Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Fe/S-catalyzed decarboxylative redox condensation of arylacetic acids with nitro arenes

Thanh Binh Nguyen,* Ludmila Ermolenko, Mathilde Corbin and Ali Al-Mourabit*

General information

Reagents were obtained from commercial supplier and used without further purification. Analytical thinlayer chromatography (TLC) was purchased from Merck KGaA (silica gel 60 F254). Visualization of the chromatogram was performed by UV light (254 nm) or phosphomolybdic acid or vanilline stains. Flash column chromatography was carried out using kieselgel 35-70 μm particle sized silica gel (230-400 mesh). NMR Chemical shifts are reported in (δ) ppm relative to tetramethylsilane (TMS) with the residual solvent as internal reference ($CDCl_3$, δ 7.26 ppm for ^1H and δ 77.0 ppm for ^{13}C; DMSO-d_6, δ 2.50 ppm for ^1H and δ 39.5 ppm for ^{13}C; CD_3OD, δ 3.31 ppm for ^1H and δ 49.0 ppm for ^{13}C). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. We note that some quaternary carbon signals in some cases of NH-benzimidazoles are difficult to observe due to tautomerism.

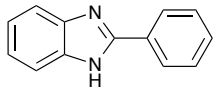
General procedure

Metal-catalyzed redox condensation reaction

A mixture of *o*-substituted nitrobenzene **1** (2.5 mmol) or **4** (1.25 mmol), aryl acetic acid **2** (3 mmol, 1.5 equiv), elemental sulfur (32 mg, 1 mmol, 40 mol %), $FeCl_2{\cdot}4H_2O$ (5 mol %, 0.125 mmol) and *N*-methylpiperidine (40 mol %, 1 mmol, 99 mg) was stirred in a 20-mL tube under an argon atmosphere for 20 h at 130 °C. The crude mixture cooled to room temperature was purified by column chromatography on silica gel (heptane : ethyl acetate) to afford the desired benzazoles.

Characterizations of Products

2-Phenylbenzimidazole (3a)[1]

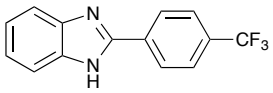


Grayish solid.

^{1}H NMR (300 MHz, CD_3OD) δ 8.09-8.07 (m, 2H), 7.63-7.60 (m, 2H), 7.54-7.47 (m, 3H), 7.28-7.22 (m, 2H).

^{13}C NMR (75 MHz, DMSO-d_6) δ 151.2, 143.8, 135.0, 130.2, 129.8, 128.9, 126.4, 122.5, 121.7, 118.9, 111.3.

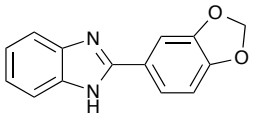
2-(4-(Trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazole (3b)[2]



^{1}H NMR (300 MHz, CD_3OD) δ 8.25 (d, J = 7.7 Hz, 2H), 7.84 (d, J = 7.7 Hz, 2H), 7.64 (s, 2H), 7.30 (m, 2H).

^{13}C NMR (75 MHz, CD_3OD) δ 151.7, 144.8 (broad peak), 136.5 (broad peak), 134.8, 132.9 (q, $J_{C\text{-}F}$ = 321 Hz), 128.5, 127.2 (q, $J_{C\text{-}F}$ = 3.7 Hz), 126.7, 124.5, 122.3, 119.8 (broad peak), 113.2 (broad peak).

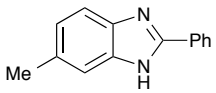
2-(Benzo[*d*][1,3]dioxol-5-yl)-1*H*-benzo[*d*]imidazole (3c)[2]



^{1}H NMR (300 MHz, CD_3OD) δ 7.57 (m, 4H), 7.23 (m, 2H), 6.95 (d, J = 8.2 Hz, 1H), 6.03 (s, 2H).

^{13}C NMR (75 MHz, CD_3OD) δ 152.0, 149.5, 148.5, 123.5, 122.5, 121.0, 114.2, 108.3, 106.5, 101.7.

6-Methyl-2-phenyl-1*H*-benzo[*d*]imidazole (3d)[1]

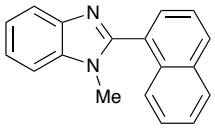


[1] T. B. Nguyen, J. Le Bescont, L. Ermolenko, and A. Al-Mourabit, *Org. Let*t. **2013**, *15*, 6218.
[2] A. J. Blacker, M. M. Farah, M. I. Hall, S. P. Marsden, O. Saidi, J. M. J. Williams, *Org. Lett*. **2009**, *11*, 2039.

^{1}H NMR (300 MHz, CD_3OD) δ 8.06 (d, J = 7.4 Hz, 2H), 7.50 (m, 4H), 7.39 (s, 1H), 7.08 (d, J = 8.4 Hz, 1H), 2.46 (s, 3H).

^{13}C NMR (75 MHz, CD_3OD) δ 151.6, 138.9, 137.5, 132.5, 129.8, 128.8, 126.4, 124.2, 114.5, 114.0, 20.2.

1-Methyl-2-(naphthalen-1-yl)-1*H*-benzo[*d*]imidazole (3e)

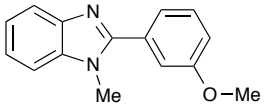


^{1}H NMR (300 MHz, CD_3OD) δ 8.12 (d, J = 7.9 Hz, 1H), 8.03 (d, J = 7.9 Hz, 1H), 7.73 (m, 3H), 7.58 (m, 4H), 7.40 (m, 3H), 3.63 (s, 3H).

^{13}C NMR (75 MHz, CD_3OD) δ 154.2, 143.4, 137.0, 135.1, 133.4, 131.2, 130.1, 129.7, 128.6, 128.5, 127.7, 126.2, 126.1, 124.4, 123.9, 119.7, 111.5, 31.4.

HRMS-ESI+: m/z [M + H]+ calcd for $C_{18}H_{15}N_2$: 259.1235; found: 259.1266.

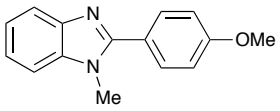
2-(3-Methoxyphenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3f)[3]



^{1}H NMR (300 MHz, CD_3OD) δ 7.87 (d, J = 8.7 Hz, 1H), 7.43 (m, 2H), 7.35 (m, 4H), 7.07 (d, J = 8.7 Hz, 1H), 3.92, 3.90 (2s, 6H).

^{13}C NMR (75 MHz, CD_3OD) δ 160.0, 153.8, 142.8, 136.7, 131.4, 129.8, 123.0, 122.7, 121.9, 120.0, 116.2, 114.8, 109.8, 55.6, 32.0.

2-(4-Methoxyphenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3g)[4]

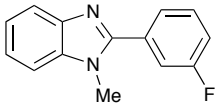


^{1}H NMR (300 MHz, CD_3OD) δ 7.83 (d, J = 8.0 Hz, 1H), 7.73 (d, J = 7.3 Hz, 2H), 7.37 (m, 1H), 7.32 (m, 2H), 7.05 (d, J = 7.3 Hz, 2H), 3.88, 3.85 (2s, 6H).

^{13}C NMR (75 MHz, CD_3OD) δ 160.8, 153.7, 142.8, 136.5, 130.9, 122.5, 122.4 (two carbons), 119.5, 114.1, 109.6, 55.4, 31.7.

[3] Z. Gu, W. Chen, L. Shao, *J. Org. Chem*. **2014**, *79*, 5806.
[4] W. Zhang, Q. Zeng, X. Zhang, Y. Tian, Y. Yue, Y. Guo, Z. Wang, *J. Org. Chem.* **2011**, *76*, 4741.

2-(3-Fluorophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3h)

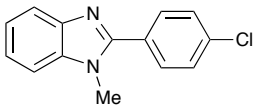


^{1}H NMR (300 MHz, CD_3OD) δ 7.91 (d, J = 8.0 Hz, 1H), 7.85 (m, 2H), 7.48 (d, J = 8.0 Hz, 1H), 7.42 (m, 2H), 7.32 (d, J = 7.3 Hz, 2H), 3.93 (s, 3H).

^{13}C NMR (75 MHz, CD_3OD) δ 160.8, 158.9, 148.8, 138.7, 132.6, 127.6, 127.5, 122.3, 119.0, 118.7, 115.8, 112.1, 111.9, 105.8, 27.7.

HRMS-ESI+: m/z [M + H]+ calcd for $C_{14}H_{12}FN_2$: 227.0985; found: 227.0966.

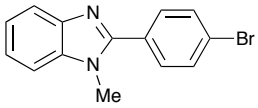
2-(4-Chlorophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3i)[5]



^{1}H NMR (300 MHz, CD_3OD) δ 7.84 (d, J = 6.8 Hz, 1H), 7.74 (d, J = 8.0 Hz, 2H), 7.52 (d, J = 8.0 Hz, 2H), 7.40 (m, 1H), 7.35 (m, 2H), 3.88 (s, 3H).

^{13}C NMR (75 MHz, CD_3OD) δ 152.6, 142.6, 136.5, 136.2, 130.7, 129.1, 128.6, 123.2, 122.8, 119.9, 109.7, 31.7.

2-(4-Bromophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3j)

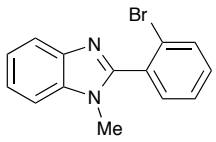


^{1}H NMR (300 MHz, CD_3OD) δ 7.84 (d, J = 7.8 Hz, 1H), 7.69 (m, 5H), 7.41 (m, 1H), 7.36 (m, 2H), 3.87 (1s, 3H).

^{13}C NMR (75 MHz, CD_3OD) δ 152.5, 142.5, 136.5, 132.0, 131.0, 128.9, 124.5, 123.2, 122.8, 120.0, 110.0, 32.0.

HRMS-ESI+: m/z [M + H]+ calcd for $C_{14}H_{12}{}^{79}BrN_2$: 287.0184; found: 287.0196.

2-(2-Bromophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3k)[6]



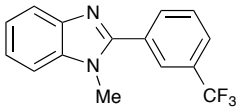
[5] L. Tang, X. Guo, Y. Yang, Z. Zha, Z. Wang, *Chem. Commun*. **2014**, *50*, 6145.

[6] A. J. Blatch, O. V. Chetina, J. A. K. Howard, L. G. F. Patrick, C. A. Smethurstb, A. Whiting, *Org. Biomol. Chem*. **2006**, *4*, 3297.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, J = 7.9 Hz, 1H), 7.74 (d, J = 7.9 Hz, 1H), 7.57 (d, J = 7.9 Hz, 1H), 7.48 (m, 1H), 7.43 (m, 2H), 7.37 (m, 3H), 3.69 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 152.5, 142.6, 135.4, 132.9, 132.5, 132.1, 131.5, 127.6, 123.9, 123.1, 122.6, 120.1, 109.8, 31.1.

1-Methyl-2-(3-(trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazole (3l)

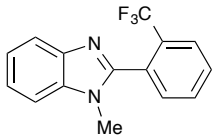


^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (s, 1H), 7.98 (d, J = 7.7 Hz, 1H), 7.87 (m, 1H), 7.80 (d, J = 7.7 Hz, 1H), 7.69 (t, J = 7.5 Hz, 1H), 7.40 (m, 3H), 3.91 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 152.0, 142.7, 136.6, 132.6, 131.2, 126.5 (two carbons, q), 126.4, 126.3, 123.4, 122.9, 120.0, 109.8, 31.7.

HRMS-ESI+: m/z [M + H]+ calcd for $C_{15}H_{12}F_3N_2$: 277.0953; found: 277.0925.

1-Methyl-2-(2-(trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazole (3m)

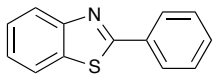


^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, J = 7.4 Hz, 2H), 7.69 (t, J = 3.7 Hz, 2H), 7.54 (d, J = 3.7 Hz, 1H), 7.38 (m, 3H), 3.58 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 150.5, 142.6, 135.4, 132.2, 131.7, 130.4, 129.0, 126.7, 126.6 (q, J = 4.4 Hz), 125.3, 123.1, 122.5, 120.1, 109.5, 30.6.

HRMS-ESI+: m/z [M + H]+ calcd for $C_{15}H_{12}F_3N_2$: 277.0953; found: 277.0978.

2-Phenylbenzo[d]thiazole (5a)[7]

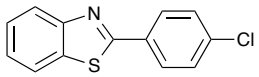


^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 (m, 2H), 7.98 (t, J = 7.8 Hz, 2H), 7.52 (m, 4H), 7.41 (t, J = 7.8 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 156.2, 137.2, 135.7, 133.5, 131.5, 129.6, 128.8, 127.9, 124.9, 124.0.

[7] T. B. Nguyen, L. Ermolenko, W. A. Dean, A. Al-Mourabit, *Org. Lett*. **2012**, *14*, 5948.

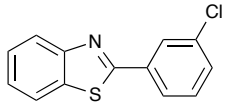
2-(4-Chlorophenyl)benzo[*d*]thiazole (5b)[8]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (d, J = 8.2 Hz, 1H), 8.02 (d, J = 8.4, 2H), 7.90 (d, J = 8.2 Hz, 1H), 7.46 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 154.1, 137.0, 135.1, 132.1, 129.2, 128.8, 126.6, 125.5, 123.3, 121.7.

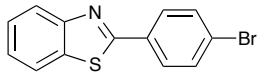
2-(3-Chlorophenyl)benzo[d]thiazole (5c)[9]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, J = 1.7 Hz, 1H), 8.06 (d, J = 8.5 Hz, 1H), 7.93-7.86 (m, 2H), 7.51-7.36 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 154.1, 135.4, 135.3, 135.2, 131.0, 130.4, 127.5, 126.7, 125.8, 125.7, 123.6, 121.8.

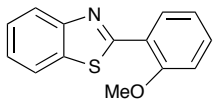
2-(4-Bromophenyl)benzo[*d*]thiazole (5d)[10]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.05 (d, J = 7.7 Hz, 1H), 7.95-7.90 (m, 2H), 7.87 (d, J = 7.9 Hz, 1H), 7.62-7.57 (m, 2H), 7.51-7.46 (m, 1H), 7.40-7.35 (m, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 154.2, 135.2, 132.7, 132.4, 129.1, 126.7, 125.6, 123.5, 121.8.

2-(2-Methoxyphenyl)benzo[*d*]thiazole (5e)[9]

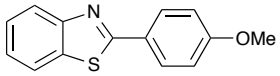


^{1}H NMR (300 MHz, $CDCl_3$) δ 8.58 (dd, J = 7.6, 1.6 Hz, 1H), 8.14 (d, J = 8.0 Hz, 1H), 7.94 (d, J = 8.0 Hz, 1H), 7.46 (m, 3H), 7.16 (t, J = 7.6 Hz, 1H), 7.06 (d, J = 8.0 Hz, 1H), 4.06 (s, 3H).

[8] Y. Liao, H. Qi, S. Chen, P. Jiang, W. Zhou, G. Deng, *Org. Lett.* **2012**, *14*, 6004.
[9] Z. Yang, X. Chen, S. Wang, J. Liu, K. Xie, A. Wang, Z. Tan, *J. Org. Chem.* **2012**, *77*, 7086.
[10] S. Liu, R. Chen, X. Guo, H. Yang, G. Deng, Chao-Jun Li, *Green Chem*. **2012**, *14*, 1577.

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2, 157.4, 152.2, 136.2, 131.9, 129.5, 125.9, 124.7, 122.9, 122.3, 121.2 (two carbons), 111.8, 55.8.

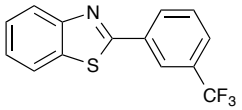
2-(4-Methoxyphenyl)benzo[*d*]thiazole (5f)[11]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04 (m, 3H), 7.87 (d, J = 8.0 Hz, 1H), 7.48 (t, J = 8.1 Hz, 1H), 7.36 (t, J = 8.0 Hz, 1H), 7.00 (d, J = 8.0 Hz, 1H), 3.87 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 167.9, 162.0, 154.3, 134.9, 133.9, 129.1, 126.3, 124.9, 123.0, 121.6, 114.5, 55.5.

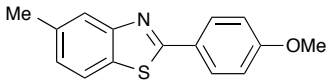
2-(3-(Trifluoromethyl)phenyl)benzo[*d*]thiazole (5g)[8]



^{1}H NMR (300 MHz, $CDCl_3$) δ 8.40 (s, 1H), 8.27 (d, J = 7.8 Hz, 1H), 8.14 (d, J = 8.0 Hz, 1H), 7.96 (d, J = 8.0 Hz, 1H), 7.77 (d, J = 8.0 Hz, 1H), 7.65 (t, J = 7.9 Hz, 1H), 7.55 (t, J = 7.9 Hz, 1H), 7.45 (t, J = 7.9 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 154.1, 135.3, 134.6, 131.8 (q, J = 32.3 Hz), 130.9, 129.8, 127.6 (q, J = 3.5 Hz), 126.9, 125.9, 124.5 (q, J = 3.5 Hz), 124.0 (q, J = 274 Hz), 123.7, 122.0.

2-(4-Methoxyphenyl)-5-methylbenzo[*d*]thiazole (5h)



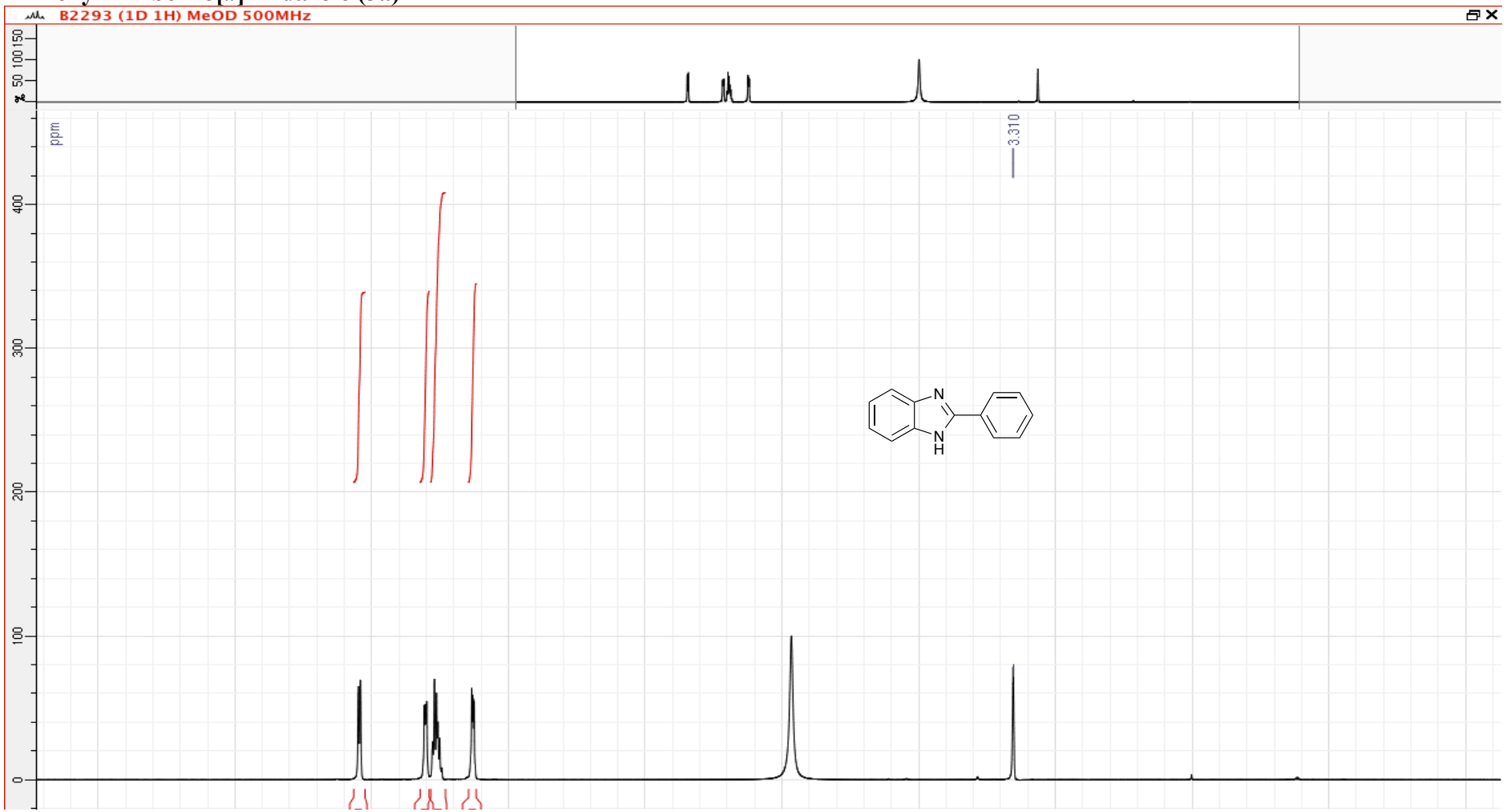
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, J = 8.7 Hz, 2H), 7.85 (s, 1H), 7.75 (d, J = 8.1 Hz, 1H), 7.19 (d, J = 8.1 Hz, 1H), 7.00 (d, J = 8.7 Hz, 2H), 3.91 (s, 3H), 2.53 (s, 3H).

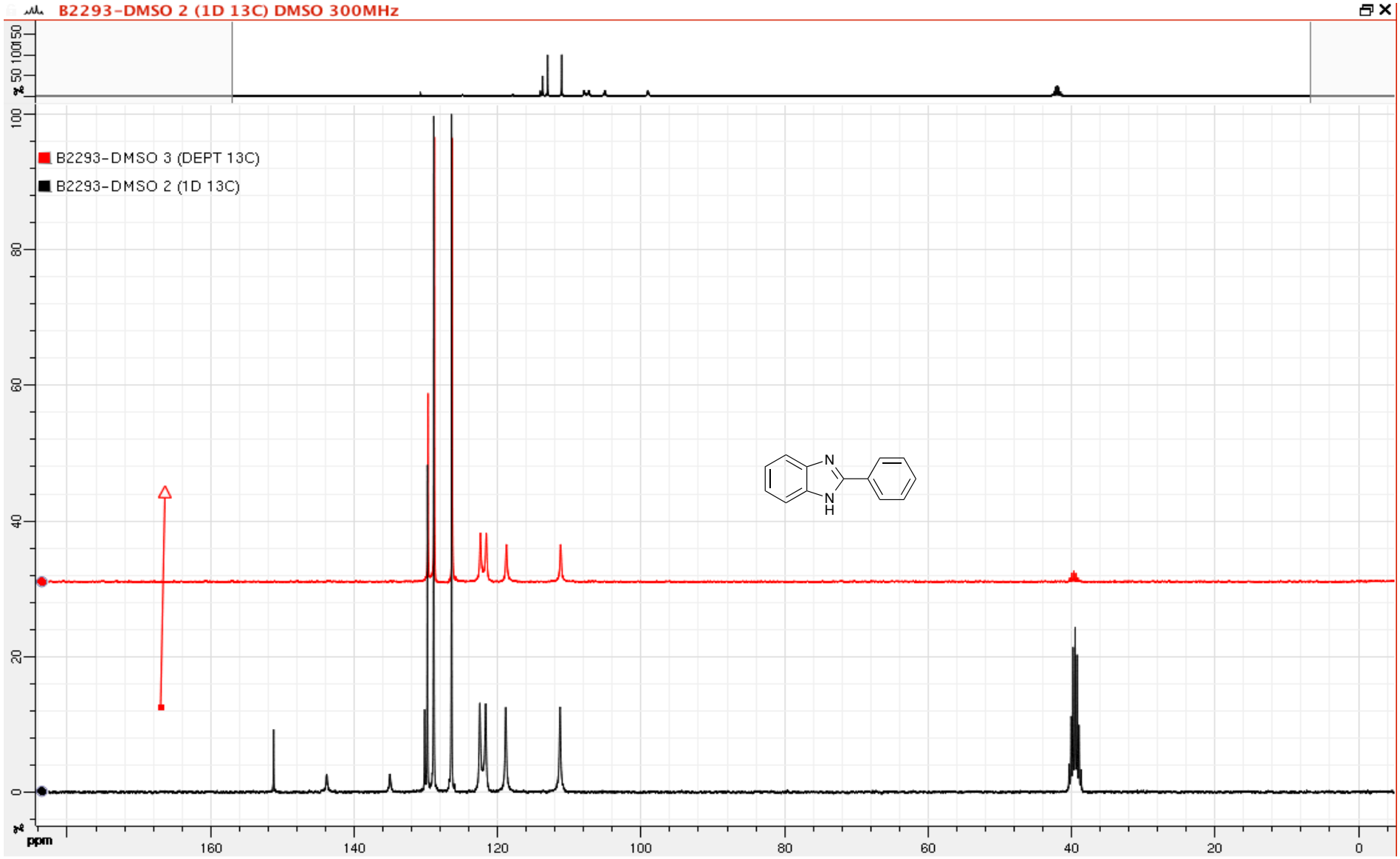
^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 161.8, 154.5, 136.3, 131.8, 129.1, 126.5, 126.4, 122.9, 121.0, 114.5, 55.5, 21.4.

HRMS-ESI+: m/z [M + H]+ calcd for $C_{15}H_{14}NOS$: 256.0796; found: 277.0753.

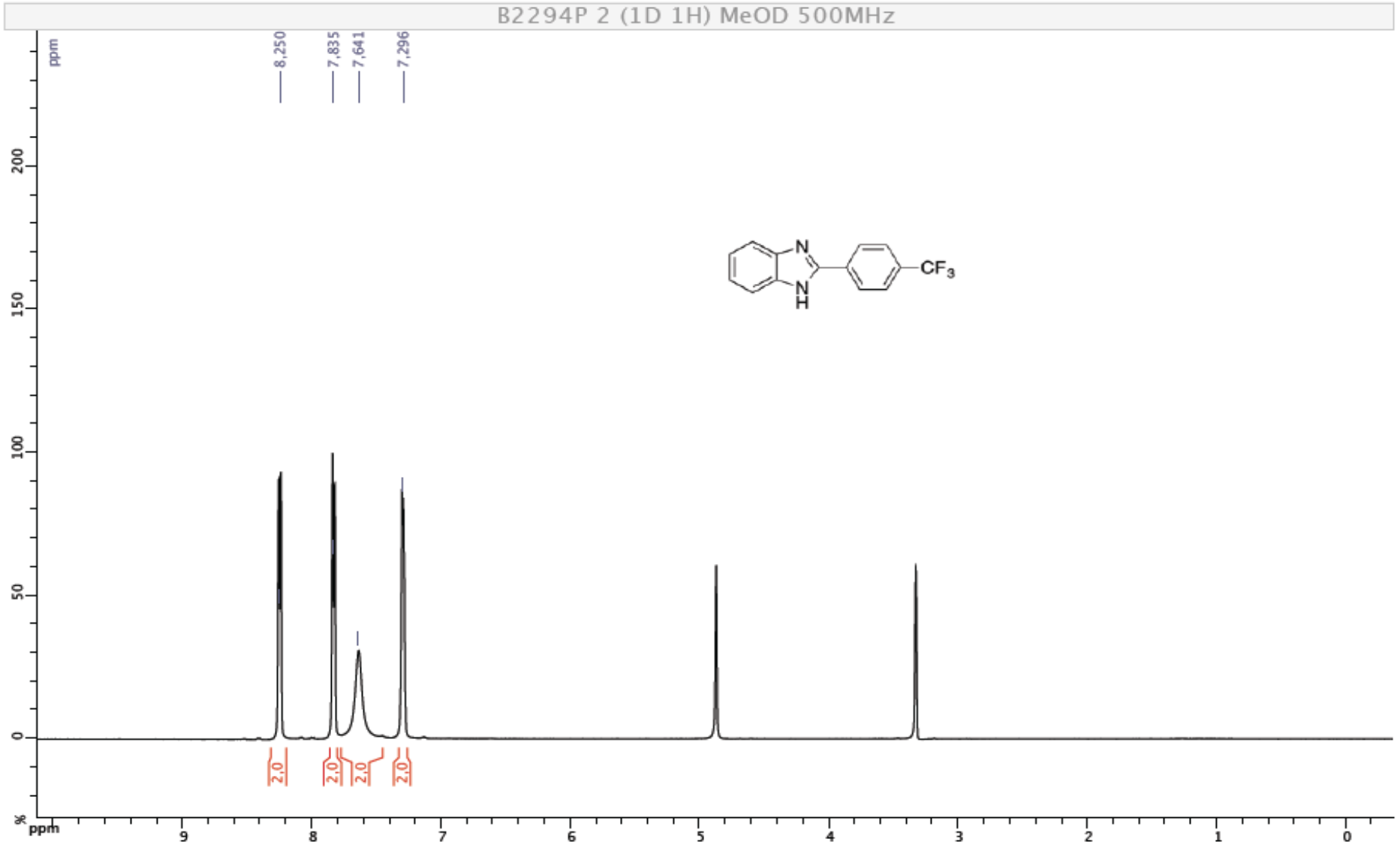
[11] T. B. Nguyen, L. Ermolenko, A. Al-Mourabit, *Green Chem*. **2013**, *15*, 2713.

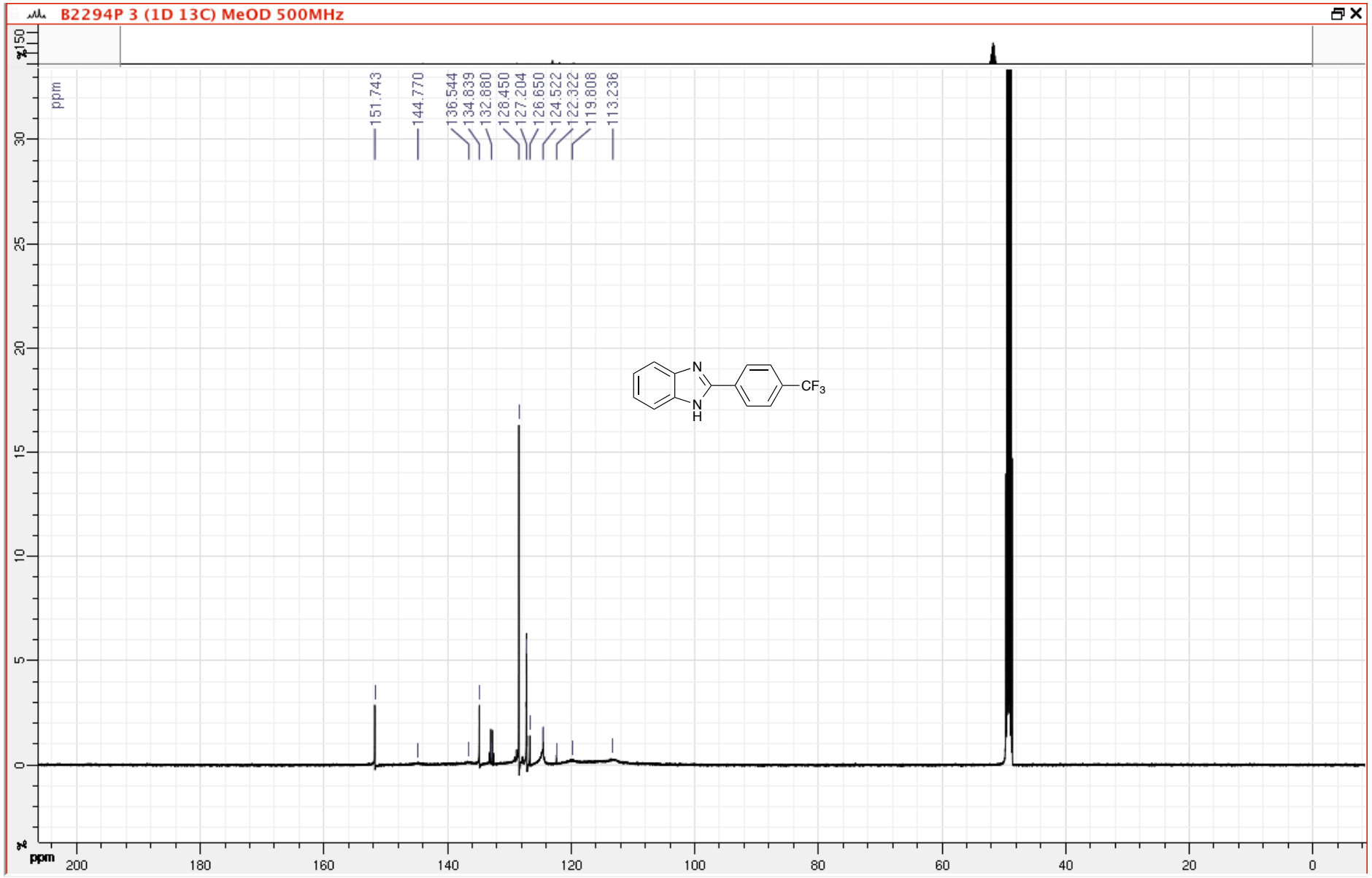
2-Phenyl-1*H*-benzo[*d*]imidazole (3a)



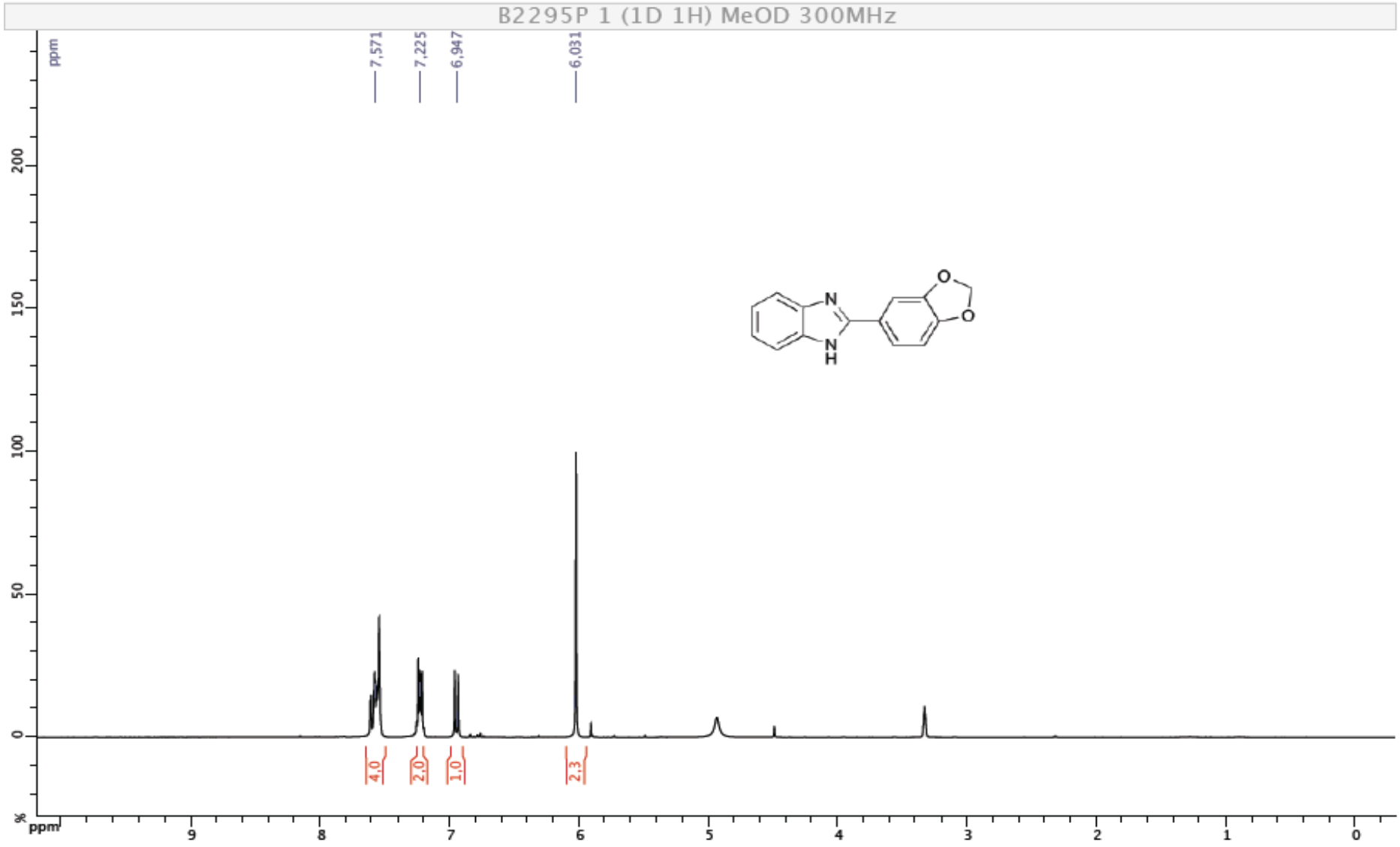

B2293-DMSO 2 (1D 13C) DMSO 300MHz
B2293-DMSO 3 (DEPT 13C)
B2293-DMSO 2 (1D 13C)
N
N
H
ppm
160
140
120
100
80
60
40
20
0

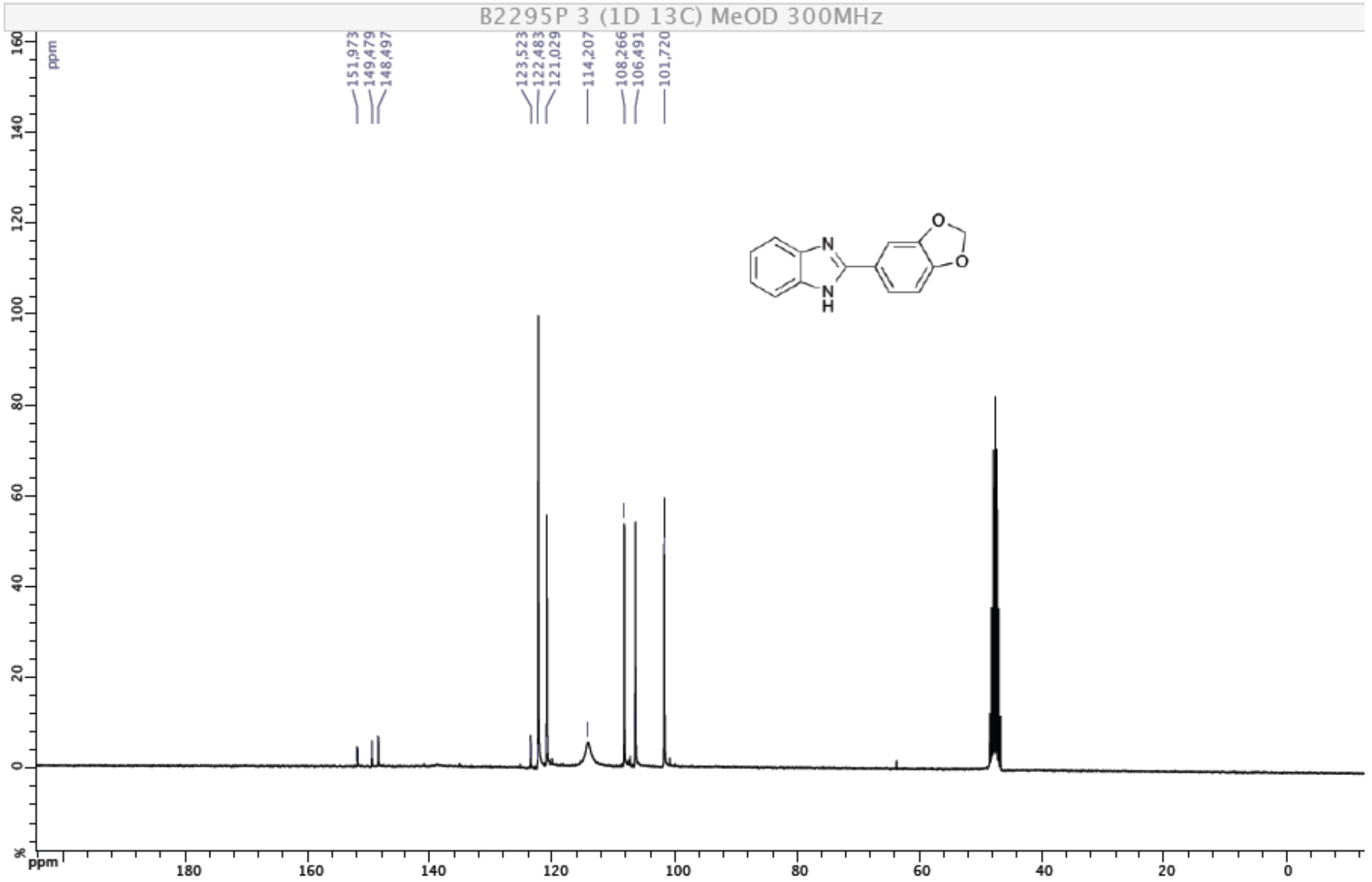
2-(4-(Trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazole (3b)



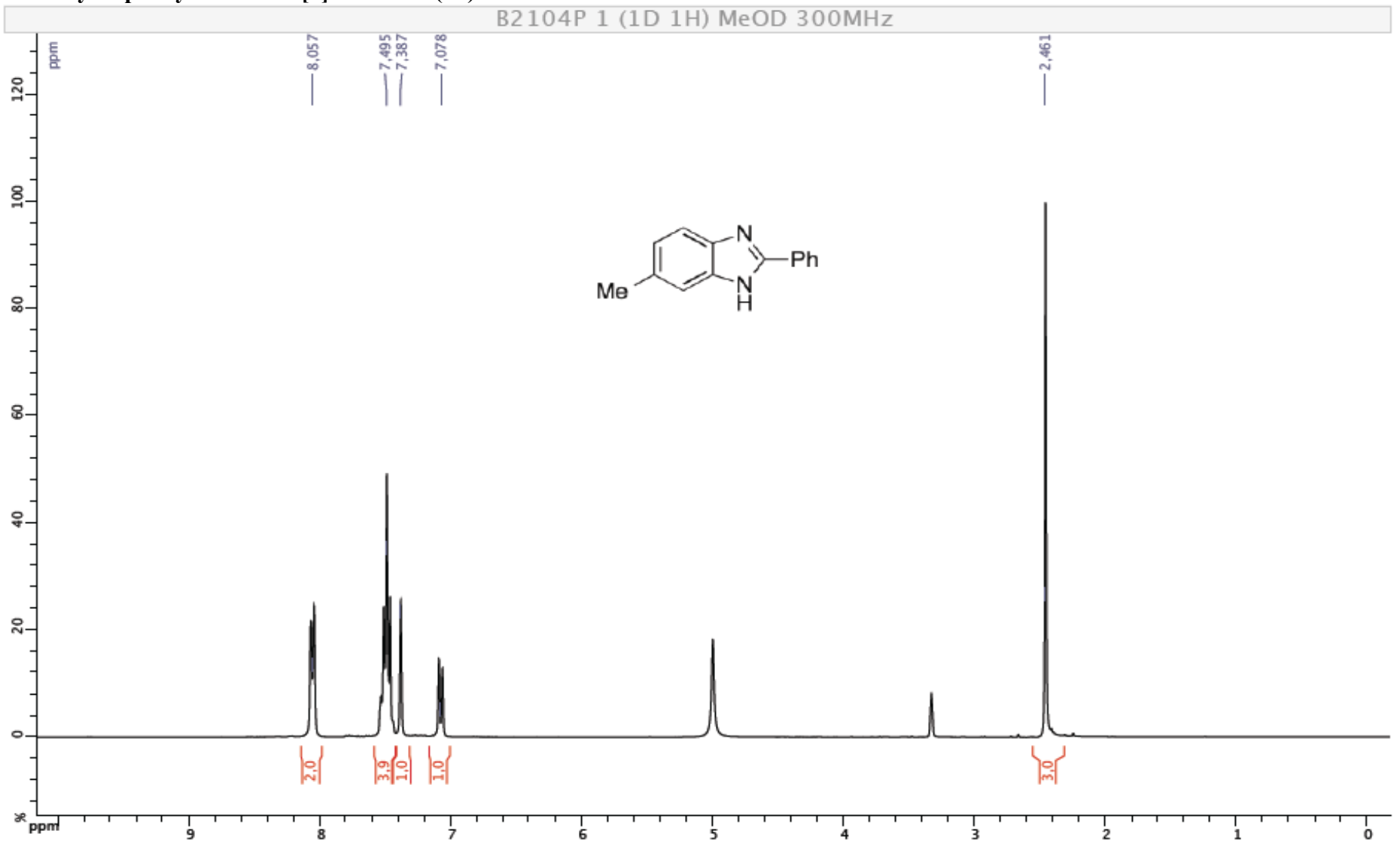

B2294P 3 (1D 13C) MeOD 500MHz
ppm
151.743
144.770
136.544
134.839
132.880
128.450
127.204
126.650
124.522
122.322
119.808
113.236
N
N
H
CF3
200
180
160
140
120
100
80
60
40
20
0

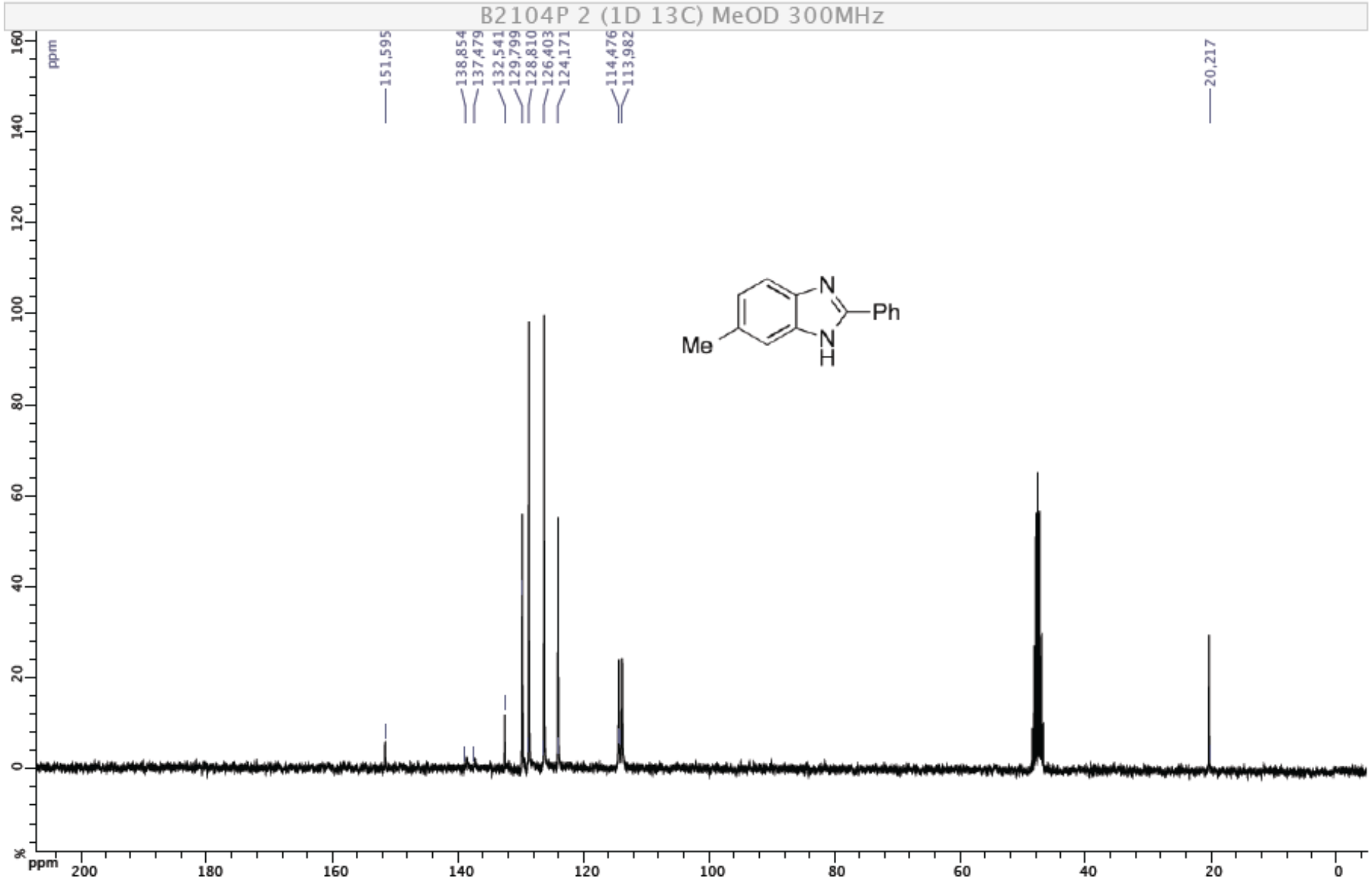
2-(Benzo[*d*][1,3]dioxol-5-yl)-1*H*-benzo[*d*]imidazole (3c)



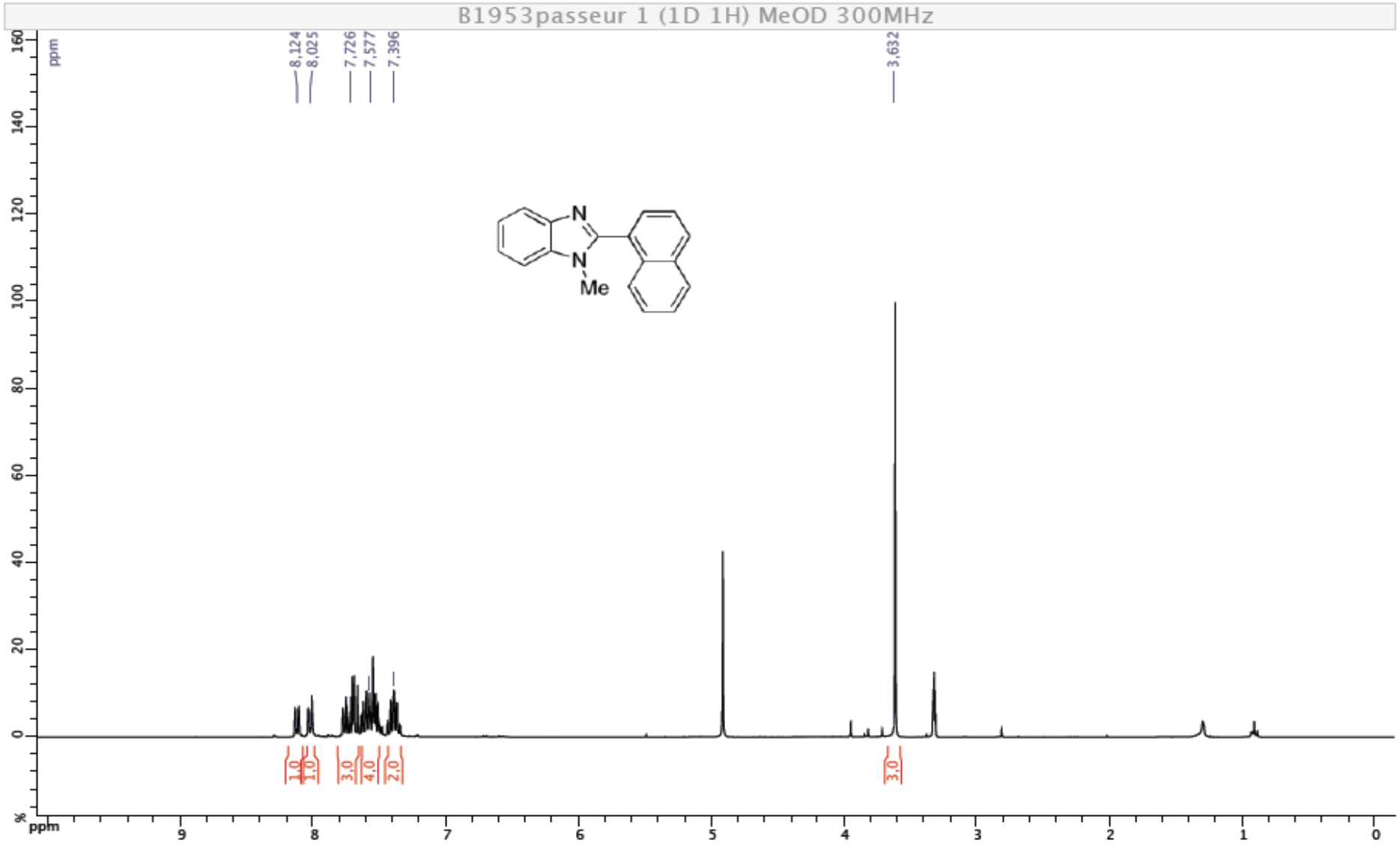

B2295P 3 (1D 13C) MeOD 300MHz
ppm
151,973
149,479
148,497
123,523
122,483
121,029
114,207
108,266
106,491
101,720
N
N
H
O
O
%
ppm
180
160
140
120
100
80
60
40
20
0

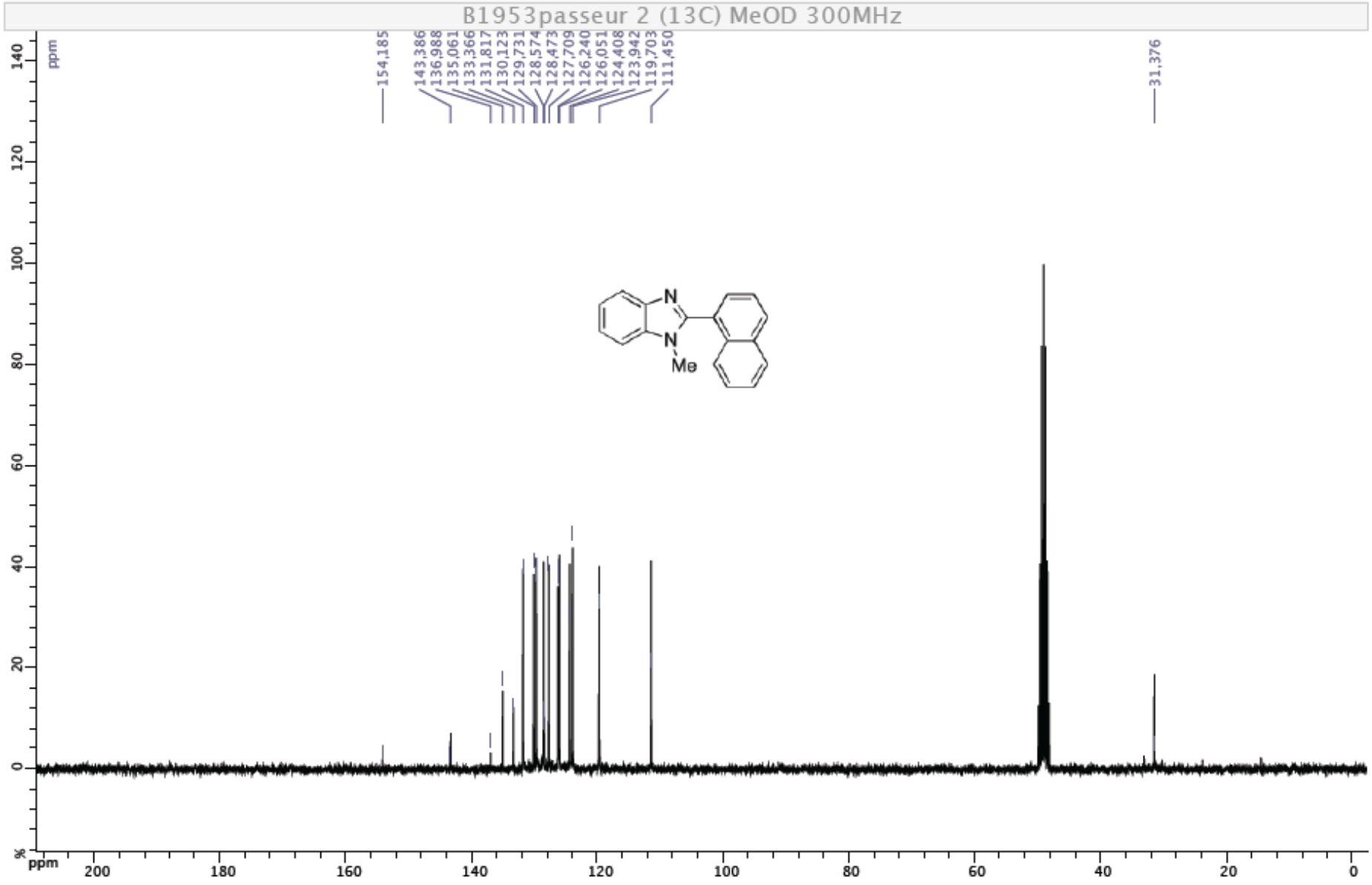
6-Methyl-2-phenyl-1*H*-benzo[*d*]imidazole (3d)



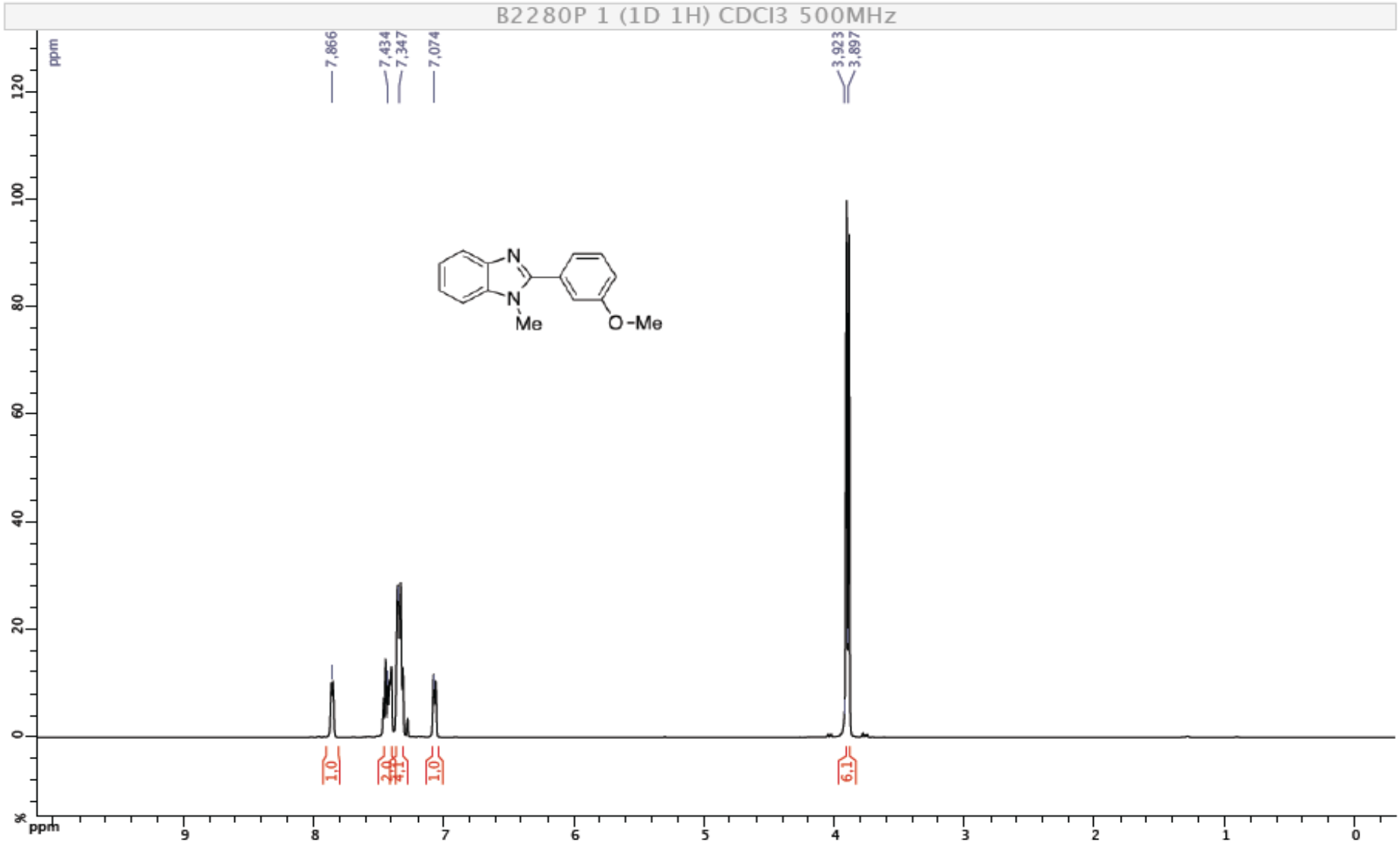

B2104P 2 (1D 13C) MeOD 300MHz
ppm
151,595
138,854
137,479
132,541
129,799
128,810
126,403
124,171
114,476
113,982
20,217
N
Ph
Me
N
H
%
ppm
200
180
160
140
120
100
80
60
40
20
0

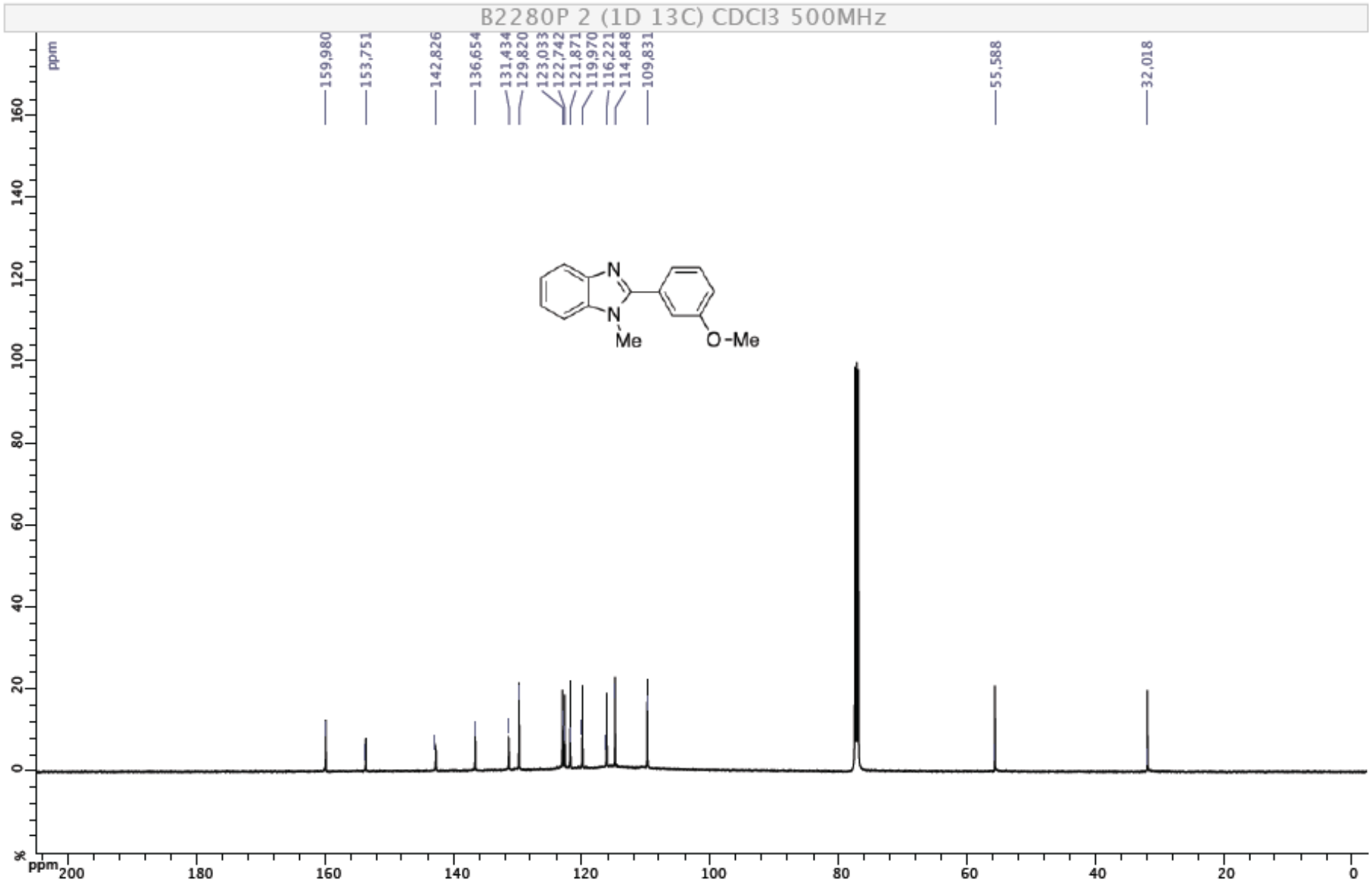
1-Methyl-2-(naphthalen-1-yl)-1*H*-benzo[*d*]imidazole (3e)



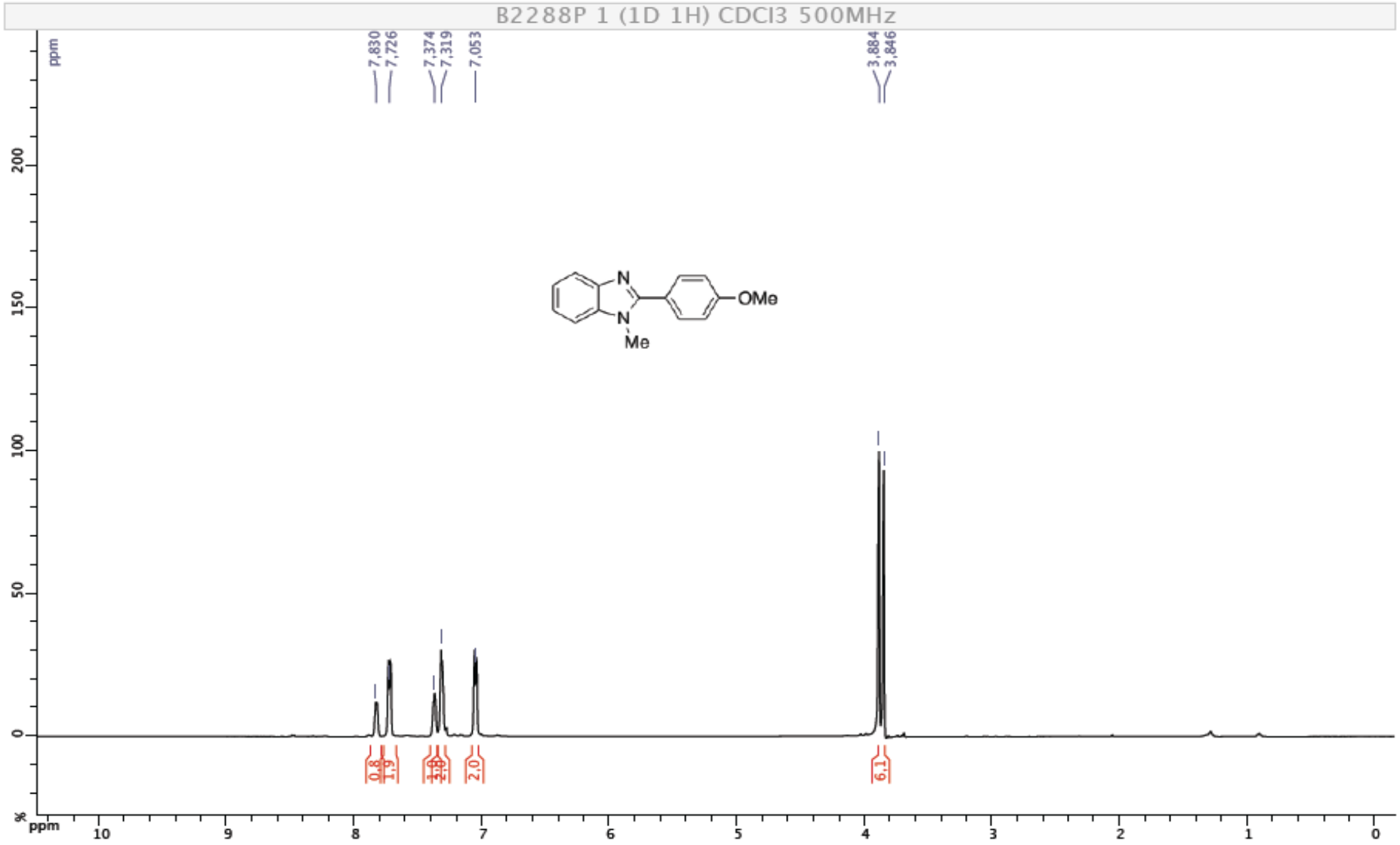

B1953passeur 2 (13C) MeOD 300MHz
ppm
154,185
143,386
136,988
135,061
133,366
131,817
130,123
129,731
128,574
128,473
127,709
126,240
126,051
124,408
123,942
119,703
111,450
31,376
N
N
Me
%
ppm
200
180
160
140
120
100
80
60
40
20
0

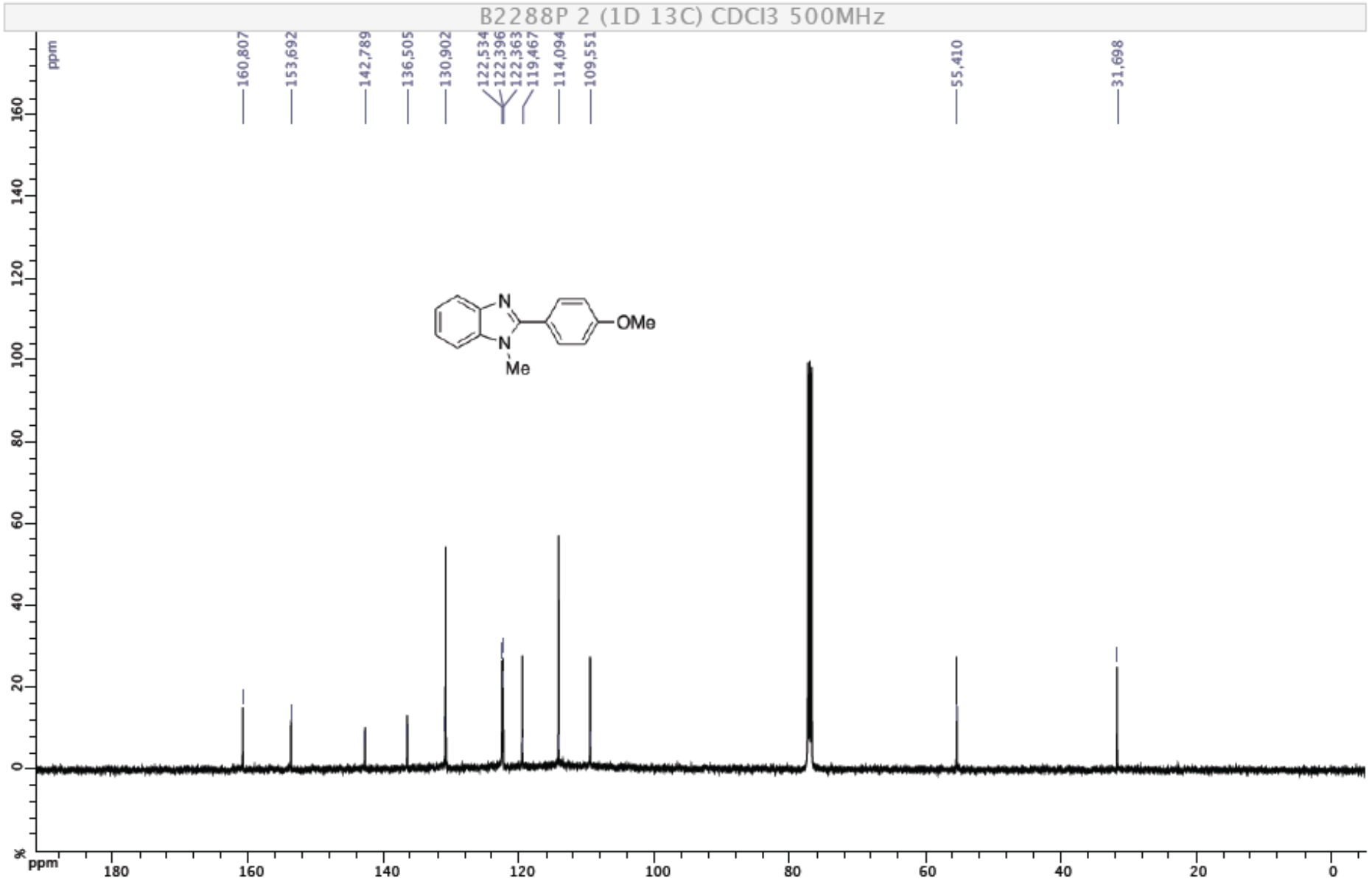
2-(3-Methoxyphenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3f)



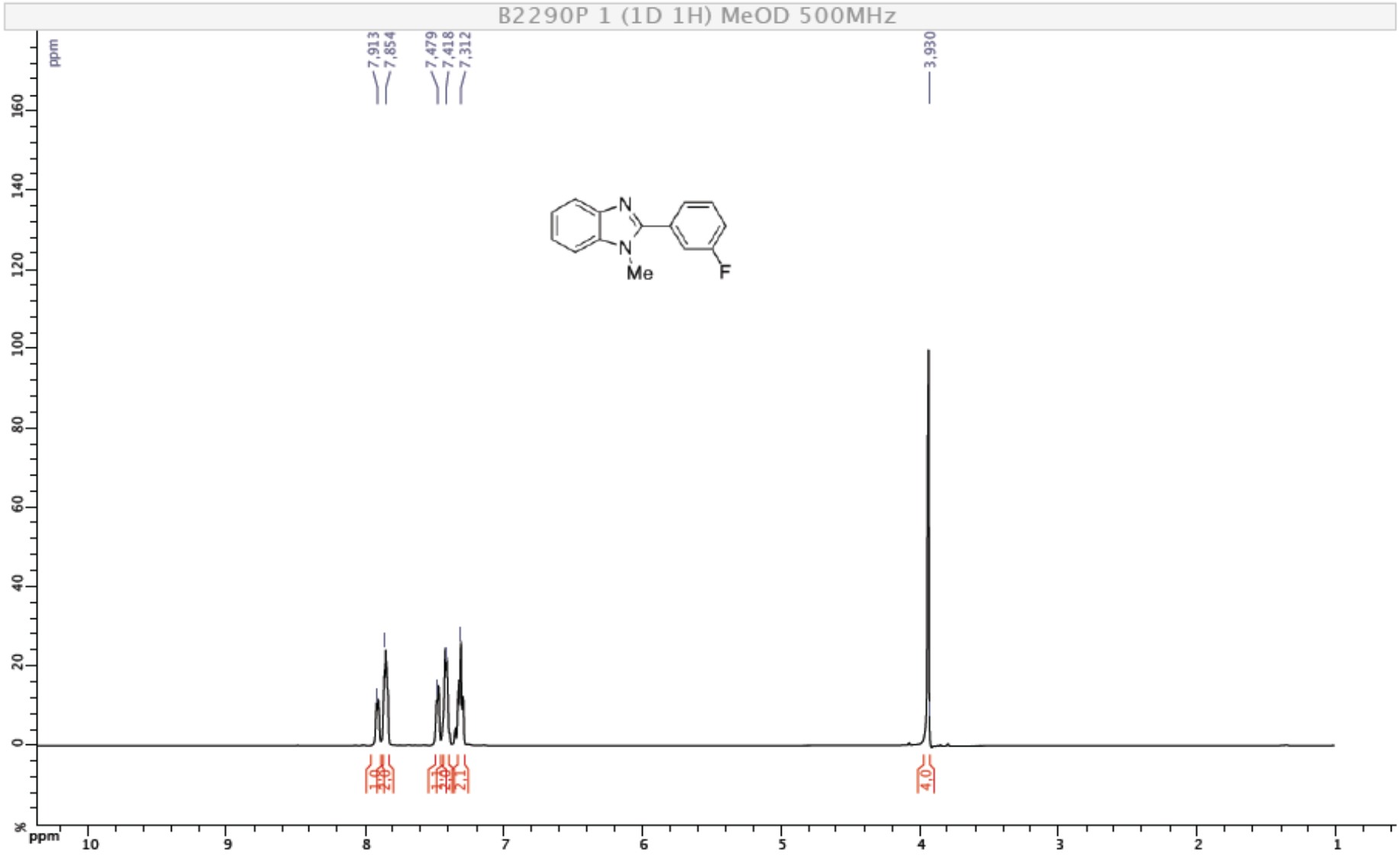

B2280P 2 (1D 13C) CDCl3 500MHz
ppm
159,980
153,751
142,826
136,654
131,434
129,820
123,033
122,742
121,871
119,970
116,221
114,848
109,831
55,588
32,018
N
N
Me
O-Me
%
ppm
200
180
160
140
120
100
80
60
40
20
0

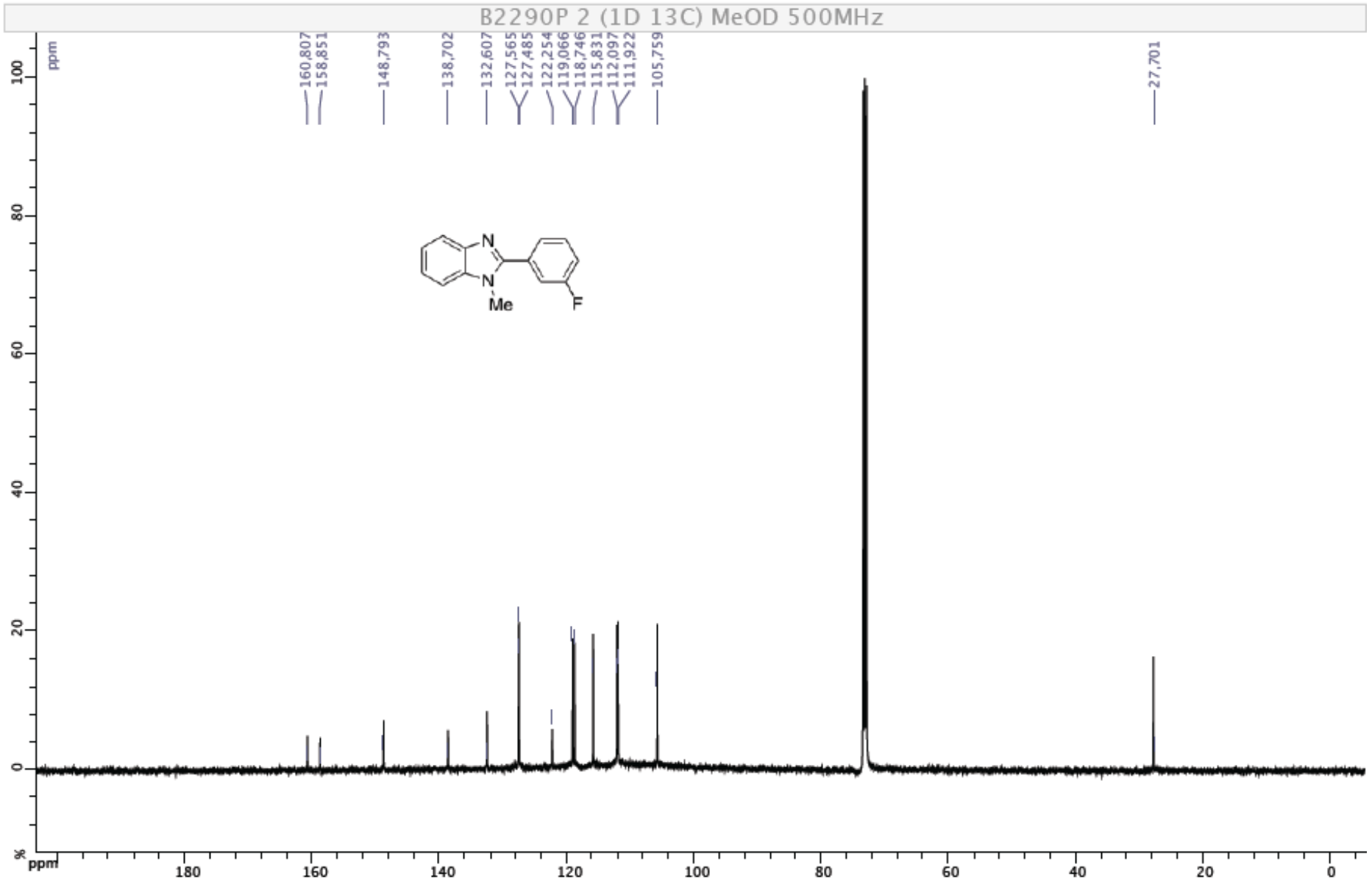
2-(4-Methoxyphenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3g)



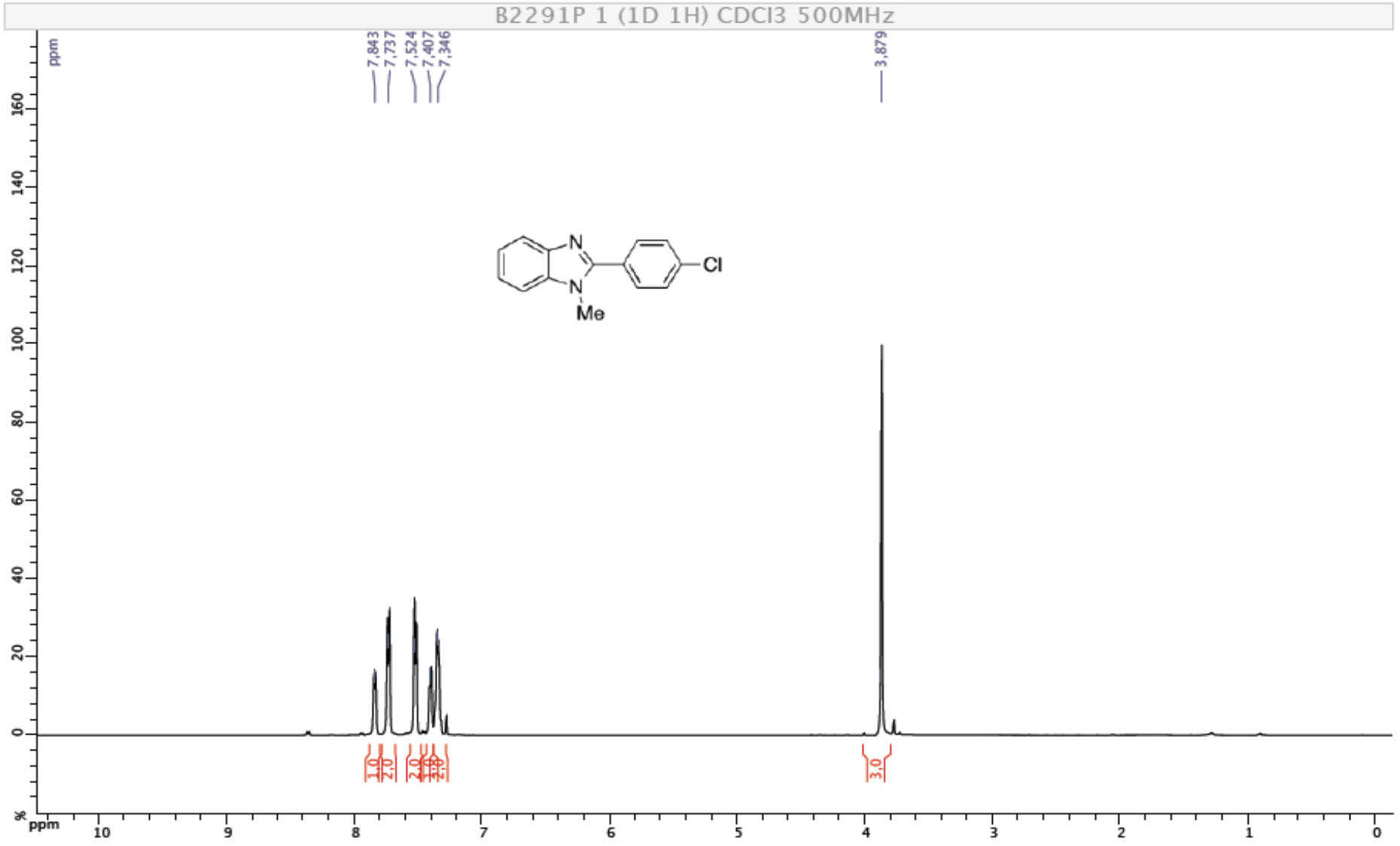

B2288P 2 (1D 13C) CDCl3 500MHz
ppm
160,807
153,692
142,789
136,505
130,902
122,534
122,396
122,363
119,467
114,094
109,551
55,410
31,698
N
N
Me
OMe
%
ppm
180
160
140
120
100
80
60
40
20
0

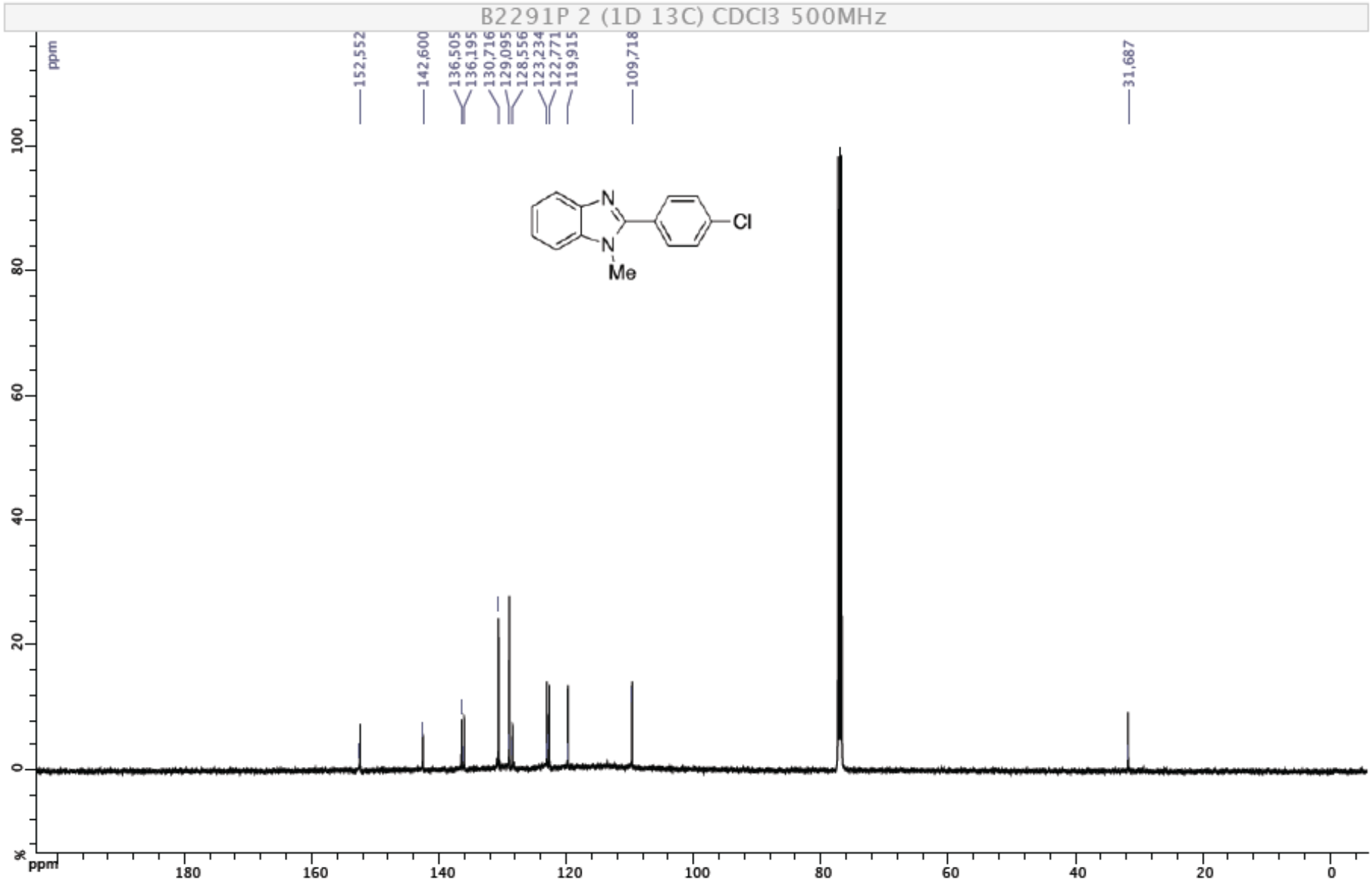
2-(3-Fluorophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3h)



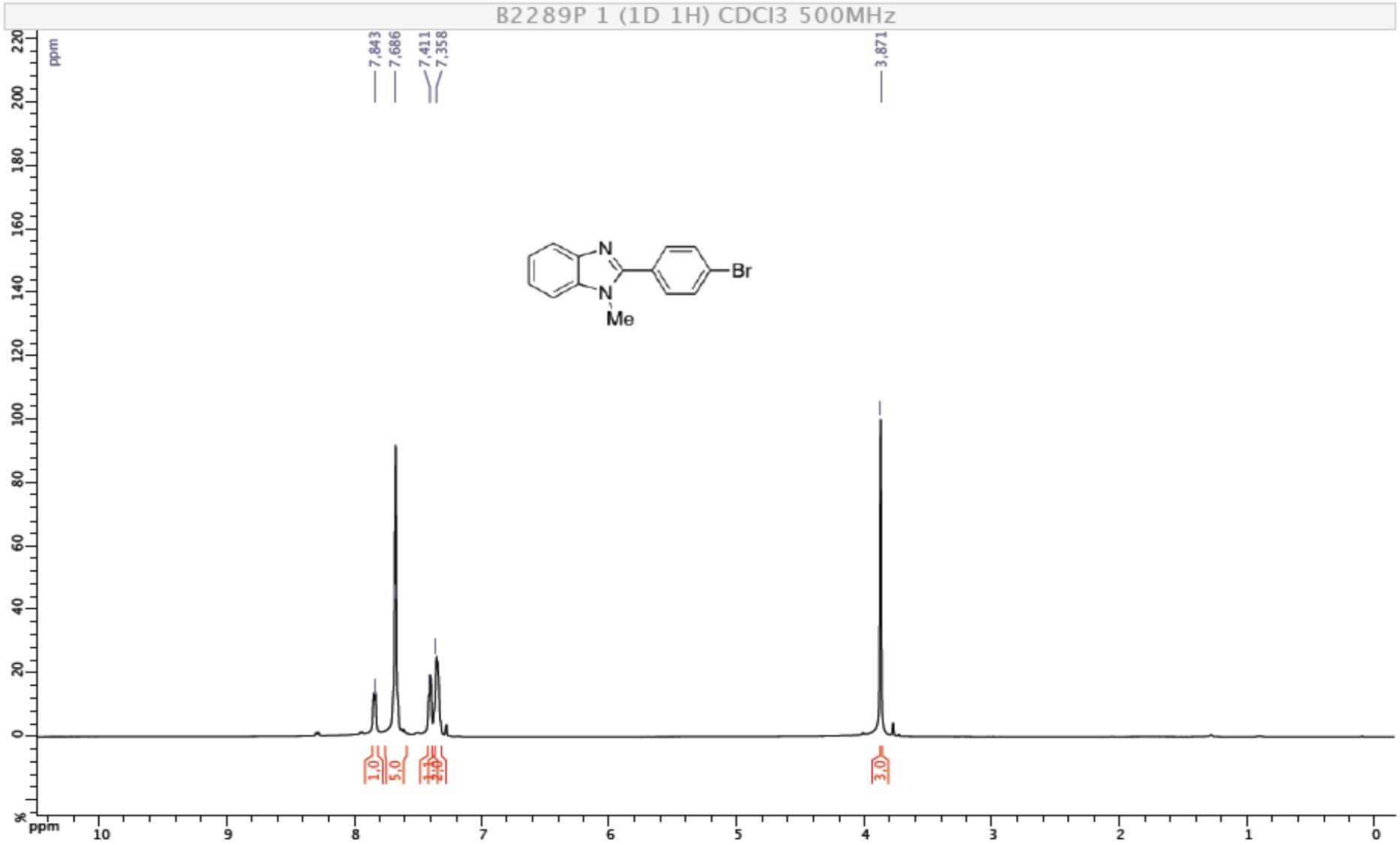

B2290P 2 (1D 13C) MeOD 500MHz
ppm
160,807
158,851
148,793
138,702
132,607
127,565
127,485
122,254
119,066
118,746
115,831
112,097
111,922
105,759
27,701
N
N
Me
F
%
ppm
180
160
140
120
100
80
60
40
20
0

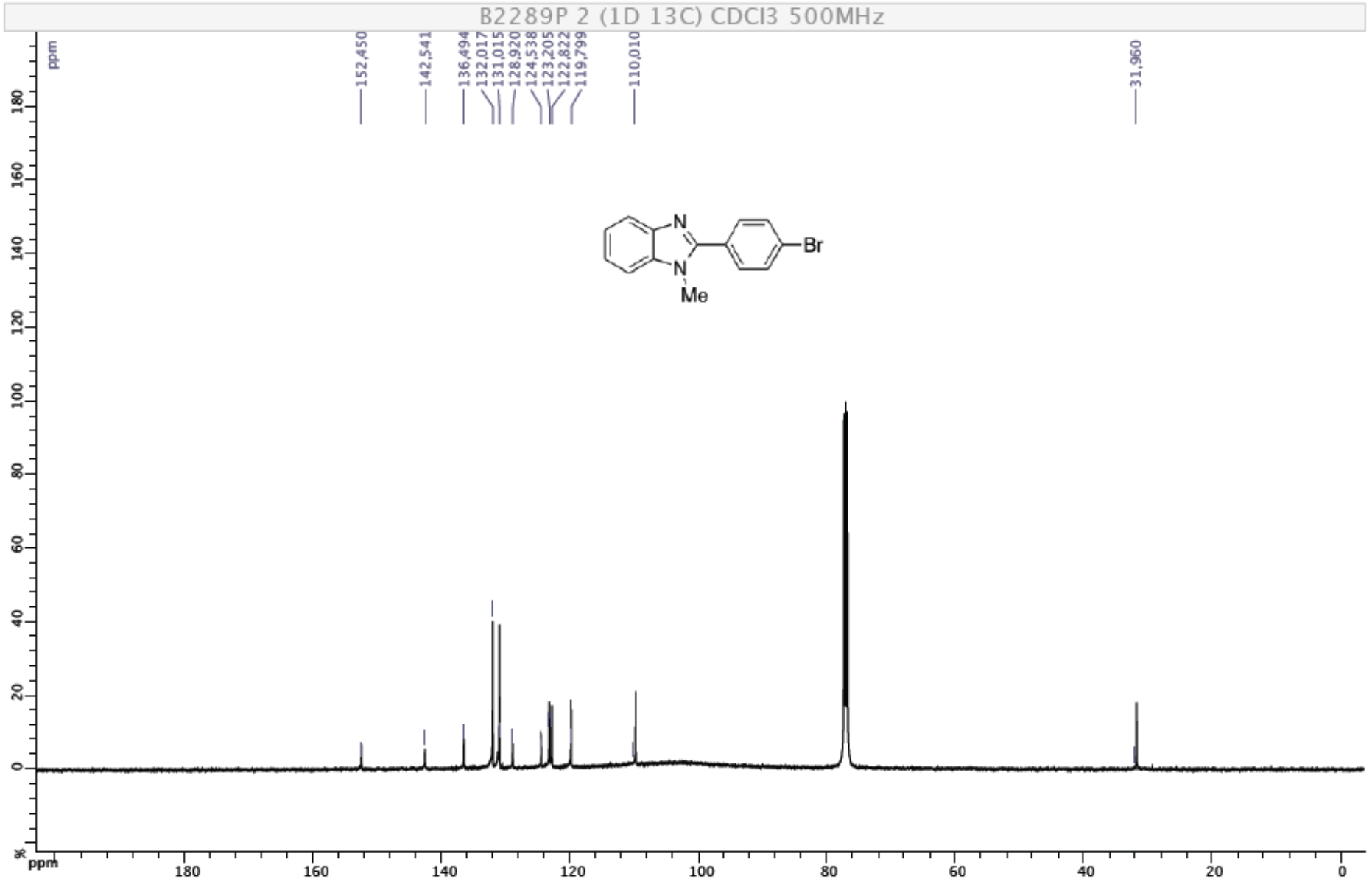
2-(4-Chlorophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3i)



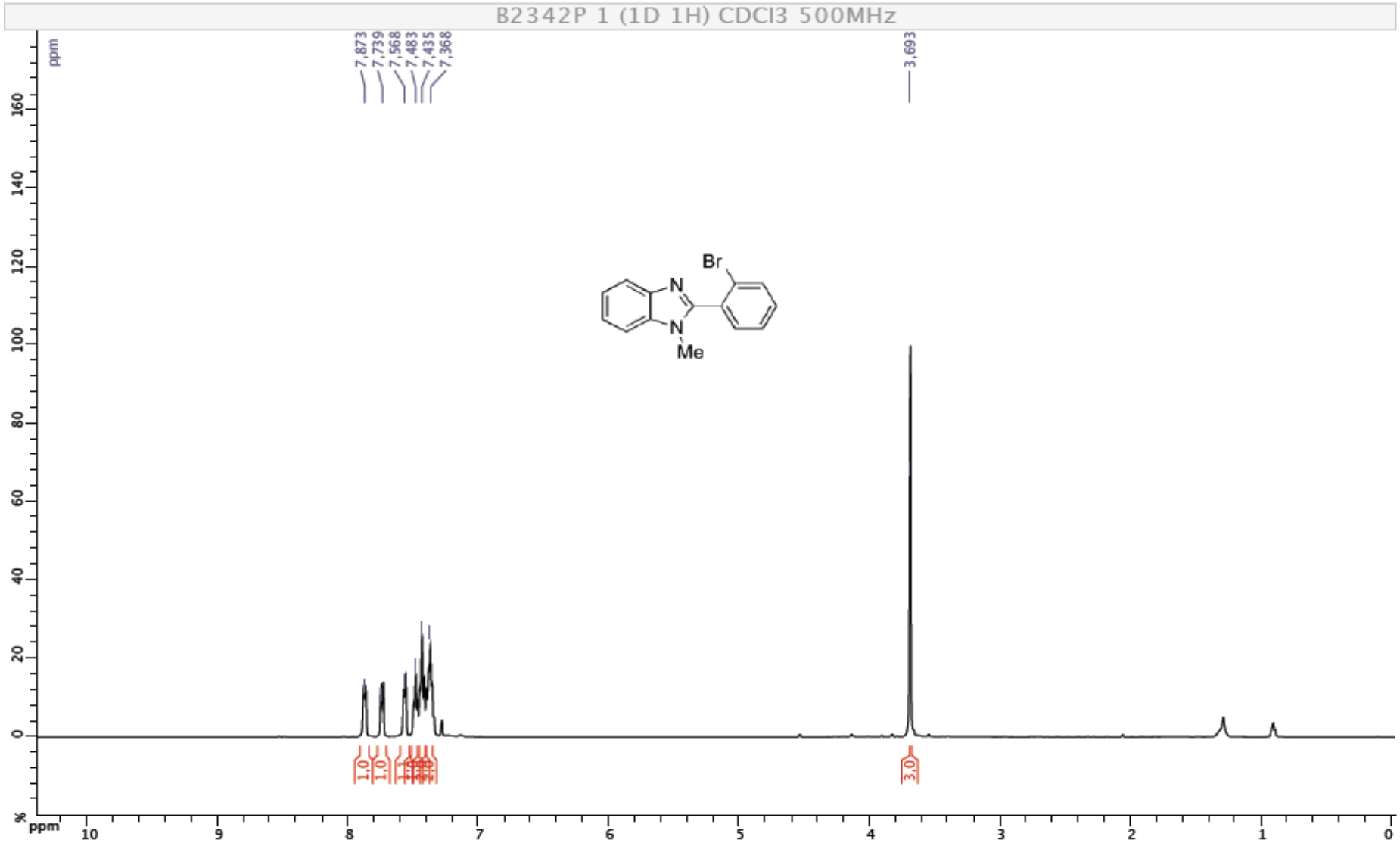

B2291P 2 (1D 13C) CDCl3 500MHz
ppm
152,552
142,600
136,505
136,195
130,716
129,095
128,556
123,234
122,771
119,915
109,718
31,687
N
N
Me
Cl
100
80
60
40
20
0
%
ppm
180
160
140
120
100
80
60
40
20
0

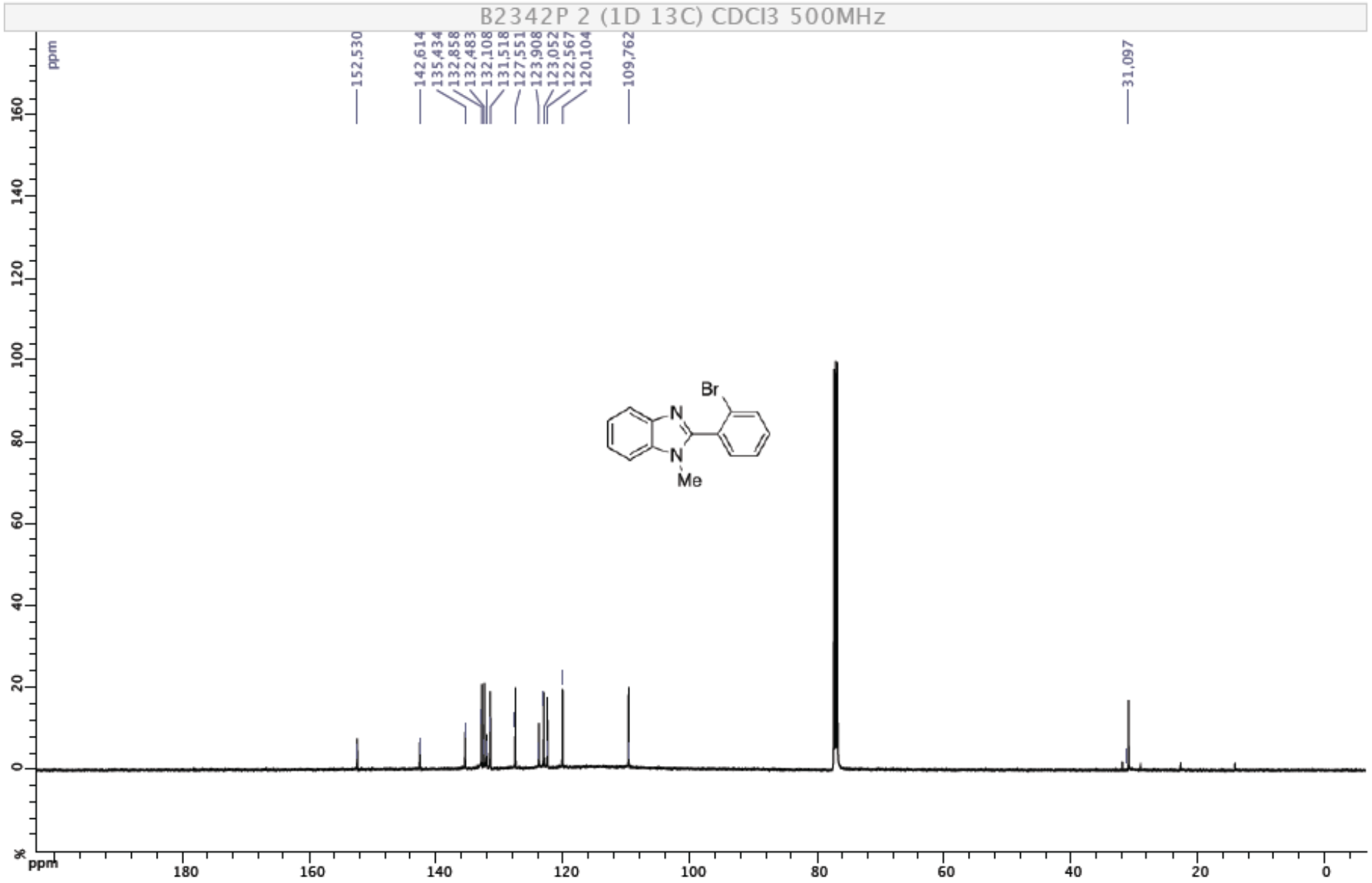
2-(4-Bromophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3j)



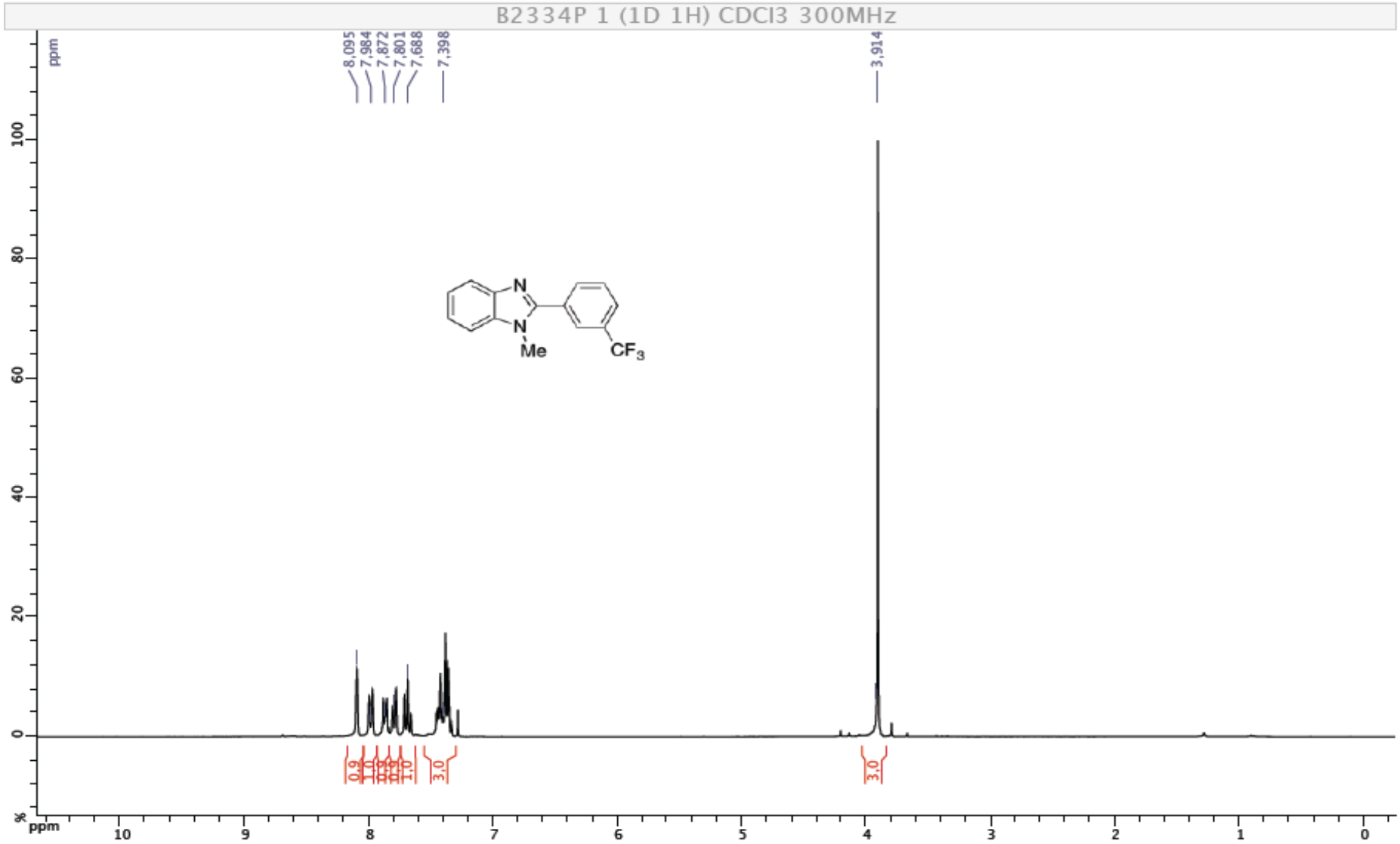

B2289P 2 (1D 13C) CDCl3 500MHz
ppm
152,450
142,541
136,494
132,017
131,015
128,920
124,538
123,205
122,822
119,799
110,010
31,960
N
N
Me
Br
%
ppm
180
160
140
120
100
80
60
40
20
0

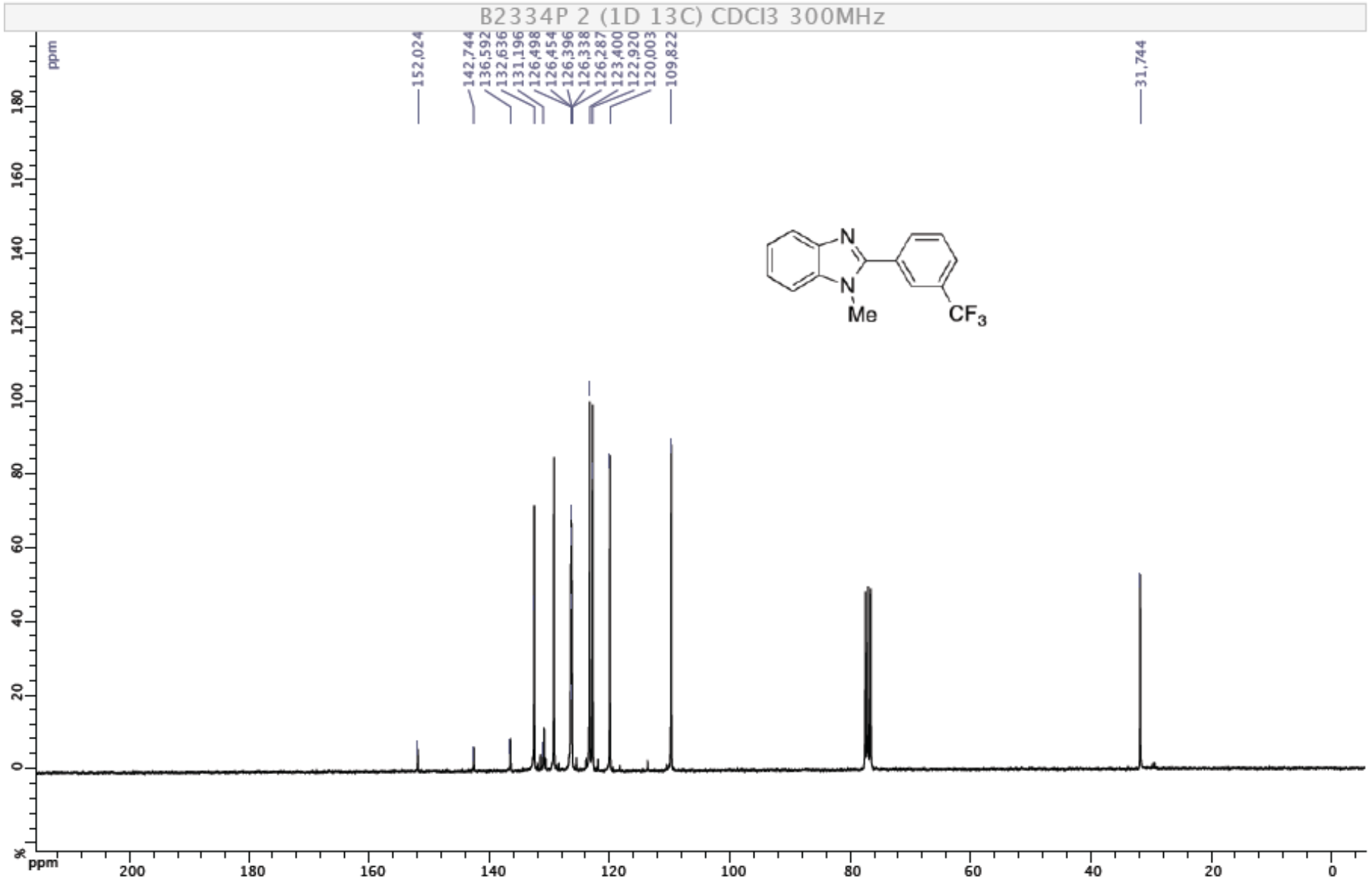
2-(2-Bromophenyl)-1-methyl-1*H*-benzo[*d*]imidazole (3k)



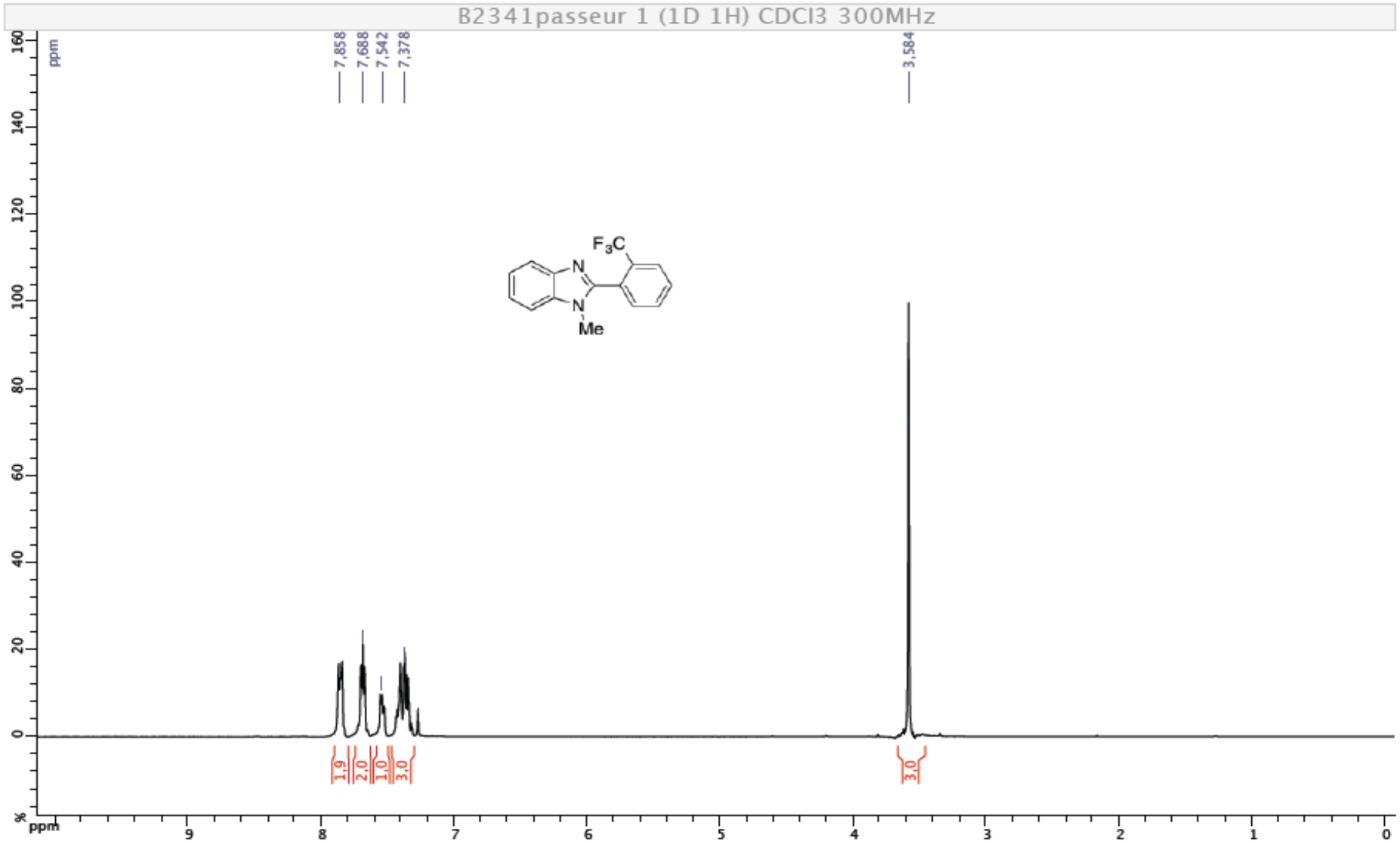

B2342P 2 (1D 13C) CDCl3 500MHz
ppm
152,530
142,614
135,434
132,858
132,483
132,108
131,518
127,551
123,908
123,052
122,567
120,104
109,762
31,097
Br
N
N
Me
%
ppm
180
160
140
120
100
80
60
40
20
0

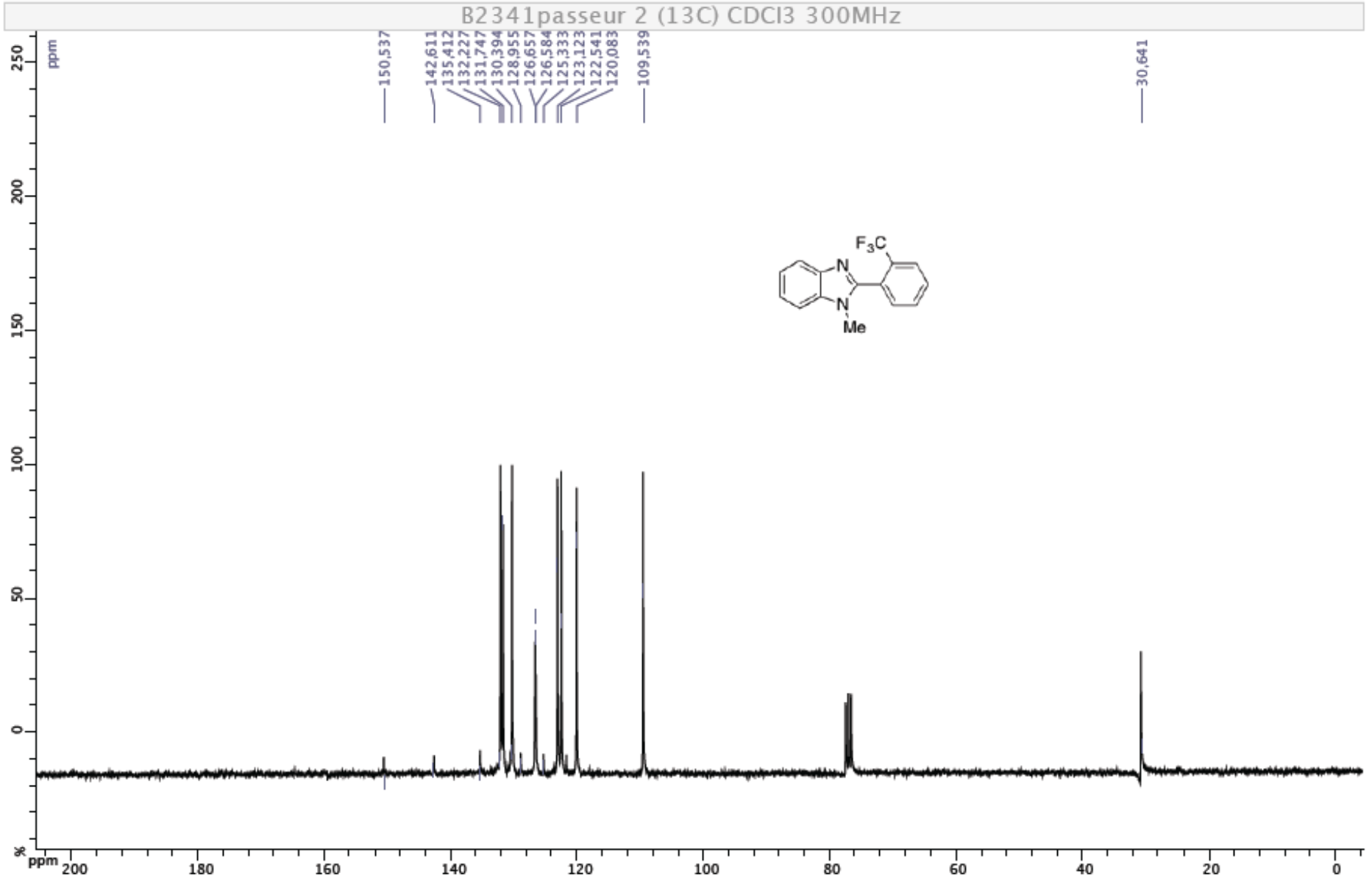
1-Methyl-2-(3-(trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazole (3l)



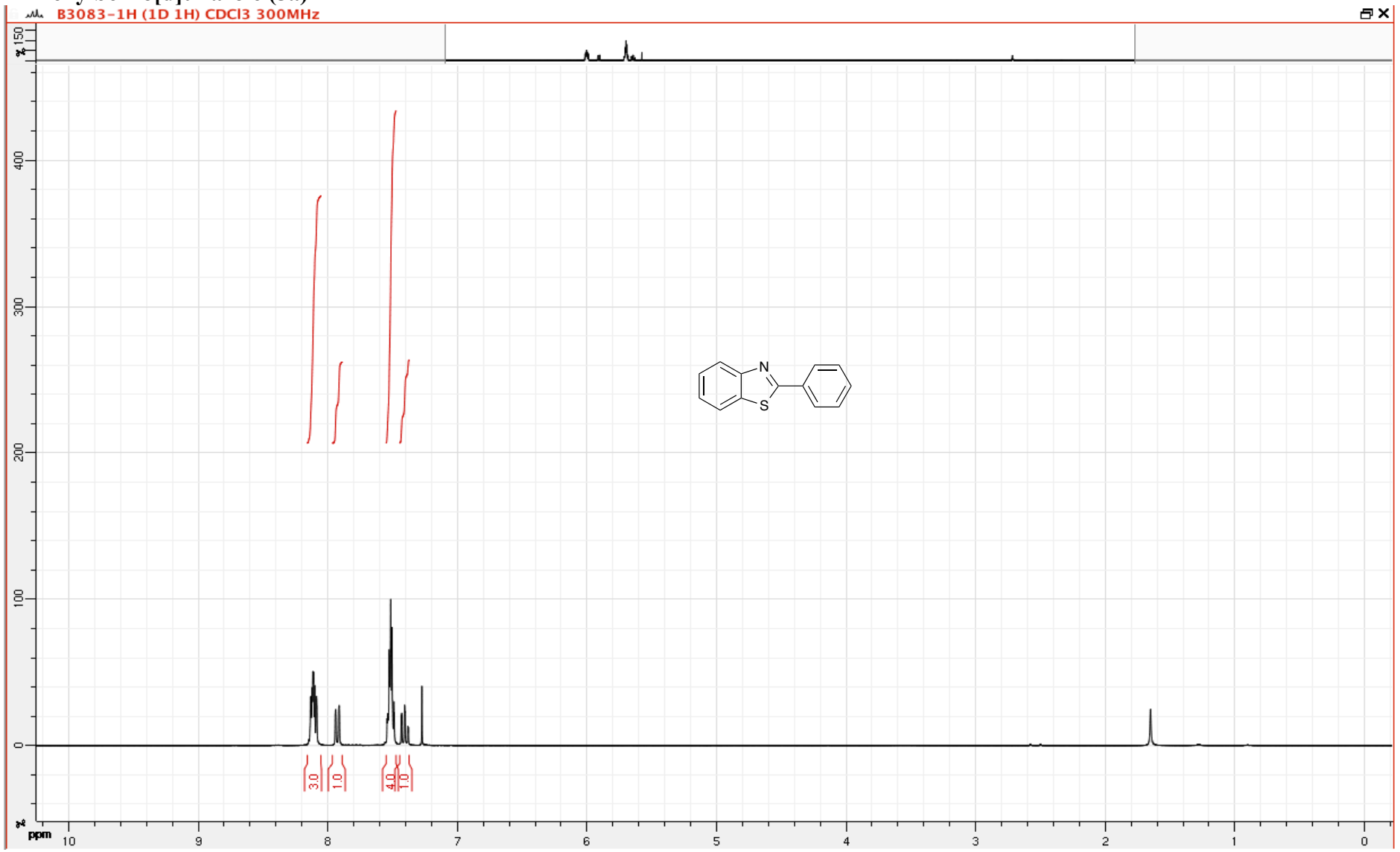

B2334P 2 (1D 13C) CDCl3 300MHz
ppm
152,024
142,744
136,592
132,636
131,196
126,498
126,454
126,396
126,338
126,287
123,400
122,920
120,003
109,822
31,744
N
N
Me
CF3
%
ppm
200
180
160
140
120
100
80
60
40
20
0

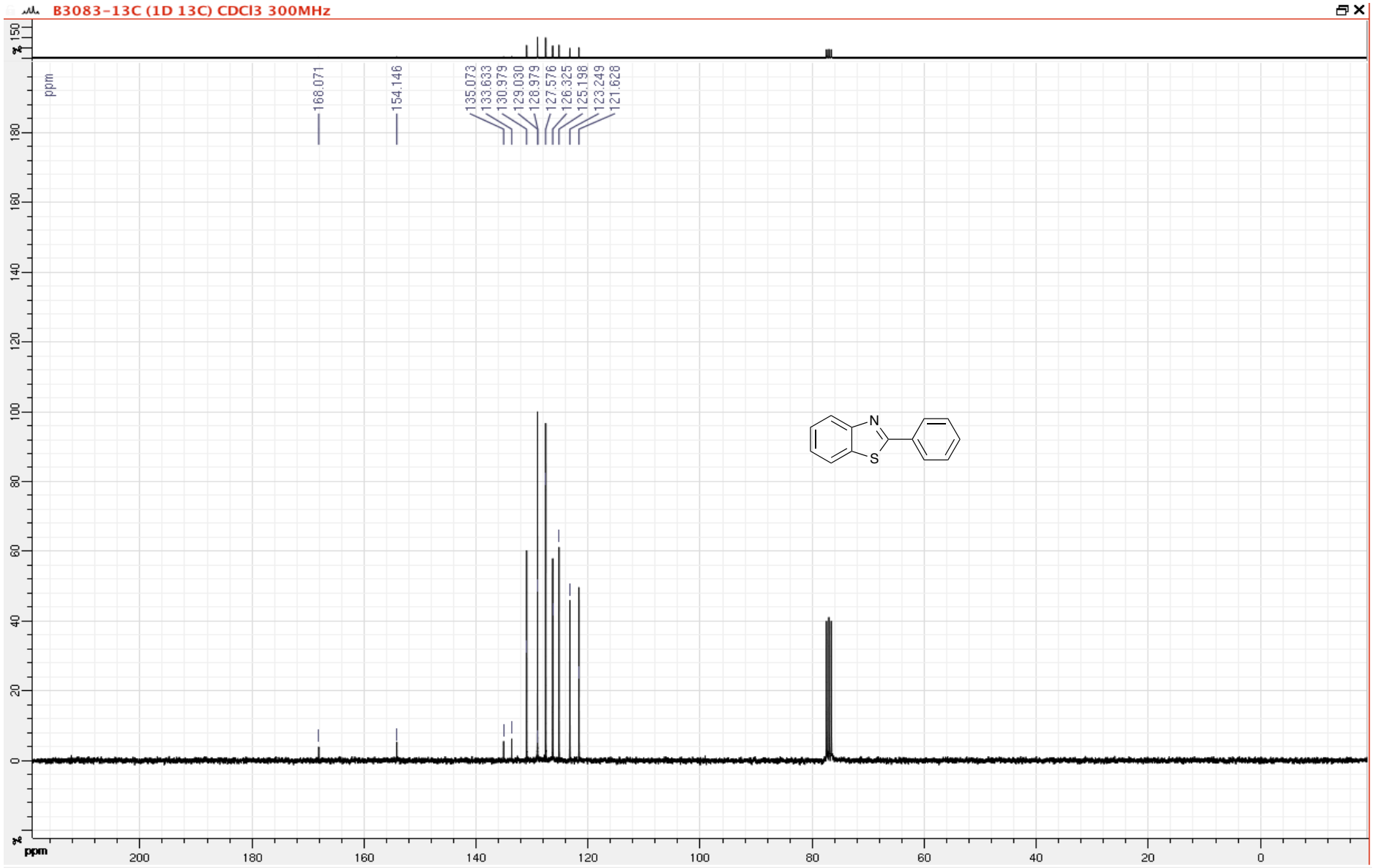
1-Methyl-2-(2-(trifluoromethyl)phenyl)-1*H*-benzo[*d*]imidazole (3m)



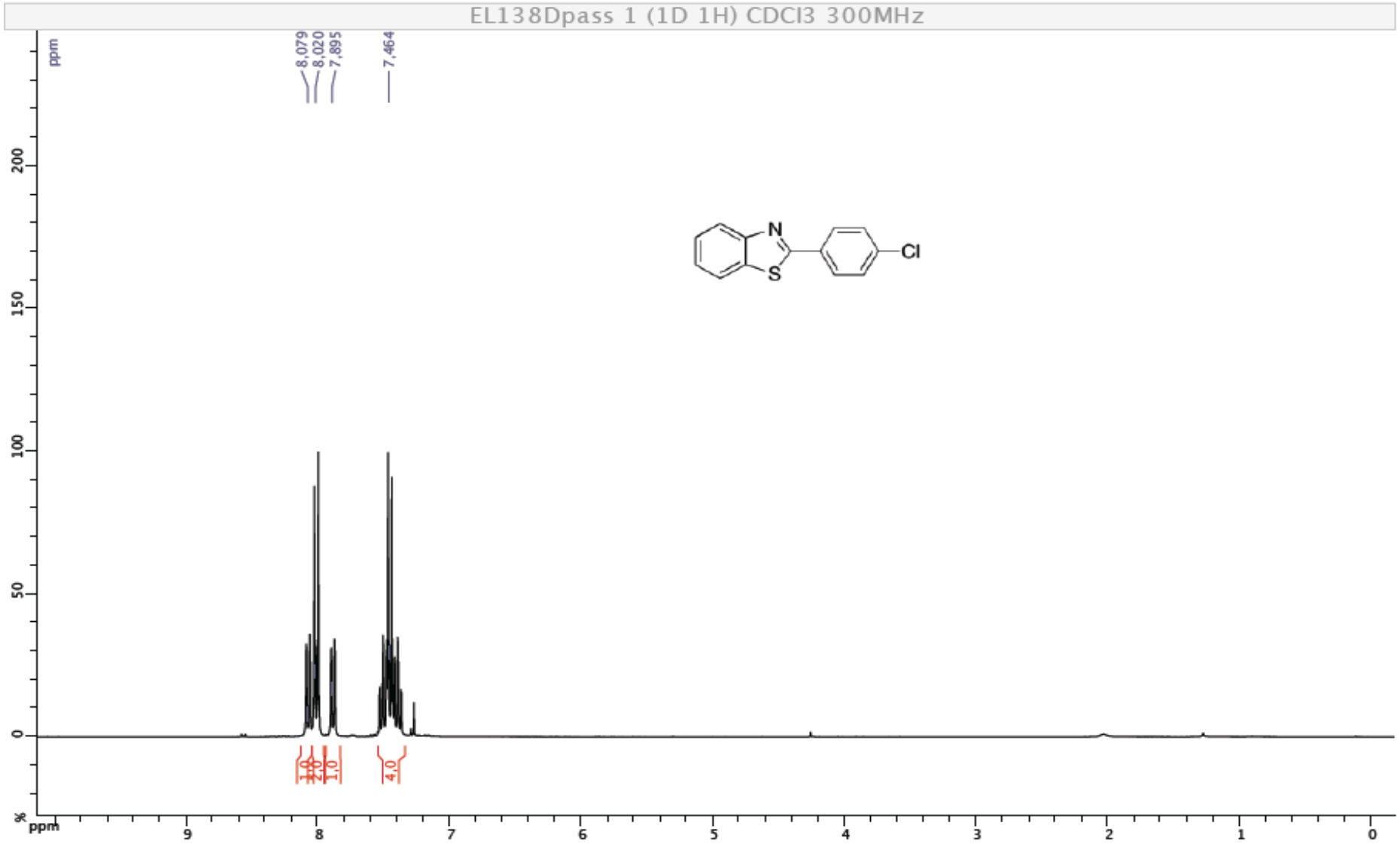

B2341passeur 2 (13C) CDCl3 300MHz
ppm
150,537
142,611
135,412
132,227
131,747
130,394
128,955
126,657
126,584
125,333
123,123
122,541
120,083
109,539
30,641
F3C
N
N
Me
250
200
150
100
50
0
%
ppm
200
180
160
140
120
100
80
60
40
20
0

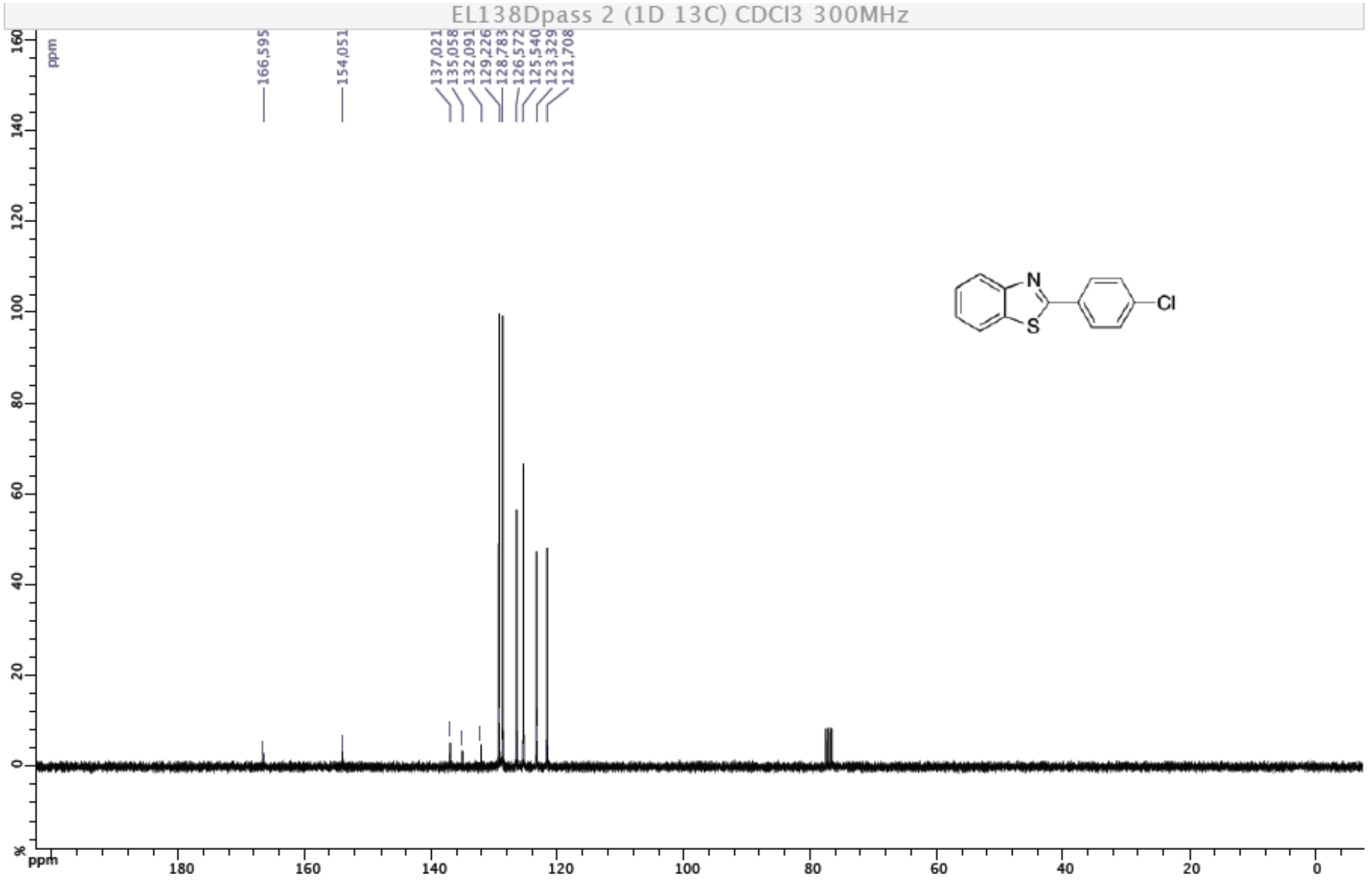
2-Phenylbenzo[d]thiazole (5a)



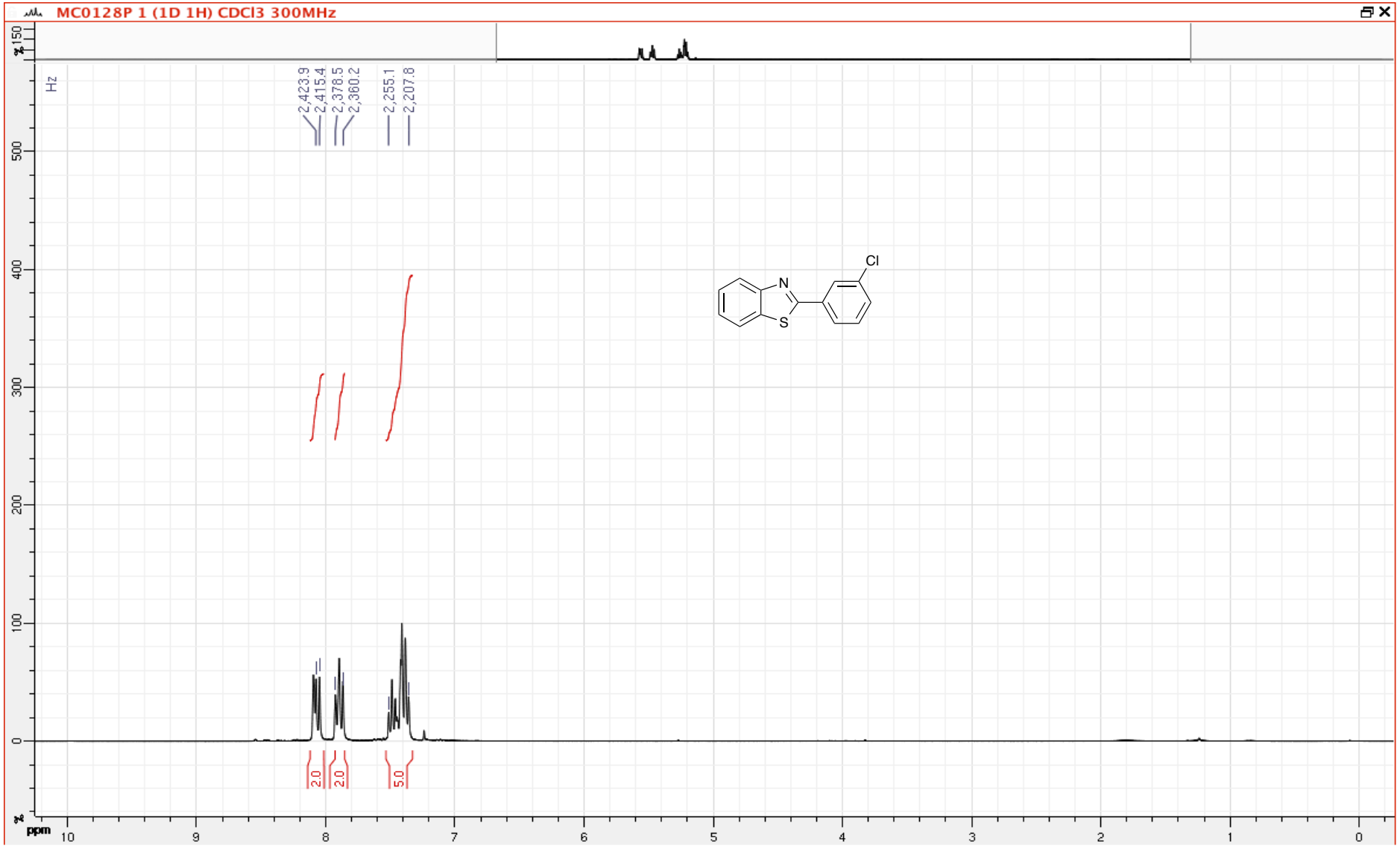

B3083-13C (1D 13C) CDCl3 300MHz
ppm
168.071
154.146
135.073
133.633
130.979
129.030
128.979
127.576
126.325
125.198
123.249
121.628
N
S
ppm
200
180
160
140
120
100
80
60
40
20
0

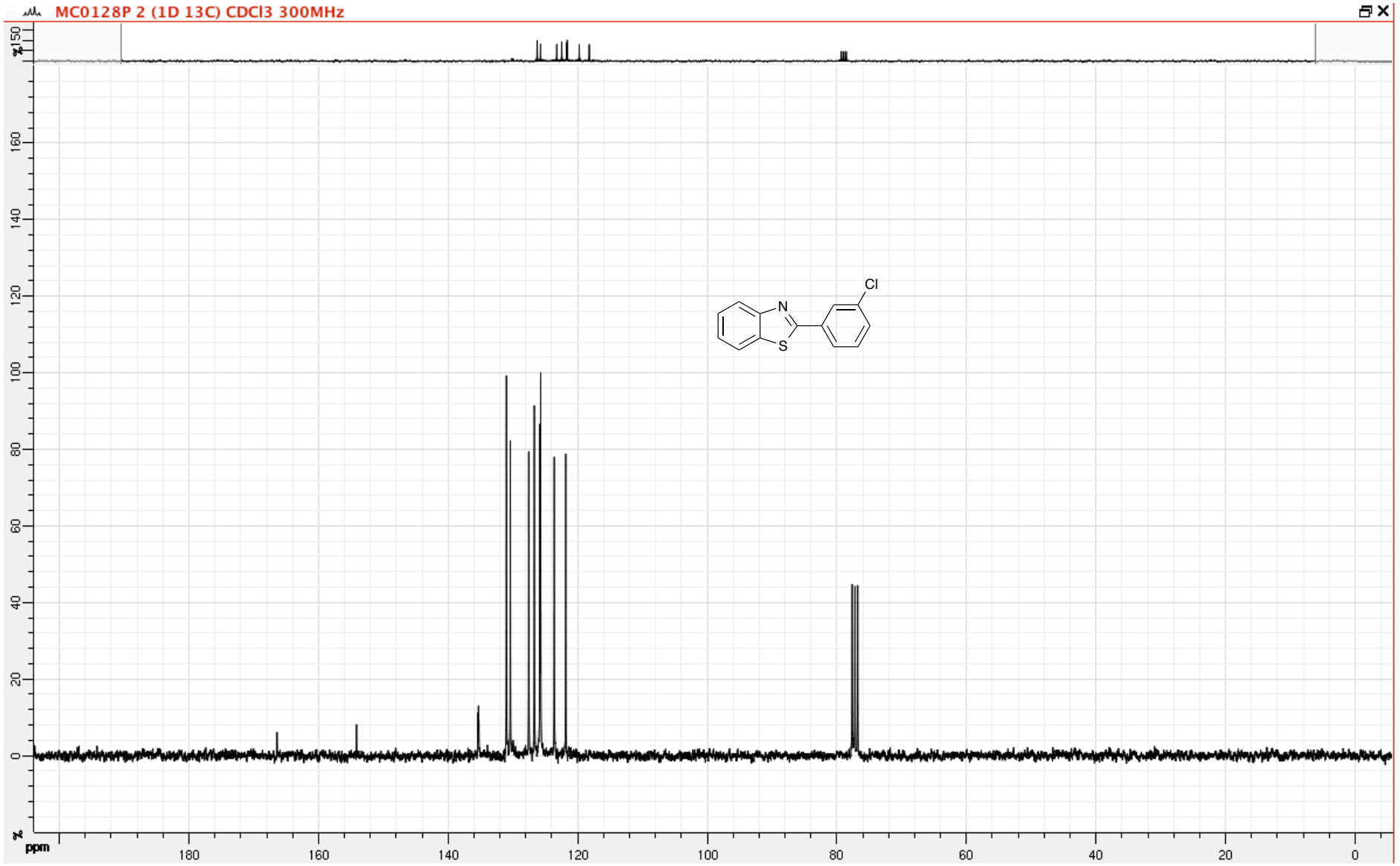
2-(4-Chlorophenyl)benzo[*d*]thiazole (5b)



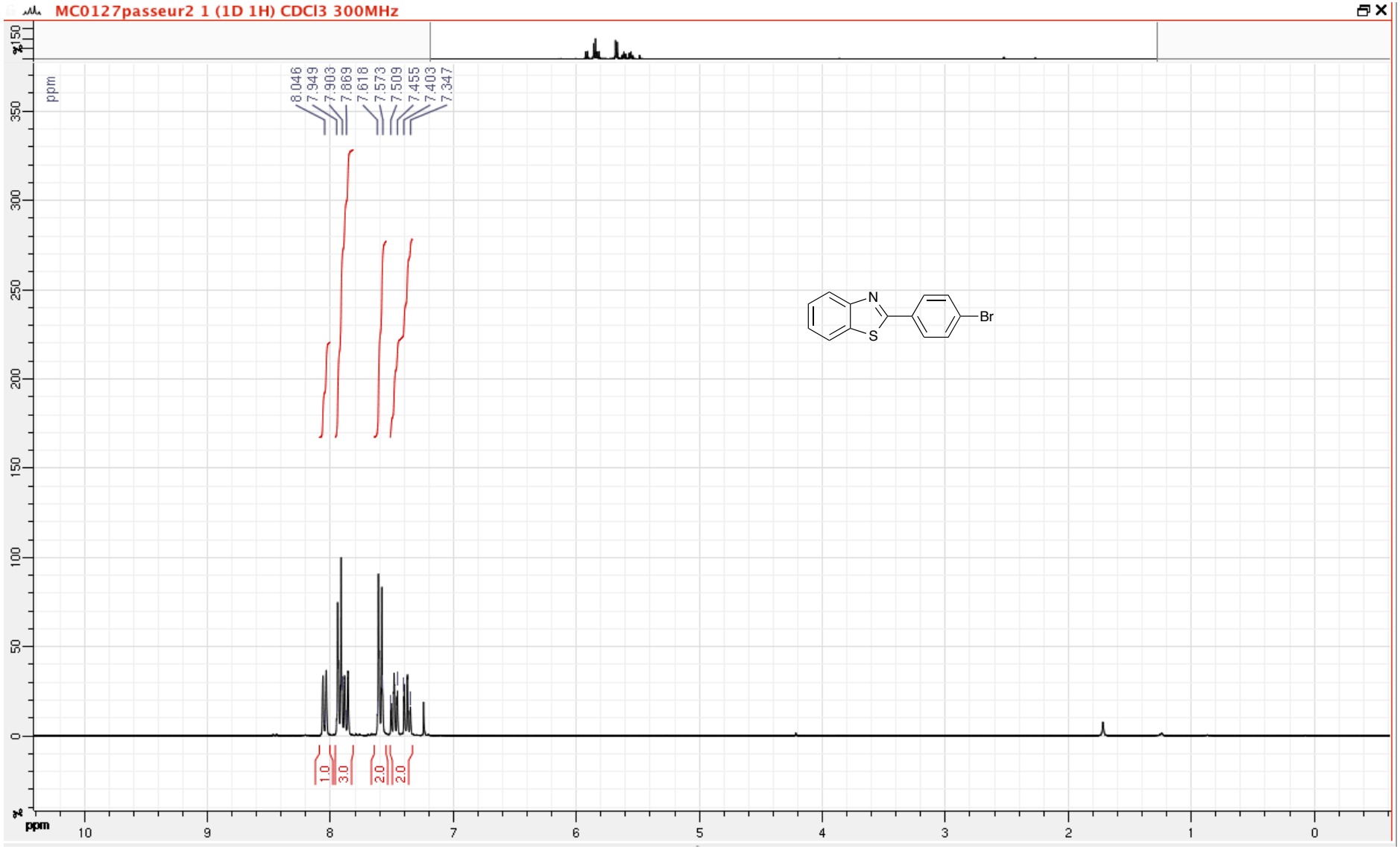

EL138Dpass 2 (1D 13C) CDCl3 300MHz
ppm
166,595
154,051
137,021
135,058
132,091
129,226
128,783
126,572
125,540
123,329
121,708
N
S
Cl
%
ppm
180
160
140
120
100
80
60
40
20
0

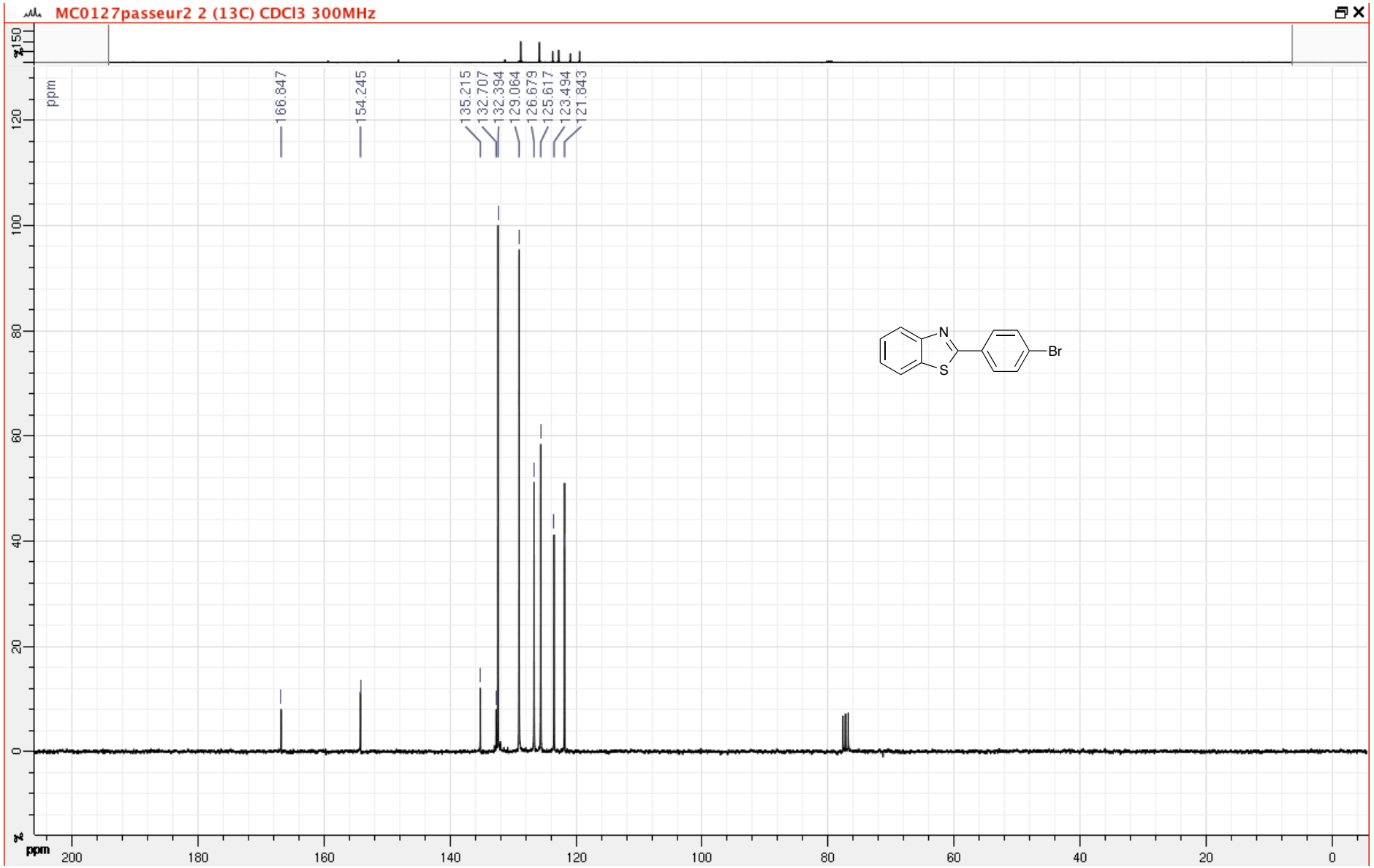
2-(3-Chlorophenyl)benzo[d]thiazole (5c)



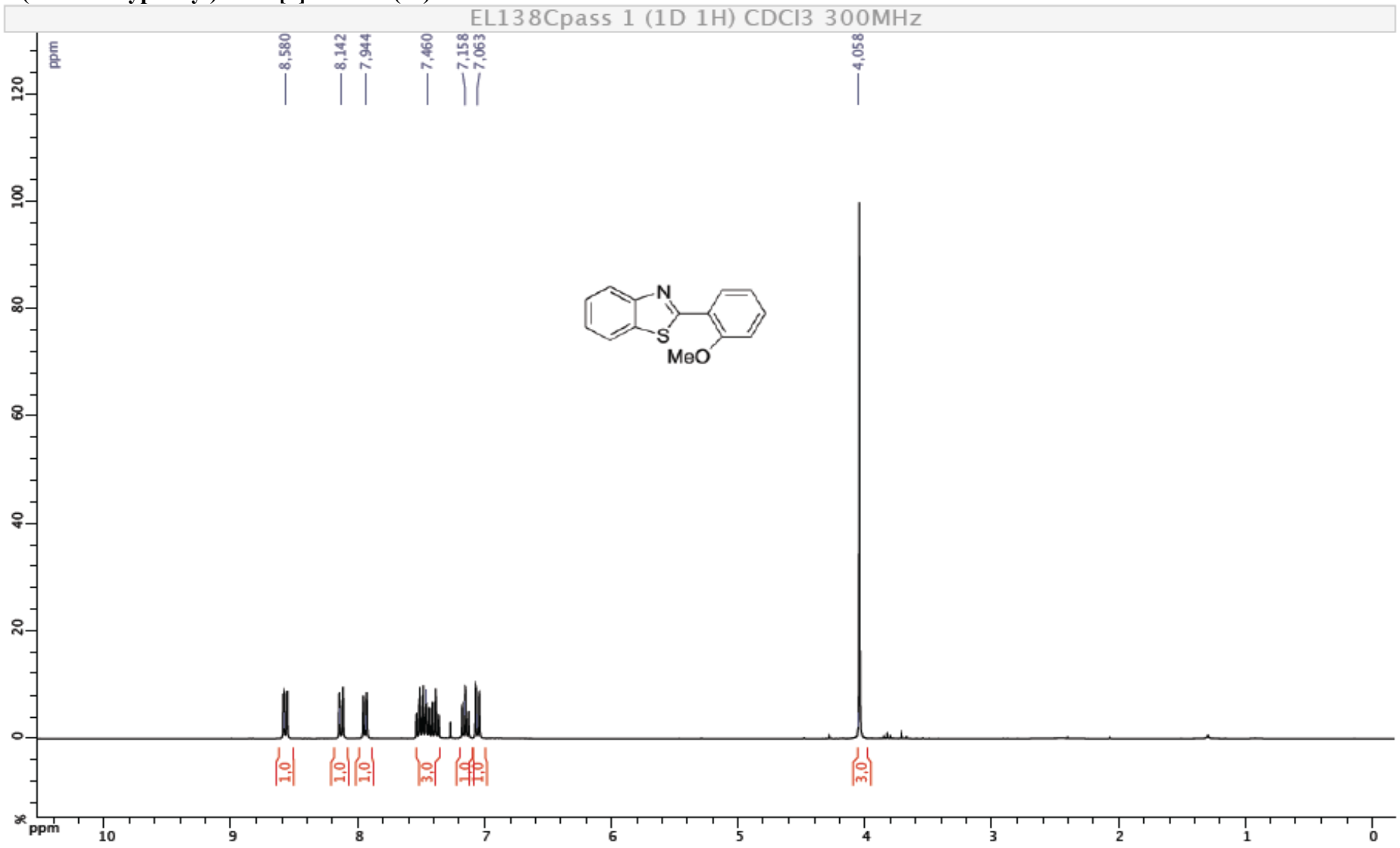

MC0128P 2 (1D 13C) CDCl3 300MHz
Cl
N
S
ppm
180
160
140
120
100
80
60
40
20
0

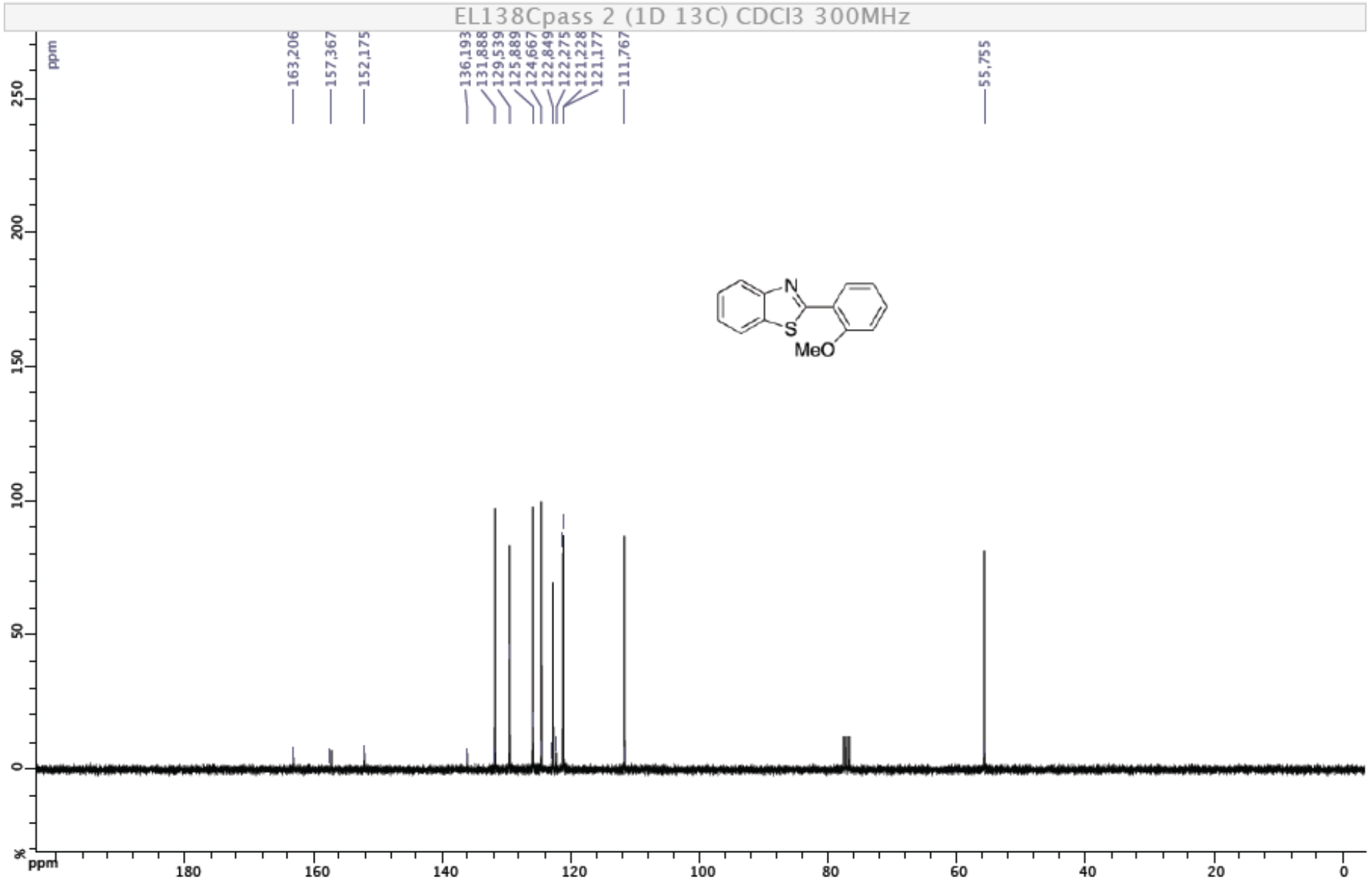
2-(4-Bromophenyl)benzo[*d*]thiazole (5d)



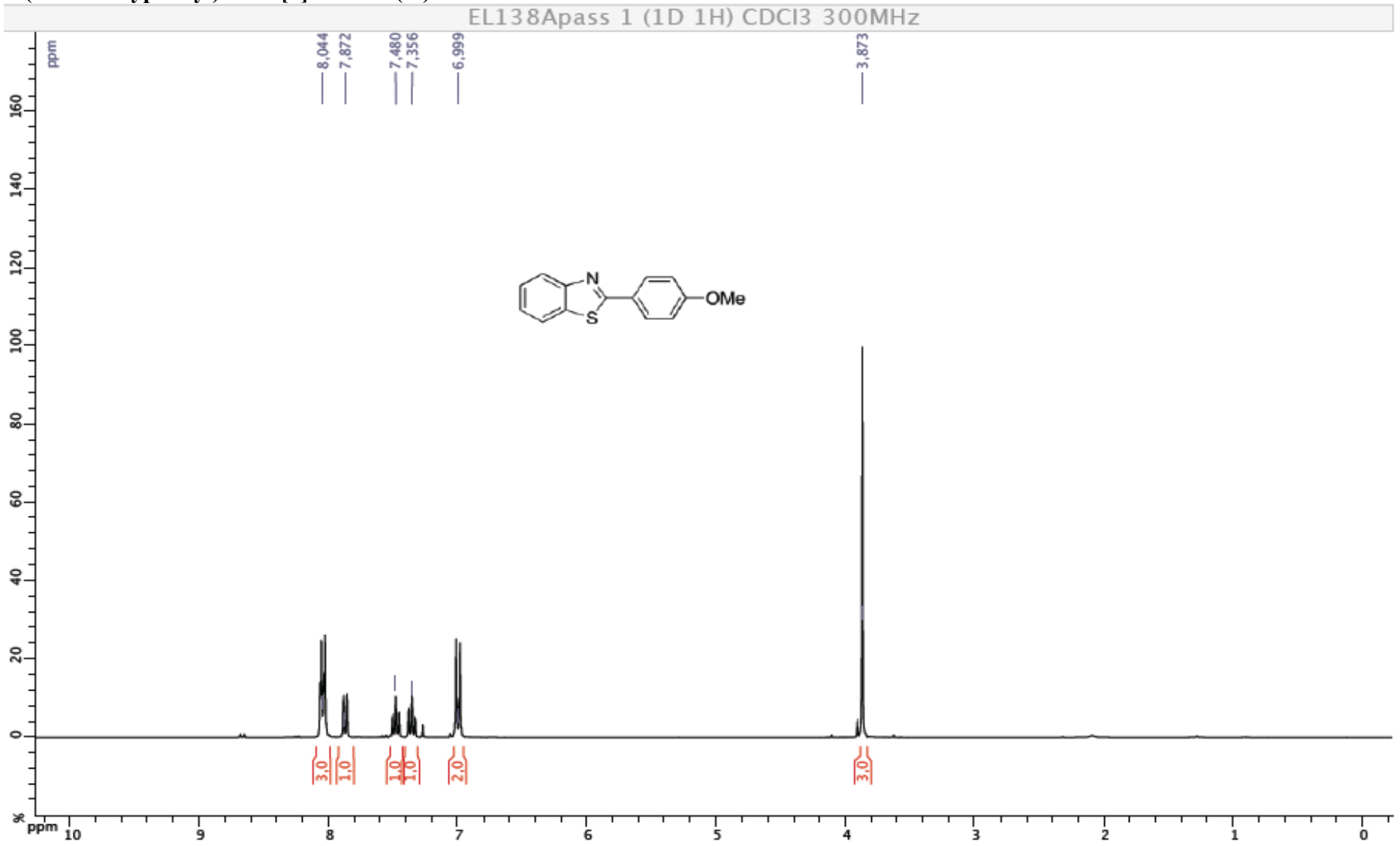

MC0127passeur2 2 (13C) CDCl3 300MHz
ppm
166.847
154.245
135.215
132.707
132.394
129.064
126.679
125.617
123.494
121.843
N
S
Br

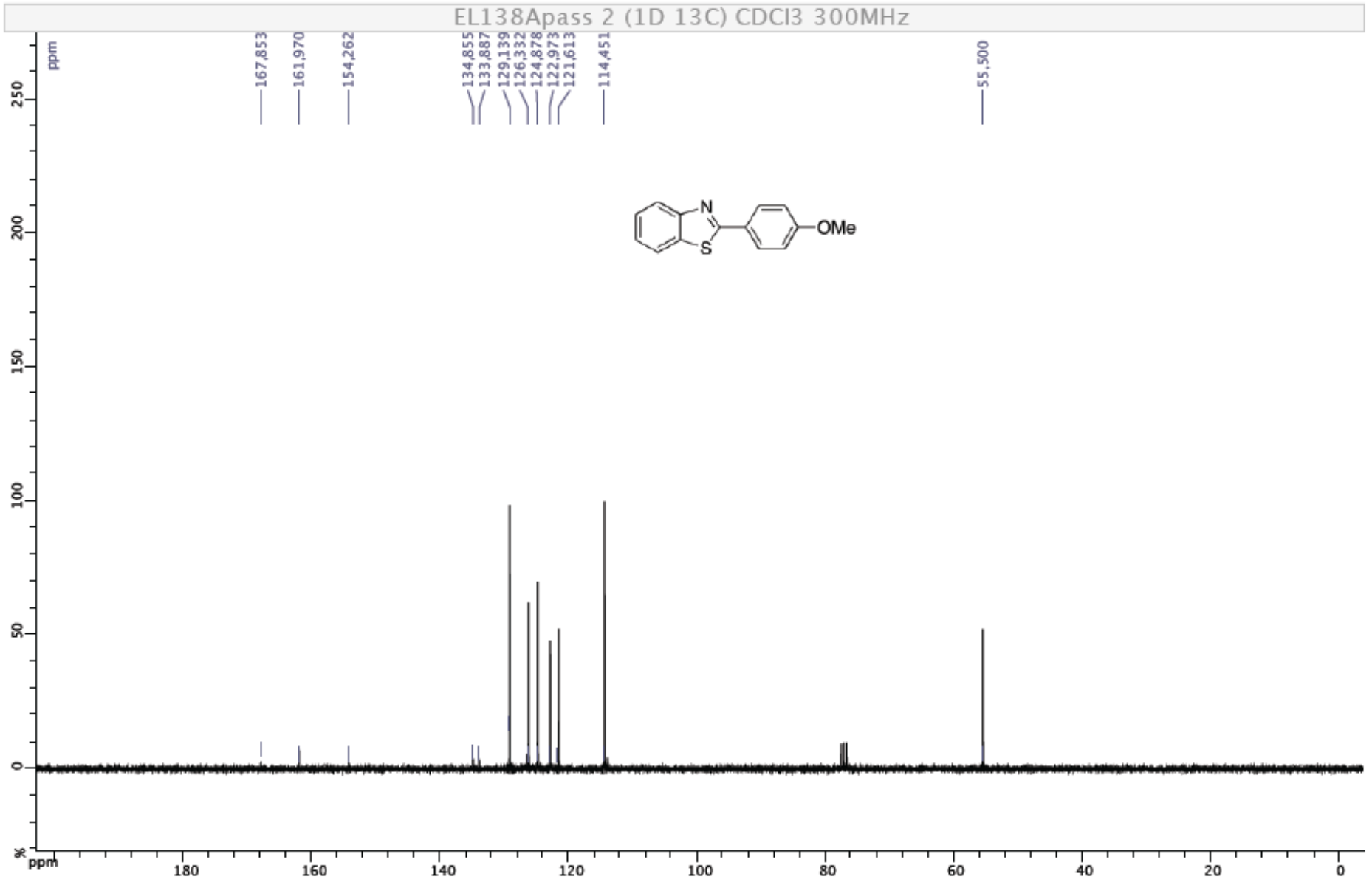
2-(2-Methoxyphenyl)benzo[*d*]thiazole (5e)



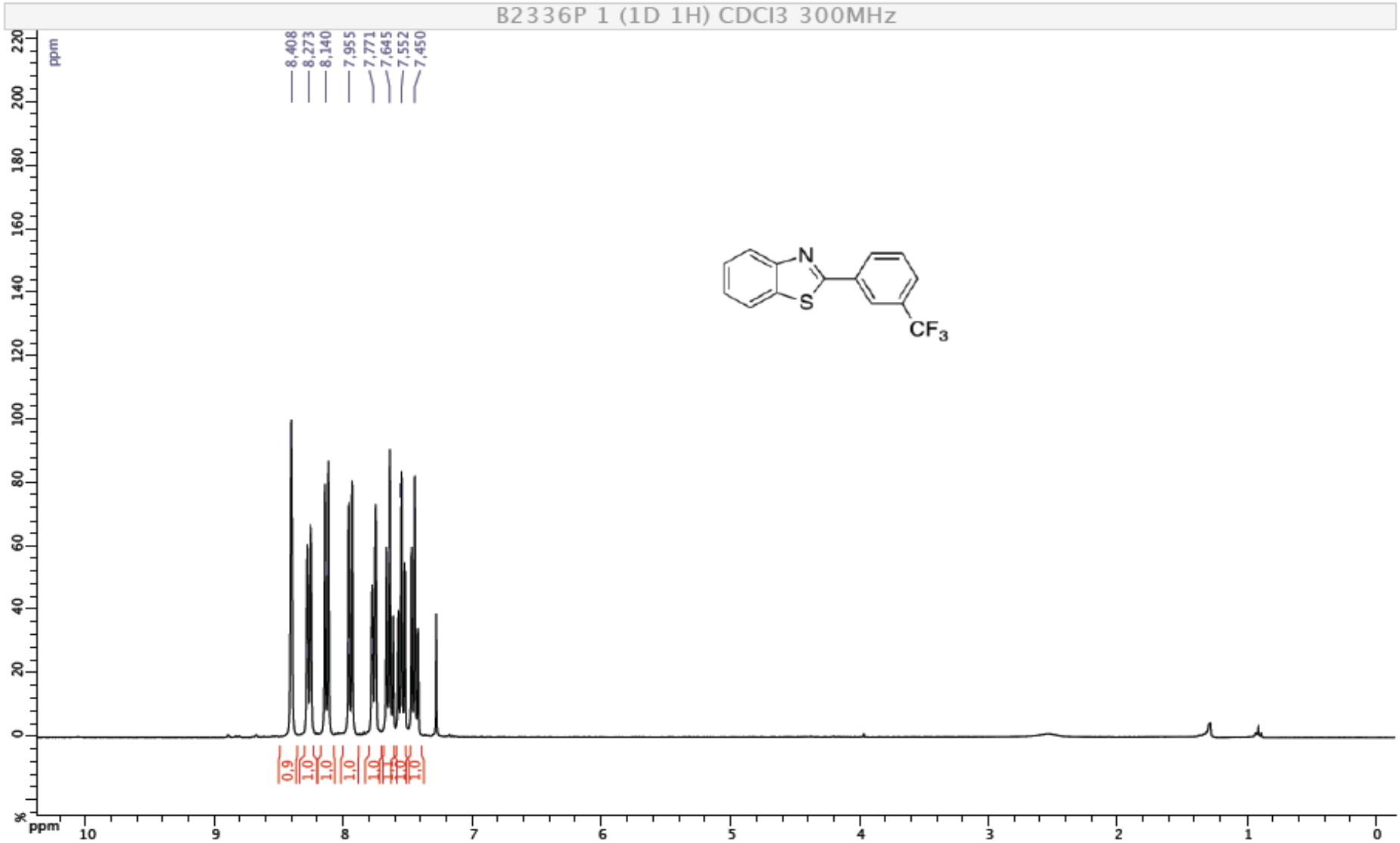

EL138Cpass 2 (1D 13C) CDCl3 300MHz
ppm
163,206
157,367
152,175
136,193
131,888
129,539
125,889
124,667
122,849
122,275
121,228
121,177
111,767
55,755
N
S
MeO
250
200
150
100
50
0
%
ppm
180
160
140
120
100
80
60
40
20
0

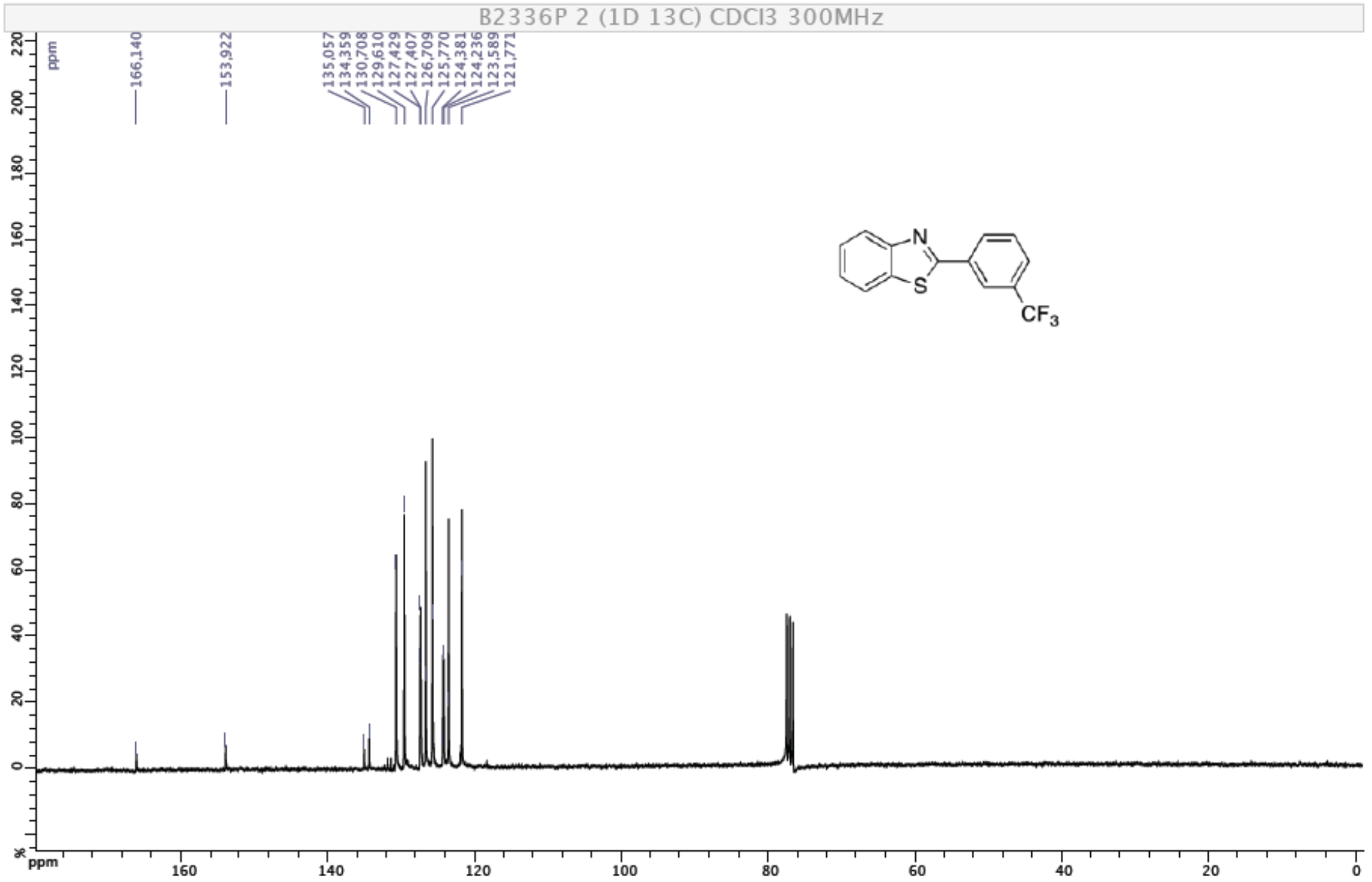
2-(4-Methoxyphenyl)benzo[*d*]thiazole (5f)



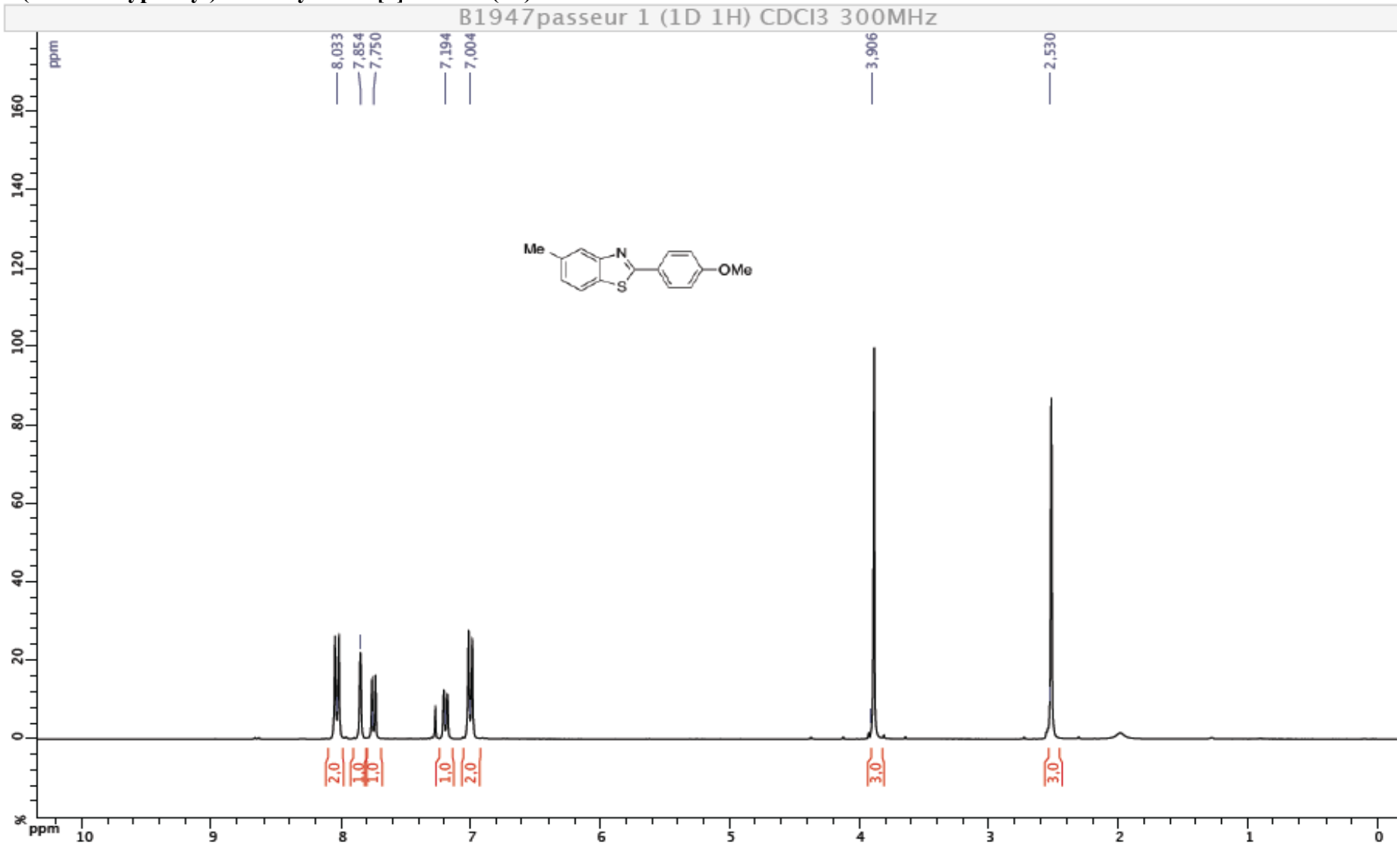

EL138Apass 2 (1D 13C) CDCl3 300MHz
ppm
167,853
161,970
154,262
134,855
133,887
129,139
126,332
124,878
122,973
121,613
114,451
55,500
N
S
OMe
250
200
150
100
50
0
%
ppm
180
160
140
120
100
80
60
40
20
0

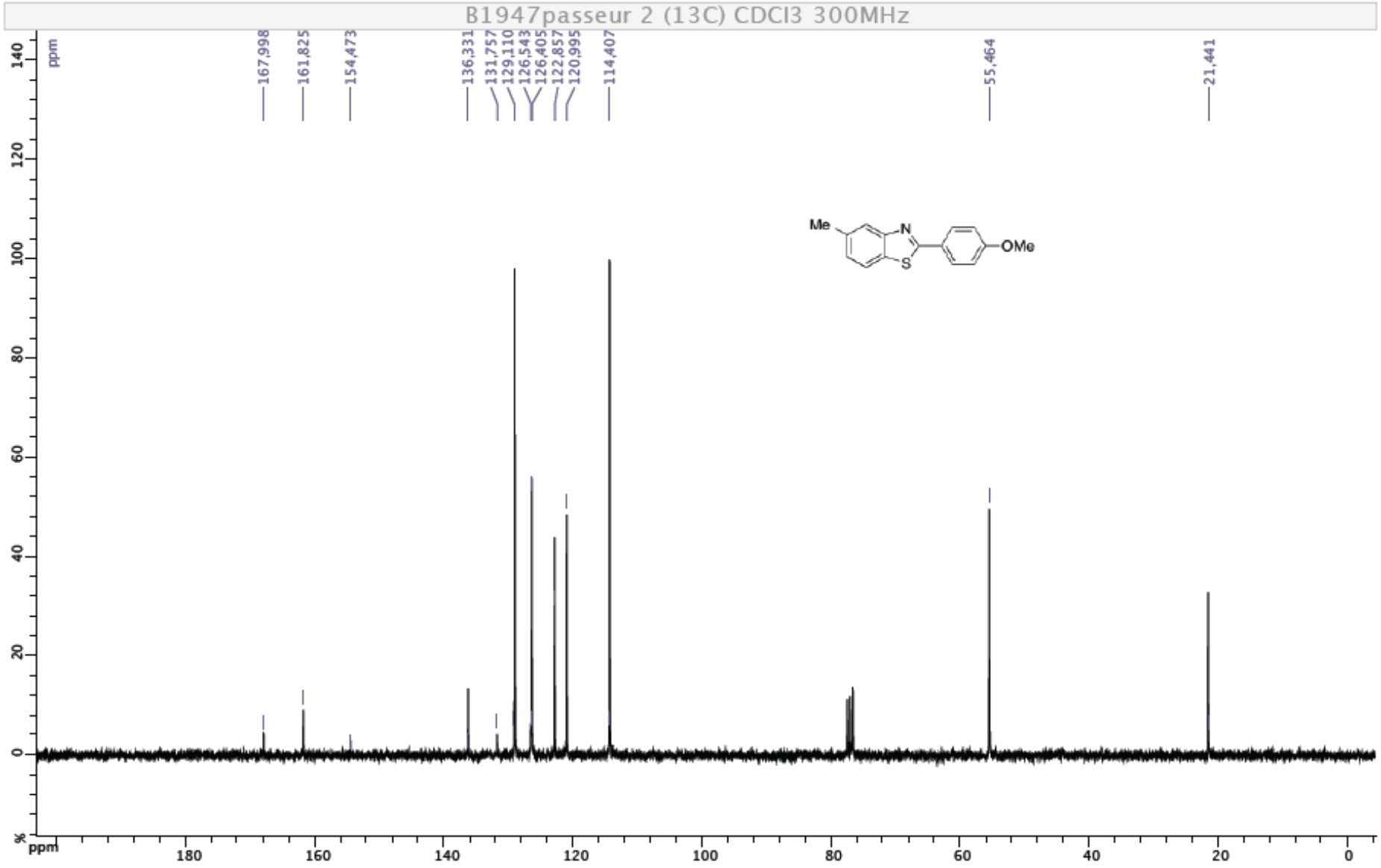
2-(3-(Trifluoromethyl)phenyl)benzo[*d*]thiazole (5g)



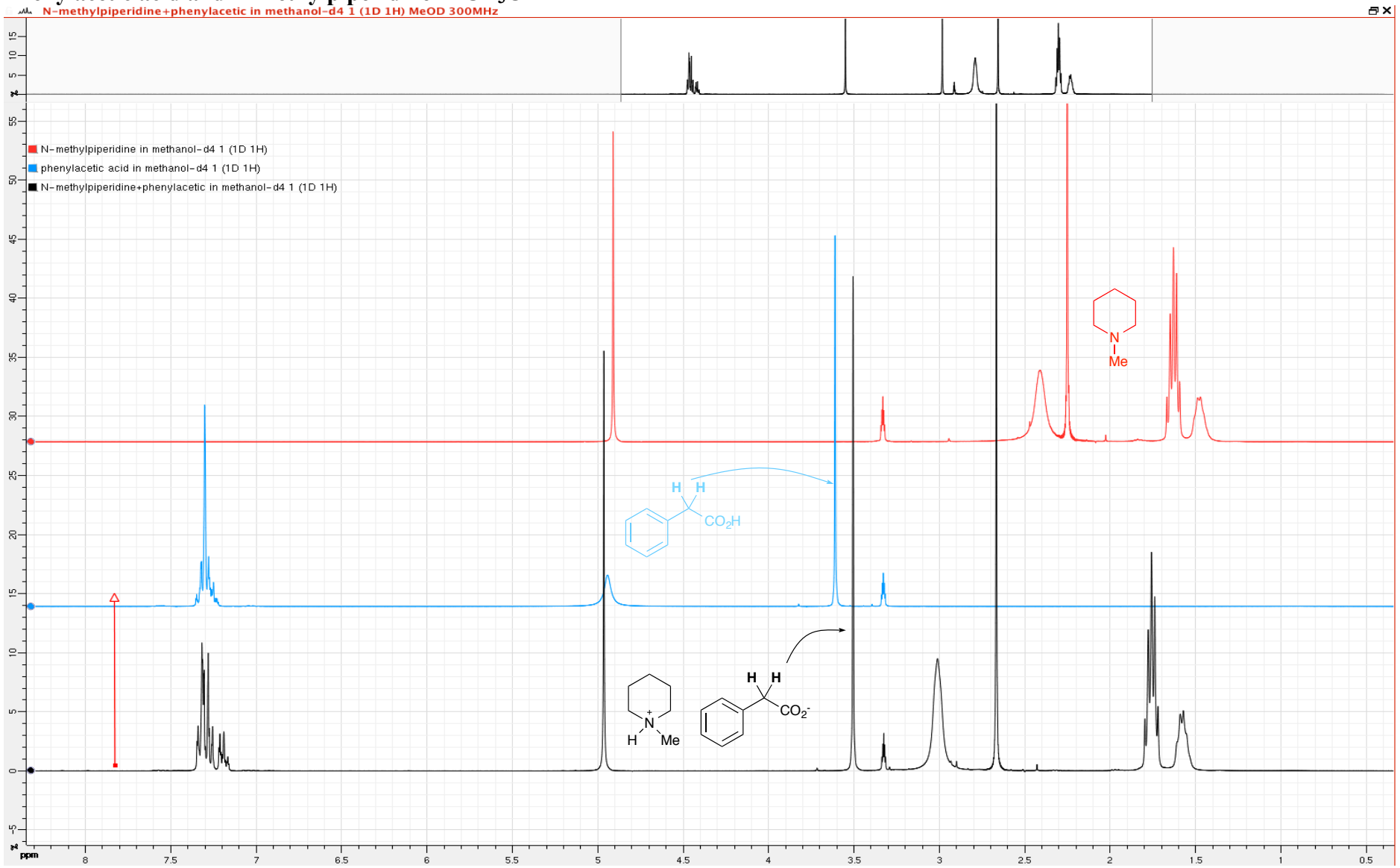

B2336P 2 (1D 13C) CDCl3 300MHz
ppm
166,140
153,922
135,057
134,359
130,708
129,610
127,429
127,407
126,709
125,770
124,381
124,236
123,589
121,771
N
S
CF3
220
200
180
160
140
120
100
80
60
40
20
0
%
ppm
160
140
120
100
80
60
40
20
0

2-(4-Methoxyphenyl)-5-methylbenzo[*d*]thiazole (5h)




B1947passeur 2 (13C) CDCl3 300MHz
ppm
167,998
161,825
154,473
136,331
131,757
129,110
126,543
126,405
122,857
120,995
114,407
55,464
21,441
Me
N
S
OMe
%
ppm
180
160
140
120
100
80
60
40
20
0

Phenylacetic acid and *N*-methylpiperidine in CD_3OD



Phenylacetic acid and 3-picoline in CD_3OD