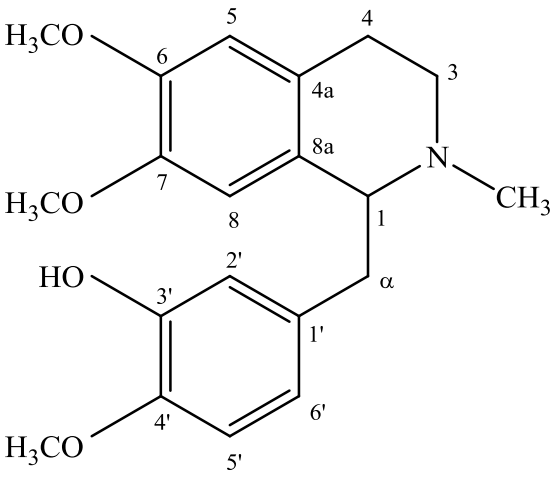
3.2.6 Alkaloid CD6: Laudanidine 2



2

Alkaloid **CD6** was obtained as a pale brownish amorphous solid. The UV spectrum showed two absorption peaks at 298 and 244 nm, which indicated the existence of the conjugated aromatic system. The IR spectrum showed an absorption band at 3390 cm^{-1}, which indicated the presence of a hydroxyl group in the molecule. The ESI (positive mode) spectrum revealed a molecular ion peak, $[M+H]^+$, at m/z 344.18 suggested a molecular formula of $C_{20}H_{25}NO_4$.

The ^1H NMR spectrum (Figure 3.32) displayed five aromatic proton signals at the lower region. There were two singlets at δ 6.53 and 6.03 attributable to H-5 and H-8 respectively. H-8 was shielded compared to H-5 because of the anisotropic effect caused by ring C (facing the ring C). Another two of the proton signals appeared at δ 6.75 (*d*, *J* = 2.07 Hz, H-2') and δ 6.70 (*d*, *J* = 8.17 Hz, H-5') as a doublet. H-6' signal appeared as a doublet of doublets (*dd*) at δ 6.50 (*dd*, *J* = 2.07, 8.17 Hz, H-6'). In addition, three distinct singlet peaks at δ 3.80, 3.54, and 3.82 were attributed to the methoxyl groups positioned at C-6, C-7 and C-4', respectively. Furthermore, a singlet resonated at δ 2.49 was a characteristic of the *N*-CH_3 group. A total of four proton

signals were observed at a higher region between δ 2.68-3.67 attributable to the aliphatic protons of H-α, H-1, H-3 and H-4. Due to the limitation of the sample amount, we were unable to analyze the compound using 2D NMR spectrum, however by comparison the spectroscopic data obtained (^{1}H NMR, ^{13}C NMR, IR, UV, MS) with the published data [100-101], confirmed that alkaloid **CD6** is laudanidine **2**.

Table 3.6: ^{1}H NMR (in $CDCl_3$, 400MHz) and ^{13}C NMR (in $CDCl_3$, 100MHz) of **2**.

Position	$\delta_{H,}$ ppm (J in Hz)	δ_C (ppm)[100]
1	3.67 (*dd*, 5.2, 7.8)	64.7
3	3.2-3.14 (*m*)	46.5
	2.78-2.73 (*m*)	
4	2.86-2.80 (*m*)	25.0
	4.5, 16.0	
4a		125.5
5	6.53 (*s*)	111.9
6	-	147.2
7	-	146.2
8	6.03 (*s*)	111.0
8a	-	129.2
α	3.11 (*dd*, 5.2, 13.7)	40.73
	2.68 (*dd*, 7.9, 13.7)	
1’	-	133.2
2’	6.75 (*d*, 2.07)	115.8
3’	-	145.5
4’	-	145.1
5’	6.70 (*d*, 8.17)	110.4
6’	6.50 (*dd*, 2.07, 8.17)	121.2
6-OMe	3.80 (*s*)	55.69
7-OMe	3.54 (*s)*	55.24
4’-OMe	3.82 (*s*)	55.94
N-Me	2.49 (*s*)	42.40

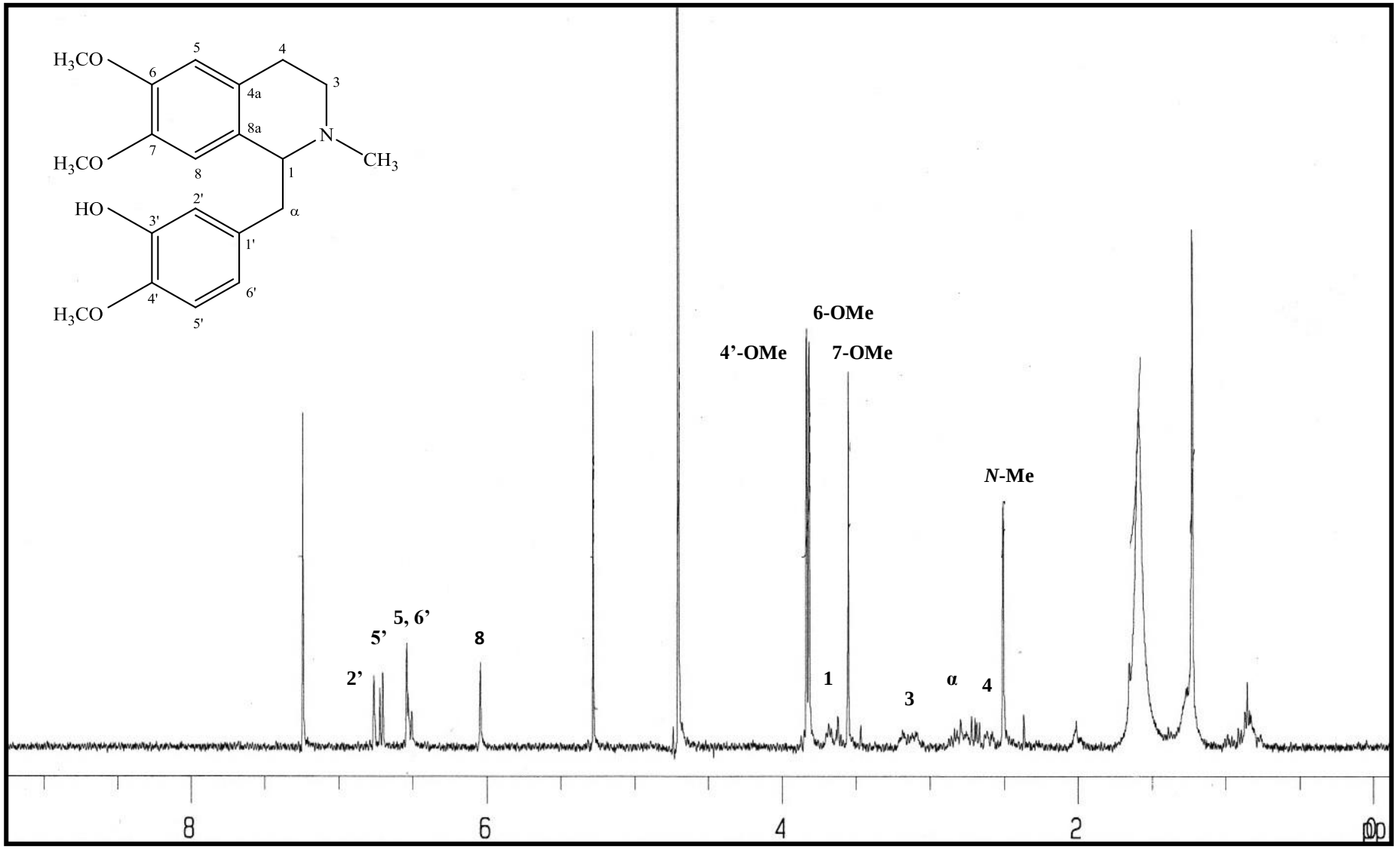


Figure 3.32: ^{1}H NMR Spectrum of Laudanidine **2**

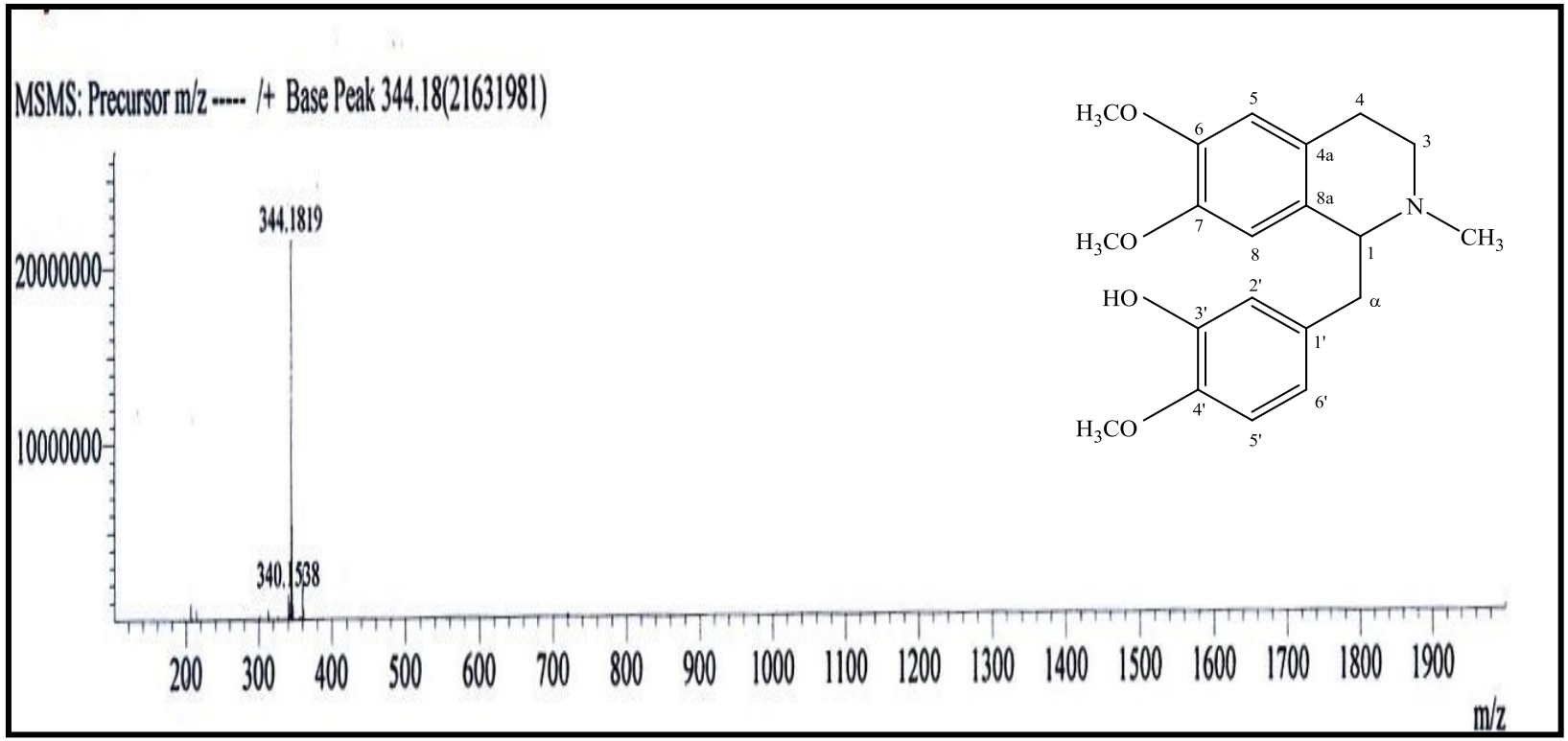


Figure 3.33: LCMS Spectrum of Laudanidine **2**