Supporting Information

Keikipukalides, Furanocembrane Diterpenes from the Antarctic Deep Sea Octocoral *Plumarella delicatissima*

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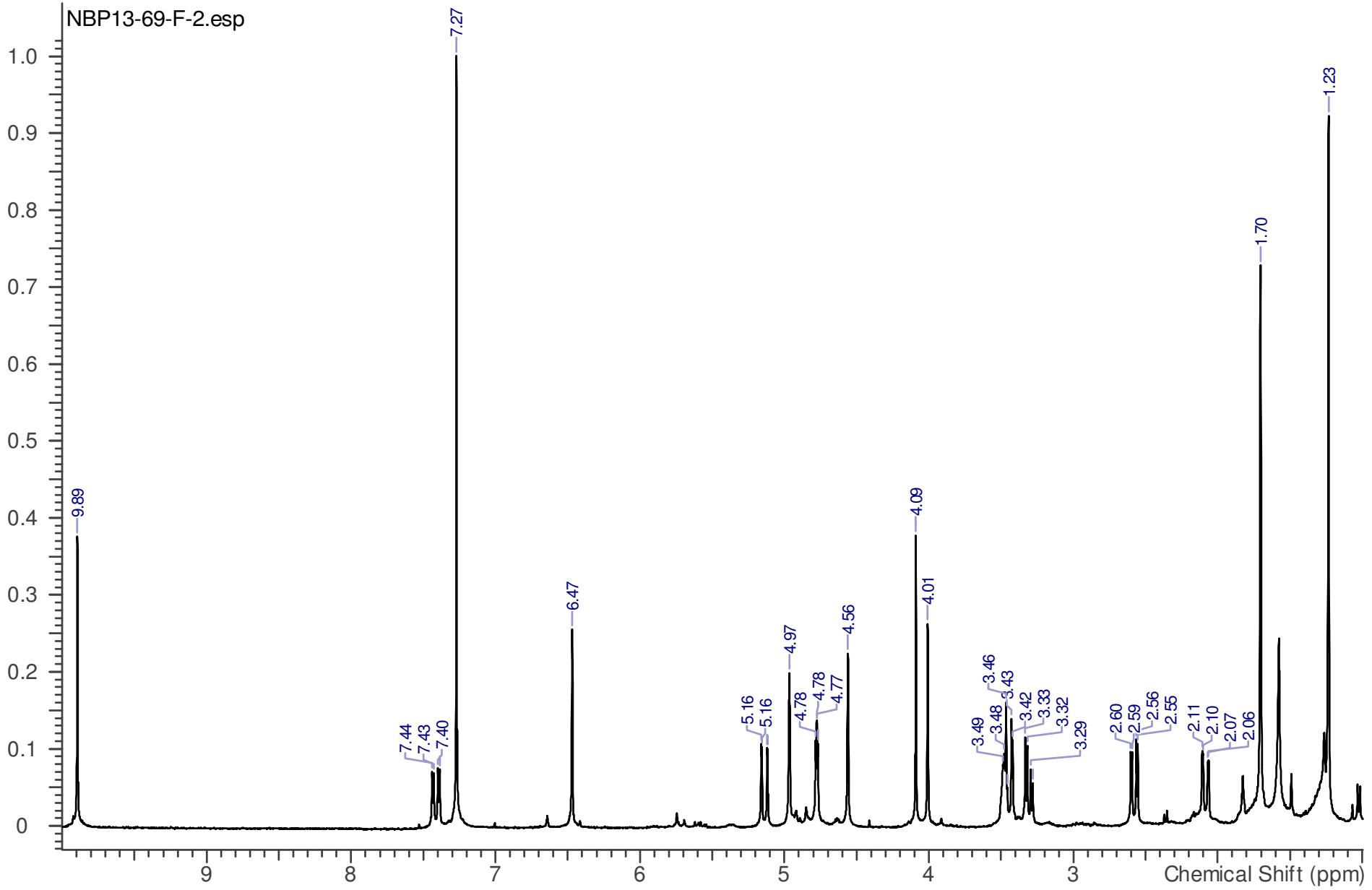

Figure S1. ^{1}H NMR spectrum of keikipukalide A (**1**) in $CDCl_3$, 400 MHz

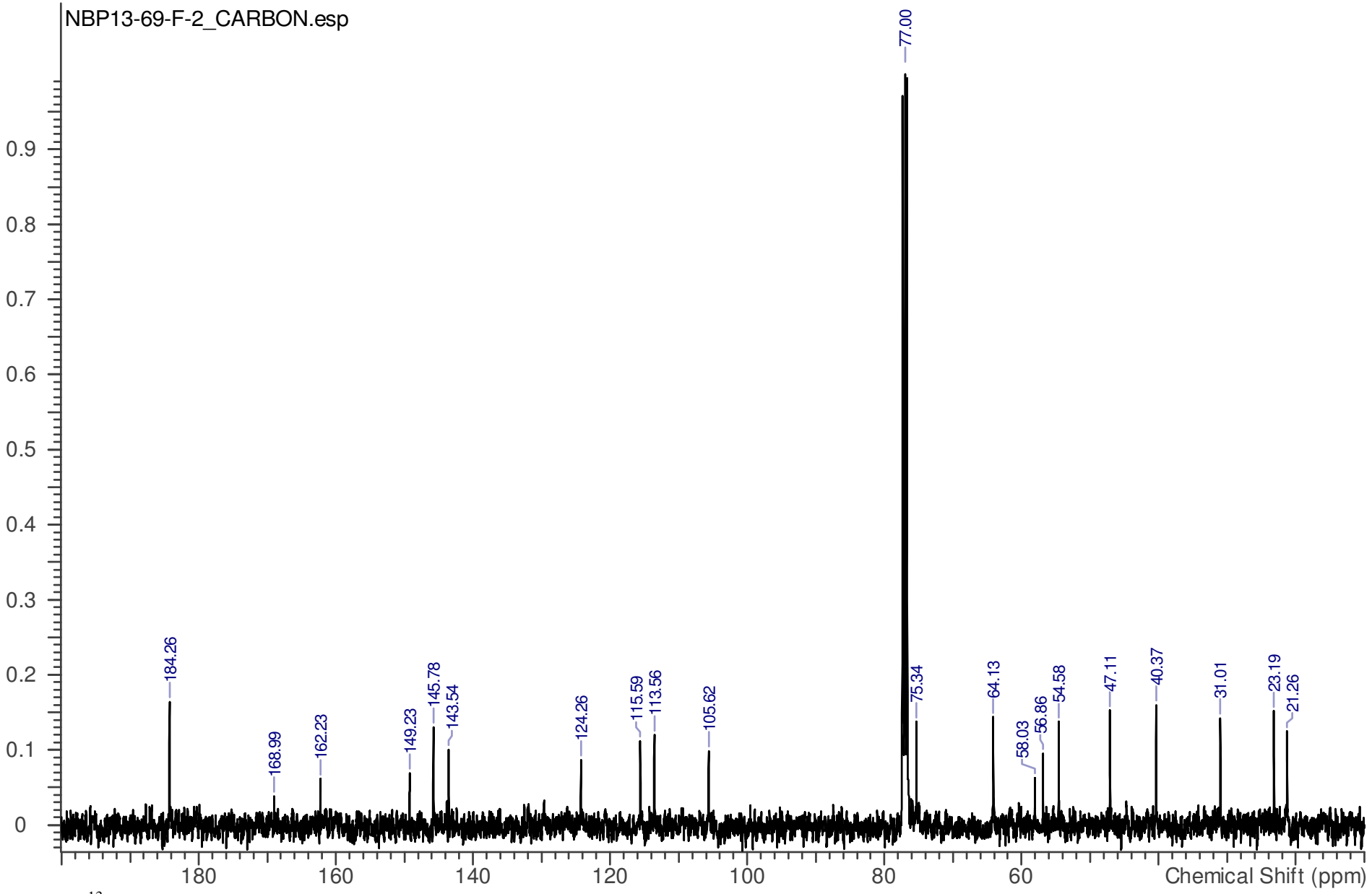

Figure S2. ^{13}C NMR spectrum of keikipukalide A (**1**) in $CDCl_3$, 100 MHz

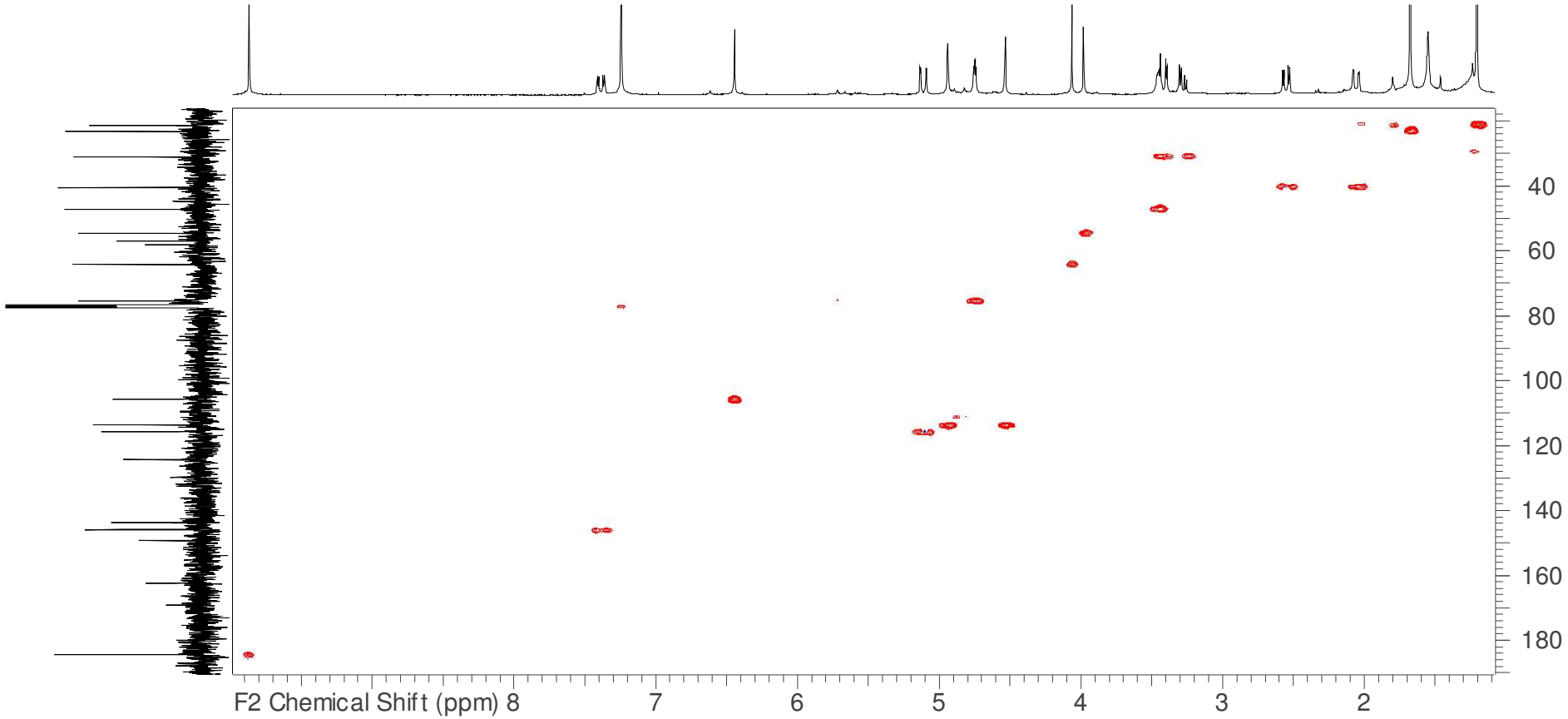

Figure S3. gHMQC of keikipukalide A (**1**) in $CDCl_3$, 400 MHz

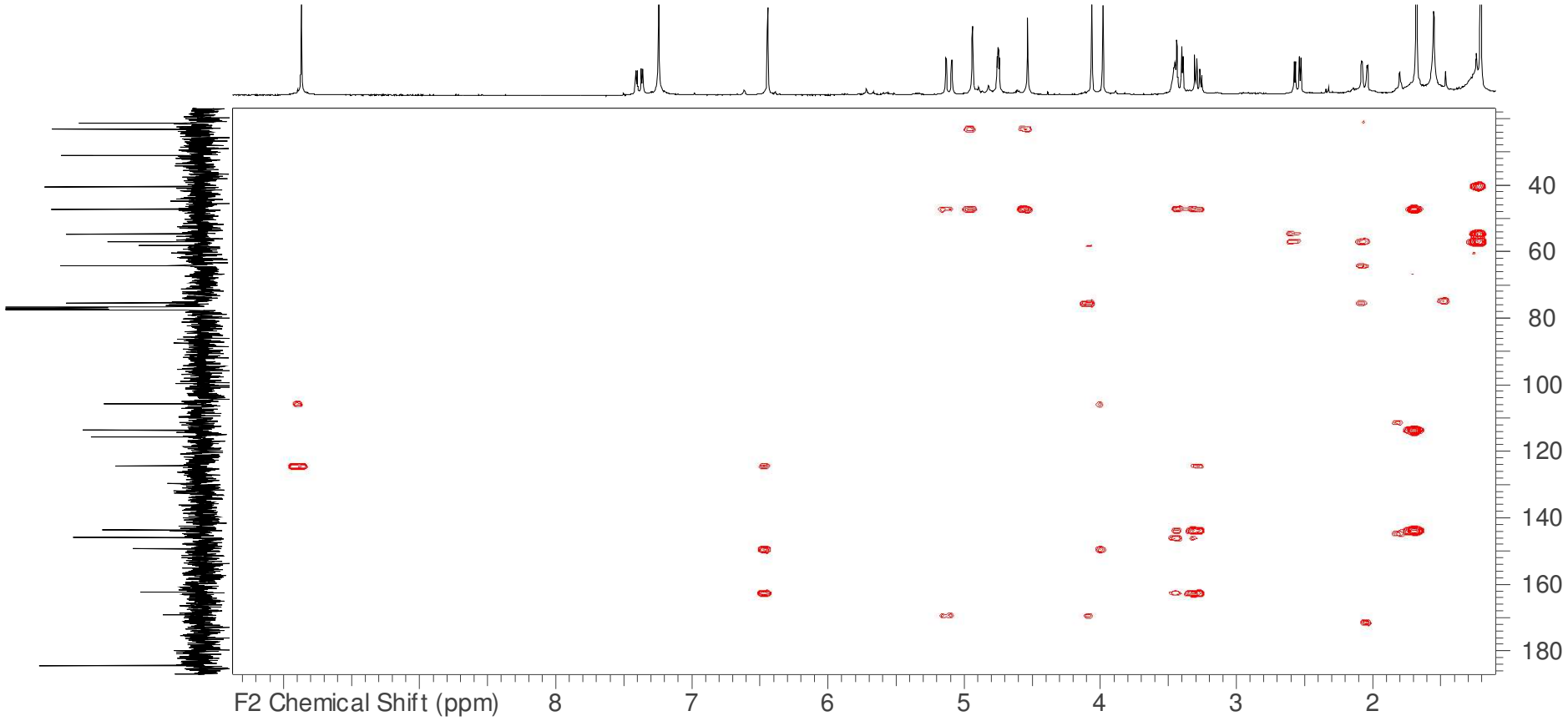

Figure S4. gHMBC of keikipukalide A (**1**) in $CDCl_3$, 400 MHz

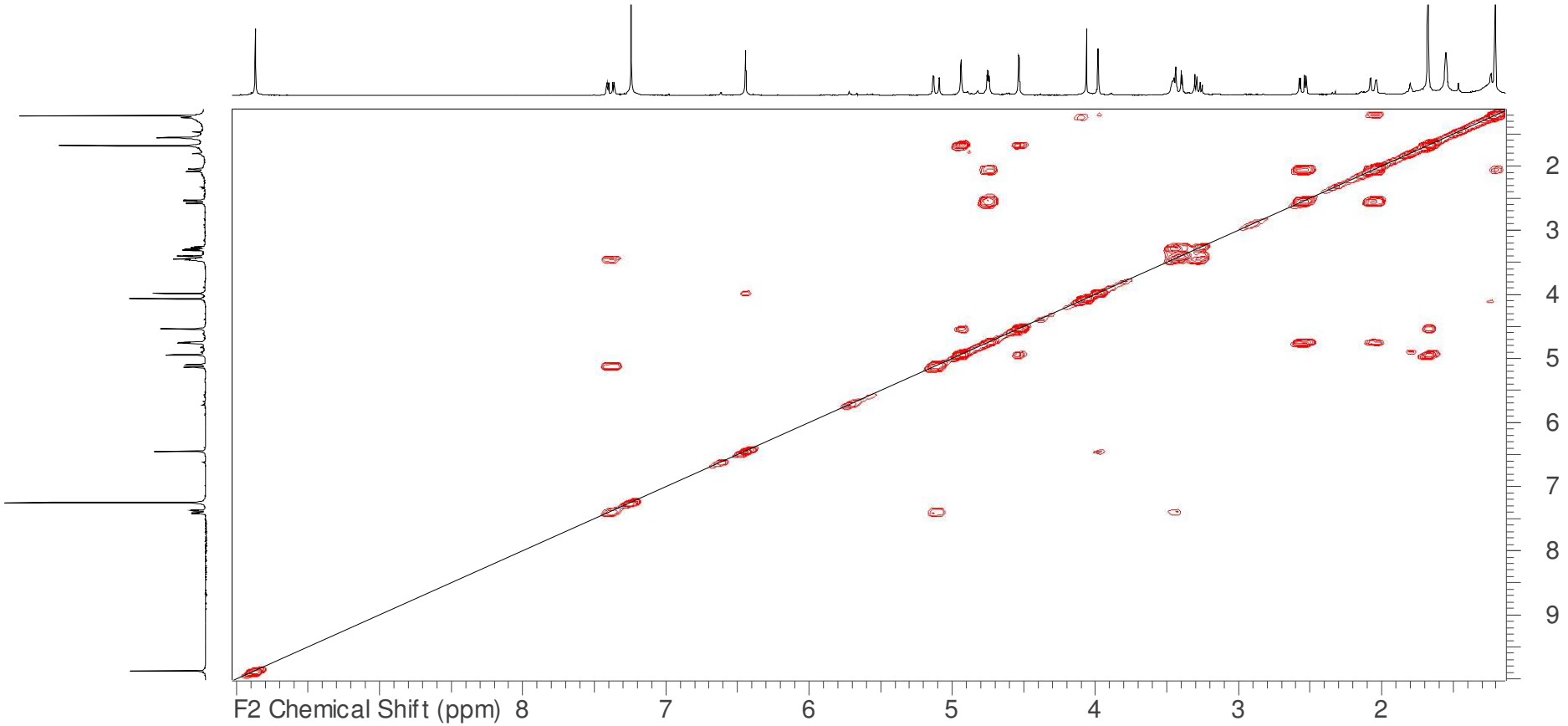

Figure S5. gCOSY of keikipukalide A (**1**) in $CDCl_3$, 400 MHz

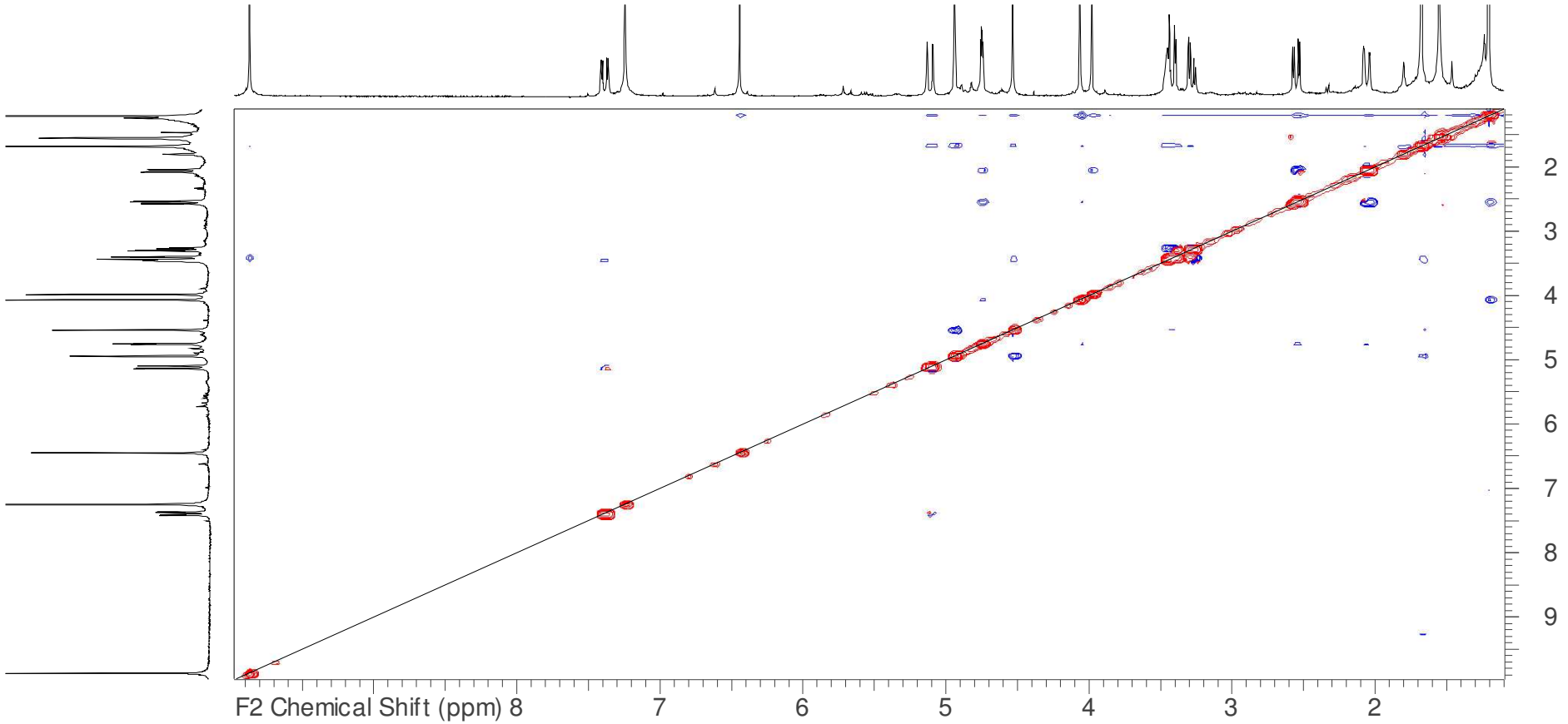

Figure S6. NOESY of keikipukalide A (**1**) in $CDCl_3$, 500 MHz

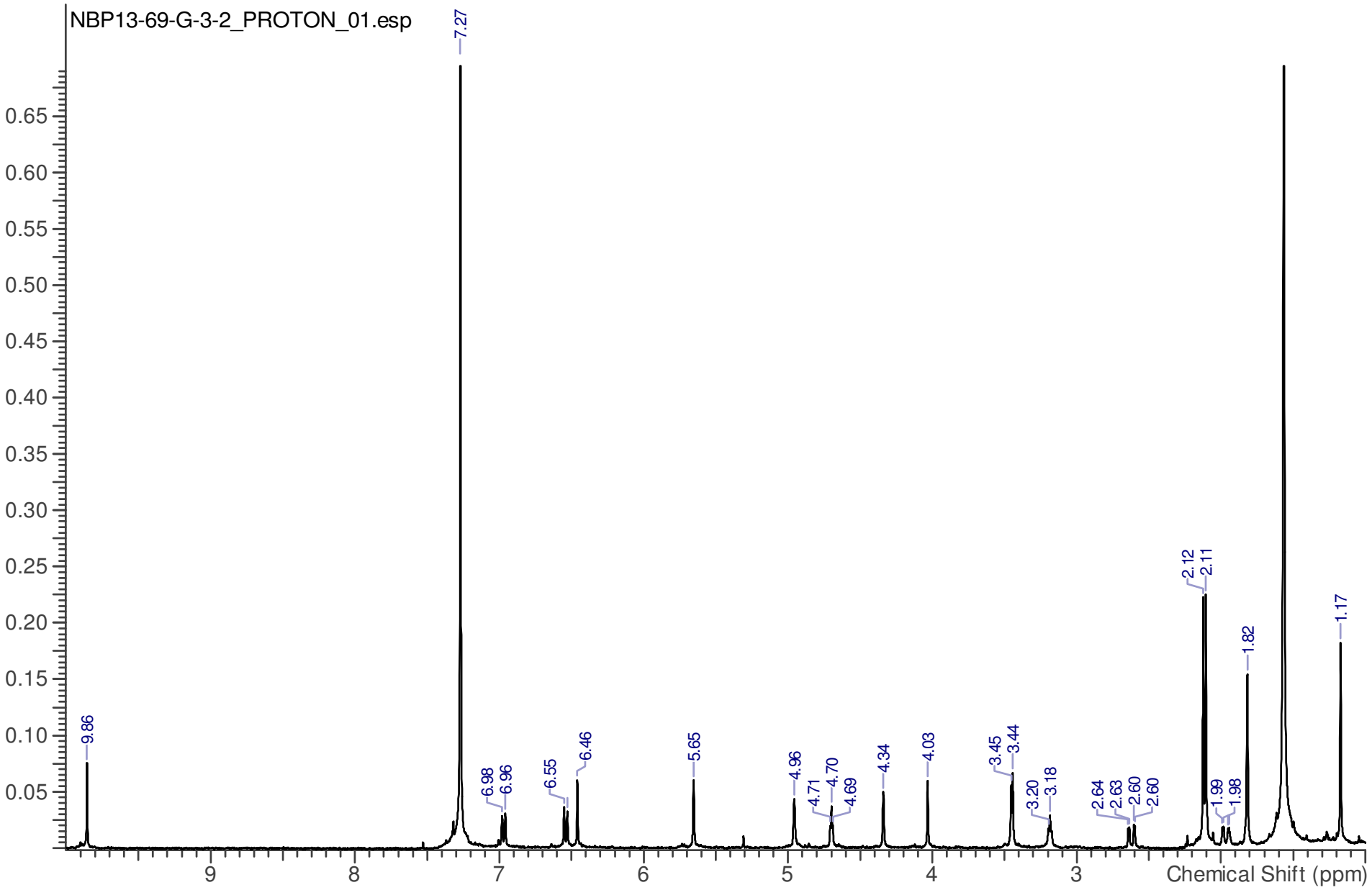

Figure S7. ^{1}H NMR spectrum of keikipukalide B (**2**) in $CDCl_3$, 400 MHz

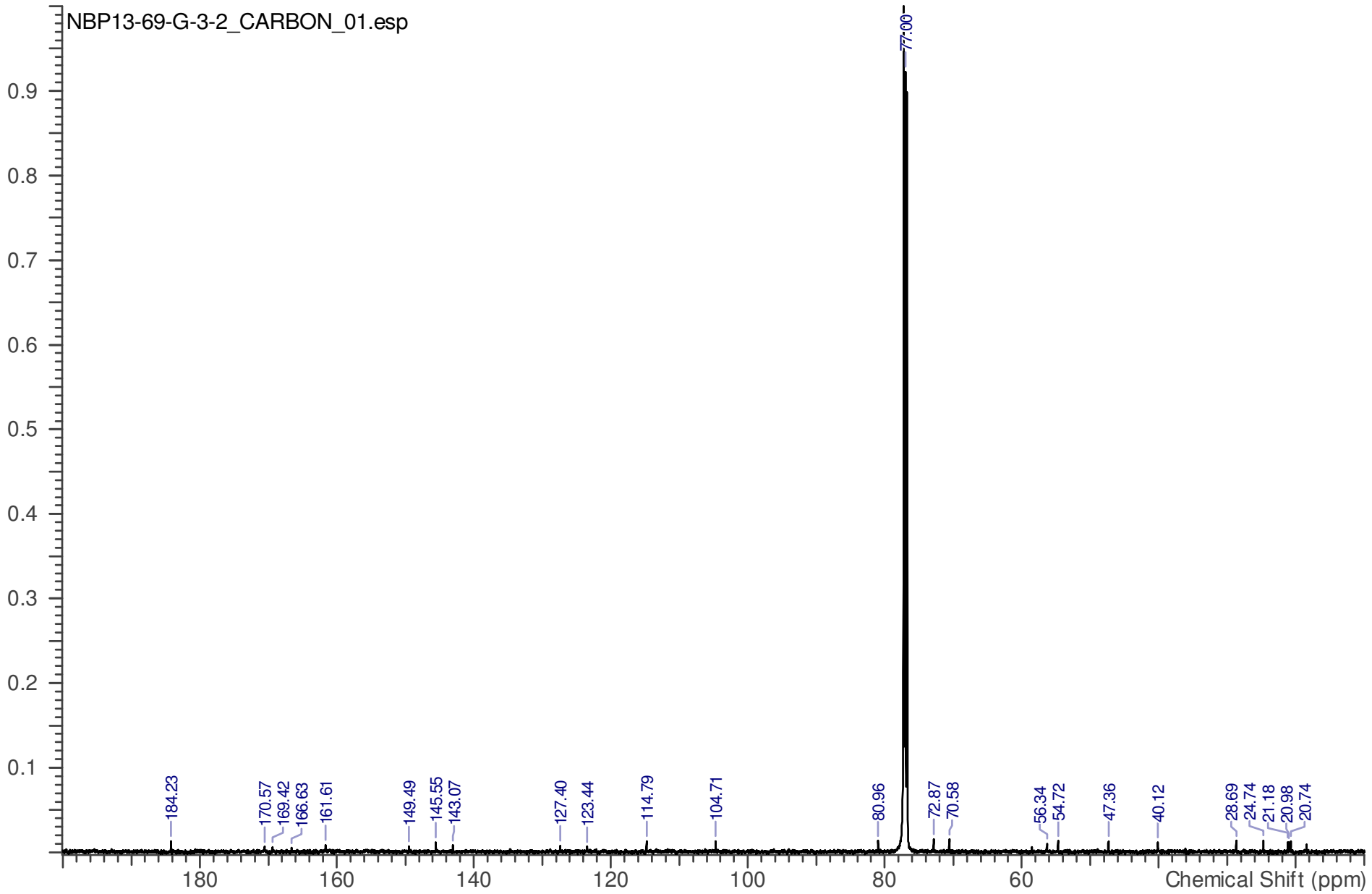

Figure S8. ^{13}C NMR spectrum of keikipukalide B (**2**) in $CDCl_3$, 125 MHz

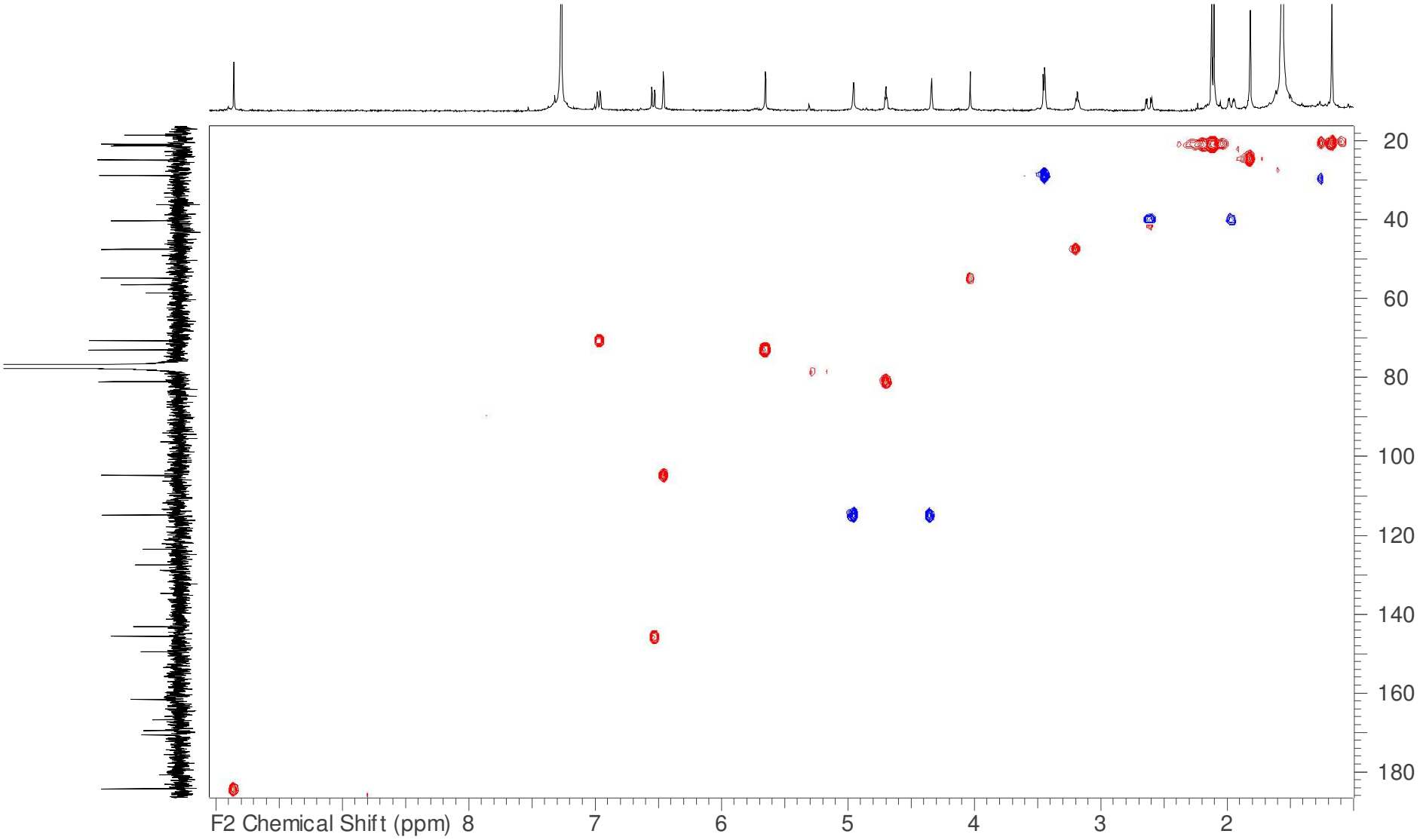

Figure S9. gHSQC of keikipukalide B (**2**) in $CDCl_3$, 600 MHz

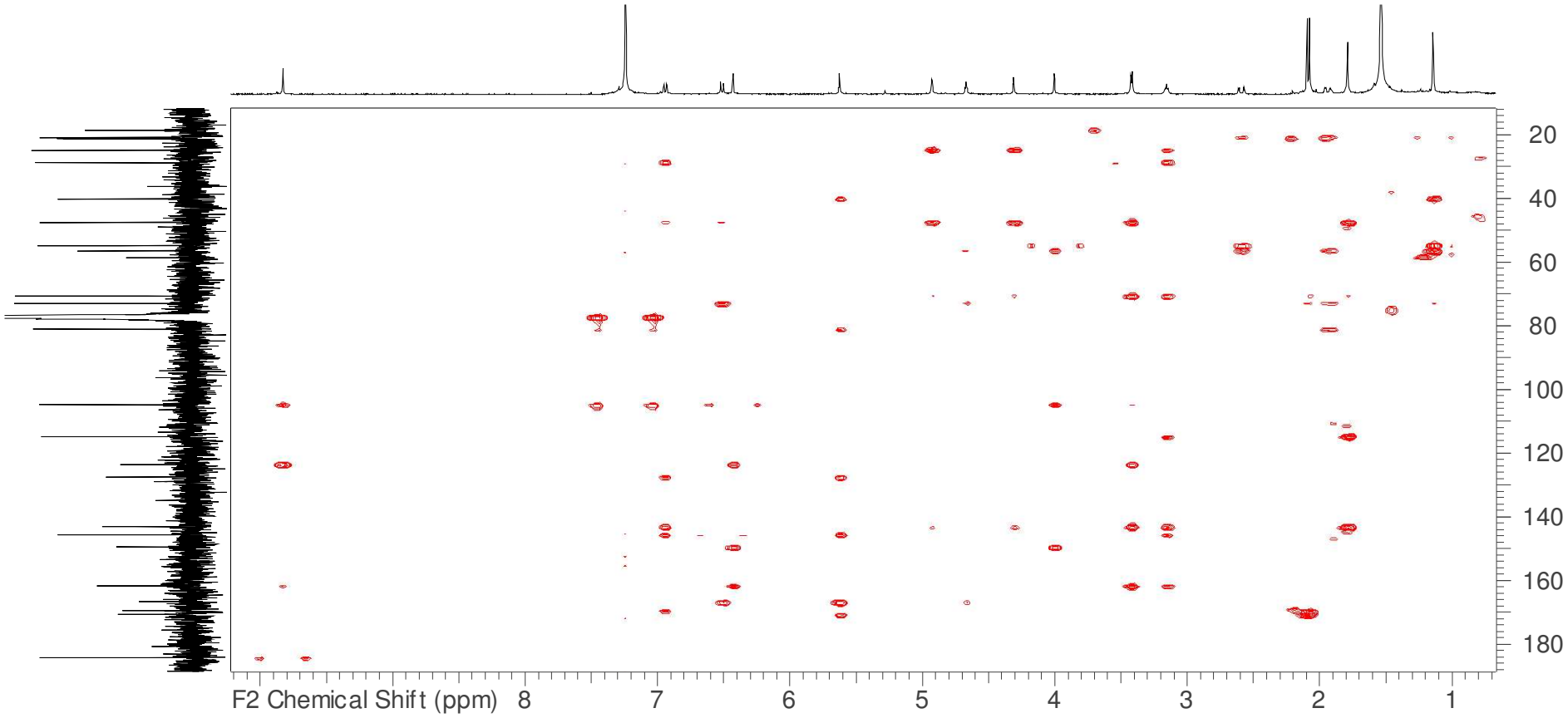

Figure S10. gHMBC of keikipukalide B (**2**) in $CDCl_3$, 500 MHz

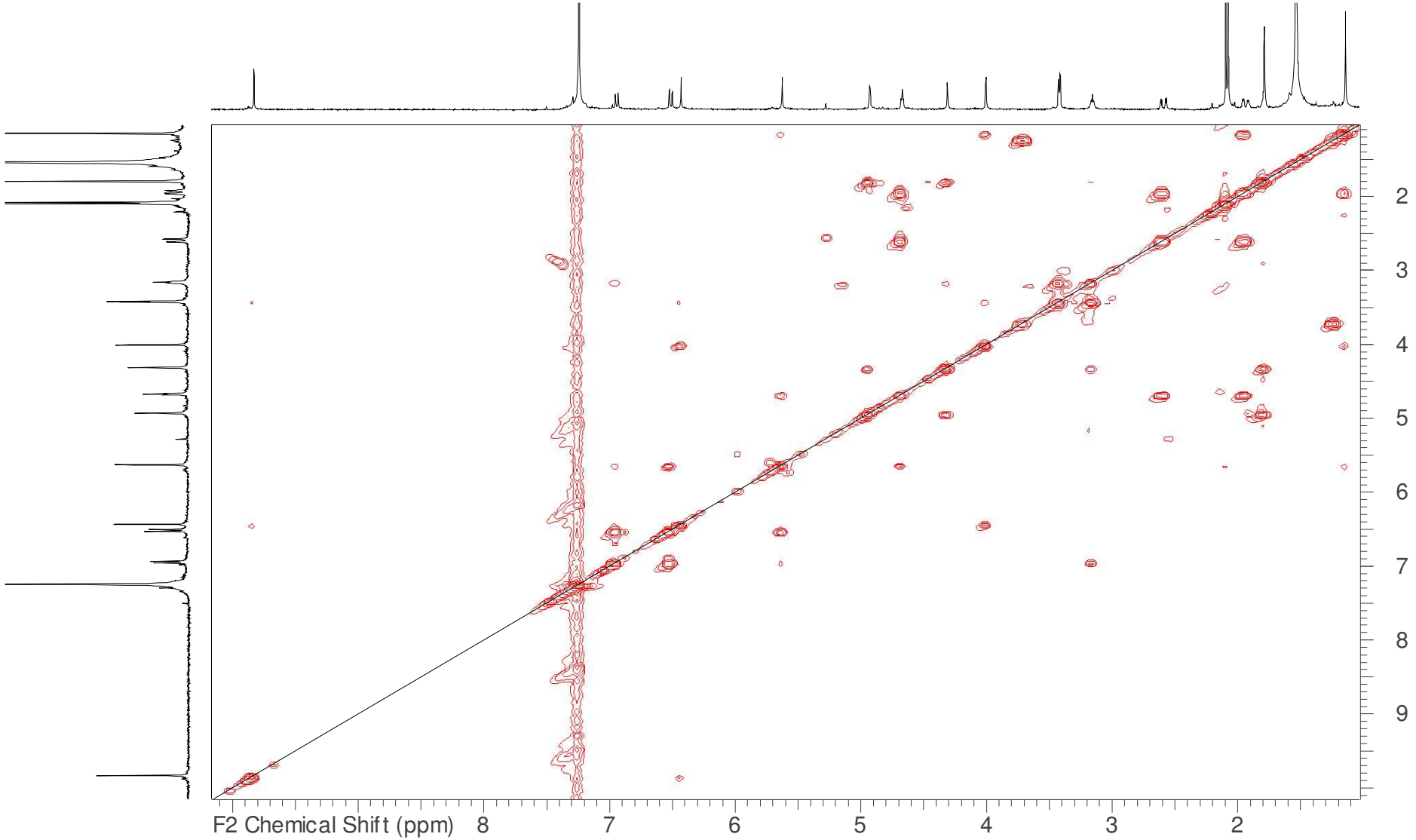

Figure S11. gCOSY of keikipukalide B (**2**) in $CDCl_3$, 500 MHz

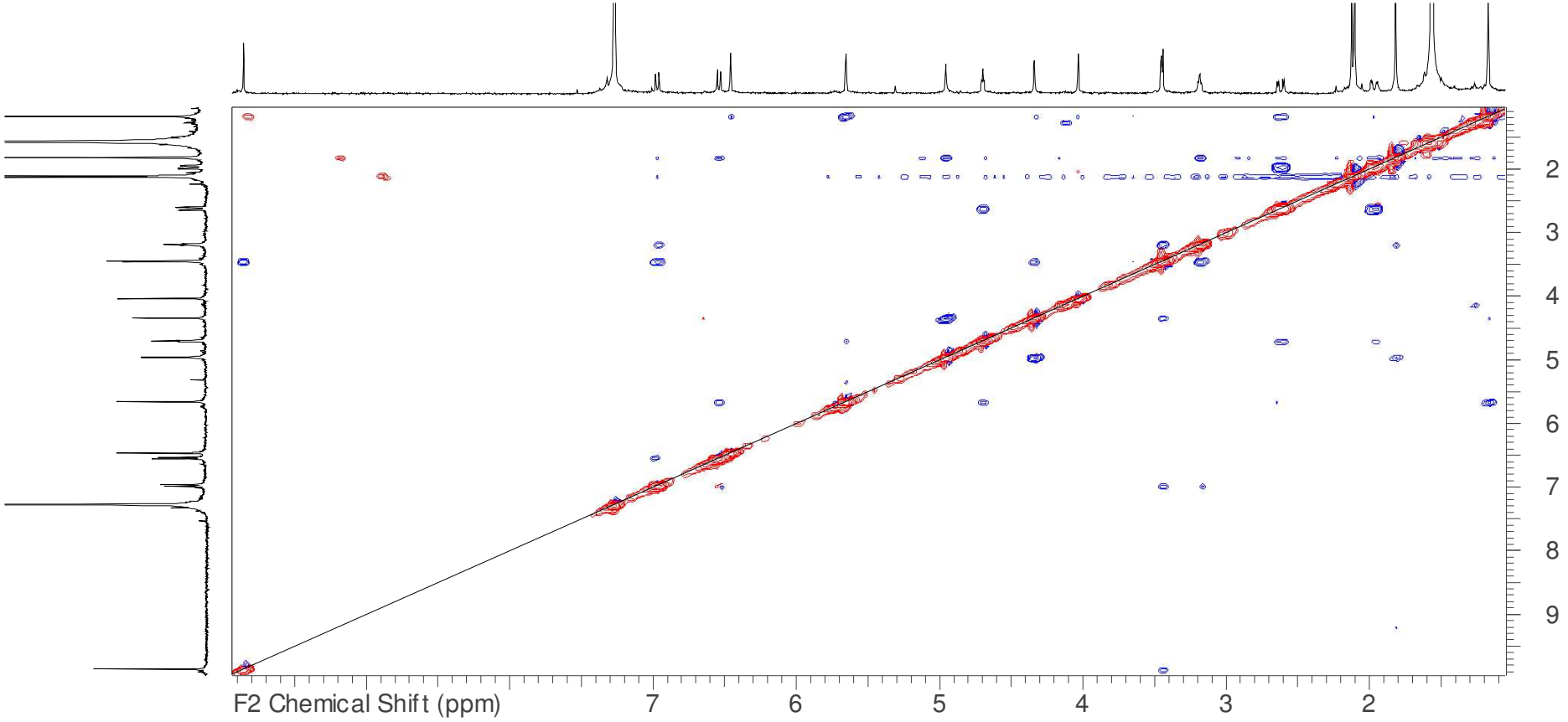

Figure S12. NOESY of keikipukalide B (**2**) in $CDCl_3$, 400 MHz

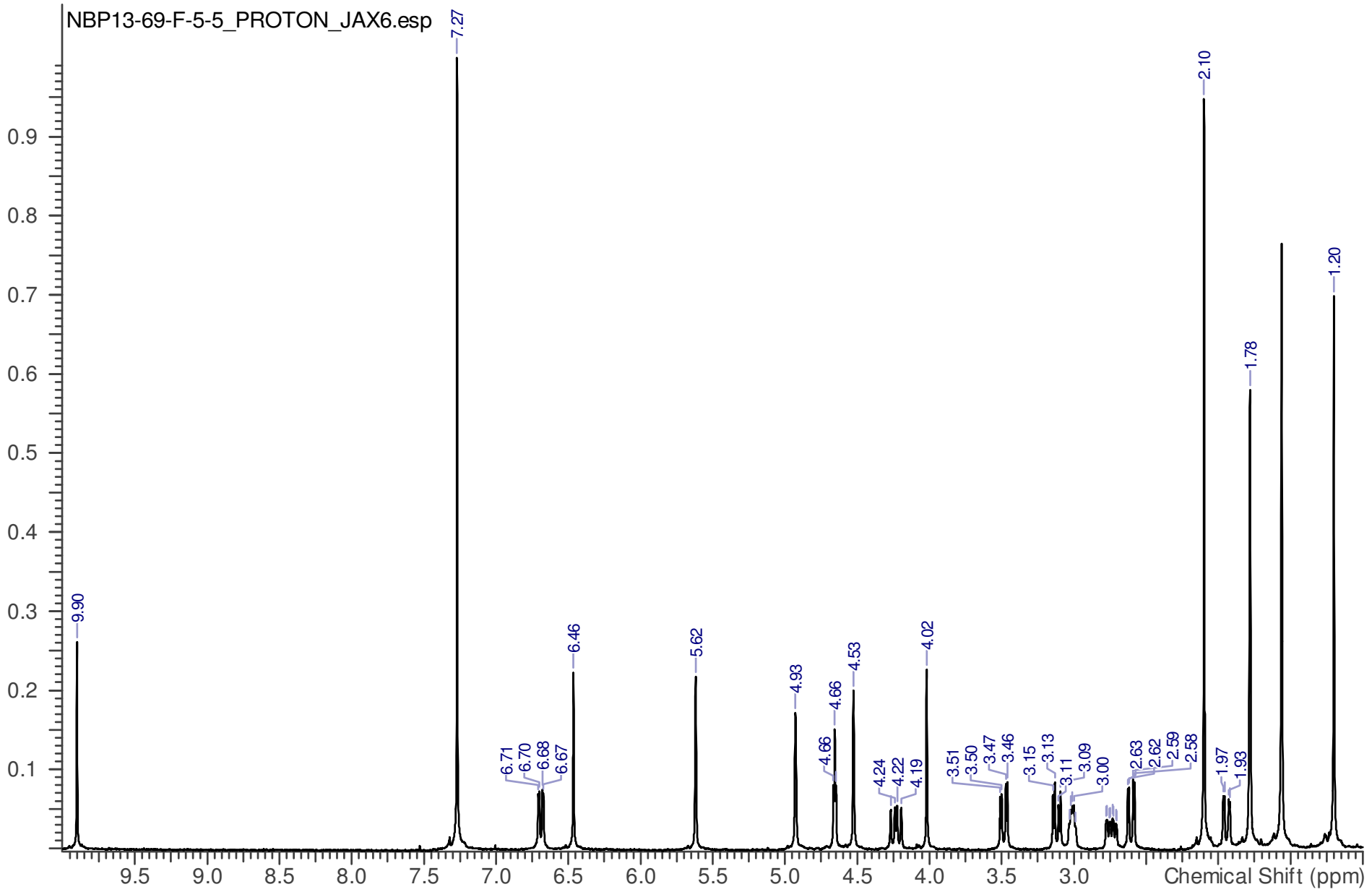

Figure S13. ^{1}H NMR spectrum of keikipukalide C (**3**) in $CDCl_3$, 400 MHz

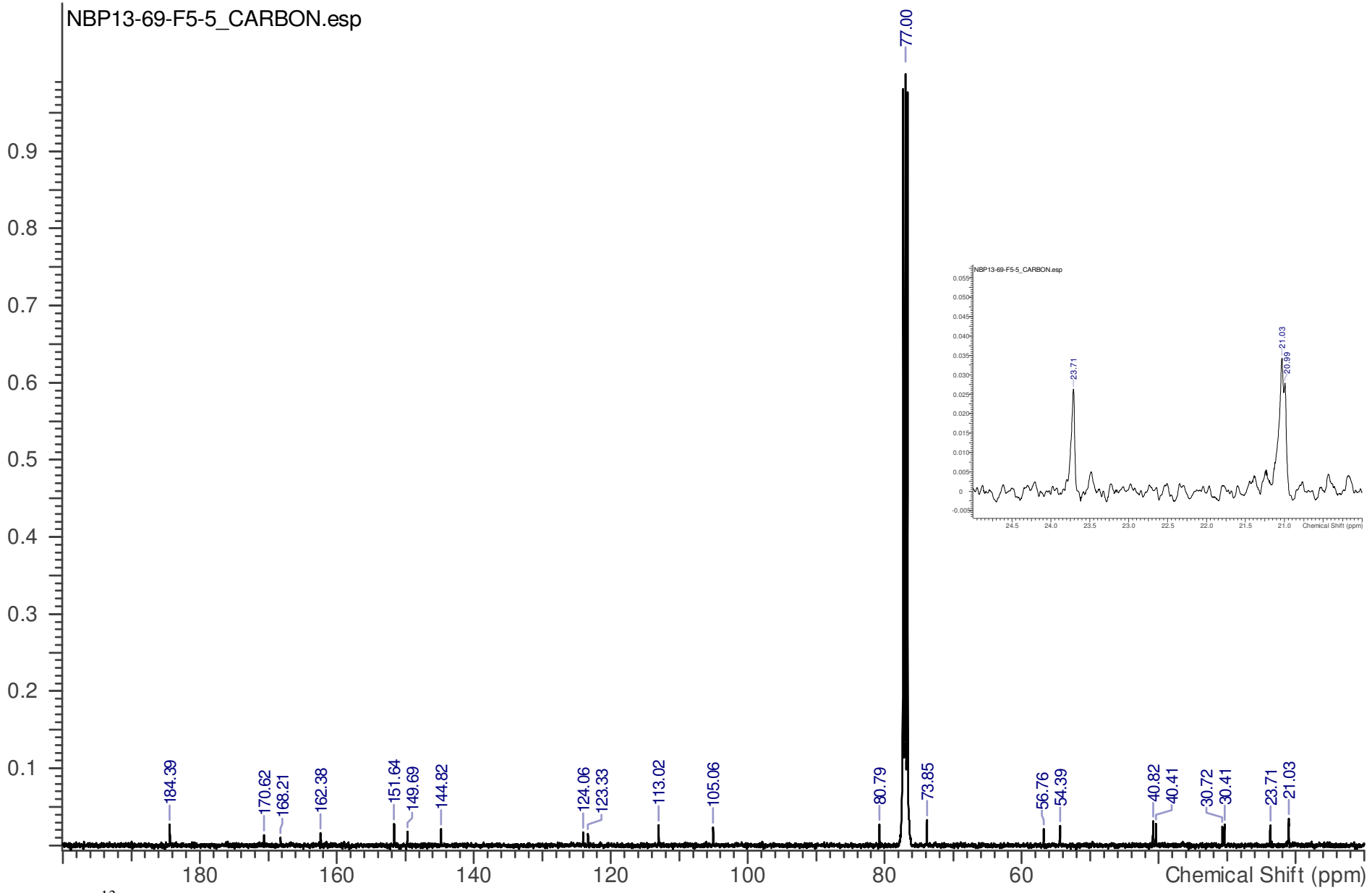

Figure S14. ^{13}C NMR spectrum of keikipukalide C (**3**) in $CDCl_3$, 100 MHz Inset: expansion of 20-25 ppm illustrating all resonances in the region, one of which does not display in the full spectrum due to congestion.

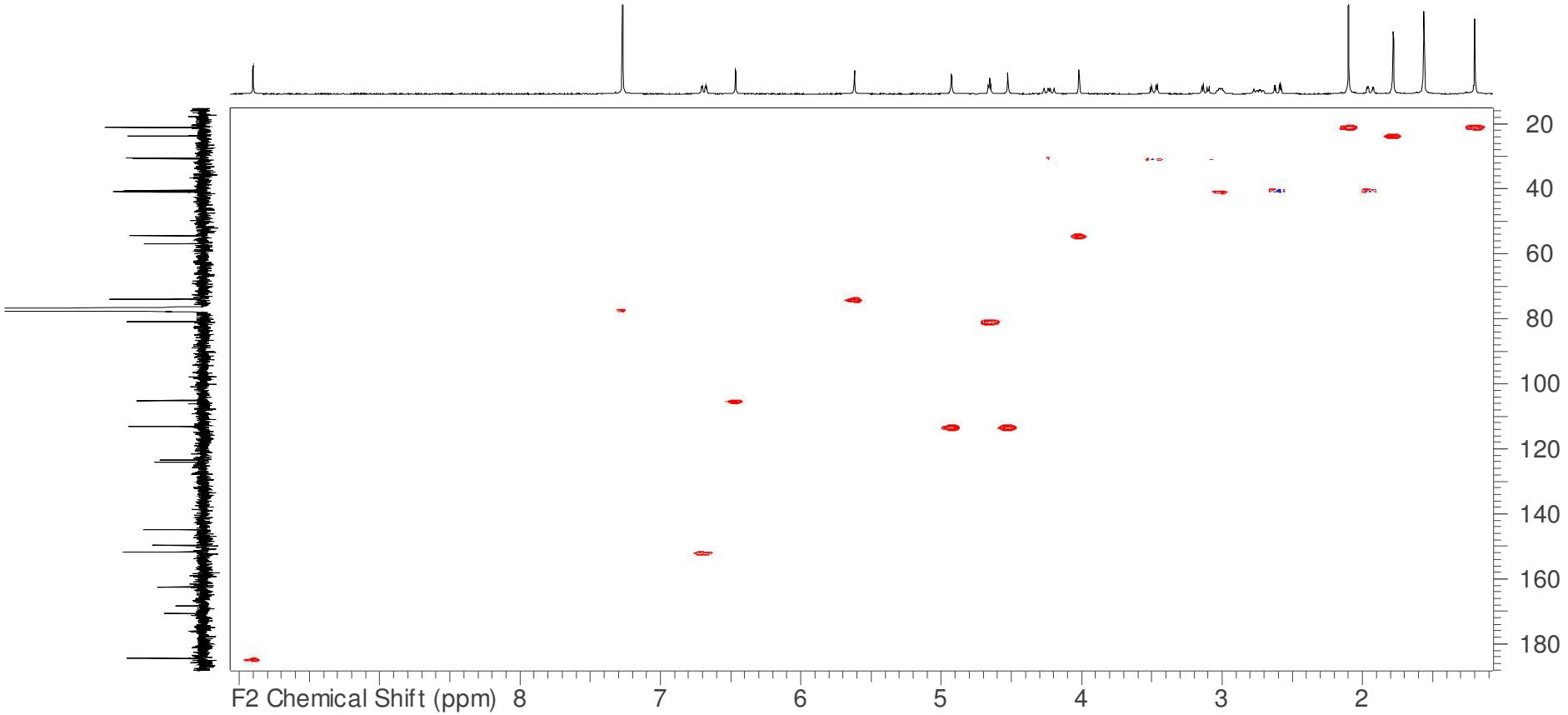

Figure S15. gHMQC of keikipukalide C (**3**) in $CDCl_3$, 400 MHz

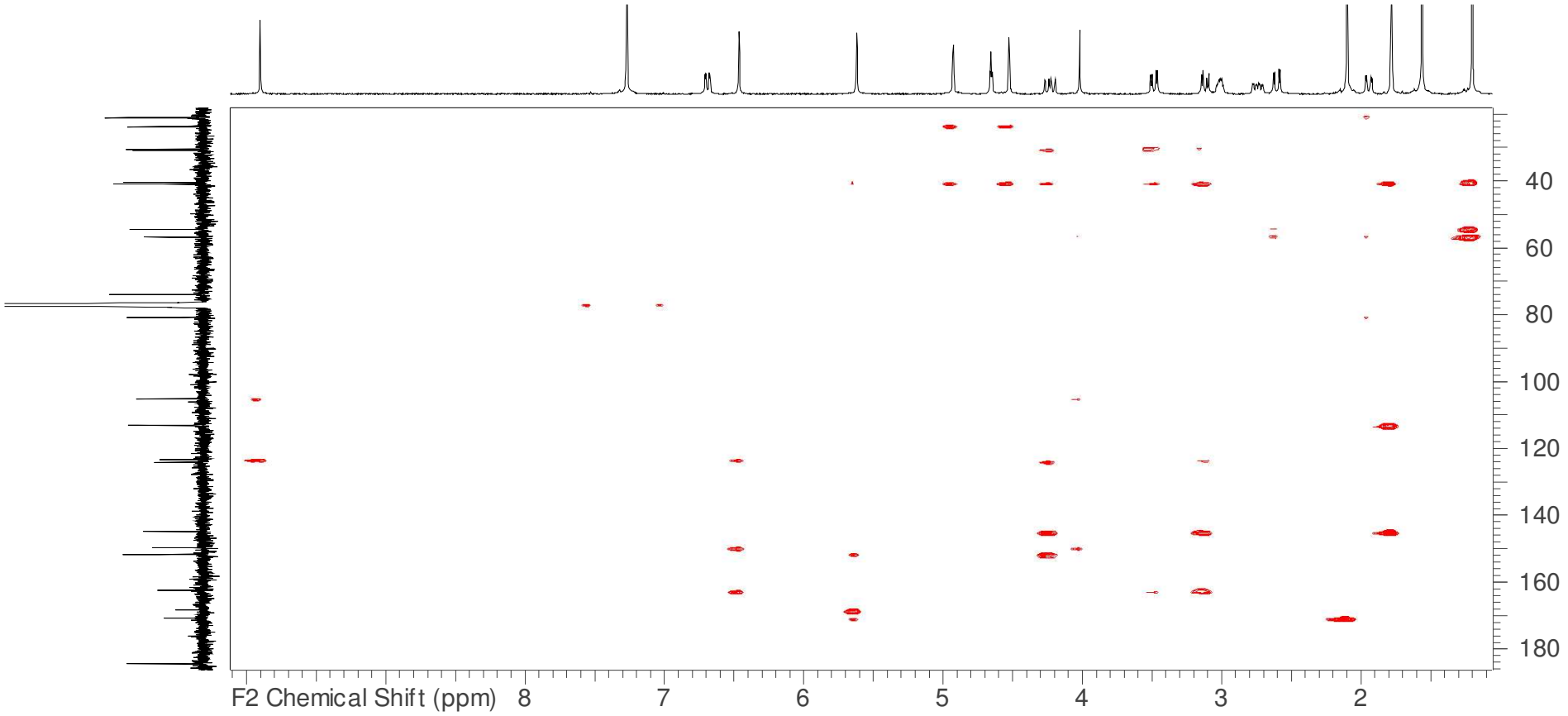

Figure S16. gHMBC of keikipukalide C (**3**) in $CDCl_3$, 400 MHz

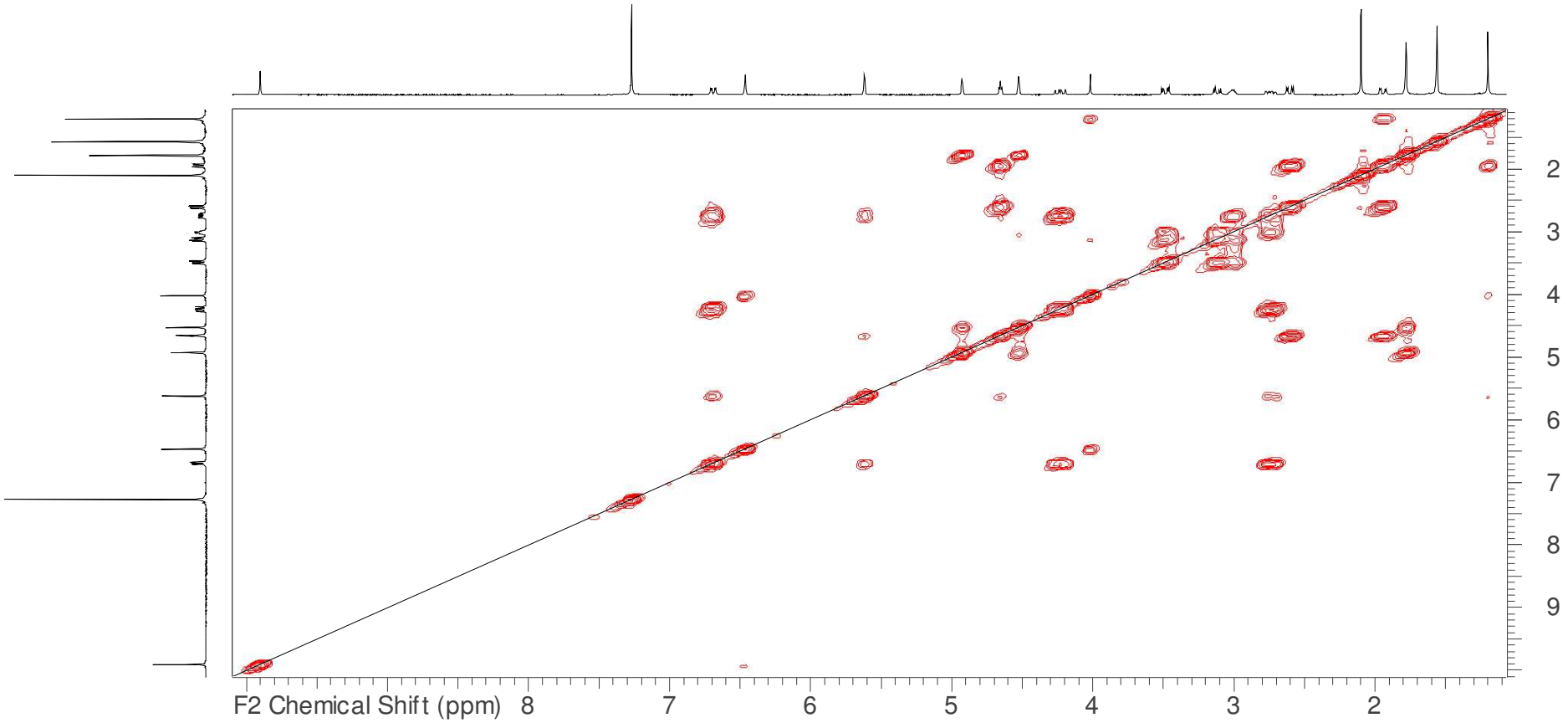

Figure S17. gCOSY of keikipukalide C (**3**) in $CDCl_3$, 400 MHz

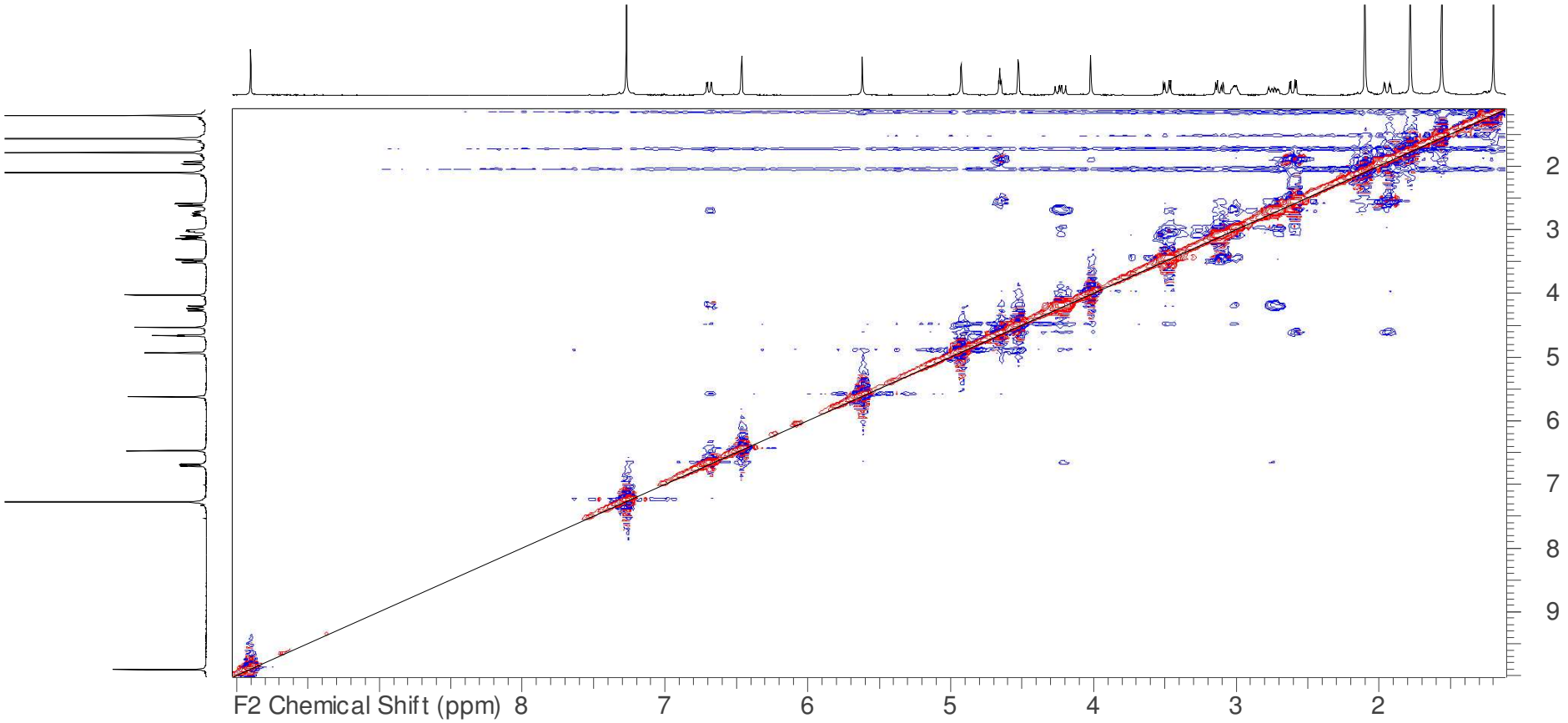

Figure S18. NOESY of keikipukalide C (**3**) in $CDCl_3$, 400 MHz

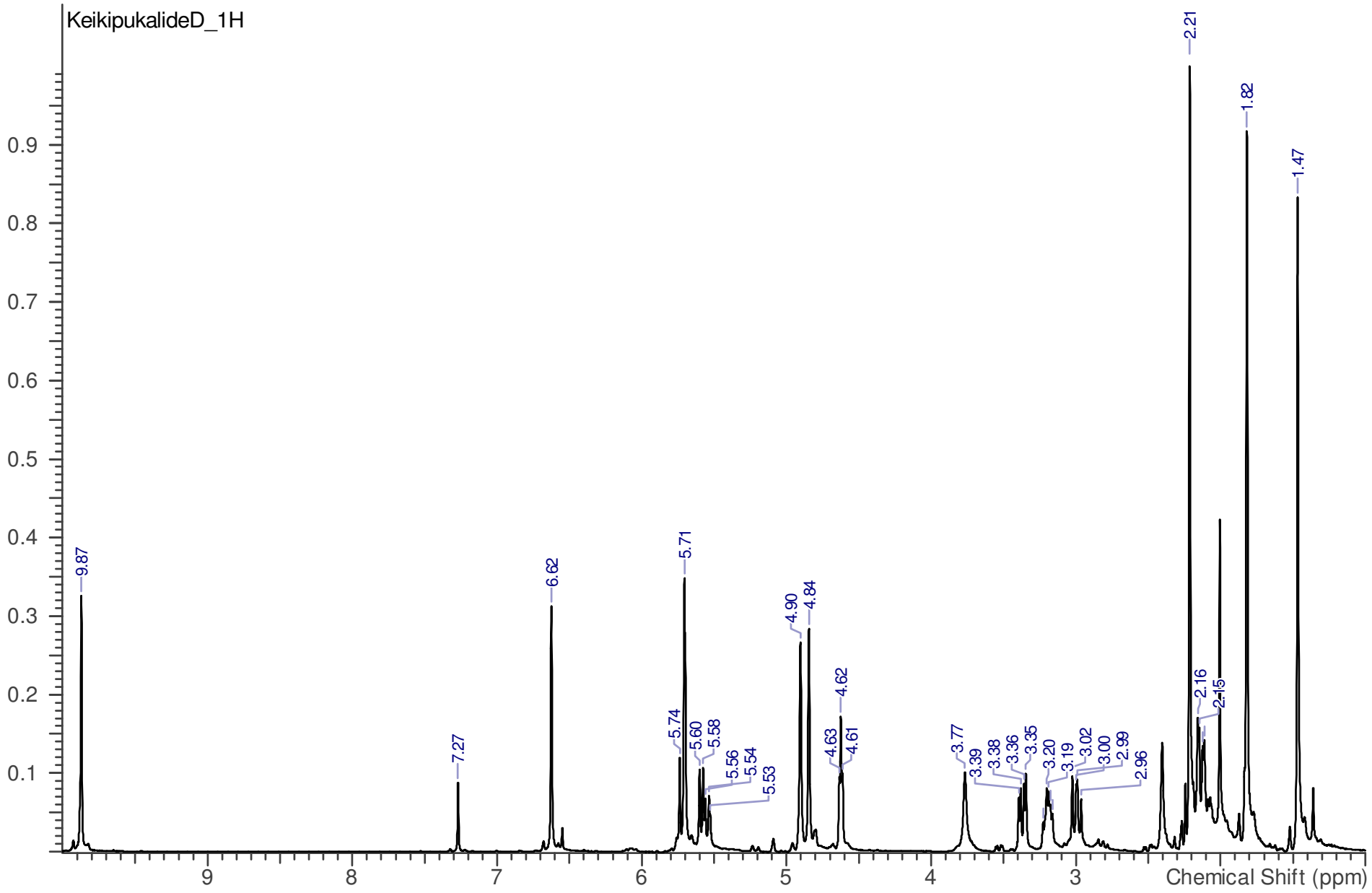

Figure S19. ^{1}H NMR spectrum of keikipukalide D (**4**) in $CDCl_3$, 400 MHz

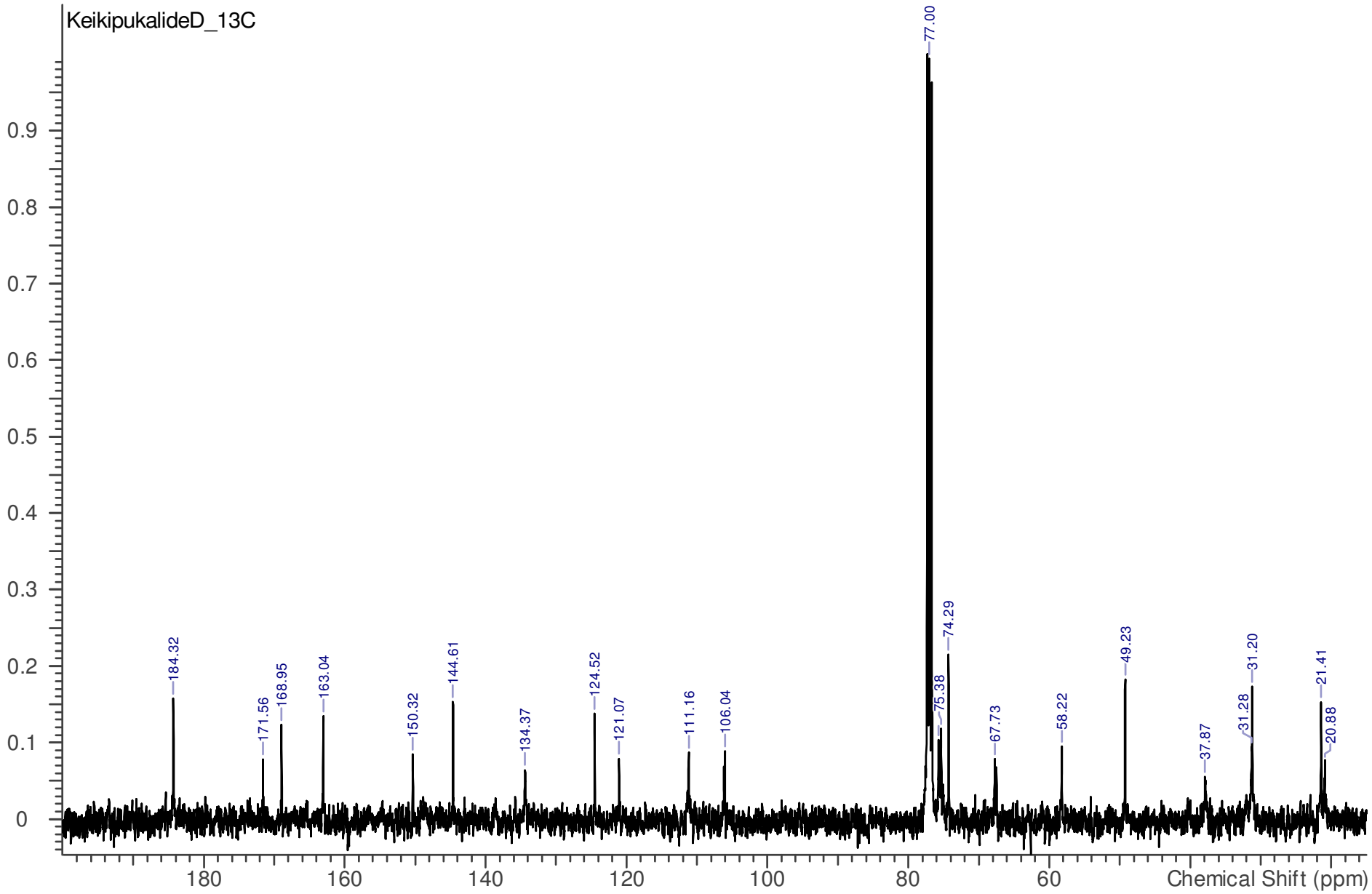

Figure S20. ^{13}C NMR spectrum of keikipukalide D (**4**) in $CDCl_3$, 100 MHz

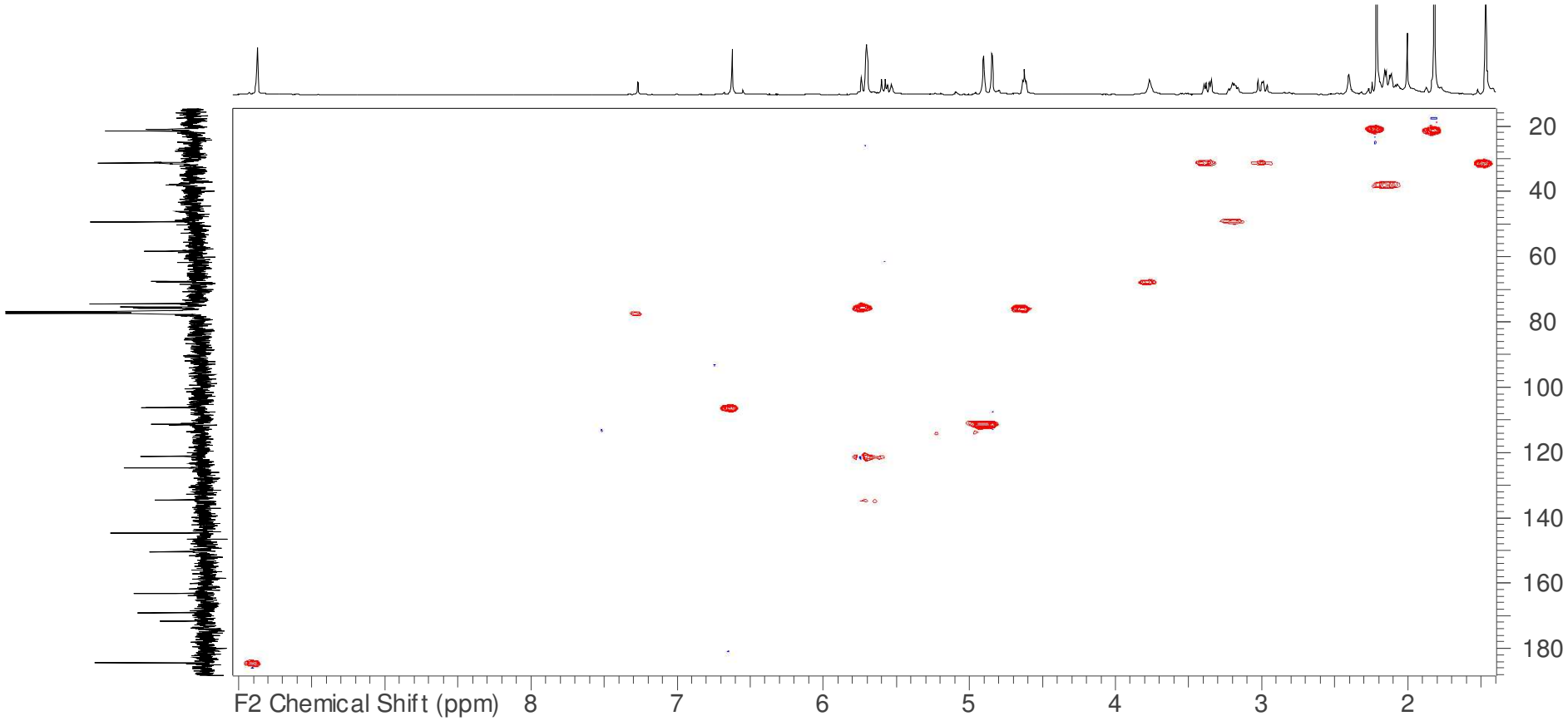

Figure S21. gHMQC of keikipukalide D (**4**) in $CDCl_3$, 400 MHz

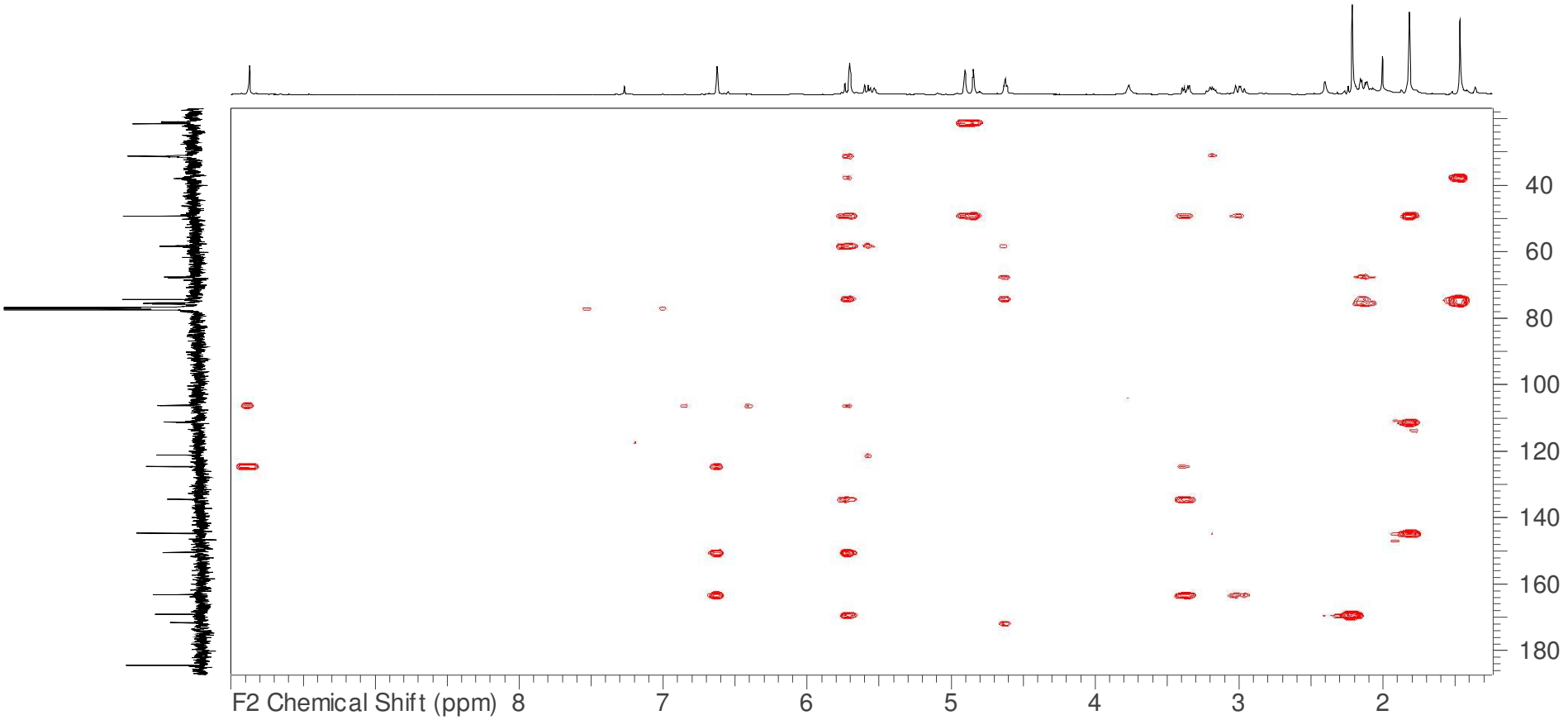

Figure S22. gHMBC of keikipukalide D (**4**) in $CDCl_3$, 400 MHz

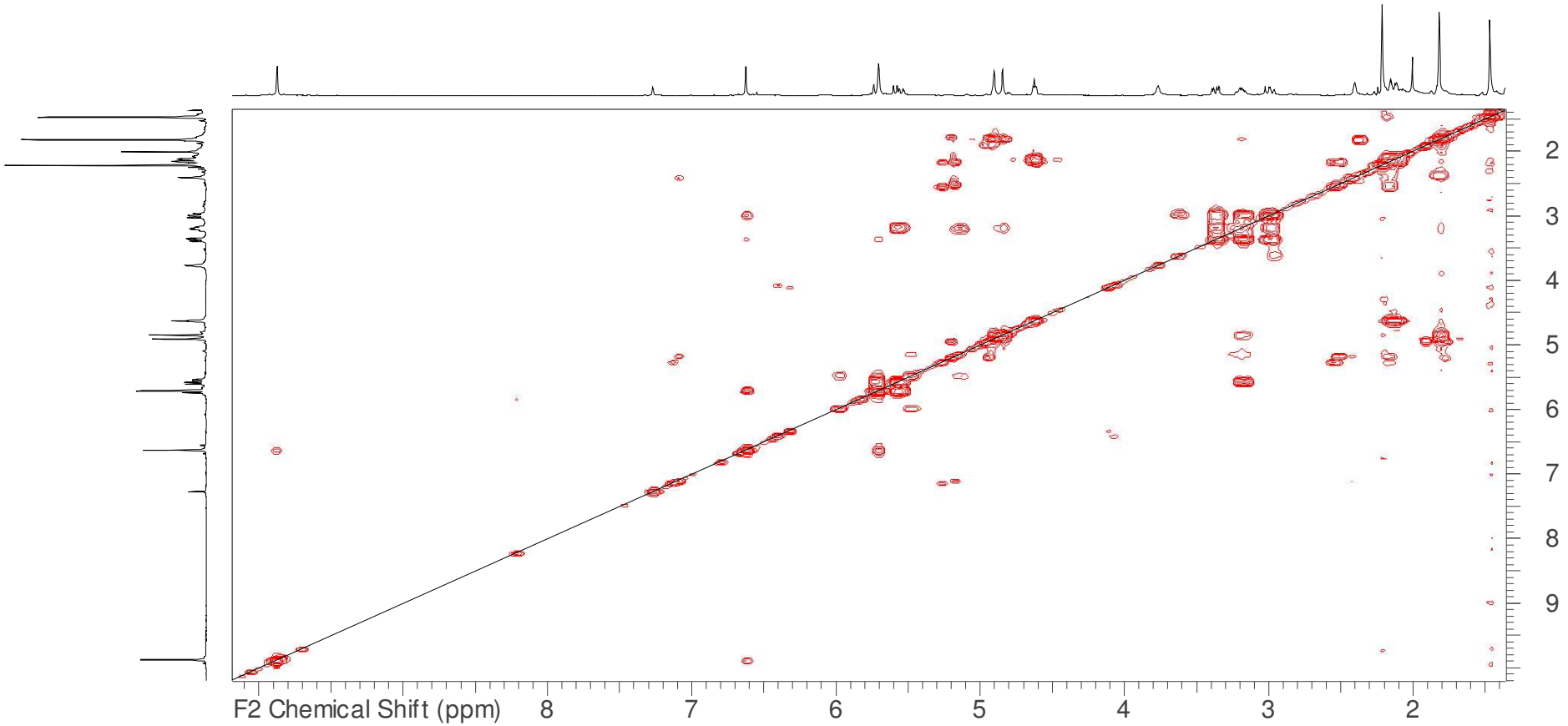

Figure S23. gCOSY of keikipukalide D (**4**) in $CDCl_3$, 500 MHz

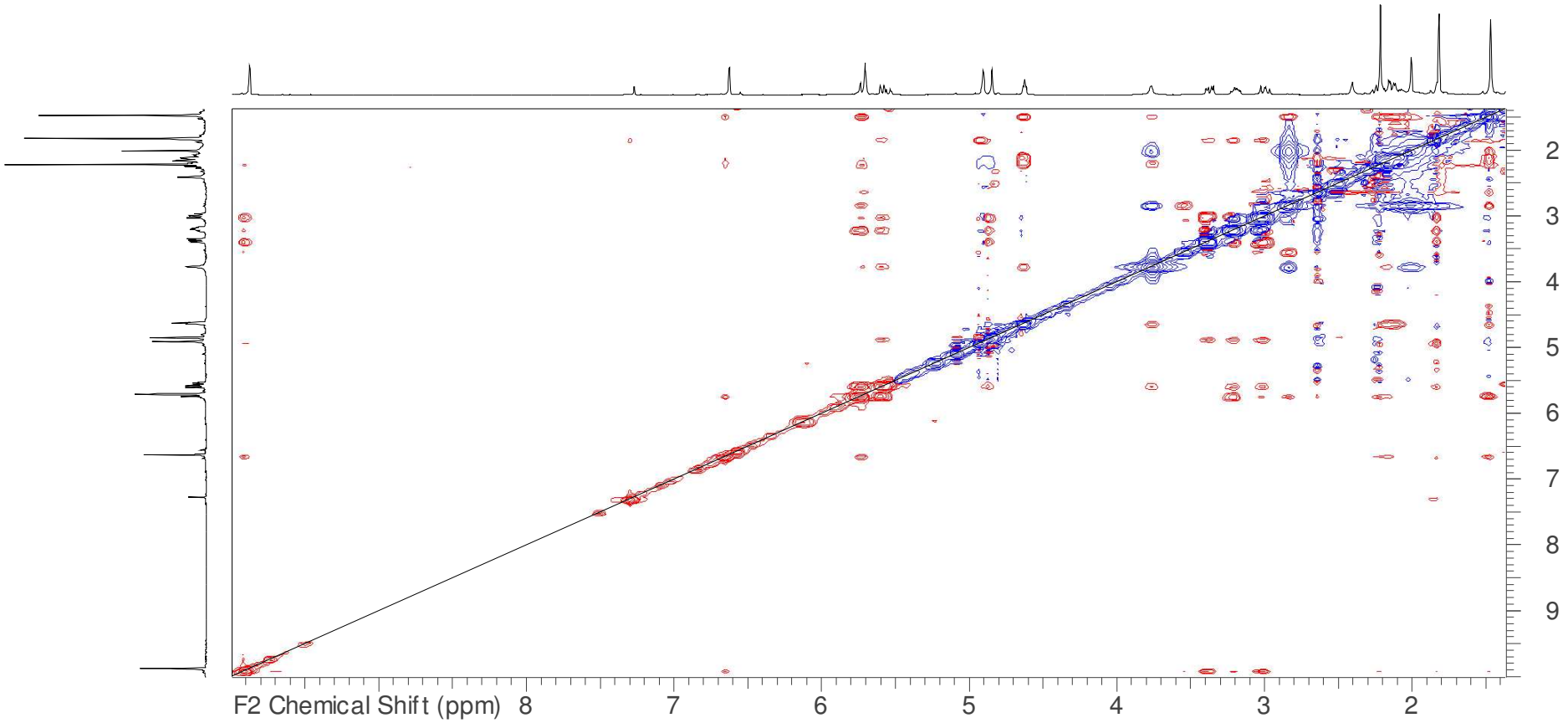

Figure S24. NOESY of keikipukalide D (**4**) in $CDCl_3$, 500 MHz

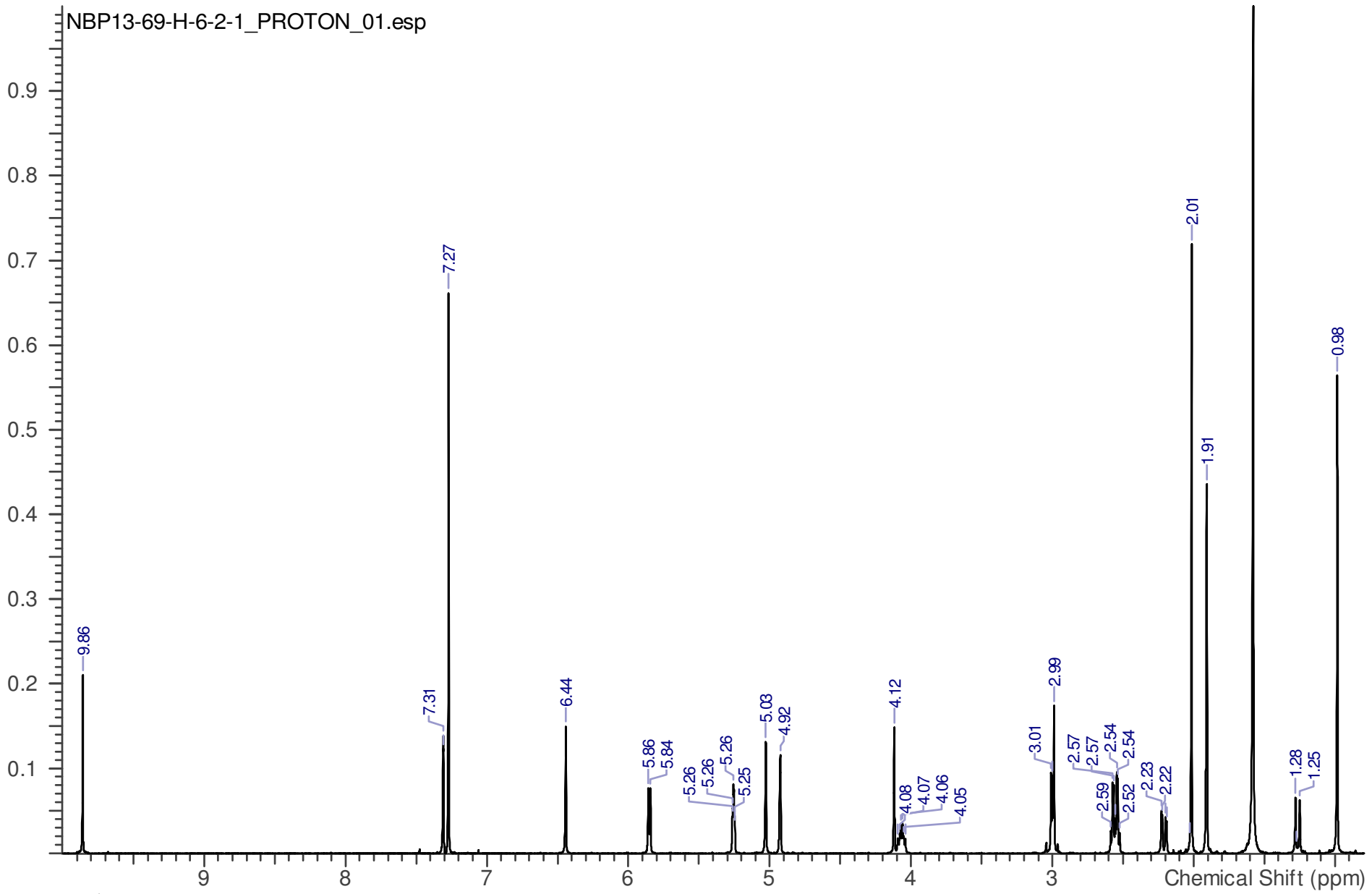

Figure S25. ^{1}H NMR spectrum of keikipukalide E (**5**) in $CDCl_3$, 500 MHz; 1.58 ppm is the water signal.

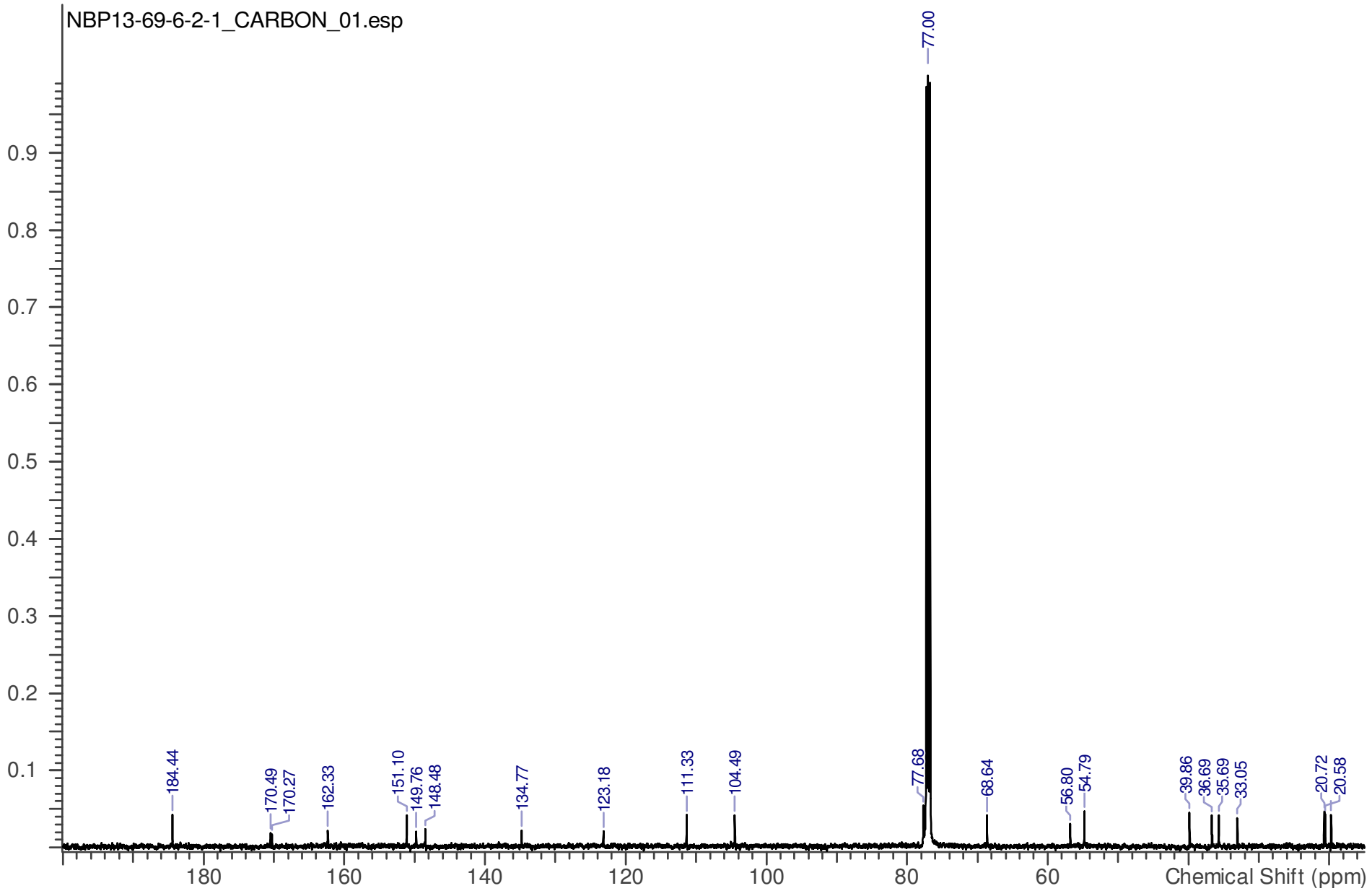

Figure S26. ^{13}C NMR spectrum of keikipukalide E (**5**) in $CDCl_3$, 125 MHz

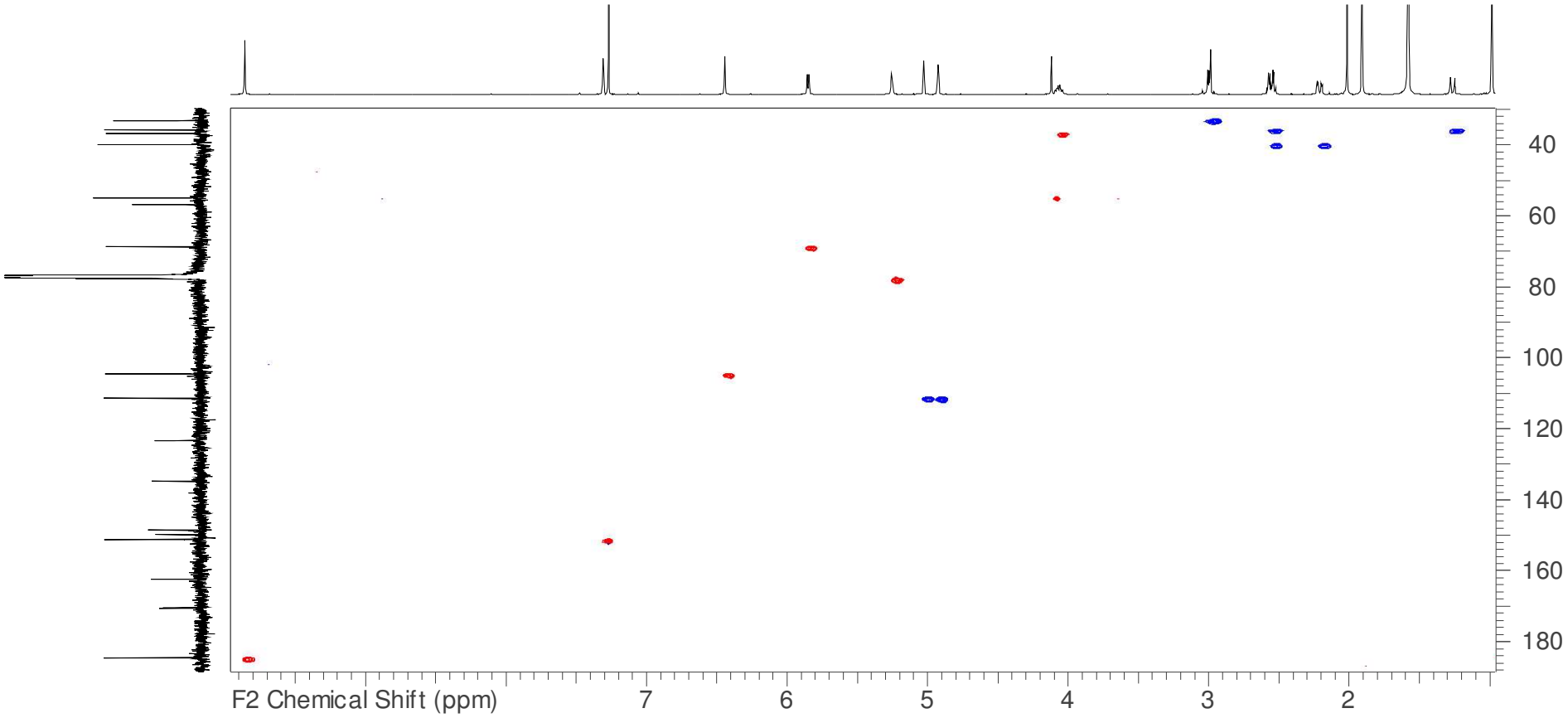

Figure S27. gHSQC of keikipukalide E (**5**) in $CDCl_3$, 600 MHz

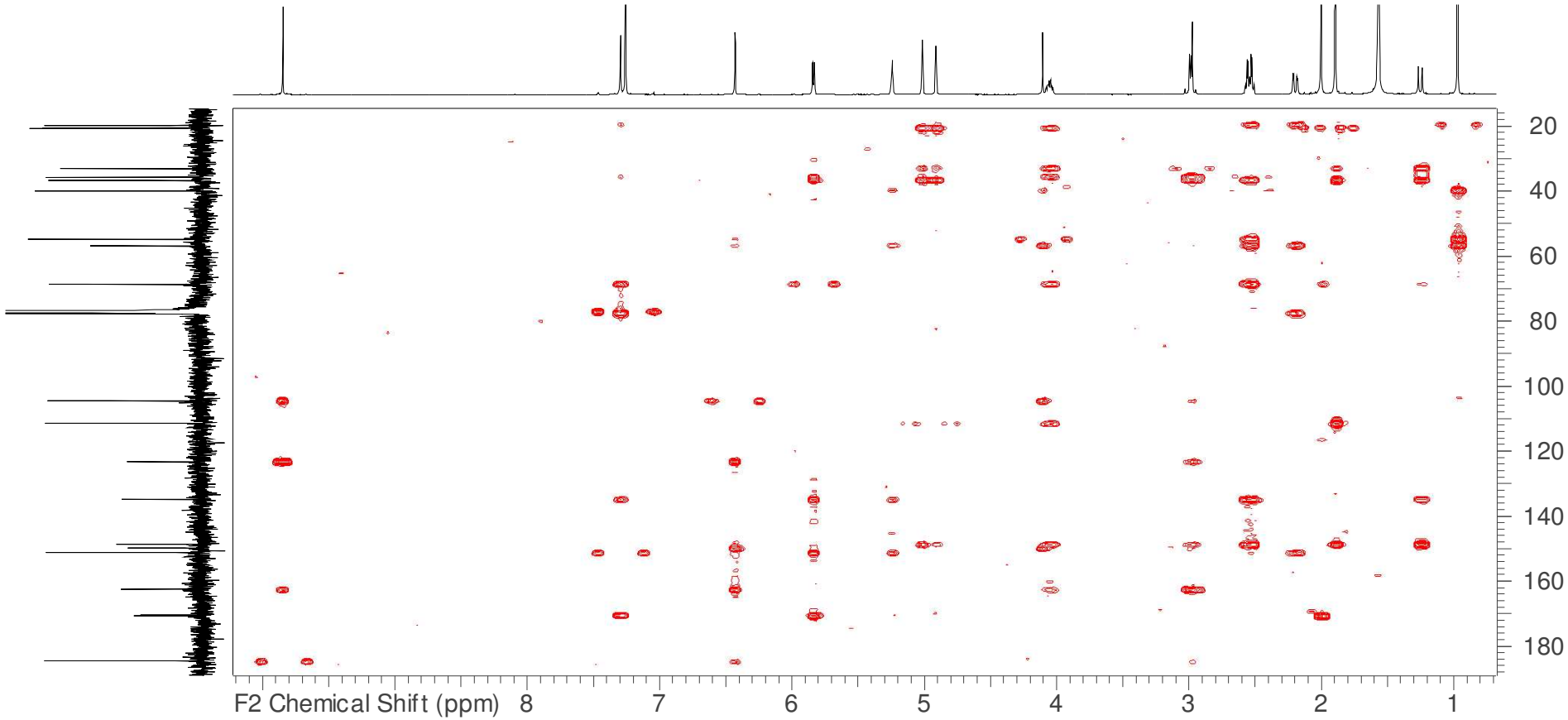

Figure S28. gHMBC of keikipukalide E (**5**) in $CDCl_3$, 500 MHz

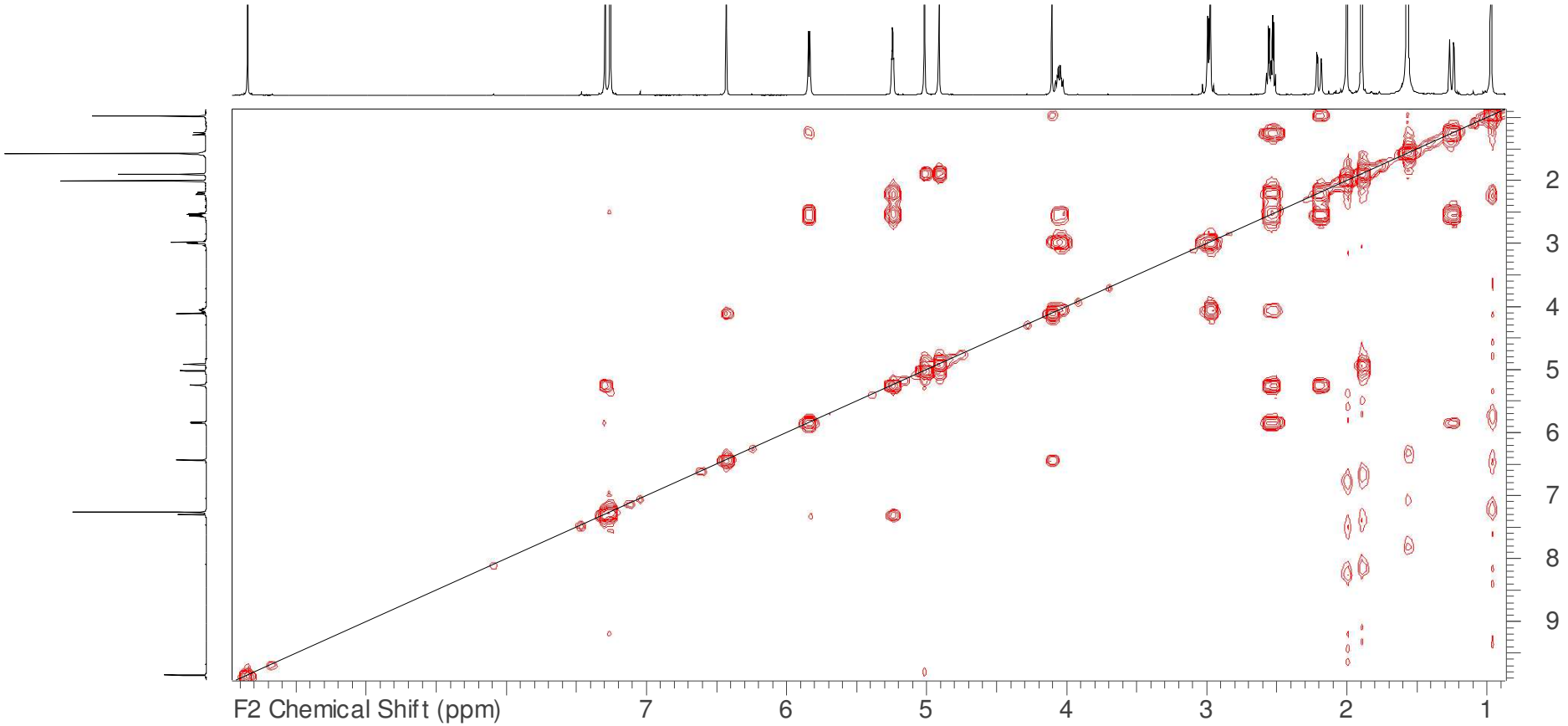

Figure S29. gCOSY of keikipukalide E (**5**) in $CDCl_3$, 500 MHz

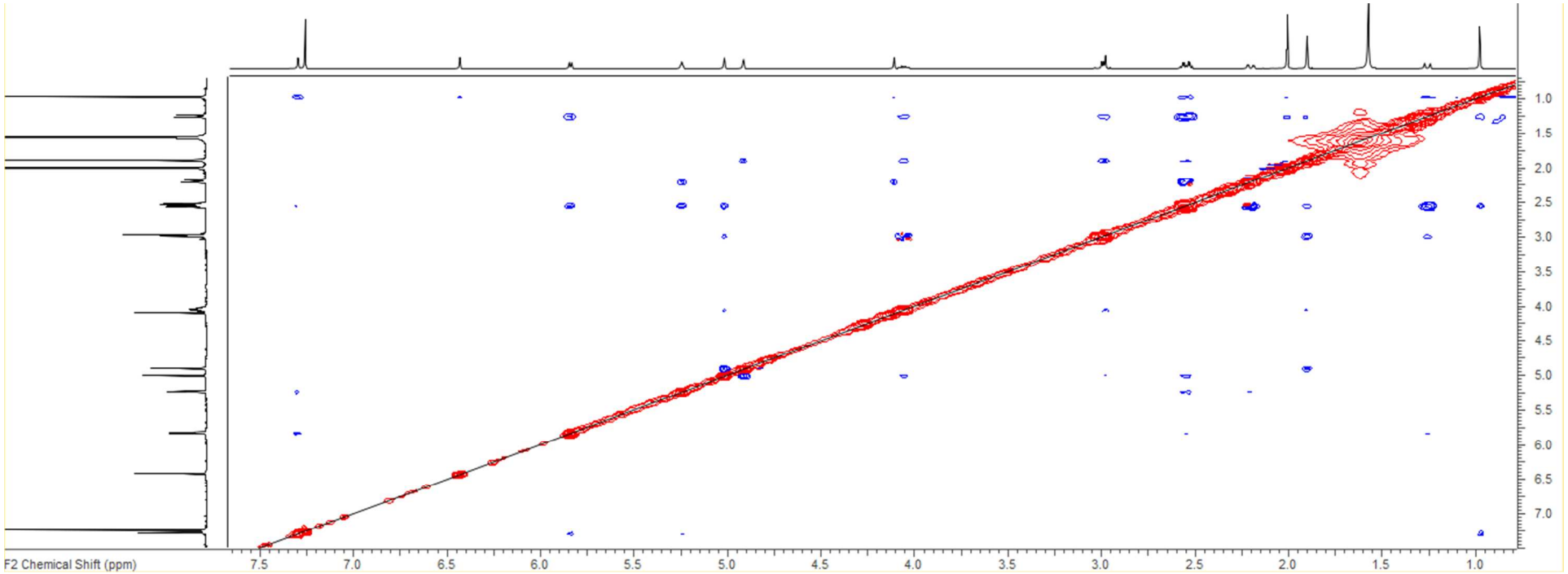

Figure S30. NOESY of keikipukalide E (**5**) in $CDCl_3$, 500 MHz

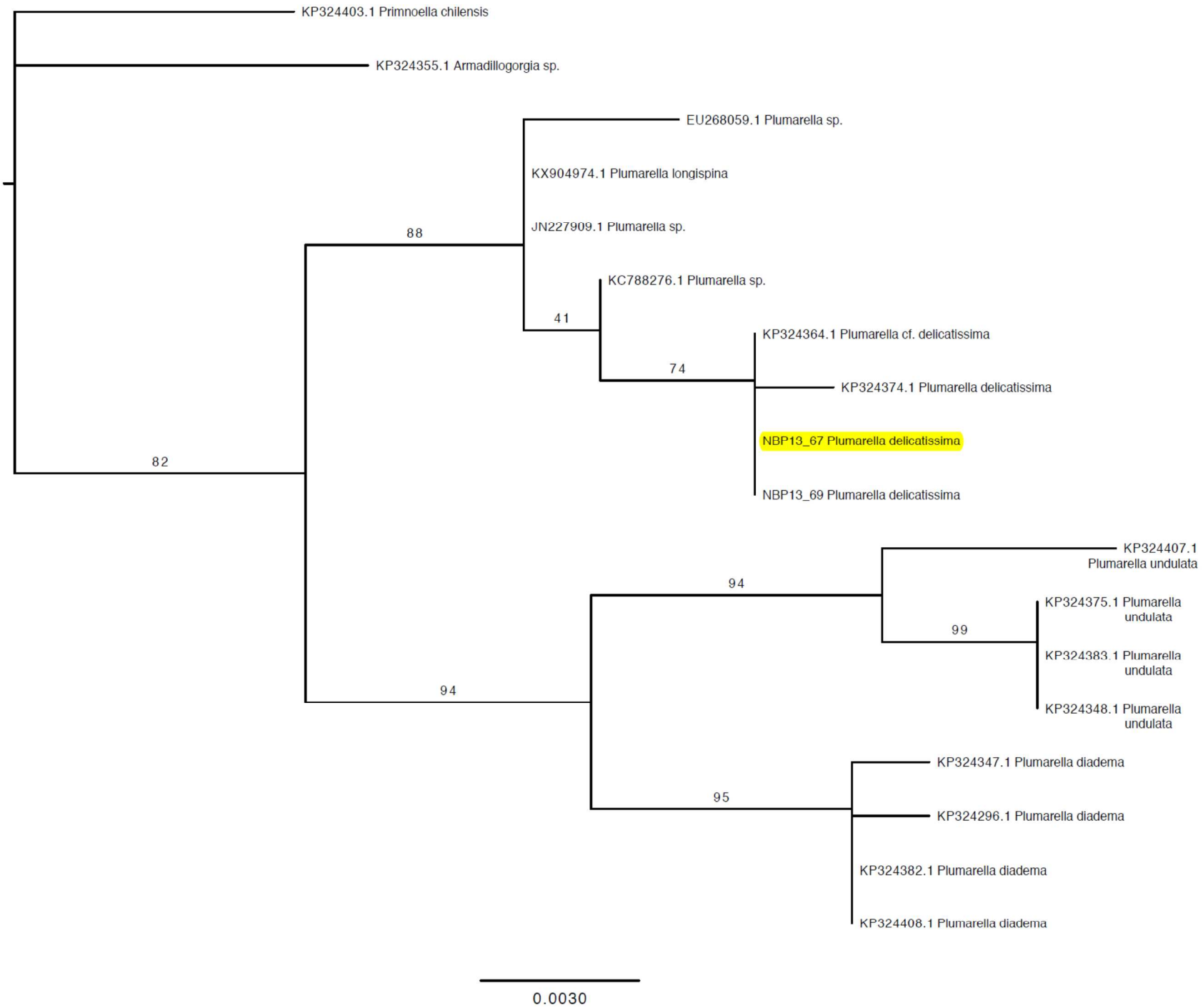

Figure S31. Maximum Likelihood tree topology comparing our *Plumarella* msh1 sequences with those available on Genbank

Table S1. Crystal Data and Structure Refinement for Keikipukalide A (**1**).	
Identification code	JAX_F2_3
Empirical formula	$C_{20}H_{20}O_6$
Formula weight	356.36
Temperature/K	100.0
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	6.74700(10)
b/Å	13.2449(3)
c/Å	18.8700(4)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1686.29(6)
Z	4
ρ_{calc}g/cm^3	1.404
μ/mm^{-1}	0.862
F(000)	752.0
Crystal size/mm^3	0.14 × 0.07 × 0.02
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/	8.156 to 154.438
Index ranges	$-8 \leq h \leq 8$, $-15 \leq k \leq 16$, $-23 \leq l \leq 23$
Reflections collected	25739
Independent reflections	3552 [R_{int} = 0.0710, R_{sigma} = 0.0356]
Data/restraints/parameters	3552/0/249
Goodness-of-fit on F^2	1.048
Final R indexes [I>=2σ (I)]	R_1 = 0.0352, wR_2 = 0.0698
Final R indexes [all data]	R_1 = 0.0433, wR_2 = 0.0731
Largest diff. peak/hole / e Å^{-3}	0.19/-0.18
Flack parameter	0.17(10)

Table S2. Results Bijvoet-Pair Analysis and Bayesian Statistics for Keikipukalide A (**1**).	
Space Group P212121	Student-T Prob. Plot
Wavelength 1.54178	Sample Size. 1478
Flack x .... 0.17(10)	Corr. Coeff. 0.999
Parsons z .. 0.20(10)	Intercept .. 0.016
	Slope 0.890
Bijvoet Pairs 1488	
Coverage ... 99	Bayesian Statistics
DiffCalcMax. 33.52	Student_T Nu 100
Outlier Crit 67.04	Select Pairs 1488
Scatter Plot	Theta_Min .. 7.73
Sigma Crit.. 0.25	Theta_Max .. 76.85
Select Pairs 106	**P2(true).... 1.000**
Number Plus 69	P3(true).... 0.981
Number Minus 37	P3(rac-twin) 0.019
Slope 0.902	P3(false) .. 0.4E-17
	G 0.6231
	G (su) 0.1761
	Hooft y ... 0.19(9)

Table S3. Crystal Data and Structure Refinement for Keikipukalide E (**5**).	
Identification code	NBP13_69_H_6_3
Empirical formula	$C_{22}H_{24}O_7$
Formula weight	400.41
Temperature/K	100
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	8.7671(2)
b/Å	11.4162(3)
c/Å	19.4875(4)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1950.44(8)
Z	4
ρ_{calc}g/cm^3	1.364
μ/mm^{-1}	0.845
F(000)	848.0
Crystal size/mm^3	0.31 × 0.116 × 0.03
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	8.978 to 154.788
Index ranges	$-11 \leq h \leq 10$, $-14 \leq k \leq 14$, $-24 \leq l \leq 24$
Reflections collected	29803
Independent reflections	4102 [R_{int} = 0.0664, R_{sigma} = 0.0323]
Data/restraints/parameters	4102/0/273
Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0344, wR_2 = 0.0782
Final R indexes [all data]	R_1 = 0.0401, wR_2 = 0.0812
Largest diff. peak/hole / e Å^{-3}	0.19/-0.18
Flack parameter	0.02(8)

Table S4. Results Bijvoet-Pair Analysis and Bayesian Statistics for Keikipukalide E (**5**).	
Space Group P212121	Student-T Prob. Plot
Wavelength 1.54178	Sample Size. 1734
Flack x .... 0.02(8)	Corr. Coeff. 0.999
Parsons z .. 0.04(8)	Intercept .. -0.029
	Slope 0.890
Bijvoet Pairs 1744	
Coverage ... 98	Bayesian Statistics
DiffCalcMax. 38.60	Student_T Nu 100
Outlier Crit 77.21	Select Pairs 1744
Scatter Plot	Theta_Min .. 6.76
Sigma Crit.. 0.25	Theta_Max .. 77.26
Select Pairs 151	**P2(true).... 1.000**
Number Plus 94	P3(true).... 1.000
Number Minus 57	P3(rac-twin) 0.9E-09
Slope 1.030	P3(false) .. 0.9E-38
	G 0.9530
	G (su) 0.1473
	Hooft y ... 0.02(7)