Silver triflate-copper(II) acetate cooperative catalysis in cascade reaction for concise synthesis of 2-carbonyl *H*-pyrazolo[5,1-*a*]isoquinolines

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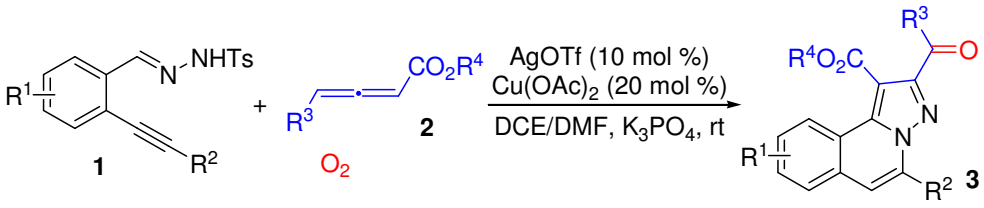
Supporting Information

1. General experimental methods (S2).
2. General experimental procedure and characterization data (S2-S9).
3. ^{1}H and ^{13}C NMR spectra of compound **3** (S10-S41).

General experimental methods:

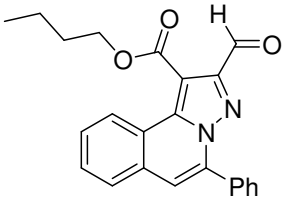
Unless otherwise stated, all commercial reagents were used as received. All solvents were dried and distilled according to standard procedures. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63μm, standard grade). Analytical thin–layer chromatography was performed using 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 experimental procedure for the reactions of N'-(2-alkynylbenzylidene)hydrazide ***1*** *with allenoate* ***2*** *in the presence of oxygen co-catalyzed by silver triflate and copper(II) acetate.*

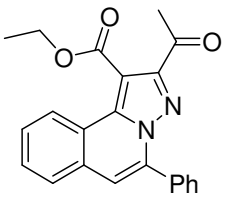


Silver triflate (10 mol %) was added to a solution of *N*`-(2-alkynylbenzylidene) hydrazide **1** (0.2 mmol) in DCE (1.0 mL). The solution was stirred at 50 °C in air for one hour. Then copper(II) acetate (20 mol %), K_3PO_4 (10 mol %), allenoate **2** (2.0 equiv), and DMF (1.5 mL) were added. The mixture was stirred at room temperature (25 °C) under O_2 atomsphere overnight. After completion of reaction as indicated by

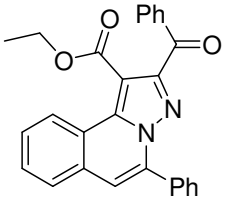
TLC, the reaction was quenched by saturated aqueous NH_4Cl (5.0 mL). The mixture was extracted with EtOAc (4.0 mL×3). The combined organic layer was dried over Na_2SO_4, and concentrated in vacuo. The residue was purified by column chromatography on silica gel to provide the desired product **3**.



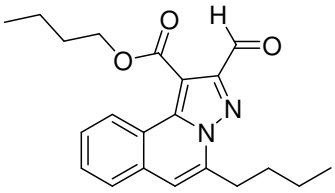
Butyl 2-formyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3a**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.00 (t, *J* = 6.9 Hz, 3H), 1.47-1.51 (m, 2H), 1.81-1.85 (m, 2H), 4.50-4.52 (m, 2H), 7.30 (s, 1H), 7.53 (d, *J* = 8.0 Hz, 3H), 7.65-7.66 (m, 2H), 7.83-7.85 (m, 3H), 8.97 (d, *J* = 8.0 Hz, 1H), 10.36 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.5, 35.4, 44.5, 72.5, 106.9, 113.8, 119.0, 120.7, 122.2, 122.7, 122.9, 126.3, 130.5, 131.6, 139.3, 151.7, 168.4, 169.7; HRMS (ESI) calcd for $C_{23}H_{20}N_2O_3$: 373.1552 ($M + H^+$), found: 373.1565.



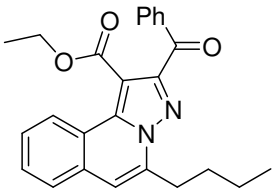
Ethyl 2-acetyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3b**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.45 (t, *J* = 8.0 Hz, 3H), 2.64 (s, 3H), 4.52-4.57 (m, 2H), 7.24 (s, 1H), 7.52-7.62 (m, 4H), 7.78 (d, *J* = 4.0 Hz, 1H), 7.86-7.87 (m, 2H), 8.48 (d, *J* = 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 27.6, 62.0, 108.0, 115.9, 123.5, 124.5, 127.6, 128.2, 128.3, 129.3, 129.6, 129.9, 132.8, 138.0, 149.2, 152.4, 165.7, 194.4; HRMS (ESI) calcd for $C_{24}H_{22}N_2O_3$: 381.1215 ($M + Na^+$), found: 381.1216.



Ethyl 2-benzoyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3c**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.00 (t, *J* = 8.0 Hz, 3H), 4.14-4.17 (m, 2H), 7.32 (s, 1H), 7.44-7.48 (m, 5H), 7.55-7.58 (m, 1H), 7.68-7.69 (m, 2H), 7.82-7.88 (m, 3H), 7.98-8.00 (m, 2H), 9.51 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.8, 69.0, 106.1, 113.0, 118.9, 121.9, 122.7, 123.7, 123.9, 129.6, 130.7, 131.5, 142.1, 151.1, 172.5; HRMS (ESI) calcd for $C_{27}H_{20}N_2O_3$: 443.1372 ($M + Na^+$), found: 443.1371.

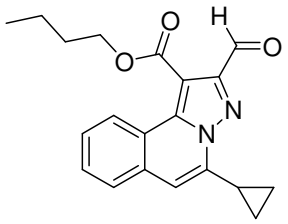


Butyl 5-butyl-2-formyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3d**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.00-1.49 (m, 10H), 1.79-1.85 (m, 4H), 3.19-3.22 (m, 2H), 4.46-4.49 (m, 2H), 7.07 (s, 1H), 7.58-7.64 (m, 2H), 7.71 (d, *J* = 8.0 Hz, 1H), 9.07 (d, *J* = 8.0 Hz, 1H), 10.48 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 14.1, 19.4, 22.5, 28.9, 30.5, 30.7, 65.8, 108.8, 114.2, 123.3, 126.1, 127.0, 127.5, 129.7, 130.5, 139.2, 139.5, 149.1, 164.4, 187.1; HRMS (ESI) calcd for $C_{21}H_{24}N_2O_3$: 375.1685 ($M + Na^+$), found: 375.1696.

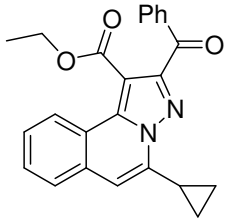


Ethyl 2-benzoyl-5-butyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3e**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90-1.01 (m, 6H), 1.45-1.51 (m, 2H), 1.83-1.88 (m, 2H), 3.19-3.22 (m, 2H), 4.12-4.17 (m, 2H), 7.10 (s, 1H), 7.46-7.49 (m, 2H), 7.58-7.68 (m, 3H), 7.76 (d, *J* = 4.0 Hz, 1H), 8.02 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$)

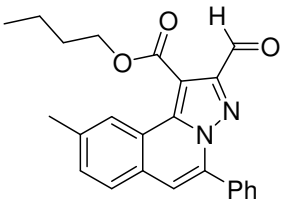
δ 30.8, 31.2, 38.0, 43.2, 44.6, 61.8, 68.9, 77.8, 95.5, 106.0, 118.6, 121.4, 121.8, 123.7, 129.7, 131.1, 131.6, 142.1, 151.1, 159.6, 169.0, 172.9; HRMS (ESI) calcd for $C_{27}H_{20}N_2O_3$: 423.1685 (M + Na^+), found: 423.1661.



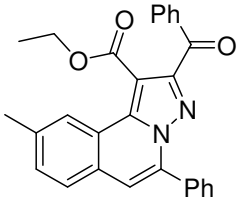
Butyl 5-cyclopropyl-2-formyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3f**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.90-0.91 (m, 2H), 0.98 (t, *J* = 8.0 Hz, 3H), 1.22-1.24 (m, 2H), 1.45-1.51 (m, 2H), 1.79-1.83 (m, 2H), 2.78 (s, 1H), 4.46-4.49 (m, 2H), 6.88 (s, 1H), 7.58-7.67 (m, 3H), 9.09 (d, *J* = 8.0 Hz, 1H), 10.51 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.8, 11.4, 13.9, 19.4, 30.6, 30.7, 65.8, 109.0, 111.3, 123.1, 126.2, 127.0, 127.5, 129.7, 130.6, 139.3, 141.1, 149.2, 164.3, 187.1; HRMS (ESI) calcd for $C_{20}H_{20}N_20_3$: 359.1372 (M + Na^+), found: 359.1389.



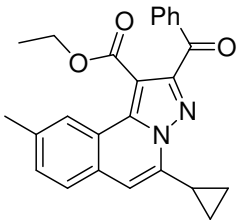
Ethyl 2-benzoyl-5-cyclopropyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3g**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94-1.00 (m, 5H), 1.19-1.21 (m, 2H), 2.74-2.79 (m, 1H), 4.11-4.16 (m, 2H), 6.89 (s, 1H), 7.47-7.50 (m, 2H), 7.59-7.69 (m, 3H), 7.72 (d, *J* = 8.0 Hz, 1H), 8.03 (d, *J* = 8.0 Hz, 1H), 9.51 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.3, 29.2, 30.1, 30.8, 68.9, 103.2, 106.0, 108.1, 110.2, 118.4, 121.4, 121.8, 126.8, 129.7, 132.9, 135.2, 142.3, 151.1, 162.6, 166.0, 173.0; HRMS (ESI) calcd for $C_{24}H_{20}N_2O_3$: 407.1372 (M + Na^+), found: 407.1364.



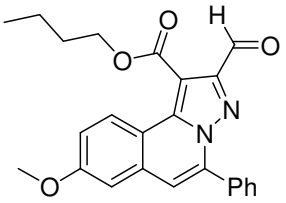
Butyl 2-formyl-9-methyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3h**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 8.0 Hz, 3H),1.46-1.54 (m, 2H), 1.61-1.83 (m, 2H), 2.56 (s, 3H), 4.49-4.52 (m, 2H), 7.25 (s, 1H), 7.46-7.50 (m, 5H), 7.51-7.81 (m, 2H), 8.69 (s, 1H), 10.33 (s, 1H);^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 19.3, 22.1, 30.6, 65.9, 108.3, 116.9, 120.1, 123.7, 125.3, 127.5, 128.1, 128.5, 129.6, 131.4, 137.4, 138.5, 139.0, 149.0, 164.6, 186.6; HRMS (ESI) calcd for $C_{24}H_{22}N_2O_3$: 409.1528 (M + Na^+), found: 409.1547.



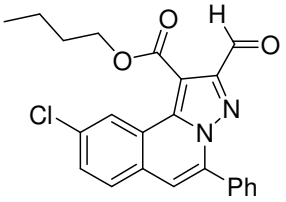
Ethyl 2-benzoyl-9-methyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3i**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.00 (t, *J* = 4.0 Hz, 3H), 2.61 (s, 3H), 4.15-4.19 (m, 2H), 7.28 (s, 1H), 7.42-7.58 (m, 7H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.87 (d, *J* = 4.0 Hz, 2H), 7.99 (d, *J* = 8.0 Hz, 2H), 9.29 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.4, 22.1, 61.1, 116.0, 126.3, 127.2, 128.2, 128.4, 129.4, 129.7, 129.9, 131.4, 133.4, 138.2, 152.4, 163.8, 190.8; HRMS (ESI) calcd for $C_{28}H_{22}N_2O_3$: 457.1528 (M + Na^+), found: 457.1522.



Ethyl 2-benzoyl-5-cyclopropyl-9-methyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3j**) ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.89-0.92 (m, 2H), 0.98 (t, *J* = 8.0 Hz, 3H), 1.13-1.17 (m, 2H), 2.56 (s, 3H), 2.70-2.73 (m, 2H), 4.10-4.15 (m, 2H), 6.84 (s, 1H), 7.44-7.48 (m, 4H), 7.56-7.60 (m, 2H), 8.02 (d, *J* = 8.0 Hz, 2H), 9.26 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.1, 29.1, 30.8, 31.6, 37.7, 68.8, 105.9, 108.1, 118.4, 121.1, 121.3, 122.8, 124.1, 129.8, 129.9, 132.1, 142.3, 151.1, 172.9; HRMS (ESI) calcd for $C_{25}H_{22}N_2O_3$: 421.1528 ($M + Na^+$), found: 421.1512.

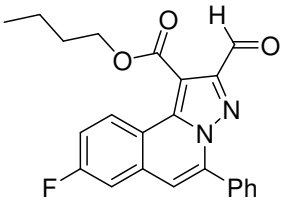


Butyl 2-formyl-8-methoxy-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3k**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.98 (t, *J* = 8.0 Hz, 3H), 1.45-1.49 (m, 2H), 1.79-1.82 (m, 2H), 3.94 (s, 3H), 4.45-4.48 (m, 2H), 7.15 (s, 1H), 7.23-7.25 (m, 2H), 7.51 (s, 3H), 7.82 (s, 2H), 9.05 (d, *J* = 8.0 Hz, 1H), 10.4 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 19.3, 30.6, 55.5, 65.6, 108.3, 116.8, 117.7, 117.8, 128.0, 128.4, 129.6, 129.8, 132.5, 138.5, 139.8, 149.3, 160.4, 164.4, 186.8; HRMS (ESI) calcd for $C_{24}H_{22}N_2O_4$: 425.1477 ($M + Na^+$), found: 425.1485.

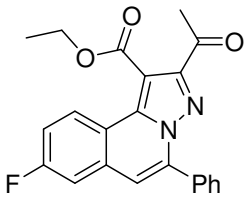


Butyl 9-chloro-2-formyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3l**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, *J* = 8.0 Hz, 3H), 1.48-1.50 (m, 2H), 1.82-1.85 (m, 2H), 4.50-4.53 (m, 2H), 7.27 (s, 1H), 7.52-7.63 (m, 4H), 7.72-7.82 (m, 3H), 9.11 (s, 1H), 10.39 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 19.4, 30.7, 66.2, 109.0, 116.4, 124.7, 125.7, 128.7, 128.9, 129.0, 129.7, 130.1, 130.5, 132.3, 134.3, 149.4,

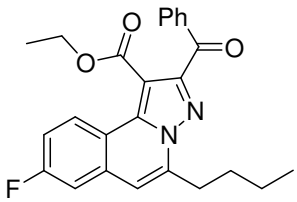
164.1, 186.6; HRMS (ESI) calcd for $C_{23}H_{19}ClN_2O_3$: 429.0982 ($M + H^+$), found: 429.0989.



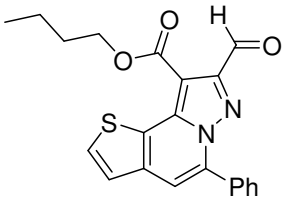
Butyl 8-fluoro-2-formyl-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3m**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, J = 8.0 Hz, 3H), 1.44-1.47 (m, 2H), 1.78-1.85 (m, 2H), 4.46-4.49 (m, 2H), 7.23 (s, 1H), 7.33-7.43 (m, 2H), 7.51-7.52 (m, 3H), 7.81-7.82 (m, 2H), 9.13-9.17 (m, 1H), 10.37 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.1, 35.5, 44.5, 72.8, 106.5, 109.9, 110.1, 113.0, 116.3, 123.1 ($^2J_{CF}$ = 30 Hz), 123.8, 125.8, 126.2, 131.5, 131.6, 139.5, 139.6, 149.4, 151.4, 151.5, 169.3; HRMS (ESI) calcd for $C_{23}H_{19}FN_2O_3$: 413.1277 ($M + Na^+$), found: 413.1295.



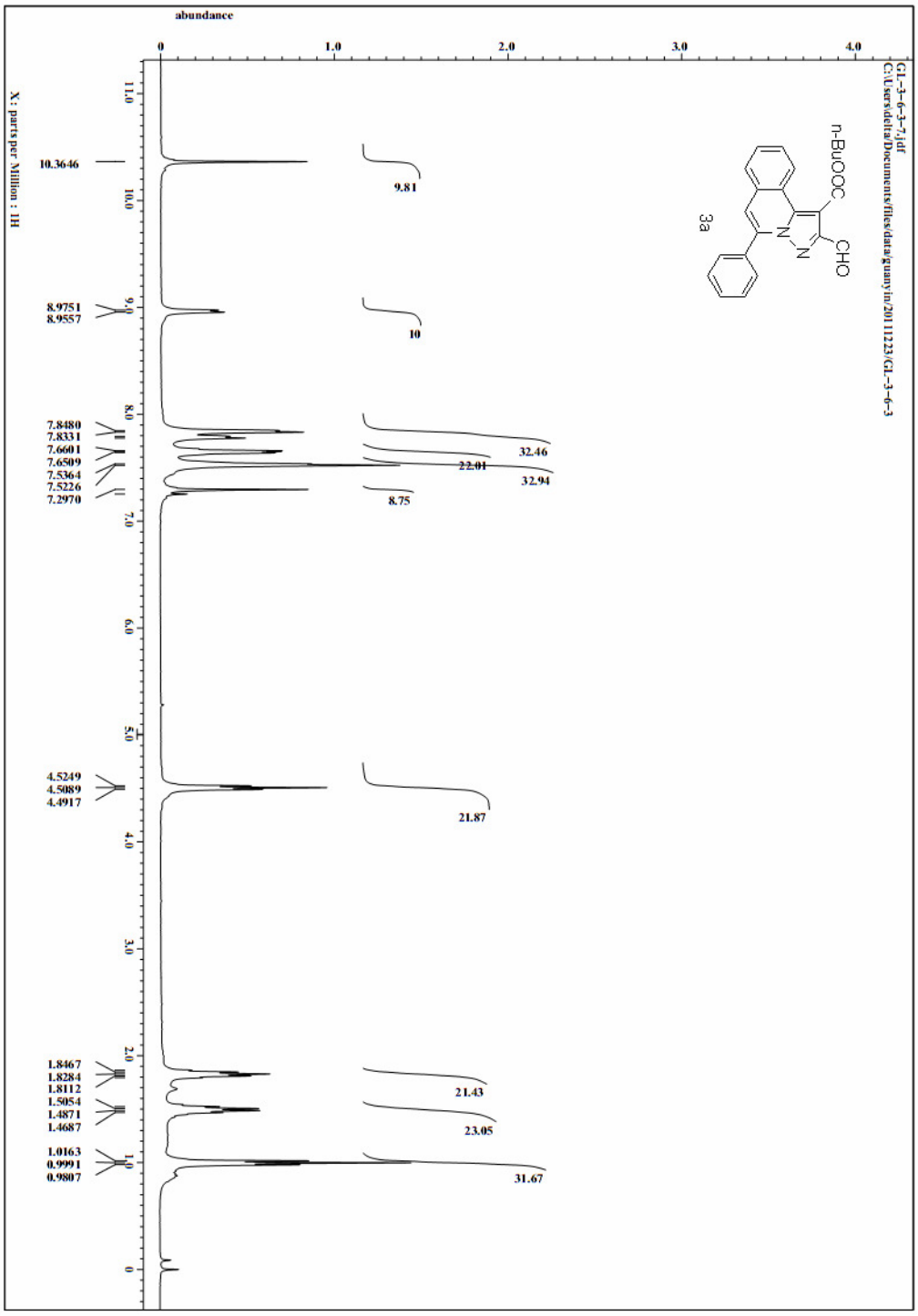
Ethyl 2-acetyl-8-fluoro-5-phenyl*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3n**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.43 (t, J = 8.0 Hz, 3H), 2.63 (s, 10H), 4.49-4.54 (m, 2H), 7.17 (s, 1H), 7.30-7.53 (m, 5H), 7.84-7.86 (m, 2H), 8.61-8.65 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.3, 69.7, 105.9, 107.6, 109.9, 110.1, 112.0 ($^2J_{CF}$ = 30 Hz), 112.9, 113.5, 113.7, 122.1, 122.8, 123.8, 124.0, 131.4, 140.1, 149.2, 151.2, 152.4, 175.7; HRMS (ESI) calcd for $C_{22}H_{17}FN_2O_3$: 399.1121 ($M + Na^+$), found: 399.1103.

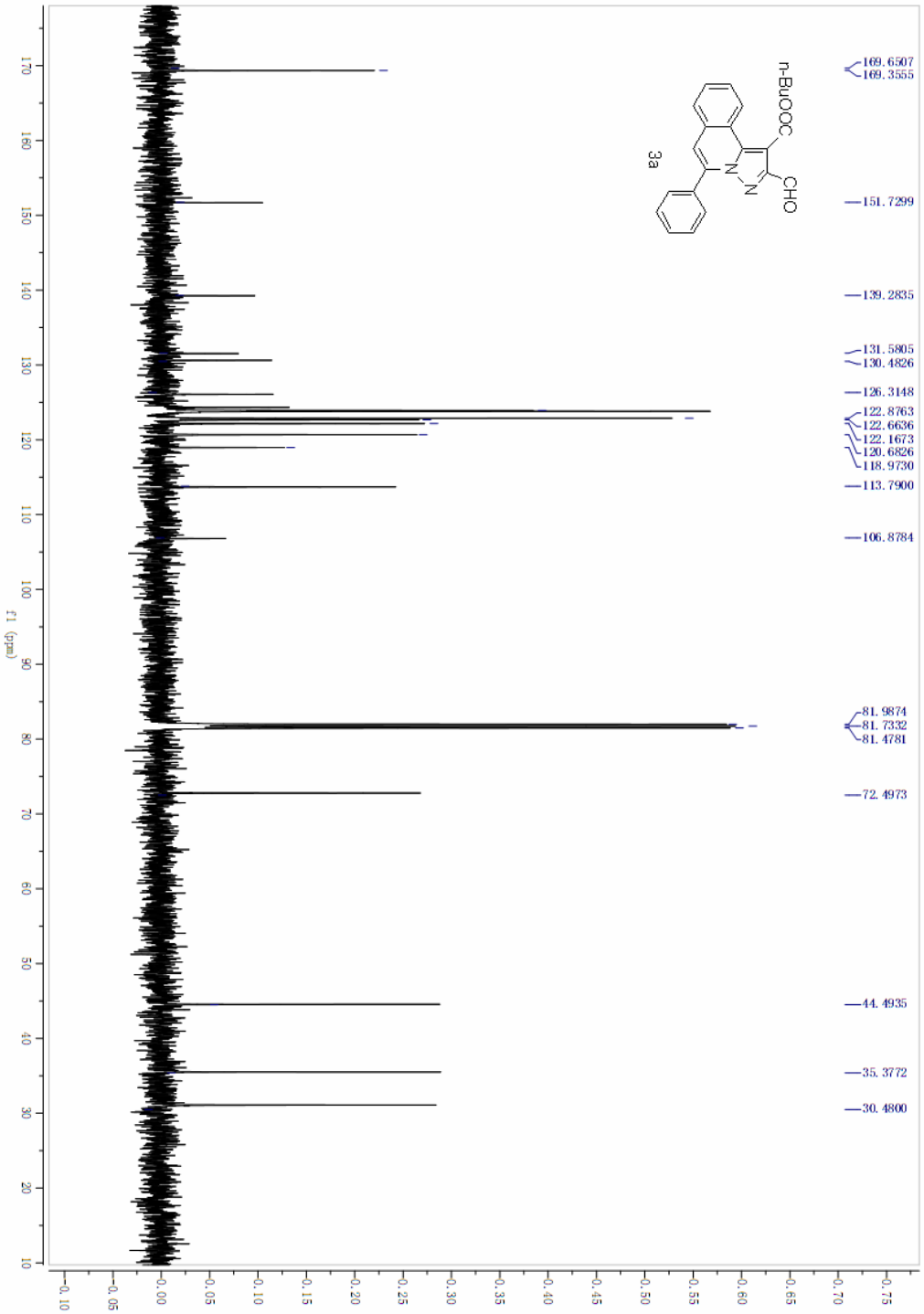


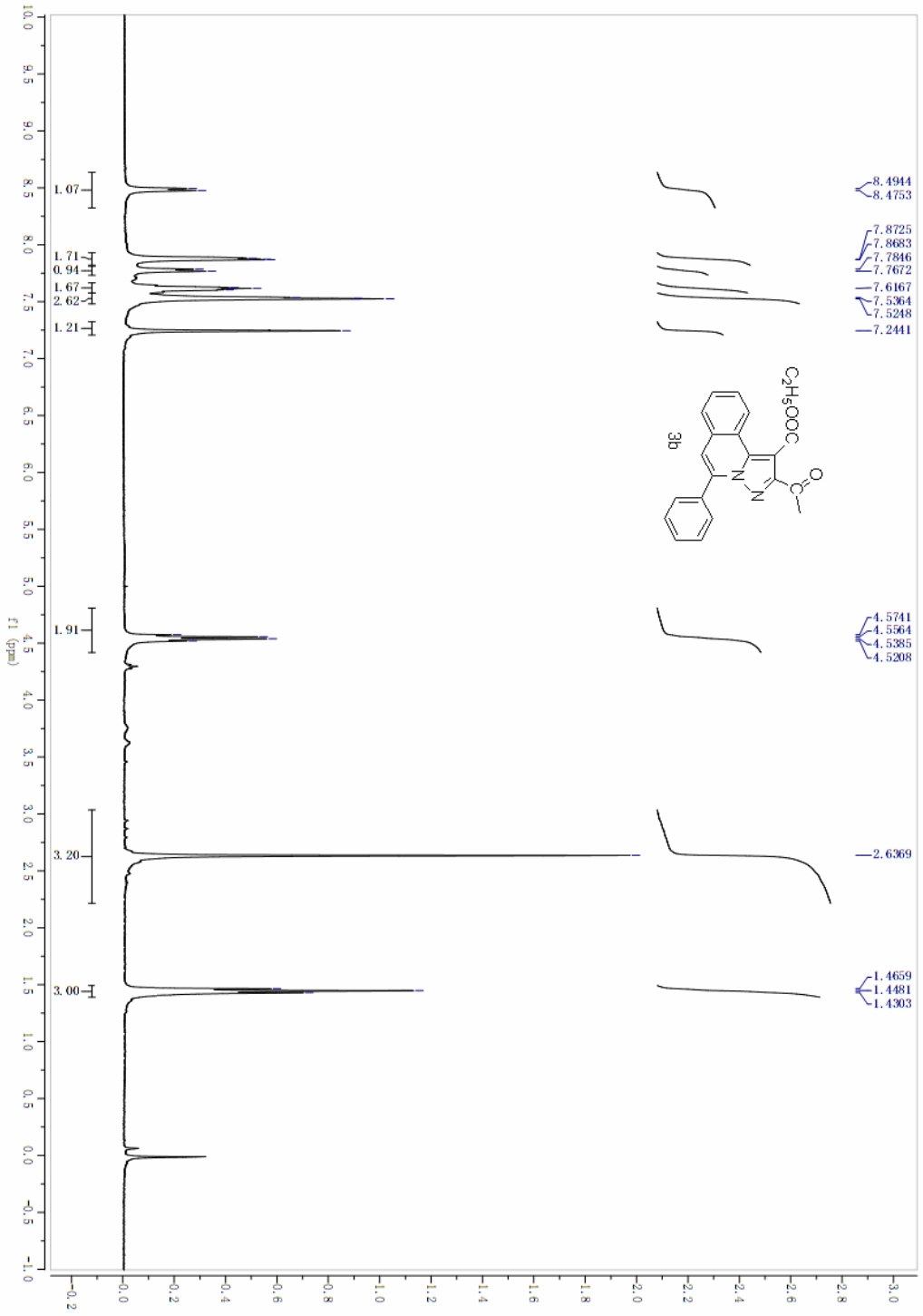
Ethyl 2-benzoyl-5-butyl-8-fluoro*H*-pyrazolo[5,1-*a*]isoquinoline-1-carboxylate (**3o**): ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.94-0.97 (m, 6H), 1.44-1.50 (m, 2H), 1.80-1.86 (m, 2H), 3.17-3.20 (m, 2H), 4.09-4.14 (m, 2H), 7.02 (s, 1H), 7.31-7.34 (m, 2H), 7.36-7.38 (m, 2H), 7.45-7.47 (m, 2H), 7.99 (d, J = 8.0 Hz, 2H), 9.62-9.66 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.8, 31.2, 38.0, 43.2, 43.8 ($^{2}J_{CF}$ = 30 Hz), 44.6, 69.0, 105.5, 109.0, 110.0, 112.7, 115.9, 122.9, 124.0, 125.3 ($^{1}J_{CF}$ = 240 Hz), 129.7, 131.0, 131.6, 132.6, 142.4, 149.3, 151.1 ($^{2}J_{CF}$ = 40 Hz), 172.8; HRMS (ESI) calcd for $C_{25}H_{23}N_2O_3$: 441.1590 ($M + H^+$), found: 441.1609.

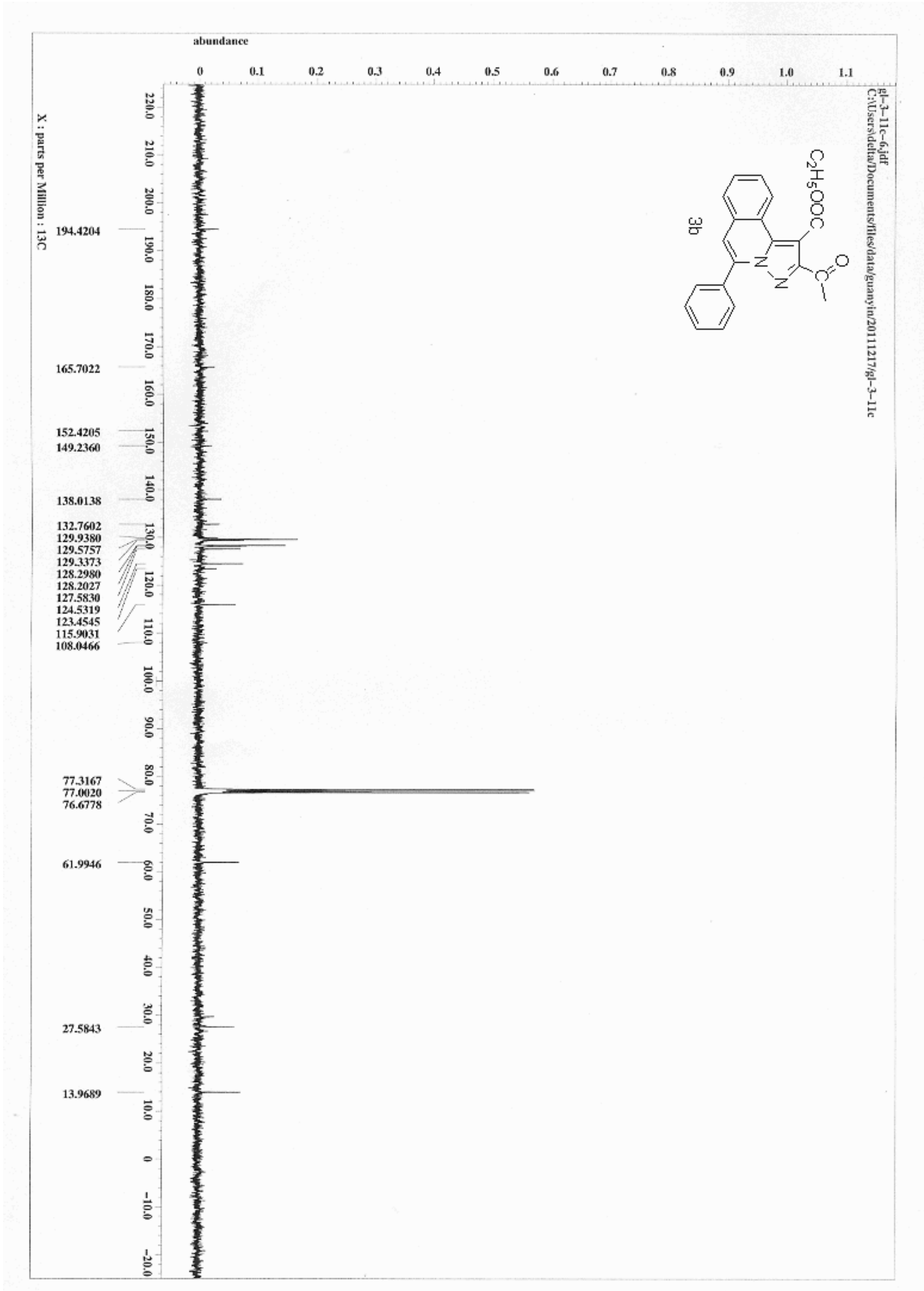


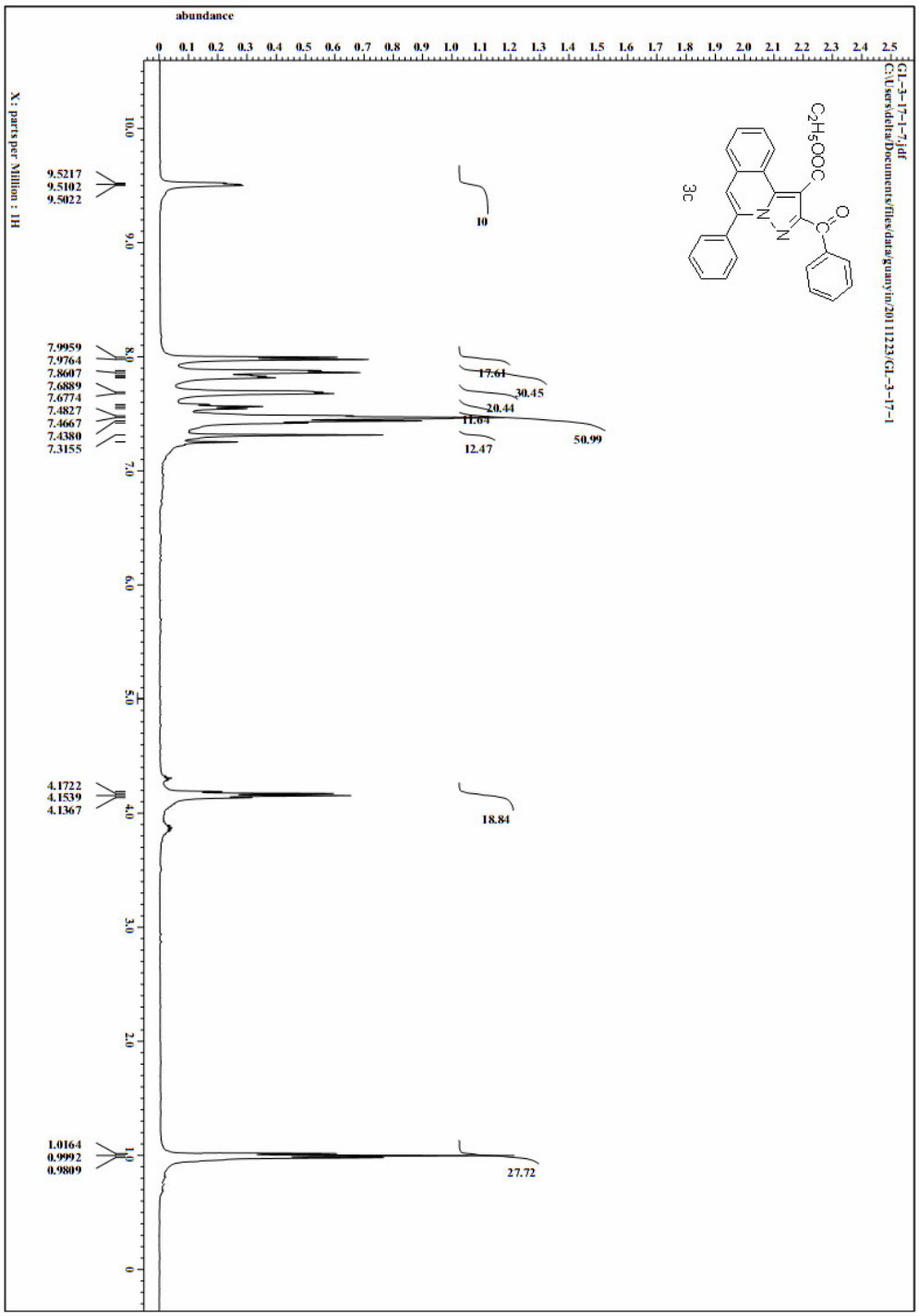
Compound **3p**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.99 (t, J = 8.0 Hz, 3H), 1.47-1.51 (m, 2H), 1.83-1.86 (m, 2H), 4.48-4.51 (m, 2H), 7.47-7.51 (m, 5H), 7.79-7.84 (m, 3H), 10.70 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.1, 35.6, 44.7, 72.2, 97.4, 110.5, 113.2, 119.2, 122.1, 123.8, 130.5, 130.8, 139.3, 150.6, 169.7, 187.9; HRMS (ESI) calcd for $C_{21}H_{18}N_2O_3S$: 401.0936 ($M + Na^+$), found: 401.0951.

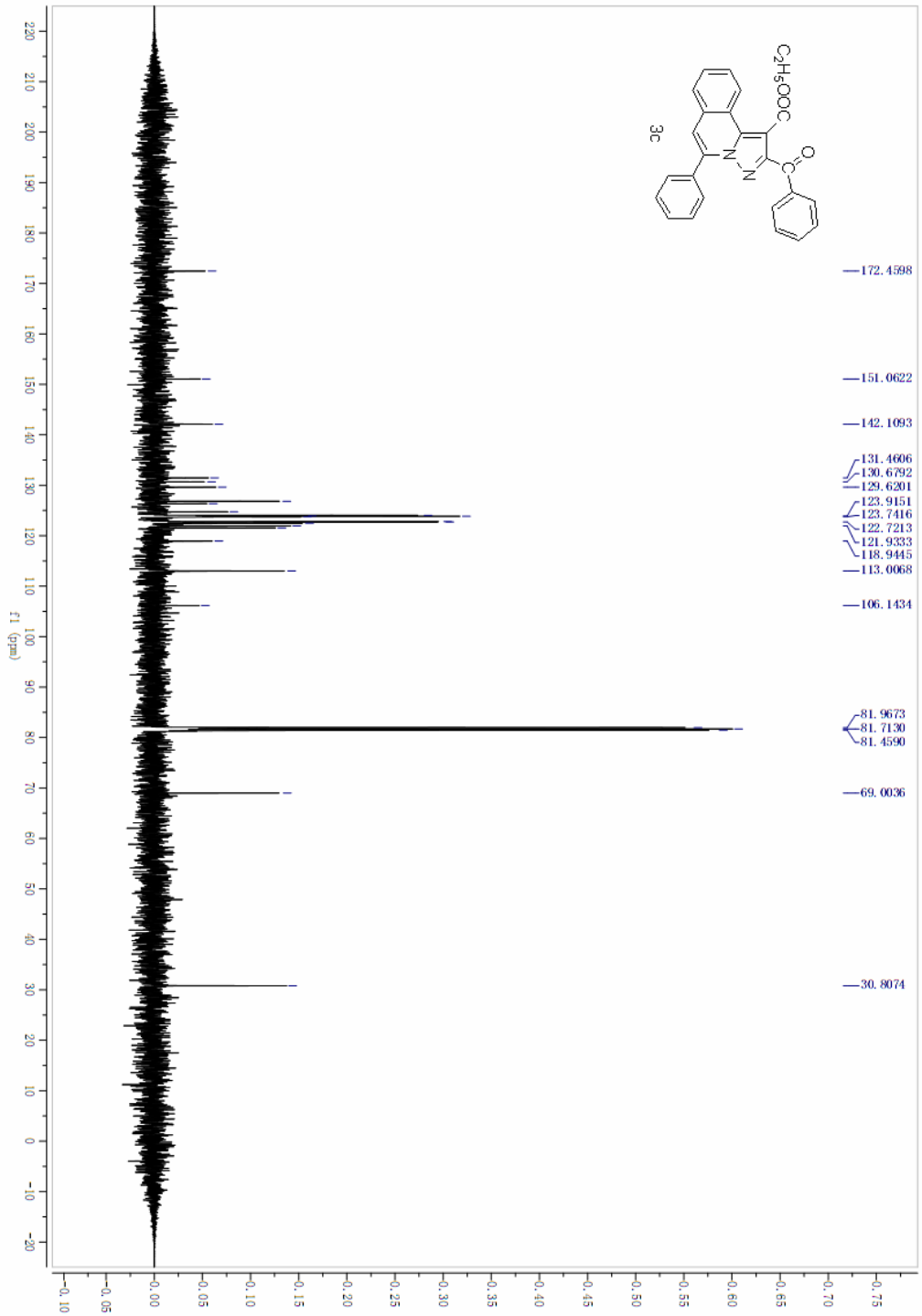

abundance
X : parts per Million : 1H
G1_3-6-3-7.jdf
C:\Users\delta\Documents\files\data\guanyin\20111223\G1_3-6-3
n-BuOOC
CHO
3a
10.3646
8.9751
8.9557
7.8480
7.8331
7.6601
7.6509
7.5364
7.5226
7.2970
4.5249
4.5089
4.4917
1.8467
1.8284
1.8112
1.5054
1.4871
1.4687
1.0163
0.9991
0.9807
9.81
10
32.46
22.01
32.94
8.75
21.87
21.43
23.05
31.67

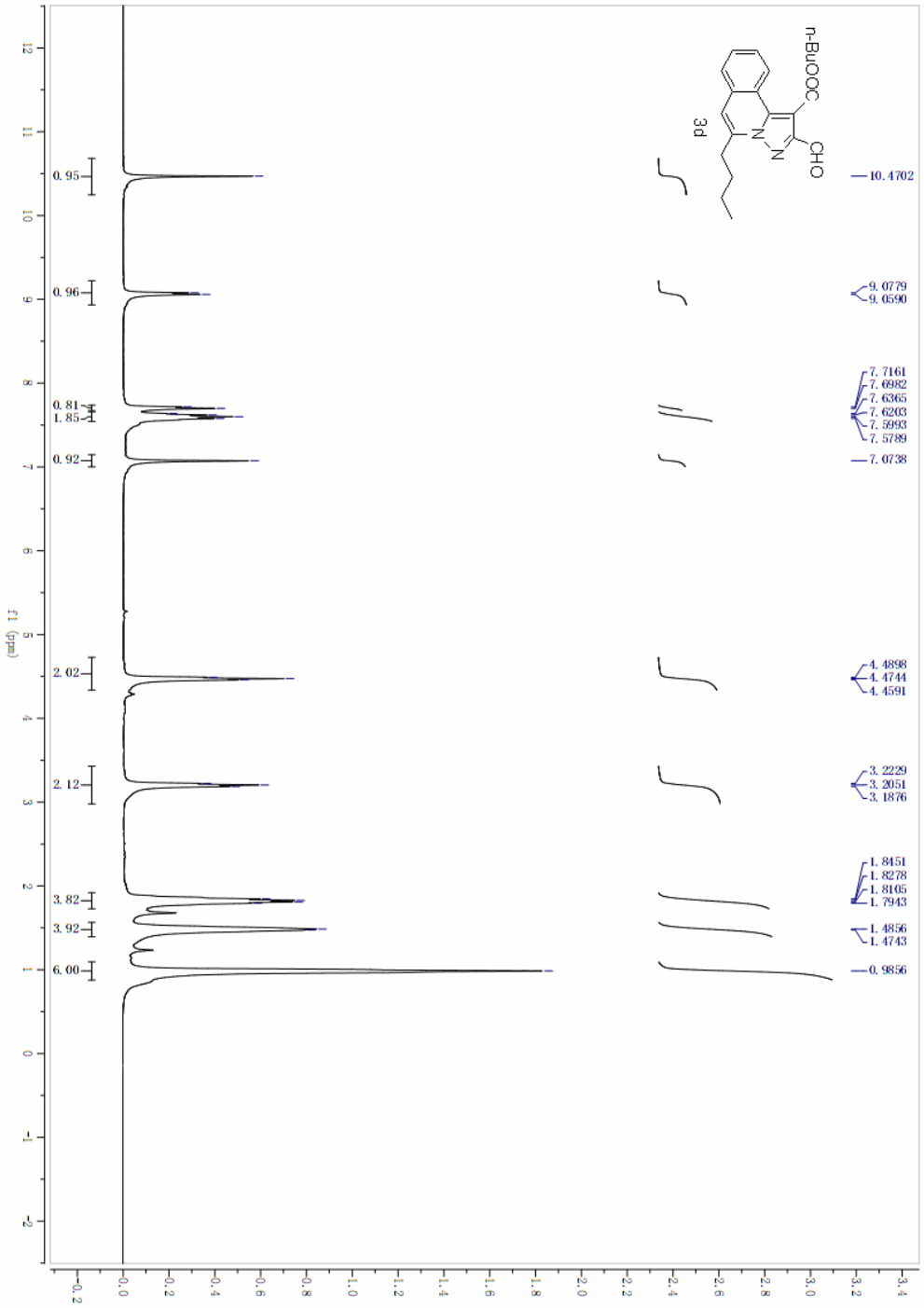

n-BuOOC
CHO
3a
169.6507
169.3555
151.7299
139.2835
131.5805
130.4826
126.3148
122.8763
122.6636
122.1673
120.6826
118.9730
113.7900
106.8784
81.9874
81.7332
81.4781
72.4973
44.4935
35.3772
30.4800
f1 (ppm)

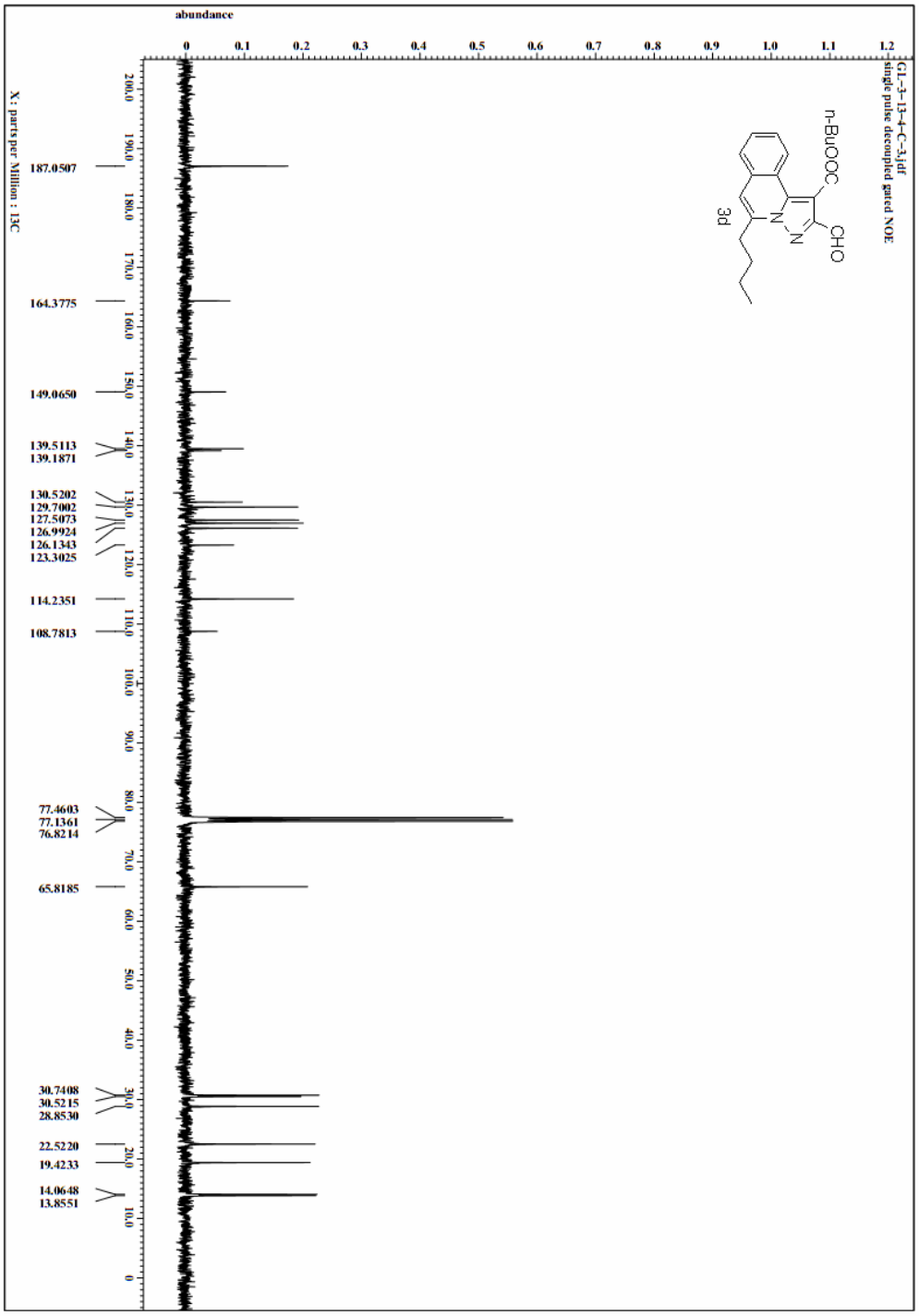

1.07
1.71
0.94
1.67
2.62
1.21
1.91
3.20
3.00
8.4944
8.4753
7.8725
7.8683
7.7846
7.7672
7.6167
7.5364
7.5248
7.2441
4.5741
4.5564
4.5385
4.5208
2.6369
1.4659
1.4481
1.4303
3b
C2H5OOC
f1 (ppm)

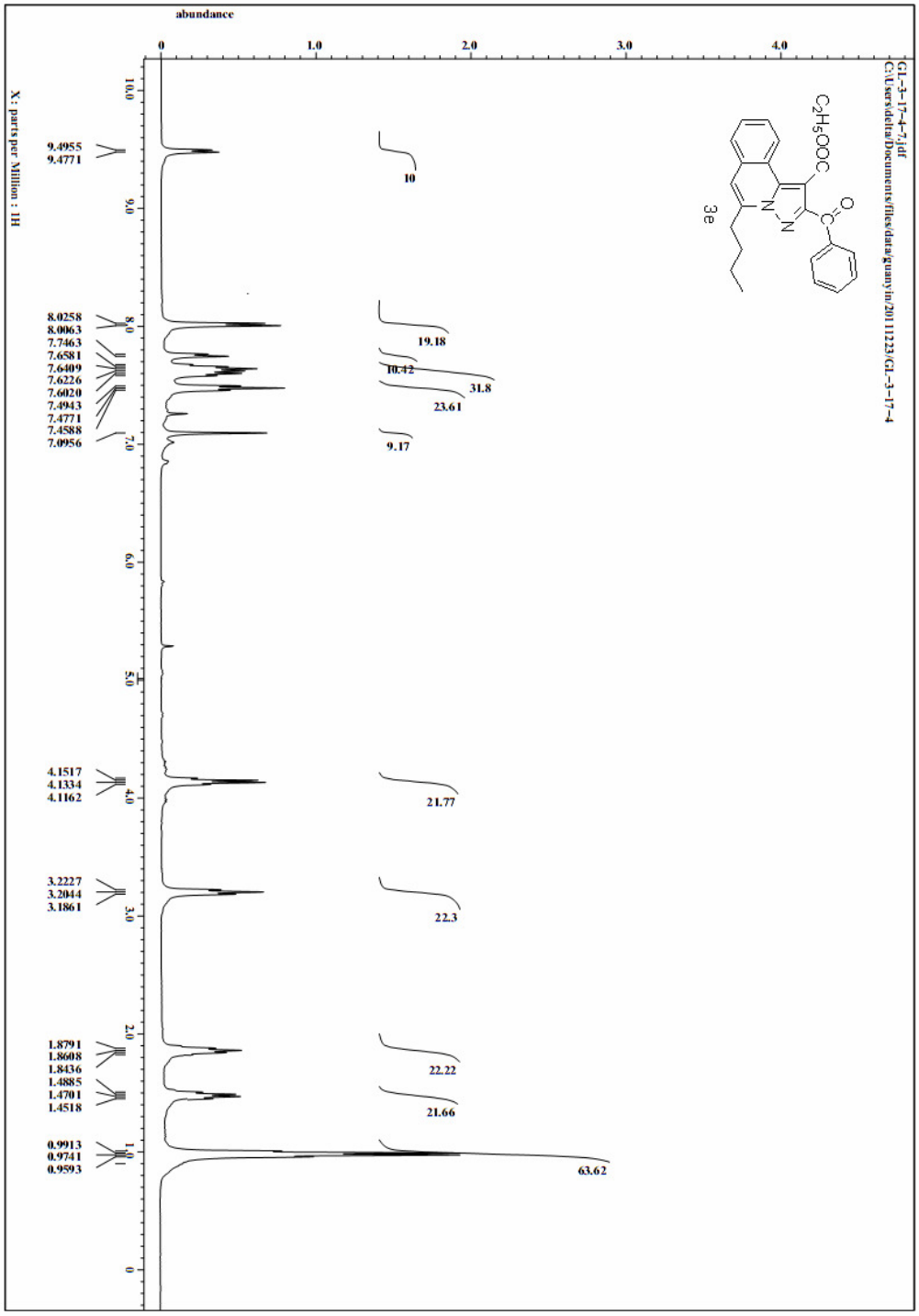

abundance
X : parts per Million : 13C
gl-3-11c-6.jdf
C:\Users\delta\Documents\files\data\guanyin\20111217\gl-3-11c
C2H5OOC
3b
194.4204
165.7022
152.4205
149.2360
138.0138
132.7602
129.9380
129.5757
129.3373
128.2980
128.2027
127.5830
124.5319
123.4545
115.9031
108.0466
77.3167
77.0020
76.6778
61.9946
27.5843
13.9689

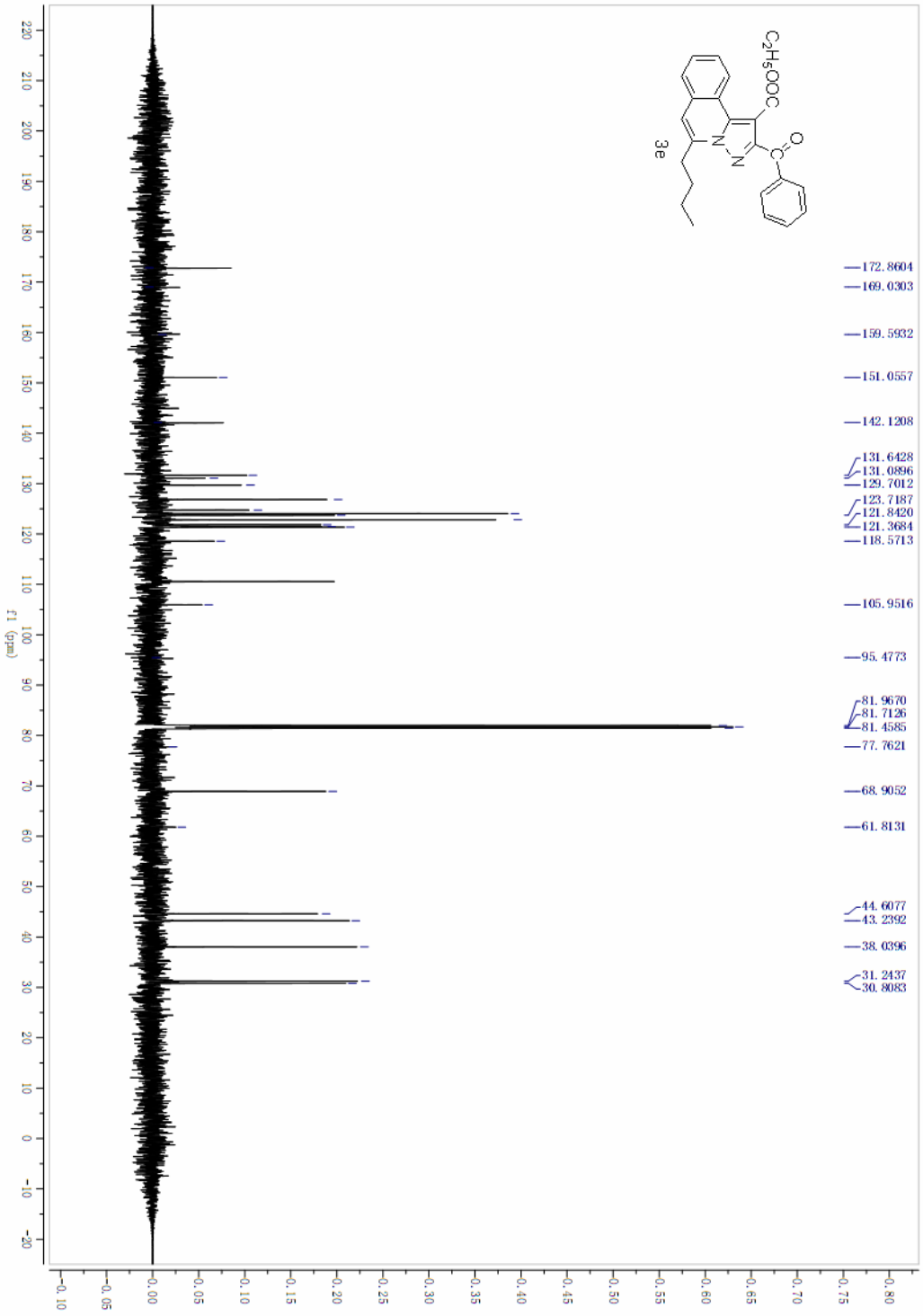

abundance
X : parts per Million : 1H
9.5217
9.5102
9.5022
7.9959
7.9764
7.8607
7.6889
7.6774
7.4827
7.4667
7.4380
7.3155
4.1722
4.1539
4.1367
1.0164
0.9992
0.9809
10
17.61
30.45
20.44
11.64
50.99
12.47
18.84
27.72
3c
C2H5OOC
G1--3--17--1--7.jdf
C:\Users\delta\Documents\files\data\guanyin\20111223\G1--3--17--1

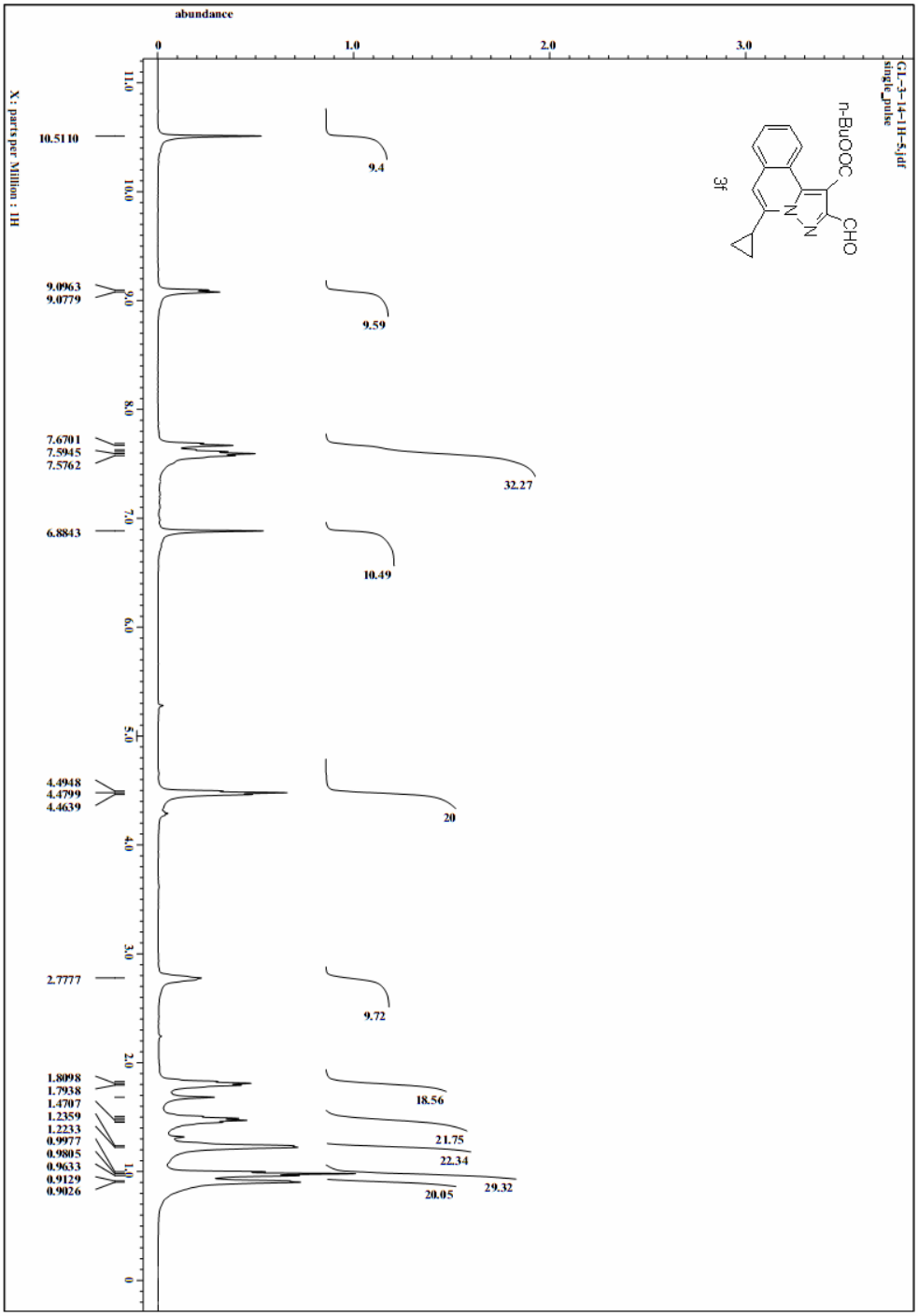

3c
C_2H_5OOC
O
O
N
N
172.4598
151.0622
142.1093
131.4606
130.6792
129.6201
123.9151
123.7416
122.7213
121.9333
118.9445
113.0068
106.1434
81.9673
81.7130
81.4590
69.0036
30.8074
f1 (ppm)

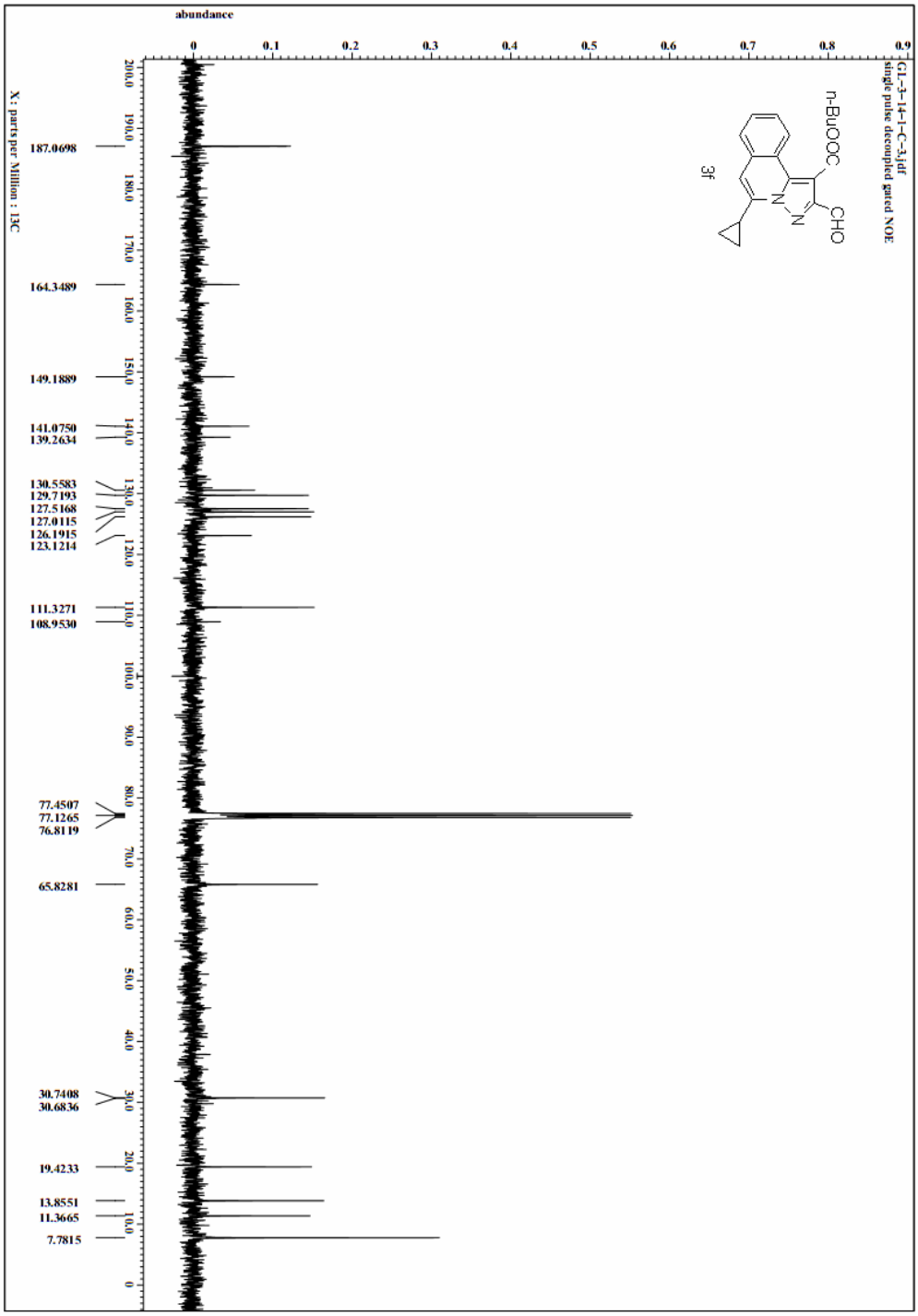

n-BuOOC
CHO
N
N
3d
0.95
0.96
0.81
1.85
0.92
2.02
2.12
3.82
3.92
6.00
10.4702
9.0779
9.0590
7.7161
7.6982
7.6365
7.6203
7.5993
7.5789
7.0738
4.4898
4.4744
4.4591
3.2229
3.2051
3.1876
1.8451
1.8278
1.8105
1.7943
1.4856
1.4743
0.9856
f1 (ppm)

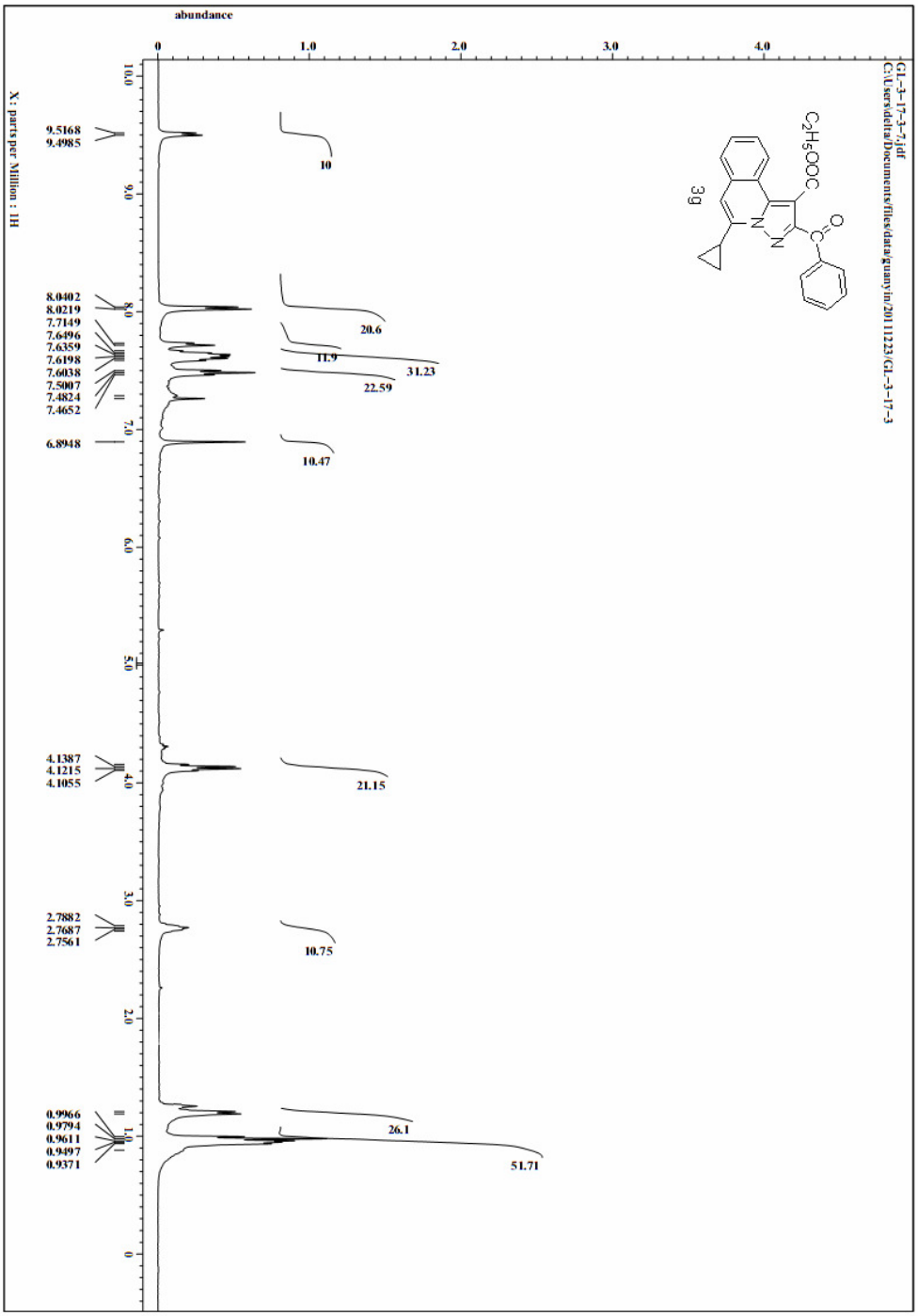

abundance
X: parts per Million : 13C
GL-3-13-4-C--3.jdf
single pulse decoupled gated NOE
n-BuOOC
CHO
3d
187.0507
164.3775
149.0650
139.5113
139.1871
130.5202
129.7002
127.5073
126.9924
126.1343
123.3025
114.2351
108.7813
77.4603
77.1361
76.8214
65.8185
30.7408
30.5215
28.8530
22.5220
19.4233
14.0648
13.8551

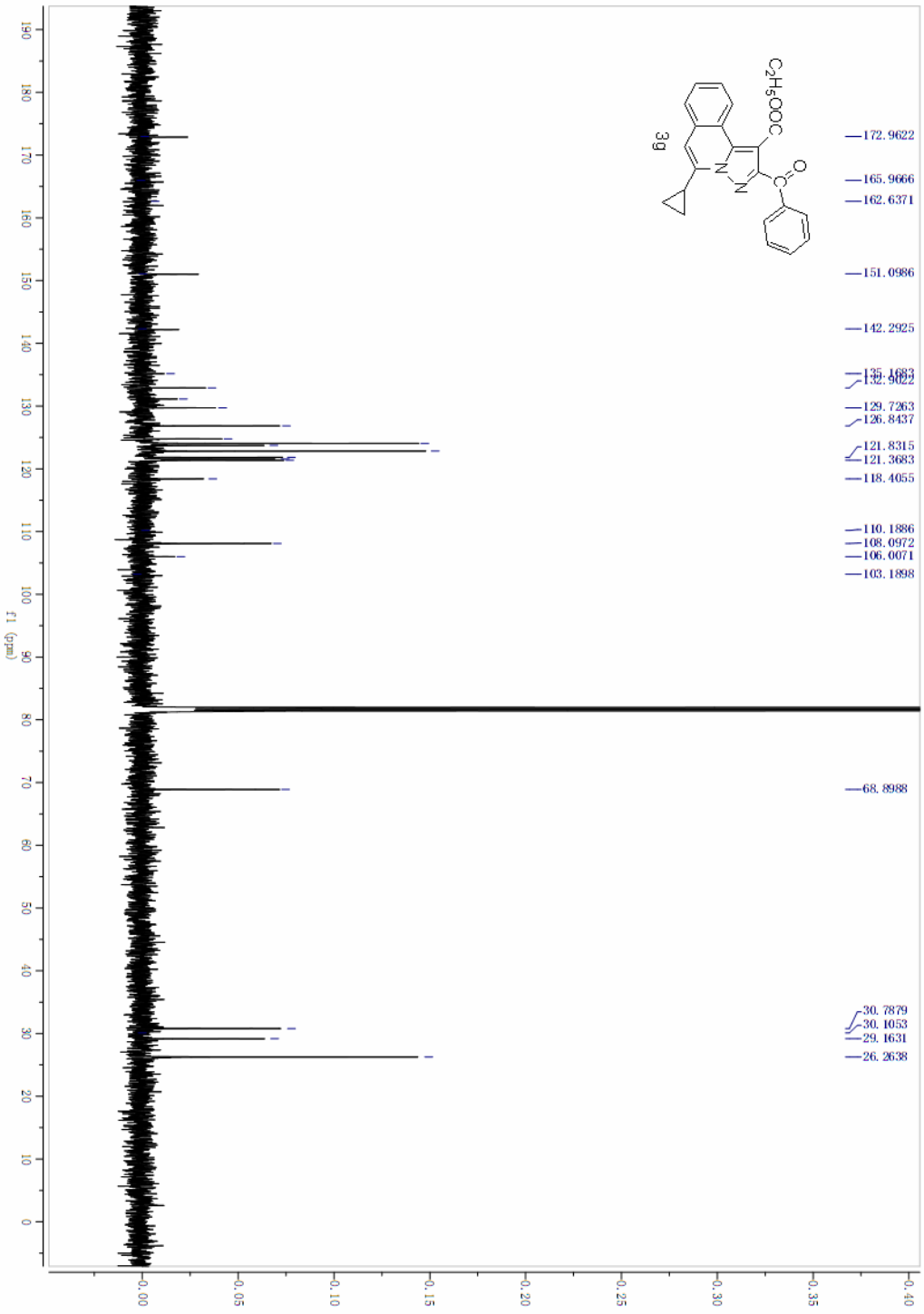

abundance
X : parts per Million : 1H
GL-3-17-4-7.jdf
C:\Users\delta\Documents\files\data\guanyin\20111223\GL-3-17-4
C2H5OOC
3e
9.4955
9.4771
8.0258
8.0063
7.7463
7.6581
7.6409
7.6226
7.6020
7.4943
7.4771
7.4588
7.0956
4.1517
4.1334
4.1162
3.2227
3.2044
3.1861
1.8791
1.8608
1.8436
1.4885
1.4701
1.4518
0.9913
0.9741
0.9593
10
19.18
10.42
31.8
23.61
9.17
21.77
22.3
22.22
21.66
63.62

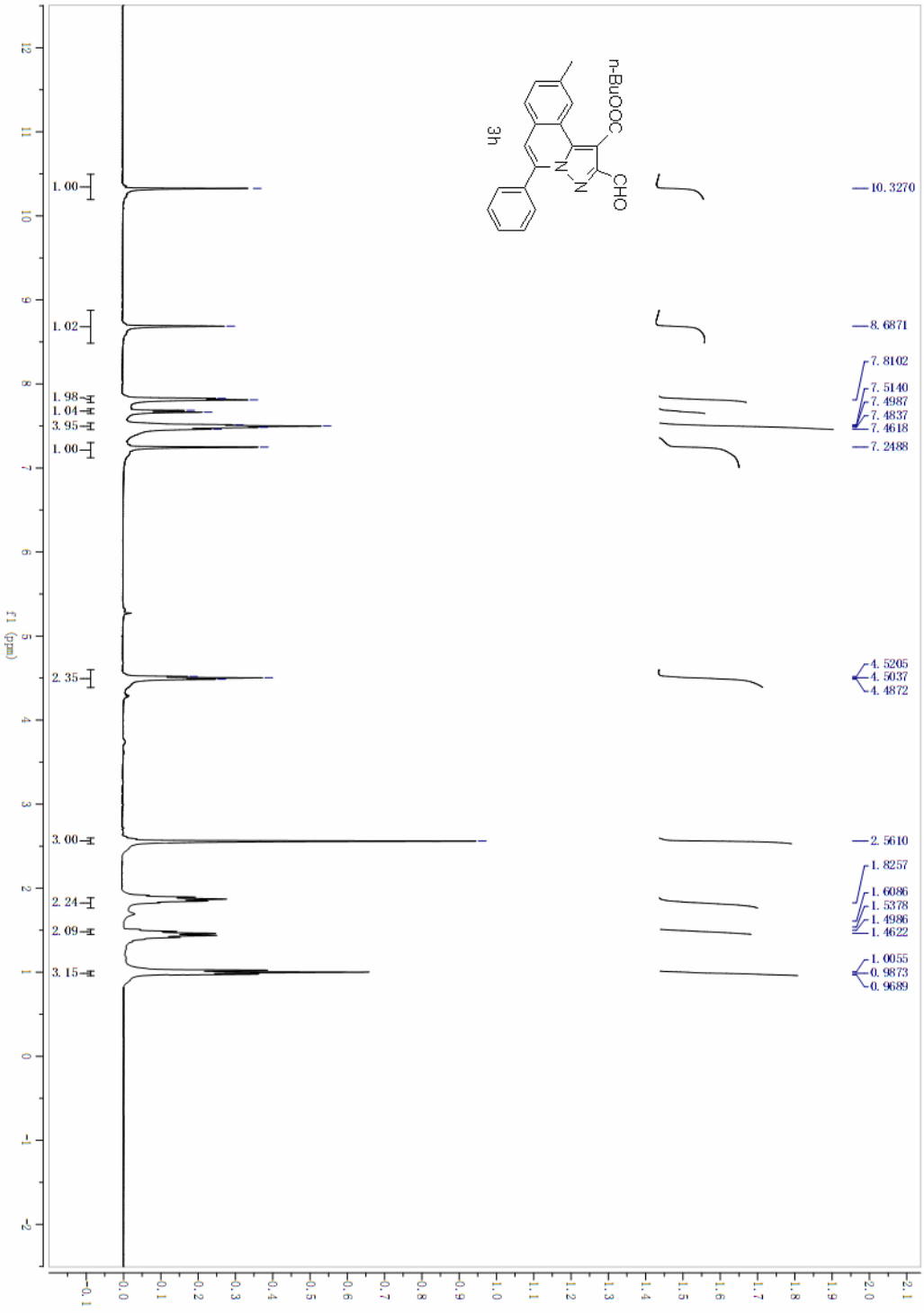

C2H5OOC
O
O
N
N
3e
f1 (ppm)
172.8604
169.0303
159.5932
151.0557
142.1208
131.6428
131.0896
129.7012
123.7187
121.8420
121.3684
118.5713
105.9516
95.4773
81.9670
81.7126
81.4585
77.7621
68.9052
61.8131
44.6077
43.2392
38.0396
31.2437
30.8083

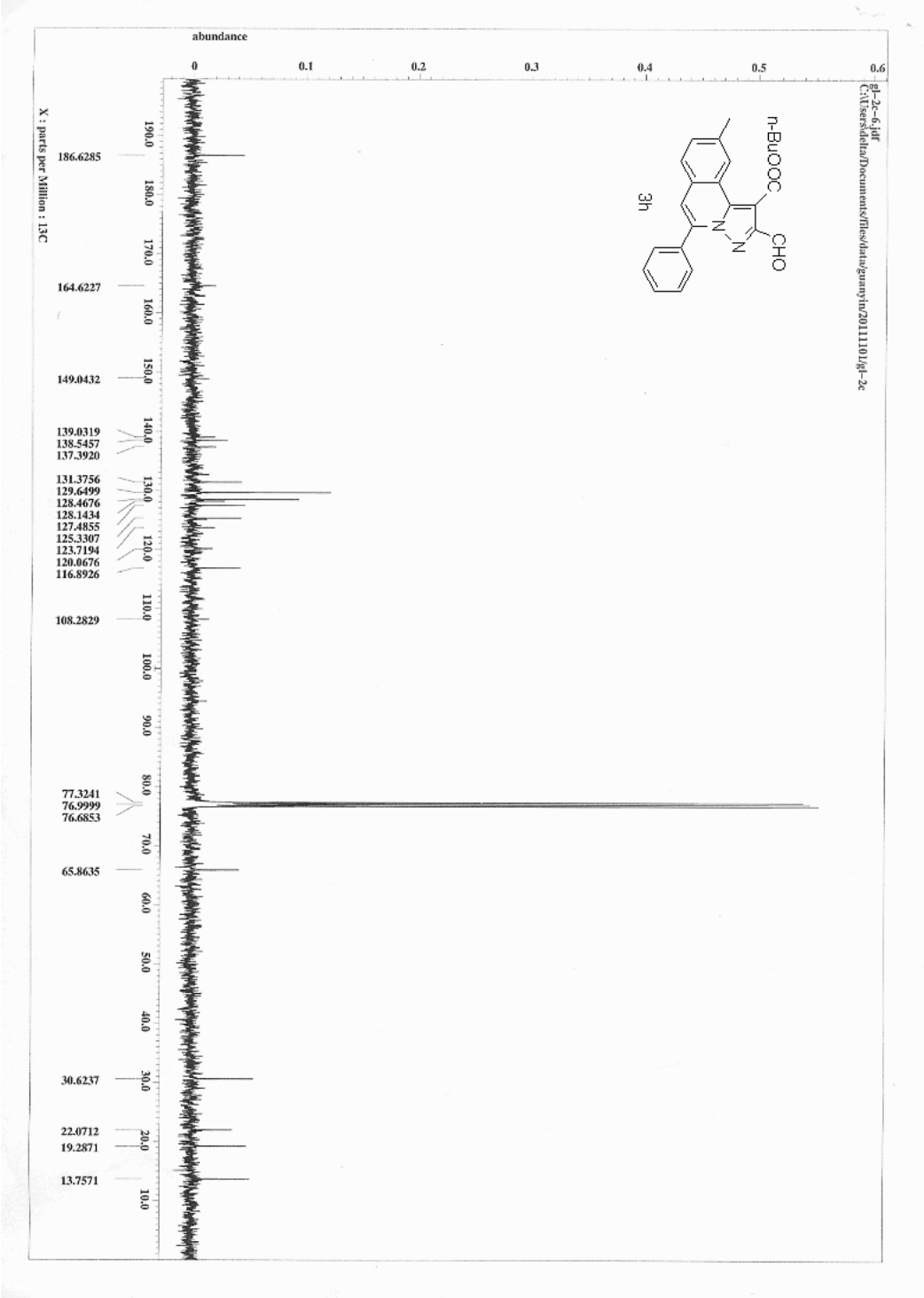

abundance
X: parts per Million : 1H
GL-3-14-111-5.jdf
single_pulse
n-BuOOC
CHO
3f
10.5110
9.0963
9.0779
7.6701
7.5945
7.5762
6.8843
4.4948
4.4799
4.4639
2.7777
1.8098
1.7938
1.4707
1.2359
1.2233
0.9977
0.9805
0.9633
0.9129
0.9026
9.4
9.59
32.27
10.49
20
9.72
18.56
21.75
22.34
29.32
20.05

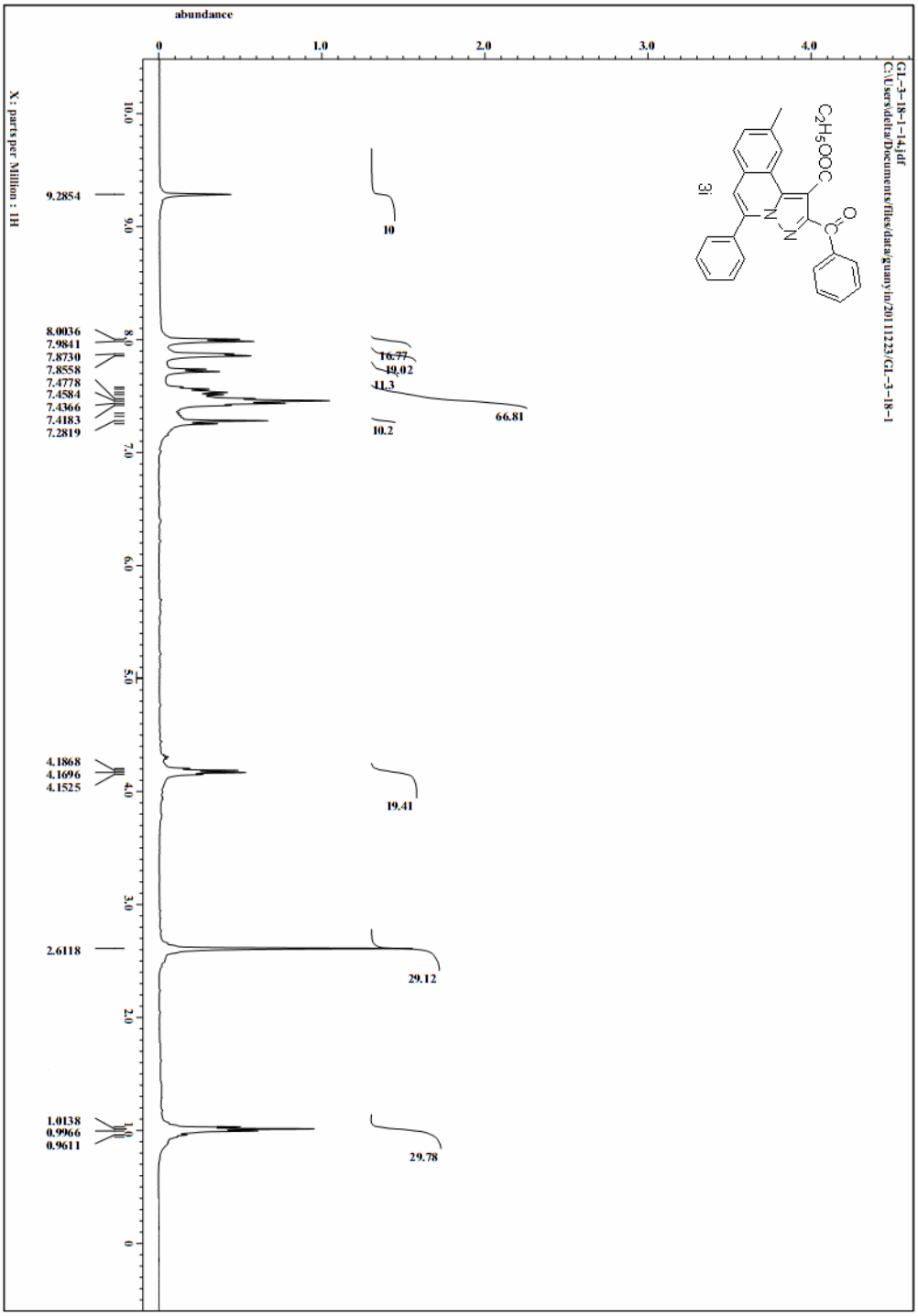

abundance
X: parts per Million : 13C
GL-3-14-1-C-3.jdf
single pulse decoupled gated NOE
n-BuOOC
CHO
3f
187.0698
164.3489
149.1889
141.0750
139.2634
130.5583
129.7193
127.5168
127.0115
126.1915
123.1214
111.3271
108.9530
77.4507
77.1265
76.8119
65.8281
30.7408
30.6836
19.4233
13.8551
11.3665
7.7815

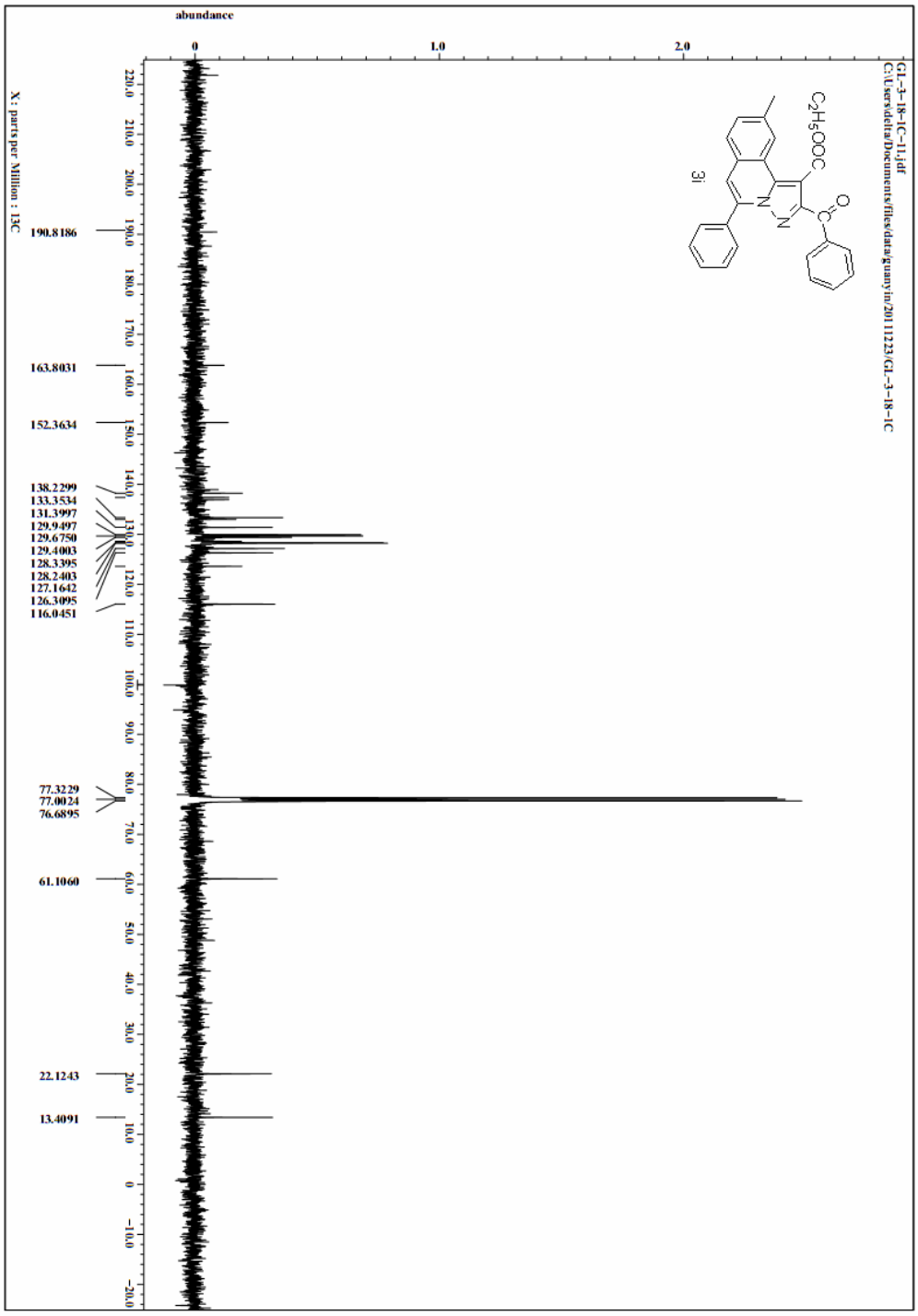

abundance
X : parts per Million : 111
9.5168
9.4985
8.0402
8.0219
7.7149
7.6496
7.6359
7.6198
7.6038
7.5007
7.4824
7.4652
6.8948
4.1387
4.1215
4.1055
2.7882
2.7687
2.7561
0.9966
0.9794
0.9611
0.9497
0.9371
10
20.6
11.9
31.23
22.59
10.47
21.15
10.75
26.1
51.71
3g
C2H5OOC
C:\Users\delta\Documents\files\data\guanyin\20111223\GL-3-17-3
GL-3-17-3-7.jdf

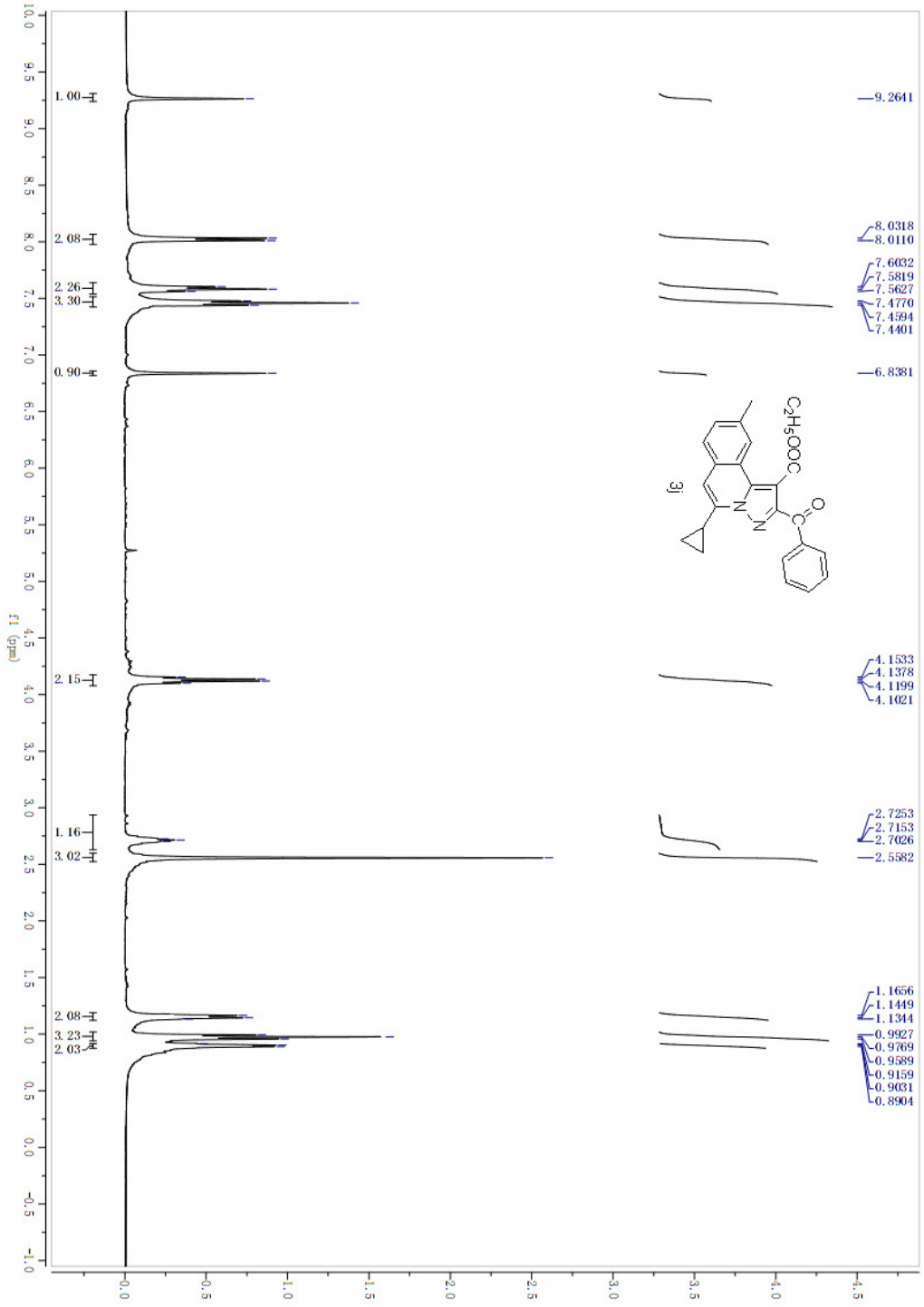

3g
C2H5OOC
O
N
N
172.9622
165.9666
162.6371
151.0986
142.2925
135.1683
132.9022
129.7263
126.8437
121.8315
121.3683
118.4055
110.1886
108.0972
106.0071
103.1898
68.8988
30.7879
30.1053
29.1631
26.2638
f1 (ppm)

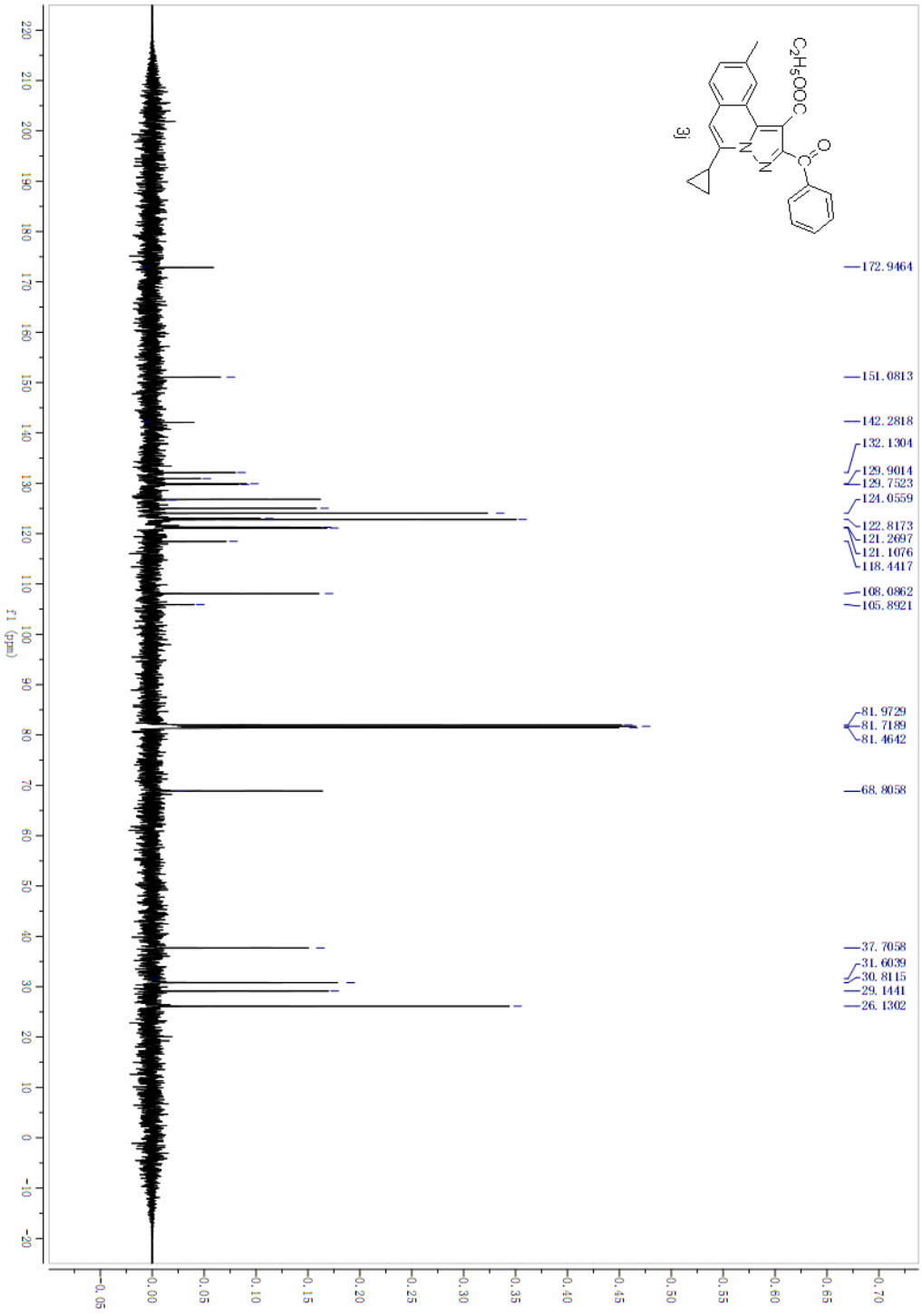

3h
n-BuOOC
CHO
1.00
1.02
1.98
1.04
3.95
1.00
2.35
3.00
2.24
2.09
3.15
10.3270
8.6871
7.8102
7.5140
7.4987
7.4837
7.4618
7.2488
4.5205
4.5037
4.4872
2.5610
1.8257
1.6086
1.5378
1.4986
1.4622
1.0055
0.9873
0.9689
f1 (ppm)

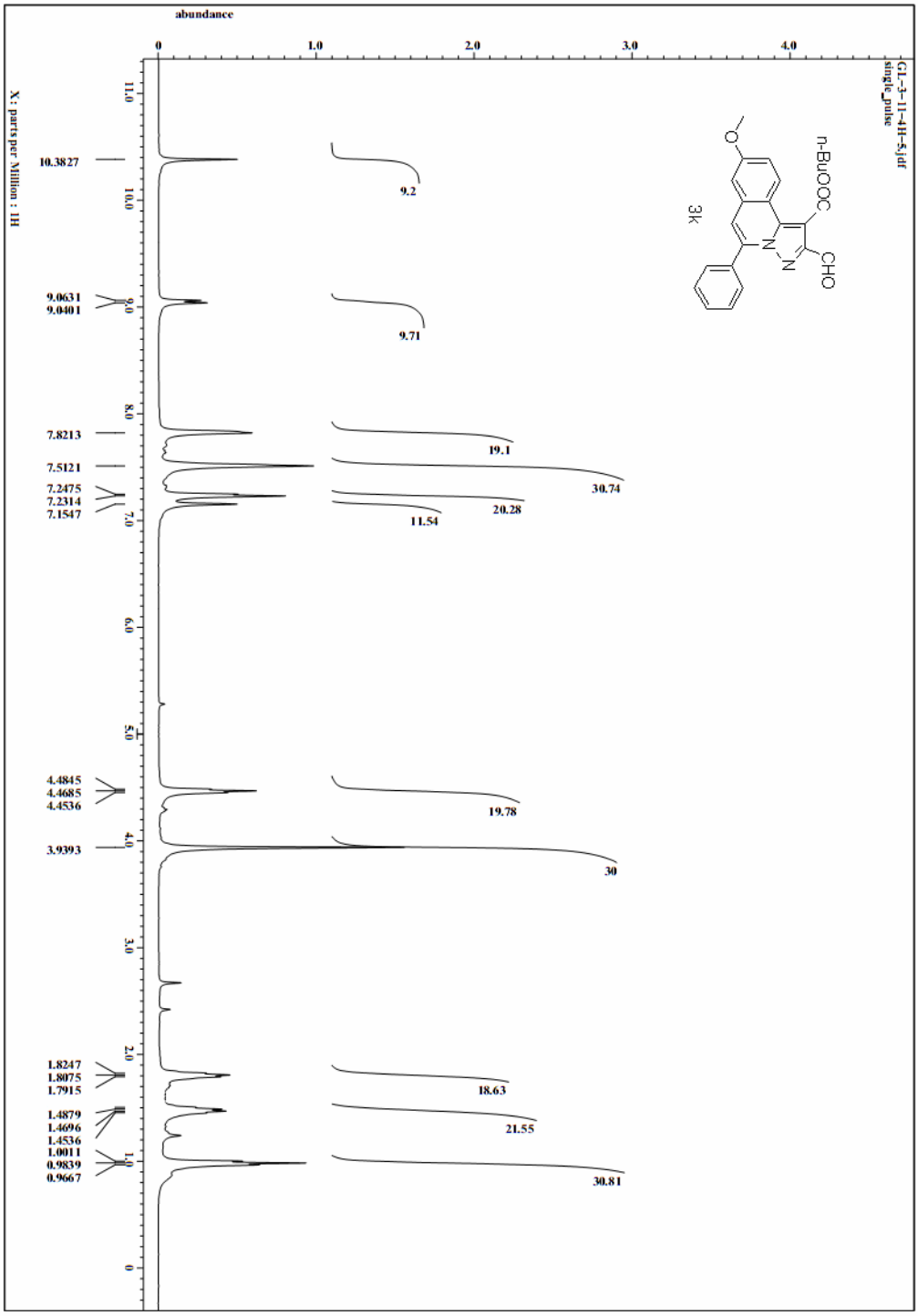

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
X : parts per Million : 13C
186.6285
164.6227
149.0432
139.0319
138.5457
137.3920
131.3756
129.6499
128.4676
128.1434
127.4855
125.3307
123.7194
120.0676
116.8926
108.2829
77.3241
76.9999
76.6853
65.8635
30.6237
22.0712
19.2871
13.7571
n-BuOOC
CHO
N
N
3h
gl-2c-6.jdf
C:\Users\dell\a\Documents\files\data\pranayjn\20111101\gl-2c

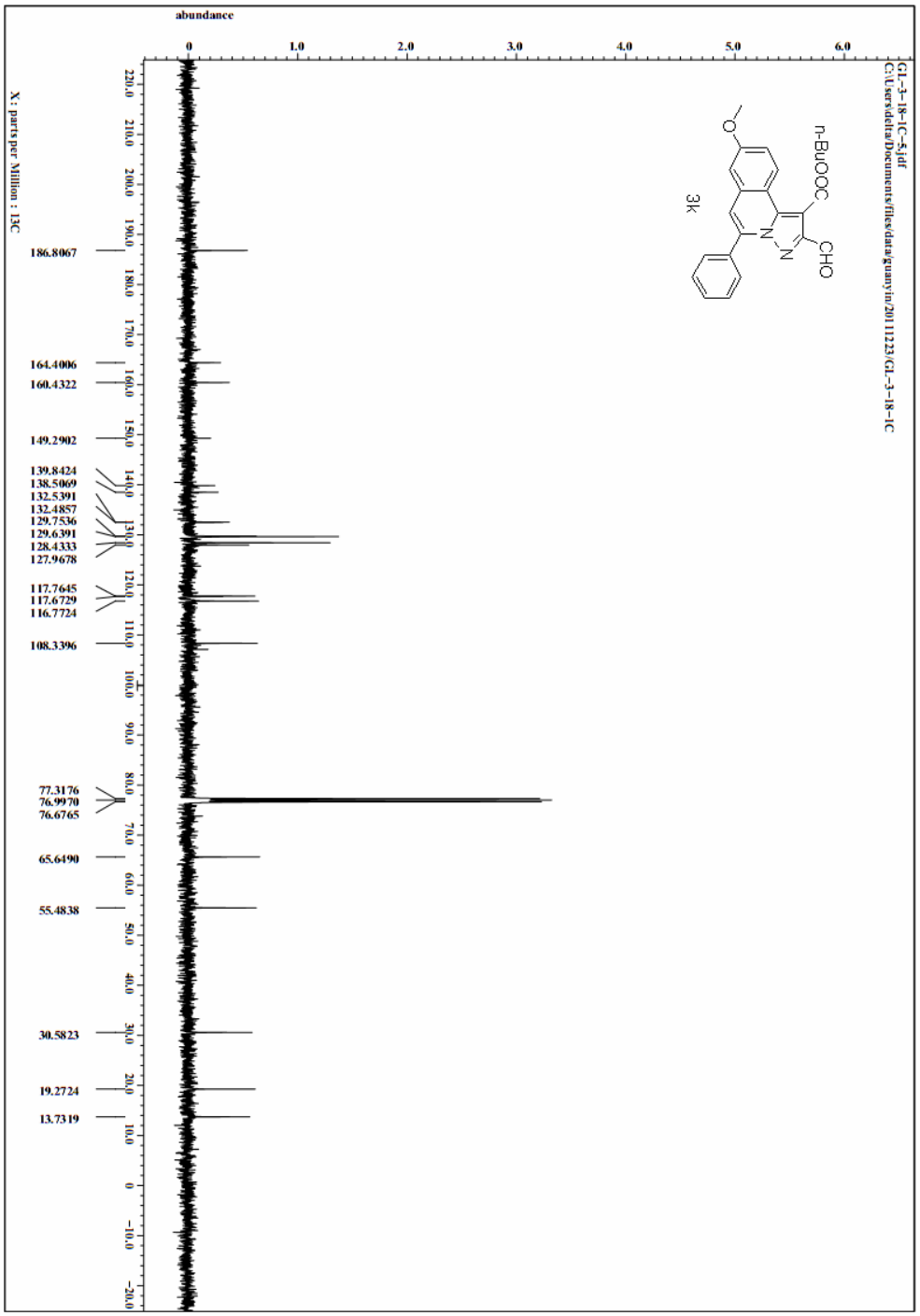

abundance
X : parts per Million : 1H
GL-3-18-1-14.jdf
C:\Users\delta\Documents\files\data\guanyin\20111223\GL-3-18-1
9.2854
8.0036
7.9841
7.8730
7.8558
7.4778
7.4584
7.4366
7.4183
7.2819
4.1868
4.1696
4.1525
2.6118
1.0138
0.9966
0.9611
10
16.77
19.02
11.3
66.81
10.2
19.41
29.12
29.78
3l
C2H5OOC
O
N
N

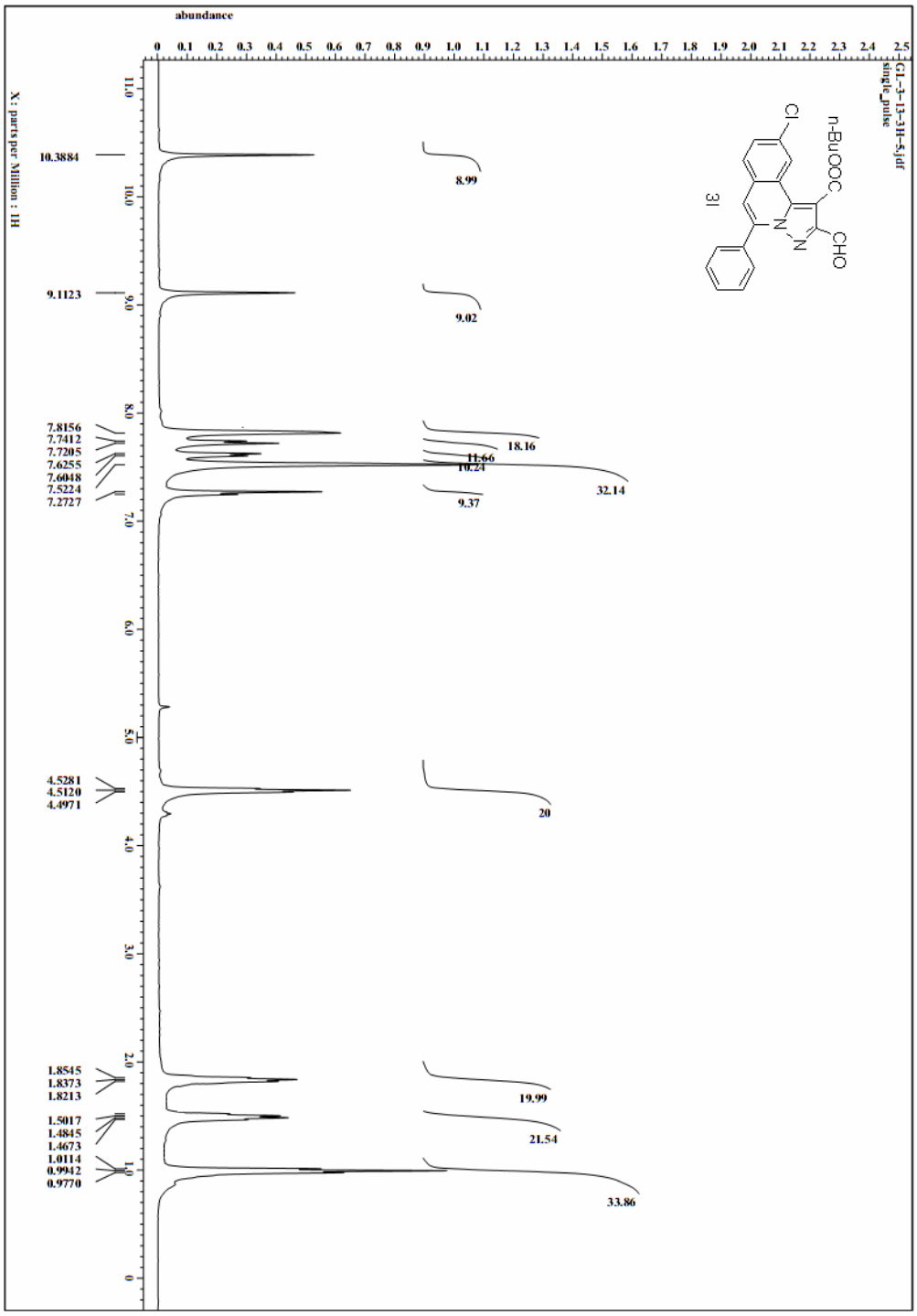

abundance
0
1.0
2.0
GL-3-18-1C-11.jdf
C:\Users\delta\Documents\files\data\guanyin\20111223\GL-3-18-1C
X: parts per Million : 13C
190.8186
163.8031
152.3634
138.2299
133.3534
131.3997
129.9497
129.6750
129.4003
128.3395
128.2403
127.1642
126.3095
116.0451
77.3229
77.0024
76.6895
61.1060
22.1243
13.4091
3i
C2H5OOC

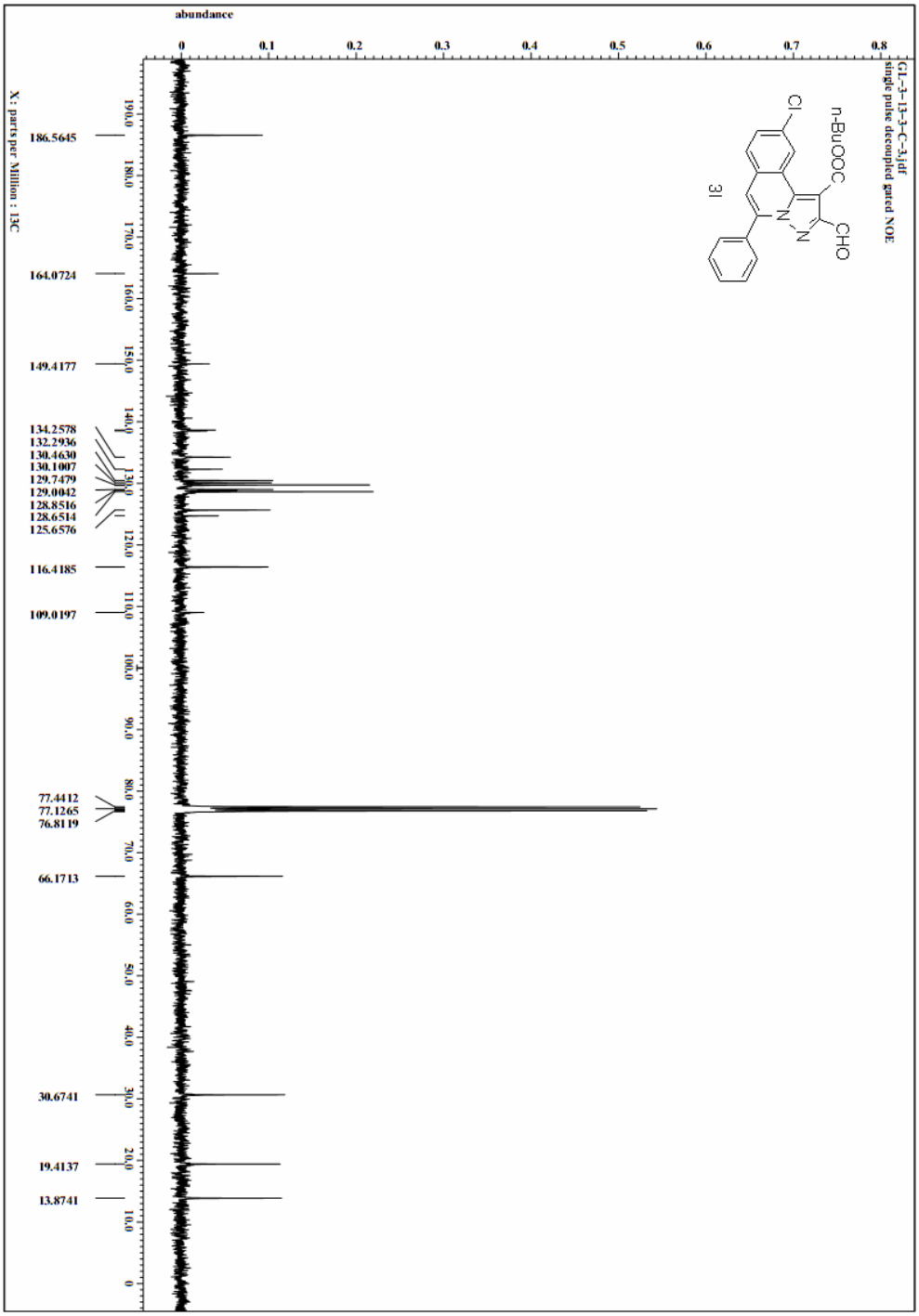

1.00
2.08
2.26
3.30
0.90
2.15
1.16
3.02
2.08
3.23
2.03
9.2641
8.0318
8.0110
7.6032
7.5819
7.5627
7.4770
7.4594
7.4401
6.8381
4.1533
4.1378
4.1199
4.1021
2.7253
2.7153
2.7026
2.5582
1.1656
1.1449
1.1344
0.9927
0.9769
0.9589
0.9159
0.9031
0.8904
C2H5OOC
3j
f1 (ppm)

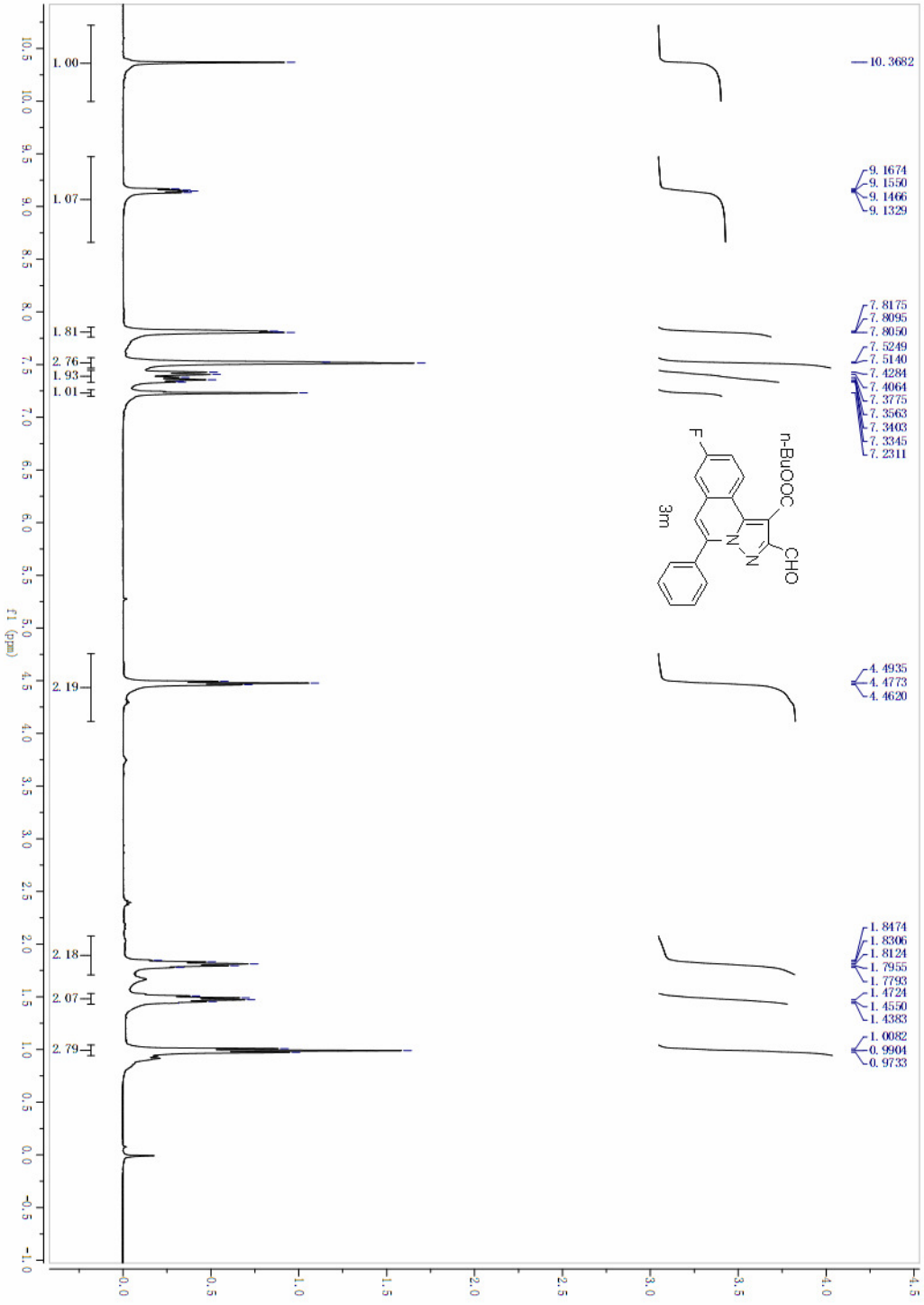

3j
C_2H_5OOC
O
O
N
N
172.9464
151.0813
142.2818
132.1304
129.9014
129.7523
124.0559
122.8173
121.2697
121.1076
118.4417
108.0862
105.8921
81.9729
81.7189
81.4642
68.8058
37.7058
31.6039
30.8115
29.1441
26.1302
f1 (ppm)

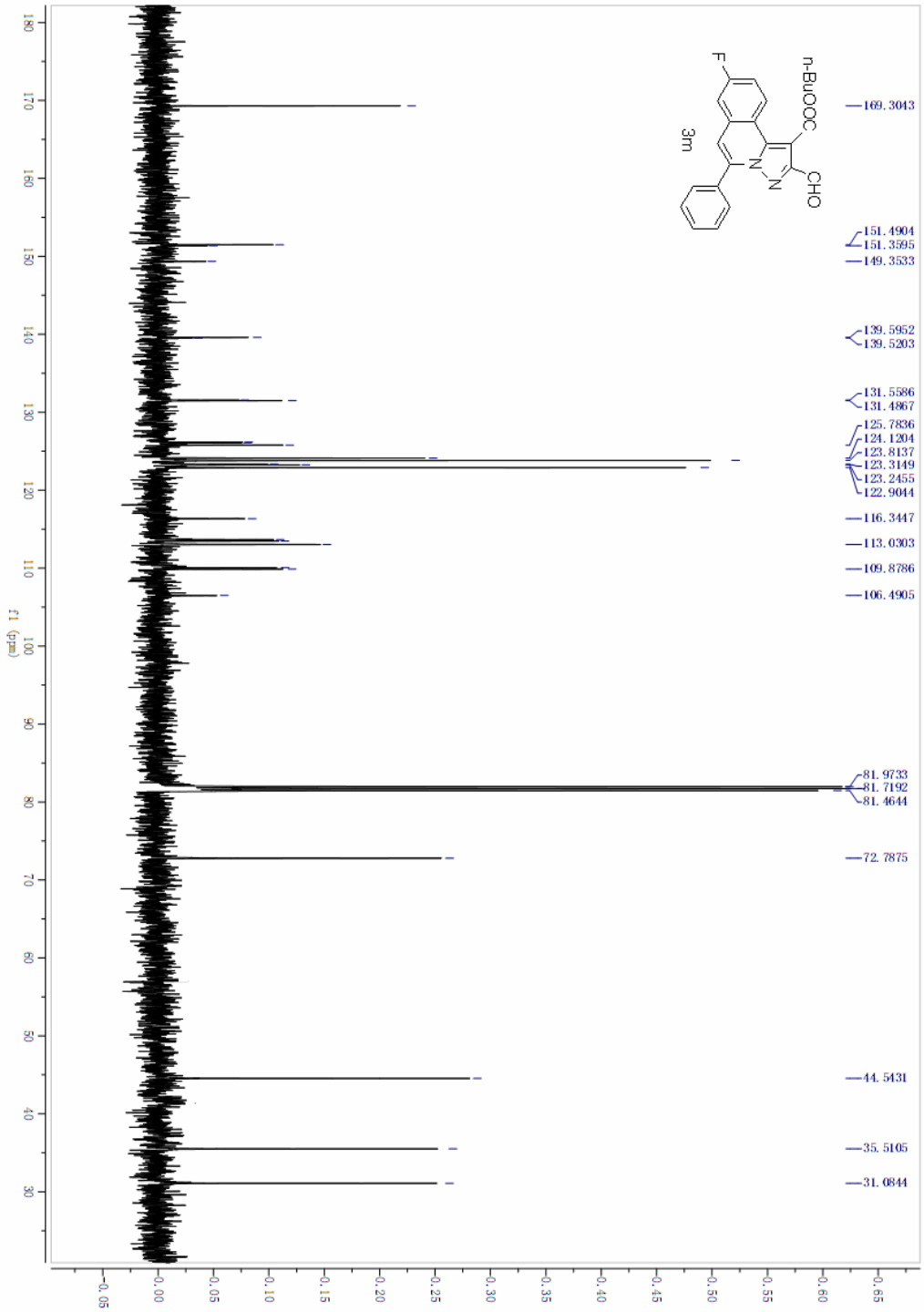

abundance
X : parts per Million : 1H
G1-3-11-4H-5.jdf
single_pulse
10.3827
9.0631
9.0401
7.8213
7.5121
7.2475
7.2314
7.1547
4.4845
4.4685
4.4536
3.9393
1.8247
1.8075
1.7915
1.4879
1.4696
1.4536
1.0011
0.9839
0.9667
9.2
9.71
19.1
30.74
20.28
11.54
19.78
30
18.63
21.55
30.81
3k
n-BuOOC
CHO

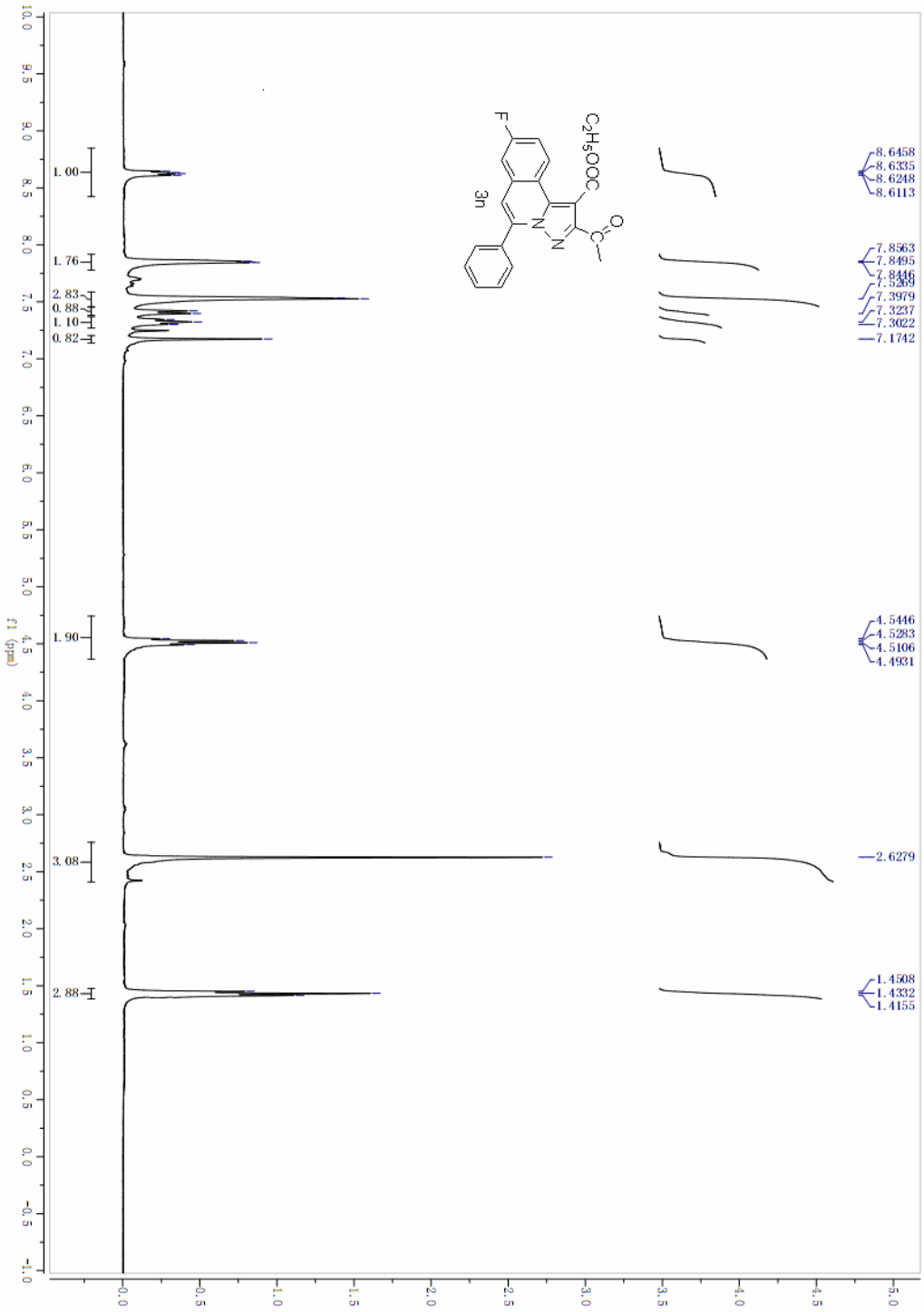

abundance
X : parts per Million : 13C
186.8067
164.4006
160.4322
149.2902
139.8424
138.5069
132.5391
132.4857
129.7536
129.6391
128.4333
127.9678
117.7645
117.6729
116.7724
108.3396
77.3176
76.9970
76.6765
65.6490
55.4838
30.5823
19.2724
13.7319
n-BuOOC
CHO
3k
C:\Users\delta\Documents\files\data\guanyin\20111223\GL-3-18-1C
GL-3-18-1C-5.jdf

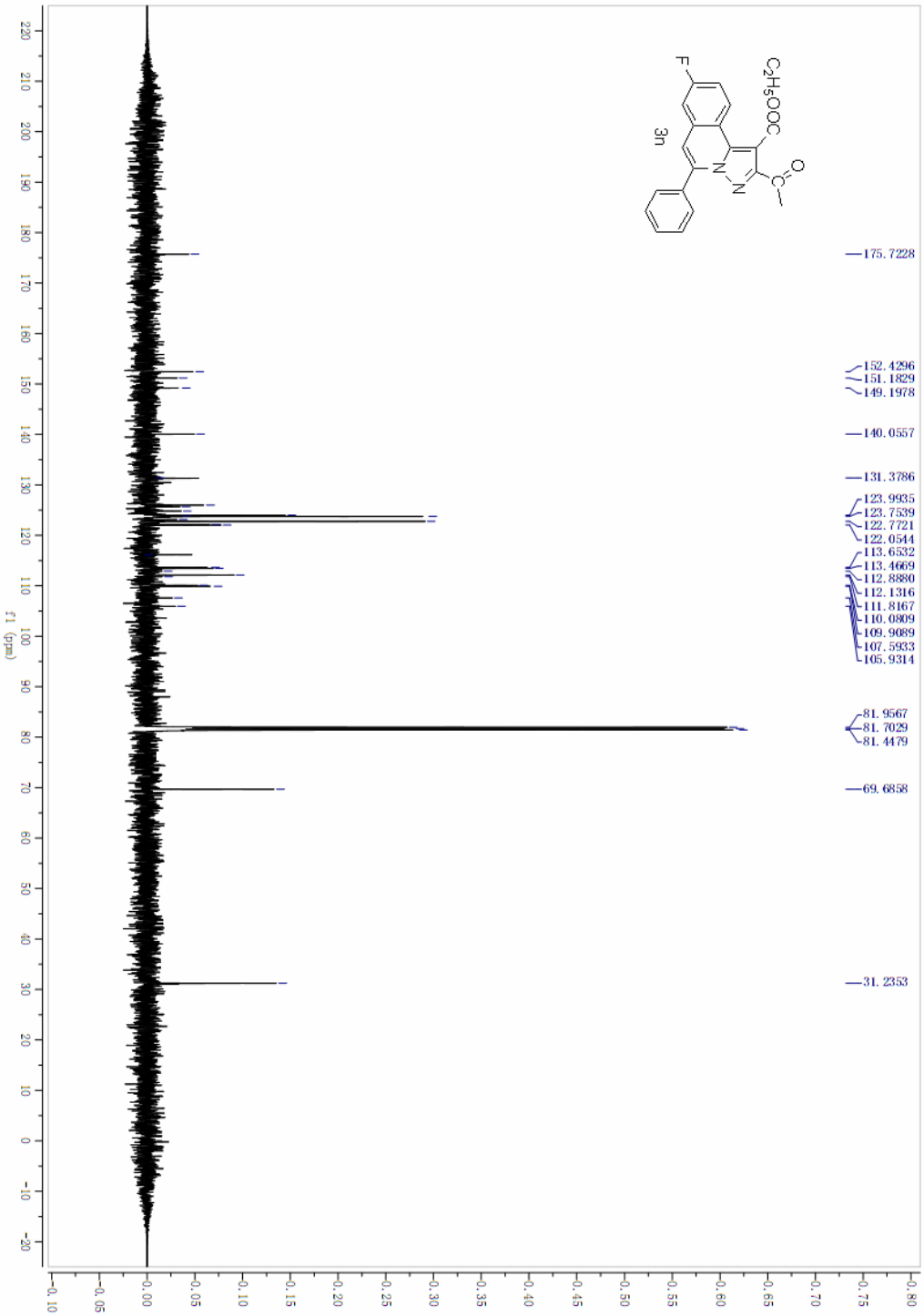

abundance
X : parts per Million : 1H
Cl-3-13-3H-5.jdf
single_pulse
10.3884
9.1123
7.8156
7.7412
7.7205
7.6255
7.6048
7.5224
7.2727
4.5281
4.5120
4.4971
1.8545
1.8373
1.8213
1.5017
1.4845
1.4673
1.0114
0.9942
0.9770
8.99
9.02
18.16
11.66
10.24
32.14
9.37
20
19.99
21.54
33.86
3l
Cl
n-BuOOC
CHO
N
N

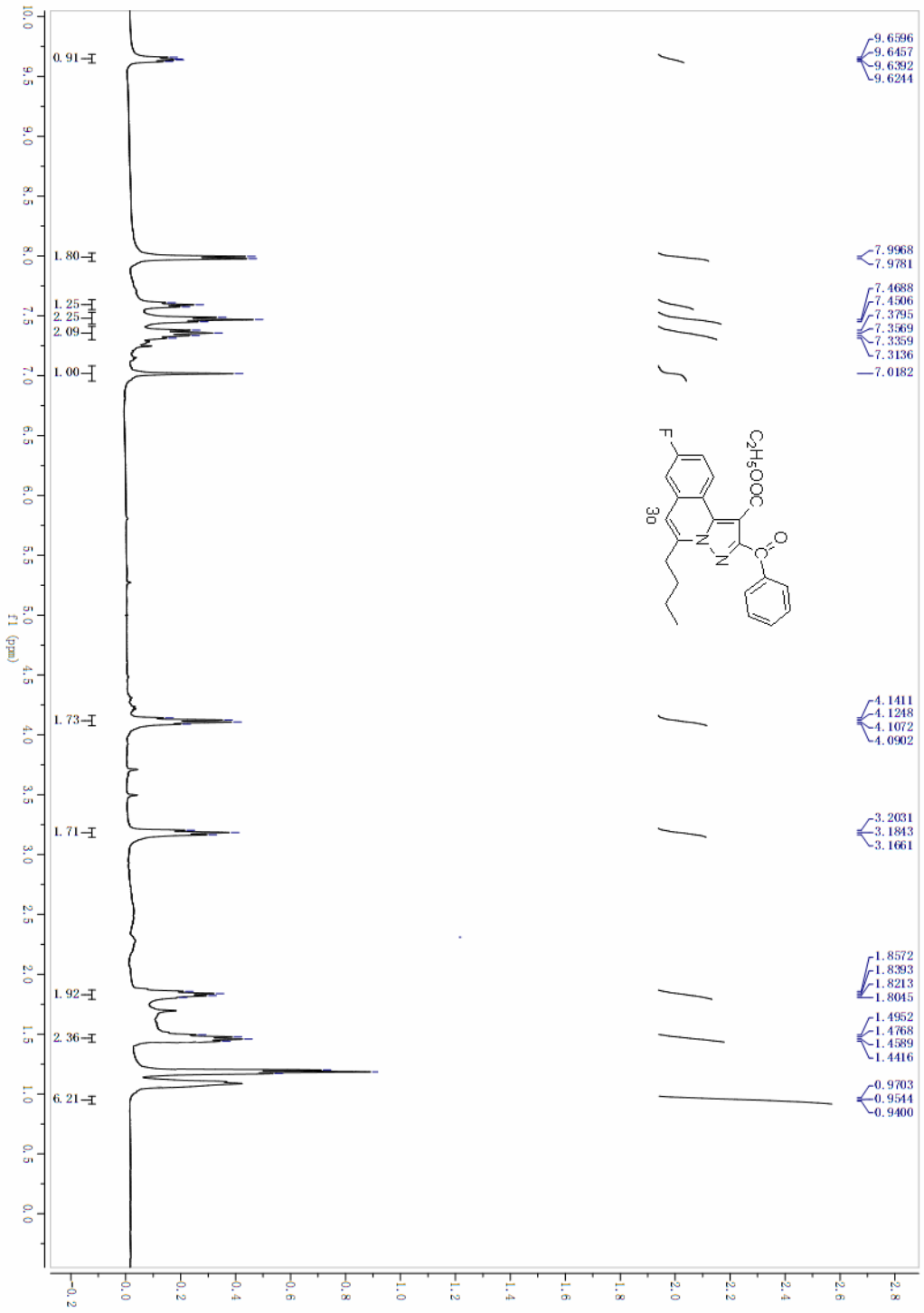

abundance
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
X: parts per Million : 13C
186.5645
164.0724
149.4177
134.2578
132.2936
130.4630
130.1007
129.7479
129.0042
128.8516
128.6514
125.6576
116.4185
109.0197
77.4412
77.1265
76.8119
66.1713
30.6741
19.4137
13.8741
G1-3-13-3-C-3.jdf
single pulse decoupled gated NOE
Cl
n-BuOOC
CHO
N
N
13

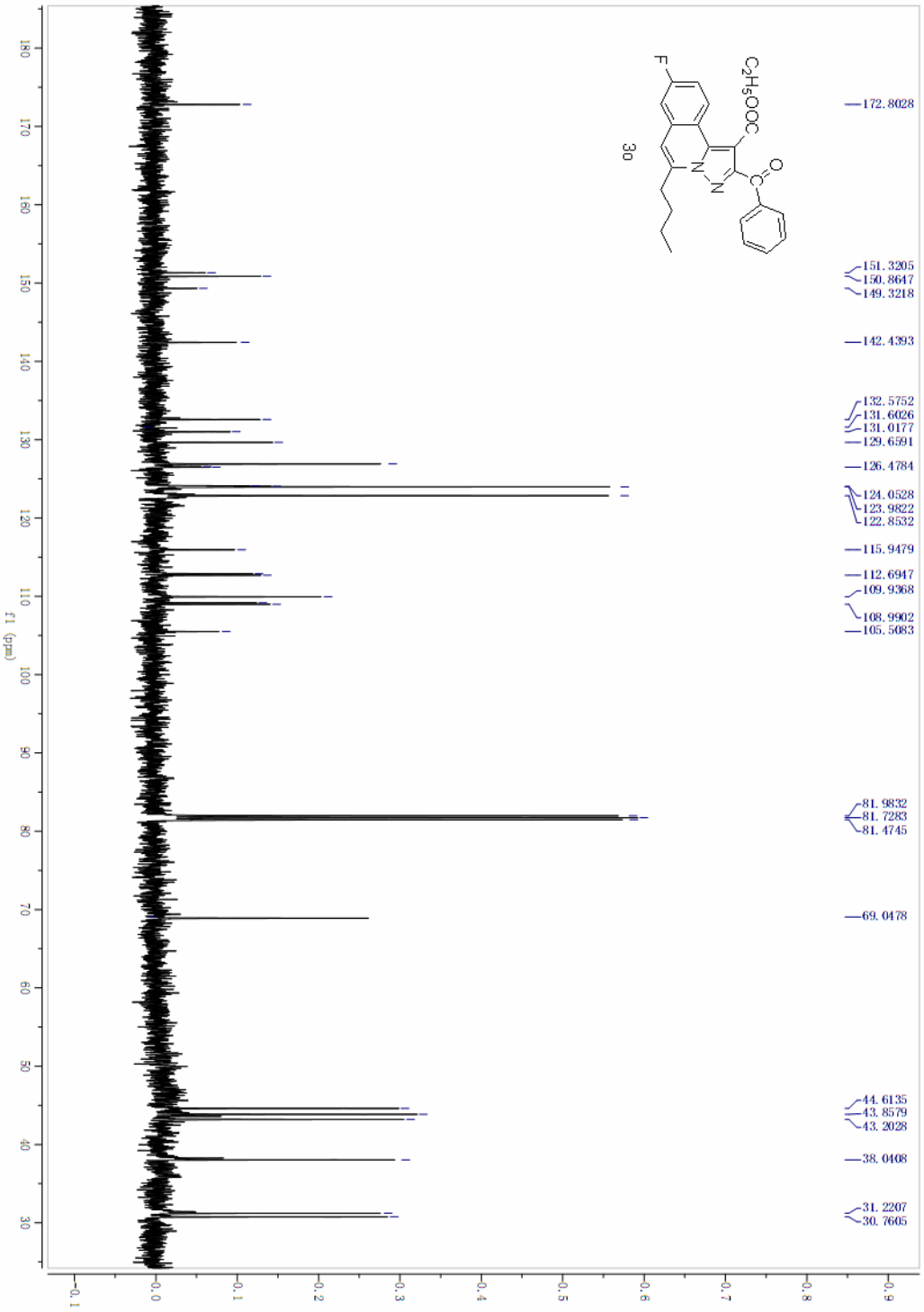

1.00
1.07
1.81
2.76
1.93
1.01
2.19
2.18
2.07
2.79
10.3682
9.1674
9.1550
9.1466
9.1329
7.8175
7.8095
7.8050
7.5249
7.5140
7.4284
7.4064
7.3775
7.3563
7.3403
7.3345
7.2311
4.4935
4.4773
4.4620
1.8474
1.8306
1.8124
1.7955
1.7793
1.4724
1.4550
1.4383
1.0082
0.9904
0.9733
F
n-BuOOC
CHO
N
N
3m
f1 (ppm)

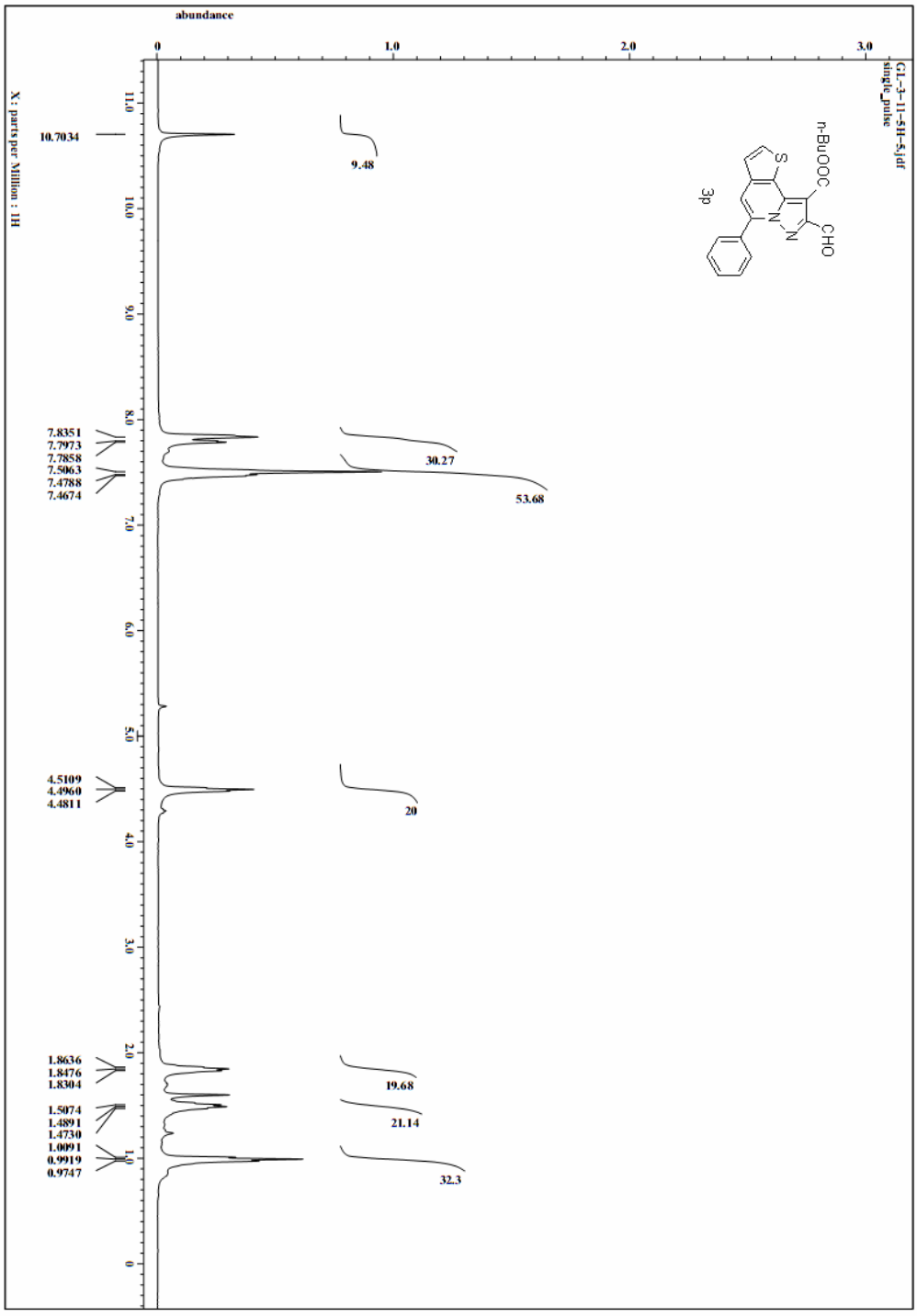

F
n-BuOOC
3m
N
N
CHO
169.3043
151.4904
151.3595
149.3533
139.5952
139.5203
131.5586
131.4867
125.7836
124.1204
123.8137
123.3149
123.2455
122.9044
116.3447
113.0303
109.8786
106.4905
81.9733
81.7192
81.4644
72.7875
44.5431
35.5105
31.0844
f1 (ppm)

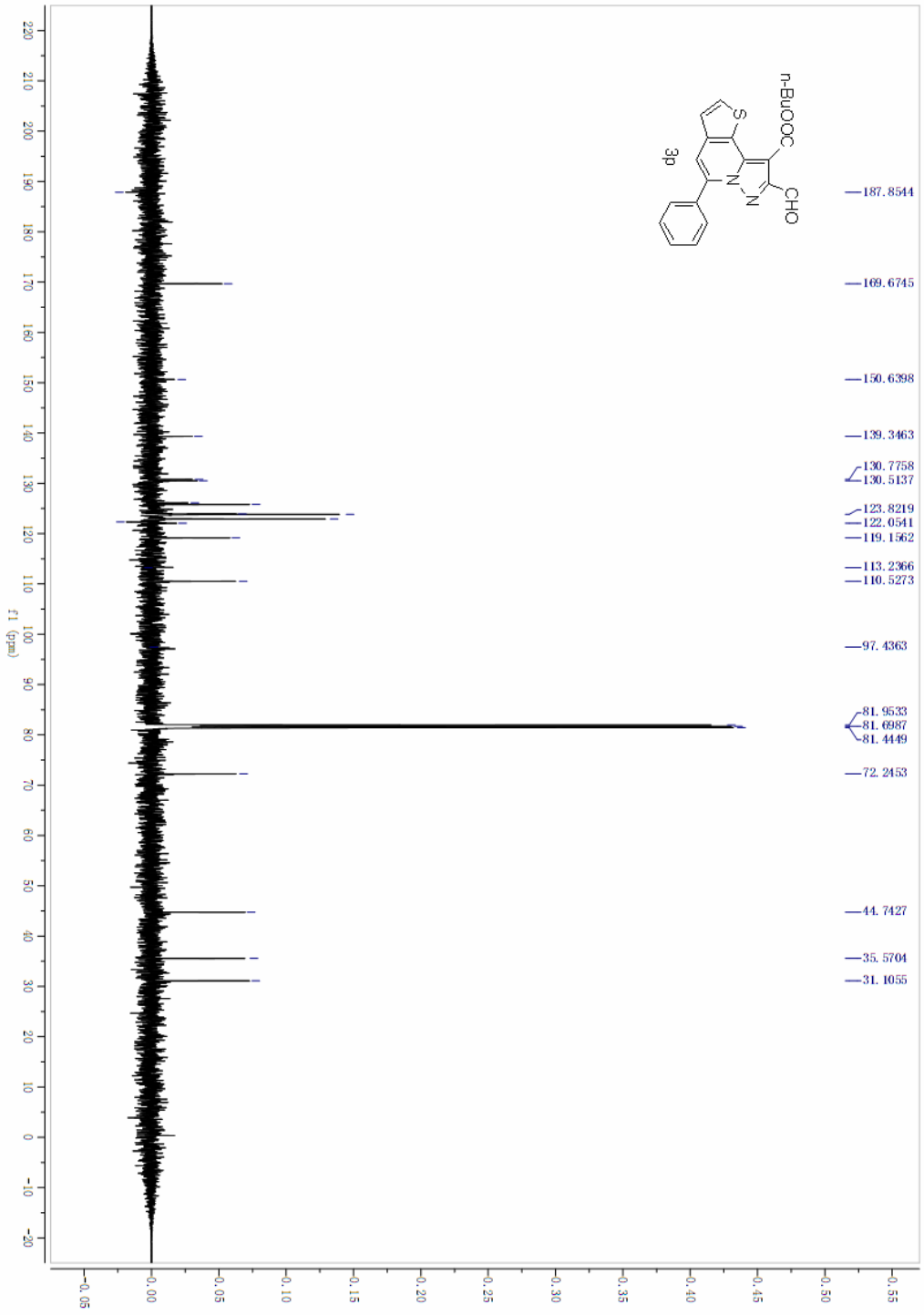

F
C2H5OOC
O
O
N
N
3n
1.00
1.76
2.83
0.88
1.10
0.82
1.90
3.08
2.88
8.6458
8.6335
8.6248
8.6113
7.8563
7.8495
7.8446
7.3269
7.3979
7.3237
7.3022
7.1742
4.5446
4.5283
4.5106
4.4931
2.6279
1.4508
1.4332
1.4155
f1 (ppm)


F
C2H5OOC
3n
N
N
O
O
175.7228
152.4296
151.1829
149.1978
140.0557
131.3786
123.9935
123.7539
122.7721
122.0544
113.6532
113.4669
112.8880
112.1316
111.8167
110.0809
109.9089
107.5933
105.9314
81.9567
81.7029
81.4479
69.6858
31.2353
f1 (ppm)


9.6596
9.6457
9.6392
9.6244
7.9968
7.9781
7.4688
7.4506
7.3795
7.3569
7.3359
7.3136
7.0182
4.1411
4.1248
4.1072
4.0902
3.2031
3.1843
3.1661
1.8572
1.8393
1.8213
1.8045
1.4952
1.4768
1.4589
1.4416
0.9703
0.9544
0.9400
0.91
1.80
1.25
2.25
2.09
1.00
1.73
1.71
1.92
2.36
6.21
F
C2H5OOC
O
N
N
O
3g
f1 (ppm)


3o
F
C2H5OOC
O
N
N
172.8028
151.3205
150.8647
149.3218
142.4393
132.5752
131.6026
131.0177
129.6591
126.4784
124.0528
123.9822
122.8532
115.9479
112.6947
109.9368
108.9902
105.5083
81.9832
81.7283
81.4745
69.0478
44.6135
43.8579
43.2028
38.0408
31.2207
30.7605
f1 (ppm)


abundance
X: parts per Million : 1H
GL-3--11-5H-5.jdf
single_pulse
10.7034
9.48
7.8351
7.7973
7.7858
7.5063
7.4788
7.4674
30.27
53.68
4.5109
4.4960
4.4811
20
1.8636
1.8476
1.8304
19.68
1.5074
1.4891
1.4730
21.14
1.0091
0.9919
0.9747
32.3
n-BuOOC
CHO
S
N
N
3p


3p
n-BuOOC
CHO
S
N
N
187.8544
169.6745
150.6398
139.3463
130.7758
130.5137
123.8219
122.0541
119.1562
113.2366
110.5273
97.4363
81.9533
81.6987
81.4449
72.2453
44.7427
35.5704
31.1055
f1 (ppm)