Supplementary NMR Appendix

Multisubstituted pyrimidines effectively inhibit bacterial growth and biofilm formation of *Staphylococcus aureus*

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[*]email: jari.yli-kauhaluoma@helsinki.fi

[⊥]These authors contributed equally to this work.

NMR Spectra

Peak reports and multiplet assignments are provided for all compounds. Compound peaks are highlighted in **green** and solvent peaks in **yellow**. All 2D-NMR spectra are edited to highlight the correlation peaks. ^{13}C-HSQC spectra include DEPT-135 information with –CH– and $–CH_3$ correlation peaks in positive phase (**red**) while $–CH_2–$ correlation peaks in negative phase (**blue**). For most of the compounds,^{15}N-HMBC spectrum and corresponding ^{15}N 1D projection are also reported.

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Compound **4c** 011

Compound **4d** 017

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Compound **5c** 043

Compound **5d** 049

Compound **5e** 055

Compound **6b** 061

Compound **6c** 067

Compound **6d** 073

Compound **6e** 079

Compound **7a** 085

Compound **7b** 091

Compound **7c** 097

Compound **7d** 103

Compound **7e** 109

Compound **7f** 115

Compound **8d** 123

Compound **9a** 130

Compound **9b** 134

Compound **9c** 140

Compound **9d** 144

Compound **9e** 150

Compound **10a** 156

Compound **10b** 160

Compound **10c** 166

Compound **10d** 172

Compound **10e** 178

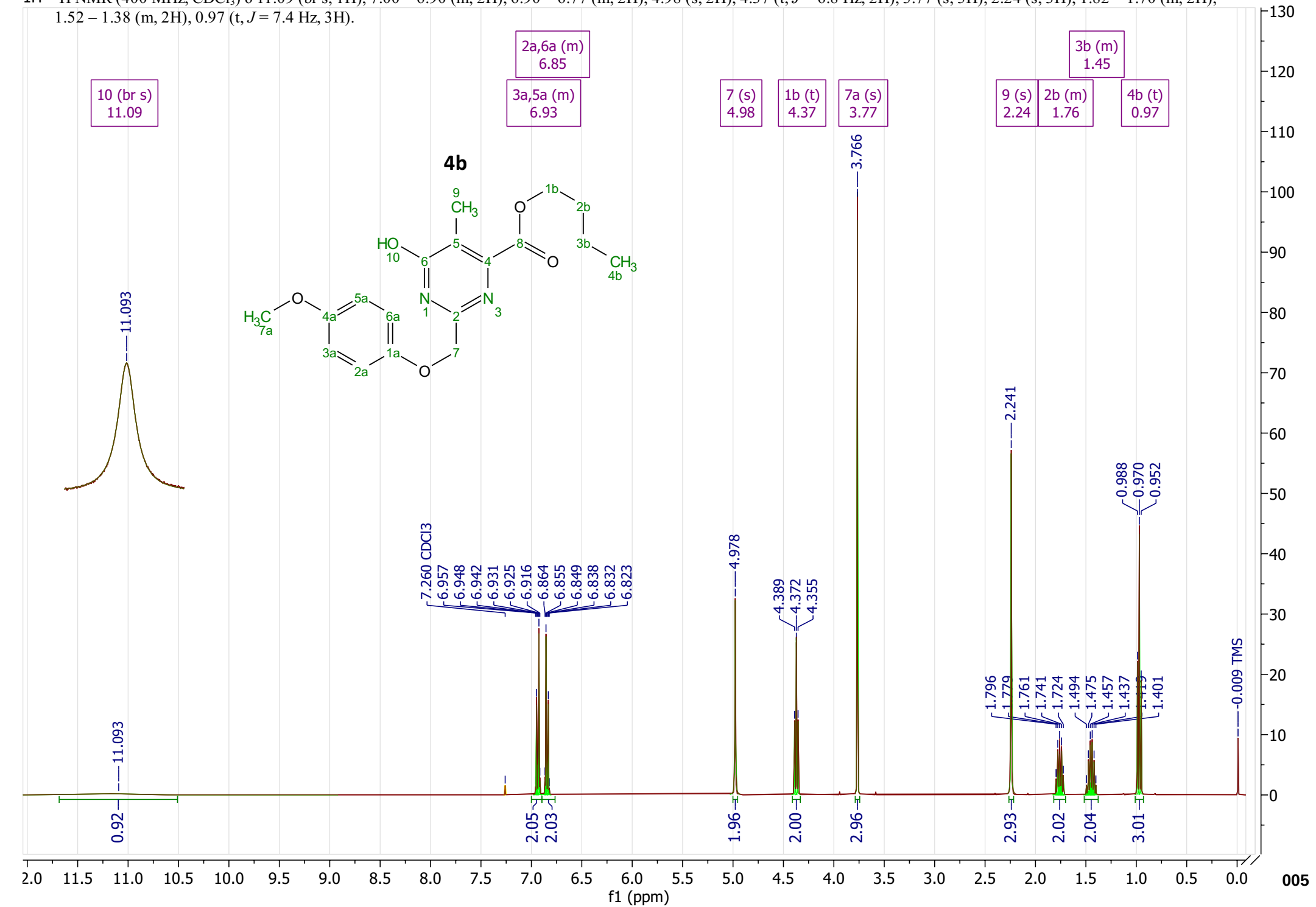
Compound **10f** 182

Compound **11d** 188

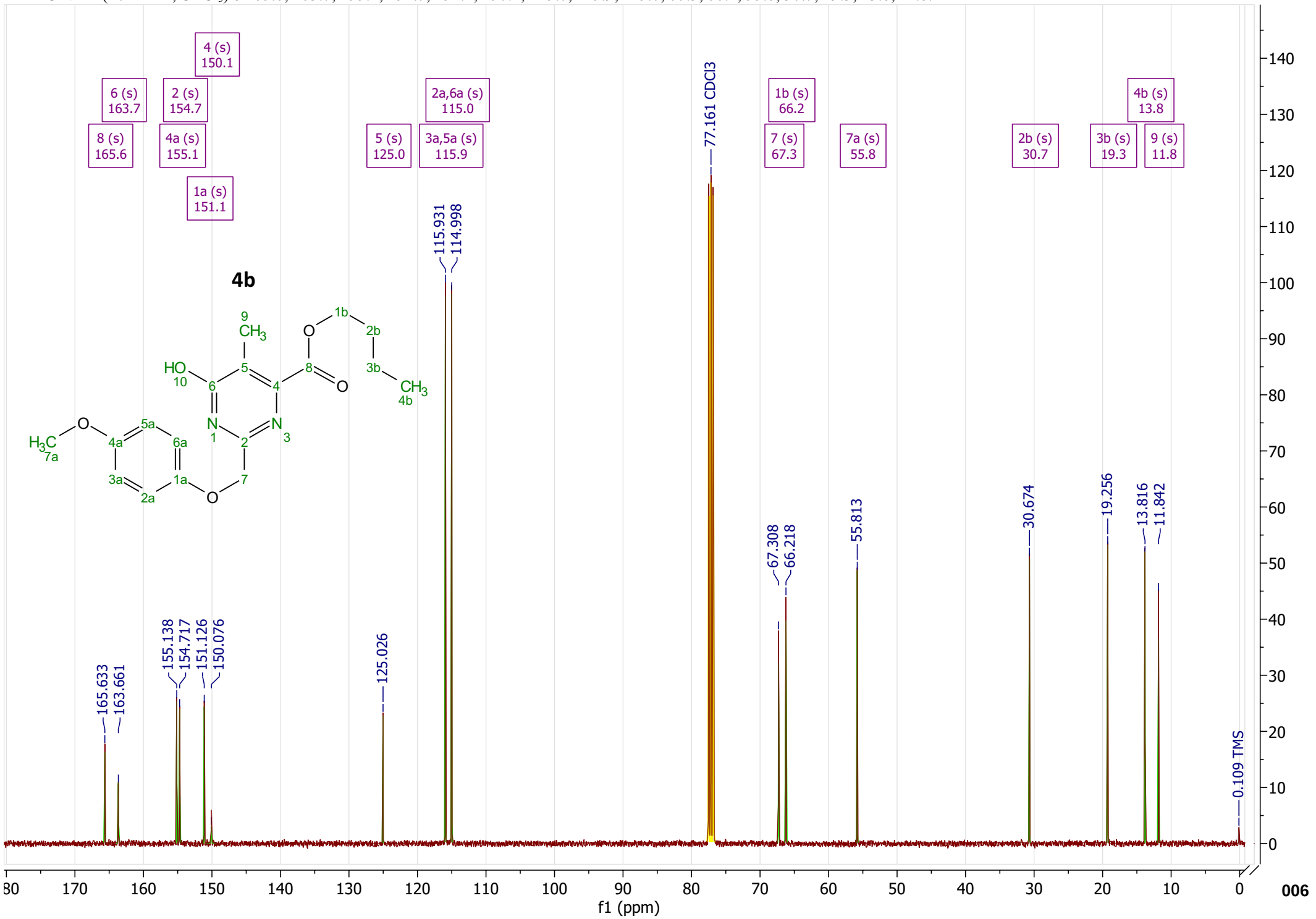
Compound **12b** 193

Compound **12d** 199

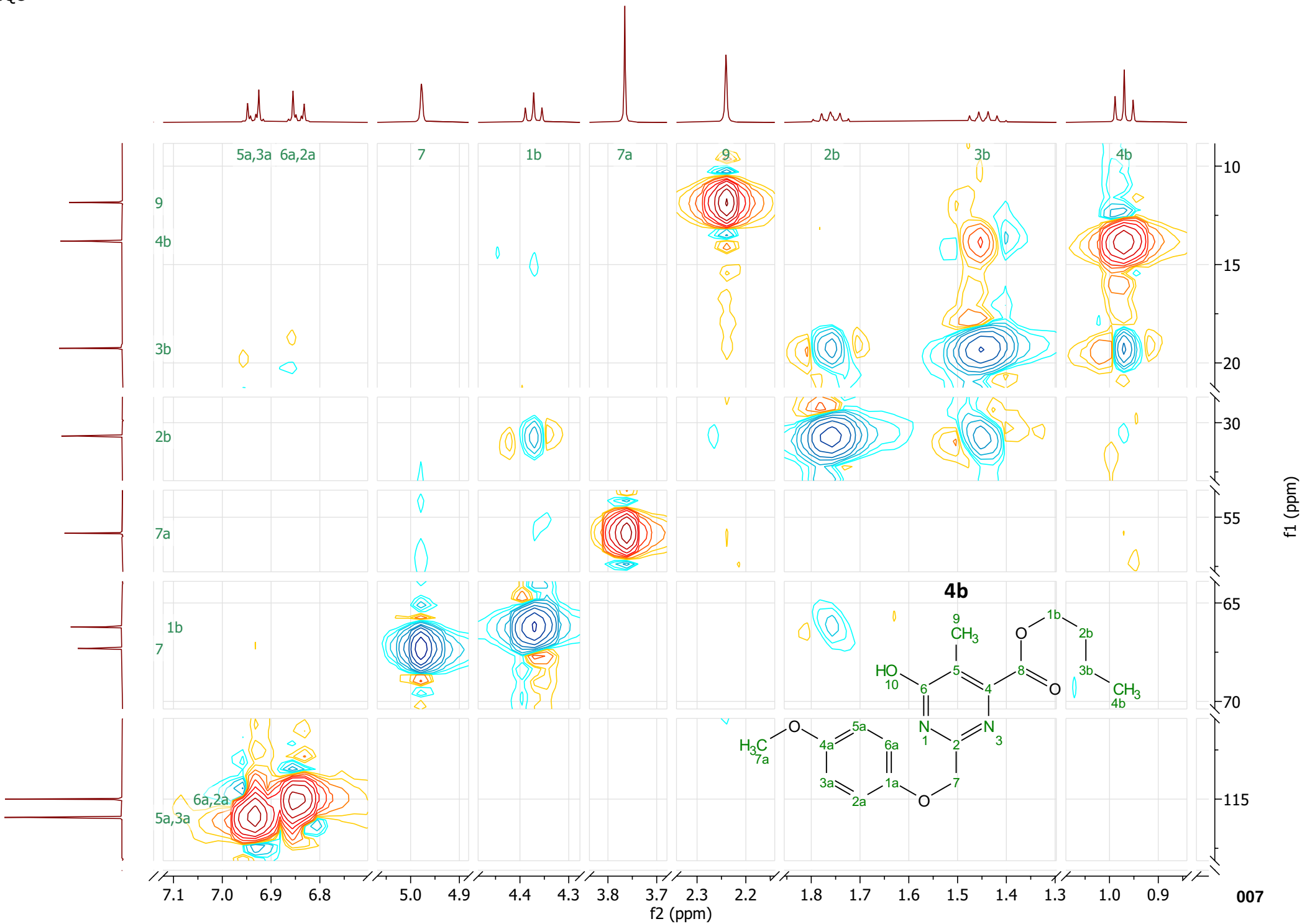
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.09 (br s, 1H), 7.00 – 6.90 (m, 2H), 6.90 – 6.77 (m, 2H), 4.98 (s, 2H), 4.37 (t, J = 6.8 Hz, 2H), 3.77 (s, 3H), 2.24 (s, 3H), 1.82 – 1.70 (m, 2H), 1.52 – 1.38 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H).



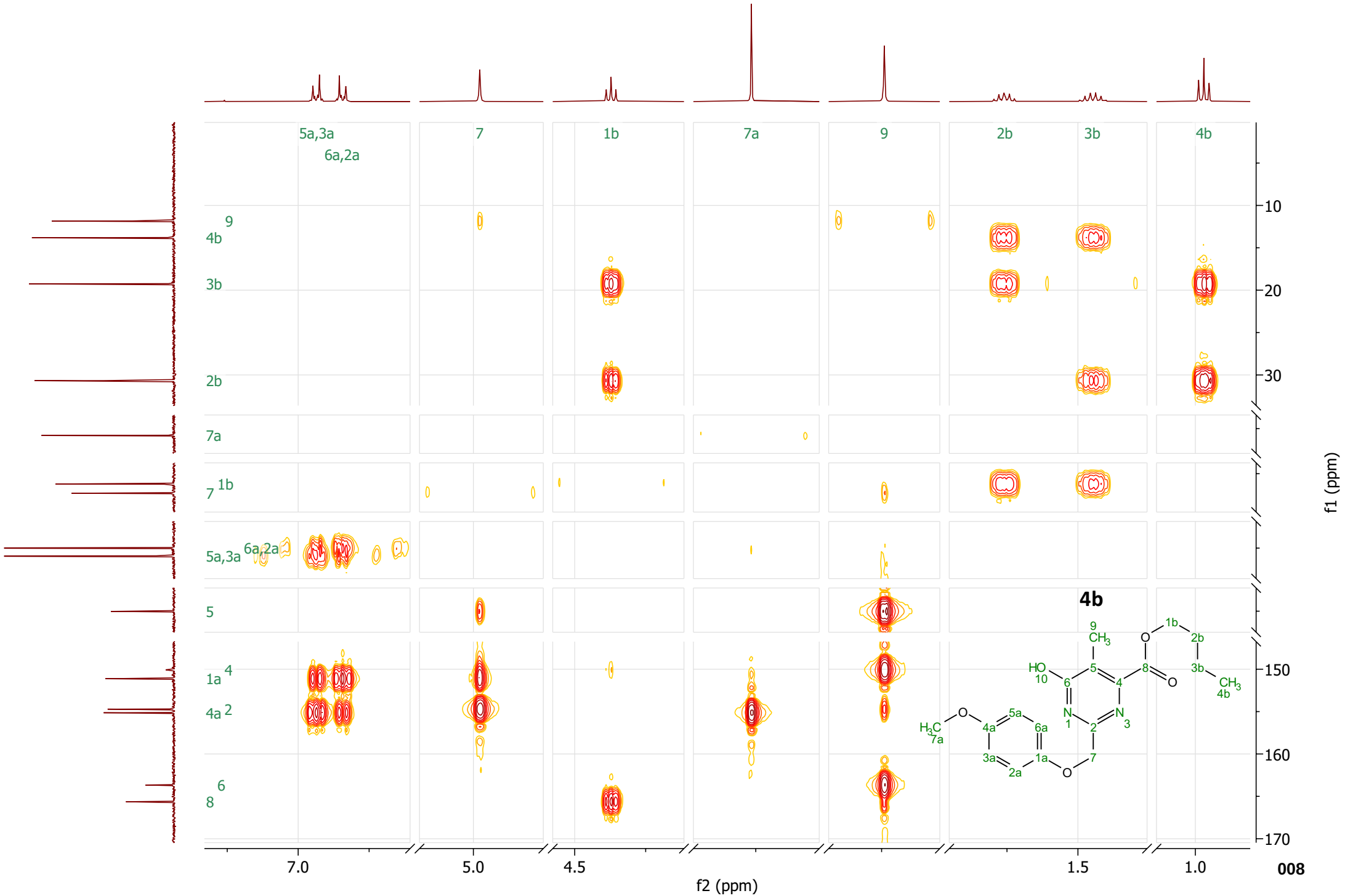
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 163.7, 155.1, 154.7, 151.1, 150.1, 125.0, 115.9, 115.0, 67.3, 66.2, 55.8, 30.7, 19.3, 13.8, 11.8.



13C HSQC



13C HMBC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6a,2a
7
1b
7a
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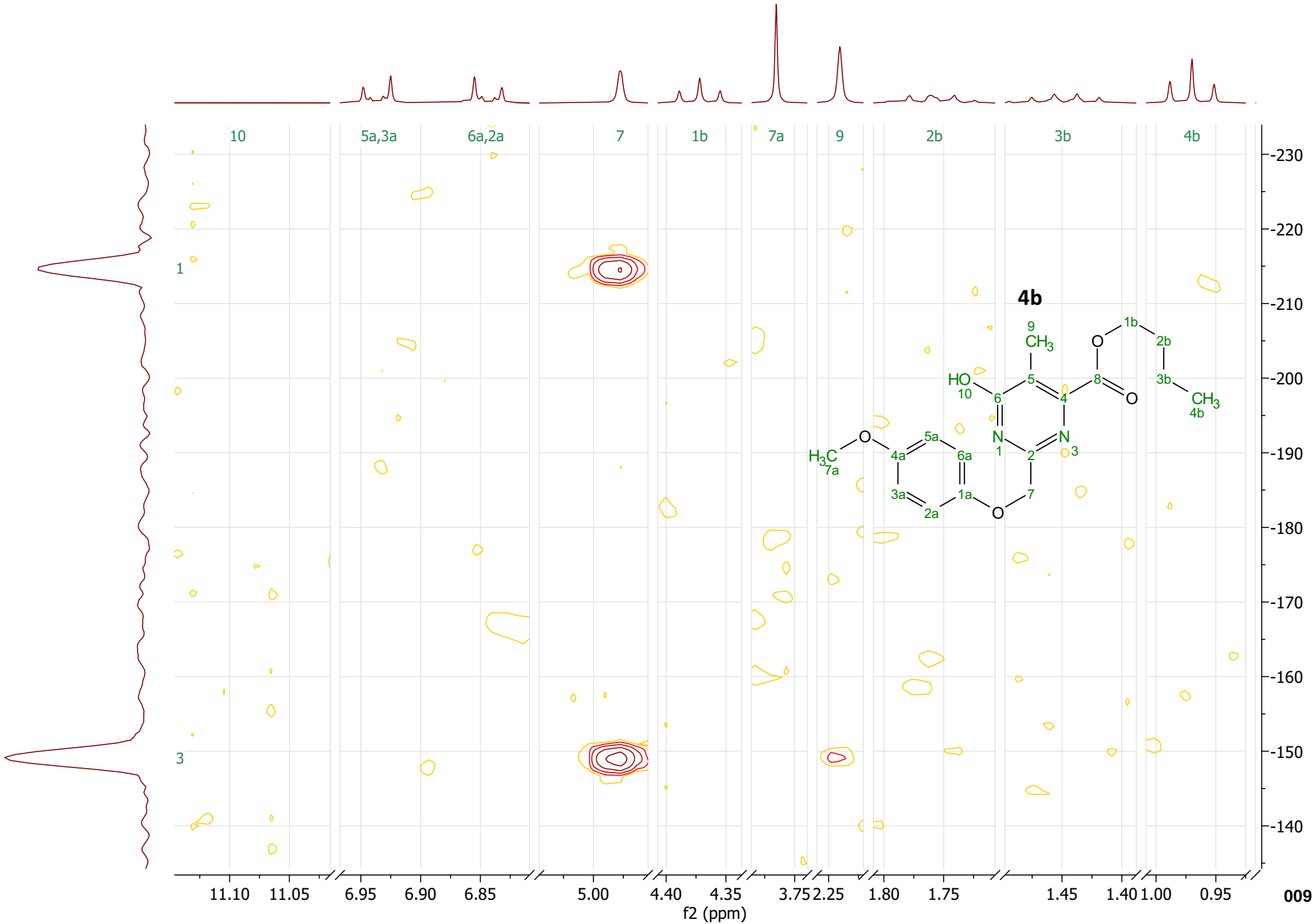
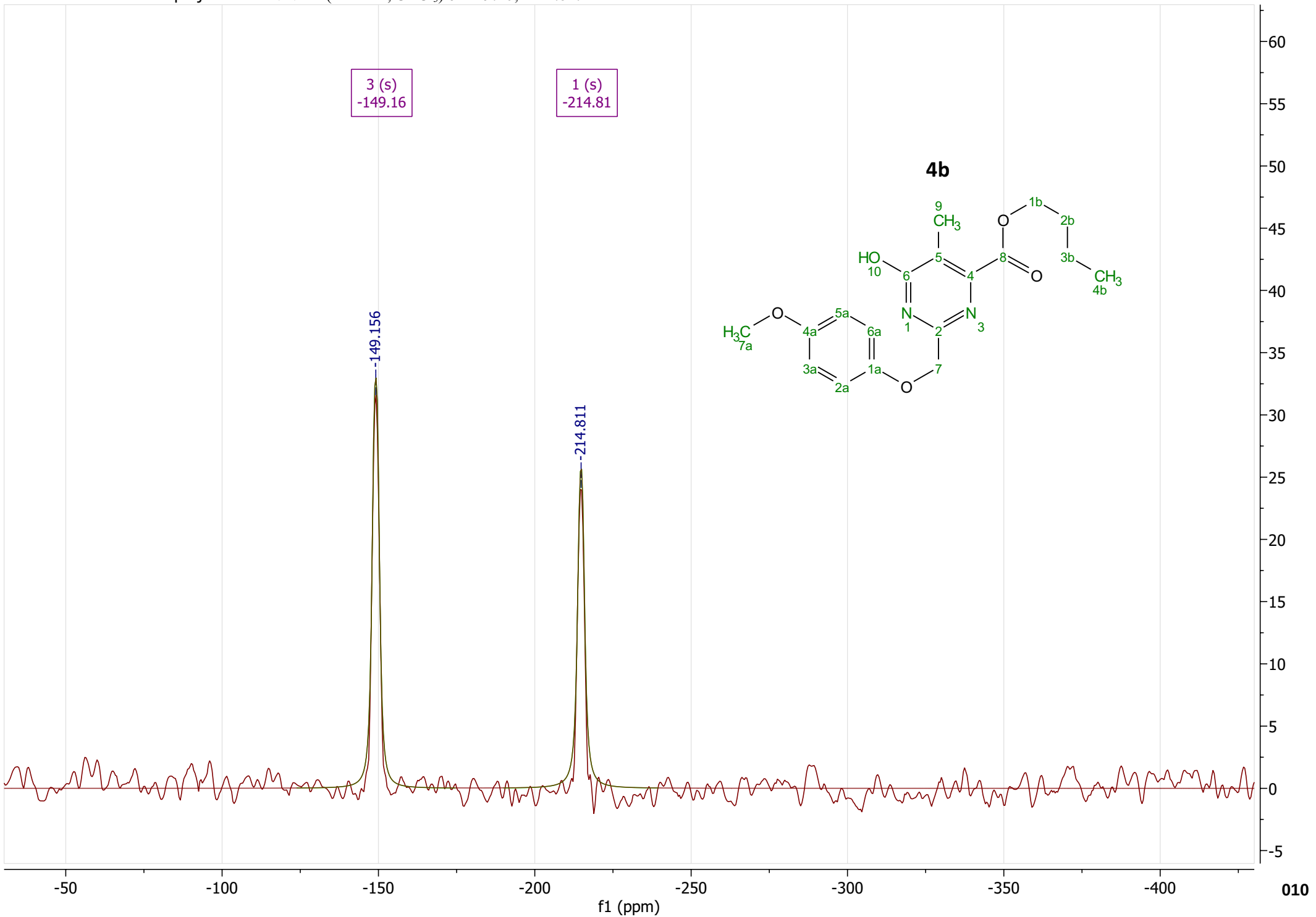
2b
3b
4b
9
4b
3b
2b
7a
7
1b
5a,3a
6a,2a
5
1a
4
4a
2
8
6
10
20
30
150
160
170
f1 (ppm)
7.0
5.0
4.5
1.5
1.0
f2 (ppm)
4b
008

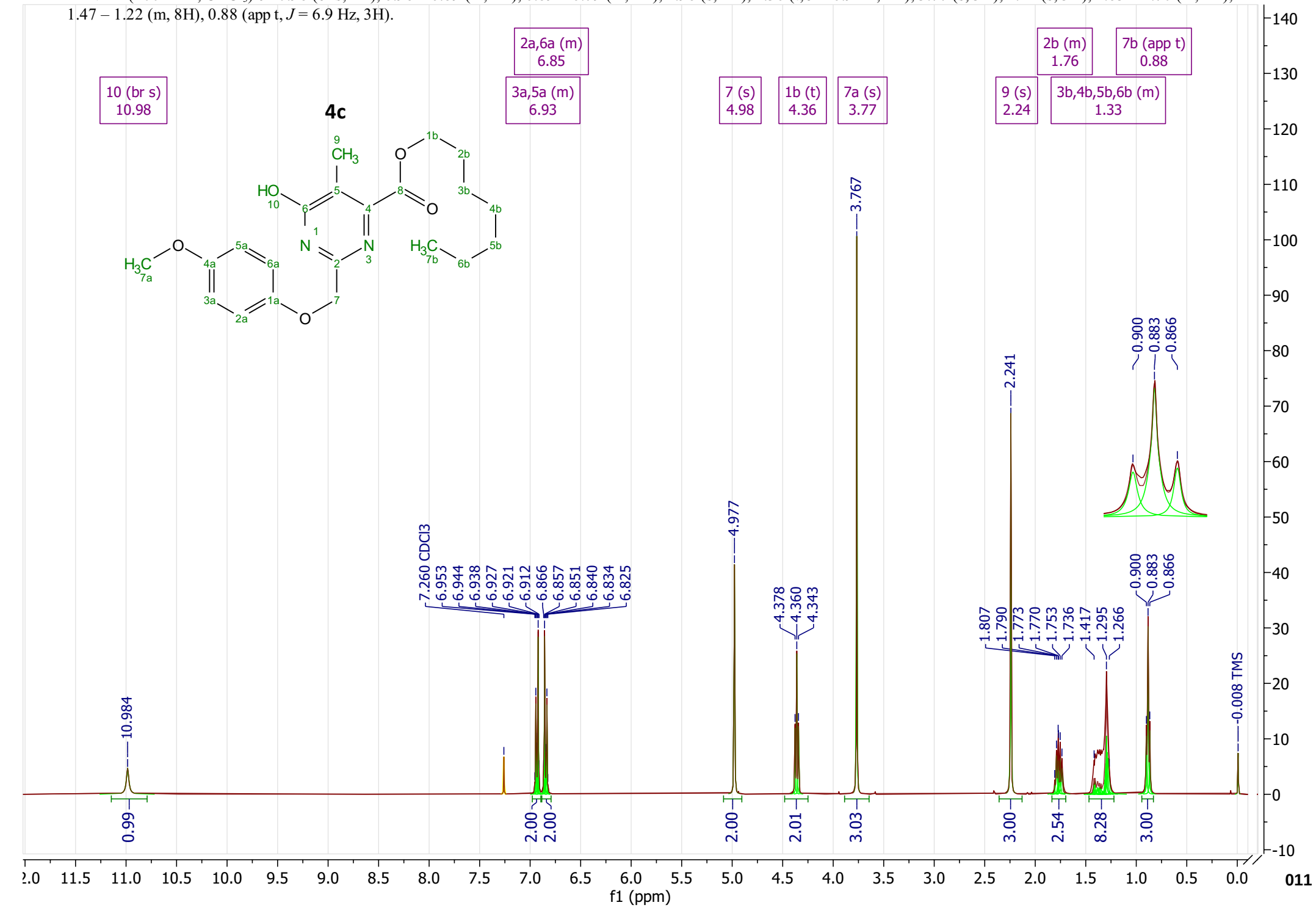
15N HMBC



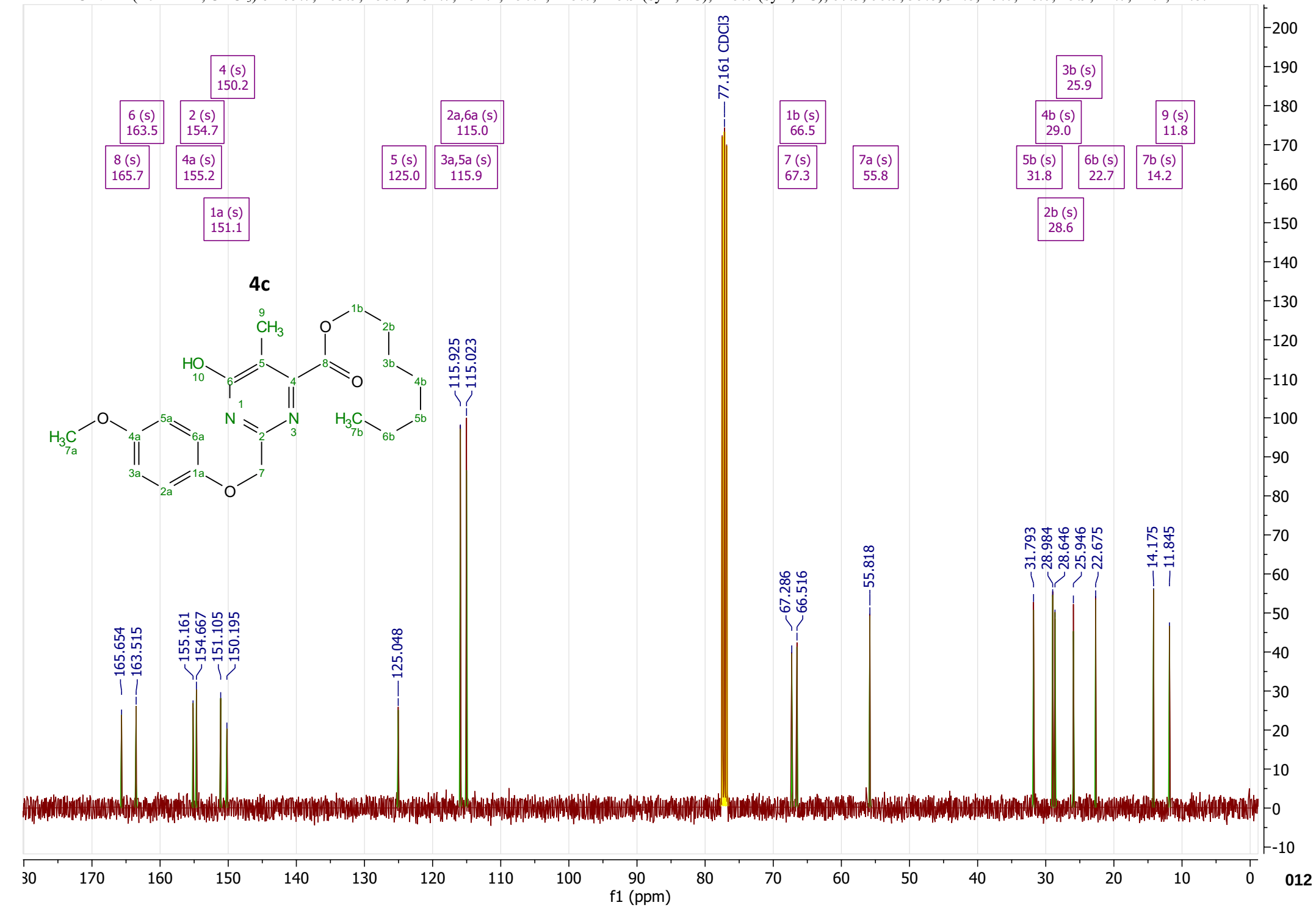
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -149.16, -214.81.



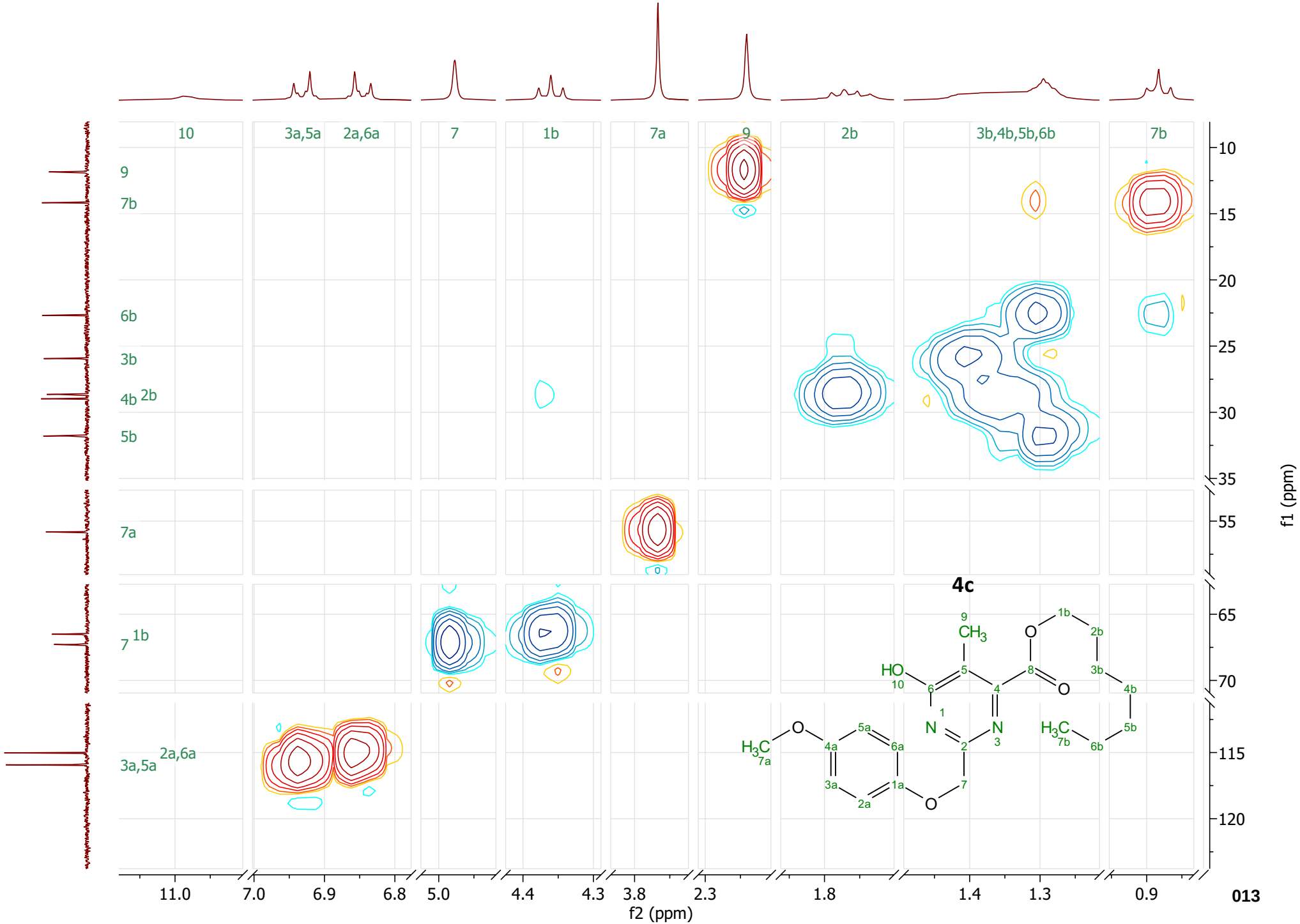
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.98 (br s, 1H), 6.98 – 6.89 (m, 2H), 6.89 – 6.79 (m, 2H), 4.98 (s, 2H), 4.36 (t, *J* = 6.9 Hz, 2H), 3.77 (s, 3H), 2.24 (s, 3H), 1.83 – 1.70 (m, 2H), 1.47 – 1.22 (m, 8H), 0.88 (app t, *J* = 6.9 Hz, 3H).



13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 163.5, 155.2, 154.7, 151.1, 150.2, 125.0, 115.9 (sym, 2C), 115.0 (sym, 2C), 67.3, 66.5, 55.8, 31.8, 29.0, 28.6, 25.9, 22.7, 14.2, 11.8.



13C HSQC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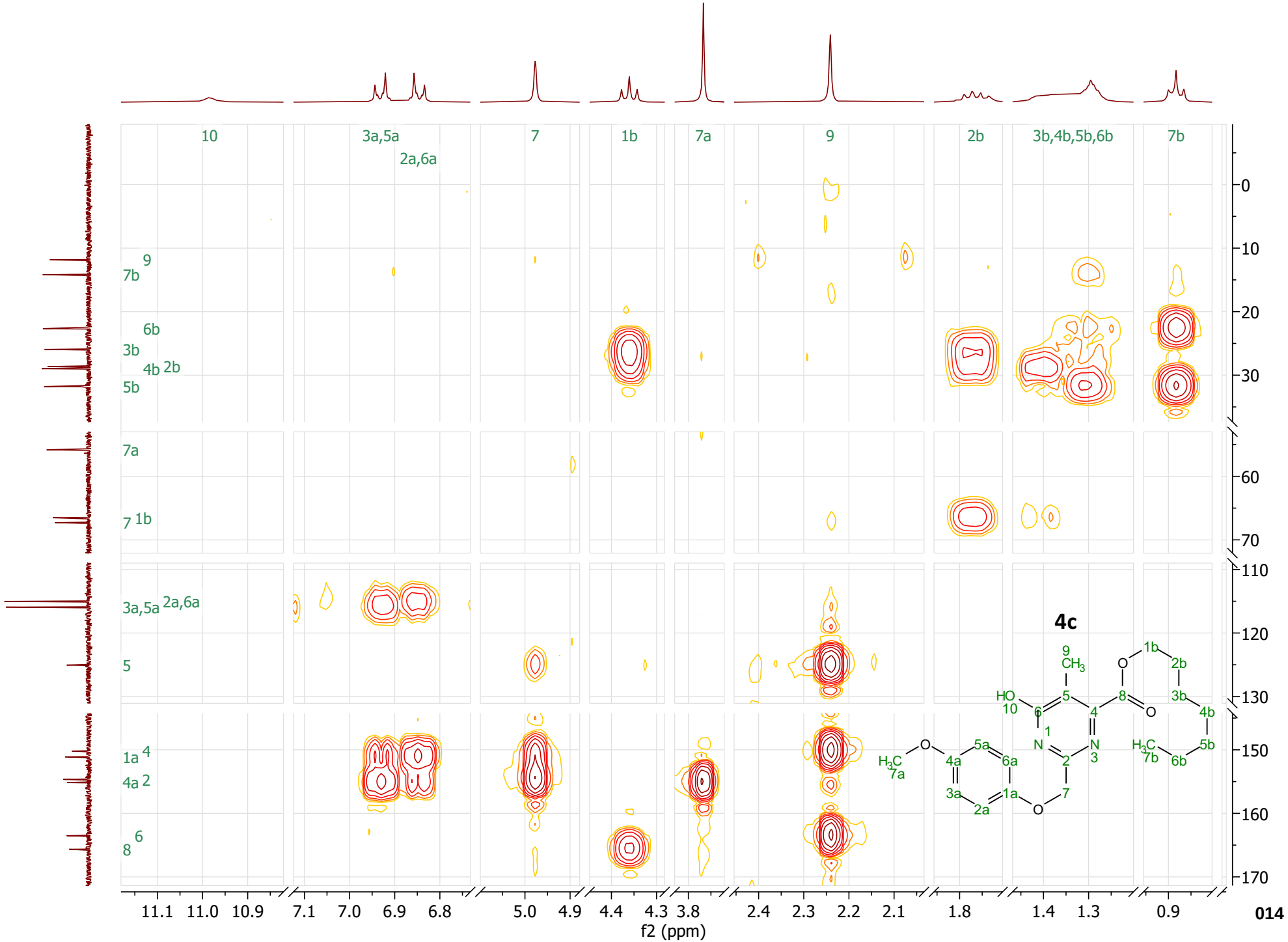
3a,5a
2a,6a
7
1b
7a
9
2b
3b,4b,5b,6b
7b
9
7b
6b
3b
4b 2b
5b
7a
7 1b
3a,5a 2a,6a
4c
f2 (ppm)
f1 (ppm)
11.0
7.0
6.9
6.8
5.0
4.4
4.3
3.8
2.3
1.8
1.4
1.3
0.9
10
15
20
25
30
35
55
65
70
115
120
013

13C HMBC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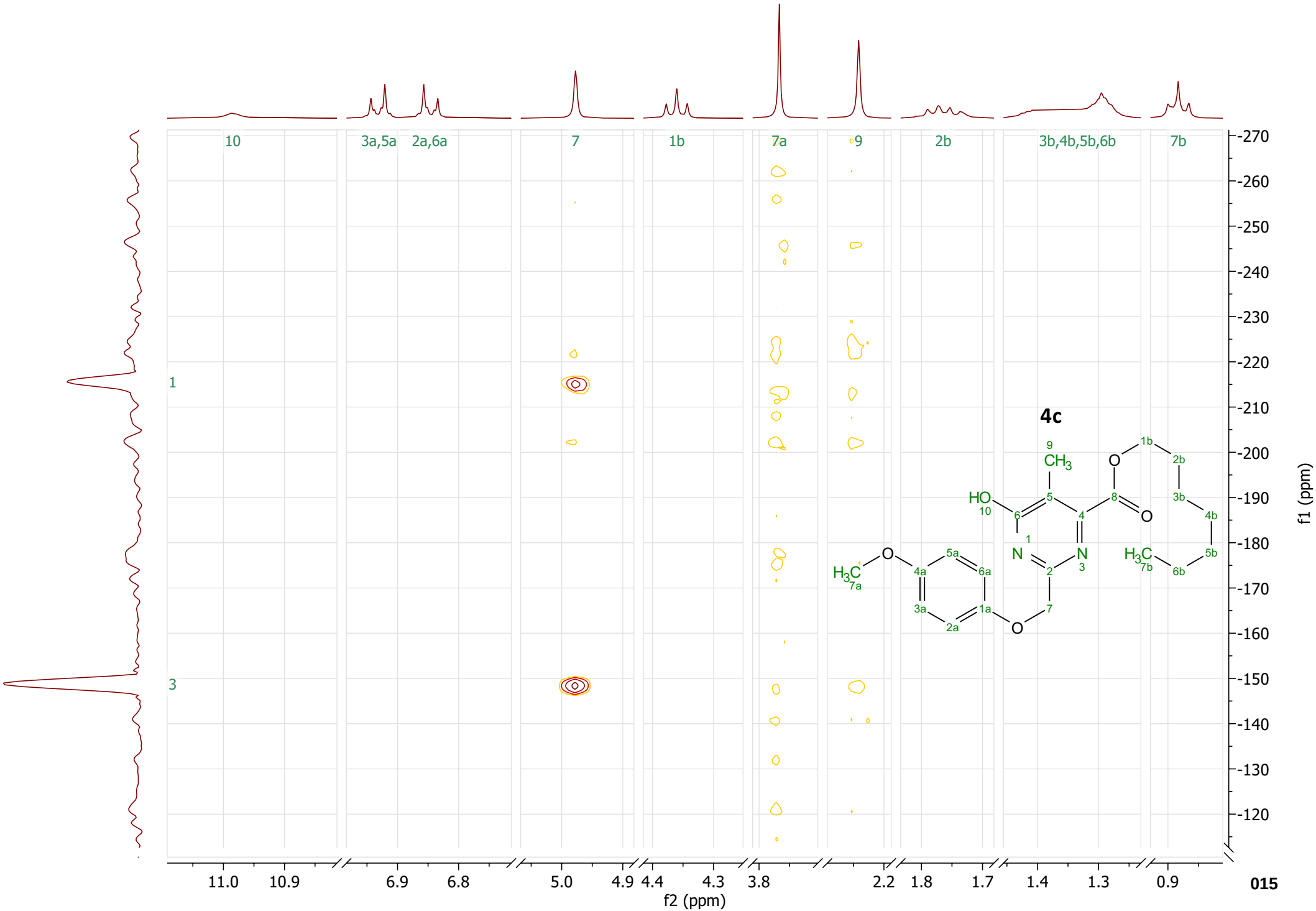
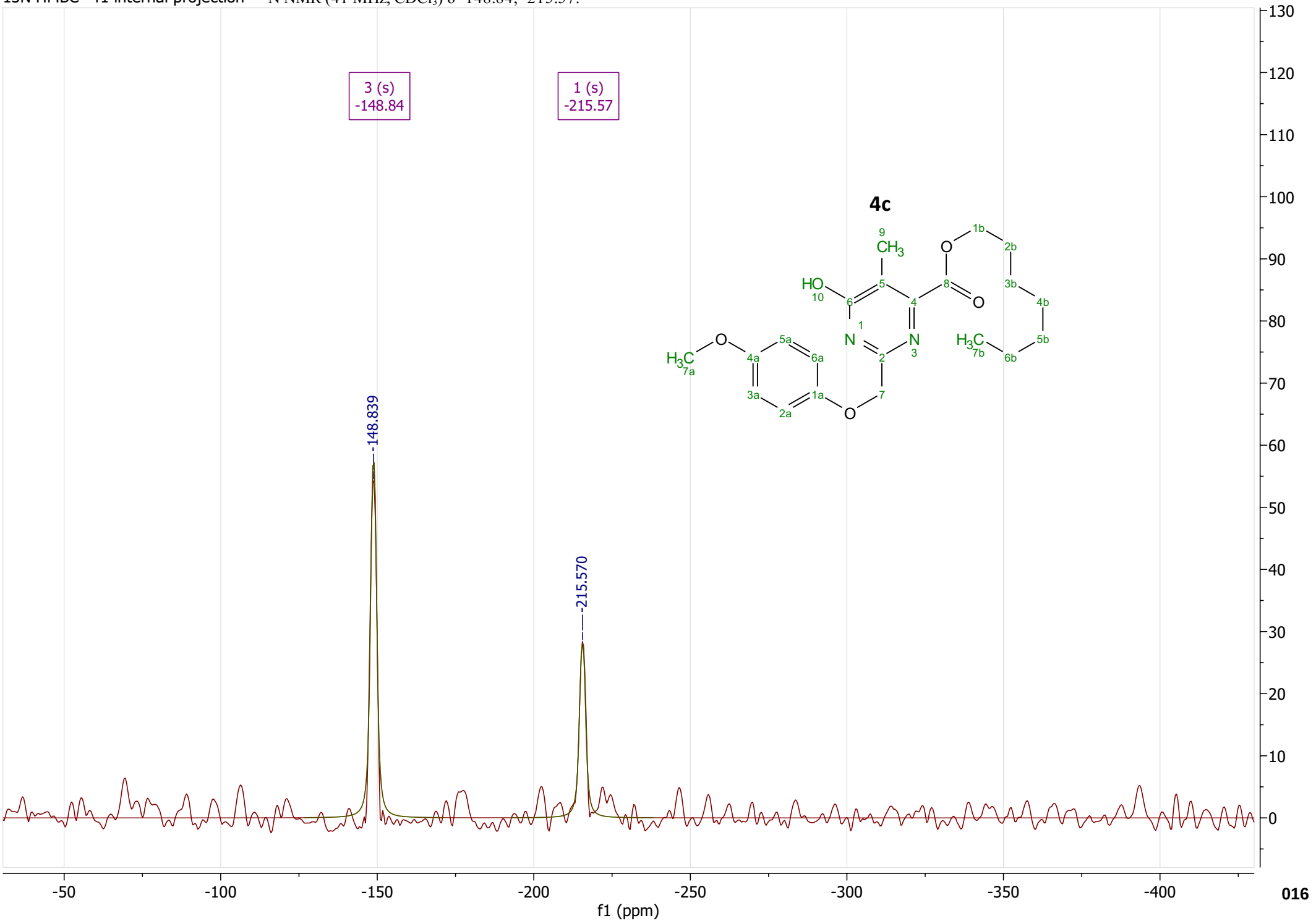
4b
2b
5b
7a
7
1b
3a,5a
2a,6a
5
1a
4
4a
2
8
6
4c
f1 (ppm)
f2 (ppm)
014

15N HMBC

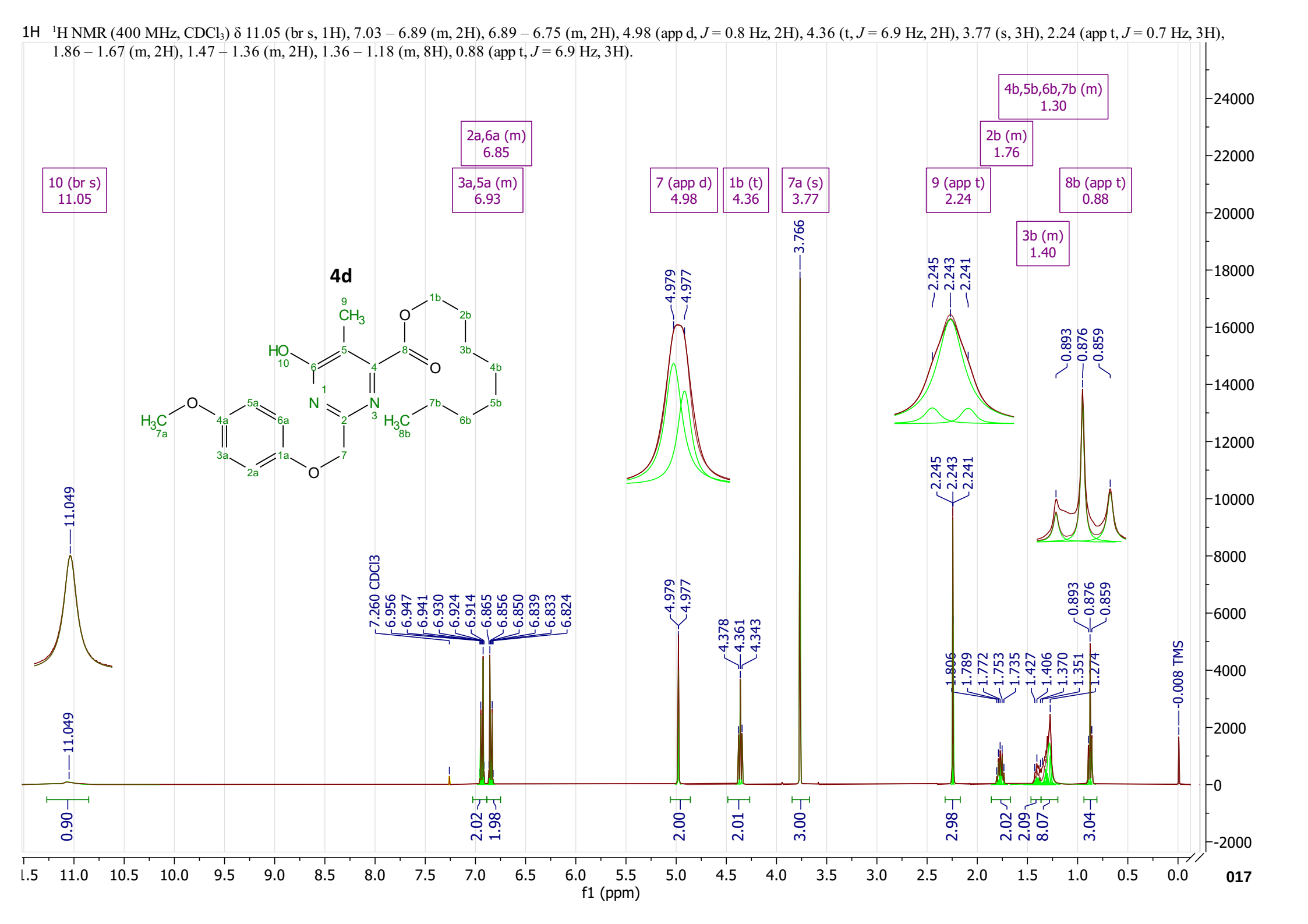


15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -148.84, -215.57.

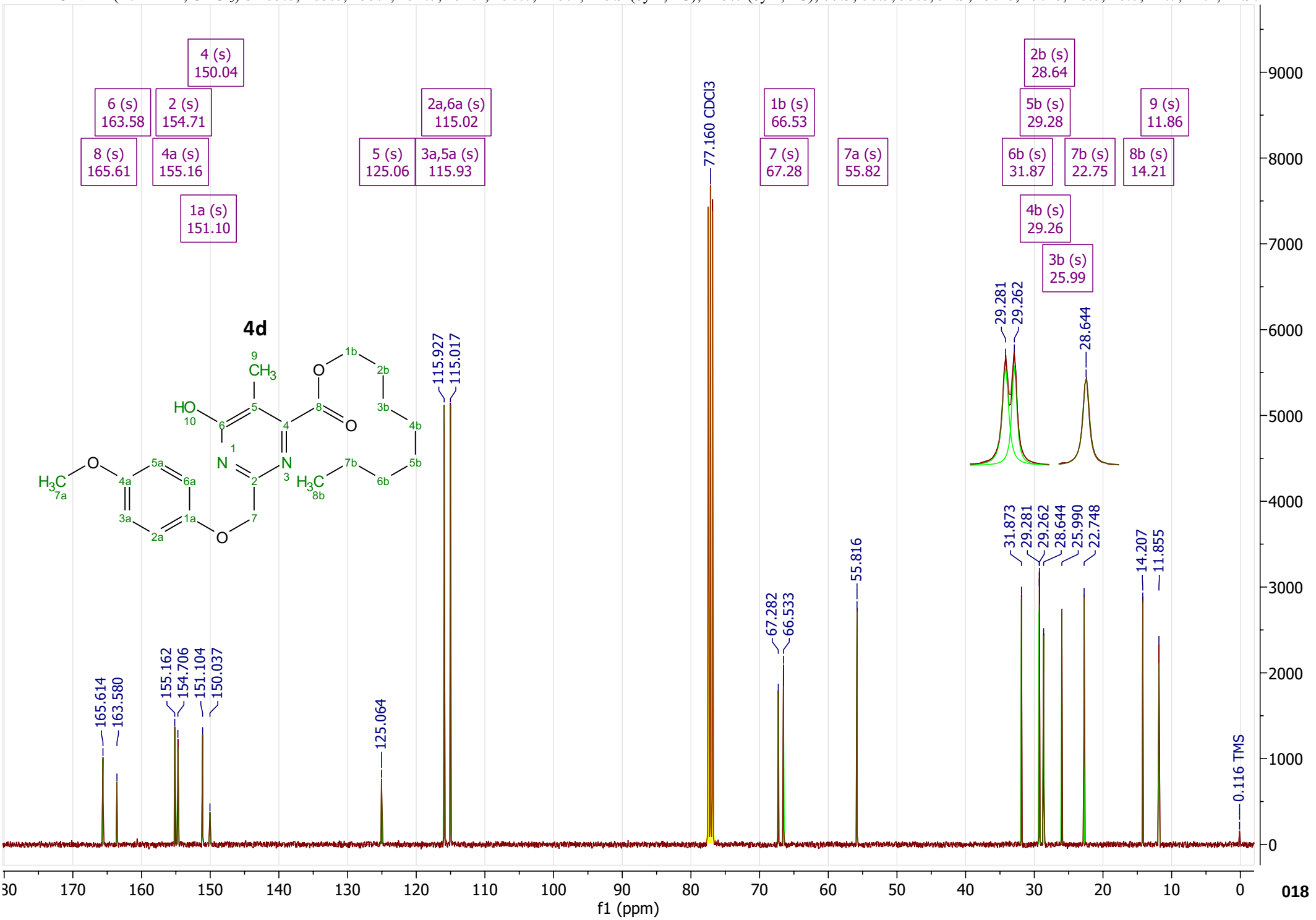


016

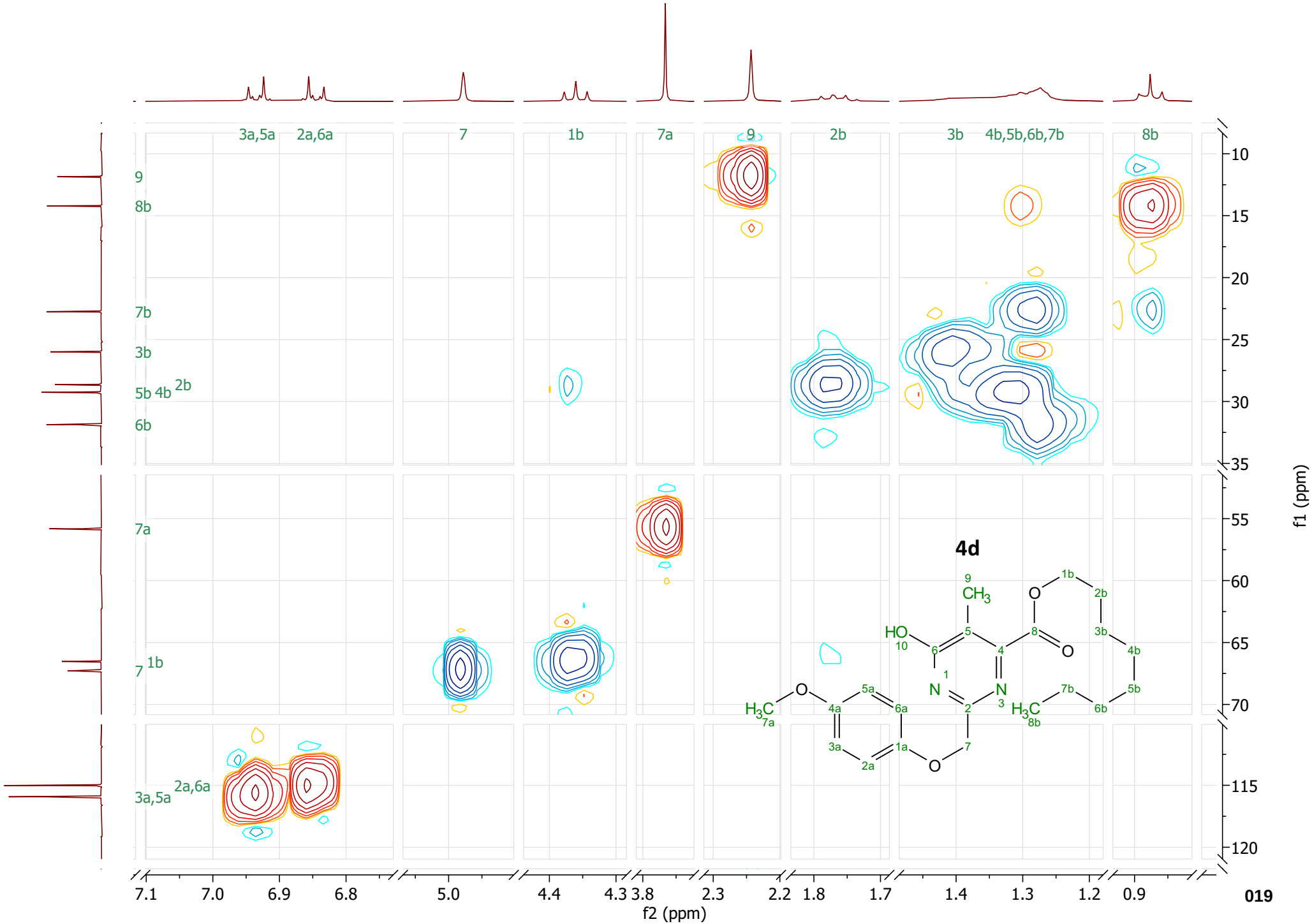
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.05 (br s, 1H), 7.03 – 6.89 (m, 2H), 6.89 – 6.75 (m, 2H), 4.98 (app d, *J* = 0.8 Hz, 2H), 4.36 (t, *J* = 6.9 Hz, 2H), 3.77 (s, 3H), 2.24 (app t, *J* = 0.7 Hz, 3H), 1.86 – 1.67 (m, 2H), 1.47 – 1.36 (m, 2H), 1.36 – 1.18 (m, 8H), 0.88 (app t, *J* = 6.9 Hz, 3H).



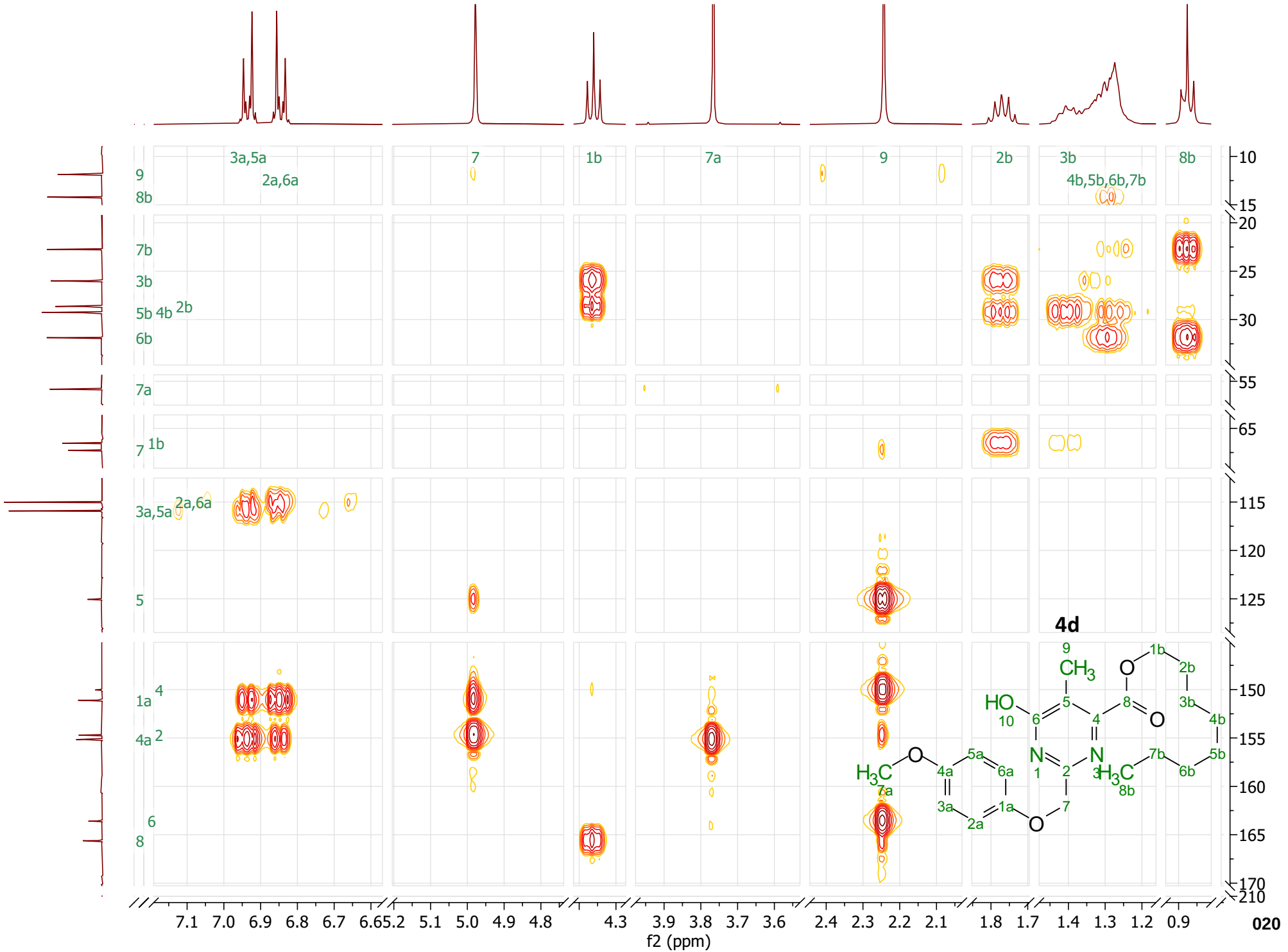
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 163.6, 155.2, 154.7, 151.1, 150.0, 125.1, 115.9 (sym, 2C), 115.0 (sym, 2C), 67.3, 66.5, 55.8, 31.9, 29.28, 29.26, 28.6, 26.0, 22.7, 14.2, 11.9.



13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
9
8b
7b
3b
5b 4b 2b
6b
7a
7 1b
3a,5a 2a,6a
4d
f2 (ppm)
f1 (ppm)
019

13C HMBC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
9
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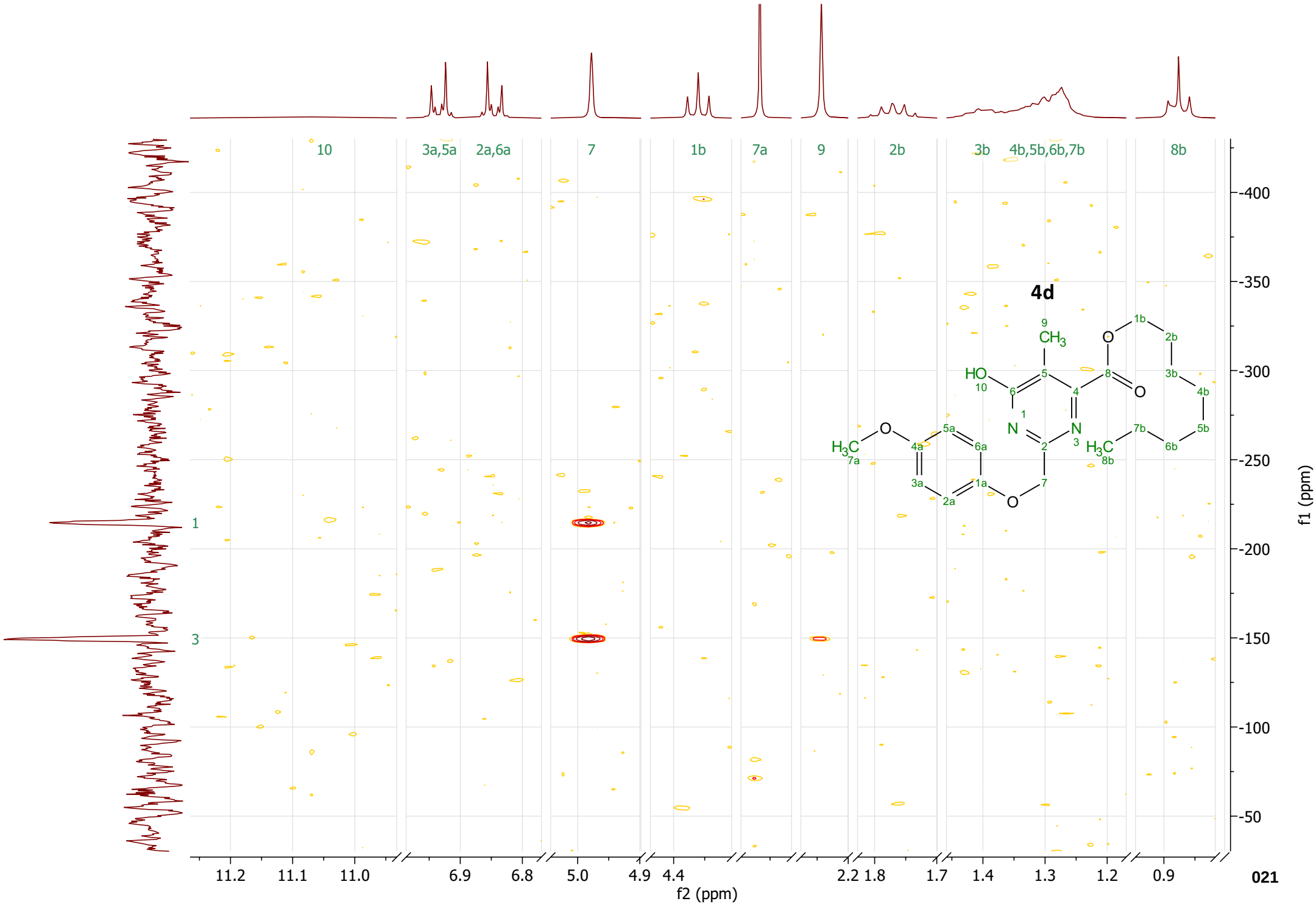
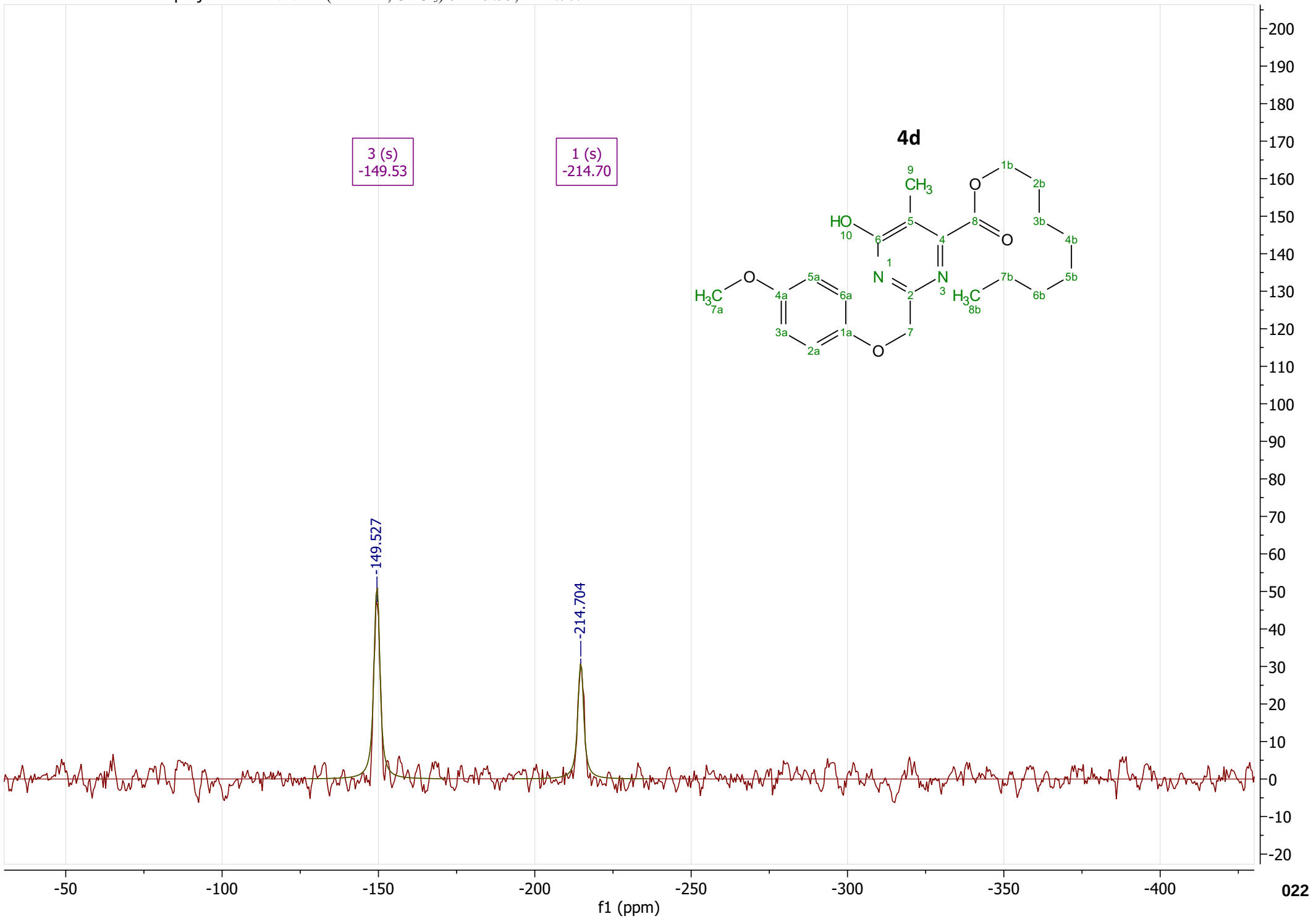
7b
3b
5b
4b
2b
6b
7a
7
1b
3a,5a
2a,6a
5
1a
4
4a
2
8
6
4d
f2 (ppm)
f1 (ppm)
020

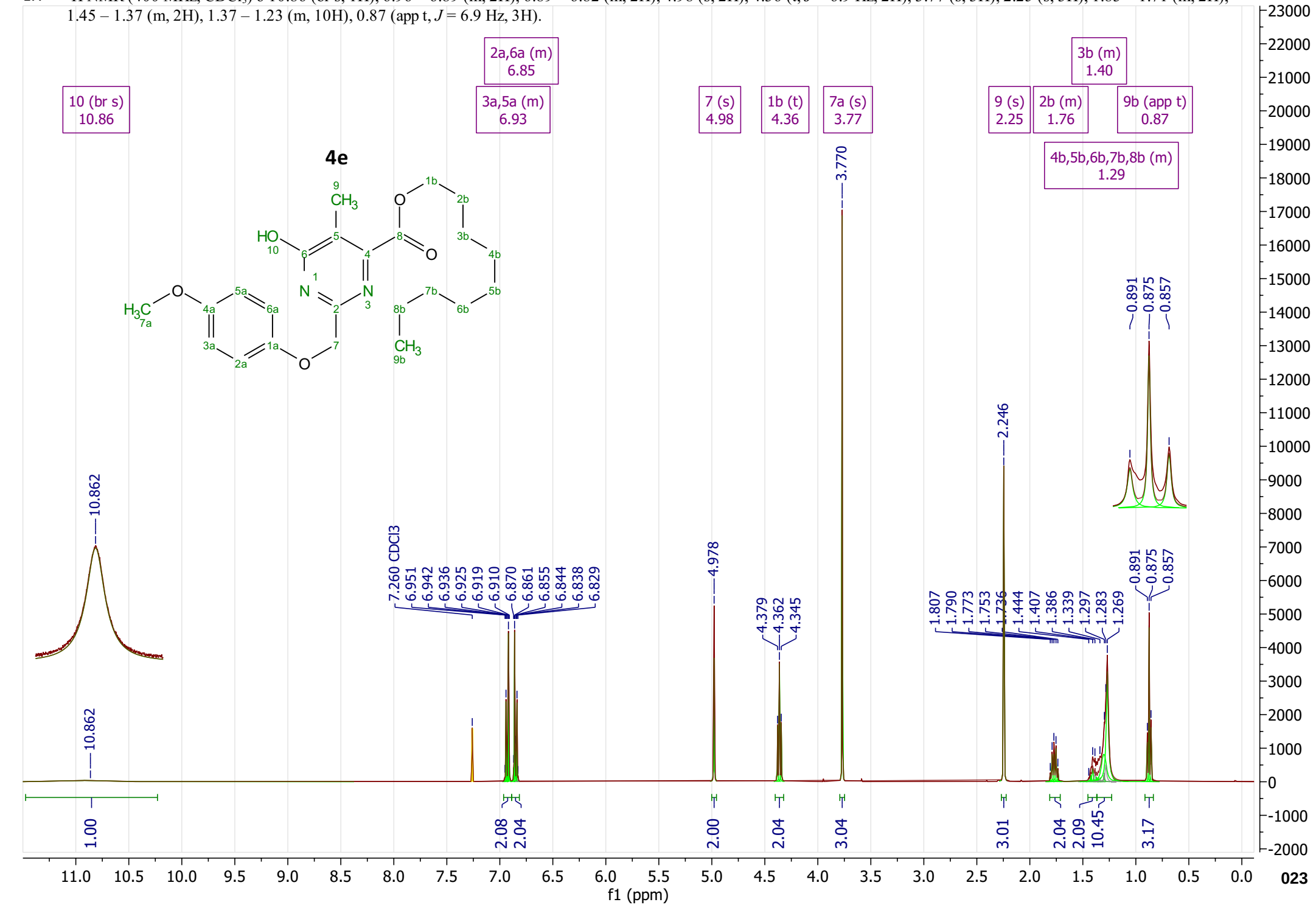
15N HMBC


10
3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
4d
1
3
-400
-350
-300
-250
-200
-150
-100
-50
f1 (ppm)
11.2
11.1
11.0
6.9
6.8
5.0
4.9
4.4
2.2
1.8
1.7
1.4
1.3
1.2
0.9
f2 (ppm)
021

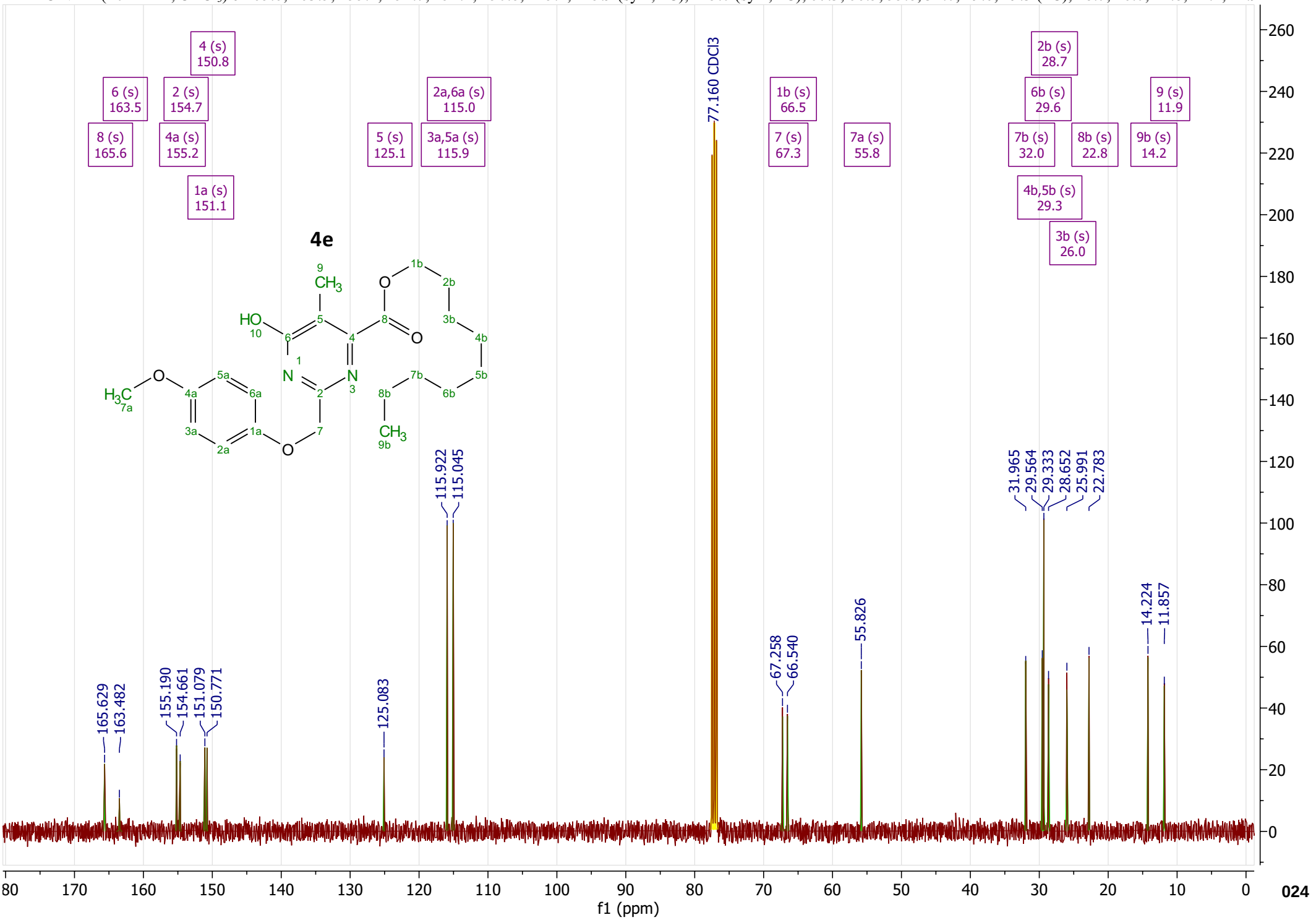
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -149.53, -214.70.



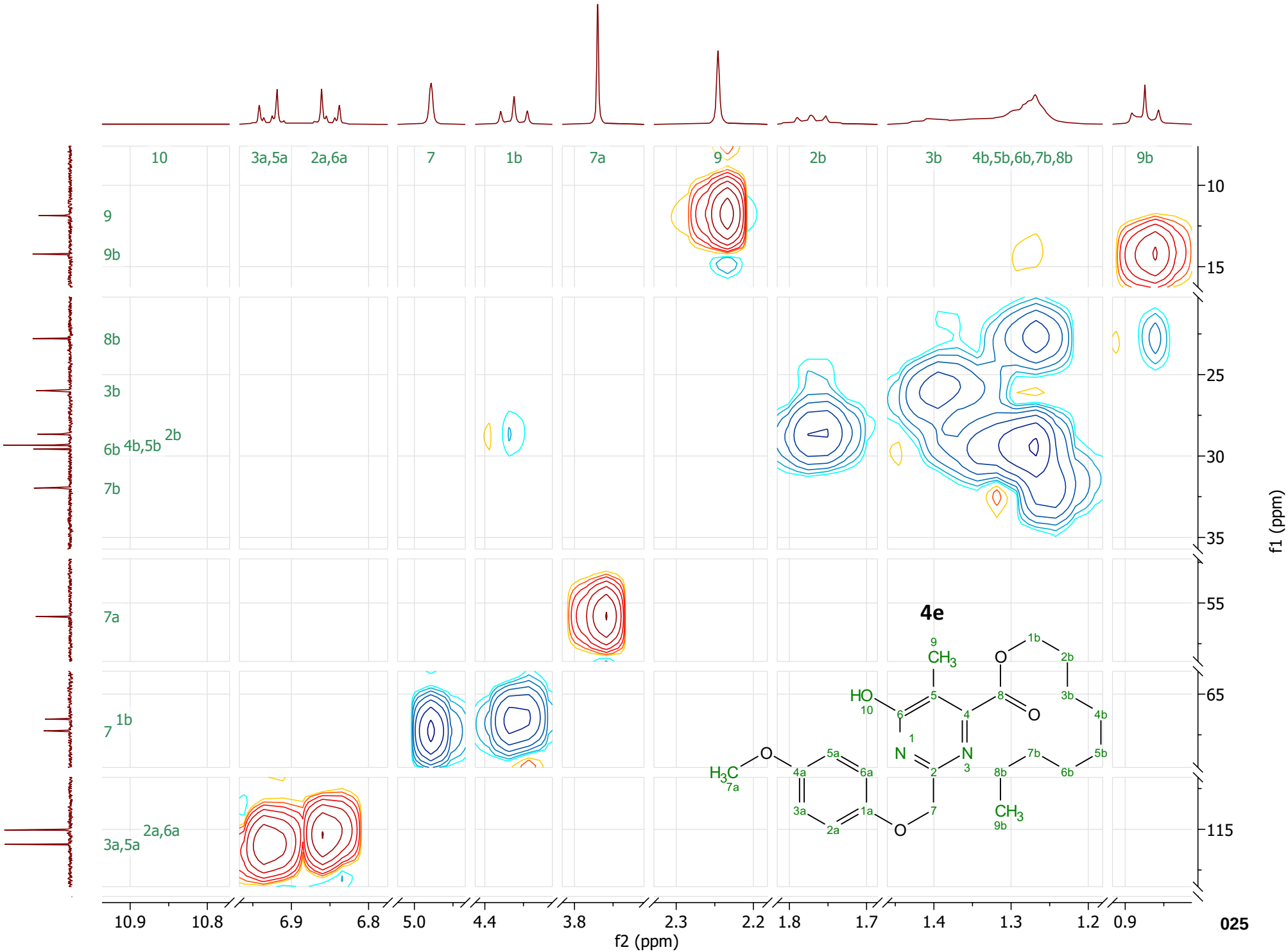
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.86 (br s, 1H), 6.96 – 6.89 (m, 2H), 6.89 – 6.82 (m, 2H), 4.98 (s, 2H), 4.36 (t, J = 6.9 Hz, 2H), 3.77 (s, 3H), 2.25 (s, 3H), 1.83 – 1.71 (m, 2H), 1.45 – 1.37 (m, 2H), 1.37 – 1.23 (m, 10H), 0.87 (app t, J = 6.9 Hz, 3H).



13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.6, 163.5, 155.2, 154.7, 151.1, 150.8, 125.1, 115.9 (sym, 2C), 115.0 (sym, 2C), 67.3, 66.5, 55.8, 32.0, 29.6, 29.3 (2C), 28.7, 26.0, 22.8, 14.2, 11.9



13C HSQC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3a,5a
2a,6a
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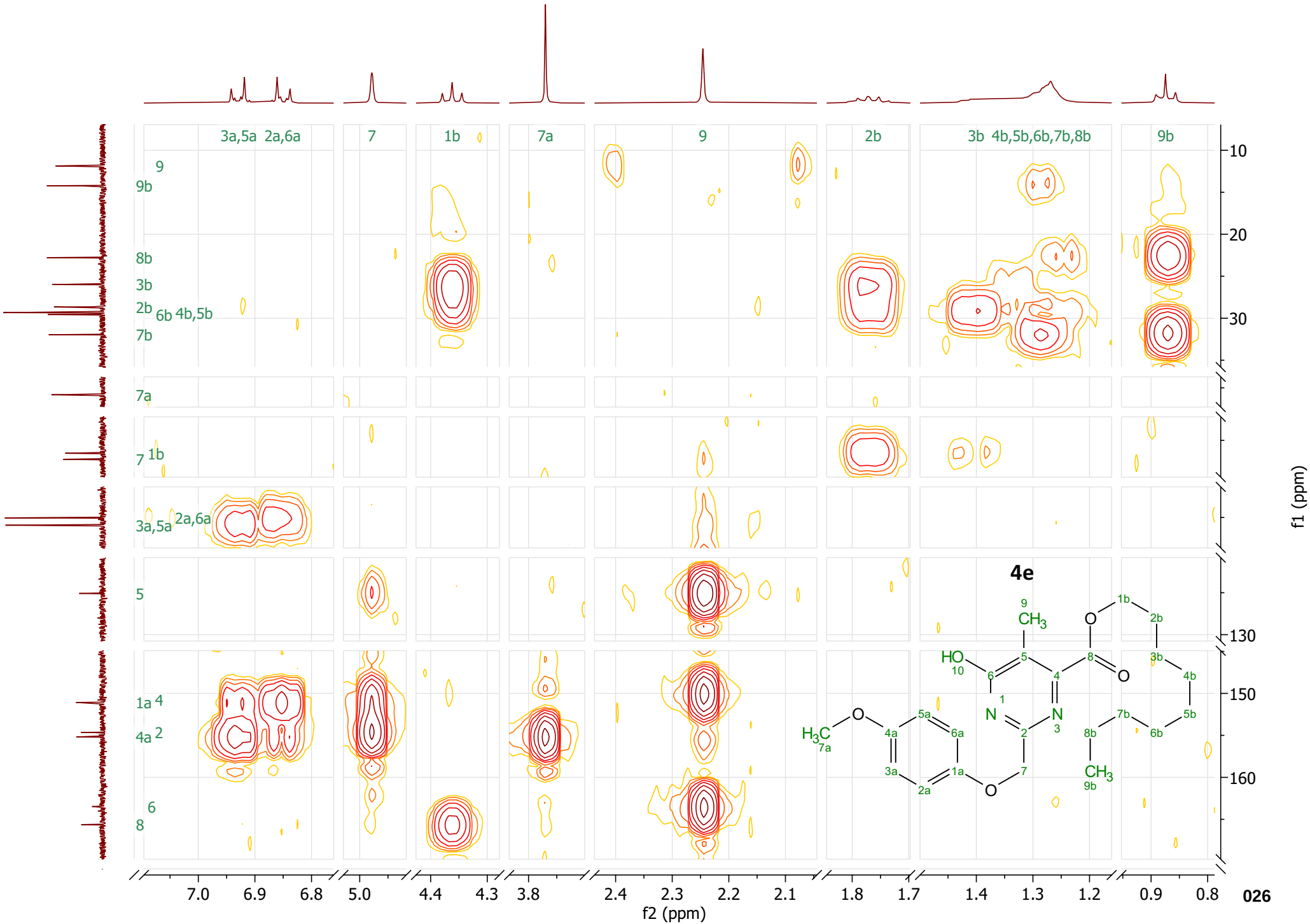
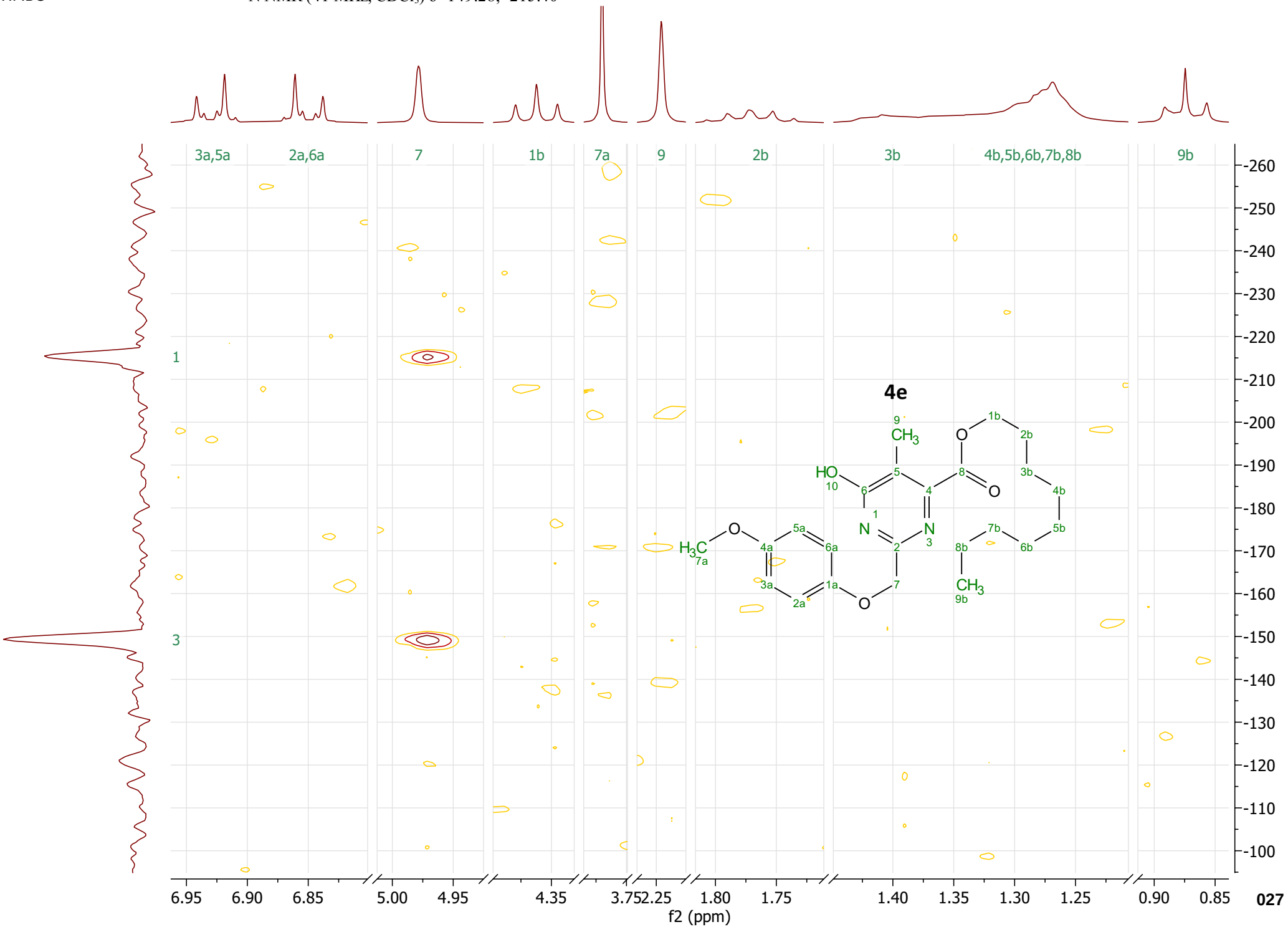
1b
7a
9
2b
3b
4b,5b,6b,7b,8b
9b
9
9b
8b
3b
6b
4b,5b
2b
7b
7a
7
1b
3a,5a
2a,6a
4e
f2 (ppm)
f1 (ppm)
025

13C HMBC


3a,5a 2a,6a
7
1b
7a
9
2b
3b 4b,5b,6b,7b,8b
9b
9
9b
8b
3b
2b 6b 4b,5b
7b
7a
7 1b
3a,5a 2a,6a
5
1a 4
4a 2
6
8
4e
f2 (ppm)
f1 (ppm)
026

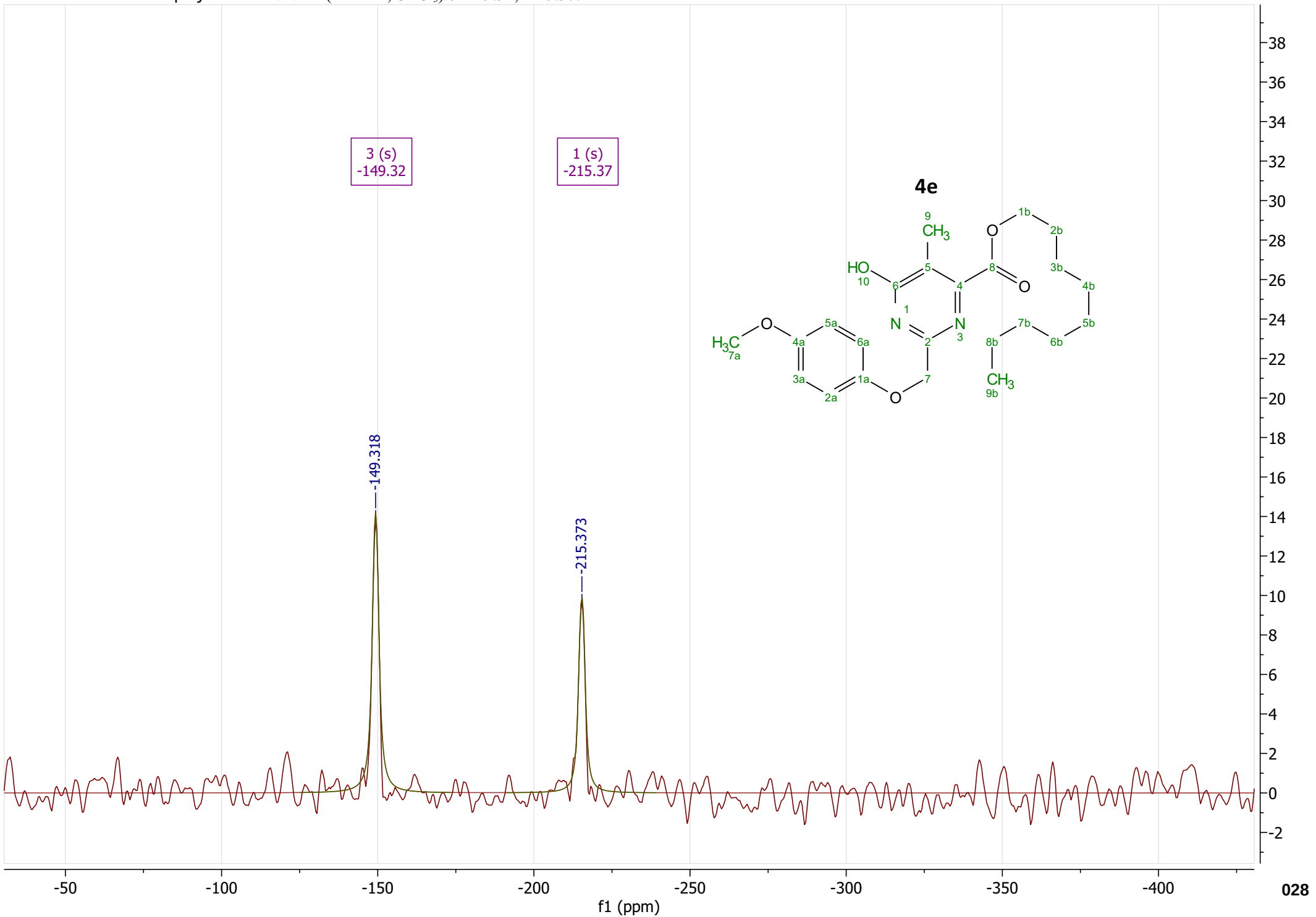
15N HMBC

^{15}N NMR (41 MHz, $CDCl_3$) δ -149.28, -215.40

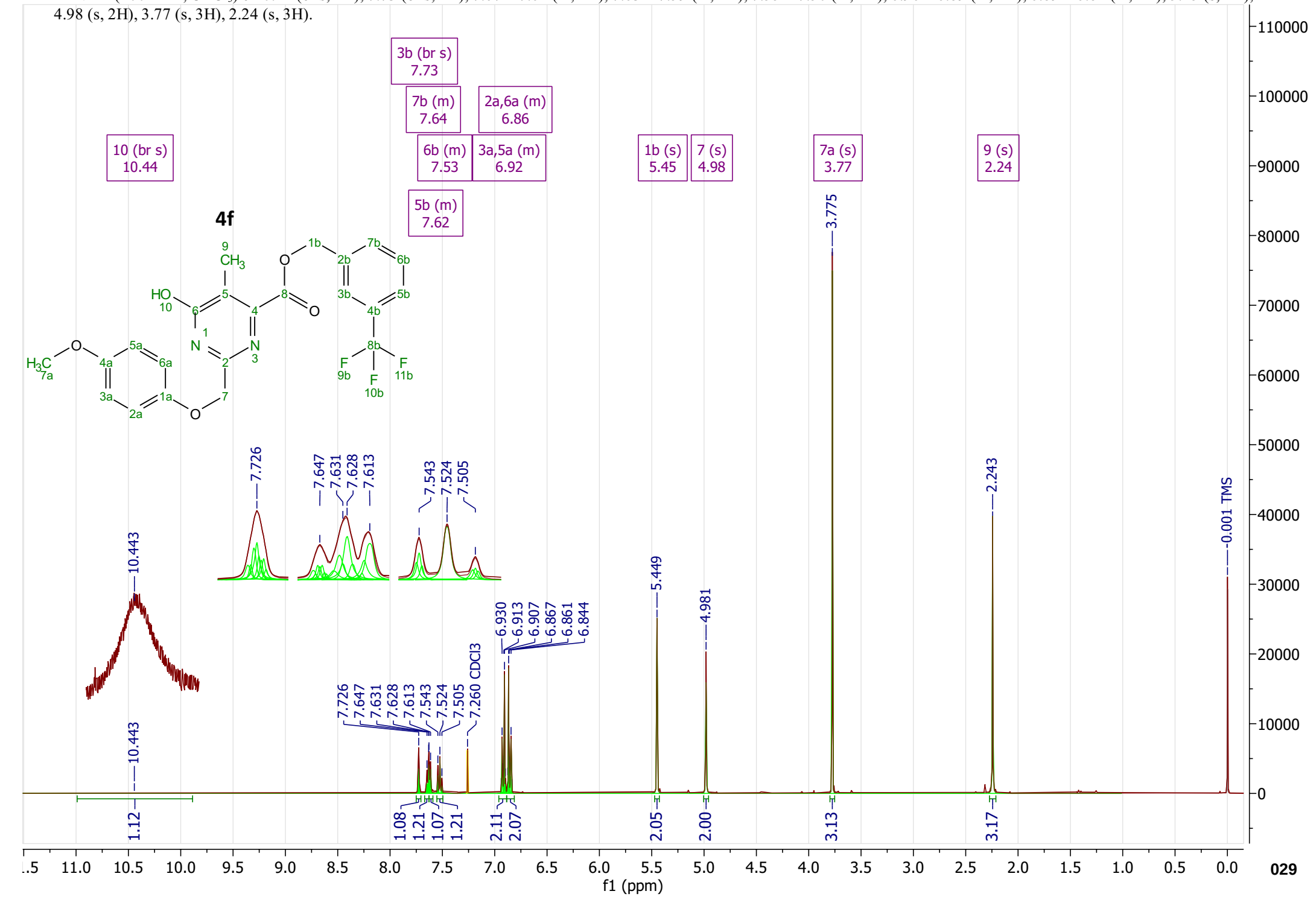


027

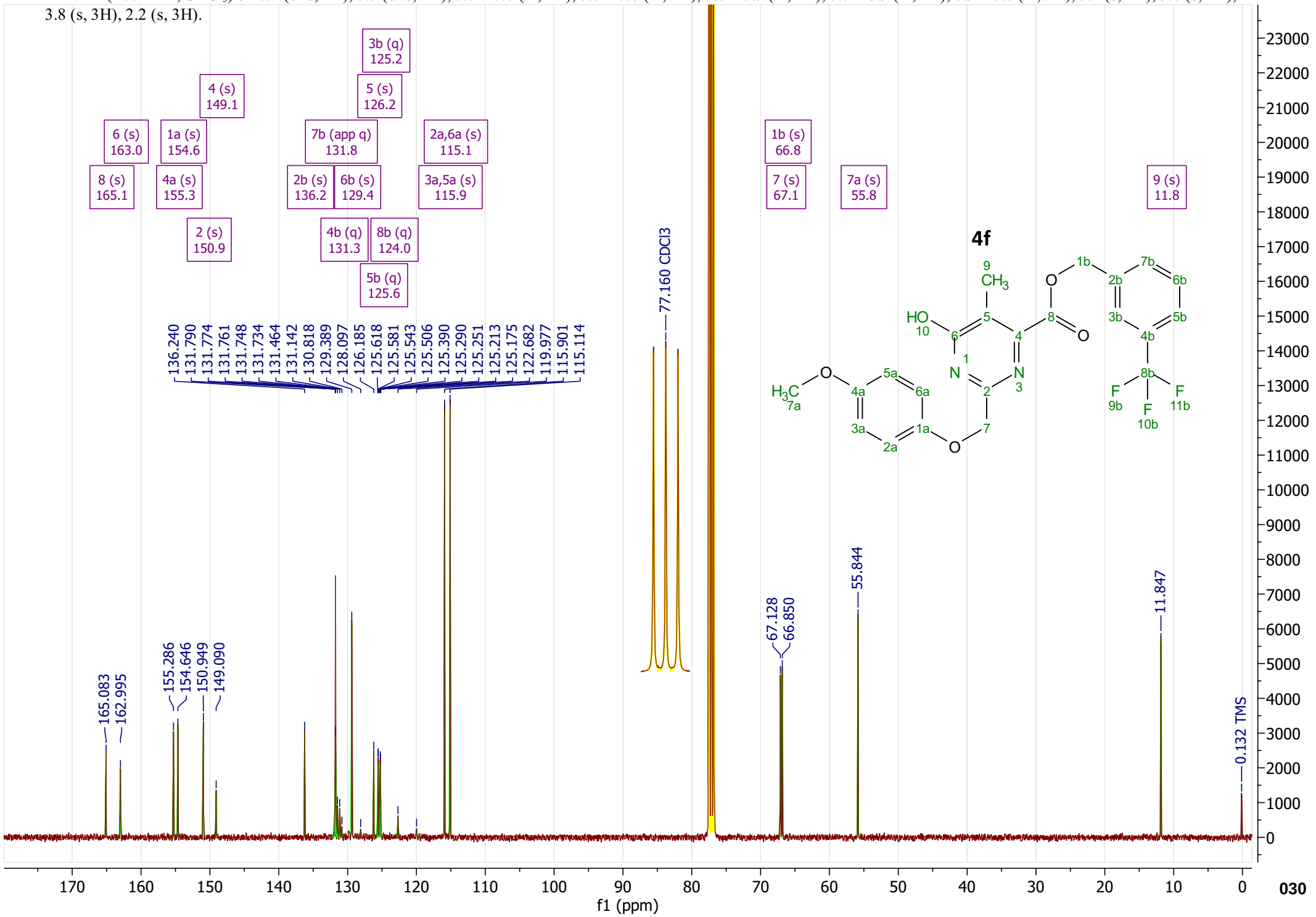
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -149.32, -215.37.



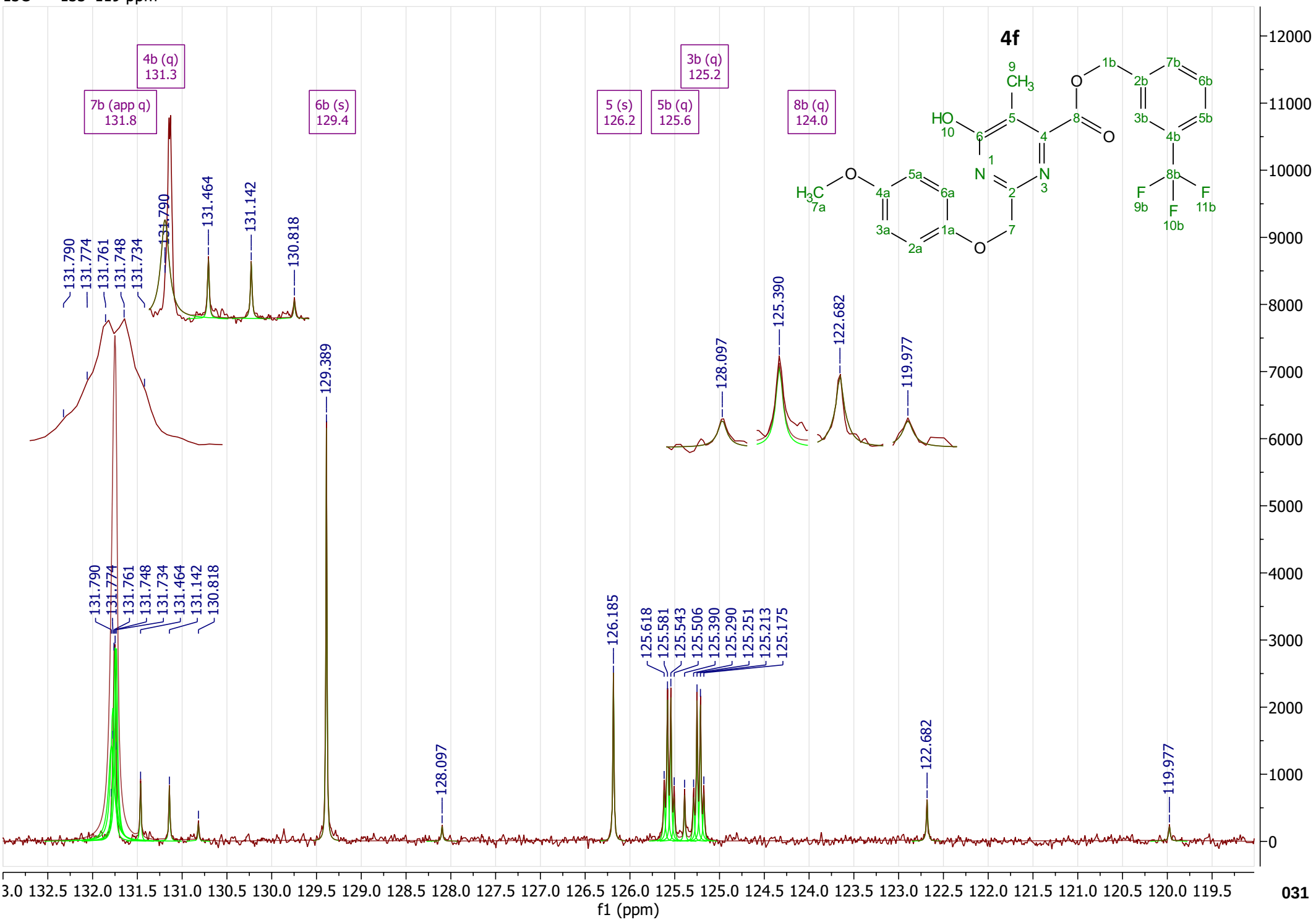
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.44 (br s, 1H), 7.73 (br s, 1H), 7.67 – 7.61 (m, 2H), 7.63 – 7.59 (m, 1H), 7.55 – 7.50 (m, 1H), 6.96 – 6.89 (m, 2H), 6.89 – 6.81 (m, 2H), 5.45 (s, 2H), 4.98 (s, 2H), 3.77 (s, 3H), 2.24 (s, 3H).



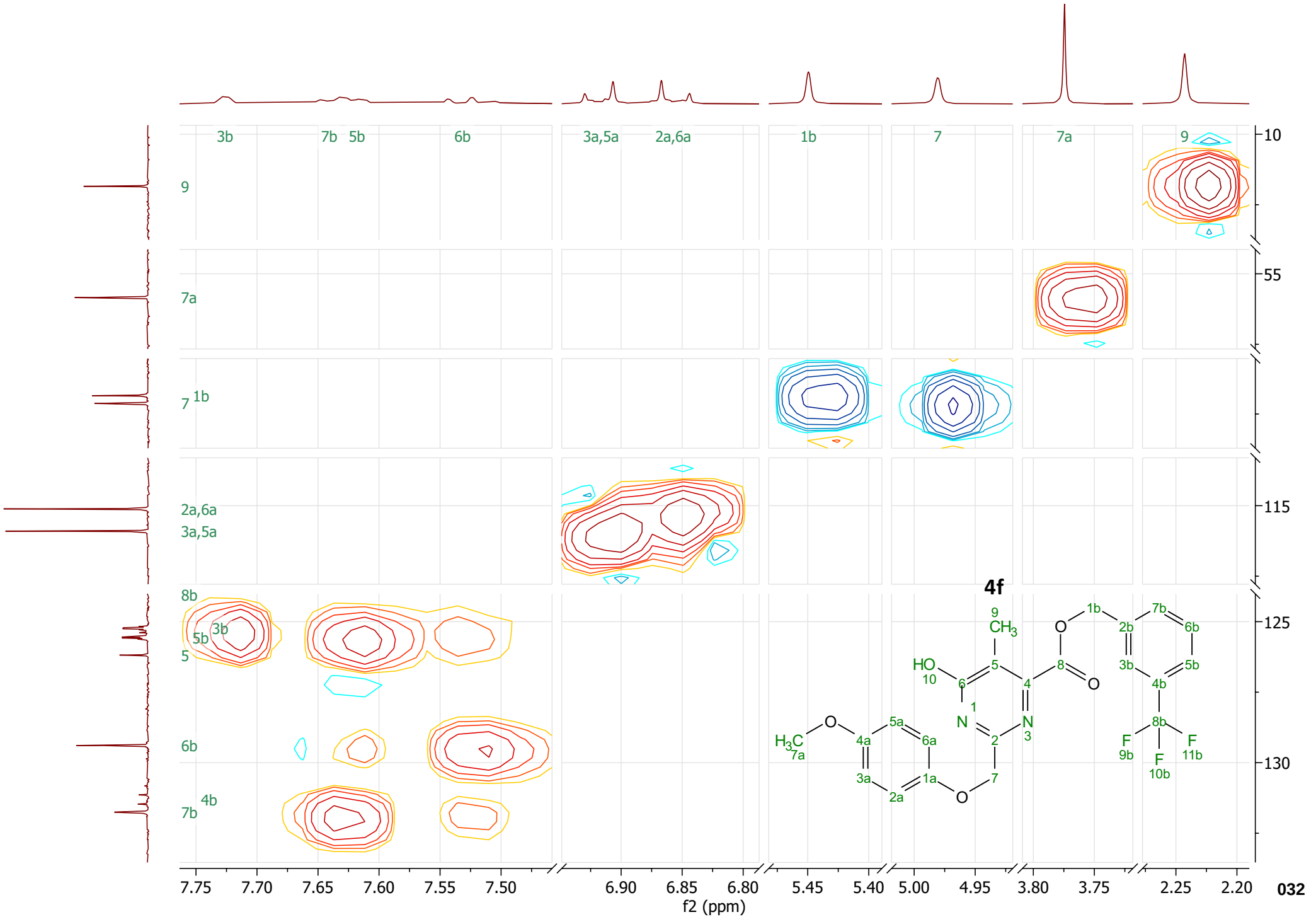
13C ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.4 (br s, 1H), 7.7 (br s, 1H), 7.7 – 7.6 (m, 2H), 7.6 – 7.6 (m, 1H), 7.6 – 7.5 (m, 1H), 7.0 – 6.9 (m, 2H), 6.9 – 6.8 (m, 2H), 5.4 (s, 2H), 5.0 (s, 2H), 3.8 (s, 3H), 2.2 (s, 3H).



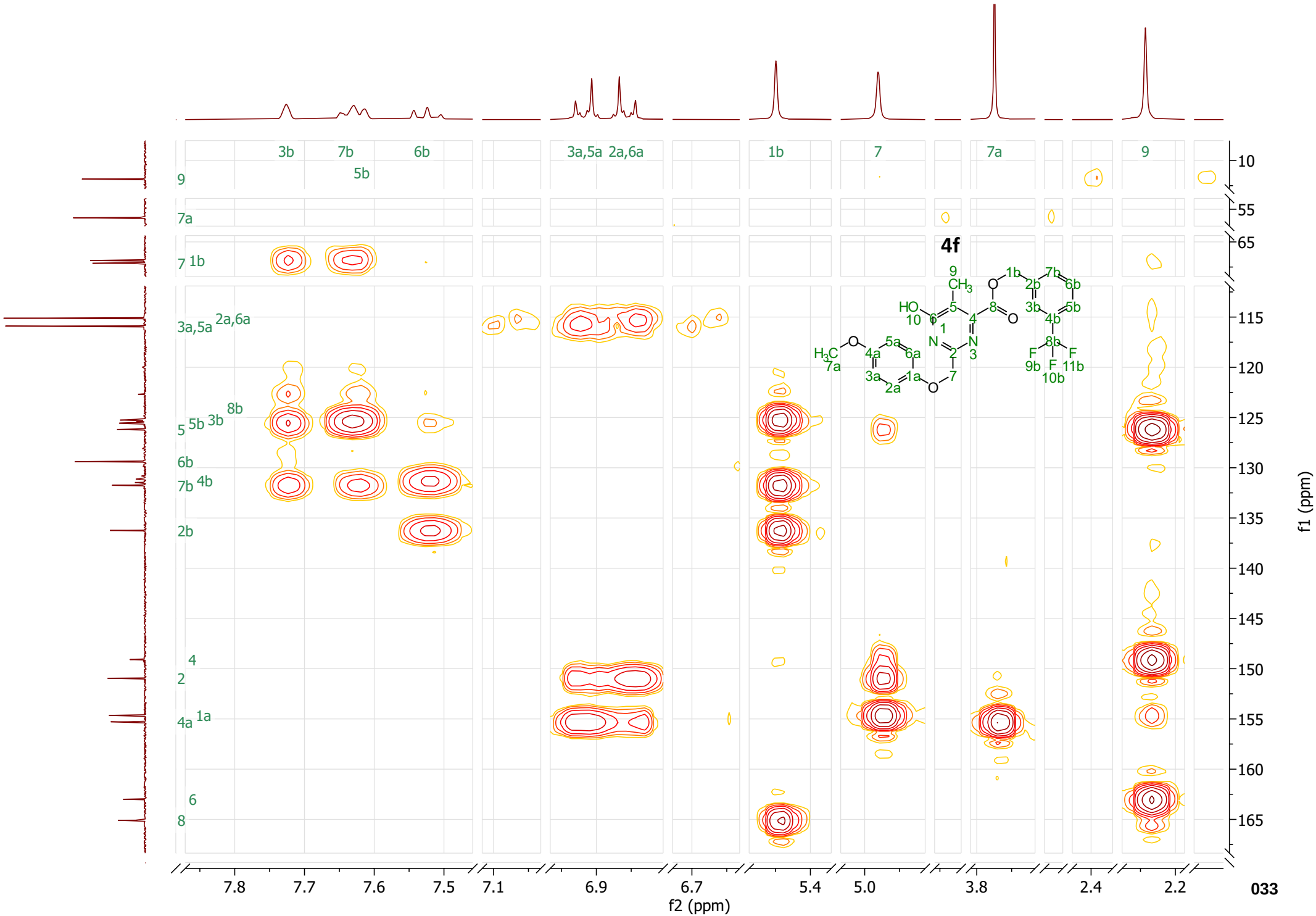
13C — 133–119 ppm



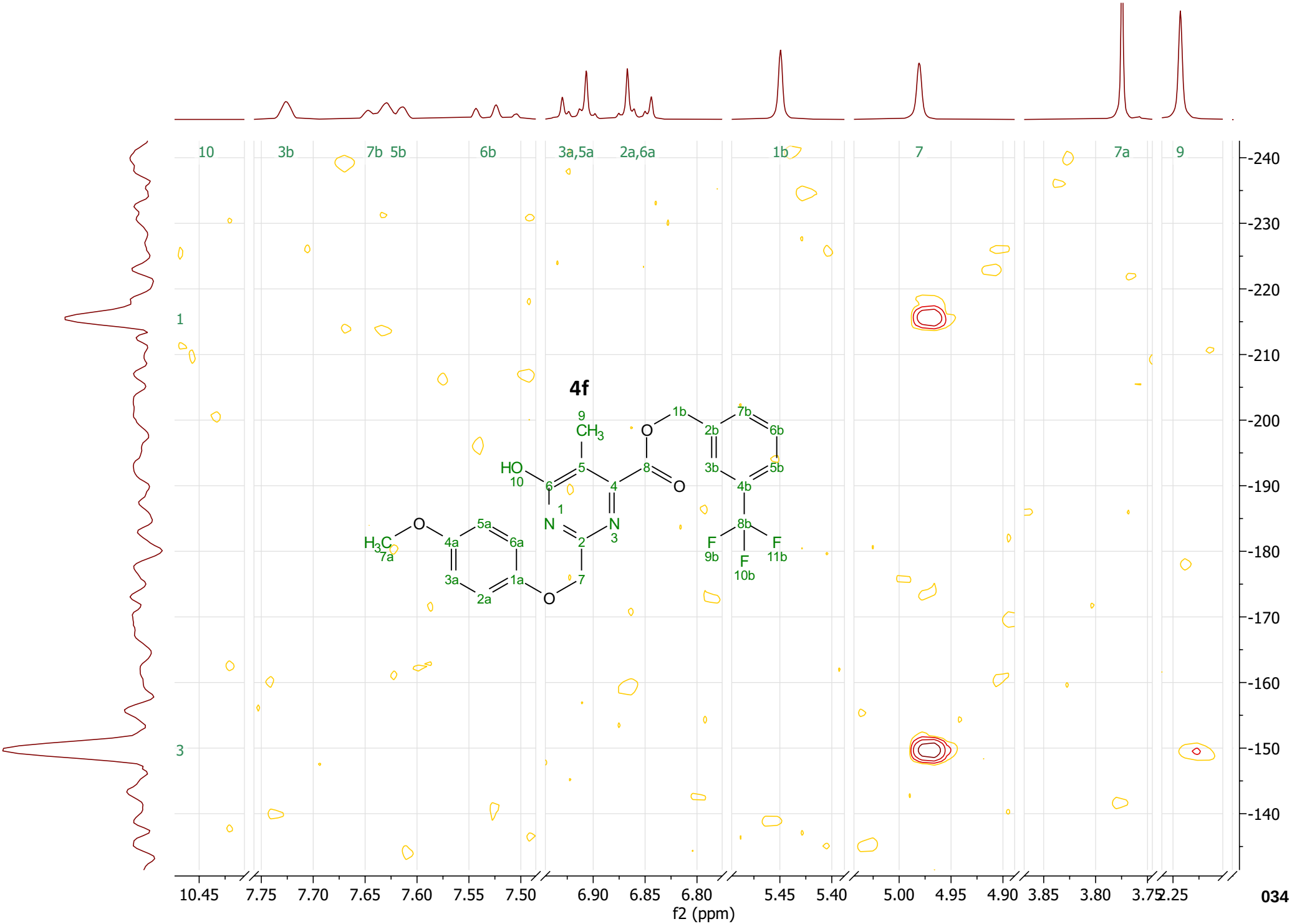
13C HSQC



13C HMBC

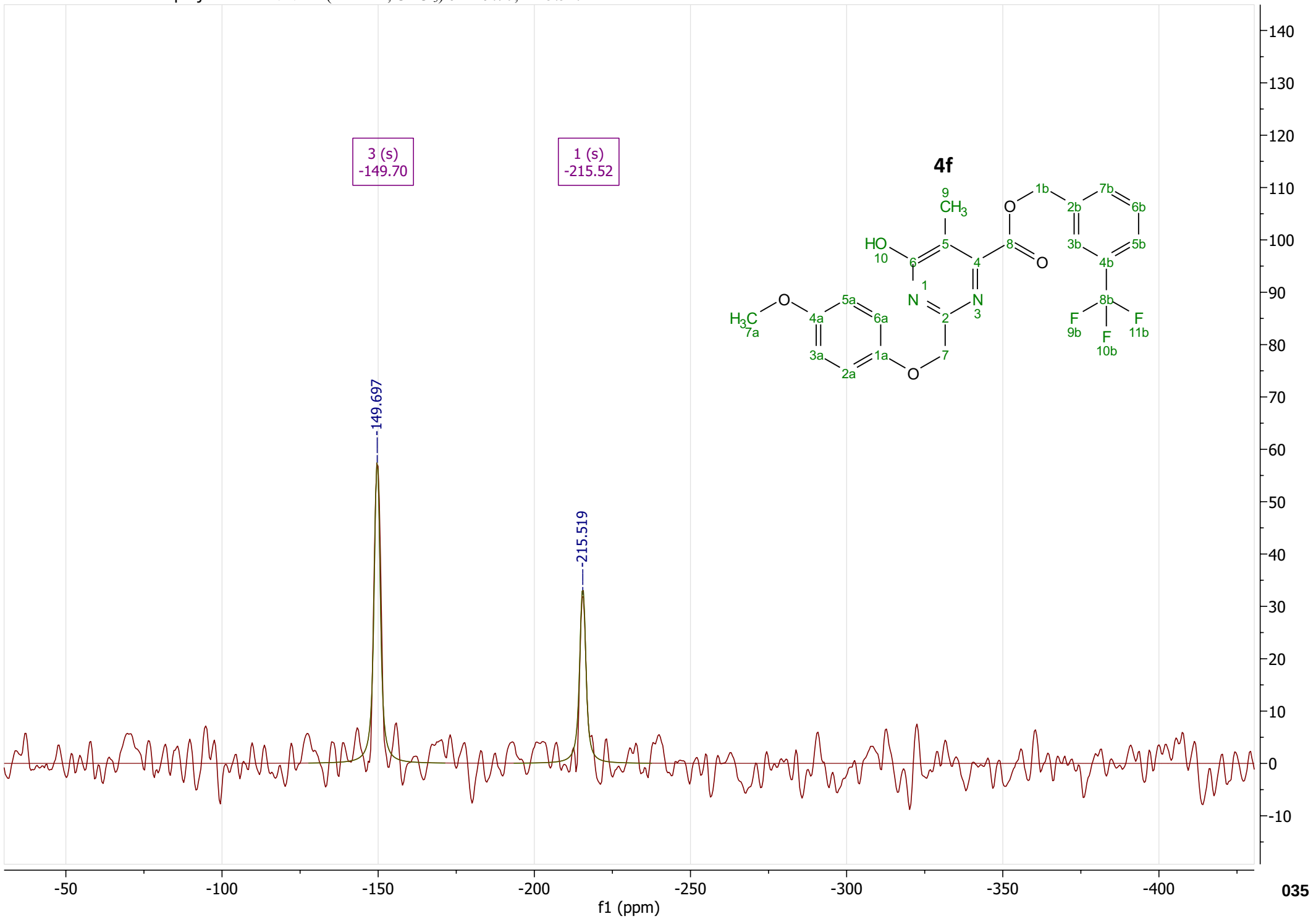


15N HMBC


10
3b
7b 5b
6b
3a,5a
2a,6a
1b
7
7a
9
1
3
4f
f2 (ppm)
f1 (ppm)
10.45
7.75
7.70
7.65
7.60
7.55
7.50
6.90
6.85
6.80
5.45
5.40
5.00
4.95
4.90
3.85
3.80
3.75
2.25
-240
-230
-220
-210
-200
-190
-180
-170
-160
-150
-140

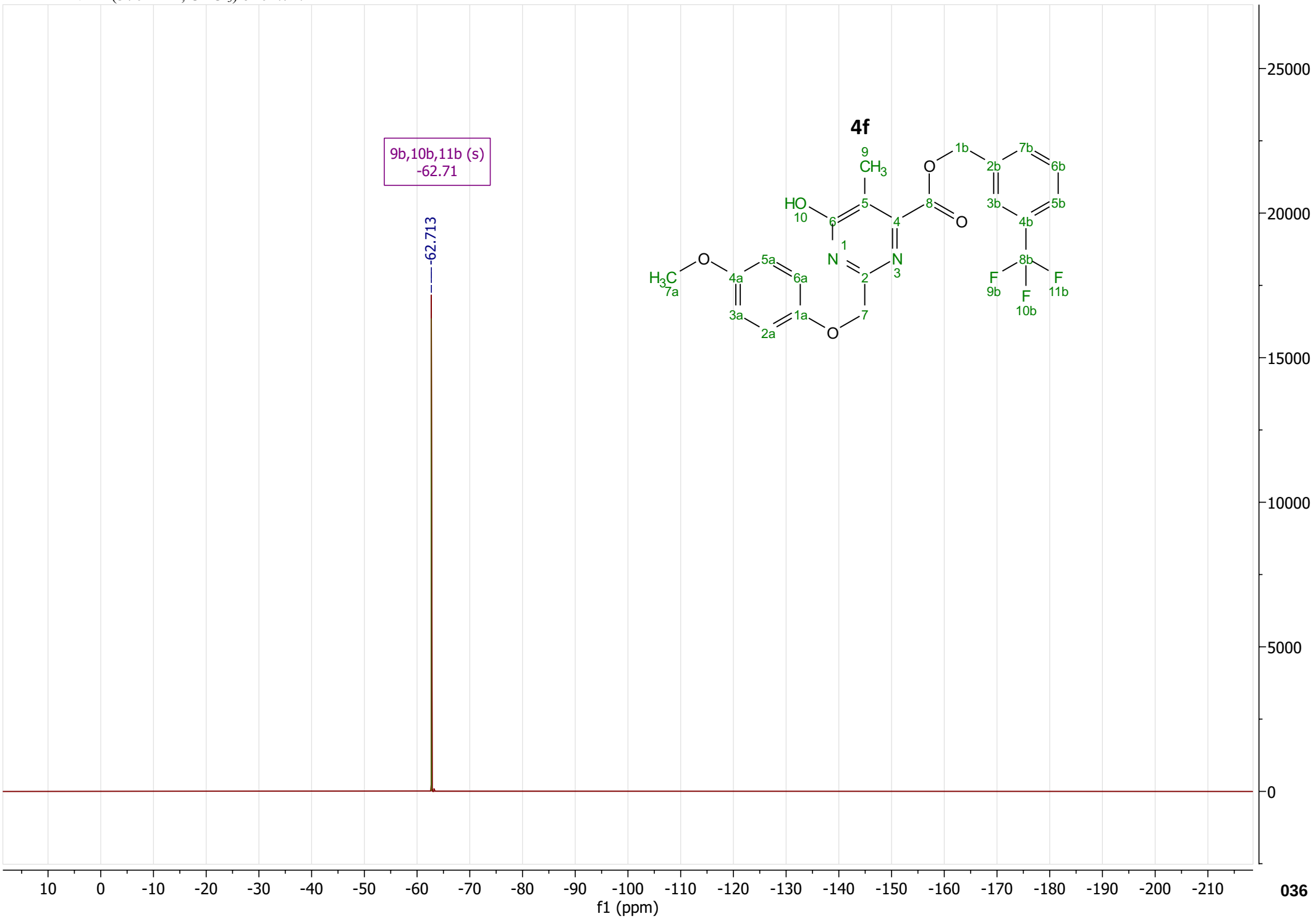
034

15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -149.70, -215.52.

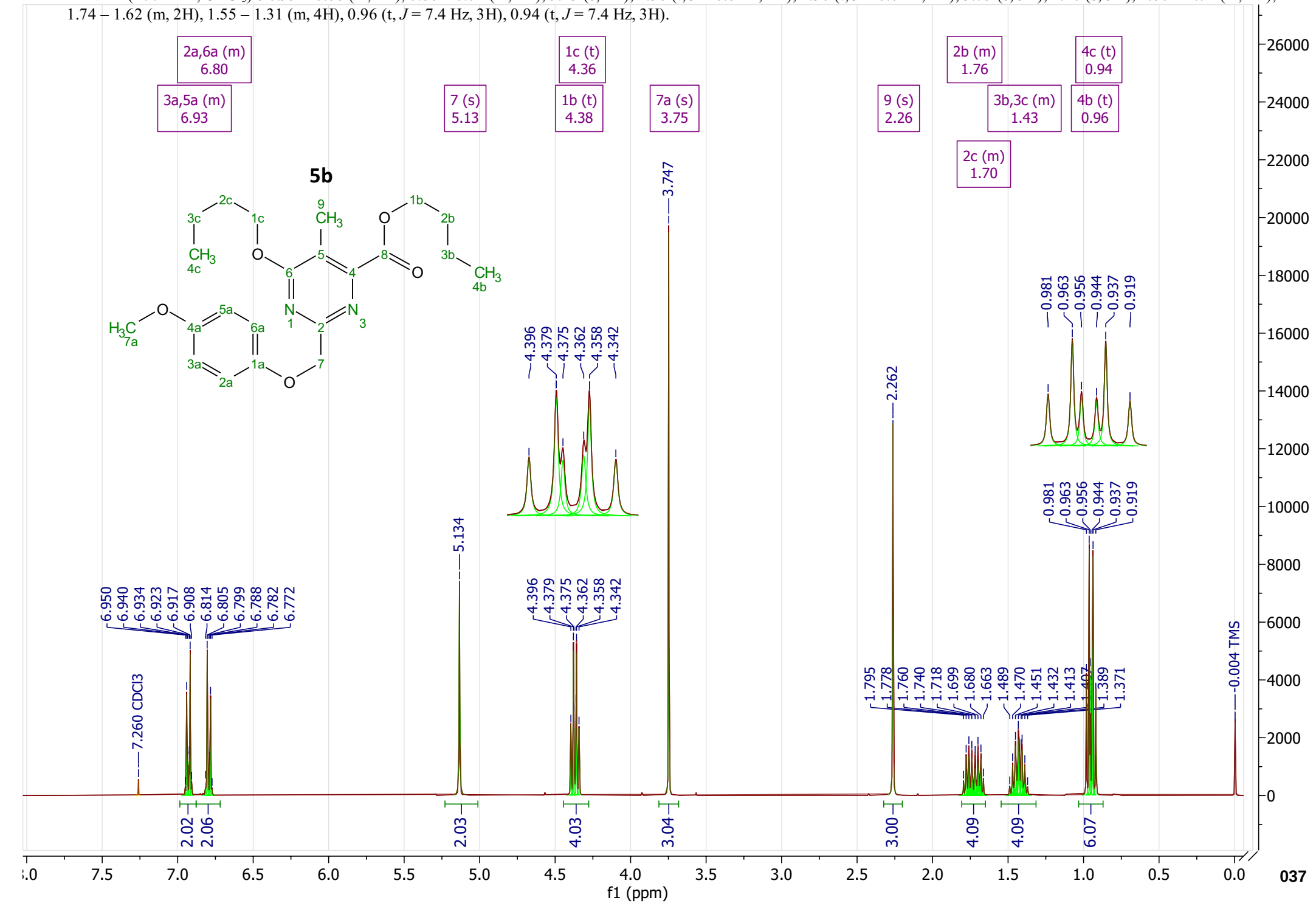


035

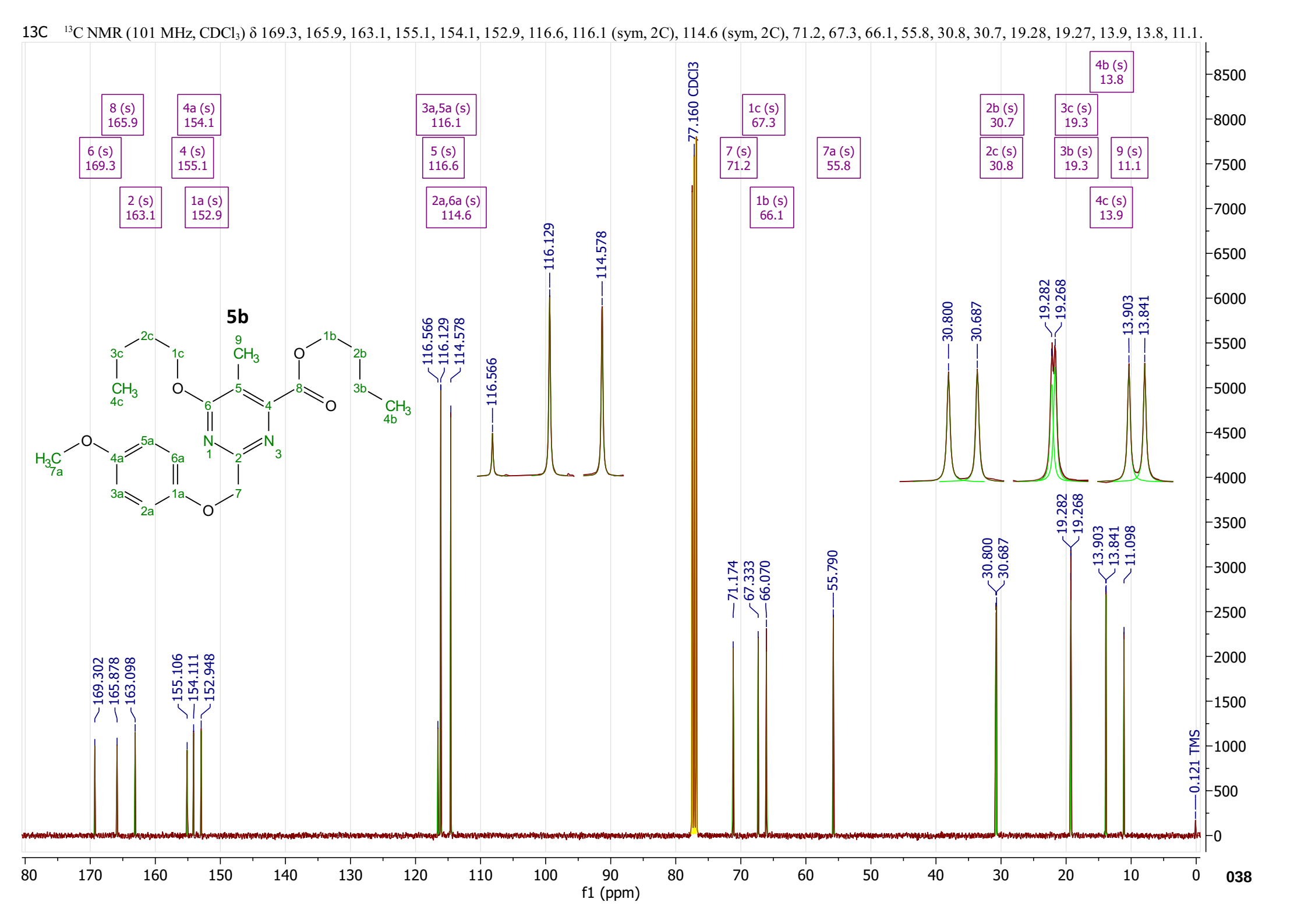
19F ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.71.



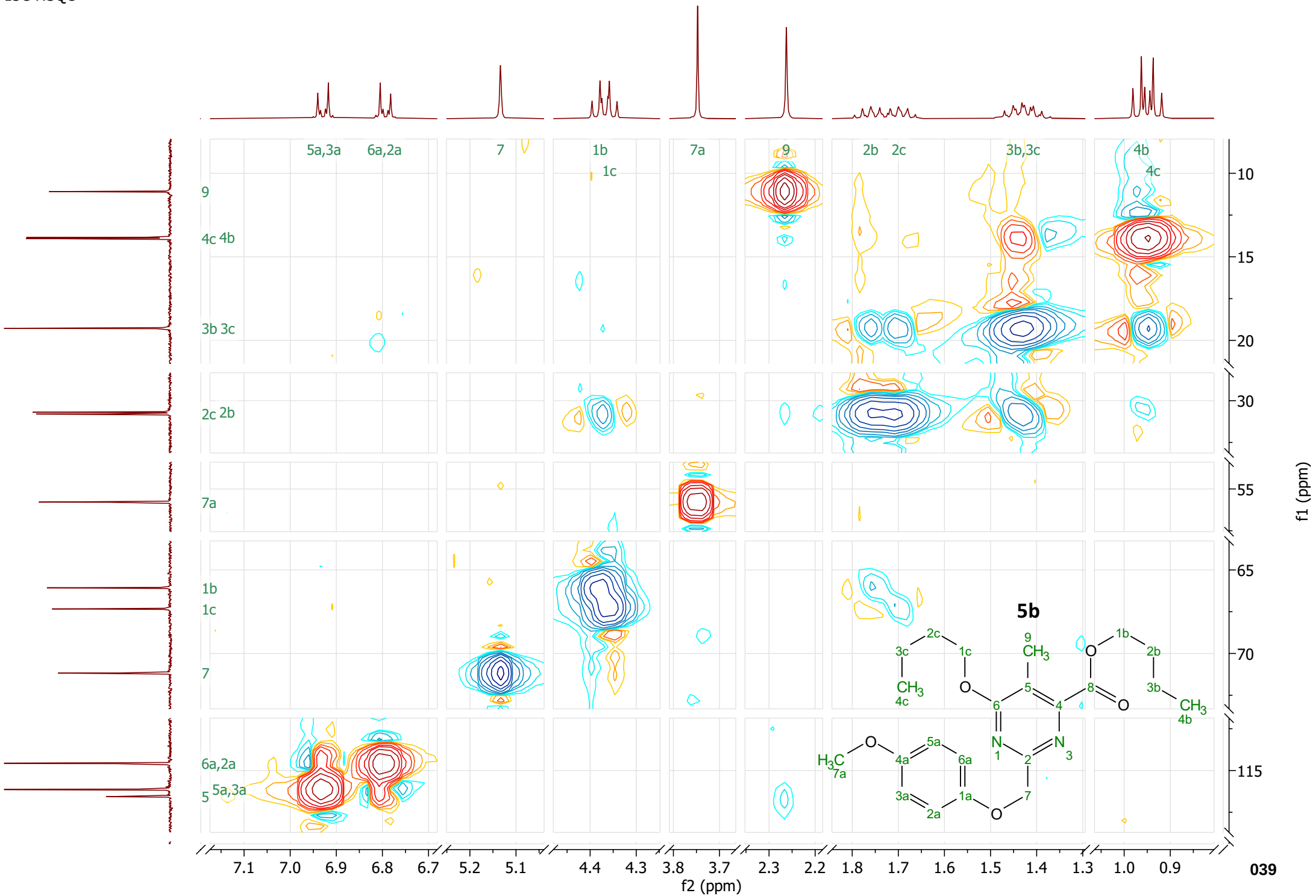
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 – 6.88 (m, 2H), 6.88 – 6.72 (m, 2H), 5.13 (s, 2H), 4.38 (t, *J* = 6.8 Hz, 2H), 4.36 (t, *J* = 6.6 Hz, 2H), 3.75 (s, 3H), 2.26 (s, 3H), 1.86 – 1.72 (m, 2H), 1.74 – 1.62 (m, 2H), 1.55 – 1.31 (m, 4H), 0.96 (t, *J* = 7.4 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H).



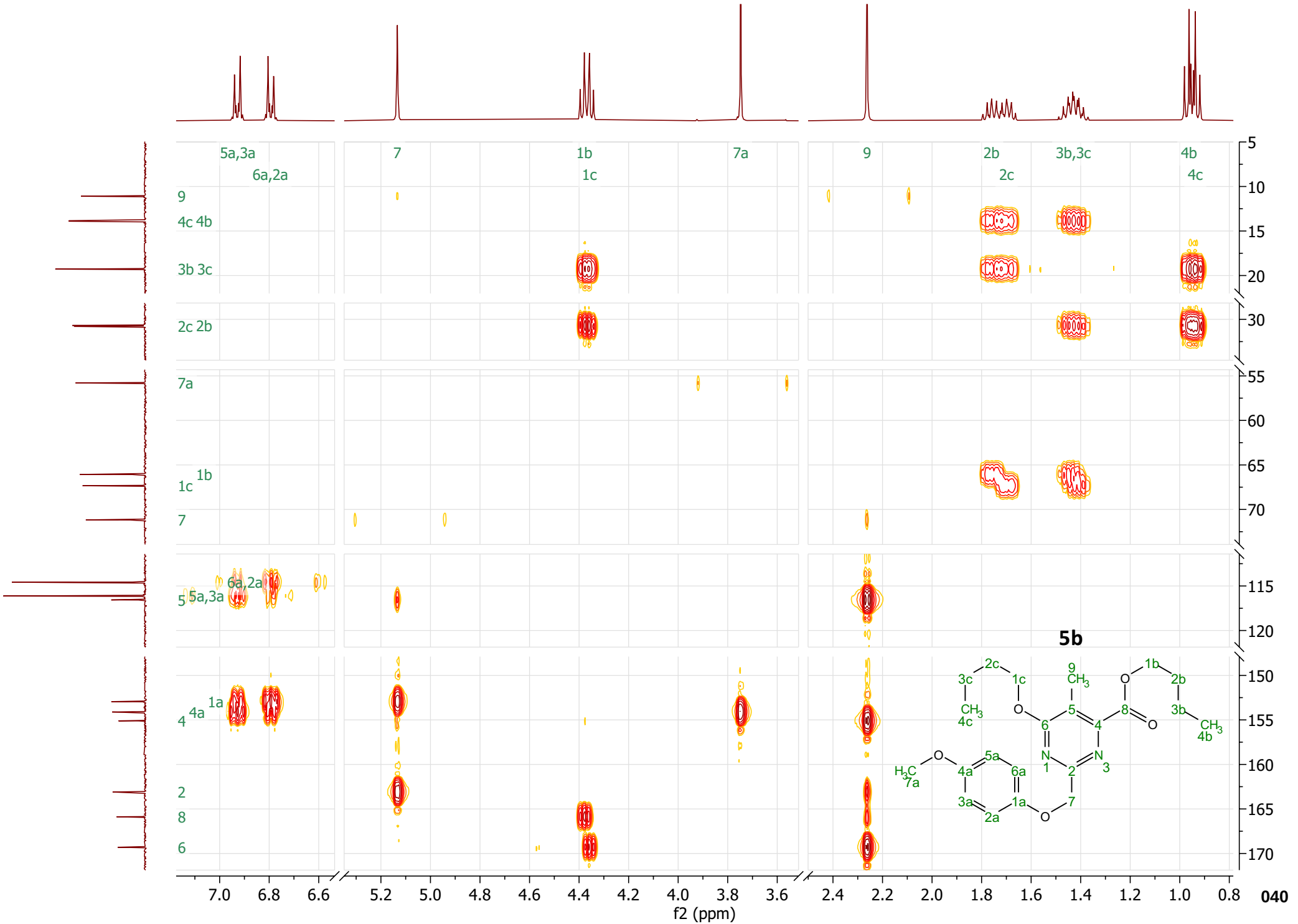
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 165.9, 163.1, 155.1, 154.1, 152.9, 116.6, 116.1 (sym, 2C), 114.6 (sym, 2C), 71.2, 67.3, 66.1, 55.8, 30.8, 30.7, 19.28, 19.27, 13.9, 13.8, 11.1.



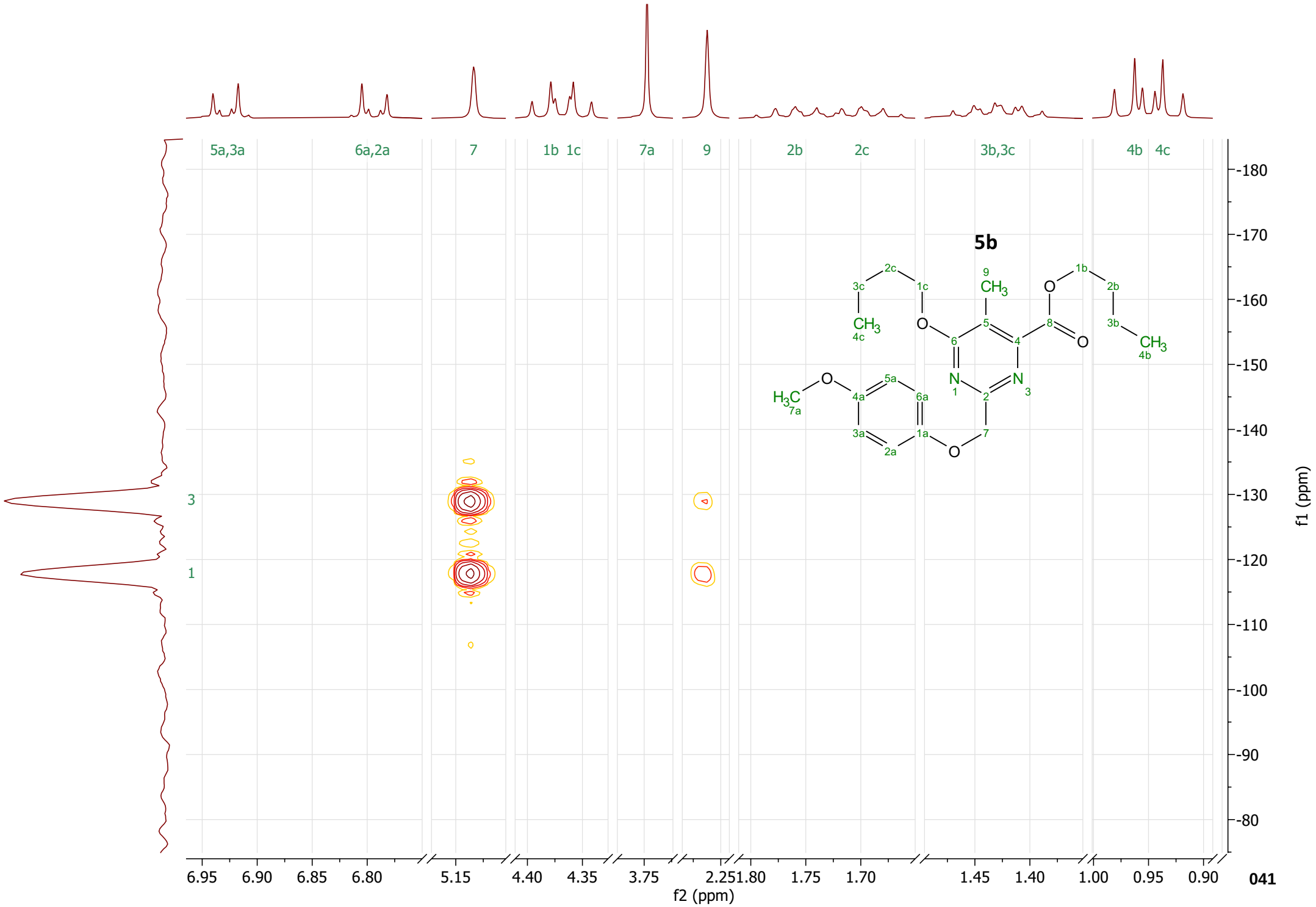
13C HSQC


5a,3a
6a,2a
7
1b
1c
7a
9
2b
2c
3b,3c
4b
4c
9
4c 4b
3b 3c
2c 2b
7a
1b
1c
7
6a,2a
5 5a,3a
5b
f2 (ppm)
f1 (ppm)
039

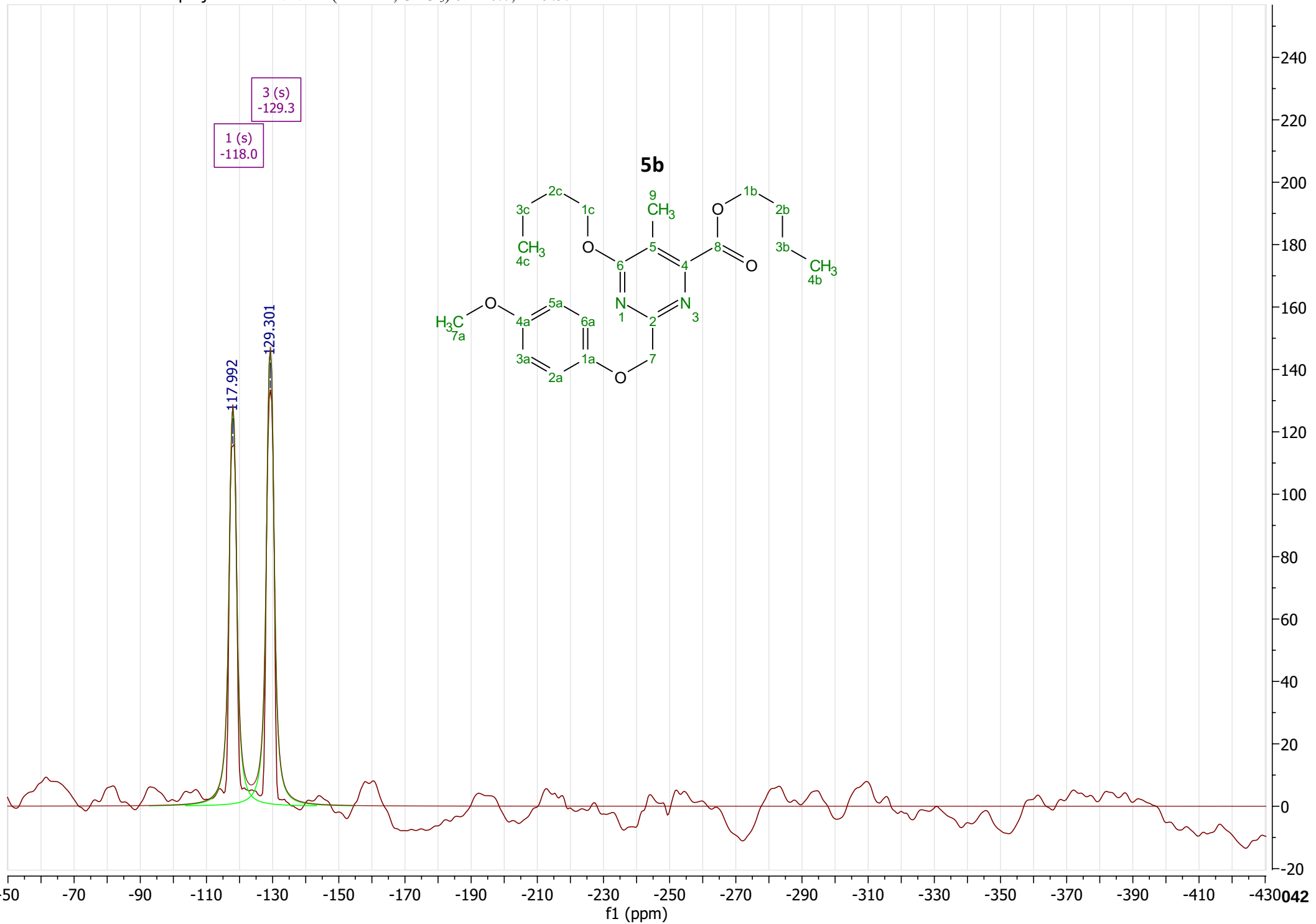
13C HMBC


5a,3a
6a,2a
7
1b
1c
7a
9
2b
2c
3b,3c
4b
4c
9
4c 4b
3b 3c
2c 2b
7a
1c 1b
7
5
5a,3a
6a,2a
4
4a
1a
2
8
6
5b
f1 (ppm)
f2 (ppm)
040

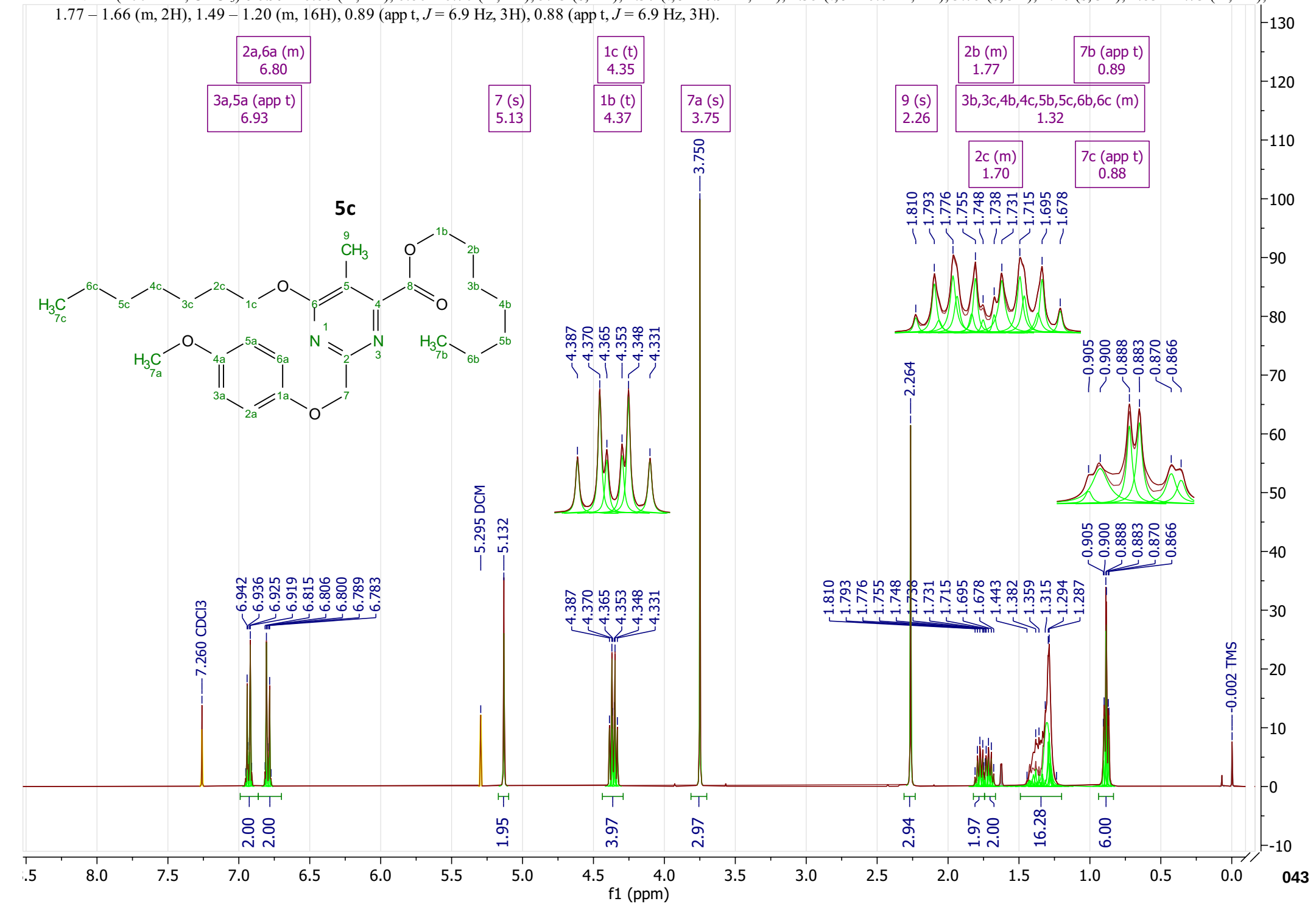
15N HMBC


5a,3a
6a,2a
7
1b 1c
7a
9
2b
2c
3b,3c
4b 4c
5b
3
1
-180
-170
-160
-150
-140
-130
-120
-110
-100
-90
-80
f1 (ppm)
6.95
6.90
6.85
6.80
5.15
4.40
4.35
3.75
2.25
1.80
1.75
1.70
1.45
1.40
1.00
0.95
0.90
f2 (ppm)
041

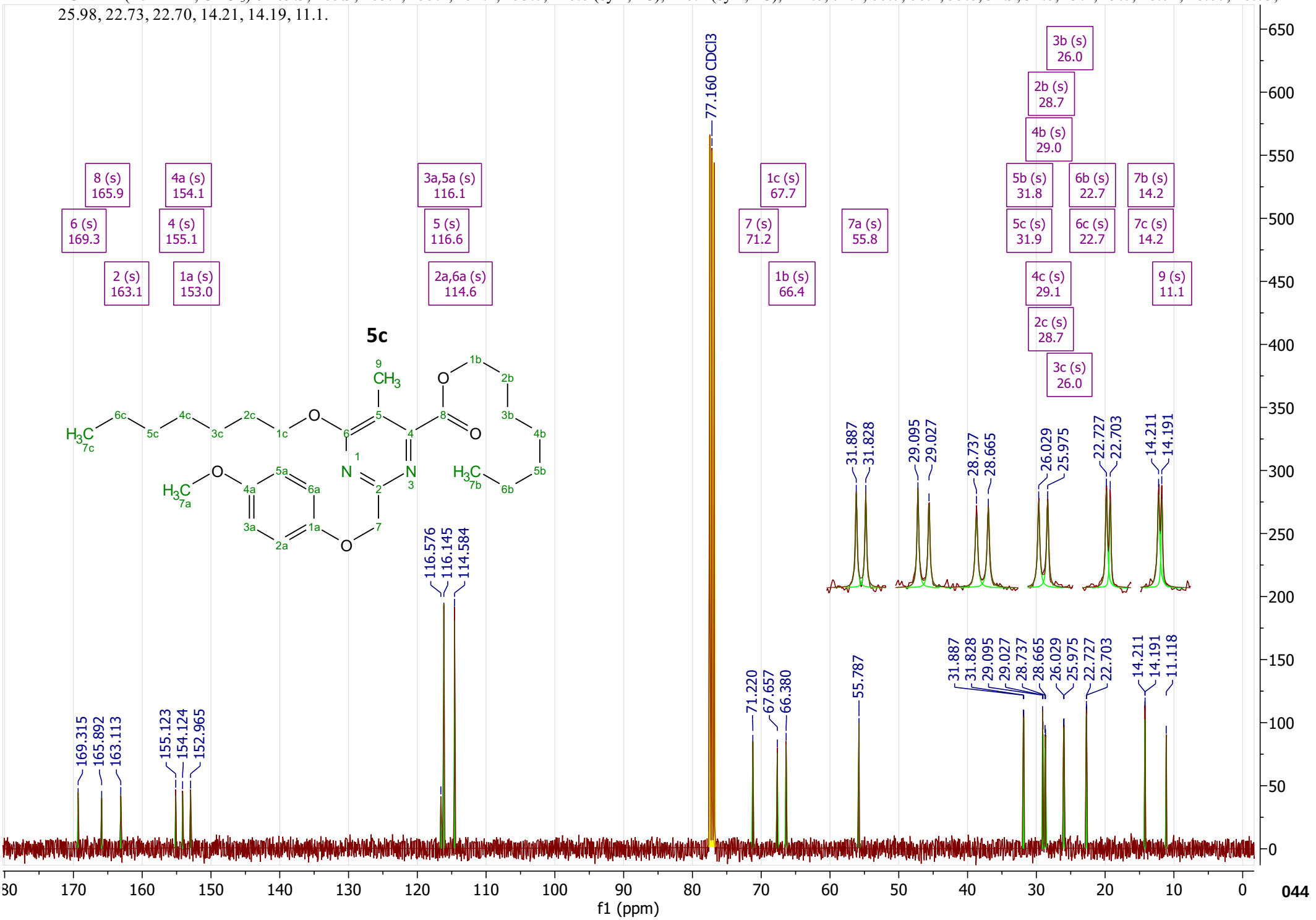
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -118.0, -129.3.



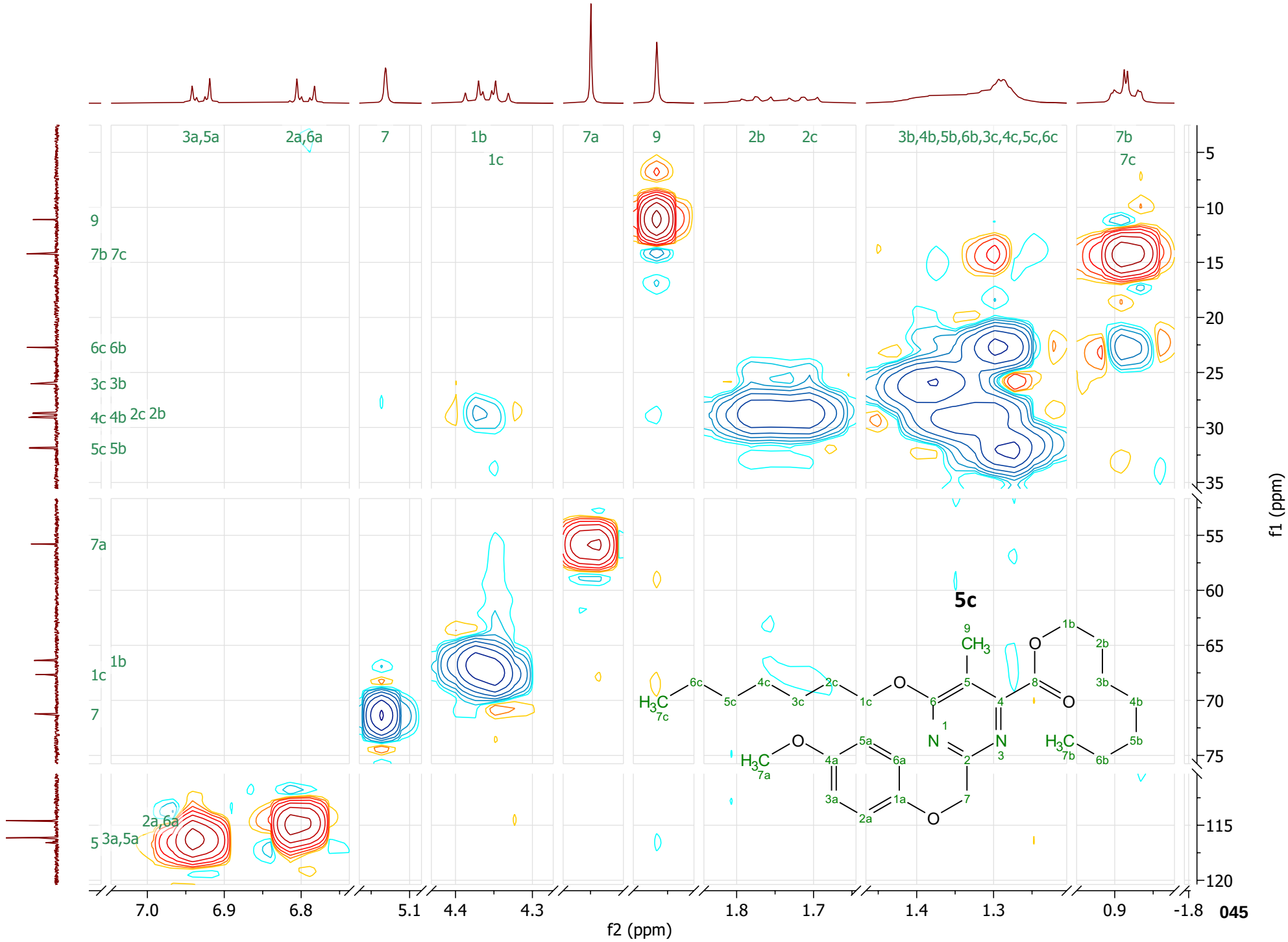
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 – 6.86 (m, 2H), 6.86 – 6.70 (m, 2H), 5.13 (s, 2H), 4.37 (t, *J* = 6.9 Hz, 2H), 4.35 (t, *J* = 6.6 Hz, 2H), 3.75 (s, 3H), 2.26 (s, 3H), 1.83 – 1.73 (m, 2H), 1.77 – 1.66 (m, 2H), 1.49 – 1.20 (m, 16H), 0.89 (app t, *J* = 6.9 Hz, 3H), 0.88 (app t, *J* = 6.9 Hz, 3H).



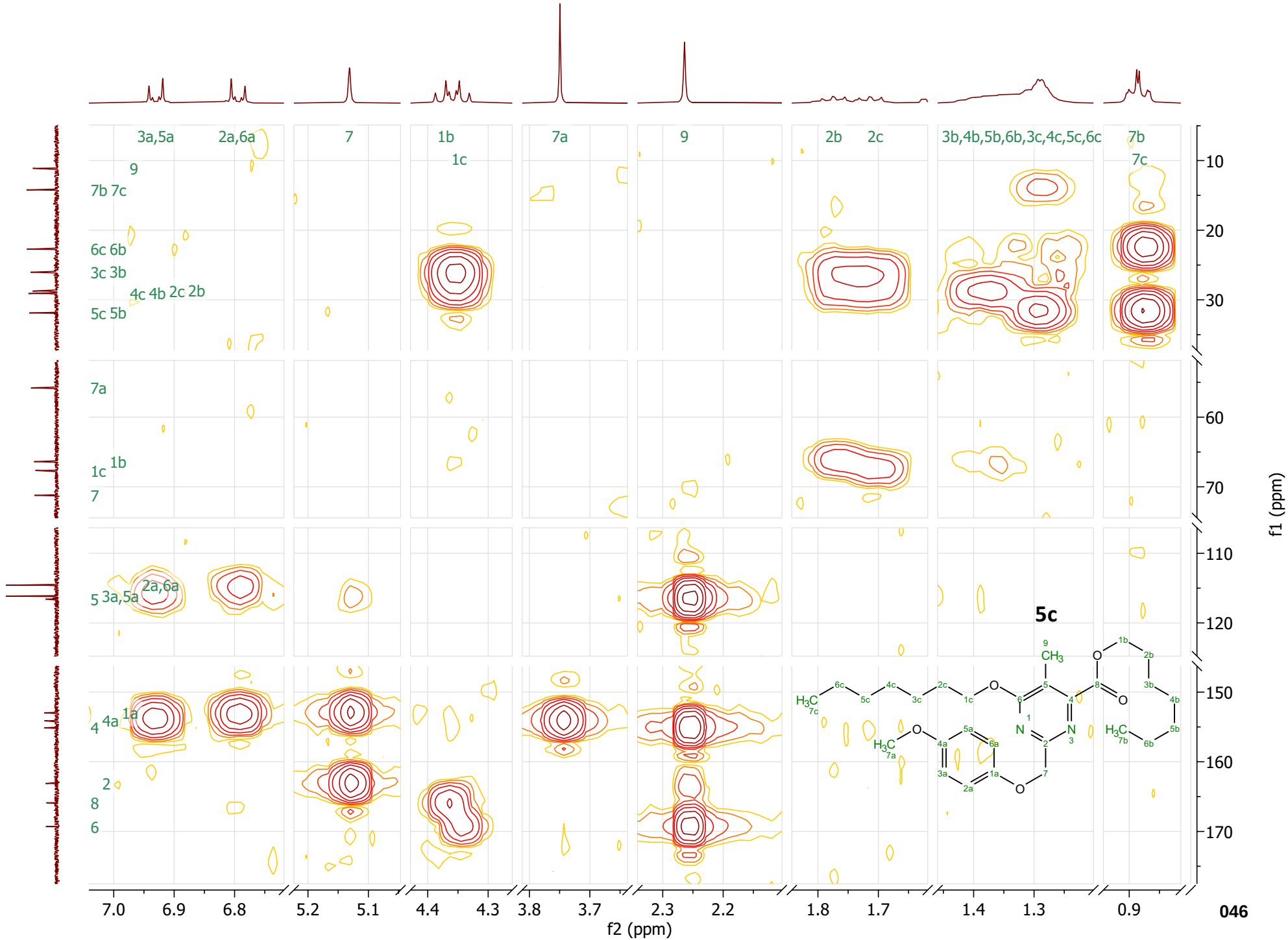
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 165.9, 163.1, 155.1, 154.1, 153.0, 116.6 (sym, 2C), 116.1 (sym, 2C), 114.6, 71.2, 67.7, 66.4, 55.8, 31.9, 31.8, 29.1, 29.0, 28.74, 28.67, 26.03, 25.98, 22.73, 22.70, 14.21, 14.19, 11.1.



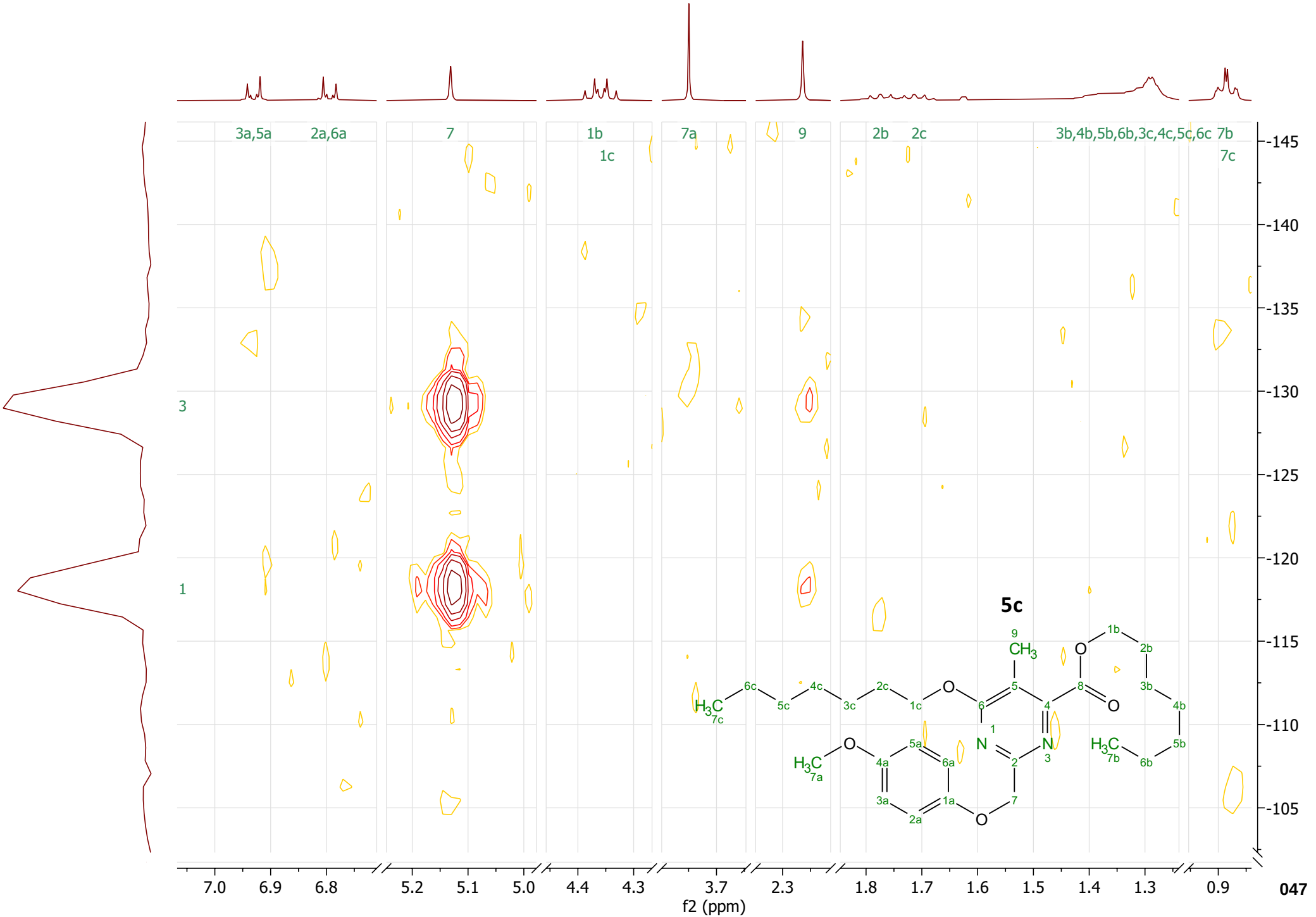
13C HSQC


3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,4b,5b,6b,3c,4c,5c,6c
7b
7c
6c 6b
3c 3b
4c 4b 2c 2b
5c 5b
1c
1b
5c
f2 (ppm)
f1 (ppm)
045

13C HMBC

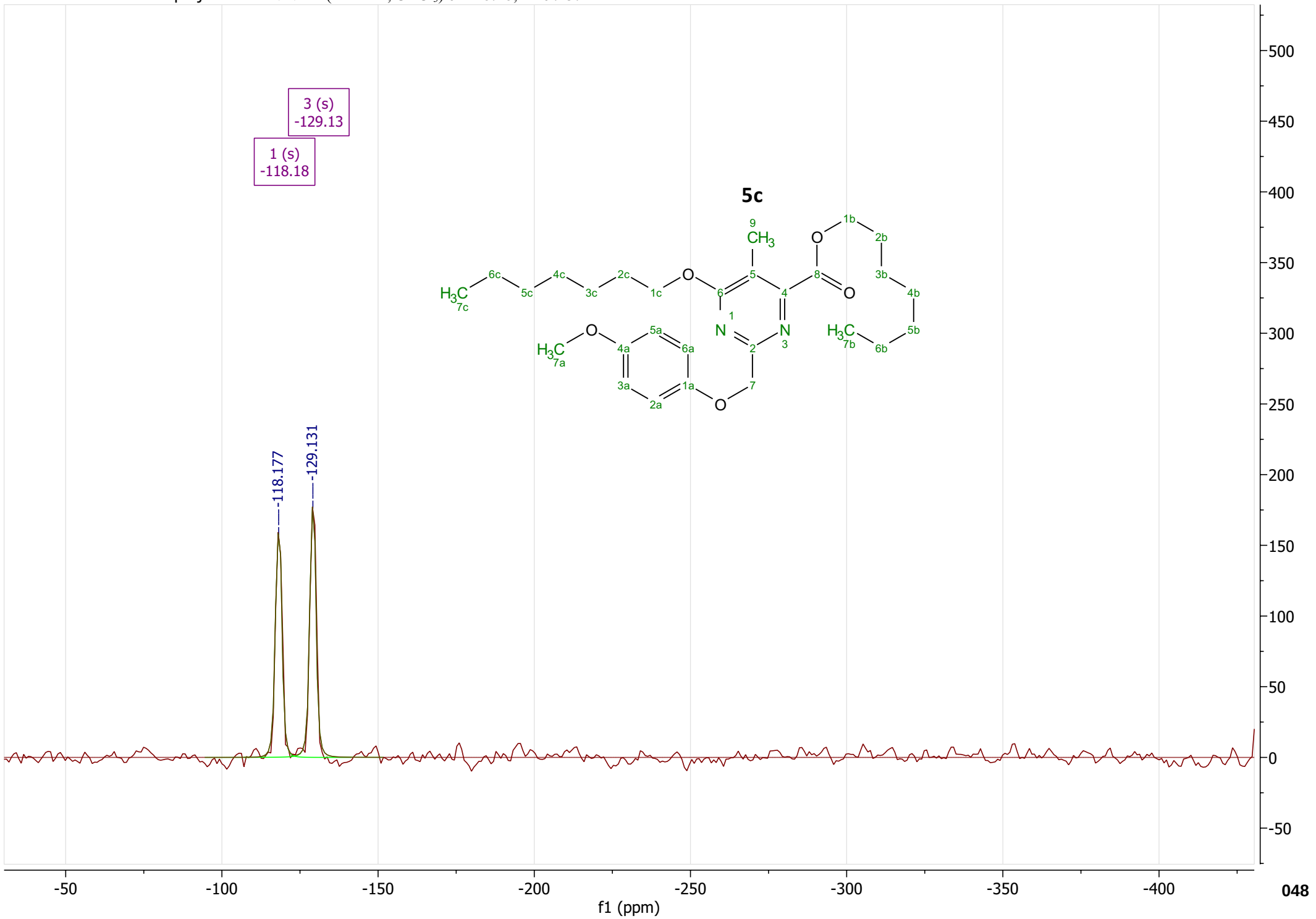

3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,4b,5b,6b,3c,4c,5c,6c
7b
7c
9
7b 7c
6c 6b
3c 3b
4c 4b 2c 2b
5c 5b
7a
1c 1b
7
5 3a,5a 2a,6a
4 4a 1a
2
8
6
5c
f1 (ppm)
f2 (ppm)
7.0
6.9
6.8
5.2
5.1
4.4
4.3
3.8
3.7
2.3
2.2
1.8
1.7
1.4
1.3
0.9
10
20
30
60
70
110
120
150
160
170
046

15N HMBC


3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,4b,5b,6b,3c,4c,5c,6c
7b
7c
3
1
5c
f1 (ppm)
f2 (ppm)
-145
-140
-135
-130
-125
-120
-115
-110
-105
7.0
6.9
6.8
5.2
5.1
5.0
4.4
4.3
3.7
2.3
1.8
1.7
1.6
1.5
1.4
1.3
0.9

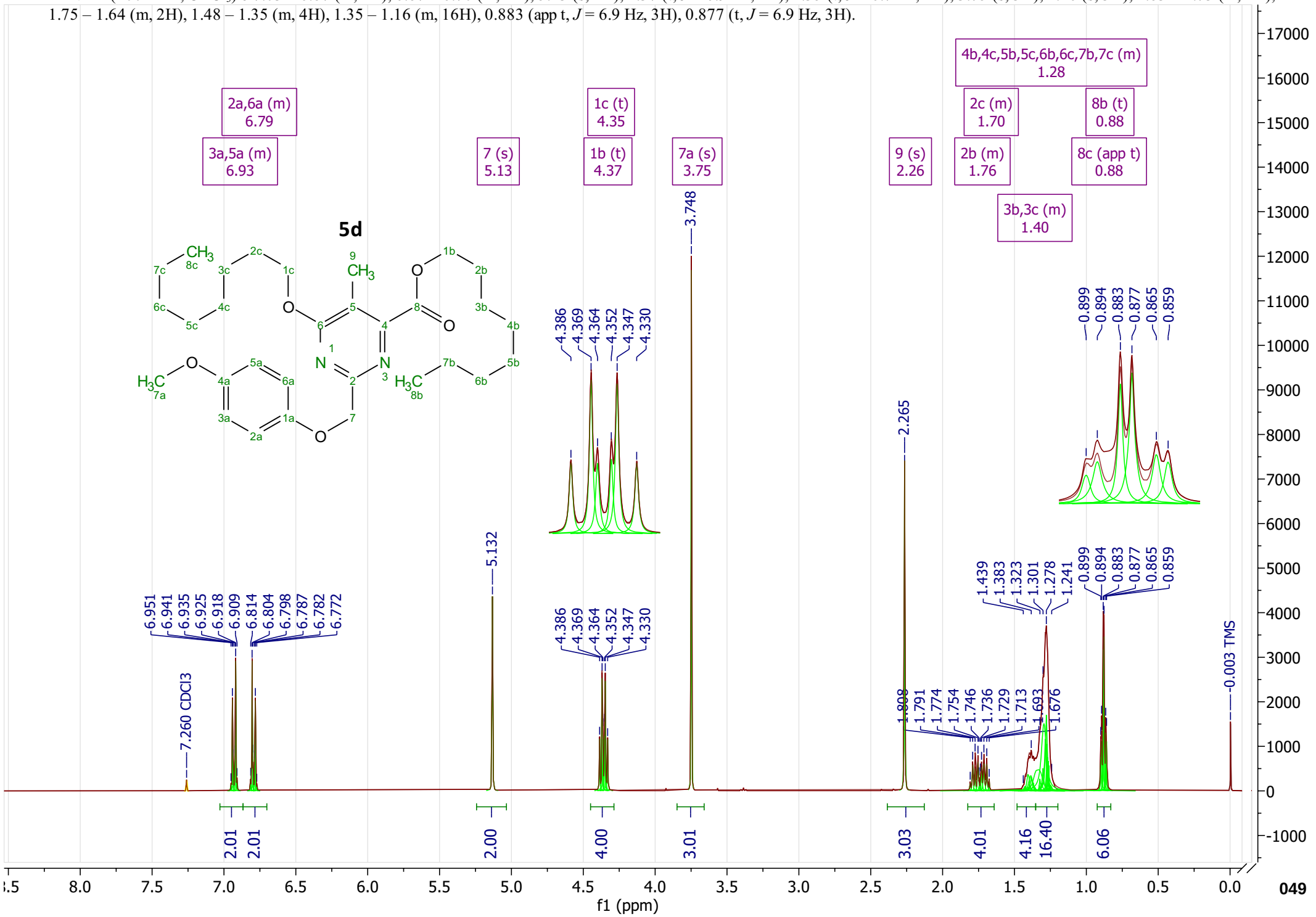
047

15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -118.18, -129.13.

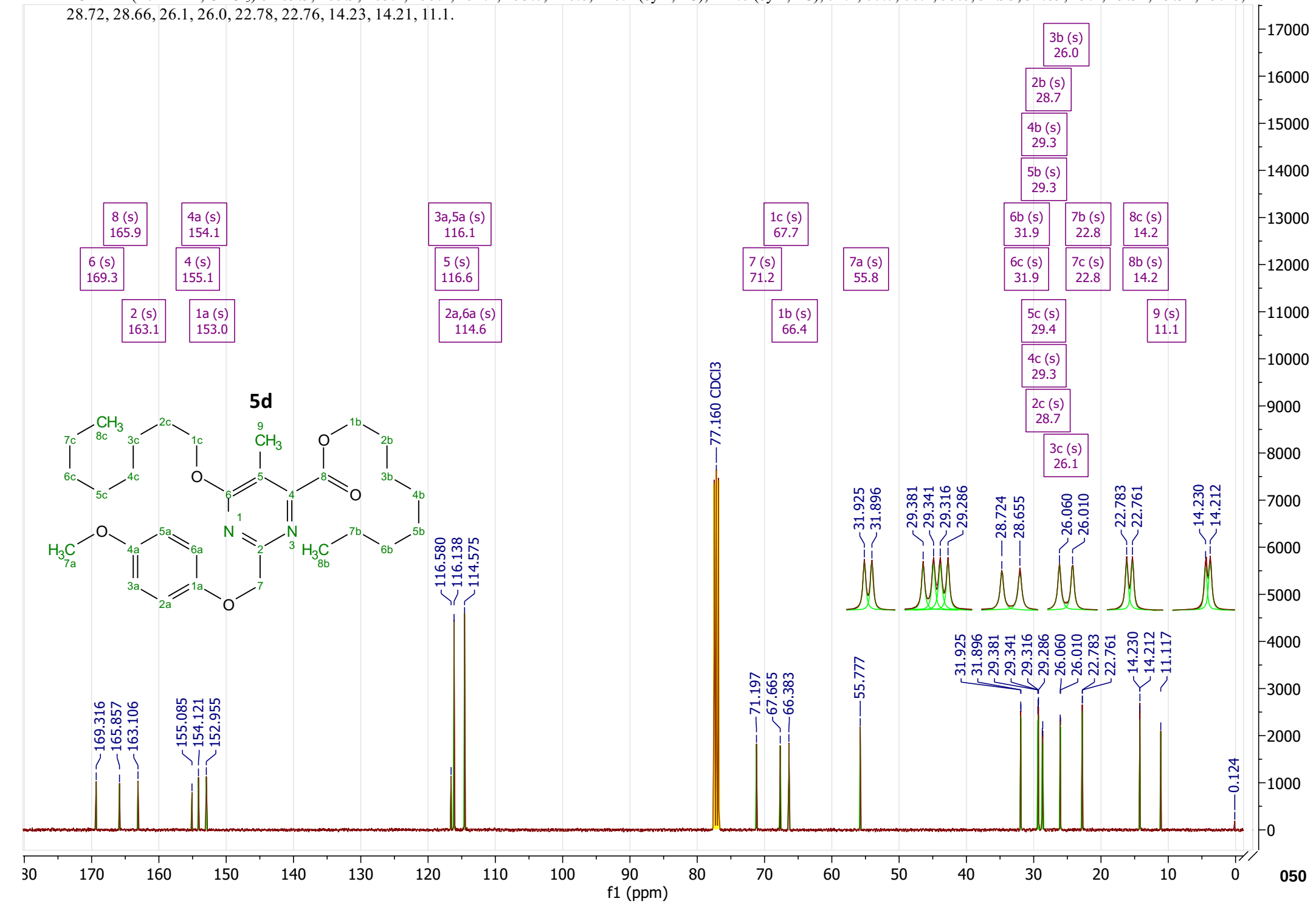


048

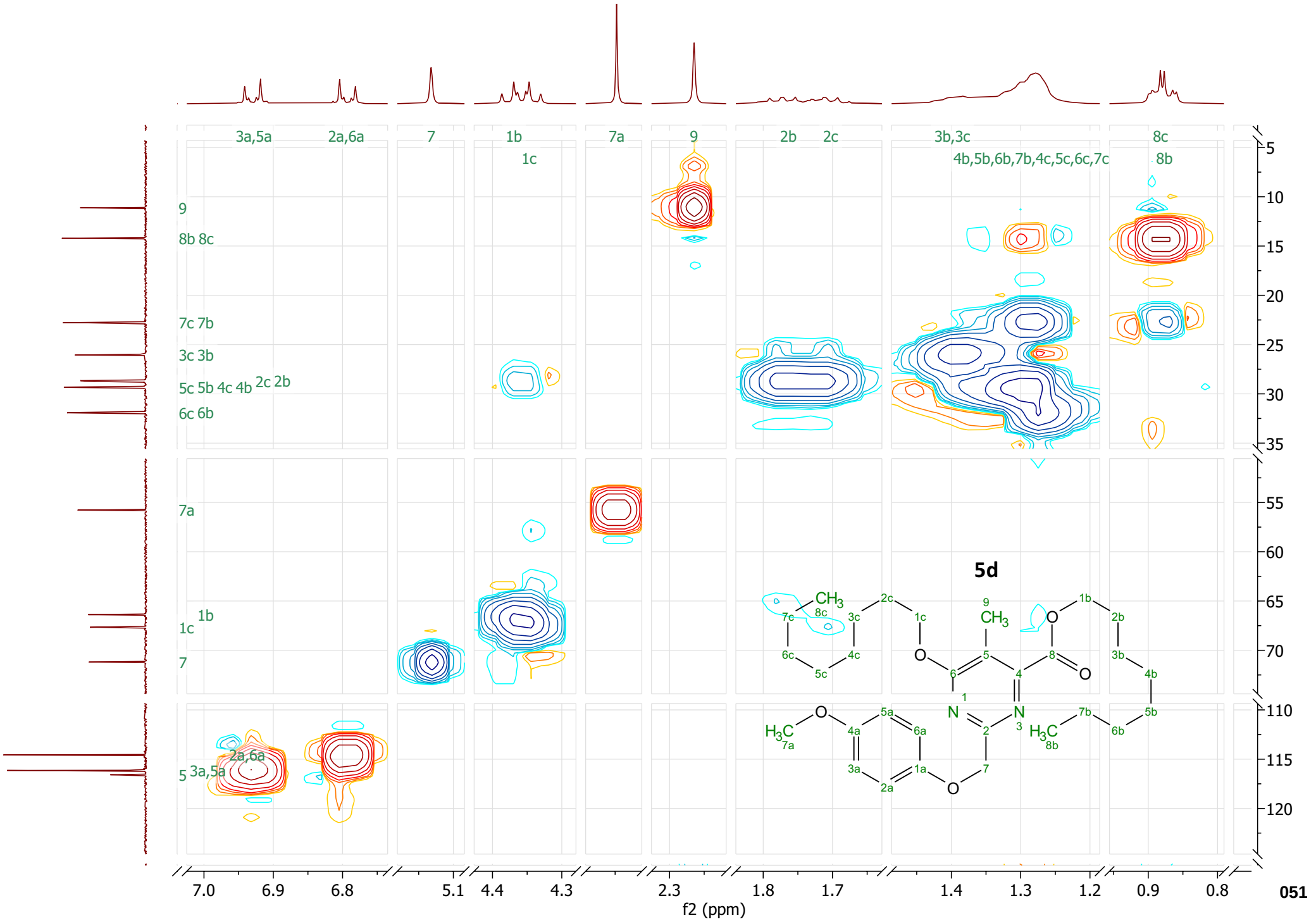
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 – 6.87 (m, 2H), 6.87 – 6.70 (m, 2H), 5.13 (s, 2H), 4.37 (t, *J* = 6.9 Hz, 2H), 4.35 (t, *J* = 6.7 Hz, 2H), 3.75 (s, 3H), 2.26 (s, 3H), 1.83 – 1.73 (m, 2H), 1.75 – 1.64 (m, 2H), 1.48 – 1.35 (m, 4H), 1.35 – 1.16 (m, 16H), 0.883 (app t, *J* = 6.9 Hz, 3H), 0.877 (t, *J* = 6.9 Hz, 3H).



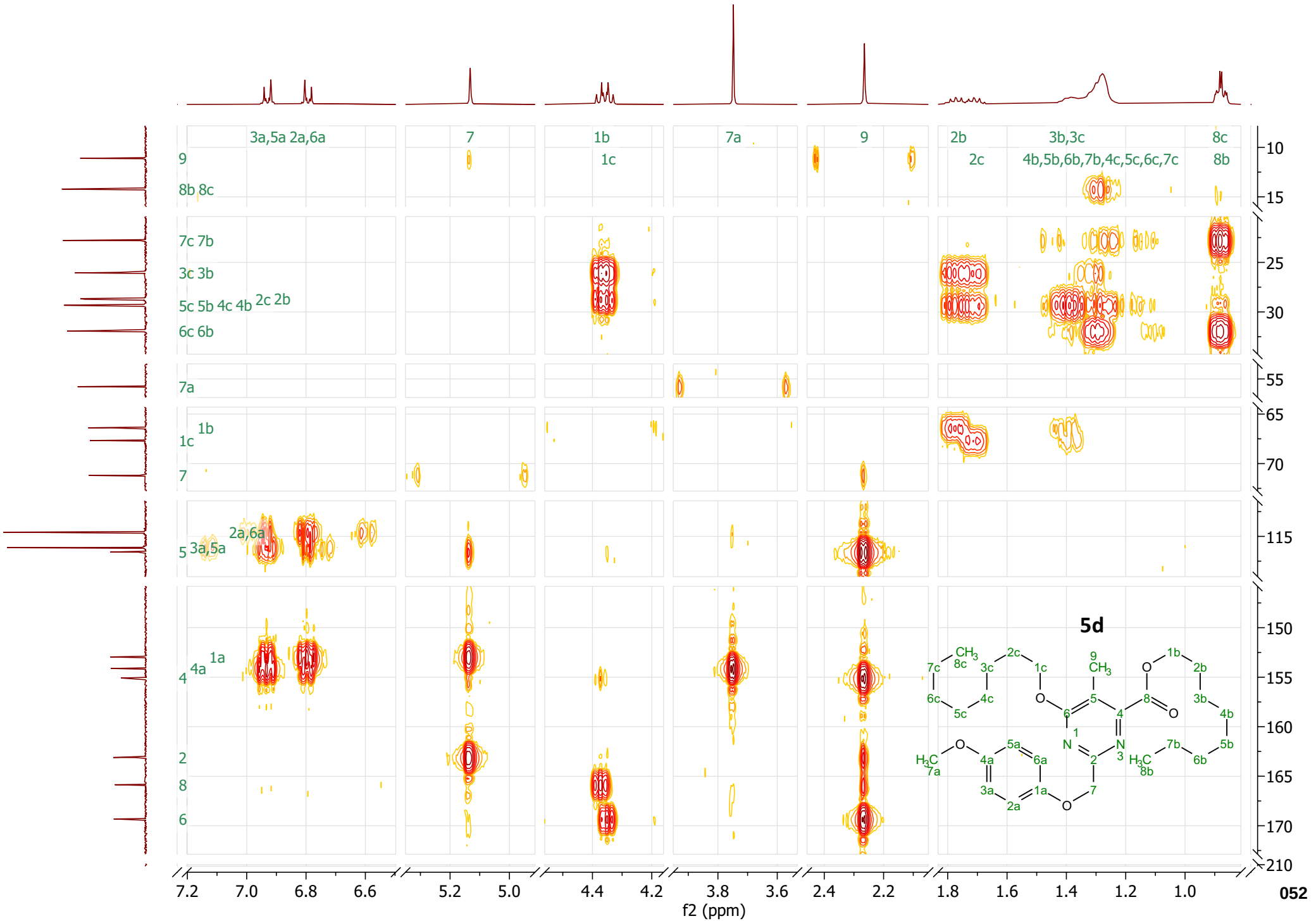
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 165.9, 163.1, 155.1, 154.1, 153.0, 116.6, 116.1 (sym, 2C), 114.6 (sym, 2C), 71.2, 67.7, 66.4, 55.8, 31.93, 31.89, 29.4, 29.34, 29.31, 29.28, 28.72, 28.66, 26.1, 26.0, 22.78, 22.76, 14.23, 14.21, 11.1.



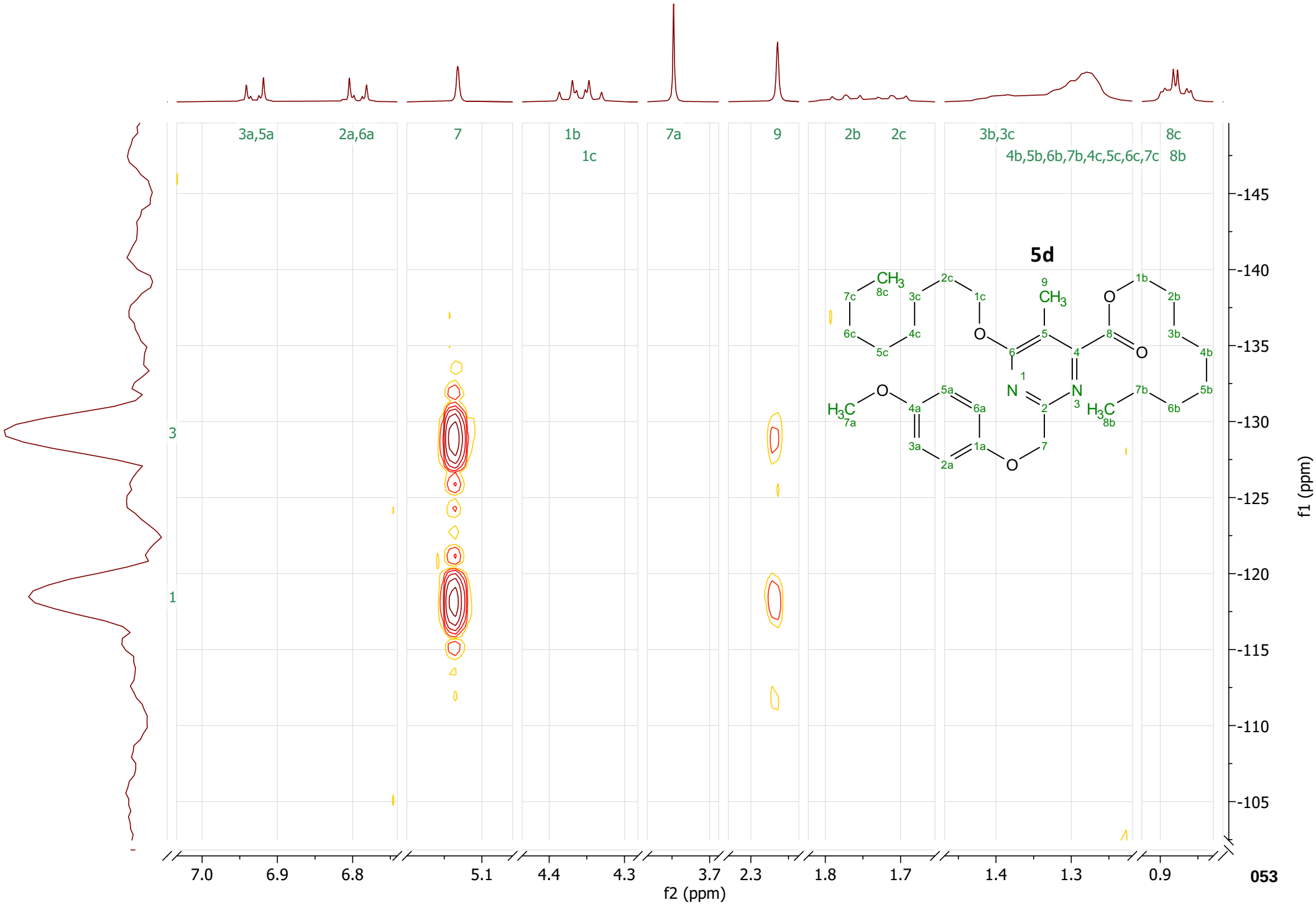
13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
2c
3b,3c
8c
1c
4b,5b,6b,7b,4c,5c,6c,7c
8b
9
8b 8c
7c 7b
3c 3b
5c 5b 4c 4b 2c 2b
6c 6b
7a
1b
1c
7
5
3a,5a
2a,6a
5d
f1 (ppm)
f2 (ppm)
7.0
6.9
6.8
5.1
4.4
4.3
2.3
1.8
1.7
1.4
1.3
1.2
0.9
0.8
051

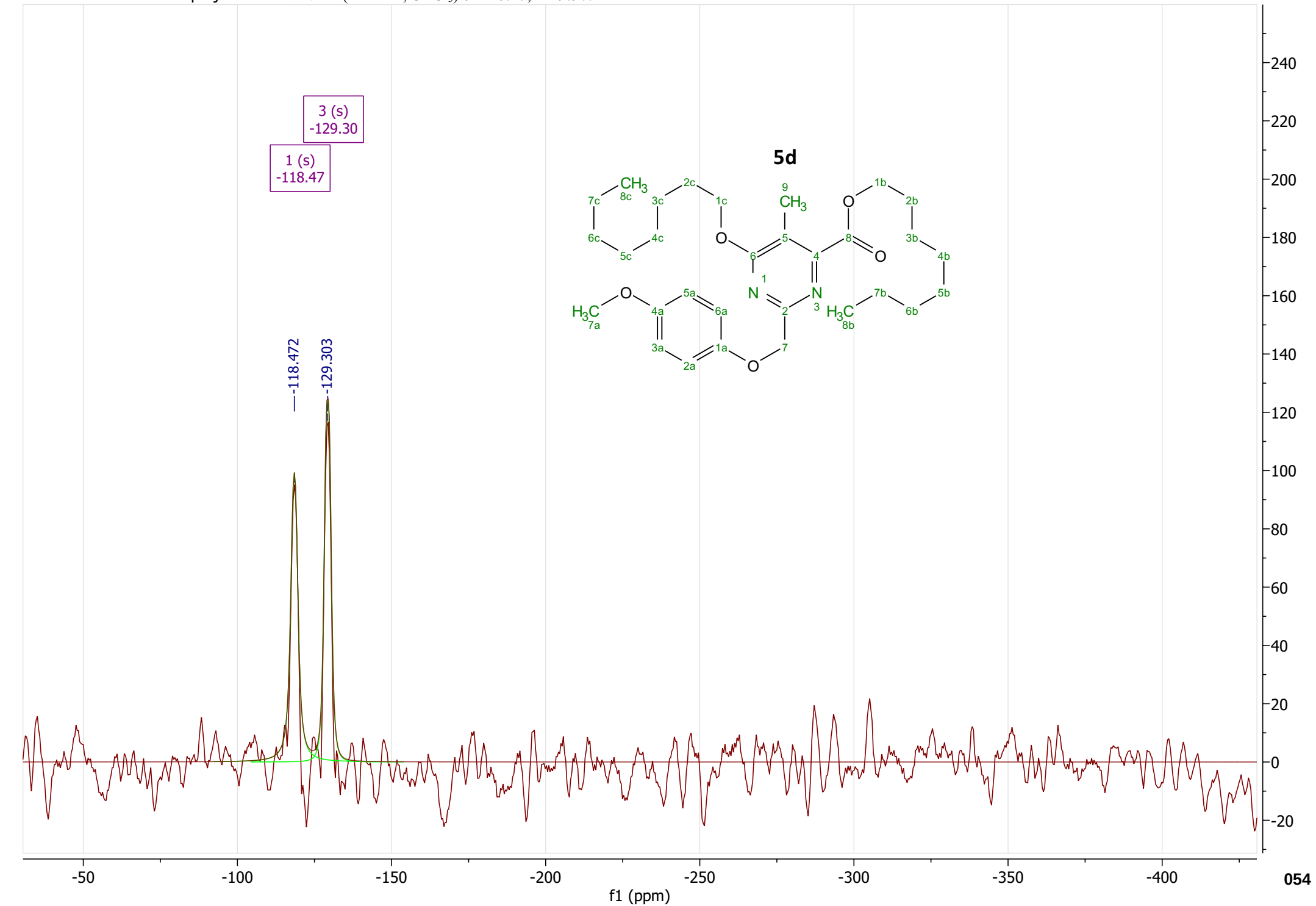
13C HMBC


3a,5a 2a,6a
7
1b
7a
9
2b
3b,3c
8c
1c
4b,5b,6b,7b,4c,5c,6c,7c
2c
8b
7c 7b
3c 3b
5c 5b 4c 4b 2c 2b
6c 6b
1b
2a,6a
3a,5a
1a
4a
5d
f2 (ppm)
f1 (ppm)
052

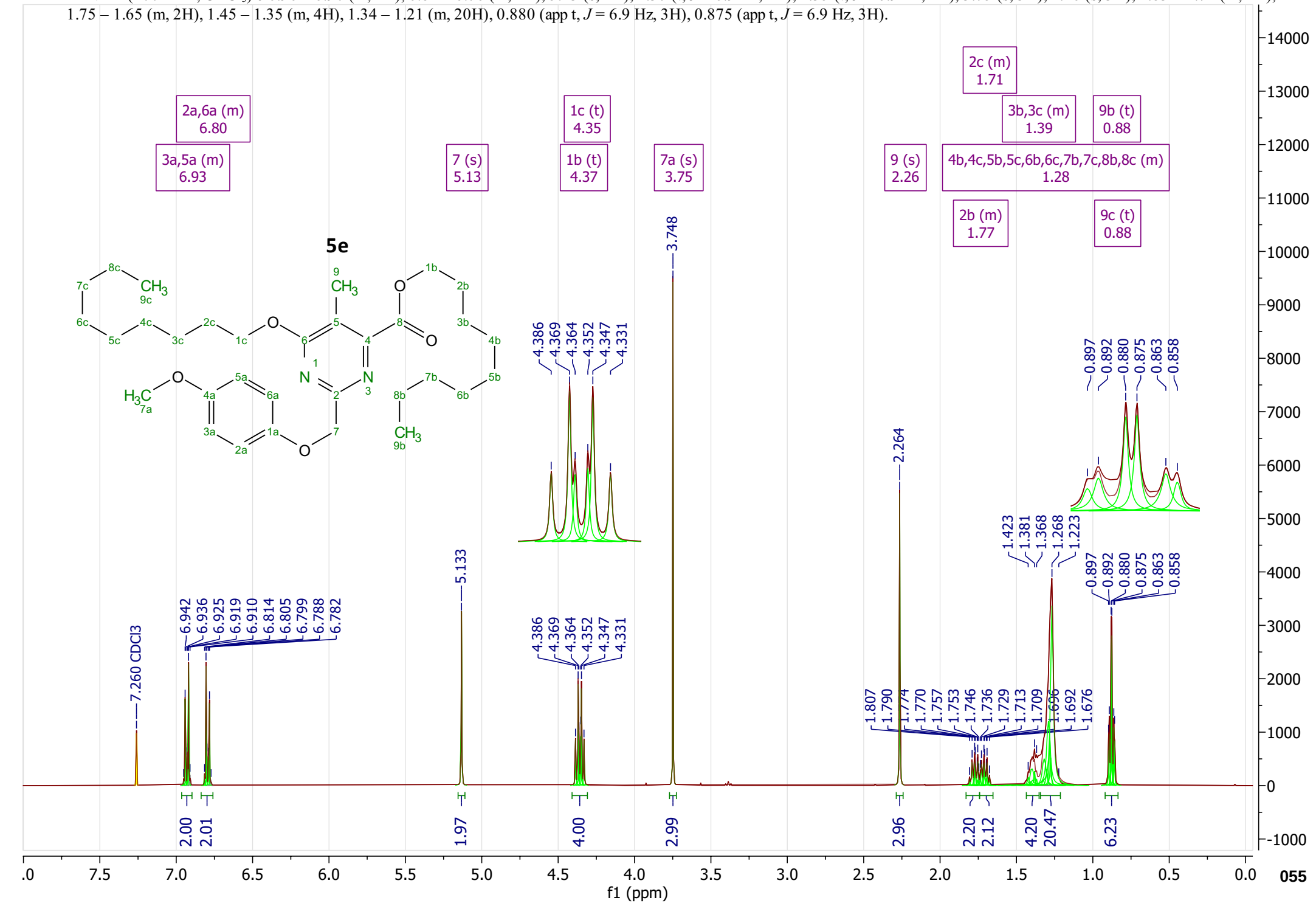
15N HMBC


3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,3c
4b,5b,6b,7b,4c,5c,6c,7c
8c
8b
5d
f2 (ppm)
f1 (ppm)
053

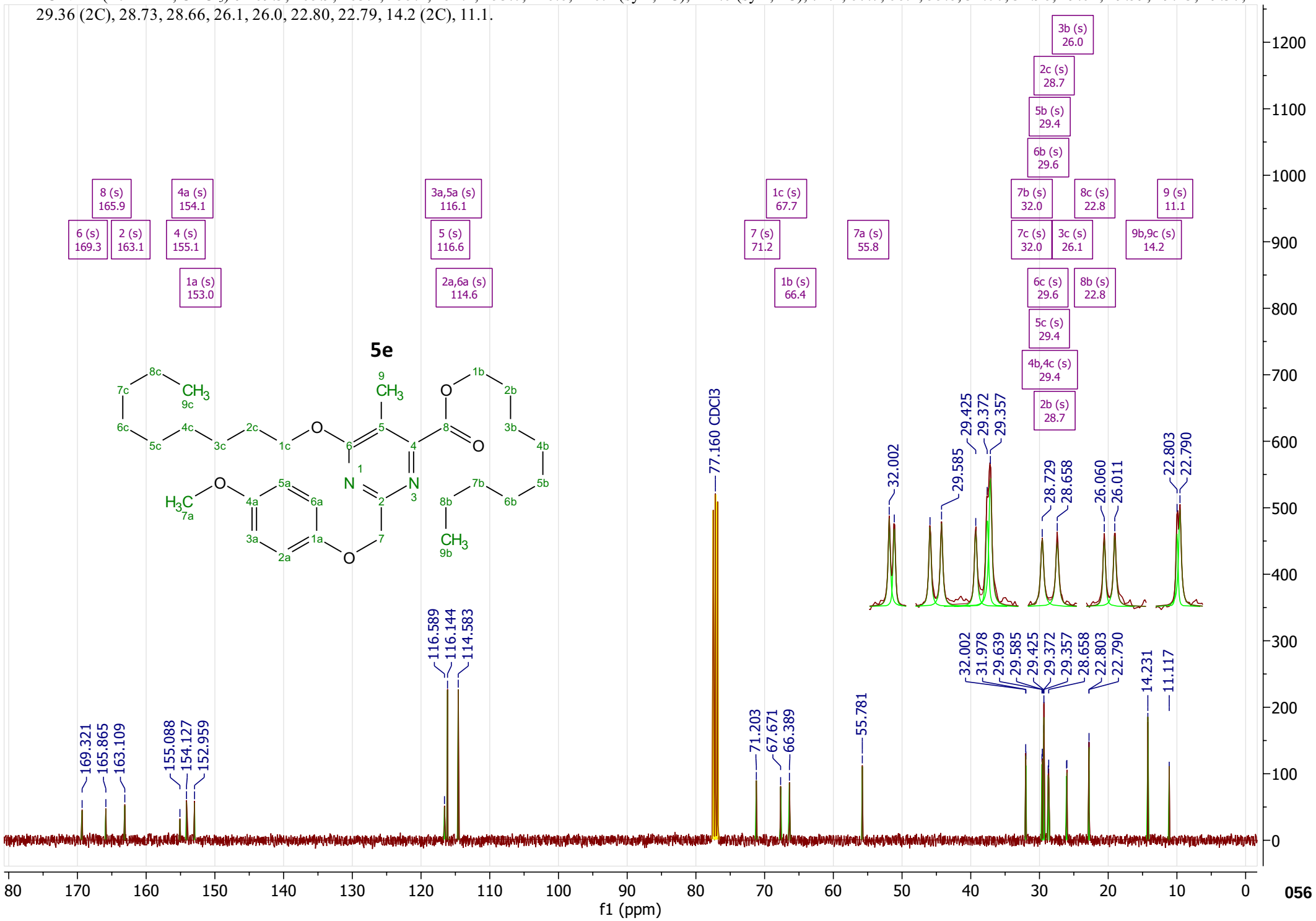
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -118.47, -129.30.



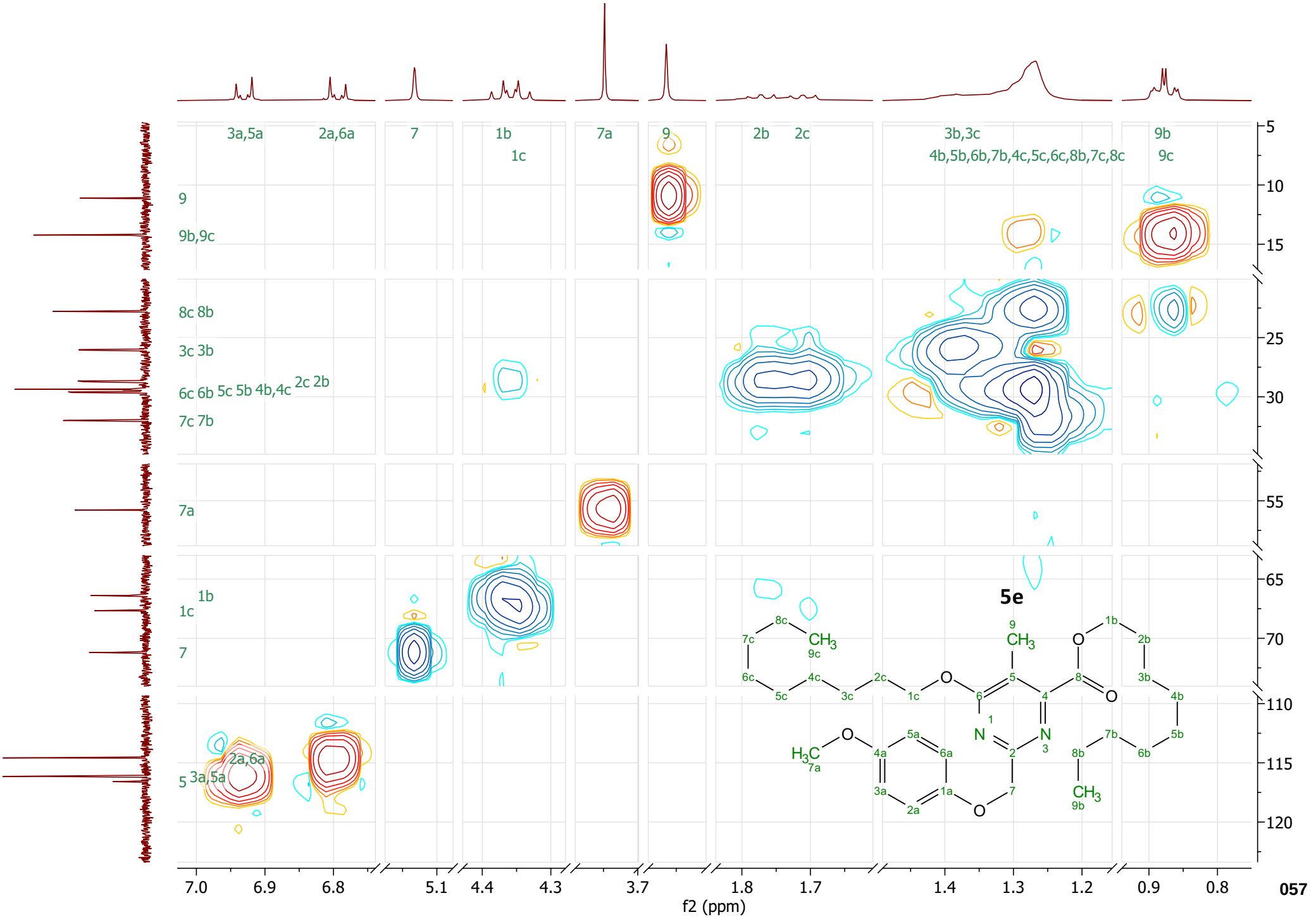
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 – 6.90 (m, 2H), 6.84 – 6.76 (m, 2H), 5.13 (s, 2H), 4.37 (t, *J* = 6.9 Hz, 2H), 4.35 (t, *J* = 6.9 Hz, 2H), 3.75 (s, 3H), 2.26 (s, 3H), 1.83 – 1.71 (m, 2H), 1.75 – 1.65 (m, 2H), 1.45 – 1.35 (m, 4H), 1.34 – 1.21 (m, 20H), 0.880 (app t, *J* = 6.9 Hz, 3H), 0.875 (app t, *J* = 6.9 Hz, 3H).



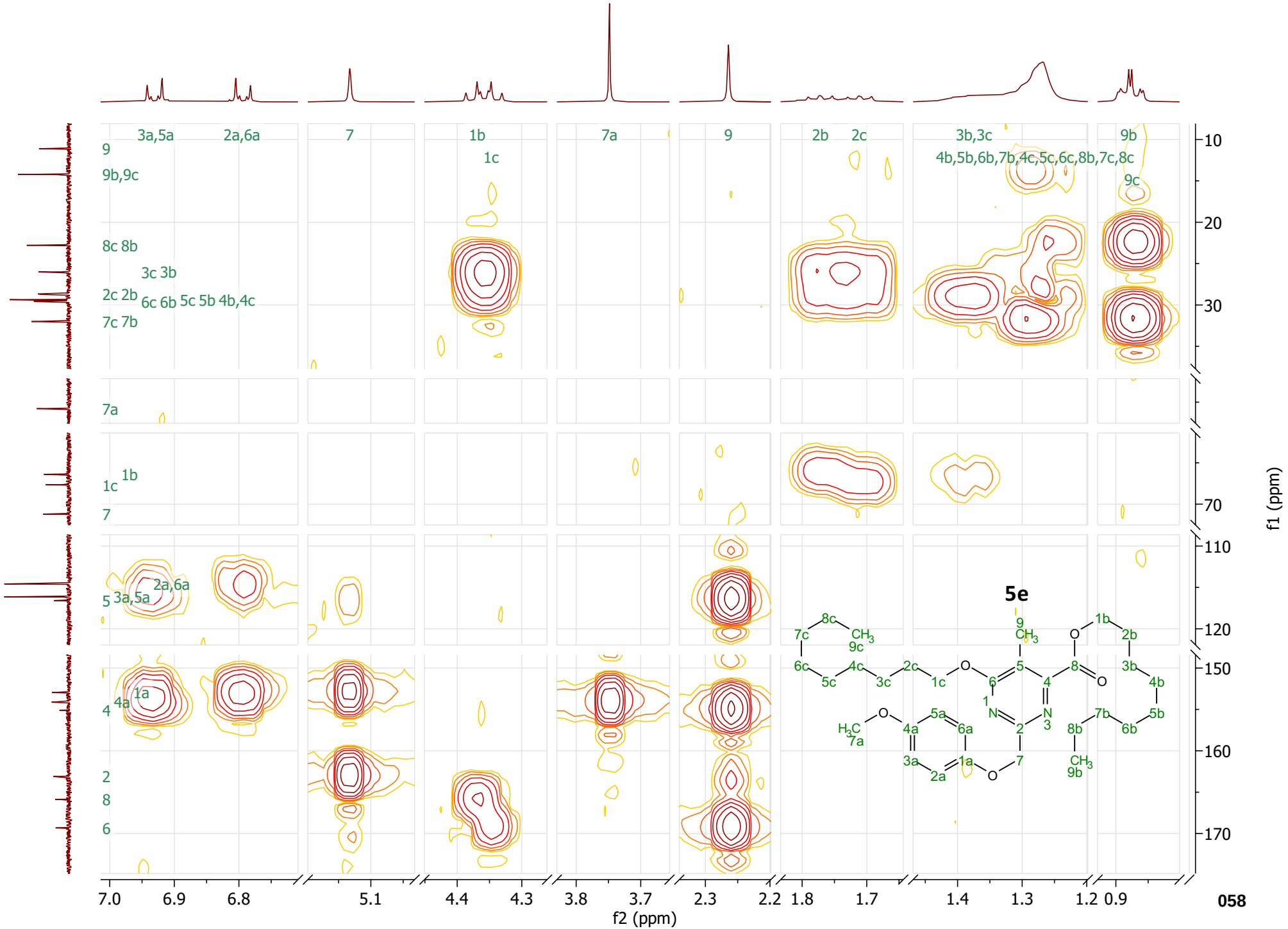
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.3, 165.9, 163.1, 155.1, 154.1, 153.0, 116.6, 116.1 (sym, 2C), 114.6 (sym, 2C), 71.2, 67.7, 66.4, 55.8, 32.00, 31.98, 29.64, 29.59, 29.43, 29.37, 29.36 (2C), 28.73, 28.66, 26.1, 26.0, 22.80, 22.79, 14.2 (2C), 11.1.



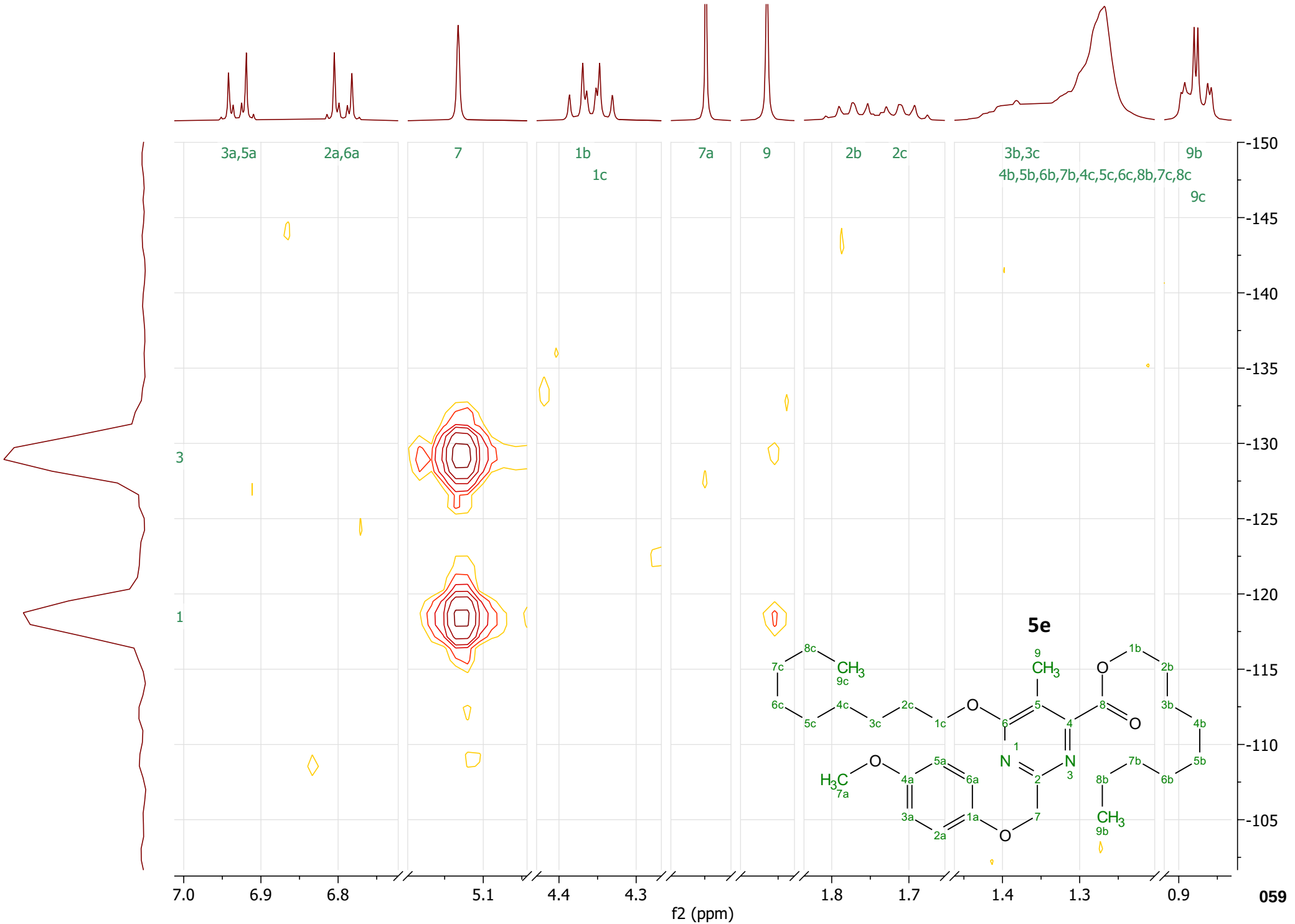
13C HSQC


3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,3c
4b,5b,6b,7b,4c,5c,6c,8b,7c,8c
9b
9c
9
9b,9c
8c 8b
3c 3b
6c 6b 5c 5b 4b,4c 2c 2b
7c 7b
7a
1b
1c
7
5
3a,5a
2a,6a
5e
f1 (ppm)
f2 (ppm)
7.0
6.9
6.8
5.1
4.4
4.3
3.7
1.8
1.7
1.4
1.3
1.2
0.9
0.8
5
10
15
25
30
55
65
70
110
115
120
057

13C HMBC

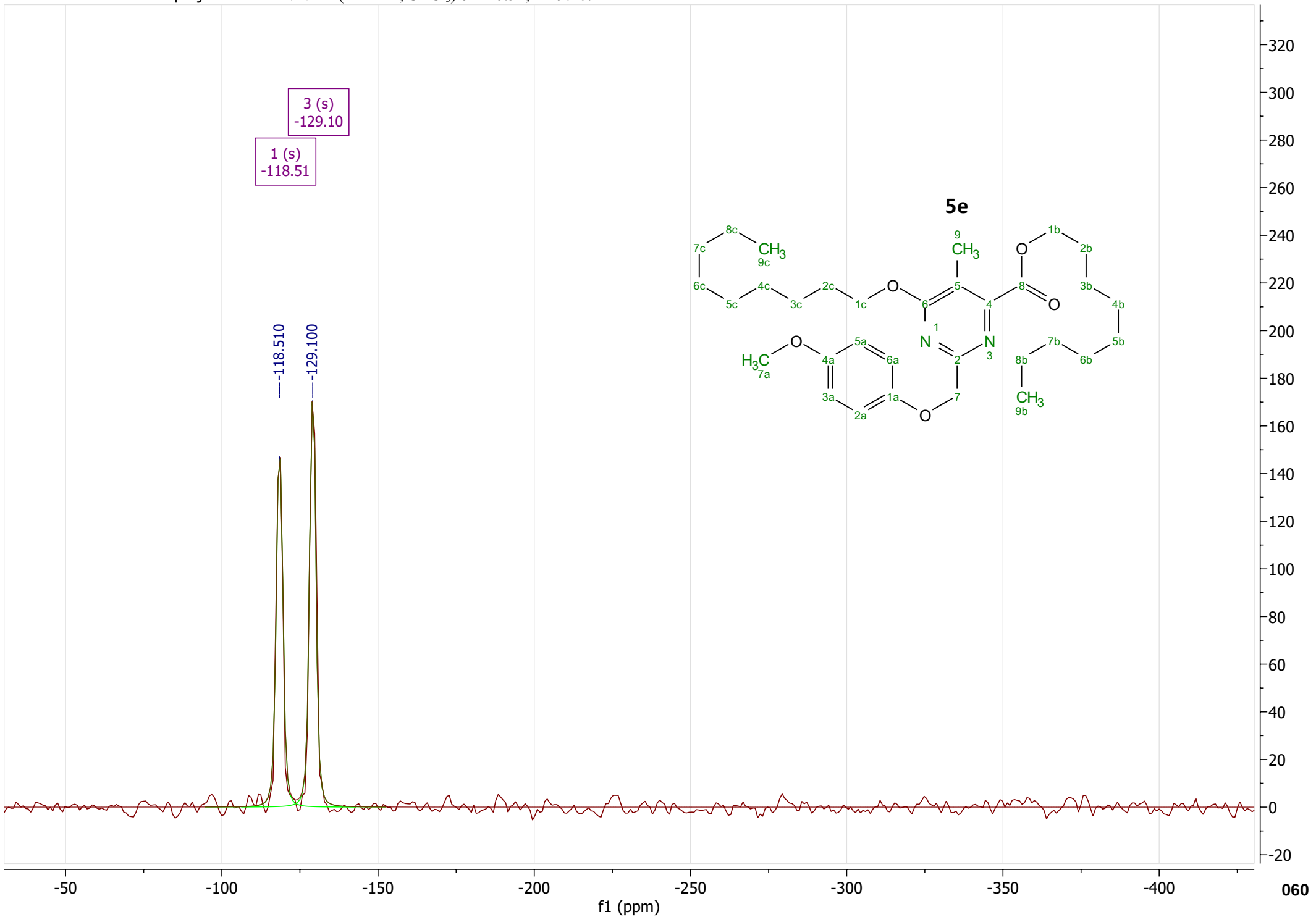

3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,3c
4b,5b,6b,7b,4c,5c,6c,8b,7c,8c
9b
9c
9
9b,9c
8c 8b
3c 3b
2c 2b
6c 6b 5c 5b 4b,4c
7c 7b
7a
1c
1b
7
5
3a,5a
2a,6a
4
4a
1a
2
8
6
5e
f1 (ppm)
f2 (ppm)
7.0
6.9
6.8
5.1
4.4
4.3
3.8
3.7
2.3
2.2
1.8
1.7
1.4
1.3
1.2
0.9
10
20
30
70
110
120
150
160
170
058

15N HMBC


3a,5a
2a,6a
7
1b
1c
7a
9
2b
2c
3b,3c
4b,5b,6b,7b,4c,5c,6c,8b,7c,8c
9b
9c
3
1
-150
-145
-140
-135
-130
-125
-120
-115
-110
-105
f1 (ppm)
7.0
6.9
6.8
5.1
4.4
4.3
1.8
1.7
1.4
1.3
0.9
f2 (ppm)
5e
CH3
9c
CH3
9
H3C
7a
CH3
9b
N
N
O
O
O
O
O
1
2
3
4
5
6
7
8
1a
1b
1c
2a
2b
2c
3a
3b
3c
4a
4b
4c
5a
5b
5c
6a
6b
6c
7b
7c
8b
8c

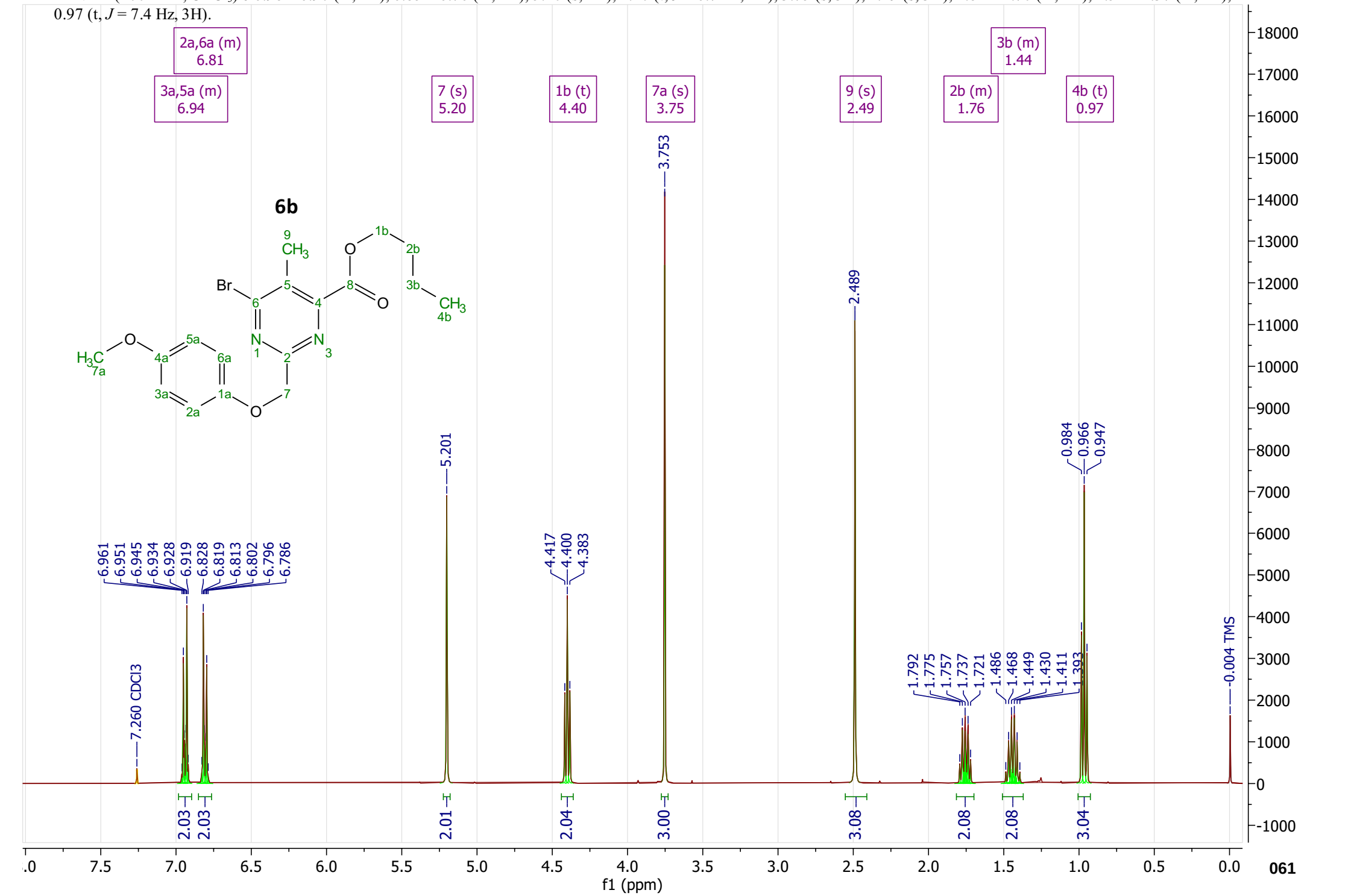
059

15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -118.51, -129.10.

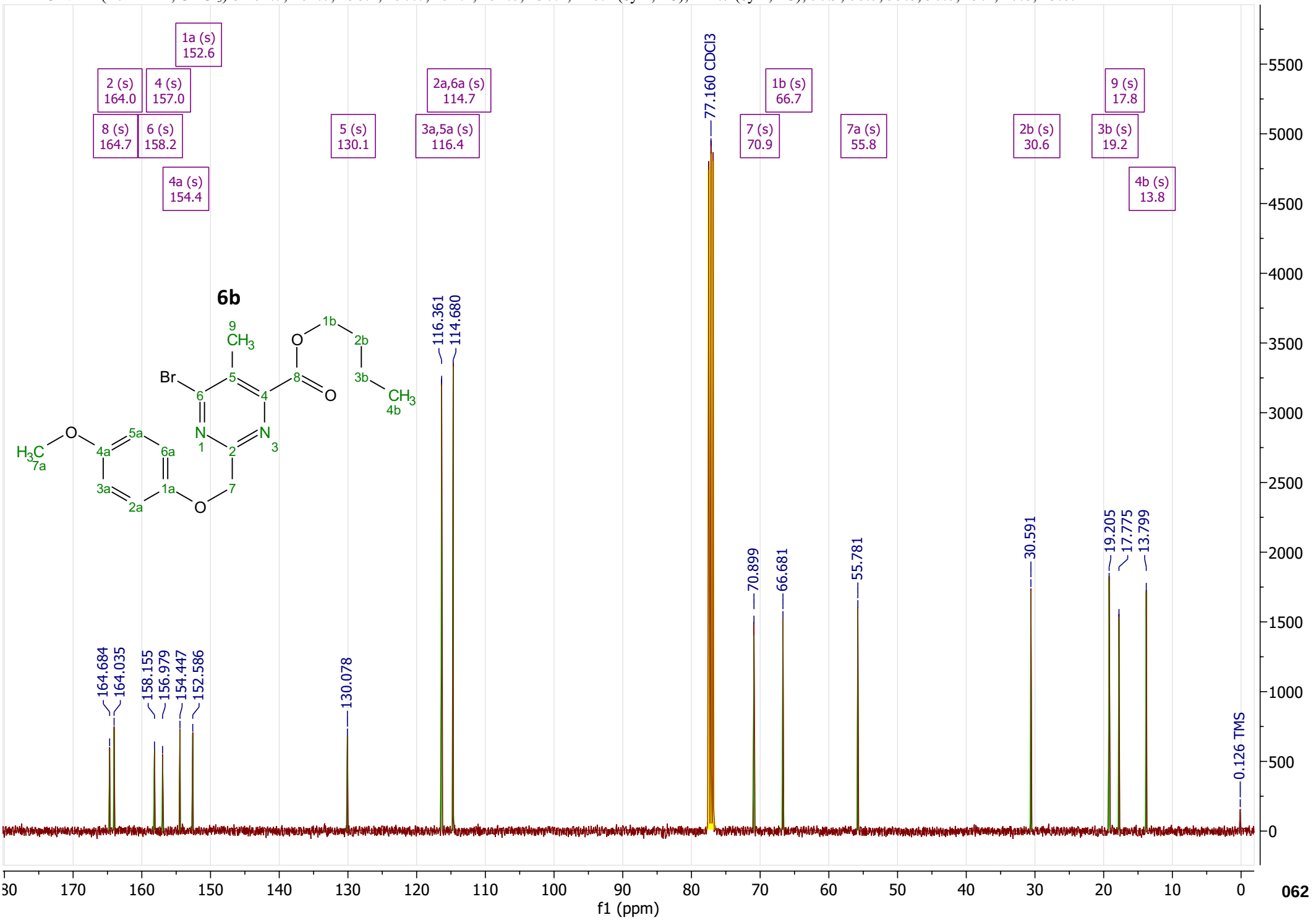


060

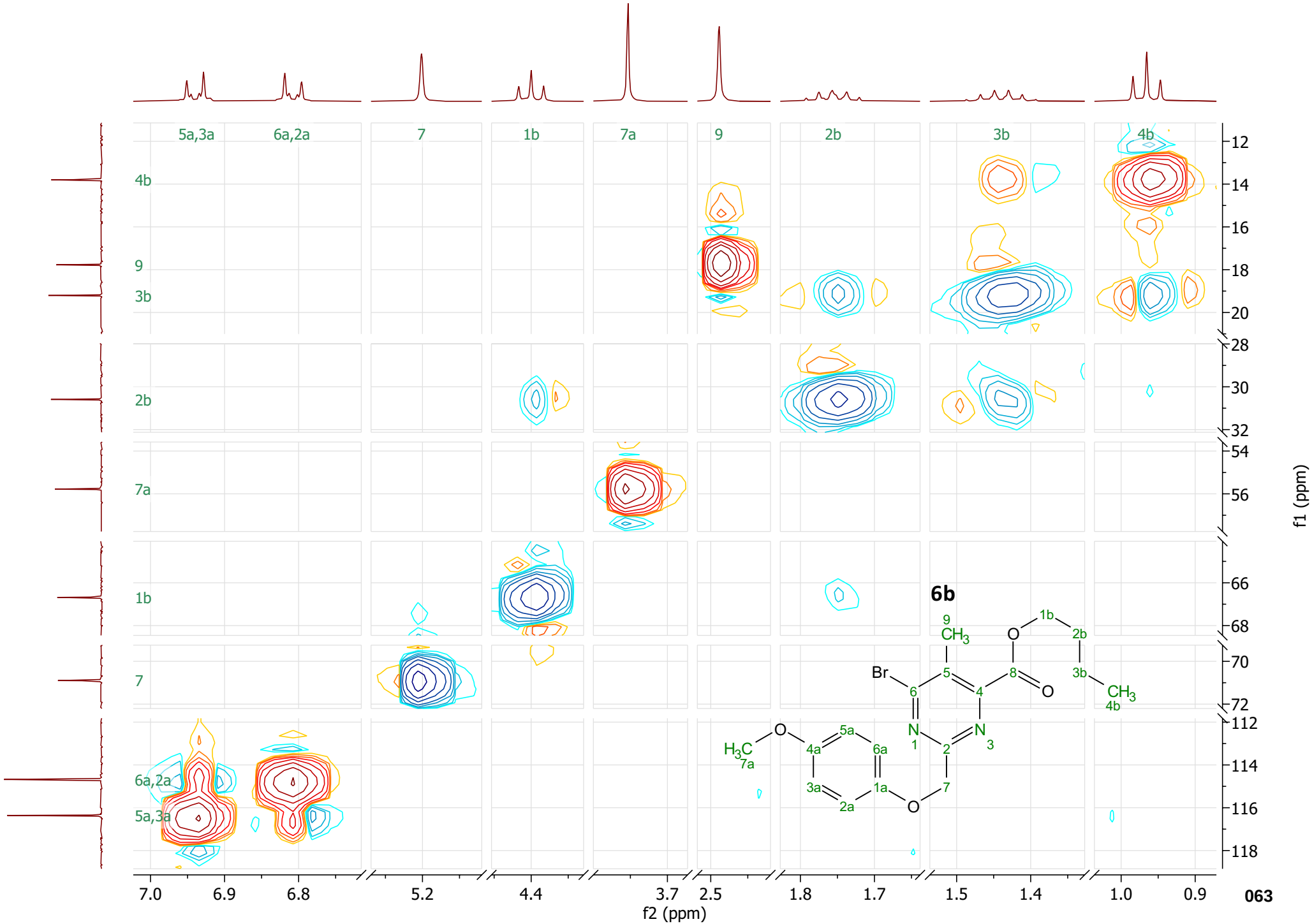
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 – 6.90 (m, 2H), 6.85 – 6.76 (m, 2H), 5.20 (s, 2H), 4.40 (t, *J* = 6.7 Hz, 2H), 3.75 (s, 3H), 2.49 (s, 3H), 1.81 – 1.70 (m, 2H), 1.51 – 1.37 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).



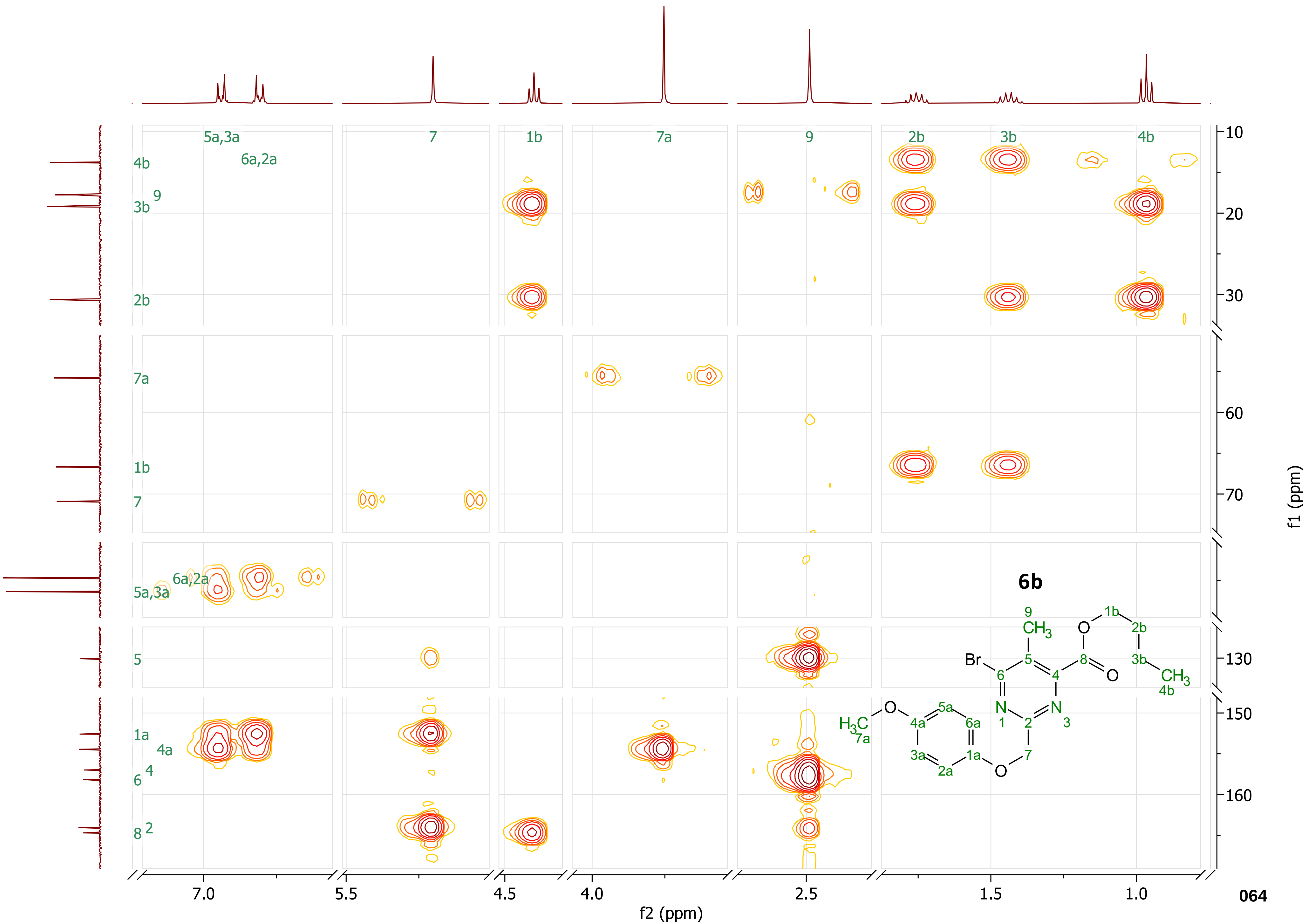
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 164.0, 158.2, 157.0, 154.4, 152.6, 130.1, 116.4 (sym, 2C), 114.7 (sym, 2C), 70.9, 66.7, 55.8, 30.6, 19.2, 17.8, 13.8.



13C HSQC

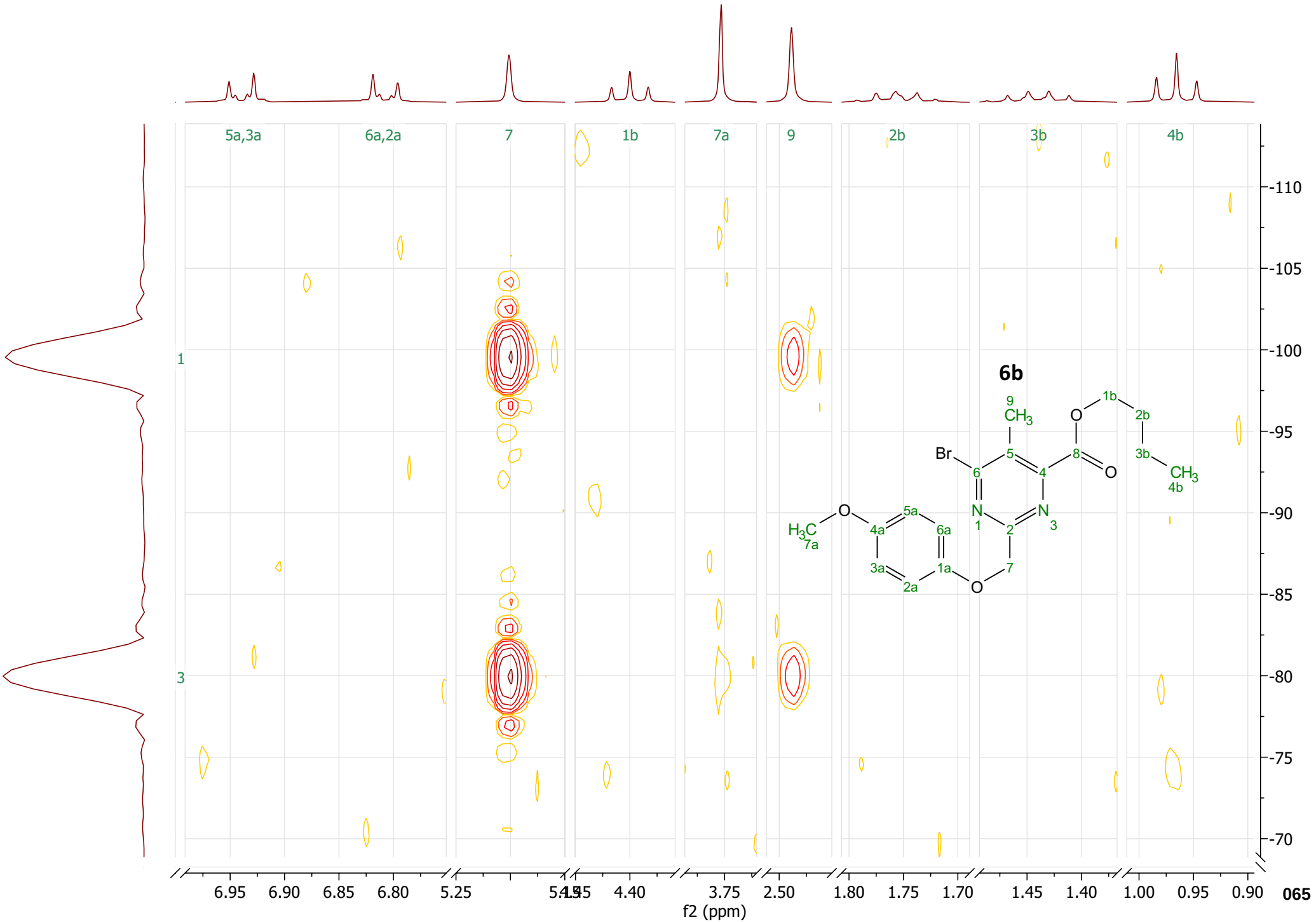


13C HMBC



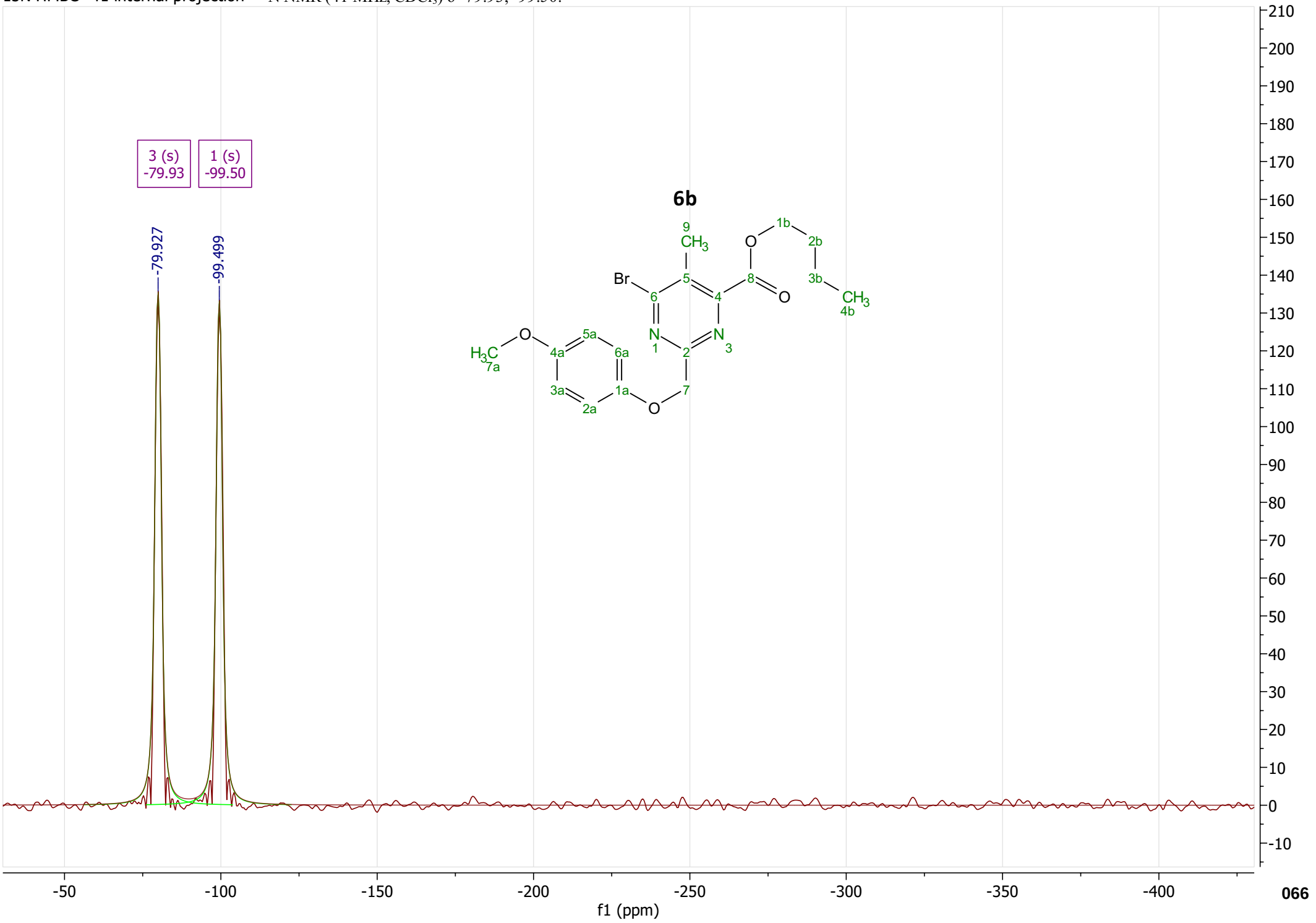
5a,3a
6a,2a
7
1b
7a
9
2b
3b
4b
5a,3a
6a,2a
7a
1b
7
5
1a
4a
4
6
8
2
10
20
30
60
70
130
150
160
7.0
5.5
4.5
4.0
2.5
1.5
1.0
f2 (ppm)
f1 (ppm)
6b
9
CH3
O
1b
2b
Br
6
5
4
8
O
3b
CH3
4b
N
1
2
N
3
H3C
7a
O
4a
5a
6a
3a
2a
1a
O
7

15N HMBC


5a,3a
6a,2a
7
1b
7a
9
2b
3b
4b
1
3
6b
f2 (ppm)
f1 (ppm)
6.95
6.90
6.85
6.80
5.25
4.40
3.75
2.50
1.80
1.75
1.70
1.45
1.40
1.00
0.95
0.90
-110
-105
-100
-95
-90
-85
-80
-75
-70

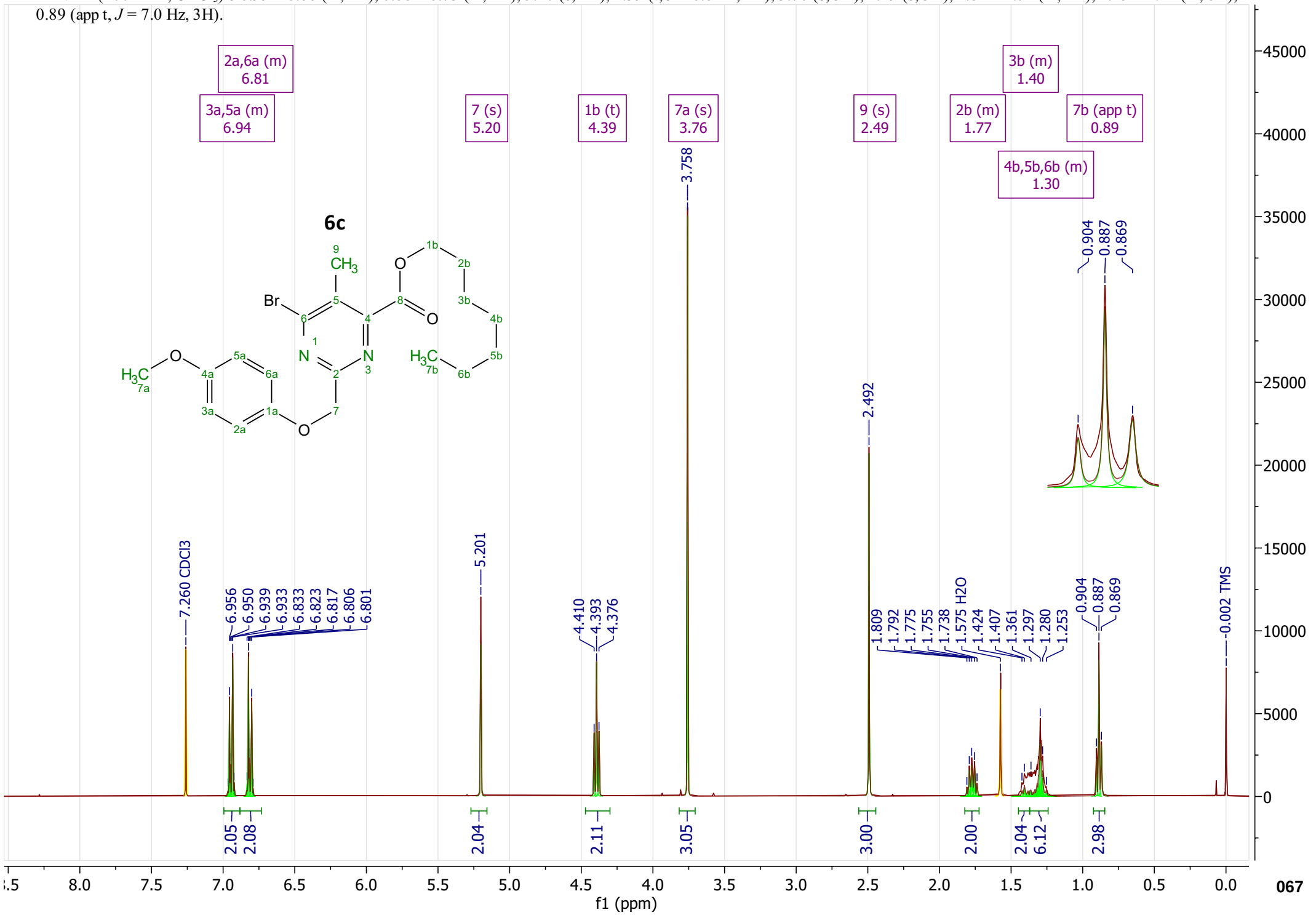
065

15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -79.93, -99.50.

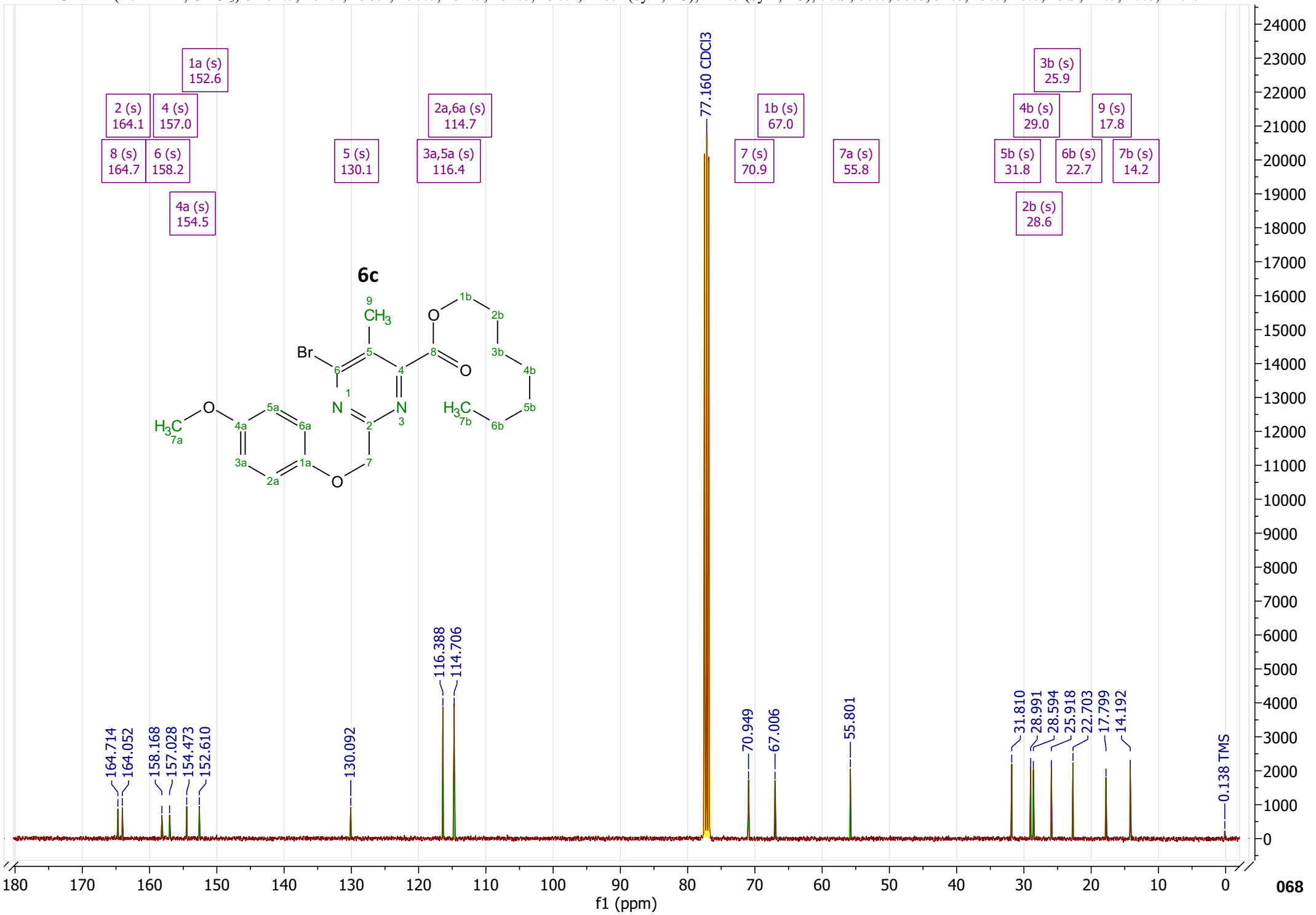


066

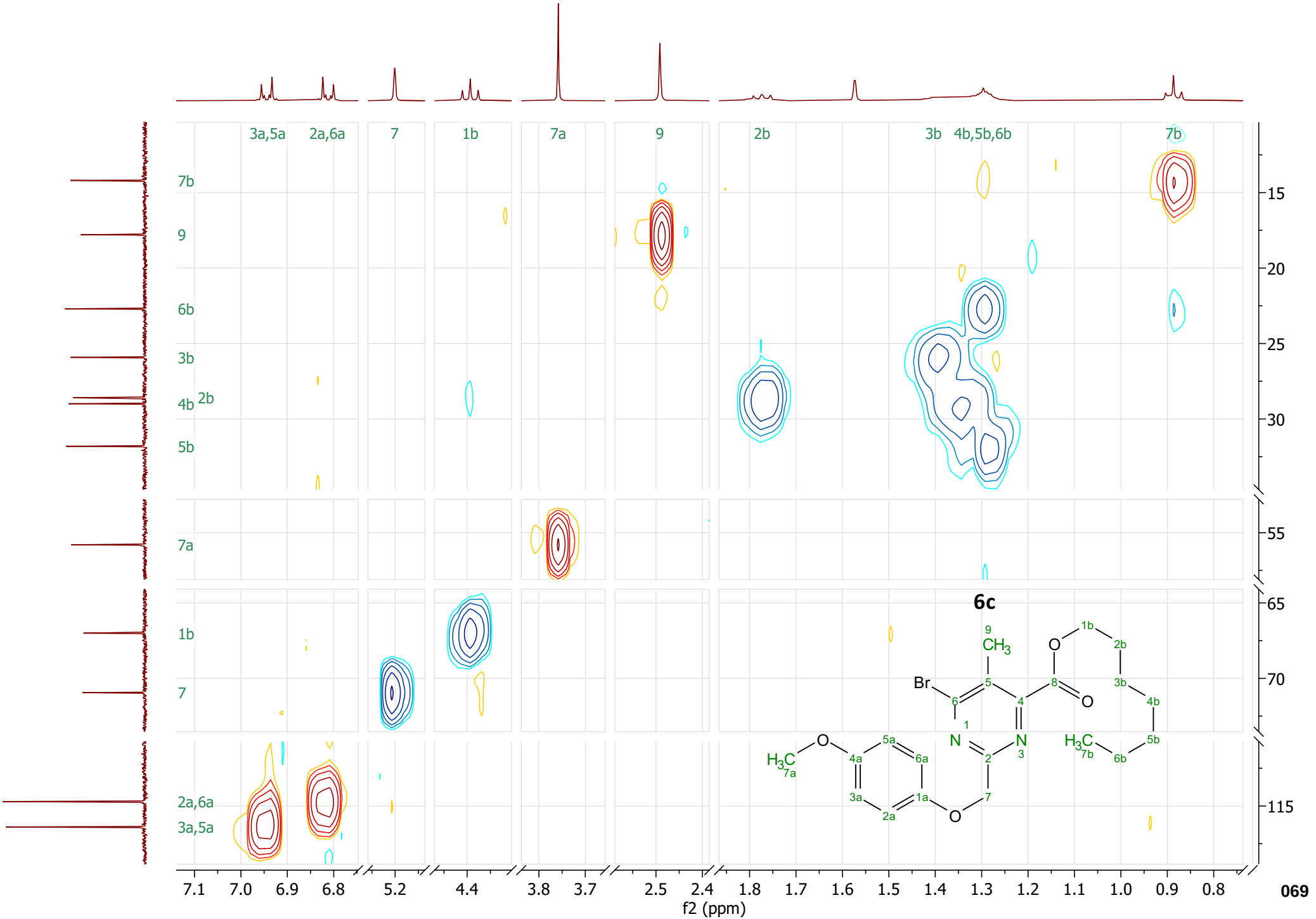
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 – 6.88 (m, 2H), 6.88 – 6.73 (m, 2H), 5.20 (s, 2H), 4.39 (t, *J* = 6.8 Hz, 2H), 3.76 (s, 3H), 2.49 (s, 3H), 1.82 – 1.72 (m, 2H), 1.45 – 1.24 (m, 8H), 0.89 (app t, *J* = 7.0 Hz, 3H).



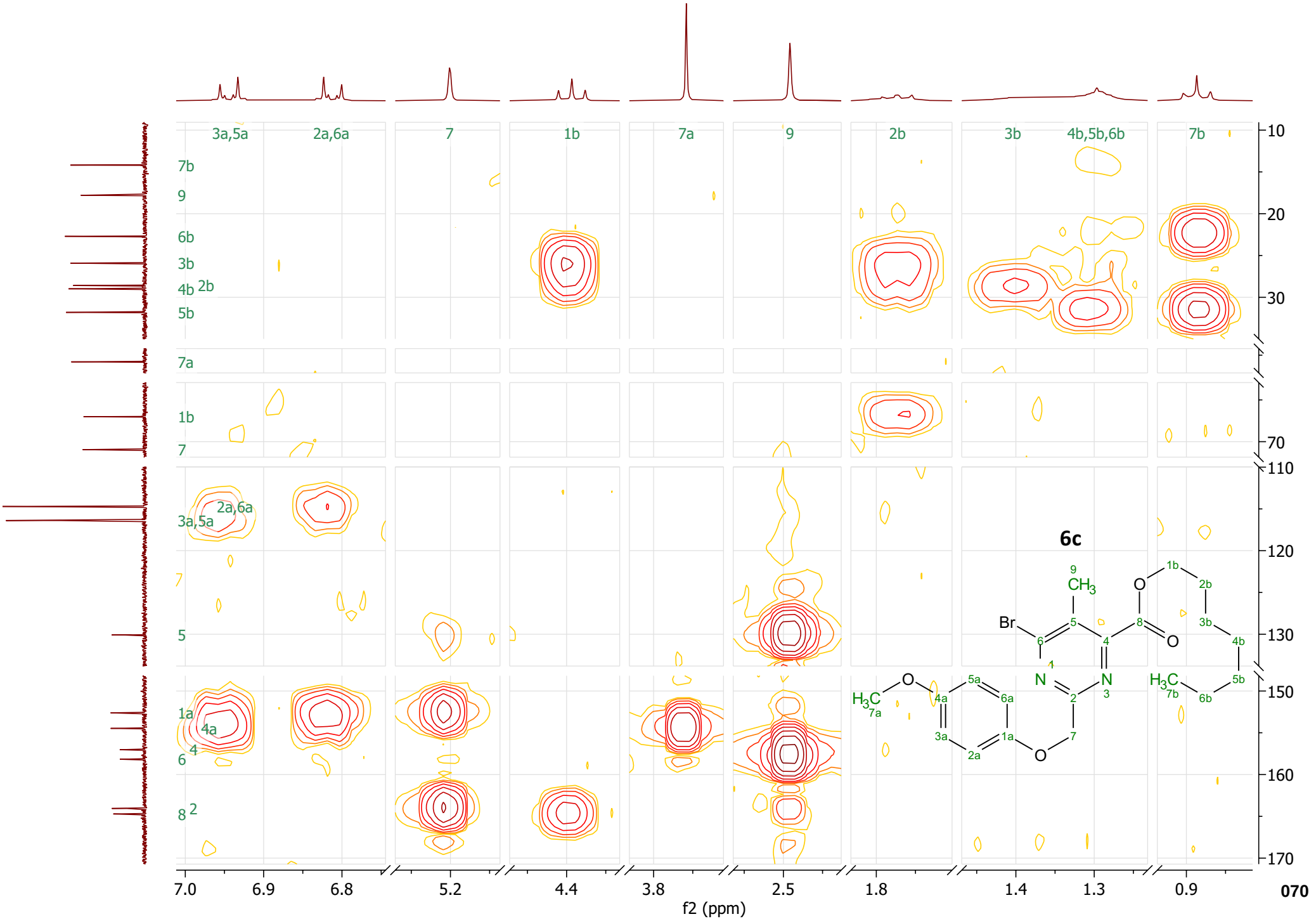
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 164.1, 158.2, 157.0, 154.5, 152.6, 130.1, 116.4 (sym, 2C), 114.7 (sym, 2C), 70.9, 67.0, 55.8, 31.8, 29.0, 28.6, 25.9, 22.7, 17.8, 14.2.



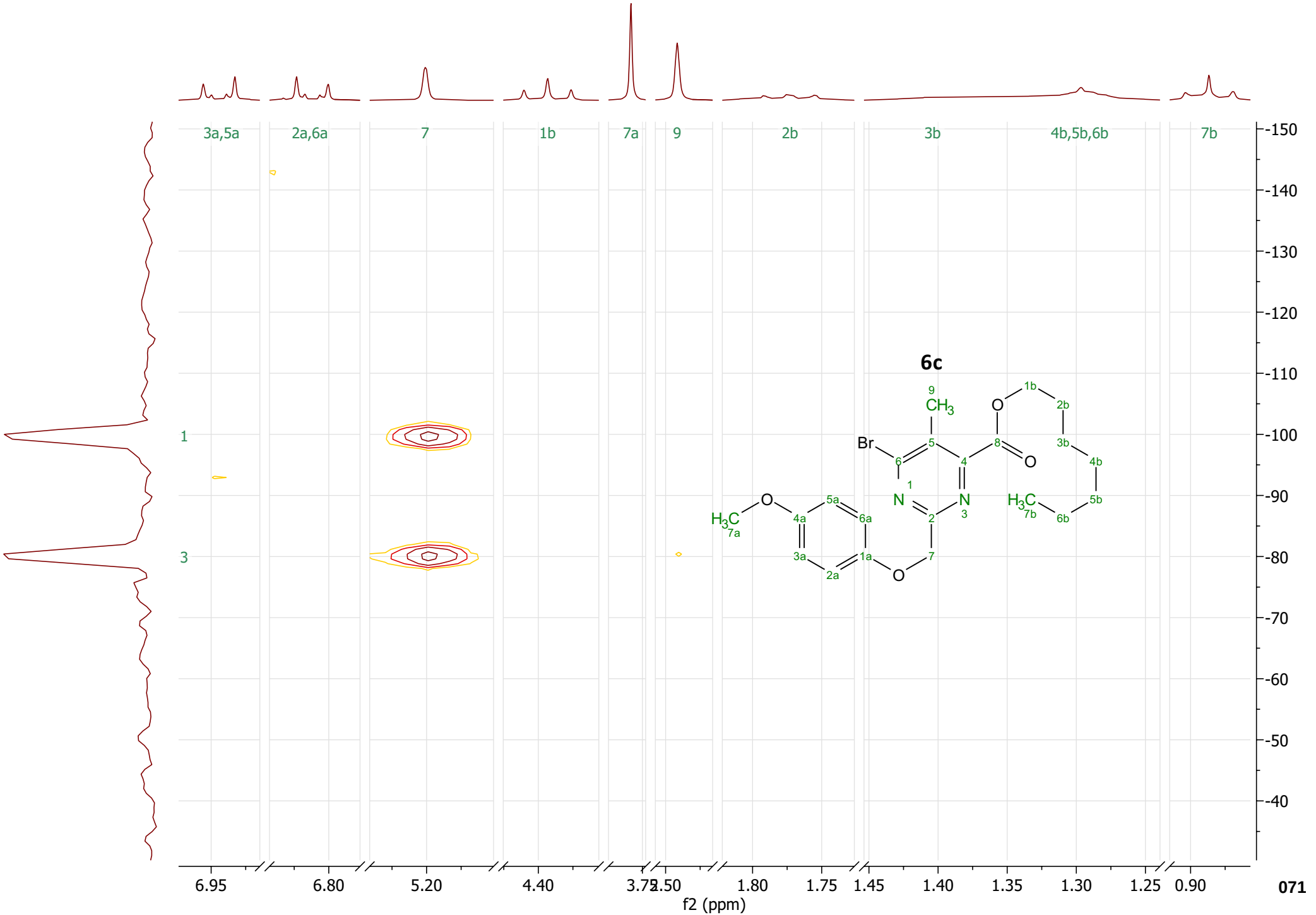
13C HSQC



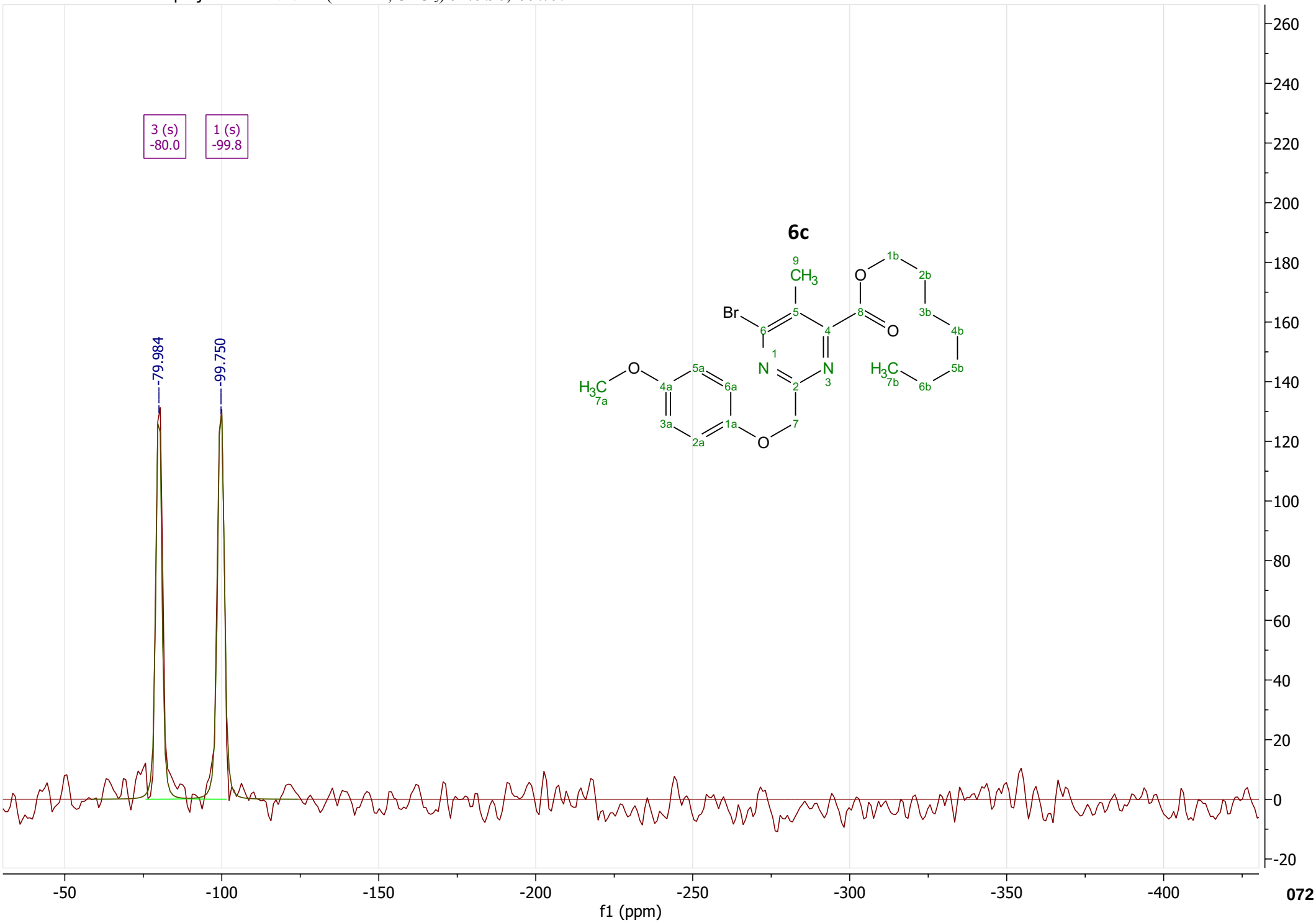
13C HMBC



15N HMBC

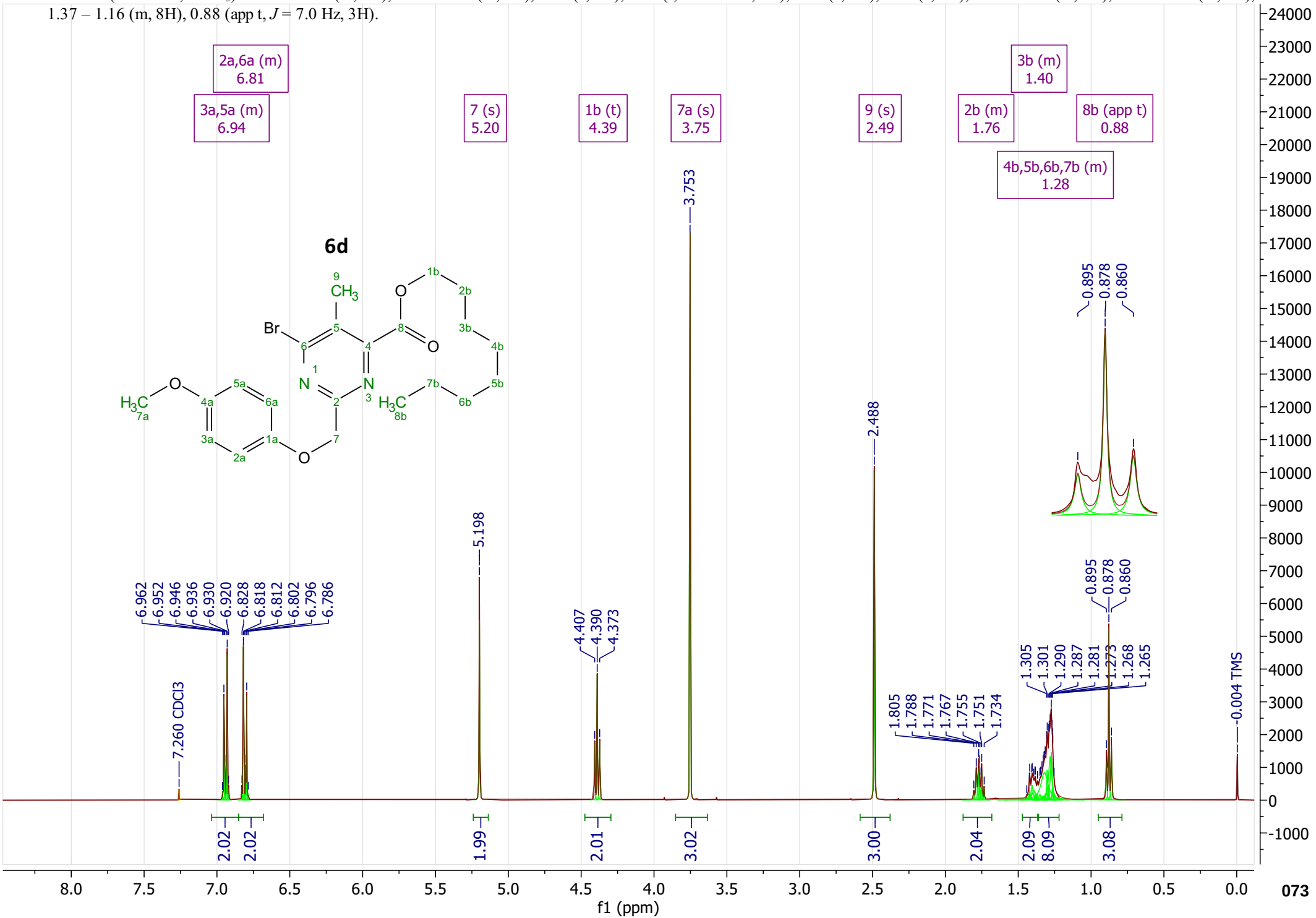


15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -79.98, -99.75.

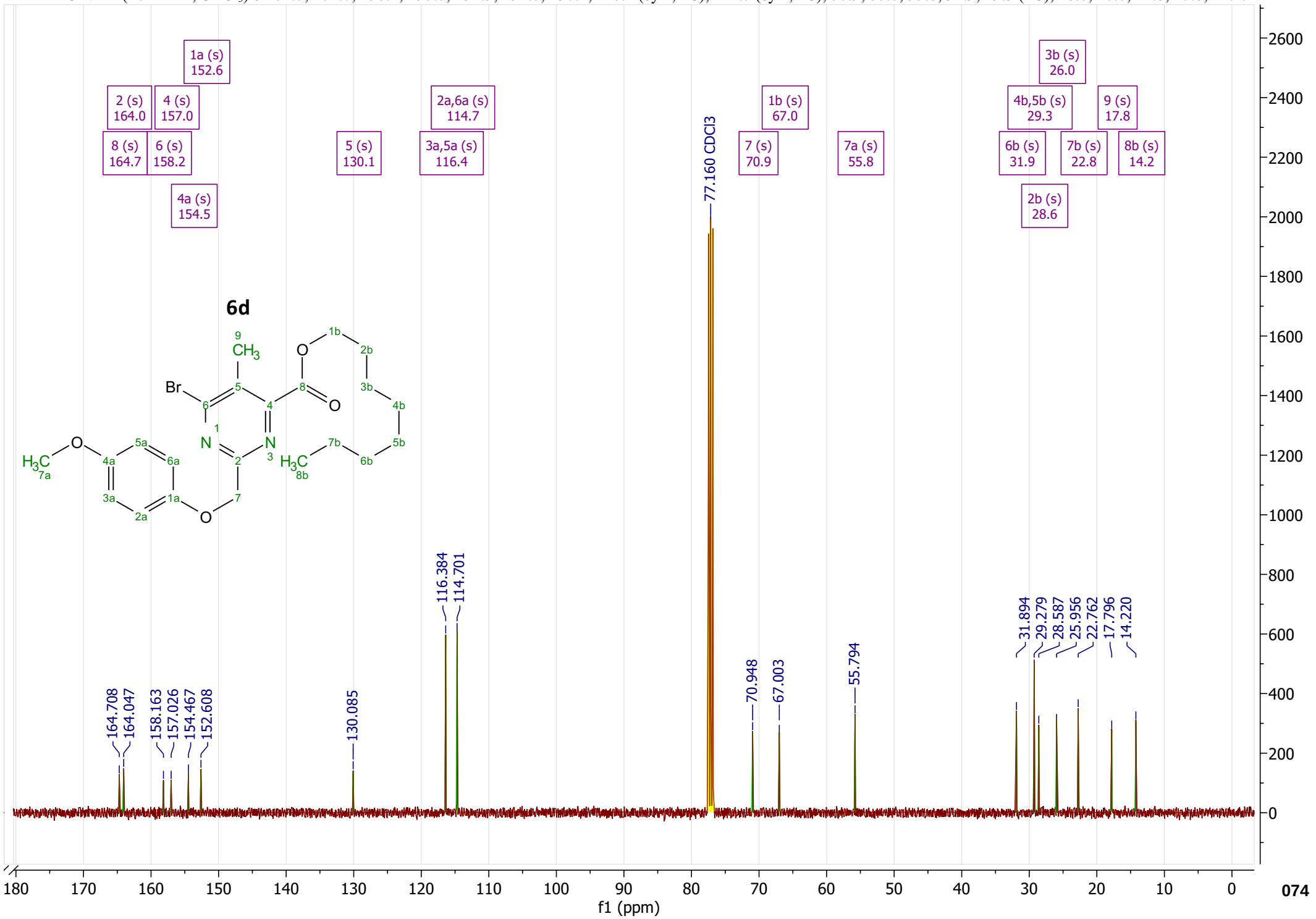


072

1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.04 – 6.85 (m, 2H), 6.85 – 6.68 (m, 2H), 5.20 (s, 2H), 4.39 (t, *J* = 6.8 Hz, 2H), 3.75 (s, 3H), 2.49 (s, 3H), 1.88 – 1.68 (m, 2H), 1.48 – 1.37 (m, 2H), 1.37 – 1.16 (m, 8H), 0.88 (app t, *J* = 7.0 Hz, 3H).

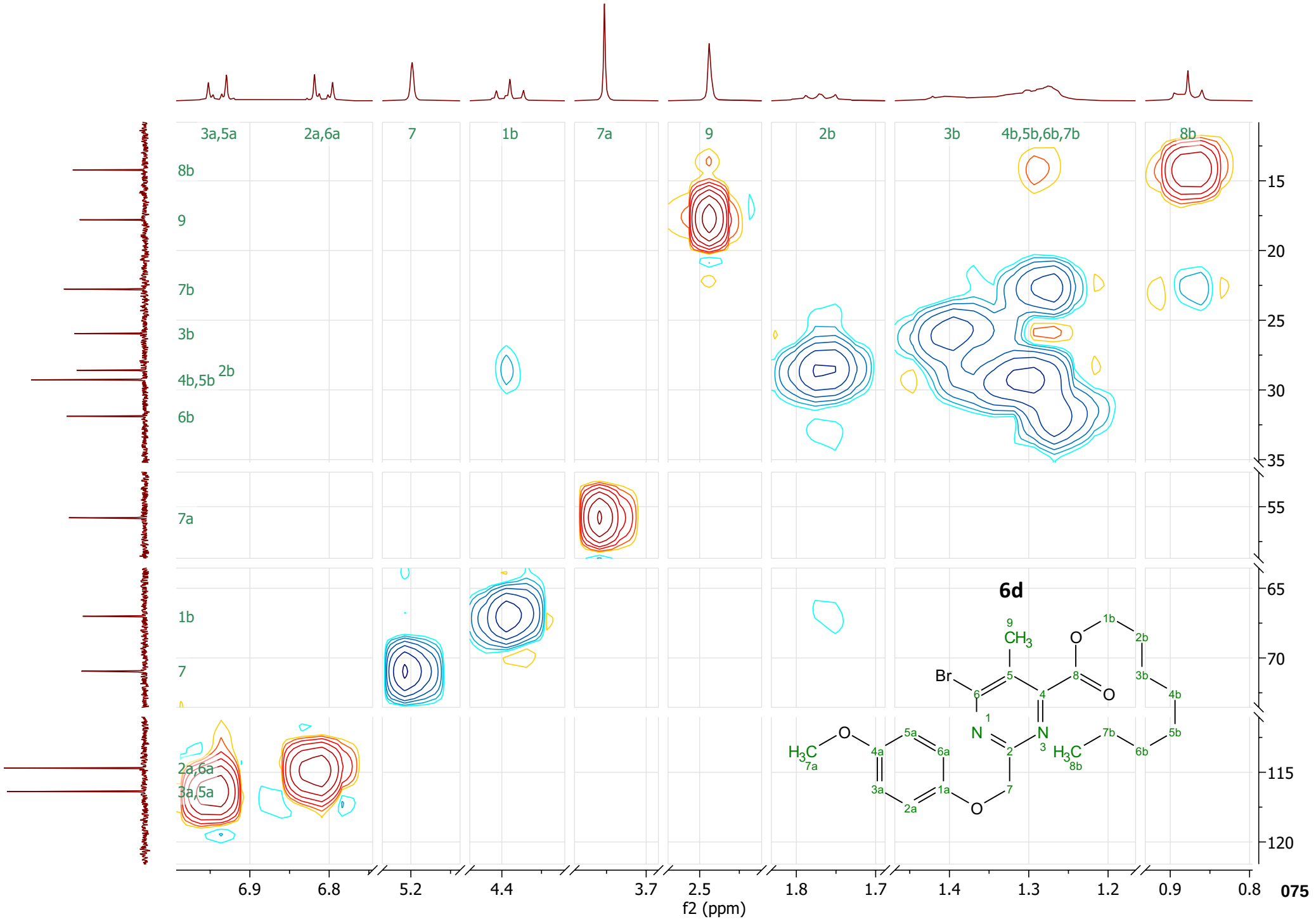


13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 164.0, 158.2, 157.0, 154.5, 152.6, 130.1, 116.4 (sym, 2C), 114.7 (sym, 2C), 70.9, 67.0, 55.8, 31.9, 29.3 (2C), 28.6, 26.0, 22.8, 17.8, 14.2.

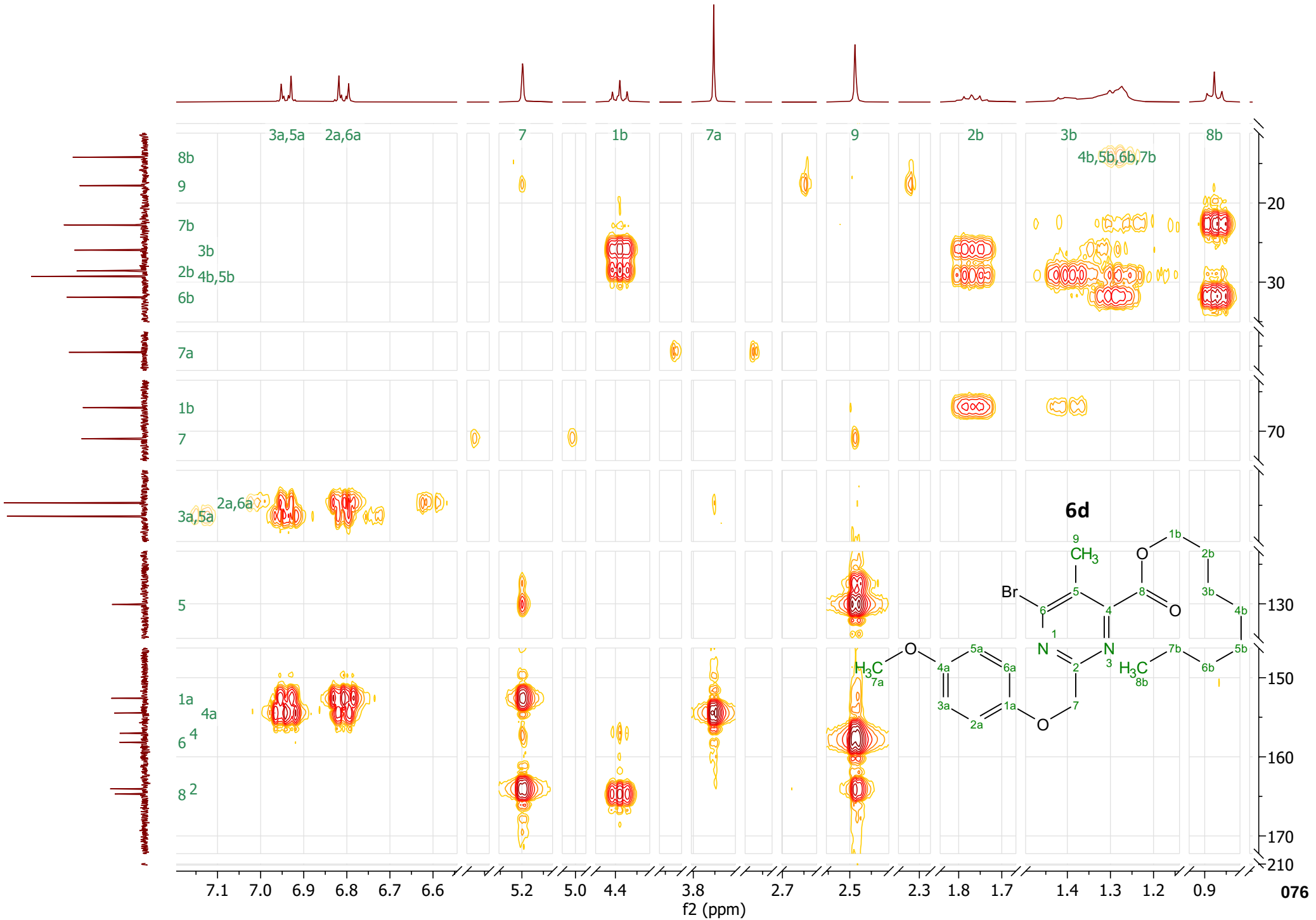


074

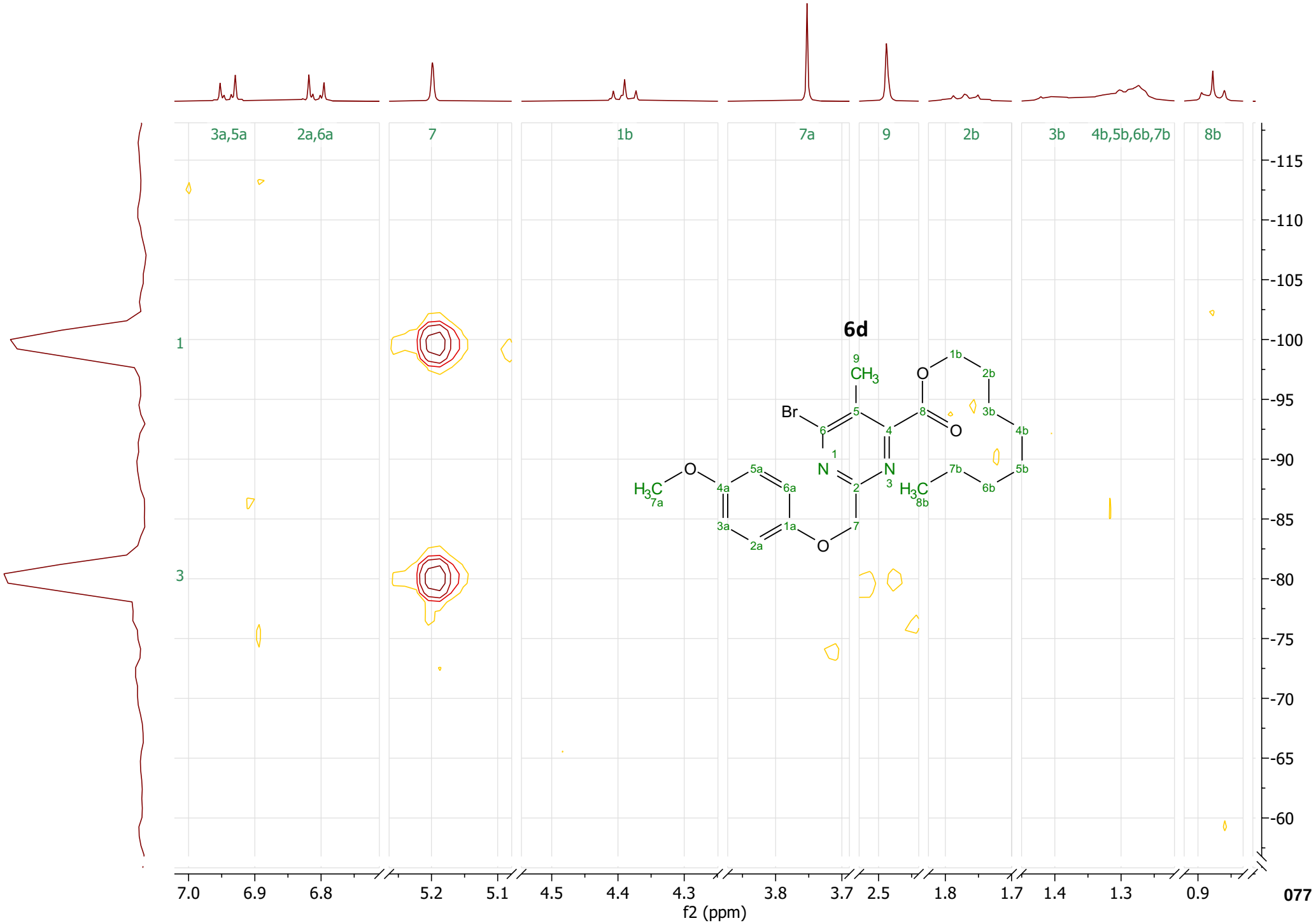
13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
8b
9
7b
3b
4b,5b
2b
6b
7a
1b
7
2a,6a
3a,5a
6d
f1 (ppm)
f2 (ppm)
6.9
6.8
5.2
4.4
3.7
2.5
1.8
1.7
1.4
1.3
1.2
0.9
0.8
075

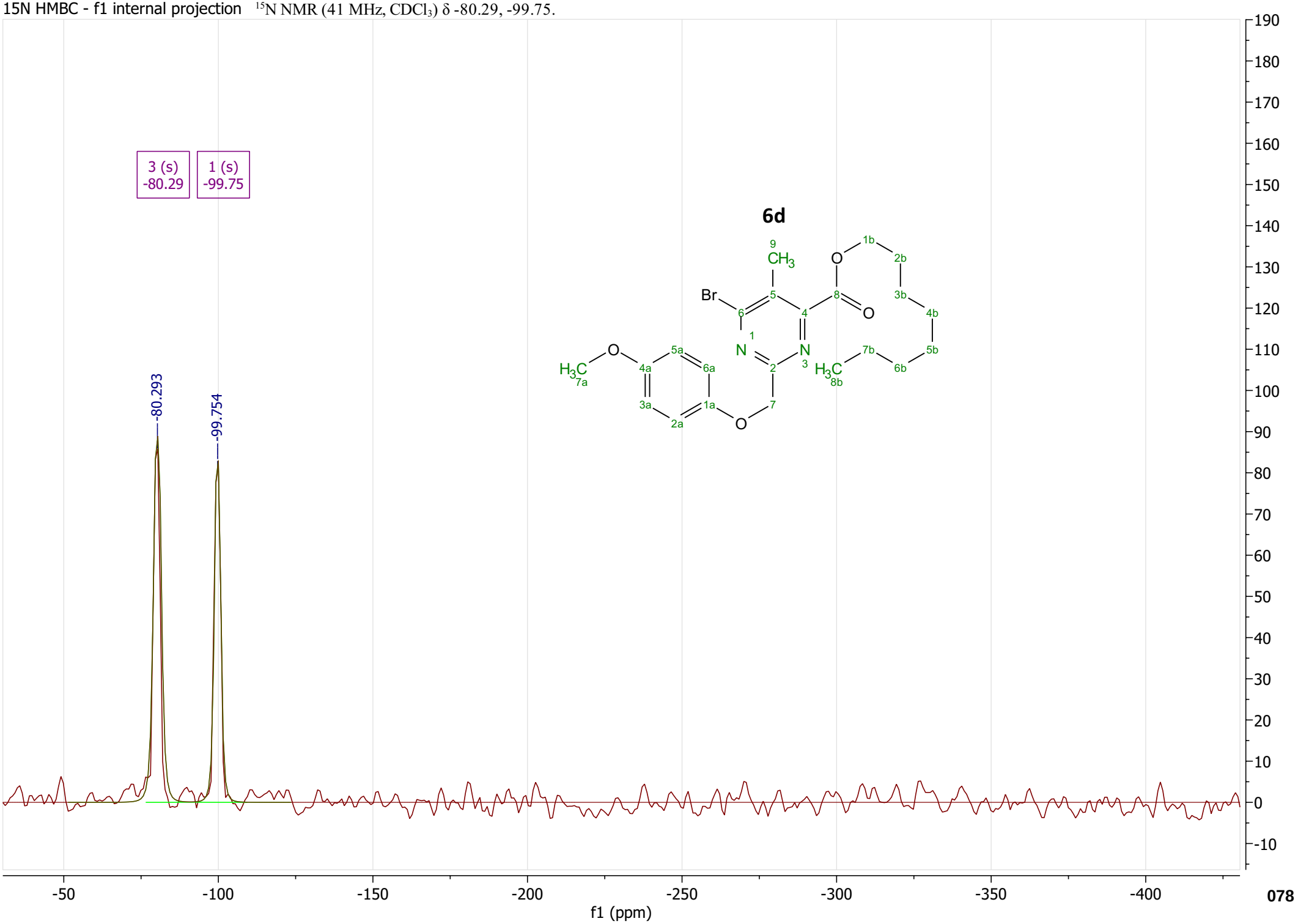
13C HMBC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
8b
9
7b
3b
2b
4b,5b
6b
7a
1b
7
3a,5a
2a,6a
5
1a
4a
6
4
8
2
6d
f2 (ppm)
f1 (ppm)
076

15N HMBC

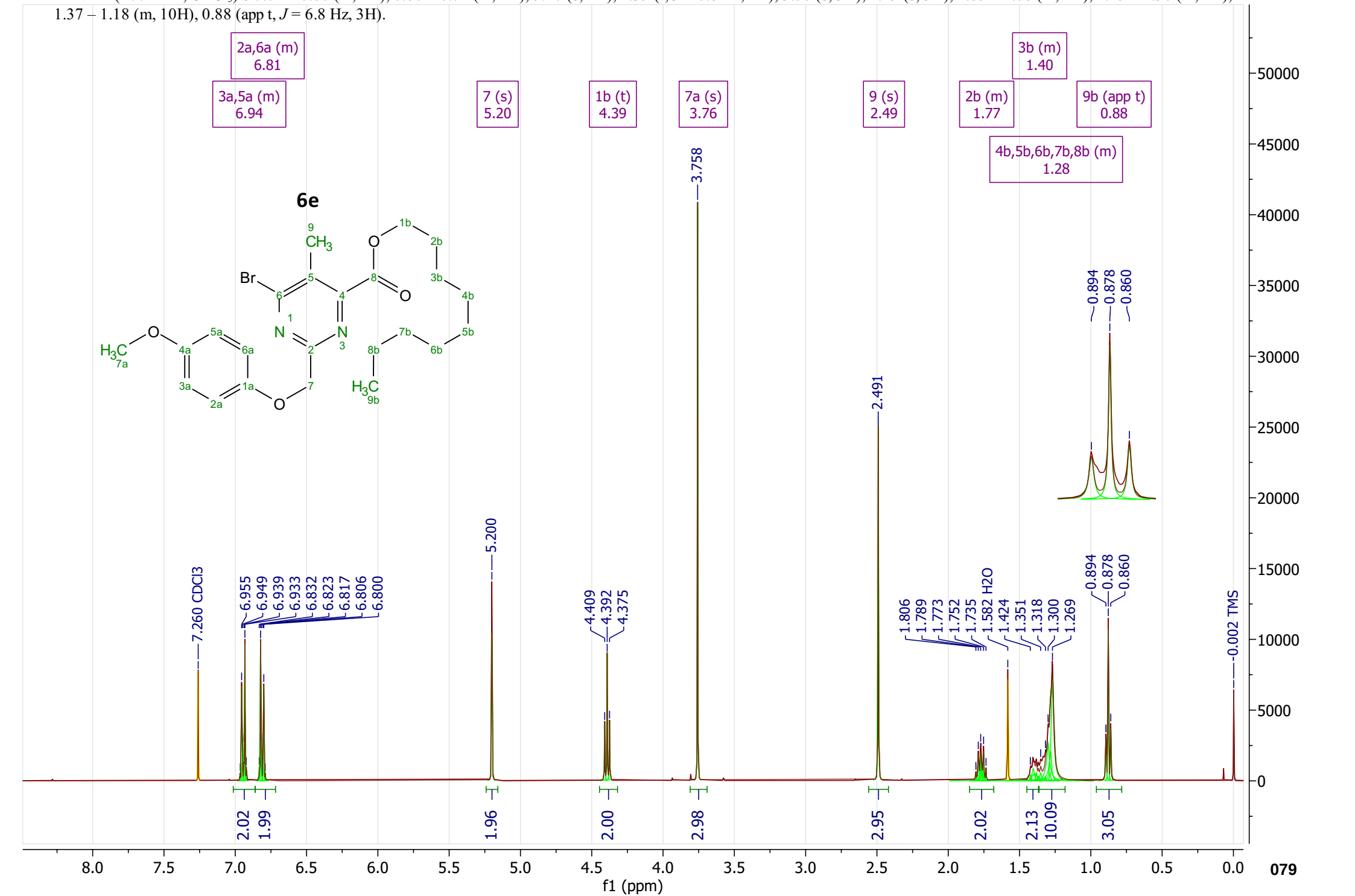


15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -80.29, -99.75.

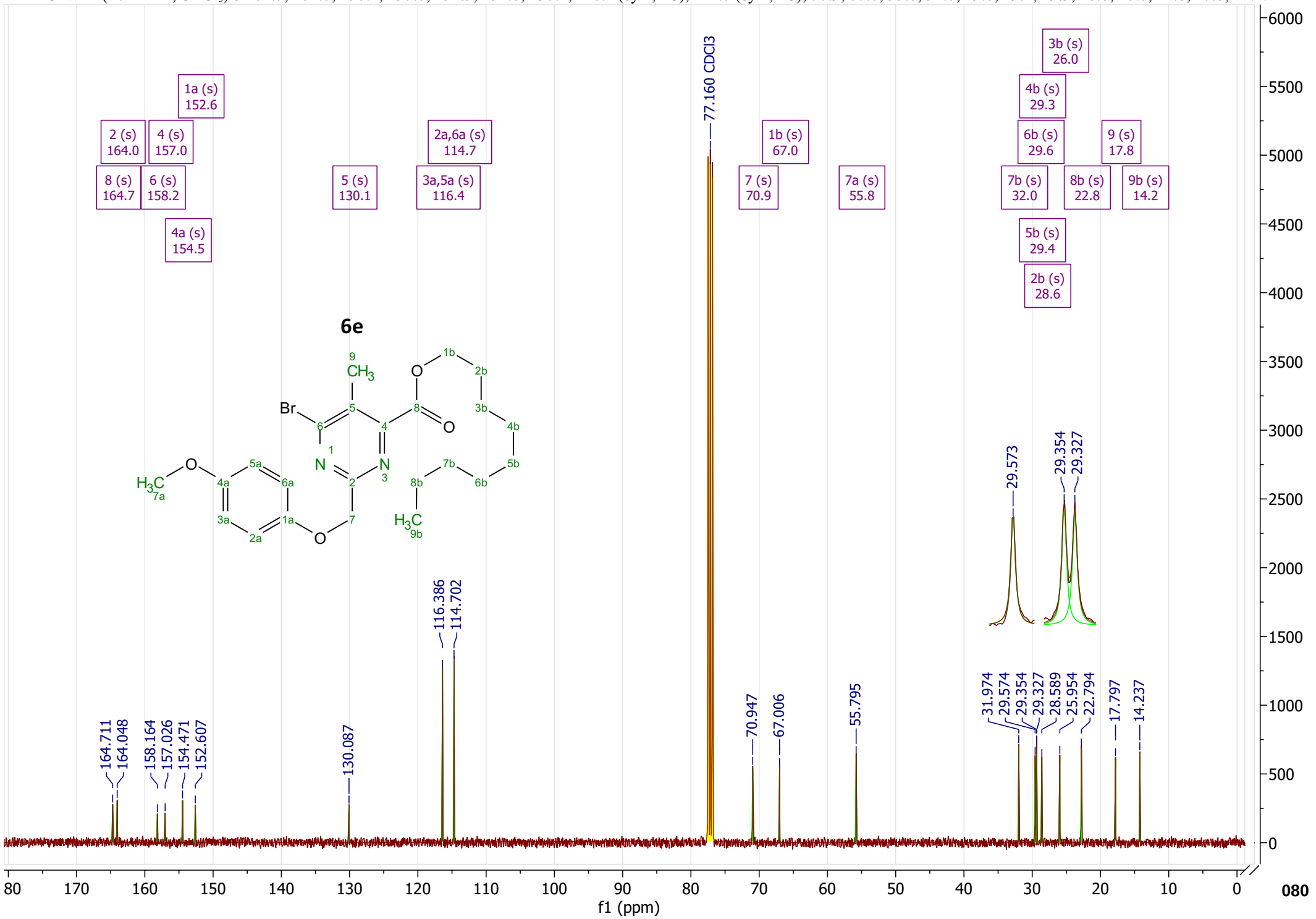


078

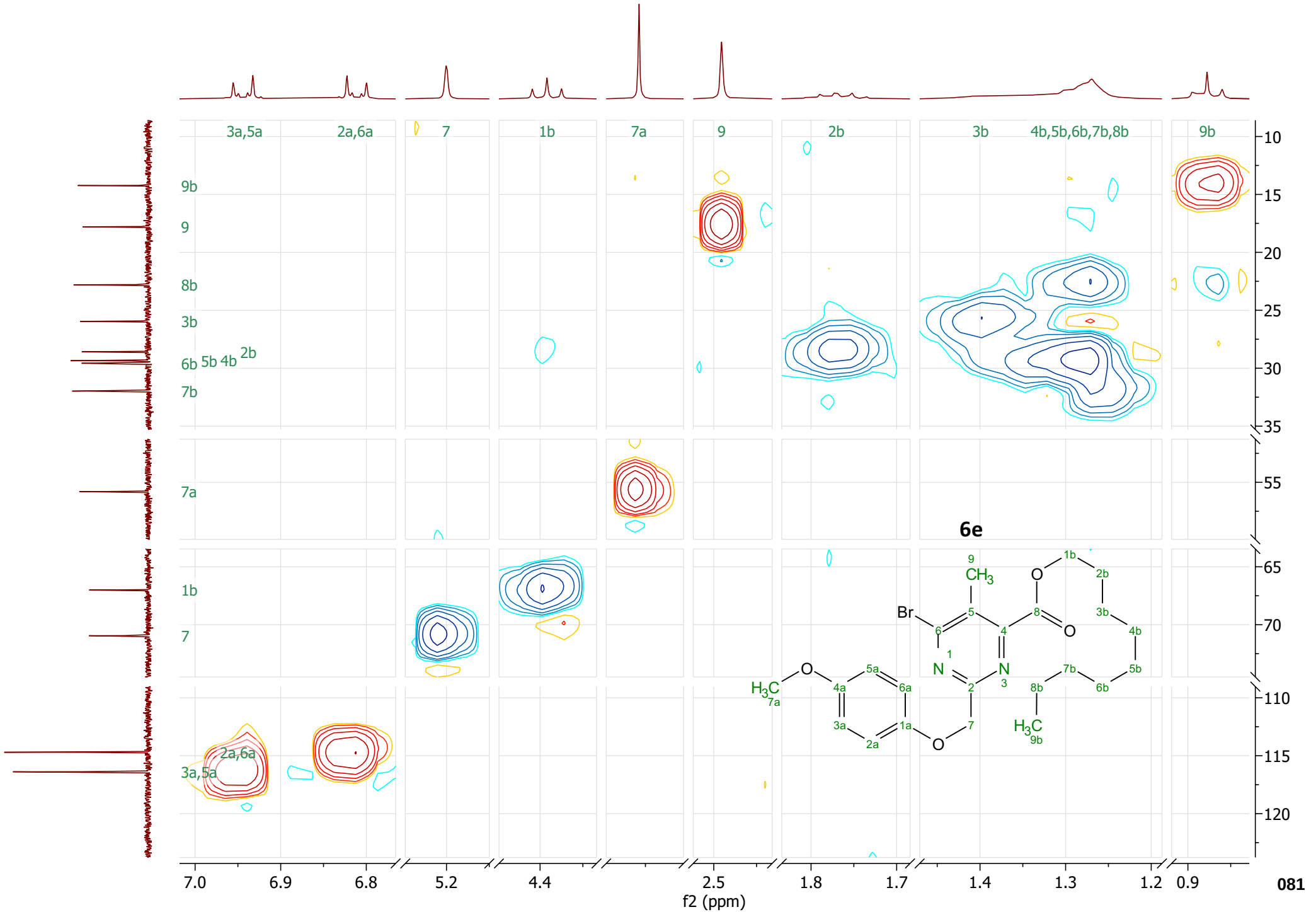
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 – 6.86 (m, 2H), 6.86 – 6.72 (m, 2H), 5.20 (s, 2H), 4.39 (t, *J* = 6.8 Hz, 2H), 3.76 (s, 3H), 2.49 (s, 3H), 1.85 – 1.68 (m, 2H), 1.45 – 1.36 (m, 2H), 1.37 – 1.18 (m, 10H), 0.88 (app t, *J* = 6.8 Hz, 3H).



13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.7, 164.0, 158.2, 157.0, 154.5, 152.6, 130.1, 116.4 (sym, 2C), 114.7 (sym, 2C), 70.9, 67.0, 55.8, 32.0, 29.6, 29.4, 29.3, 28.6, 26.0, 22.8, 17.8, 14.2.

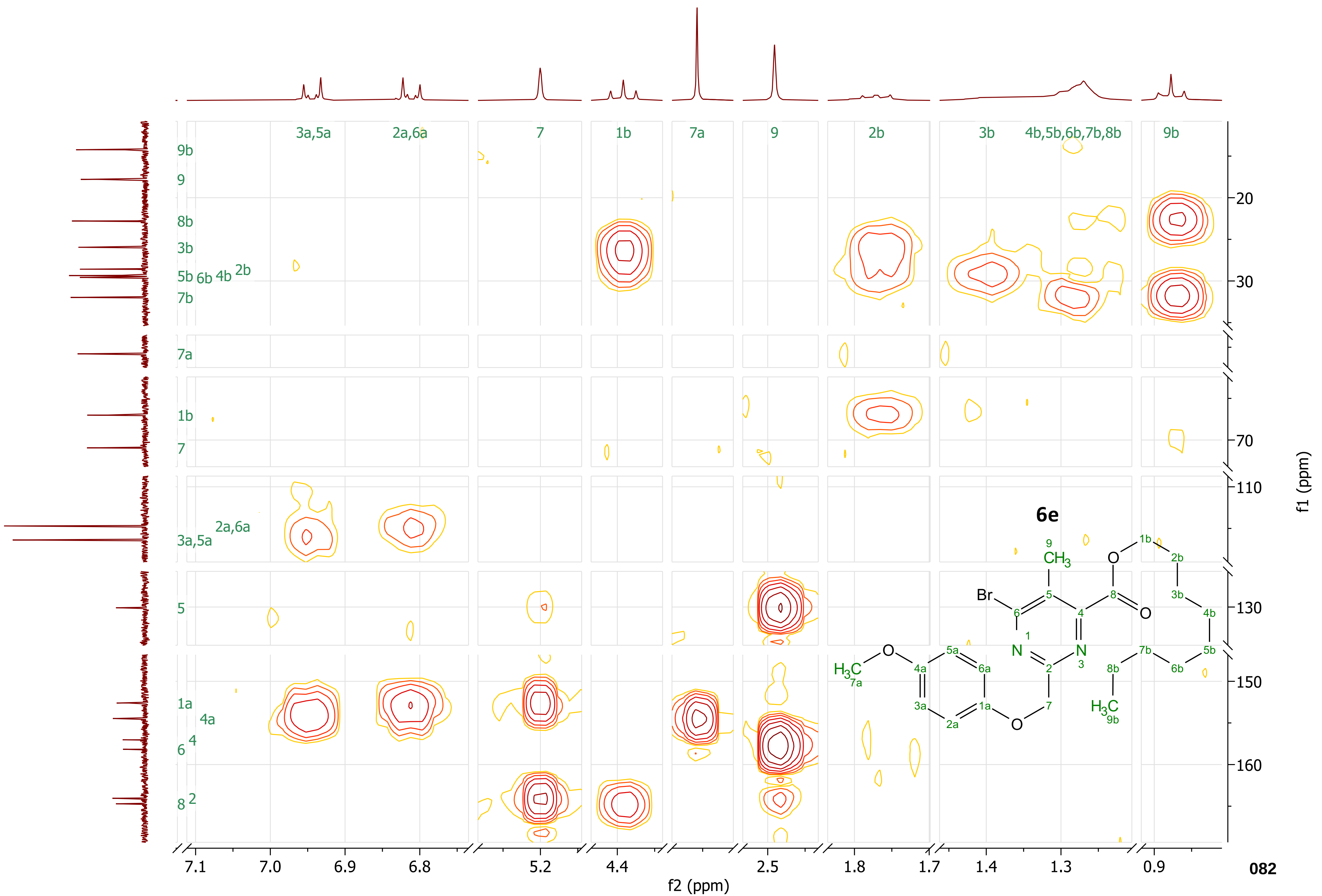


13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b,8b
9b
9b
9
8b
3b
6b 5b 4b 2b
7b
7a
1b
7
2a,6a
3a,5a
6e
f2 (ppm)
f1 (ppm)
7.0
6.9
6.8
5.2
4.4
2.5
1.8
1.7
1.4
1.3
1.2
0.9
10
15
20
25
30
35
55
65
70
110
115
120

081

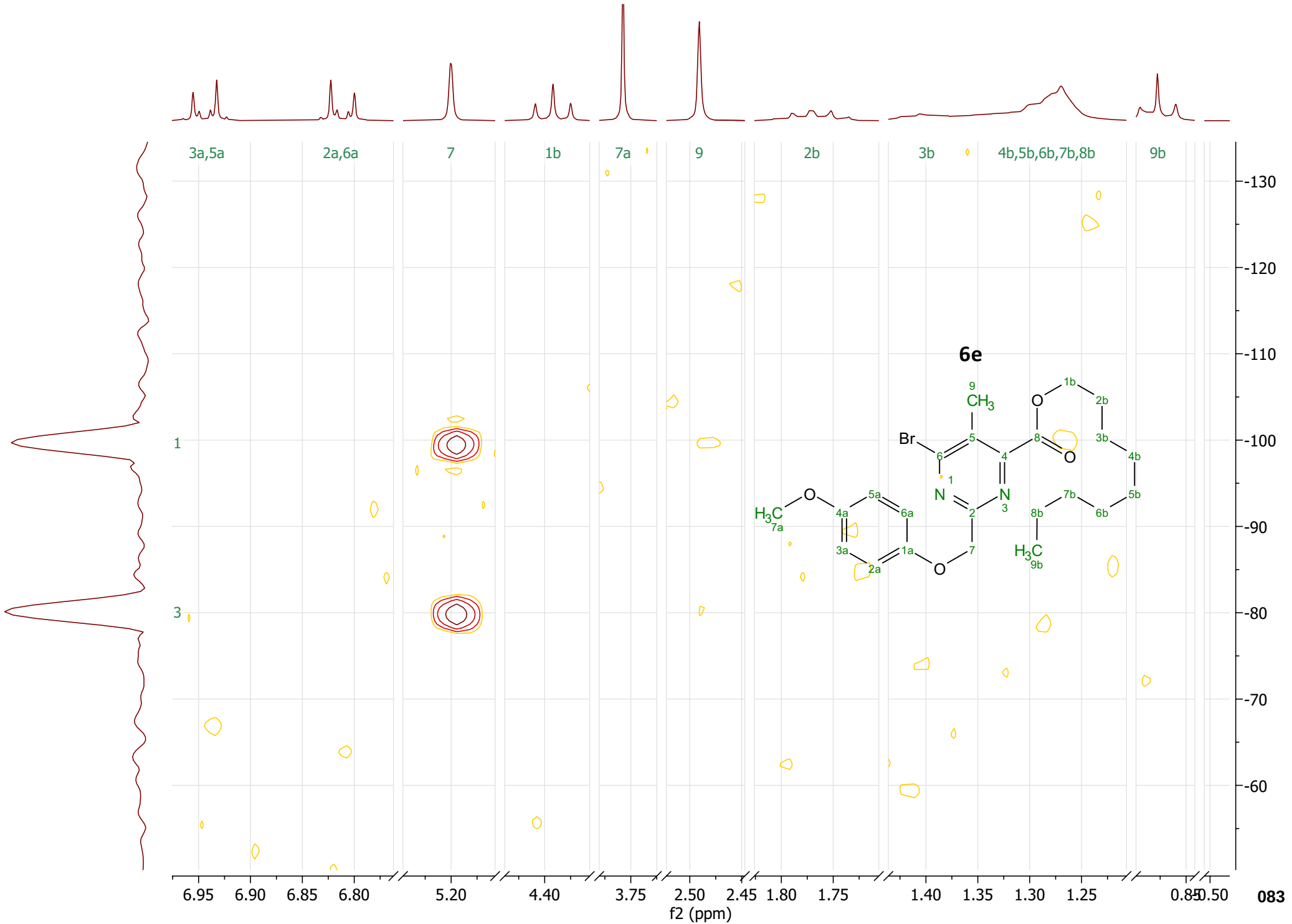
13C HMBC



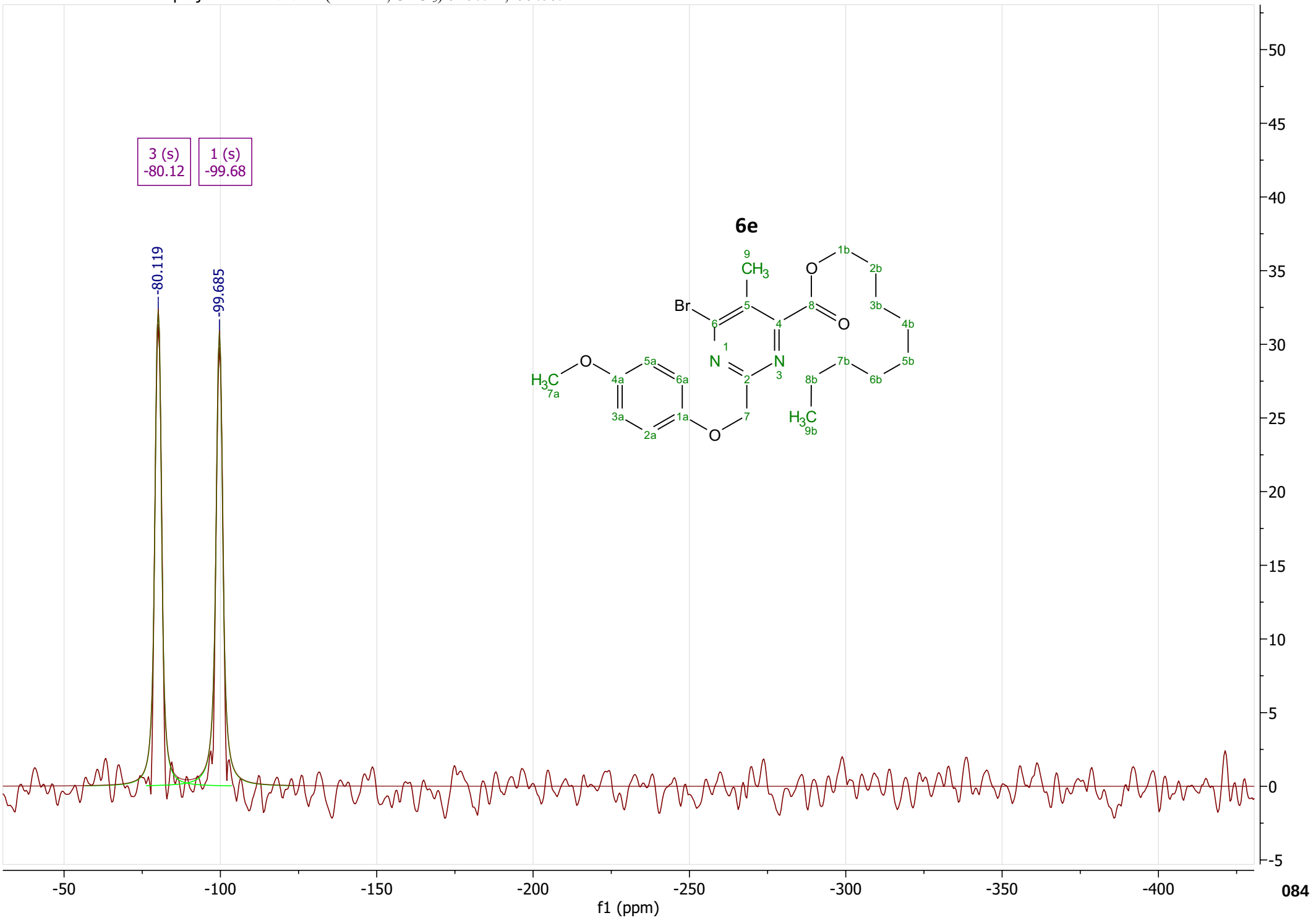
3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b,8b
9b
9b
9
8b
3b
5b 6b 4b 2b
7b
7a
1b
7
2a,6a
3a,5a
5
1a
4a
6 4
8 2
20
30
70
110
130
150
160
f1 (ppm)
7.1
7.0
6.9
6.8
5.2
4.4
2.5
1.8
1.7
1.4
1.3
0.9
f2 (ppm)
6e

082

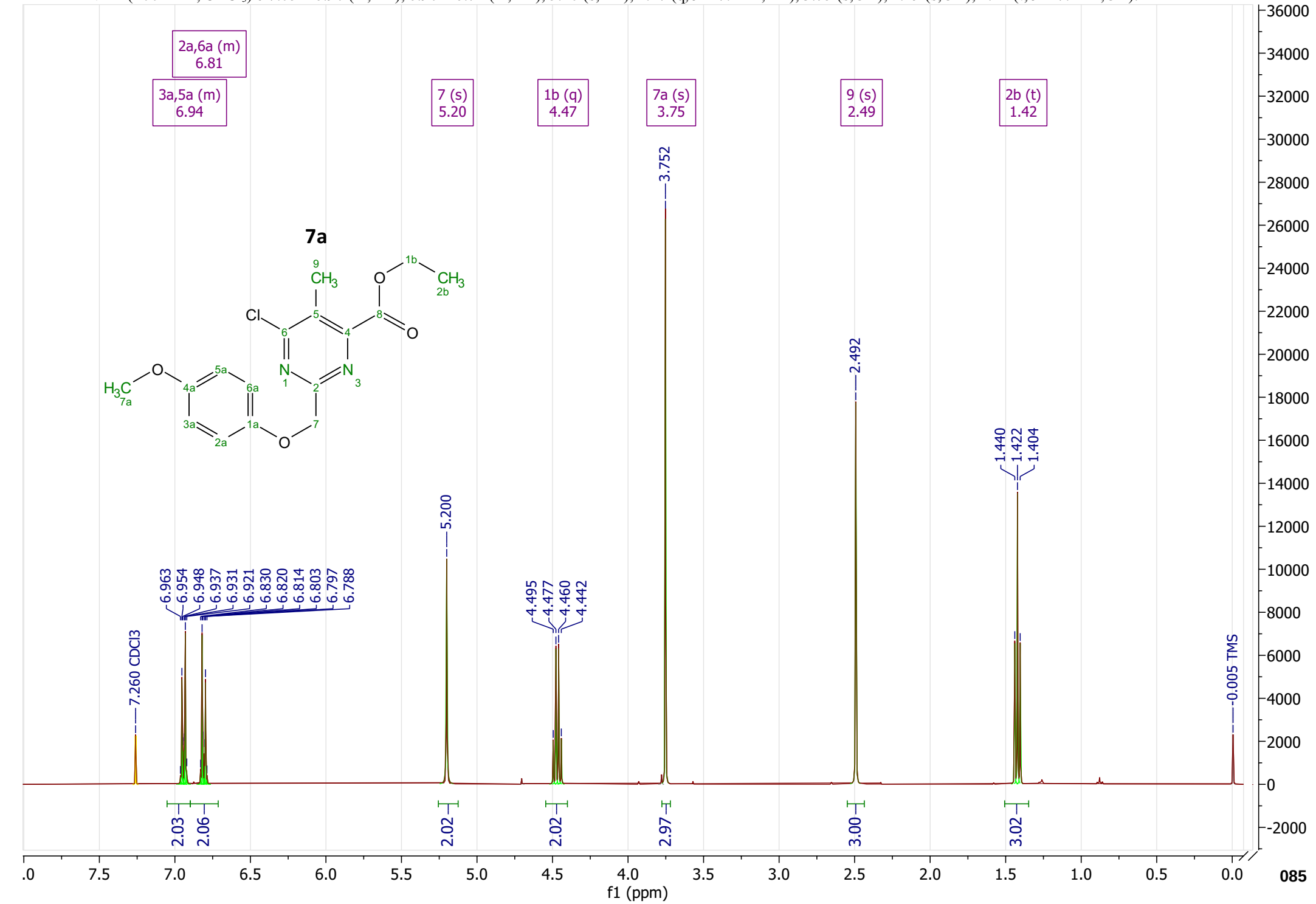
15N HMBC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b,8b
9b
1
3
-130
-120
-110
-100
-90
-80
-70
-60
f1 (ppm)
6.95
6.90
6.85
6.80
5.20
4.40
3.75
2.50
2.45
1.80
1.75
1.40
1.35
1.30
1.25
0.85
0.50
f2 (ppm)
6e
Br
CH3
O
N
H3C
083

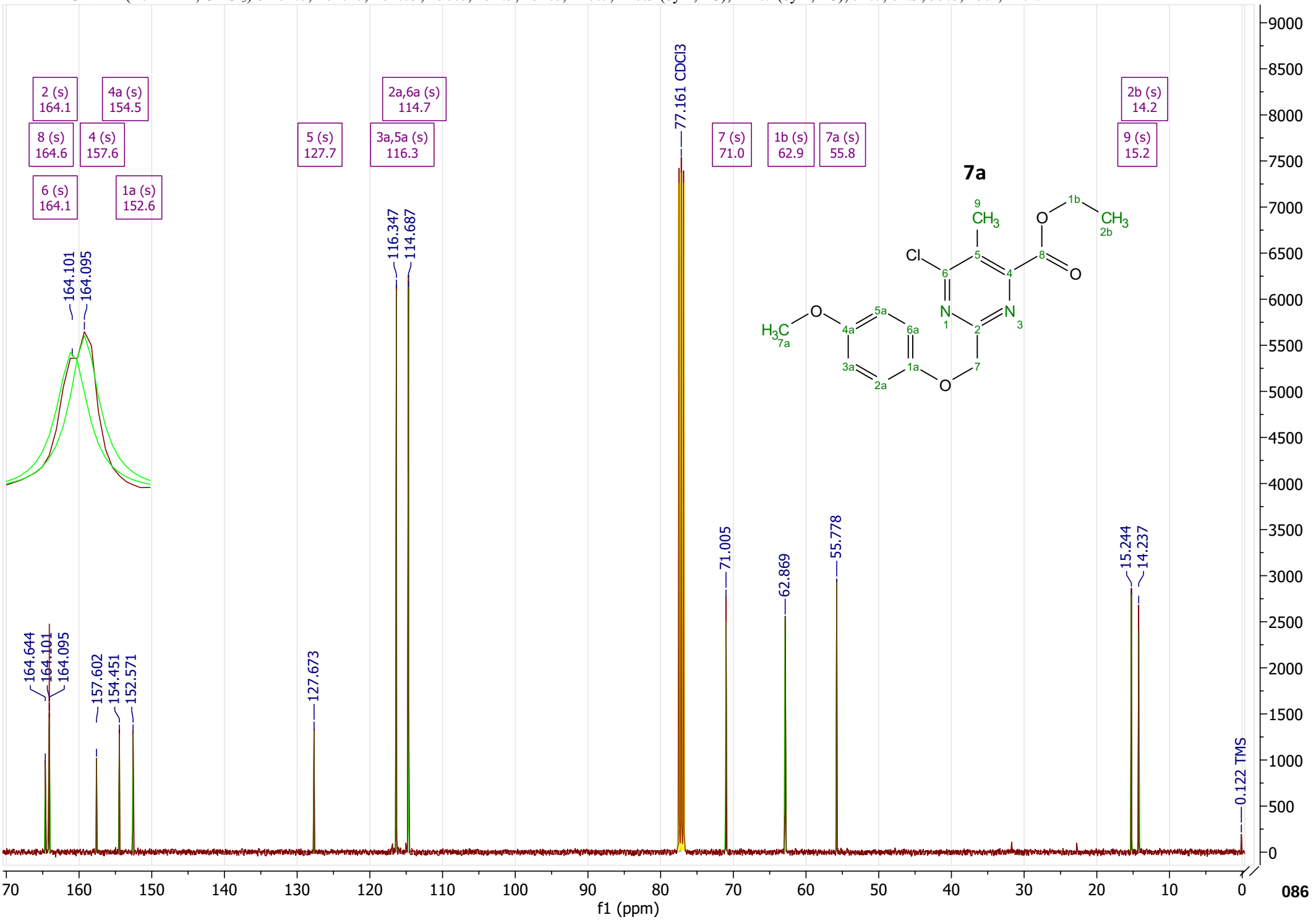
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -80.12, -99.68.



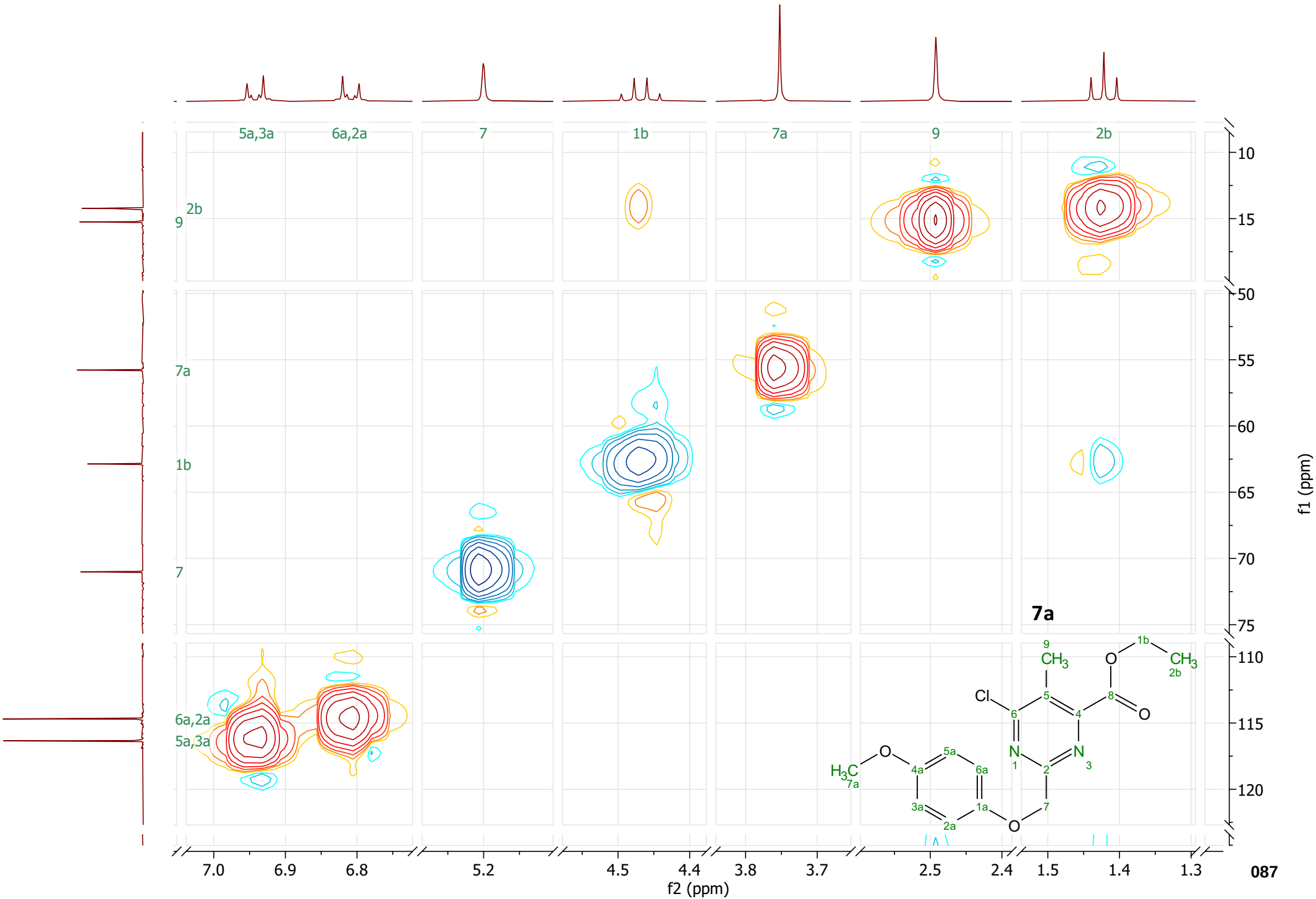
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 – 6.90 (m, 2H), 6.90 – 6.71 (m, 2H), 5.20 (s, 2H), 4.47 (q, *J* = 7.2 Hz, 2H), 3.75 (s, 3H), 2.49 (s, 3H), 1.42 (t, *J* = 7.1 Hz, 3H).



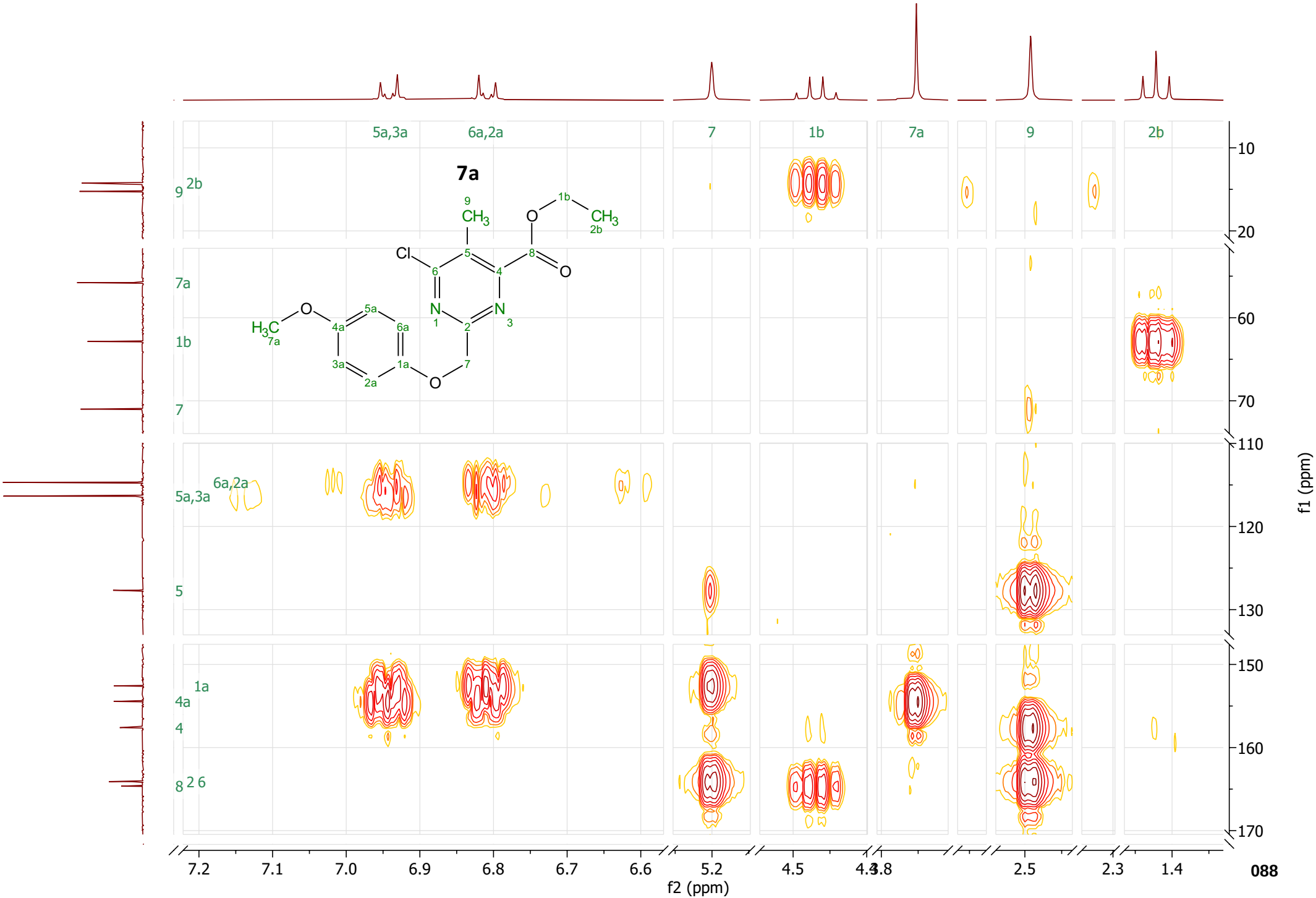
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.6, 164.10, 164.09, 157.6, 154.5, 152.6, 127.7, 116.3 (sym, 2C), 114.7 (sym, 2C), 71.0, 62.9, 55.8, 15.2, 14.2.



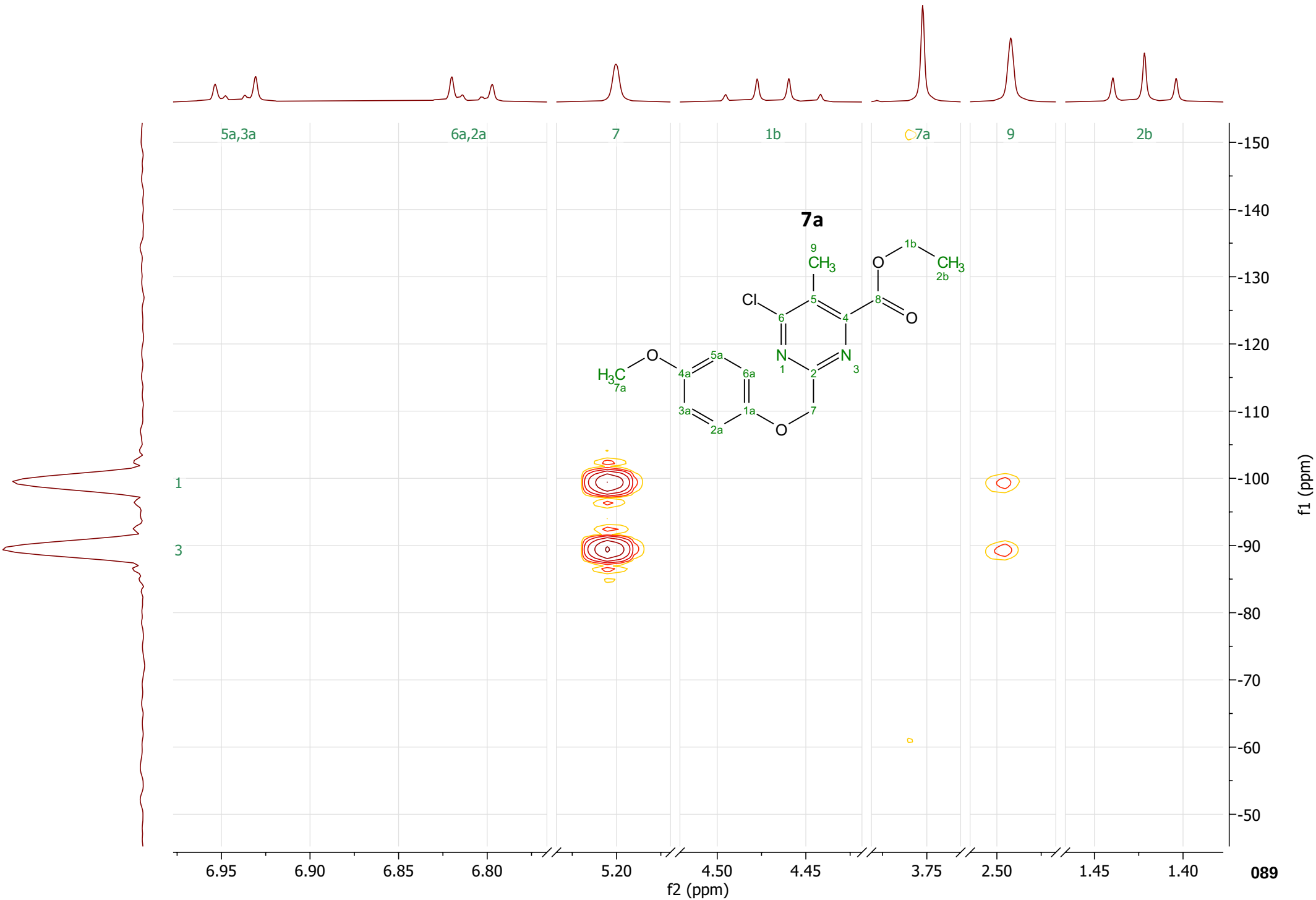
13C HSQC


5a,3a
6a,2a
7
1b
7a
9
2b
9
2b
7a
1b
7
6a,2a
5a,3a
7a
f1 (ppm)
f2 (ppm)
7.0
6.9
6.8
5.2
4.5
4.4
3.8
3.7
2.5
2.4
1.5
1.4
1.3
10
15
50
55
60
65
70
75
110
115
120
087

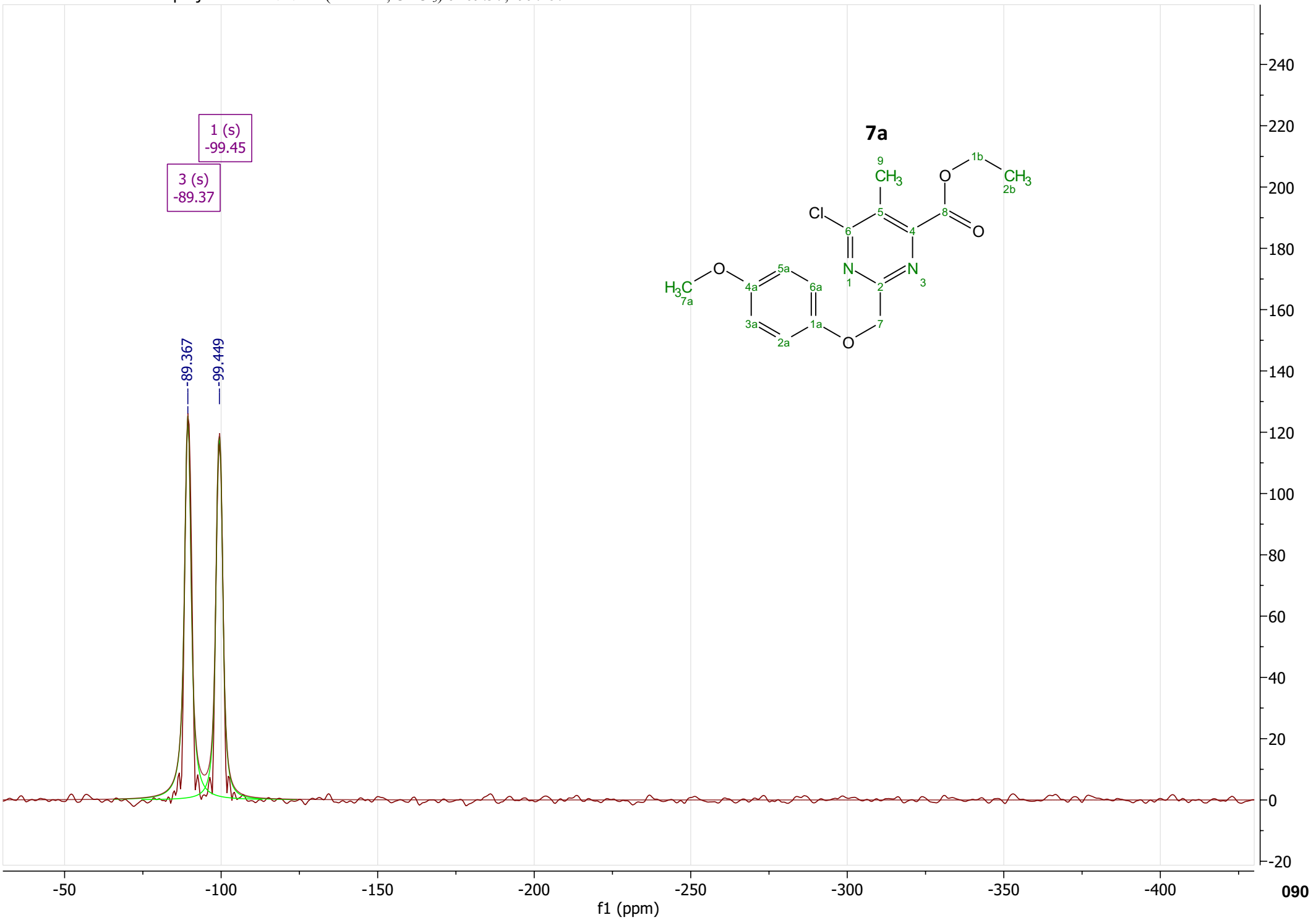
13C HMBC



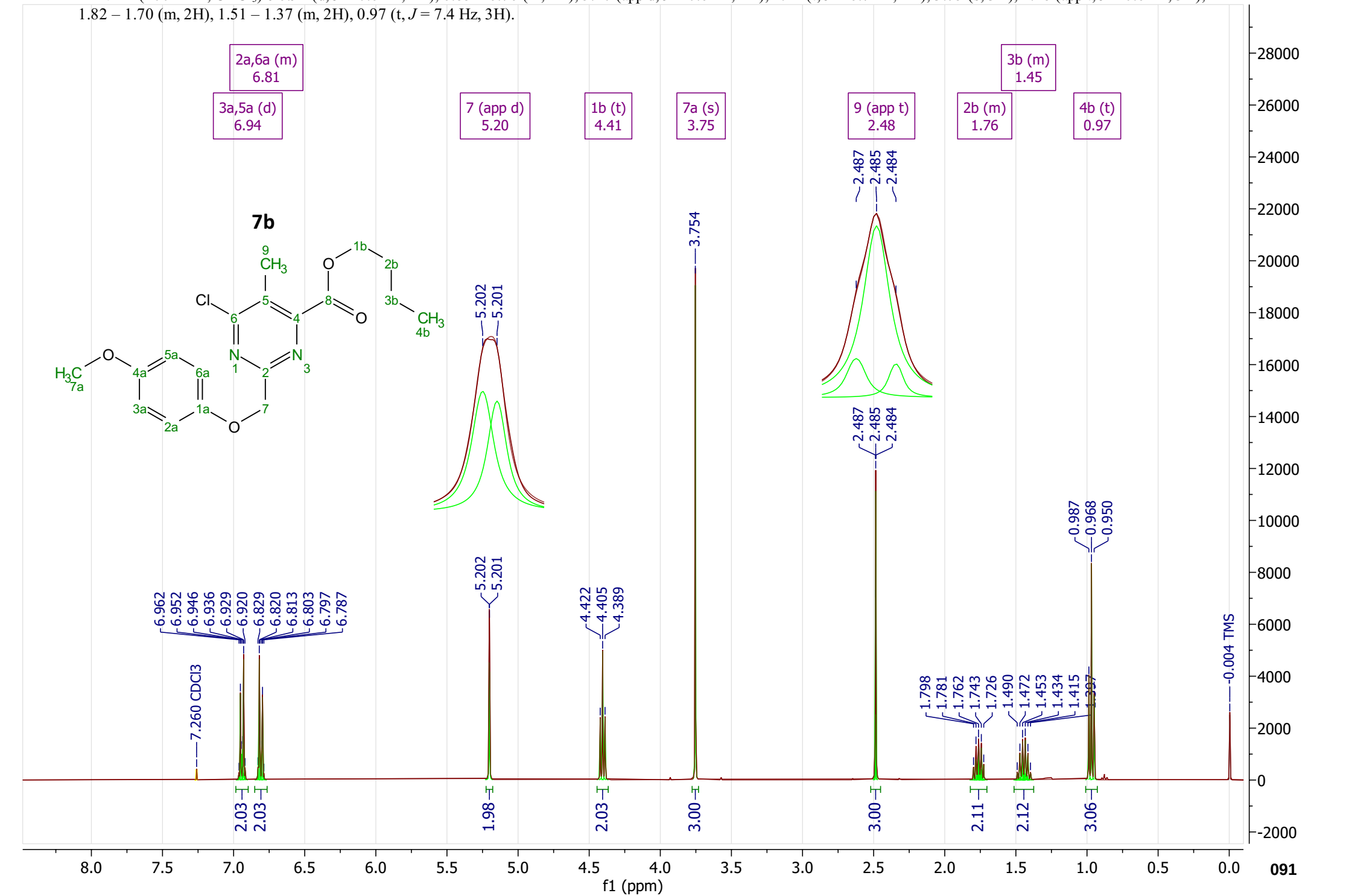
15N HMBC


5a,3a
6a,2a
7
1b
7a
9
2b
7a
1
3
-150
-140
-130
-120
-110
-100
-90
-80
-70
-60
-50
f1 (ppm)
6.95
6.90
6.85
6.80
5.20
4.50
4.45
3.75
2.50
1.45
1.40
f2 (ppm)
089

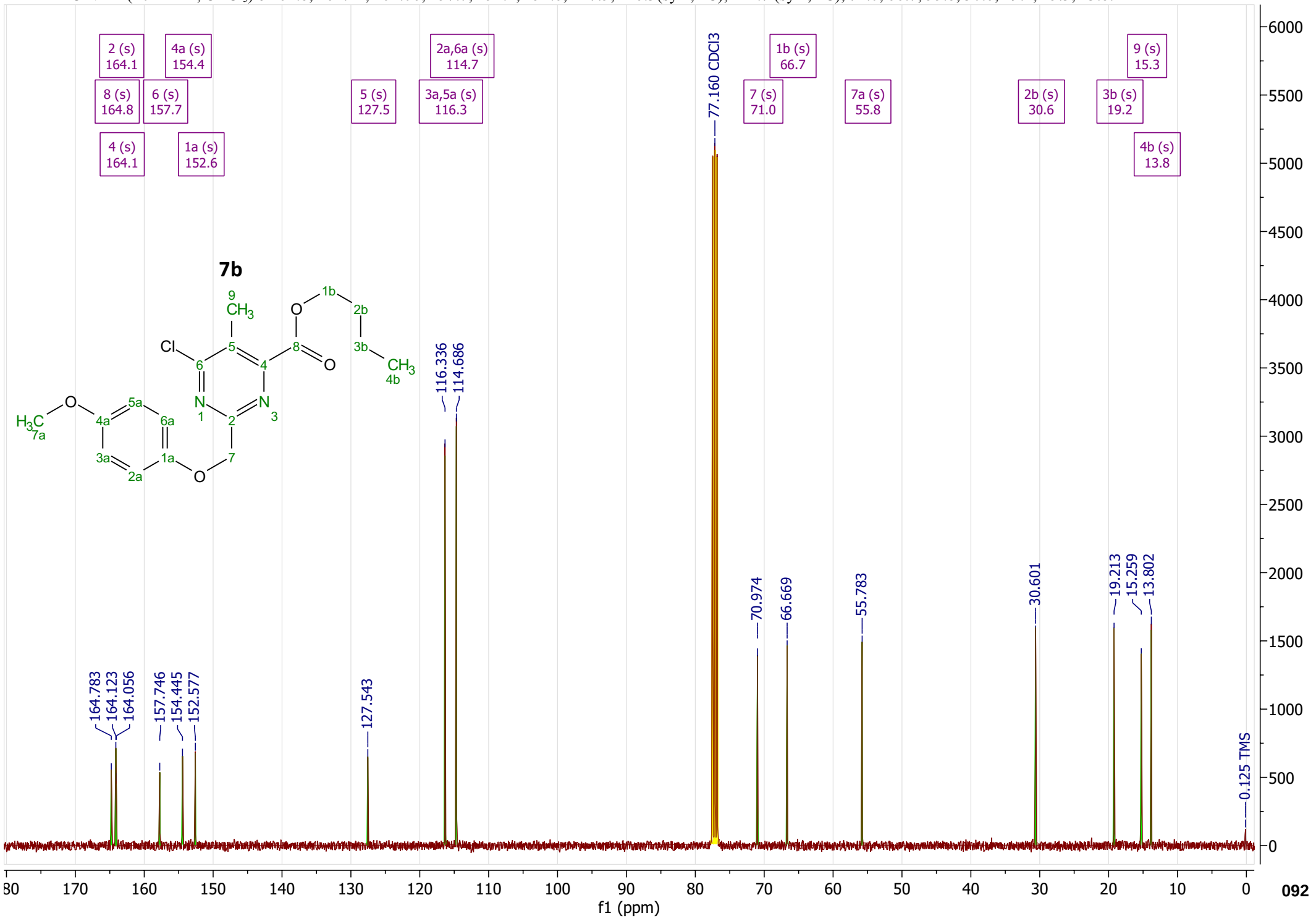
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -89.37, -99.45.



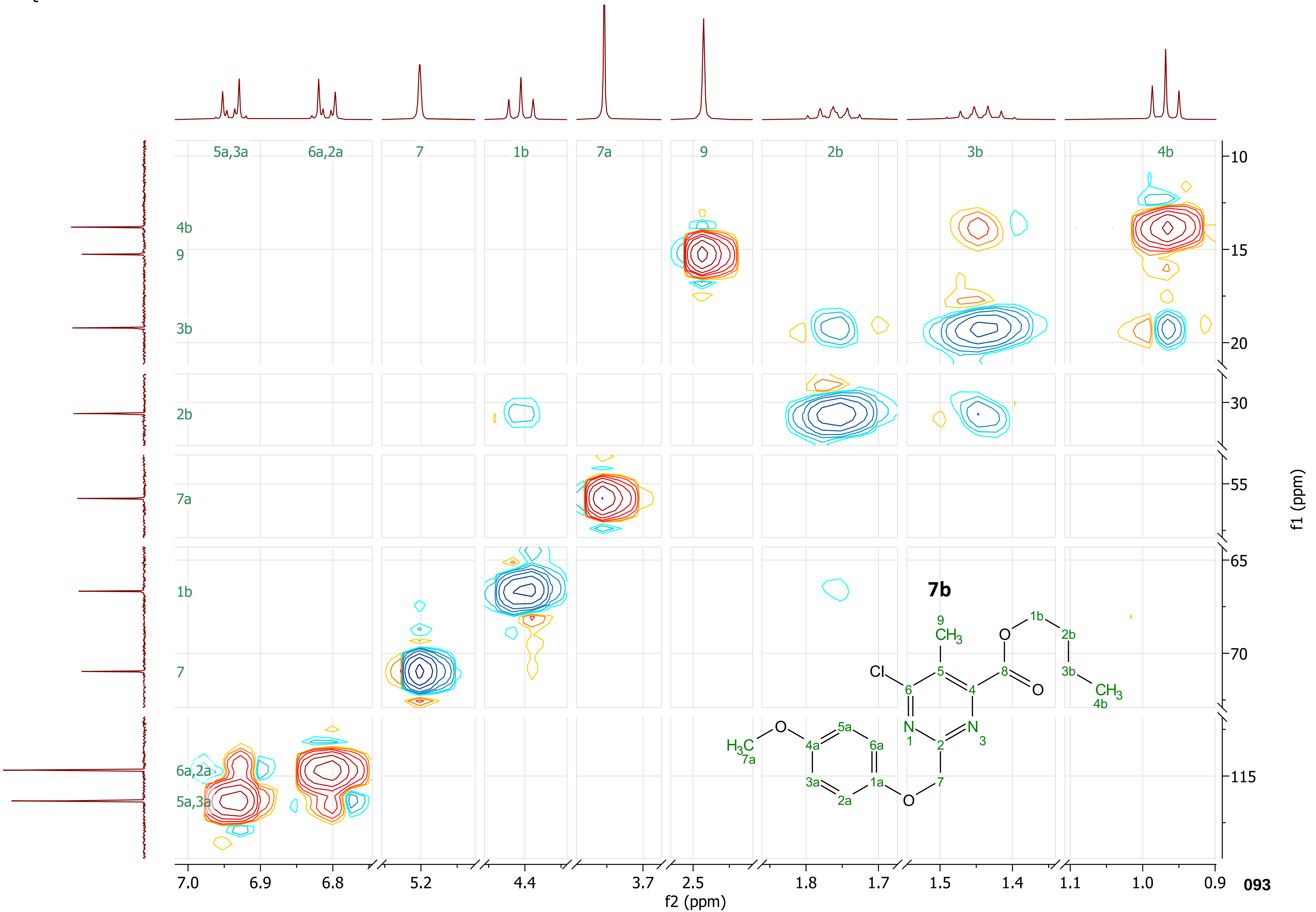
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.94 (d, J = 0.6 Hz, 2H), 6.85 – 6.76 (m, 2H), 5.20 (app d, J = 0.6 Hz, 2H), 4.41 (t, J = 6.7 Hz, 2H), 3.75 (s, 3H), 2.48 (app t, J = 0.6 Hz, 3H), 1.82 – 1.70 (m, 2H), 1.51 – 1.37 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H).



13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 164.12, 164.06, 157.7, 154.4, 152.6, 127.5, 116.3(sym, 2C), 114.7 (sym, 2C), 71.0, 66.7, 55.8, 30.6, 19.2, 15.3, 13.8.

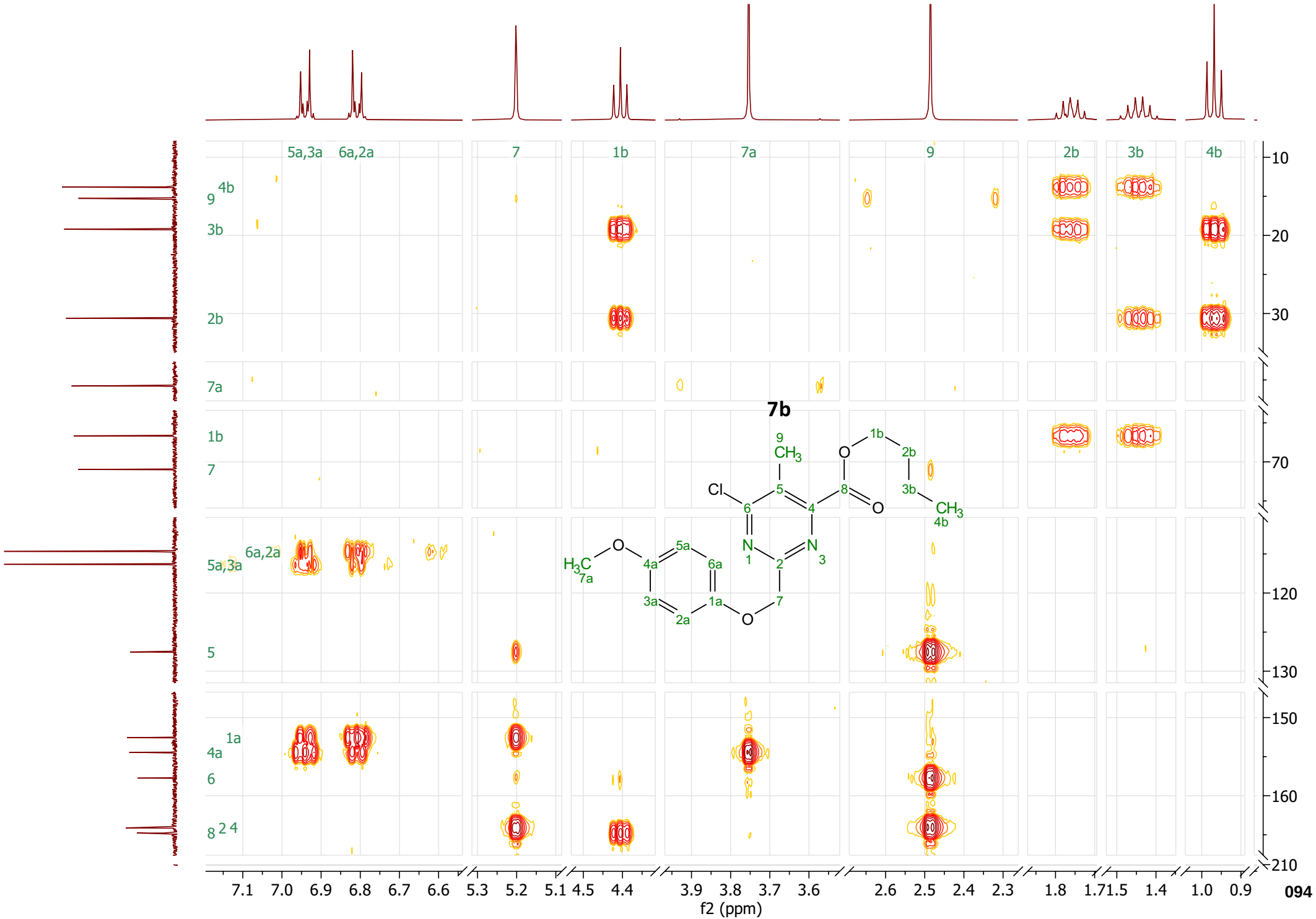


13C HSQC

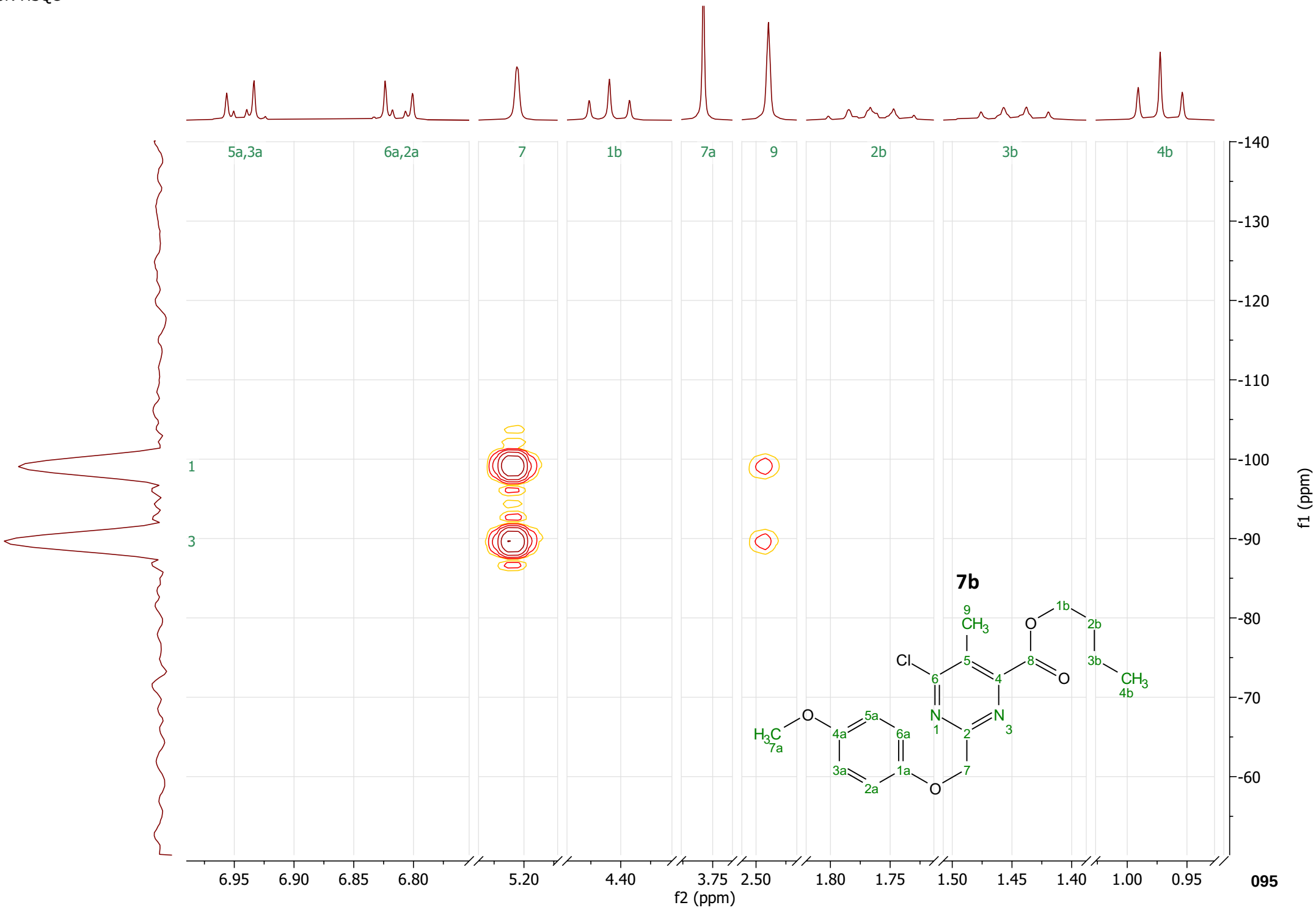


5a,3a
6a,2a
7
1b
7a
9
2b
3b
4b
6a,2a
5a,3a
7b
9
CH3
O
1b
2b
Cl
5
8
4
6
3b
O
CH3
4b
N
1
2
N
3
H3C
7a
O
4a
5a
6a
3a
1a
2a
O
7
7.0
6.9
6.8
5.2
4.4
3.7
2.5
1.8
1.7
1.5
1.4
1.1
1.0
0.9
f2 (ppm)
10
15
20
30
55
65
70
115
f1 (ppm)

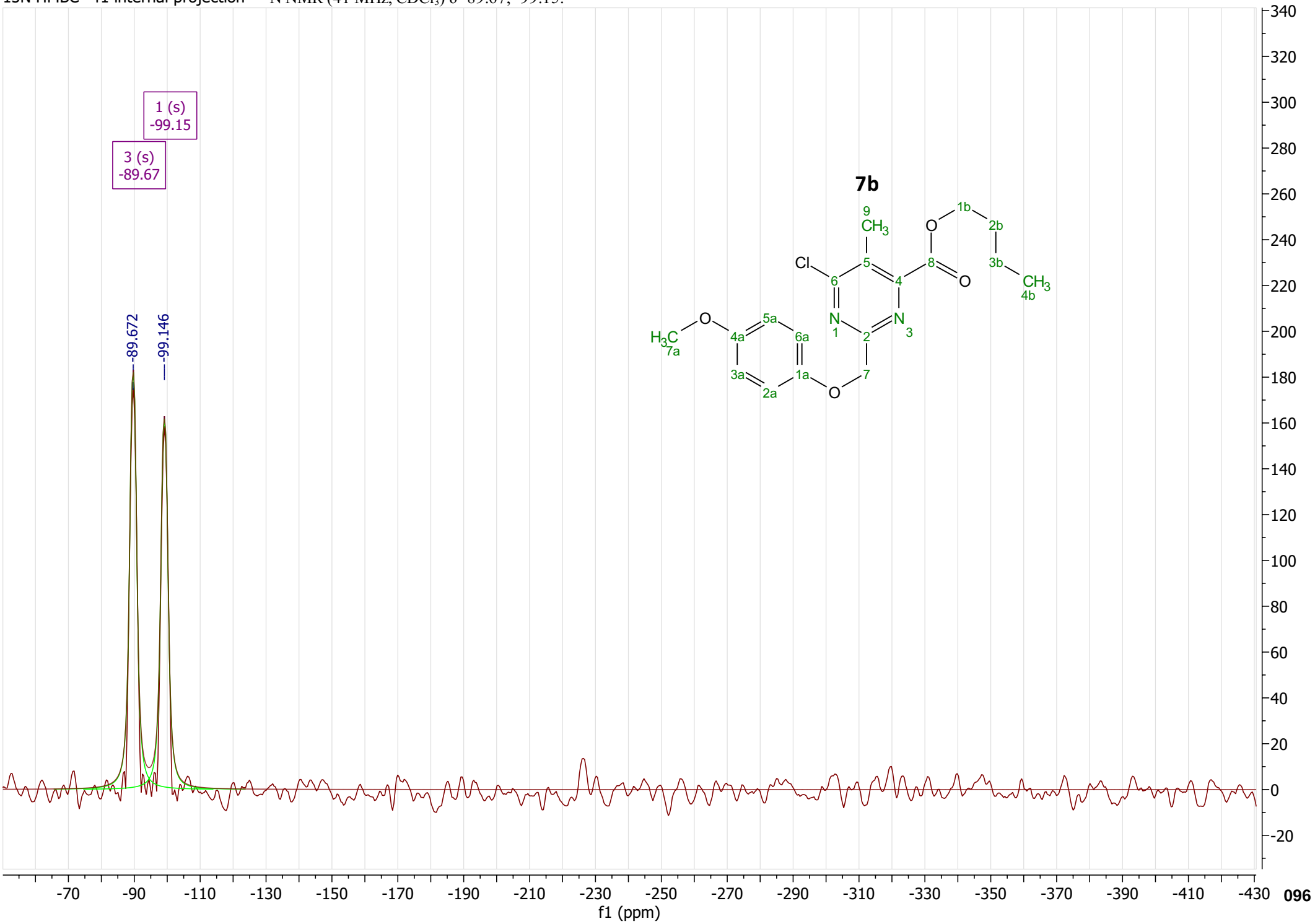
13C HMBC



15N HSQC

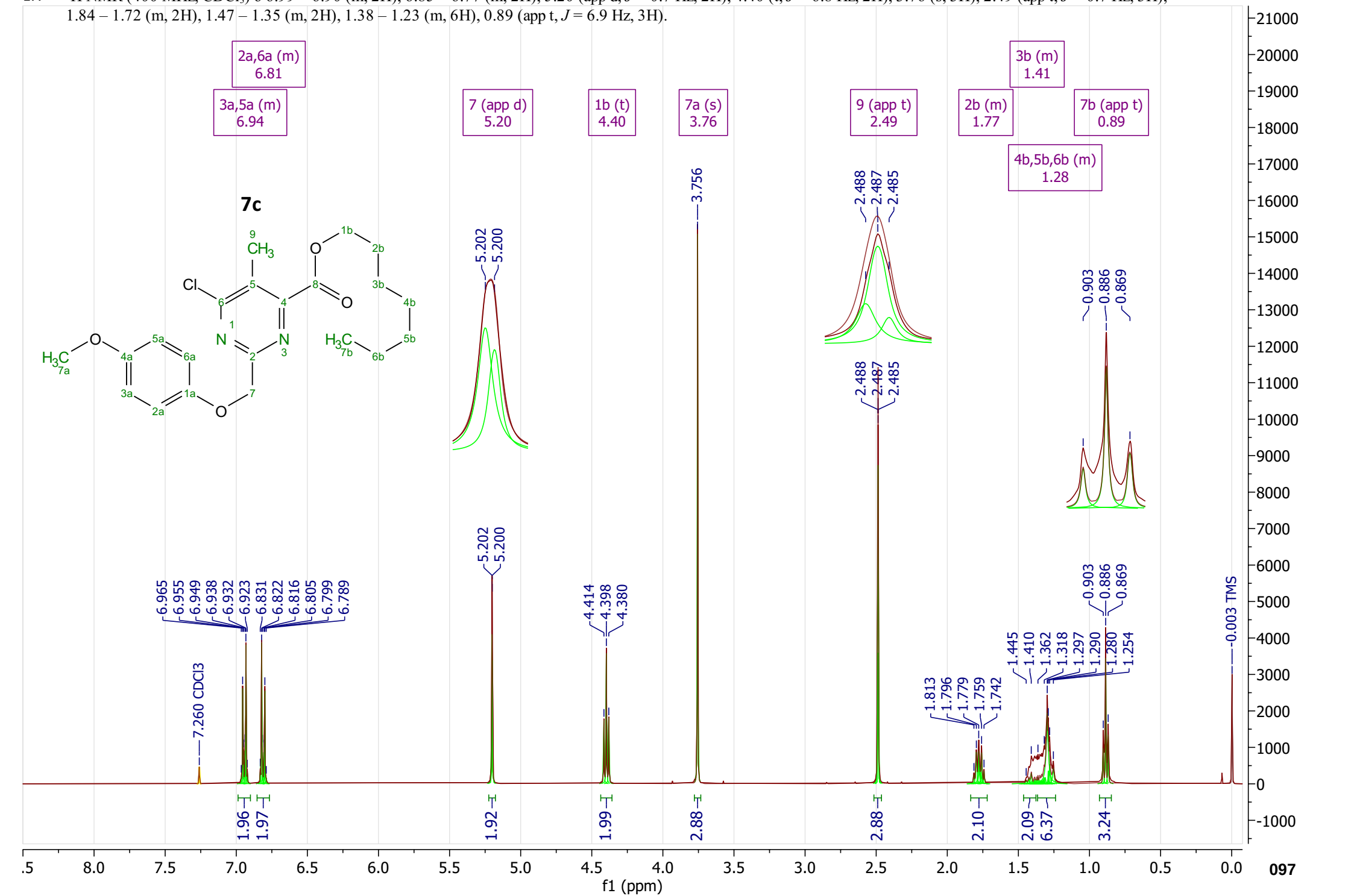

5a,3a
6a,2a
7
1b
7a
9
2b
3b
4b
1
3
-140
-130
-120
-110
-100
-90
-80
-70
-60
f1 (ppm)
6.95
6.90
6.85
6.80
5.20
4.40
3.75
2.50
1.80
1.75
1.50
1.45
1.40
1.00
0.95
f2 (ppm)
7b
095

15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -89.67, -99.15.

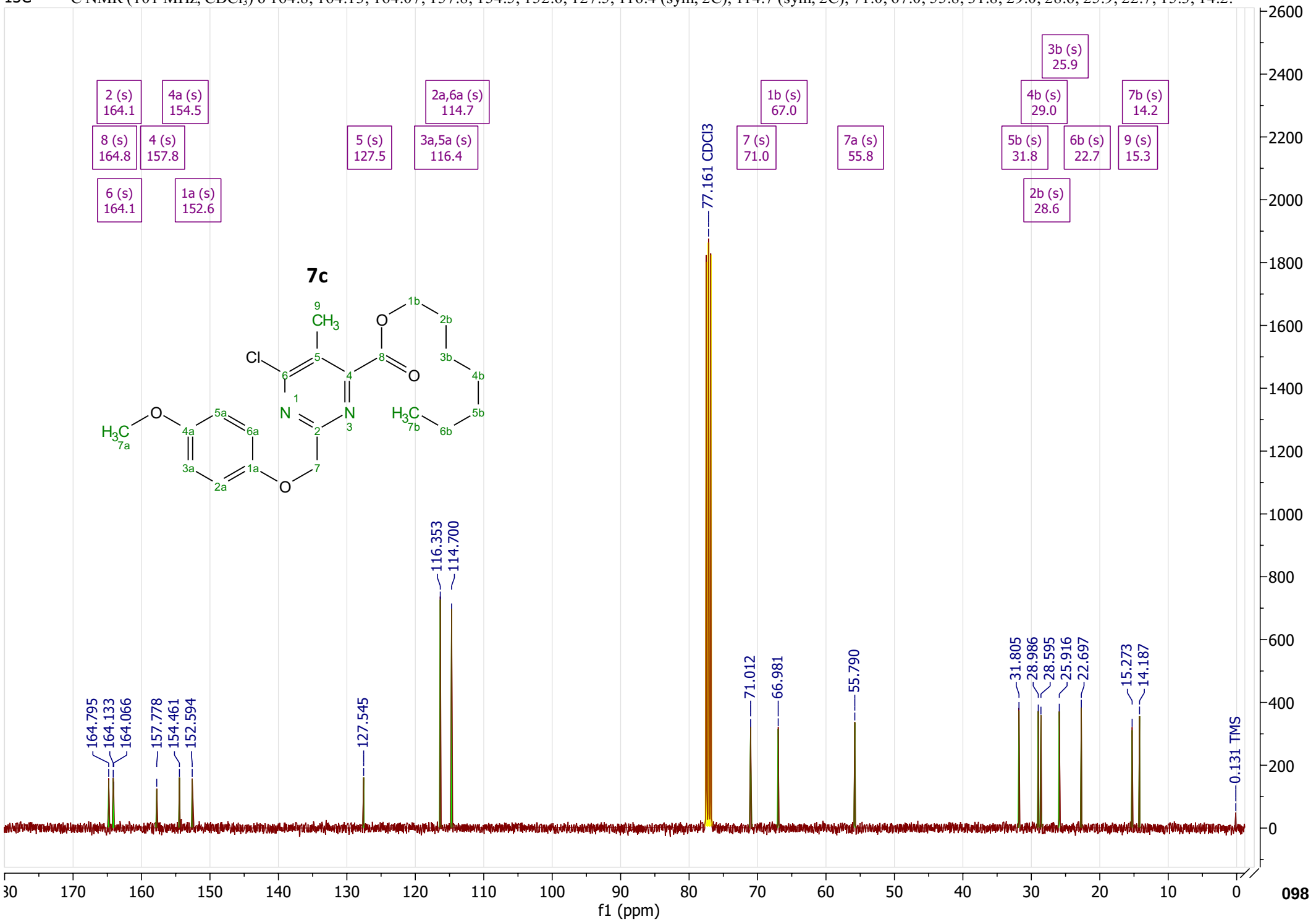


096

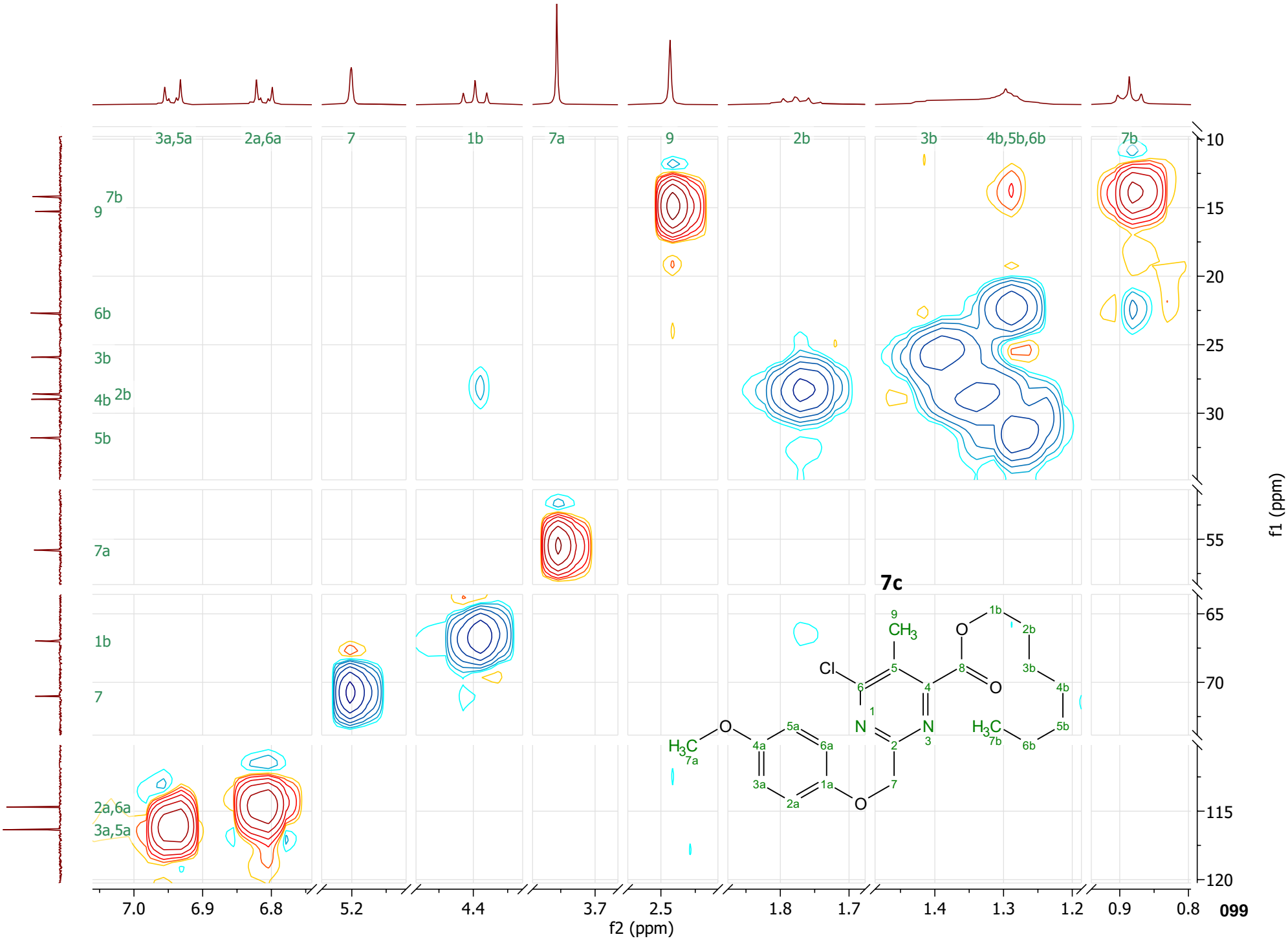
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 – 6.90 (m, 2H), 6.85 – 6.77 (m, 2H), 5.20 (app d, *J* = 0.7 Hz, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.76 (s, 3H), 2.49 (app t, *J* = 0.7 Hz, 3H), 1.84 – 1.72 (m, 2H), 1.47 – 1.35 (m, 2H), 1.38 – 1.23 (m, 6H), 0.89 (app t, *J* = 6.9 Hz, 3H).



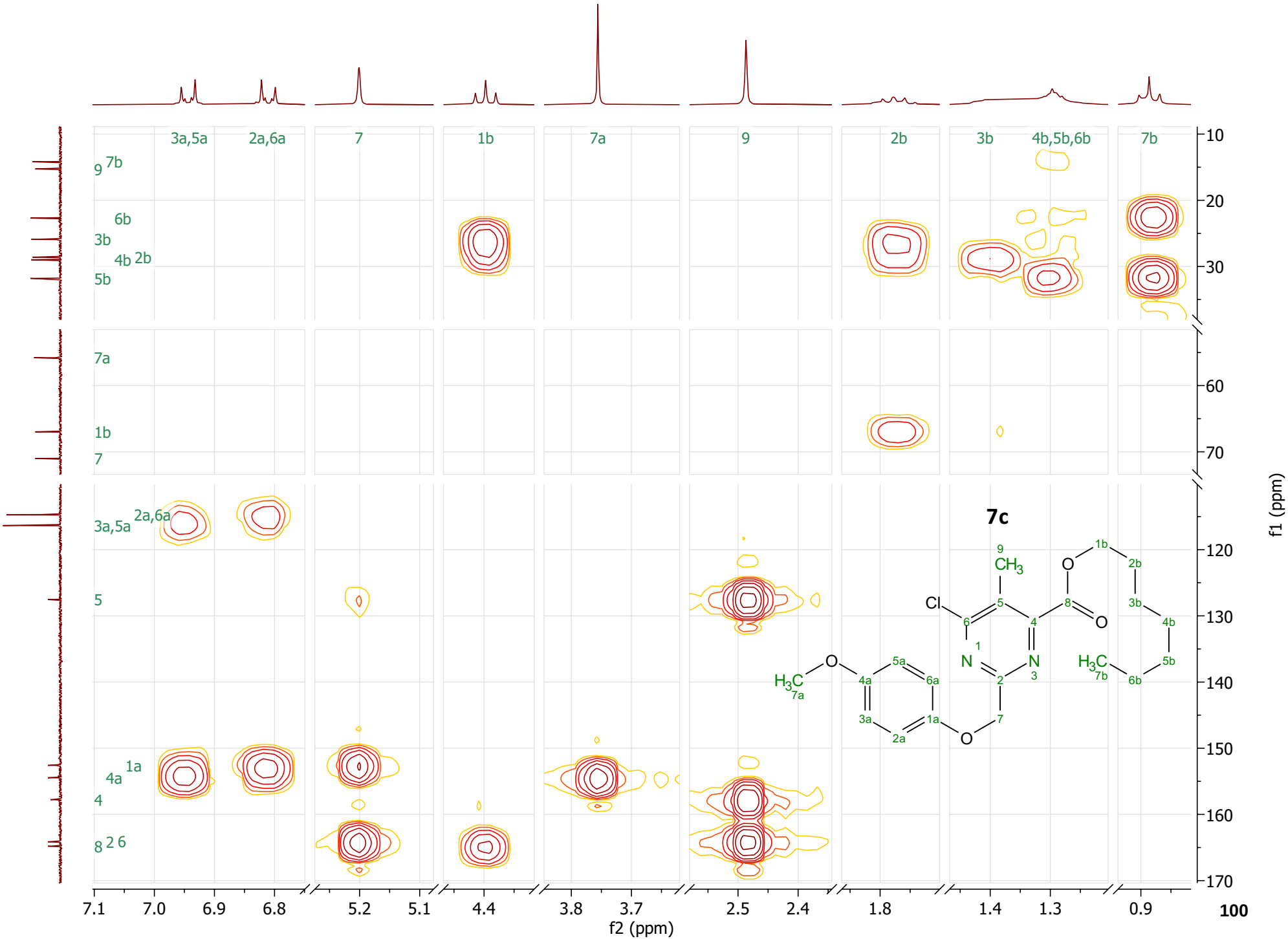
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 164.13, 164.07, 157.8, 154.5, 152.6, 127.5, 116.4 (sym, 2C), 114.7 (sym, 2C), 71.0, 67.0, 55.8, 31.8, 29.0, 28.6, 25.9, 22.7, 15.3, 14.2.



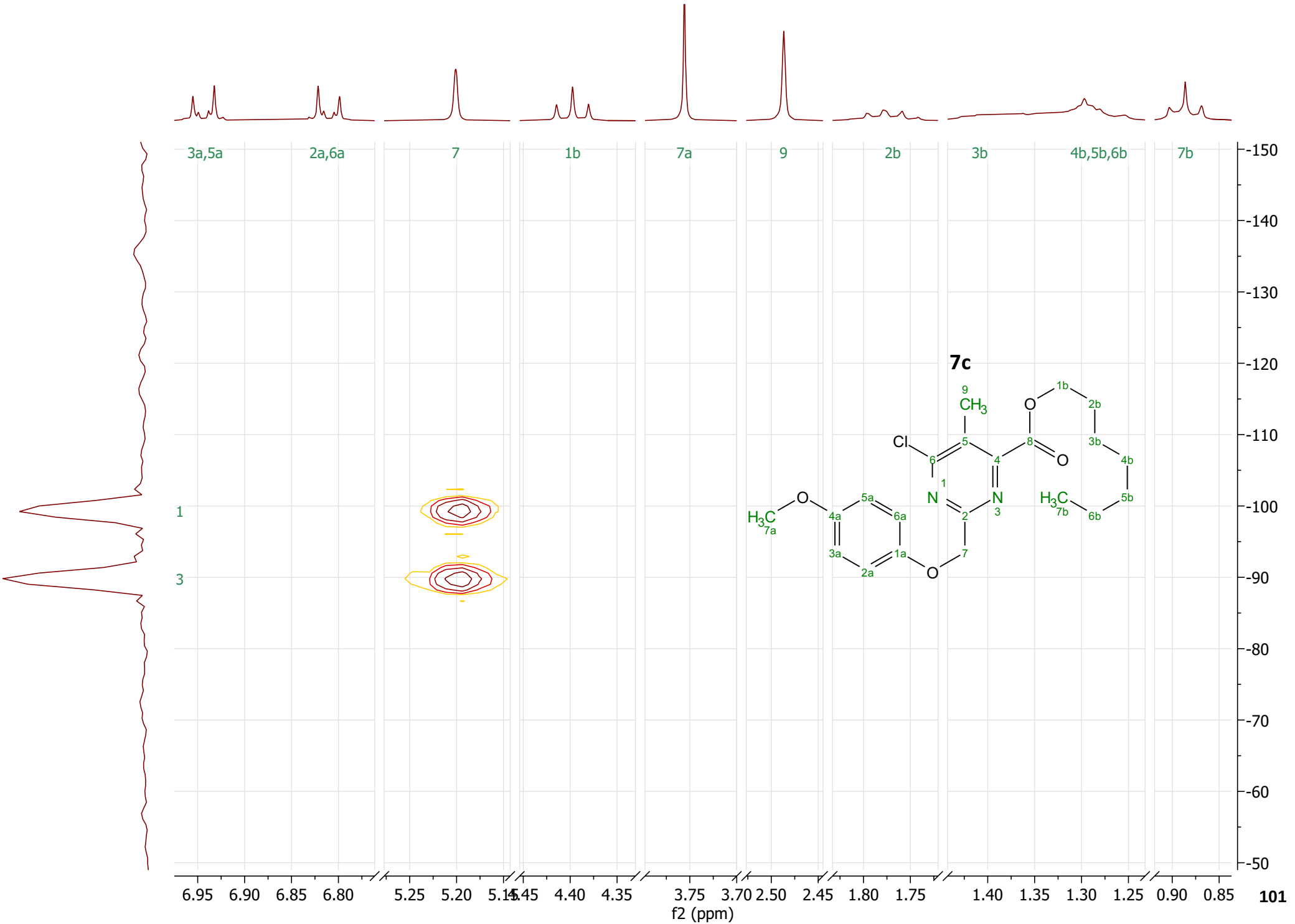
13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b
7b
9
7b
6b
3b
4b
2b
5b
7a
1b
7
2a,6a
3a,5a
7c
f2 (ppm)
f1 (ppm)
099

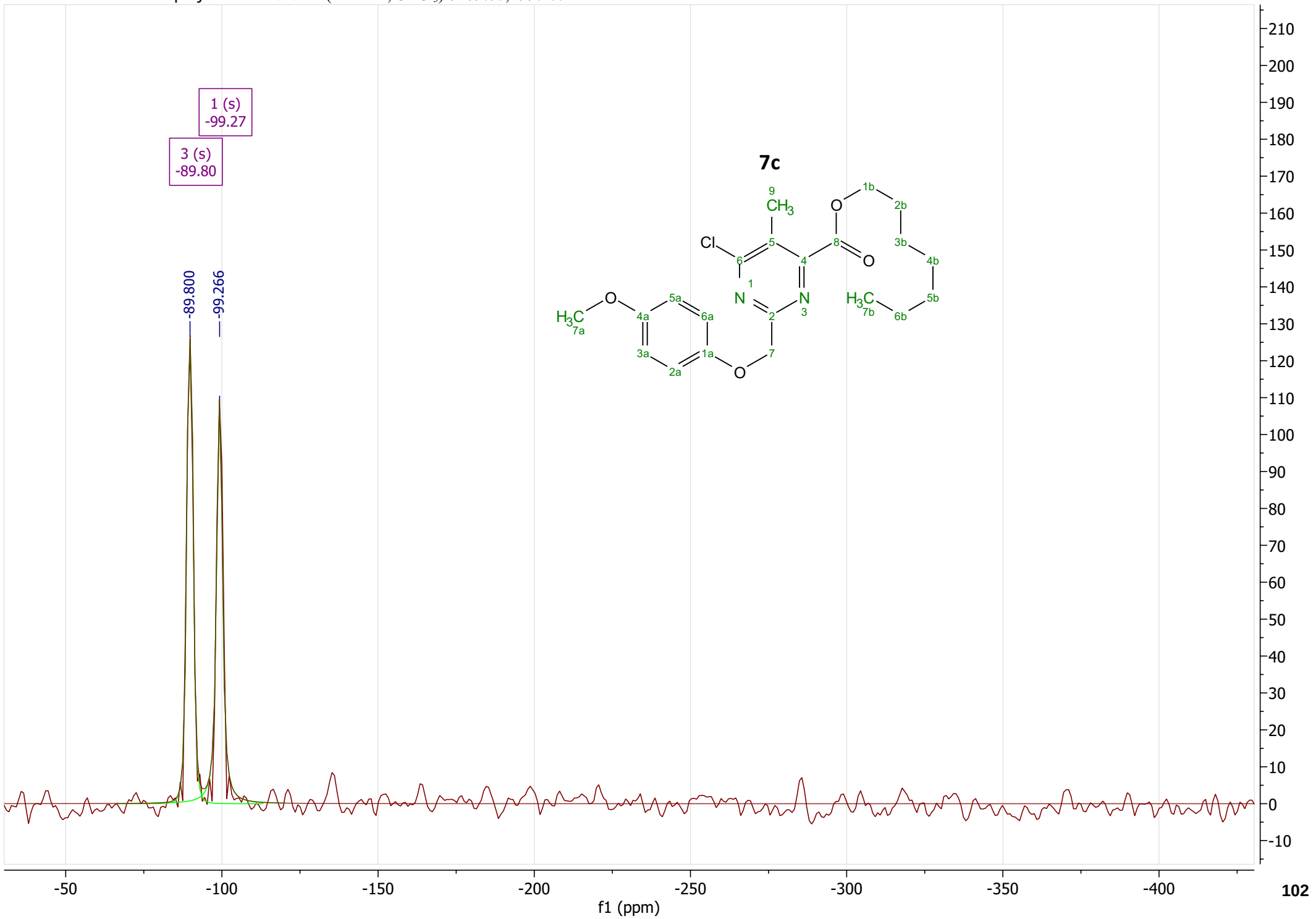
13C HMBC



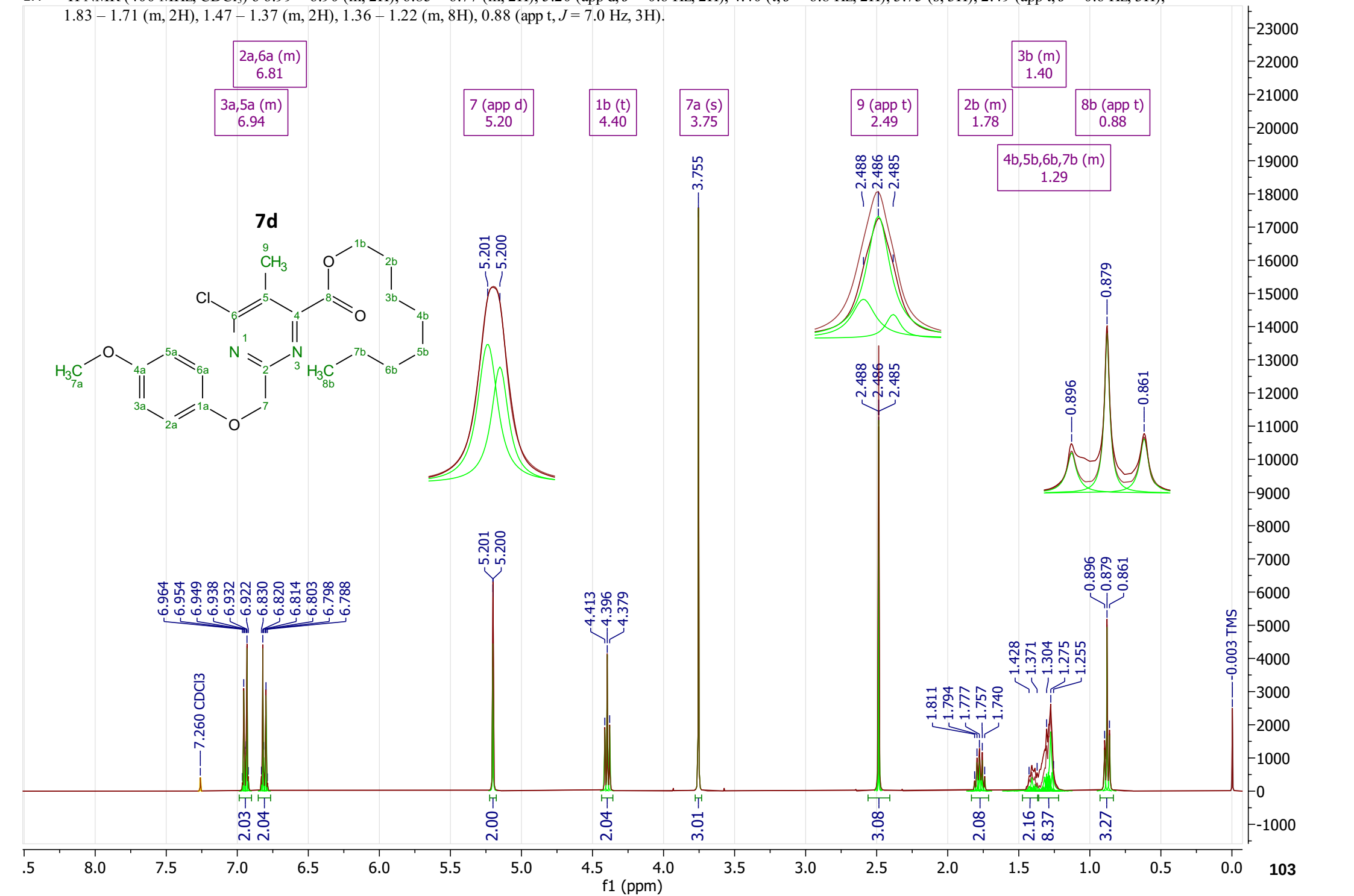
15N HMBC



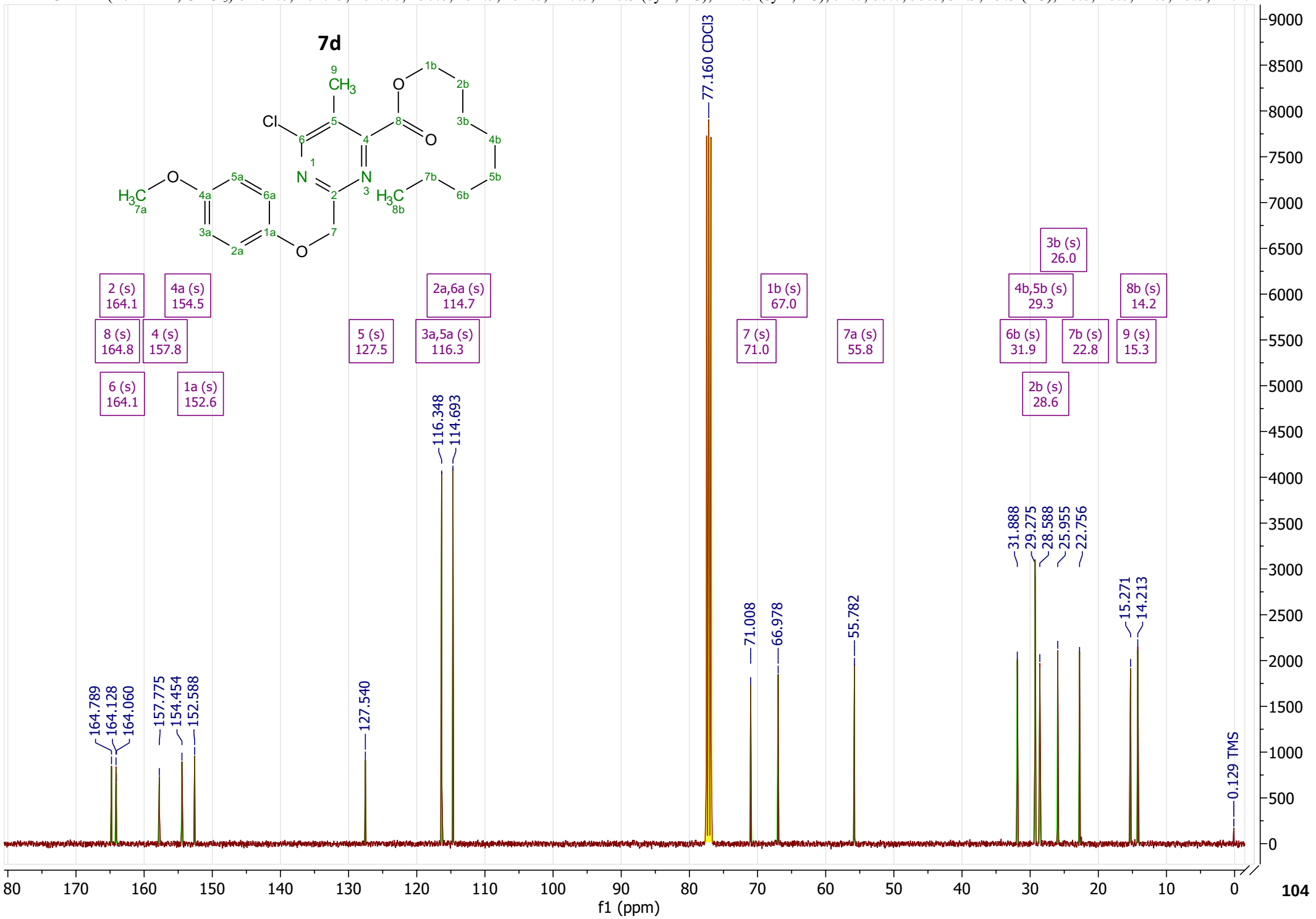
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -89.80, -99.27.



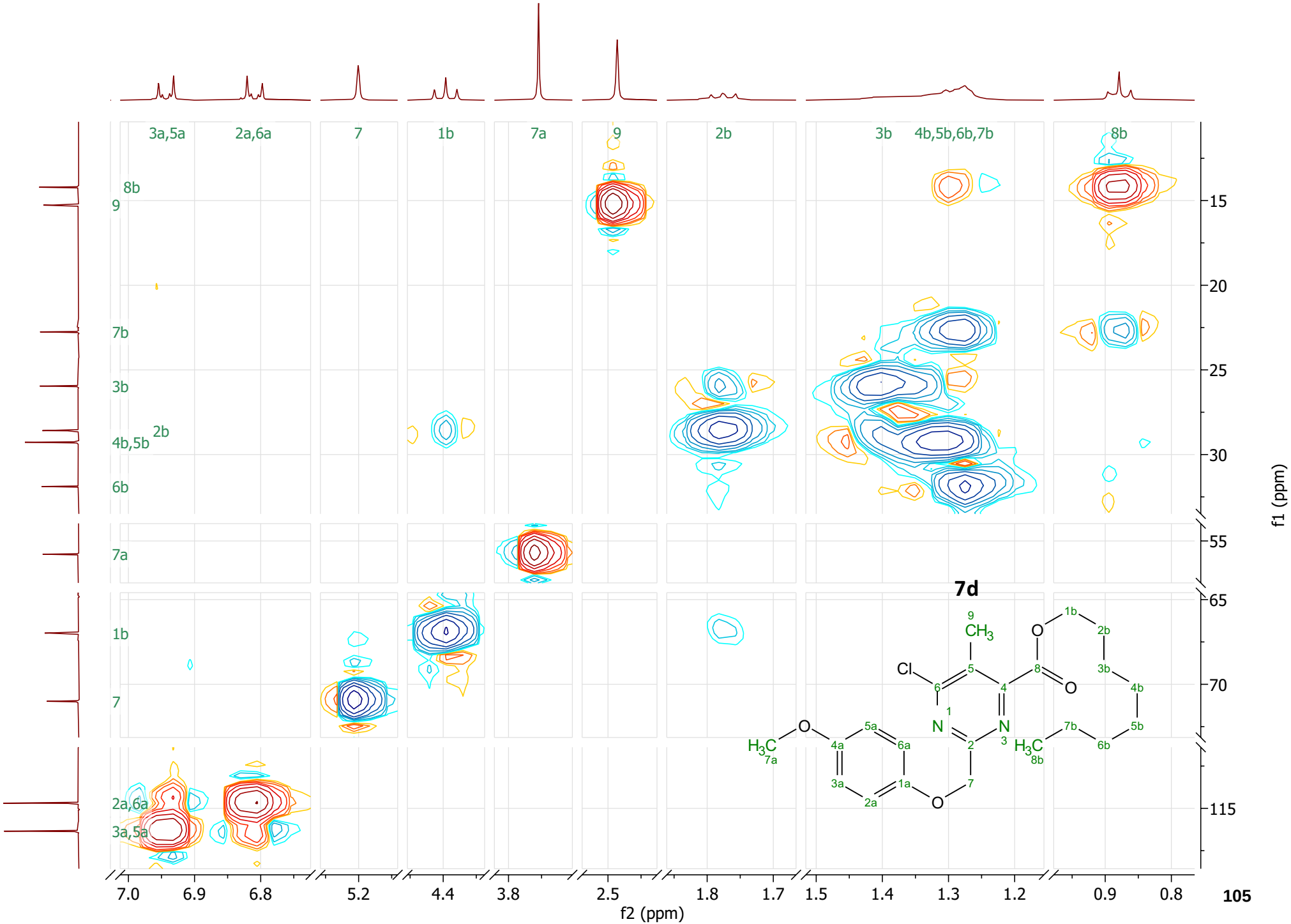
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 – 6.90 (m, 2H), 6.85 – 6.77 (m, 2H), 5.20 (app d, *J* = 0.6 Hz, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.75 (s, 3H), 2.49 (app t, *J* = 0.6 Hz, 3H), 1.83 – 1.71 (m, 2H), 1.47 – 1.37 (m, 2H), 1.36 – 1.22 (m, 8H), 0.88 (app t, *J* = 7.0 Hz, 3H).



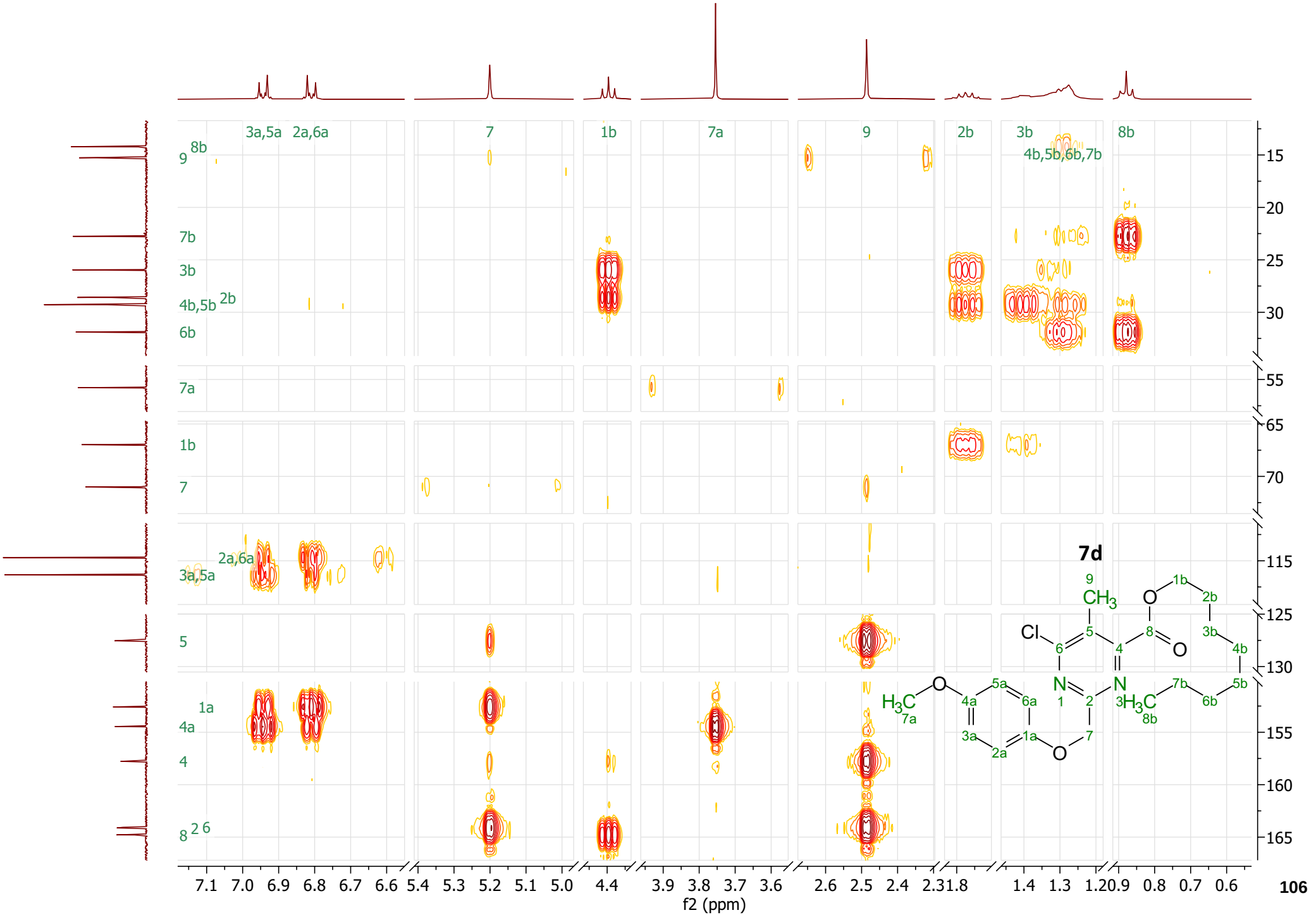
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 164.13, 164.06, 157.8, 154.5, 152.6, 127.5, 116.3 (sym, 2C), 114.7 (sym, 2C), 71.0, 67.0, 55.8, 31.9, 29.3 (2C), 28.6, 26.0, 22.8, 15.3, 14.2.



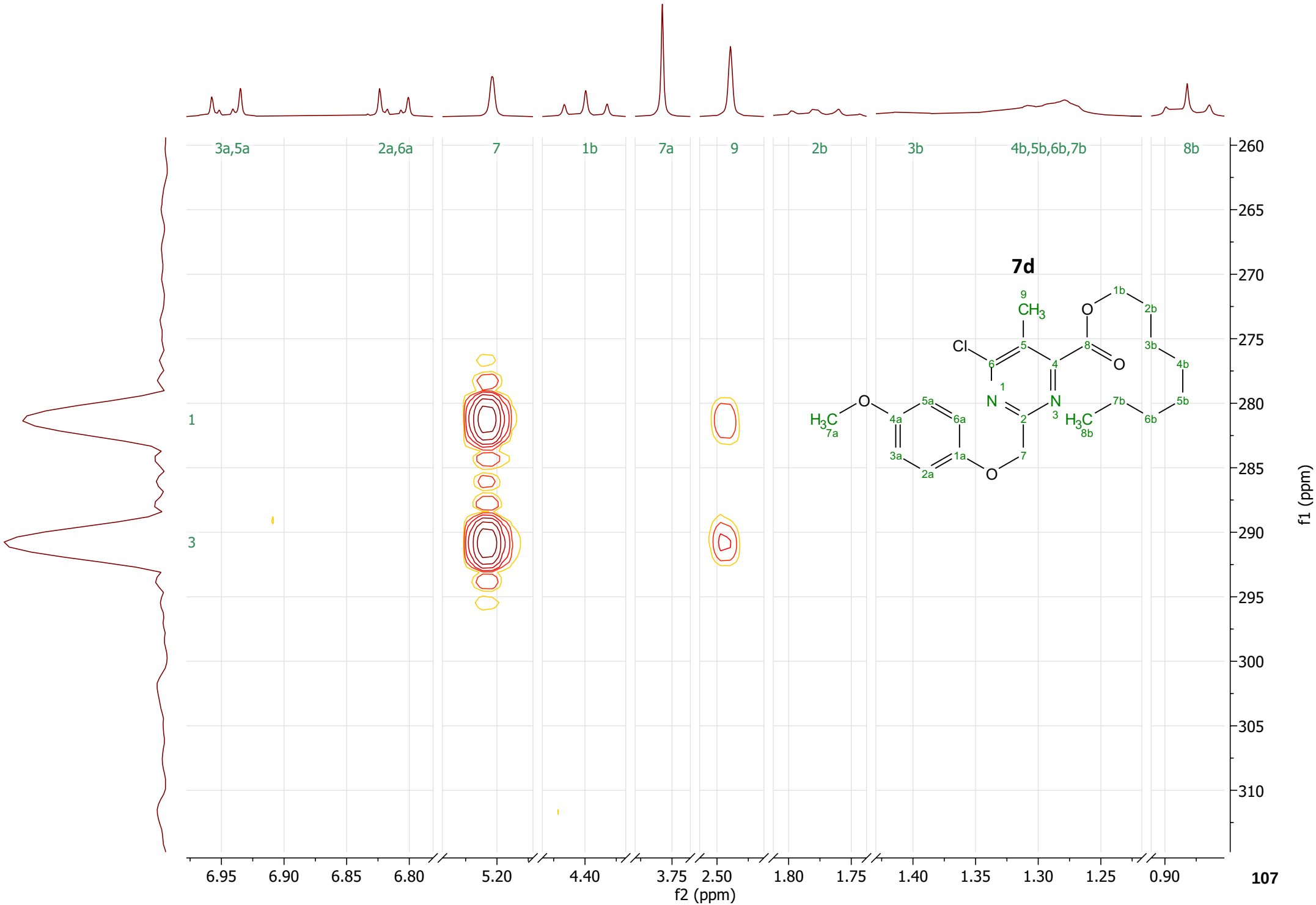
13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
8b
9
7b
3b
4b,5b
2b
6b
7a
1b
7
2a,6a
3a,5a
7d
f1 (ppm)
f2 (ppm)
7.0
6.9
6.8
5.2
4.4
3.8
2.5
1.8
1.7
1.5
1.4
1.3
1.2
0.9
0.8
15
20
25
30
55
65
70
115

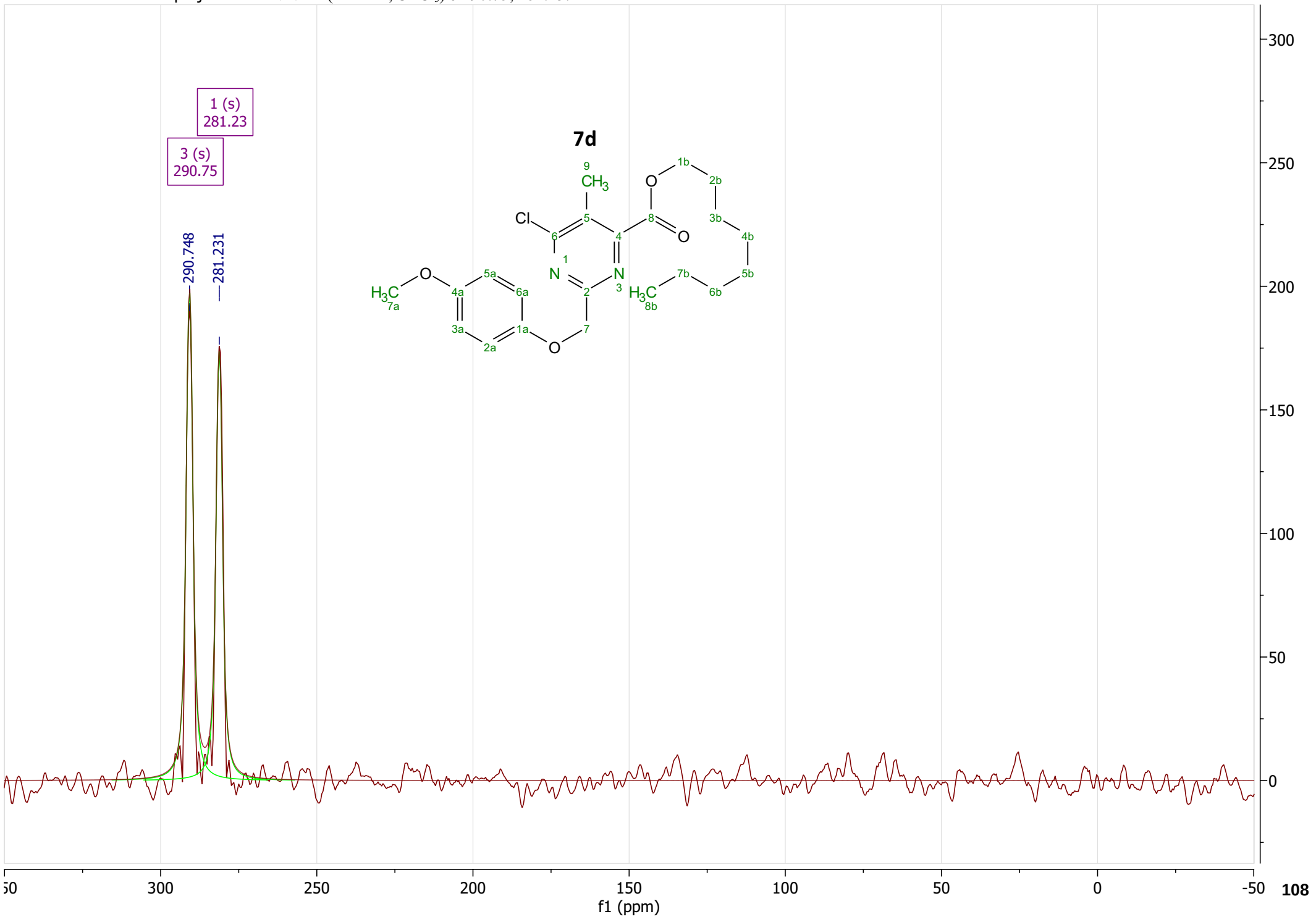
13C HMBC



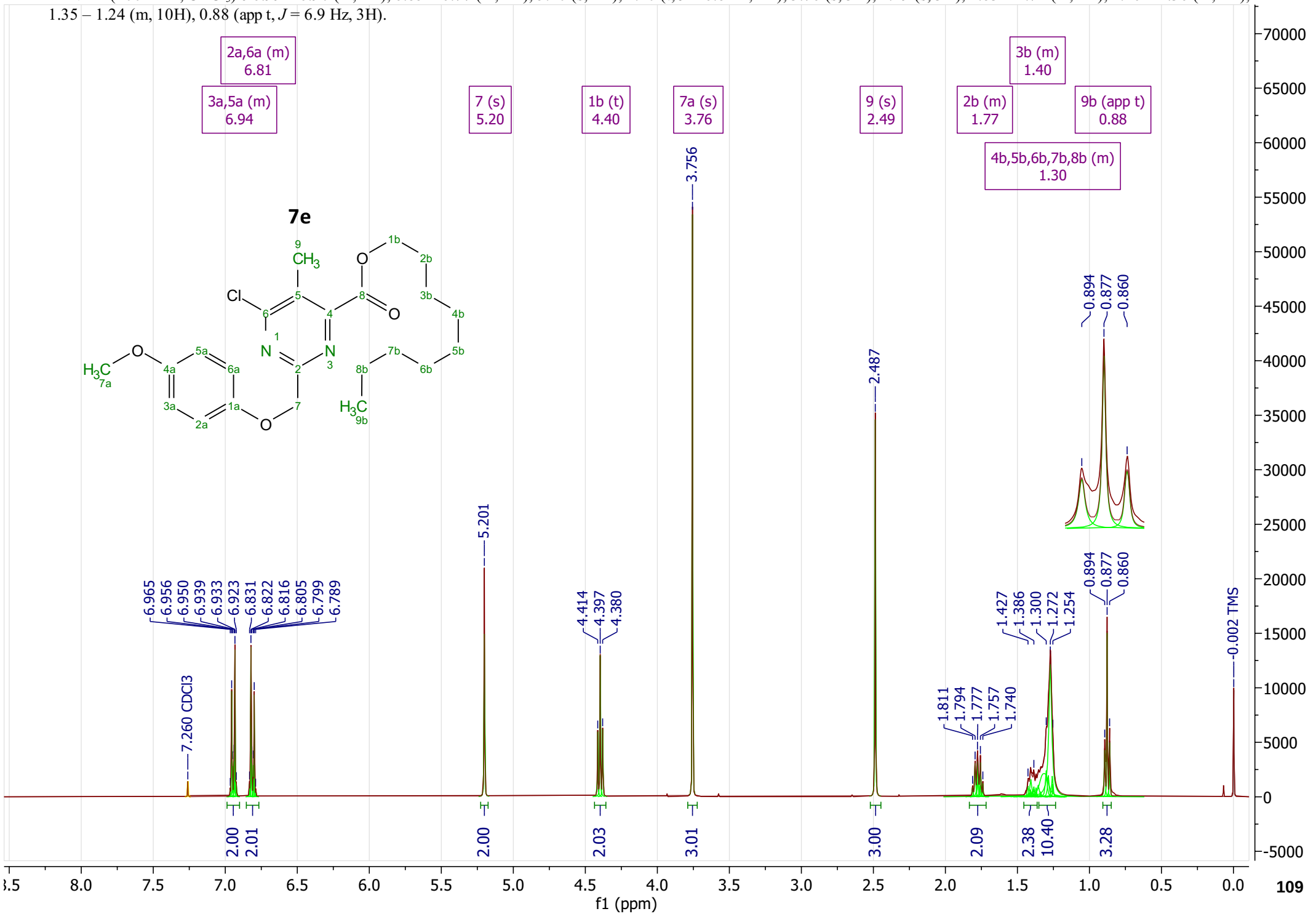
15N HMBC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
1
3
7d
f2 (ppm)
f1 (ppm)
6.95
6.90
6.85
6.80
5.20
4.40
3.75
2.50
1.80
1.75
1.40
1.35
1.30
1.25
0.90
260
265
270
275
280
285
290
295
300
305
310

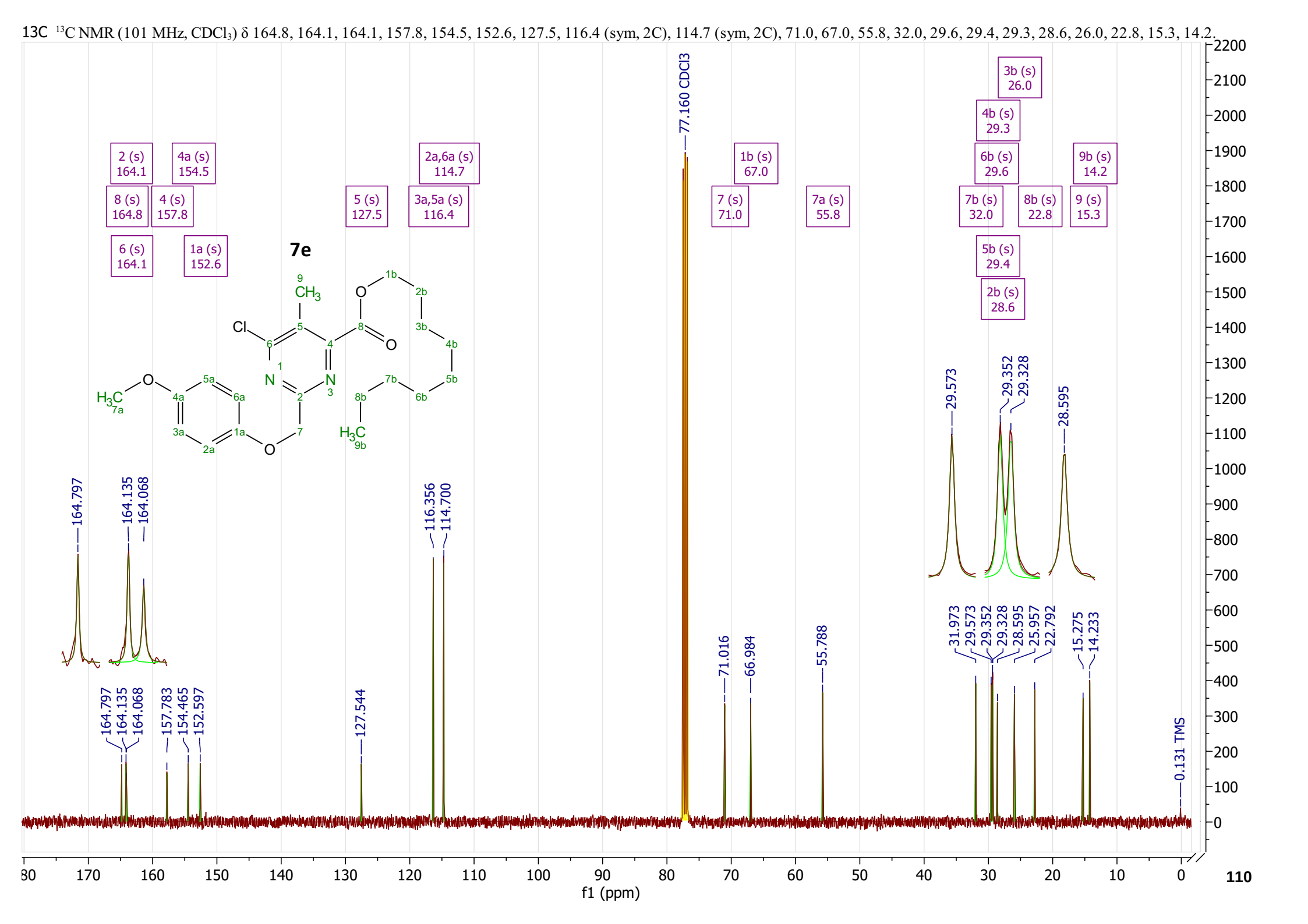
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ 290.75, 281.23.



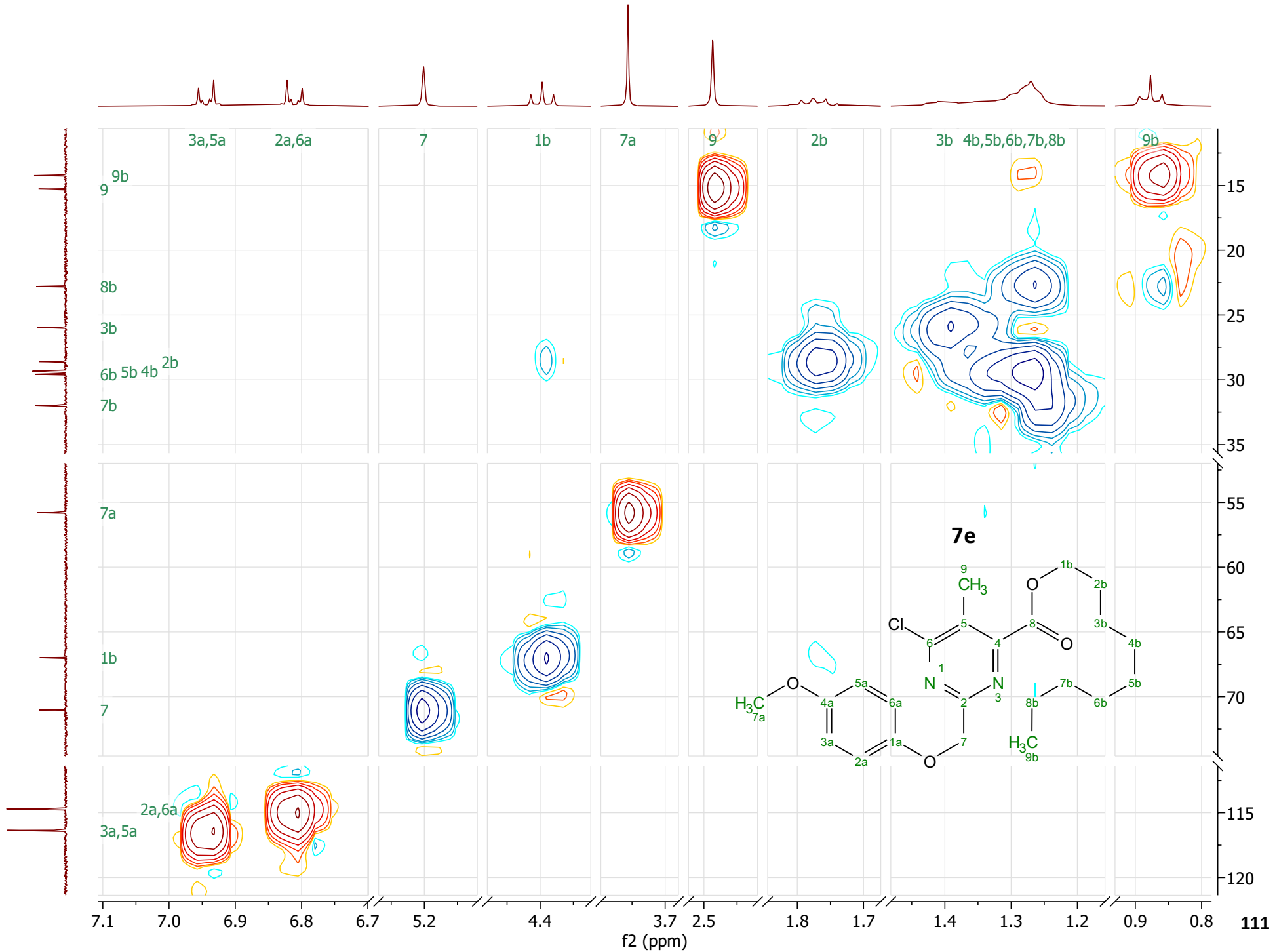
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 – 6.90 (m, 2H), 6.85 – 6.77 (m, 2H), 5.20 (s, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.76 (s, 3H), 2.49 (s, 3H), 1.83 – 1.72 (m, 2H), 1.46 – 1.36 (m, 2H), 1.35 – 1.24 (m, 10H), 0.88 (app t, *J* = 6.9 Hz, 3H).



13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.8, 164.1, 164.1, 157.8, 154.5, 152.6, 127.5, 116.4 (sym, 2C), 114.7 (sym, 2C), 71.0, 67.0, 55.8, 32.0, 29.6, 29.4, 29.3, 28.6, 26.0, 22.8, 15.3, 14.2.

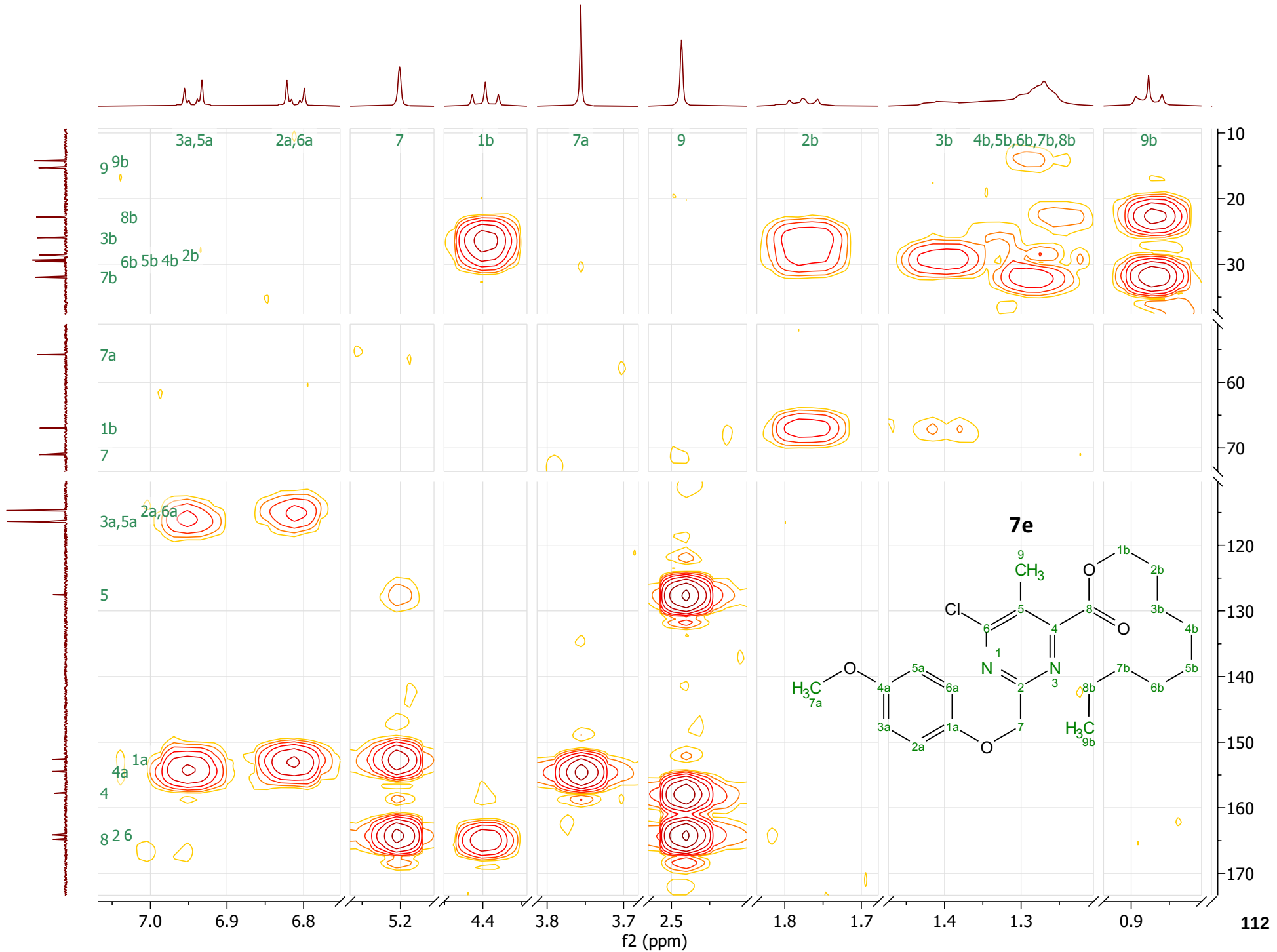


13C HSQC

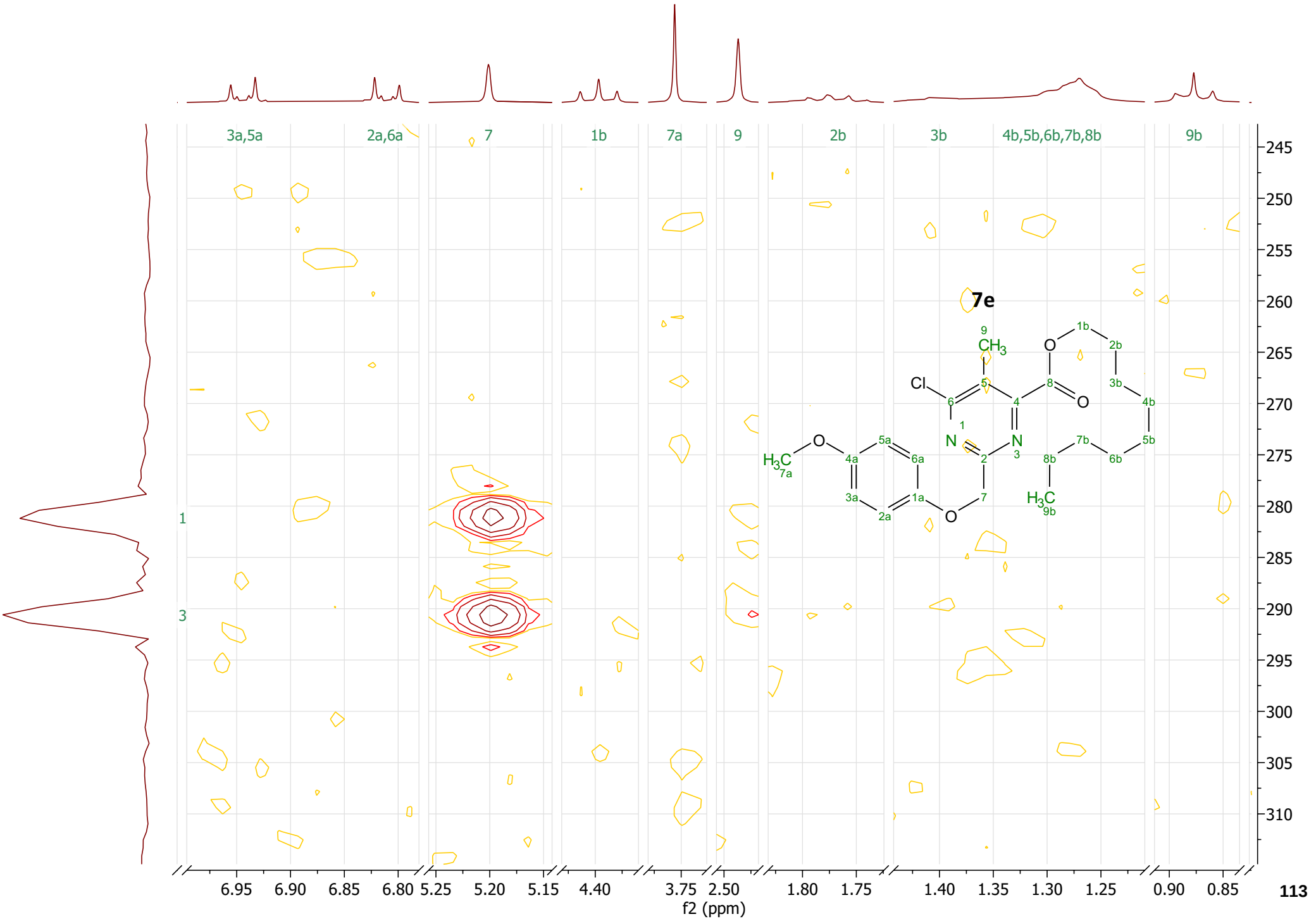

3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b,8b
9b
9
9b
8b
3b
6b
5b
4b
2b
7b
7a
1b
7
2a,6a
3a,5a
7e
f2 (ppm)
f1 (ppm)
7.1
7.0
6.9
6.8
6.7
5.2
4.4
3.7
2.5
1.8
1.7
1.4
1.3
1.2
0.9
0.8
15
20
25
30
35
55
60
65
70
115
120

111

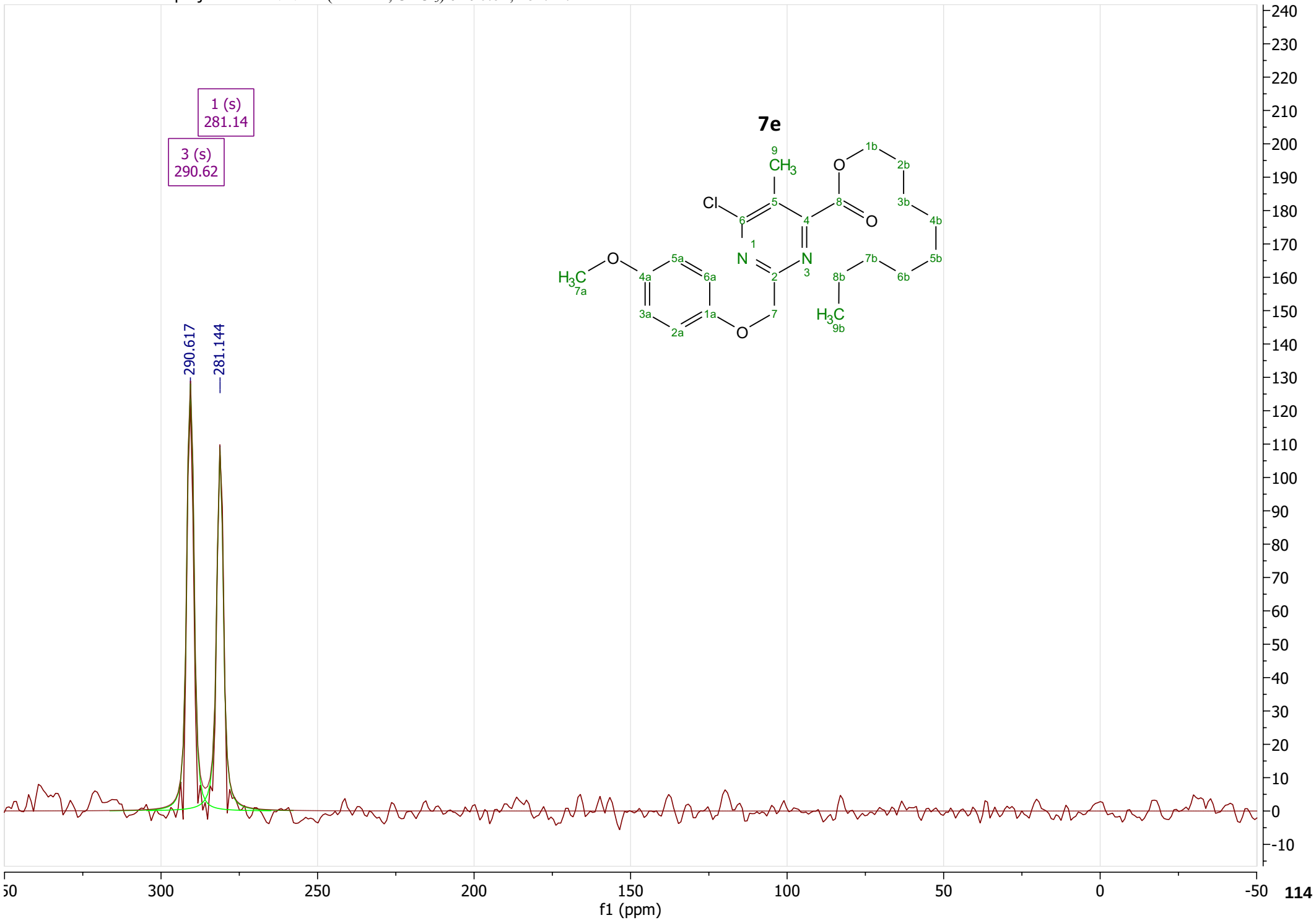
13C HMBC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b,8b
9b
9 9b
8b
3b
6b 5b 4b 2b
7b
7a
1b
7
3a,5a
2a,6a
5
4a
1a
4
8 2 6
7e
f2 (ppm)
f1 (ppm)
7.0
6.9
6.8
5.2
4.4
3.8
3.7
2.5
1.8
1.7
1.4
1.3
0.9
10
20
30
60
70
120
130
140
150
160
170

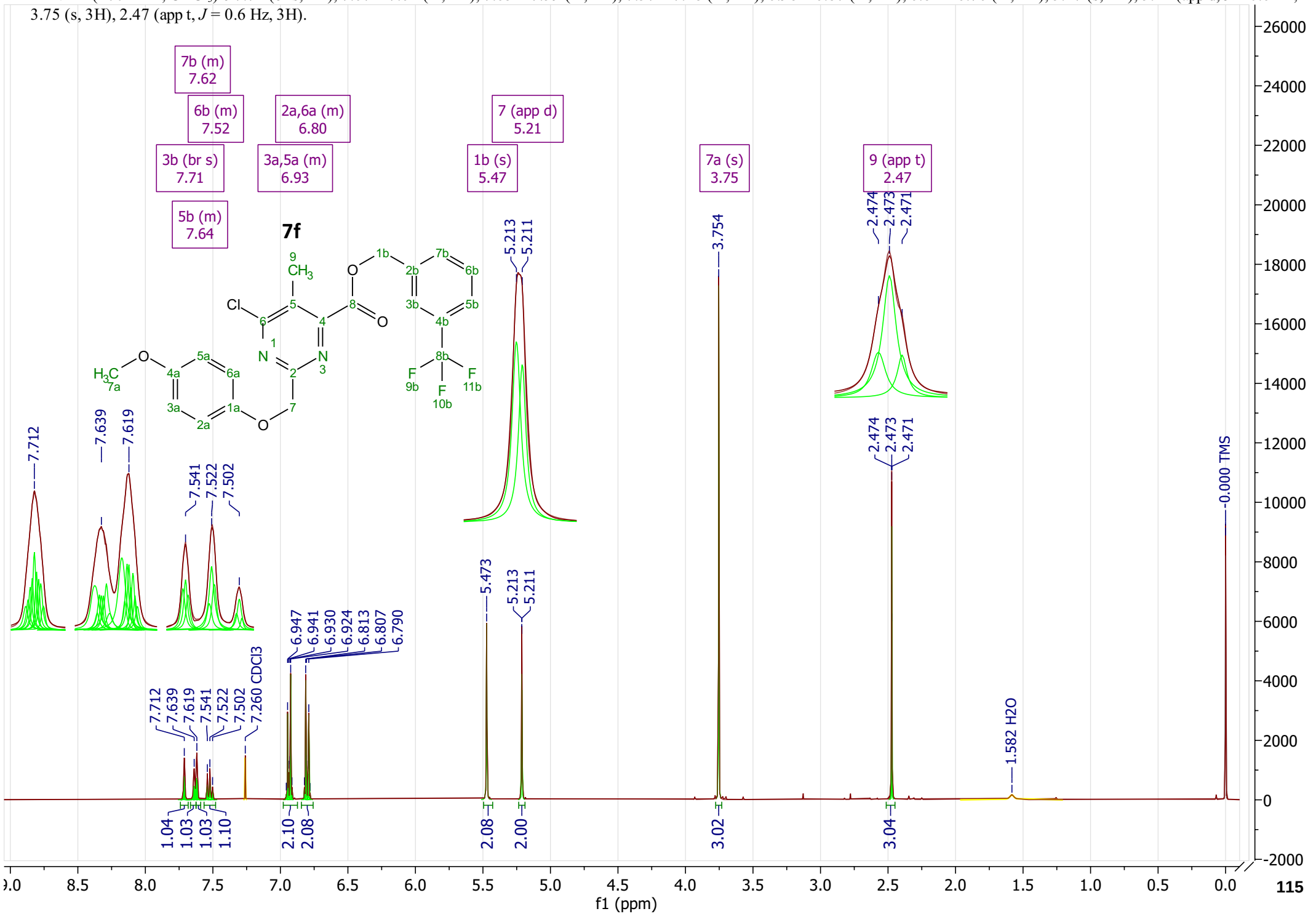
15N HMBC



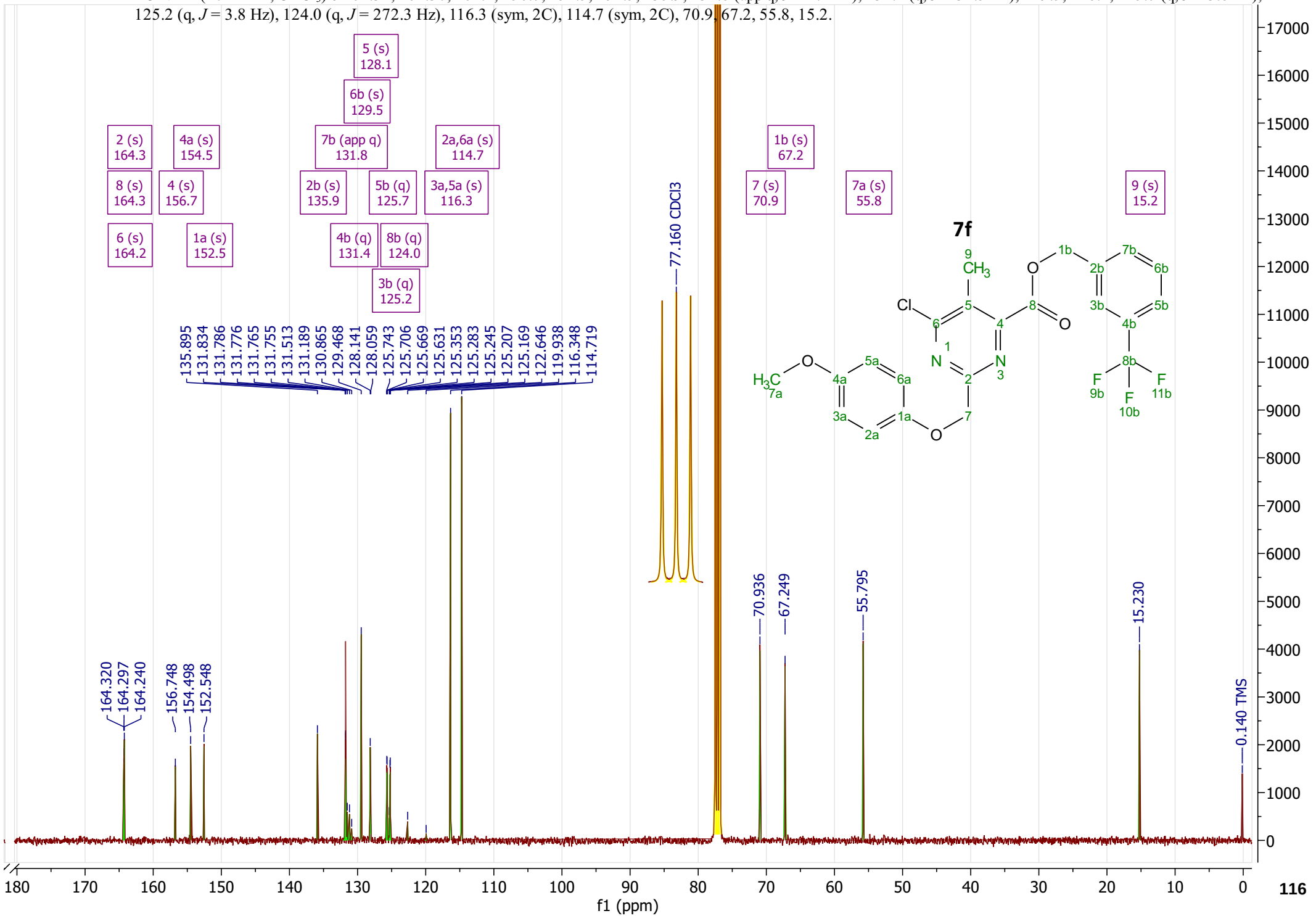
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ 290.62, 281.14.



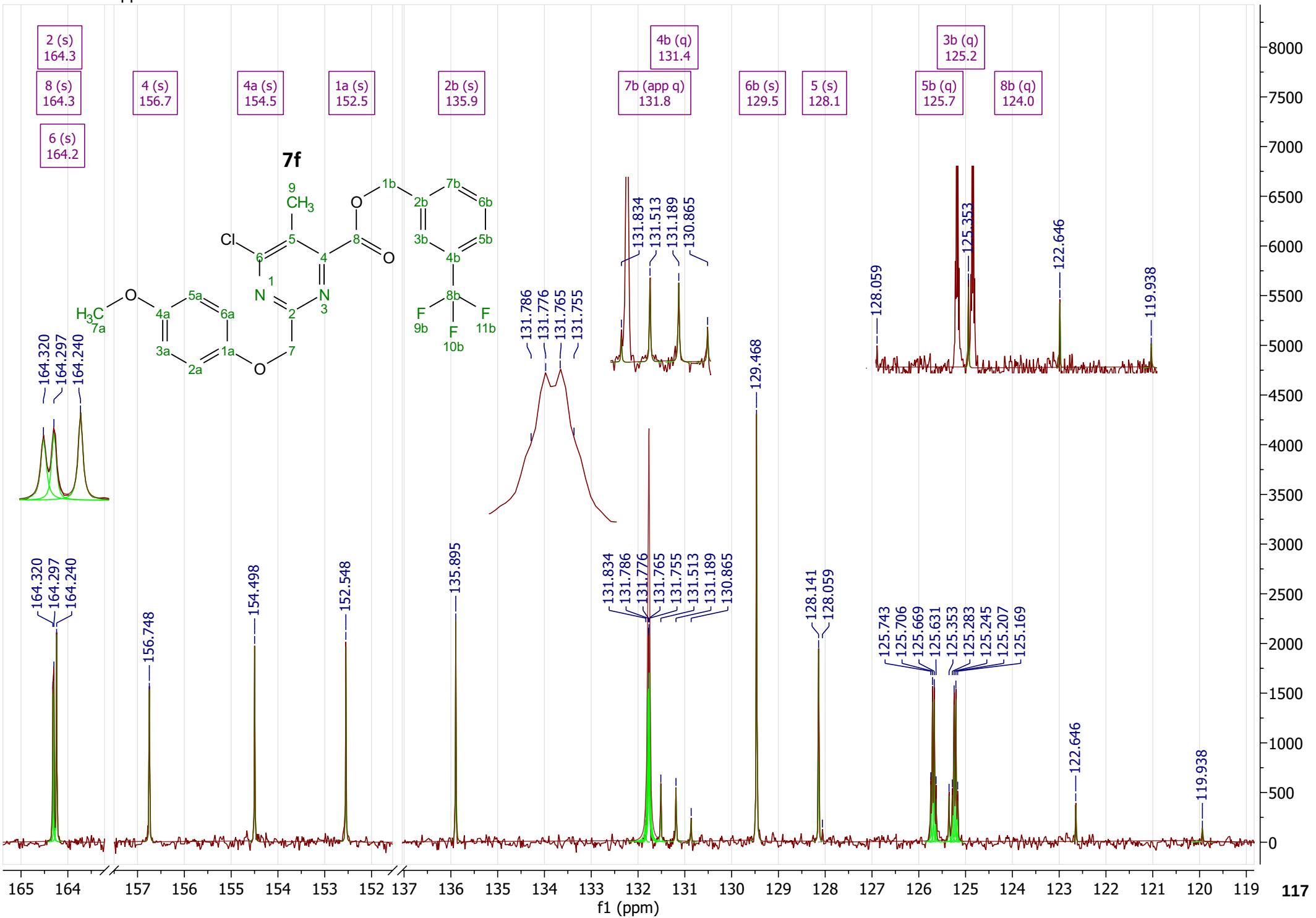
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (br s, 1H), 7.67 – 7.61 (m, 1H), 7.65 – 7.59 (m, 1H), 7.57 – 7.48 (m, 1H), 6.98 – 6.87 (m, 2H), 6.84 – 6.76 (m, 2H), 5.47 (s, 2H), 5.21 (app d, *J* = 0.6 Hz, 2H), 3.75 (s, 3H), 2.47 (app t, *J* = 0.6 Hz, 3H).



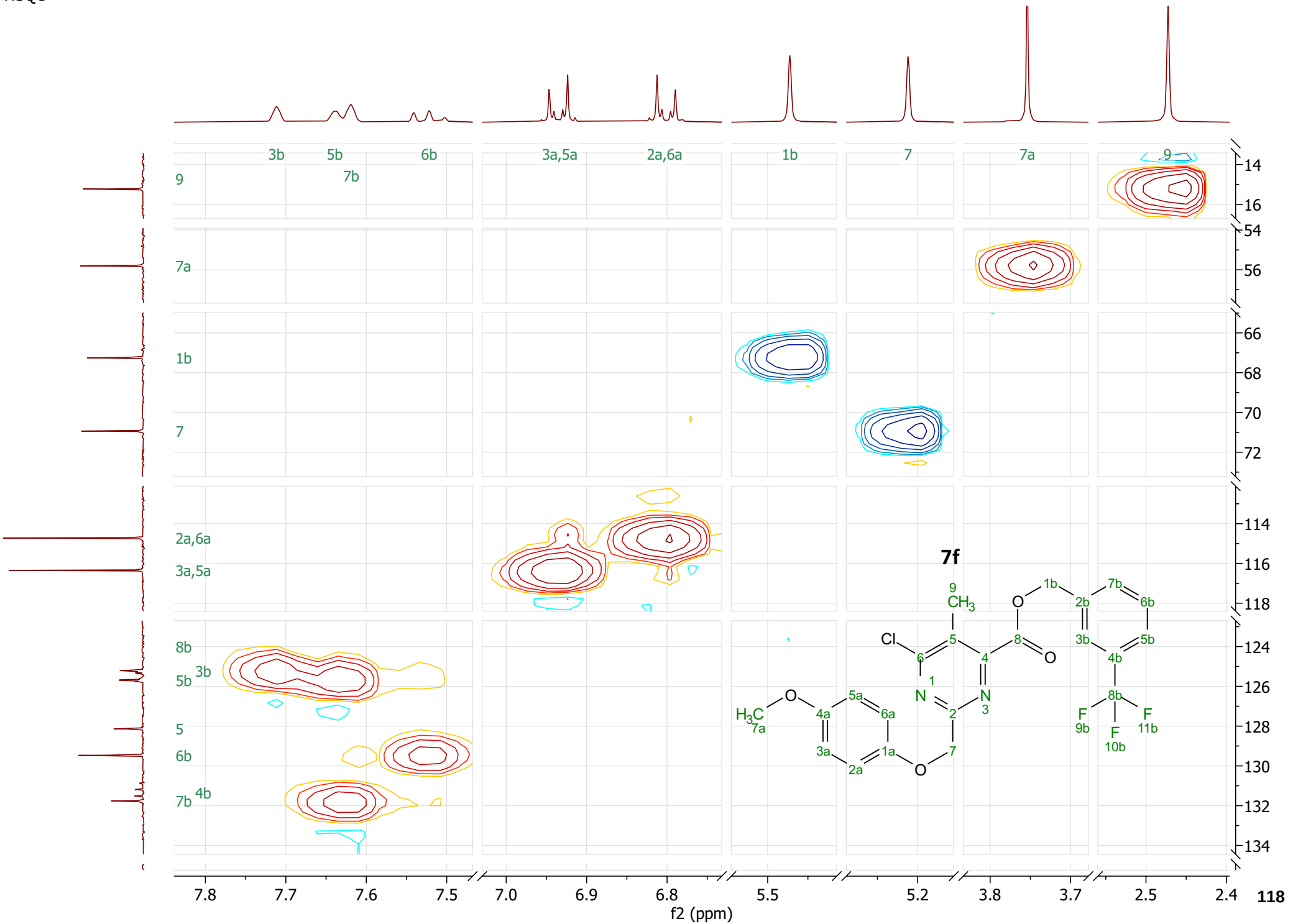
GHG103.10.fid — ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.32, 164.30, 164.2, 156.7, 154.5, 152.5, 135.9, 131.8 (app q, J = 1.1 Hz), 131.4 (q, J = 32.5 Hz), 129.5, 128.1, 125.7 (q, J = 3.8 Hz), 125.2 (q, J = 3.8 Hz), 124.0 (q, J = 272.3 Hz), 116.3 (sym, 2C), 114.7 (sym, 2C), 70.9, 67.2, 55.8, 15.2.



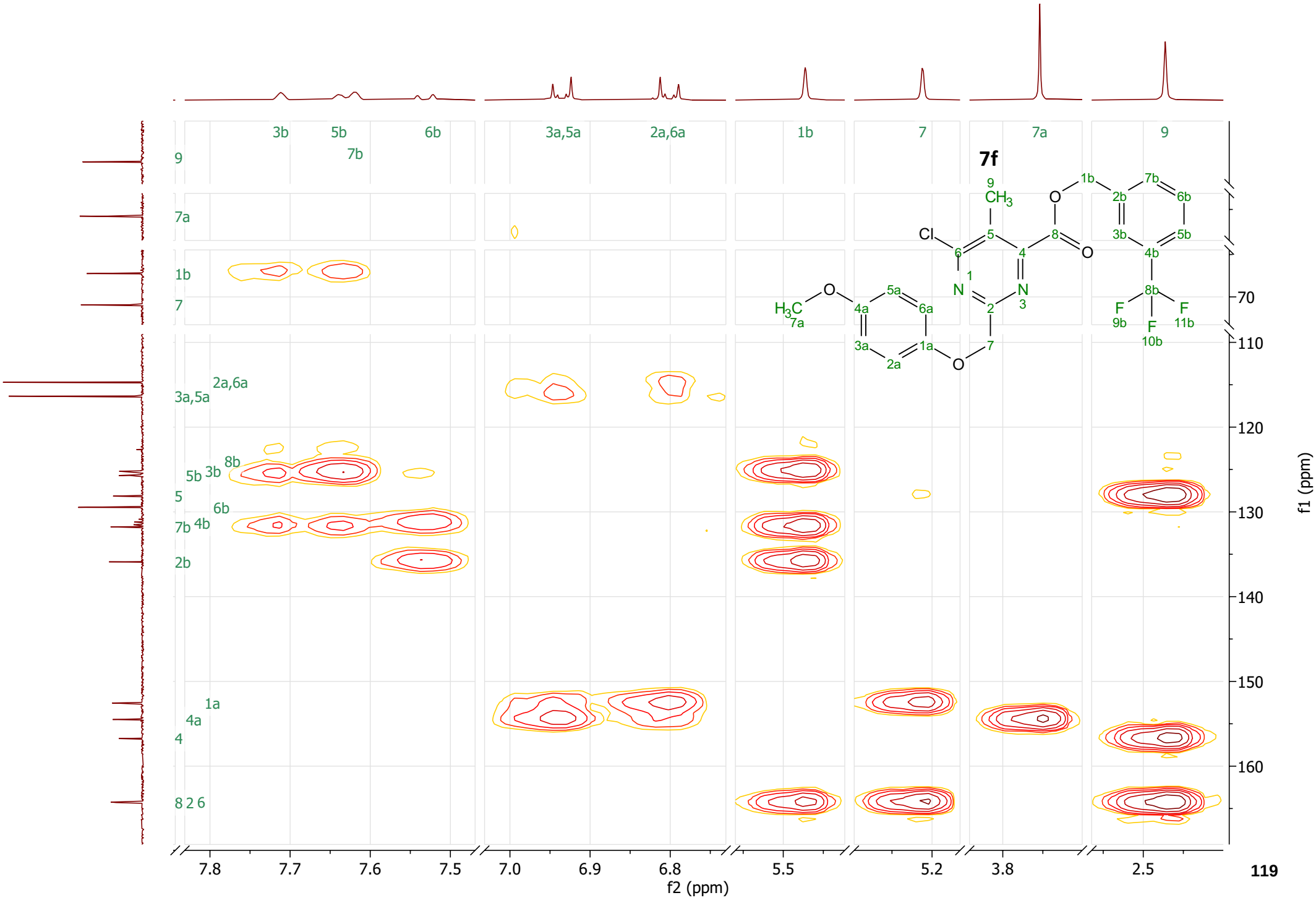
13C — 165–119 ppm



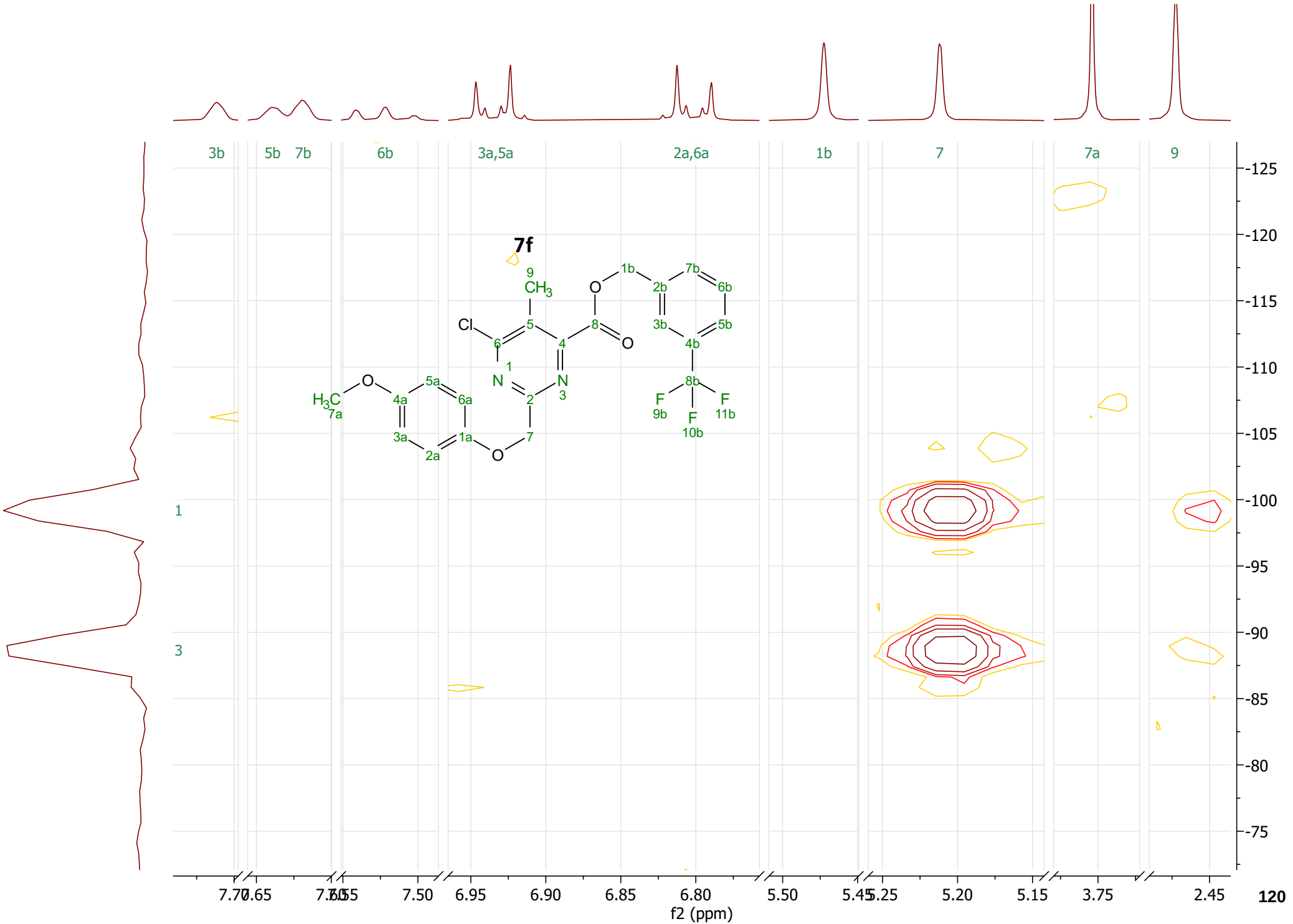
13C HSQC


3b
5b
7b
6b
3a,5a
2a,6a
1b
7
7a
9
9
7a
1b
2a,6a
3a,5a
8b
5b
3b
5
6b
7b
4b
14
16
54
56
66
68
70
72
114
116
118
124
126
128
130
132
134
f1 (ppm)
7.8
7.7
7.6
7.5
7.0
6.9
6.8
5.5
5.2
3.8
3.7
2.5
2.4
f2 (ppm)
7f
CH3
Cl
O
N
H3C
F
F
F

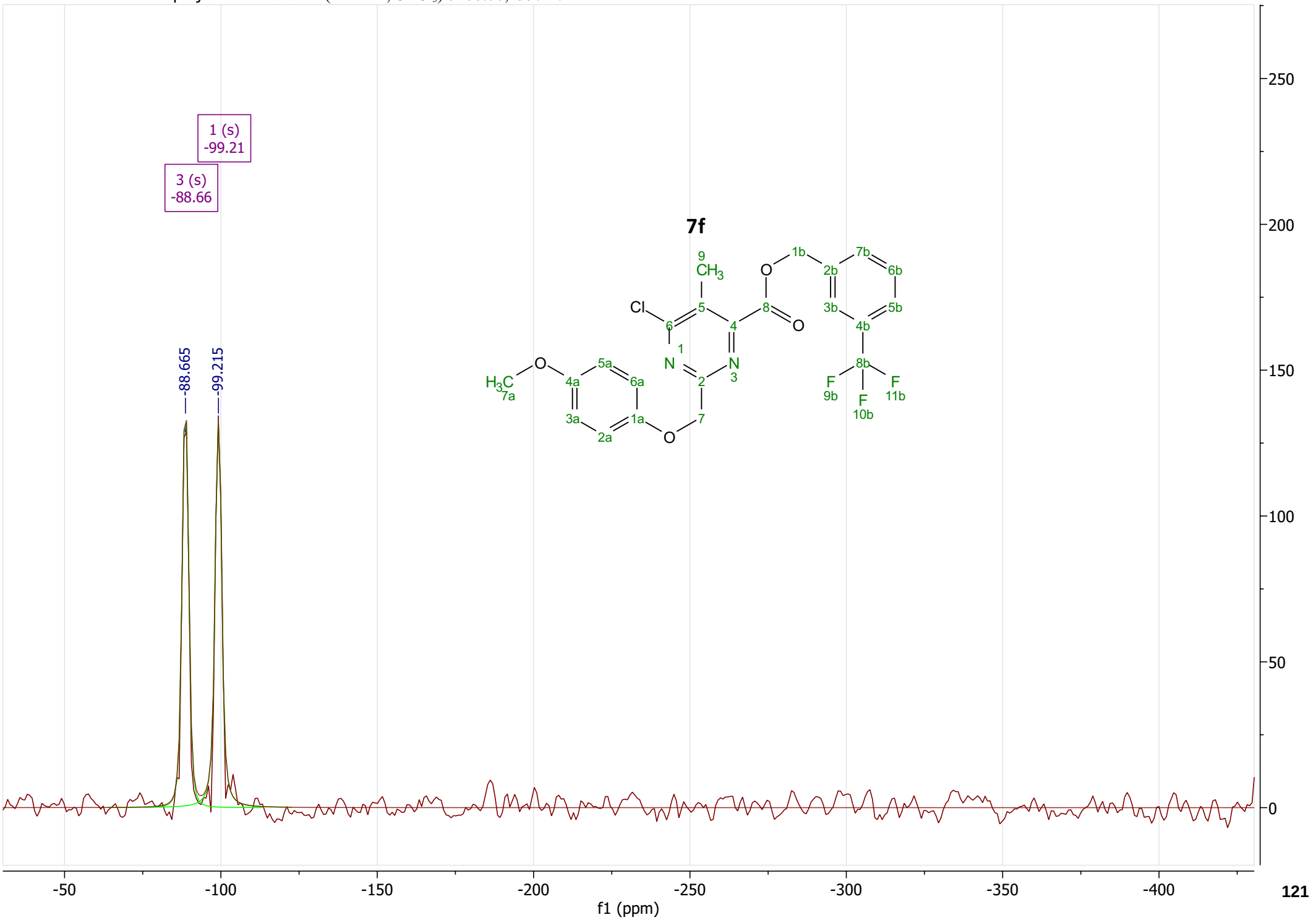
13C HMBC


7f
3b
5b
6b
7b
3a,5a
2a,6a
1b
7
7a
9
f2 (ppm)
f1 (ppm)
7.8
7.7
7.6
7.5
7.0
6.9
6.8
5.5
5.2
3.8
2.5
70
110
120
130
140
150
160

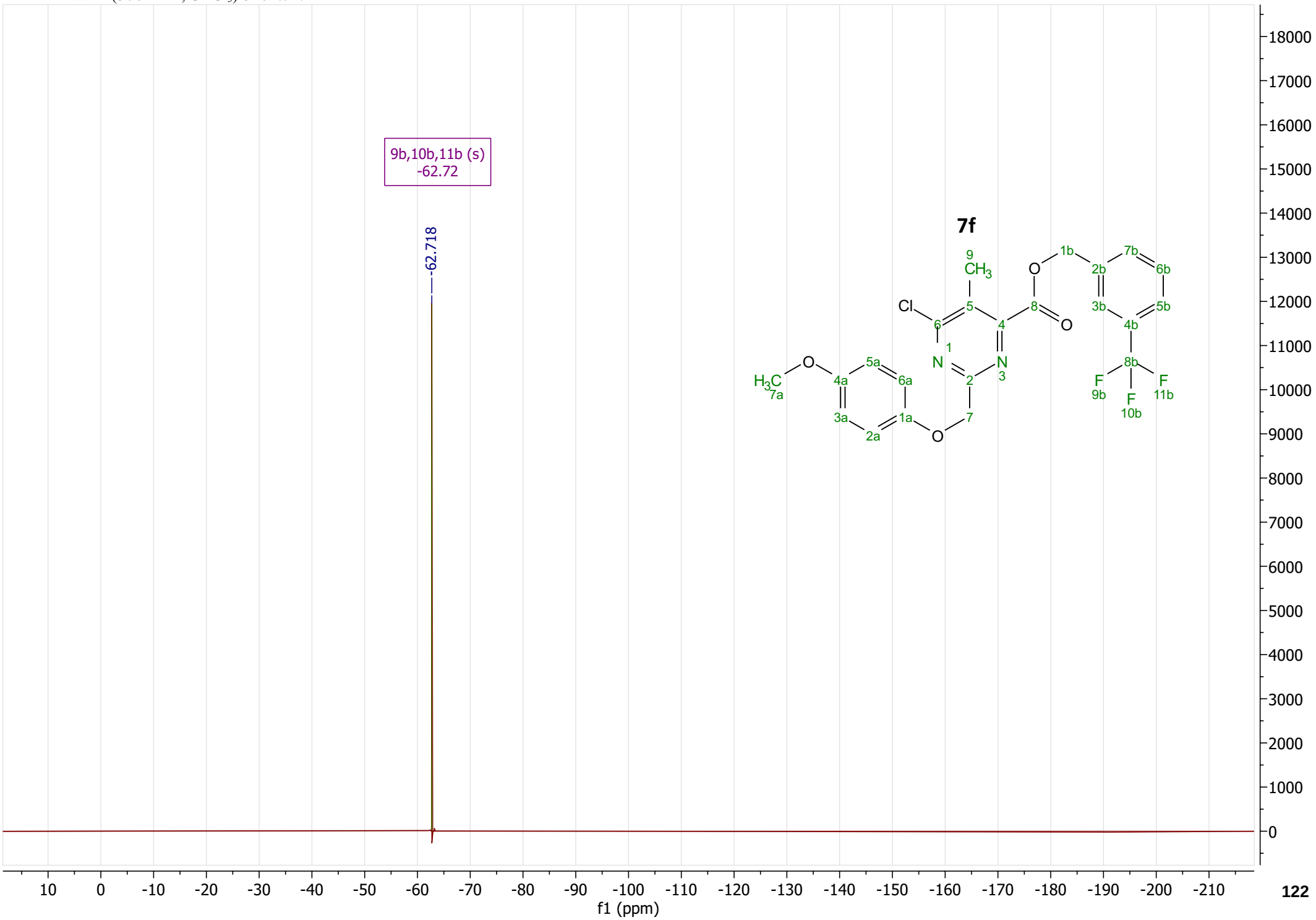
15N HMBC


3b
5b
7b
6b
3a,5a
2a,6a
1b
7
7a
9
7f
f2 (ppm)
f1 (ppm)
-125
-120
-115
-110
-105
-100
-95
-90
-85
-80
-75
1
3
7.70
7.65
7.55
7.50
6.95
6.90
6.85
6.80
5.50
5.45
5.25
5.20
5.15
3.75
2.45
120

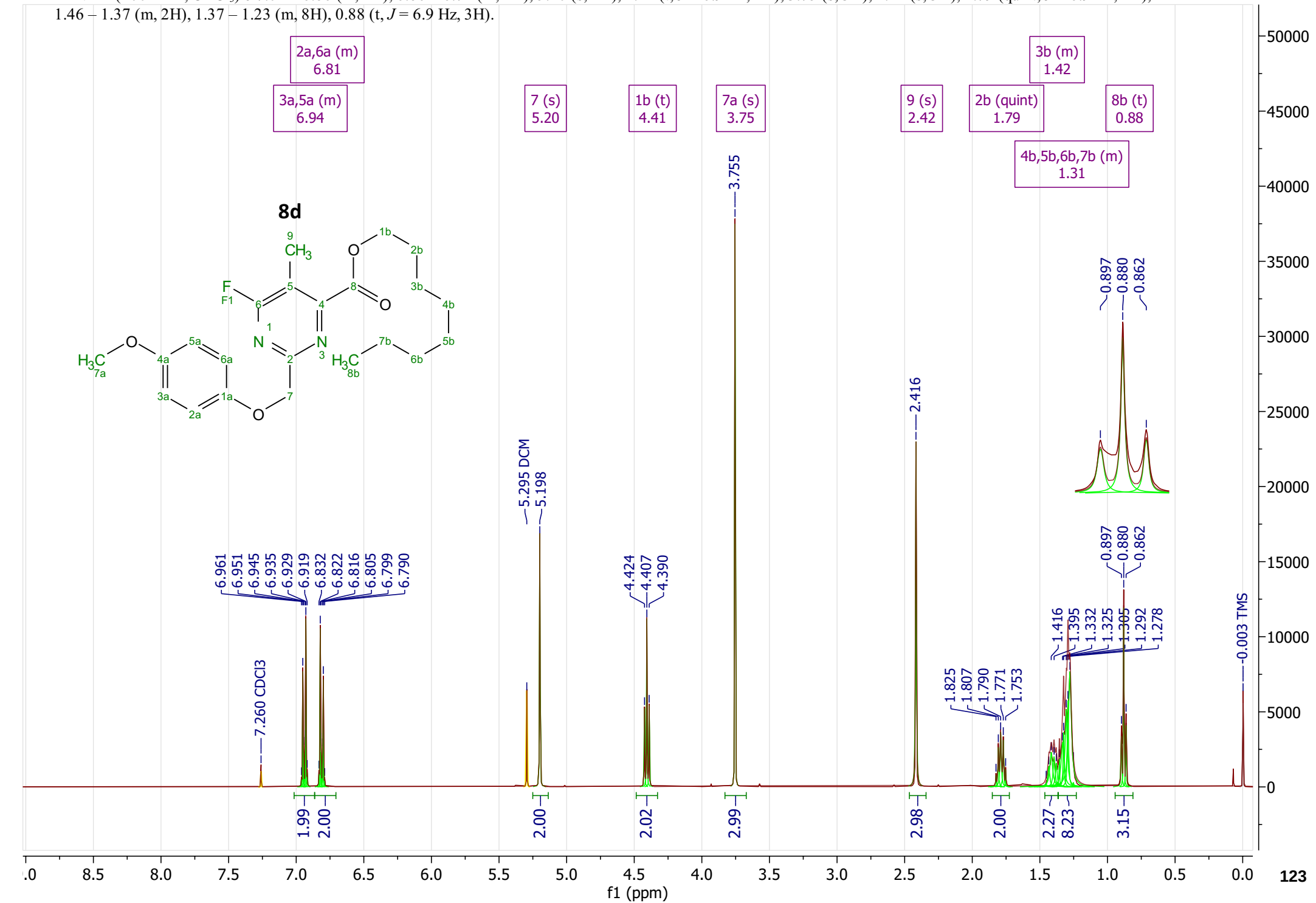
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -88.66, -99.21.



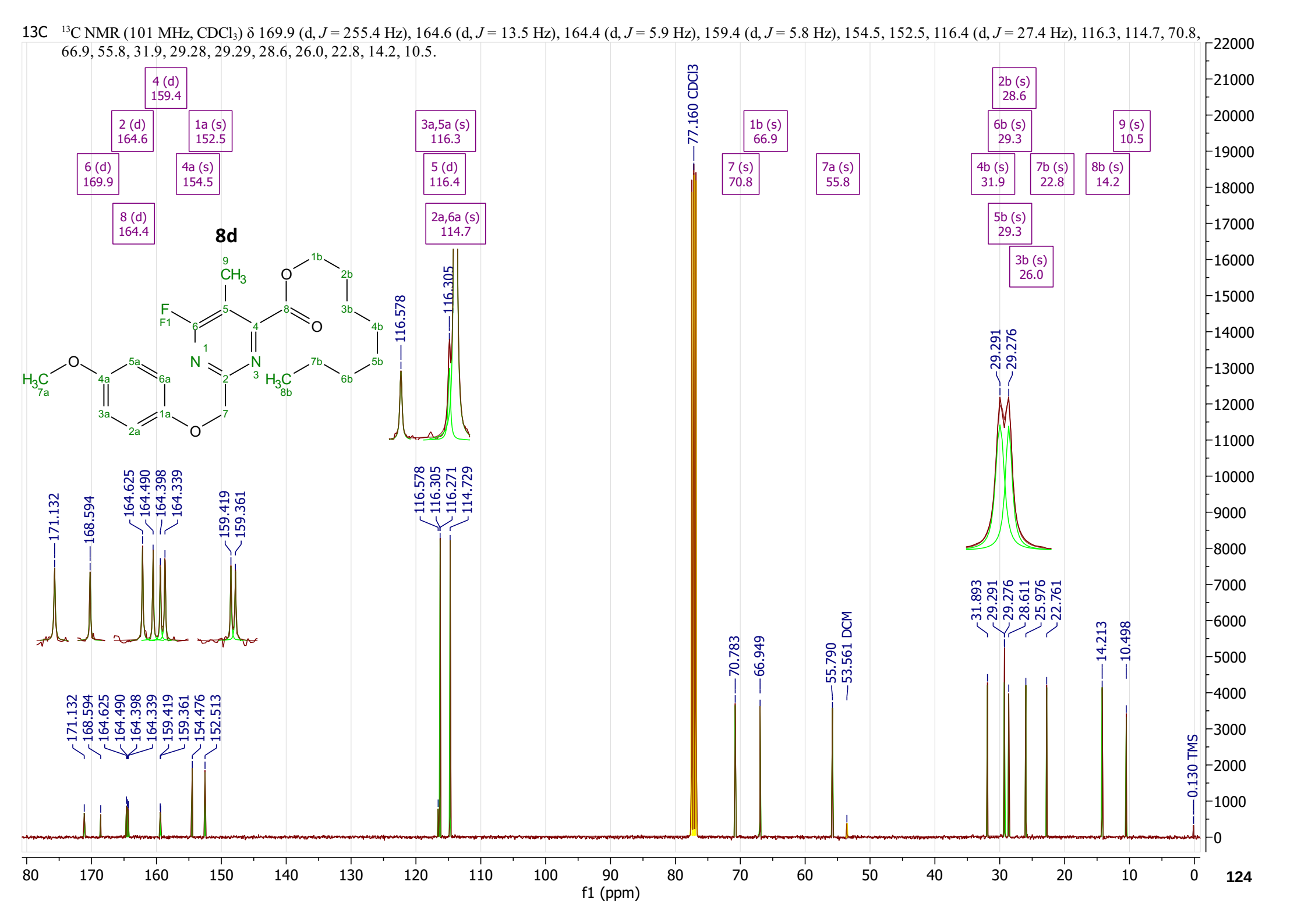
19F ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.72.



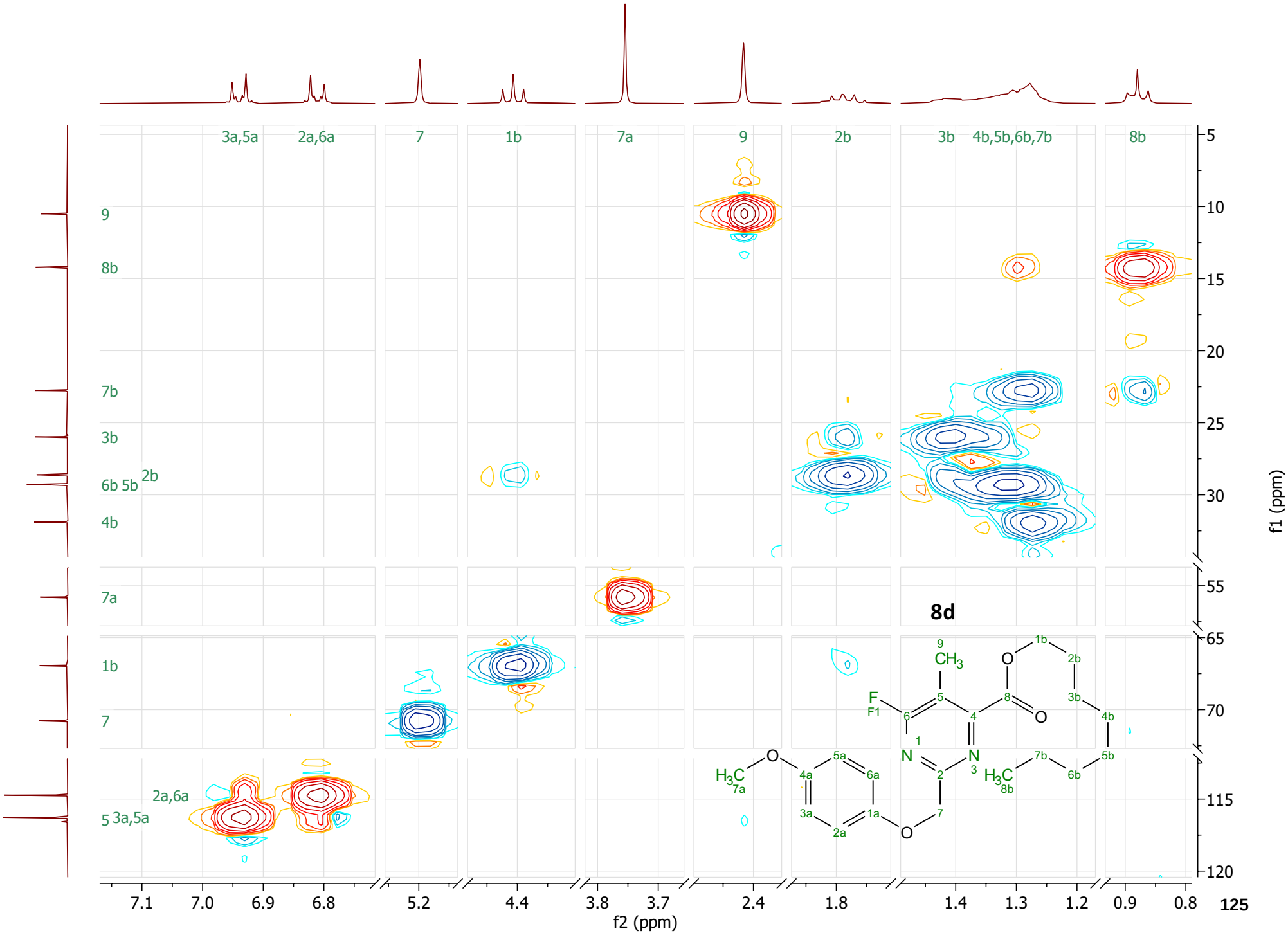
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.02 – 6.86 (m, 2H), 6.86 – 6.71 (m, 2H), 5.20 (s, 2H), 4.41 (t, *J* = 6.9 Hz, 2H), 3.75 (s, 3H), 2.42 (s, 3H), 1.79 (quint, *J* = 6.9 Hz, 2H), 1.46 – 1.37 (m, 2H), 1.37 – 1.23 (m, 8H), 0.88 (t, *J* = 6.9 Hz, 3H).



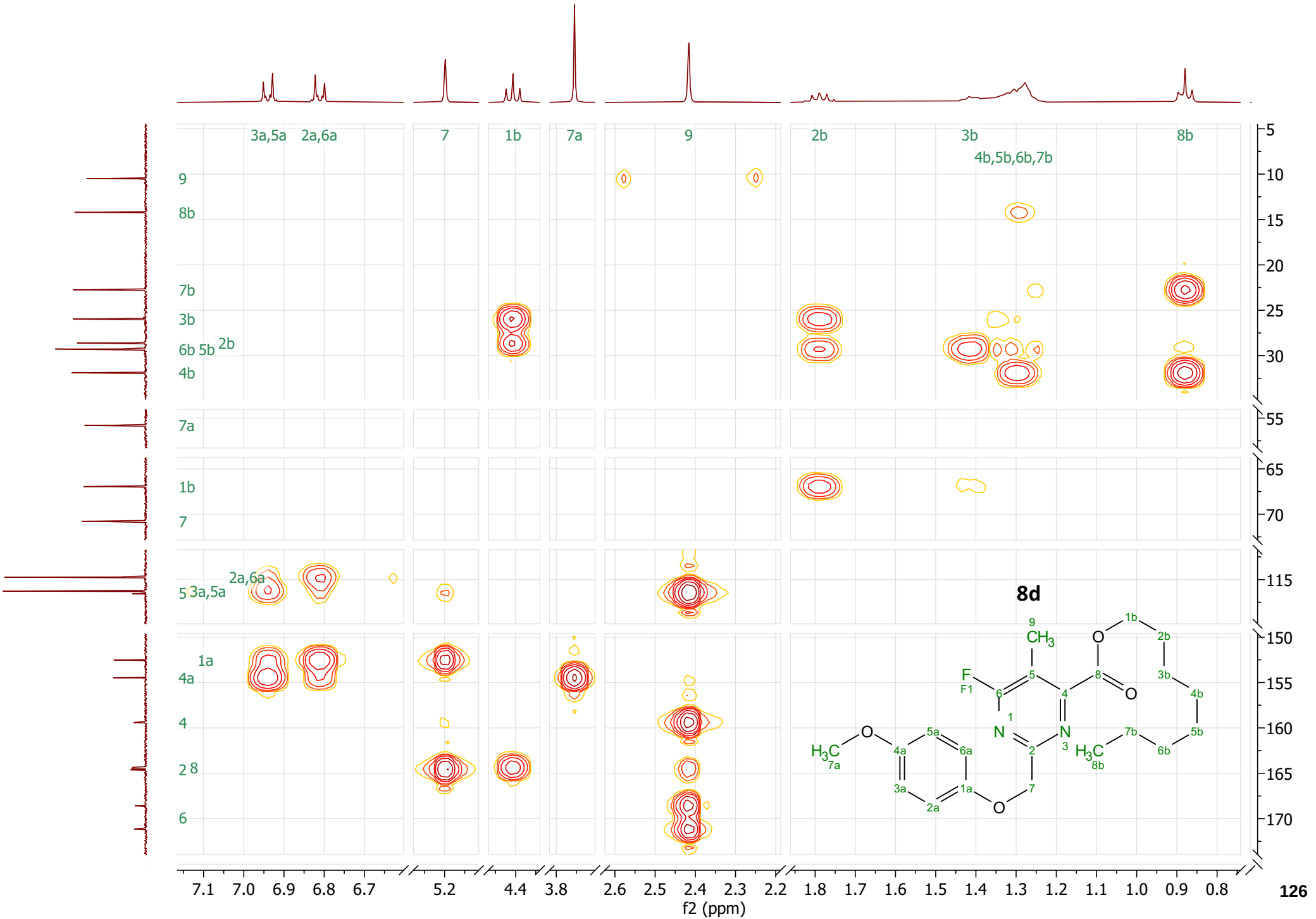
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.9 (d, J = 255.4 Hz), 164.6 (d, J = 13.5 Hz), 164.4 (d, J = 5.9 Hz), 159.4 (d, J = 5.8 Hz), 154.5, 152.5, 116.4 (d, J = 27.4 Hz), 116.3, 114.7, 70.8, 66.9, 55.8, 31.9, 29.28, 29.29, 28.6, 26.0, 22.8, 14.2, 10.5.



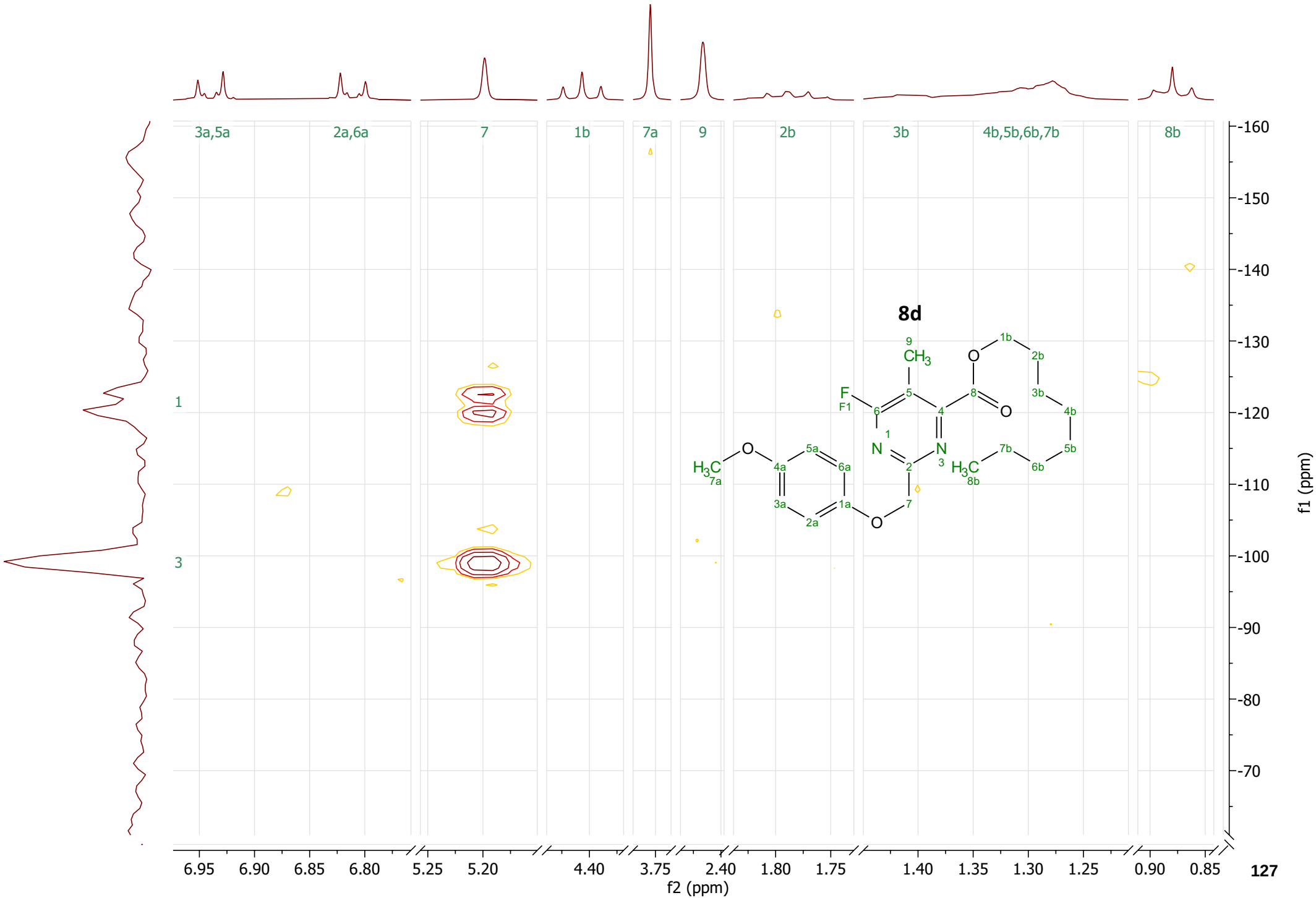
13C HSQC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
9
8b
7b
3b
6b 5b 2b
4b
7a
1b
7
5 3a,5a
2a,6a
8d
f2 (ppm)
f1 (ppm)
7.1
7.0
6.9
6.8
5.2
4.4
3.8
3.7
2.4
1.8
1.4
1.3
1.2
0.9
0.8
125
5
10
15
20
25
30
55
65
70
115
120

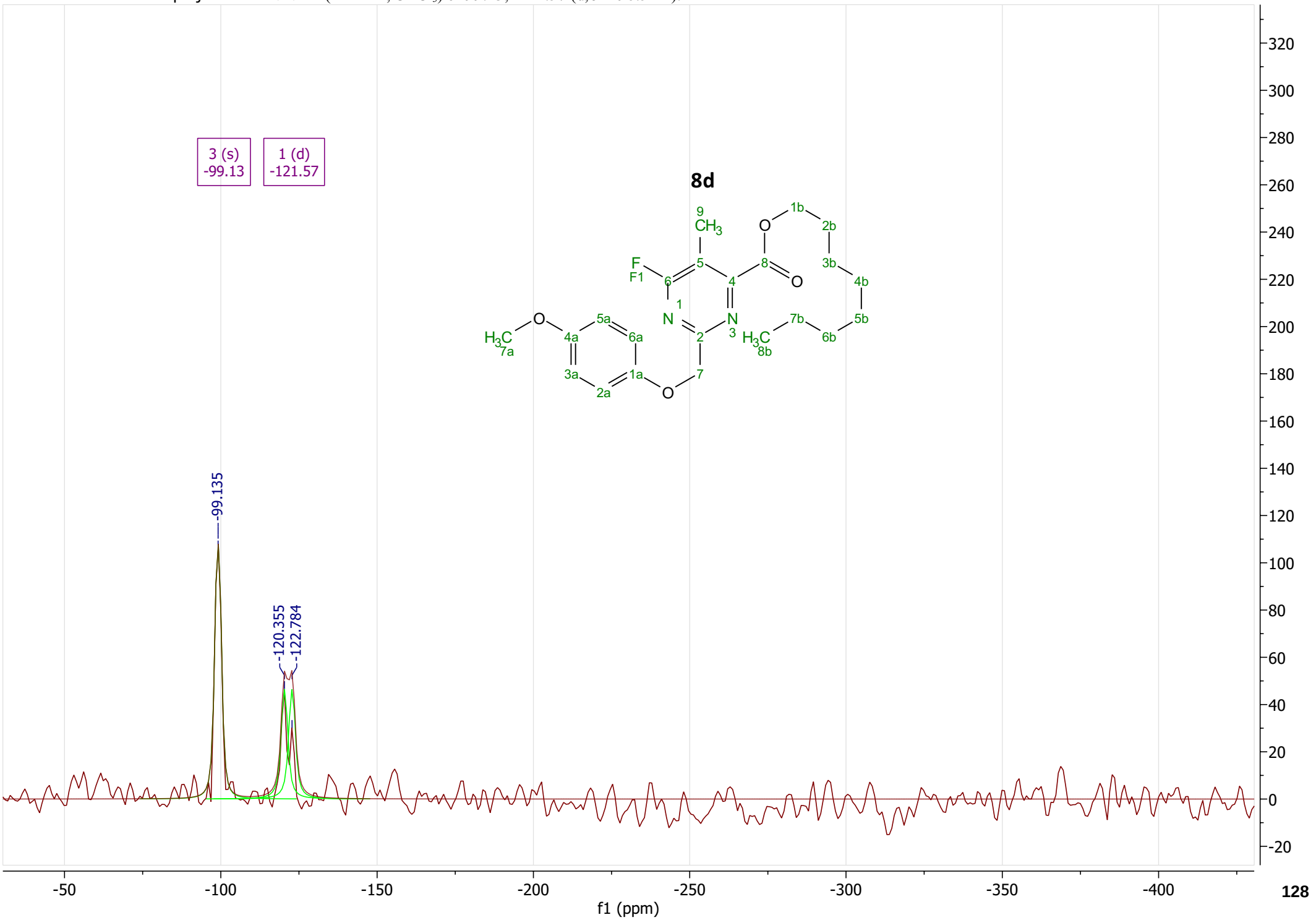
13C HMBC


3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
9
8b
7b
3b
6b 5b 2b
4b
7a
1b
7
5
3a,5a
2a,6a
1a
4a
4
2 8
6
8d
f2 (ppm)
f1 (ppm)
7.1 7.0 6.9 6.8 6.7 5.2 4.4 3.8 2.6 2.5 2.4 2.3 2.2 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8
5 10 15 20 25 30 55 65 70 115 150 155 160 165 170

15N HMBC

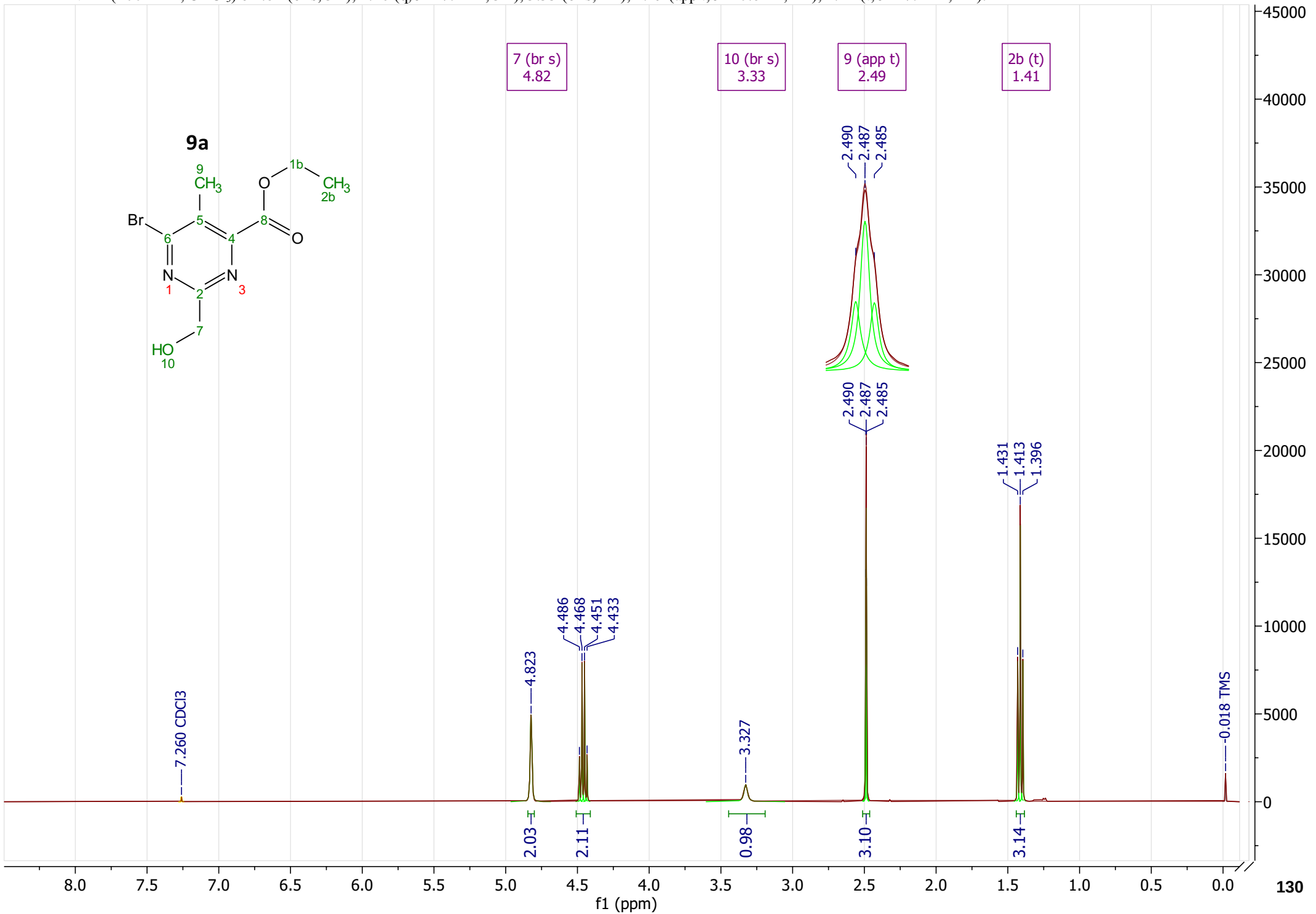

3a,5a
2a,6a
7
1b
7a
9
2b
3b
4b,5b,6b,7b
8b
1
3
8d
f2 (ppm)
f1 (ppm)
6.95
6.90
6.85
6.80
5.25
5.20
4.40
3.75
2.40
1.80
1.75
1.40
1.35
1.30
1.25
0.90
0.85
-160
-150
-140
-130
-120
-110
-100
-90
-80
-70

15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -99.13, -121.57 (d, J = 98.5 Hz).

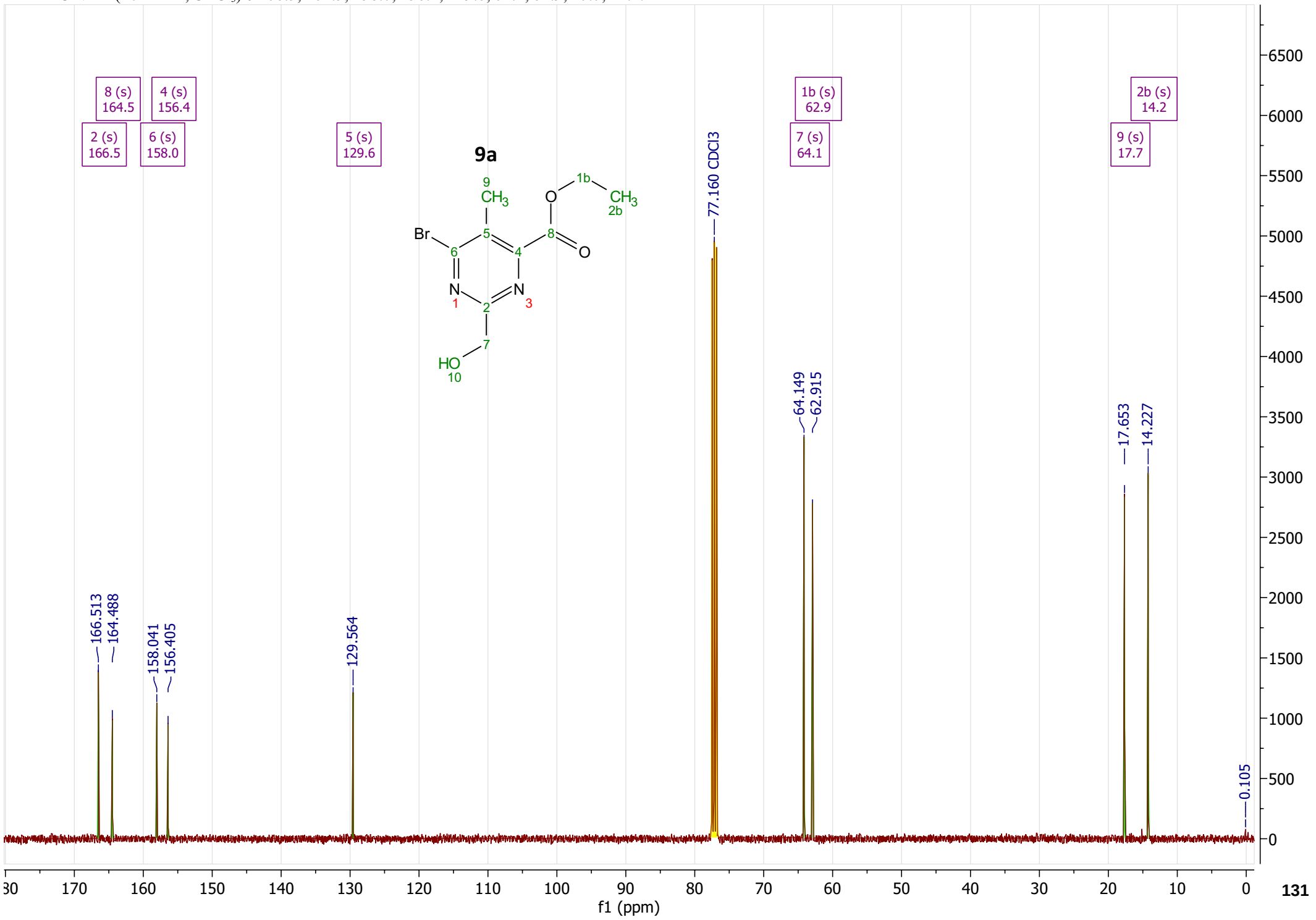


19F ^{19}F NMR (376 MHz, $CDCl_3$) δ -60.82.

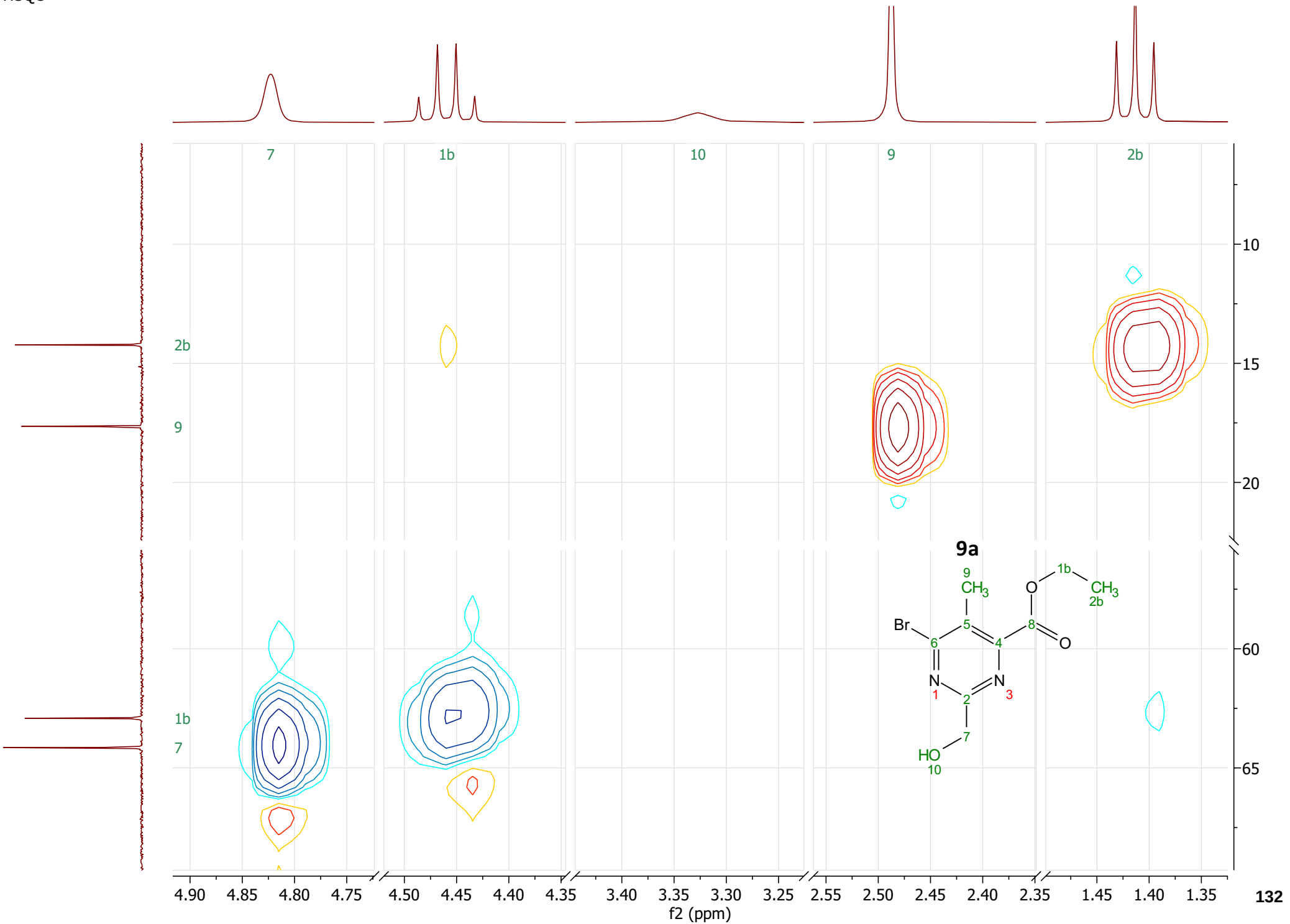
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (br s, 3H), 4.46 (q, J = 7.2 Hz, 3H), 3.33 (br s, 1H), 2.49 (app t, J = 0.8 Hz, 4H), 1.41 (t, J = 7.2 Hz, 4H).



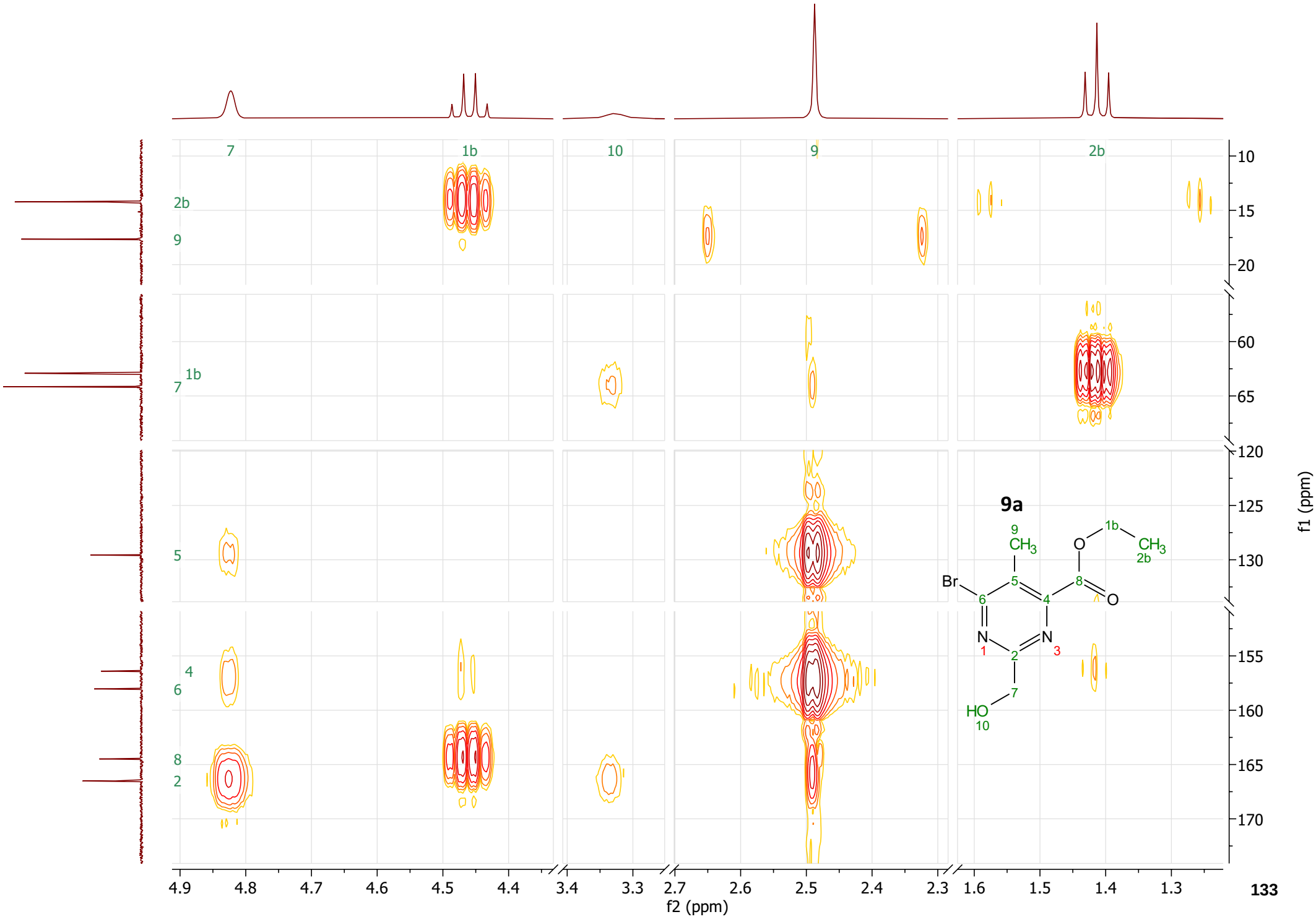
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.5, 158.0, 156.4, 129.6, 64.1, 62.9, 17.7, 14.2.



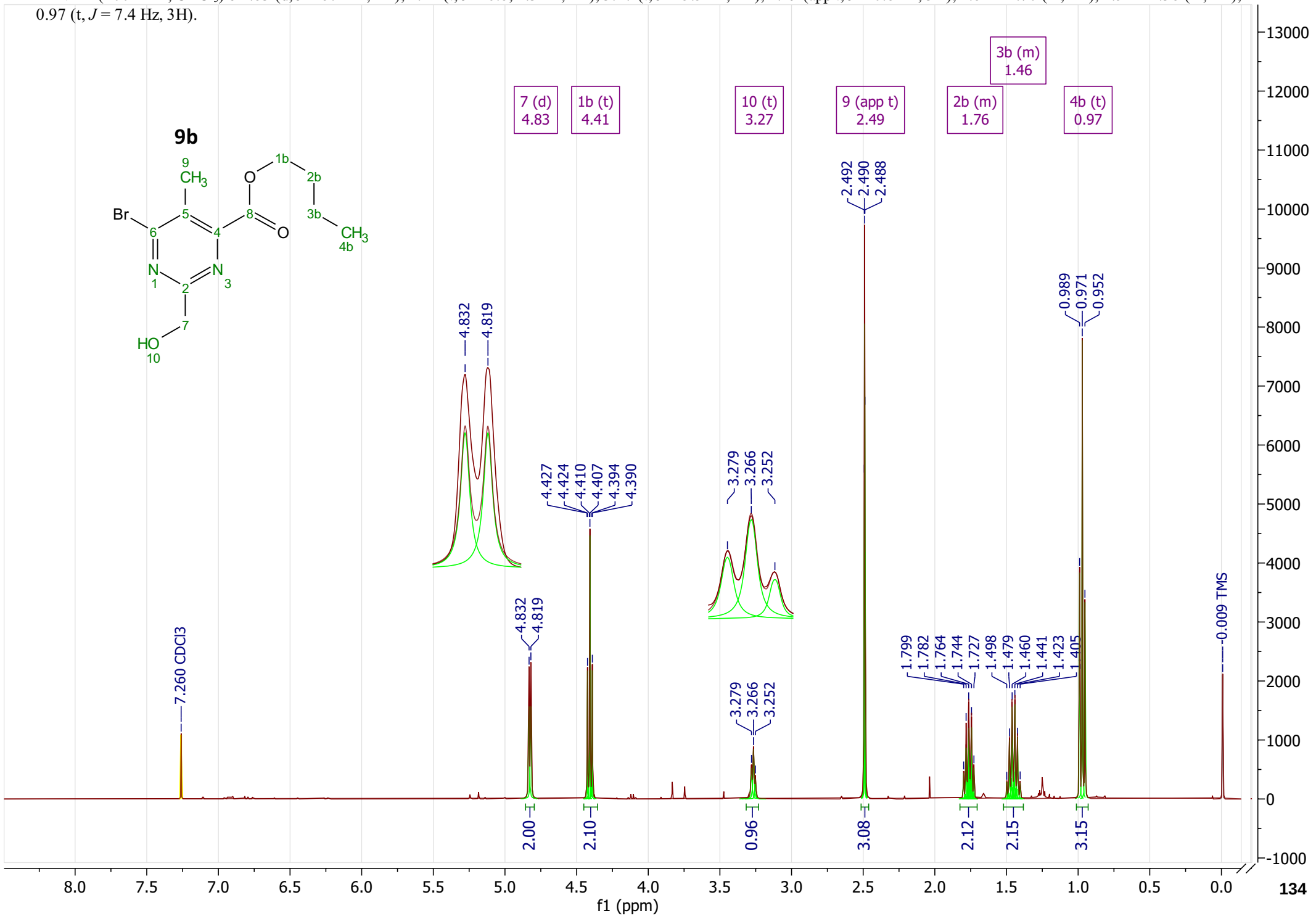
13C HSQC


7
1b
10
9
2b
2b
9
1b
7
10
15
20
60
65
f1 (ppm)
4.90
4.85
4.80
4.75
4.50
4.45
4.40
4.35
3.40
3.35
3.30
3.25
2.55
2.50
2.45
2.40
2.35
1.45
1.40
1.35
f2 (ppm)
9a
Br
CH3
O
CH3
O
N
N
HO

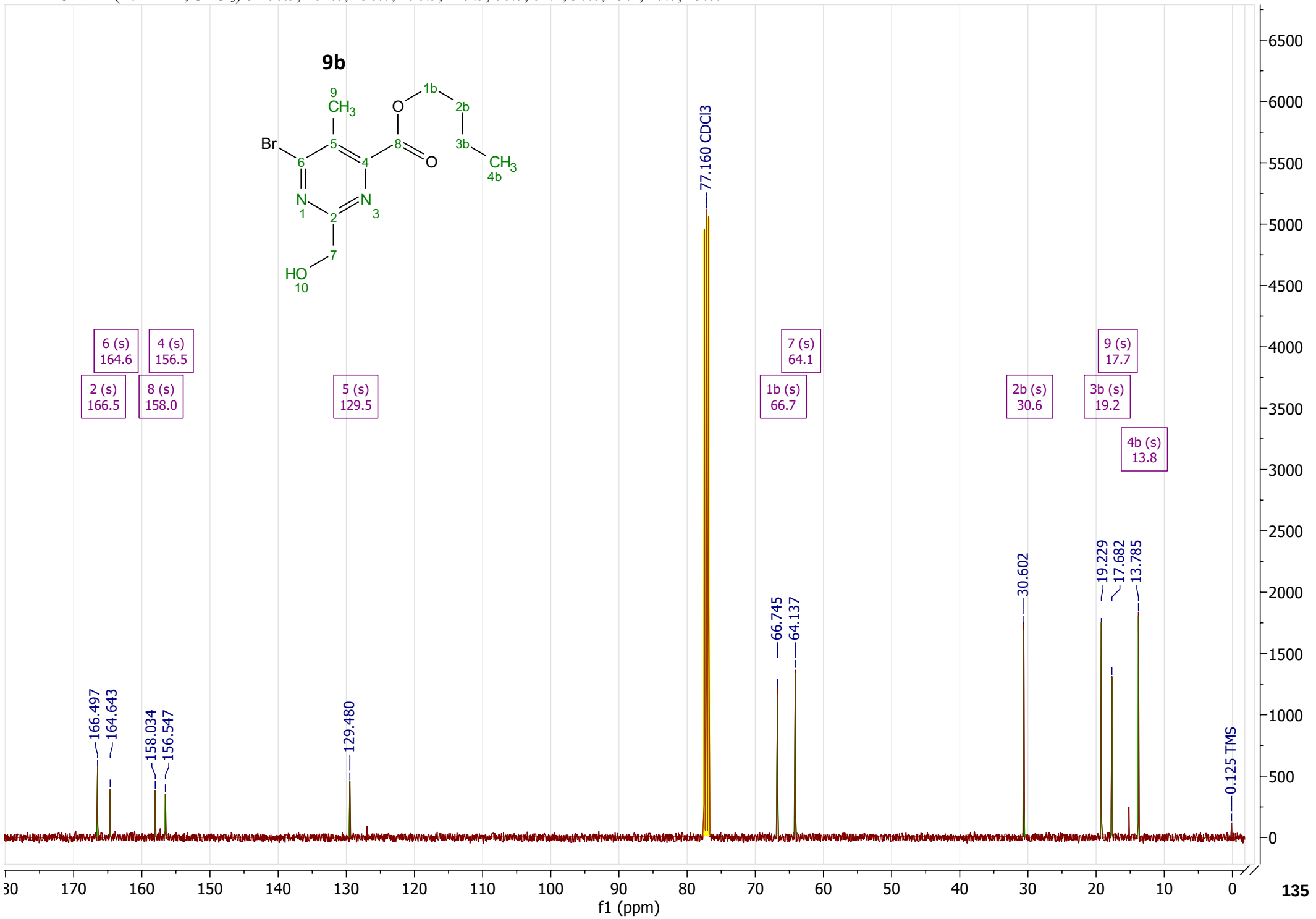
13C HMBC


7
1b
10
9
2b
2b
9
7
1b
5
6
4
8
2
9a
9
CH3
1b
O
CH3
2b
Br
5
8
6
4
O
N
N
1
2
3
7
HO
10
10
15
20
60
65
120
125
130
155
160
165
170
f1 (ppm)
4.9
4.8
4.7
4.6
4.5
4.4
3.4
3.3
2.7
2.6
2.5
2.4
2.3
1.6
1.5
1.4
1.3
f2 (ppm)

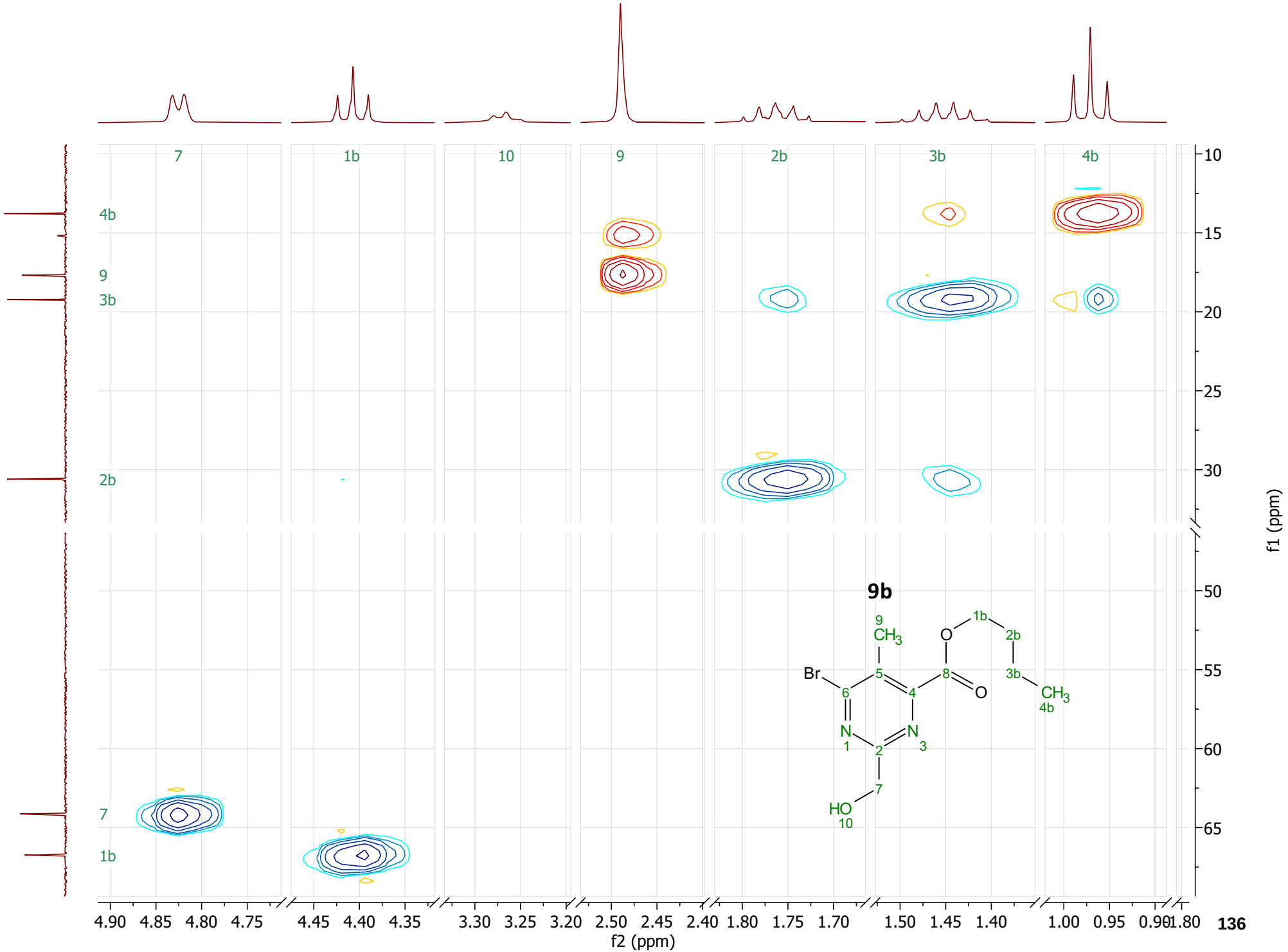
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (d, J = 5.1 Hz, 2H), 4.41 (t, J = 6.8, 1.3 Hz, 2H), 3.27 (t, J = 5.5 Hz, 1H), 2.49 (app t, J = 0.8 Hz, 3H), 1.82 – 1.70 (m, 2H), 1.52 – 1.38 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H).



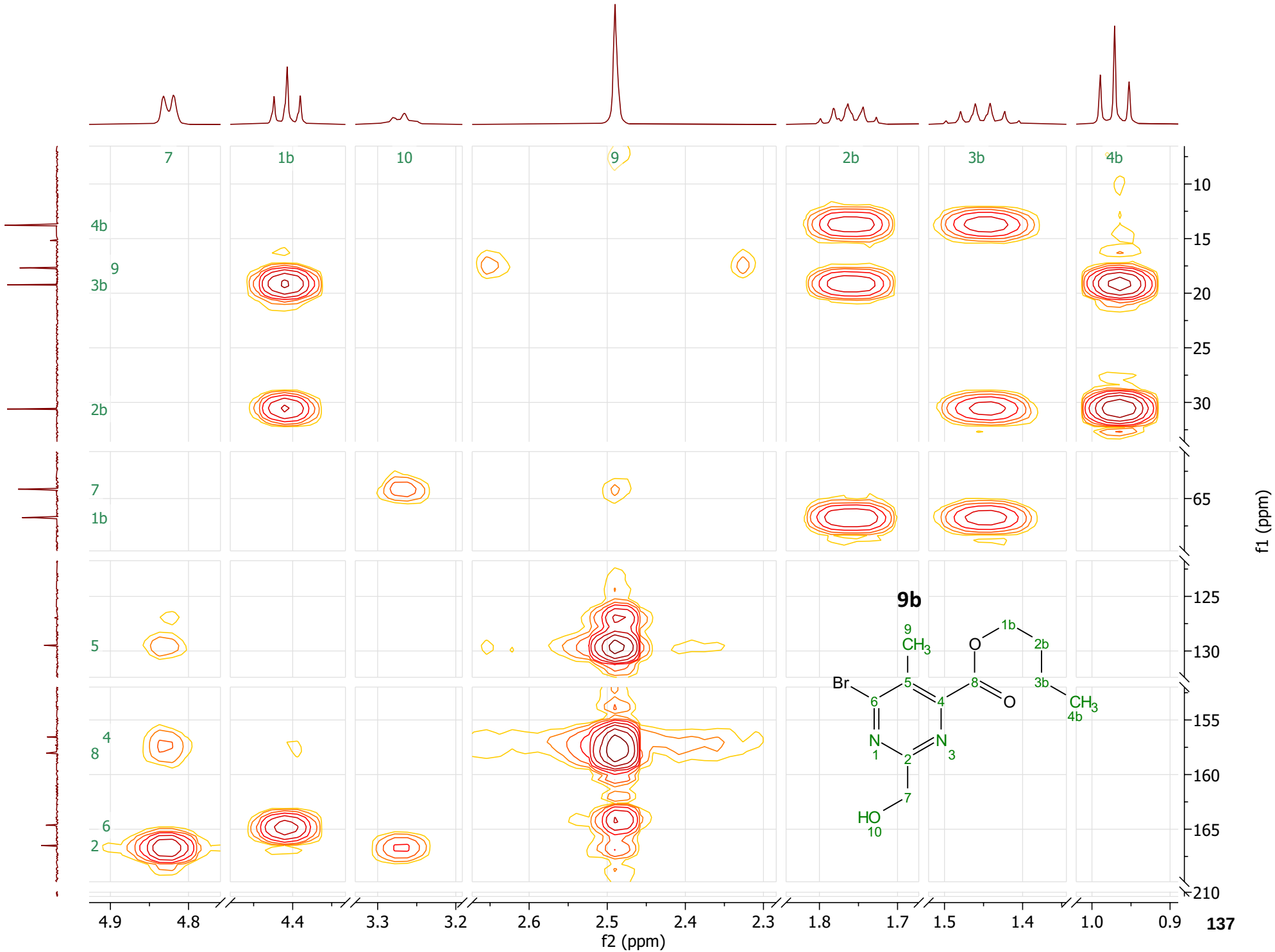
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.6, 158.0, 156.5, 129.5, 66.7, 64.1, 30.6, 19.2, 17.7, 13.8.



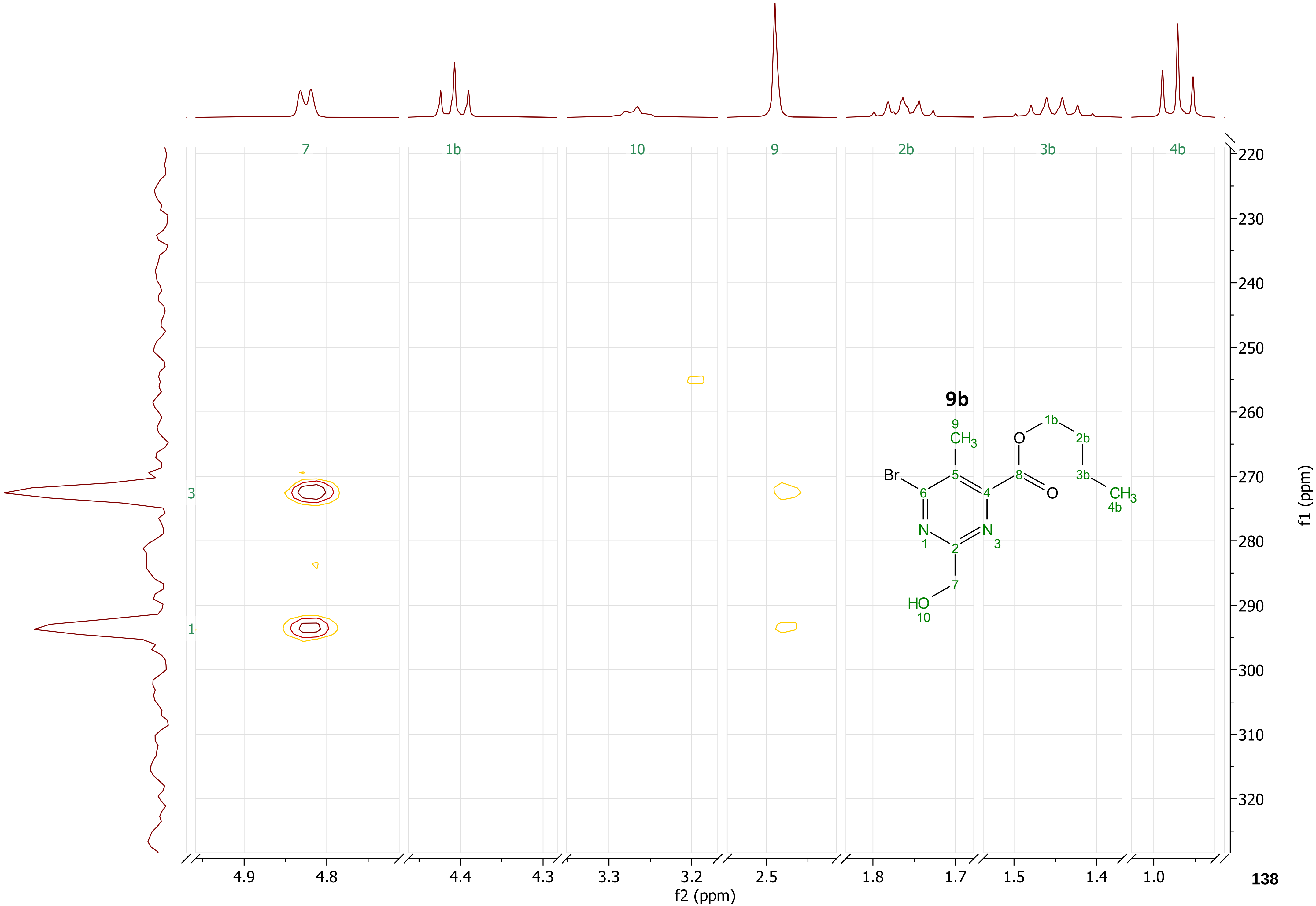
13C HSQC


7
1b
10
9
2b
3b
4b
4b
9
3b
2b
7
1b
9b
9
CH3
O
1b
2b
Br
5
8
3b
CH3
6
4
O
4b
N
1
N
3
2
7
HO
10
10
15
20
25
30
50
55
60
65
4.90 4.85 4.80 4.75
4.45 4.40 4.35
3.30 3.25 3.20
2.50 2.45 2.40
1.80 1.75 1.70
1.50 1.45 1.40
1.00 0.95 0.90
1.80
f2 (ppm)
f1 (ppm)

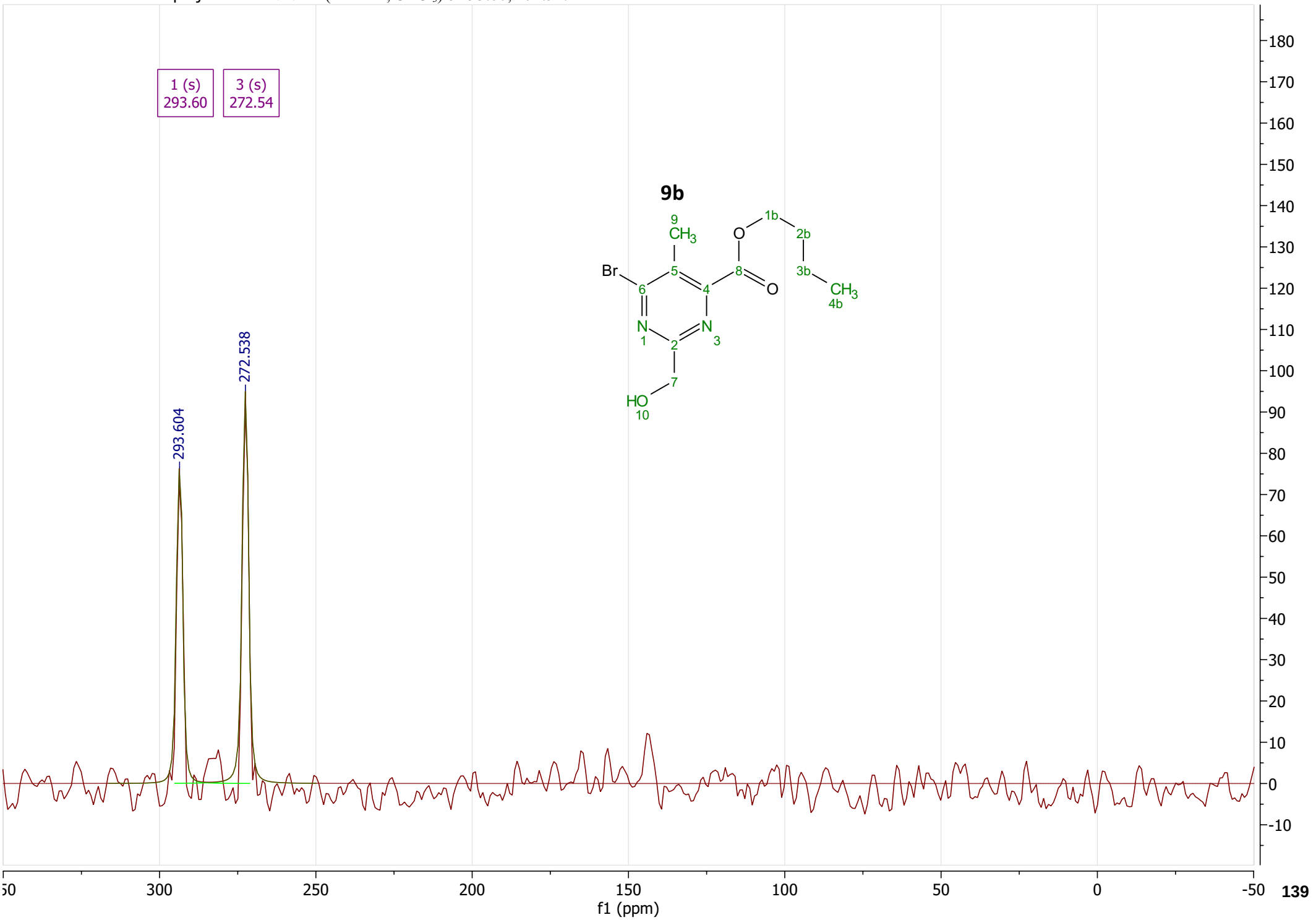
13C HMBC



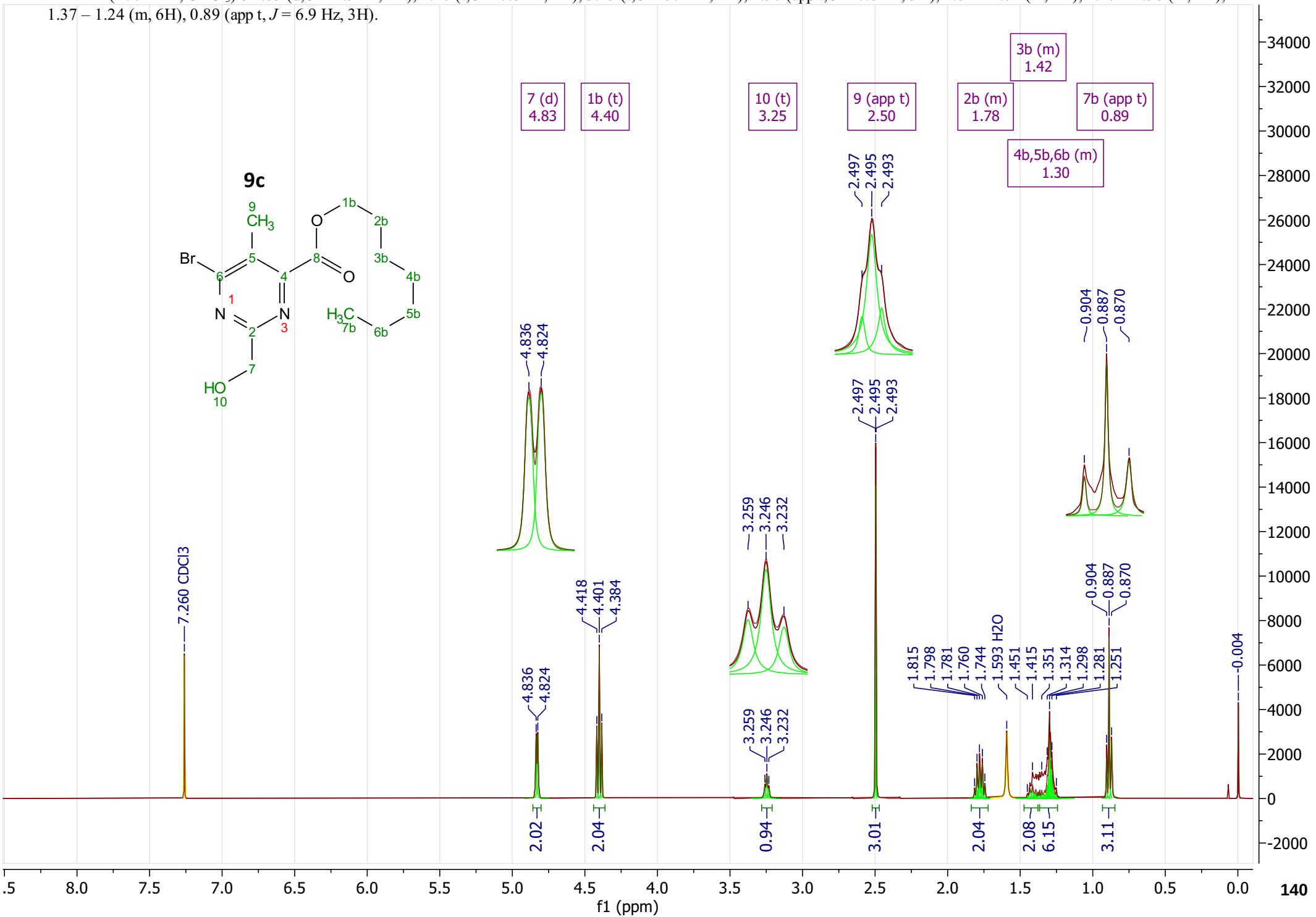
15N HMBC



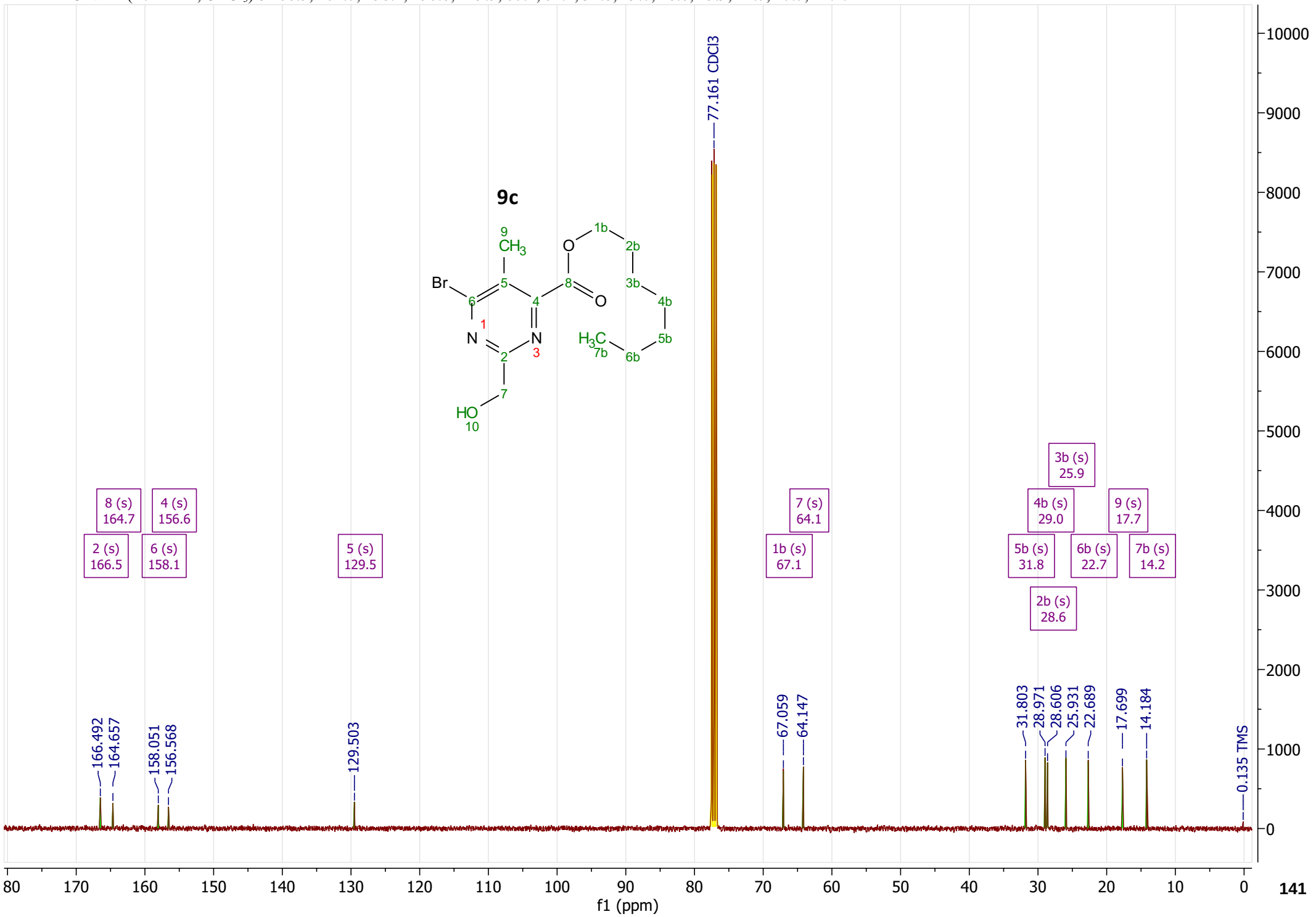
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ 293.60, 272.54.



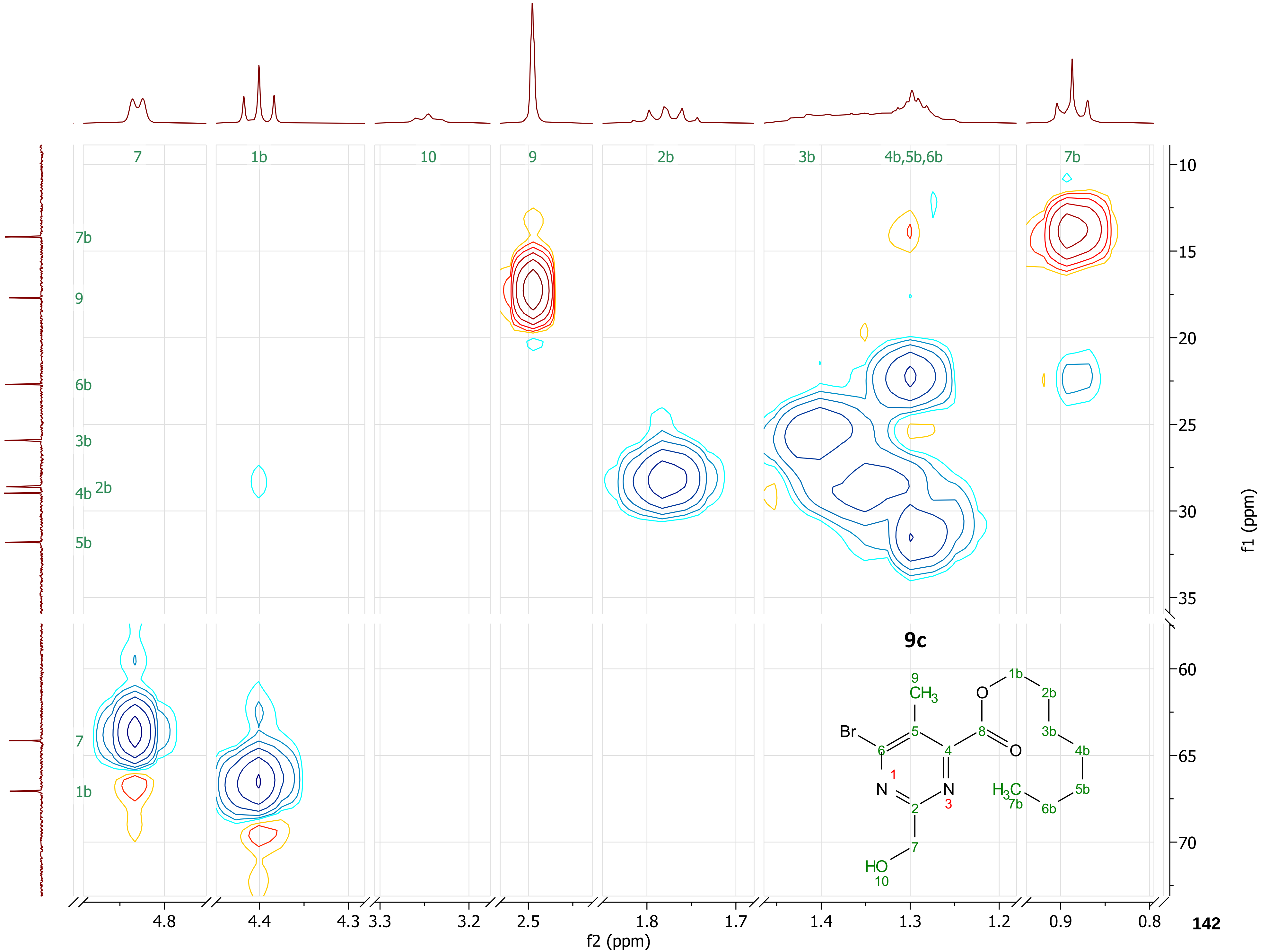
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (d, J = 4.5 Hz, 2H), 4.40 (t, J = 6.8 Hz, 2H), 3.25 (t, J = 5.4 Hz, 1H), 2.50 (app t, J = 0.8 Hz, 3H), 1.84 – 1.72 (m, 2H), 1.47 – 1.38 (m, 2H), 1.37 – 1.24 (m, 6H), 0.89 (app t, J = 6.9 Hz, 3H).



13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.7, 158.1, 156.6, 129.5, 67.1, 64.1, 31.8, 29.0, 28.6, 25.9, 22.7, 17.7, 14.2.

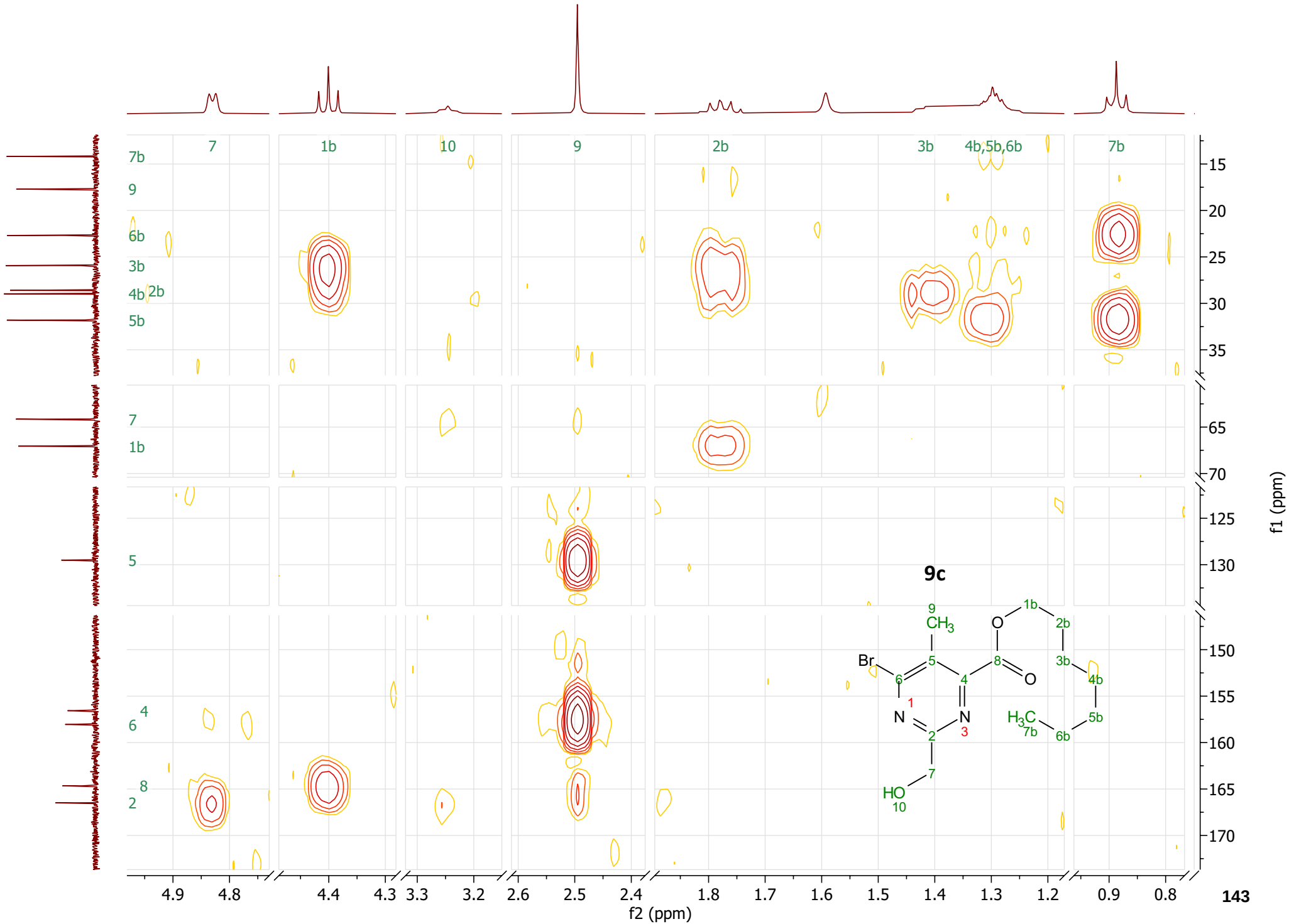


13C HSQC

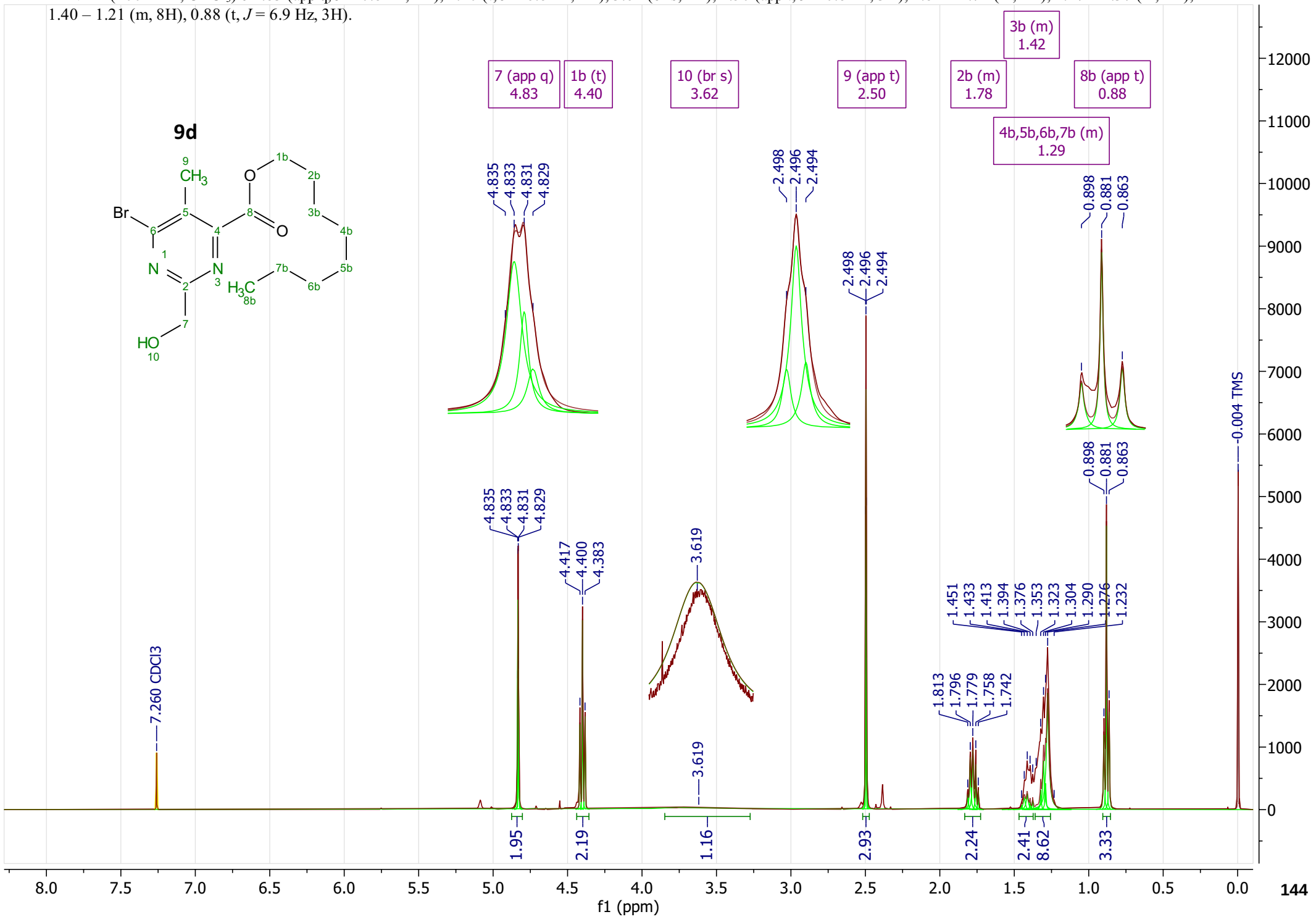


7
1b
10
9
2b
3b
4b,5b,6b
7b
7b
9
6b
3b
4b
2b
5b
7
1b
9c
9
CH3
O
1b
2b
3b
4b
5b
6b
H3C
7b
Br
6
5
4
8
O
1
N
N
3
2
7
HO
10
4.8
4.4
4.3
3.3
3.2
2.5
1.8
1.7
1.4
1.3
1.2
0.9
0.8
f2 (ppm)
10
15
20
25
30
35
60
65
70
f1 (ppm)

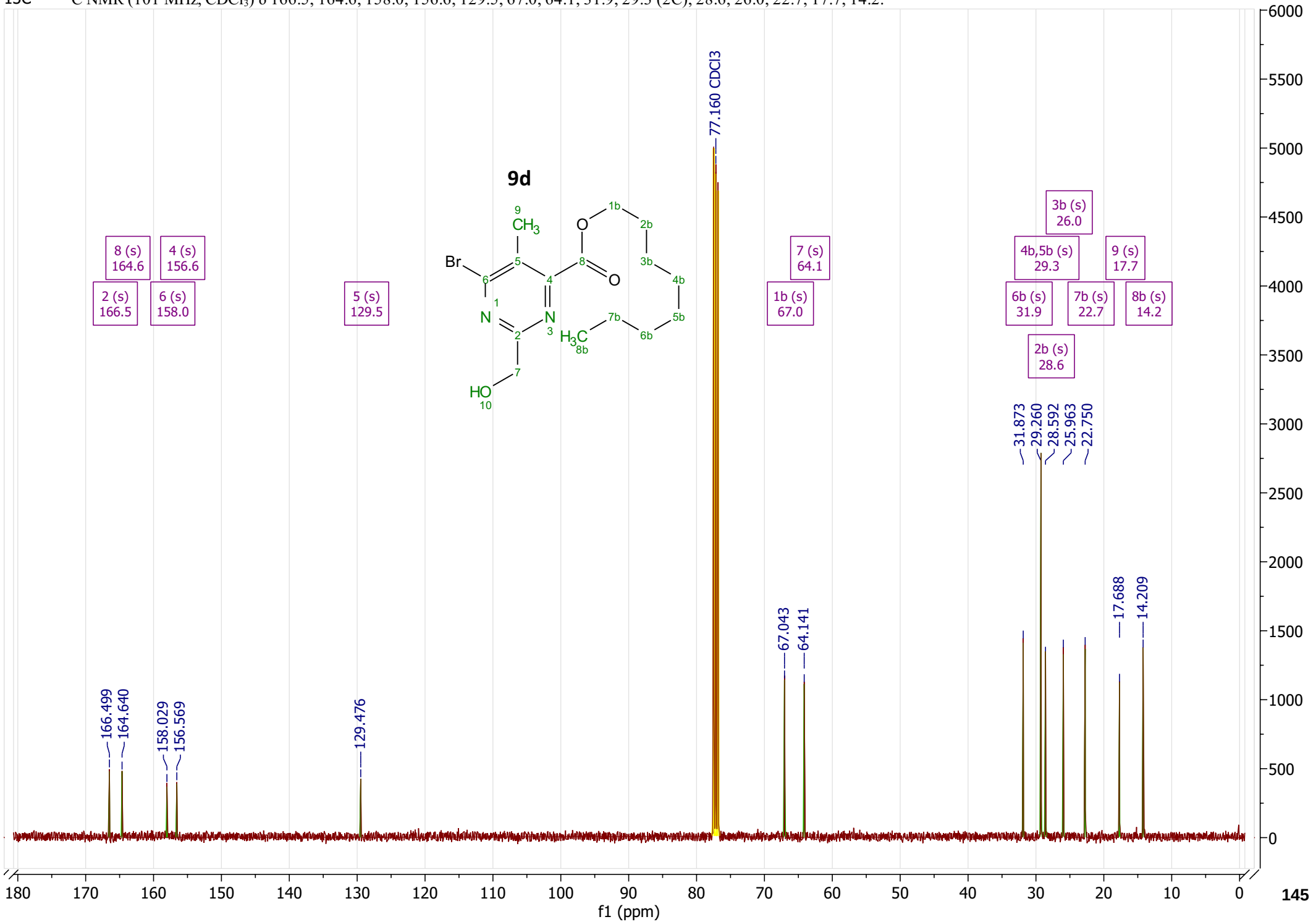
13C HMBC


7
1b
10
9
2b
3b
4b,5b,6b
7b
7b
9
6b
3b
4b 2b
5b
7
1b
5
6 4
8
2
9c
9
CH3
1b
O
2b
Br
5
8
3b
4b
6
4
O
1
N
N
2
3
H3C
7b
6b
5b
7
HO
10
15
20
25
30
35
65
70
125
130
150
155
160
165
170
f1 (ppm)
4.9
4.8
4.4
4.3
3.3
3.2
2.6
2.5
2.4
1.8
1.7
1.6
1.5
1.4
1.3
1.2
0.9
0.8
f2 (ppm)

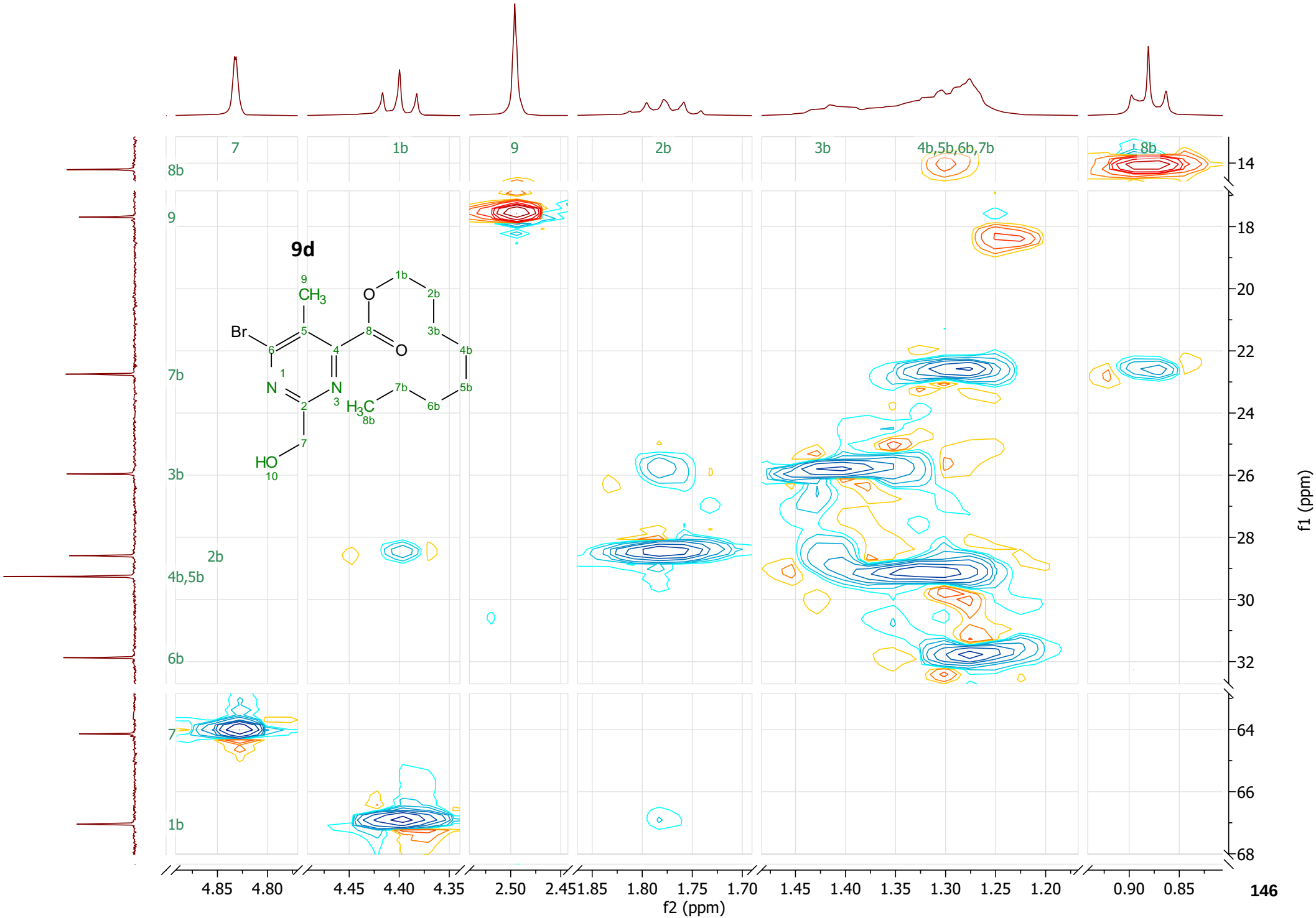
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (app q, J = 0.8 Hz, 2H), 4.40 (t, J = 6.8 Hz, 2H), 3.62 (br s, 1H), 2.50 (app t, J = 0.8 Hz, 3H), 1.84 – 1.72 (m, 2H), 1.47 – 1.37 (m, 2H), 1.40 – 1.21 (m, 8H), 0.88 (t, J = 6.9 Hz, 3H).



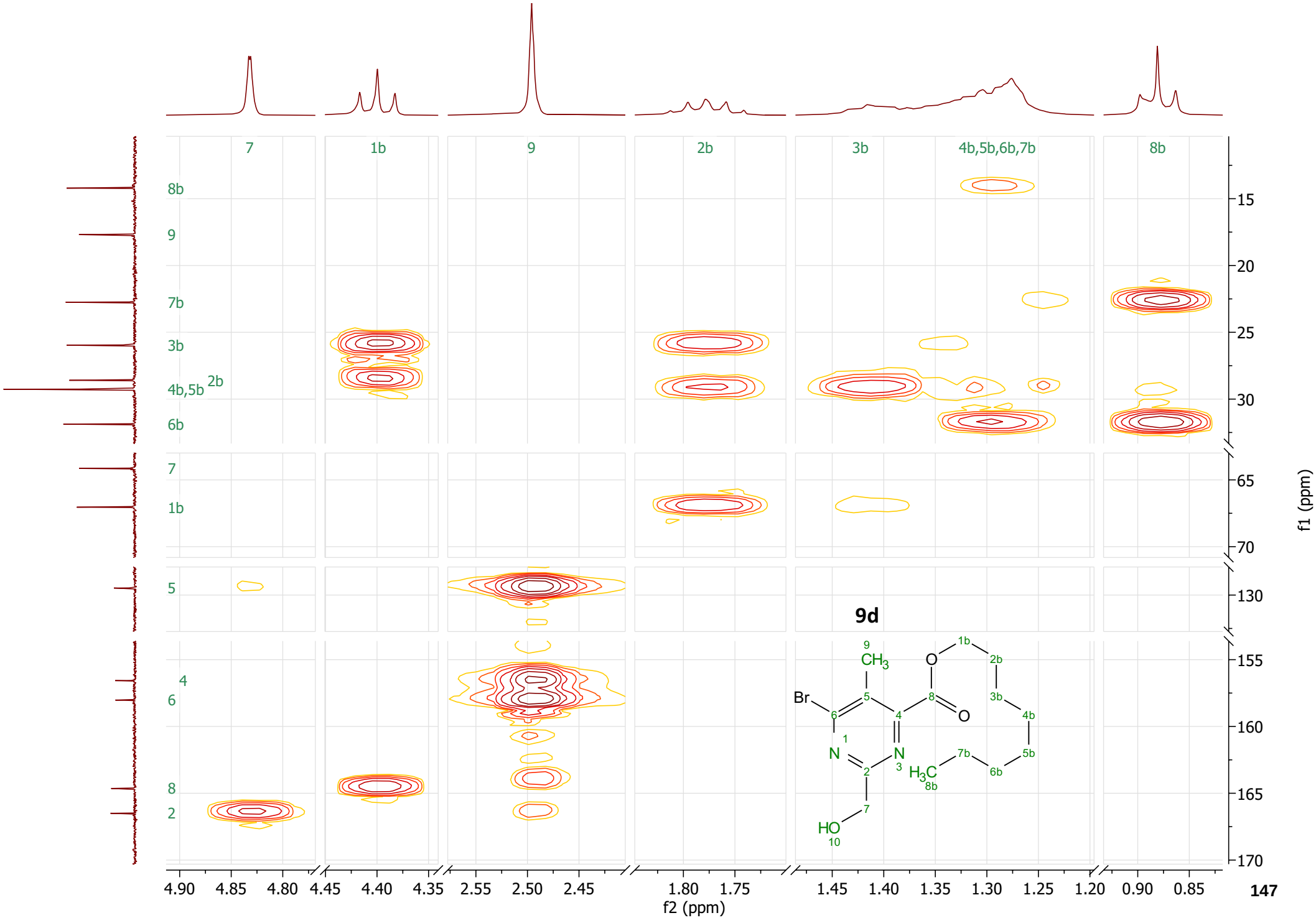
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.6, 158.0, 156.6, 129.5, 67.0, 64.1, 31.9, 29.3 (2C), 28.6, 26.0, 22.7, 17.7, 14.2.



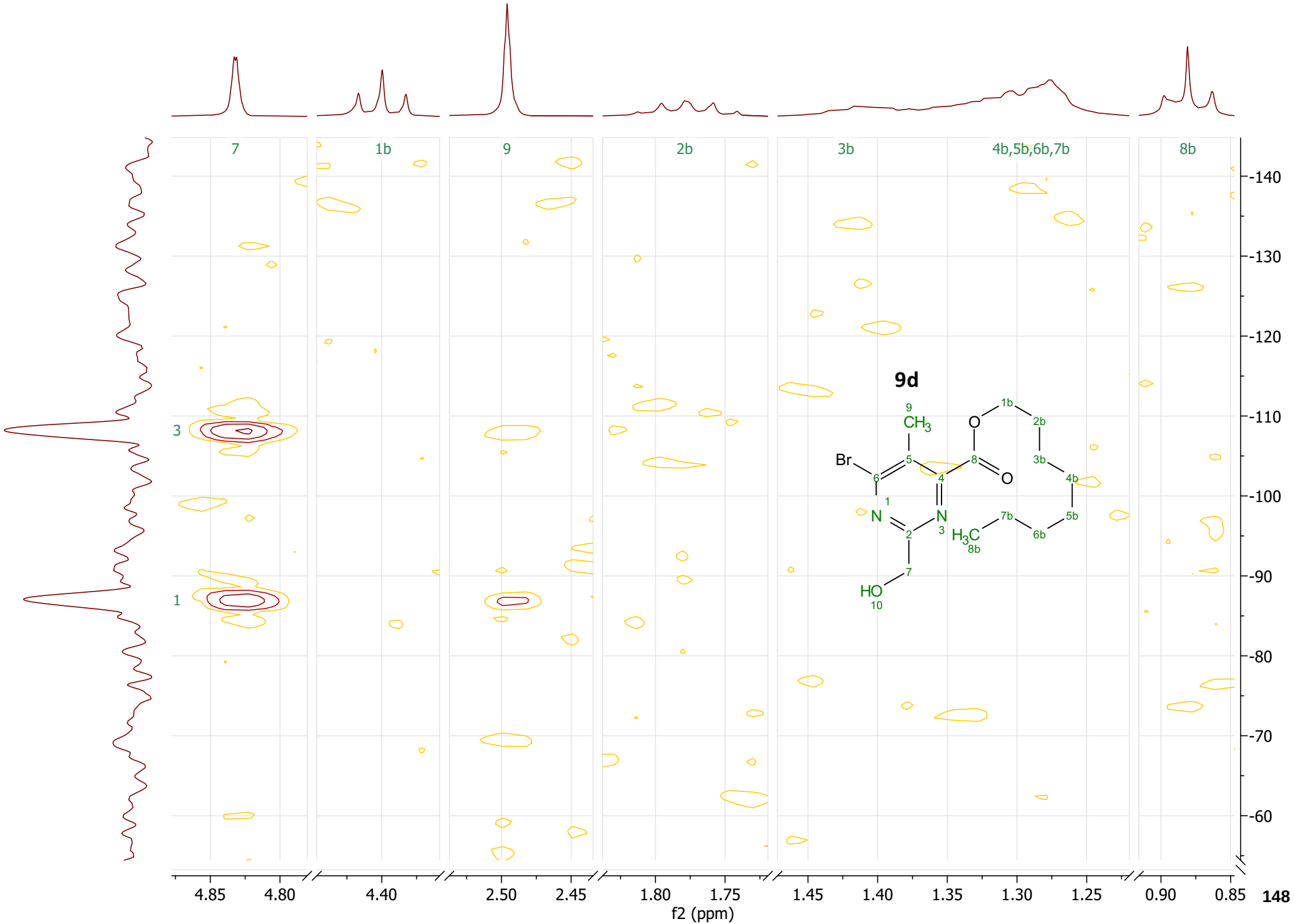
13C HSQC


7
1b
9
2b
3b
4b,5b,6b,7b
8b
8b
9
7b
3b
2b
4b,5b
6b
7
1b
9d
Br
CH3
O
O
N
N
H3C
HO
14
18
20
22
24
26
28
30
32
64
66
68
f1 (ppm)
4.85
4.80
4.45
4.40
4.35
2.50
2.45
1.85
1.80
1.75
1.70
1.45
1.40
1.35
1.30
1.25
1.20
0.90
0.85
f2 (ppm)

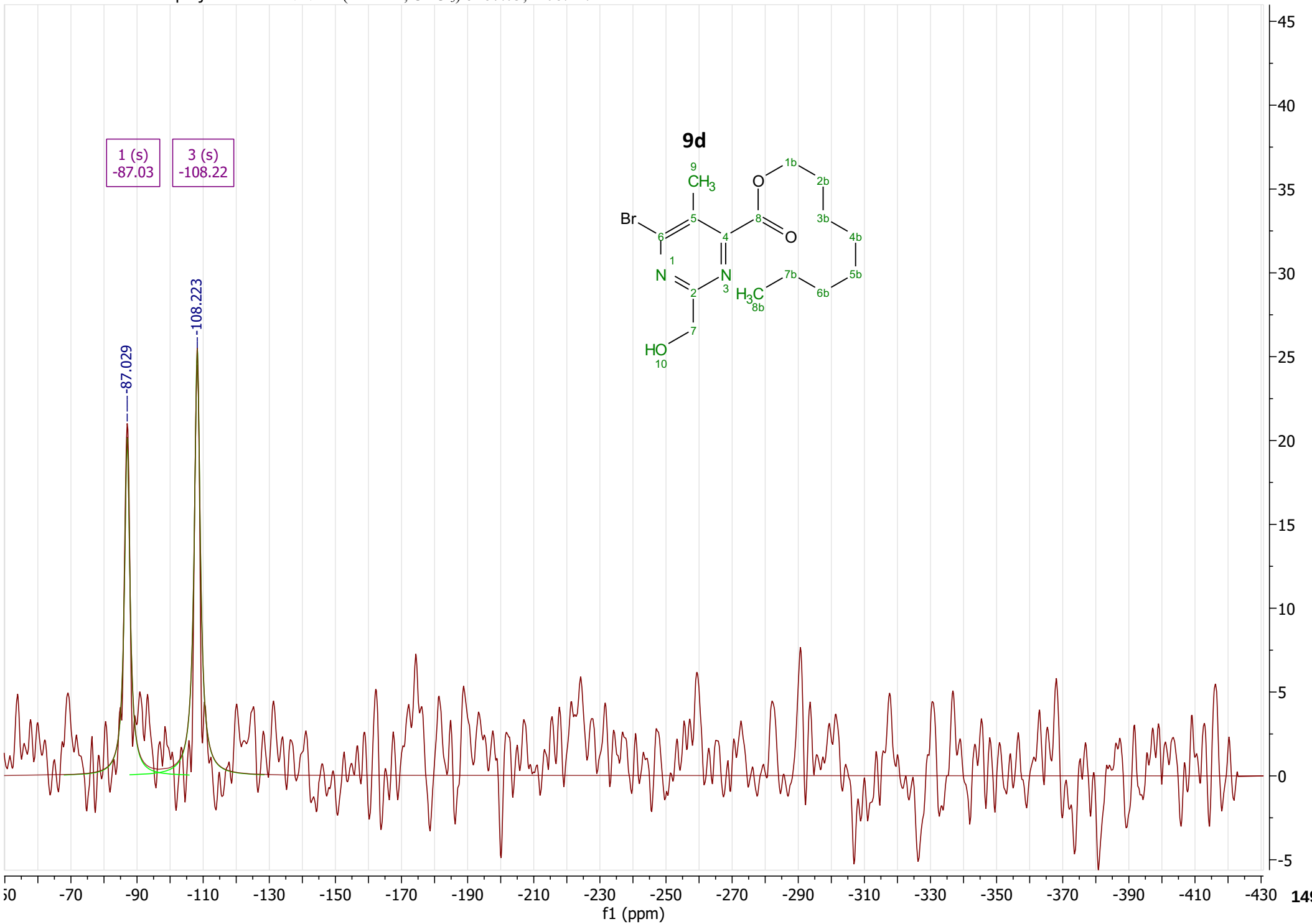
13C HMBC


7
1b
9
2b
3b
4b,5b,6b,7b
8b
8b
9
7b
3b
4b,5b 2b
6b
7
1b
5
4
6
8
2
9d
f1 (ppm)
f2 (ppm)
4.90 4.85 4.80 4.45 4.40 4.35 2.55 2.50 2.45 1.80 1.75 1.45 1.40 1.35 1.30 1.25 1.20 0.90 0.85
15 20 25 30 65 70 130 155 160 165 170

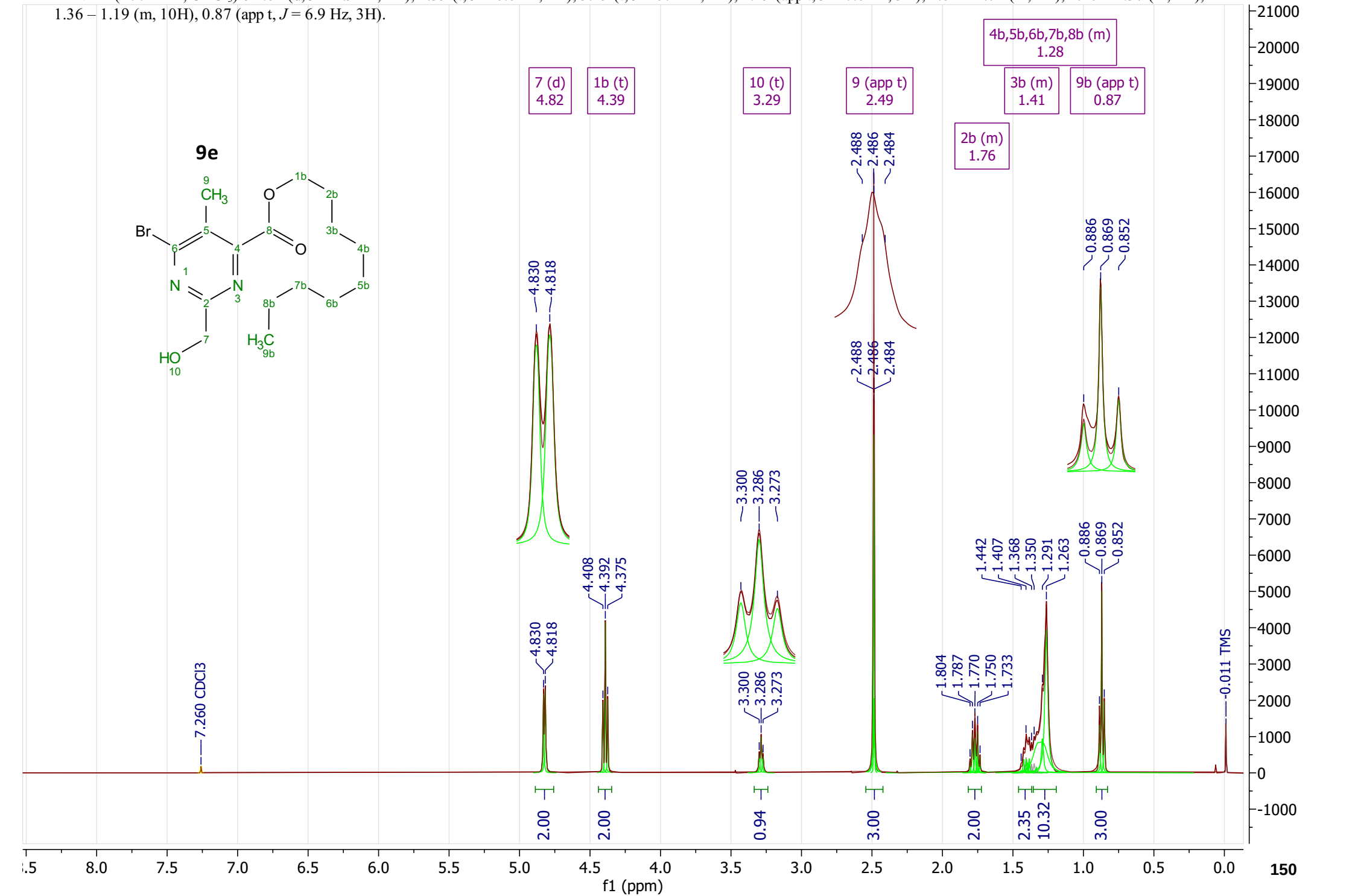
15N HMBC


7
1b
9
2b
3b
4b,5b,6b,7b
8b
3
1
9d
f2 (ppm)
f1 (ppm)
4.85
4.80
4.40
2.50
2.45
1.80
1.75
1.45
1.40
1.35
1.30
1.25
0.90
0.85
-140
-130
-120
-110
-100
-90
-80
-70
-60

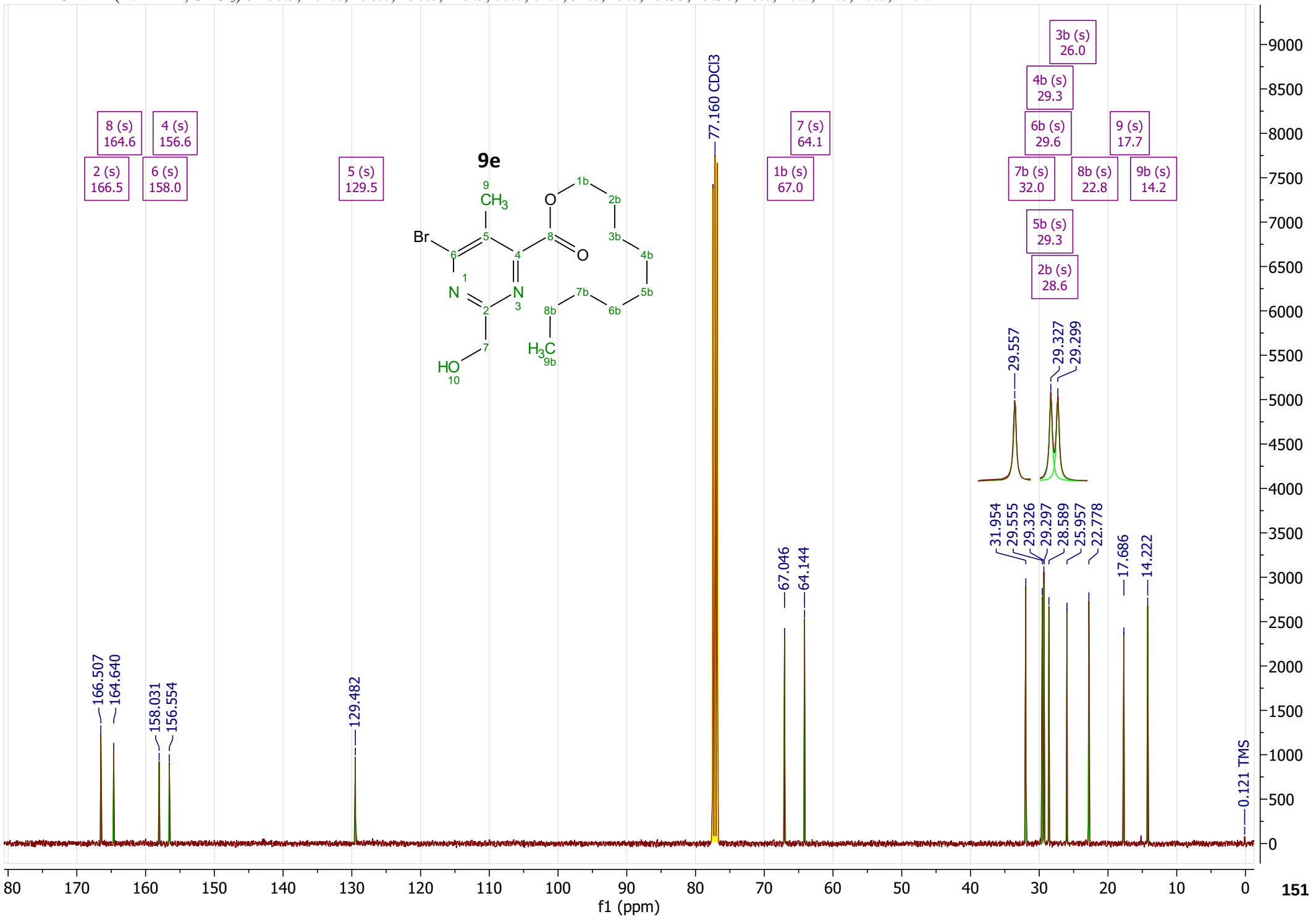
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -87.03, -108.22.



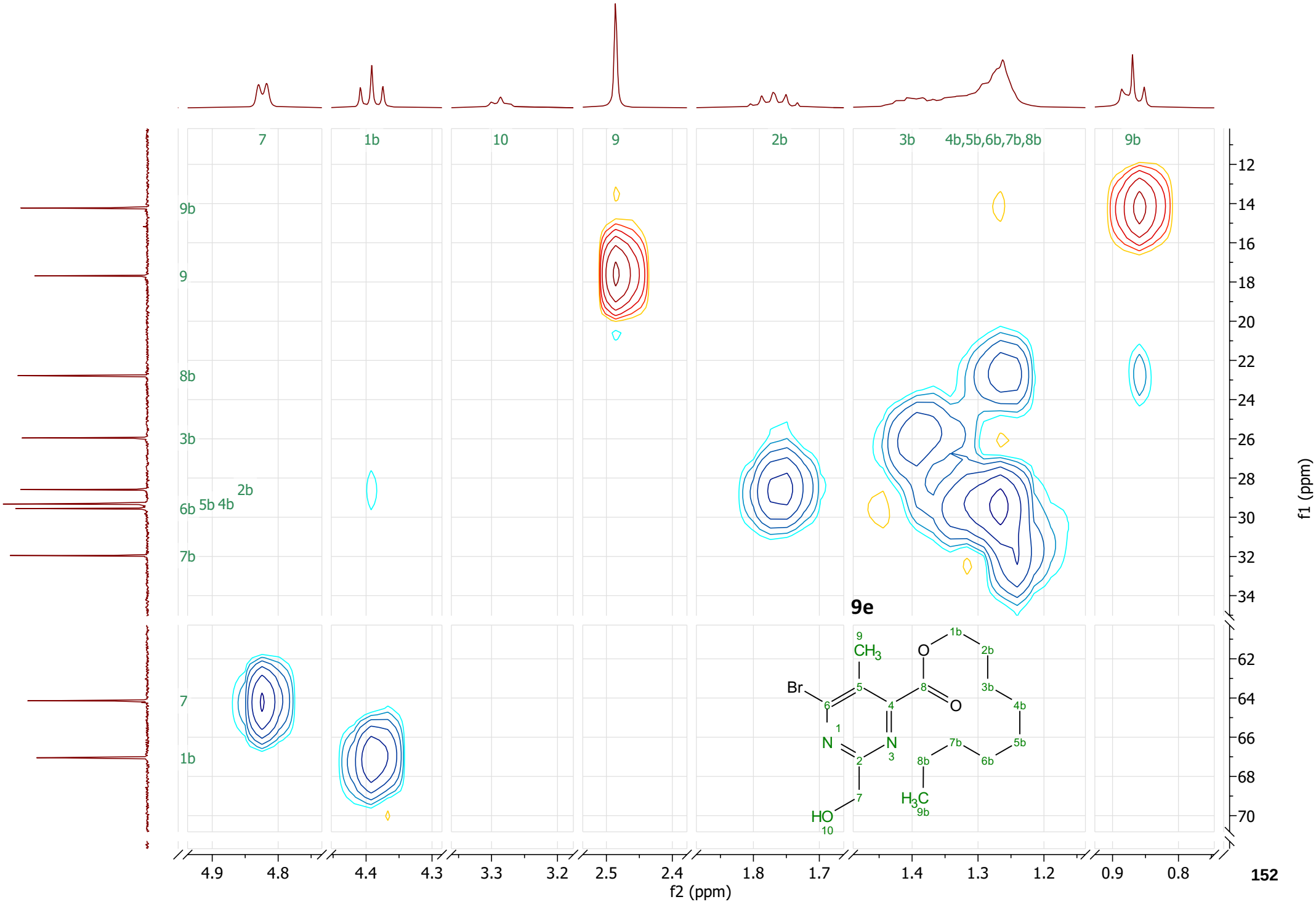
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (d, *J* = 4.9 Hz, 2H), 4.39 (t, *J* = 6.8 Hz, 2H), 3.29 (t, *J* = 5.4 Hz, 1H), 2.49 (app t, *J* = 0.8 Hz, 3H), 1.82 – 1.72 (m, 2H), 1.46 – 1.37 (m, 2H), 1.36 – 1.19 (m, 10H), 0.87 (app t, *J* = 6.9 Hz, 3H).



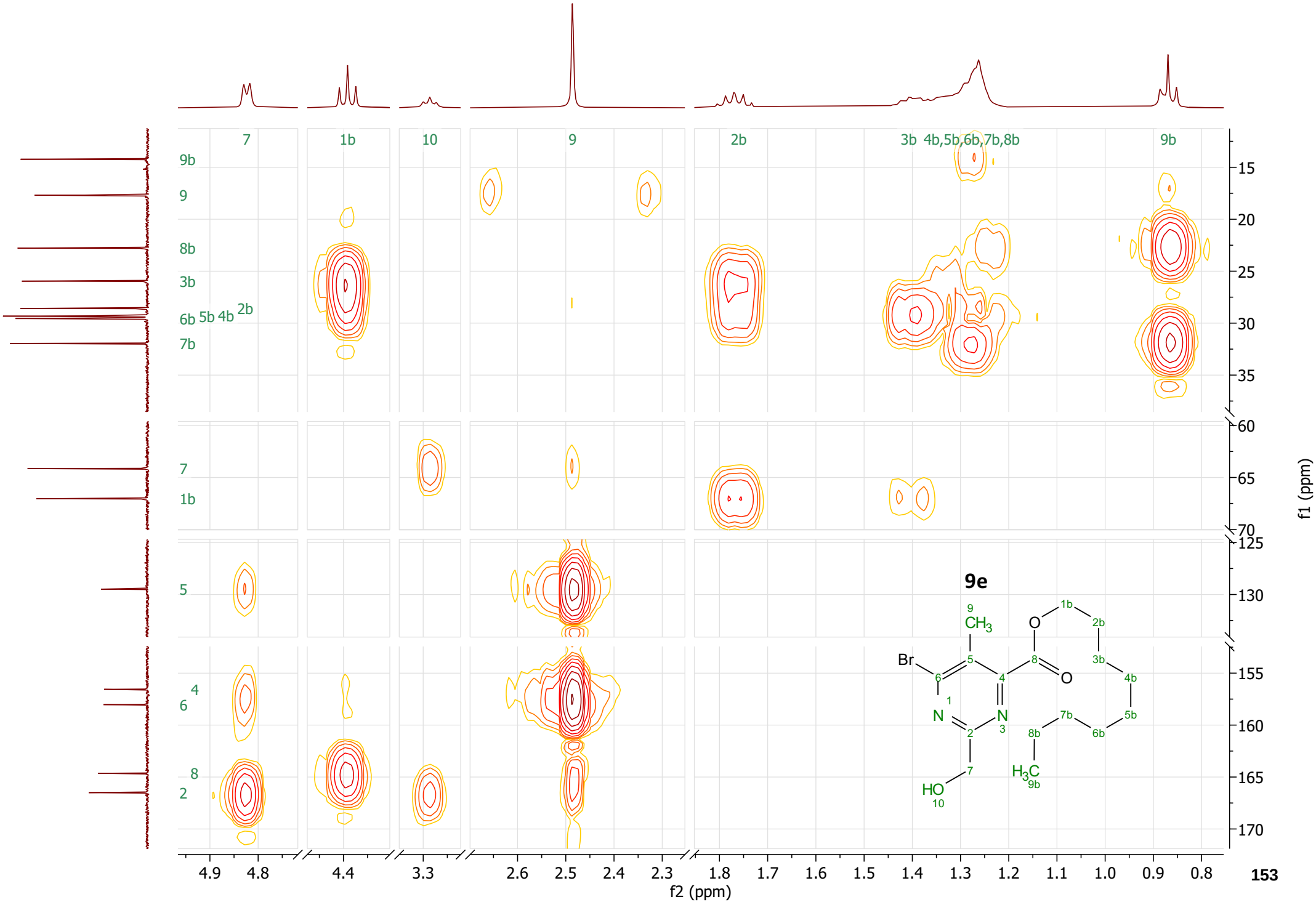
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.6, 158.0, 156.6, 129.5, 67.0, 64.1, 32.0, 29.6, 29.33, 29.30, 28.6, 26.0, 22.8, 17.7, 14.2.



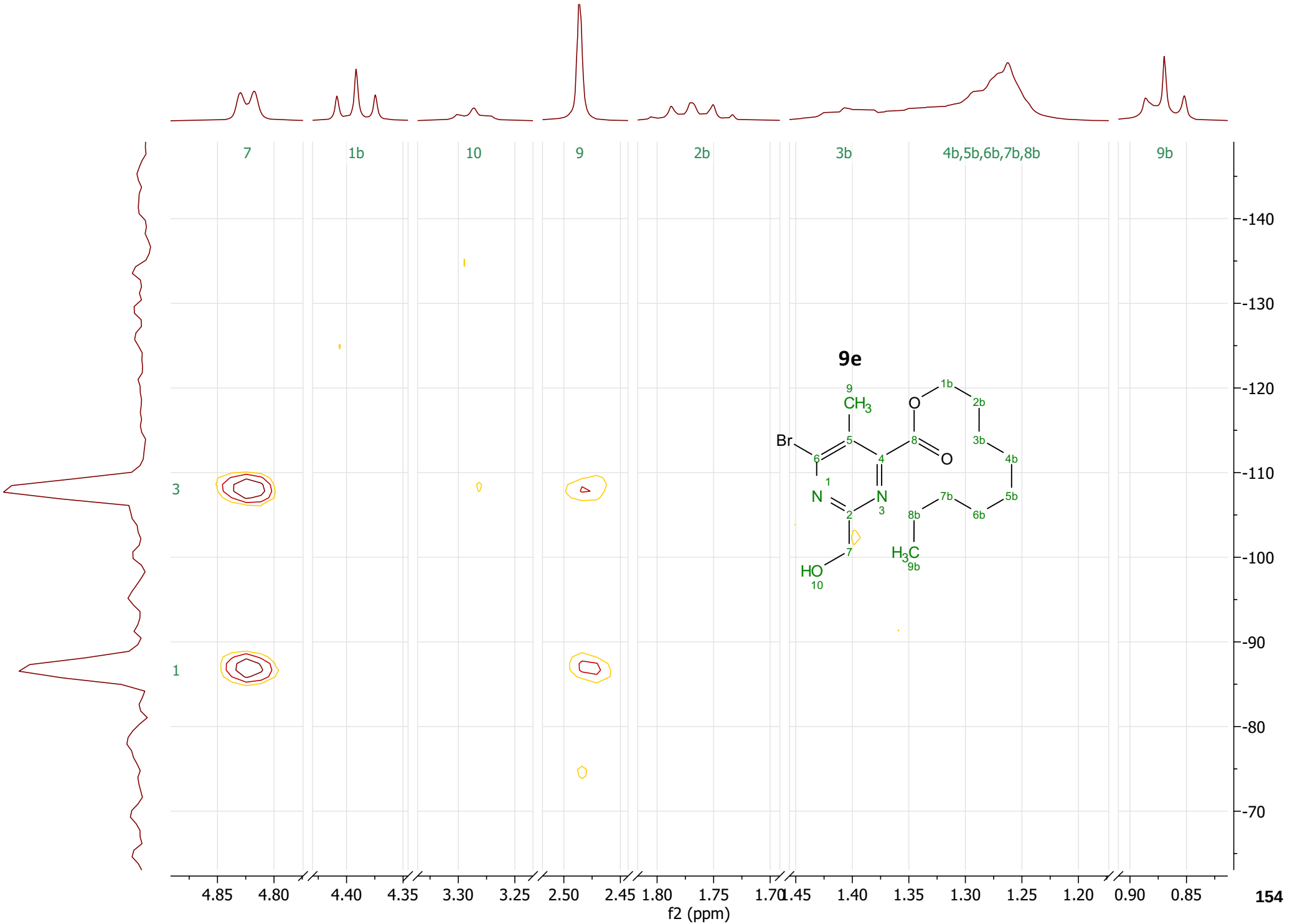
13C HSQC


7
1b
10
9
2b
3b
4b,5b,6b,7b,8b
9b
9b
9
8b
3b
6b
5b 4b
2b
7b
7
1b
9e
f1 (ppm)
12
14
16
18
20
22
24
26
28
30
32
34
62
64
66
68
70
4.9
4.8
4.4
4.3
3.3
3.2
2.5
2.4
1.8
1.7
1.4
1.3
1.2
0.9
0.8
f2 (ppm)
Br
CH3
O
O
N
N
HO
H3C
1
2
3
4
5
6
7
8
9
10
1b
2b
3b
4b
5b
6b
7b
8b
9b

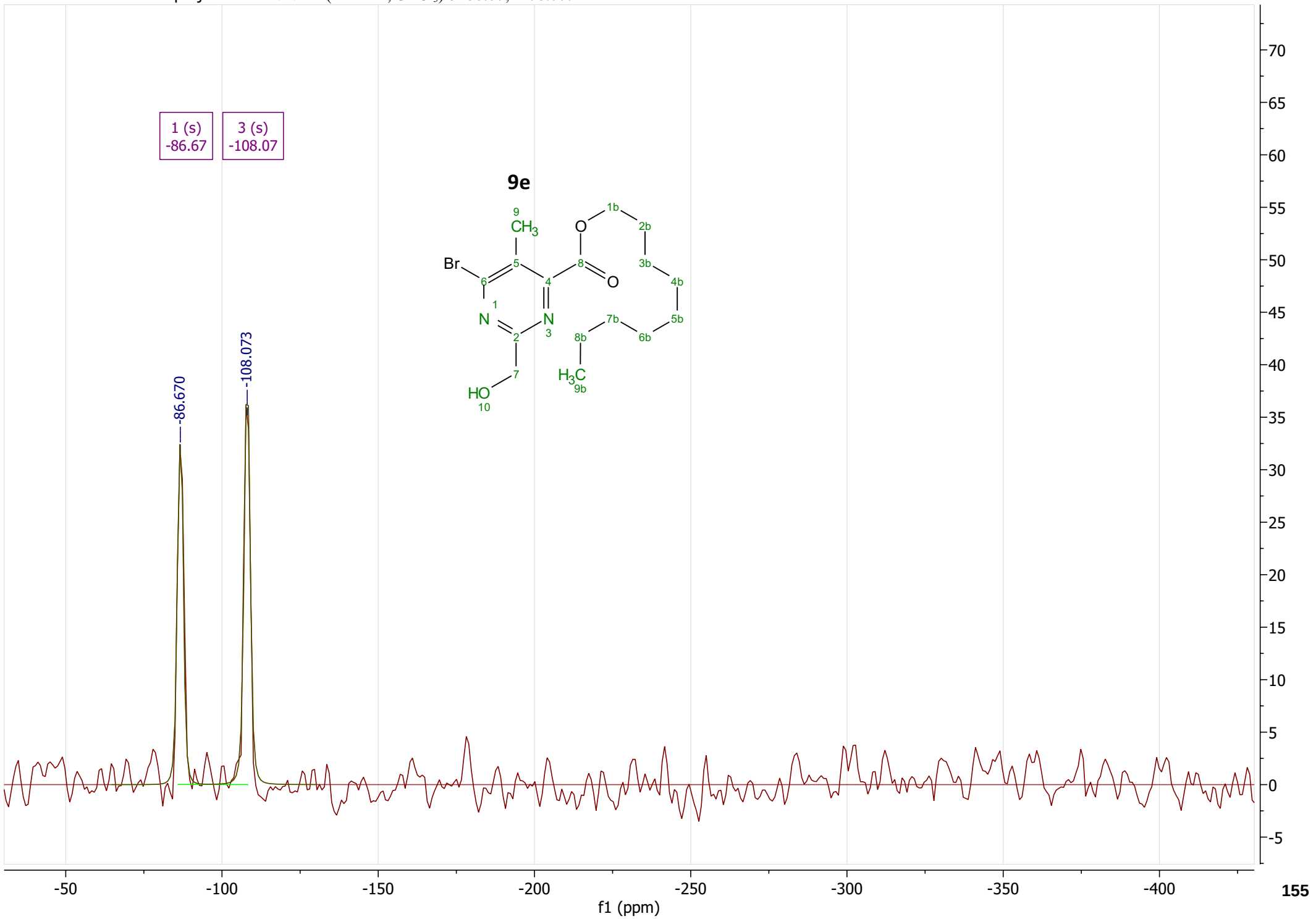
13C HMBC


7
1b
10
9
2b
3b 4b,5b,6b,7b,8b
9b
9b
9
8b
3b
6b 5b 4b 2b
7b
7
1b
5
6 4
8
2
f2 (ppm)
f1 (ppm)
9e
Br
CH3
HO
H3C
N
O

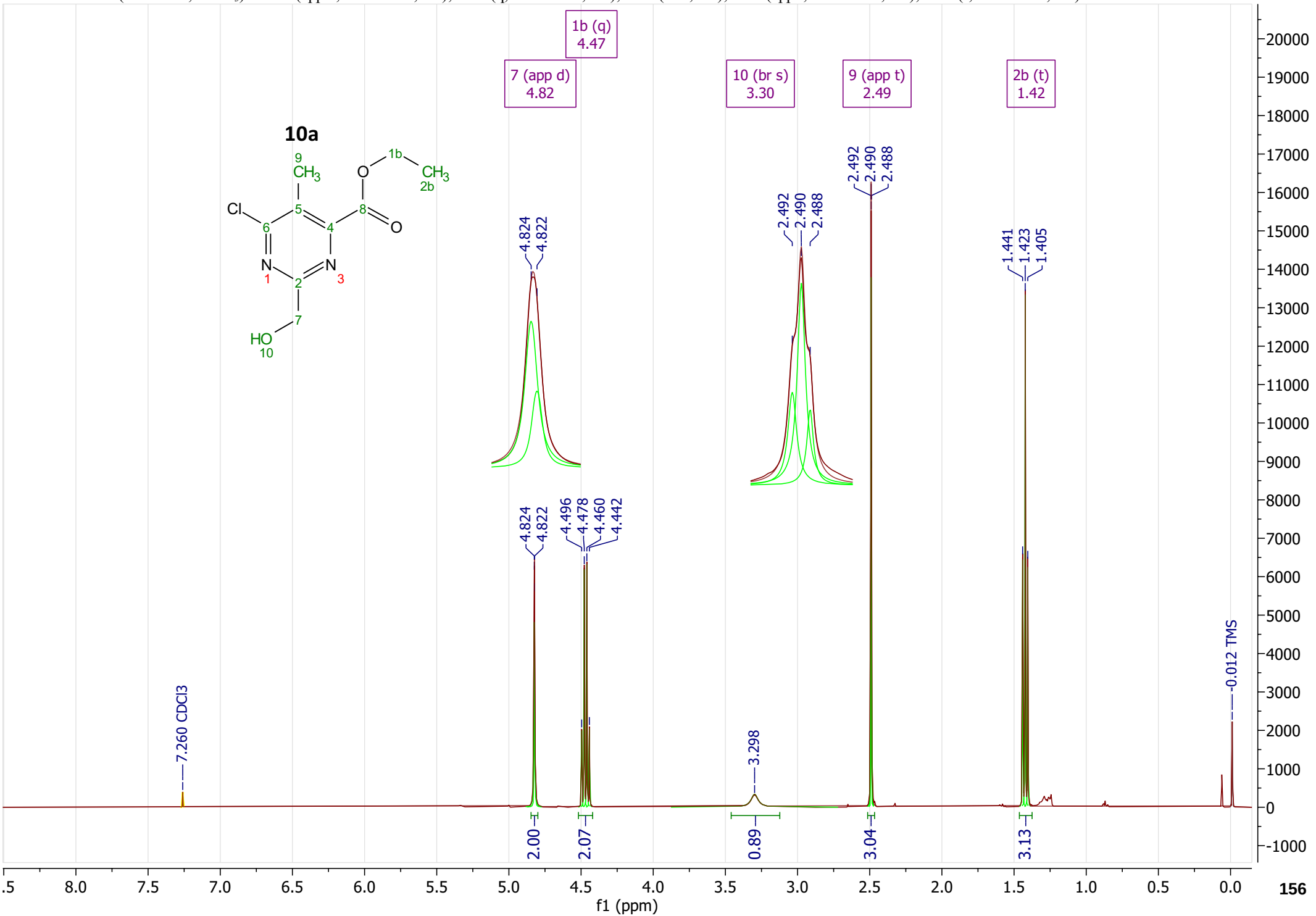
15N HMBC


7
1b
10
9
2b
3b
4b,5b,6b,7b,8b
9b
3
1
-140
-130
-120
-110
-100
-90
-80
-70
f1 (ppm)
4.85
4.80
4.40
4.35
3.30
3.25
2.50
2.45
1.80
1.75
1.70
1.45
1.40
1.35
1.30
1.25
1.20
0.90
0.85
f2 (ppm)
9e
CH3
Br
O
HO
N
H3C
9b
10

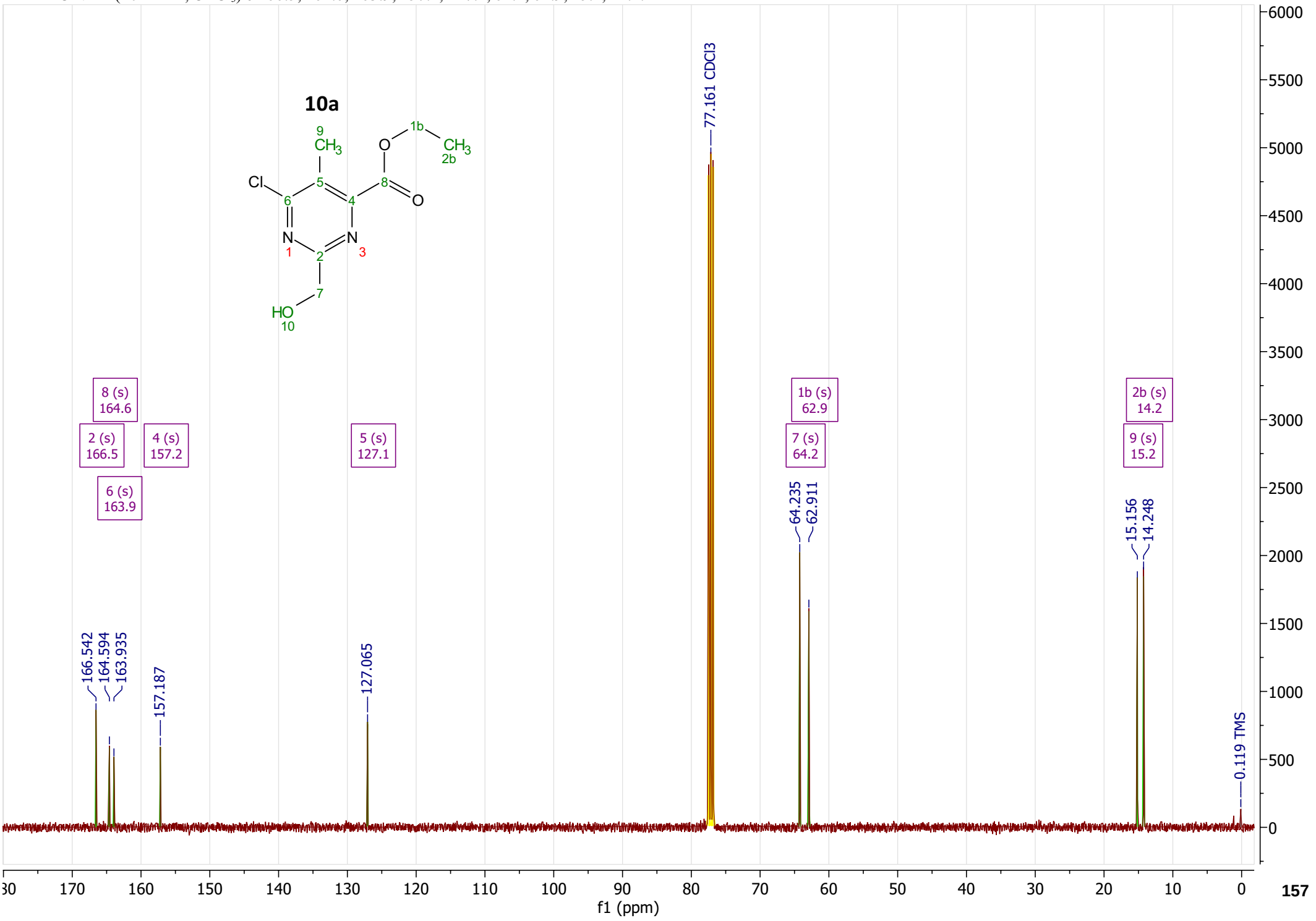
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -86.67, -108.07.



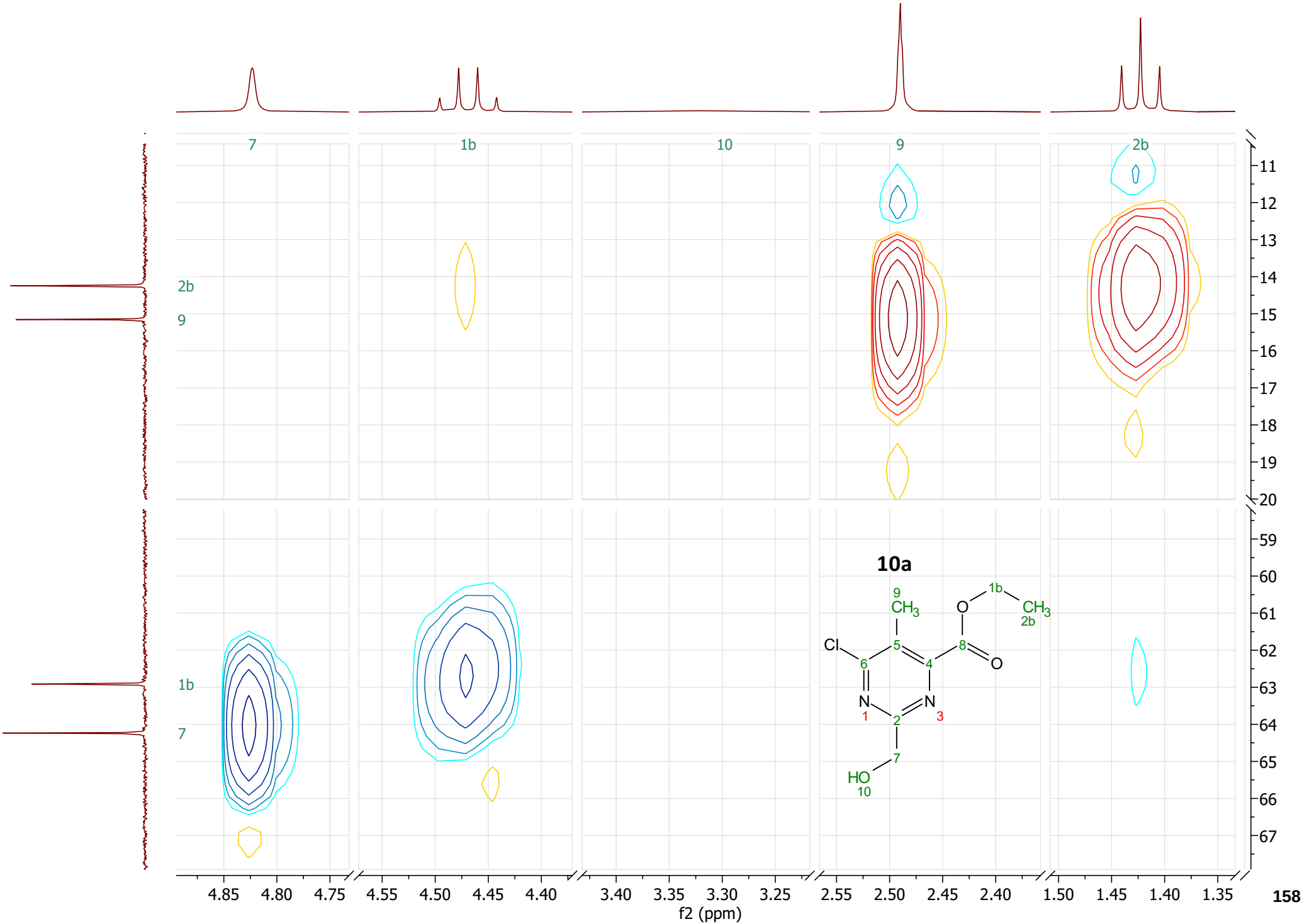
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (app d, J = 0.8 Hz, 2H), 4.47 (q, J = 7.1 Hz, 2H), 3.30 (br s, 1H), 2.49 (app t, J = 0.8 Hz, 3H), 1.42 (t, J = 7.1 Hz, 3H).



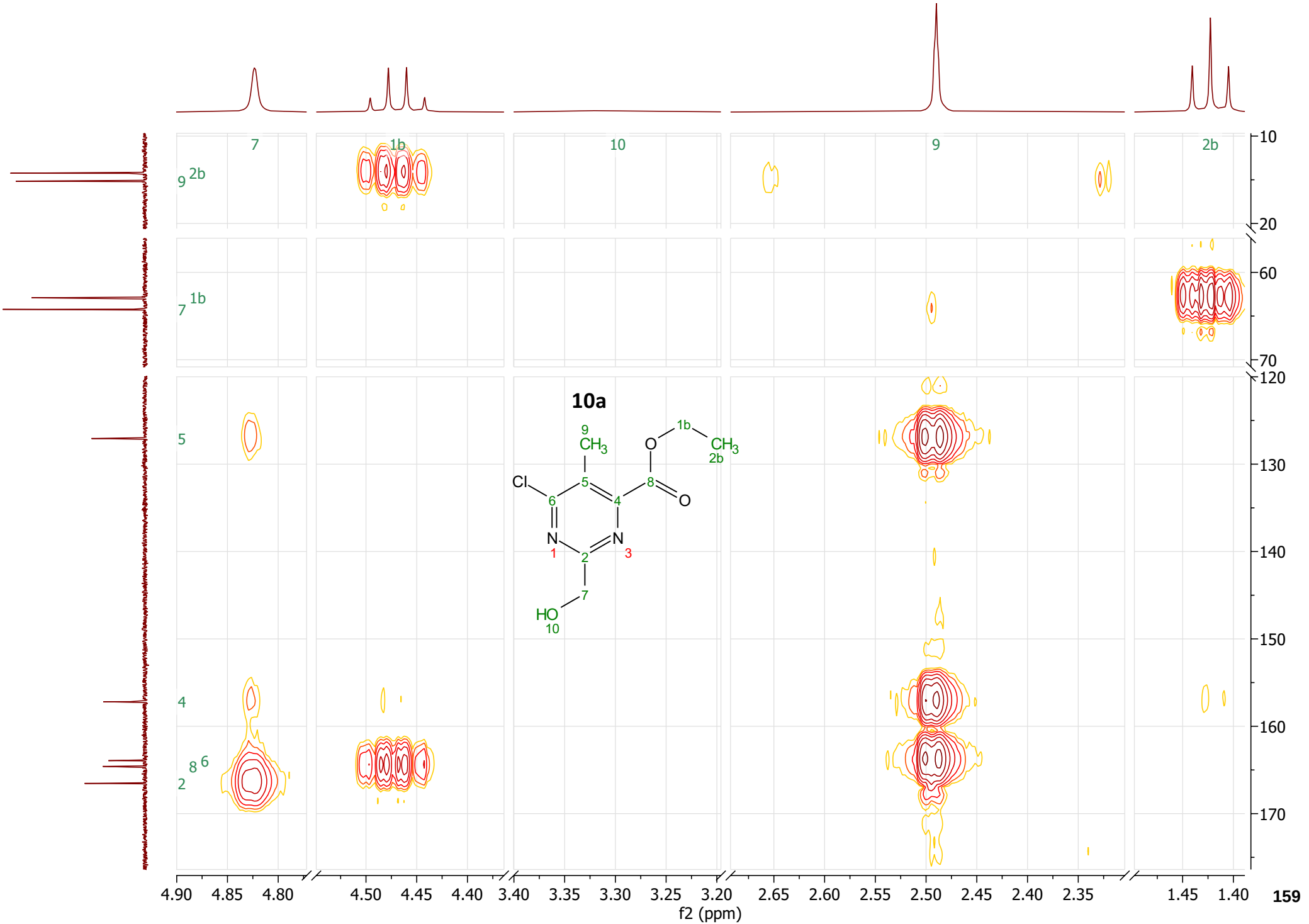
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.6, 163.9, 157.2, 127.1, 64.2, 62.9, 15.2, 14.2.



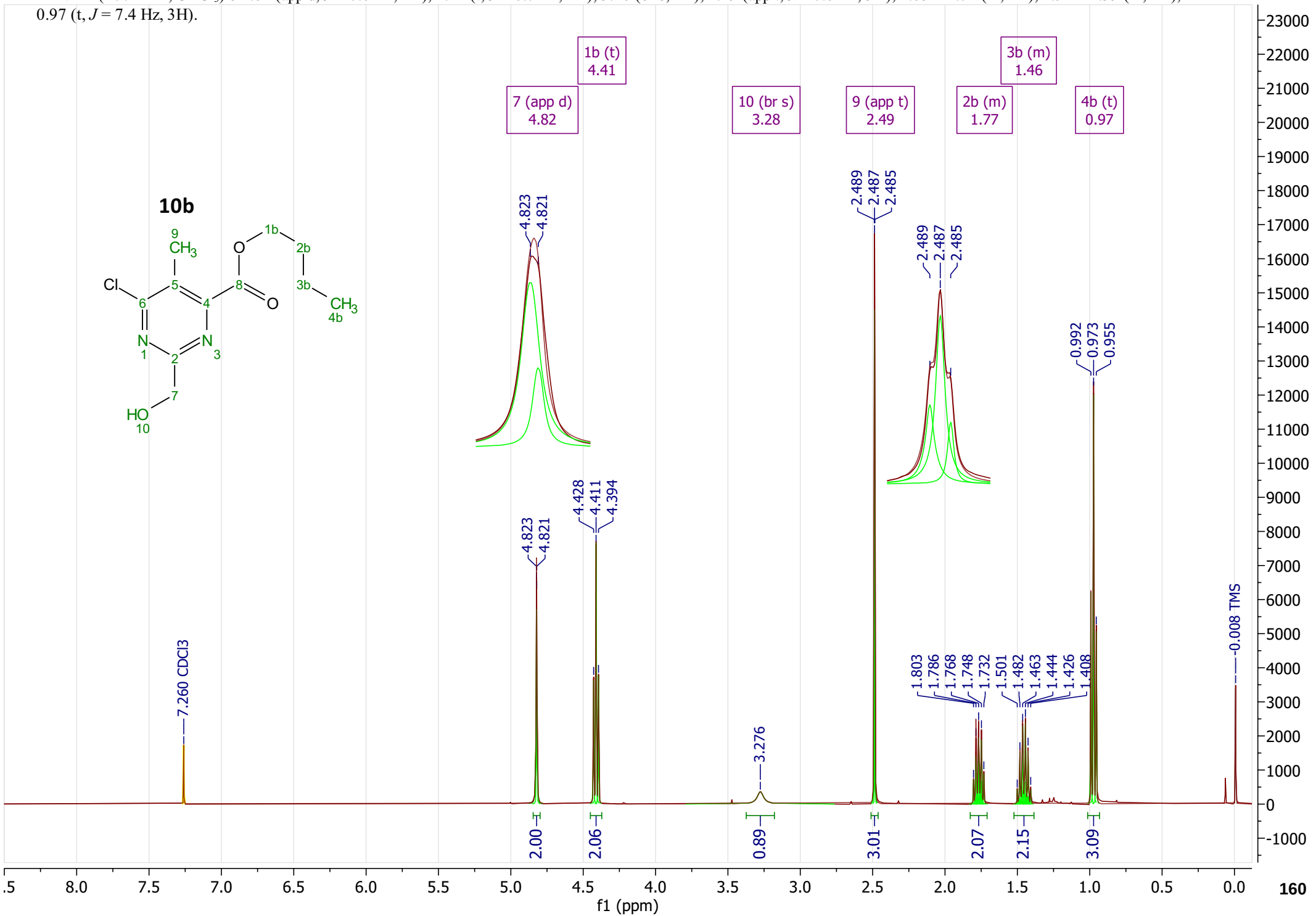
13C HSQC


7
1b
10
9
2b
2b
9
1b
7
10a
9
CH3
1b
O
CH3
2b
Cl
5
8
6
4
O
N
N
1
2
3
7
HO
10
f2 (ppm)
f1 (ppm)

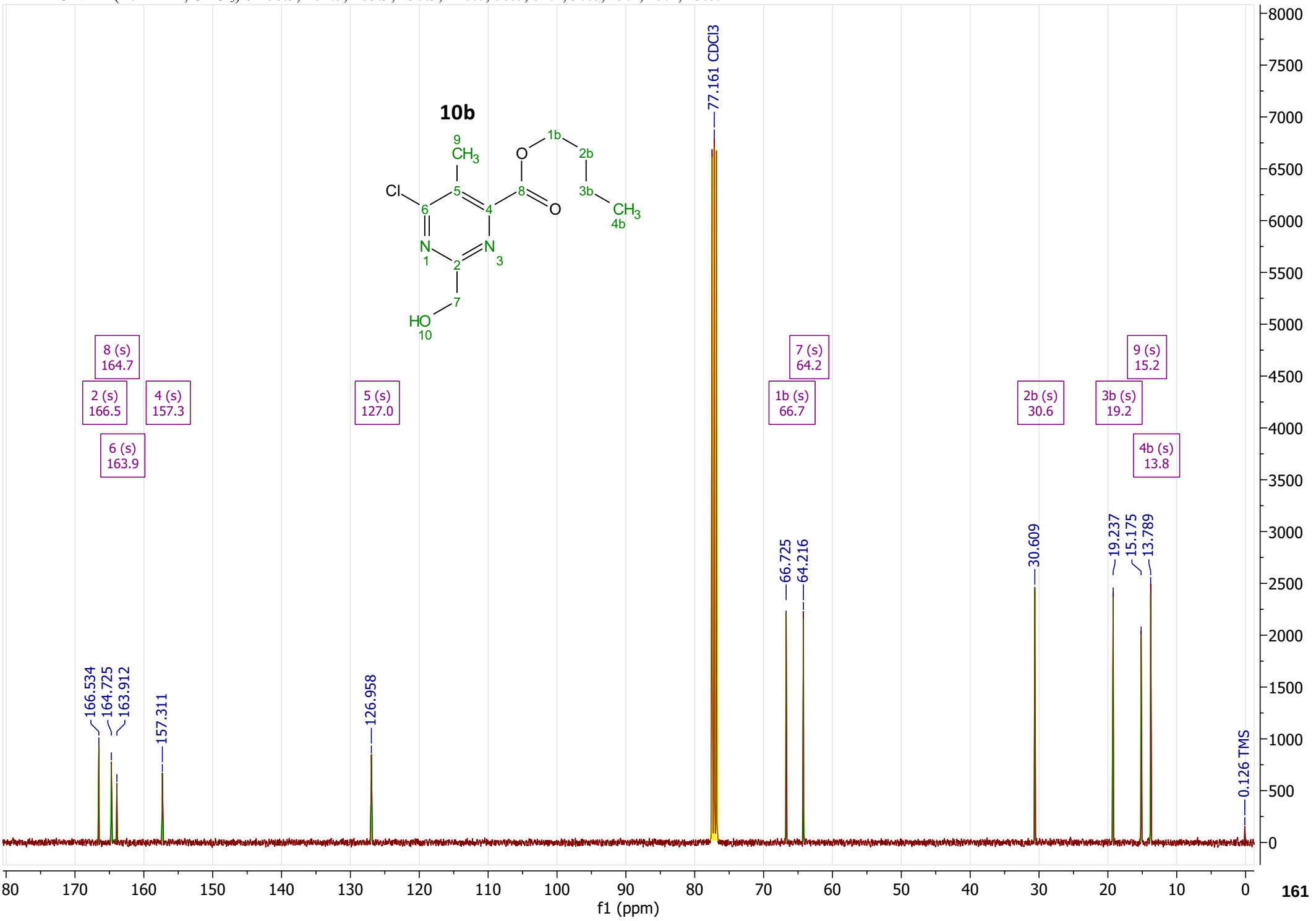
13C HMBC


10a
9
CH3
1b
O
CH3
2b
Cl
6
5
4
8
O
N
1
2
N
3
7
HO
10
7
1b
9
2b
10
9
2b
5
4
8
6
2
f2 (ppm)
f1 (ppm)
4.90
4.85
4.80
4.50
4.45
4.40
3.40
3.35
3.30
3.25
3.20
2.65
2.60
2.55
2.50
2.45
2.40
2.35
1.45
1.40
10
20
60
70
120
130
140
150
160
170

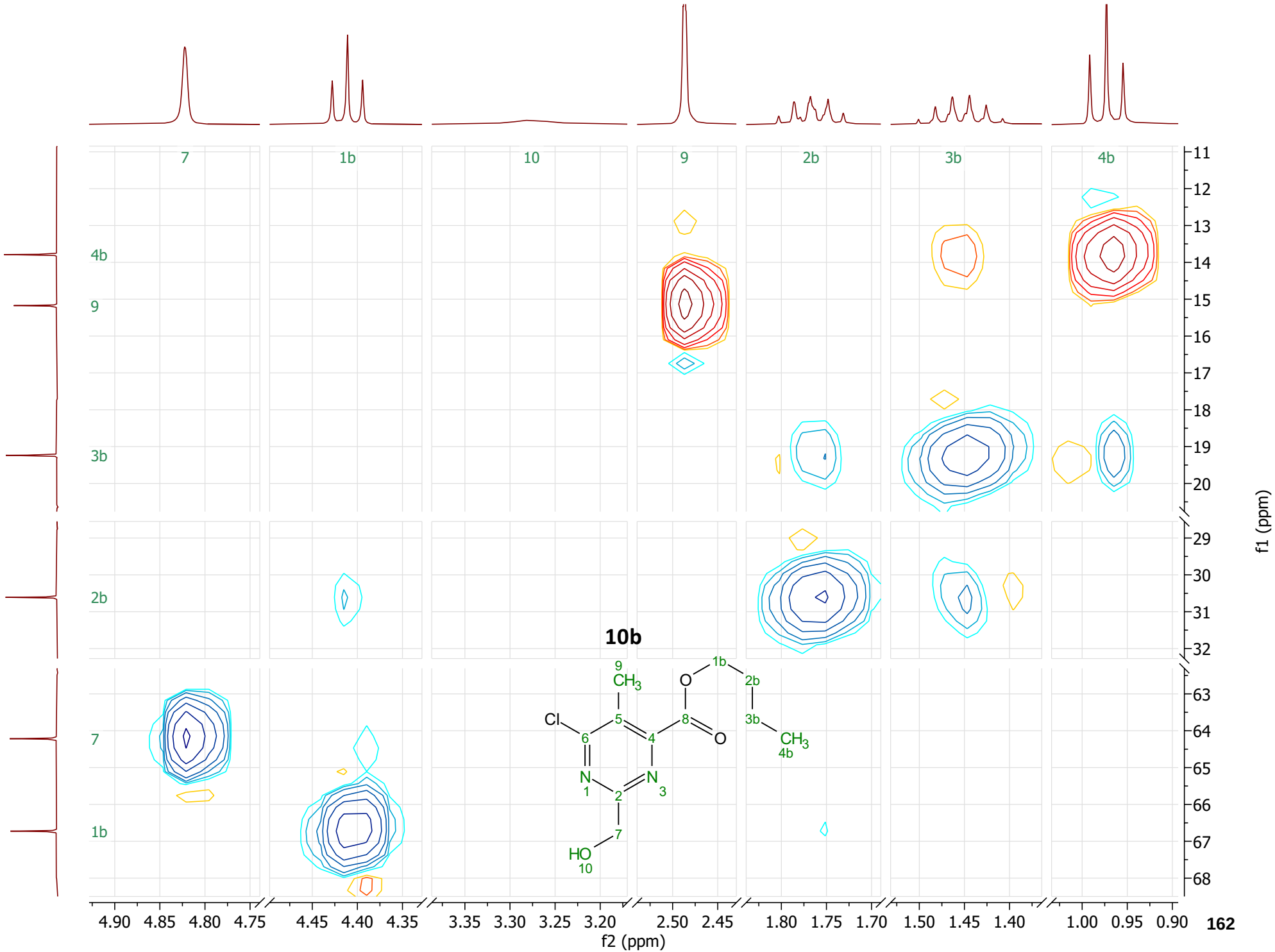
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (app d, *J* = 0.8 Hz, 2H), 4.41 (t, *J* = 6.7 Hz, 2H), 3.28 (br s, 1H), 2.49 (app t, *J* = 0.8 Hz, 3H), 1.83 – 1.71 (m, 2H), 1.52 – 1.39 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H).



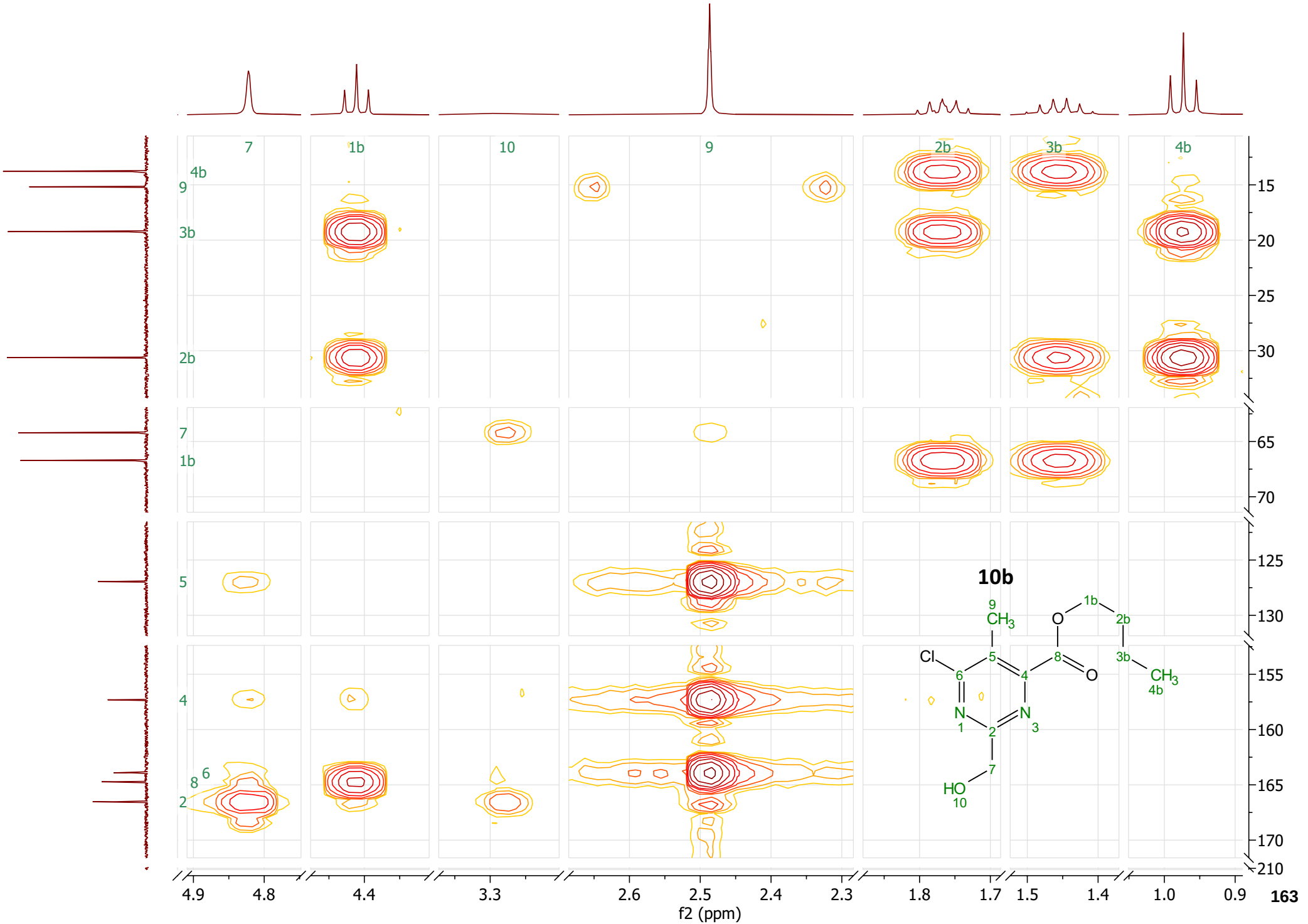
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.7, 163.9, 157.3, 127.0, 66.7, 64.2, 30.6, 19.2, 15.2, 13.8.



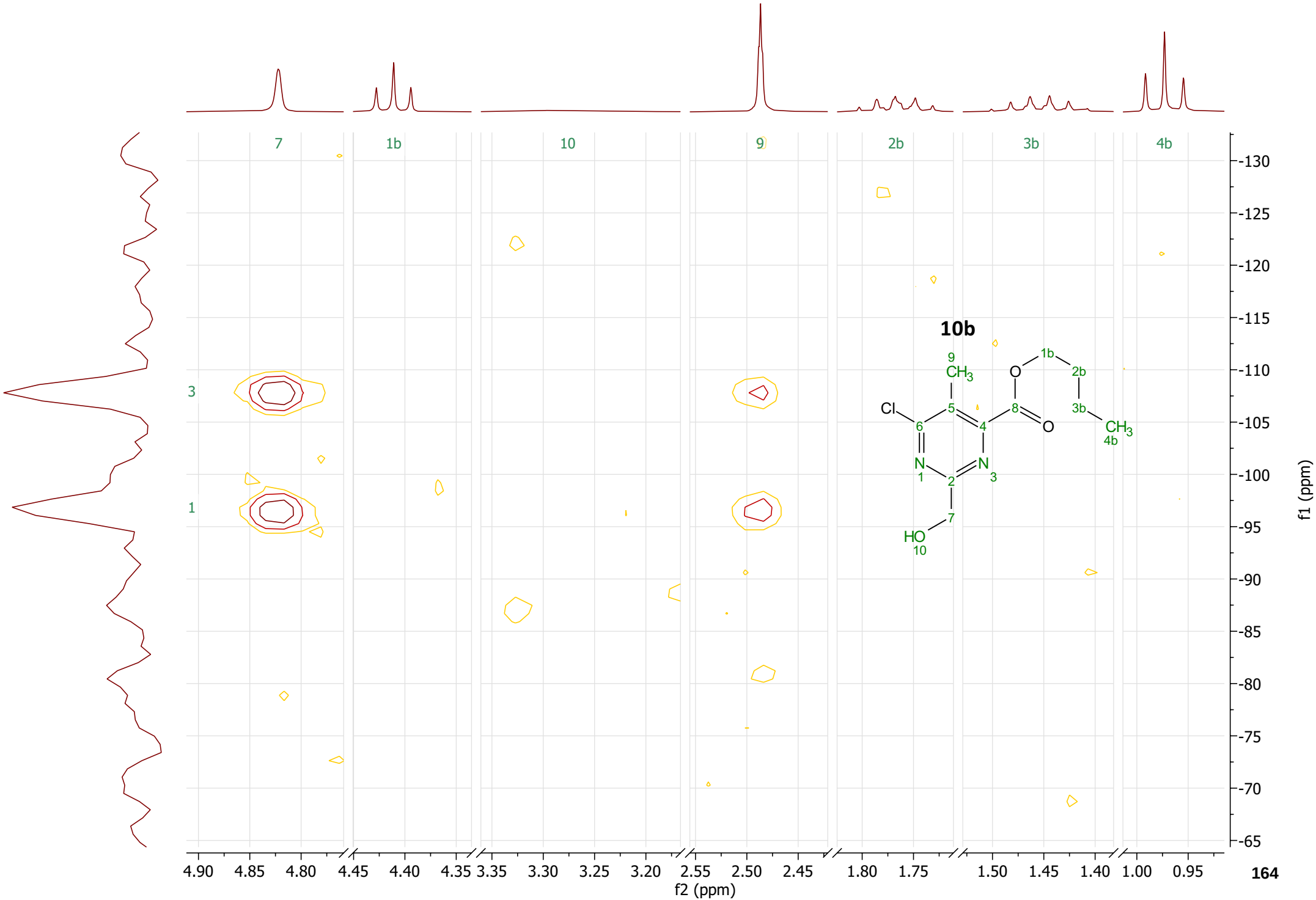
13C HSQC


7
1b
10
9
2b
3b
4b
4b
9
3b
2b
7
1b
10b
f2 (ppm)
f1 (ppm)
4.90 4.85 4.80 4.75 4.45 4.40 4.35 3.35 3.30 3.25 3.20 2.50 2.45 1.80 1.75 1.70 1.50 1.45 1.40 1.00 0.95 0.90
11 12 13 14 15 16 17 18 19 20 29 30 31 32 63 64 65 66 67 68

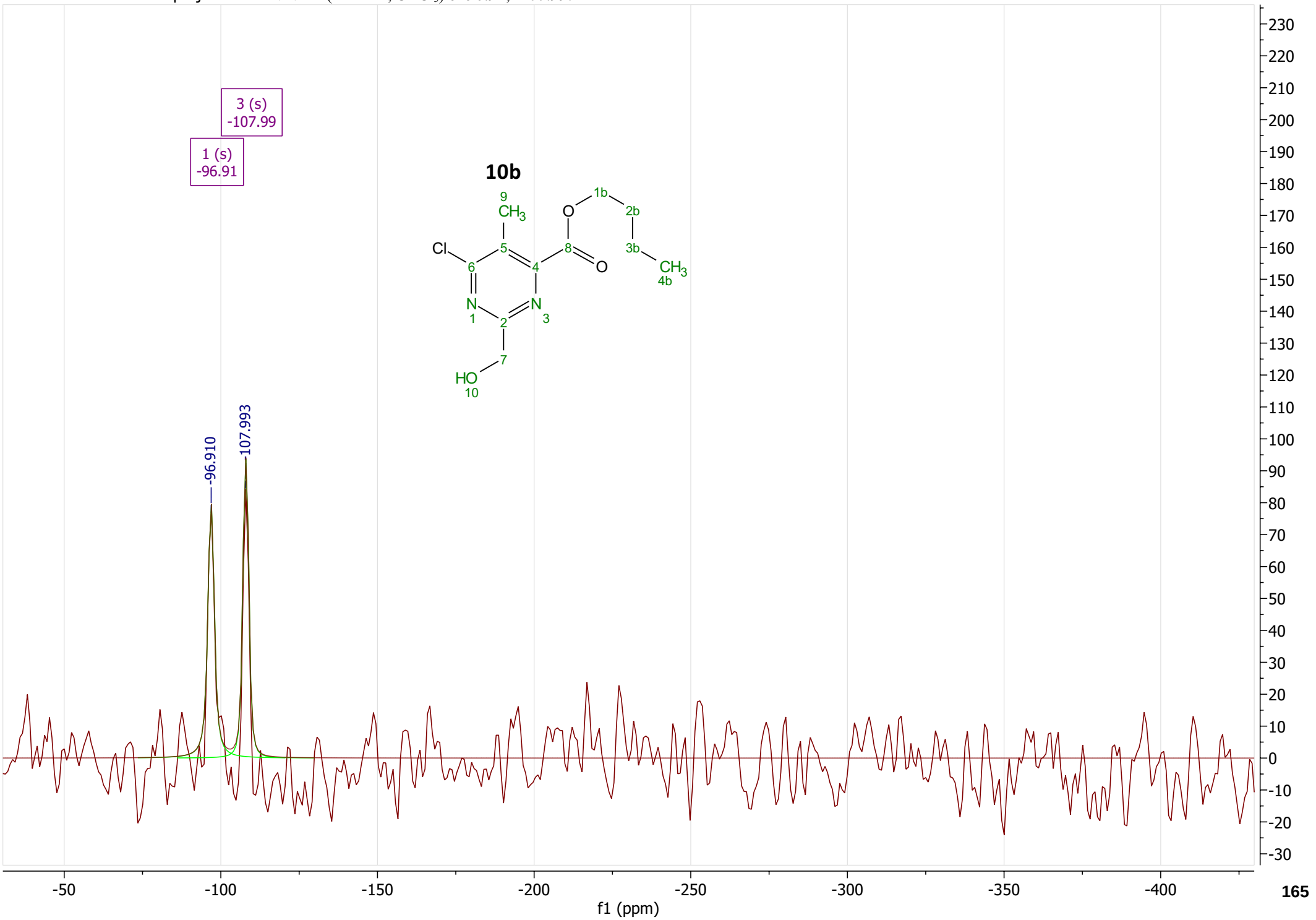
13C HMBC


7
1b
10
9
2b
3b
4b
9
4b
3b
2b
7
1b
5
4
8
6
2
10b
f1 (ppm)
f2 (ppm)
4.9
4.8
4.4
3.3
2.6
2.5
2.4
2.3
1.8
1.7
1.5
1.4
1.0
0.9
15
20
25
30
65
70
125
130
155
160
165
170
210

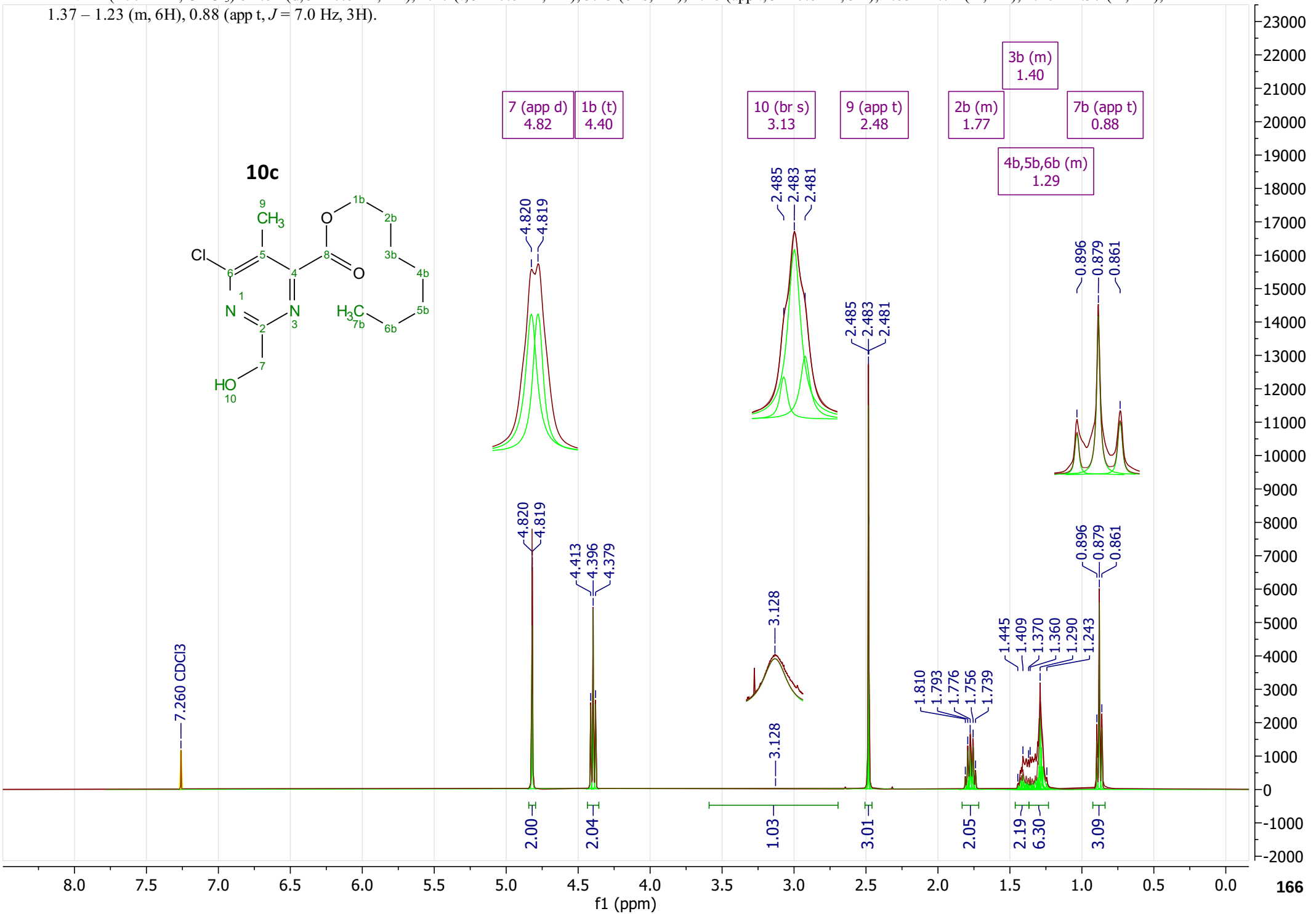
15N HMBC


7
1b
10
9
2b
3b
4b
3
1
10b
9
CH3
1b
O
2b
Cl
5
8
3b
CH3
6
4
O
4b
N
N
1
2
3
7
HO
10
-130
-125
-120
-115
-110
-105
-100
-95
-90
-85
-80
-75
-70
-65
f1 (ppm)
4.90
4.85
4.80
4.45
4.40
4.35
3.35
3.30
3.25
3.20
2.55
2.50
2.45
1.80
1.75
1.50
1.45
1.40
1.00
0.95
f2 (ppm)

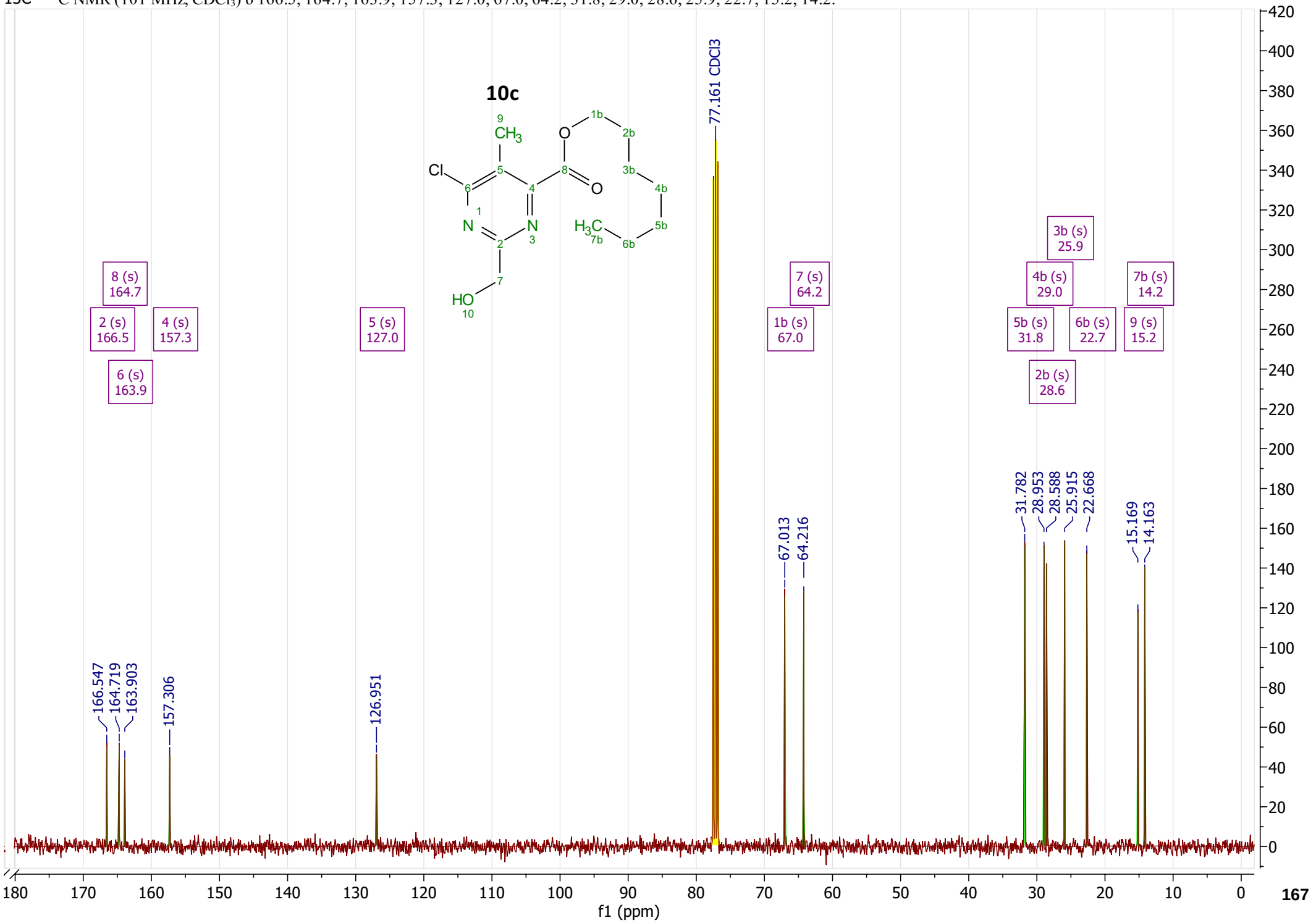
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -96.91, -107.99.



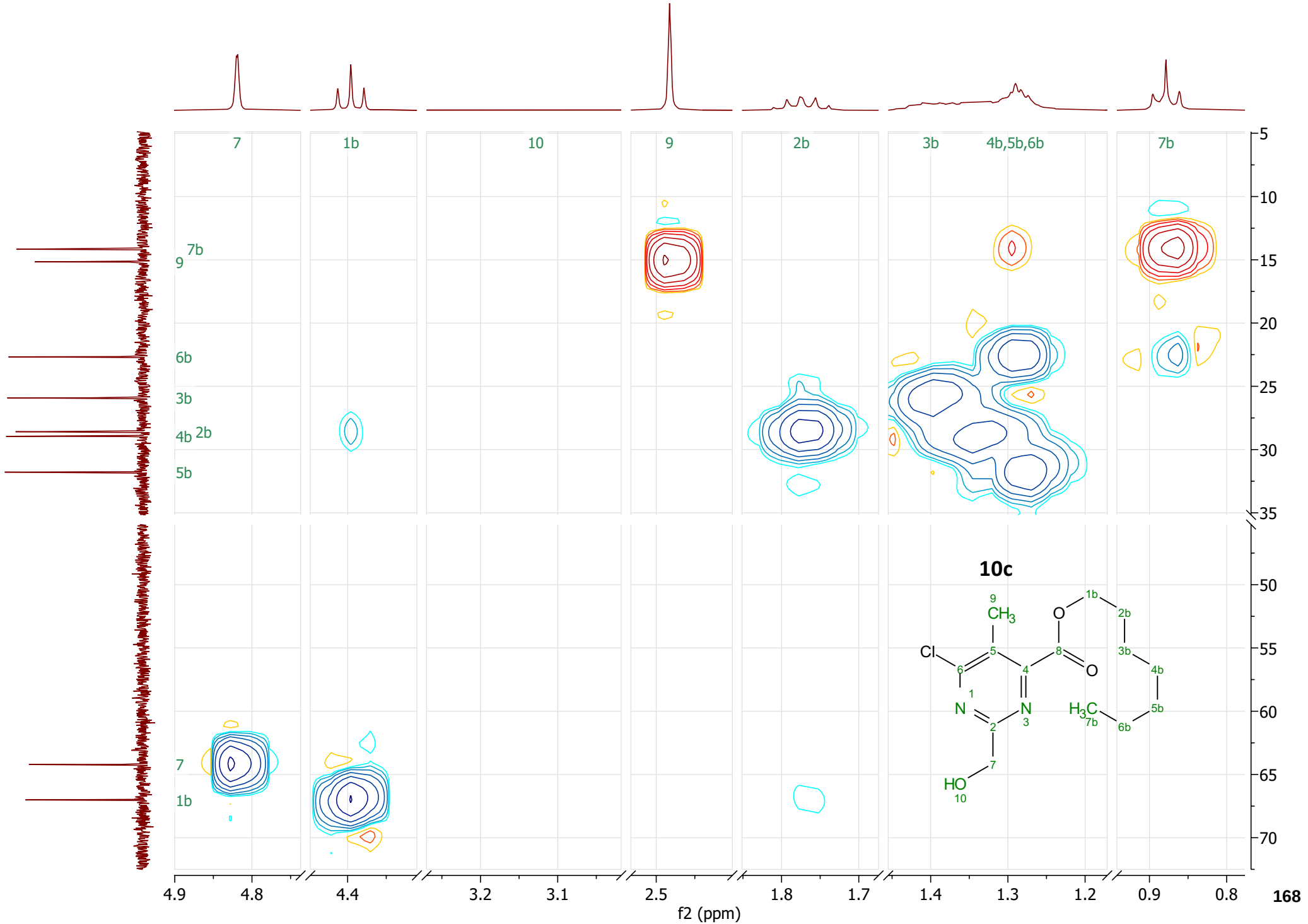
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (d, *J* = 0.8 Hz, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.13 (br s, 1H), 2.48 (app t, *J* = 0.8 Hz, 3H), 1.83 – 1.72 (m, 2H), 1.46 – 1.37 (m, 2H), 1.37 – 1.23 (m, 6H), 0.88 (app t, *J* = 7.0 Hz, 3H).



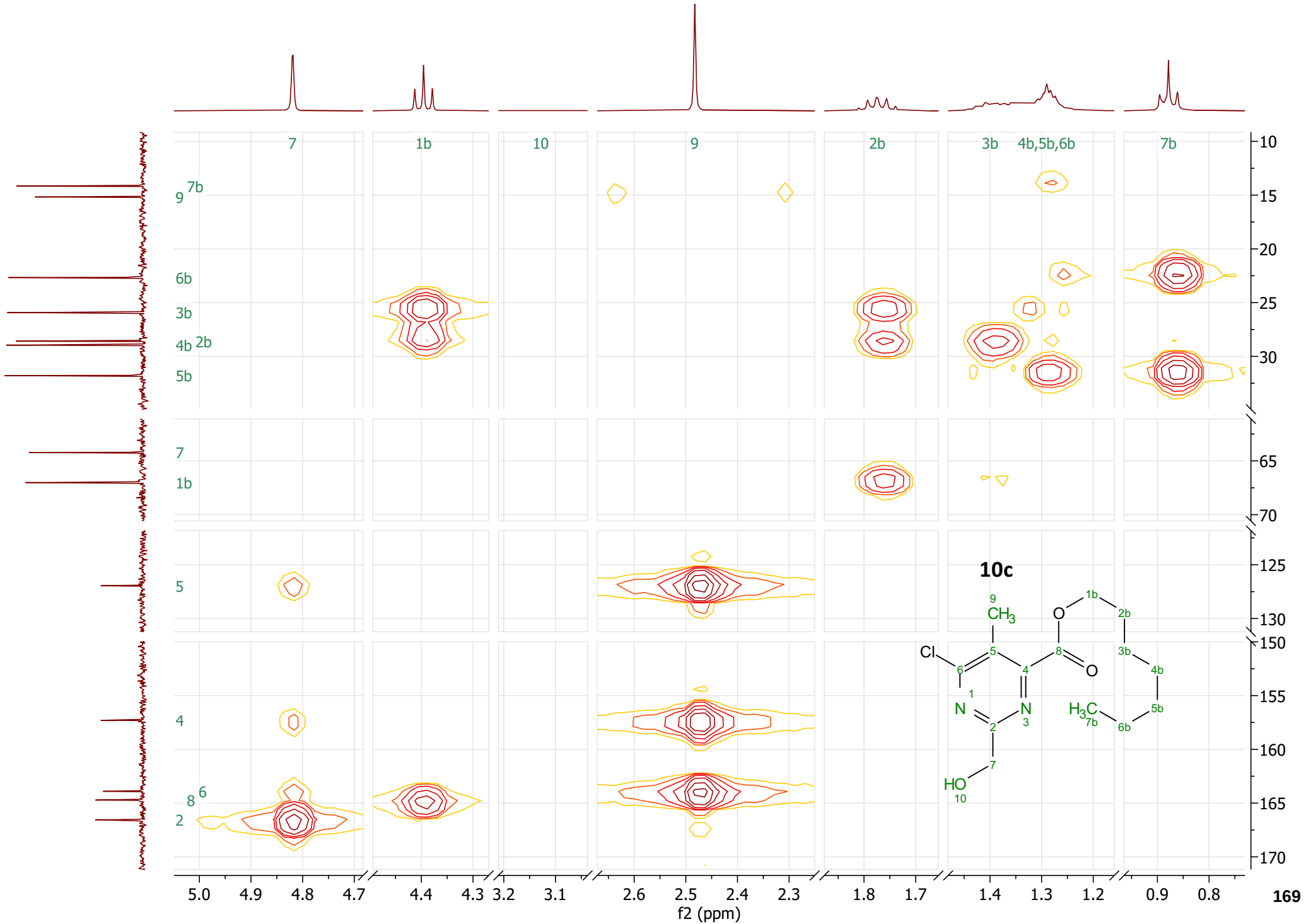
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.7, 163.9, 157.3, 127.0, 67.0, 64.2, 31.8, 29.0, 28.6, 25.9, 22.7, 15.2, 14.2.



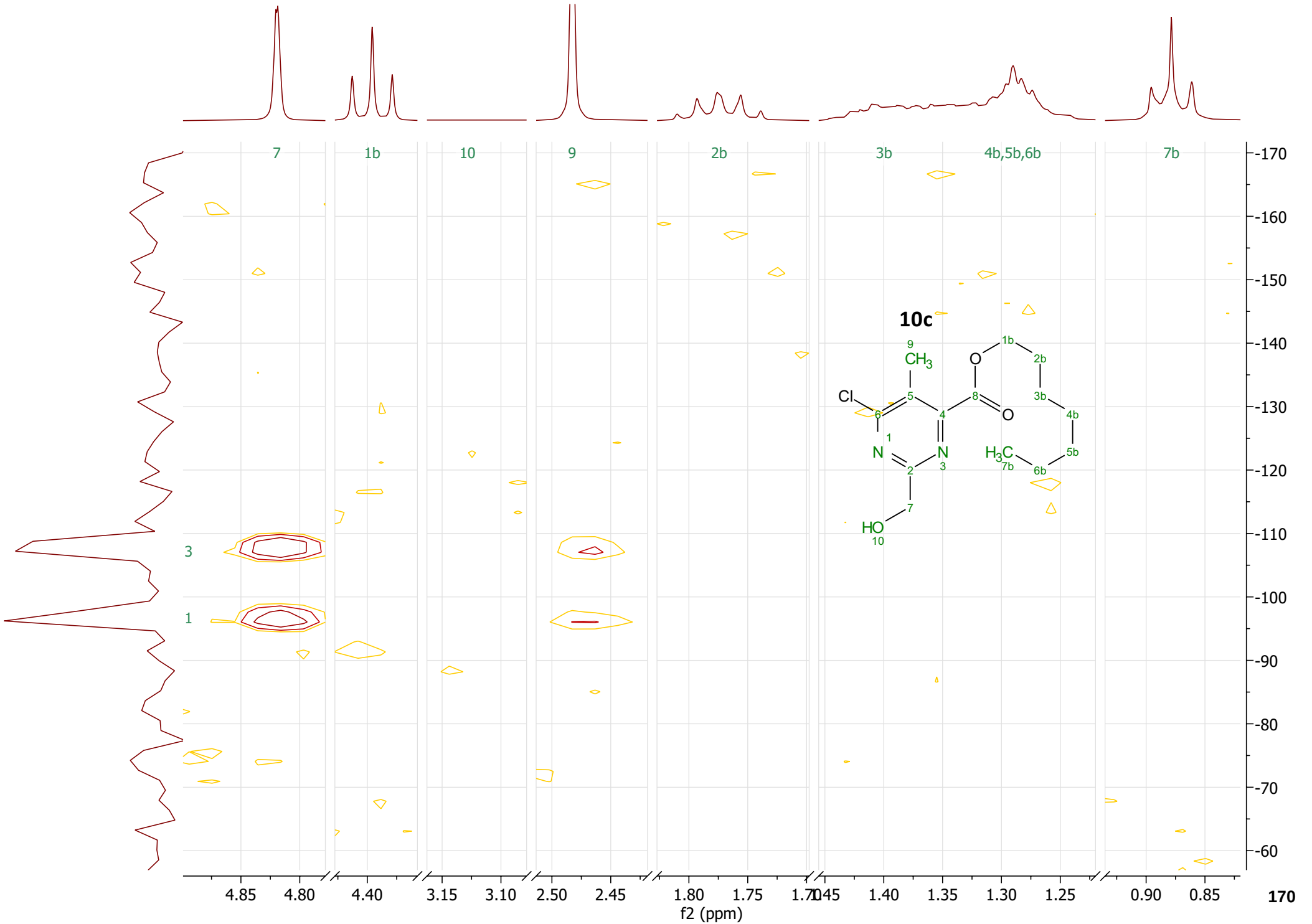
13C HSQC


7
1b
10
9
2b
3b
4b,5b,6b
7b
9
7b
6b
3b
4b
2b
5b
7
1b
10c
9
CH3
O
1b
2b
Cl
5
8
3b
4b
6
4
O
1
N
N
H3C
5b
2
3
7b
6b
7
HO
10
f1 (ppm)
f2 (ppm)
4.9
4.8
4.4
3.2
3.1
2.5
1.8
1.7
1.4
1.3
1.2
0.9
0.8
5
10
15
20
25
30
35
50
55
60
65
70

13C HMBC

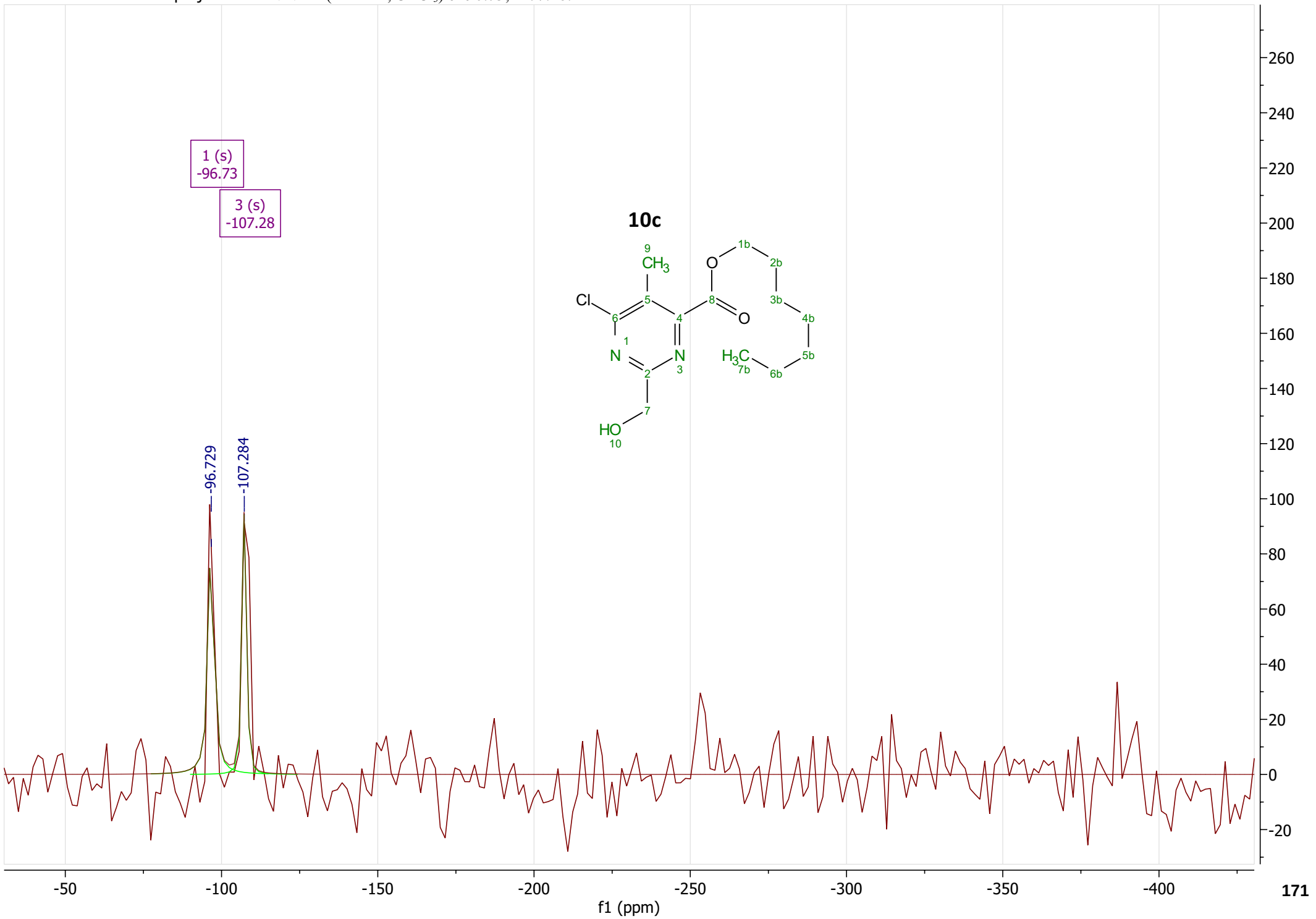

7
1b
10
9
2b
3b
4b,5b,6b
7b
9
7b
6b
3b
4b
2b
5b
7
1b
5
4
8
6
2
10c
f2 (ppm)
f1 (ppm)
169

15N HMBC

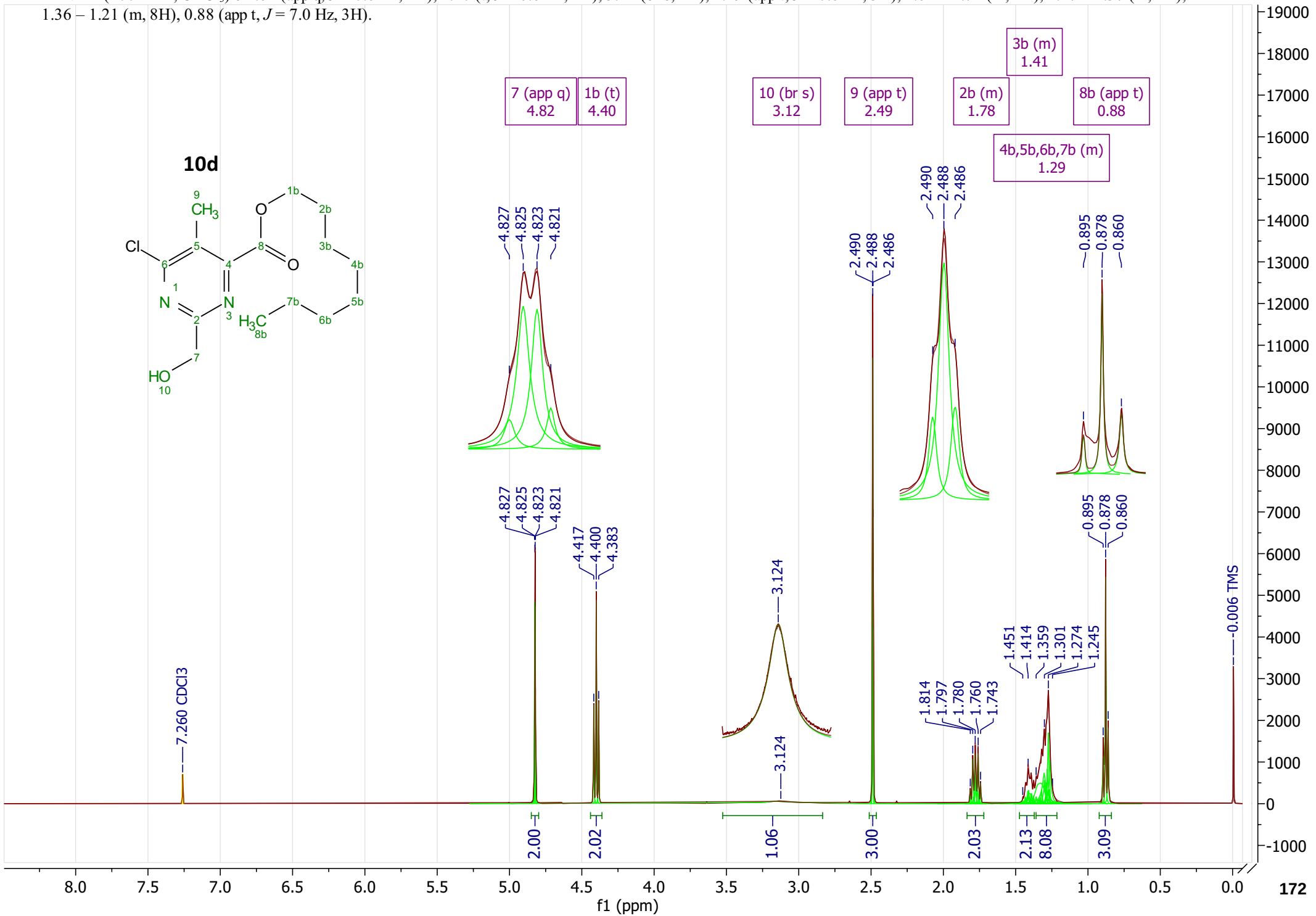

7
1b
10
9
2b
3b
4b,5b,6b
7b
3
1
10c
f2 (ppm)
f1 (ppm)
4.85
4.80
4.40
3.15
3.10
2.50
2.45
1.80
1.75
1.70
1.45
1.40
1.35
1.30
1.25
0.90
0.85
-170
-160
-150
-140
-130
-120
-110
-100
-90
-80
-70
-60

170

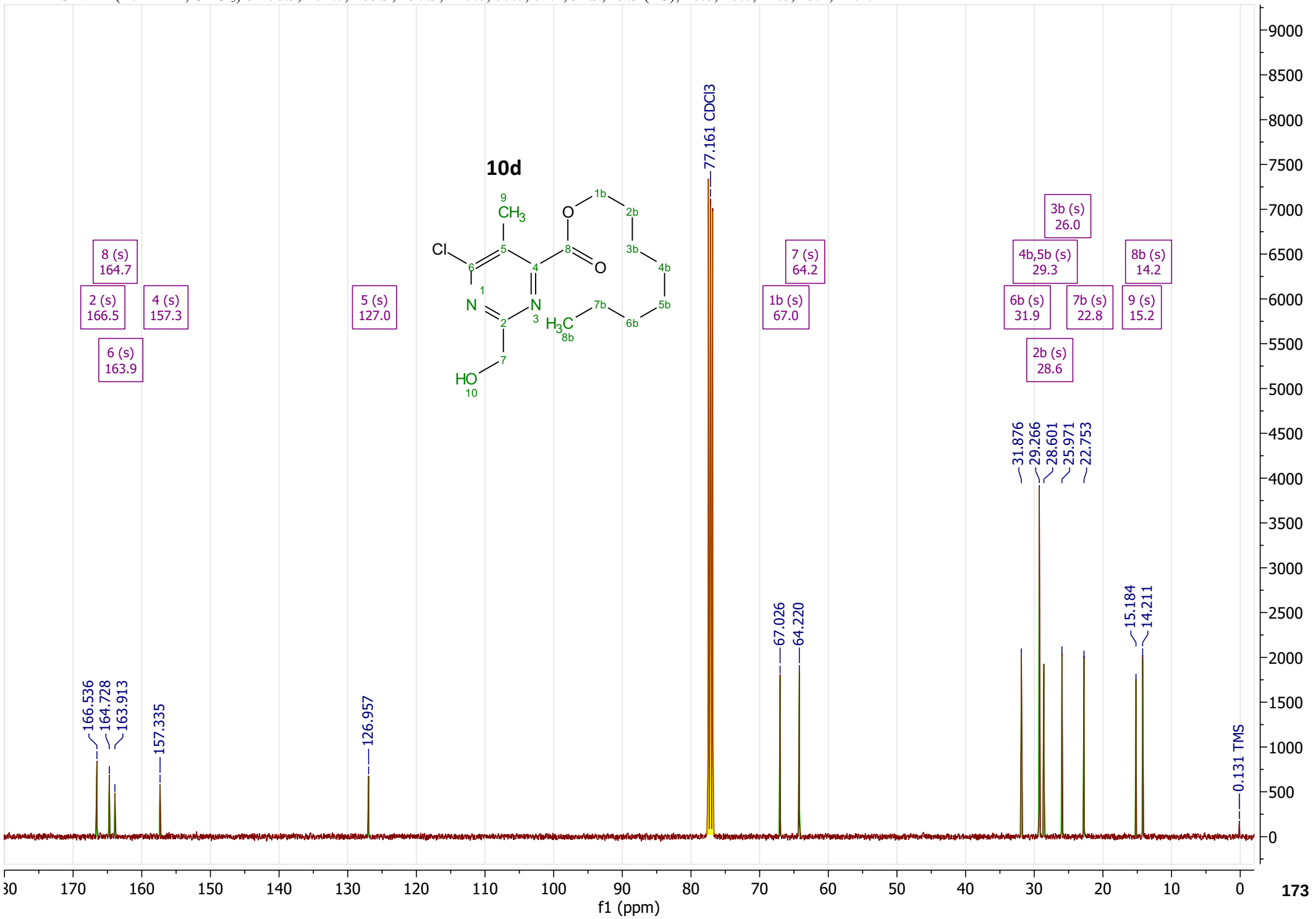
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -96.73, -107.28.



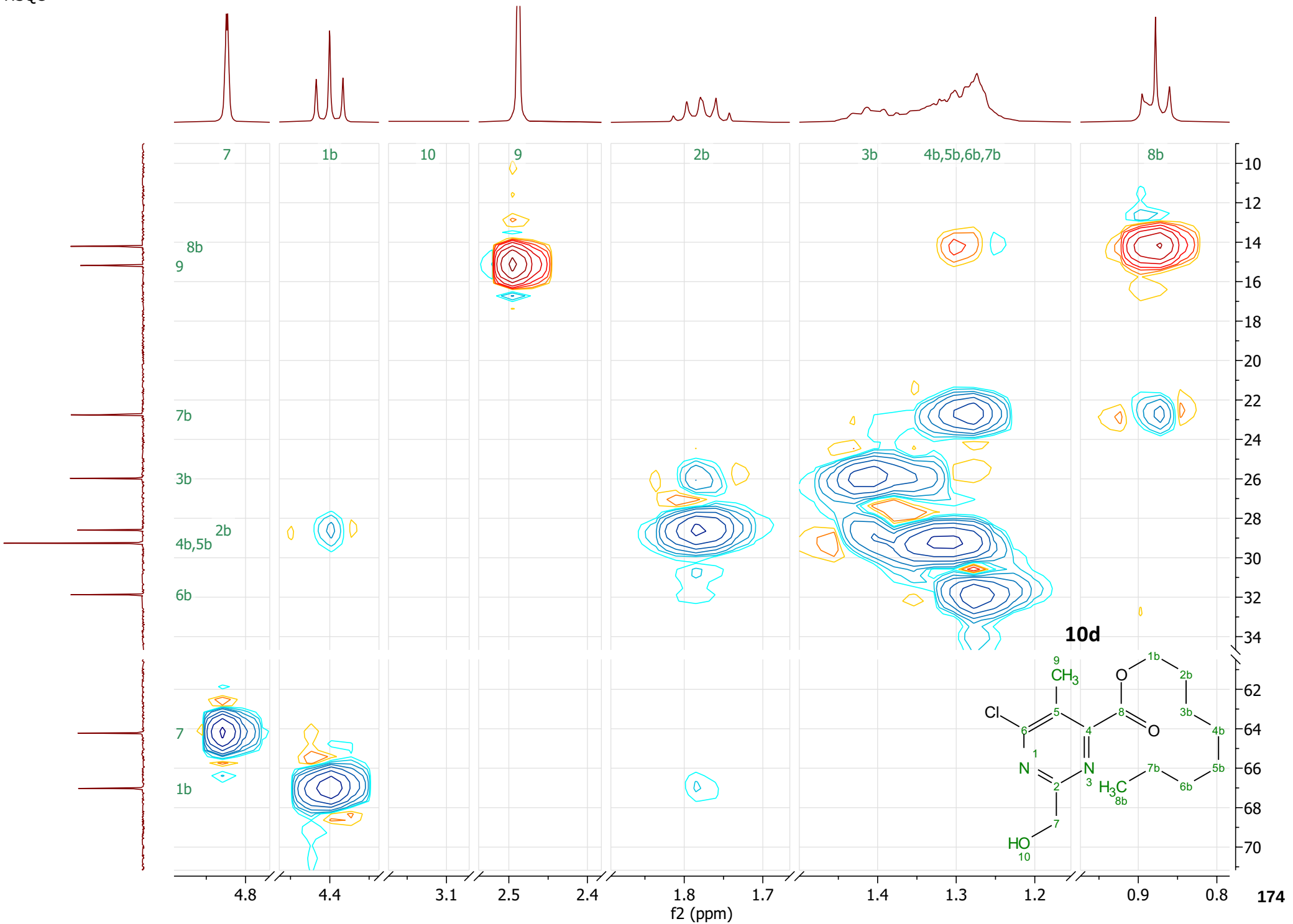
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 (app q, *J* = 0.8 Hz, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.12 (br s, 1H), 2.49 (app t, *J* = 0.8 Hz, 3H), 1.84 – 1.72 (m, 2H), 1.47 – 1.37 (m, 2H), 1.36 – 1.21 (m, 8H), 0.88 (app t, *J* = 7.0 Hz, 3H).



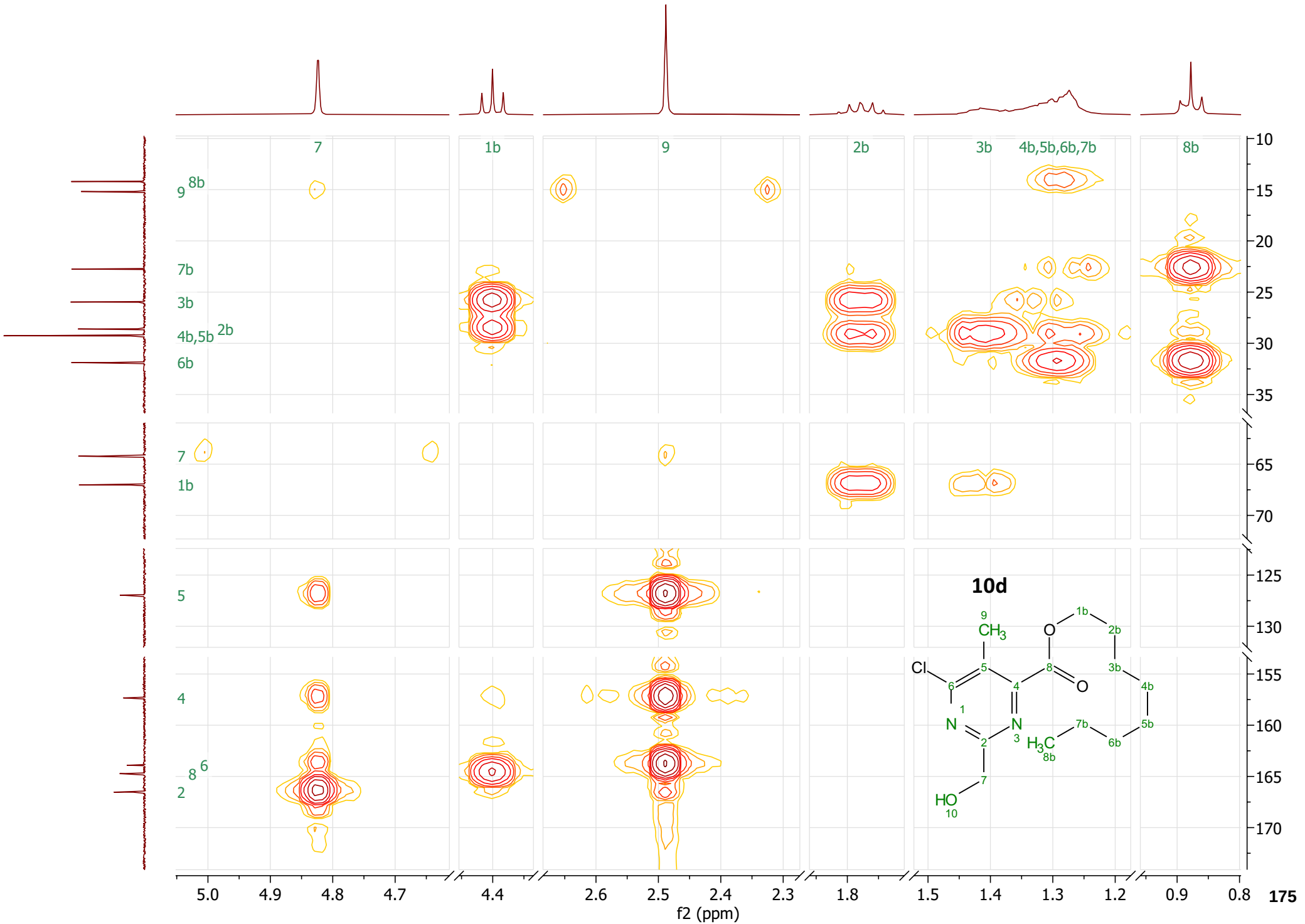
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.7, 163.9, 157.3, 127.0, 67.0, 64.2, 31.9, 29.3 (2C), 28.6, 26.0, 22.8, 15.2, 14.2.



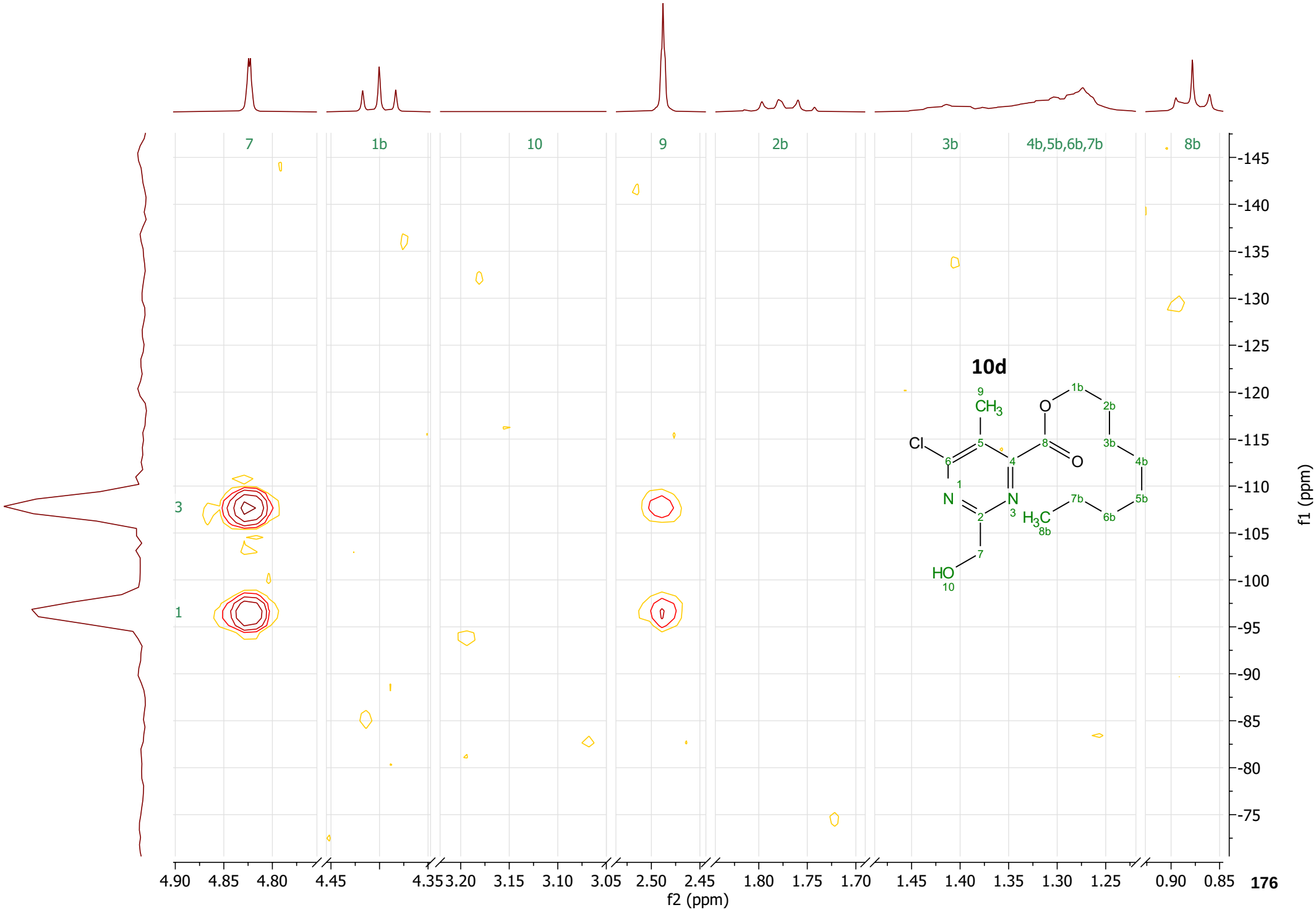
13C HSQC


7
1b
10
9
2b
3b
4b,5b,6b,7b
8b
8b
9
7b
3b
2b
4b,5b
6b
7
1b
4.8
4.4
3.1
2.5
2.4
1.8
1.7
1.4
1.3
1.2
0.9
0.8
f2 (ppm)
f1 (ppm)
10d
CH3
Cl
O
O
N
N
H3C
HO

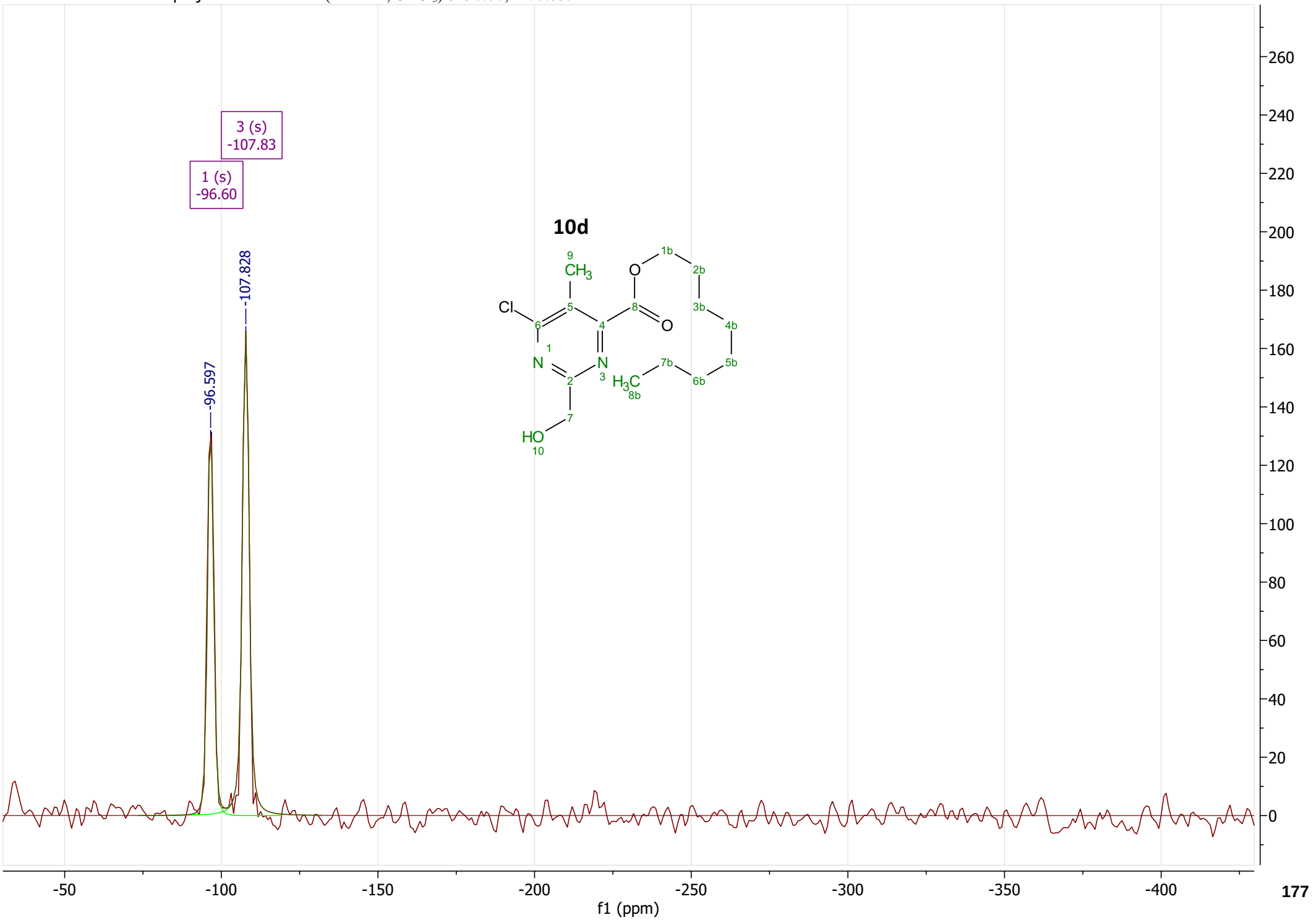
13C HMBC


7
1b
9
2b
3b
4b,5b,6b,7b
8b
9 8b
7b
3b
4b,5b 2b
6b
7
1b
5
4
2 8 6
10d
f2 (ppm)
f1 (ppm)

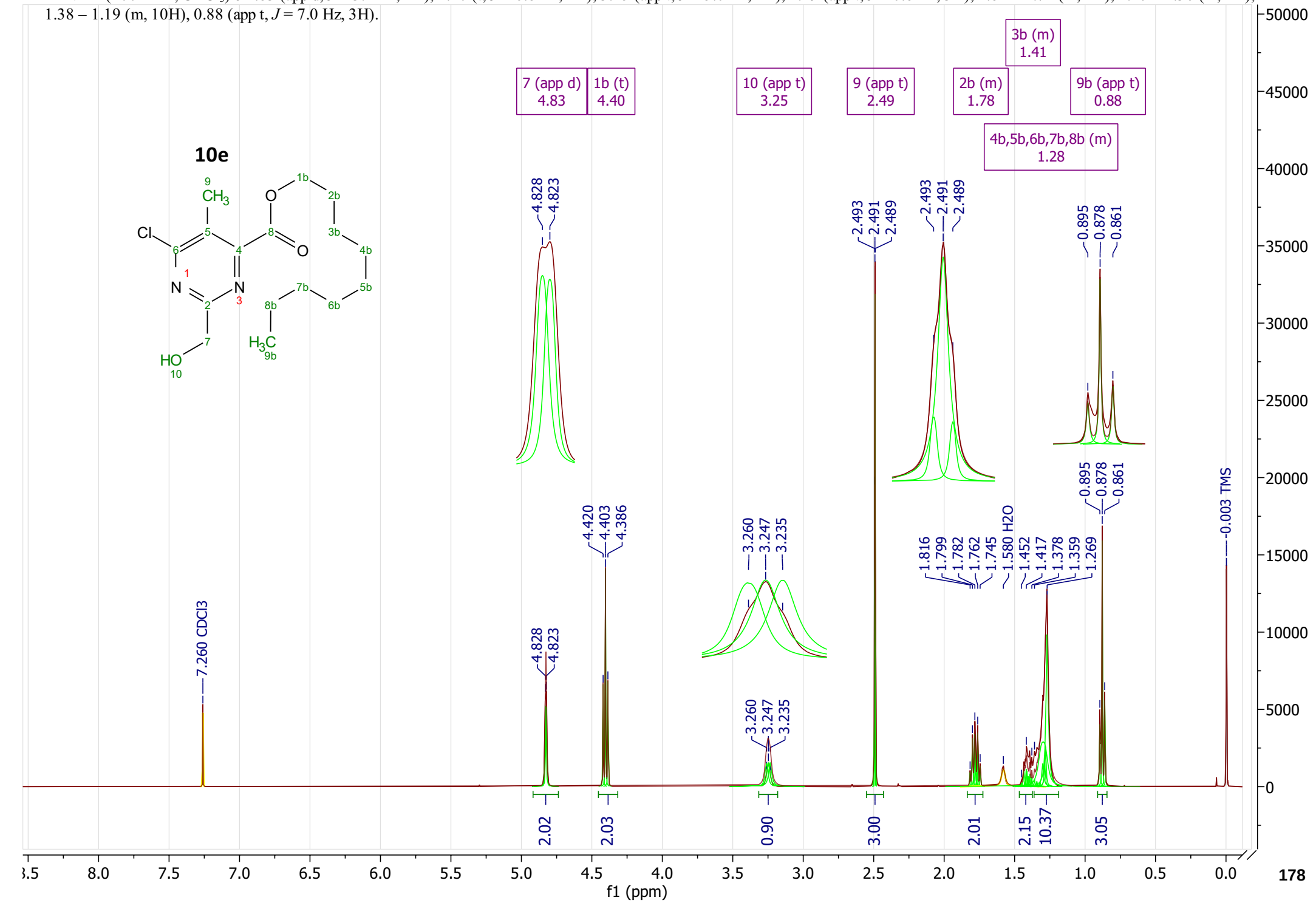
15N HMBC


7
1b
10
9
2b
3b
4b,5b,6b,7b
8b
3
1
10d
f2 (ppm)
f1 (ppm)
4.90
4.85
4.80
4.45
4.35
3.20
3.15
3.10
3.05
2.50
2.45
1.80
1.75
1.70
1.45
1.40
1.35
1.30
1.25
0.90
0.85
-145
-140
-135
-130
-125
-120
-115
-110
-105
-100
-95
-90
-85
-80
-75

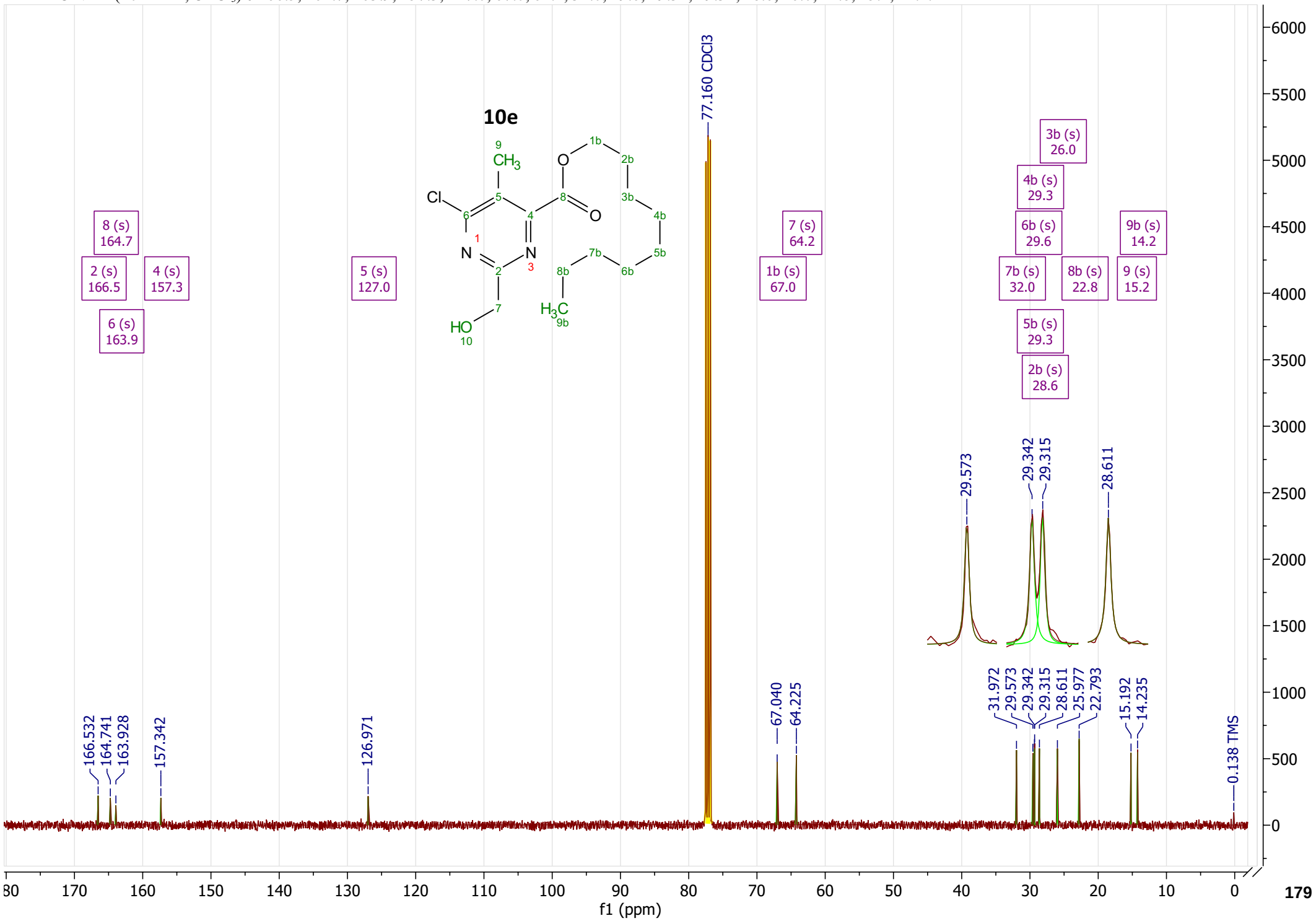
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -96.60, -107.83.



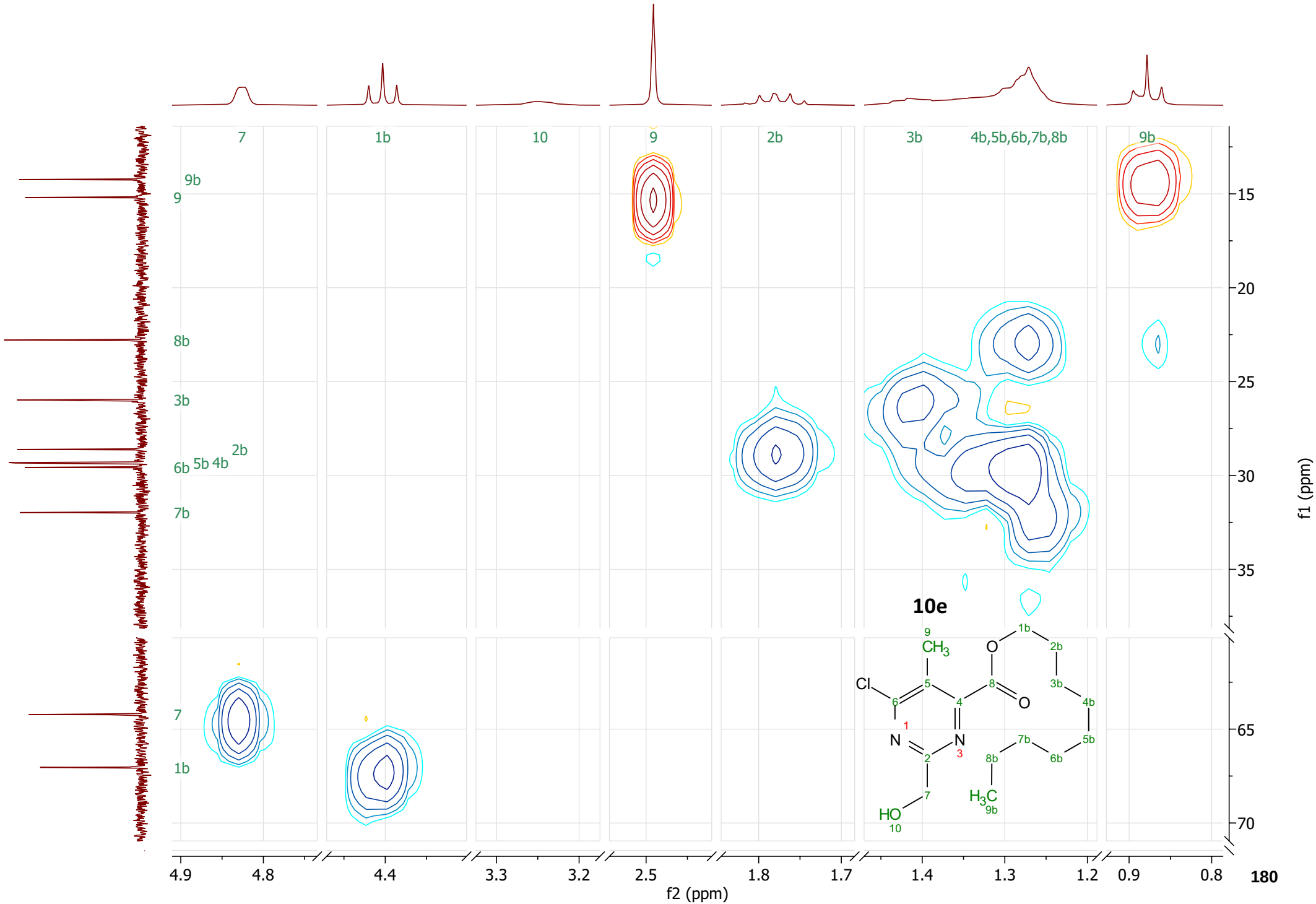
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.83 (app d, *J* = 3.4 Hz, 2H), 4.40 (t, *J* = 6.8 Hz, 2H), 3.25 (app t, *J* = 5.0 Hz, 1H), 2.49 (app t, *J* = 0.8 Hz, 3H), 1.84 – 1.72 (m, 2H), 1.47 – 1.36 (m, 2H), 1.38 – 1.19 (m, 10H), 0.88 (app t, *J* = 7.0 Hz, 3H).



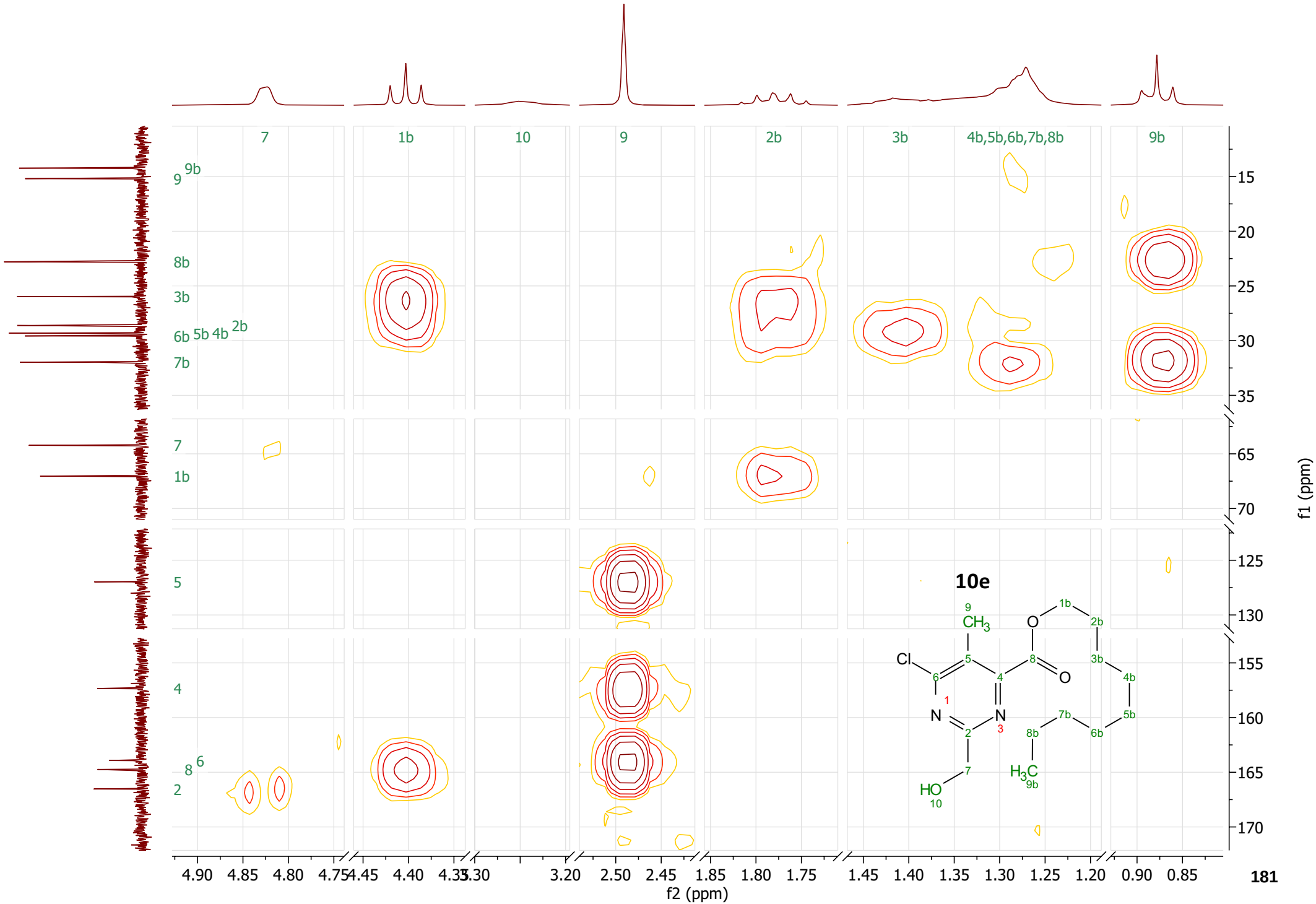
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 164.7, 163.9, 157.3, 127.0, 67.0, 64.2, 32.0, 29.6, 29.34, 29.32, 28.6, 26.0, 22.8, 15.2, 14.2.



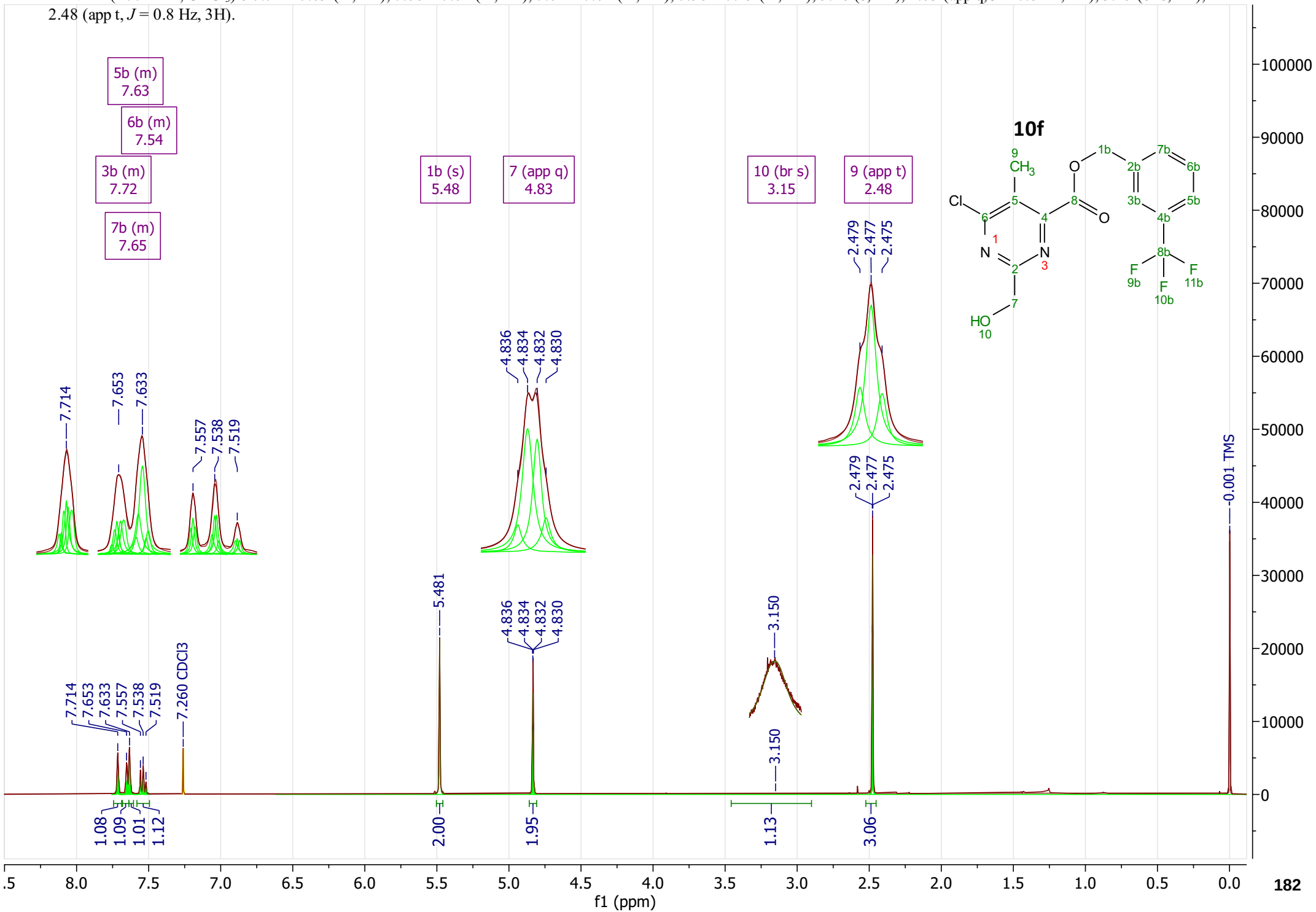
13C HSQC


7
1b
10
9
2b
3b
4b,5b,6b,7b,8b
9b
9b
9
8b
3b
6b 5b 4b 2b
7b
7
1b
15
20
25
30
35
65
70
f1 (ppm)
4.9
4.8
4.4
3.3
3.2
2.5
1.8
1.7
1.4
1.3
1.2
0.9
0.8
180
f2 (ppm)
10e
CH3
Cl
O
O
N
N
HO
H3C

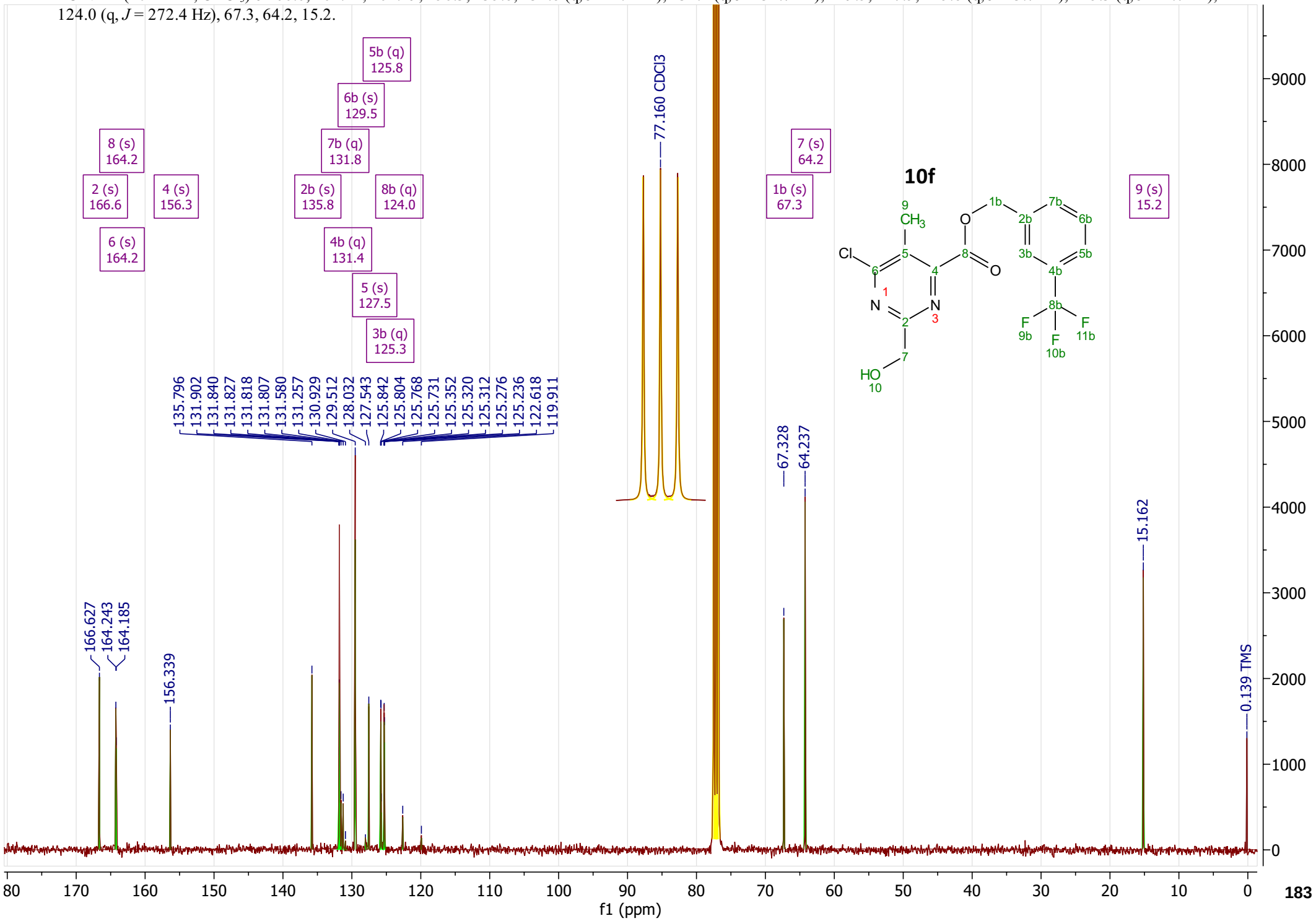
13C HMBC


7
1b
10
9
2b
3b
4b,5b,6b,7b,8b
9b
9 9b
8b
3b
6b 5b 4b 2b
7b
7
1b
5
4
8 6
2
10e
9
CH3
1b
O
2b
Cl
5
8
3b
4b
6
4
O
1
N
N
2
3
7b
5b
8b
6b
7
H3C
9b
HO
10
15
20
25
30
35
65
70
125
130
155
160
165
170
4.90 4.85 4.80 4.75 4.45 4.40 4.35 3.30 3.20 2.50 2.45 1.85 1.80 1.75 1.45 1.40 1.35 1.30 1.25 1.20 0.90 0.85
f2 (ppm)
f1 (ppm)

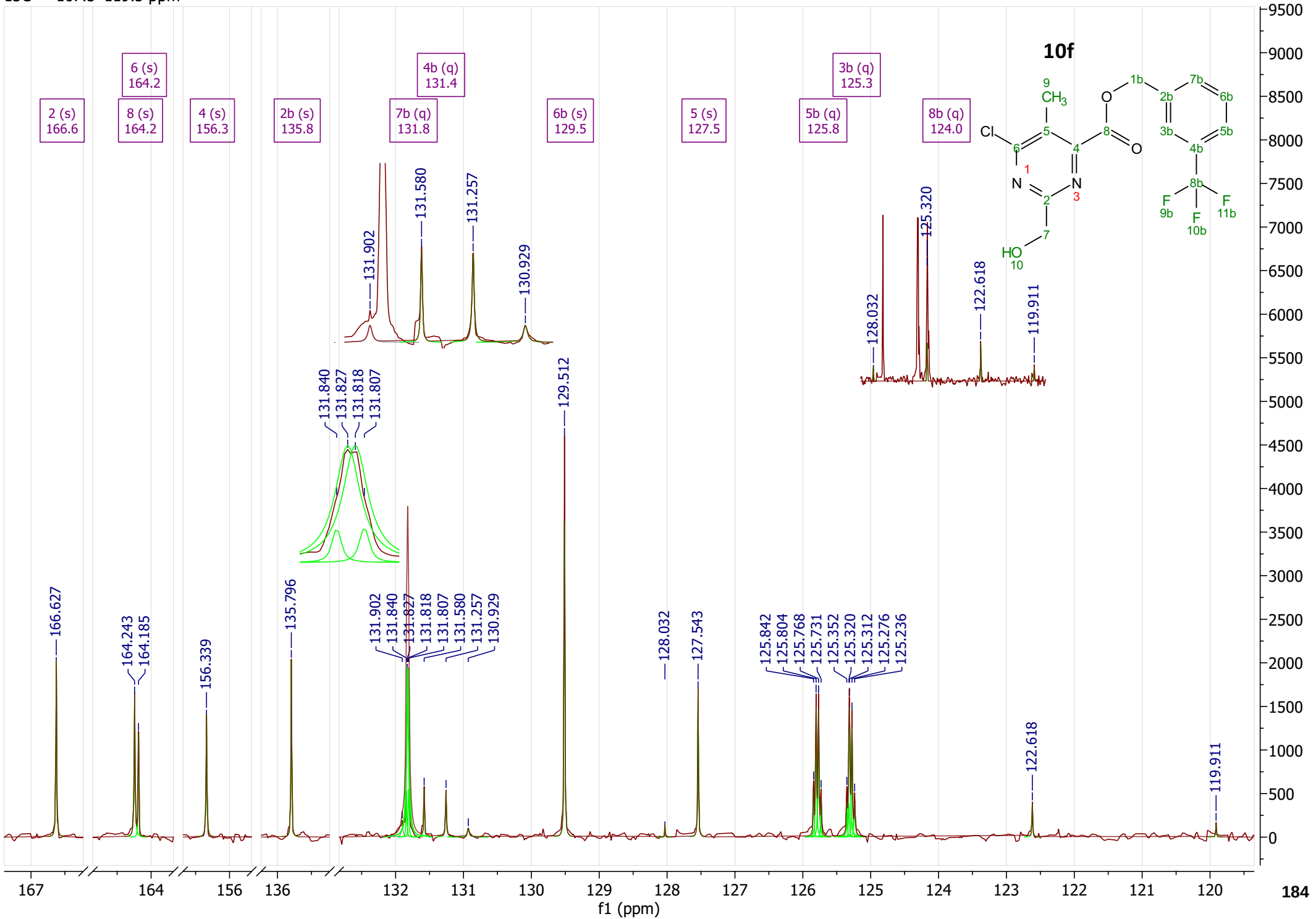
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 – 7.69 (m, 1H), 7.68 – 7.64 (m, 1H), 7.64 – 7.61 (m, 1H), 7.58 – 7.49 (m, 1H), 5.48 (s, 2H), 4.83 (app q, *J* = 0.8 Hz, 2H), 3.15 (br s, 1H), 2.48 (app t, *J* = 0.8 Hz, 3H).



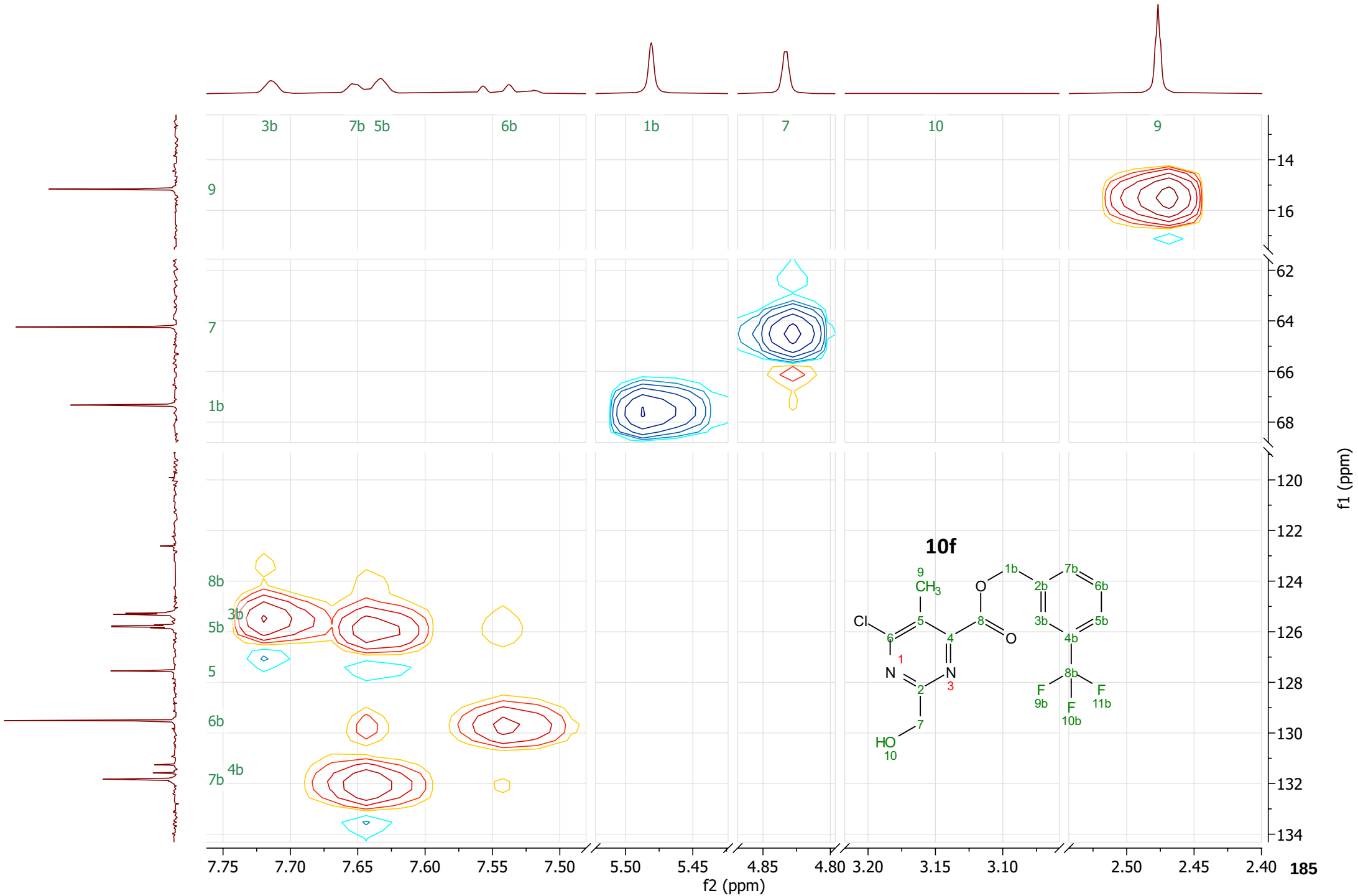
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.6, 164.24, 164.19, 156.3, 135.8, 131.8 (q, J = 1.1 Hz), 131.4 (q, J = 32.7 Hz), 129.5, 127.5, 125.8 (q, J = 3.7 Hz), 125.3 (q, J = 4.0 Hz), 124.0 (q, J = 272.4 Hz), 67.3, 64.2, 15.2.



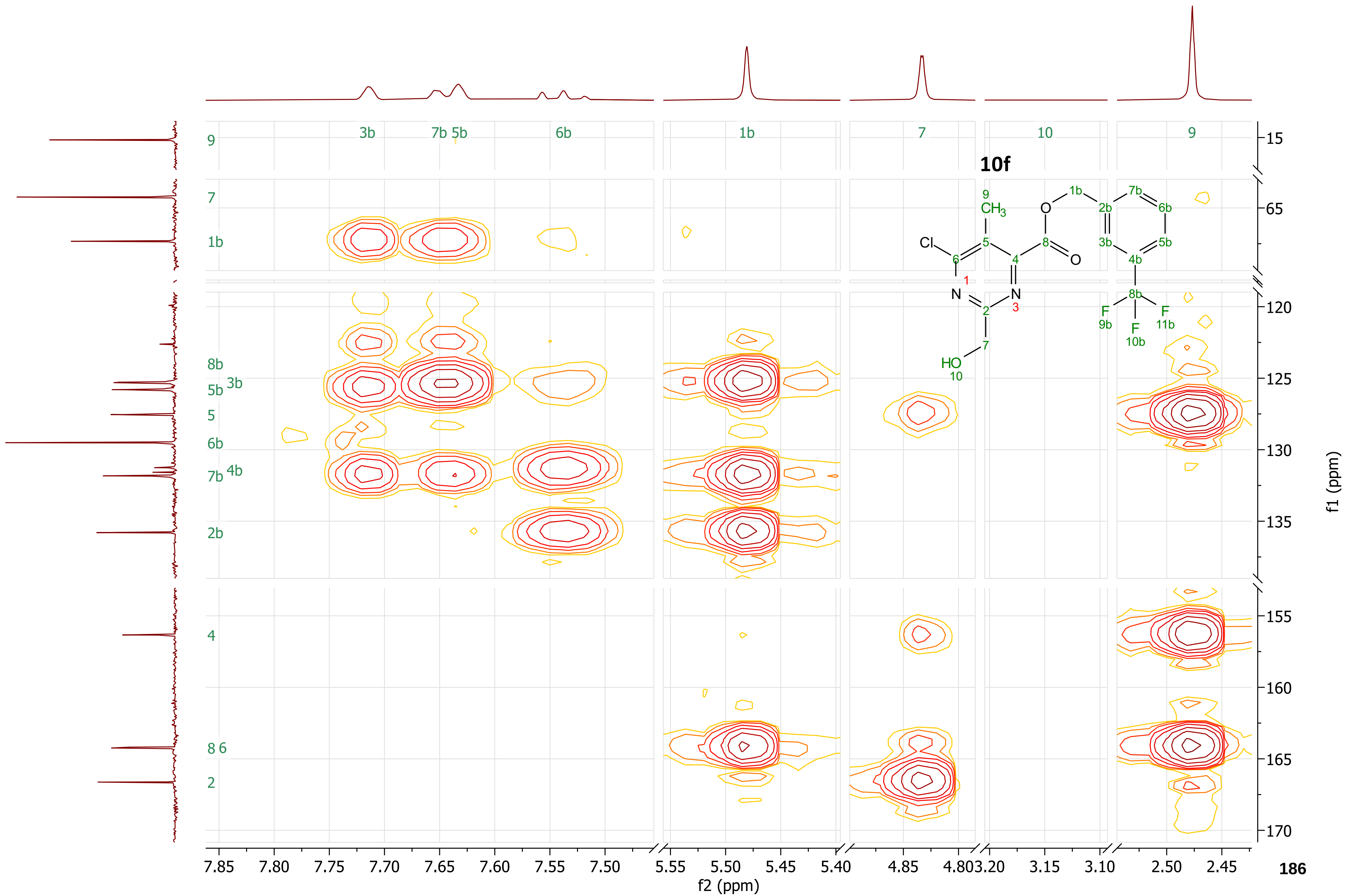
13C — 167.5–119.5 ppm



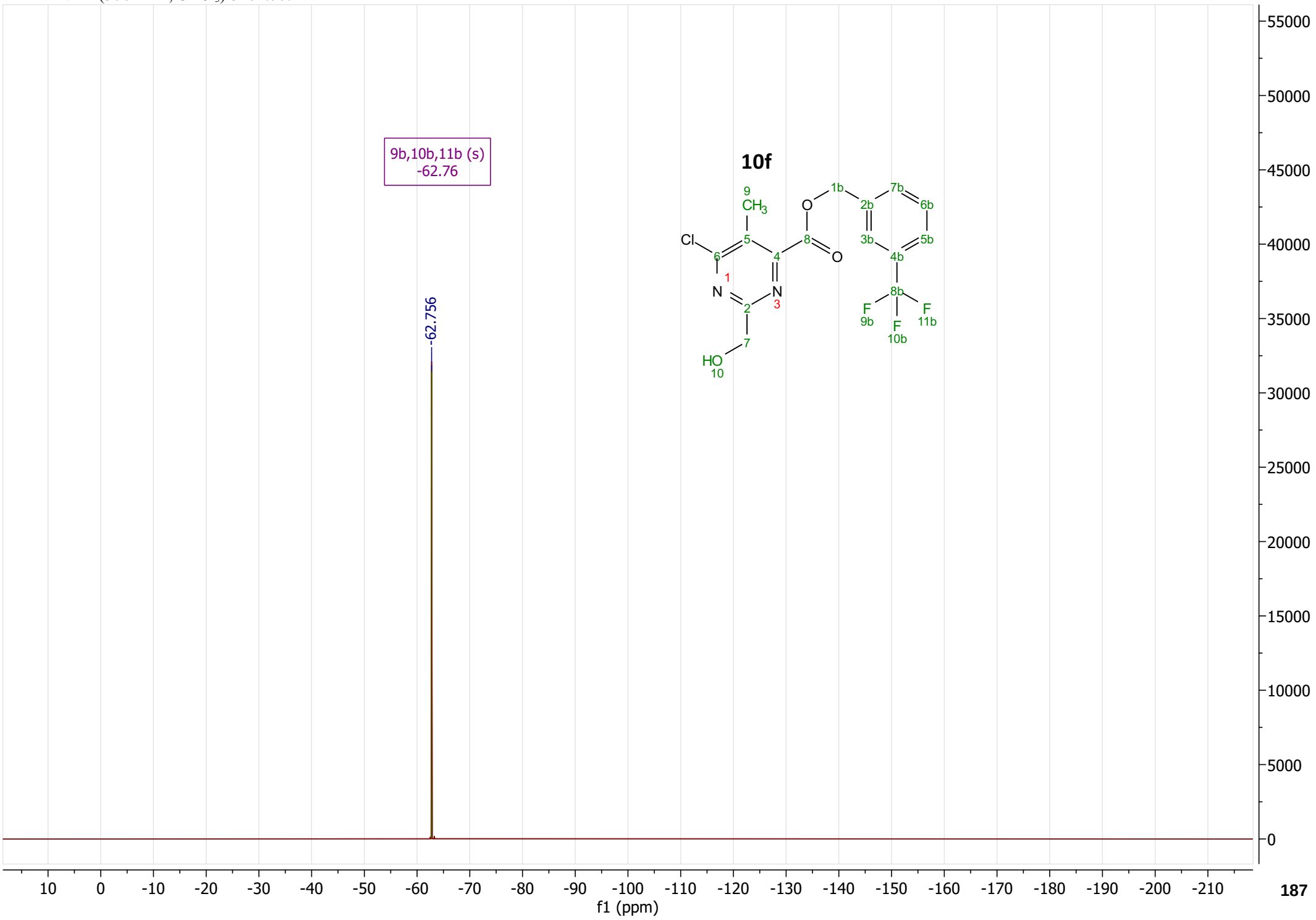
13C HSQC


3b
7b 5b
6b
1b
7
10
9
9
7
1b
8b
3b
5b
5
6b
7b
4b
10f
f2 (ppm)
f1 (ppm)
7.75
7.70
7.65
7.60
7.55
7.50
5.50
5.45
4.85
4.80
3.20
3.15
3.10
2.50
2.45
2.40
14
16
62
64
66
68
120
122
124
126
128
130
132
134

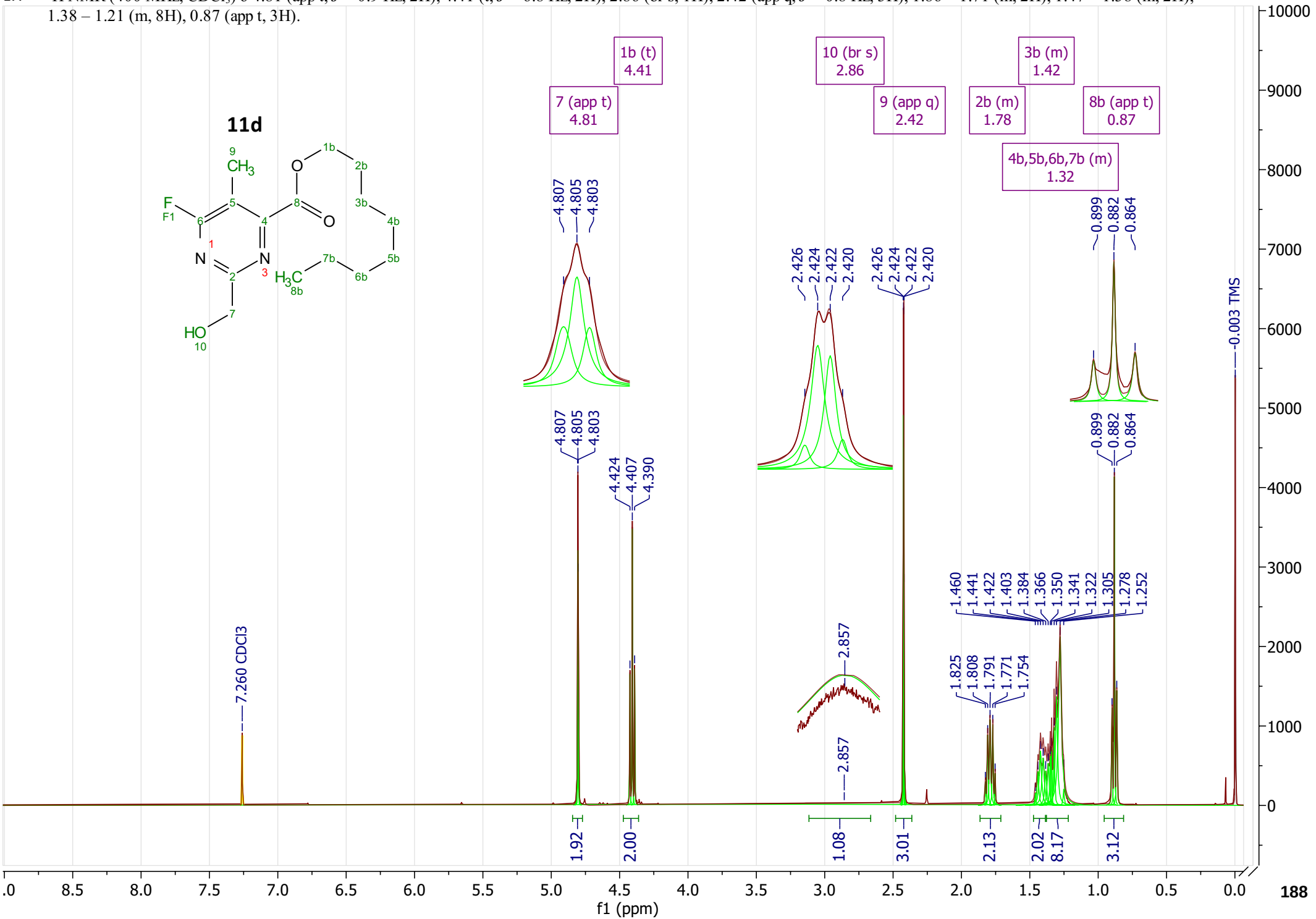
13C HMBC


10f
9
CH3
Cl
O
1b
2b
7b
6b
5b
3b
4b
8b
F
F
F
9b
10b
11b
HO
10
N
N
1
2
3
4
5
6
7
8
O
3b
7b 5b
6b
1b
7
10
9
8b
5b 3b
5
6b
7b 4b
2b
4
8 6
2
f2 (ppm)
f1 (ppm)

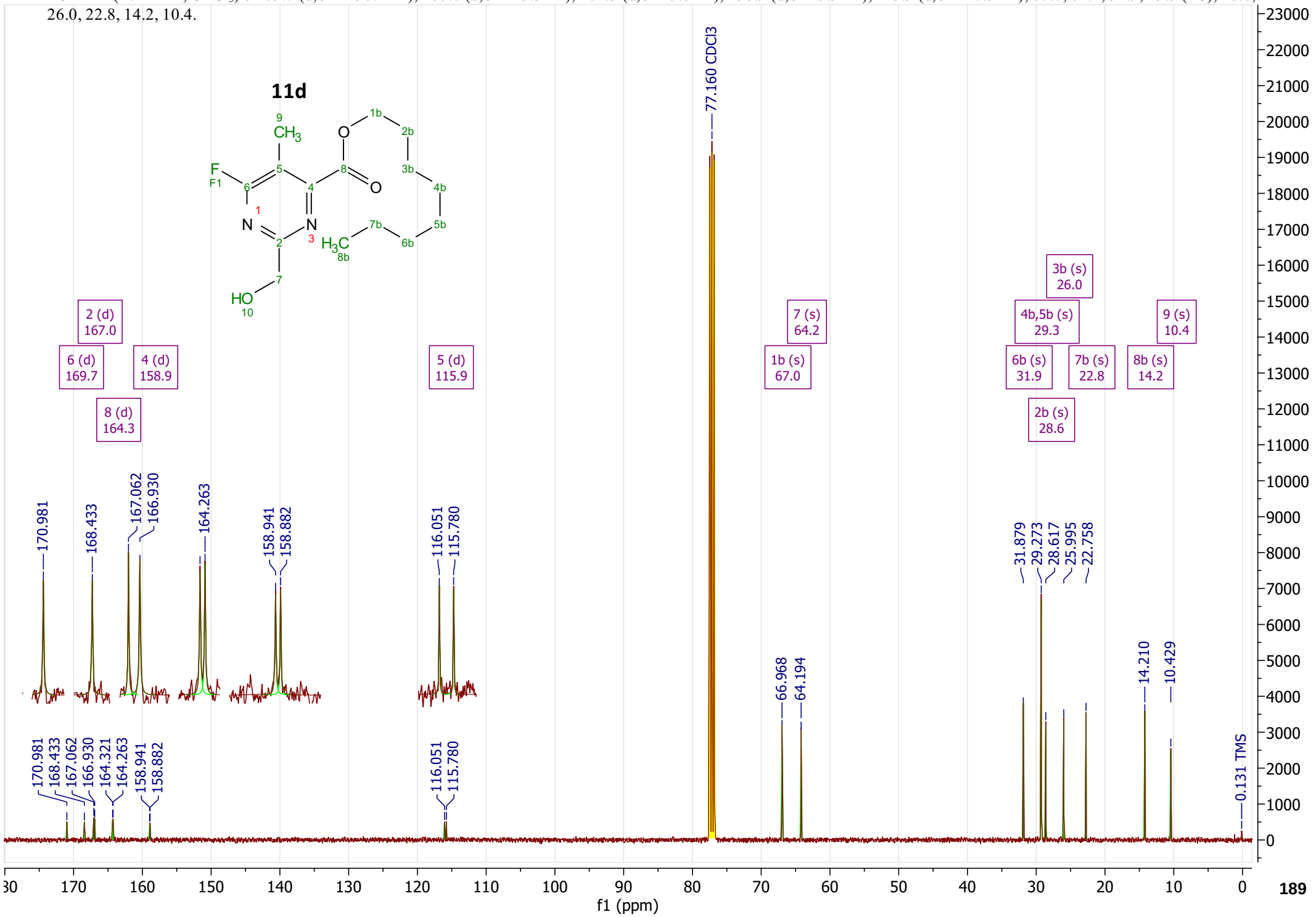
19F ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.76.



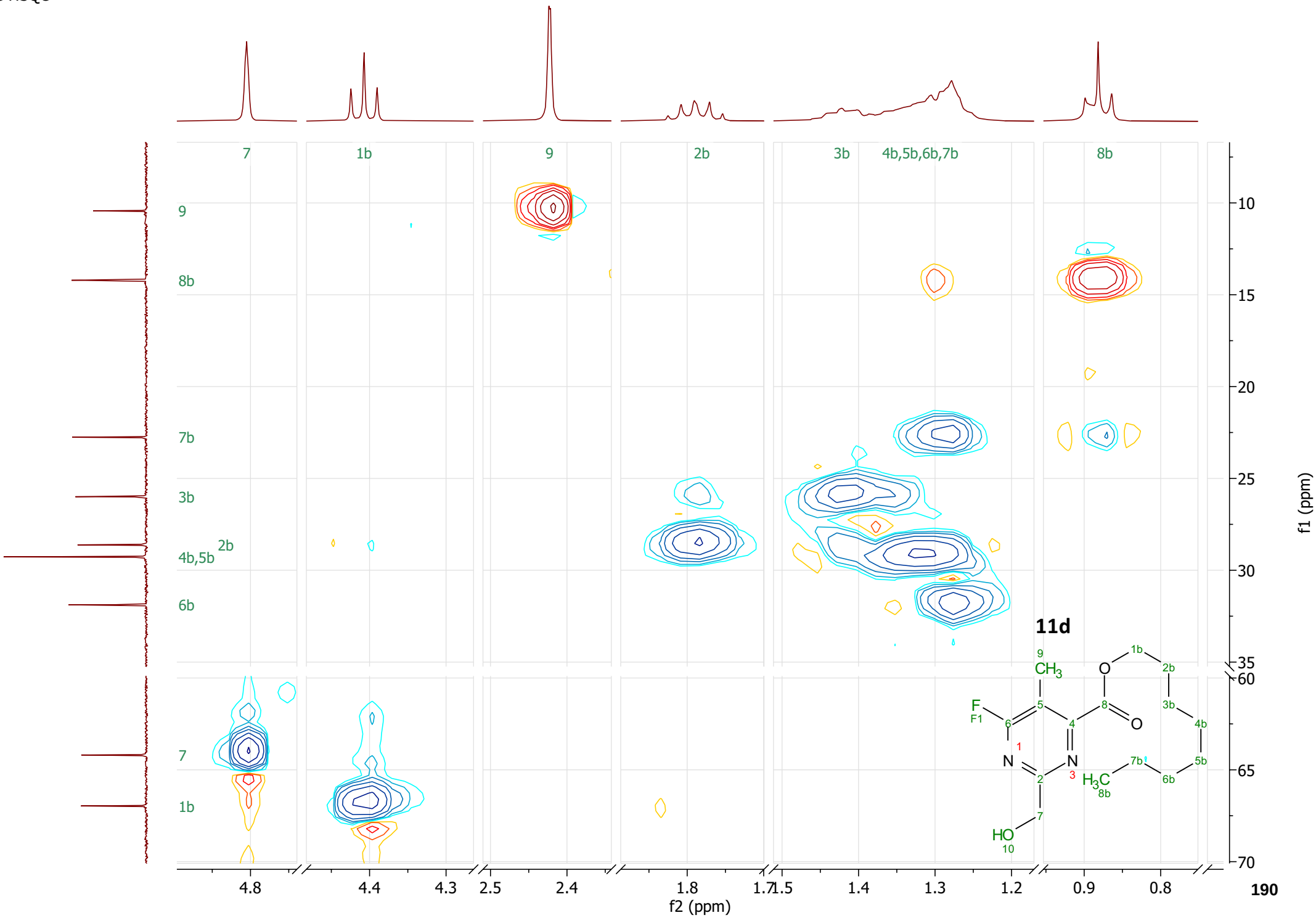
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.81 (app t, *J* = 0.9 Hz, 2H), 4.41 (t, *J* = 6.8 Hz, 2H), 2.86 (br s, 1H), 2.42 (app q, *J* = 0.8 Hz, 3H), 1.86 – 1.71 (m, 2H), 1.47 – 1.38 (m, 2H), 1.38 – 1.21 (m, 8H), 0.87 (app t, 3H).



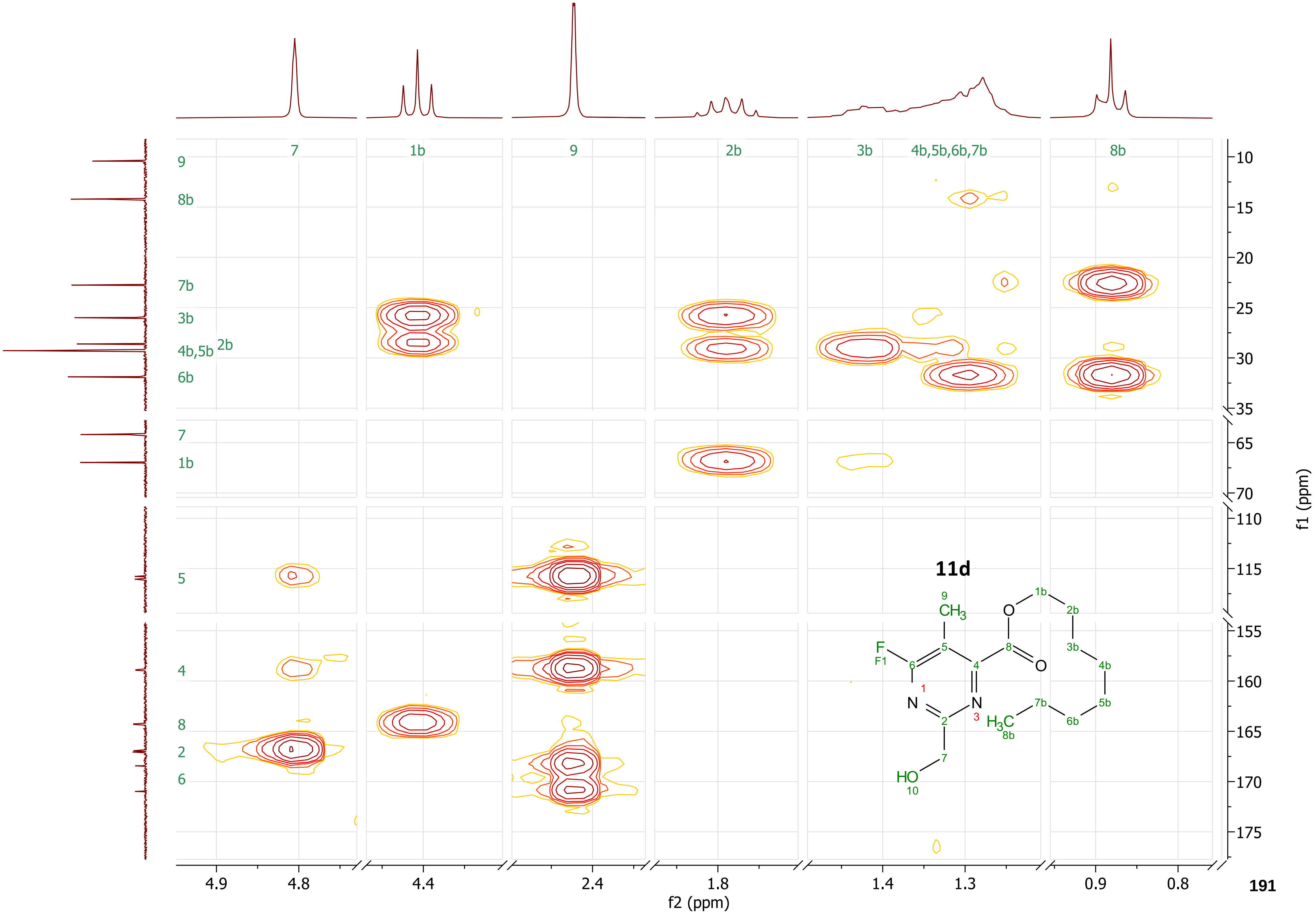
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.7 (d, J = 256.4 Hz), 167.0 (d, J = 13.3 Hz), 164.3 (d, J = 5.8 Hz), 158.9 (d, J = 5.9 Hz), 115.9 (d, J = 27.3 Hz), 67.0, 64.2, 31.9, 29.3 (2C), 28.6, 26.0, 22.8, 14.2, 10.4.



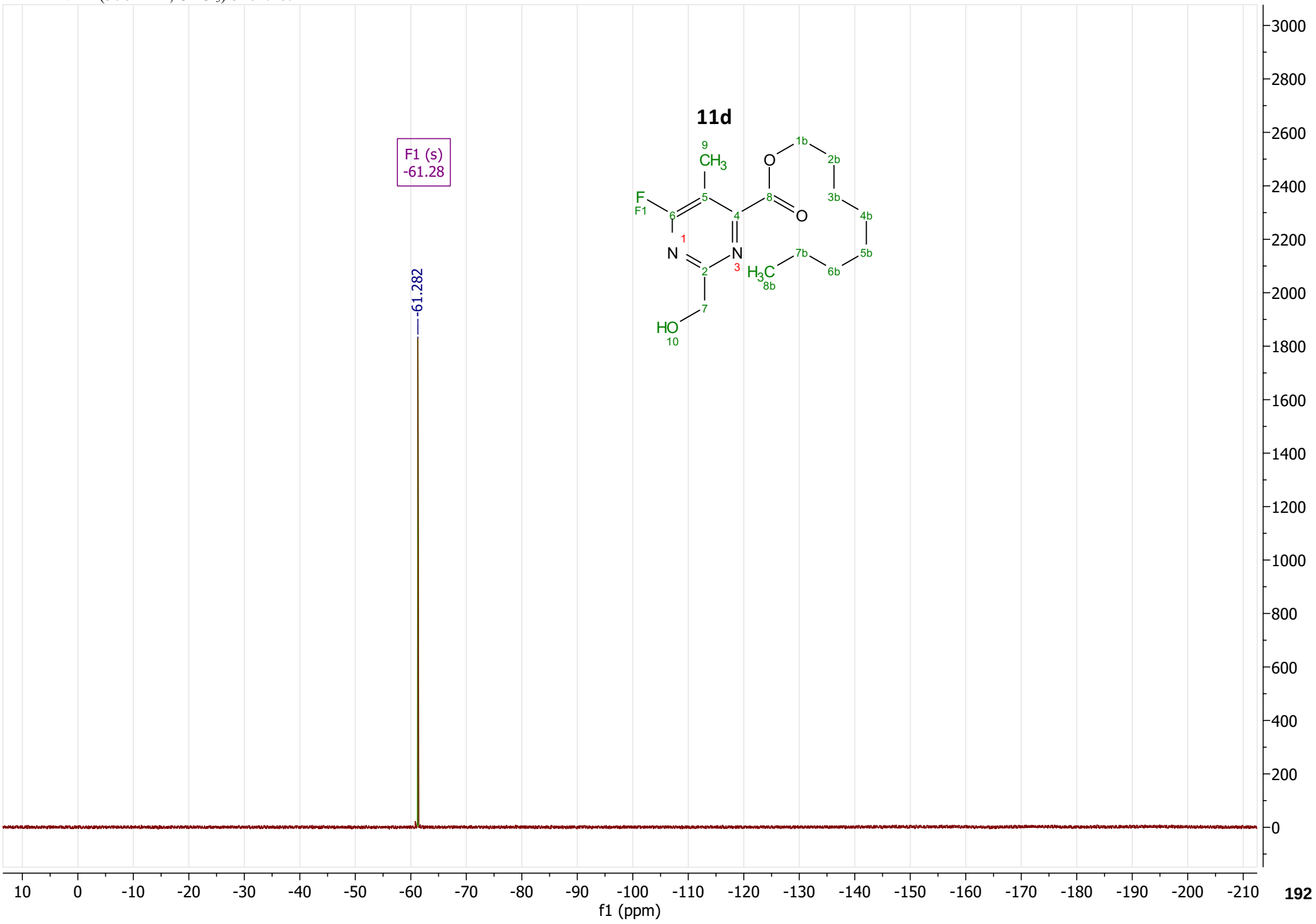
13C HSQC


7
1b
9
2b
3b
4b,5b,6b,7b
8b
9
8b
7b
3b
4b,5b
2b
6b
7
1b
11d
f2 (ppm)
f1 (ppm)
190

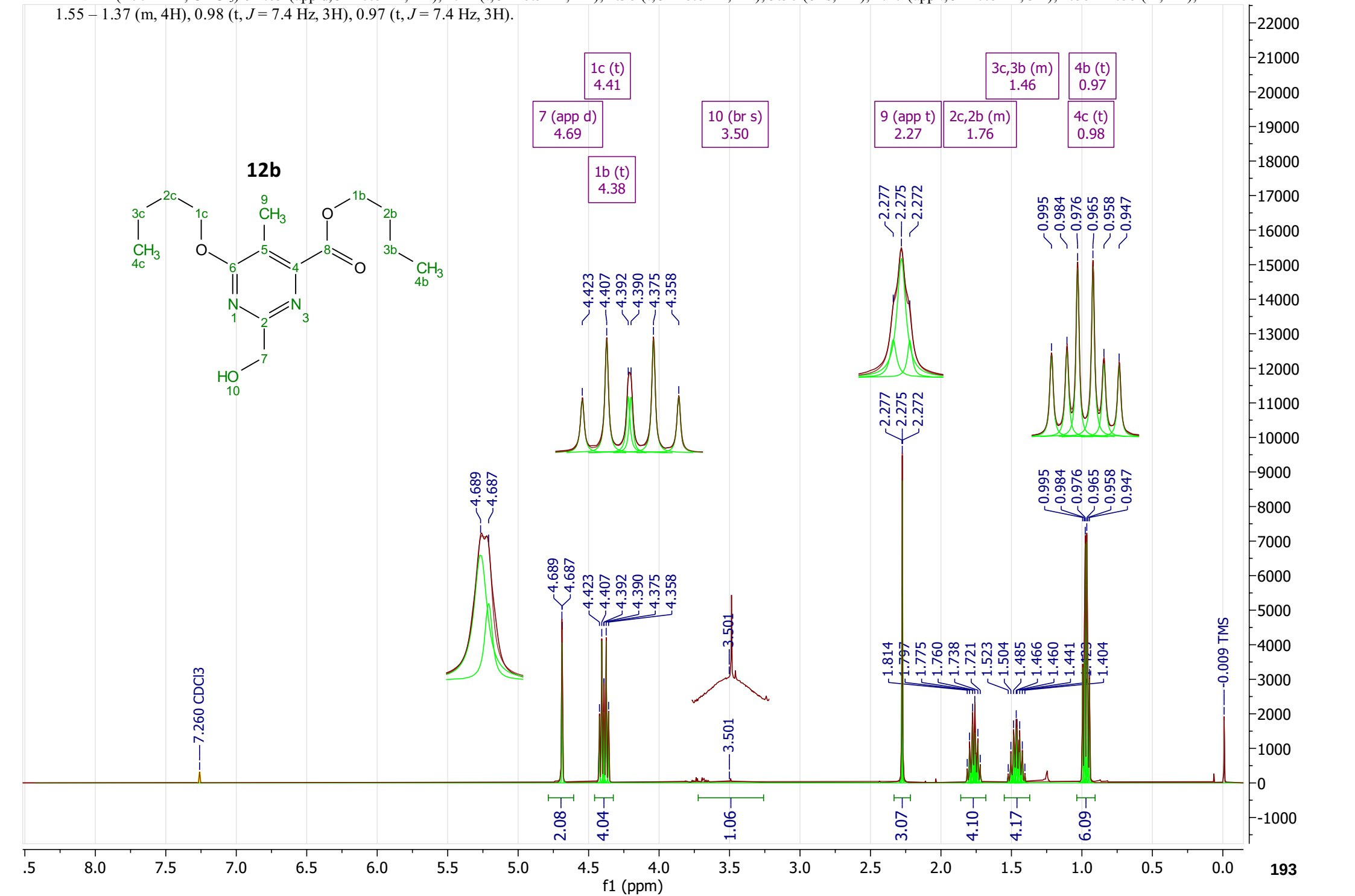
13C HMBC


7
1b
9
2b
3b
4b,5b,6b,7b
8b
9
8b
7b
3b
4b,5b 2b
6b
7
1b
5
4
8
2
6
11d
f2 (ppm)
f1 (ppm)
4.9
4.8
4.4
2.4
1.8
1.4
1.3
0.9
0.8

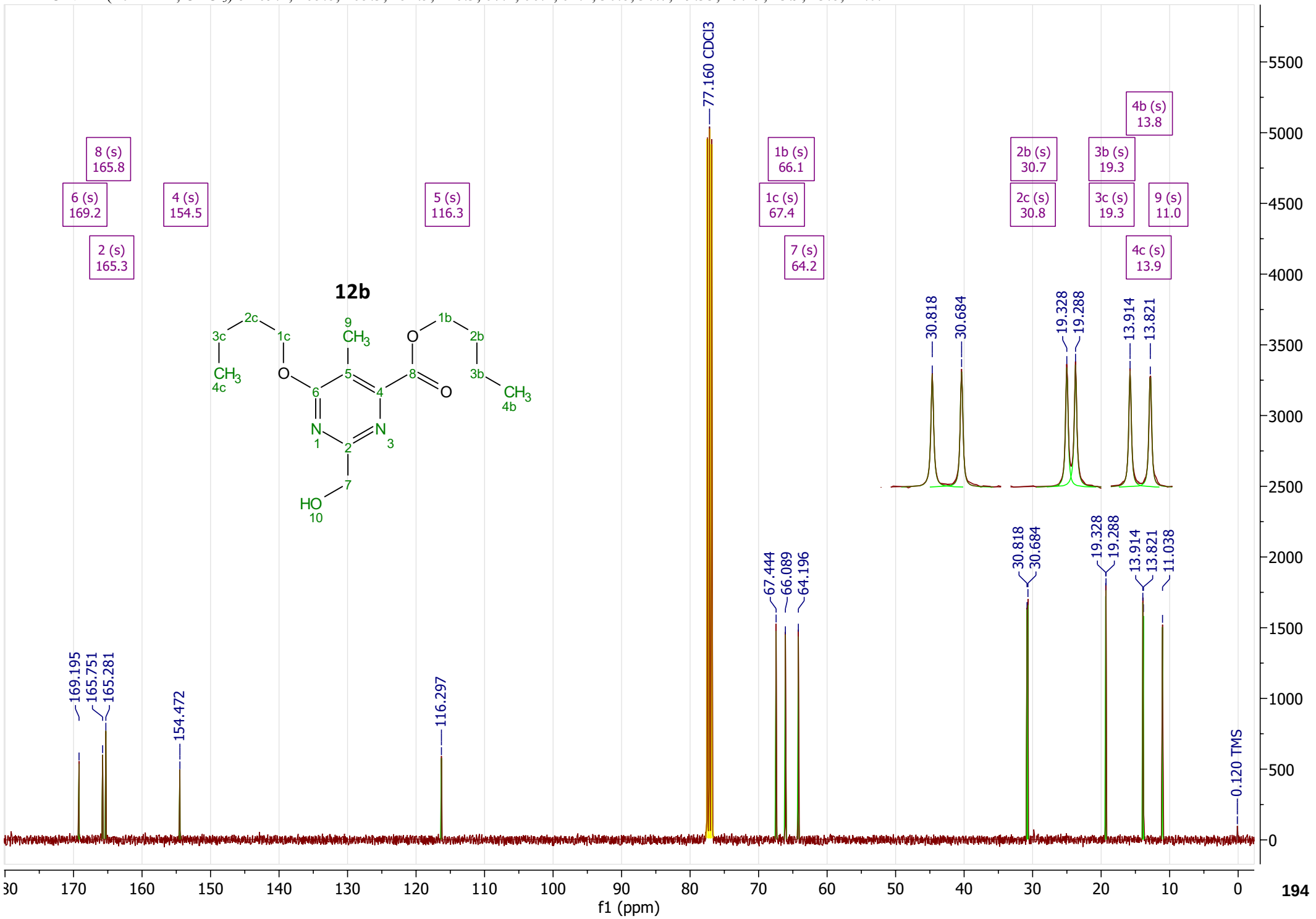
19F ^{19}F NMR (376 MHz, $CDCl_3$) δ -61.28.



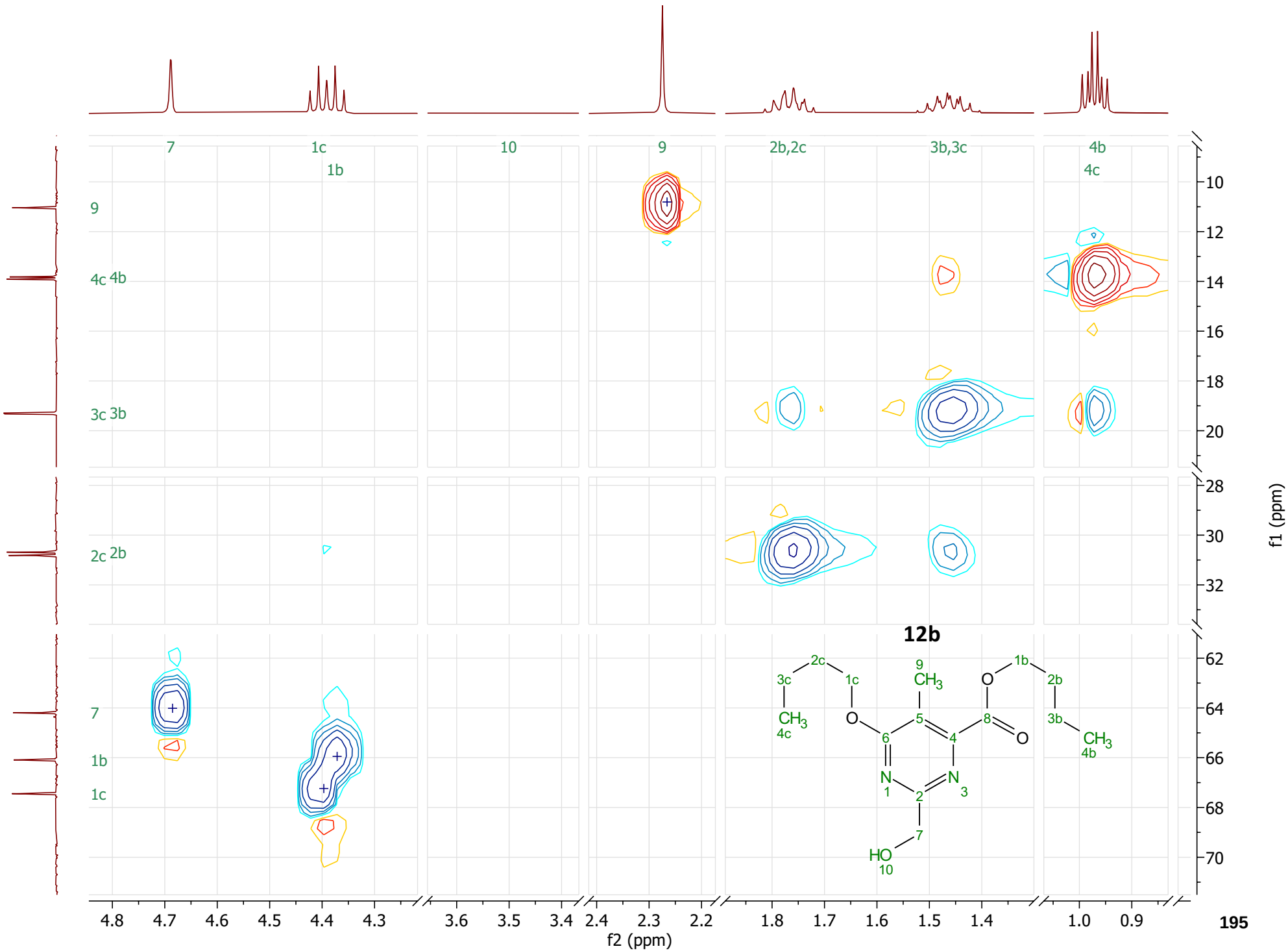
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.69 (app d, *J* = 0.8 Hz, 2H), 4.41 (t, *J* = 6.5 Hz, 2H), 4.38 (t, *J* = 6.8 Hz, 2H), 3.50 (br s, 1H), 2.27 (app t, *J* = 0.8 Hz, 3H), 1.86 – 1.68 (m, 4H), 1.55 – 1.37 (m, 4H), 0.98 (t, *J* = 7.4 Hz, 3H), 0.97 (t, *J* = 7.4 Hz, 3H).



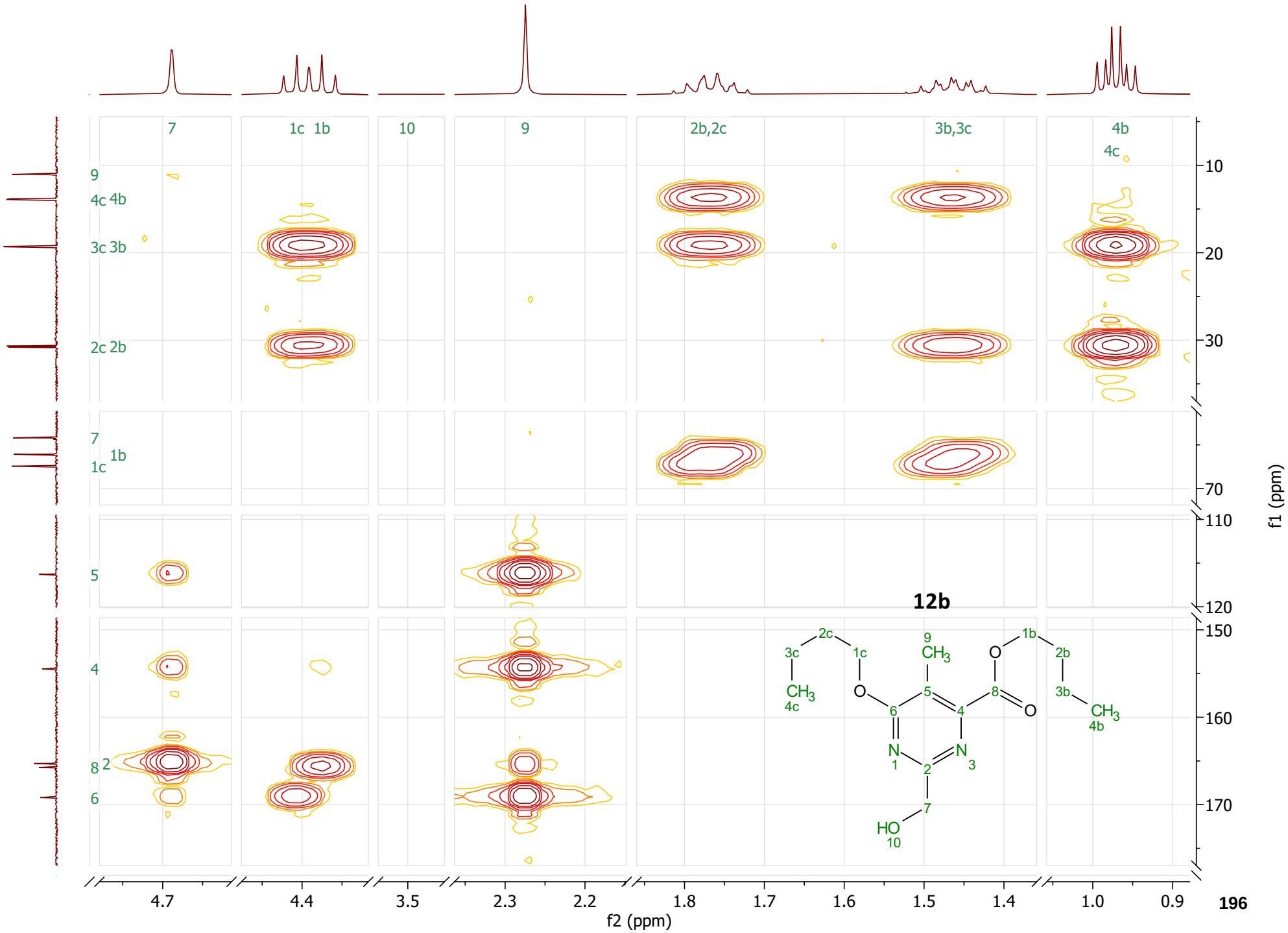
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 165.8, 165.3, 154.5, 116.3, 67.4, 66.1, 64.2, 30.8, 30.7, 19.33, 19.29, 13.9, 13.8, 11.0.



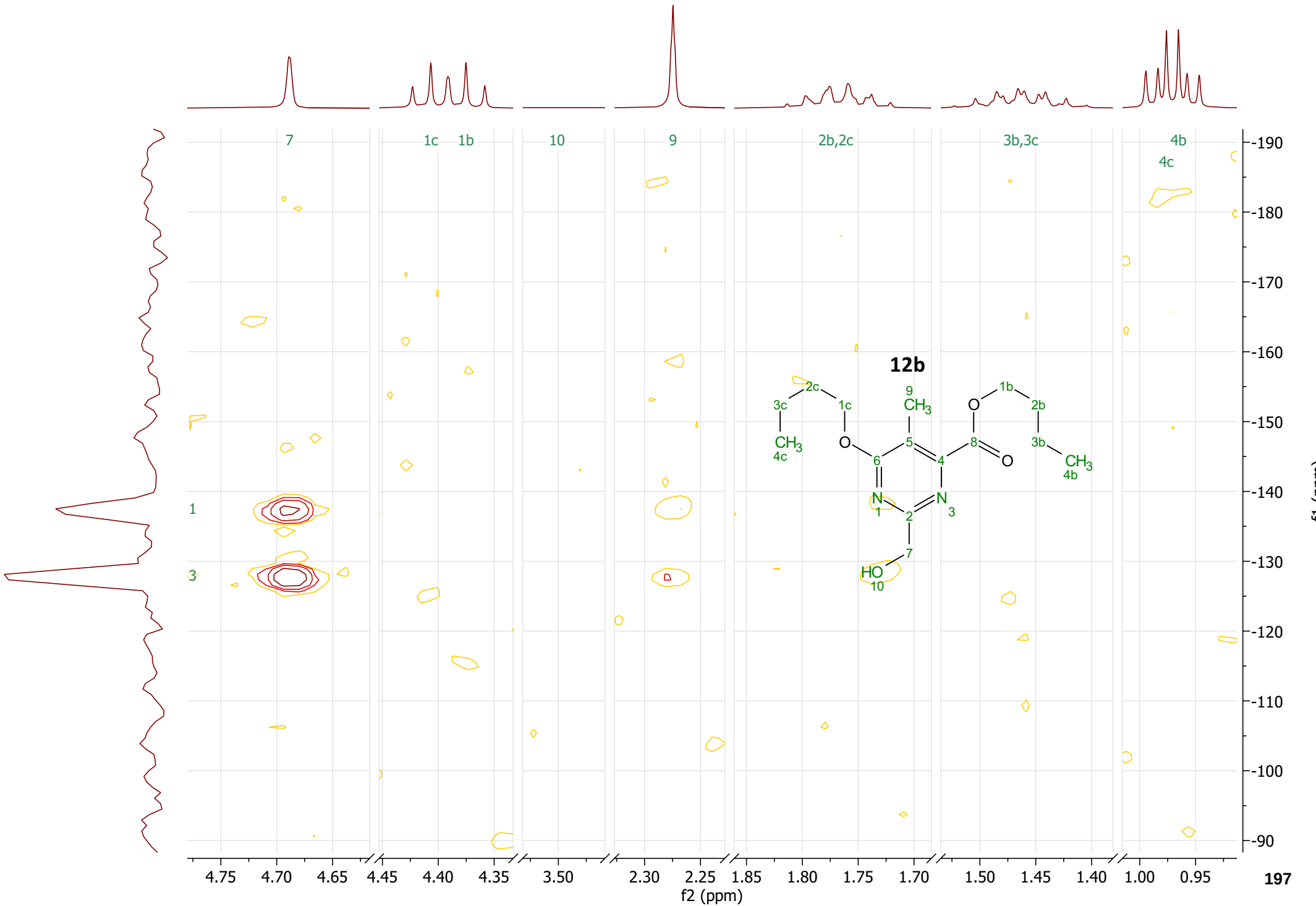
13C HSQC


7
1c
1b
10
9
2b,2c
3b,3c
4b
4c
9
4c 4b
3c 3b
2c 2b
7
1b
1c
12b
f2 (ppm)
f1 (ppm)

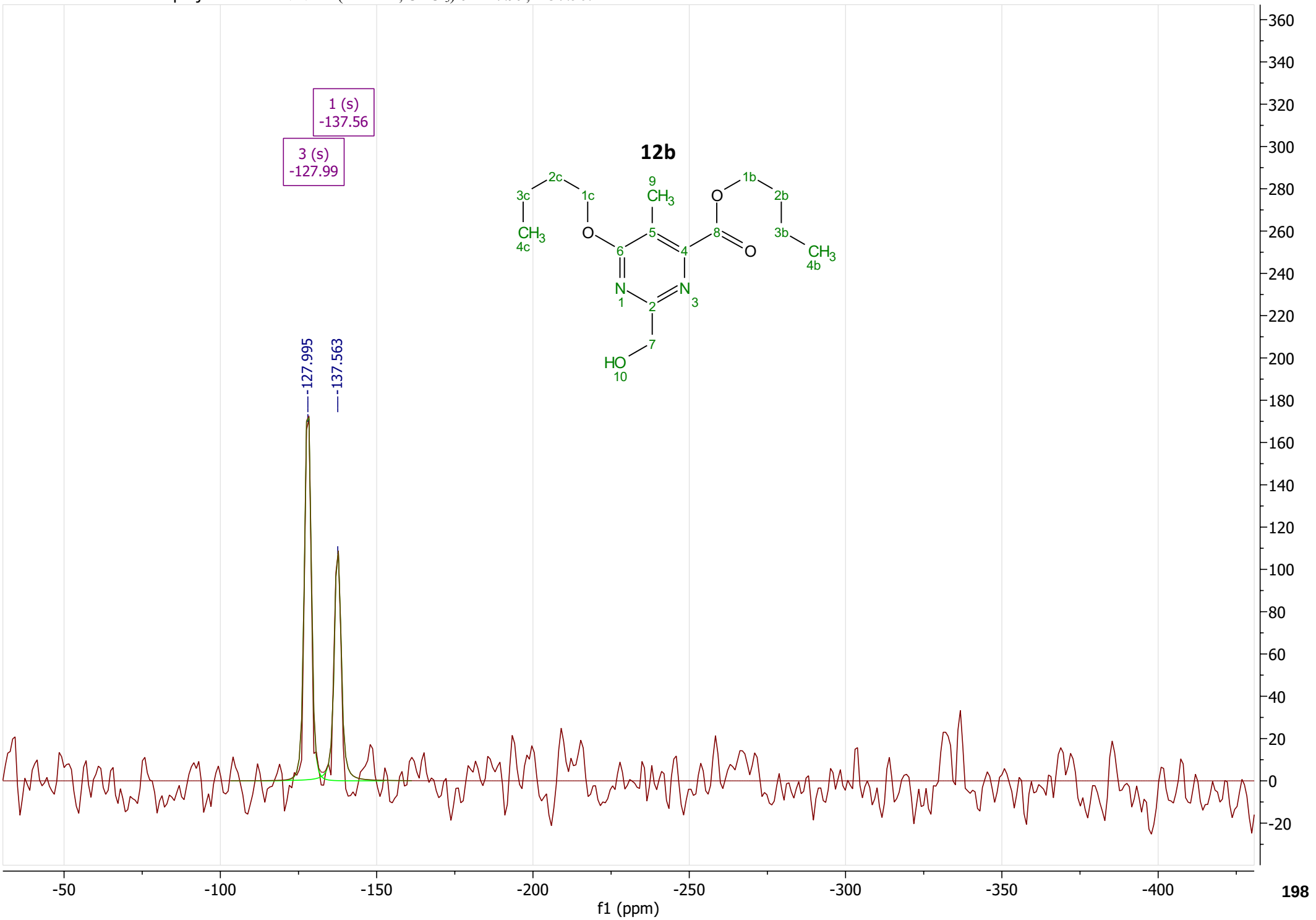
13C HMBC


7
1c 1b
10
9
2b,2c
3b,3c
4b
4c
9
4c 4b
3c 3b
2c 2b
7
1c 1b
5
4
8 2
6
10
20
30
70
110
120
150
160
170
f1 (ppm)
4.7
4.4
3.5
2.3
2.2
1.8
1.7
1.6
1.5
1.4
1.0
0.9
f2 (ppm)
12b
CH3
4c
3c
2c
1c
O
6
5
9
CH3
4
8
O
1b
O
2b
3b
CH3
4b
N
1
2
N
3
7
HO
10

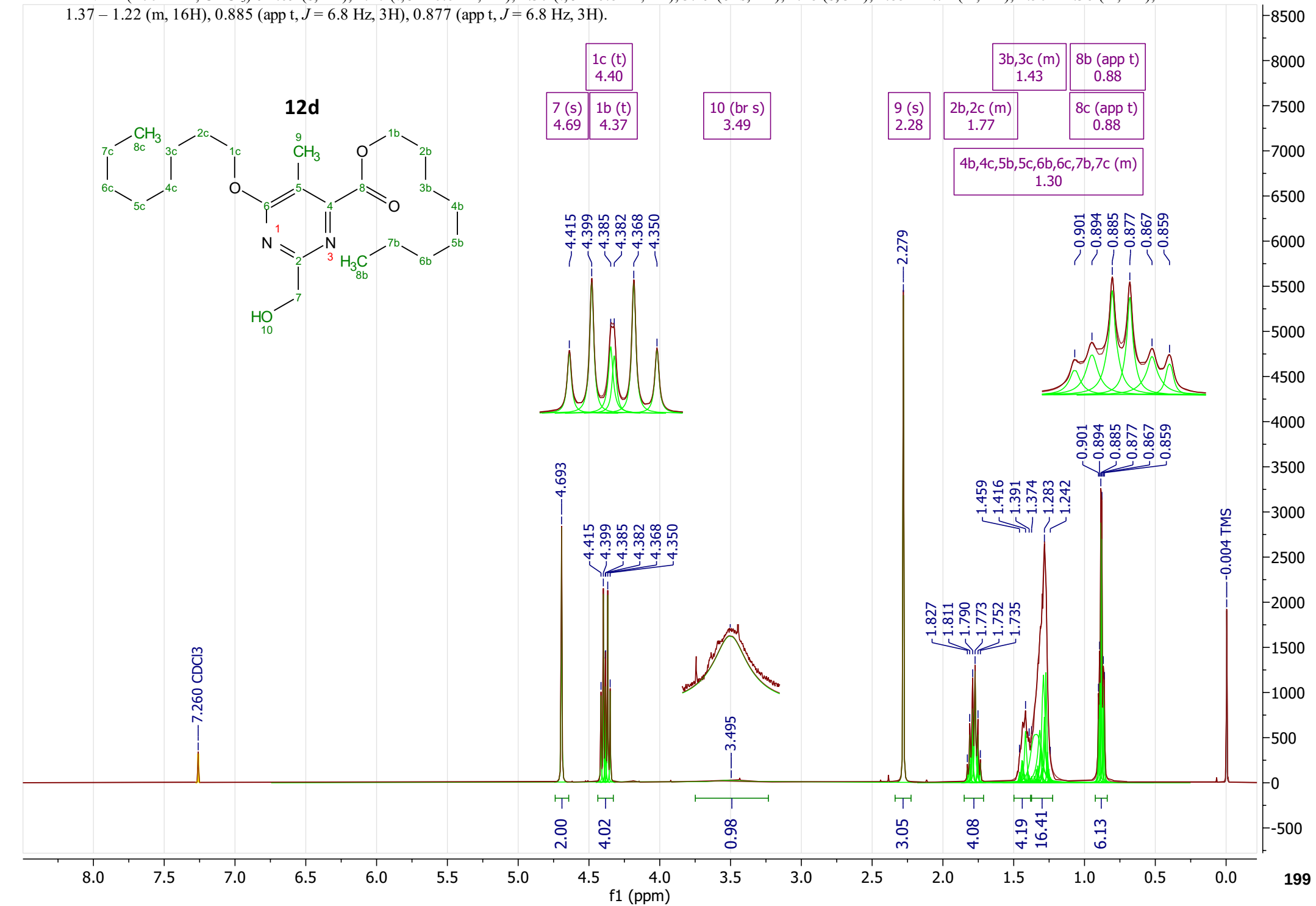
15N HMBC


7
1c
1b
10
9
2b,2c
3b,3c
4b
4c
1
3
12b
9
CH3
2c
3c
1c
CH3
4c
O
6
5
4
8
O
1b
2b
3b
CH3
4b
N
1
2
N
3
7
HO
10
-190
-180
-170
-160
-150
-140
-130
-120
-110
-100
-90
f1 (ppm)
4.75
4.70
4.65
4.45
4.40
4.35
3.50
2.30
2.25
1.85
1.80
1.75
1.70
1.50
1.45
1.40
1.00
0.95
f2 (ppm)

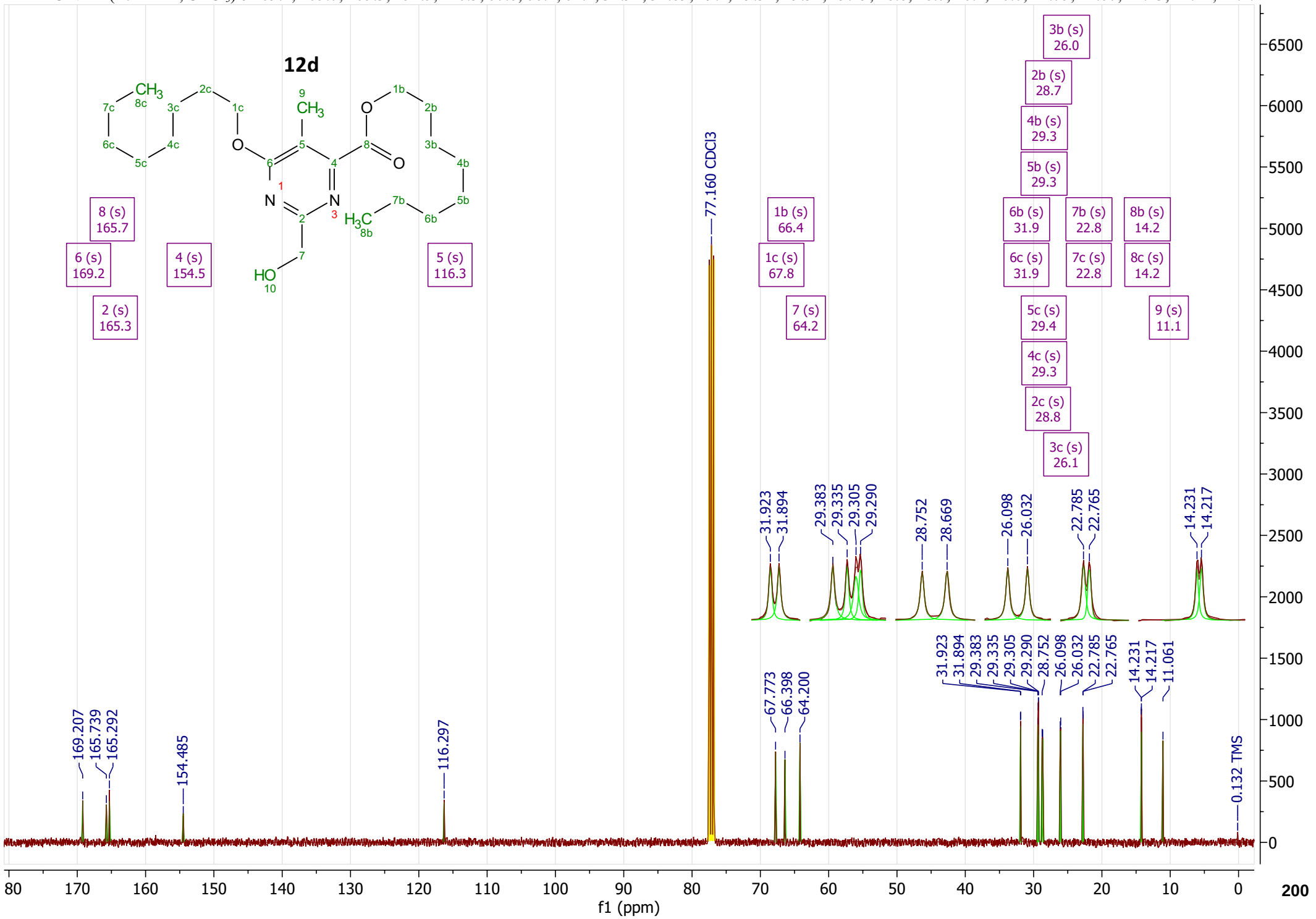
15N HMBC - f1 internal projection ^{15}N NMR (41 MHz, $CDCl_3$) δ -127.99, -137.56.



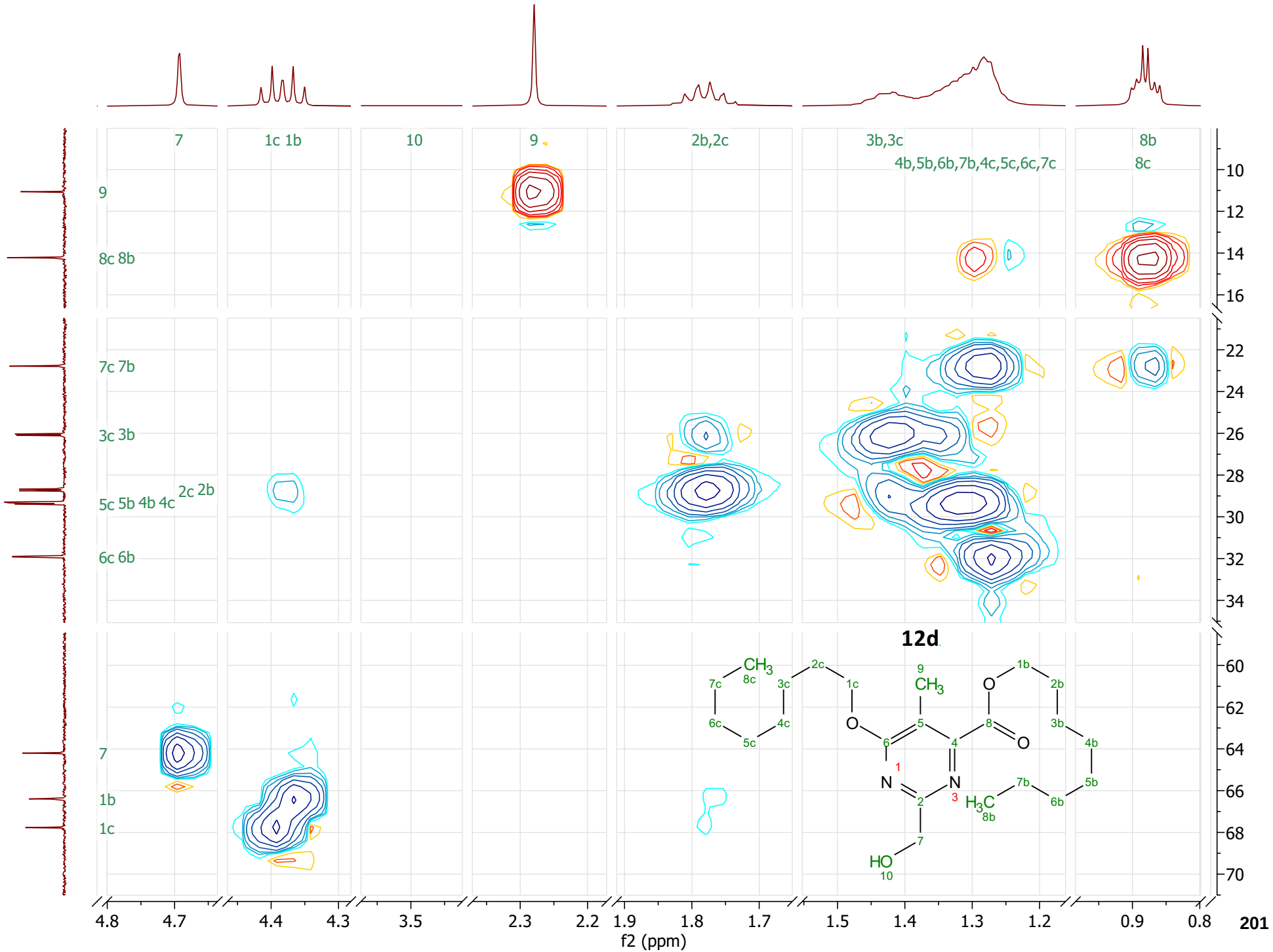
1H ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.69 (s, 2H), 4.40 (t, *J* = 6.6 Hz, 2H), 4.37 (t, *J* = 6.8 Hz, 2H), 3.49 (br s, 1H), 2.28 (s, 3H), 1.85 – 1.71 (m, 4H), 1.50 – 1.38 (m, 4H), 1.37 – 1.22 (m, 16H), 0.885 (app t, *J* = 6.8 Hz, 3H), 0.877 (app t, *J* = 6.8 Hz, 3H).



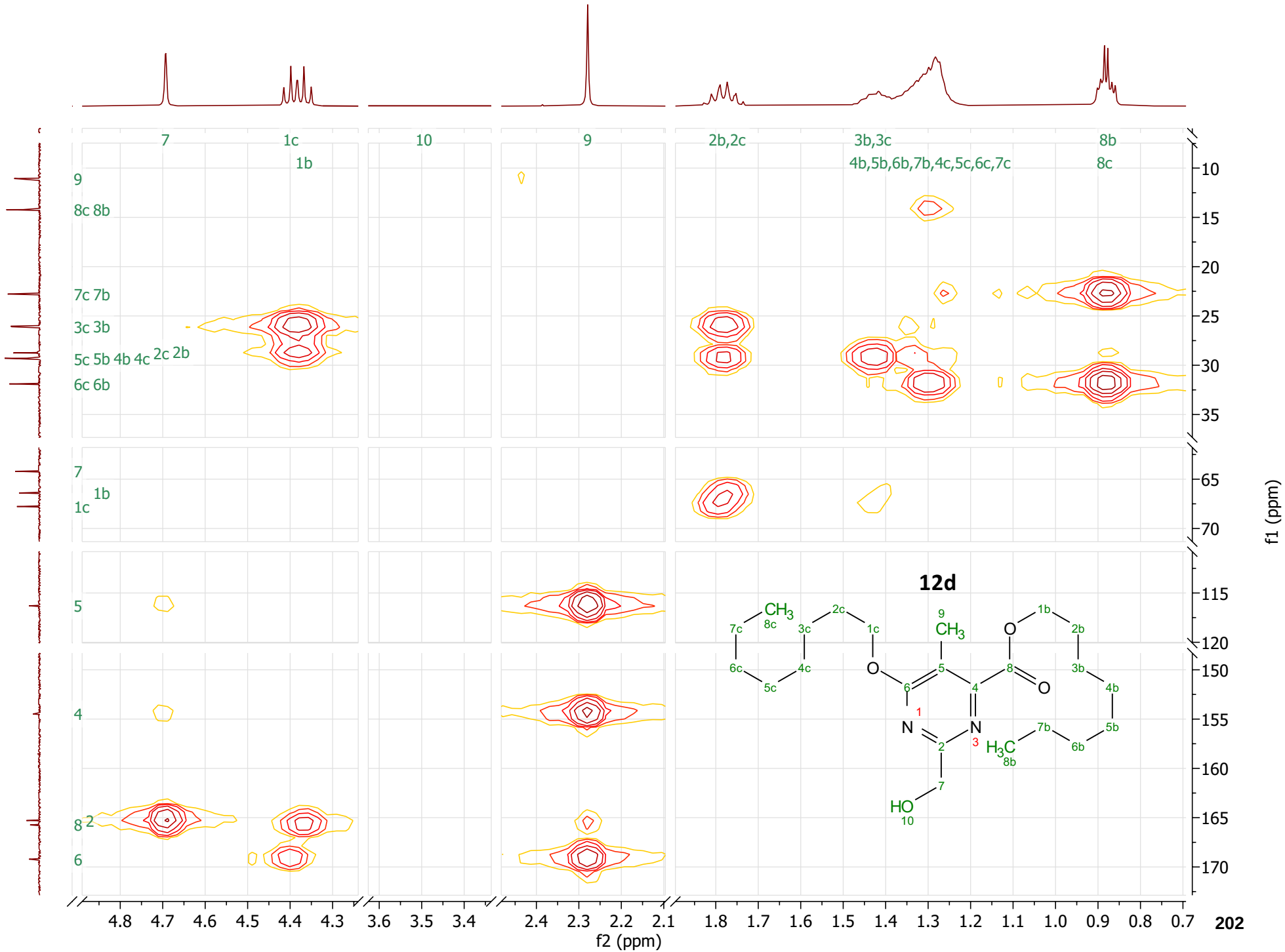
13C ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 165.7, 165.3, 154.5, 116.3, 67.8, 66.4, 64.2, 31.92, 31.89, 29.4, 29.34, 29.31, 29.29, 28.8, 28.7, 26.1, 26.0, 22.75, 22.67, 14.23, 14.22, 11.1.



13C HSQC


7
1c 1b
10
9
2b,2c
3b,3c
4b,5b,6b,7b,4c,5c,6c,7c
8b
8c
9
8c 8b
7c 7b
3c 3b
5c 5b 4b 4c
2c 2b
6c 6b
7
1b
1c
12d
f1 (ppm)
f2 (ppm)
4.8
4.7
4.4
4.3
3.5
2.3
2.2
1.9
1.8
1.7
1.5
1.4
1.3
1.2
0.9
0.8
10
12
14
16
22
24
26
28
30
32
34
60
62
64
66
68
70

13C HMBC


7
1c
1b
10
9
2b,2c
3b,3c
4b,5b,6b,7b,4c,5c,6c,7c
8b
8c
9
8c 8b
7c 7b
3c 3b
5c 5b 4b 4c 2c 2b
6c 6b
7
1c
1b
5
4
8
2
6
12d
f2 (ppm)
f1 (ppm)
202