Total Synthesis of (–)-Basiliskamide A and NMR Studies on the Conversion of Basiliskamide A to Basiliskamide B

Luiz C. Dias* and Caroline C. S. Gonçalves

Instituto de Química, Universidade Estadual de Campinas, CP 6154, 13084-971 Campinas-SP, Brazil



Figure S1. ¹H NMR of acetonide 6 (DMSO-*d*₆, 250 MHz).



Figure S2. ¹³C NMR of acetonide 6 (DMSO- d_6 , 62.5 MHz).



Figure S3. IR of acetonide 6.



Figure S4. ¹H NMR of diol 7 (DMSO-*d*₆, 250 MHz).



Figure S5. 13 C NMR of diol 7 (DMSO- d_6 , 62.5 MHz).









Figure S7. HRMS of diol 7.



Figure S8. ¹H NMR of basiliskamide A (1), (DMSO-*d*₆, 500 MHz).



Figure S9. ¹³C NMR of basiliskamide A (1), (DMSO- d_6 , 125 MHz).



Figure S10. IR of basiliskamide A (1).

Table 1. Comparison of ¹H and ¹³C NMR data for natural¹ and our synthetic basiliskamide A (DMSO-*d*₆)

¹ H NMR (400 MHz) δ /ppm (number of hydrogens, multiplicity, coupling constant) Natural	¹ H NMR (500 MHz) δ/ppm (number of hydrogens, multiplicity, coupling constant) Synthetic	13 C NMR (100 MHz) ¹ δ (ppm) Natural	¹³ C NMR (125 MHz) ¹ δ (ppm) Synthetic
0.84 (3H, d, 7.0 Hz)	0.84 (3H, d, 7.1 Hz)	10.1	10.1
0.87 (3H, t, 7.5 Hz)	0.87 (3H, t, 7.7 Hz)	11.6	11.7
0.90 (3H, d, 7.0 Hz)	0.89 (3H, d, 6.6 Hz)	12.8	12.8
1.11 (1H, m)	1.11 (1H, m)	26.4	26.4
1.25 (1H, m)	1.25 (1H, m)	34.7	34.7
1.67 (1H, m)	1.67 (1H, m)	35.5	35.5
1.99 (1H, m)	2.02 (1H, m)	40.7	40.8
2.06 (1H, m)	2.04 (1H, m)	69.6	69.7
2.28 (1H, m)	2.28 (1H, m)	76.3	76.3
3.49 (1H, m)	3.48 (1H, m)	118.0	118.0
4.57 (1H, d, 5.0 Hz)	4.58 (1H, d, 5.4 Hz)	119.3	119.4
4.92 (1H, dd, 2.0 and 9.5 Hz)	4.92 (1H, dd, 2.6 and 9.5 Hz)	128.2	128.3
5.55 (1H, d, 11.0 Hz)	5.55 (1H, d, 11.5 Hz)	128.4	128.4
5.91 (1H, dt, 7.0 and 15.0 Hz)	5.91 (1H, dt, 7.4 and 14.9 Hz)	128.9	128.9
6.31 (1H, dd, 11.0 and 11.0 Hz)	6.31 (1H, apt, 11.3 Hz)	130.4	130.4
6.61 (1H, d, 16.0 Hz)	6.62 (1H, d, 15.9 Hz)	134.0	134.0
6.83 (1H, s)	6.84 (1H, brs)	140.5	140.51
7.31 (1H, s)	7.33 (1H, brs)	-	140.56
7.40 (1H, dd, 11.0 and 15.0 Hz)	7.39 (1H, m)	144.6	144.6
7.40 (1H, m)	7.41 (3H, m)	166.0	166.1
7.41 (2H, m)	-	167.4	167.5
7.65 (1H, d, 16.0 Hz)	7.66 (1H, d, 16.0 Hz)	-	-
7.71 (2H, m)	7.72 (2H, m)	-	-

1. Barsby, T.; Kelly, M. T.; Andersen R. J.; J. Nat. Prod. 2002, 65, 1447; Kelly, M. T.; Anderson, R. J.; Barsby, T.; US Patent 2005/0277779 A1, 2005.