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Supplementary material

Additional bioactive guanidine alkaloids from the Mediterranean sponge *Crambe crambe*

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NMR spectra

| Crambescin A2 (1) | |
|--|------|
| ¹ H NMR spectrum (500 MHz, CD3OD) | S-2 |
| ¹³ C NMR spectrum (125 MHz, CD3OD) | S-3 |
| ¹ H- ¹ H COSY spectrum (500 MHz, CD3OD) | S-4 |
| Crambescin A1 (4) | |
| ¹ H NMR spectrum (500 MHz, CD3OD) | S-5 |
| ¹³ C NMR spectrum (125 MHz, CD3OD) | S-6 |
| ¹ H- ¹ H COSY spectrum (500 MHz, CD3OD) | S-7 |
| Didehydrocrambescin A1 (5) | |
| ¹ H NMR spectrum (500 MHz, CD3OD) | S-8 |
| ¹ H- ¹ H COSY spectrum (500 MHz, CD3OD) | S-9 |
| Crambescin B1 (7) | |
| ¹ H NMR spectrum (500 MHz, CD3OD) | S-10 |
| ¹³ C NMR spectrum (125 MHz, CD3OD) | S-11 |
| ¹ H- ¹ H COSY spectrum (500 MHz, CD3OD) | S-12 |
| Crambescin C1 (10) | |
| ¹ H NMR spectrum (500 MHz, CD3OD) | S-13 |
| ¹ H- ¹ H COSY spectrum (500 MHz, CD3OD) | S-14 |
| ¹ H- ¹³ C HMBC spectrum (500 MHz, CD3OD) | S-15 |
| ¹ H NMR spectra of crambescins A2 (1-3) and crambescin A1 (4) | S-16 |
| ¹ H NMR spectra of crambescins B2 (6-8) | S-16 |
| ¹ H NMR spectra of crambescins C1 (9-11) | S-17 |
| ¹ H NMR spectrum of crambescidine 816 (12) | S-18 |
| ¹³ C NMR spectrum of crambescidine 816 (12) | S-19 |

HRMS/MS data

| S-20 |
|------|
| S-20 |
| S-21 |
| S-21 |
| S-22 |
| S-22 |
| S-23 |
| S-23 |
| S-24 |
| S-24 |
| S-25 |
| |





¹³C NMR spectrum (125 MHz, CD3OD) of crambescin A2 (1)



¹H-¹H COSY spectrum (500 MHz, CD3OD) of crambescin A2 (1)



¹H NMR spectrum (500 MHz, CD3OD) of crambescin A1 (4)



¹³C NMR spectrum (125 MHz, CD3OD) of crambescin A1 (4)



¹H-¹H COSY spectrum (500 MHz, CD3OD) of crambescin A1 (4)





¹H-¹H COSY spectrum (500 MHz, CD3OD) of didehydrocrambescin A1 (5)





¹³C NMR spectrum (125 MHz, CD3OD) of crambescin B1 (7)



¹H-¹H COSY spectrum (500 MHz, CD3OD) of crambescin B1 (7)



¹H NMR spectrum (500 MHz, CD3OD) of crambescin C1 (10)



¹H-¹H COSY spectrum (500 MHz, CD3OD) of crambescin C1 (10)



¹H-¹³C HMBC spectrum (500 MHz, CD3OD) of crambescin C1 (10)



¹H NMR spectra of crambescins A2 (1-3) and crambescin A1 (4)







¹H NMR spectra of crambescins C1 (9-11)





¹³ C NMR spectrum of crambescidin 816 (12)





HRMS/MS spectrum of crambescin A1 (4)





198.19643

200

142.13374

150

0

300 m/z 368.26563

350

400

322.29929

249.18483

250

464.10568

450

• • • •



HRMS/MS spectrum of crambescin B1 (7)



HRMS/MS spectrum of crambescin B1 (8)









HRMS/MS spectrum of crambescin C1 (10)



HRMS/MS spectrum of crambescin C1 (11)