Generation of benzoimidazo[1,5-a]imidazoles via a copper-catalyzed tandem reaction of carbodiimide and isocyanoacetate

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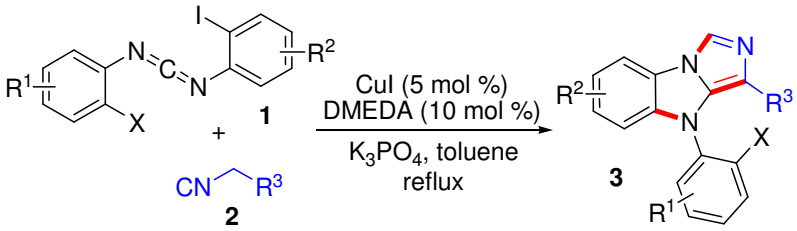
Supporting Information

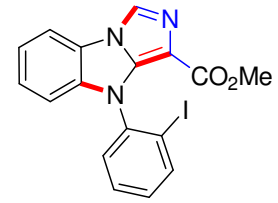
General Materials and Methods:

Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light. Organic solutions were concentrated on rotary evaporators at ~20 Torr at 25–35°C. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on a Bruker DRX-400 spectrometer operating at 400 MHz and 100 MHz, respectively. All chemical shift values are quoted in ppm and coupling constants quoted in Hz. High resolution mass spectrometry (HRMS) spectra were obtained on a micrOTOF II Instrument.

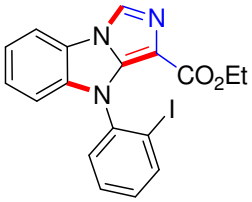
*General procedure of the synthesis of benzoimidazo[1,5-a]imidazoles **3** via a copper-catalyzed tandem reaction of carbodiimide **1** and isocyanoacetate **2***



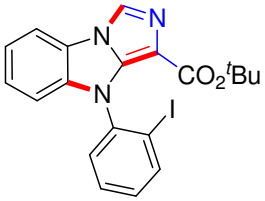
Copper(I) iodide (10 mol %), DMEDA (20 mol %), and K_3PO_4 (2 equiv) were added to a solution of carbodiimide **1** (0.5 mmol) in toluene (5 mL) under N_2 atmosphere at room temperature. After 2 minutes, isocyanoacetate (1.2 euqiv) was added, and the mixture was stirred at reflux. After completion of reaction as indicated by TLC (8-12 hrs), the solvent was evaporated. The residue was purified on silica gel providing benzoimidazo[1,5-a]imidazole **3**.



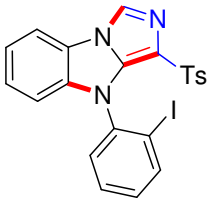
Benzoimidazo[1,5-a]imidazole **3a**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.49 (s, 3H), 6.92 (d, *J* = 7.2 Hz, 1H), 7.27-7.34 (m, 3H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.54 (d, *J* = 7.6 Hz, 1H), 7.77 (d, *J* = 7.6 Hz, 1H), 8.01-8.04 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.7, 98.7, 106.1, 111.3, 111.8, 120.7, 121.8, 122.7, 125.4, 128.9, 129.8, 130.7, 138.4, 138.6, 139.3, 162.1; HRMS Calcd for $C_{17}H_{13}IN_3O_2^+$ (ESI, M+H$^+$): 418.0047; found: 418.0057.



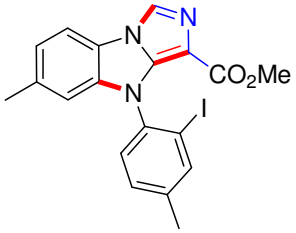
Benzoimidazo[1,5-a]imidazole **3b**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (t, *J* = 6.4 Hz, 3H), 3.97-4.01 (m, 2H), 6.90 (d, *J* = 7.6 Hz, 1H), 7.23-7.41 (m, 3H), 7.47-7.55 (m, 2H), 7.75 (d, *J* = 7.6 Hz, 1H), 7.99-8.04 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 59.5, 99.0, 111.6, 111.8, 120.3, 120.8, 121.9, 122.3, 125.6, 129.2, 129.8, 130.3, 130.8, 138.6, 139.6, 140.4, 162.1; HRMS Calcd for $C_{18}H_{15}IN_3O_2^+$ (ESI, M+H$^+$): 432.0203; found: 432.0202.



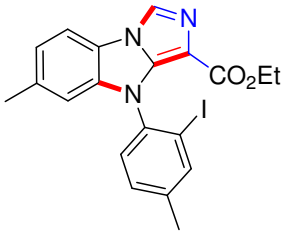
Benzoimidazo[1,5-a]imidazole **3c**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.26 (s, 3H), 6.85 (d, *J* = 7.6 Hz, 1H), 7.22-7.30 (m, 3H), 7.46-7.53 (m, 2H), 7.75 (d, *J* = 7.6 Hz, 1H), 7.97-8.04 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.1, 50.8, 98.6, 109.3, 111.7, 111.8, 120.3, 121.9, 122.3, 125.4, 129.3, 129.7, 129.8, 130.7, 139.2, 140.0, 140.4, 162.3; HRMS Calcd for $C_{20}H_{19}IN_3O_2^+$ (ESI, M+H$^+$): 460.0516; found: 460.0505.



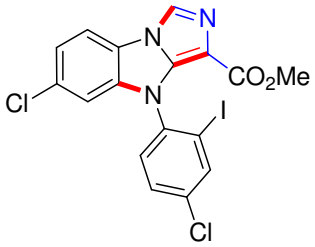
Benzoimidazo[1,5-a]imidazole **3d**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.67 (s, 3H), 6.61 (d, *J* = 7.6 Hz, 1H), 6.89-6.92 (m, 1H), 7.07-7.30 (m, 9H), 7.47 (d, *J* = 7.6 Hz, 1H), 7.89-7.94 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 98.6, 109.4, 116.8, 120.4, 122.3, 129.8, 130.4, 130.8, 135.8, 140.4, 141.8, 156.3; HRMS Calcd for $C_{22}H_{17}IN_3O_2S^+$ (ESI, $M+H^+$): 514.0081; found: 514.0084.



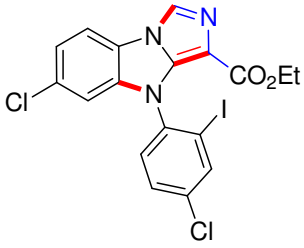
Benzoimidazo[1,5-a]imidazole **3e**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.41 (s, 3H), 2.45 (s, 3H), 3.49 (s, 3H), 6.69 (s, 1H), 7.10 (d, *J* = 8.0 Hz, 1H), 7.33-7.35 (m, 2H), 7.60 (d, *J* = 7.6 Hz, 1H), 7.87 (s, 1H), 7.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.8, 21.7, 50.9, 98.7, 106.3, 111.4, 111.6, 120.6, 120.9, 122.7, 129.4, 129.8, 136.0, 139.2, 139.8, 162.4; HRMS Calcd for $C_{19}H_{17}IN_3O_2^+$ (ESI, $M+H^+$): 446.0360; found: 446.0347.



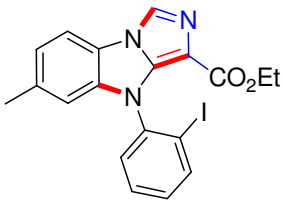
Benzoimidazo[1,5-a]imidazole **3f**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.05 (t, *J* = 6.4 Hz, 3H), 2.41 (s, 3H), 2.45 (s, 3H), 4.02-4.05 (m, 2H), 6.67 (s, 1H), 7.09 (d, *J* = 7.6 Hz, 1H), 7.27-7.33 (m, 2H), 7.60 (d, *J* = 8.0 Hz, 1H), 7.86 (s, 1H), 7.95 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4, 20.7, 21.7, 59.5, 98.7, 111.4, 111.6, 122.6, 123.2, 129.4, 129.8, 130.5, 135.9, 136.2, 139.2, 139.9, 140.6, 141.2, 162.0; HRMS Calcd for $C_{20}H_{19}IN_3O_2^+$ (ESI, $M+H^+$): 460.0516; found: 460.0508.



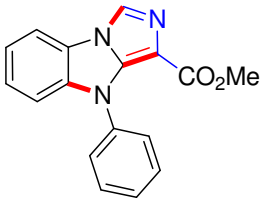
Benzoimidazo[1,5-a]imidazole **3g**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.59 (s, 3H), 6.90 (s, 1H), 7.28-7.31 (m, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.69 (d, *J* = 8.8 Hz, 1H), 7.98 (s, 1H), 8.04 (s,1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 51.1, 99.0, 106.9, 111.6, 112.8, 121.7, 122.5, 129.5, 130.4, 131.6, 136.2, 139.1, 139.4, 162.1; HRMS Calcd for $C_{17}H_{11}Cl_2IN_3O_2^+$ (ESI, M+H$^+$): 485.9268; found: 485.9250.



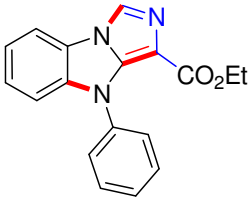
Benzoimidazo[1,5-a]imidazole **3h**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.12 (t, *J* = 6.4 Hz, 3H),4.07-4.10 (m, 2H), 6.87 (s, 1H), 7.27-7.31 (m, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.98 (s, 1H), 8.04 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 59.8, 99.2, 107.3, 111.6, 112.8, 120.9, 121.7, 122.4, 129.6, 130.4, 131.6, 136.2, 136.7, 139.2, 161.7; HRMS Calcd for $C_{18}H_{13}Cl_2IN_3O_2^+$ (ESI, M+H$^+$): 499.9424; found: 499.9414.



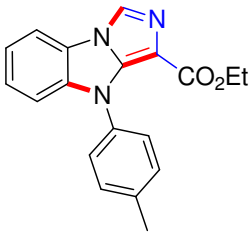
Benzoimidazo[1,5-a]imidazole **3i**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.12 (t, *J* = 6.4 Hz, 3H), 2.41 (s, 3H), 3.98-4.10 (m, 2H), 6.86 (s, 1H), 6.90-7.98 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 21.7, 59.7, 99.9, 109.7, 111.6, 111.7, 122.0, 122.9, 125.7, 129.6, 130.9, 132.4, 134.7, 136.3, 139.8, 140.2, 140.8, 161.7; HRMS Calcd for $C_{19}H_{17}Cl_2IN_3O_2^+$ (ESI, M+H$^+$): 446.0360; found: 446.0349.



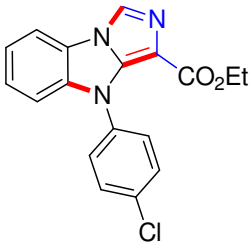
Benzoimidazo[1,5-a]imidazole **3j**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.61 (s, 3H), 7.11 (d, *J* = 8.0 Hz, 1H), 7.20-7.29 (m, 2H), 7.40 (d, *J* = 7.6 Hz, 2H), 7.46-7.61 (m, 3H), 7.68 (d, *J* = 7.6 Hz, 1H), 7.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 50.7, 105.5, 111.1, 111.7, 120.5, 121.7, 122.8, 125.4, 127.3, 128.5, 128.8, 135.6, 139.3, 141.4, 162.1; HRMS Calcd for $C_{17}H_{13}N_3NaO_2^+$ (ESI, M+Na$^+$): 314.0900; found: 314.0883.



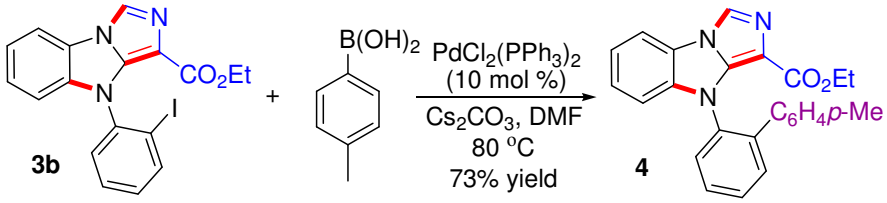
Benzoimidazo[1,5-a]imidazole **3k**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.20 (t, *J* = 6.4 Hz, 3H), 4.03-4.08 (m, 3H), 7.11 (d, *J* = 8.0 Hz, 1H), 7.20-7.28 (m, 2H), 7.29-7.52 (m, 5H), 7.68 (d, *J* = 7.6 Hz, 1H), 7.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.6, 59.9, 106.1, 111.7, 112.7, 120.7, 121.9, 123.3, 125.8, 127.5, 130.0, 133.6, 138.9, 140.1, 162.4; HRMS Calcd for $C_{18}H_{16}N_3O_2^+$ (ESI, M+H$^+$): 306.1237; found: 306.1258.



Benzoimidazo[1,5-a]imidazole **3l**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.09 (t, *J* = 6.4 Hz, 3H), 2.47 (s, 3H), 4.08-4.11 (m, 3H), 7.16 (d, *J* = 8.0 Hz, 1H), 7.34-7.35 (m, 6H), 7.72 (d, *J* = 7.2 Hz, 1H), 7.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 21.3, 59.6, 105.8, 111.4, 111.7, 120.5, 121.7, 122.9, 125.5, 127.2, 129.7, 133.3, 138.6, 139.8, 162.1; HRMS Calcd for $C_{19}H_{18}N_3O_2^+$ (ESI, M+H$^+$): 320.1394; found: 320.1392.

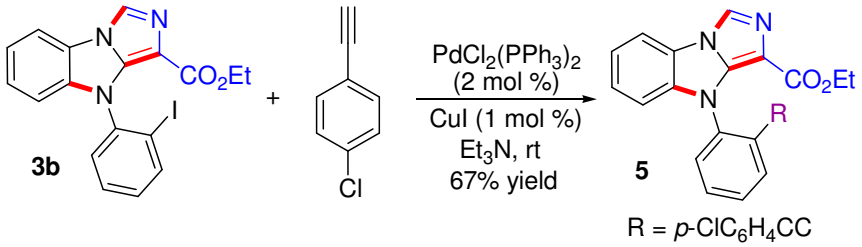


Benzoimidazo[1,5-a]imidazole **3m**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.02 (t, *J* = 6.4 Hz, 3H), 4.01-4.06 (m, 3H), 7.09 (d, *J* = 8.0 Hz, 1H), 7.19-7.21 (m, 1H), 7.23-7.28 (m, 5H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.91 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.6, 60.2, 106.5, 111.9, 112.3, 120.7, 122.2, 123.5, 126.0, 127.7, 130.2, 139.1, 140.3, 162.6; HRMS Calcd for $C_{18}H_{15}ClN_3O_2^+$ (ESI, $M+H^+$): 340.0847; found: 340.0842.



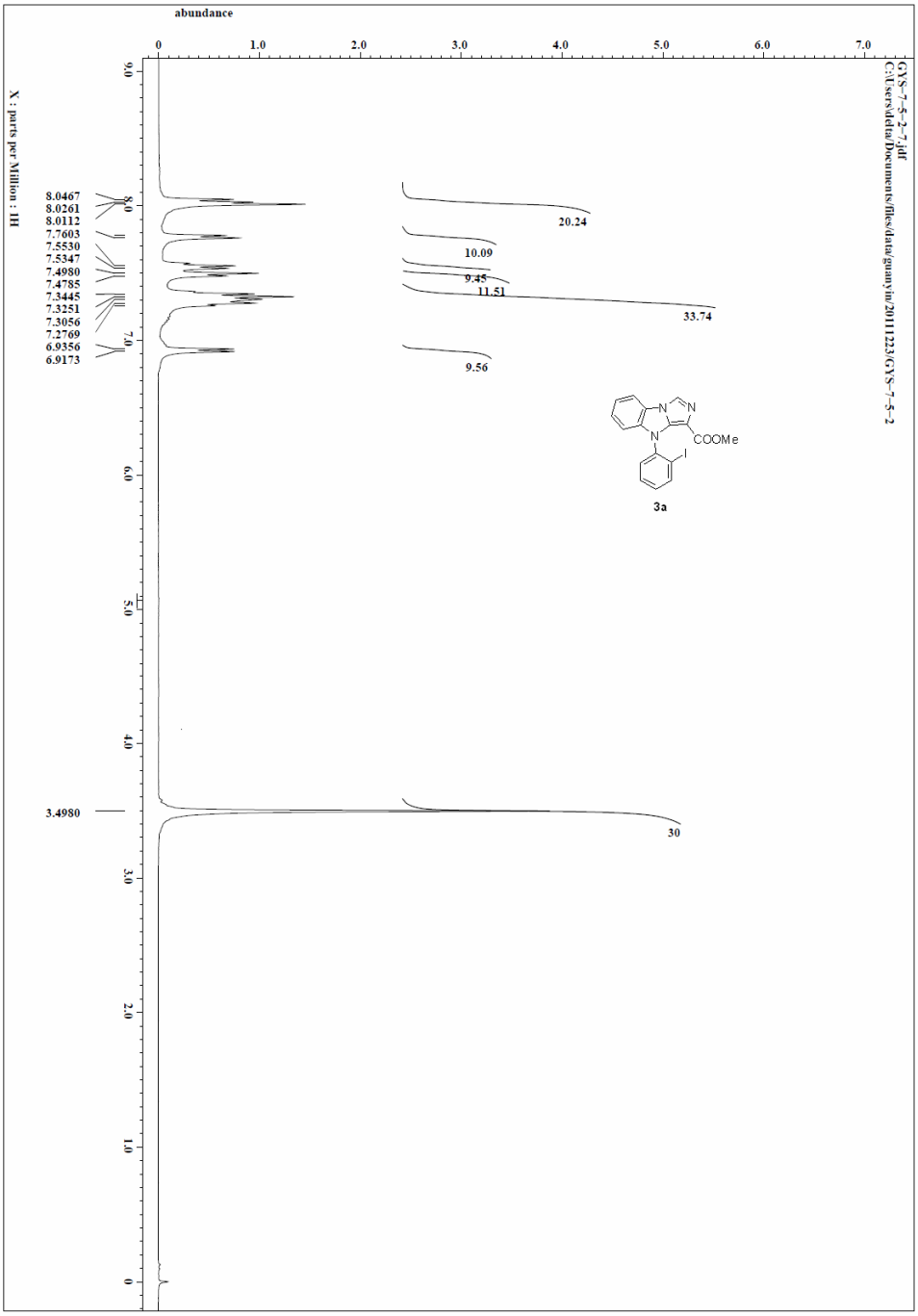
4-Methylphenylboronic acid (1.2 equiv), $PdCl_2(PPh_3)_2$ (10 mol %), and Cs_2CO_3 (2 equiv) were added to a solution of benzoimidazo[1,5-*a*]imidazole **3b** (0.2 mmol) in DMF under N_2 atmosphere at 80 °C. After completion of reaction as indicated by TLC, the solvent was evaporated. The residue was purified on silica gel providing benzoimidazo[1,5-a]imidazole **4**.

^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91 (t, *J* = 6.4 Hz, 3H), 2.16 (s, 3H), 3.99-4.04 (m, 2H), 6.87-6.91 (m, 2H), 7.01 (d, *J* = 7.2 Hz, 1H), 7.17-7.21 (m, 3H), 7.44-7.48 (m, 3H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.82 (d, *J* = 7.2 Hz, 1H), 7.92 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 20.9, 59.7, 105.7, 111.5, 111.7, 121.6, 127.7, 127.9, 128.3, 128.5, 128.6, 128.9, 130.9, 131.9, 132.0, 134.3, 141.3, 161.3; HRMS Calcd for $C_{25}H_{22}N_3O_2^+$ (ESI, $M+H^+$): 396.1707; found: 396.1715.



1-Chloro-4-ethynylbenzene (1.2 equiv), $PdCl_2(PPh_3)_2$ (2 mol %) , CuI (1 mol %), and benzoimidazo[1,5-*a*]imidazole **3b** (0.2 mmol) were added into a test tube. Dried triethylamine (2.0 mL) was added under N_2 atmosphere. The mixture was stirred at room temperature. After completion of reaction as indicated by TLC, the solvent was evaporated. The residue was purified on silica gel providing benzoimidazo[1,5-a]imidazole **5**.

^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (t, *J* = 6.4 Hz, 3H), 4.02-4.06 (m, 2H), 6.79 (d, *J* = 7.2 Hz, 1H), 7.10-7.13 (m, 2H), 7.29-7.31 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 1H), 7.54-7.57 (m, 3H), 7.70-7.72 (m, 3H), 8.01 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 59.6, 85.6, 94.1, 111.8, 120.6, 121.9, 122.9, 125.5, 128.4, 128.5, 128.7, 129.0, 131.9, 132.1, 132.4, 132.6, 134.3, 138.9, 139.6, 141.3, 162.1; HRMS Calcd for $C_{26}H_{19}ClN_3O_2^+$ (ESI, $M+H^+$): 440.1160; found: 440.1166.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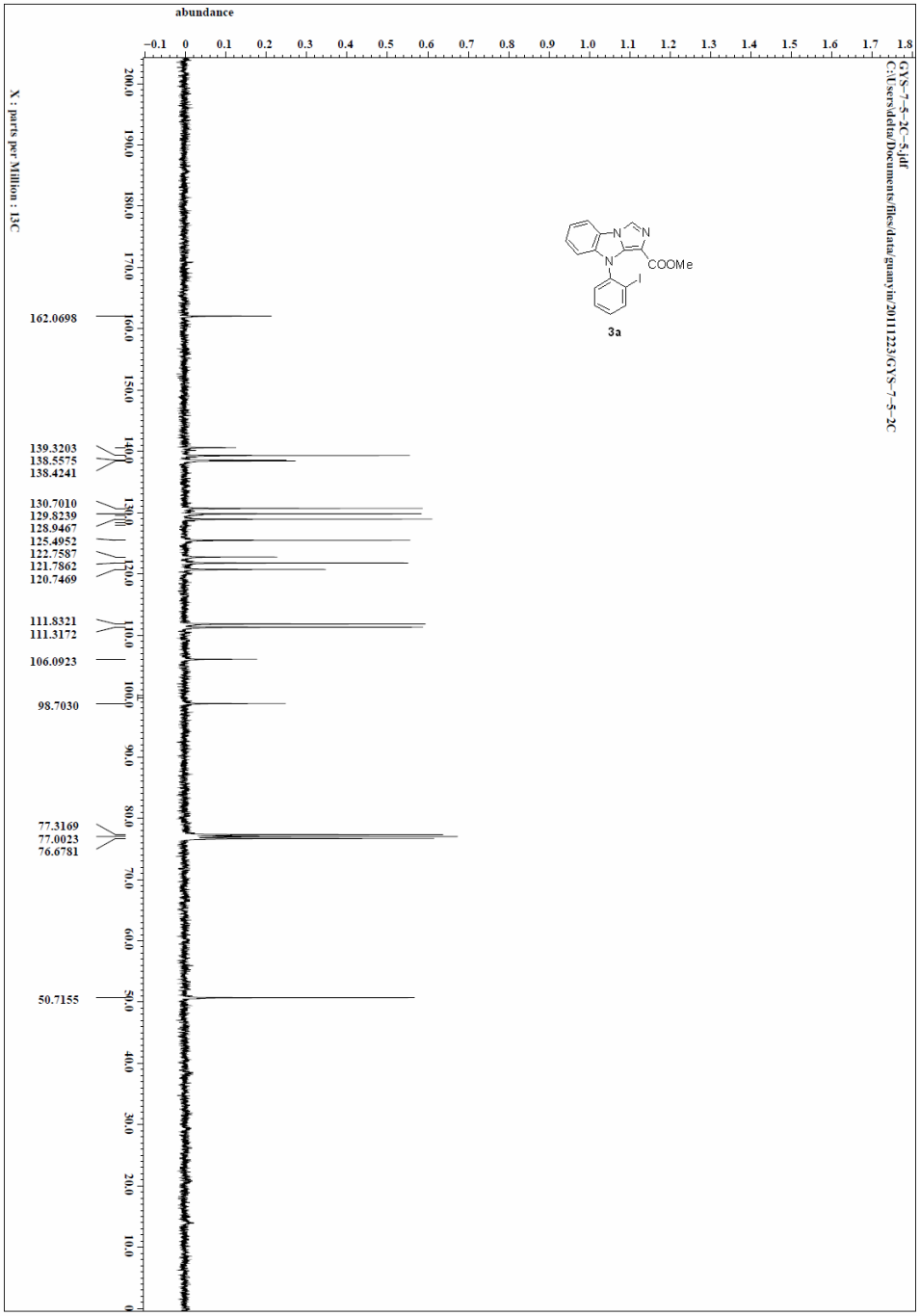
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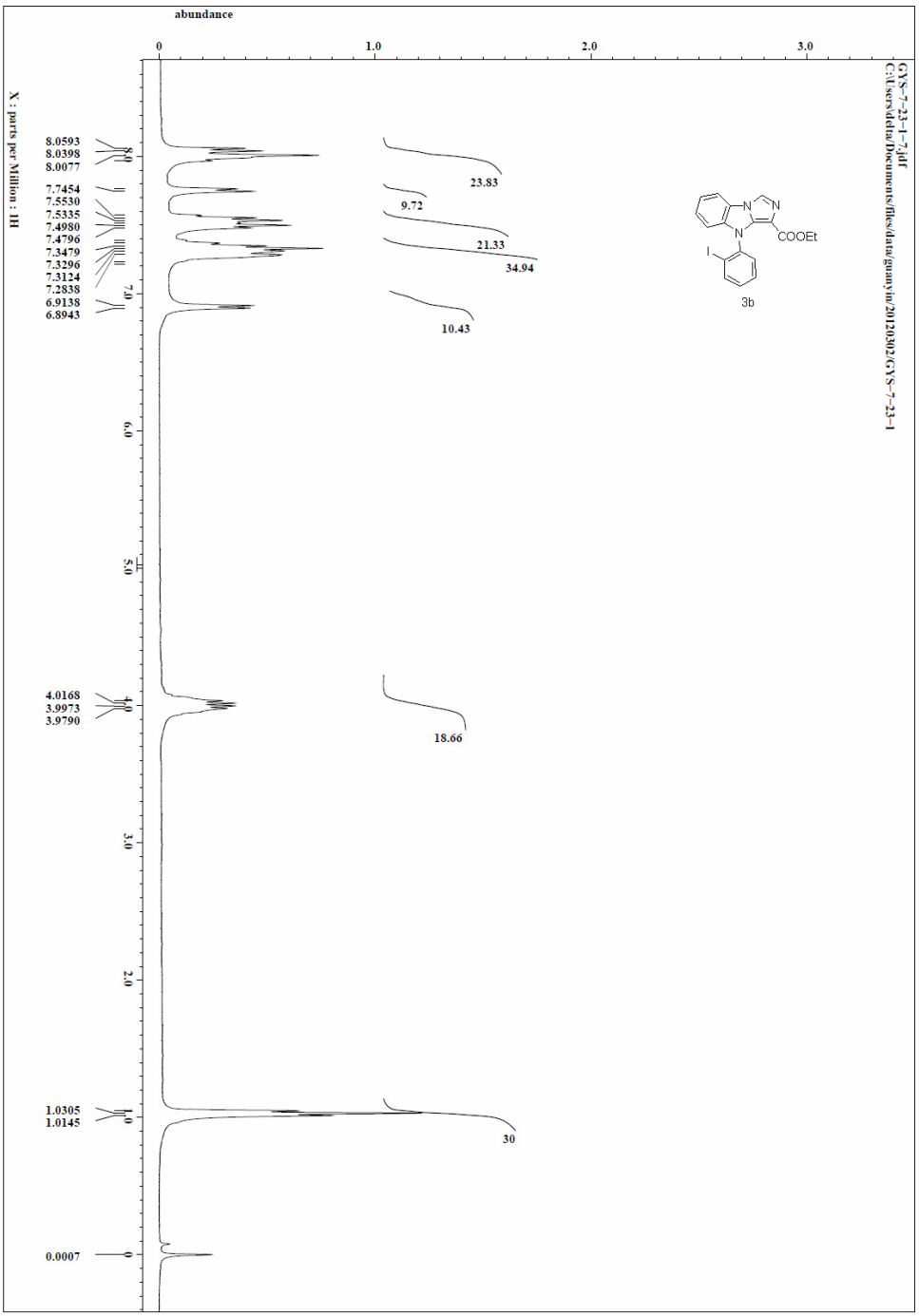
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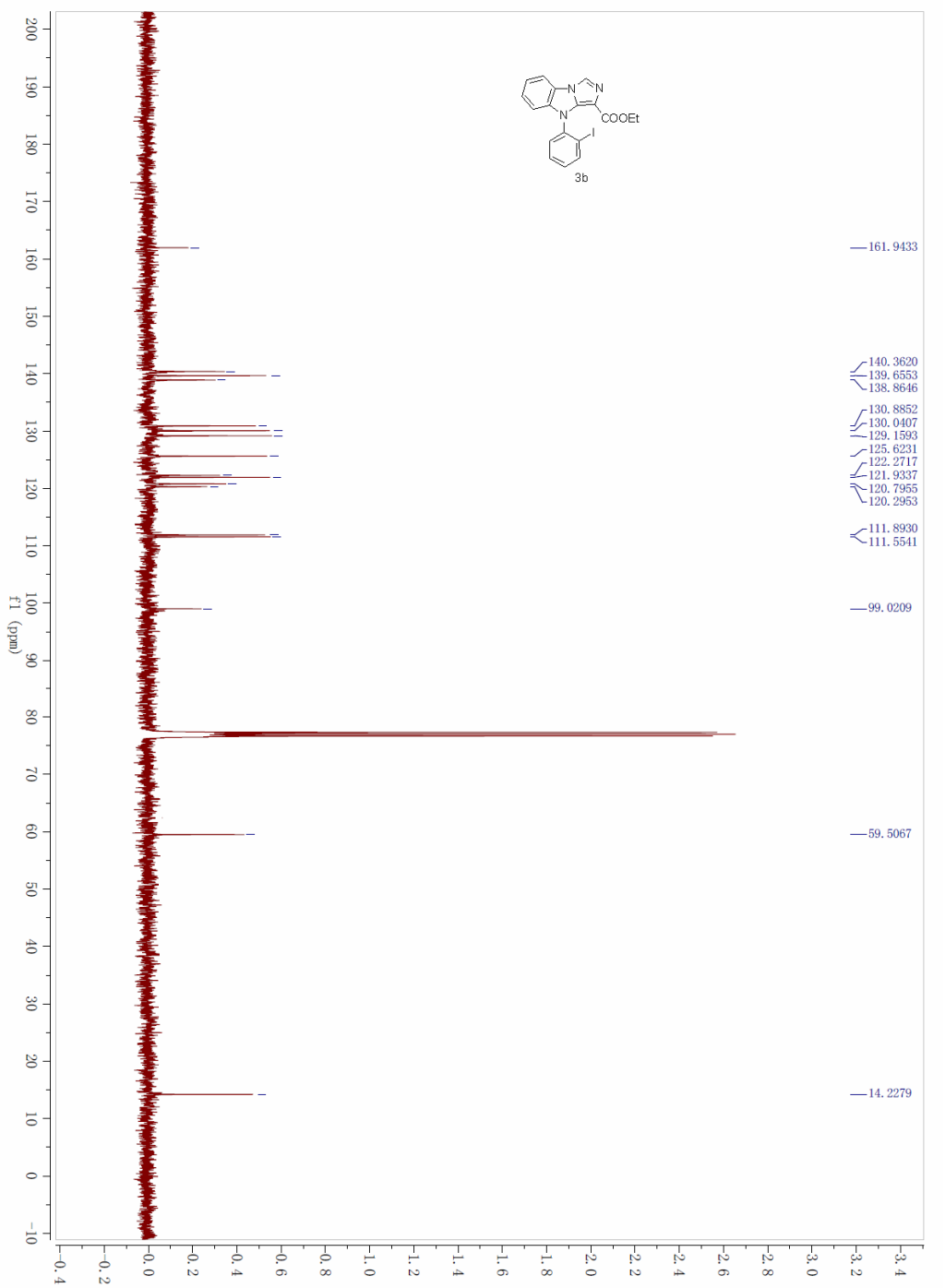
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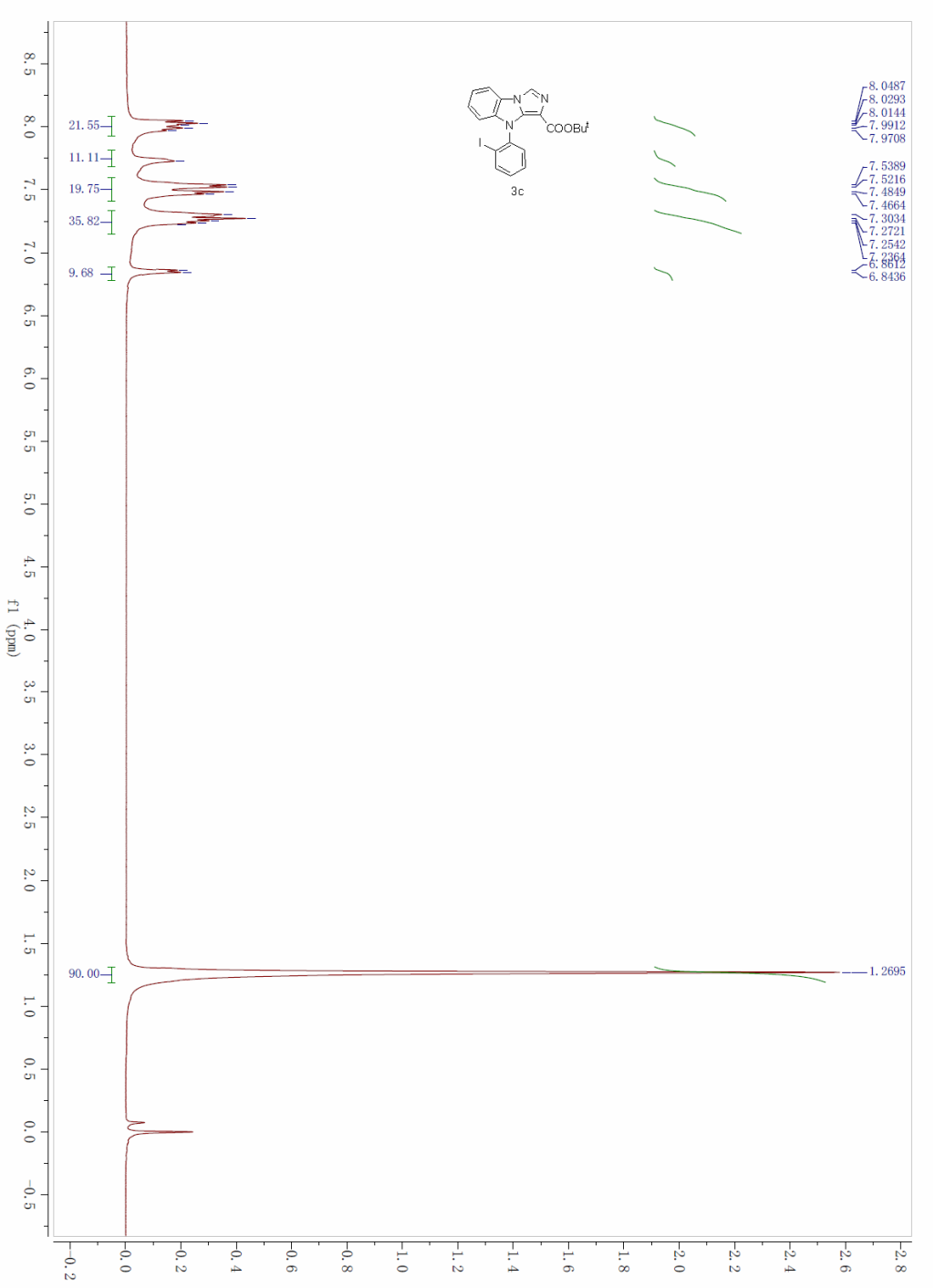
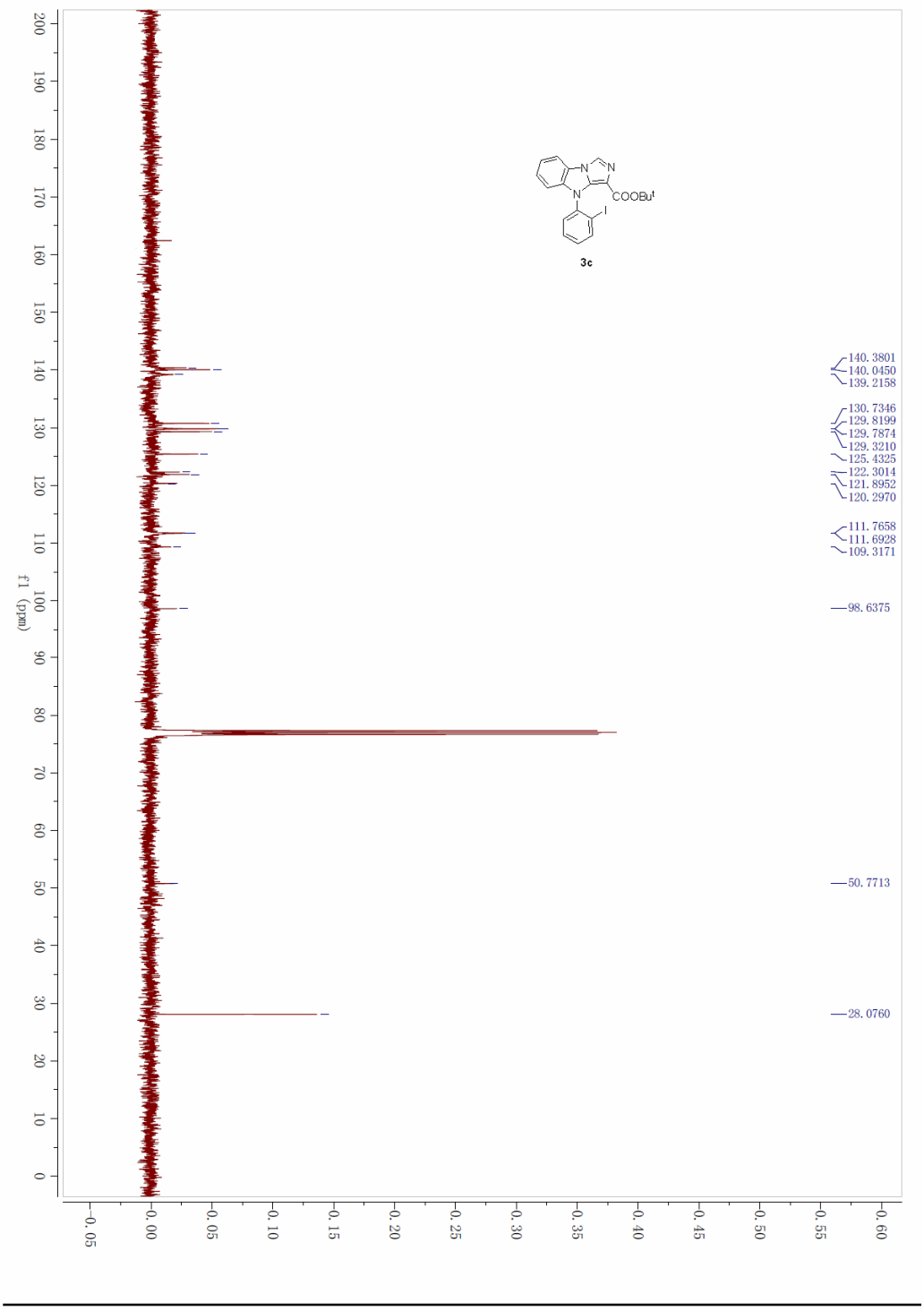
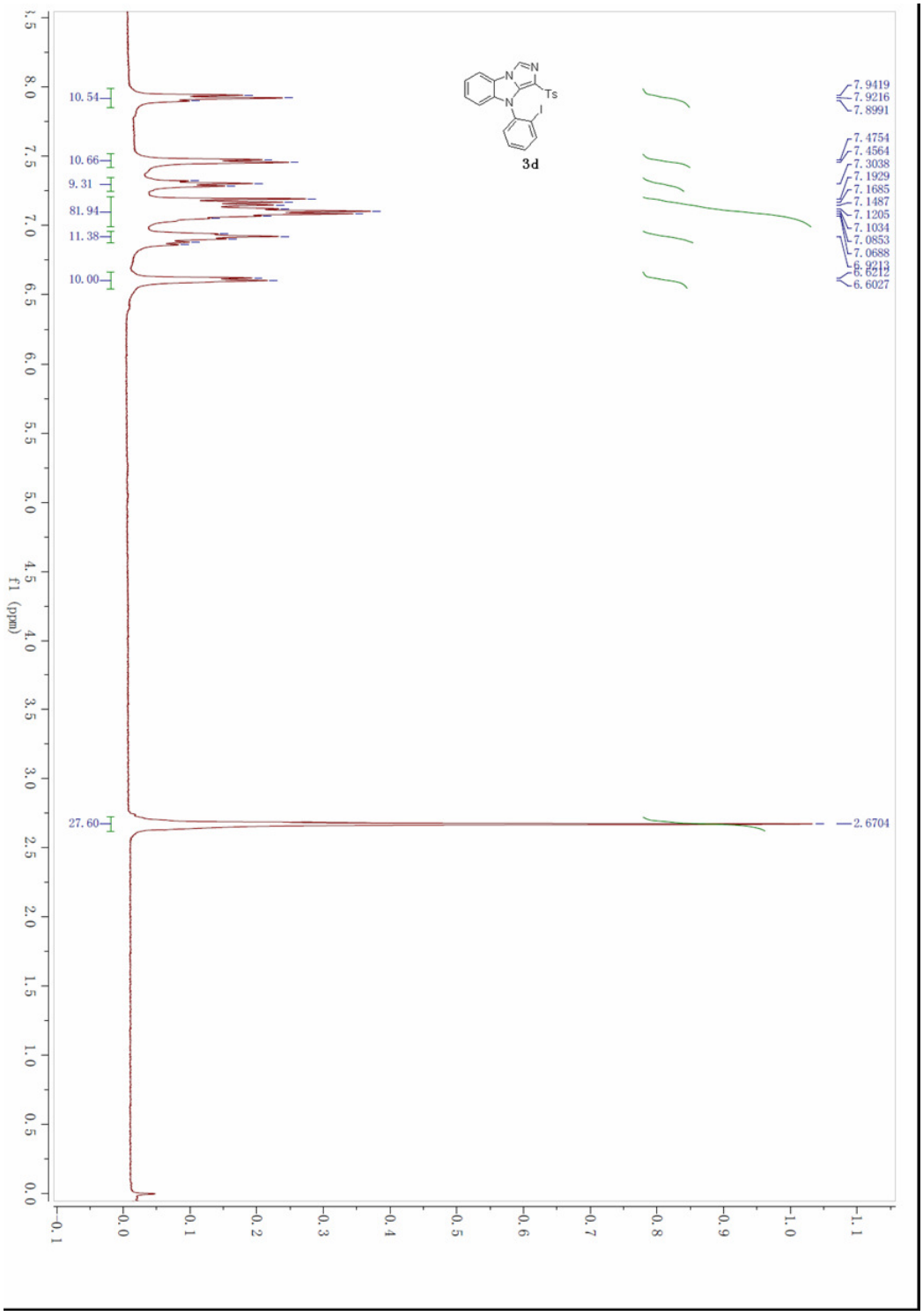
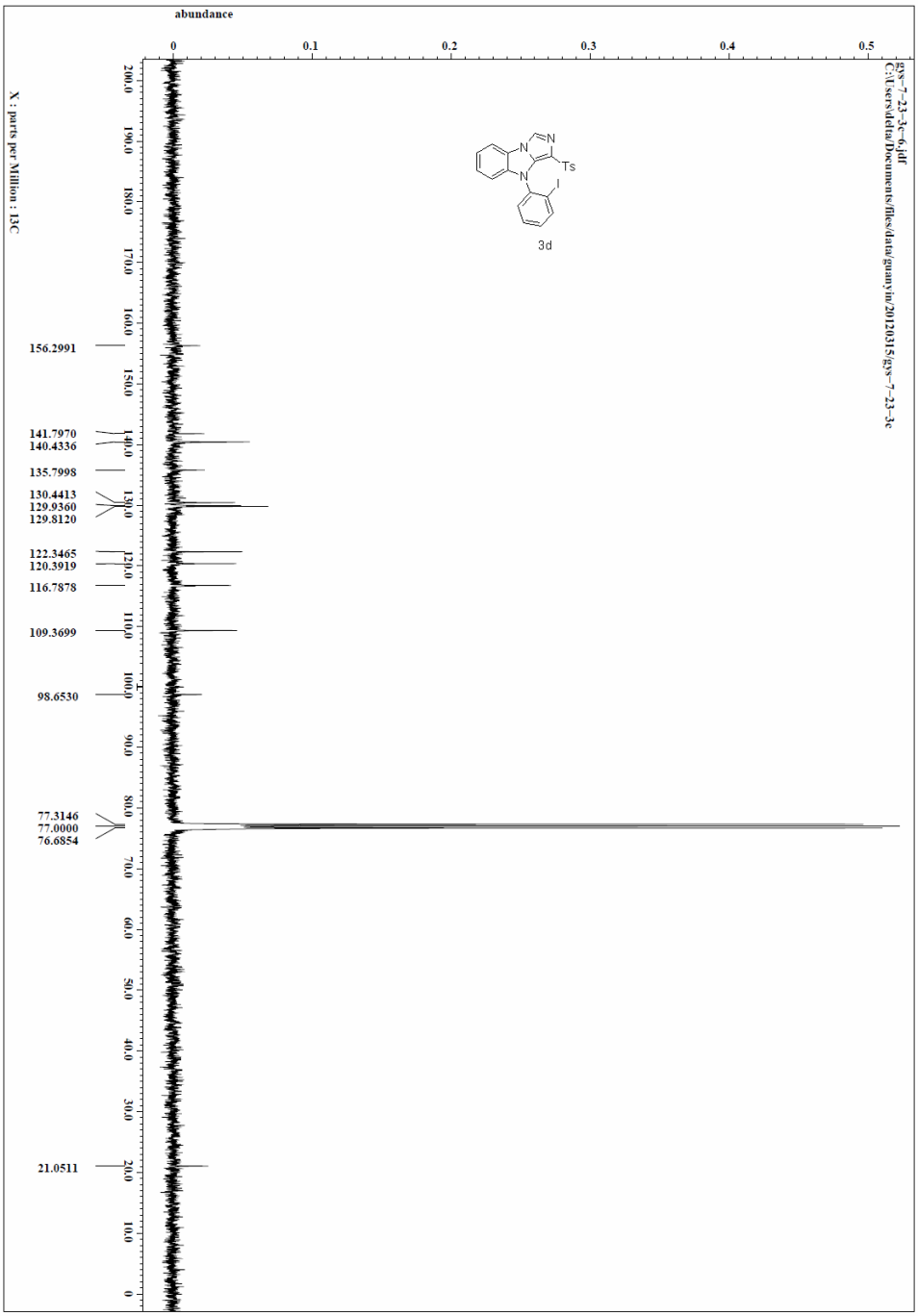
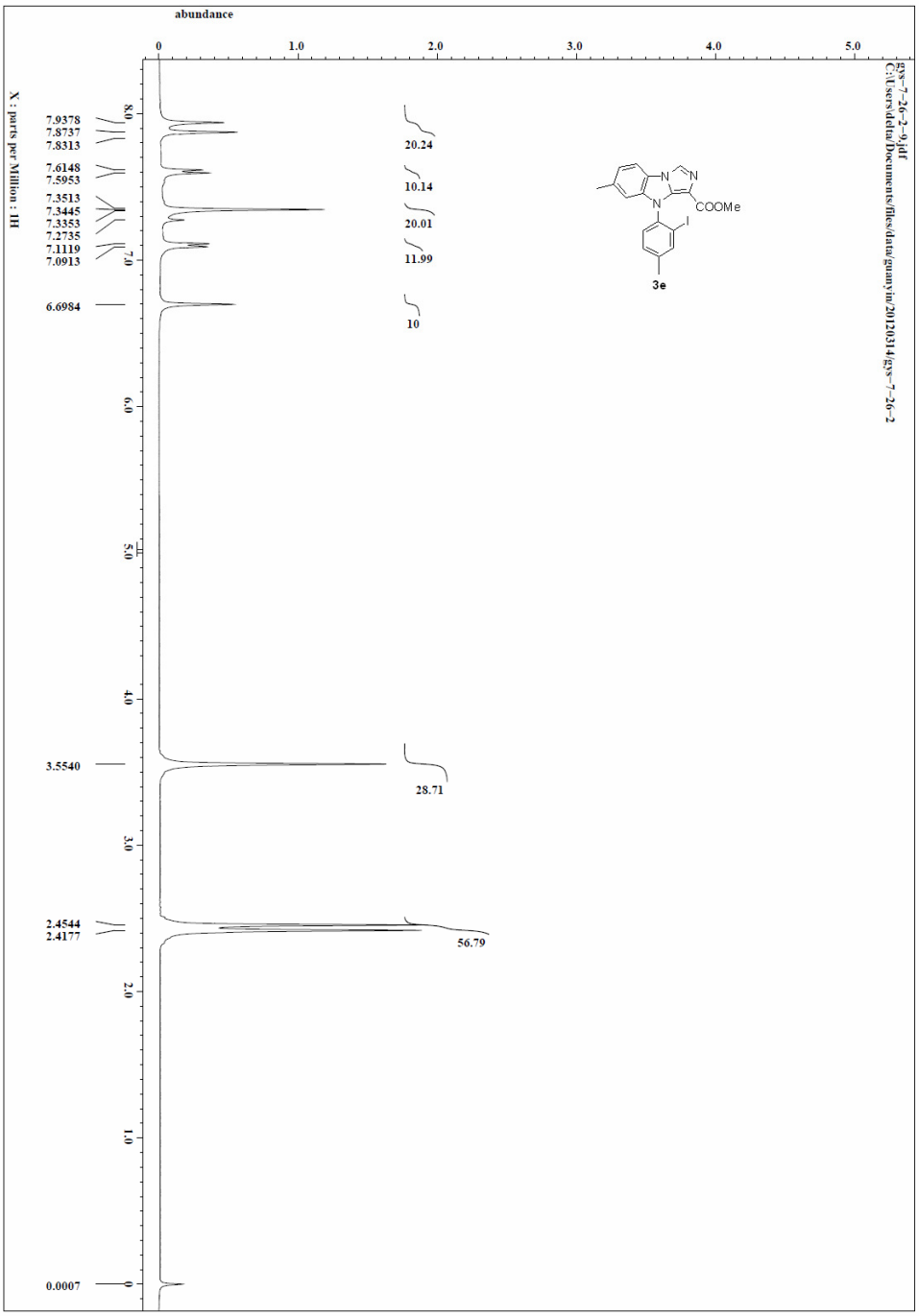
14.2279
f1 (ppm)

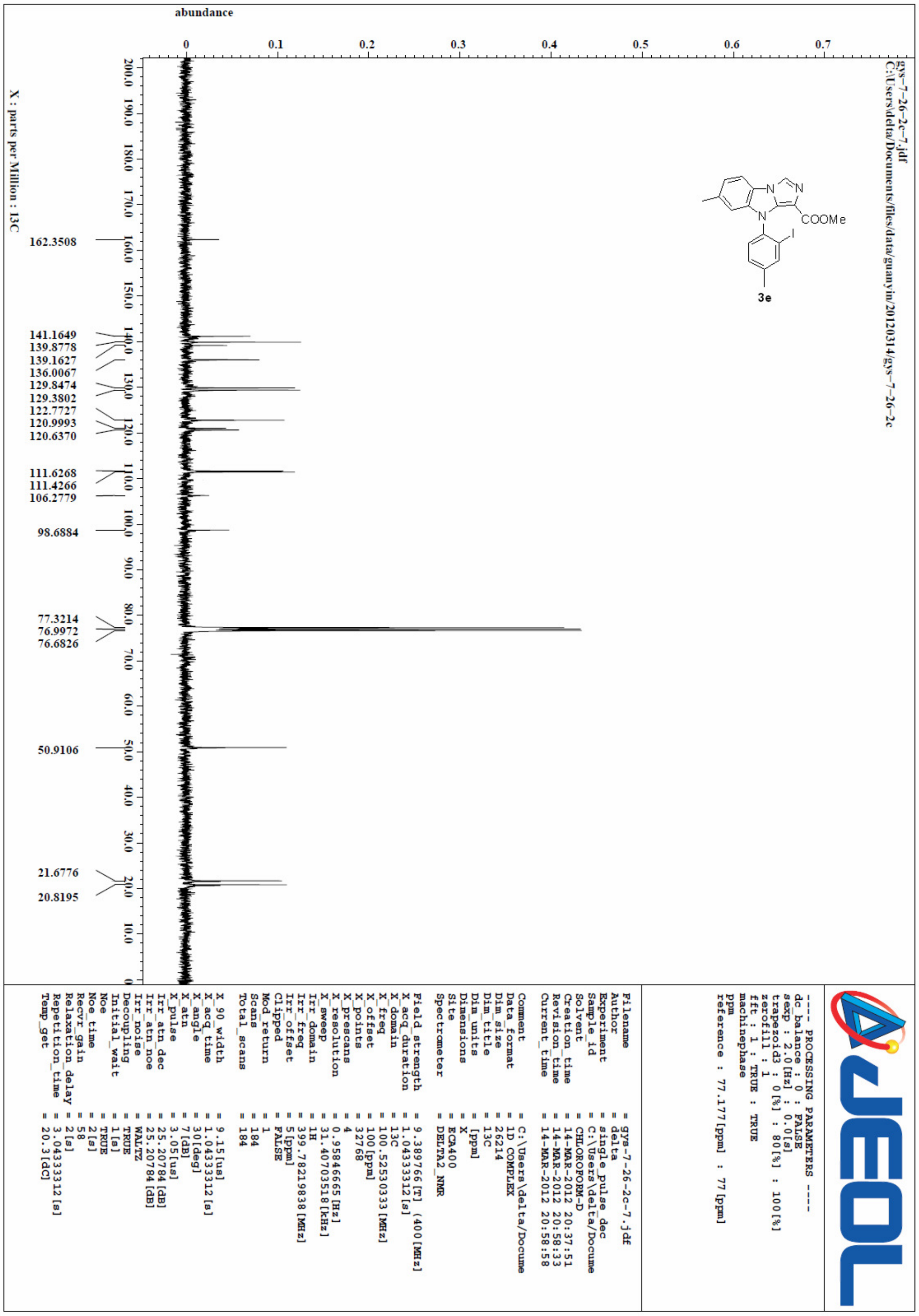

21.55
11.11
19.75
35.82
9.68
90.00
COOBu^t
3c
8.0487
8.0293
8.0144
7.9912
7.9708
7.5389
7.5216
7.4849
7.4664
7.3034
7.2721
7.2542
7.2364
6.8612
6.8436
1.2695
f1 (ppm)

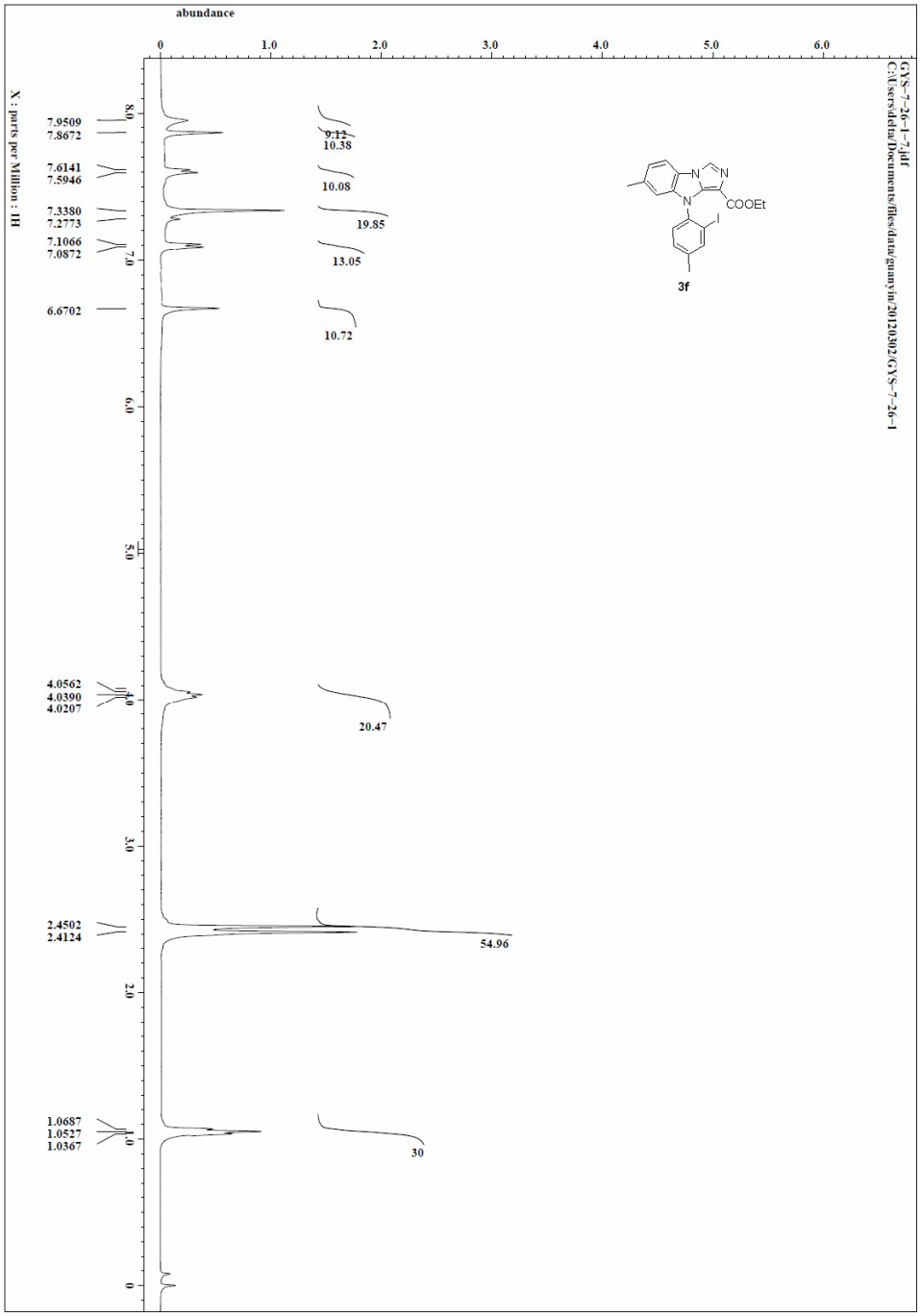

COOBu^t
I
3c
140.3801
140.0450
139.2158
130.7346
129.8199
129.7874
129.3210
125.4325
122.3014
121.8952
120.2970
111.7658
111.6928
109.3171
98.6375
50.7713
28.0760
f1 (ppm)

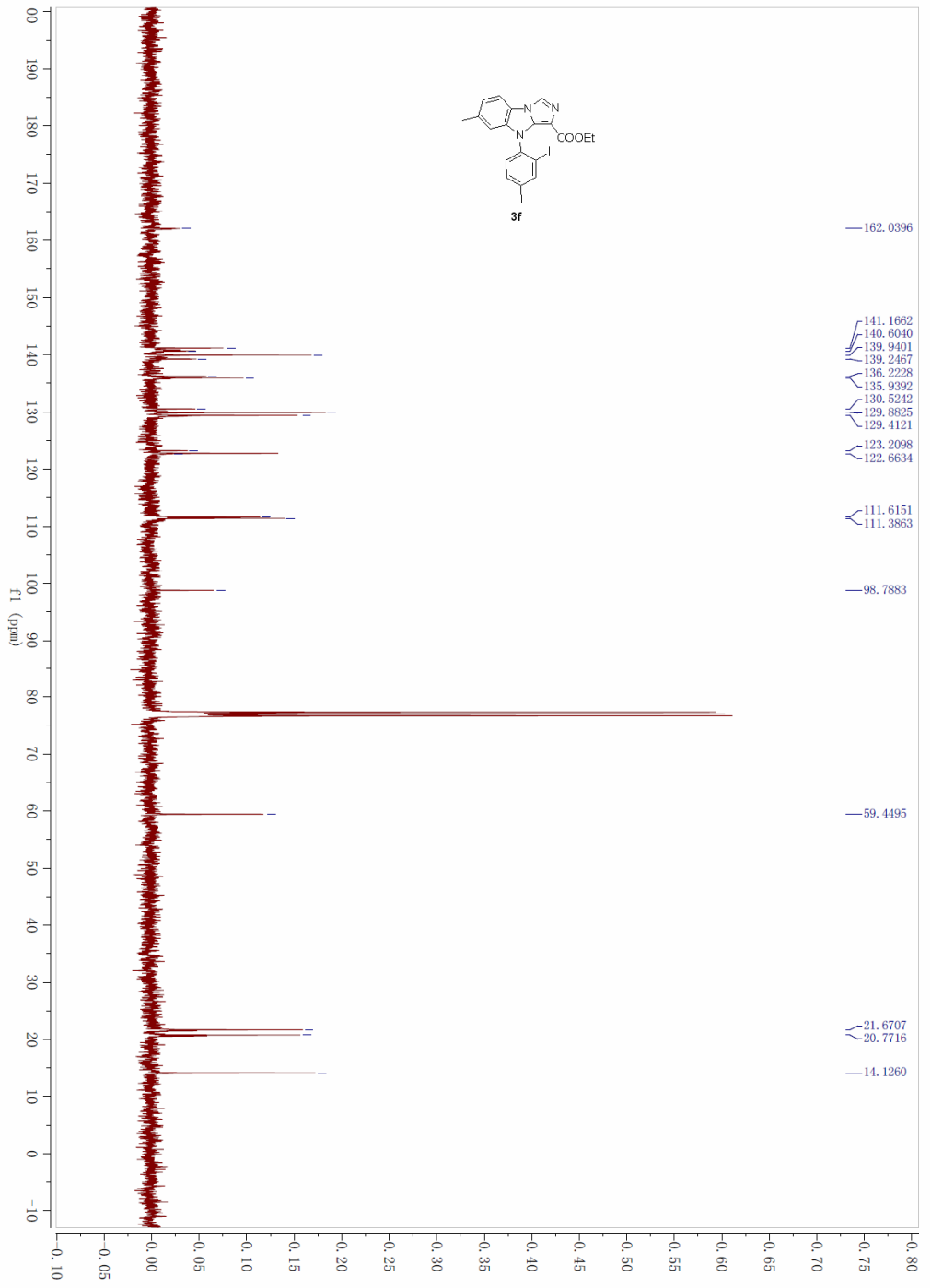

10.54
10.66
9.31
81.94
11.38
10.00
27.60
2.6704
7.9419
7.9216
7.8991
7.4754
7.4564
7.3038
7.1929
7.1685
7.1487
7.1205
7.1034
7.0853
7.0688
6.9213
6.6212
6.6027
Ts
I
3d
f1 (ppm)

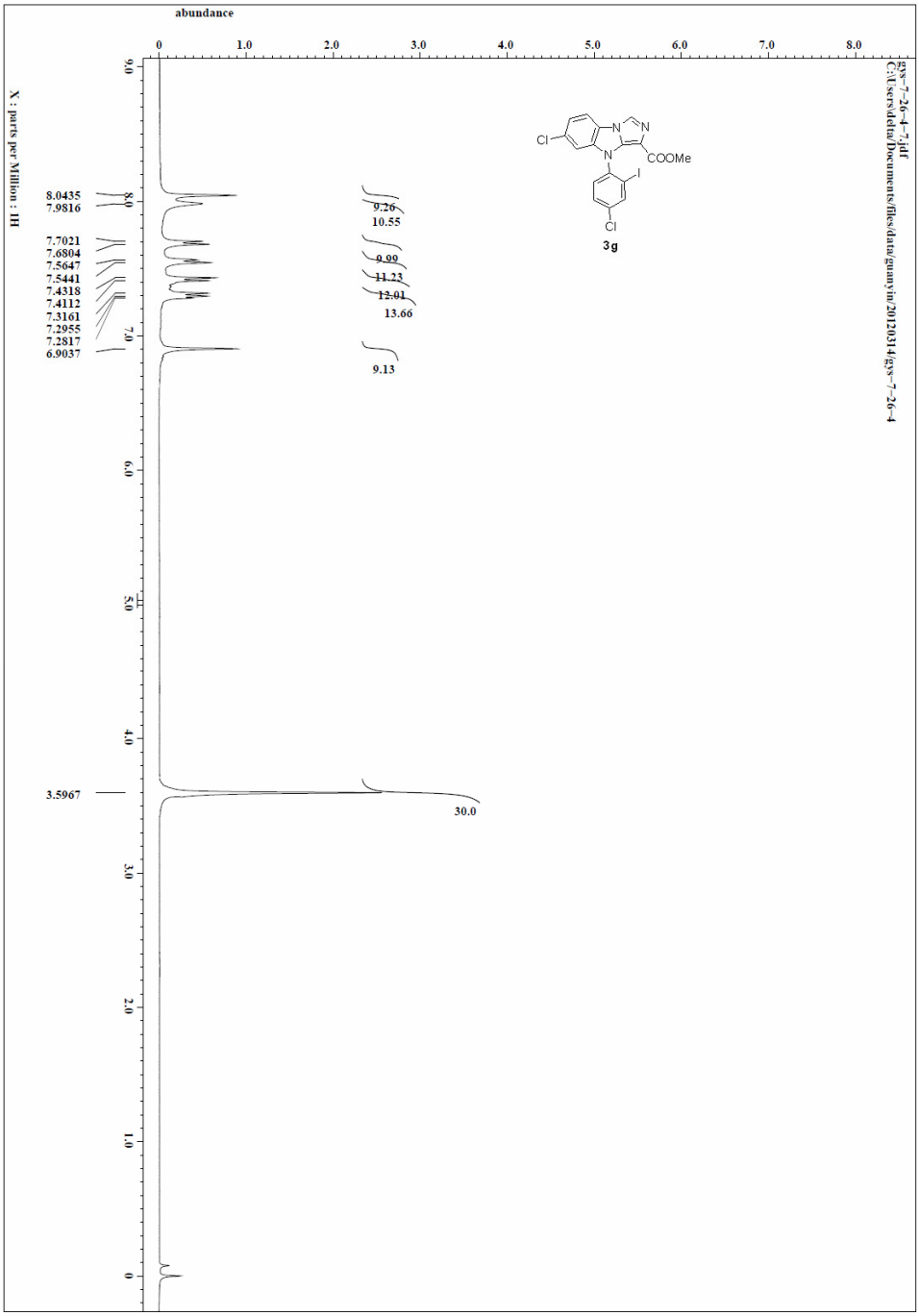

abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
156.2991
141.7970
140.4336
135.7998
130.4413
129.9360
129.8120
122.3465
120.3919
116.7878
109.3699
98.6530
77.3146
77.0000
76.6854
21.0511
Ts
I
3d
gyx-7-23-3c-6.jdf
C:\Users\delta\Documents\flkes\data\guanyin\20120315\gyx-7-23-3c

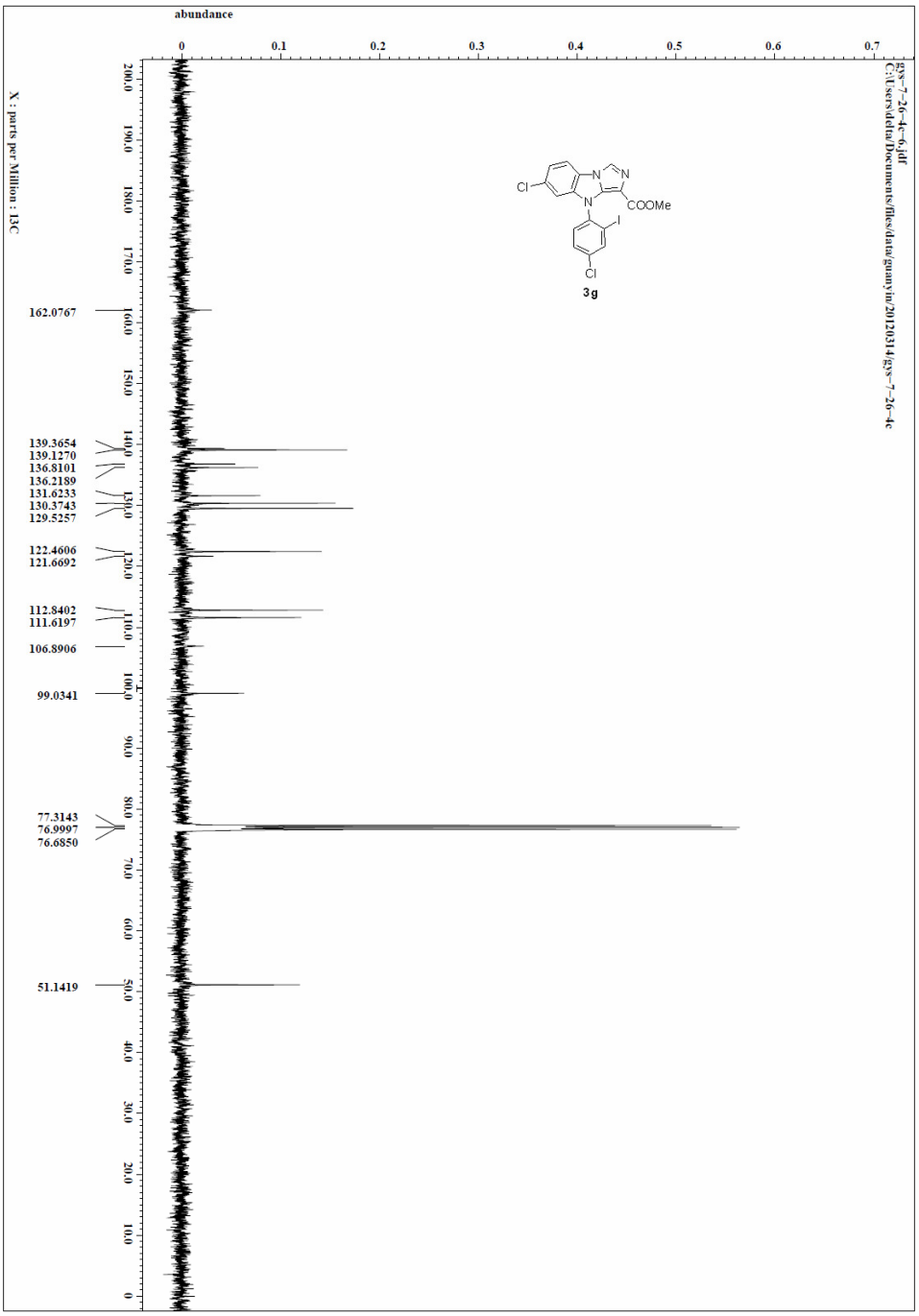

abundance
X : parts per Million : 1H
7.9378
7.8737
7.8313
7.6148
7.5953
7.3513
7.3445
7.3353
7.2735
7.1119
7.0913
6.6984
3.5540
2.4544
2.4177
0.0007
20.24
10.14
20.01
11.99
10
28.71
56.79
COOMe
I
3e
C:\Users\delta\Documents\files\data\guanyin\20120314\gys-7-26-2_9.jdf
gys-7-26-2_9.jdf

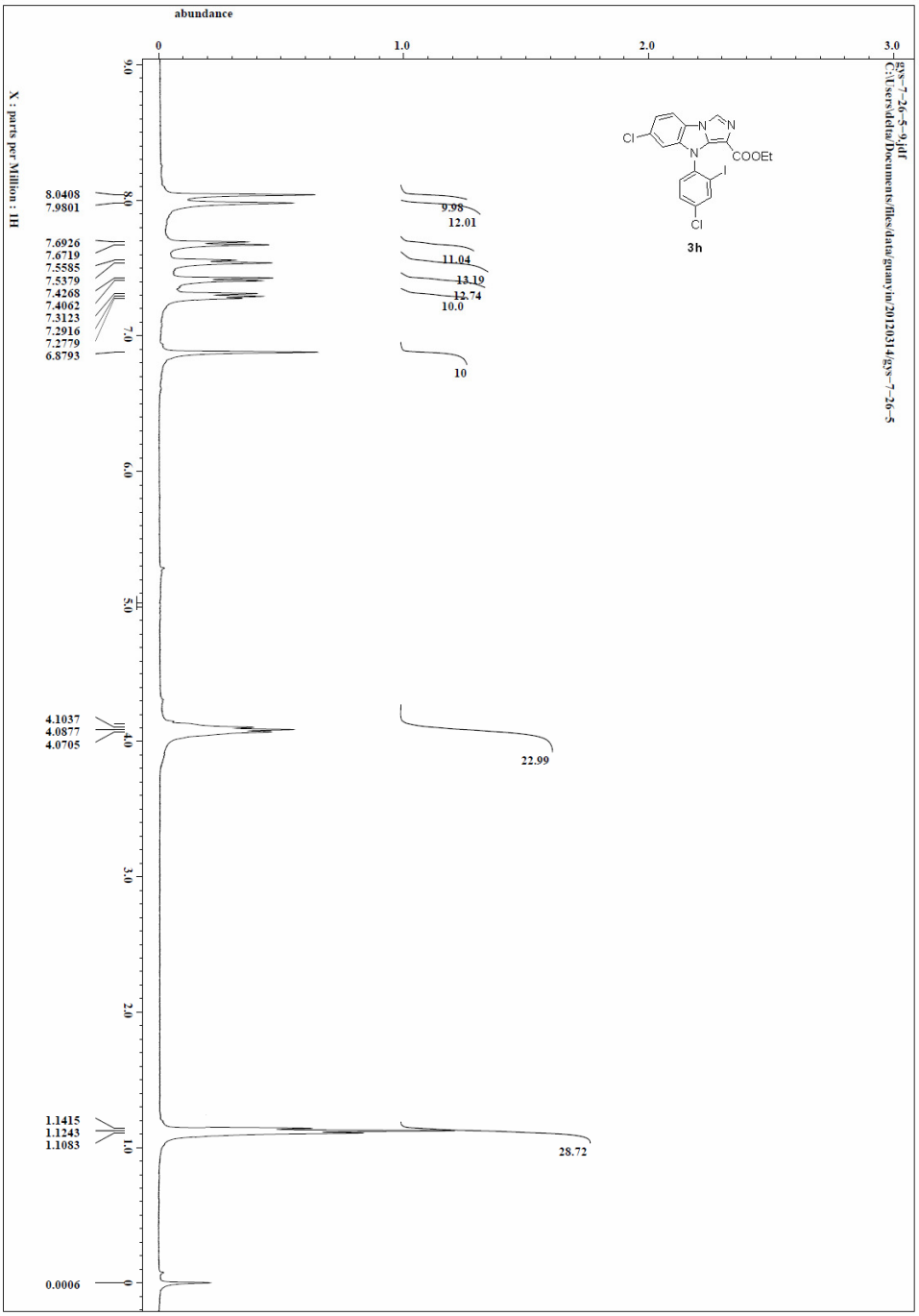

abundance
X : parts per Million : 13C
3e
COOMe
162.3508
141.1649
139.8778
139.1627
136.0067
129.8474
129.3802
122.7727
120.9993
120.6370
111.6268
111.4266
106.2779
98.6884
77.3214
76.9972
76.6826
50.9106
21.6776
20.8195

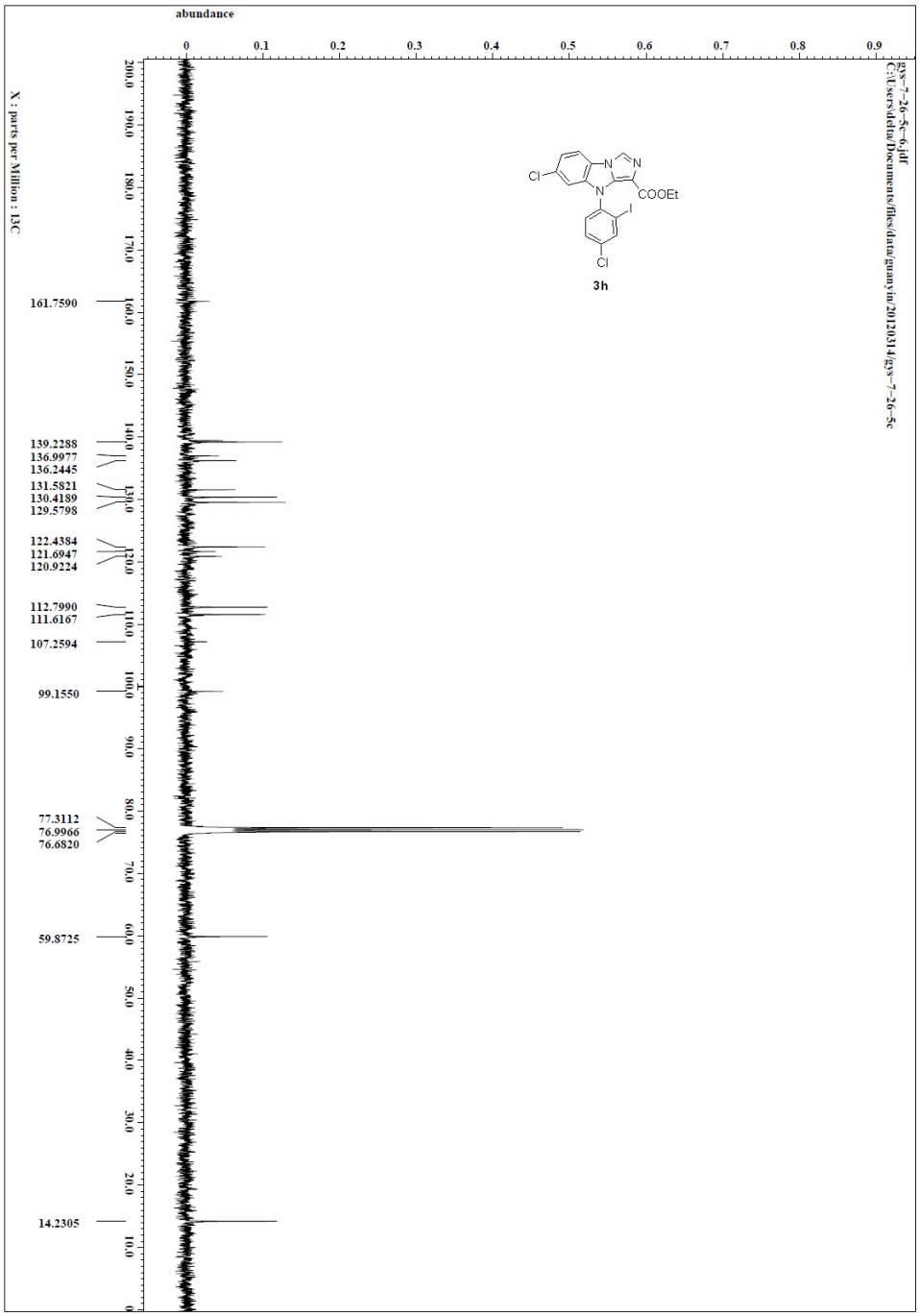

abundance
X : parts per Million : 1H
7.9509
7.8672
7.6141
7.5946
7.3380
7.2773
7.1066
7.0872
6.6702
4.0562
4.0390
4.0207
2.4502
2.4124
1.0687
1.0527
1.0367
9.12
10.38
10.08
19.85
13.05
10.72
20.47
54.96
30
COOEt
I
3f
C:\Users\delta\Documents\files\data\guanyin\20120302\GYS-7-26-1-7.pdf
GYS-7-26-1-7.pdf

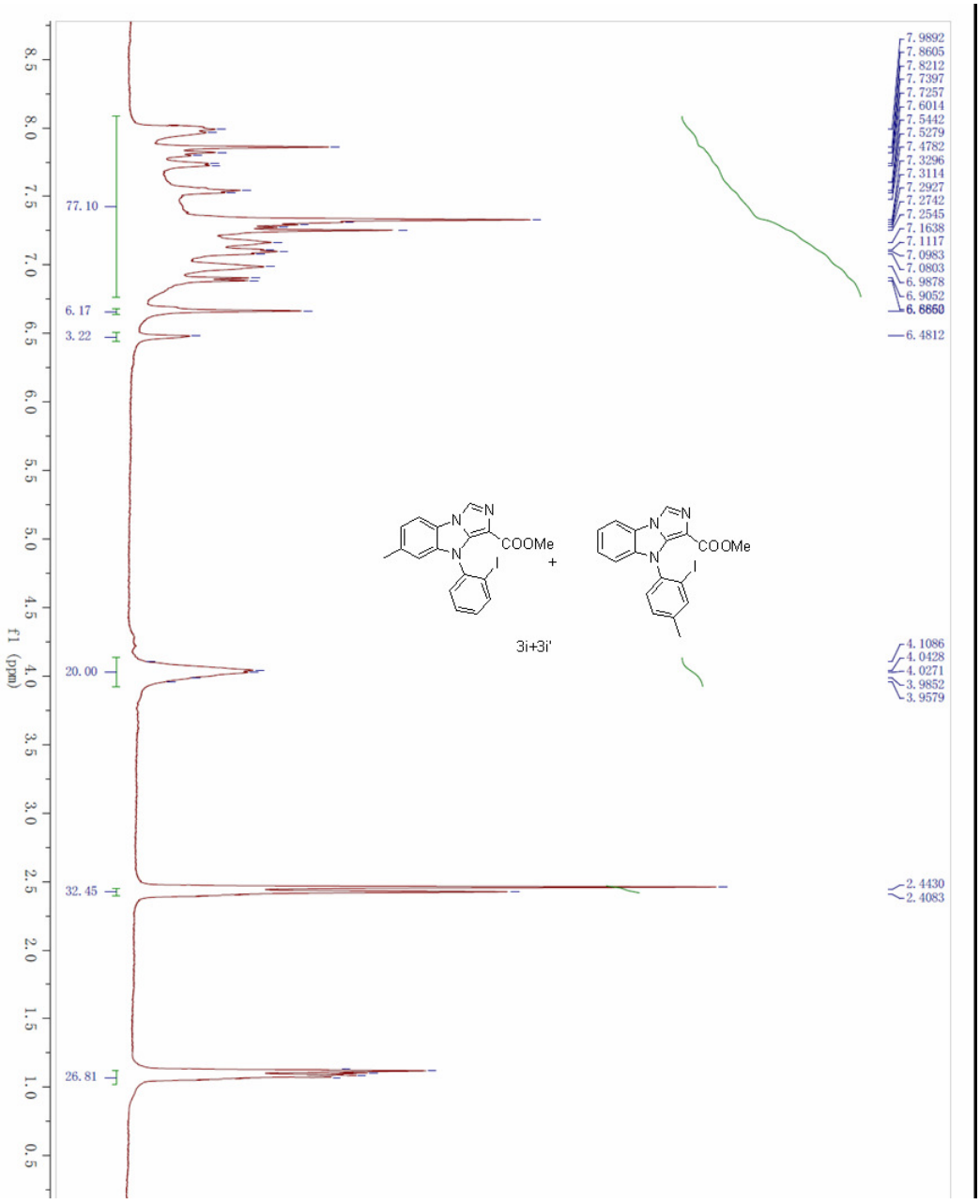

COOEt
I
3f
162.0396
141.1662
140.6040
139.9401
139.2467
136.2228
135.9392
130.5242
129.8825
129.4121
123.2098
122.6634
111.6151
111.3863
98.7883
59.4495
21.6707
20.7716
14.1260
f1 (ppm)

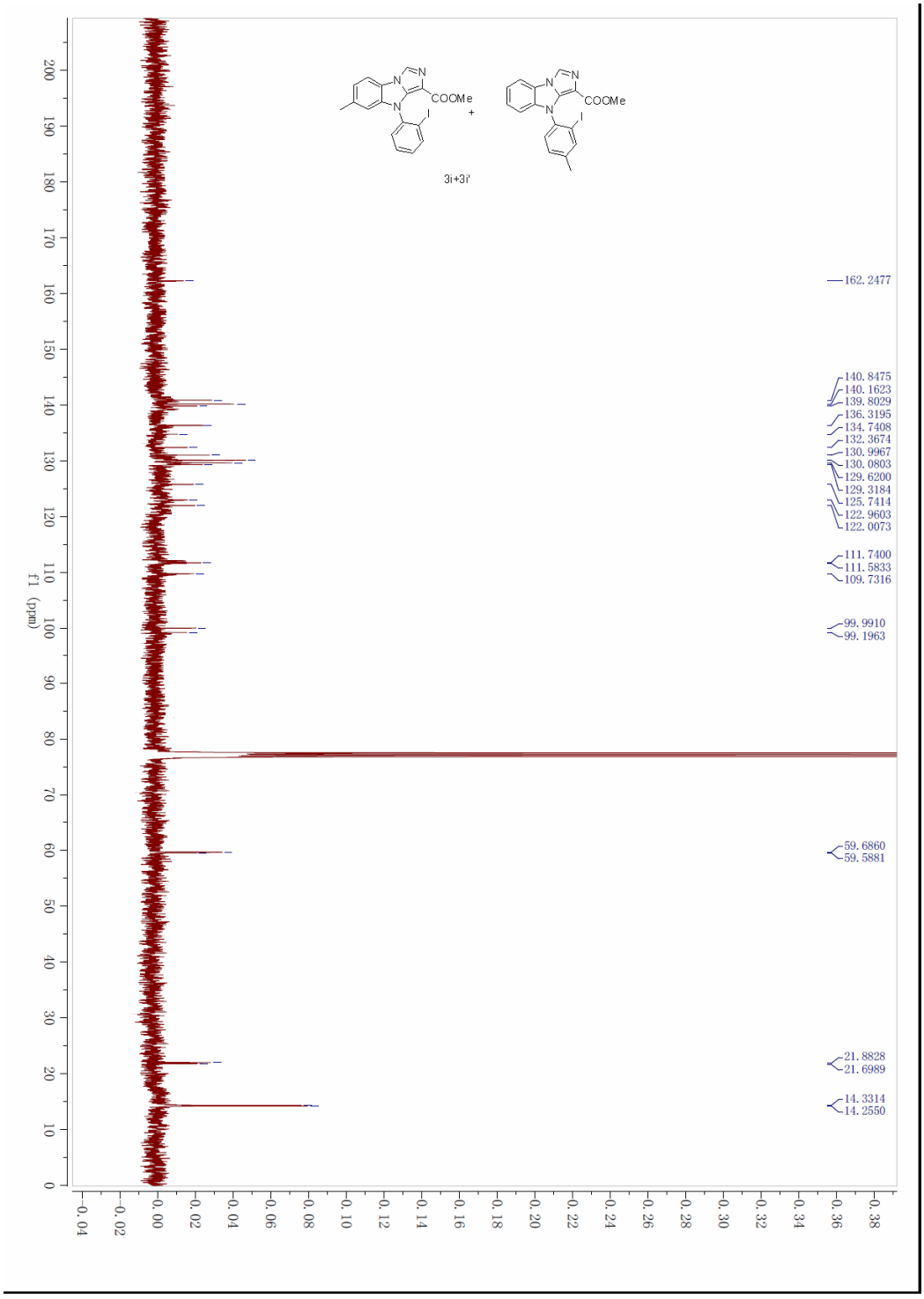

abundance
X : parts per Million : 1H
8.0435
7.9816
7.7021
7.6804
7.5647
7.5441
7.4318
7.4112
7.3161
7.2955
7.2817
6.9037
3.5967
9.26
10.55
9.99
11.23
12.01
13.66
9.13
30.0
3g
gys-7-26-4-7.jdf
C:\Users\delta\Documents\flies\data\guanyin\20120314\gys-7-26-4

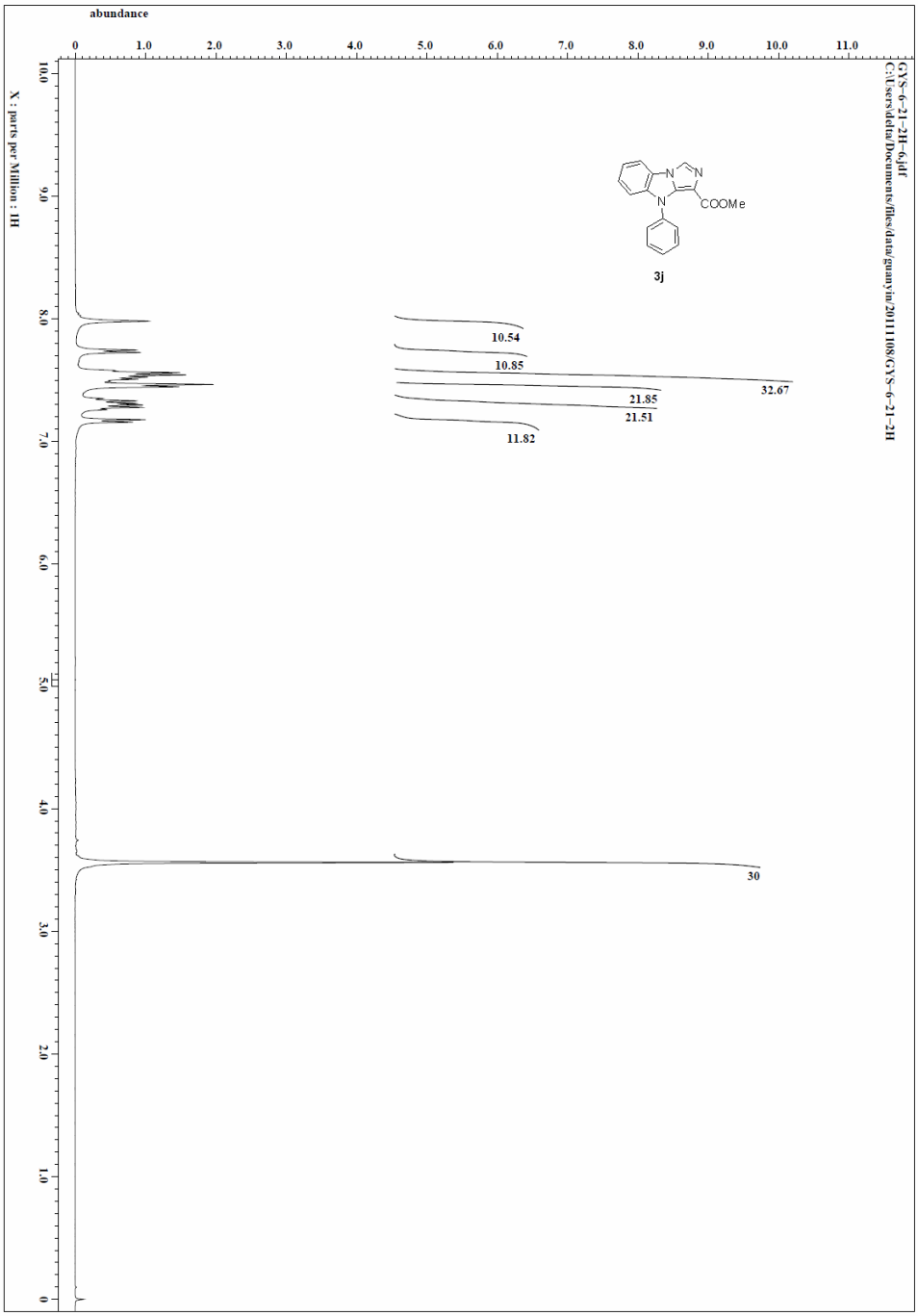

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
X : parts per Million : 13C
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
162.0767
139.3654
139.1270
136.8101
136.2189
131.6233
130.3743
129.5257
122.4606
121.6692
112.8402
111.6197
106.8906
99.0341
77.3143
76.9997
76.6850
51.1419
gsy-7-26-4-c-6.jdf
C:\Users\delta\Documents\files\data\guansiu\20120314\gsy-7-26-4c
Cl
N
N
N
COOMe
I
Cl
3g

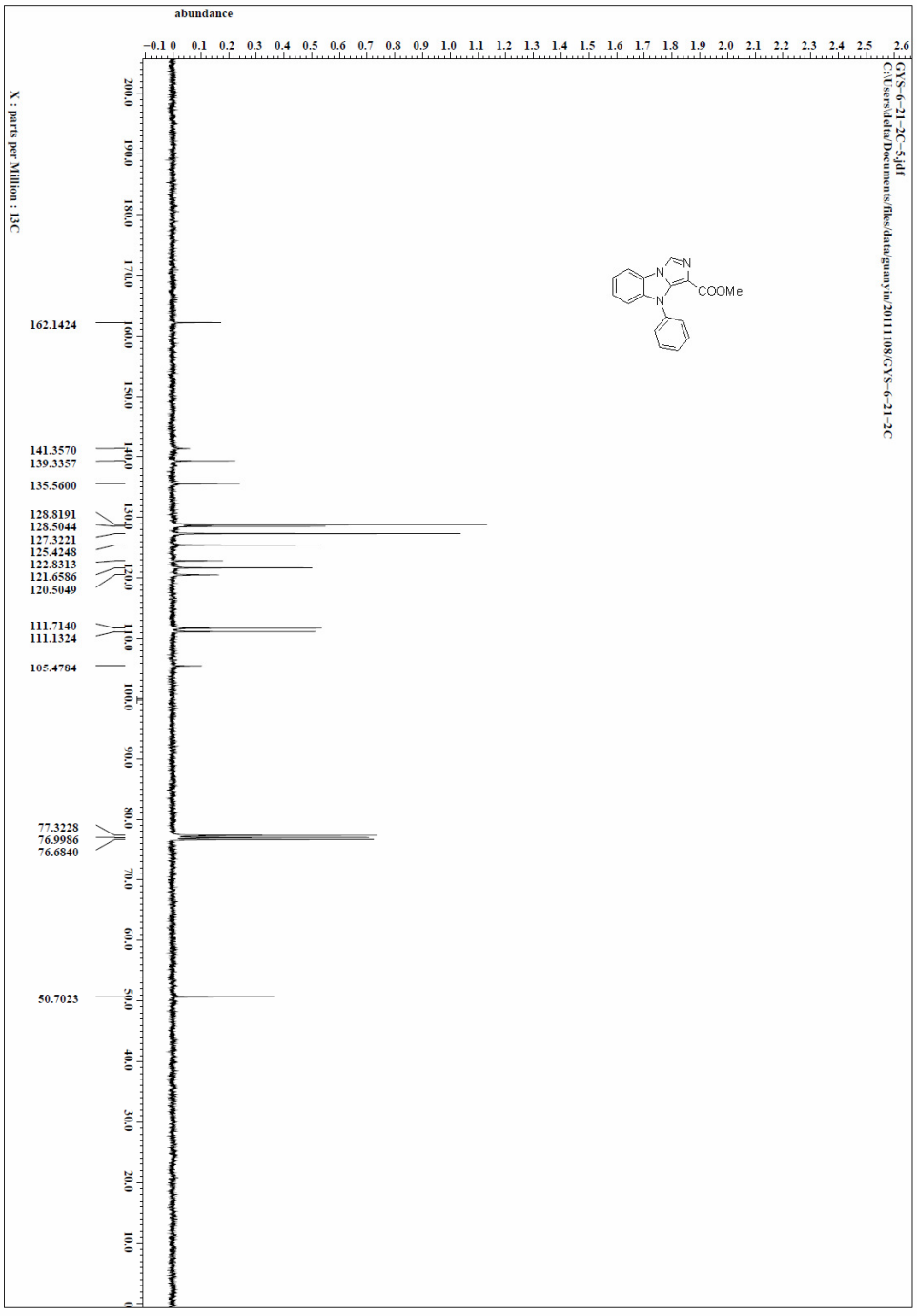

abundance
X : parts per Million : 1H
8.0408
7.9801
7.6926
7.6719
7.5585
7.5379
7.4268
7.4062
7.3123
7.2916
7.2779
6.8793
4.1037
4.0877
4.0705
1.1415
1.1243
1.1083
0.0006
9.98
12.01
11.04
13.19
12.74
10.0
10
22.99
28.72
3h
gsx-7-26-5-9.jdf
C:\Users\delta\Documents\files\data\guanyin\20120314\gsx-7-26-5

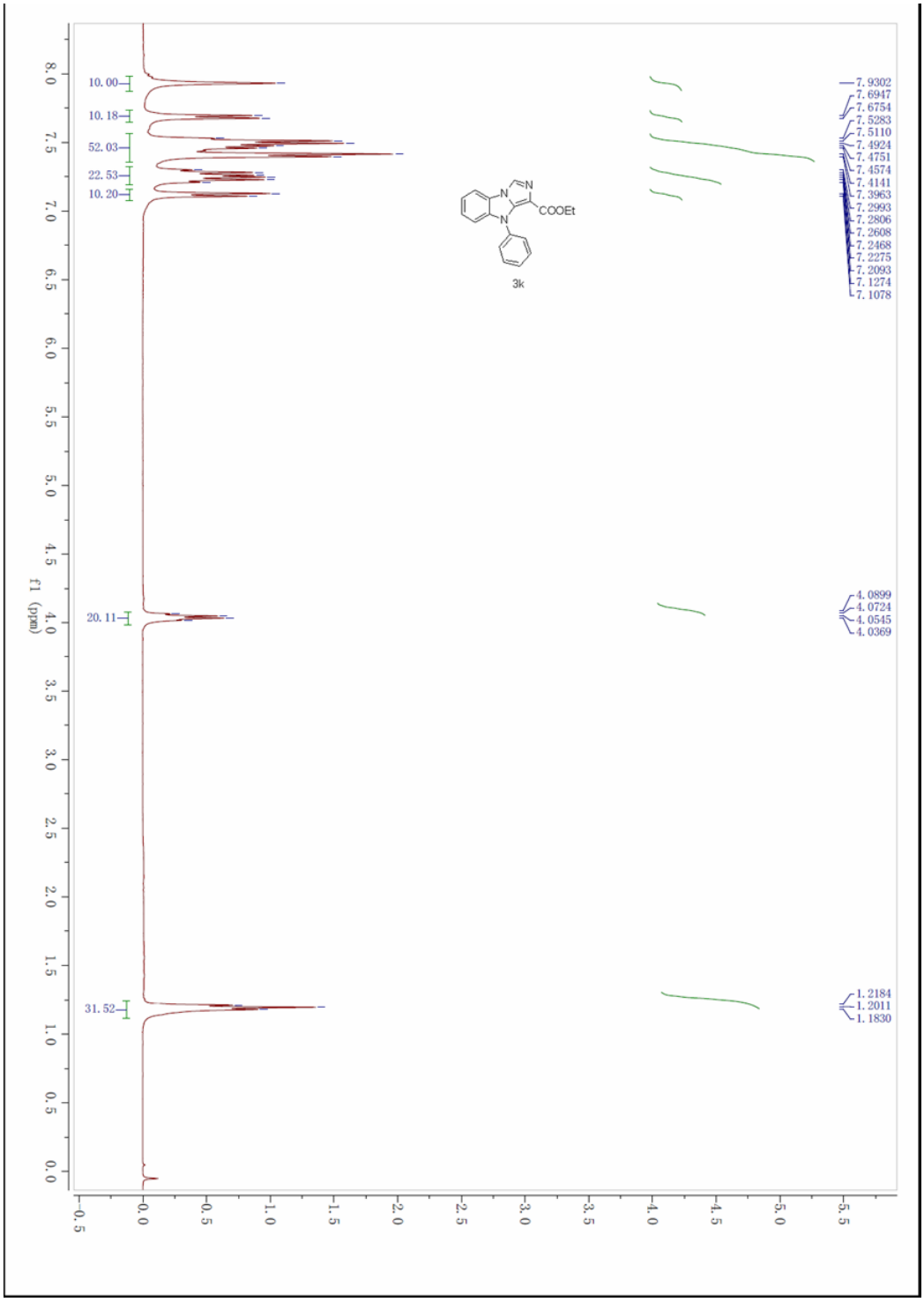

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
X : parts per Million : 13C
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
161.7590
139.2288
136.9977
136.2445
131.5821
130.4189
129.5798
122.4384
121.6947
120.9224
112.7990
111.6167
107.2594
99.1550
77.3112
76.9966
76.6820
59.8725
14.2305
Cl
N
N
N
COOEt
I
Cl
3h
gys-7-26-5c-6.jdf
C:\Users\delta\Documents\files\data\guanyin\20120314\gys-7-26-5c

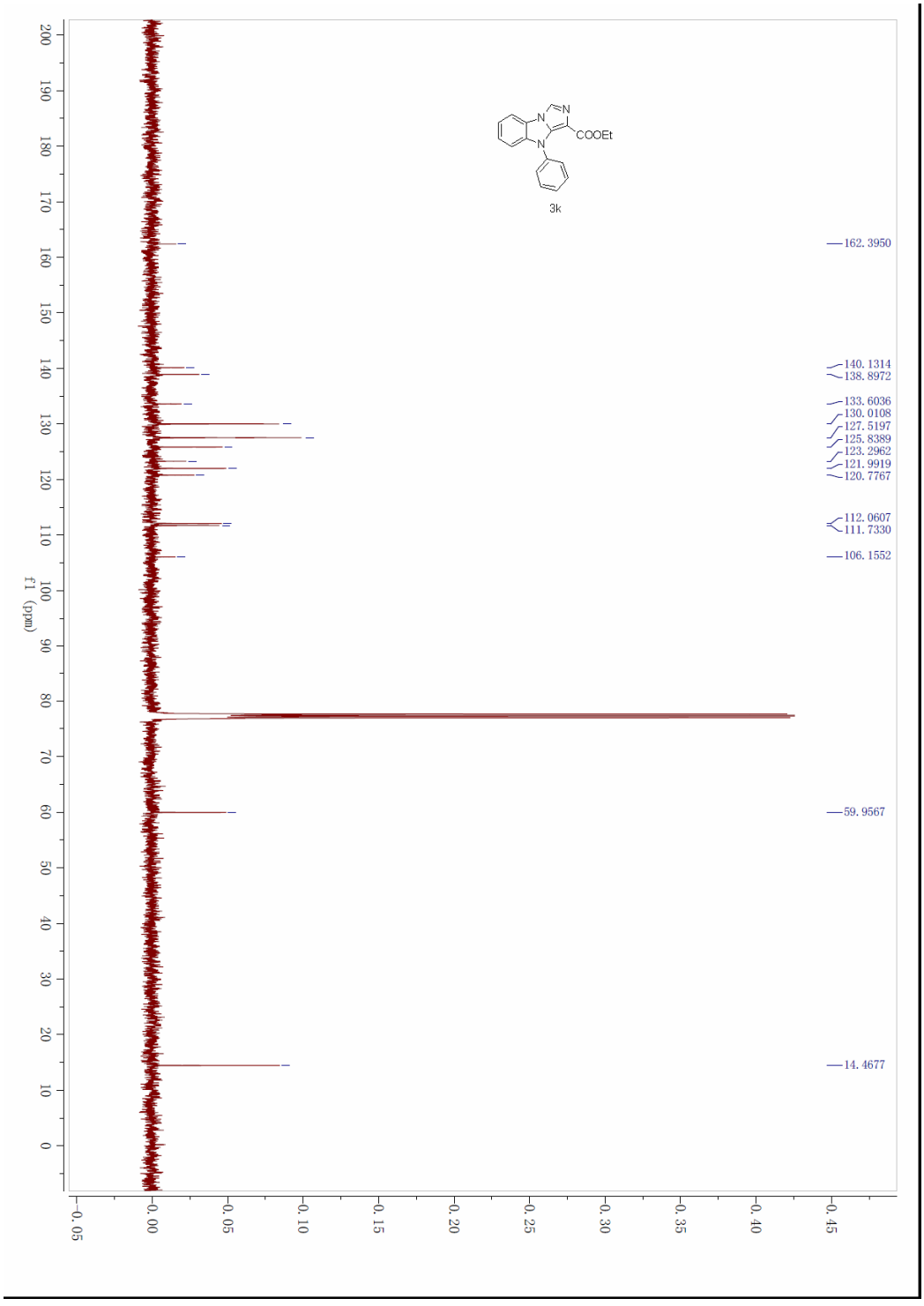

7.9892
7.8605
7.8212
7.7397
7.7257
7.6014
7.5442
7.5279
7.4782
7.3296
7.3114
7.2927
7.2742
7.2545
7.1638
7.1117
7.0983
7.0803
6.9878
6.9052
6.8650
6.4812
4.1086
4.0428
4.0271
3.9852
3.9579
2.4430
2.4083
77.10
6.17
3.22
20.00
32.45
26.81
f1 (ppm)
COOMe
+
COOMe
3i+3i'

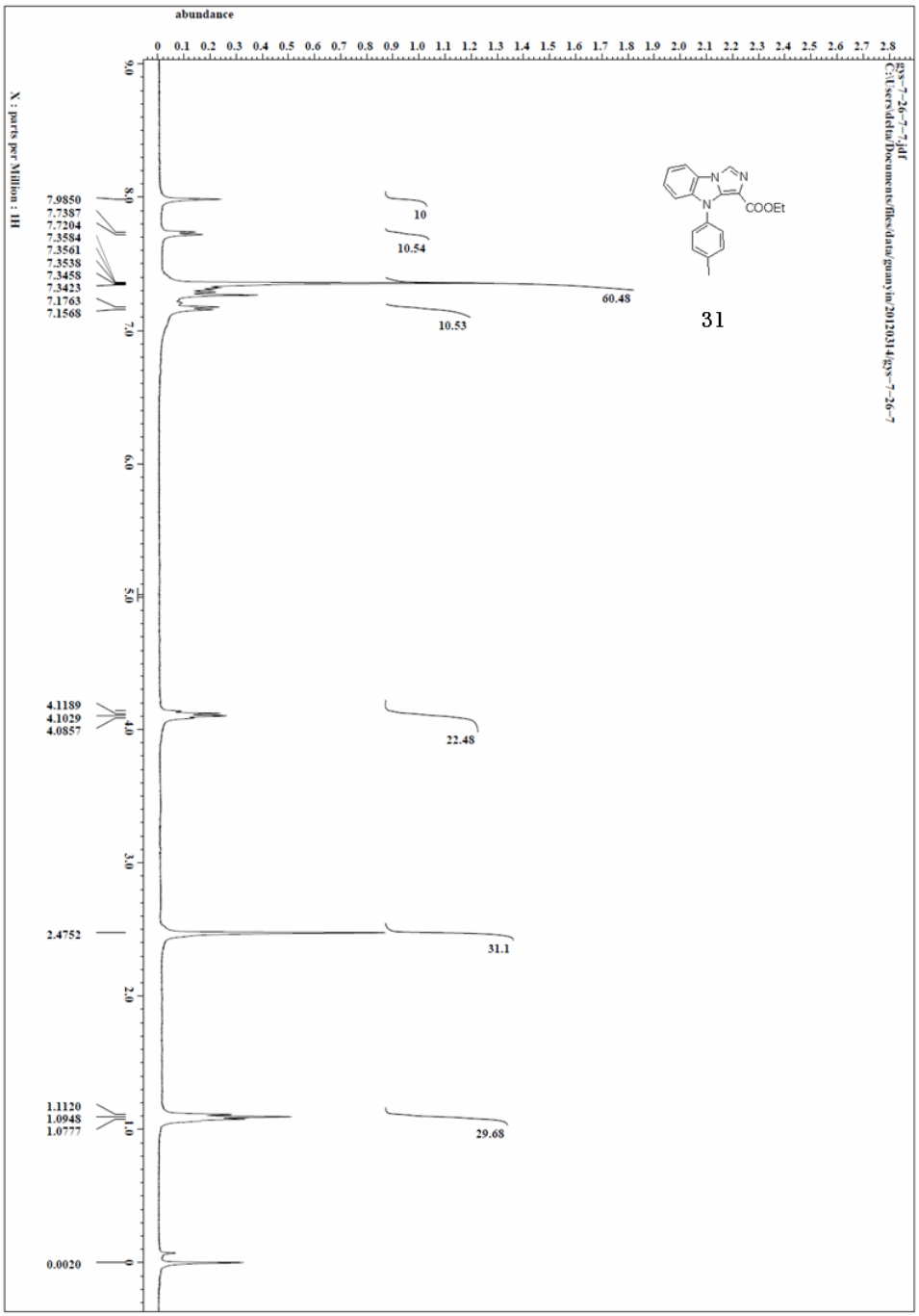

COOMe
+
COOMe
3i+3i'
162.2477
140.8475
140.1623
139.8029
136.3195
134.7408
132.3674
130.9967
130.0803
129.6200
129.3184
125.7414
122.9603
122.0073
111.7400
111.5833
109.7316
99.9910
99.1963
59.6860
59.5881
21.8828
21.6989
14.3314
14.2550
f1 (ppm)

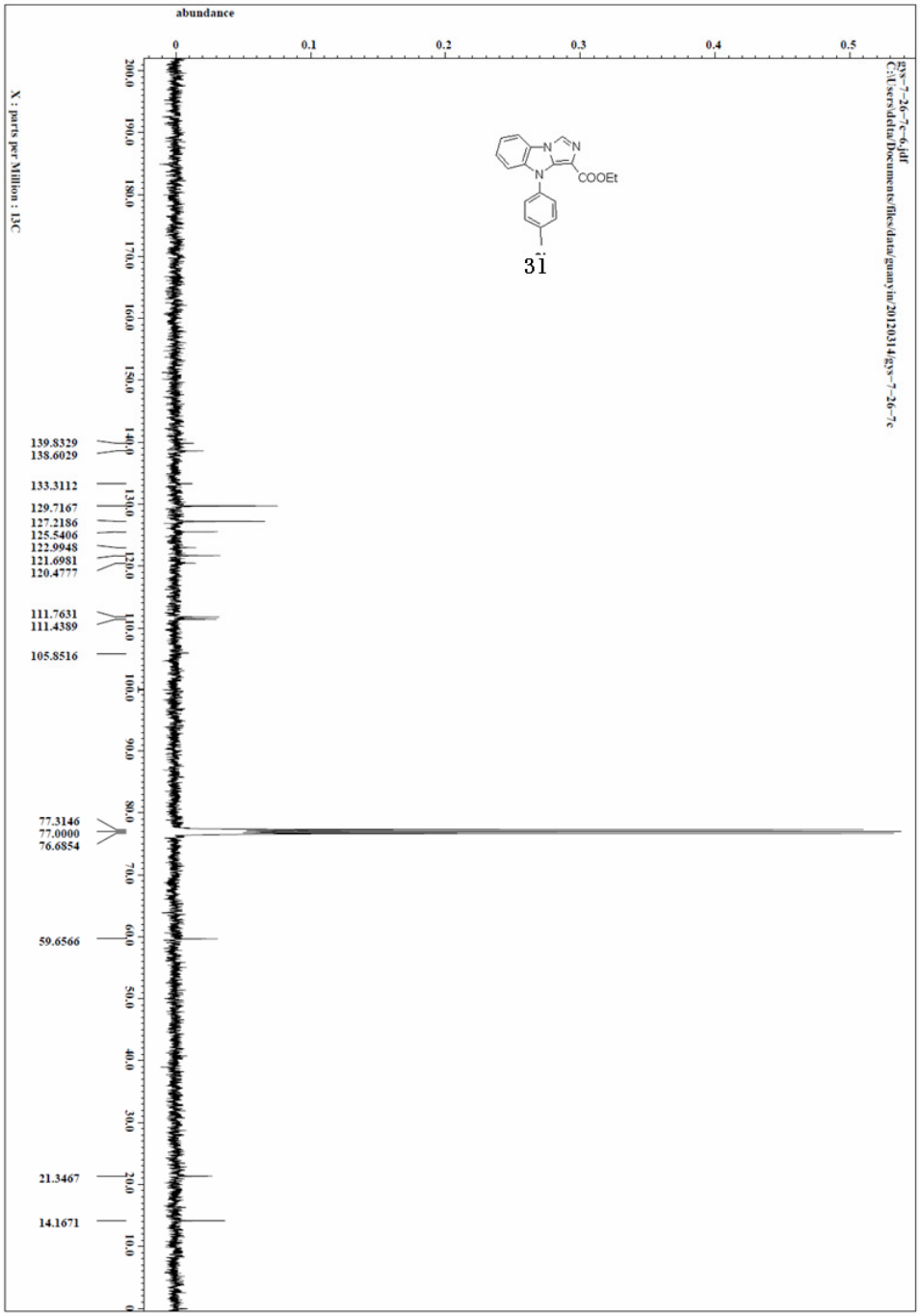

abundance
X : parts per Million : 1H
GYS-6-21-2H-6.jdf
C:\Users\delta\Documents\files\data\guanyin\20111108\GYS-6-21-2H
COOMe
3j
10.54
10.85
32.67
21.85
21.51
11.82
30

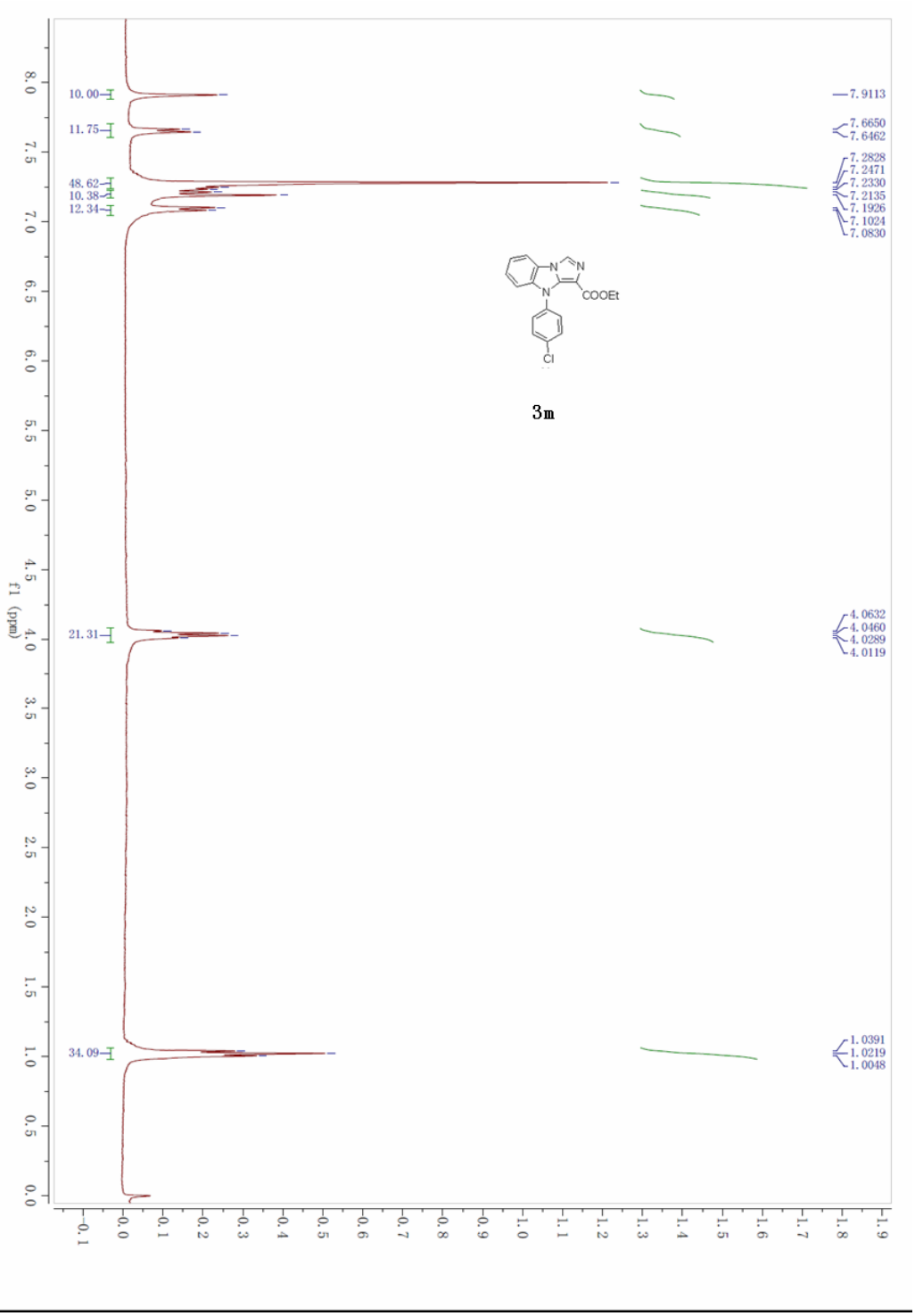

abundance
X : parts per Million : 13C
GYS-6-21-2C-5.jdf
C:\Users\delta\Documents\files\data\guanyin\20111108\GYS-6-21-2C
COOMe
162.1424
141.3570
139.3357
135.5600
128.8191
128.5044
127.3221
125.4248
122.8313
121.6586
120.5049
111.7140
111.1324
105.4784
77.3228
76.9986
76.6840
50.7023


10.00
10.18
52.03
22.53
10.20
20.11
31.52
COOEt
3k
7.9302
7.6947
7.6754
7.5283
7.5110
7.4924
7.4751
7.4574
7.4141
7.3963
7.2993
7.2806
7.2608
7.2468
7.2275
7.2093
7.1274
7.1078
4.0899
4.0724
4.0545
4.0369
1.2184
1.2011
1.1830
f1 (ppm)

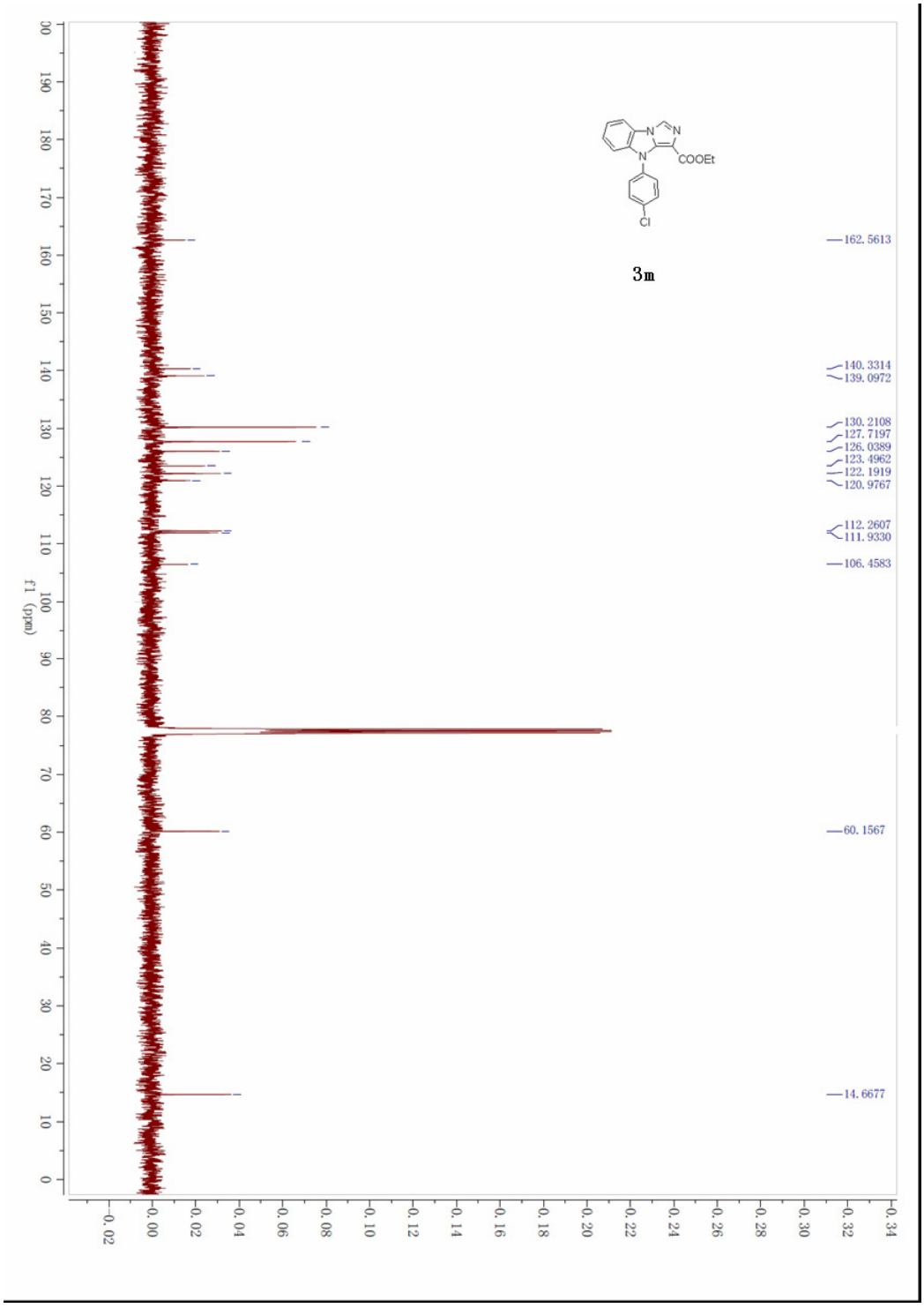

COOEt
3k
162.3950
140.1314
138.8972
133.6036
130.0108
127.5197
125.8389
123.2962
121.9919
120.7767
112.0607
111.7330
106.1552
59.9567
14.4677
f1 (ppm)

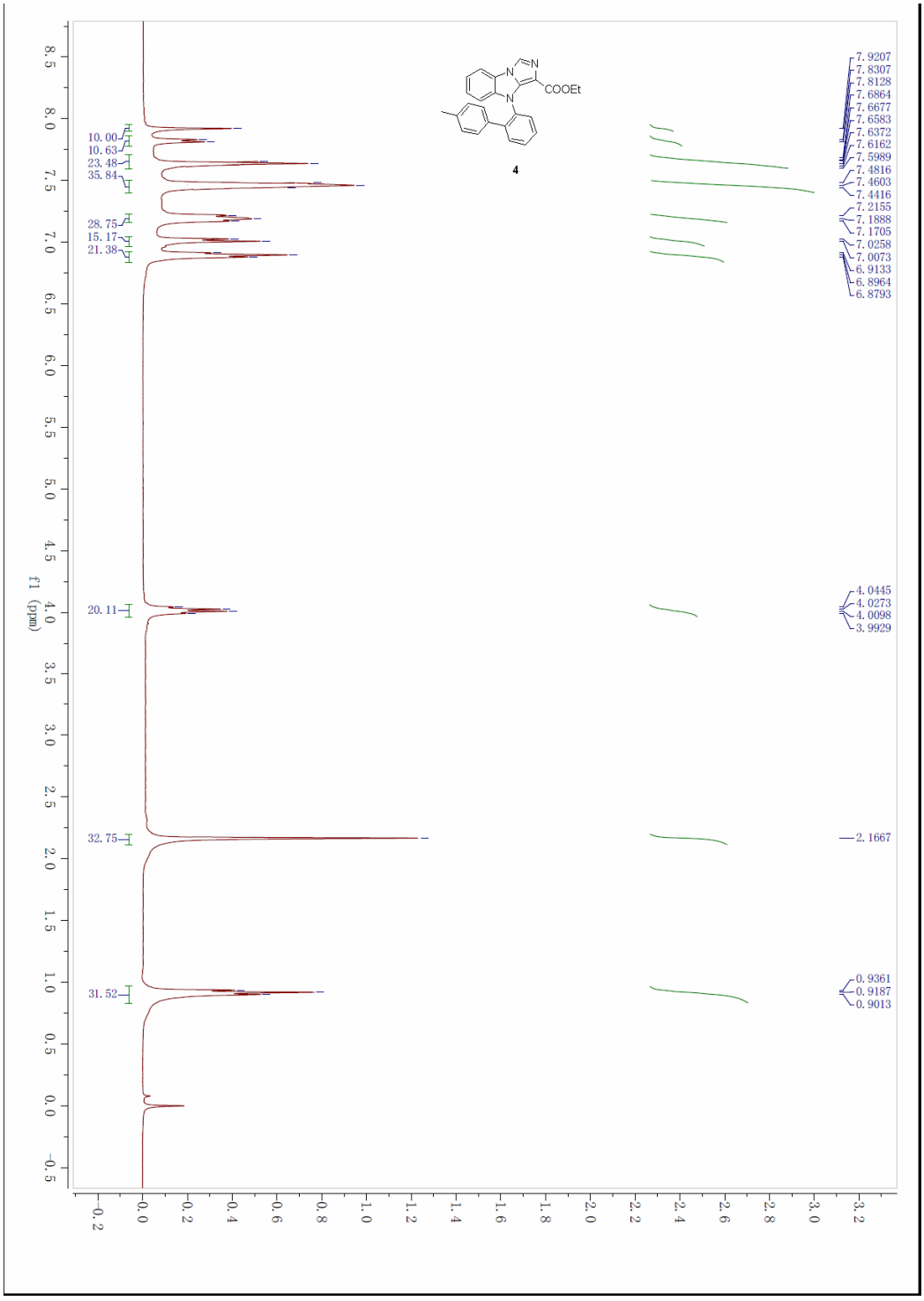

abundance
X : parts per Million : 1H
7.9850
7.7387
7.7204
7.3584
7.3561
7.3538
7.3458
7.3423
7.1763
7.1568
4.1189
4.1029
4.0857
2.4752
1.1120
1.0948
1.0777
0.0020
10
10.54
60.48
10.53
22.48
31.1
29.68
COOEt
31

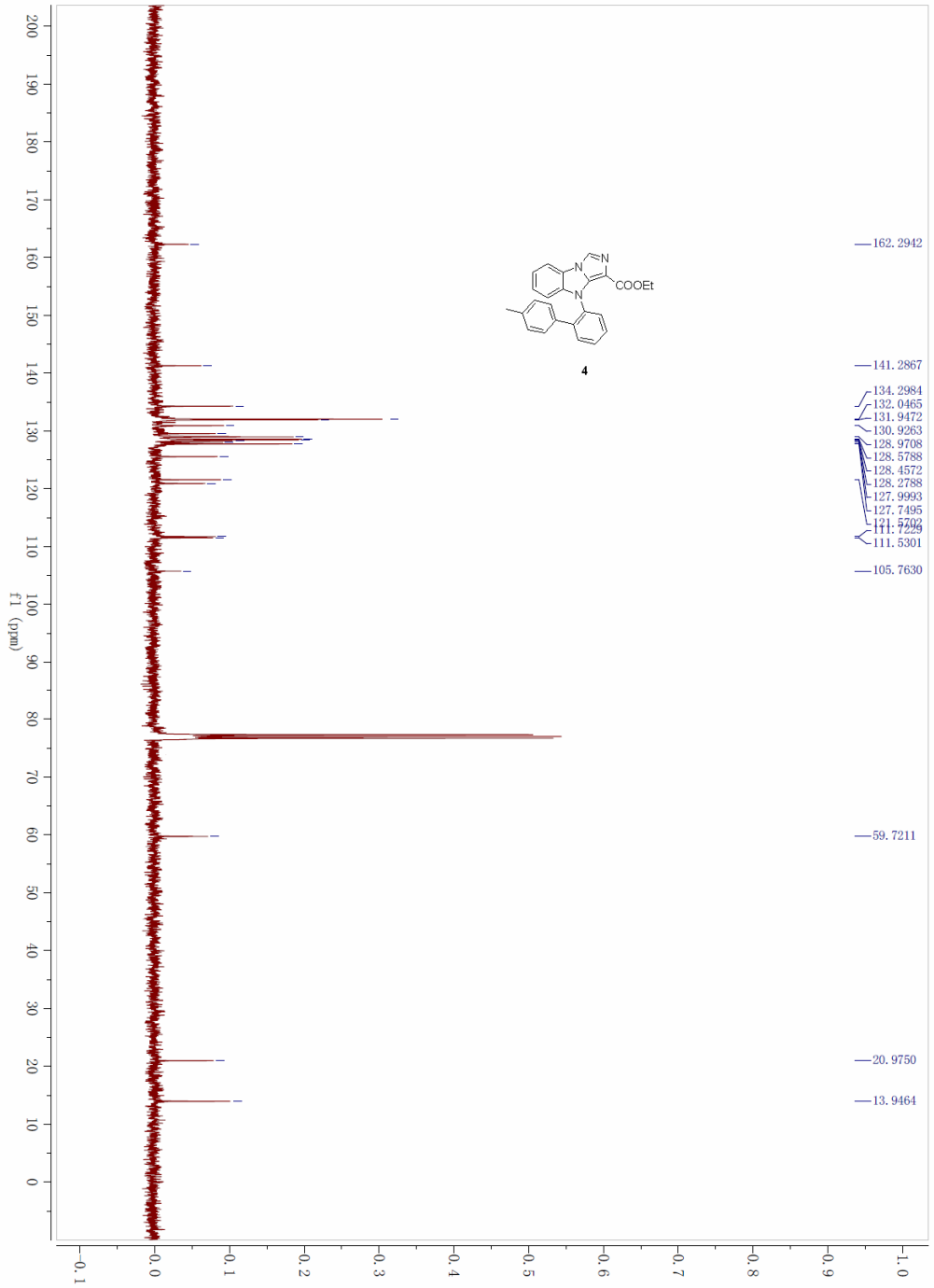

abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
139.8329
138.6029
133.3112
129.7167
127.2186
125.5406
122.9948
121.6981
120.4777
111.7631
111.4389
105.8516
77.3146
77.0000
76.6854
59.6566
21.3467
14.1671
COOEt
31

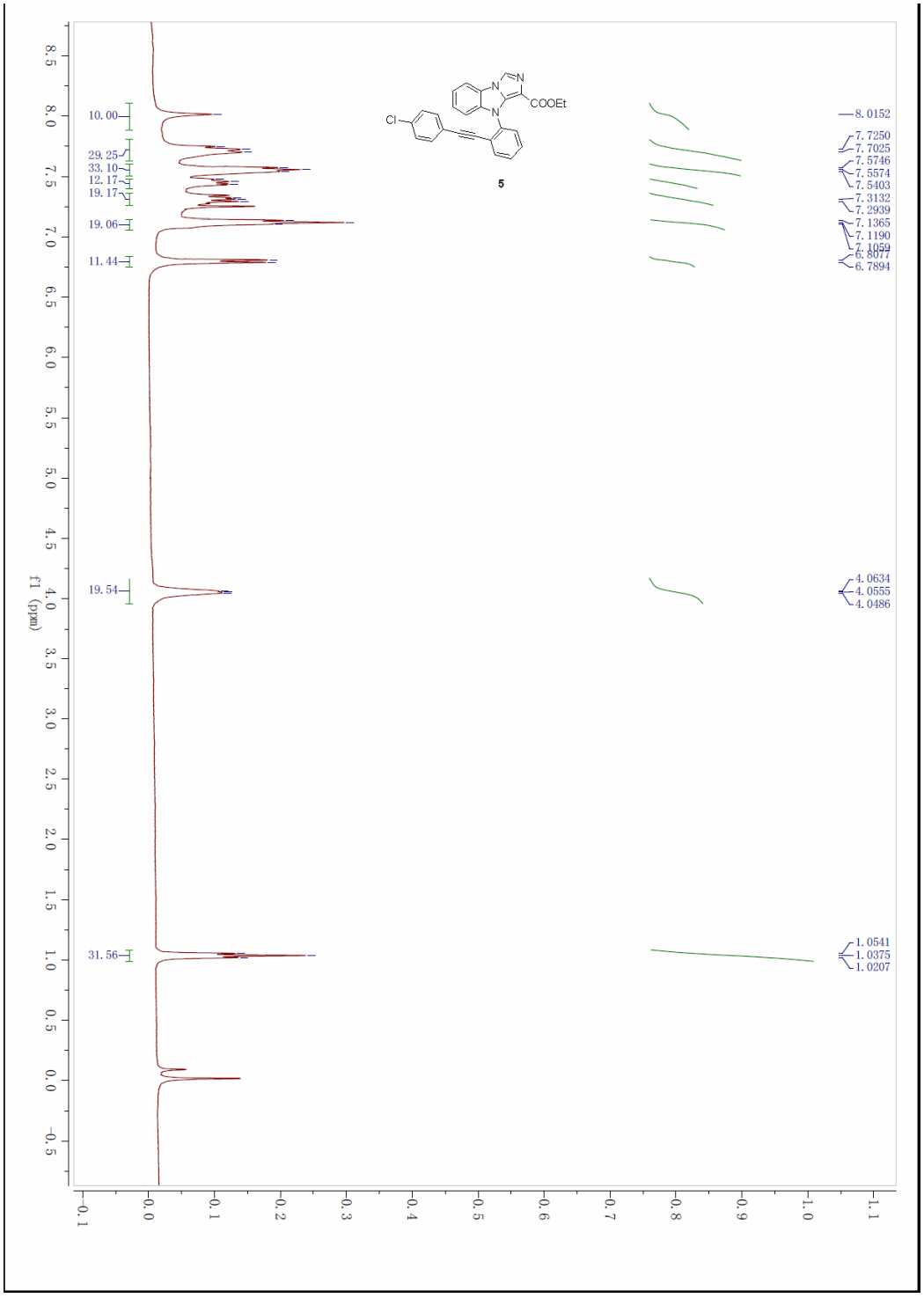

10.00
11.75
48.62
10.38
12.34
21.31
34.09
7.9113
7.6650
7.6462
7.2828
7.2471
7.2330
7.2135
7.1926
7.1024
7.0830
4.0632
4.0460
4.0289
4.0119
1.0391
1.0219
1.0048
COOEt
Cl
f1 (ppm)

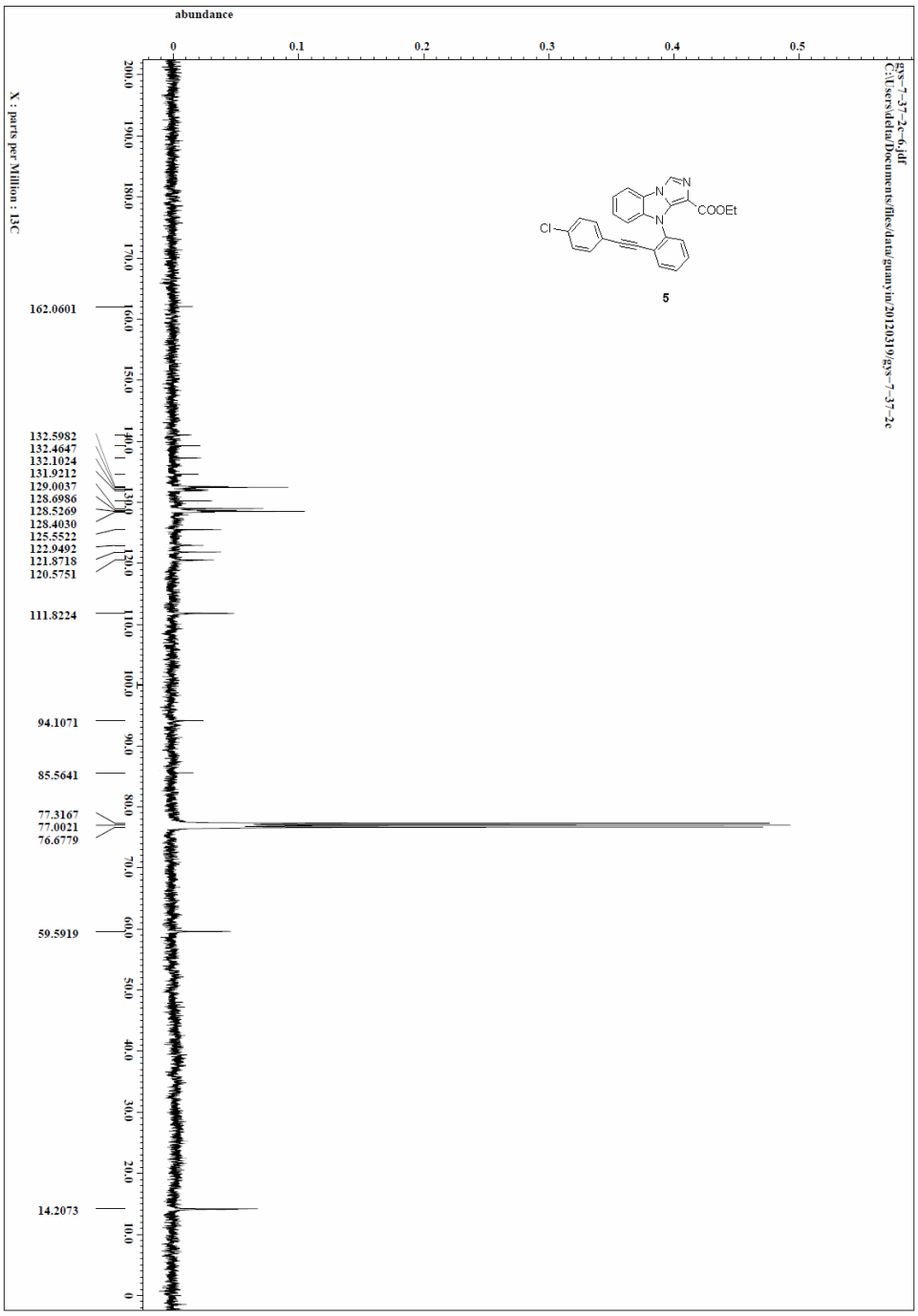
3m


COOEt
Cl
3m
162.5613
140.3314
139.0972
130.2108
127.7197
126.0389
123.4962
122.1919
120.9767
112.2607
111.9330
106.4583
60.1567
14.6677
f1 (ppm)


COOEt
4
10.00
10.63
23.48
35.84
28.75
15.17
21.38
20.11
32.75
31.52
7.9207
7.8307
7.8128
7.6864
7.6677
7.6583
7.6372
7.6162
7.5989
7.4816
7.4603
7.4416
7.2155
7.1888
7.1705
7.0258
7.0073
6.9133
6.8964
6.8793
4.0445
4.0273
4.0098
3.9929
2.1667
0.9361
0.9187
0.9013
f1 (ppm)


162.2942
141.2867
134.2984
132.0465
131.9472
130.9263
128.9708
128.5788
128.4572
128.2788
127.9993
127.7495
111.5301
105.7630
59.7211
20.9750
13.9464
COOEt
4
f1 (ppm)


10.00
29.25
33.10
12.17
19.17
19.06
11.44
19.54
31.56
COOEt
Cl
5
8.0152
7.7250
7.7025
7.5746
7.5574
7.5403
7.3132
7.2939
7.1365
7.1190
7.1059
6.8077
6.7894
4.0634
4.0555
4.0486
1.0541
1.0375
1.0207
f1 (ppm)


abundance
0
0.1
0.2
0.3
0.4
0.5
X : parts per Million : 13C
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
162.0601
132.5982
132.4647
132.1024
131.9212
129.0037
128.6986
128.5269
128.4030
125.5522
122.9492
121.8718
120.5751
111.8224
94.1071
85.5641
77.3167
77.0021
76.6779
59.5919
14.2073
N
N
COOEt
Cl
5
gss-7-37-2c-6.jdf
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