C-Hexaphenyl-substituted trianglamine as chiral solvating agent for carboxylic acids

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Electronic Supplementary Information

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Calculated ECD and UV spectra for **1b** and **1c**: S13-S14

Relative energies, percentage populations and values of torsion angles calculated for **1b** and **1c**: S15-S16

General information:

NMR spectra were recorded on a Varian MR 400 (400 MHz) instrument at 25 °C using $CDCl_3$ as a solvent purchased from Sigma-Aldrich. Chemical shifts are reported in ppm relative to the solvent residual peak of $CDCl_3$ (δ_H 7.27). Spectral assignments were obtained by analysis of chemical shifts and interpretation of ^1H, ^{13}C, gradient COSY, adiabatic gradient HSQC, adiabatic gradient HMBC NMR spectra for a 1:2 solution of **1c** and racemic **3a** in $CDCl_3$. Gradient COSY spectrum was acquired with 4 transient for 256 increments with a relaxation delay of 1 s; adiabatic gradient HSQC and HMBC were acquired with 4 transient for 1024 increments with a relaxation delay of 1 s and processed by weighting both dimensions with a square sine bell function. Bidimensional ROESY was acquired with a mixing time of 500 ms, a relaxation delay of 1 s by collecting 16 transients for each of 1024 increments and processed by weighting both dimensions with a square sine bell function. Selective 1D-ROESY spectra were acquired by irradiation of a 25 Hz band with a mixing time of 600 ms, a relaxation time of 1 s and by collecting 11000 FIDs.

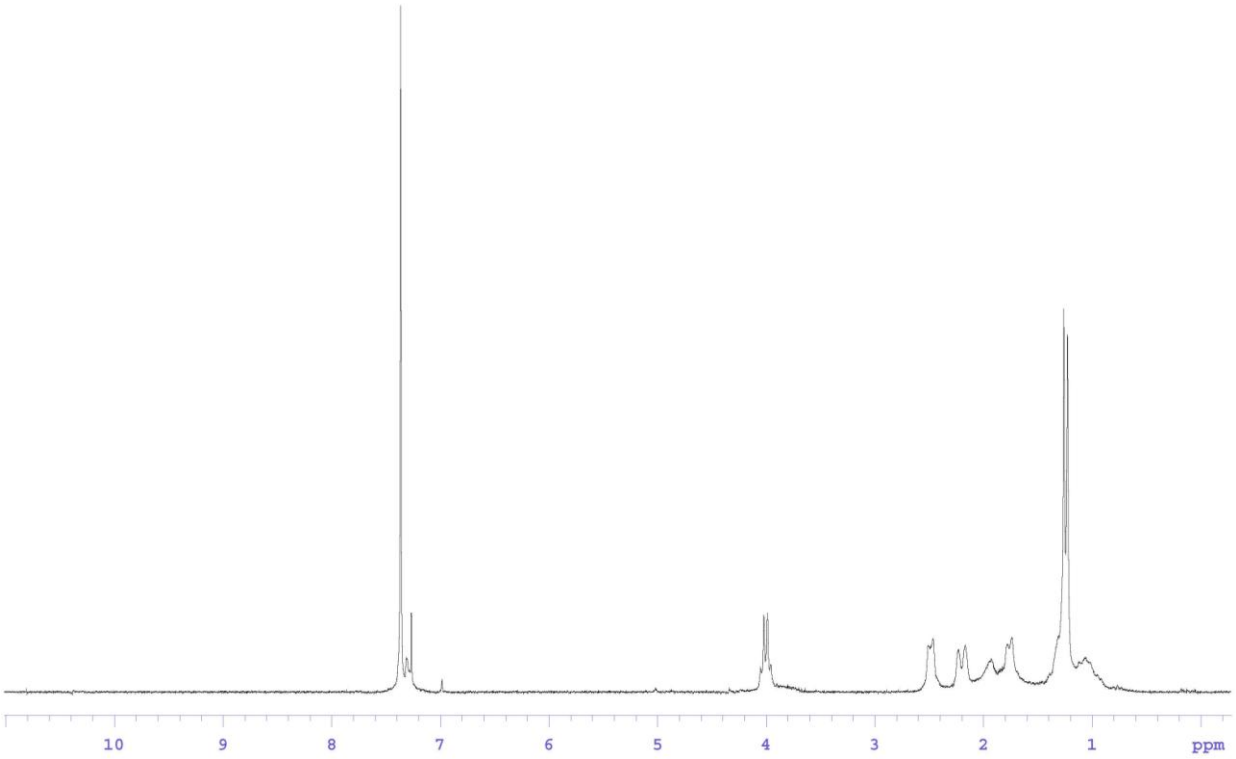

Figure 1. ^{1}H NMR spectra of **1b**

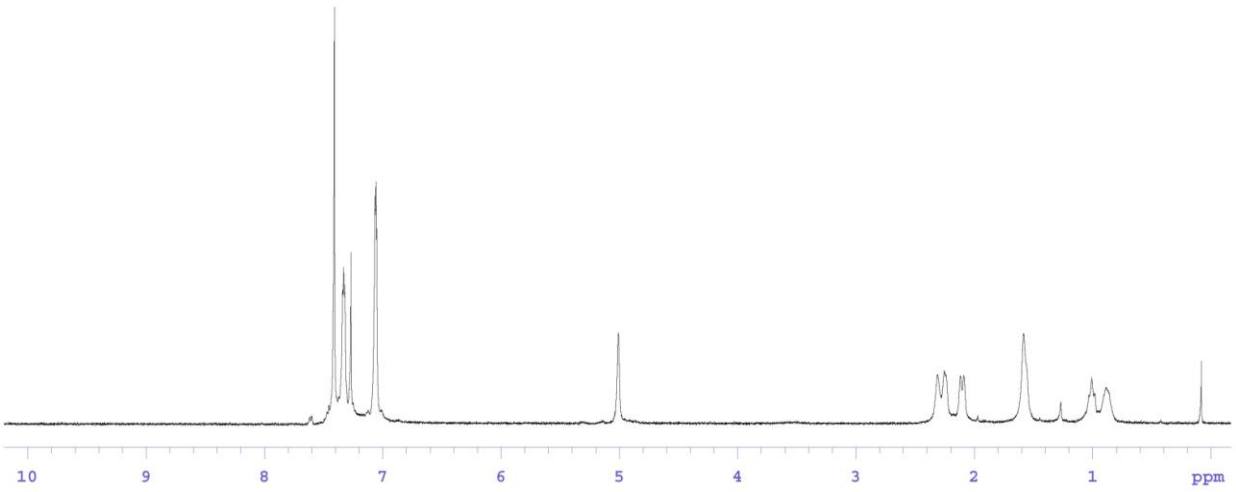

Figure 2. ^{1}H NMR spectra of **1c**

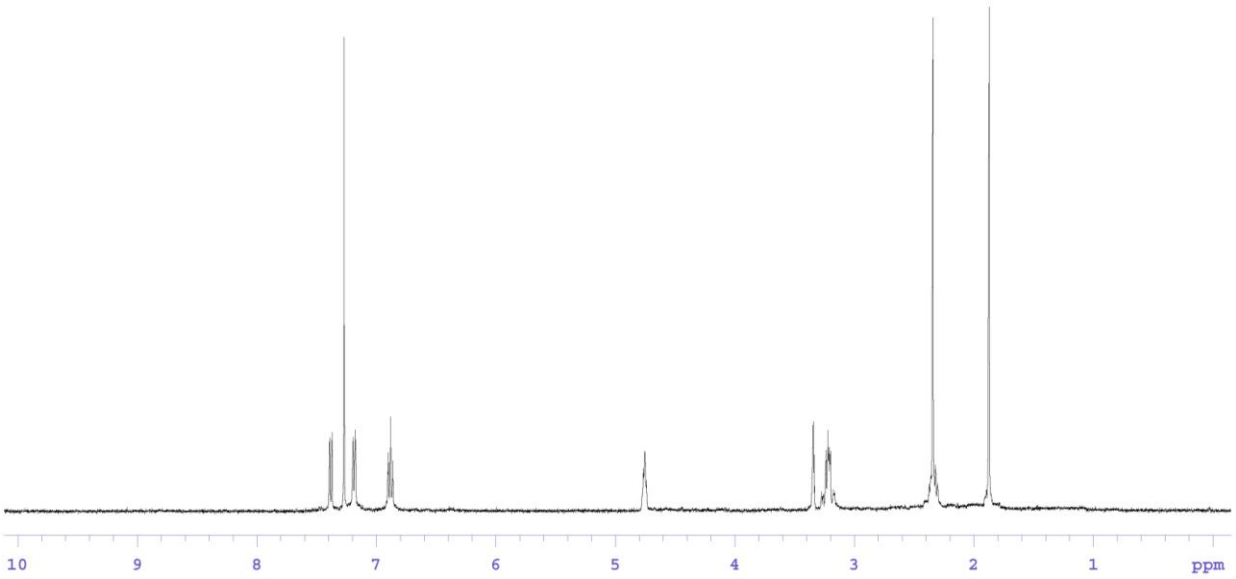

Figure 3. ^{1}H NMR spectra of **6**

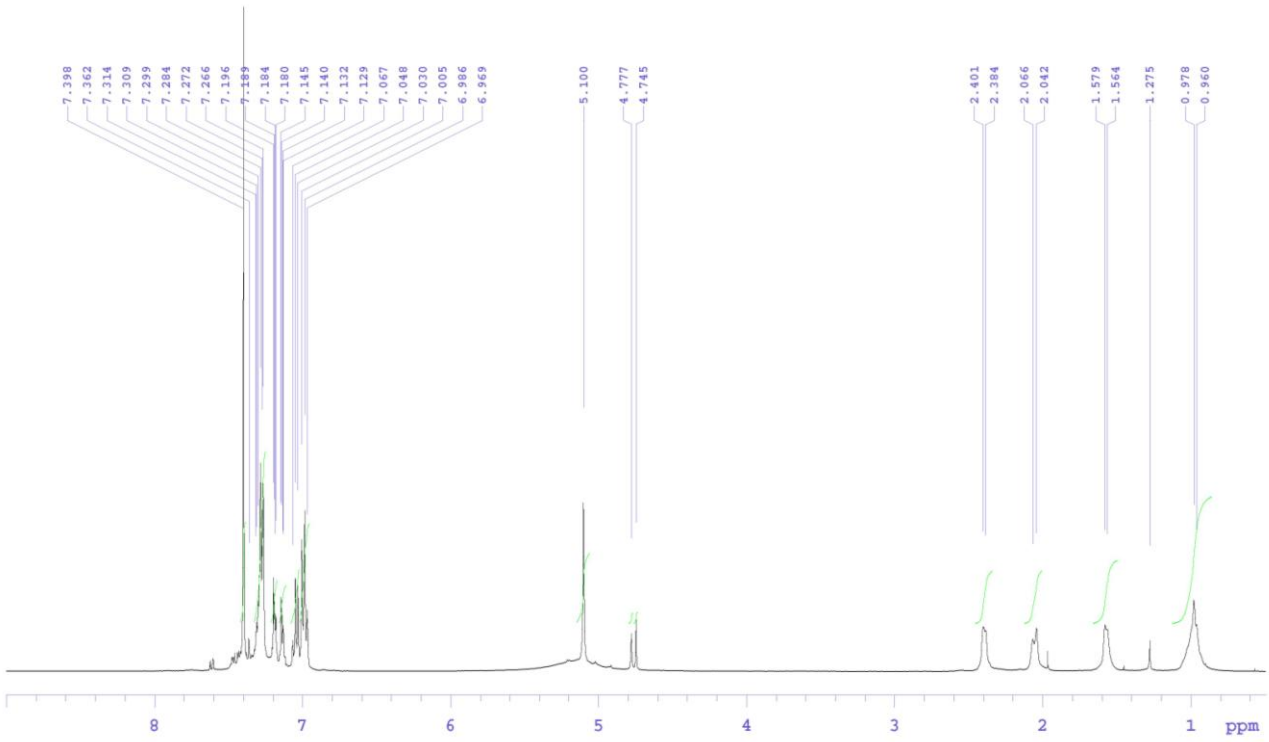

Figure 4. ^{1}H NMR spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

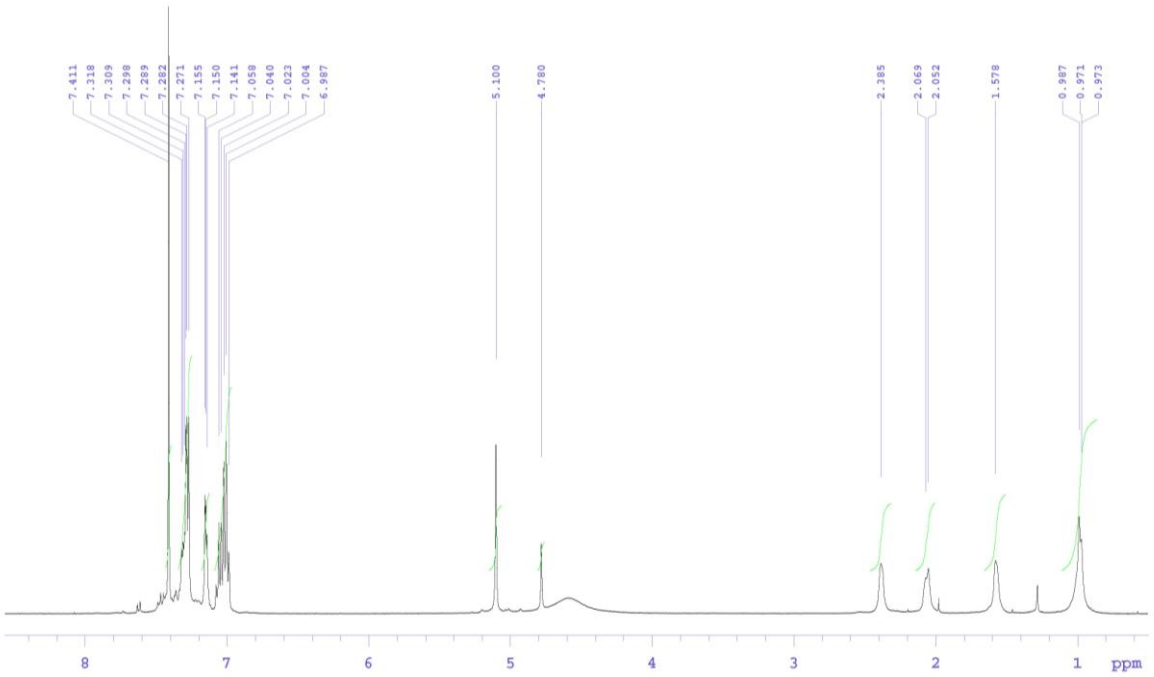

Figure 5. ^{1}H NMR spectra of a 1:2 mixture of **1c** with (*R*)-**3a**.

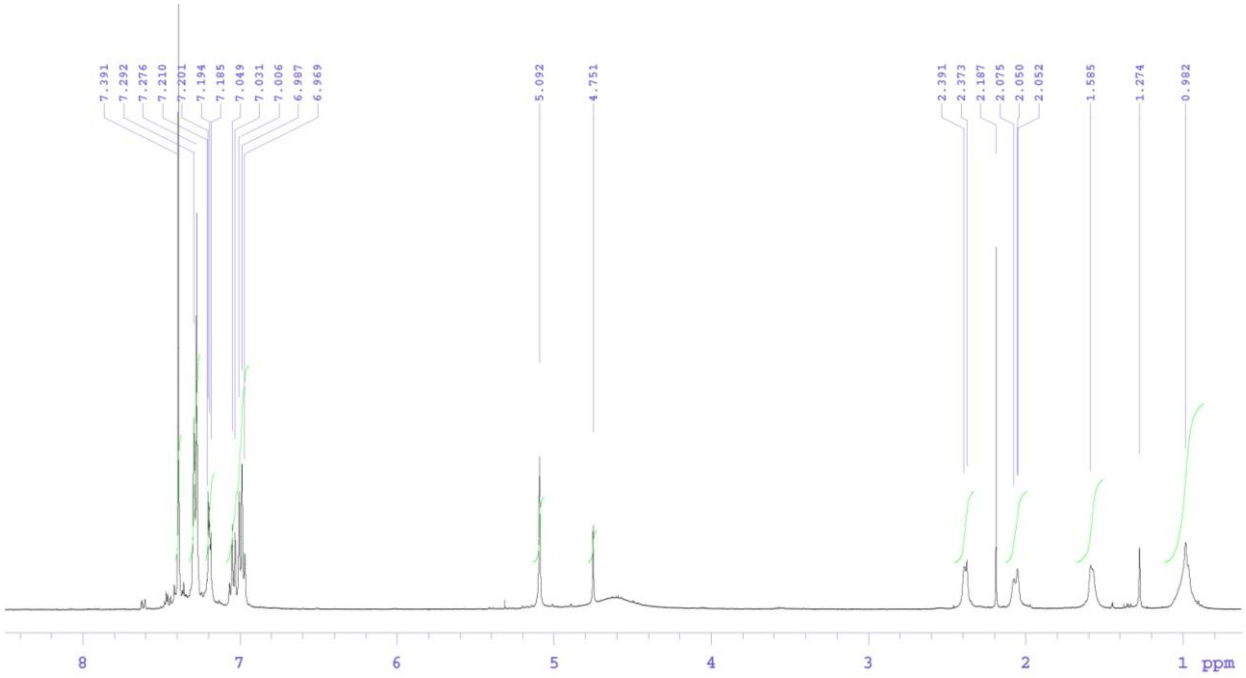

Figure 6. [1]H NMR spectra of a 1:2 mixture of **1c** with (*S*)-**3a**.

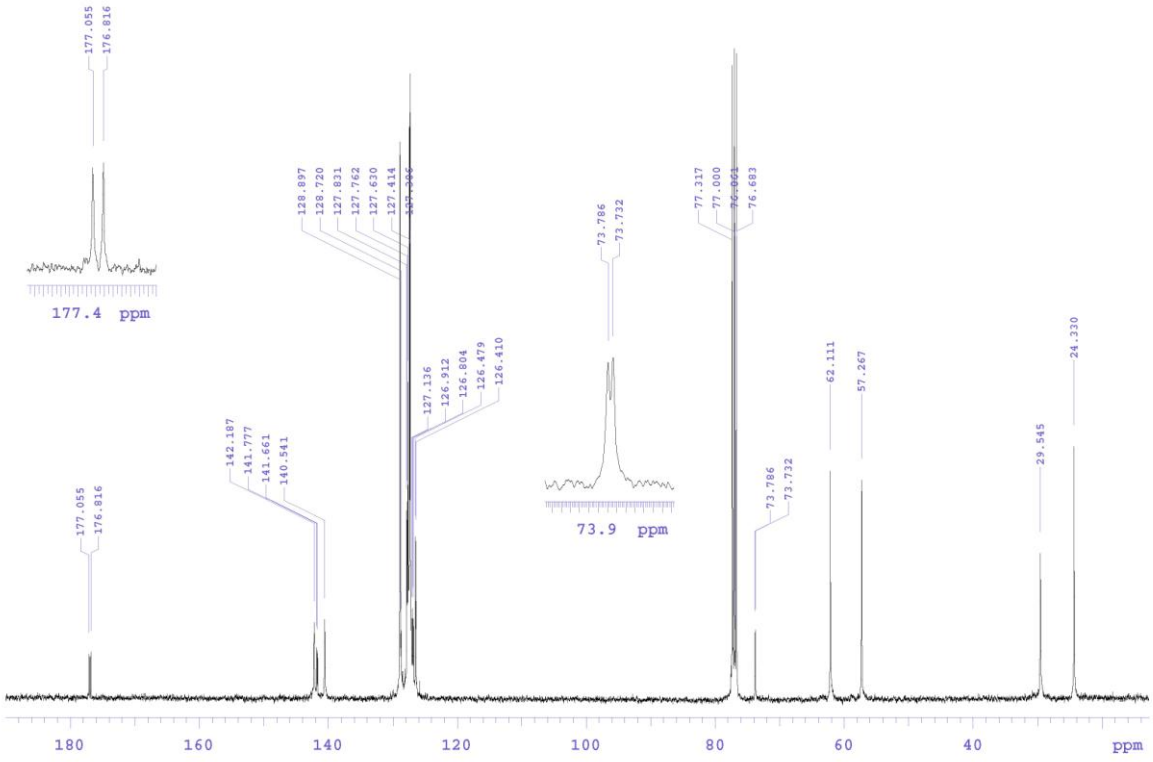

Figure 7. [13]C NMR spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

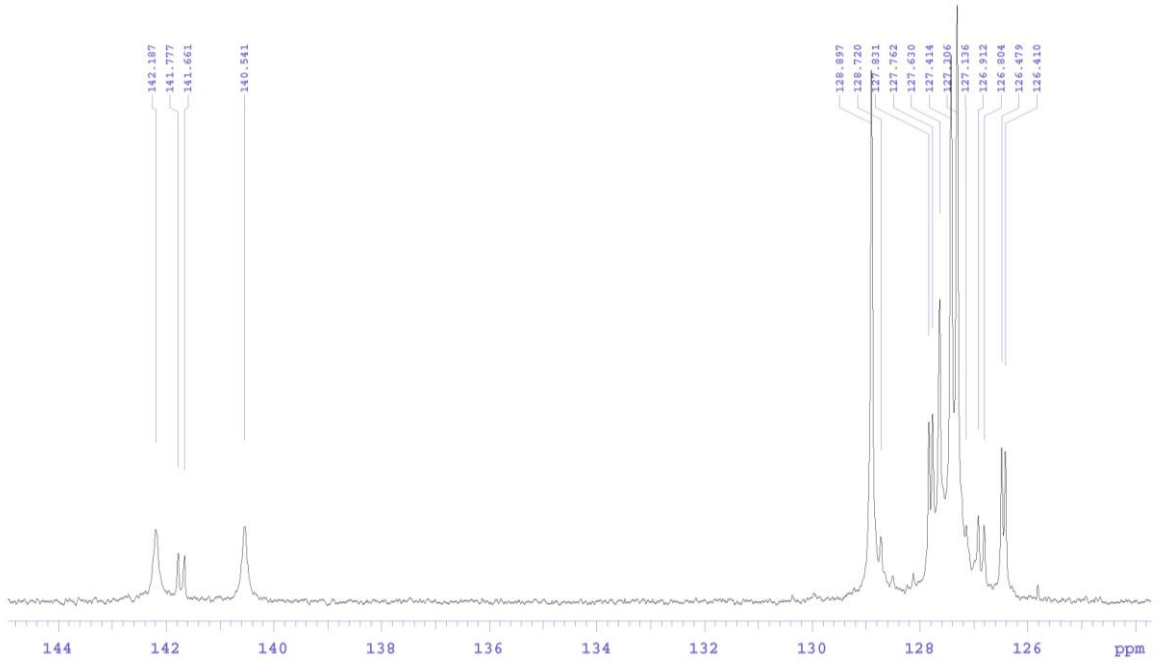

Figure 8. Partially ^{13}C NMR spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

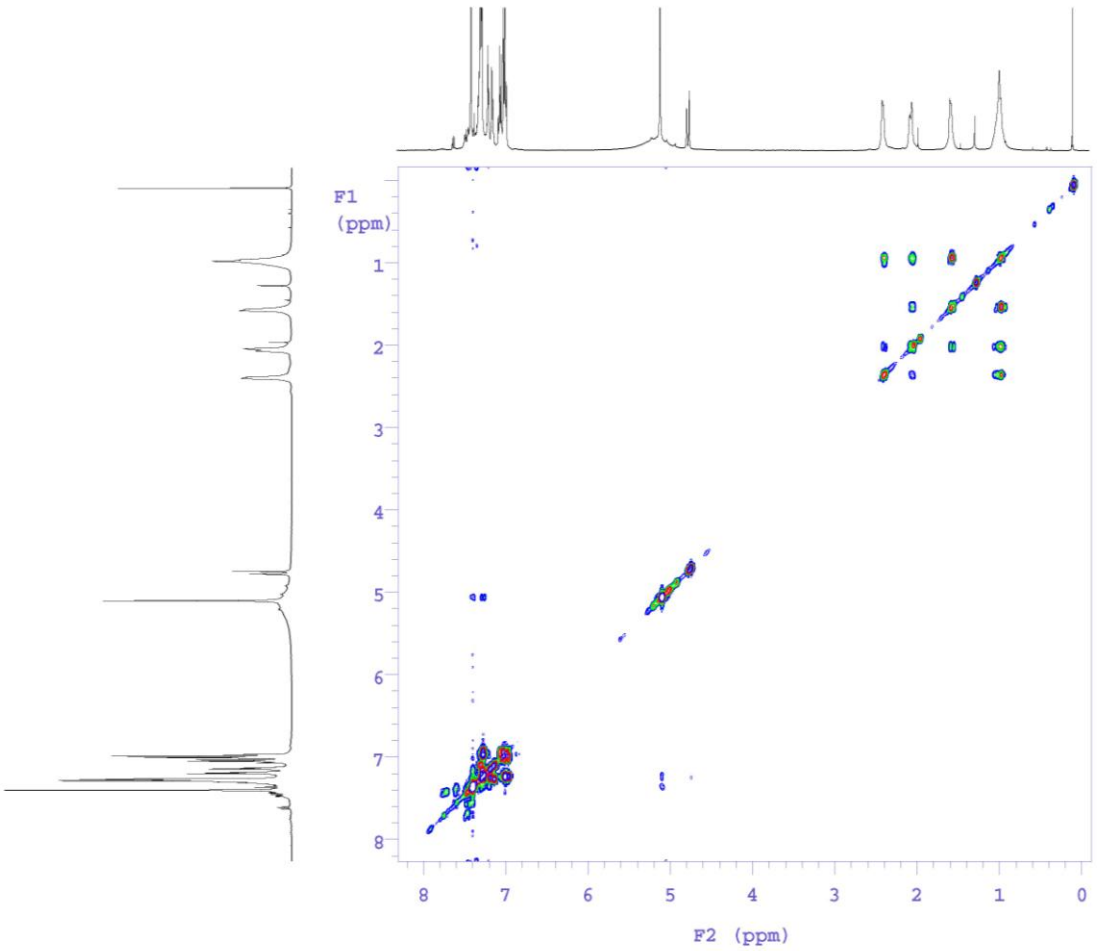

Figure 9. gCOSY spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

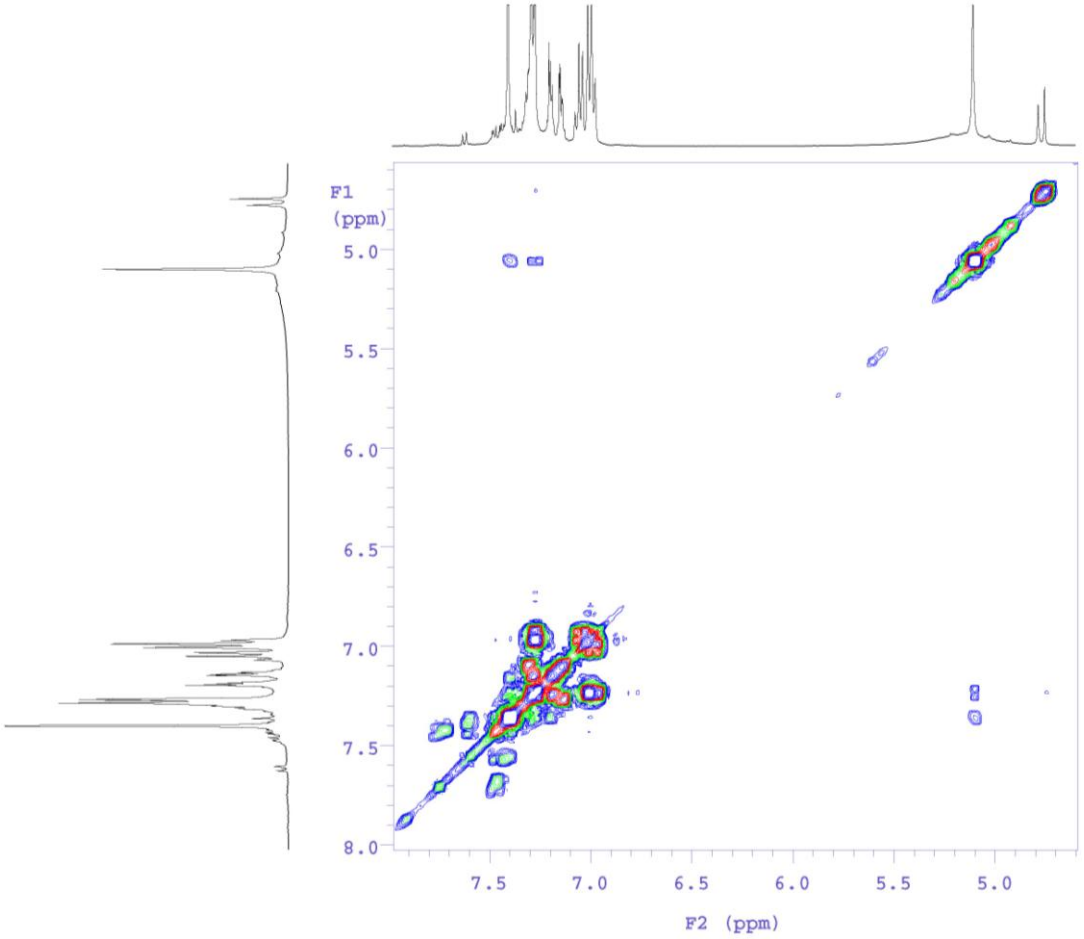

Figure 10. Partially gCOSY spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

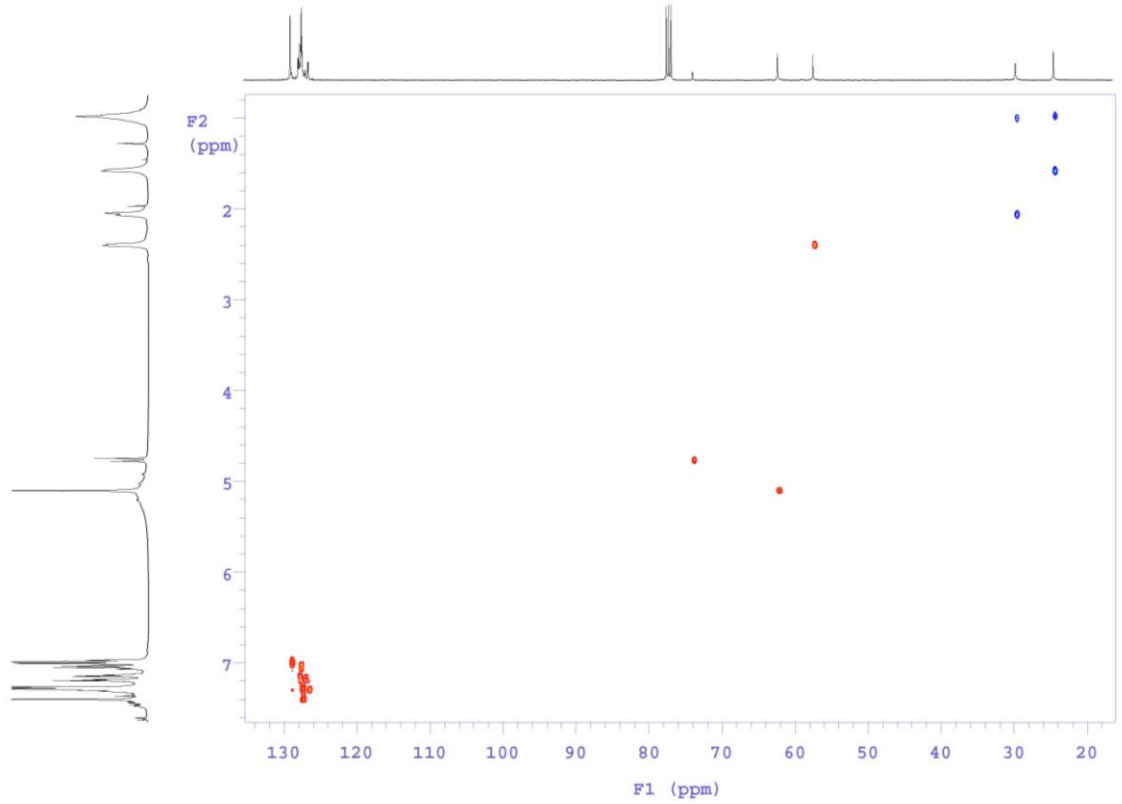

Figure 11. gHSQC spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

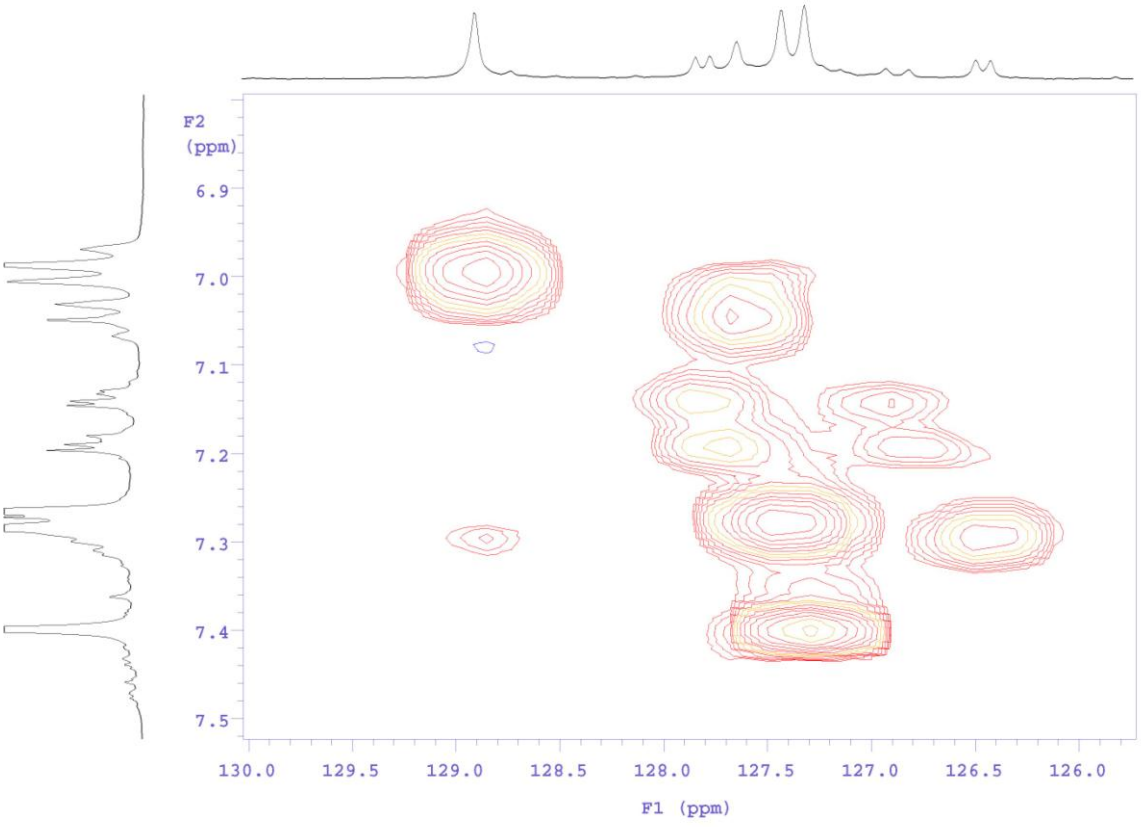

Figure 12. Partially gHSQC spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

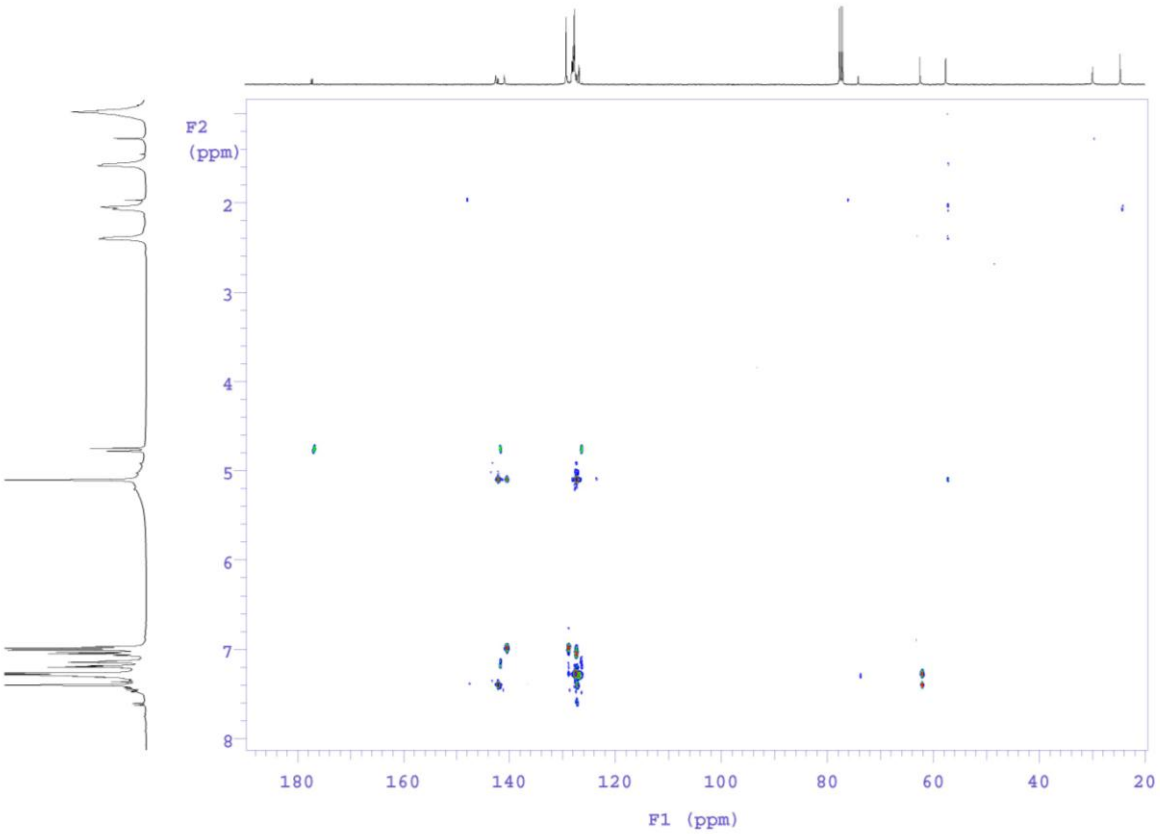

Figure 13. gHMBC spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

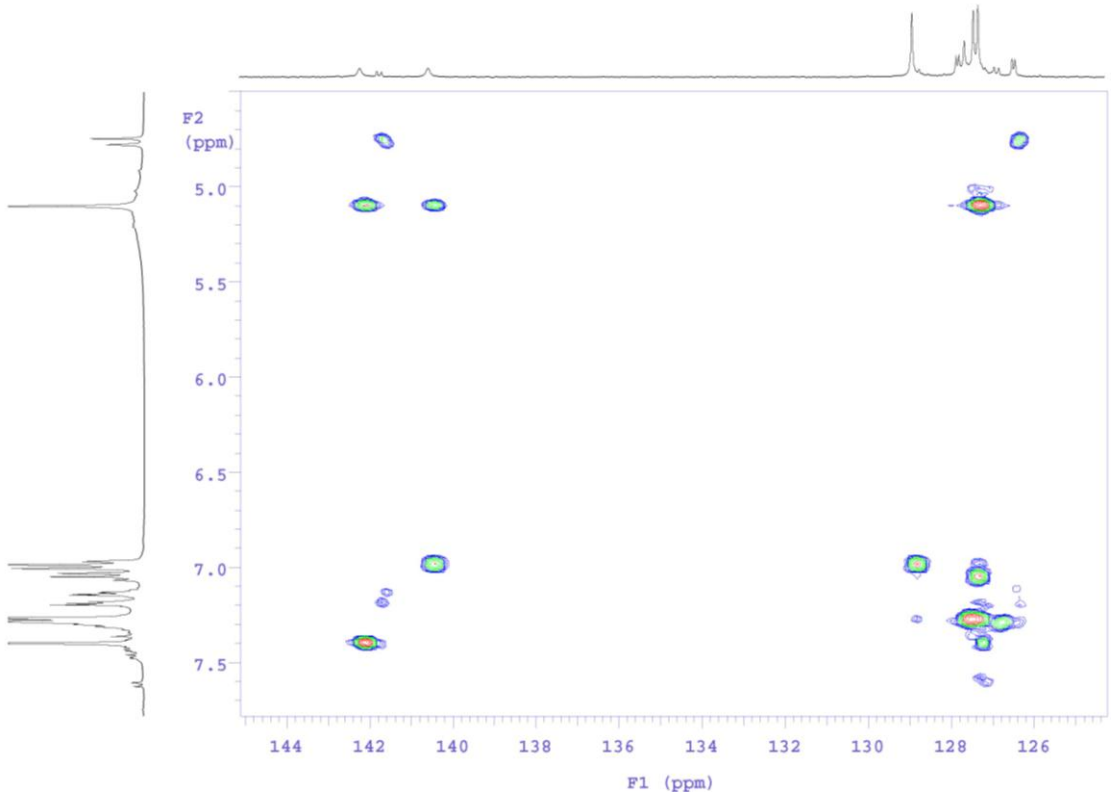

Figure 14. Partially gHMBC spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

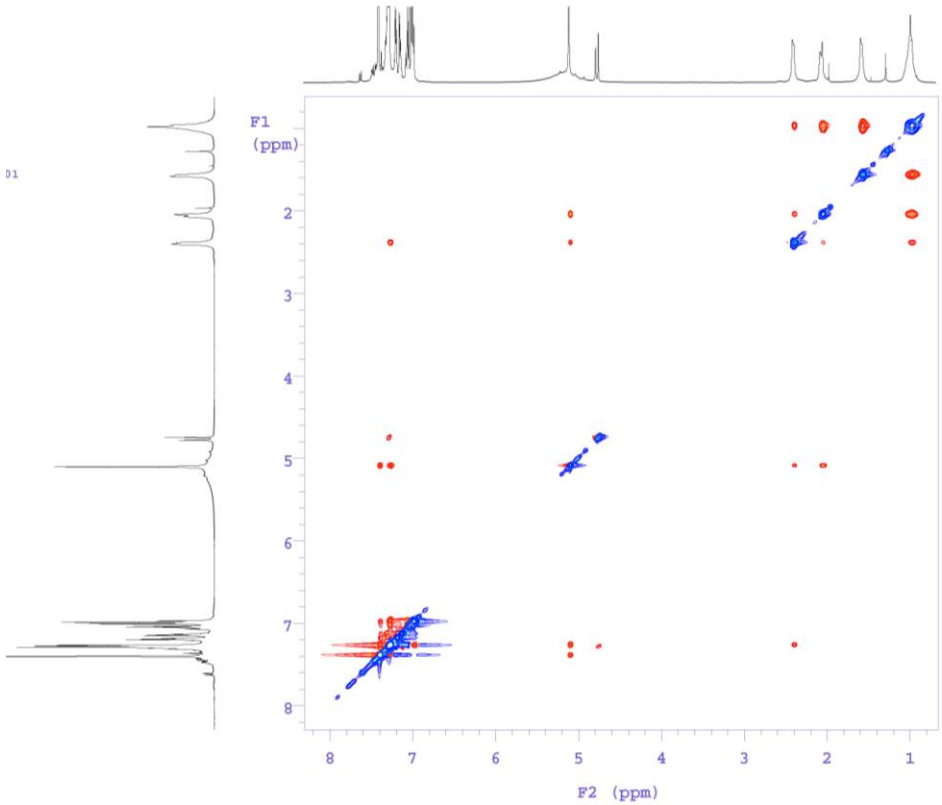

Figure 15. ROESY spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

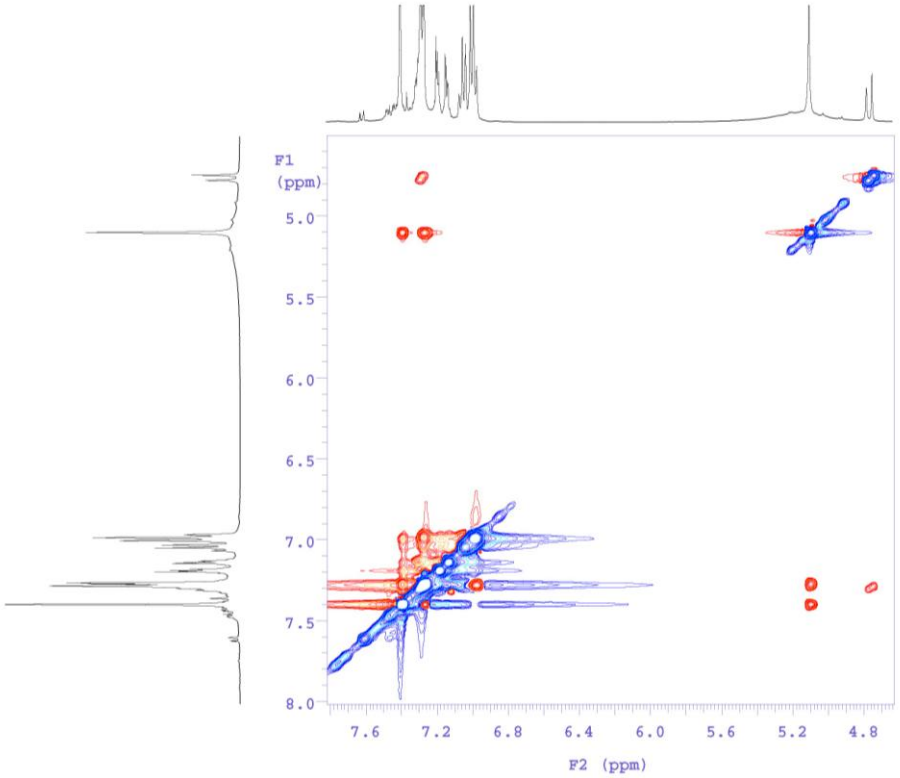

Figure 16. Partially ROESY spectra of a 1:2 mixture of **1c** with *rac*-**3a**.

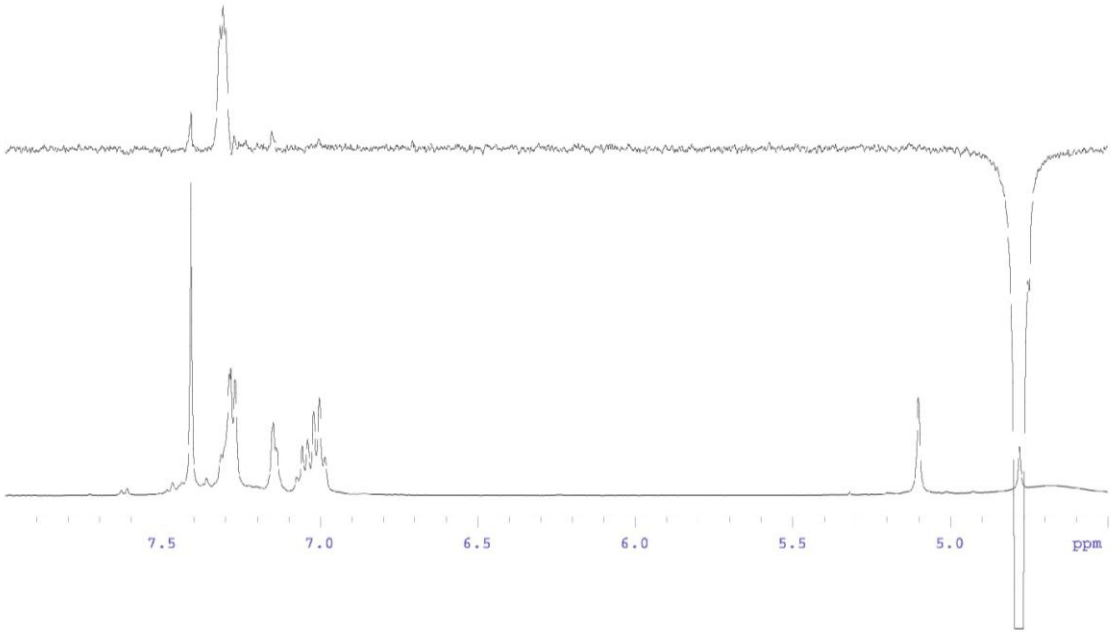

Figure 17. Partially 1D ROESY and ^{1}H NMR spectra of a 1:2 mixture of **1c** with (*R*)-**3a**.

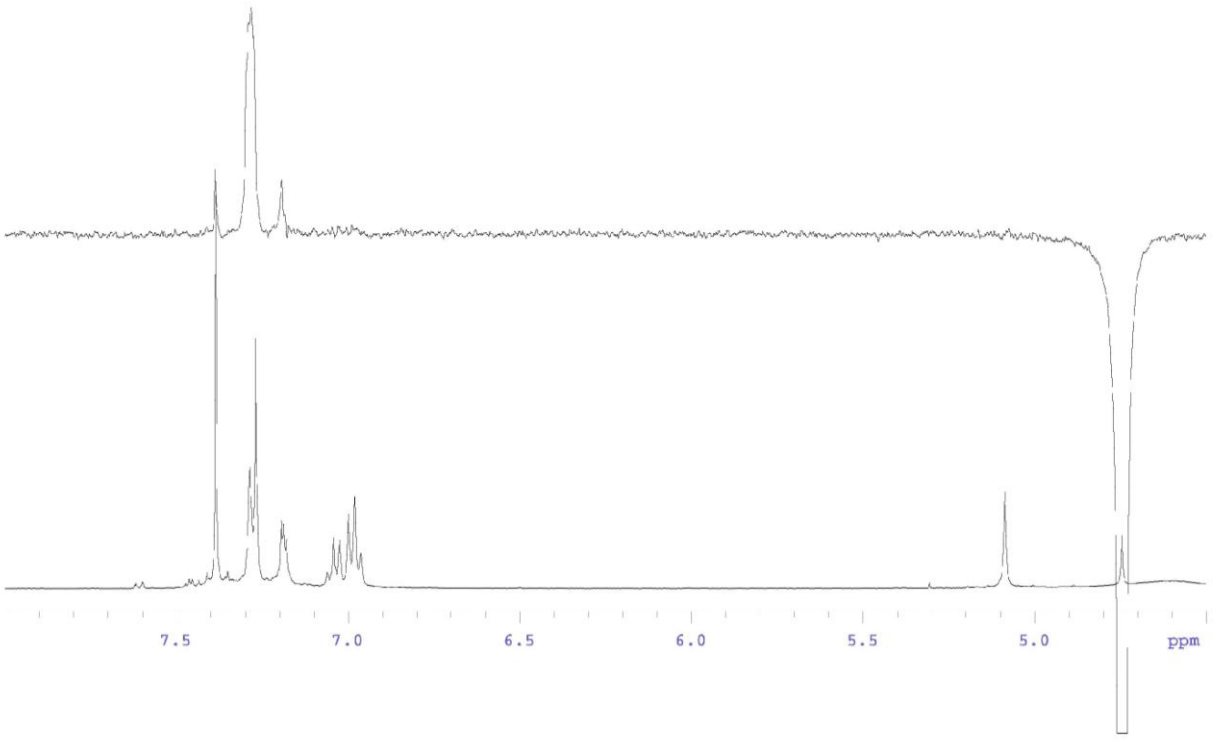

Figure 18. Partially 1D ROESY and ^{1}H NMR spectra of a 1:2 mixture of **1c** with (*S*)-**3a**.

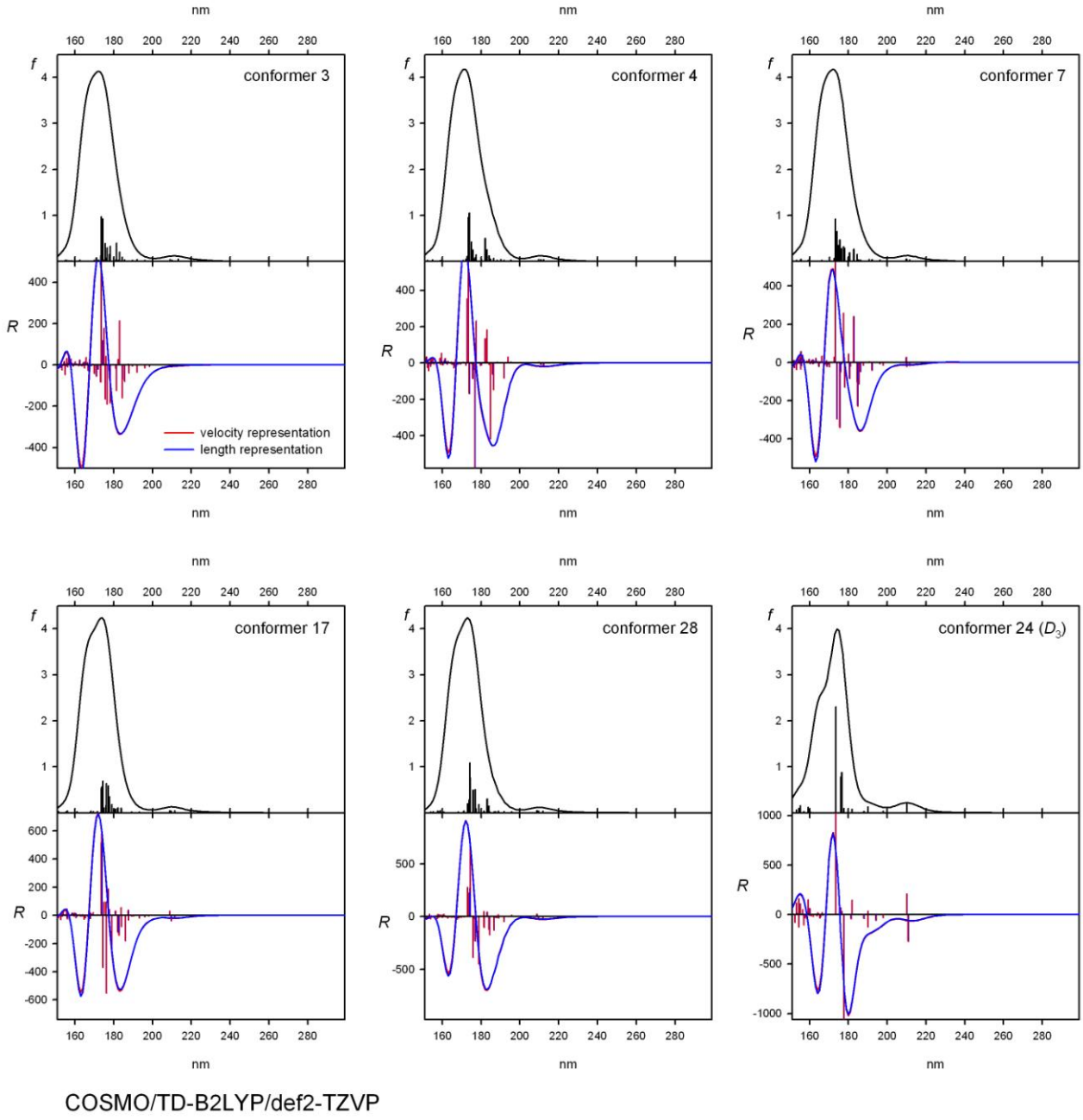

Figure 19. ECD and UV spectra calculated for low-energy conformers of **1b** at the PCM/B2LYP/def2-TZVP level. Wavelength not corrected. Conformers are numbered in order of their appearance during conformational search.

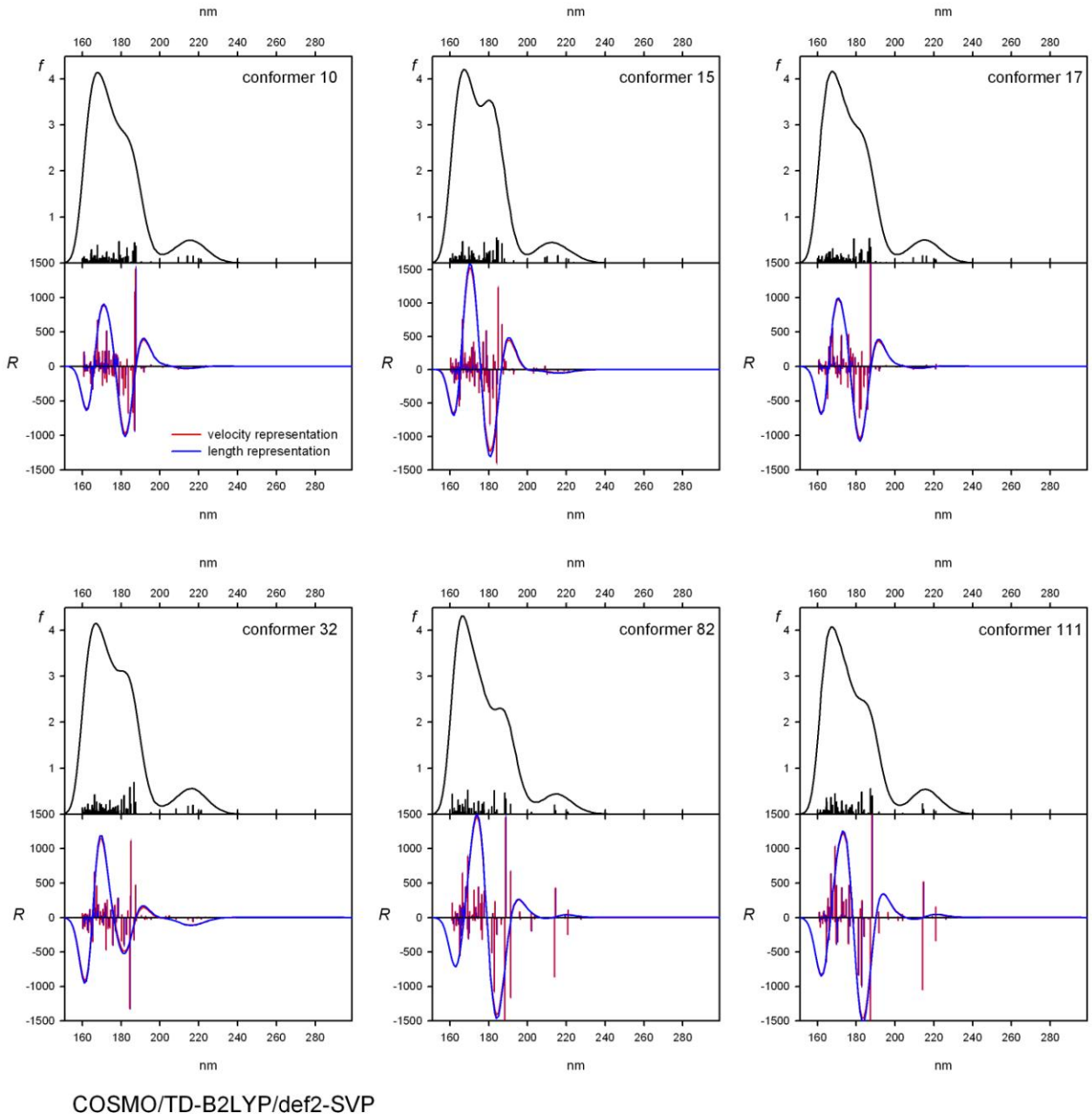

Figure 20. ECD and UV spectra calculated for low-energy conformers of **1c** at the PCM/B2LYP/def2-SVP level. Wavelength not corrected. Conformers are numbered in order of their appearance during conformational search.

Table 1. Relative energies (ΔE, in kcal mol^{-1}), percentage populations and values of torsion angles calculated at the PCM/PBE0/6-31G* level for low-energy conformers of trianglamines **1b**, **1c** and **1c×6H**$^{+}$.

Conformer[a]	ΔE	Population	Sequences of torsion angles $\beta\alpha\alpha'\beta'$
1b (1)	0.00	55	158.8; 163.0; 83.4; 69.4; –178.8; 171.5; 163.8; –171.3; –177.4; 173.0; 158.7; 168.8
1b (4)	1.98	2	172.2; 175.2; 160.3; 170.3; 61.3. 78.6; 176.8; 87.2; 173.5; 163.3; 174.0; 175.7
1b (7)	0.35	30	–177.0; 175.1; 158.1; 168.9; 156.6; 162.4; 82.9; 70.4; –173.0; 164.4; 176.1; –179.3
1b (17)	0.96	11	–172.9; 168.9; 157.0; 170.1; 156.6; 159.1; 79.4; 70.2; –173.2; 166.0; 173.9; 179.1
1b (28)	2.00	2	175.4; 165.6; 168.4; –173.1; 175.8; 173.2; 161.3; 170.8; 61.0; 76.5; 178.0; 81.4
1c (10)	0.00	33	169.7; 176.8; 153.1; 172.1; –179.0; 168.7; 158.2; 169.7; 177.6; 164.3; 167.6; 171.3
1c (15)	0.64	11	178.8; 154.4; 177.5; 170.2; 171.2; 161.5; 165.9; 171.4; 170.9; 175.7; 155.5; 177.6
1c(17)	0.10	29	–178.7; 166.7; 159.4; 168.6; 179.4; 165.6; 163.9; 169.3; 170.2; 178.1; 154.3; 173.7
1c(32)	1.20	4	–179.4; 162.1; 170.4; 173.5; 167.9. 169.2; 168.0; 178.6; 168.8; 175.1; 155.8; 176.8
1c (82)	0.59	12	178.6; 165.7; 160.0; 170.6;

			178.0; 165.0; 161.0; 171.0;
			178.0; 165.6; 160.2; 170.7
1c (111)	0.63	11	178.3; 165.7; 160.1; 170.6;
			178.0; 165.1; 160.0; 170.8;
			178.1; 165.2; 160.0; 170.9
1c×6H$^+$	-	~100	179.7; 138.0. 134.6; –177.3;
			179.7; 138.0. 134.6; –177.3;
			179.7; 138.0. 134.6; –177.3

[a] Conformers are numbered in order of their appearance during conformational search.