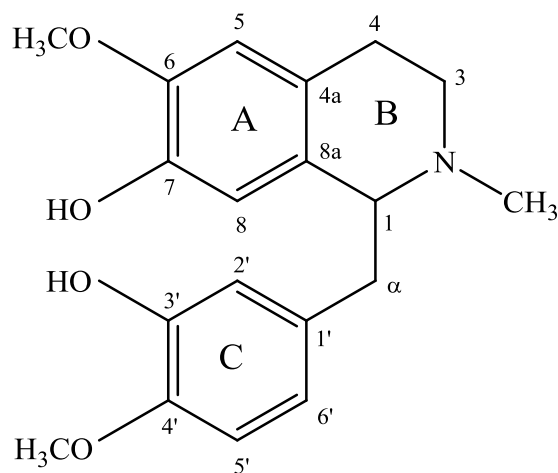


3.2.5 Alkaloid CD5: Reticuline 14



14

Alkaloid **CD5** was afforded as a brownish amorphous solid. The UV spectrum showed absorption band at 293 nm which was a characteristic of a benzylisoquinoline alkaloid⁵⁸. The IR spectrum showed strong absorptions at 3350 and 2936 cm⁻¹ due to the stretching of hydroxyl group and C-H aromatic respectively. ESI (positive mode) mass spectrum gave a pseudomolecular ion peak, [M+H]⁺ at m/z 329.2 consistent with the molecular formula of C₁₉H₂₃NO₄, with nine degrees of unsaturation.

The ¹H NMR spectrum (Figure 3.26) demonstrated two methoxyl groups overlapped to each other at δ 3.84, corresponded to 6-OMe and 4'-OMe. In addition, there were five aromatic protons appeared at δ 6.75 (*d*, *J* = 1.96 Hz, 1H, H-2'), δ 6.71 (*d*, *J* = 8.32 Hz, 1H, H-5'), δ 6.57 (*dd*, *J*₁ = 8.04 Hz, *J*₂ = 1.96 Hz, 1H, H-6'), δ 6.52 (*s*, 1H, H-5) and δ 6.38 (*s*, 1H, H-8). H-8 was shielded compared to H-5 because of the anisotropic effect caused by ring C (facing the ring C). Furthermore, the presence of *N*-methyl proton was observed as a singlet at δ 2.43. A total of seven proton signals were observed at a higher region between δ 2.59-3.17 attributable to the aliphatic protons of

H- α , H-4 and H-3. The complete assignments for the proton and carbon signals are tabulated in Table 3.5.

The ^{13}C NMR (Figure 3.27) spectrum established the presence of nineteen carbons which is in agreement with the molecular formula of reticuline. The DEPT experiment showed three methyls, three methylenes, six methines and seven quaternary carbons in the skeleton. The COSY spectrum (Figure 3.28) showed that H-5' was only correlated with H-6' while in the shielded area, H- α only correlated with H- α '.

In HMBC spectrum (Figure 3.29) the cross-peaks of the proton at δ 6.52 (H-5) correlated with δ 24.93 (C-4), δ 125.15 (C-8a) and δ 143.45 (C-7) while the proton at δ 6.38 (H-8) correlated with δ 64.60 (C-1), δ 145.10 (C-6) and 129.99 (C-4a). C-7 and C-3' bearing hydroxyl groups were observed at δ 143.45 and δ 145.23 respectively through the long range coupling of H-5 (δ 6.52) to C-7 (δ 143.45) and H-5' (δ 6.71) to C-3' (δ 145.23).

Finally, unambiguous assignment of all proton and carbon signals using DEPT, HMQC and COSY and by comparison with literature data⁹⁵⁻⁹⁹ showed that the alkaloid **CD5** was reticuline **14**.

Table 3.5: ¹H NMR (in CDCl₃, 400 MHz) and ¹³C NMR (in CDCl₃, 400 MHz) of **14**

Position	δ_{H} , ppm (J in Hz)	δ_{C} (ppm)	HMBC (H→C)	HMQC
1	3.63-3.66 (<i>m</i>)	64.60	α , 1', 3, 4a, 8a	H-1
3	2.69-3.18 (<i>m</i>)	46.75	4a	H-3
4	2.53-2.82 (<i>m</i>)	24.93	8a	H-4
4a	-	129.99	-	-
5	6.52 (<i>s</i>)	110.50	4, 8a, 7	H-5
6	-	145.10	-	-
7	-	143.45	-	-
8	6.38 (<i>s</i>)	113.74	1, 6, 4a	H-8
8a	-	125.15	-	-
α	2.69-3.02 (<i>m</i>)	41.03	8a, 2', 6', 1', 1	H- α
1'		133.06	-	
2'	6.75 (<i>d</i> , 1.96)	115.65	α , 3', 6'	H-2'
3'	-	145.23		
4'	-	145.36		-
5'	6.71 (<i>d</i> , 8.32)	110.62	1', 4'	H-5'
6'	6.57 (<i>dd</i> , 1.96, 8.04)	121.01	α , 2', 4'	H-6'
4'-OMe	3.83 (<i>s</i>)	55.98	4'	-
6-OMe	3.83 (<i>s</i>)	55.98	6	-
N-Me	2.43 (<i>s</i>)	42.43	1, 3	-

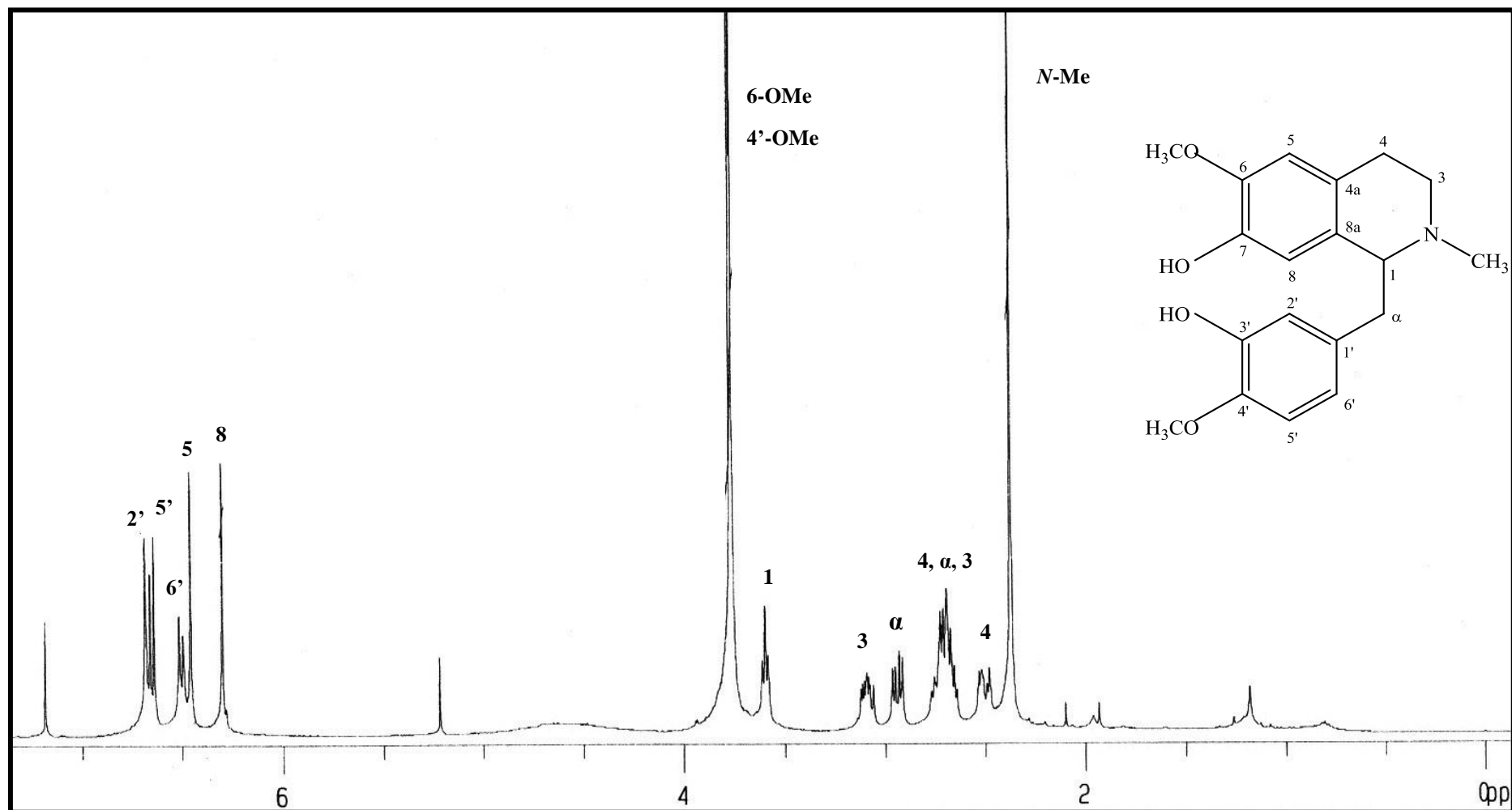


Figure 3.26: ¹H NMR spectrum of Reticuline 14

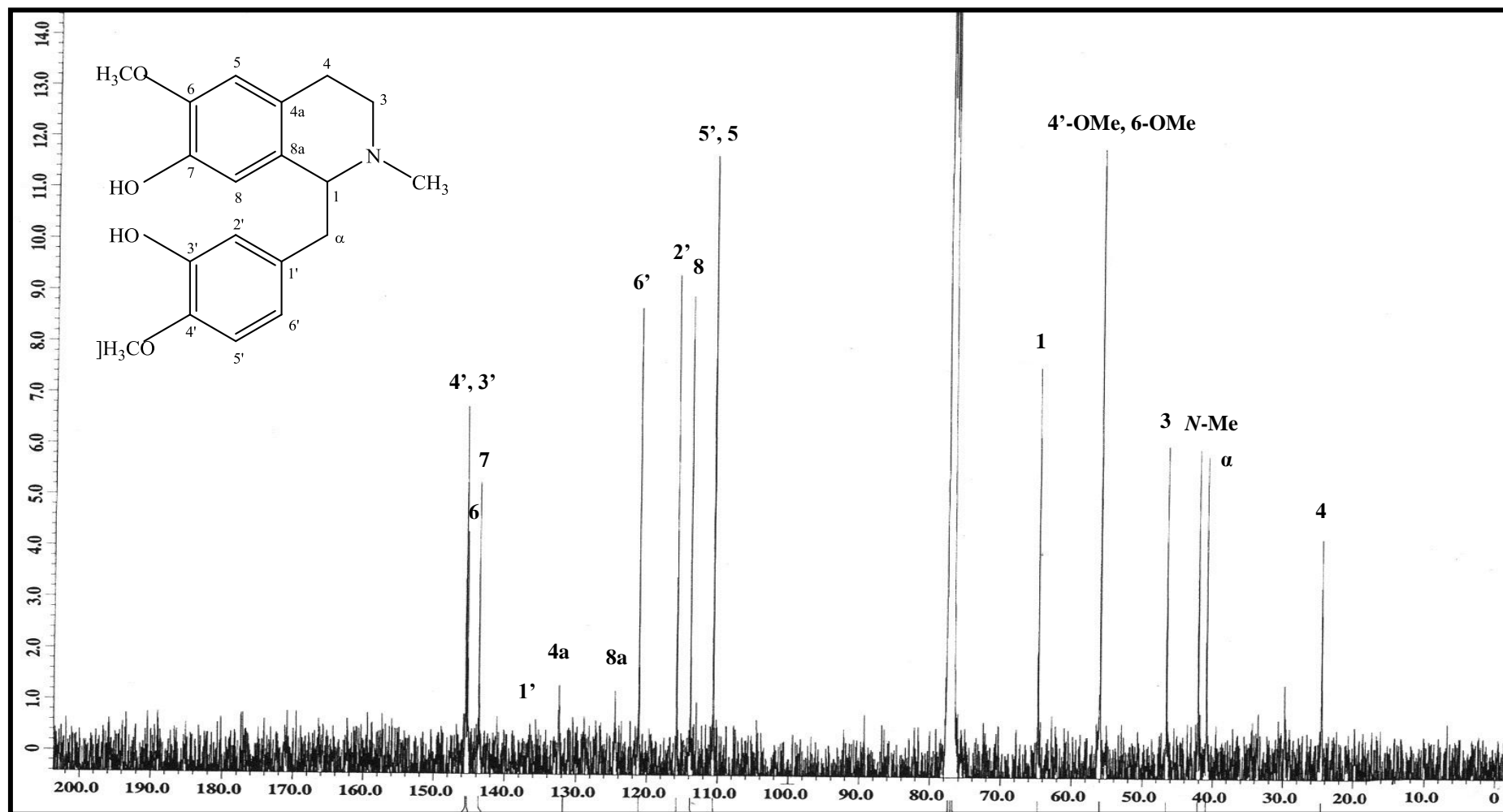


Figure 3.27: ^{13}C NMR spectrum of Reticuline 14

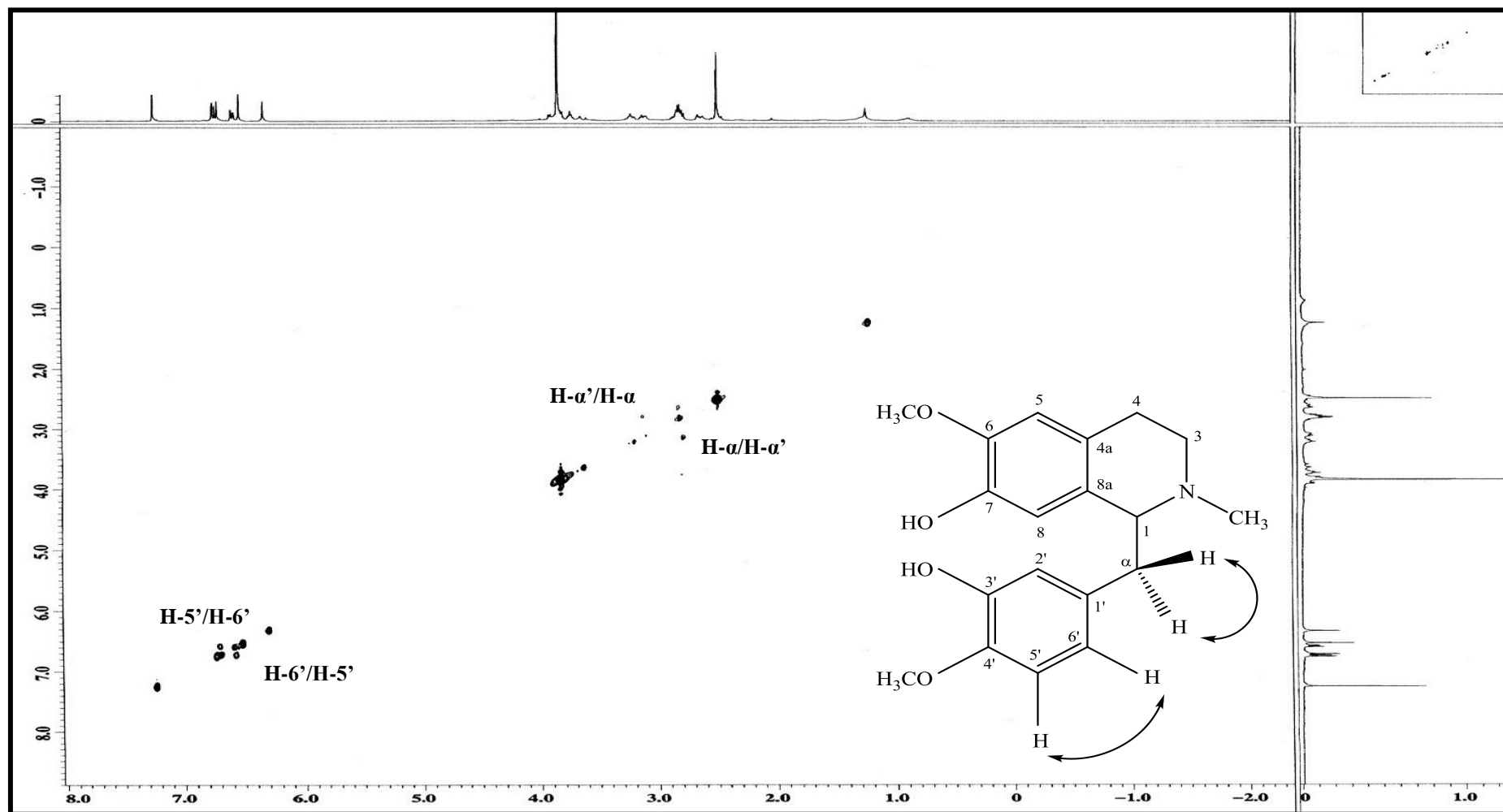


Figure 3.28: COSY spectrum of Reticuline 14

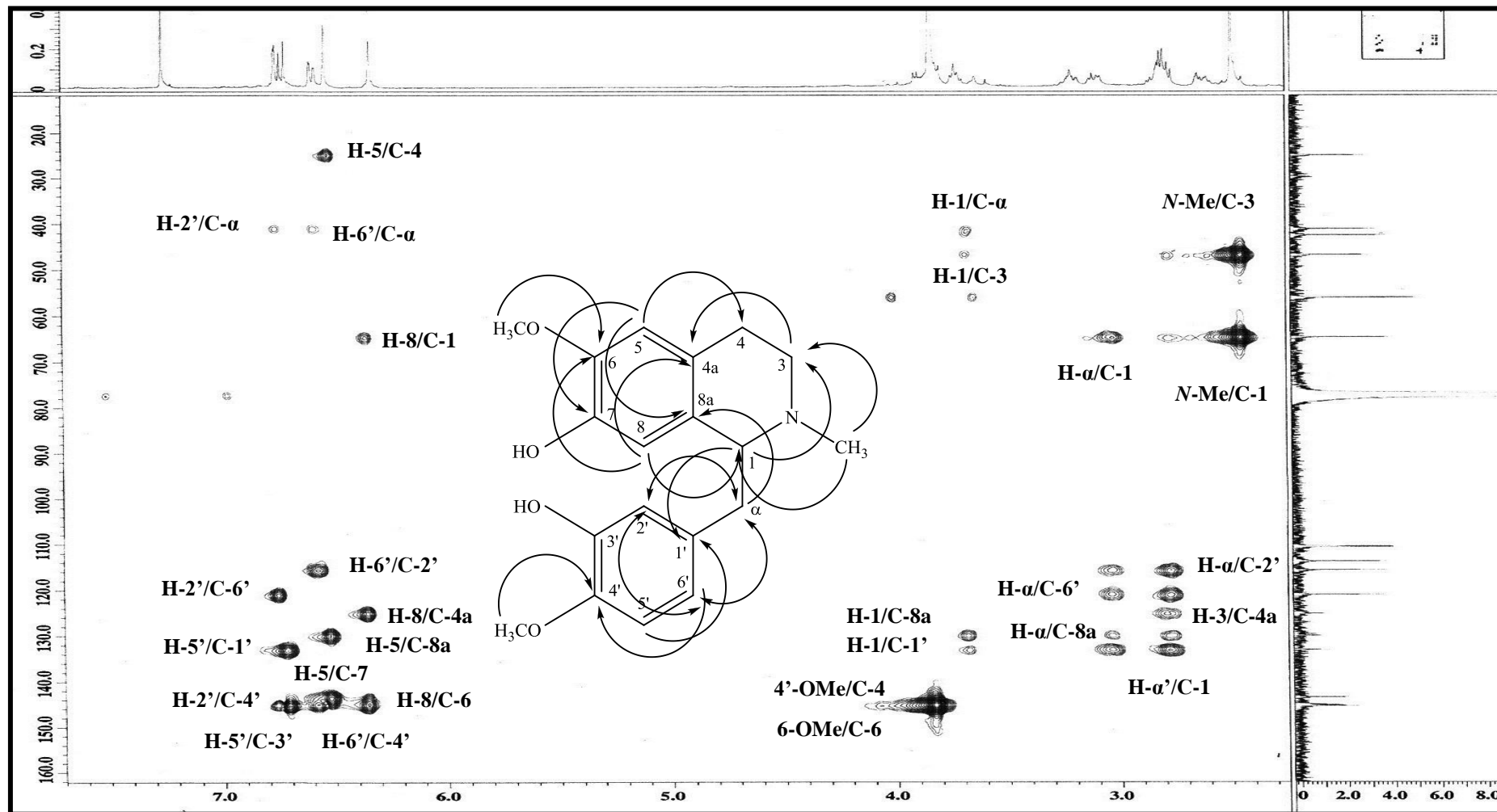


Figure 3.29: HMBC spectrum of Reticuline 14

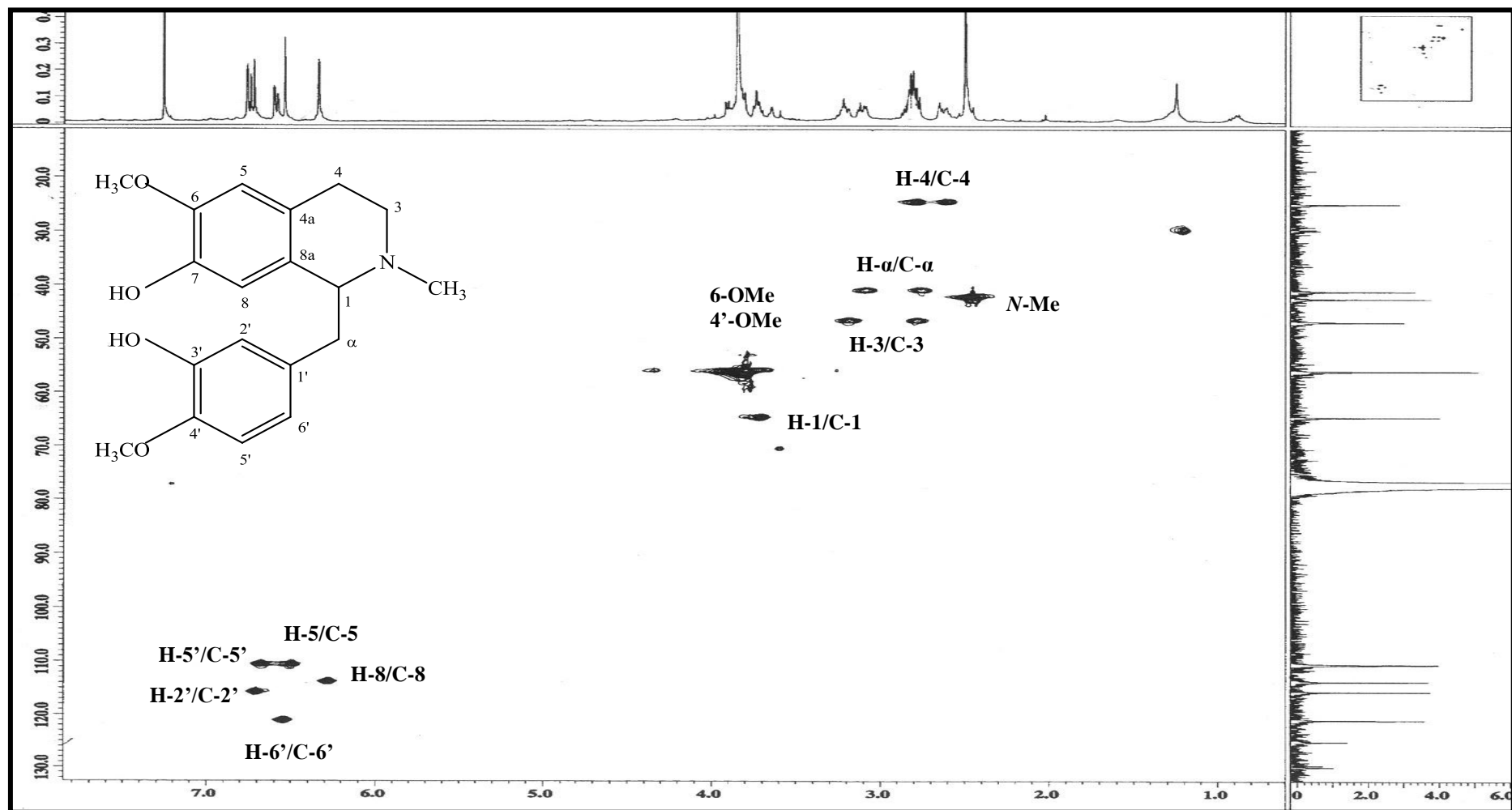


Figure 3.30: HMQC spectrum of Reticuline 14

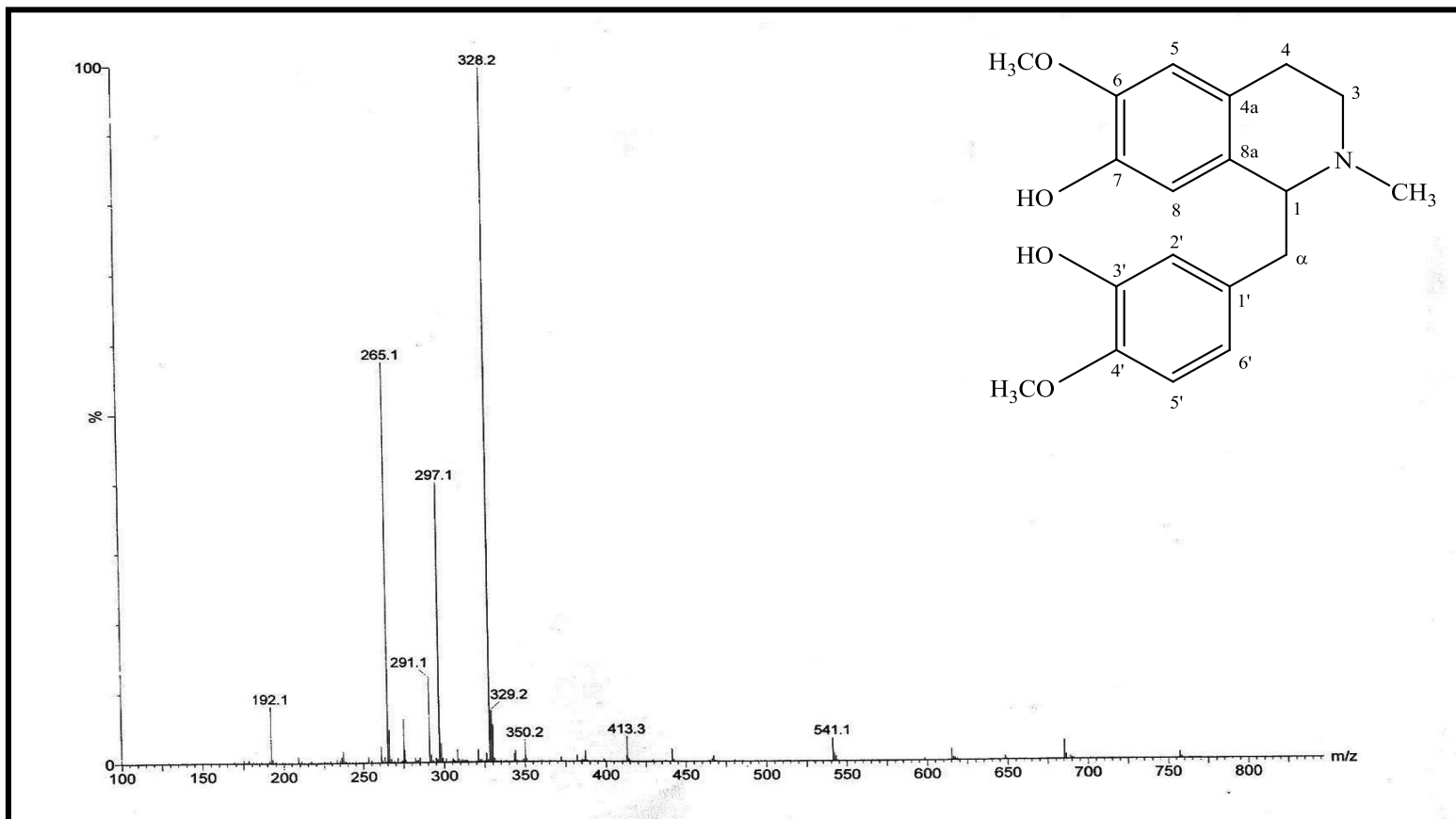


Figure 3.31: LCMS spectrum of Reticuline 14