Electronic Supplementary Material (ESI) for Chemical Communications

Tetrabutylammonium Iodide Catalyzed Allylic Sulfonylation of α-Methyl Styrene Derivatives with Sulfonylhydrazides

Xiaoqing Li,* Xiangsheng Xu, Can Zhou

College of Chemical Engineering and Materials Science, Zhejiang University of Technology,

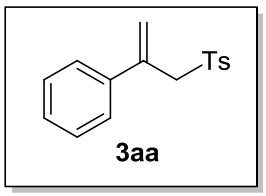
Hangzhou 310014, (P. R. China)

E-mail: xqli@zjut.edu.cn

Table of contents

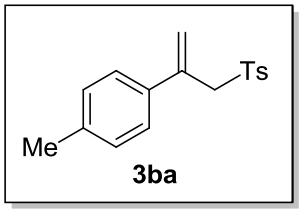
General Methods: All reagents and solvents were purchased from commercial suppliers and used without purifications. Melting points are uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 ℃ in $CDCl_3$ at 500 and 125 MHz, respectively, with TMS as the internal standard. Chemical shifts (δ) are expressed in ppm and coupling constants J are given in Hz. High resolution mass spectra (HRMS) were obtained on a TOF MS instrument with ESI source. The alkenes were synthesized form the corresponding ketones with methyltriphenylphosphonium bromide according to literature method.[1]

General procedure for the synthesis of compounds 3: alkenes (0.5 mmol) and TBHP (1 mmol) was added to a solution of sulfonylhydrazides (0.6 mmol) and tetrabutylammonium iodide (0.1 mmol) in 2 mL of MeCN. After the reaction mixture was stirred at 80 ^{o}C for 20 h, the solvent was removed under reduced pressure, and the residual was treated with silica gel chromatography to give sulfones **3**.



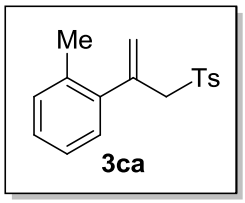
3aa

1-methyl-4-((2-phenylallyl)sulfonyl)benzene(3aa): white solid. mp. 97-98 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.2 Hz, 2H), 7.28 – 7.18 (m, 7H), 5.57 (s, 1H), 5.20 (s, 1H), 4.24 (s, 2H), 2.37 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 138.9, 136.6, 135.5, 129.4, 128.6, 128.3, 127.8, 126.2, 121.6, 62.1, 21.5. HRMS (ESI) calcd for $C_{16}H_{17}O_2S$ $(M + H)^+$: 273.0949; found: 273.0954.



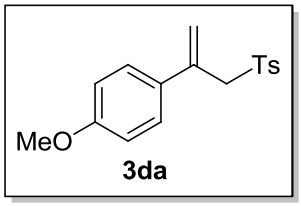
3ba

1-methyl-4-((2-(p-tolyl)allyl)sulfonyl)benzene(3ba): white solid. mp. 81-83 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 5.54 (s, 1H), 5.12 (s, 1H), 4.22 (s, 2H), 2.39 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 137.8, 136.5, 136.0, 135.6, 129.4, 129.0, 128.7, 126.1, 120.8, 62.2, 21.5, 21.0. HRMS (ESI) calcd for $C_{17}H_{19}O_2S$ $(M + H)^+$: 287.1106; found: 287.1110.



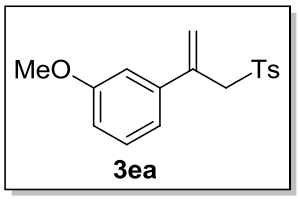
3ca

1-methyl-2-(3-tosylprop-1-en-2-yl)benzene(3ca): colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.14 – 6.99 (m, 3H), 6.94 (dd, *J* = 7.6, 0.9 Hz, 1H), 5.48 (d, *J* = 0.8 Hz, 1H), 5.27 (d, *J* = 1.0 Hz, 1H), 4.13 (s, 2H), 2.38 (s, 3H), 2.23 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 140.0, 136.8, 136.1, 134.5, 130.2, 129.5, 128.6, 128.2, 127.5, 125.5, 124.0, 63.2, 21.4, 19.9. HRMS (ESI) calcd for $C_{17}H_{19}O_2S$ $(M + H)^+$: 287.1106; found: 287.1109.



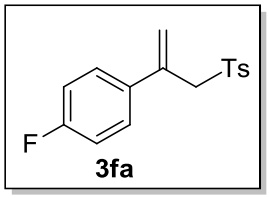
3da

1-methoxy-4-(3-tosylprop-1-en-2-yl)benzene(3da): yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.3 Hz, 2H), 7.23 (dt, *J* = 5.3, 2.1 Hz, 4H), 6.78 (d, *J* = 8.8 Hz, 2H), 5.49 (s, 1H), 5.07 (s, 1H), 4.22 (s, 2H), 3.79 (s, 3H), 2.40 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.5, 144.5, 136.0, 135.6, 131.3, 129.5, 128.7, 127.5, 119.9, 113.7, 62.3, 55.3, 21.6. HRMS (ESI) calcd for $C_{17}H_{19}O_3S$ $(M + H)^+$: 303.1055; found: 303.1059.



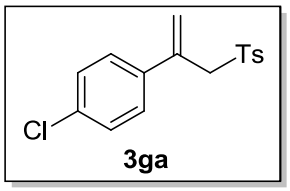
3ea

1-methoxy-3-(3-tosylprop-1-en-2-yl)benzene(3ea): white solid. mp. 89-90 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.2 Hz, 2H), 7.23 – 7.12 (m, 3H), 6.85 (d, *J* = 7.7 Hz, 1H), 6.79 – 6.74 (m, 2H), 5.58 (s, 1H), 5.22 (s, 1H), 4.23 (s, 2H), 3.76 (s, 3H), 2.38 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.5, 144.5, 140.4, 136.6, 135.5, 129.4, 129.3, 128.6, 121.8, 118.7, 113.3, 112.1, 62.2, 55.2, 21.5. HRMS (ESI) calcd for $C_{17}H_{19}O_3S$ $(M + H)^+$: 303.1055; found: 303.1058.



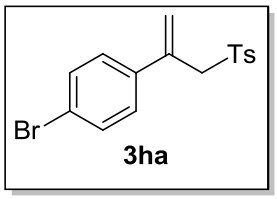
3fa

1-fluoro-4-(3-tosylprop-1-en-2-yl)benzene(3fa): white solid. mp. 110-111 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.3 Hz, 2H), 7.30 – 7.19 (m, 4H), 6.97 – 6.88 (m, 2H), 5.52 (s, 1H), 5.17 (s, 1H), 4.21 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.5, 161.6, 144.7, 135.7, 135.5, 135.1, 135.0, 129.5, 128.6, 128.1, 128.0, 121.7, 115.3, 115.1, 62.3, 21.5. HRMS (ESI) calcd for $C_{16}H_{16}FO_2S$ $(M + H)^+$: 291.0855; found: 291.0858.



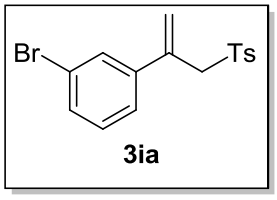
3ga

1-chloro-4-(3-tosylprop-1-en-2-yl)benzene(3ga): white solid. mp. 110-112 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.3 Hz, 2H), 7.25 – 7.17 (m, 6H), 5.56 (s, 1H), 5.20 (s, 1H), 4.20 (s, 2H), 2.40 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 137.3, 135.7, 135.4, 133.9, 129.6, 128.6, 128.4, 127.6, 122.2, 62.1, 21.6. HRMS (ESI) calcd for $C_{16}H_{16}ClO_2S$ $(M + H)^+$: 307.0560; found: 307.0564.



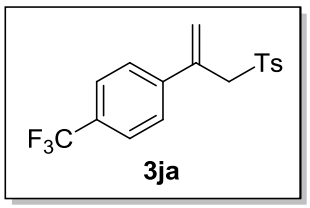
3ha

1-bromo-4-(3-tosylprop-1-en-2-yl)benzene(3ha): white solid. mp. 130-132 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.6 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.14 (d, *J* = 8.6 Hz, 2H), 5.57 (s, 1H), 5.22 (s, 1H), 4.20 (s, 2H), 2.41 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 137.8, 135.8, 135.4, 131.5, 129.6, 128.6, 127.9, 122.3, 122.1, 62.1, 21.6. HRMS (ESI) calcd for $C_{16}H_{16}BrO_2S$ $(M + H)^+$: 351.0054; found: 351.0057.



3ia

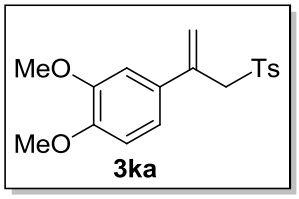
1-bromo-3-(3-tosylprop-1-en-2-yl)benzene(3ia): yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.3 Hz, 2H), 7.32 (m, 1H), 7.21 (m, 4H), 7.09 (t, *J* = 7.9 Hz, 1H), 5.57 (s, 1H), 5.29 (s, 1H), 4.20 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.8, 140.9, 135.5, 135.2, 130.7, 129.8, 129.5, 129.3, 128.5, 124.9, 122.9, 122.4, 61.9, 21.5. HRMS (ESI) calcd for $C_{16}H_{16}BrO_2S$ $(M + H)^+$: 351.0054; found: 351.0057.



3ja

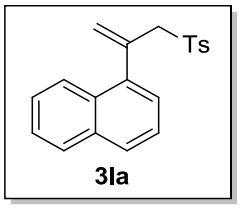
1-methyl-4-((2-(4-(trifluoromethyl)phenyl)allyl)sulfonyl)benzene(3ja): white solid. mp. 124-126 ^{o}C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, J = 8.3 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.1 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 5.66 (s, 1H), 5.35 (s, 1H), 4.25 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.9, 142.4, 135.8,

135.4, 130.1, 129.8, 129.7, 129.6, 129.5, 128.6, 127.9, 127.3, 126.7, 125.6, 125.6, 125.4, 125.3, 125.3, 125.3, 125.1, 123.7, 122.9, 62.1, 21.4. HRMS (ESI) calcd for $C_{17}H_{16}F_3O_2S$ $(M + H)^+$: 341.0823; found: 341.0826.



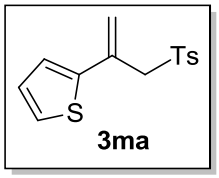
3ka

1,2-dimethoxy-4-(3-tosylprop-1-en-2-yl)benzene(3ka): yellow solid. mp. 69-71 ^oC . ^1H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H), 6.83 – 6.77 (m, 2H), 6.70 (d, *J* = 8.3 Hz, 1H), 5.48 (s, 1H), 5.09 (s, 1H), 4.20 (s, 2H), 3.82 (s, 3H), 3.80(s, 3H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 148.9, 148.5, 144.4, 136.1, 135.5, 131.6, 129.3, 128.5, 120.0, 118.9, 110.8, 109.6, 62.2, 55.7, 55.7, 21.3. HRMS (ESI) calcd for $C_{18}H_{21}O_4S$ $(M + H)^+$: 333.1161; found: 333.1167.

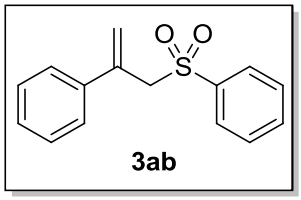


3la

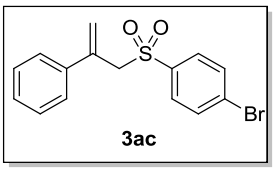
1-(3-tosylprop-1-en-2-yl)naphthalene(3la): yellow oil. ^1H NMR (500 MHz, $CDCl_3$) δ 7.82 – 7.74 (m, 2H), 7.67 (d, *J* = 8.2 Hz, 1H), 7.53 (d, *J* = 8.3 Hz, 2H), 7.45 – 7.39 (m, 2H), 7.27 (dd, *J* = 8.1, 7.1 Hz, 1H), 7.14 (dd, *J* = 7.1, 1.1 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 2H), 5.71 (d, *J* = 0.8 Hz, 1H), 5.49 (d, *J* = 1.1 Hz, 1H), 4.31 (s, 2H), 2.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.2, 138.0, 135.8, 133.5, 130.4, 129.2, 128.4, 128.1, 128.0, 126.1, 125.9, 125.7, 125.1, 125.0, 124.8, 63.6, 21.4. HRMS (ESI) calcd for $C_{20}H_{19}F_3O_2S$ $(M + H)^+$: 323.1106; found: 323.1110.



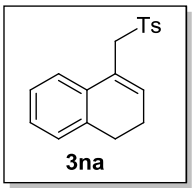
2-(3-tosylprop-1-en-2-yl)thiophene(3ma): yellow solid. mp. 65-66 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.3 Hz, 4H), 7.26 (d, *J* = 8.0 Hz, 6H), 7.13 (dd, *J* = 5.1, 1.0 Hz, 2H), 6.93 (dd, *J* = 3.6, 1.0 Hz, 2H), 6.87 (dd, *J* = 5.1, 3.7 Hz, 2H), 5.62 (s, 2H), 5.01 (s, 2H), 4.19 (s, 4H), 2.41 (s, 6H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.7, 142.7, 135.3, 130.2, 129.5, 128.7, 127.5, 125.3, 125.2, 119.5, 62.4, 21.5. HRMS (ESI) calcd for $C_{14}H_{15}O_2S_2$ $(M + H)^+$: 279.0513; found: 279.0516.



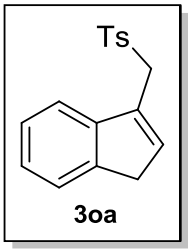
((2-phenylallyl)sulfonyl)benzene(3ab): white solid. mp. 60-61 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 – 7.74 (m, 2H), 7.58 – 7.49 (m, 1H), 7.45 – 7.37 (m, 2H), 7.29 – 7.18 (m, 5H), 5.58 (s, 1H), 5.21 (s, 1H), 4.27 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.7, 138.4, 136.5, 133.6, 128.8, 128.6, 128.3, 128.0, 126.1, 121.7, 62.0. HRMS (ESI) calcd for $C_{15}H_{15}O_2S$ $(M + H)^+$: 259.0793; found:259.0796.



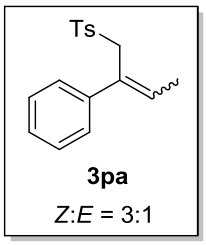
1-bromo-4-((2-phenylallyl)sulfonyl)benzene(3ac): white solid. mp. 127-128 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 – 7.56 (m, 2H), 7.54 – 7.49 (m, 2H), 7.25 – 7.19 (m, 5H), 5.59 (s, 1H), 5.24 (s, 1H), 4.27 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.5, 137.4, 136.4, 132.1, 130.2, 129.0, 128.4, 128.1, 126.2, 122.1, 62.2. HRMS (ESI) calcd for $C_{15}H_{14}BrO_2S$ $(M + H)^+$: 336.9898; found: 336.9902.



4-(tosylmethyl)-1,2-dihydronaphthalene(3na): white solid. mp. 76-77 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 8.1 Hz, 2H), 7.16 – 7.02 (m, 4H), 5.90 (t, *J* = 4.6 Hz, 1H), 4.19 (s, 2H), 2.68 (t, *J* = 8.1 Hz, 2H), 2.36 (d, *J* = 5.6 Hz, 3H), 2.21 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.5, 135.9, 135.5, 134.7, 132.7, 129.4, 128.7, 127.5, 127.2, 126.3, 125.9, 123.2, 60.0, 27.7, 23.3, 21.5. HRMS (ESI) calcd for $C_{18}H_{19}O_2S$ $(M + H)^+$: 299.1106; found: 299.1113.

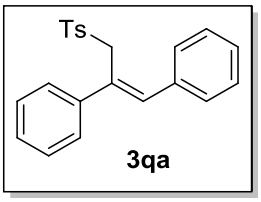


3-(tosylmethyl)-1H-indene(3oa): white solid. mp. 130-132 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 7.0 Hz, 1H), 7.27 – 7.12 (m, 5H), 6.33 (s, 1H), 4.32 (d, *J* = 0.7 Hz, 2H), 3.31 (s, 2H), 2.36 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.6, 143.3, 143.1, 137.1, 135.5, 131.9, 129.4, 128.5, 126.1, 125.0, 123.6, 119.5, 55.7, 38.3, 21.4. HRMS (ESI) calcd for $C_{17}H_{17}O_2S$ $(M + H)^+$: 285.0949; found: 285.0953.



1-methyl-4-((2-phenylbut-2-en-1-yl)sulfonyl)benzene(3pa): colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) ***Z* isomer** δ 7.62 (d, *J* = 8.3 Hz, 2H), 7.23 – 7.04 (m, 7H), 6.09 (q, *J* = 7.1 Hz, 1H), 4.34 (s, 2H), 2.37 (s, 3H), 1.67 (d, *J* = 7.1 Hz, 3H); ***E* isomer** δ 7.62

(d, *J* = 8.3 Hz, 2H), 7.23 – 7.04 (m, 7H), 5.78 (q, *J* = 7.0 Hz, 1H), 4.10 (s, 2H), 2.40 (s, 3H), 1.64 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4, 141.0, 136.2, 132.8, 132.7, 129.4, 129.2, 128.7, 128.5, 128.4, 128.2, 128.1, 127.0, 126.9, 126.3, 65.1, 57.6, 21.5, 21.5, 15.3, 15.0. HRMS (ESI) calcd for $C_{17}H_{19}O_2S$ $(M + H)^+$: 287.1106; found: 284.1110.



3qa

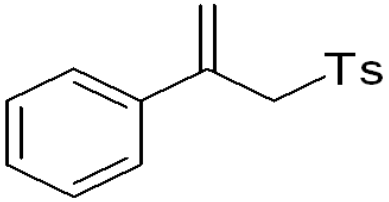
(3-tosylprop-1-ene-1,2-diyl)dibenzene(3qa): white solid. mp. 128-130 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.3 Hz, 2H), 7.38 – 7.20 (m, 10H), 7.06 (d, *J* = 8.0 Hz, 2H), 7.00 (s, 1H), 4.55 (s, 2H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.2, 140.4, 136.8, 136.5, 136.1, 130.5, 129.4, 128.6, 128.5, 128.4, 128.3, 127.8, 127.6, 126.8, 58.0, 21.5. HRMS (ESI) calcd for $C_{22}H_{21}O_2S$ $(M + H)^+$: 349.1262; found: 349.1265.

References

[1] S. Ohsugi, K. Nishide, M. Node, *Tetrahedron*, **2003**, *59*, 1859.

120716
lxq146-1 CDCl3

7.6559
7.6395
7.2621
7.2552
7.2512
7.2349
7.2279
7.2186
7.2138
7.2060
7.1897
5.5722
5.2008
4.2387
2.3677



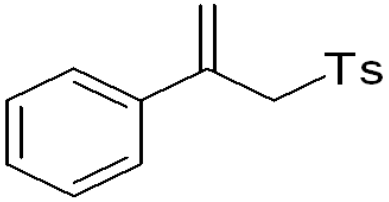
3aa

1.96
7.06
1.00
1.01
2.06
3.00

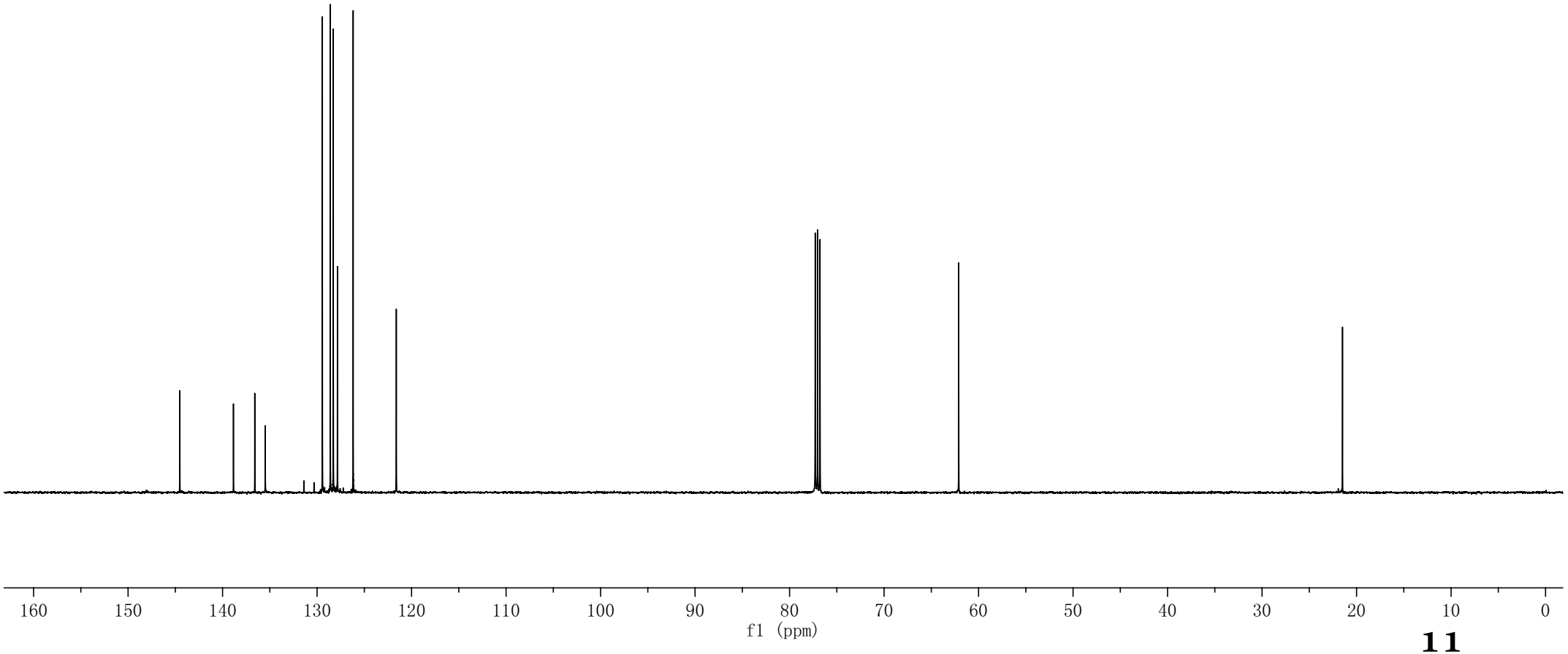
f1 (ppm)

120716
lxq146-1 CDCl3

144.5216
138.8503
136.5925
135.4891
129.4383
128.5957
128.2804
127.8463
126.1804
121.6137
77.2865
77.0323
76.7777
62.0996
21.4842

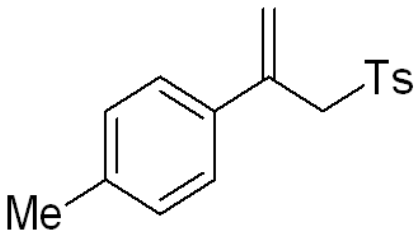


3aa



120718
LXQ146-4 CDCl3

7.6621
7.6456
7.2547
7.2242
7.2081
7.1753
7.1589
7.0545
7.0385
5.5390
5.1224
4.2246
2.3892
2.3071



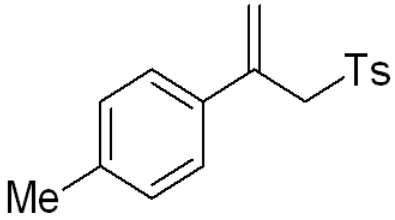
3ba

1.99
2.04
2.09
2.03
1.02
1.02
2.05
2.96
3.00

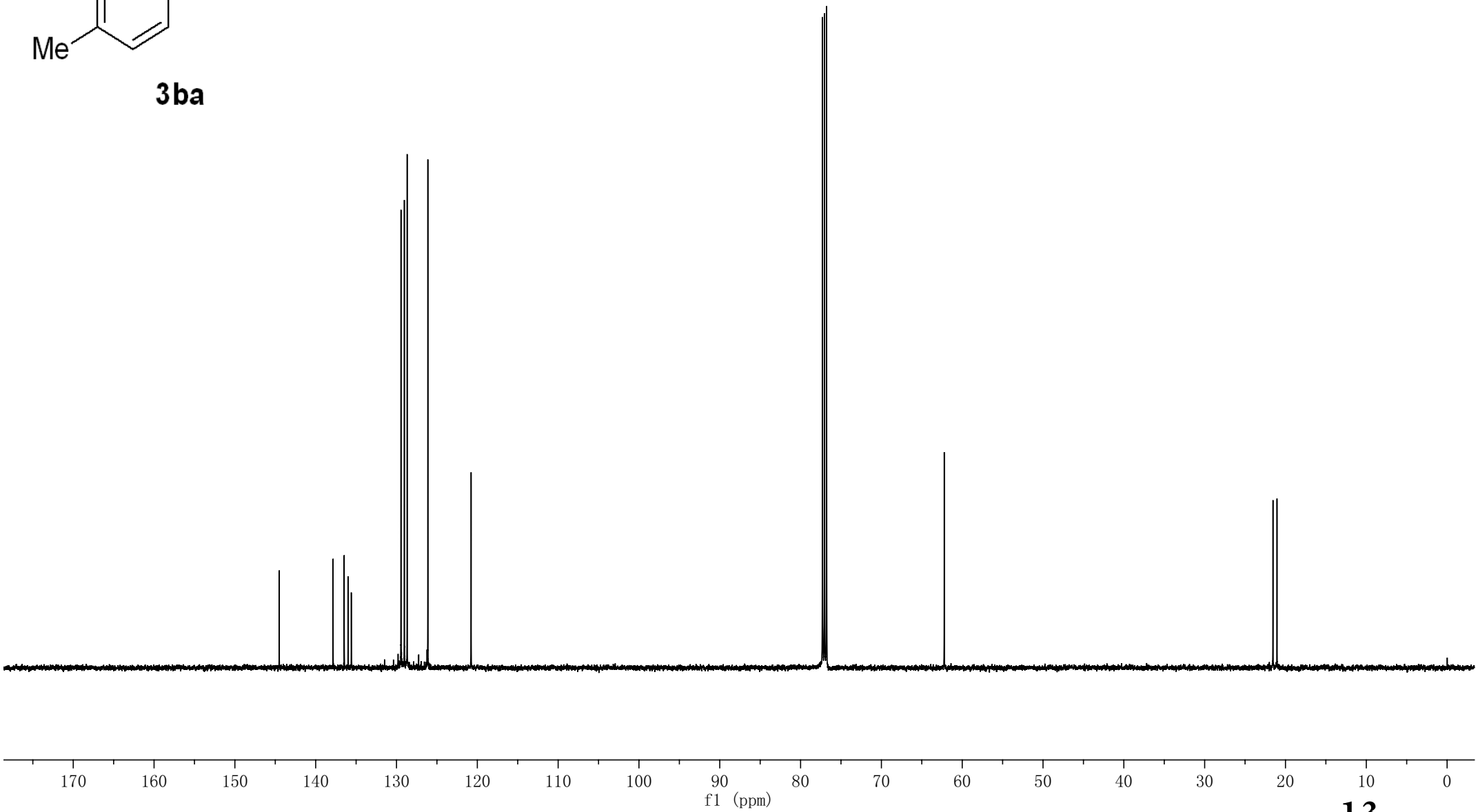
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

120718
LXQ146-4 CDCl3

144.5092
137.8482
136.4724
135.9915
135.5938
129.4474
129.0307
128.6829
126.1219
120.7670
77.2880
77.0337
76.7795
62.2259
21.5453
21.0457

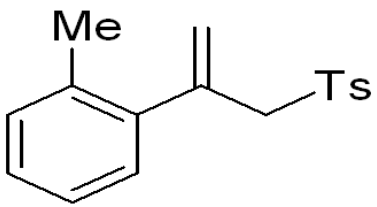


3ba



120726
lxq146-12 CDCl3

7.6528
7.6363
7.2239
7.2078
7.1094
7.1072
7.0951
7.0924
7.0742
7.0609
7.0393
7.0241
7.0222
6.9497
6.9478
6.9345
6.9327
5.4797
5.4782
5.2697
5.2677
4.1299
2.3812
2.2260



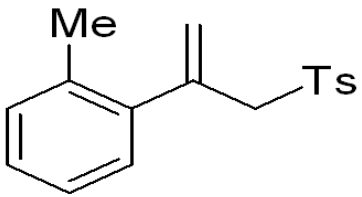
3ca

1.96
1.99
3.03
0.99
0.98
0.98
2.02
3.00
2.96

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)

120726
lxq146-12 CDCl3

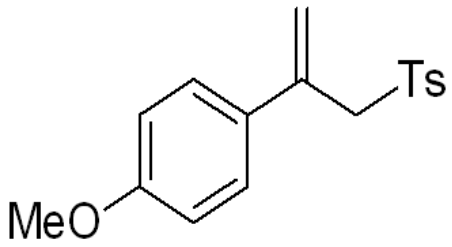
144.3708
139.9603
136.8314
136.0786
134.5466
130.1811
129.4585
128.5768
128.1963
127.4926
125.5050
124.0429
77.2865
77.0320
76.7774
63.2105
21.4431
19.9162



3ca

160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

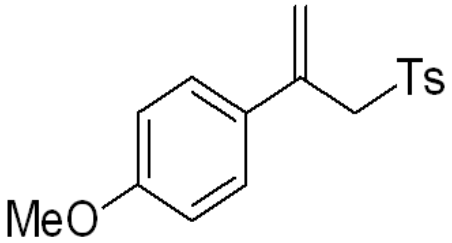
121024
LXQ146-22 CDCl3



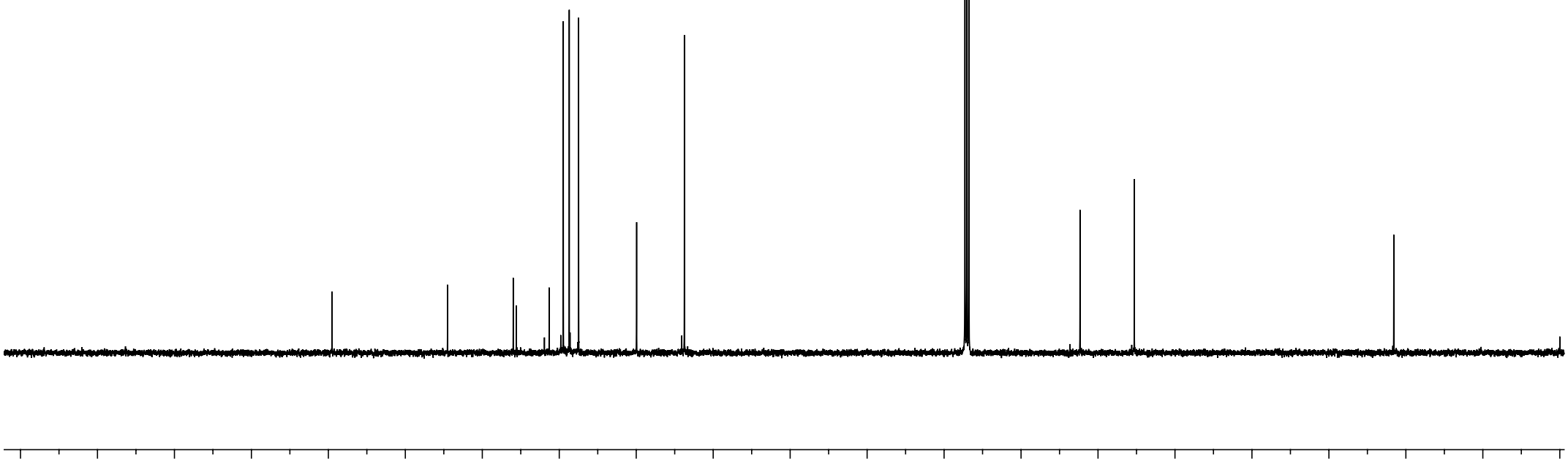
3da

121024
LXQ146-22 CDCl3

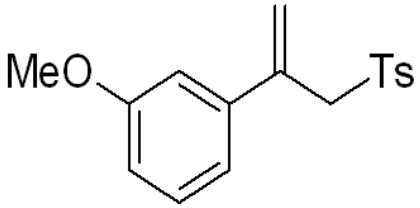
159.5212
144.5183
135.9817
135.5818
131.3127
129.4735
128.7011
127.4821
119.9464
113.7448
77.2871
77.0331
76.7791
62.3407
55.2891
21.5711



3da



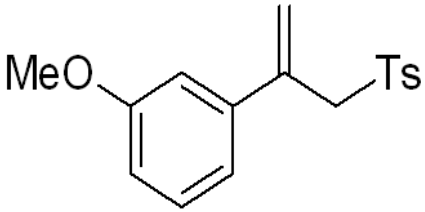
120724
lxq146-8 CDCl3



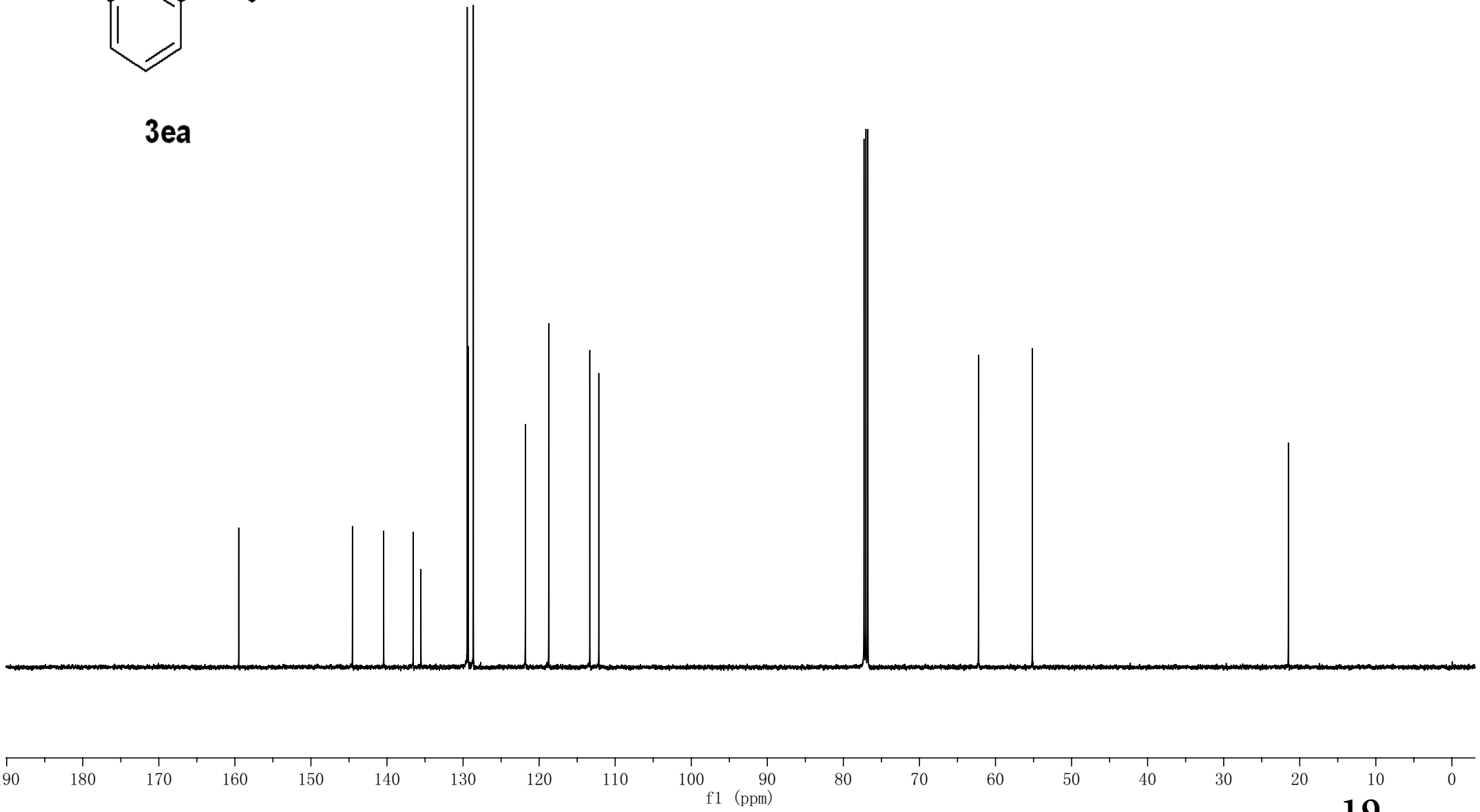
3ea

120724
1xq146-8 CDCl3

159.4940
144.5474
140.4329
136.5658
135.5436
129.4483
129.3102
128.6478
121.8018
118.7400
113.3344
112.1502
77.2863
77.0318
76.7776
62.2401
55.1608
21.4993

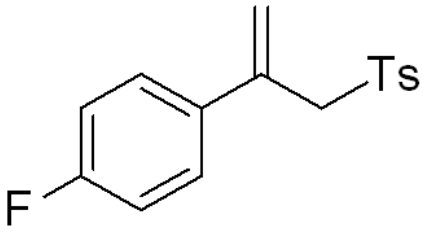


3ea



120827
LXQ146-19 CDCl3

7.6614
7.6449
7.2533
7.2425
7.2256
6.9425
6.9383
6.9289
6.9252
6.9214
6.9121
6.9079
5.5242
5.1698
4.2115
2.3963



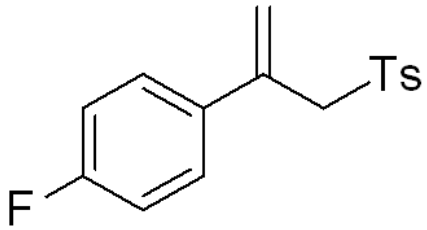
3fa

1.88
3.98
1.89
1.00
1.00
2.01
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)

120827
LXQ146-19 CDCl3

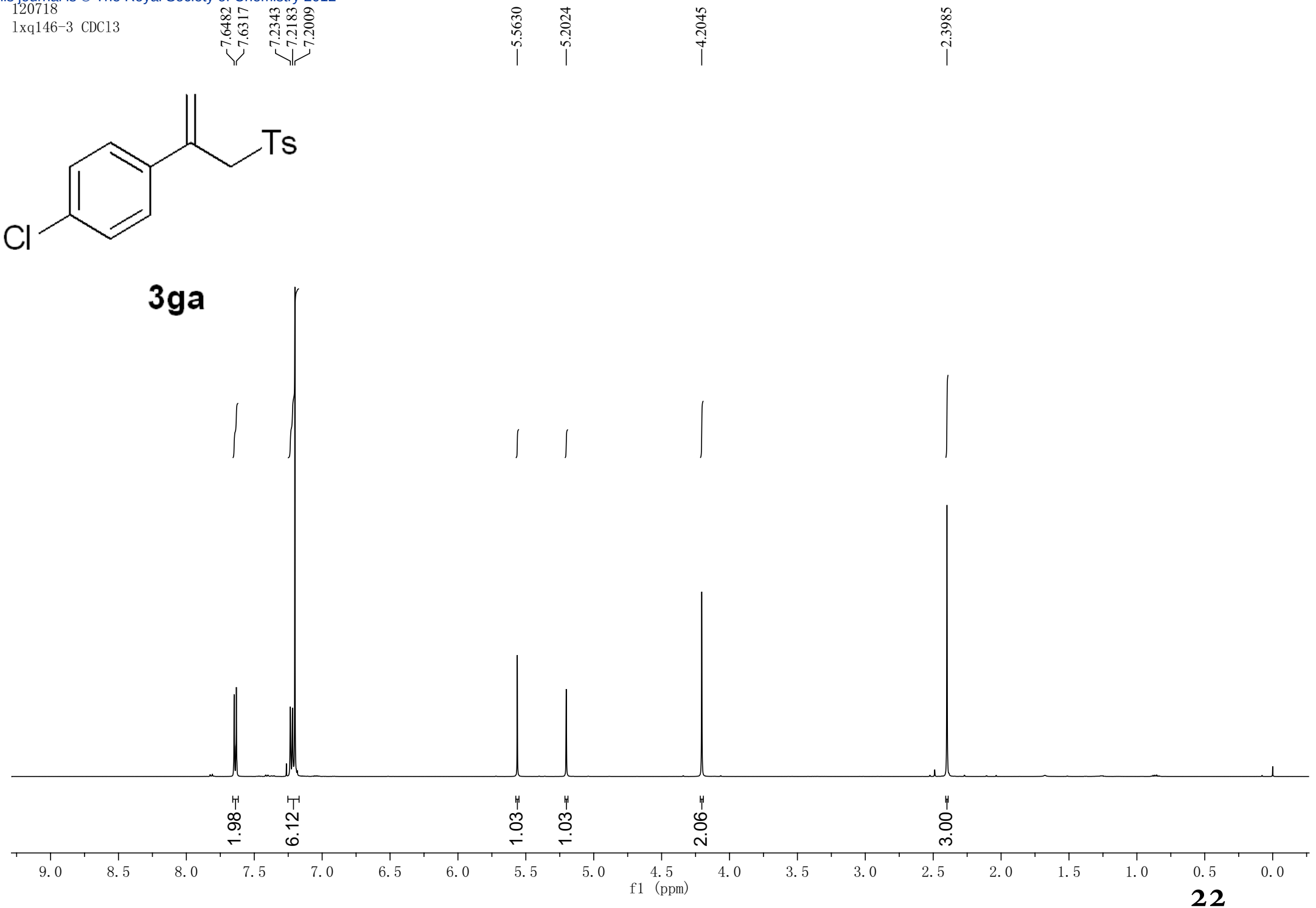
163.5205
161.5505
144.7260
135.6988
135.5013
135.0520
135.0261
129.5350
128.6066
128.0790
128.0144
121.6621
115.2919
115.1196
77.2885
77.0344
76.7801
62.3000
21.5167



3fa

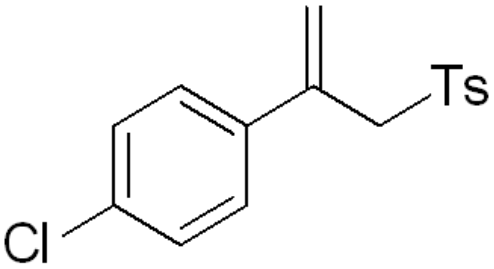
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

120718
1xq146-3 CDCl3

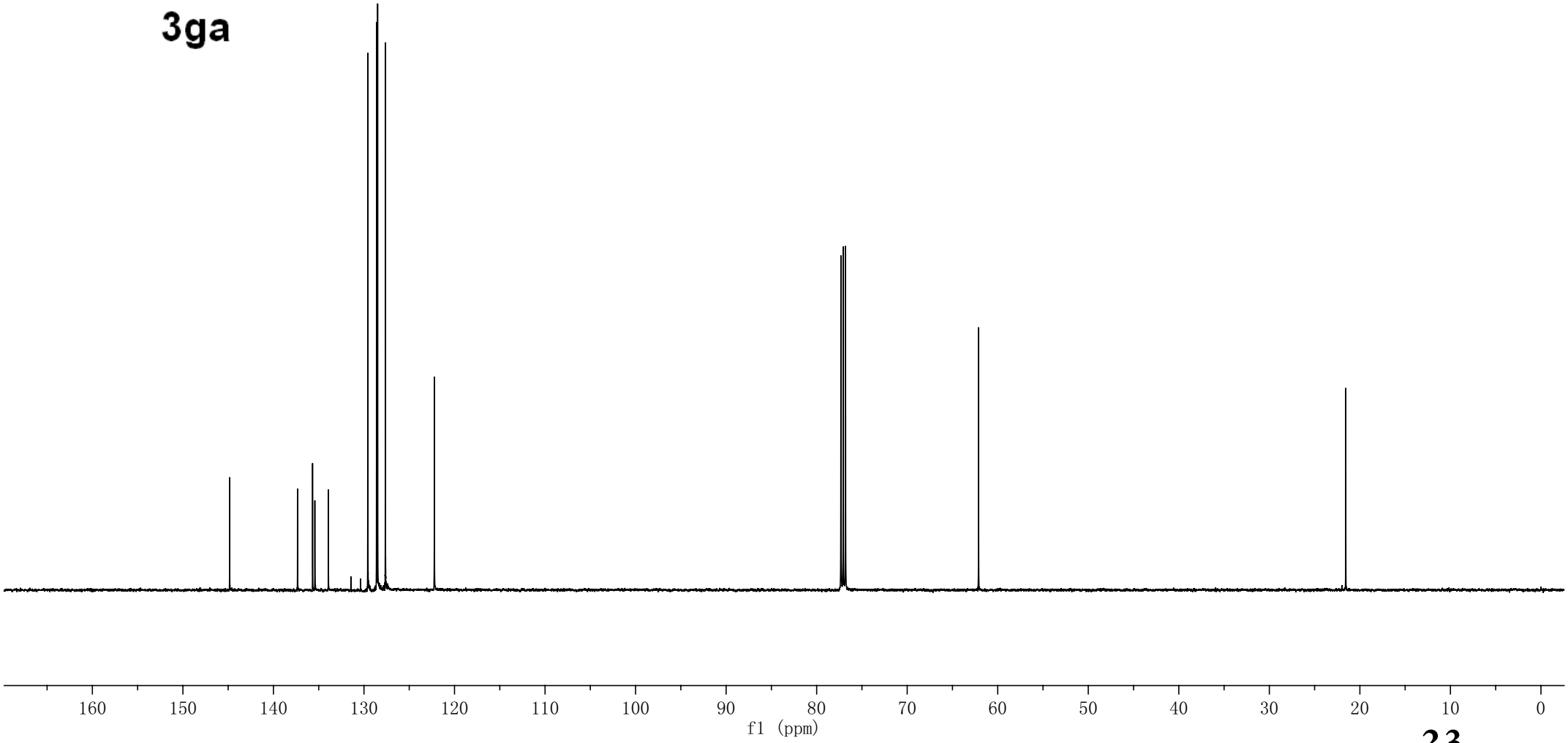


120718
1xq146-3 CDCl3

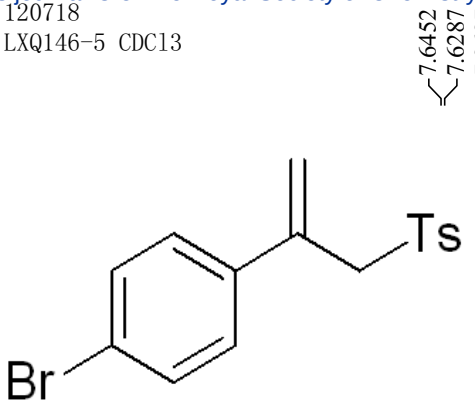
144.8361
137.3388
135.6770
135.4246
133.9301
129.5779
128.6131
128.4890
127.6365
122.2218
77.3199
77.0654
76.8112
62.1174
21.5568



3ga



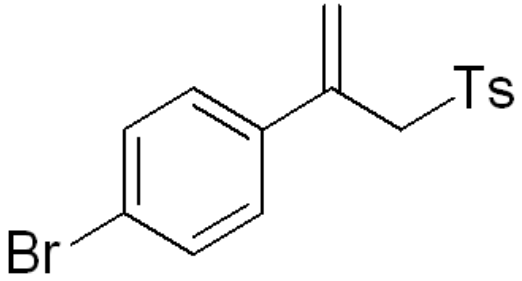
120718
LXQ146-5 CDCl3



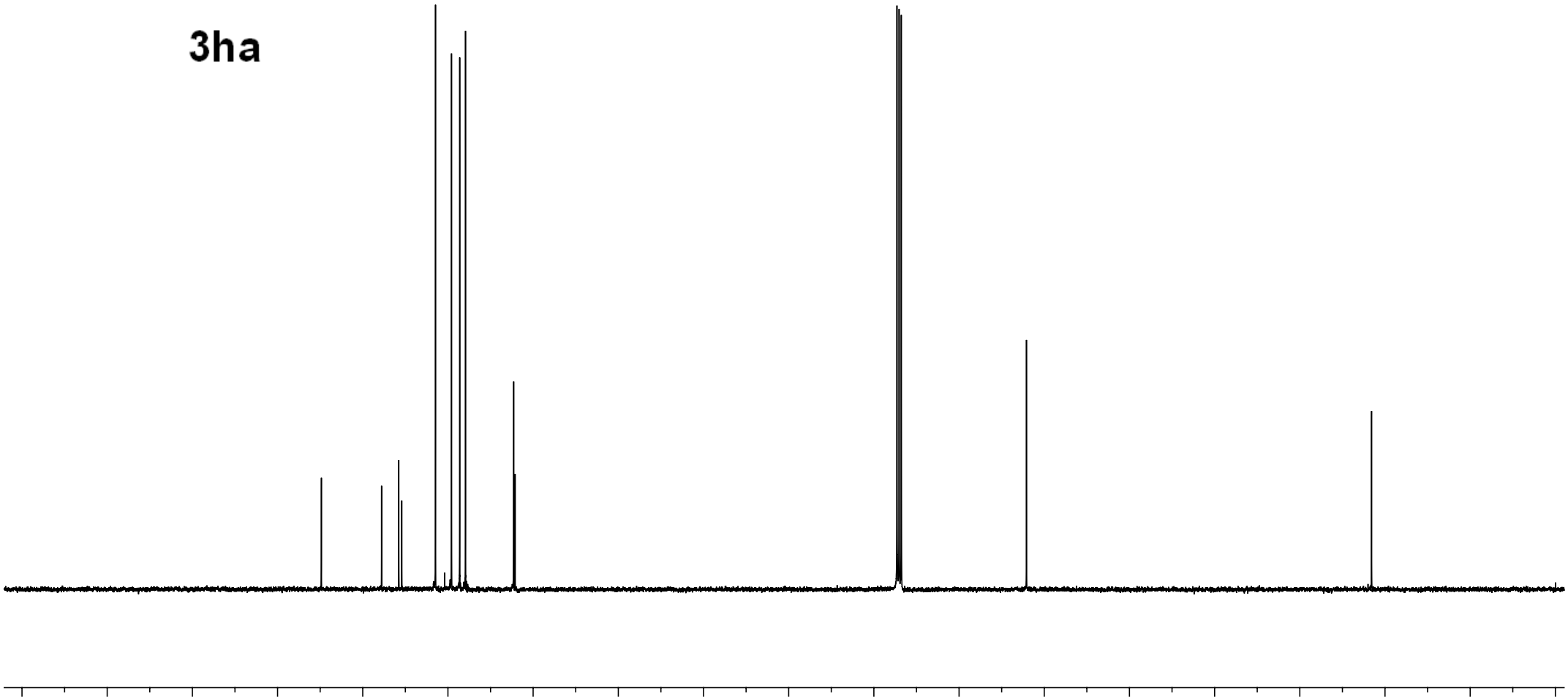
3ha

120718
LXQ146-5 CDCl3

144.8533
137.7933
135.7695
135.4206
131.4590
129.5789
128.6173
127.9373
122.2800
122.1104
77.2889
77.0348
76.7804
62.0894
21.5776



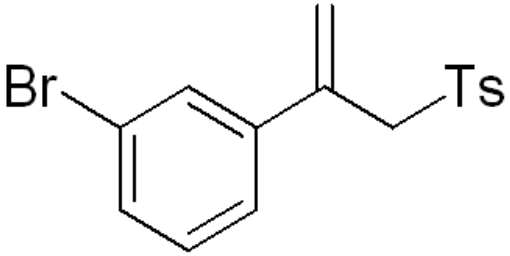
3ha



180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

120827
LXQ146-20 CDCl3

7.6326
7.6161
7.3113
7.3096
7.3078
7.2334
7.2298
7.2262
7.2153
7.2126
7.2067
7.1993
7.1947
7.1083
7.0926
7.0769
5.3725
5.2863
4.1990
2.3834



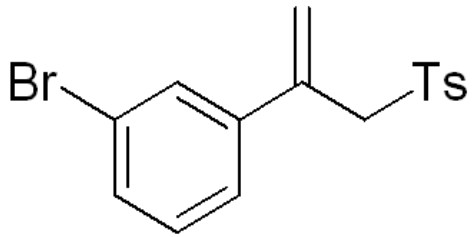
3ia

1.92
0.94
3.93
1.08
1.00
1.00
2.02
3.06

f1 (ppm)

120827
LXQ146-20 CDCl3

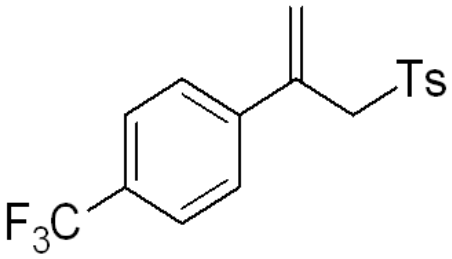
144.7983
140.9350
135.5035
135.2284
130.6863
129.7780
129.5157
129.2795
128.5173
124.8550
122.8887
122.3948
77.2843
77.0297
76.7753
61.9301
21.5099



3ia

121023
lxq146-112 CDCl3

7.6336
7.6171
7.4855
7.4690
7.3644
7.3482
7.2613
7.2110
7.1950
5.6647
5.3479
4.2545
2.3837
1.5454
1.2533



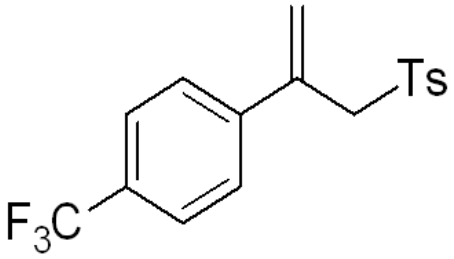
3ja

1.97
1.97
2.05
2.05
1.00
1.00
2.00
3.01

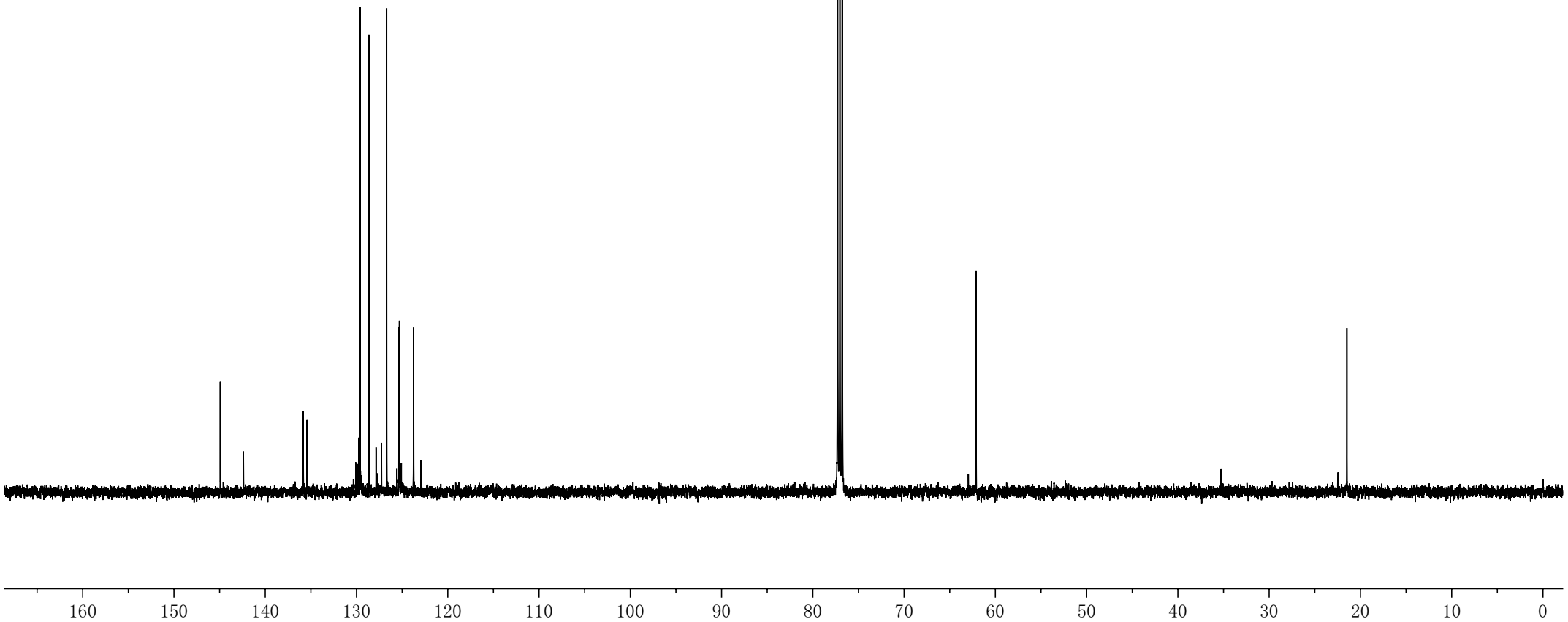
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

120724
lxq146-11 CDCl3

144.9257
142.3904
135.8362
135.4224
130.0837
129.8235
129.7396
129.6165
129.4508
128.6388
127.8588
127.2596
126.6937
125.5978
125.5677
125.3617
125.3320
125.3015
125.2721
125.0858
123.7433
122.9240
77.2853
77.0314
76.7773
62.1045
21.4485

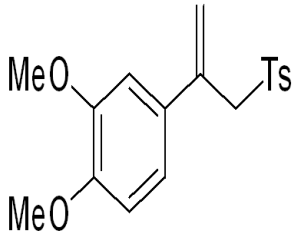


3ja



120827
LXQ146-18 CDCl3

7.6369
7.6204
7.1968
7.1807
6.8239
6.8196
6.8073
6.8030
6.7863
6.7820
6.7058
6.6892
5.4839
5.0888
4.2041
3.8150
3.7983
2.3533



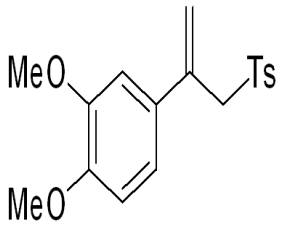
3ka

1.97
2.02
2.06
1.02
1.01
1.01
1.99
6.08
3.00

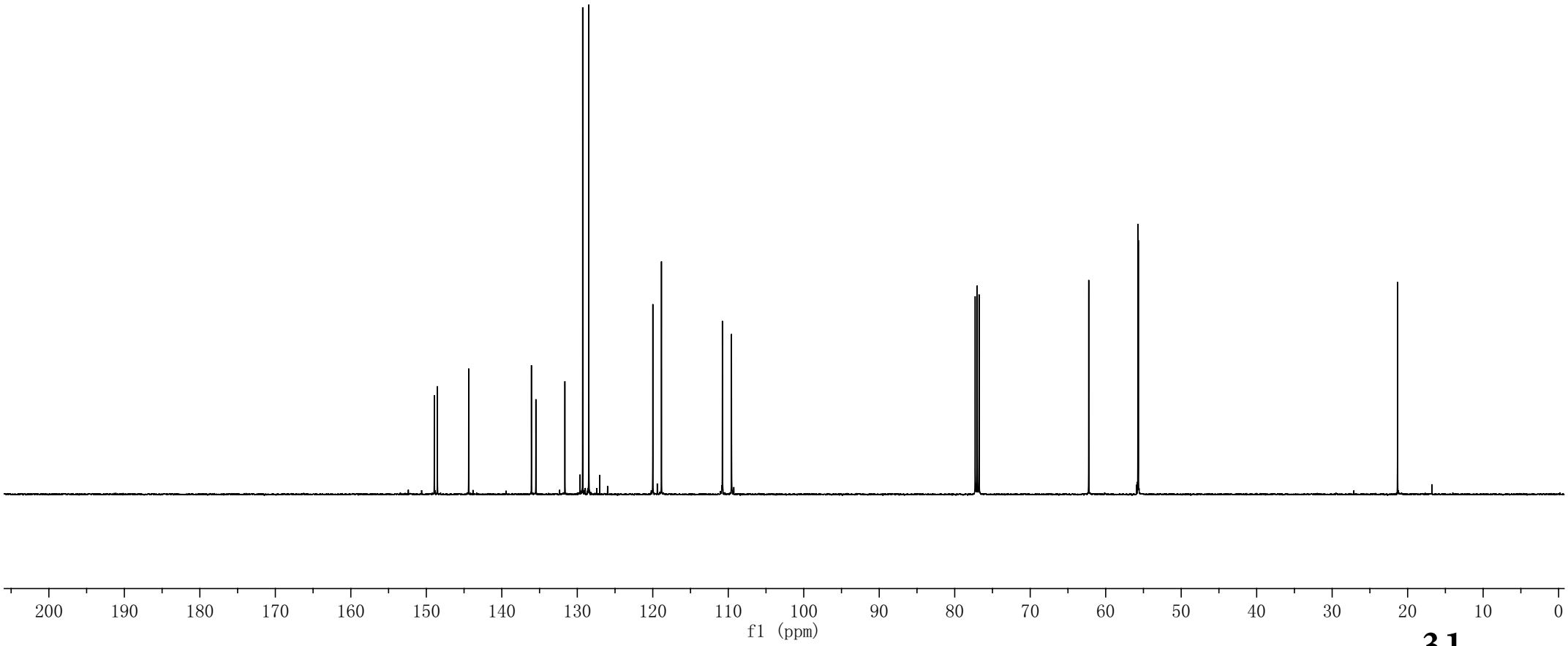
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

120827
LXQ146-18 CDCl3

148.9444
148.5361
144.3870
136.0772
135.4887
131.6417
129.2866
128.4717
119.9903
118.8516
110.7778
109.5759
77.2863
77.0310
76.7762
62.2461
55.7376
55.6689
21.3349

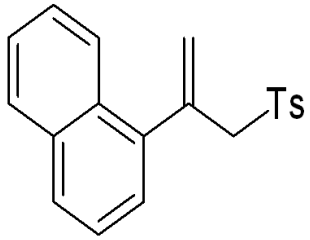


3ka

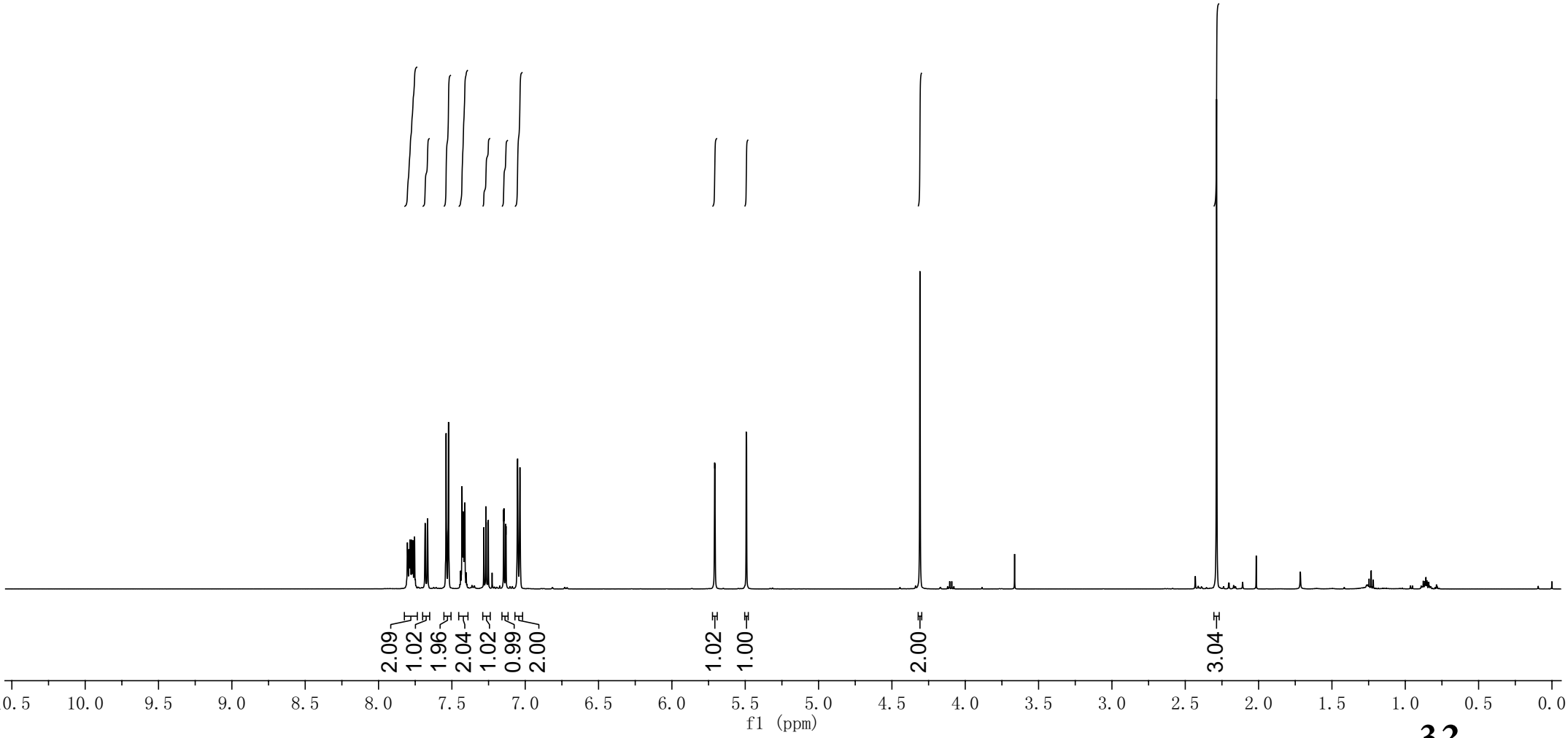


120726
1xq146-62 CDCl3

7.8032 7.7838 7.7736 7.7646 7.7545 7.6823 7.6658 7.5395 7.5230 7.4310 7.4268 7.4224 7.4205 7.4157 7.4120 7.2823 7.2680 7.2660 7.2517 7.1472 7.1451 7.1331 7.1309 7.0520 7.0361 5.7082 5.7067 5.4931 5.4909 4.3089 2.2863

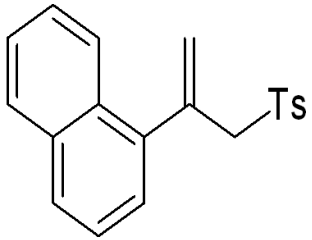


3la

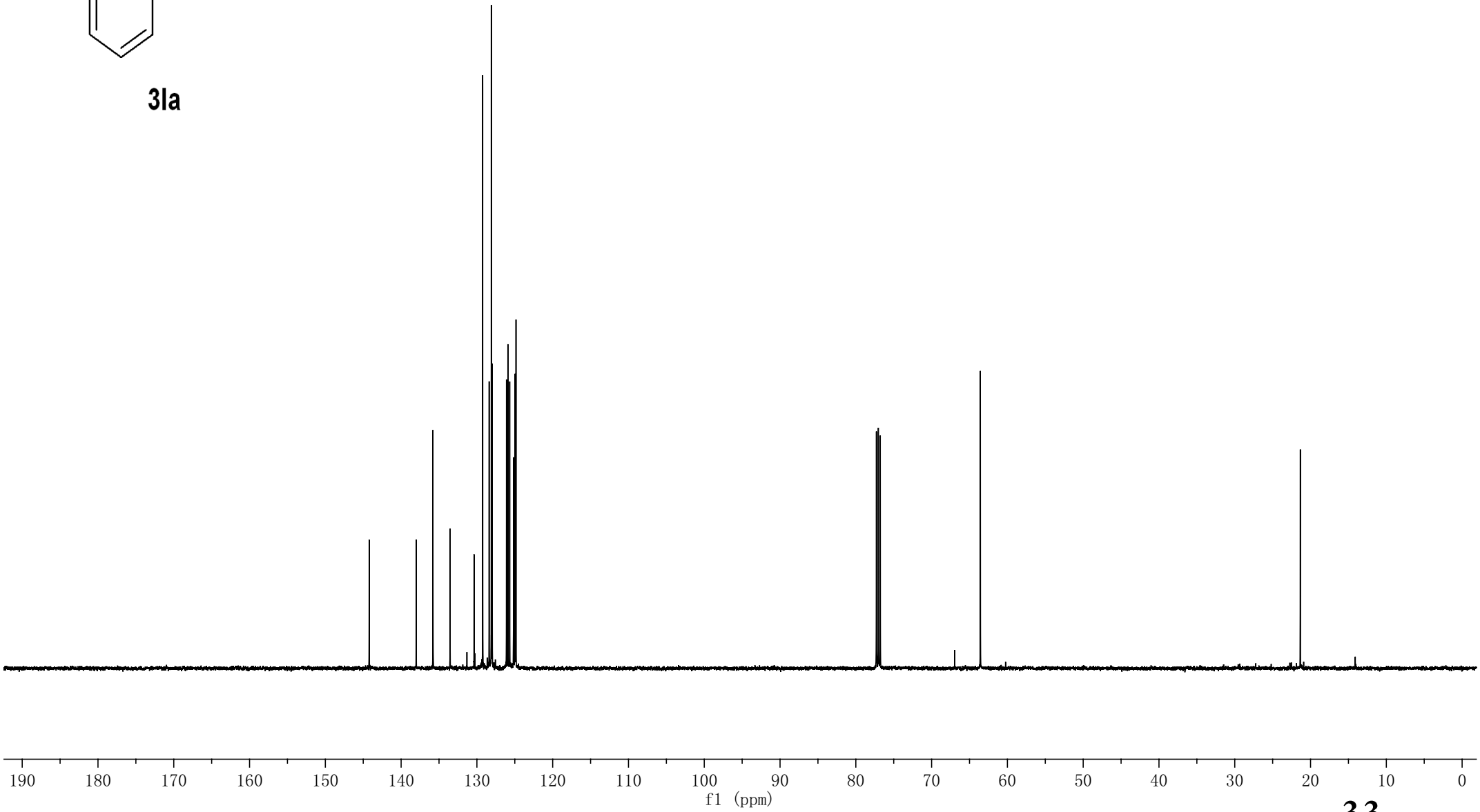


120726
lxq146-62 CDCl3

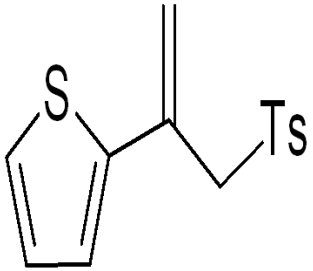
144.2105
138.0128
135.8063
133.5398
130.3755
129.2362
128.3854
128.0998
127.9801
126.0960
125.9107
125.6703
125.1484
124.9808
124.8401
77.2850
77.0305
76.7756
63.5681
21.3514



3la



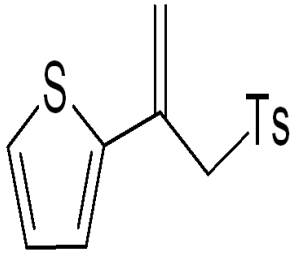
121025
146-103 CDCl3



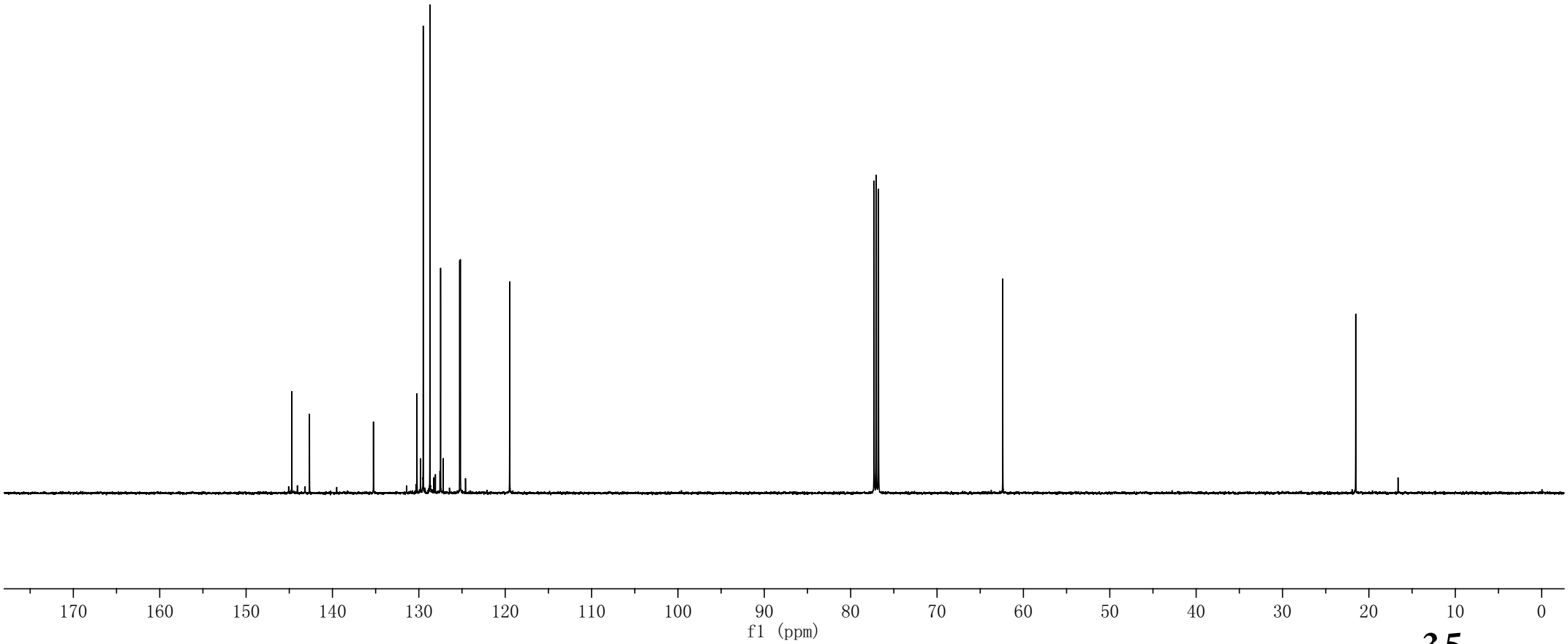
3ma

120724
lxq146-10 CDCl3

144.7054
142.6587
135.2561
129.4915
128.7158
127.4781
125.2690
125.1802
119.4808
77.2858
77.0315
76.7771
62.3925
21.5258

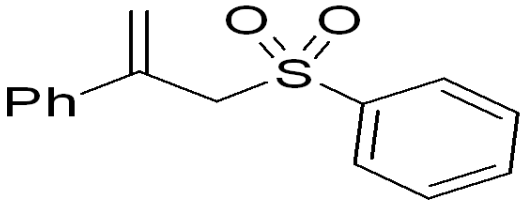


3ma

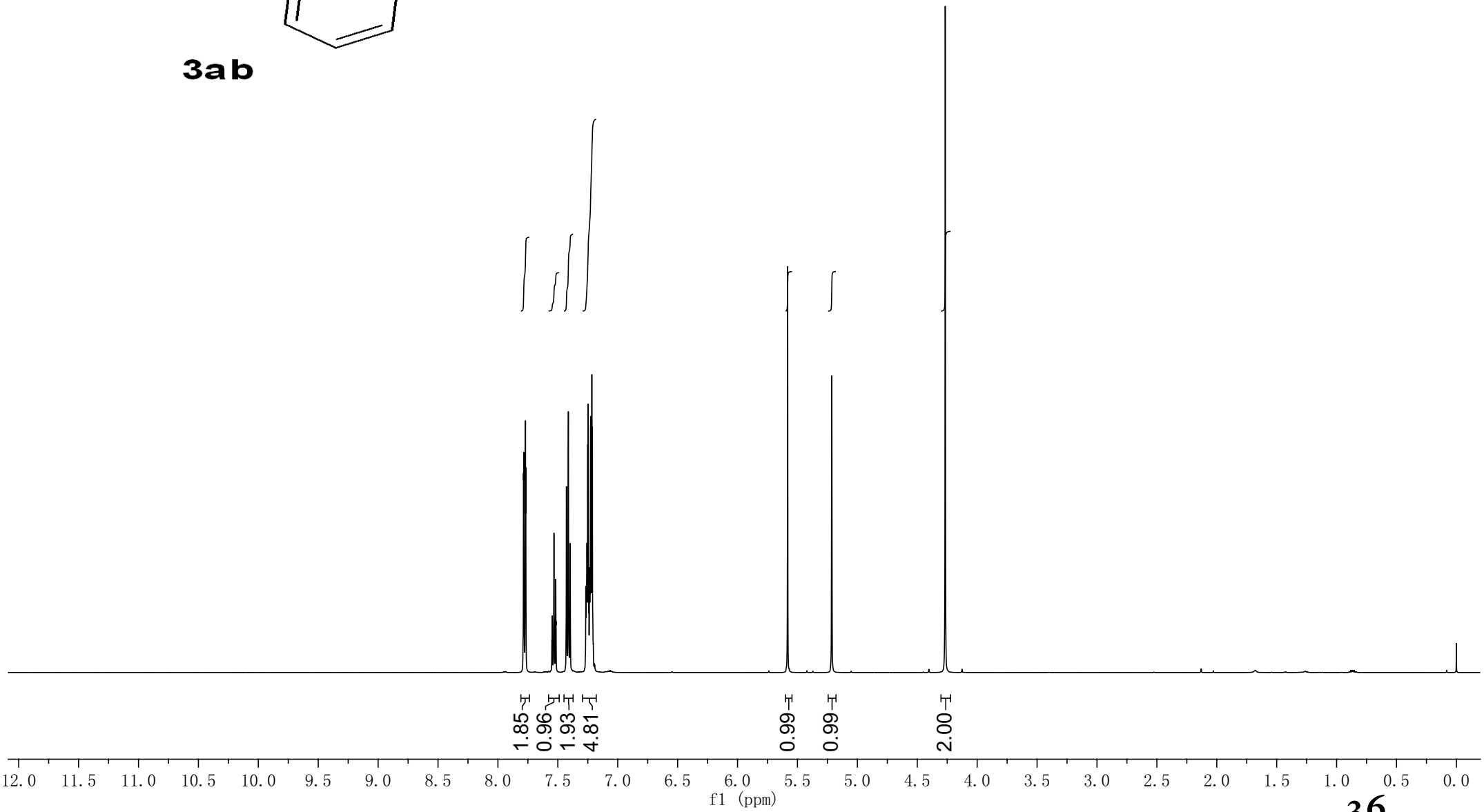


120727
LXQ148 CDC13

7.7864 7.7845 7.7737 7.7701 7.7675 7.5493 7.5471 7.5448 7.5322 7.5195 7.5173 7.5150 7.4281 7.4250 7.4121 7.4002 7.3969 7.2665 7.2640 7.2614 7.2567 7.2503 7.2463 7.2410 7.2344 7.2313 7.2260 7.2218 7.2160 7.2117 7.2042 5.5820 5.2137 4.2664

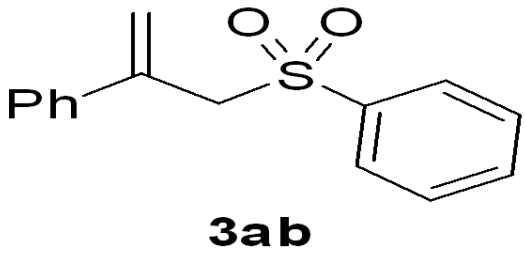


3ab



120727
LXQ148 CDC13

138.7415
138.4471
136.4632
133.5541
128.8286
128.5664
128.3334
127.9780
126.1411
121.7474
77.2890
77.0346
76.7800
62.0052

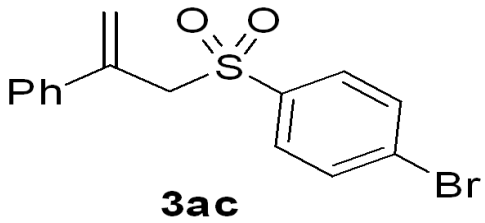


3ab

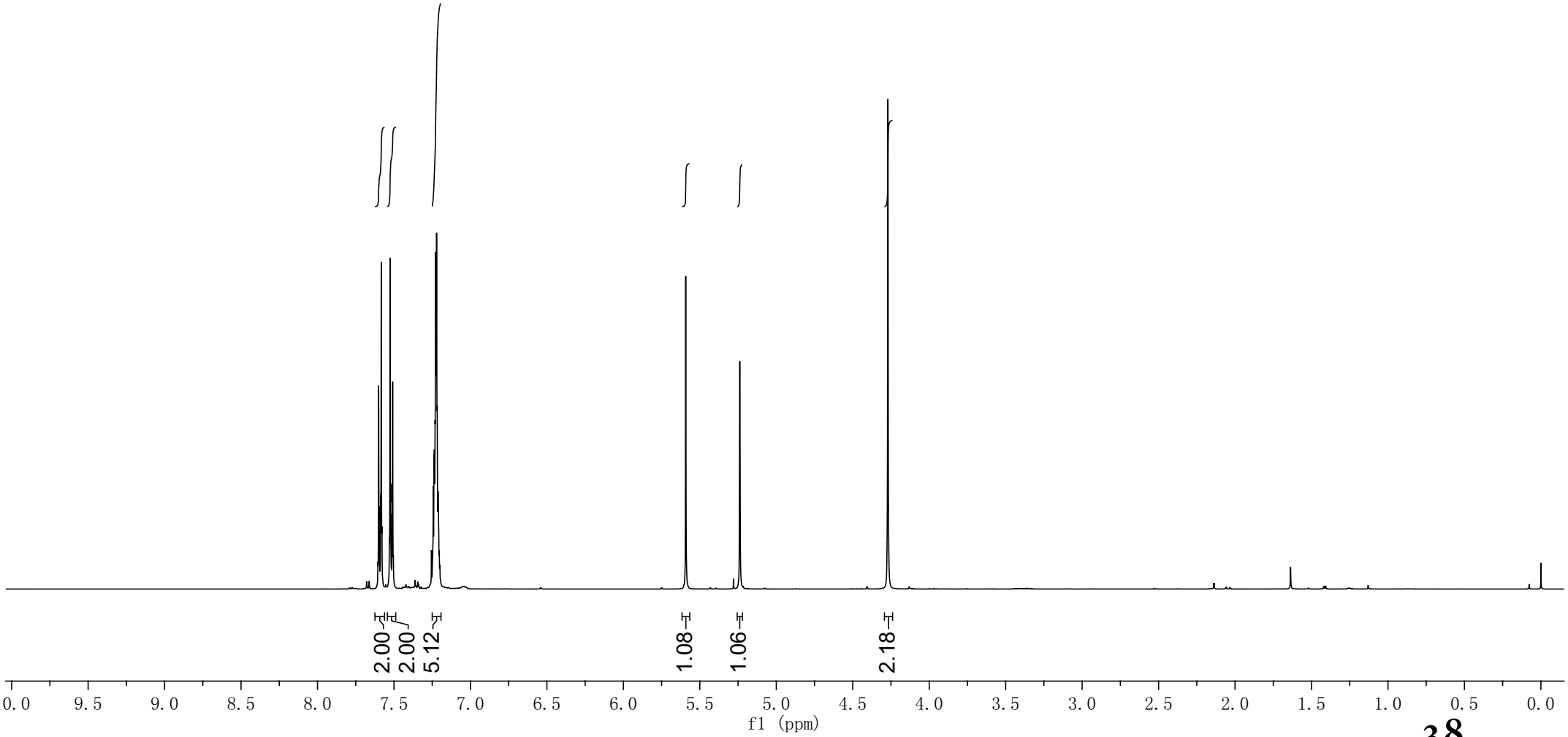
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

120730
1xq146-149 CDCl3

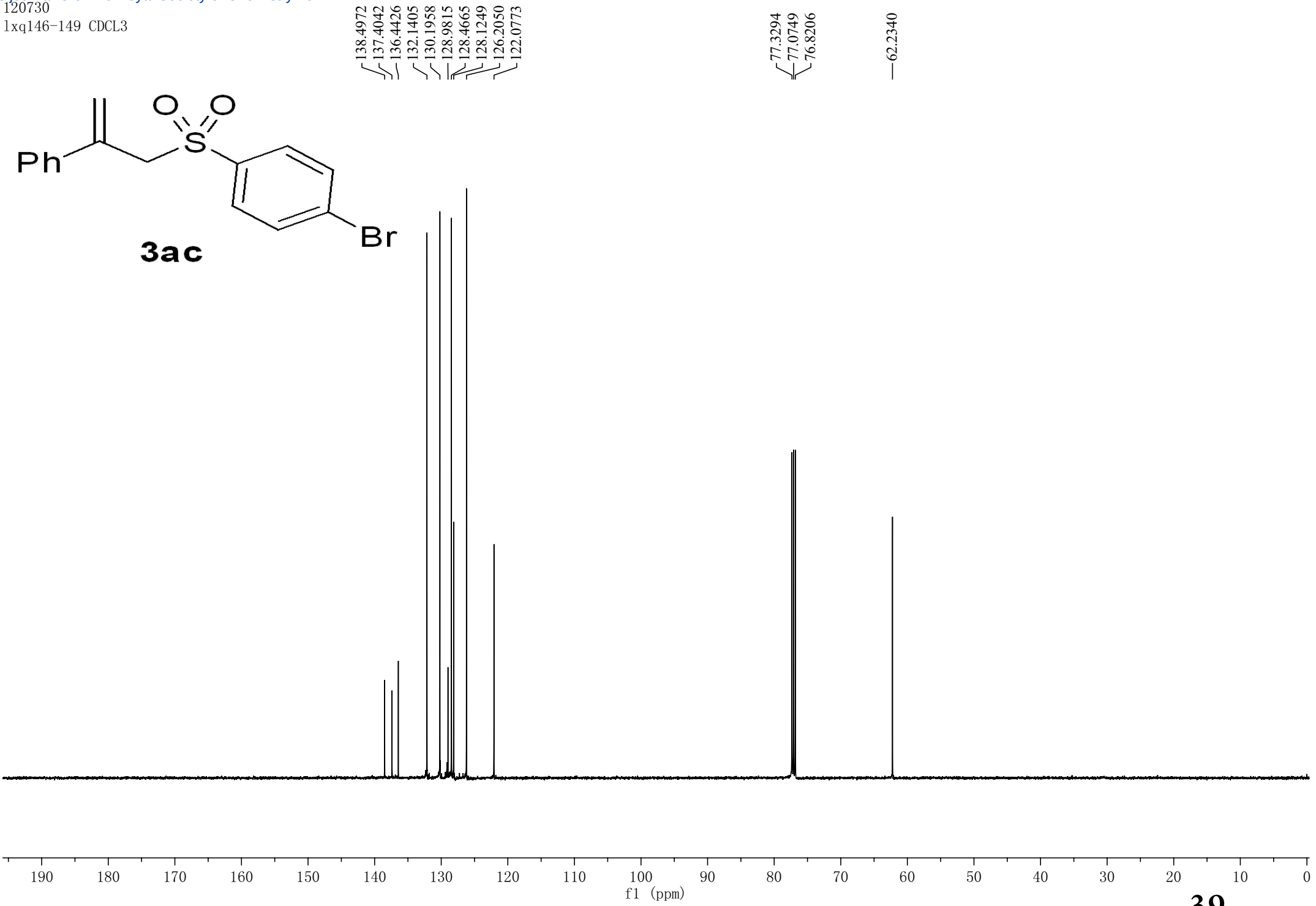
7.6049 7.6004 7.5967 7.5871 7.5831 7.5789 7.5300 7.5257 7.5218 7.5122 7.5084 7.5040 7.2548 7.2431 7.2380 7.2360 7.2310 7.2274 7.2213 7.2176 7.2115 7.2099 7.2041 7.2003 5.5921 5.2386 4.2702



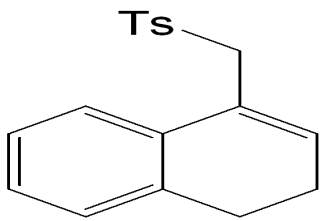
3ac



120730
lxq146-149 CDCL3



120726
lxq146-72 CDCl3



3na

7.6746 7.6582 7.2099 7.1938 7.1306 7.1159 7.0914 7.0788 7.0756 7.0648 7.0538 7.0475 7.0067 5.8974 5.8881 4.1888 2.6950 2.6791 2.6628 2.3639 2.3527 2.2335 2.2238 2.2171 2.2078 2.2018 2.1920

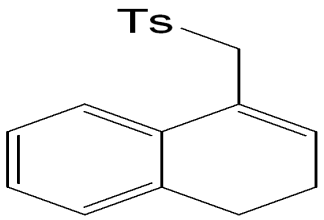
1.95 2.06 4.05 0.98 2.08 2.03 3.00 2.06

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

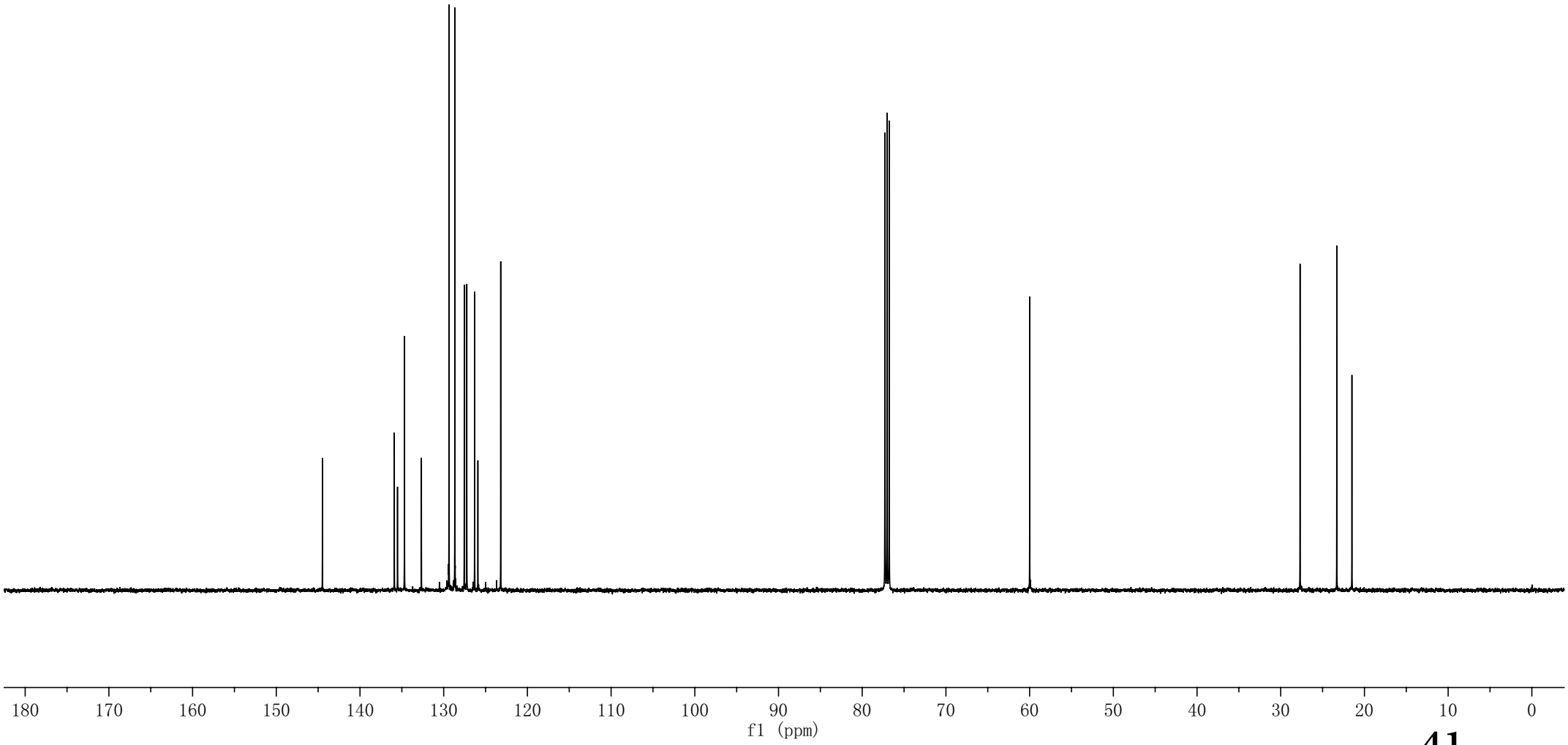
f1 (ppm)

120726
lxq146-72 CDCl3

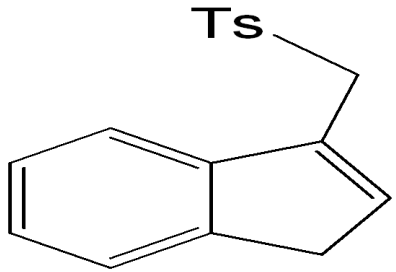
144.4873
135.8956
135.5196
134.6827
132.6605
129.3621
128.6689
127.5411
127.2377
126.3057
125.9157
123.1886
77.2896
77.0351
76.7810
59.9924
27.6921
23.3054
21.4937



3na

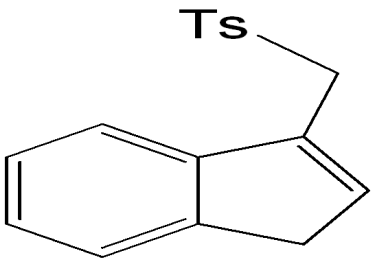


120820
lxq146-16 CDCl3



3oa

120820
lxq146-16 CDCl3



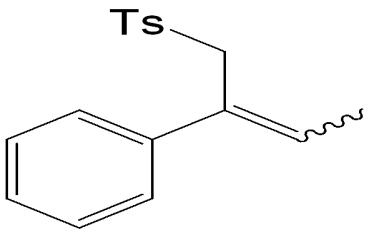
3oa

144.5727
143.2847
143.0668
137.1359
135.4542
131.9217
129.4052
128.4605
126.0818
125.0295
123.6355
119.4557
77.2844
77.0296
76.7752
55.7237
38.2513
21.4442

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

121026
3pa2 CDCl3

7.6301
7.6136
7.2598
7.2203
7.2041
7.1919
7.1833
7.1760
7.1693
7.1611
7.1109
6.0967
6.0826
6.0684
5.8042
5.7902
5.7761
5.7621
4.3442
4.0994
2.3974
2.3683
1.6825
1.6683
1.6448
1.6307
1.5607



3pa

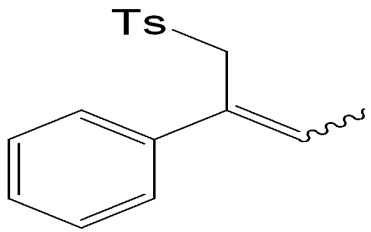
Z/E isomers

2.56
9.52
0.97
0.34
2.00
0.69
1.05
3.14
2.99
1.03

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)

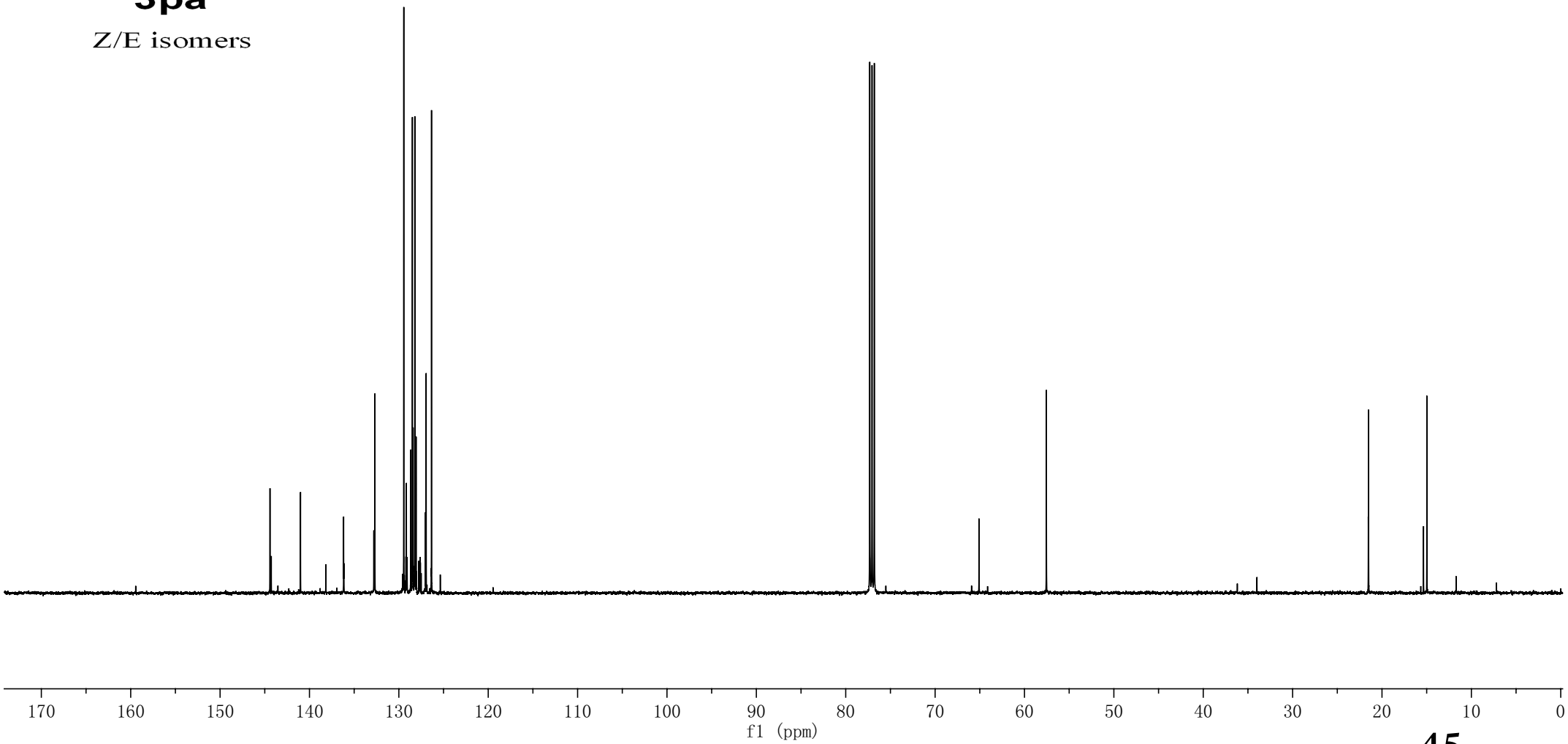
120801
lxq146-132 CDCl3

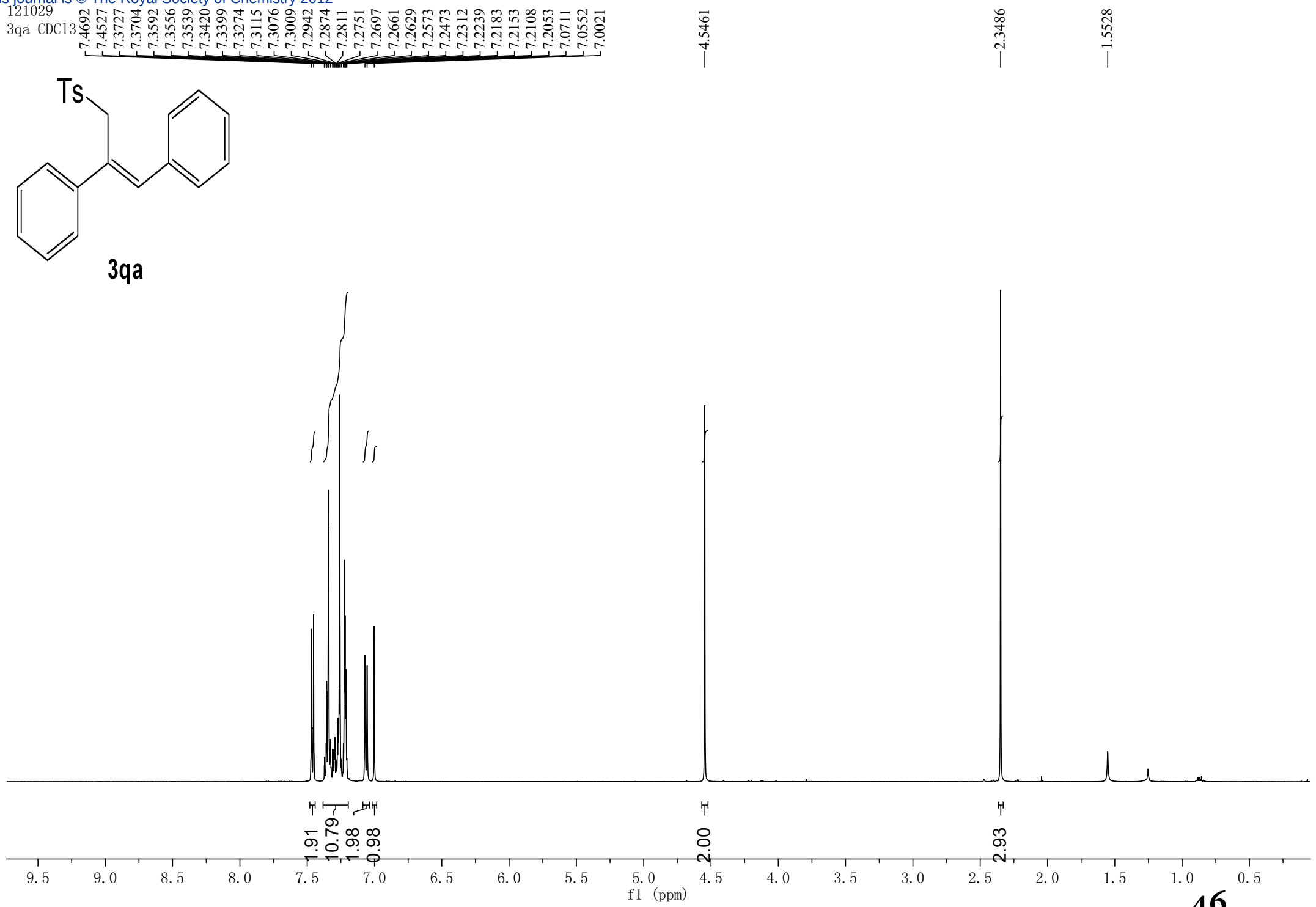
144.4143
141.0011
136.1797
132.7980
132.6848
129.4474
129.1561
128.6665
128.4862
128.4072
128.2135
128.0661
127.0192
126.9434
126.3444
77.3118
77.0575
76.8030
65.0871
57.5519
21.5283
21.5068
15.3538
14.9793



3pa

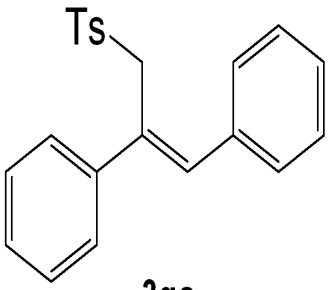
Z/E isomers





120823
lxq146-17 CDCl3

144.2455
140.3773
136.7687
136.5446
136.1084
130.4605
129.3571
128.6280
128.5370
128.3606
128.3094
127.7644
127.5777
126.8068
77.2864
77.0321
76.7781
58.0261
21.5003



3qa