Electronic Supplementary Material (ESI) for RSC Advances.

Isohexide hydroxy esters: synthesis and application of a new class of biobased AB-type building blocks

Shanmugam Thiyagarajan,[a,b] Jing Wu,[a,b] Rutger J. I. Knoop,[a] Jacco van Haveren,[a,b] Martin Lutz,[c] and Daan S. van Es*[a,b]

[a] Food & Bio-based Research, Wageningen University and Research Centre, P.O. Box 17, 6700 AA Wageningen, The Netherlands
Fax: +31 317 483011; Tel: +31 317 481160
E-mail: daan.vanes@wur.nl; shanmugam.thiyagarajan@wur.nl

[b] Dutch Polymer Institute, P.O. Box 902, 5600 AX, Eindhoven, The Netherlands

[c] Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584, Utrecht, The Netherlands

Supporting information:

Figure 12. ^{13}C NMR spectrum of (3S,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4b**) ($CDCl_3$)

Figure 13. ^{1}H NMR spectrum of (3R,6S)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4c**) ($CDCl_3$)

Figure 14. ^{13}C NMR spectrum of (3R,6S)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4c**) ($CDCl_3$)

Figure 15. ^{1}H NMR spectrum of (3R,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4d**) ($CDCl_3$)

Figure 16. ^{13}C NMR spectrum of (3R,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4d**) ($CDCl_3$)

Figure 17. ^{1}H NMR spectrum of (3S,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5a**) ($CDCl_3$)

Figure 18. ^{13}C NMR spectrum of (3S,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5a**) ($CDCl_3$)

Figure 19. 2D-COSY spectrum of (3S,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5a**) ($CDCl_3$)

Figure 20. ^{1}H NMR spectrum of (3R,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5b**) ($CDCl_3$)

Figure 21. ^{13}C NMR spectrum of (3R,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5b**) ($CDCl_3$)

Figure 22. 2D-COSY spectrum of (3R,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5b**) ($CDCl_3$)

Figure 23. ^{1}H NMR spectrum of (3S,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5c**) ($CDCl_3$)

Figure 24. ^{13}C NMR spectrum of (3S,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5c**) ($CDCl_3$)

Figure 25. 2D-COSY spectrum of (3S,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5c**) ($CDCl_3$)

Figure 26. ^{1}H NMR spectrum of (3R,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5d**) ($CDCl_3$)

Figure 27. ^{13}C NMR spectrum of (3R,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5d**) ($CDCl_3$)

Figure 28. 2D-COSY spectrum of (3R,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5d**) ($CDCl_3$)

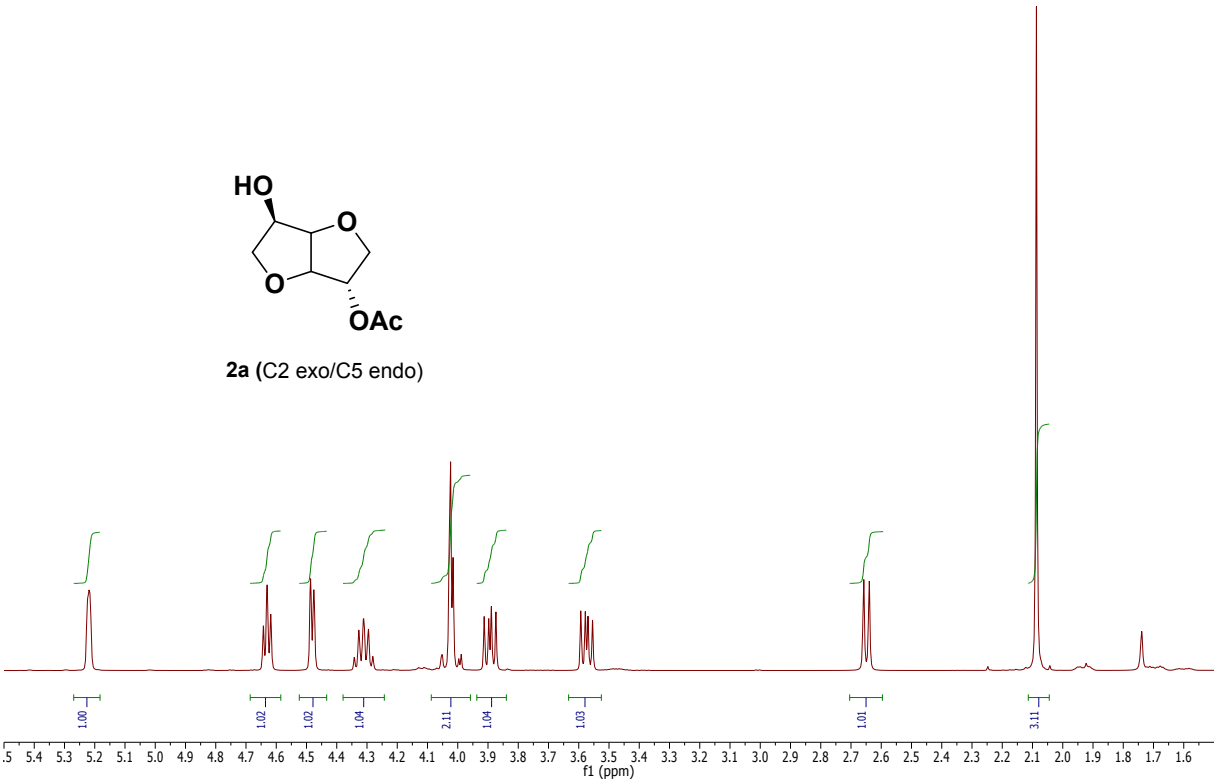

Figure 1. ^{1}H NMR spectrum of (3S,6R)-6-hydroxyhexahydrofuro[3,2-b]furan-3-yl acetate (**2a**) ($CDCl_3$)

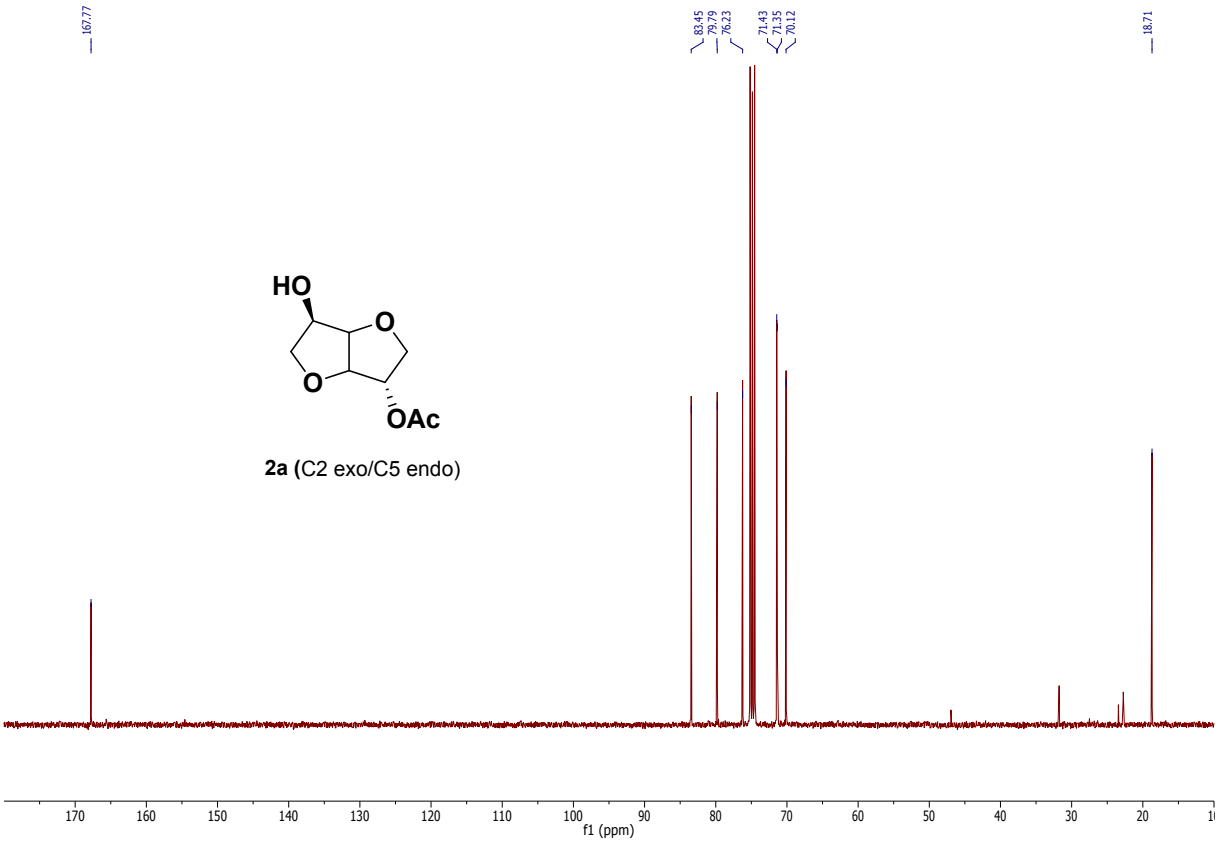

Figure 2. ^{13}C NMR spectrum of (3S,6R)-6-hydroxyhexahydrofuro[3,2-b]furan-3-yl acetate (**2a**) ($CDCl_3$)

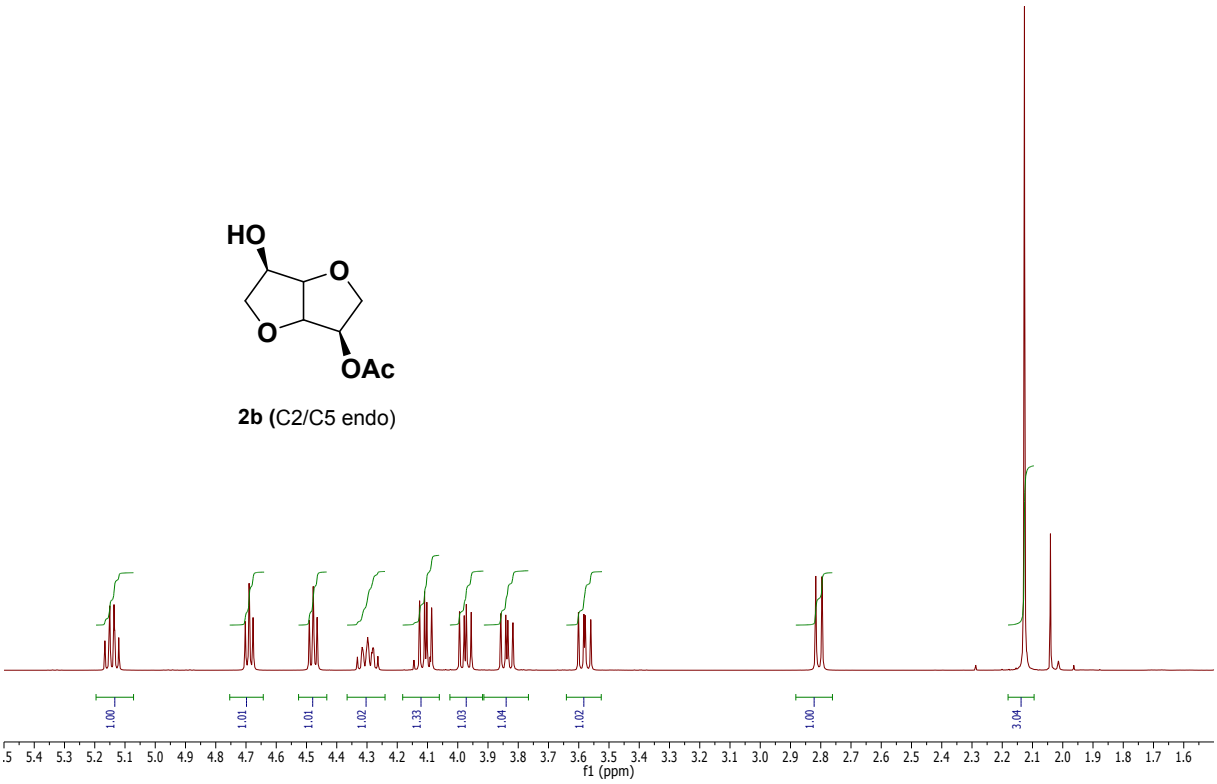

Figure 3. ^{1}H NMR spectrum of (3R,6R)-6-hydroxyhexahydrofuro[3,2-b]furan-3-yl acetate (**2b**) ($CDCl_3$)

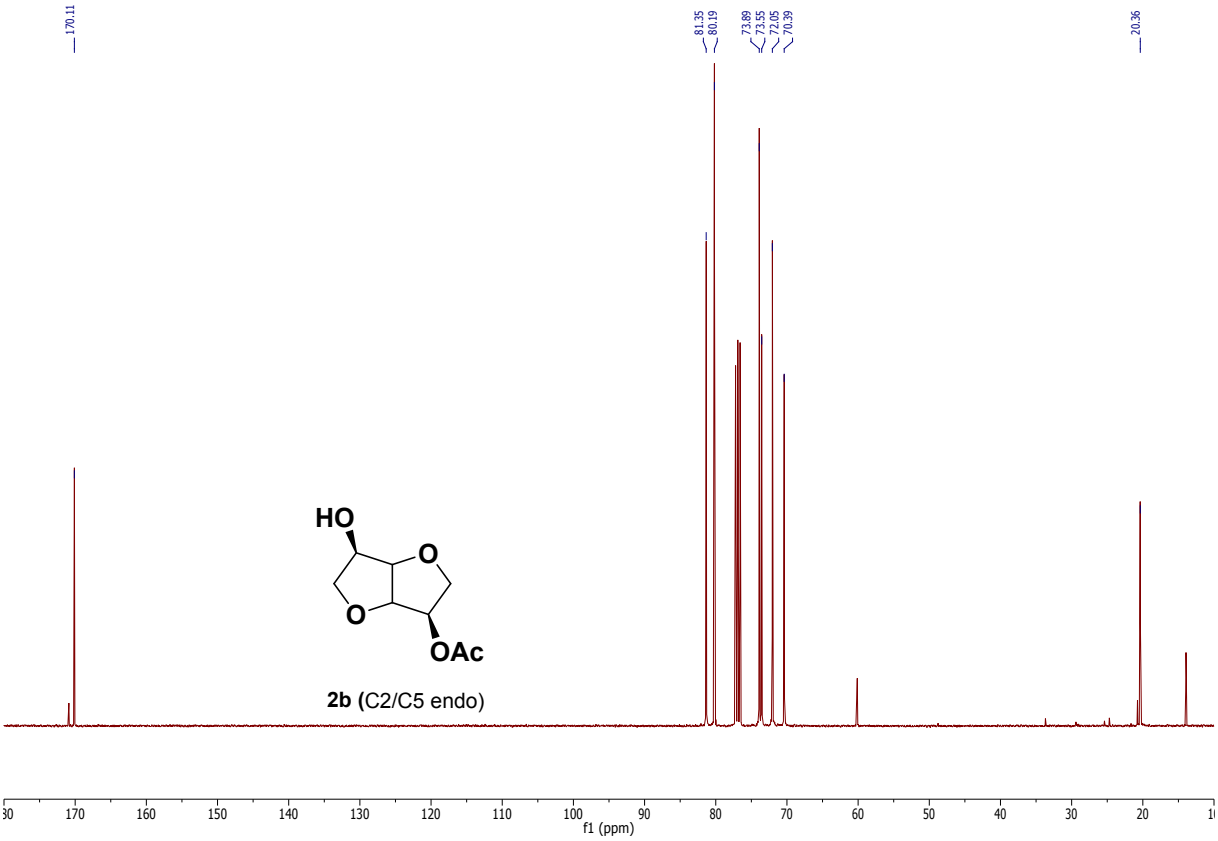

Figure 4. ^{13}C NMR spectrum of (3R,6R)-6-hydroxyhexahydrofuro[3,2-b]furan-3-yl acetate (**2b**) ($CDCl_3$)

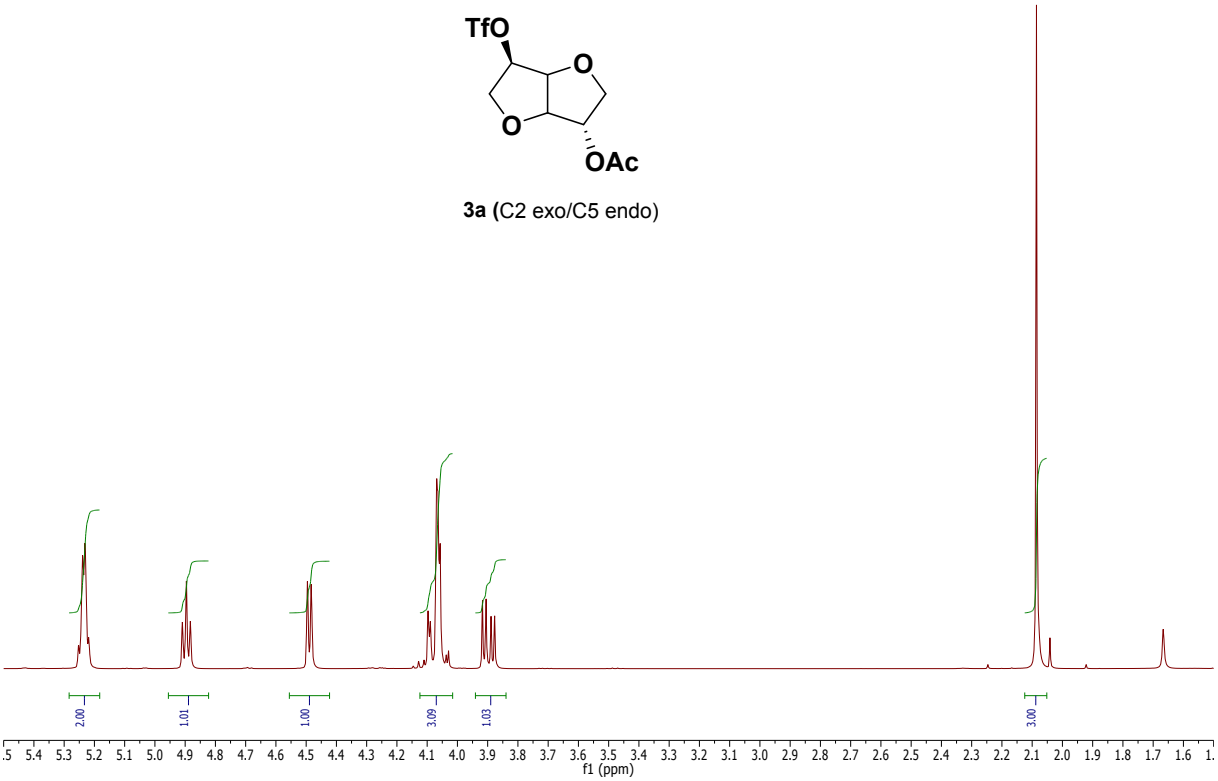

Figure 5. ^{1}H NMR spectrum of (3S,6R)-6-(((trifluoromethyl)sulfonyl)oxy)hexahydrofuro[3,2-b]furan-3-yl acetate (**3a**) ($CDCl_3$)

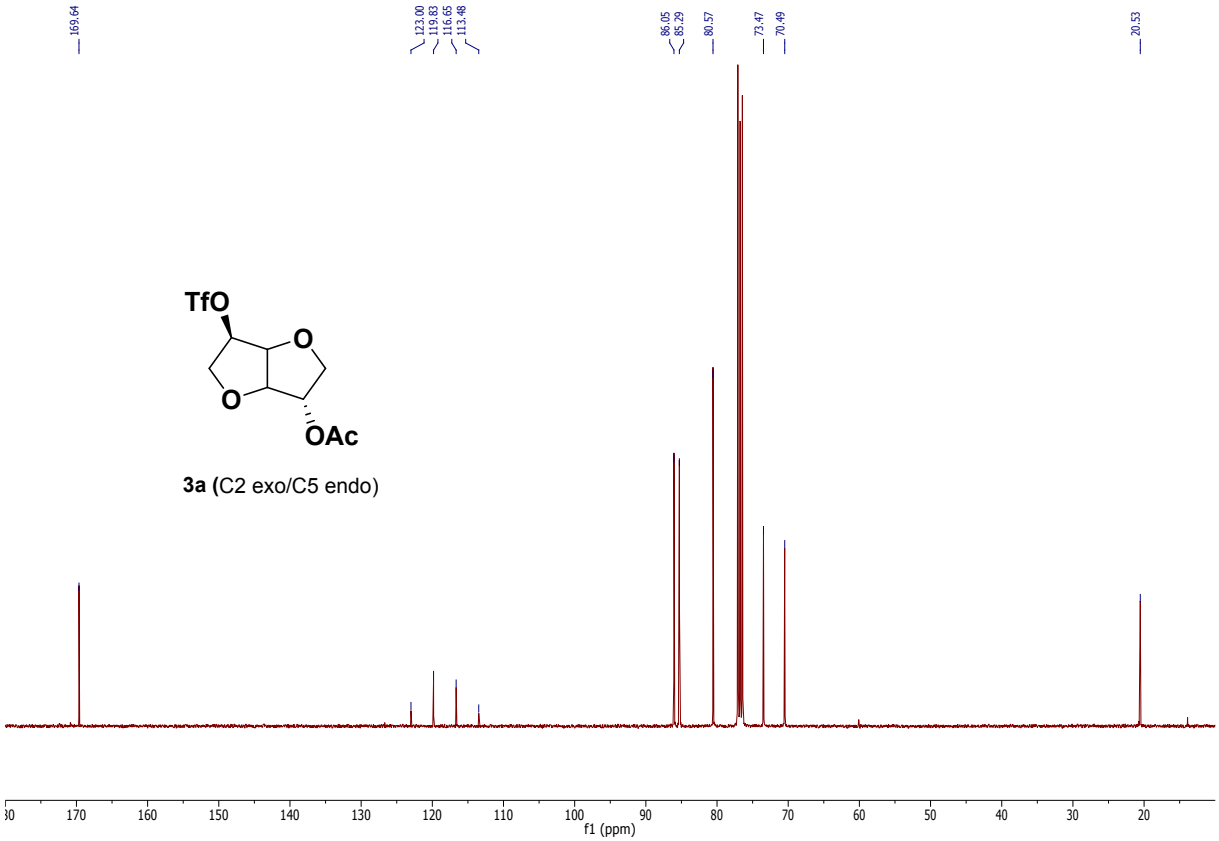

Figure 6. ^{13}C NMR spectrum of (3S,6R)-6-(((trifluoromethyl)sulfonyl)oxy)hexahydrofuro[3,2-b]furan-3-yl acetate (**3a**) ($CDCl_3$)

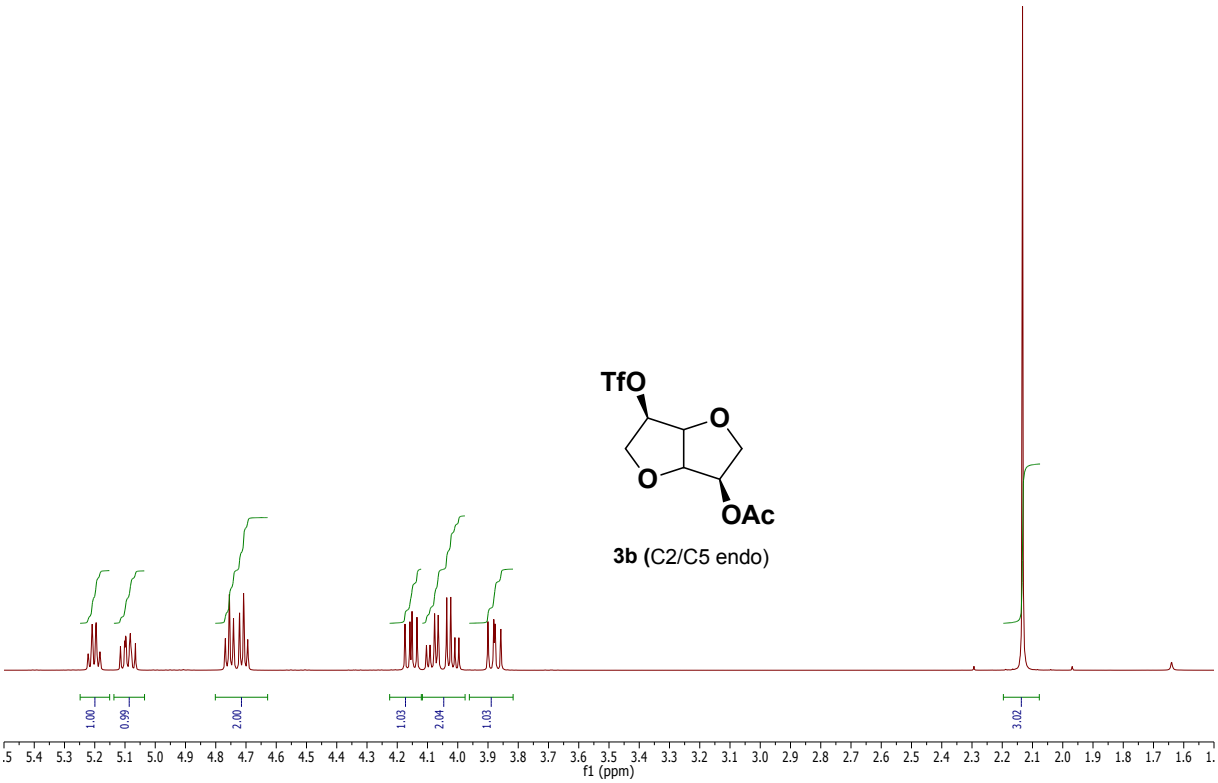

Figure 7. ^{1}H NMR spectrum of (3R,6R)-6-(((trifluoromethyl)sulfonyl)oxy)hexahydrofuro[3,2-b]furan-3-yl acetate (**3b**) ($CDCl_3$)

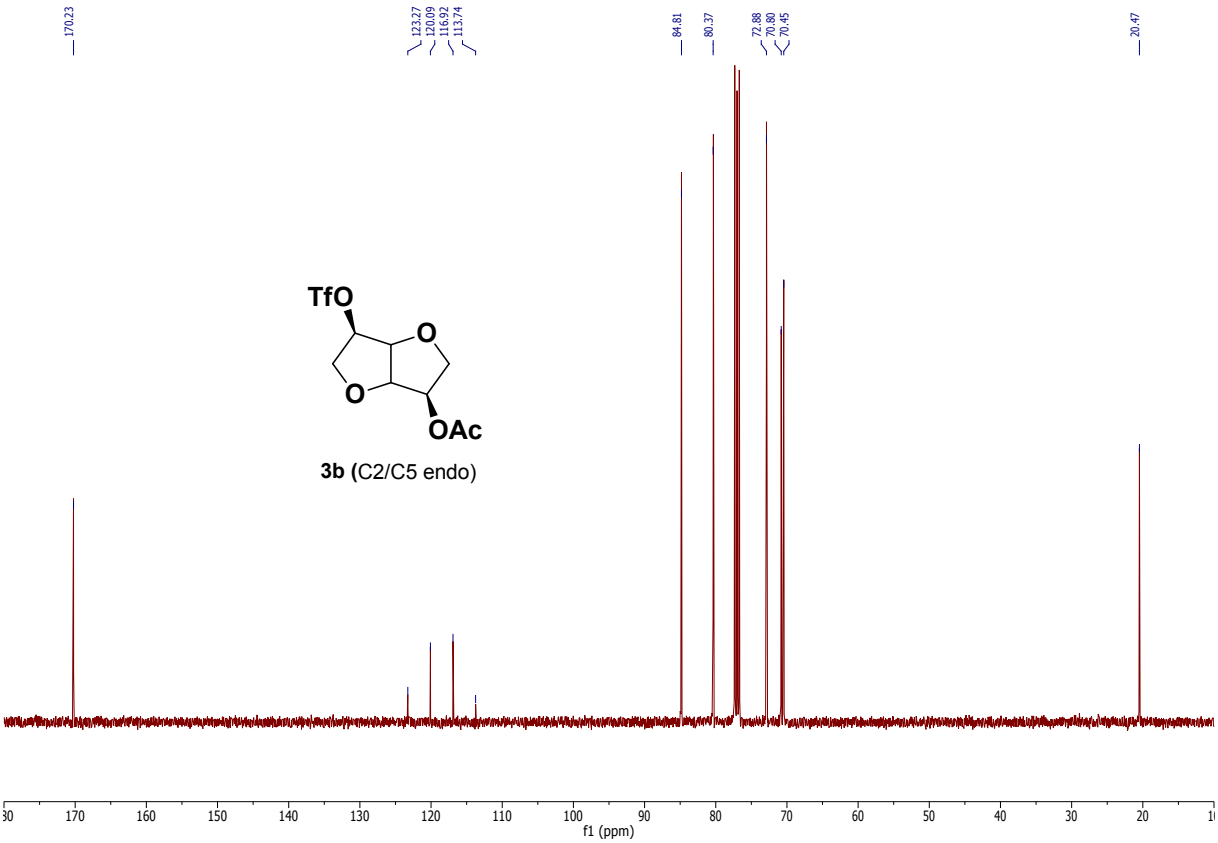

Figure 8. ^{13}C NMR spectrum of (3R,6R)-6-(((trifluoromethyl)sulfonyl)oxy)hexahydrofuro[3,2-b]furan-3-yl acetate (**3b**) ($CDCl_3$)

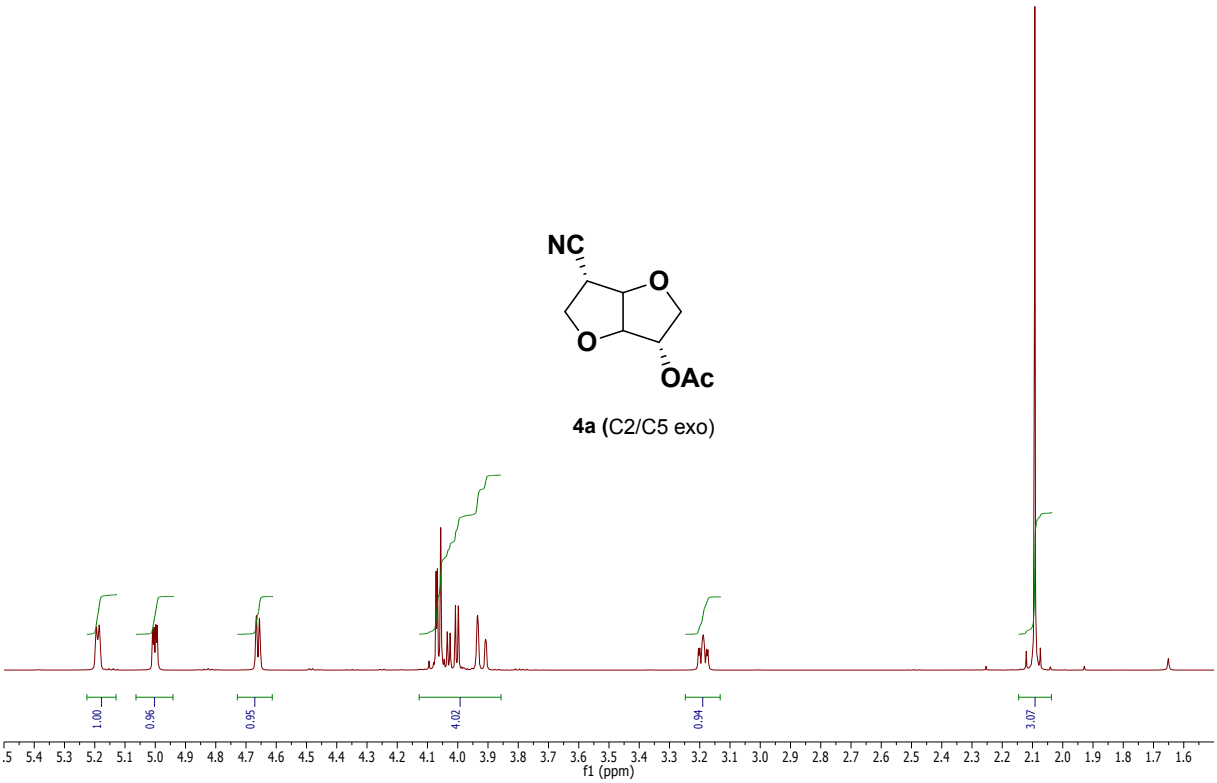

Figure 9. ^{1}H NMR spectrum of (3S,6S)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4a**) ($CDCl_3$)

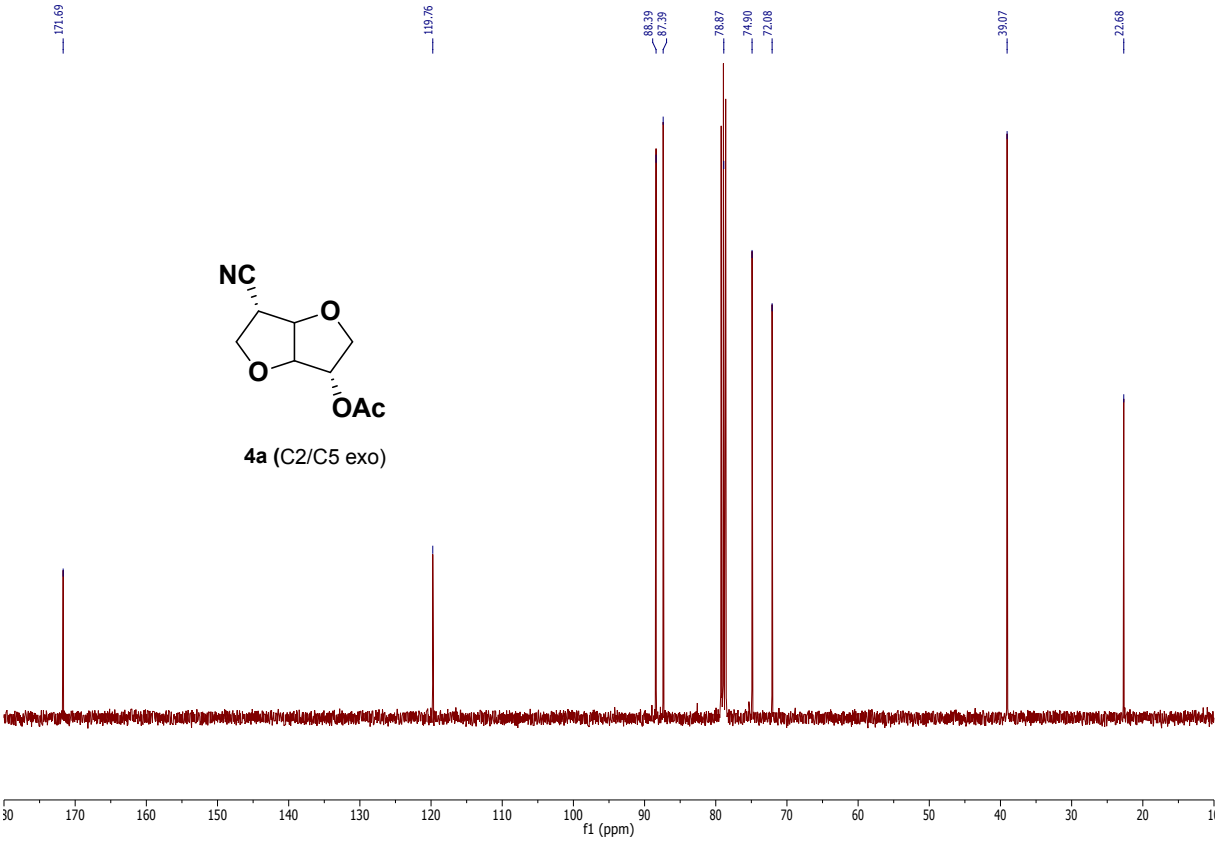

Figure 10. ^{13}C NMR spectrum of (3S,6S)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4a**) ($CDCl_3$)

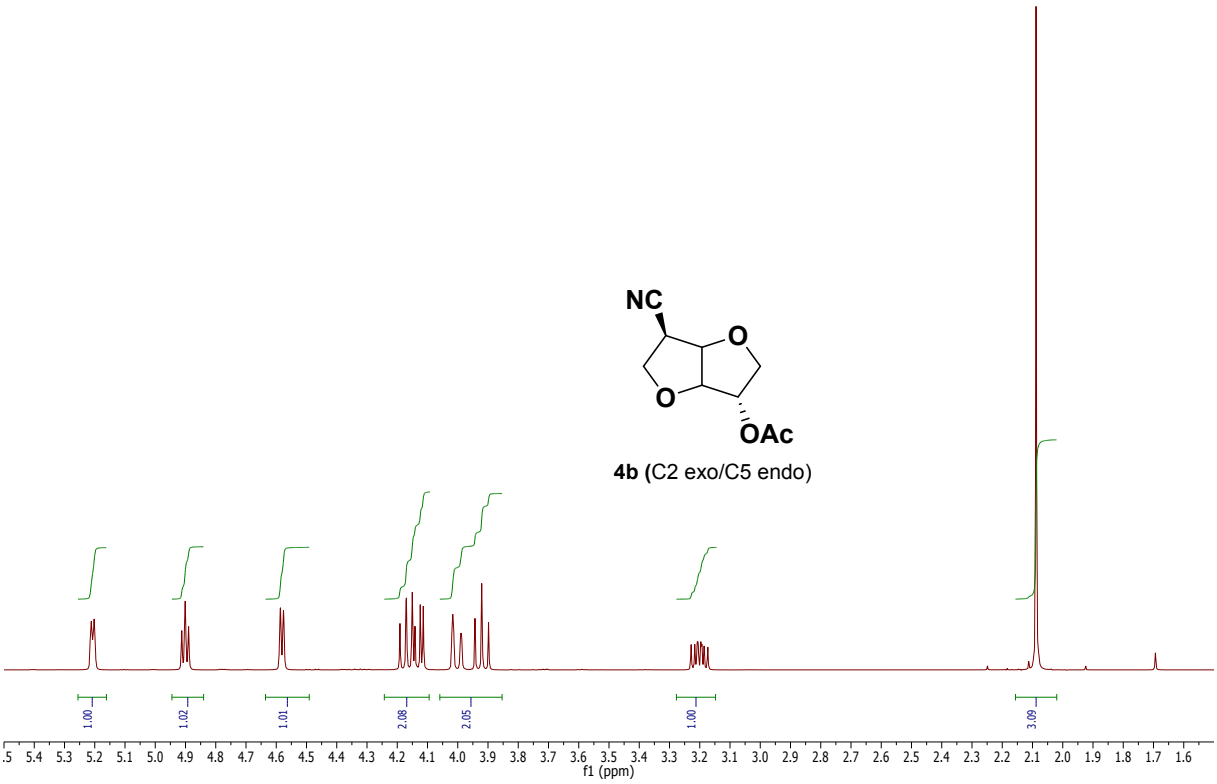

Figure 11. ^{1}H NMR spectrum of (3S,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4b**) ($CDCl_3$)

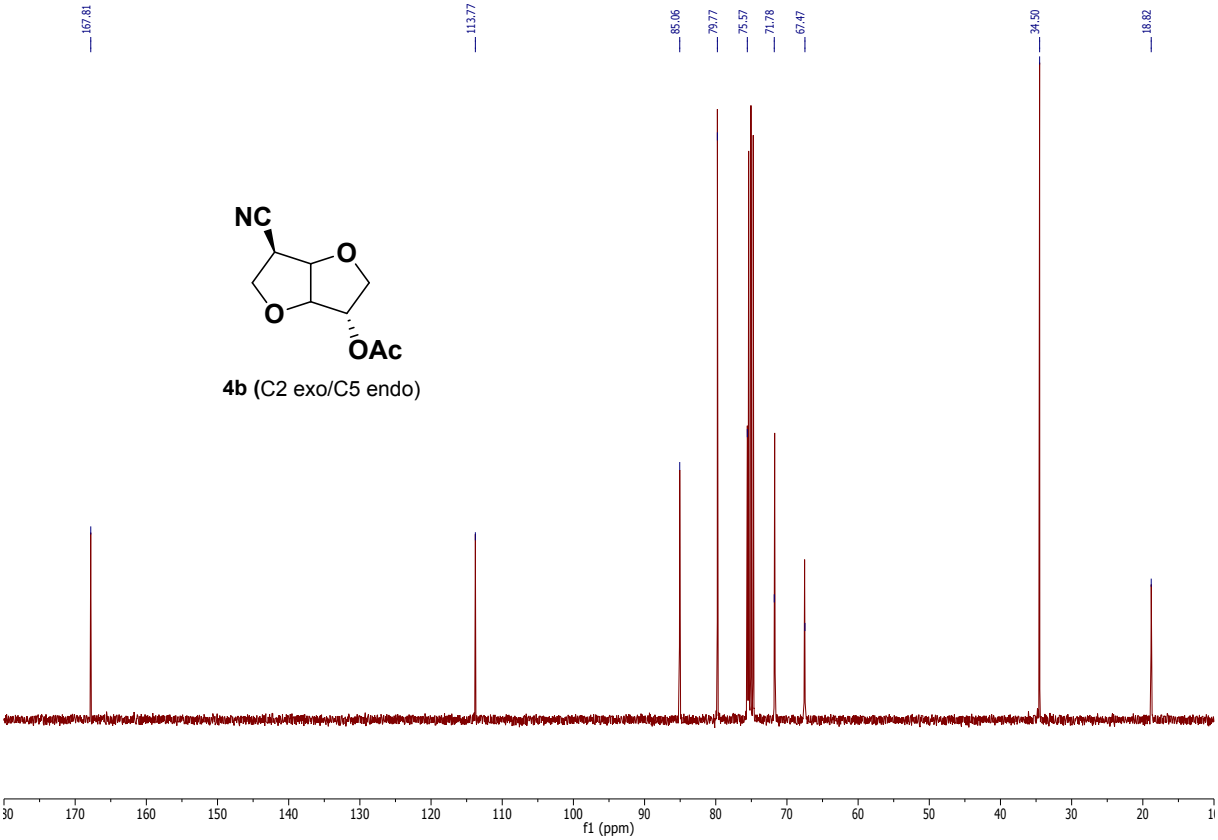

Figure 12. ^{13}C NMR spectrum of (3S,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4b**) ($CDCl_3$)

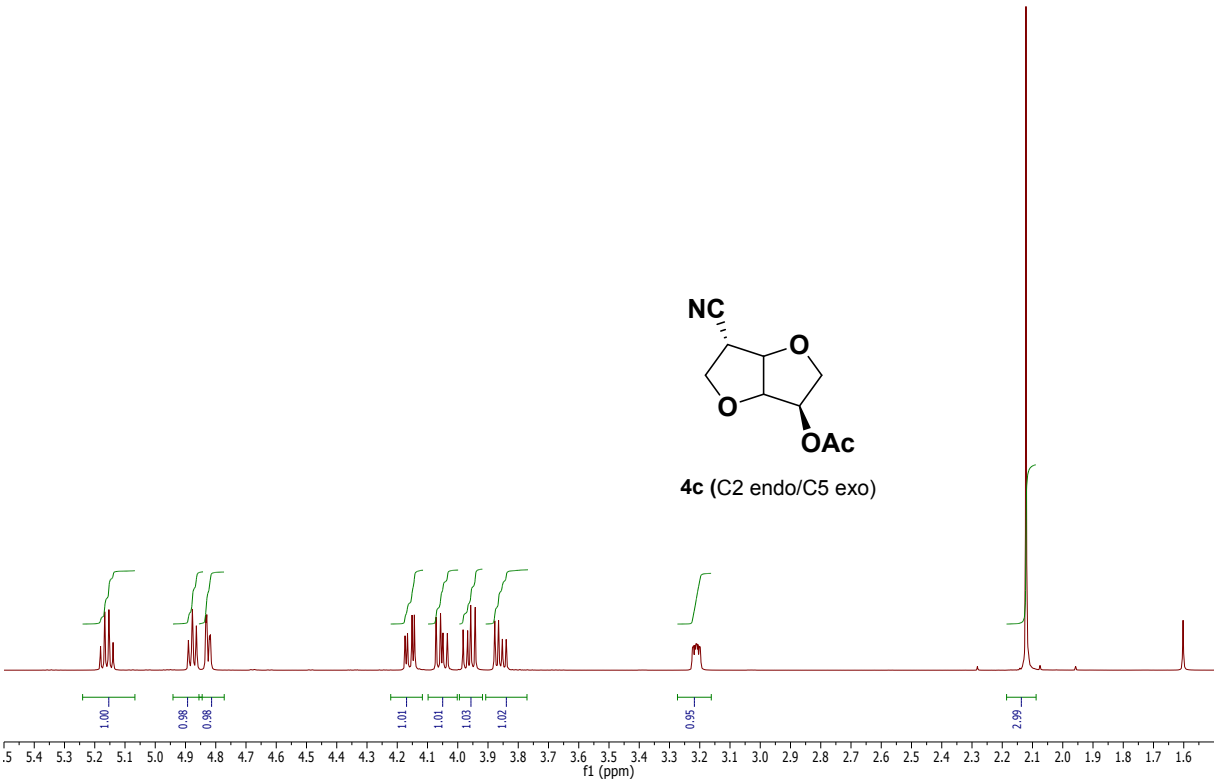

Figure 13. ^{1}H NMR spectrum of (3R,6S)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4c**) ($CDCl_3$)

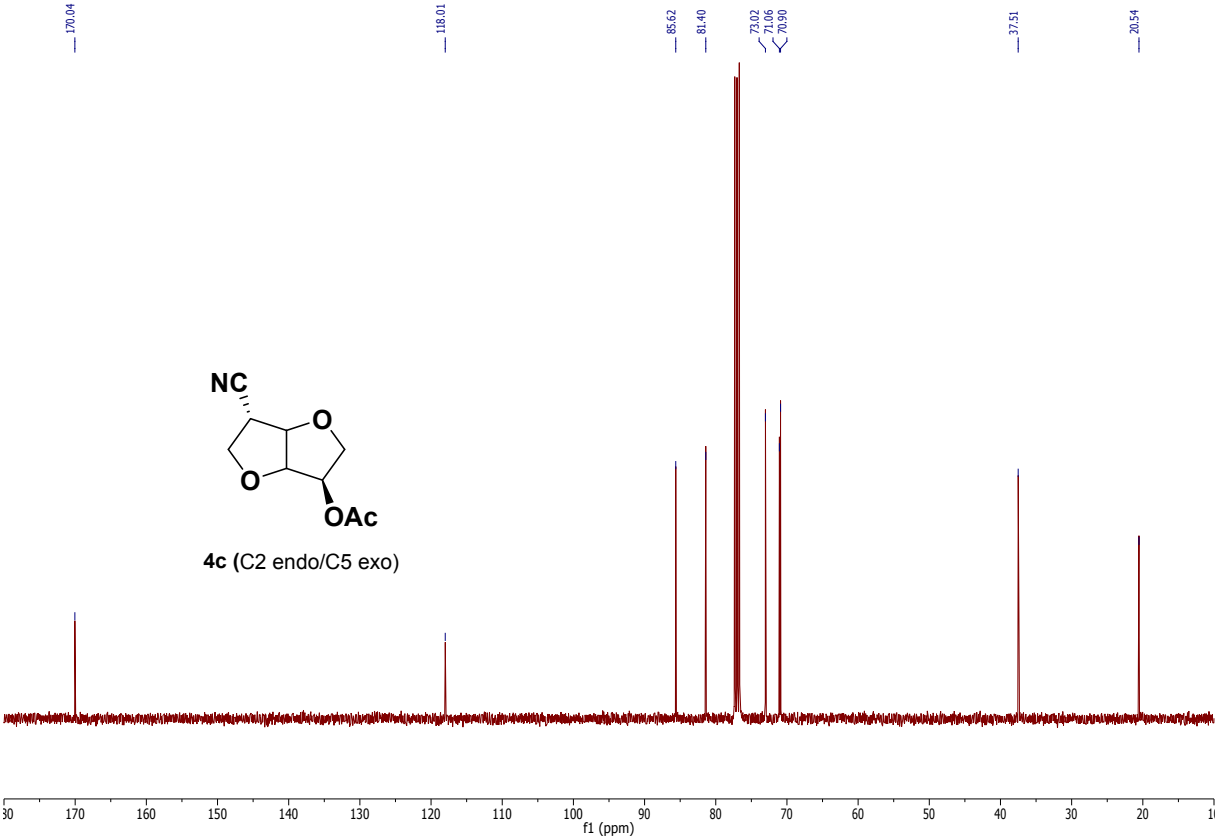

Figure 14. ^{13}C NMR spectrum of (3R,6S)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4c**) ($CDCl_3$)

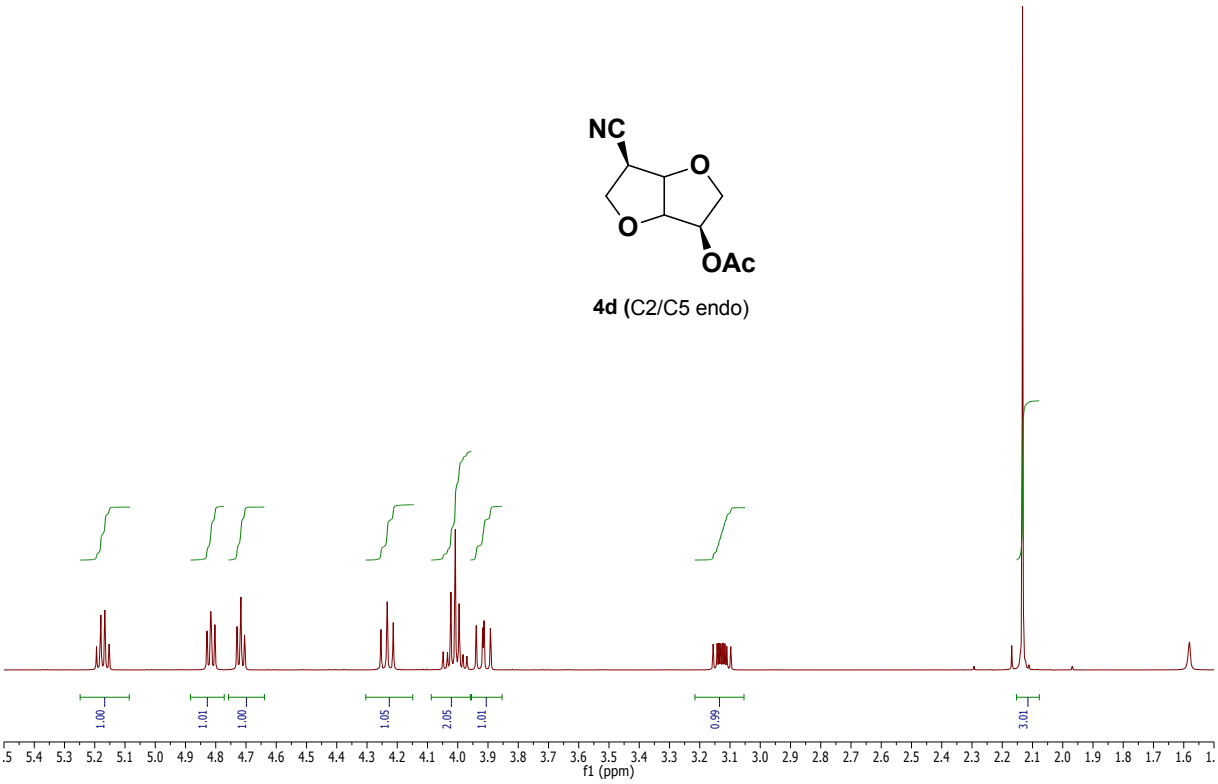

Figure 15. ^{1}H NMR spectrum of (3R,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4d**) ($CDCl_3$)

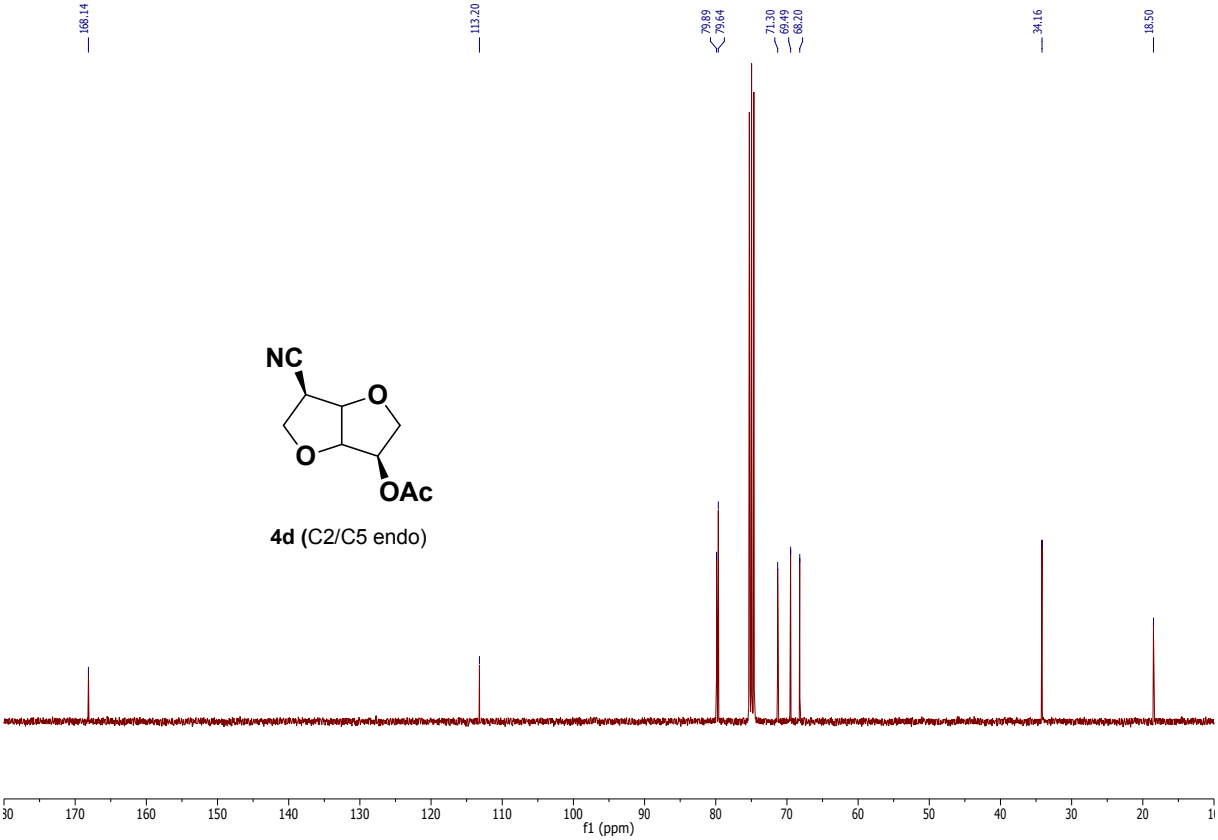

Figure 16. ^{13}C NMR spectrum of (3R,6R)-6-cyanohexahydrofuro[3,2-b]furan-3-yl acetate (**4d**) ($CDCl_3$)

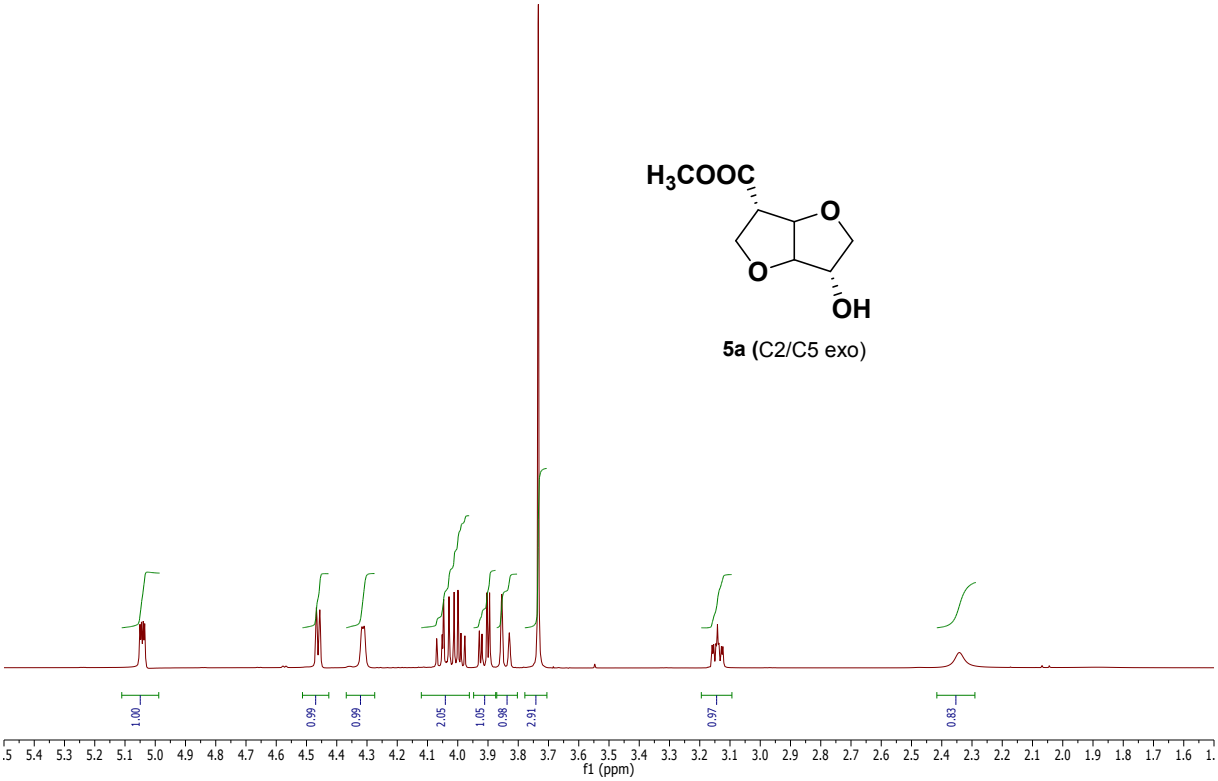

Figure 17. ^{1}H NMR spectrum of (3S,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5a**) ($CDCl_3$)

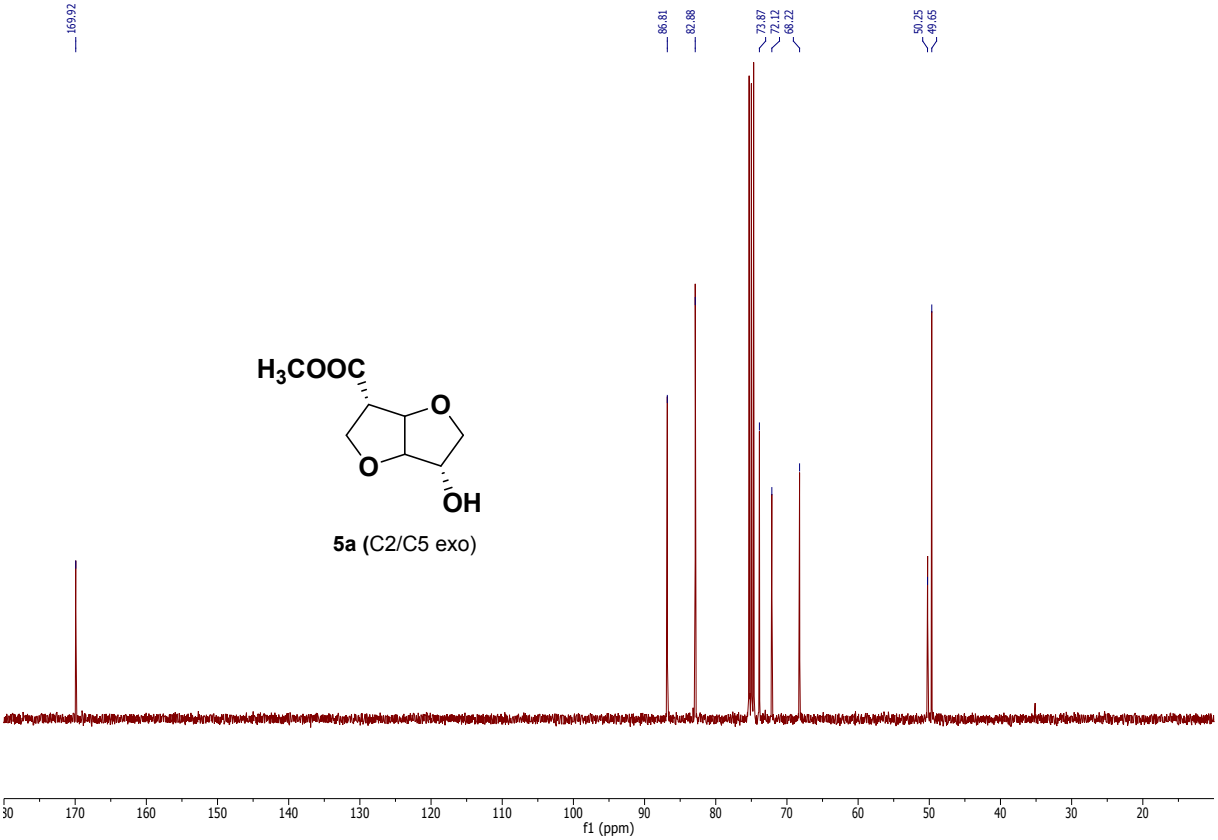

Figure 18. ^{13}C NMR spectrum of (3S,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5a**) ($CDCl_3$)

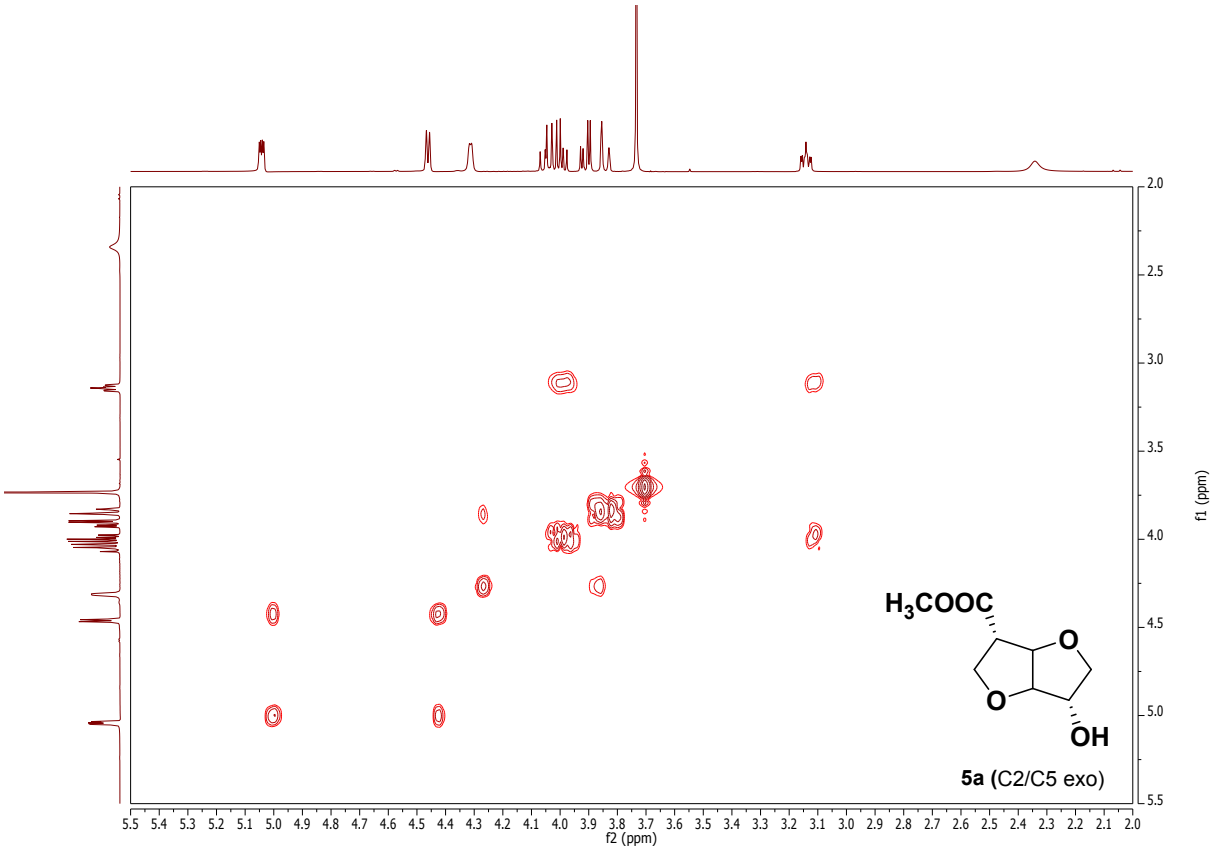

Figure 19. 2D-COSY spectrum of (3S,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5a**) ($CDCl_3$)

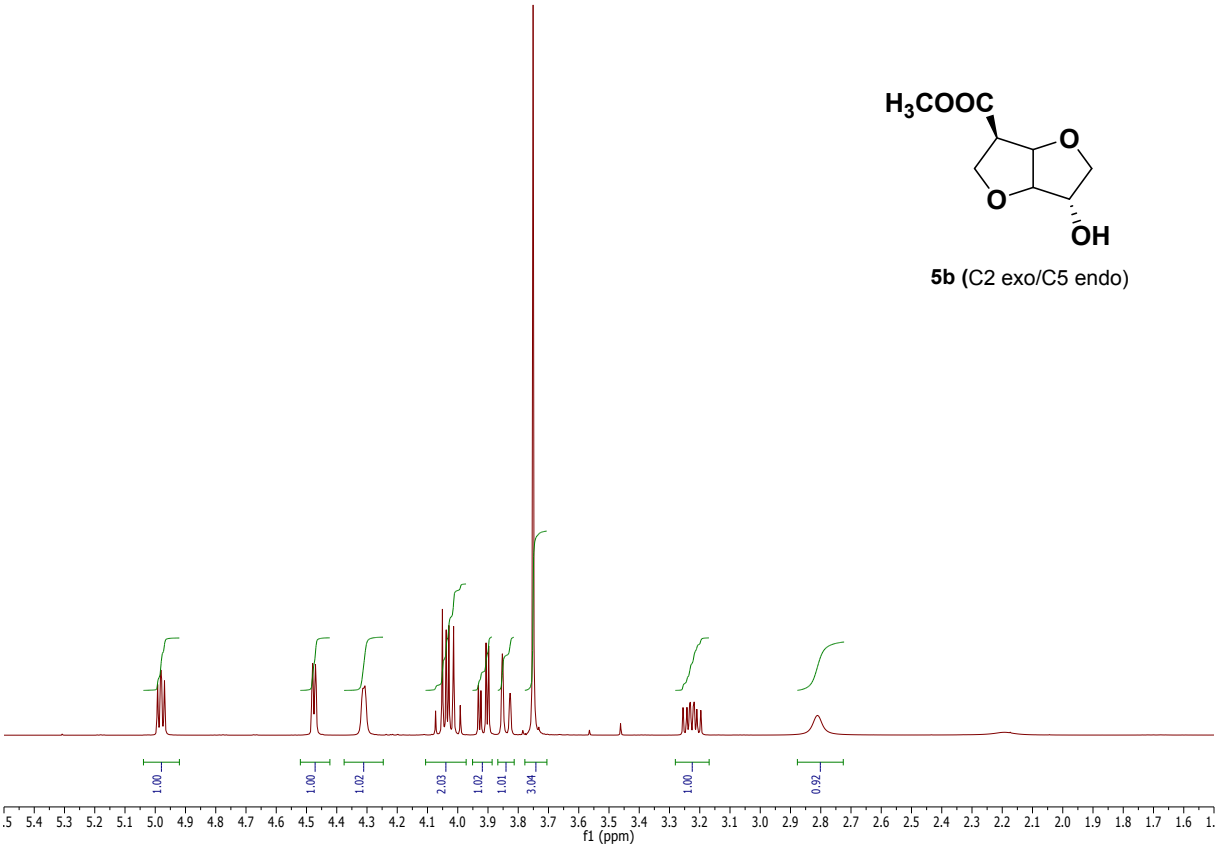

Figure 20. ^1H NMR spectrum of (3R,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5b**) ($CDCl_3$)

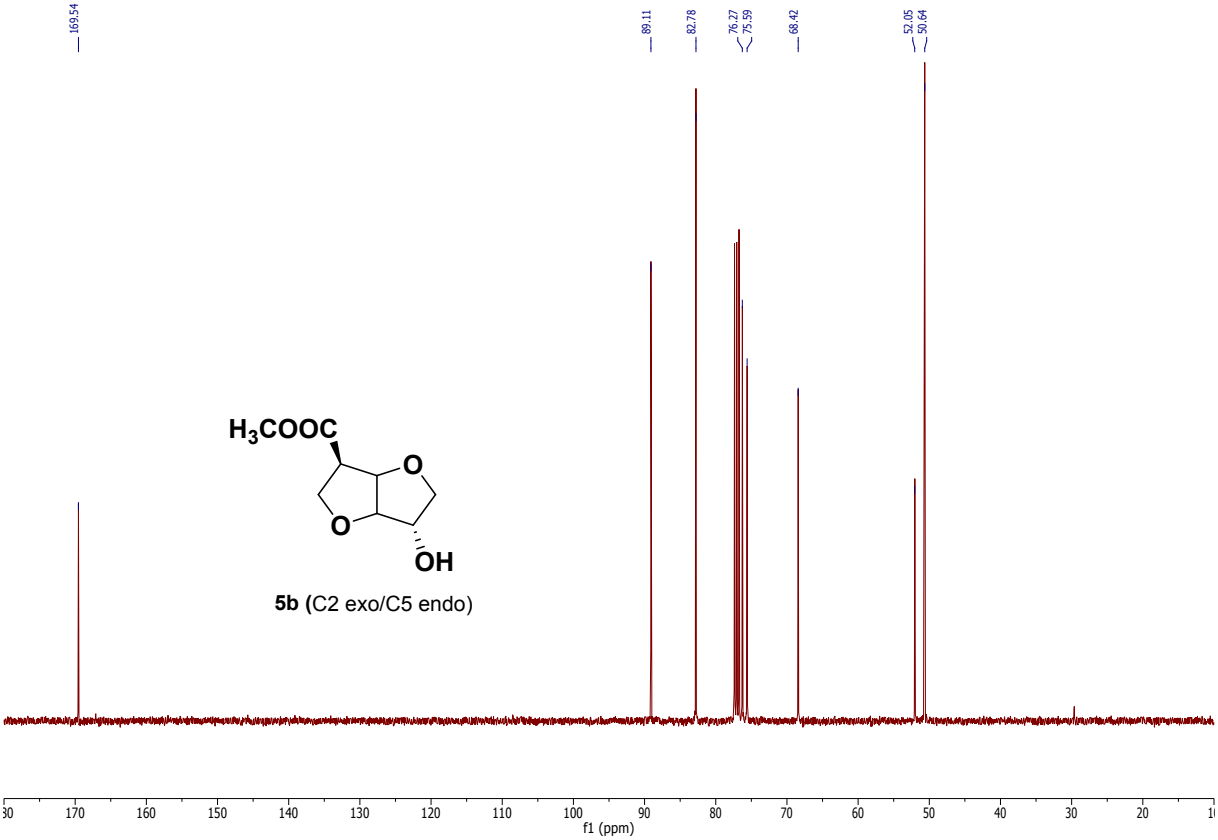

Figure 21. ^{13}C NMR spectrum of (3R,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5b**) ($CDCl_3$)

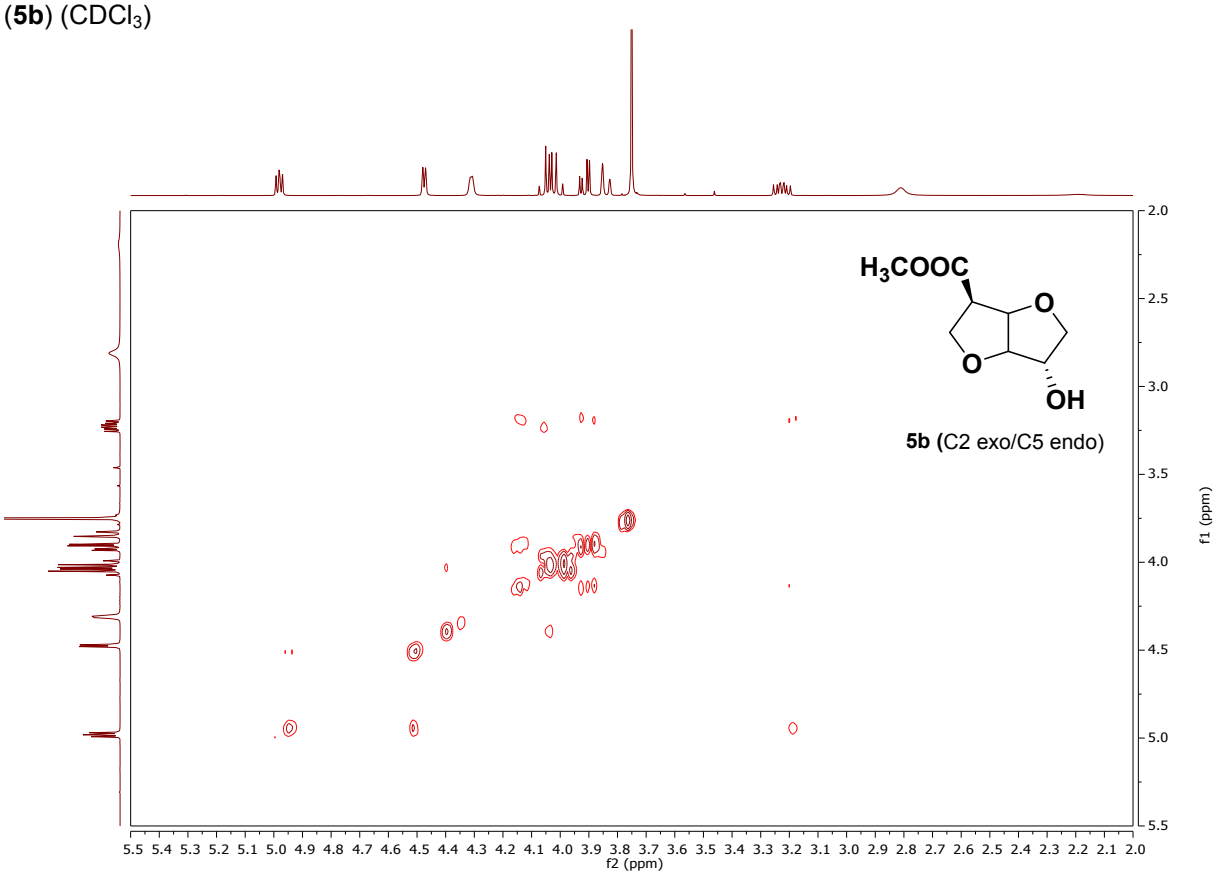

Figure 22. 2D-COSY spectrum of (3R,6S)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5b**) ($CDCl_3$)

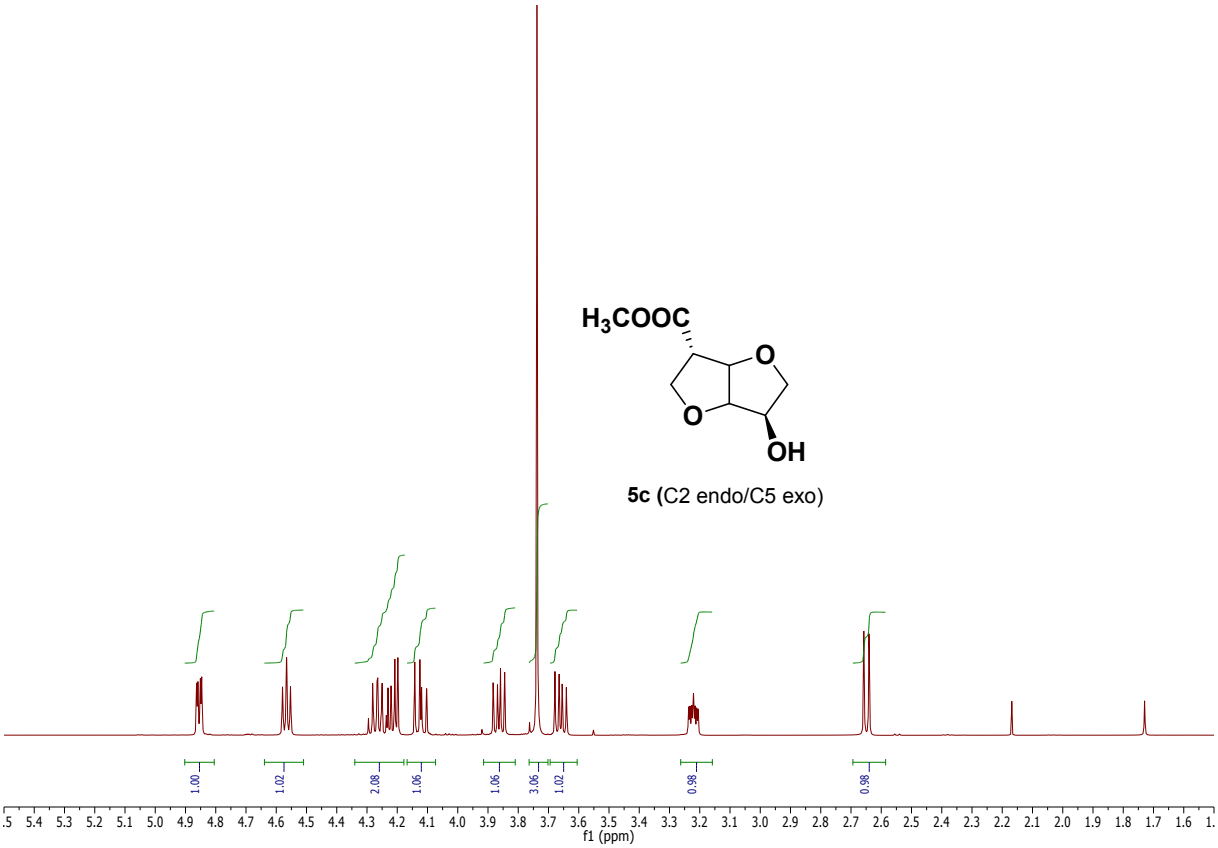

Figure 23. ^{1}H NMR spectrum of (3S,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5c**) ($CDCl_3$)

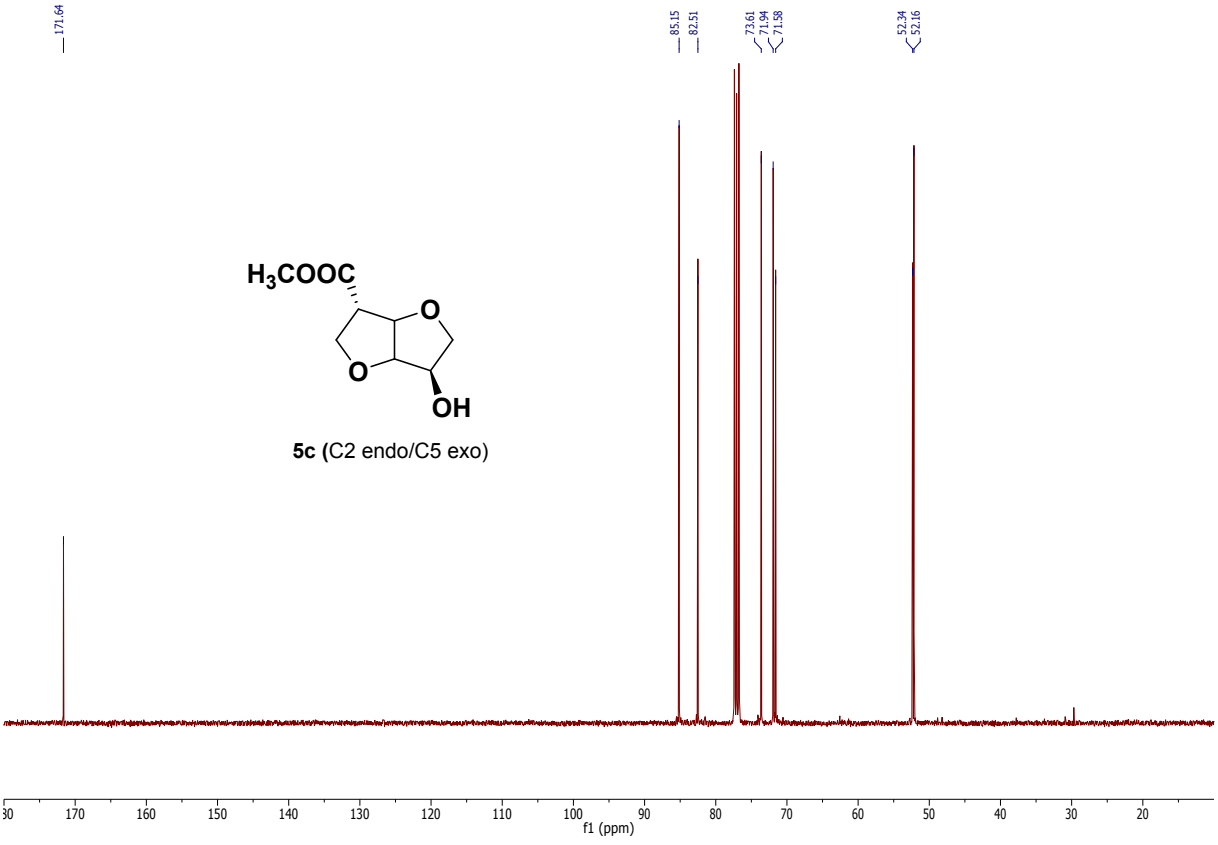

Figure 24. ^{13}C NMR spectrum of (3S,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5c**) ($CDCl_3$)

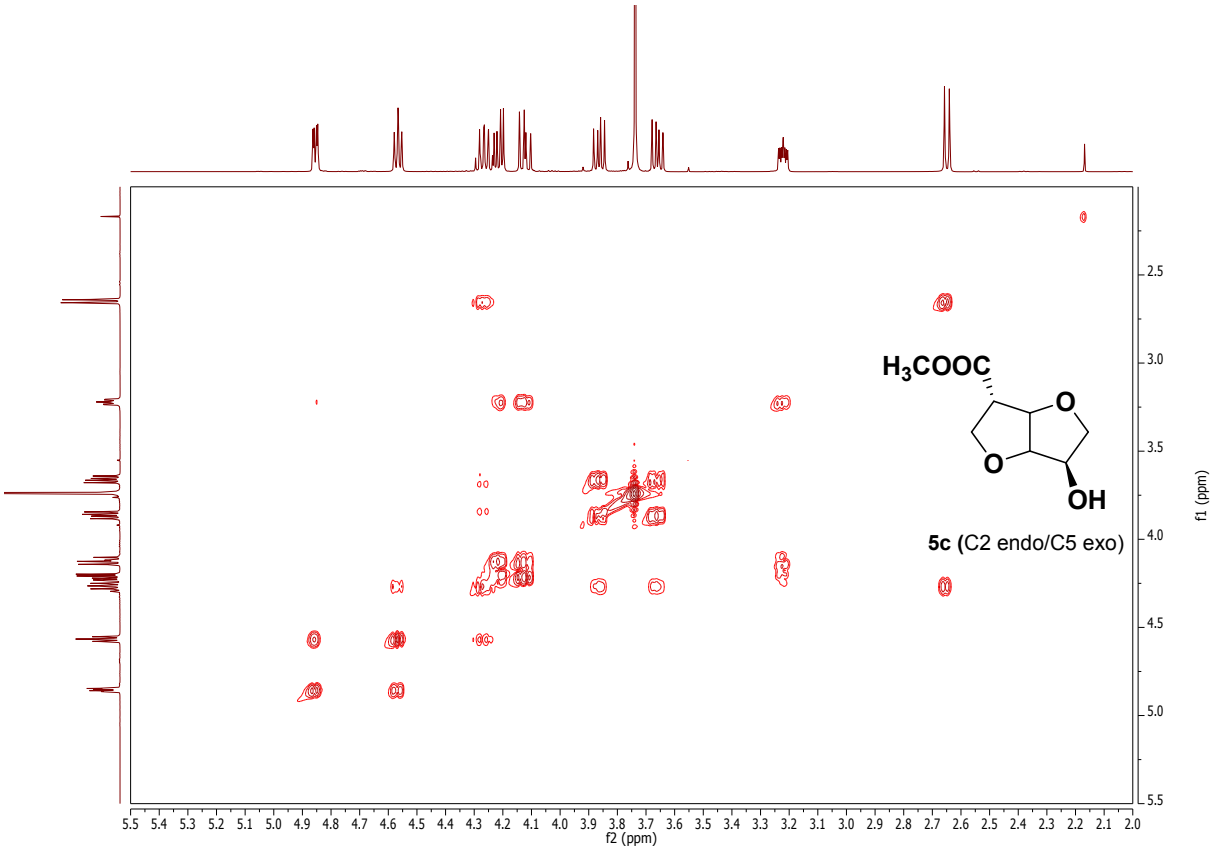

Figure 25. 2D-COSY spectrum of (3S,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5c**) ($CDCl_3$)

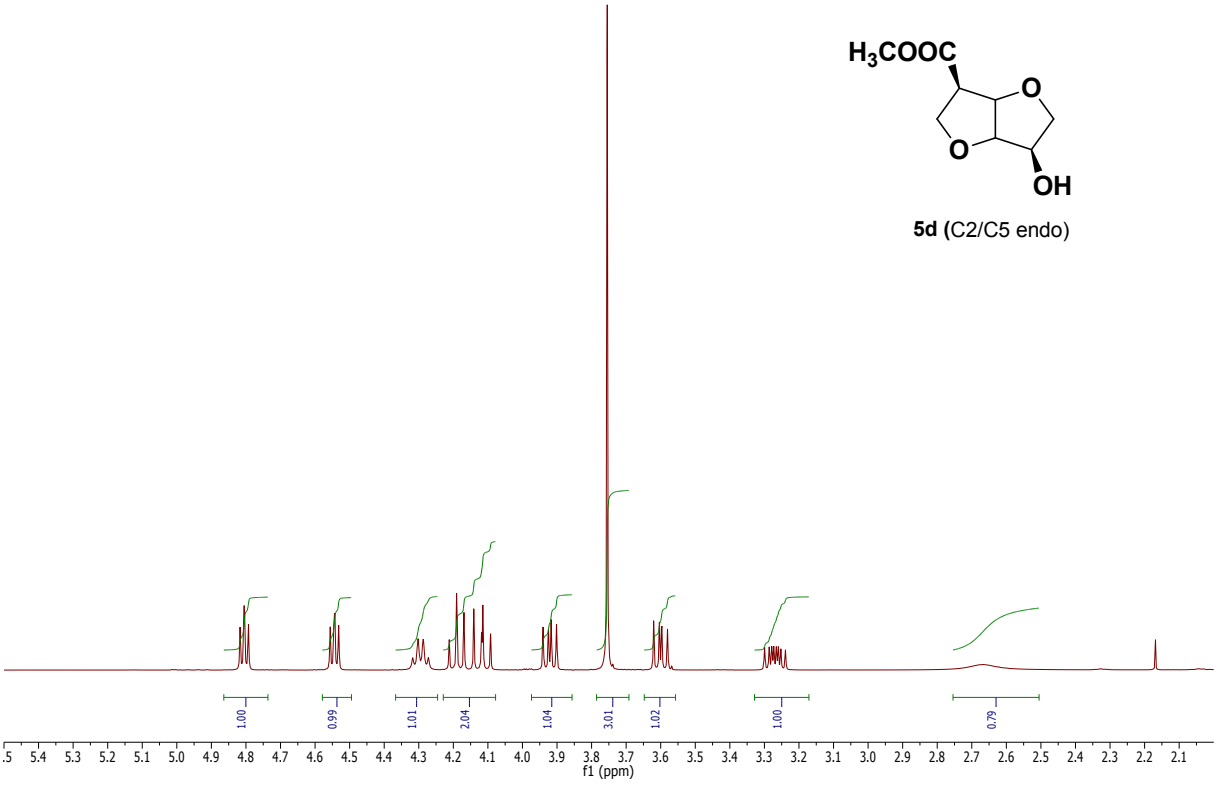

Figure 26. ^{1}H NMR spectrum of (3R,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5d**) ($CDCl_3$)

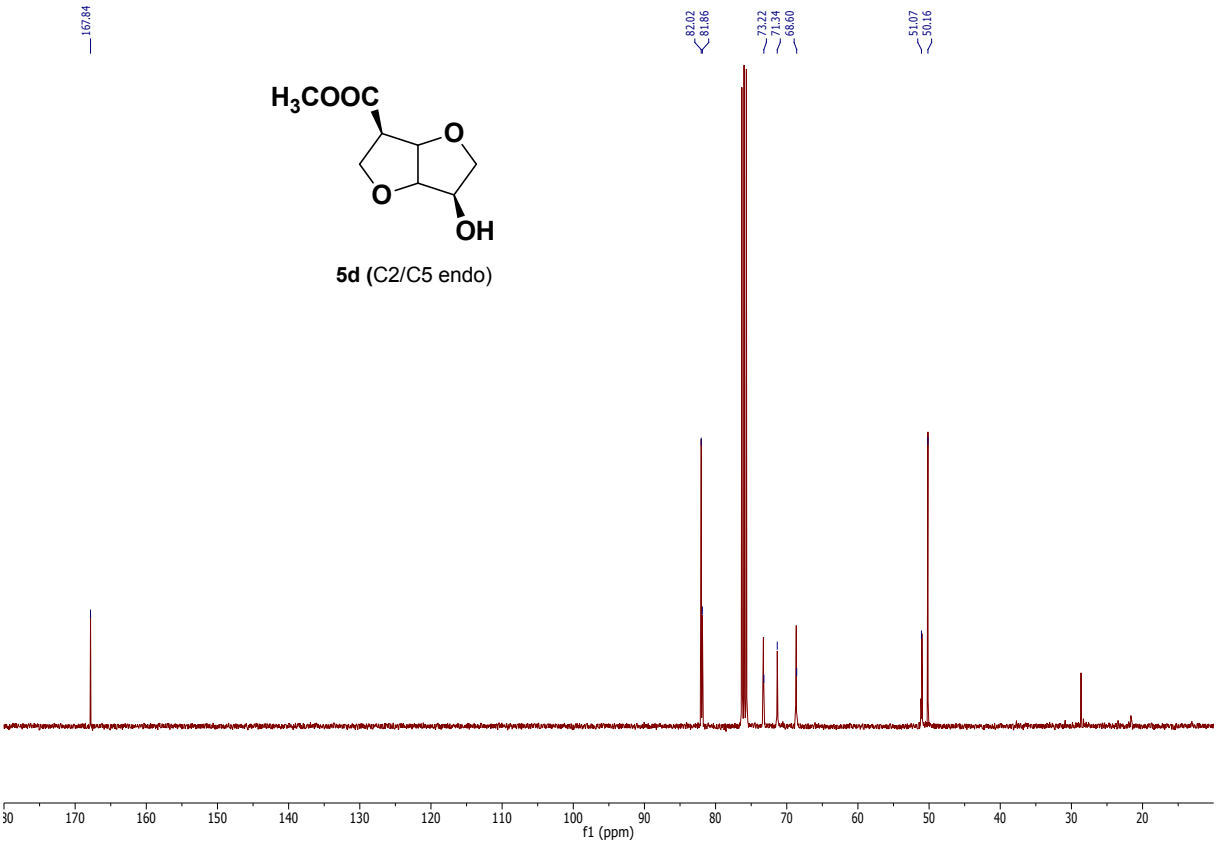

Figure 27. ^{13}C NMR spectrum of (3R,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5d**) ($CDCl_3$)

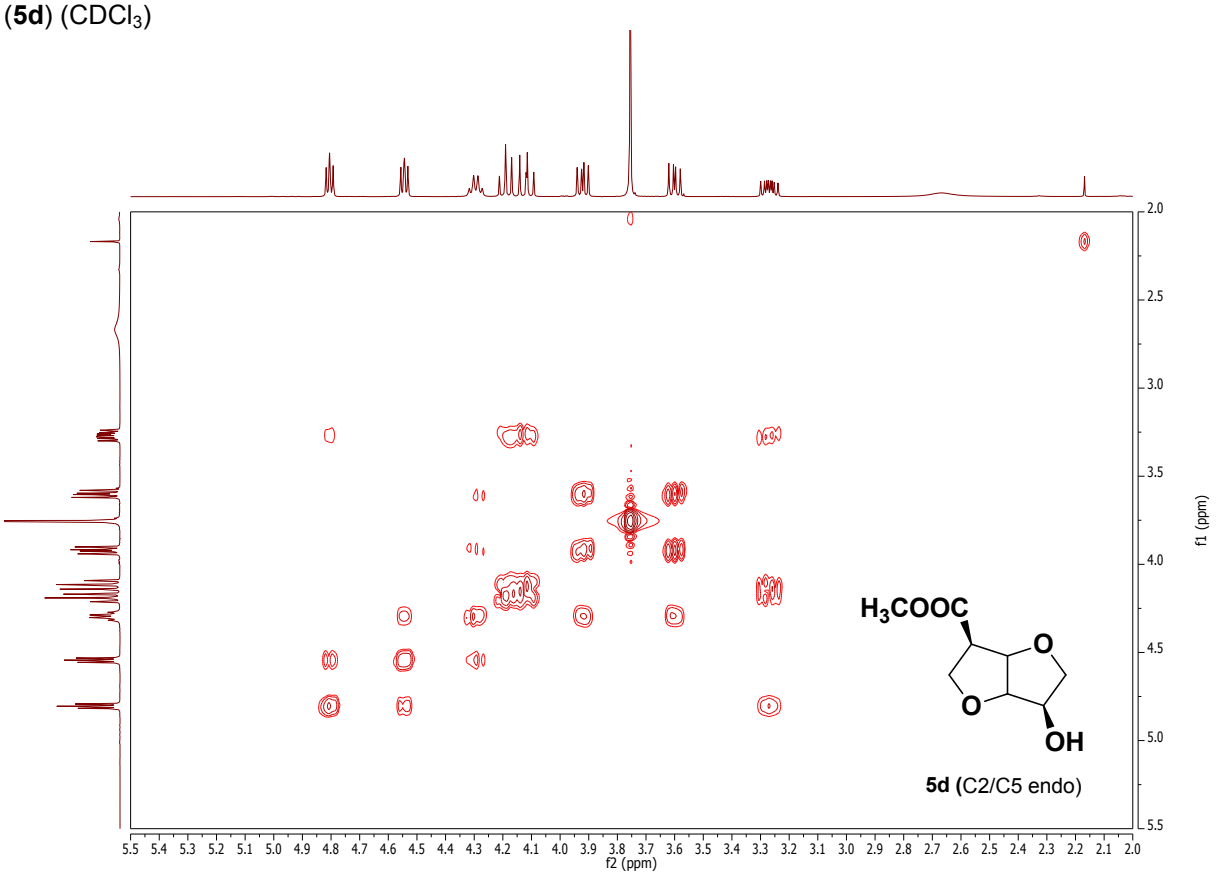

Figure 28. 2D-COSY spectrum of (3R,6R)-methyl 6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylate (**5d**) ($CDCl_3$)

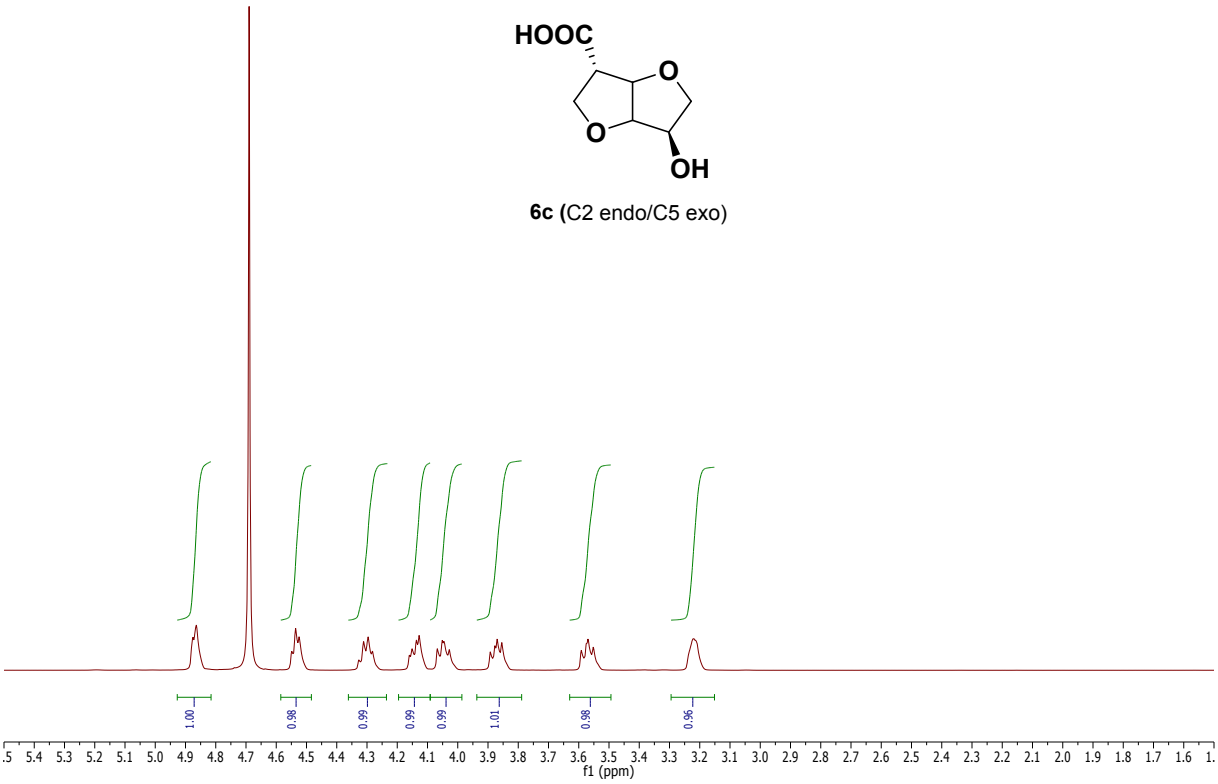

Figure 29. ^{1}H NMR spectrum of (3S,6R)-6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylic acid (**6c**) (D_2O)

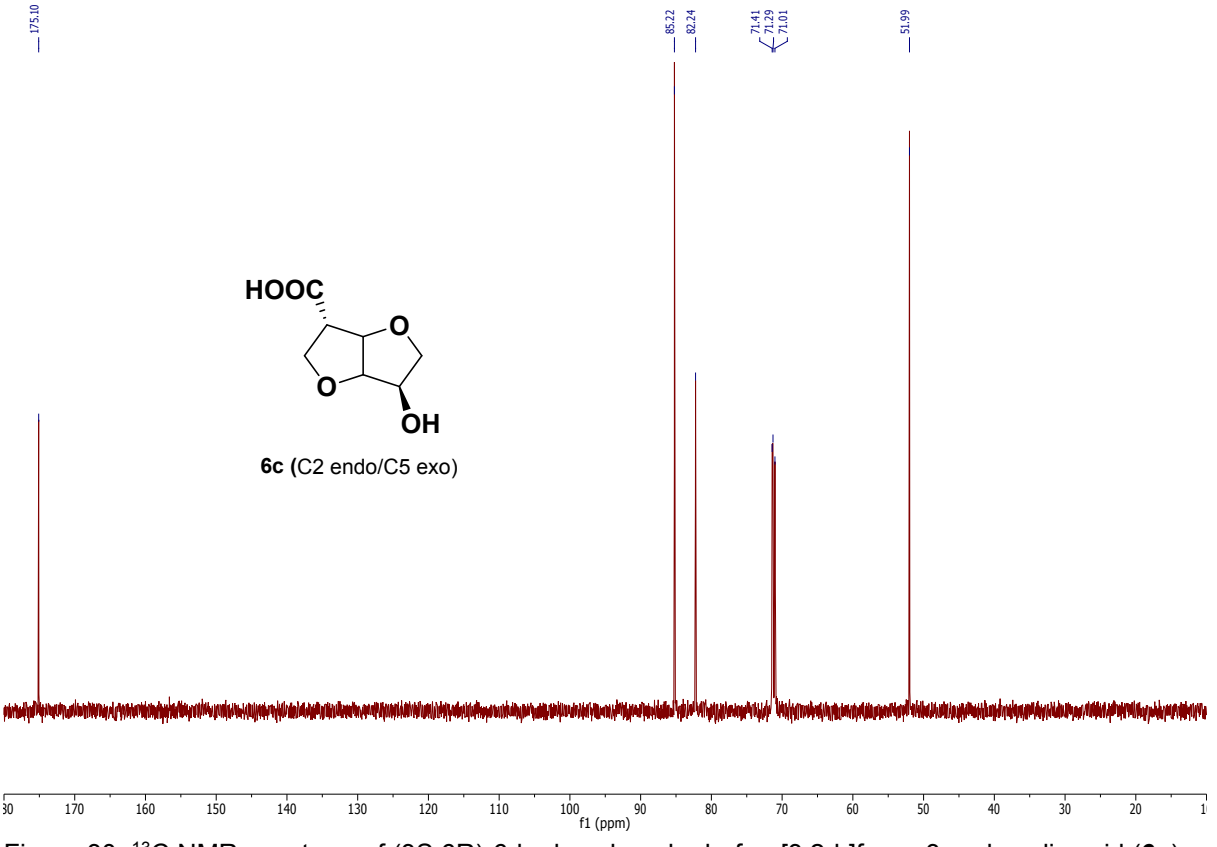

Figure 30. ^{13}C NMR spectrum of (3S,6R)-6-hydroxyhexahydrofuro[3,2-b]furan-3-carboxylic acid (**6c**) (D_2O)

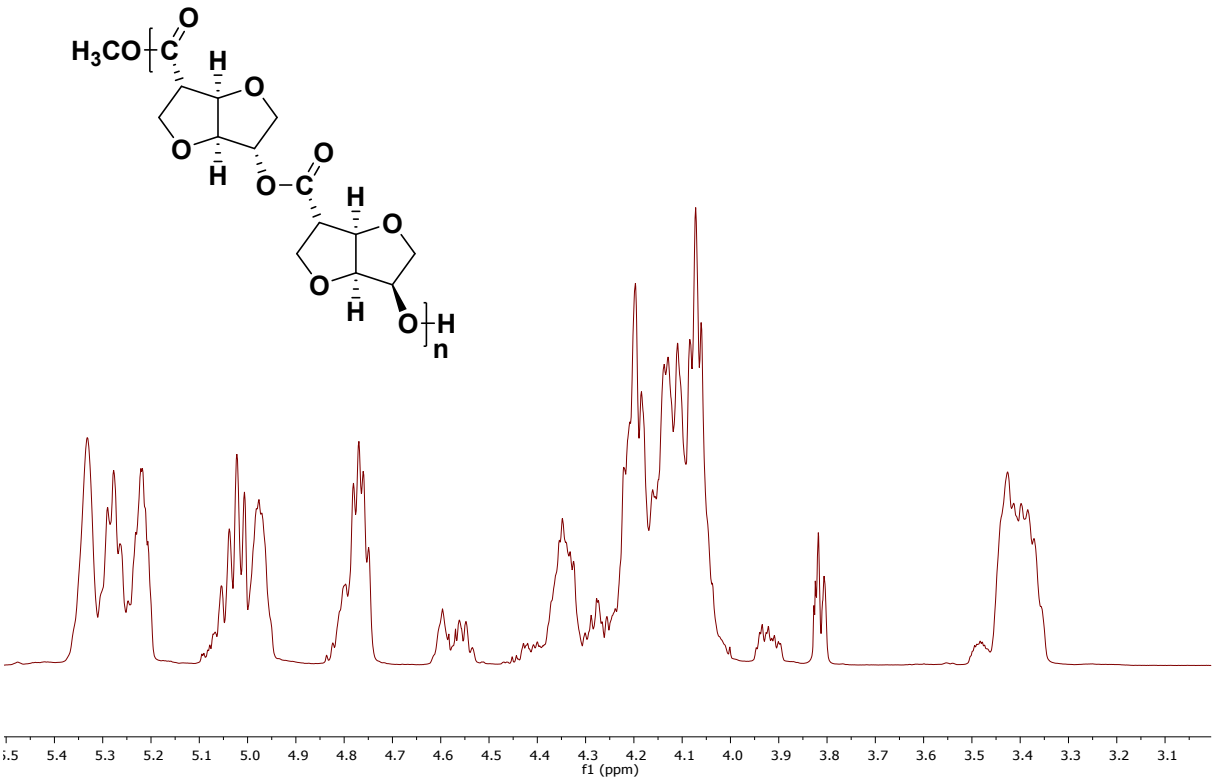

Figure 31. ^{1}H NMR spectrum of PE-5 recorded in (6:1) $CDCl_3$ /TFA (trifluoroacetic acid-d).

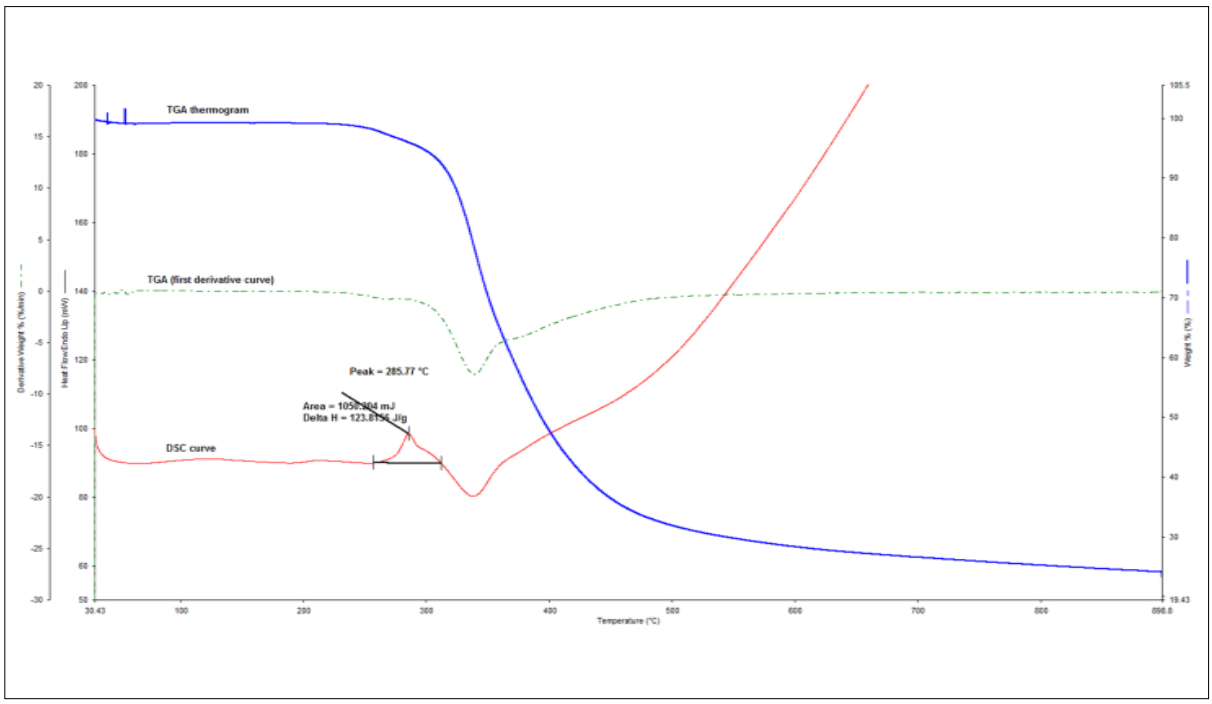

Figure 32. Simultaneous Thermal Analysis (STA) of PE-1 recorded from 30 to 900 °C at 10 °C min−1 under a N_2 atmosphere.

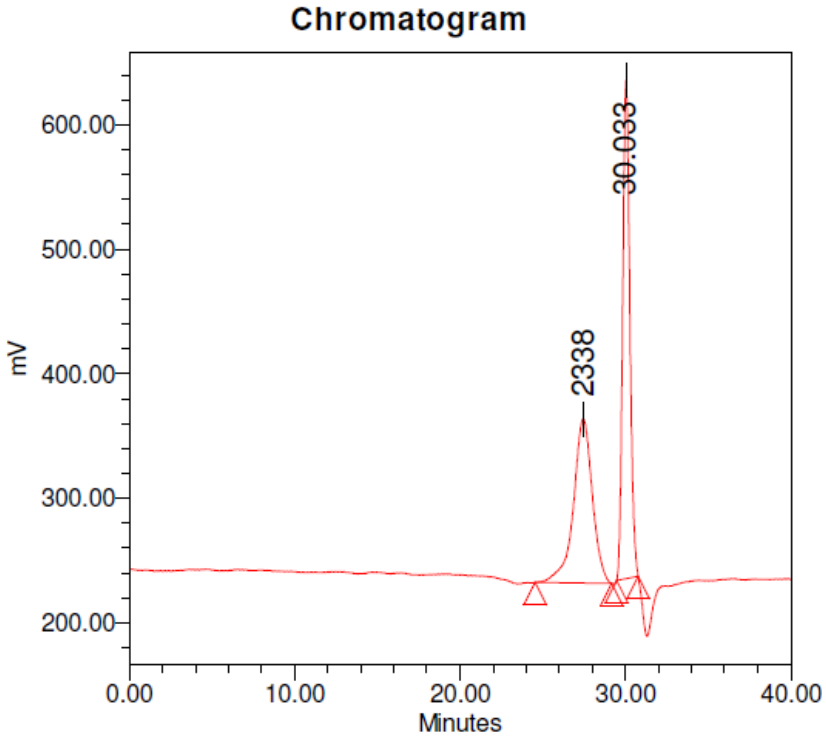

Figure 33. GPC chromatogram of PE-1 measured using HFIP as solvent.

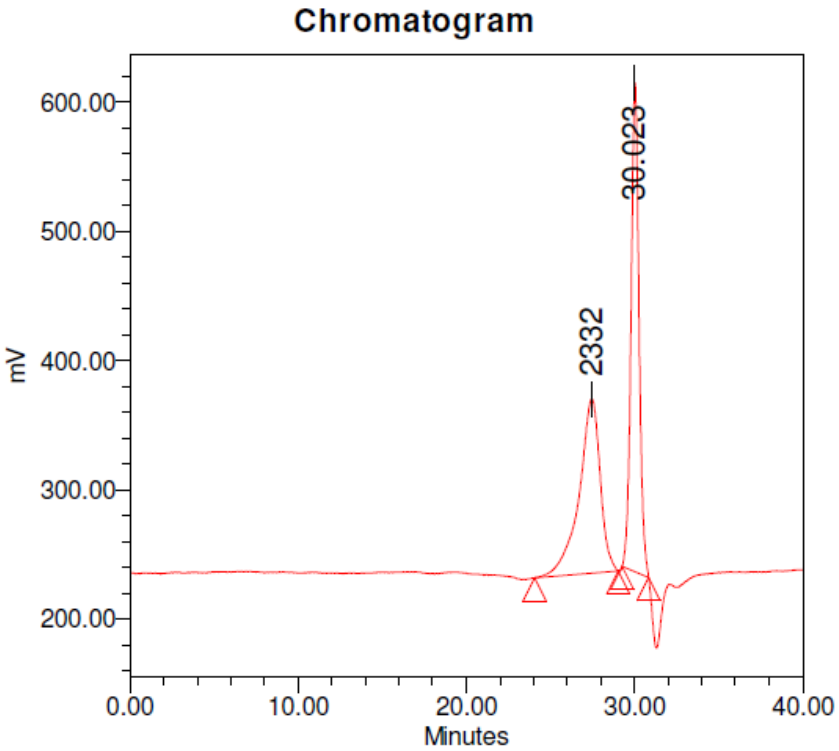

Figure 34. GPC chromatogram of PE-2 measured using HFIP as solvent.

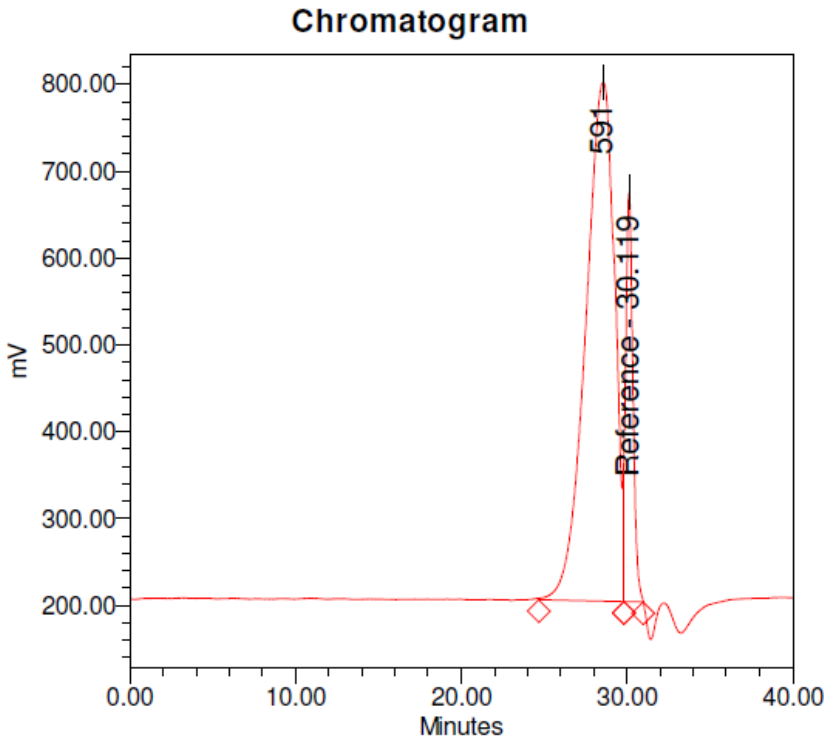

Figure 35. GPC chromatogram of PE-3 measured using HFIP as solvent.

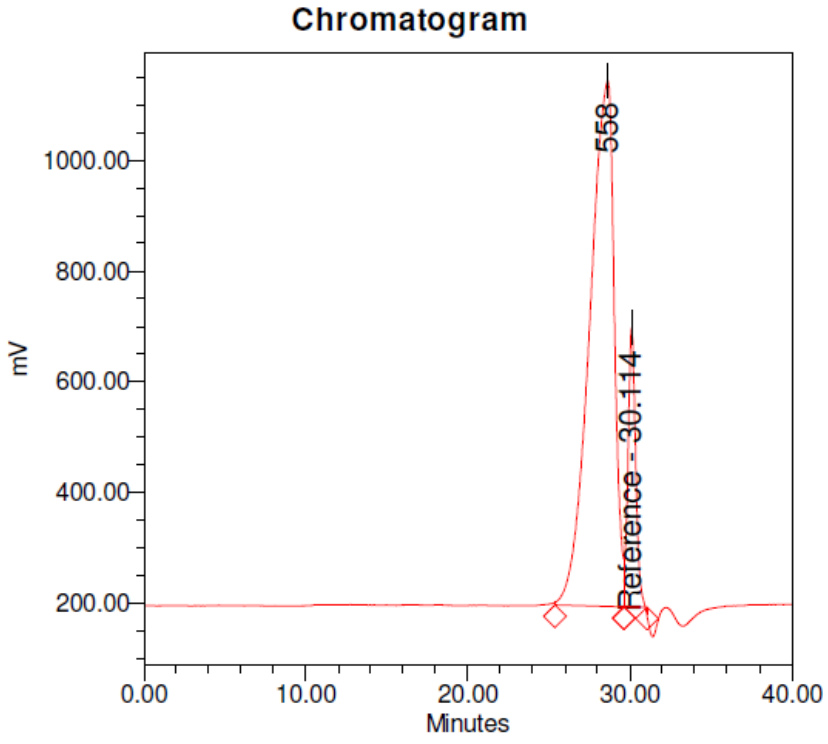

Figure 36. GPC chromatogram of PE-4 measured using HFIP as solvent.

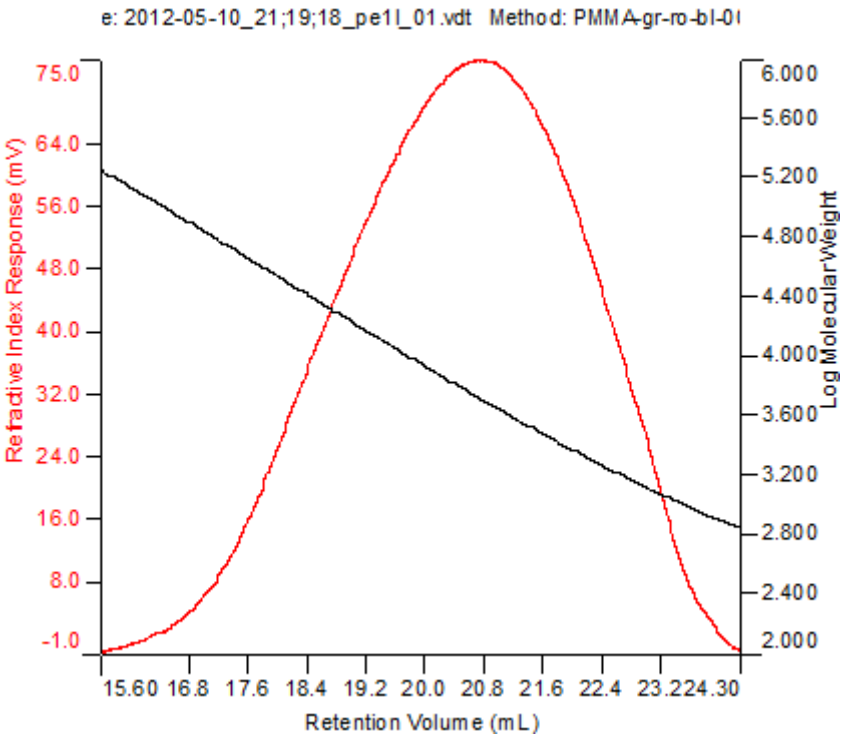

Figure 37. GPC chromatogram of PE-5 measured using HFIP as solvent.

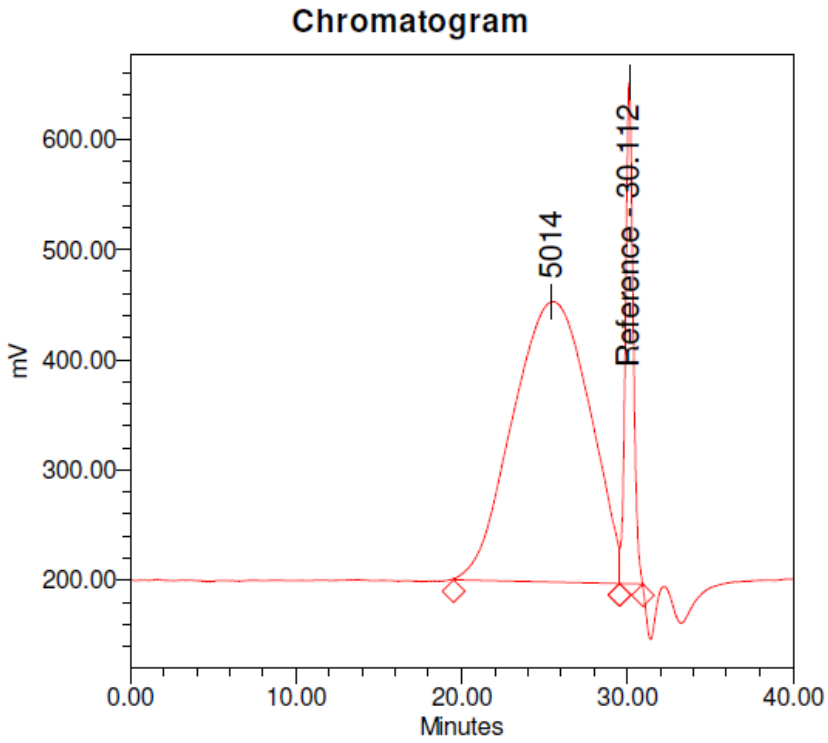

Figure 38. GPC chromatogram of PE-6 measured using HFIP as solvent.