

Tetrabutylammonium Iodide Catalyzed Allylic Sulfonylation of Baylis–Hillman Acetates with Sulfonylhydrazides in Water

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Supporting Information

The characterization data for known compounds-----S2-S4
¹H and ¹³C NMR spectra of compounds **3**-----S5-S44

(Z)-methyl 3-phenyl-2-(tosylmethyl)acrylate (3aa)¹: ¹H NMR (500 MHz, CDCl₃) δ 7.92 (s, 1H), 7.71 (d, *J* = 8.1 Hz, 2H), 7.47 – 7.45 (m, 2H), 7.38 – 7.34 (m, 3H), 7.26 (d, *J* = 7.1 Hz, 2H), 4.47 (s, 2H), 3.61 (s, 3H), 2.42 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.9, 146.1, 144.7, 136.3, 133.7, 129.6, 129.1, 128.7, 128.5, 121.1, 55.1, 52.3, 21.6.

(Z)-methyl 3-(4-chlorophenyl)-2-(tosylmethyl)acrylate (3fa)²: ¹H NMR (500 MHz, CDCl₃) δ 7.86 (s, 1H), 7.72 – 7.68 (m, 2H), 7.46 – 7.41 (m, 2H), 7.36 – 7.30 (m, 2H), 7.29 – 7.24 (m, 2H), 4.42 (s, 2H), 3.59 (s, 3H), 2.42 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.6, 144.8, 144.6, 135.7, 132.1, 130.5, 129.6, 128.9, 128.4, 121.6, 55.0, 52.3, 21.5.

(Z)-ethyl 3-(4-chlorophenyl)-2-(tosylmethyl)acrylate (3ga)³: ¹H NMR (500 MHz, CDCl₃) δ 7.87 (s, 1H), 7.71 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 4.44 (s, 2H), 4.06 (q, *J* = 7.1 Hz, 2H), 2.43 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.2, 144.9, 144.4, 136.4, 135.7, 132.2, 130.5, 129.7, 128.9, 128.5, 122.0, 61.6, 55.0, 21.6, 14.0.

(Z)-methyl 3-(2-bromophenyl)-2-(tosylmethyl)acrylate (3ha)¹: ¹H NMR (500 MHz, CDCl₃) δ 7.94 (s, 1H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.61 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.55 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.36 – 7.32 (m, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.23 – 7.20 (m, 1H), 4.35 (s, 2H), 3.64 (s, 3H), 2.42 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.3, 144.8, 144.7, 136.3, 134.0, 132.8, 130.6, 130.1, 129.7, 128.4, 127.5, 124.0, 123.0, 54.9, 52.4, 21.5.

(Z)-ethyl 3-(3-nitrophenyl)-2-(tosylmethyl)acrylate (3ka)³: ¹H NMR (500 MHz, CDCl₃) δ 8.21 (d, *J* = 6.5 Hz, 2H), 7.94 (s, 1H), 7.89 (d, *J* = 7.8 Hz, 1H), 7.72 (d, *J* = 8.2 Hz, 2H), 7.62 – 7.55 (m, 1H), 7.28 (d, *J* = 7.8 Hz, 2H), 4.43 (s, 2H), 4.16 (q, *J* =

7.1 Hz, 2H), 2.41 (s, 3H), 1.29 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 165.7, 148.3, 145.2, 142.6, 136.1, 135.4, 134.6, 129.9, 129.8, 128.5, 124.3, 123.9, 123.8, 62.0, 54.6, 21.5, 14.0.

(E)-3-phenyl-2-(tosylmethyl)acrylonitrile (3la)²: ^1H NMR (500 MHz, CDCl_3) δ 7.81 – 7.77 (m, 2H), 7.72 – 7.68 (m, 2H), 7.47 – 7.36 (m, 5H), 7.10 (s, 1H), 4.03 (s, 2H), 2.46 (d, $J = 5.3$ Hz, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 151.7, 145.8, 134.7, 132.6, 131.5, 130.2, 129.3, 129.0, 128.8, 117.1, 98.3, 61.4, 21.7.

(2Z,4E)-methyl 5-phenyl-2-(tosylmethyl)penta-2,4-dienoate (3ma)¹: ^1H NMR (500 MHz, CDCl_3) δ 7.78 – 7.74 (m, 2H), 7.59 (d, $J = 11.6$ Hz, 1H), 7.40 – 7.25 (m, 7H) 6.91 (d, $J = 15.3$ Hz, 1H), 6.78 (m, 1H), 4.42 (s, 2H), 3.60 (s, 3H), 2.26 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 166.5, 145.2, 145.0, 143.3, 135.7, 135.6, 129.7, 129.6, 128.8, 128.8, 127.6, 122.7, 118.4, 54.7, 52.2, 21.4.

(Z)-methyl 3-(thiophen-2-yl)-2-(tosylmethyl)acrylate (3na)³: ^1H NMR (500 MHz, CDCl_3) δ 8.05 (s, 1H), 7.81 – 7.75 (m, 2H), 7.52 (t, $J = 4.8$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 7.11 (dd, $J = 5.0, 3.8$ Hz, 1H), 4.61 (s, 2H), 3.54 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 166.8, 144.7, 138.1, 136.8, 136.5, 134.0, 130.7, 129.5, 128.7, 127.8, 116.5, 56.1, 52.3, 21.6.

(Z)-methyl 3-phenyl-2-((phenylsulfonyl)methyl)acrylate (3ab)¹: ^1H NMR (500 MHz, CDCl_3) δ 7.94 (s, 1H), 7.85 – 7.83 (m, 2H), 7.63 – 7.56 (m, 1H), 7.50 – 7.46 (m, 4H), 7.39 – 7.34 (m, 3H), 4.49 (s, 2H), 3.59 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ 166.9, 146.3, 139.4, 133.8, 133.7, 129.7, 129.2, 129.0, 128.8, 128.5, 121.0, 55.2, 52.4.

(Z)-methyl 3-(2-bromophenyl)-2-((phenylsulfonyl)methyl) acrylate (3hb)¹: ¹H NMR (500 MHz, CDCl₃) δ 7.97 (s, 1H), 7.87 – 7.82 (m, 2H), 7.65 – 7.49 (m, 5H), 7.36 (td, *J* = 7.6, 0.7 Hz, 1H), 7.23 (td, *J* = 7.8, 1.4 Hz, 1H), 4.36 (s, 2H), 3.63 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 166.3, 145.2, 139.5, 134.1, 133.7, 132.9, 130.7, 130.1, 129.1, 128.4, 127.6, 124.0, 122.9, 55.0, 52.5.

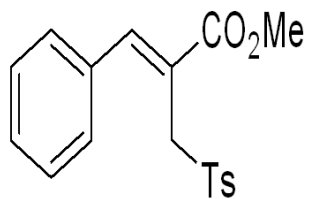
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References

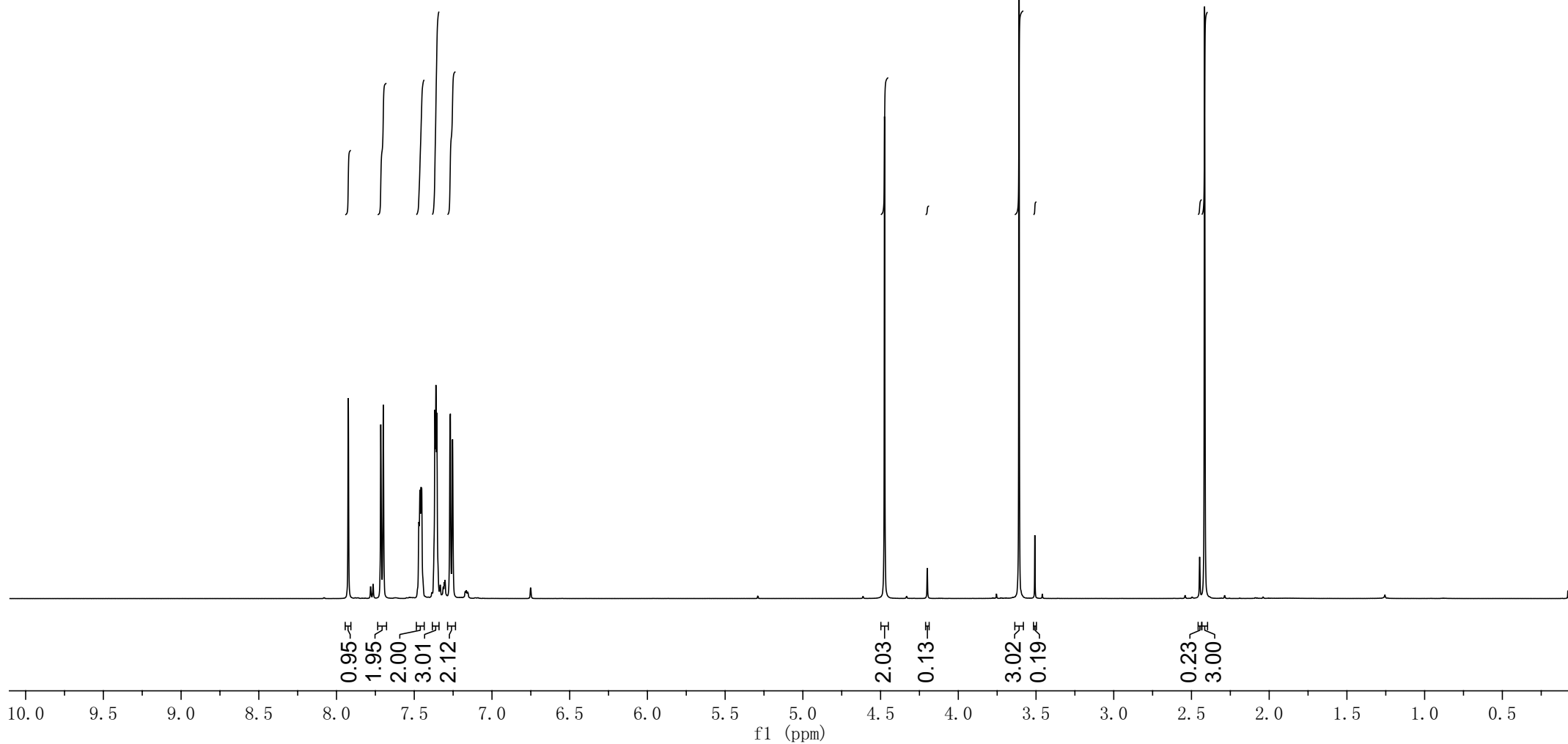
1. L. R. Reddy, B. Hu, M. Prashad, and K. Prasad, *Angew. Chem. Int. Ed.*, 2009, **48**, 172
2. G. W. Kabalka, B. Venkataiaha and G. Dong, *Tetrahedron Lett.*, 2003, **44**, 4673
3. K. Karnakar, J. Shankar, S. N. Murthy, and Y. V. D. Nageswar, *Helv. Chem. Acta*, 2011, **94**, 875

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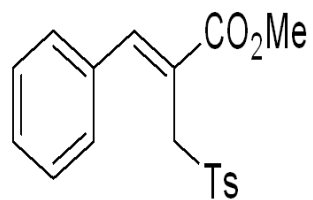
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3aa

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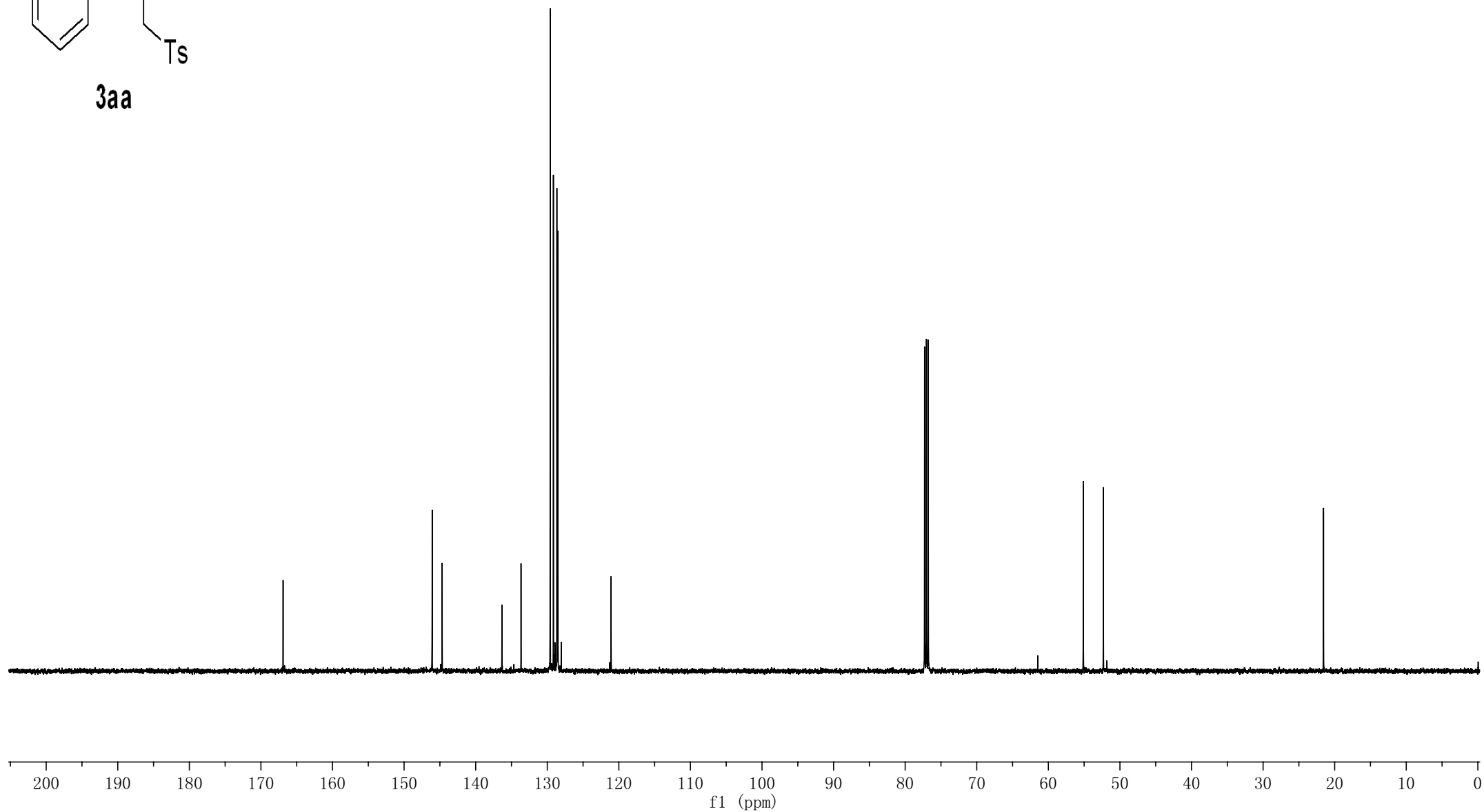
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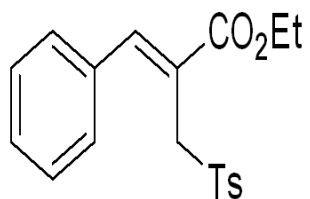
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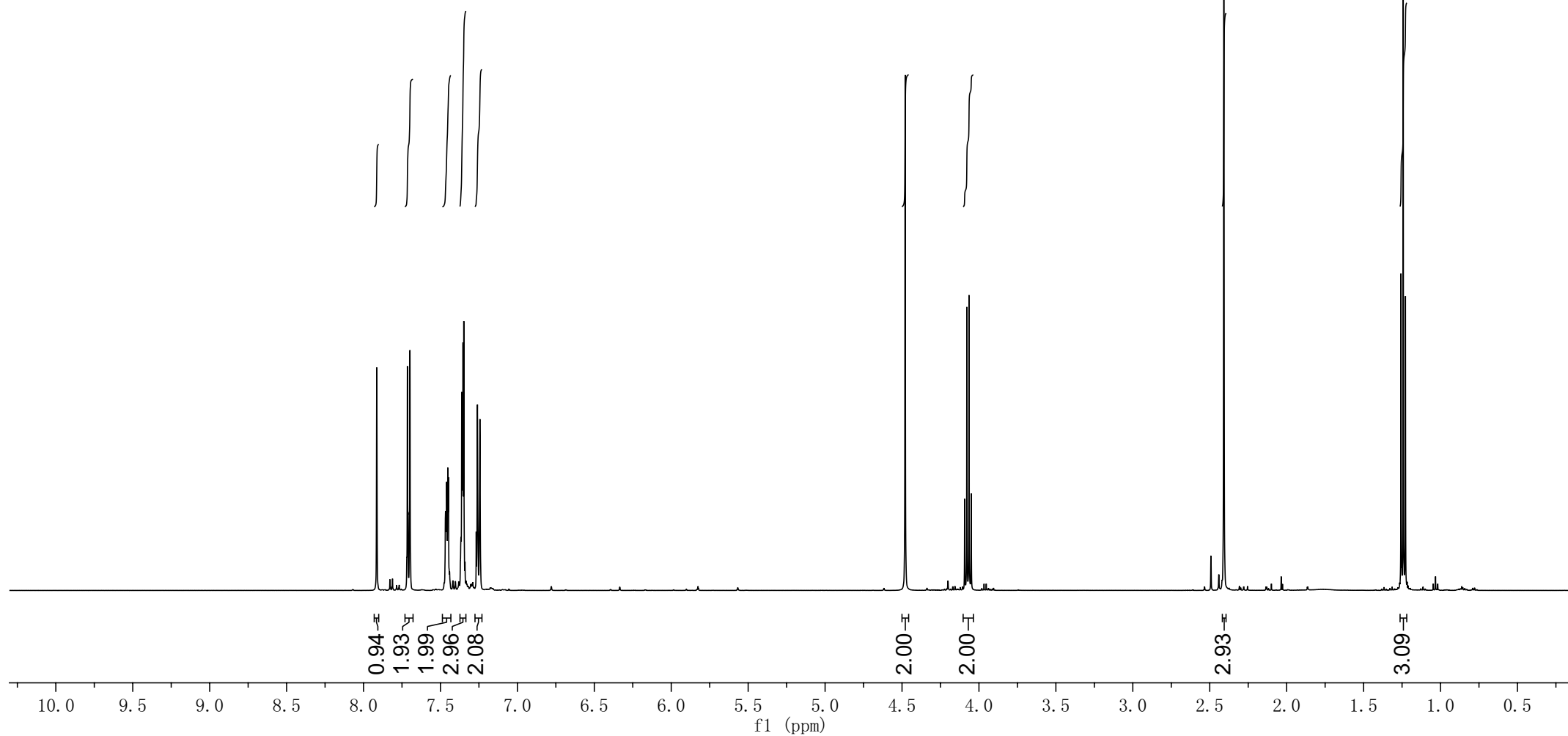
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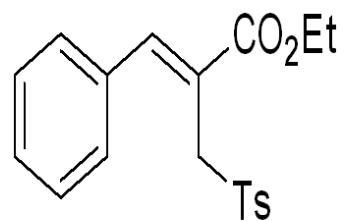
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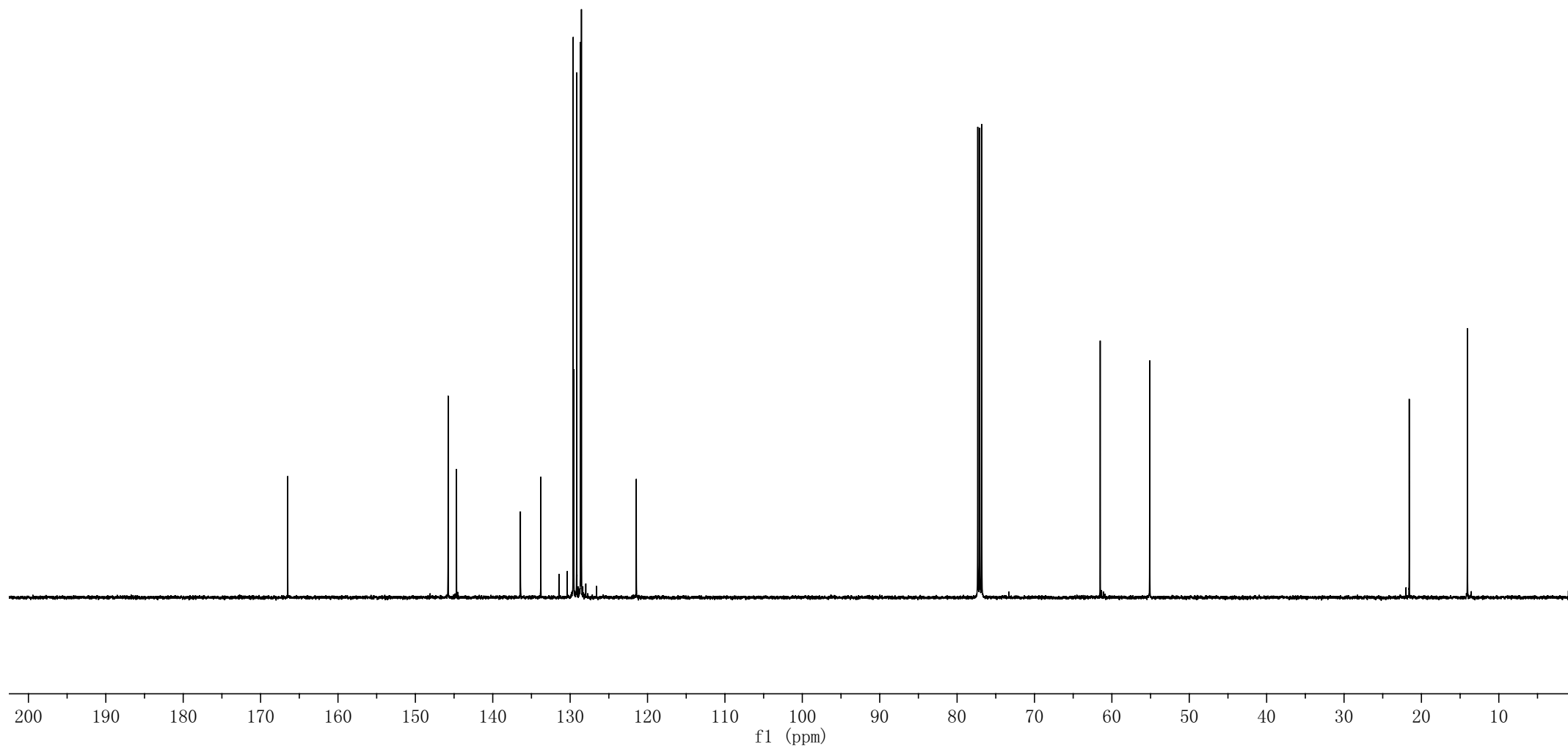


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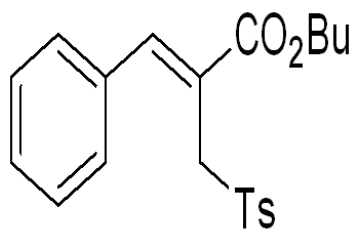


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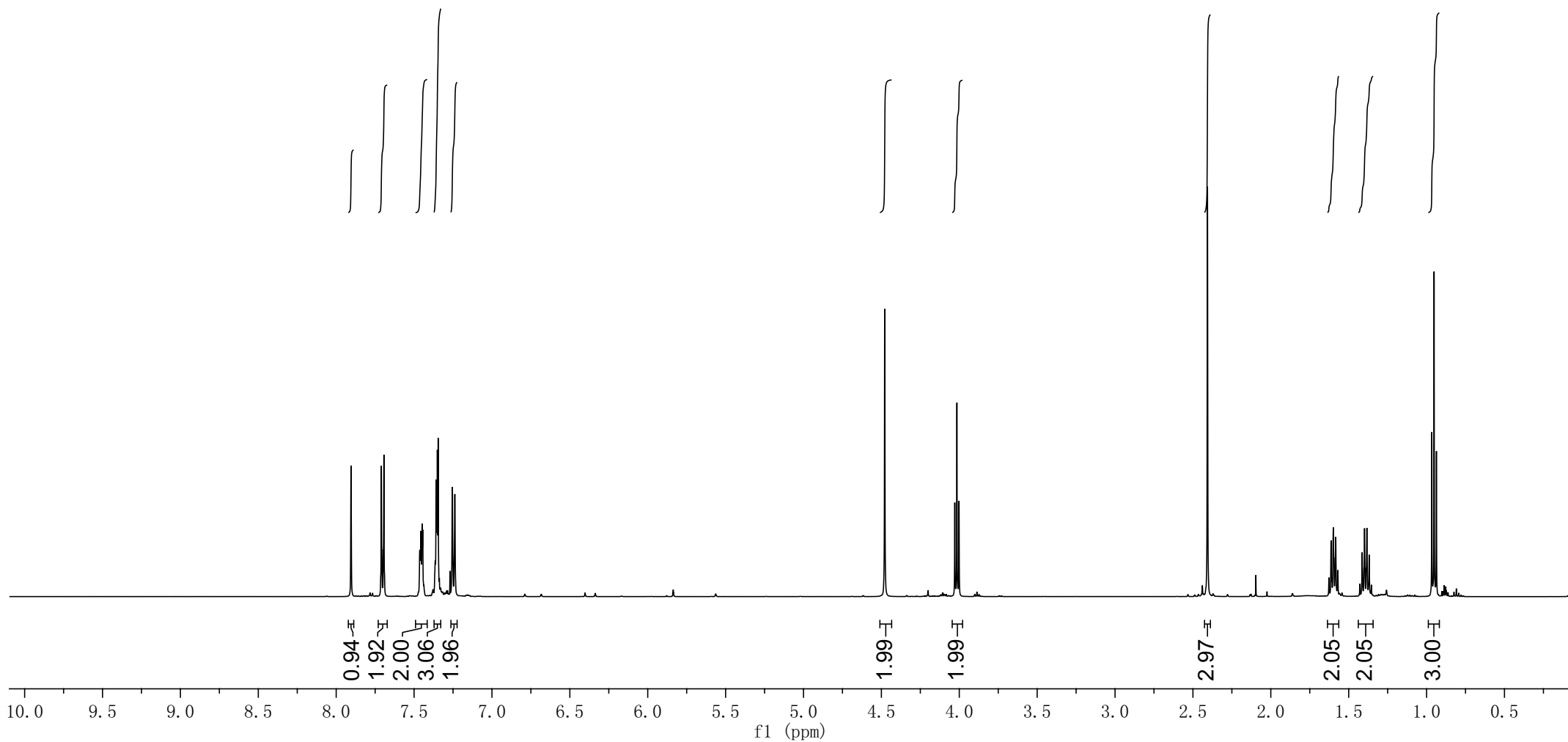
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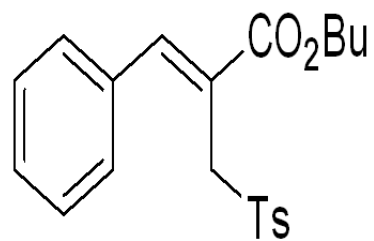
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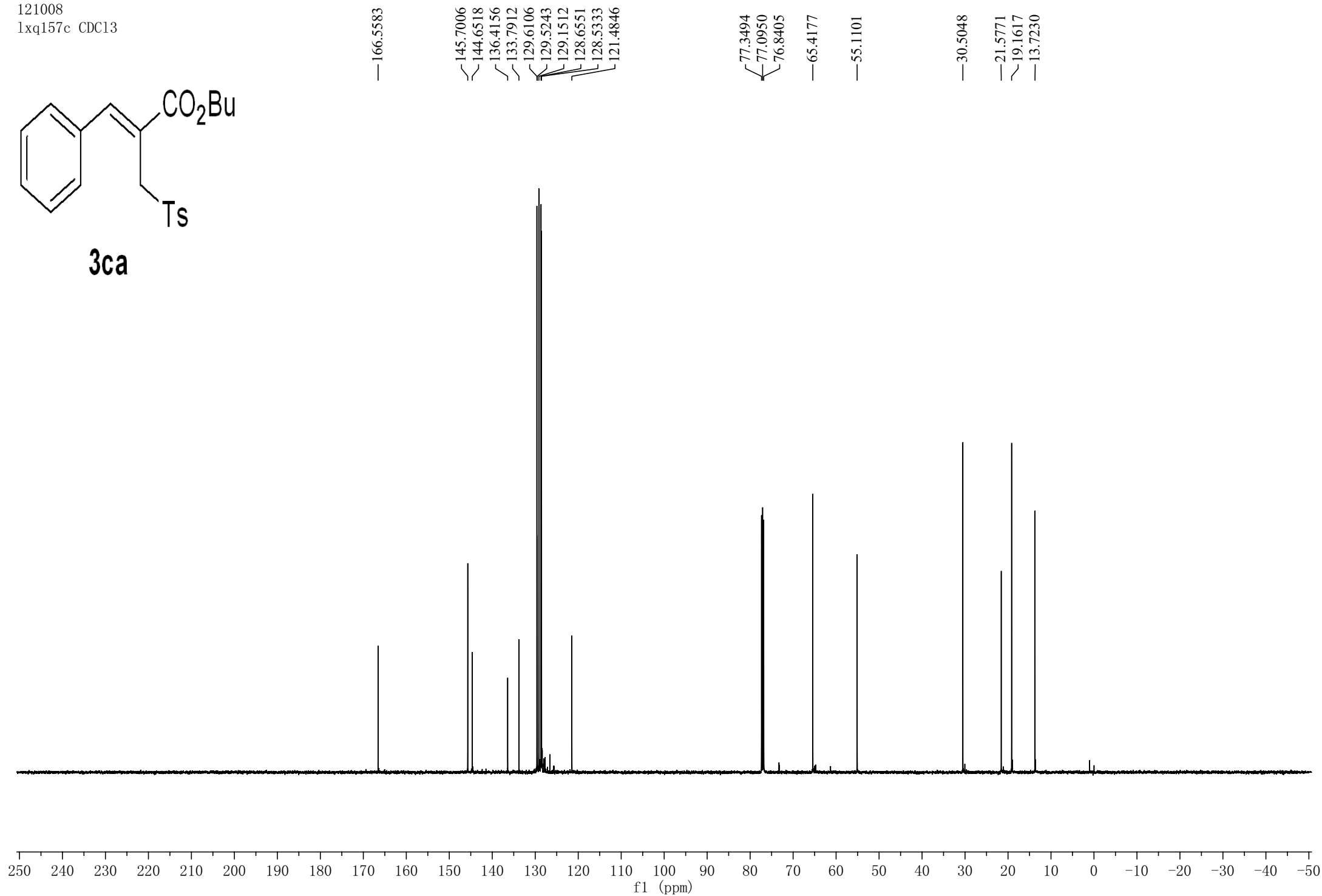
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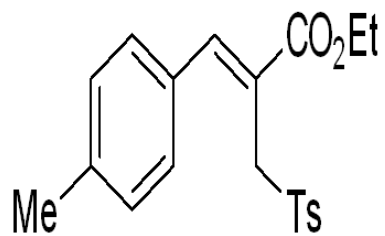
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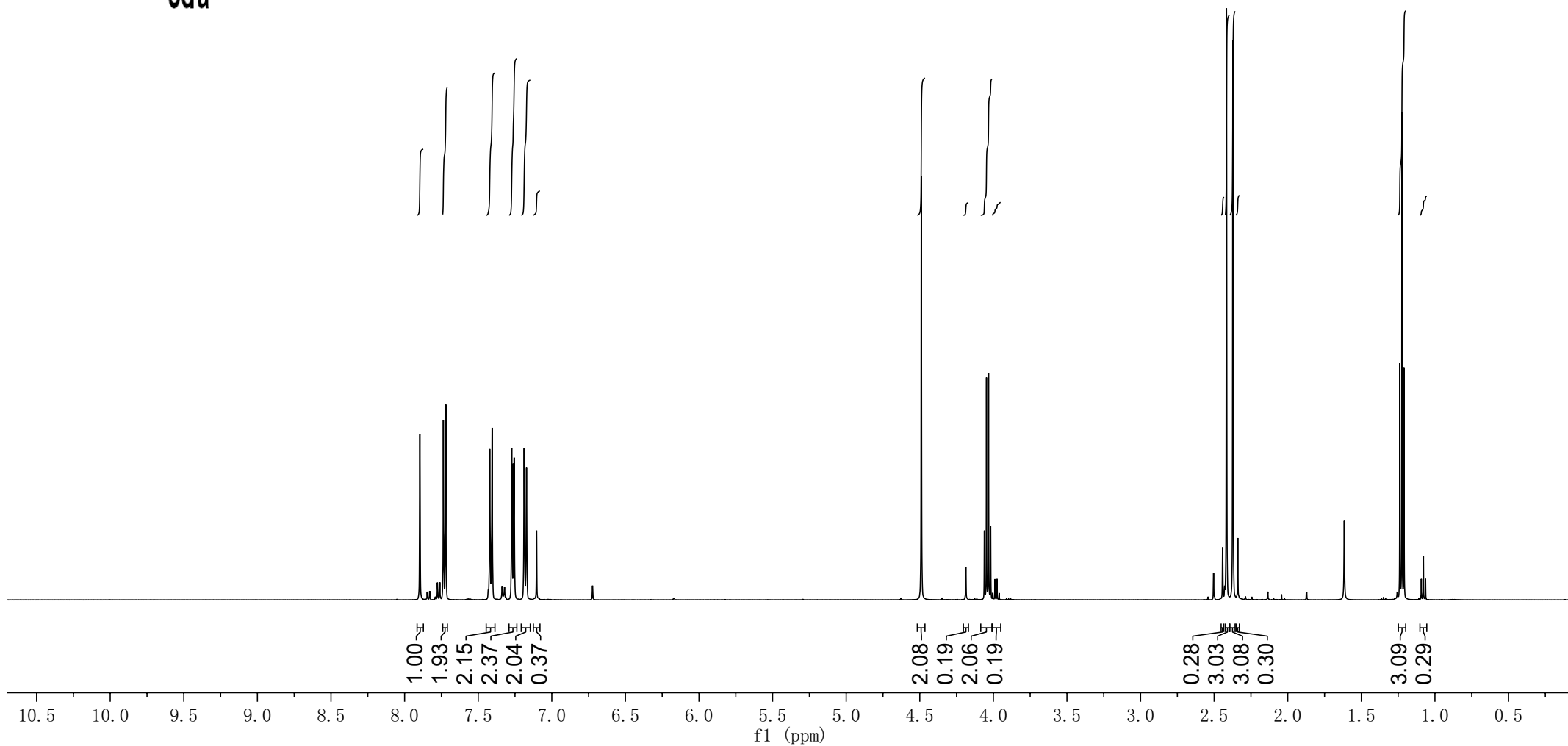
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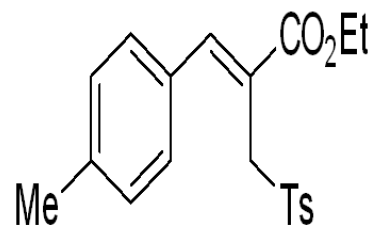
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121008
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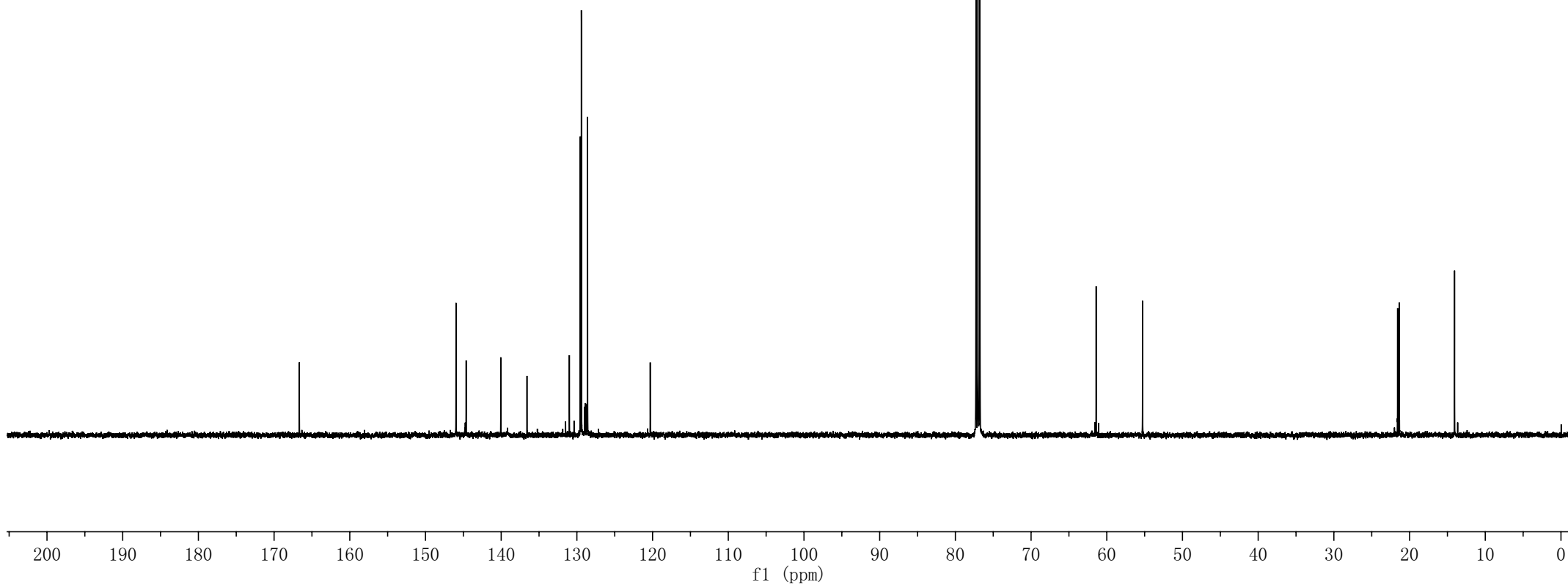
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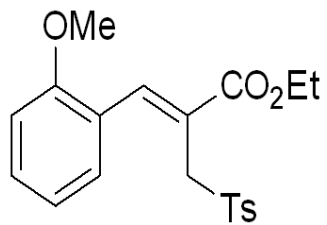
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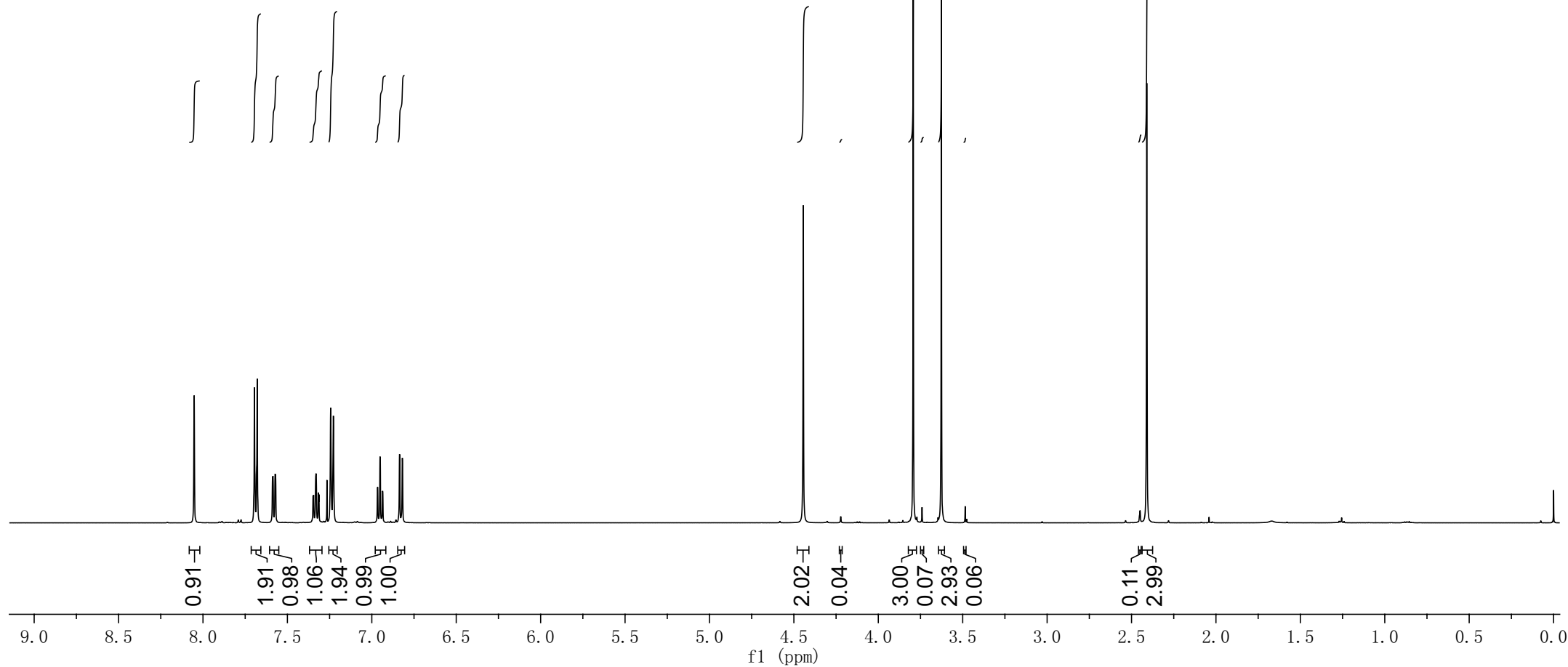
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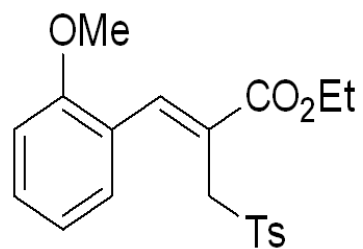
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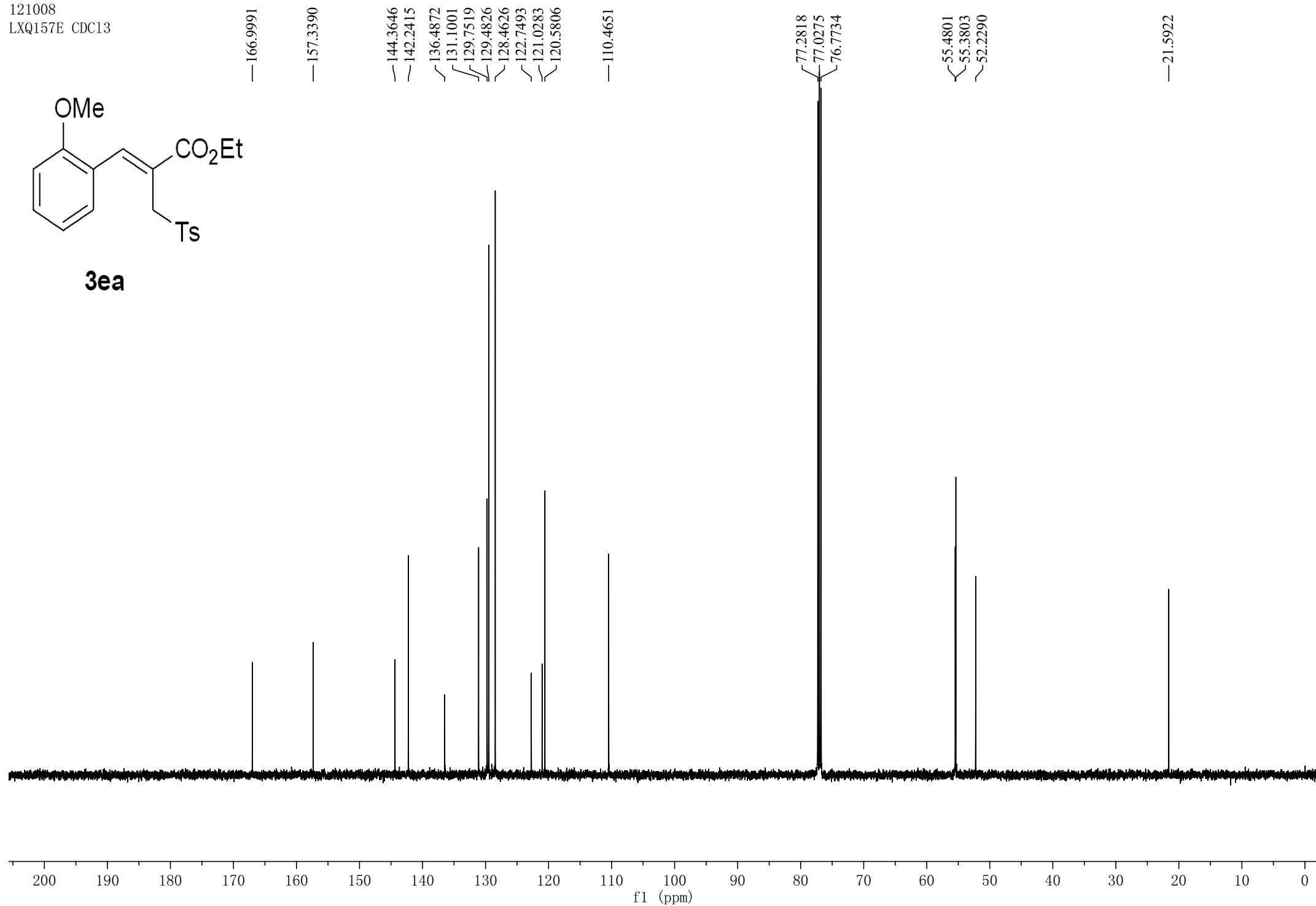
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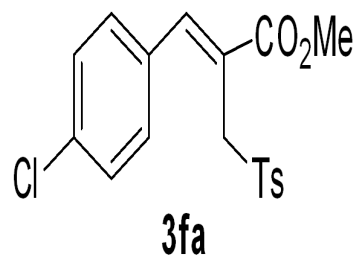
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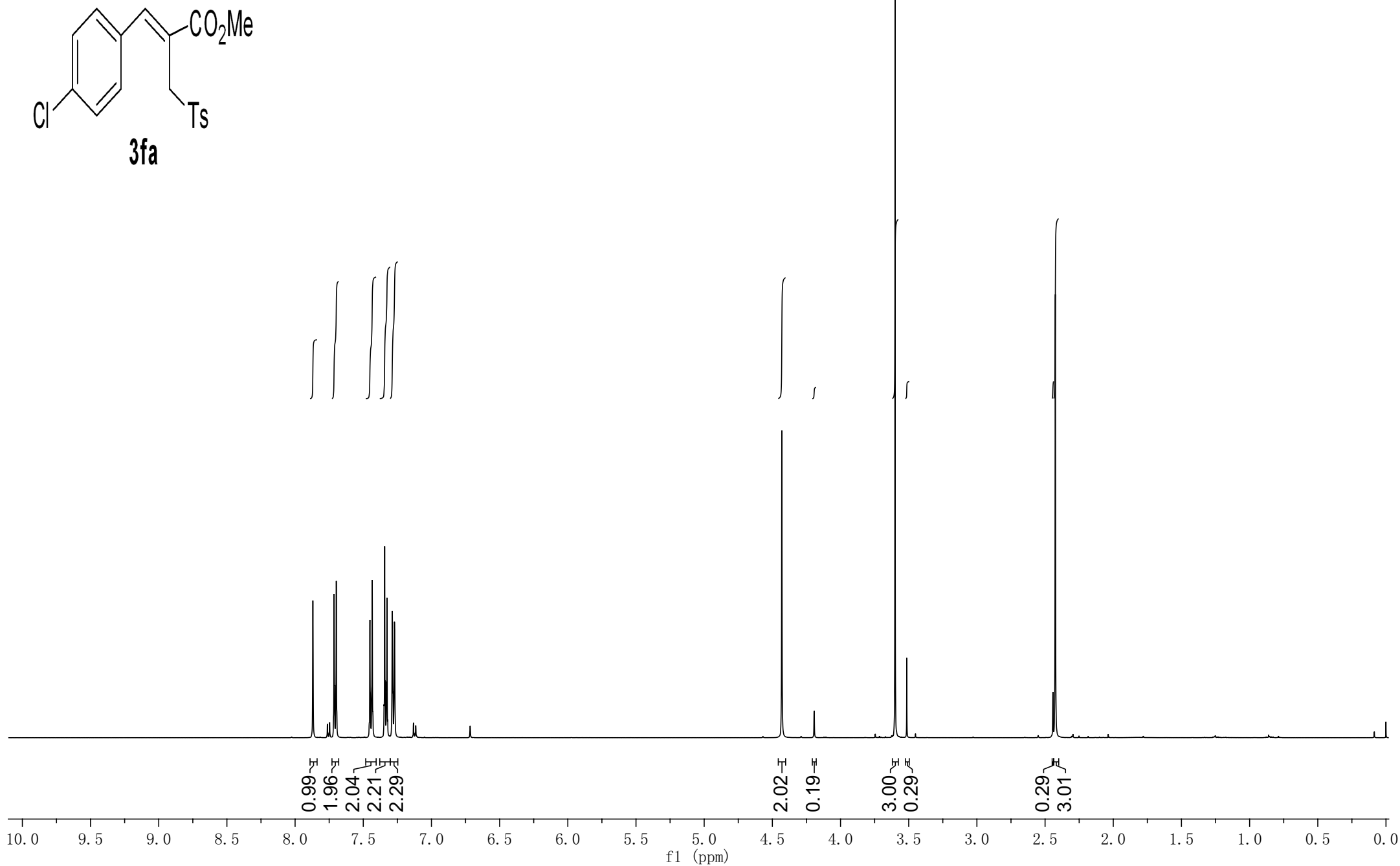
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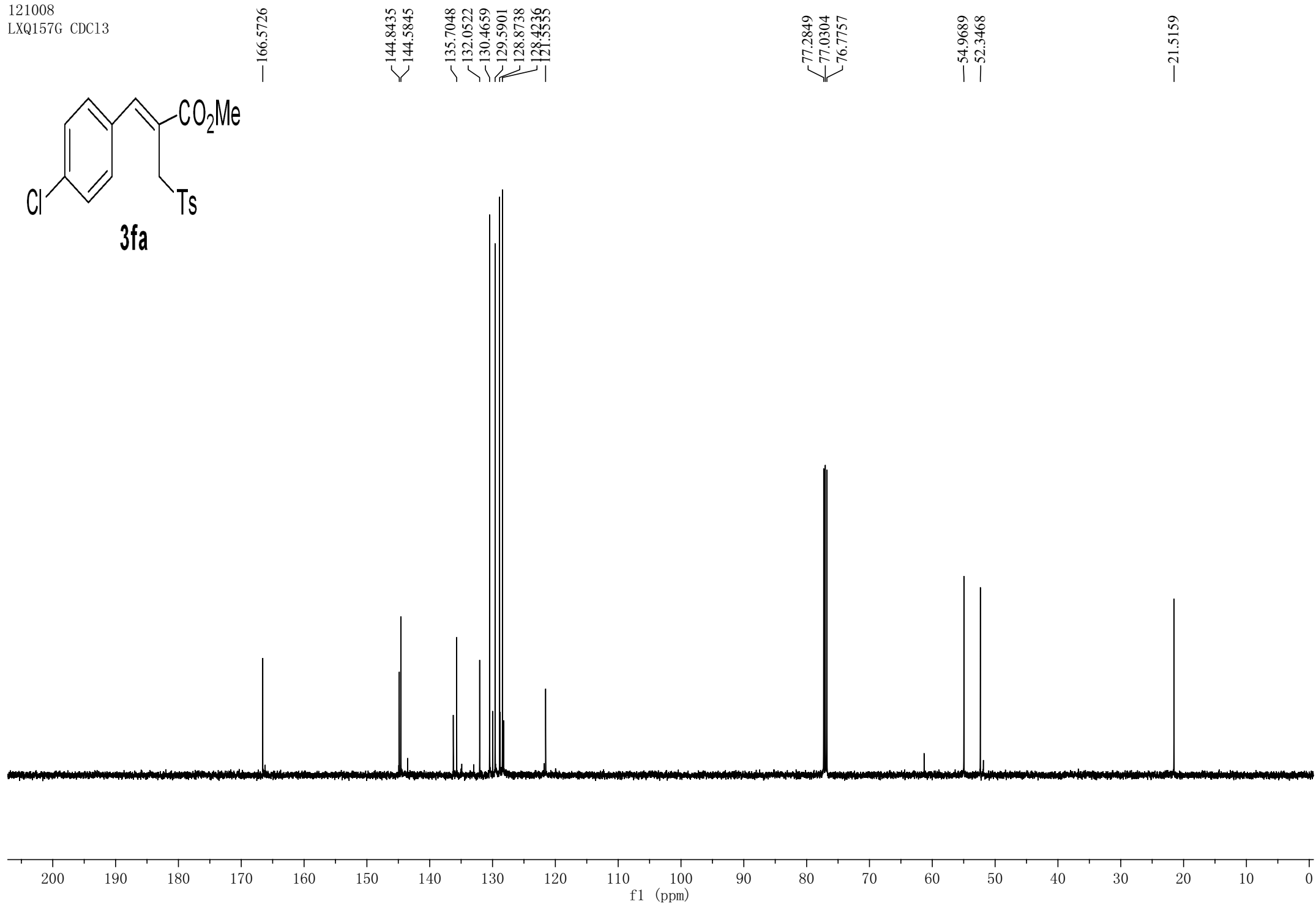
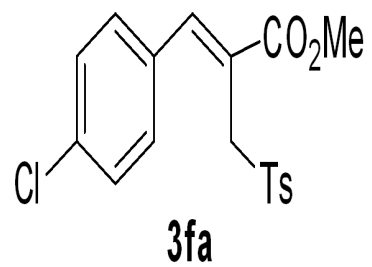
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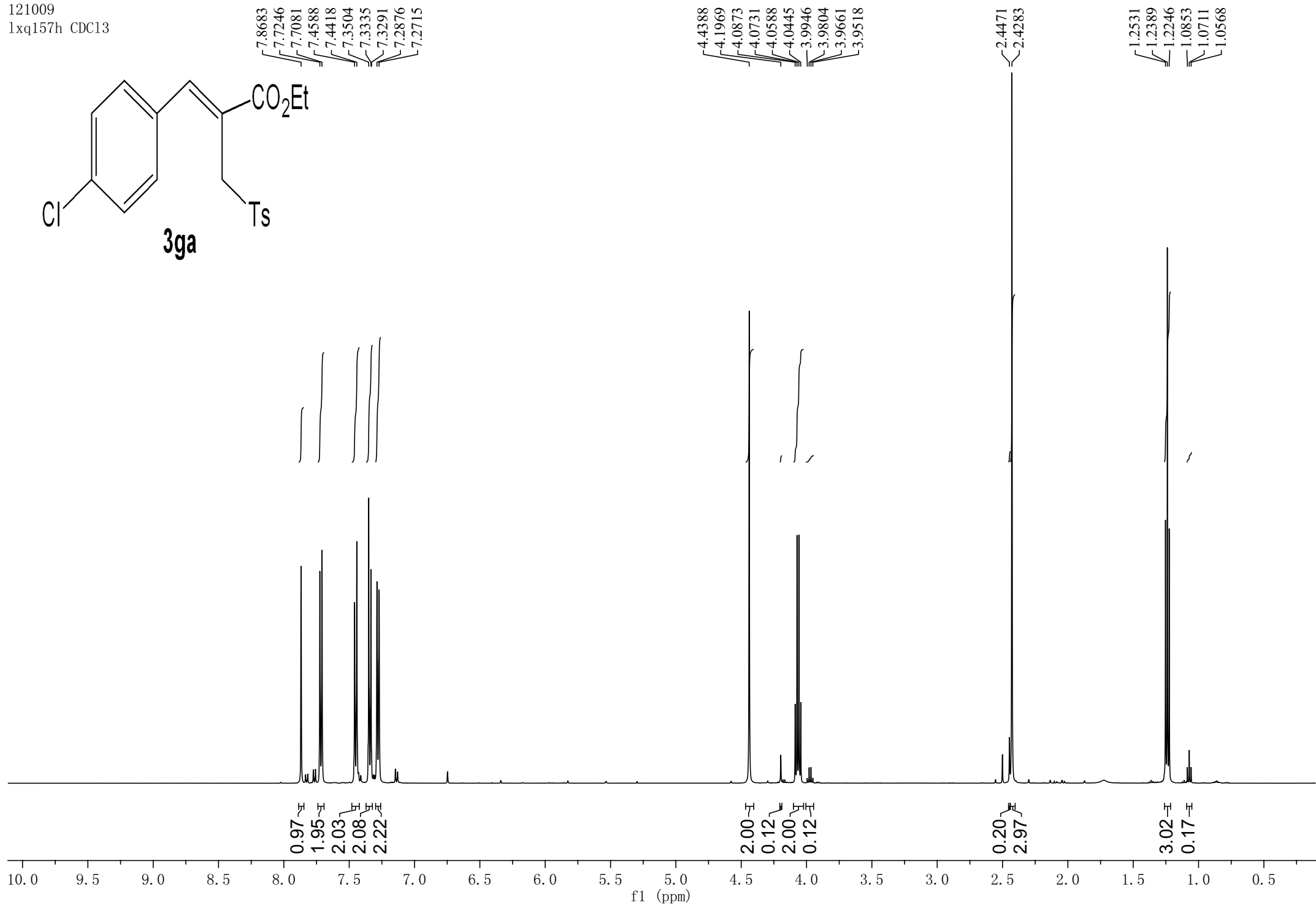
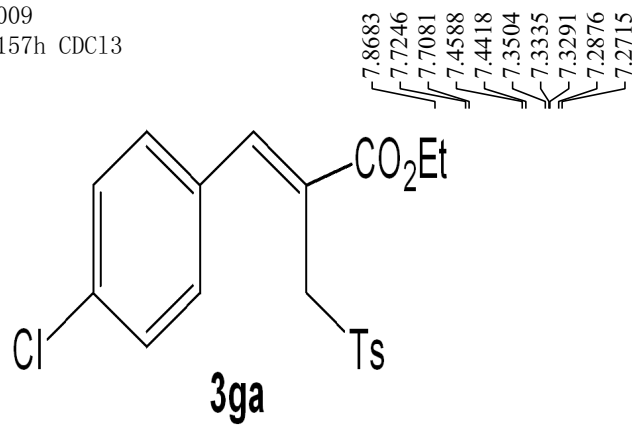
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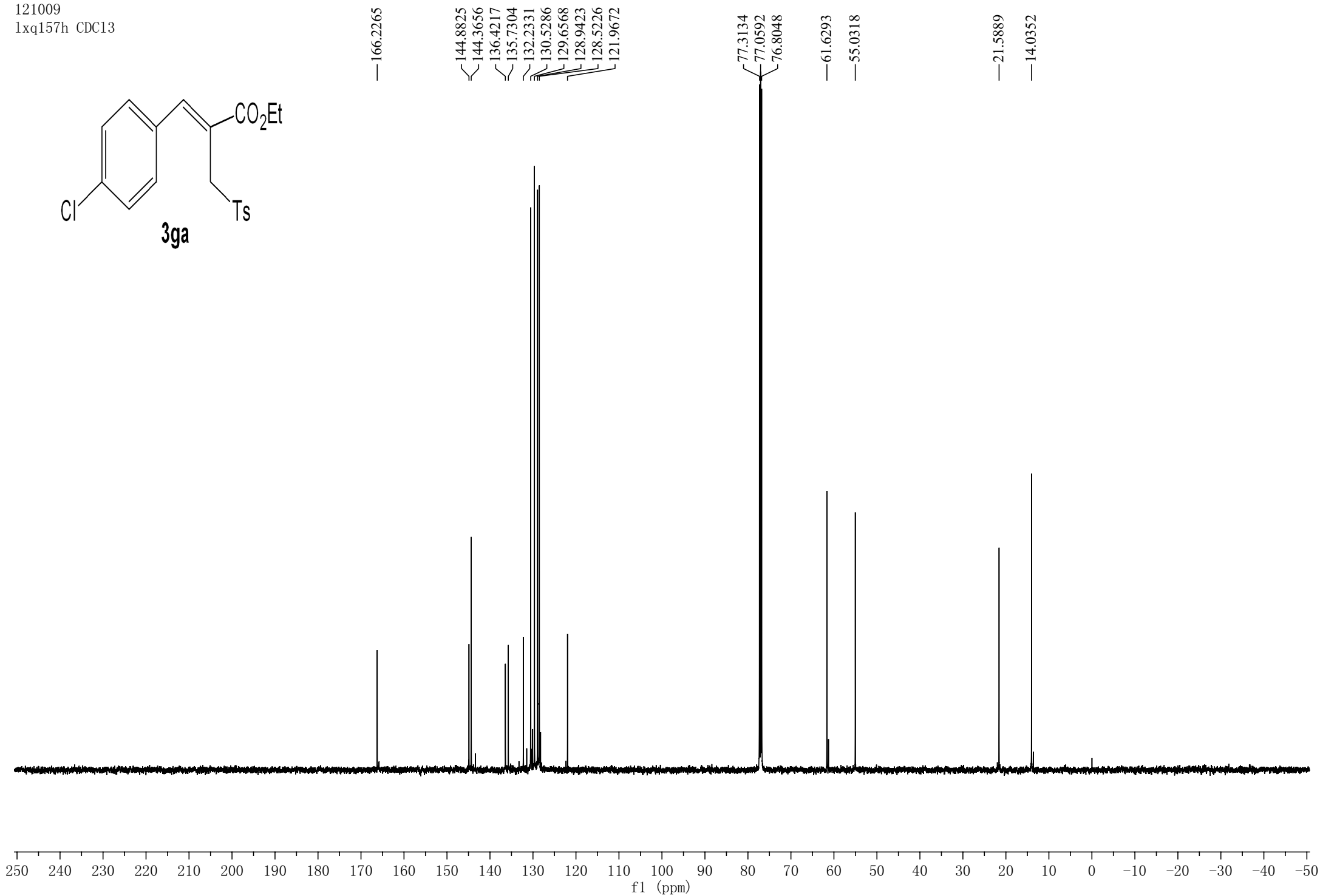
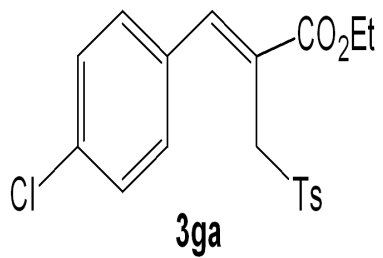
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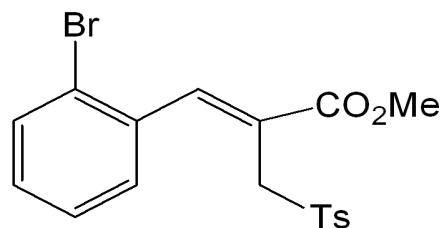
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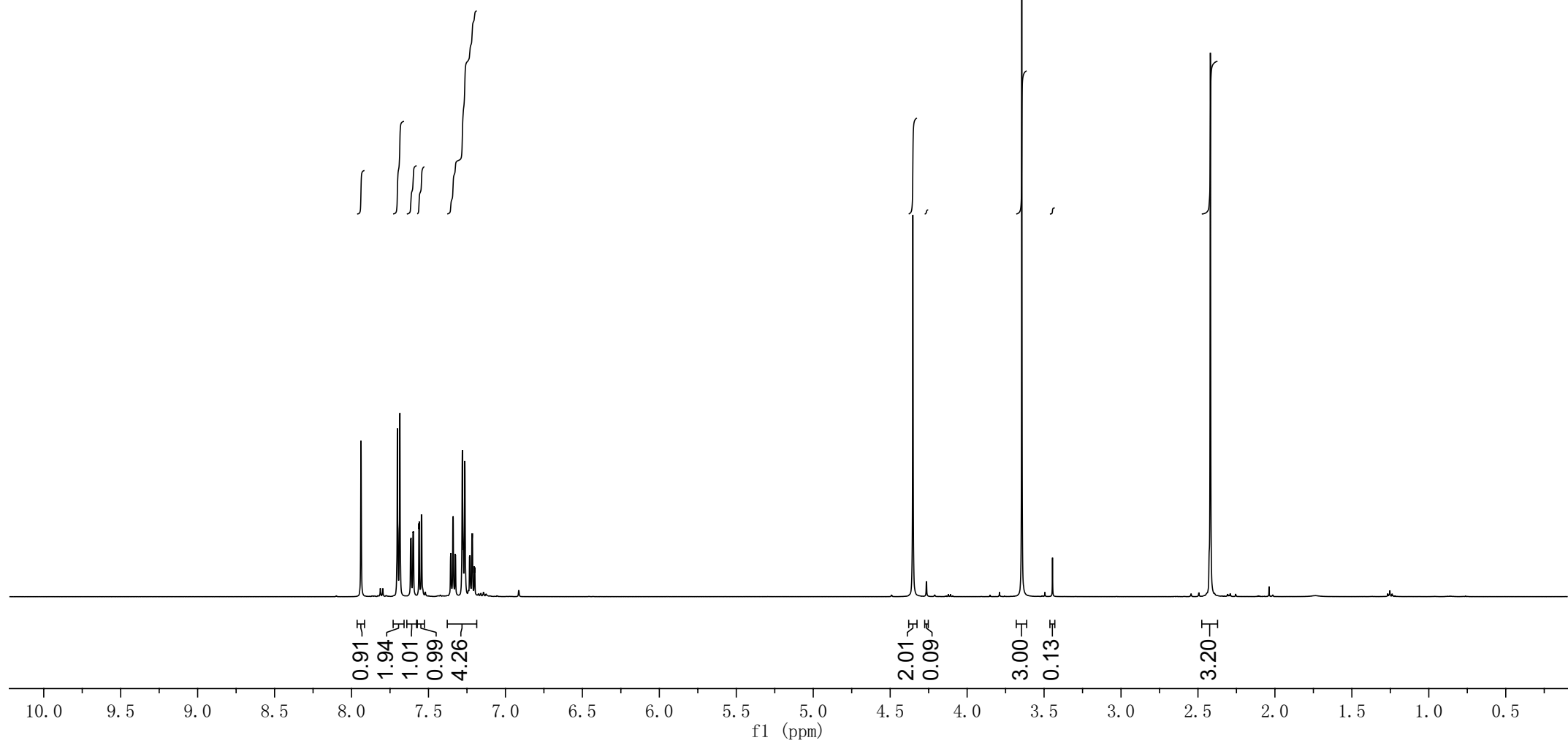


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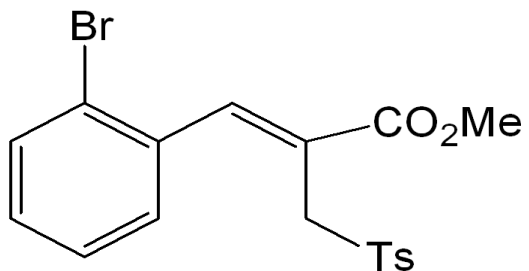


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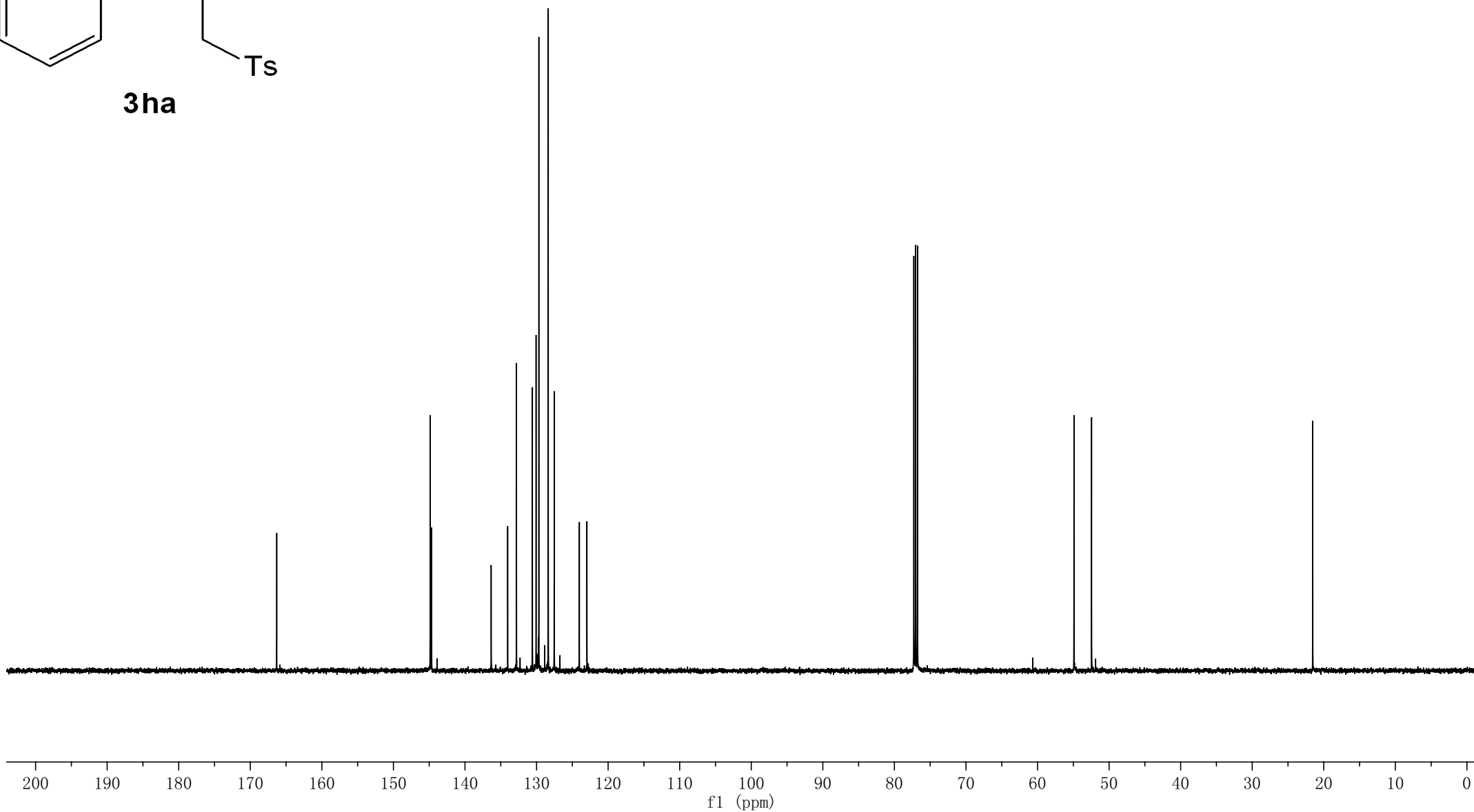
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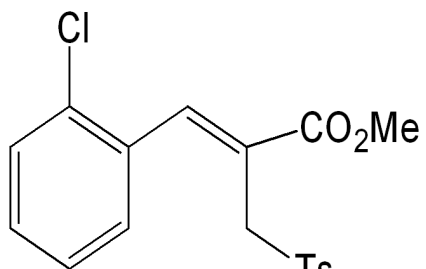
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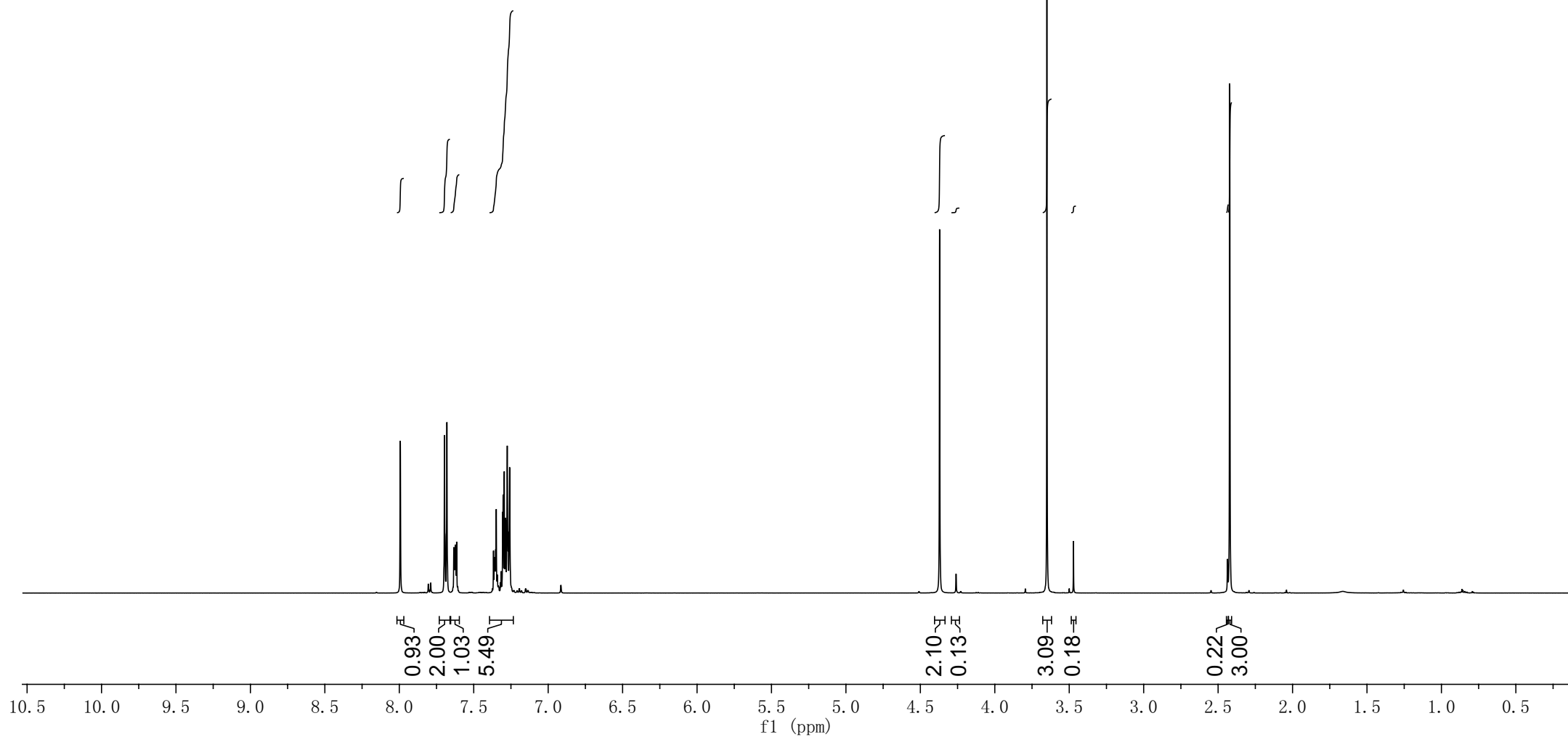
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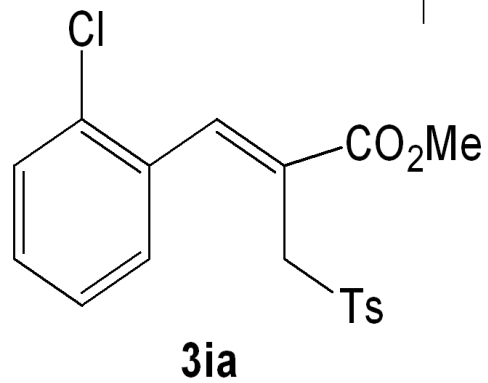
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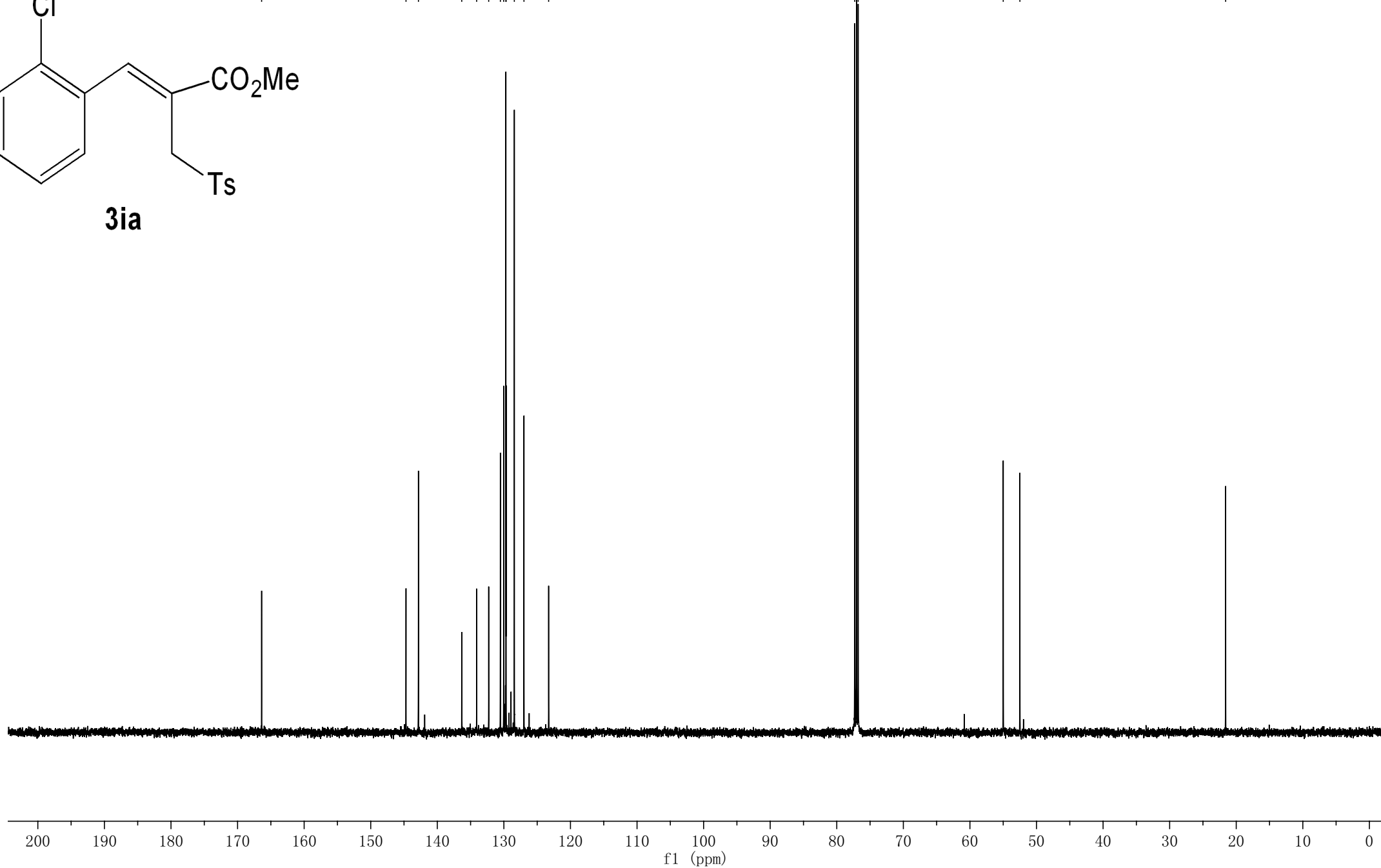
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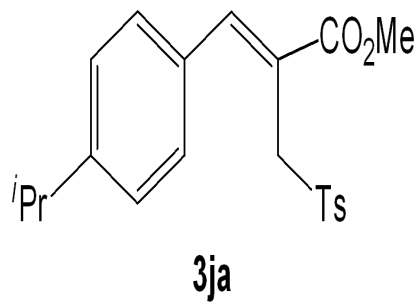
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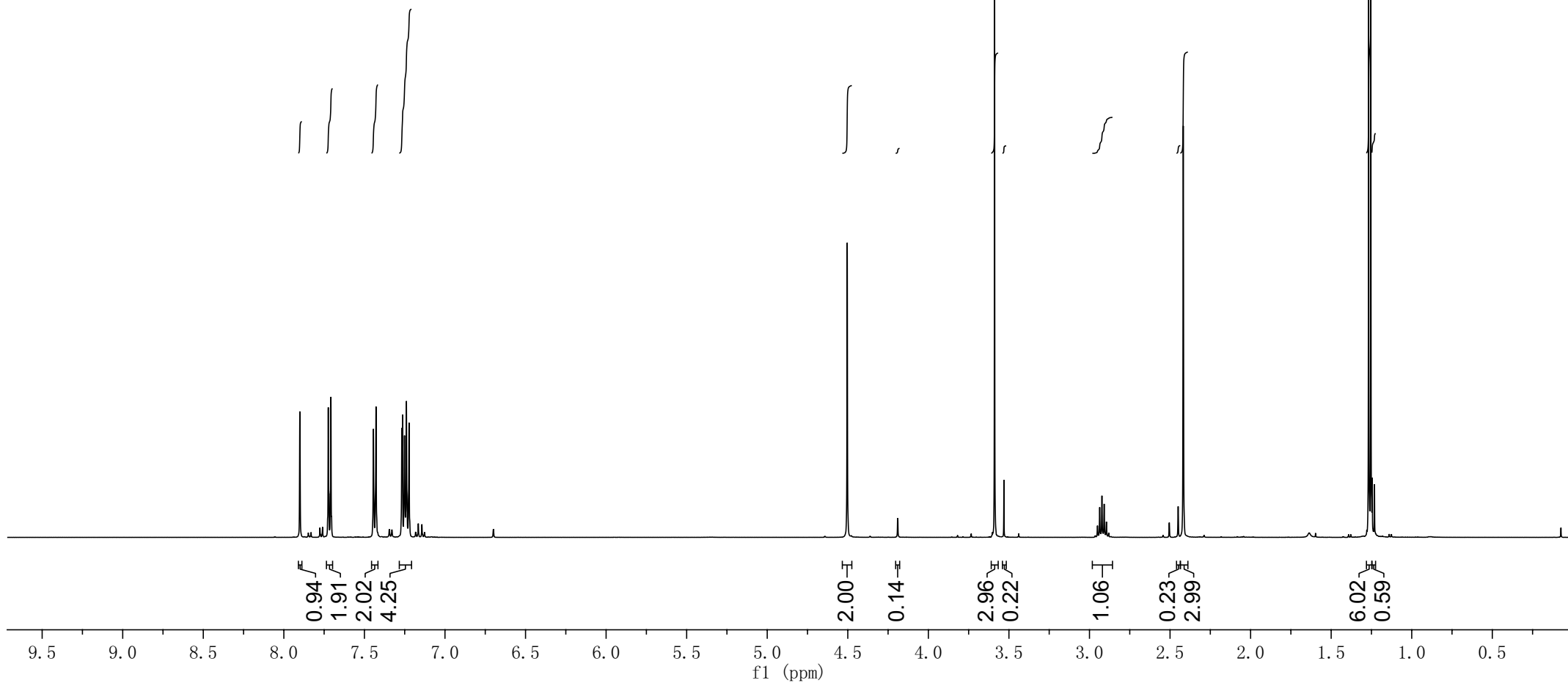
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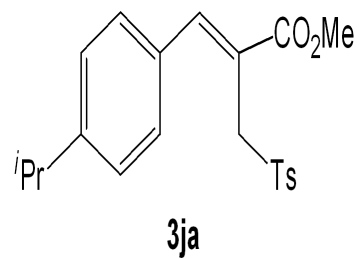
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121029
lxq1571 CDCl₃



— 167.1182

— 150.9974

— 146.2503

— 144.6098

— 136.4425

— 131.2278

— 129.5529

— 128.6020

— 126.8219

— 120.0075

— 77.2862

— 77.0320

— 76.7778

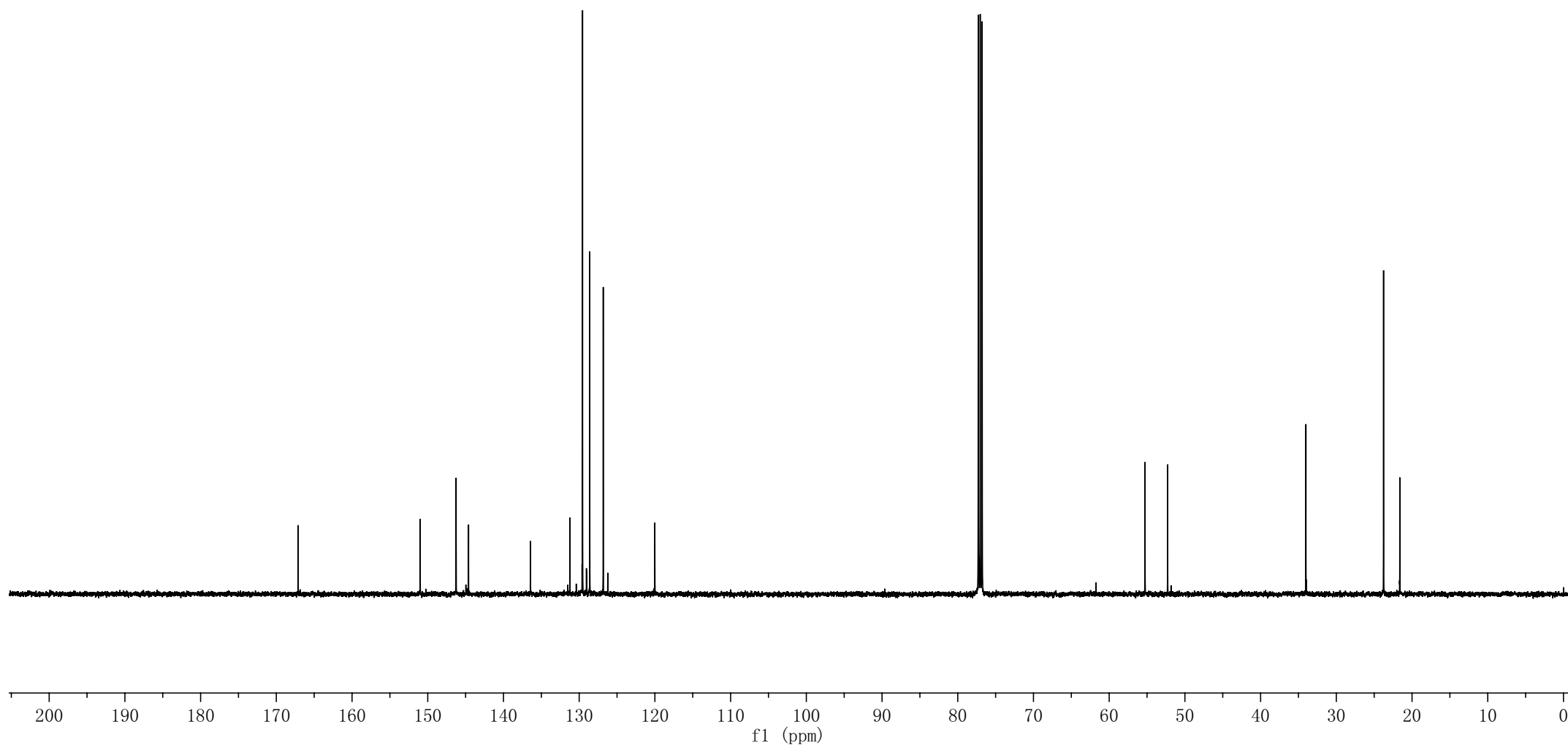
— 55.2706

— 52.2756

— 34.0150

— 23.7600

— 21.6082



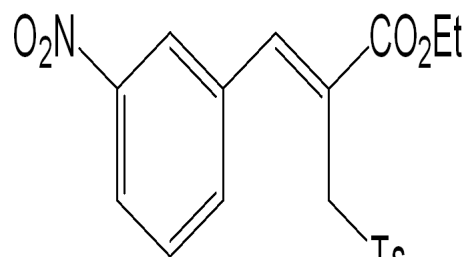
121112
lxq157m CDCl3

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7.9398
7.8974
7.8818
7.7264
7.7099
7.6073
7.5918
7.5739
7.2887
7.2731

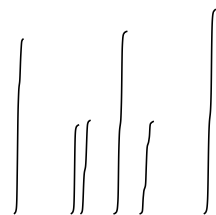
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4.1622
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4.1336

2.4098

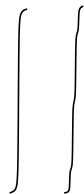
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1.2931
1.2788



3ka



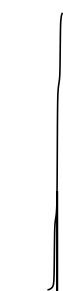
1.96
1.00
1.05
2.05
1.04
2.29



2.06
2.10



3.12

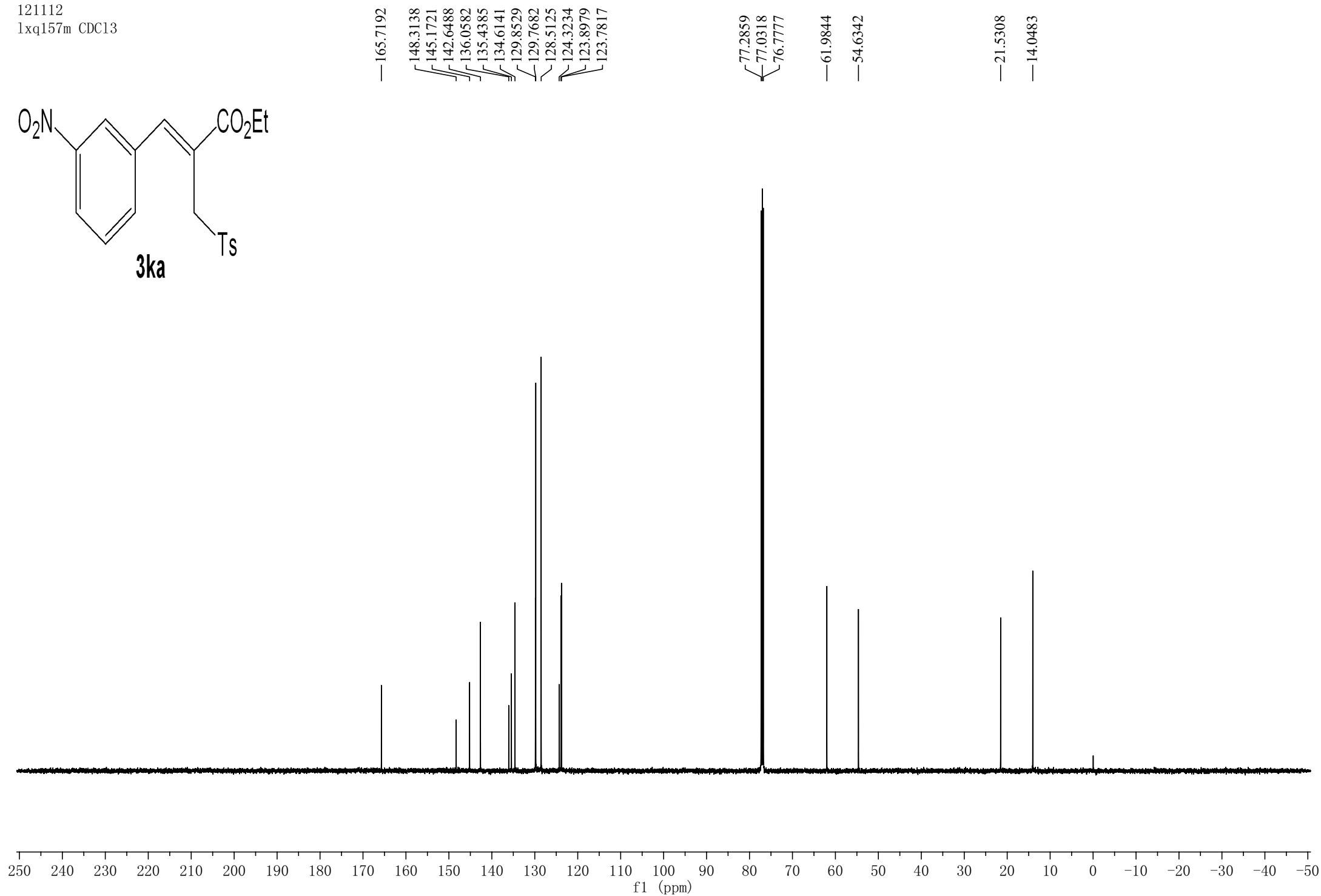
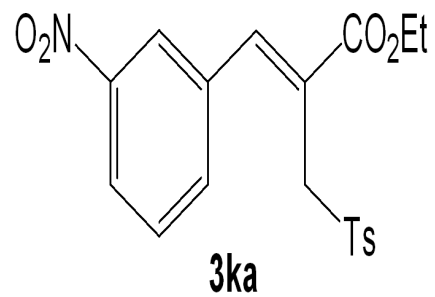


3.10

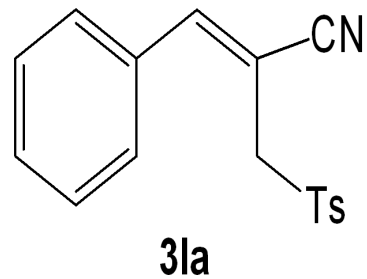
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f1 (ppm)

121112
lxq157m CDCl₃



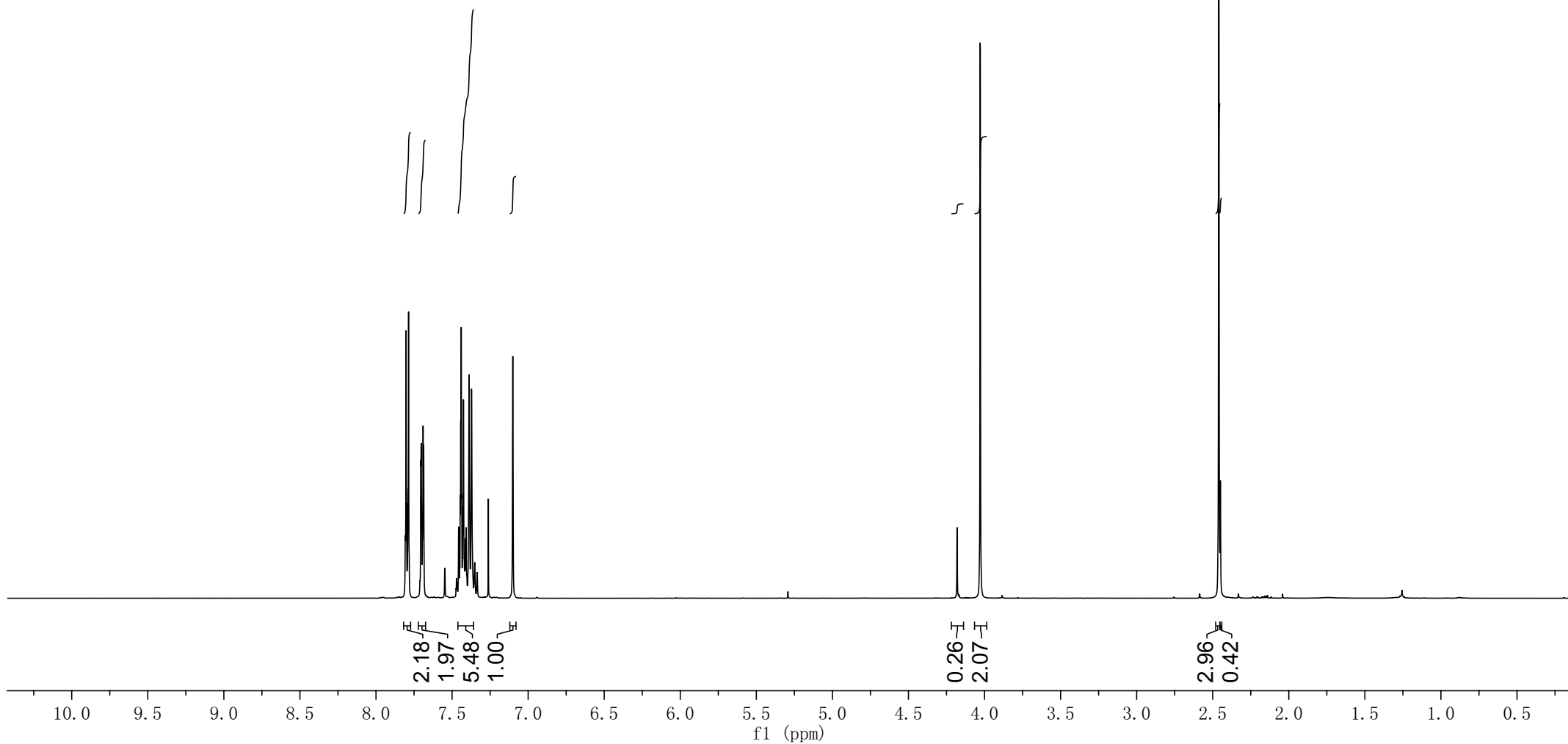
121018
lxq157k CDC13



7.8070
7.8026
7.7992
7.7896
7.7859
7.7054
7.7027
7.6904
7.6869
7.4571
7.4555
7.4465
7.4432
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7.4123
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7.3719
7.2613
7.1007

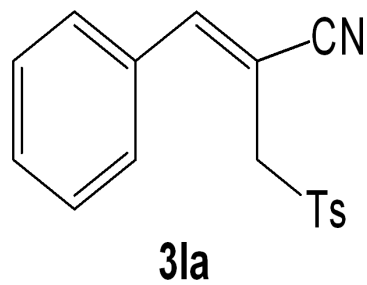
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4.0287

2.4602
2.4495

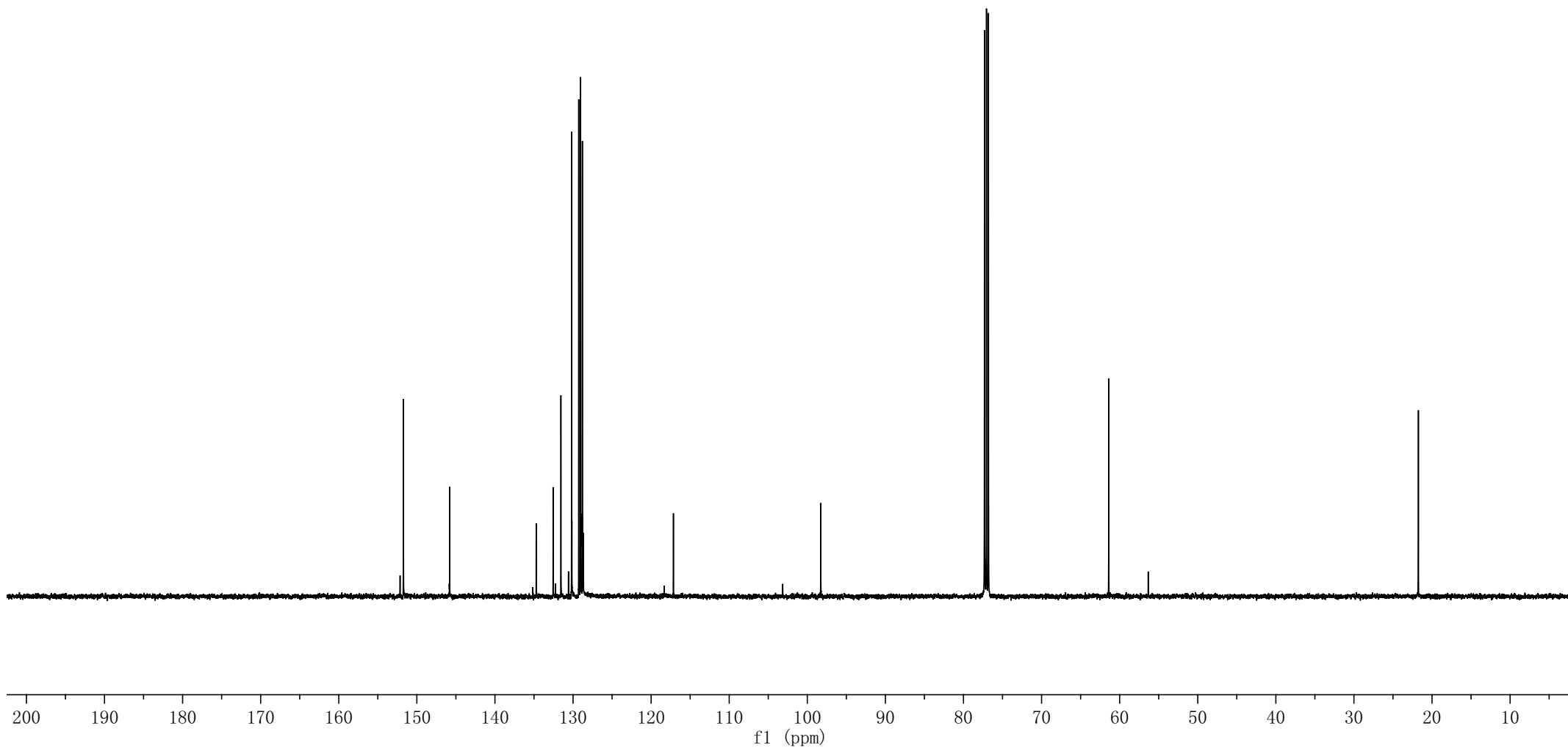


121018
lxq157k CDC13

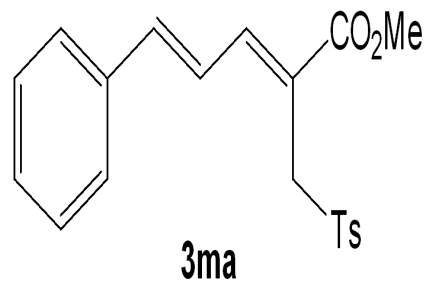
Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is © The Royal Society of Chemistry 2013



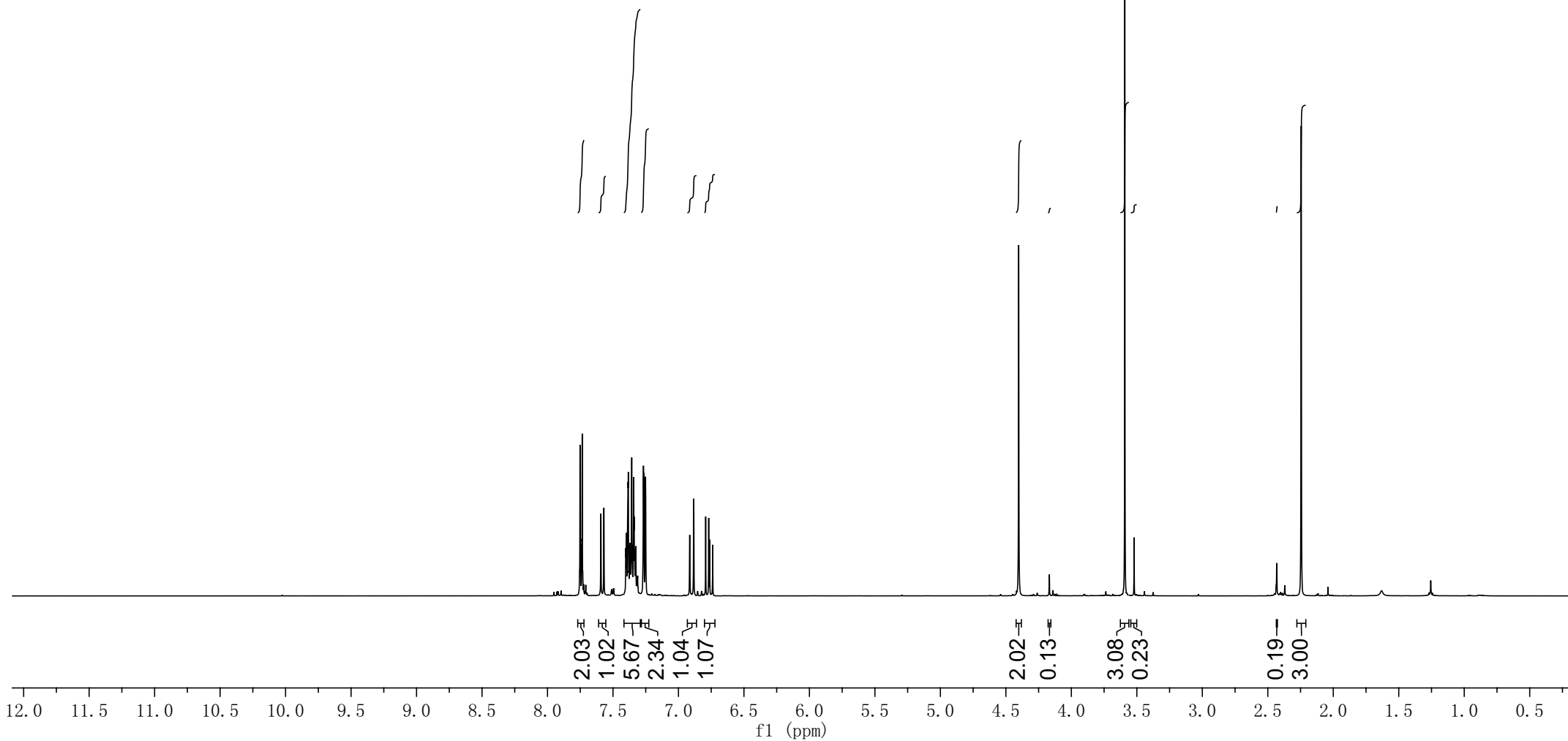
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— 145.7990
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132.5531
131.5489
130.1845
129.2716
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— 117.1424
— 98.2804
77.3104
77.0559
76.8018
— 61.3988
— 21.7417



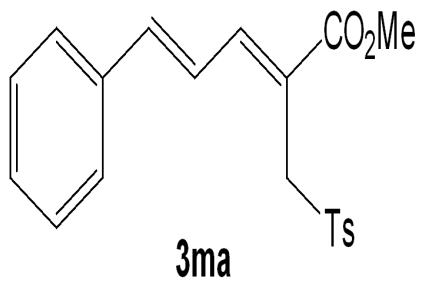
121008
lxql57n



3ma



121008
lxq157n CDC13



— 166.5016

145.2265
144.9504
143.2564

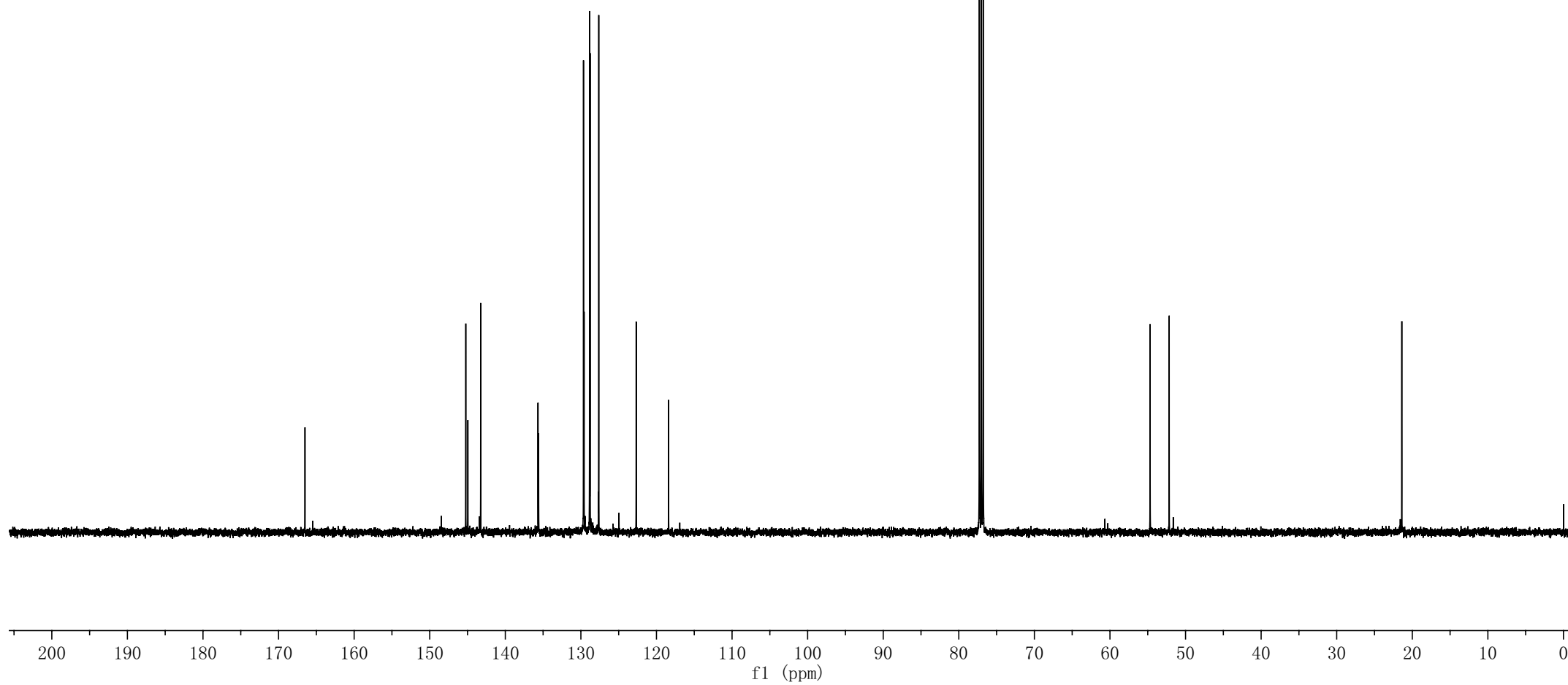
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129.6550
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128.8482
128.7805
127.6341
122.6858
118.4018

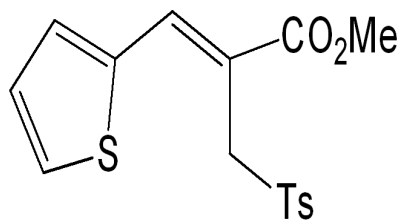
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77.0253
76.7709

— 54.6913
— 52.1631

— 21.3872



121015
lxq15702 CDC13



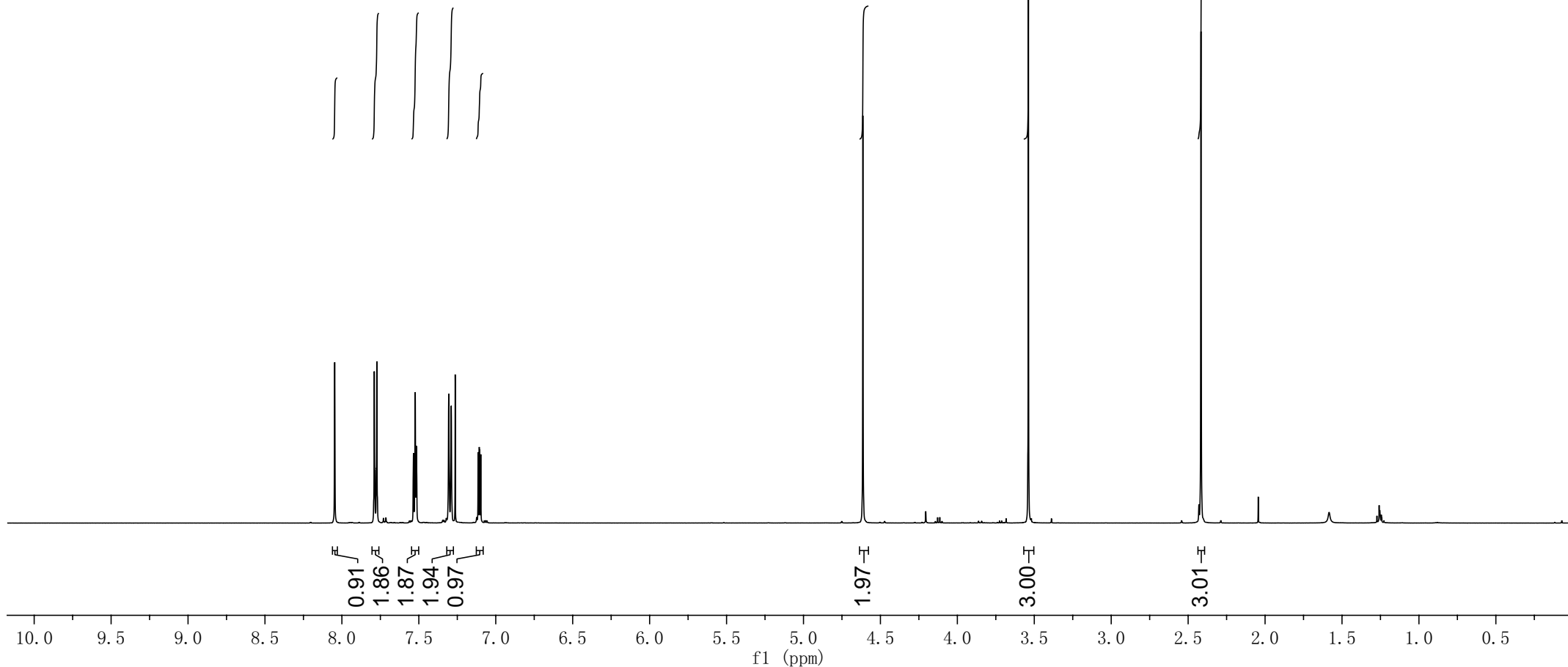
3na

8.0468
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7.7729
7.5347
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7.5154
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7.2895
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7.1148
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7.1047
7.0972

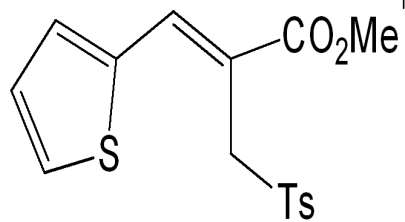
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3.5383

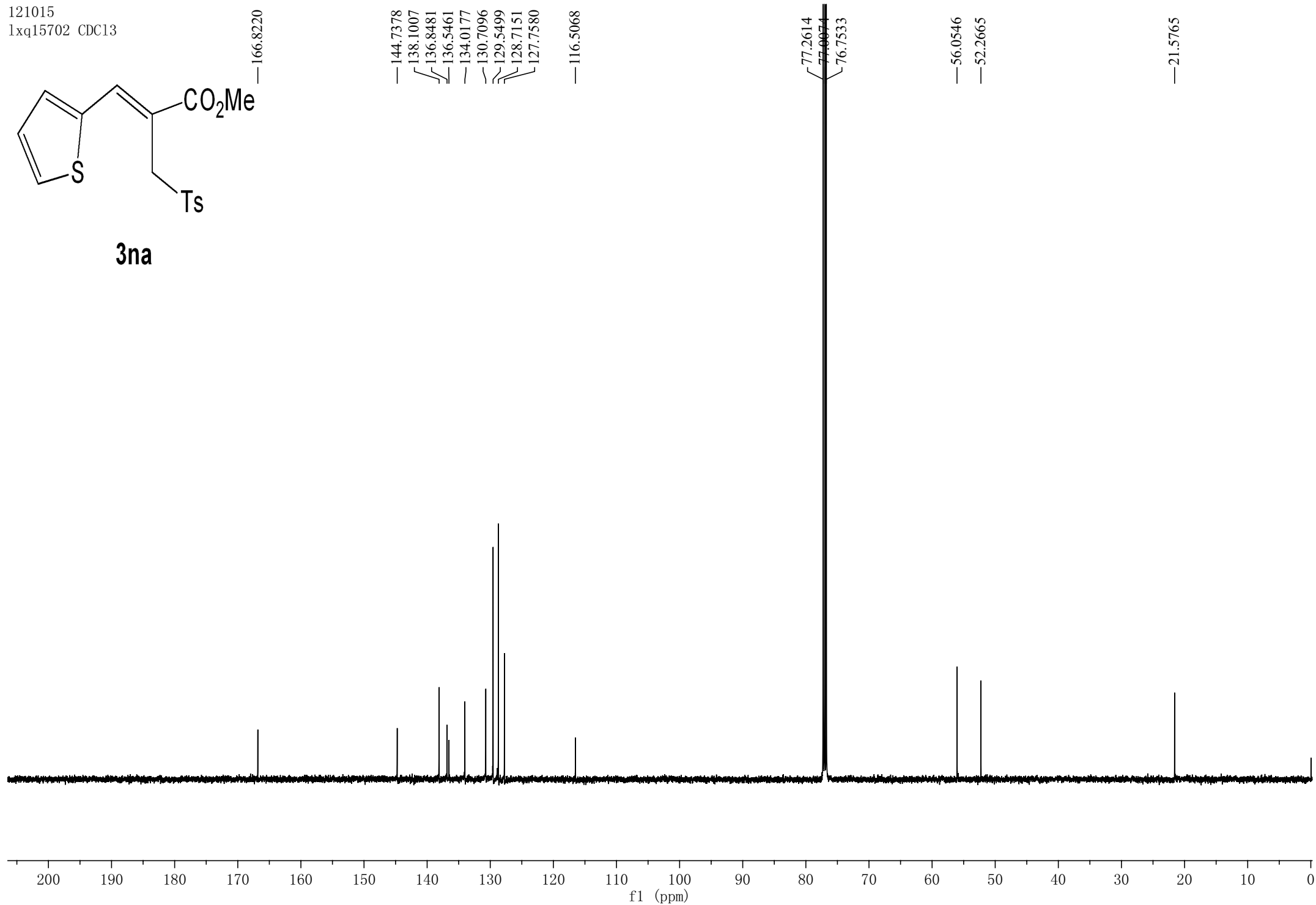
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121015
lxq15702 CDC13



3na



121018
lxq157qCDC13

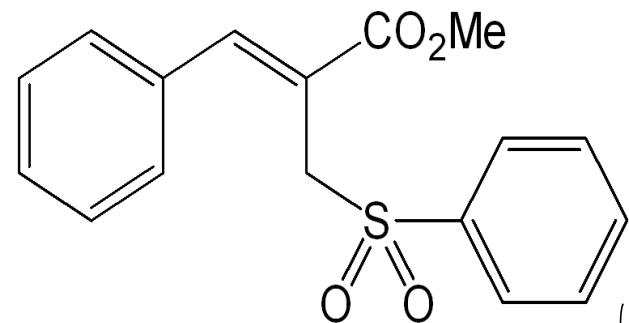
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7.8347
7.6002
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7.4993
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7.4765
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7.3610
7.3563
7.2732

4.4903

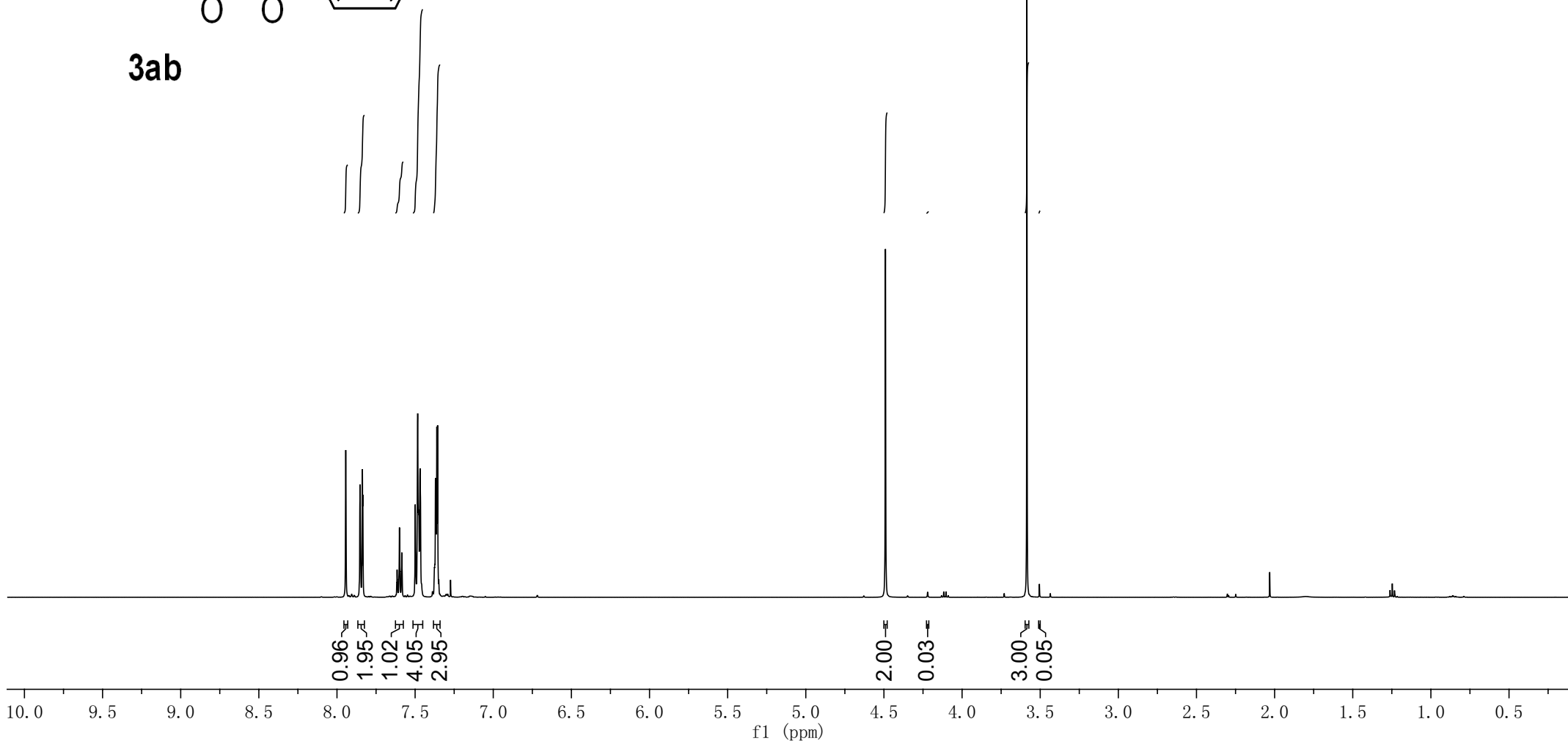
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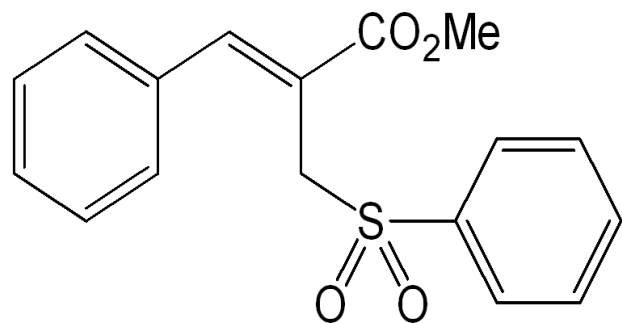
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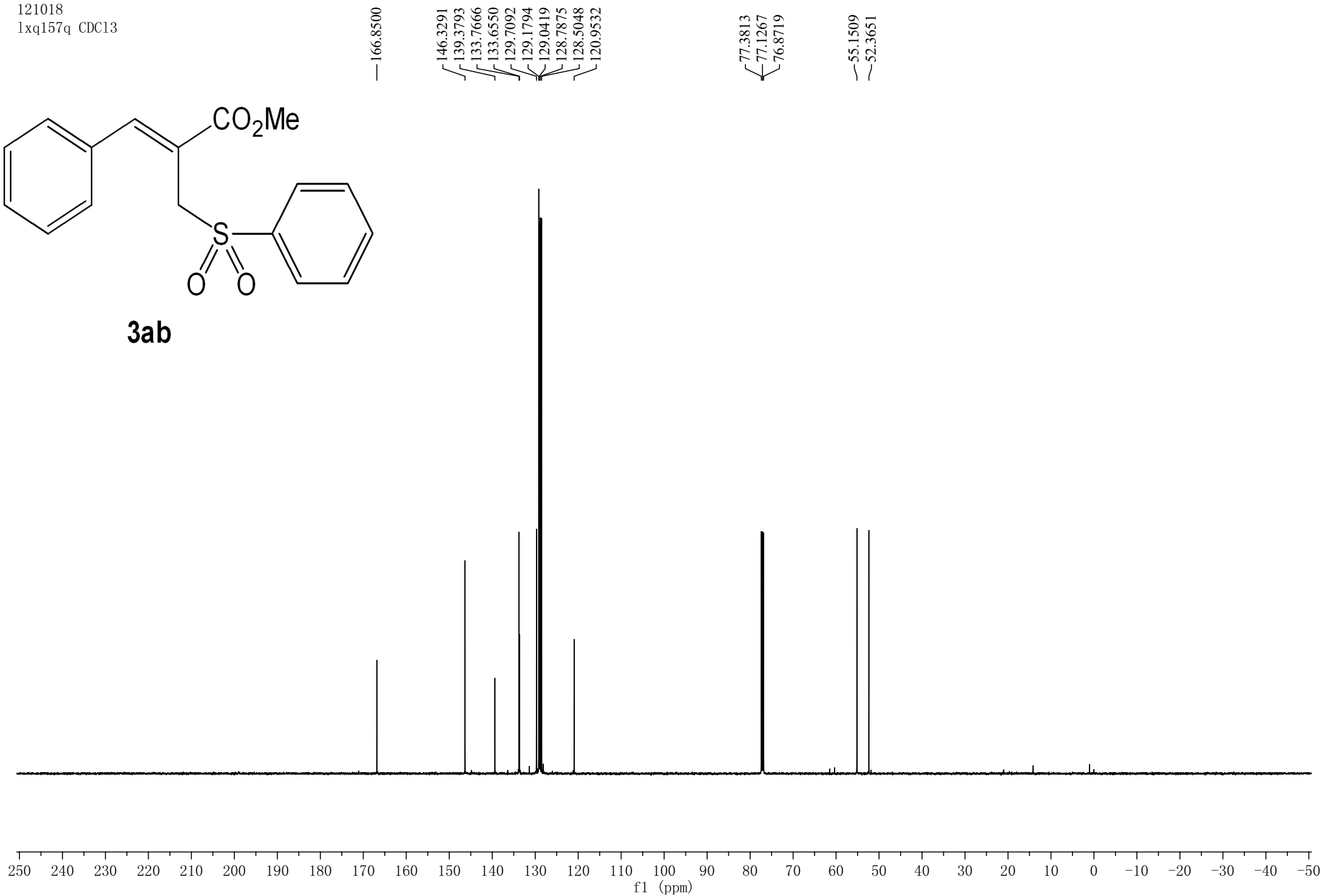
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121018
lxq157q CDC13



3ab

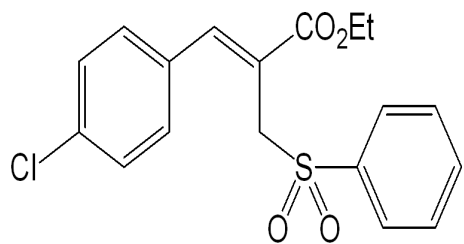


121016
lxq157q CDC13

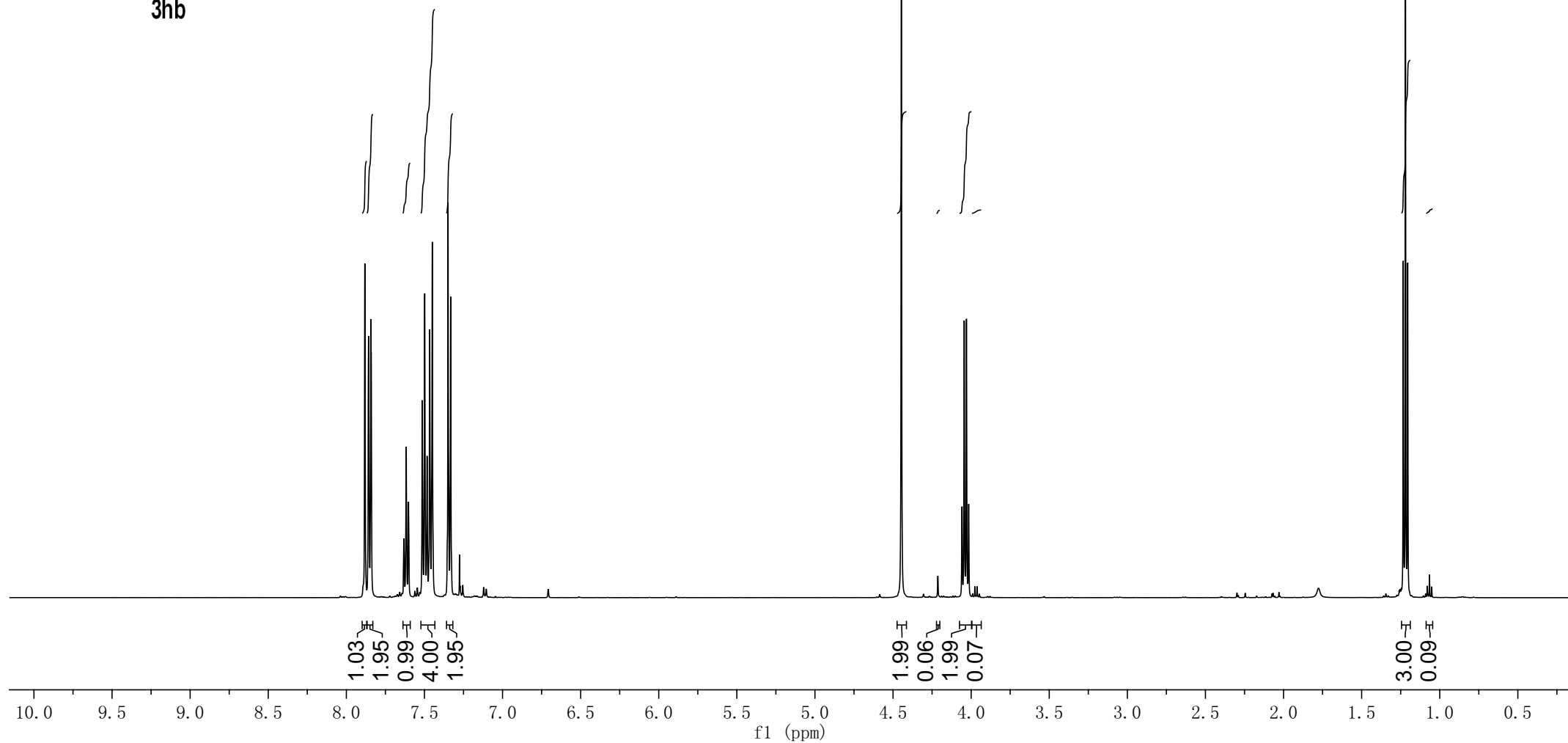
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7.8410
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7.6021
7.5142
7.4984
7.4830
7.4666
7.4498
7.3490
7.3320
7.2755

4.4468
4.2131
4.0585
4.0442
4.0299
4.0157
3.9904
3.9761
3.9618
3.9476

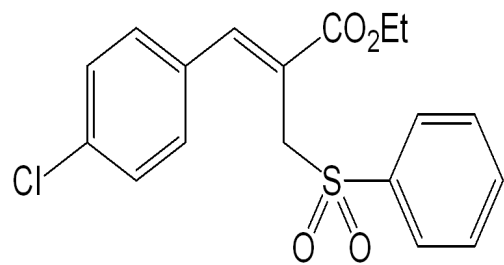
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3hb



121016
lxq157q CDC13



3hb

—166.0731

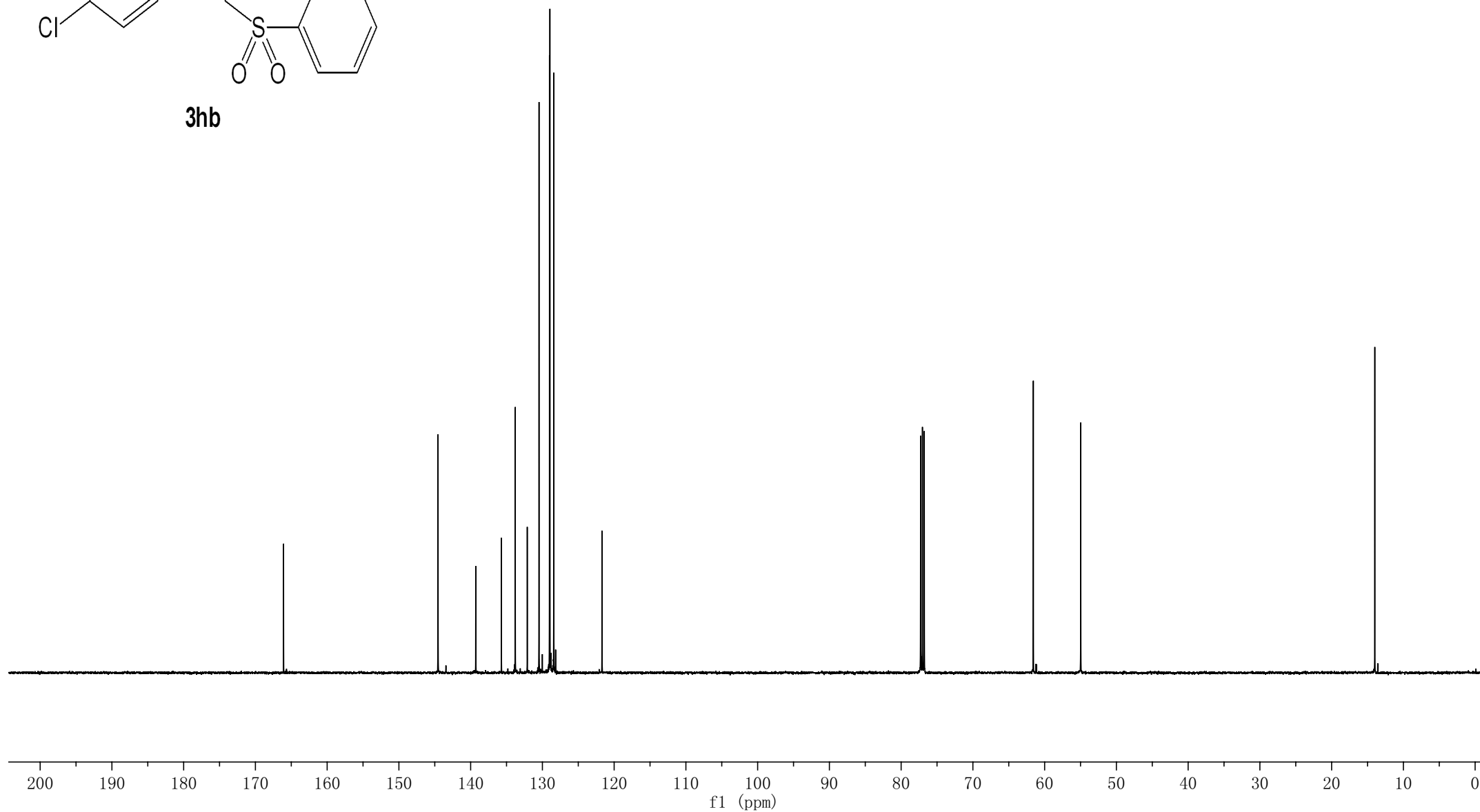
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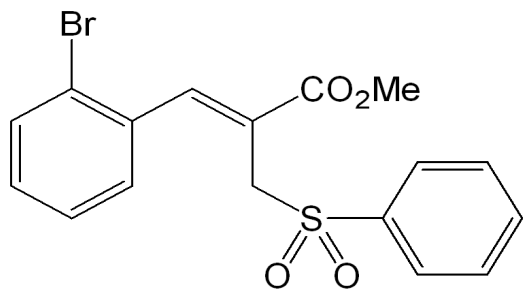
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—13.9773

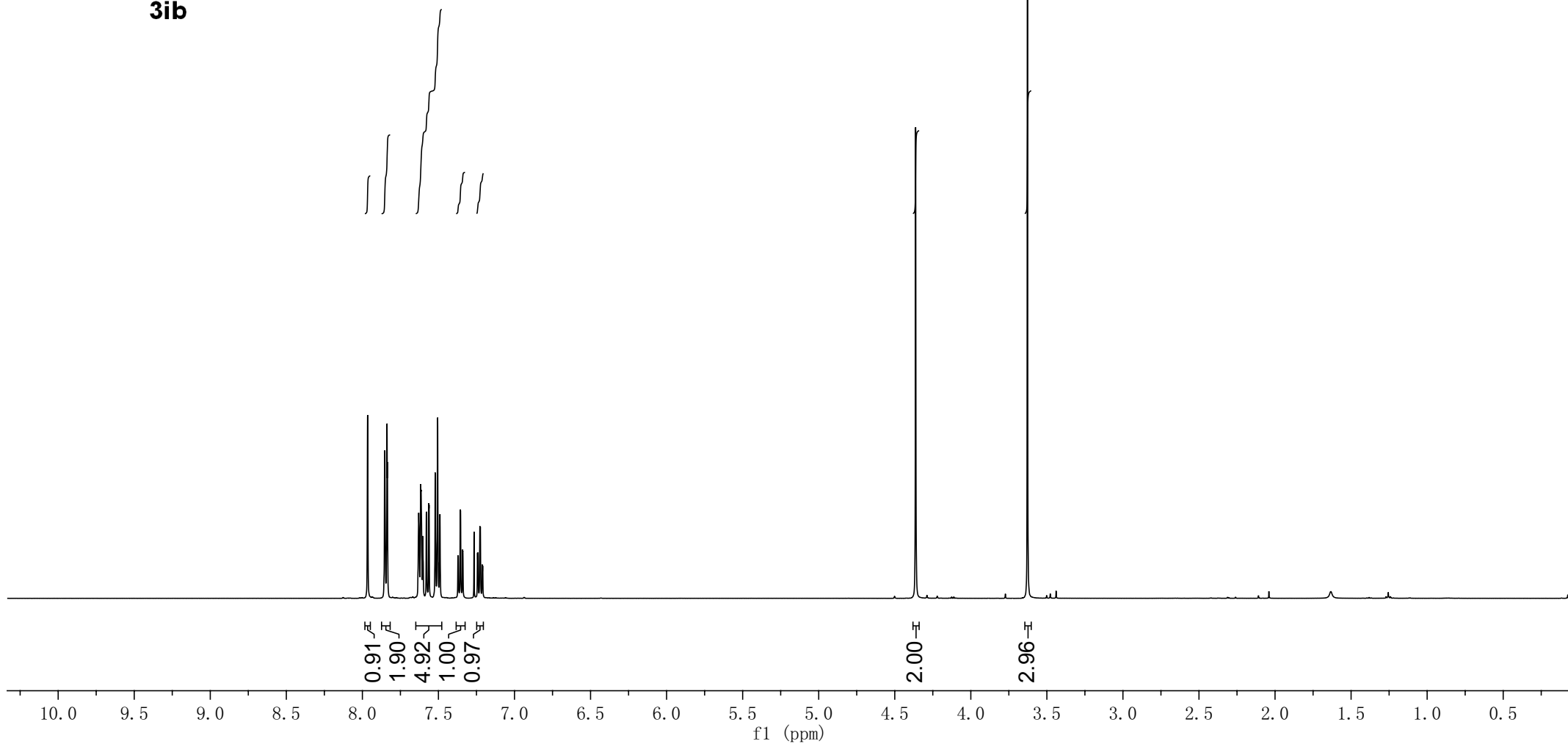


121016
lxq157r CDCl₃

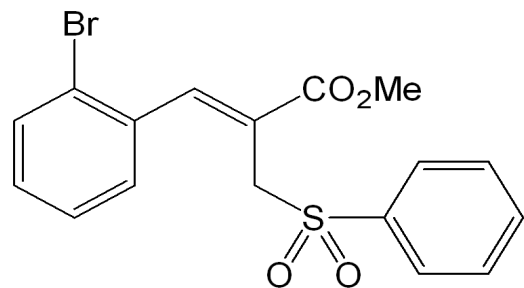
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7.5616
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7.2246
7.2120
7.2091



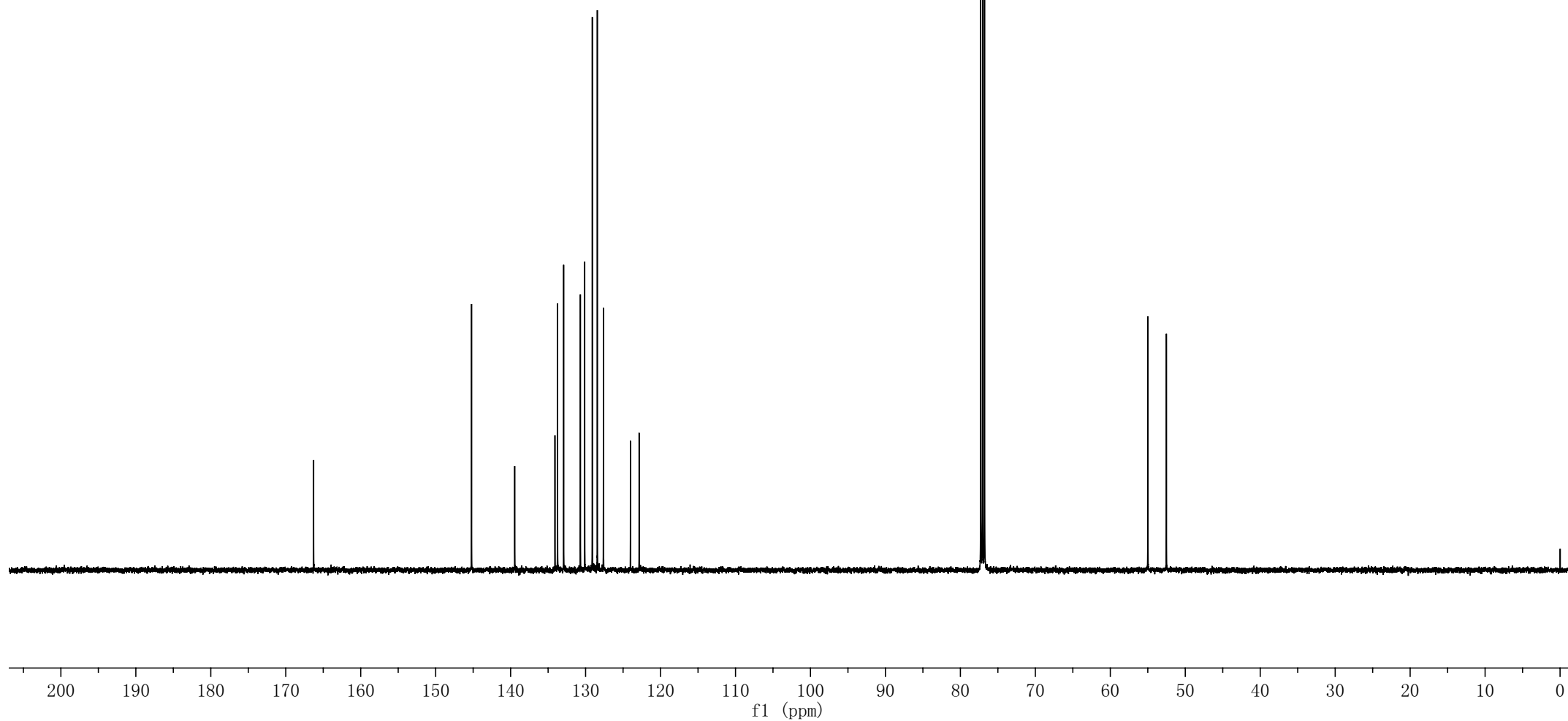
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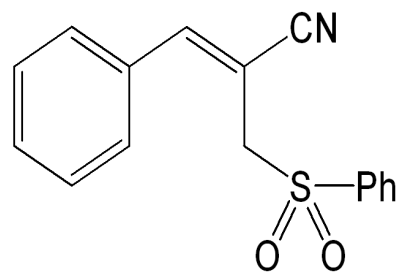
121016
lxq157r CDC13



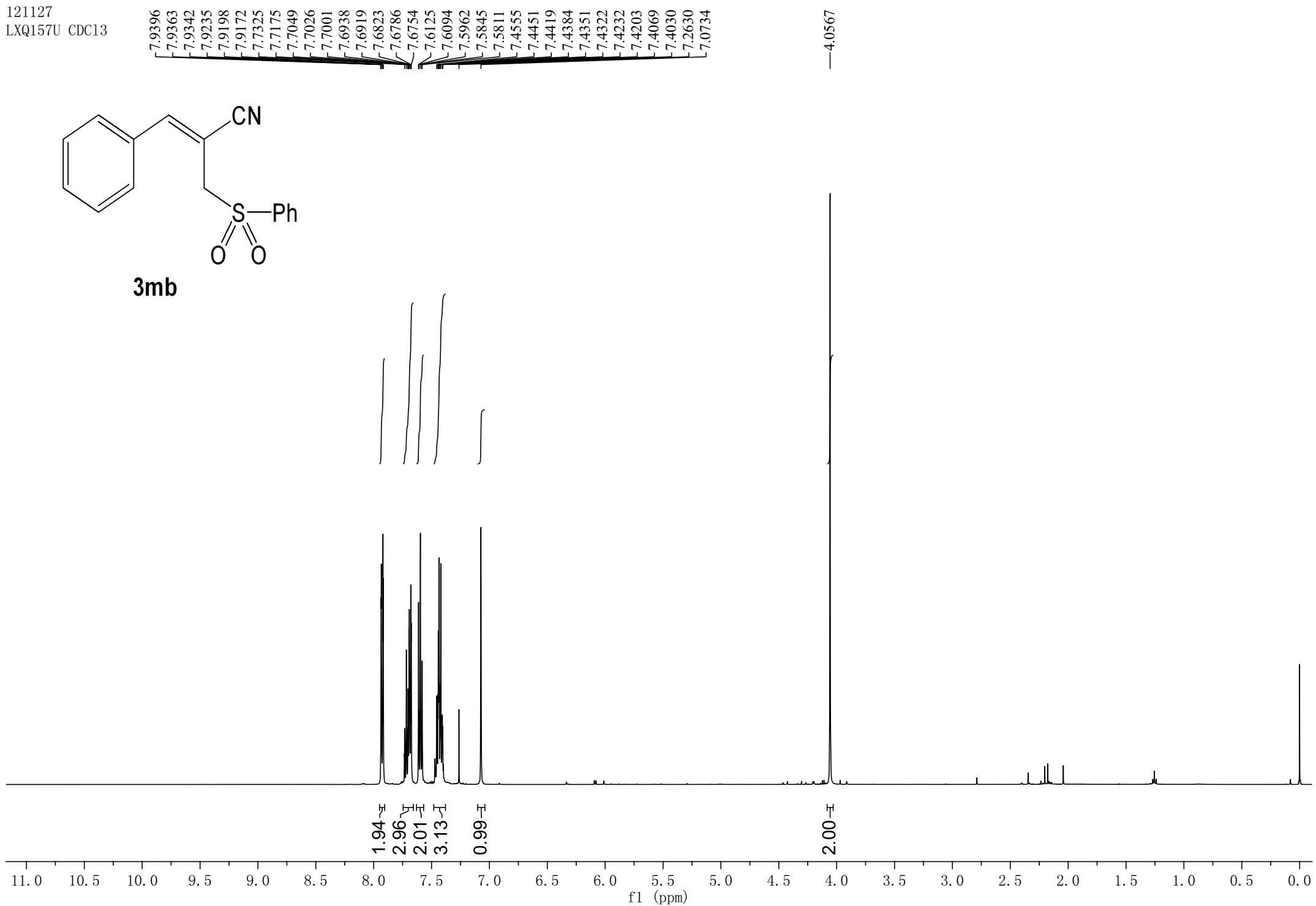
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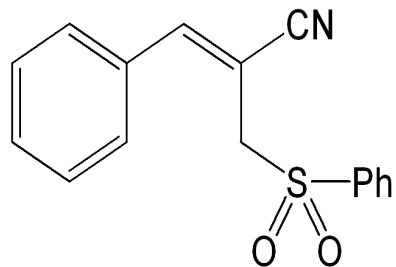
121127
LXQ157U CDC13



3mb

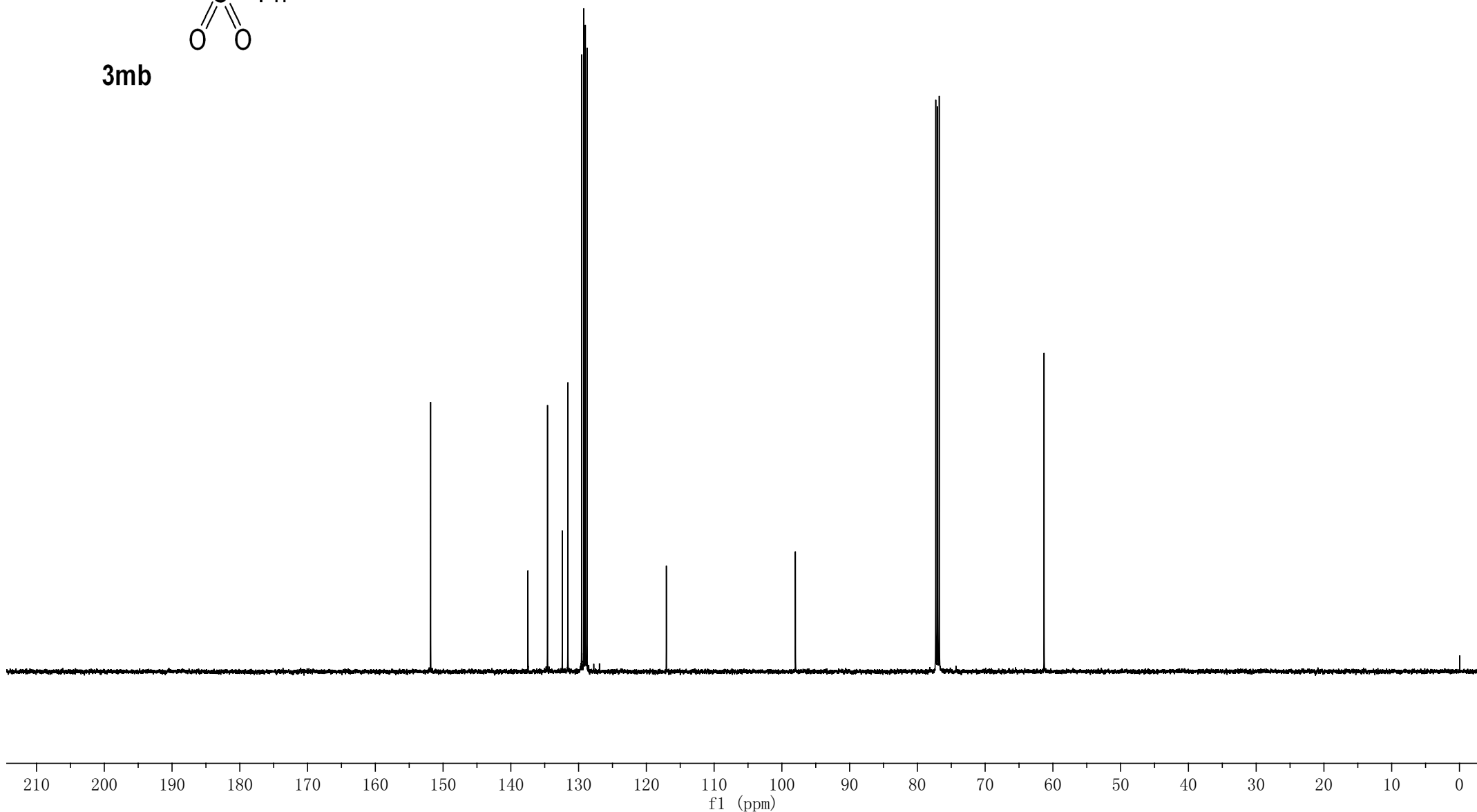


121127
LXQ157U CDC13



3mb

— 151.8355
— 137.4771
— 134.5845
— 132.4061
— 131.5764
— 129.5407
— 129.2279
— 129.0006
— 128.7601
— 117.0463
— 98.0083
— 77.2860
— 77.0316
— 76.7772
— 61.3016

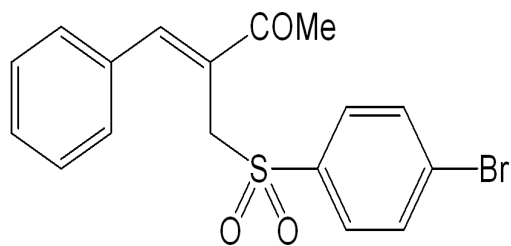


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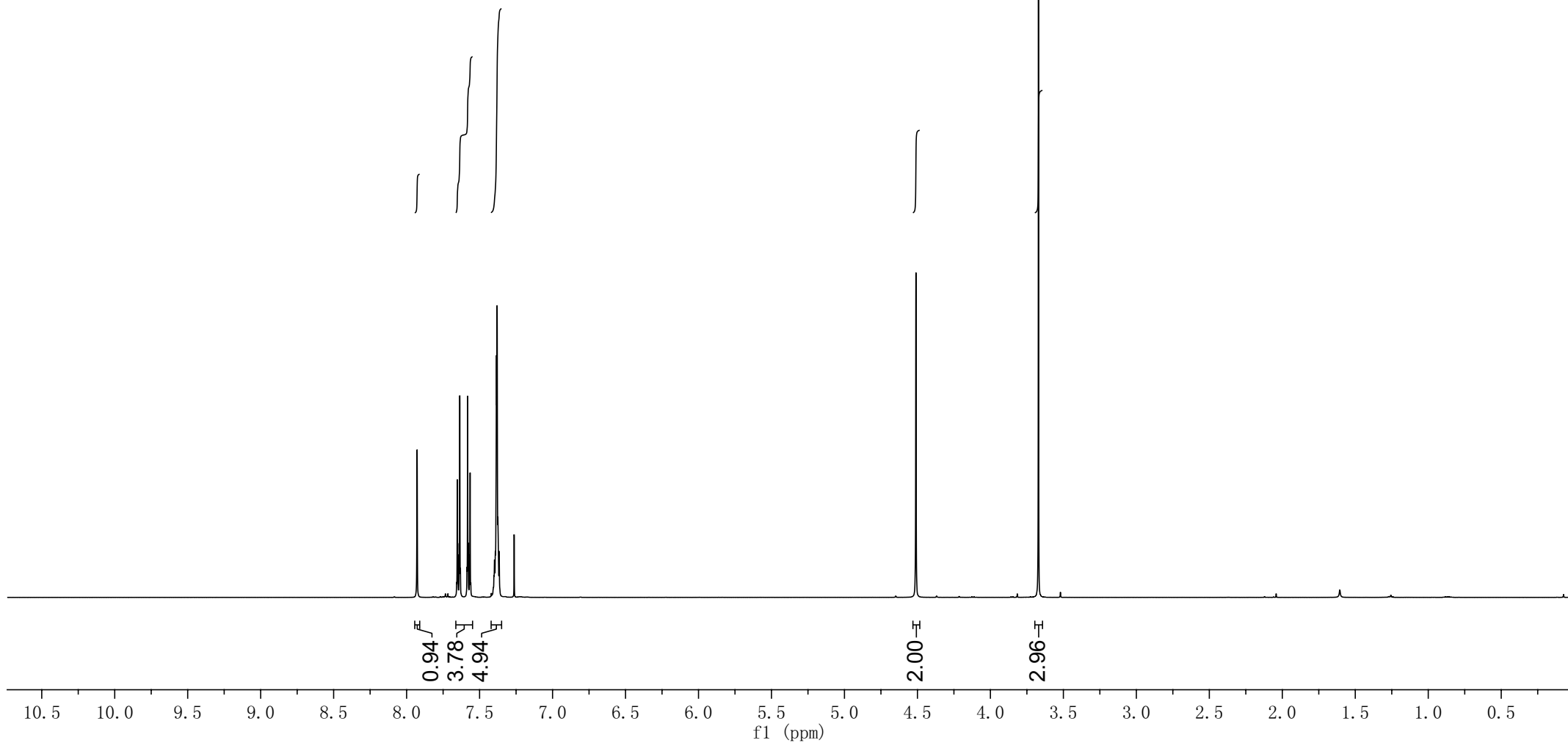
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7.2631

4.5102

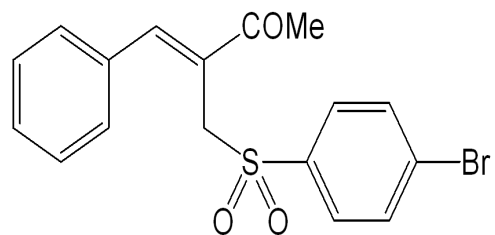
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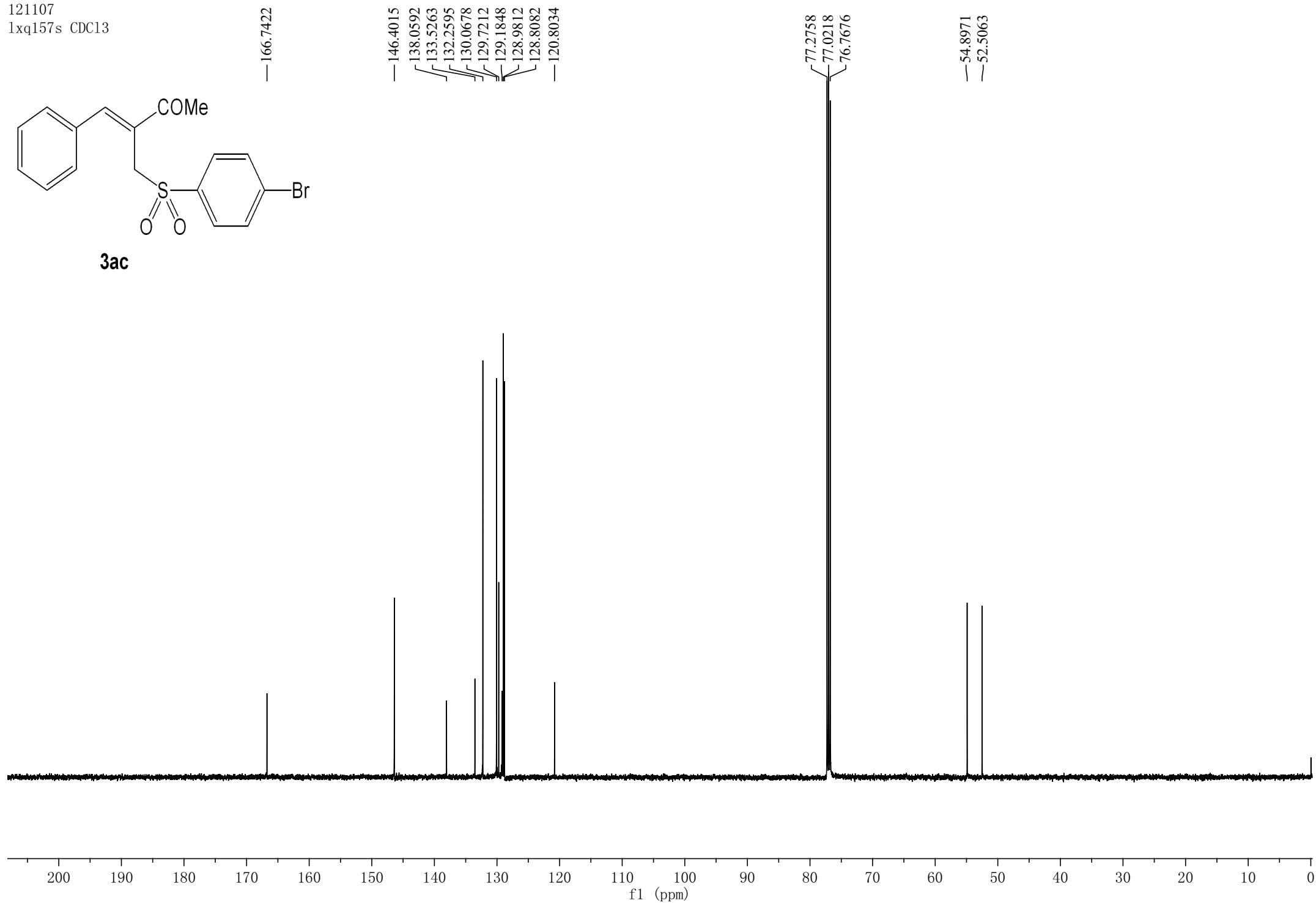
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121107
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3ac



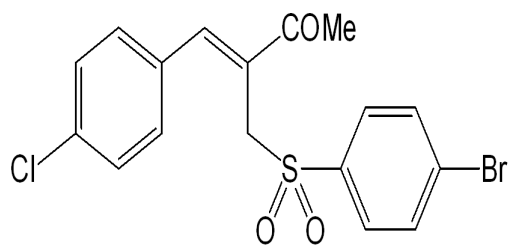
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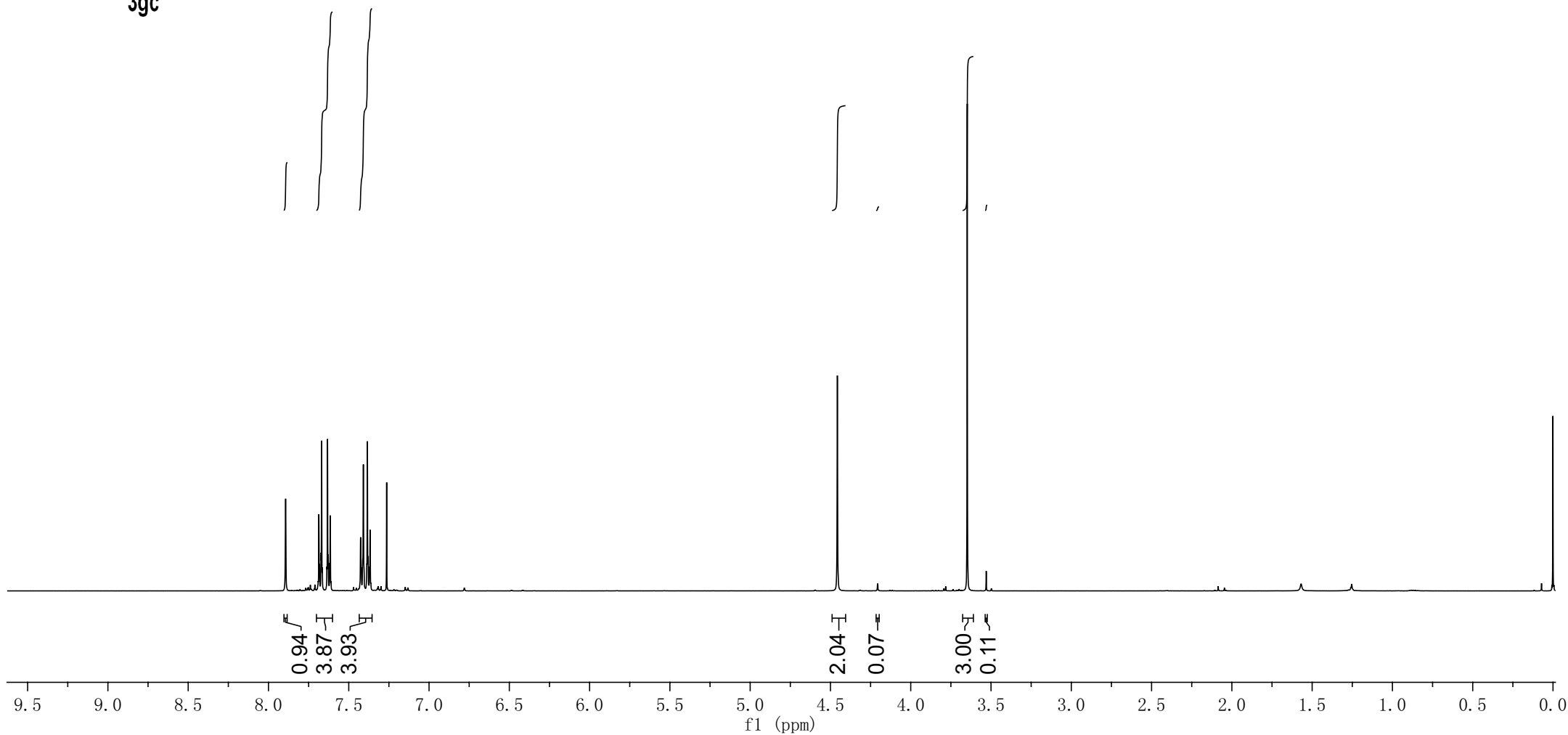
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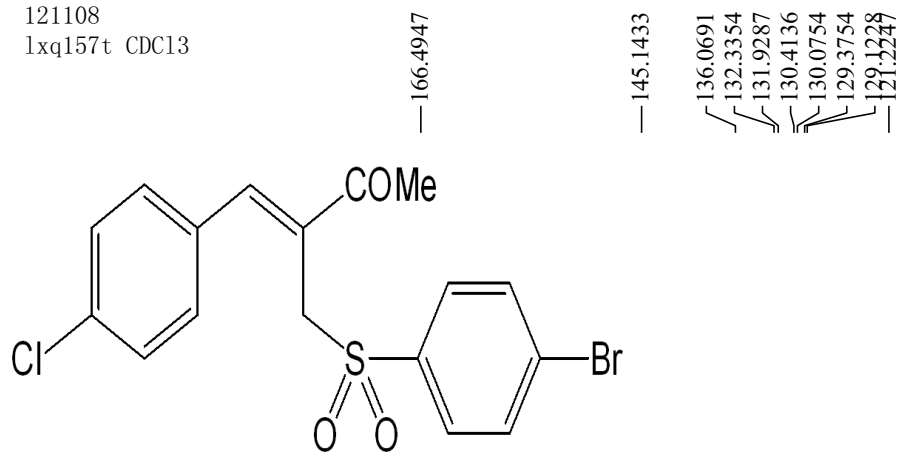
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3gc



121108
lxq157t CDC13



3gc

