SUPPLEMENTARY MATERIAL

The structure proposed for apteniol D is different from that of the compound obtained by total synthesis

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ABSTRACT

We describe the synthesis of 4,4'-oxyneolignan, the proposed structure for naturally occurring apteniol D. The diphenyl ether moiety in 4,4'-oxyneolignan was formed via classical Ullmann ether synthesis using excess copper powder in *N,N*-dimethylacetamide. The spectral data of synthesized apteniol D show differences compared to those of naturally occurring apteniol D.

Keywords: *Aptenia cordifolia*, diphenyl ether, Ullmann ether synthesis, apteniol

Supplemental file (Figure) Legends

Table S1. ^1H - and ^{13}C -NMR data for synthesized and naturally occurring apteniol D.

Figure S1. ^1H NMR spectrum of Compound **8**. (CDCl_3 , 400 MHz)

Figure S2. ^{13}C NMR spectrum of Compound **8**. (CDCl_3 , 100 MHz)

Figure S3. ^1H NMR spectrum of Compound **9**. (CDCl_3 , 400 MHz)

Figure S4. ^{13}C NMR spectrum of Compound **9**. (CDCl_3 , 100 MHz)

Figure S5. ^1H NMR spectrum of Compound **10**. (CDCl_3 , 400 MHz)

Figure S6. ^{13}C NMR spectrum of Compound **10**. (CDCl_3 , 100 MHz)

Figure S7. ^1H NMR spectrum of Compound **4**. (CDCl_3 , 400 MHz)

Figure S8. ^{13}C NMR spectrum of Compound **4**. (CDCl_3 , 100 MHz)

Table S1. ^1H - and ^{13}C -NMR data^a for synthesized and naturally occurring apteniol D.

position	synthesized apteniol D ^b		reported apteniol D ^c	
	$\delta(\text{H})$	$\delta(\text{C})$	$\delta(\text{H})$	$\delta(\text{C})$
1	-	137.4	-	133.1
2	6.48 s	105.5	6.44 s	104.8
3	-	153.4	-	146.9
4	-	131.9	-	131.9
5	-	153.4	-	146.9
6	6.48 s	105.5	6.44 s	104.8
7	2.95 t (7.2)	31.0	2.90 t (7.5)	30.7
8	2.72 t (7.2)	35.4	2.67 t (7.5)	35.7
9	-	178.2	-	177.8
1'	-	129.3	-	131.2
2'	6.76 d (3.1)	111.9	6.72 s	110.8
3'	-	154.0	-	146.9
4'	-	150.8	-	144.0
5'	6.37 d (8.8)	113.5	6.82 d (8.5)	114.3
6'	6.54 dd (3.1, 8.8)	116.0	6.70 d (8.5)	120.7
7'	3.09 t (7.8)	26.3	2.90 t (7.5)	30.2
8'	2.89 t (7.8)	33.6	2.67 t (7.5)	35.7
9'	-	179.2	-	177.8
3-OMe	3.72 s	56.1	3.87 s	56.1
5-OMe	3.72 s	56.1	3.87 s	56.1
3'-OMe	3.72 s	55.6	3.87 s	55.7

^a The coupling constants (J in Hz) are in parentheses. ^b ^1H and ^{13}C NMR data were recorded at 400 and 100 MHz, respectively, in CDCl_3 .

^c DellaGreca et al. 2005. Tetrahedron. **61**, 11924-11929. ^1H and ^{13}C NMR data were recorded at 500 and 125 MHz, respectively, in CDCl_3 .

Figure.S1

Figure.S2

Figure.S3

Figure.S4

Figure.S5

Figure.S6

Figure.S7

Figure.S8