#### An Efficient Solid-Phase Strategy for Total Synthesis of Naturally Occurring Amphiphilic Marine Siderophores: Amphibactin-T and Moanachelin ala-B

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Crude HPLC profile of Fmoc-L-Orn-OBn•TFA (3)

Fmoc-Orn-OBn.TFA on 5 g scale



HPLC Conditions

Column: Phenomenex column C18 (3  $\mu$ m x 4.6 x 50 mm) Eluent: 5 - 95% CH<sub>3</sub>CN / 0.1% TFA in 15 min Sol. A:100% H<sub>2</sub>O/0.1% TFA, Sol. B: 100% CH<sub>3</sub>CN/0.1% TFA Wavelength: 220 nm Flow rate: 1 mL/ min



<sup>1</sup>H NMR & <sup>13</sup>C NMR copies of Fmoc-L-Orn-OBn•TFA (**3**)

## HRMS (ESI-QTOF) profile of Fmoc-L-Orn-OBn•TFA (3); Calc. for $C_{27}H_{28}N_2O_4$ 445.2121, found 445.2167.





<sup>1</sup>H NMR & <sup>13</sup>C NMR copies of Fmoc-L-Orn(Ac,OBz)-OBn (4)

# HRMS (ESI-QTOF) profile of Fmoc-L-Orn(Ac,OBz)-OBn (4); Calc. for $C_{36}H_{34}N_2O_7 607.2438$ , found 607.2403.





<sup>1</sup>H NMR & <sup>13</sup>C NMR copies of Fmoc-L-Orn(Bz)-OBn (5)

### HRMS (ESI-QTOF) profile of Fmoc-L-Orn(Bz)-OBn (**5**); Calc. for $C_{34}H_{32}N_2O_5$ 549.2383, found 549.2342.





<sup>1</sup>H NMR & <sup>13</sup>C NMR copies of Fmoc-L-Orn(Ac,OBz)-OH (1)

# HRMS (ESI-QTOF) profile of Fmoc-L-Orn(Ac,OBz)-OH (1); Calc. for Calc. for $C_{29}H_{28}N_2O_7$ 517.1969, found 517.1975.



**Model peptide:** DA-Lys-Ser-Orn(Ac,OBz)-OH; Calc. for  $C_{33}H_{53}N_5O_9$  663.3843, found 664; DA-Lys-Ser-Orn(Ac,OH)-OH; Calc. for  $C_{26}H_{49}N_5O_8$  559.3581, found 560 (M+1).



Purified HPLC profile of amphibactin-T

After prep-HPLC



Prep-HPLC Conditions

Column: Phenomenex C18 column (10  $\mu$ m x 10 x 250 mm) Eluent: 5 - 60% CH<sub>3</sub>CN / 0.1% TFA in 30 min Sol. A: 100% H<sub>2</sub>O/0.1% TFA, Sol. B: 100% CH<sub>3</sub>CN/0.1% TFA Wavelength: 220 nm Flow rate: 7 mL/ min

## HRMS (ESI-QTOF) profile of amphibactin-T; Calc. for $C_{36}H_{65}N_7O_{13}$ 804.4713, found 804.4707.



Purified HPLC profile of moanachelin ala-B

After prep-HPLC



**Prep-HPLC Conditions** 

Column: Phenomenex C18 column (10  $\mu$ m x 10 x 250 mm) Eluent: 5 - 60% CH<sub>3</sub>CN / 0.1% TFA in 30 min Sol. A: 100% H<sub>2</sub>O/0.1% TFA, Sol. B: 100% CH<sub>3</sub>CN/0.1% TFA Wavelength: 220 nm Flow rate: 7 mL/ min

# HRMS (ESI-QTOF) profile of moanachelin ala-B; Calc. for $C_{36}H_{65}N_7O_{12}$ 788.4763, found 788.4770.

