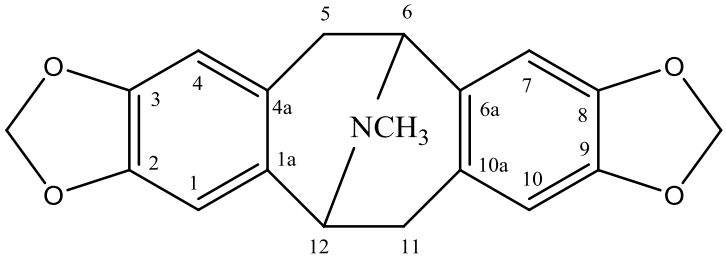
3.2.8 Alkaloid CD8: Crychine 60



60

Alkaloids **CD8** was isolated as a brownish amorphous solid. The IR spectrum showed the existence of methylenedioxy absorptions at 935.85 and 1039.51 cm^{-1}. The ESI^+ (positive mode) spectrum showed a molecular ion peak, $[M+H]^+$ at m/z 324.12 corresponding to a molecular formula of $C_{19}H_{17}NO_4$, with twelve degrees of unsaturation.

The ^1H NMR spectrum (Figure 3.36) revealed two overlapped aromatic singlets at δ 6.40 and 6.55 which could be belonged to H-4/10 and H-1/7 respectively. AMX patterns at δ 3.93 (*d*, *J* = 5.6 Hz, H-6), 3.34 (*dd*, 5.88, *J* = 16.12 Hz, H-11), 2.52 (*d*, *J* = 16.6 Hz, H-5) and methylenedioxy signals at δ 5.79, 5.84 indicated a highly symmetrical pavine structure[107].

The ^{13}C NMR spectrum (Figure 3.37) displayed nine overlapped signals and one *N*-Me. The DEPT spectrum (Figure 3.38) showed the presence of one methyl, two methylene, three methine and eight quaternary carbons. The assignments of all proton and carbon signals were confirmed by the 2D (COSY, HMQC and HMBC) spectra.

The COSY spectrum (Figure 3.39) indicated correlations between H-5/H-6, H-11/H-12, H-5α/H-5β and H-11α/H-11β. The symmetrical of this compound is clearly shown by cross-peaks seen in HMBC spectrum (Figure 3.41). For instance, H-1/H-7 correlated to C-3, C-4a, or C-9, C-10a respectively.

Finally, comparison of these spectroscopic data obtained with those of literature values[16, 107], confirmed the alkaloid **CD8** was crychine **60**.

Table 3.8 ^{1}H NMR (in $CDCl_3$, 400 MHz) and ^{13}C NMR (in $CDCl_3$, 400 MHz) of **60**

Position	$\delta_{H,}$ ppm (J in Hz)	δ_C (ppm)	COSY	HMQC	HMBC (H→C)
1	6.55 (*s*)	107.09	-	H-1	12, 4a, 3
1a	-	130.87	-	-	
2	-	146.08	-	-	
3	-	146.45	-	-	
4	6.40 (*s*)	108.73	-	H-4	1a, 2, 5
4a	-	124.94	-	-	
5	3.34 (*dd*,16.12, 5.88) 2.52 (*d*,16.6)	34.13	H-5α/H-5β	H-5	4, 6, 4a, 1a
6	3.93 (*d* 5.6)	56.74	H-6/H-5	H-6	5, 7, 6a, 12, 4a
6a	-	130.87	-	-	
7	6.55 (*s*)	107.09	-	H-7	6, 10a, 9
8	-	146.08	-	-	
9	-	146.45	-	-	
10	6.40 (*s*)	108.73	-	H-10	11, 6a, 8
10a	-	124.94	-	-	
11	3.34 (*dd*,16.12, 5.88) 2.52 (*d*,16.6)	34.13	H-11α/H-11β	H-11	10, 12, 10a, 6a
12	3.93 (*d*, 5.6)	56.74	H-12/H-11	H-12	1, 11, 10a, 6, 1a
6, 12-*N*-Me	2.49 (*s*)	40.87	-	-	
2/3 and 8/9-OCH_2O	5.79 (*d*, 1.20) 5.84 (*d*, 1.20)	100.68	-	-	

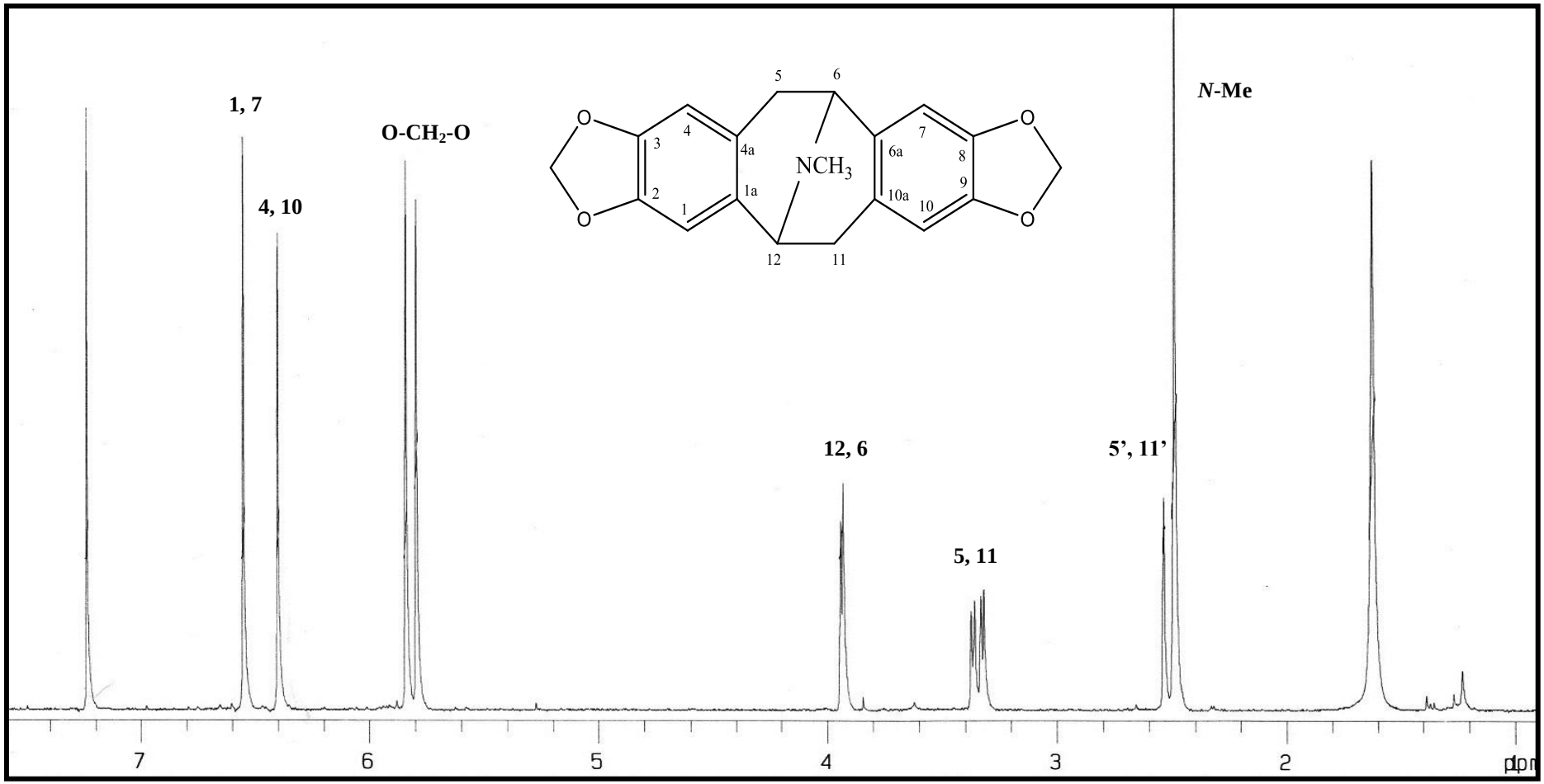


Figure 3.36: ^{1}H NMR Spectrum of Crychine **60**

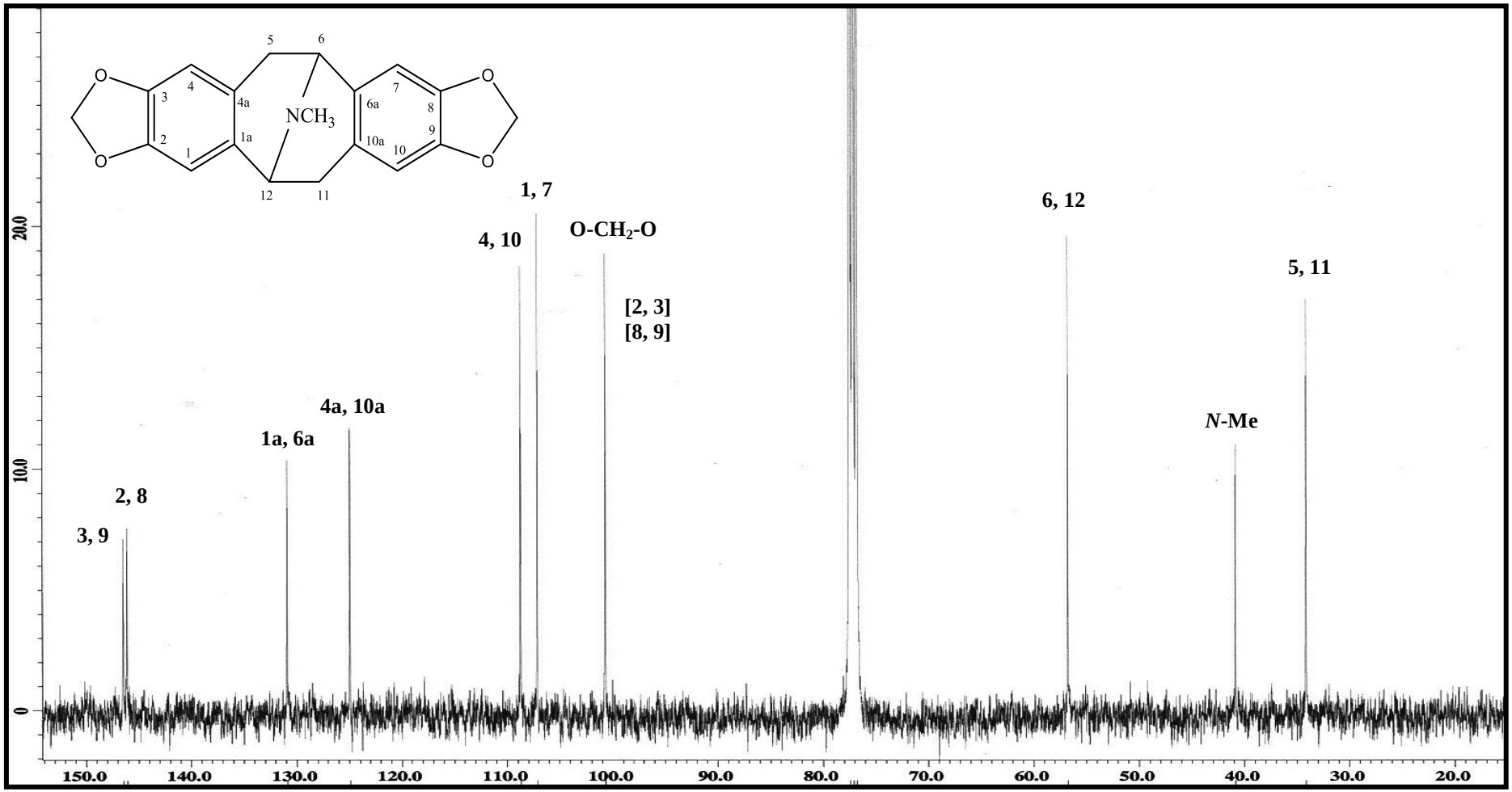


Figure 3.37: ^{13}C NMR Spectrum of Crychine **60**

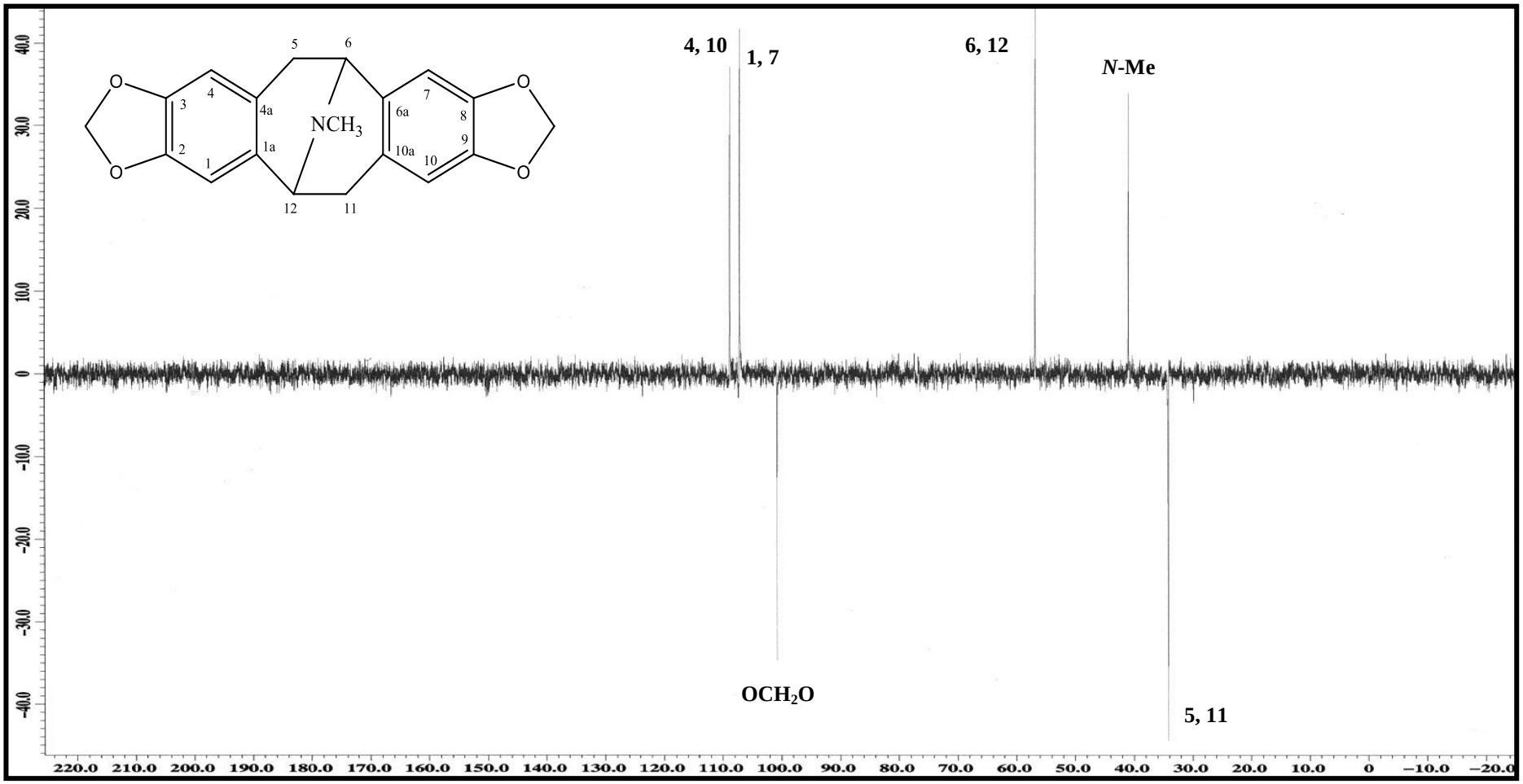


Figure 3.38: DEPT Spectrum of Crychine **60**

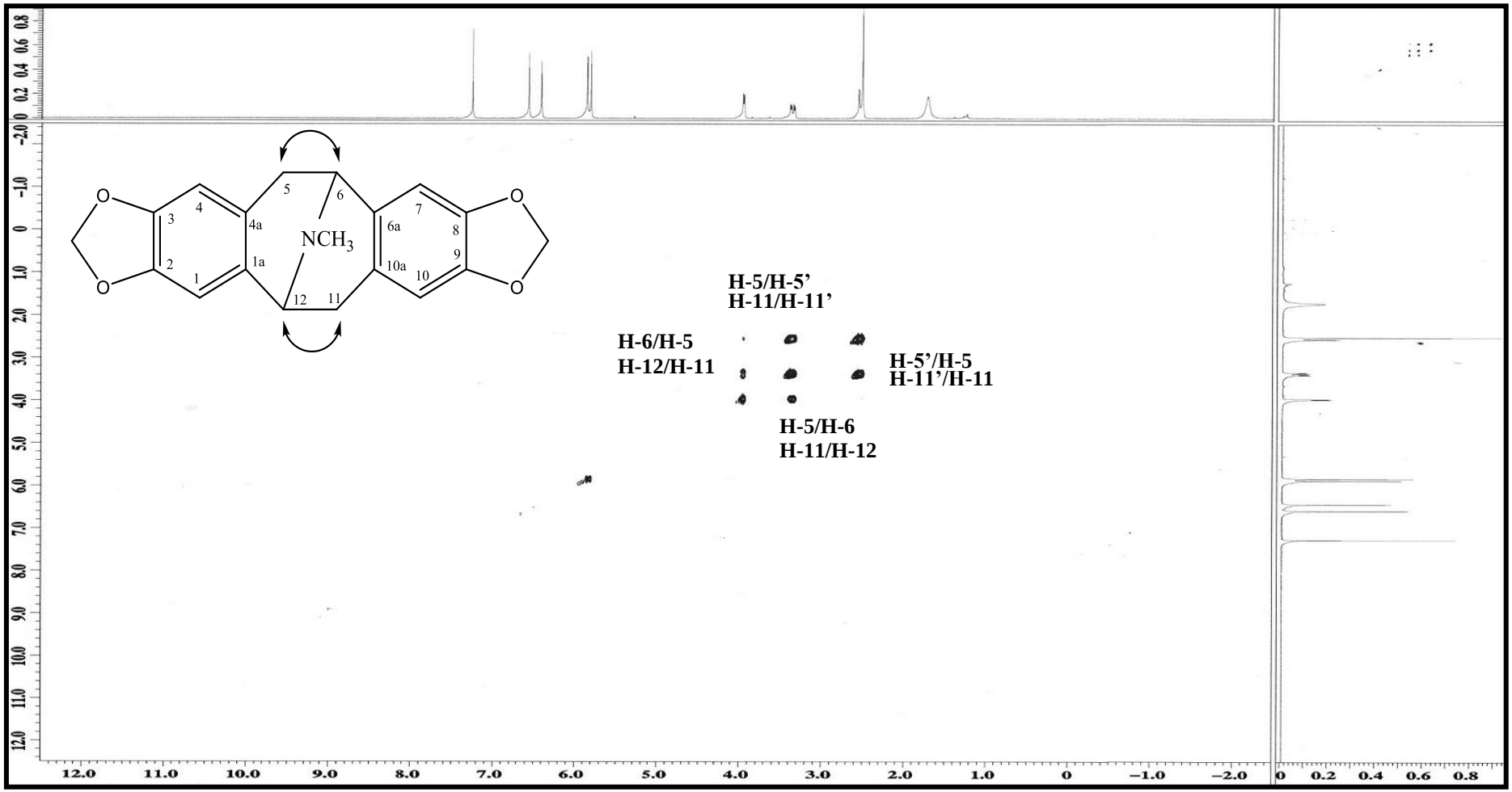


Figure 3.39: COSY Spectrum of Crychine **60**

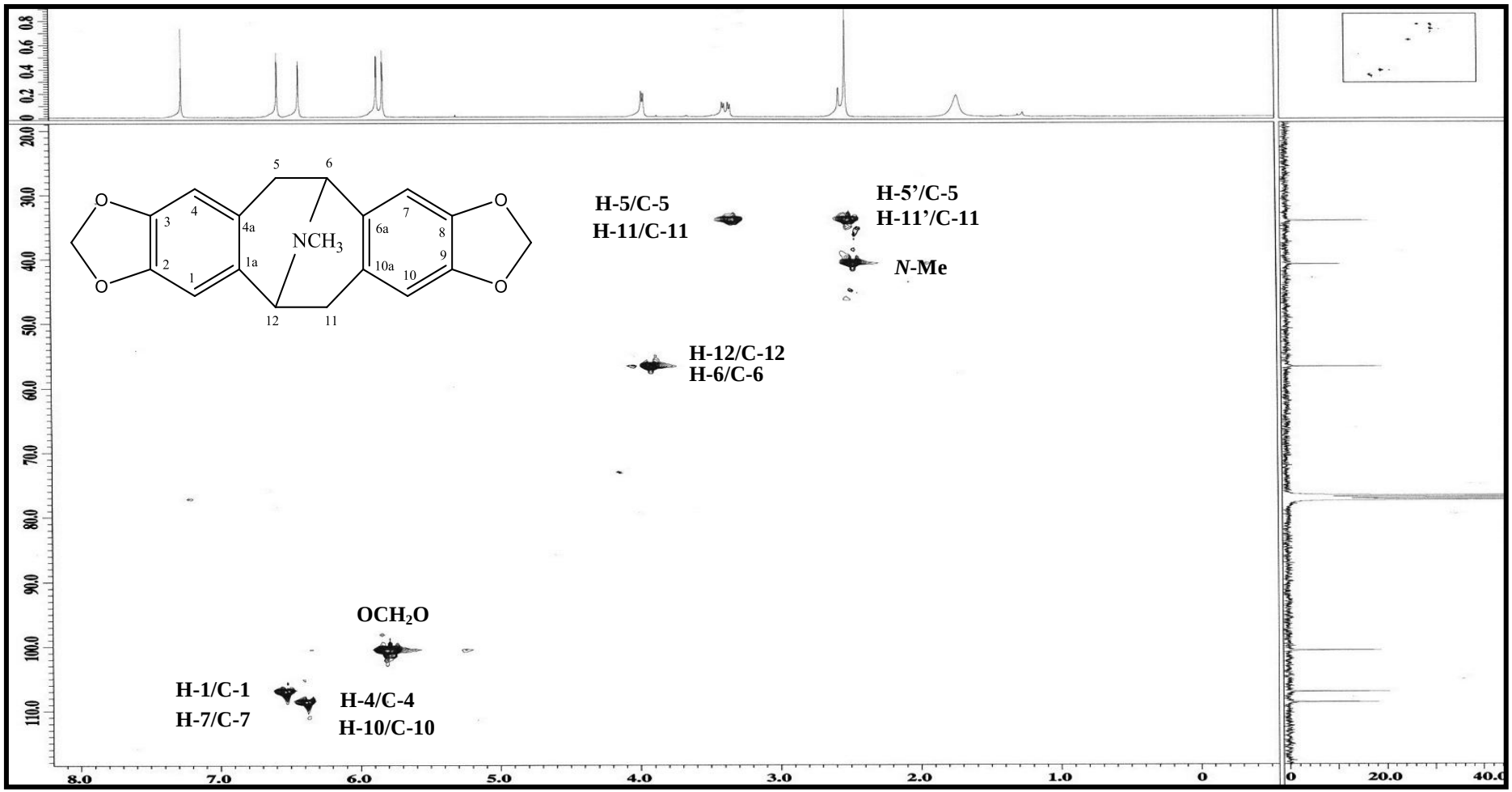


Figure 3.40: HMQC Spectrum of Crychine **60**

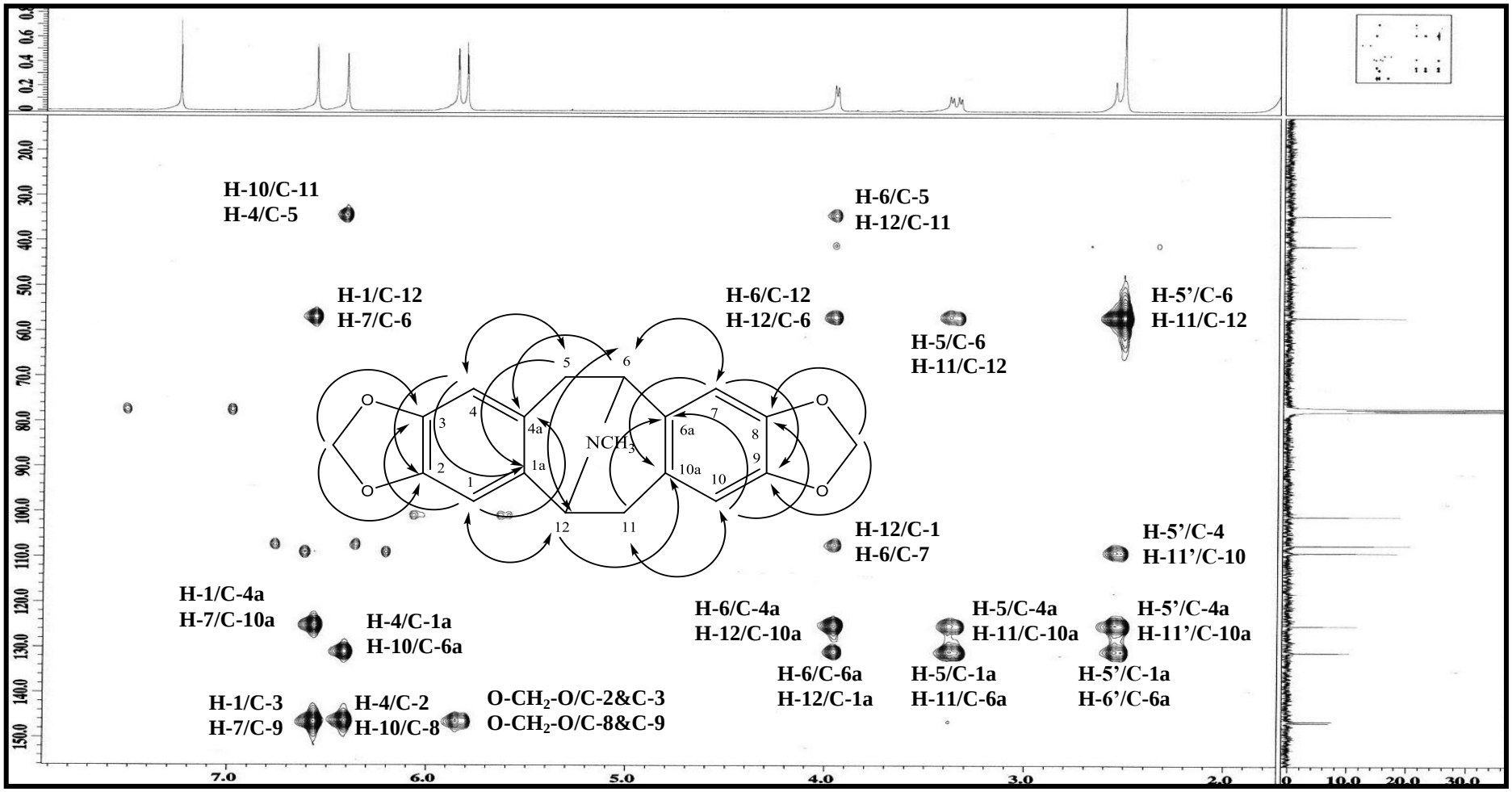


Figure 3.41: HMBC Spectrum of Crychine **60**

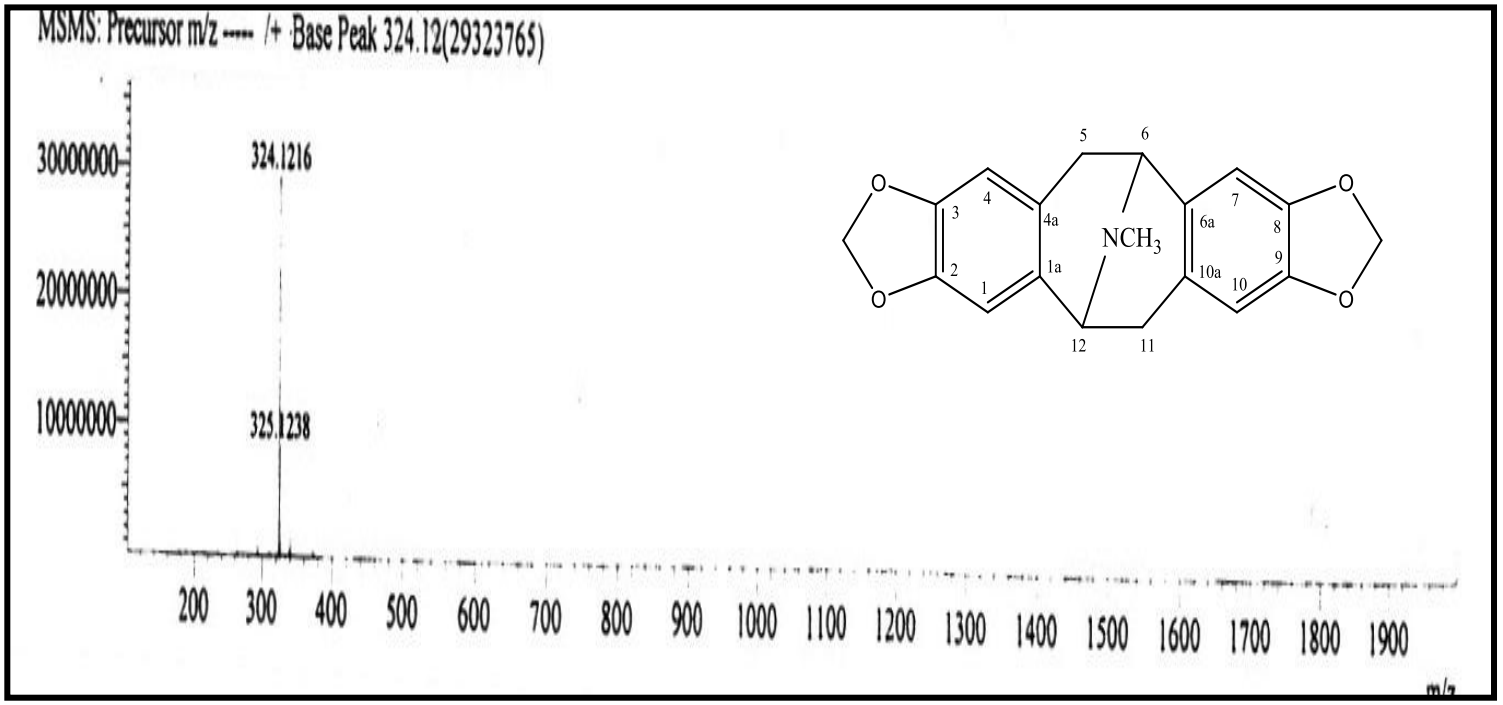


Figure 3.42: LCMS Spectrum of Crychine **60**

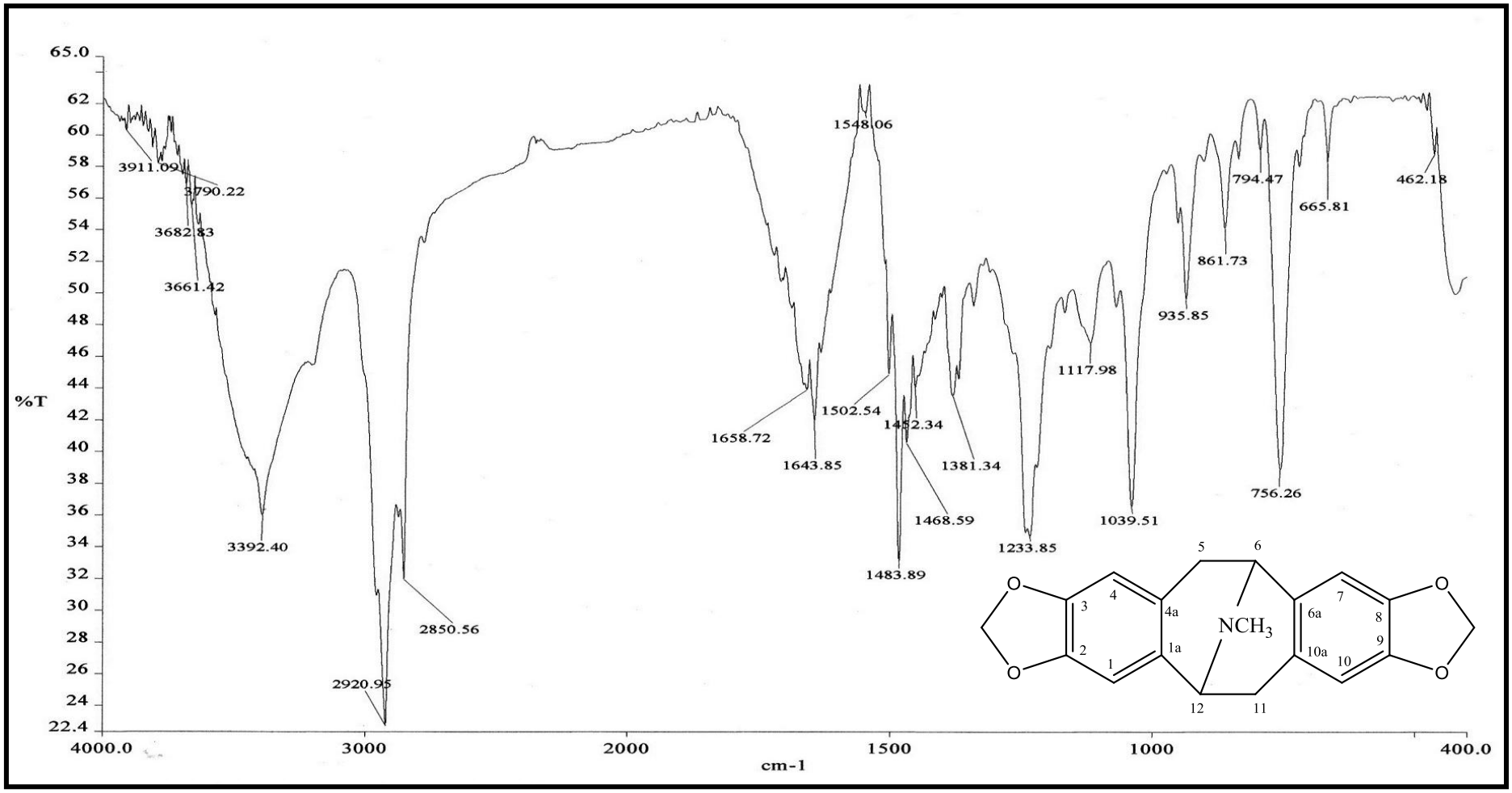


Figure 3.43: IR Spectrum of Crychine **60**