Supporting Information

4,4′-Bismoschamine: Biomimetic Synthesis and Evidence to Support Structural Equivalency to Montamine

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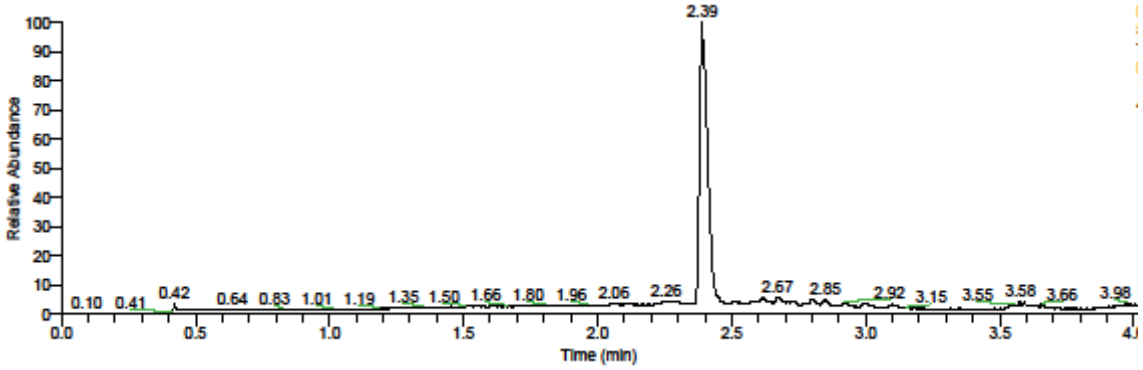
Contents

LCMS of 4,4'-bis(*N*-Boc)serotonin (**5**)



LCMS of 4,4'-bismoschamine (**3**)

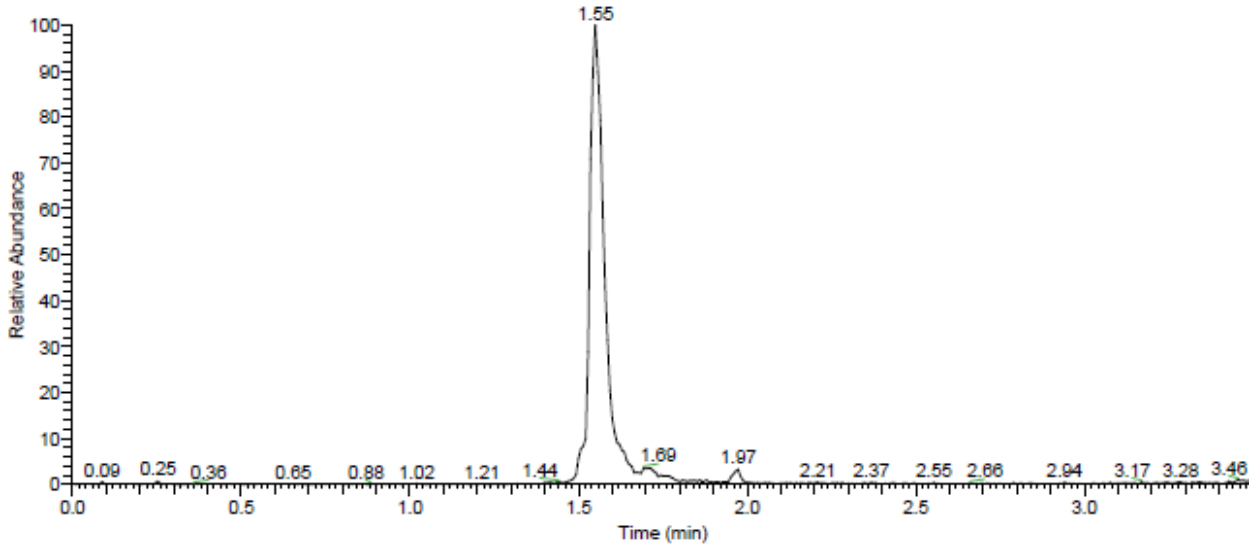
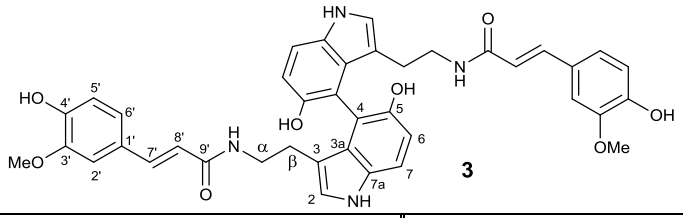


Table S1. Comparison of NMR data (DMSO-d_6) for synthetic 4,4′-bismoschamine prepared herein, 4,4′-bismoschamine prepared by Xia[1] and the natural product 4,4′-bismoschamine.[2]



3

	¹H (ppm), multiplicity, *J* (Hz)			¹³C (ppm), multiplicity, *J* (Hz)			
Carbon	synthetic **3** 400 MHz	synthetic **3**[1] **(Xia)** 400 MHz	natural **3**[2] 500 MHz	synthetic **3** 100 MHz solv. ref. 39.5 ppm	synthetic **3** 100 MHz solv. ref. 40.1 ppm[a]	Xia's synthetic **3**[1] 100 MHz solv. ref. 40.1 ppm[a]	natural **3**[2] 125 MHz spectrum not published
2	6.90-6.93, m	6.90, d (1.8)	6.90, d (1.8)	123.3	123.9	123.8	123.9
3	-	-	-	112.6	113.2	113.2	113.2
3a	-	-	-	127.3	127.9	127.8	127.9
4	-	-	-	113.8	114.4	114.4	114.4[b]
5	-	-	-	147.8	148.4	148.4	148.4
6	6.75, d (8.6)	6.73, d (8.5)	6.74, d (8.5)	111.0	111.6	111.6	111.6
7	7.15, d (8.6)	7.14, d (8.6)	7.14, d (8.5)	110.4	111.0	111.0	111.0
7a	-	-	-	131.1	131.7	131.7	131.7
β-CH_2	2.15-2.02 m	2.07-2.03 m	2.07, m	25.1	25.7	25.7	25.7
α-CH_2	2.91-2.71 m	2.91-2.70 m	2.80, m	39.2	39.8[c]	40.7	40.7
1′	-	-	-	126.5	127.1	127.0	127.1[d]
2′	7.03, d (1.9)	7.02, d (1.8)	7.02, d (1.8)	110.7	111.3	111.2	111.3
3′	-	-	-	147.8	148.4	148.3	148.4
4′	-	-	-	148.1	148.7	148.7	148.8
5′	6.74, d, (8.1)	6.73, d, (7.9)	6.74, d, (7.9)	115.6	116.2	116.2	116.2
6′	6.90-6.93 m	6.90, dd, (7.9, 1.8)	6.90, dd, (1.8, 7.9)	121.4	122.0	122.0	122.0
7′	7.22, d (15.7)	7.21, d (15.6)	7.21, d (15.8)	138.5	139.1	139.1	139.2
8′	6.41, d (15.7)	6.40, d (15.7)	6.40, d (15.8)	119.2	119.8	119.8	119.8[e]
9′	-	-	-	165.1	165.7	165.7	165.8
OMe	3.73, s	3.73, s	3.72, s	55.4	56.0	56.0	56.1
Indole NH	10.44, d (2.2)	10.42, s	10.4, s	-	-	-	-
Amide NH	7.38, t (5.3)	7.37, t (4.9)	7.38, t	-	-	-	-
5-OH	7.77, br s	7.75, s	7.75, s	-	-	-	-

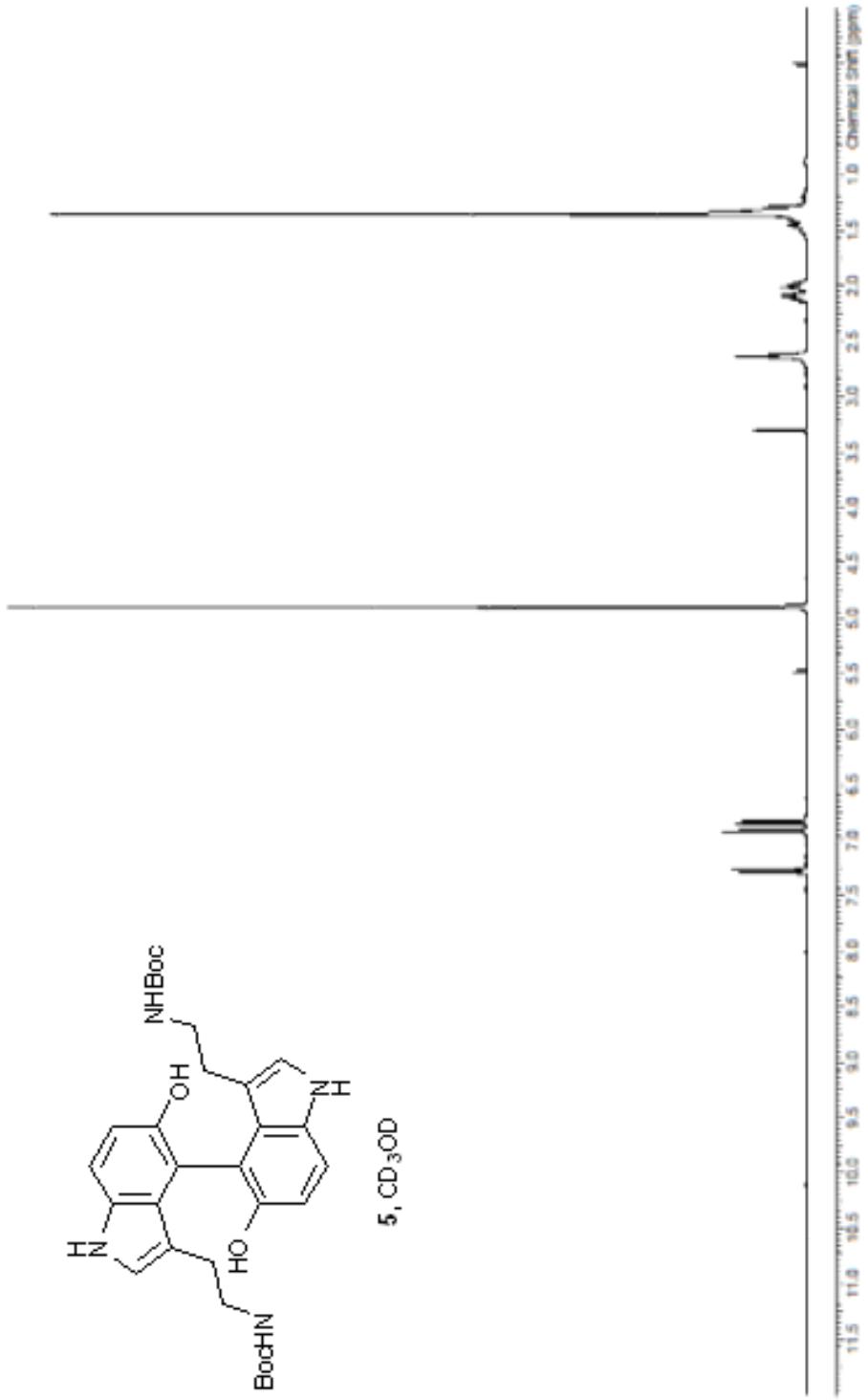
[a] Xia et al reported[1] the synthesis of **3** with ^{13}C NMR data that was not referenced to standard solvent signals. We calibrated our data to the standard residual DMSO as well as to the reference these authors used. [b] The original safflower natural product report[2] cites a resonance of 114.0, but this was revised to 114.4 by Xia[1] after correspondence with the isolation authors. [c] This region of the spectrum is obscured by residual DMSO. See page 16 of this document for a view of this region of the spectrum. [d] The original safflower natural product report[2] cites a resonance of 125.0, but this was revised to 127.1 by Xia[1] after correspondence with the isolation authors. [e] The C8′ resonance in **3** was correctly given at 119.8 ppm in the original report,[2a] but misquoted at 129.8 ppm in the subsequent full paper.[2b]

Notes and References

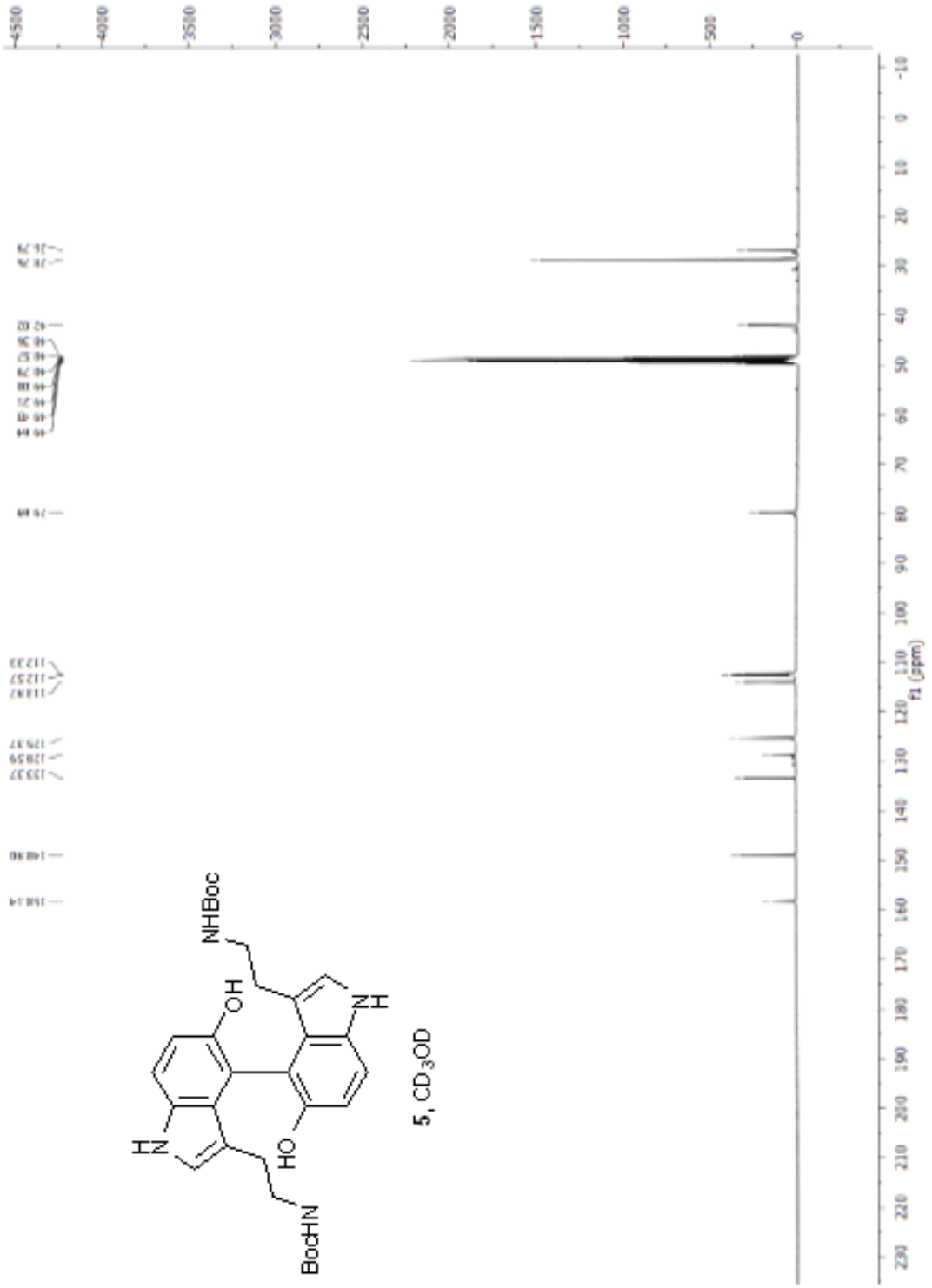
1. K. Liang, J. Yang, X. Tong, W. Shang, Z. Pan, and C. Xia. *Org. Lett.* 2016, **18**, 1474-1477.

2. (a) H. L. Zhang, A. Nagatsu, and J. Sakakibara. *Chem. Pharm. Bull.* 1996, **44**, 874-876; (b) H. L. Zhang, A. Nagatsu, T. Watanabe, J. Sakakibara, and H. Okuyama. *Chem. Pharm. Bull.* 1997, **45**, 1910-1914.

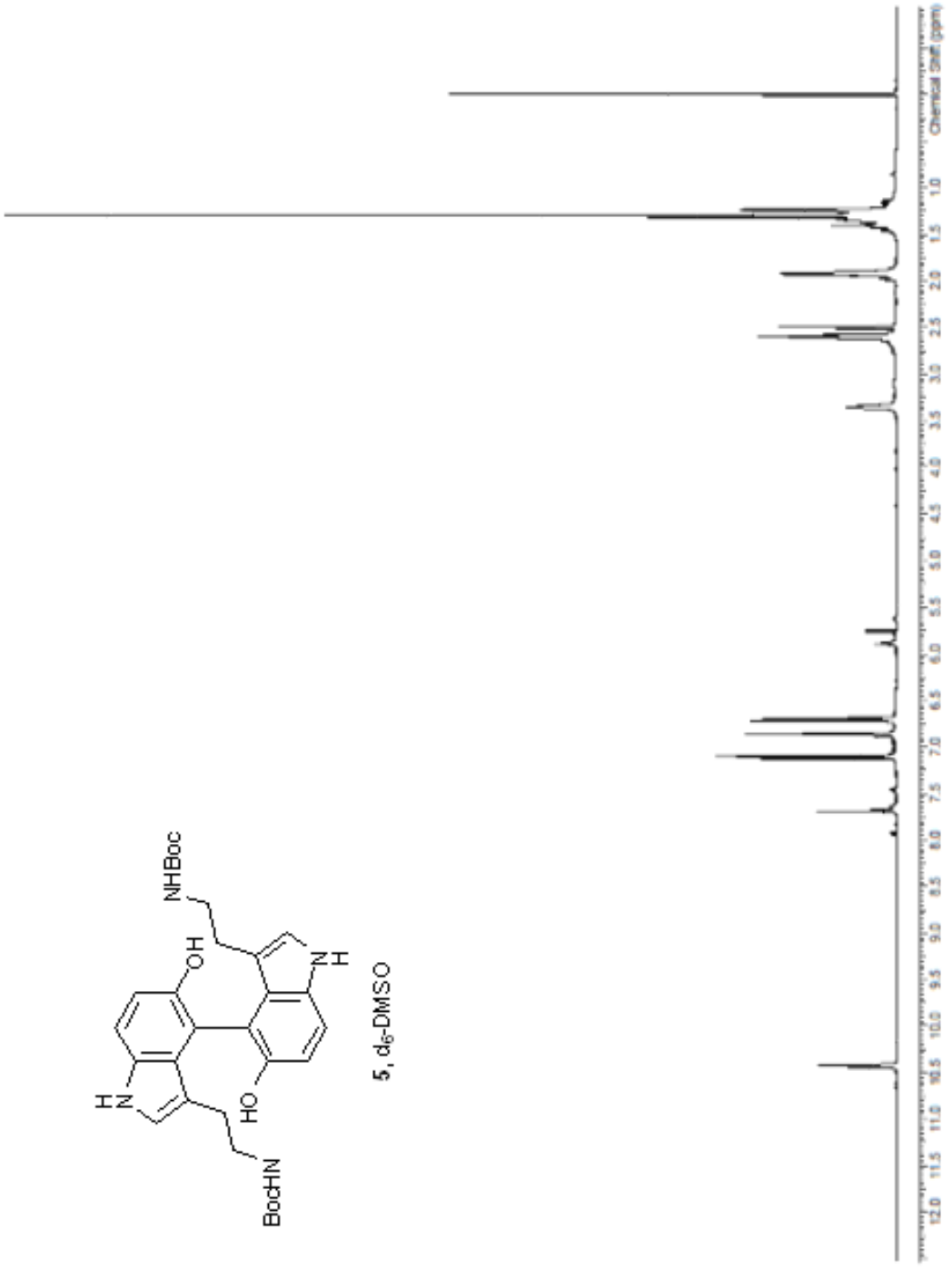
^{1}H NMR spectrum of 5 in CD_3OD



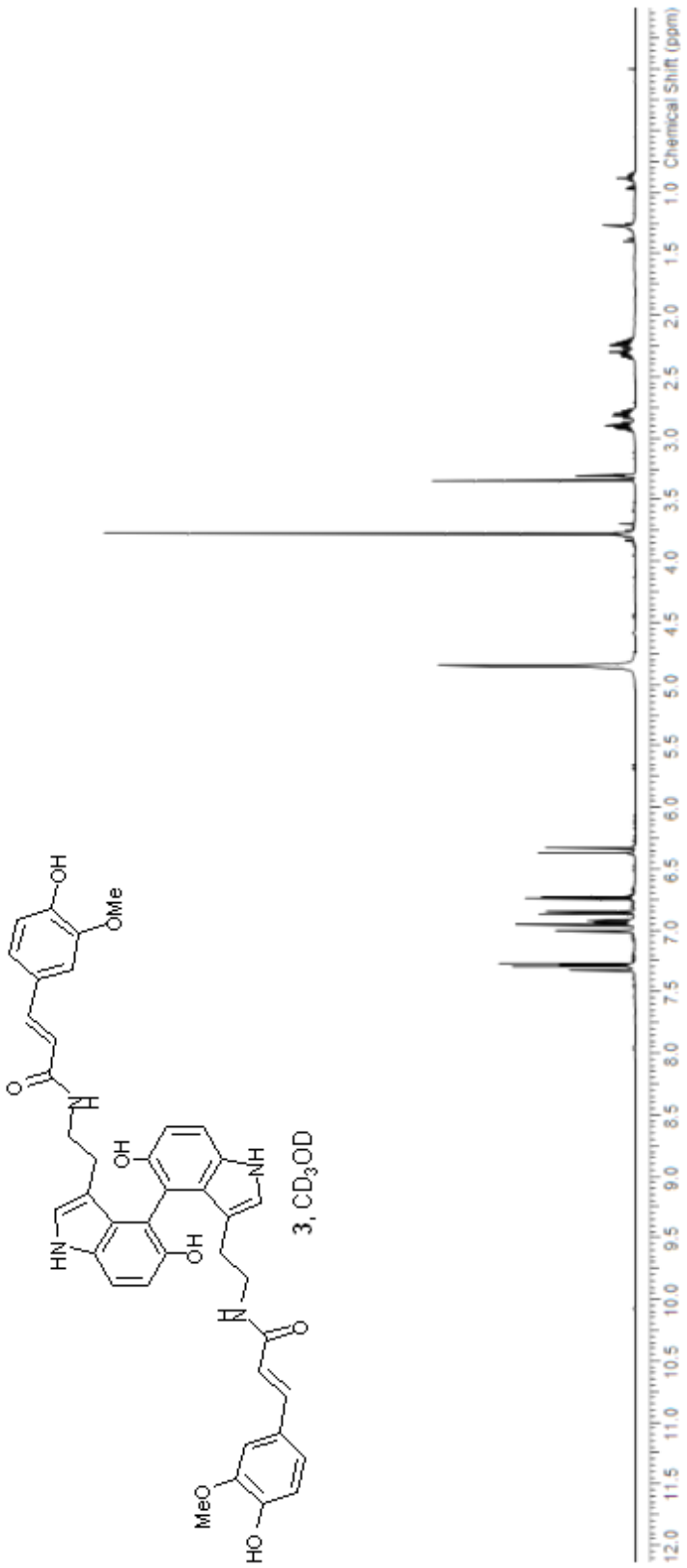
^{13}C NMR spectrum of 5 in CD_3OD



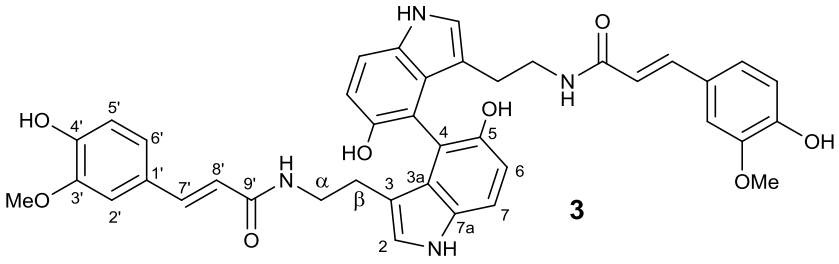
^{1}H NMR spectrum of 5 in DMSO-d_6

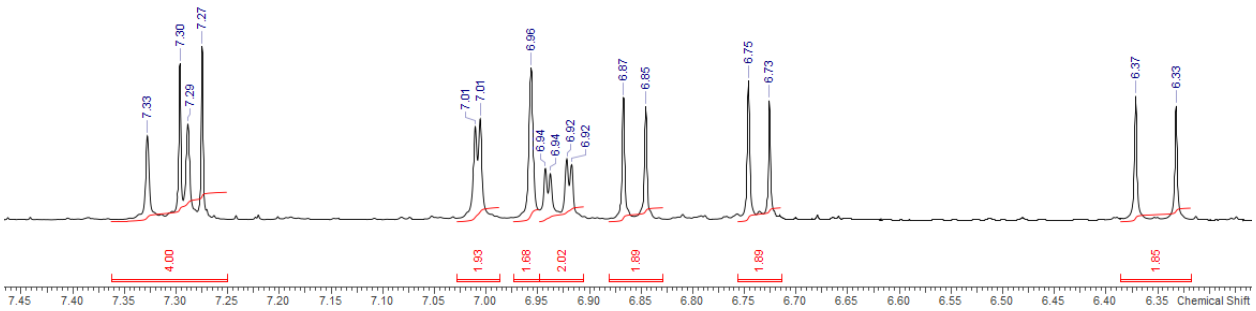


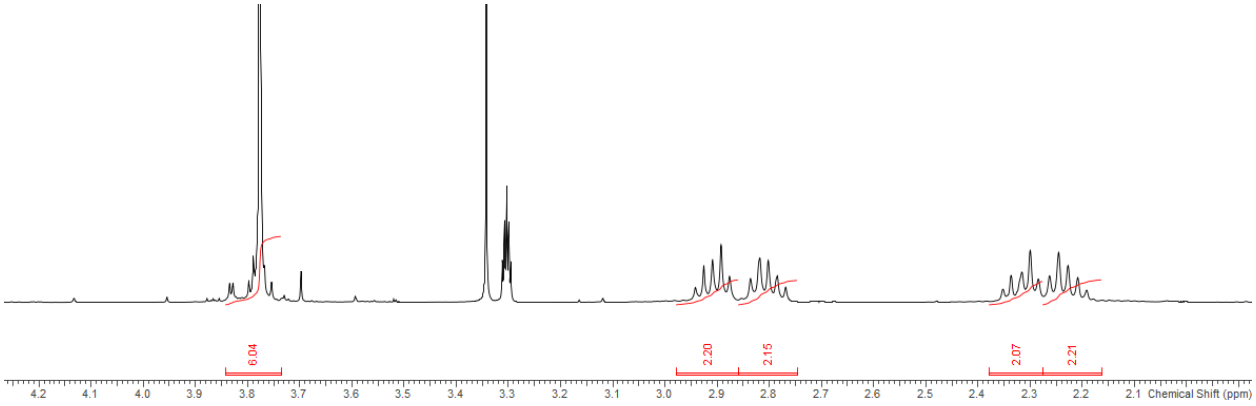
^{1}H NMR spectrum of 3 in CD_3OD:



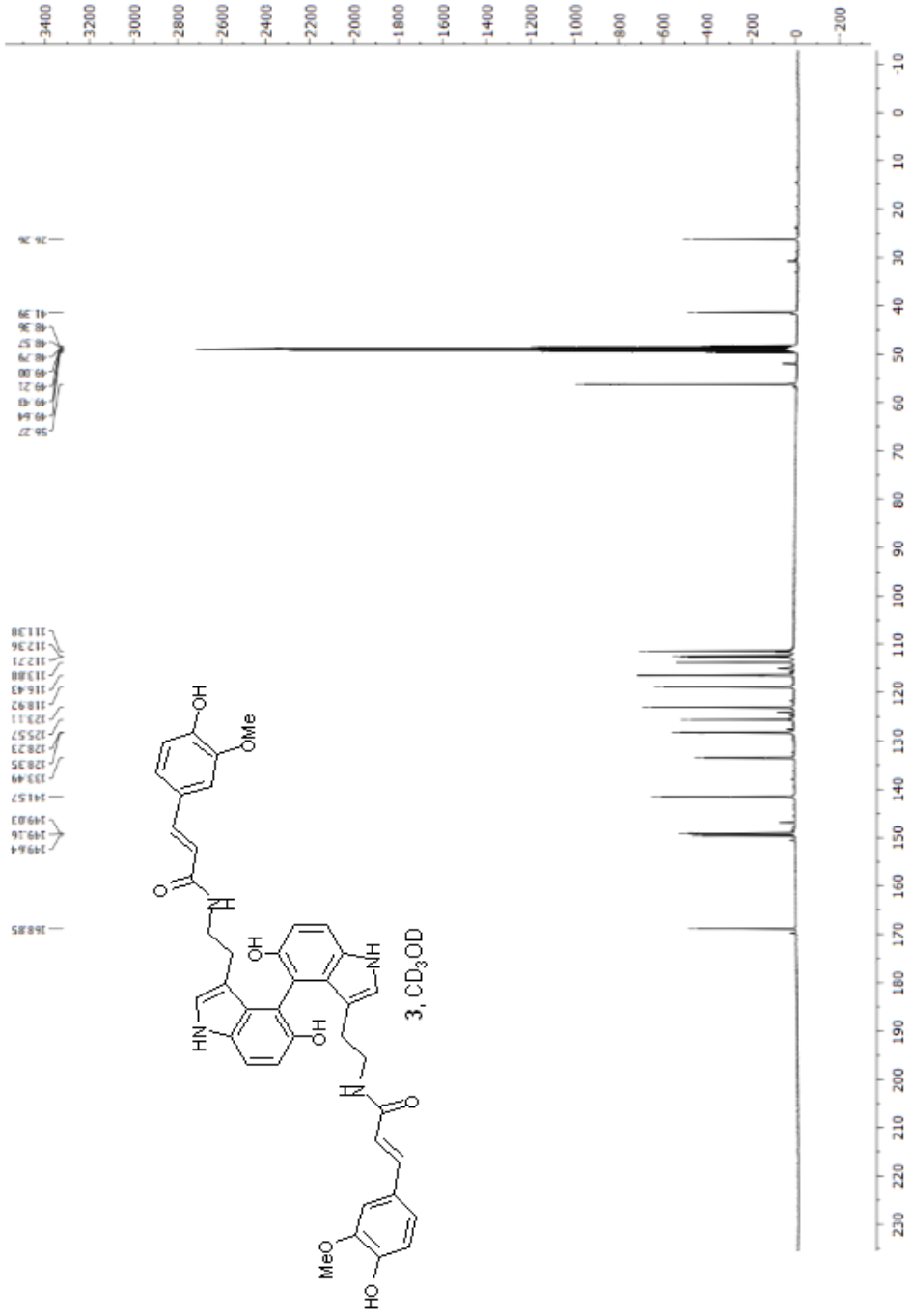
^{1}H NMR spectrum of 3 in CD_3OD: expanded spectral regions with integration shown



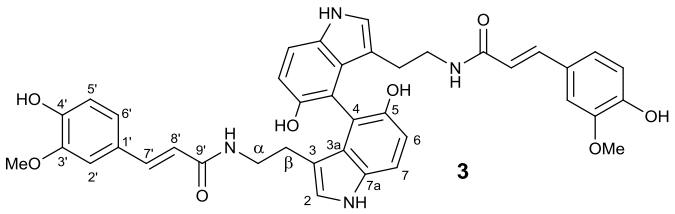


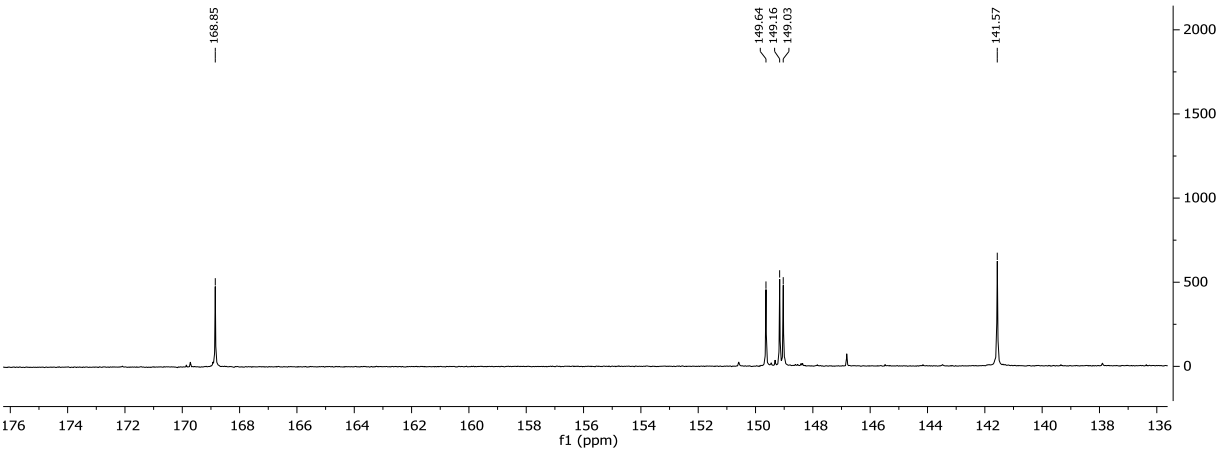


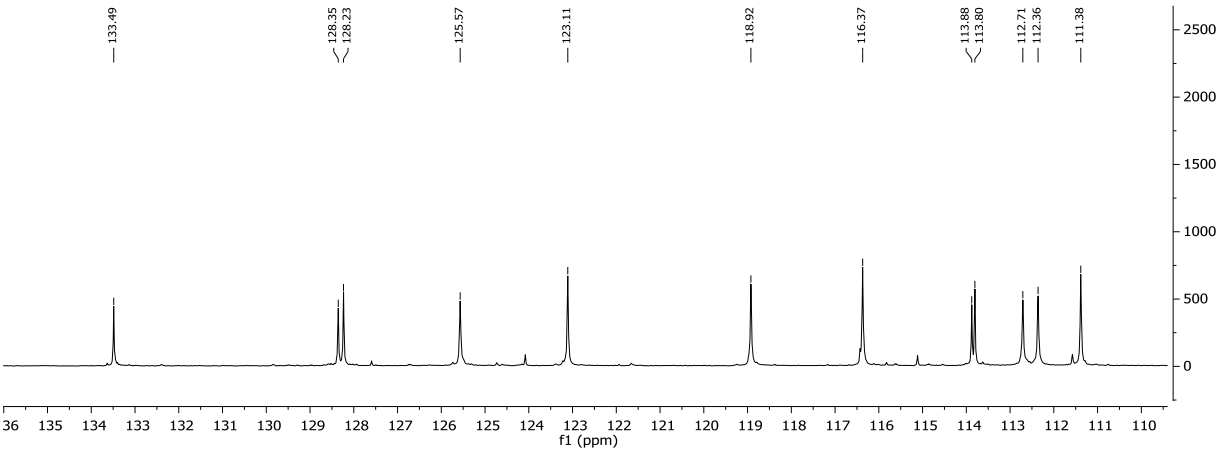
^{13}C NMR spectrum of 3 in CD_3OD

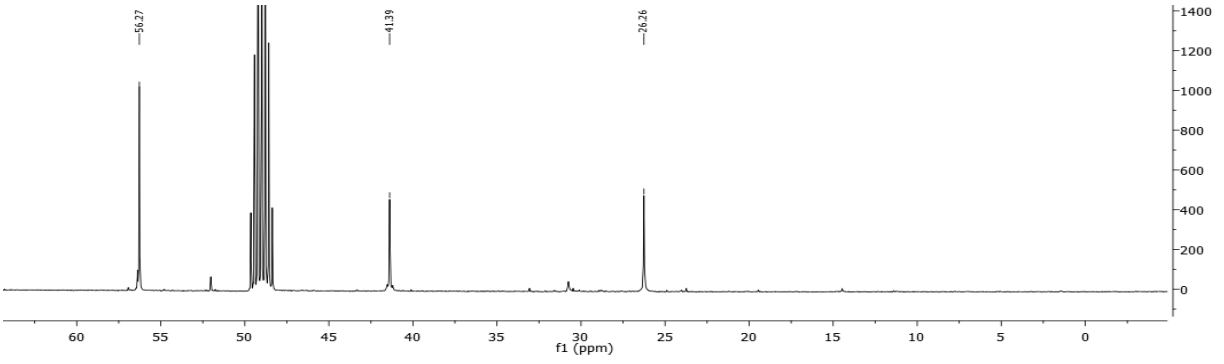


^{13}C NMR spectrum of 3 in CD_3OD: expanded spectral regions

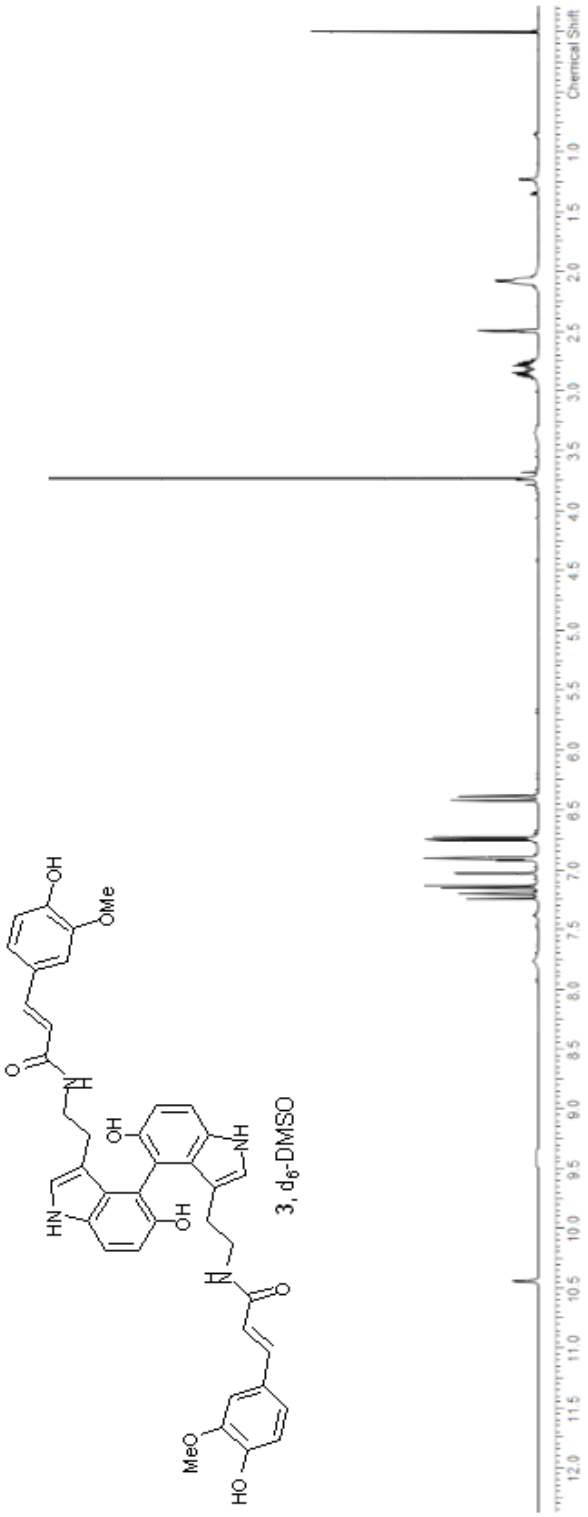




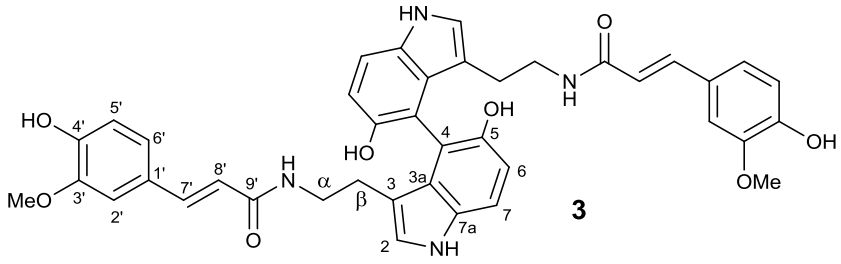


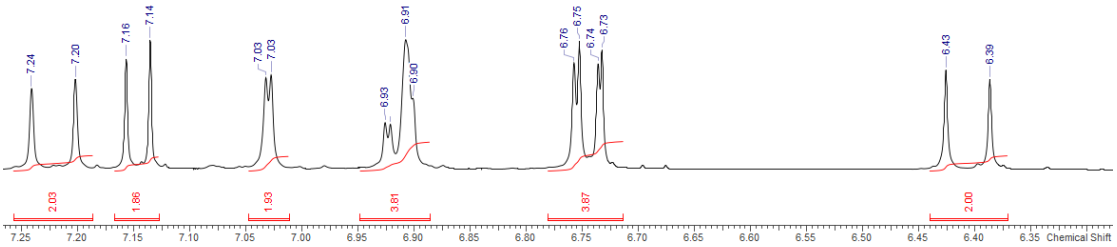


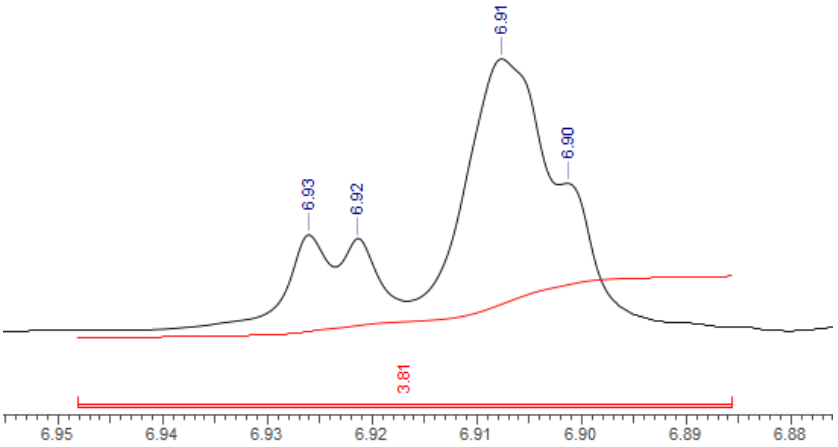
^{1}H NMR spectrum of 3 in DMSO-d_6

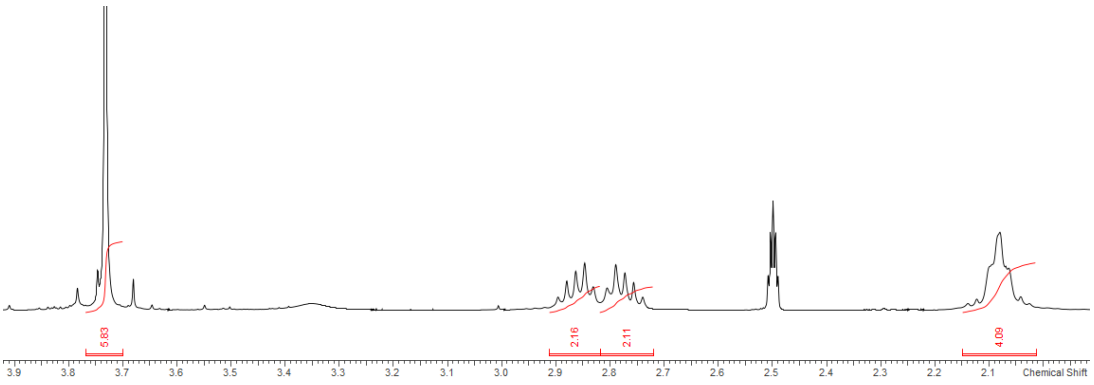


^{1}H NMR spectrum of 3 in DMSO-d$_6$: expanded spectral regions with integration shown

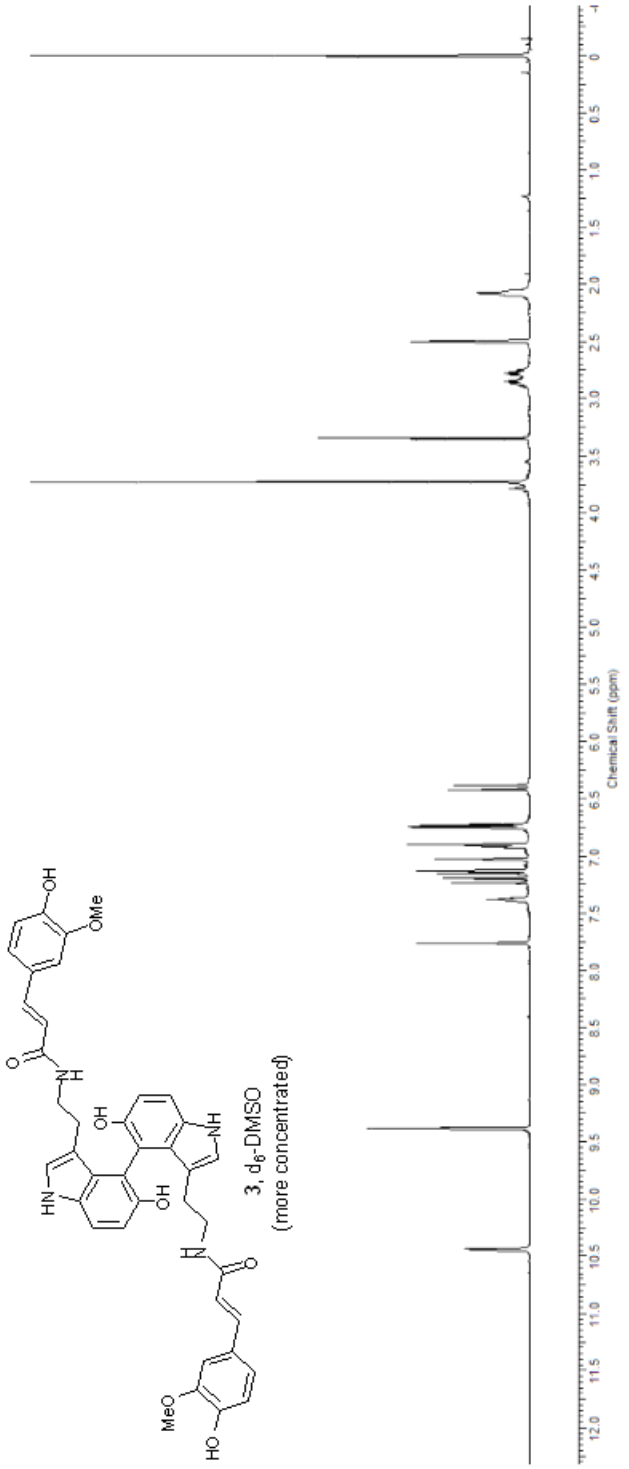




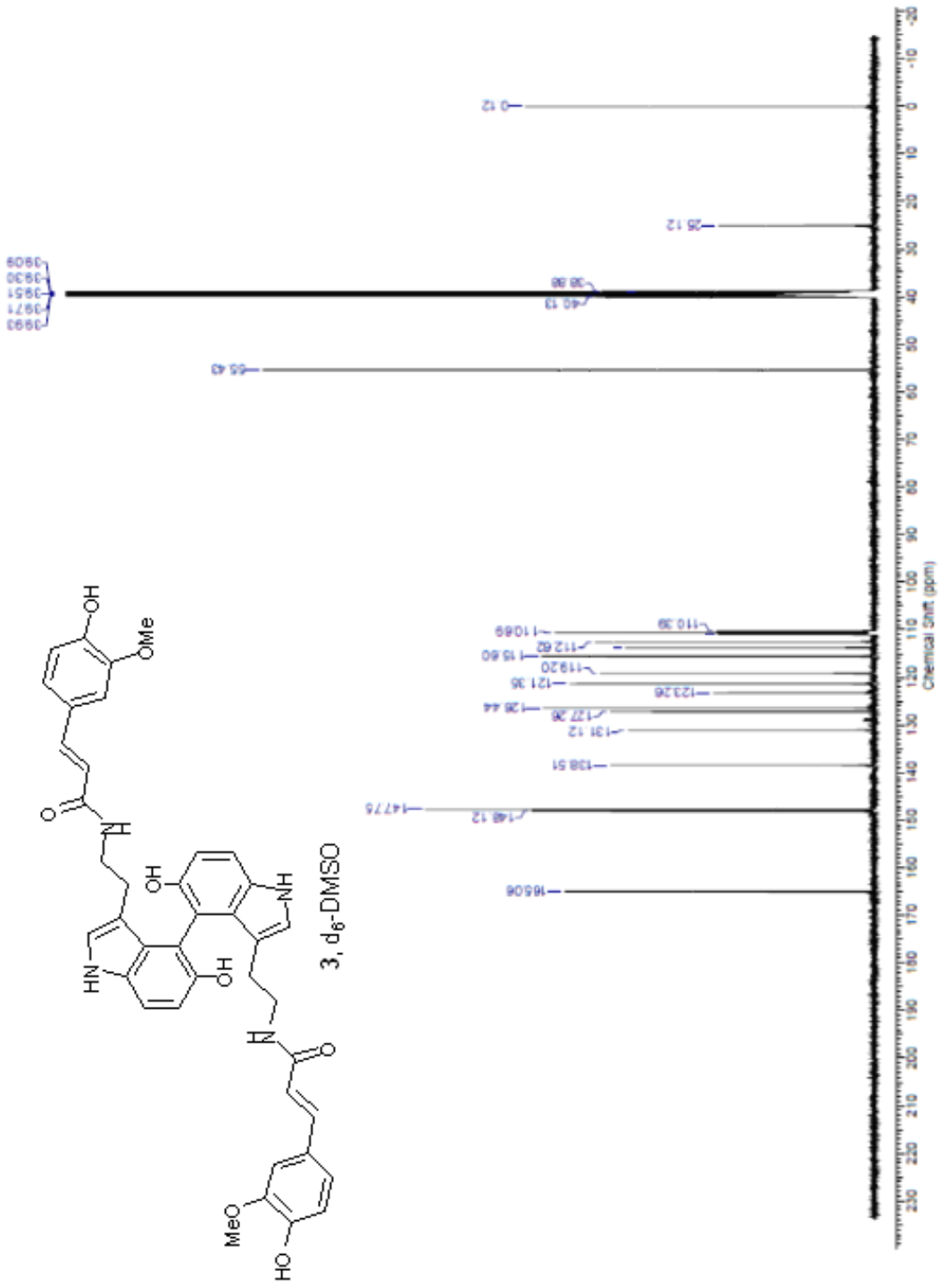




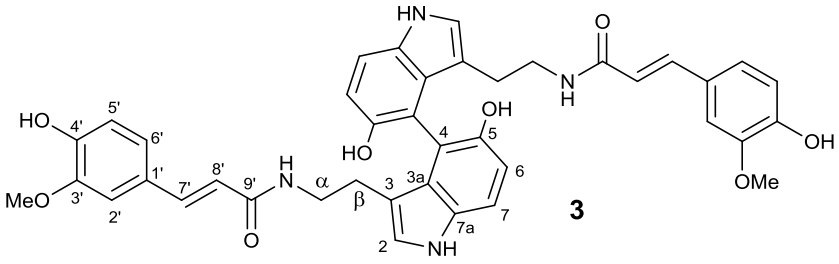
^{1}H NMR spectrum of 3 in DMSO-d_6: more concentrated sample



^{13}C NMR spectrum of 3 in DMSO- d_6



^{13}C NMR spectrum of 3 in DMSO- d_6: expanded spectral regions



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