Rh(Ⅲ)-Catalyzed *ortho*-C–H Alkynylation of *N*-Phenoxyacetamides with Hypervalent Iodine-alkyne Reagents at Room Temperature

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Supporting Information

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I. General

All reactions were carried out under an atmosphere of nitrogen. Commercially available chemicals were obtained from Sigma-Aldrich, Alfa Aesar, TCI and Aladdin and used as received unless otherwise stated. Dichloro(η^5-pentamethylcyclopentadienyl)rhodium(III) dimer (99%) was purchased from Sinocompound Technology Co.,Ltd.

Reactions were monitored with analytical thin-layer chromatography (TLC) on silica. ^{1}H NMR and ^{13}C NMR data were recorded on Bruker nuclear resonance (500 MHz, 400 MHz and 300MHz) spectrometers unless otherwise specified, respectively. Chemical shifts (δ) are given in ppm relative to TMS. The residual solvent signals were used as references and the chemical shifts converted to the TMS scale ($CDCl_3$: δ_H=7.26 ppm, δ_C=77.16 ppm; DMSO-d_6: δ_H=2.50 ppm, δ_C=39.52 ppm; MeOD-d_4: δ_H=3.31 ppm, δ_C=49.00 ppm; Acetone-d_6: δ_H=2.05 ppm, δ_C=29.84 ppm, 206.26 ppm). HRMS (ESI) analysis was performed by The Analytical Instrumentation Center at Peking University, Shenzhen Graduate School and (HRMS) data were reported with ion mass/charge (m/z) ratios as values in atomic mass units.

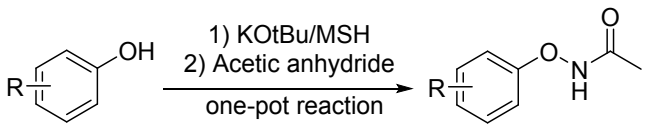
II. Preparation of starting materials

General procedure A:

Following literature reports[1,2], Phenols (1 equiv.) was dissolved in 4 mL of methanol, and then potassium *tert*-butoxide (1 equiv.) was added. The mixture was allowed to stir for 0.5 h under N_2 atmosphere. The methanol was removed, and the residue was taken up in 2 mL of dichloromethane. Then the freshly prepared *O*-mesitylsulfonylhydroxyl-amine (378 mg, 1.76 mmol) in dichloromethane (0.2 M) was added under ice cooling. The mixture was allowed to stir for 1 h, dichloromethane was then removed under reduce pressure to afford the corresponding *N*-aryloxyamine.

In a 20 mL round-bottom flask, *N*-aryloxyamine (1.0 equiv.) was dissolved in ether (0.2 M).The flask was cooled in an ice bath, to which acetic anhydride (2.0 equiv.) was slowly added. The ice bath was allowed to warm to room temperature and the mixture was stirred for 3 h at room temperature. The reaction mixture was concentrated under reduced pressure and purified by flash silica gel column chromatography to give the corresponding *N*-phenoxyacetamide.

In our work, most of substrate **1** were synthesized by general procedure A.



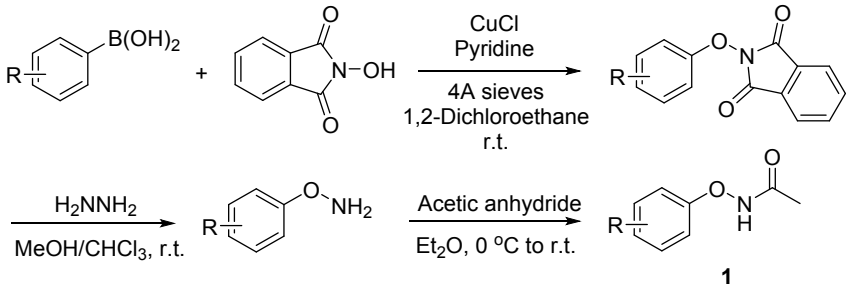
General procedure B:

Following literature reports[3,4], in a 50mL round-bottom flask, *N*-hydroxy -phthalimide (1.0 equiv.), cooper (I) chloride (1.0 equiv.), freshly activated 4 Å molecular sieves (250 mg/mmol), and phenylboronic acid (2.0 equiv.) were combined in 1,2-dichloroethane (0.2 M). The pyridine (1.1 equiv.) was then added to the suspension. The reaction mixture was open to the atmosphere and

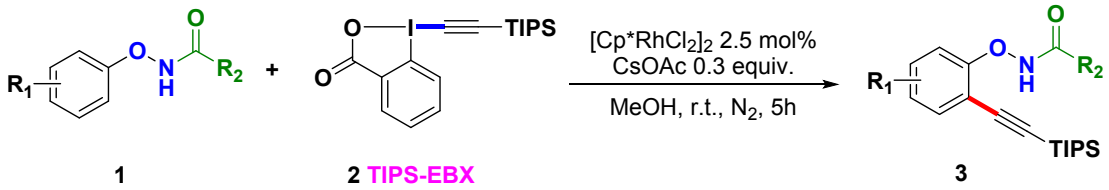
stirred at room temperature over 24-48 h. Upon completion, silica gel was added to the flask and the solvent was removed under vacuum. The desired *N*-aryloxyphthalimides were obtained by flash column chromatography on silica gel.

Hydrazine monohydrate (3.0 equiv.) was added to the solution of *N*-aryloxyphth -alimide (1.0 equiv.) in 10% MeOH in $CHCl_3$ (0.1 M). The reaction was allowed to stir at room temperature over 12 h. Upon completion, the reaction mixture was filtered off and washed with CH_2Cl_2. The filtrate was concentrated under reduced pressure, and purified by flash silica gel column chromatography to give the corresponding *N*-aryloxyamine.

In a 20 mL round-bottom flask, *N*-aryloxyamine (1.0 equiv.) was dissolved in ether (0.2 M).The flask was cooled in an ice bath, to which acetic anhydride (2.0 equiv.) was slowly added. The ice bath was allowed to warm to room temperature and the mixture was stirred for 3 h at room temperature. The reaction mixture was concentrated under reduced pressure and purified by flash silica gel column chromatography to give the corresponding *N*-phenoxyacetamide.



III. General procedure for the *ortho* C–H Alkynylation



N-Acetyl aryloxyamine (**1**) (0.4 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol%), TIPS—EBX (0.1mmol), and CsOAc (0.03 mmol) were weighed into a 25mL pressure tube, to which was added MeOH (1 mL) in a glove box. The reaction vessel was stirred at room temperature for 5 h. Then the mixture was concentrated under vacuum and the residue was purified by column chromatography on silica gel with a gradient eluent of petroleum ether and ethyl acetate to afford the corresponding product **3**.

Characterization of products 3:

***N*-(2-Methyl-6-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3a)** A white solid ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.32 (d, *J* = 7.5 Hz, 1H), 7.19 (dd, *J* = 7.5, 0.8 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 2.53 (s, 3H), 1.95 (d, *J* = 6.9 Hz, 3H), 1.18 (d, *J* = 2.6 Hz, 21H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 166.44, 159.05, 131.97, 131.85, 125.10, 115.24, 103.82, 96.95, 29.78, 18.64, 16.07, 11.22. HRMS (ESI) calcd for $C_{20}H_{33}NO_2Si$ $[M+H]^+$ 346.2202 found 346.2204.

***N*-(5-Methyl-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3b)** A white solid ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.21 (s, 1H), 6.97 (s, 1H), 2.24 (s, 3H), 2.18 (s, 3H), 2.14 (s, 3H), 1.14 (s, 21H). ^{13}C

NMR (126 MHz, $CDCl_3$) δ: 157.97, 139.66, 138.67, 131.83, 130.96, 129.67, 125.95, 121.88, 93.80, 27.73, 19.81, 16.70, 9.38. HRMS (ESI) calcd for $C_{20}H_{33}NO_2Si$ $[M+H]^+$ 346.2202 found 346.2204.

***N*-(4-Methyl-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3c)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ： 7.27 (s, 1H), 7.10 (s, 2H), 2.28 (s, 3H), 2.14 (s, 2H), 1.14 (s, 17H). ^{13}C NMR (101 MHz, $CDCl_3$) δ: 157.98, 141.67, 134.27, 133.07, 131.71, 130.39, 127.94, 111.82, 94.52, 29.68, 20.29, 18.64, 11.26. HRMS (ESI) calcd for $C_{20}H_{33}NO_2Si$ $[M+H]^+$ 346.2202 found 346.2202.

***N*-(4-(*tert*-Butyl)-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3d)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ: 7.44 (s, 1H), 7.32 (d, *J* = 7.1 Hz, 1H), 7.13 (d, *J* = 8.7 Hz, 1H), 2.15 (s, 3H), 1.30 (s, 9H), 1.15 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ: 157.97, 141.71, 133.13, 131.76, 130.74, 127.94, 126.97, 111.55, 94.55, 34.20, 31.27, 29.68, 18.66, 11.29. HRMS (ESI) calcd for $C_{23}H_{38}NO_2Si$ $[M+H]^+$ 388.2762 found 388.2760.

***N*-(2-((Triisopropylsilyl)ethynyl)phenoxy)acetamide (3e)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ: 7.47 (d, *J* = 7.4 Hz, 1H), 7.30 (d, *J* = 7.7 Hz, 1H), 7.21 (d, *J* = 8.2 Hz, 1H), 7.03 (t, *J* = 7.1 Hz, 1H), 2.12 (s, 3H), 1.14 (d, *J* = 1.6 Hz, 21H) ^{13}C NMR (75 MHz, $CDCl_3$) δ: 165.87, 133.95, 133.95, 129.74, 122.94, 122.94, 111.58, 96.74, 29.65, 11.22. HRMS (ESI) calcd for $C_{19}H_{30}NO_2Si$ $[M+H]^+$ 332.2046 found 332.2047.

***N*-(4,5-Dimethyl-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3f)** A white solid ^1H NMR (500 MHz, $CDCl_3$) δ: 7.21 (s, 1H), 6.97 (s, 1H), 2.24 (s, 3H), 2.18 (s, 3H), 2.14 (s, 3H), 1.14 (s, 21H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.40, 141.83, 139.27, 134.86, 133.07, 131.80, 128.13, 113.66, 95.71, 29.95, 20.43, 19.79, 18.92, 11.60. HRMS (ESI) calcd for $C_{21}H_{34}NO_2Si$ $[M+H]^+$ 360.2359 found 360.2358.

***N*-(4-Chloro-2-methyl-6-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3g)** A white solid ^1H NMR (500 MHz, $CDCl_3$) δ: 7.26 (d, *J* = 2.4 Hz, 1H), 7.14 (d, *J* = 2.0 Hz, 1H), 2.48 (s, 3H), 1.94 (s, 3H), 1.16 (s, 21H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 157.74, 131.57, 131.07, 130.68, 130.01, 116.73, 102.34, 98.53, 29.58, 18.58, 16.04, 11.20. HRMS (ESI) calcd for $C_{20}H_{31}ClNO_2Si$ $[M+H]^+$ 380.1813 found 380.1815.

***N*-(5-Fluoro-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3h)** A white solid ^1H NMR (400 MHz, Chloroform-*d*) δ 8.98 (s, 1H), 7.22 (q, *J* = 7.9 Hz, 1H), 7.00 (s, 1H), 6.80 (t, *J* = 8.4 Hz, 1H), 2.10 (s, 3H), 1.14 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.74, 165.28, 162.77, 161.26, 129.89, 129.79, 124.63, 124.11, 110.46, 110.25, 107.66, 103.02, 94.06, 77.16, 19.74, 18.73, 11.37. HRMS (ESI) calcd for $C_{19}H_{29}FNO_2Si$ $[M+H]^+$ 350.1952 found 350.1950.

***N*-(4-Fluoro-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3i)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ: 7.24 – 7.06 (m, 1H), 6.99 (s, 1H), 2.13 (s, 3H), 1.13 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ: 156.29, 141.69, 136.31, 133.09, 131.69, 127.96, 120.15(*J*=22.2Hz), 116.46(*J*=23.2Hz), 94.52, 29.67, 18.60, 11.19. HRMS (ESI) calcd for $C_{19}H_{29}FNO_2Si$ $[M+H]^+$ 350.1952 found 350.1953.

***N*-(5-Chloro-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3j)** A white solid ^1H NMR (500 MHz,

$CDCl_3$) δ: 7.42 (s, 1H), 7.24 (d, *J* = 5.8 Hz, 1H), 7.15 (s, 1H), 2.10 (s, 3H), 1.13 (s, 21H). ^{13}C NMR (126 MHz, $CDCl_3$) δ: 158.84, 133.41, 132.60, 129.75, 129.61, 128.12, 115.40, 98.68, 29.71, 18.64, 11.30. HRMS (ESI) calcd for $C_{19}H_{29}ClNO_2Si$ $[M+H]^+$ 366.1656 found 366.1659.

***N*-(4-Chloro-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3k)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ: 7.38 (d, *J* = 8.1 Hz, 1H), 7.24 (s, 1H), 7.01 (d, *J* = 6.9 Hz, 1H), 2.13 (s, 3H), 1.13 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.46, 135.34, 134.66, 133.85, 123.34, 121.87, 114.46, 109.66, 98.03, 29.68, 18.61, 11.21. HRMS (ESI) calcd for $C_{19}H_{29}ClNO_2Si$ $[M+H]^+$ 366.1656 found 366.1659.

***N*-(5-Bromo-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3l)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ：7.38 (s, 1H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.16 (d, *J* = 7.3 Hz, 1H), 2.12 (s, 3H), 1.12 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ：160.33, 134.83, 126.23, 123.18, 117.20, 115.67, 110.00, 98.29, 29.68, 18.61, 11.20. HRMS (ESI) calcd for $C_{19}H_{29}BrNO_2Si$ $[M+H]^+$ 410.1151found 410.1154.

***N*-(4-Bromo-2-((triisopropylsilyl)ethynyl)phenoxy)acetamide (3m)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ：7.56 (s, 1H), 7.39 (d, *J* = 11.2 Hz, 1H), 7.09 (s, 1H), 2.12 (s, 3H), 1.13 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ：158.92, 141.68, 136.18, 133.10, 132.53, 131.69, 127.95, 115.05, 94.57, 29.79, 18.61, 11.19. HRMS (ESI) calcd for $C_{19}H_{29}BrNO_2Si$ $[M+H]^+$ 410.1151found 410.1152.

Methyl 4-(acetamidooxy)-3-((triisopropylsilyl)ethynyl)benzoate 3n A white solid ^1H NMR (500 MHz, $CDCl_3$) δ: 8.11 (s, 1H), 7.95 (d, *J* = 5.1 Hz, 10H), 7.26 (s, 1H), 3.90 (s,30H), 2.11 (s, 3H), 1.13 (s, 21H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.02, 163.26, 135.67, 131.53, 125.03, 112.17, 111.14, 100.24, 98.01, 52.29, 18.77, 11.42 HRMS (ESI) calcd for $C_{21}H_{32}NO_4Si$ $[M+H]^+$ 390.2101 found 390.2103.

***N*-(2-((Triisopropylsilyl)ethynyl)phenoxy)benzamide (3o)** A white solid ^1H NMR (400 MHz, $CDCl_3$) δ: 10.95 (s, 1H), 8.18 (dd, *J* = 5.5, 3.5 Hz, 1H), 7.64 – 7.56 (m, 1H), 7.55 – 7.42 (m, 2H), 7.34 – 7.28 (m, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 7.05 (t, *J* = 7.3 Hz, 1H), 1.11 (s, 21H). ^{13}C NMR (101 MHz, $CDCl_3$) δ: 165.25, 159.50, 134.15, 132.29, 131.55, 130.64, 129.37, 122.98, 119.68, 113.48, 113.40, 105.62, 100.00, 18.54, 11.13. HRMS (ESI) calcd for $C_{24}H_{32}NO_2Si$ $[M+H]^+$ 394.2202 found 394.2203.

***N*-(4,5-dimethyl-2-(phenylethynyl)phenoxy)acetamide (3q)** A white solid ^1H NMR (300 MHz, Chloroform-*d*) δ 7.53 (dd, *J* = 6.7, 3.1 Hz, 2H), 7.40 – 7.30 (m, 3H), 7.28 (s, 1H), 7.00 (s, 1H), 2.27 (s, 3H), 2.22 (s, 3H), 2.17 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.53, 139.07, 134.18, 131.64, 131.55, 128.35, 128.28, 123.28, 93.70, 77.47, 77.04, 76.62, 20.28, 19.81, 18.76. HRMS (ESI) calcd for $C_{18}H_{17}NNaO_2$ $[M+Na]^+$: 302.1157 found 302.1153.

Characterization of other compounds:

2-methoxy-3,4-dimethyl-6-((triisopropylsilyl)ethynyl)phenol (4a) A white solid ^1H NMR (400 MHz, Chloroform-*d*) δ 6.93 (s, 1H), 5.72 (s, 1H), 3.80 (s, 3H), 2.18 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.48, 145.10, 132.07, 128.92, 127.48, 107.66, 101.45, 96.74, 77.33, 77.01, 76.69, 60.36, 19.27, 18.69, 12.52, 11.26. HRMS (ESI) calcd for $C_{20}H_{33}O_2Si$ Exact Mass: $[M+H]^+$ 333.2250 found 333.2234.

***N*-(2-ethynyl-4,5-dimethylphenoxy)acetamide (4b)** A white solid ^1H NMR (400 MHz, Methanol-d_4) δ 7.17 (s, 1H), 6.87 (s, 1H), 3.57 (s, 1H), 2.26 – 2.22 (m, 3H), 2.17 (d, *J* = 2.4 Hz, 3H), 2.03 (d, *J* = 2.3

Hz, 3H). ^{13}C NMR (101 MHz, MeOD) δ 170.51, 159.66, 140.38, 135.53, 132.10, 114.70, 108.02, 82.76, 79.68, 20.21, 19.40, 18.67. HRMS (ESI) calcd for $C_{12}H_{14}NO_2$ $[M+H]^+$ 204.1025 found 204.1030.

1-(phenylethynyl)-1l3-benzo[d][1,2]iodaoxol-3(1H)-one (2d) A white solid ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.32 (d, *J* = 8.3 Hz, 1H), 8.13 (dd, *J* = 7.4, 1.7 Hz, 1H), 7.94 – 7.88 (m, 1H), 7.83 – 7.77 (m, 1H), 7.74 – 7.68 (m, 2H), 7.59 – 7.48 (m, 3H). ^{13}C NMR (101 MHz, DMSO) δ 166.25, 135.13, 132.56, 132.05, 131.32, 131.30, 130.66, 129.04, 127.49, 120.50, 116.35, 104.31, 52.10. HRMS (ESI) calcd for $C_{15}H_{10}IO_2$ $[M+H]^+$: 348.9725 found 348.9729.

Intermediate A A red solid ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (s, 1H), 7.07 (s, 1H), 2.51 (s, 3H), 2.31 (s, 3H), 2.24 (s, 3H), 1.86 (s, 15H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.94, 165.72, 137.07, 135.46, 126.07, 110.33, 97.49, 97.42, 21.92, 20.36, 19.51, 10.49. HRMS (ESI) calcd for $C_{20}H_{27}NO_2Rh$ $[M+H]^+$: 416.1097 found 416.1027.

IV. References

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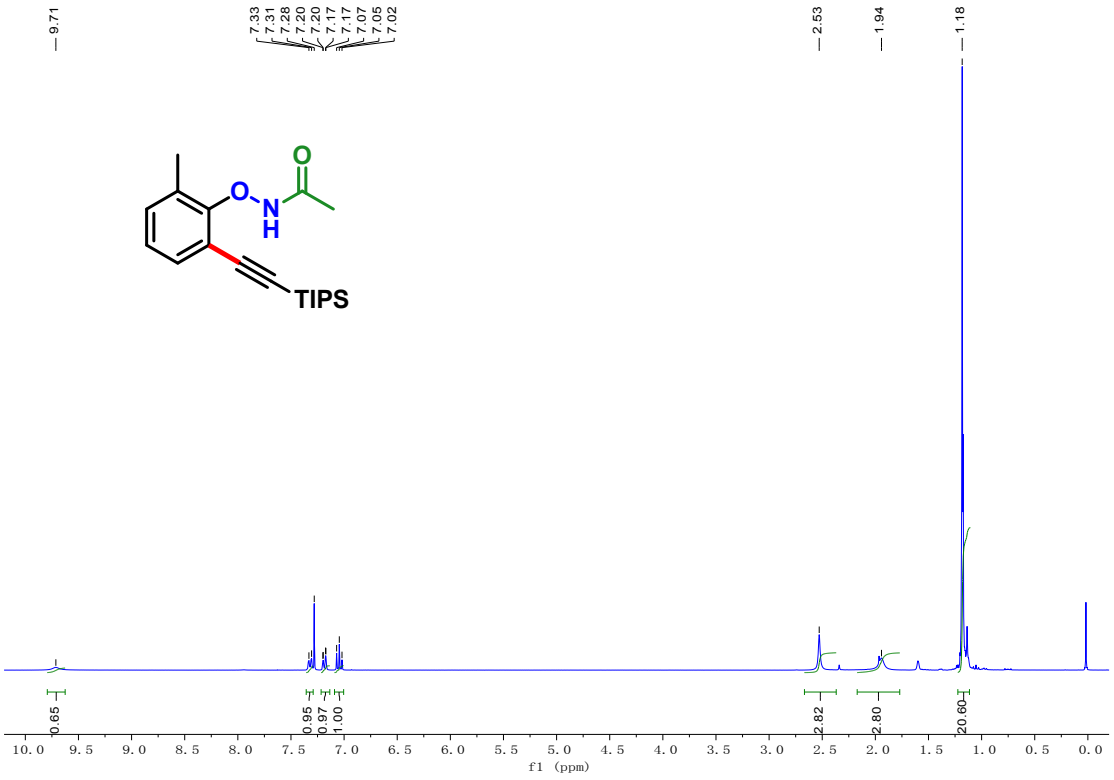
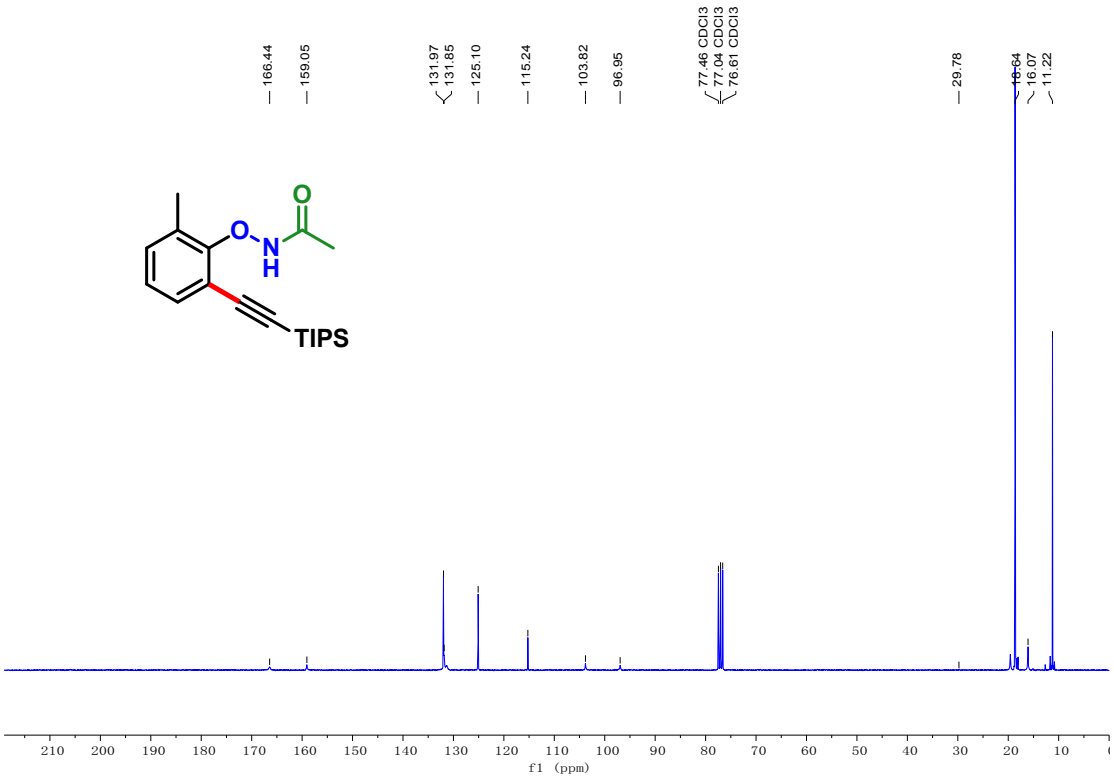
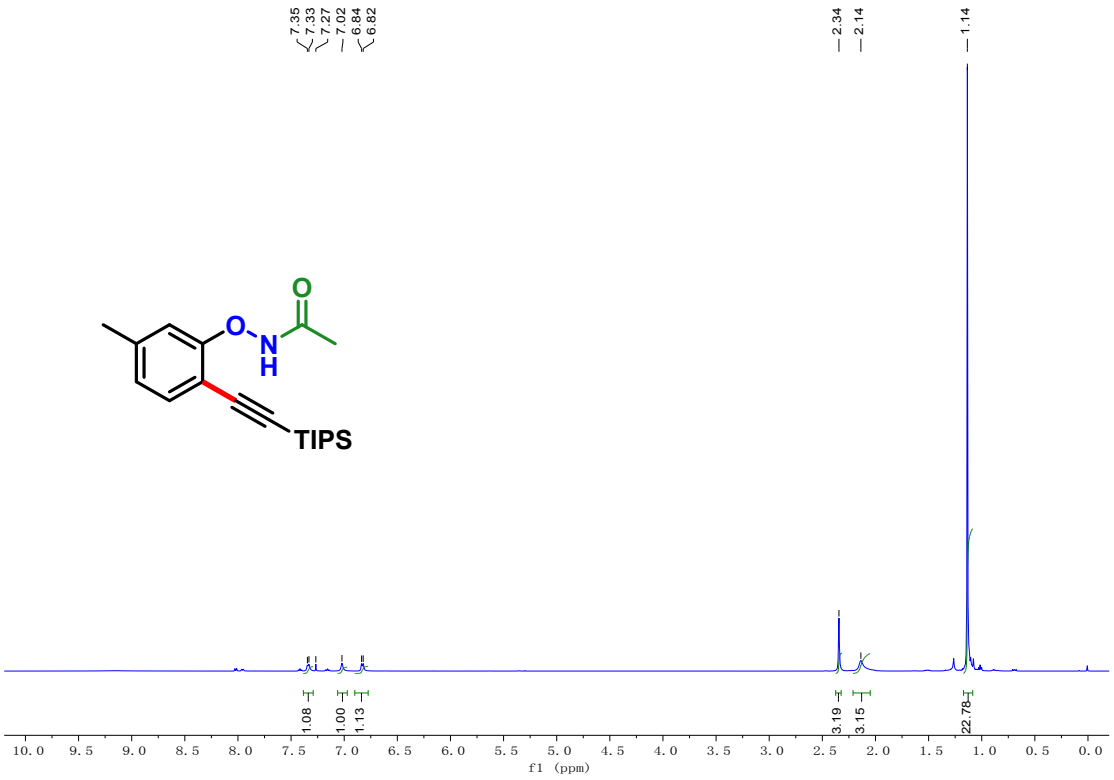
V. ^{1}H and ^{13}C NMR spectra



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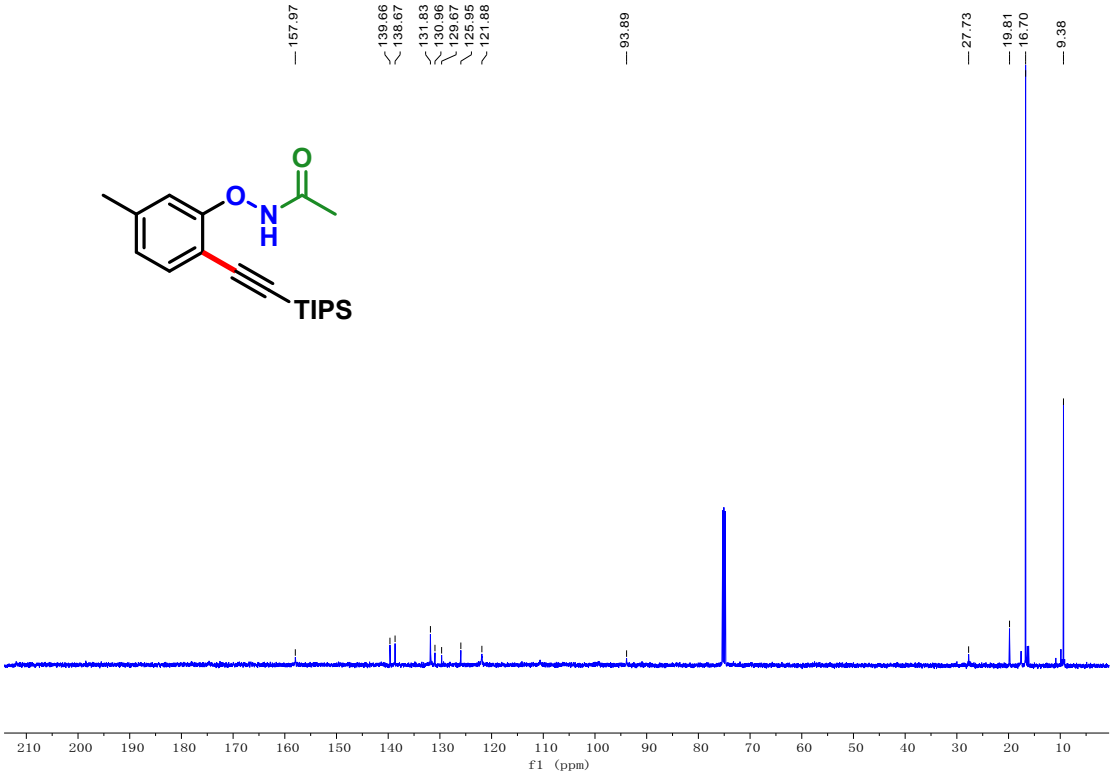
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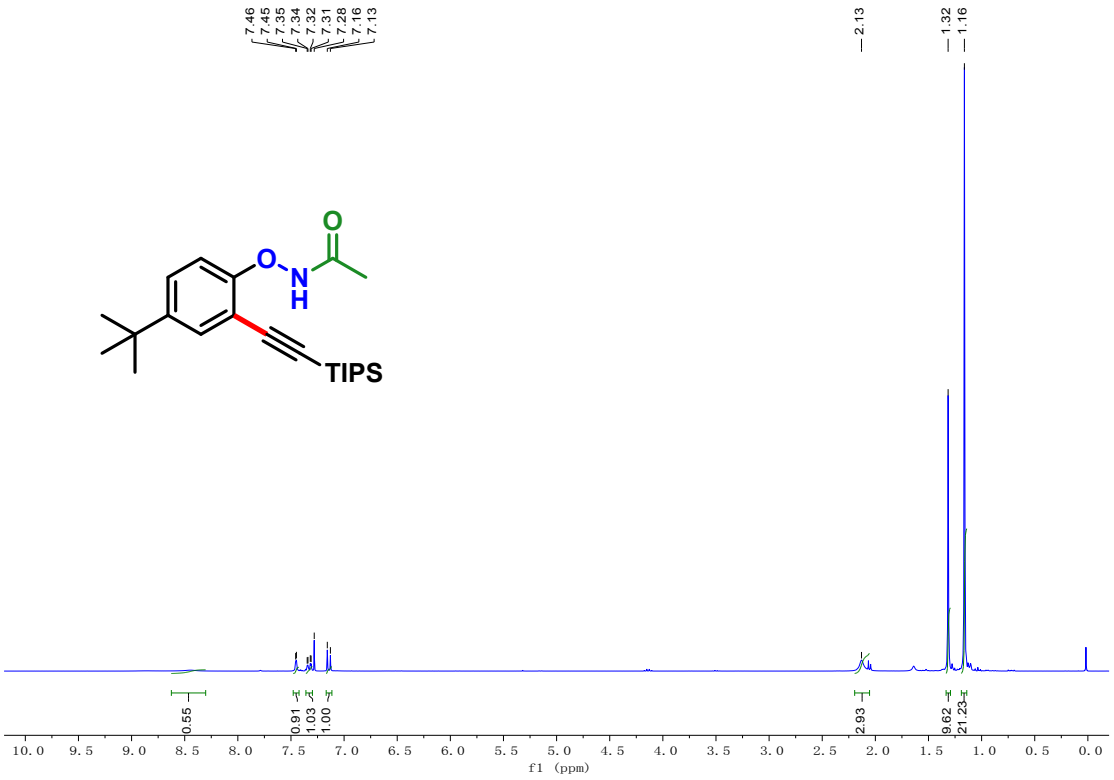
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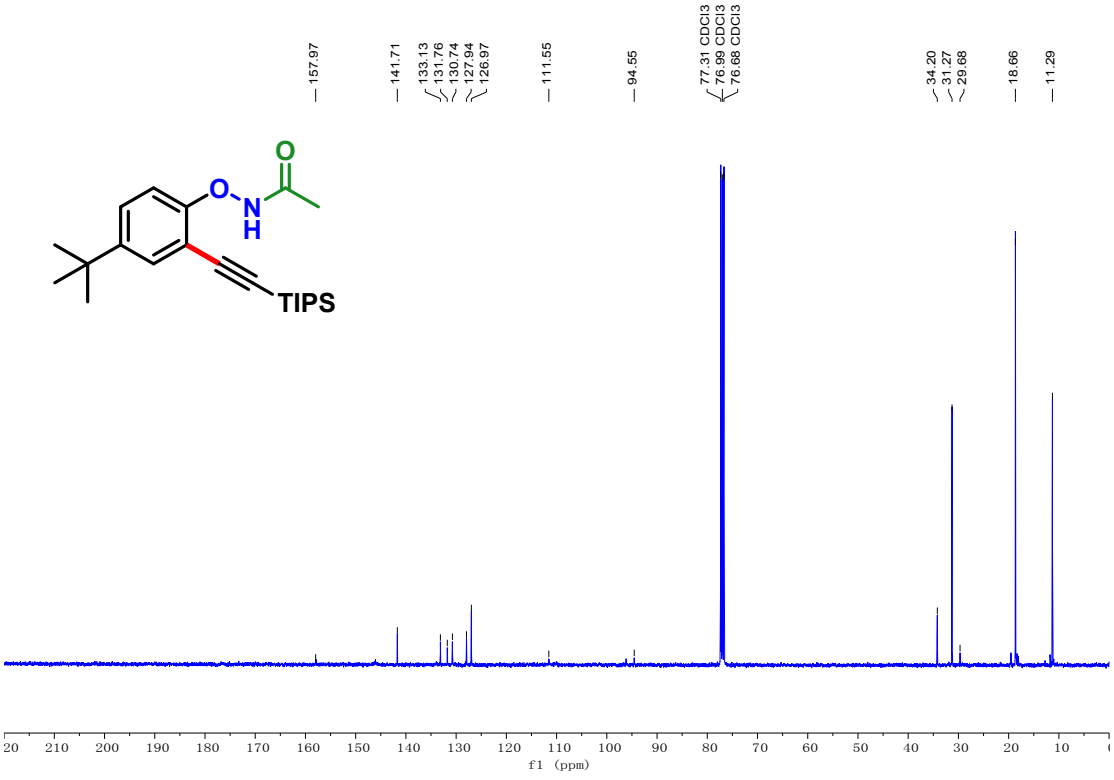
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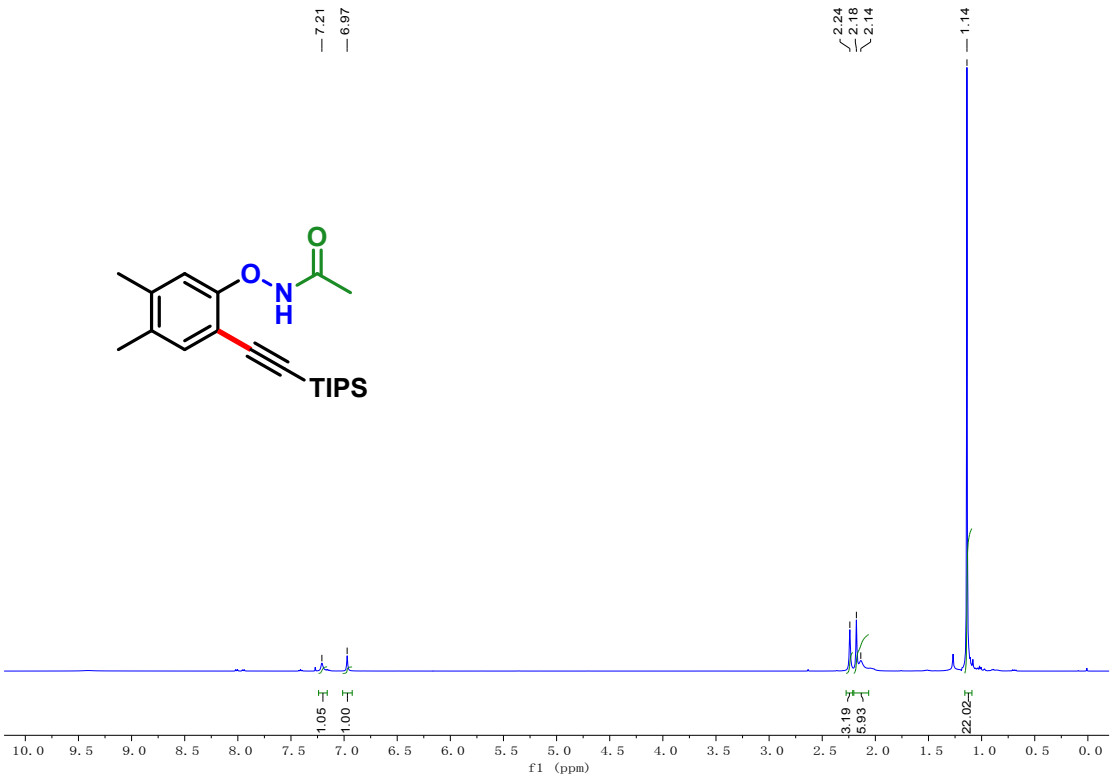
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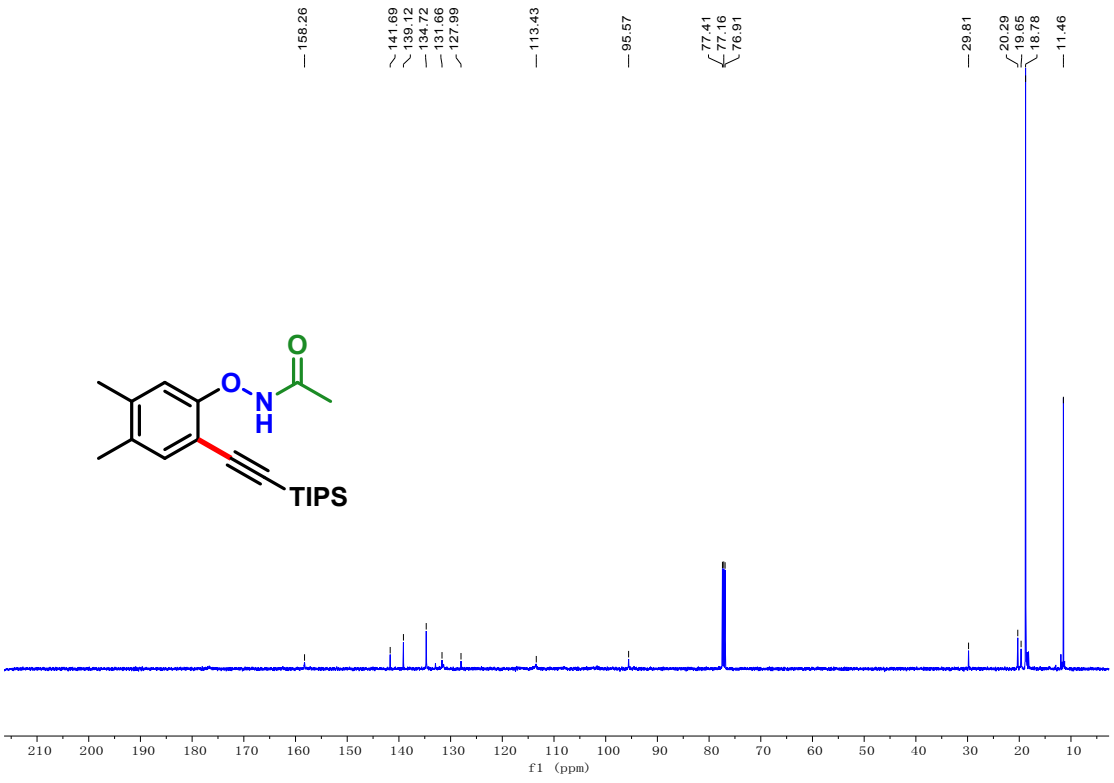
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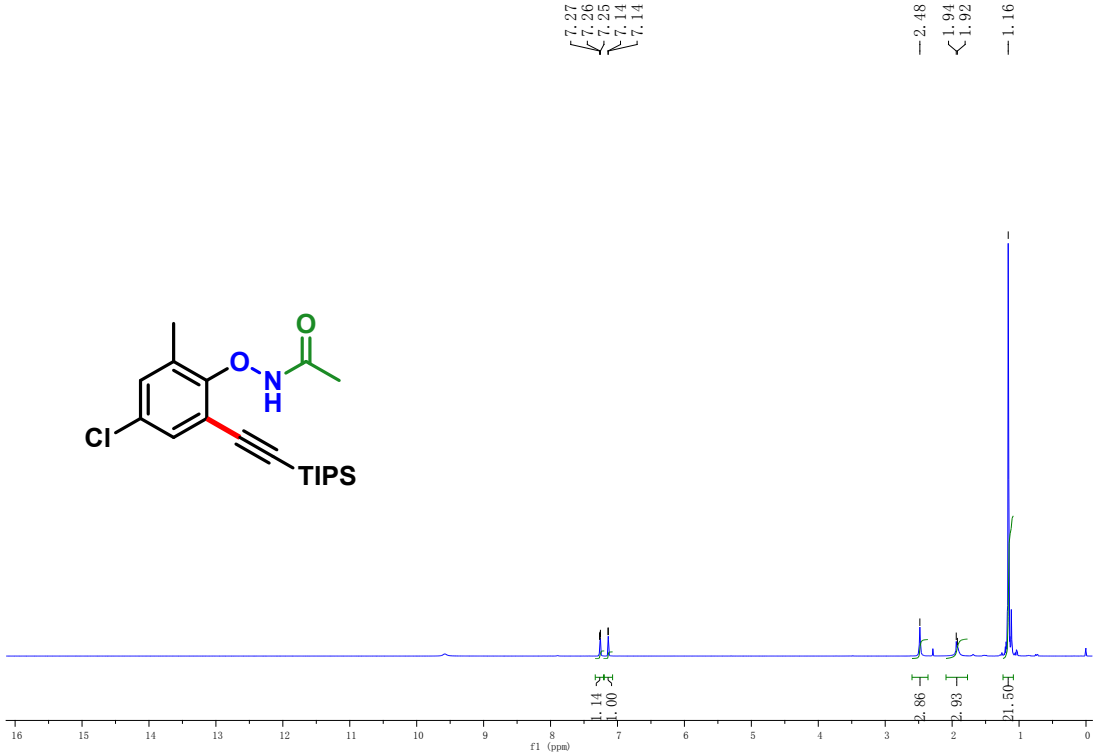
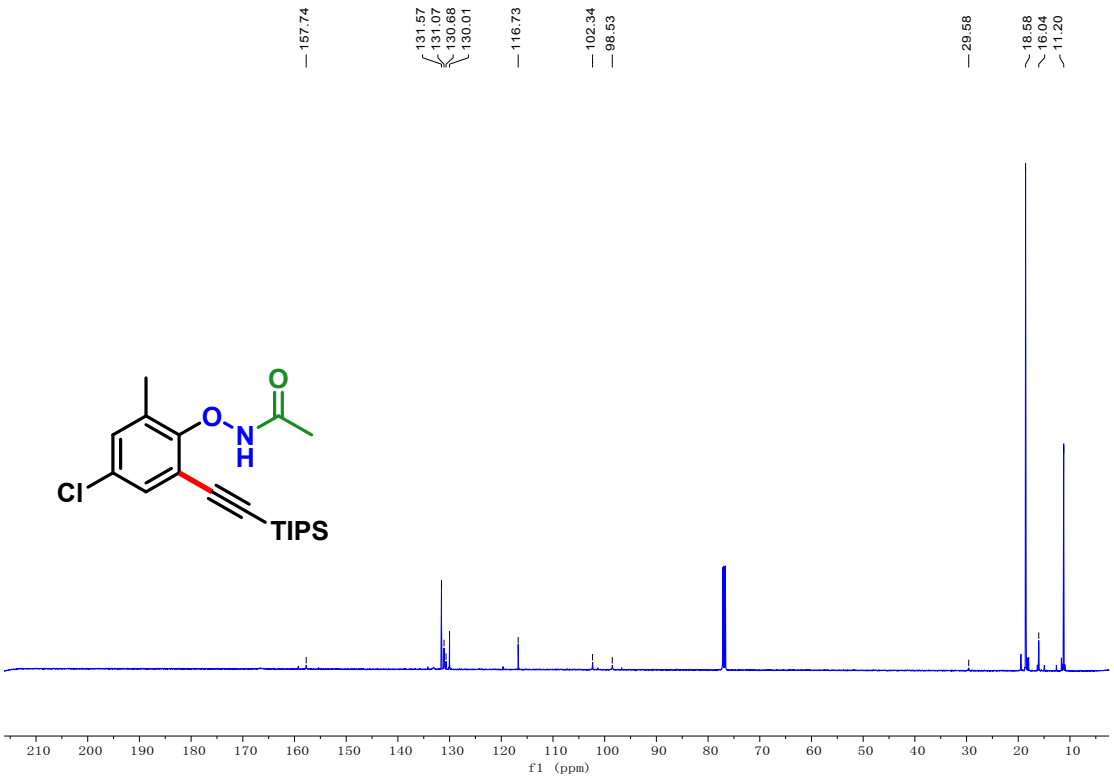
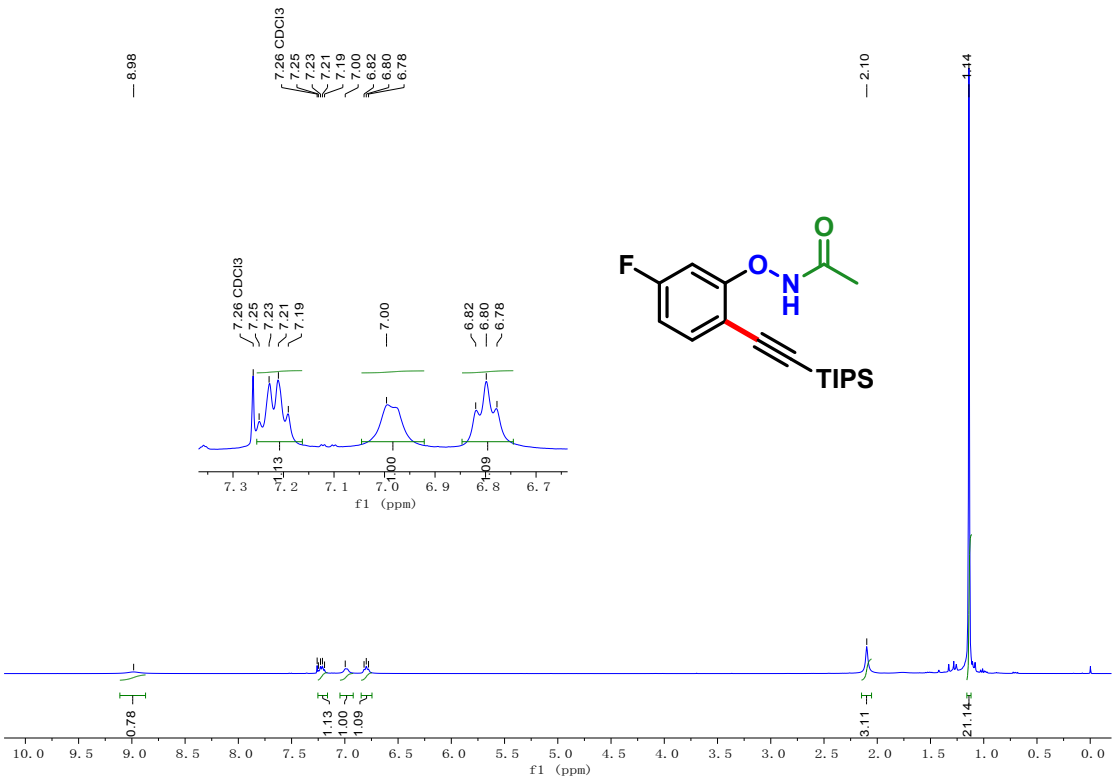
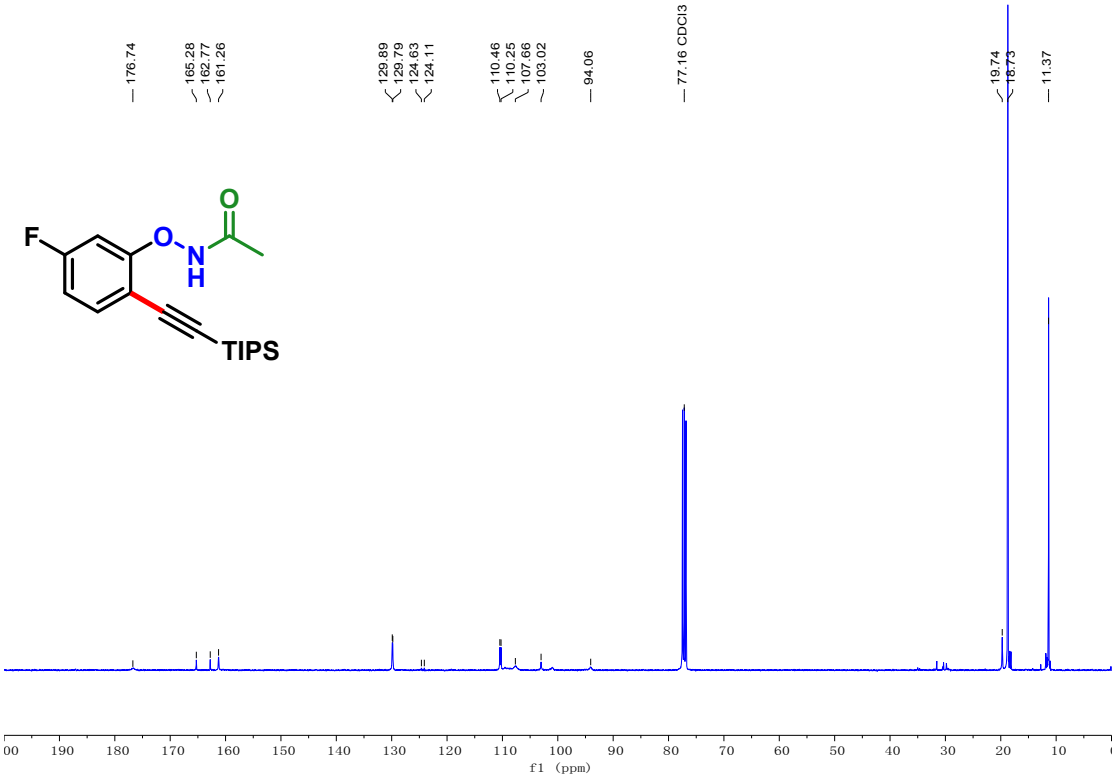
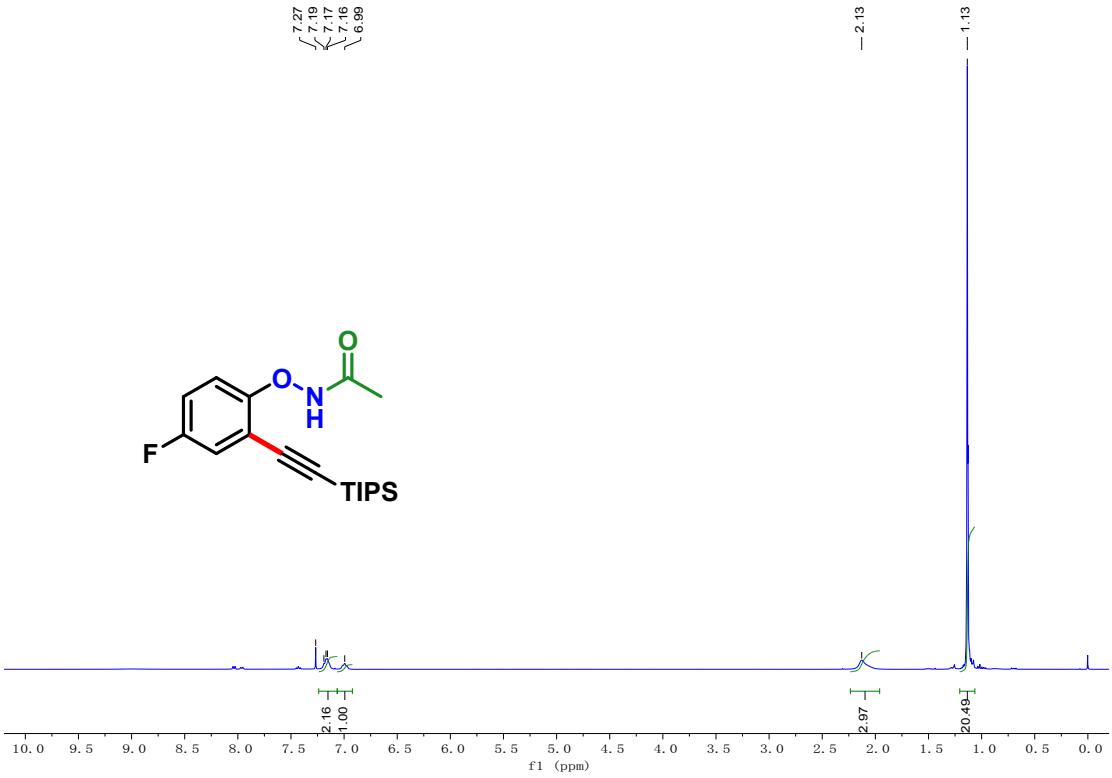
TIPS
f1 (ppm)

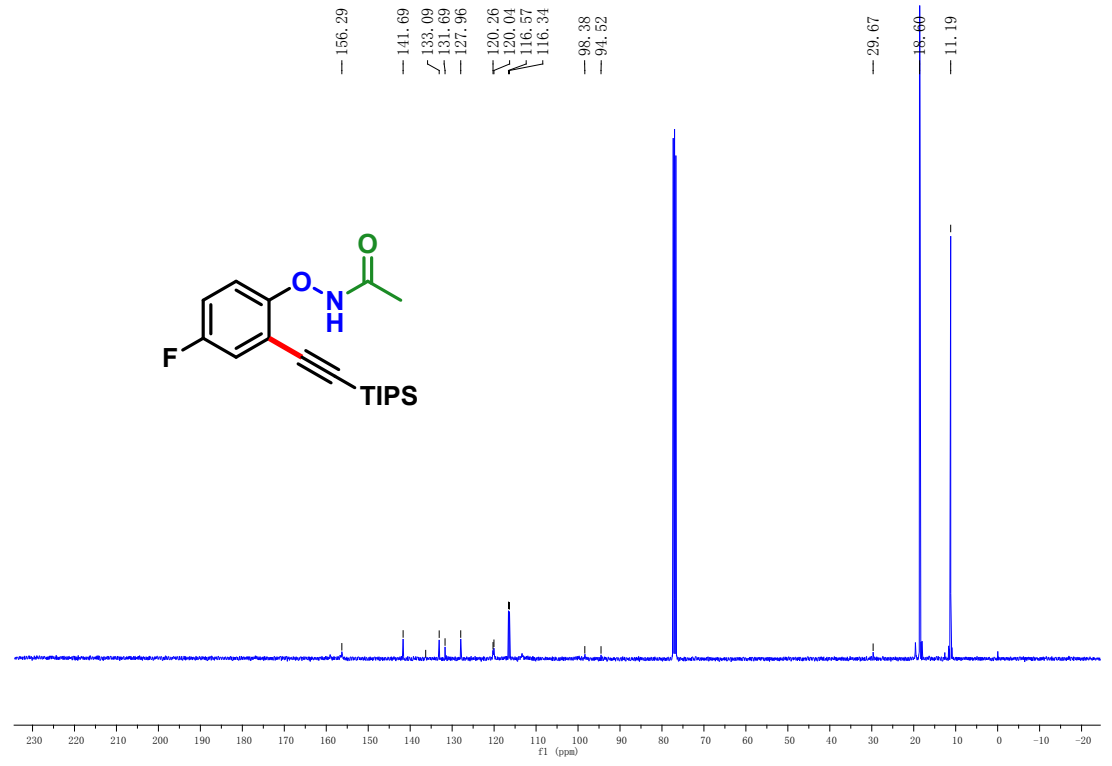

7.27
7.26
7.25
7.14
7.14
2.48
1.94
1.92
1.16
O
N
H
Cl
TIPS
1.14
1.00
2.86
2.93
21.50
f1 (ppm)

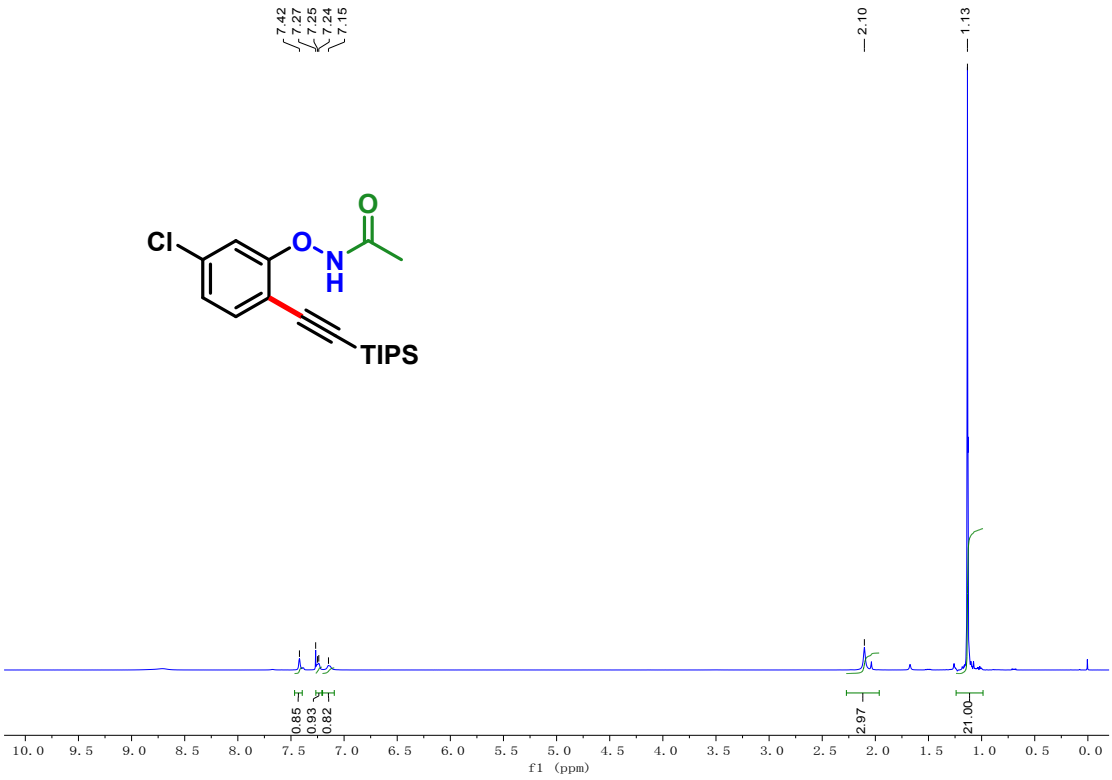

157.74
131.57
131.07
130.88
130.01
116.73
102.34
98.53
29.58
18.58
16.04
11.20
O
N
H
Cl
TIPS
f1 (ppm)

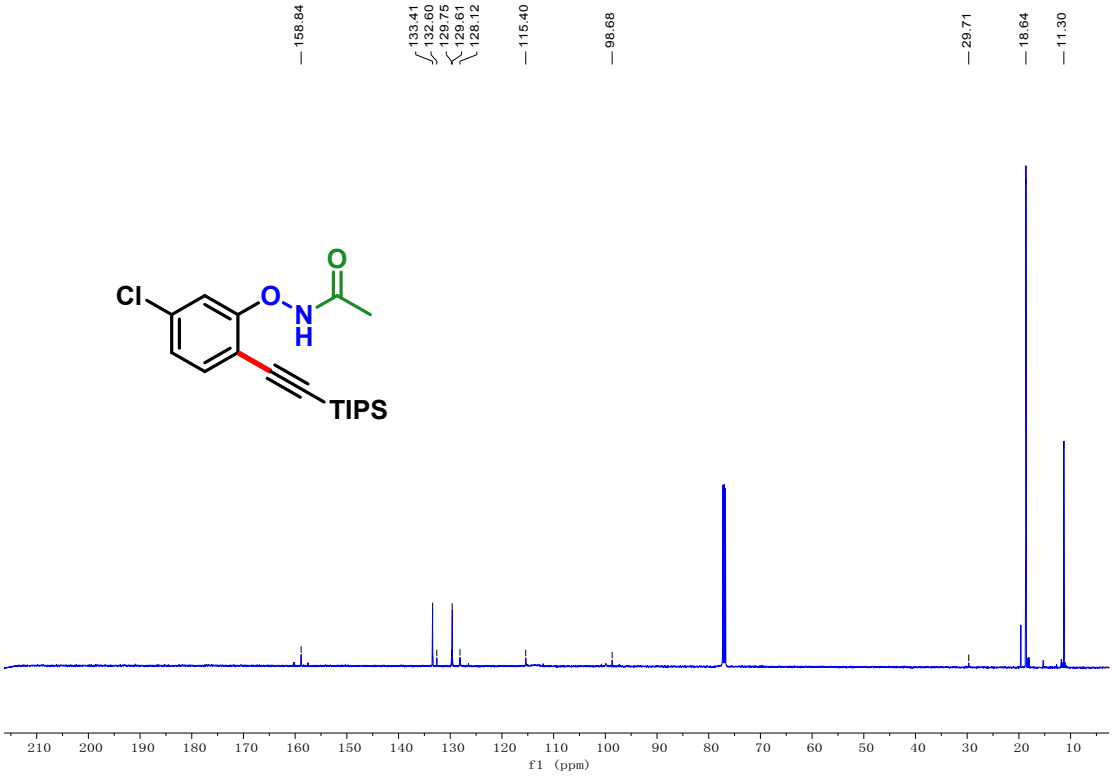

8.98
7.26 CDCl3
7.25
7.23
7.21
7.19
7.00
6.82
6.80
6.78
2.10
1.14
F
O
N
H
TIPS
0.78
1.13
1.00
1.09
3.11
21.14
f1 (ppm)

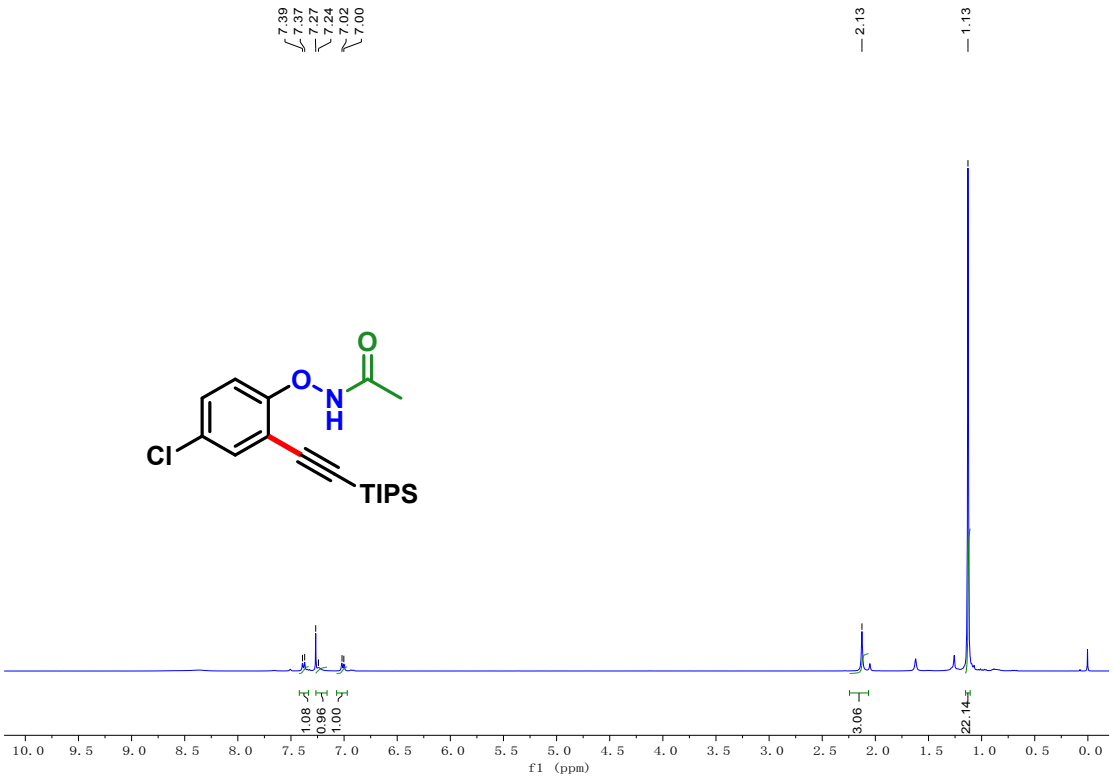

176.74
165.28
162.77
161.26
129.89
129.79
124.63
124.11
110.46
110.25
107.66
103.02
94.06
77.16 CDCl3
19.74
18.73
11.37
F
O
N
H
TIPS
f1 (ppm)

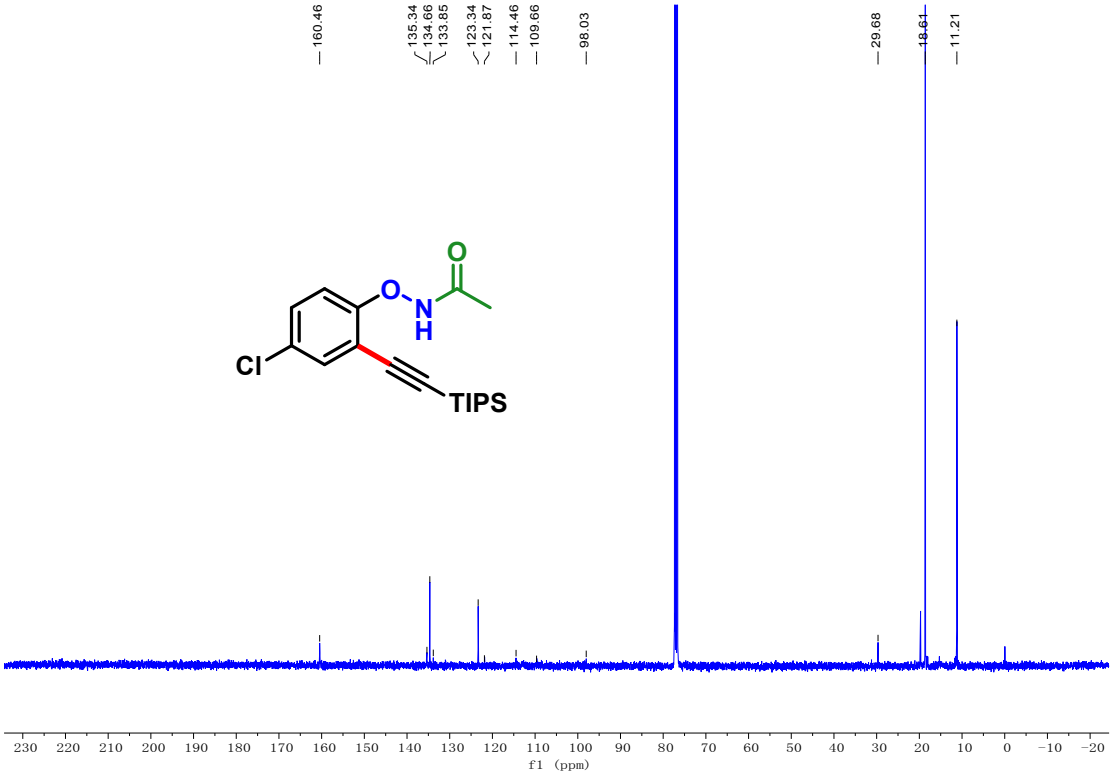

7.27
7.19
7.17
7.16
6.99
2.13
1.13
O
O
N
H
F
TIPS
2.16
1.00
2.97
20.49
f1 (ppm)

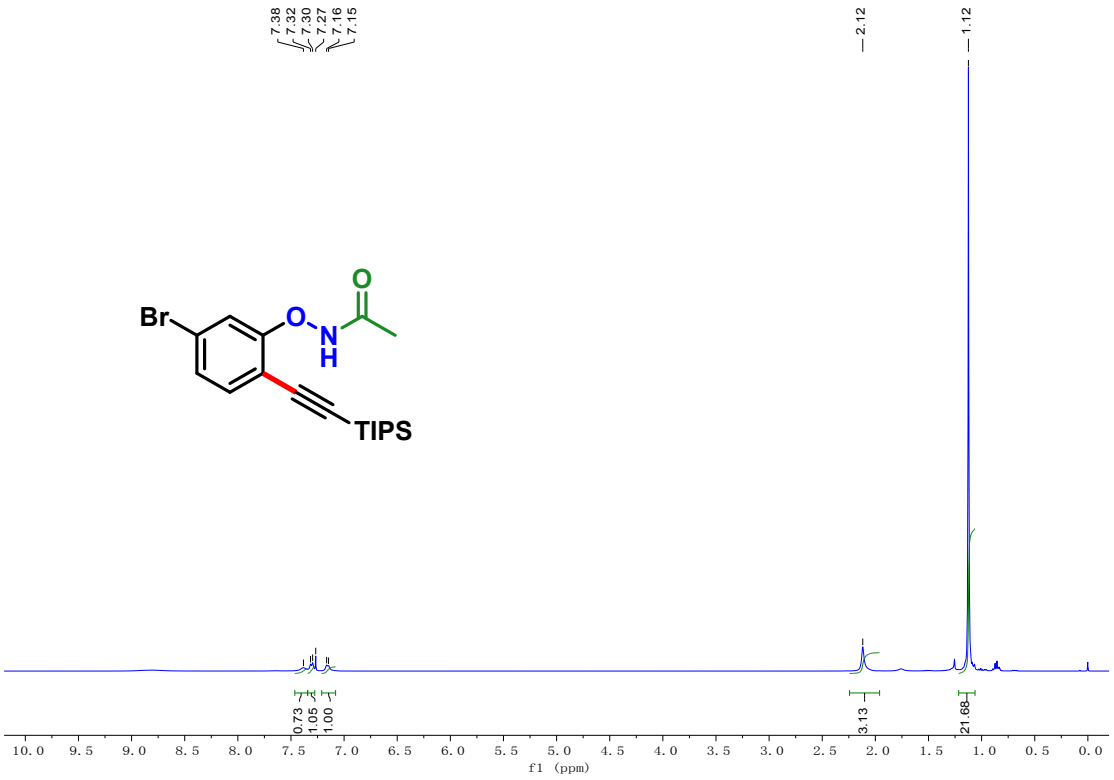

156.29
141.69
133.09
131.69
127.96
120.26
120.04
116.57
116.34
98.38
94.52
29.67
18.60
11.19
O
O
N
H
F
TIPS
f1 (ppm)

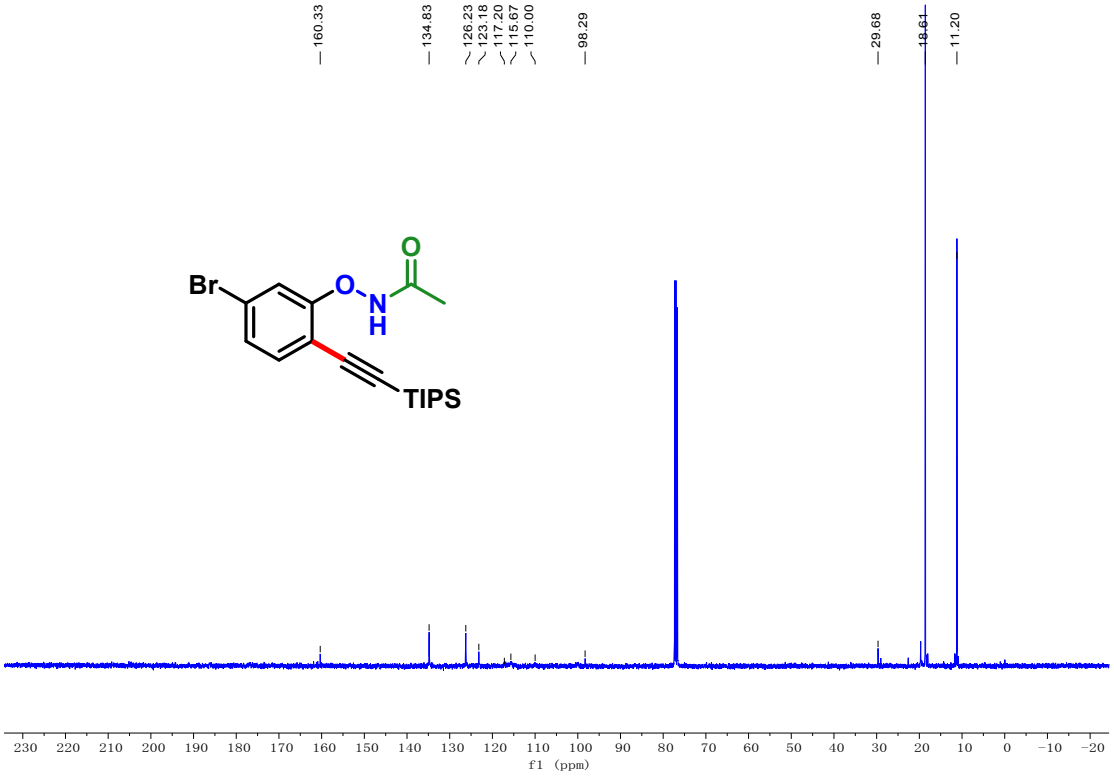

7.42
7.27
7.25
7.24
7.15
2.10
1.13
Cl
O
N
H
O
TIPS
0.85
0.93
0.82
2.97
21.00
f1 (ppm)

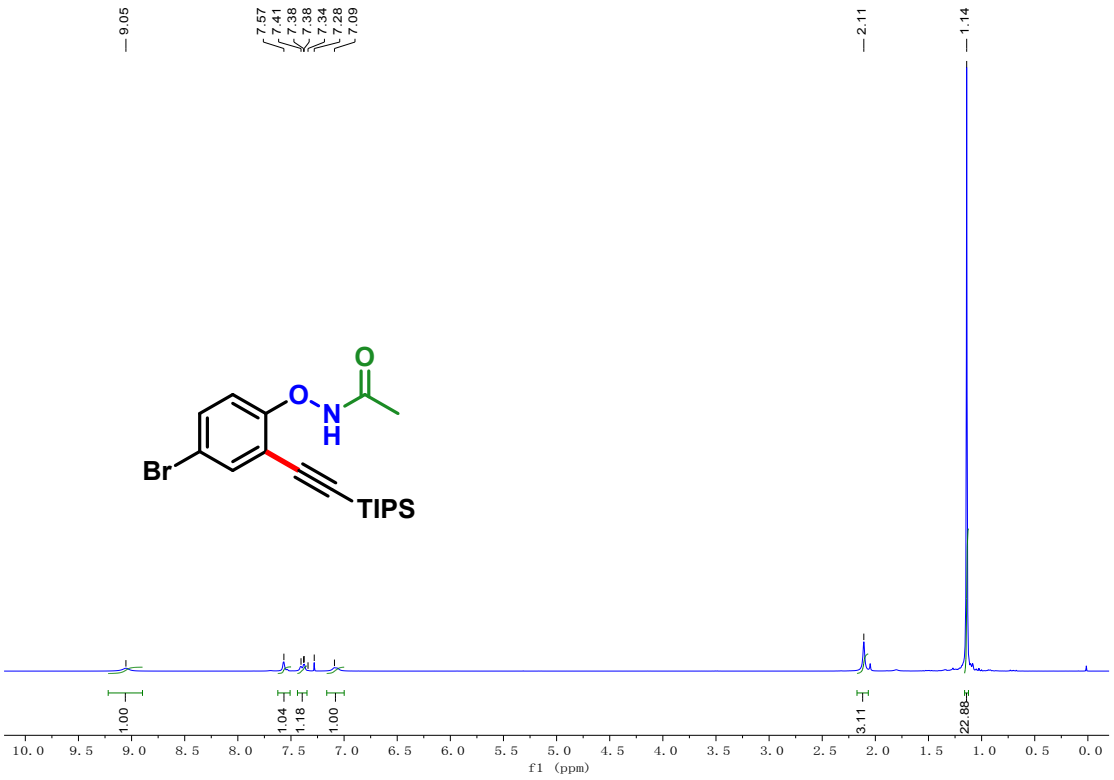

158.84
133.41
132.60
129.75
129.61
128.12
115.40
98.68
29.71
18.64
11.30
Cl
O
N
H
O
TIPS
f1 (ppm)

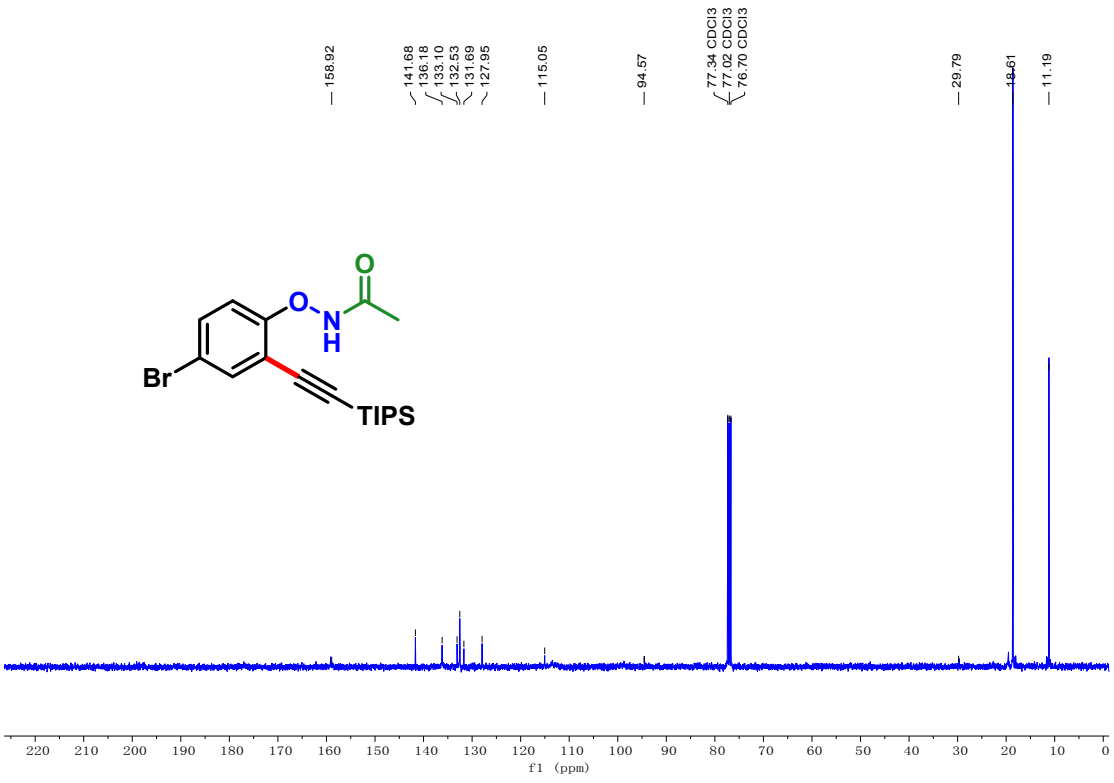

7.39
7.37
7.27
7.24
7.02
7.00
2.13
1.13
O
O
N
H
Cl
TIPS
1.08
0.96
1.00
3.06
22.14
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

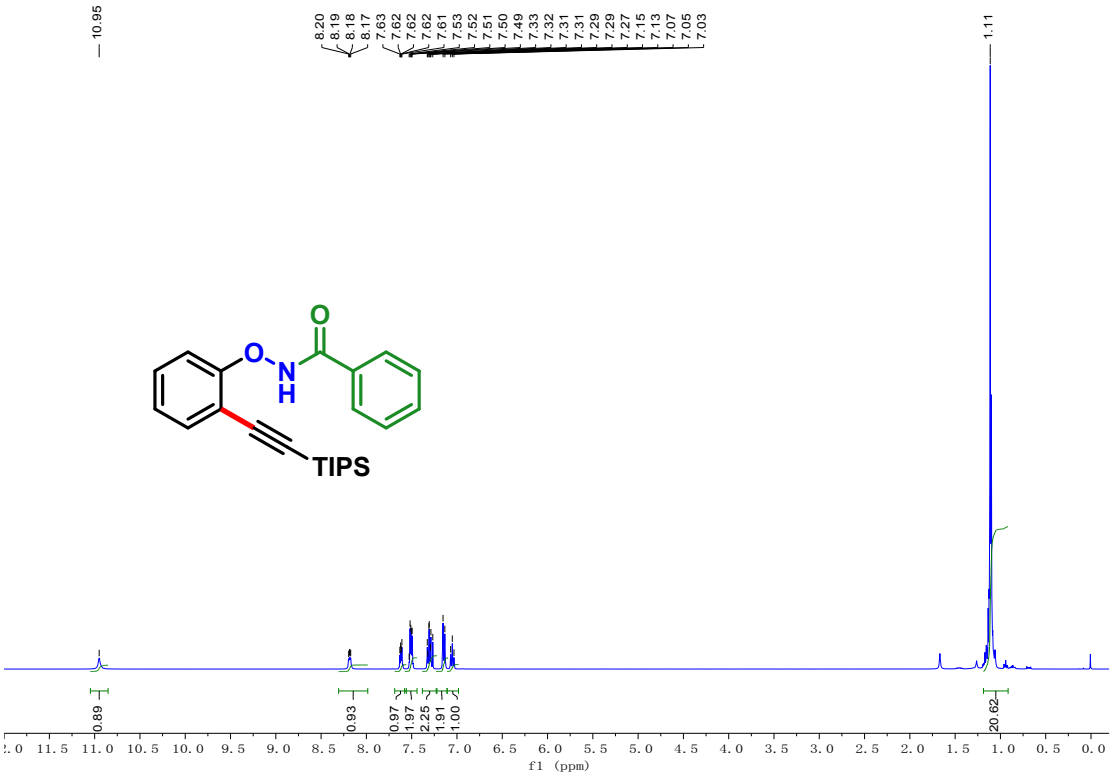

160.46
135.34
134.66
133.85
123.34
121.87
114.46
109.66
98.03
29.68
18.61
11.21
O
O
N
H
Cl
TIPS
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20
f1 (ppm)

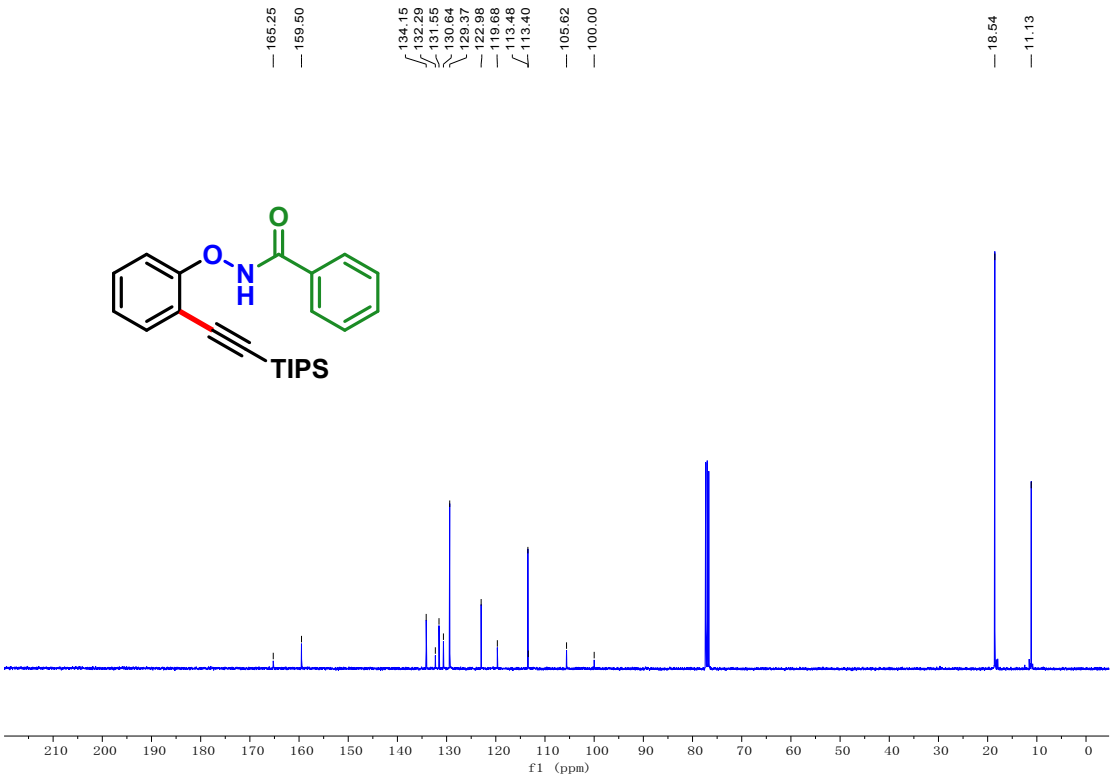

7.38
7.32
7.30
7.27
7.16
7.15
2.12
1.12
Br
O
N
H
O
TIPS
0.73
1.05
1.00
3.13
21.68
f1 (ppm)

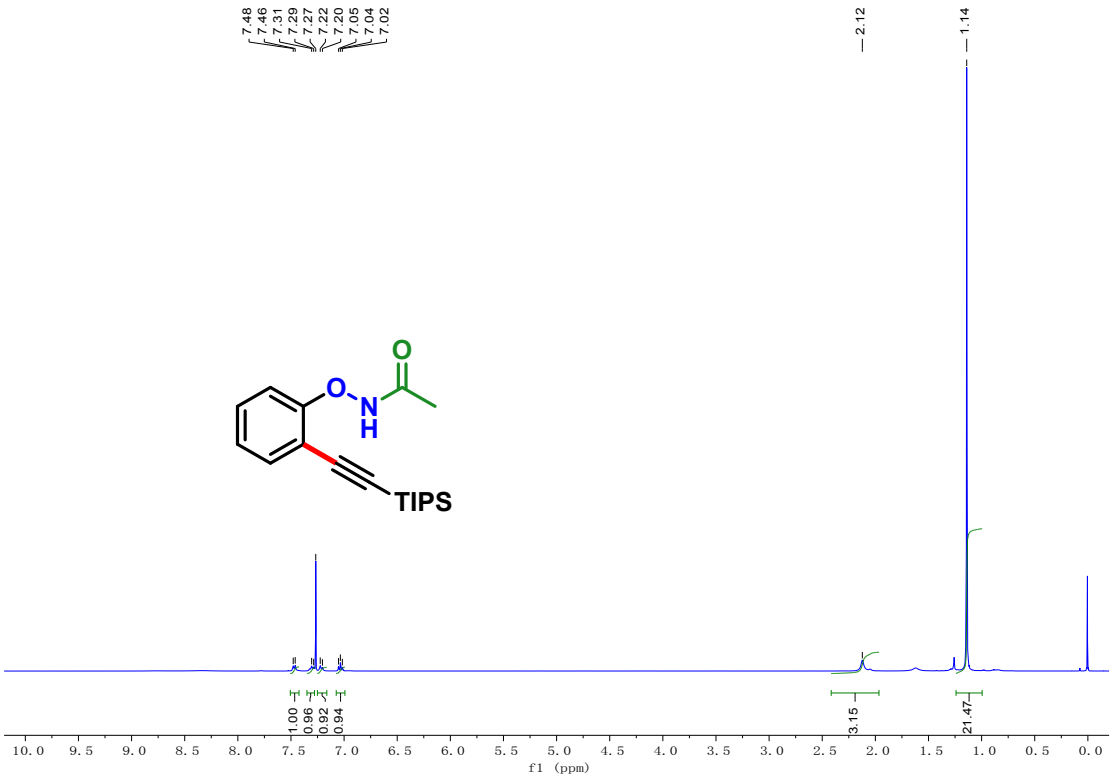

160.33
134.83
126.23
123.18
117.20
115.67
110.00
98.29
29.68
18.61
11.20
Br
O
N
H
O
TIPS
f1 (ppm)

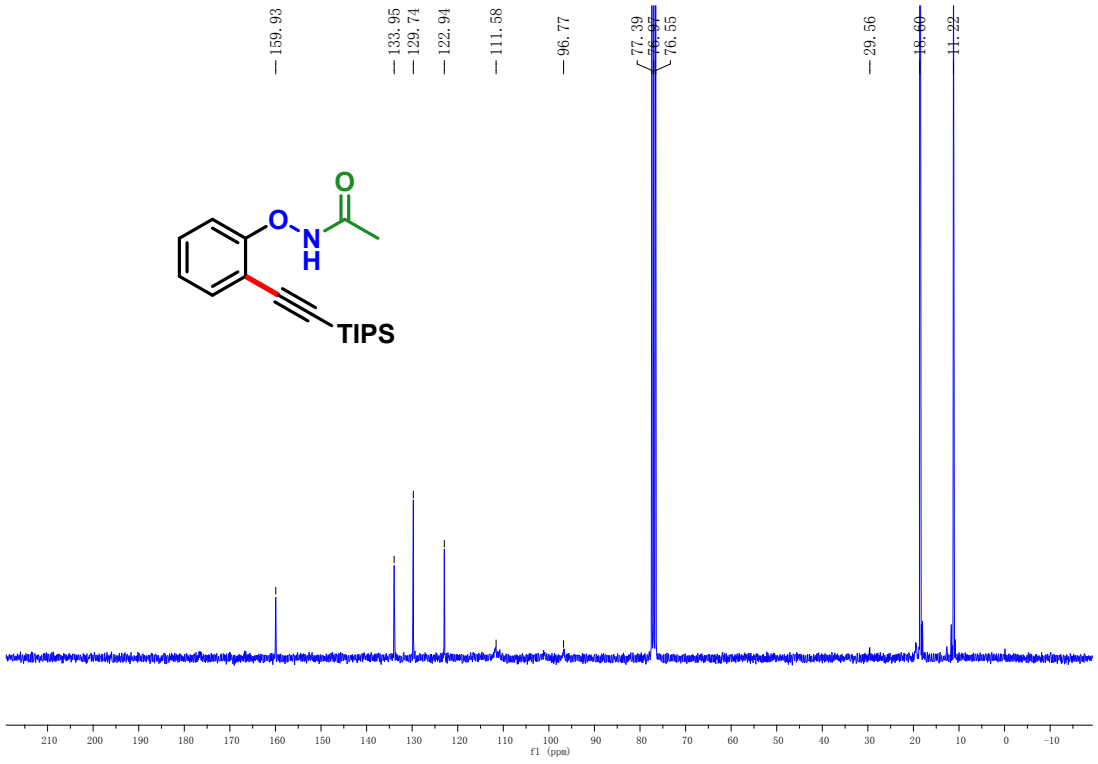

9.05
7.57
7.41
7.38
7.38
7.34
7.28
7.09
2.11
1.14
O
O
N
H
Br
TIPS
1.00
1.04
1.18
1.00
3.11
22.88
f1 (ppm)

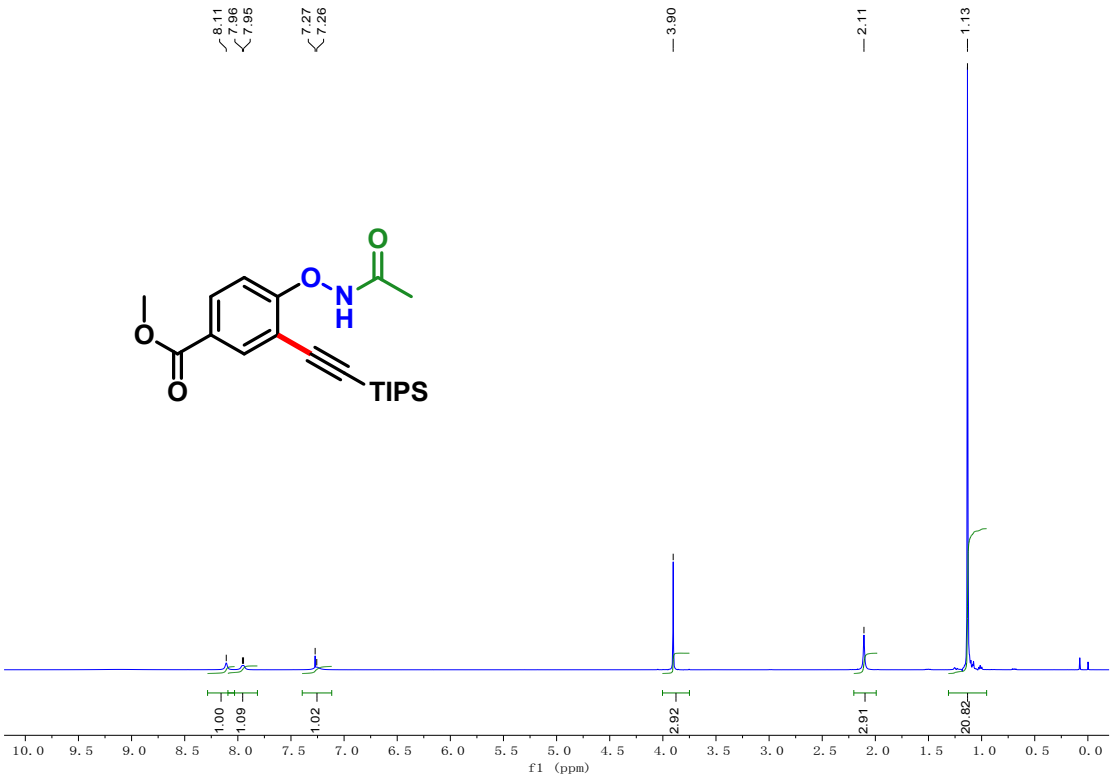

158.92
141.68
136.18
133.10
132.53
131.69
127.95
115.05
94.57
77.34 CDCl3
77.02 CDCl3
76.70 CDCl3
29.79
18.61
11.19
O
O
N
H
Br
TIPS
f1 (ppm)

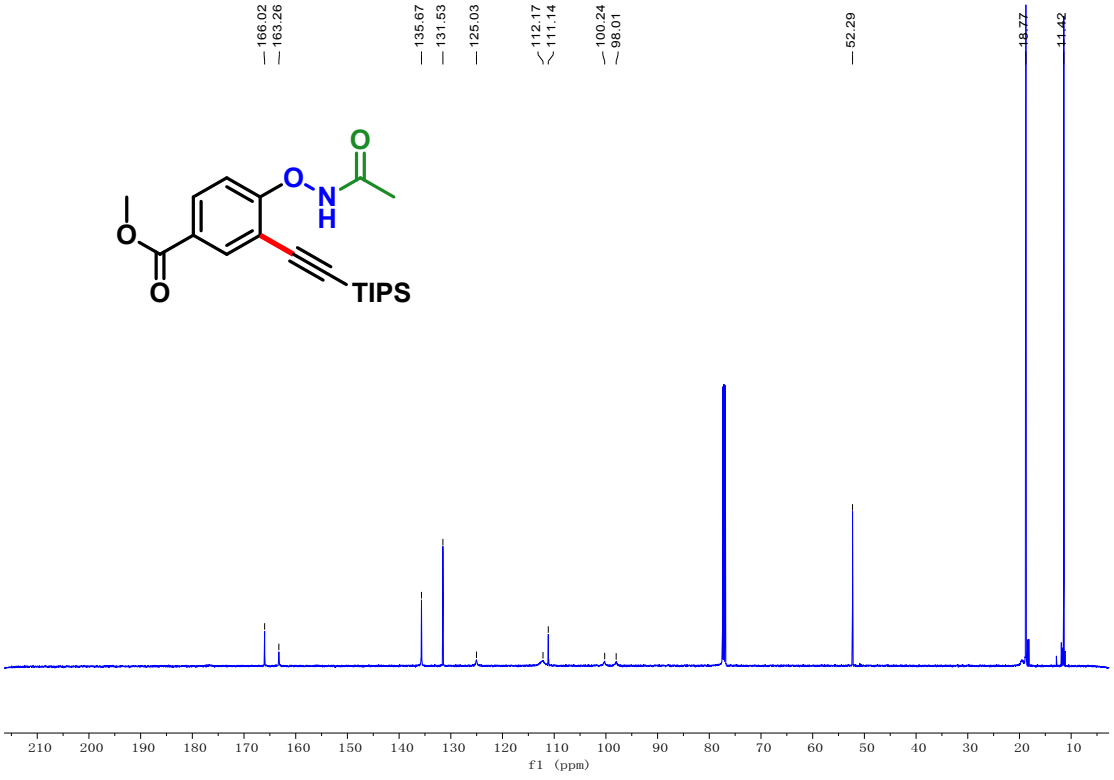

10.95
8.20
8.19
8.18
8.17
7.63
7.62
7.62
7.62
7.61
7.53
7.52
7.51
7.50
7.49
7.33
7.32
7.31
7.31
7.29
7.29
7.27
7.15
7.13
7.07
7.05
7.03
1.11
O
N
H
TIPS
0.89
0.93
0.97
1.97
2.25
1.91
1.00
20.62
f1 (ppm)

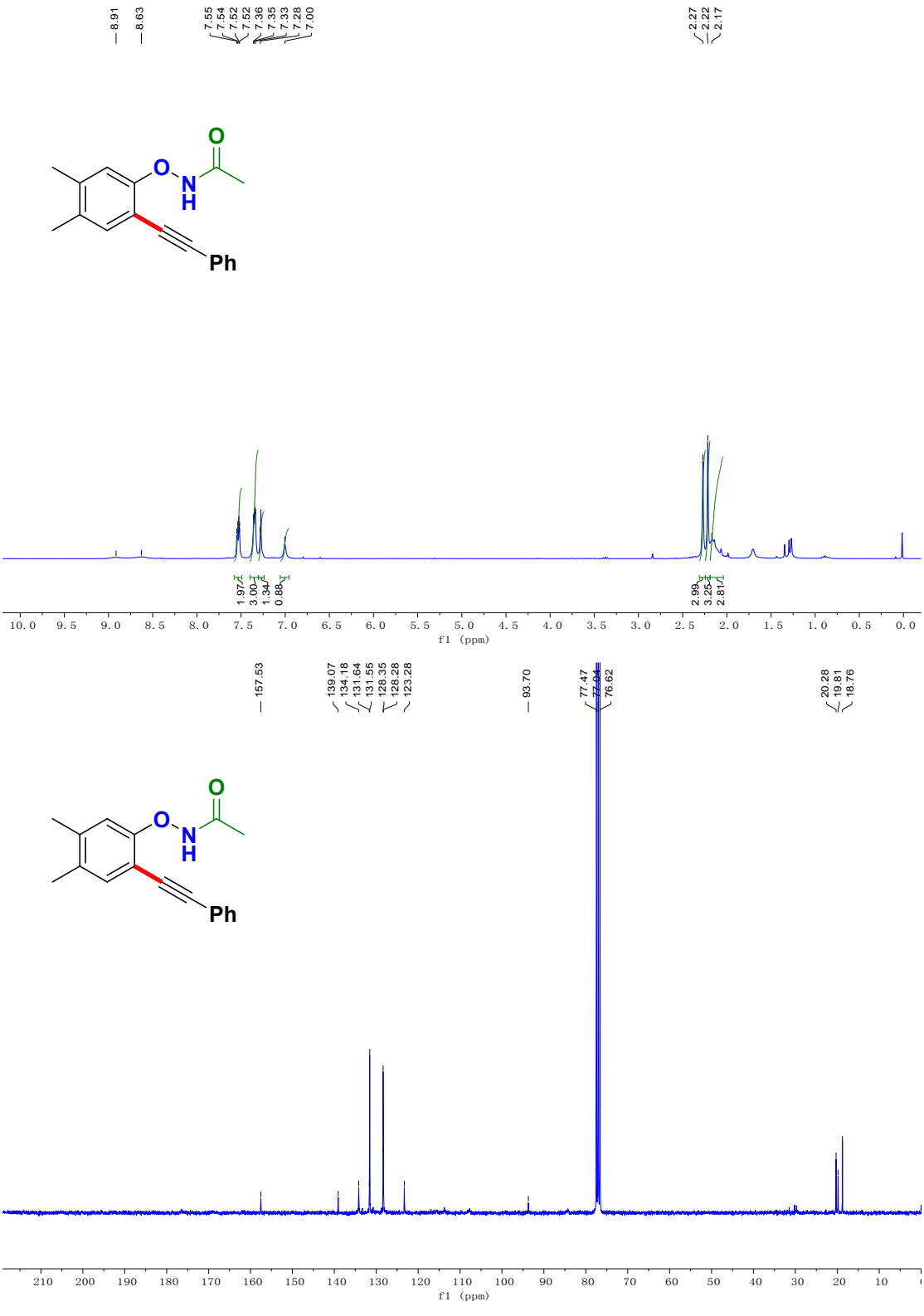

165.25
159.50
134.15
132.29
131.55
130.64
129.37
122.98
119.68
113.48
113.40
105.62
100.00
18.54
11.13
O
N
H
TIPS
f1 (ppm)

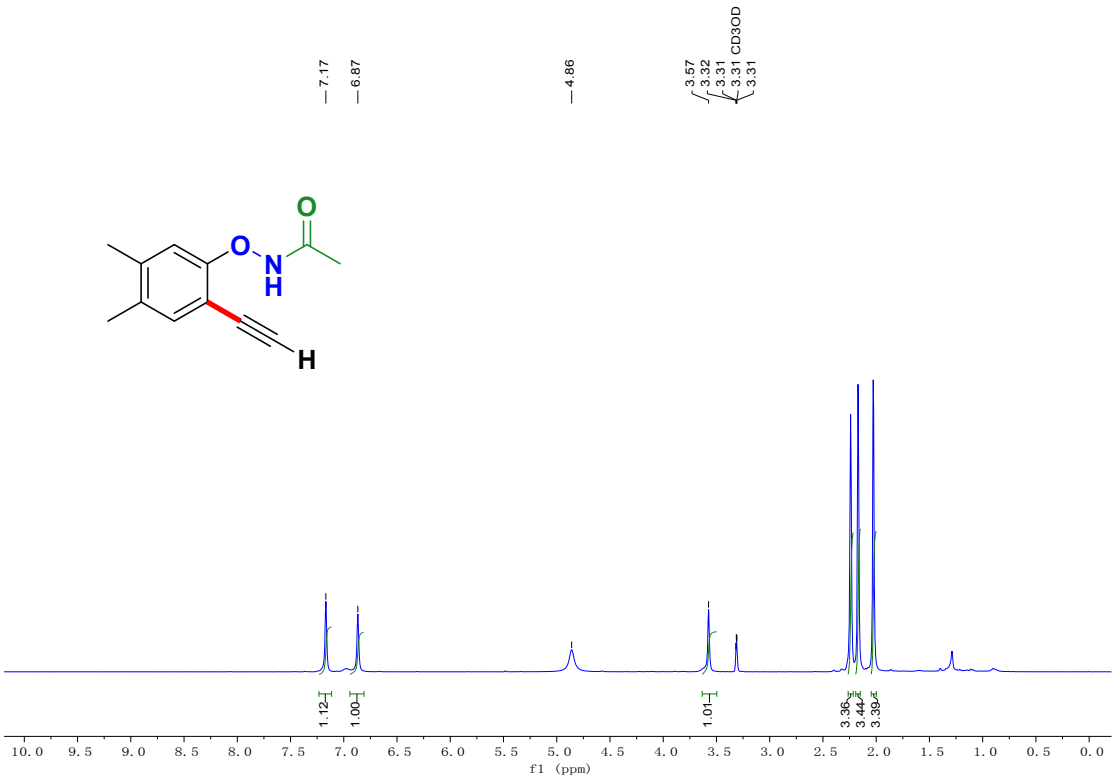

7.48
7.46
7.31
7.29
7.27
7.22
7.20
7.05
7.04
7.02
2.12
1.14
O
O
N
H
TIPS
1.00
0.96
0.92
0.94
3.15
21.47
f1 (ppm)

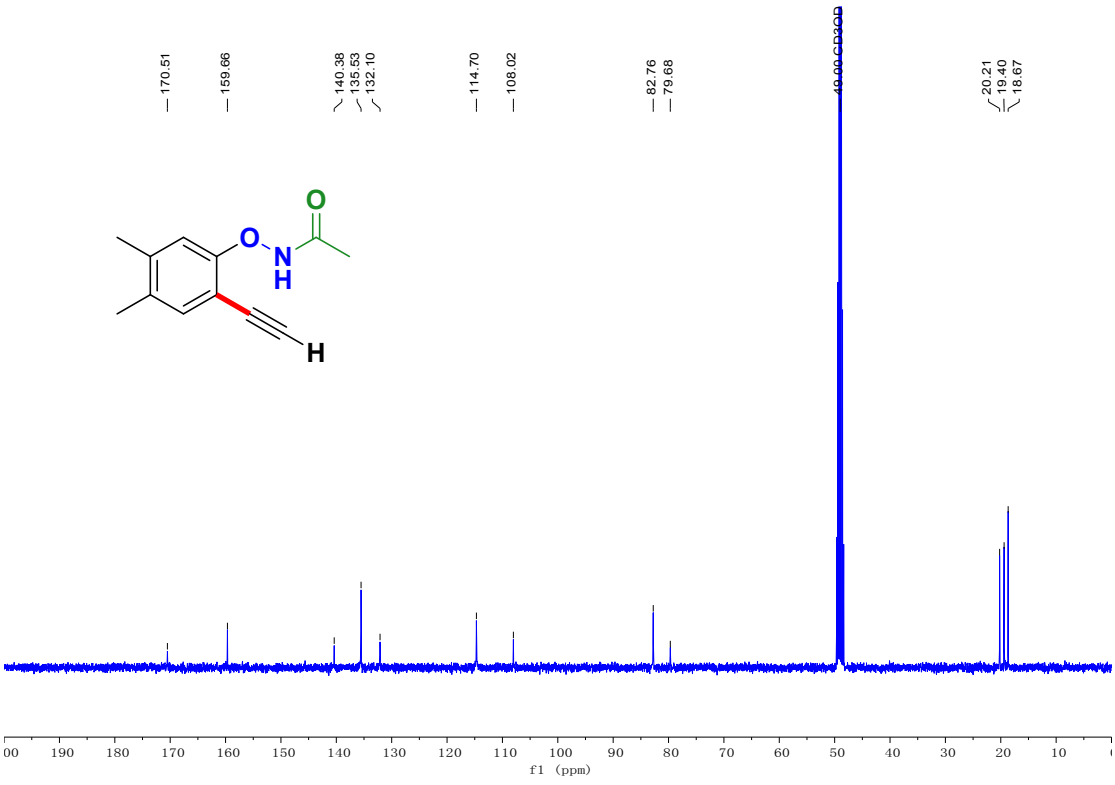

159.93
133.95
129.74
122.94
111.58
96.77
77.39
76.97
76.55
29.56
18.60
11.22
O
O
N
H
TIPS
f1 (ppm)

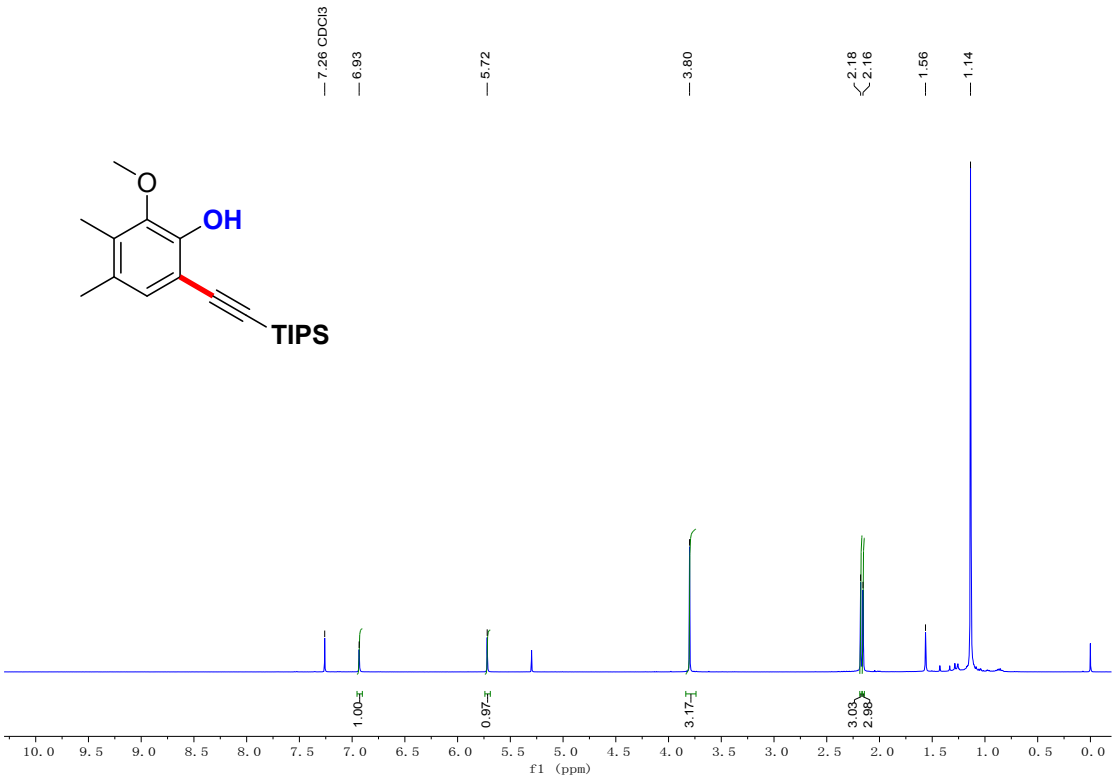

8.11
7.96
7.95
7.27
7.26
3.90
2.11
1.13
O
O
N
H
O
O
TIPS
1.00
1.09
1.02
2.92
2.91
20.82
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

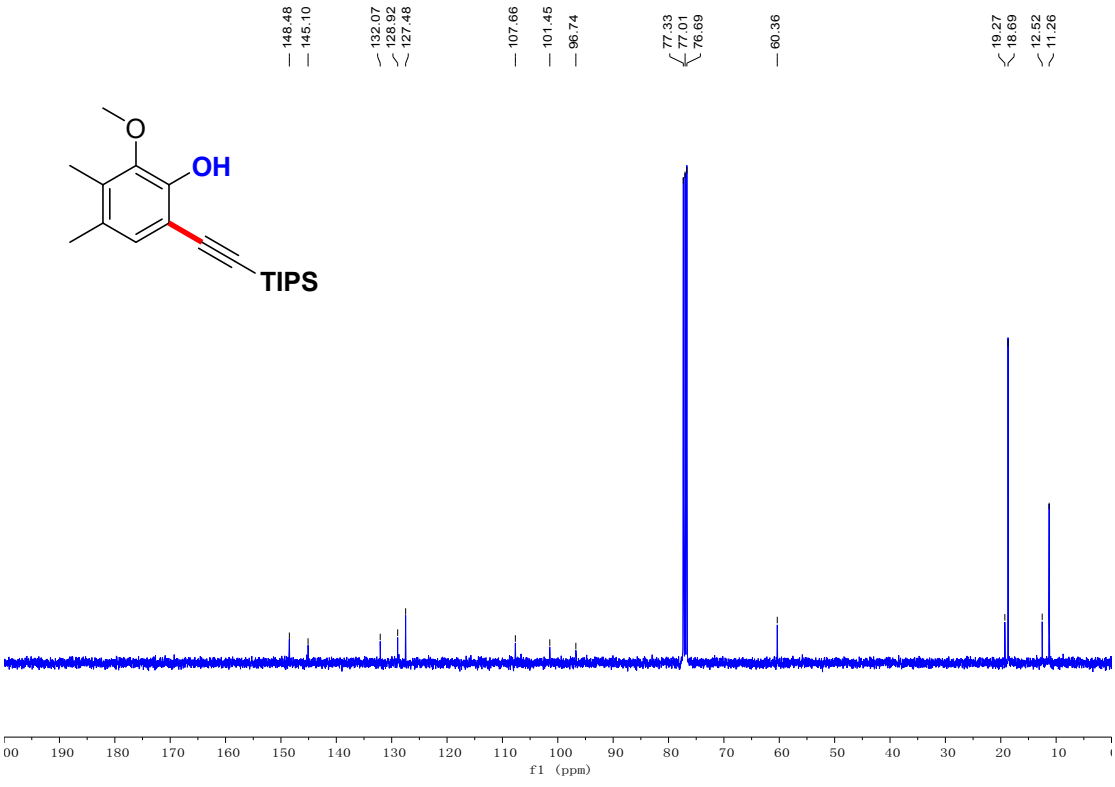

166.02
163.26
135.67
131.53
125.03
112.17
111.14
100.24
98.01
52.29
18.77
11.42
O
O
N
H
O
O
TIPS
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

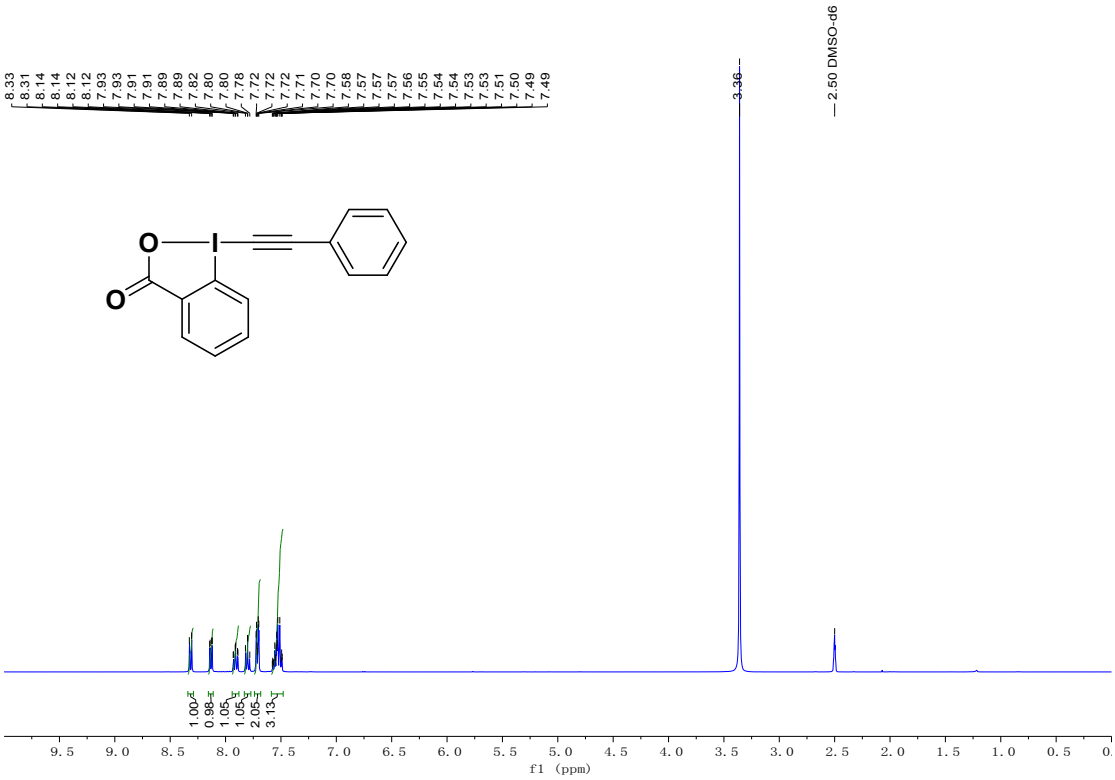

8.91
8.63
7.55
7.54
7.52
7.52
7.36
7.35
7.33
7.28
7.00
2.27
2.22
2.17
O
O
N
H
Ph
1.97
3.00
1.34
0.88
2.99
3.25
2.81
f1 (ppm)
157.53
139.07
134.18
131.64
131.55
128.35
128.28
123.28
93.70
77.47
77.04
76.62
20.28
19.81
18.76
f1 (ppm)

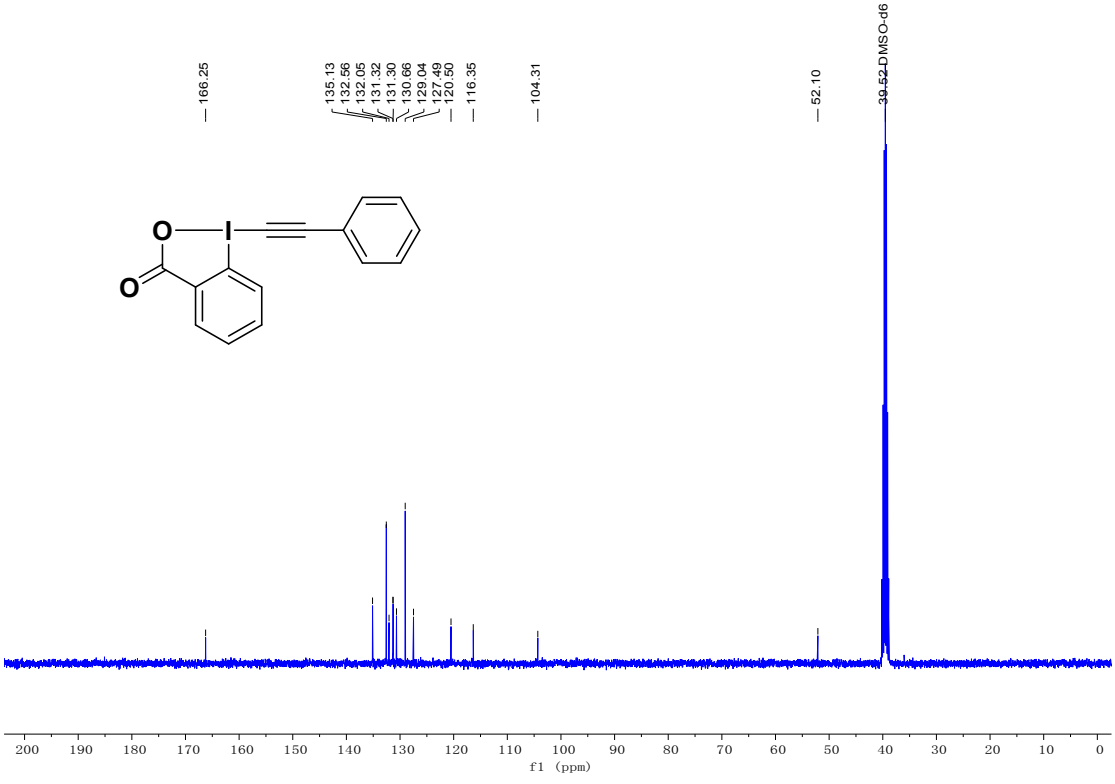

7.17
6.87
4.86
3.57
3.32
3.31
3.31 CD3OD
3.31
O
O
N
H
H
1.12
1.00
1.01
3.36
3.44
3.39
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

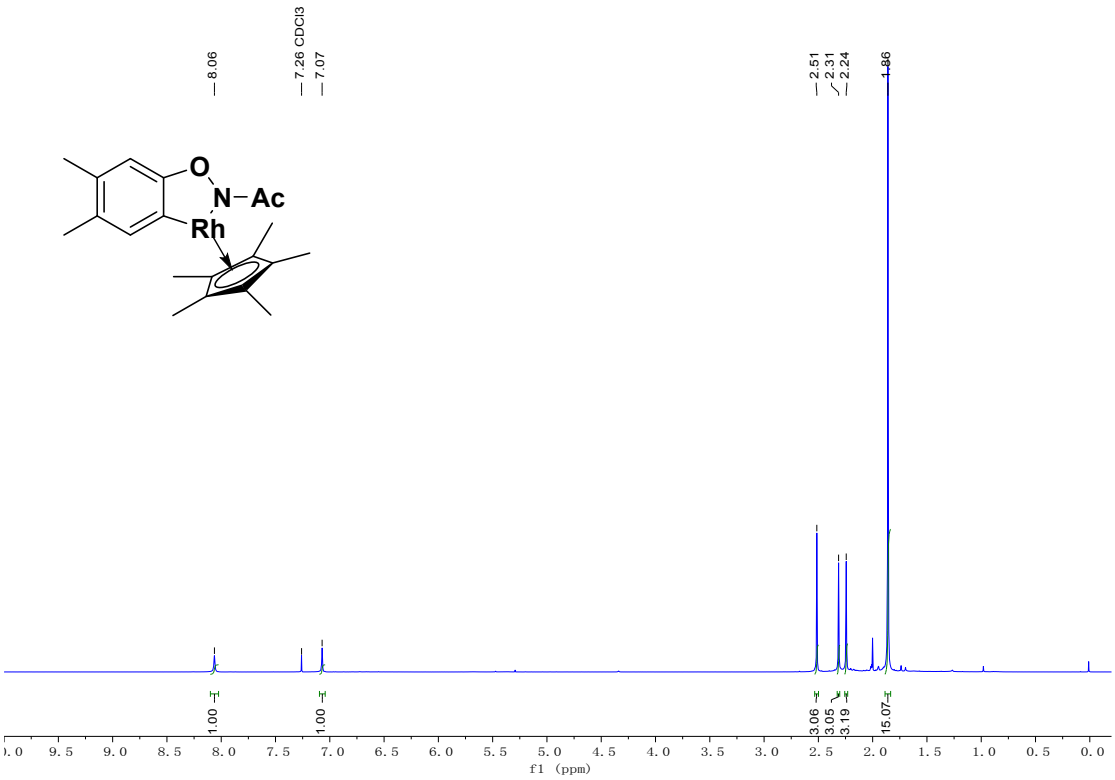

170.51
159.66
140.38
135.53
132.10
114.70
108.02
82.76
79.68
49.00 CD3OD
20.21
19.40
18.67
O
O
N
H
H
00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

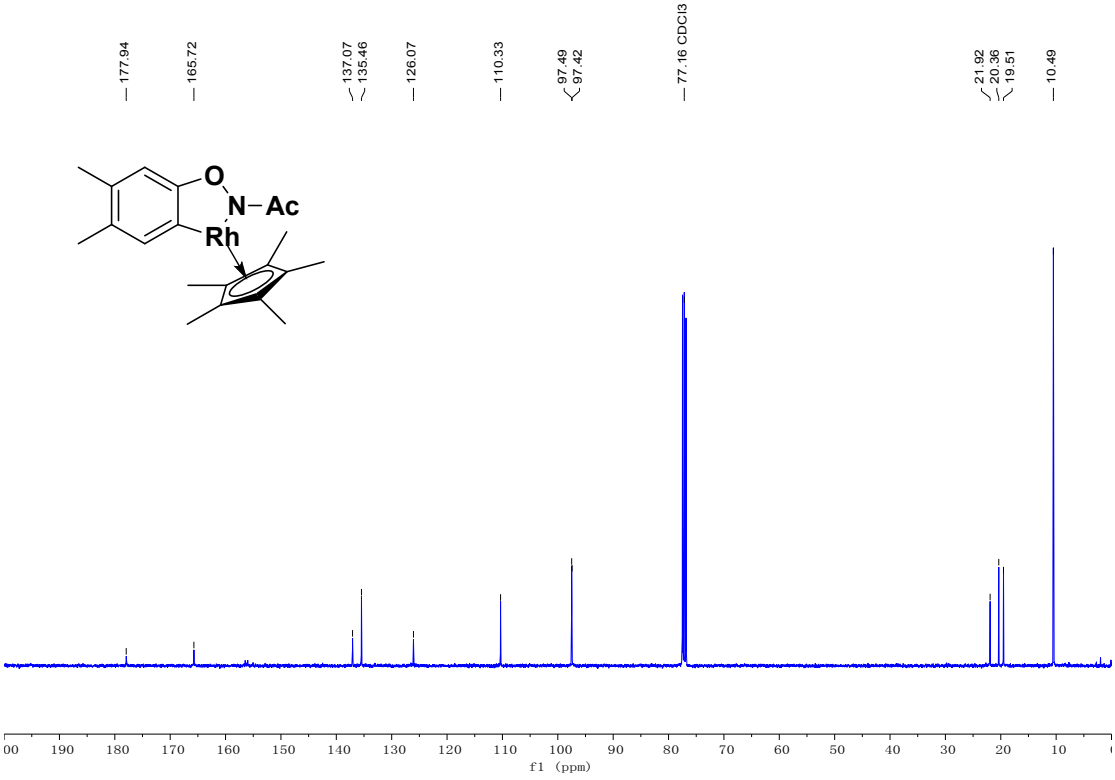

7.26 CDCl3
6.93
5.72
3.80
2.18
2.16
1.56
1.14
O
OH
TIPS
1.00
0.97
3.17
3.03
2.98
f1 (ppm)


148.48
145.10
132.07
128.92
127.48
107.66
101.45
96.74
77.33
77.01
76.69
60.36
19.27
18.69
12.52
11.26
O
OH
TIPS
f1 (ppm)


8.33
8.31
8.14
8.14
8.12
8.12
7.93
7.93
7.91
7.91
7.89
7.89
7.82
7.80
7.80
7.78
7.72
7.72
7.72
7.71
7.70
7.70
7.58
7.57
7.57
7.57
7.56
7.55
7.54
7.54
7.53
7.53
7.51
7.50
7.49
7.49
3.36
2.50 DMSO-d6
O
I
O
1.00
0.98
1.05
1.05
2.05
3.13
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


166.25
135.13
132.56
132.05
131.32
131.30
130.66
129.04
127.49
120.50
116.35
104.31
52.10
39.52 DMSO-d6
O
I
O
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.06
7.26 CDCl3
7.07
2.51
2.31
2.24
1.86
O
N−Ac
Rh
1.00
1.00
3.06
3.05
3.19
15.07
f1 (ppm)


177.94
165.72
137.07
135.46
126.07
110.33
97.49
97.42
77.16 CDCl3
21.92
20.36
19.51
10.49
O
N−Ac
Rh
f1 (ppm)