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## Antiostatin $A_1$ (1a)



C- or N-atom no., number of protons	<sup>1</sup> H NMR data of natural A <sub>1</sub> (acetone-d <sub>6</sub> )	<sup>1</sup> H NMR data of synthetic A <sub>1</sub> (acetone-d <sub>6</sub> )
1a, 2 H	2.97 (t)	3.00 (m)
1b, 1c, 1d, 3 x 2 H	1.37, 1.45, 1.65 (m)	1.37–1.42, 1.43–1.51, 1.65–1.71 (m)
1e, 3 H	0.89 (t)	0.92 (t)
3, OH	8.05 (s)	8.08 and 8.10 (s)
4c, NH	9.68 (br s)	9.72 (br s)
4e, 3 H	2.47 (s)	2.50 (s)
5, 1 H	8.13 (d)	8.16 (d)
6, 1 H	7.10 (t)	7.12 (t)
7, 1 H	7.30 (t)	7.33 (t)
8, 1 H	7.45 (d)	7.46 (d)
9, NH	10.16 (br s)	10.21 (br s)
10, 3 H	2.40 (s)	2.41 (s)

C-atom no.	<sup>13</sup> C NMR data natural A <sub>1</sub> (acetone-d <sub>6</sub> )	<sup>13</sup> C NMR data of synthetic A <sub>1</sub> (acetone-d <sub>6</sub> )
1	123.3 (C)	122.90 (C)
1a	30.0 (CH <sub>2</sub> )	29.12 (CH <sub>2</sub> )
1b	30.1 (CH <sub>2</sub> )	30.05 (CH <sub>2</sub> )
1c	33.1 (CH <sub>2</sub> )	32.77 (CH <sub>2</sub> )
1d	23.5 (CH <sub>2</sub> )	23.35 (CH <sub>2</sub> )
1e	13.0 (CH <sub>3</sub> )	12.68 (CH <sub>3</sub> )
2	125.8 (C)	125.46 (C)
3	144.3 (C)	143.95 (C)
4	118.3 (C)	117.95 (C)
4a	115.5 (C)	115.07 (C)
4b	123.8 (C)	123.39 (C)
4d	172.1 (C)	171.78 (C)
4e	23.7 (CH <sub>3</sub> )	23.18 (CH <sub>3</sub> )
5	122.8 (CH)	122.51 (CH)
6	119.2 (CH)	118.85 (CH)
7	125.8 (CH)	125.36 (CH)
8	111.8 (CH)	111.48 (CH)
8a	141.4 (C)	140.98 (C)
9a	135.1 (C)	134.69 (C)
10	14.6 (CH <sub>3</sub> )	14.35 (CH <sub>3</sub> )

## Antiostatin $A_2$ (1b)



C- or N-atom no., number of protons	<sup>1</sup> H NMR data of natural A <sub>2</sub> (acetone-d <sub>6</sub> )	<sup>1</sup> H NMR data of synthetic A <sub>2</sub> (acetone-d <sub>6</sub> )
1a, 2 H	3.00 (m)	3.00 (m)
1c, 1 H	1.23–1.30 (m)	1.23–1.30 (m)
1b, 2 H; 1d, 2 H	1.46 –1.58 (m); 1.64–1.71 (m)	1.45–1.59 (m); 1.65–1.71 (m)
1e, 3 H	0.94 (t)	0.95 (t)
1f, 3 H	1.05 (d)	1.06 (d)
3, OH	8.08 (br s)	_
4c, NH	9.78 (br s)	9.74 (br s)
4e, 3 H	2.50 (s)	2.51 (s)
5, 1 H	8.16 (d)	8.17 (d)
6, 1 H	7.12 (dt)	7.12 (dt)
7, 1 H	7.33 (dt)	7.33 (dt)
8, 1 H	7.47 (d)	7.47 (d)
9, NH	10.23 (br s)	10.20 (br s)
10, 3 H	2.42 (s)	2.42 (s)

## Antiostatin B<sub>4</sub> (2c)



C-atom no.	<sup>13</sup> C NMR data of natural B <sub>4</sub> (acetone-d <sub>6</sub> )	<sup>13</sup> C NMR data of synthetic B <sub>4</sub> (acetone-d <sub>6</sub> )
1, 4b	123.7 and 123.7 (2 C)	122.81 and 122.92 (2 C)
1a	29.7 (CH <sub>2</sub> )	30.05 (CH <sub>2</sub> )
1b	31.1 (CH <sub>2</sub> )	30.57 (CH <sub>2</sub> )
1c	31.0 (CH <sub>2</sub> )	30.47 (CH <sub>2</sub> )
1d	30.6 (CH <sub>2</sub> )	29.17 (CH <sub>2</sub> )
1e	33.2 (CH <sub>2</sub> )	32.64 (CH <sub>2</sub> )
1f	23.8 (CH <sub>2</sub> )	23.29 (CH <sub>2</sub> )
1g	14.9 (CH <sub>3</sub> )	14.33 (CH <sub>3</sub> )
2	126.0 (C)	125.52 (C)
3	143.9 (C)	142.99 (C)
4	117.6 (C)	116.67 (C)
4a	115.7 (C)	114.90 (C)
4d	155.9 (C=O)	154.73 (C=O)
4f	156.8 (C=O)	155.99 (C=O)
4h	48.3 (CH <sub>2</sub> )	47.58 (CH <sub>2</sub> )
4i	30.1 (CH)	29.51 (CH)
4j	20.7 (CH <sub>3</sub> )	20.21 (CH <sub>3</sub> )
4k	20.7 (CH <sub>3</sub> )	20.21 (CH <sub>3</sub> )
5	122.6 (CH)	121.99 (CH)
6	119.7 (CH)	119.11 (CH)
7	126.0 (CH)	125.44 (CH)
8	112.1 (CH)	111.45 (CH)
8a	141.8 (C)	140.84 (C)
9a	135.3 (C)	134.71 (C)
10	13.3 (CH <sub>3</sub> )	12.78 (CH <sub>3</sub> )

## Melting points:

antiostatin	natural product	synthetic product
$A_1$ ( <b>1a</b> )	180–183 °C	190–192 °C
$A_2(\mathbf{1b})$	195–197 °C	185 °C
A <sub>3</sub> ( <b>1c</b> )	190–192 °C	191–192 °C
$A_4(\mathbf{1d})$	191–193 °C	180–183 °C
$B_2(2a)$	119–120 °C	119–120 °C
B <sub>3</sub> ( <b>2b</b> )	117–118 °C	119–120 °C
B <sub>4</sub> ( <b>2c</b> )	118–120 °C	117–120 °C
$B_5(2d)$	92–94 °C	92 °C