CHAPTER 3

RESULTS AND DISCUSSION

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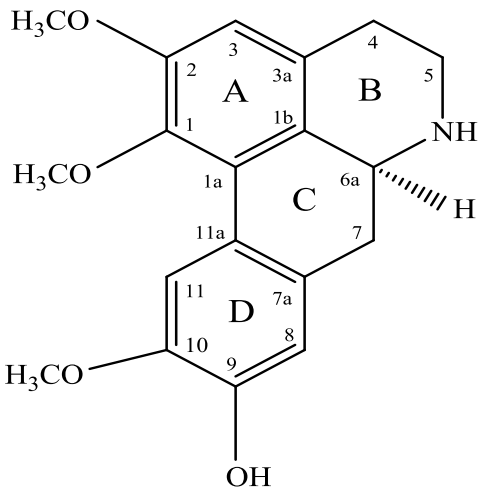
3.1 Introduction

In recent years, chemists are interested in isolation of natural product compounds from the plants due to their pharmacological activities and can be a source of much useful therapies. In this course of studies the plant from the family of Lauraceae, *Cryptocarya densiflora* has been selected to study its alkaloid contents.

3.2 Alkaloids of *Cryptocarya densiflora*

The barks and leaves of *Cryptocarya densiflora* were chosen for the investigations and the isolation techniques were carried out using the conventional methods (CC, TLC, PTLC). The alkaloids from barks and leaves of *Cryptocarya densiflora* were isolated by using acid base extraction as mention in chapter 5 (experimental part). The crude alkaloid obtained consists of aporphine, oxoaporphine, benzylisoquinoline and pavine types. Isolation from the barks gave six alkaloids while from leaves yielded four alkaloids. The alkaloids include laurotetanine **63**, isocaryachine **64**, *N*-demethylphyllocryptine **65**, nornantenine **66**, reticuline **14**, laudanidine **2**, dicentrinone **67**, crychine **60**, *N*-methyllaurotetanine **53** and a novel alkaloid cryptocaryadine **68**.

3.2.1 Alkaloid CD1: Laurotetanine 63



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Alkaloid **CD1**, $[\alpha]^{26}_{D}$ = + 125.0^{o} (c = 2.24, MeOH) was afforded as a pale brownish amorphous solid. The UV spectrum showed absorptions at 220, 281, 302 and 312 nm, which were typical of an aporphinic nature, thus suggesting a 1,2,9,10-tetrasubstituted aporphine skeleton[60]. The IR spectrum showed a strong absorption peak at 3429.87 cm^{-1} due to the stretching of O-H group of phenolic hydroxyl moiety. Other absorption peaks were at 1508, 1238 and 1464 cm^{-1} which indicated the C-H aromatic, C-O and CH_2 respectively. The ESIMS (positive mode) spectrum showed an intense pseudomolecular ion peak, $[M+H]^+$ at m/z 328.16 corresponding to the molecular formula of $C_{19}H_{21}NO_4$.

The ^{1}H NMR spectrum of **CD1** (Table 3.1 and Figure 3.1) exhibited three distinct methoxyl signals at δ 3.64, 3.86 and 3.87 which were most probably positioned at C-1, C-2 and C-10. The former was assigned to the methoxyl at C-1 since the protons were shielded by the anisotropic effect caused by ring D. A one proton singlet at δ 6.57 was observed in the spectrum, confirming that C-1 and C-2 are substituted.

Furthermore, the singlet at δ 6.77 could be attributed to H-8. This value was typical of a 9, 10-substitution pattern[80-81]. It was clear that, the low field signal of H-11 at δ 8.06 suggested that C-10 was substituted by a methoxyl group. The aliphatic protons gave a multiplet signal between δ 3.80-2.64. The above observations were reinforced by COSY experiment which displayed correlations of H-4/H-5 and H-6a/H-7.

The ^{13}C NMR spectrum (Figure 3.2) established the resonances of nineteen carbons while DEPT experiment (Figure 3.3) showed three methyls, three methylenes, four methines and nine quaternary carbon signals in the molecule, consistent with the structure proposed. In the HMBC spectrum (Figure 3.4), the cross-peaks were observed between H-8/C-10, C-7/C-11a; 10-OMe/C-10 suggesting that a methoxyl group attached to C-10 instead of C-9. The complete assignment of proton and carbon were obtained through the aid of the 2D (COSY, HMQC and HMBC) experiments.

Finally, comparison of the spectroscopic data obtained with the literature values, alkaloid **CD1** was deduced as laurotetanine[82-86] **63**, which existed widely among the family of Annonaceae, Monimiaceae, Papaveraceae and Lauraceae[86-89].

Table 3.1: ^{1}H NMR (in $CDCl_3$, 400MHz) and ^{13}C NMR (in $CDCl_3$, 100MHz) of **63**

Position	δ_H, ppm (J in Hz)	δ_C (ppm)	HMBC (H→C)	HMQC
1	-	144.31		
1a	-	126.83		
1b	-	127.42		
2	-	152.23		
3	6.57 (*s*)	110.83	4, 1b, 1, 2	H-3
3a	-	129.00		
4	2.74 (*dd*, 13.68, 4.64)	29.09	6a, 1b	H-4
5	3.01 (*dd*, 12.92, 4.1) 3.65 (*m*)	43.14	6a, 3a	H-5
6a	3.80 (*dd*, 4.40, 13.20)	53.78		H-6a
7	2.64 (*d*, 13.68)	36.59	1b, 6a, 8, 11a	H-7
7a	-	129.79		
8	6.77 (*s*)	113.97	7, 11a, 10	H-8
9	-	145.37		
10	-	144.98		
11	8.06 (*s*)	111.36	7a, 9	H-11
11a	-	124.05		
1-OMe	3.64 (*s*)	60.28	1	
2-OMe	3.86 (*s*)	56.11	2	
10-OMe	3.87 (*s*)	55.92	10	

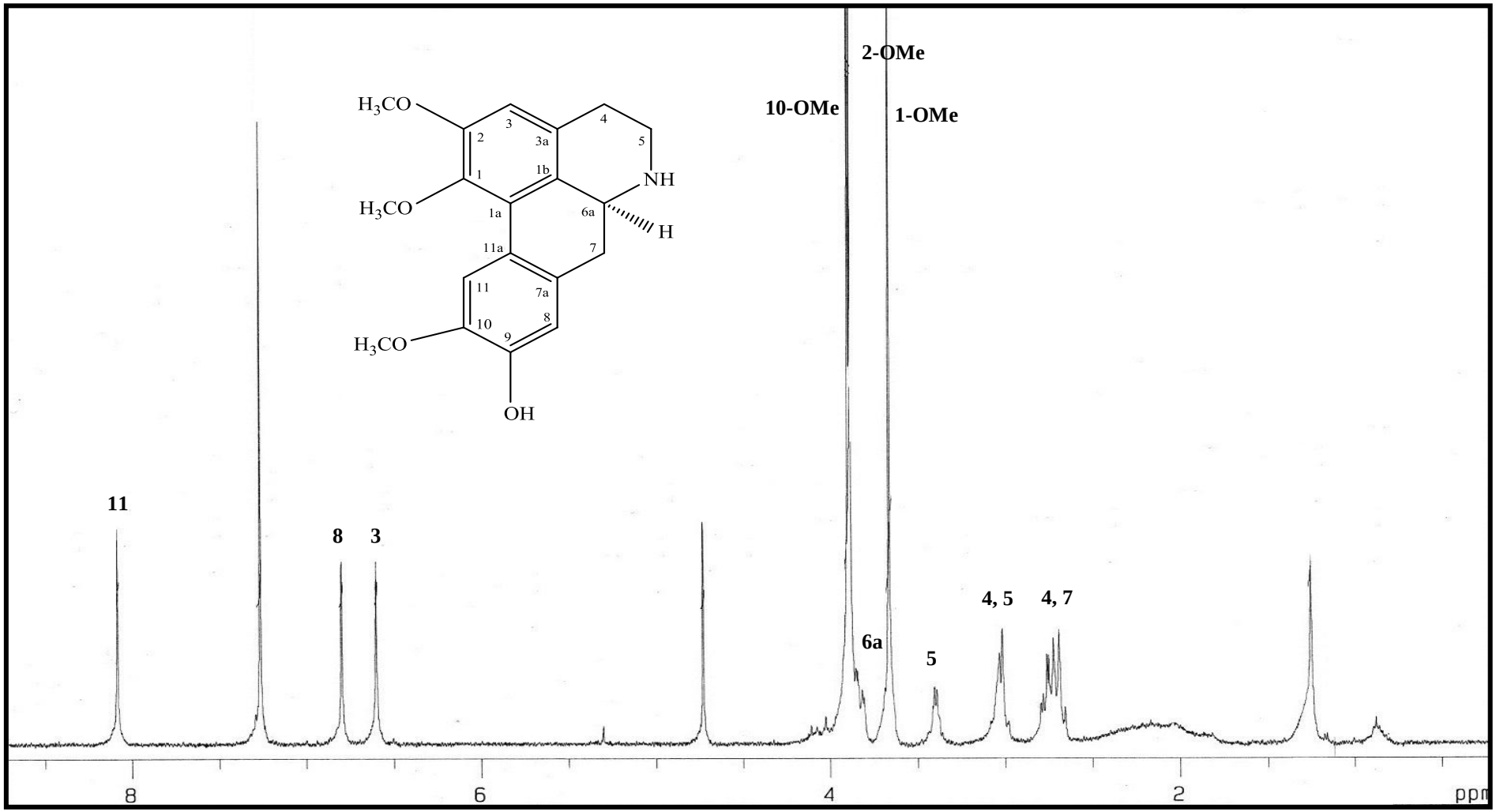

Figure 3.1: ^{1}H NMR Spectrum of Laurotetanine **63**

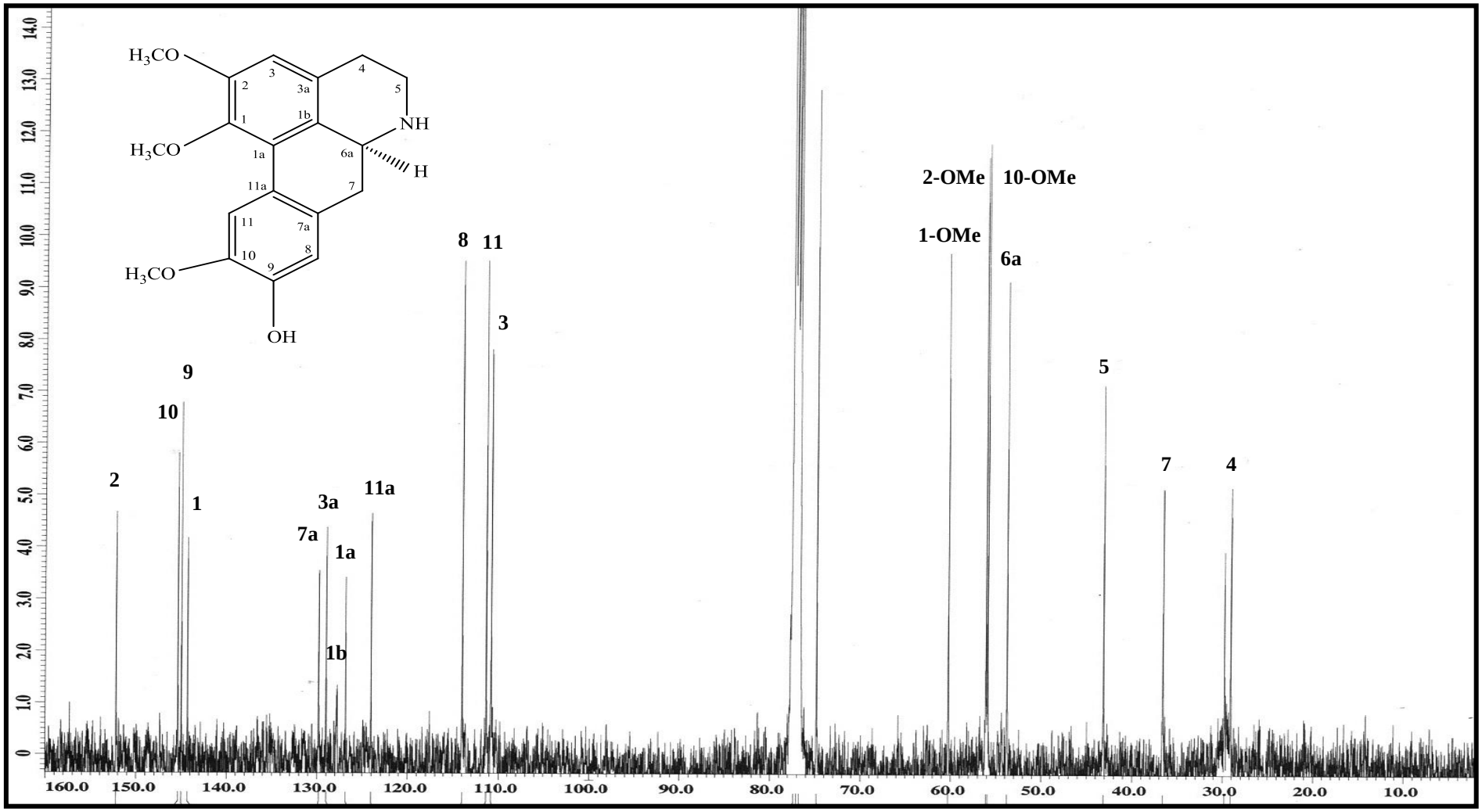

Figure 3.2: ^{13}C NMR Spectrum of Laurotetanine **63**

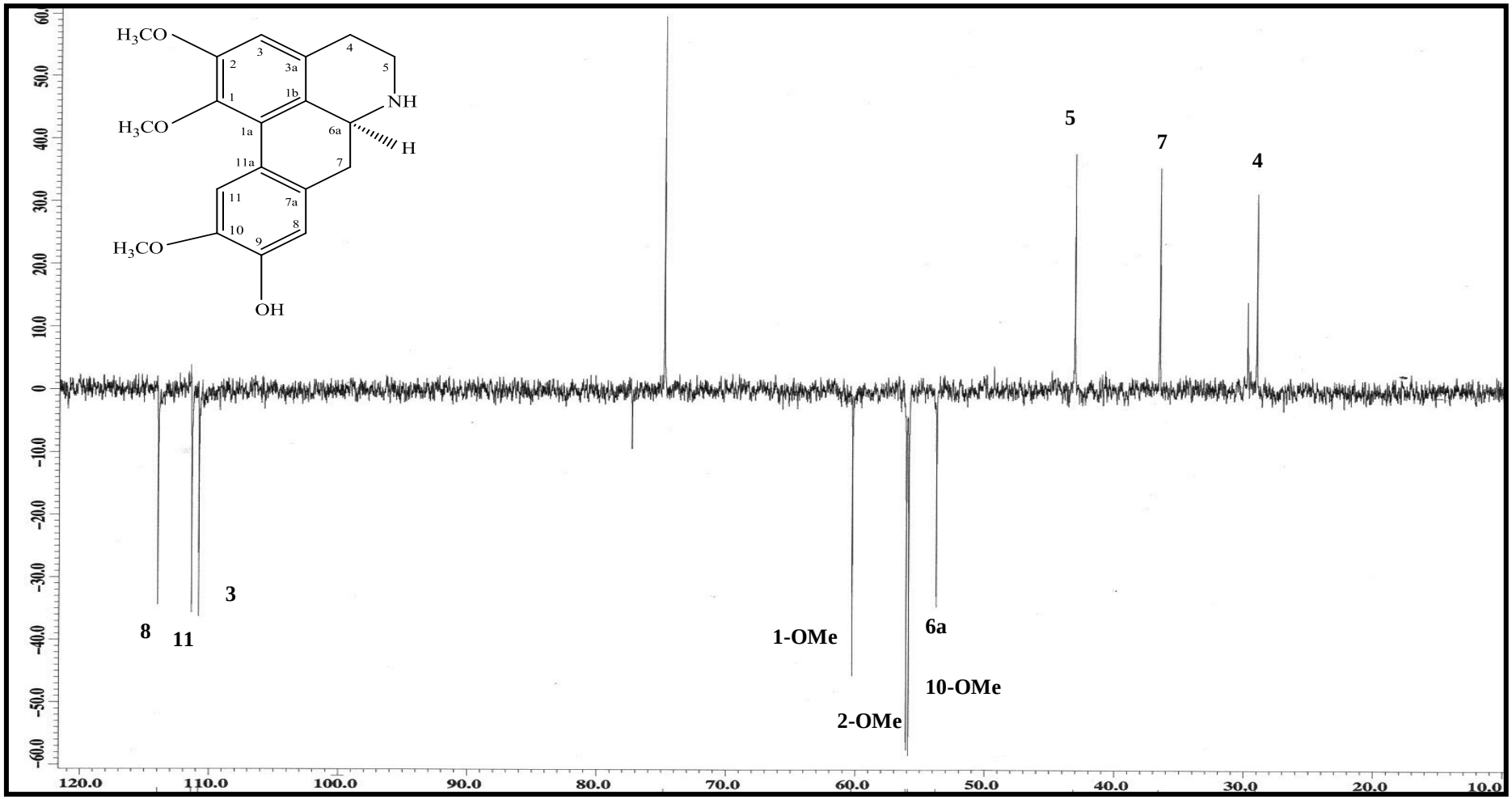

Figure 3.3: DEPT Spectrum of Laurotetanine **63**

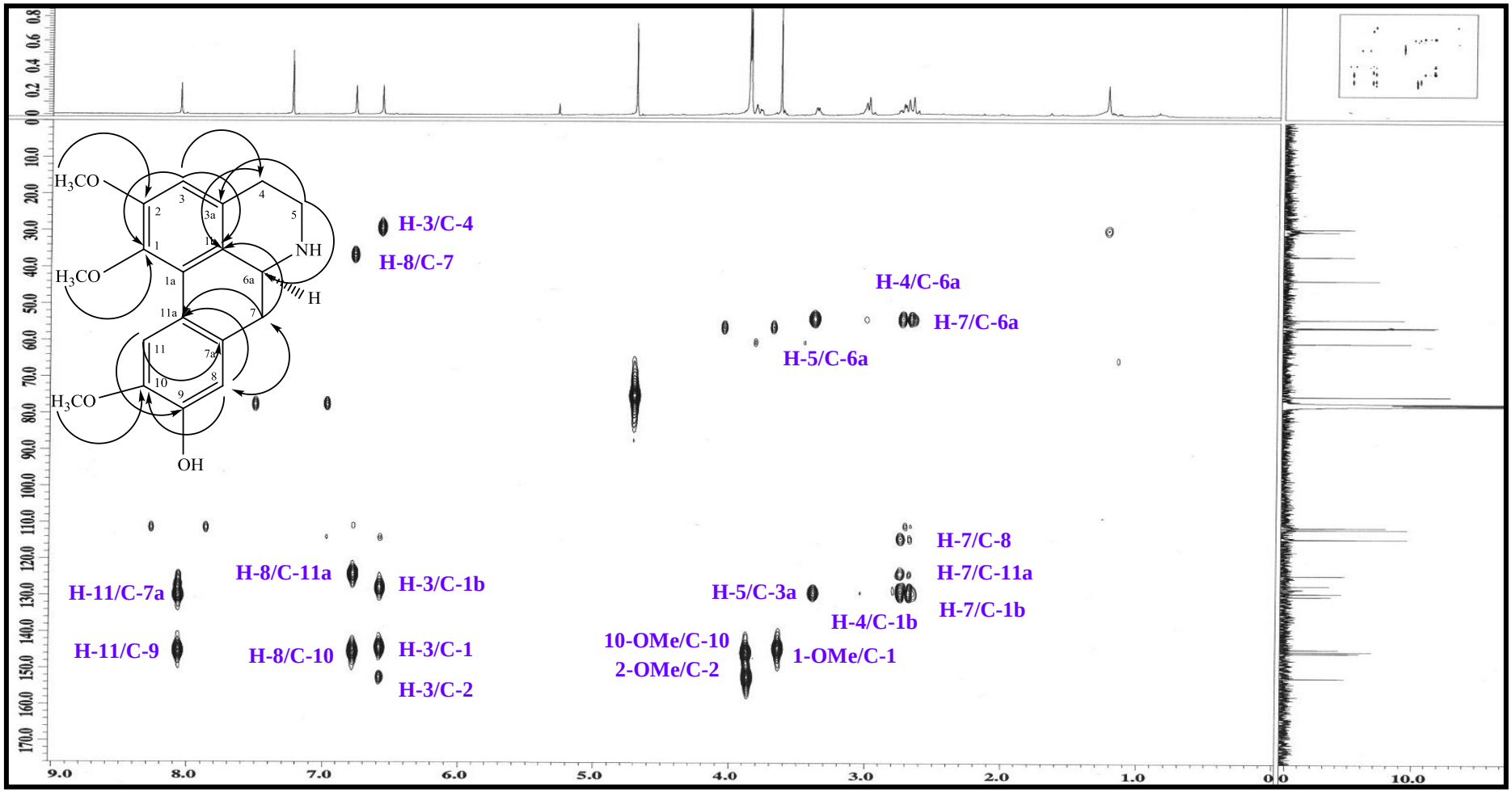

Figure 3.4: HMBC Spectrum of Laurotetanine **63**

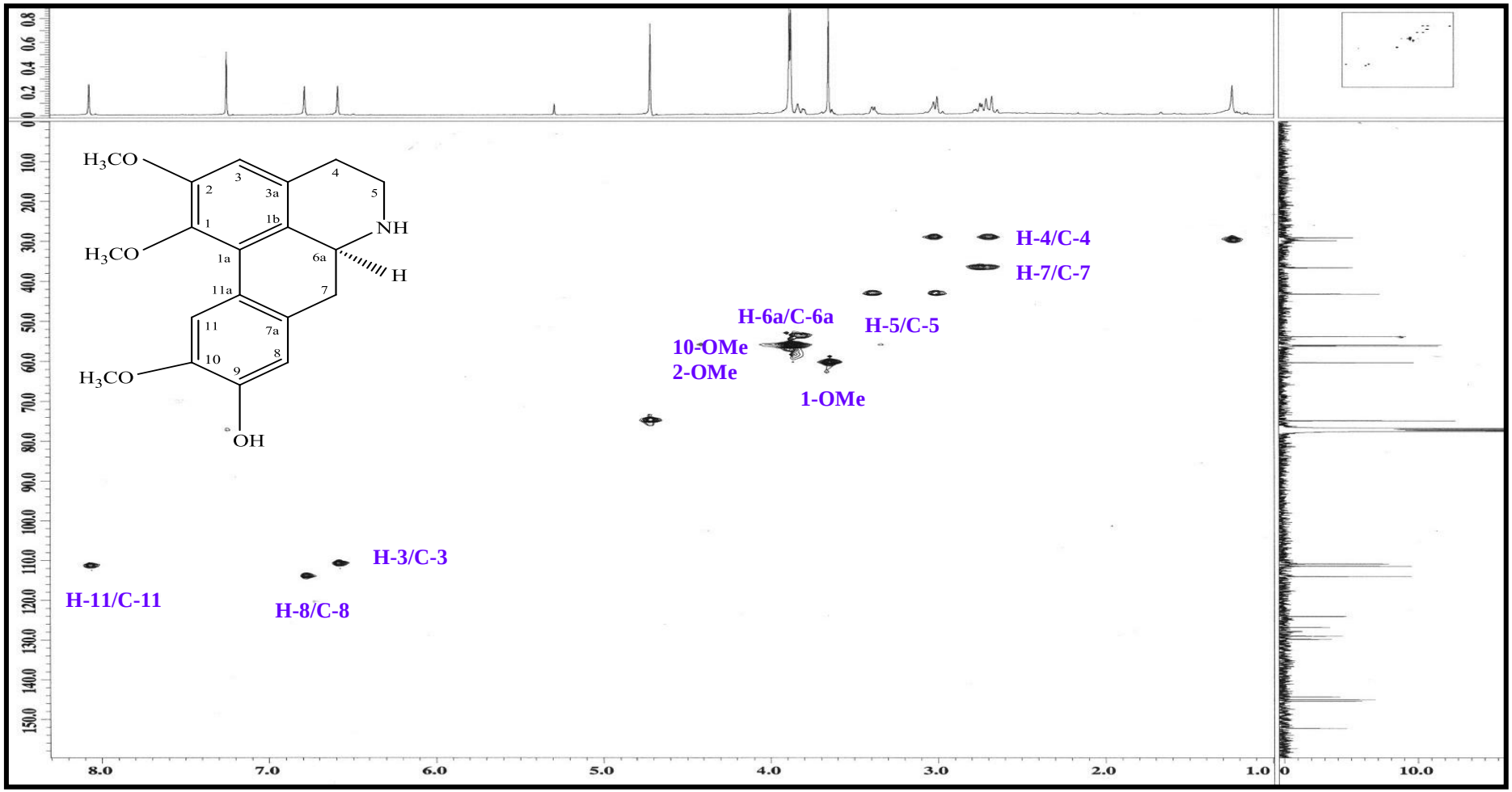

Figure 3.5: HMQC Spectrum of Laurotetanine **63**

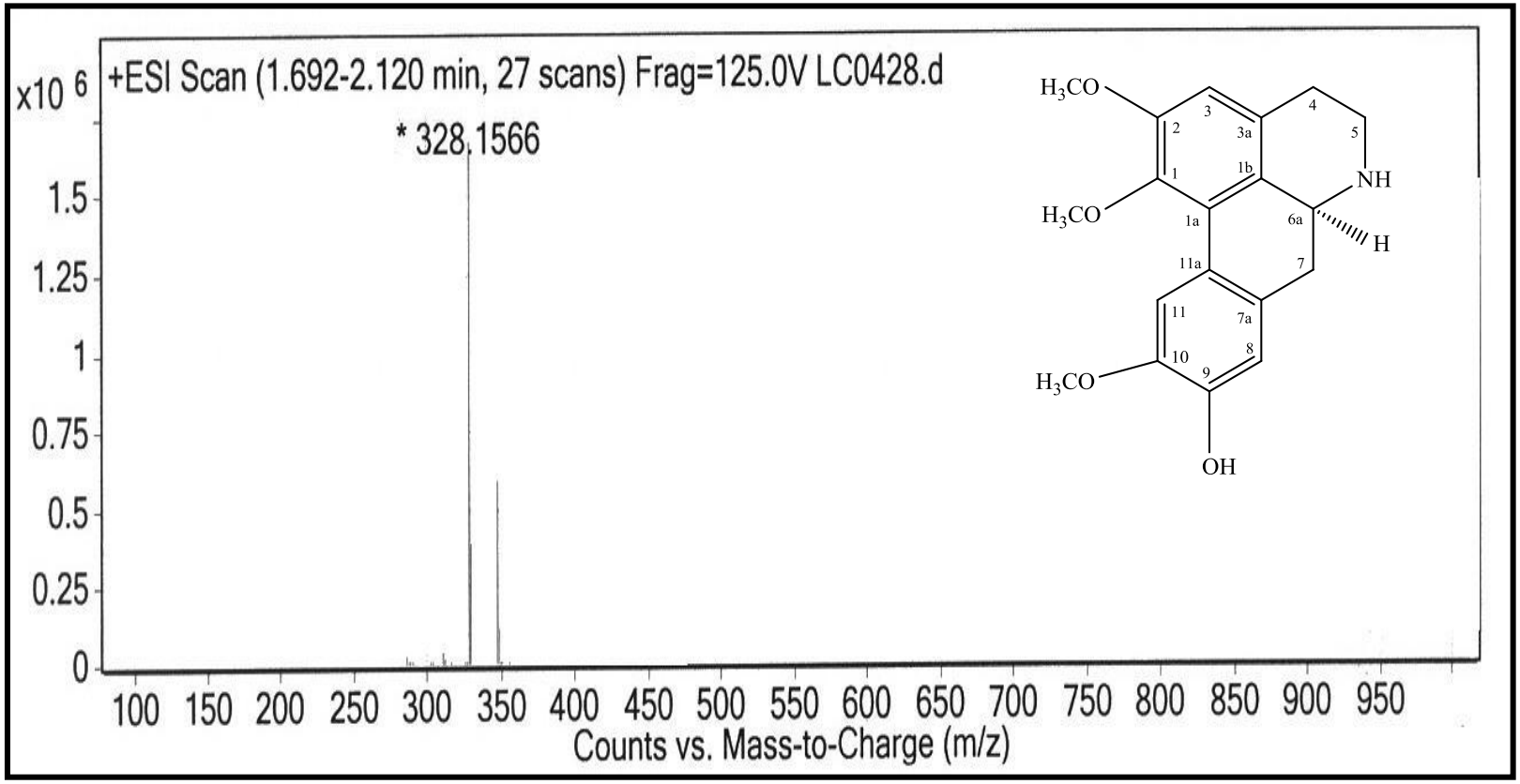

Figure 3.6: LCMS Spectrum of Laurotetanine **63**

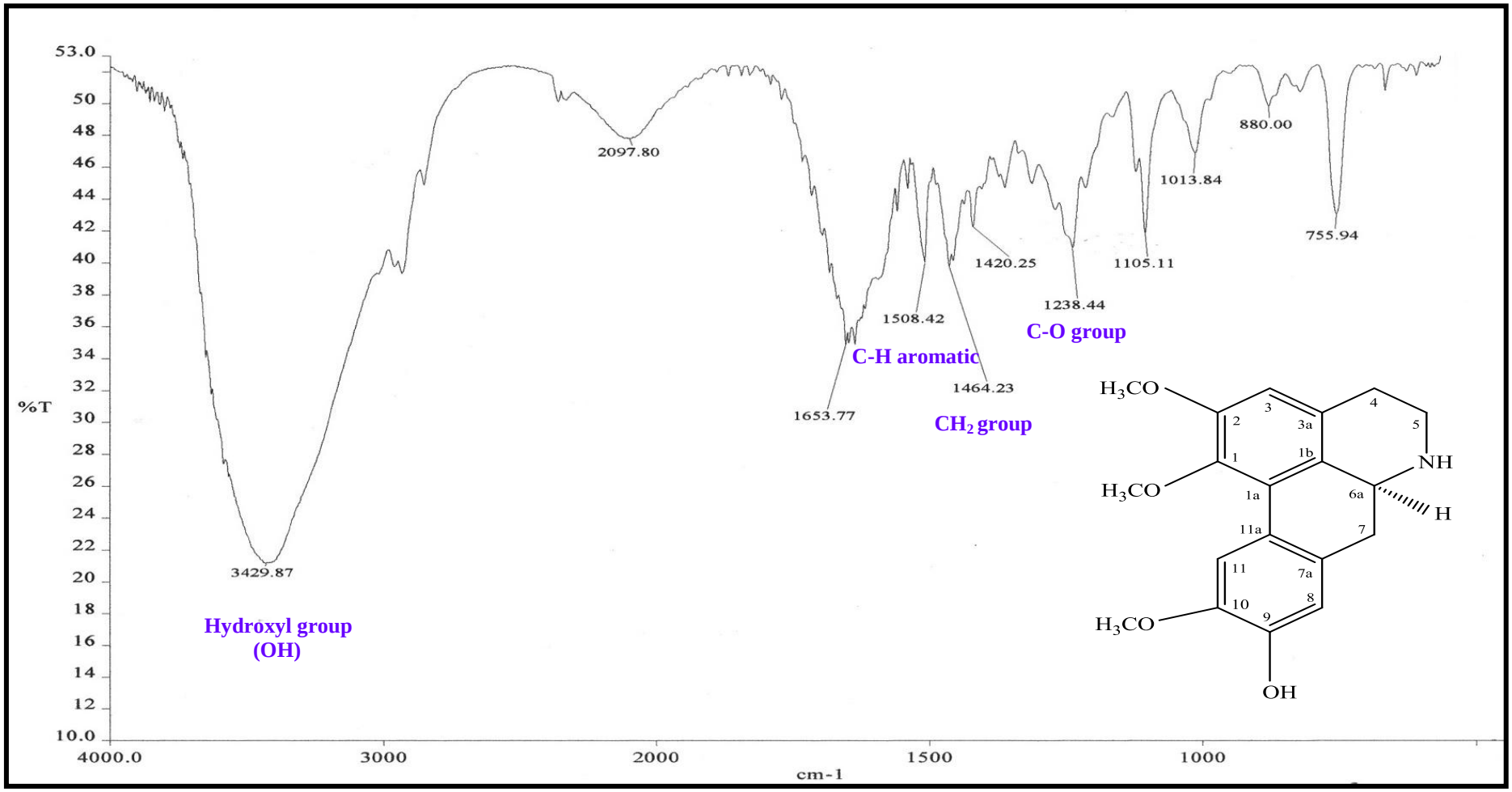

Figure 3.7: IR Spectrum of Laurotetanine **63**