Supplementary Material (ESI) for Chemical Communications

AgOTf-Catalyzed Tandem Reaction of *N'*-(2-Alkynylbenzylidene)-hydrazide with Alkyne

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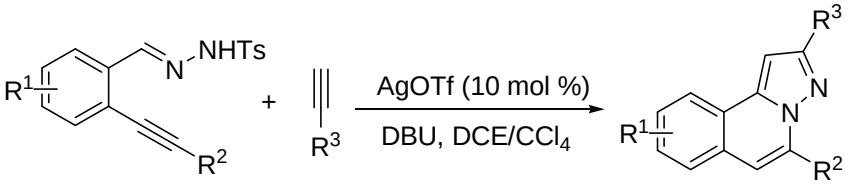
Supporting Information

General experimental methods:

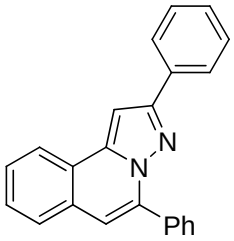
All reactions were performed in reaction tubes under nitrogen atmosphere. Flash column chromatography was performed using silica gel (60-Å pore size, 32–63 μm, standard grade). Analytical thin–layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated 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 (house vacuum) at 25–35°C. Commercial reagents and solvents were used as received. Nuclear magnetic resonance (NMR) spectra are recorded in parts per million from internal tetramethylsilane on the δ scale. N'-(2-Alkynylbenzylidene)hydrazide was synthesized according to the literature method (Anderson, P. N.; Sharp, J. T.; et al, J. Chem. Soc., Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999), **1980**, (6), 1331-1334.)

General procedure for AgOTf-catalyzed tandem reaction of N'-(2-alkynylbenzylidene)hydrazide with alkyne:



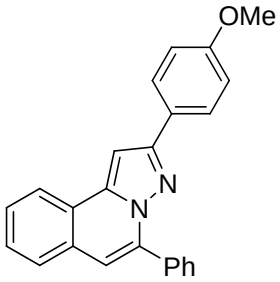
1 **2** **3**

A mixture of *N*'-(2-alkynylbenzylidene)hydrazide **1** (0.20 mmol) and AgOTf (10 mol %) in anhydrous DCE (0.5 mL) was heated at 70 °C for 3 hours. The mixture was then cooled to room temperature. A solution of DBU (0.6 mmol, 3.0 equiv) and alkyne **2** (0.3 mmol, 1.5 equiv) in CCl_4 (2.0 mL) was added subsequently. The mixture was stirred at room temperature overnight. After completion of the reaction as indicated by TLC, the solvent was evaporated. The residue was diluted with EtOAc (10 mL), washed with H_2O (10 mL), dried by anhydrous $MgSO_4$. Evaporation of the solvent followed by purification on silica gel provided the corresponding product **3**.



3a. 2,5-diphenylpyrazolo[5,1-a]isoquinoline.

Yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$): 7.05 (s, 1H), 7.32-7.35 (m, 1H), 7.38 (s, 1H), 7.41-7.44 (t, *J* = 7.3 Hz, 2H), 7.50-7.56 (m, 5H), 7.71-7.73 (m, 1H), 7.98-8.05 (m, *4*H), 8.12-8.15(m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 94.7, 112.5, 123.5,123.9, 126.4, 127.2, 127.3, 127.9, 128.1, 128.6, 129.2, 129.3, 129.6, 133.4, 133.7, 138.4, 140.7, 152.2; HRMS calcd. for $C_{23}H_{16}N_2^+$ $[M+H]^+$: 321.1392, found 321.1383.



3b. 2-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline.

Yield: 74%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.85 (s, 3H), 6.96 (d, *J* = 8.3 Hz, 2H), 7.05 (s, 1H), 7.32 (s, 1H), 7.50-7.58 (m, 5H), 7.72-7.74 (m, 1H), 7.92-7.94 (m, 2H), 8.02-8.05 (m, 2H), 8.13-8.15 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 55.3, 94.2, 112.2, 113.9, 123.5, 123.8, 126.1, 127.1, 127.6, 127.9, 128.1, 129.2, 129.3, 129.6, 133.8, 138.3, 140.7, 152.1, 159.7; HRMS calcd. for $C_{24}H_{18}N_2O^+$ $[M+H]^+$: 351.1497, found 351.1497

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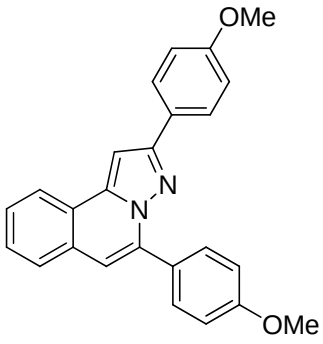
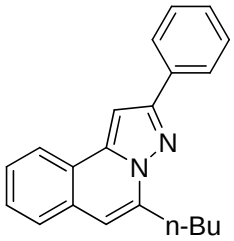


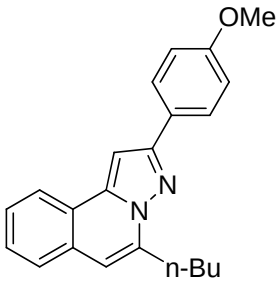
3c. 2,5-bis(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline.

Yield: 70%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.85 (s, 3H), 3.90 (s, 3H), 6.96-7.00 (m, 3H), 7.05-7.08 (m, 2H), 7.30 (s, 1H), 7.49-7.55 (m, 2H), 7.69-7.71 (m, 1H), 7.93-7.96 (m, 2H), 7.99-8.03 (m, 2H), 8.10-8.12 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 55.5, 94.3, 111.5, 113.7, 114.1, 123.6, 123.7, 126.3, 127.0, 127.1, 127.8, 127.9, 129.5, 131.1, 138.2, 140.9, 152.1, 159.8, 160.4; HRMS calcd. for $C_{25}H_{20}N_2O_2^+$ $[M+H]^+$: 381.1603, found 381.1612.



3d. 5-butyl-2-phenylpyrazolo[5,1-a]isoquinoline.

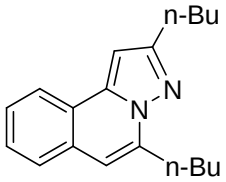
Yield: 99%; ^{1}H NMR (400 MHz, $CDCl_3$): 1.02 (t, *J*= 7.3 Hz, 3H), 1.50-1.53 (m, 2H), 1.89-1.93 (m, 2H), 3.21 (t, *J*= 7.3 Hz, 2H), 6.78 (s, 1H), 7.29 (s, 1H), 7.34-7.38 (m, 1H), 7.44-7.50 (m, 4H), 7.62-7.64 (m, 1H), 8.04- 8.08 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 22.5, 28.9, 30.5, 94.5, 109.3, 123.4, 126.3, 126.4, 126.5, 127.6, 128.1, 128.7, 129.2, 133.6, 139.6, 140.0, 151. 9; HRMS calcd. for $C_{21}H_{20}N_2^+$ $[M+H]^+$: 301.1705, found 301.1689.



3e. 5-butyl-2-(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline.

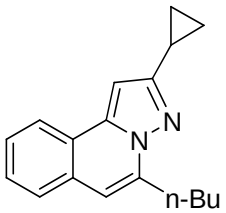
Yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$)): 1.01 (t, *J*= 7.3 Hz, 3H), 1.49-1.55 (m, 2H), 1.87-1.94 (m, 2H), 3.20 (t, *J*= 7.3 Hz, 2H), 3.84 (s, 3H), 6.75 (s, 1H), 6.98 (d, *J*= 8.7 Hz, 2H), 7.20 (s, 1H), 7.44-7.47 (m, 2H), 7.60-7.62 (m, 1H), 7.95-7.99 (d, *J*= 8.7 Hz, 2H), 8.02- 8.06 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.6, 28.9, 30.5, 55.3,

94.0, 108.9, 114.0, 123.2, 123.3, 126.3, 126.4, 127.5, 129.2, 139.6, 140.0, 151.8, 159.6; HRMS calcd. for $C_{22}H_{22}N_2O^+$ $[M+H]^+$: 331.1810, found 331.1811.



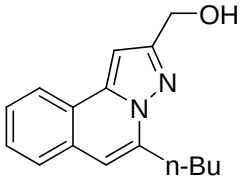
3f. 2,5-dibutylpyrazolo[5,1-a]isoquinoline.

Yield: 75%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.93-1.02 (m, 6H), 1.41-1.59 (m, 4H), 1.72-1.81 (m, 2H), 1.82-1.91 (m, 2H), 2.89 (t, *J* = 7.8 Hz, 2H), 3.15 (t, *J* = 7.8 Hz, 2H), 6.73 (s, 1H), 6.82 (s, 1H), 7.44-7.46 (m, 2H), 7.61-7.63 (m, 1H), 8.00-8.02 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 14.0, 22.5, 22.6, 28.5, 28.8, 30.4, 32.1, 96.1, 108.1, 123.2, 123.3, 126.1, 126.3, 127.4, 129.2, 139.3, 139.4, 154.9; HRMS calcd. for $C_{19}H_{24}N_2^+$ $[M+H]^+$: 281.2018, found 281.2018.



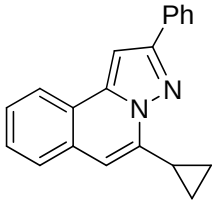
3g. 5-butyl-2-cyclopropylpyrazolo[5,1-a]isoquinoline.

Yield: 62%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.88-0.92 (m, 2H), 0.97-1.02 (t, *J* = 7.4 Hz, 3H), 1.03-1.06 (m, 2H), 1.47-1.58 (m, 2H), 1.84-1.94 (m, 2H), 2.19-2.22 (m, 1H), 3.14 (t, *J* = 7.4 Hz, 2H), 3.86 (s, 3H), 6.62 (s, 1H), 6.72 (s, 1H), 7.44-7.47 (m, 2H), 7.61-7.63 (m, 1H), 7.96-7.99 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 8.91, 9.70, 14.0, 22.5, 28.8, 30.4, 93.1, 108.1, 123.0, 123.3, 126.1, 126.3, 127.4, 129.2, 139.2, 139.5, 156.8; HRMS calcd. for $C_{18}H_{20}N_2^+$ $[M+H]^+$: 265.1705, found 265.1697.



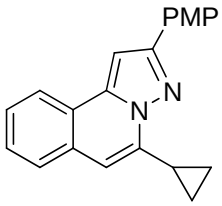
3h. (5-butylpyrazolo[5,1-a]isoquinolin-2-yl)methanol.

Yield: 64%; ^{1}H NMR (400 MHz, $CDCl_3$)): 0.99 (t, *J*= 7.3 Hz, 3H), 1.44-1.54 (m, 2H), 1.81-1.88 (m, 2H), 2.69 (br, 1H), 3.12 (t, *J*= 7.3 Hz, 2H), 4.96 (s, 2H), 6.78 (s, 1H), 6.99 (s, 1H), 7.46-7.52 (m, 2H), 7.63-7.65 (m, 1H), 8.00- 8.03 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.5, 28.8, 30.4, 59.5, 95.8, 109.2, 123.2, 123.4, 126.4, 126.5, 127.8, 129.1, 139.3, 139.7, 153.2; HRMS calcd. for $C_{16}H_{18}N_2O^+$ $[M+H]^+$: 255.1497, found 255.1492.



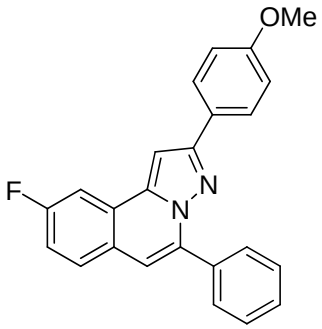
3i. 5-cyclopropyl-2-phenylpyrazolo[5,1-a]isoquinoline.

Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.94-0.96 (m, 2H), 1.18-1.21 (m, 2H), 2.83-2.88 (m, 1H), 6.55 (s, 1H), 7.31 (s, 1H), 7.34-7.37 (m, 1H), 7.44-7.48 (m, 4H), 7.57-7.59 (m, 1H), 8.04-8.07 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.78, 11.3, 94.9, 106.1, 123.3, 123.5, 126.5, 126.6, 126.7, 127.8, 128.3, 128.8, 129.4, 133.8, 140.2, 141.4, 152.3; HRMS calcd. for $C_{20}H_{16}N_2^+$ $[M+H]^+$: 285.1392, found 285.1390.



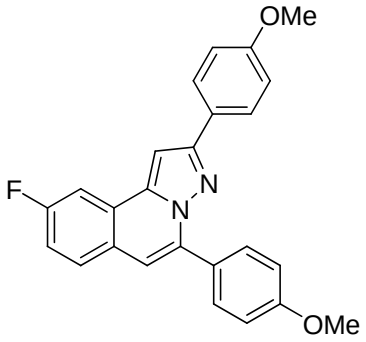
3j. 5-cyclopropyl-2-(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline.

Yield: 78%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.94- 0.97(m, 2H), 1.21- 1.25(m, 2H), 2.85- 2.88 (m, 1H), 3.86 (s, 3H), 6.56 (s, 1H), 6.99-7.01 (d, *J*= 8.68 Hz, 1H), 7.26 (s, 1H), 7.46-7.48 (m, 2H), 7.57-7.61 (m, 1H), 7.98-8.00 (d, *J*= 8.72 Hz, 2H), 8.06-8.08 (d, *J*= 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.63, 11.1, 55.3, 94.2, 105.5, 114.1, 123.0, 123.4, 126.3, 126.4, 127.6, 127.7, 129.3, 140.1, 141.3, 152.1, 159.7; HRMS calcd. for $C_{21}H_{18}N_2O^+$ $[M+H]^+$: 315.1497, found 315.1491.



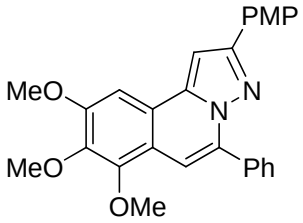
3k. 9-fluoro-2-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline.

Yield: 62%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.83 (s, 3H), 6.94-6.98 (m, 3H), 7.23-7.26 (m, 2H), 7.47-7.54 (m, 3H), 7.65-7.74 (m, 2H), 7.89 (d, J=8.68Hz, 2H), 7.98-8.01 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 94.7, 108.7(d, $^{2}J_{CF}$ = 22.9 Hz), 111.4, 114.0, 116.6(d, $^{2}J_{CF}$ = 23.8 Hz), 119.1, 125.0, 125.9, 127.6, 128.1, 129.2, 129.3, 129.6, 133.6, 137.7, 139.9, 152.1, 159.8, 161.5 (d, $^{1}J_{CF}$ = 246.0 Hz) ; HRMS calcd. for $C_{24}H_{17}FN_2O^{+}$ $[M+H]^{+}$: 369.1403, found 369.1406.



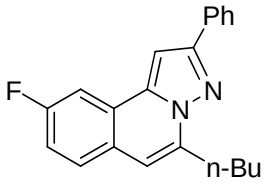
3l. 9-fluoro-2,5-bis(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline.

Yield: 67%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.85 (s, 3H), 3.90 (s, 3H), 6.94-6.97 (m, 3H), 7.04-7.06 (d, J = 8.3 Hz, 2H), 7.23-7.25 (m, 2H), 7.65-7.73 (m, 2H), 7.90-7.97 (m,4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4，55.5，94.8，108.8 (d, $^{2}J_{CF}$ = 22.9 Hz), 110.8，113.7，114.1，116.7(d, $^{2}J_{CF}$ = 23.8 Hz),，124.9，126.0，126.1，127.7，129.3，131.1，137.5，140.1, 152.1, 159.9, 160.4, 161.5 (d, $^{1}J_{CF}$ = 246.0 Hz) ; HRMS calcd. for $C_{25}H_{19}FN_2O_2^{+}$ $[M+H]^{+}$: 399.1509, found 399.1522.



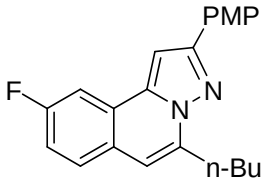
3m. 7,8,9-trimethoxy-2-(4-methoxyphenyl)-5-phenylpyrazolo[5,1-a]isoquinoline

Yield: 83%; ^{1}H NMR (400 MHz, $CDCl_3$): 3.84 (s, 3H), 3.99 (s, 3H), 4.04 (s, 3H), 4.05 (s, 3H), 6.94 (s, 1H), 6.97 (s, 1H), 7.20 (s, 1H), 7.27-7.31 (m, 2H), 7.48-7.56 (m, 3H), 7.91-7.94 (d, *J*= 8.8 Hz, 2H), 8.04-8.06 (d, *J*= 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 56.3, 61.3, 61.7, 93.7, 100.2, 106.5, 114.1, 118.9, 120.6, 126.3, 127.7, 128.2, 129.0, 129.8, 134.3, 136.6, 140.3, 142.1, 149.0, 152.0, 153.9, 159.8; HRMS calcd. for $C_{27}H_{24}N_2O_4^+$ $[M+H]^+$: 441.1814, found 441.1809.



3n. 5-butyl-9-fluoro-2-phenylpyrazolo[5,1-a]isoquinoline.

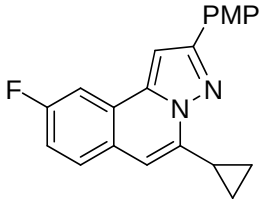
Yield: 88%; ^{1}H NMR (400 MHz, $CDCl_3$): 1.02 (t, *J* = 7.3 Hz, 3H), 1.49-1.59 (m, 2H), 1.85-1.93 (m, 2H), 3.18 (t, *J* = 7.3 Hz, 2H), 6.74 (s, 1H), 7.20-7.23 (m, 2H), 7.35-7.38 (m, 1H), 7.45-7.48 (m, 2H), 7.57-7.61 (m, 1H), 7.68 (dd, *J1* = 0.7 Hz, *J2* = 2.4 Hz, 1H), 8.02 (s, 1H), 8.03 (d, *J* = 1.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 22.5, 28.9, 30.4, 95.0, 108.5(d, $^{2}J_{CF}$ = 21.9 Hz), 108.6, 116.3(d, $^{2}J_{CF}$ = 22.9 Hz), 124.5, 125.7, 126.3, 128.2, 128.6, 128.7, 133.4, 138.9, 139.3, 152.0, 161.1 (d, $^{1}J_{CF}$ = 245.0 Hz) ; HRMS calcd. for $C_{21}H_{19}FN_2^+$ $[M+H]^+$: 319.1611, found 319.1611.



3o. 5-butyl-9-fluoro-2-(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline.

Yield: 64%; ^{1}H NMR (400 MHz, $CDCl_3$): 1.02 (t, *J* = 7.3 Hz, 3H), 1.48-1.58 (m, 2H),

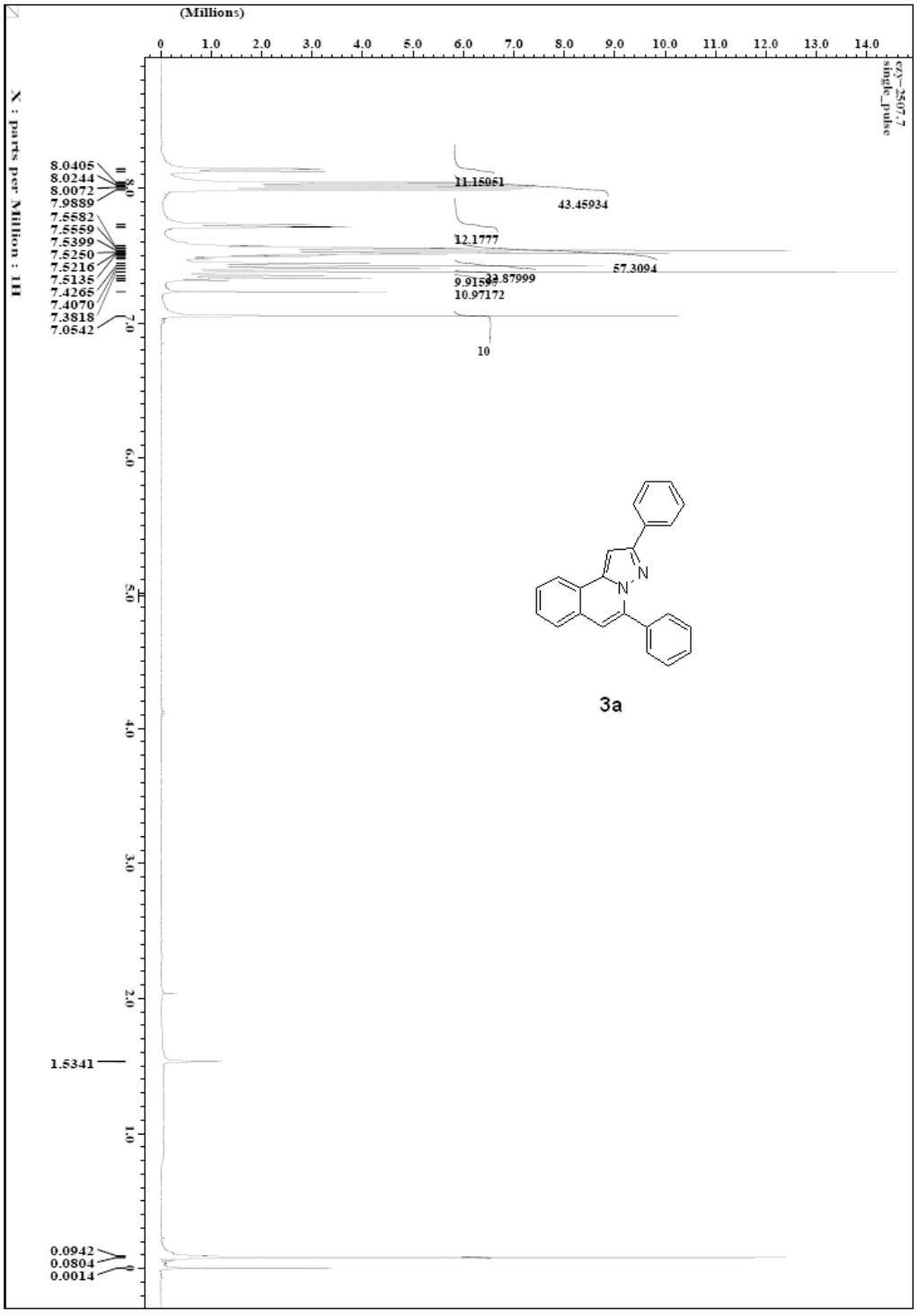
1.84-1.94 (m, 2H), 3.19 (t, *J* = 7.3 Hz, 2H), 3.86 (s, 3H), 6.74 (s, 1H), 6.97-7.01 (m, 2H), 7.16 (s, 1H), 7.21-7.25 (m, 1H), 7.59-7.62 (m, 1H), 7.68 (dd, *J1* = 0.69 Hz, *J2* = 2.4 Hz, 1H), 7.94-7.97 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.7, 29.0, 30.6, 55.4, 94.6, 108.4, 108.6(d, $^{2}J_{CF}$ = 22.9 Hz), 116.3(d, $^{2}J_{CF}$ = 23.8 Hz), 124.5, 125.7, 126.3, 127.7, 128.6, 139.0, 139.4, 152.0, 159.8, 161.1 (d, $^{1}J_{CF}$ = 245.0 Hz) ; HRMS calcd. for $C_{22}H_{21}FN_2O^+$ $[M+H]^+$: 349.1716, found 349.1721.



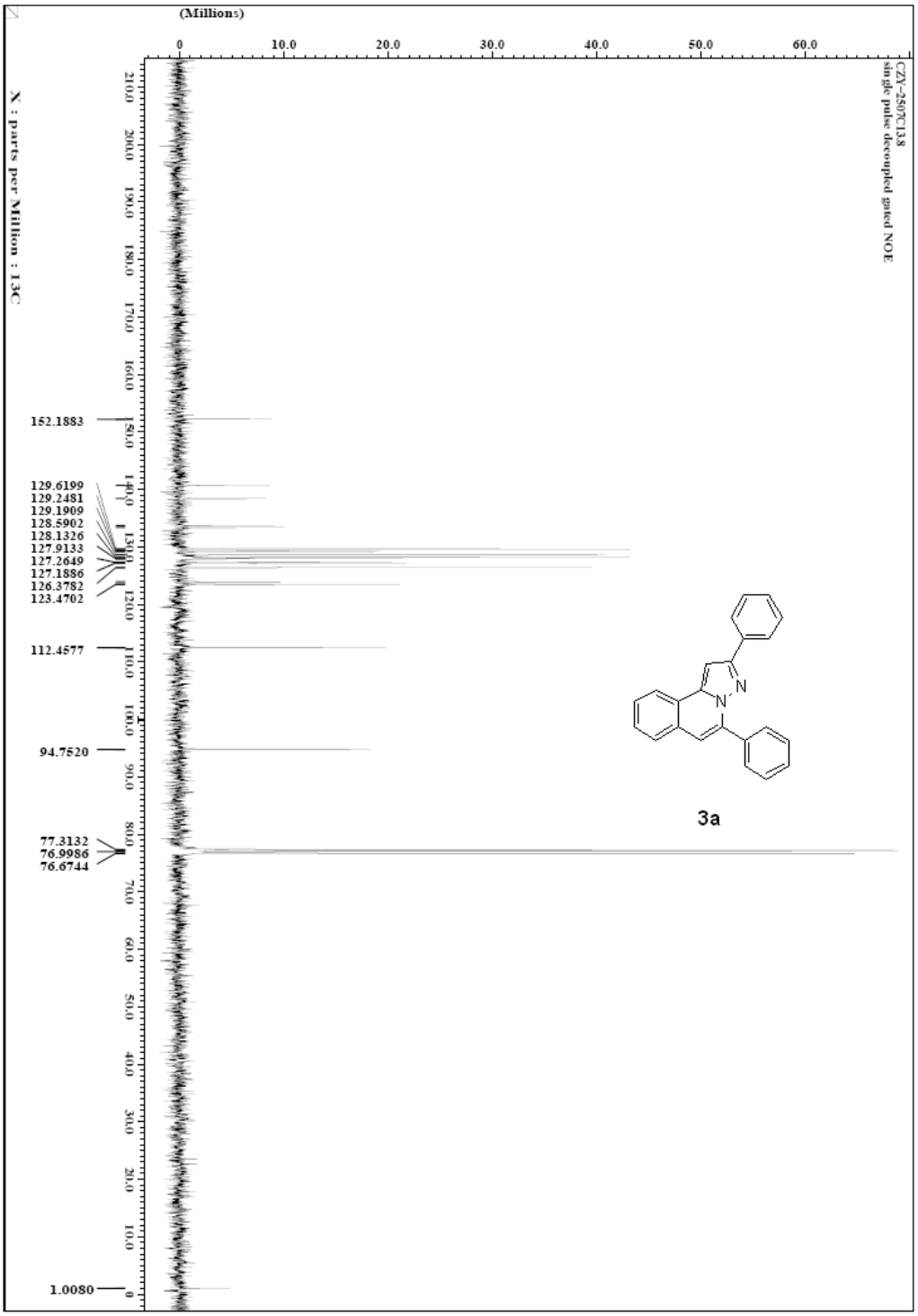
3p. 5-cyclopropyl-9-fluoro-2-(4-methoxyphenyl)pyrazolo[5,1-a]isoquinoline.

Yield: 62%; ^{1}H NMR (400 MHz, $CDCl_3$): 0.93-0.95 (m, 2H), 1.18-1.22 (m, 2H), 2.80-2.84 (m, 1H), 3.86 (s, 3H), 6.53 (s, 1H), 6.97-7.01 (m, 2H), 7.19-7.22 (m, 2H), 7.54-7.58 (m, 1H), 7.68 (dd, *J1* = 0.69 Hz, *J2* = 2.4 Hz, 1H), 7.96-7.98 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 7.52, 11.0, 55.3, 94.7, 105.0, 108.5(d, $^{2}J_{CF}$ = 21.9 Hz), 114.1, 116.3(d, $^{2}J_{CF}$ = 22.9 Hz), 124.1, 125.8, 126.2, 127.6, 128.6, 139.3, 140.5, 152.1, 161.0 (d, $^{1}J_{CF}$ = 242.2 Hz) ; HRMS calcd. for $C_{21}H_{17}FN_2O^+$ $[M+H]^+$: 333.1403, found 333.1402.

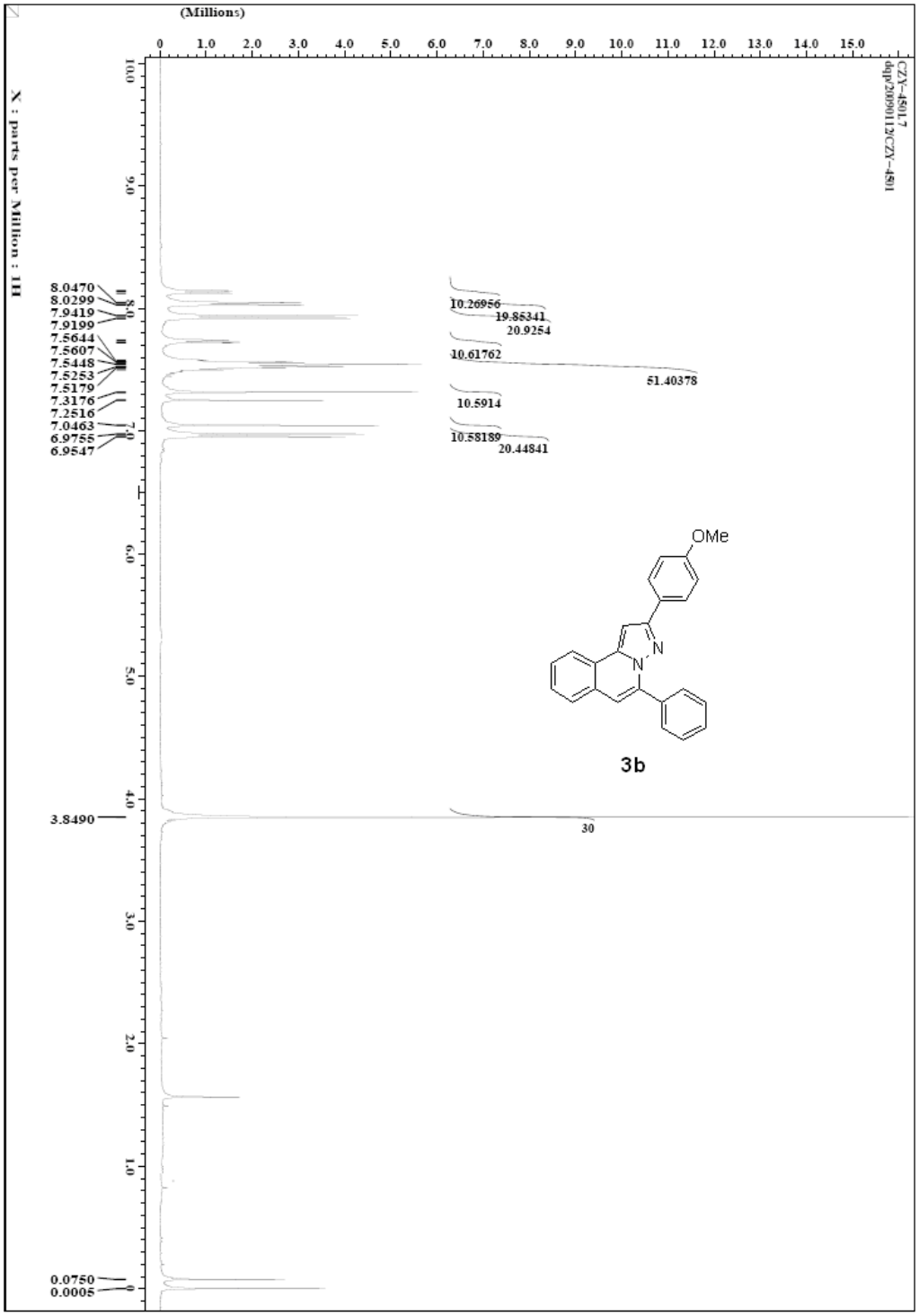
3a


(Millions)
X : parts per Million : 1H
8.0405
8.0244
8.0072
7.9889
7.5582
7.5559
7.5399
7.5250
7.5216
7.5135
7.4265
7.4070
7.3818
7.0542
11.15051
43.45934
12.1777
57.3094
33.87999
9.91599
10.97172
10
1.5341
0.0942
0.0804
0.0014
3a

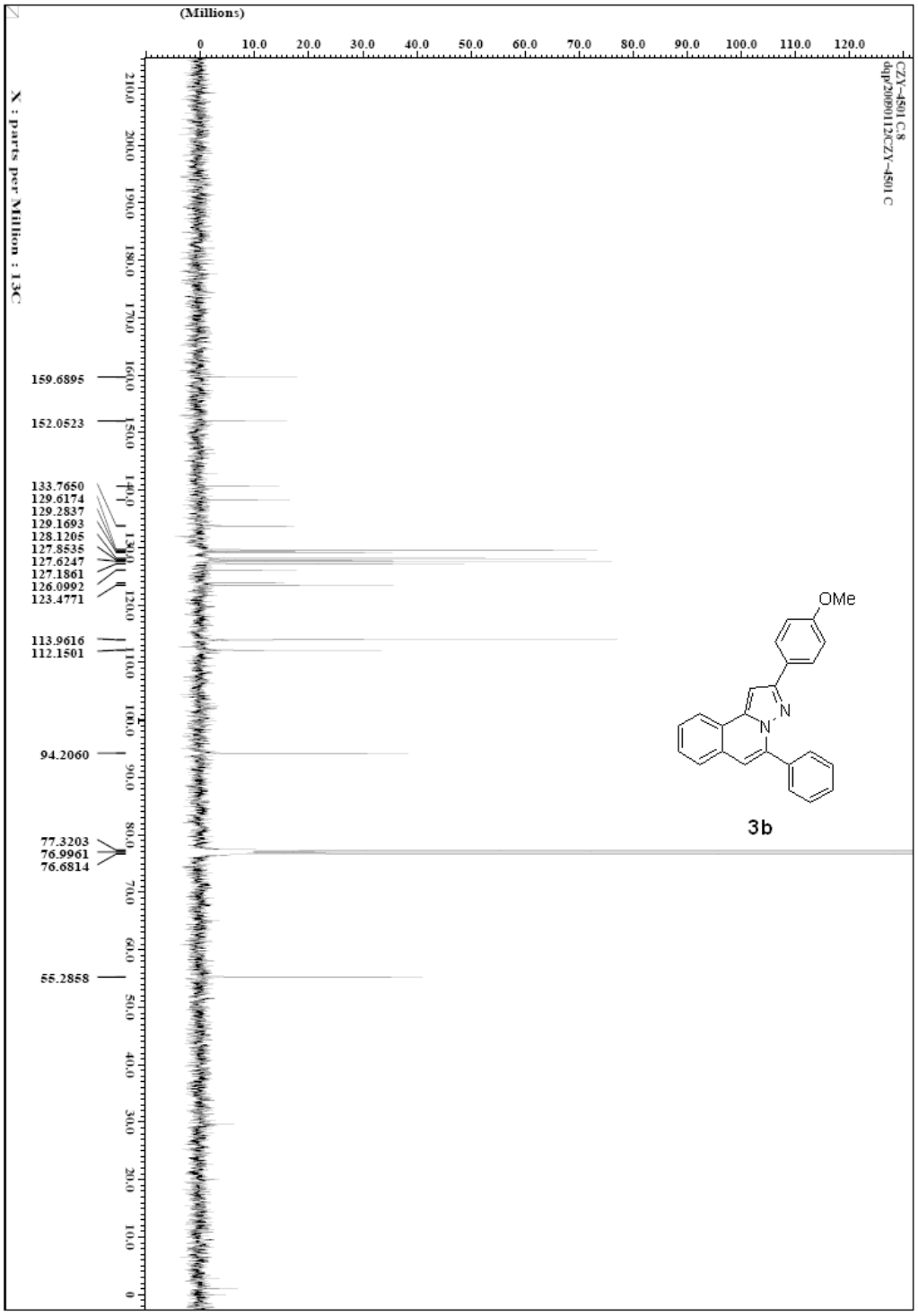
3a


(Millions)
X : parts per Million : 13C
CZY-250TC13.8
single pulse decoupled gated NOE
152.1883
129.6199
129.2481
129.1909
128.5902
128.1326
127.9133
127.2649
127.1886
126.3782
123.4702
112.4577
94.7520
77.3132
76.9986
76.6744
1.0080
3a

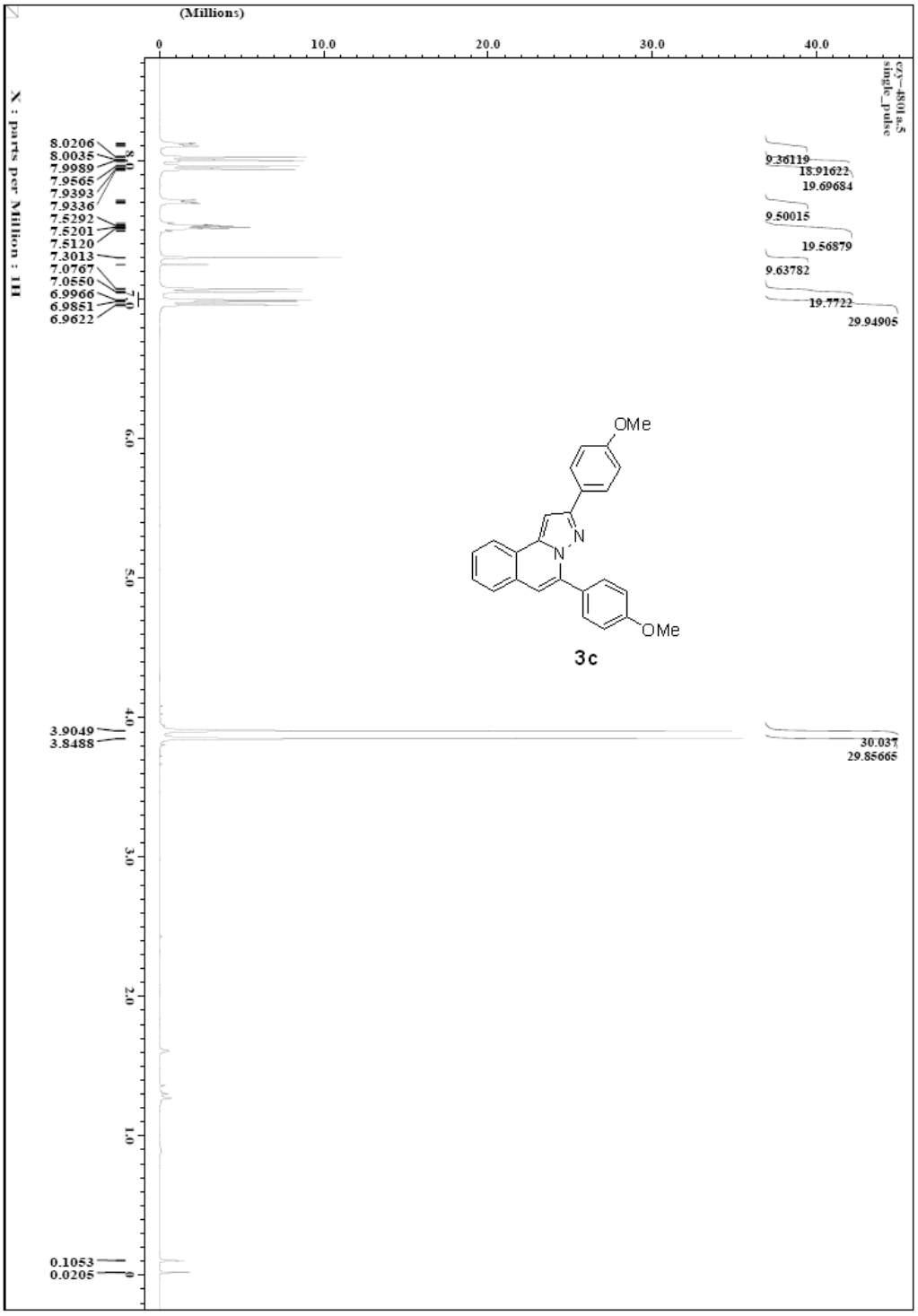
3b


(Millions)
X : parts per Million : 1H
8.0470
8.0299
7.9419
7.9199
7.5644
7.5607
7.5448
7.5253
7.5179
7.3176
7.2516
7.0463
6.9755
6.9547
3.8490
0.0750
0.0005
10.26956
19.85341
20.9254
10.61762
51.40378
10.5914
10.58189
20.44841
30
OMe
N
N
3b

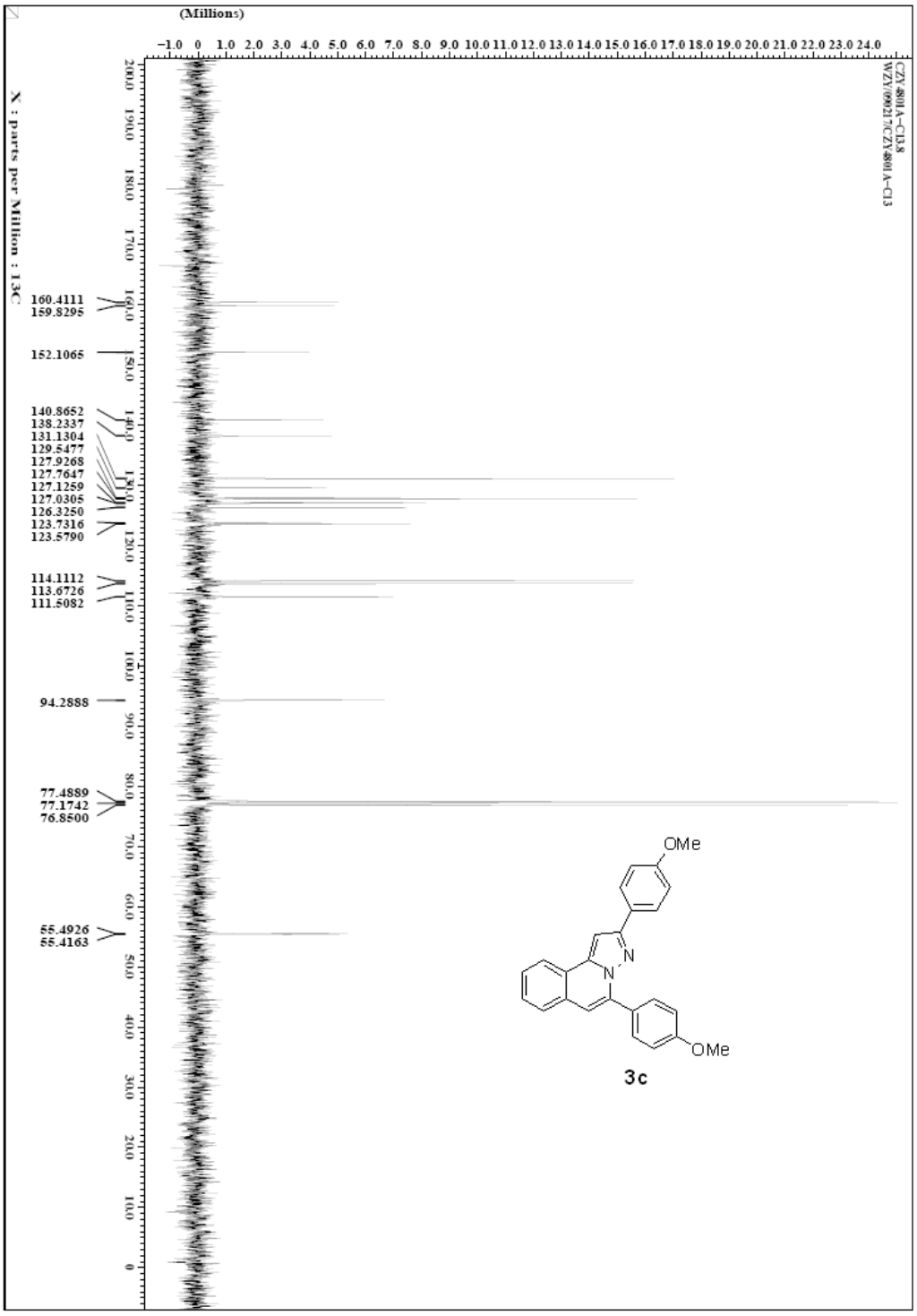
3b


(Millions)
X : parts per Million : 13C
159.6895
152.0523
133.7650
129.6174
129.2837
129.1693
128.1205
127.8535
127.6247
127.1861
126.0992
123.4771
113.9616
112.1501
94.2060
77.3203
76.9961
76.6814
55.2858
OMe
N
N
3b

3c

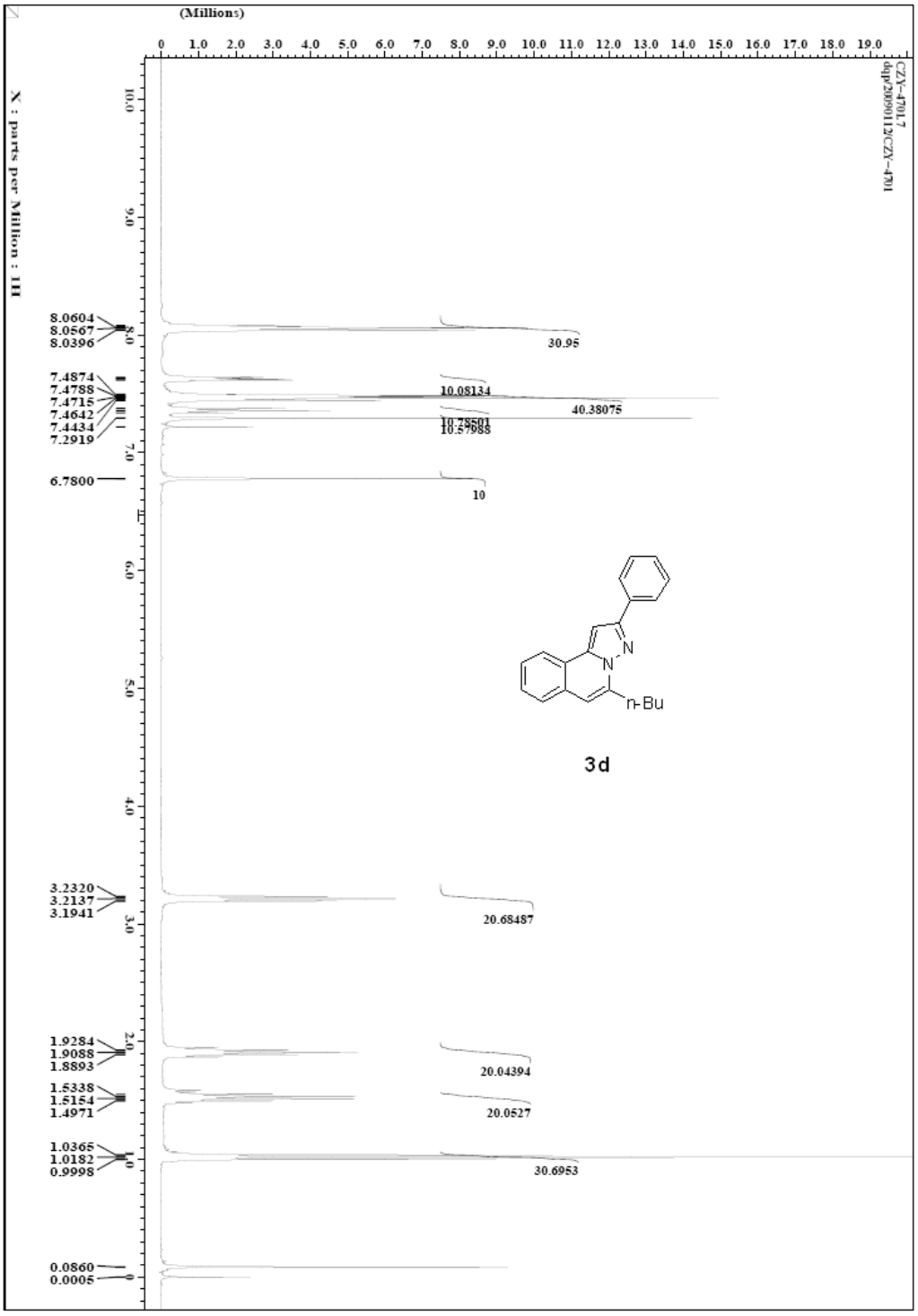

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 1H
8.0206
8.0035
7.9989
7.9565
7.9393
7.9336
7.5292
7.5201
7.5120
7.3013
7.0767
7.0550
6.9966
6.9851
6.9622
3.9049
3.8488
0.1053
0.0205
9.36119
18.91622
19.69684
9.50015
19.56879
9.63782
19.7722
29.94905
30.037
29.85665
OMe
N
N
OMe
3c

3c

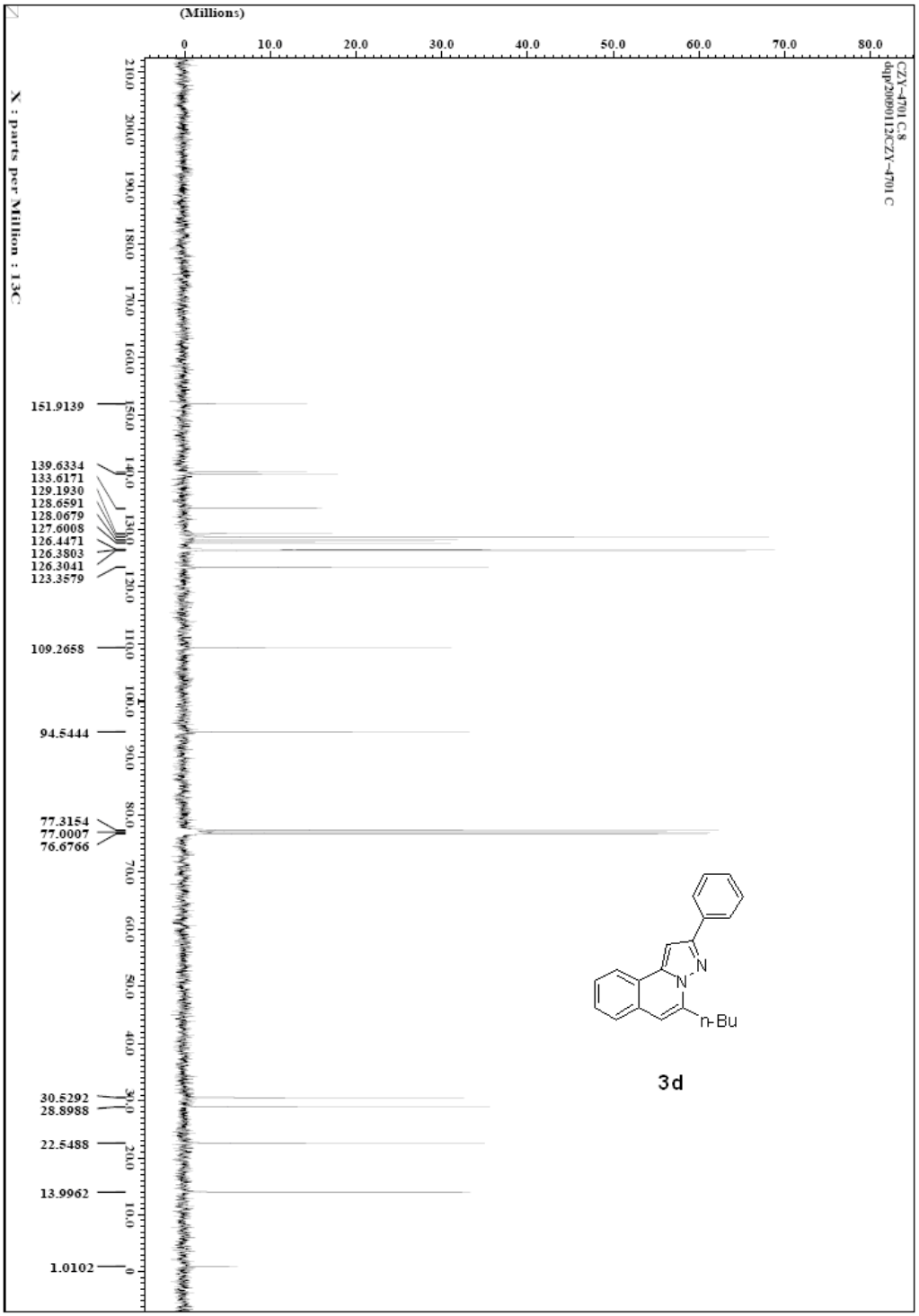

(Millions)
X : parts per Million : 13C
160.4111
159.8295
152.1065
140.8652
138.2337
131.1304
129.5477
127.9268
127.7647
127.1259
127.0305
126.3250
123.7316
123.5790
114.1112
113.6726
111.5082
94.2888
77.4889
77.1742
76.8500
55.4926
55.4163
OMe
N
N
OMe

3c

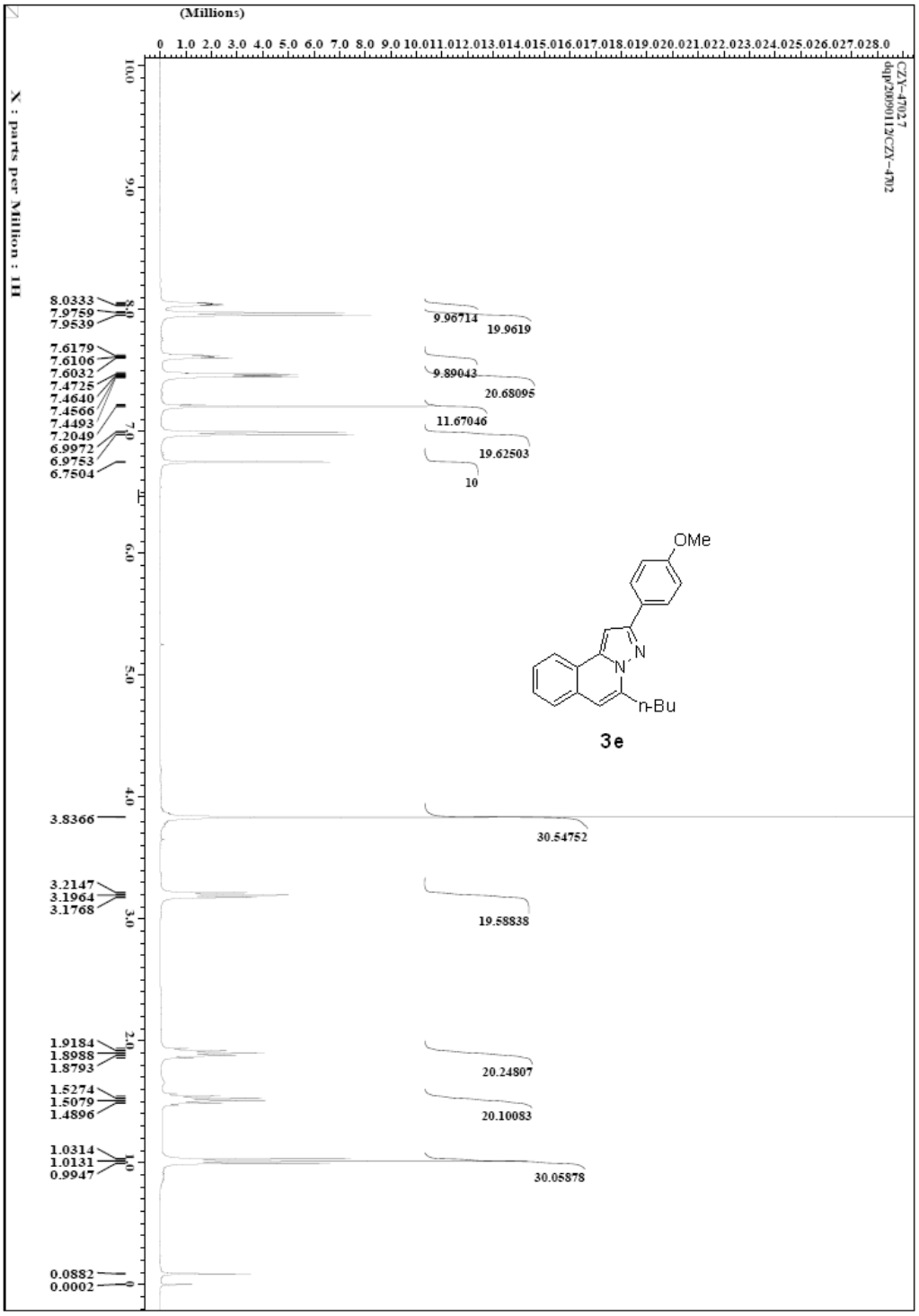
3d


(Millions)
X : parts per Million : 1H
8.0604
8.0567
8.0396
7.4874
7.4788
7.4715
7.4642
7.4434
7.2919
6.7800
3.2320
3.2137
3.1941
1.9284
1.9088
1.8893
1.5338
1.5154
1.4971
1.0365
1.0182
0.9998
0.0860
0.0005
30.95
10.08134
40.38075
10.57988
10
20.68487
20.04394
20.0527
30.6953
N
N
n-Bu
3d

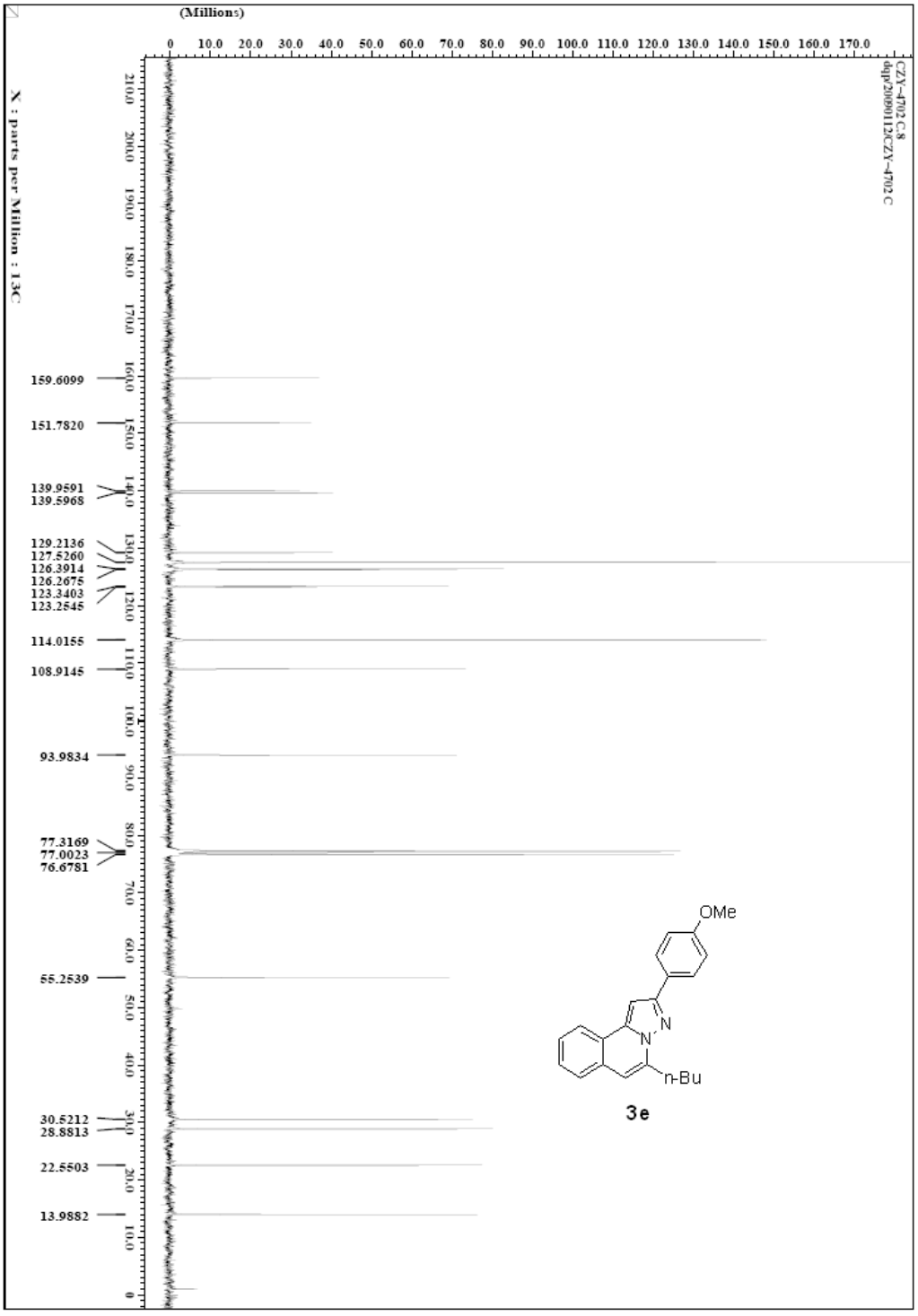
3d


(Millions)
X : parts per Million : 13C
151.9139
139.6334
133.6171
129.1930
128.6591
128.0679
127.6008
126.4471
126.3803
126.3041
123.3579
109.2658
94.5444
77.3154
77.0007
76.6766
30.5292
28.8988
22.5488
13.9962
1.0102
n-Bu
3d

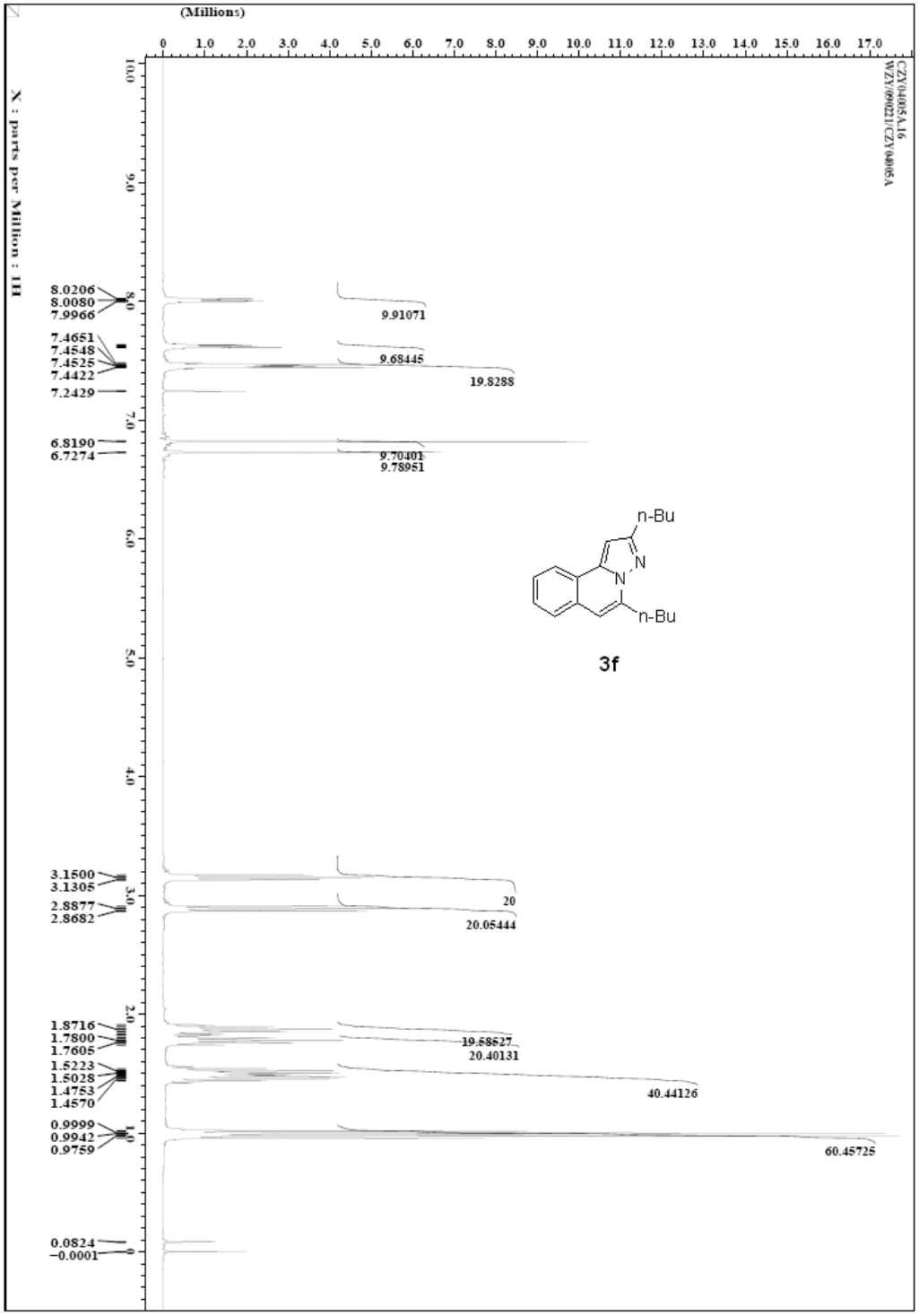
3e


(Millions)
X : parts per Million : 1H
8.0333
7.9759
7.9539
7.6179
7.6106
7.6032
7.4725
7.4640
7.4566
7.4493
7.2049
6.9972
6.9753
6.7504
3.8366
3.2147
3.1964
3.1768
1.9184
1.8988
1.8793
1.5274
1.5079
1.4896
1.0314
1.0131
0.9947
0.0882
0.0002
9.96714
19.9619
9.89043
20.68095
11.67046
19.62503
10
30.54752
19.58838
20.24807
20.10083
30.05878
OMe
N
N
n-Bu
3e

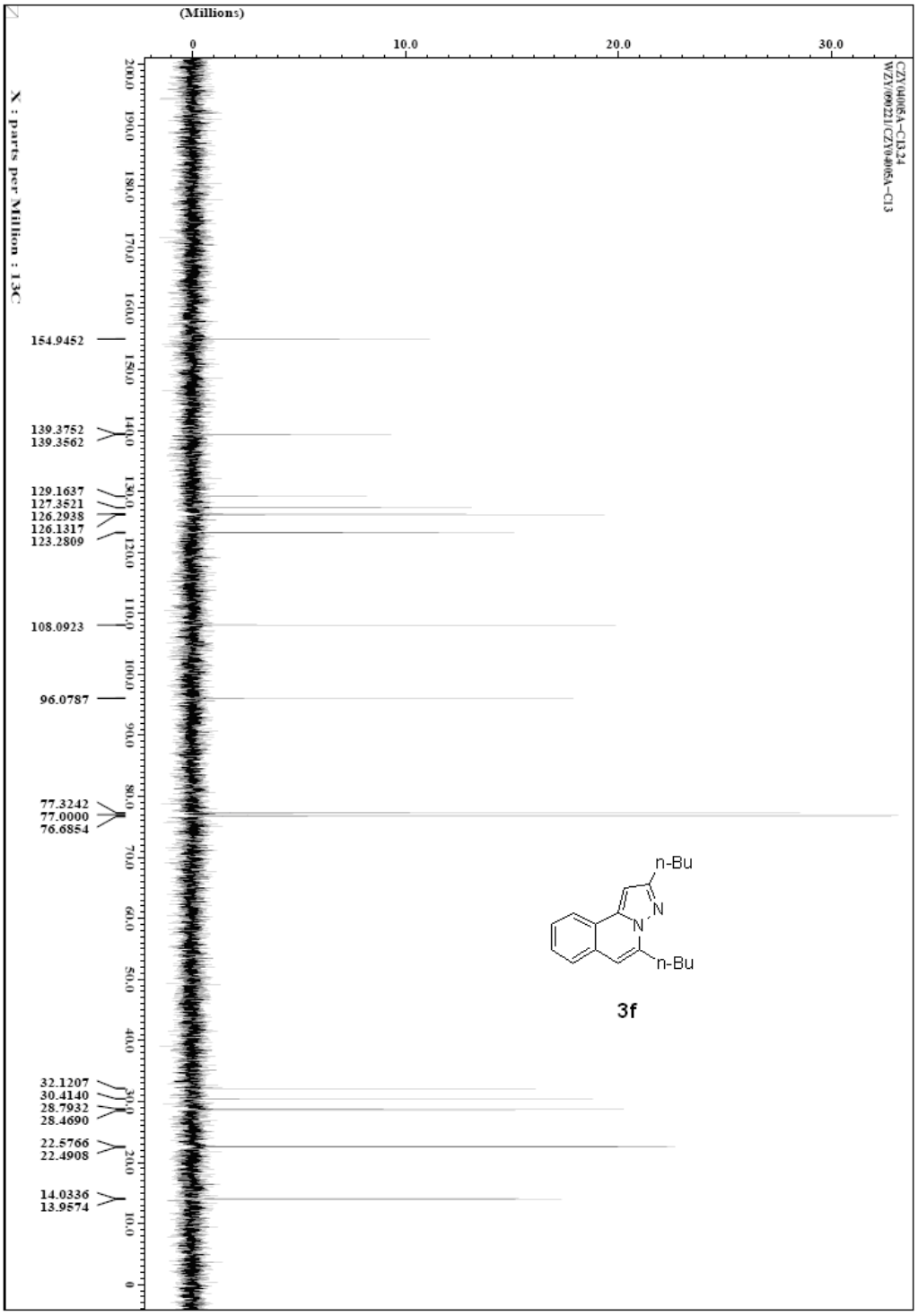
3e


(Millions)
X : parts per Million : 13C
159.6099
151.7820
139.9591
139.5968
129.2136
127.5260
126.3914
126.2675
123.3403
123.2545
114.0155
108.9145
93.9834
77.3169
77.0023
76.6781
55.2539
30.5212
28.8813
22.5503
13.9882
OMe
N
N
n-Bu
3e

3f

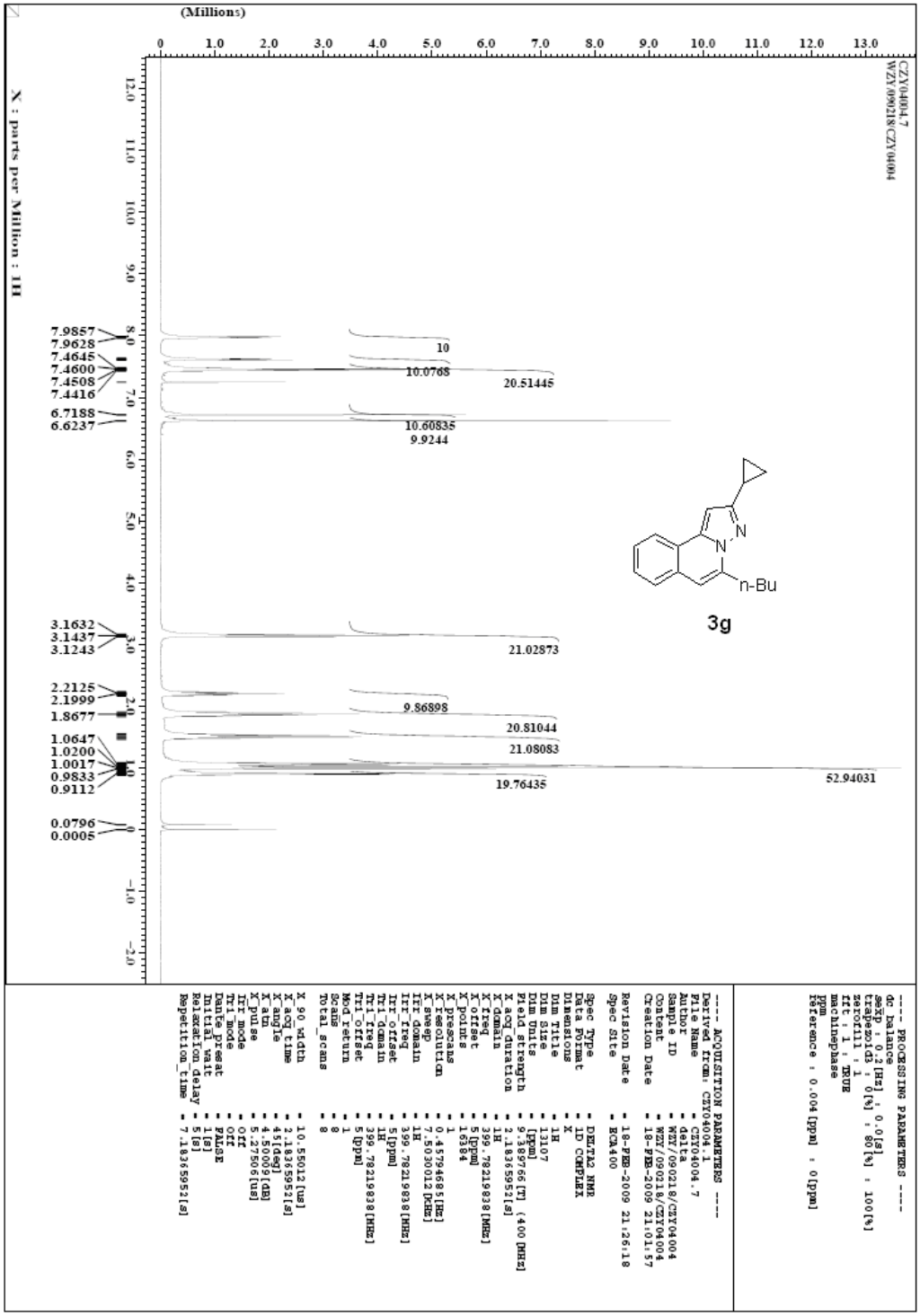

(Millions)
X : parts per Million : 1H
8.0206
8.0080
7.9966
7.4651
7.4548
7.4525
7.4422
7.2429
6.8190
6.7274
3.1500
3.1305
2.8877
2.8682
1.8716
1.7800
1.7605
1.5223
1.5028
1.4753
1.4570
0.9999
0.9942
0.9759
0.0824
-0.0001
9.91071
9.68445
19.8288
9.70401
9.78951
20
20.05444
19.58627
20.40131
40.44126
60.45725
n-Bu
N
N
n-Bu
3f

3f

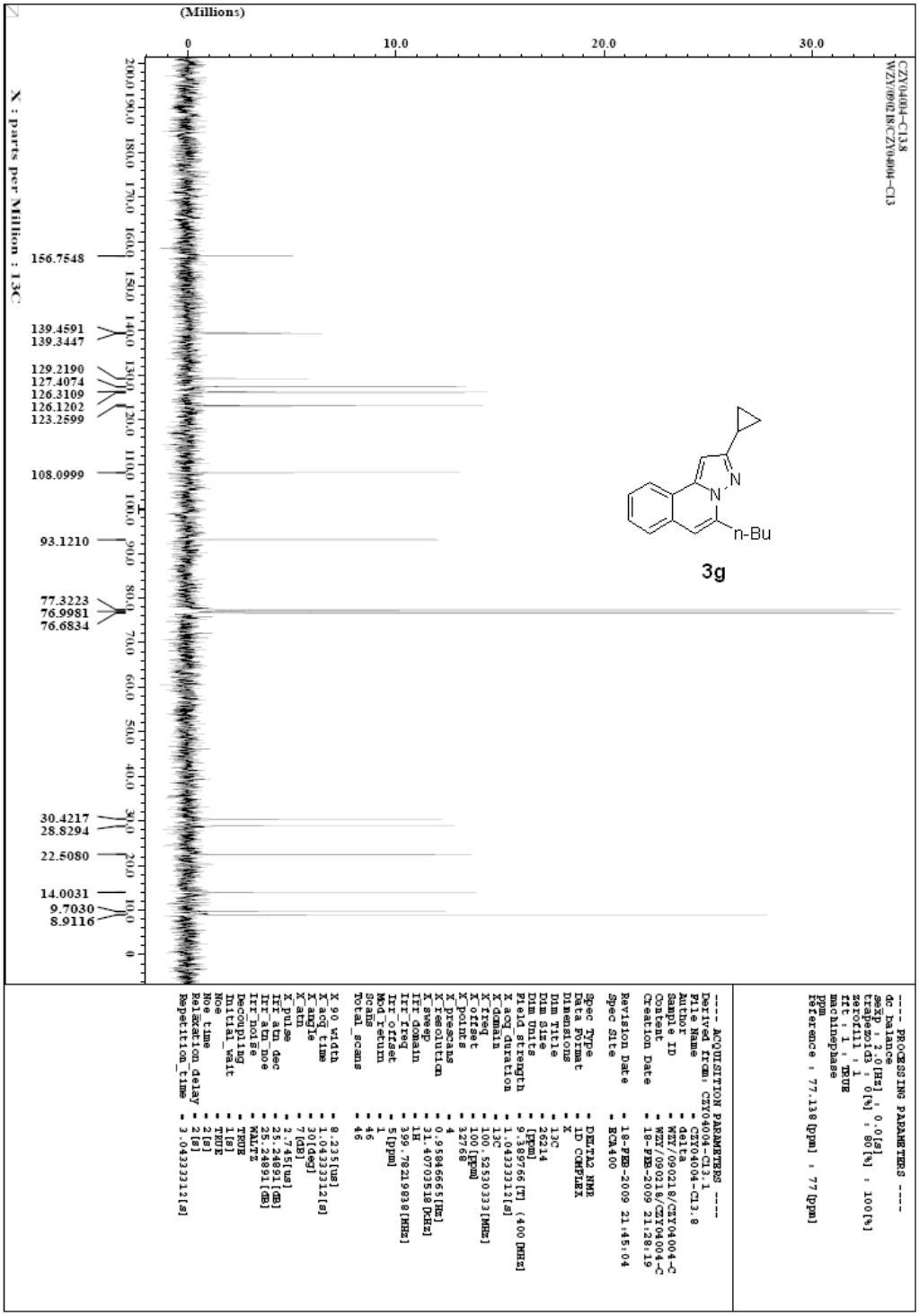

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
CZY04005A-C13-24
WZY090221/CZY04005A-C13
154.9452
139.3752
139.3562
129.1637
127.3521
126.2938
126.1317
123.2809
108.0923
96.0787
77.3242
77.0000
76.6854
32.1207
30.4140
28.7932
28.4690
22.5766
22.4908
14.0336
13.9574
n-Bu
N
N
n-Bu

3f

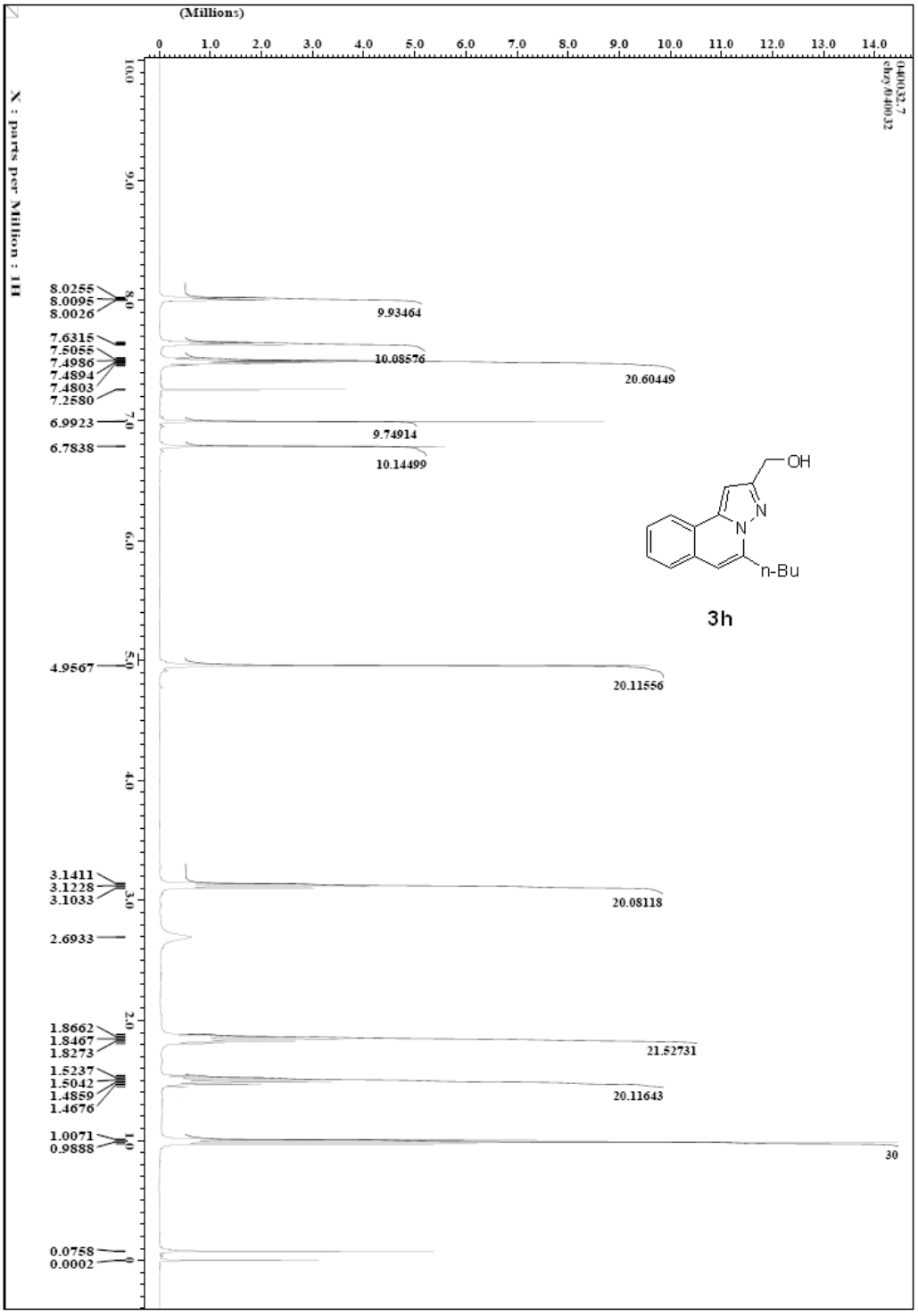
3g


(Millions)
X : parts per Million : 1H
7.9857
7.9628
7.4645
7.4600
7.4508
7.4416
6.7188
6.6237
3.1632
3.1437
3.1243
2.2125
2.1999
1.8677
1.0647
1.0200
1.0017
0.9833
0.9112
0.0796
0.0005
10
10.0768
20.51445
10.60835
9.9244
21.02873
9.86898
20.81044
21.08083
19.76435
52.94031
n-Bu
3g

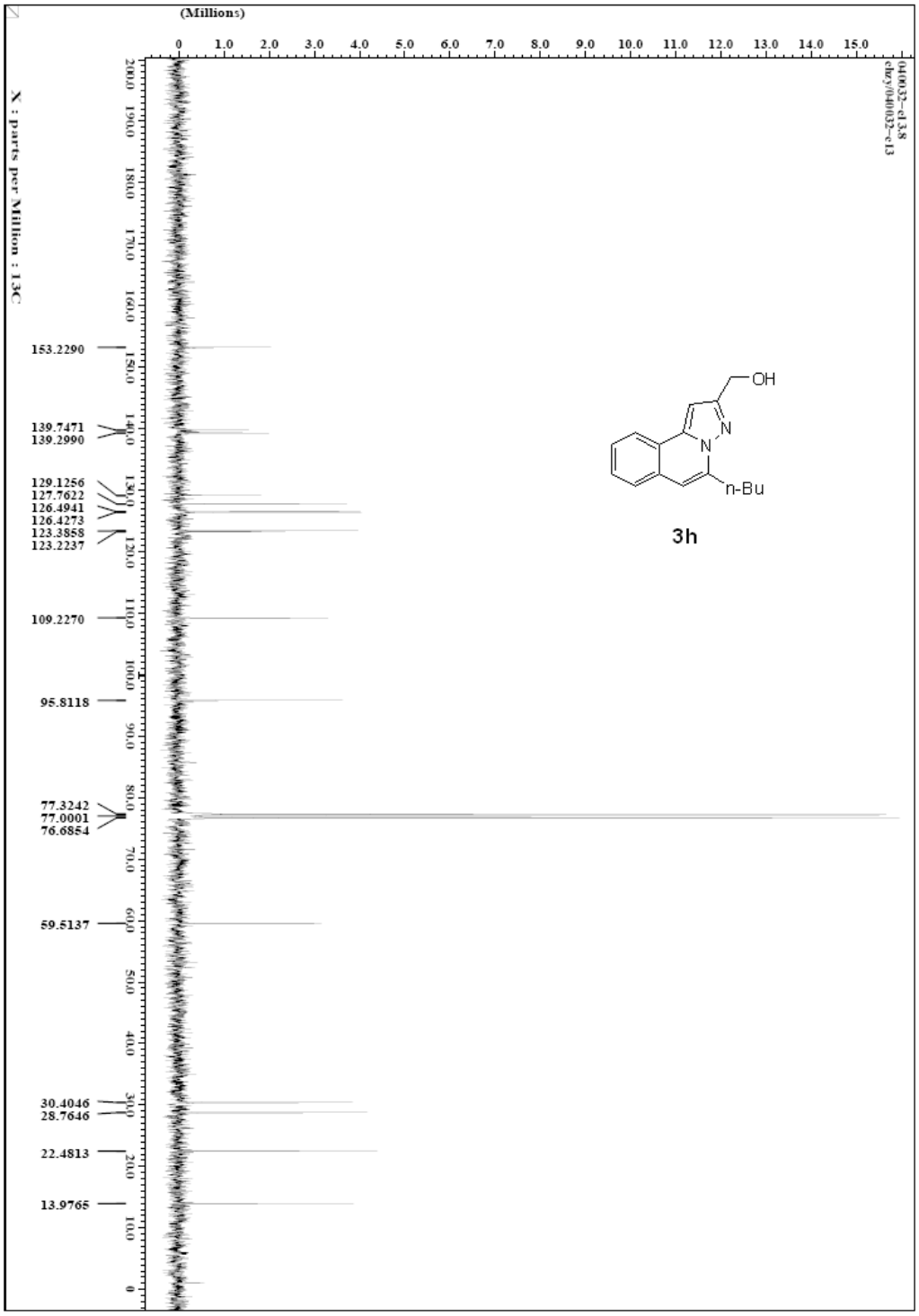
3g


(Millions)
X : parts per Million : 13C
156.7548
139.4591
139.3447
129.2190
127.4074
126.3109
126.1202
123.2599
108.0999
93.1210
77.3223
76.9981
76.6834
30.4217
28.8294
22.5080
14.0031
9.7030
8.9116
n-Bu
3g

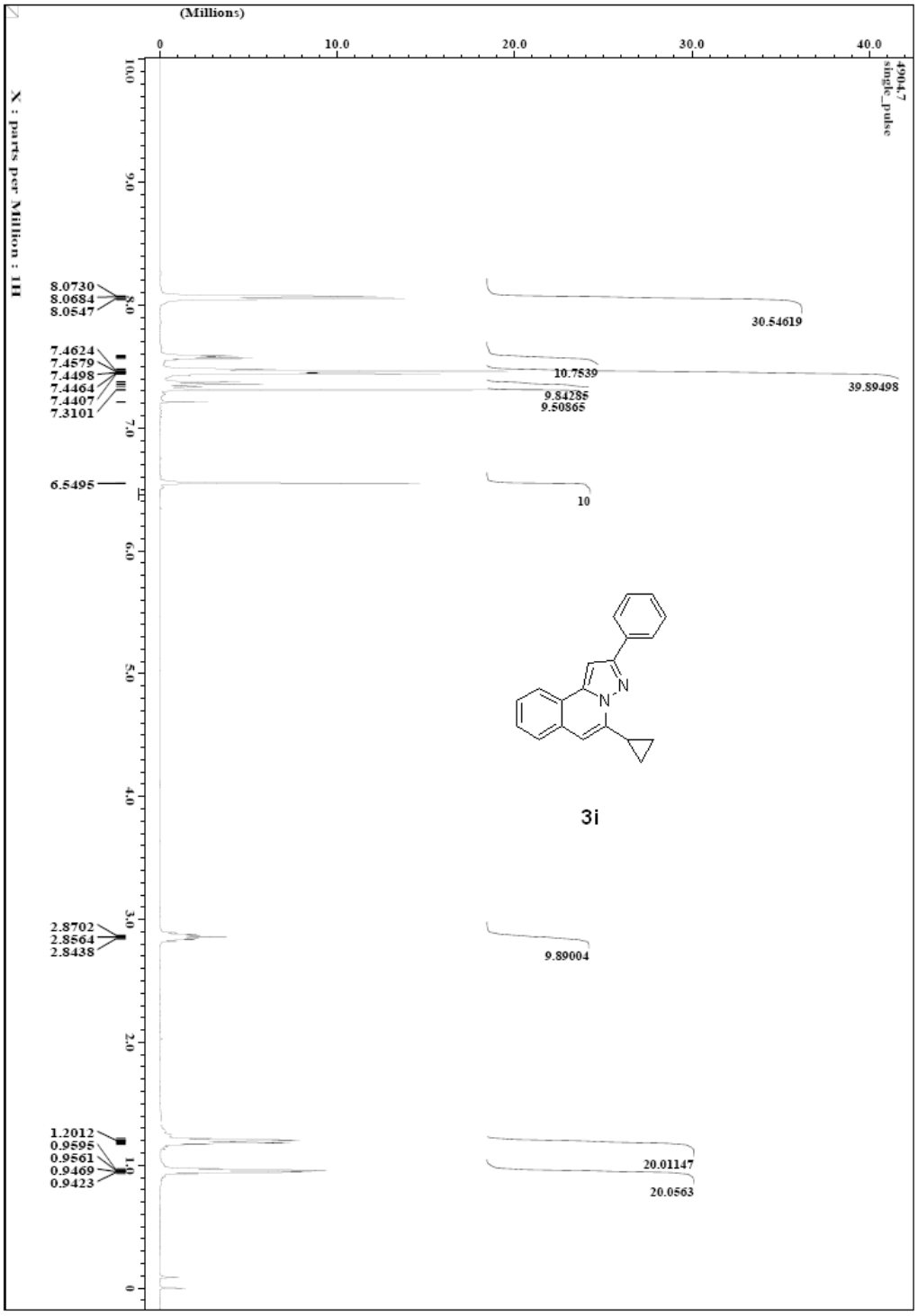
3h


(Millions)
X : parts per Million : 1H
8.0255
8.0095
8.0026
7.6315
7.5055
7.4986
7.4894
7.4803
7.2580
6.9923
6.7838
4.9567
3.1411
3.1228
3.1033
2.6933
1.8662
1.8467
1.8273
1.5237
1.5042
1.4859
1.4676
1.0071
0.9888
0.0758
0.0002
9.93464
10.08576
20.60449
9.74914
10.14499
20.11556
20.08118
21.52731
20.11643
30
OH
N
N
n-Bu
3h

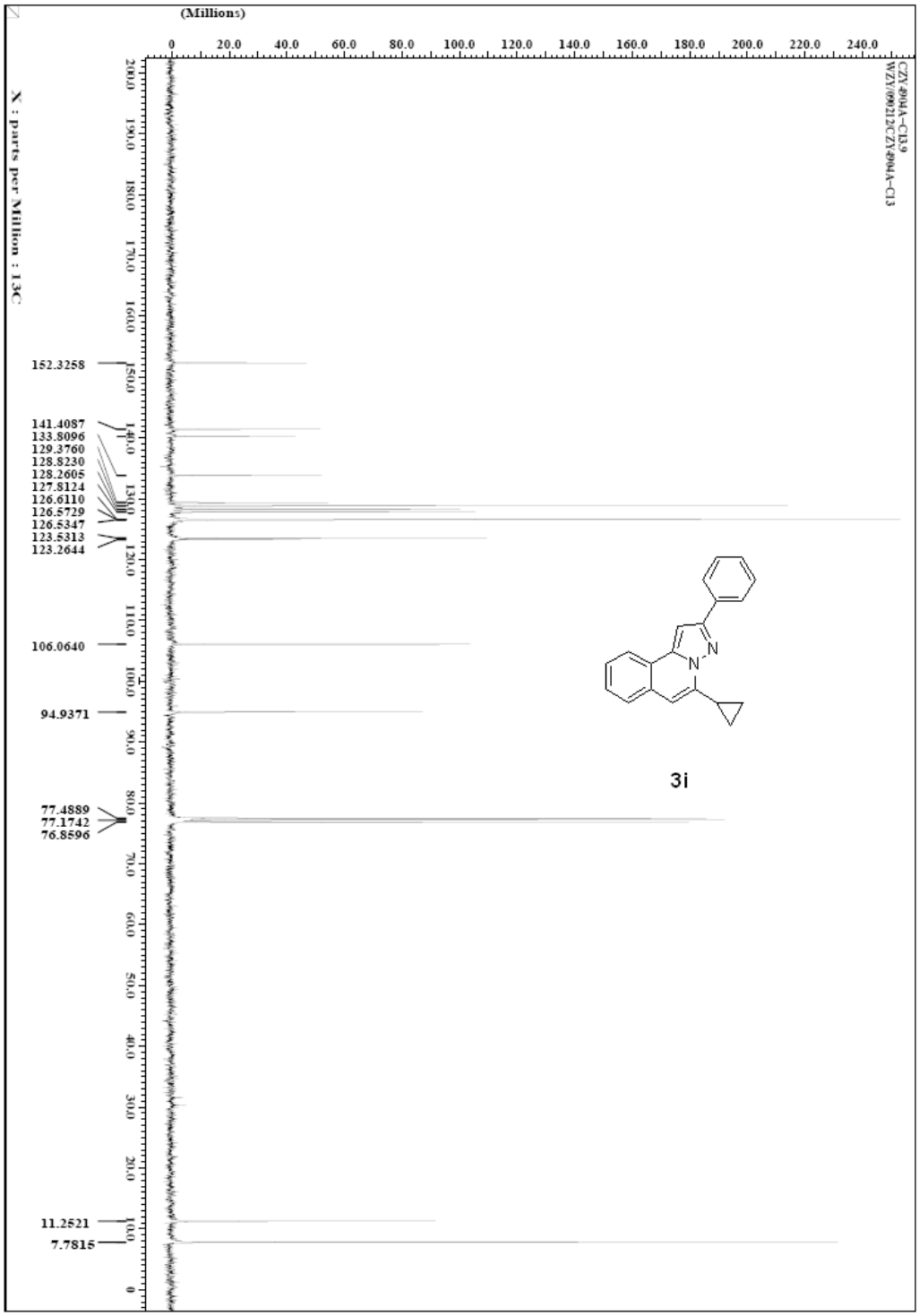
3h


(Millions)
X : parts per Million : 13C
153.2290
139.7471
139.2990
129.1256
127.7622
126.4941
126.4273
123.3858
123.2237
109.2270
95.8118
77.3242
77.0001
76.6854
59.5137
30.4046
28.7646
22.4813
13.9765
OH
N
N
n-Bu
3h

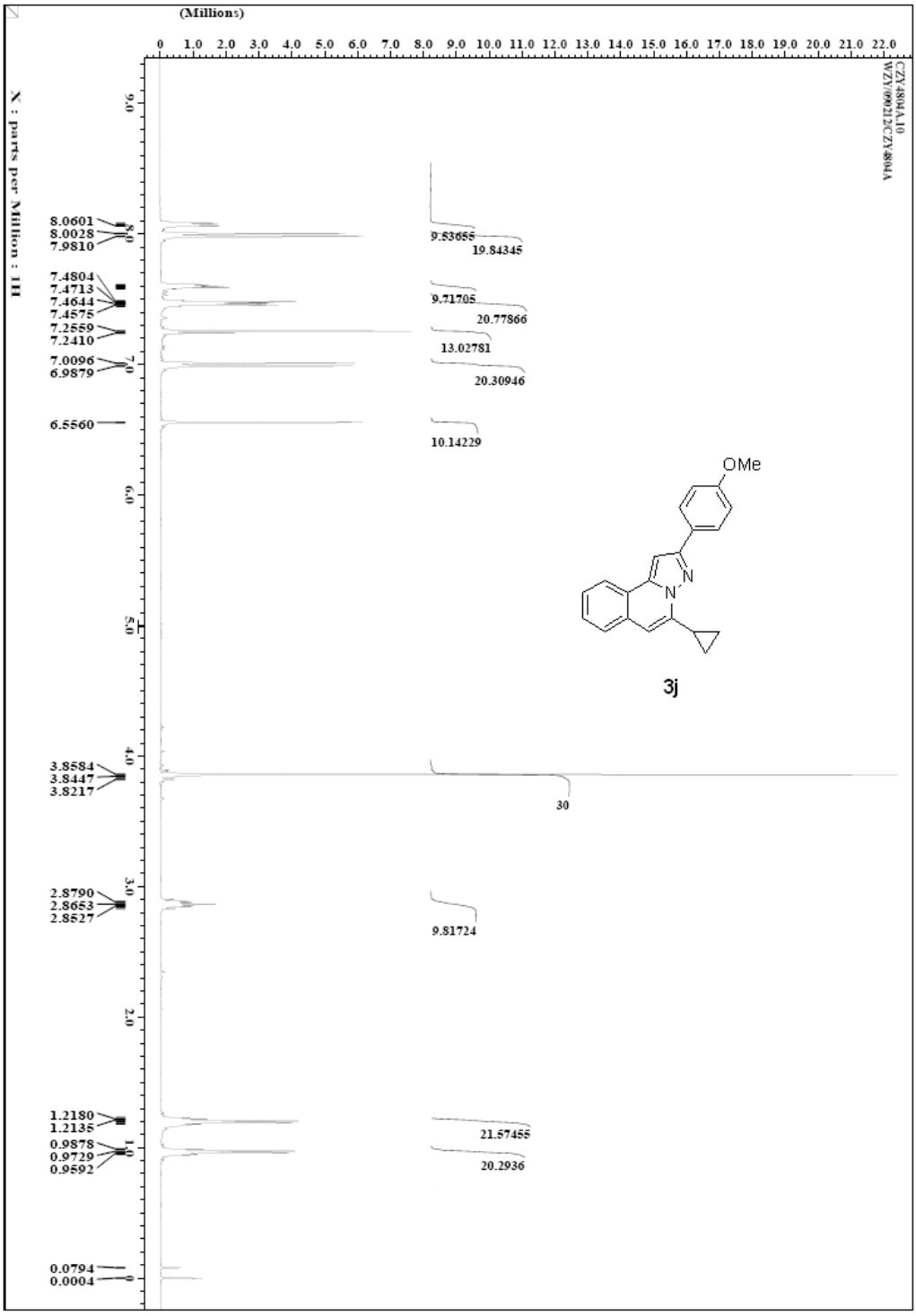
3i


(Millions)
X : parts per Million : 1H
8.0730
8.0684
8.0547
7.4624
7.4579
7.4498
7.4464
7.4407
7.3101
6.5495
2.8702
2.8564
2.8438
1.2012
0.9595
0.9561
0.9469
0.9423
30.54619
10.7539
39.89498
9.84285
9.50865
10
9.89004
20.01147
20.0563
3i

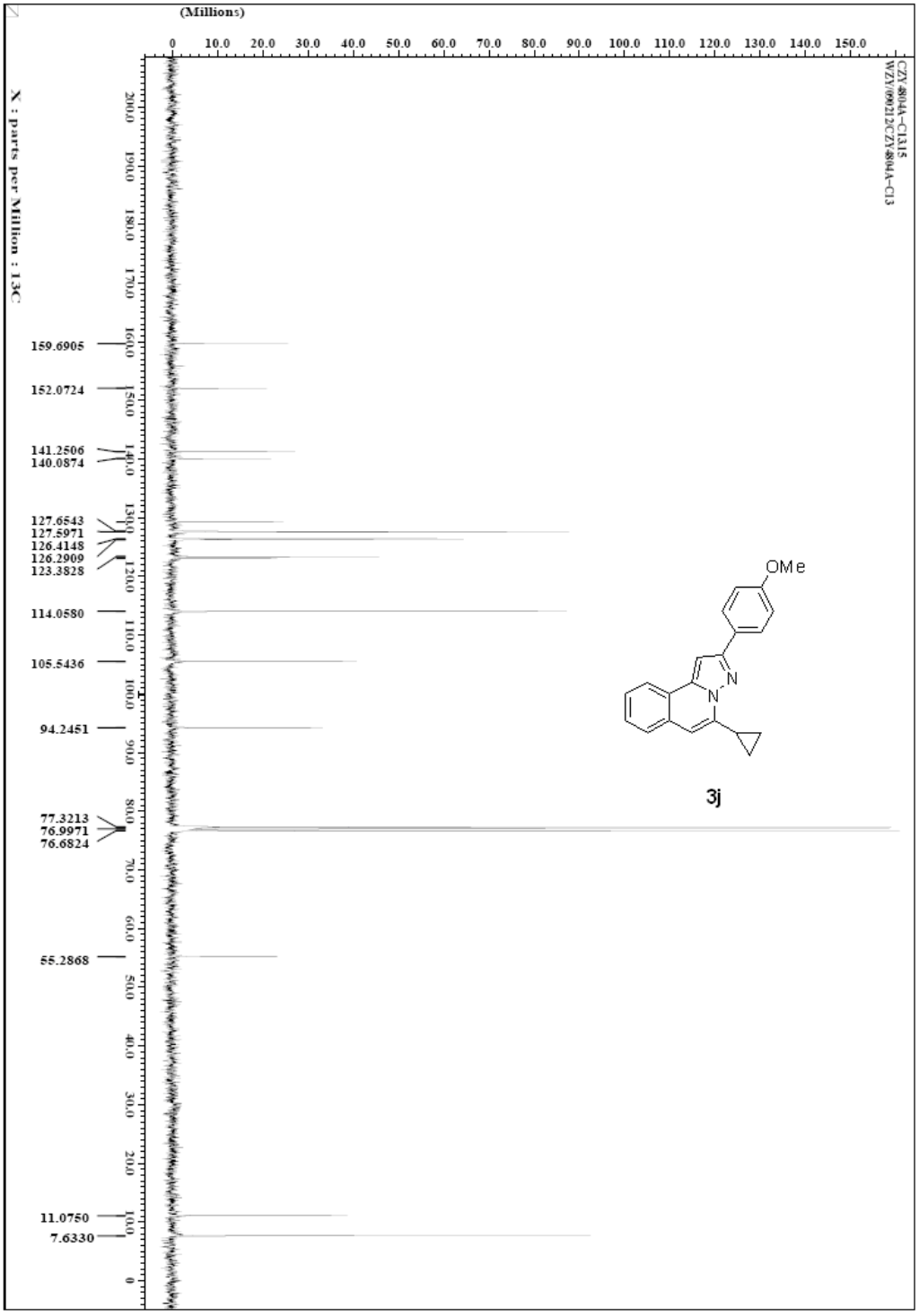
3i


(Millions)
X : parts per Million : 13C
152.3258
141.4087
133.8096
129.3760
128.8230
128.2605
127.8124
126.6110
126.5729
126.5347
123.5313
123.2644
106.0640
94.9371
77.4889
77.1742
76.8596
11.2521
7.7815
3i

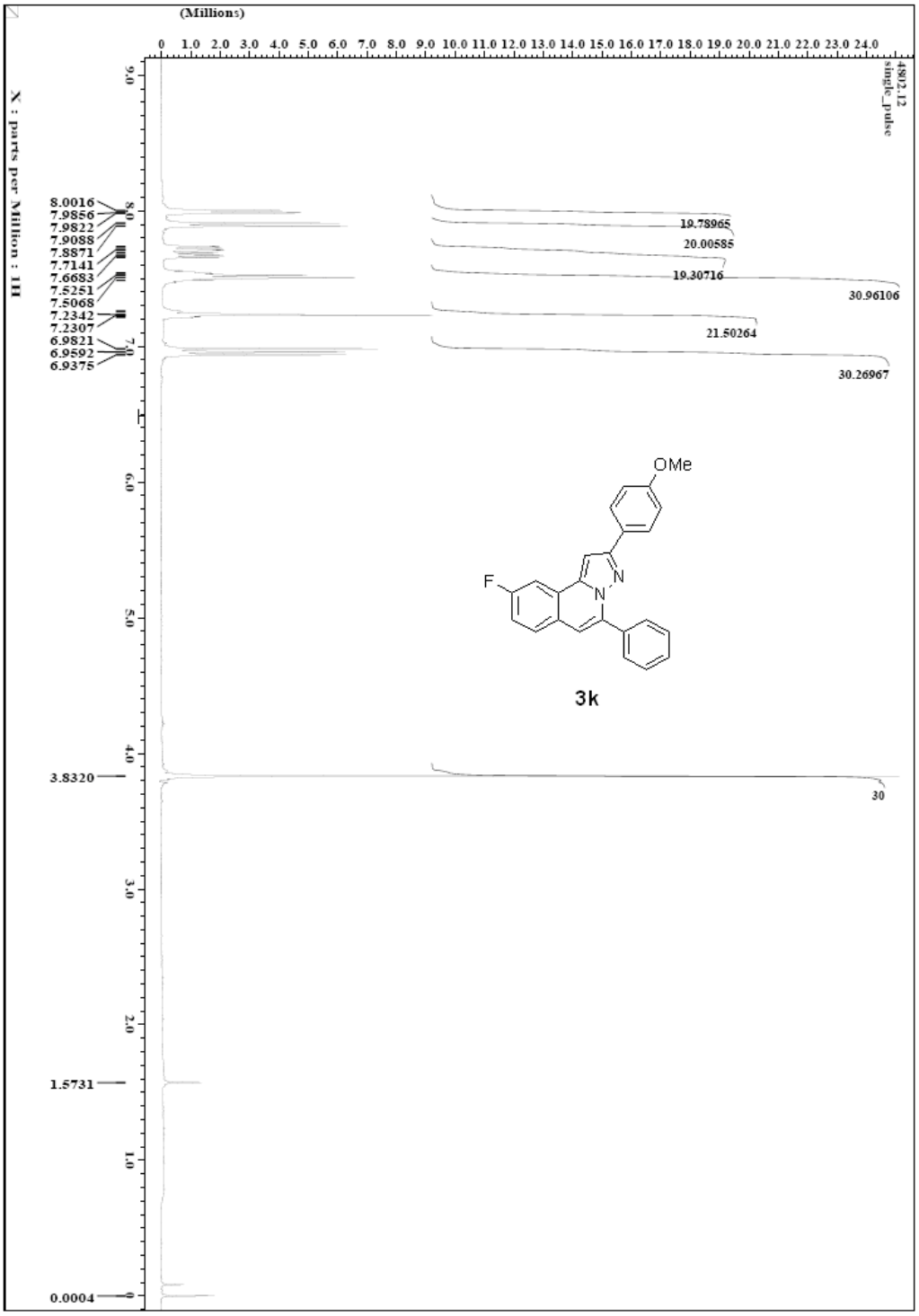
3j


(Millions)
X : parts per Million : 1H
8.0601
8.0028
7.9810
7.4804
7.4713
7.4644
7.4575
7.2569
7.2410
7.0096
6.9879
6.5560
3.8584
3.8447
3.8217
2.8790
2.8653
2.8527
1.2180
1.2135
0.9878
0.9729
0.9592
0.0794
0.0004
9.53655
19.84345
9.71705
20.77866
13.02781
20.30946
10.14229
30
9.81724
21.57455
20.2936
OMe
N
N
3j

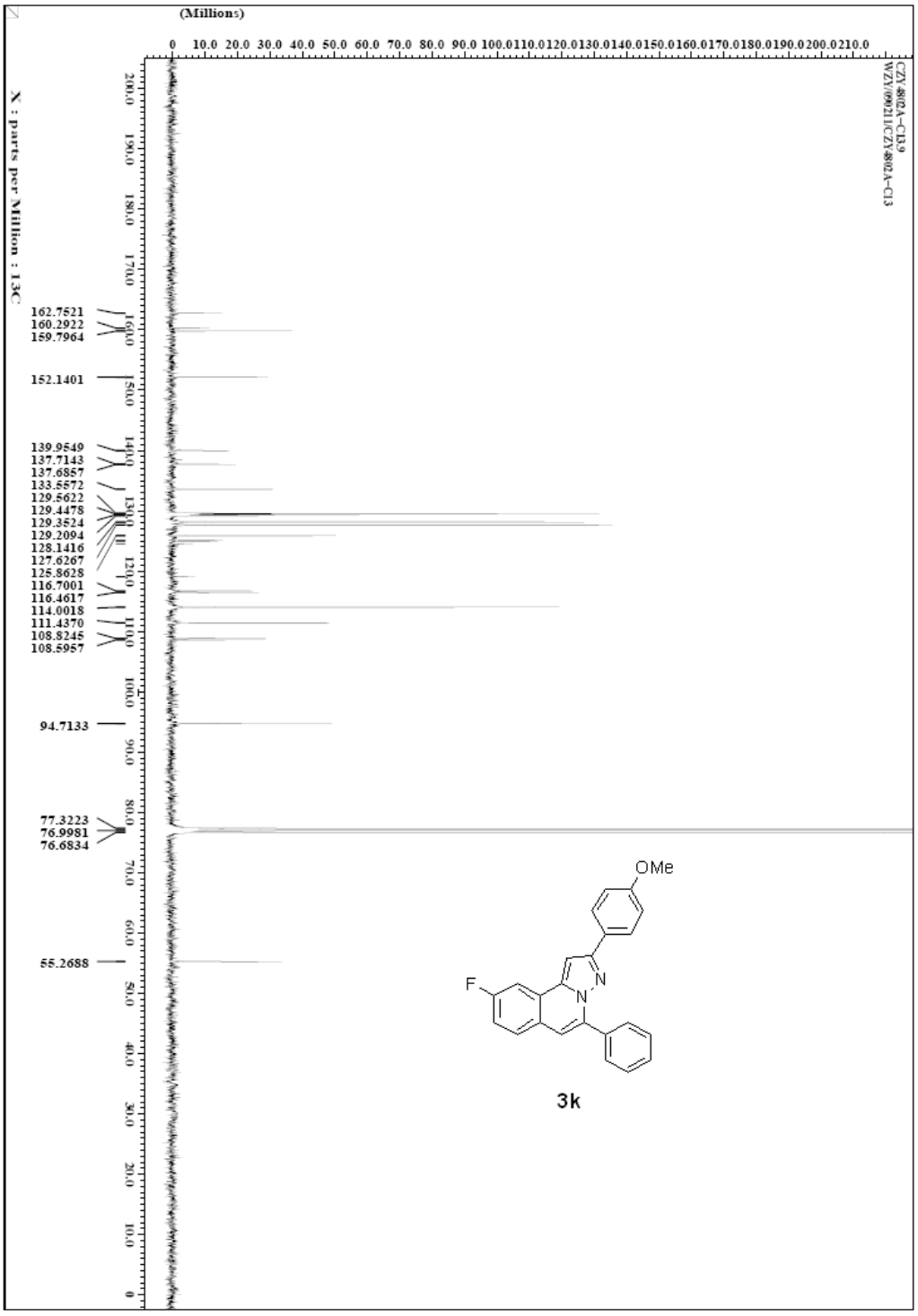
3j


(Millions)
X : parts per Million : 13C
159.6905
152.0724
141.2506
140.0874
127.6543
127.5971
126.4148
126.2909
123.3828
114.0580
105.5436
94.2451
77.3213
76.9971
76.6824
55.2868
11.0750
7.6330
OMe
N
N
3j

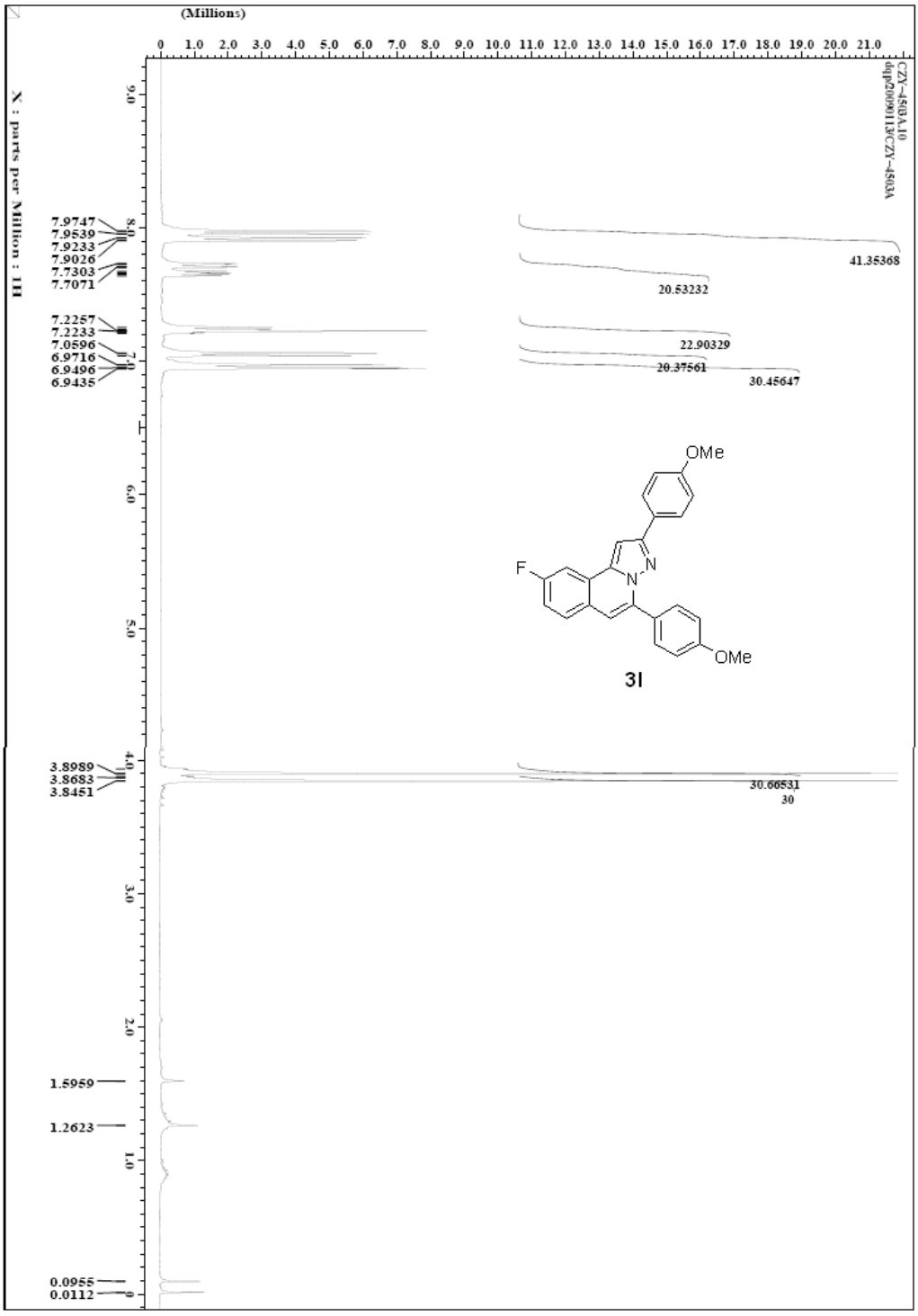
3k


(Millions)
X : parts per Million : 1H
4802.12
single_pulse
8.0016
7.9856
7.9822
7.9088
7.8871
7.7141
7.6683
7.5251
7.5068
7.2342
7.2307
6.9821
6.9592
6.9375
3.8320
1.5731
0.0004
19.78965
20.00585
19.30716
30.96106
21.50264
30.26967
30
OMe
F
N
N
3k

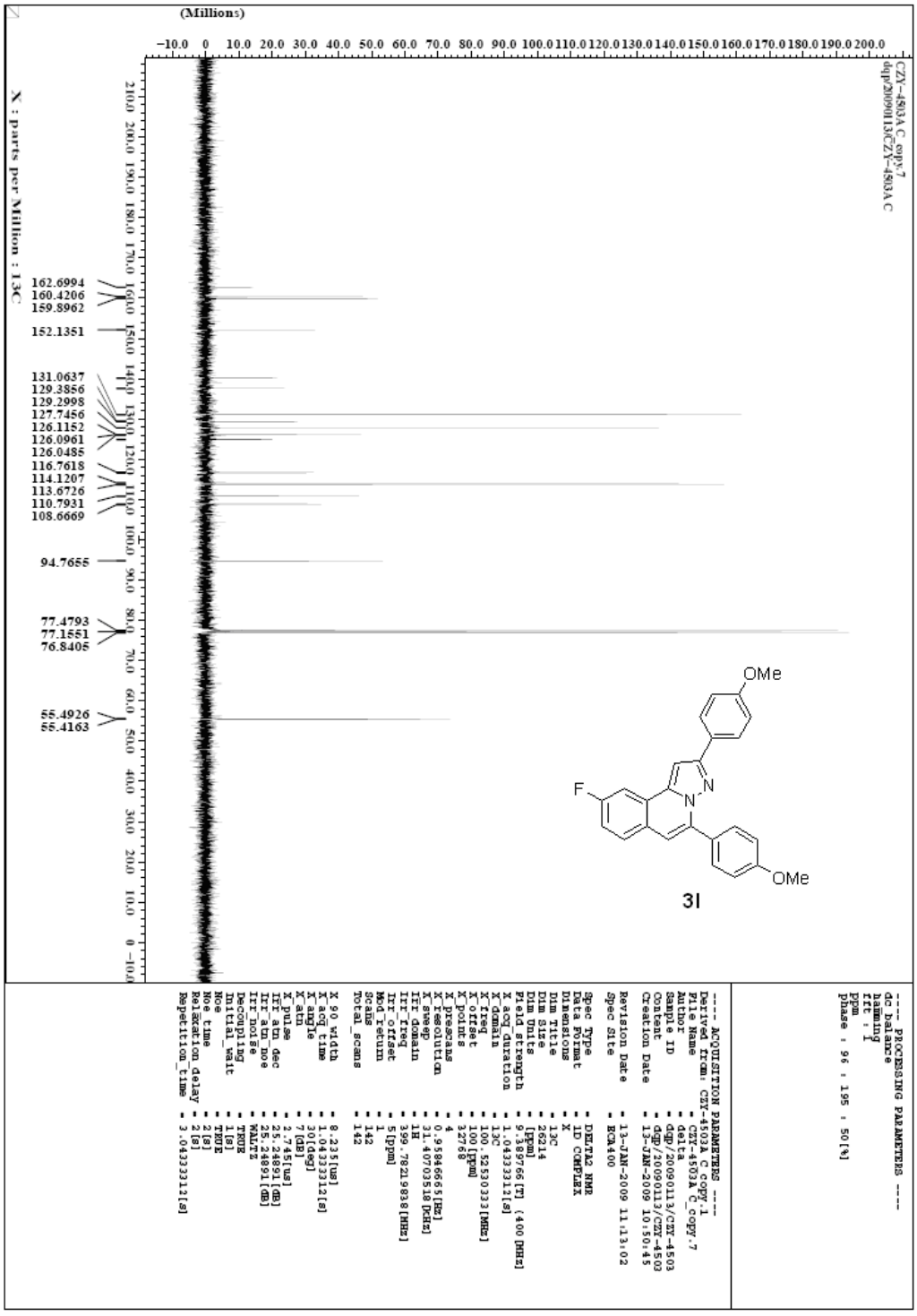
3k


(Millions)
X : parts per Million : 13C
162.7521
160.2922
159.7964
152.1401
139.9549
137.7143
137.6857
133.5572
129.5622
129.4478
129.3524
129.2094
128.1416
127.6267
125.8628
116.7001
116.4617
114.0018
111.4370
108.8245
108.5957
94.7133
77.3223
76.9981
76.6834
55.2688
OMe
F
N
N
3k

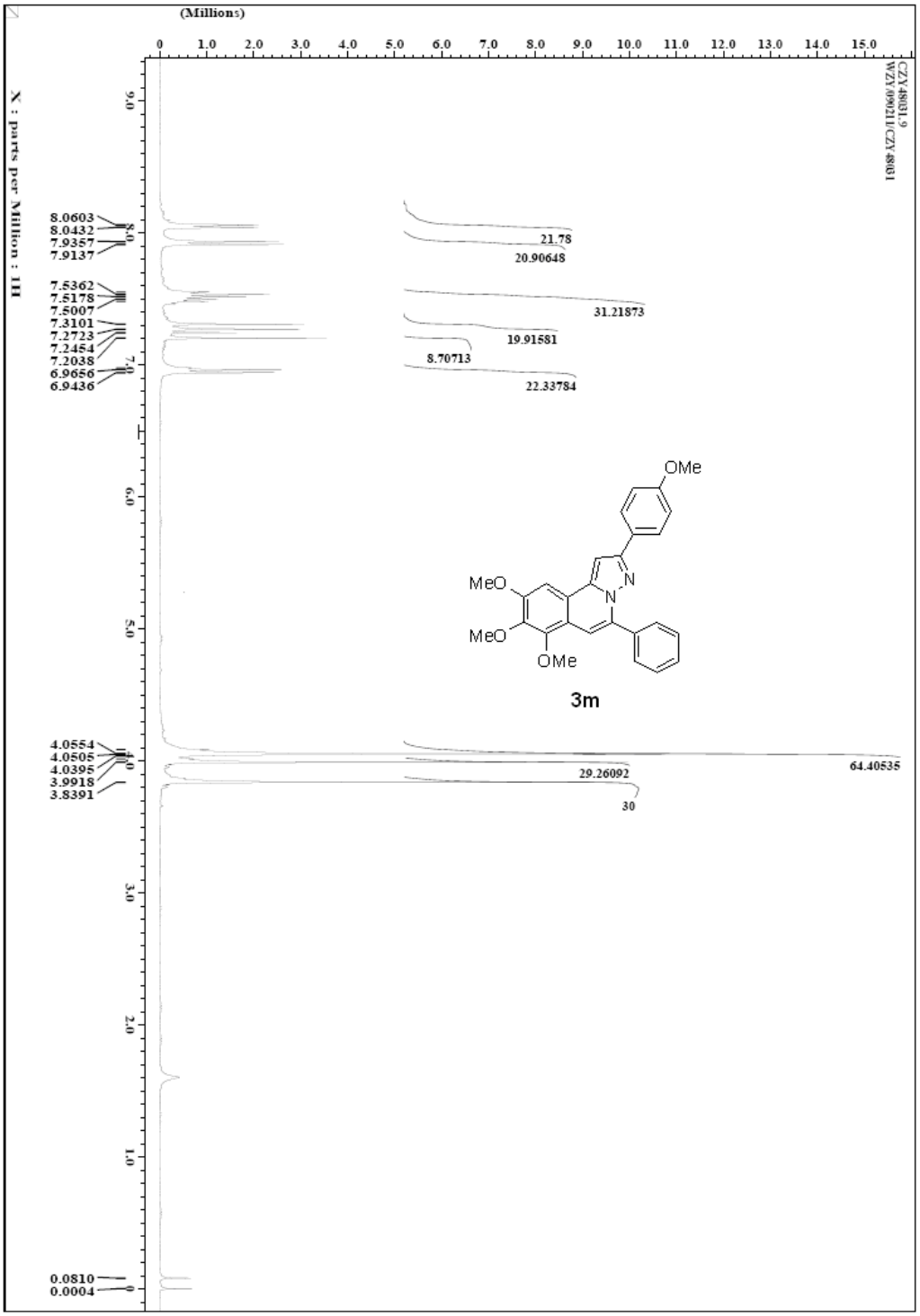
3l


(Millions)
X : parts per Million : 1H
7.9747
7.9539
7.9233
7.9026
7.7303
7.7071
7.2257
7.2233
7.0596
6.9716
6.9496
6.9435
3.8989
3.8683
3.8451
1.5959
1.2623
0.0955
0.0112
41.35368
20.53232
22.90329
20.37561
30.45647
30.66531
OMe
F
N
N
OMe
3l

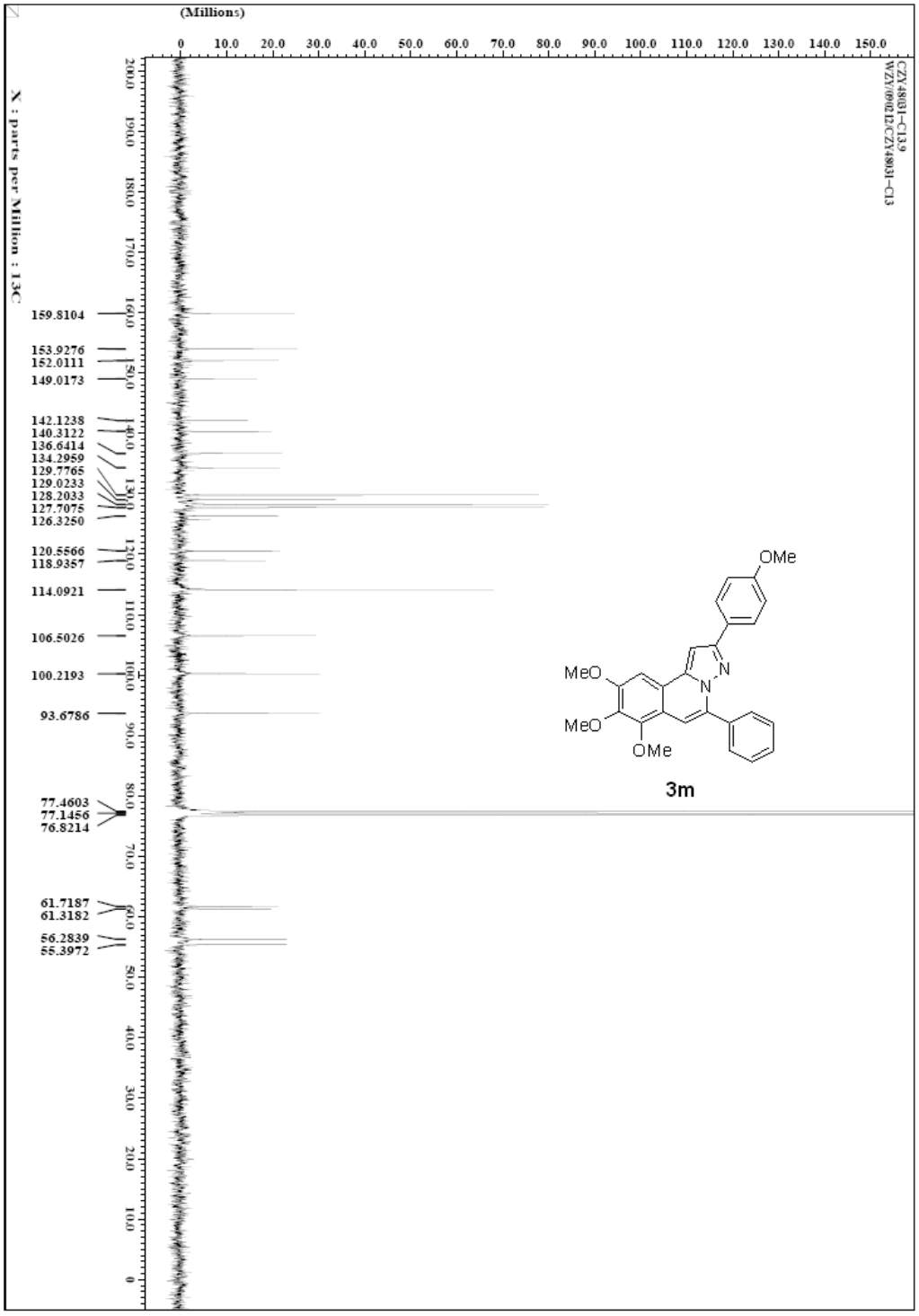
3l


(Millions)
X : parts per Million : 13C
162.6994
160.4206
159.8962
152.1351
131.0637
129.3856
129.2998
127.7456
126.1152
126.0961
126.0485
116.7618
114.1207
113.6726
110.7931
108.6669
94.7655
77.4793
77.1551
76.8405
55.4926
55.4163
OMe
F
N
N
OMe
3l
---- PROCESSING PARAMETERS ----
dc_balance
Running
ft
ppm : 1
phase : 96 : 195 : 50[%]
---- ACQUISITION PARAMETERS ----
Derived from: CZY-4503A C_copy.1
File Name - CZY-4503A C_copy.7
Author - delta
Sample ID - dqp/20090113/CZY-4503
Content - dqp/20090113/CZY-4503
Creation Date - 13-JAN-2009 10:50:45
Revision Date - 13-JAN-2009 11:13:02
Spec Site - ECA400
Spec Type - DELTA2_NMR
Nucleus - 13C
Dimensions - 1D COMPLEX
Dim Title - 13C
Dim Size - 26214
Dim Units - [ppm]
Field_strength - 9.389766 [T] (400 [MHz]
X_acq_duration - 1.043333112[s]
X_domain - 13C
X_freq - 100.525303333 [MHz]
X_offset - 100 [ppm]
X_points - 32768
X_prescans - 4
X_resolution - 0.958466651 [Hz]
X_sweep - 31.44070318 [kHz]
Irr_domain - 1H
Irr_freq - 399.782219838 [MHz]
Irr_offset - 5 [ppm]
Mod_return - 1
Scans - 142
Total_scans - 142
X_90_width - 8.235[us]
X_acq_time - 1.043333112[s]
X_angle - 30[deg]
X_atn - 7[dB]
X_pulse - 2.745[us]
Irr_atn_dec - 25.24891[dB]
Irr_atn_one - 25.24891[dB]
Irr_noise - WALTZ
Decoupling - TRUE
Initial_wait - 1[s]
Mode - TRUE
Noe_time - 2[s]
Relaxation_delay - 2[s]
Repetition_time - 3.043333112[s]

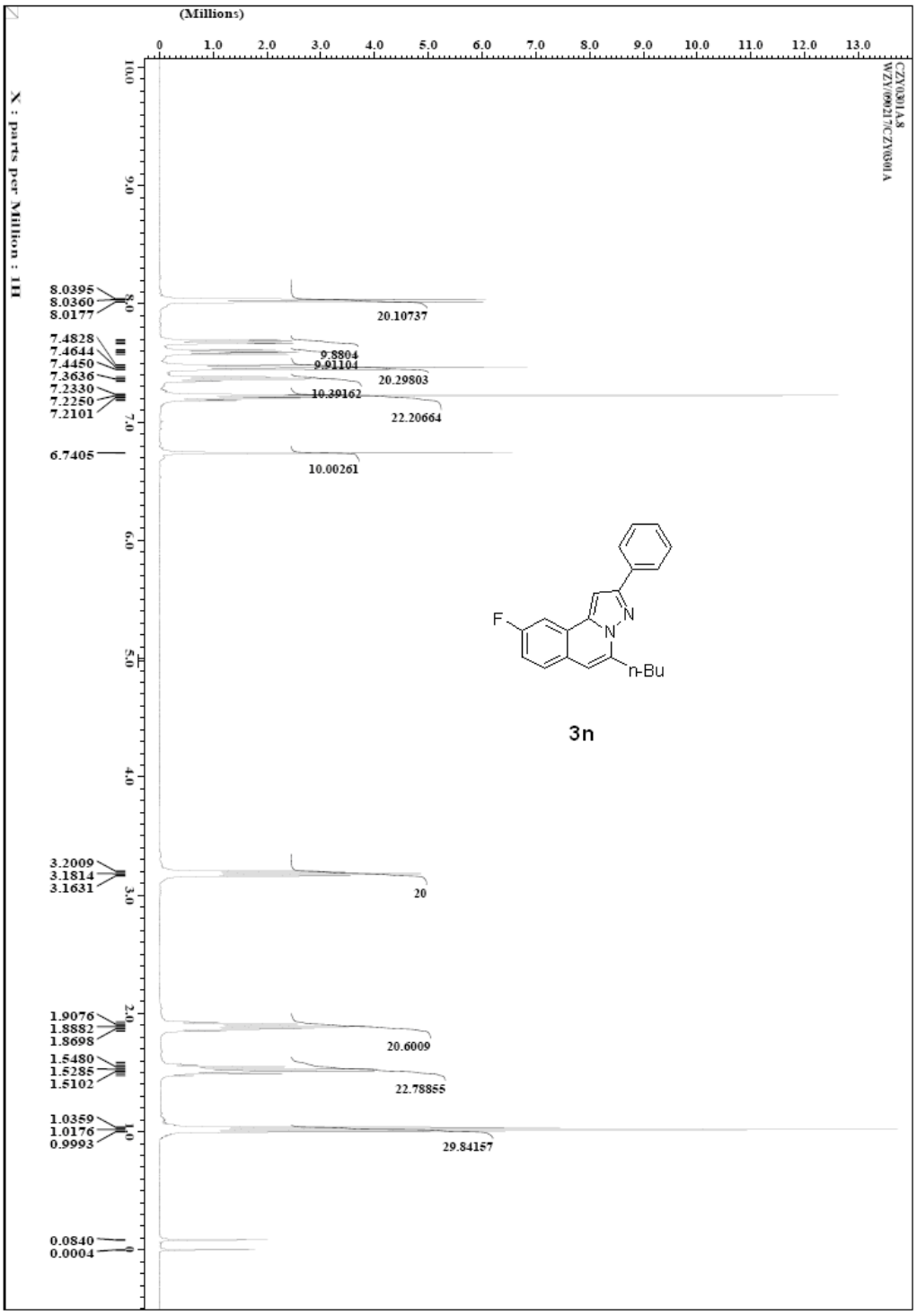
3m


(Millions)
X : parts per Million : 1H
8.0603
8.0432
7.9357
7.9137
7.5362
7.5178
7.5007
7.3101
7.2723
7.2454
7.2038
6.9656
6.9436
4.0554
4.0505
4.0395
3.9918
3.8391
0.0810
0.0004
21.78
20.90648
31.21873
19.91581
8.70713
22.33784
64.40535
29.26092
30
OMe
MeO
MeO
OMe
N
N
3m

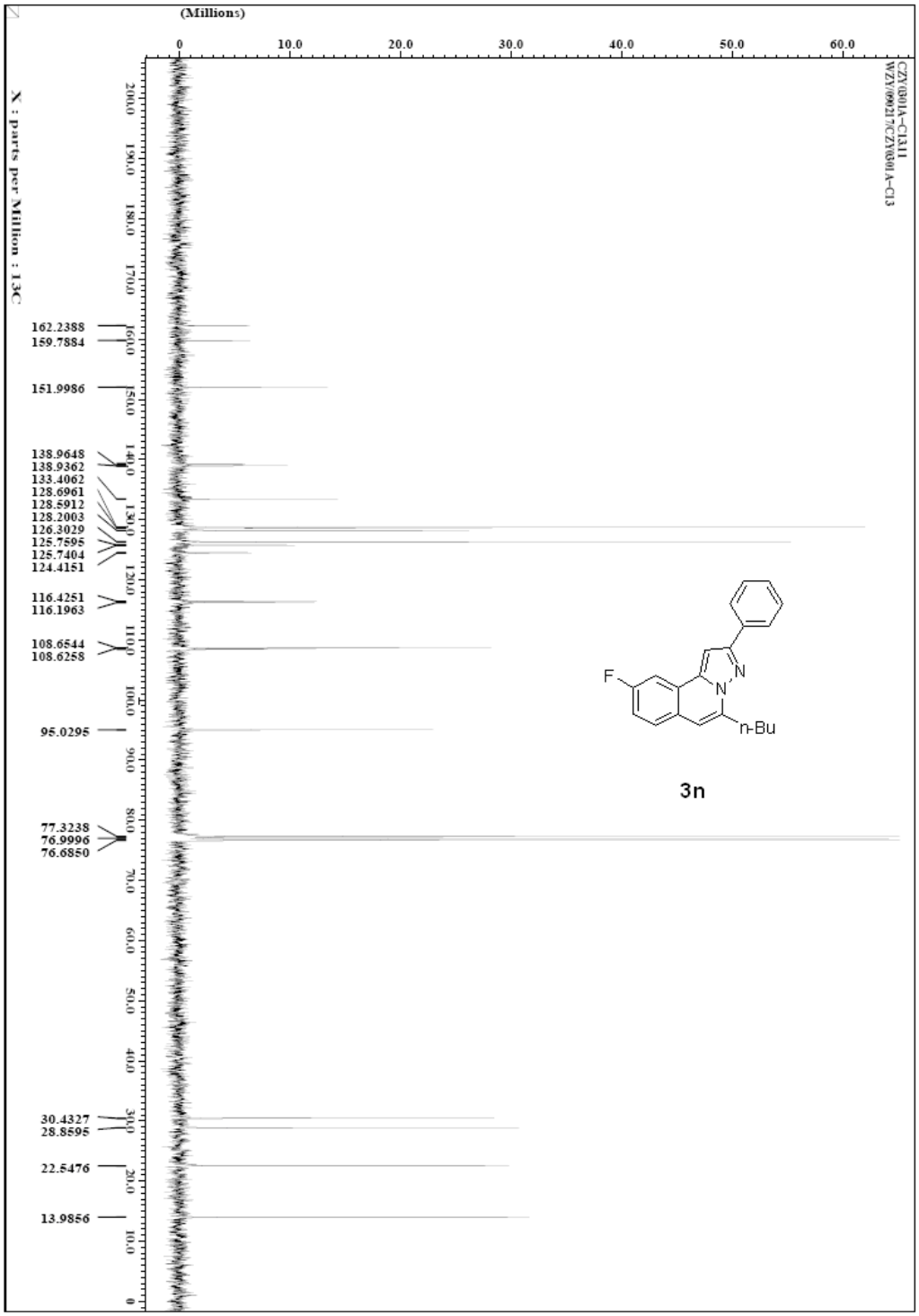
3m


(Millions)
X : parts per Million : 13C
159.8104
153.9276
152.0111
149.0173
142.1238
140.3122
136.6414
134.2959
129.7765
129.0233
128.2033
127.7075
126.3250
120.5566
118.9357
114.0921
106.5026
100.2193
93.6786
77.4603
77.1456
76.8214
61.7187
61.3182
56.2839
55.3972
OMe
MeO
MeO
OMe
N
N
3m

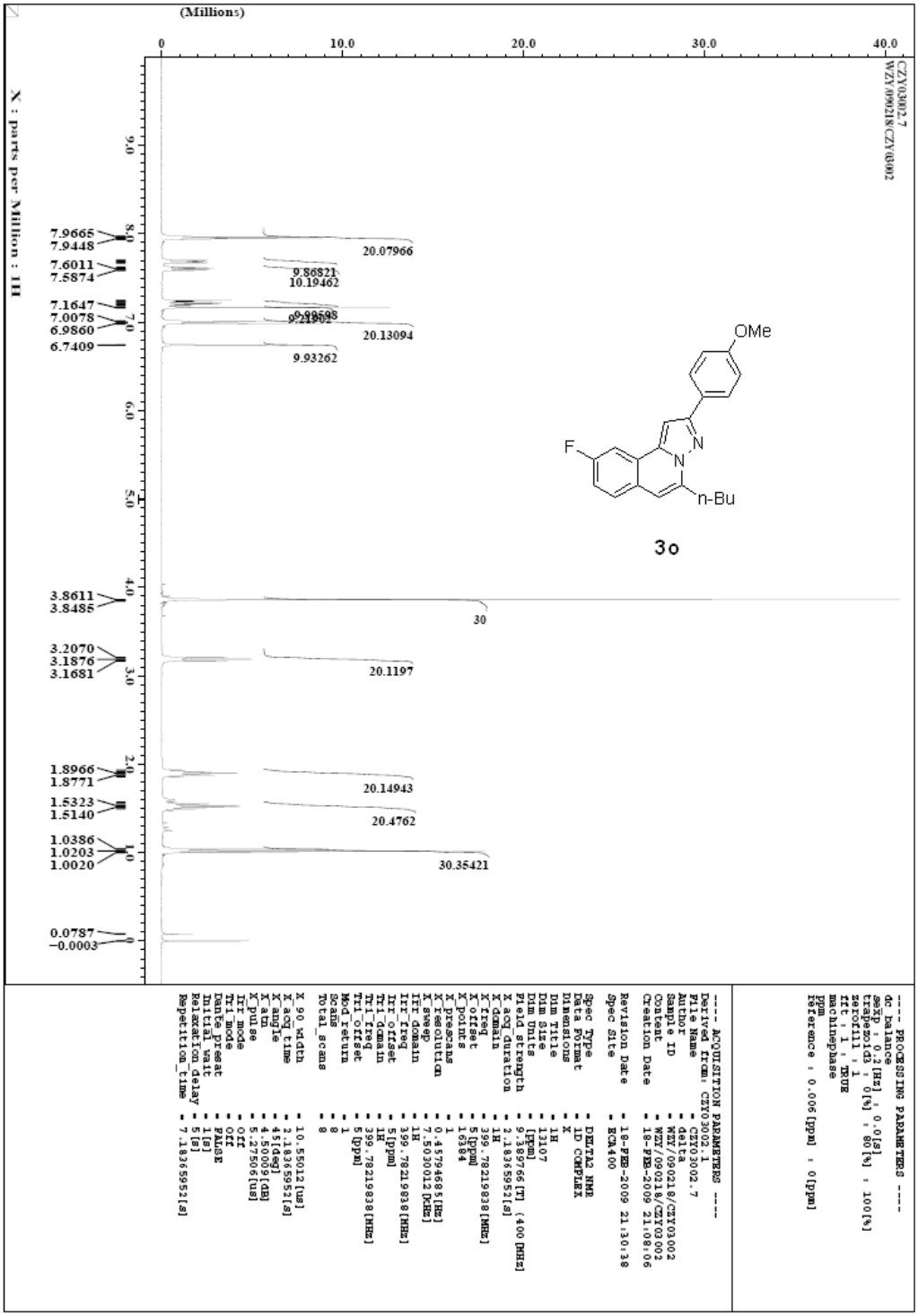
3n


(Millions)
X : parts per Million : 1H
8.0395
8.0360
8.0177
7.4828
7.4644
7.4450
7.3636
7.2330
7.2250
7.2101
6.7405
3.2009
3.1814
3.1631
1.9076
1.8882
1.8698
1.5480
1.5285
1.5102
1.0359
1.0176
0.9993
0.0840
0.0004
20.10737
9.8804
9.91104
20.29803
10.39162
22.20664
10.00261
20
20.6009
22.78855
29.84157
F
N
N
n-Bu
3n

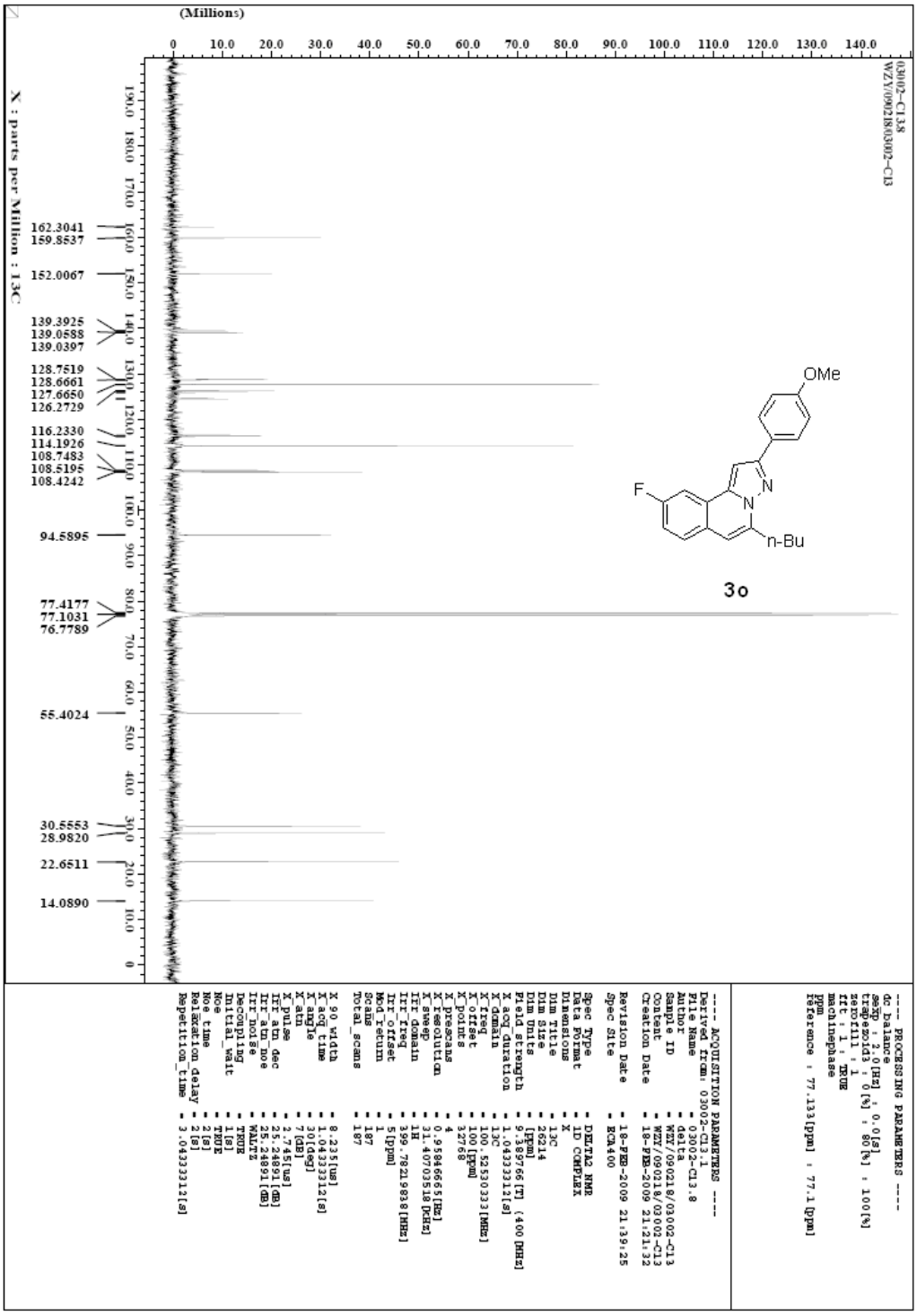
3n


(Millions)
X : parts per Million : 13C
162.2388
159.7884
151.9986
138.9648
138.9362
133.4062
128.6961
128.5912
128.2003
126.3029
125.7595
125.7404
124.4151
116.4251
116.1963
108.6544
108.6258
95.0295
77.3238
76.9996
76.6850
30.4327
28.8595
22.5476
13.9856
F
N
N
n-Bu
3n

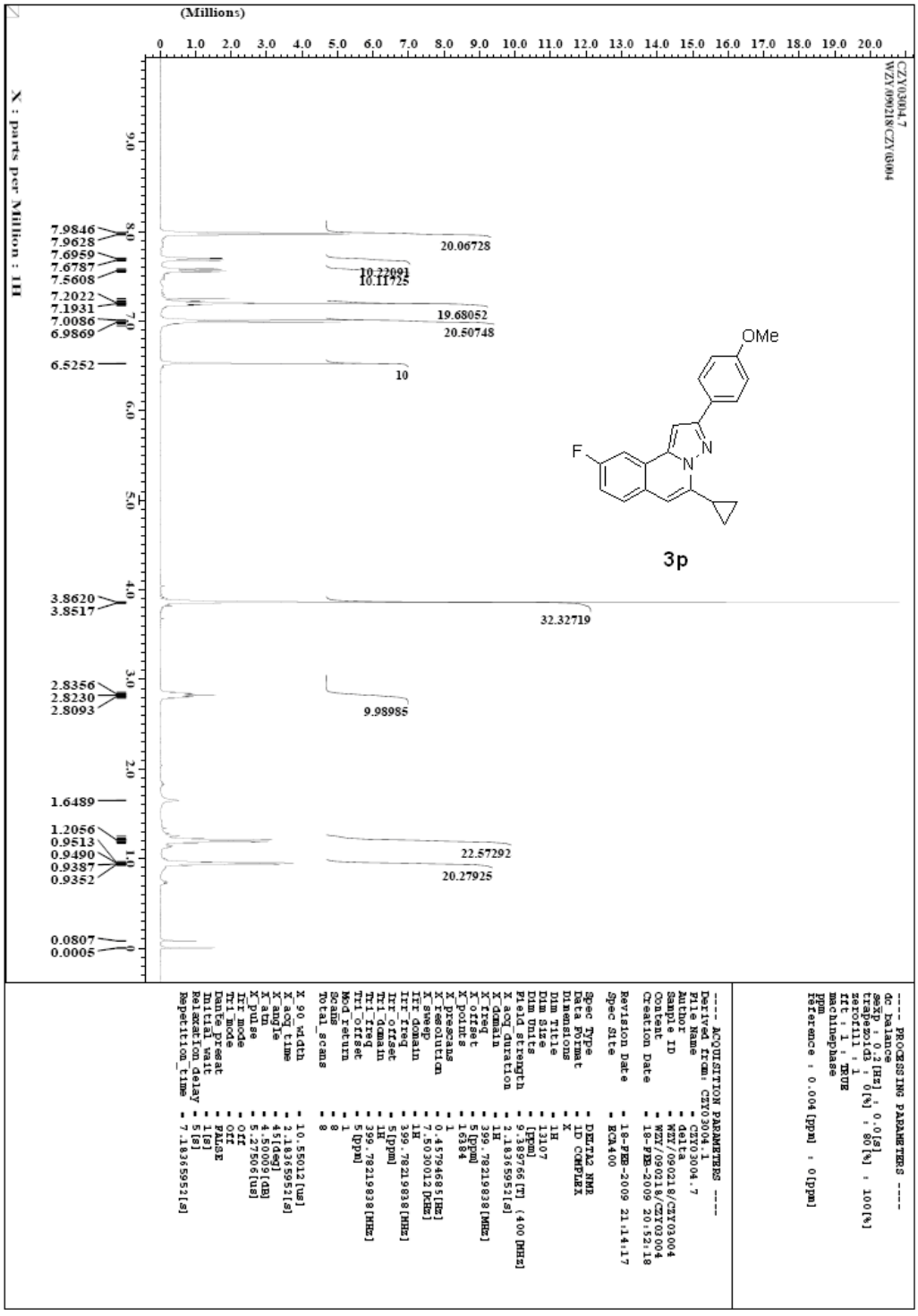
3o


(Millions)
X : parts per Million : 1H
7.9665
7.9448
7.6011
7.5874
7.1647
7.0078
6.9860
6.7409
3.8611
3.8485
3.2070
3.1876
3.1681
1.8966
1.8771
1.5323
1.5140
1.0386
1.0203
1.0020
0.0787
-0.0003
20.07966
9.86821
10.19462
9.98598
9.21902
20.13094
9.93262
30
20.1197
20.14943
20.4762
30.35421
OMe
F
N
N
n-Bu
3o

3o


(Millions)
X : parts per Million : 13C
162.3041
159.8537
152.0067
139.3925
139.0588
139.0397
128.7519
128.6661
127.6650
126.2729
116.2330
114.1926
108.7483
108.5195
108.4242
94.5895
77.4177
77.1031
76.7789
55.4024
30.5553
28.9820
22.6511
14.0890
OMe
F
N
N
n-Bu
3o

3p

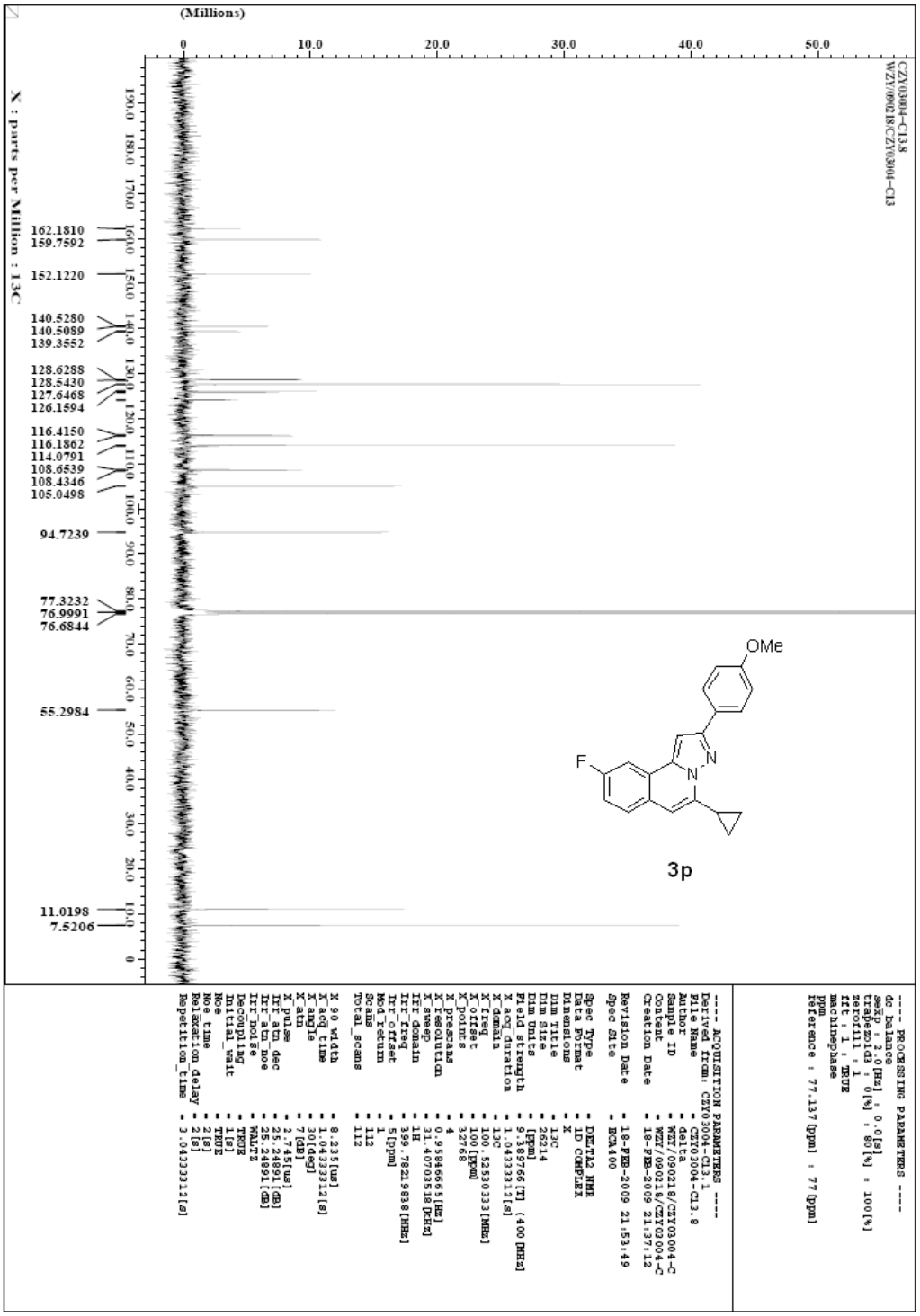


CZY03047
WZY090218/CZY03004

--- PROCESSING PARAMETERS ----
gc_balance
sexp : 0[Hz] ; 0.0[s]
trapezoid : 0[%] ; 80[%] ; 100[%]
zerofill : 1
fft : 1 ; TRUE
machinephase
ppm
reference : 0.004[ppm] ; 0[ppm]

---- ACQUISITION PARAMETERS ----
Derived from: CZY03004.1
File Name - CZY03004.7
Author - delta
Sample ID - WZY/090218/CZY03004
Content - WZY/090218/CZY03004
Creation Date - 18-FEB-2009 20:52:18
Revision Date - 18-FEB-2009 21:14:17
Spec Site - ECA400
Spec Type - DELTA2_NMR
Data Format - 1D COMPLEX
Dimensions - X
Dim Title - 1H
Dim Size - 13107
Dim Units - [ppm]
Field strength - 9.389766[T] (400[MHz]
X acq_duration - 2.183659552[s]
X domain - 1H
X freq - 399.78219838[MHz]
X offset - 5[ppm]
X points - 16384
X prescans - 1
X resolution - 0.457946[Hz]
X sweep - 7.50300012[kHz]
Irr domain - 1H
Irr freq - 399.78219838[MHz]
Irr offset - 5[ppm]
Tri domain - 1H
Tri freq - 399.78219838[MHz]
Tri offset - 5[ppm]
Mod return - 1
Scans - 8
Total scans - 8
X 90 width - 10.55012[us]
X acq_time - 2.183659552[s]
X angle - 45[deg]
X atn - 4.50000[dB]
X pulse - 5.27506[us]
Irr mode - off
Tri mode - off
Dante preset - FALSE
Initial wait - 1[s]
Relaxation_delay - 5[s]
Repetition_time - 7.183659552[s]

3p


(Millions)
X : parts per Million : 13C
162.1810
159.7592
152.1220
140.5280
140.5089
139.3552
128.6288
128.5430
127.6468
126.1594
116.4150
116.1862
114.0791
108.6539
108.4346
105.0498
94.7239
77.3232
76.9991
76.6844
55.2984
11.0198
7.5206
OMe
F
N
N
3p