

3,4-Unsubstituted 2-*tert*-butyl-pyrrolidine-1-oxyls with hydrophilic functional groups in the side chains

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1. IR spectral data

1.1 Methyl 3-(5-*tert*-butyl-2-ethyl-1-oxido-3,4-dihydro-2*H*-pyrrol-2-yl)propanoate (**4a**)

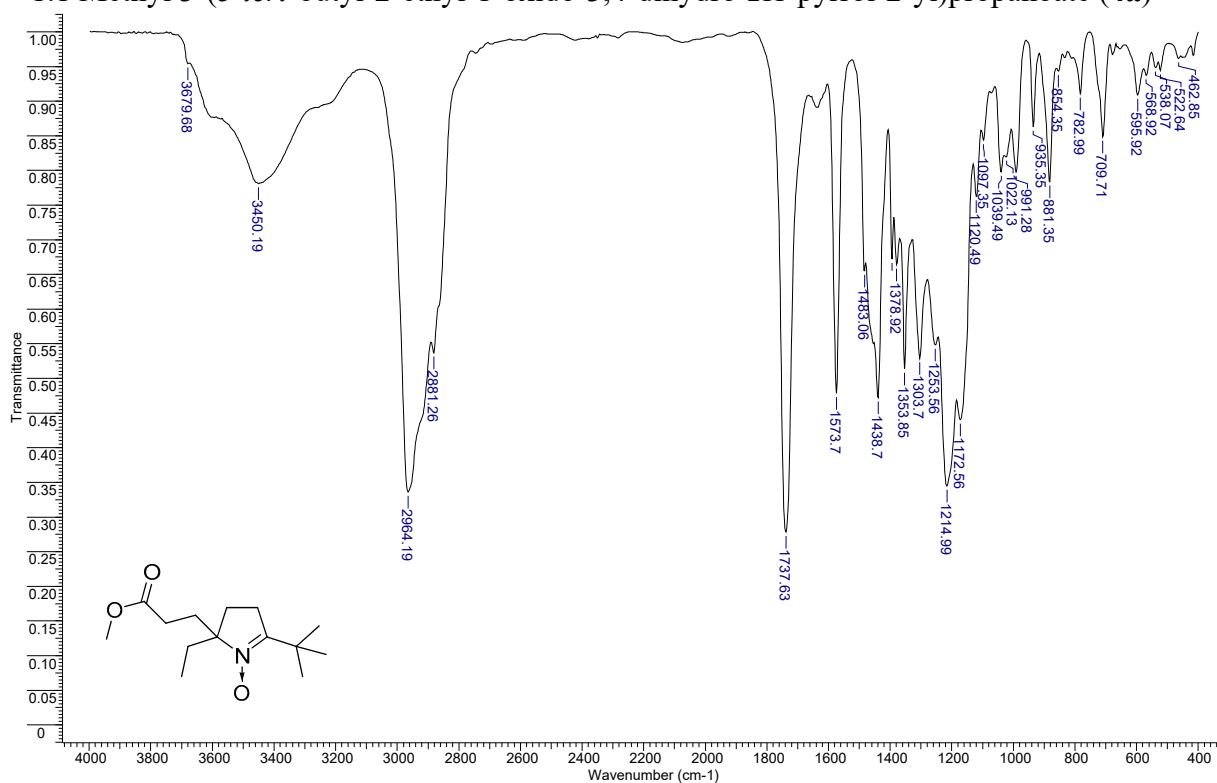


Figure S1. IR spectrum of **4a** (neat)

1.2 Dimethyl 3,3'-(5-*tert*-butyl-1-oxido-3,4-dihydro-2*H*-pyrrole-2,2-diyl)dipropionate (**4b**)

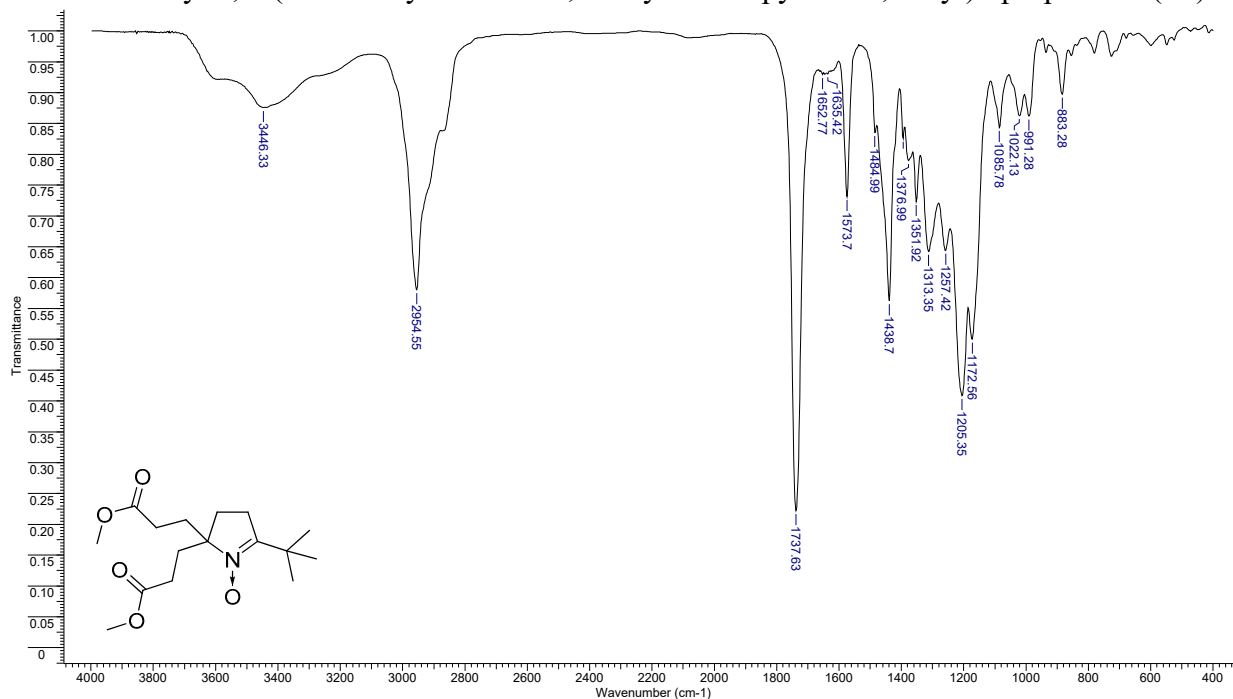


Figure S2. IR spectrum of **4b** (neat)

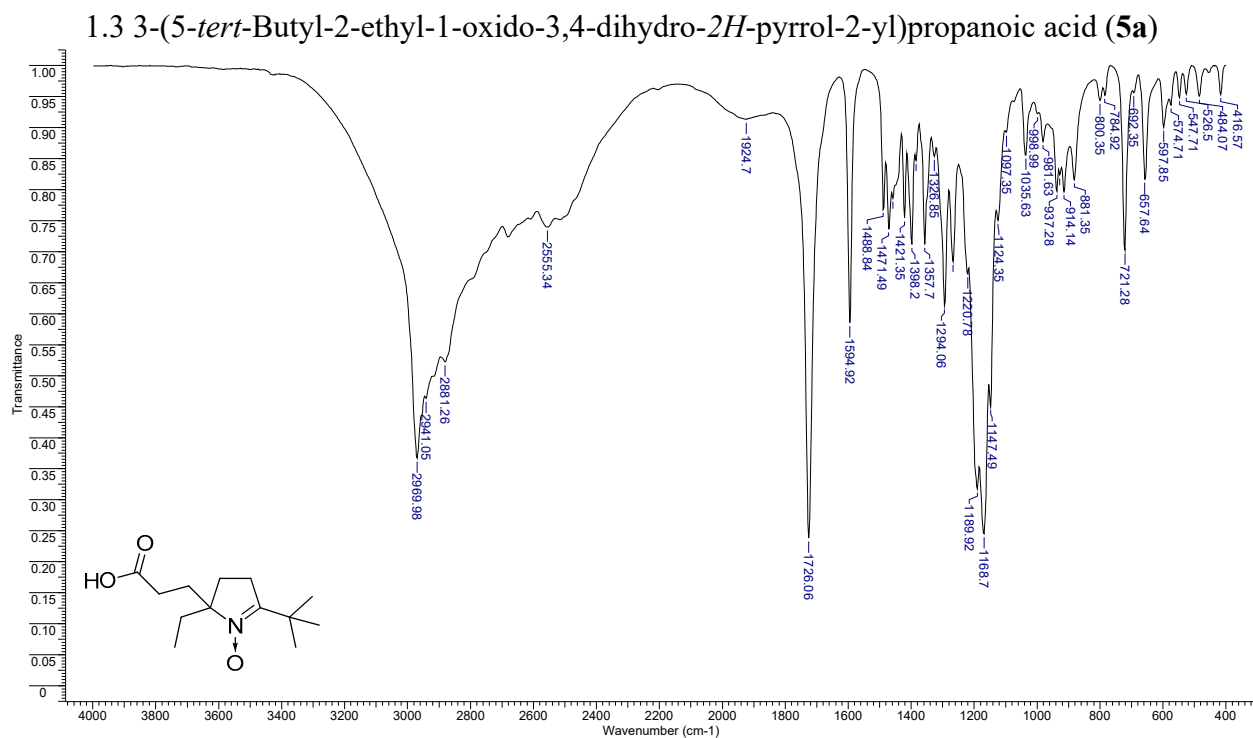


Figure S3. IR spectrum of **5a** (KBr)

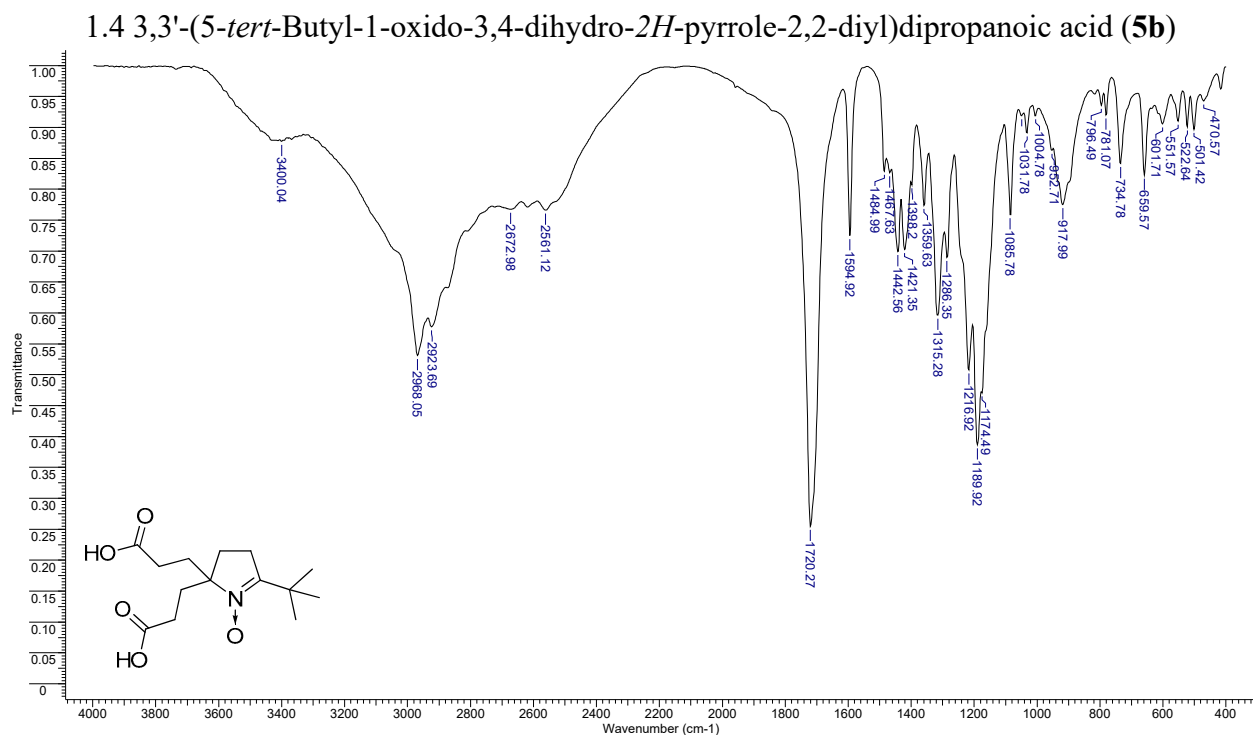


Figure S4. IR spectrum of **5b** (KBr)

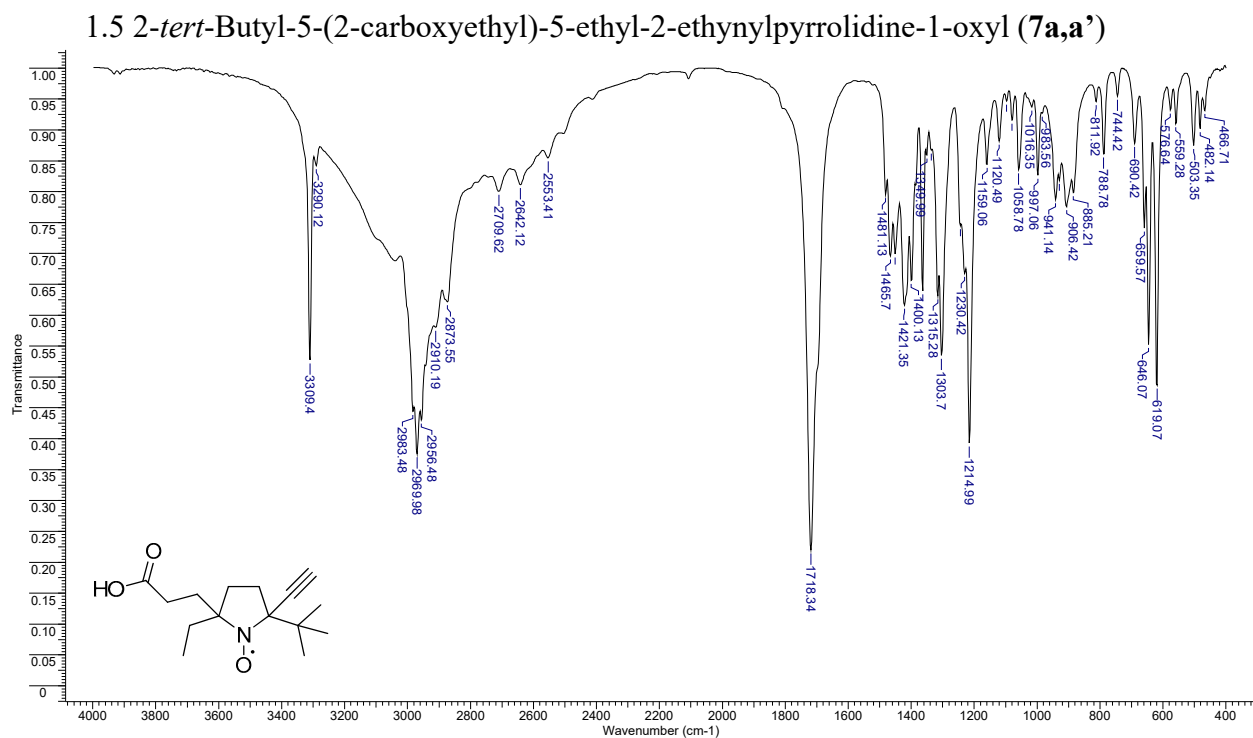


Figure S5. IR spectrum of **7a,a'**

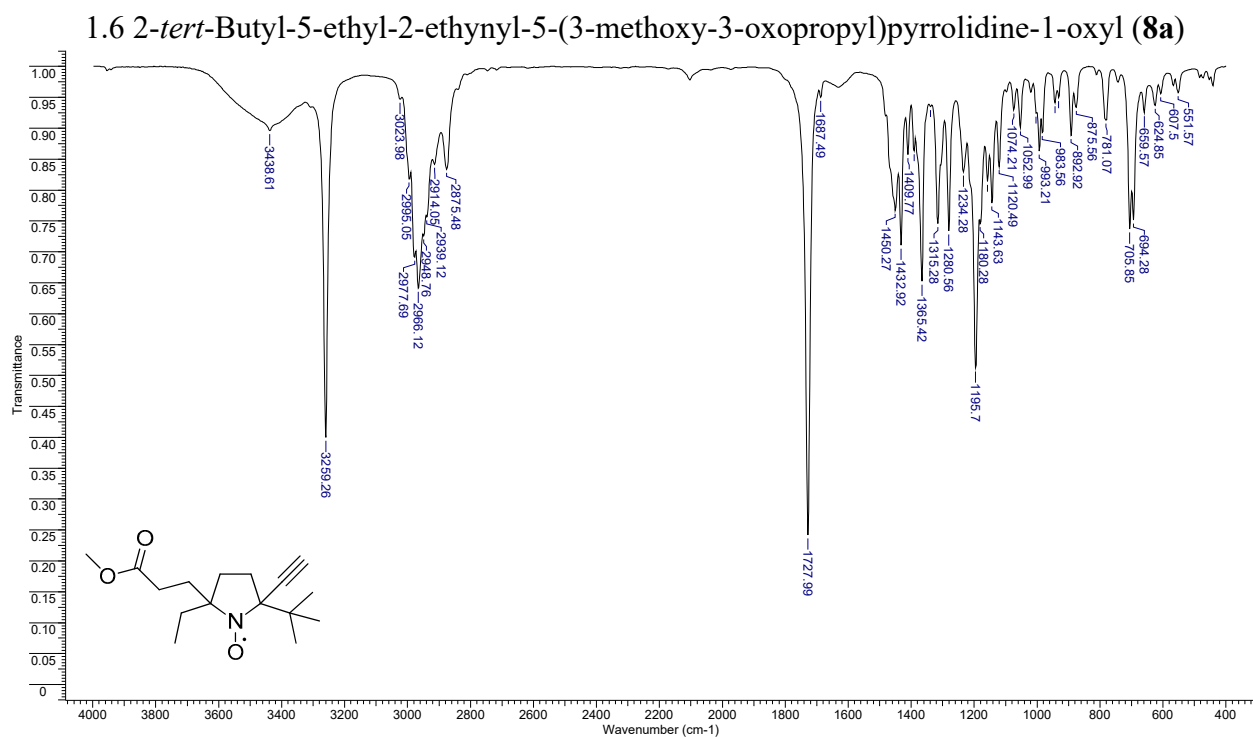


Figure S6. IR spectrum of **8a** (KBr)

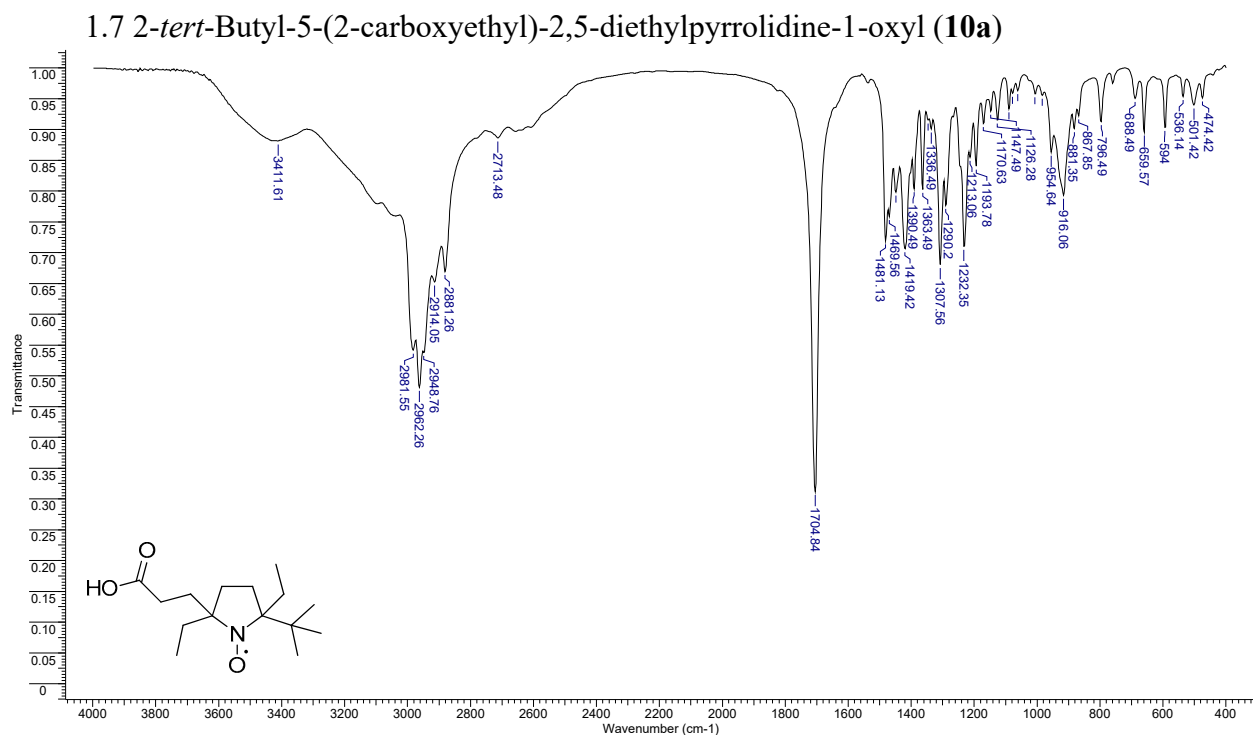


Figure S7. IR spectrum of **10a** (KBr)

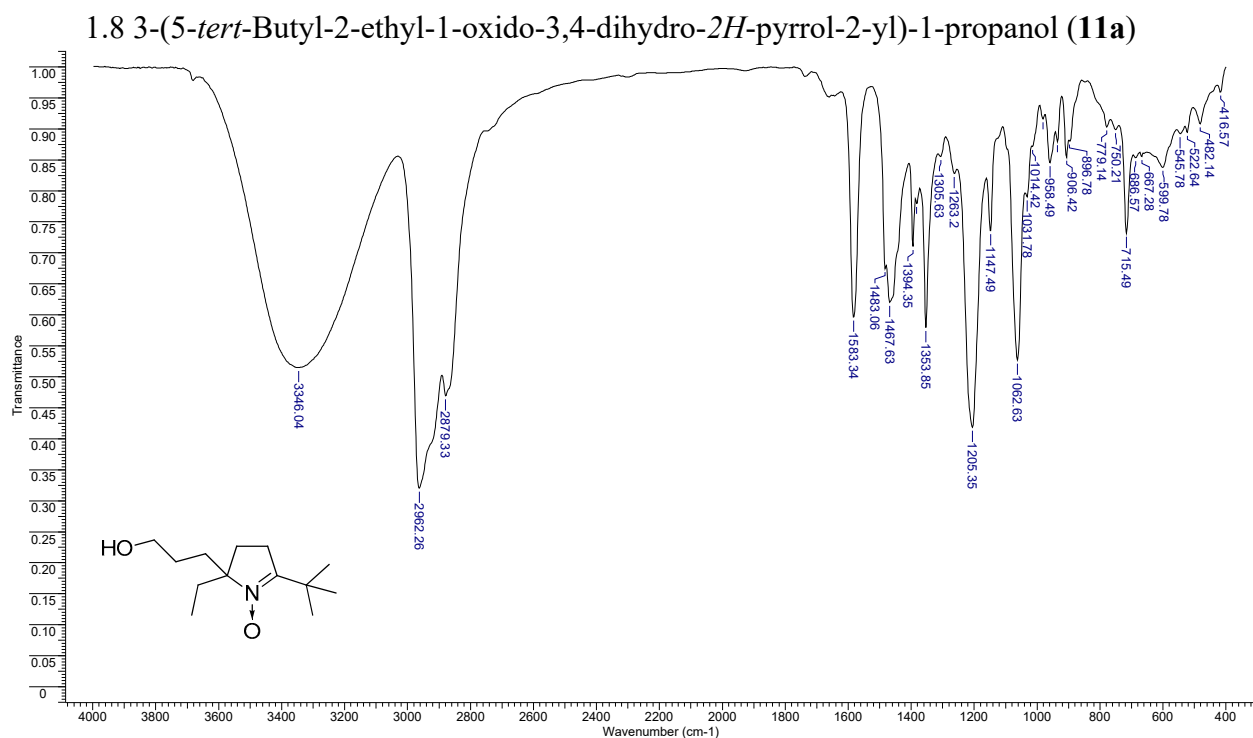


Figure S8. IR spectrum of **11a** (neat)

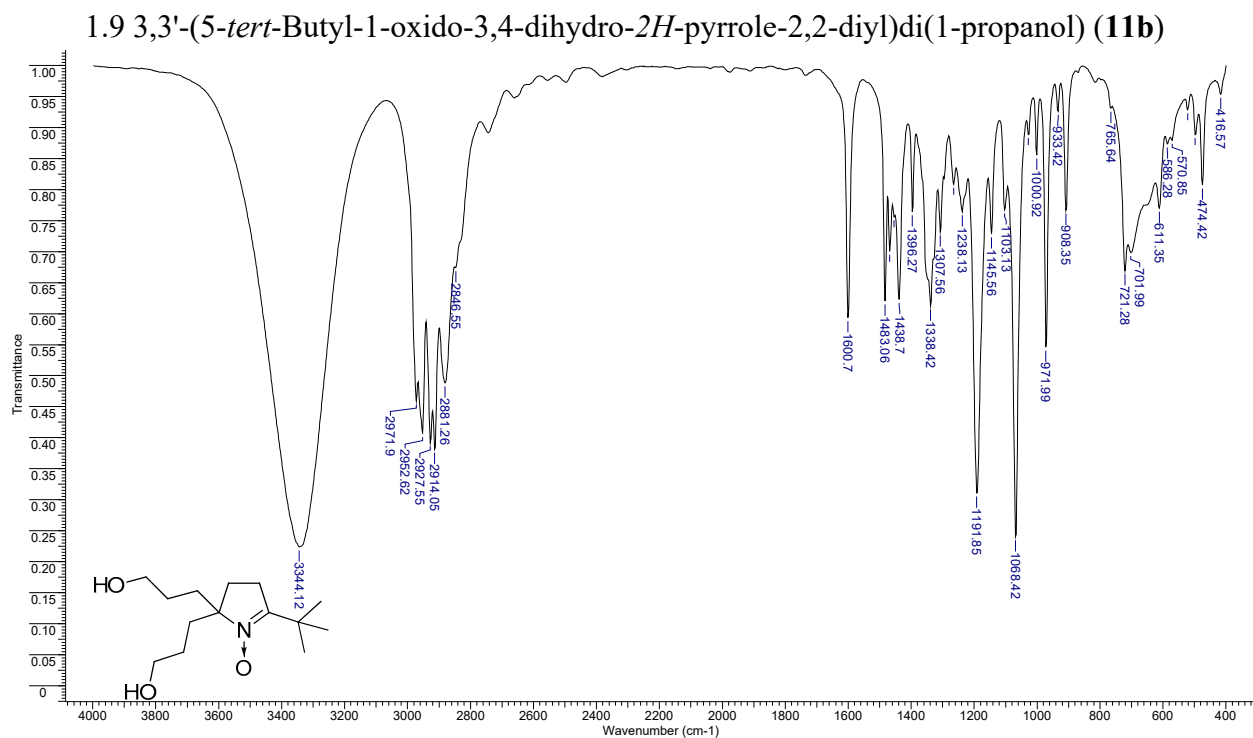


Figure S9. IR spectrum of **11b** (KBr)

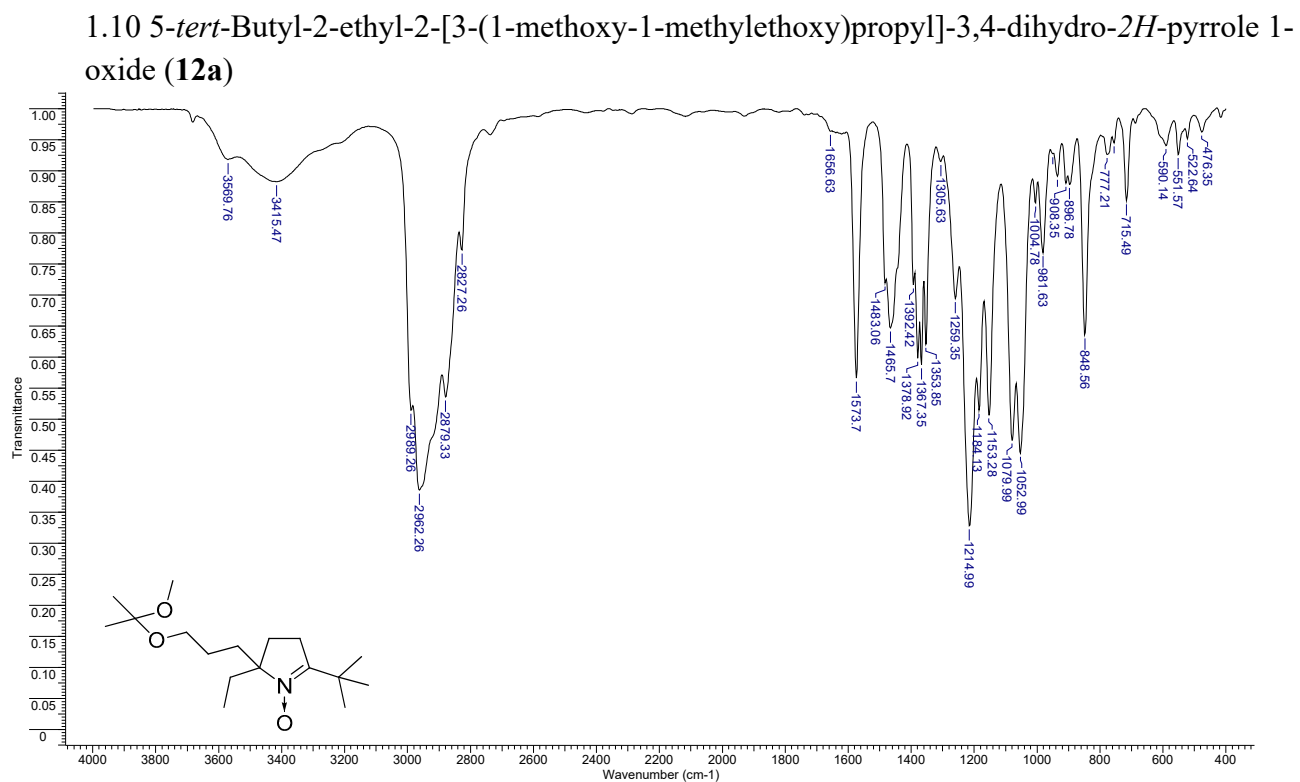


Figure S10. IR spectrum of **12a** (neat)

1.11 5-*tert*-Butyl-2,2-bis[3-(1-methoxy-1-methylethoxy)propyl]-3,4-dihydro-2*H*-pyrrole 1-oxide (12b)

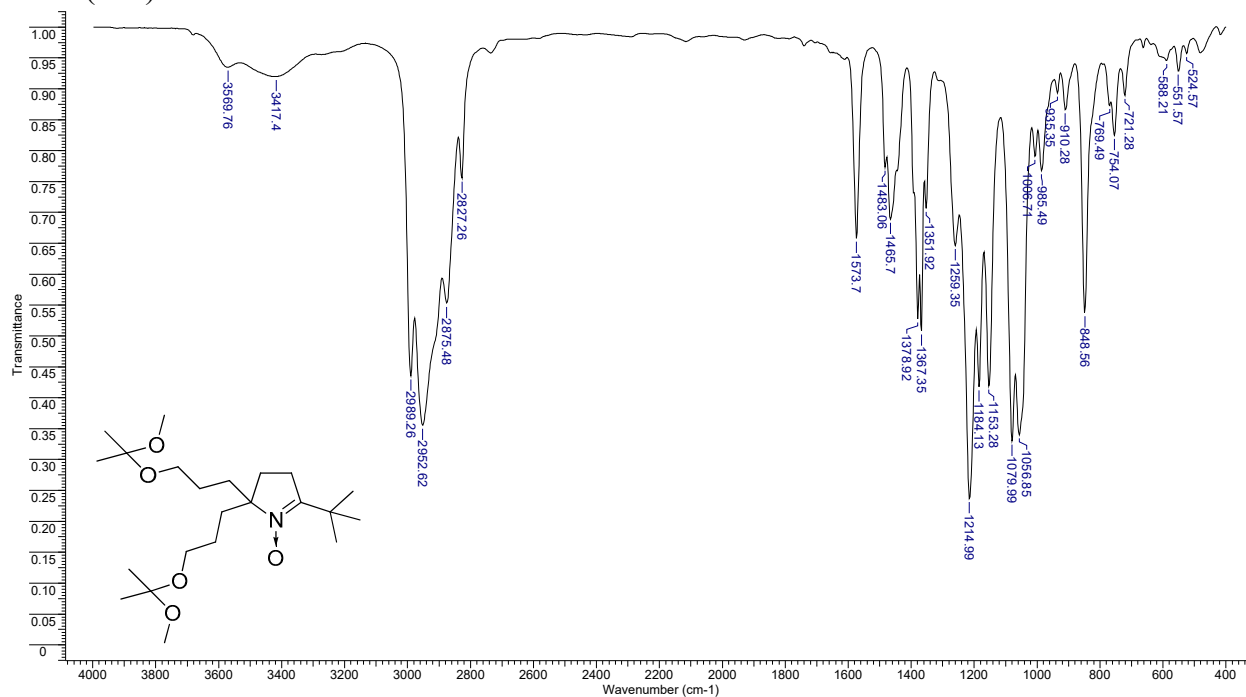


Figure S11. IR spectrum of 12b (neat)

1.12 2-*tert*-Butyl-2-ethyl-5,5-bis(3-hydroxypropyl)pyrrolidine-1-oxyl (14a)

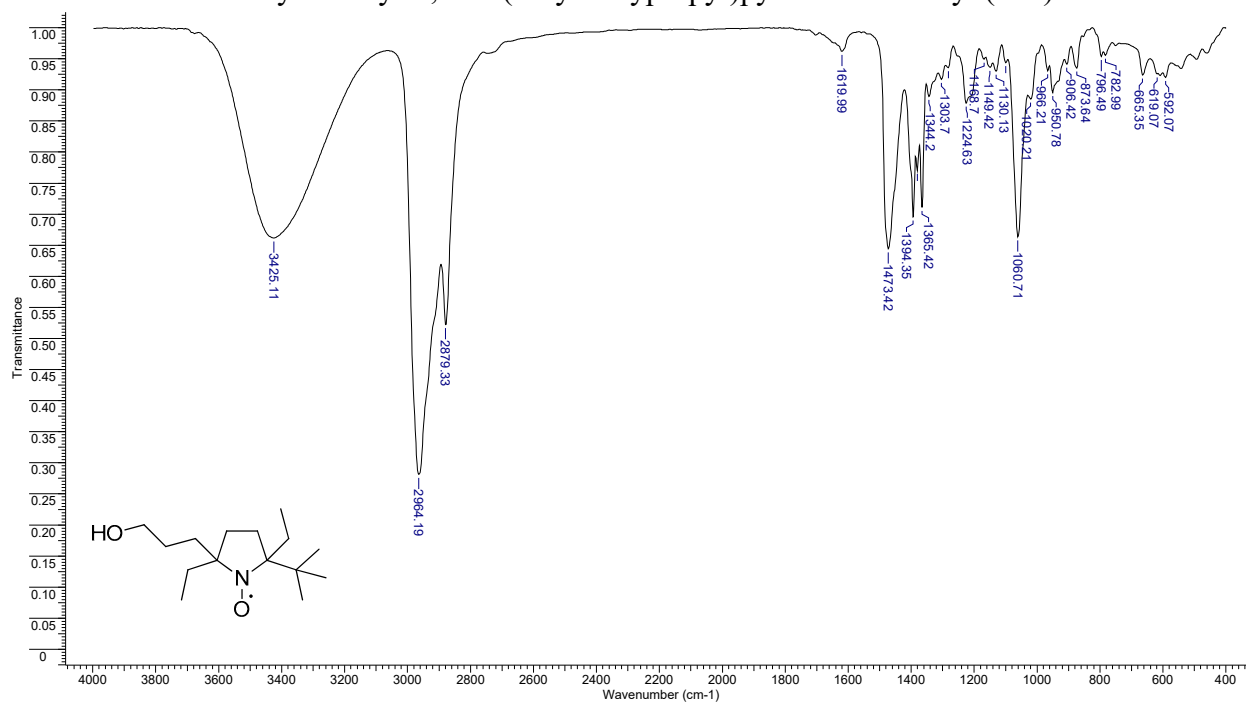


Figure S12. IR spectrum of 14a (neat)

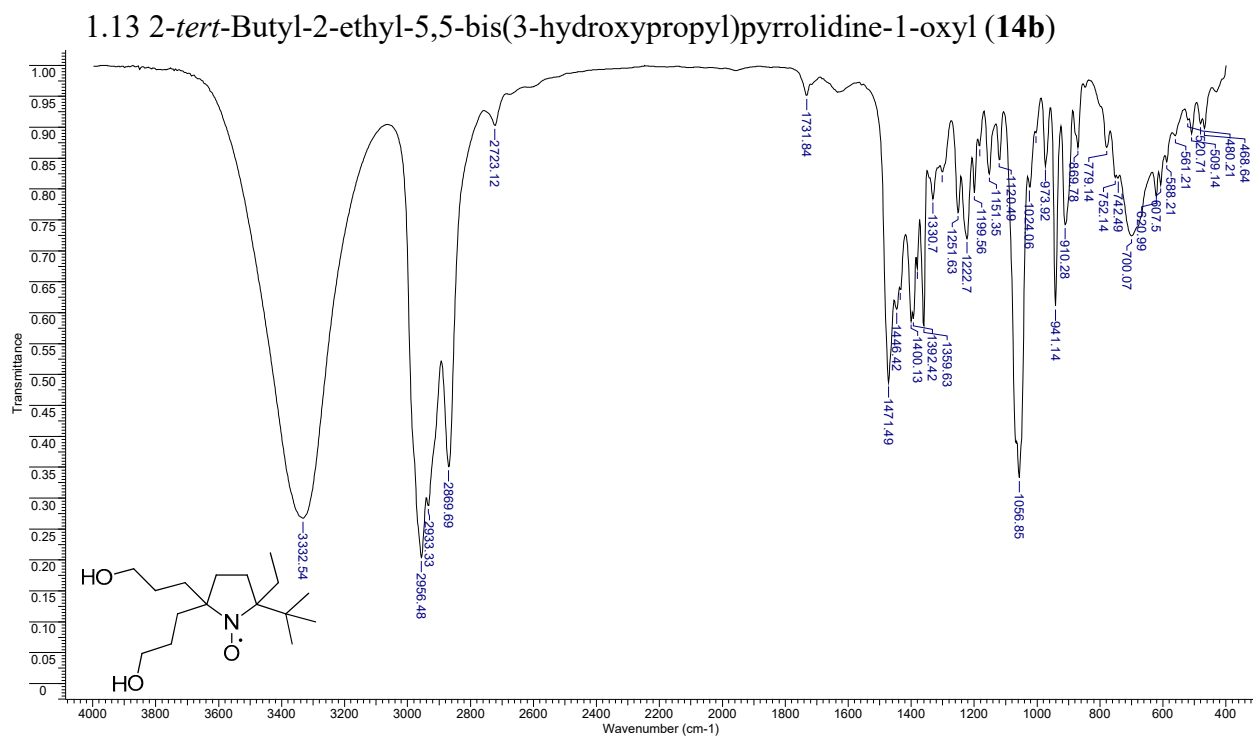


Figure S13. IR spectrum of **14b** (KBr)

2. ^1H and ^{13}C NMR spectral data

2.1 Methyl 3-(5-*tert*-butyl-2-ethyl-1-oxido-3,4-dihydro-2*H*-pyrrol-2-yl)propanoate (**4a**)

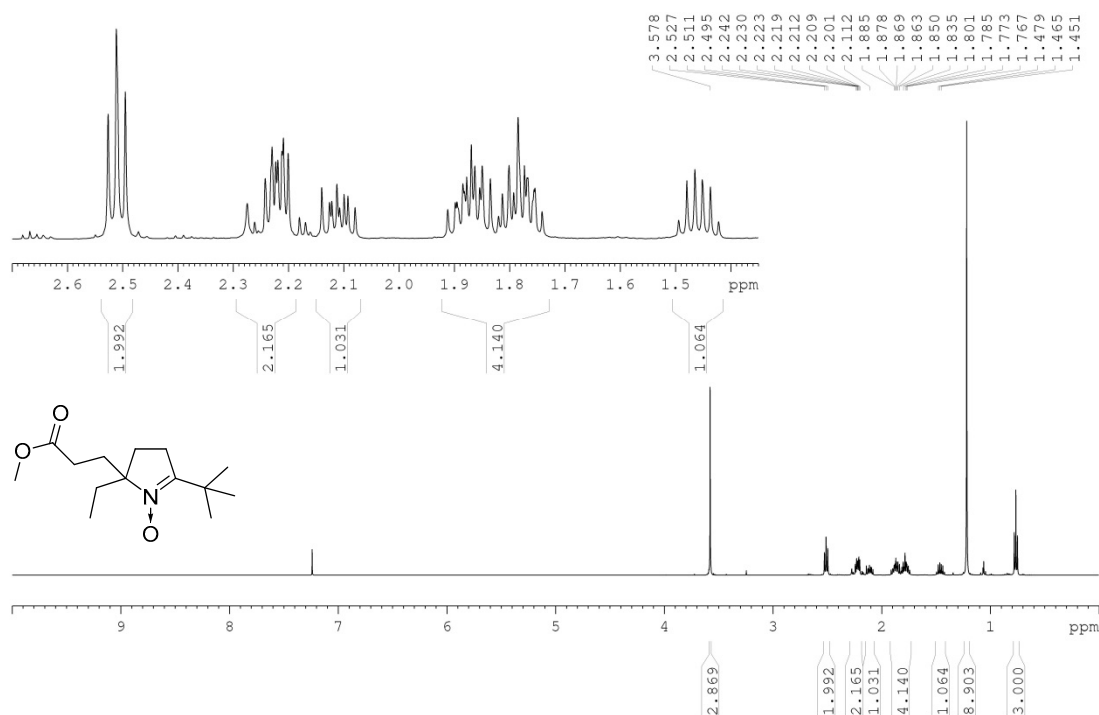


Figure S14. ^1H NMR spectrum of **4a** in CDCl_3 at 500 MHz

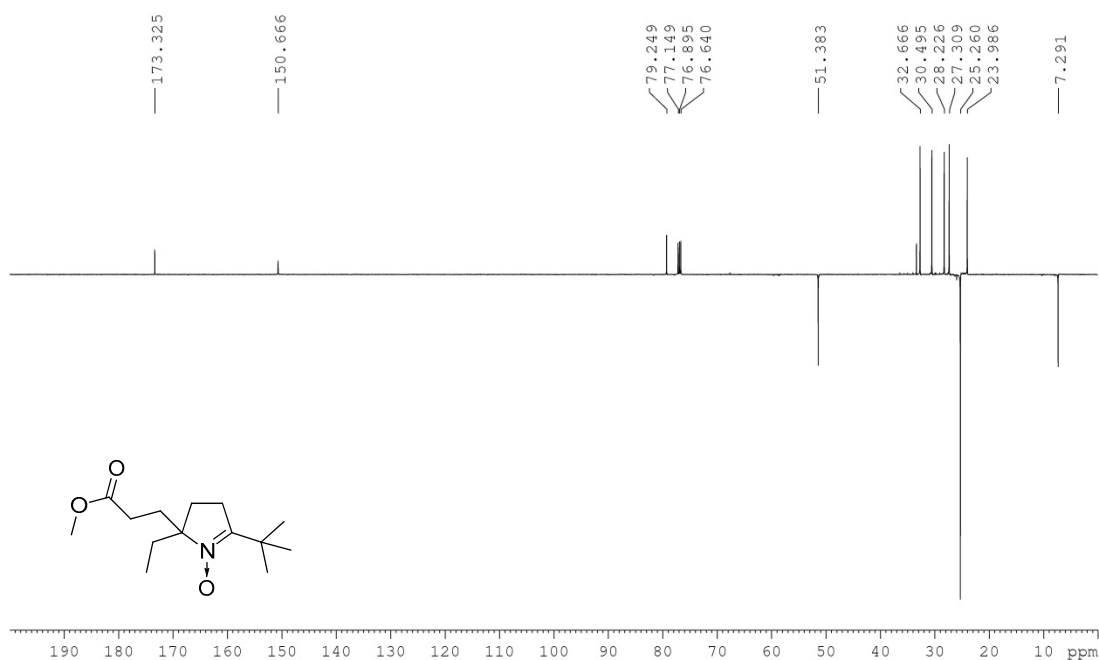


Figure S15. ^{13}C NMR spectrum of **4a** in CDCl_3 at 125 MHz

2.2 Dimethyl 3,3'-(5-*tert*-butyl-1-oxido-3,4-dihydro-2*H*-pyrrole-2,2-diyl)dipropionate (**4b**)

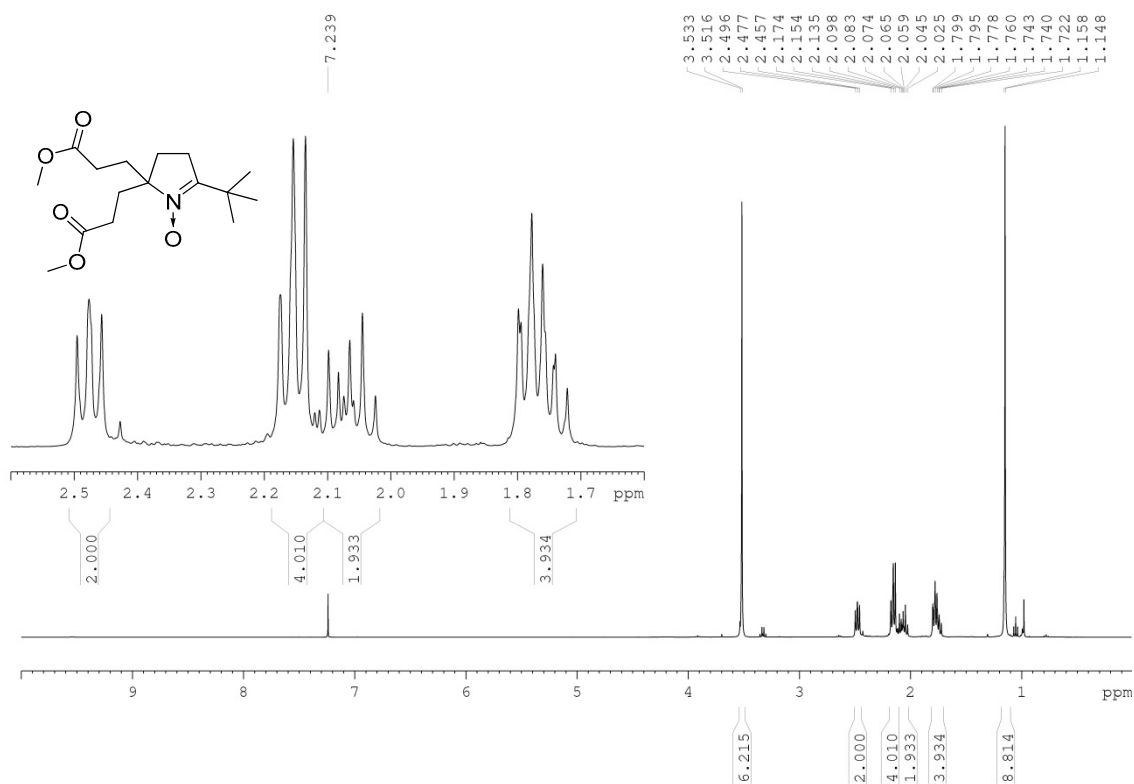


Figure S16. ¹H NMR spectrum of **4b** in CDCl₃ at 400 MHz



Figure S17. ¹³C NMR spectrum of **4b** in CDCl₃ at 100 MHz

2.3 3-(5-*tert*-Butyl-2-ethyl-1-oxido-3,4-dihydro-2*H*-pyrrol-2-yl)propanoic acid (**5a**)

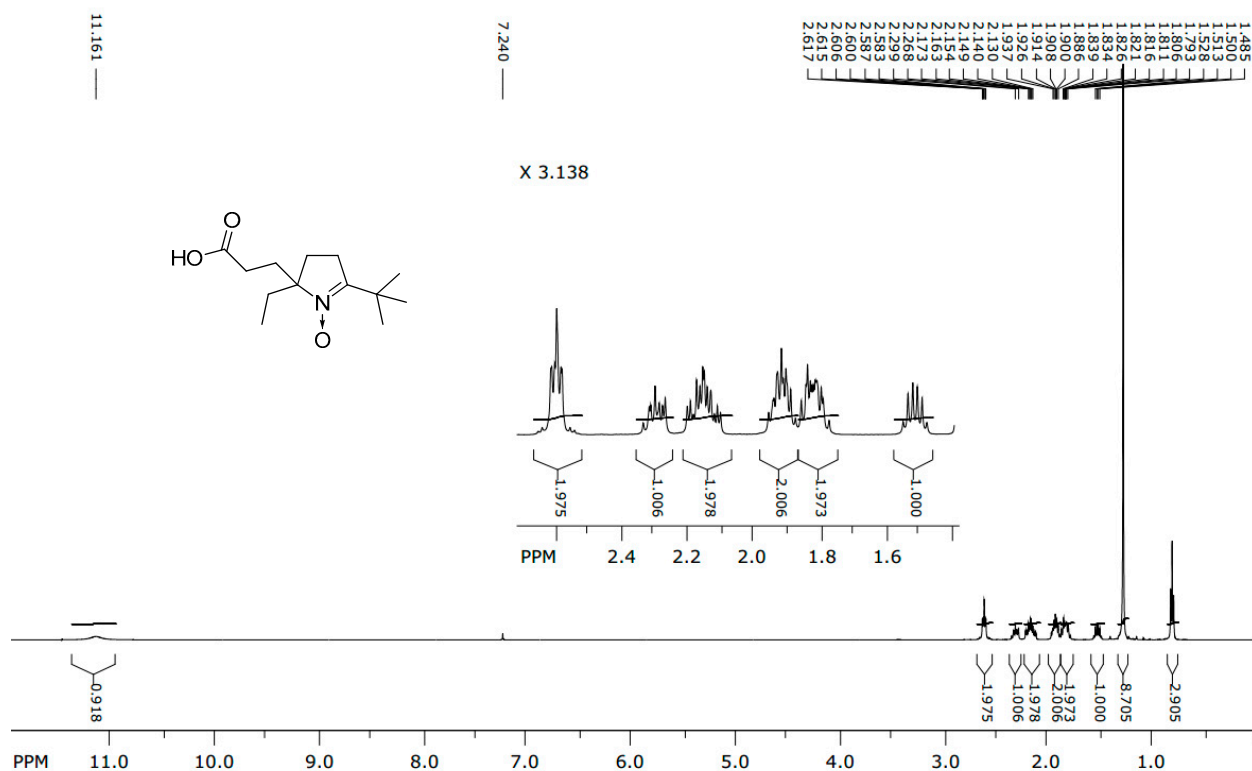


Figure S18. ¹H NMR spectrum of **5a** in CDCl₃ at 500 MHz

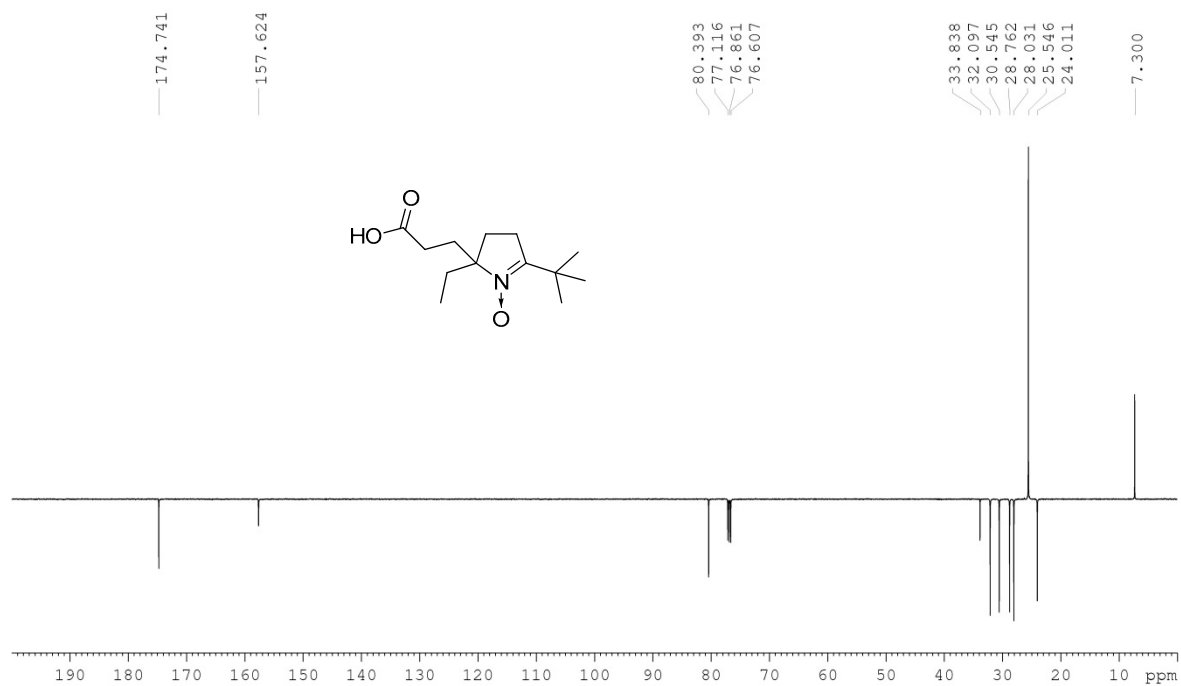


Figure S19. ¹³C NMR spectrum of **5a** in CDCl₃ at 125 MHz

2.4 3,3'-(5-*tert*-Butyl-1-oxido-3,4-dihydro-2*H*-pyrrole-2,2-diyl)dipropionic acid (**5b**)

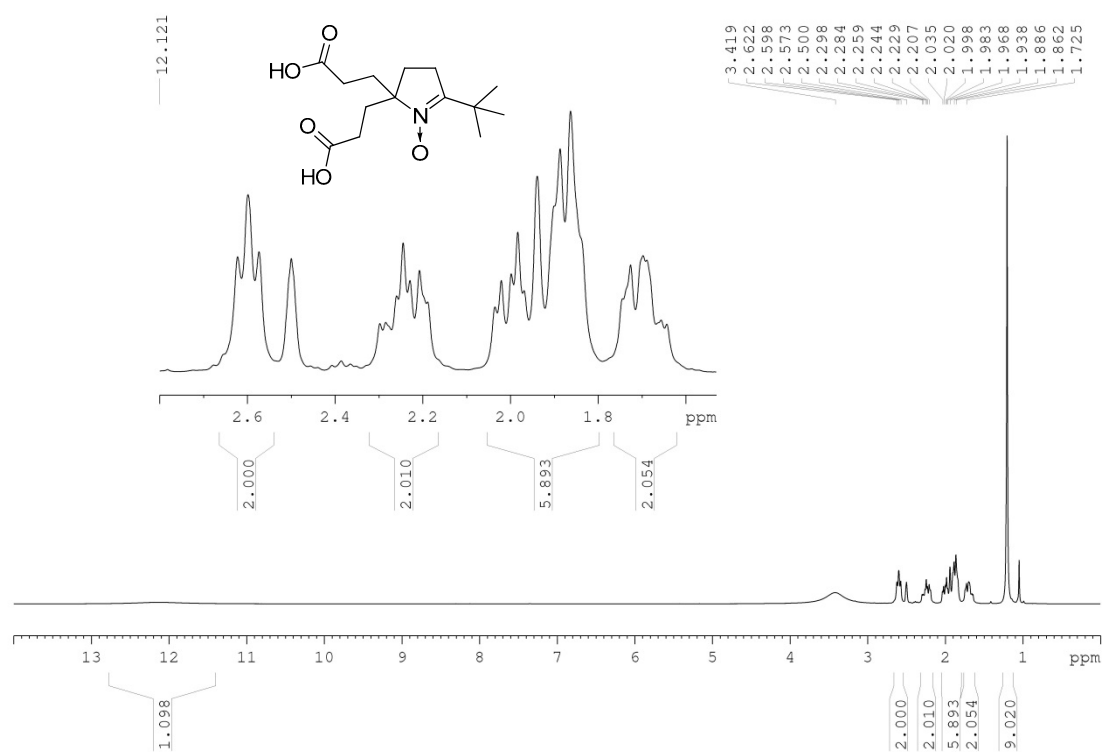


Figure S20. ¹H NMR spectrum of **5b** in DMSO-d₆ at 300 MHz

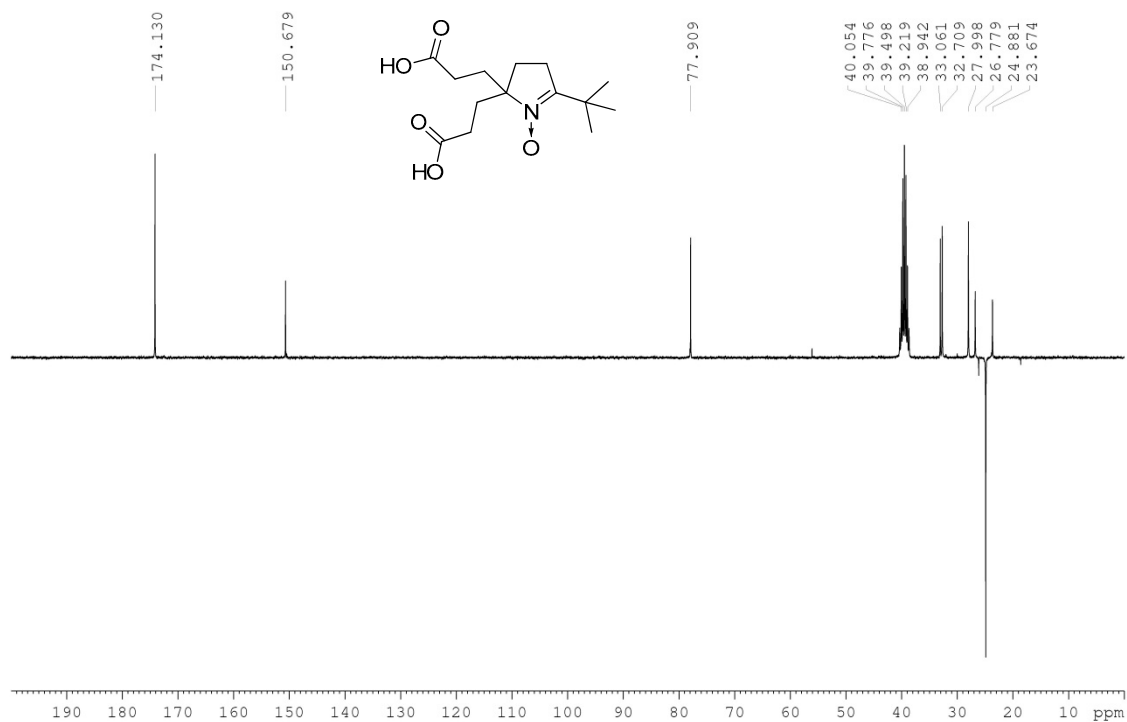


Figure S21. ¹³C NMR spectrum of **5b** in DMSO-d₆ at 75 MHz

2.5 2-*tert*-Butyl-5-(2-carboxyethyl)-5-ethyl-2-ethynylpyrrolidinium trifluoroacetate (mixture of diastereomers) (**7a,a'**_{red})

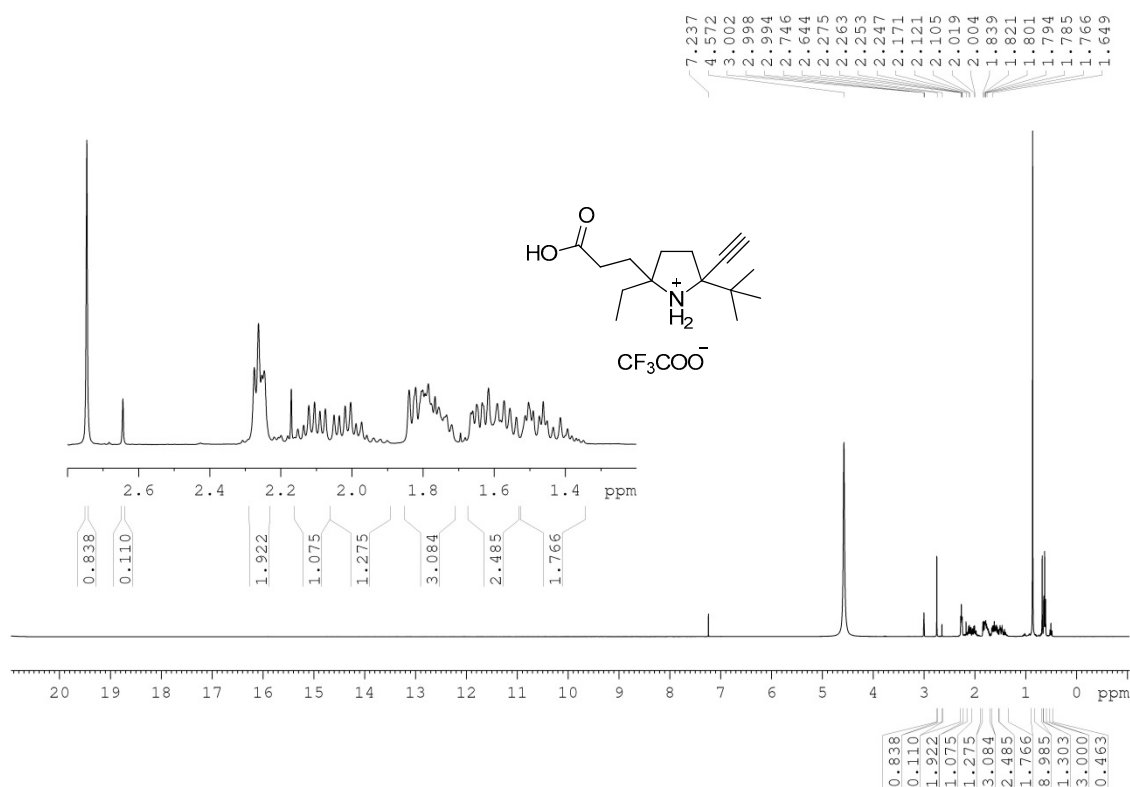


Figure S22. ^1H NMR spectrum of **7a,a'**_{red} in CDCl_3 - CD_3OD at 400 MHz

2.6 2-*tert*-Butyl-5-ethyl-2-ethynyl-5-(3-methoxy-3-oxopropyl)pyrrolidinium trifluoroacetate (**8a_red**)

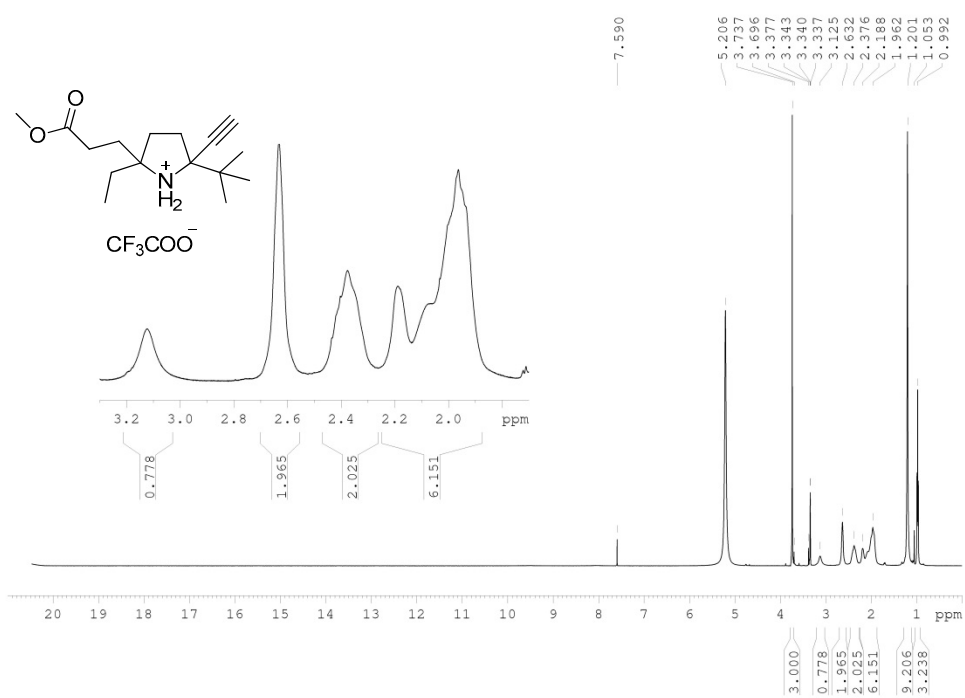


Figure S23. ^1H NMR spectrum of **8a_red** in CDCl_3 - CD_3OD at 500 MHz at 298 K

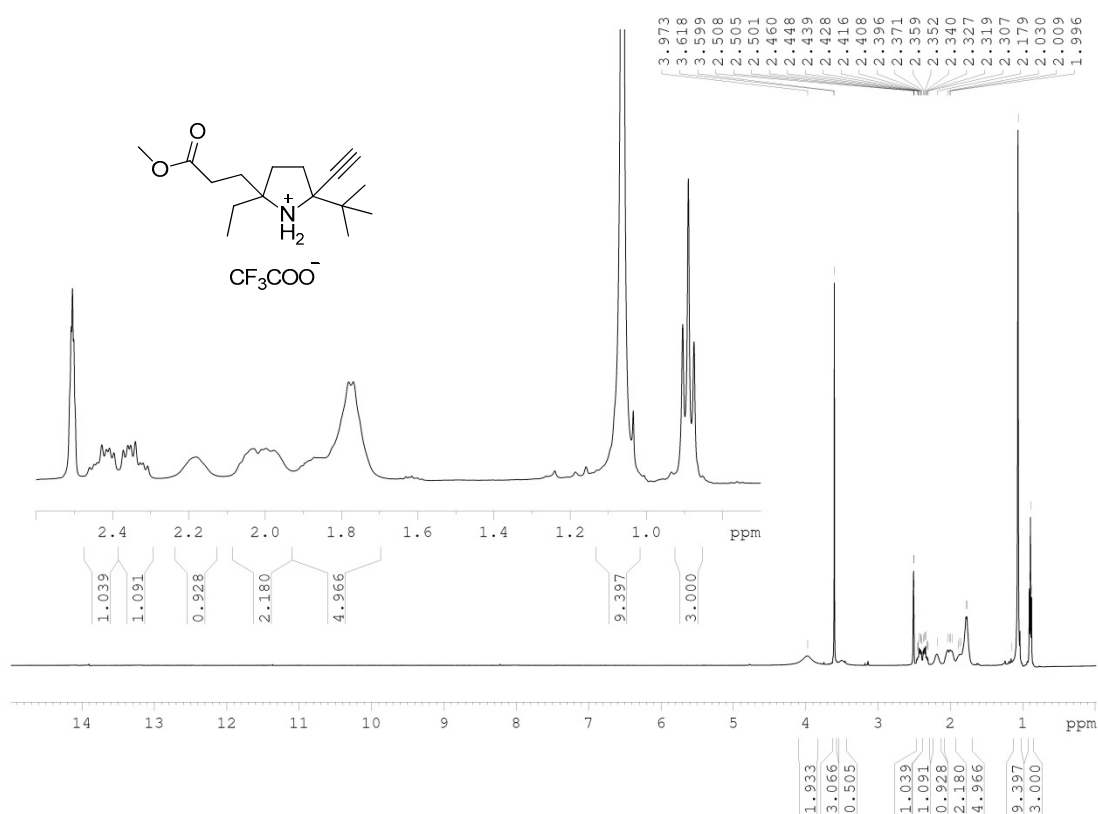


Figure S24. ¹H NMR spectrum of **8a_{red}** in DMSO-*d*₆ at 500 MHz at 333 K

2.7 2-*tert*-Butyl-5-(2-carboxyethyl)-2,5-diethylpyrrolidinium trifluoroacetate (**10a_{red}**)

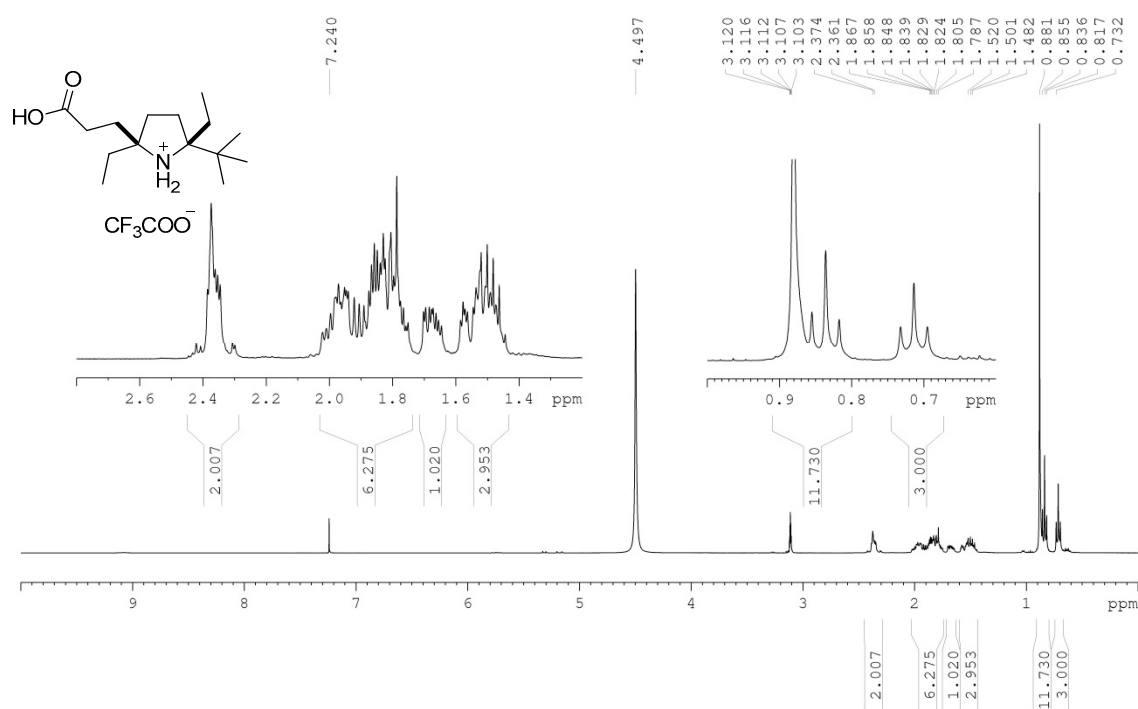


Figure S25. ¹H NMR spectrum of **10a_{red}** in CDCl₃-CD₃OD at 400 MHz

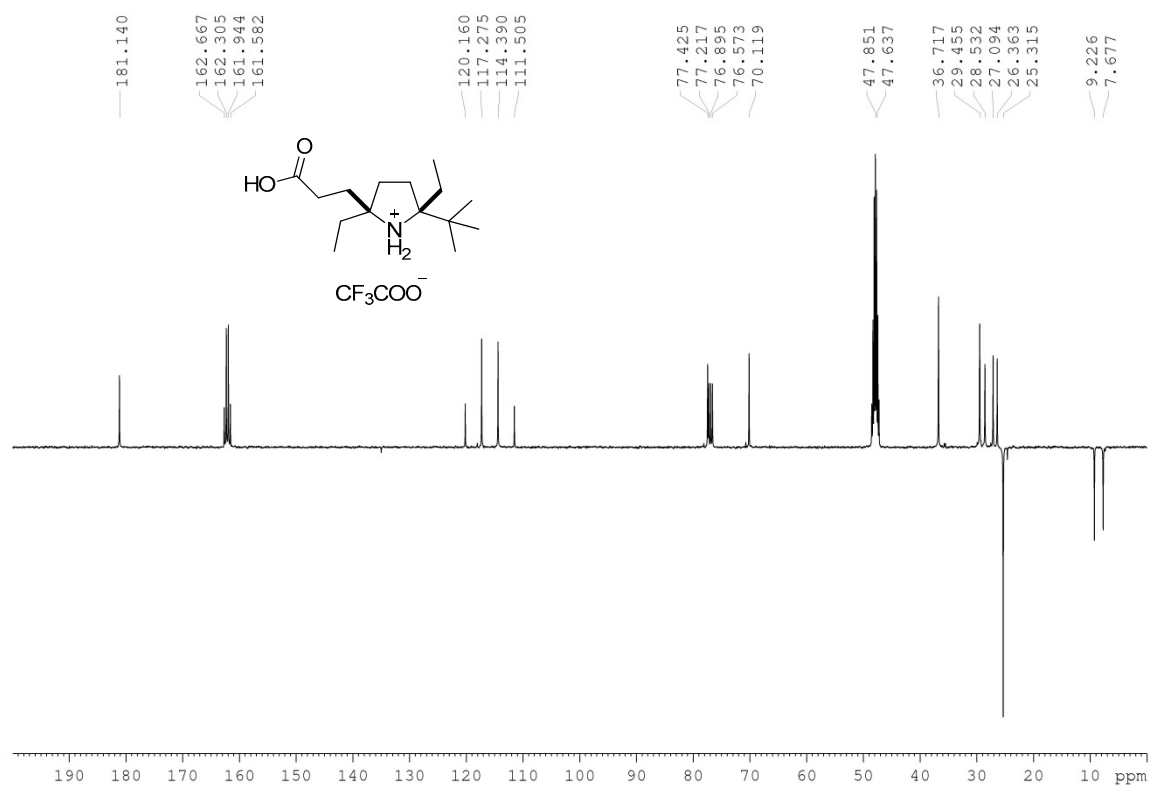


Figure S26. ¹³C NMR spectrum of **10a_{red}** in CDCl₃-CD₃OD at 100 MHz

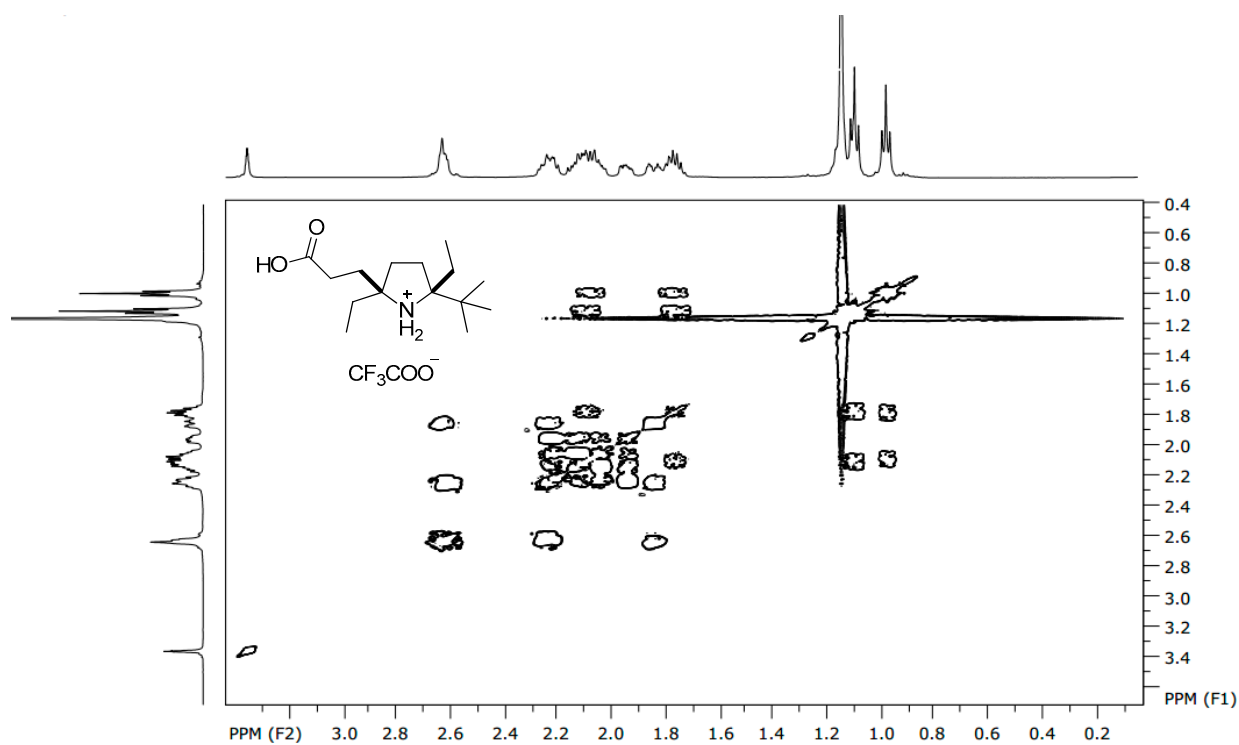


Figure S27. ¹H-¹H COSY NMR spectrum of **10a_{red}** in CDCl₃-CD₃OD

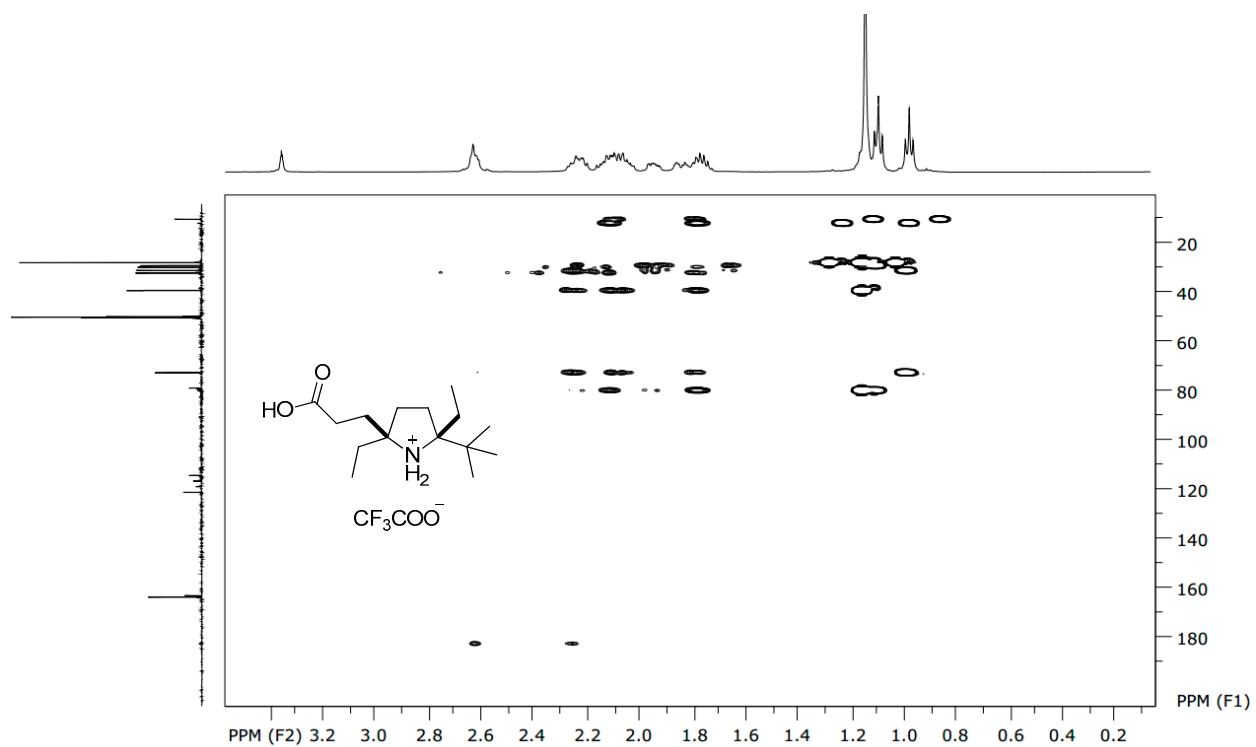


Figure S28. ^1H - ^{13}C HMBC NMR spectrum of **10a_{red}** in CDCl_3 - CD_3OD

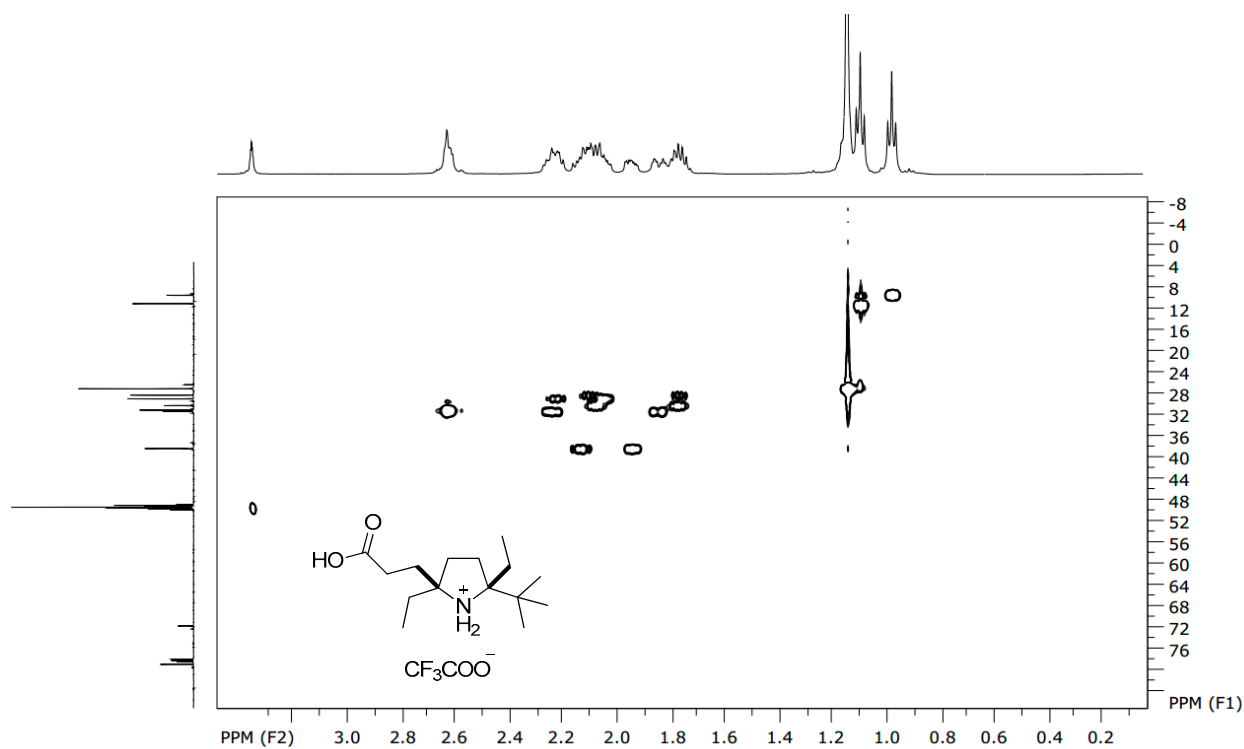


Figure S29. ^1H - ^{13}C HSQC NMR spectrum of **10a_{red}** in CDCl_3 - CD_3OD

2.8 3-(5-*tert*-Butyl-2-ethyl-1-oxido-3,4-dihydro-2*H*-pyrrol-2-yl)-1-propanol (**11a**)

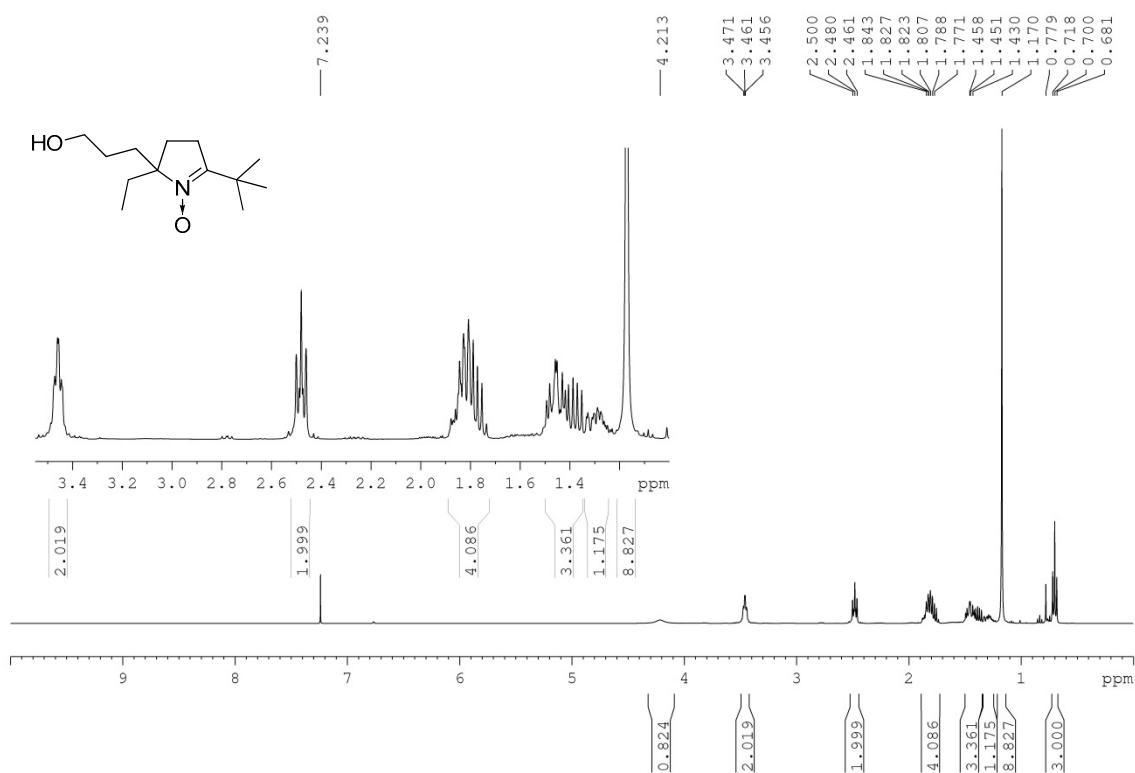


Figure S30. ¹H NMR spectrum of **11a** in CDCl₃ at 400 MHz

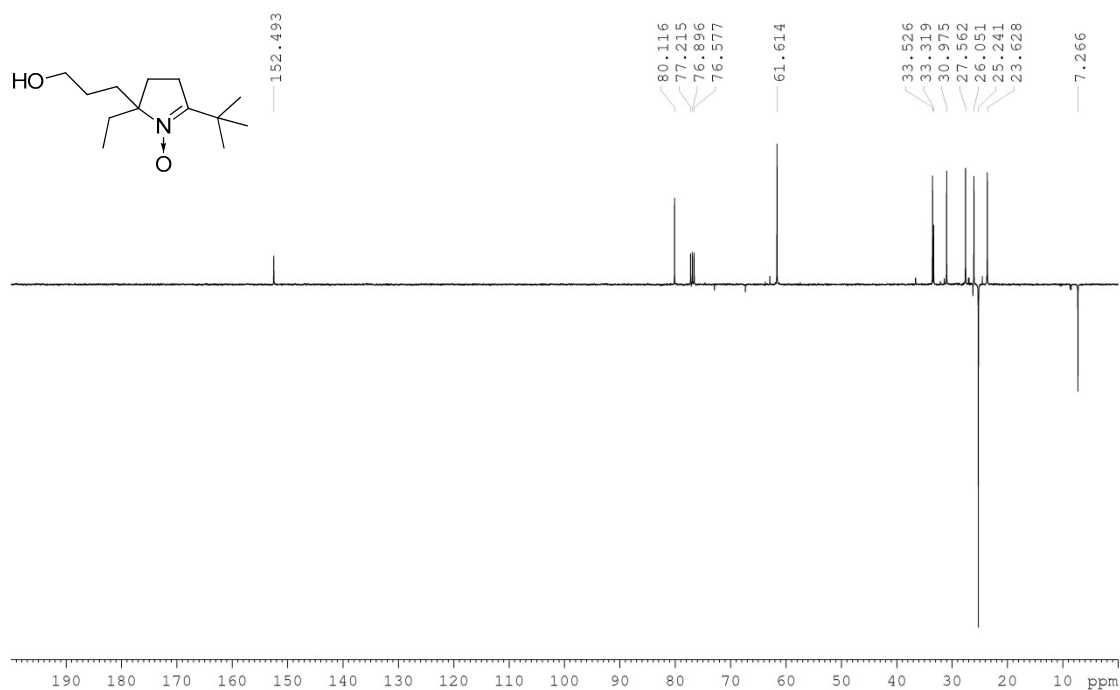


Figure S31. ¹³C NMR spectrum of **11a** in CDCl₃ at 100 MHz

2.9 3,3'-(5-*tert*-Butyl-1-oxido-3,4-dihydro-2*H*-pyrrole-2,2-diyl)di(1-propanol) (**11b**)

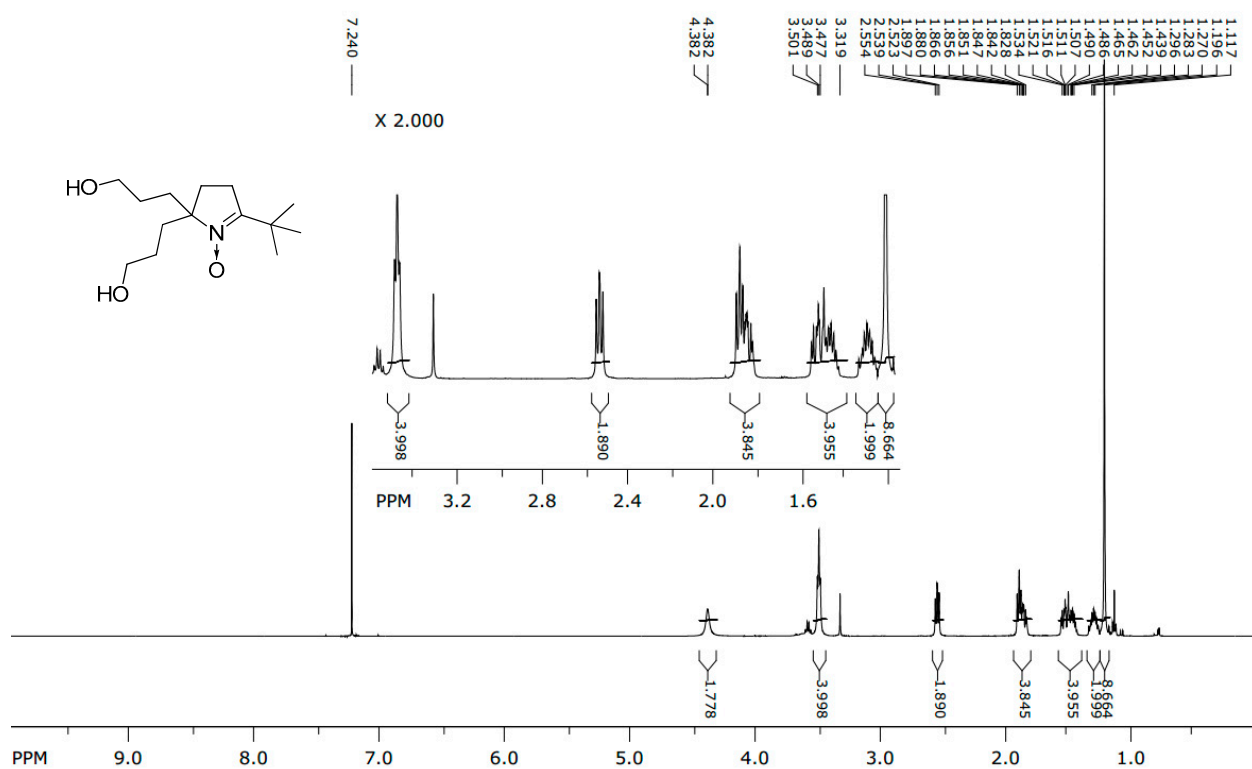


Figure S32. ¹H NMR spectrum of **11b** in CDCl₃ at 500 MHz

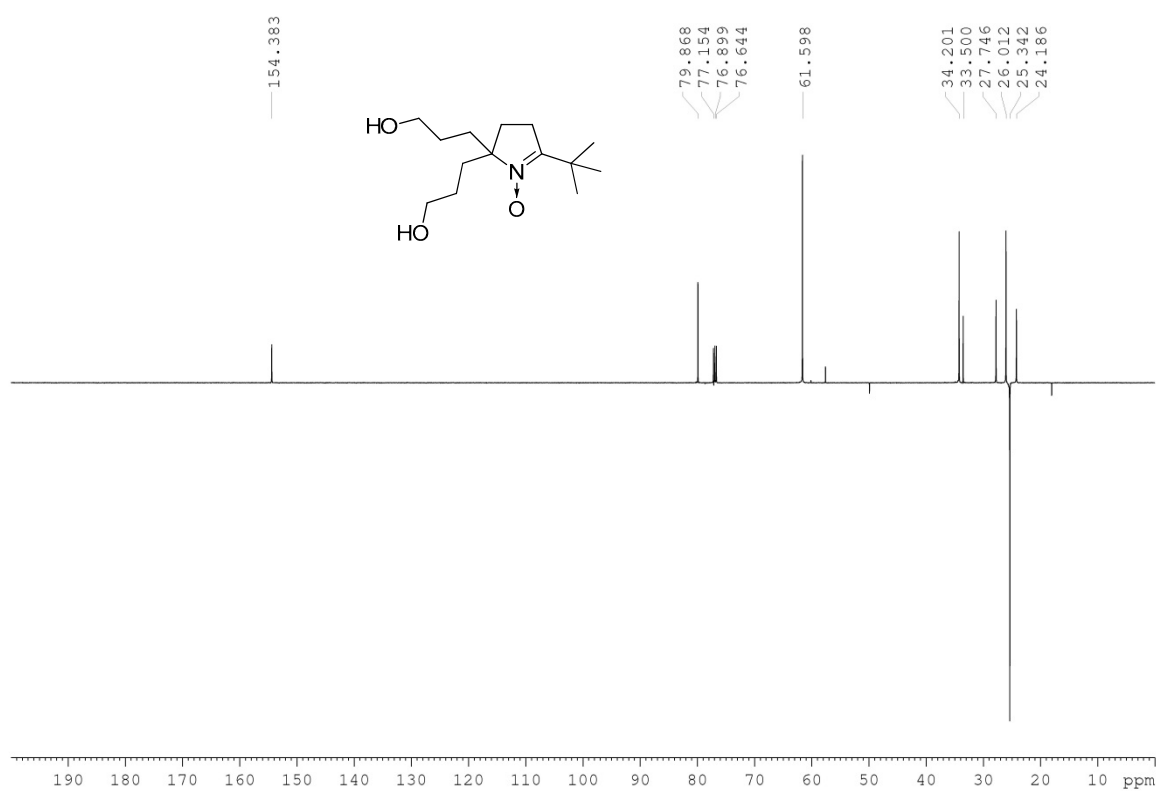


Figure S33. ¹³C NMR spectrum of **11b** in CDCl₃ at 125 MHz

2.10 5-*tert*-Butyl-2-ethyl-2-[3-(1-methoxy-1-methylethoxy)propyl]-3,4-dihydro-2*H*-pyrrole 1-oxide (**12a**)

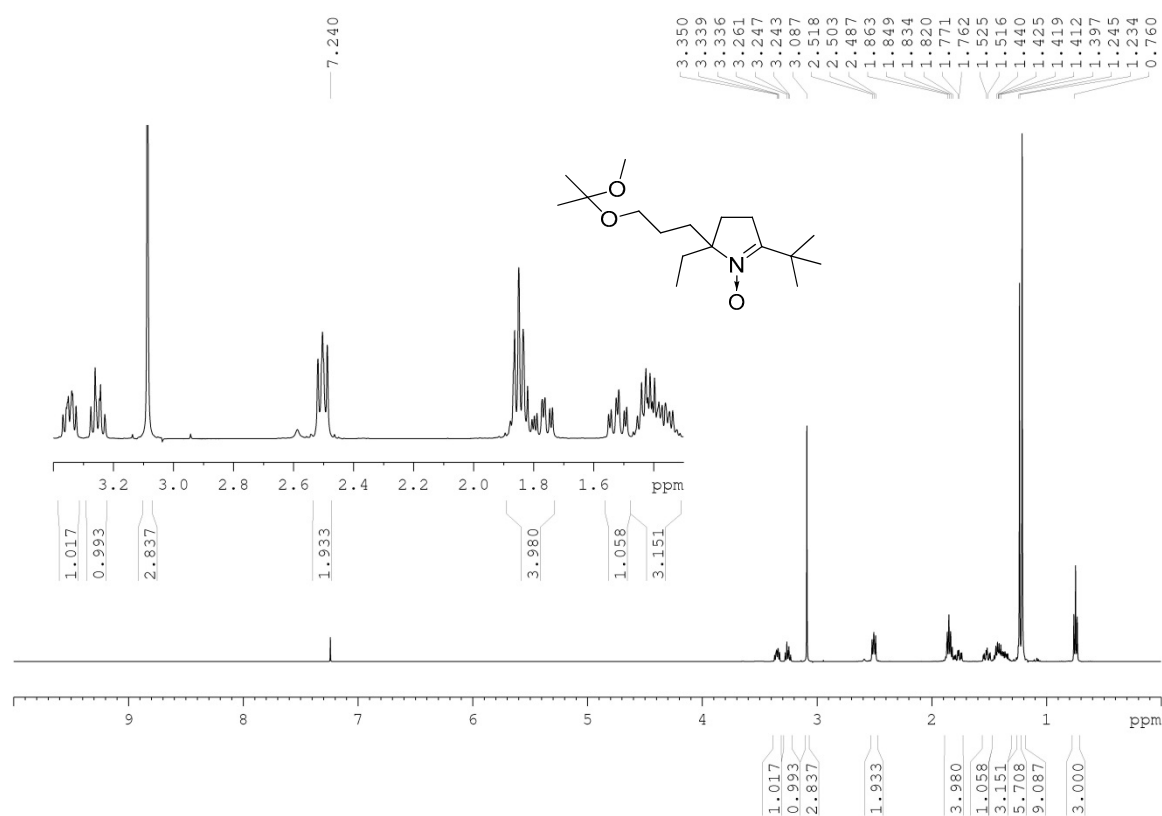


Figure S34. ¹H NMR spectrum of **12a** in CDCl₃ at 500 MHz

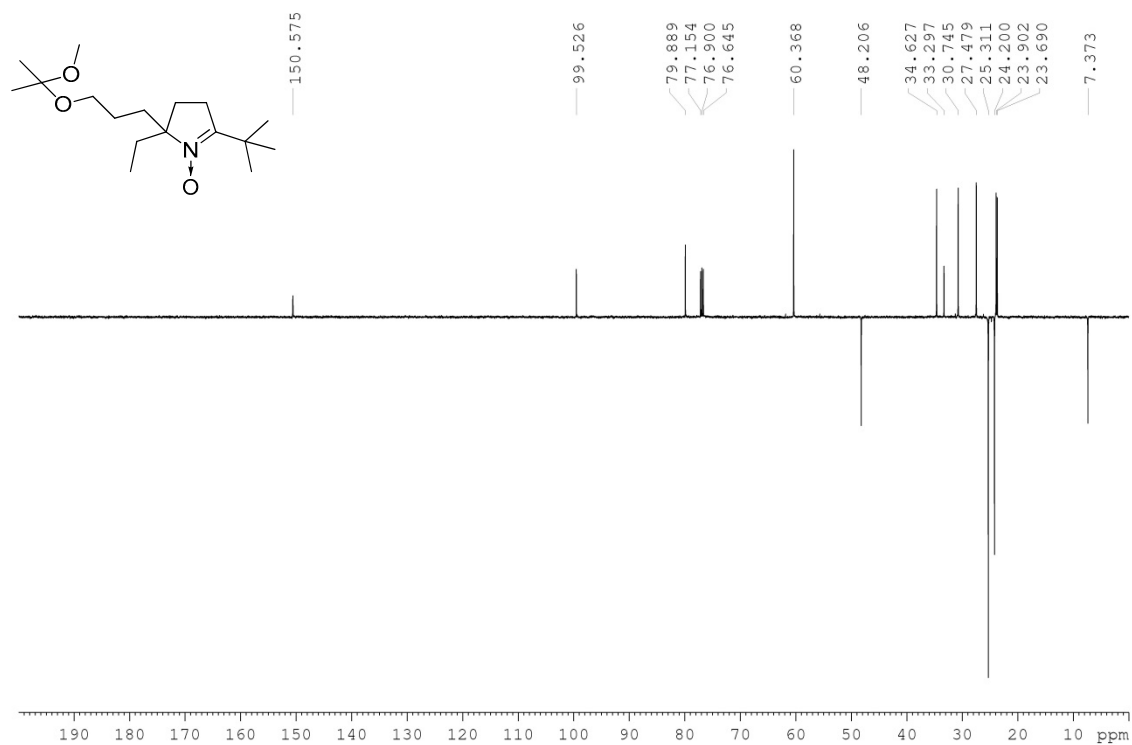


Figure S35. ¹³C NMR spectrum of **12a** in CDCl₃ at 125 MHz

2.11 5-*tert*-Butyl-2,2-bis[3-(1-methoxy-1-methylethoxy)propyl]-3,4-dihydro-2*H*-pyrrole 1-oxide (**12b**)

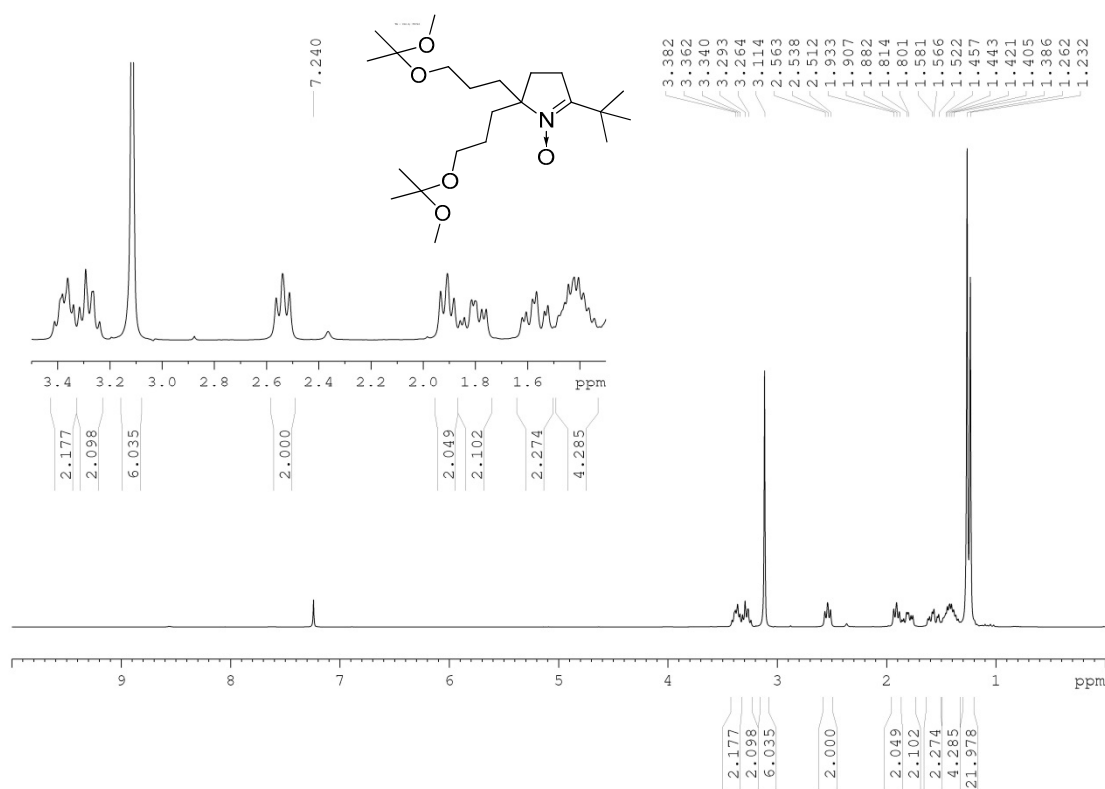


Figure S36. ¹H NMR spectrum of **12b** in CDCl₃ at 300 MHz

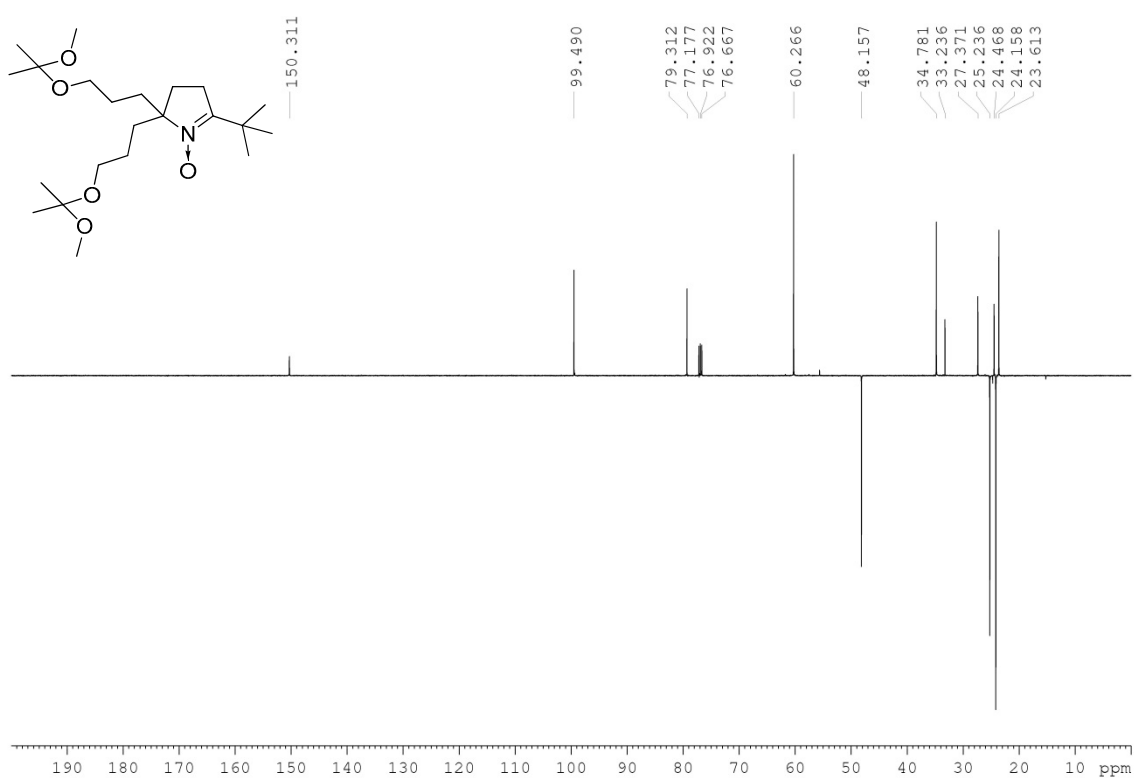


Figure S37. ¹³C NMR spectrum of **12b** in CDCl₃ at 125 MHz

2.12 2-*tert*-Butyl-2,5-diethyl-5-(3-hydroxypropyl)pyrrolidinium trifluoroacetate (mixture of diastereomers) (**14a_{red}**)

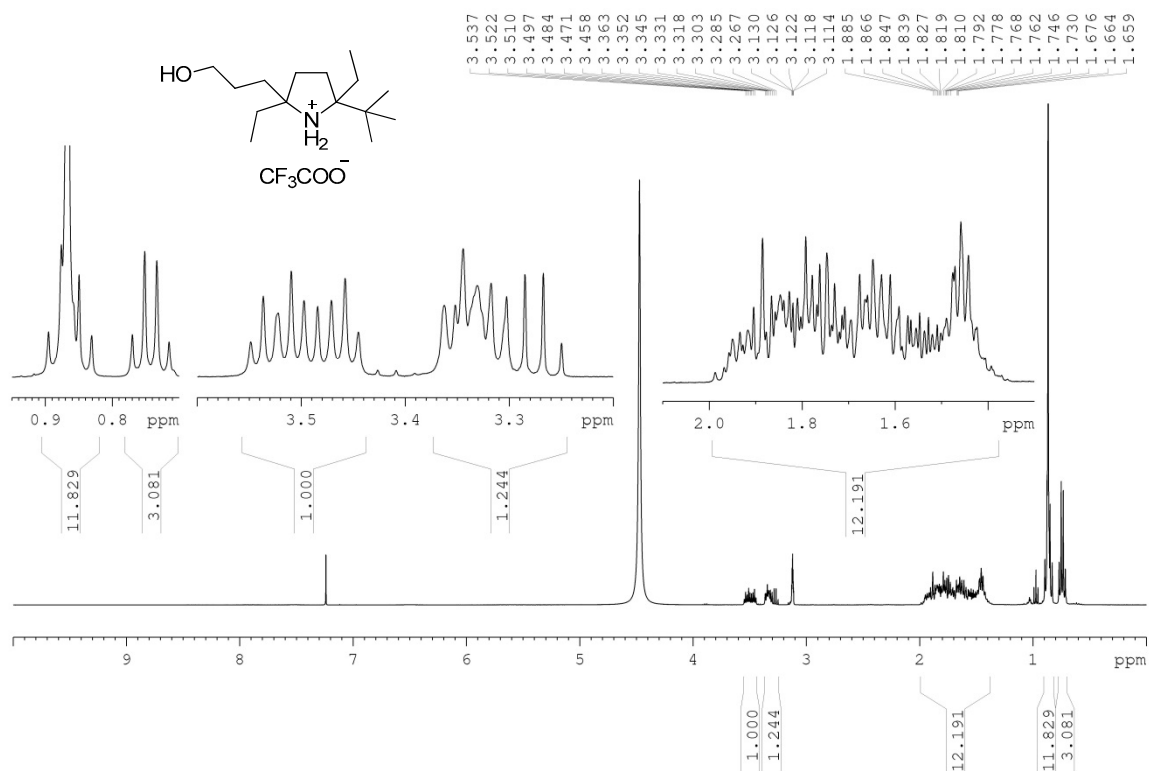


Figure S38. ¹H NMR spectrum of **14a_{red}** in CDCl₃-CD₃OD at 400 MHz

2.13 2-*tert*-Butyl-2-ethyl-5,5-bis(3-hydroxypropyl)pyrrolidinium trifluoroacetate (**14b_{red}**)

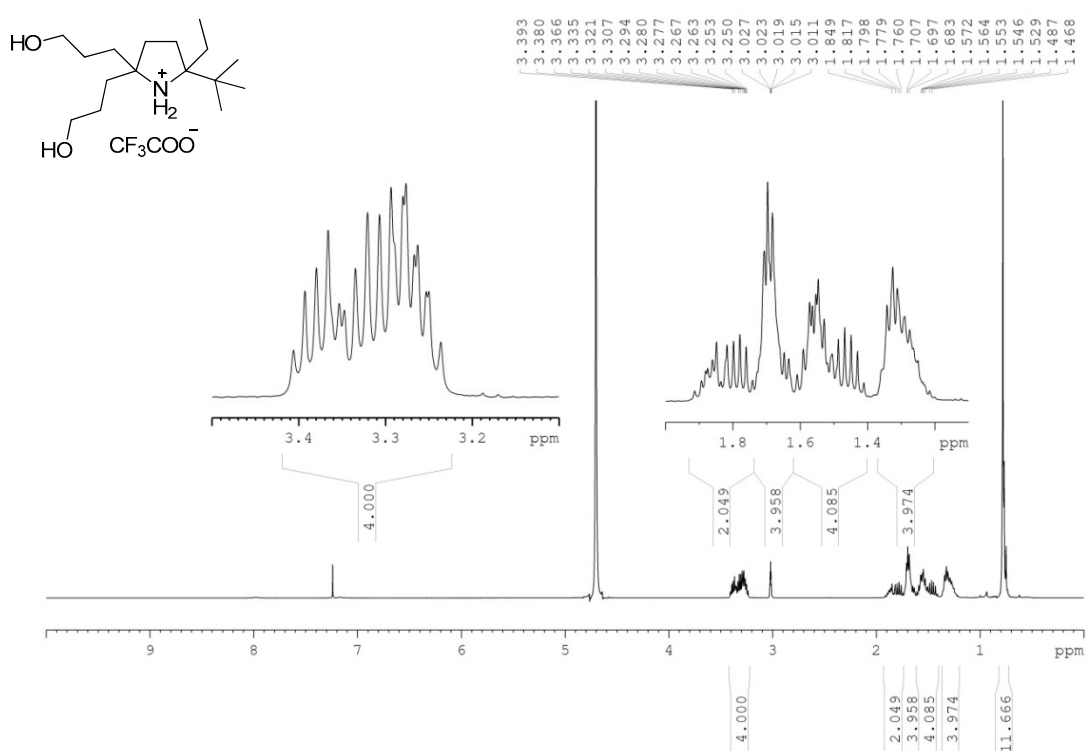


Figure S39. ¹H NMR spectrum of **14b_{red}** in CDCl₃-CD₃OD at 400 MHz

3. HPLC analysis

3.1 HPLC analysis of 2-*tert*-Butyl-2-ethyl-5,5-bis(3-hydroxypropyl)pyrrolidine-1-oxyl (**14a**)

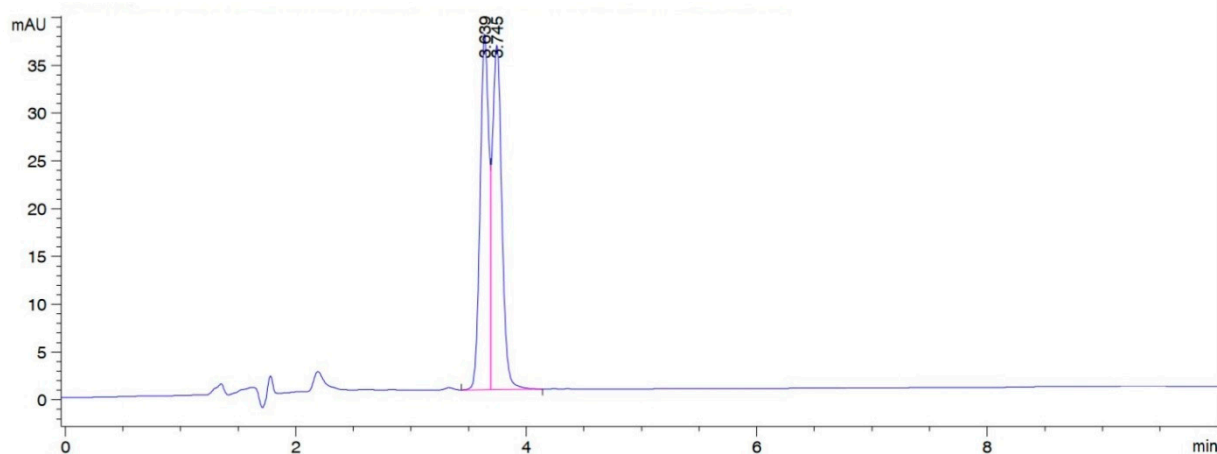


Figure S40. The HPLC analysis of **14a**