BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Complexation of chiral amines by resorcin[4]arene sulfonic acids in polar media – circular dichroism and diffusion studies of chirality transfer and solvent dependence

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Copies of NMR, UV, and CD spectra

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1.1. NMR spectra of cavitands

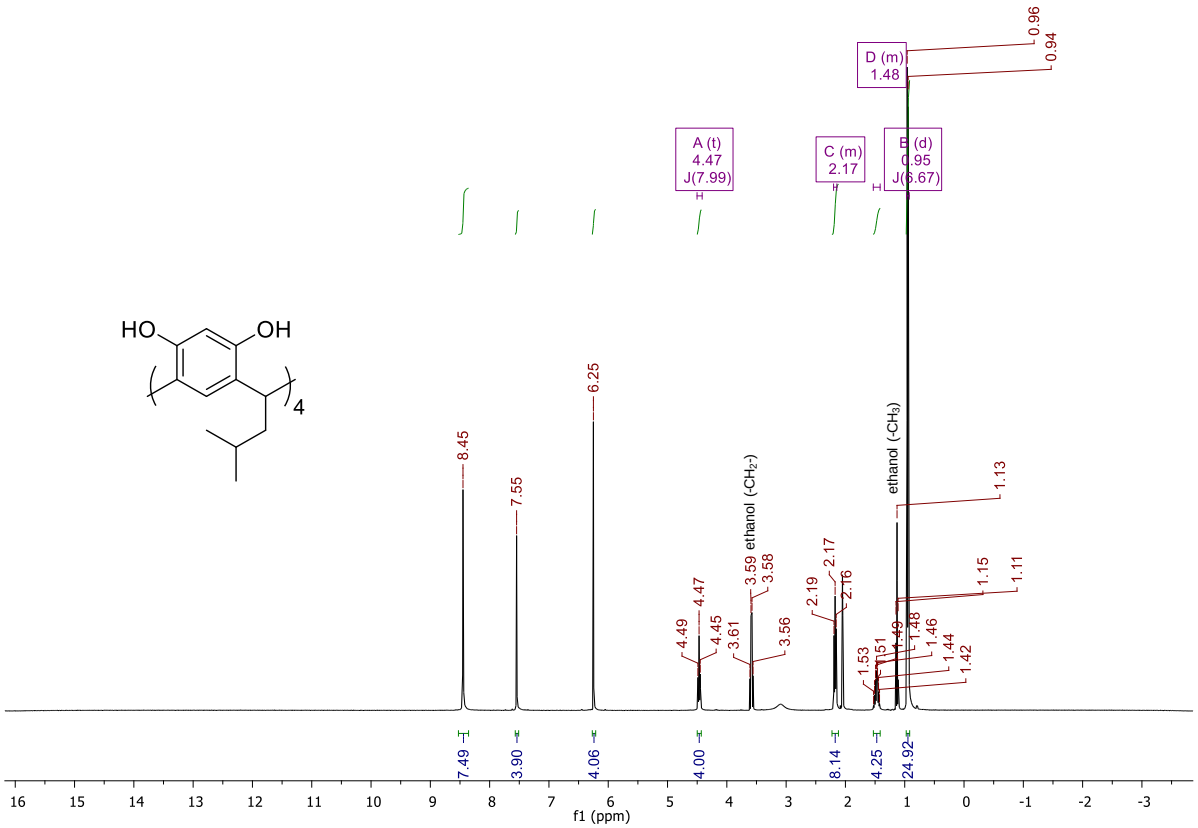

Figure S1. ^{1}H NMR (400 MHz, acetone-d_6) spectrum of 2,8,14,20-tetraisobutyl-resorcin[4]arene.

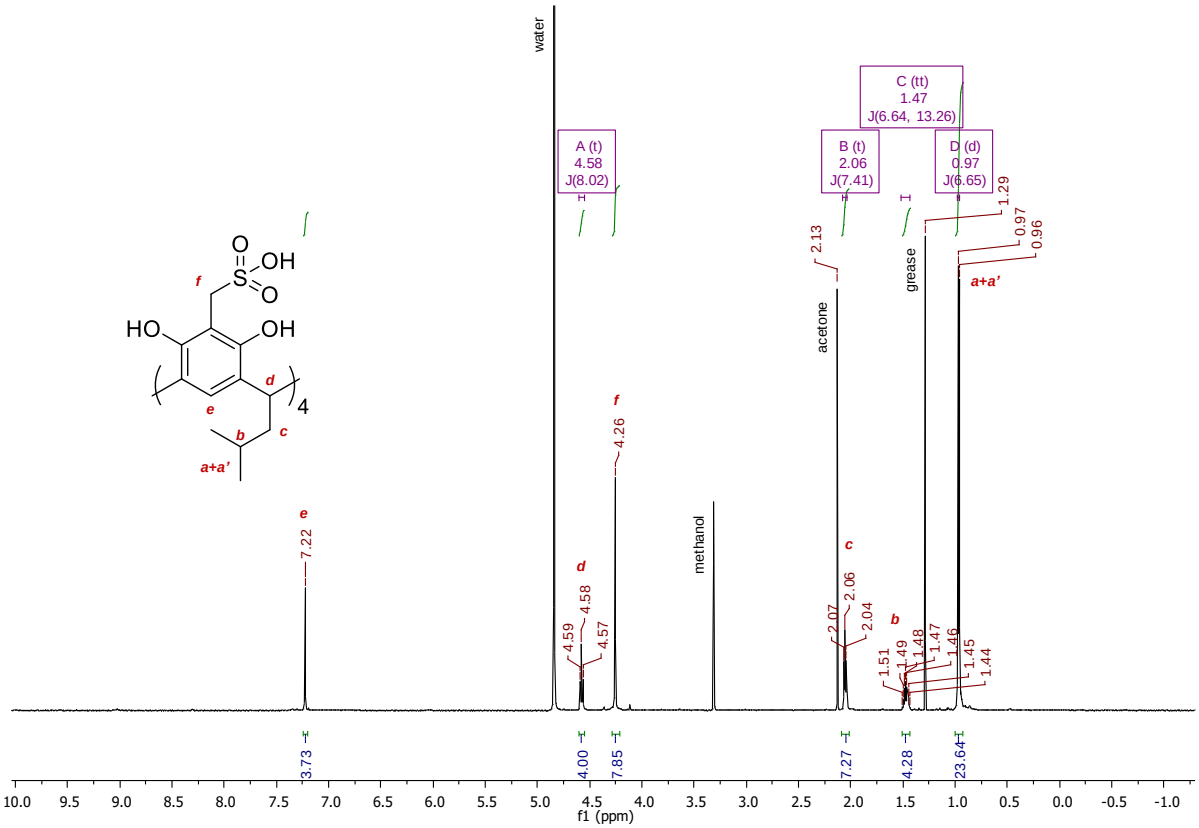

Figure S2. ^{1}H NMR (600 MHz, methanol-d_4) spectrum of **1**.

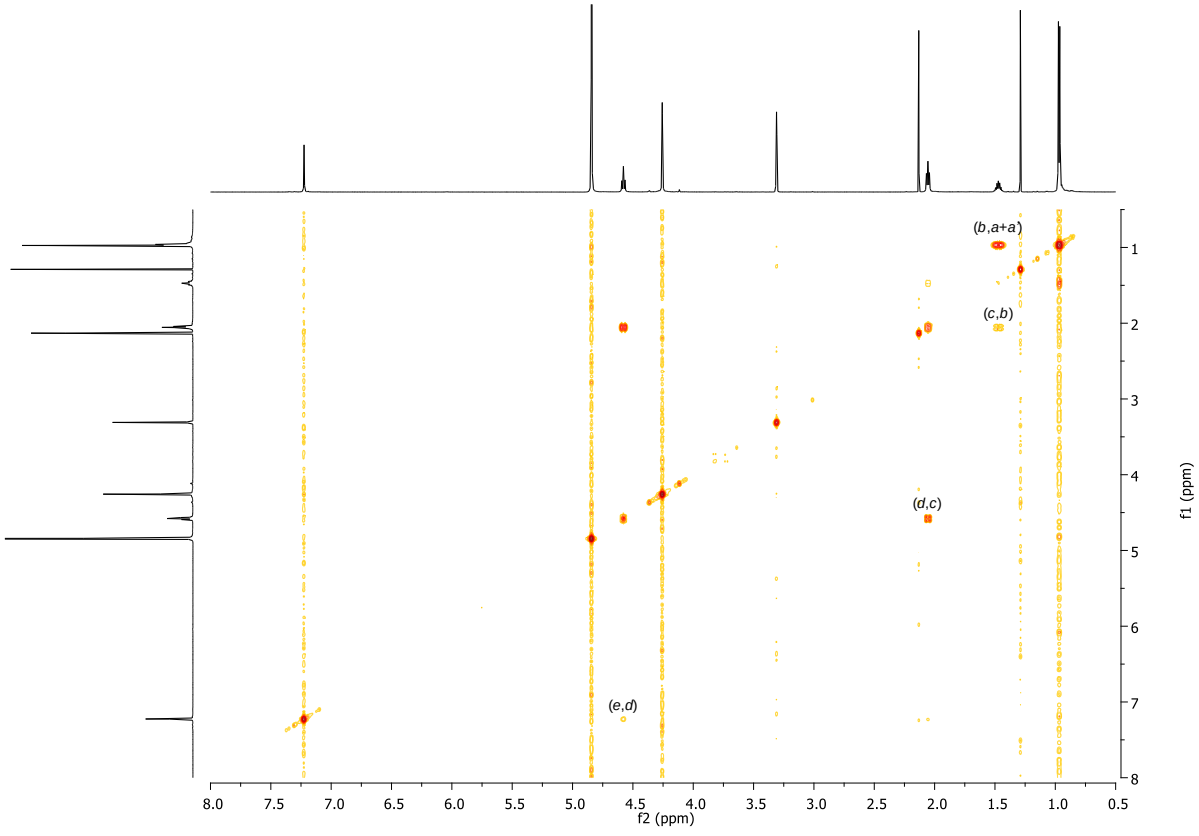

Figure S3. COSY NMR (600 MHz, methanol-d_4) spectrum of **1**.

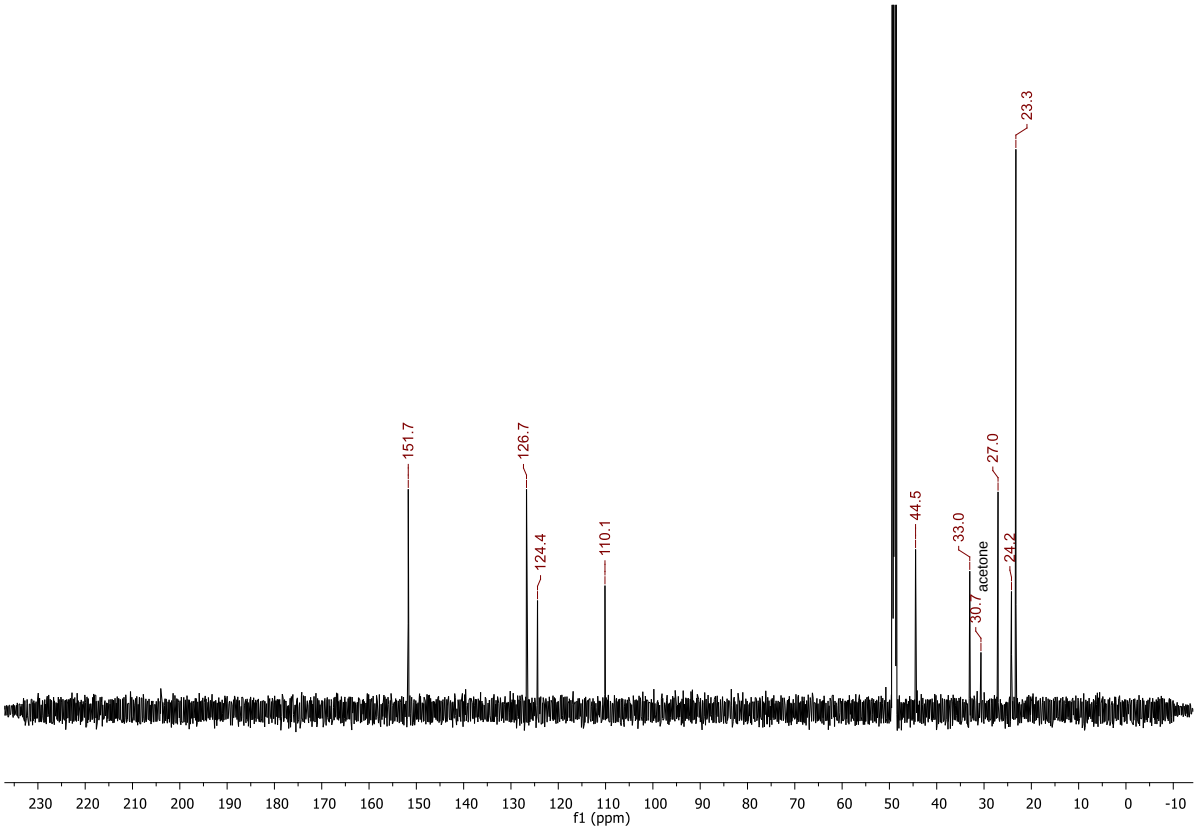

Figure S4. ^{13}C NMR (150 MHz, methanol-d_4) spectrum of **1**.

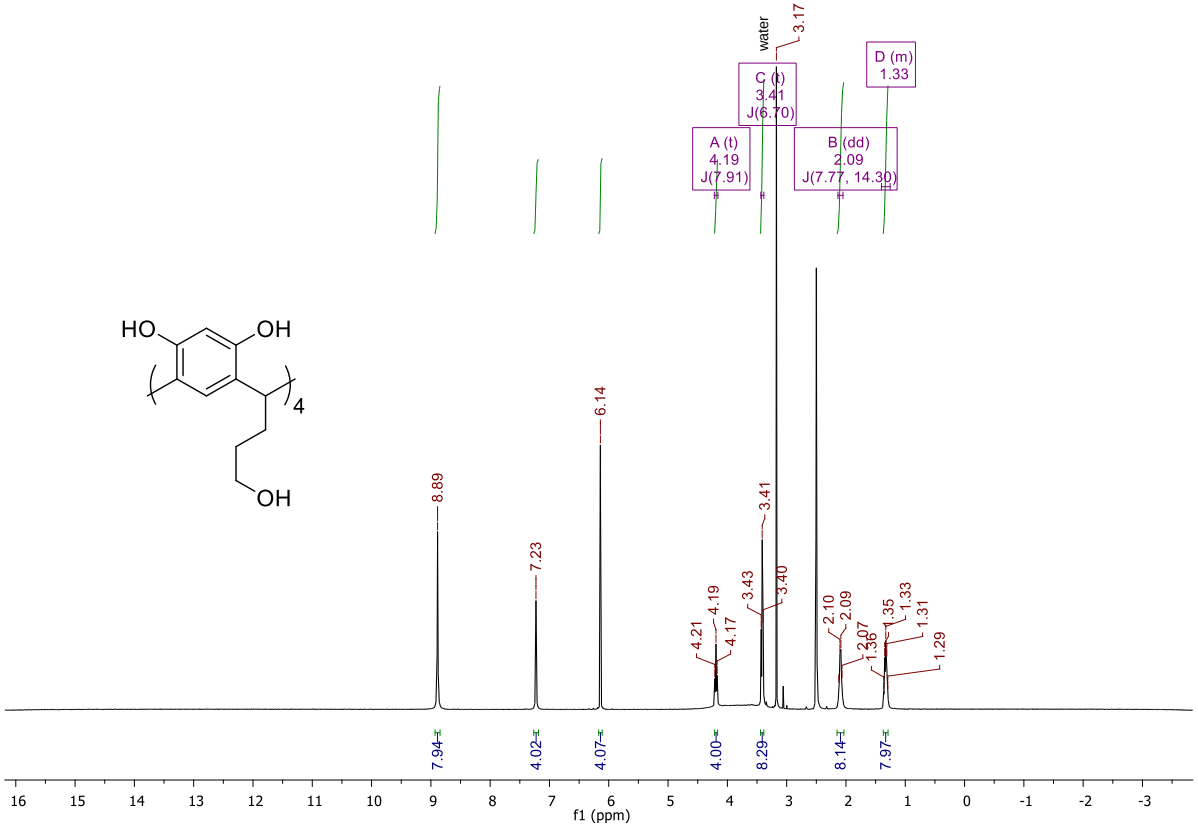

Figure S5. ^{1}H NMR (400 MHz, dimethyl sulfoxide-d_6) spectrum of C-(3-hydroxypropyl)-resorcin[4]arene.

1.2. NMR spectra of complexes of tetrakis(sulfonatomethyl)resorcin[4]arene **1** with amino acids esters

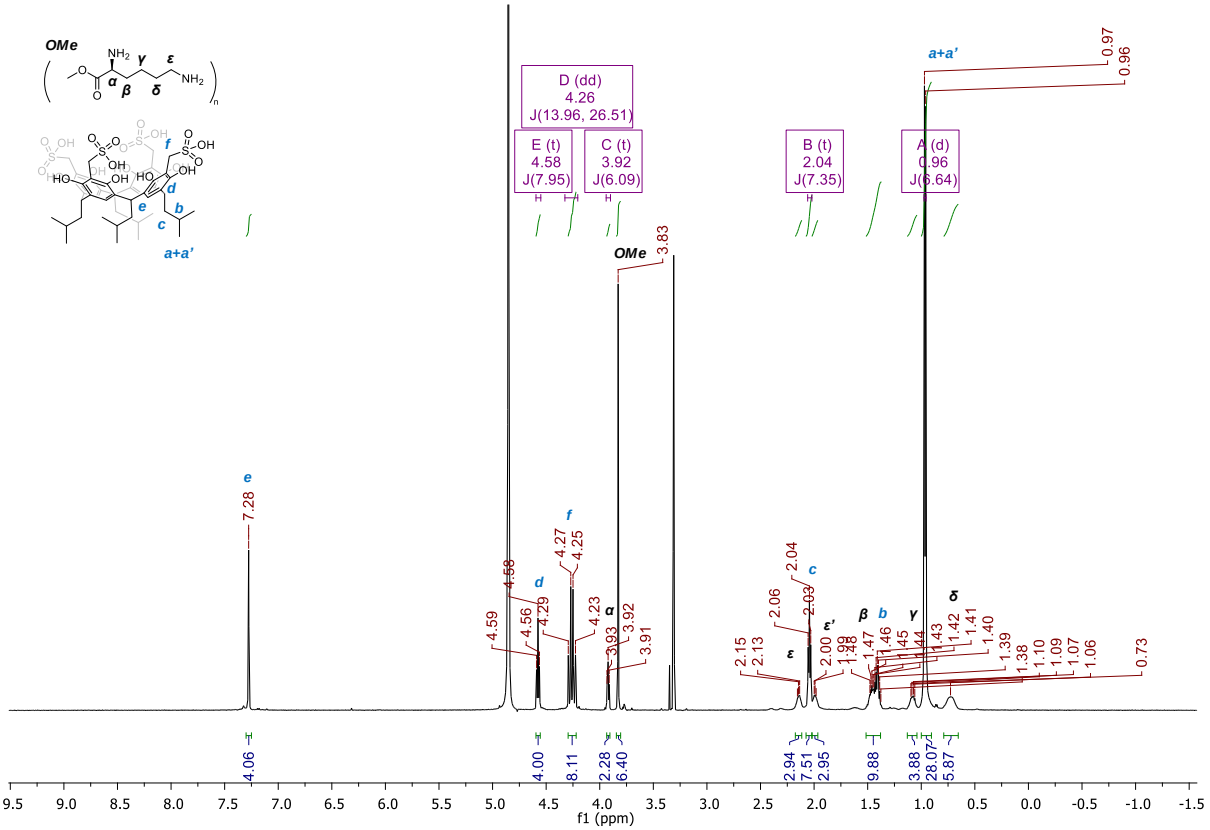

Figure S6. ^{1}H NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-lysine methyl ester.

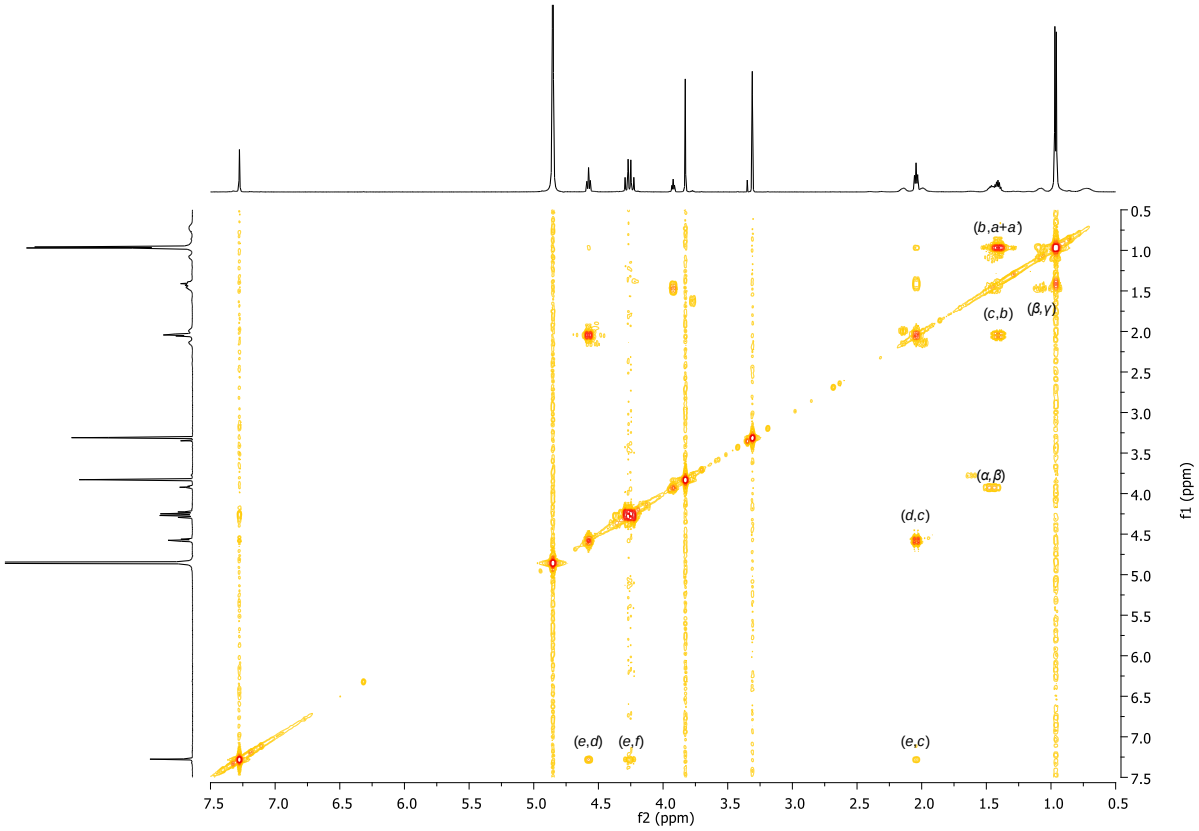

Figure S7. COSY NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-lysine methyl ester.

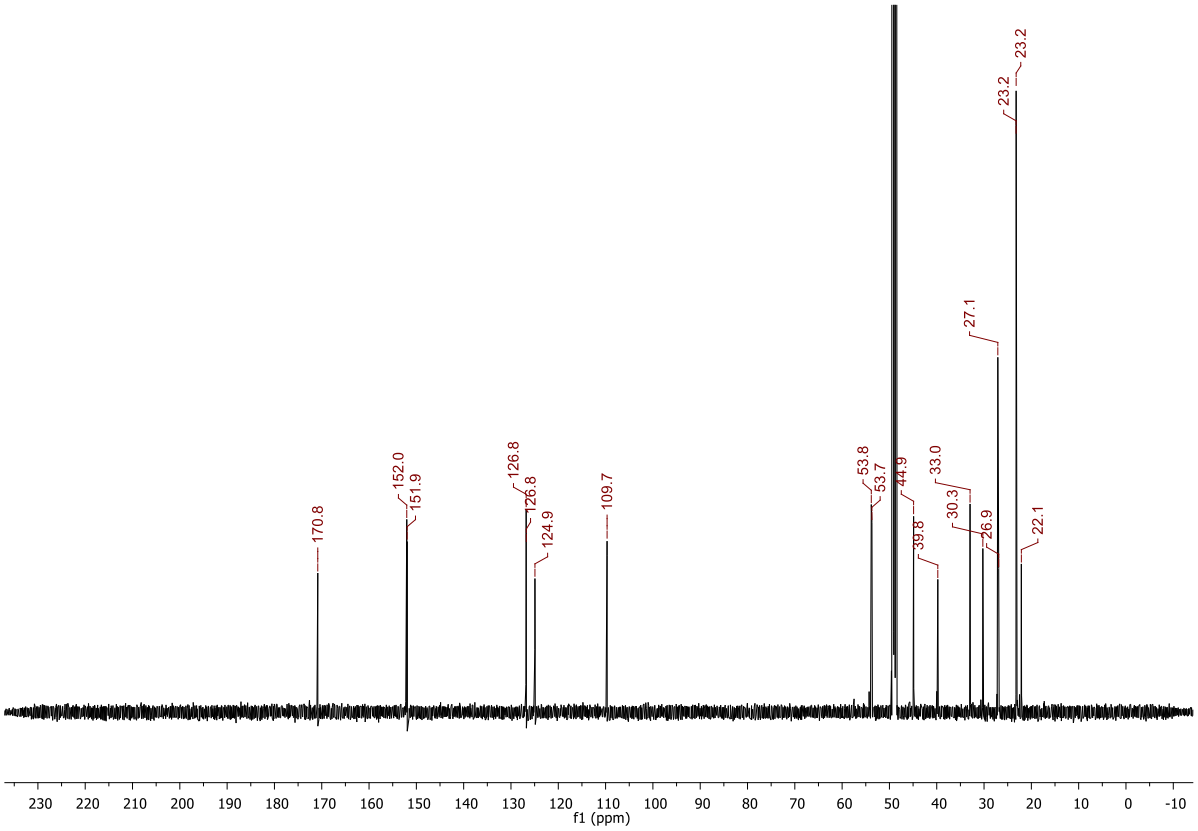

Figure S8. ^{13}C NMR (150 MHz, methanol-d_4) spectrum of complex of **1** with L-lysine methyl ester.

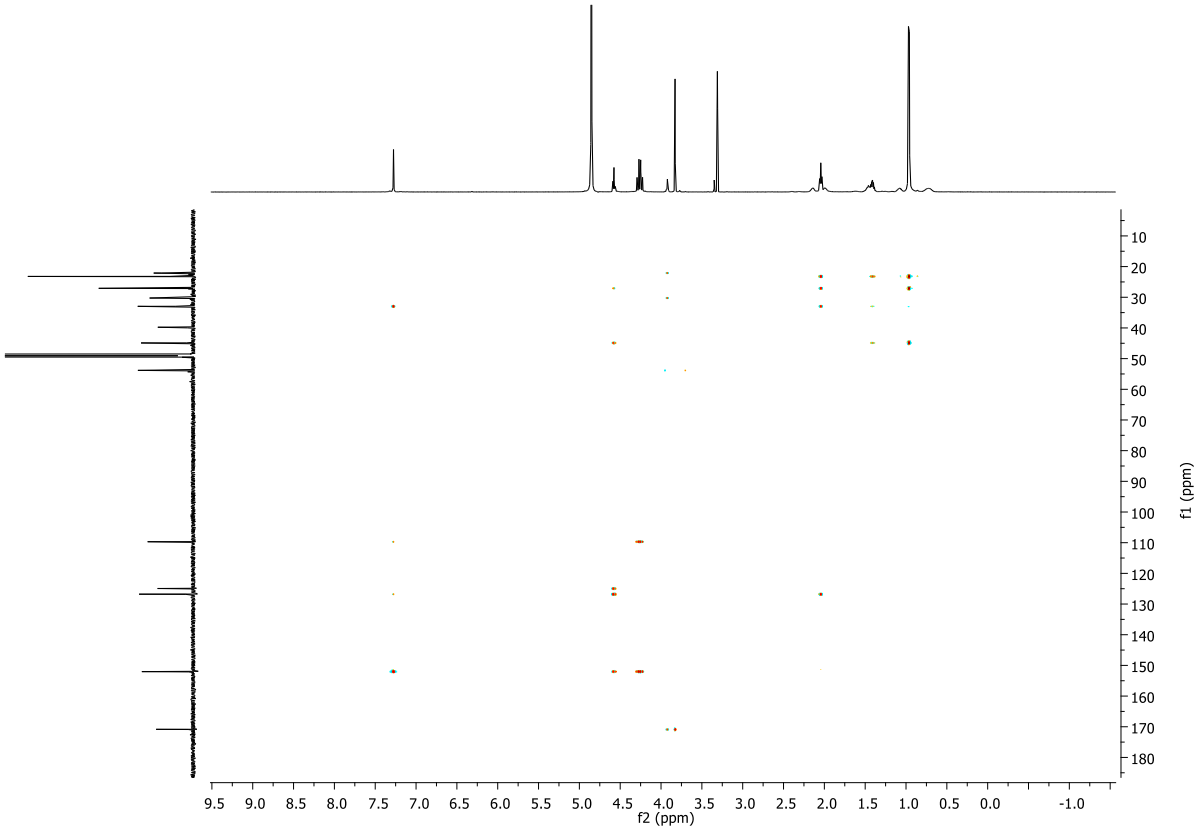

Figure S9. HMBC NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-lysine methyl ester.

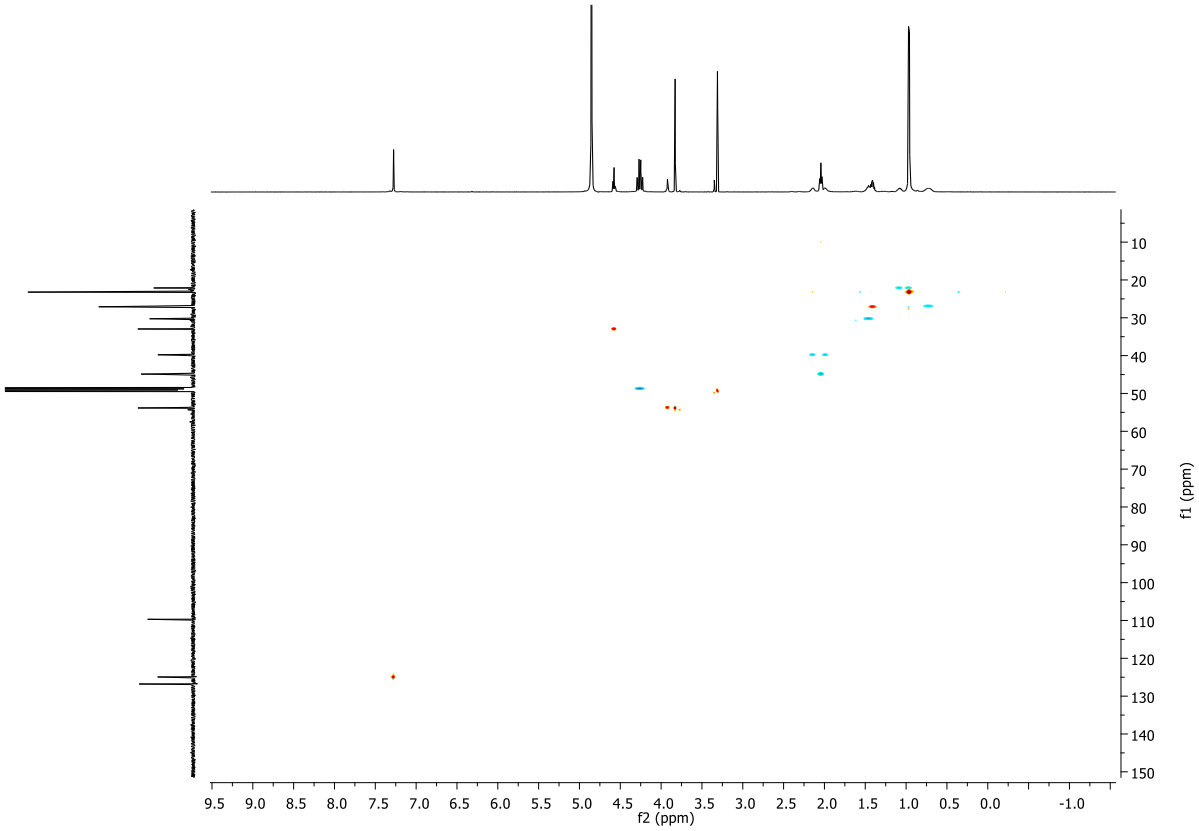

Figure S10. HSQC NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-lysine methyl ester.

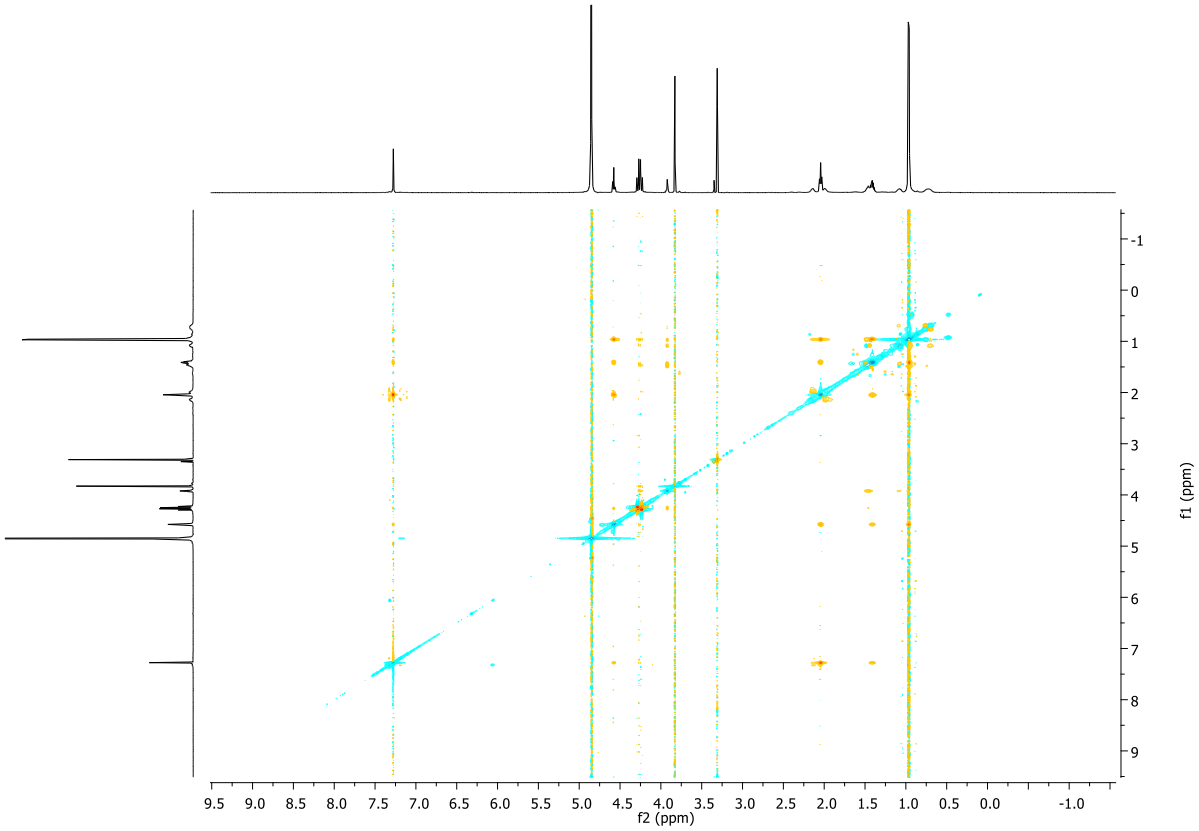

Figure S11. ROESY NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-lysine methyl ester.

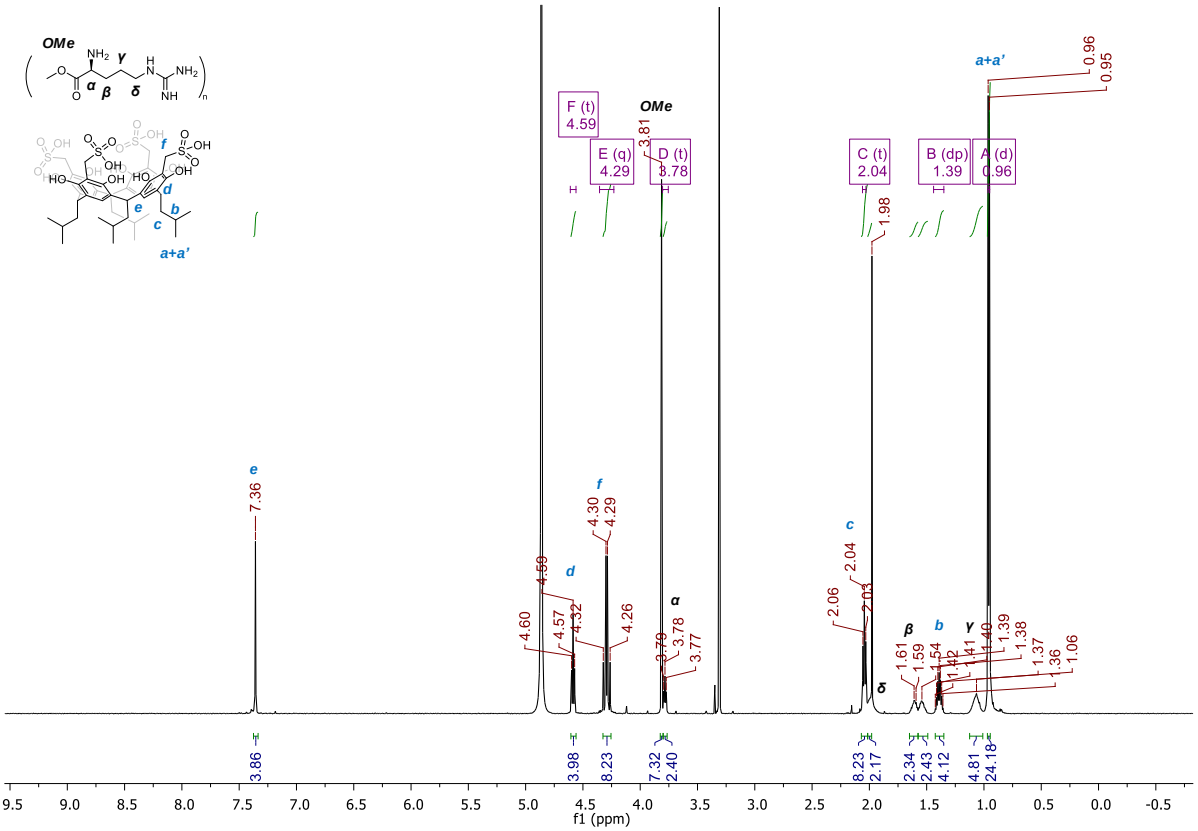

Figure S12. ^{1}H NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-arginine methyl ester.

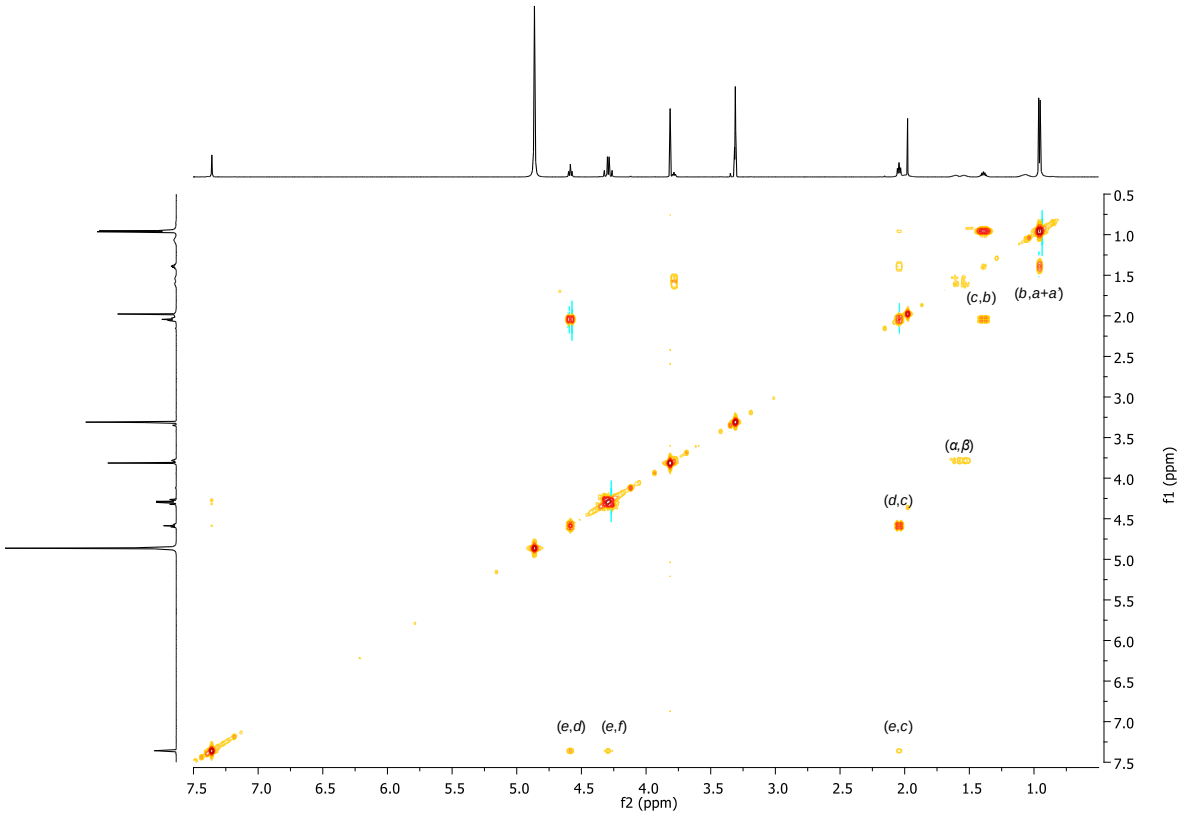

Figure S13. COSY NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-arginine methyl ester.

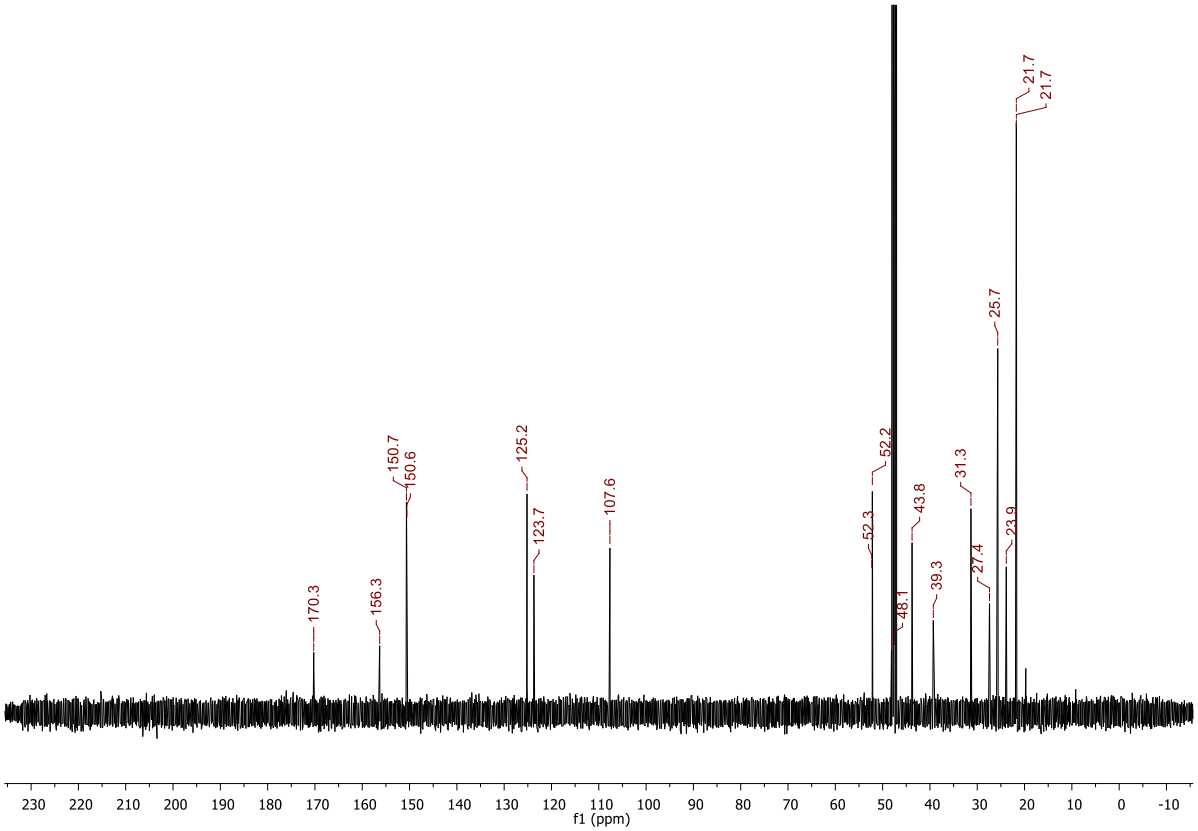

Figure S14. ^{13}C NMR (150 MHz, methanol-d_4) spectrum of complex of **1** with L-arginine methyl ester.

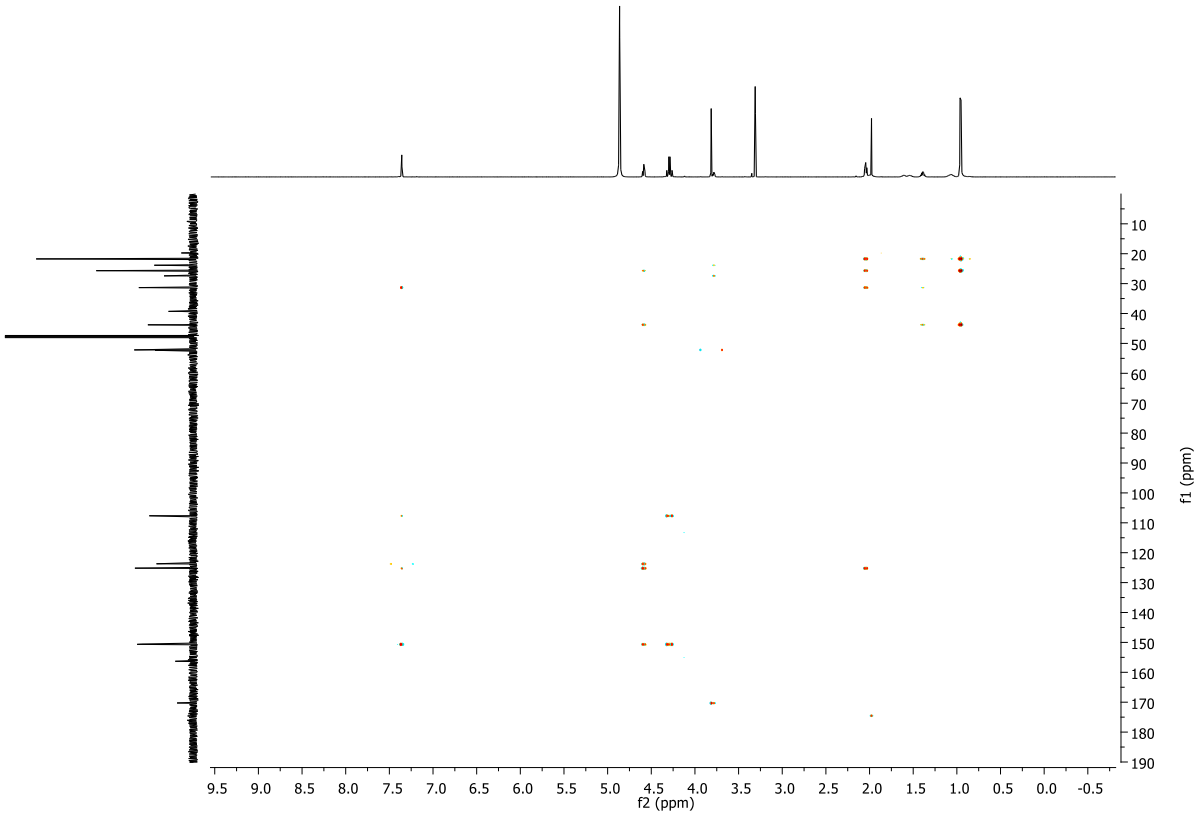

Figure S15. HMBC NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-arginine methyl ester.

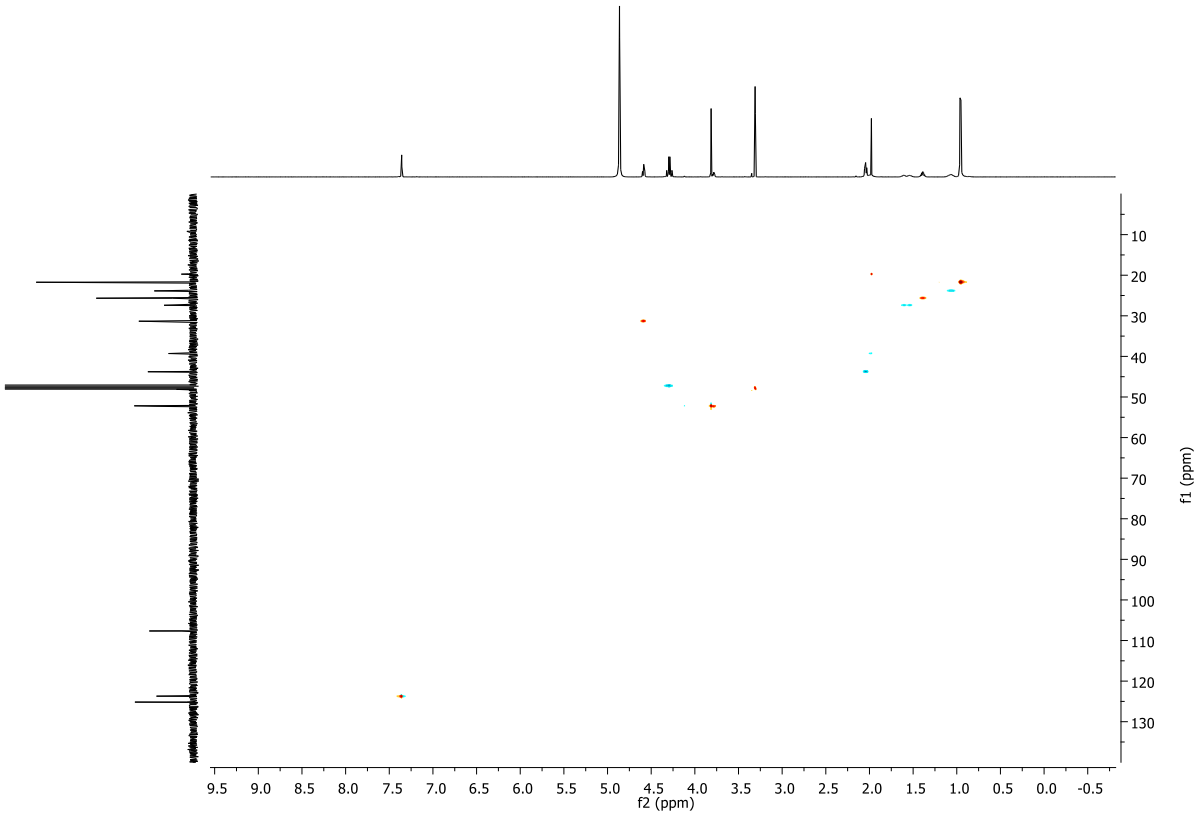

Figure S16. HSQC NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-arginine methyl ester.

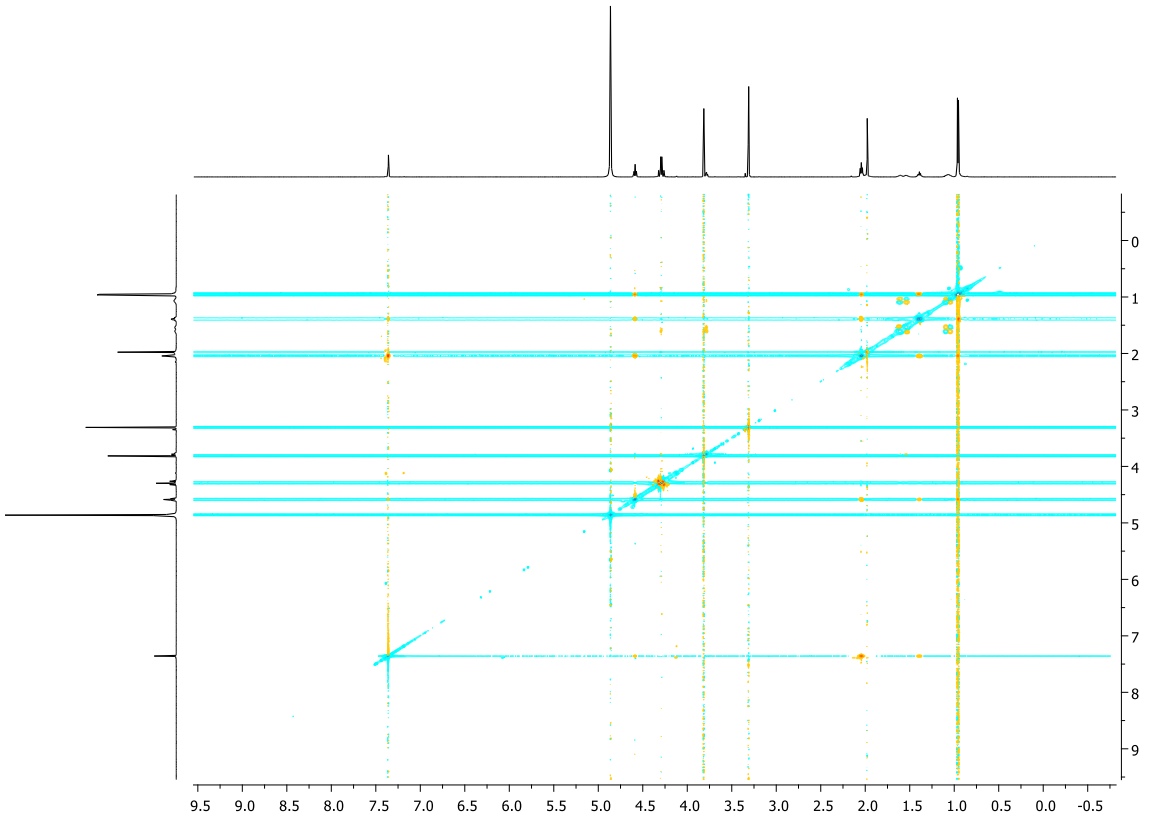

Figure S17. ROESY NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-arginine methyl ester.

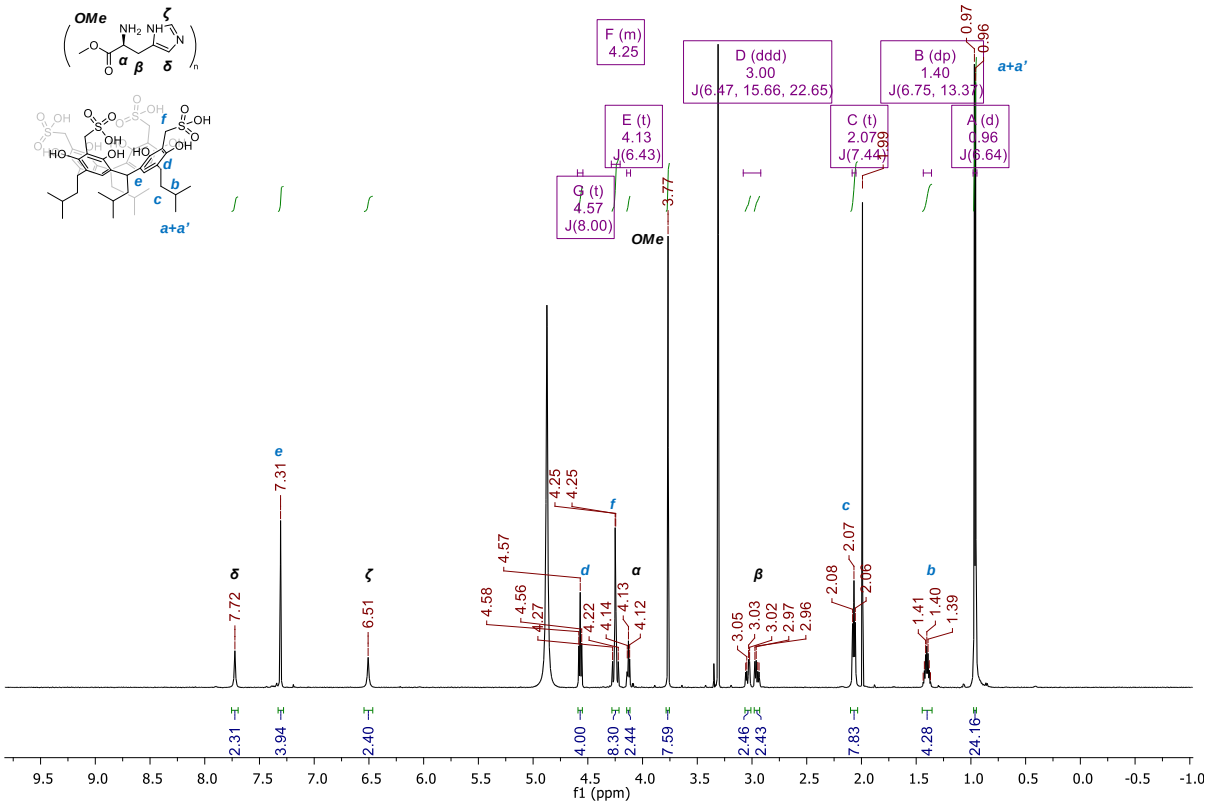

Figure S18. ^{1}H NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-histidine methyl ester.

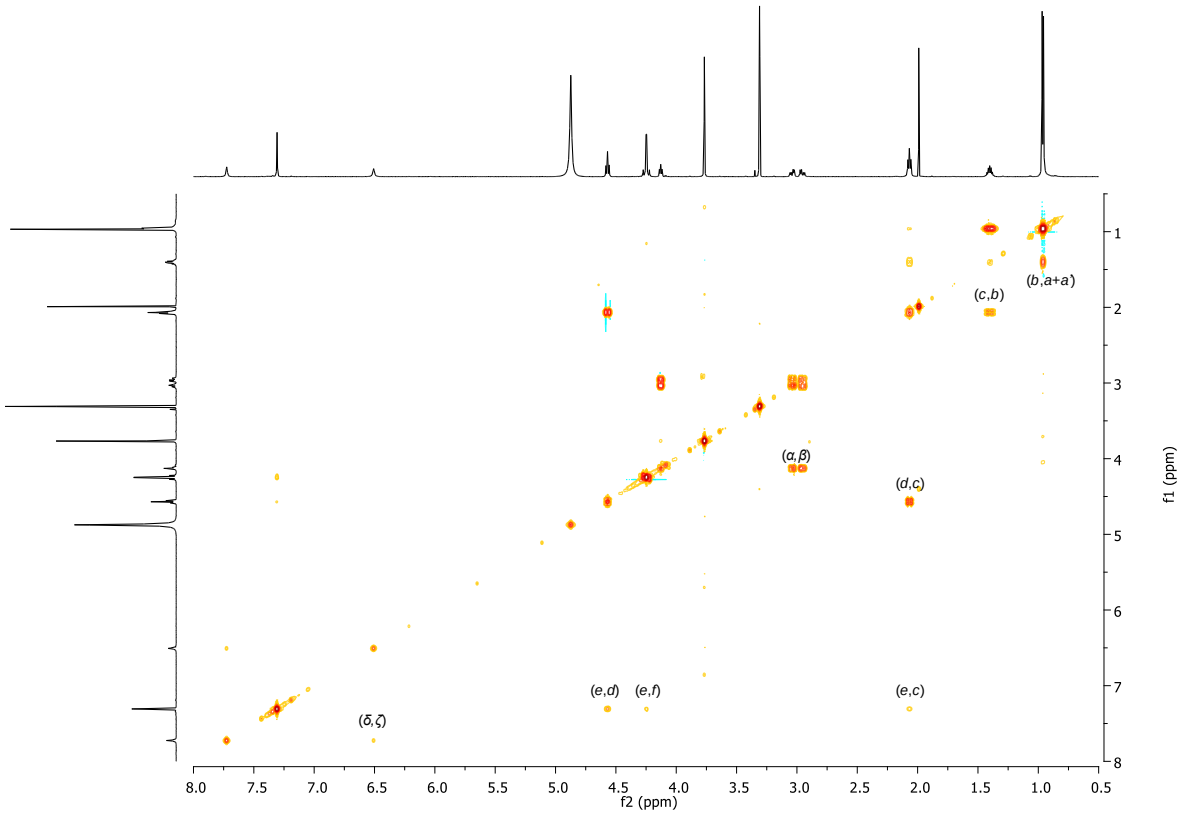

Figure S19. COSY NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-histidine methyl ester.

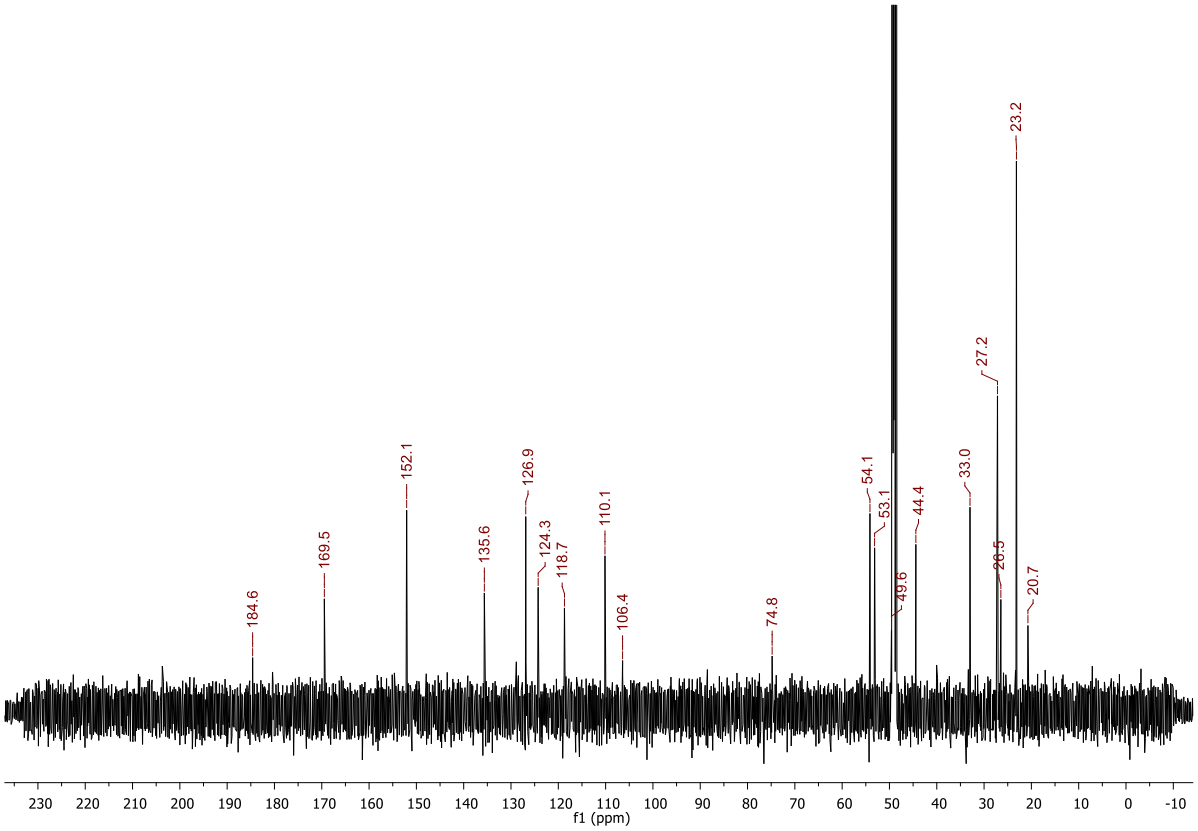

Figure S20. ^{13}C NMR (150 MHz, methanol-d_4) spectrum of complex of **1** with L-histidine methyl ester.

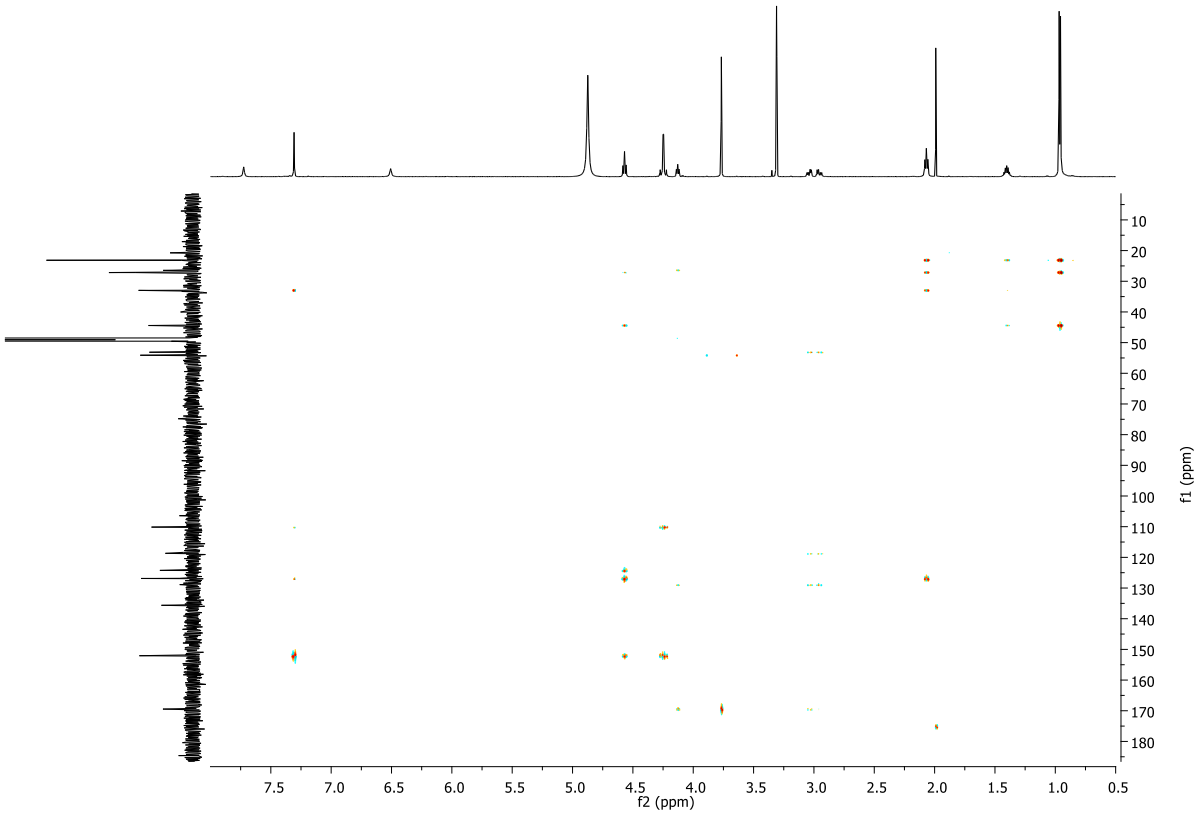

Figure S21. HMBC NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-histidine methyl ester.

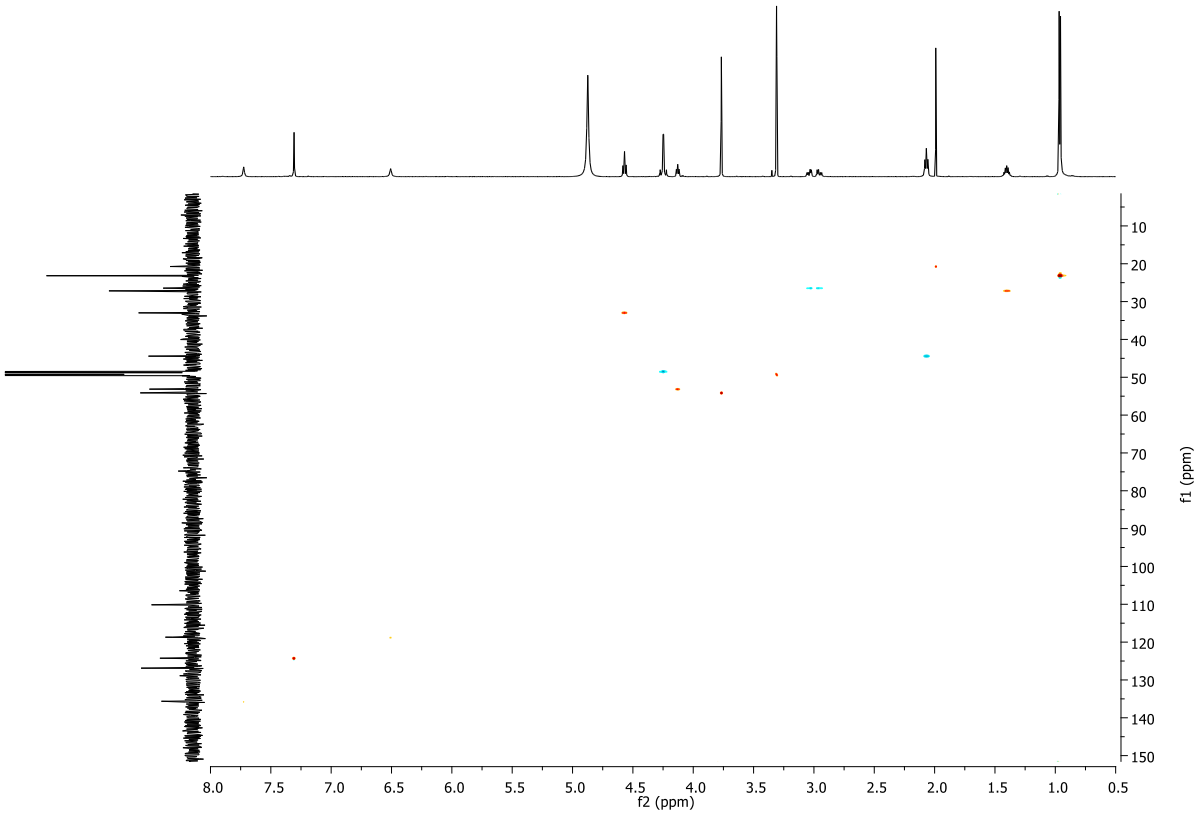

Figure S22. HSQC NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-histidine methyl ester.

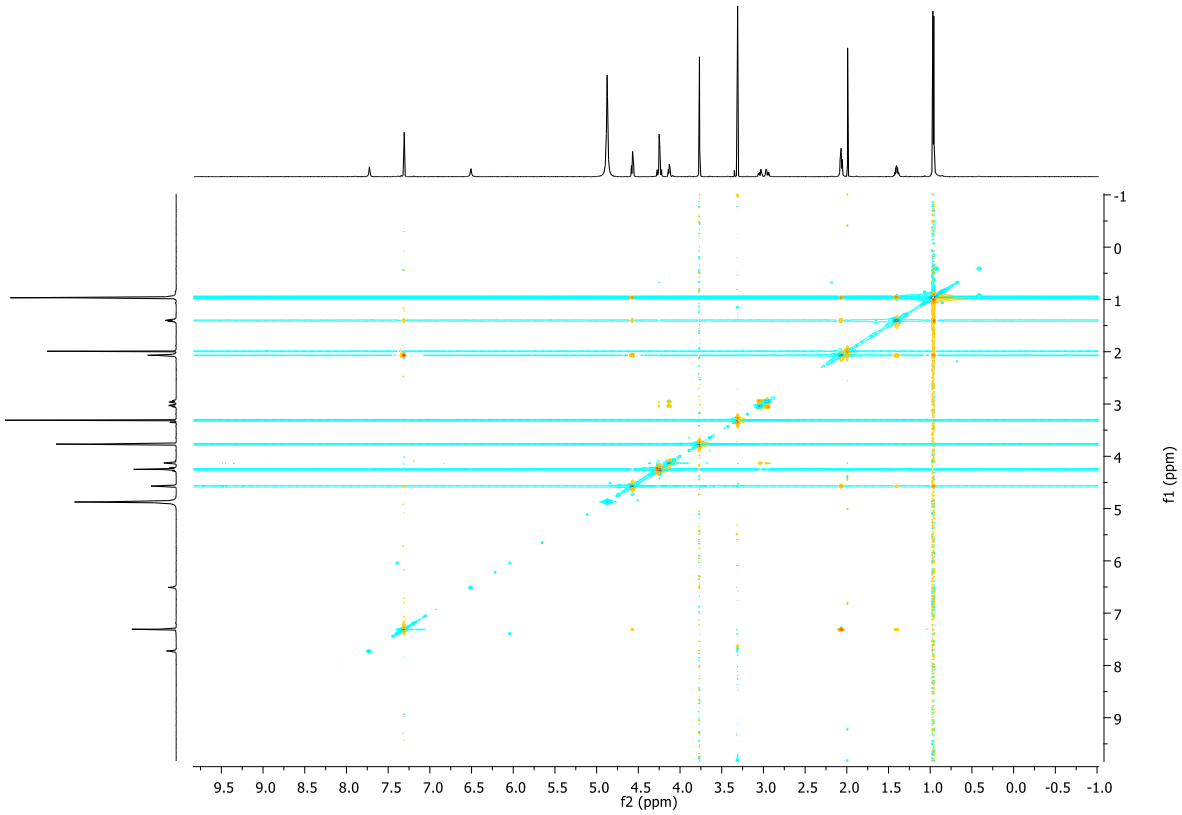

Figure S23. ROESY NMR (600 MHz, methanol-d_4) spectrum of complex of **1** with L-histidine methyl ester.

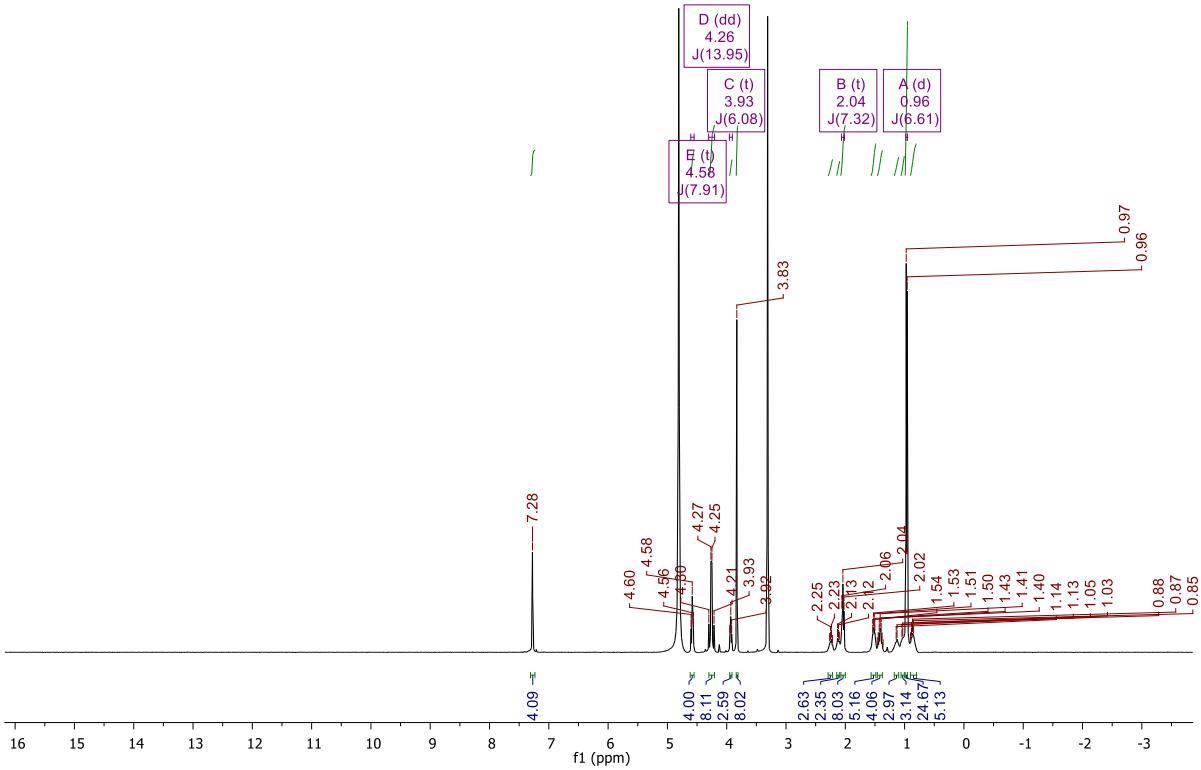

Figure S24. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of complex of **1** with D-lysine methyl ester.

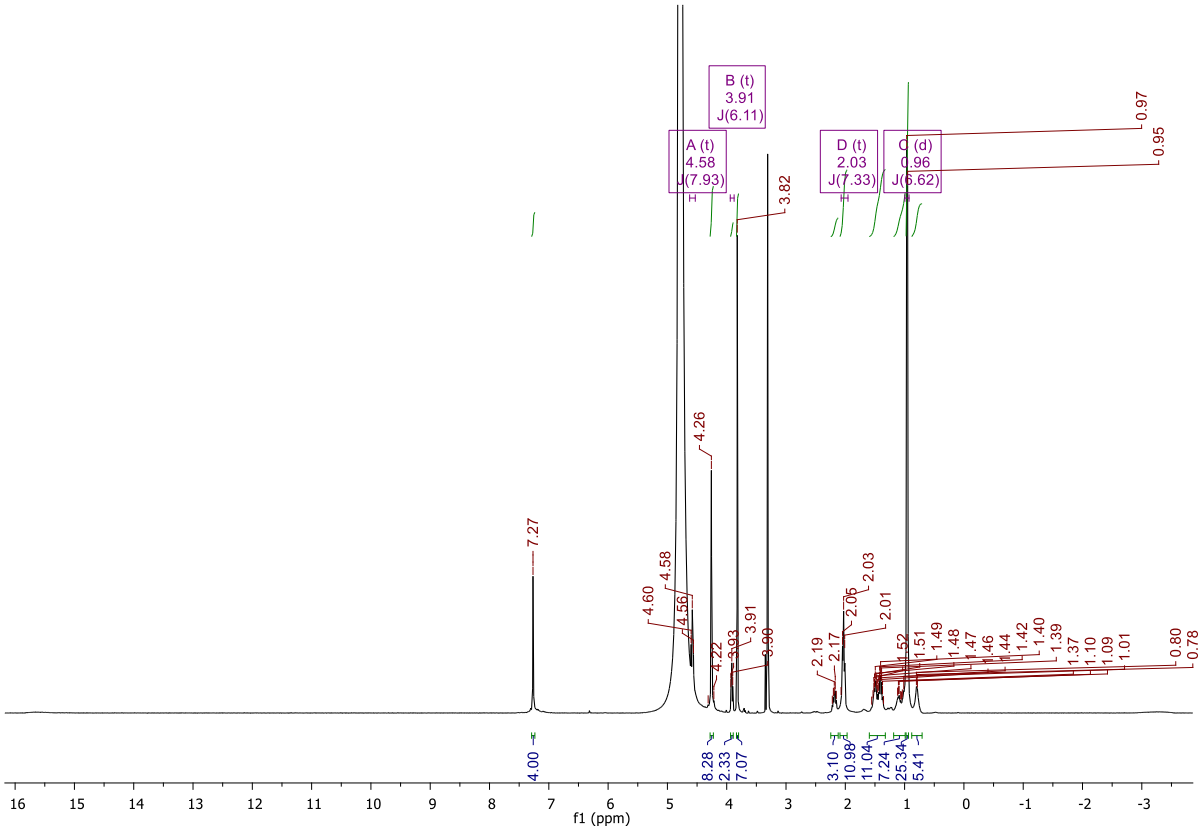

Figure S25. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of complex of **1** with (*rac*)-lysine methyl ester.

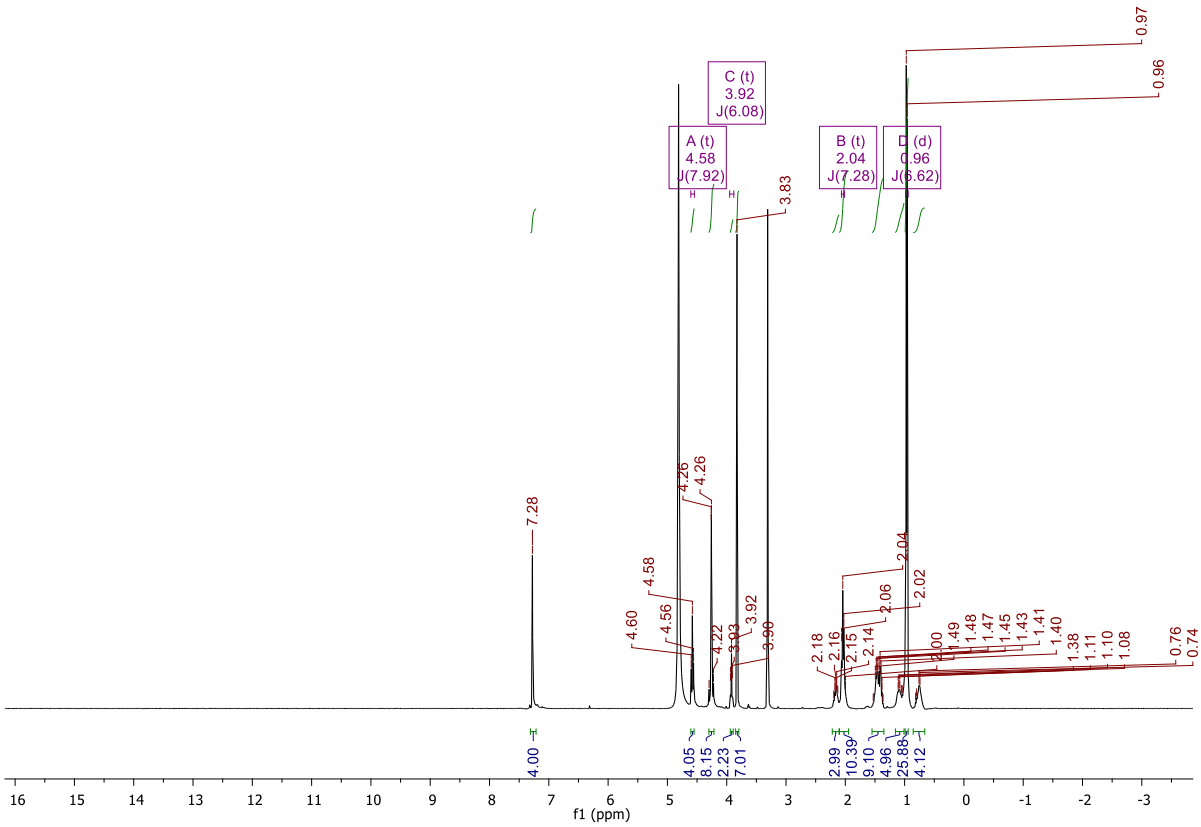

Figure S26. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of complex of **1** with L- and (*rac*)-lysine methyl ester.

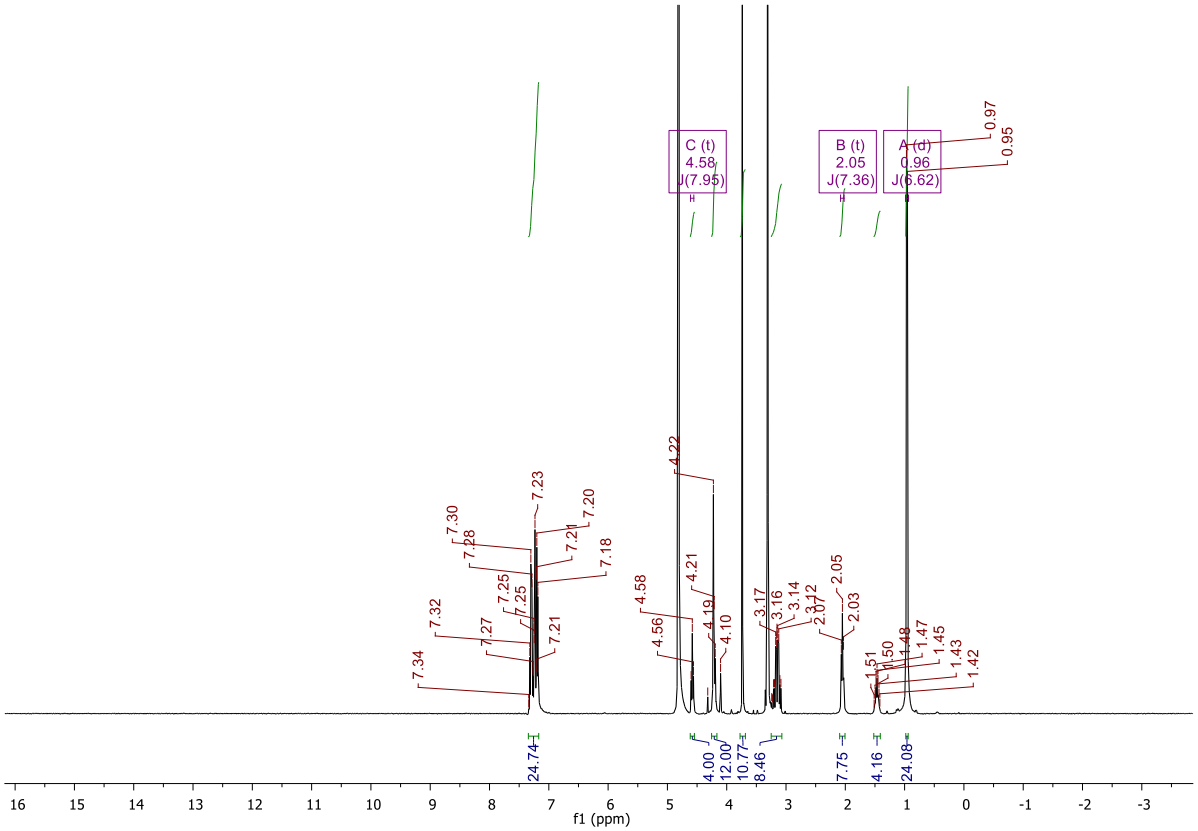

Figure S27. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of complex of **1** with L-phenylalanine methyl ester.

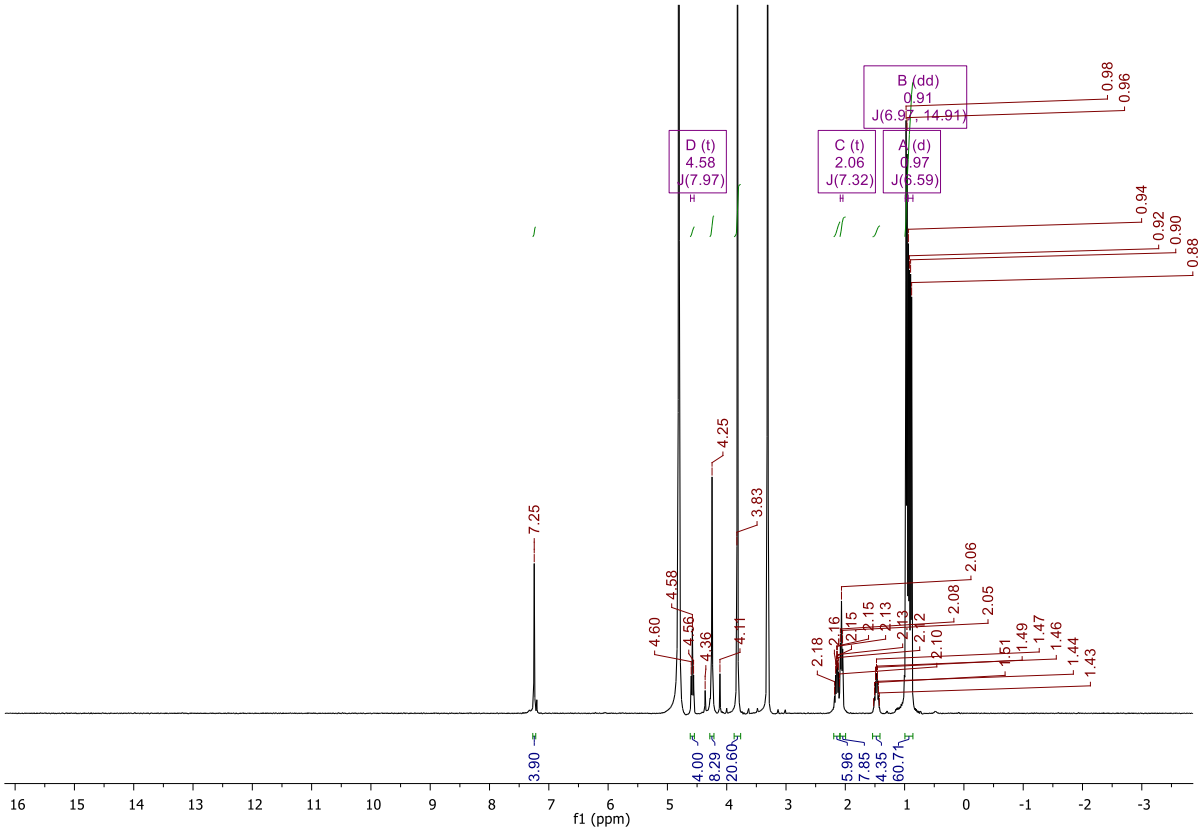

Figure S28. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of complex of **1** with L-valine methyl ester.

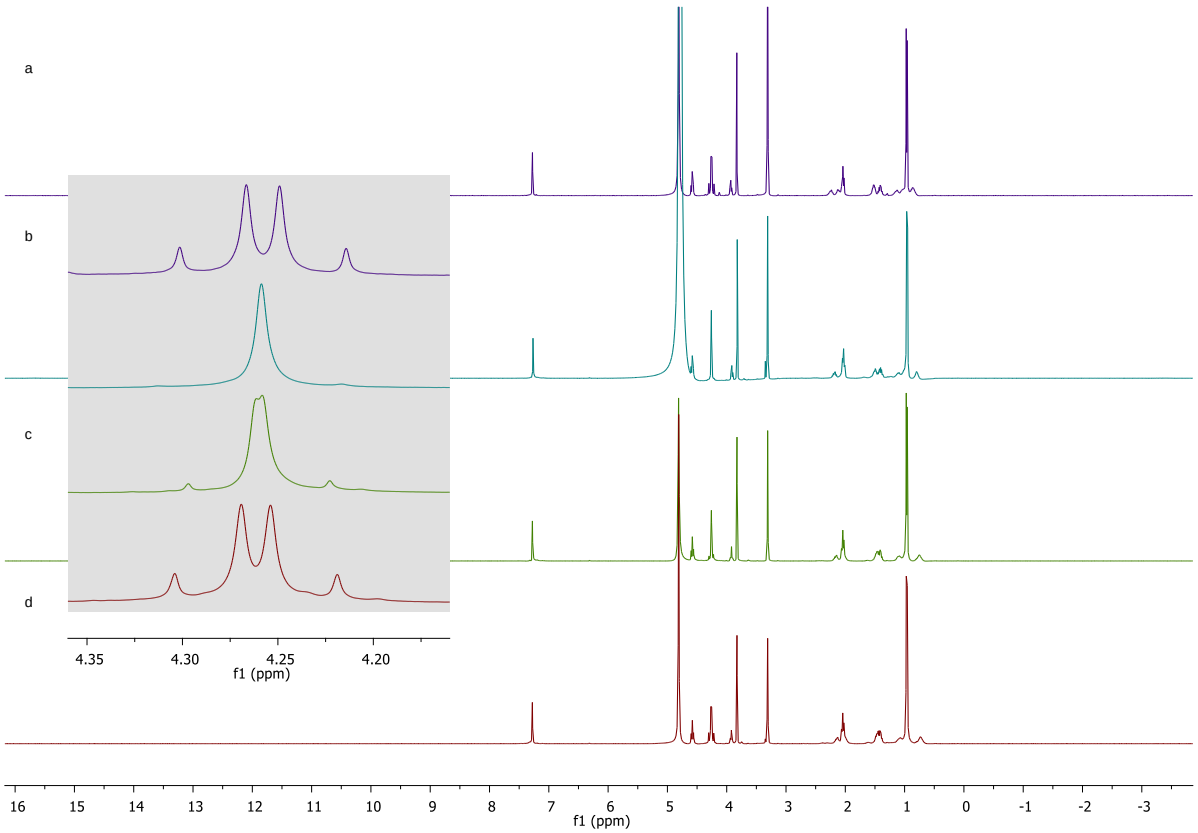

Figure S29. ^{1}H NMR (400 MHz, methanol-d_4) spectra of complexes of **1** with (a) L–lysine methyl ester; (b) (*rac*)-lysine methyl ester; (c) mixture of L- and (*rac*)-lysine methyl ester and (d) D–lysine methyl ester. Inset (grey) depict *f* protons of **1**.

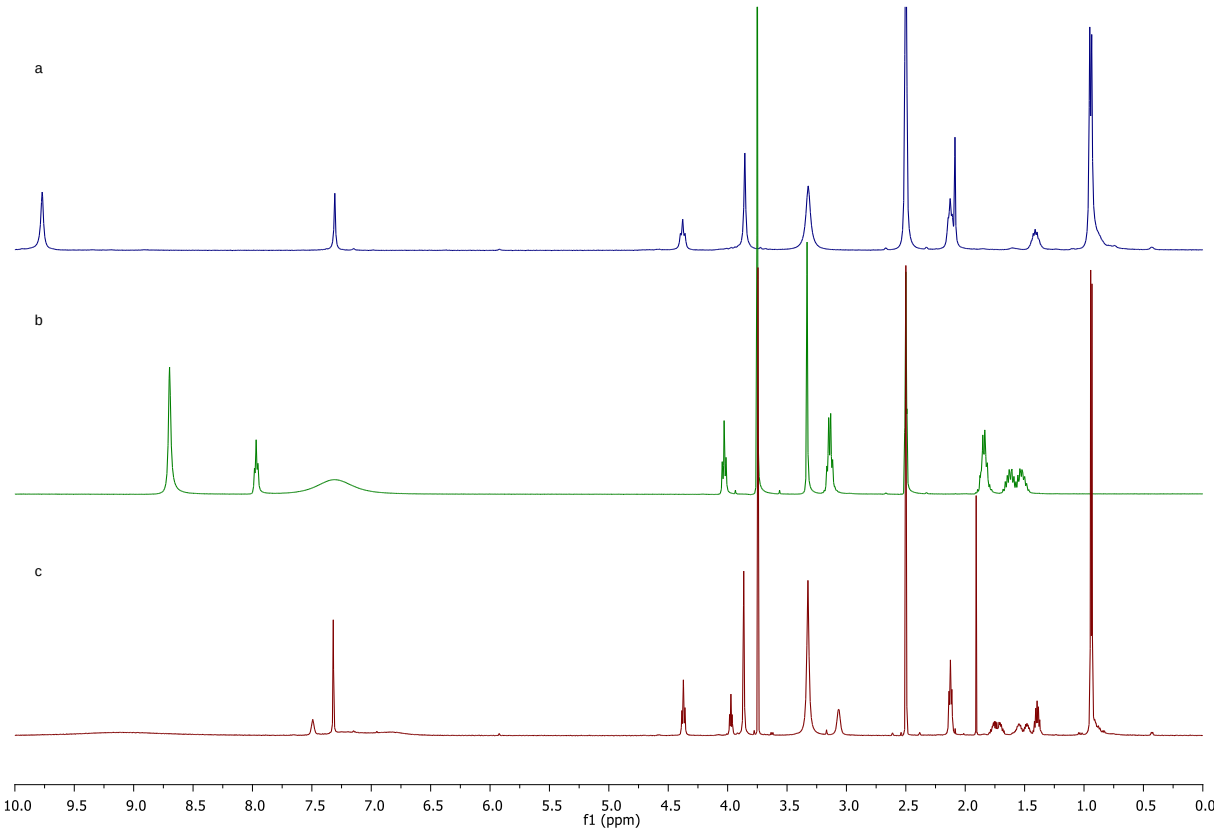

Figure S30. ^{1}H NMR (400 MHz and 600MHz, DMSO-d_6) spectra of (a) **1**; (b) L-arginine methyl ester; (c) **1**(ArgOMe)$_2$.

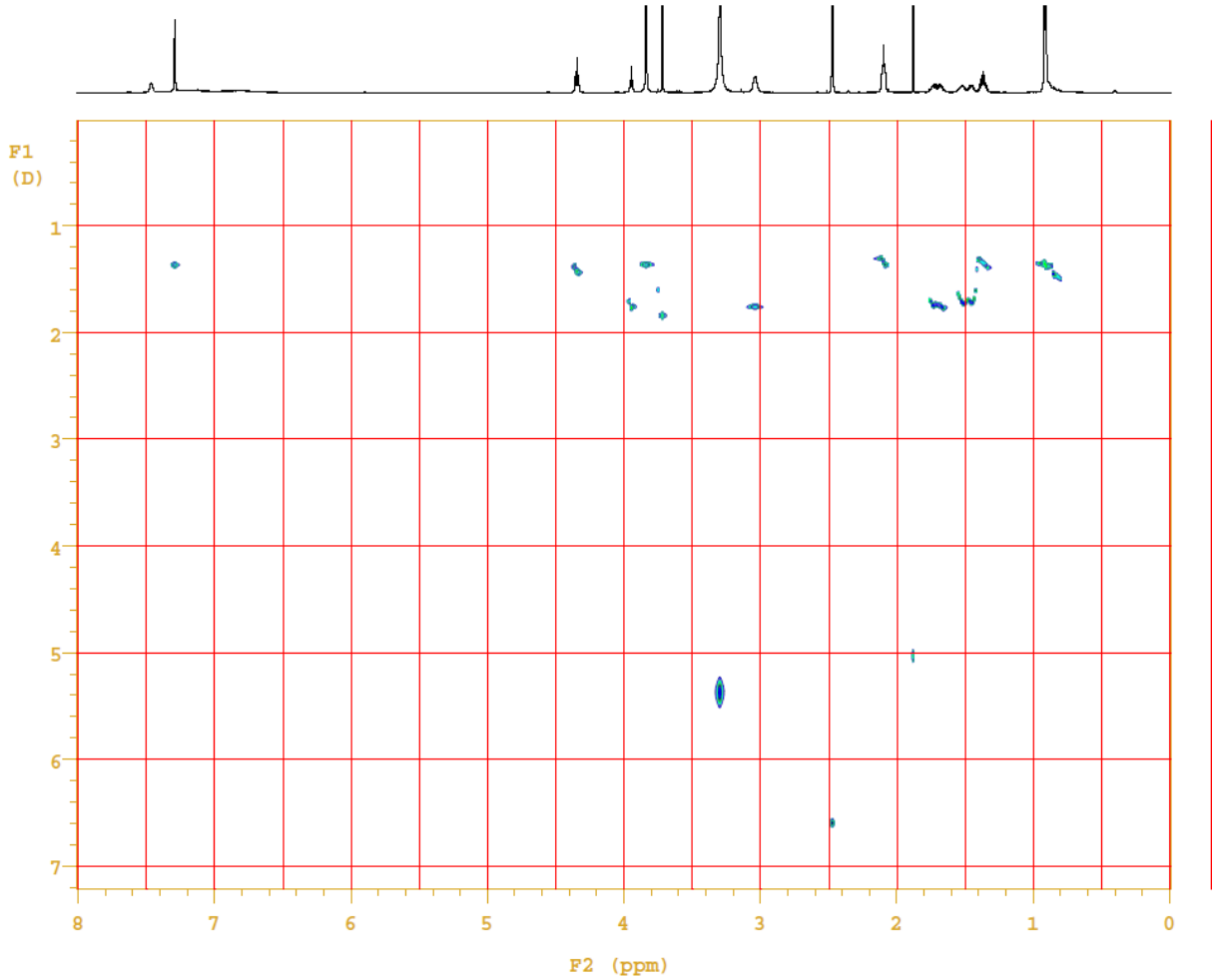

Figure S31. DOSY NMR spectrum of **1**(ArgOMe)$_2$ DMSO-d_6, at 298K, 600 MHz.

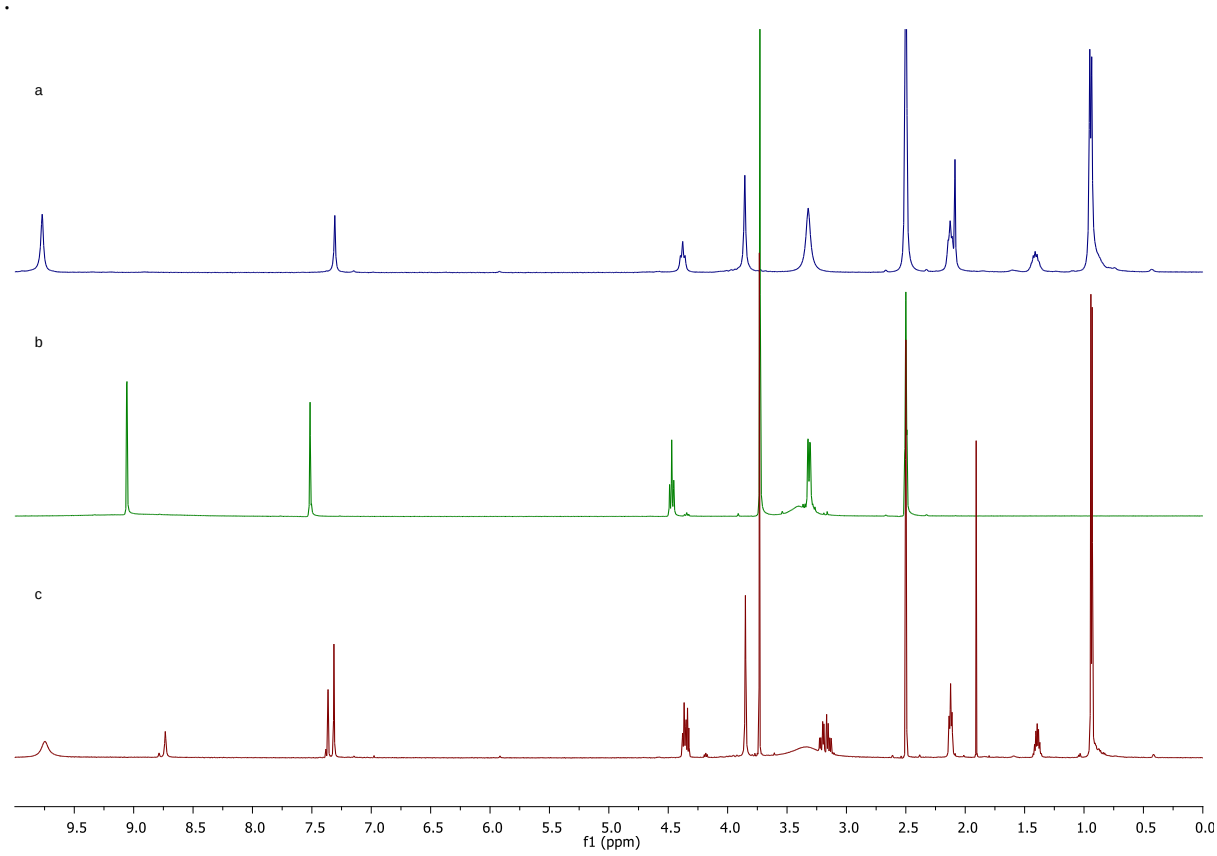

Figure S32. ^{1}H NMR (400 MHz and 600MHz, DMSO-d_6) spectra of (a) **1**; (b) L-histidine methyl ester; (c) **1**(HisOMe)$_2$.

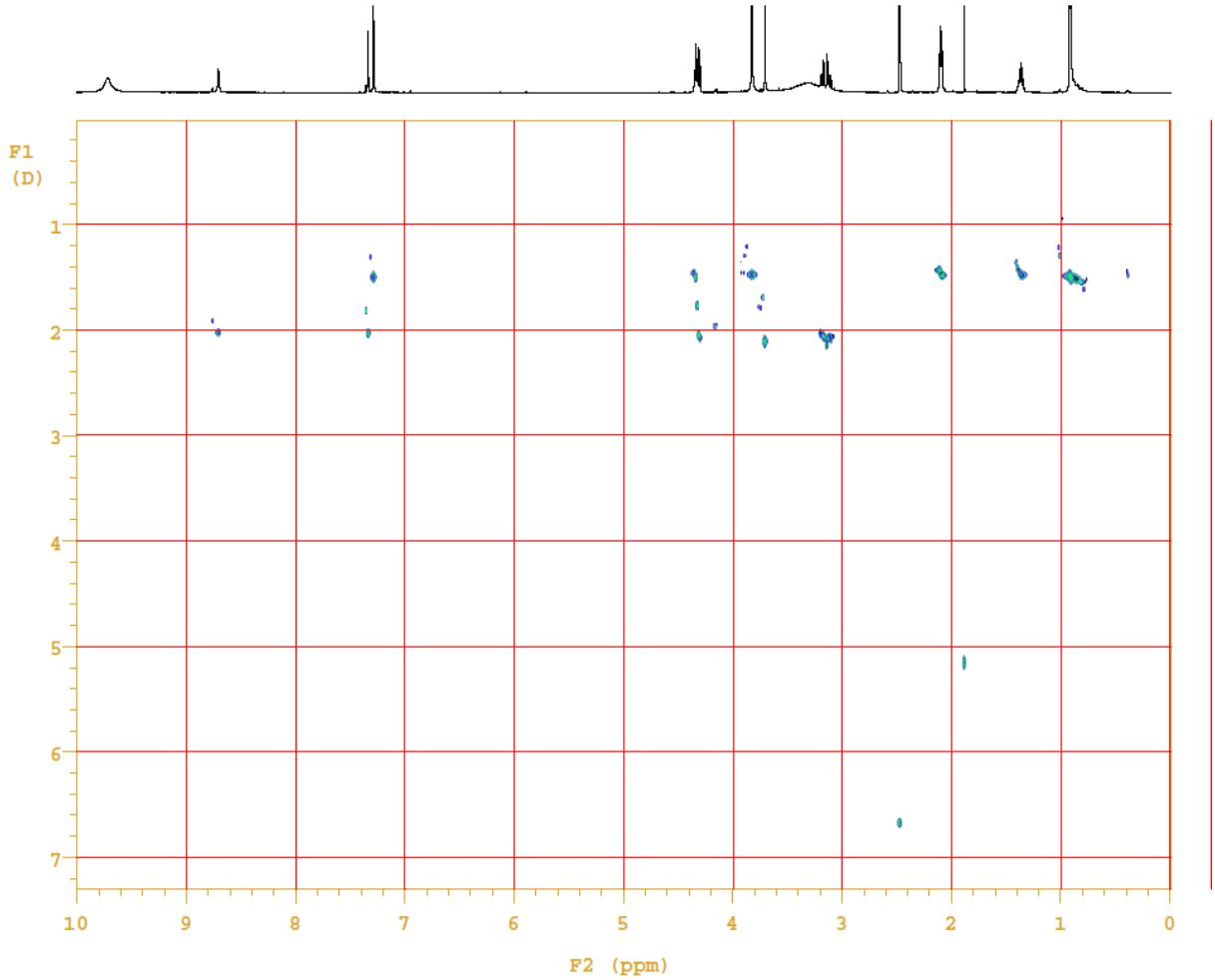

Figure S33. DOSY NMR spectrum of **1**(HisOMe)$_2$ DMSO-d_6, at 298K, 600 MHz.

1.3. NMR spectra of chiral cavitands

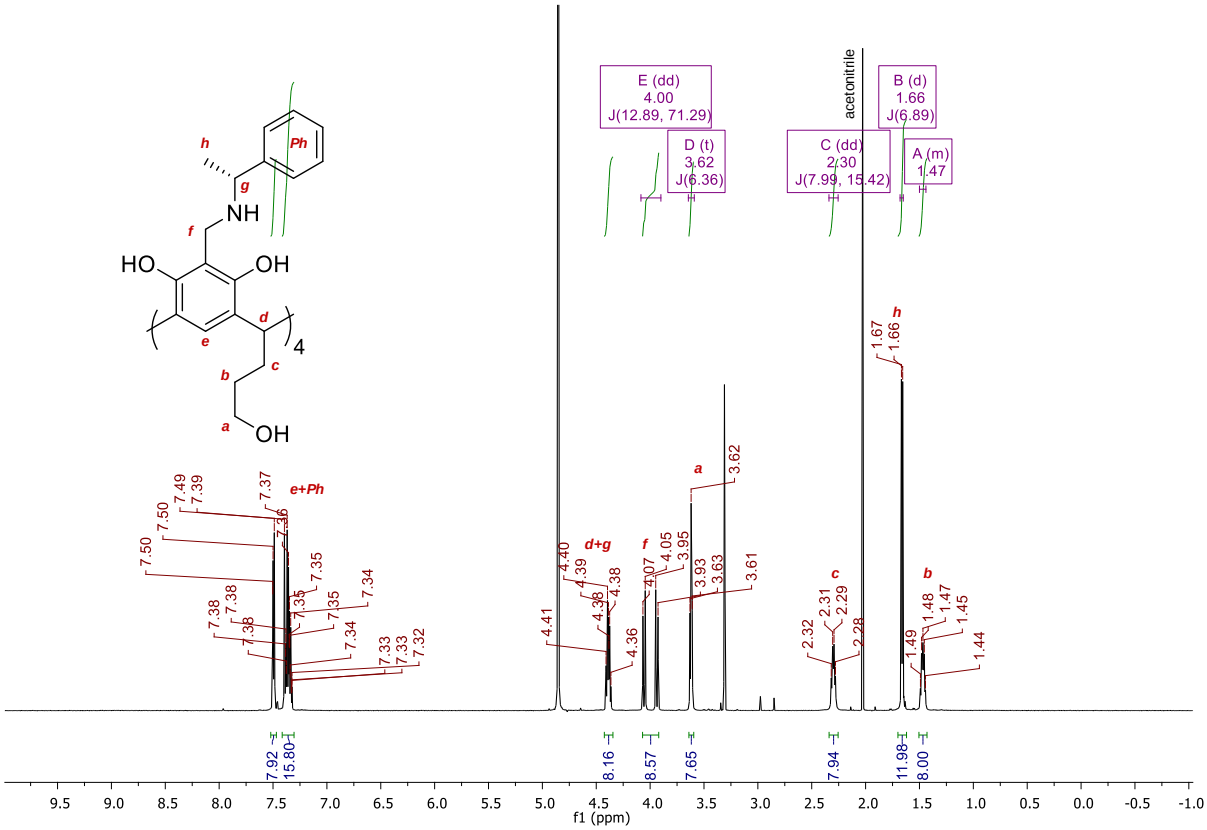

Figure S34. ^{1}H NMR (600 MHz, methanol-d_4) spectrum of (*R*)-**2**.

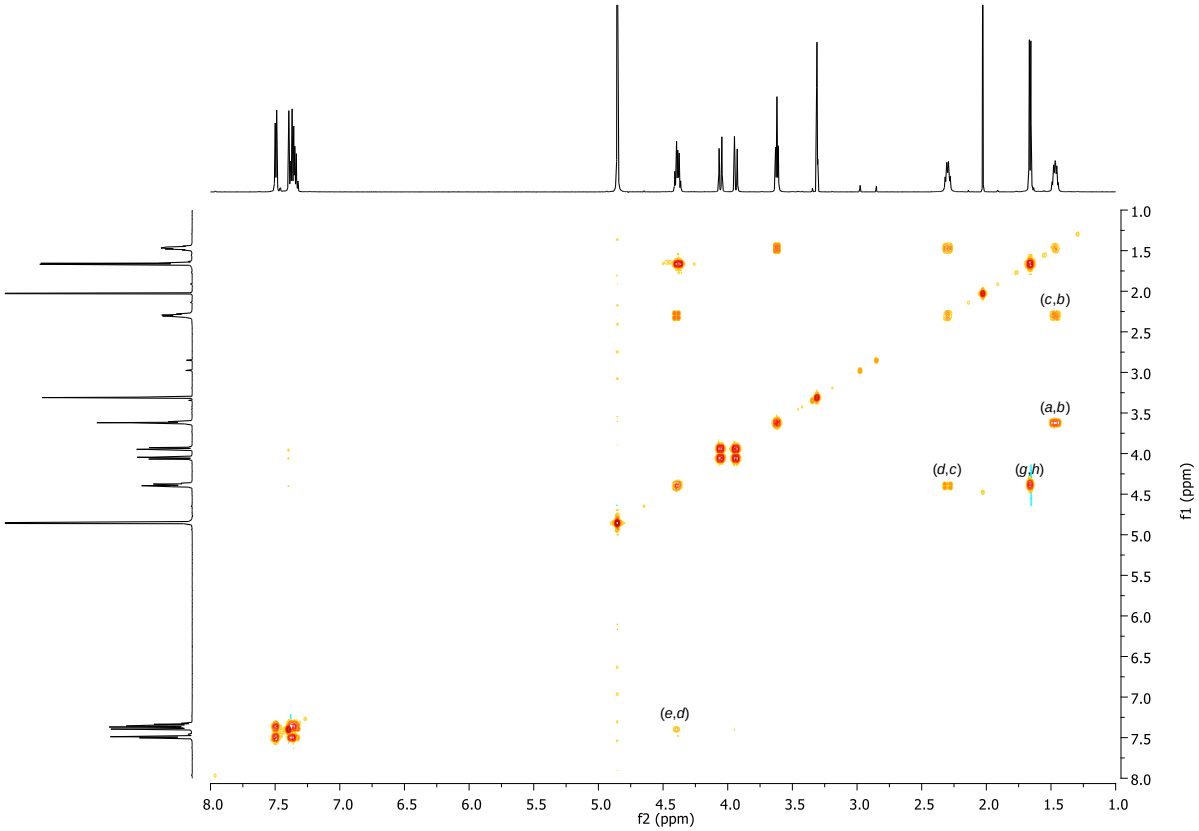

Figure S35. COSY NMR (600 MHz, methanol-d_4) spectrum of (*R*)-**2**.

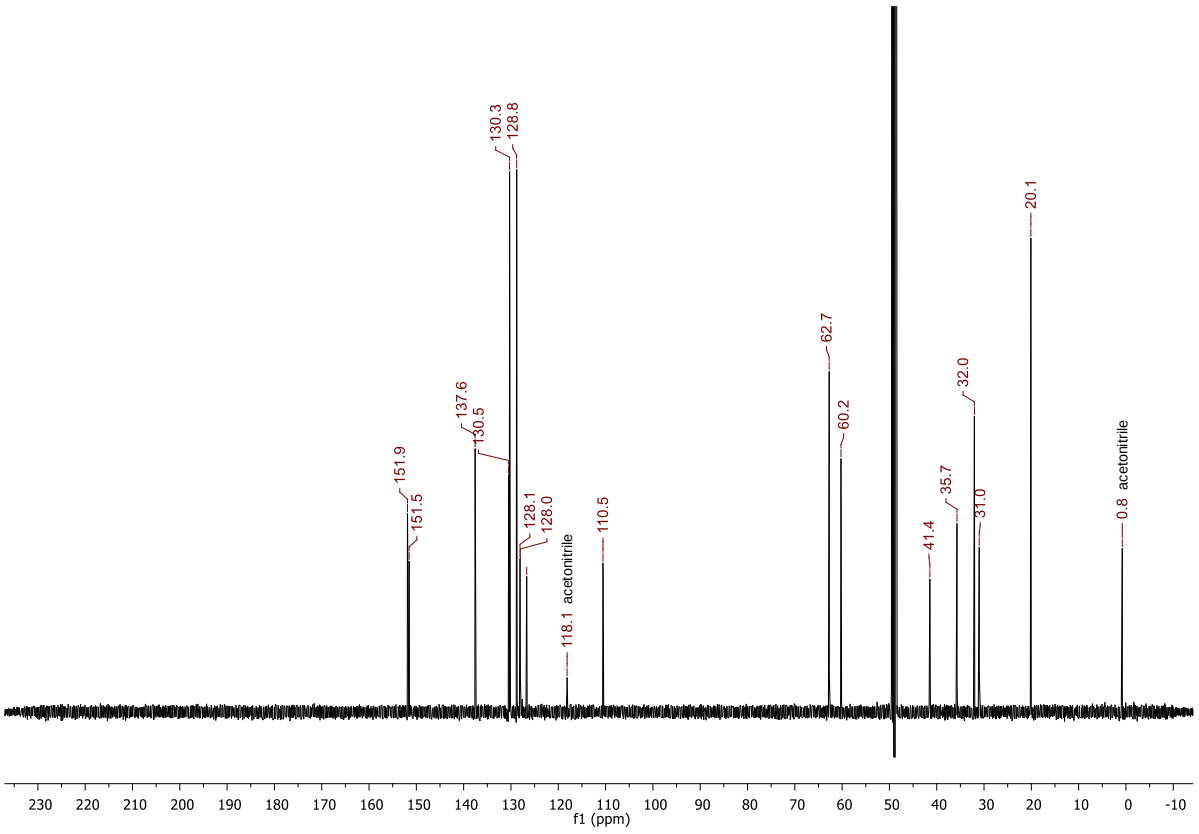

Figure S36. ^{13}C NMR (150 MHz, methanol-d_4) spectrum of (*R*)-**2**.

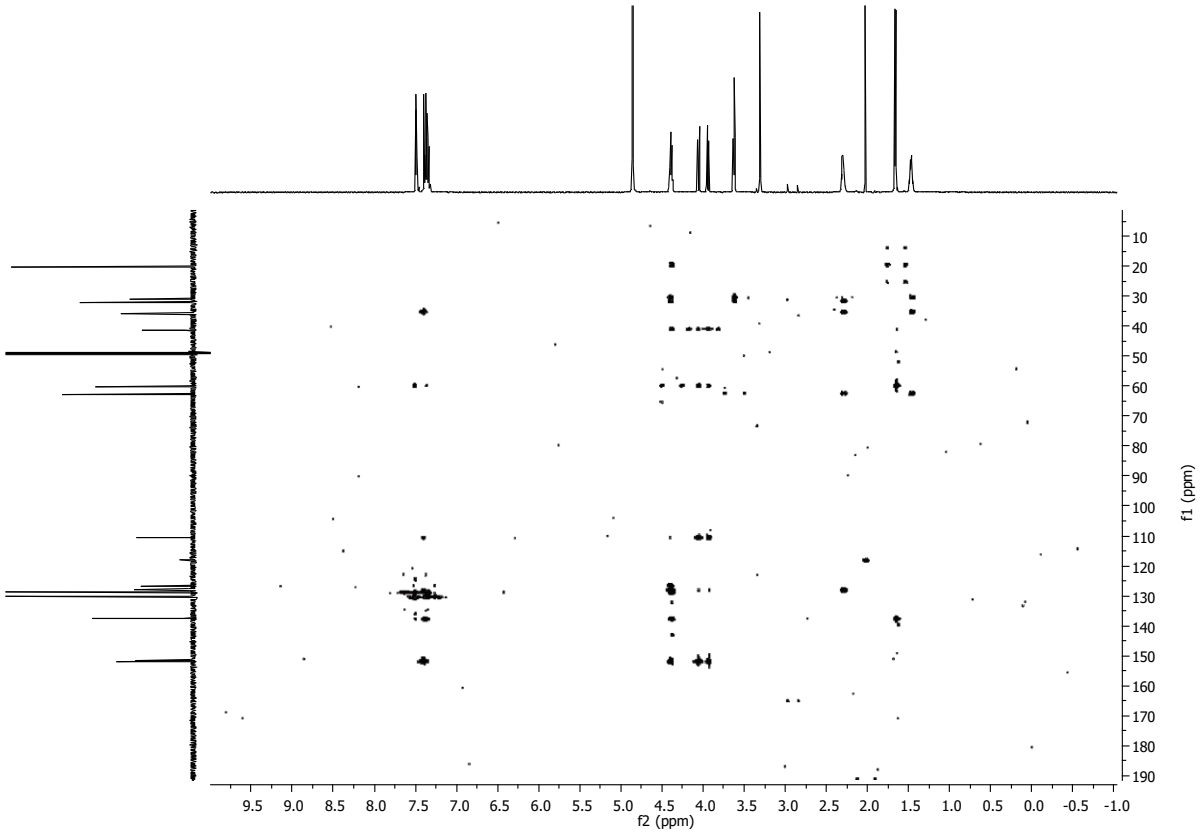

Figure S37. HMBC NMR (600 MHz, methanol-d_4) spectrum of (*R*)-**2**.

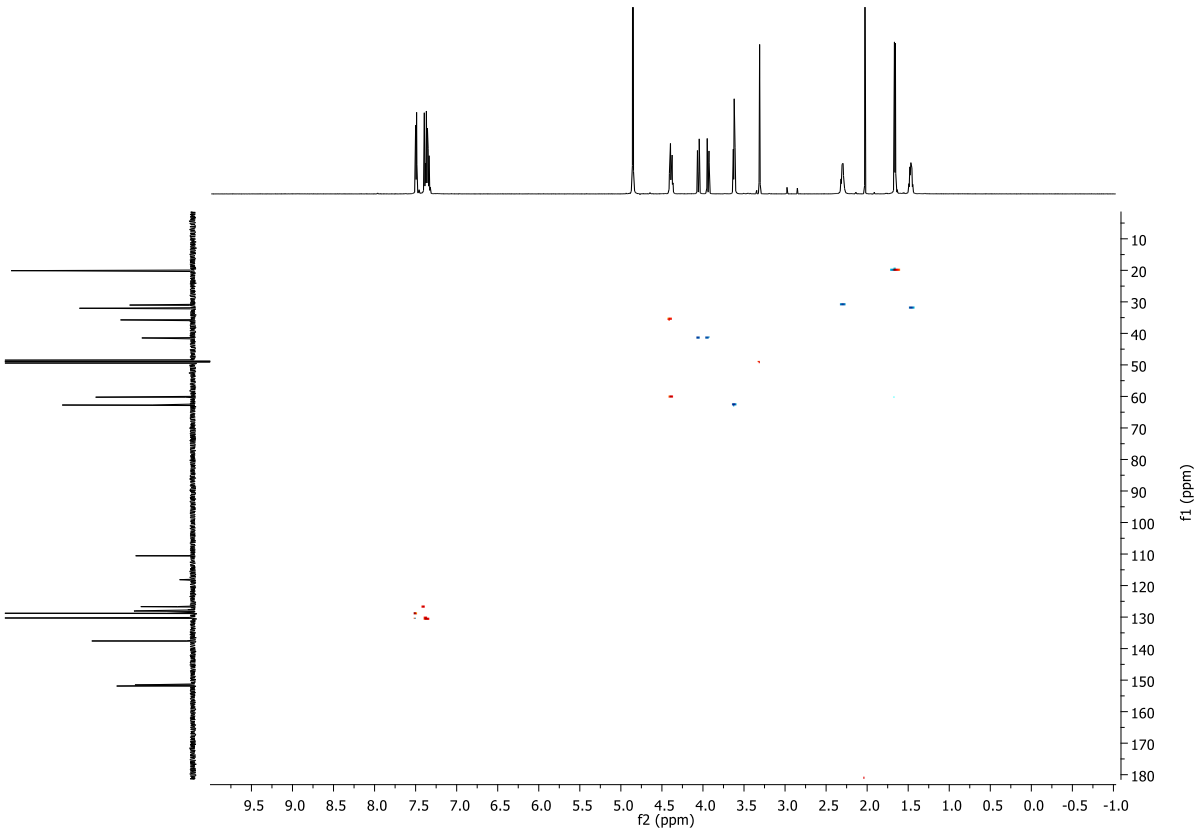

Figure S38. HSQC NMR (600 MHz, methanol-d_4) spectrum of (*R*)-**2**.

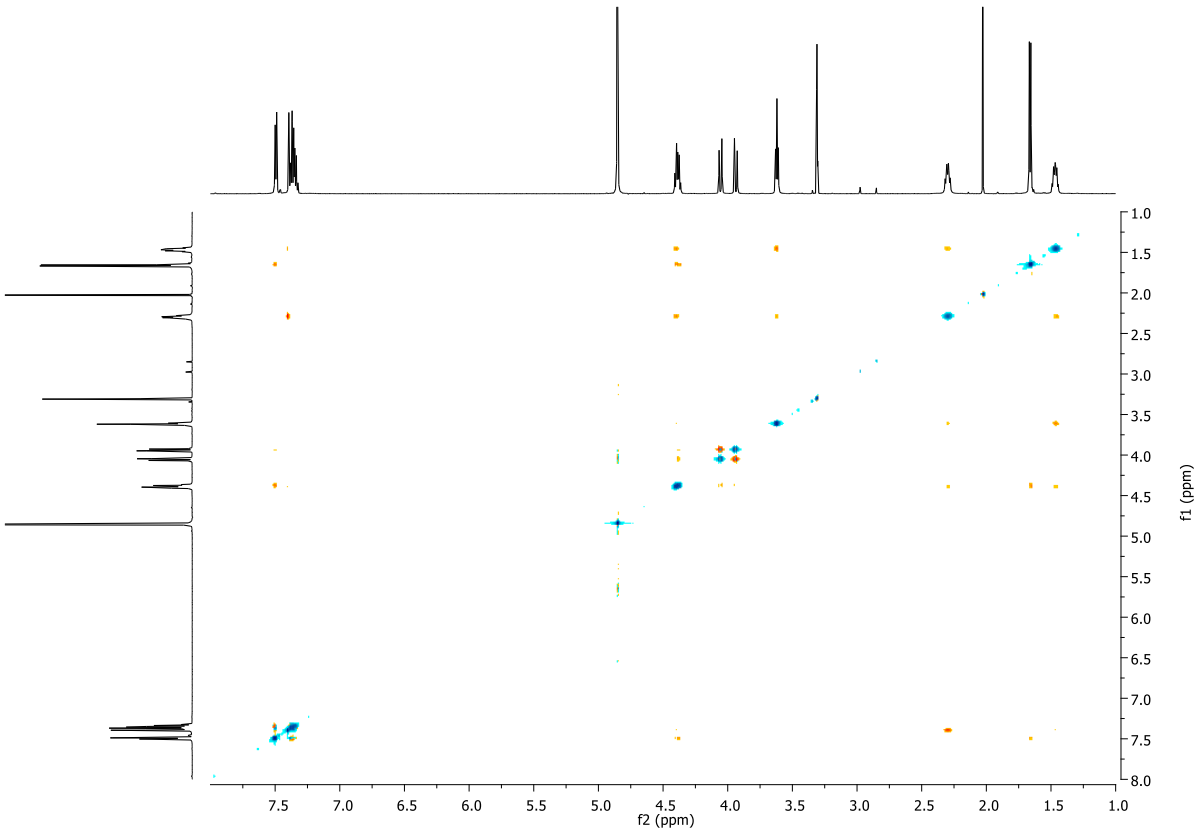

Figure S39. ROESY NMR (600 MHz, methanol-d_4) spectrum of (*R*)-**2**.

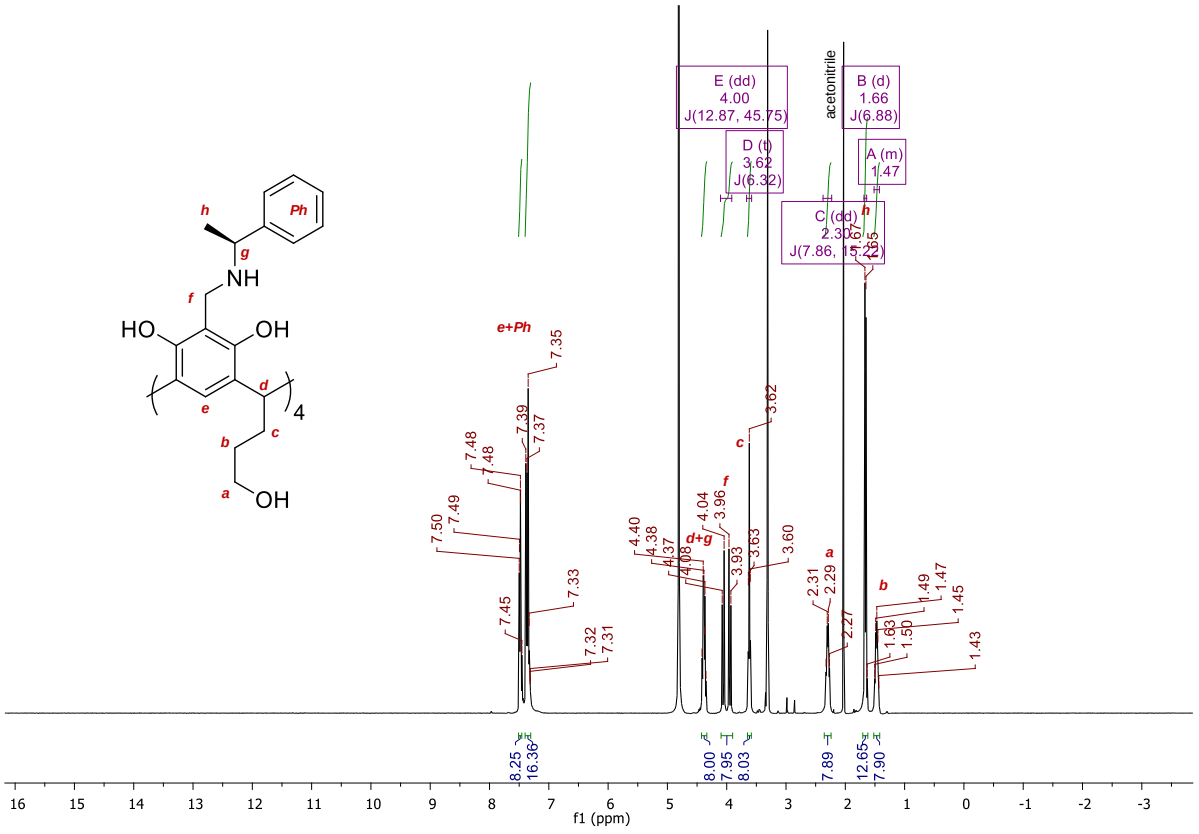

Figure S40. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of (*S*)-**2**.

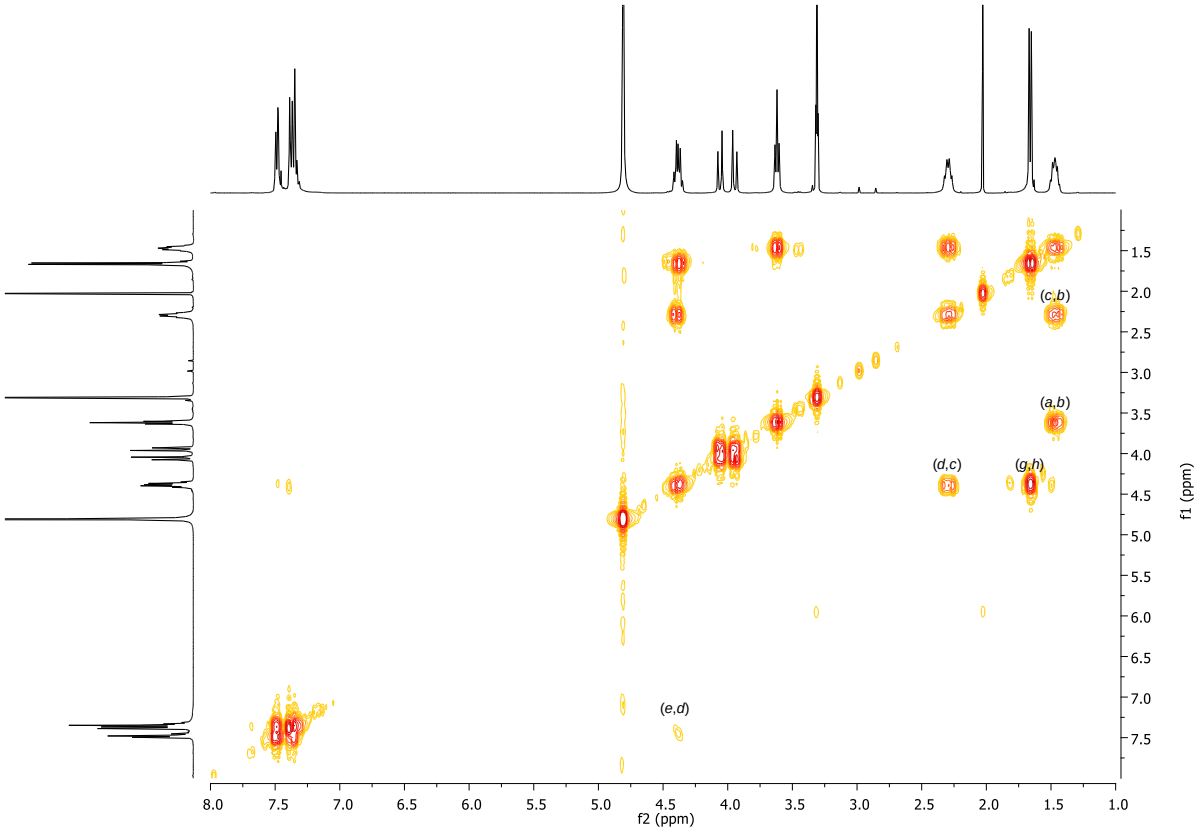

Figure S41 COSY NMR (400 MHz, methanol-d_4) spectrum of (*S*)-**2**.

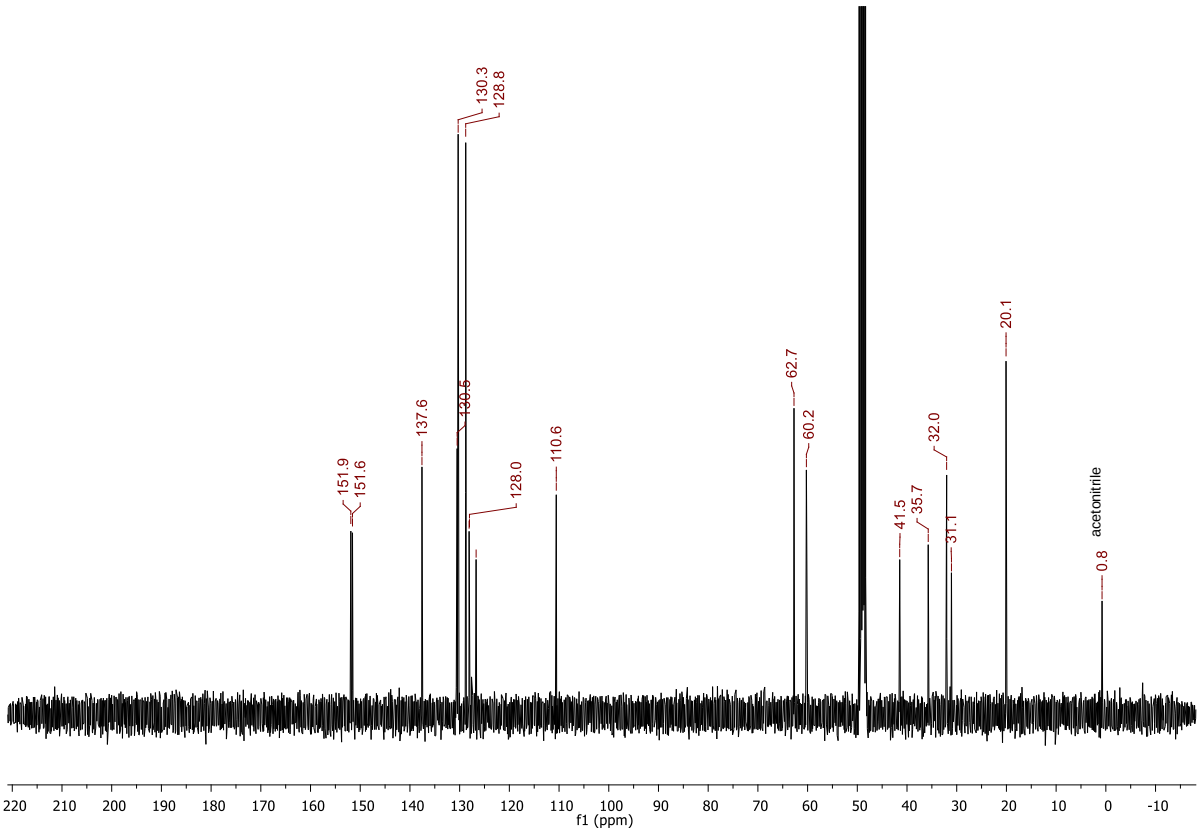

Figure S42. ^{13}C NMR (100 MHz, methanol-d_4) spectrum of (*S*)-**2**.

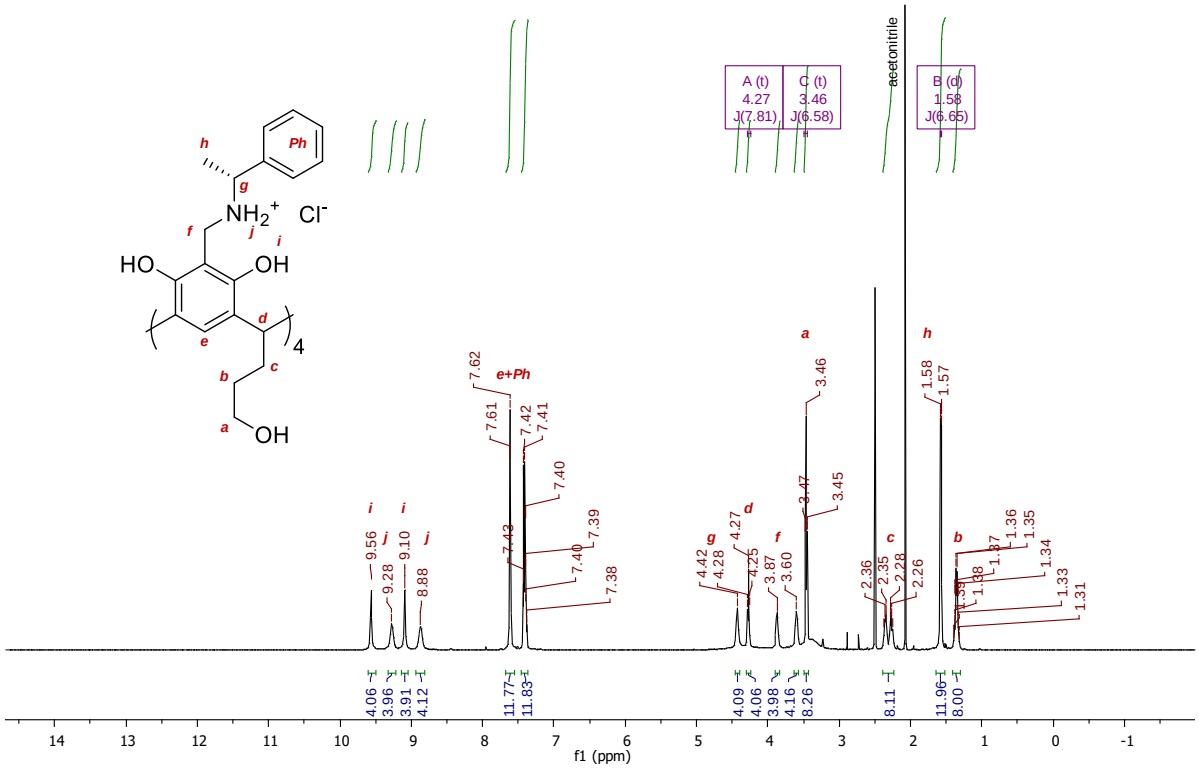

Figure S43. ^{1}H NMR (600 MHz, DMSO-d_6) spectrum of (*R*)-**2**.

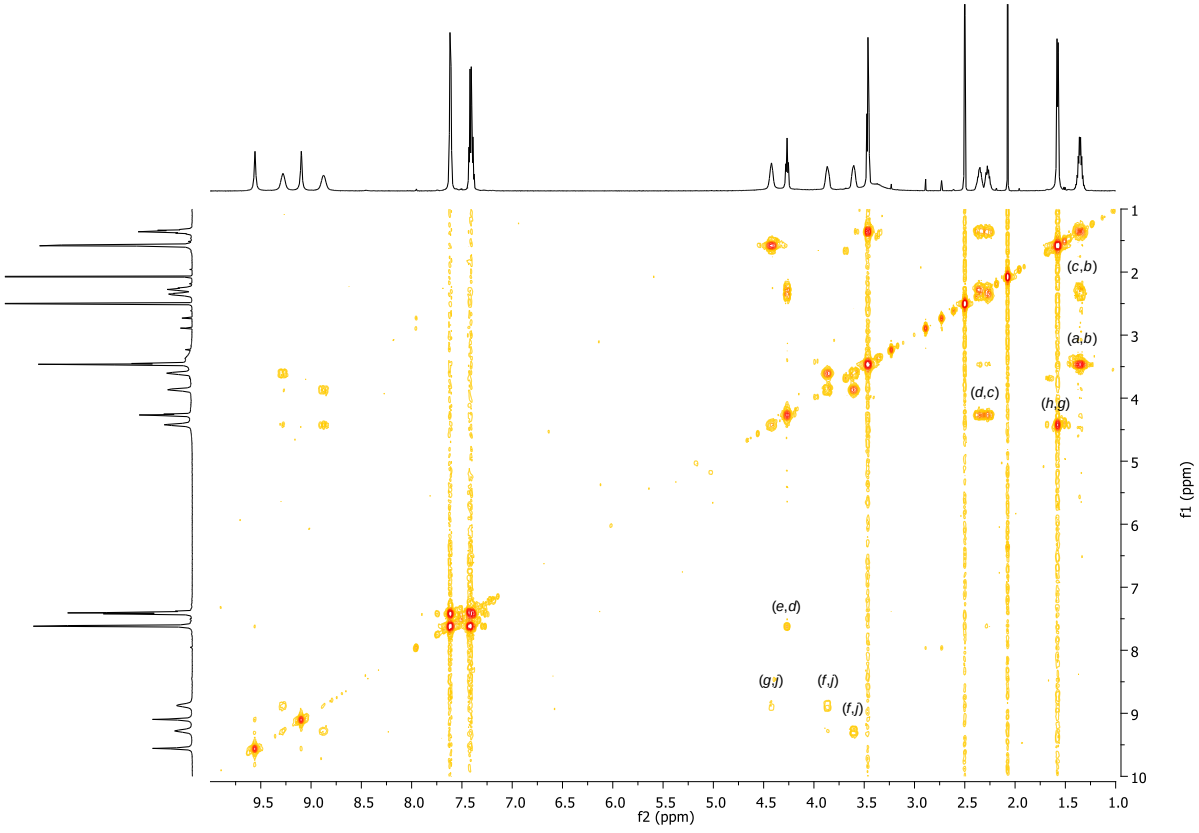

Figure S44. COSY NMR (600 MHz, DMSO-d_6) spectrum of (*R*)-**2**.

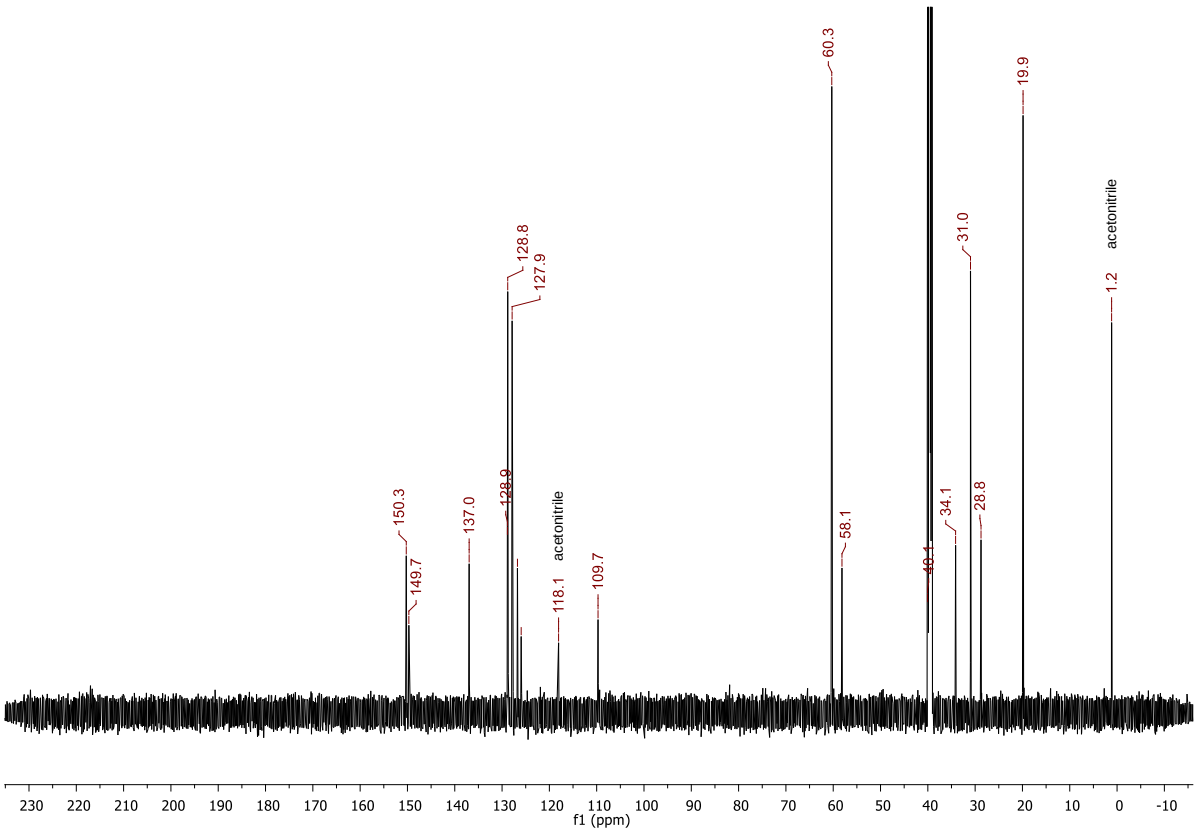

Figure S45. ^{13}C NMR (150 MHz, DMSO-d_6) spectrum of (*R*)-**2**.

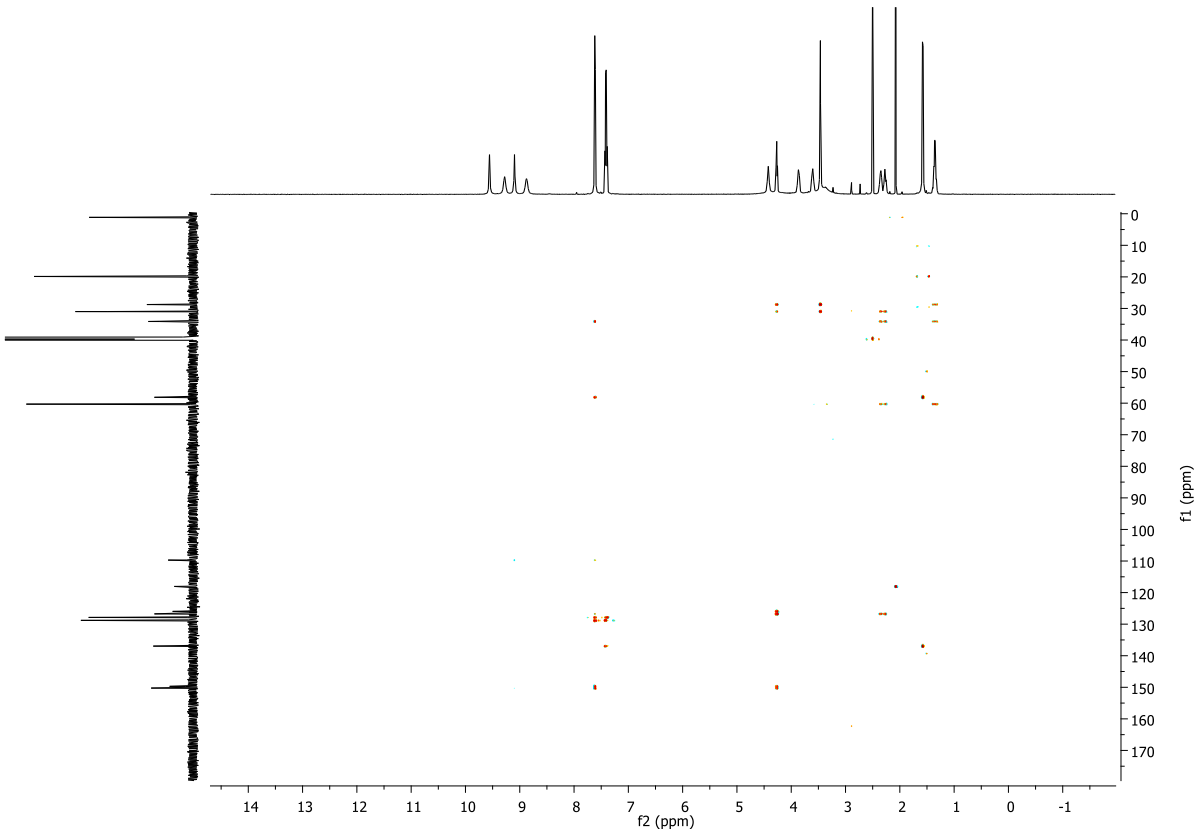

Figure S46. HMBC NMR (600 MHz, DMSO-d_6) spectrum of (*R*)-**2**.

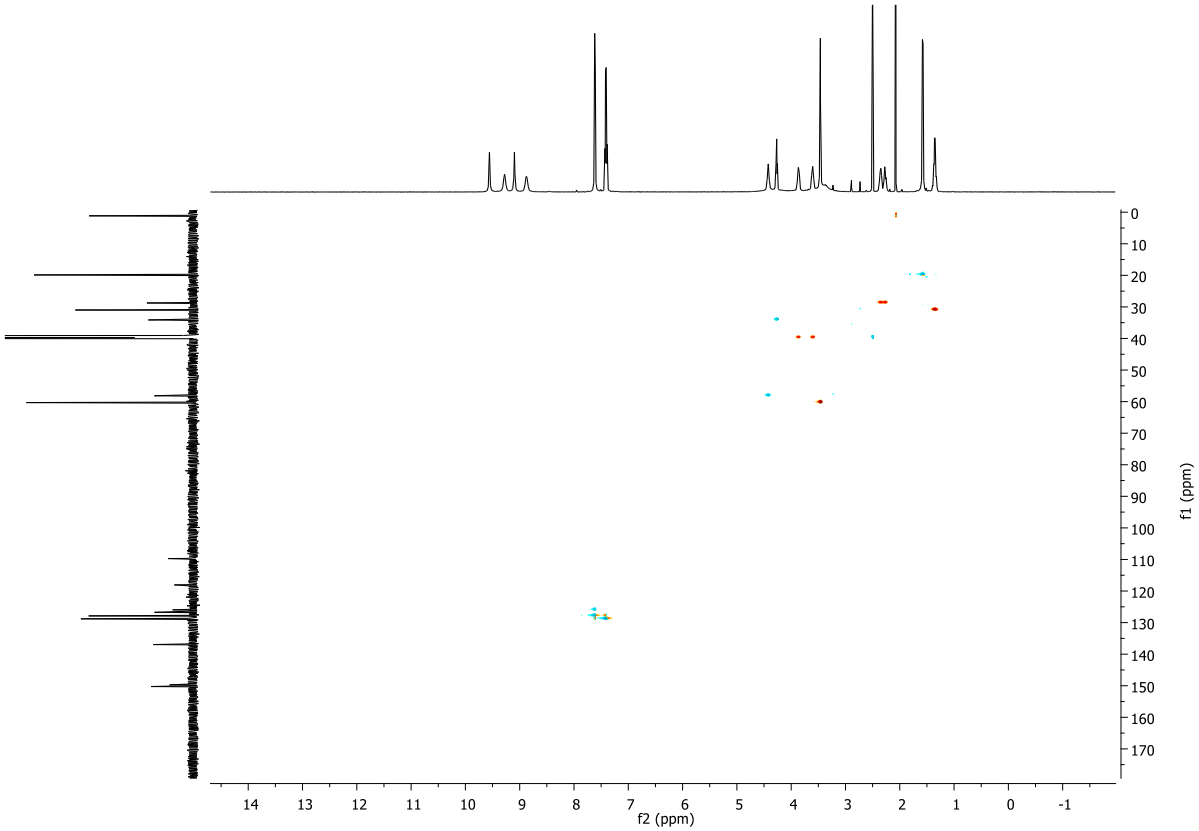

Figure S47. HSQC NMR (600 MHz, DMSO-d_6) spectrum of (*R*)-**2**.

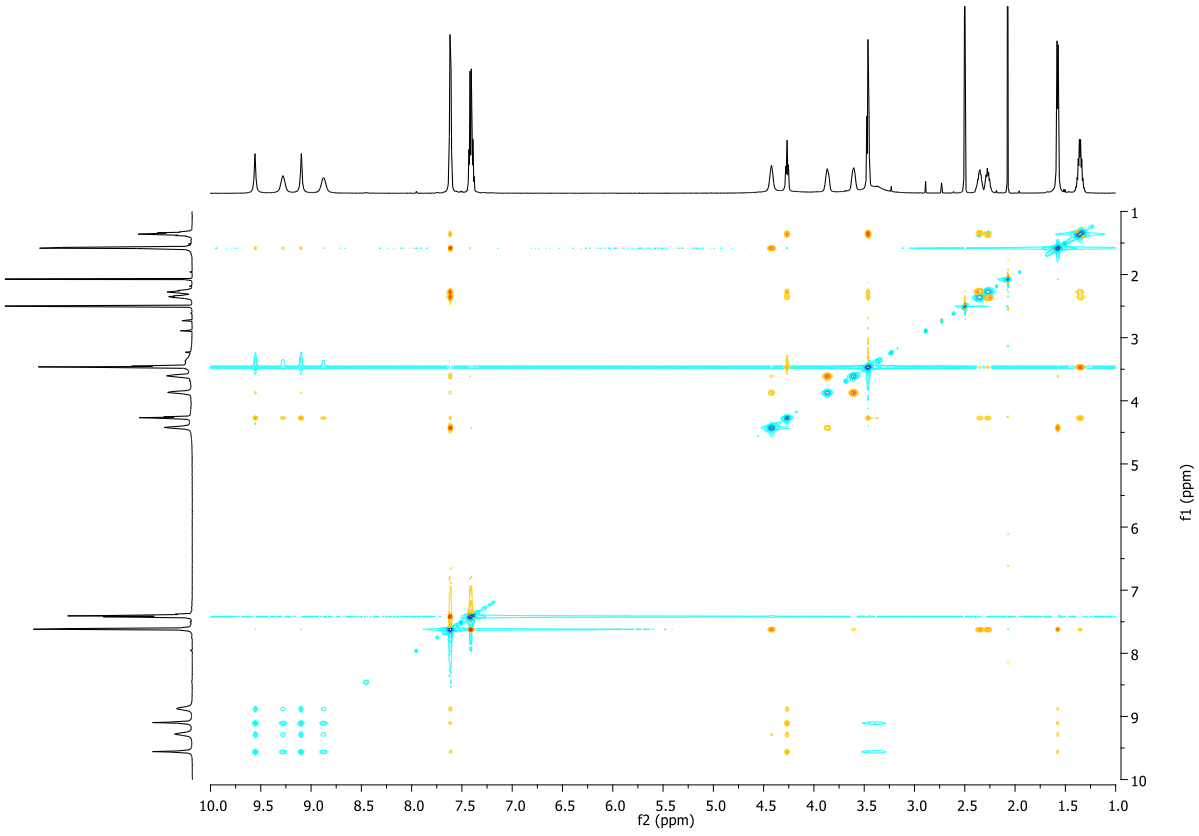

Figure S48. ROESY NMR (600 MHz, DMSO-d_6) spectrum of (*R*)-**2**.

1.4. NMR spectra of capsules

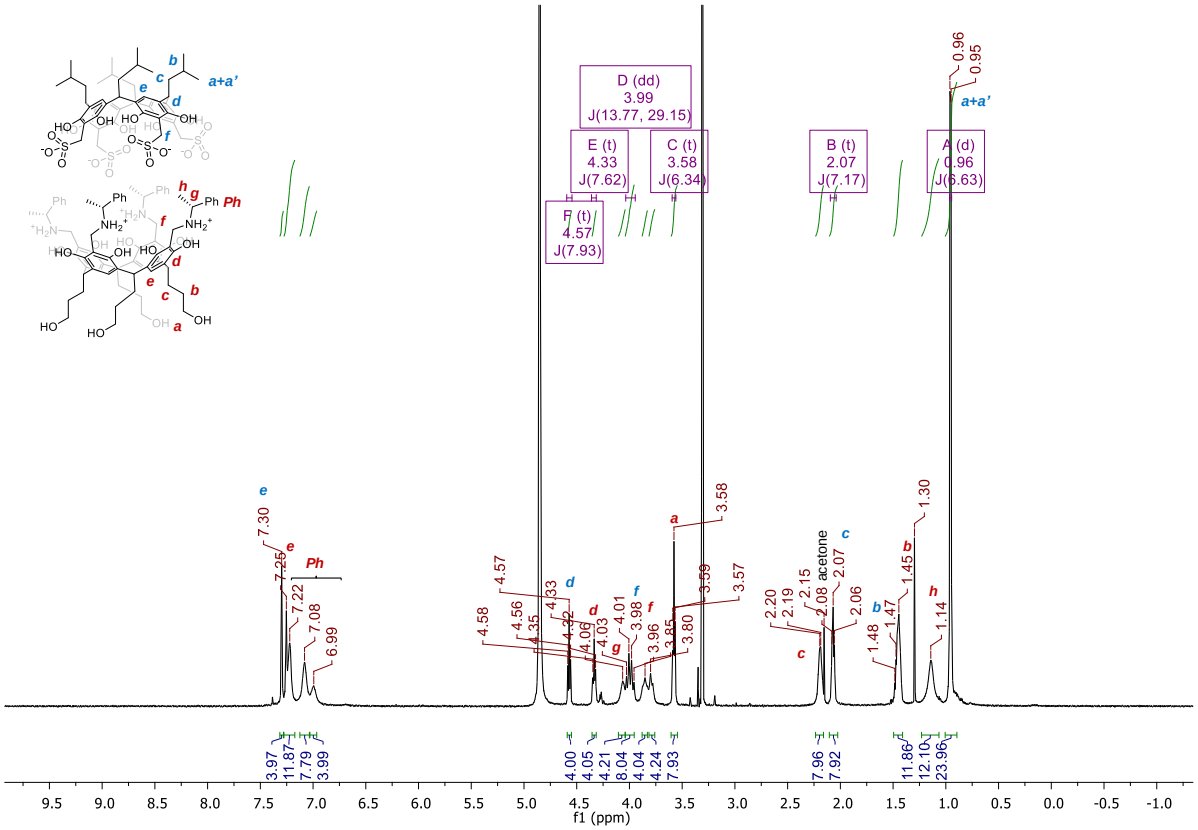

Figure S49. ^{1}H NMR (600 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*R*)-**2**.

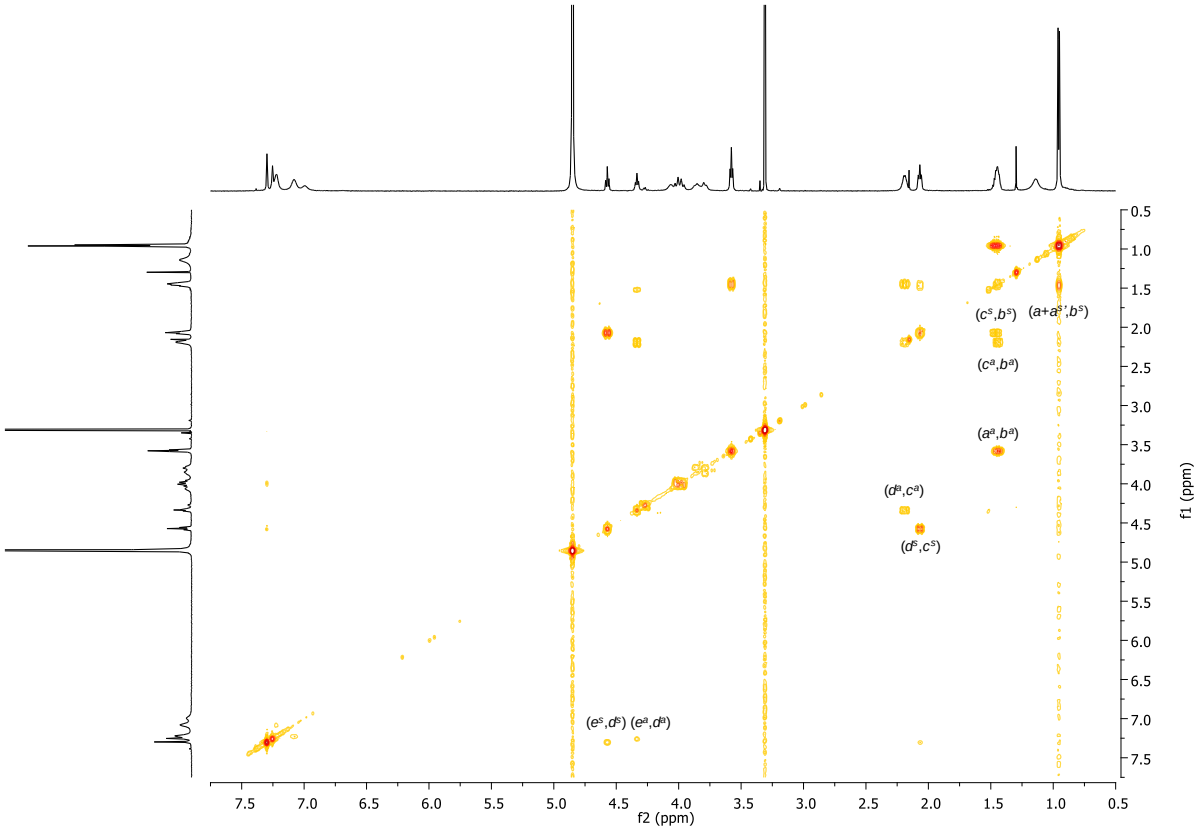

Figure S50. COSY NMR (600 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*R*)-**2**.

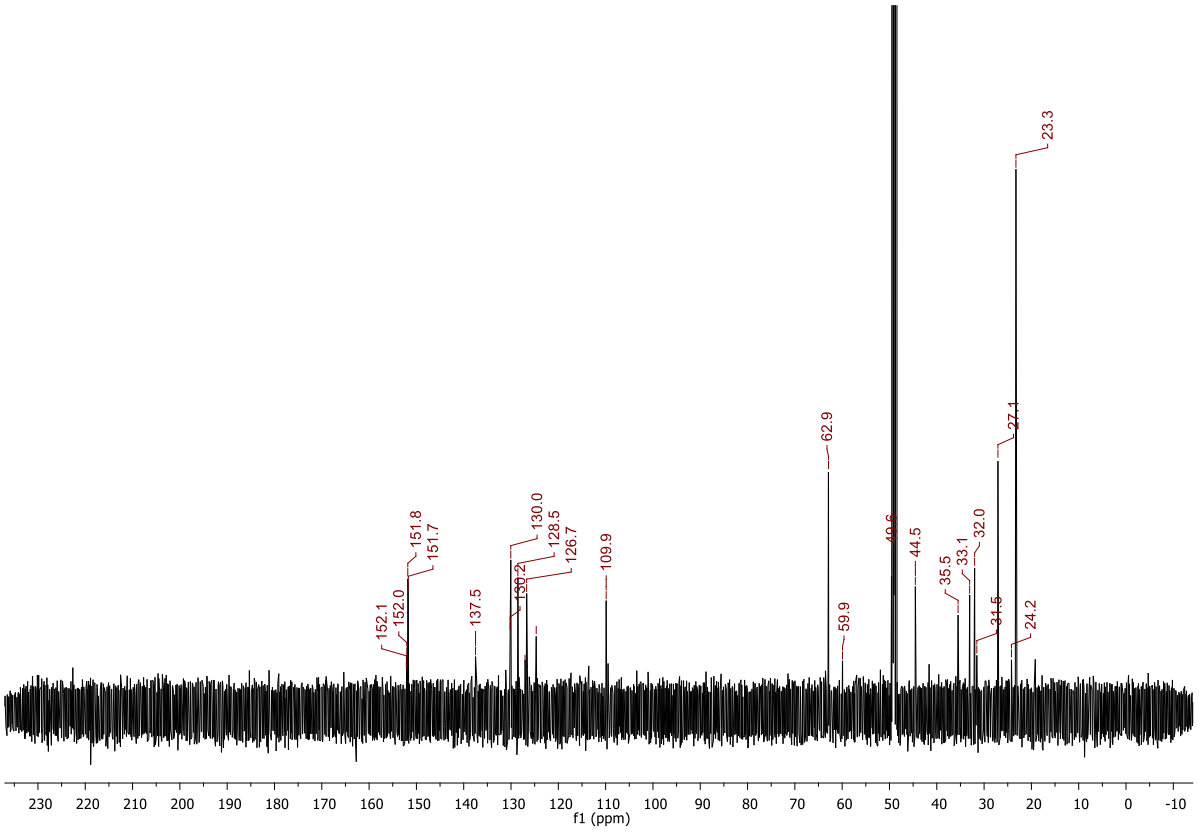

Figure S51. ^{13}C NMR (150 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*R*)-**2**.

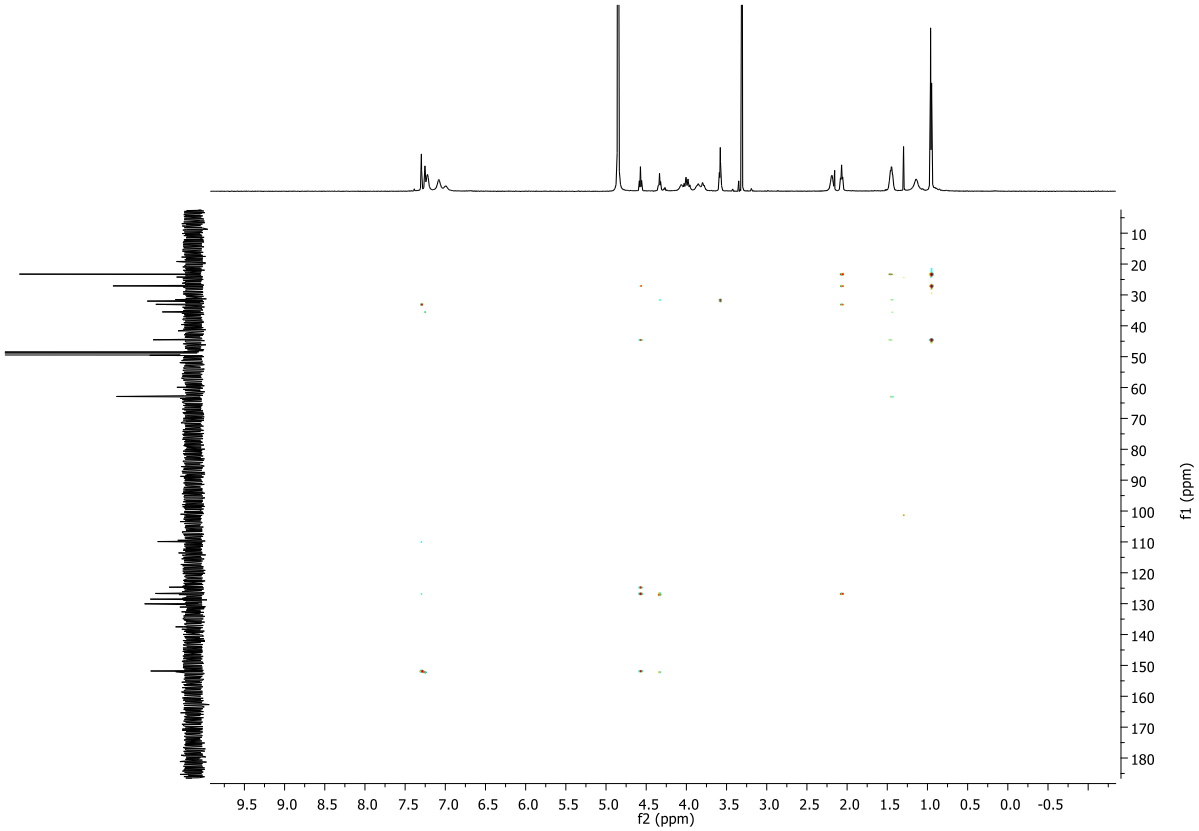

Figure S52. HMBC NMR (600 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*R*)-**2**.

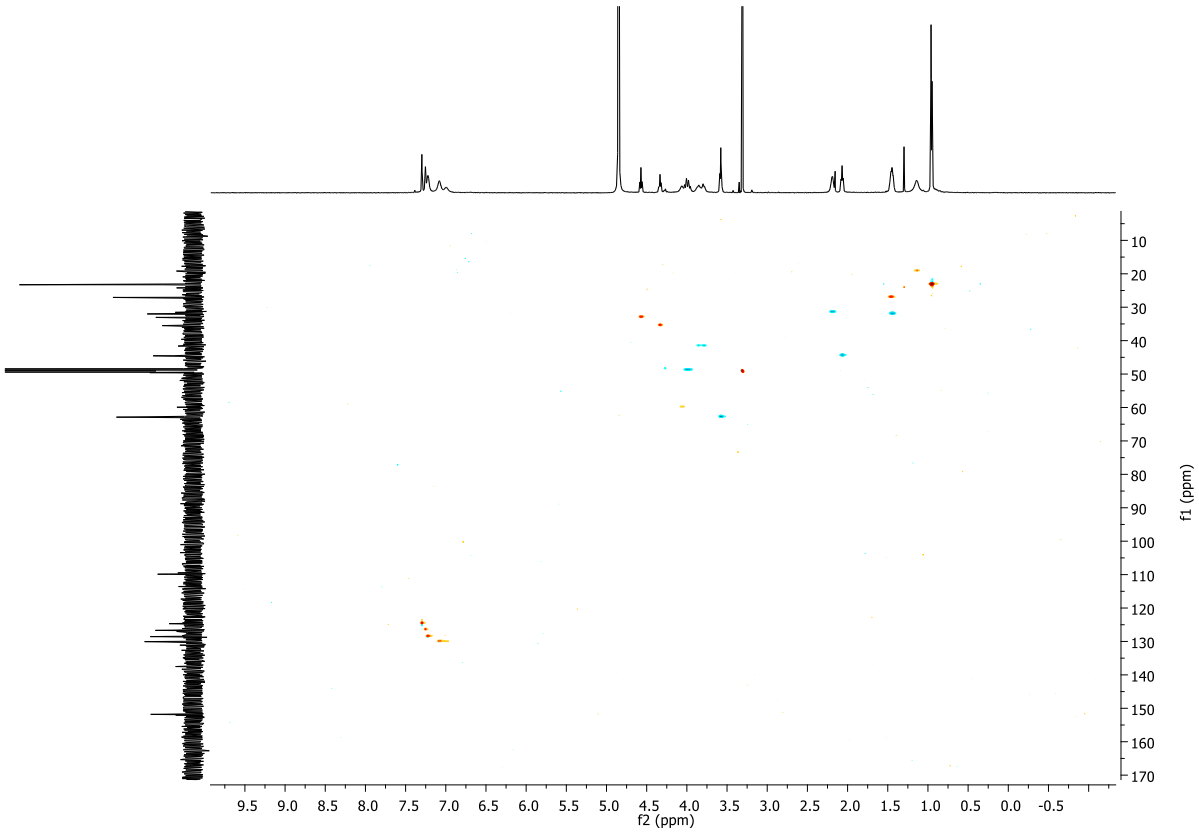

Figure S53. HSQC NMR (600 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*R*)-**2**.

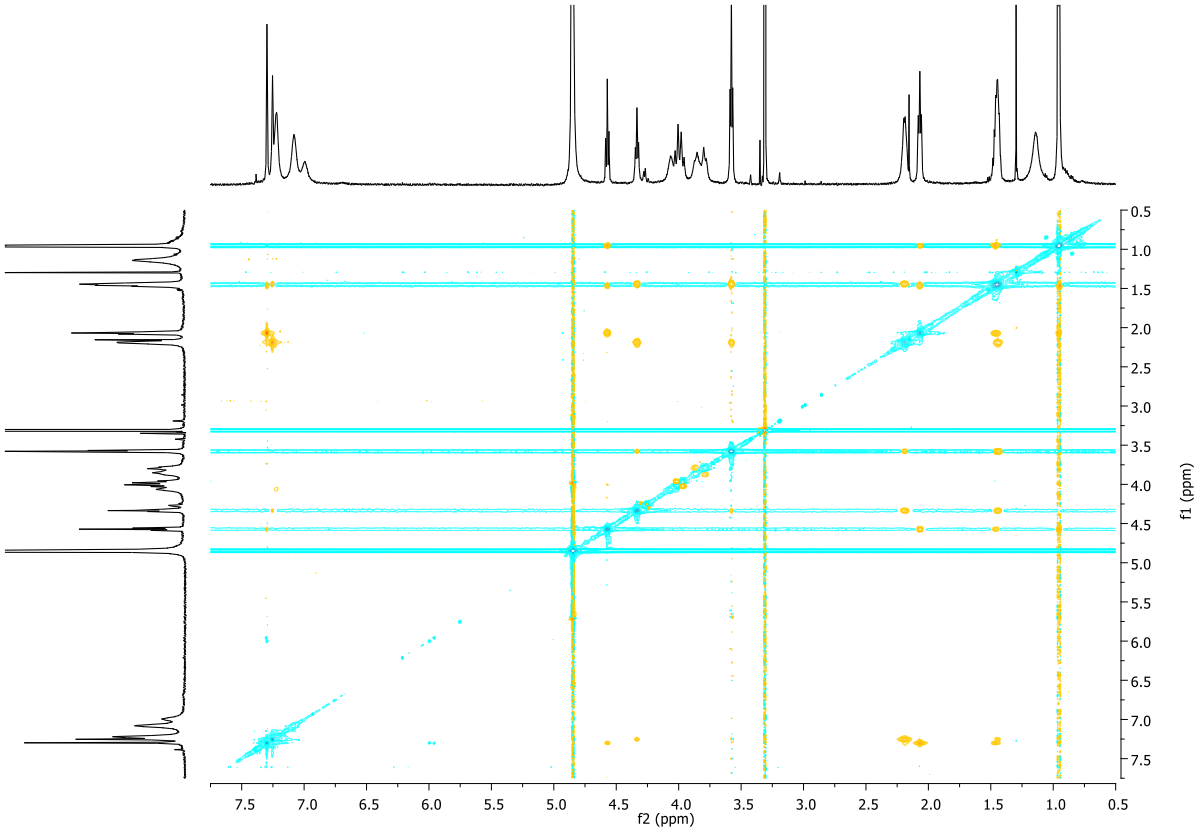

Figure S54. ROESY NMR (600 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*R*)-**2**.

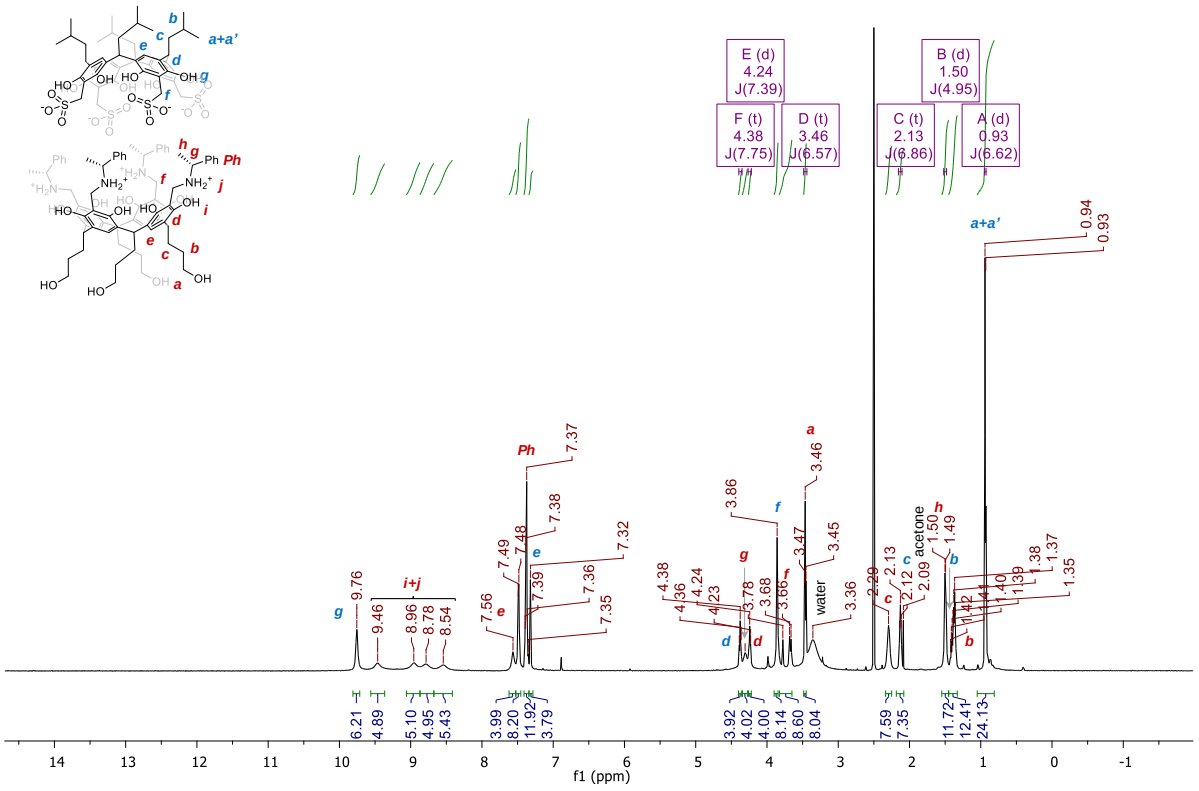

Figure S55. ^{1}H NMR (600 MHz, DMSO-d_6) spectrum of capsule composed of **1** + (*R*)-**2**.

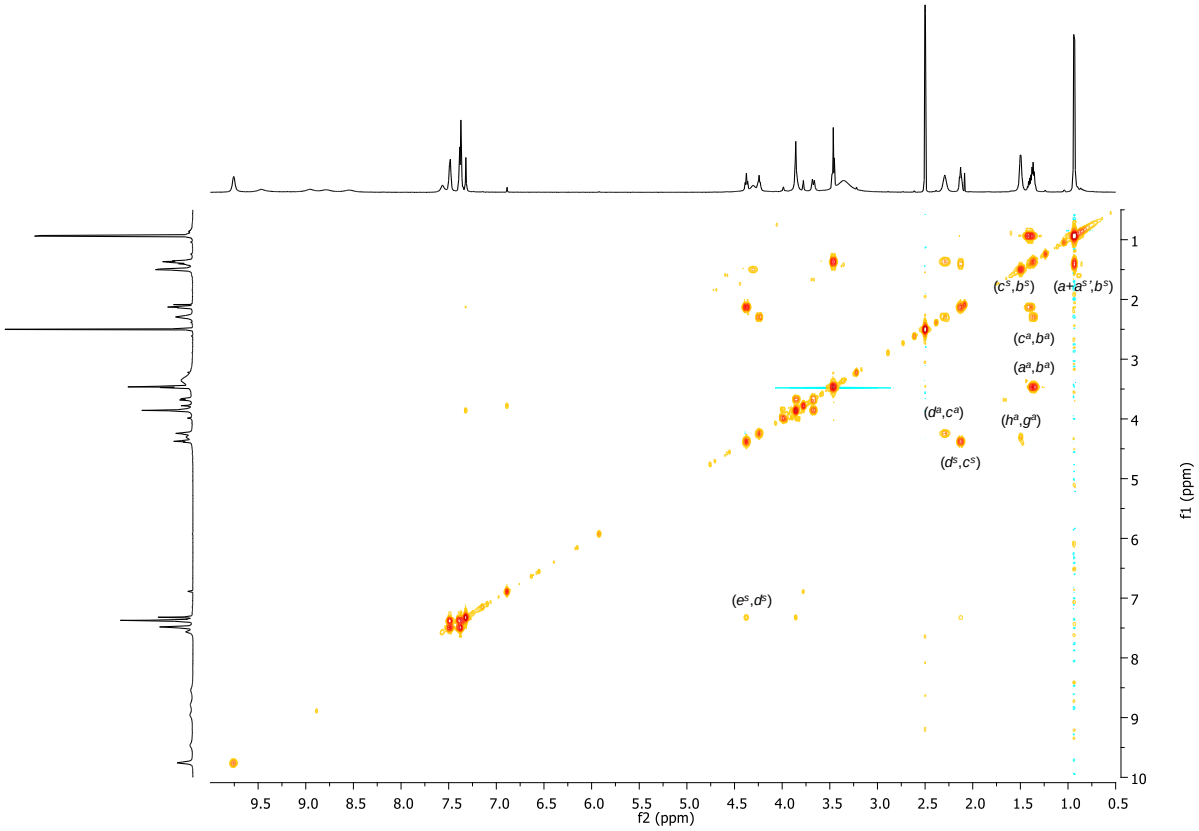

Figure S56. COSY NMR (600 MHz, DMSO-d_6) spectrum of capsule composed of **1** + (*R*)-**2**.

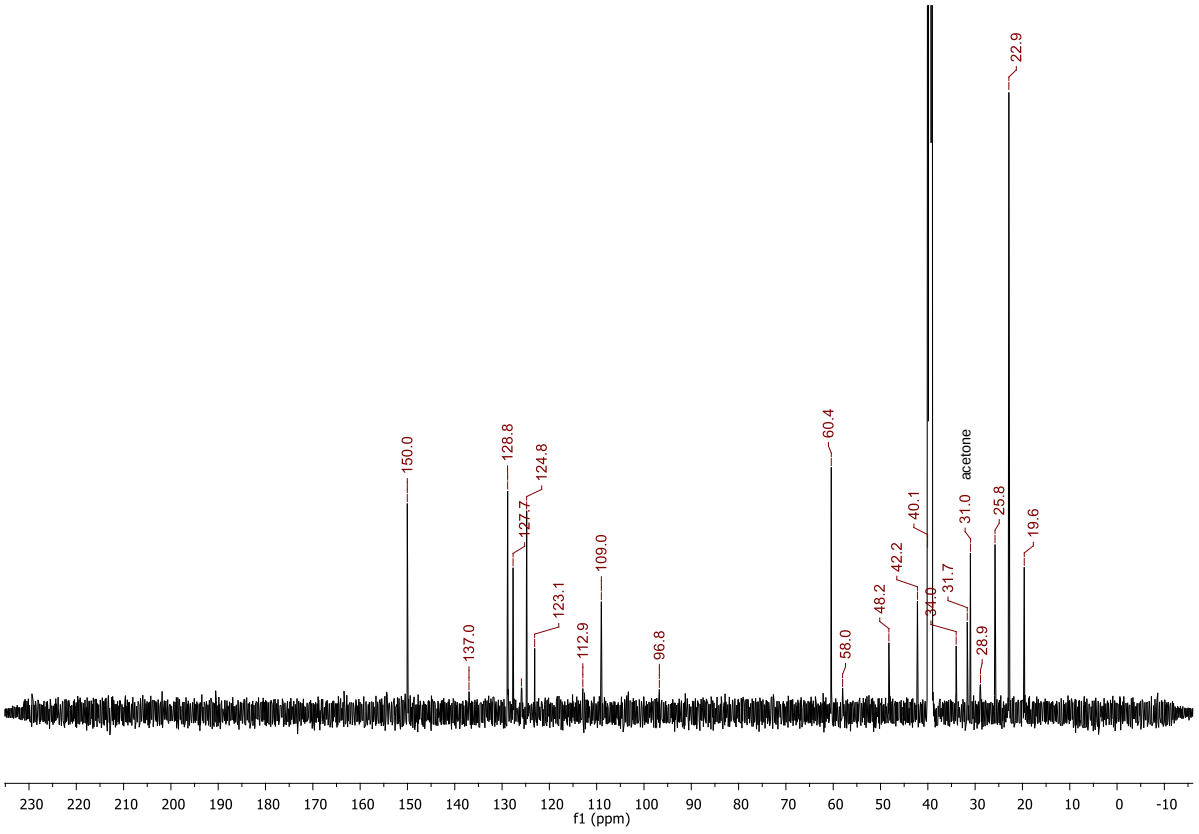

Figure S57. ^{13}C NMR (150 MHz, DMSO-d_6) spectrum of capsule composed of **1** + (*R*)-**2**.

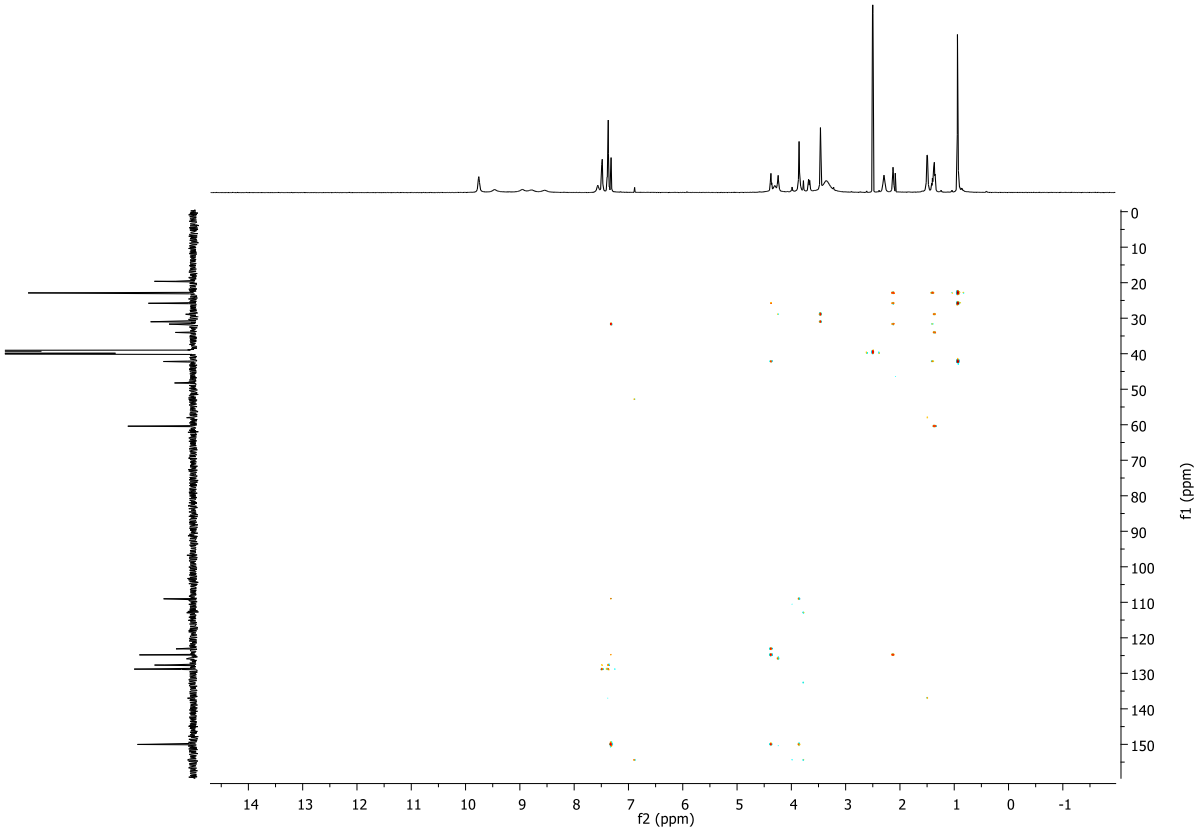

Figure S58. HMBC NMR (600 MHz, DMSO-d_6) spectrum of capsule composed of **1** + (*R*)-**2**.

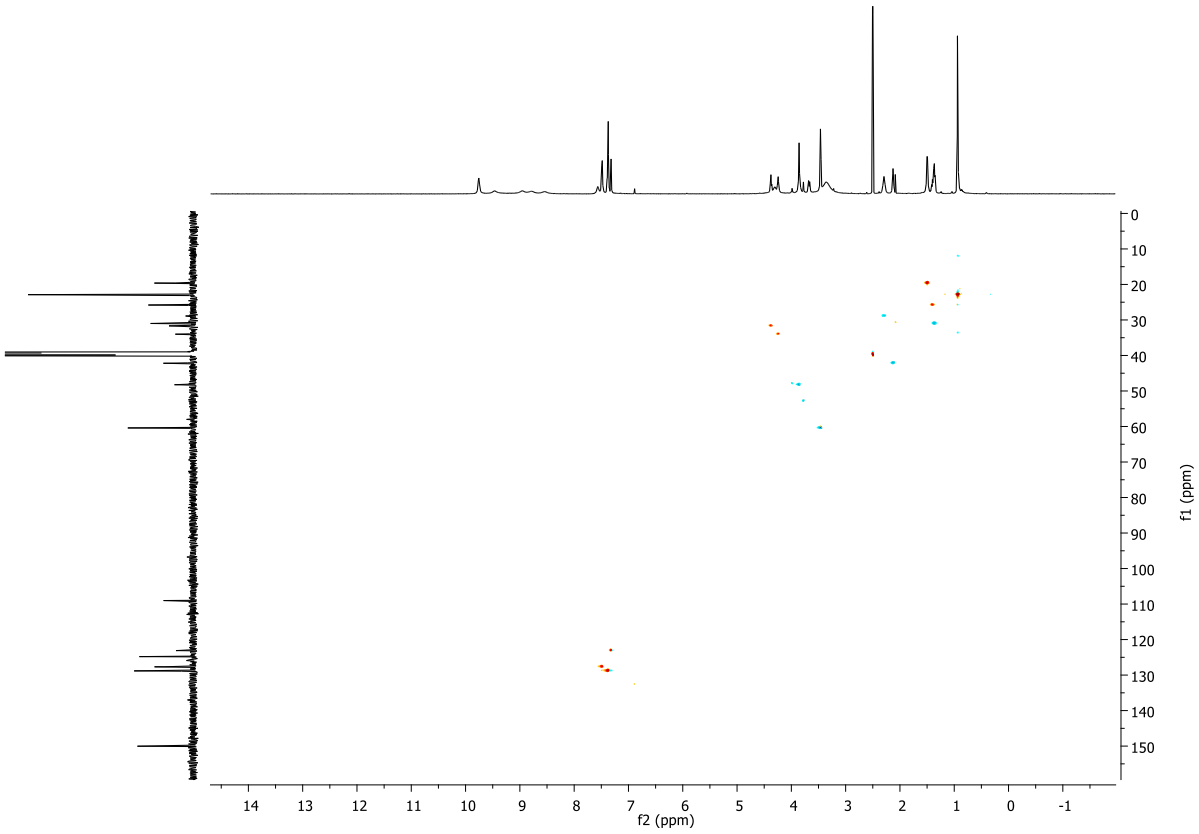

Figure S59. HSQC NMR (600 MHz, DMSO-d_6) spectrum of capsule composed of **1** + (*R*)-**2**.

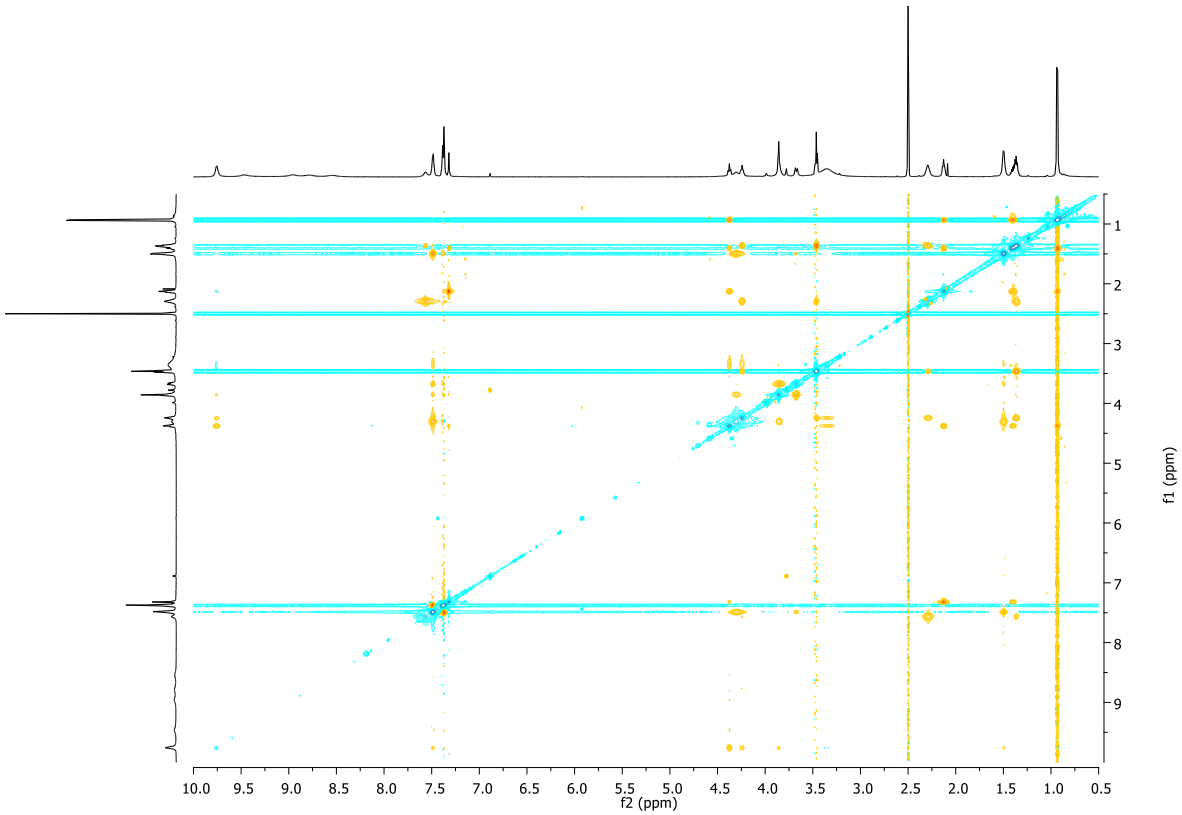

Figure S60. ROESY NMR (600 MHz, DMSO-d_6) spectrum of capsule composed of **1** + (*R*)-**2**.

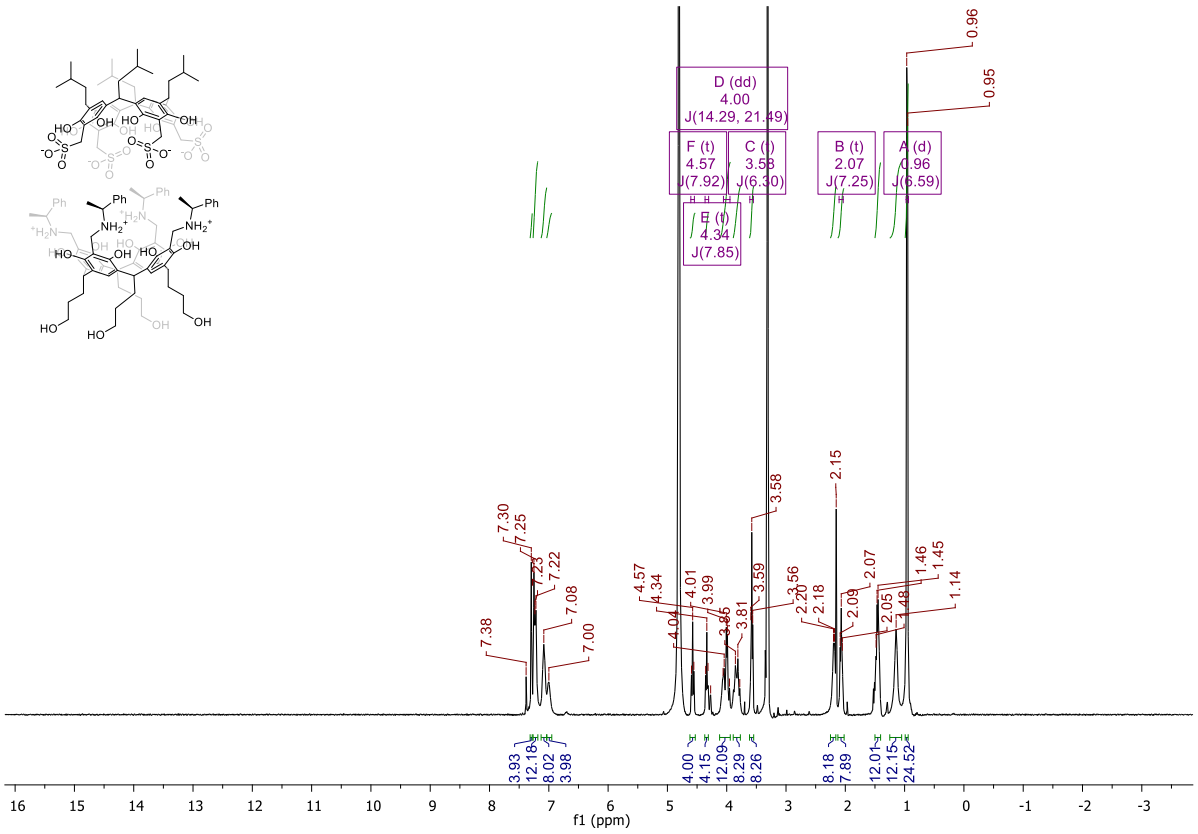

Figure S61. ^{1}H NMR (400 MHz, methanol-d_4) spectrum of capsule composed of **1** + (*S*)-**2**.

2. UV and ECD spectra

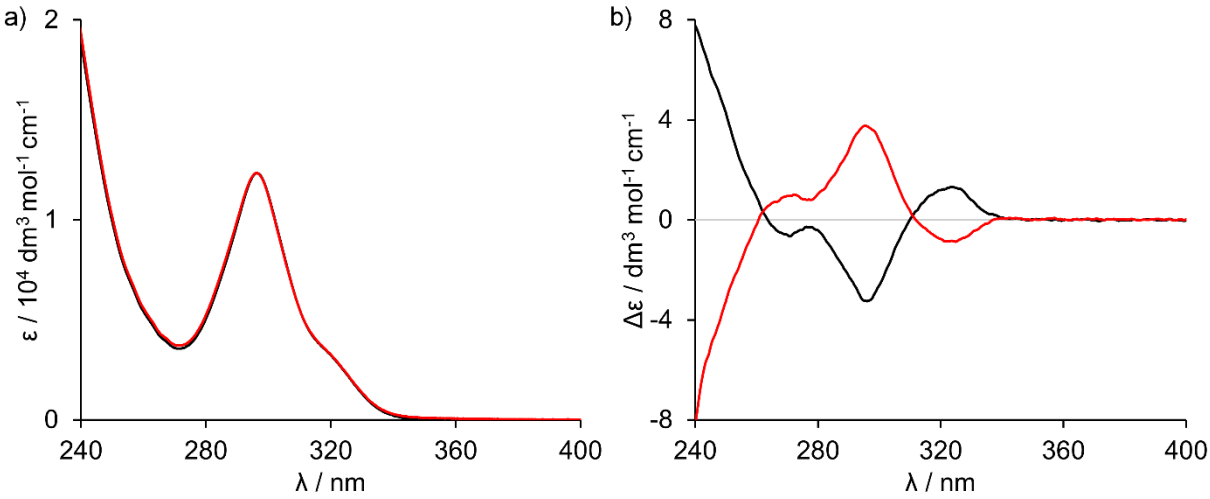

Figure S62. a) UV spectra of (*R*)-**2** (**black**) and (*S*)-**2** (**red**) in H_2O; b) ECD spectra of (*R*)-**2** (**black**) and (*S*)-**2** (**red**) in H_2O. The intensities of all spectra were normalized by using the UV band at 296 nm of sample (*R*)-**2**.

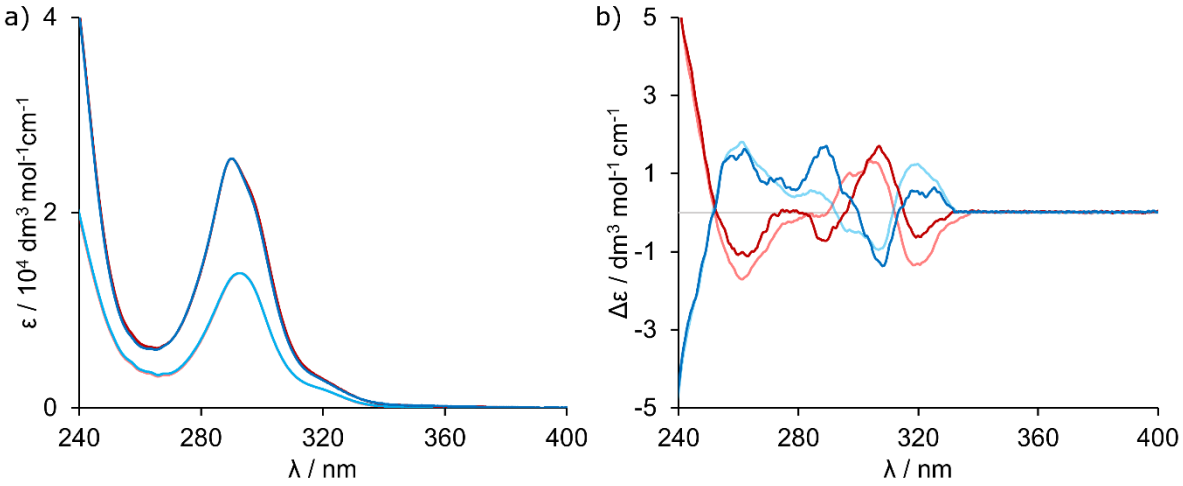

Figure S63. a) UV spectra of (*R*)-**2** (**light red**), (*S*)-**2** (**light blue**) and complexes **1** + (*R*)-**2** (**red**), **1** + (*S*)-**2** (**blue**) in CH_3OH; b) ECD spectra of (*R*)-**2** (**light red**), (*S*)-**2** (**light blue**) and complexes **1** + (*R*)-**2** (**red**), **1** + (*S*)-**2** (**blue**) in CH_3OH. The intensities of cavitand spectra were normalized by using the UV band at 290 nm of sample (*R*)-**2** whereas intensities of capsules spectra were normalized by using the UV band at 308 nm of sample **1** + (*R*)-**2**. The intensities of capsules spectra were multiplied by 2.

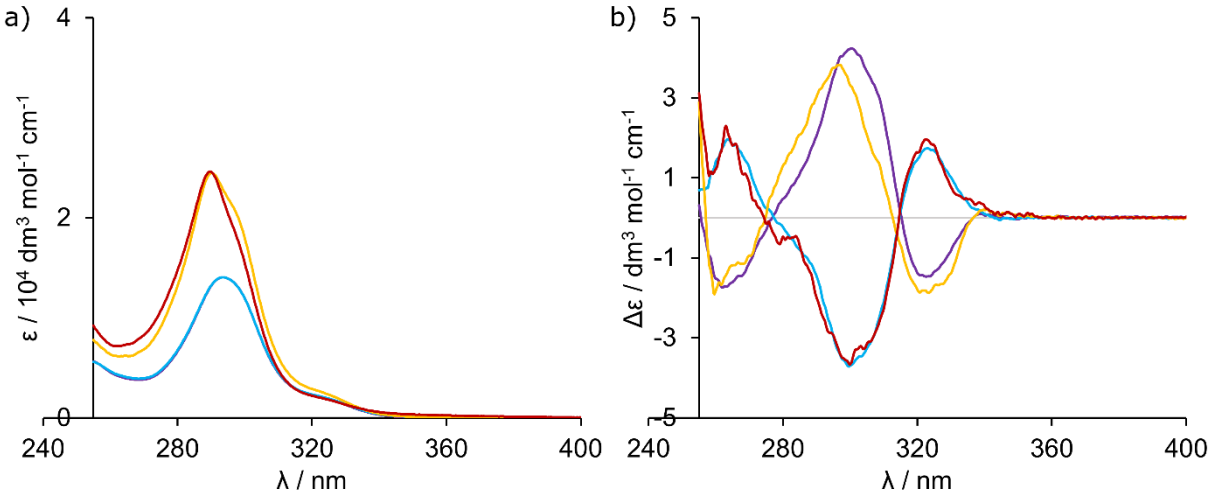

Figure S64. a) UV spectra of (*R*)-**2** (**violent**), (*S*)-**2** (**light blue**) and complexes **1** + (*R*)-**2** (**yellow**), **1** + (*S*)-**2** (**red**) in DMSO; b) ECD spectra of (*R*)-**2** (**violent**), (*S*)-**2** (**light blue**) and complexes **1** + (*R*)-**2** (**yellow**), **1** + (*S*)-**2** (**red**) in DMSO. The intensities of cavitand spectra were normalized by using the UV band at 293 nm of sample (*R*)-**2** whereas intensities of capsules spectra were normalized by using the UV band at 290 nm of sample **1** + (*R*)-**2**. The intensities of capsules spectra were multiplied by 2.

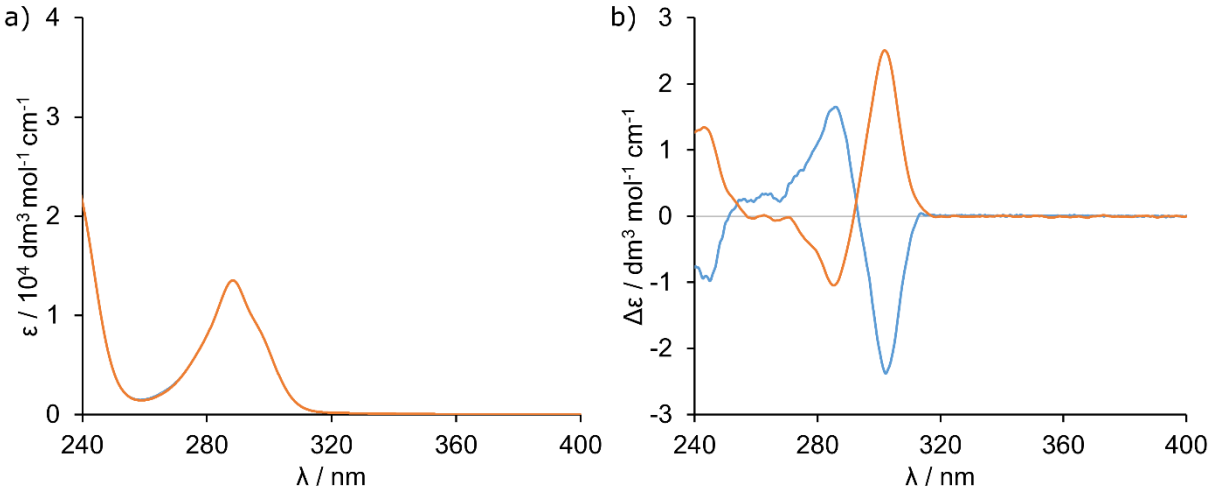

Figure S65. a) UV spectra of **1**(L–LysOMe)$_2$ (**orange**) and **1**(D–LysOMe)$_2$ (**blue**) in CH_3OH; b) ECD spectra of **1**(L–LysOMe)$_2$ (**orange**) and **1**(D–LysOMe)$_2$ (**blue**) in CH_3OH. The intensities of all spectra were normalized by using the UV band at 296 nm of sample **1**(L–LysOMe)$_2$.

3. ESIMS spectrum

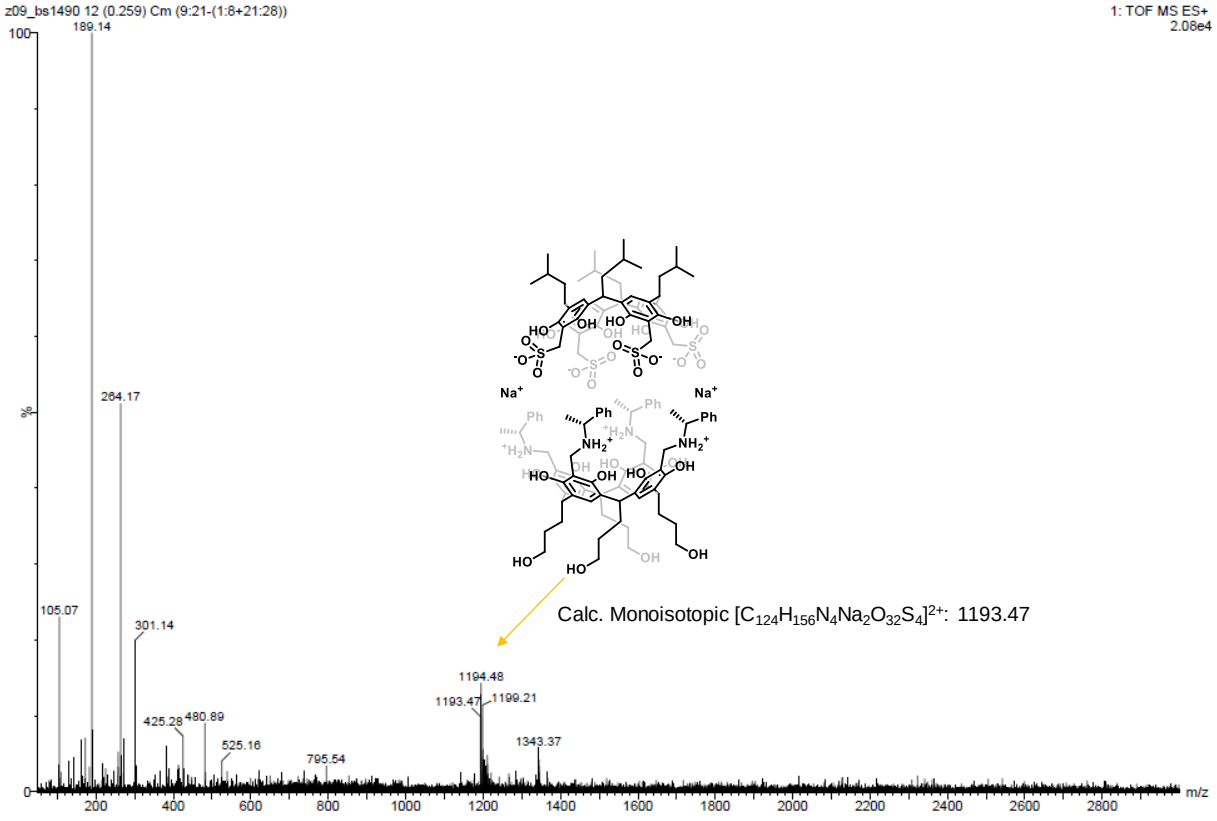

Figure 66S. ESIMS spectrum of **1** + (*R*)-**2**.