Supporting information

Reduction of sulfoxides and pyridine-N-oxides over iron powder with water as hydrogen source promoted by carbon dioxide

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Contents Table

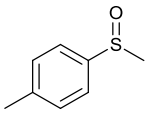
1. General procedure for the reduction of sulfoxides

A mixture of sulfoxides (1 mmol), Fe (2 mmol, 0.1117 g), H_2O (2 mL) was placed in a 50 mL stainless steel autoclave equipped with an inner glass tube at room temperature. The vessel was sealed and CO_2 was subsequently introduced into the autoclave. The system was heated under the predetermined reaction temperature for 25 min to reach the equilibration, then the final pressure was adjusted to the desired pressure by introducing the correct amount of CO_2. After the reaction was finished, the vessel was cooled within an ice-bath and the pressure was released slowly to atmospheric pressure. The products were diluted with ethyl acetate and analyzed by GC. The residue was purified by column chromatography on silica gel (200-300 mesh, eluting with *n*-hexane) to afford the desired product. The isolated products were further identified with NMR spectra and GC-MS, which are consistent with those reported in the literature.

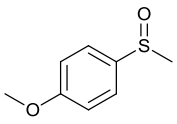
2. General procedure for the reduction of pyridine-N-oxides

A mixture of pyridine-N-oxides (1 mmol), Fe (2 mmol, 0.1117 g), H_2O (2 mL) was placed in a 50 mL stainless steel autoclave equipped with an inner glass tube at room temperature. The vessel was sealed and CO_2 was subsequently introduced into the autoclave. The system was heated under the predetermined reaction temperature for 25 min to reach the equilibration, then the final pressure was adjusted to the desired pressure by introducing the correct amount of CO_2. After the reaction was finished, the vessel was cooled with an ice-bath and the pressure was released slowly to atmospheric pressure. The products were diluted with diethyl ether or acetone and analyzed by GC. The residue was purified by column chromatography on silica gel (200-300 mesh, eluting with *n*-hexane and ethyl acetate or methanol) to afford the desired product. The isolated products were further identified with NMR spectra and GC-MS, which are consistent with those reported in the literature.

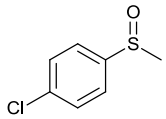
3. Analytic data of the substrate and products



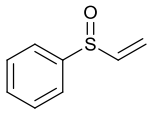
p-Tolyl methyl sulfoxide.[1] The product was obtained as a yellow liquid. ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 7.52 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.4 Hz, 2H), 2.69 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):142.44, 141.48, 130.02, 123.52, 43.96, 21.37.



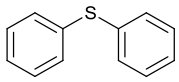
4-Methoxyphenyl methyl sulfoxide.[1] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.60 – 7.48 (m, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 3.79 (s, 3H), 2.64 (s, 3H).^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm): 161.96, 136.51, 125.46, 114.85, 55.53, 43.96.



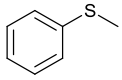
p-Chlorophenyl methyl sulfoxide. [1] The product was obtained as a yellow solid. ^{1}H NMR (400 MHz, CDCl3) δ (ppm):7.58 – 7.52 (m, 2H), 7.49 – 7.43 (m, 2H), 2.68 (s, 3H). ^{13}C NMR (100.6 MHz, CDCl3) δ (ppm):144.14, 137.22, 129.64, 124.98, 43.99.



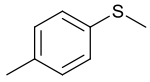
Phenyl vinyl sulfoxide.[2] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.67 – 7.40 (m, 5H), 6.58 (dd, *J* = 16.5, 9.6 Hz, 1H), 6.19 (d, *J* = 16.5 Hz, 1H), 5.88 (d, *J* = 9.6 Hz, 1H).^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):143.17, 142.86, 131.30, 129.48, 124.69, 120.80.



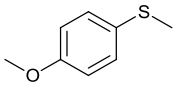
Diphenyl sulfide.[3] The product was obtained as a pale yellow liquid (0.169g, 90.9% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.39 (d, *J* = 7.4 Hz, 4H), 7.34 (dd, *J* = 9.9, 4.8 Hz, 4H), 7.28 (dd, *J* = 7.6, 5.1 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):135.86, 131.10, 129.25, 127.10. EI-MS, *m/z* (%): 186 (100) [M^+].



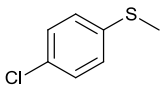
Phenyl methyl sulfide.[3] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.32 – 7.20 (m, 4H), 7.17 – 7.07 (m, 1H), 2.47 (s, 3H).^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):138.40, 128.80, 126.61, 125.00, 15.83. EI-MS, *m/z* (%): 124 (100) [M^+].



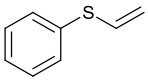
p-Tolyl methyl sulfide.[4] The product was obtained as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.24 – 7.16 (m, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 2.48 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):135.03, 134.68, 129.59, 127.27, 20.91, 16.51. EI-MS, *m/z* (%): 138 (100) [M^+].



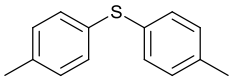
4-Methoxyphenyl methyl sulfide.[4] The product was obtained as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.23 (d, *J* = 8.6 Hz, 2H), 6.81 (d, *J* = 8.6 Hz, 2H), 3.75 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):158.21, 130.21, 128.78, 114.62, 55.37, 18.09. EI-MS, *m/z* (%): 154 (93) [M^+].



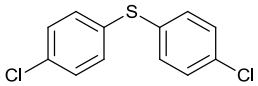
p-Chlorophenyl methyl sulfide.[4] The product was obtained as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.24 (d, *J* = 8.5 Hz, 2H), 7.20 – 7.12 (m, 2H), 2.46 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):136.99, 130.89, 128.90, 127.90, 16.09. EI-MS, *m/z* (%): 158 (100) [M^+].



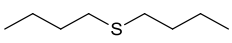
Phenyl vinyl sulfide.[5] The product was obtained as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.50 (d, *J* = 7.5 Hz, 1H), 7.39 (d, *J* = 7.3 Hz, 2H), 7.36 – 7.30 (m, 3H), 6.55 (dd, *J* = 16.6, 9.6 Hz, 1H), 5.36 (dd, *J* = 13.1, 5.7 Hz, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):134.26, 131.89, 130.52, 129.13, 127.52, 127.16, 127.13, 115.49. EI-MS, *m/z* 136 (77) [M^+].



4,4'-Dimethyldiphenylsulfide.[6] The product was obtained as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.23 (d, *J* = 8.1 Hz, 4H), 7.10 (d, *J* = 8.0 Hz, 4H), 2.32 (s, 6H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):136.91, 132.66, 131.06, 129.91, 21.07. EI-MS, *m/z* (%): 214 (100) [M^+].



4-CHLOROPHENYL SULFIDE.[6] The product was obtained as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):7.36 – 7.13 (m, 8H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):133.93, 133.46, 132.30, 129.48. EI-MS, *m/z* 255 (14) [M^+].



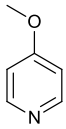
Di-N-butyl sulfide.[6] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):2.56 – 2.44 (m, 4H), 1.56 (ddd, *J* = 12.5, 8.8, 7.2 Hz, 4H), 1.40 (dq, *J* = 14.3, 7.2 Hz, 4H), 0.91 (t, *J* = 7.3 Hz, 6H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ

(ppm):31.85, 22.07, 13.72. EI-MS, *m/z* (%): 146 (43) [M^+].

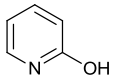
Tetrahydrothiophene.[7] The product was obtained as a pale yellow liquid.^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):2.95 – 2.64 (m, 4H), 2.03 – 1.78 (m, 4H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):31.80, 31.08. EI-MS, *m/z* (%): 88 (74) [M^+].

Pyridine.[8] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):8.67 – 8.55 (m, 2H), 7.66 (dd, *J* = 10.7, 4.6 Hz, 1H), 7.34 – 7.22 (m, 2H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):149.86, 135.93, 123.72. EI-MS, *m/z* (%): 88 (74) [M^+].

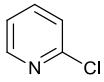
2-Methylpyridine.[9] The product was obtained as a pale yellow liquid.^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):8.47 (d, *J* = 4.4 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.12 (d, *J* = 7.8 Hz, 1H), 7.09 – 6.99 (m, 1H), 2.53 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):158.39, 149.14, 136.24, 123.25, 120.68, 24.48. EI-MS, *m/z* (%): 93 (100) [M^+].



4-Methoxypyridine.[10] The product was obtained as a yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):8.40 (d, *J* = 5.2 Hz, 2H), 6.79 (d, *J* = 6.2 Hz, 2H), 3.82 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):165.59, 151.02, 109.87, 55.06. EI-MS, *m/z* (%): 109 (100) [M^+].

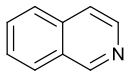


2-Hydroxypyridine.[11] The product was obtained as a brown solid. ^{1}H NMR (400 MHz, MeOD) δ (ppm):7.74–7.26 (m, 2H), 6.71–6.22 (m, 2H). ^{13}C NMR (100.6 MHz, MeOD) δ (ppm):165.84, 143.80, 136.05, 120.85, 108.59. EI-MS, *m/z* (%): 95 (100) [M^+].

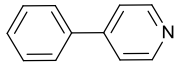


2-Chloropyridine.[12] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, MeOD) δ (ppm):8.48 (dd, *J* = 6.4, 1.4 Hz, 1H), 7.80 (dd, *J* = 8.1, 1.9 Hz, 1H),

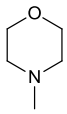
7.57 (td, *J* = 7.9, 1.5 Hz, 1H), 7.53–7.46 (m, 1H). ^{13}C NMR (100.6 MHz, MeOD) δ (ppm):142.13, 140.39, 130.95, 129.22, 126.32. EI-MS, *m/z* (%): 113 (61) [M^+].



Isoquinoline.[13] The product was obtained as a yellow liquid.^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):9.25 (s, 1H), 8.52 (d, *J* = 5.8 Hz, 1H), 7.96 (d, *J* = 8.2 Hz, 1H), 7.81 (d, *J* = 8.2 Hz, 1H), 7.73–7.52 (m, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):152.51, 142.96, 135.79, 130.37, 128.68, 127.64, 127.26, 126.47, 120.48. EI-MS, *m/z* (%): 129 (100) [M^+].



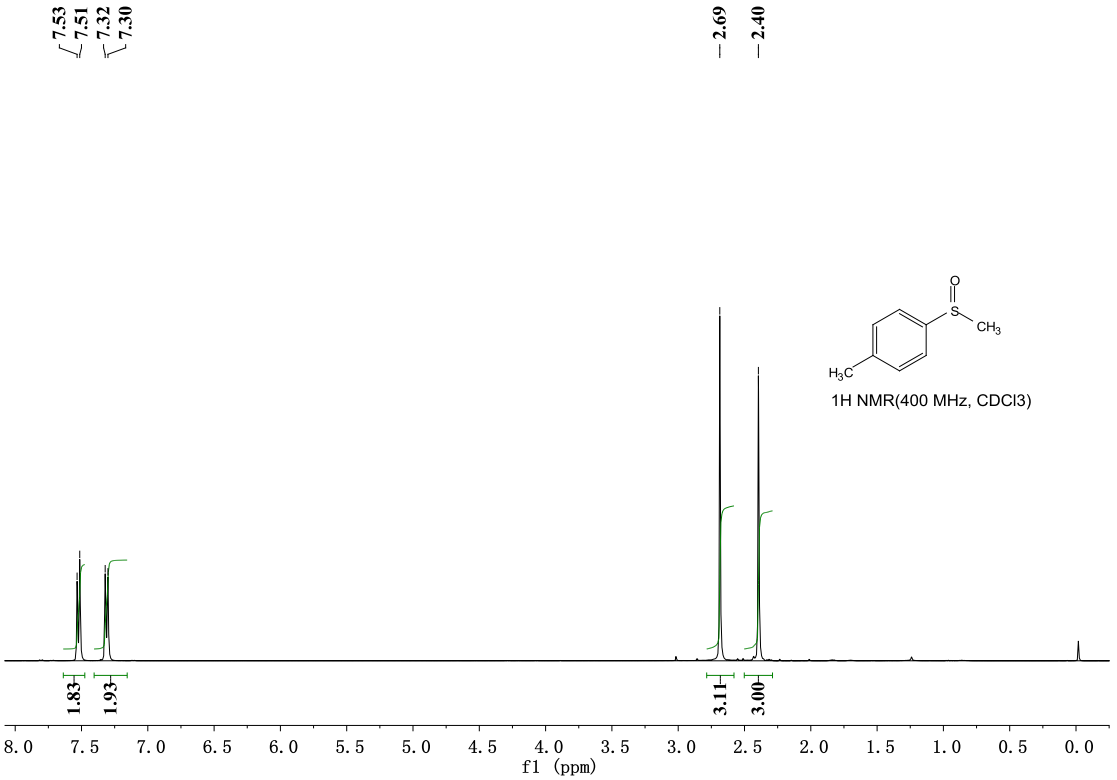
4-Phenylpyridine.[11] The product was obtained as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):8.67 (d, *J* = 5.3 Hz, 2H), 7.65 (d, *J* = 7.2 Hz, 2H), 7.59–7.40 (m, 5H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm):150.19, 148.47, 138.11, 129.13, 127.01, 121.67. EI-MS, *m/z* (%): 155 (100) [M^+].

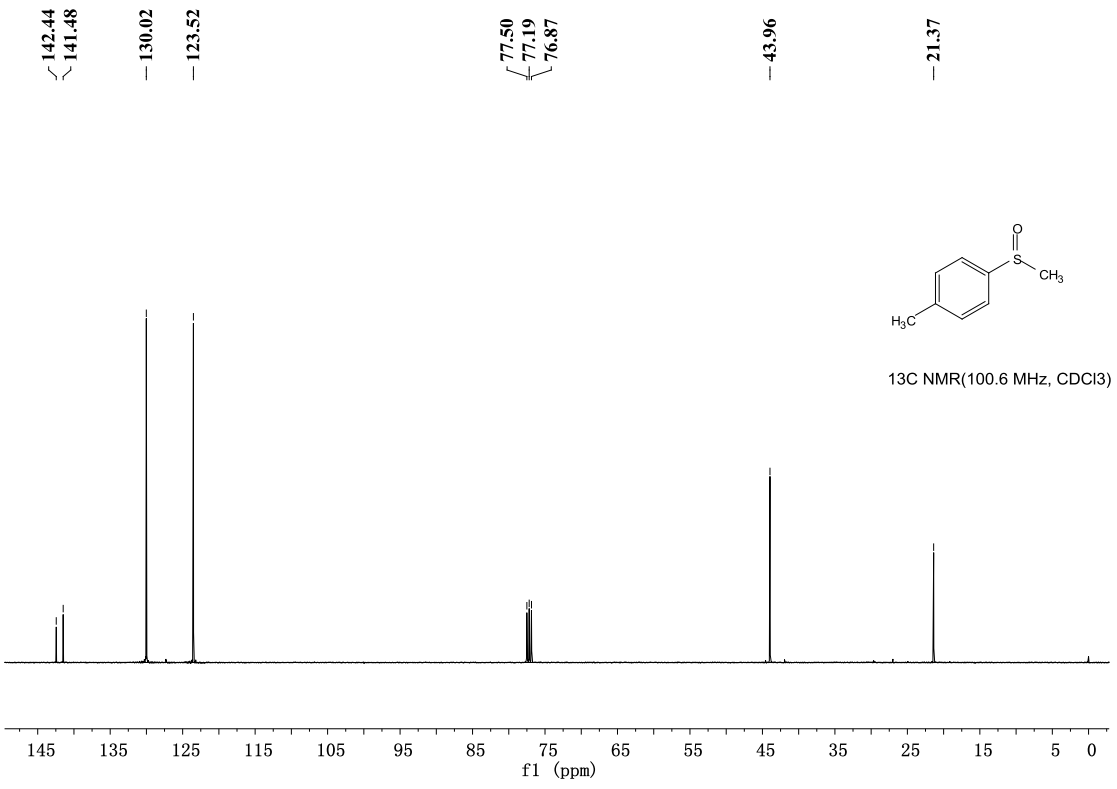


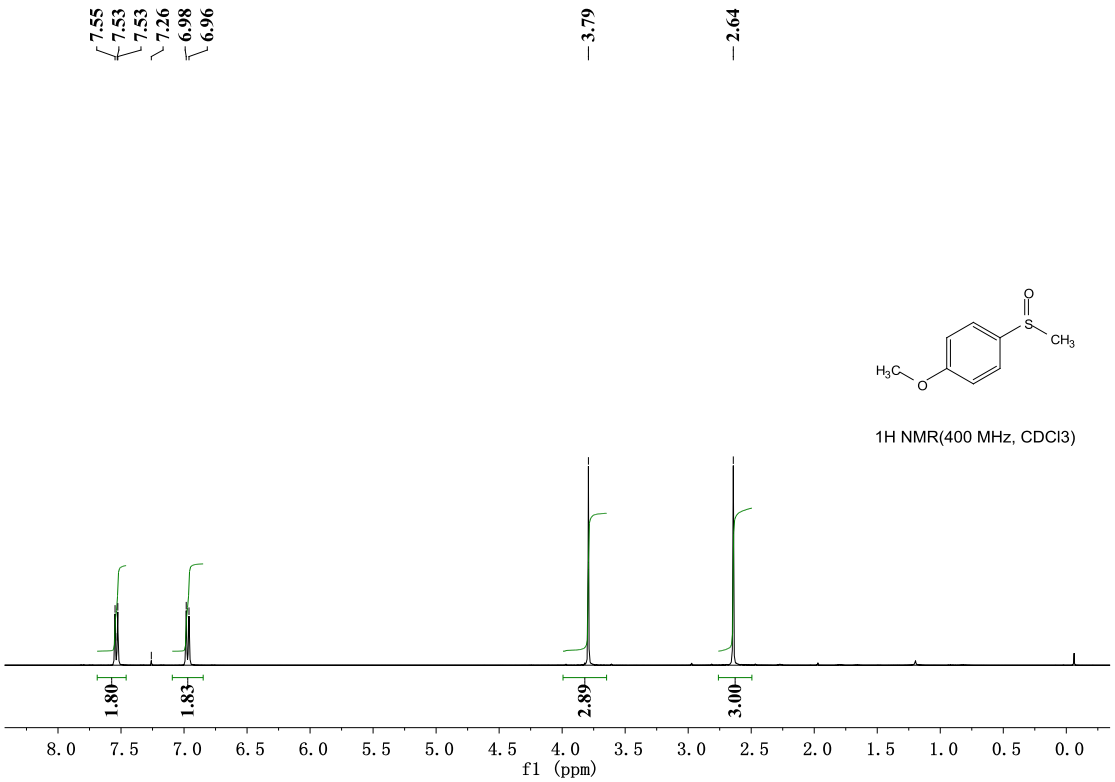
4-methyl morpholine.[14] The product was obtained as a pale yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm):3.90–3.59 (m, 4H), 2.39 (s, 4H), 2.28 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$) δ (ppm): 66.95, 55.46, 46.47. EI-MS, *m/z* (%): 101 (100) [M^+].

4. NMR and GC-MS spectra of the substrate and products

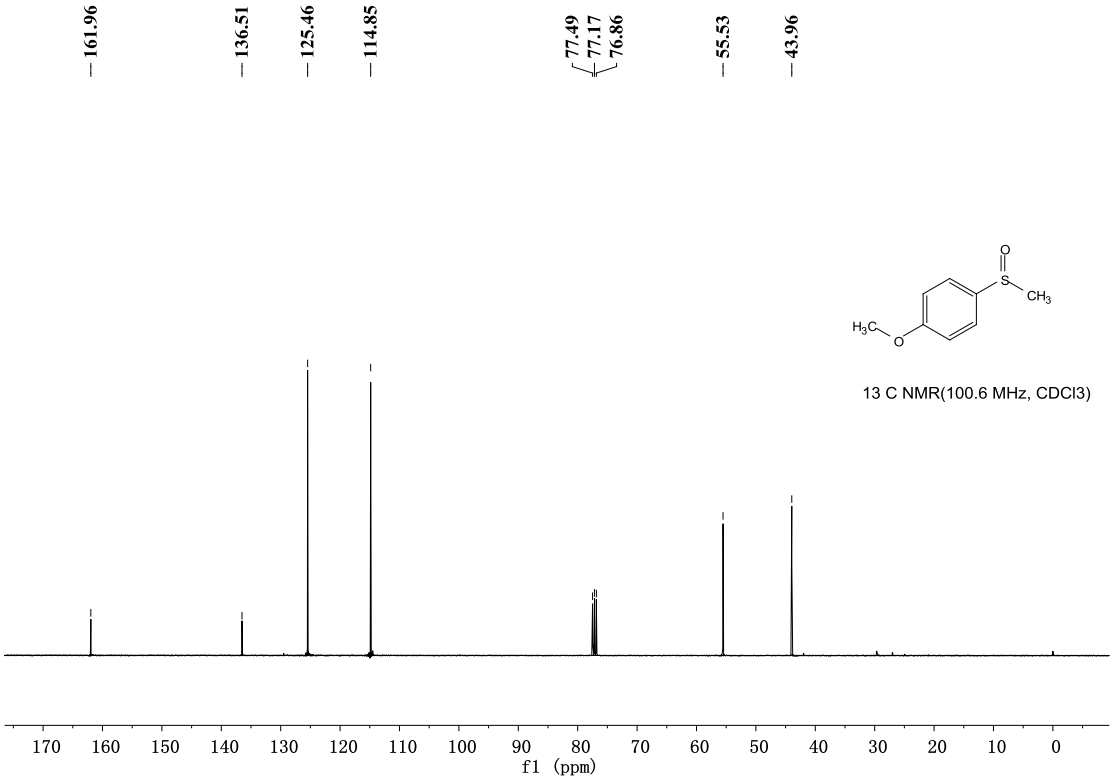
4.1 ^{1}H NMR and ^{13}C NMR spectra of the substrate and products



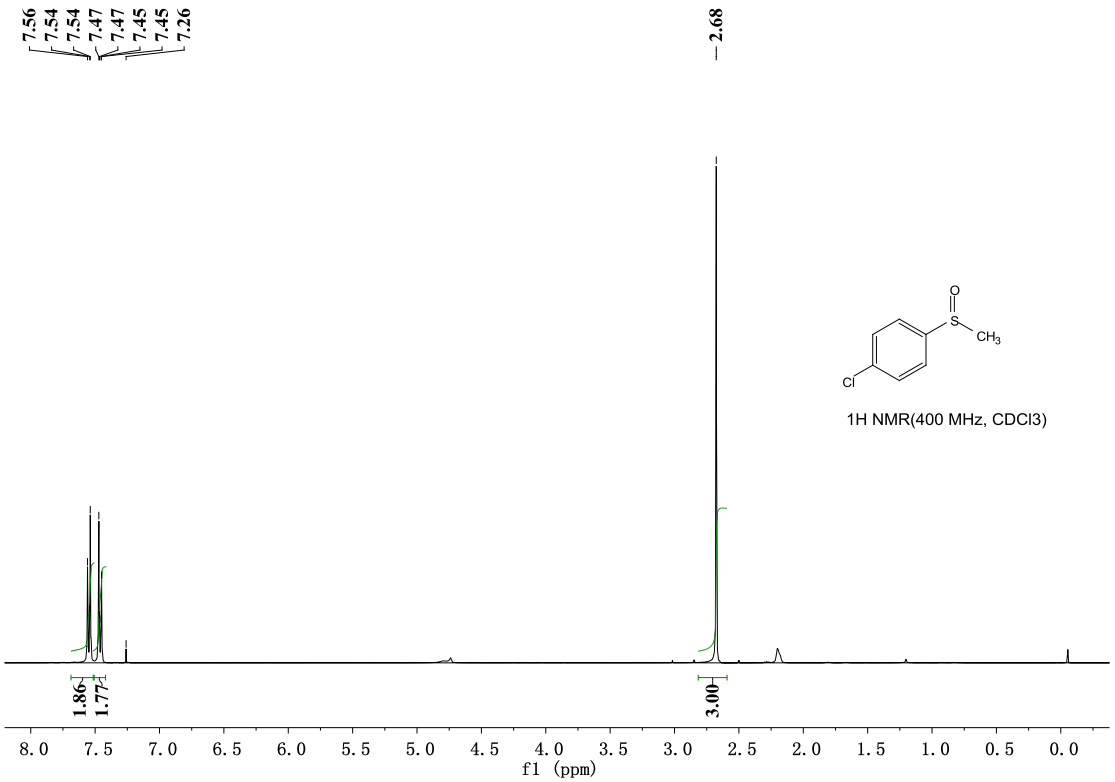




1H NMR(400 MHz, CDCl3)



13 C NMR(100.6 MHz, CDCl3)


7.56
7.54
7.54
7.47
7.47
7.45
7.45
7.26
2.68
O
S
CH3
Cl
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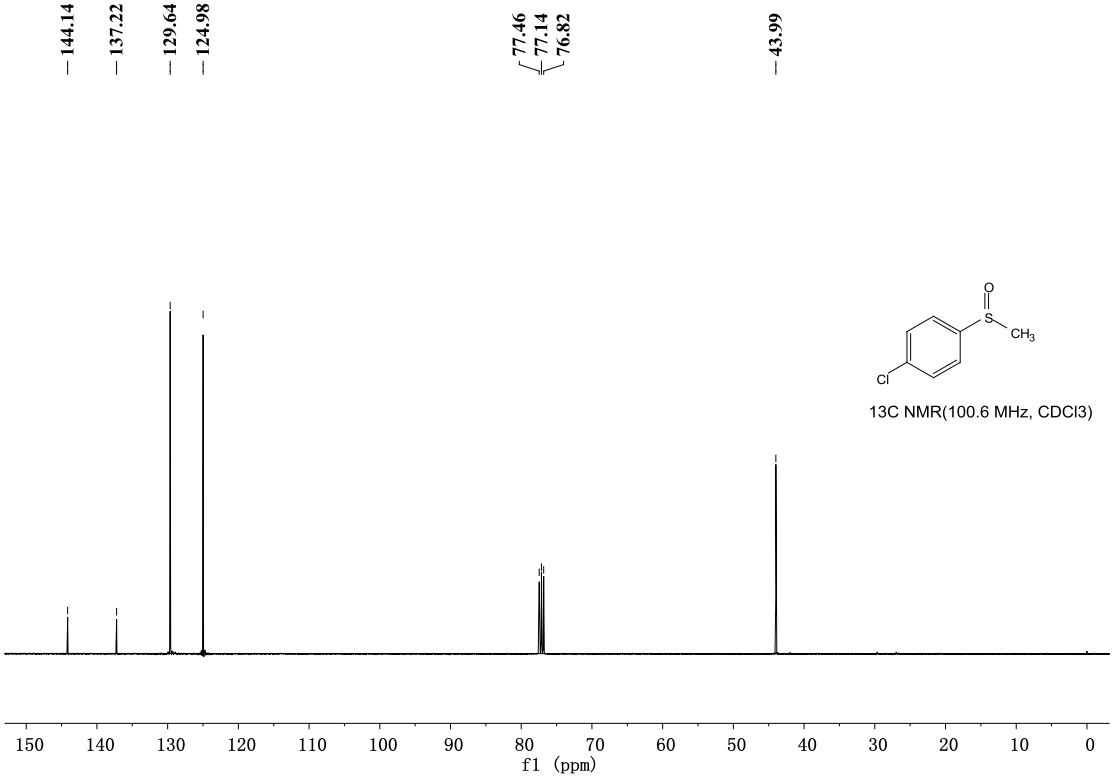
1.86
1.77
3.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


144.14
137.22
129.64
124.98
77.46
77.14
76.82
43.99
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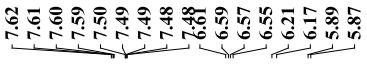
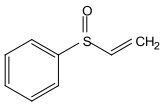
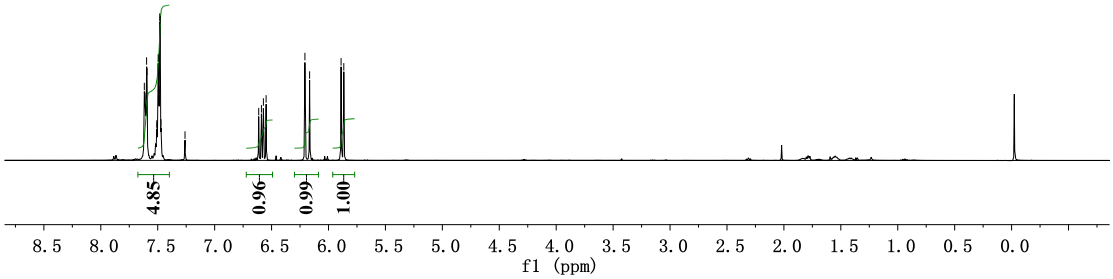
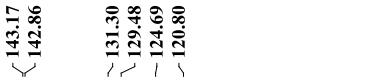
S
CH3
Cl
13C NMR(100.6 MHz, CDCl3)
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

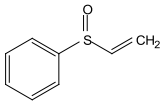




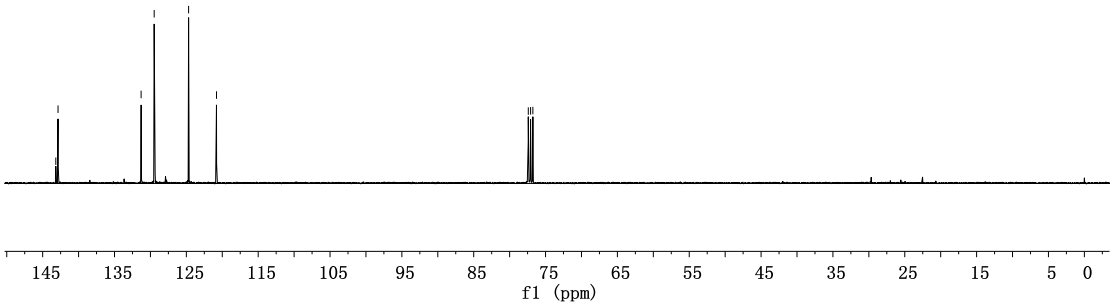
1H NMR(400 MHz, CDCl3)

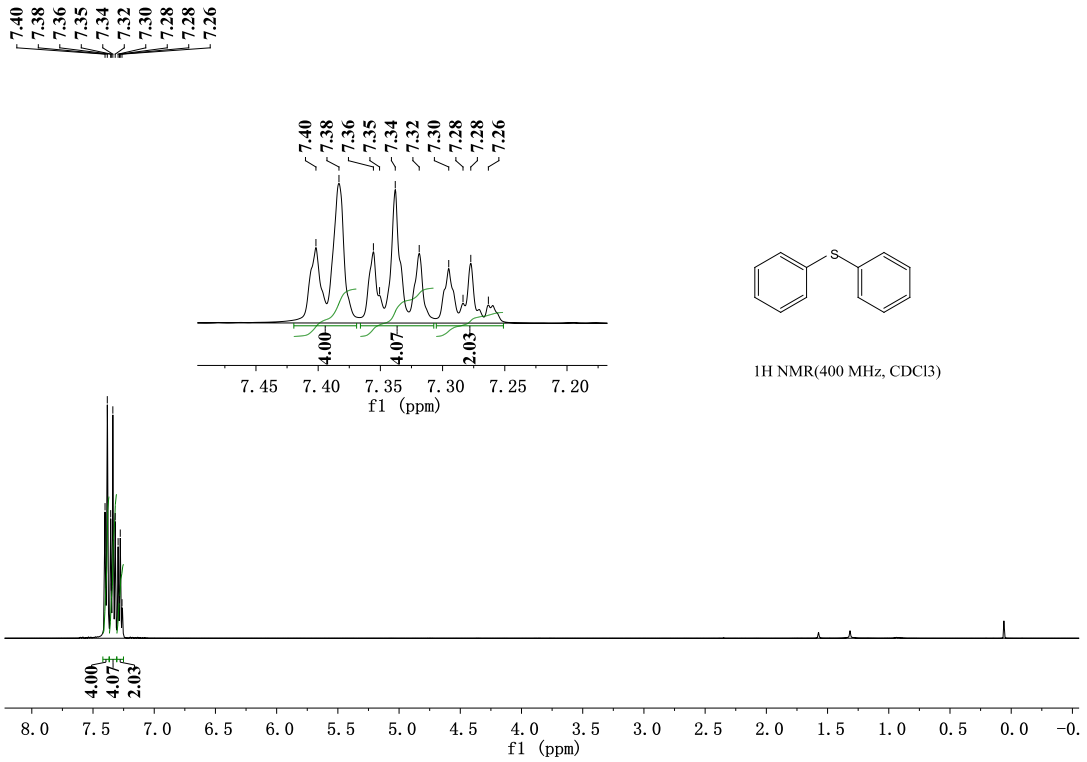


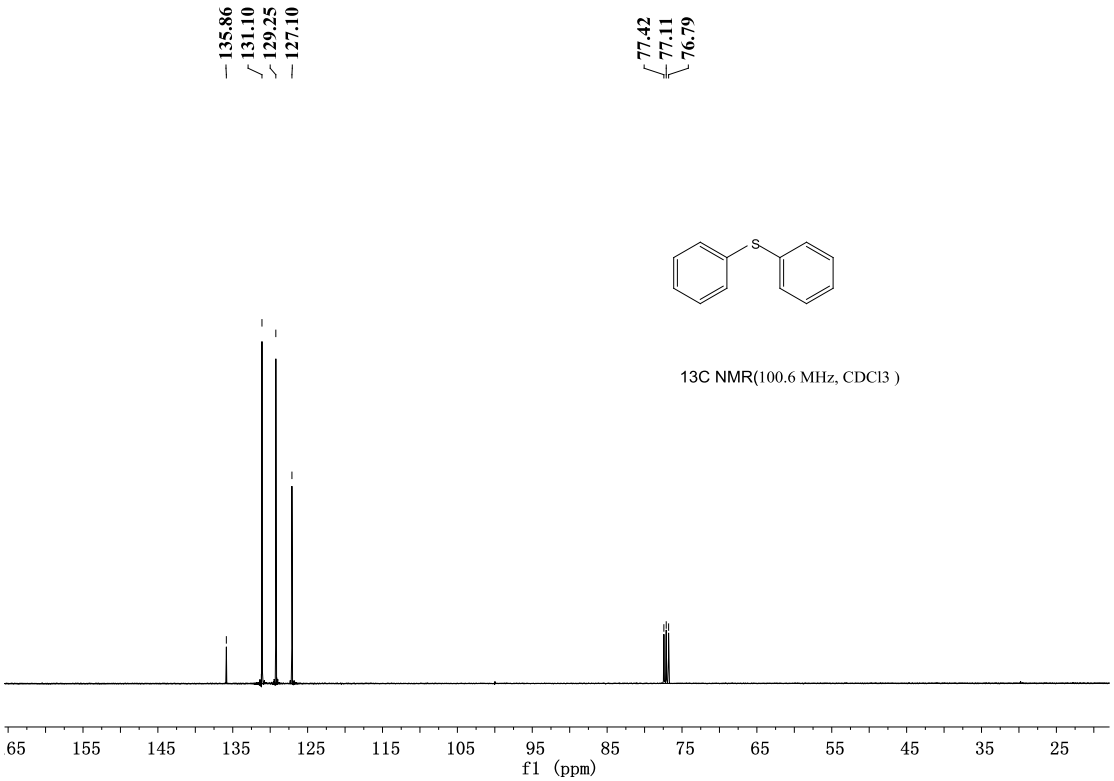


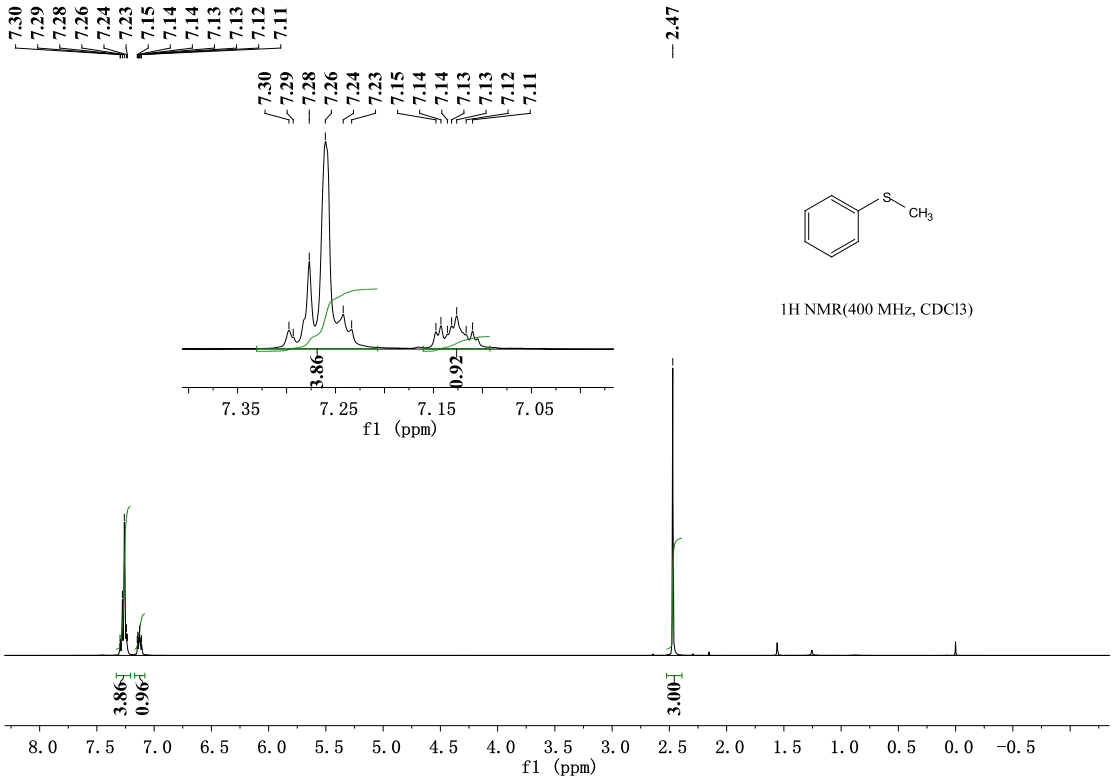


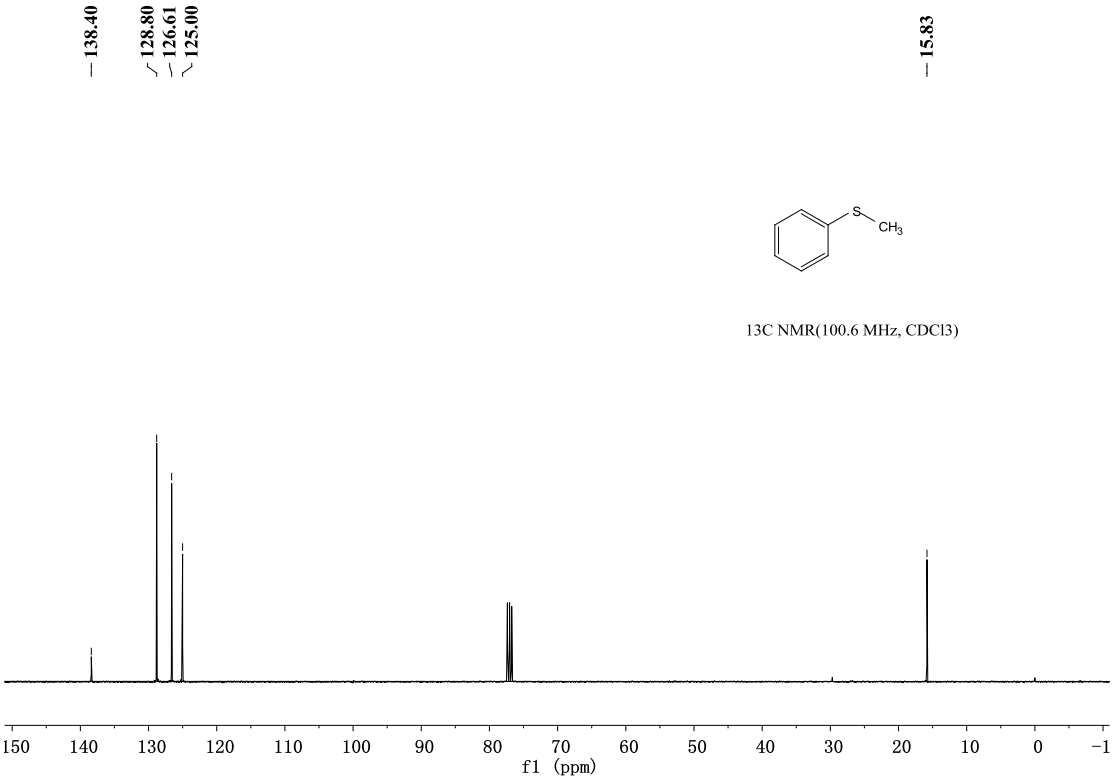
13C NMR(100.6 MHz, CDCl3)

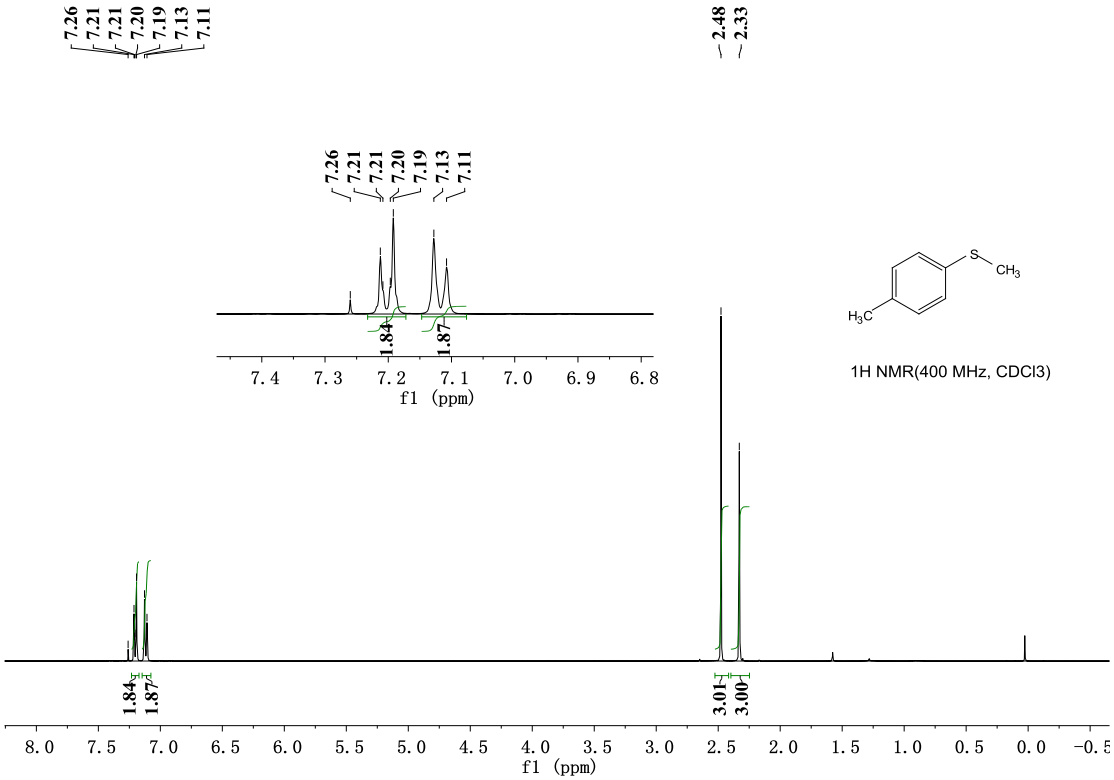


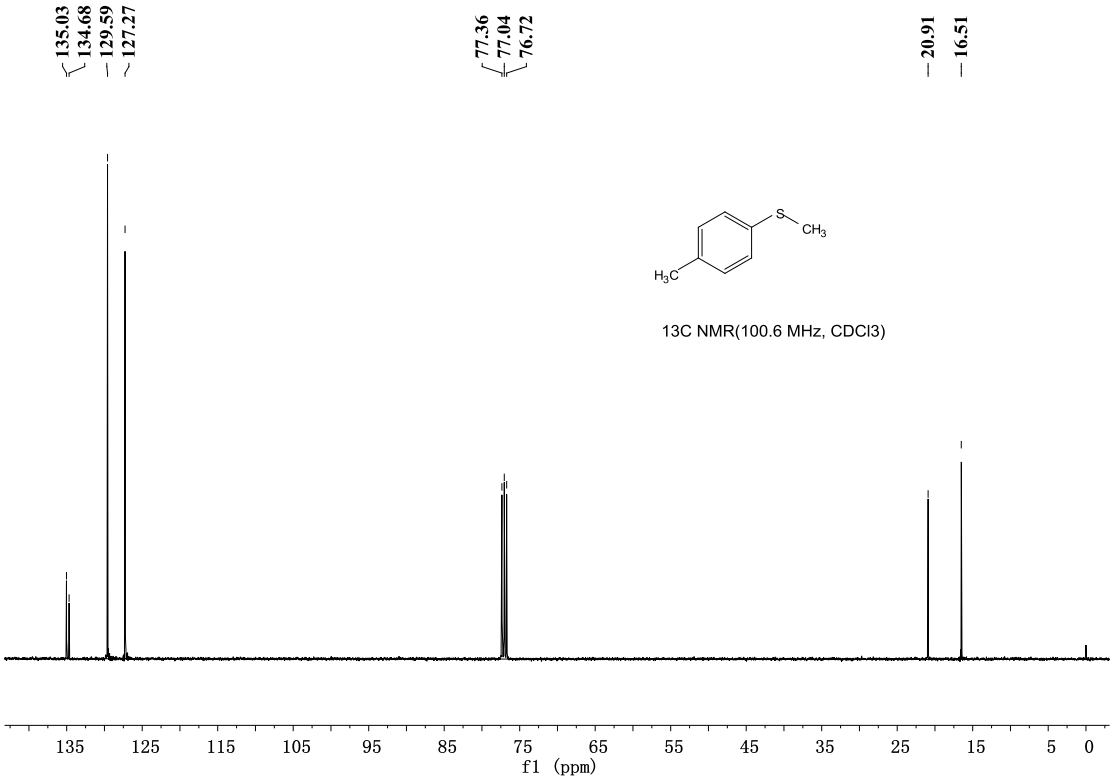

7.40
7.38
7.36
7.35
7.34
7.32
7.30
7.28
7.28
7.26
4.00
4.07
2.03
f1 (ppm)
S
1H NMR(400 MHz, CDCl3)

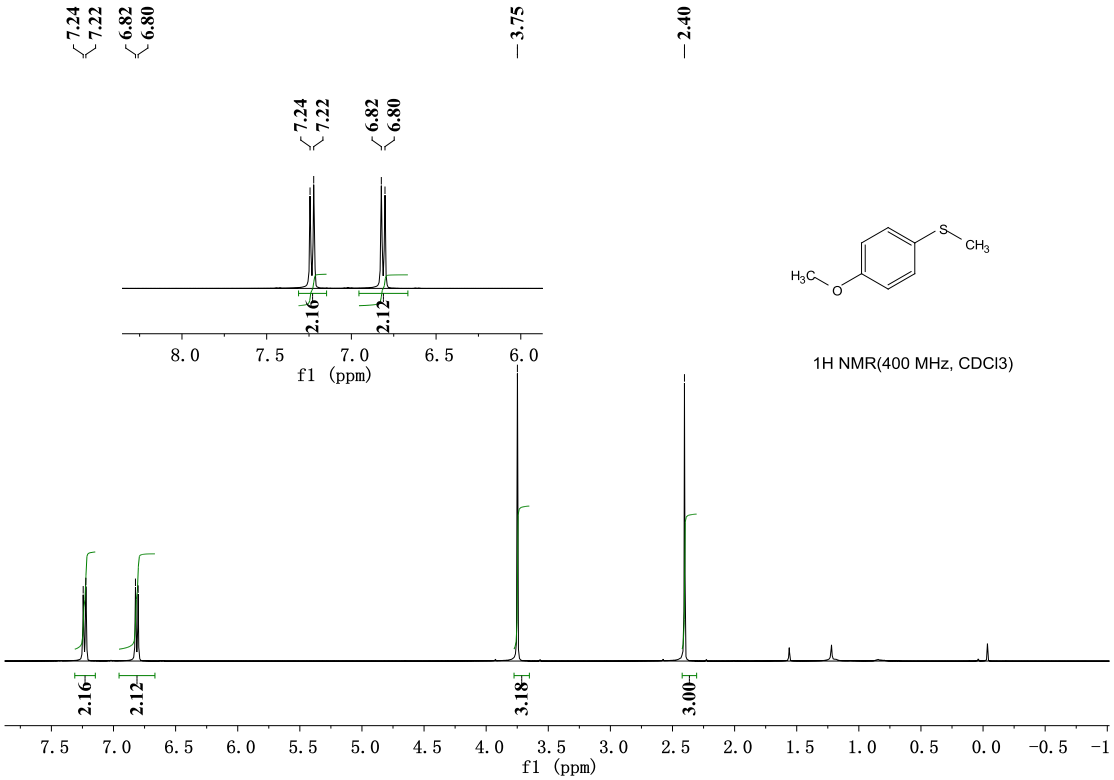

135.86
131.10
129.25
127.10
77.42
77.11
76.79
S
13C NMR(100.6 MHz, CDCl3)
f1 (ppm)

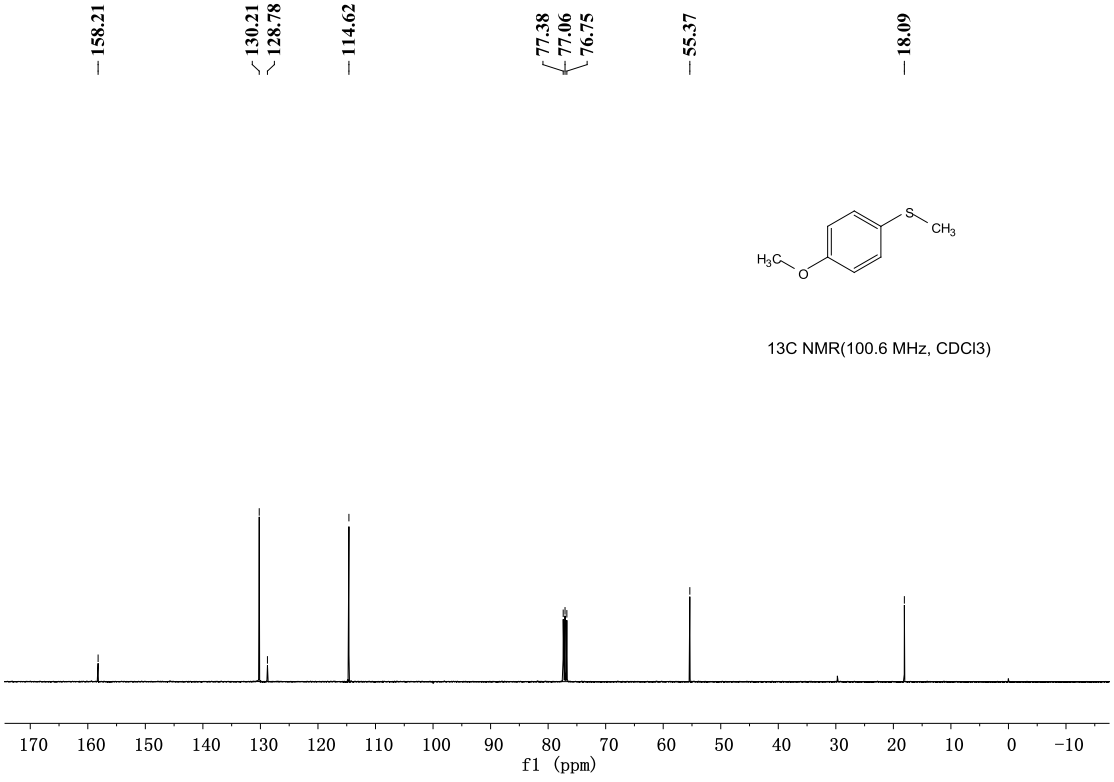


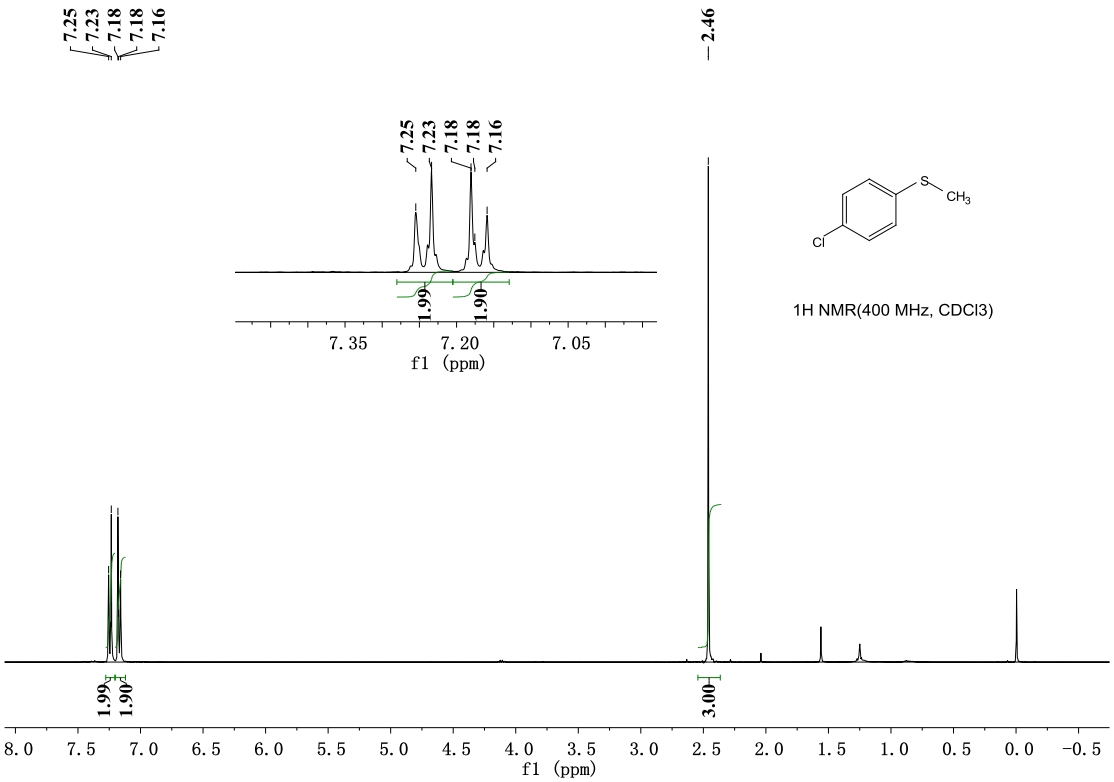



7.26
7.21
7.21
7.20
7.19
7.13
7.11
2.48
2.33
1.84
1.87
3.01
3.00
f1 (ppm)
1H NMR(400 MHz, CDCl3)

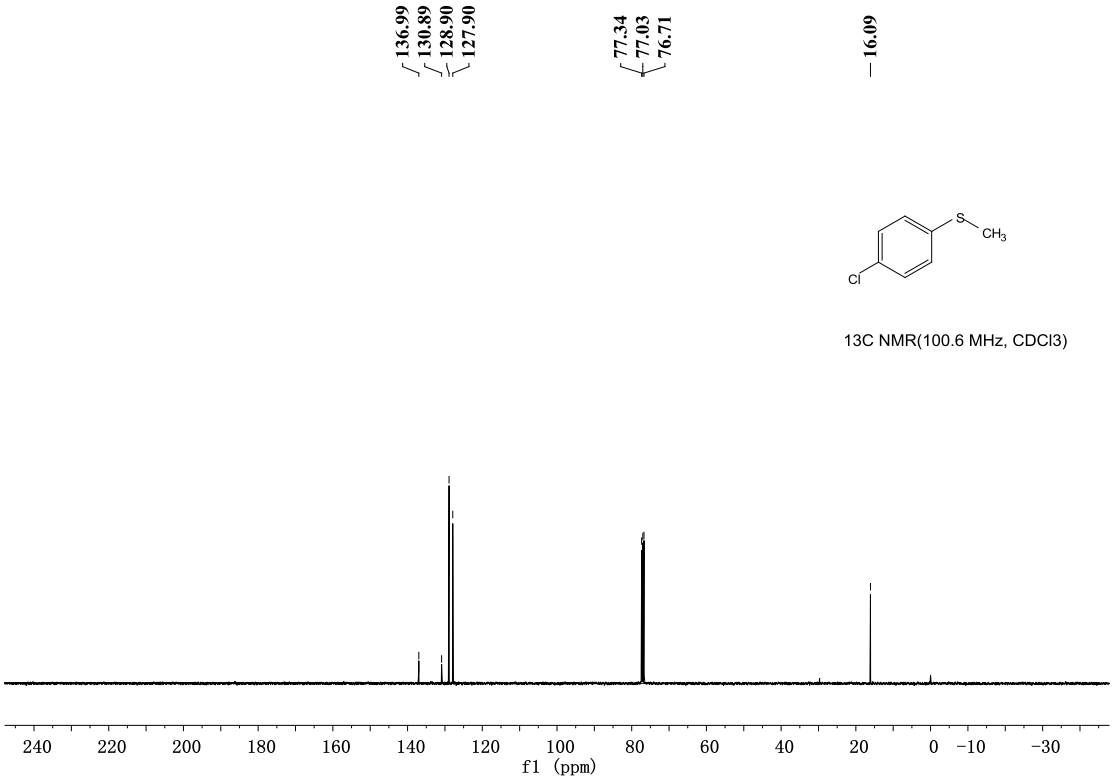

135.03
134.68
129.59
127.27
77.36
77.04
76.72
20.91
16.51
f1 (ppm)
13C NMR(100.6 MHz, CDCl3)


7.24
7.22
6.82
6.80
3.75
2.40
2.16
2.12
f1 (ppm)
S
CH3
H3C
O
1H NMR(400 MHz, CDCl3)
2.16
2.12
3.18
3.00
f1 (ppm)

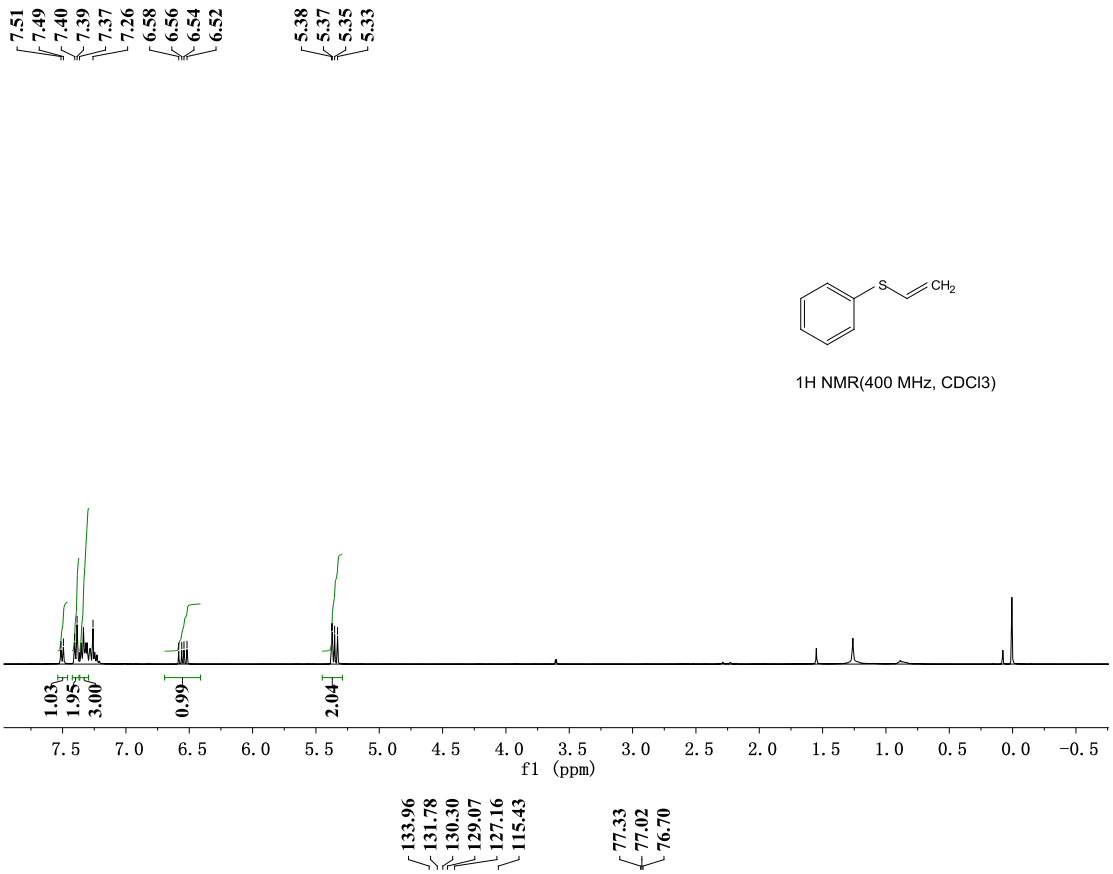

158.21
130.21
128.78
114.62
77.38
77.06
76.75
55.37
18.09
S
CH3
H3C
O
13C NMR(100.6 MHz, CDCl3)
f1 (ppm)

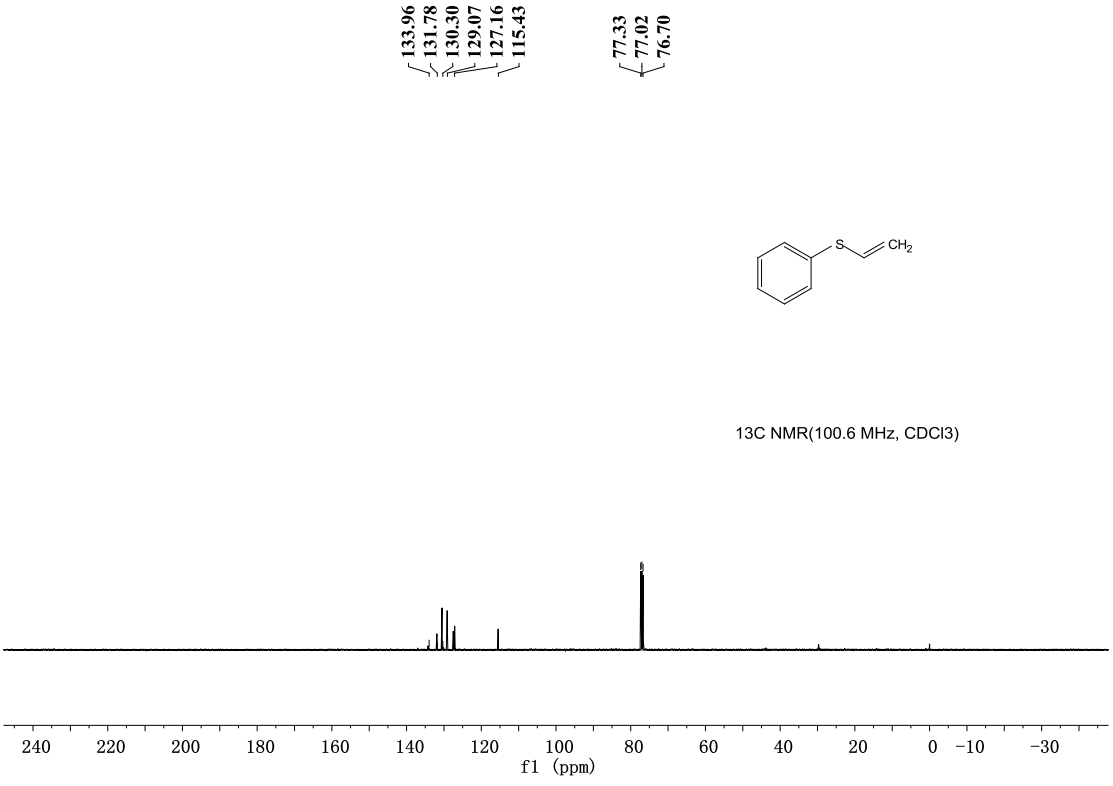


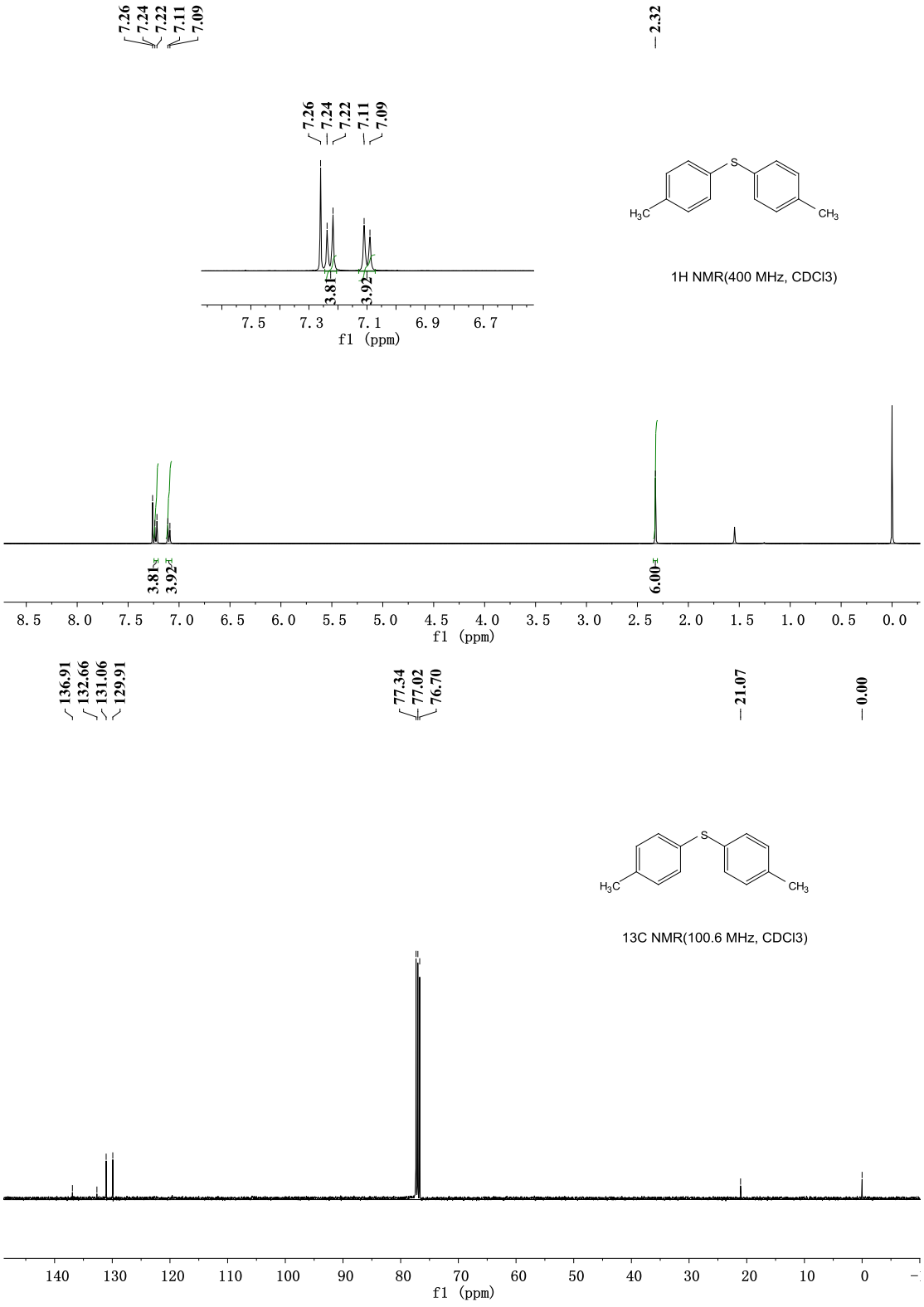
1H NMR(400 MHz, CDCl3)

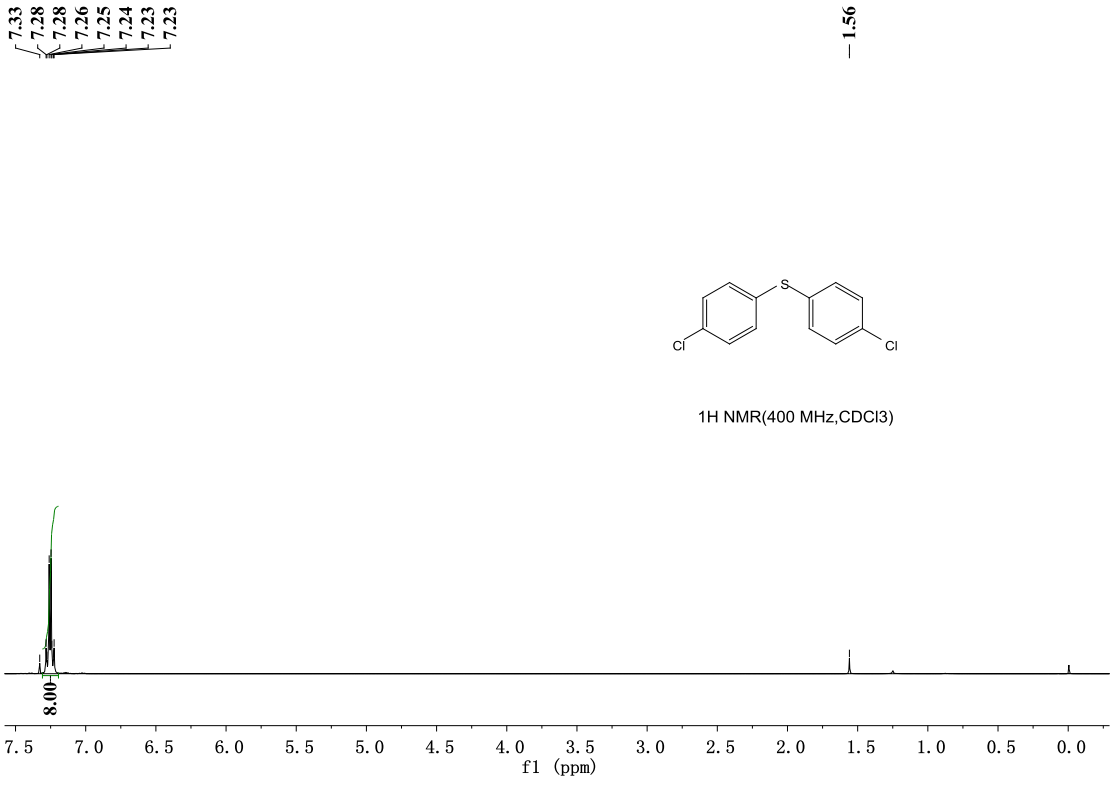


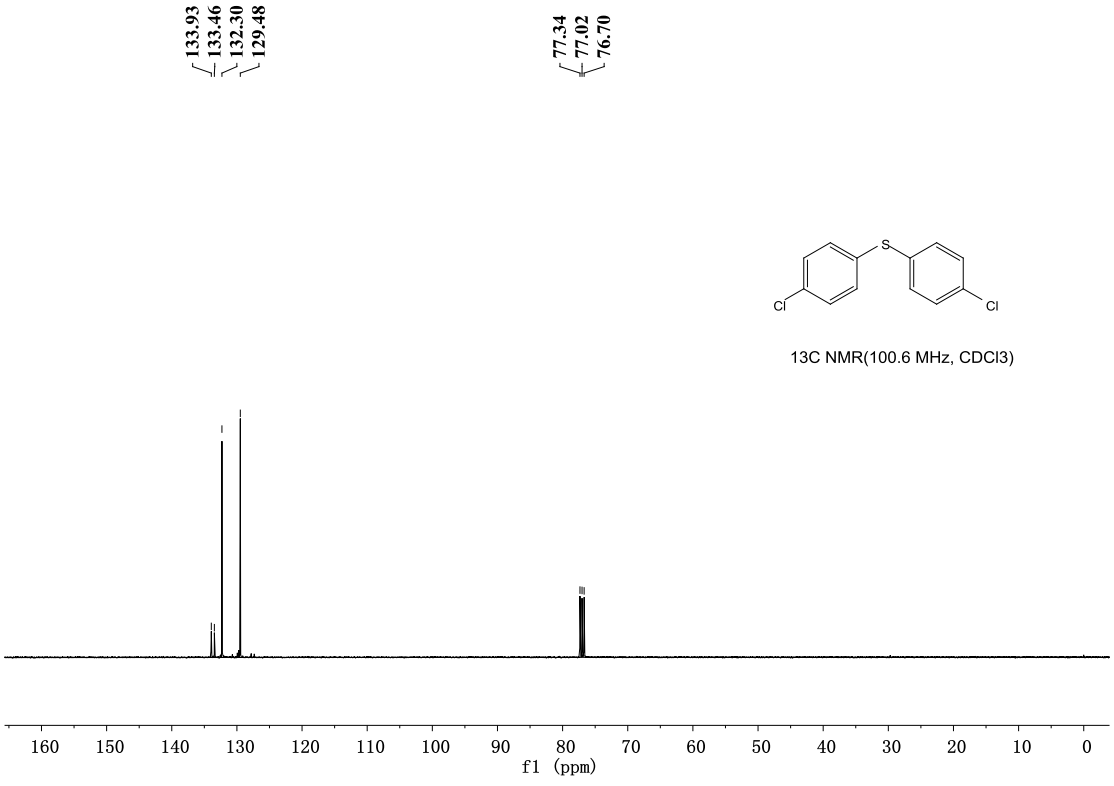
13C NMR(100.6 MHz, CDCl3)

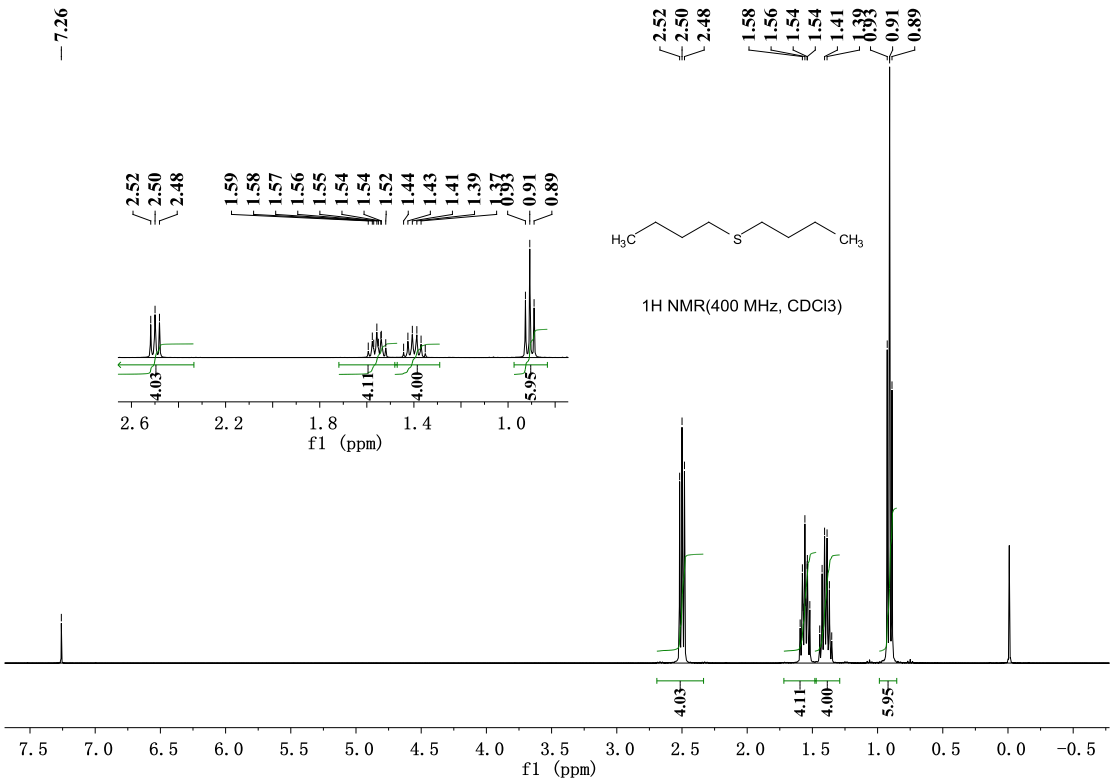

7.51
7.49
7.40
7.39
7.37
7.26
6.58
6.56
6.54
6.52
5.38
5.37
5.35
5.33
S
CH2
1H NMR(400 MHz, CDCl3)
1.03
1.95
3.00
0.99
2.04
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

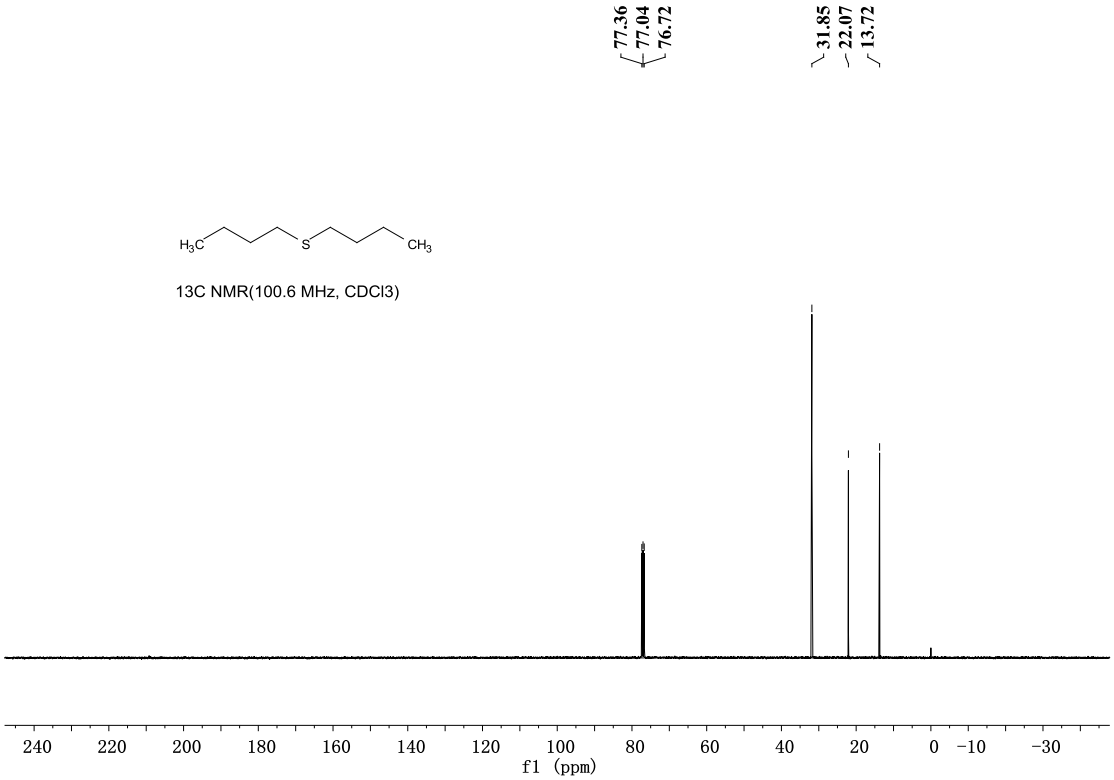

133.96
131.78
130.30
129.07
127.16
115.43
77.33
77.02
76.70
S
CH2
13C NMR(100.6 MHz, CDCl3)
240 220 200 180 160 140 120 100 80 60 40 20 0 −10 −30
f1 (ppm)

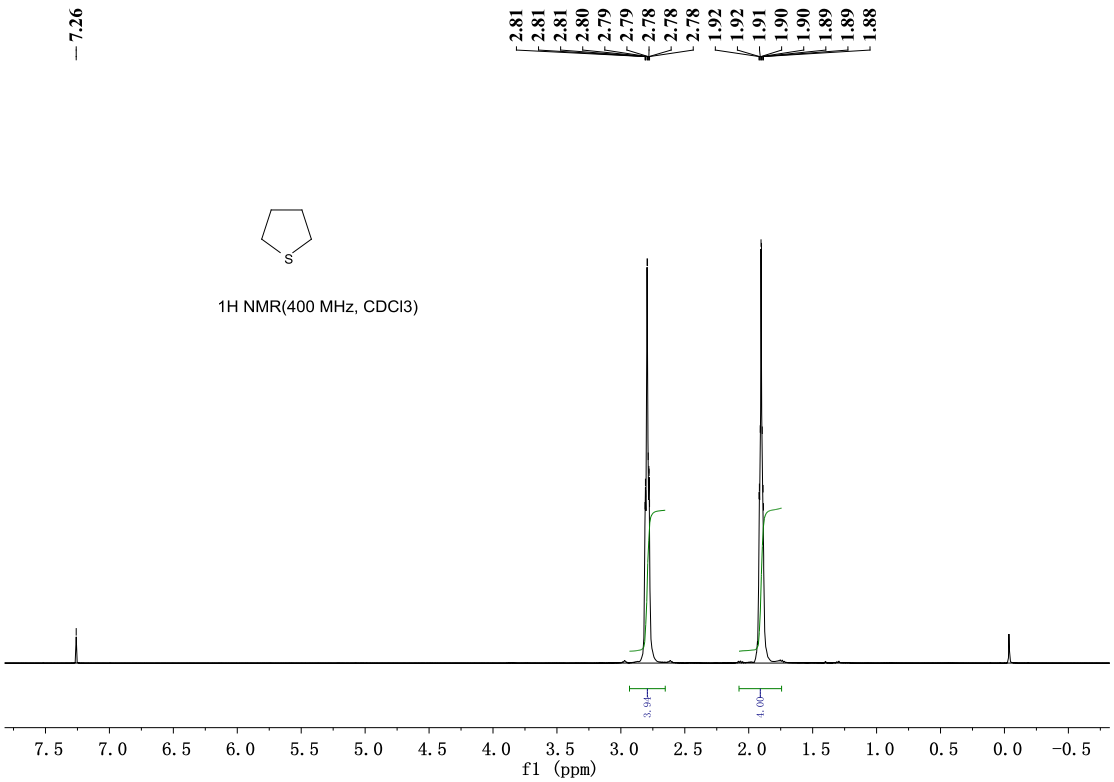

7.26
7.24
7.22
7.11
7.09
2.32
3.81
3.92
6.00
f1 (ppm)
1H NMR(400 MHz, CDCl3)
136.91
132.66
131.06
129.91
77.34
77.02
76.70
21.07
0.00
13C NMR(100.6 MHz, CDCl3)

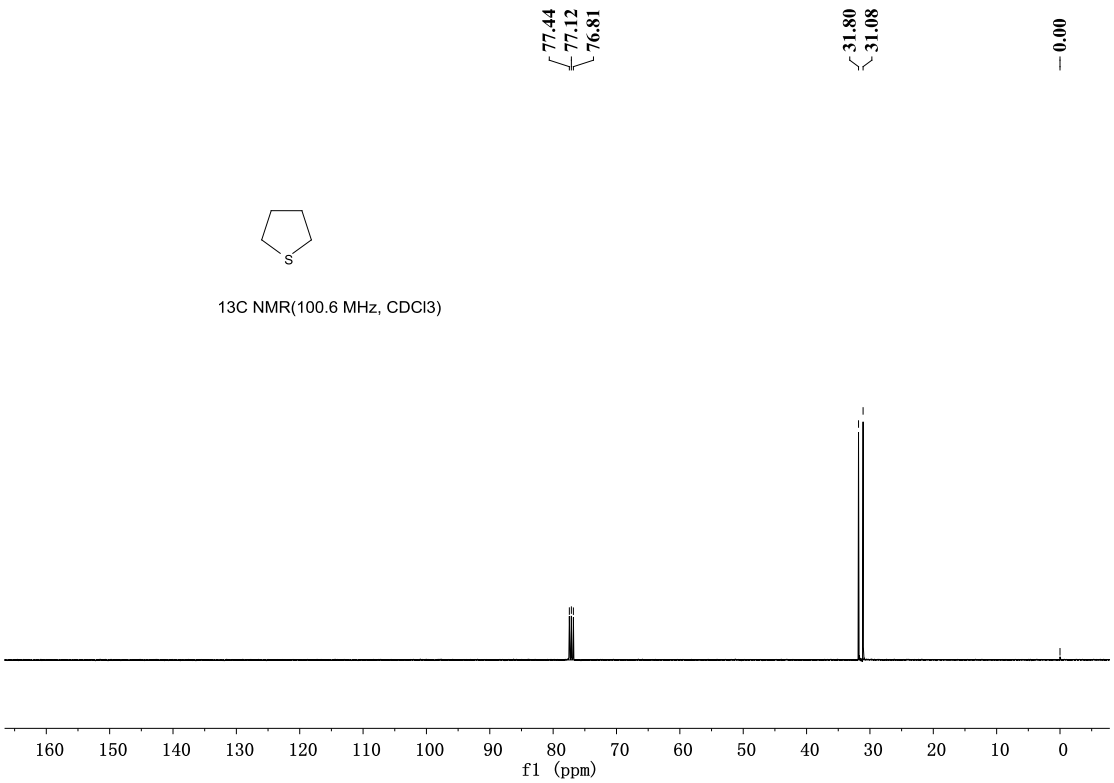

7.33
7.28
7.28
7.26
7.25
7.24
7.23
7.23
1.56
S
Cl
Cl
1H NMR(400 MHz,CDCl3)
8.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

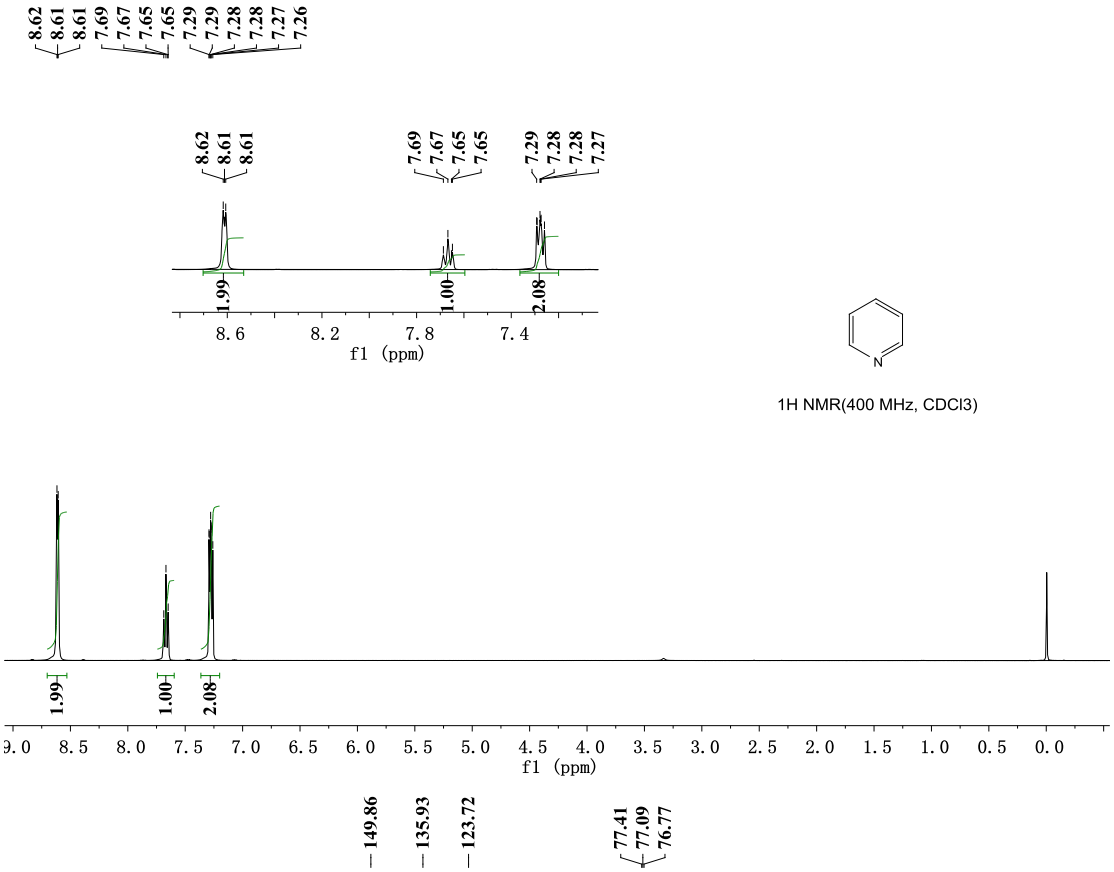

133.93
133.46
132.30
129.48
77.34
77.02
76.70
S
Cl
Cl
13C NMR(100.6 MHz, CDCl3)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

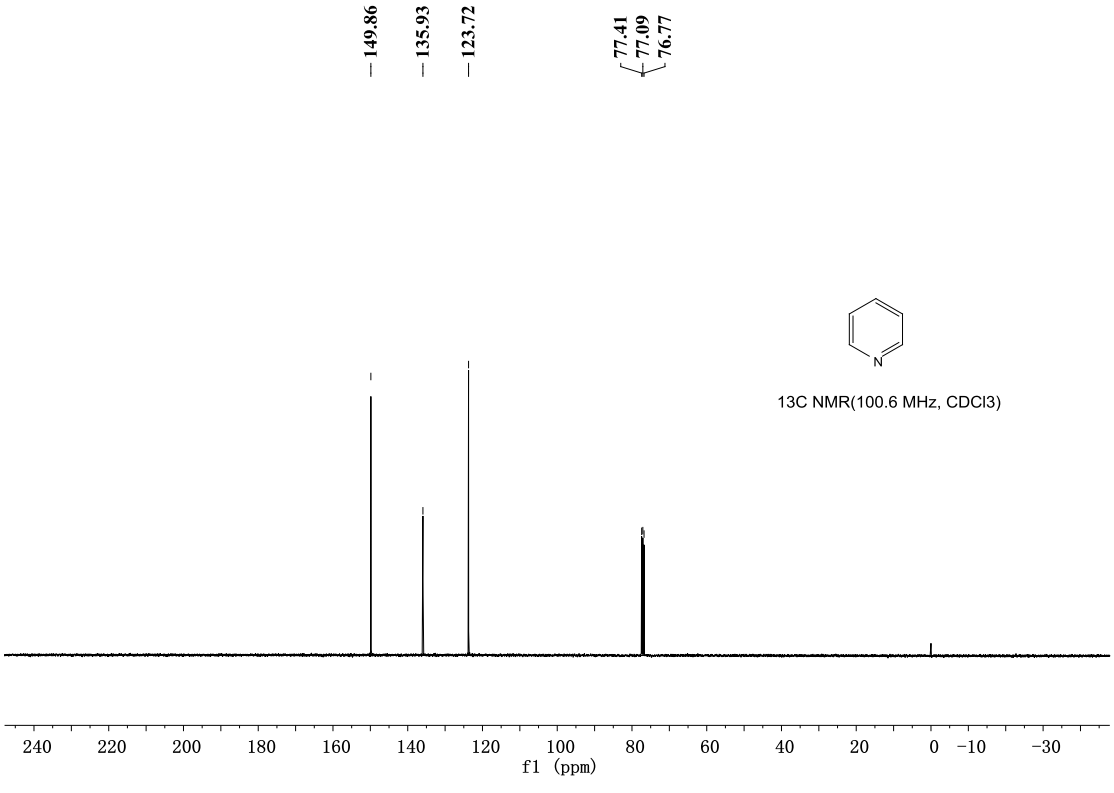

7.26
2.52
2.50
2.48
1.58
1.56
1.54
1.54
1.41
0.93
0.91
0.89
H3C
S
CH3
1H NMR(400 MHz, CDCl3)
4.03
4.11
4.00
5.95
f1 (ppm)

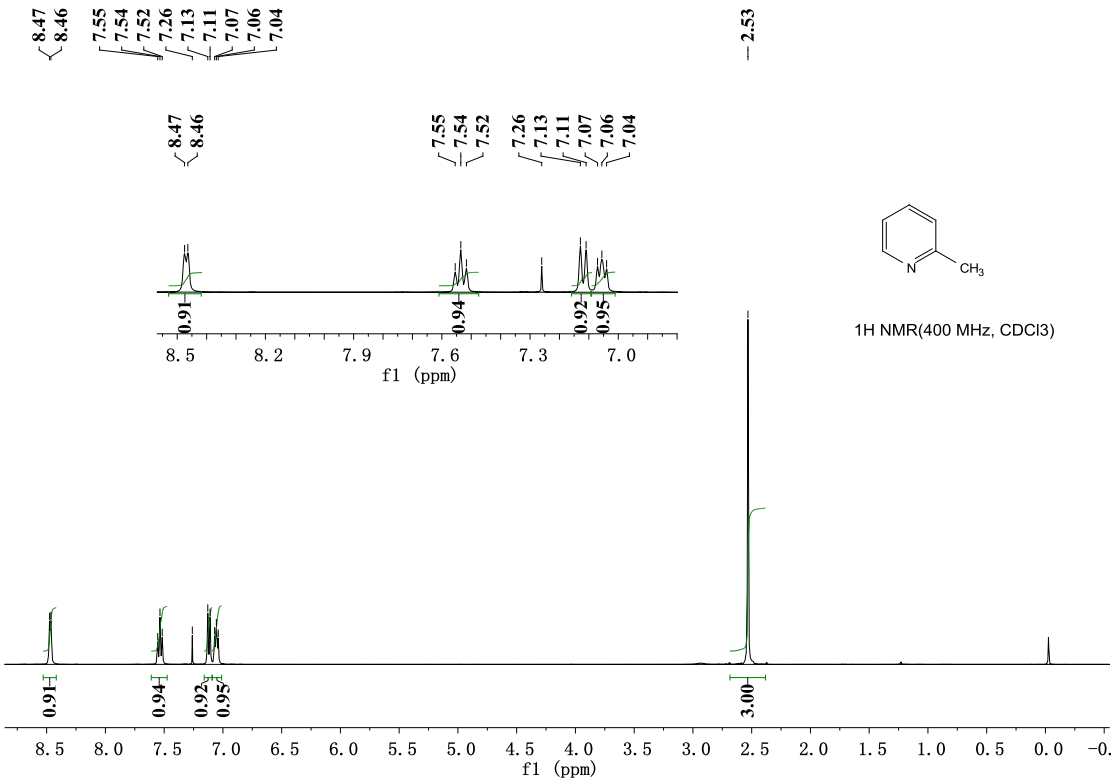

77.36
77.04
76.72
31.85
22.07
13.72
H3C
S
CH3
13C NMR(100.6 MHz, CDCl3)
f1 (ppm)


7.26
2.81
2.81
2.81
2.80
2.79
2.79
2.78
2.78
2.78
1.92
1.92
1.91
1.90
1.90
1.89
1.89
1.88
S
1H NMR(400 MHz, CDCl3)
3.91
4.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)

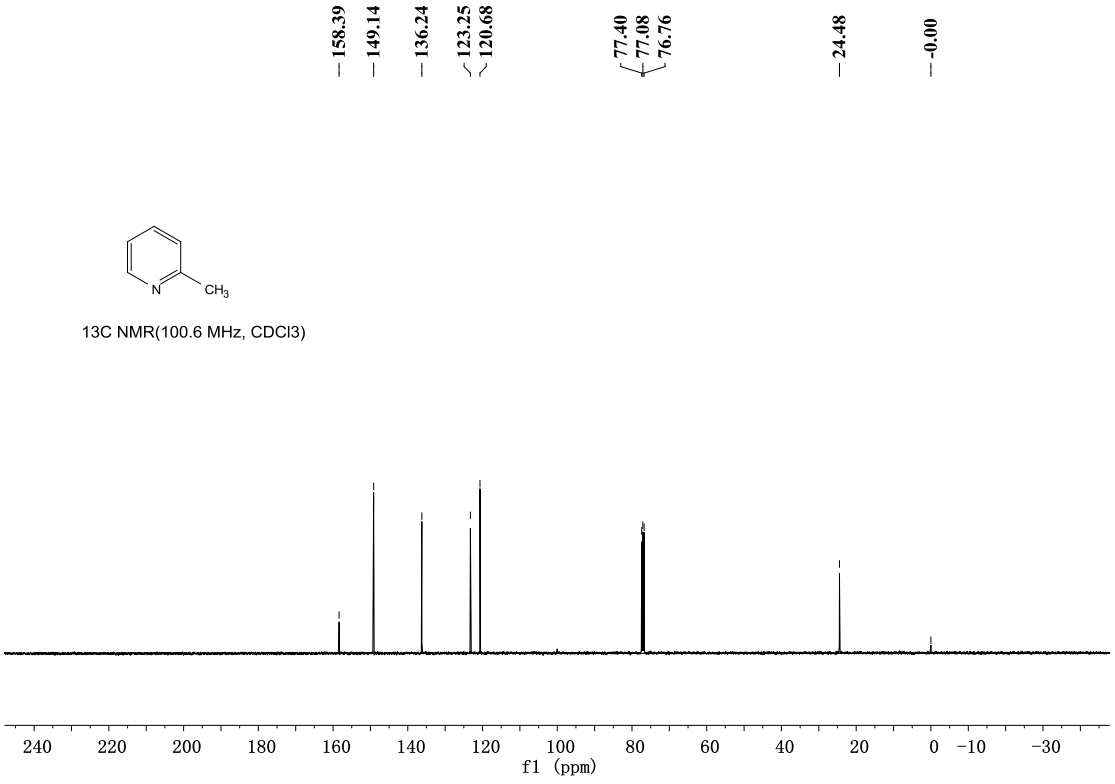

77.44
77.12
76.81
31.80
31.08
0.00
S
13C NMR(100.6 MHz, CDCl3)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.62
8.61
8.61
7.69
7.67
7.65
7.65
7.29
7.29
7.28
7.28
7.27
7.26
1.99
1.00
2.08
f1 (ppm)
1H NMR(400 MHz, CDCl3)

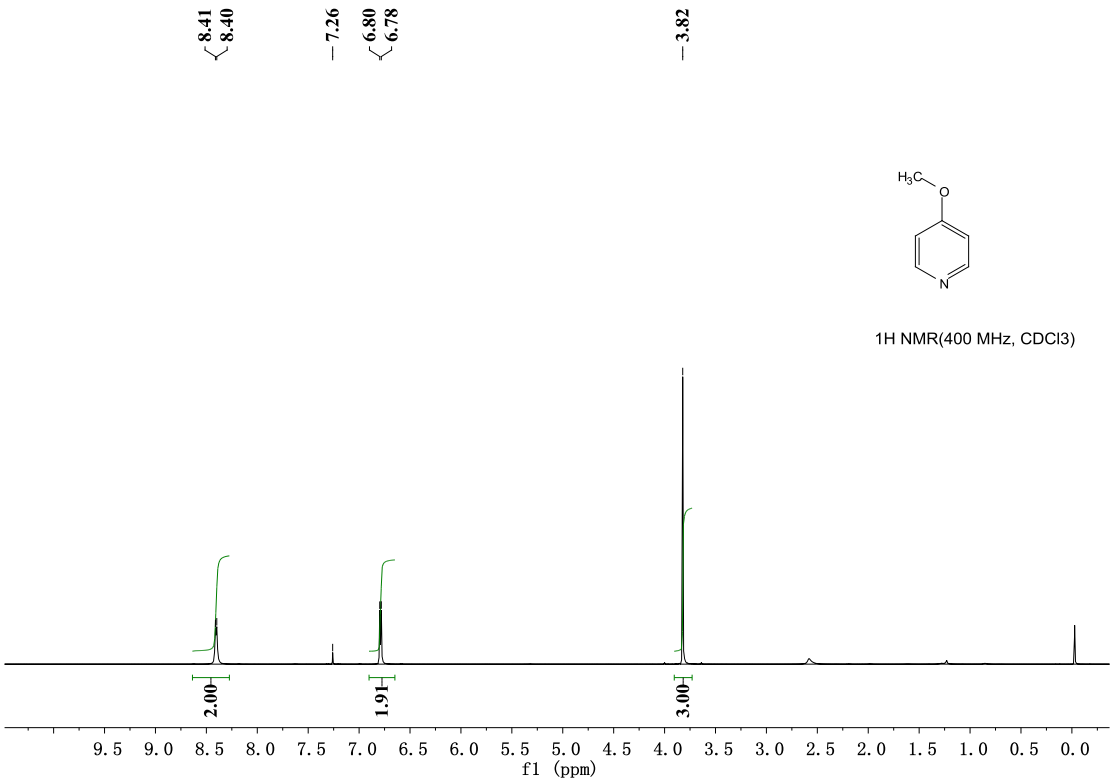

149.86
135.93
123.72
77.41
77.09
76.77
f1 (ppm)
13C NMR(100.6 MHz, CDCl3)

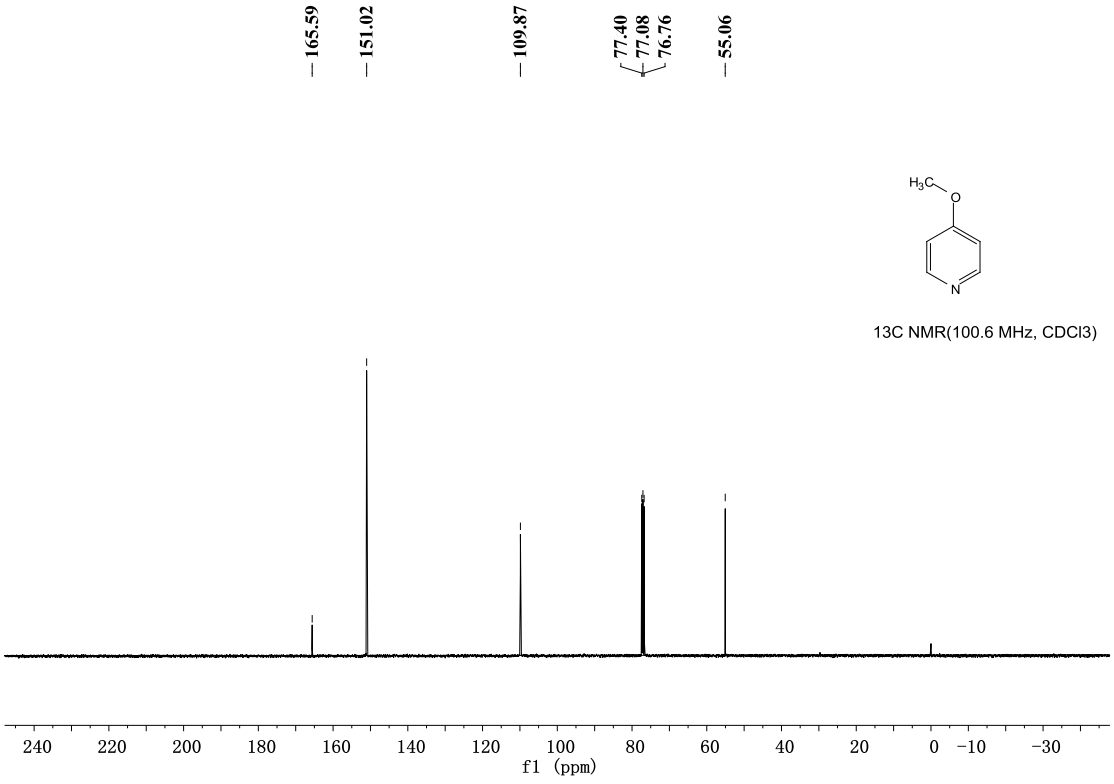


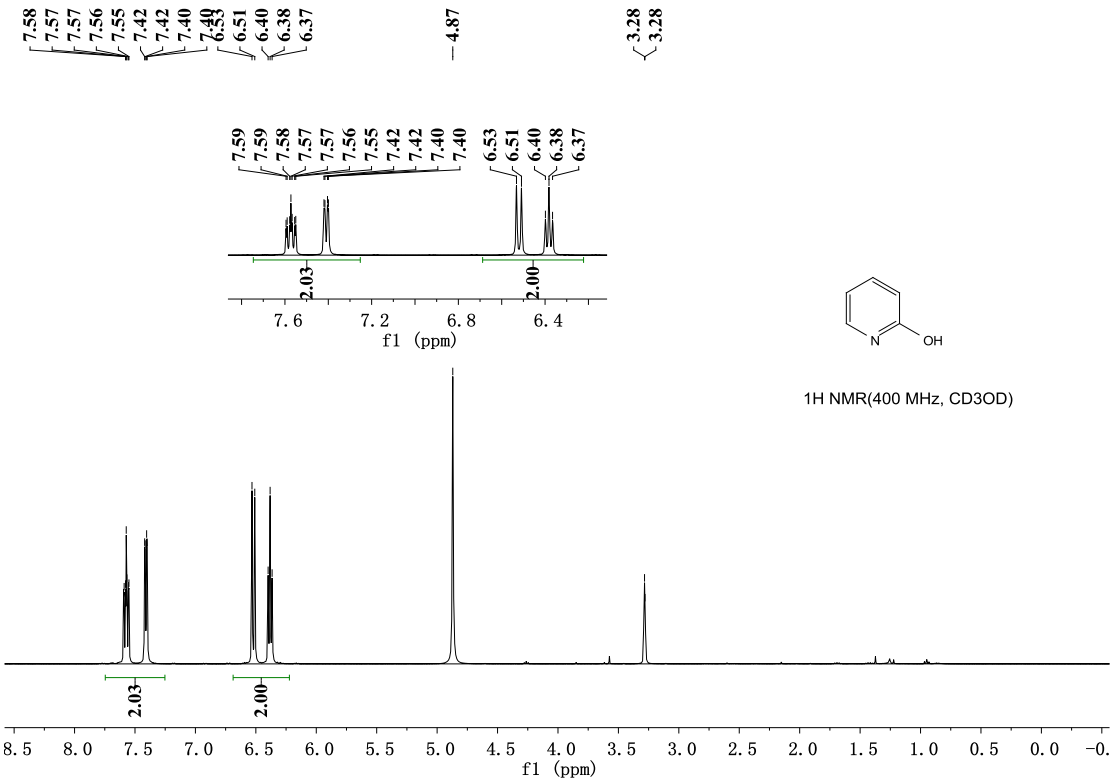
1H NMR(400 MHz, CDCl3)

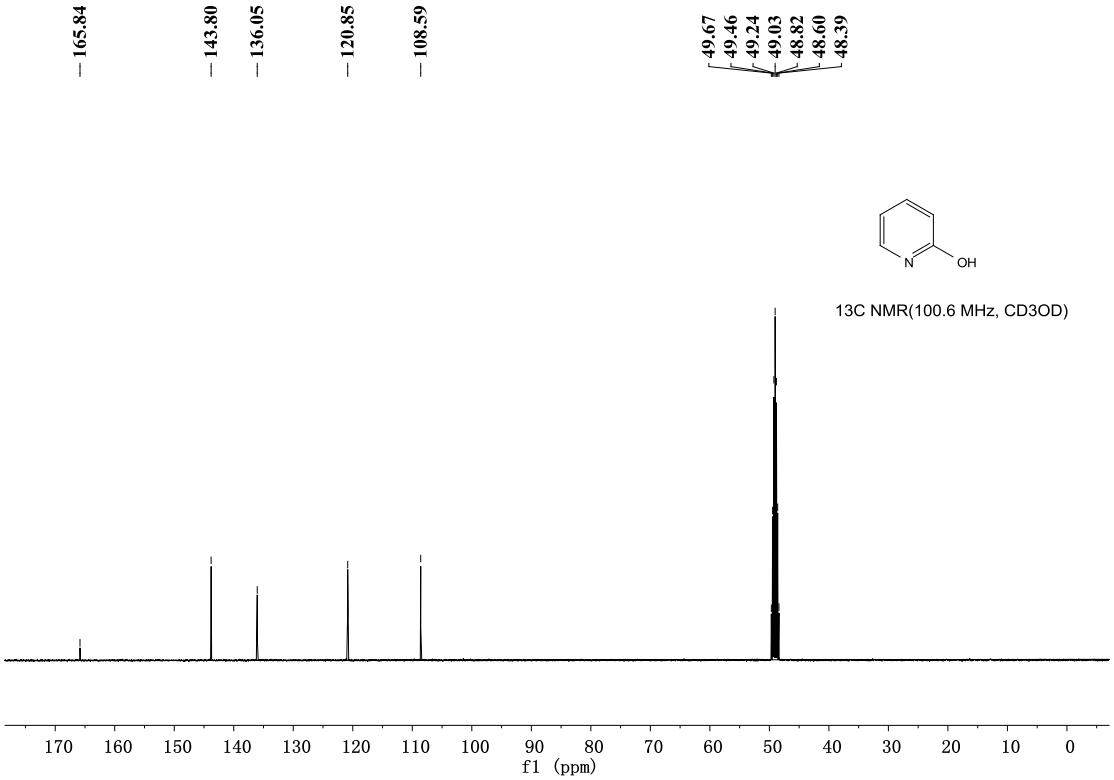


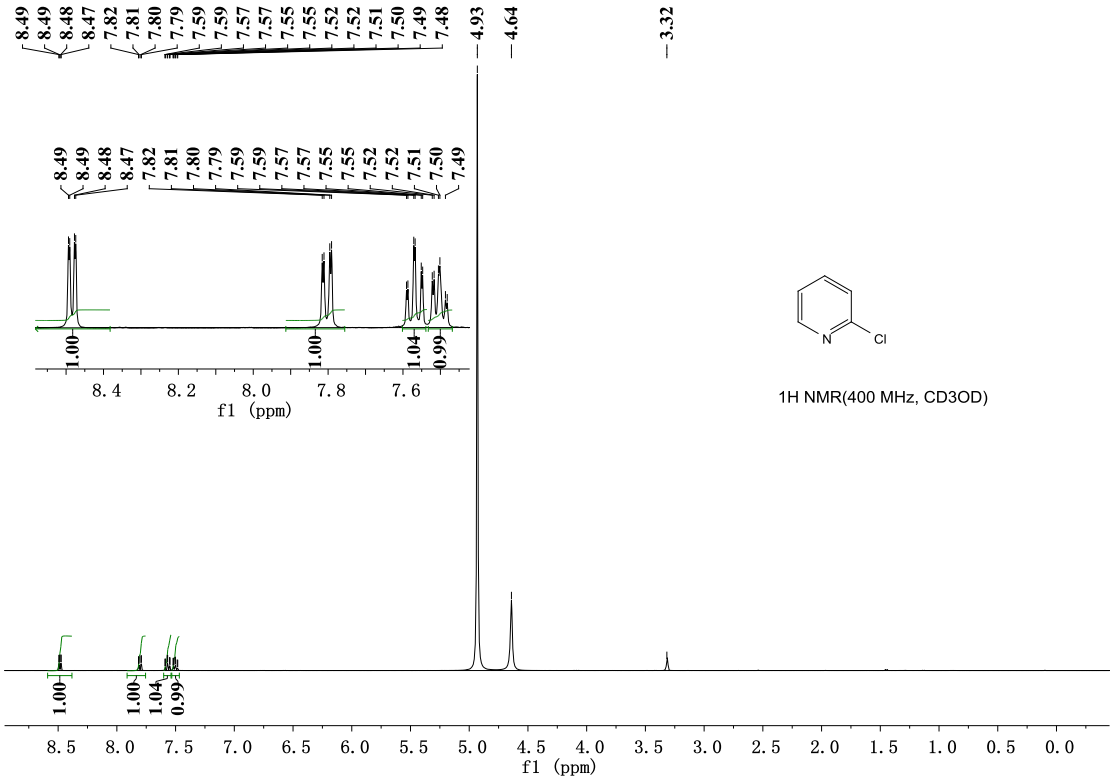
13C NMR(100.6 MHz, CDCl3)

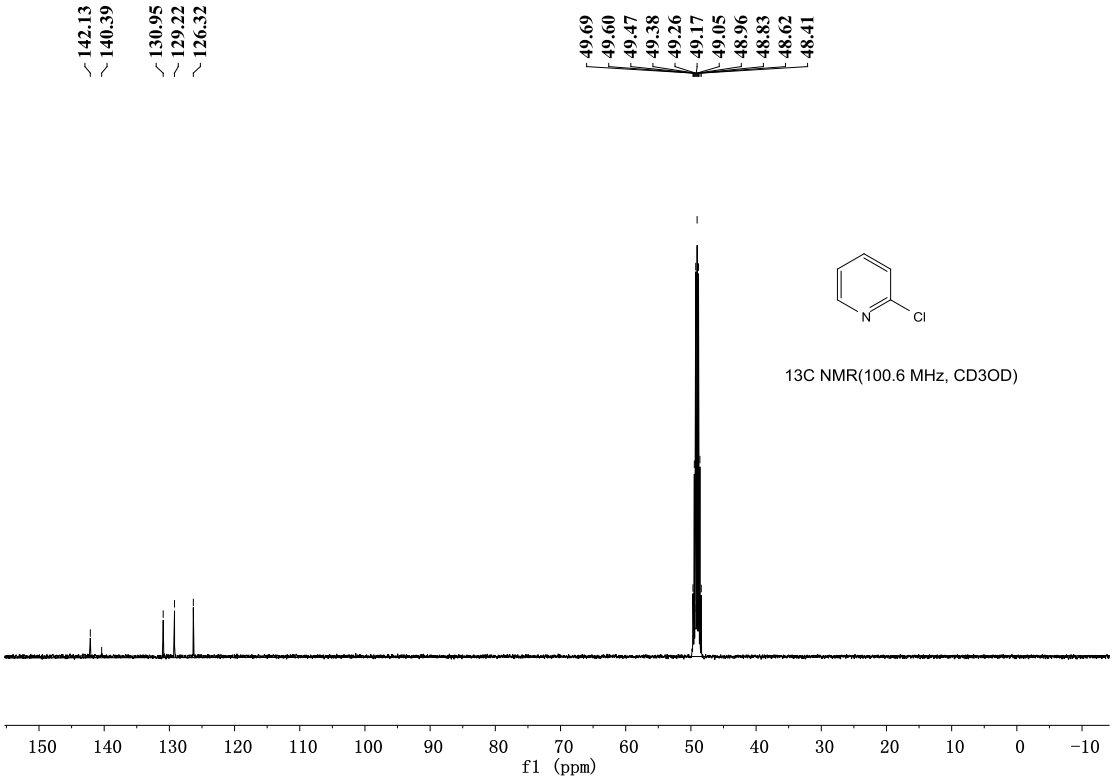

8.41
8.40
7.26
6.80
6.78
3.82
H3C
O
N
1H NMR(400 MHz, CDCl3)
2.00
1.91
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

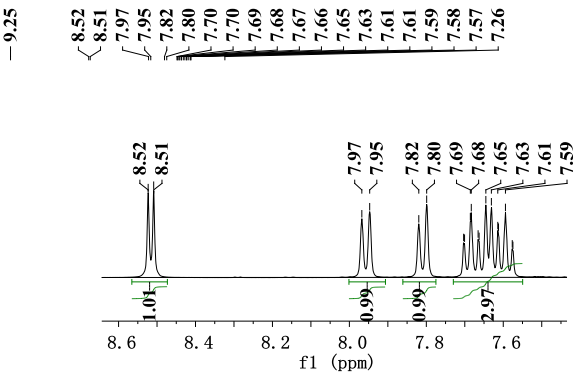

165.59
151.02
109.87
77.40
77.08
76.76
55.06
H3C
O
N
13C NMR(100.6 MHz, CDCl3)
240 220 200 180 160 140 120 100 80 60 40 20 0 −10 −30
f1 (ppm)

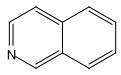

7.58
7.57
7.57
7.56
7.55
7.42
7.42
7.40
7.40
6.53
6.51
6.40
6.38
6.37
4.87
3.28
3.28
2.03
2.00
f1 (ppm)
N
OH
1H NMR(400 MHz, CD3OD)


165.84
143.80
136.05
120.85
108.59
49.67
49.46
49.24
49.03
48.82
48.60
48.39
N
OH
13C NMR(100.6 MHz, CD3OD)
f1 (ppm)

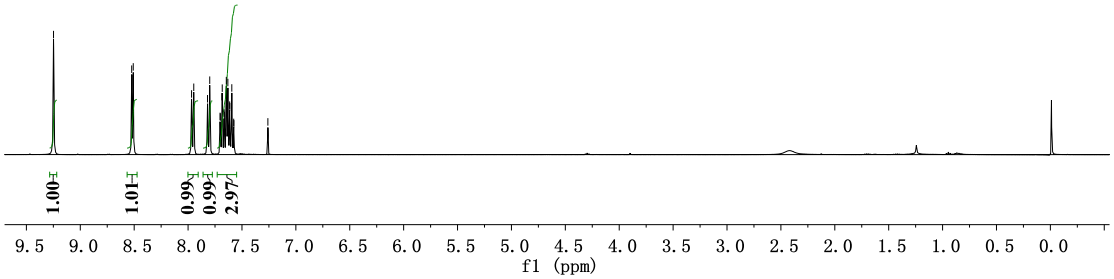

8.49
8.49
8.48
8.47
7.82
7.81
7.80
7.79
7.59
7.59
7.57
7.57
7.55
7.55
7.52
7.52
7.51
7.50
7.49
7.48
4.93
4.64
3.32
1.00
1.00
1.04
0.99
f1 (ppm)
N
Cl
1H NMR(400 MHz, CD3OD)

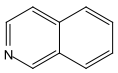

142.13
140.39
130.95
129.22
126.32
49.69
49.60
49.47
49.38
49.26
49.17
49.05
48.96
48.83
48.62
48.41
f1 (ppm)
N
Cl
13C NMR(100.6 MHz, CD3OD)



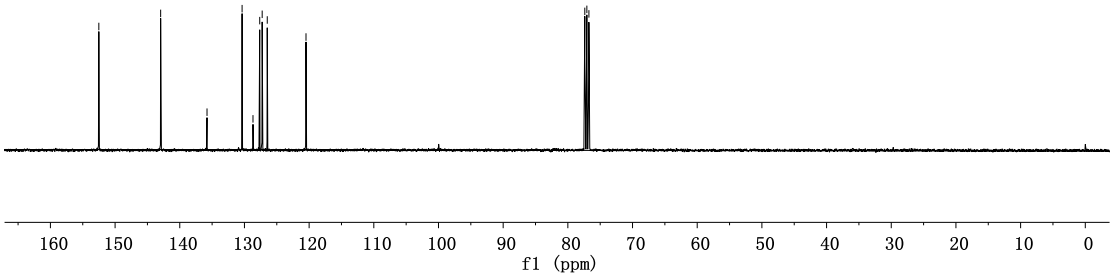


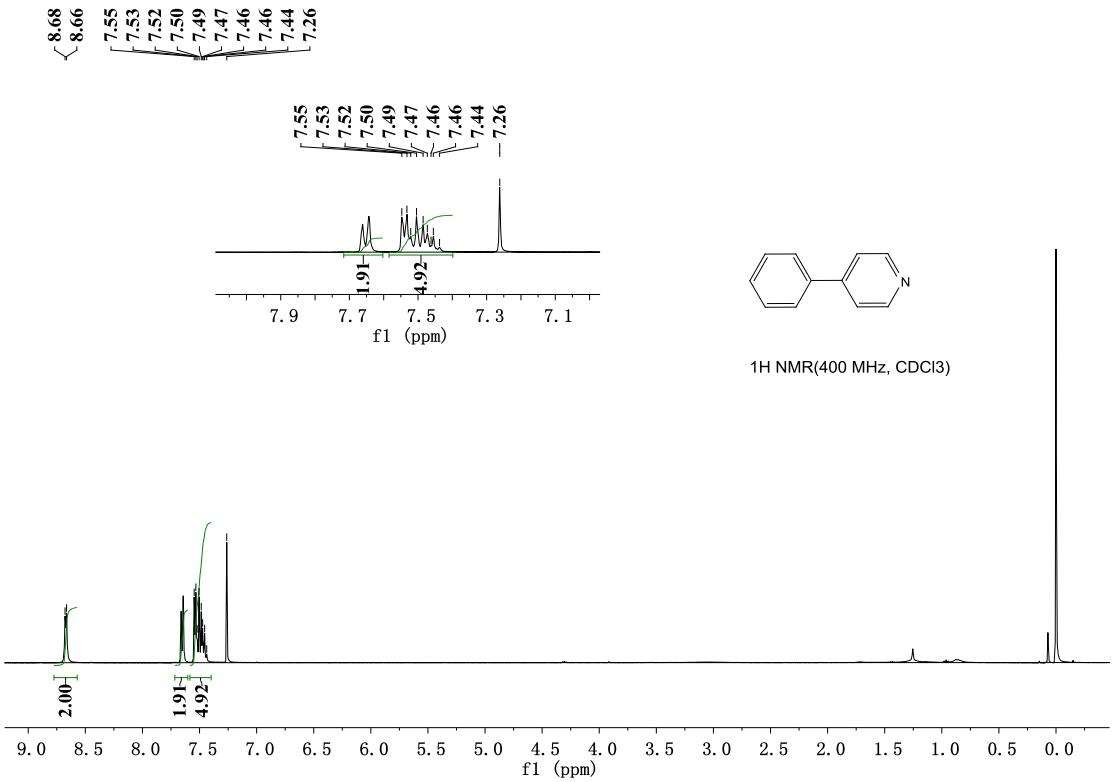
1H NMR(400 MHz, CDCl3)

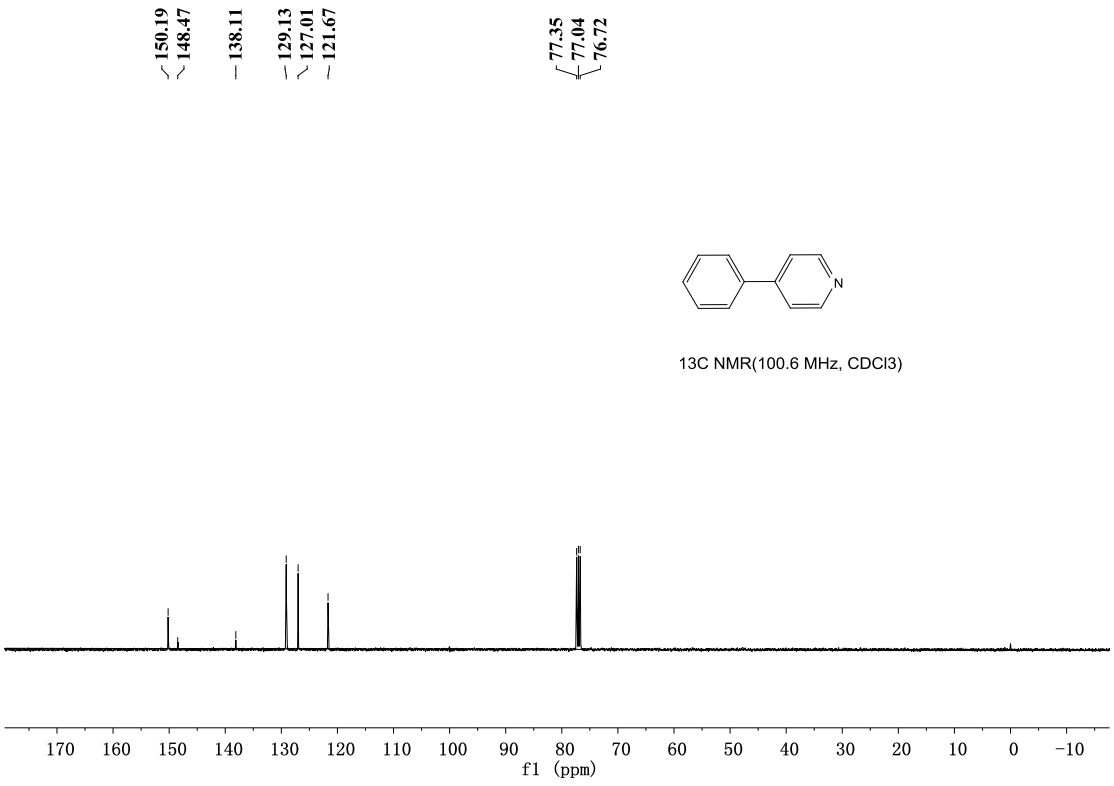


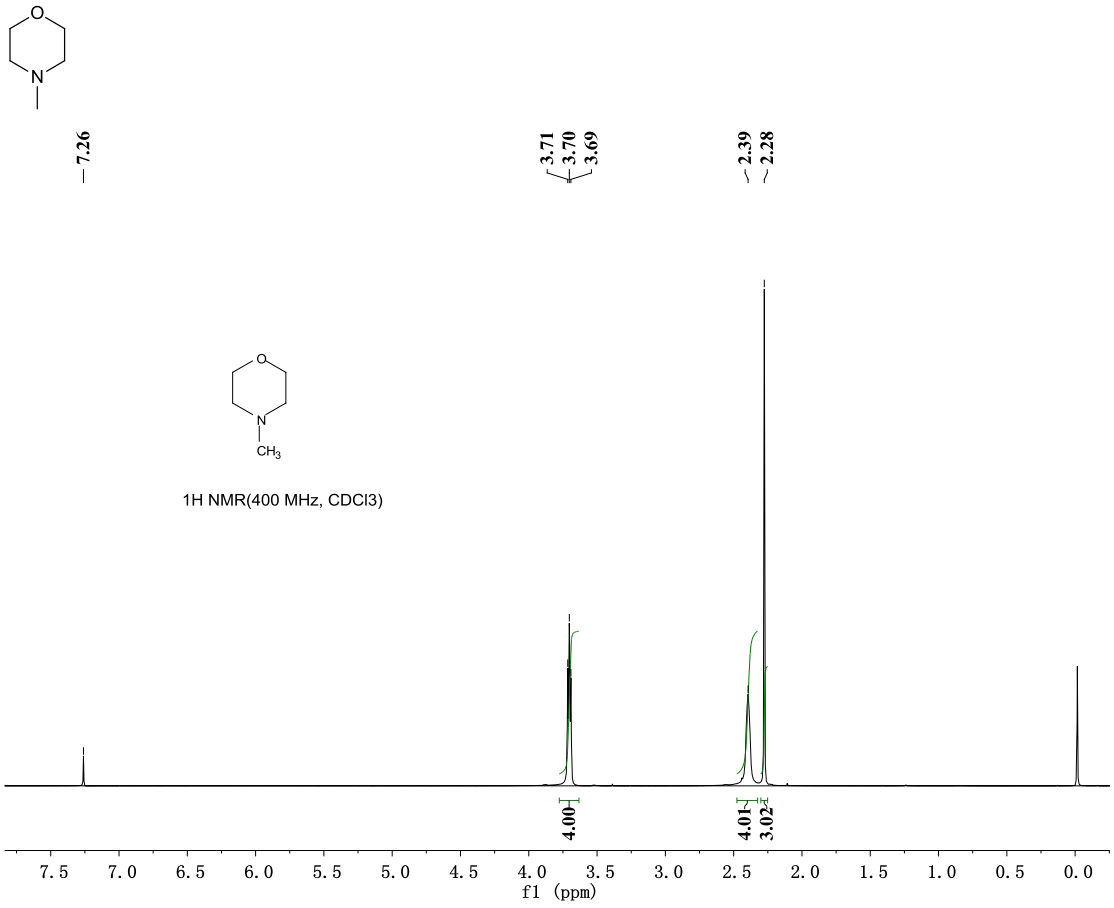


13C NMR(100.6 MHz, CDCl3)

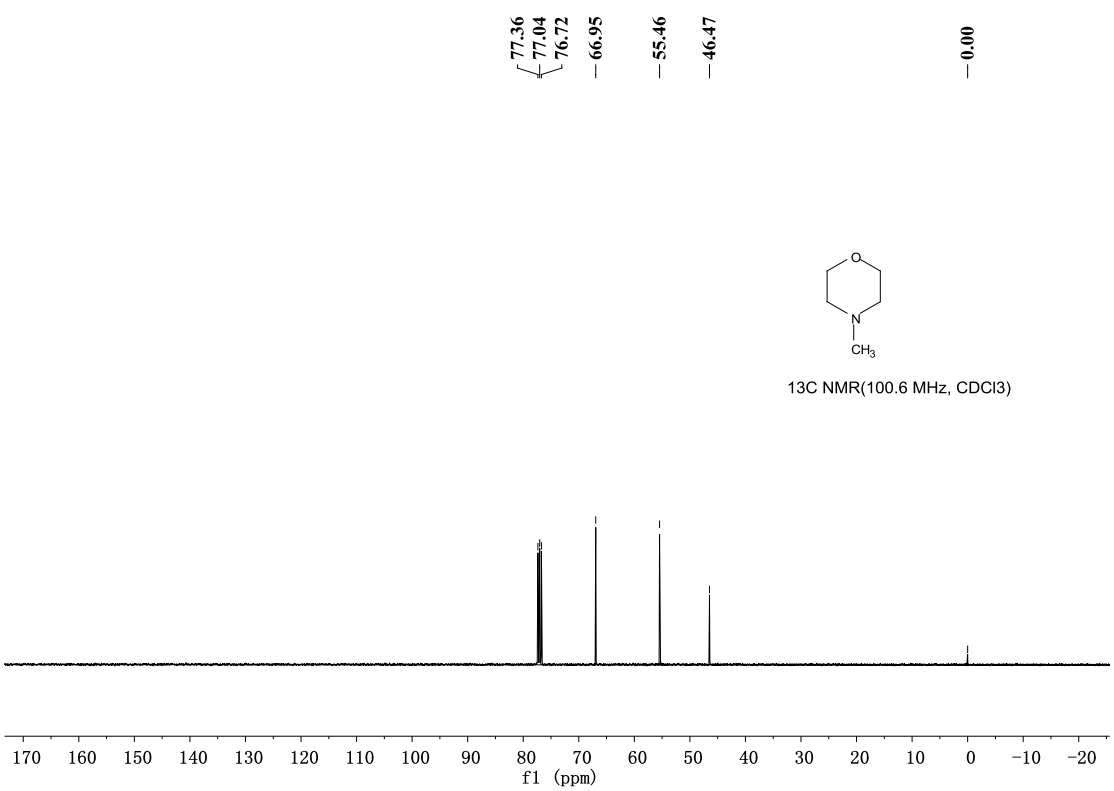



8.68
8.66
7.55
7.53
7.52
7.50
7.49
7.47
7.46
7.46
7.44
7.26
1.91
4.92
7.9
7.7
7.5
7.3
7.1
f1 (ppm)
N
1H NMR(400 MHz, CDCl3)
2.00
1.91
4.92
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


150.19
148.47
138.11
129.13
127.01
121.67
77.35
77.04
76.72
N
13C NMR(100.6 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

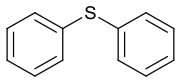


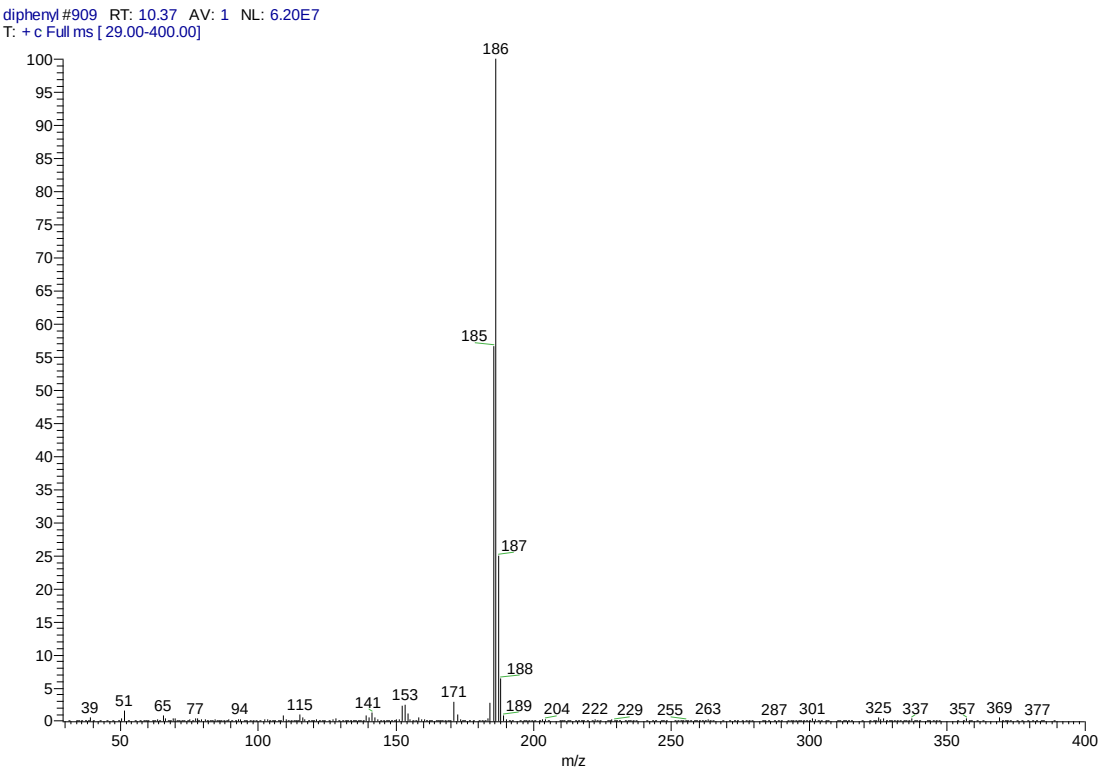
1H NMR(400 MHz, CDCl3)

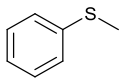


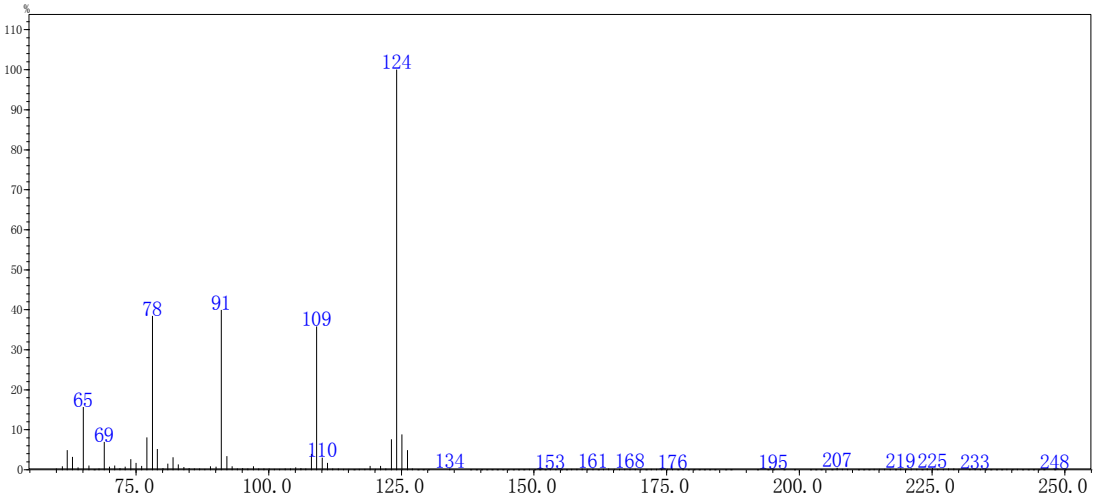
13C NMR(100.6 MHz, CDCl3)

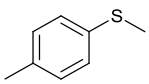
4.2 GC-MS Spectral of the Products.

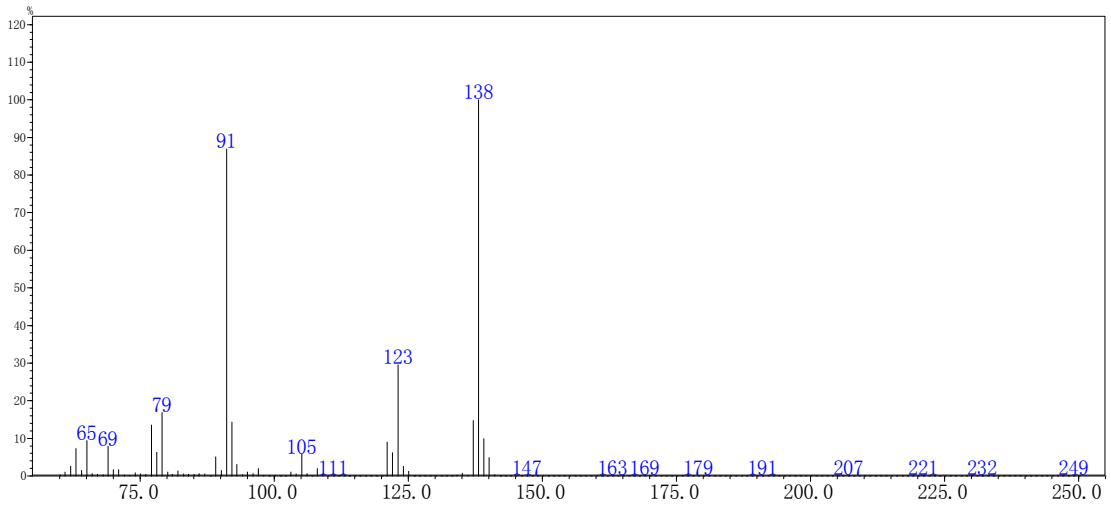


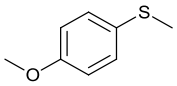


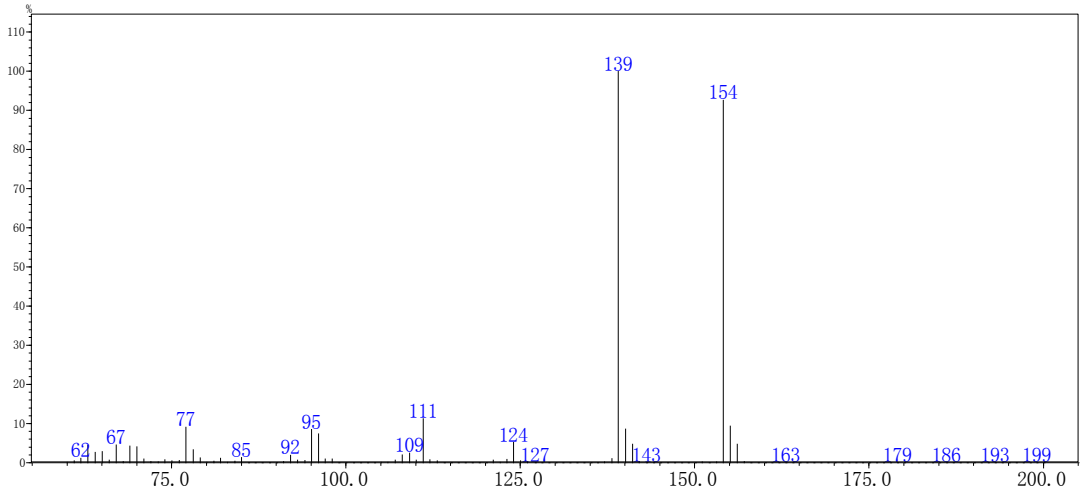


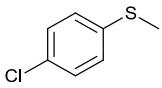


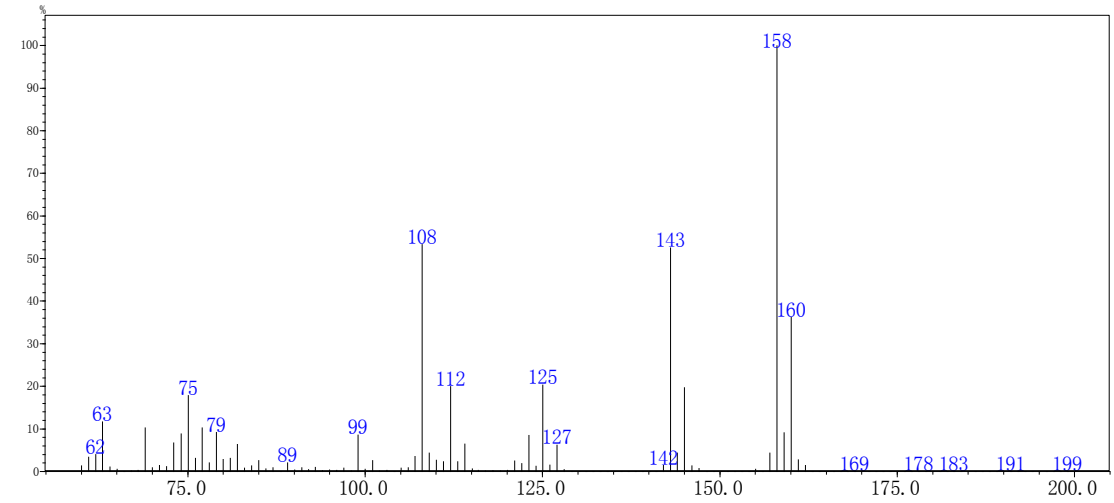








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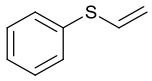
200.0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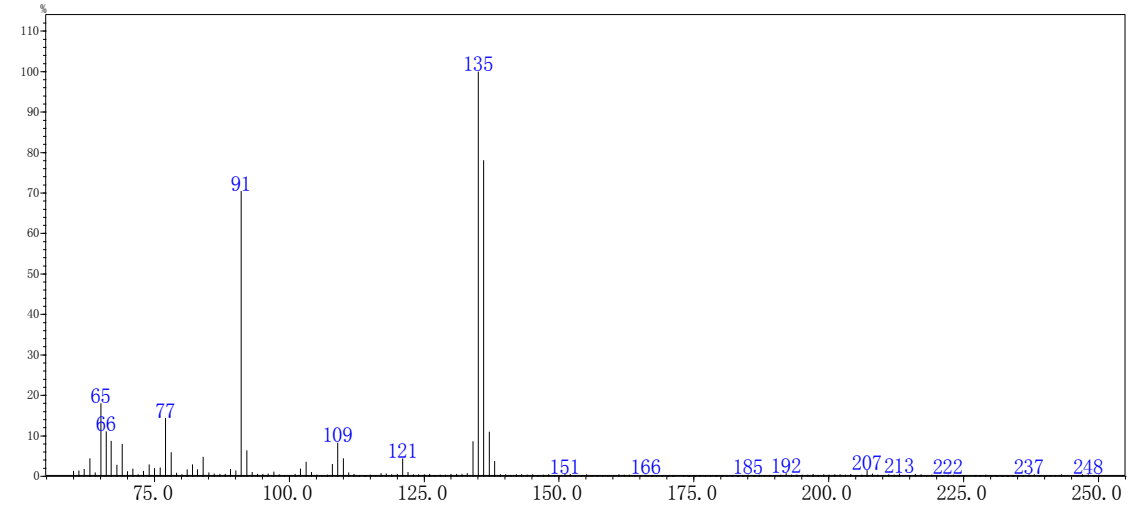
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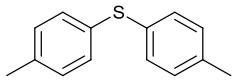
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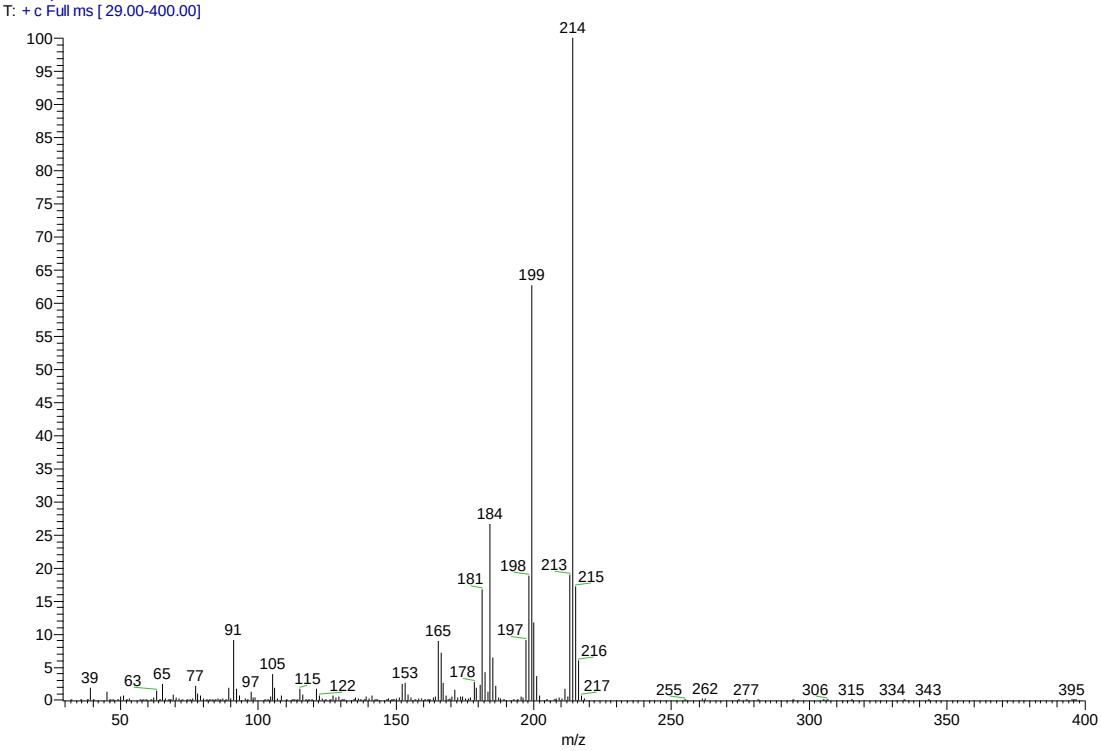
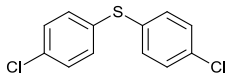
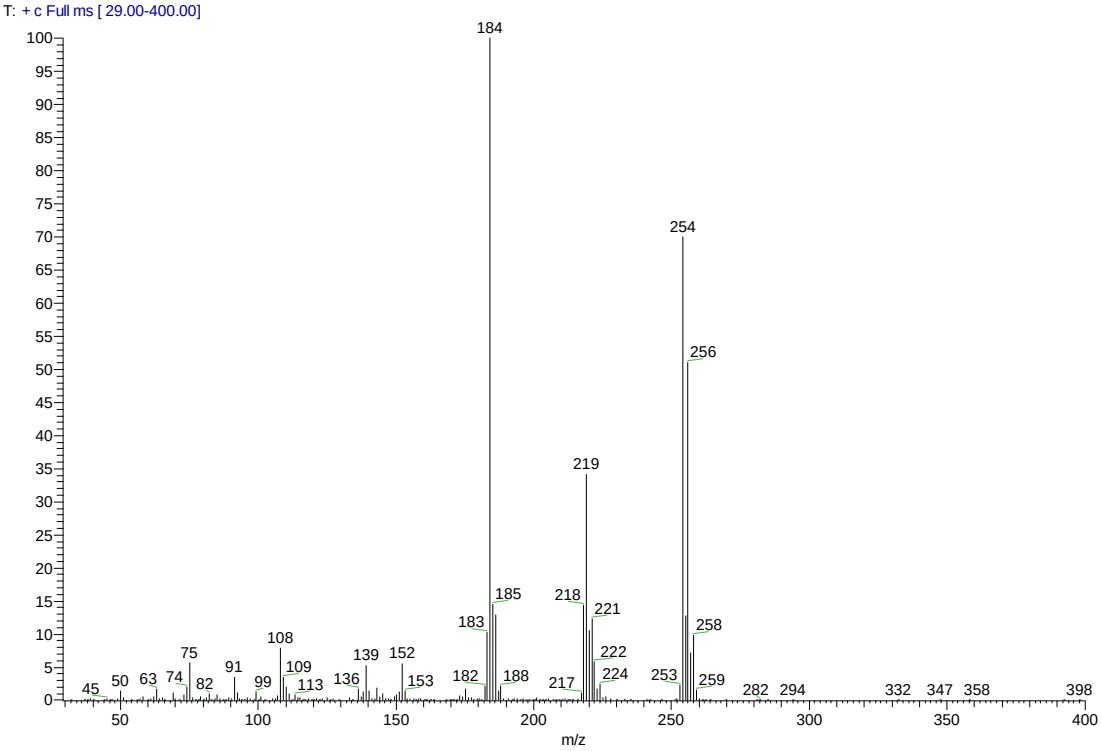
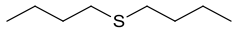
dimethyl #1045 RT: 12.06 AV: 1 NL: 3.89E6
T: + c Full ms [29.00-400.00]

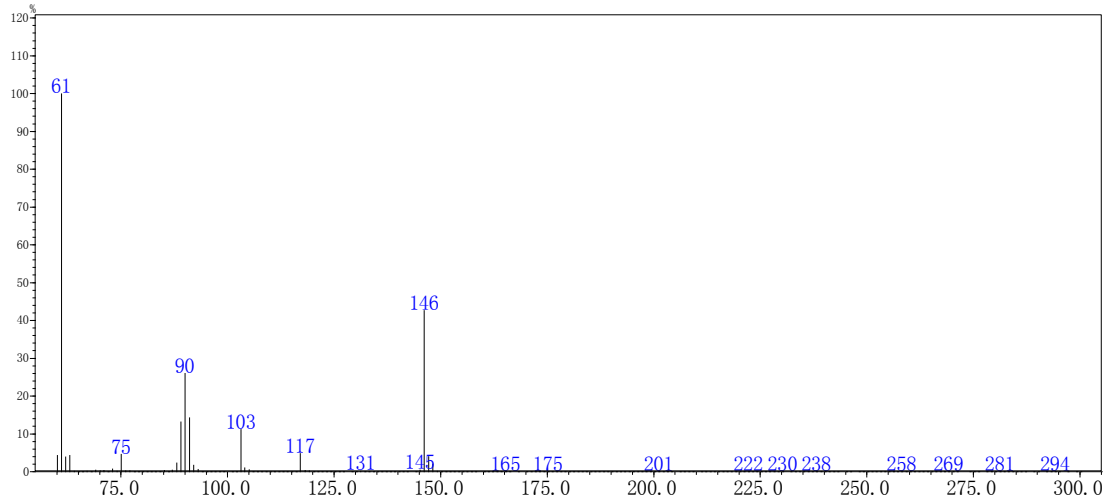


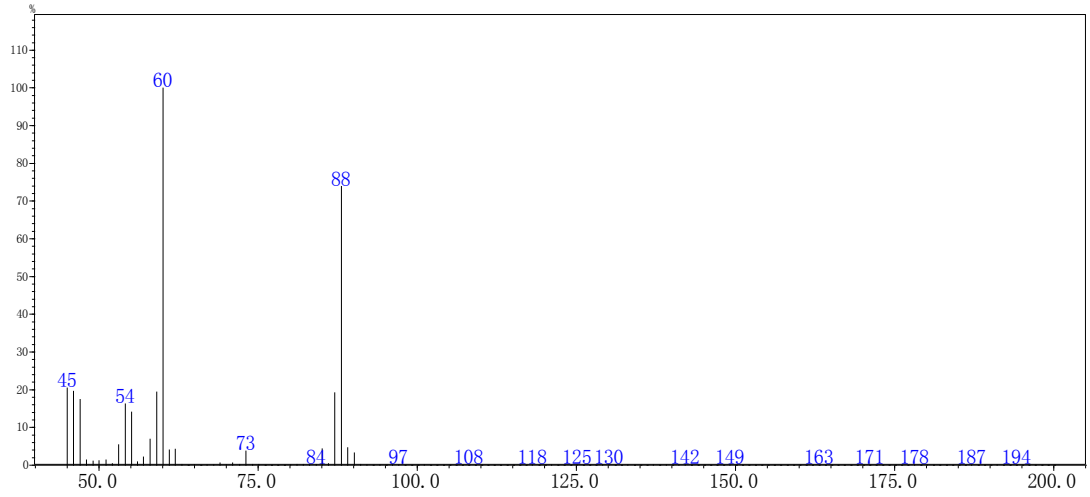


dichloide #1010 RT: 11.92 AV: 1 NL: 5.17E6
T: + c Full ms [29.00-400.00]

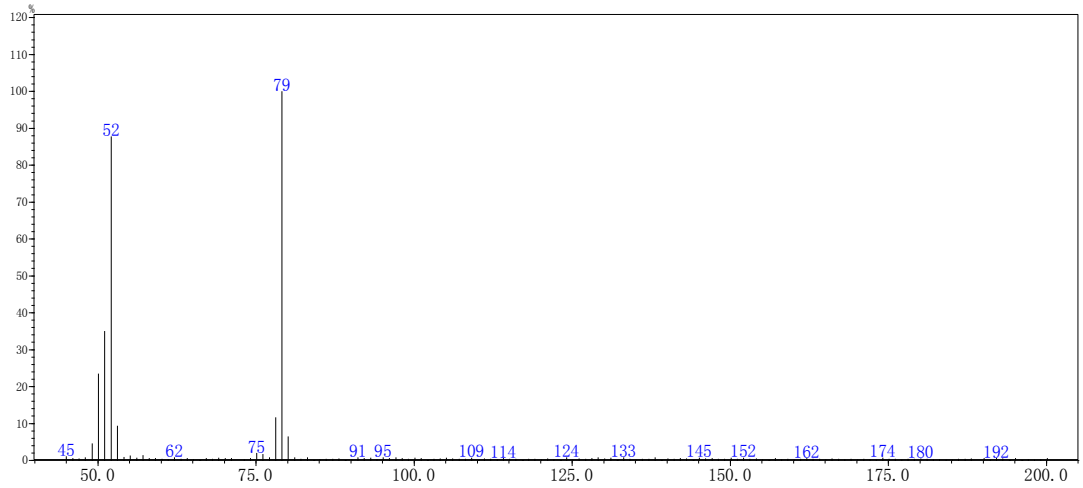


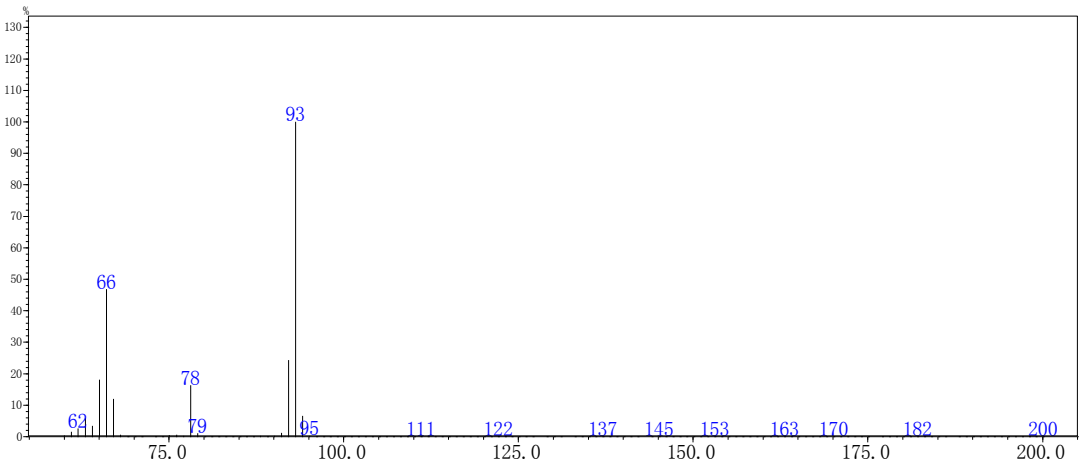






2.2 GC-MS Spectral Data of the Pyridines

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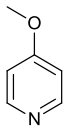
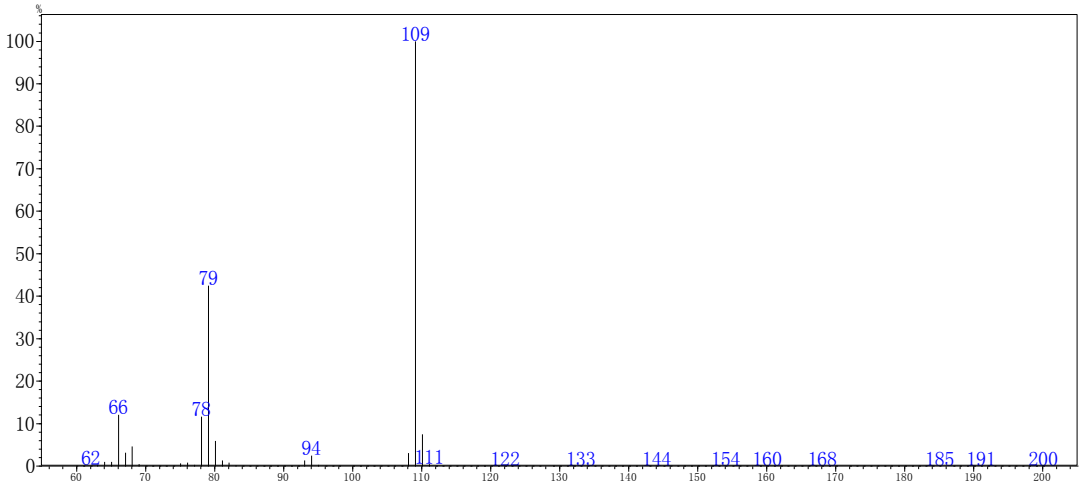
200.0

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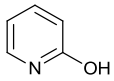

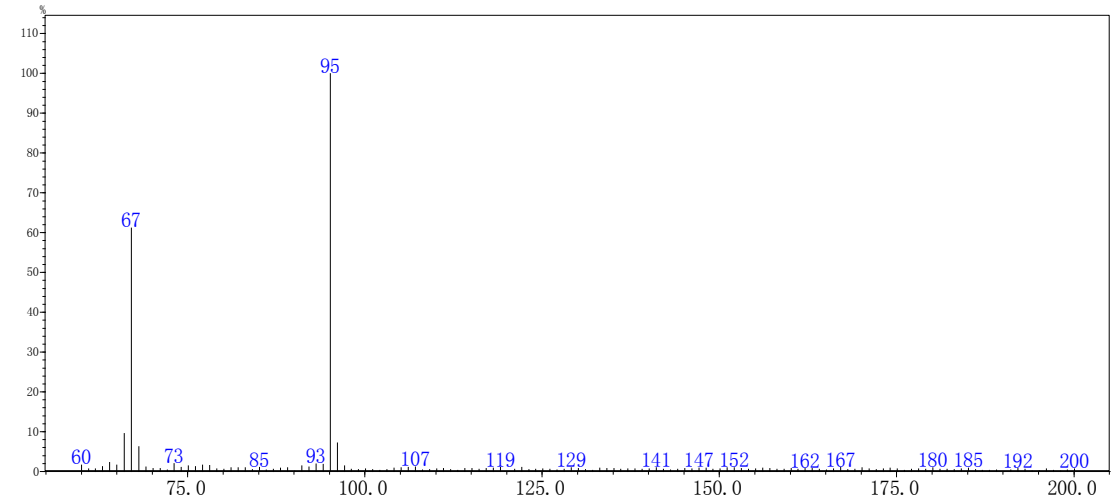
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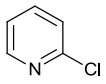
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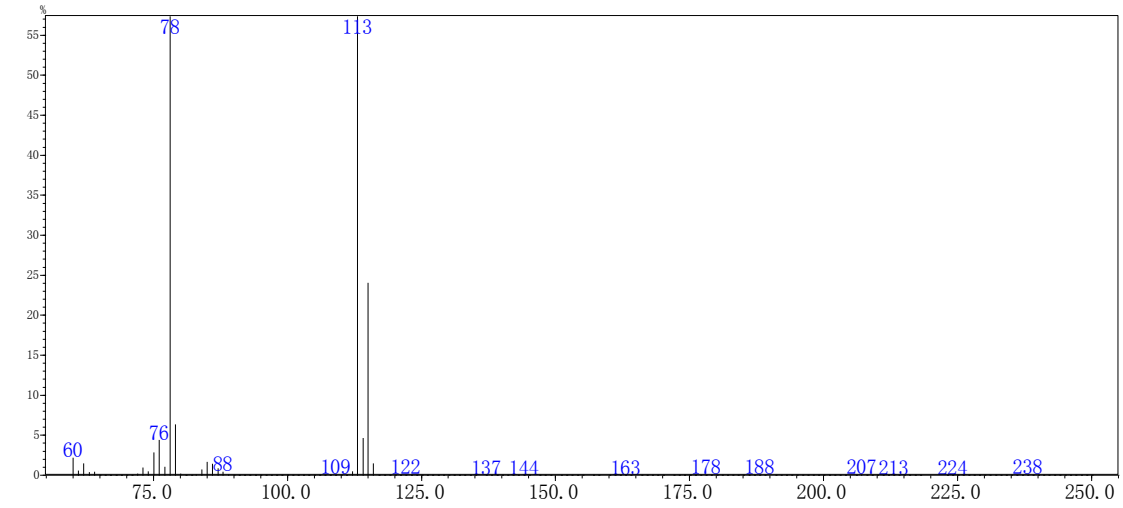
Cl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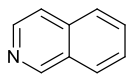
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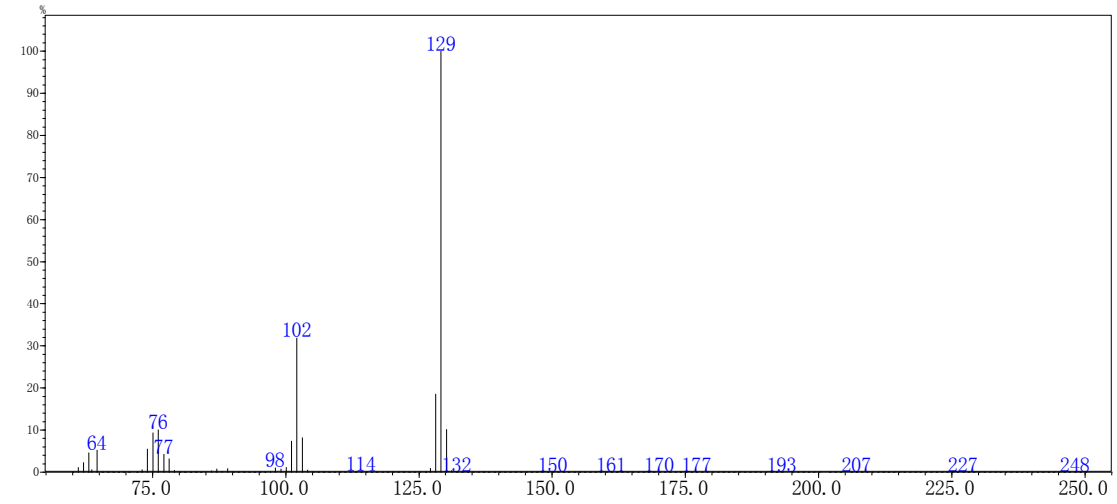
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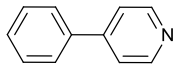

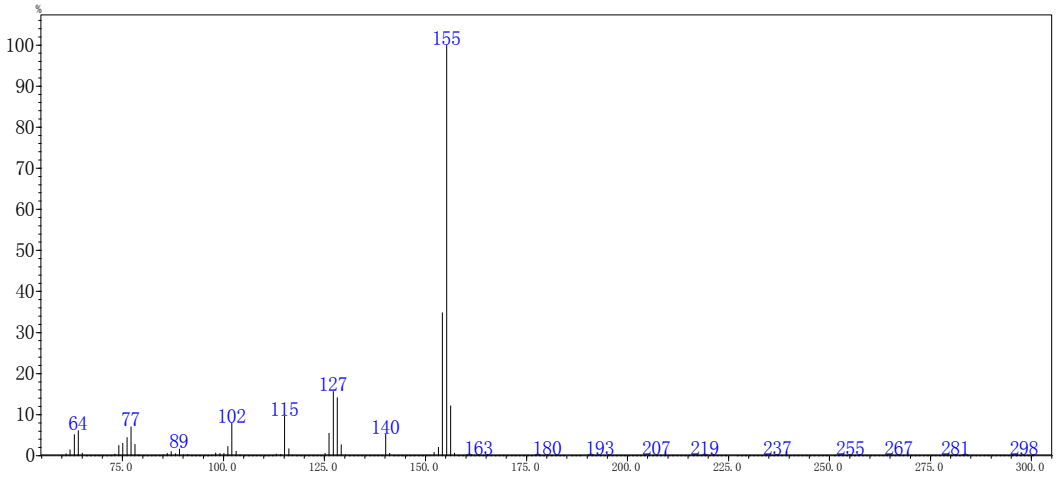

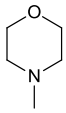

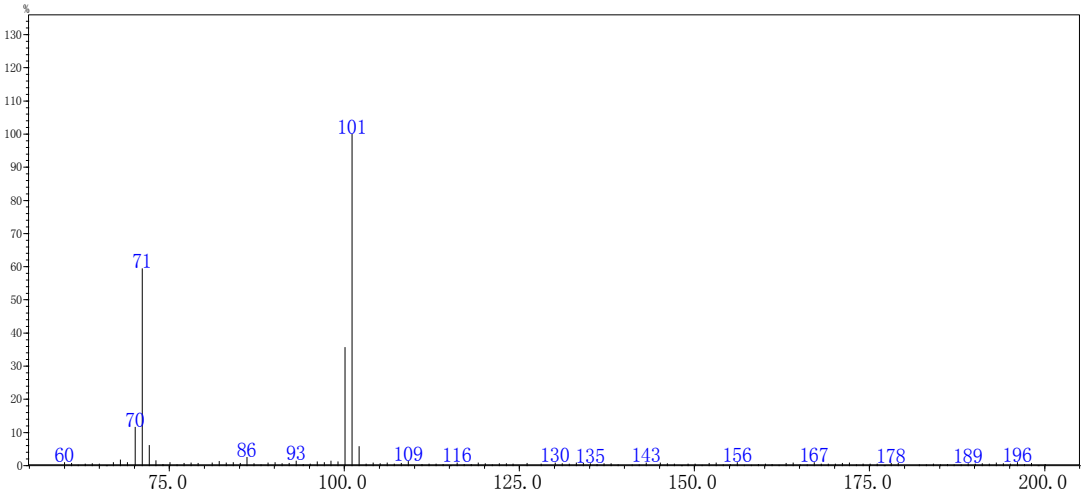

5. References

1. B. Yu, A.-H. Liu, L.-N. He, B. Li, Z.-F. Diao and Y.-N. Li, Green Chem, 2012, 14, 957-962.
2. S. P. Das, J. J. Boruah, H. Chetry and N. S. Islam, Tetrahedron Lett, 2012, 53, 1163-1168.
3. A. C. Fernandes, J. A. Fernandes, C. C. Romão, L. F. Veiros and M. J. Calhorda, Organometallics, 2010, 29, 5517-5525.
4. M. Arisawa, Y. Nihei, T. Suzuki and M. Yamaguchi, Org Lett, 2012, 14, 855-857.
5. R. V. C. Carr, R. V. Williams and L. A. Paquette, J Org Chem, 1983, 48, 4976-4986.
6. N. García, P. García-García, M. A. Fernández-Rodríguez, R. Rubio, M. R. Pedrosa, F. J. Arnáiz and R. Sanz, Adv Synth Catal, 2012, 354, 321-327.
7. K. Smith, G. A. El-Hiti and A. J. Al-Zuhairi, Journal of Sulfur Chemistry, 2011, 32, 521-531.
8. P. Kircher, G. Huttner, K. Heinze, B. Schiemenz, L. Zsolnai, M. Büchner and A. Driess, Eur J Inorg Chem, 1998, 1998, 703-720.
9. N. B. Gowda, G. K. Rao and R. A. Ramakrishna, Tetrahedron Lett, 2010, 51, 5690-5693.
10. J. R. Hwu, W. N. Tseng, H. V. Patel, F. F. Wong, D.-N. Horng, B. R. Liaw and L. C. Lin, J Org Chem, 1999, 64, 2211-2218.
11. M. Toganoh, K. Fujino, S. Ikeda and H. Furuta, Tetrahedron Lett, 2008, 49, 1488-1491.
12. S. C. Schou, Journal of Labelled Compounds and Radiopharmaceuticals, 2009, 52, 376-381.

13. T. Tanaka, K.-i. Okunaga and M. Hayashi, Tetrahedron Lett, 2010, 51, 4633-4635.
14. S. Ouk, S. Thiébaud, E. Borredon and B. Chabaud, Synth Commun, 2005, 35, 3021-3026.