Electronic Supplementary Material (ESI) for MedChemComm.

Crystallomycin revisited after 60 years: aspartocins B and C

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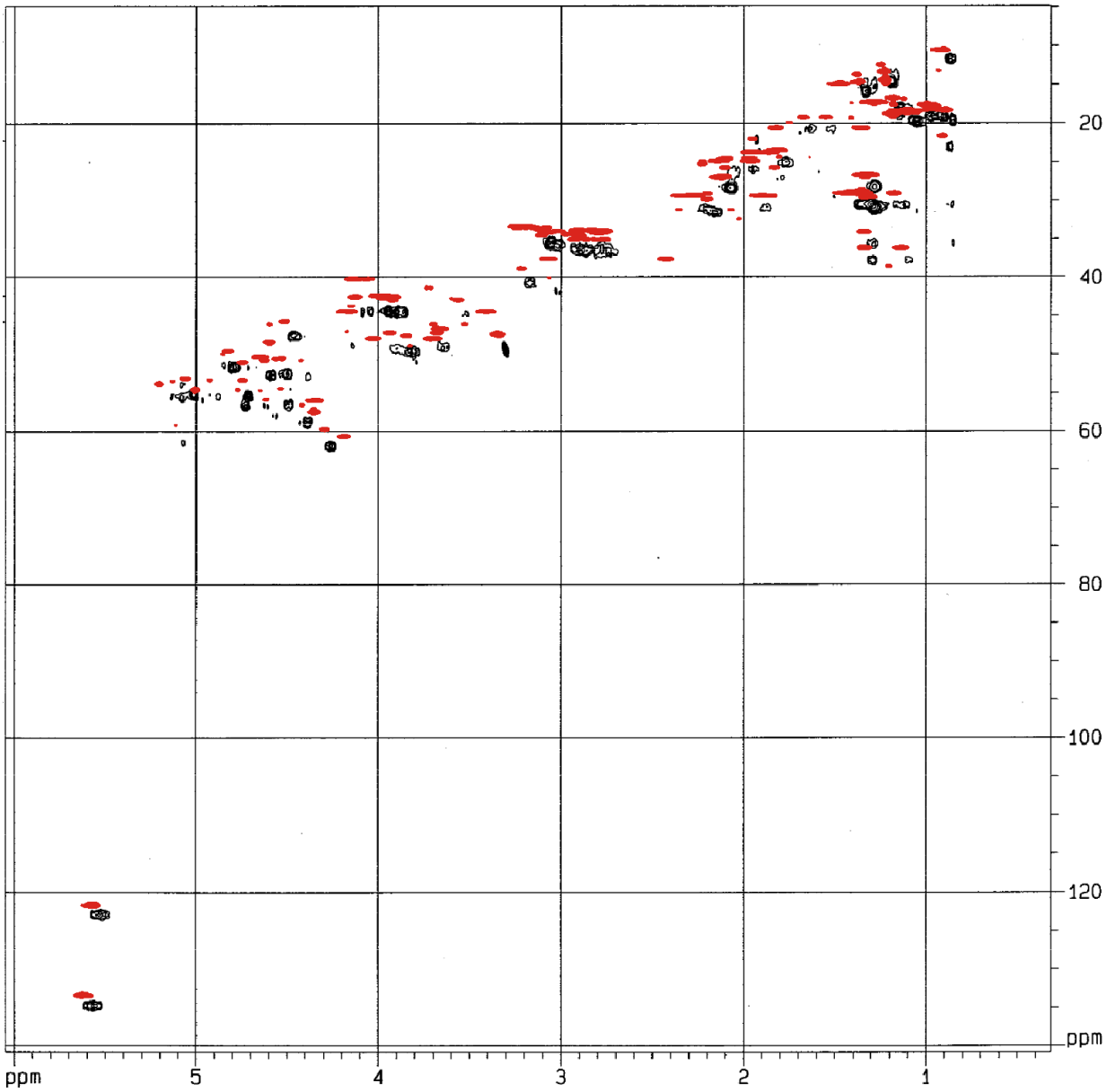
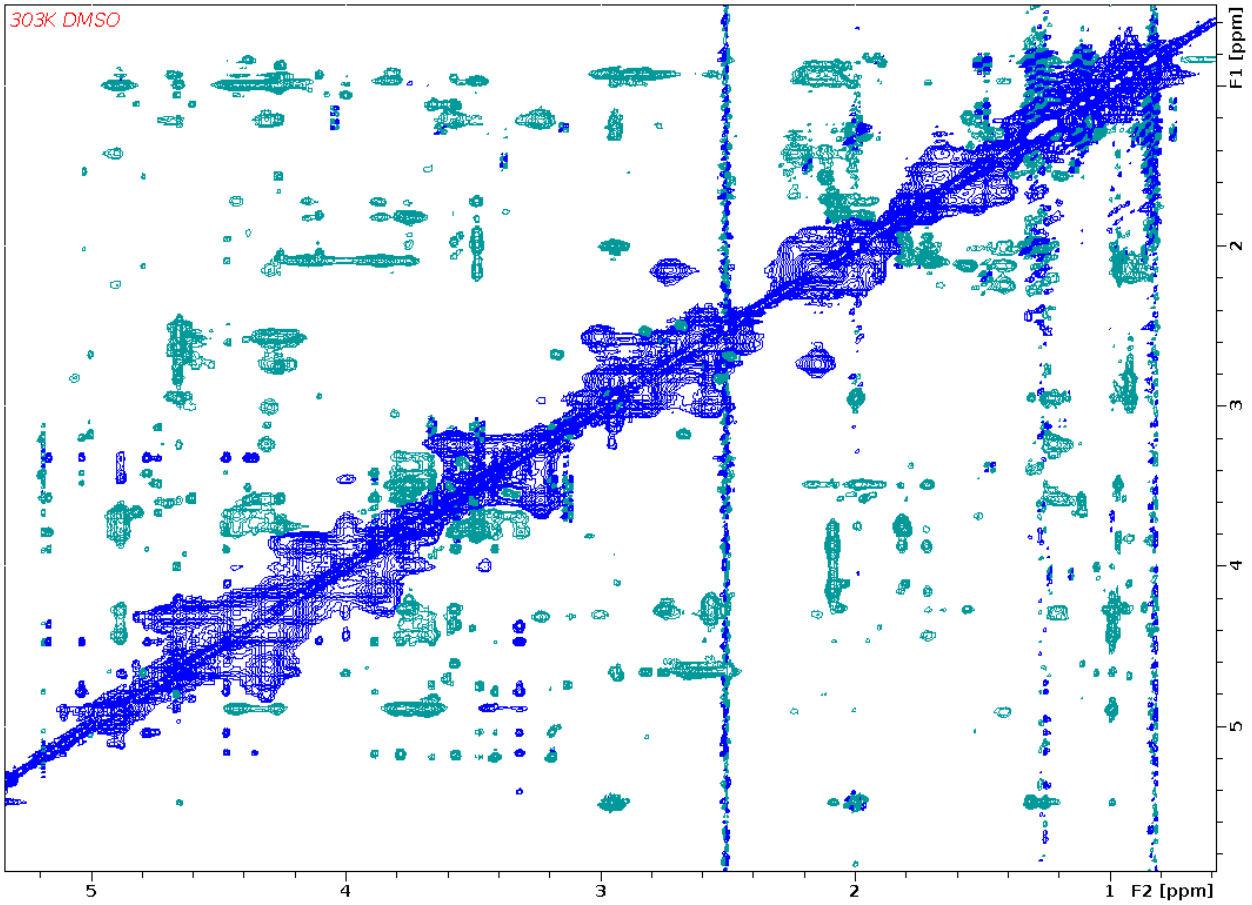
Fig. S1. Comparison of ^{13}C-HSQC spectra of aspartocin B (black contours, CD_3OD, 27°C, 400 MHz) and Cryst-2 (red contours, CD_3OD, 30°C, 800 MHz).



The spectrum of aspartocin B was taken from Supplementary Materials of (F. Kong, K. Janota, J.S. Ashcroft and G.T. Carter, *Rec. Nat. Prod.*, 2010, **4**, 131). The shift of the resonances in the ^{1}H and ^{13}C dimensions is probably originated by differences in experimental conditions (temperature, ionic strength, pH, NMR spectrometer frequency) and differences in the reference method used.

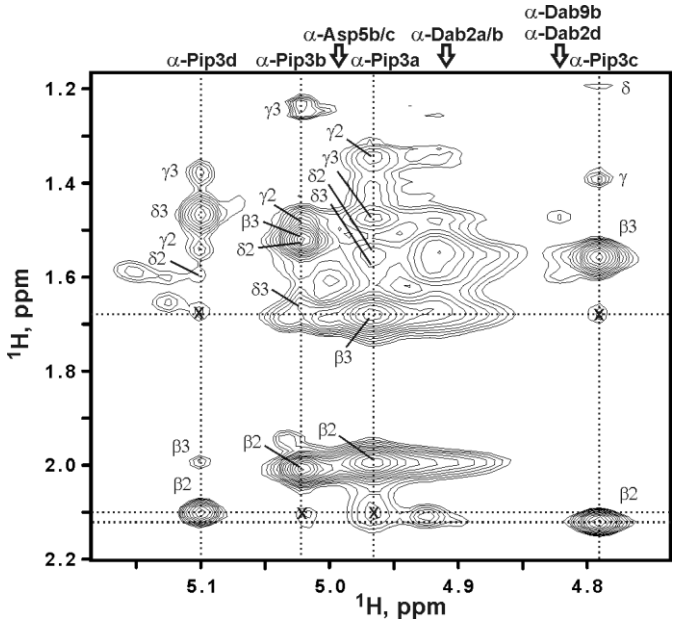
Fig. S2. The aliphatic fragment of 2D ^{1}H,^{1}H-ROESY spectrum of Cryst-2 (t_m = 200 ms, DMSO-d_6, 30°C, 800 MHz).



The blue contours (positive intensity) correspond to diagonal cross-peaks and peaks originated by conformational exchange processes. The green contours (negative intensity) correspond to NOE cross-peaks. The spectral regions near the diagonal are completely covered by exchange cross-peaks between four structural states of Cryst-2. Due to extreme crowding these peaks are run into the large blurs.

The chain of exchange cross-peaks at the water frequency (3.3 ppm) probably corresponds to OH-groups from carbohydrate impurity in the sample. These resonances are not connected with the spin-systems of Cryst-2 in NOESY, ROESY and TOCSY spectra. The Crist-2 compound itself does not contain OH-groups.

Fig. S3. The fragment of 2D ^{1}H,^{1}H-NOESY spectrum (tm = 200 ms, DMSO-d_6, 30°C, 800 MHz) that reveal the exchange processes between the four conformational sates (a,b,c,d) of Cryst-2 in solution.



The assignment of intraresidual NOEs for the Pip3 residue in the four Cryst-2 conformers is shown. The exchange originated (inter-conformer) cross-peaks are marked by crosses. The corresponding exchange cross-peaks have negative intensity in the ROESY spectrum (Fig. S2) and, therefore, could not be easily differentiated from NOE cross-peaks.

Fig. S4. HRESI-MS spectrum of Cryst-1 (**1**)

Analysis Info

Analysis Name	D:\Data\Ant-21 (Crys-1)_17_01_1044.d	Acquisition Date	5/24/2017 6:38:52 PM
Method	la_2.2.m	Operator	BDAL@DE
Sample Name	Ant-21 (Crys-1)	Instrument	compact 8255754.20088
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C

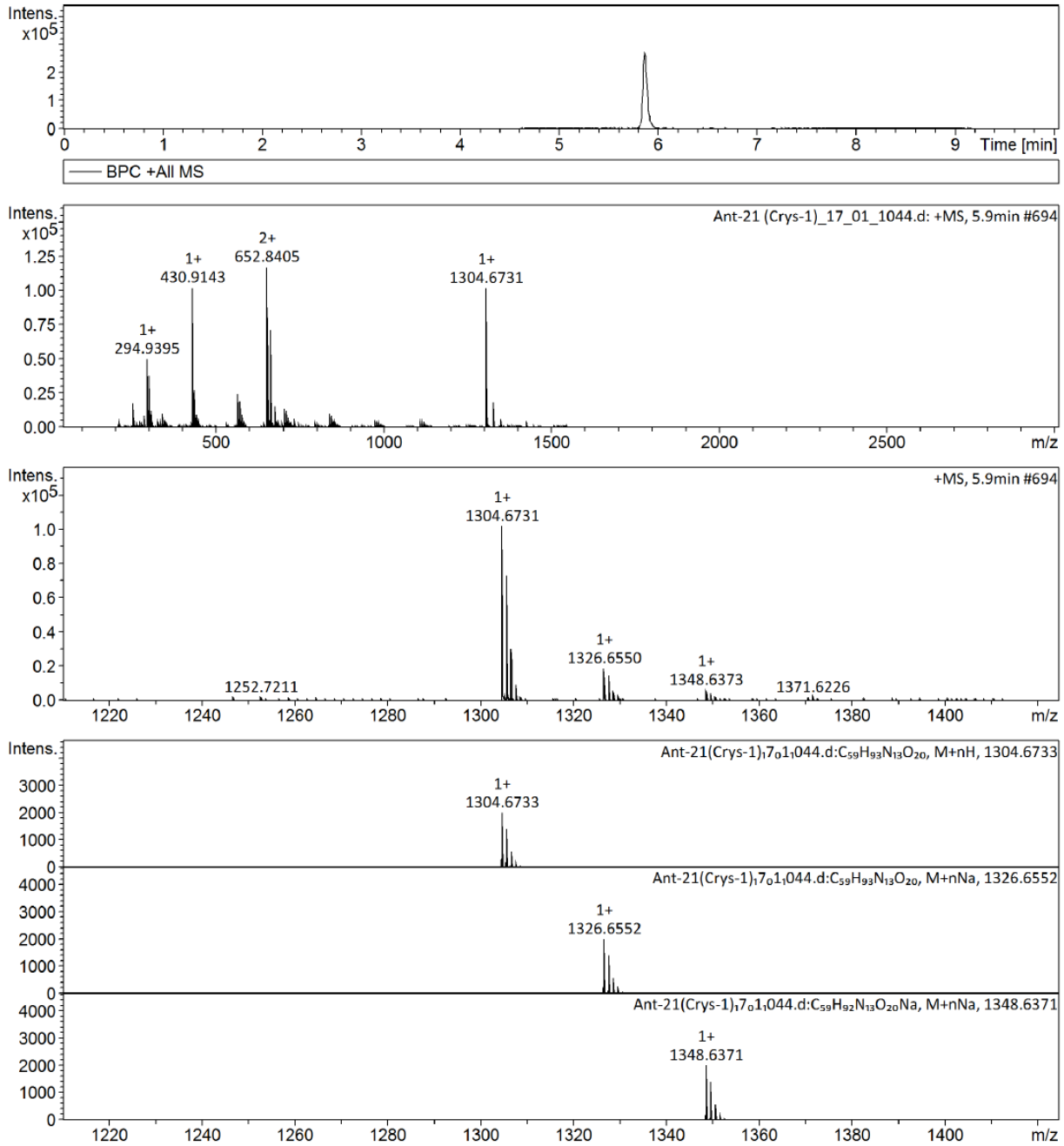


Fig. S5. HRESI-MS spectrum of Cryst-2 (**2**)

Analysis Info

Analysis Name	D:\Data\Ant-22 (Crys-2)_18_01_1045.d	Acquisition Date	5/24/2017 6:50:24 PM
Method	la_2.2.m	Operator	BDAL@DE
Sample Name	Ant-22 (Crys-2)	Instrument	compact 8255754.20088
Comment			

Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C

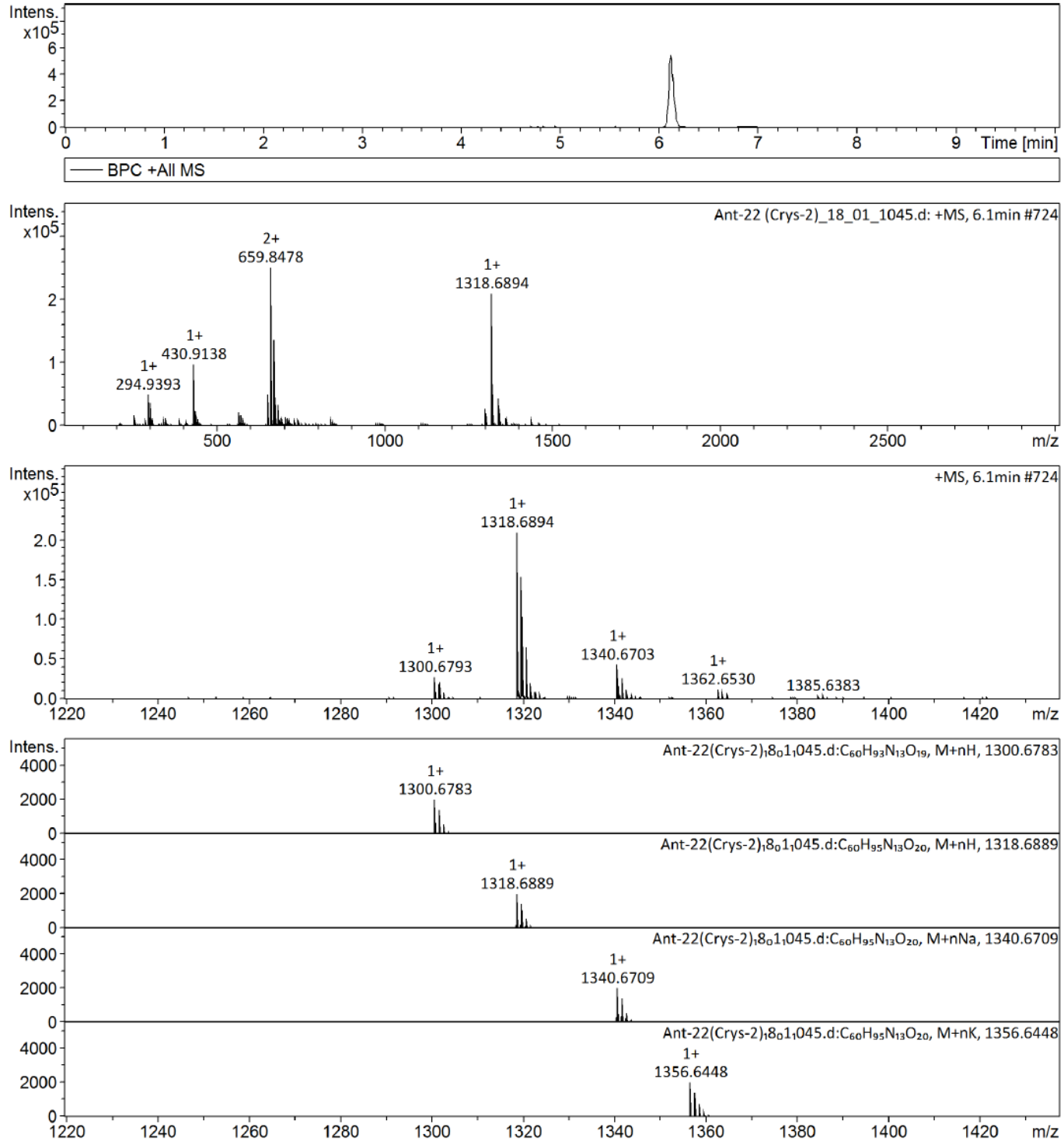

Fig. S6. HPLC-MS analysis of the extract from producing strain *Streptomyces griseorubens* INA 00887

Analysis Info

Analysis Name	D:\Data\Ant-44_12_01_1627.d	Acquisition Date	1/23/2018 1:08:23 PM
Method	la_2.2.m	Operator	BDAL@DE
Sample Name	Ant-44	Instrument	compact 8255754.20088
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	6.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C