

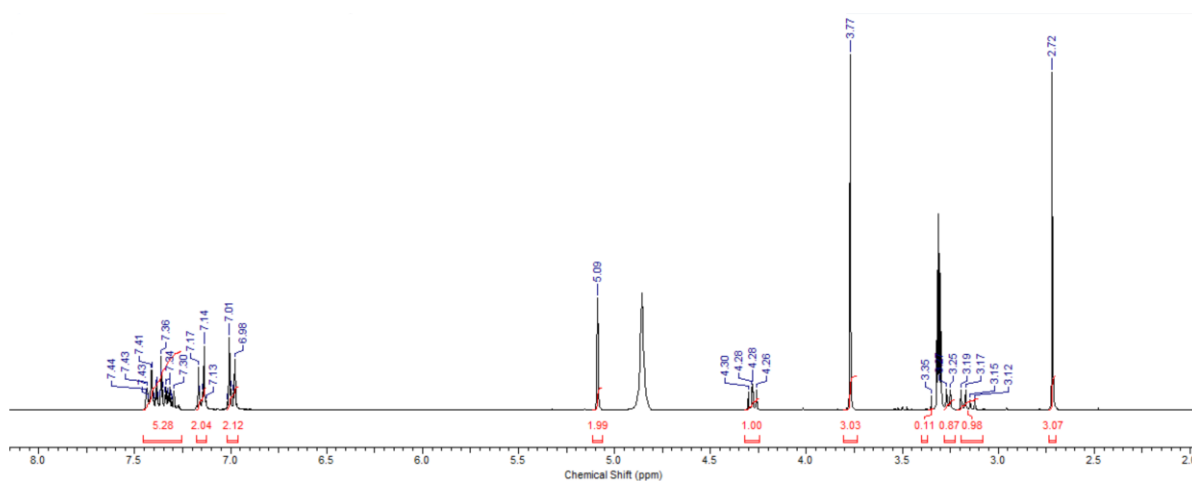
Supporting Information

An expeditious synthesis of the ascomycete metabolite rigidiusculamide B

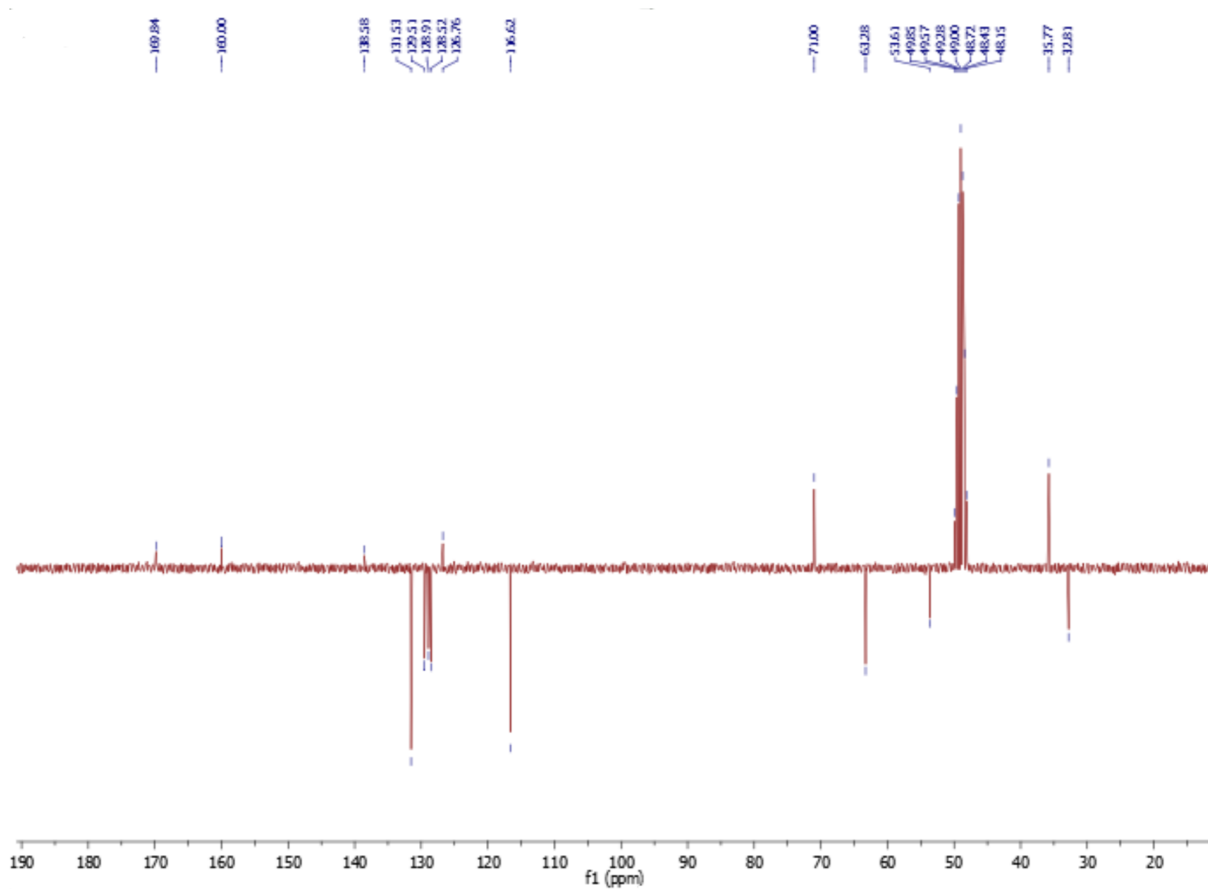
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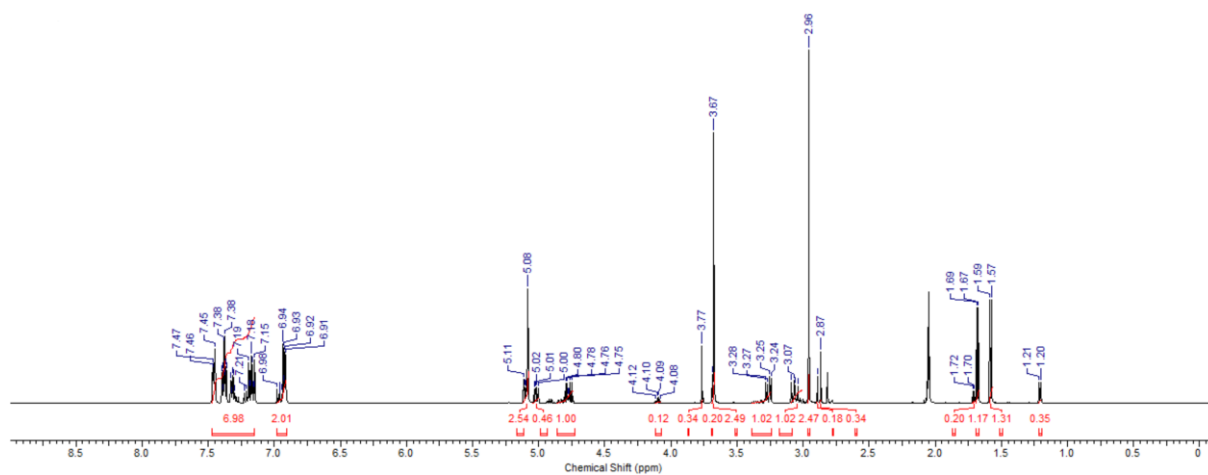
^1H NMR (300 MHz, MeOD) Methyl *N*-methyl-*O*-benzyl-L-tyrosinate hydrochloride (**7**)



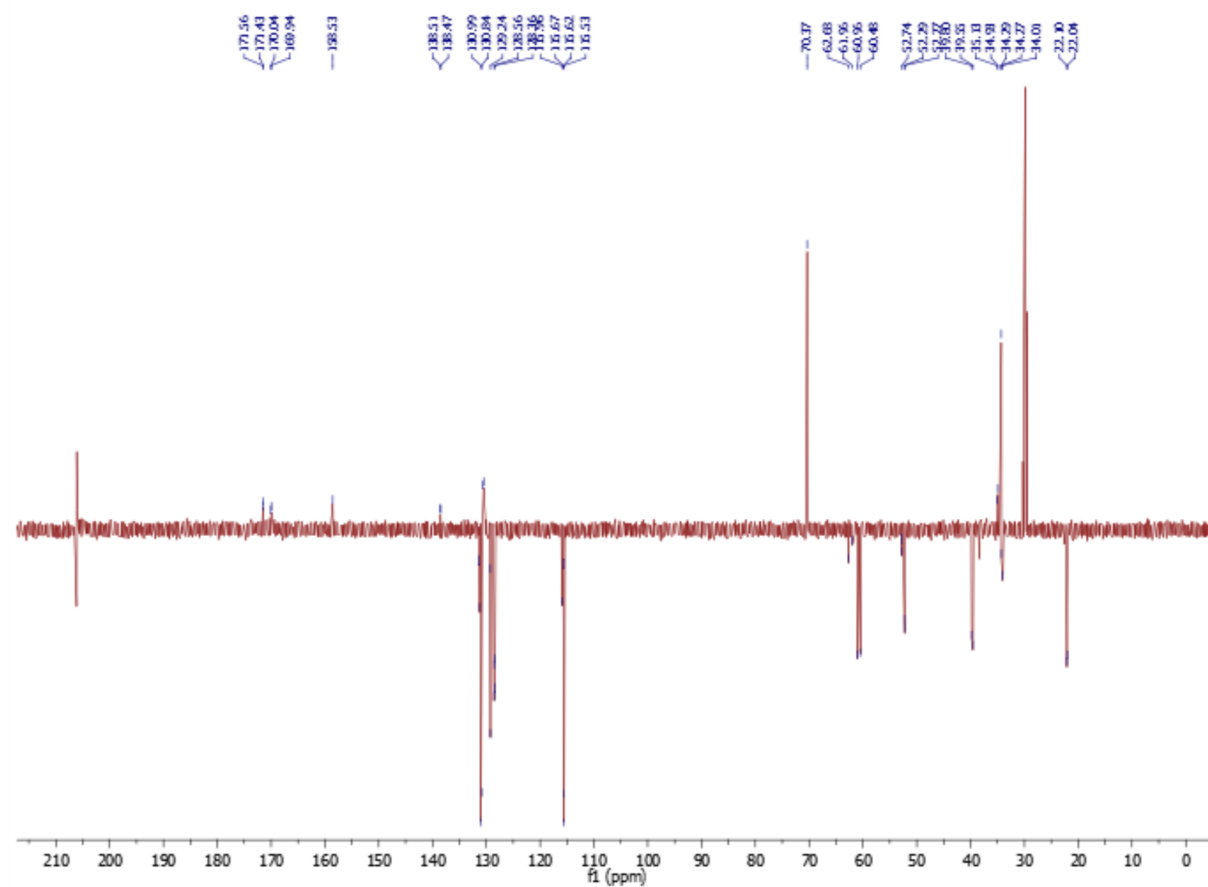
^{13}C NMR (75 MHz, MeOD) Methyl *N*-methyl-*O*-benzyl-L-tyrosinate hydrochloride (**7**)



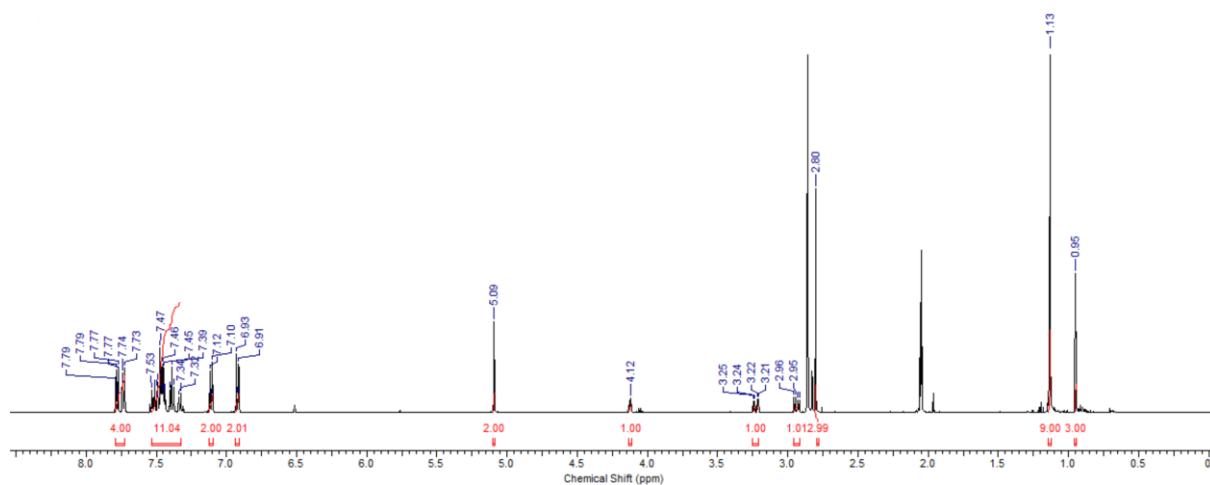
^1H NMR (500 MHz, $(\text{CD}_3)_2\text{CO}$) (*S*)-Methyl 3-(4-(benzyloxy)phenyl)-2-(2-bromo-*N*-methylpropanamido)propanoate (**9**)



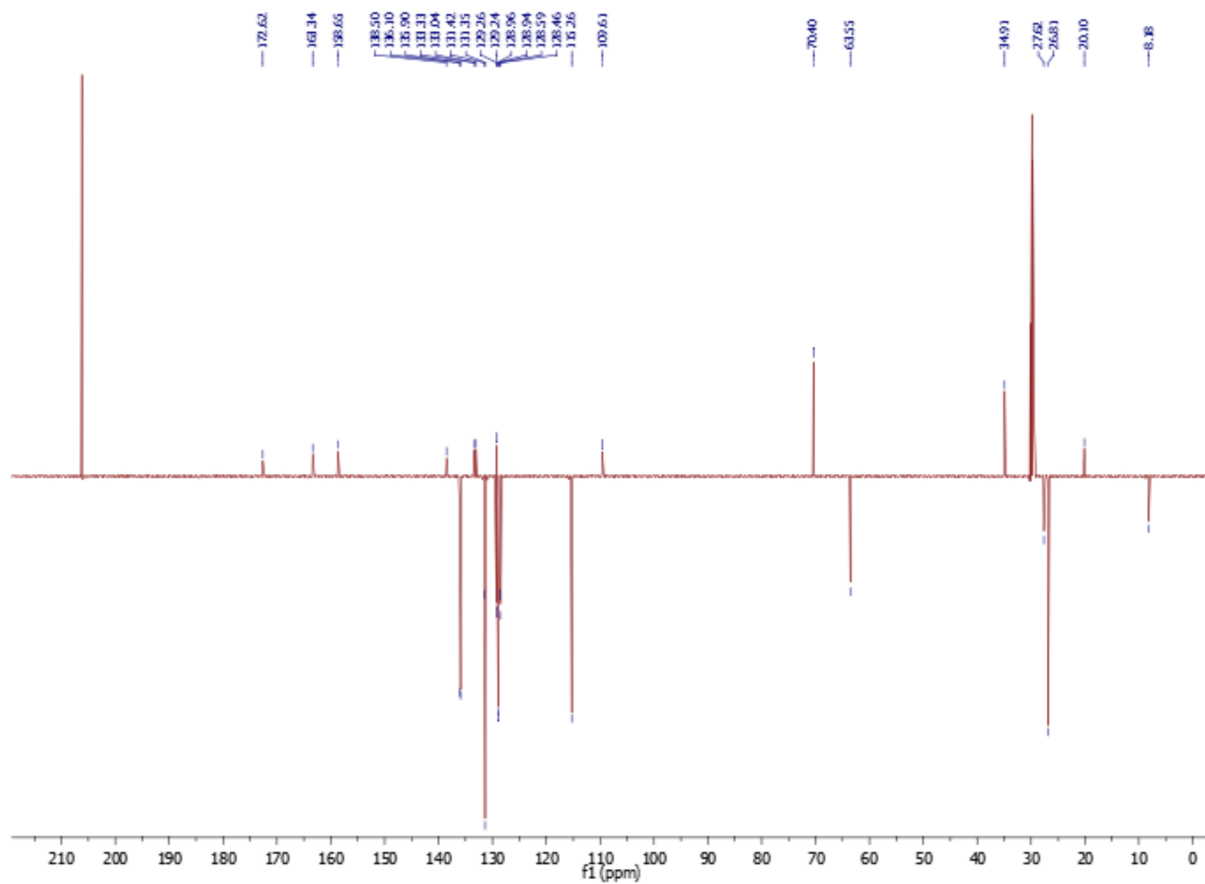
^{13}C NMR (125 MHz, $(\text{CD}_3)_2\text{CO}$) (*S*)-Methyl 3-(4-(benzyloxy)phenyl)-2-(2-bromo-*N*-methylpropanamido)propanoate (**9**)



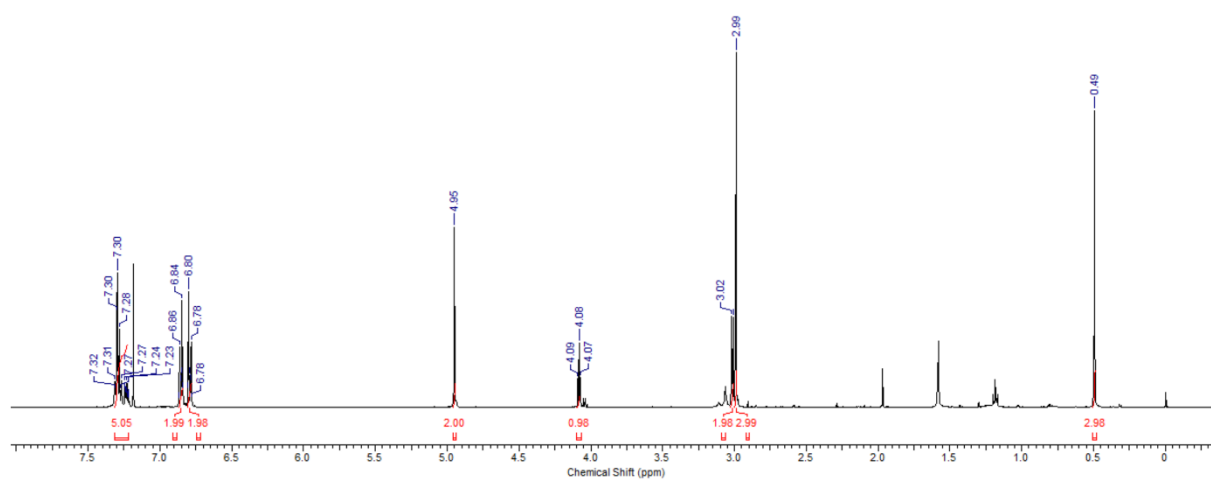
^1H NMR (500 MHz, $(\text{CD}_3)_2\text{CO}$) (S)-5-(4-(Benzyloxy)benzyl)-4-(*tert*-butyldiphenylsilyloxy)-1,3-dimethyl-1*H*-pyrrol-2(5*H*)-one (**10**)



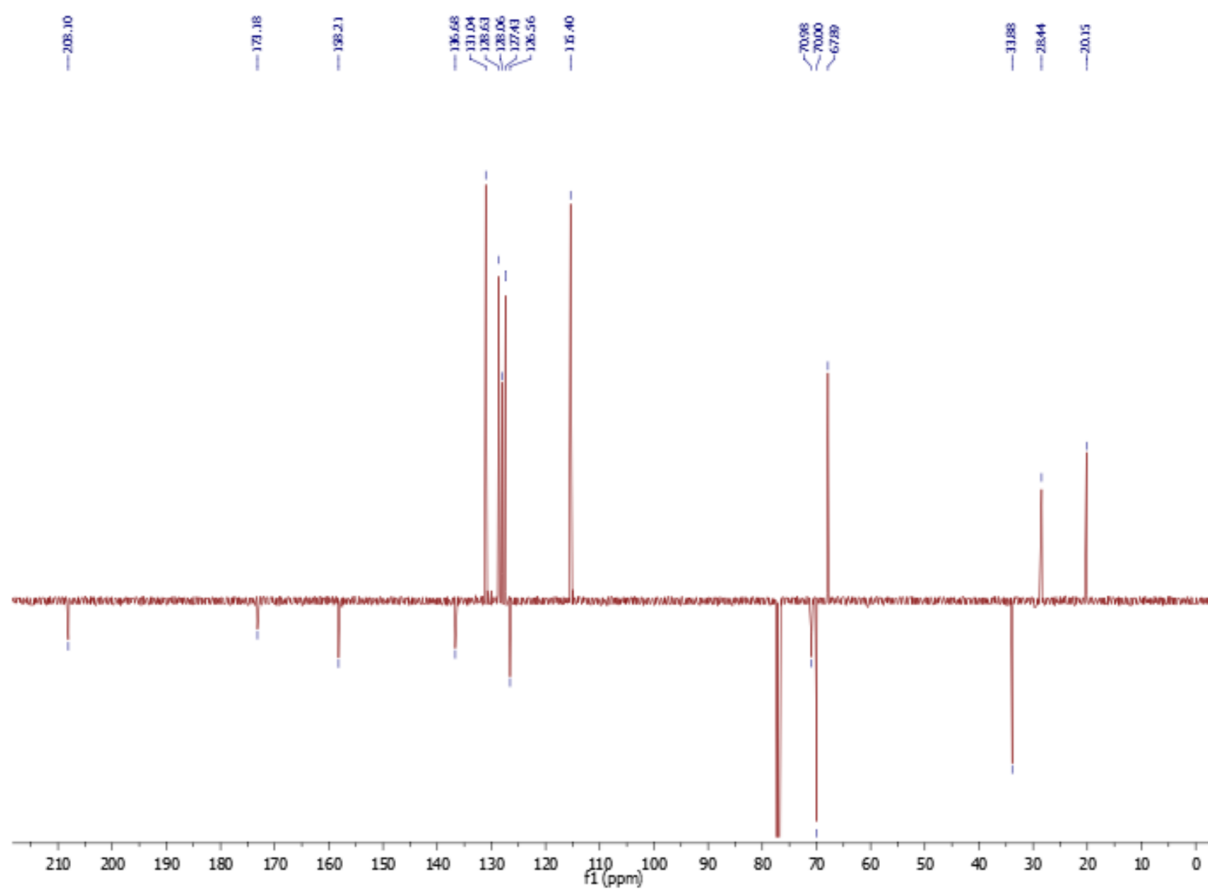
^{13}C NMR (125 MHz, $(\text{CD}_3)_2\text{CO}$) (S)-5-(4-(Benzyloxy)benzyl)-4-(*tert*-butyldiphenylsilyloxy)-1,3-dimethyl-1*H*-pyrrol-2(5*H*)-one (**10**)



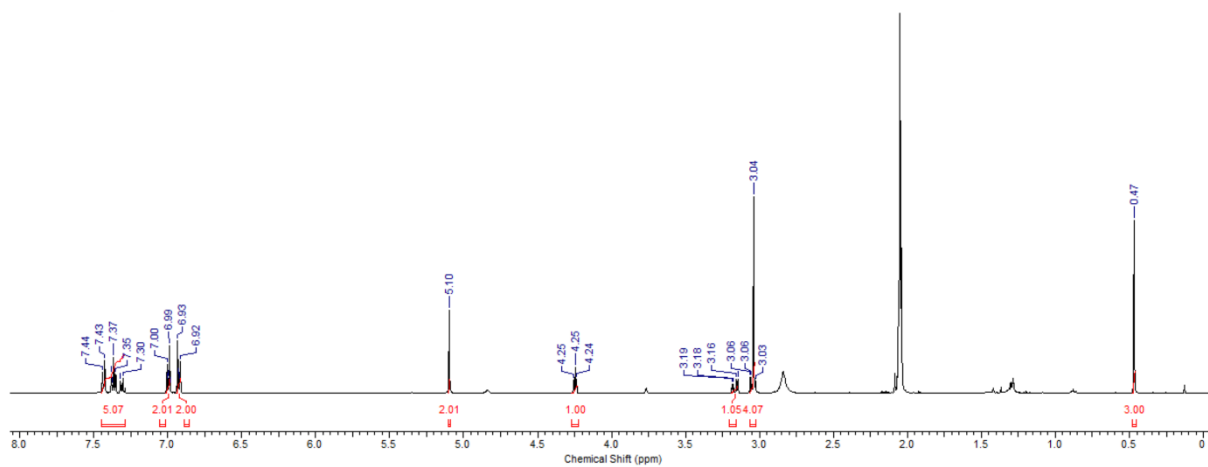
^1H NMR (500 MHz, CDCl_3) (3S,5S)-5-(4-(Benzyloxy)benzyl)-3-hydroxy-1,3-dimethylpyrrolidine-2,4-dione (**12**):



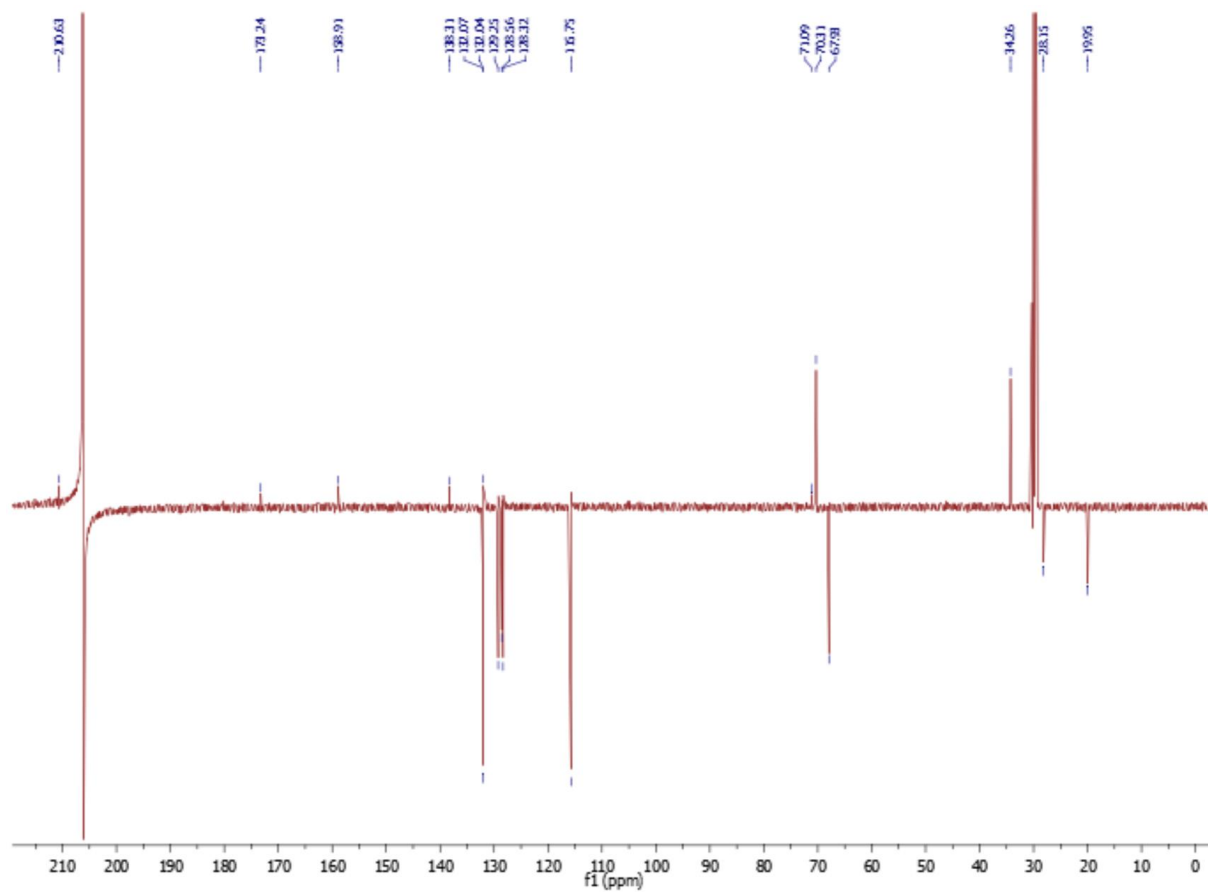
^{13}C NMR (125 MHz, CDCl_3) (3S,5S)-5-(4-(Benzyloxy)benzyl)-3-hydroxy-1,3-dimethylpyrrolidine-2,4-dione (**12**):



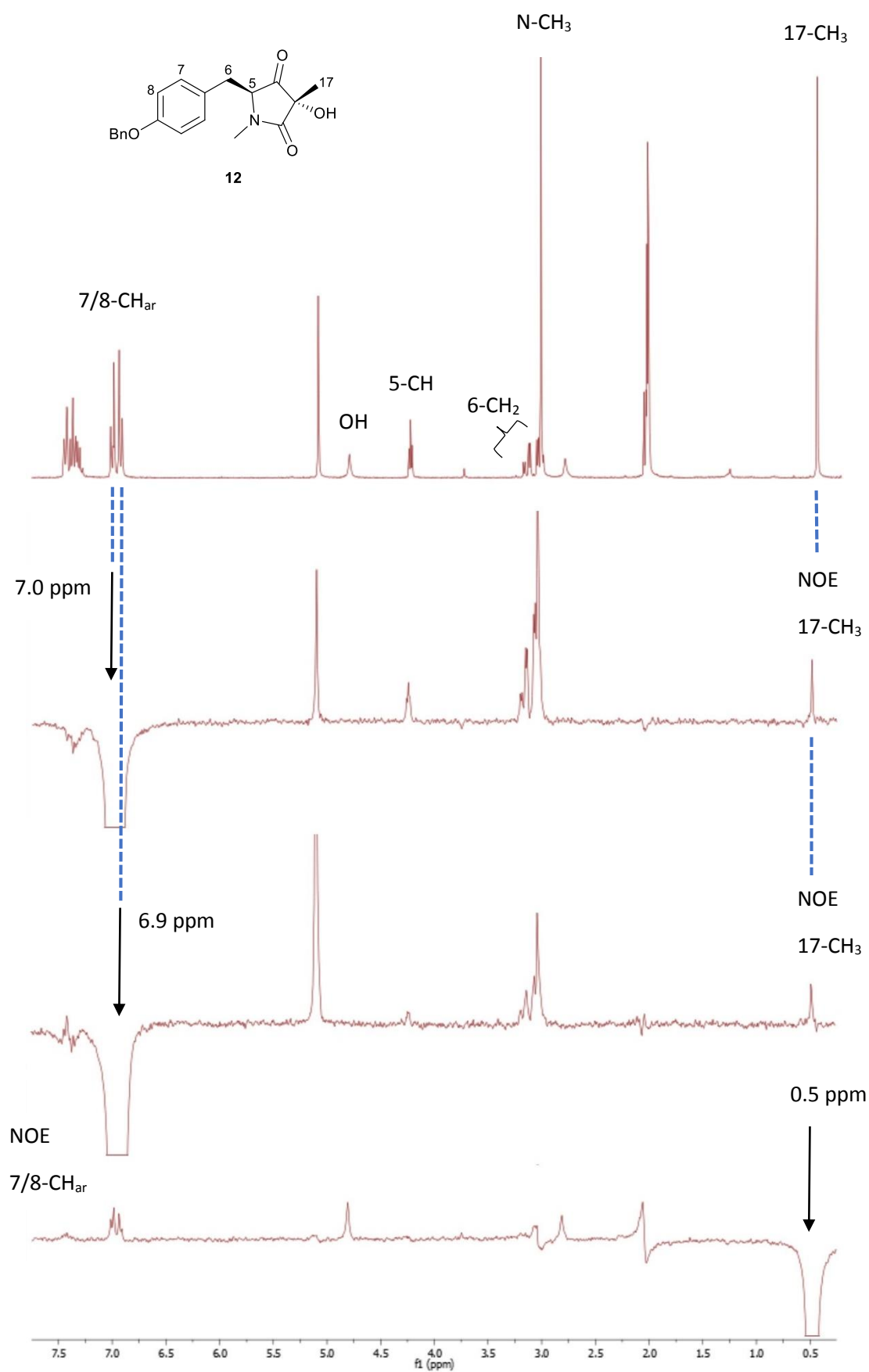
^1H NMR (500 MHz, $(\text{CD}_3)_2\text{CO}$) (3*S*,5*S*)-5-(4-(Benzyloxy)benzyl)-3-hydroxy-1,3-dimethylpyrrolidine-2,4-dione (**12**)



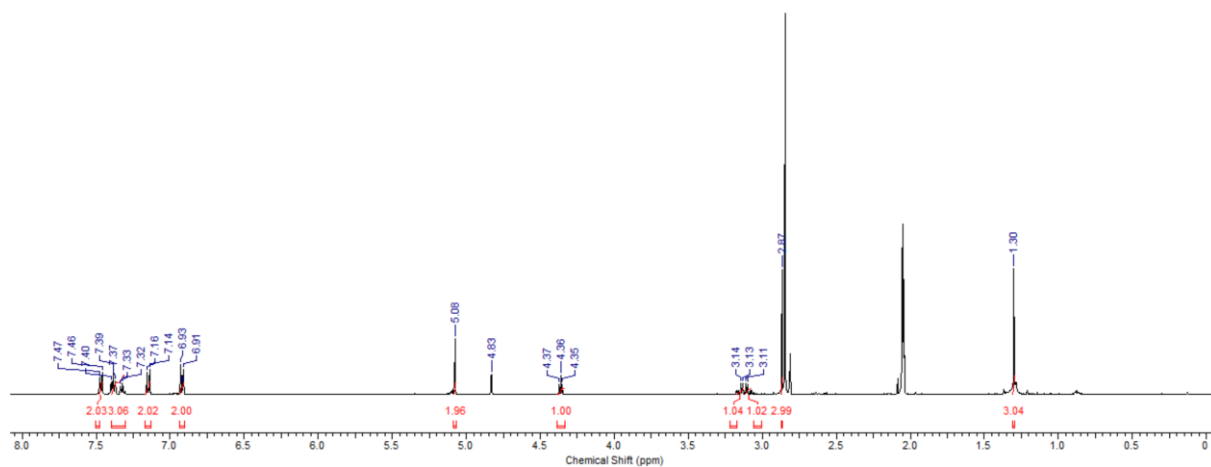
^{13}C NMR (125 MHz, $(\text{CD}_3)_2\text{CO}$) (3*S*,5*S*)-5-(4-(Benzyloxy)benzyl)-3-hydroxy-1,3-dimethylpyrrolidine-2,4-dione (**12**)



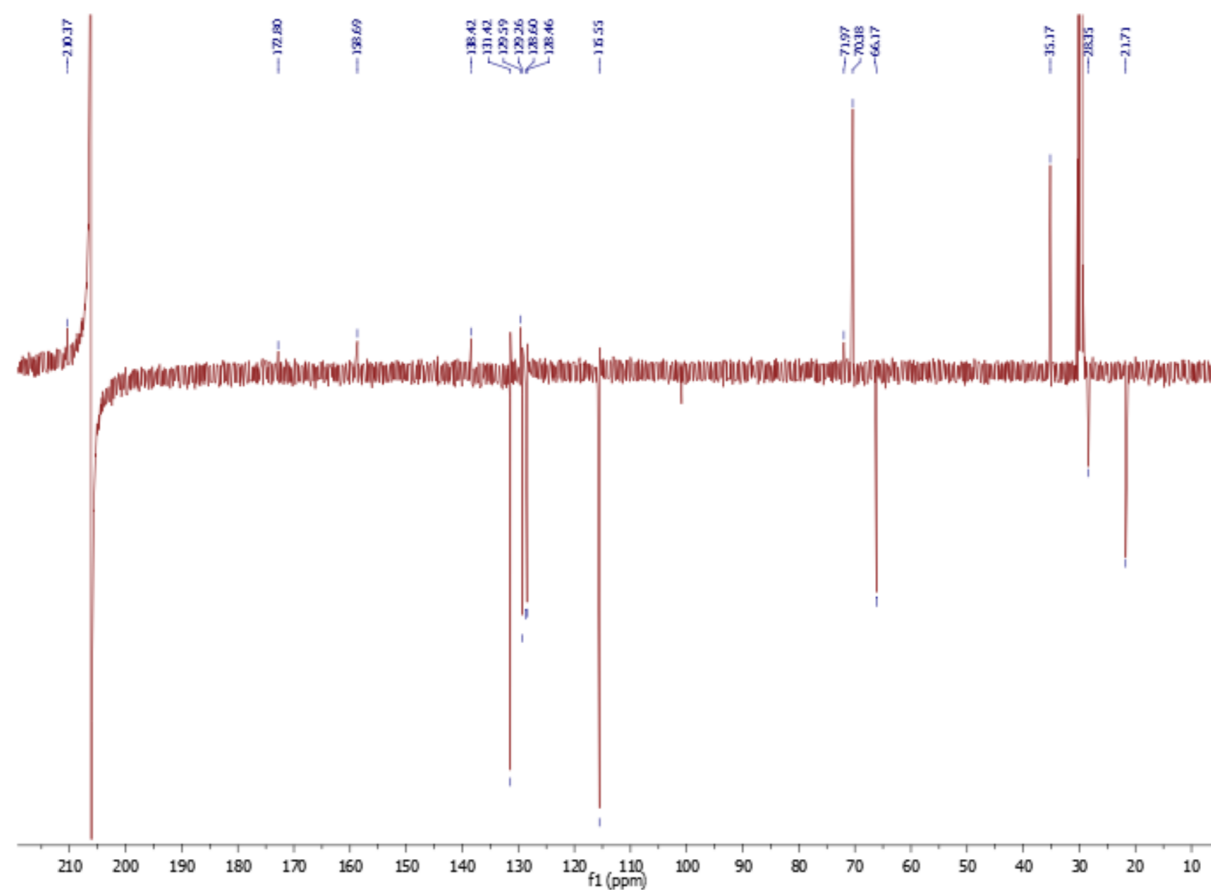
1D-NOE-difference-spectra) (300 MHz, $(\text{CD}_3)_2\text{CO}$) (3S,5S)-**12**



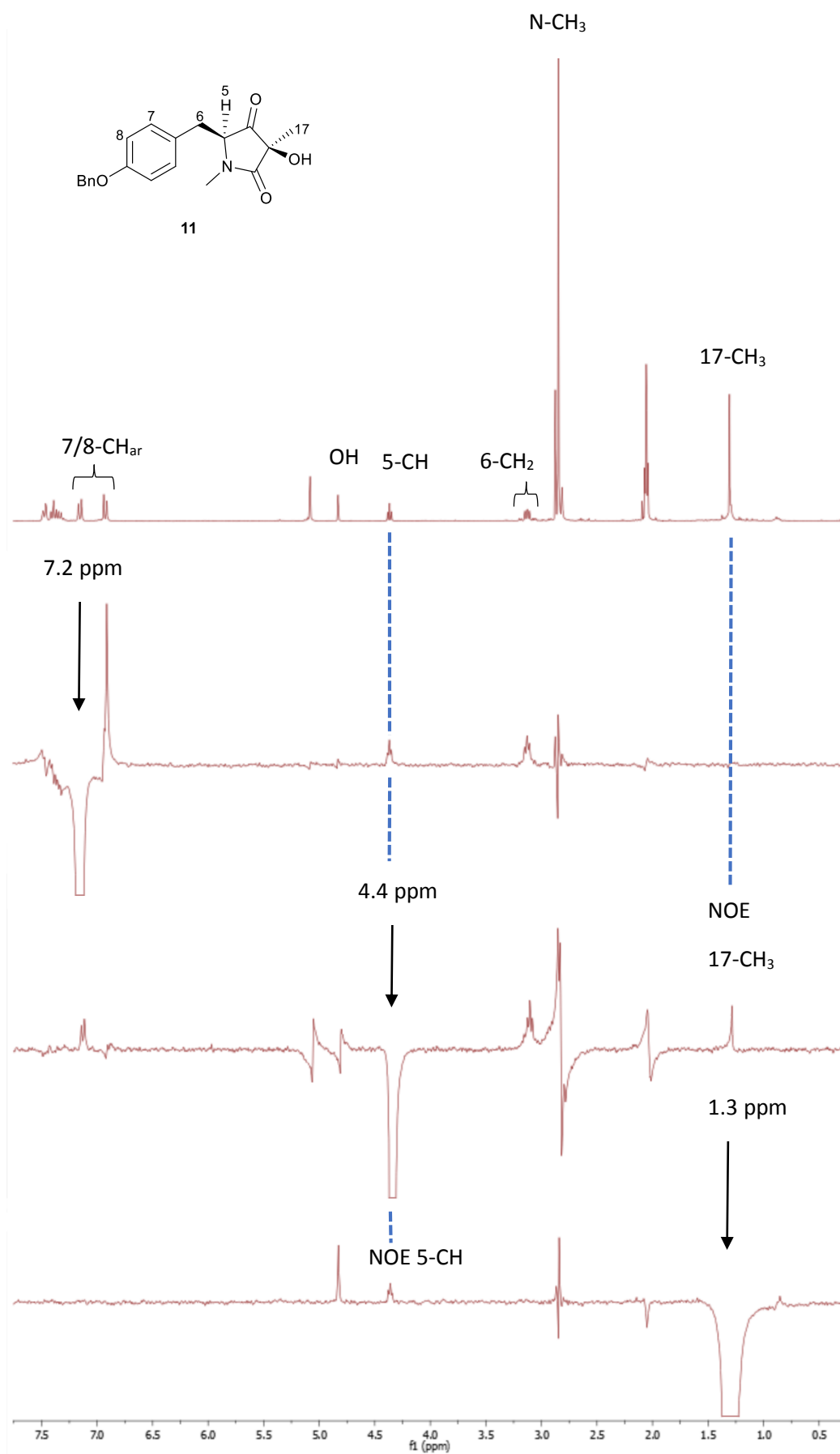
^1H NMR (500 MHz, $(\text{CD}_3)_2\text{CO}$) (3*R*,5*S*)-5-(4-(Benzyloxy)benzyl)-3-hydroxy-1,3-dimethylpyrrolidine-2,4-dione (**11**)



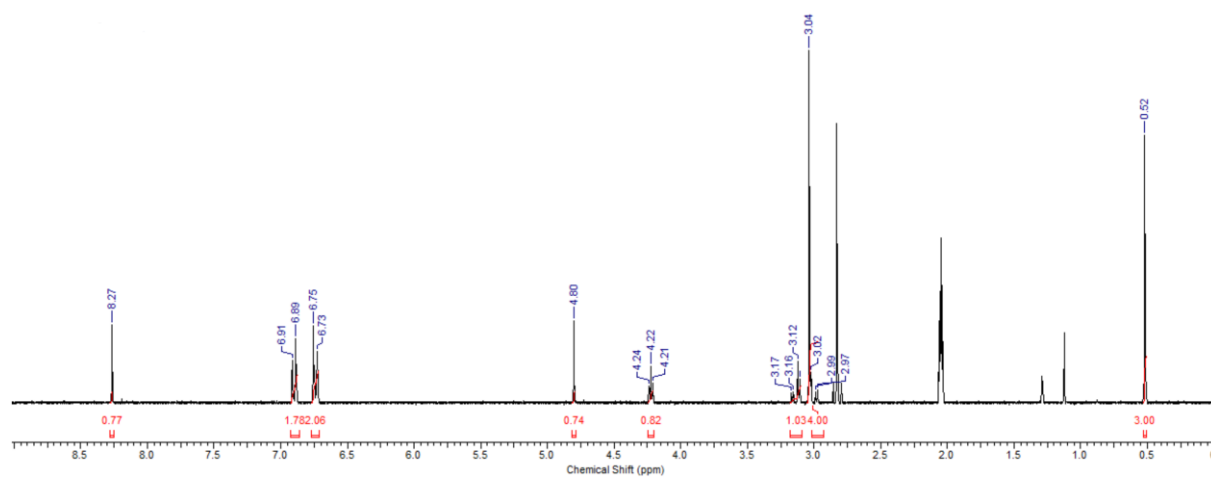
^{13}C NMR (500 MHz, $(\text{CD}_3)_2\text{CO}$) (3*R*,5*S*)-5-(4-(Benzyloxy)benzyl)-3-hydroxy-1,3-dimethylpyrrolidine-2,4-dione (**11**)



1D-NOE-difference-spectra) (300 MHz, (CD₃)₂CO) (3*R*,5*S*)-**11**



^1H NMR (300 MHz, $(\text{CD}_3)_2\text{CO}$) Rigidiusculamide B ((3*S*,5*S*)-2)



^{13}C NMR (125 MHz, $(\text{CD}_3)_2\text{CO}$) Rigidiusculamide B ((3*S*,5*S*)-2)

