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Conformational Comparison of Cyclic $\alpha_3\beta$ Tetra- *vs* $\alpha_4\beta$ Pentapeptides

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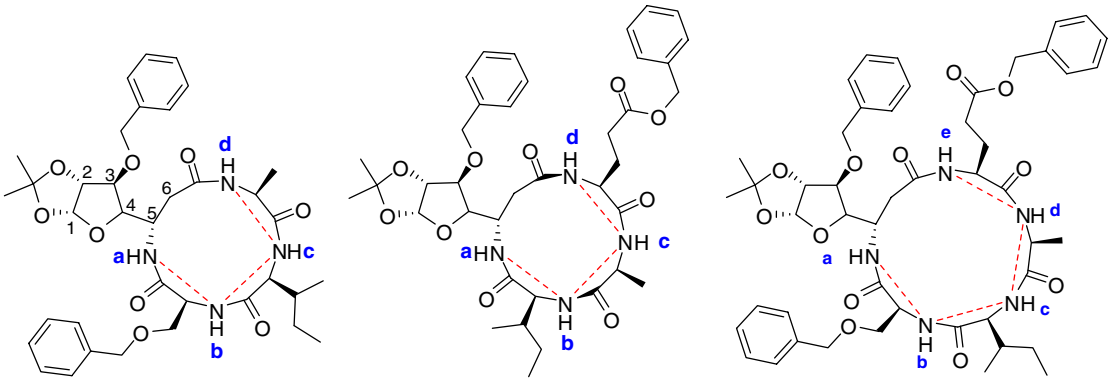
Material and Methods

High resolution mass spectroscopic analysis was performed on a Bruker MicroTOF QII mass spectrometer in positive mode with an internal calibration. All ^{1}H, ^{13}C COSY, HSQC, HMBC spectra were recorded on a 9.4T Bruker AVANCE III 600 MHz instrument at room temperature (298K) with DMSO as an internal standard using a sample concentration in the range of 20 (mg/ml) and all ROESY Experiments were performed at room temperature (298K). Samples were dissolved in 450 µl of deuterated DMSO-d_6 and measured at 25 °C.. Processing and assignments were carried out using the Topspin 2.3 software from Bruker Karlsruhe.



2a **2b** **3**

Table 1: ^{1}H NMR Chemical Shifts (δ, ppm) and Coupling Constants (*J*, Hz) of glycopeptides **2a**, **2b** and **3**

No.	2a	2b	3
Glycosyl			
1	5.81 (d, *J*= 3.8Hz)	5.82 (d, *J*=3.9Hz)	5.80 (d, *J*=3.0Hz)
2	4.77 (d, *J*=3.8Hz)	4.77 (d, *J*=3.9Hz)	4.76 (d, *J*=3.0Hz)
3	4.00 (d, *J*=2.7Hz)	4.04 (d, *J*=2.9Hz)	3.88-3.90 (m)
4	4.69 (dd, Merged with H-8, *J*=10.8, 2.7Hz)	4.60 (dd, *J*=10.0, 2.9Hz)	4.41-4.49 (m)
5	4.27 (m)	4.21 (t, *J*=7.8Hz)	4.20-4.24 (m)
6a	2.35 (m)	2.25 (d, *J*=15.1Hz)	2.34 (d, *J*=14.3Hz)
6b	2.53 (dd, *J*=6.1, 6.0Hz)	2.57 (dd, *J*=15.1,	2.58 (d, *J*= 14.3Hz)
NH	6.85 (d, *J*=9.7Hz)	6.95 (d, *J*=9.0Hz)	7.75 (d, *J*=8.8Hz)

Glutamic			
NH		8.05 (d, *J*=6.9Hz)	7.96 (d, *J*=5.1Hz)
$C_{\alpha H}$		3.98-4.02 (m)	3.98 (d, *J*=7.9Hz)
$C_{\beta H}$		1.85-1.88 (m)	1.81-1.92 (m)
$C_{\gamma H}$		2.44 (t, *J*=15.1Hz)	2.41-2.42 (m)
Alanine			
NH	8.24 (m)	7.70 (d, *J*=8.6Hz)	8.10 (d, *J*=7.9Hz)
$C_{\alpha H}$	3.93 (m)	4.21 (t, *J*=7.8Hz)	4.12 (t, *J*=7.9Hz)
$C_{\beta H}$	1.22 (d, *J*=7.3Hz)	1.23 (d, *J*=7.3Hz)	1.29 (d, *J*=7.0Hz)
Isoleucine			
NH	7.21 (d, *J*=7.3Hz)	7.66 (d, *J*=9.0Hz)	7.60 (d, *J*=6.9Hz)
$C_{\alpha H}$	3.91 (m)	3.98-4.02 (m)	3.88-3.90 (m)
$C_{\beta H}$	1.62 (m)	1.85-1.88 (m)	1.82-1.91 (m)
$C_{\gamma H}$	1.01 (m), 1.38 (m),	0.96 (m), 1.39 (m),	1.06-1.08 (m), 1.40 (m),
$C_{\delta H}$	0.82 (m)	0.80-0.83 (m)	0.75-0.80 (m)
Serine			
NH	8.23 (m)		8.19 (d, *J*=6.4Hz)
$C_{\alpha H}$	4.29 (m)		4.20-4.24 (m)
$C_{\beta H1}$	3.56 (dd, *J*=9.7, 5.8Hz)		3.71-3.73 (m)
$C_{\beta H2}$	3.72 (dd, *J*=9.7, 5.8Hz)		

Figure 1 ^{1}H NMR spectrum of **2a**

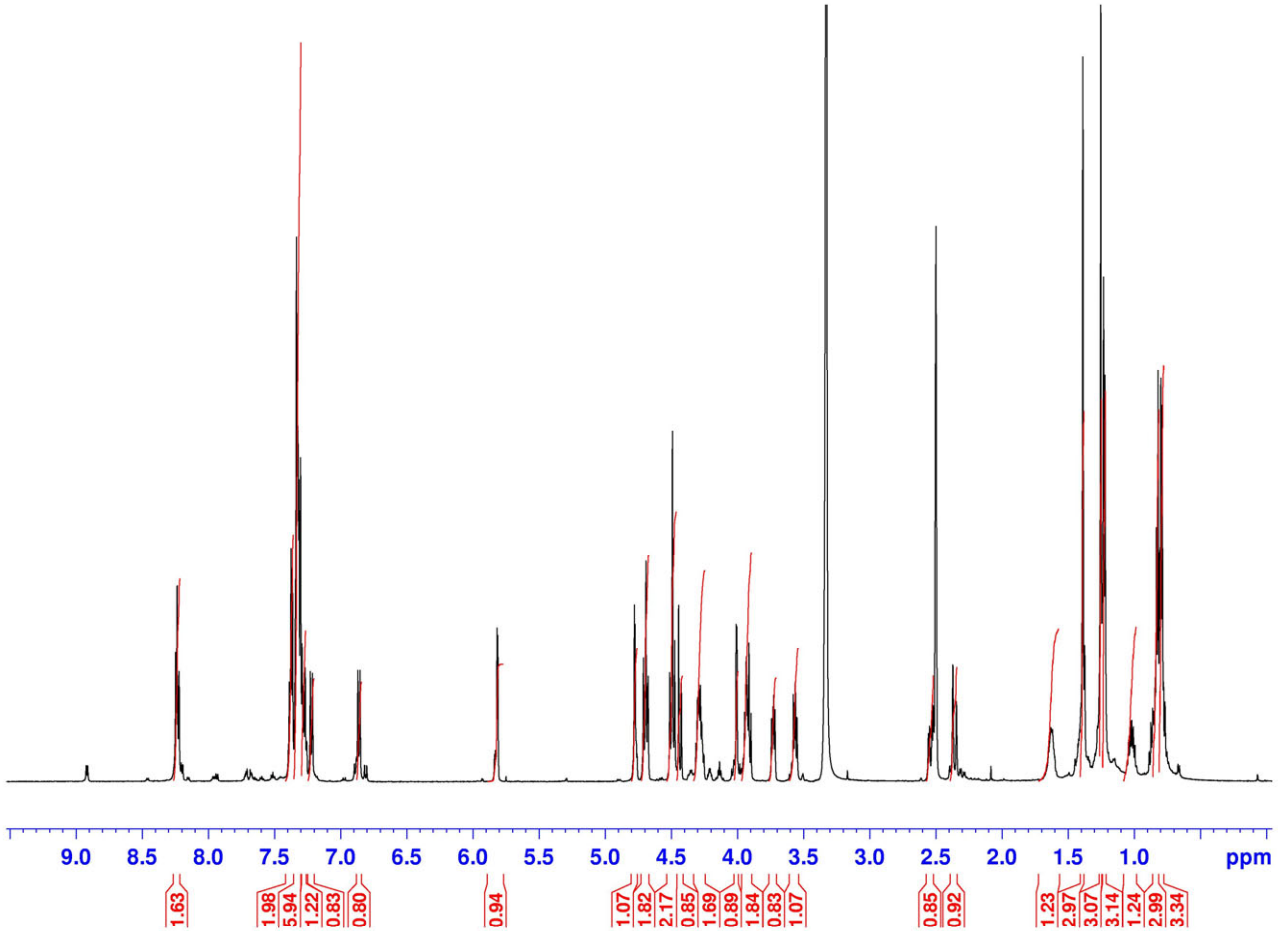


Figure 2 ^{13}C NMR spectrum of **2a**

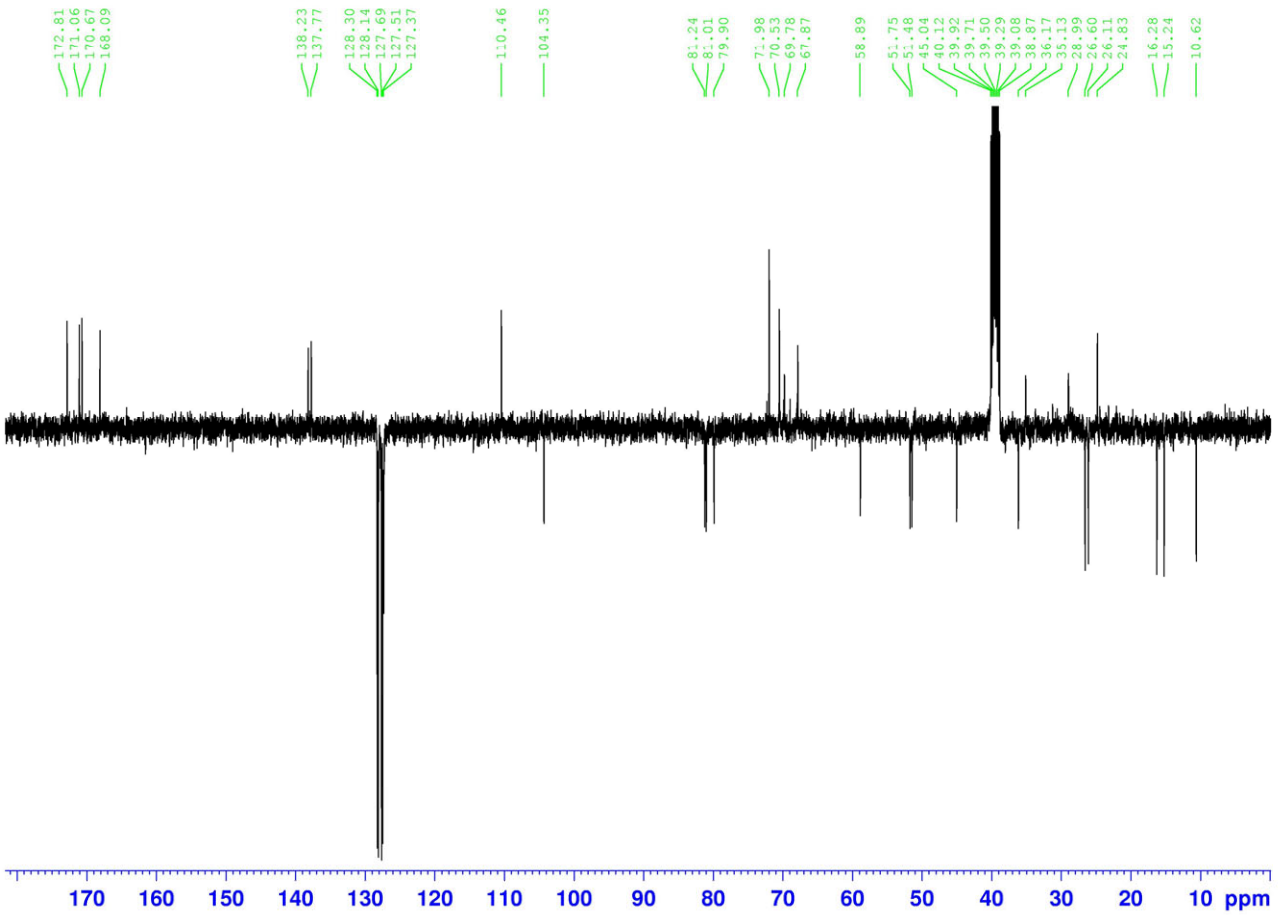


Figure 3 ROESY spectrum of **2a**

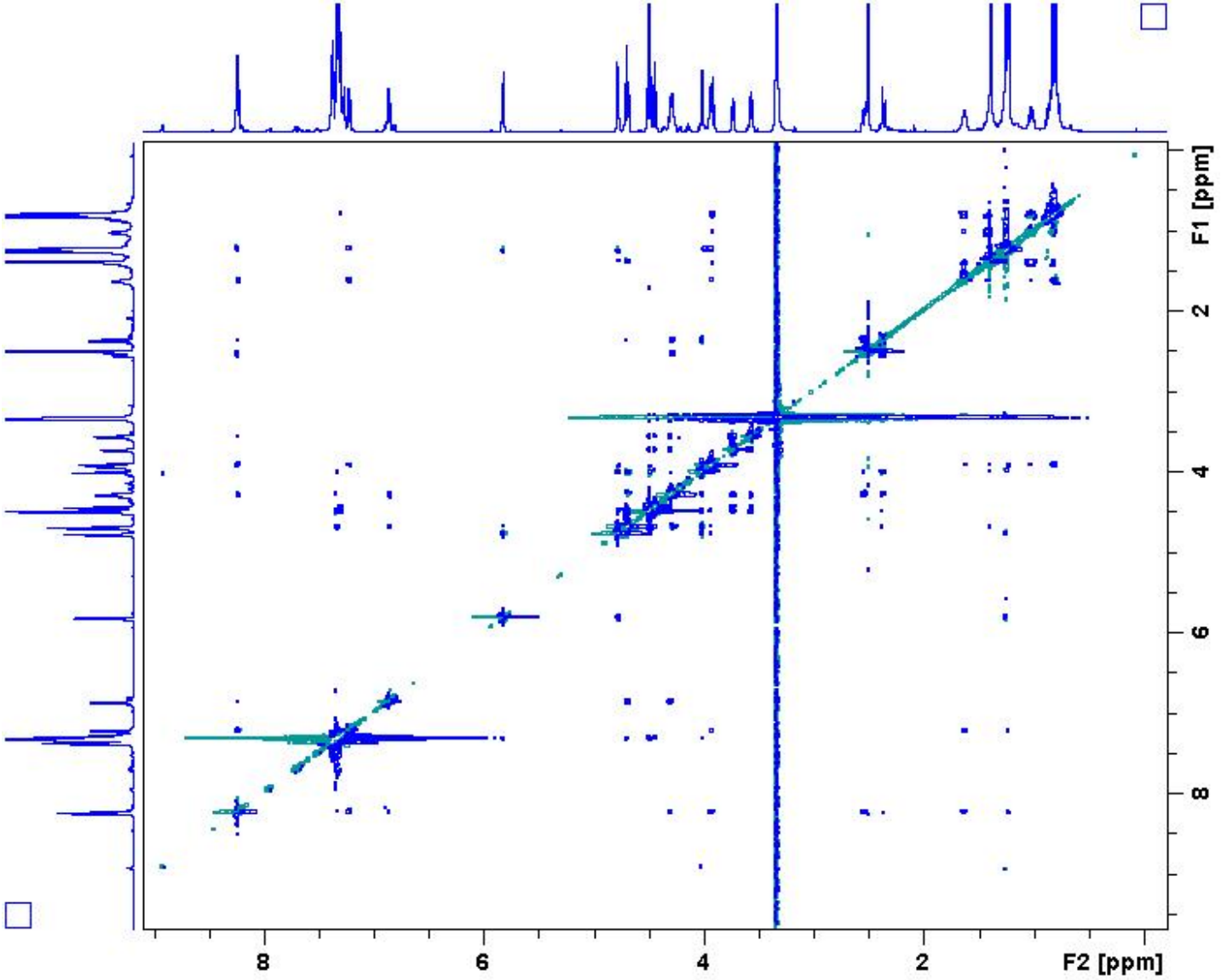


Figure 4 COSY spectrum of **2a**

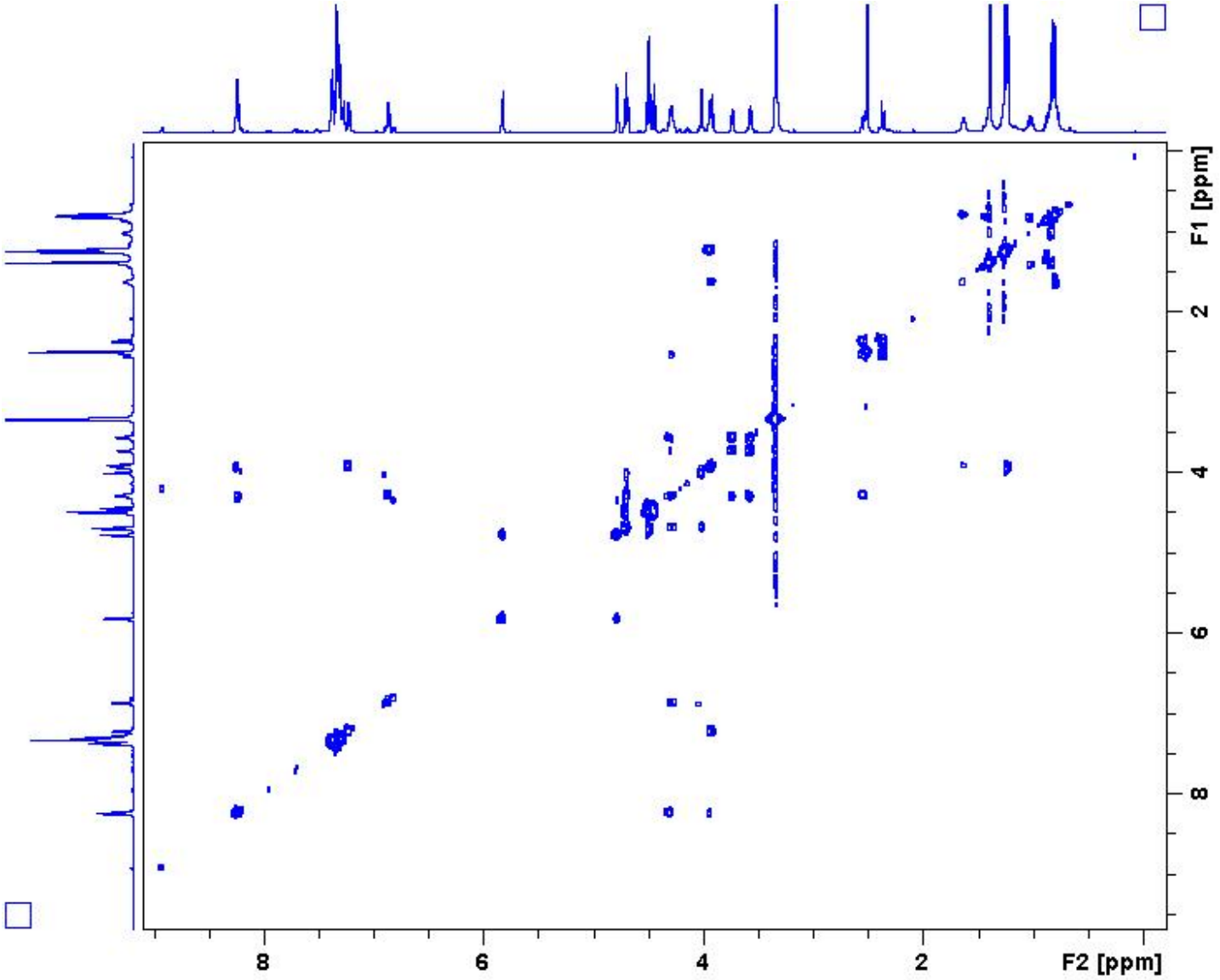


Figure 5 HSQC spectrum of **2a**

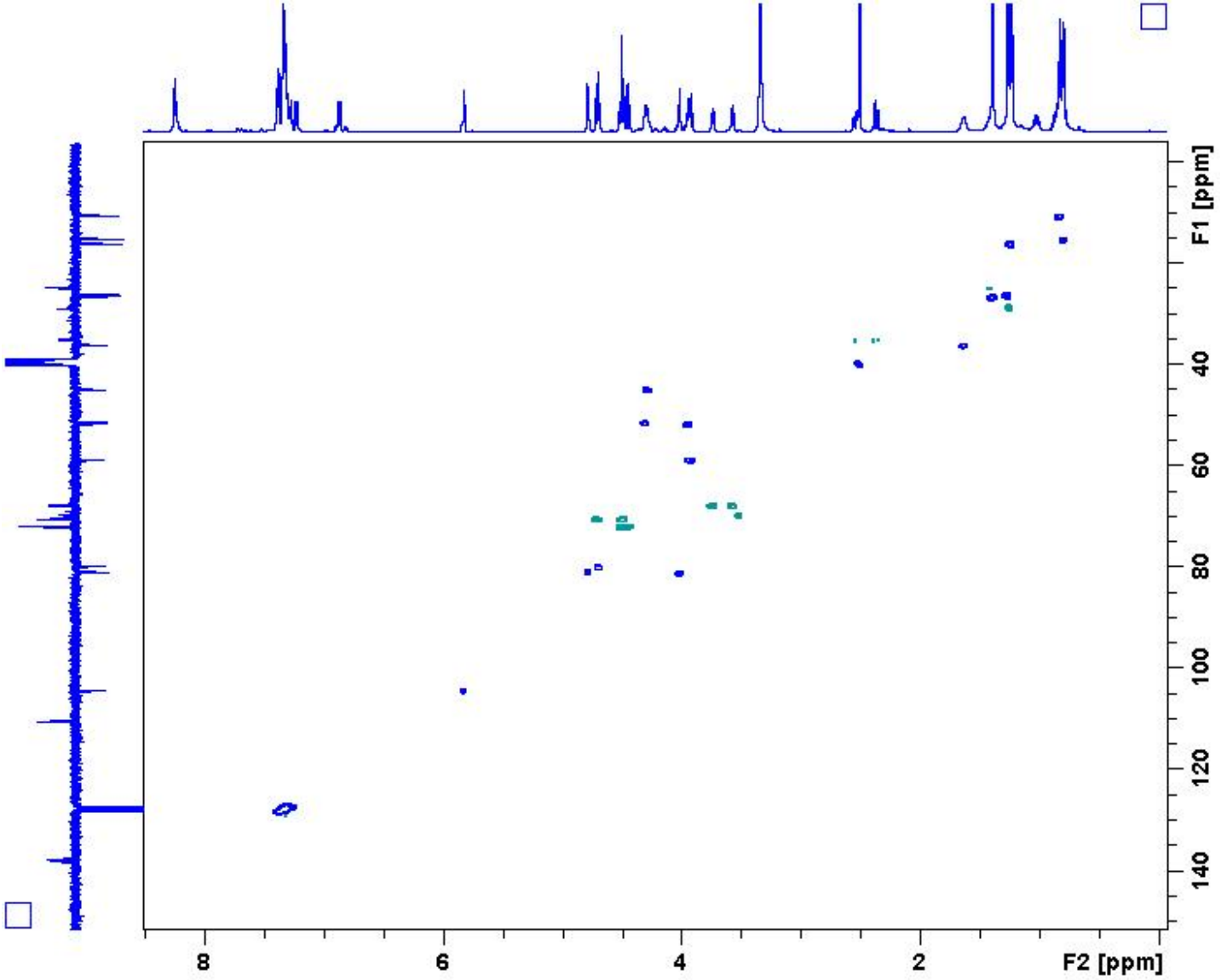


Figure 6 HMBC spectrum of **2a**

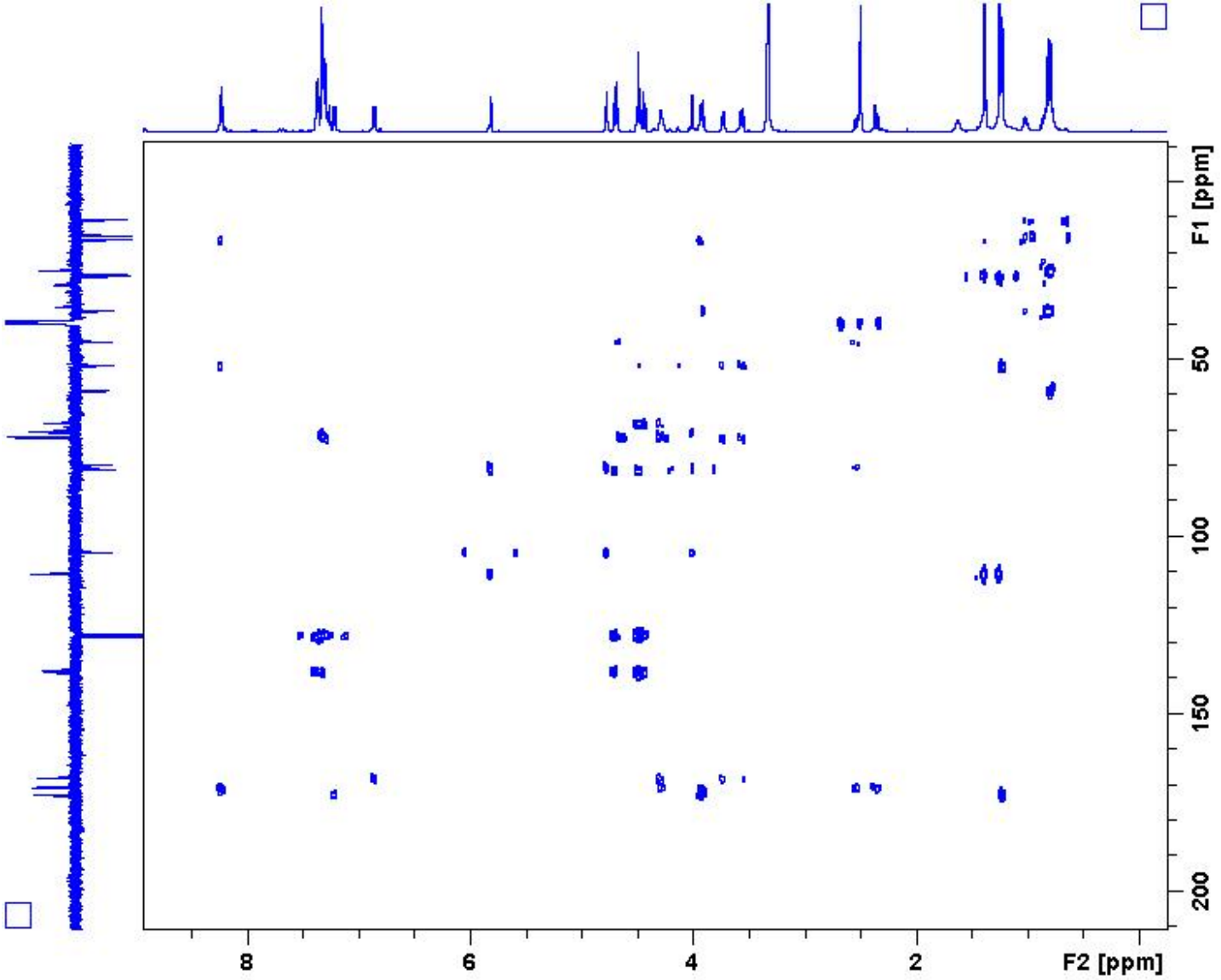


Figure 7 ^{1}H NMR spectrum of **2b**

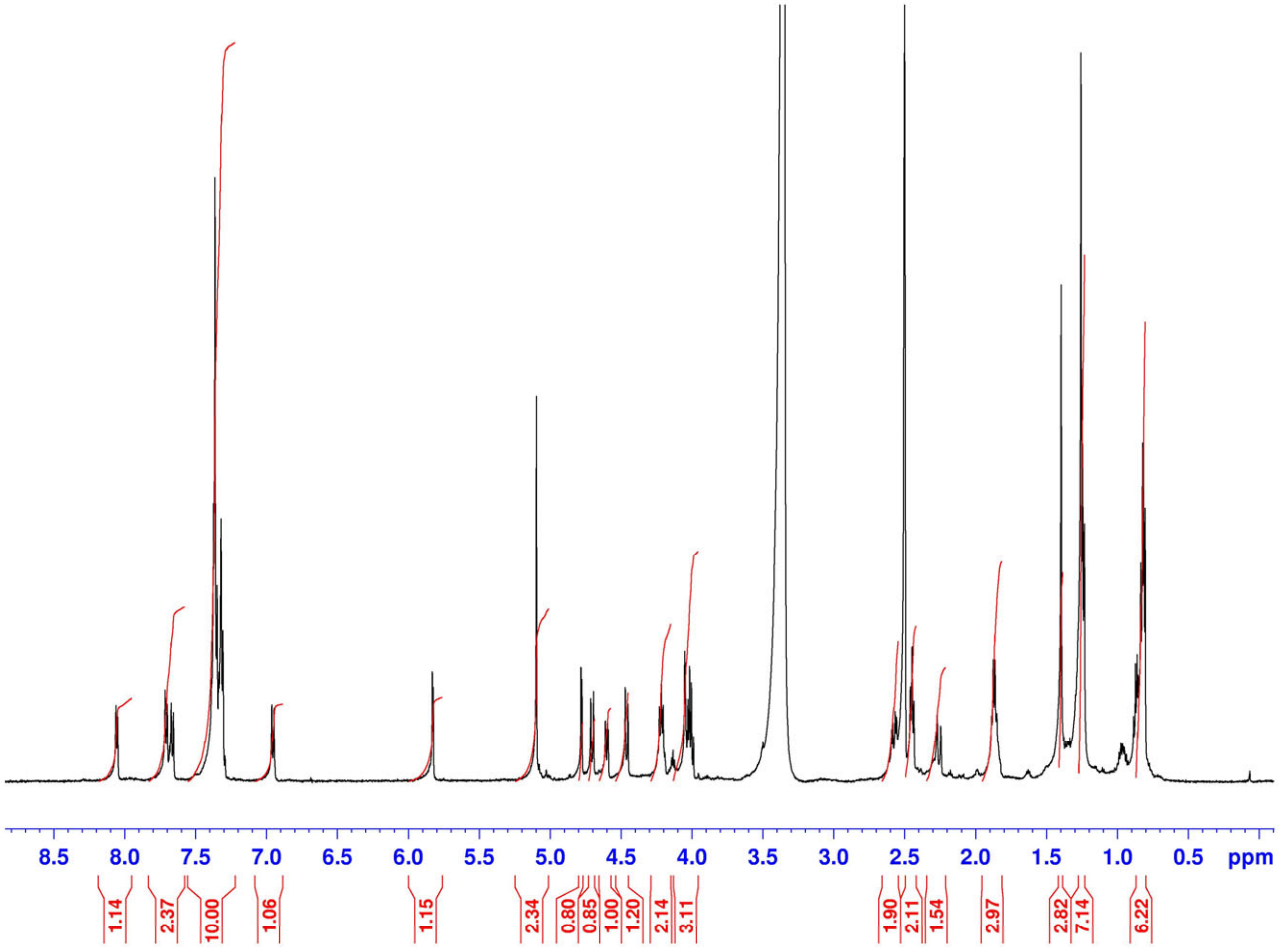


Figure 8 ^{13}C NMR spectrum of **2b**

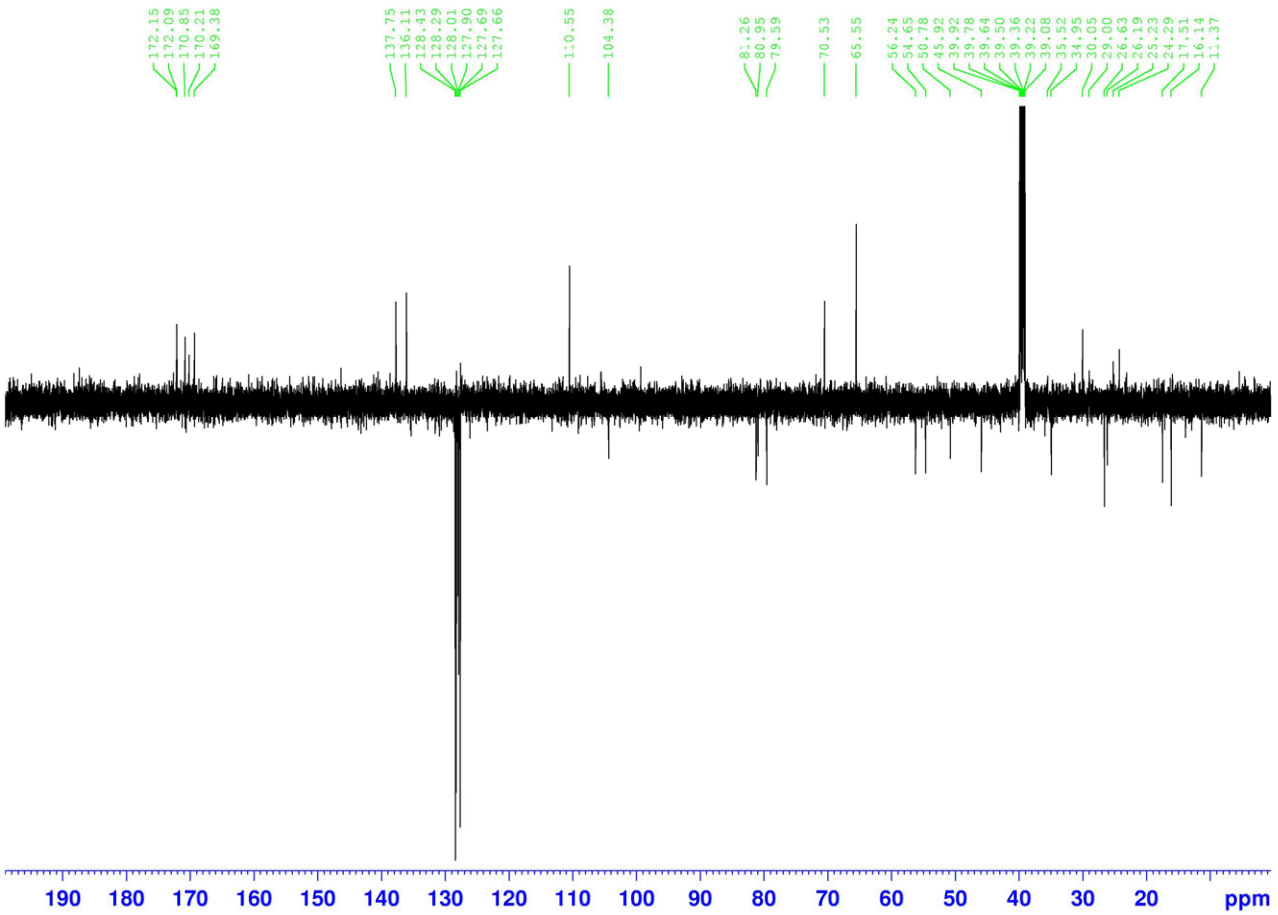


Figure 9 ROESY spectrum of **2b**

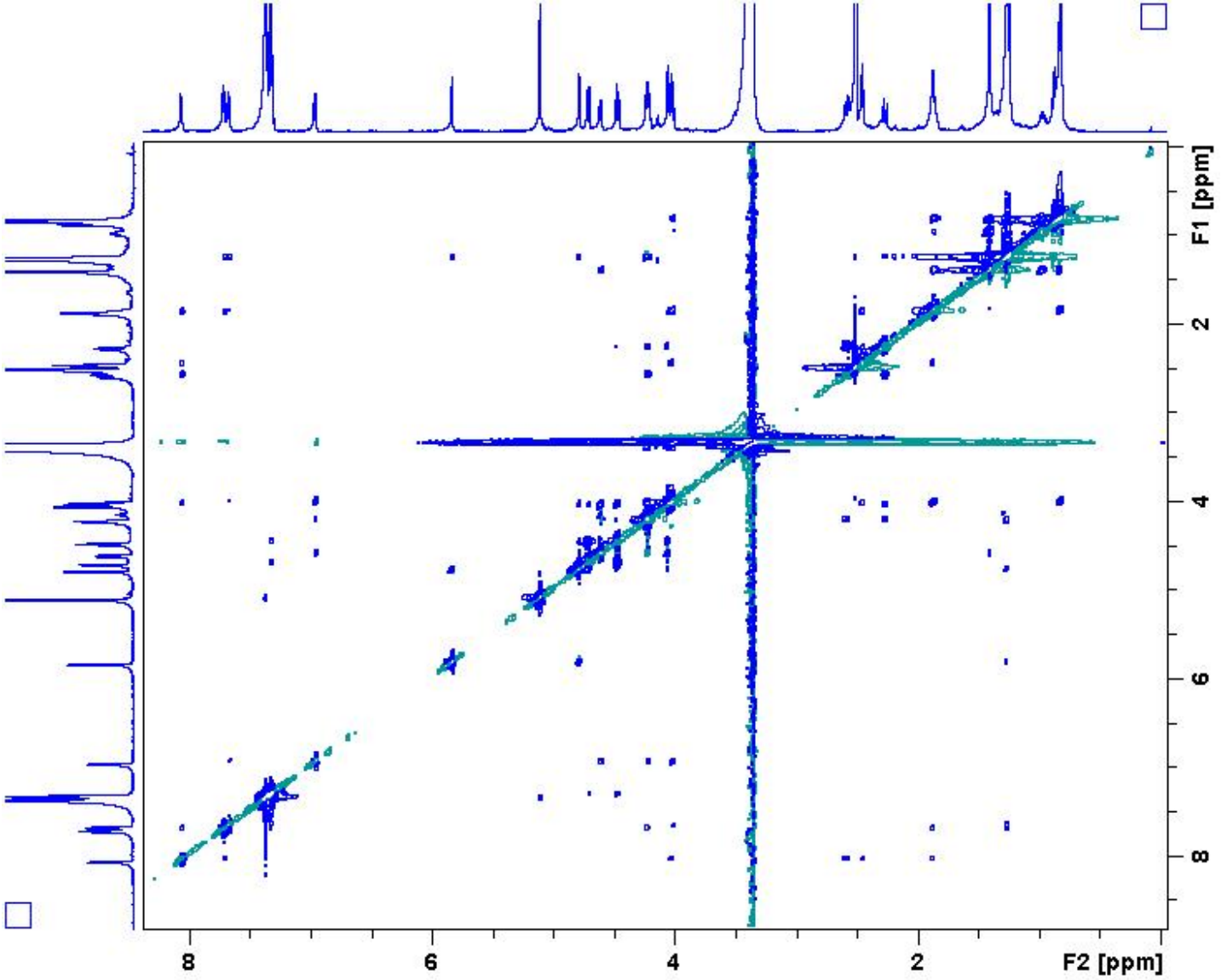


Figure 10 COSY spectrum of **2b**

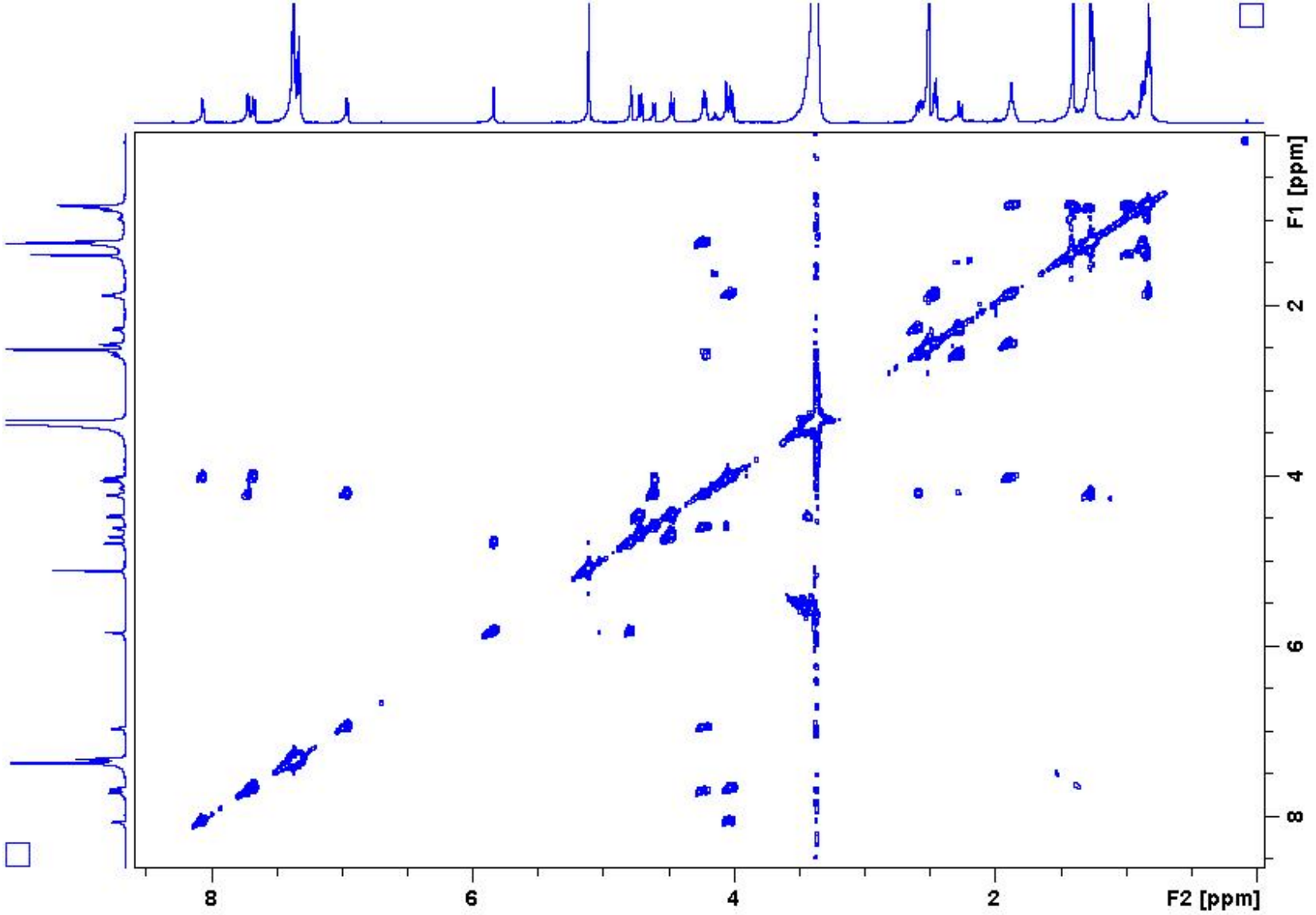


Figure 11 HSQC spectrum of **2b**

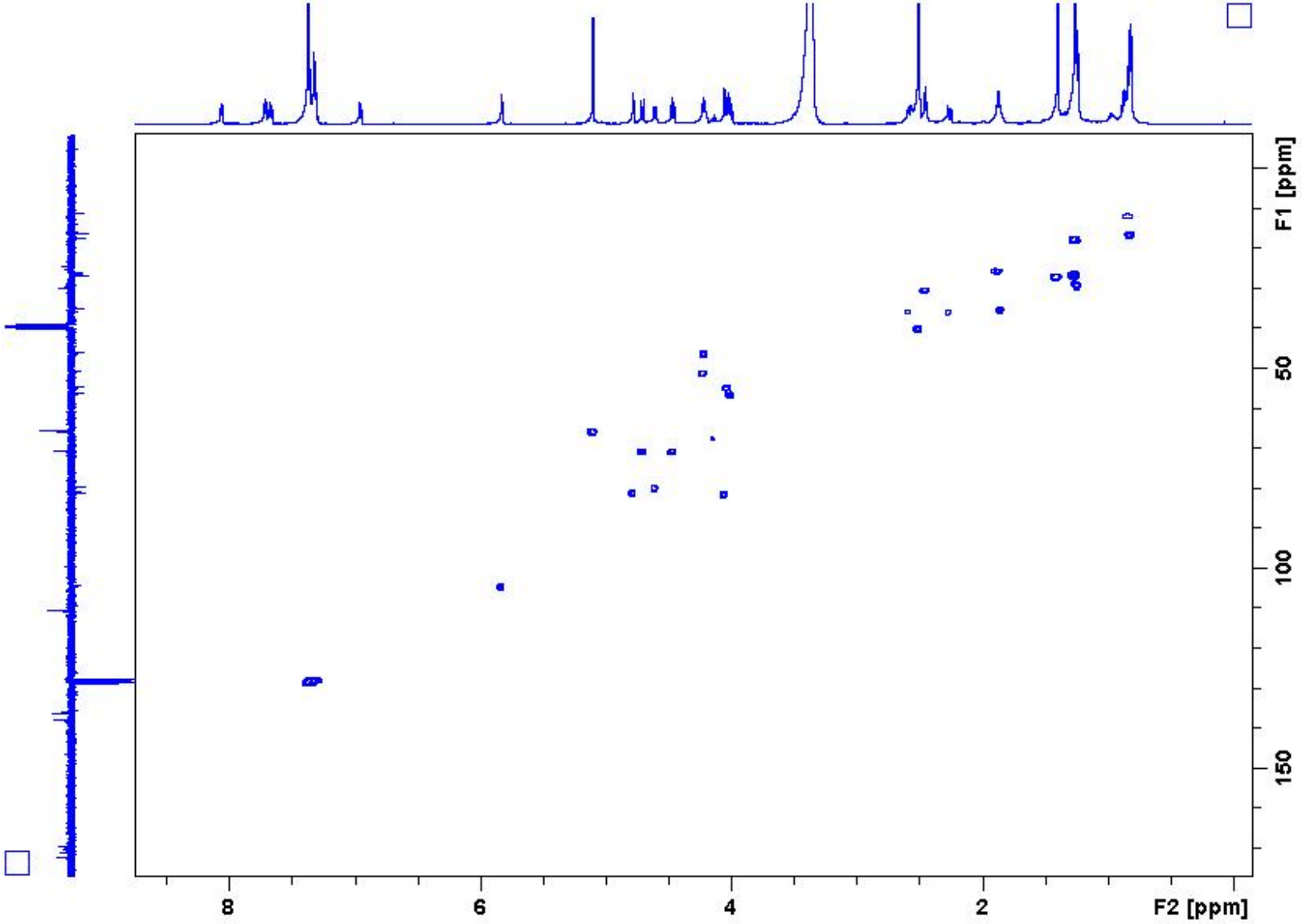


Figure 12 HMBC spectrum of **2b**

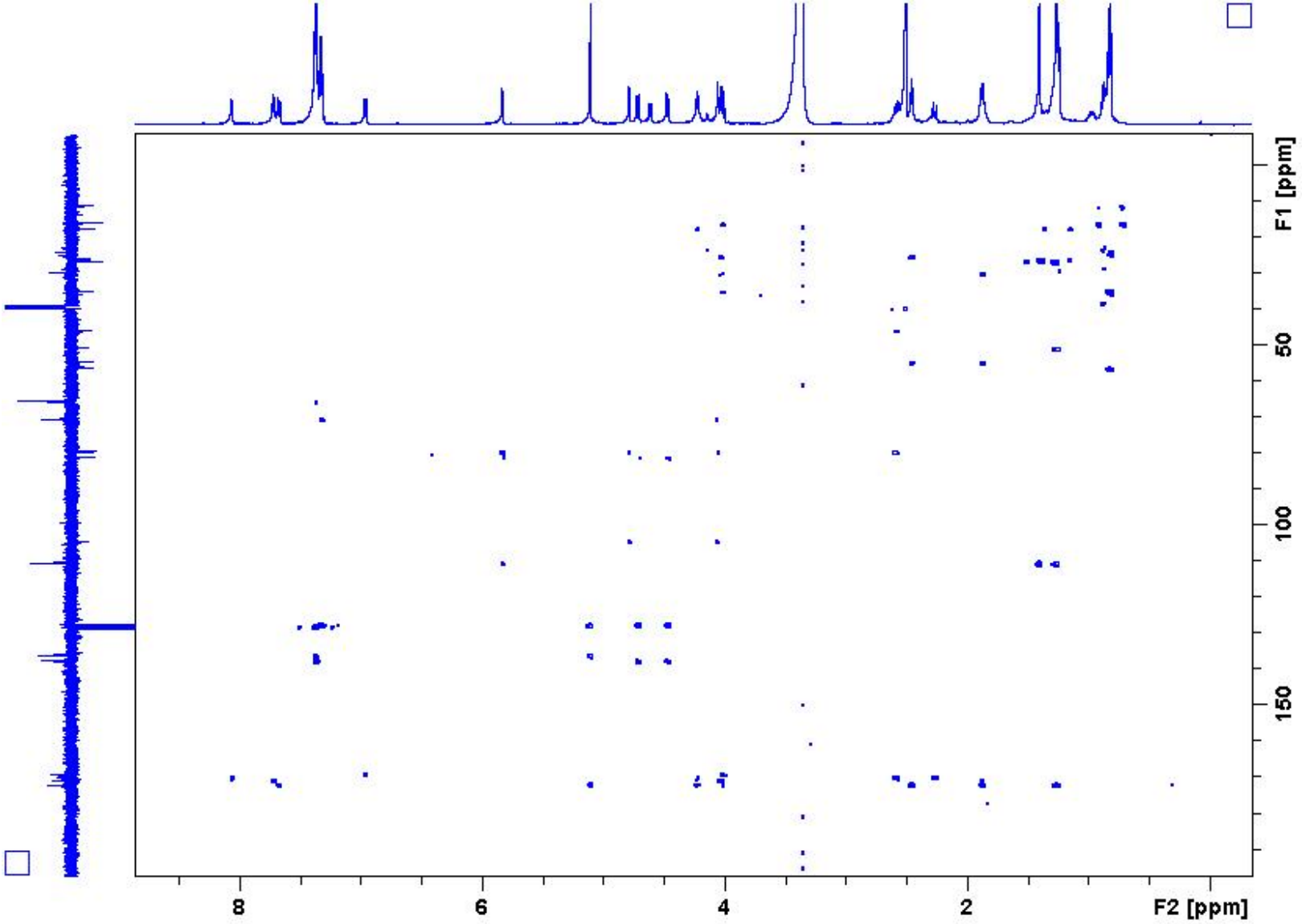


Figure 23 ^{1}H NMR spectrum of **3**

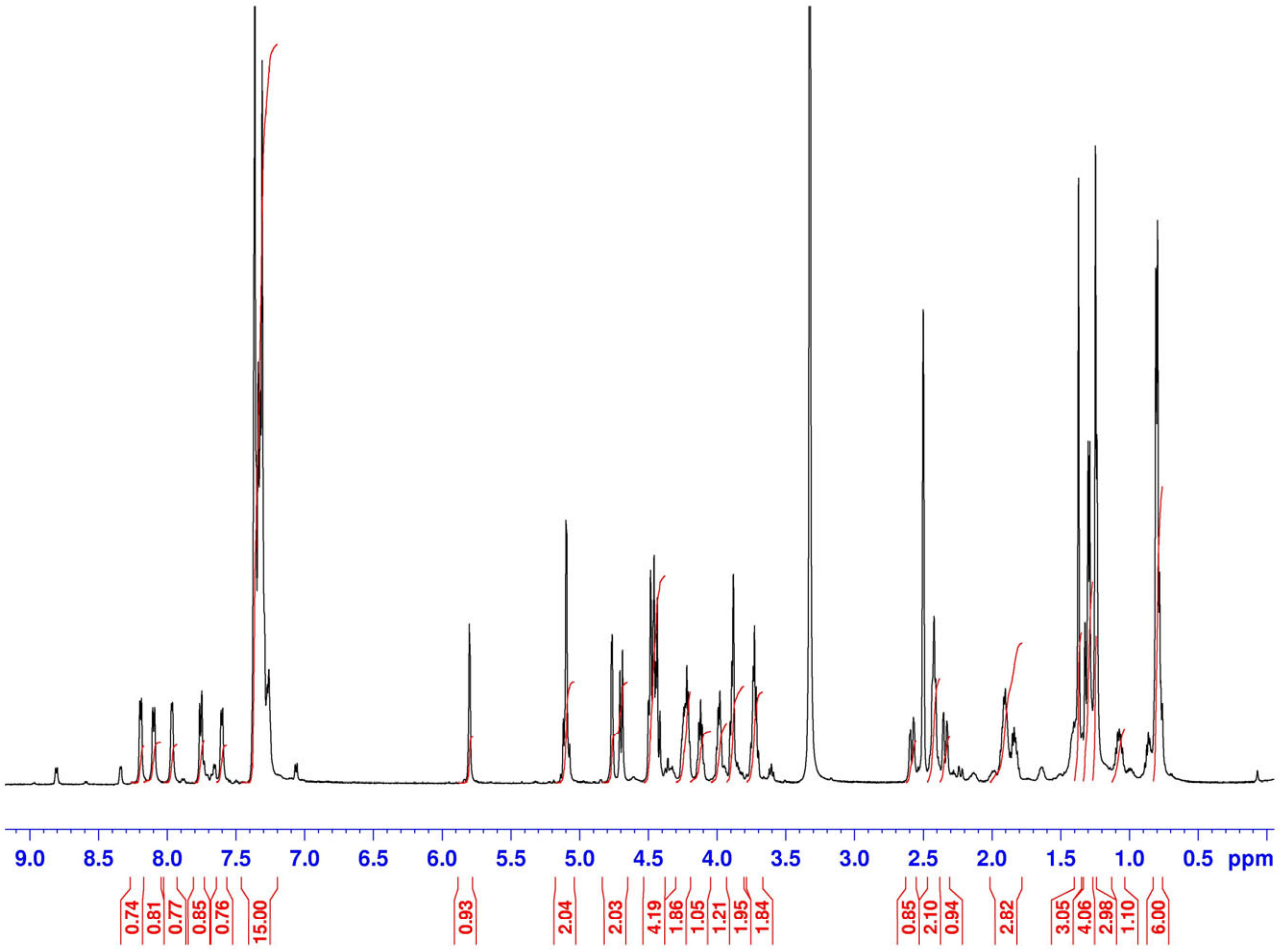


Figure 14 ^{13}C NMR spectrum of **3**

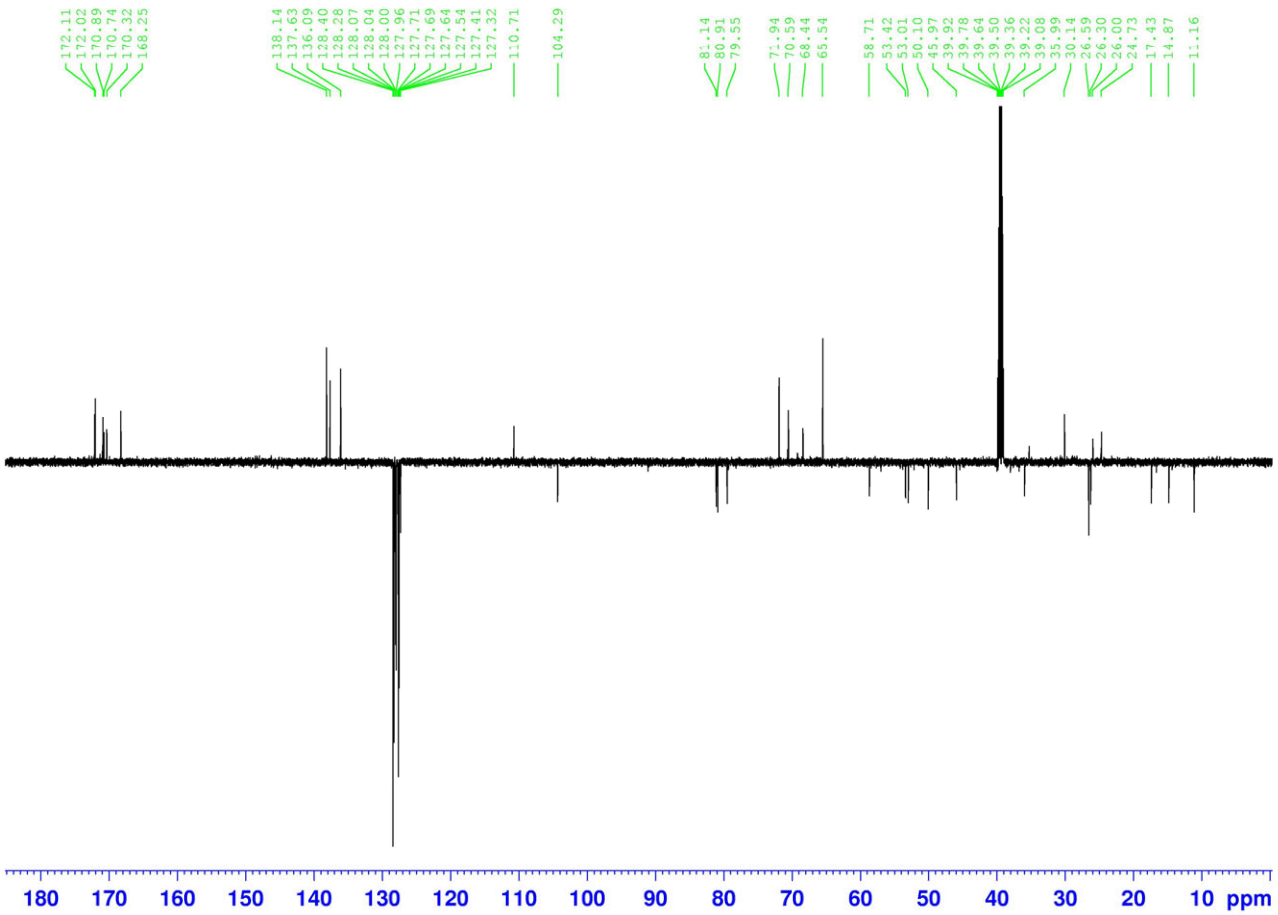


Figure 15 ROESY spectrum of **3**

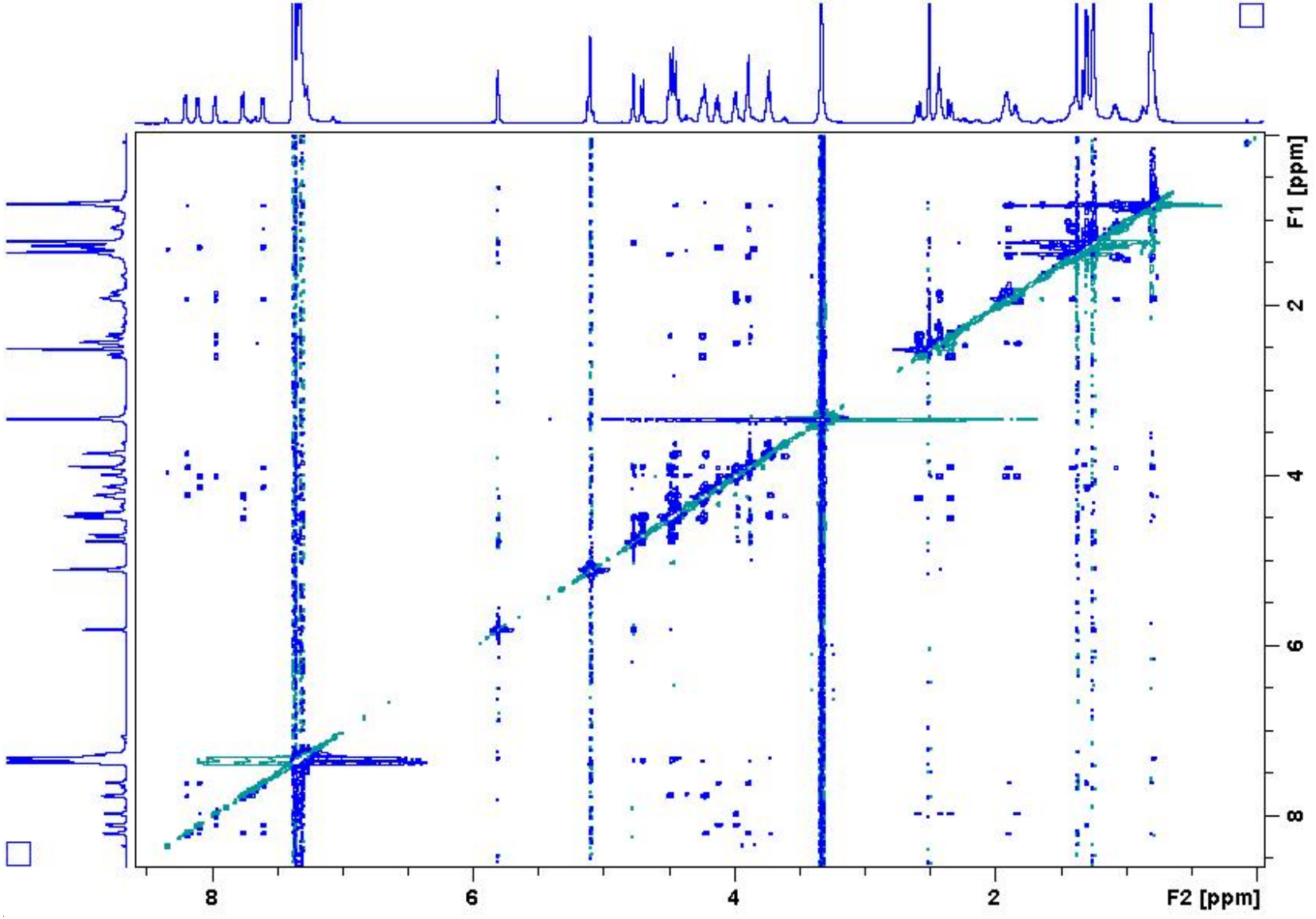


Figure 16 COSY spectrum of **3**

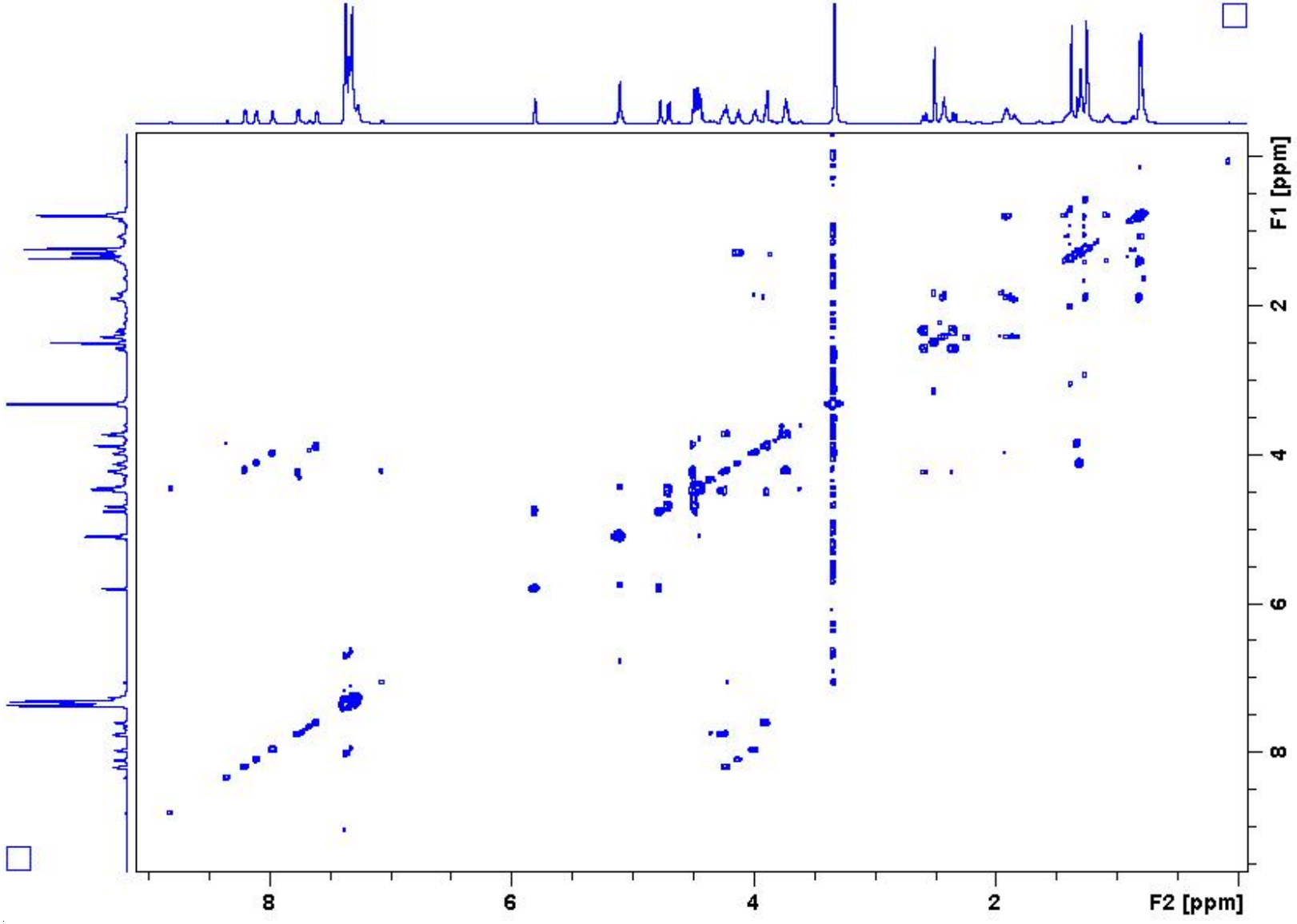


Figure 17 HSQC spectrum of **3**

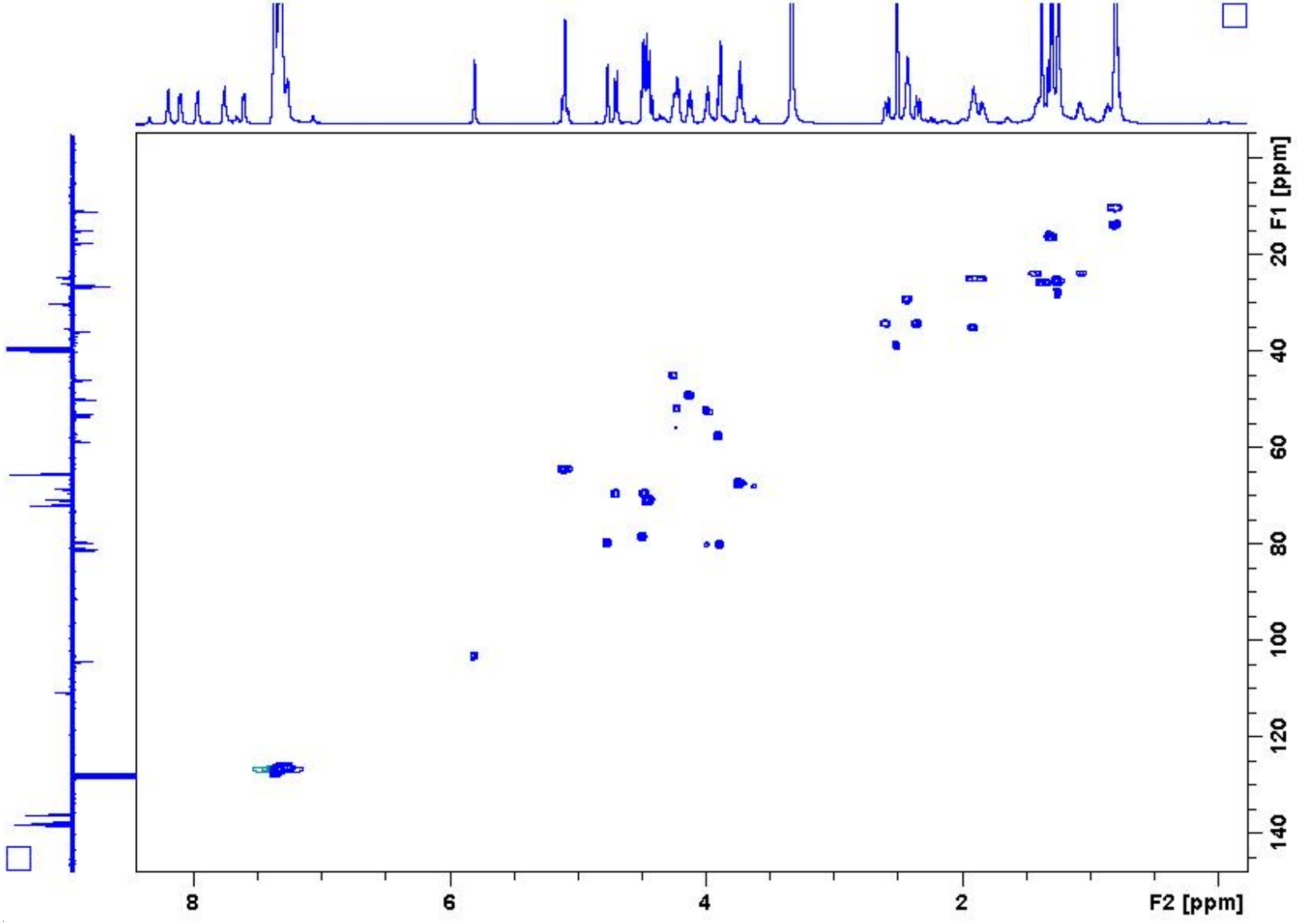


Figure 18 HMBC spectrum of **3**

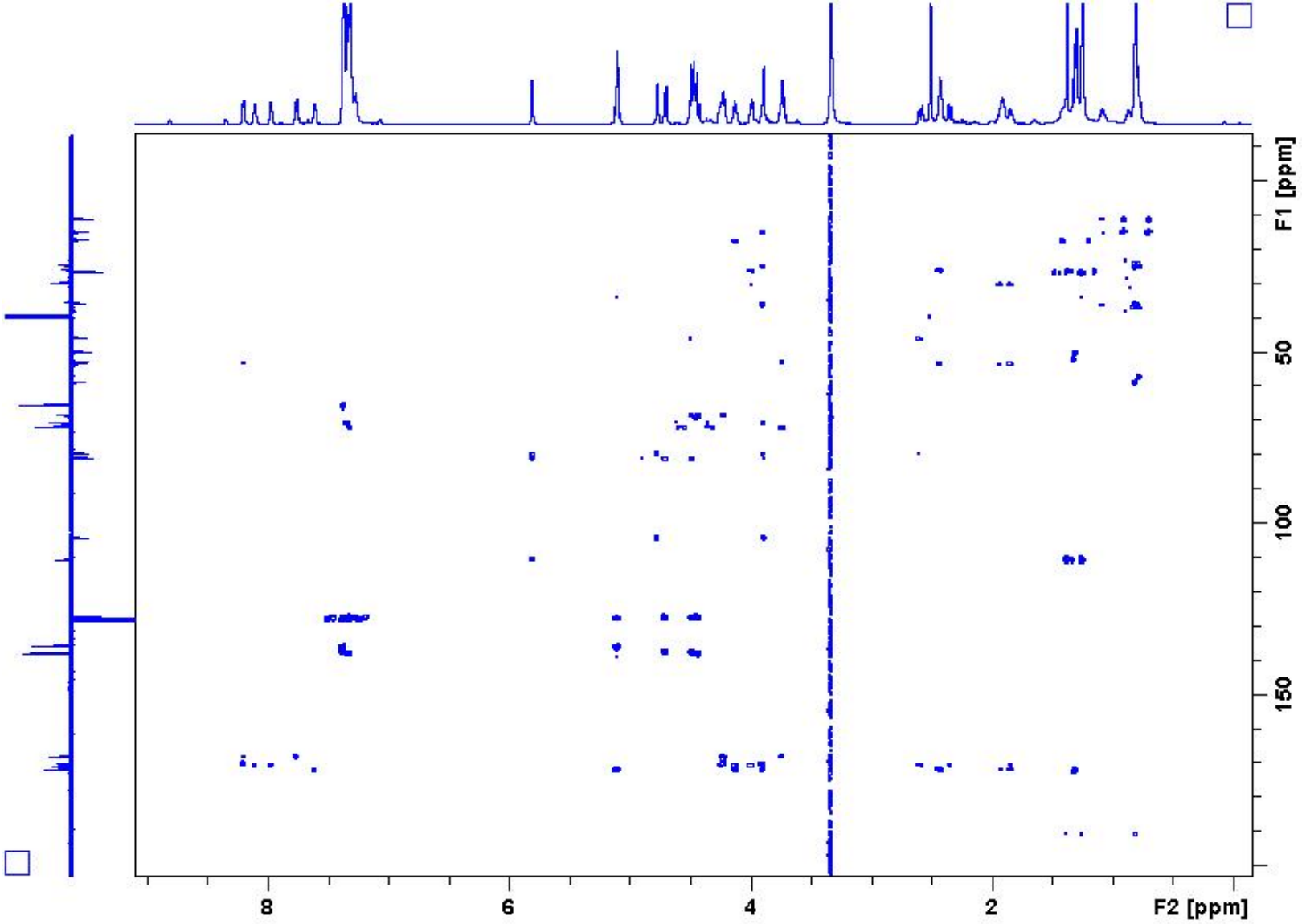


Figure 19 ^{1}H NMR spectrum of **4**

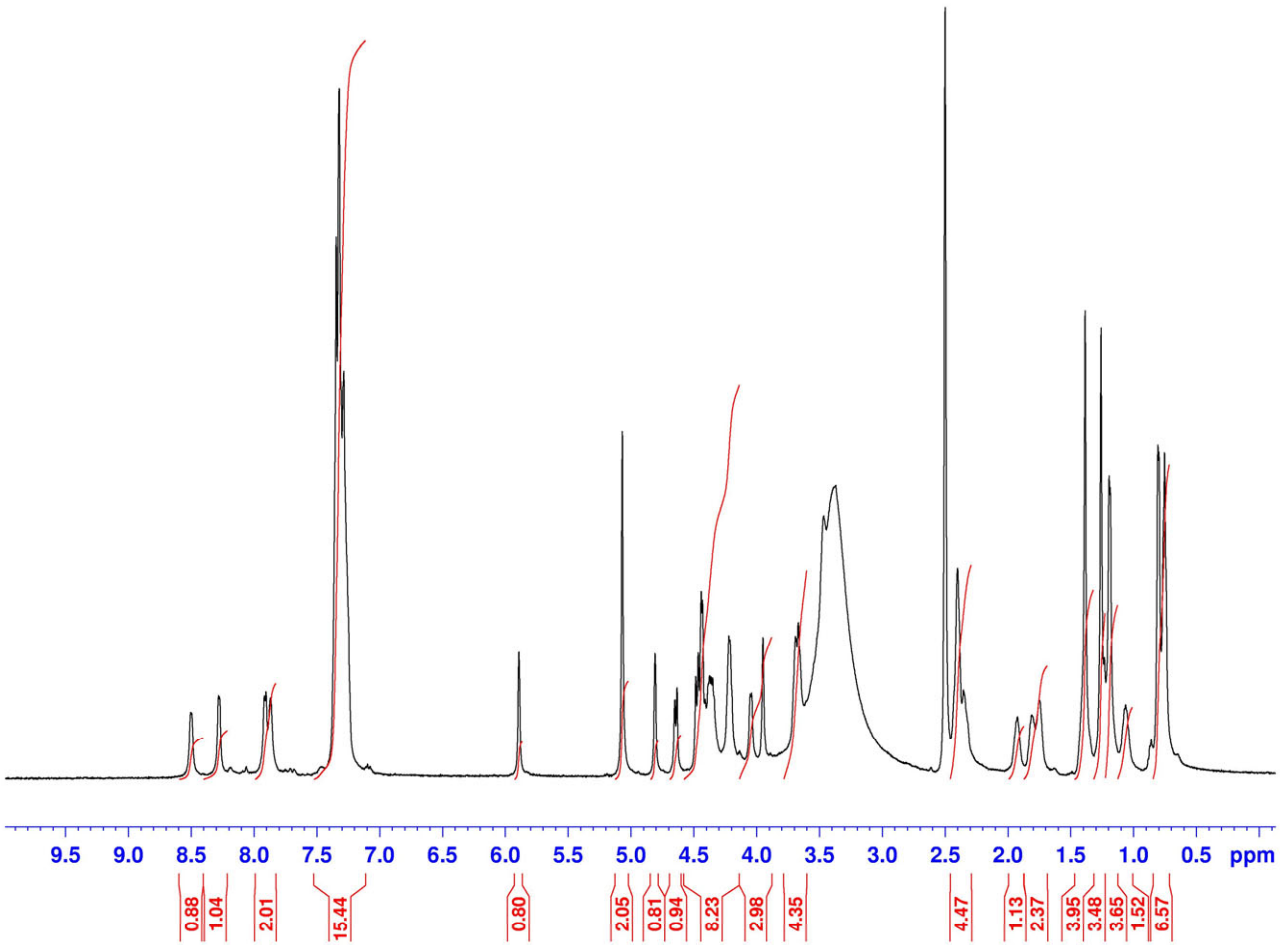


Figure 20 ^{13}C NMR spectrum of **4**

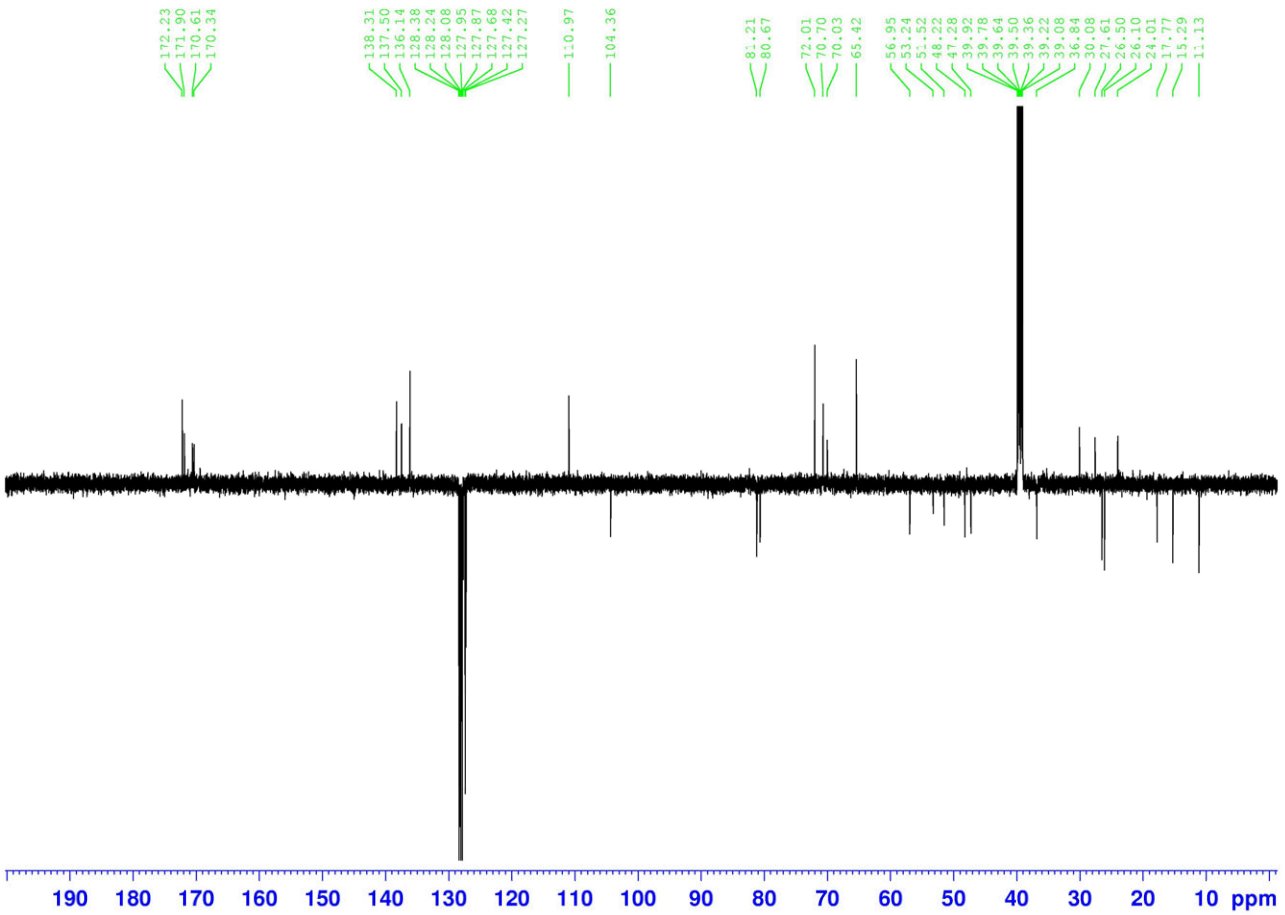
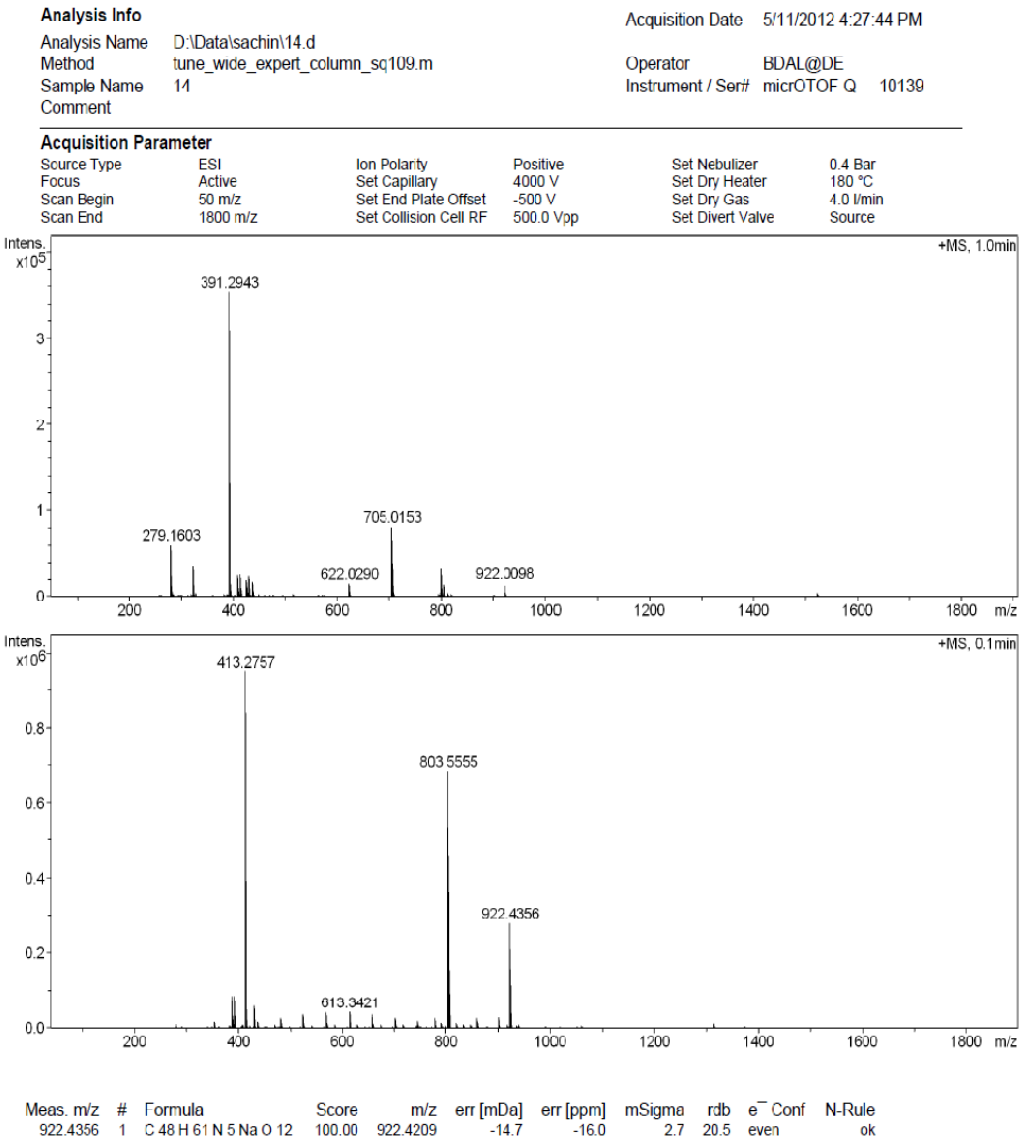


Figure 21 HRMS spectrum of **3**



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule
922.4356	1	C 48 H 61 N 5 Na O 12	100.00	922.4209	-14.7	-16.0	2.7	20.5	even	ok

Figure 22 HRMS spectrum of **4**