Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Electronic Supplementary Information

A Site Selective C-H Arylation of Free-(NH_2) Adenines with Aryl Chlorides: Application to the Synthesis of 6,8-Disubstituted Adenines

Sophian Sahnoun, Samir Messaoudi, Jean-Daniel Brion, and Mouâd Alami*

Univ Paris-Sud, BioCIS UMR-8076, Laboratoire de Chimie Thérapeutique, Faculté de Pharmacie, 5 rue J.-B. Clément, Châtenay-Malabry, F-92296, France

mouad.alami@u-psud.fr,

Phone: 33(0)1.46.83.58.87 ; Fax: 33(0)1.46.83.58.28

Contents

General experimental methods

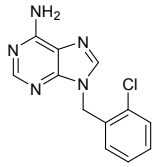
All reactions were conducted under an argon atmosphere. Solvents: cyclohexane, ethyl acetate (EtOAc), methylene chloride (CH_2Cl_2), methanol (MeOH) for extraction and chromatography were technical grade. Diethylether (Et_2O) and tetrahydrofuran (THF) were distilled under argon from sodium-benzophenone ketyl and piperidine, *n*-butylamine, aniline, morpholine from potassium hydroxide.

Instrumentation

The compounds were all identified by usual physical methods, i.e. ^{1}H-NMR, ^{13}C-NMR, IR, elemental analysis. ^{1}H and ^{13}C NMR spectra were measured in $CDCl_3$ or DMSO-d_6 on a 300 MHz spectrometer. ^{1}H chemical shifts are reported in ppm from an internal standard TMS or of residual chloroform (7.27 ppm). The following abreviation are used: m (multiplet), s (singlet), br s (broad singlet), d (doublet), t (triplet) dd (doublet of doublet), td (triplet of doublet), q (quadruplet), quint (quintuplet). ^{13}C chemical shifts are reported in ppm from the central peak of deuteriochloroform (77.14). IR spectra were acquired on a FT-IR and are reported in wave numbers (cm^{-1}). Elemental analyses were performed with a Perkin-Elmer 240 analyser. R_f values refer to TLC on 0.25 mm silica gel plates (60-F_{254}). Flash chromatography was performed on silica gel 60 (0.040-0.063 mm). Melting points (m.p.) were determined on a capillary melting point apparatus and were uncorrected. Aryls chlorides, bromides and iodides are commercially available compounds. Adenines **1a**[1]**, 1b**[2] **and 1c**[2] were synthesized as described.

Synthetic details for adenine compounds 1d[2] and 2a[3]

Compound 1d

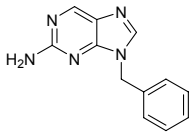


In a double-necked round bottom flask (100 mL) equipped with a condenser was added a mixture, consisting of alcohol (0.01 mol), TsIm (0.012 mol), Et_3N (0.02 mol), K_2CO_3 (0.01 mol), and adenine (0.01 mol) in DMF (30 mL). The mixture was heated at reflux, and in most cases, darkening occurred. Heating was continued until TLC indicated no further improvement in the conversion. The solvent was evaporated under vacuum and the remaining foam was dissolved in $CHCl_3$ (100 mL) and subsequently washed with water (2×100 mL). The organic layer was dried (Na_2SO_4) and evaporated. The crude product was purified by column chromatography on silica gel eluting with CH_2Cl_2/MeOH : 97/3 yielded the desired product **1d**.

Yield: 54% (1.38 g); white solid; mp: 259-261 °C; TLC : R_f 0.48 (CH_2Cl_2/MeOH : 9/1); IR (neat): ν(cm^{-1}) 3078, 2361, 1678, 1602, 1574, 1474, 1416, 1306, 1242, 1142, 1081, 1050, 751; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.18 (s, 1H), 8.12 (s, 1H), 7.51 (m, 1H), 7.33 (m, 2H), 7.26 (s, 2H, N*H*), 6.98 (m, 1H), 5.46 (s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ156.0, 152.6, 149.5, 134.0, 131.8, 129.5, 129.4, 129.1, 127.5, 118.5, 44; *m/z* MS (ES+) ($M+H^+$) 260; Anal. Calcd for $C_{12}H_{10}ClN_5$ (259,69): C 55.50, H 3.88, N 26.97; found: C 55.93, H 4.09, N 27.13.

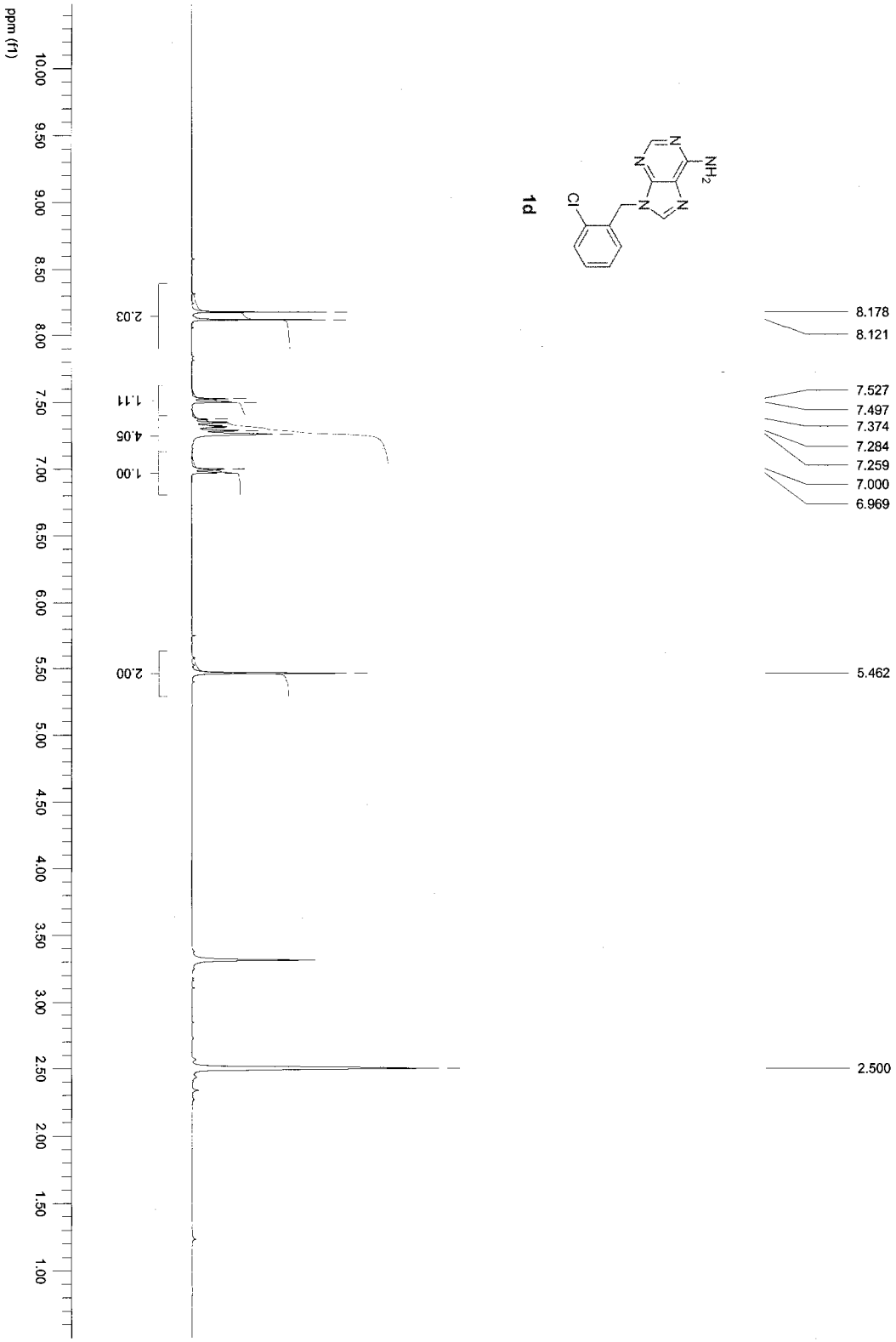
1 Sun, Z.; Hosmane, R.-S. *Synth. Commun*. **2001**, *31*, 549-554
2 Soltani Rad, A. A.; Khalafi-Nezhad, M. N.; Behrouz, S.; Faghihi, M. A.; Zare, A.; Parhami*, A. Tetrahedron*, **2008**, *64***,** 1778-1785.
3 Isobe, Y.; Kurimoto, A.; Tobe, M.; Hashimoto, K.; Nakamura, T.; Norimura, K.; Ogita, H.; Takaku H. *J. Med. Chem.* **2006**, *49*, 2088-2095.

Compound 2a



To a suspension of 2-amino purine (0.68 g, 4 mmol) and potassium carbonate (0.7 g, 5 mmol) in DMF (10 mL) was added 2-chlorobenzyl chloride (1.0 g, 6.2 mmol) and tetrabutyl ammonium iodide (catalytic amount). The mixture was stirred for 24h at room temperature. The reaction mixture was concentrated and the crude product was purified by column chromatography on silica gel eluting with CH_2Cl_2/MeOH : 97/3.

Yield: 30% (270 mg); light yellow solid; mp: 179-181 °C; TLC : R_f 0.40 (CH_2Cl_2/MeOH : 9/1); IR (neat): ν(cm^{-1}) 3463, 3279, 3131, 2924, 1609, 1576, 1516, 1460, 1423, 1363, 1296, 1259, 1211, 1168, 1027, 973, 951, 848, 790; ^{1}H NMR (300 MHz, DMSO-d_6): δ 8.59 (s, 1H), 8.15 (s, 1H), 7.30 (m, 5H),6.52 (s, 2H, N*H*), 5.29 (s, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 160.6, 152.9, 149.0, 142.6, 136.9, 128.6 (2C), 127.5, 127.0 (2C), 126.6, 45.3; *m/z* MS (ES+) ($M+H^+$) 226; Anal. Calcd for $C_{12}H_{11}N_5$ (225,1): C 6.99, H 4.92, N 31.09; found C 64.14, H 5.10, N 31.31.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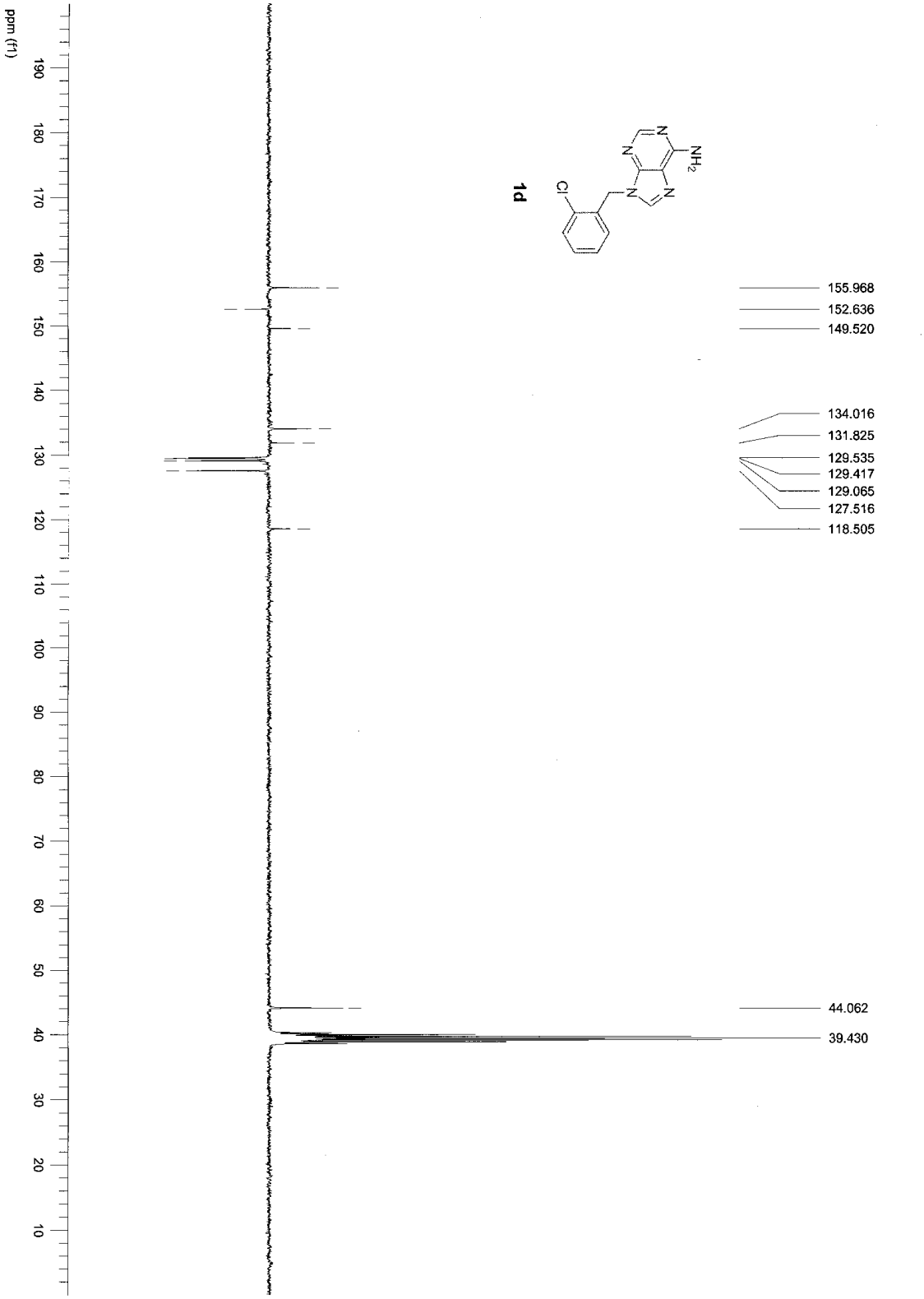
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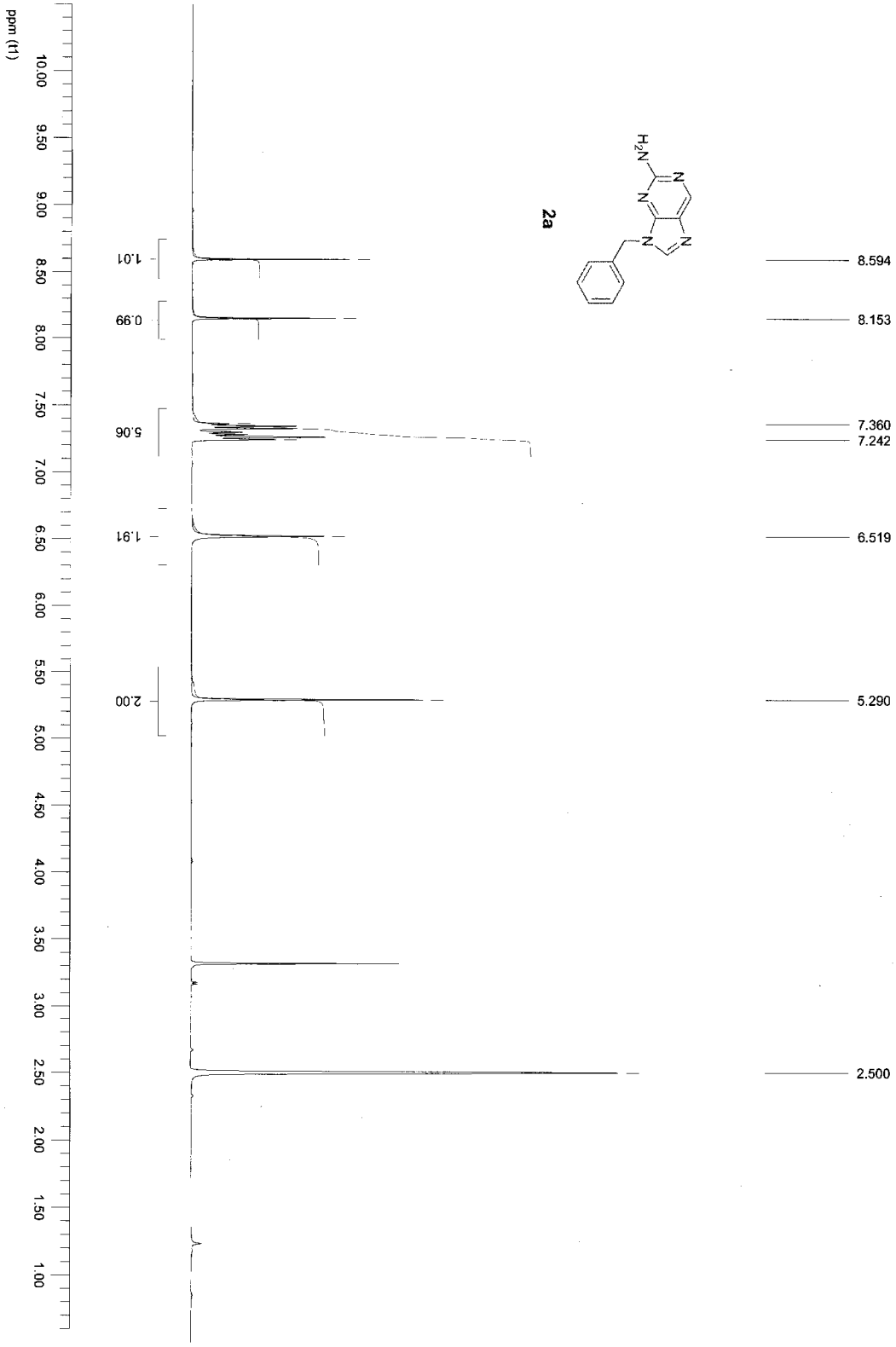
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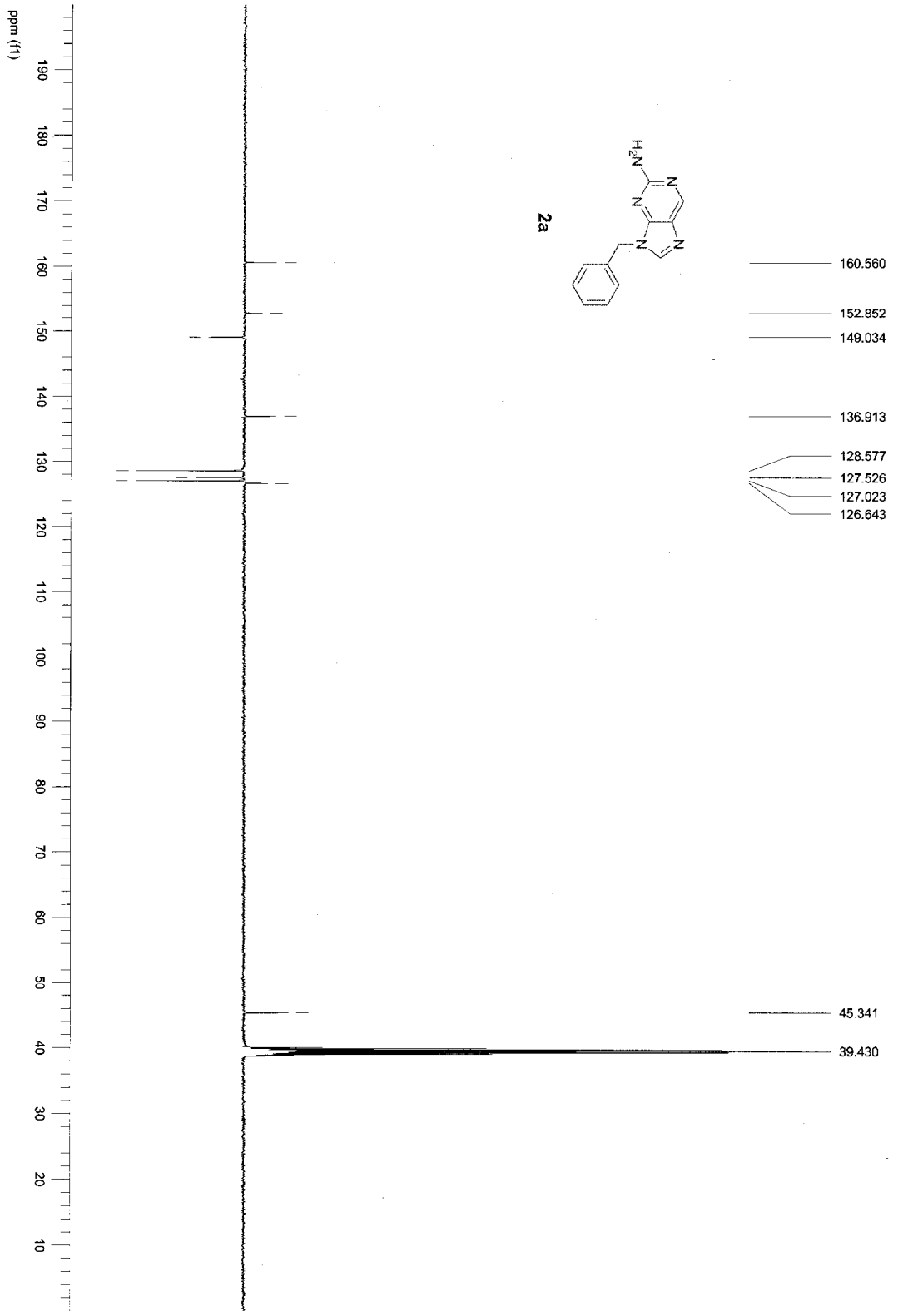
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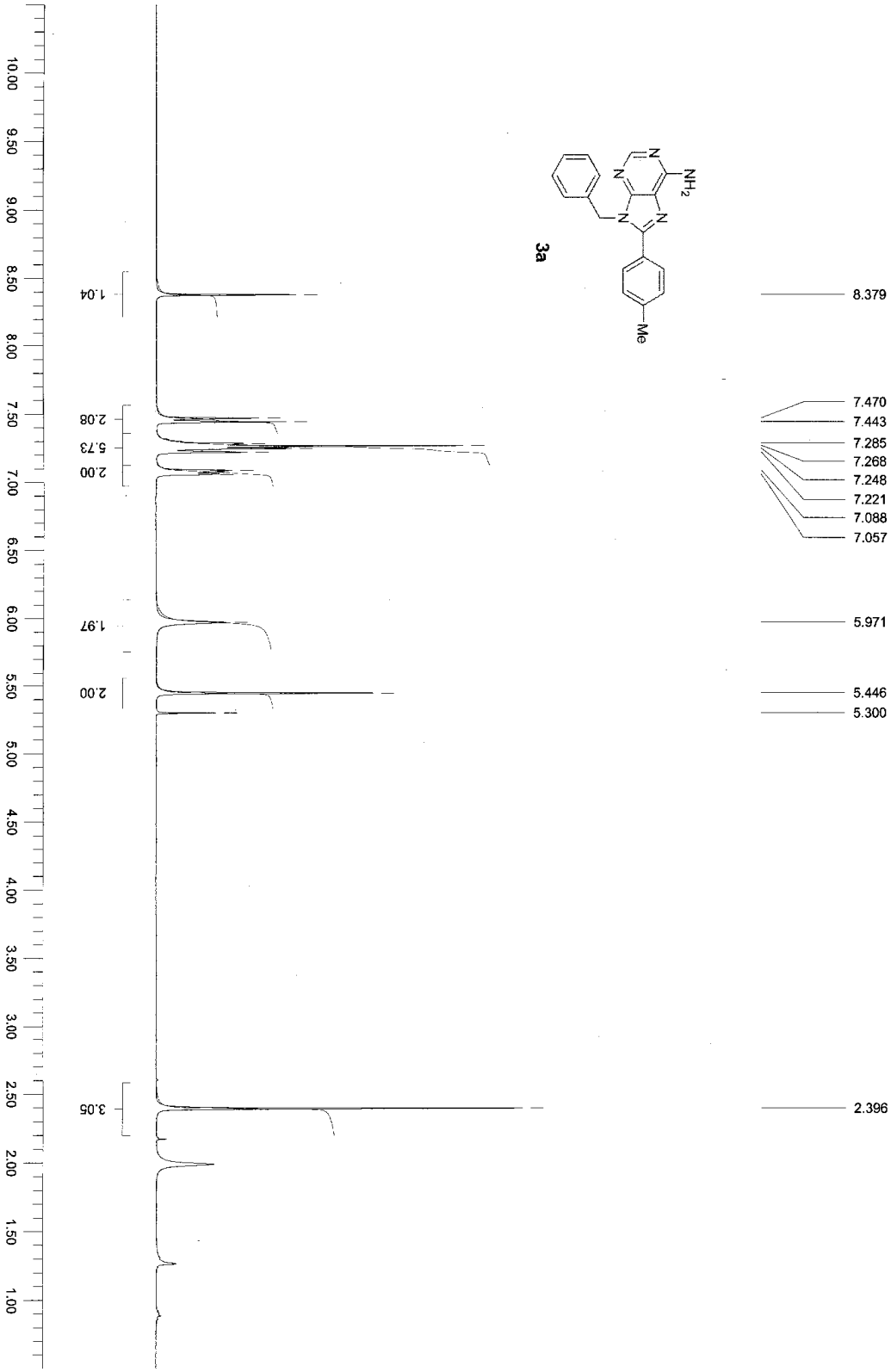
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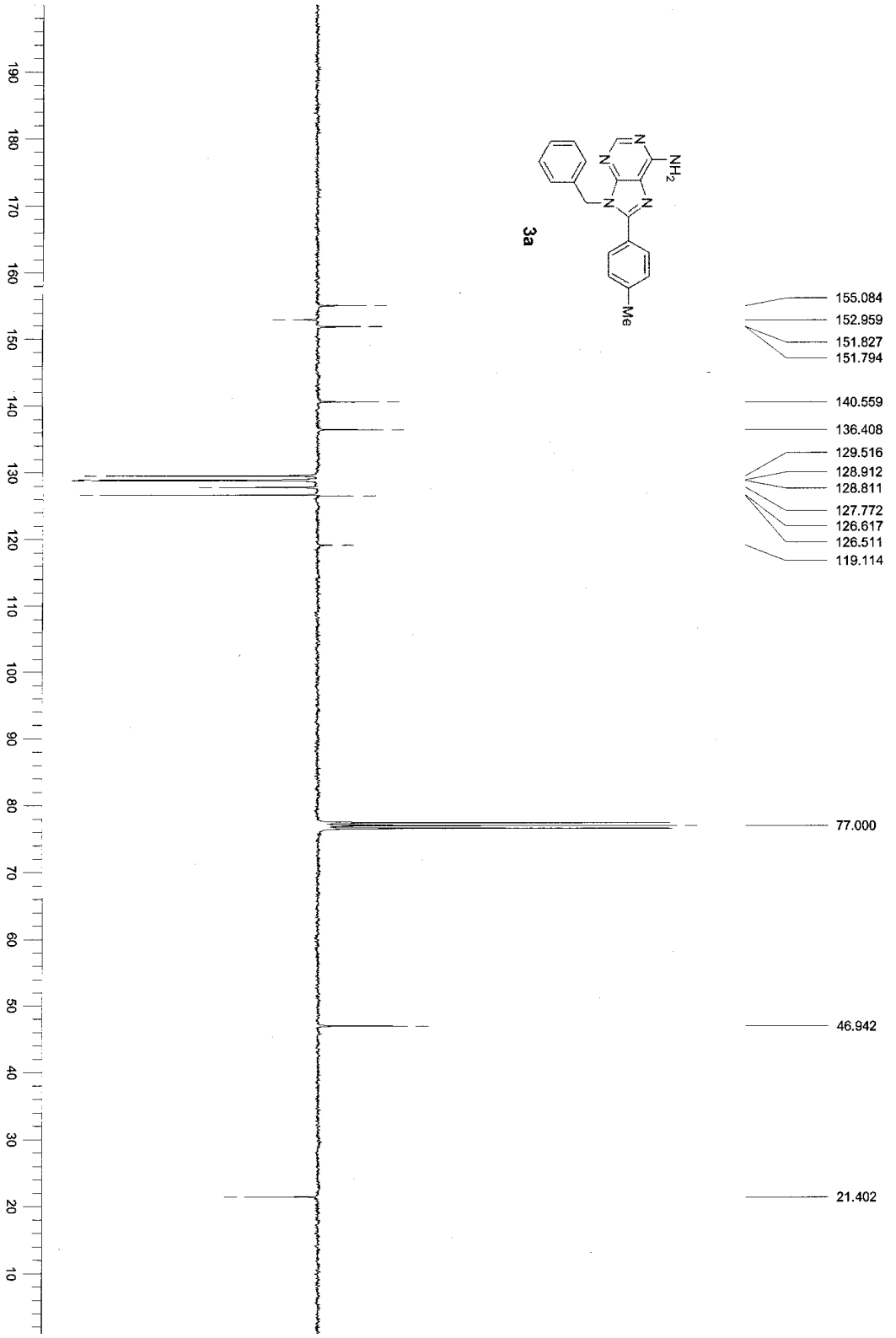
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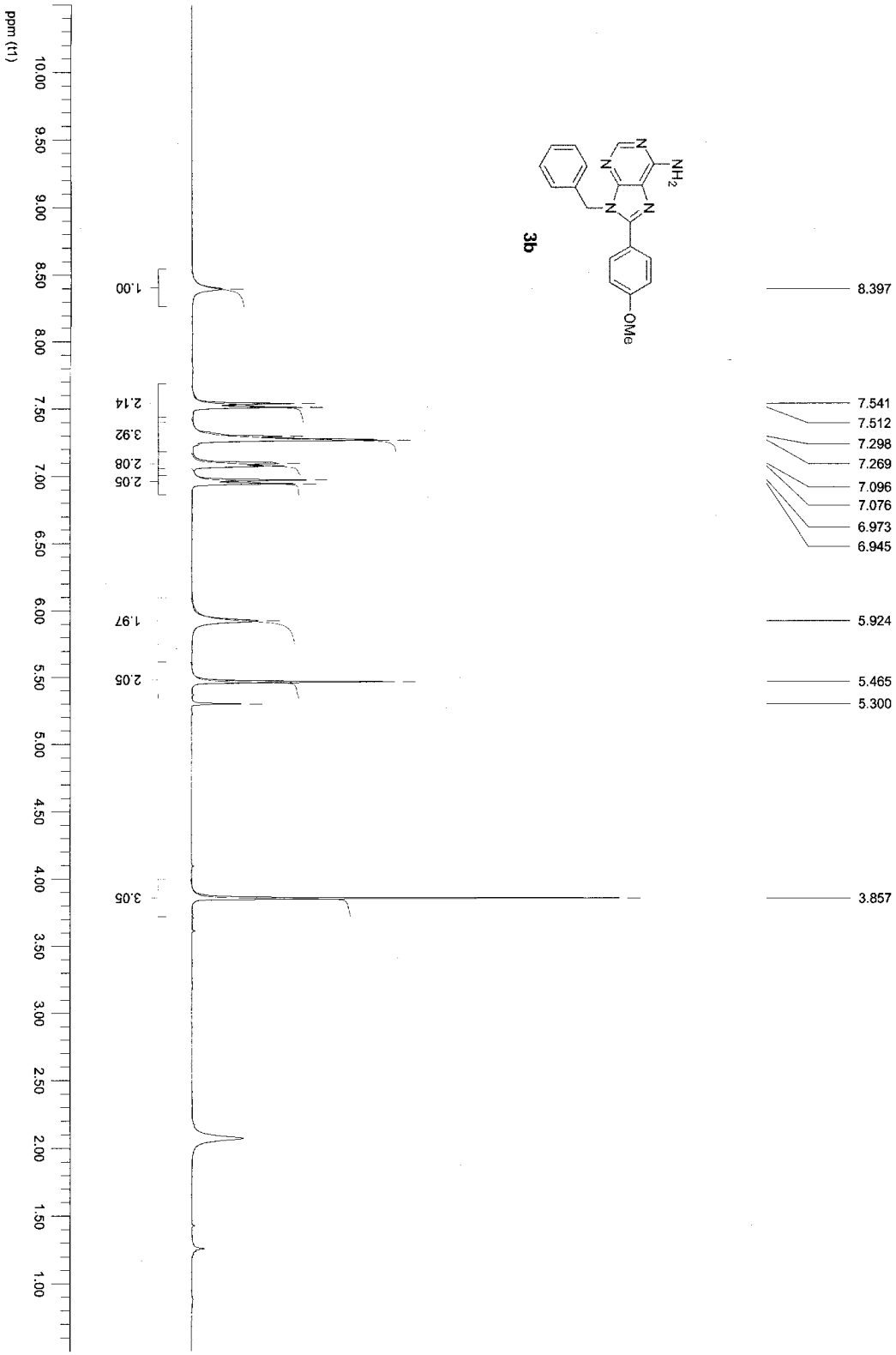
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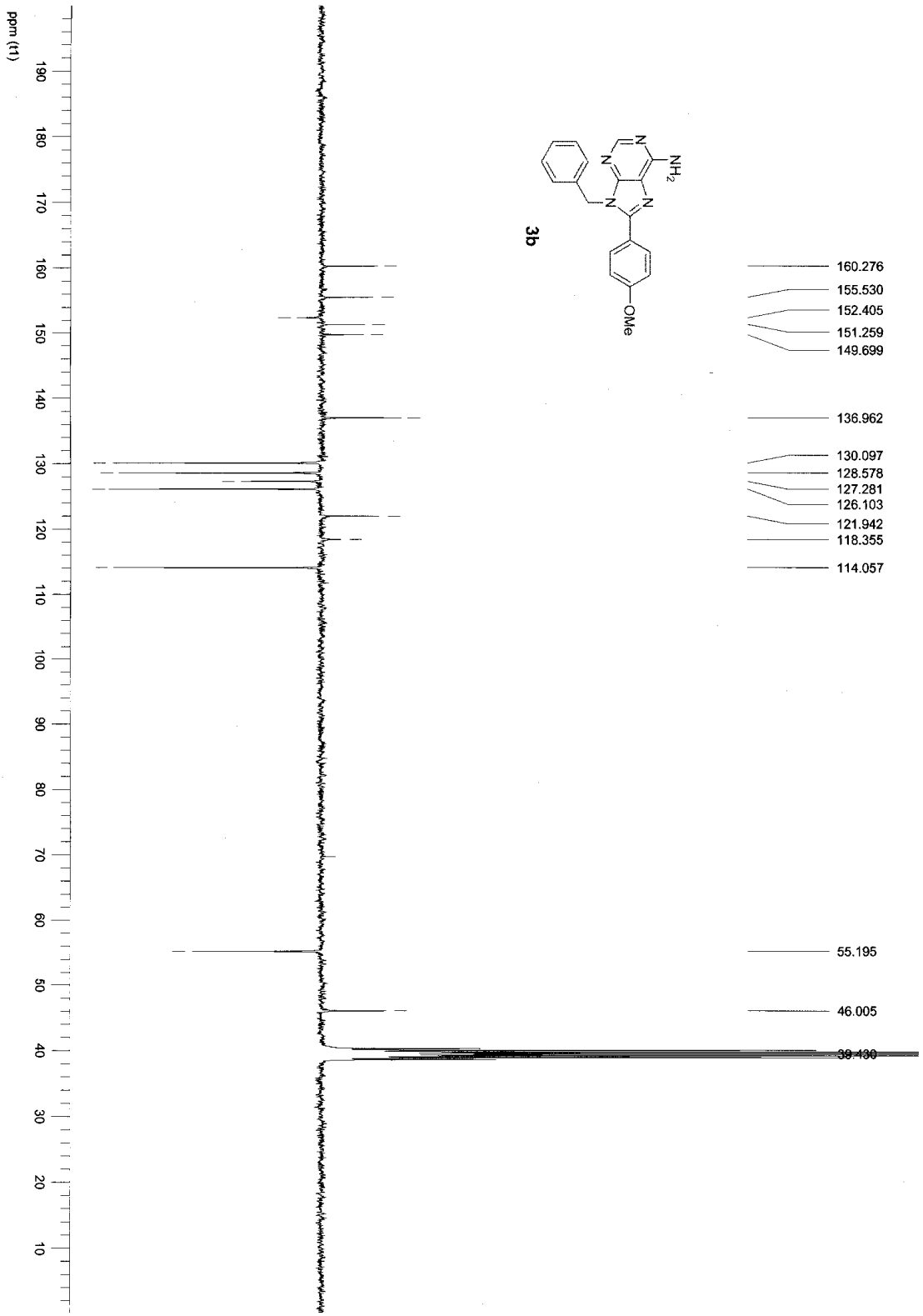
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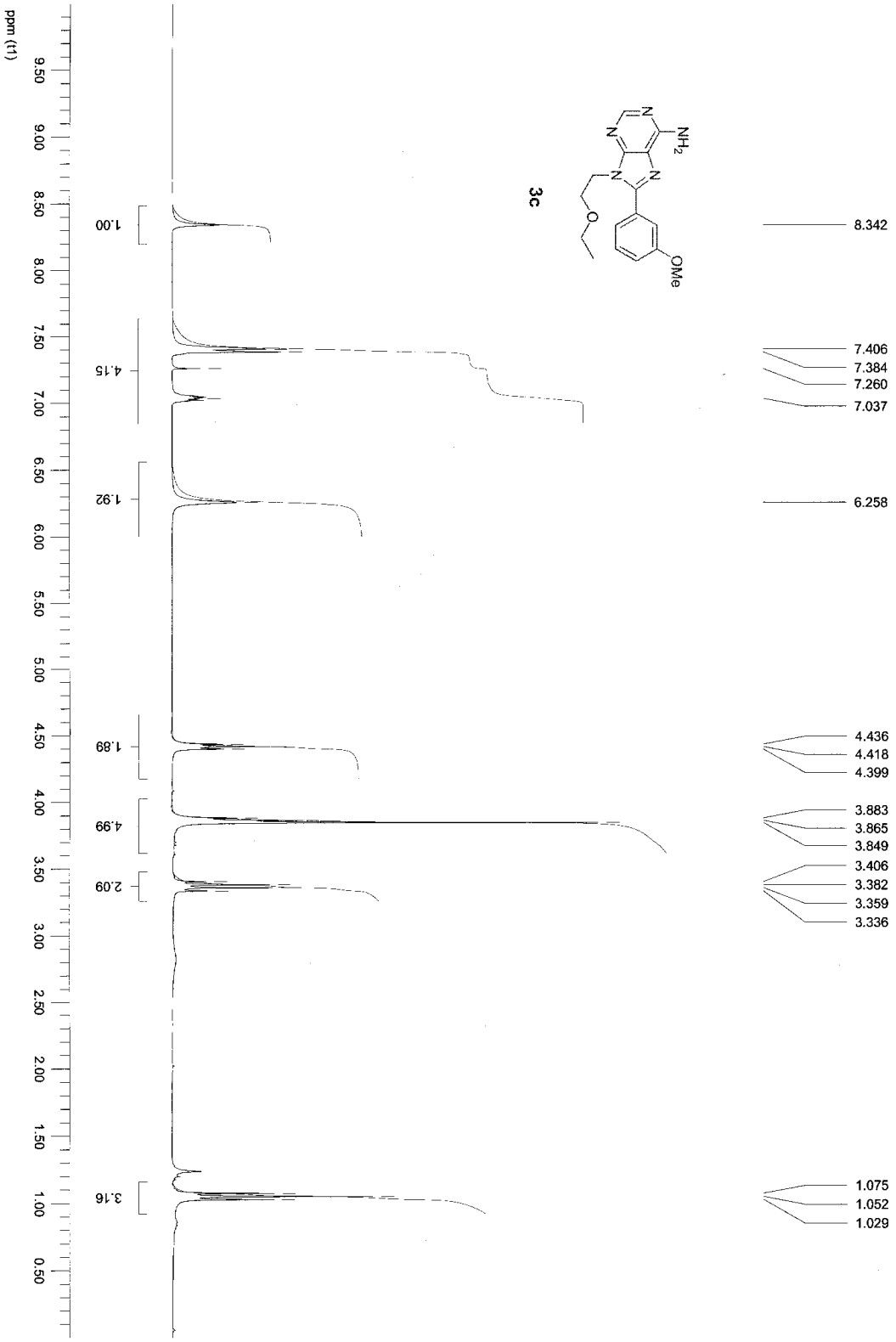
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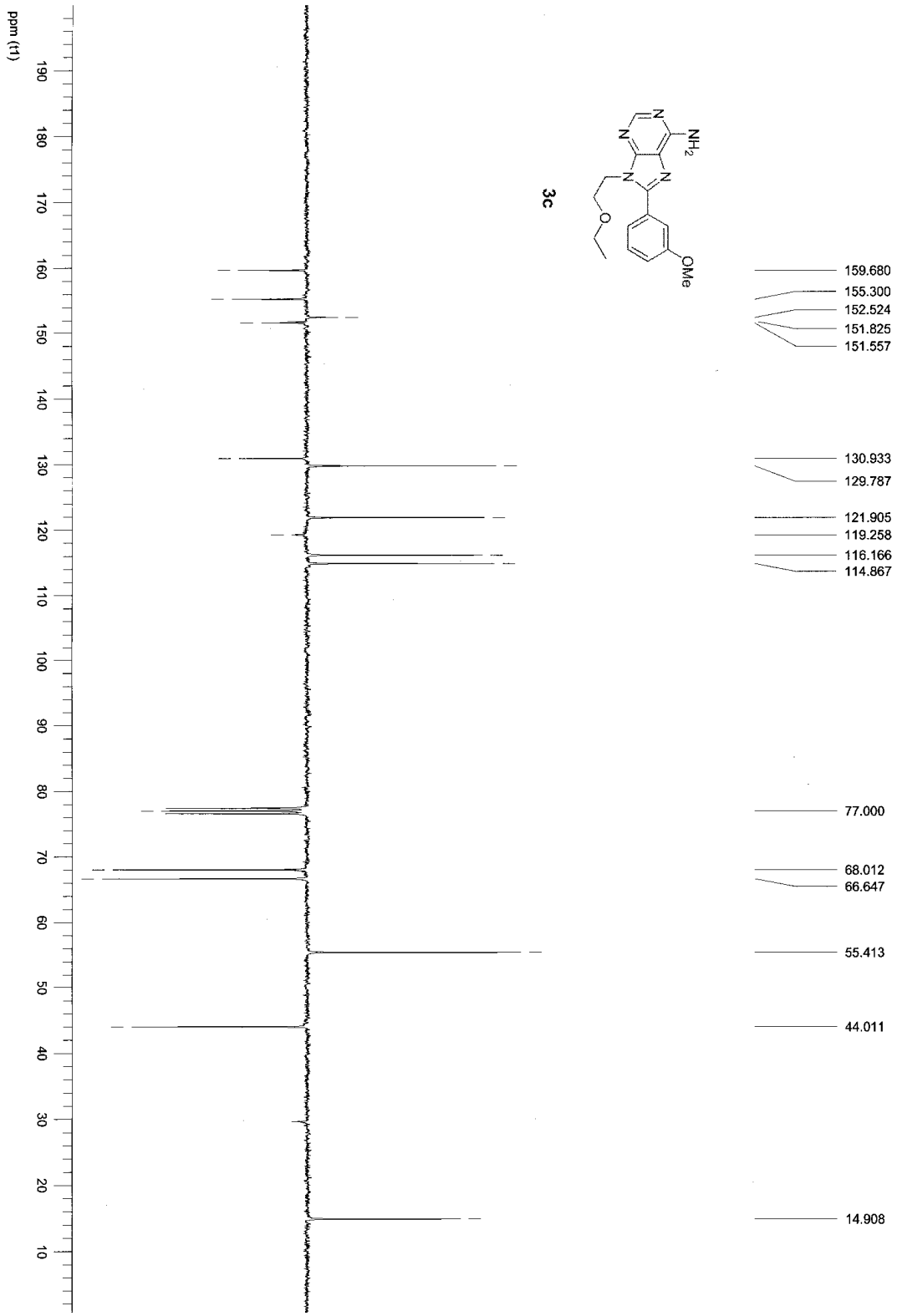
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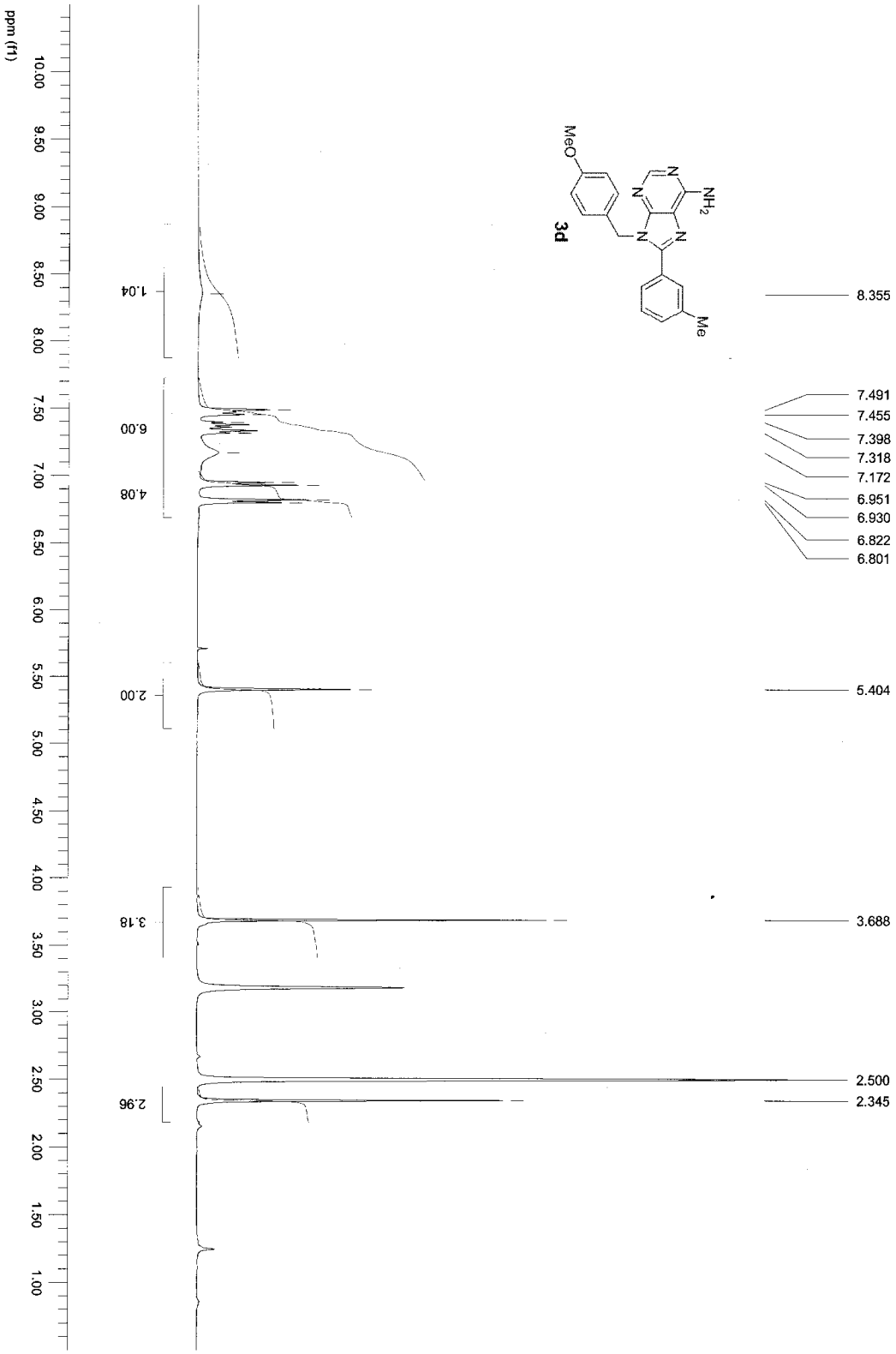
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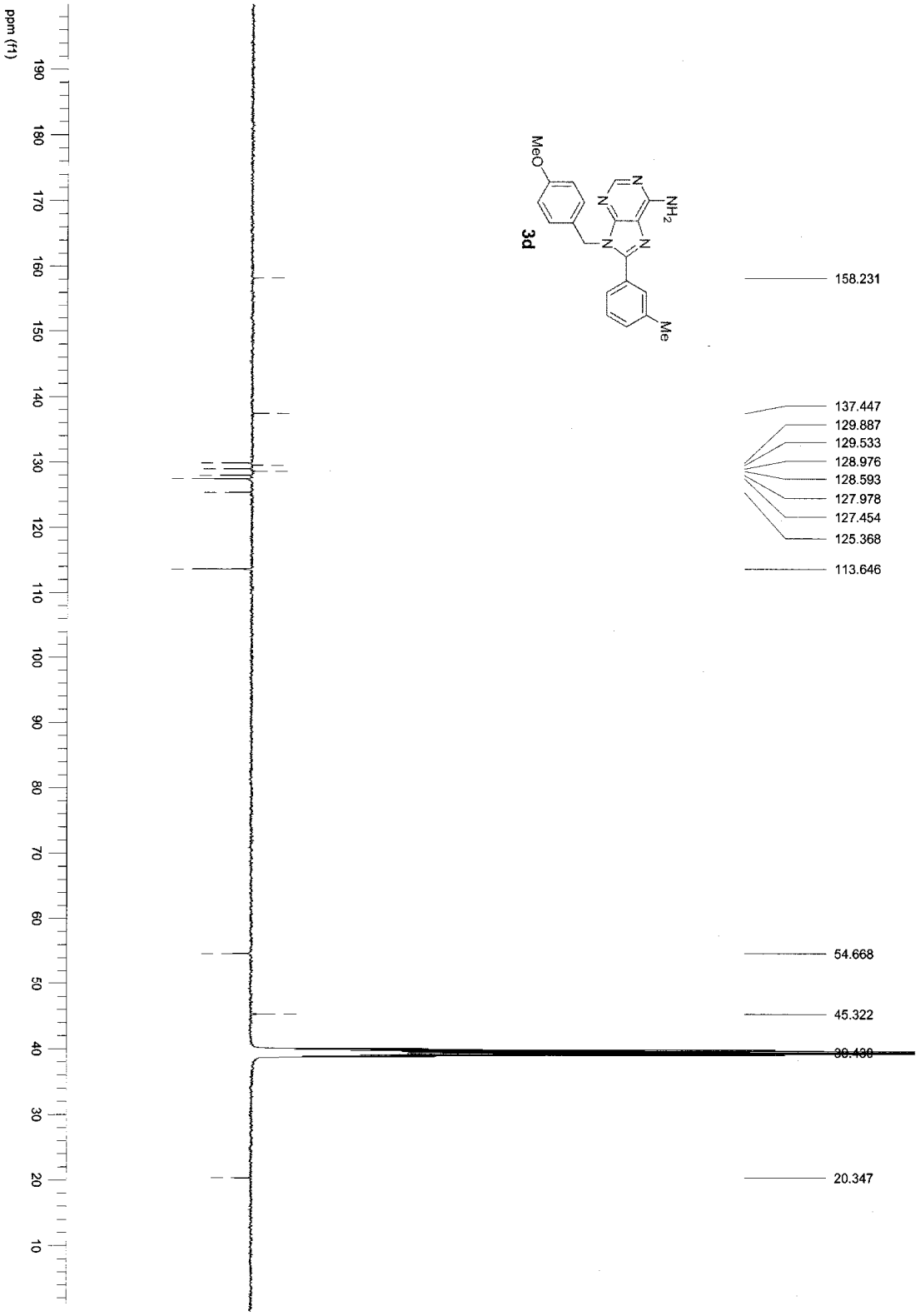
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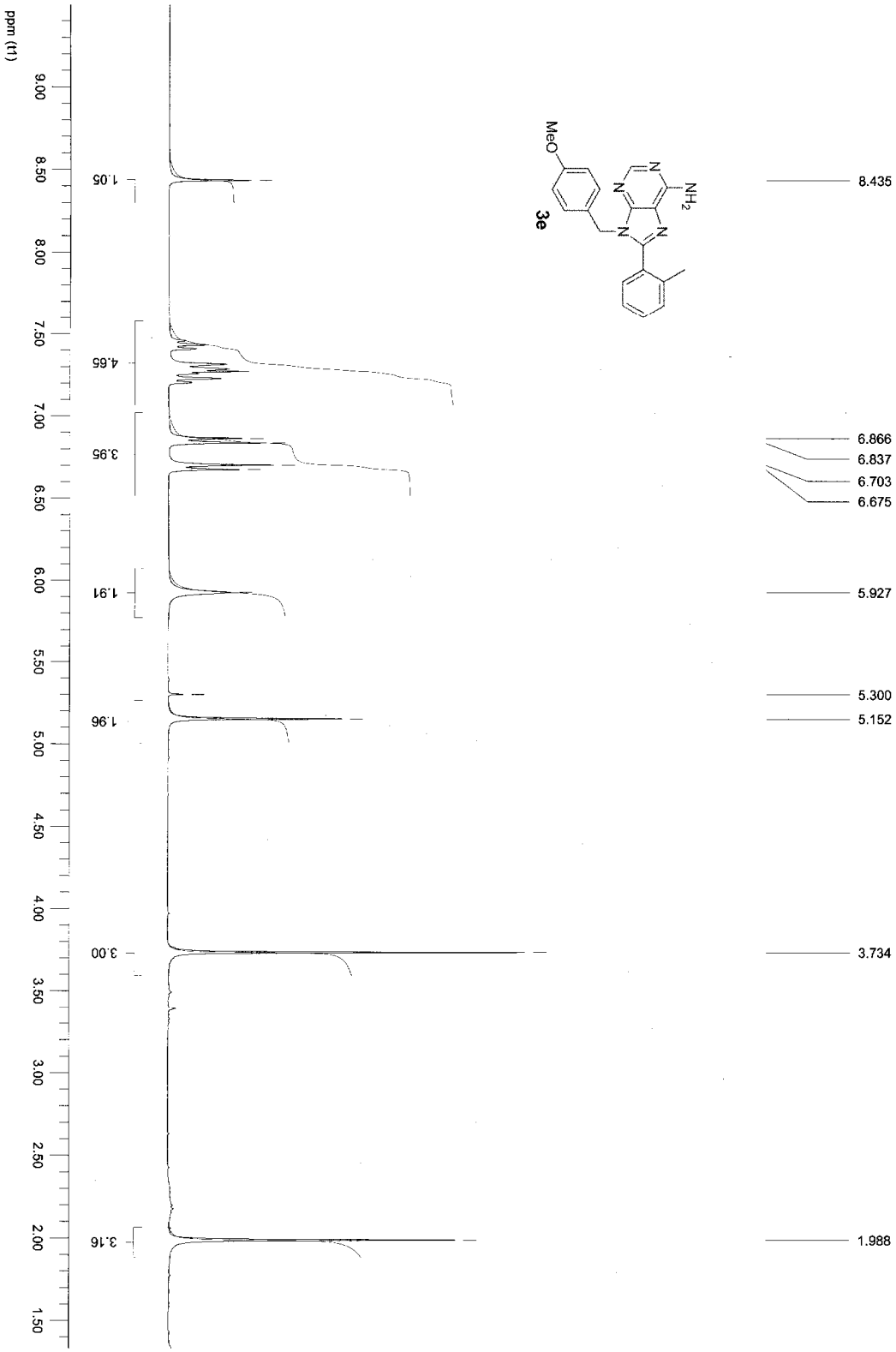
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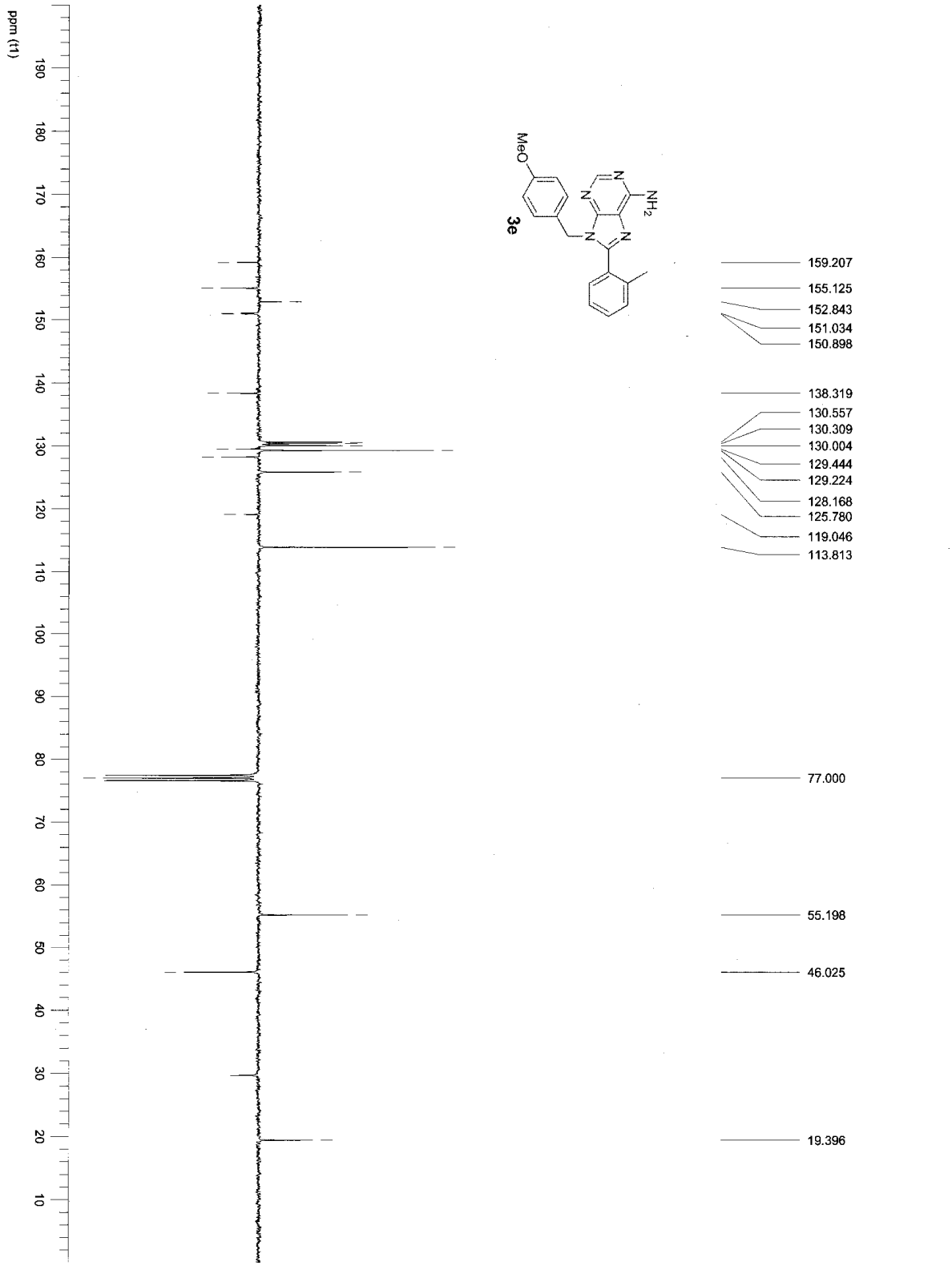
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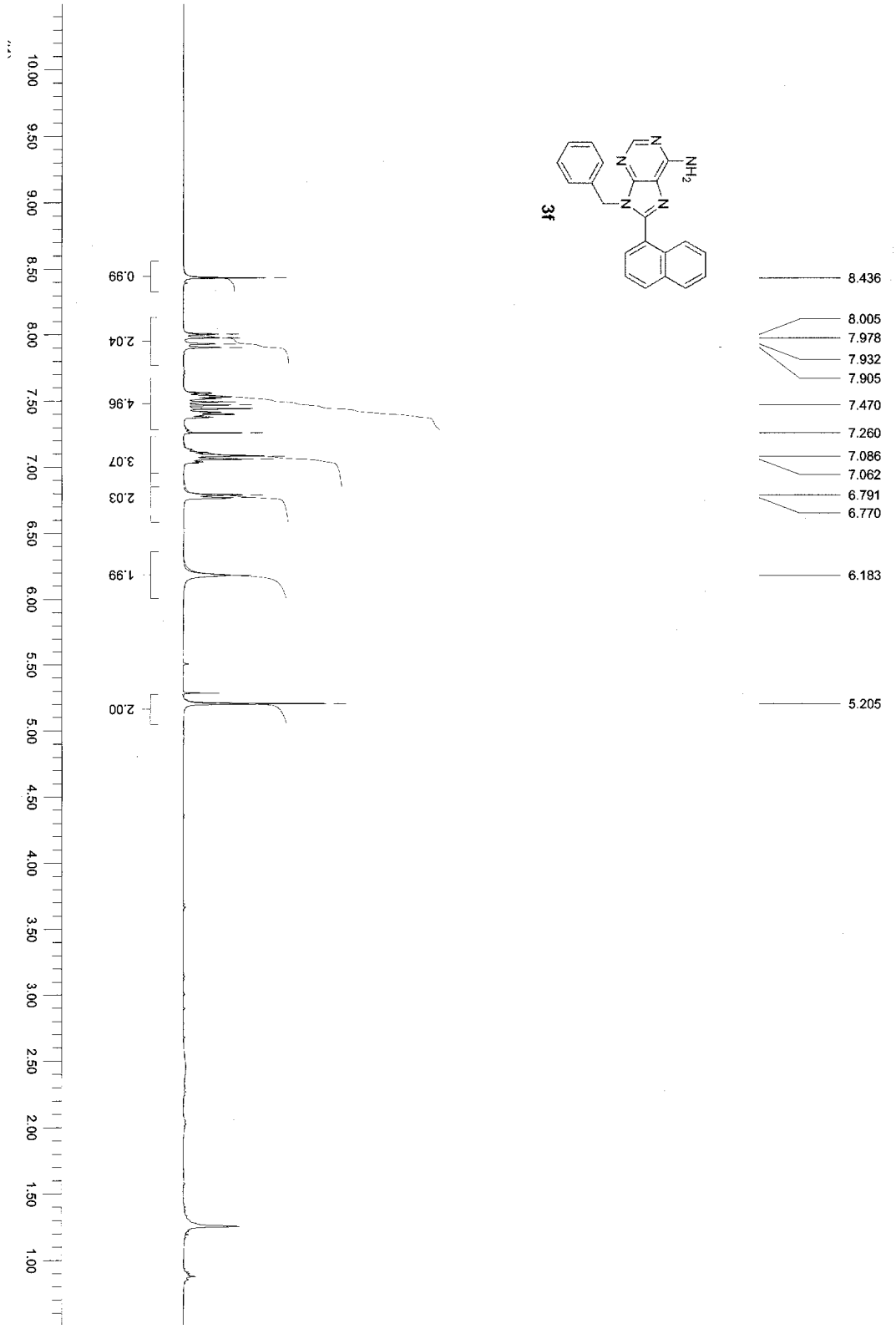
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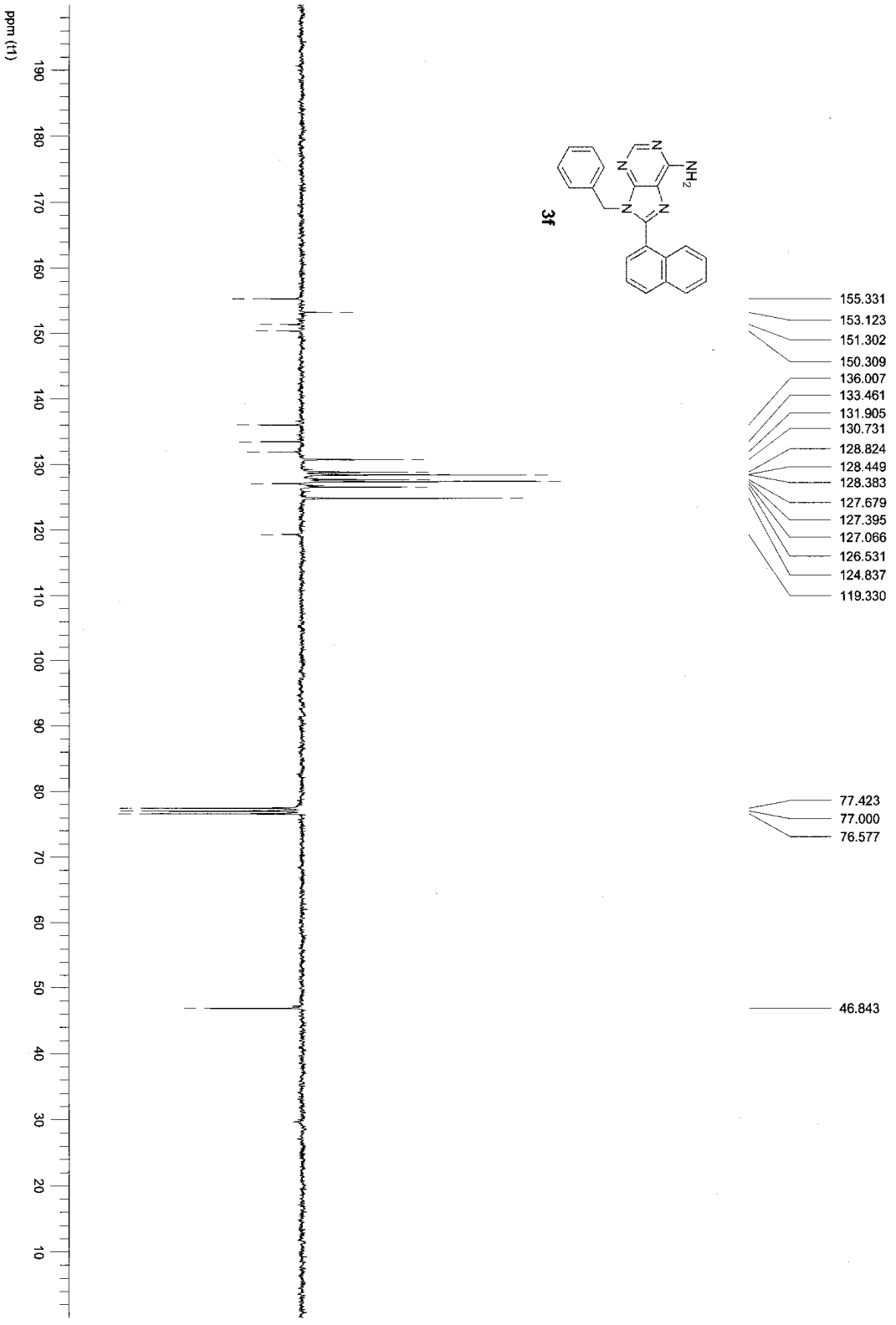
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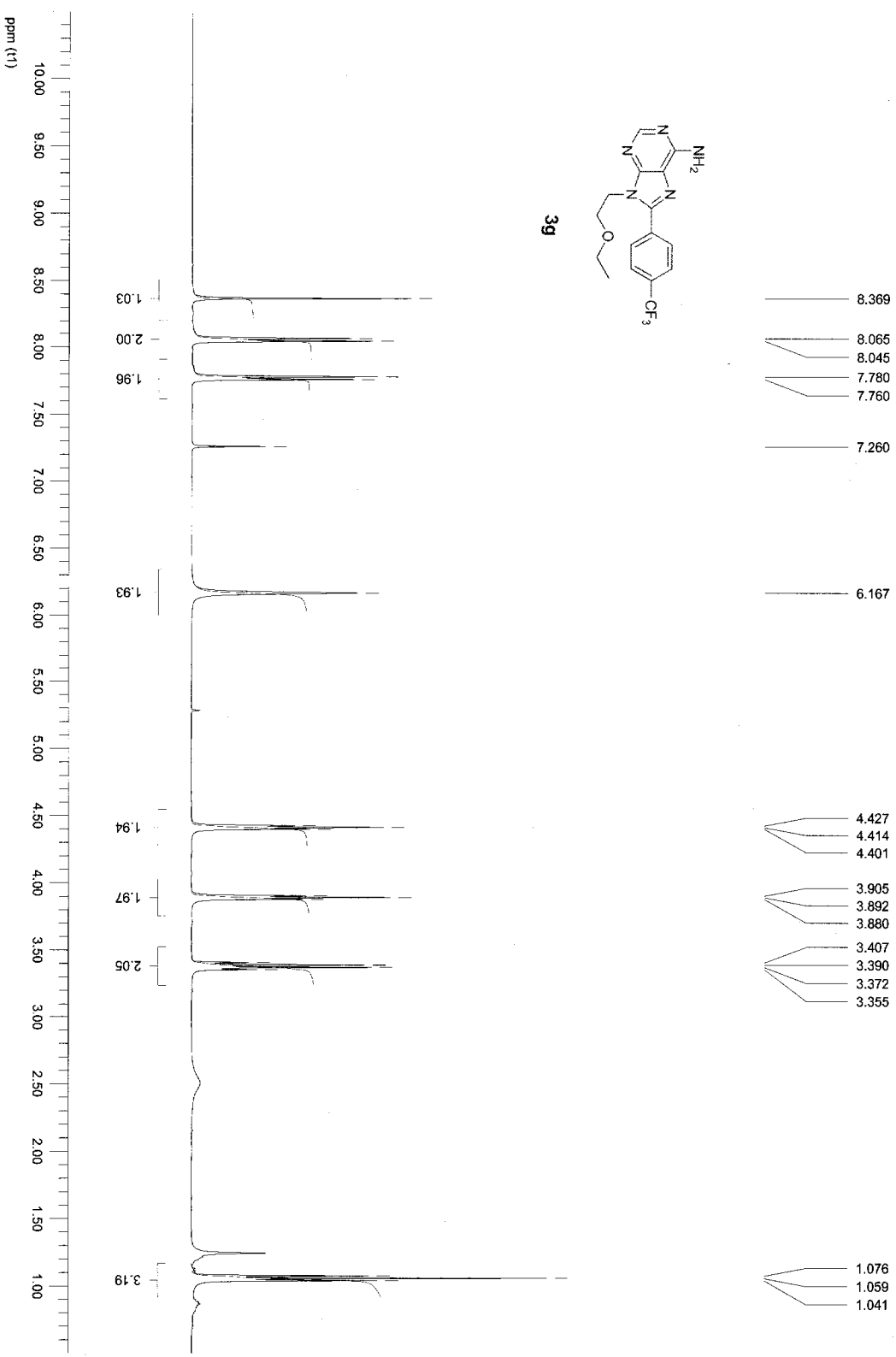
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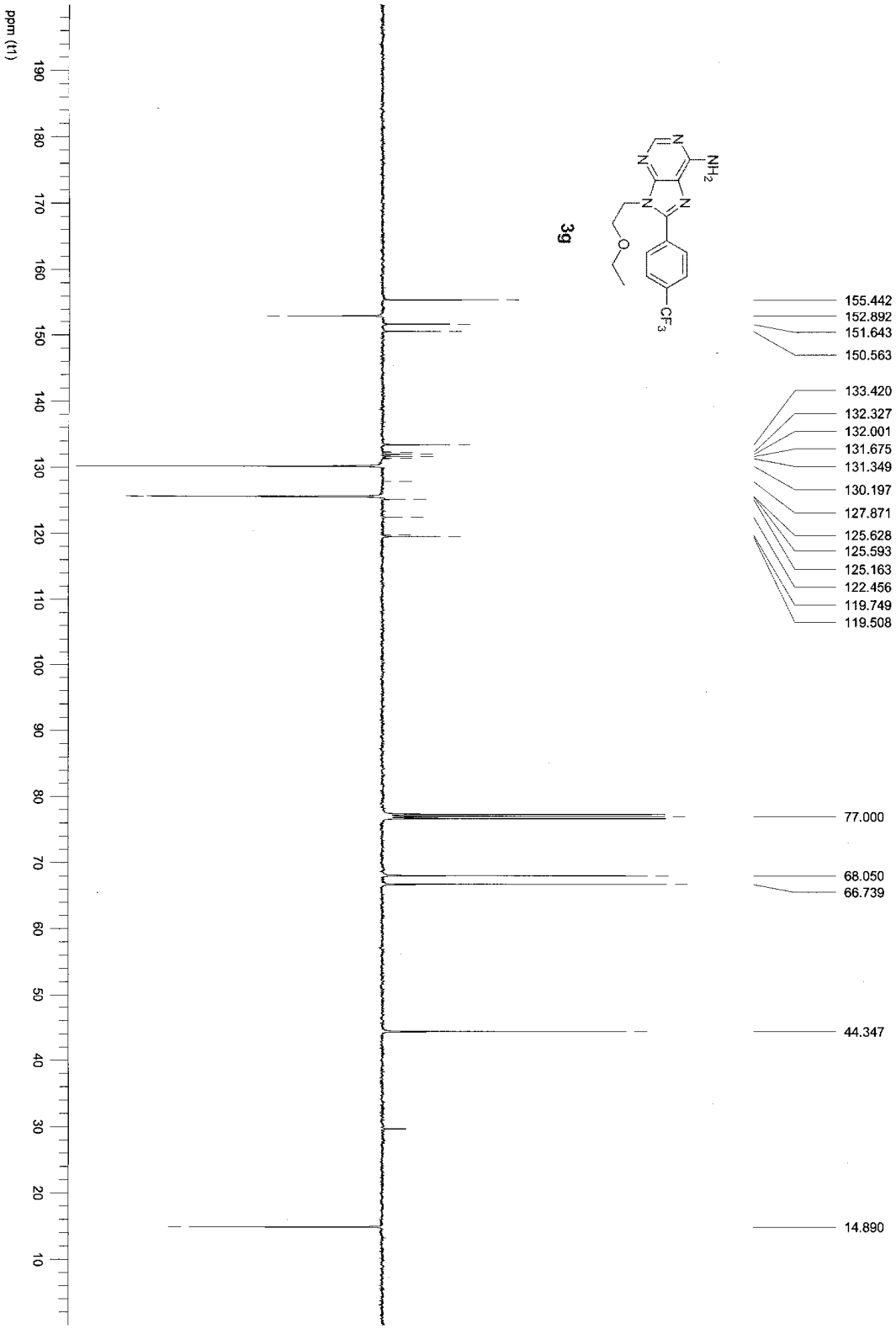
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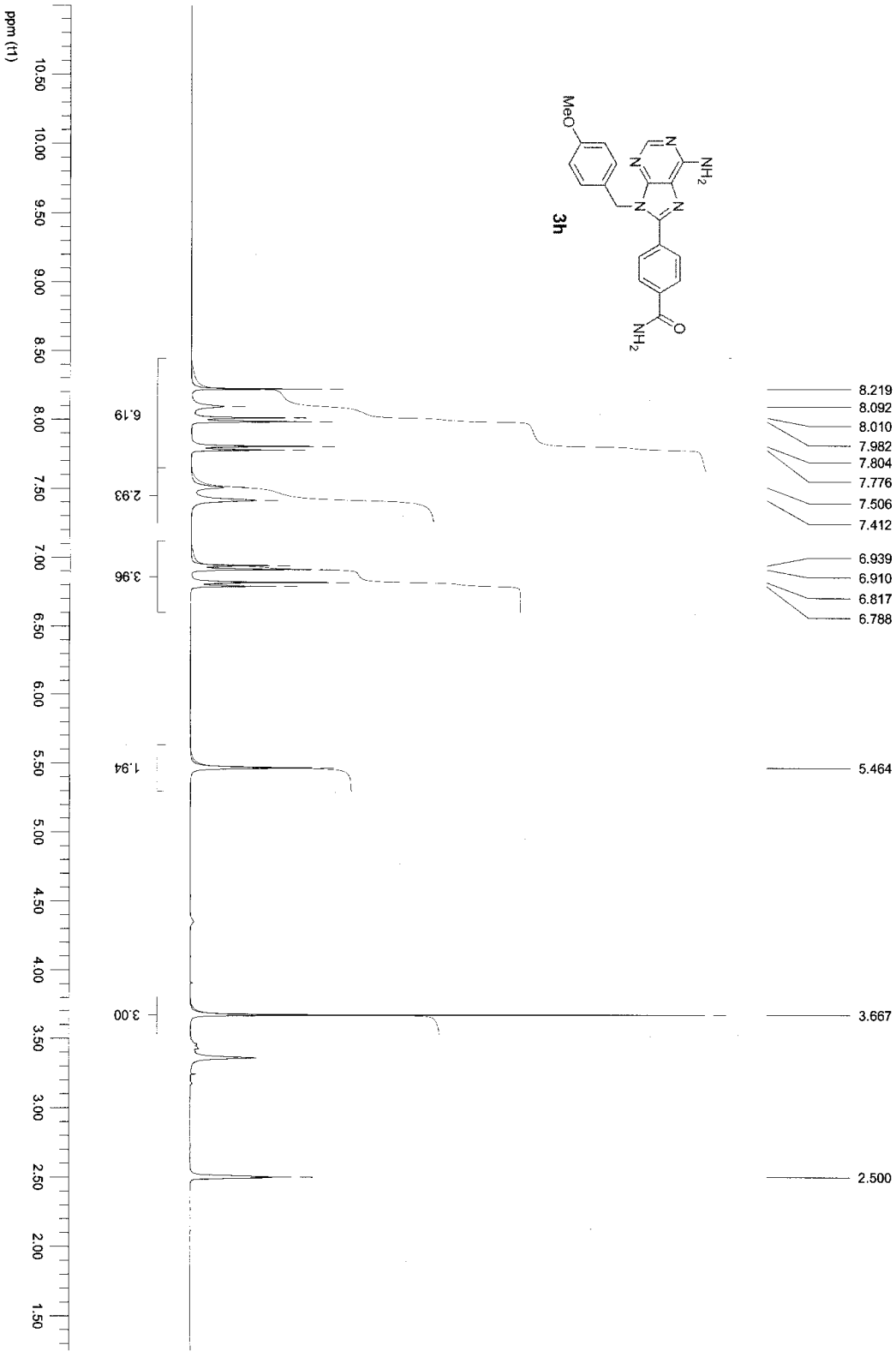
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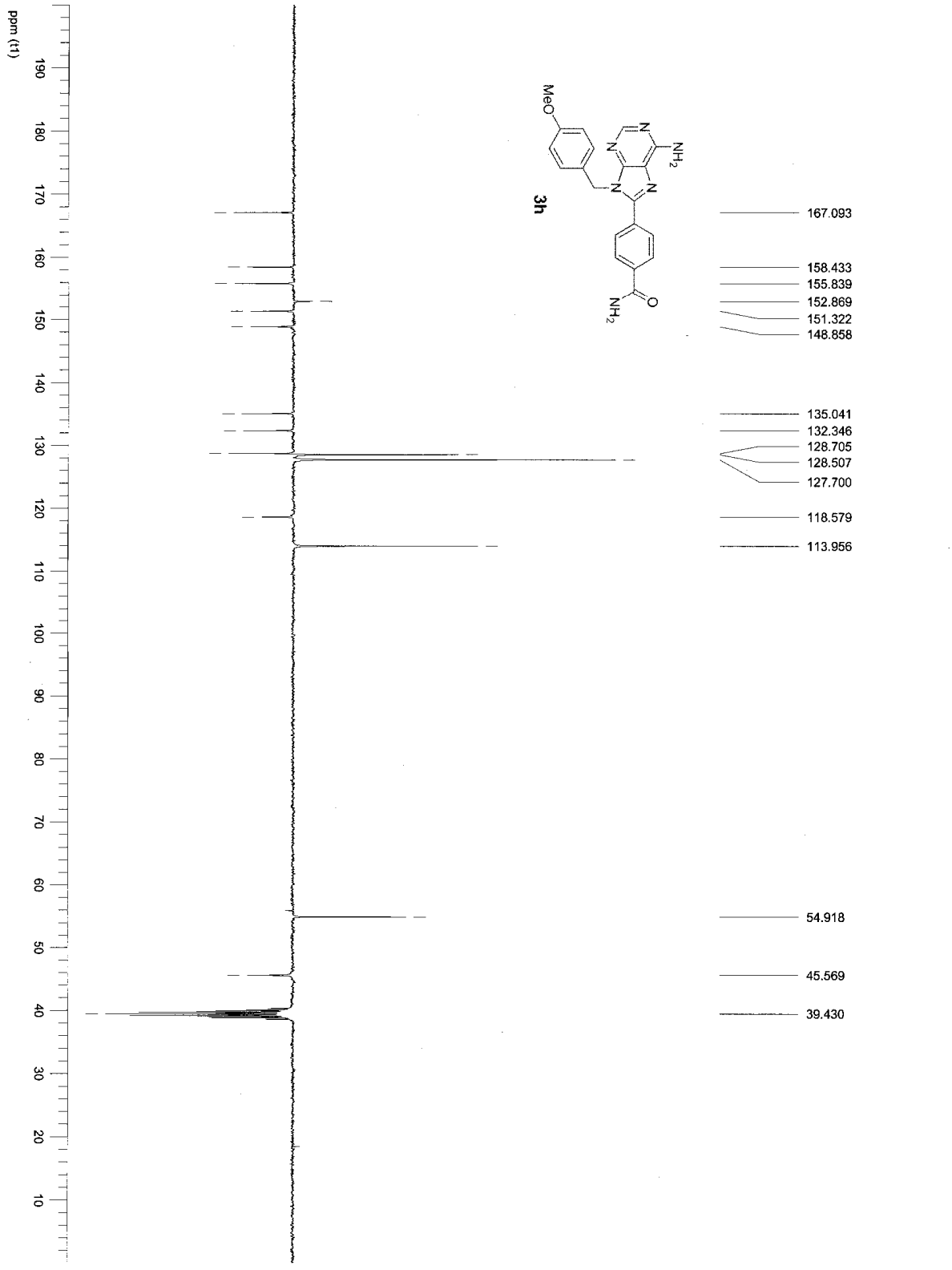
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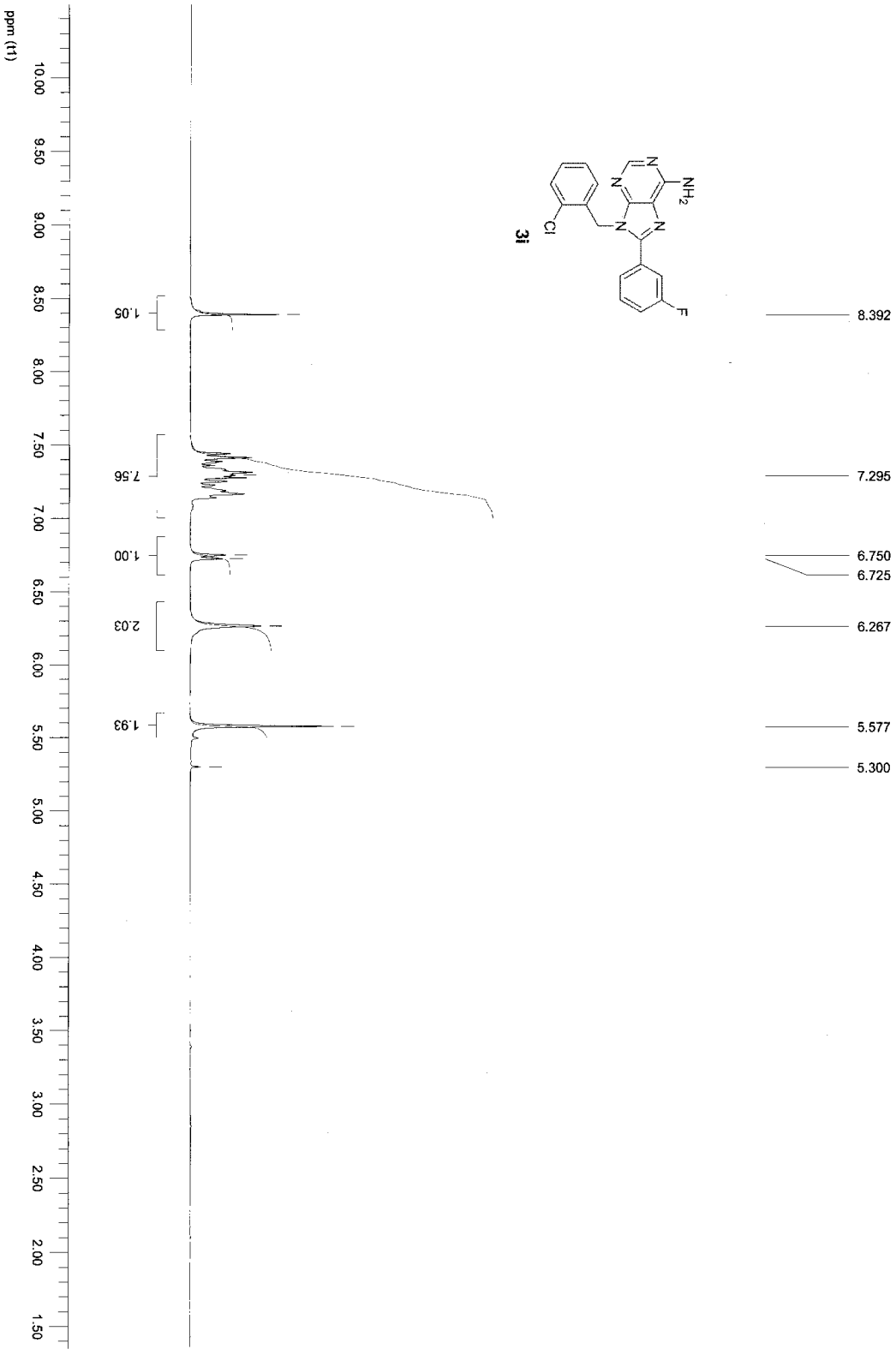
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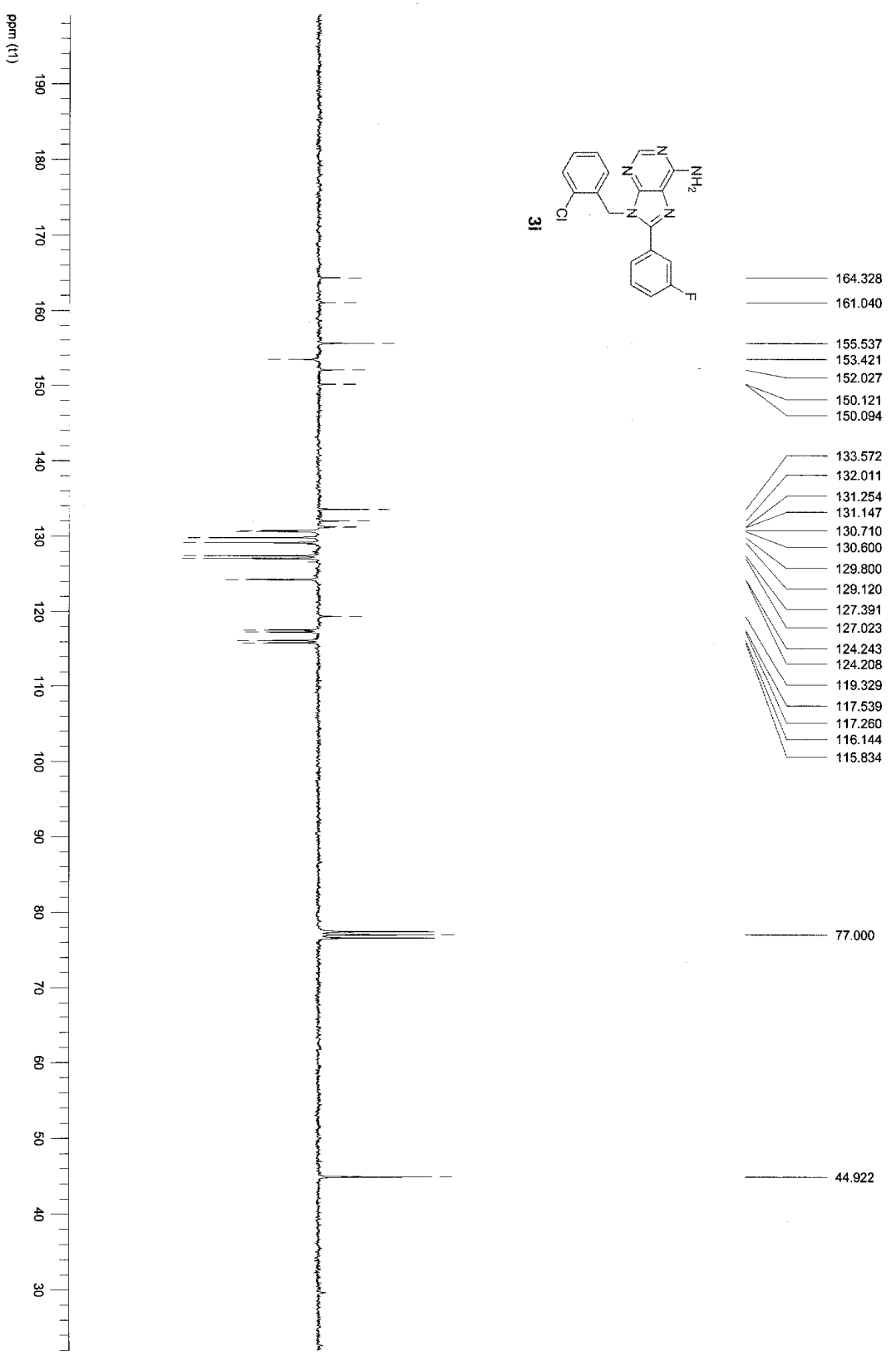
7.295
6.750
6.725
6.267
5.577
5.300
3i
Cl
F
NH2
N


ppm (t1)
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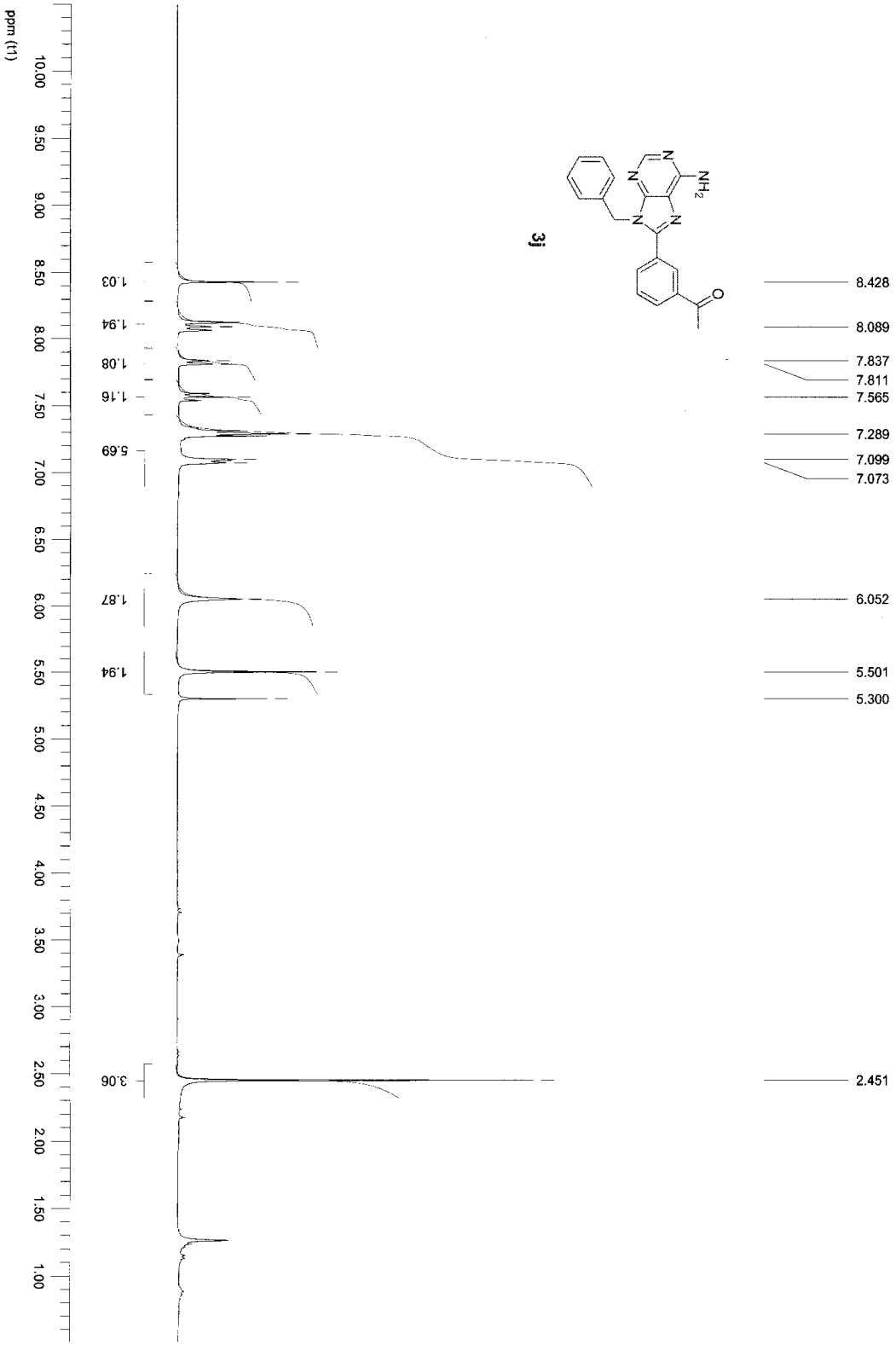
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
3i
Cl
F
NH2
164.328
161.040
155.537
153.421
152.027
150.121
150.094
133.572
132.011
131.254
131.147
130.710
130.600
129.800
129.120
127.391
127.023
124.243
124.208
119.329
117.539
117.260
116.144
115.834
77.000
44.922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9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
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4.00
3.50
3.00
2.50
2.00
1.50
1.00
1.03
1.94
1.08
1.16
5.69
1.87
1.94
3.06
3i
8.428
8.089
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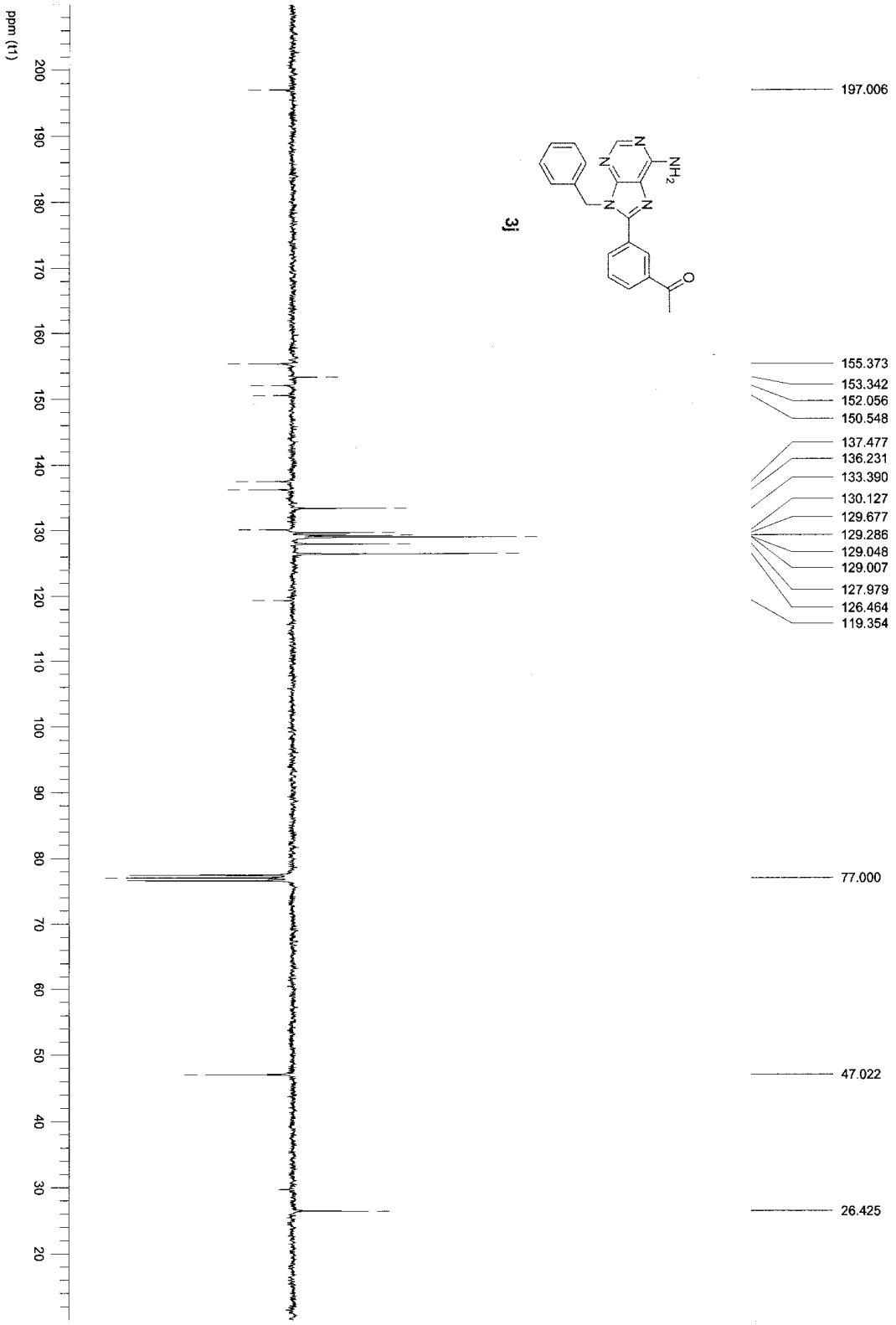
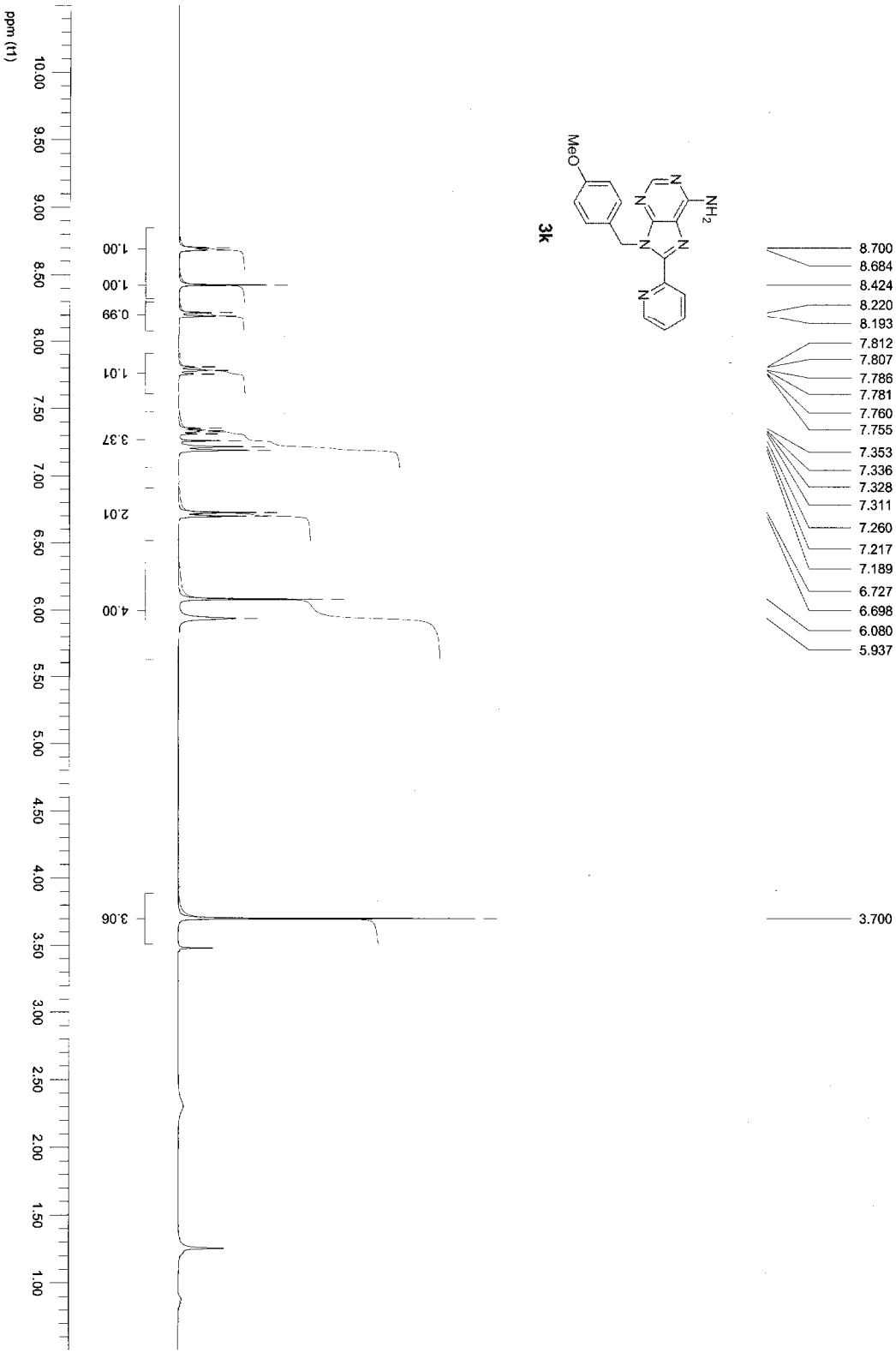
7.811
7.565
7.289
7.099
7.073
6.052
5.501
5.300
2.451


ppm (t1)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
3j
N
N
N
N
NH2
O
197.006
155.373
153.342
152.056
150.548
137.477
136.231
133.390
130.127
129.677
129.286
129.048
129.007
127.979
126.464
119.354
77.000
47.022
26.425


ppm (t1)
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9.00
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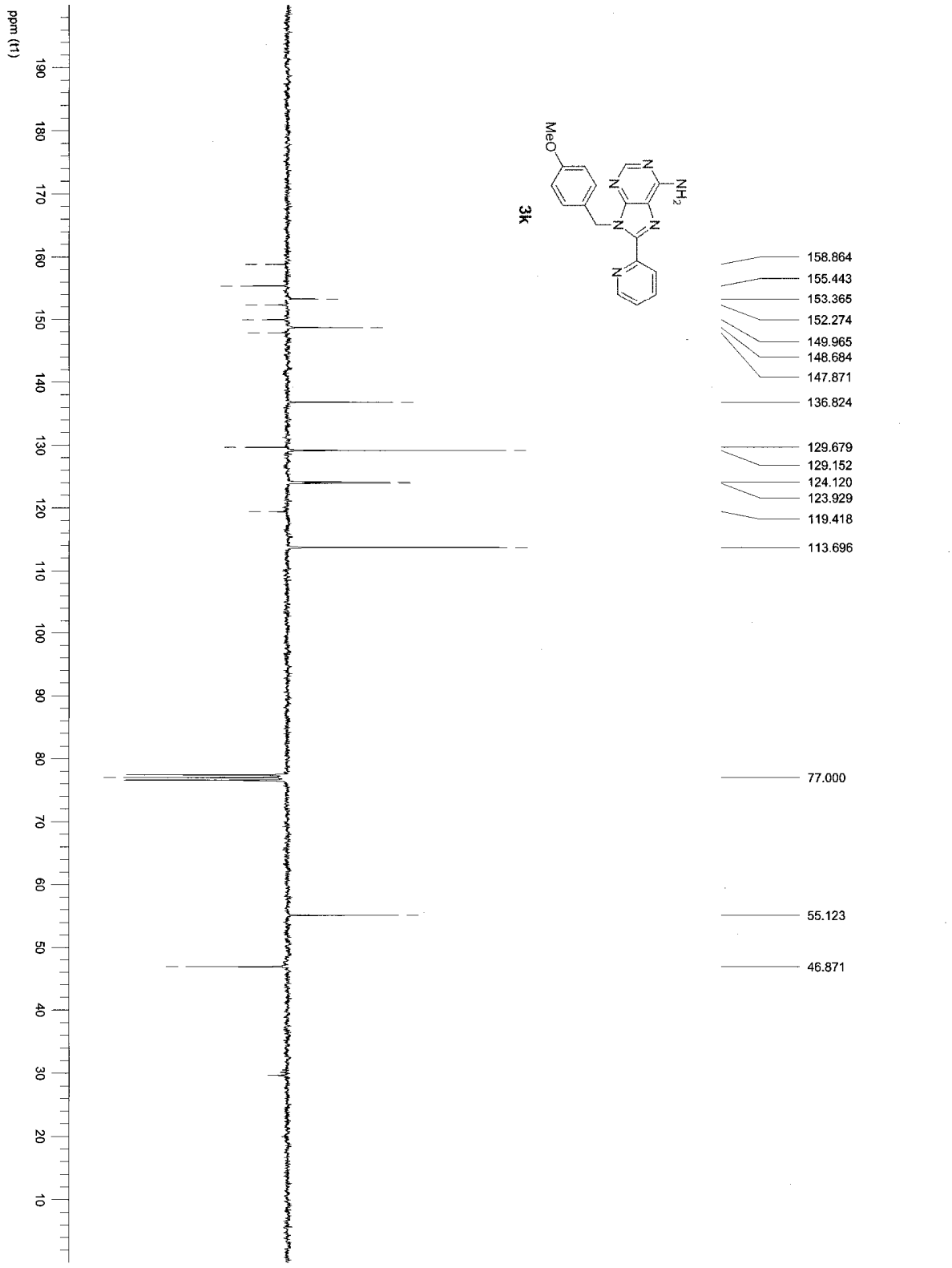
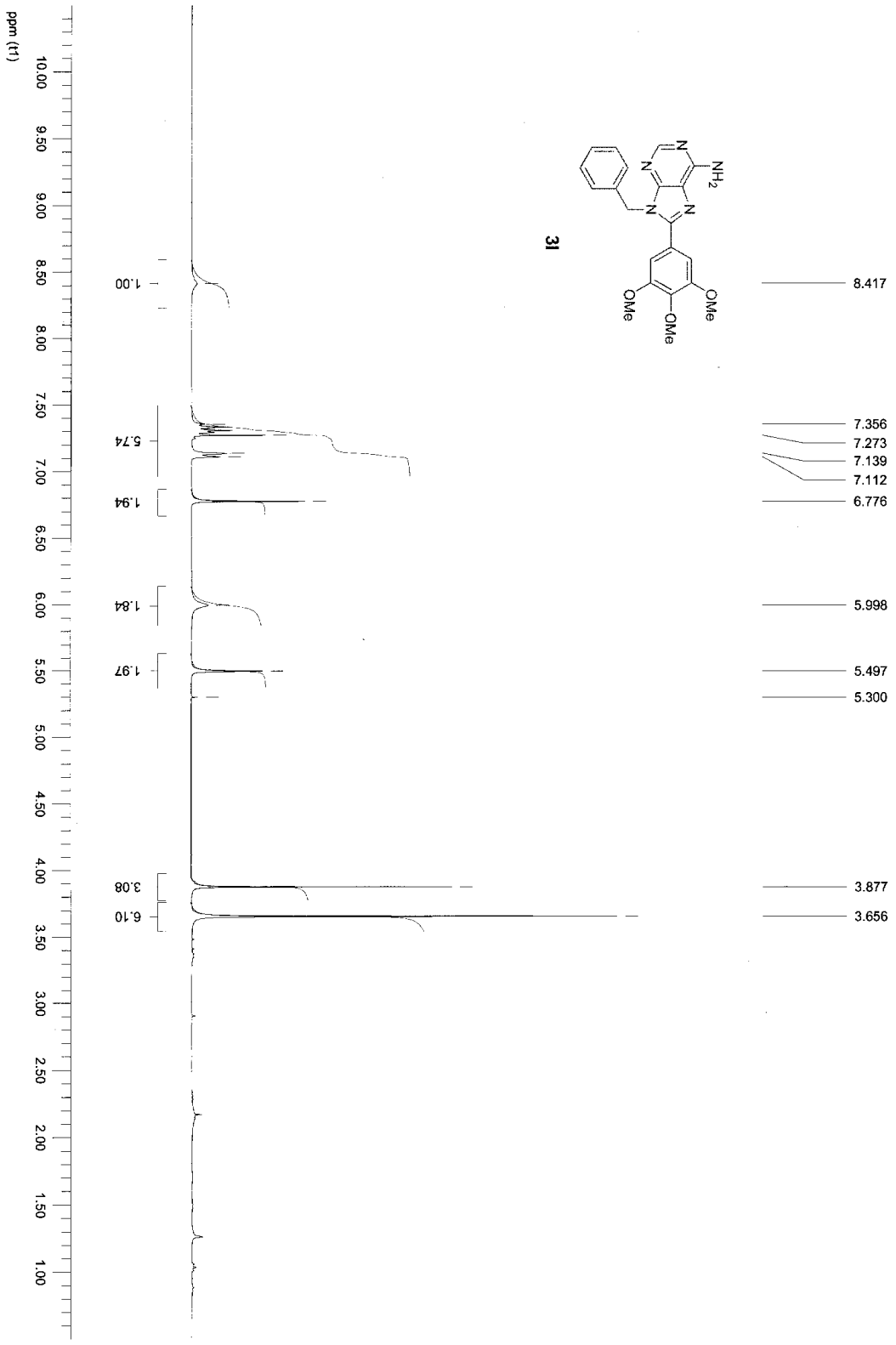
8.00
7.50
7.00
6.50
6.00
5.50
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
1.00
1.00
0.99
1.01
3.37
2.01
4.00
3.06
MeO
N
N
N
N
N
NH2
3k
8.700
8.684
8.424
8.220
8.193
7.812
7.807
7.786
7.781
7.760
7.755
7.353
7.336
7.328
7.311
7.260
7.217
7.189
6.727
6.698
6.080
5.937
3.700


ppm (t1)
MeO
NH2
3k
158.864
155.443
153.365
152.274
149.965
148.684
147.871
136.824
129.679
129.152
124.120
123.929
119.418
113.696
77.000
55.123
46.871


ppm (t1)
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9.50
9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
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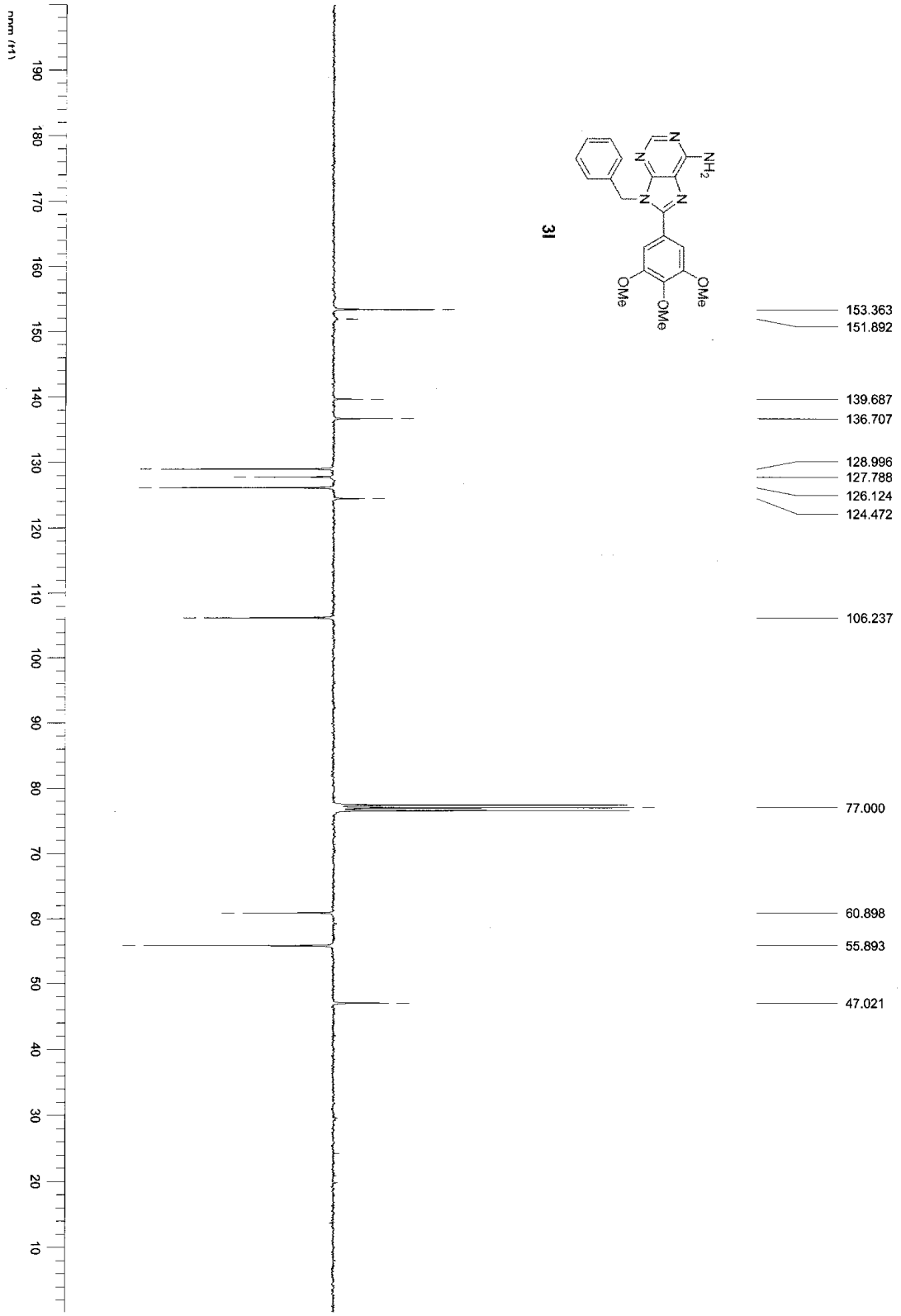
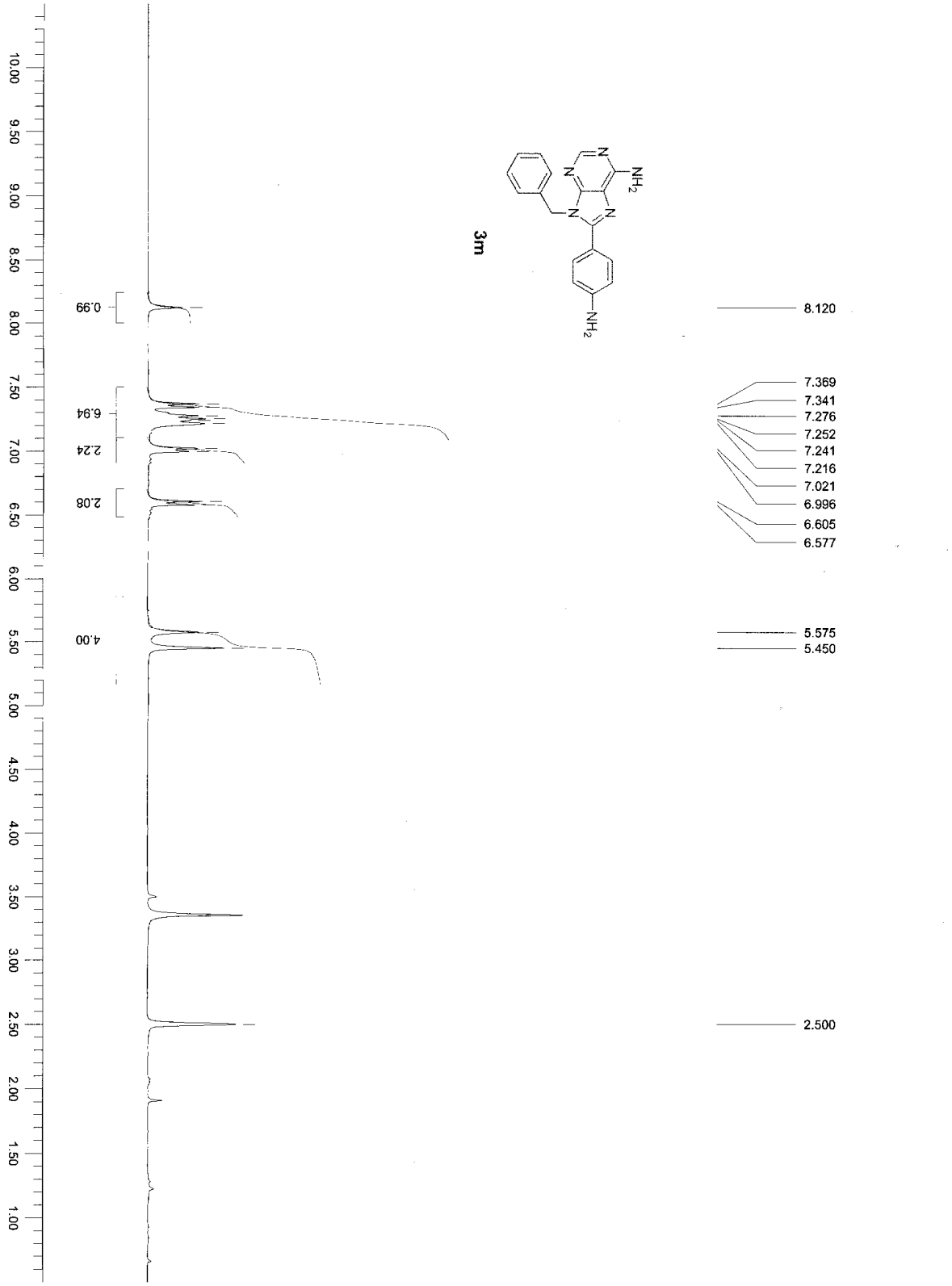
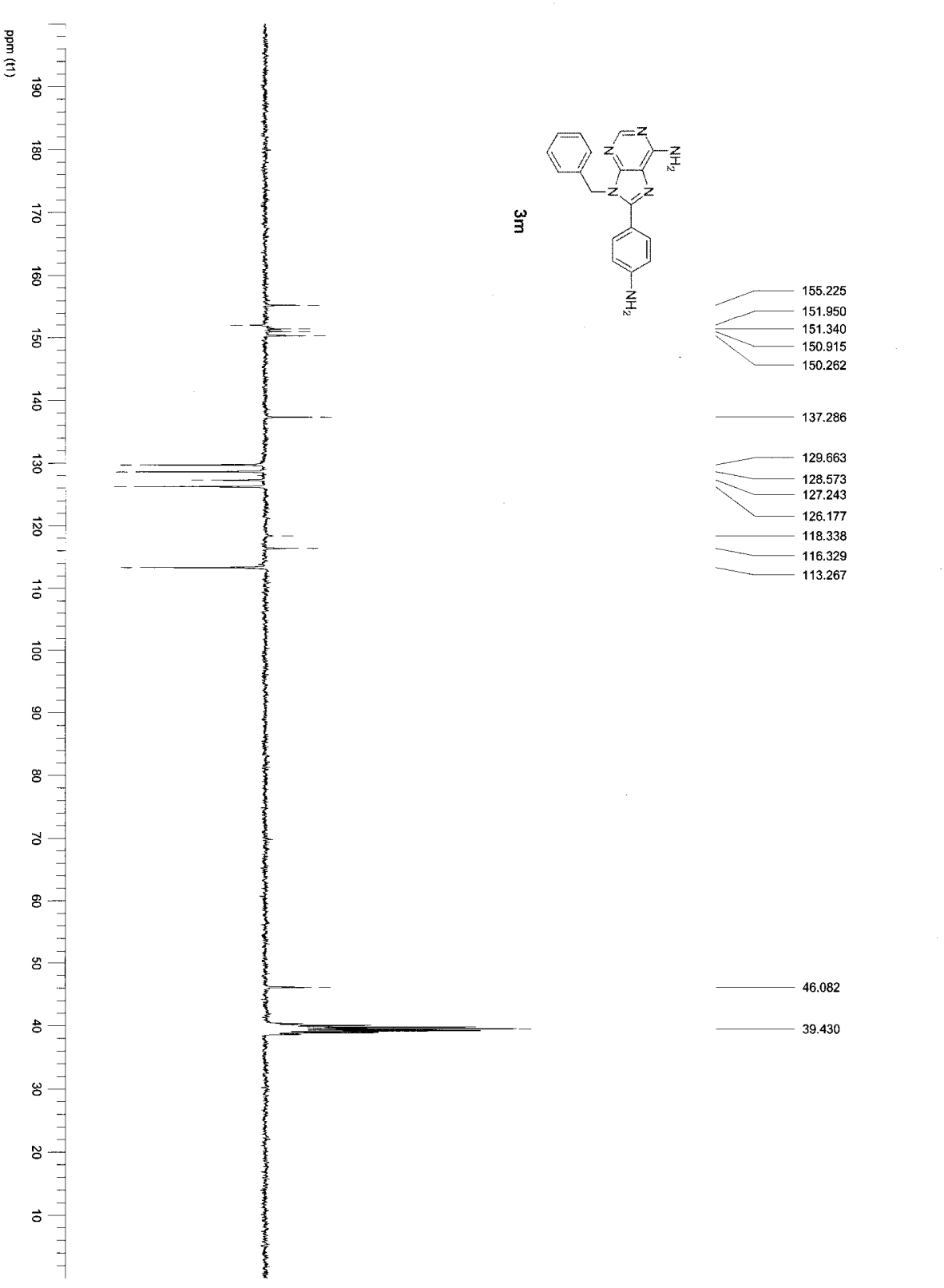
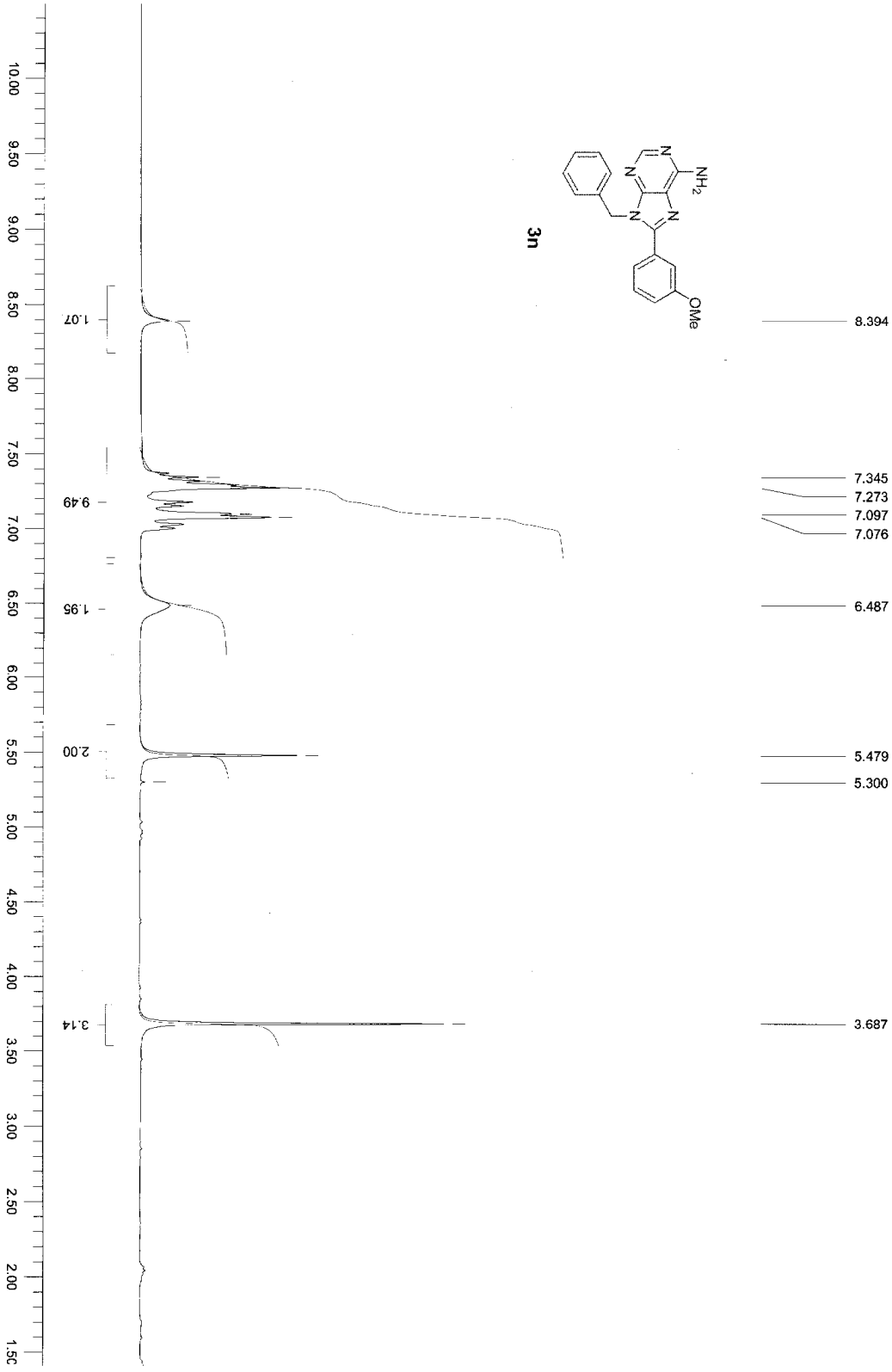
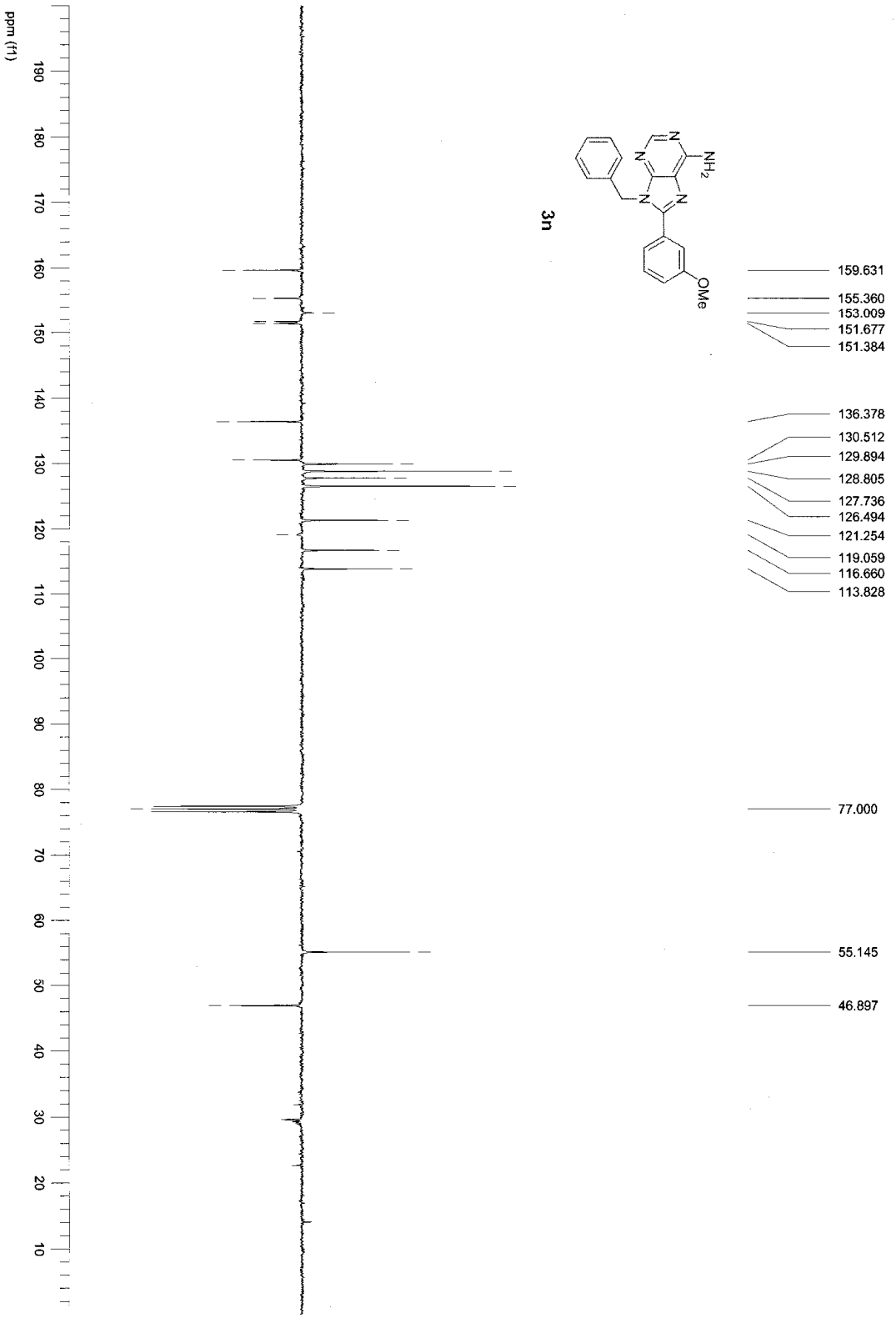
4.00
3.50
3.00
2.50
2.00
1.50
1.00
1.00
5.74
1.94
1.84
1.97
3.08
6.10
31
NH2
OMe
OMe
OMe
8.417
7.356
7.273
7.139
7.112
6.776
5.998
5.497
5.300
3.877
3.656

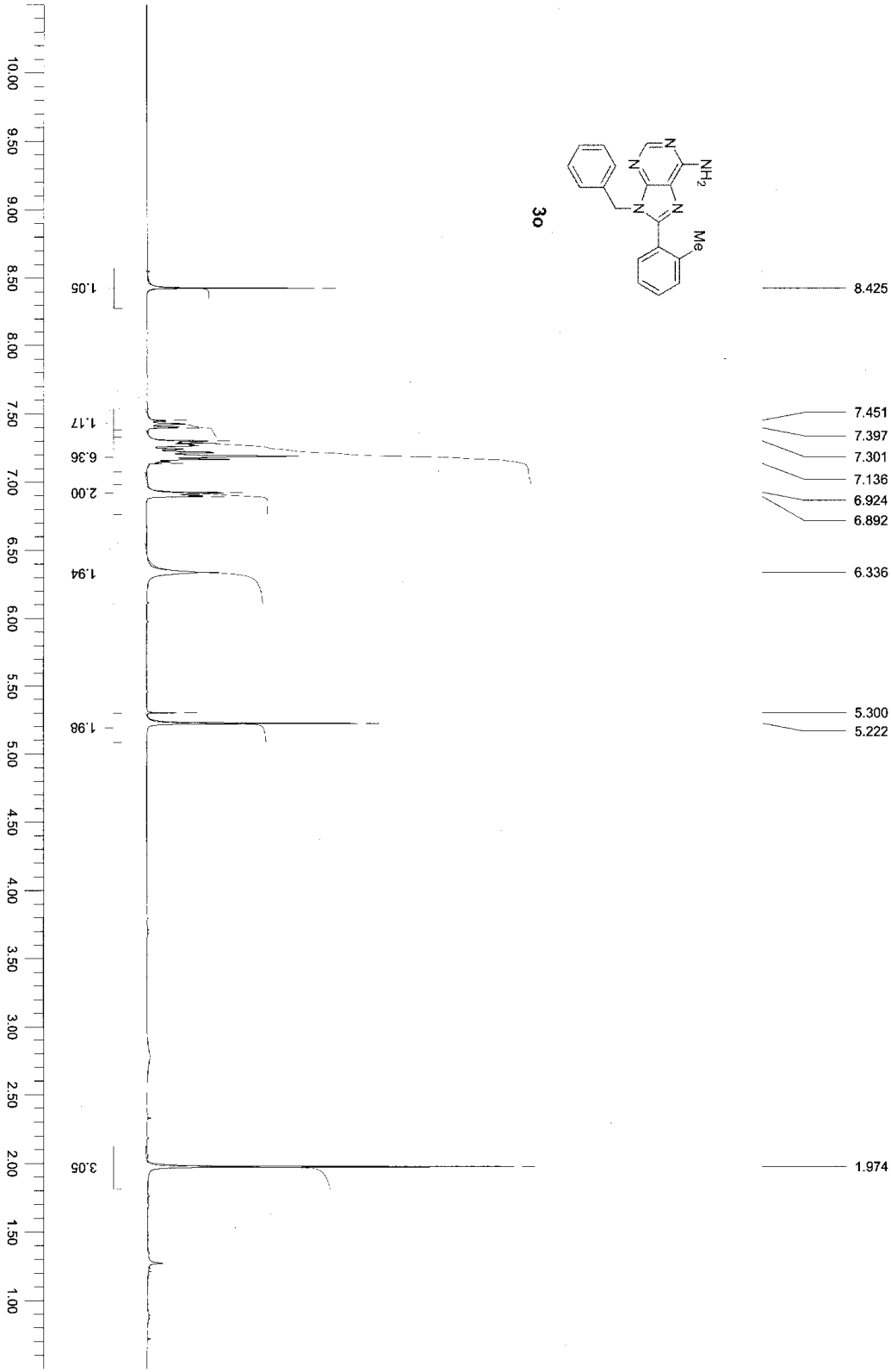

ppm (t1)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
3l
NH2
OMe
OMe
OMe
153.363
151.892
139.687
136.707
128.996
127.788
126.124
124.472
106.237
77.000
60.898
55.893
47.021

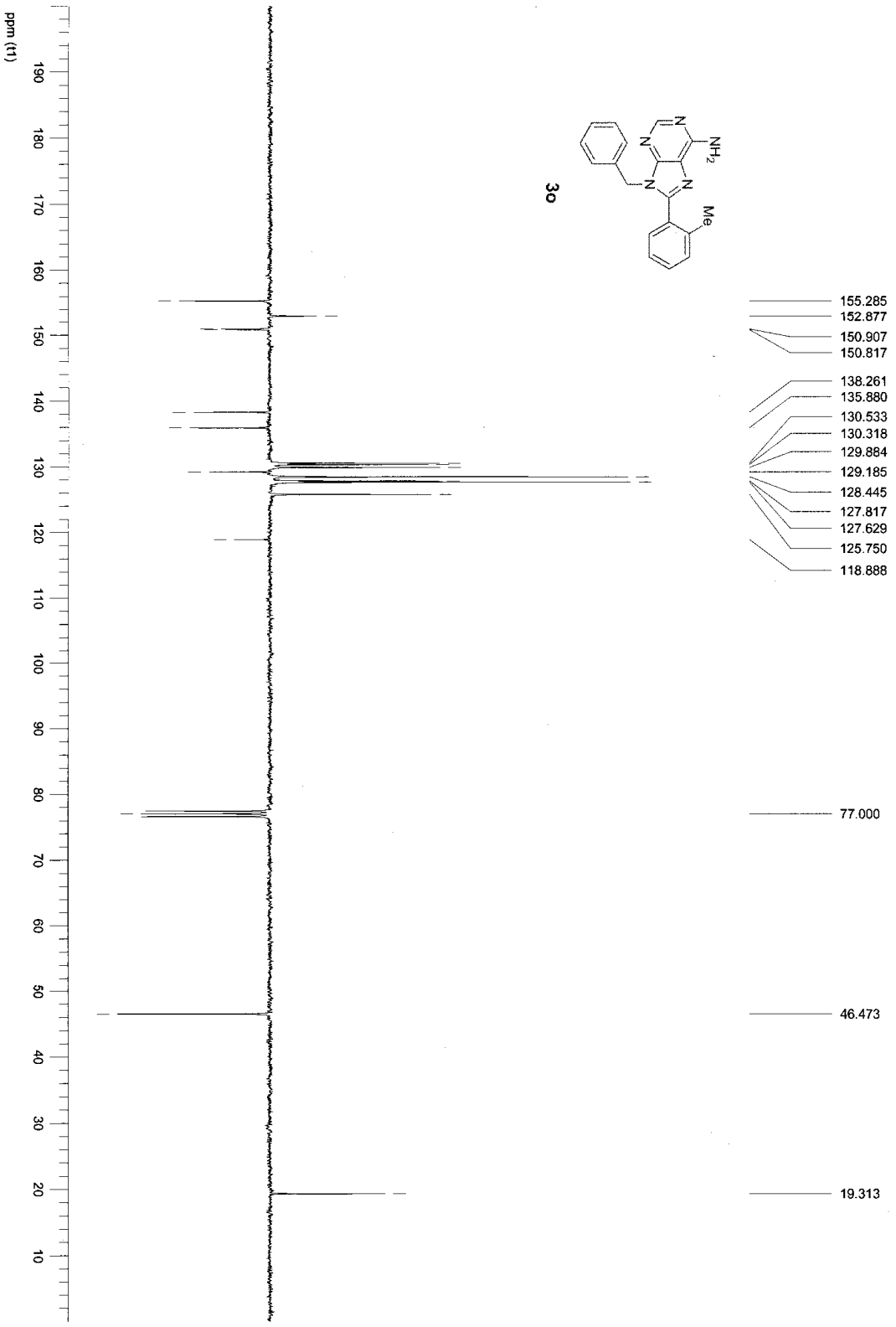

3m
8.120
7.369
7.341
7.276
7.252
7.241
7.216
7.021
6.996
6.605
6.577
5.575
5.450
2.500
0.99
6.94
2.24
2.08
4.00

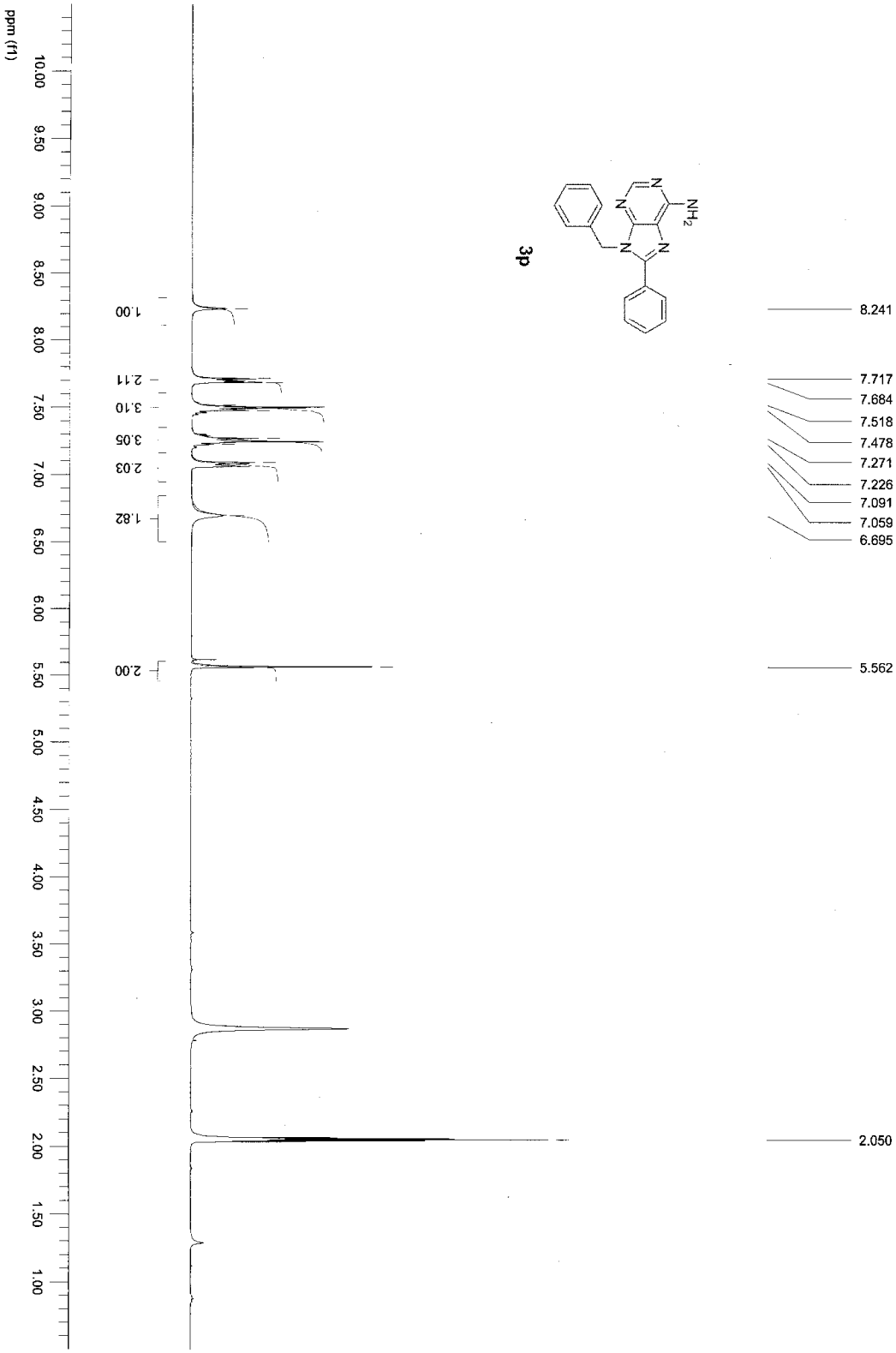

ppm (t1)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
3m
NH2
NH2
155.225
151.950
151.340
150.915
150.262
137.286
129.663
128.573
127.243
126.177
118.338
116.329
113.267
46.082
39.430


3n
NH2
OMe
8.394
7.345
7.273
7.097
7.076
6.487
5.479
5.300
3.687
1.07
9.49
1.95
2.00
3.14


ppm (f1)
3n
159.631
155.360
153.009
151.677
151.384
136.378
130.512
129.894
128.805
127.736
126.494
121.254
119.059
116.660
113.828
77.000
55.145
46.897


3o
NH2
Me
8.425
7.451
7.397
7.301
7.136
6.924
6.892
6.336
5.300
5.222
1.974
1.05
1.17
6.36
2.00
1.94
1.98
3.05


ppm (t1)
3o
NH2
Me
155.285
152.877
150.907
150.817
138.261
135.880
130.533
130.318
129.884
129.185
128.445
127.817
127.629
125.750
118.888
77.000
46.473
19.313


ppm (f1)
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9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
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4.00
3.50
3.00
2.50
2.00
1.50
1.00
1.00
2.11
3.10
3.05
2.03
1.82
2.00
3p
N
N
N
N
NH2
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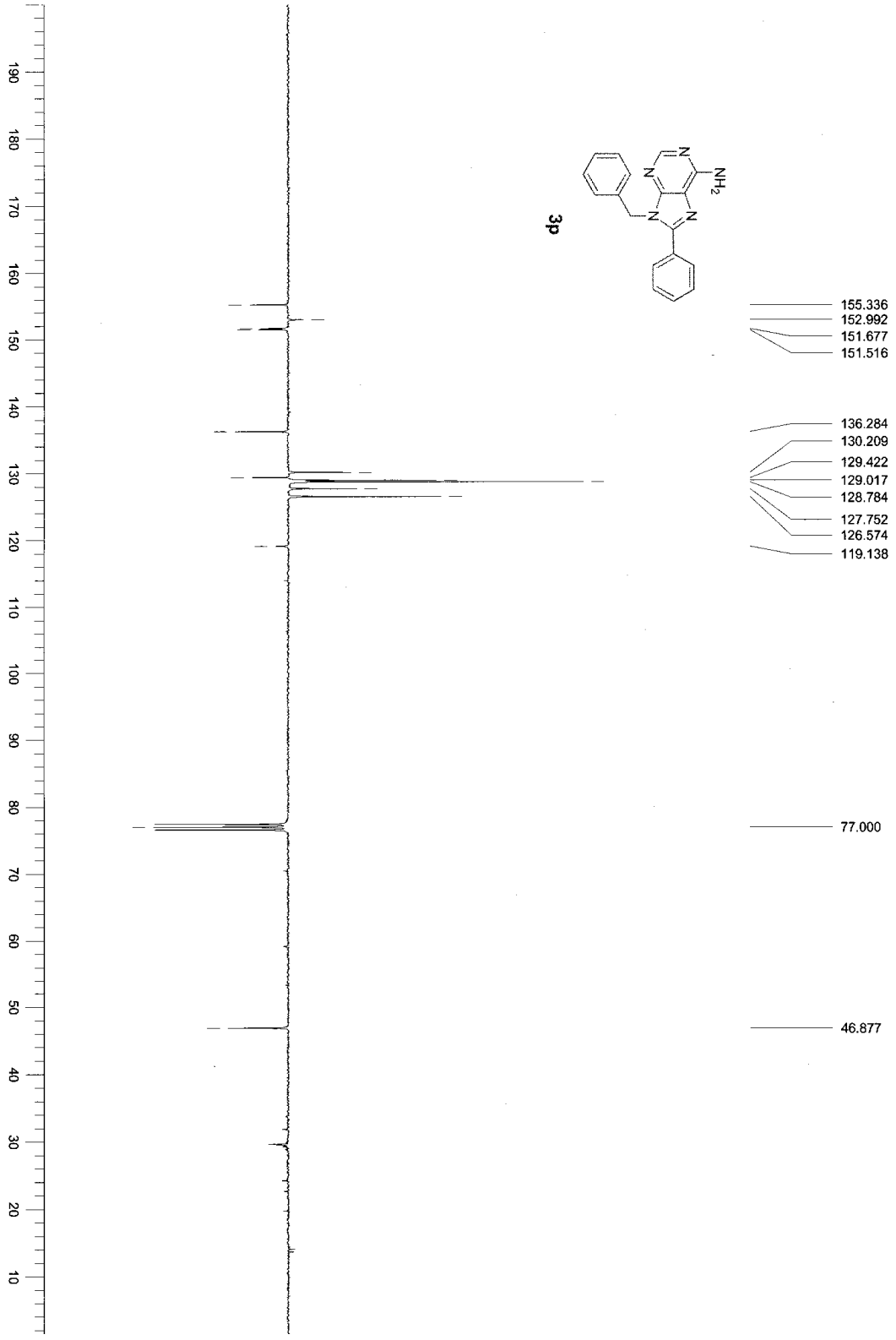
7.717
7.684
7.518
7.478
7.271
7.226
7.091
7.059
6.695
5.562
2.050


3p
155.336
152.992
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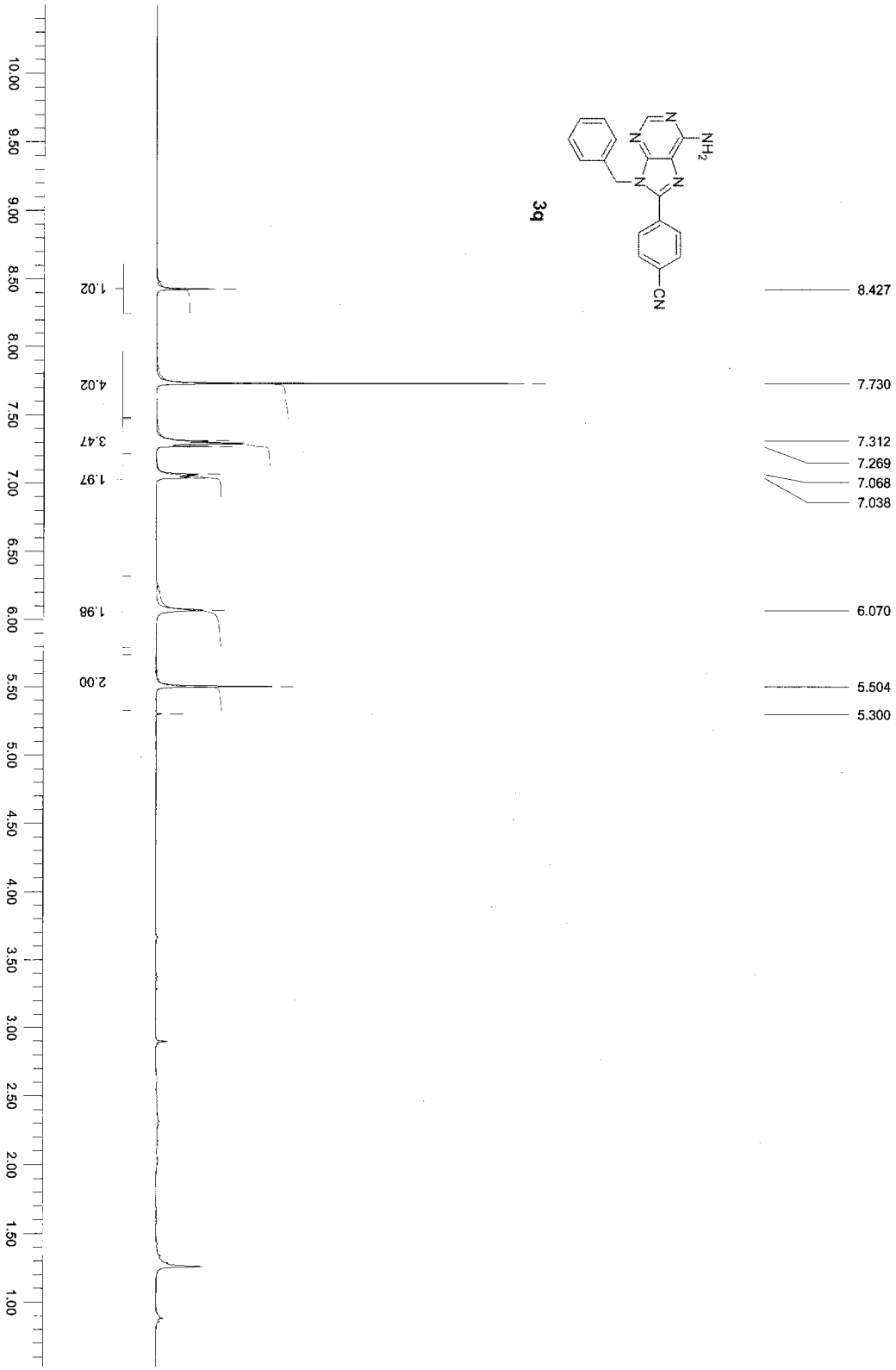
151.516
136.284
130.209
129.422
129.017
128.784
127.752
126.574
119.138
77.000
46.877


3q
NH2
CN
8.427
7.730
7.312
7.269
7.068
7.038
6.070
5.504
5.300
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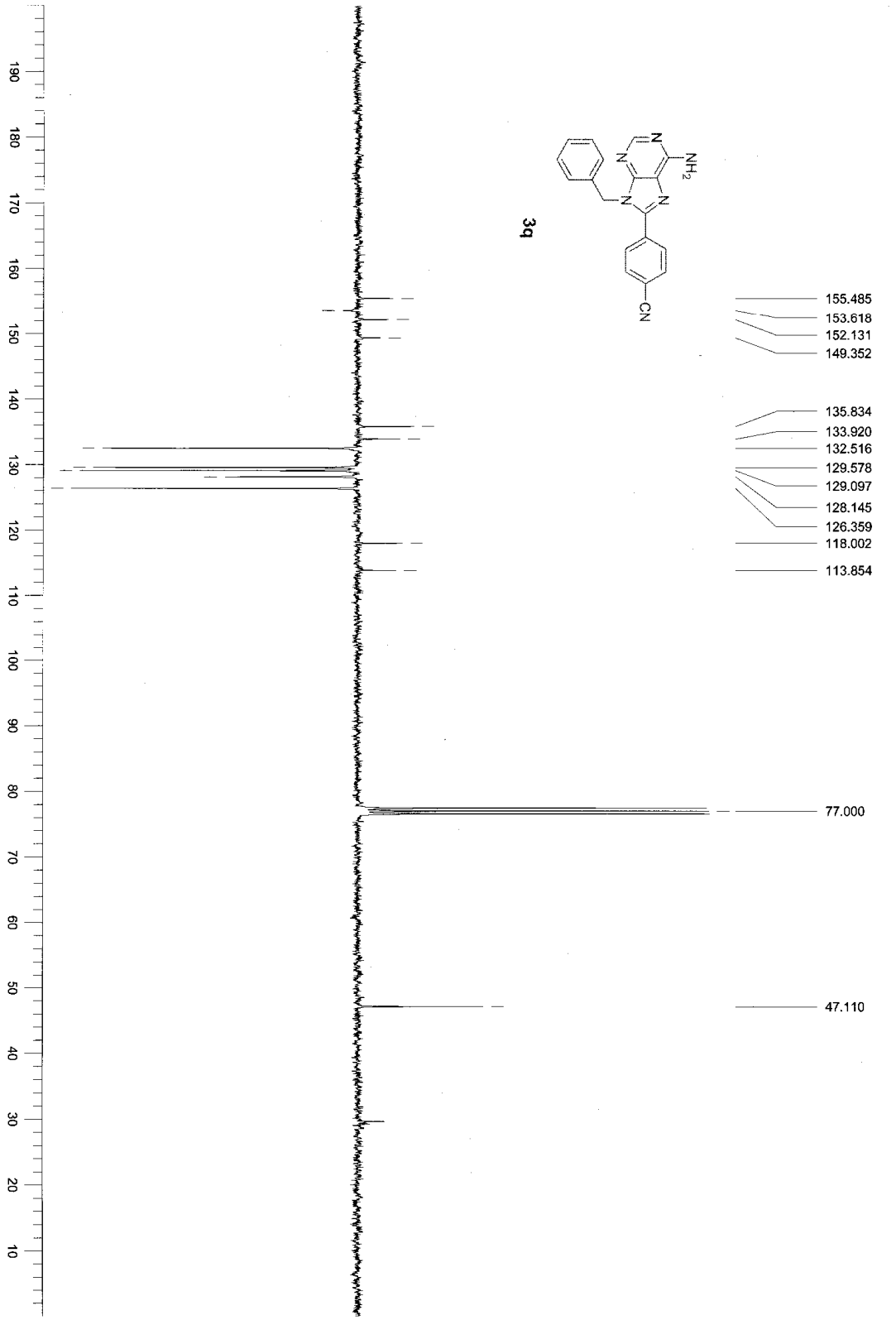
4.02
3.47
1.97
1.98
2.00


3q
NH2
CN
155.485
153.618
152.131
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133.920
132.516
129.578
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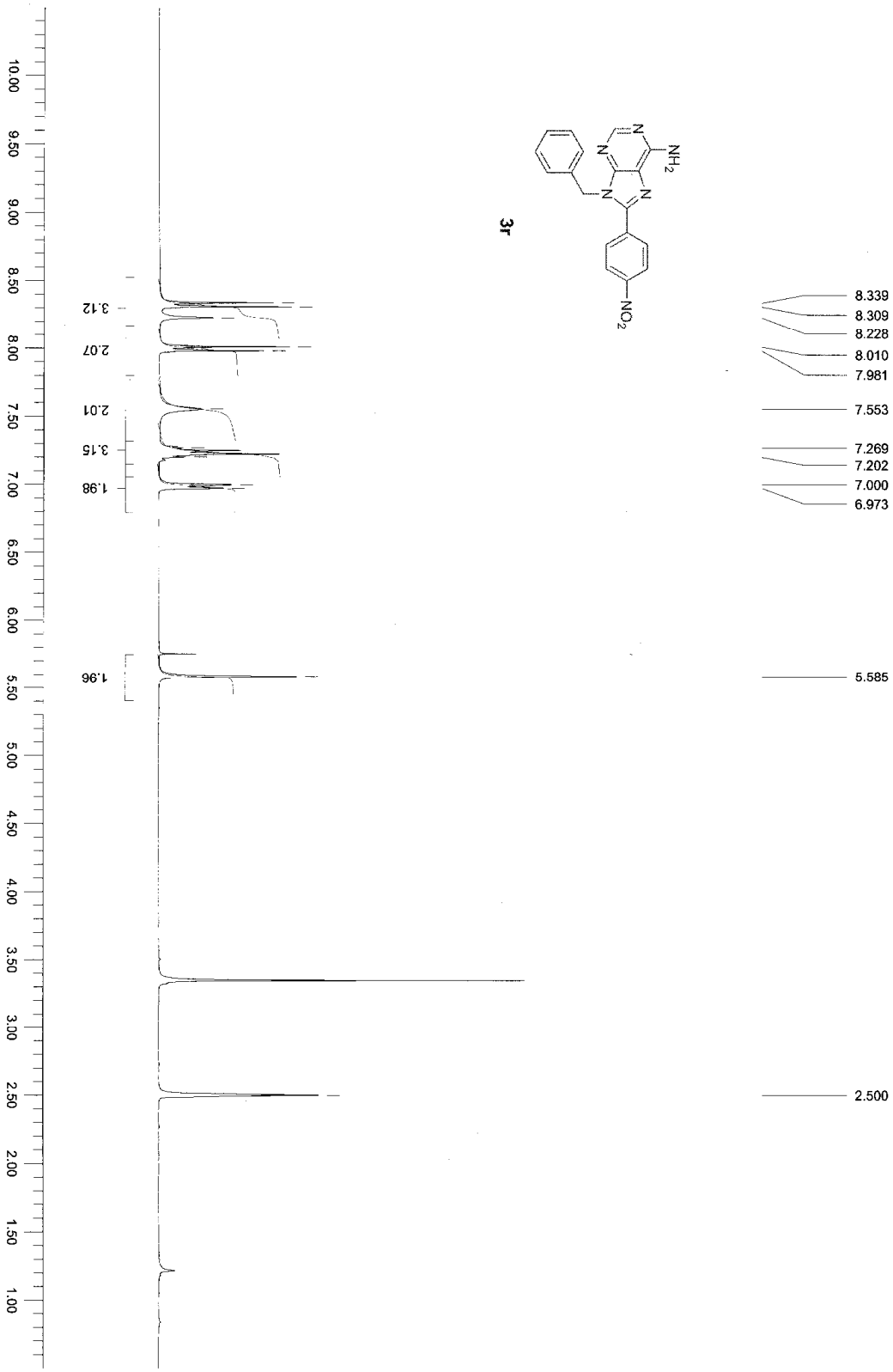
126.359
118.002
113.854
77.000
47.110


3r
NH2
NO2
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8.309
8.228
8.010
7.981
7.553
7.269
7.202
7.000
6.973
5.585
2.500
3.12
2.07
2.01
3.15
1.98
1.96
10.00
9.50
9.00
8.50
8.00
7.50
7.00
6.50
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4.50
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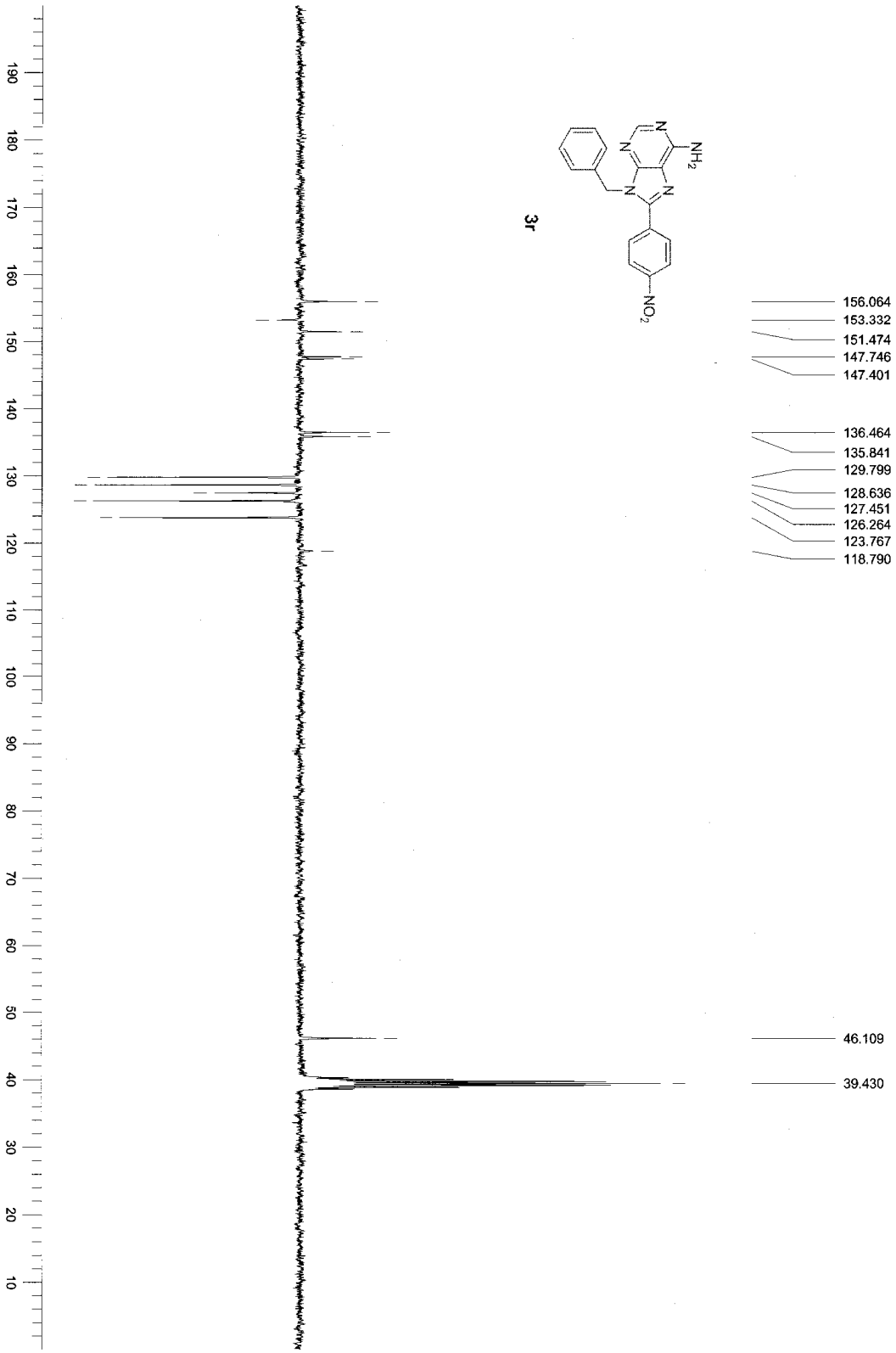
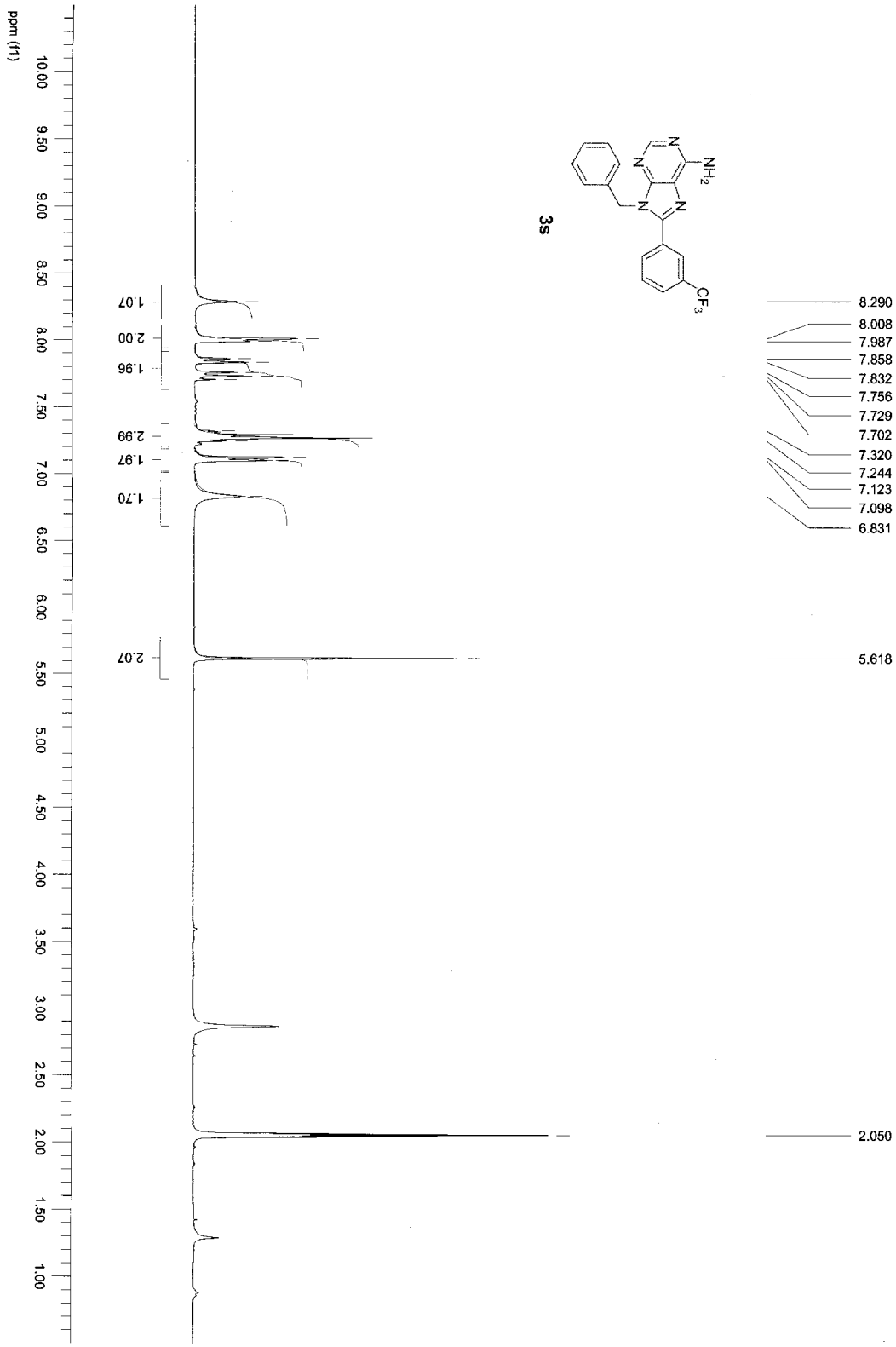
3.00
2.50
2.00
1.50
1.00


3r
156.064
153.332
151.474
147.746
147.401
136.464
135.841
129.799
128.636
127.451
126.264
123.767
118.790
46.109
39.430


ppm (f1)
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9.00
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8.00
7.50
7.00
6.50
6.00
5.50
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3.50
3.00
2.50
2.00
1.50
1.00
1.07
2.00
1.96
2.99
1.97
1.70
2.07
3s
NH2
CF3
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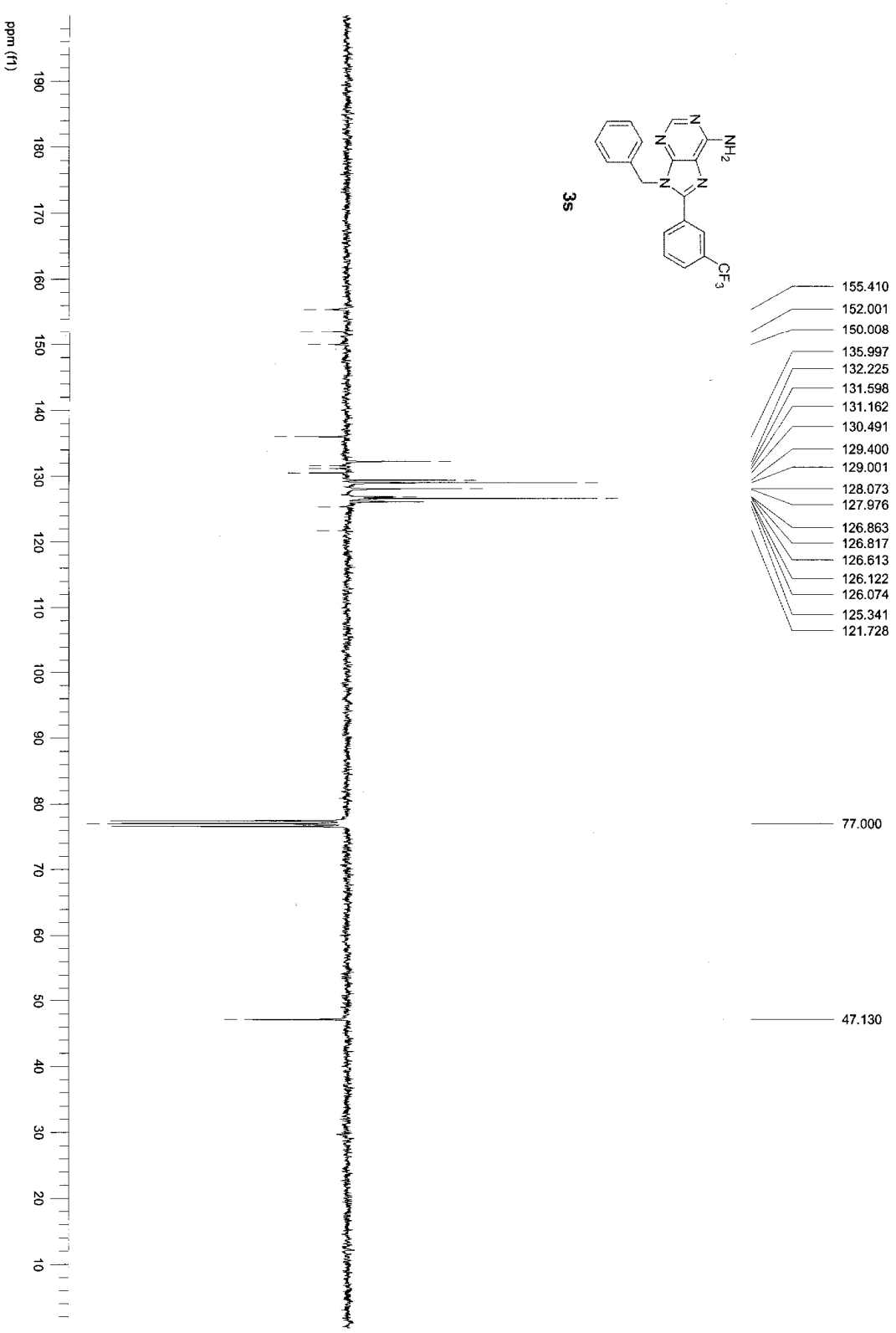
8.008
7.987
7.858
7.832
7.756
7.729
7.702
7.320
7.244
7.123
7.098
6.831
5.618
2.050


ppm (f1)
3s
155.410
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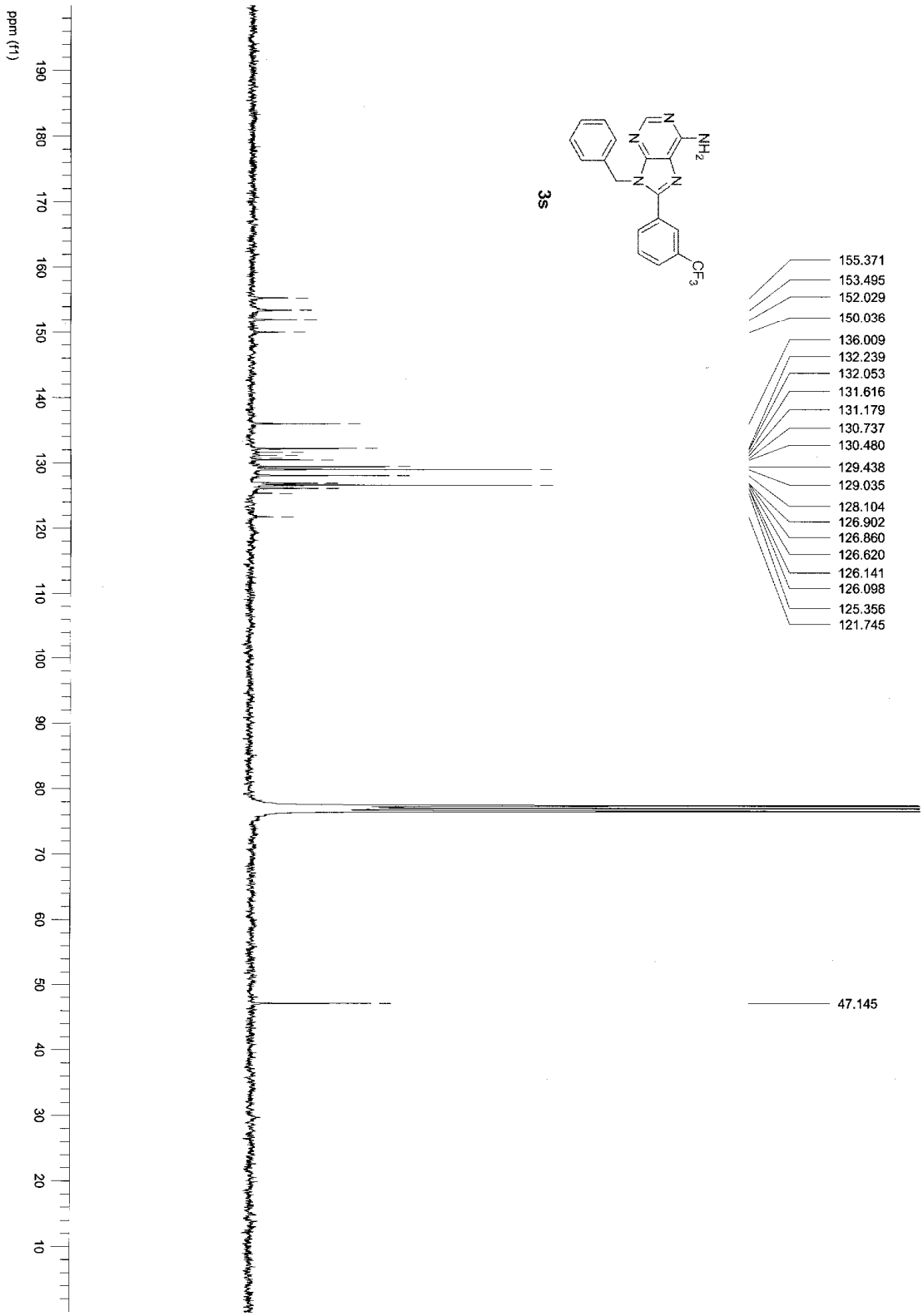
150.008
135.997
132.225
131.598
131.162
130.491
129.400
129.001
128.073
127.976
126.863
126.817
126.613
126.122
126.074
125.341
121.728
77.000
47.130


ppm (f1)
3s
155.371
153.495
152.029
150.036
136.009
132.239
132.053
131.616
131.179
130.737
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129.438
129.035
128.104
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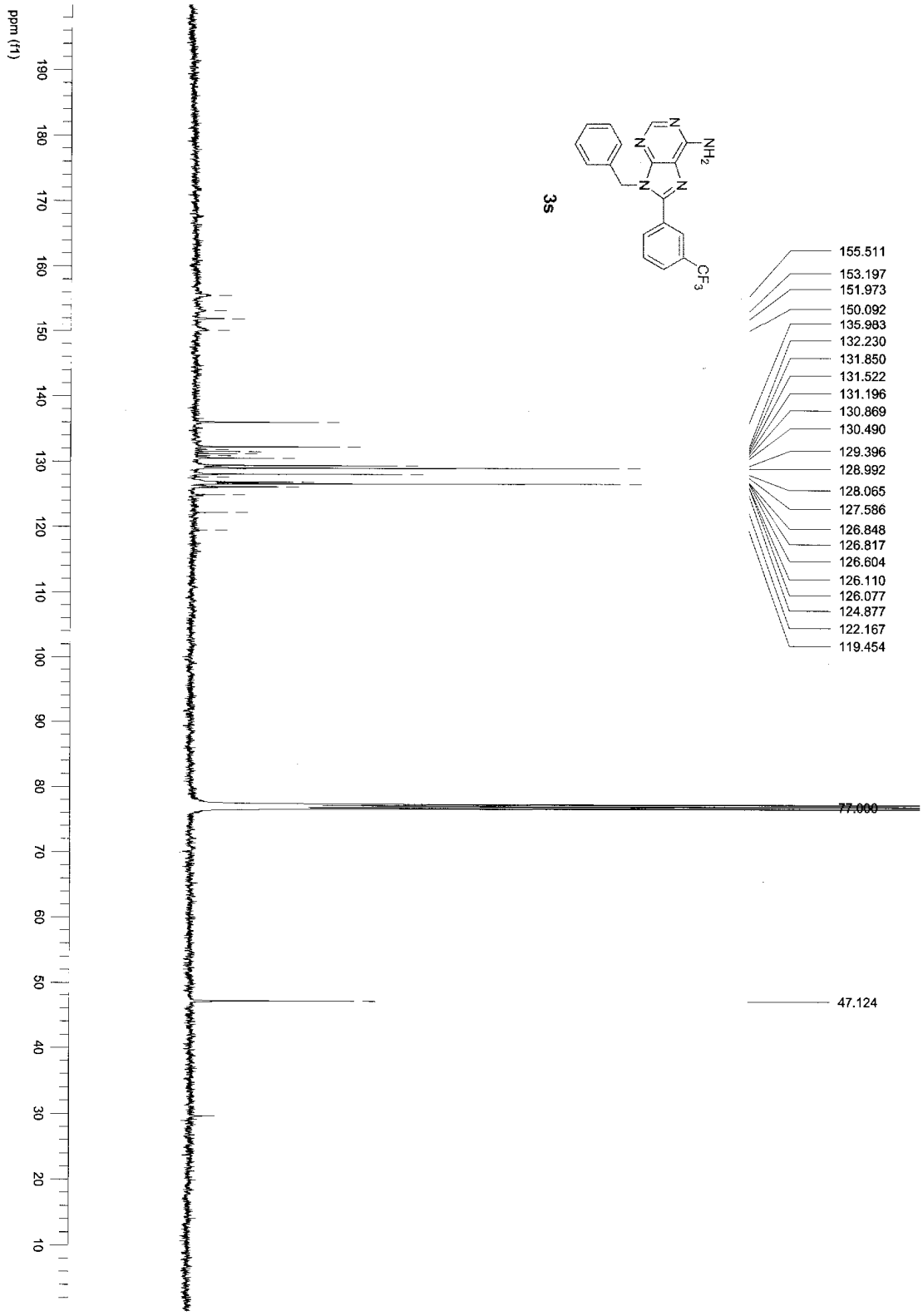
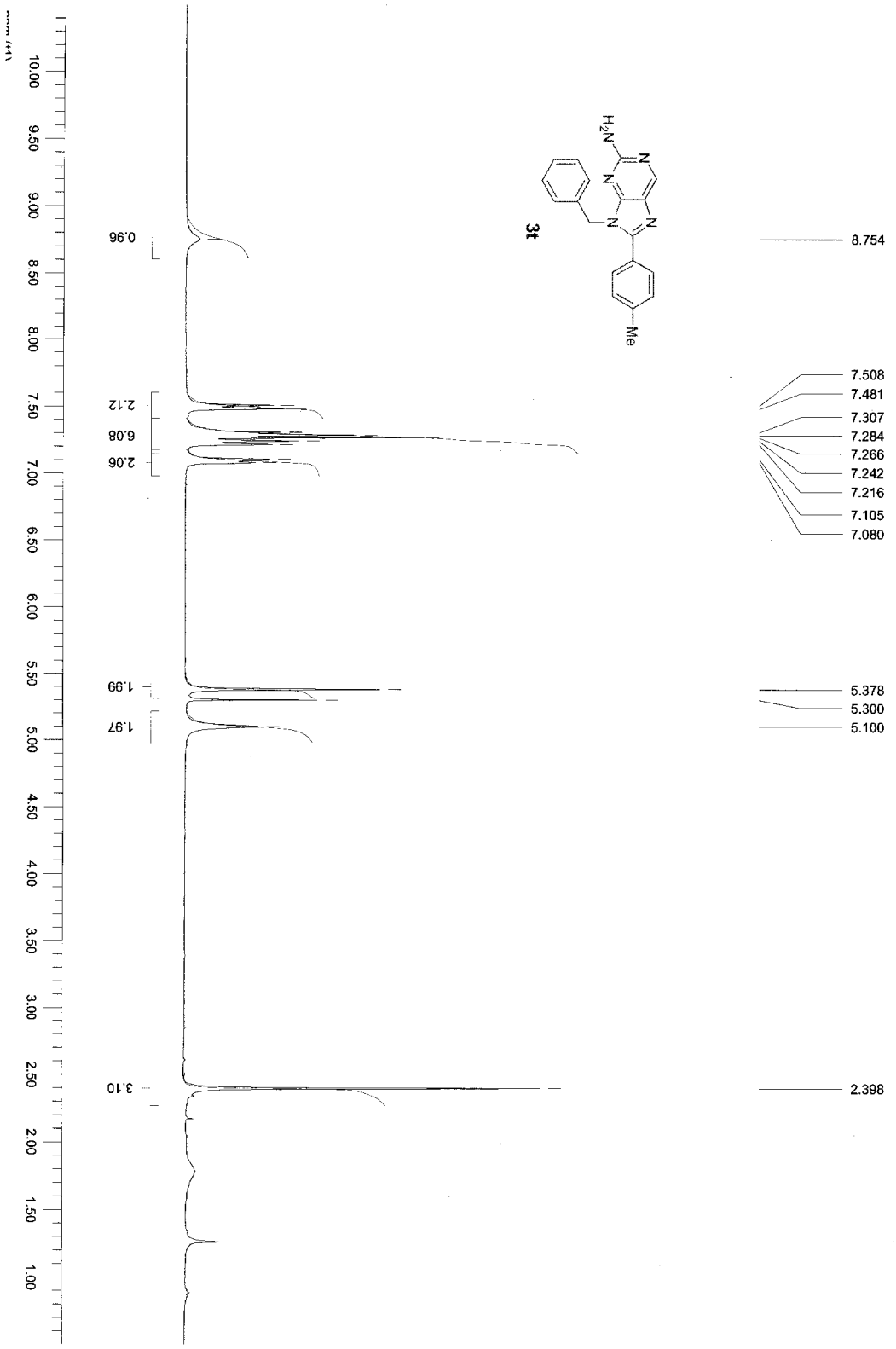
126.860
126.620
126.141
126.098
125.356
121.745
47.145


ppm (f1)
3s
155.511
153.197
151.973
150.092
135.983
132.230
131.850
131.522
131.196
130.869
130.490
129.396
128.992
128.065
127.586
126.848
126.817
126.604
126.110
126.077
124.877
122.167
119.454
77.000
47.124


3t
Me
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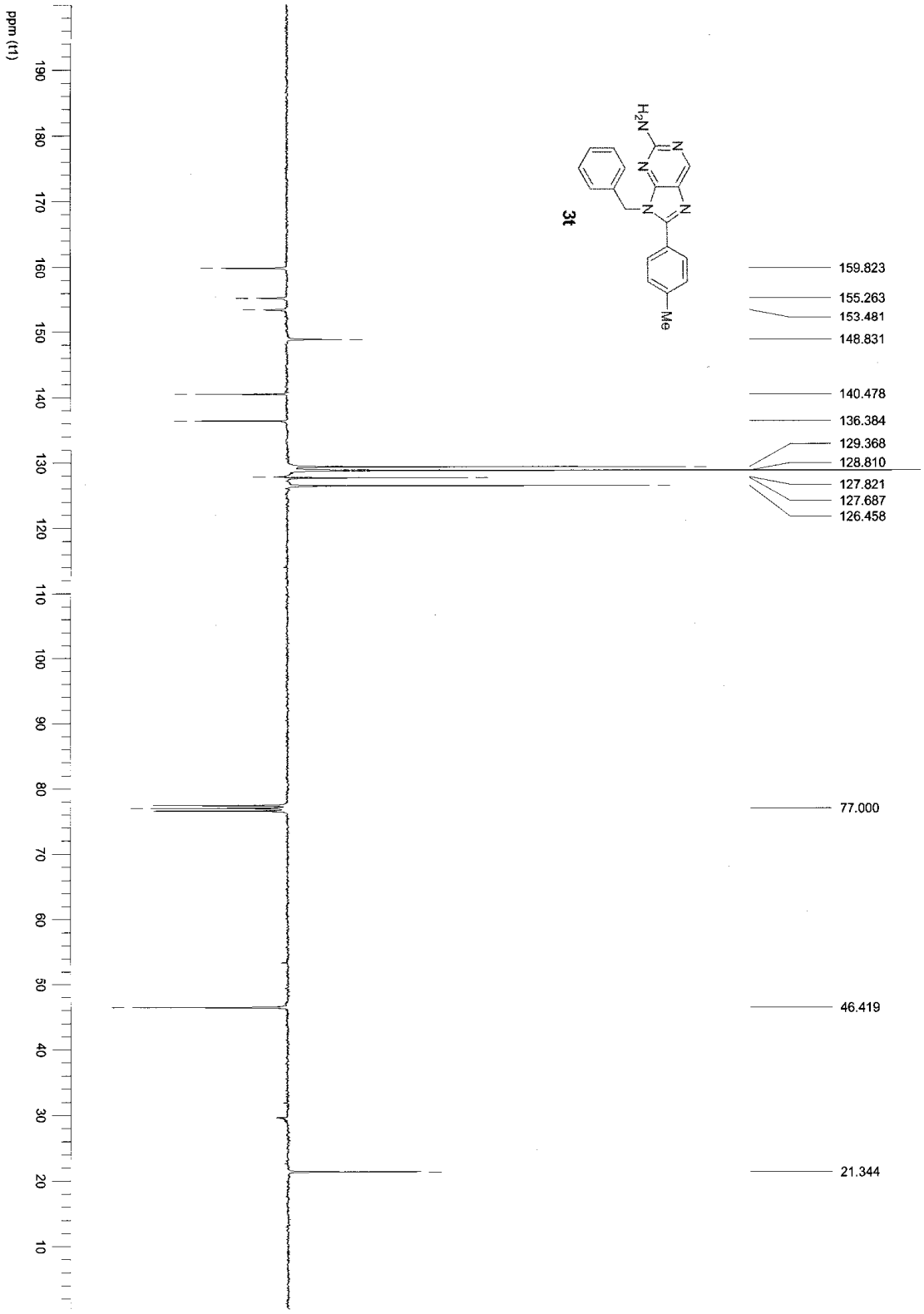
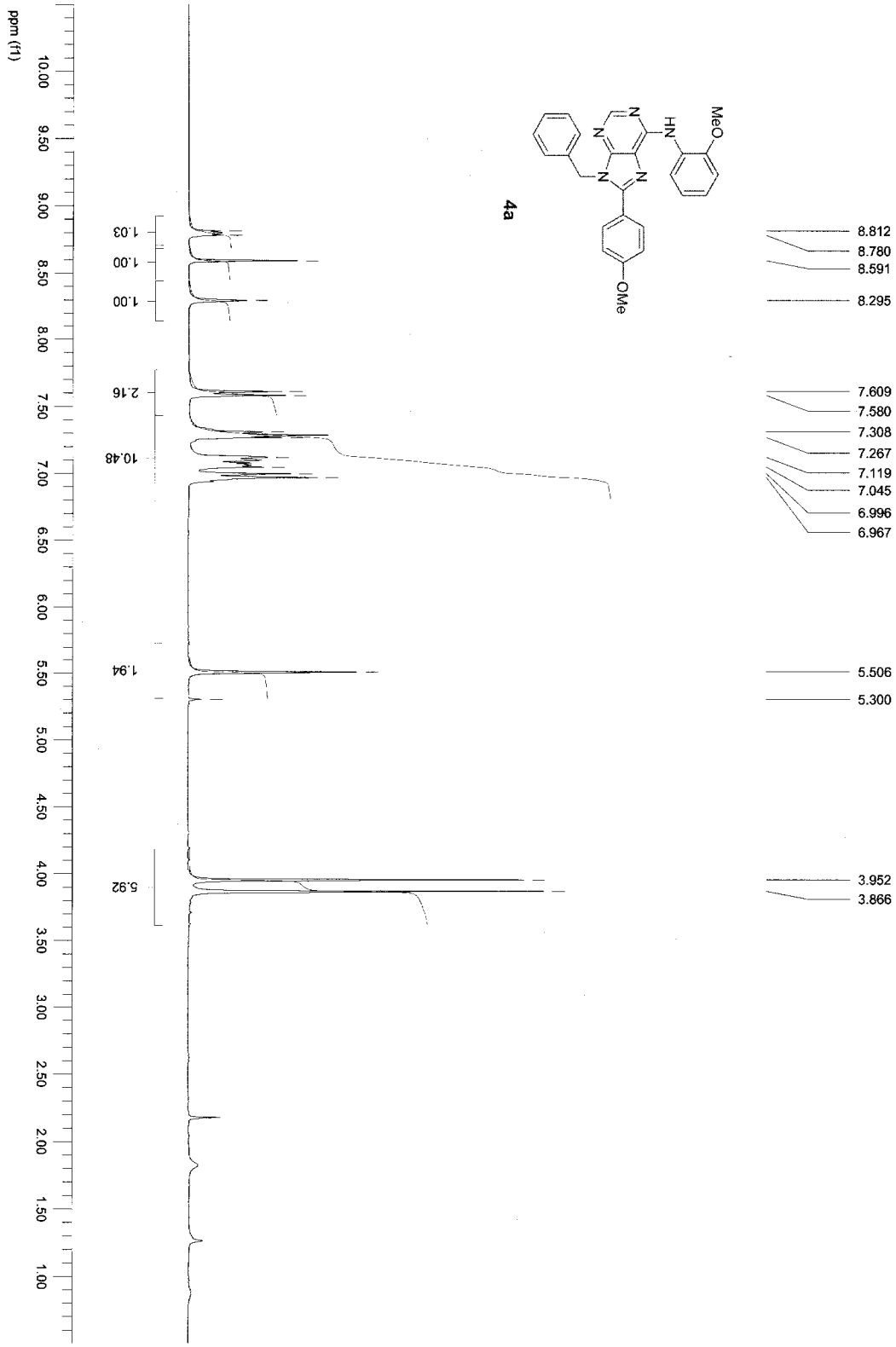
7.508
7.481
7.307
7.284
7.266
7.242
7.216
7.105
7.080
5.378
5.300
5.100
2.398
0.96
2.12
6.08
2.06
1.99
1.97
3.10


ppm (t1)
3t
159.823
155.263
153.481
148.831
140.478
136.384
129.368
128.810
127.821
127.687
126.458
77.000
46.419
21.344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9.00
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8.00
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7.00
6.50
6.00
5.50
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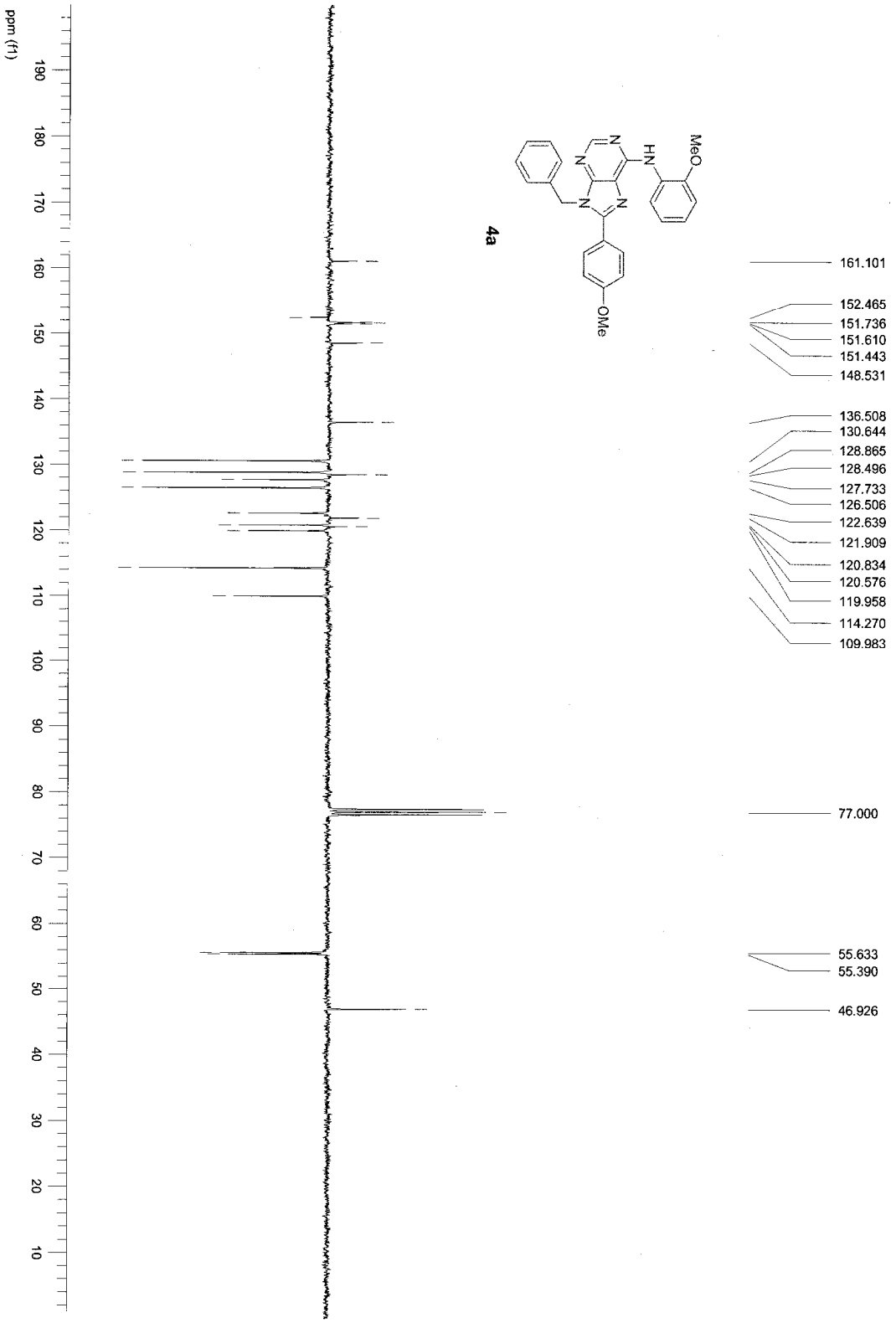
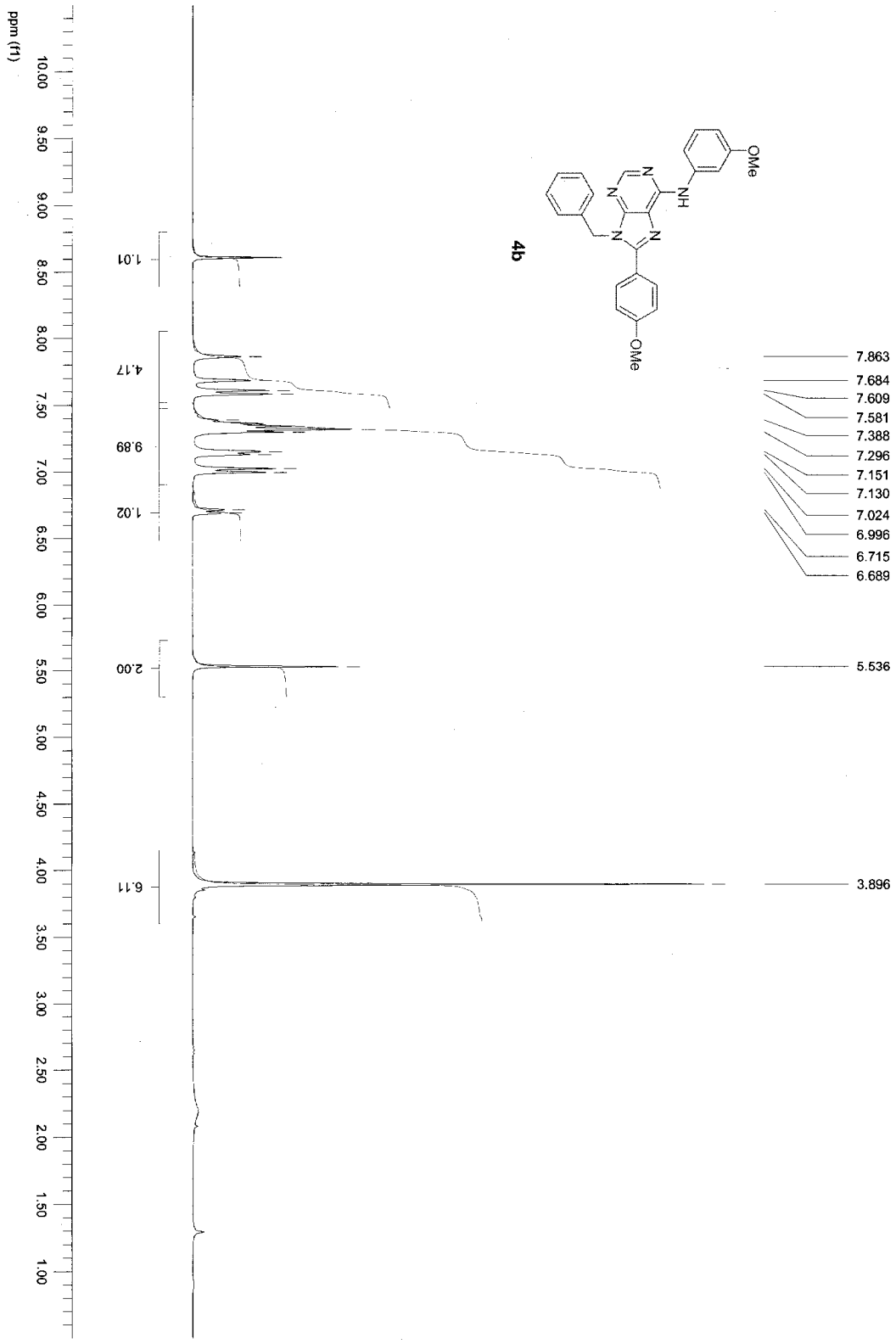
2.50
2.00
1.50
1.00
1.03
1.00
1.00
2.16
10.48
1.94
5.92
4a
N
N
N
N
HN
MeO
OMe
8.812
8.780
8.591
8.295
7.609
7.580
7.308
7.267
7.119
7.045
6.996
6.967
5.506
5.300
3.952
3.866


ppm (f1)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
4a
MeO
HN
OMe
161.101
152.465
151.736
151.610
151.443
148.531
136.508
130.644
128.865
128.496
127.733
126.506
122.639
121.909
120.834
120.576
119.958
114.270
109.983
77.000
55.633
55.390
46.926


ppm (f1)
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9.50
9.00
8.50
8.00
7.50
7.00
6.50
6.00
5.50
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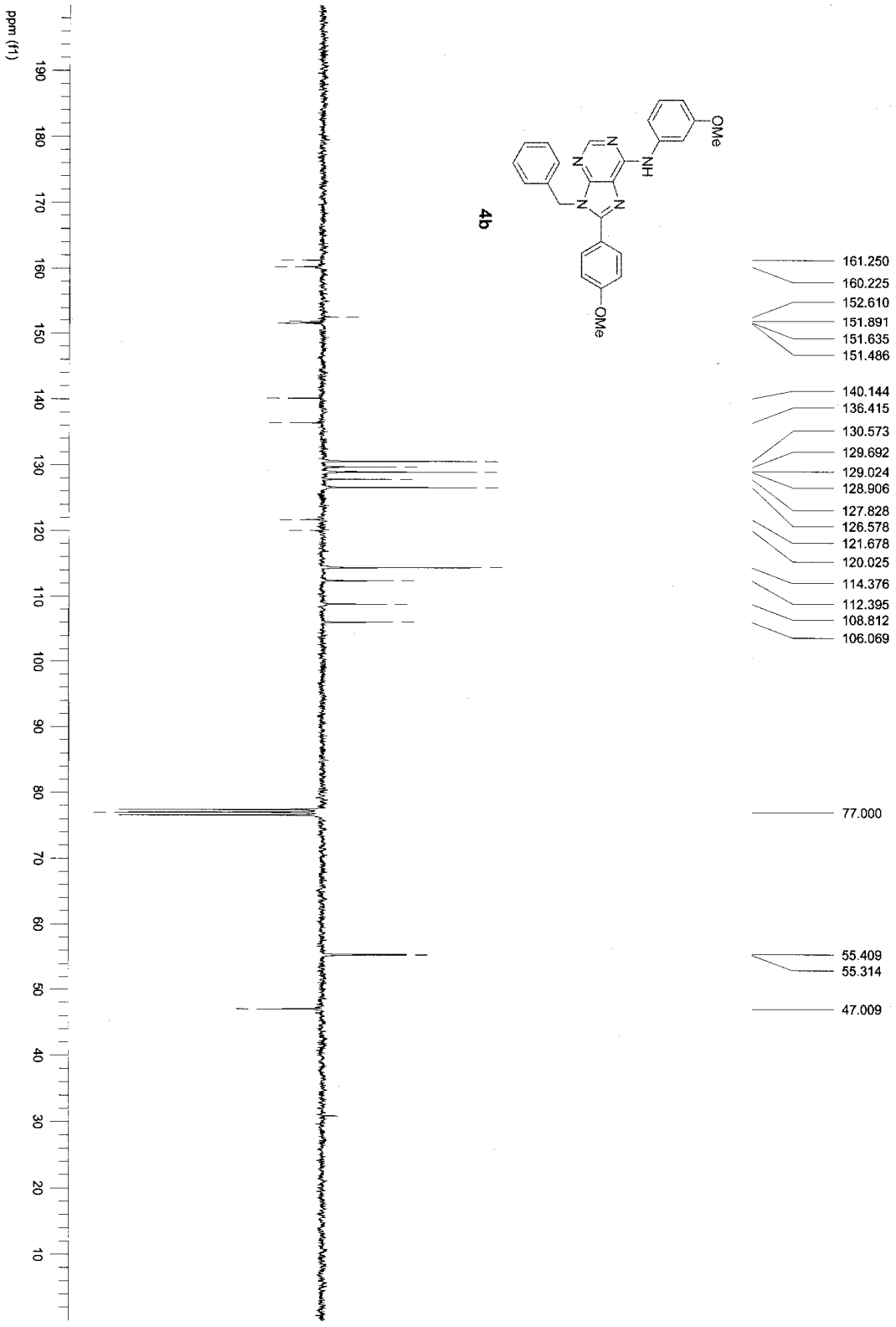
3.00
2.50
2.00
1.50
1.00
1.01
4.17
9.89
1.02
2.00
6.11
4b
OMe
NH
OMe
7.863
7.684
7.609
7.581
7.388
7.296
7.151
7.130
7.024
6.996
6.715
6.689
5.536
3.896


ppm (f1)
4b
OMe
NH
OMe
161.250
160.225
152.610
151.891
151.635
151.486
140.144
136.415
130.573
129.692
129.024
128.906
127.828
126.578
121.678
120.025
114.376
112.395
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106.069
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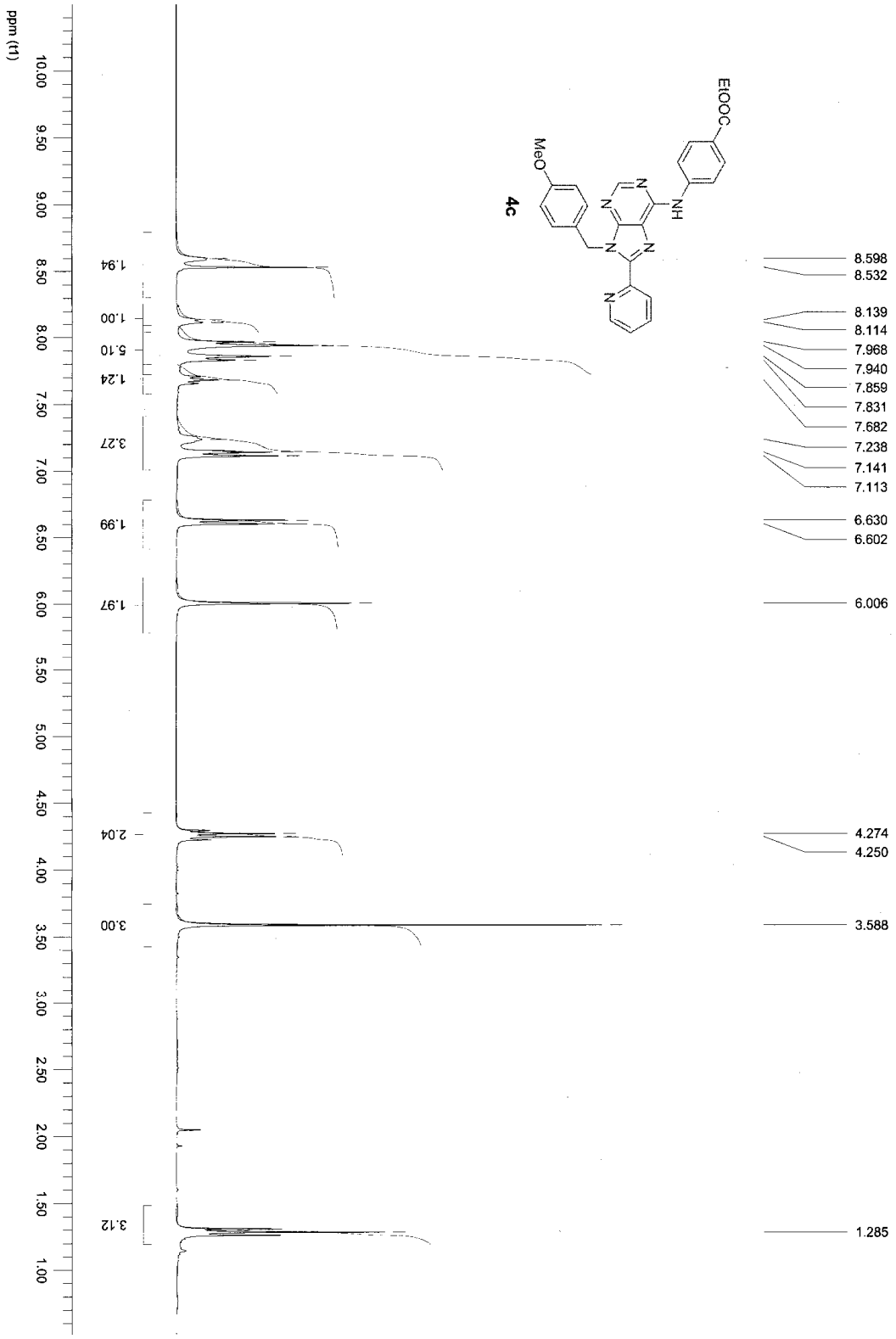
55.314
47.009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7.00
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5.50
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4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
1.94
1.00
5.10
1.24
3.27
1.99
1.97
2.04
3.00
3.12
MeO
EtOOC
NH
N
4c
8.598
8.532
8.139
8.114
7.968
7.940
7.859
7.831
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7.238
7.141
7.113
6.630
6.602
6.006
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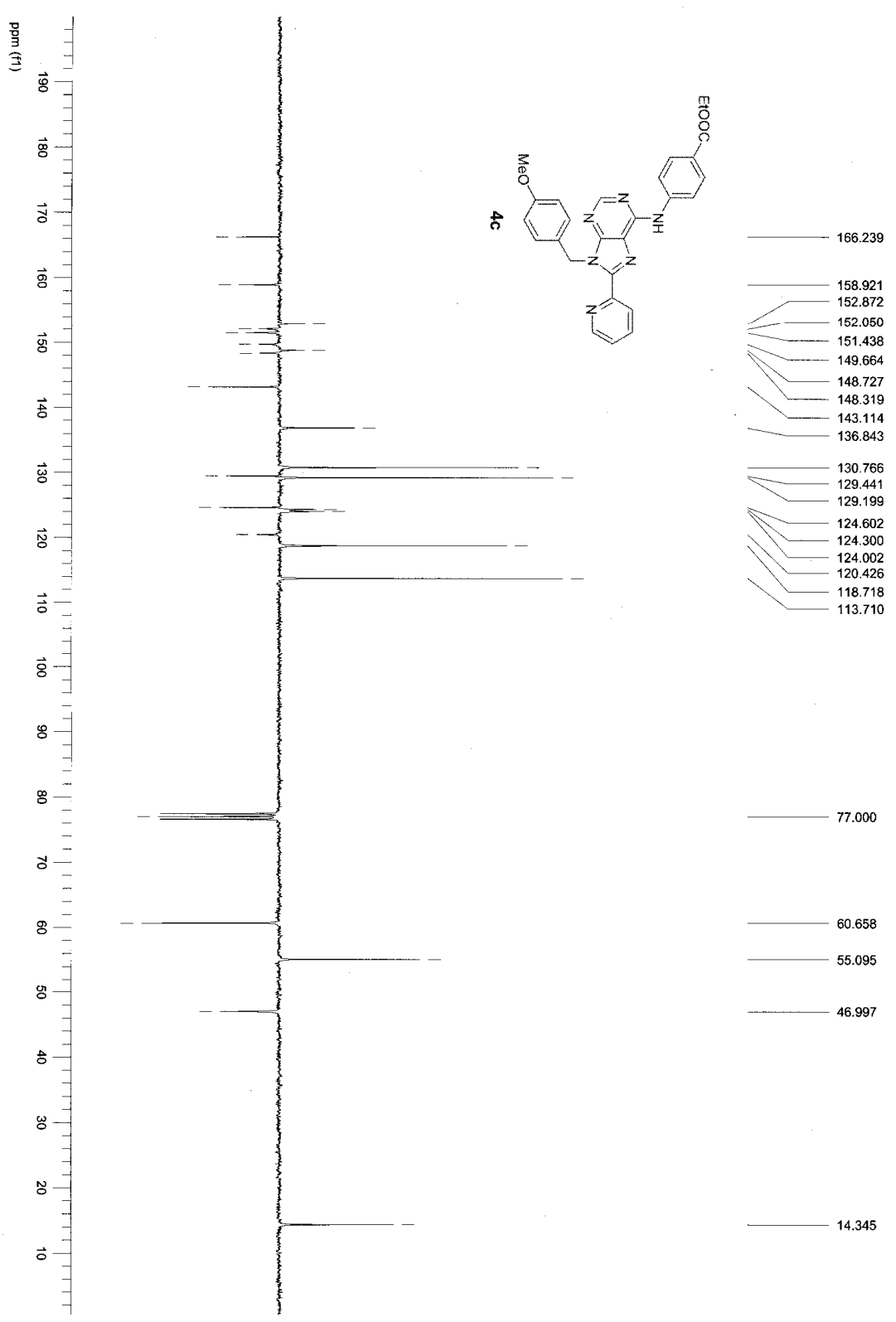
1.285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180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
MeO
EtOOC
NH
N
4c
166.239
158.921
152.872
152.050
151.438
149.664
148.727
148.319
143.114
136.843
130.766
129.441
129.199
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124.002
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77.000
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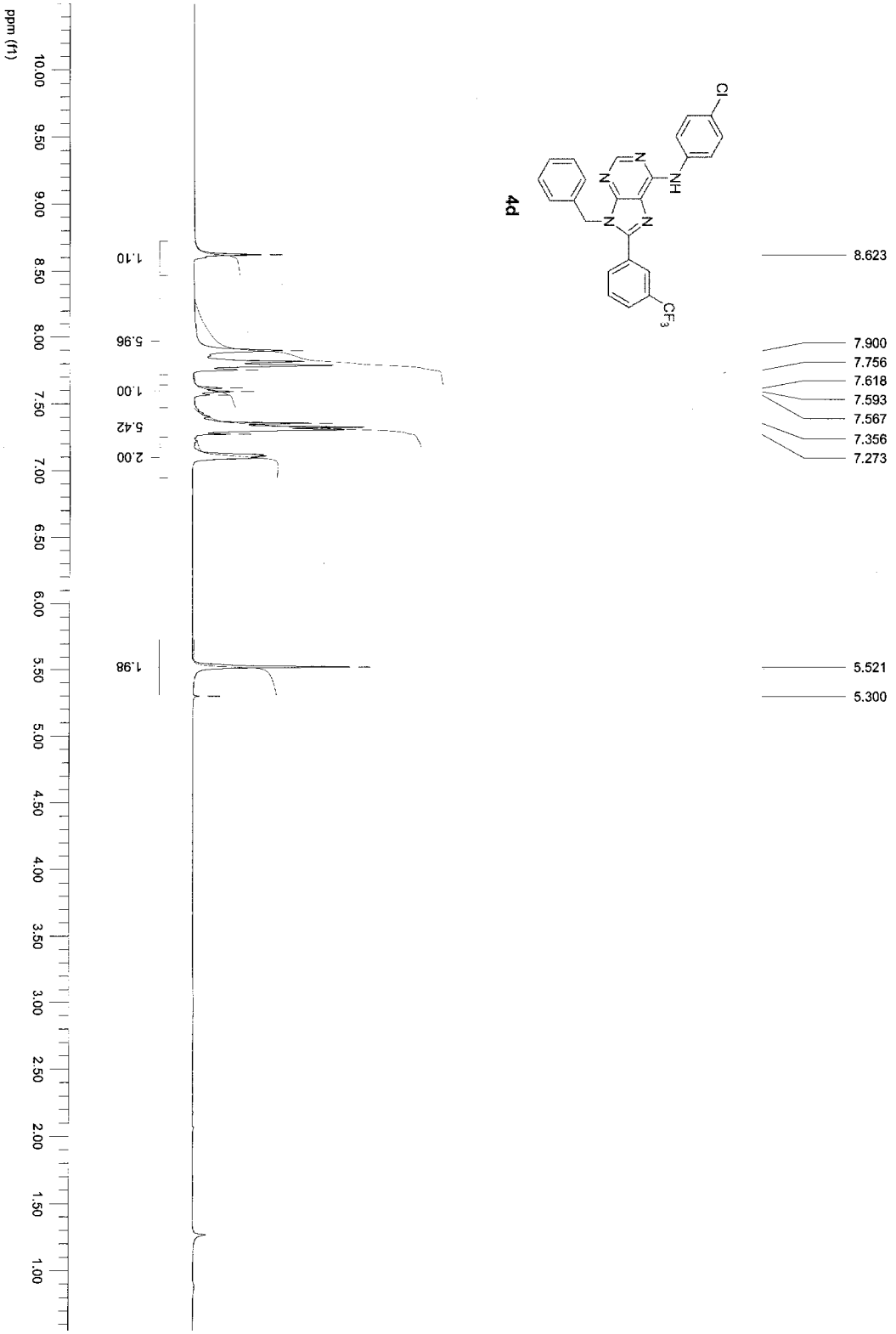
46.997
14.345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6.00
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3.00
2.50
2.00
1.50
1.00
1.10
5.96
1.00
5.42
2.00
1.98
4d
Cl
NH
N
CF3
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7.900
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7.618
7.593
7.567
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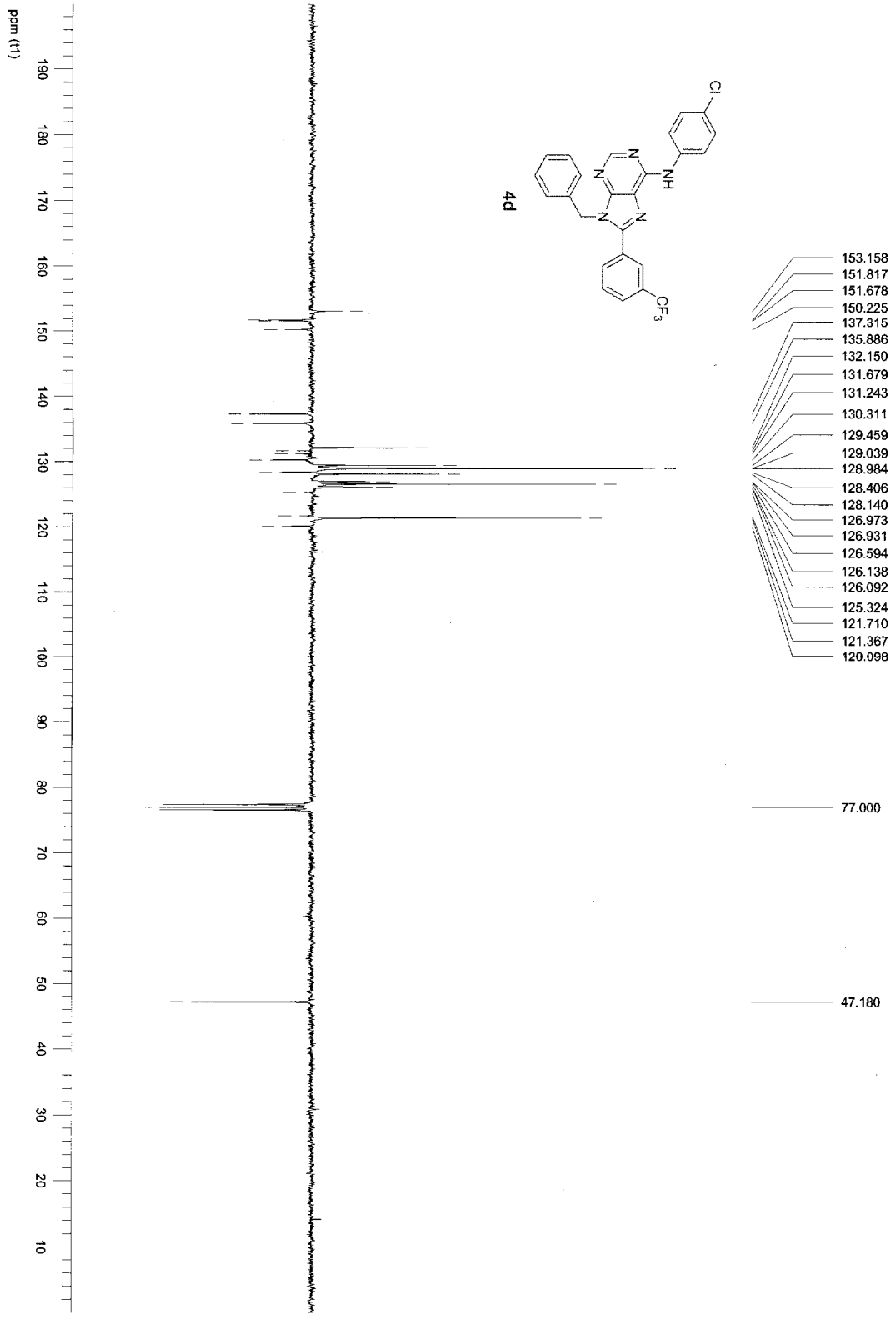
5.300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N
N
N
CF3
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135.886
132.150
131.679
131.243
130.311
129.459
129.039
128.984
128.406
128.140
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126.594
126.138
126.092
125.324
121.710
121.367
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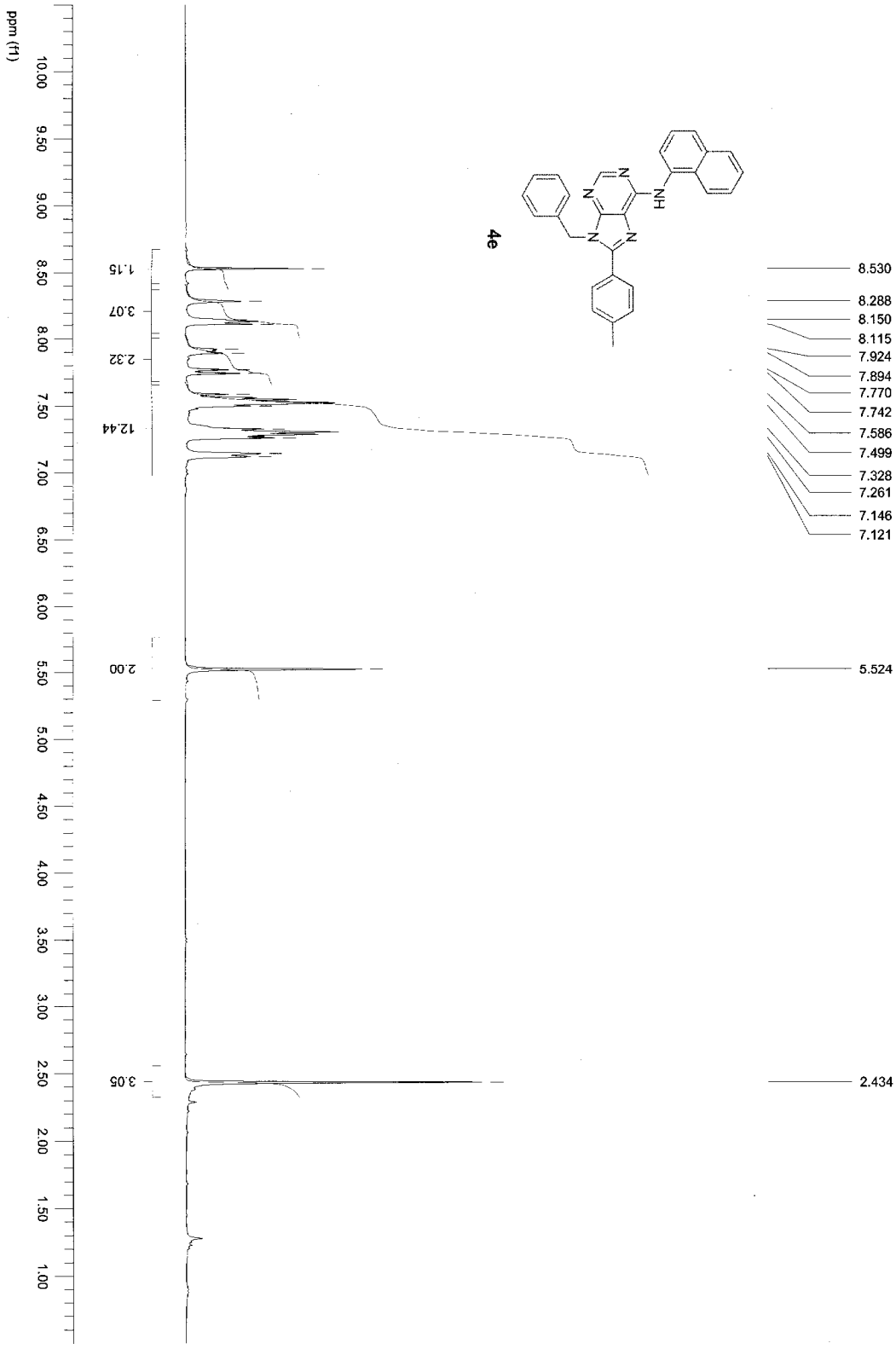
77.000
47.180


4e
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8.150
8.115
7.924
7.894
7.770
7.742
7.586
7.499
7.328
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7.146
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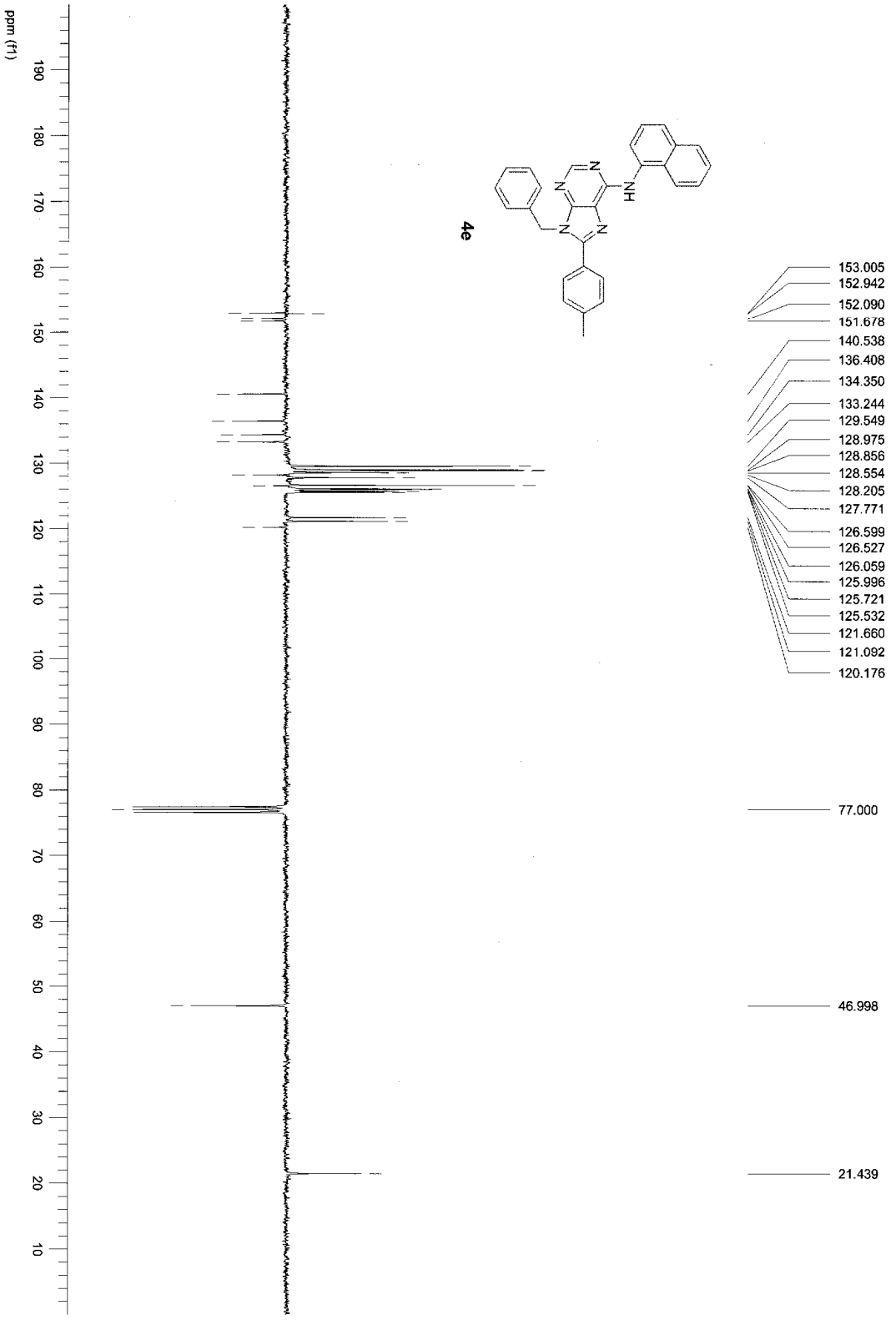
2.434
1.15
3.07
2.32
12.44
2.00
3.05
ppm (f1)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128.856
128.554
128.205
127.771
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125.996
125.721
125.532
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46.998
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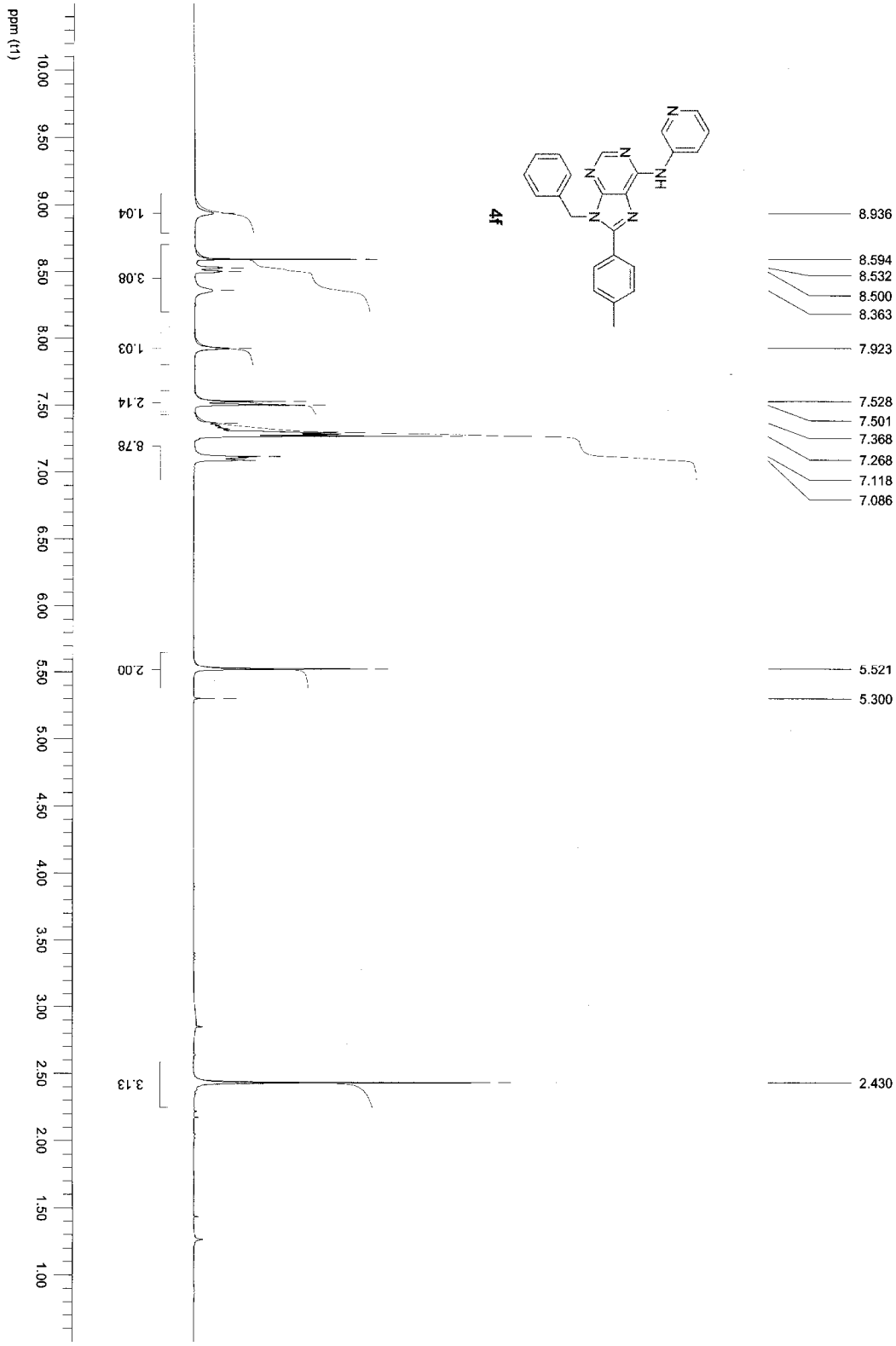
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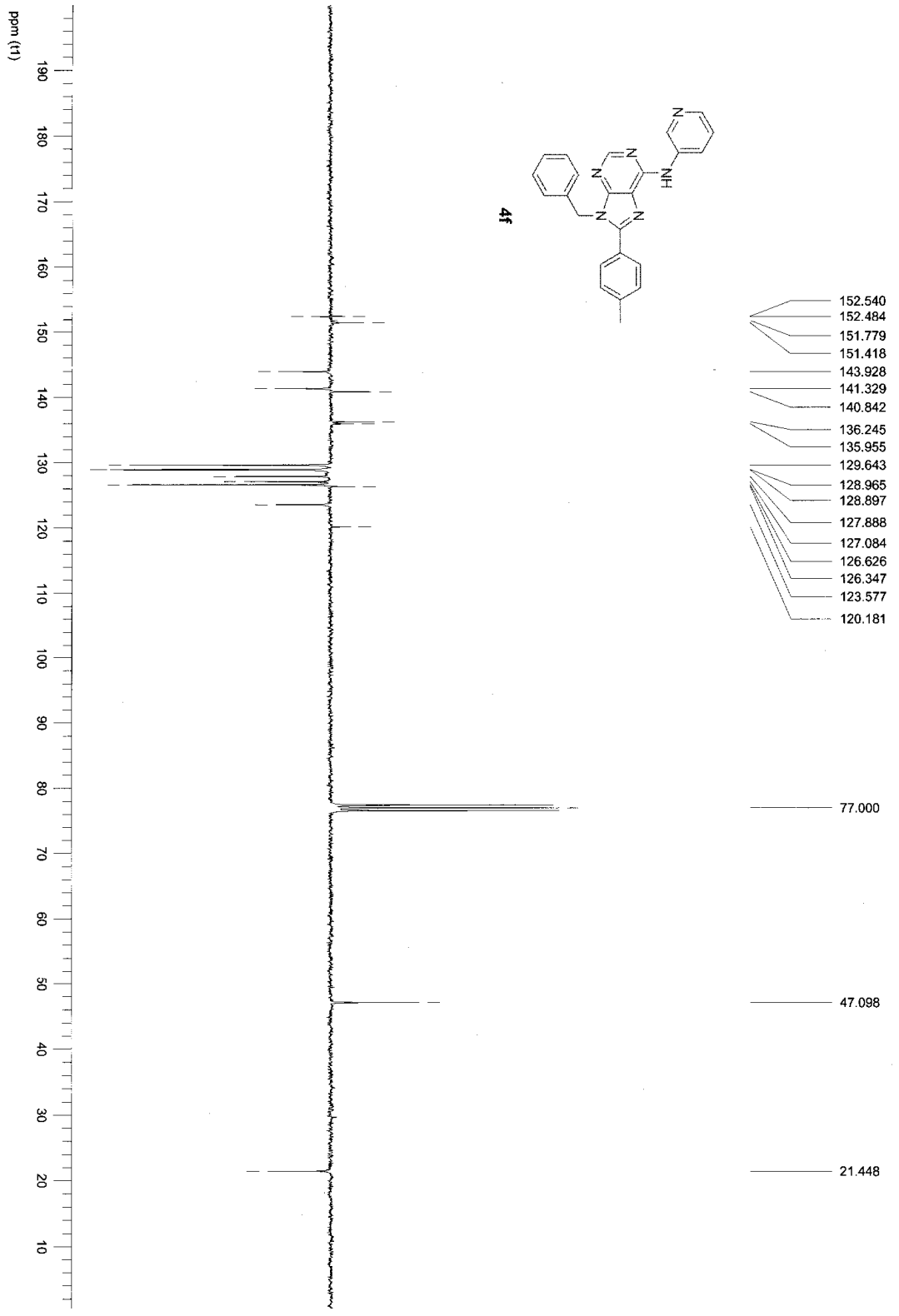
1.50
1.00
1.04
3.08
1.03
2.14
8.78
2.00
3.13
4f
8.936
8.594
8.532
8.500
8.363
7.923
7.528
7.501
7.368
7.268
7.118
7.086
5.521
5.300
2.430


ppm (t1)
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136.245
135.955
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128.897
127.888
127.084
126.626
126.347
123.577
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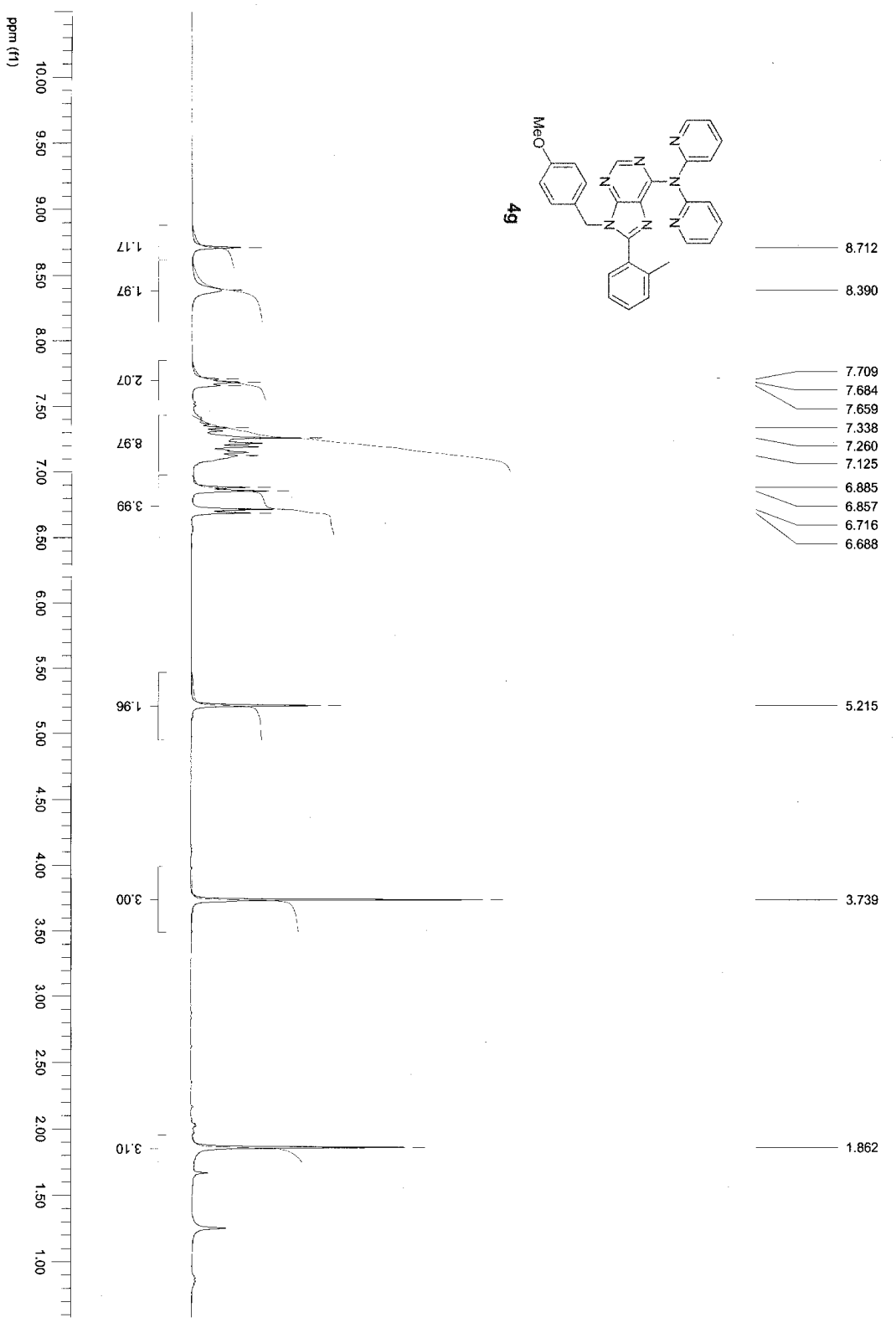
77.000
47.098
21.448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8.00
7.50
7.00
6.50
6.00
5.50
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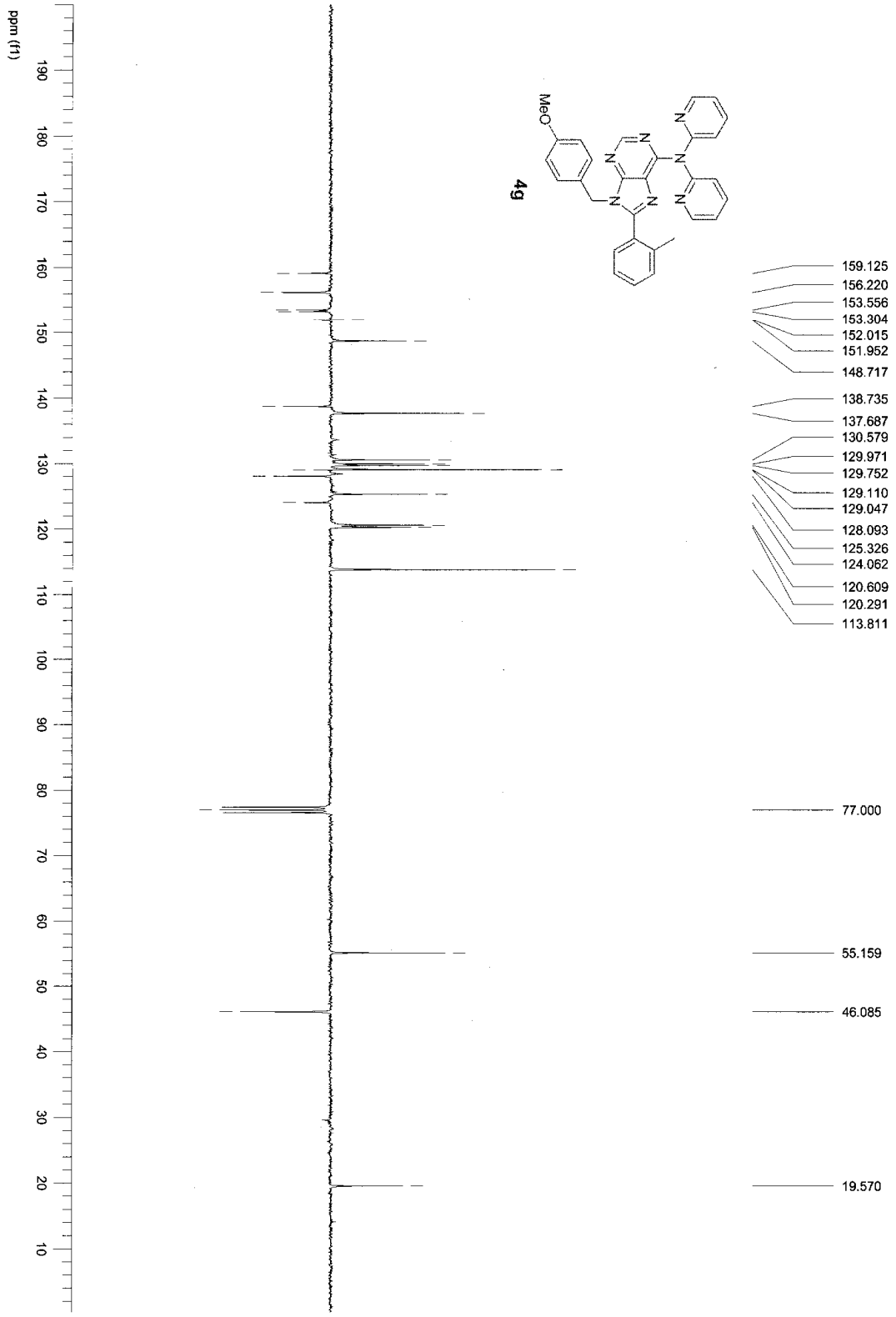
3.00
2.50
2.00
1.50
1.00
1.17
1.97
2.07
8.97
3.99
1.96
3.00
3.10
MeO
4g
8.712
8.390
7.709
7.684
7.659
7.338
7.260
7.125
6.885
6.857
6.716
6.688
5.215
3.739
1.862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4g
MeO
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152.015
151.952
148.717
138.735
137.687
130.579
129.971
129.752
129.110
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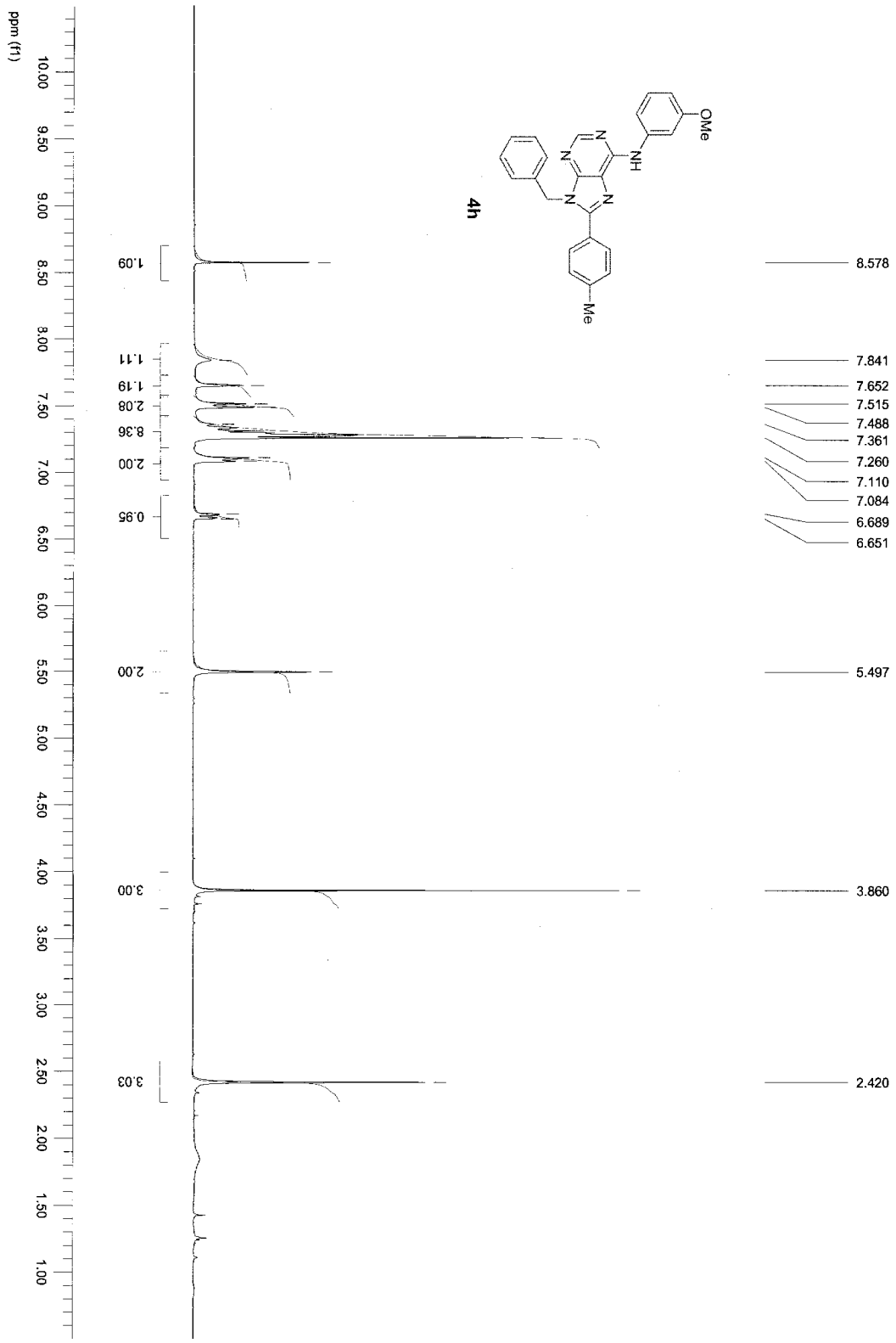
125.326
124.062
120.609
120.291
113.811
77.000
55.159
46.085
19.570


4h
OMe
NH
N
N
N
N
Me
ppm (f1)
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9.00
8.50
8.00
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3.50
3.00
2.50
2.00
1.50
1.00
1.09
1.11
1.19
2.08
8.36
2.00
0.95
2.00
3.00
3.03
8.578
7.841
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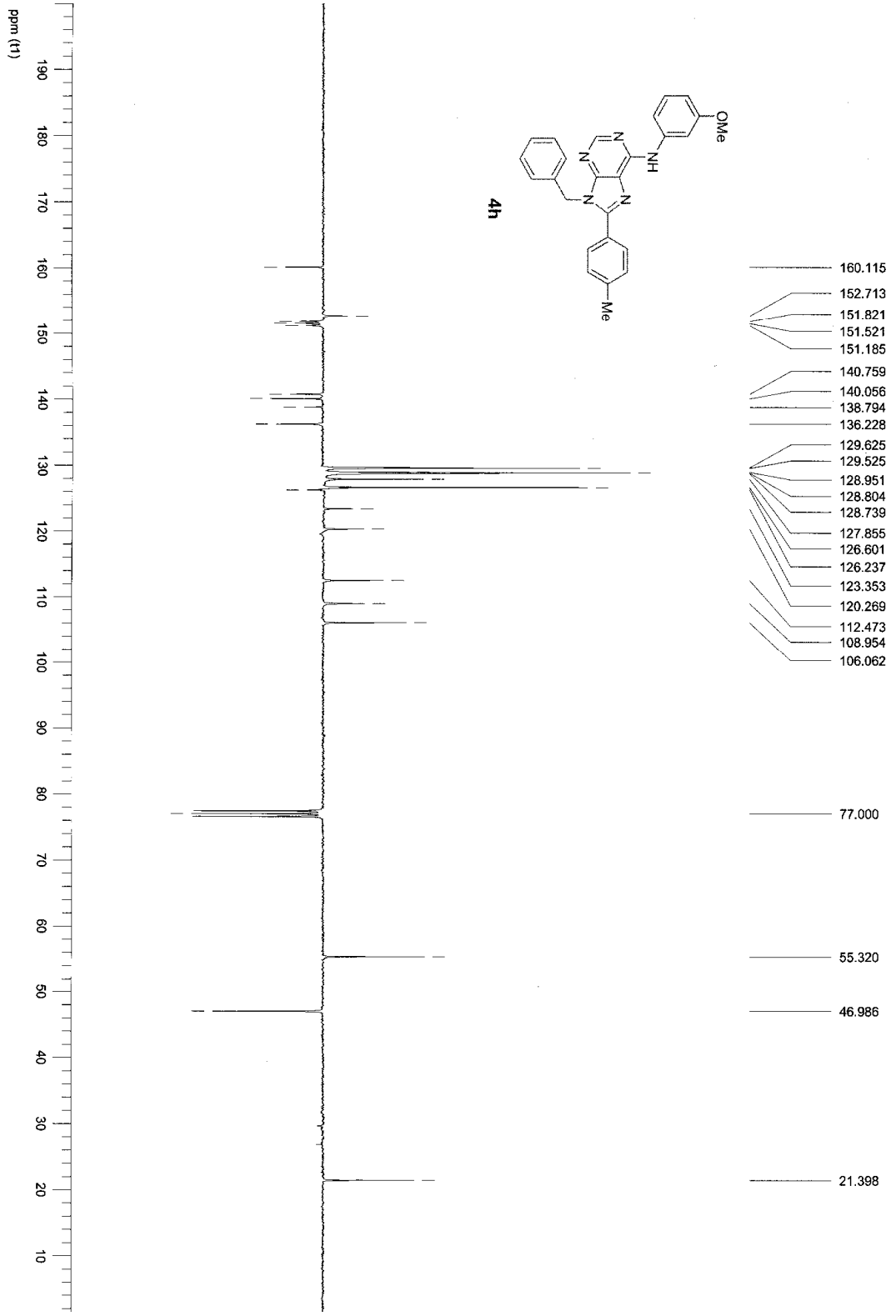
7.361
7.260
7.110
7.084
6.689
6.651
5.497
3.860
2.420


ppm (t1)
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OMe
NH
Me
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151.521
151.185
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140.056
138.794
136.228
129.625
129.525
128.951
128.804
128.739
127.855
126.601
126.237
123.353
120.269
112.473
108.954
106.062
77.000
55.320
46.986
21.398