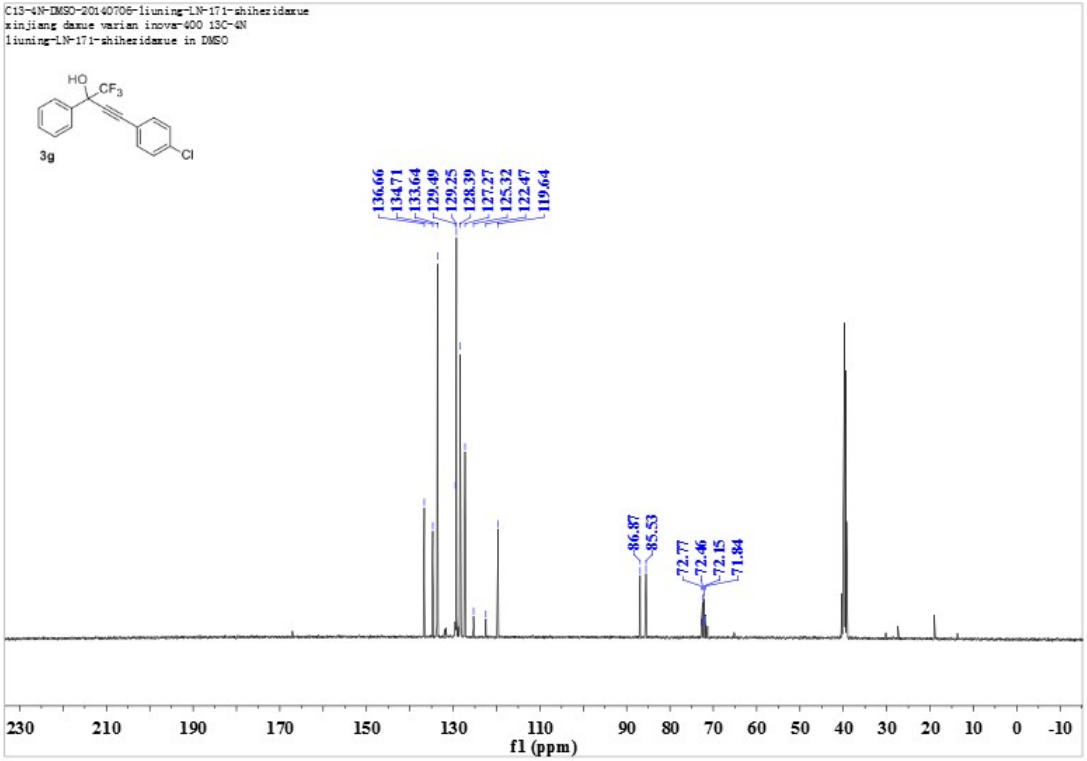
CF3
Cl
3g
8.02 7.81 7.79 7.63 7.63 7.62 7.61 7.60 7.60 7.54 7.54 7.53 7.52 7.52 7.50 7.48 7.46 7.44
1.00 2.01 2.04 5.09
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)
7.63 7.63 7.62 7.61 7.60 7.60
7.54 7.54 7.53 7.52 7.52 7.50 7.48 7.46 7.44
2.04
5.09
7.60 7.54 7.48
f1 (ppm)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CF3
Cl
3g
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133.64
129.49
129.25
128.39
127.27
125.32
122.47
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72.15
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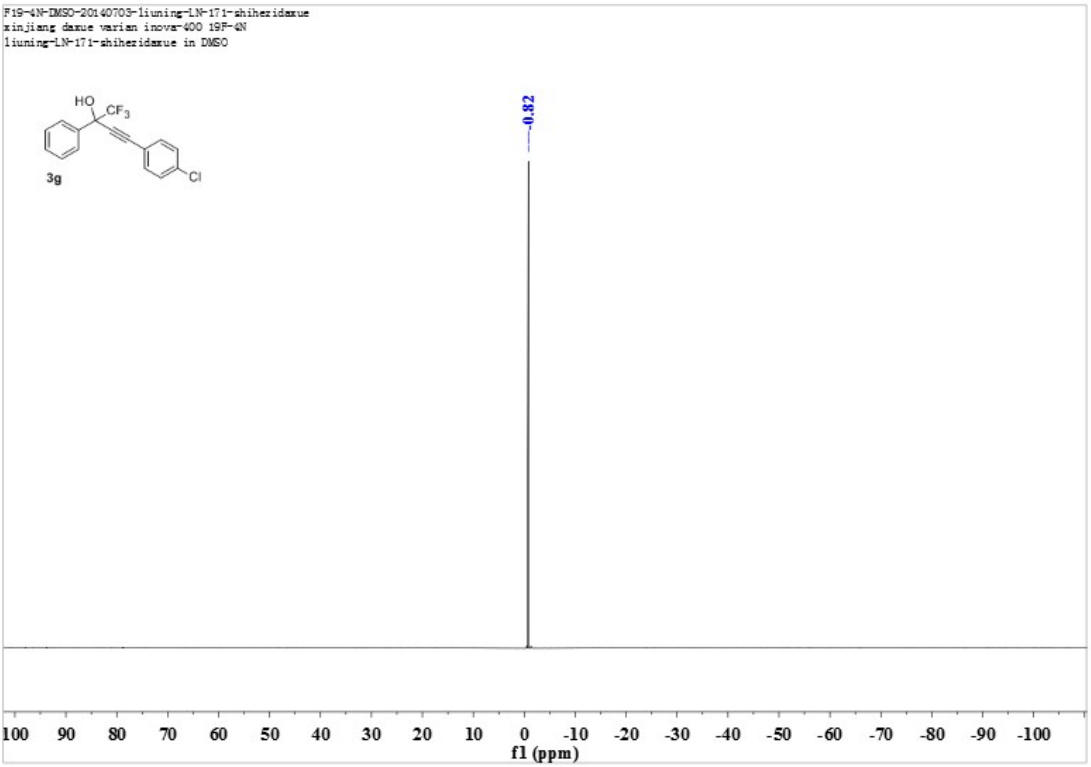
f1 (ppm)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CF3
Cl
3g
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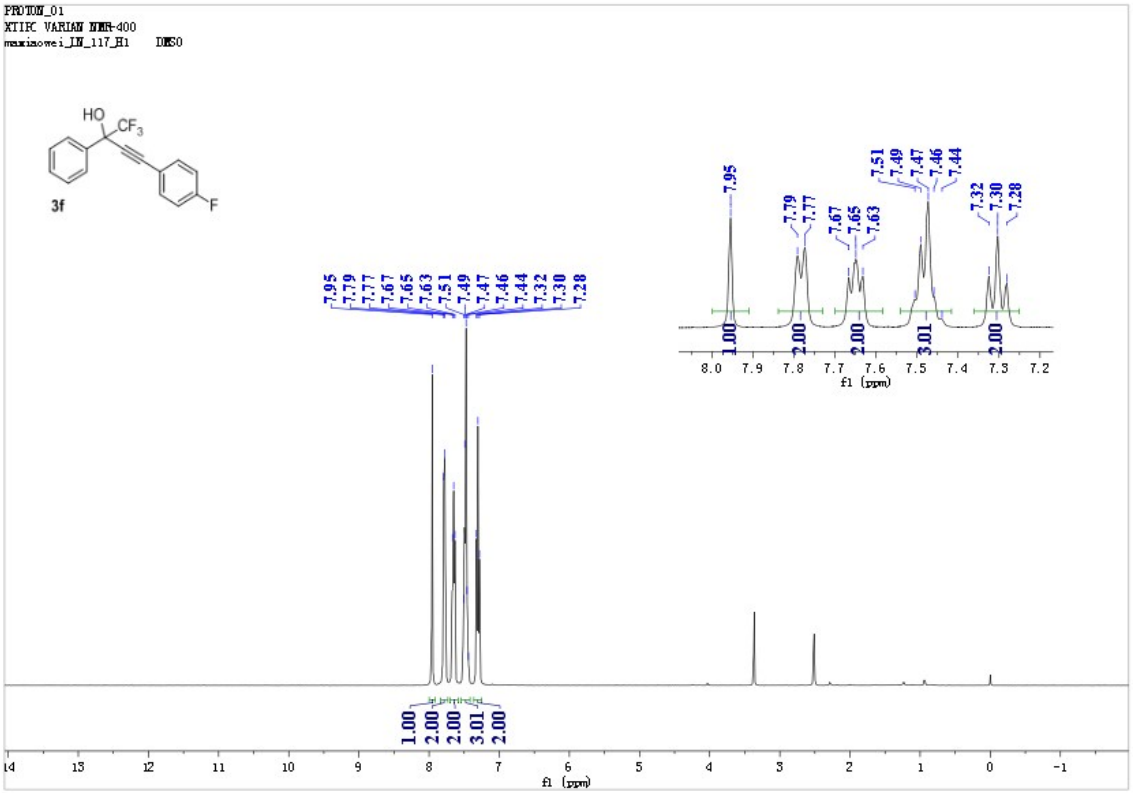
f1 (ppm)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CF3
F
3f
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7.79
7.77
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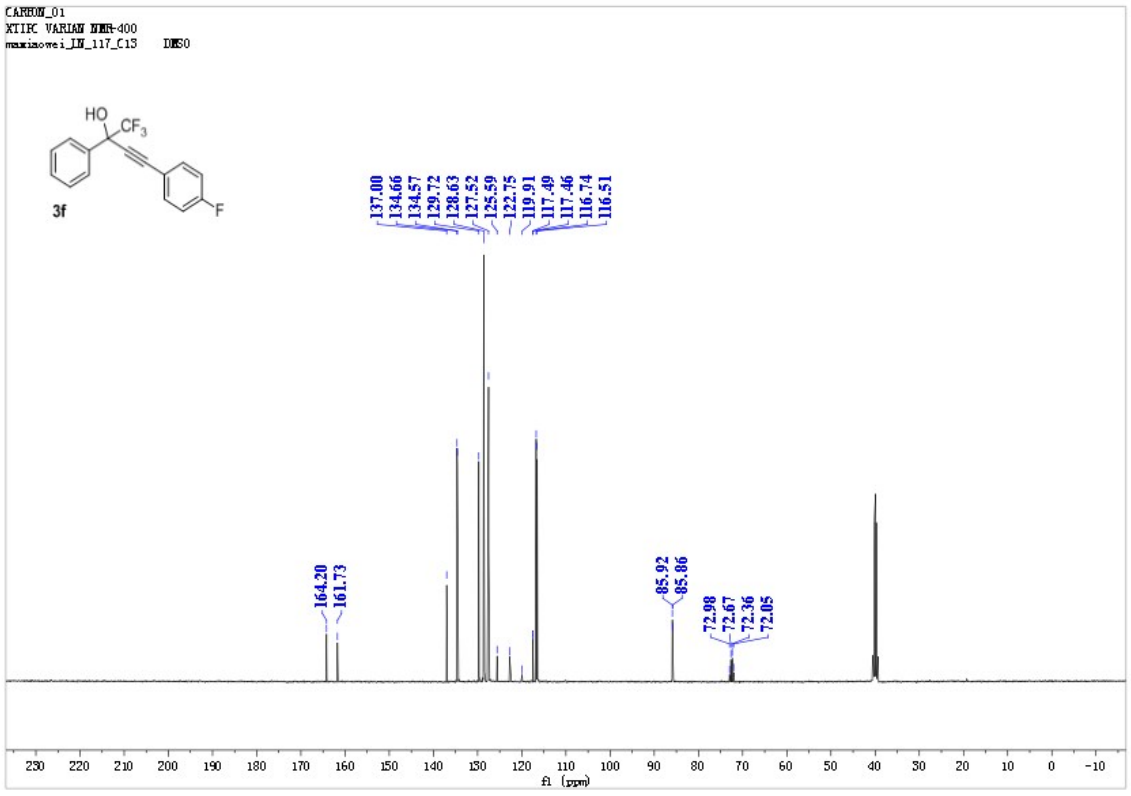
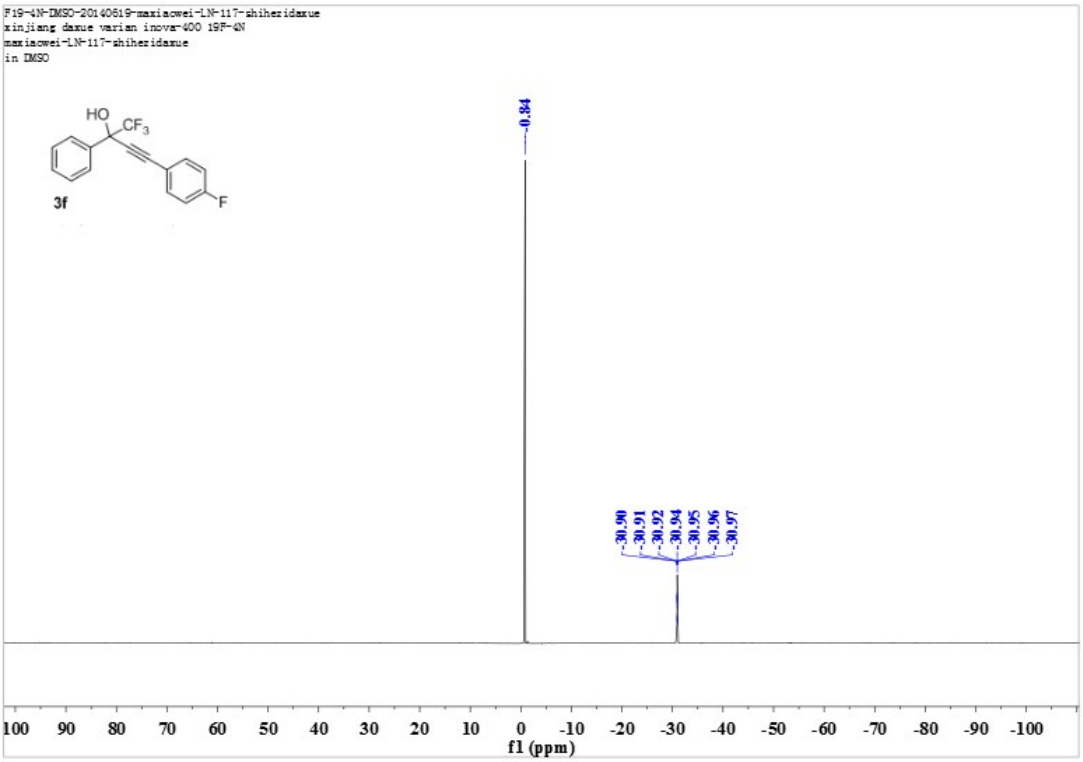
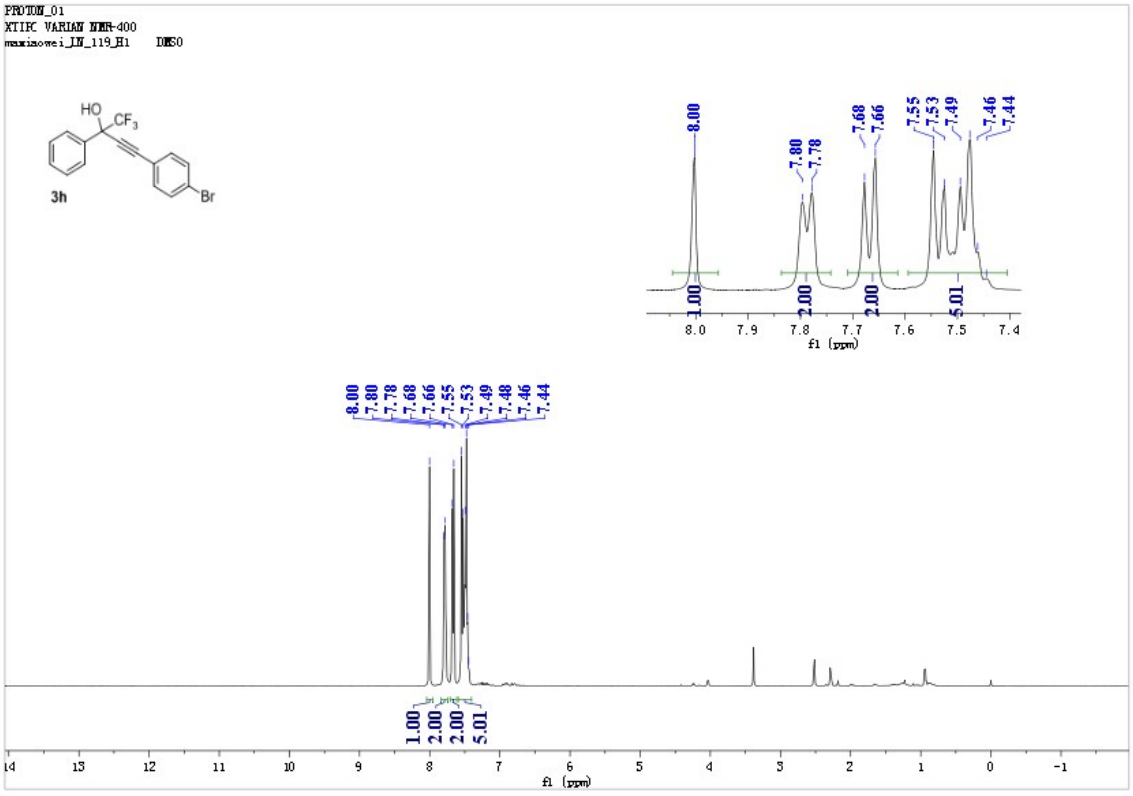
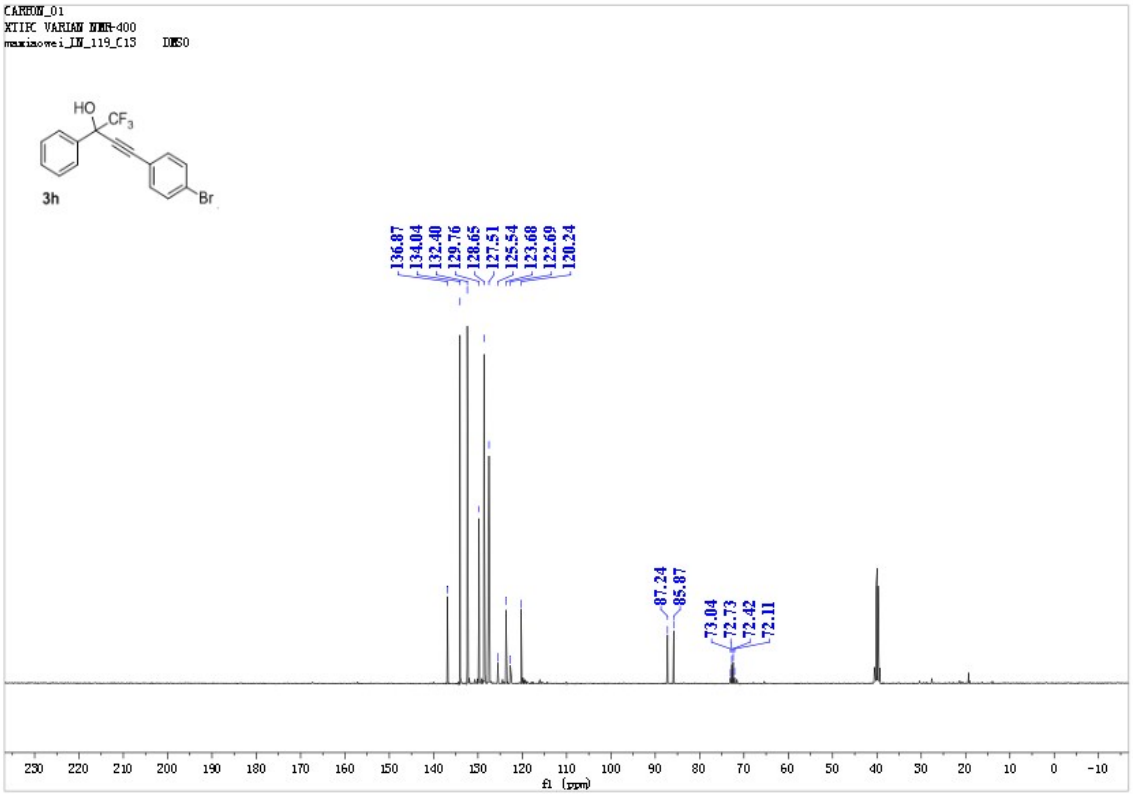
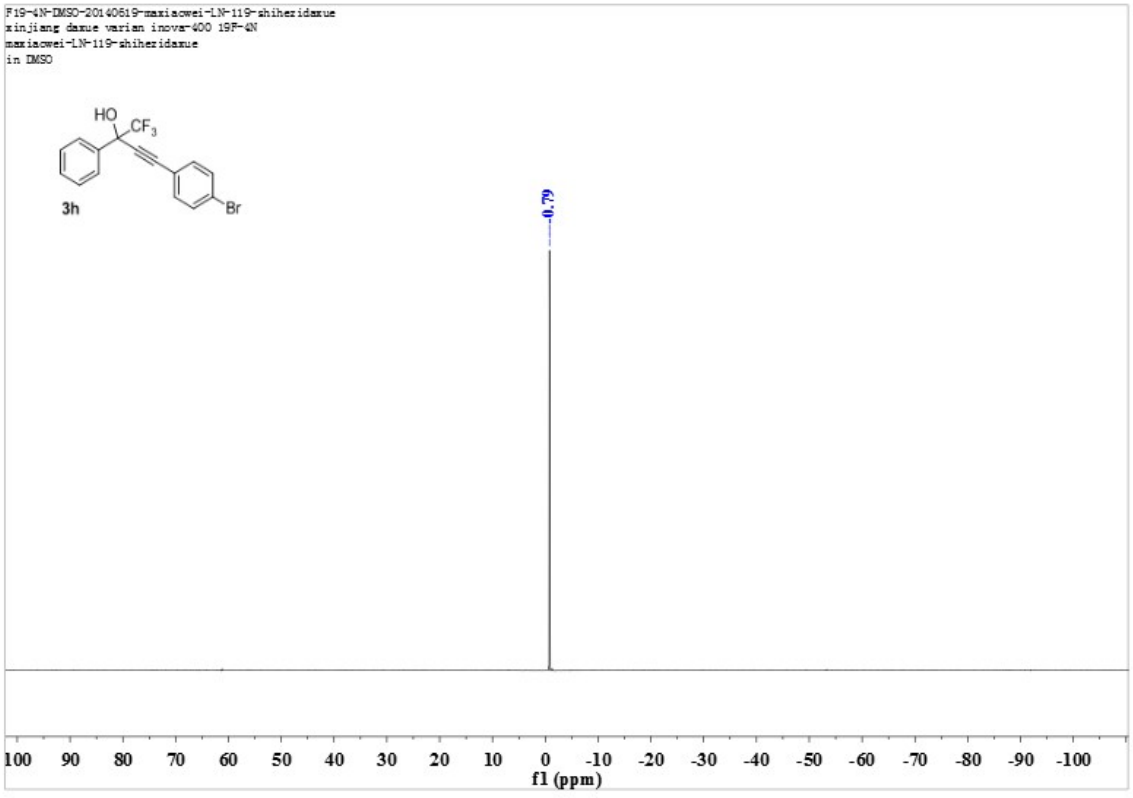
7.47
7.46
7.44
7.32
7.30
7.28
1.00
2.00
2.00
3.01
2.00
f1 (ppm)

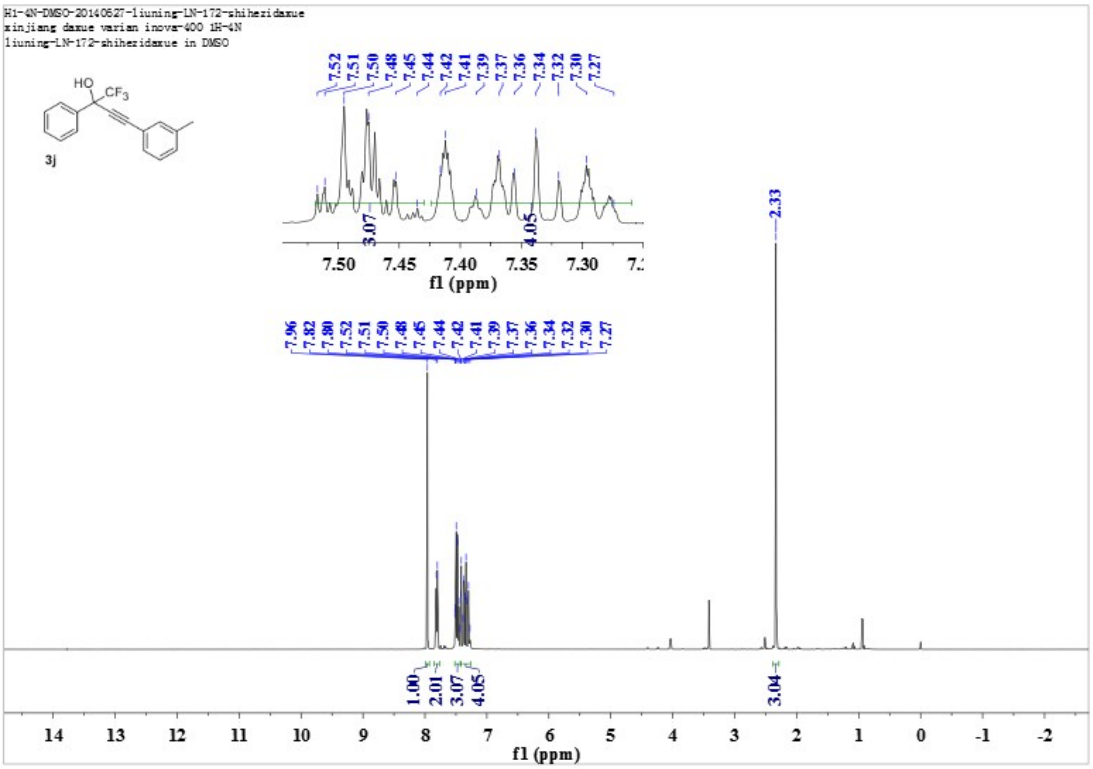

CARBON_01
XTIPC VARIAN NMR-400
mamiaowei_LN_117_C13 DMSO
HO
CF3
F
3f
137.00
134.66
134.57
129.72
128.63
127.52
125.59
122.75
119.91
117.49
117.46
116.74
116.51
164.20
161.73
85.92
85.86
72.98
72.67
72.36
72.05
f1 (ppm)

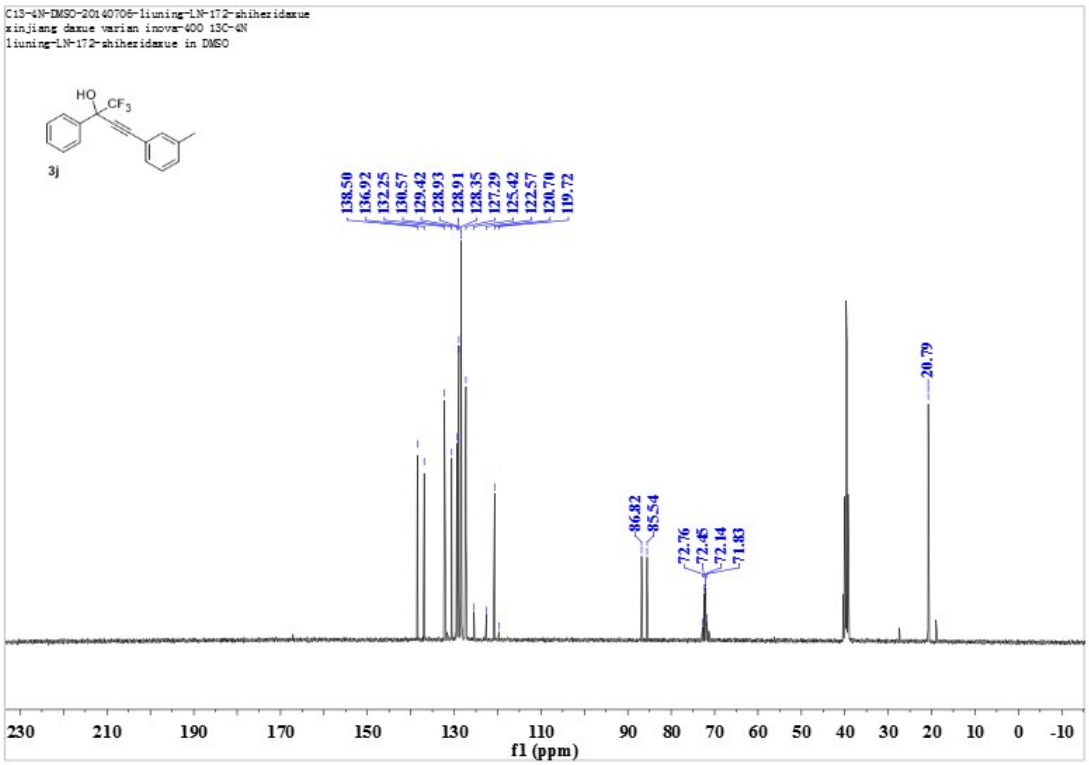

F19-4N-DMSO-20140619-maxiaowei-LN-117-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-117-shiheridaxue
in DMSO
HO
CF3
3f
F
-0.84
-30.90
-30.91
-30.92
-30.94
-30.95
-30.96
-30.97
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

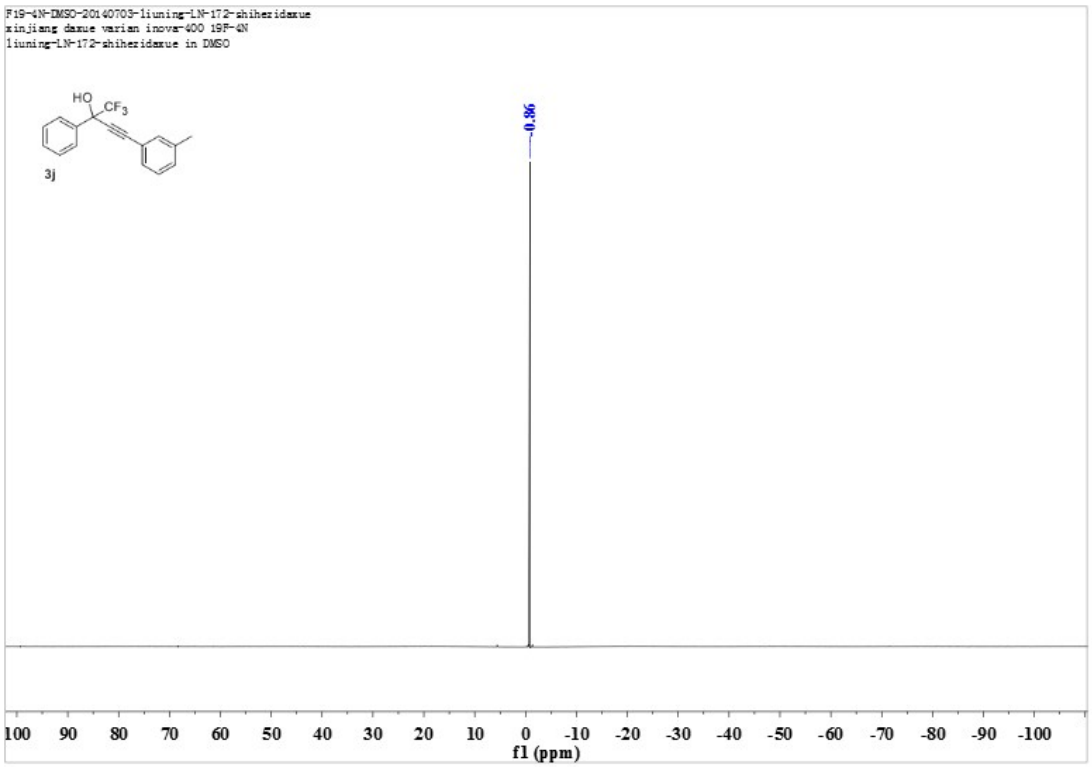

PROTON_01
XTIHC VARIAN NMR-400
maxiaowei_LN_119_H1 DMSO
HO
CF3
3h
Br
8.00
7.80
7.78
7.68
7.66
7.55
7.53
7.49
7.46
7.44
1.00
2.00
2.00
5.01
f1 (ppm)

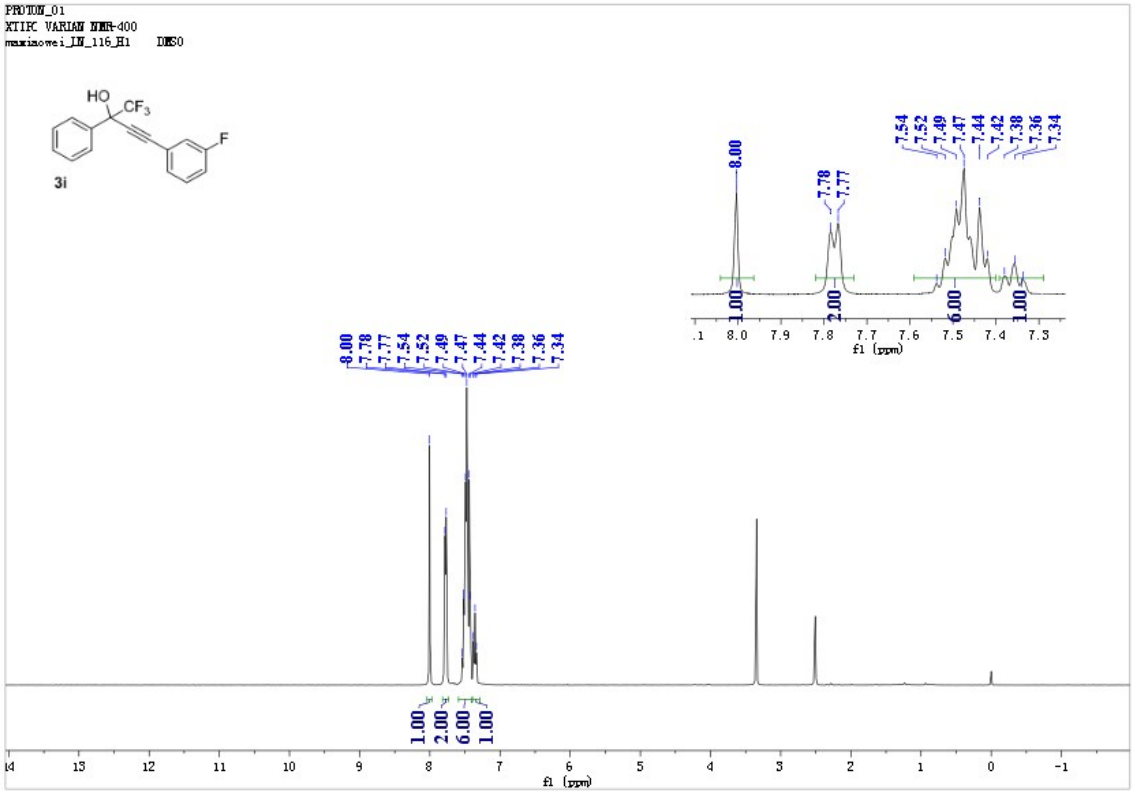

CARBON_01
XTIPC VARIAN NMR-400
maxiaowei_LN_119_C13 DMSO
HO
CF3
3h
Br
136.87
134.04
132.40
129.76
128.65
127.51
125.54
123.68
122.69
120.24
87.24
85.87
73.04
72.73
72.42
72.11
f1 (ppm)

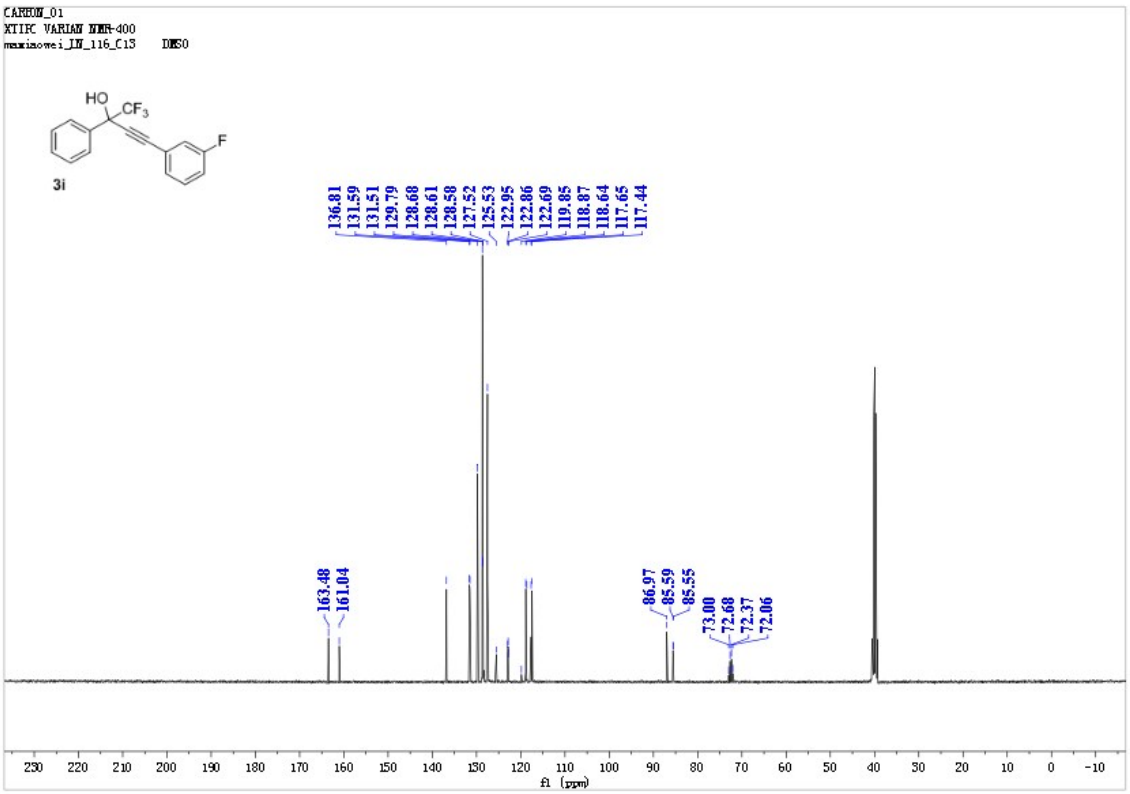

F19-4N-DMSO-20140619-maxiaowei-LN-119-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-119-shiheridaxue
in DMSO
HO
CF3
3h
Br
-0.79
f1 (ppm)

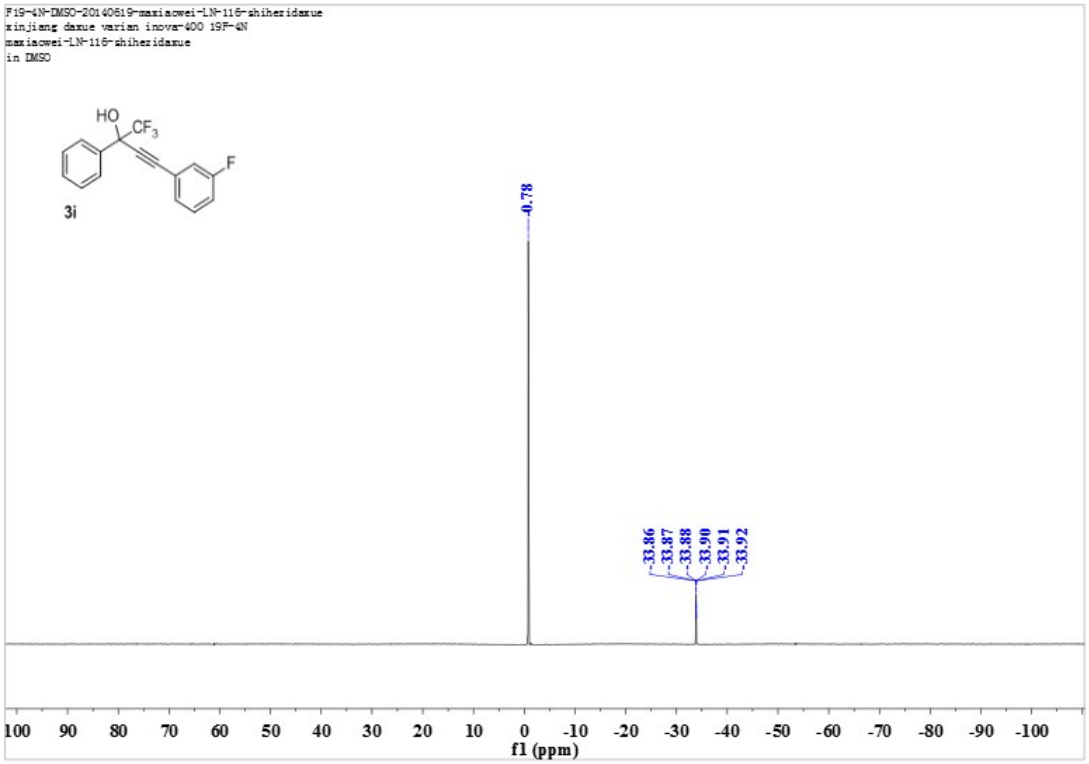

H1-4N-DMSO-20140627-liuning-LN-172-shiheridaxue
xinjiang daxue varian inova-400 1H-4N
liuning-LN-172-shiheridaxue in DMSO
HO
CF3
3j
7.52
7.51
7.50
7.48
7.45
7.44
7.42
7.41
7.39
7.37
7.36
7.34
7.32
7.30
7.27
3.07
4.05
2.33
1.00
2.01
3.07
4.05
3.04
f1 (ppm)

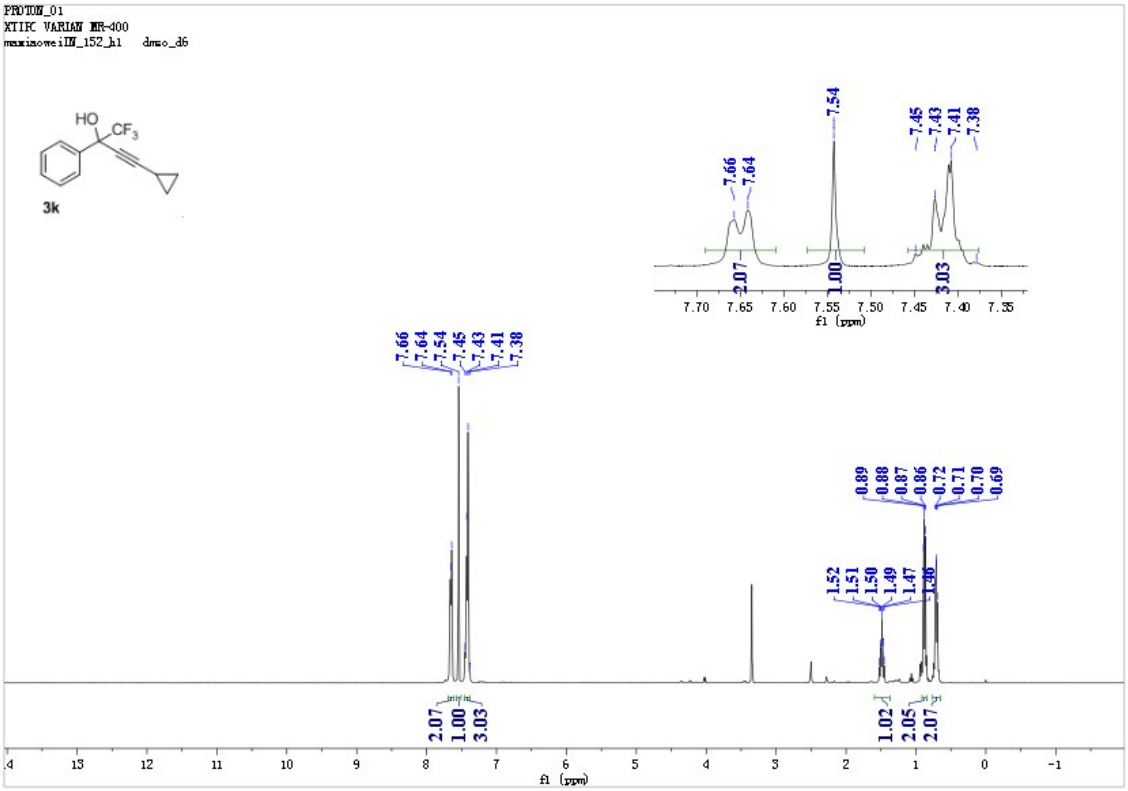

C13-4N-DMSO-20140706-liuning-LN-172-shiheridaxue
xinjiang daxue varian inova-400 13C-4N
liuning-LN-172-shiheridaxue in DMSO
HO
CF3
3j
138.50
136.92
132.25
130.57
129.42
128.93
128.91
128.35
127.29
125.42
122.57
120.70
119.72
86.82
85.54
72.76
72.45
72.14
71.83
20.79
f1 (ppm)

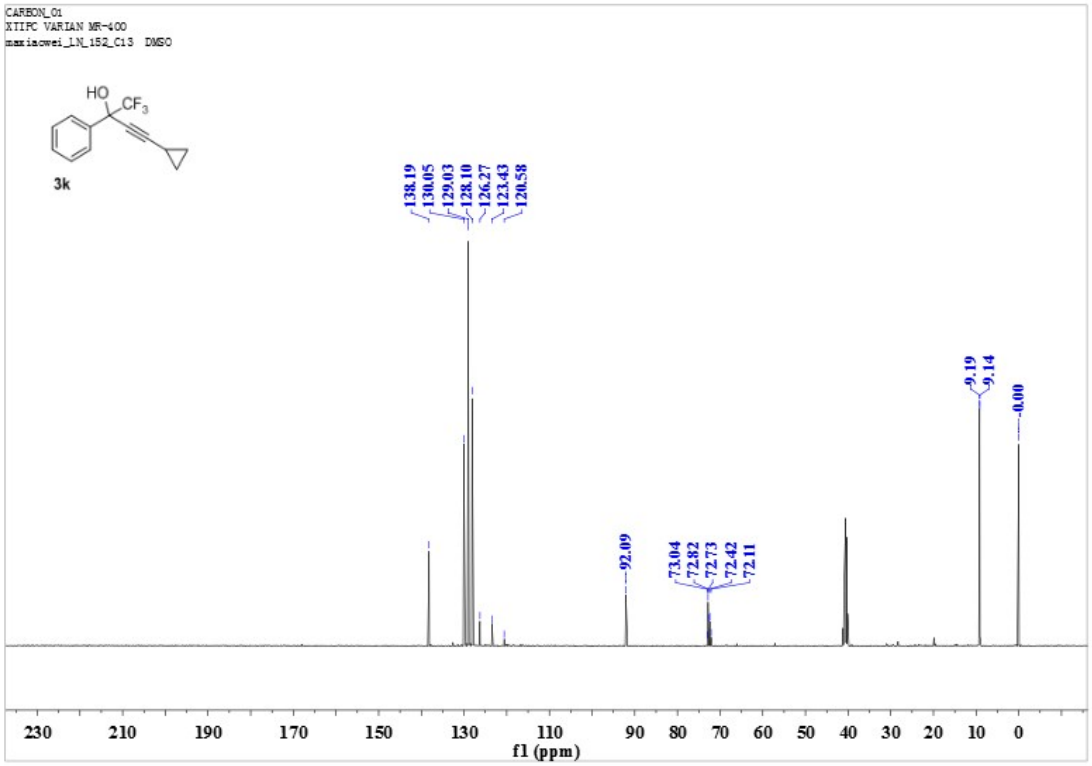

F19-4N-DMSO-20140703-liuning-LN-172-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-172-shiheridaxue in DMSO
HO
CF3
3j
-0.86
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

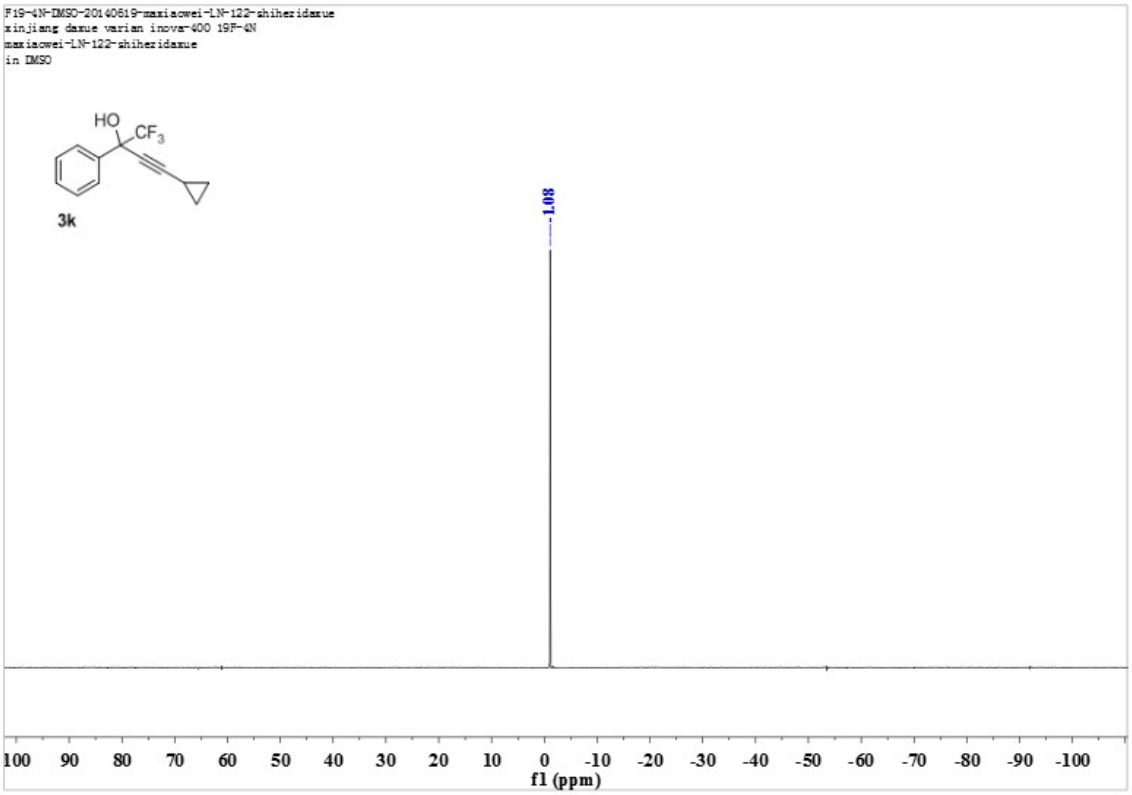

PROTON_01
XTIIC VARIAN NMR-400
maximowei_LN_116_H1 DMSO
HO
CF3
F
3i
8.00
7.78
7.77
7.54
7.52
7.49
7.47
7.44
7.42
7.38
7.36
7.34
1.00
2.00
6.00
1.00
f1 (ppm)

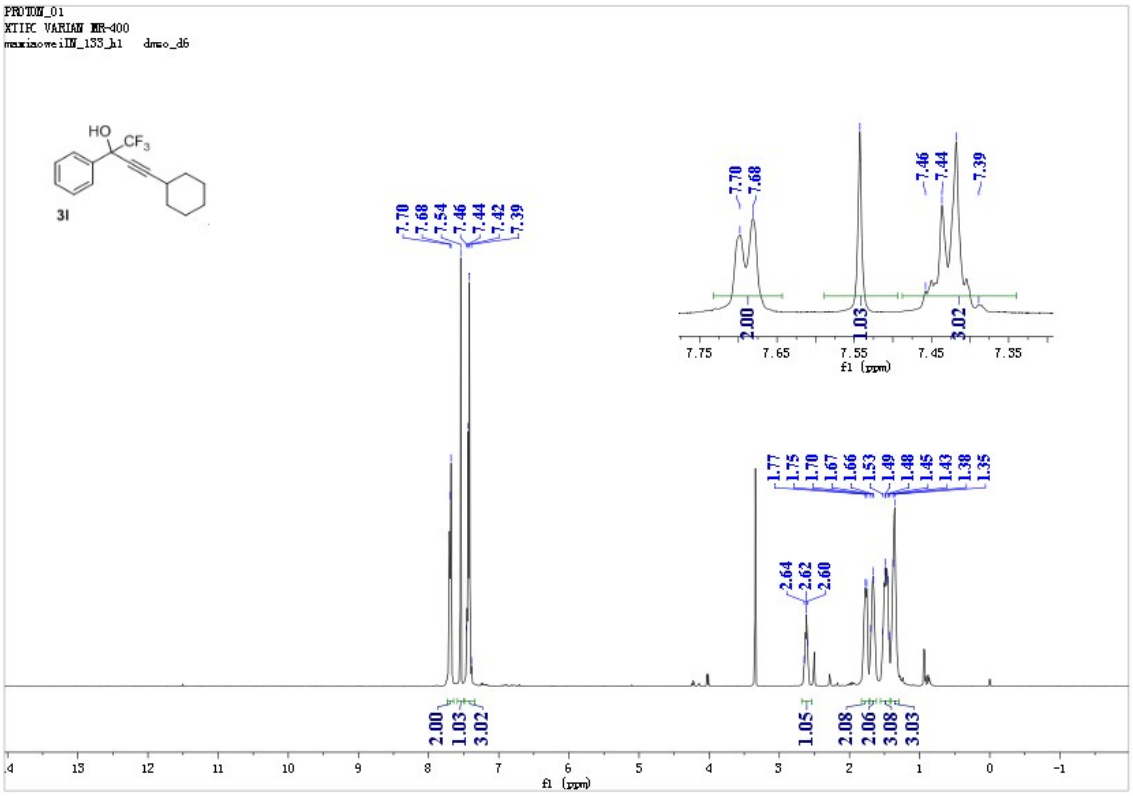

CARBON_01
XTIPC VARIAN NMR-400
maxiaowei_LN_116_C13 DMSO
HO
CF3
F
3i
136.81
131.59
131.51
129.79
128.68
128.61
128.58
127.52
125.53
122.95
122.86
122.69
119.85
118.87
118.64
117.65
117.44
163.48
161.04
86.97
85.59
85.55
73.00
72.68
72.37
72.06
f1 (ppm)

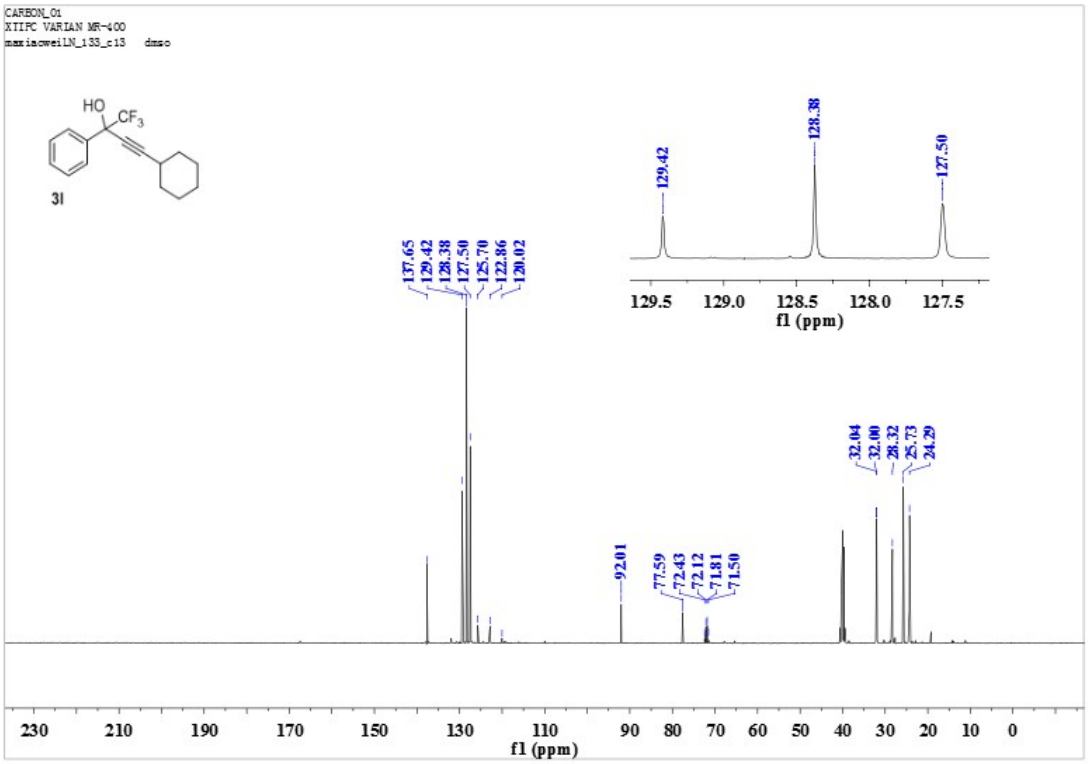

F19-4N-DMSO-20140619-maxiaowei-LN-116-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-116-shiheridaxue
in DMSO
HO
CF3
F
3i
-0.78
-33.86
-33.87
-33.88
-33.90
-33.91
-33.92
f1 (ppm)

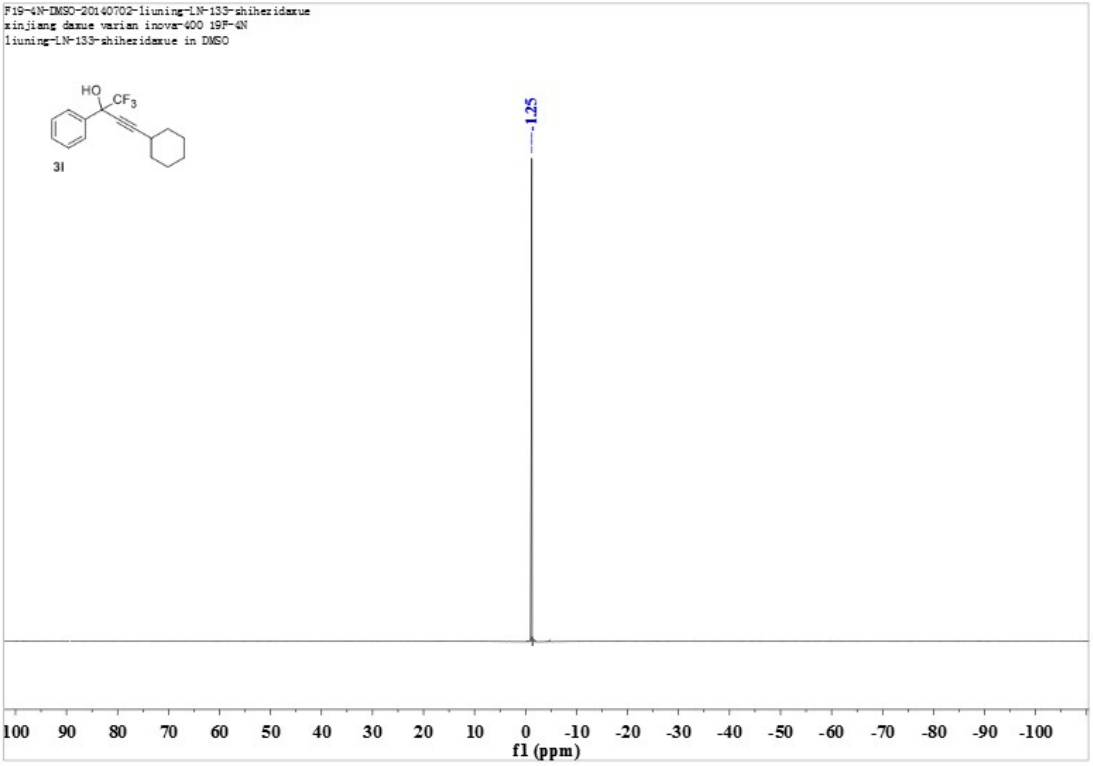

PROTON_01
XTIPC VARIAN MR-400
maxiaowei1N_152_h1 dmso_d6
HO
CF3
3k
7.66
7.64
7.54
7.45
7.43
7.41
7.38
2.07
1.00
3.03
0.89
0.88
0.87
0.86
0.72
0.71
0.70
0.69
1.52
1.51
1.50
1.49
1.47
1.36
1.02
2.05
2.07
f1 (ppm)

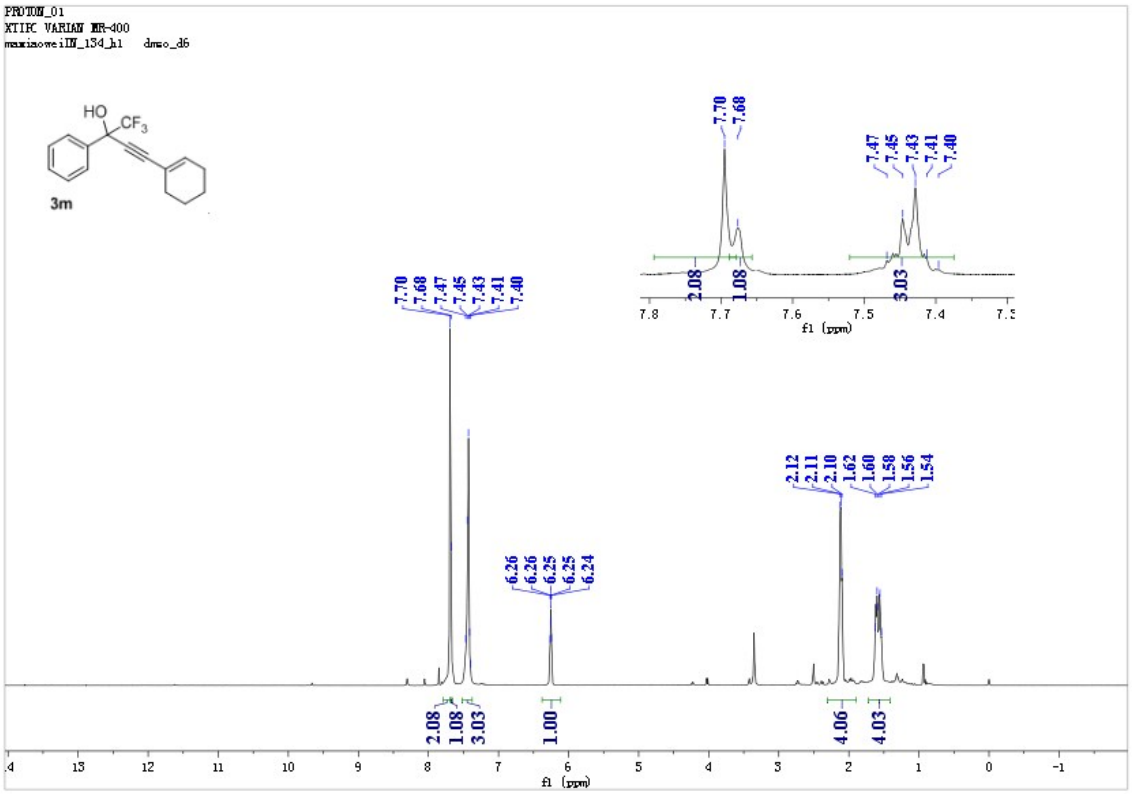

CARBON_01
XTIPC VARIAN MR-400
maxiaowei_1N_152_C13 DMSO
HO
CF3
3k
138.19
130.05
129.03
128.10
126.27
123.43
120.58
92.09
73.04
72.82
72.73
72.42
72.11
9.19
9.14
0.00
f1 (ppm)

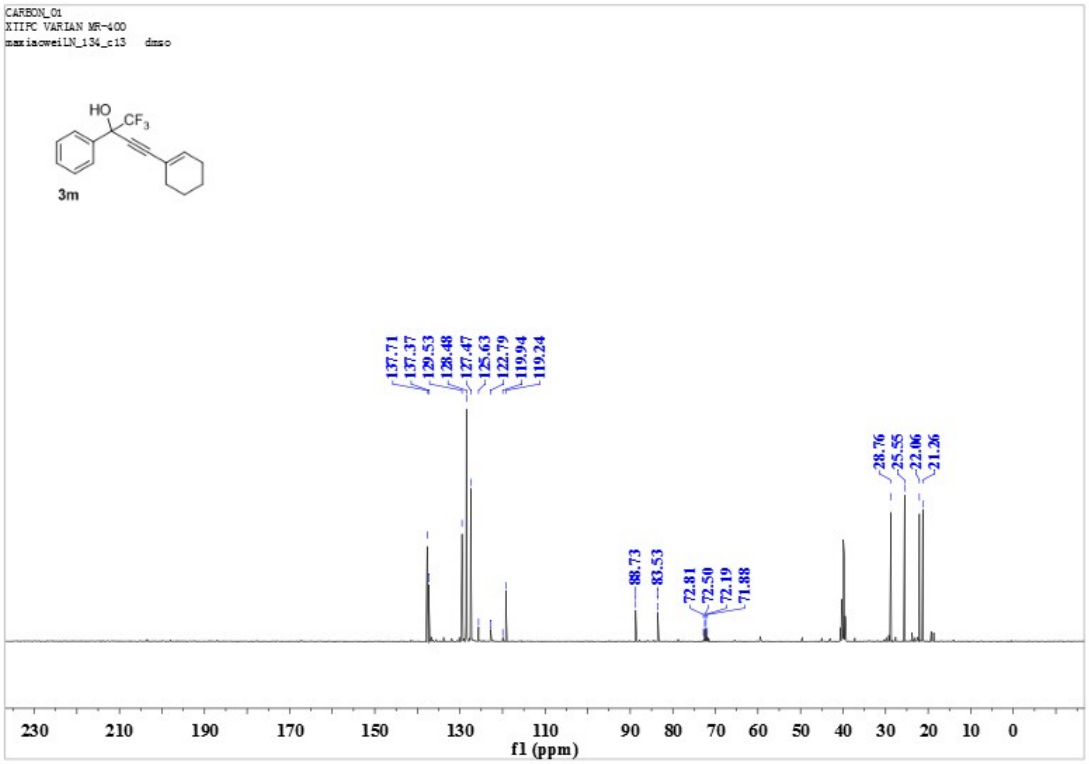

F19-4N-DMSO-20140619-maxiaowei-LN-122-shihezidaxue
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-122-shihezidaxue
in DMSO
HO
CF3
3k
-1.08
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

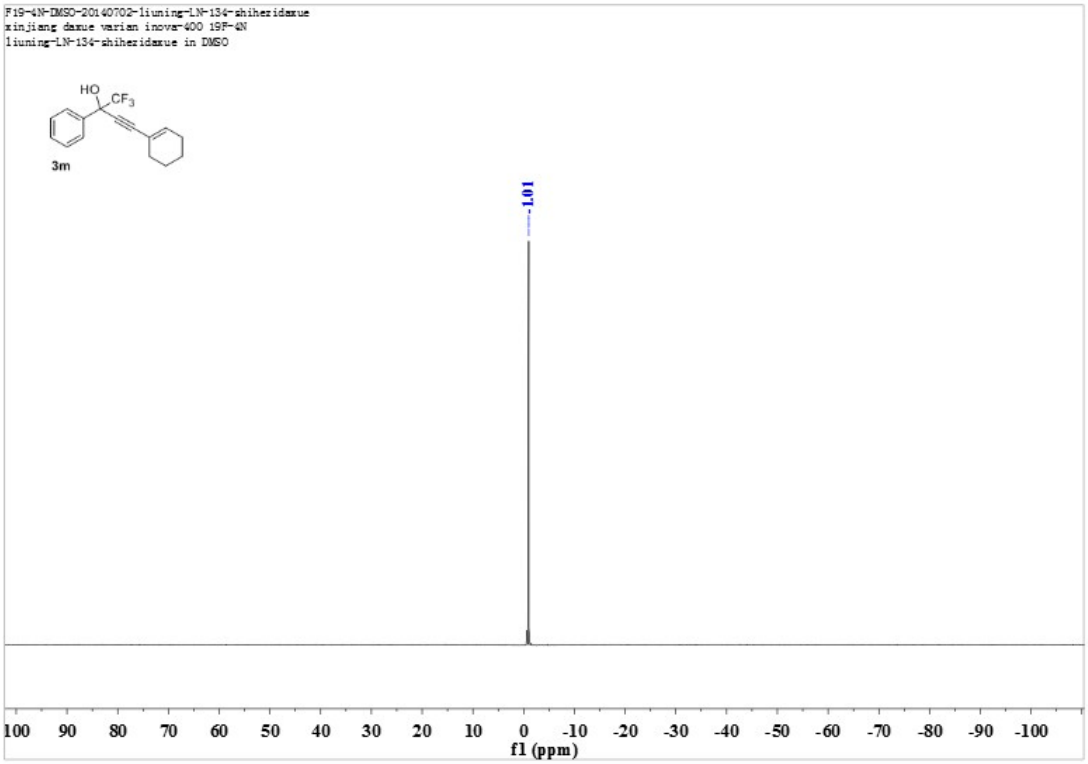

PROTON_01
XTIHC VARIAN MR-400
maxiaoweiLN_133_h1 dmso_d6
HO
CF3
3l
7.70
7.68
7.54
7.46
7.44
7.42
7.39
2.00
1.03
3.02
7.75 7.65 7.55 7.45 7.35
f1 (ppm)
1.77
1.75
1.70
1.67
1.66
1.53
1.49
1.48
1.45
1.43
1.38
1.35
2.64
2.62
2.60
1.05
2.08
2.06
3.08
3.03
f1 (ppm)

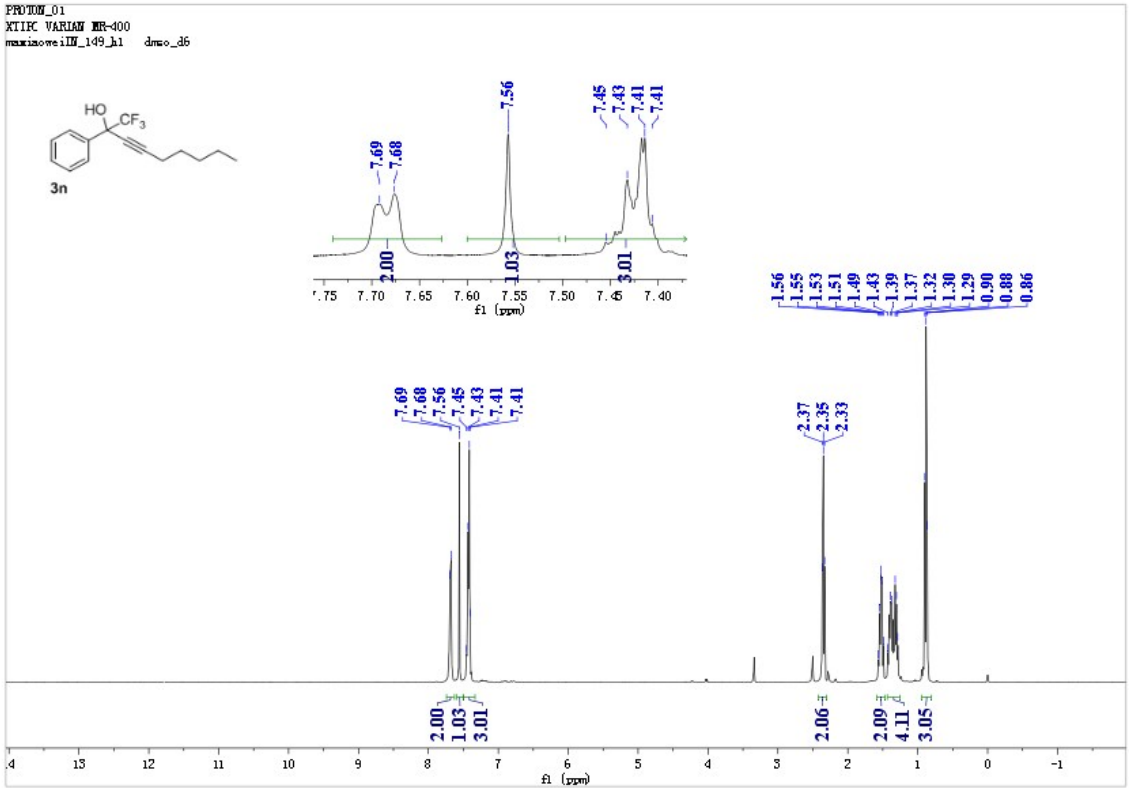

CARBON_01
XTIFC VARIAN MR-400
maxiaoweiLN_133_c13 dmso
HO
CF3
3l
137.65
129.42
128.38
127.50
125.70
122.86
120.02
92.01
77.59
72.43
72.12
71.81
71.50
32.04
32.00
28.32
25.73
24.29
129.42
128.38
127.50
129.5 129.0 128.5 128.0 127.5
f1 (ppm)
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

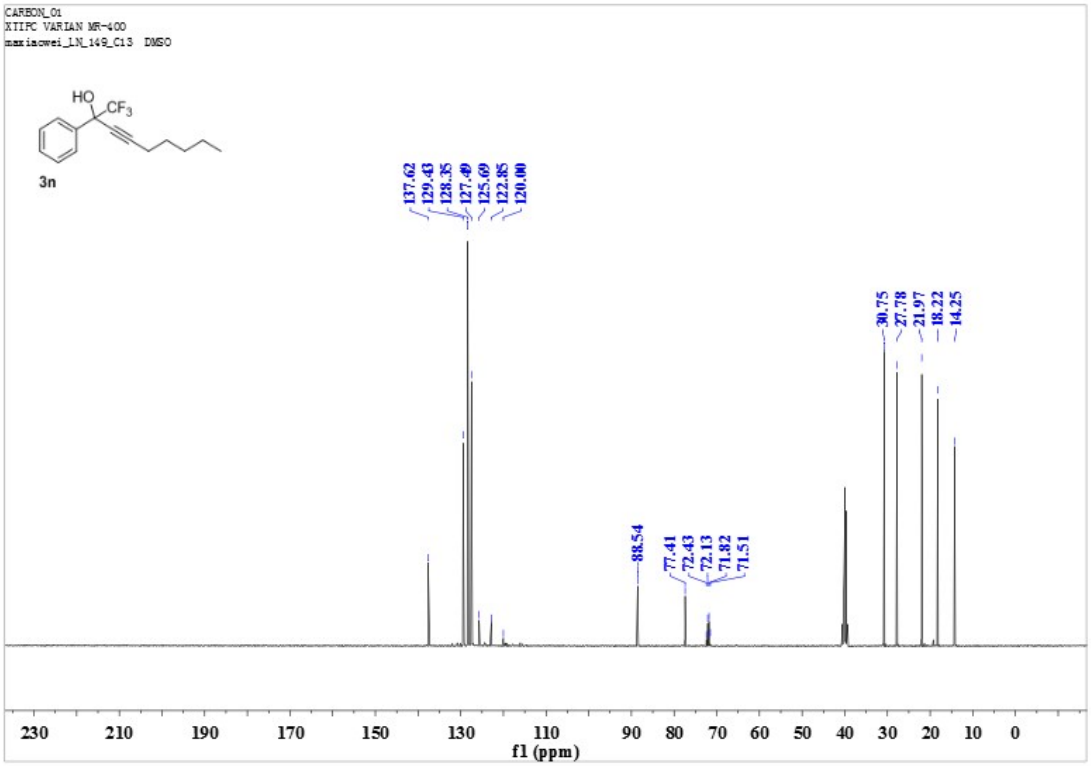

F19-4N-DMSO-20140702-liuning-LN-133-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-133-shiheridaxue in DMSO
HO
CF3
3l
-1.25
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

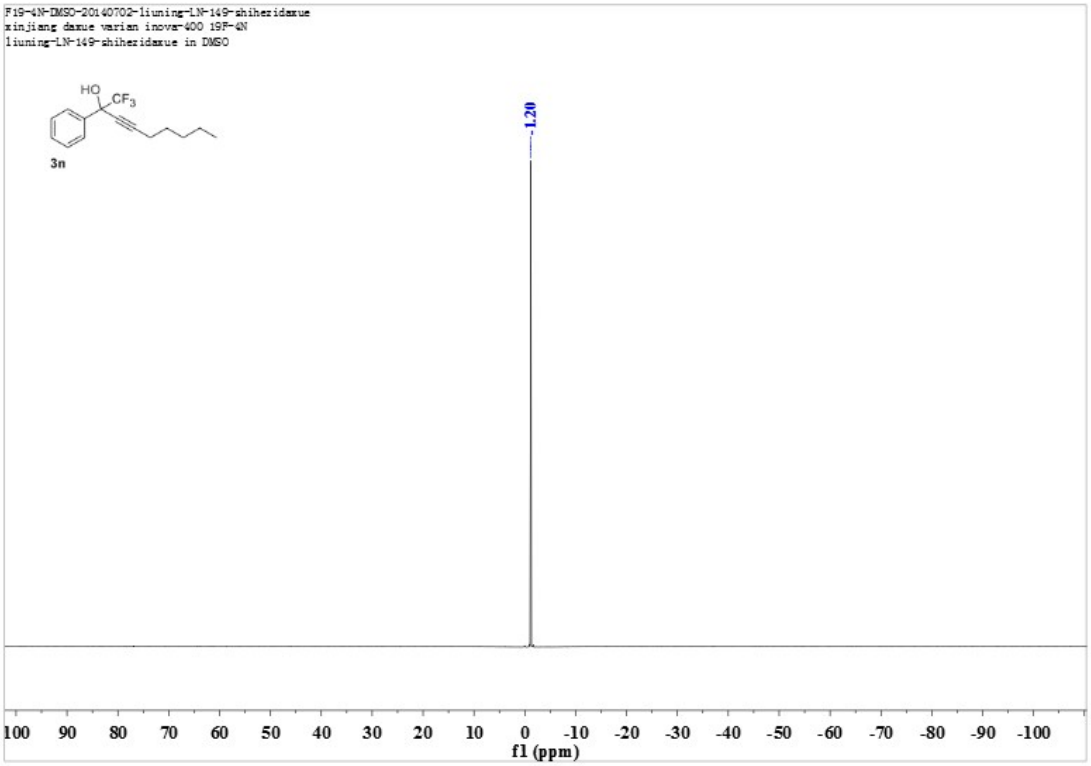

PROTON_01
XTIPC VARIAN MR-400
maxiaoweiLN_134_h1 dmso_d6
HO
CF3
3m
7.70
7.68
7.47
7.45
7.43
7.41
7.40
2.08
1.08
3.03
6.26
6.25
6.24
2.12
2.11
2.10
1.62
1.60
1.58
1.56
1.54
1.00
4.06
4.03
f1 (ppm)

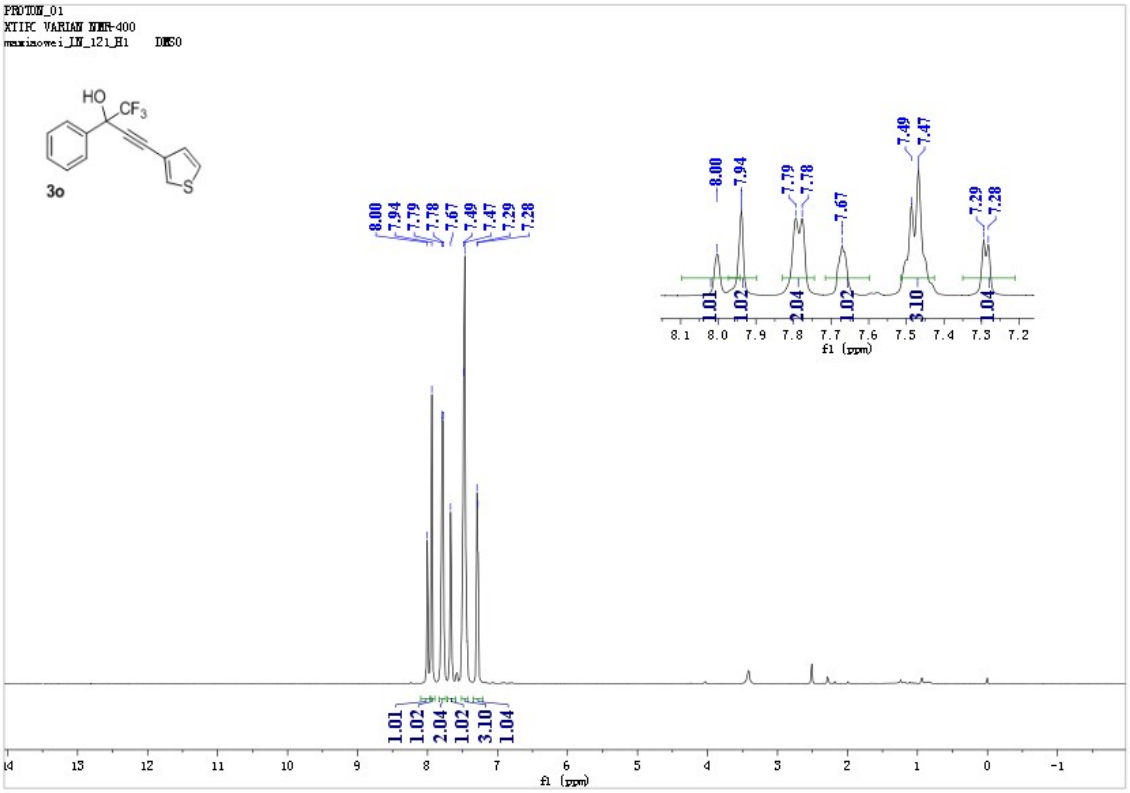

CARBON_01
XTIPC VARIAN MR-400
maxiaoweiLN_134_c13 dmso
HO
CF3
3m
137.71
137.37
129.53
128.48
127.47
125.63
122.79
119.94
119.24
88.73
83.53
72.81
72.50
72.19
71.88
28.76
25.55
22.06
21.26
f1 (ppm)

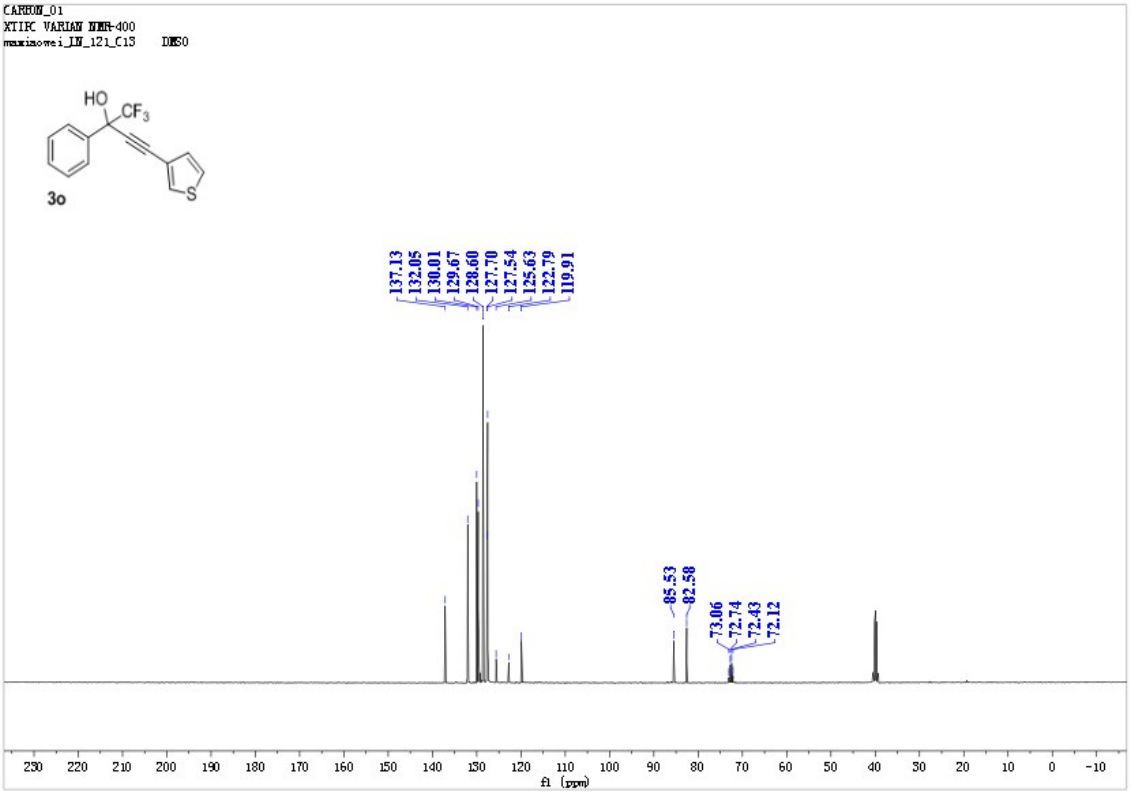

F19-4N-DMSO-20140702-liuning-LN-134-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-134-shiheridaxue in DMSO
HO
CF3
3m
-1.01
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

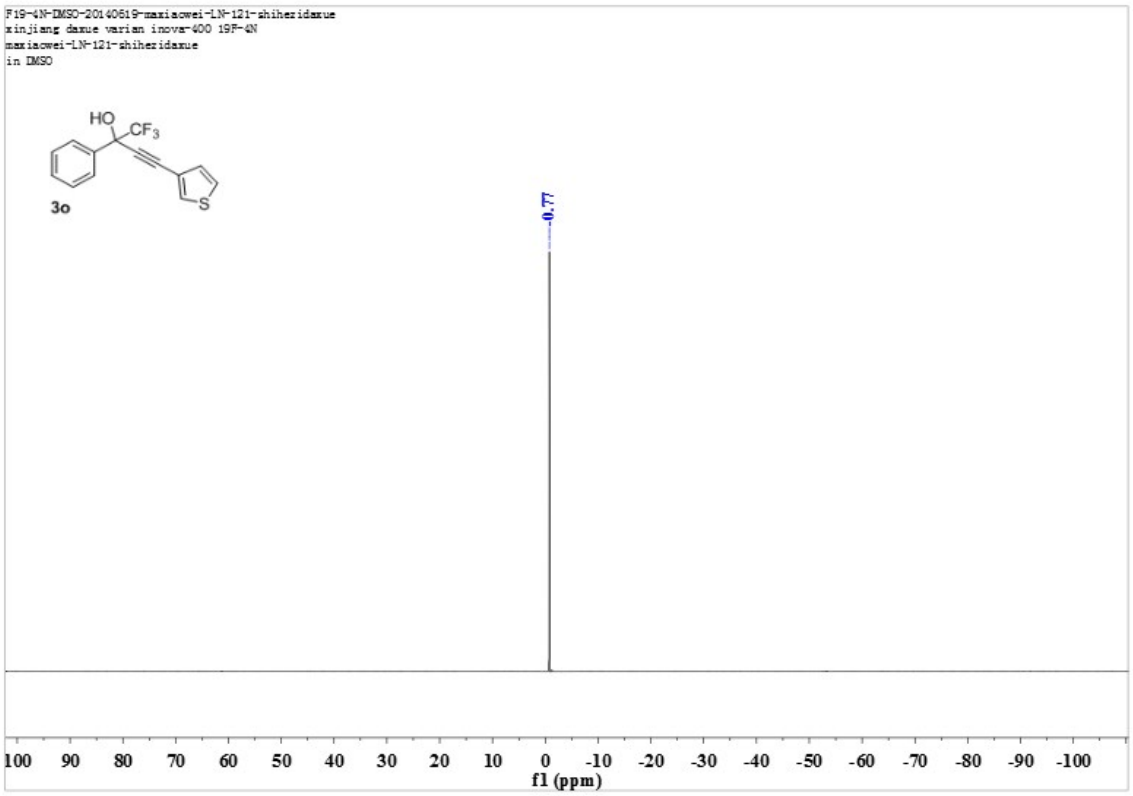

PROTON_01
XTIPC VARIAN MR-400
maxiaoweiLN_149_h1 dmso_d6
HO
CF3
3n
7.69
7.68
7.56
7.45
7.43
7.41
7.41
2.00
1.03
3.01
7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40
f1 (ppm)
1.56
1.55
1.53
1.51
1.49
1.43
1.39
1.37
1.32
1.30
1.29
0.90
0.88
0.86
2.37
2.35
2.33
2.00
1.03
3.01
2.06
2.09
4.11
3.05
f1 (ppm)

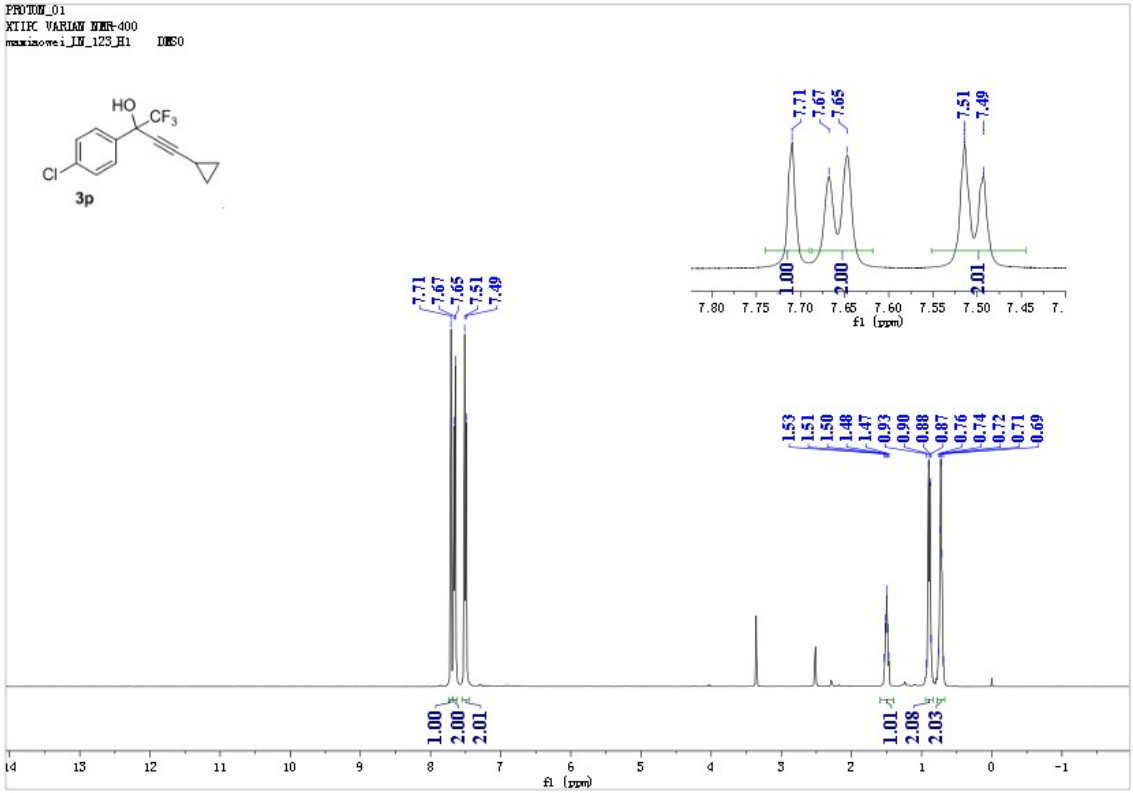

CARBON_01
XTIPC VARIAN MR-400
maxiaowei_LN_149_C13 DMSO
HO
CF3
3n
137.62
129.43
128.35
127.49
125.69
122.85
120.00
88.54
77.41
72.43
72.13
71.82
71.51
30.75
27.78
21.97
18.22
14.25
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

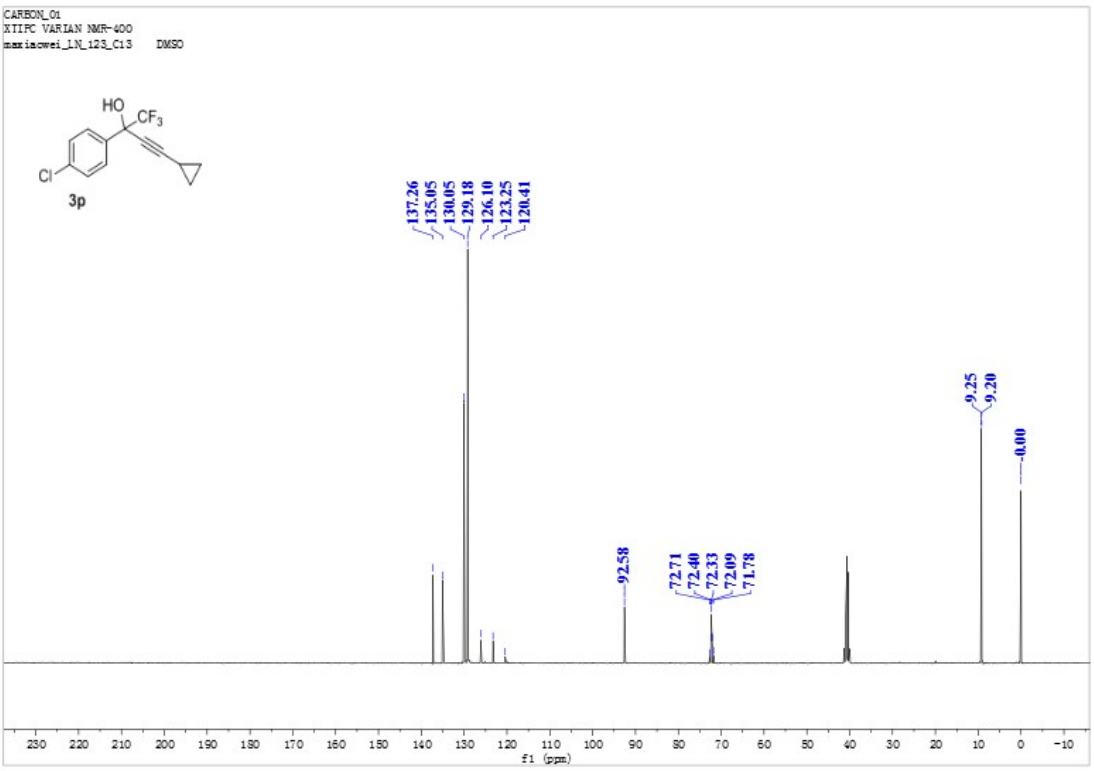

F19-4N-DMSO-20140702-liuning-LN-149-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-149-shiheridaxue in DMSO
HO
CF3
3n
-1.20
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

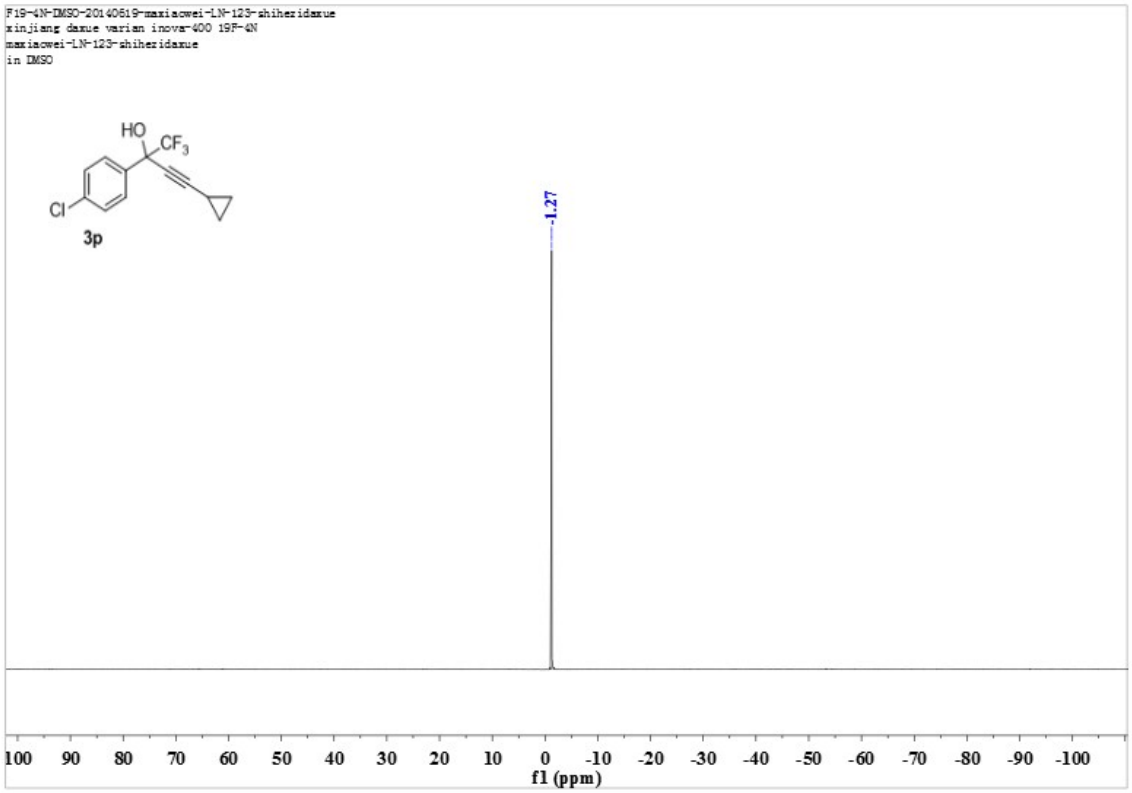

PROTON_01
XTIPC VARIAN NMR-400
maxiaowei_LW_121_H1 DMSO
HO
CF3
S
3o
8.00
7.94
7.79
7.78
7.67
7.49
7.47
7.29
7.28
1.01
1.02
2.04
1.02
3.10
1.04
f1 (ppm)

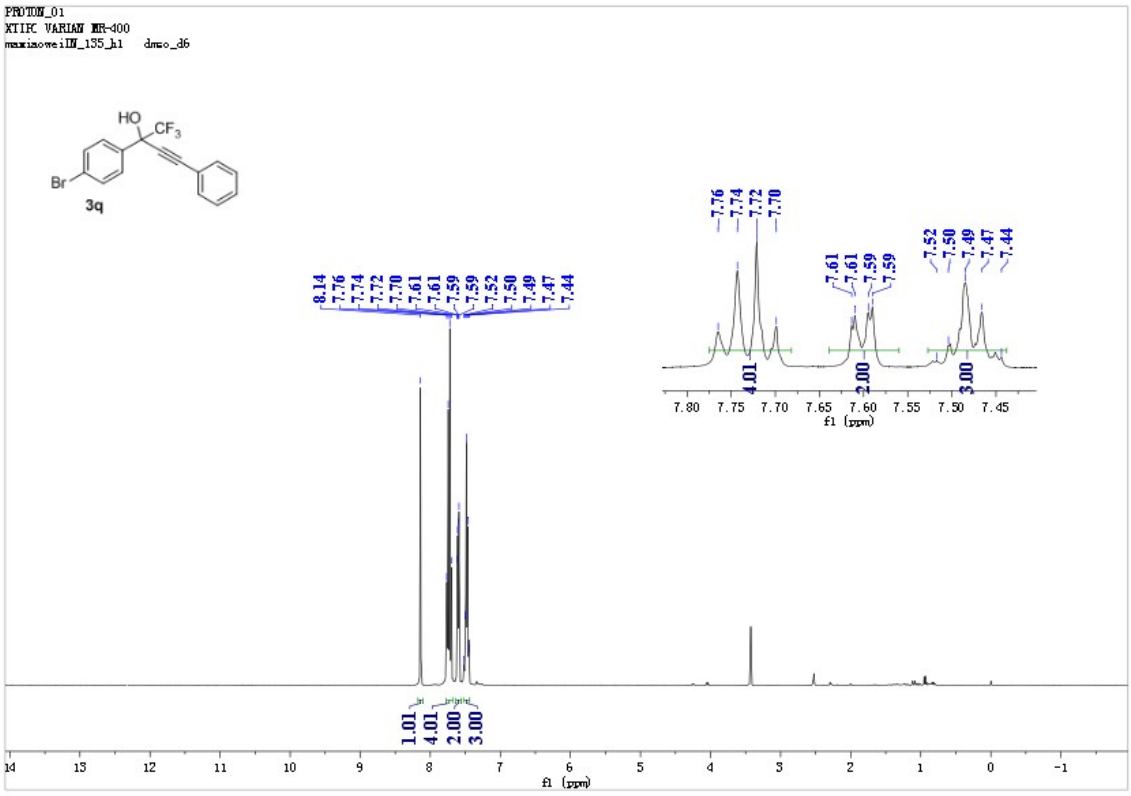

CARBON_01
XTIPC VARIAN NMR-400
maxiaowei_LW_121_C13 DMSO
HO
CF3
S
3o
137.13
132.05
130.01
129.67
128.60
127.70
127.54
125.63
122.79
119.91
85.53
82.58
73.06
72.74
72.43
72.12
f1 (ppm)

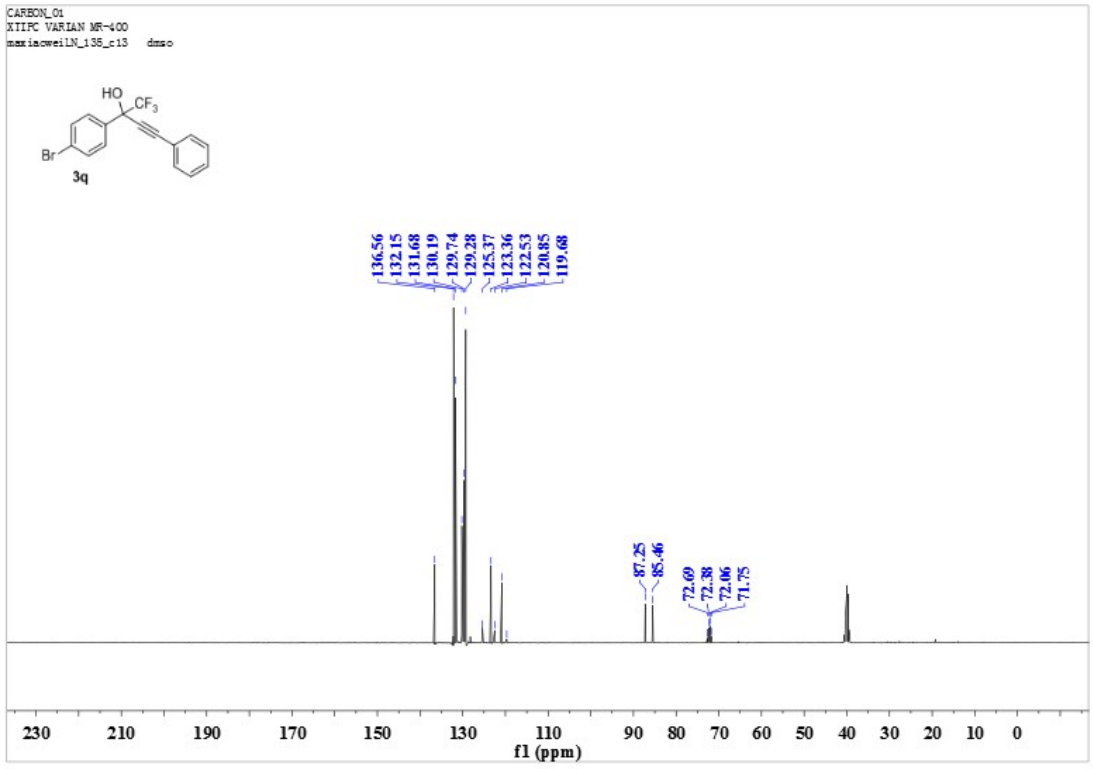

F19-4N-DMSO-20140619-maxiaowei-LN-121-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-121-shiheridaxue
in DMSO
HO
CF3
S
3o
-0.77
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

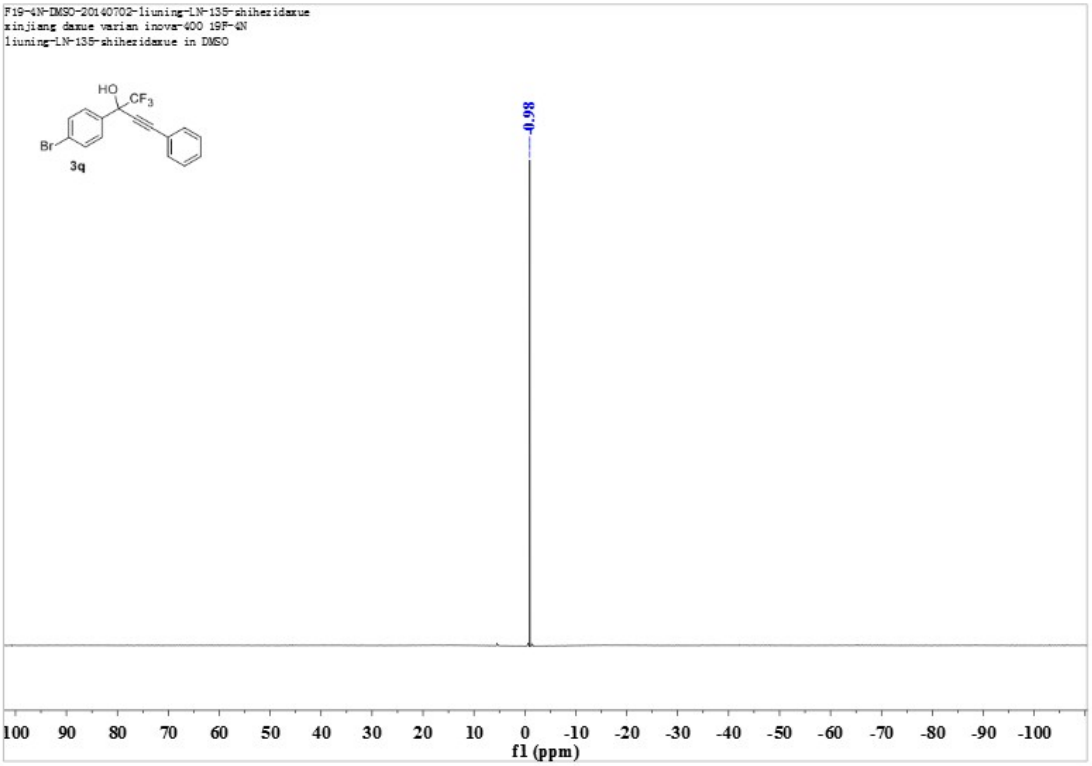

PROTON_01
XTIPC VARIAN NMR-400
maxiaowei_LN_123_H1 DMSO
HO
CF3
Cl
3p
7.71
7.67
7.65
7.51
7.49
1.00
2.00
2.01
1.53
1.51
1.50
1.48
1.47
0.93
0.90
0.88
0.87
0.76
0.74
0.72
0.71
0.69
1.01
2.08
2.03
f1 (ppm)

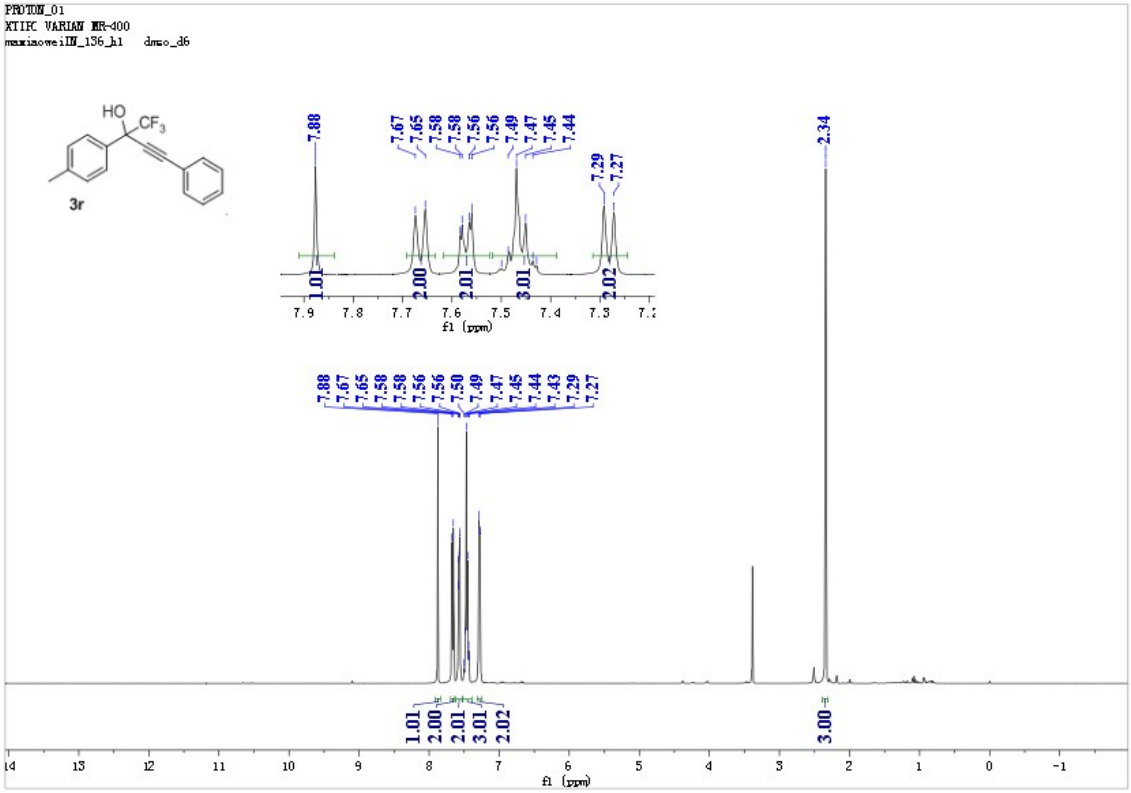

CARBON_01
XIIPC VARIAN NMR-400
maxiaowei_LN_123_C13 DMSO
HO
CF3
Cl
3p
137.26
135.05
130.05
129.18
126.10
123.25
120.41
92.58
72.71
72.40
72.33
72.09
71.78
9.25
9.20
0.00
f1 (ppm)

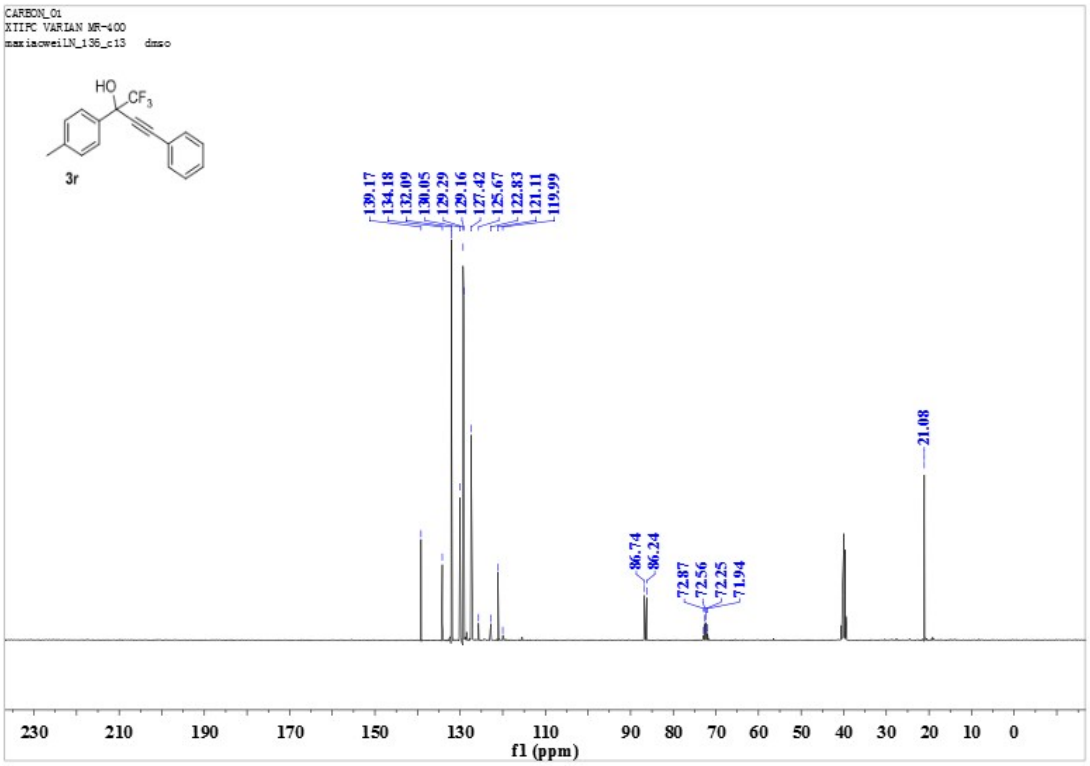

F19-4N-DMSO-20140619-maxiaowei-LN-123-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
maxiaowei-LN-123-shiheridaxue
in DMSO
HO
CF3
Cl
3p
-1.27
f1 (ppm)

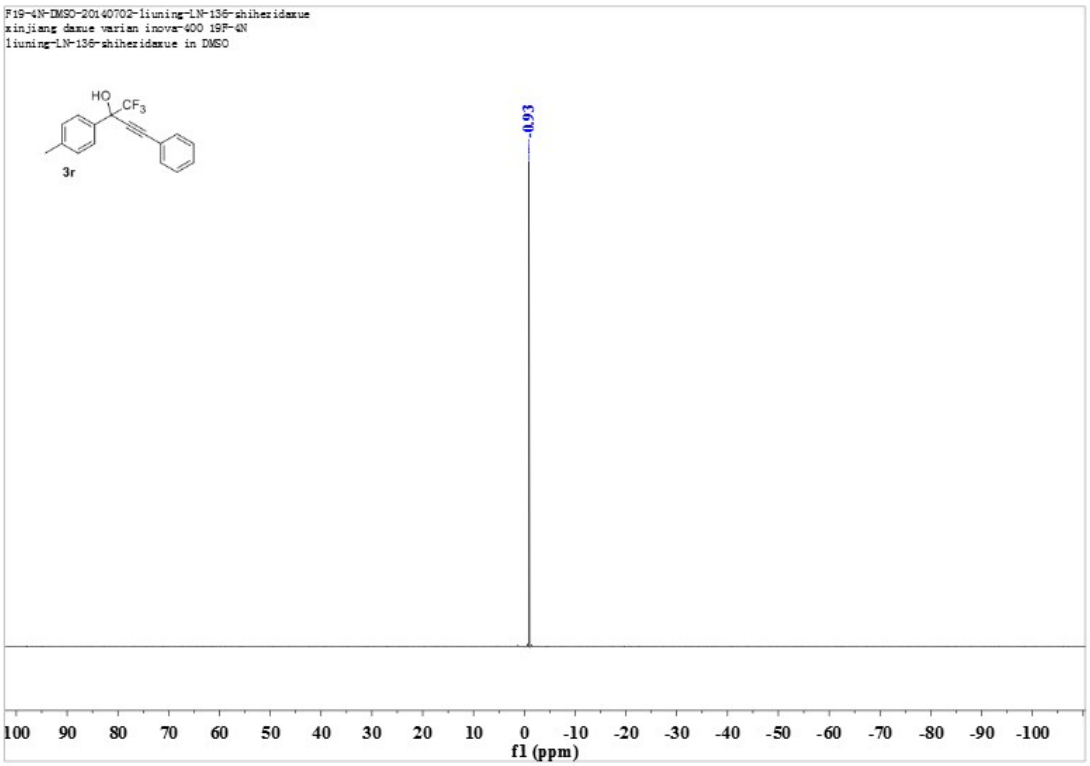

PROTON_01
XTIPC VARIAN MR-400
maxiaoweiLN_135_h1 dmso_d6
HO
CF3
Br
3q
8.14
7.76
7.74
7.72
7.70
7.61
7.61
7.59
7.59
7.52
7.50
7.49
7.47
7.44
1.01
4.01
2.00
3.00
f1 (ppm)

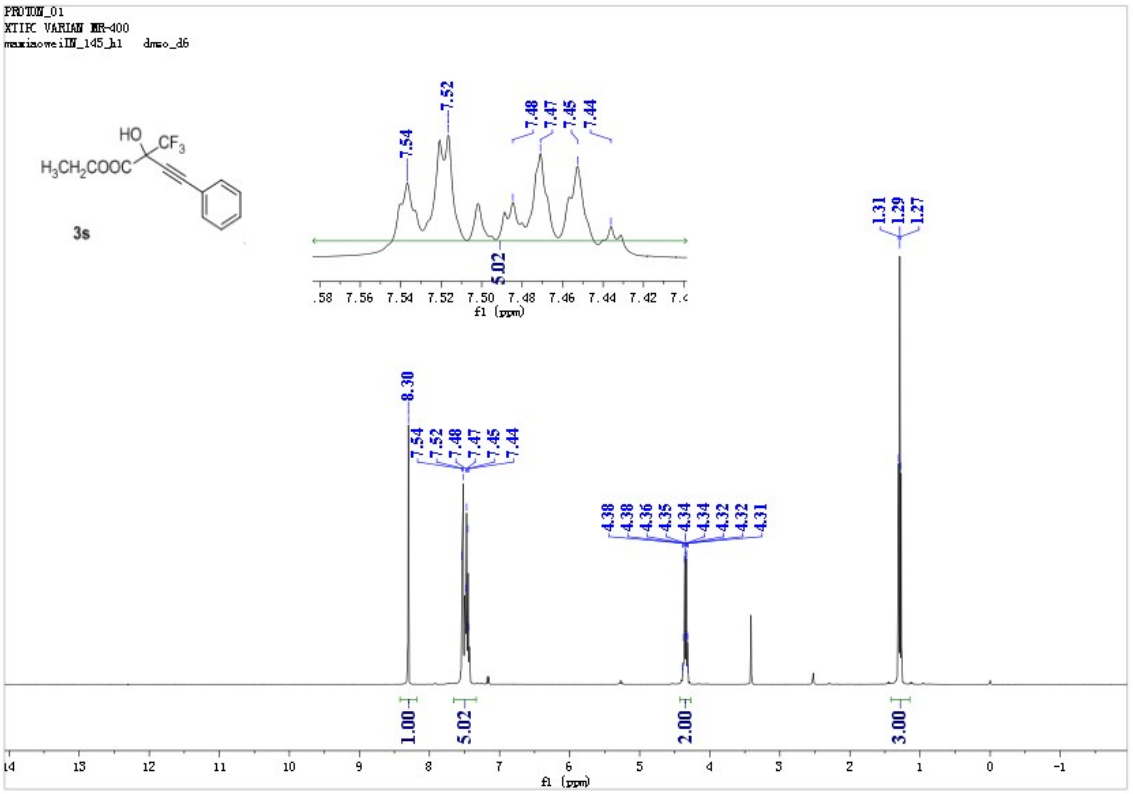

CARBON_01
XTIPC VARIAN MR-400
maxiaoweiLN_135_c13 dmso
HO
CF3
Br
3q
136.56
132.15
131.68
130.19
129.74
129.28
125.37
123.36
122.53
120.85
119.68
87.25
85.46
72.69
72.38
72.06
71.75
f1 (ppm)

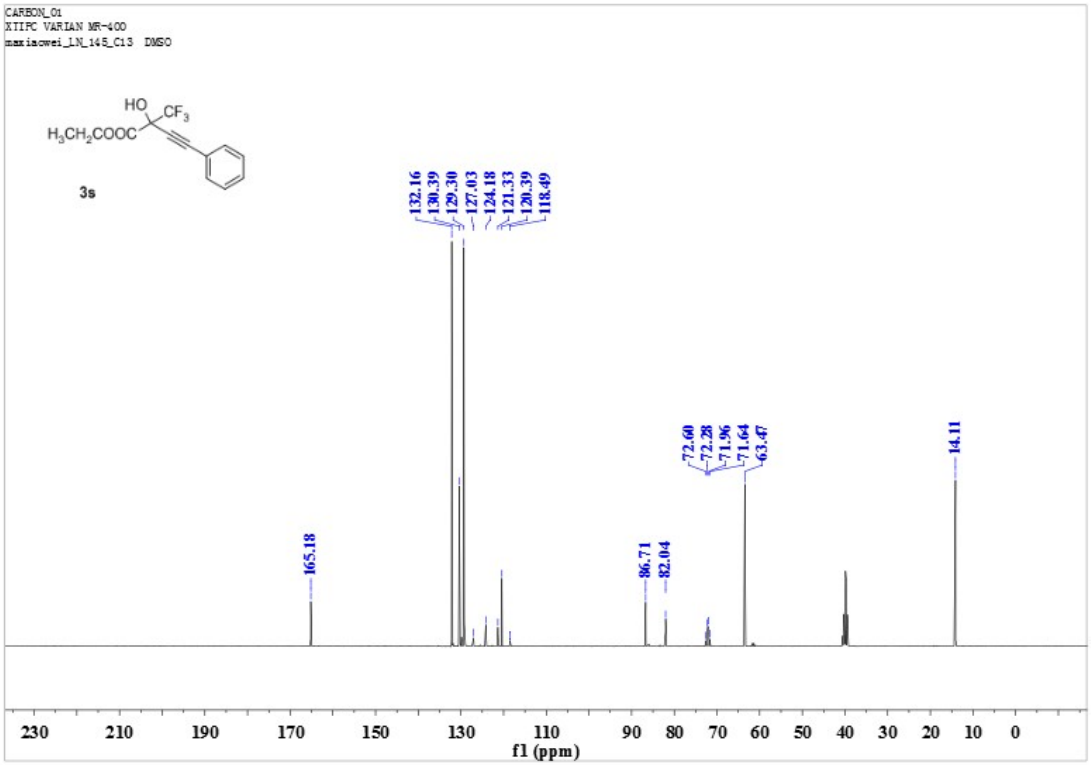

F19-4N-DMSO-20140702-liuning-LN-135-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-135-shiheridaxue in DMSO
HO
CF3
Br
3q
-0.98
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)

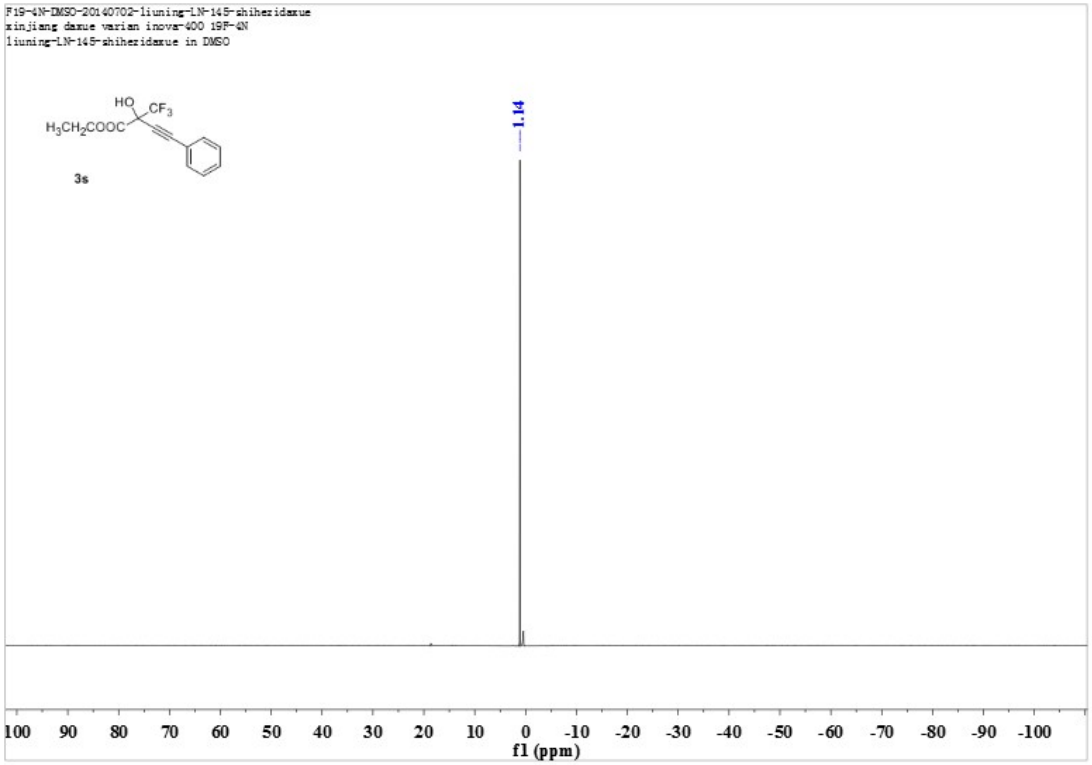

PROTON_01
XTIPC VARIAN MR-400
maximoweiLN_136_h1 dmso_d6
HO
CF3
3r
7.88
7.67
7.65
7.58
7.58
7.56
7.56
7.49
7.47
7.45
7.44
7.29
7.27
2.34
1.01
2.00
2.01
3.01
2.02
3.00
f1 (ppm)


CARBON_01
XTIPC VARIAN MR-400
maxiaoweiLN_136_c13 dmso
HO
CF3
3r
139.17
134.18
132.09
130.05
129.29
129.16
127.42
125.67
122.83
121.11
119.99
86.74
86.24
72.87
72.56
72.25
71.94
21.08
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


F19-4N-DMSO-20140702-liuning-LN-136-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-136-shiheridaxue in DMSO
HO
CF3
3r
-0.93
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)


PROTON_01
XTIPC VARIAN MR-400
maxiaoweiLN_145_h1 dmso_d6
HO
CF3
H3CH2COOC
3s
7.54
7.52
7.48
7.47
7.45
7.44
5.02
f1 (ppm)
8.30
4.38
4.36
4.35
4.34
4.32
4.31
1.31
1.29
1.27
1.00
5.02
2.00
3.00
f1 (ppm)


CARBON_01
XTIPC VARIAN MR-400
maxiaowei_LN_145_C13 DMSO
HO
CF3
H3CH2COOC
3s
132.16
130.39
129.30
127.03
124.18
121.33
120.39
118.49
165.18
86.71
82.04
72.60
72.28
71.96
71.64
63.47
14.11
f1 (ppm)


F19-4N-DMSO-20140702-liuning-LN-145-shiheridaxue
xinjiang daxue varian inova-400 19F-4N
liuning-LN-145-shiheridaxue in DMSO
HO
CF3
H3CH2COOC
3s
-1.14
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100
f1 (ppm)