Palladium-Catalyzed Decarboxylative Cross-Coupling Reaction of Acrylic Acid with Aryl Iodide

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Supporting Information

General. All reactions were performed in test 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. Solvents were re-distilled prior to use in the reactions. NMR spectra were obtained on JEOL ECA-400 systems using $CDCl_3$ as solvent, with proton and carbon resonances at 400 and 100 MHz, respectively.

General experimental procedure:
A mixture of acrylic acid **1** (0.30 mmol), aryl iodide **2** (0.36 mmol, 1.2 equiv), Ag_2CO_3 (0.90 mmol, 247 mg, 3.0 equiv), CyJohnPhos (0.06 mmol, 21 mg, 0.2 equiv), and $PdCl_2$ (0.03 mmol, 5.3 mg, 0.1 equiv) in DMA (2.0 mL) was stirred at 150 °C under nitrogen for 12 h. After completion of reaction as indicated by TLC, the mixture was cooled and filtered with Celite. After adding ethyl acetate (10 mL) to the filtrate, the organic phase was washed with saturated NH_4Cl, dried with $MgSO_4$, and concentrated under reduced vacuum. The residue was then purified by flash chromatography on silica gel to afford product **3**.

(*E*)-1-(4-methoxystyryl)benzene (**3a**)[1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.49-7.44 (m, 4H), 7.34 (t, *J* = 7.8 Hz, 2H), 7.24-7.21 (m, 1H), 7.06 (d, *J* = 16.1 Hz, 1H), 6.97 (d, *J* = 16.1 Hz, 1H), 6.91-6.88 (m 2H), 3.82 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 159.3, 137.7, 130.2, 128.7, 128.2, 127.8, 127.2, 126.7, 126.3, 114.2, 55.4.

(*E*)-1,2-diphenylethene (**3b**) [2] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.51 (d, *J* = 7.8 Hz, 4H), 7.37-7.31 (m, 4H), 7.25 (t, *J* = 7.8 Hz, 2H), 7.10 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 137.4, 128.7, 127.7, 126.6.

(*E*)-1-fluoro-4-styrylbenzene (**3c**) [1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.50-7.44 (m, 4H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.27-7.24 (m, 1H), 7.09-6.99 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 163.8 ($^1J_{C\text{-}F}$ = 248.8 Hz), 137.2, 133.6 ($^3J_{C\text{-}F}$ = 3.8 Hz), 128.8, 128.6, 128.5, 128.1, 128.0, 127.7, 127.5, 126.5, 115.7 ($^2J_{C\text{-}F}$ = 21.9 Hz).

(*E*)-1-(3-methoxystyryl)benzene (**3d**) [1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.52 (d, *J* = 7.8 Hz, 2H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.30-7.25 (m, 2H), 7.14-7.06 (m, 4H), 6.85-6.82 (m, 1H), 3.85 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 160.0, 138.9, 137.3, 129.7, 129.1, 128.7, 128.6, 127.7, 126.6, 119.3, 113.4, 111.8, 55.3.

(*E*)-1-(4-styrylphenyl)ethanone (**3e**) [3] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.95-7.93 (m, 2H), 7.58 (d, *J* = 8.3 Hz, 2H), 7.54-7.52 (m, 2H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.31-7.28 (m, 1H), 7.22 (d, *J* = 16.6 Hz, 1H), 7.12 (d, *J* = 16.1 Hz, 1H), 2.60 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 197.6, 142.1, 136.7, 131.5, 128.9, 128.8,

128.4, 127.5, 126.9, 126.5, 26.7.

(*E*)-1-(3-(trifluoromethyl)styryl)benzene (**3f**) [1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.74 (s, 1H), 7.67 (d, *J* = 7.3 Hz, 1H), 7.53-7.44 (m, 4H), 7.39-7.27 (m, 3H), 7.17 (d, *J* =16.6 Hz, 1H), 7.11 (d, *J* = 16.1 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 138.1, 136.7, 130.6, 129.6, 129.2, 128.8, 128.2, 127.2, 126.7, 124.1, 123.1.

(*E*)-1-methyl-4-styrylbenzene (**3g**) [4] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.50 (d, *J* = 7.8 Hz, 2H), 7.41 (d, *J* = 8.3 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 2H), 7.26-7.24 (m, 1H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.07 (d, *J* = 2.4 Hz, 2H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 137.6, 134.6, 129.4, 128.7, 127.7, 127.4, 126.5, 126.4, 21.3.

(*E*)-methyl 2-styrylbenzoate (**3h**) [5] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.98 (d, *J* = 16.1 Hz, 1H), 7.92 (d, *J* = 7.8 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.55-7.50 (m, 3H), 7.38-7.25 (m, 4H), 7.00 (d, *J* = 16.1 Hz, 1H), 3.92 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 168.0, 139.4, 137.5, 132.2, 131.5, 130.7, 128.7, 128.6, 127.9, 127.5, 127.2, 127.0, 126.9, 52.2.

(*E*)-1-(4-methoxystyryl)-4-nitrobenzene (**3i**) [6] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 8.20 (d, *J* = 8.8 Hz, 2H), 7.59 (d, *J* = 8.8 Hz, 2H), 7.49 (d, *J* = 8.8 Hz, 2H), 7.22 (d, *J* = 16.6 Hz, 1H), 7.00 (d, *J* = 16.6 Hz, 1H), 6.94-6.91 (m, 2H), 3.84 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 160.3, 146.5, 144.3, 133.0, 129.0, 128.5, 126.5, 124.2, 124.1, 114.4, 55.4.

(*E*)-1-(4-methoxystyryl)-3-nitrobenzene (**3j**) [7] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 8.33-8.32 (m, 1H), 8.05-8.04 (m, 1H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.51-7.47 (m, 3H), 7.18 (d, *J* = 16.6 Hz, 1H), 6.99 (d, *J* = 16.1 Hz, 1H), 6.93-6.91 (m, 2H), 3.84 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 160.0, 148.8, 139.6, 132.0, 131.3, 129.5, 129.1, 128.2, 124.0, 121.6, 120.6, 114.3, 55.4.

(*E*)-1-(4-methoxystyryl)-4-chlorobenzene (**3k**) [8] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.45-7.39 (m, 4H), 7.31-7.29 (m, 2H), 7.03 (d, *J* = 16.6 Hz, 1H), 6.93-6.88 (m, 3H), 3.83 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 159.5, 136.2, 132.7, 129.8, 128.9, 128.8, 127.8, 127.4, 125.3, 114.2, 55.4.

(*E*)-4-(4-methoxystyryl)benzonitrile (**3l**) [1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.61-7.59 (m, 2H), 7.54 (d, *J* = 8.3 Hz, 2H), 7.48-7.46 (m, 2H), 7.16 (d, *J* = 16.1 Hz, 1H), 6.96-6.90 (m, 3H), 3.84 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 160.1, 142.3, 132.5, 132.0, 129.1, 128.3, 126.6, 124.6, 119.2, 114.3, 110.1, 55.4.

(*E*)-1-(4-methoxystyryl)-4-fluorobenzene (**3m**) [1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.46-7.43 (m, 4H), 7.06-7.01 (m, 2H), 6.95 (d, *J* = 5.9 Hz, 2H), 6.91-6.89 (m, 2H), 3.83 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 162.1 ($^1J_{C\text{-}F}$ = 245 Hz), 159.4, 133.7 ($^3J_{C\text{-}F}$ = 3.8 Hz), 130.0, 128.1, 128.0, 127.8, 127.7, 125.4, 115.6 ($^2J_{C\text{-}F}$ =

20.9 Hz), 114.2, 55.4.

(*E*)-1-(4-fluorostyryl)-4-methylbenzene (**3n**)[1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.47-7.43 (m, 2H), 7.39 (d, *J* = 7.8 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.05-6.99 (m, 4H), 2.37 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 162.5 ($^1J_{C\text{-}F}$ = 246.9 Hz), 137.6, 134.4, 129.5, 128.4, 127.9, 127.8, 126.5, 126.4, 115.6 ($^2J_{C\text{-}F}$ = 20.9 Hz), 21.3.

(*E*)-2-(4-methoxystyryl)furan (**3o**)[10] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.40-7.37 (m, 3H), 6.98 (d, *J* = 16.1 Hz, 1H), 6.89-6.86 (m, 2H), 6.76 (d, *J* = 16.6 Hz, 1H), 6.40-6.39 (m, 1H), 6.29 (d, *J* = 2.9 Hz, 1H), 3.82 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 159.3, 153.6, 141.8, 129.9, 127.6, 126.8, 114.7, 114.2, 111.6, 107.7, 55.4.

(*E*)-1-(4-methoxystyryl)-4-methylbenzene (**3p**) [1] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.45-7.41 (m, 2H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.14 (d, *J* = 8.3 Hz, 2H), 7.01 (d, *J* = 16.1 Hz, 1H), 6.93 (d, *J* = 16.1 Hz, 1H), 6.89-6.86 (m, 2H), 3.81 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 159.1, 137.0, 134.8, 130.2, 129.3, 127.5, 127.1, 126.5, 126.1, 114.0, 55.2, 21.2.

(*E*)-methyl 2-(4-methylstyryl)benzoate (**3q**)[11] ^{1}H NMR (400 MHz, CDCl3) δ (ppm) 7.95-7.90 (m, 2H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.51-7.43 (m, 3H), 7.31-7.27 (m, 1H), 7.16 (d, *J* = 7.8 Hz, 2H), 6.98 (d, *J* = 16.6 Hz, 1H), 3.91 (s, 3H), 2.35 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 168.0, 139.4, 137.8, 134.7, 132.2, 131.5, 130.7, 129.4, 128.5, 127.0, 126.9, 126.8, 126.4, 52.2, 21.3.

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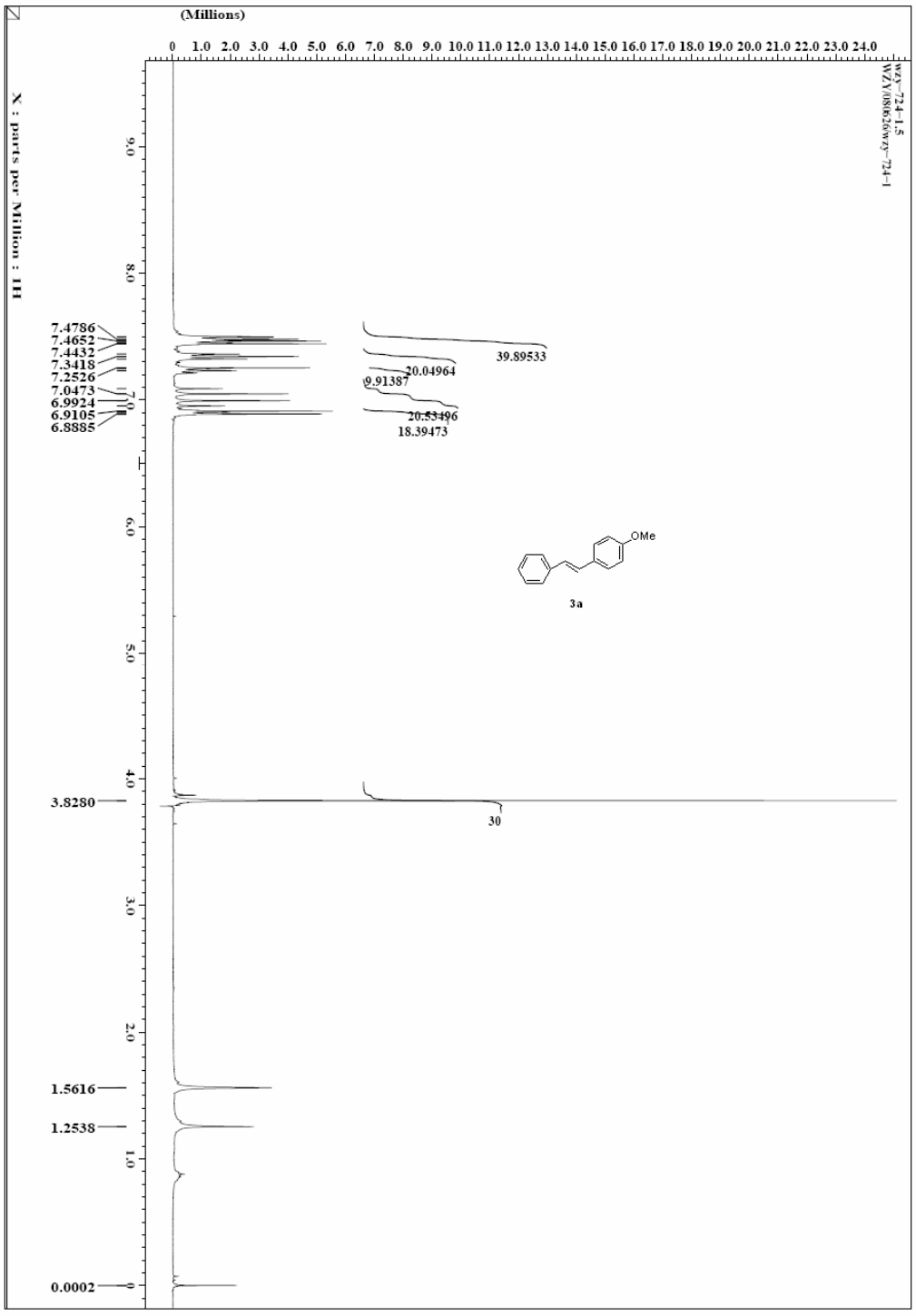
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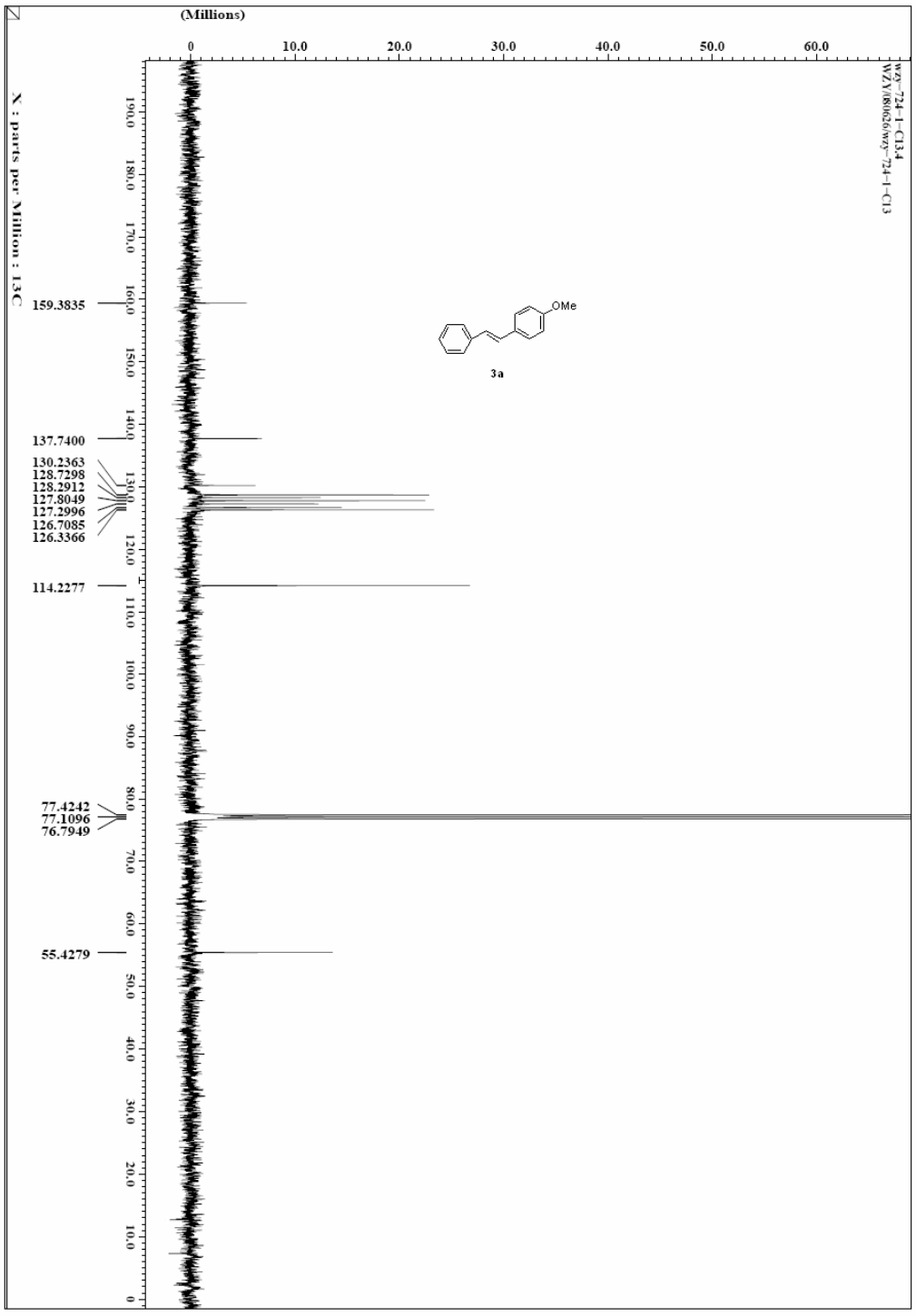
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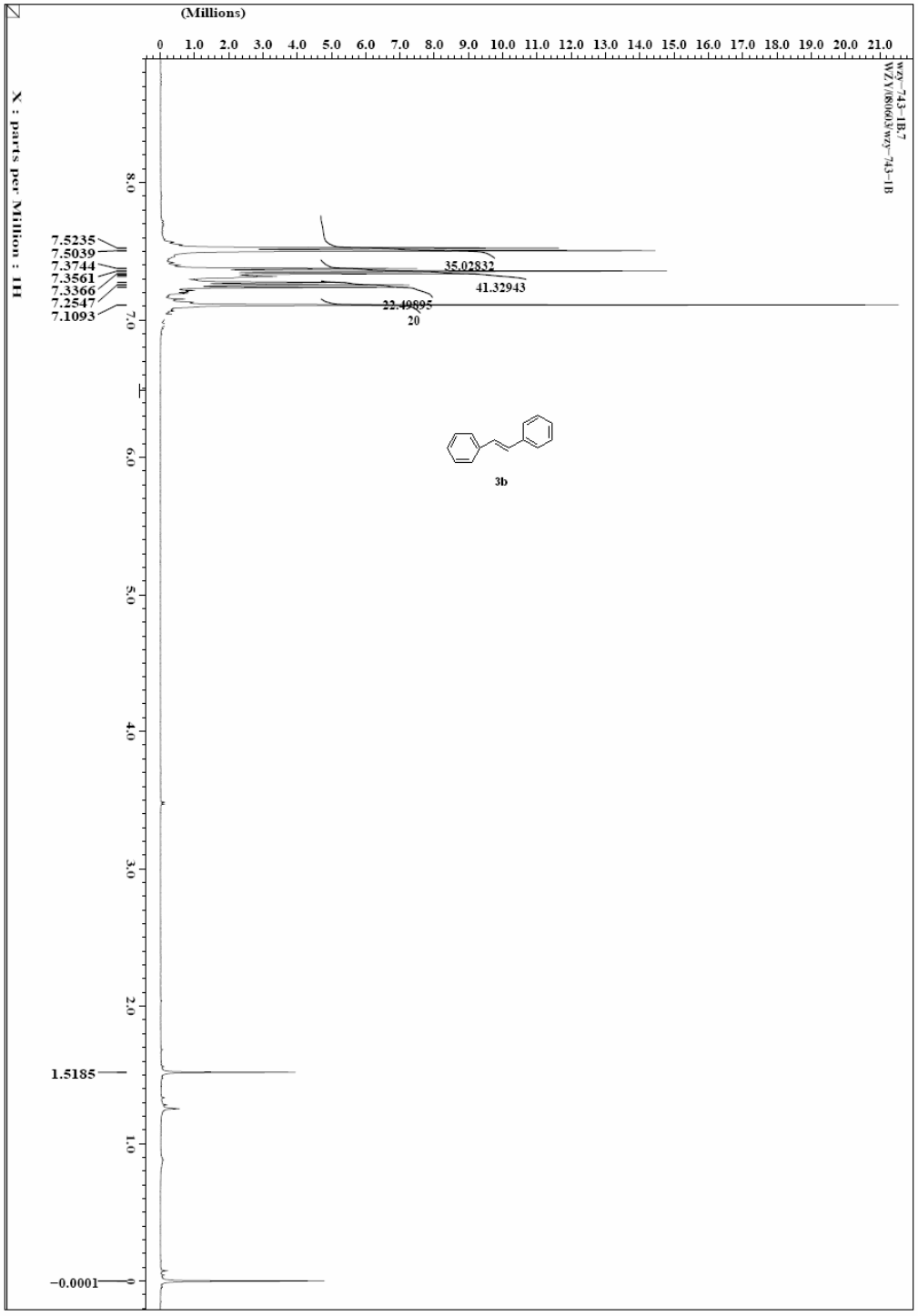
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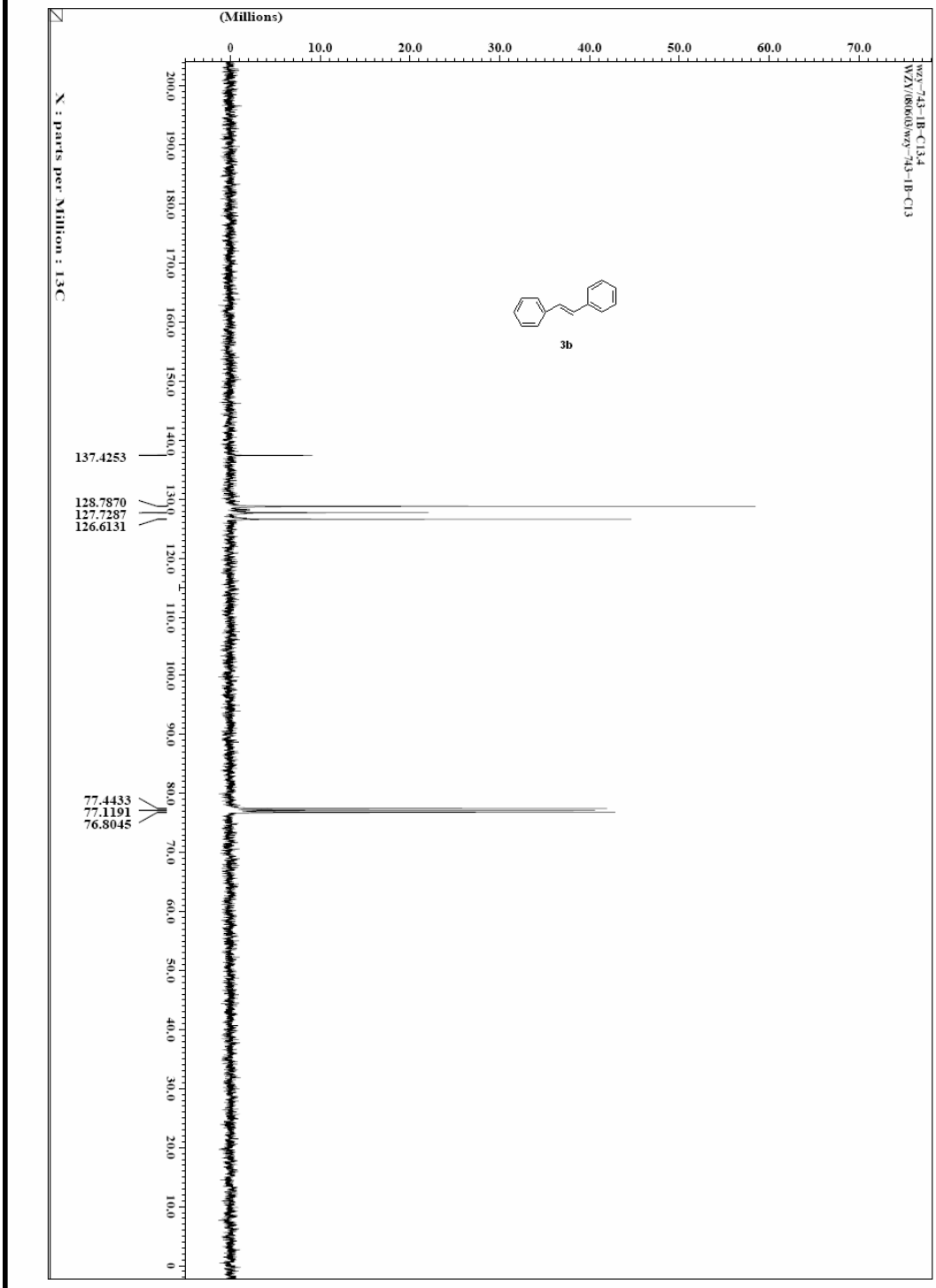
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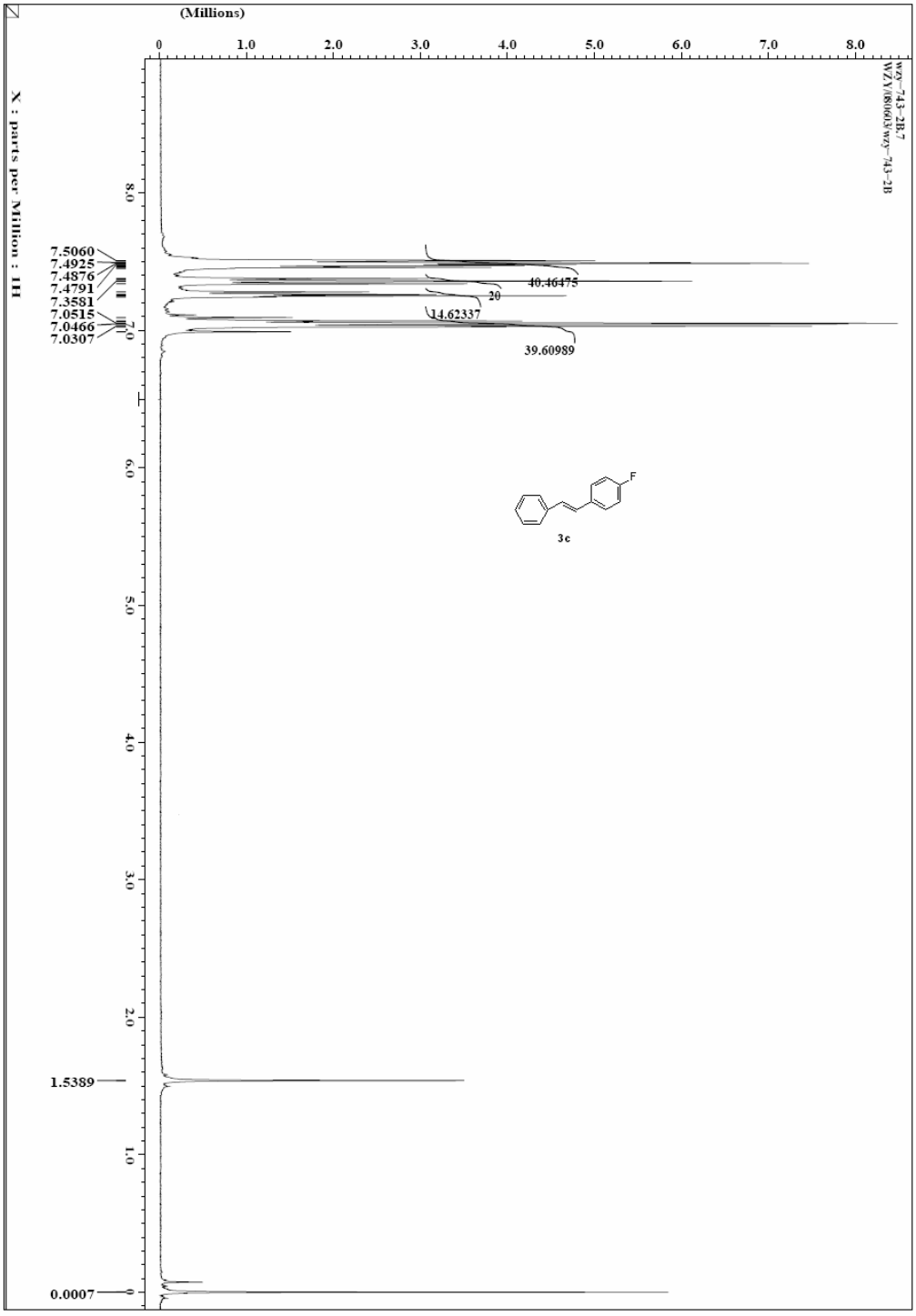
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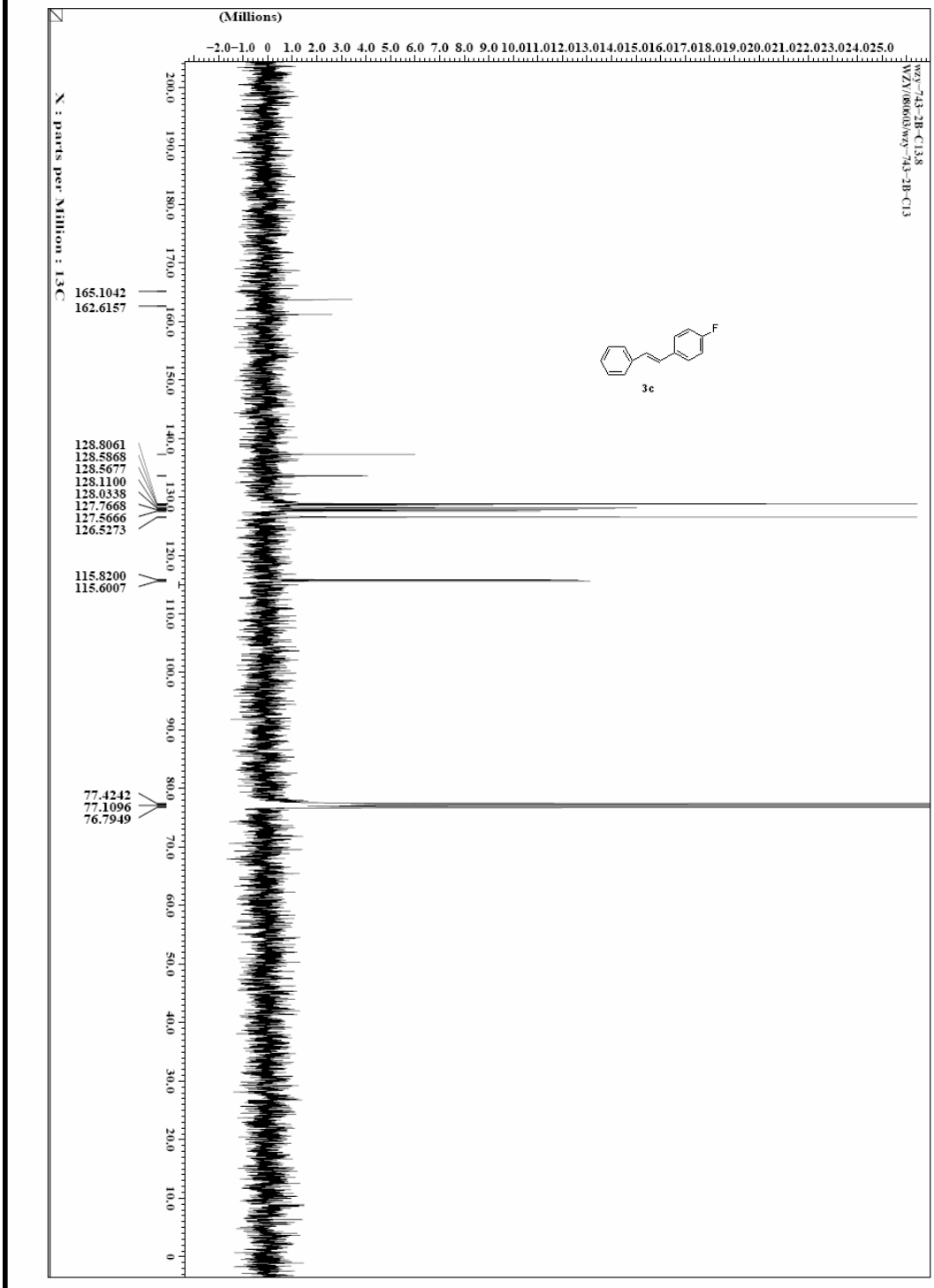
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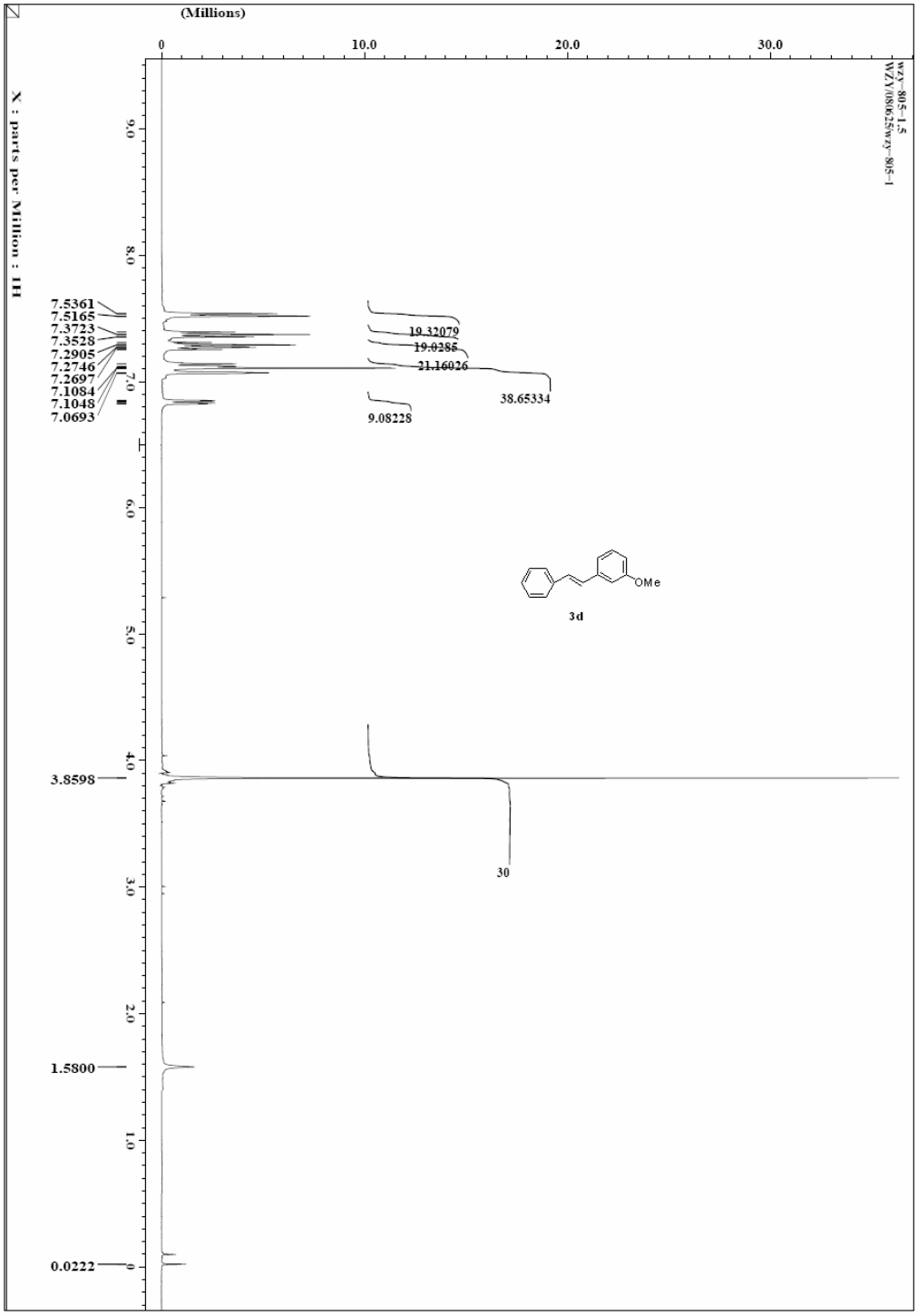
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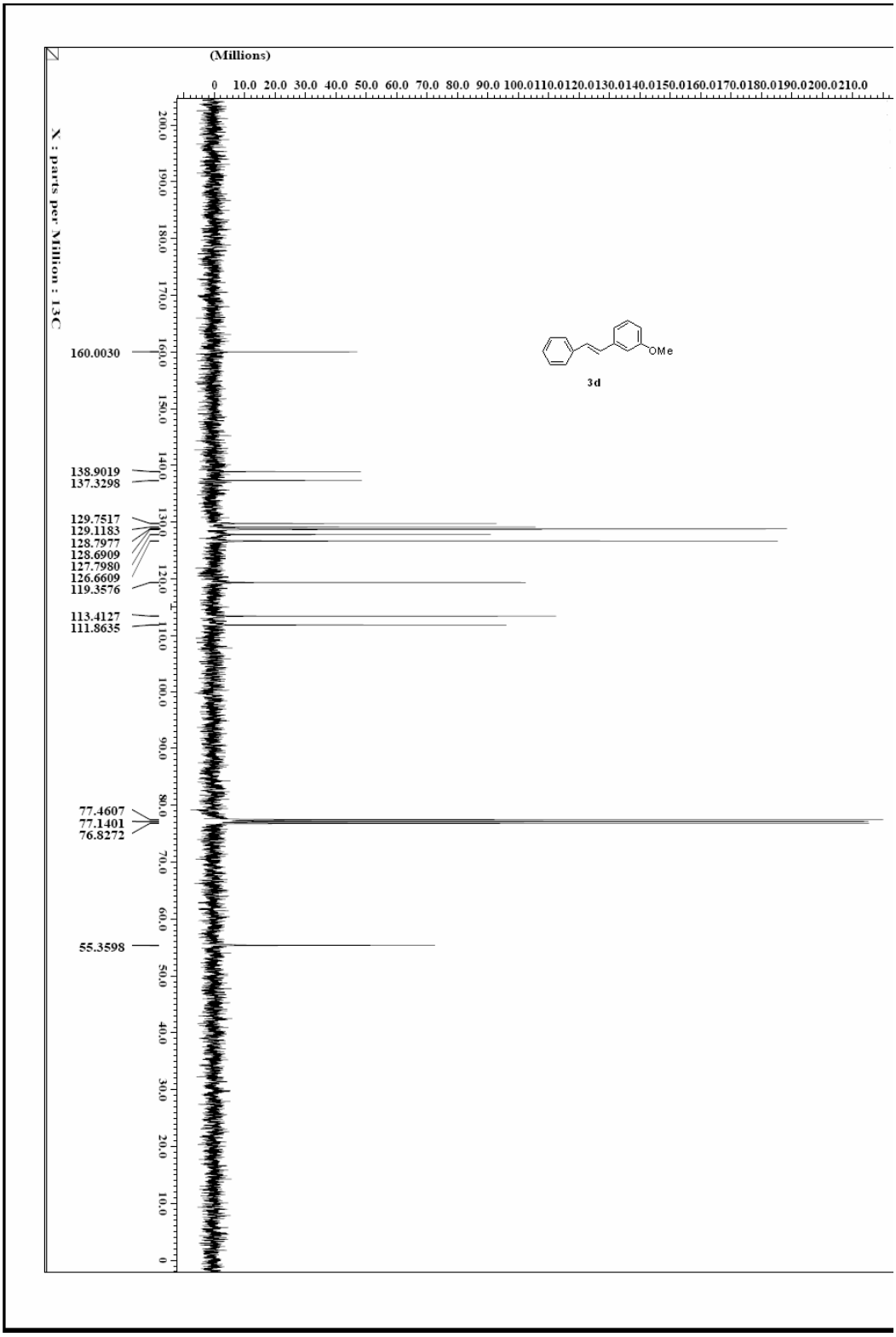
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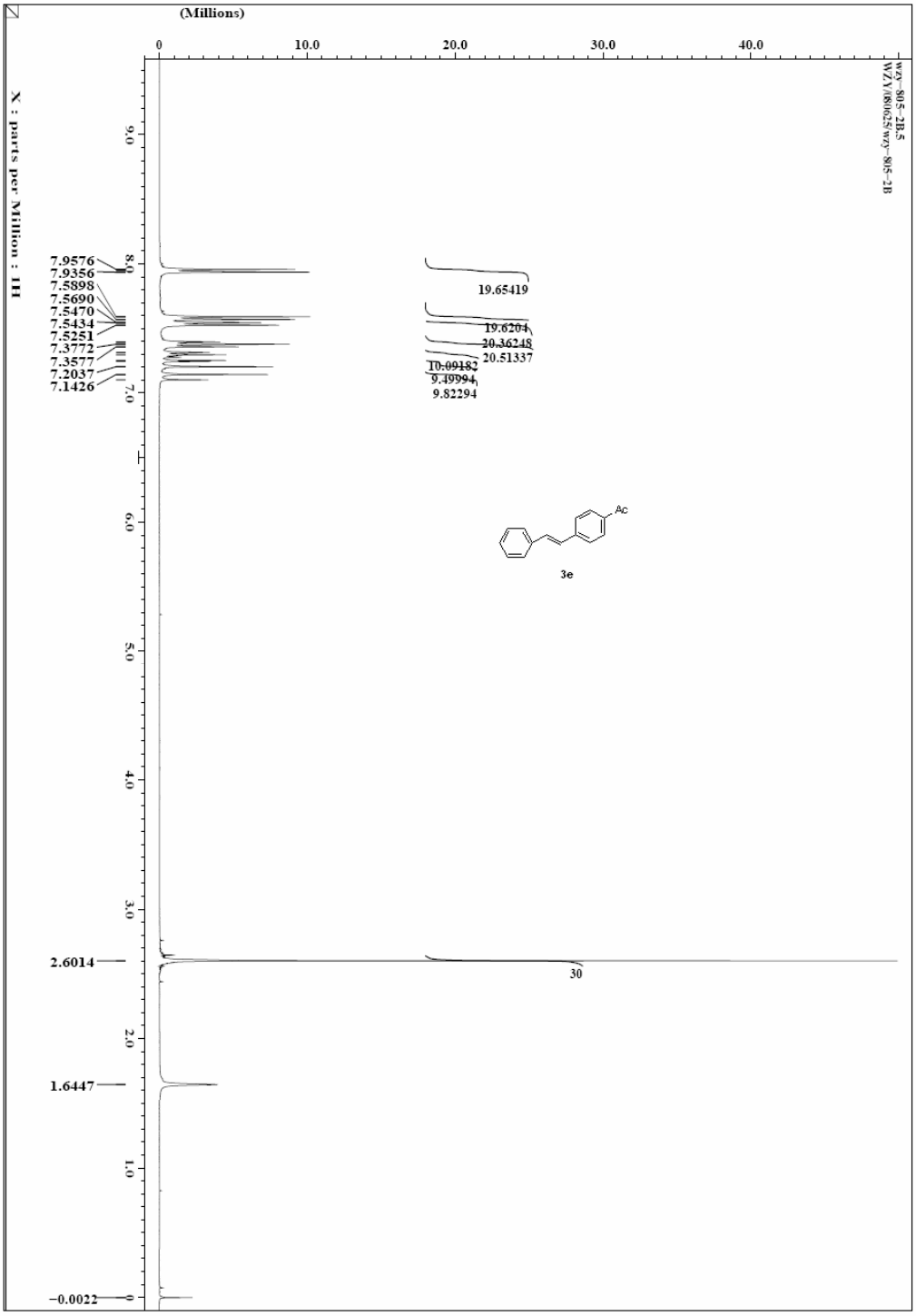
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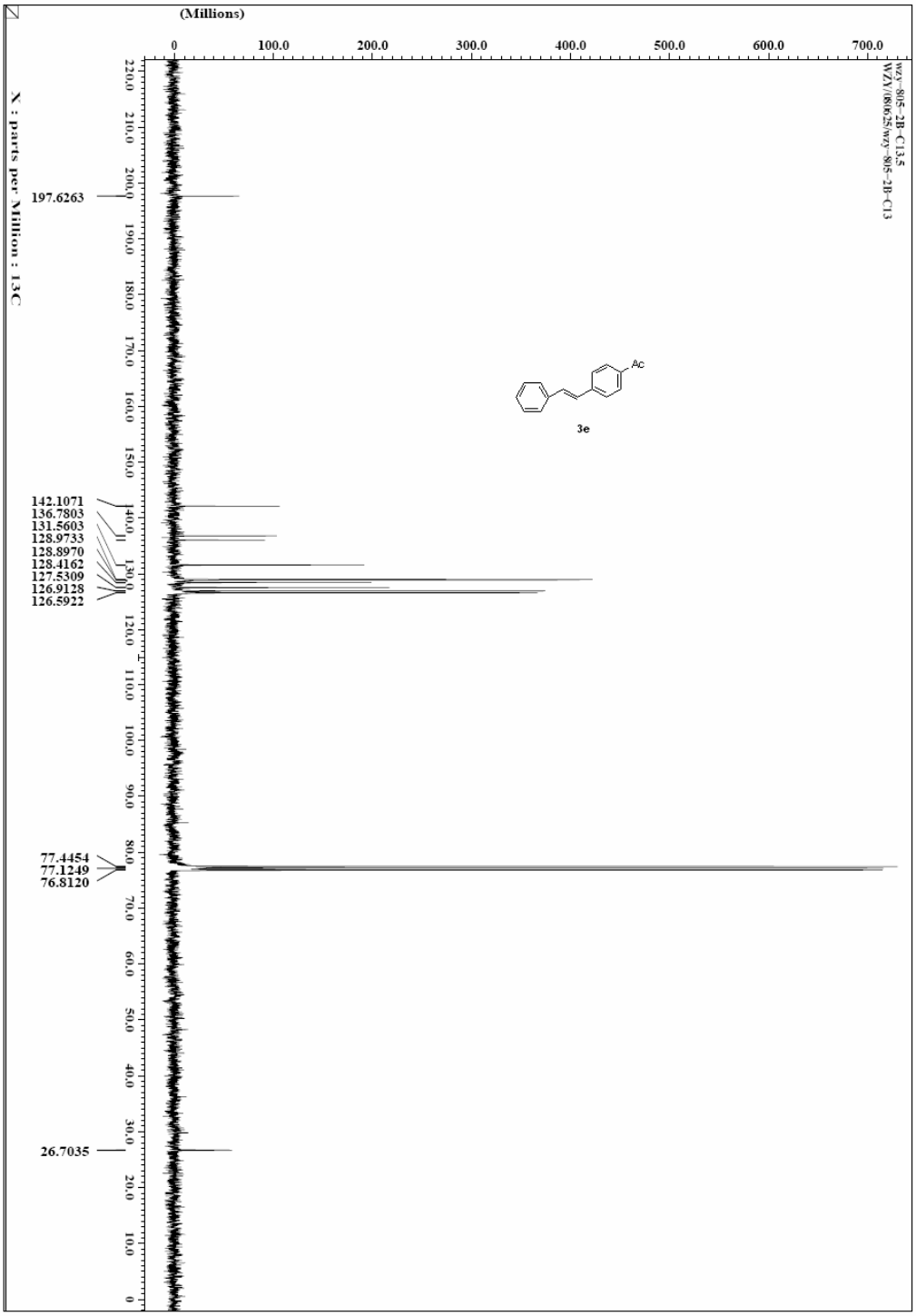
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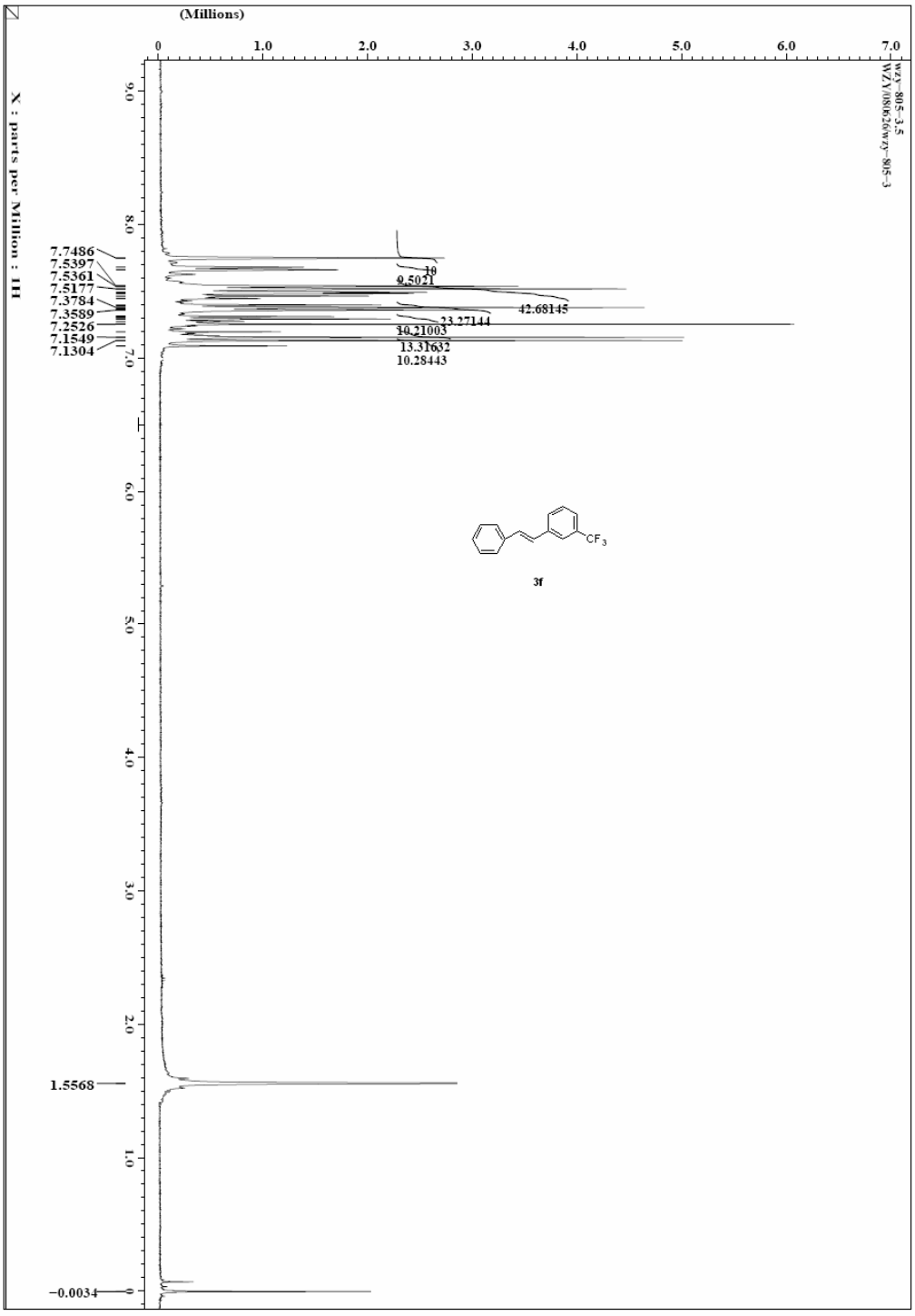
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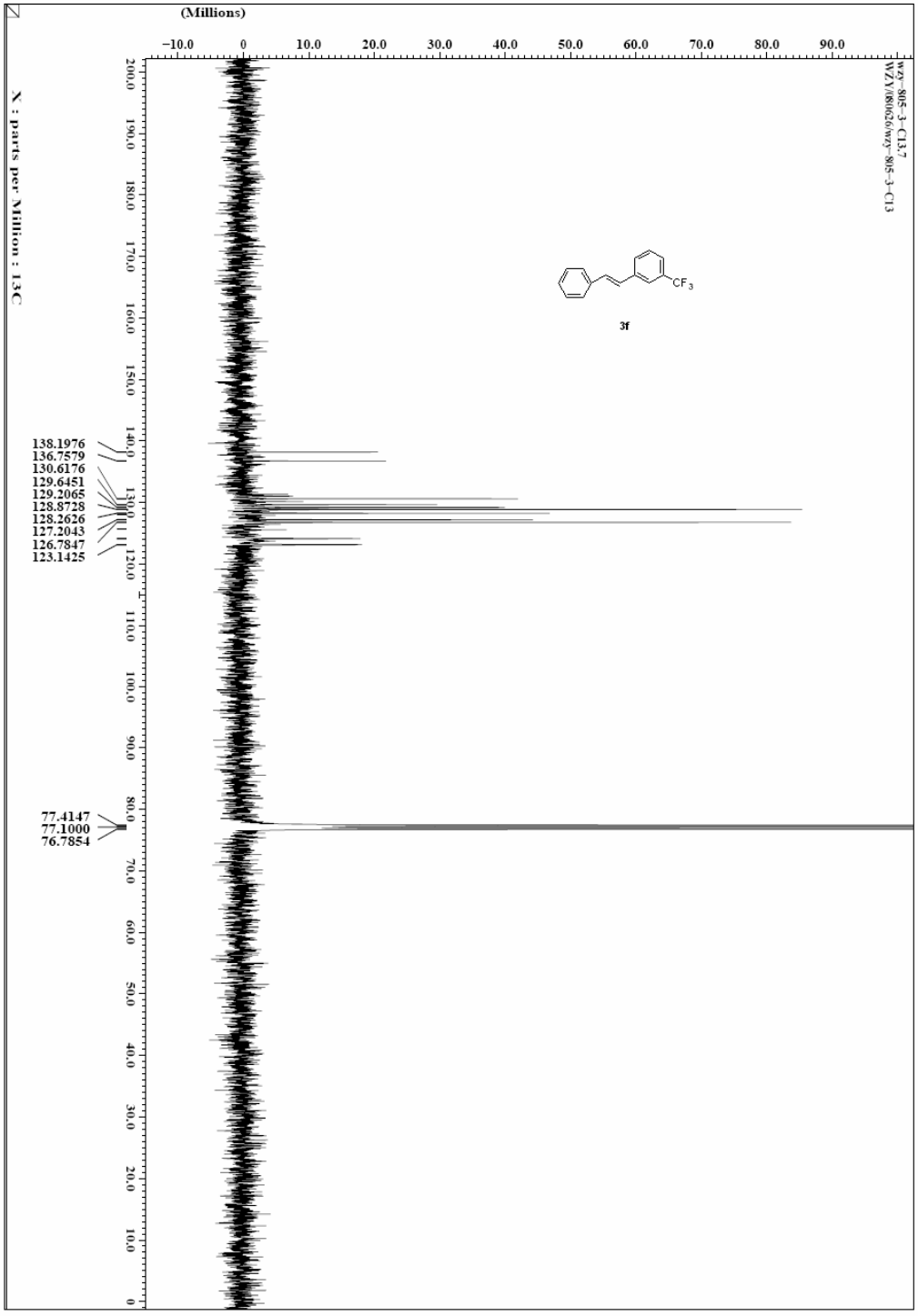
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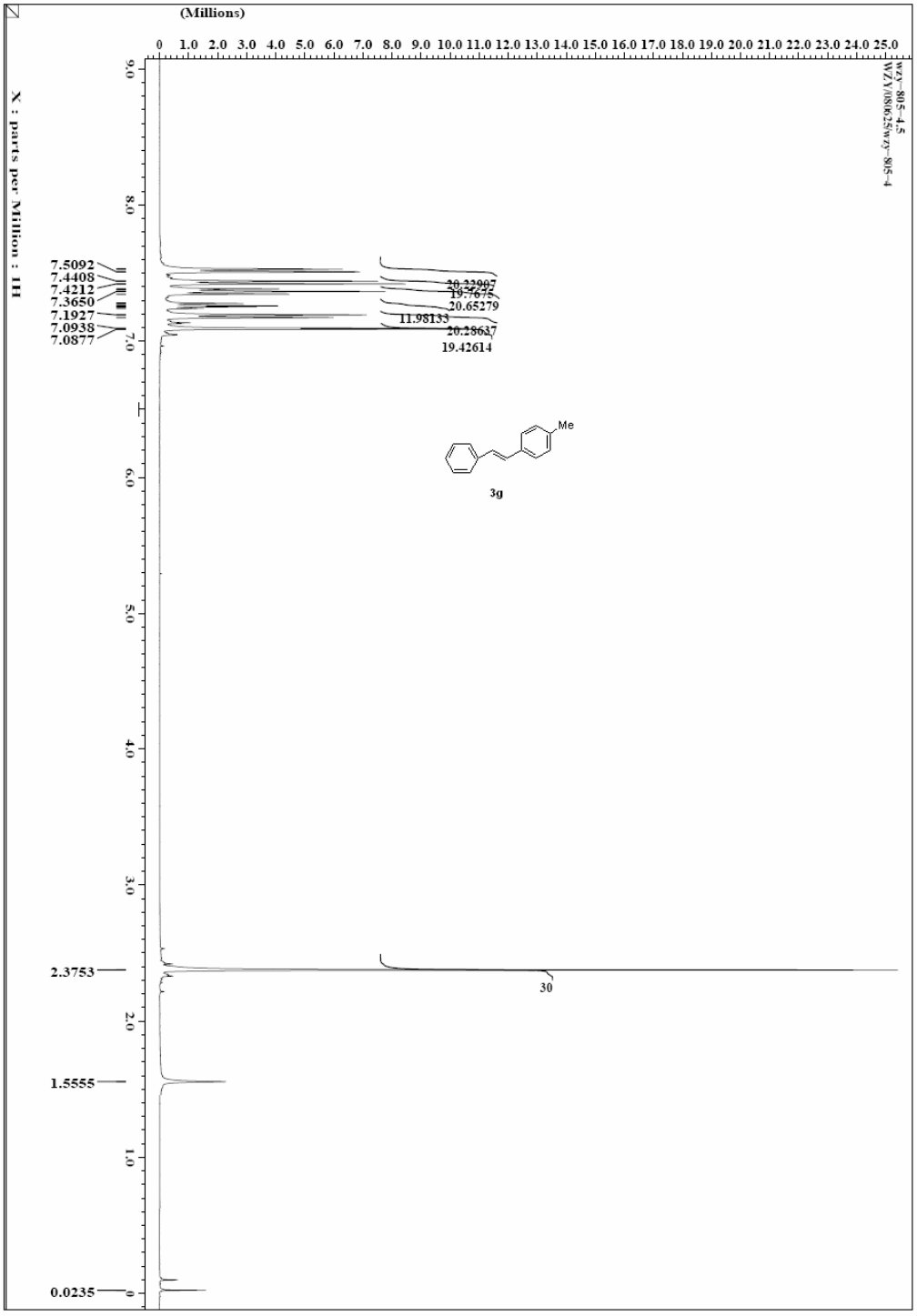
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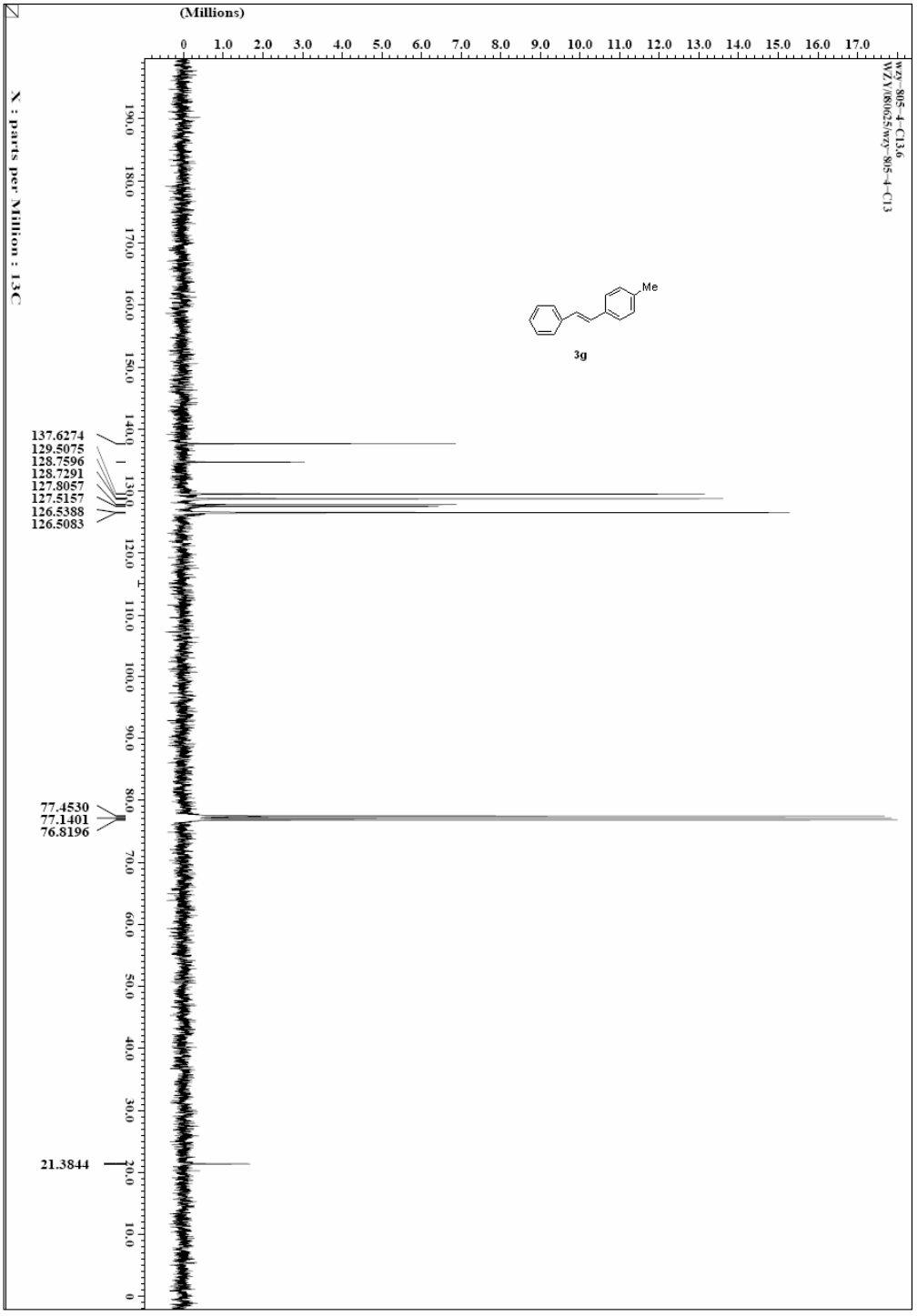
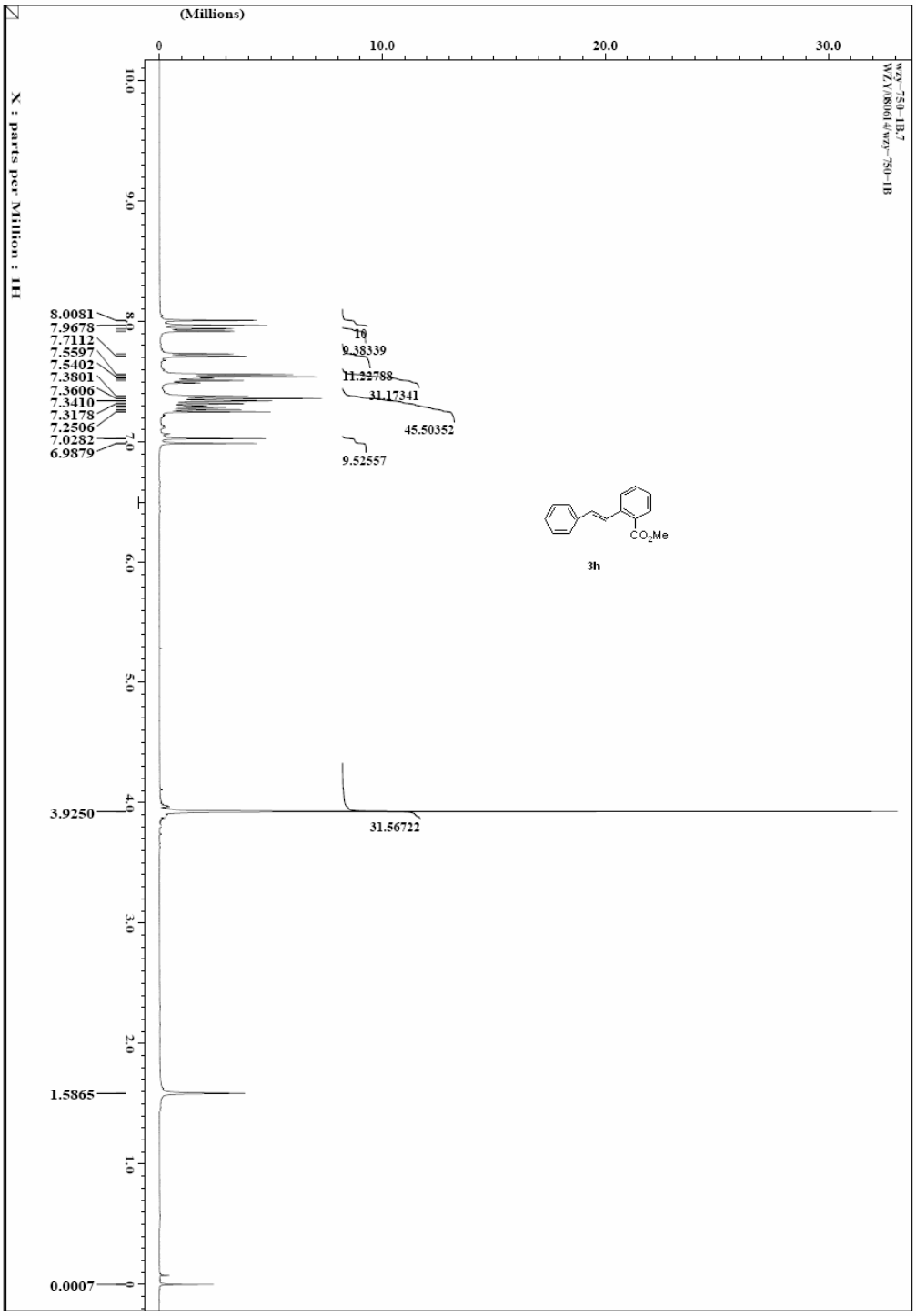
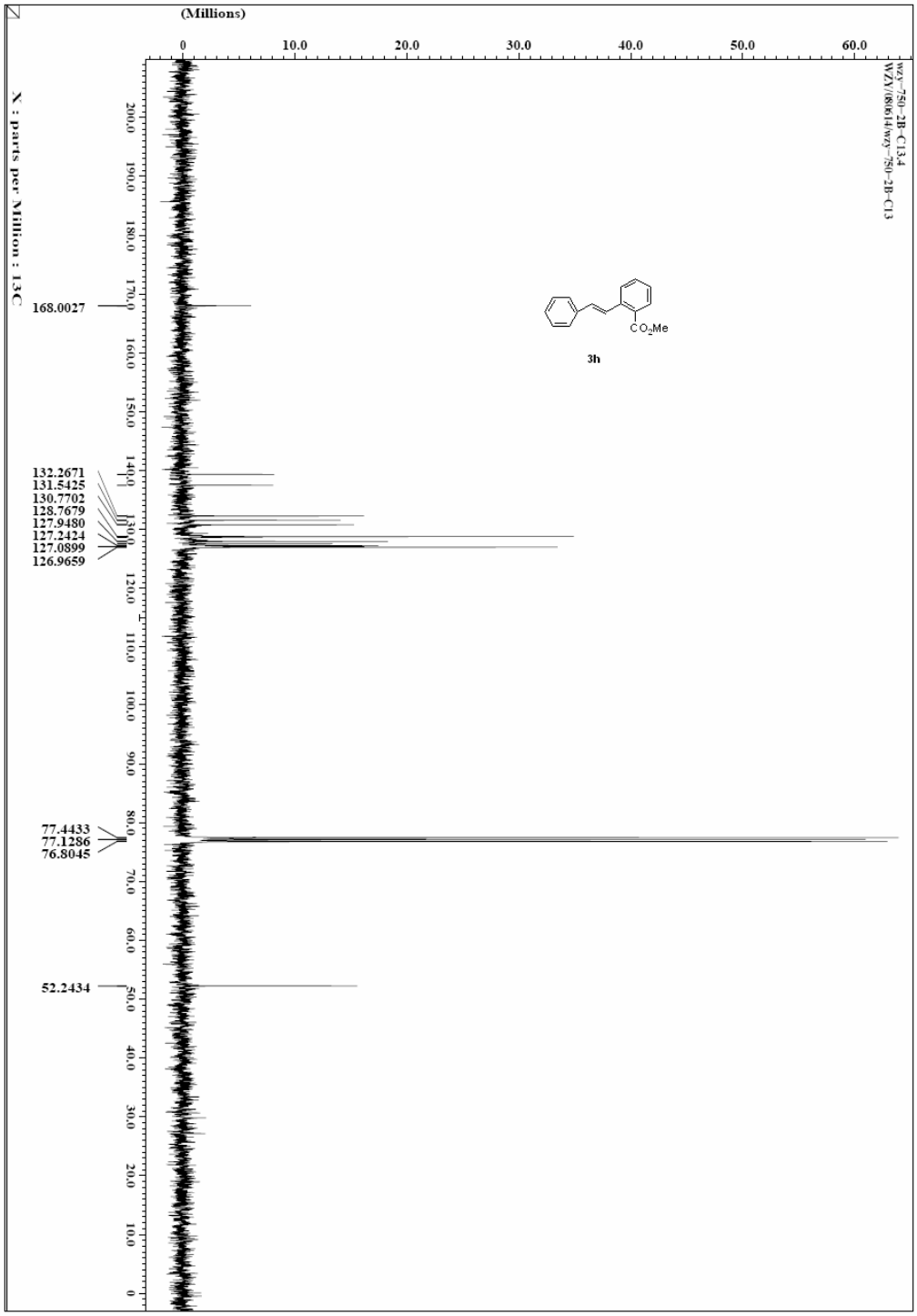
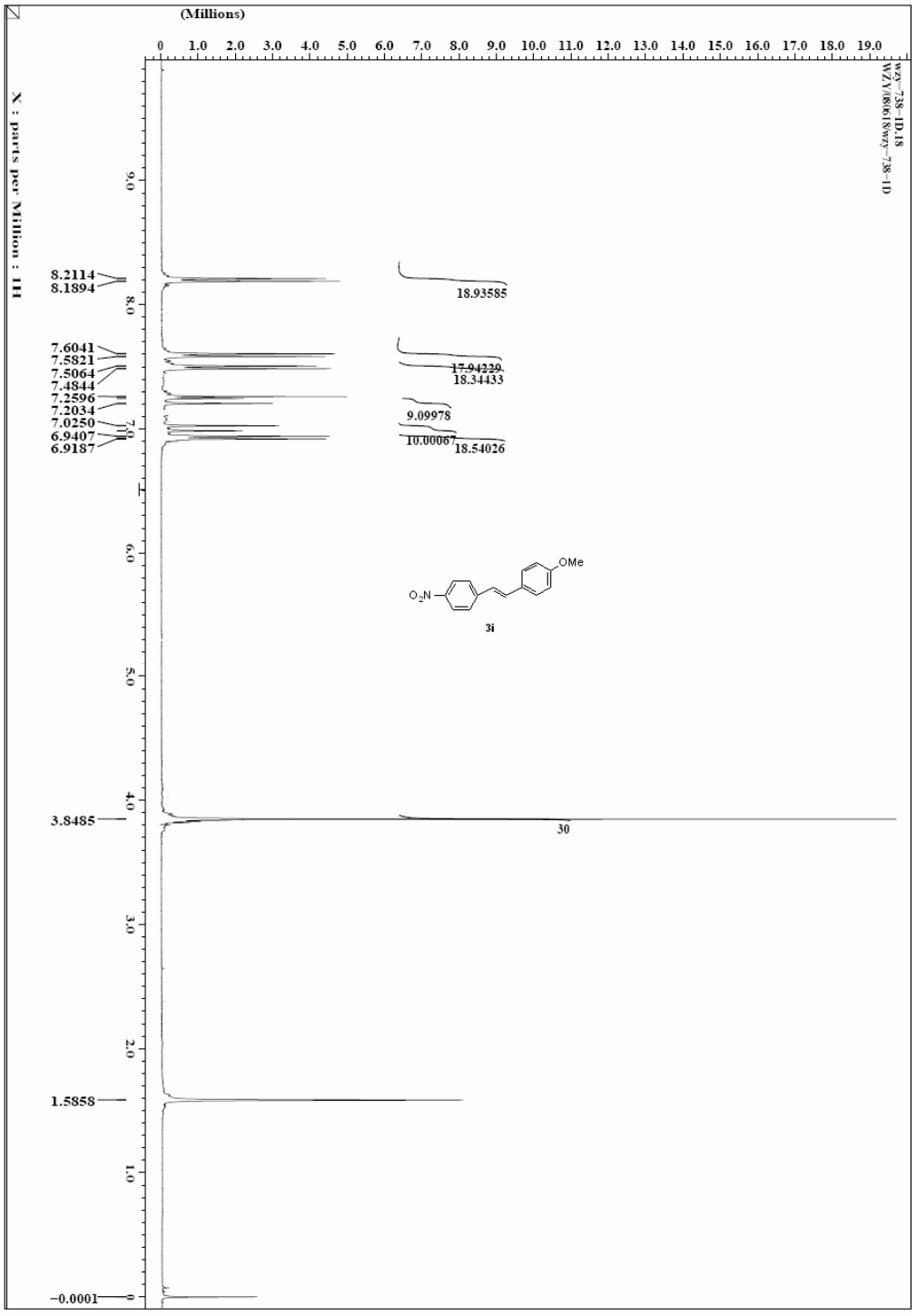
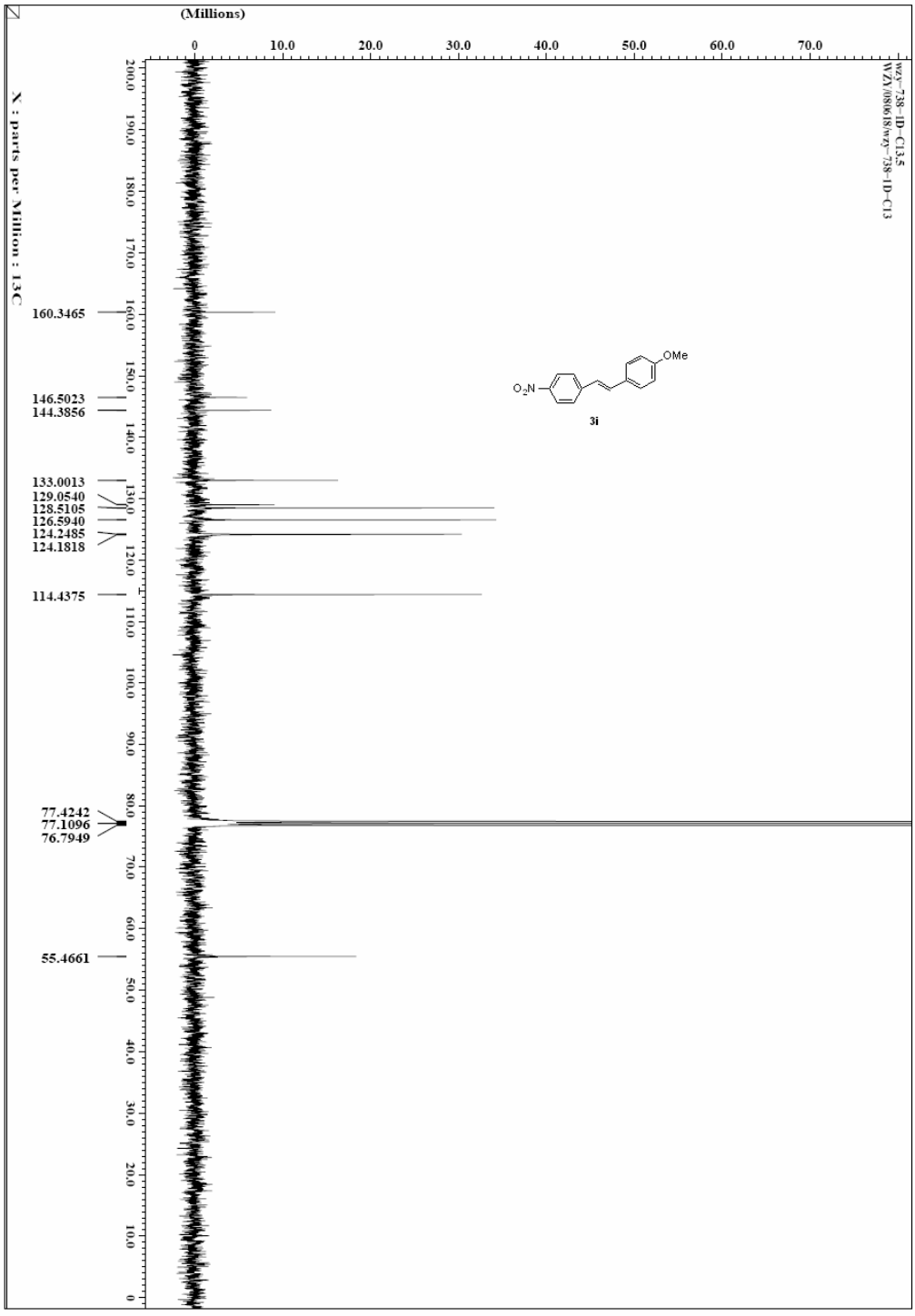
0.0235

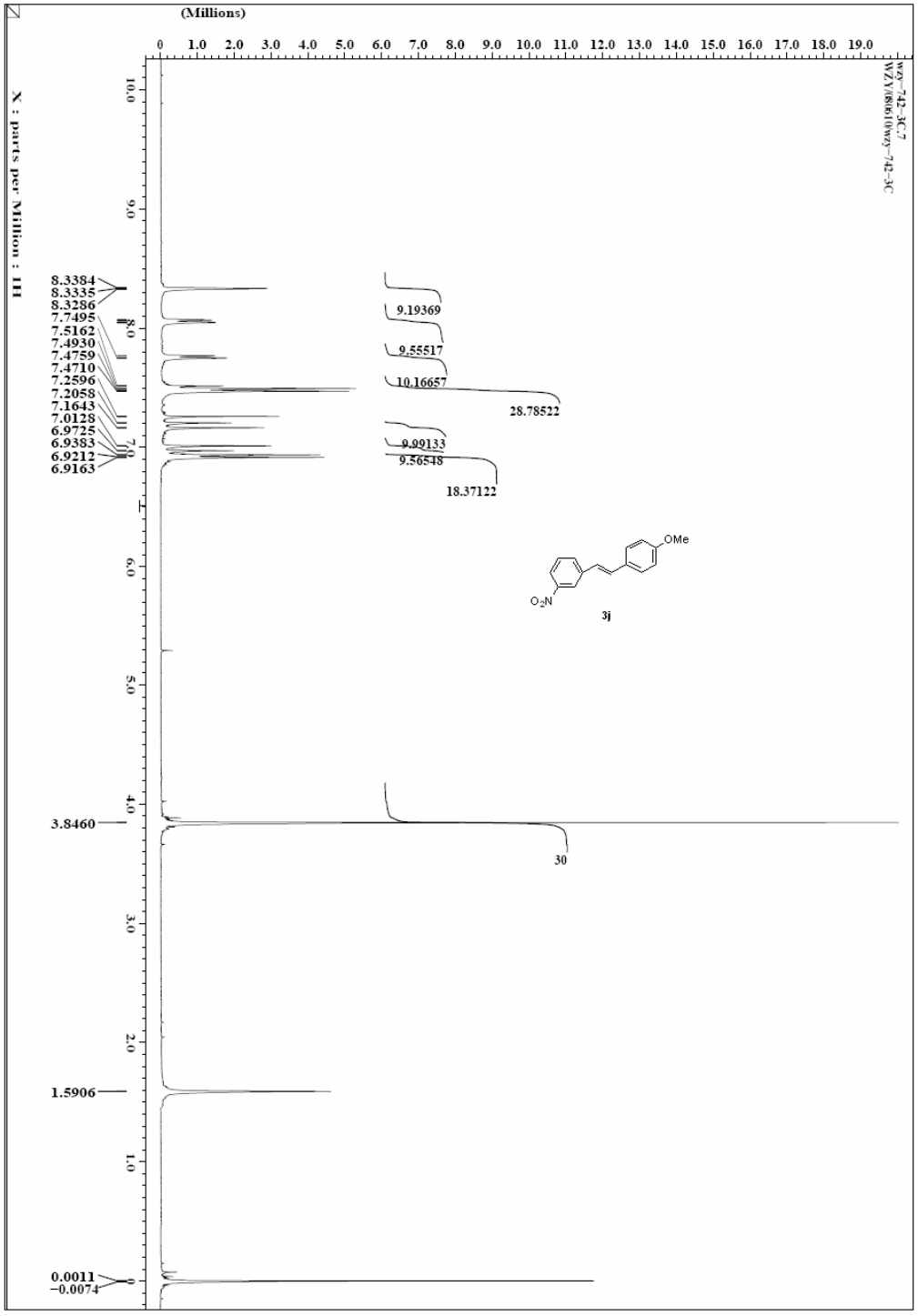

(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0
X : parts per Million : 13C
wzy-805-4-C13.6
WZY/080625/wzy-805-4-C13
Me
3g
137.6274
129.5075
128.7596
128.7291
127.8057
127.5157
126.5388
126.5083
77.4530
77.1401
76.8196
21.3844

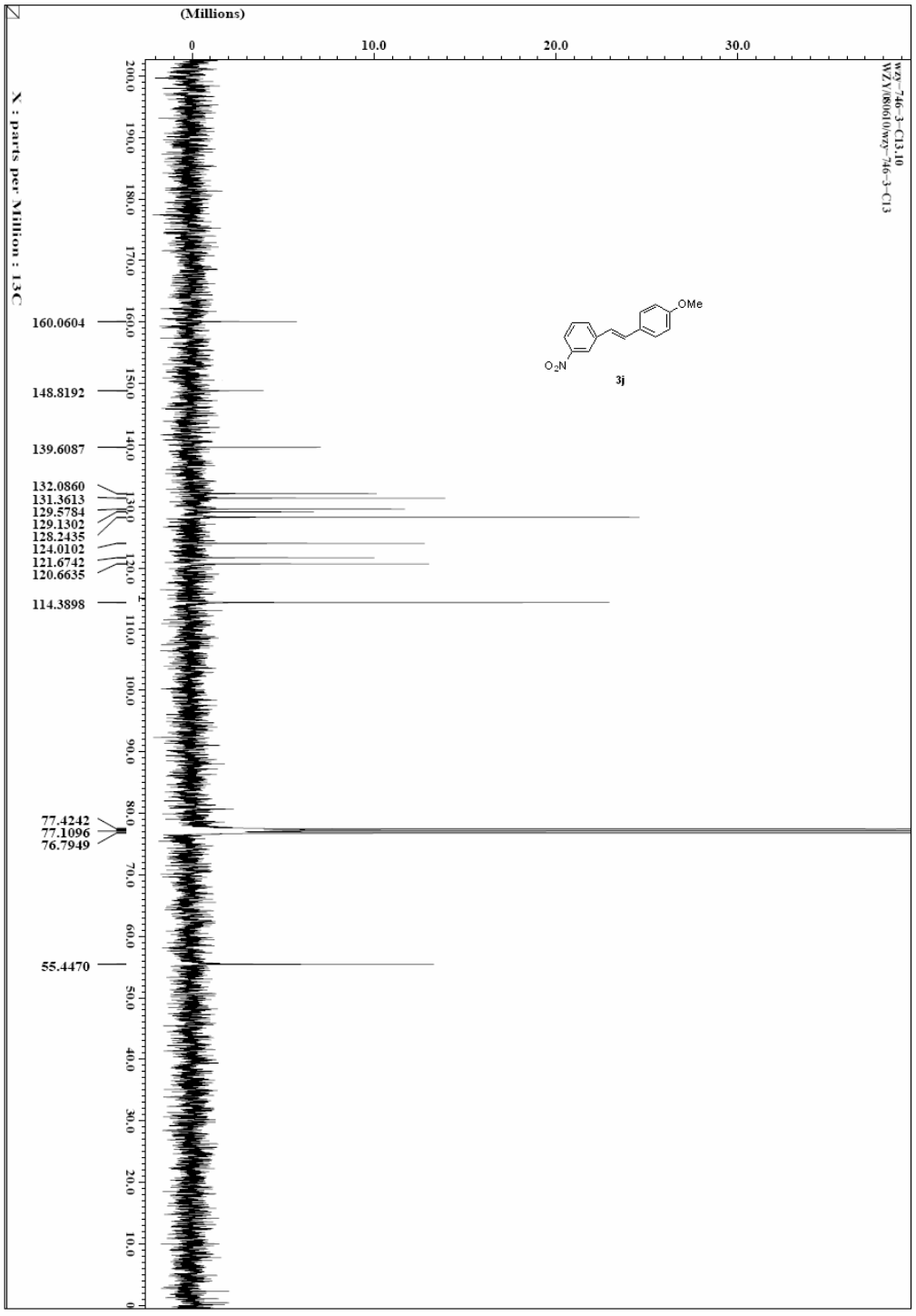

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
wzy-750-1B-7
WZY18060.4.wzy-750-1B
8.0081
7.9678
7.7112
7.5597
7.5402
7.3801
7.3606
7.3410
7.3178
7.2506
7.0282
6.9879
3.9250
1.5865
0.0007
10
9.38339
11.22788
31.17341
45.50352
9.52557
31.56722
CO2Me
3h

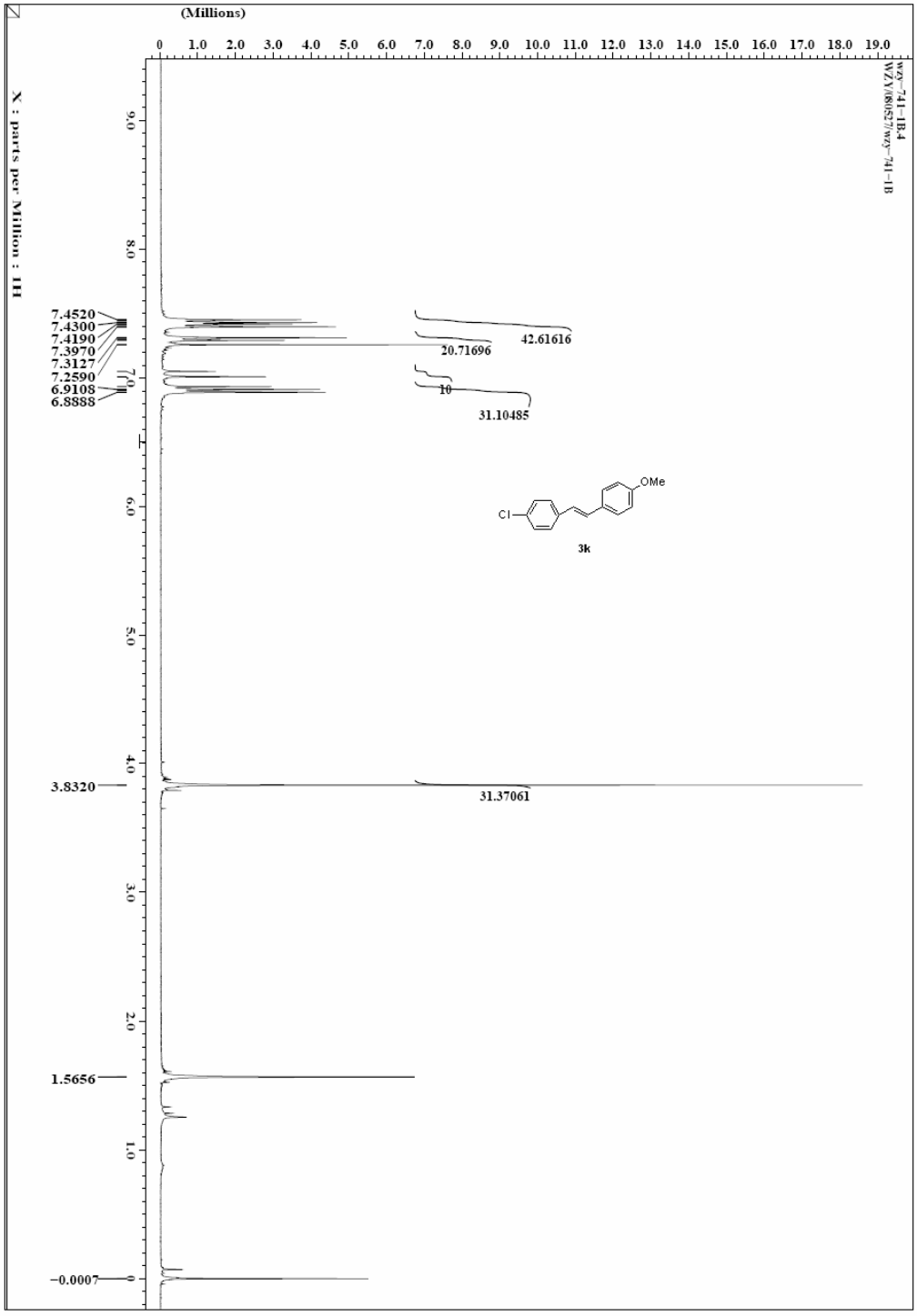

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
X : parts per Million : 13C
wzy-750-2B-C13-4
WZY/080414/wzy-750-2B-C13
168.0027
132.2671
131.5425
130.7702
128.7679
127.9480
127.2424
127.0899
126.9659
77.4433
77.1286
76.8045
52.2434
CO2Me
3h

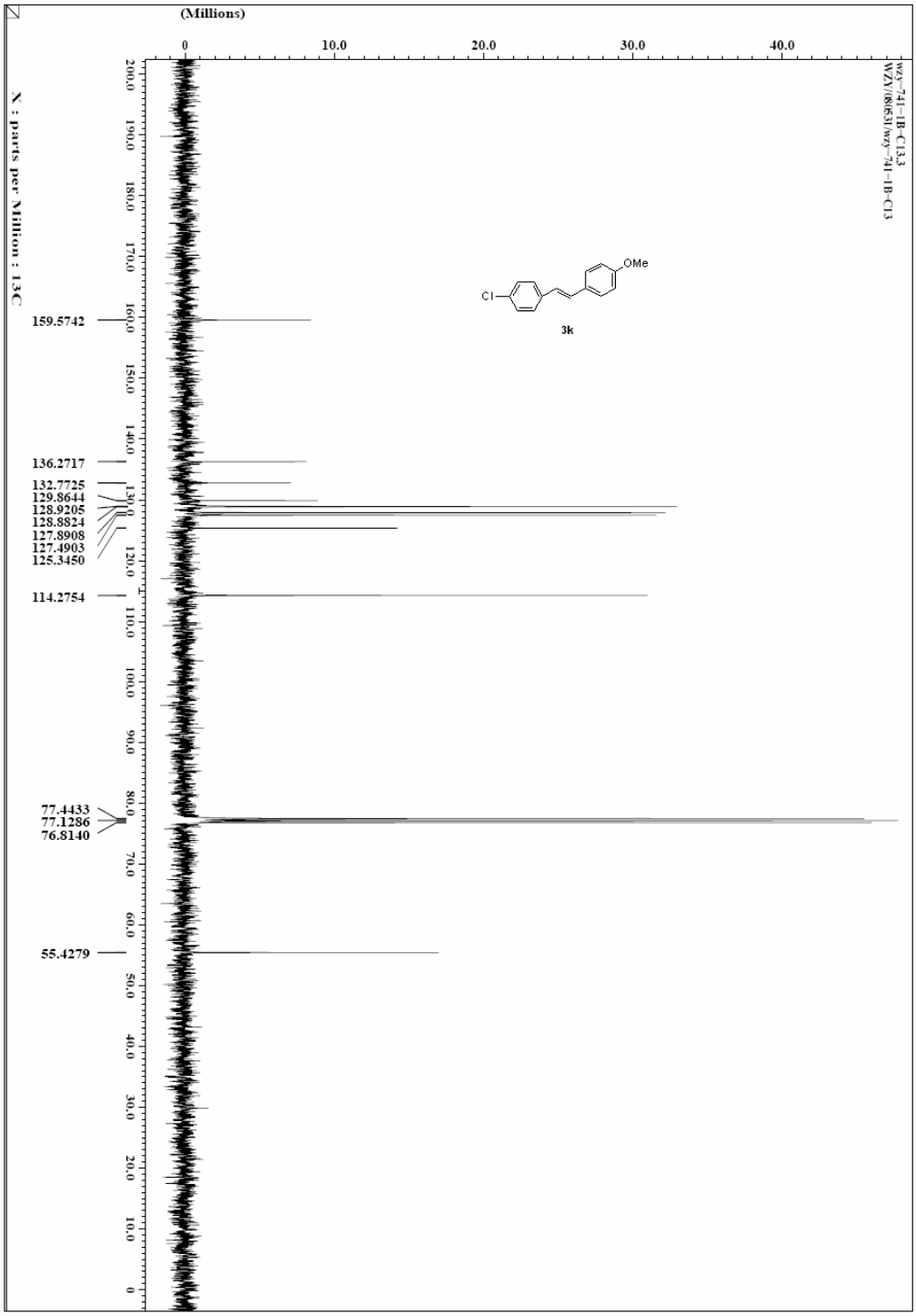

(Millions)
X : parts per Million : 1H
8.2114
8.1894
7.6041
7.5821
7.5064
7.4844
7.2596
7.2034
7.0250
6.9407
6.9187
3.8485
1.5858
−0.0001
18.93585
17.94239
18.34433
9.09978
10.00067
18.54026
30
OMe
O2N
3i

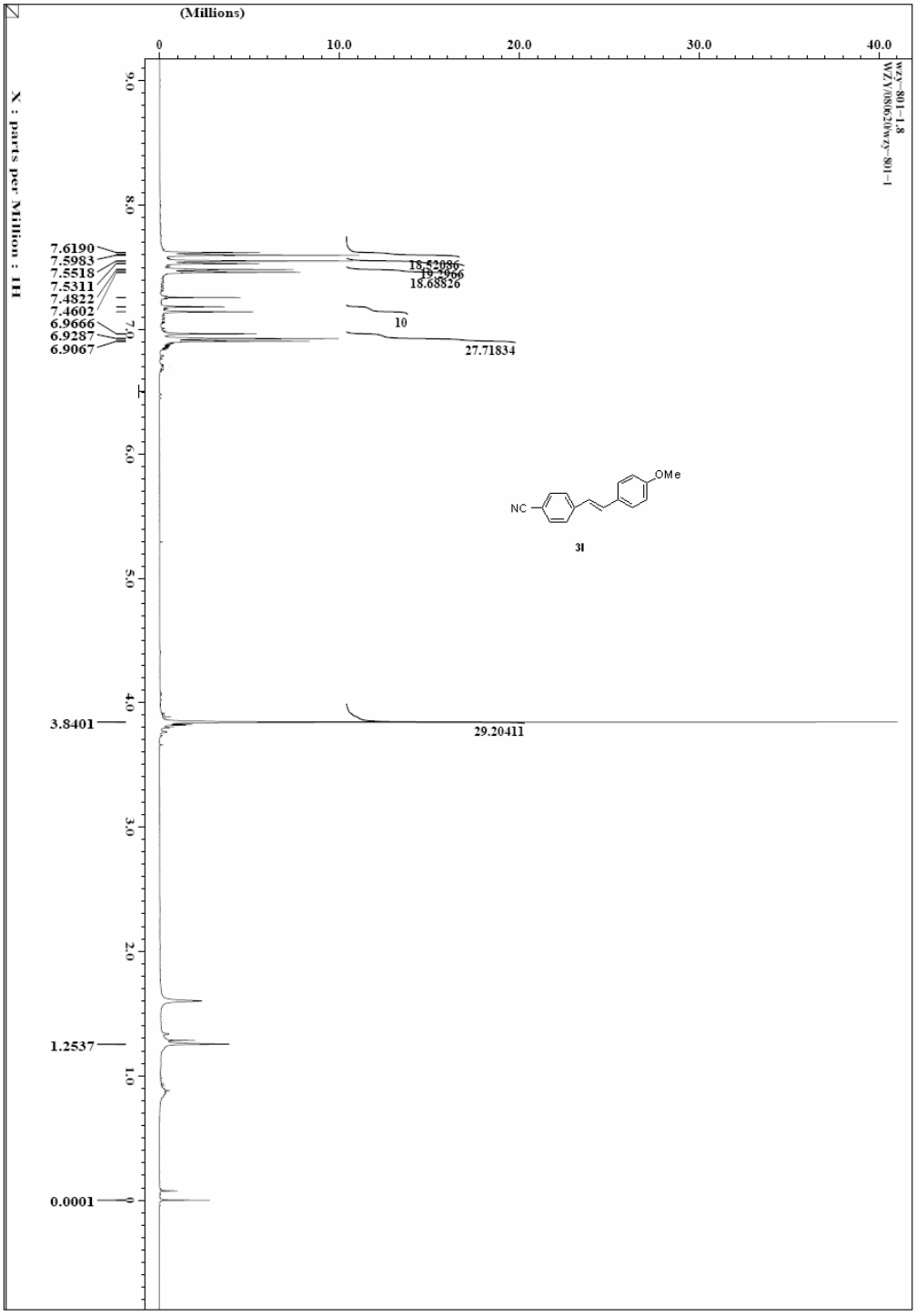

(Millions)
0
10.0
20.0
30.0
40.0
50.0
60.0
70.0
X : parts per Million : 13C
160.3465
146.5023
144.3856
133.0013
129.0540
128.5105
126.5940
124.2485
124.1818
114.4375
77.4242
77.1096
76.7949
55.4661
OMe
O2N
3i

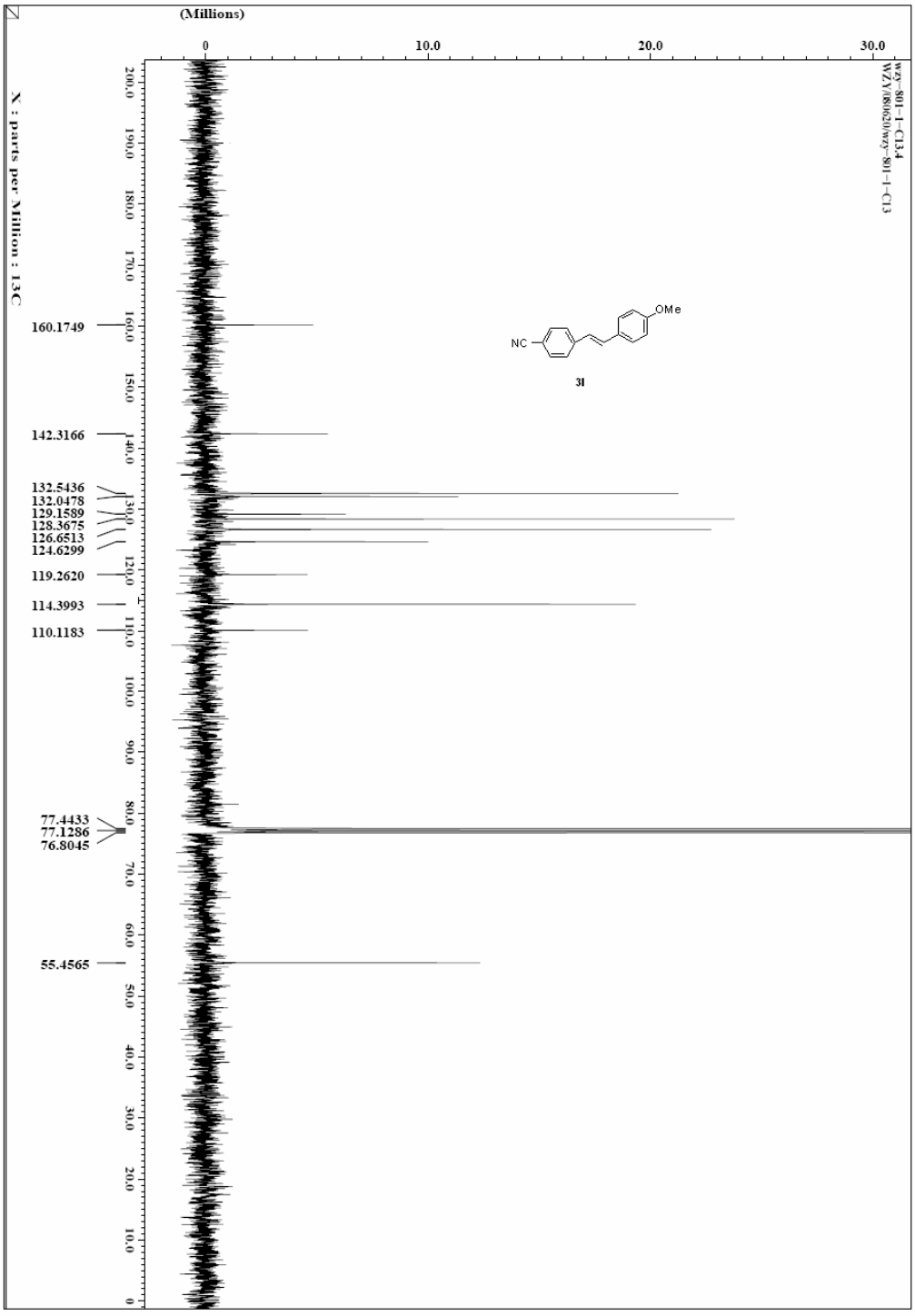

(Millions)
X : parts per Million : 1H
8.3384
8.3335
8.3286
7.7495
7.5162
7.4930
7.4759
7.4710
7.2596
7.2058
7.1643
7.0128
6.9725
6.9383
6.9212
6.9163
3.8460
1.5906
0.0011
-0.0074
9.19369
9.55517
10.16657
28.78522
9.99133
9.56548
18.37122
30
OMe
O2N
3j

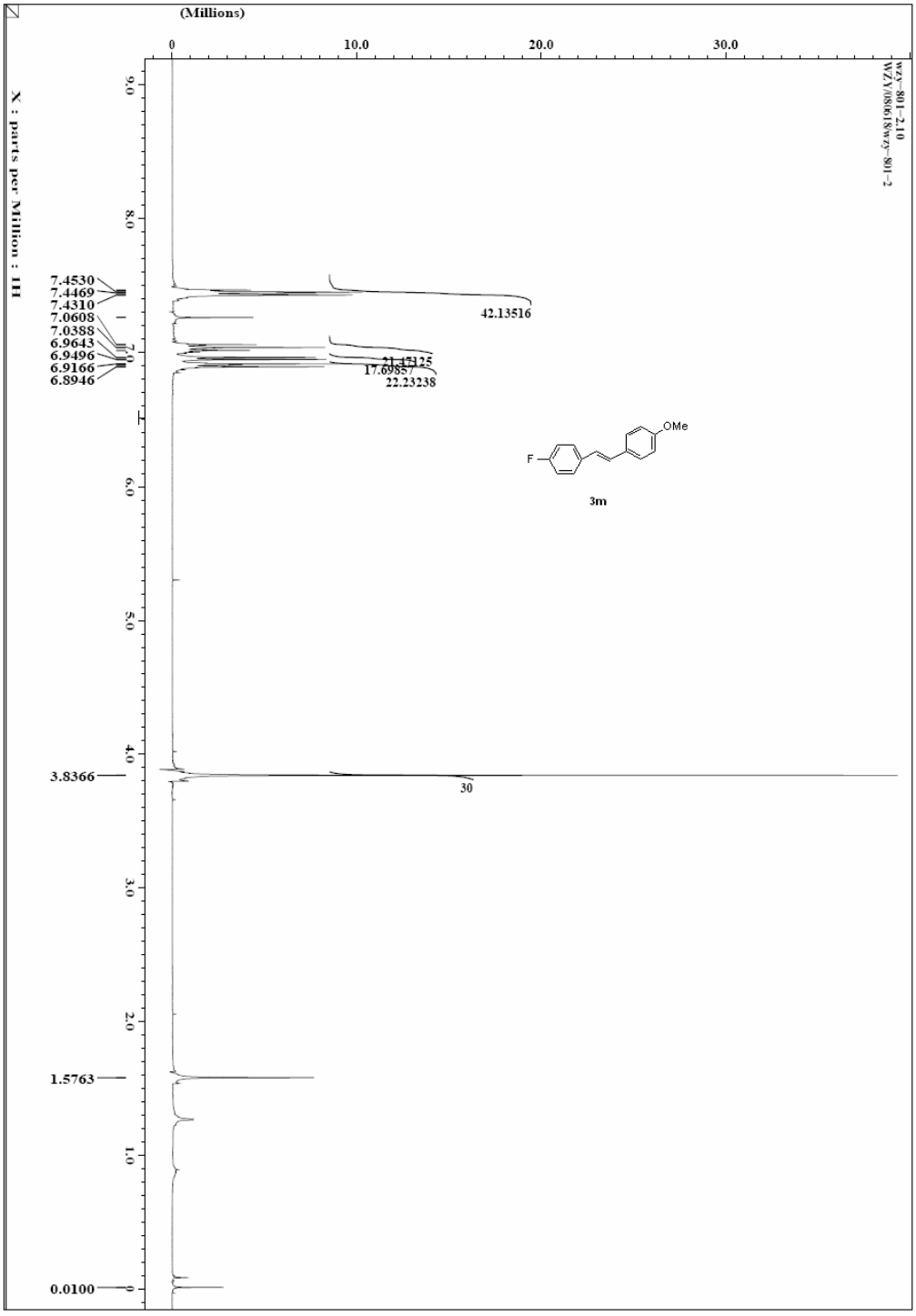

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
160.0604
148.8192
139.6087
132.0860
131.3613
129.5784
129.1302
128.2435
124.0102
121.6742
120.6635
114.3898
77.4242
77.1096
76.7949
55.4470
OMe
O2N
3j

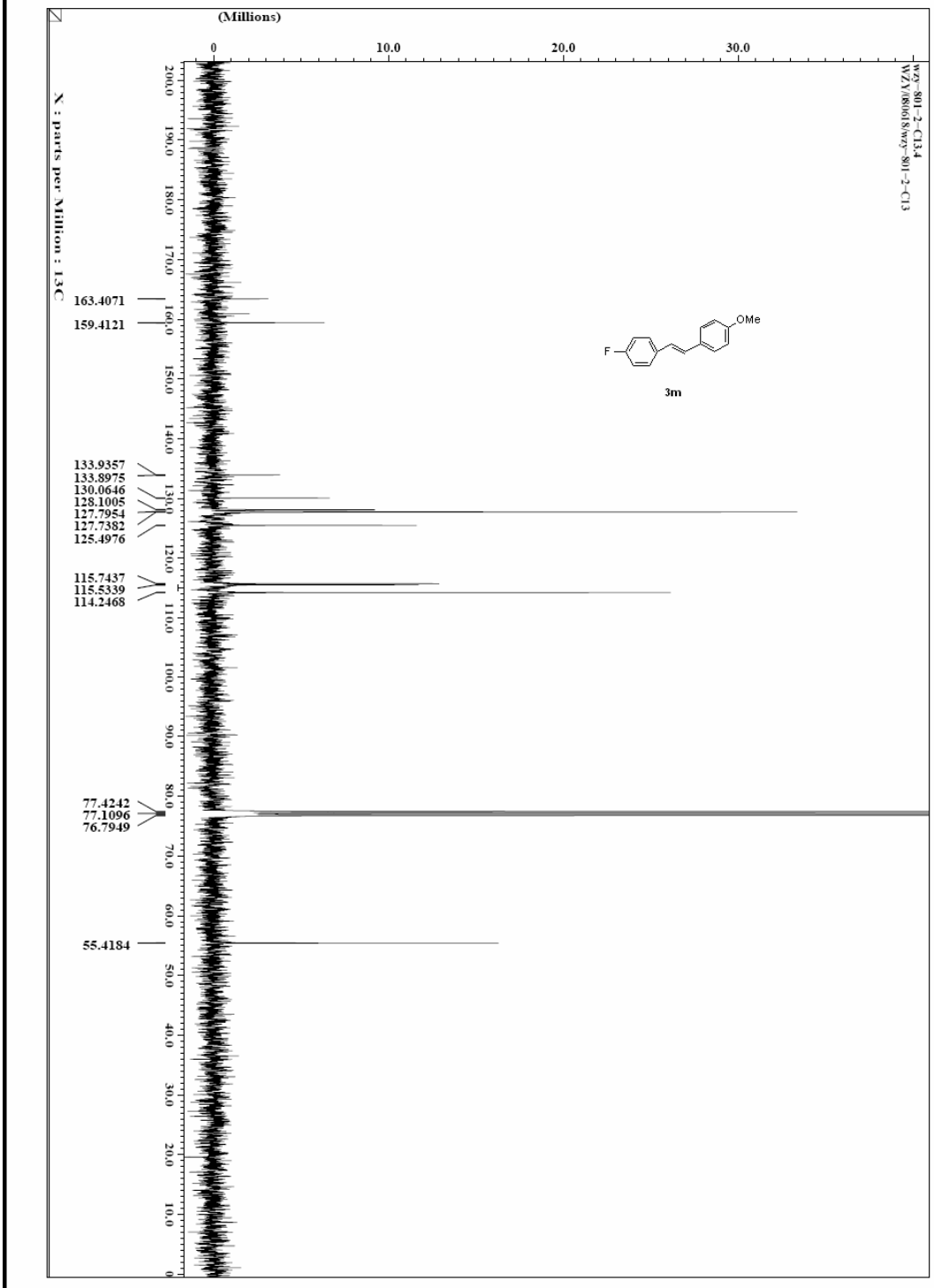

(Millions)
X : parts per Million : 1H
7.4520
7.4300
7.4190
7.3970
7.3127
7.2590
6.9108
6.8888
3.8320
1.5656
-0.0007
42.61616
20.71696
10
31.10485
31.37061
OMe
Cl
3k

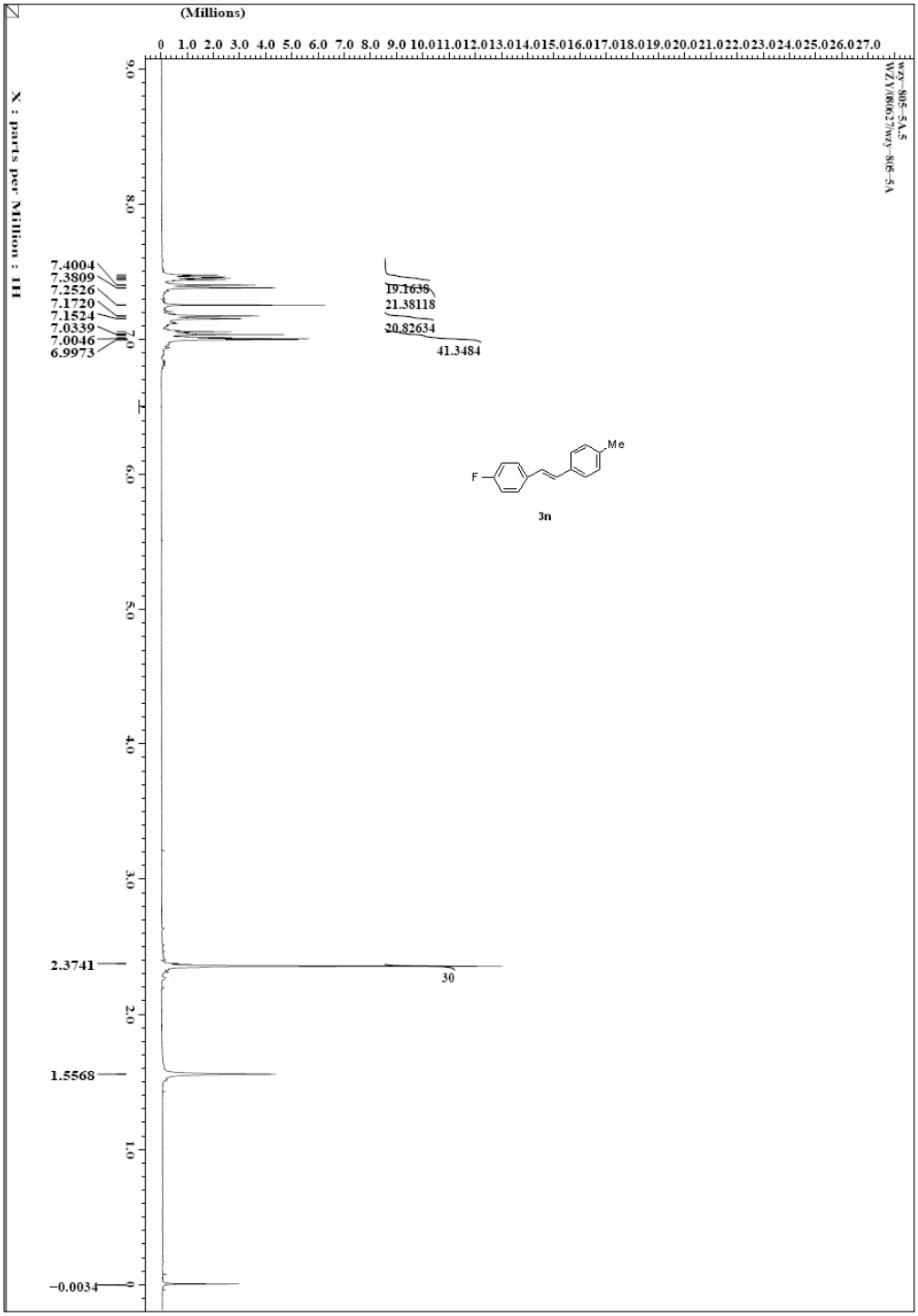

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 13C
wzy-741-1B-C13,3
WZY\080531\wzy-741-1B-C13
159.5742
136.2717
132.7725
129.8644
128.9205
128.8824
127.8908
127.4903
125.3450
114.2754
77.4433
77.1286
76.8140
55.4279
OMe
Cl
3k

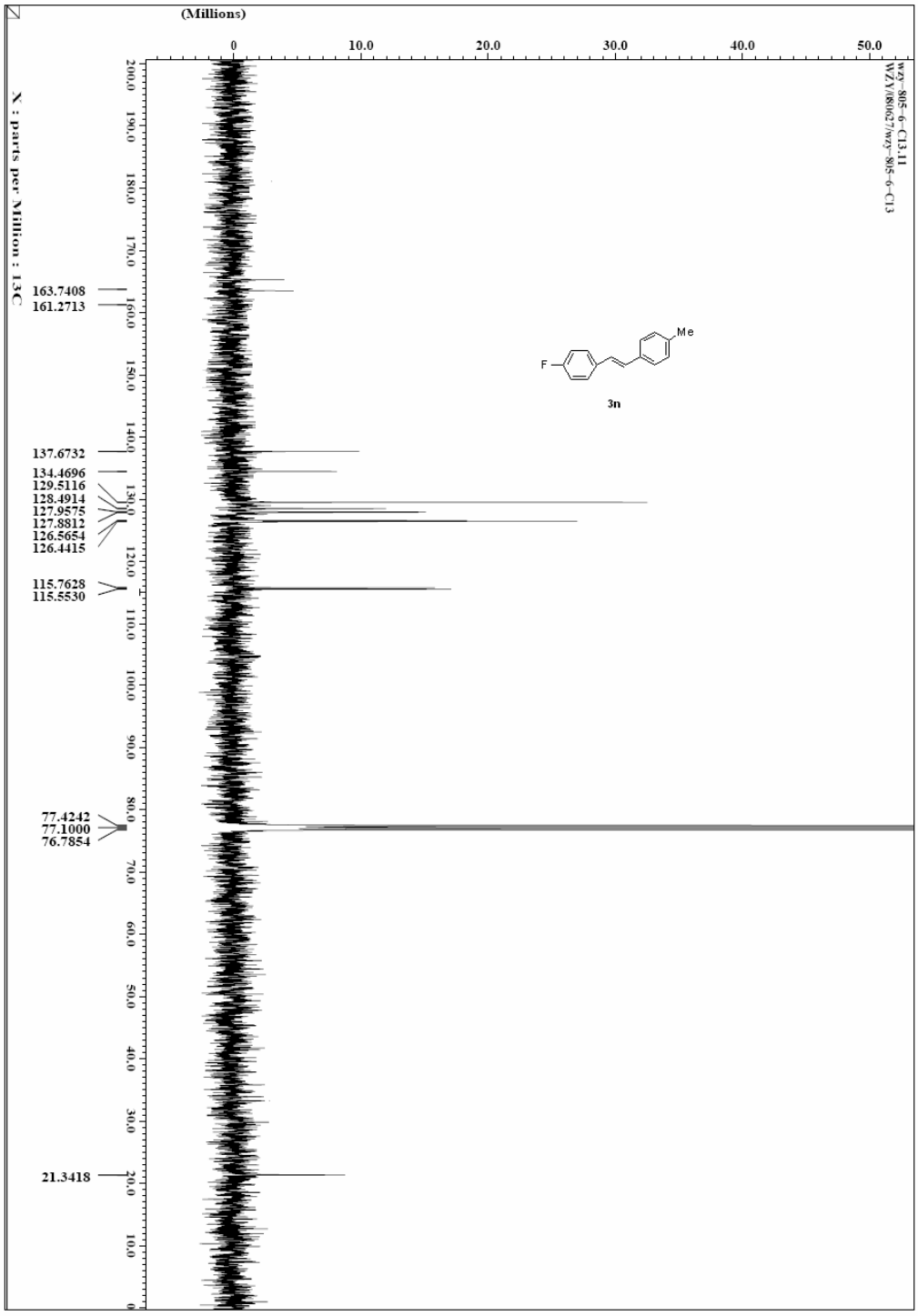

(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 1H
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.6190
7.5983
7.5518
7.5311
7.4822
7.4602
6.9666
6.9287
6.9067
3.8401
1.2537
0.0001
18.52086
18.68826
10
27.71834
29.20411
OMe
NC
3l

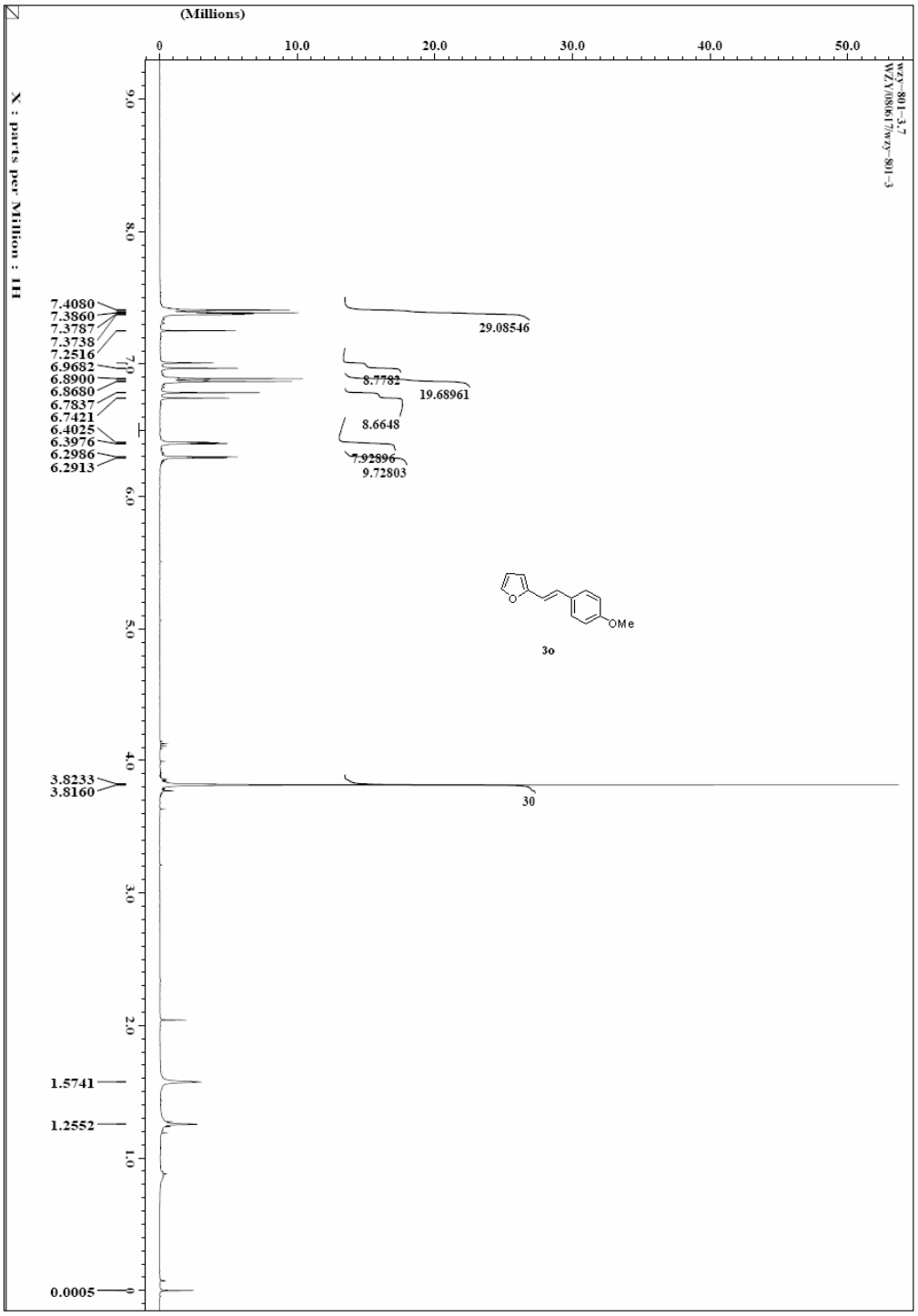

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
160.1749
142.3166
132.5436
132.0478
129.1589
128.3675
126.6513
124.6299
119.2620
114.3993
110.1183
77.4433
77.1286
76.8045
55.4565
NC
OMe
3l

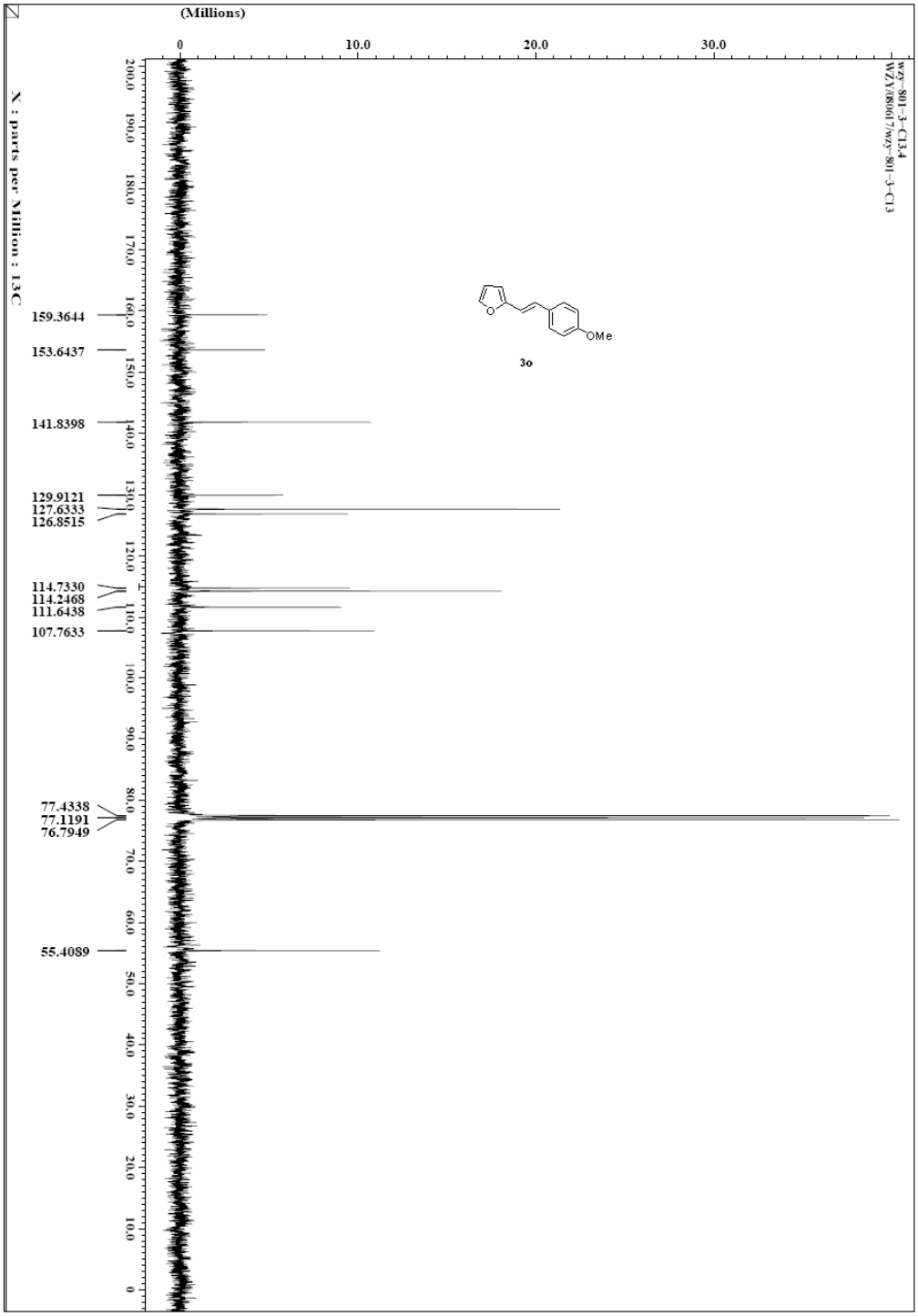

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 1H
7.4530
7.4469
7.4310
7.0608
7.0388
6.9643
6.9496
6.9166
6.8946
42.13516
17.69857
22.23238
3.8366
30
1.5763
0.0100
OMe
F
3m

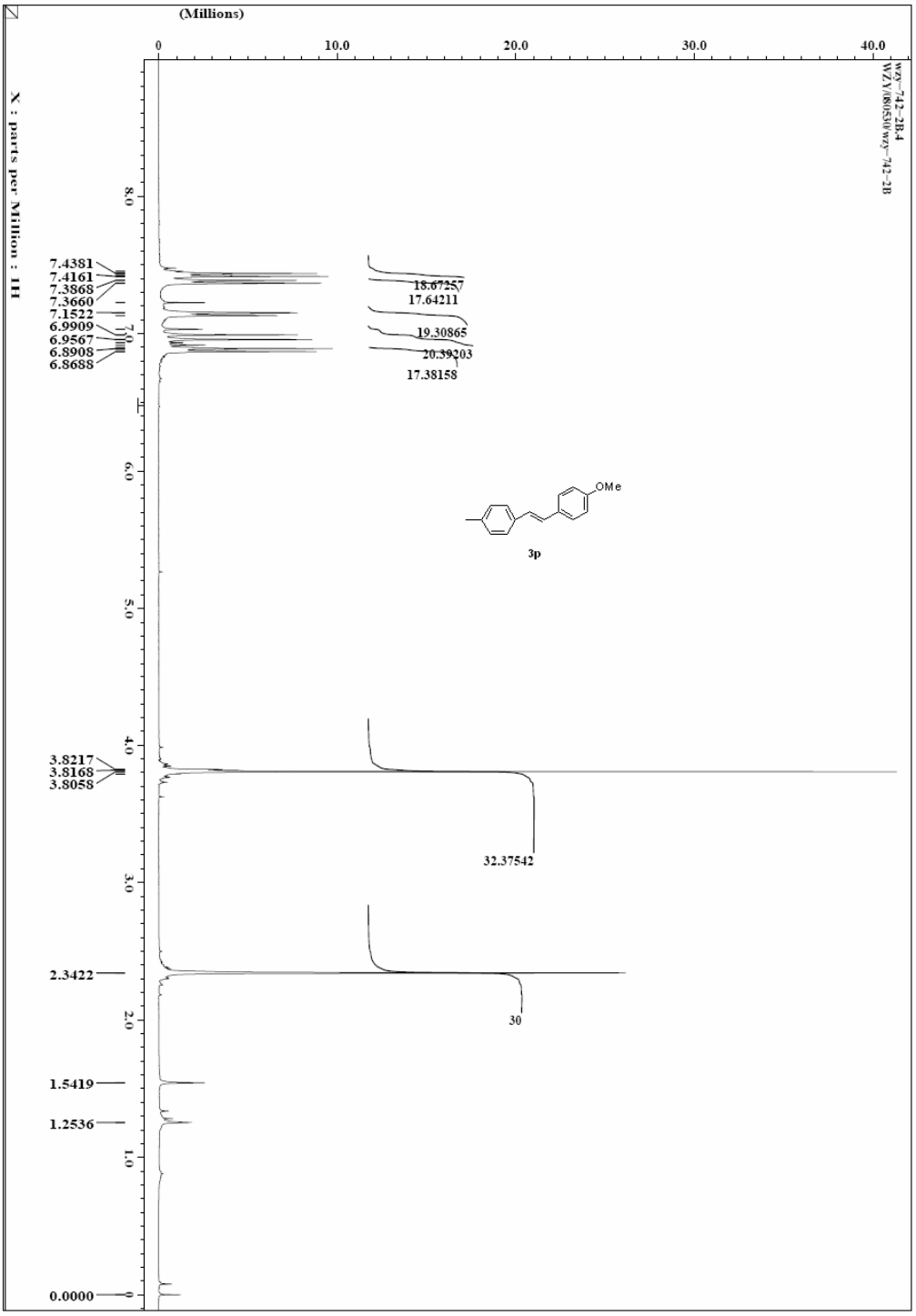

(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
wzy-801-2-C13.4
WZY080618wzy-801-2-C13
163.4071
159.4121
133.9357
133.8975
130.0646
128.1005
127.7954
127.7382
125.4976
115.7437
115.5339
114.2468
77.4242
77.1096
76.7949
55.4184
OMe
F
3m

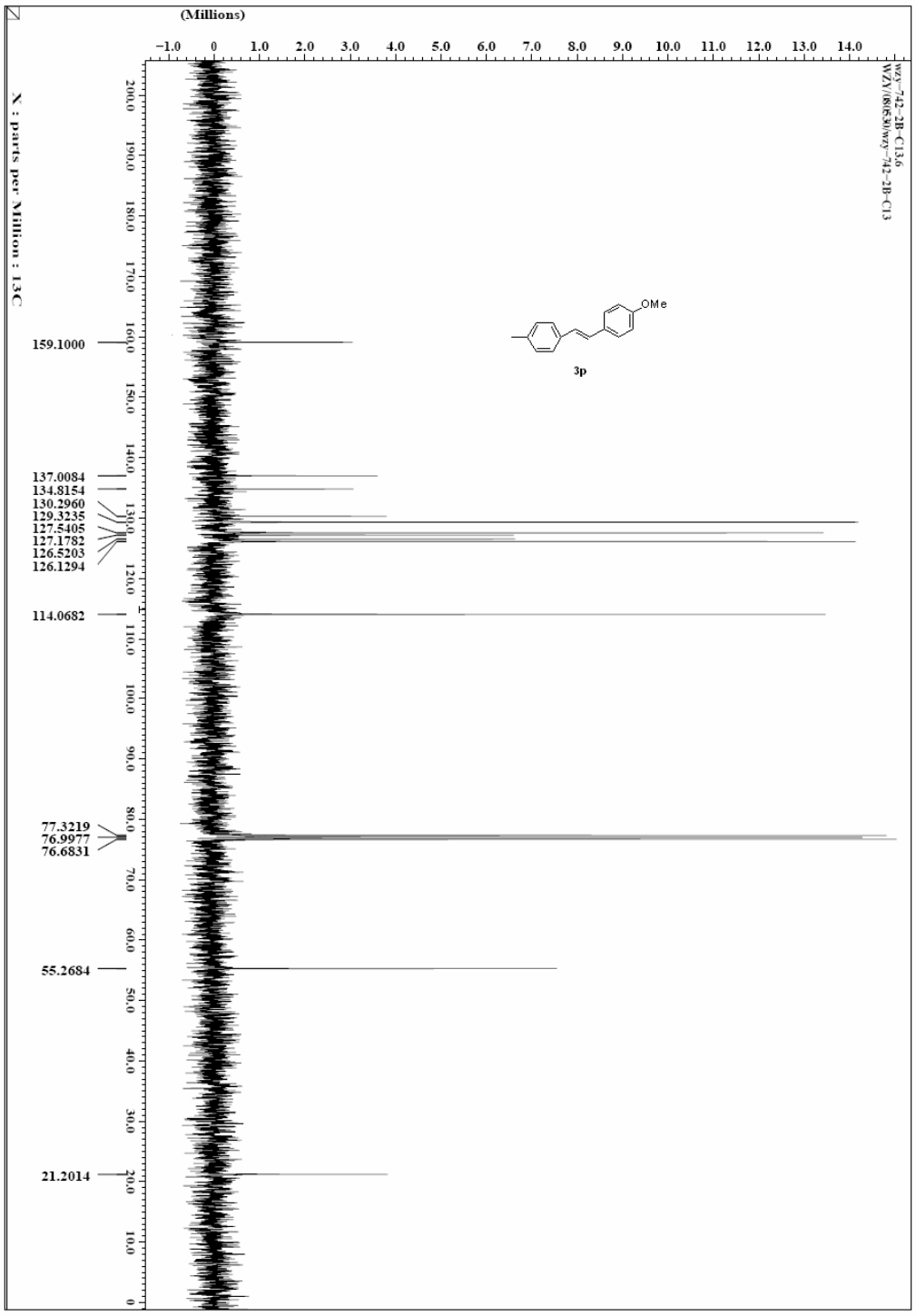

(Millions)
X : parts per Million : 1H
7.4004
7.3809
7.2526
7.1720
7.1524
7.0339
7.0046
6.9973
19.1636
21.38118
20.82634
41.3484
2.3741
30
1.5568
−0.0034
Me
F
3n

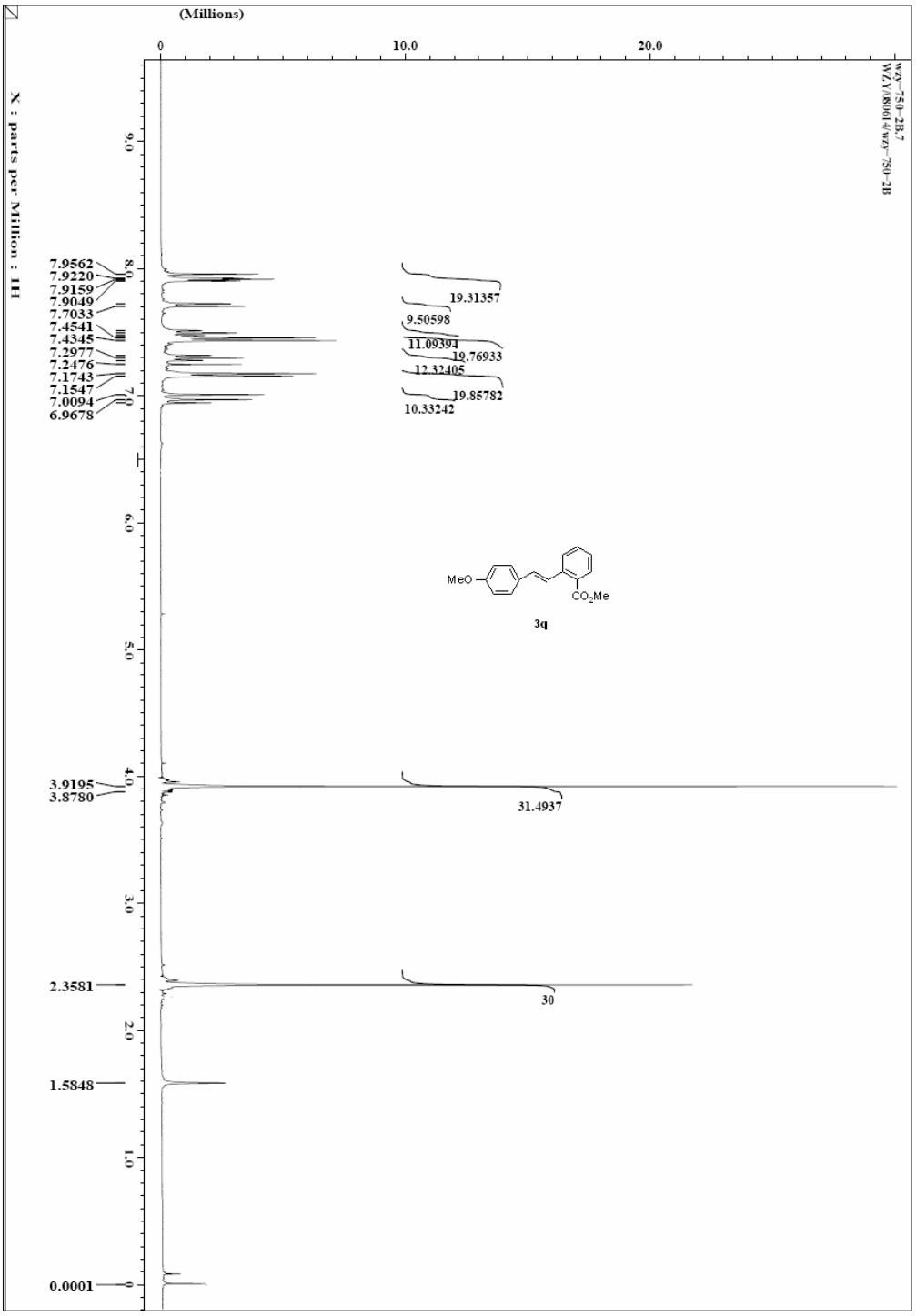

(Millions)
0
10.0
20.0
30.0
40.0
50.0
wzy-805-6-C13.11
WZY080627/wzy-805-6-C13
X : parts per Million : 13C
163.7408
161.2713
137.6732
134.4696
129.5116
128.4914
127.9575
127.8812
126.5654
126.4415
115.7628
115.5530
77.4242
77.1000
76.7854
21.3418
F
Me
3n

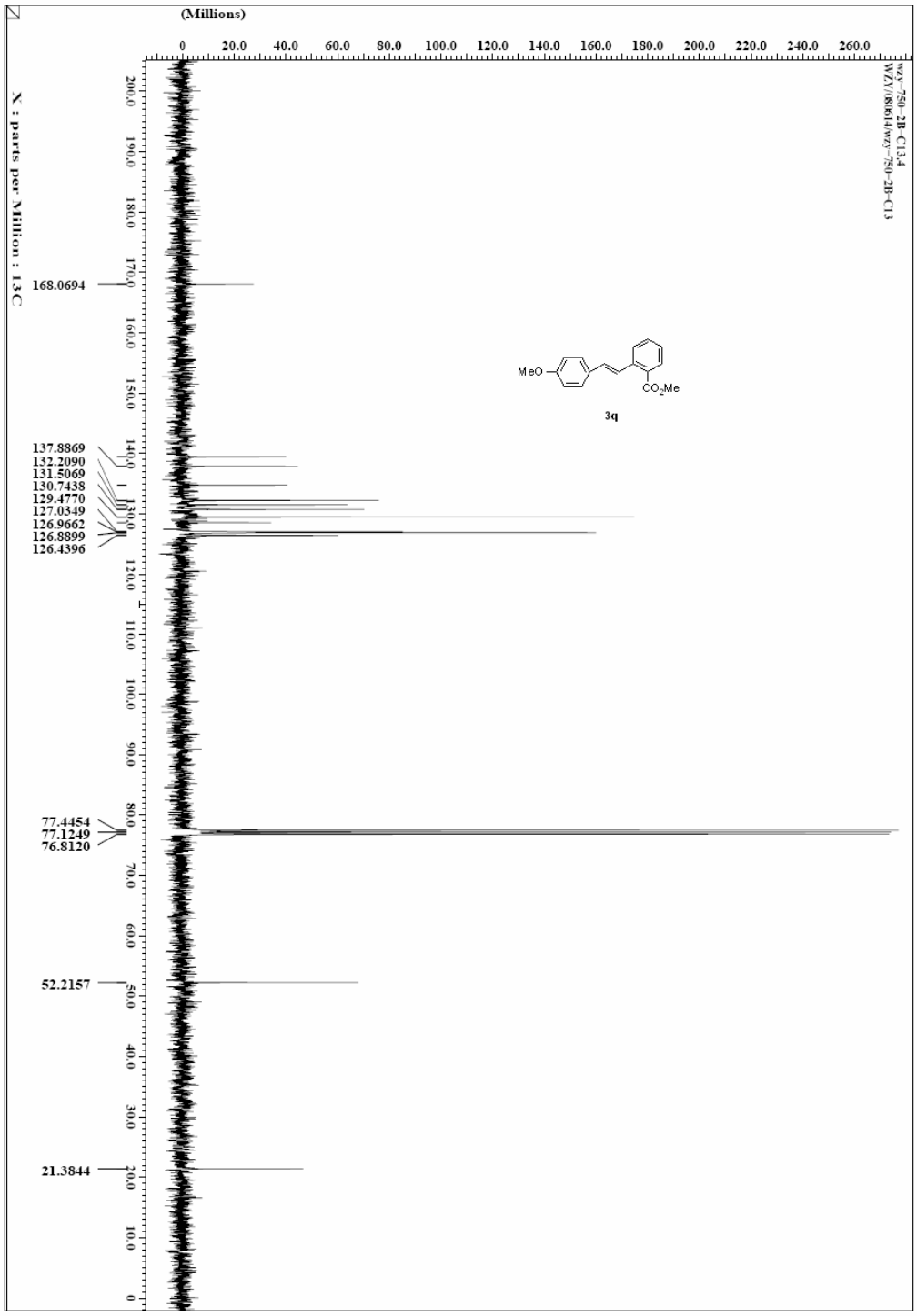

(Millions)
0
10.0
20.0
30.0
40.0
50.0
X : parts per Million : 1H
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.4080
7.3860
7.3787
7.3738
7.2516
6.9682
6.8900
6.8680
6.7837
6.7421
6.4025
6.3976
6.2986
6.2913
3.8233
3.8160
1.5741
1.2552
0.0005
29.08546
8.7782
19.68961
8.6648
7.92896
9.72803
30
OMe
3o


(Millions)
0
10.0
20.0
30.0
X : parts per Million : 13C
159.3644
153.6437
141.8398
129.9121
127.6333
126.8515
114.7330
114.2468
111.6438
107.7633
77.4338
77.1191
76.7949
55.4089
OMe
3o


(Millions)
0
10.0
20.0
30.0
40.0
X : parts per Million : 1H
wzy-742-2B-4
WZY080530 wzy-742-2B
7.4381
7.4161
7.3868
7.3660
7.1522
6.9909
6.9567
6.8908
6.8688
18.67257
17.64211
19.30865
20.39203
17.38158
OMe
3p
3.8217
3.8168
3.8058
32.37542
2.3422
30
1.5419
1.2536
0.0000


(Millions)
X : parts per Million : 13C
159.1000
137.0084
134.8154
130.2960
129.3235
127.5405
127.1782
126.5203
126.1294
114.0682
77.3219
76.9977
76.6831
55.2684
21.2014
OMe
3p


(Millions)
0
10.0
20.0
X : parts per Million : 111
wzy-750-2B-7
WZY08060-4 wzy-750-2B
7.9562
7.9220
7.9159
7.9049
7.7033
7.4541
7.4345
7.2977
7.2476
7.1743
7.1547
7.0094
6.9678
19.31357
9.50598
11.09394
19.76933
12.32405
19.85782
10.33242
MeO
CO2Me
3q
3.9195
3.8780
31.4937
2.3581
30
1.5848
0.0001


(Millions)
N : parts per Million : 13C
wzy-750-2B-C13.4
WZY080614/wzy-750-2B-C13
168.0694
137.8869
132.2090
131.5069
130.7438
129.4770
127.0349
126.9662
126.8899
126.4396
77.4454
77.1249
76.8120
52.2157
21.3844
MeO
CO2Me
3q