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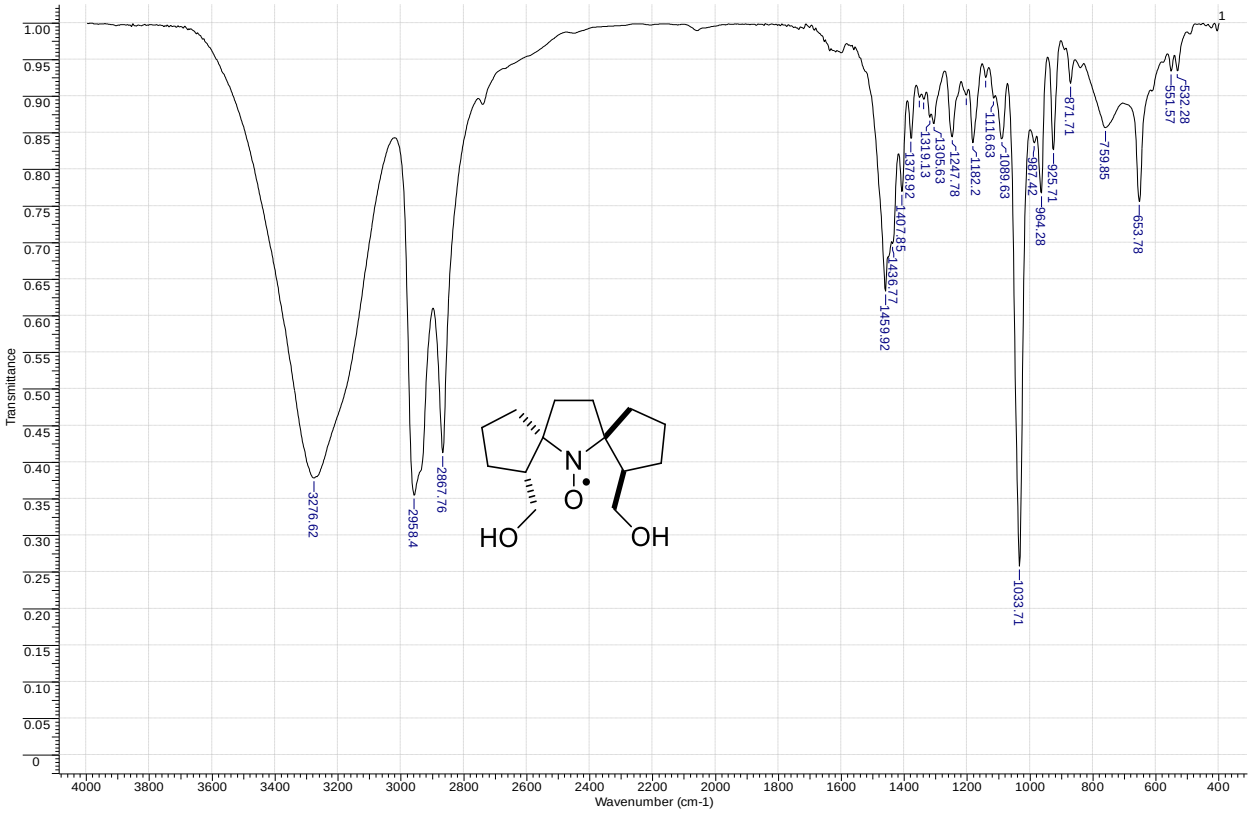


**Fig. S1.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis(hydroxymethyl)-6-azadispiro[4.1.4.2]tridecane 6-oxyl (**5**) (KBr)

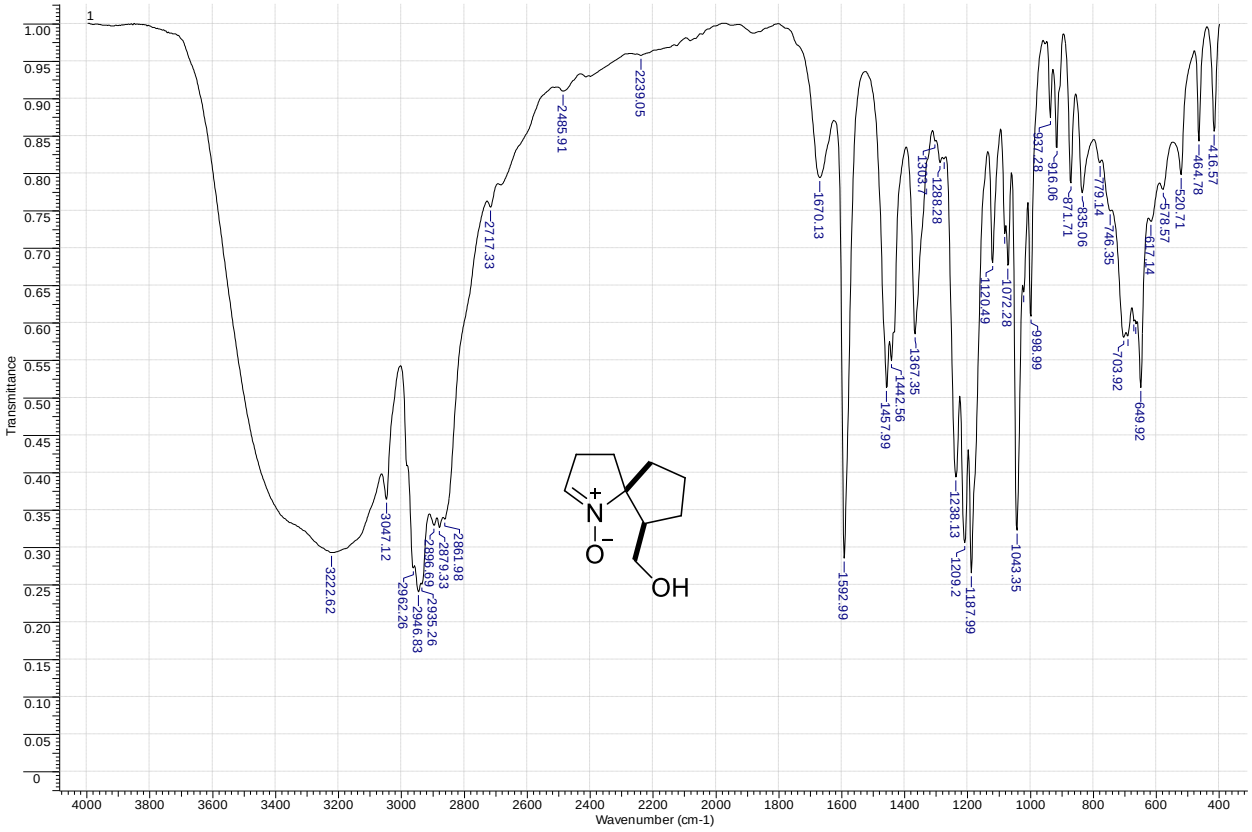


**Fig. S2.** IR spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-1-azaspiro[4.4]non-1-en-6-yl]methanol (**9**) (KBr)

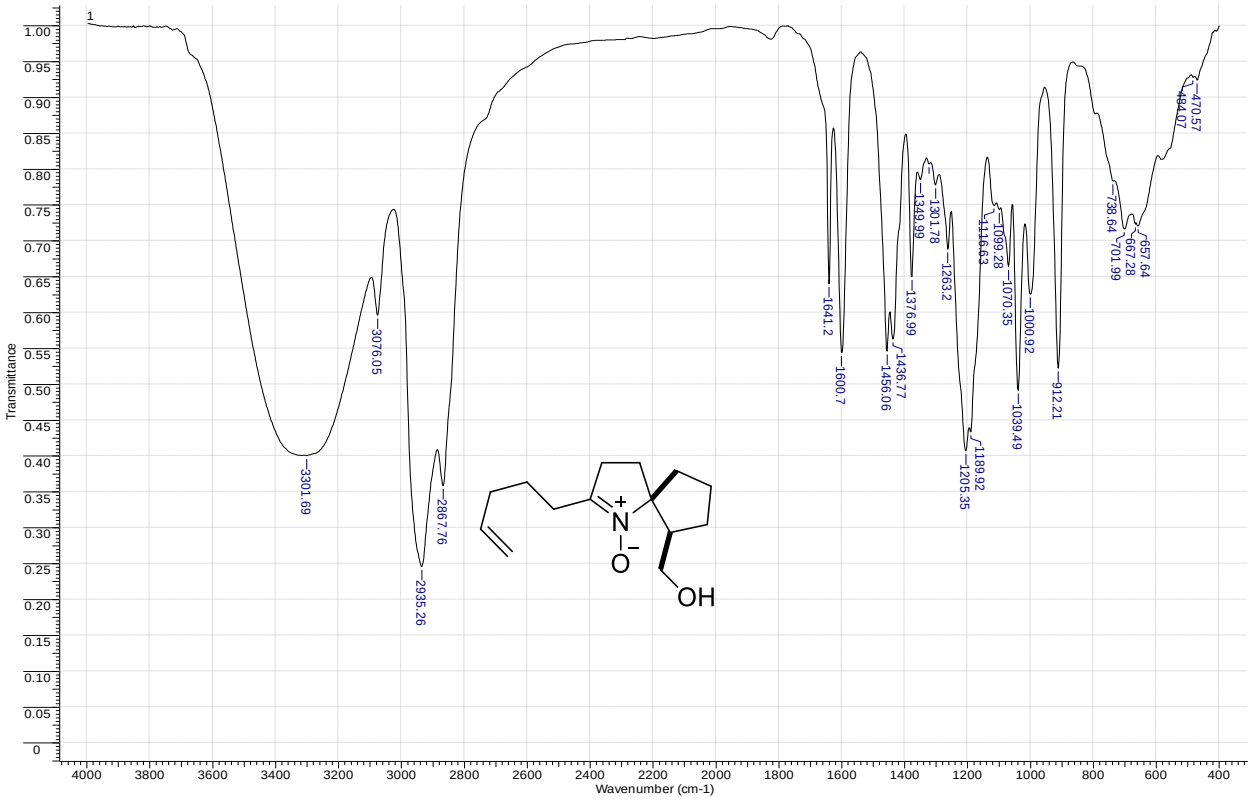


**Fig. S3.** IR spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-2-pent-4-en-1-yl-1-azaspiro[4.4]non-1-en-6-yl]methanol (**12**) (neat)

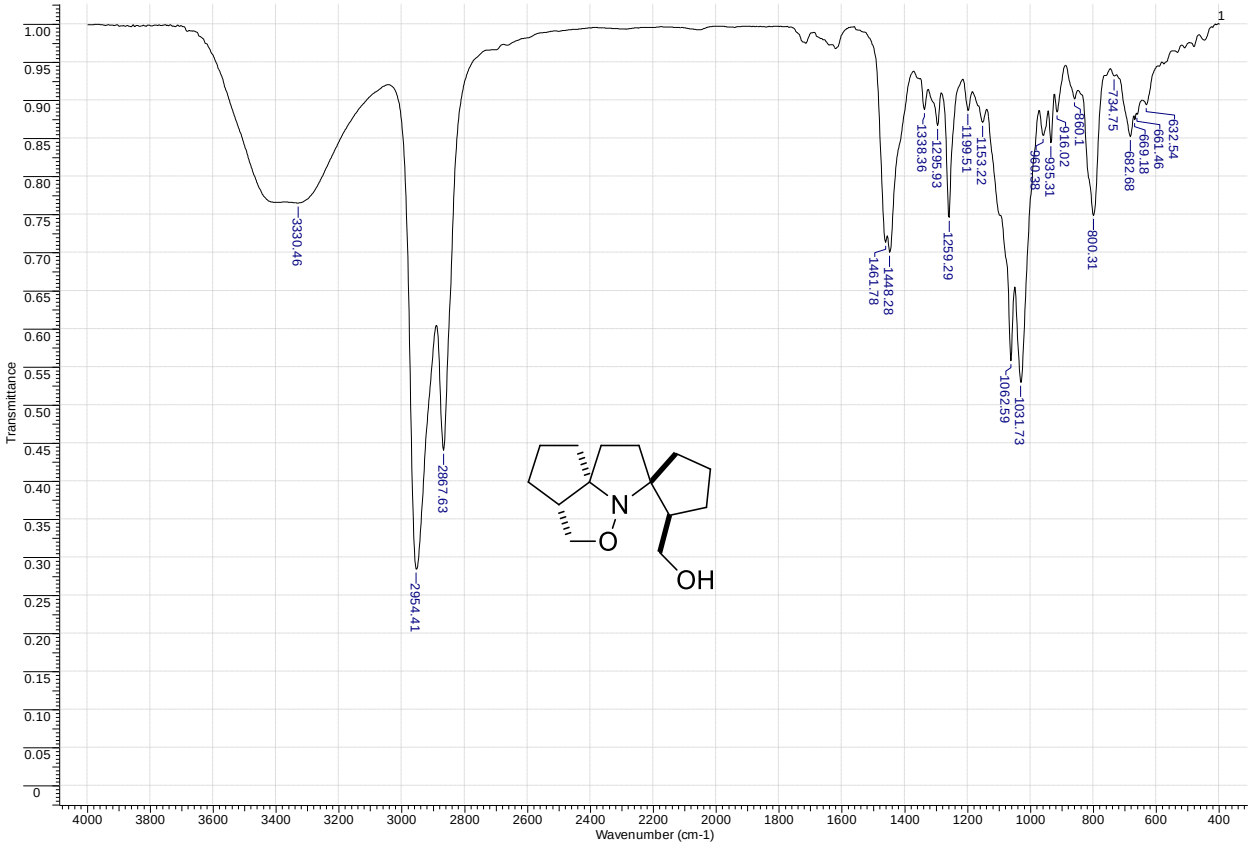


**Fig. S4.** IR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*))-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) (neat)

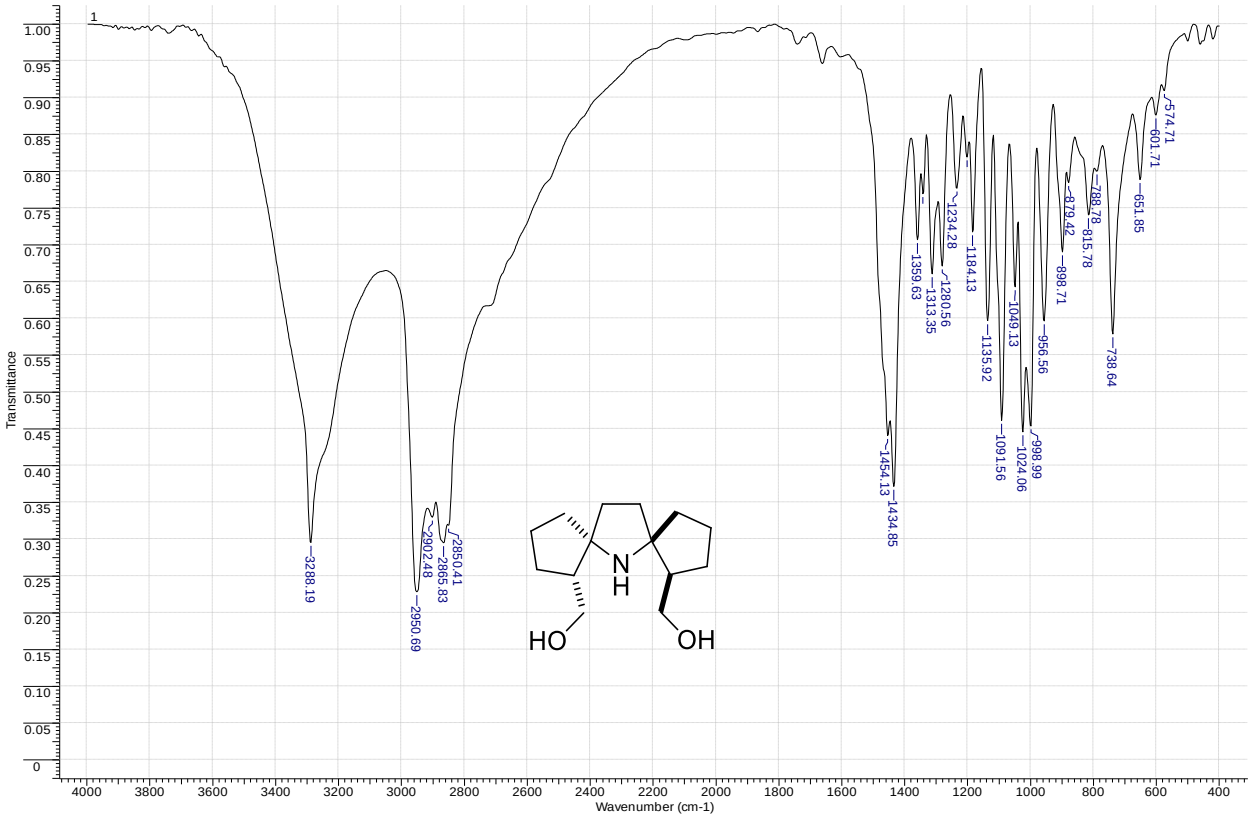


**Fig. S5.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-diyldimethanol (**14**) (KBr)


**Fig. S6.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-diyldi(methylene) diacetate (**16**) (neat)

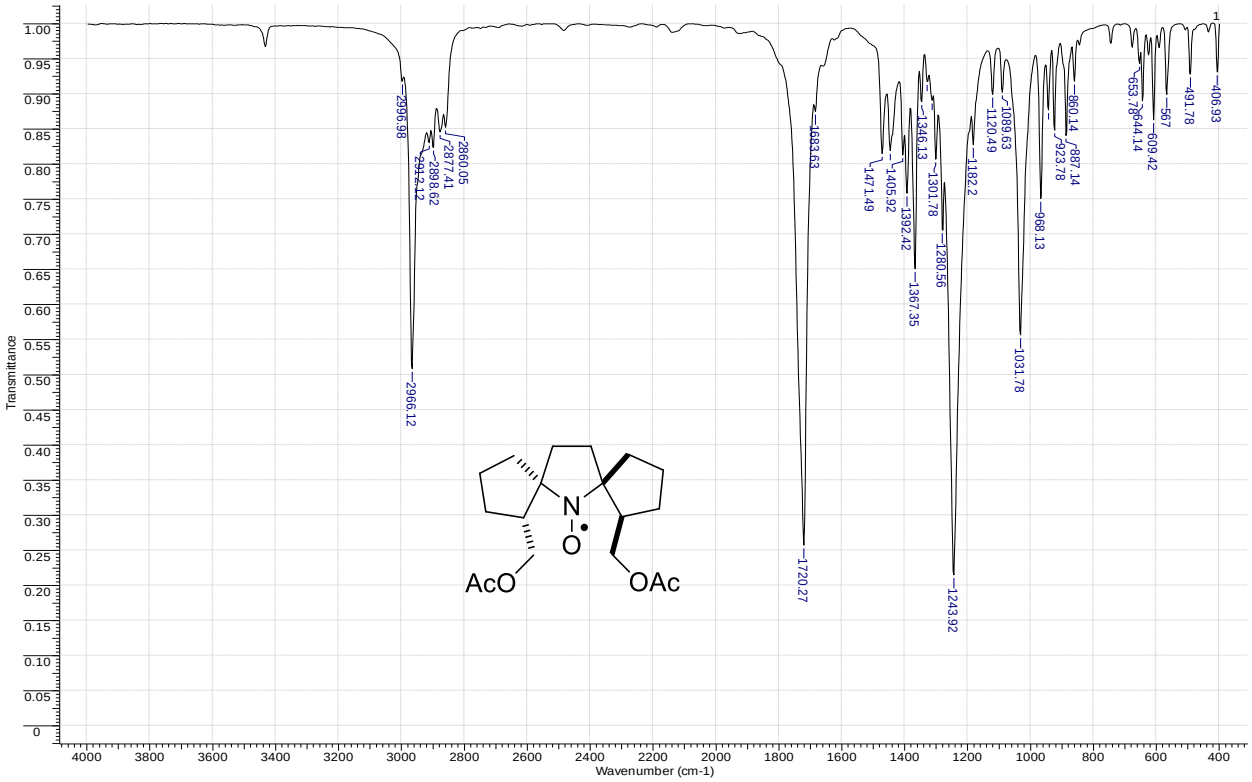


**Fig. S7.** IR spectrum of 1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(acetyloxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**17**) (KBr)

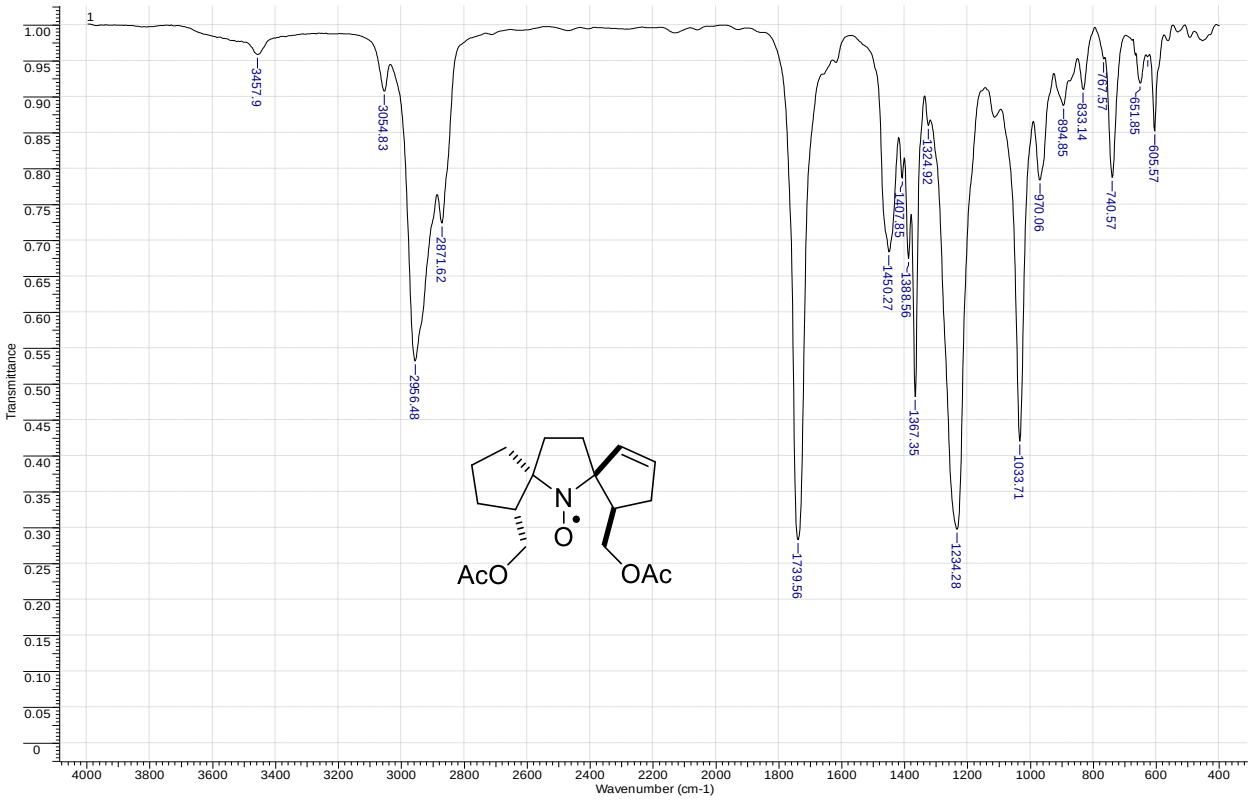


**Fig. S8.** IR spectrum of (4*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-4,8-Bis[(acetyloxy)methyl]-6-azadispiro[4.1.4.2]tridec-1-ene-6-oxyl (**18**) (neat)

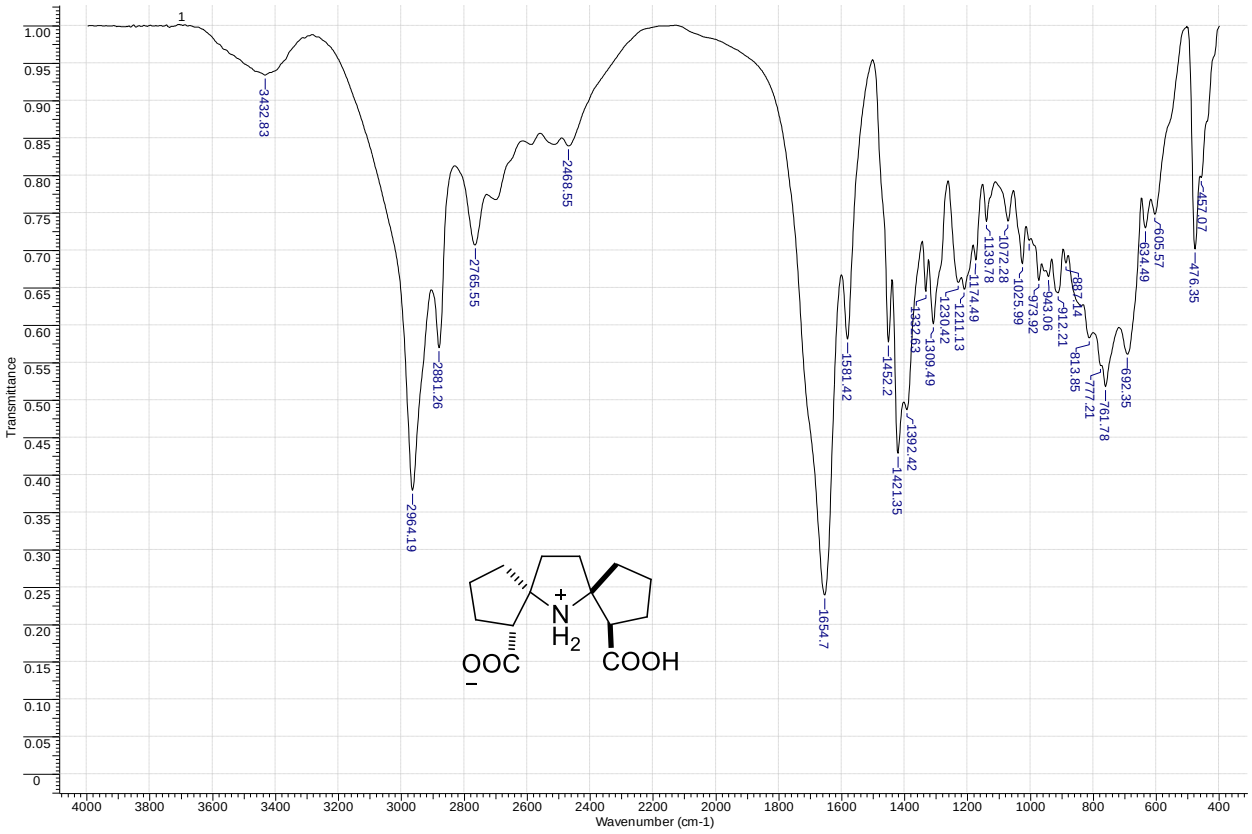


**Fig. S9.** IR spectrum of. (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-Azadispiro[4.1.4.2]tridecan-1,8-dicarboxylic acid **20** (KBr)

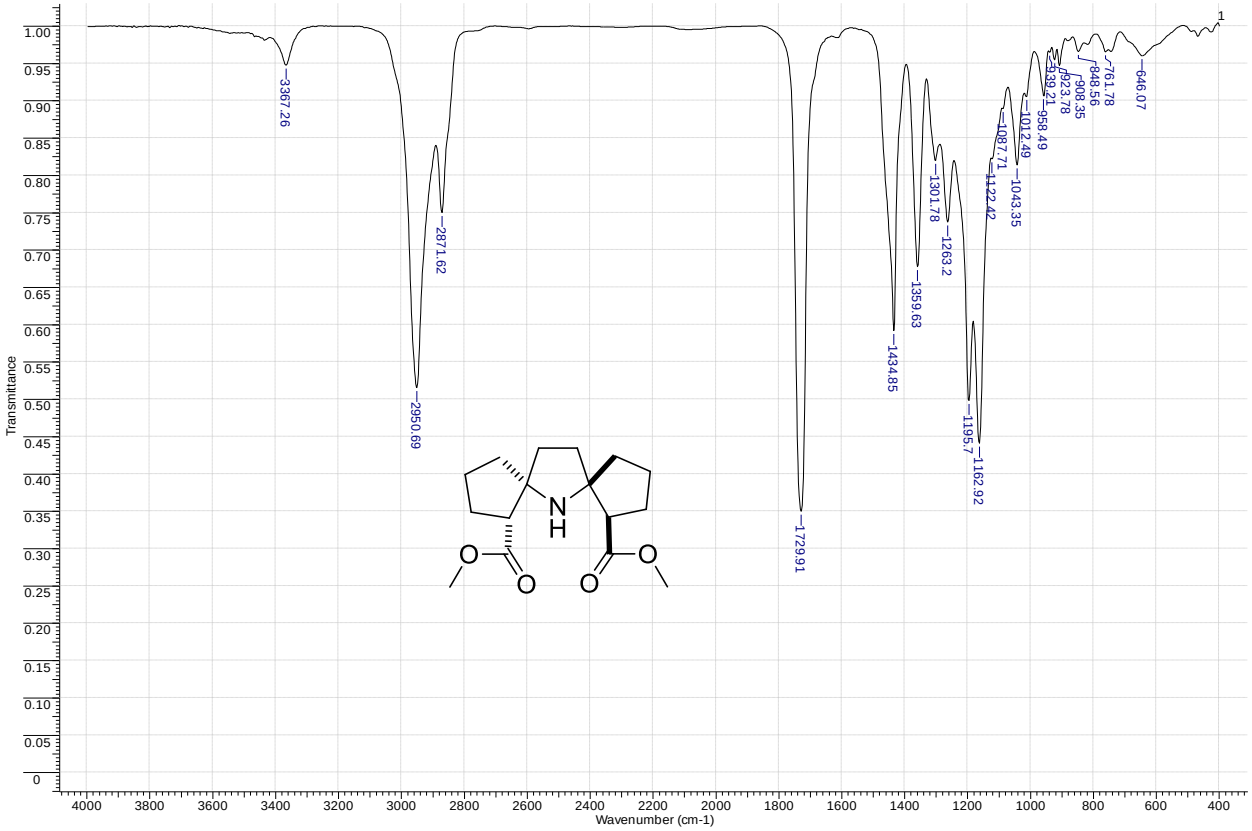


**Fig. S10.** IR spectrum of Dimethyl (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-dicarboxylate (**21**) (neat)

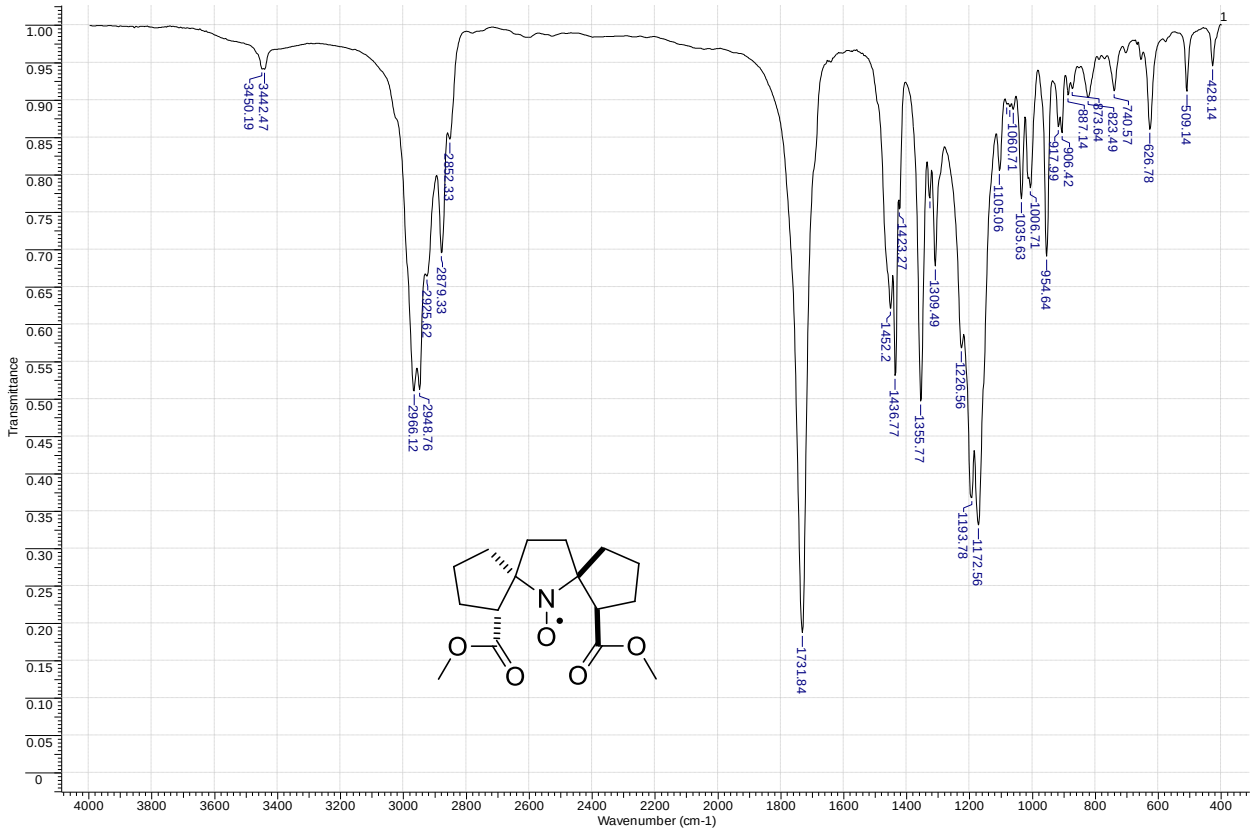


**Fig. S11.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis(methoxycarbonyl)-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**22**) (KBr)


**Fig. S12.** IR spectrum of 1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Dicarboxy-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**23a**) (KBr)

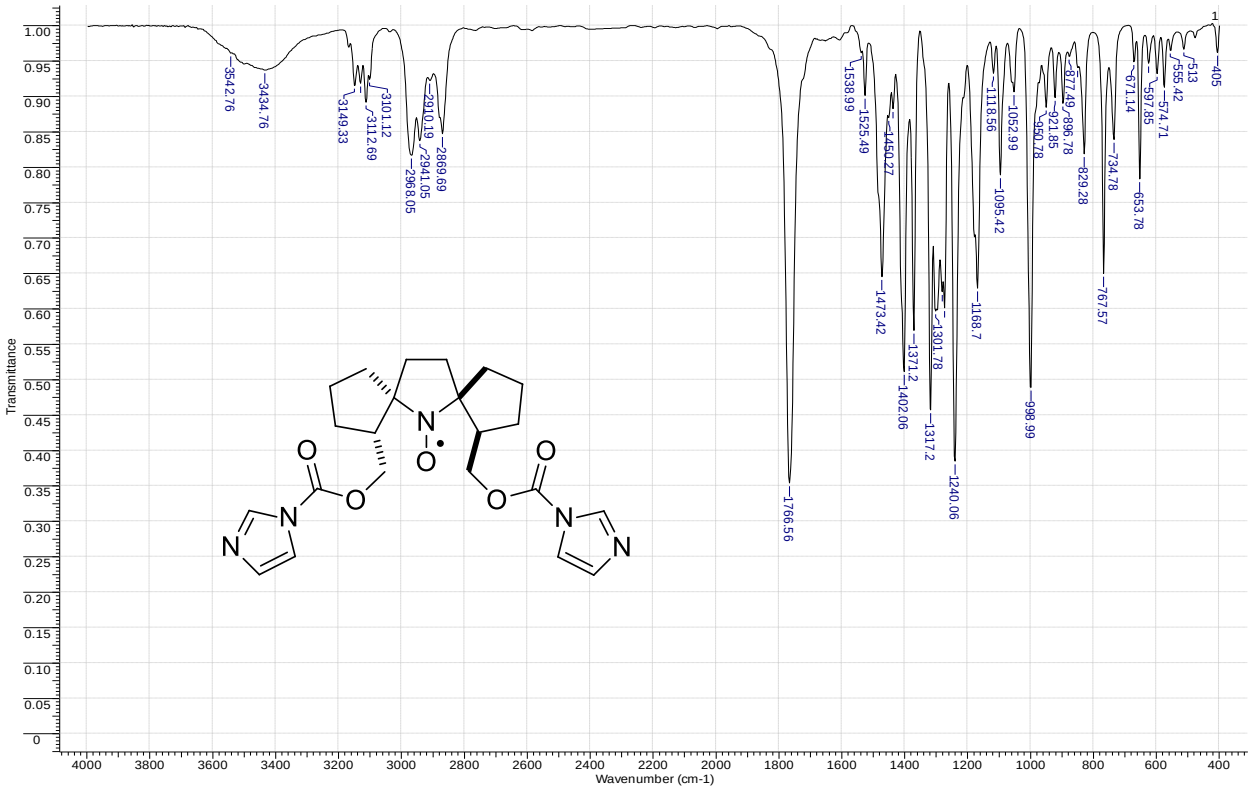


**Fig. S13.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[({1*H*-imidazol-1-ylcarbonyl}oxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**26**) (KBr)

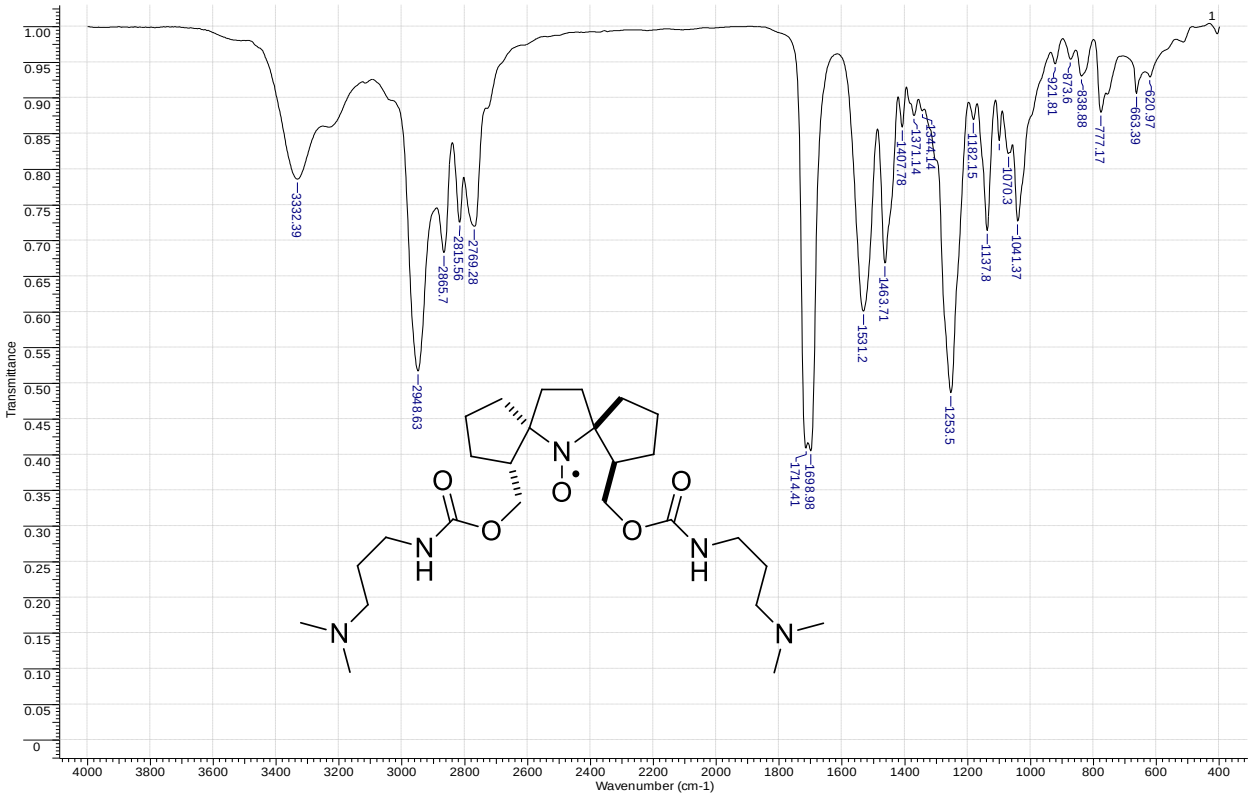


**Fig. S14.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(([{3-(dimethylamino)propyl}amino]carbonyl)oxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**27**) (neat)

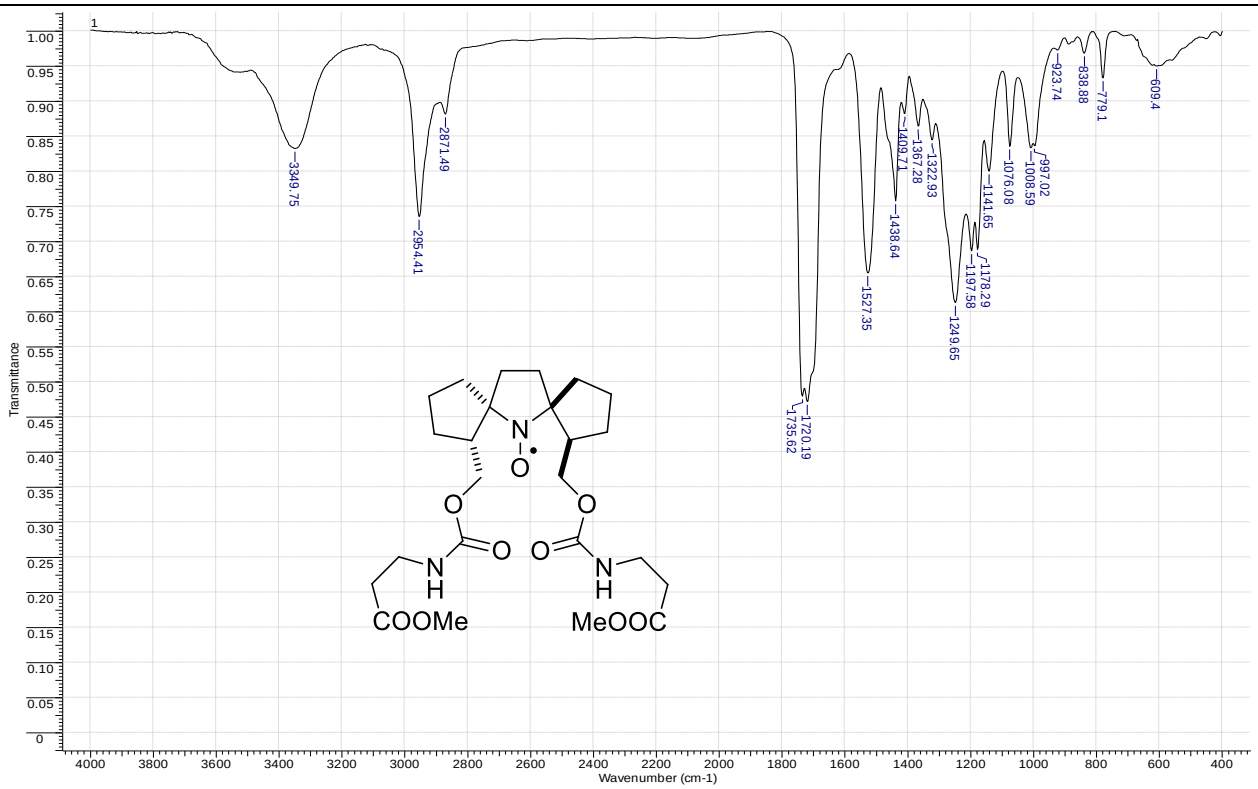


**Fig. S15.** IR spectrum of 1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*)-1,8-Bis[(([{3-methoxy-3-oxopropyl}amino]carbonyl)oxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**28**) (neat)

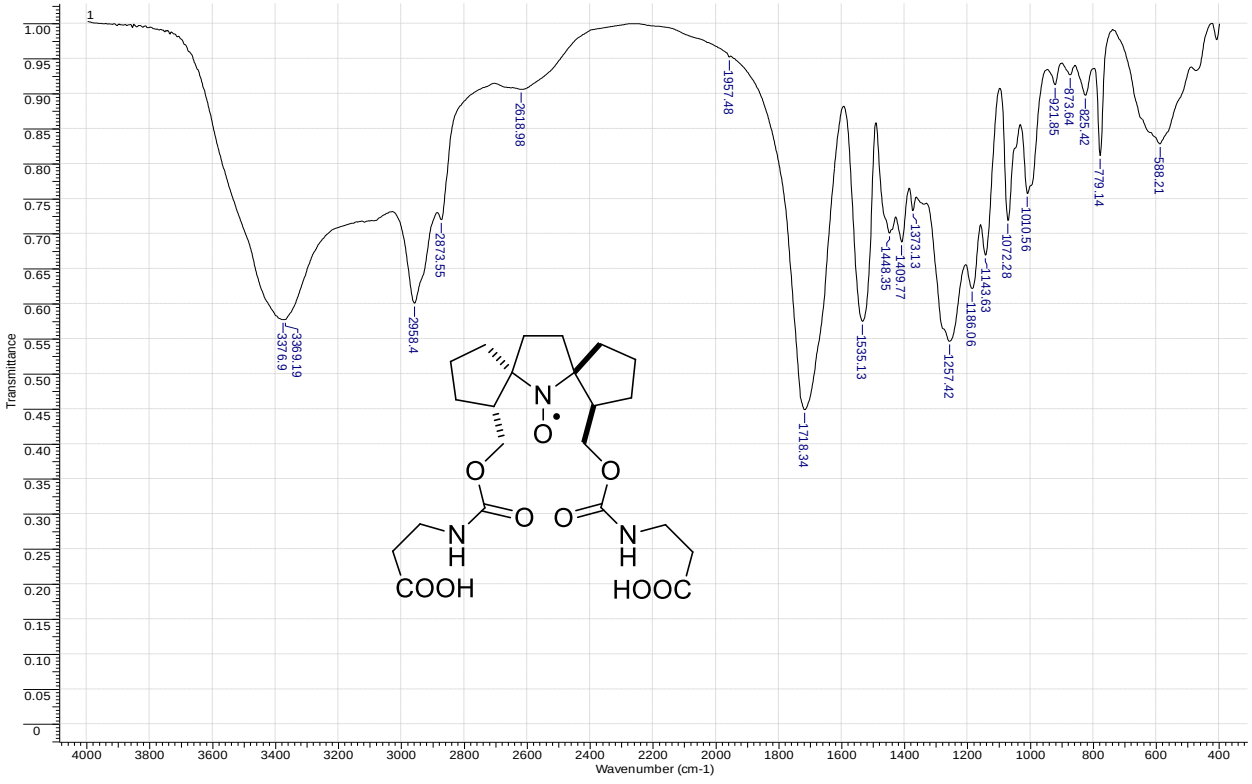


**Fig. S16.** IR spectrum of 1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*)-1,8-Bis[(([{2-carboxyethyl}amino]carbonyl)oxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**29**) (KBr)


**Fig. S17.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(prop-2-yn-1-yloxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**30**) (neat)

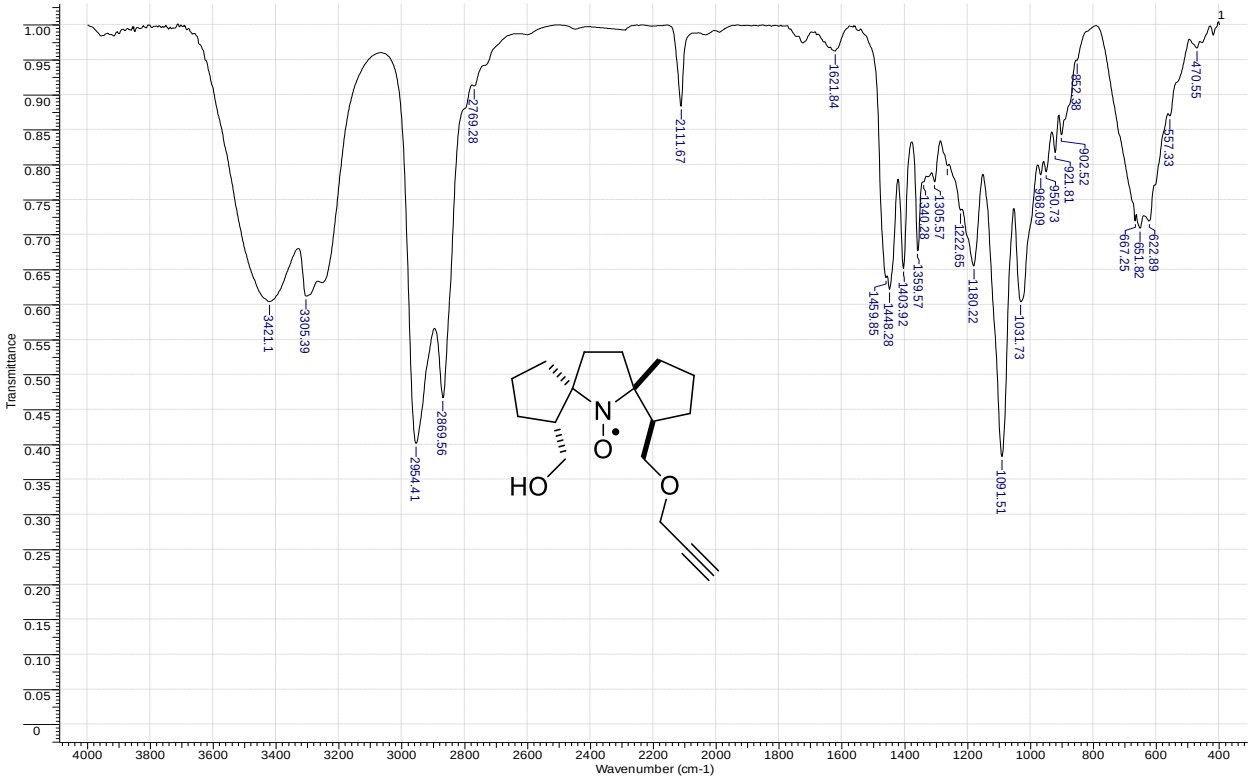


**Fig. S18.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-[(prop-2-yn-1-yloxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**31**) (neat)

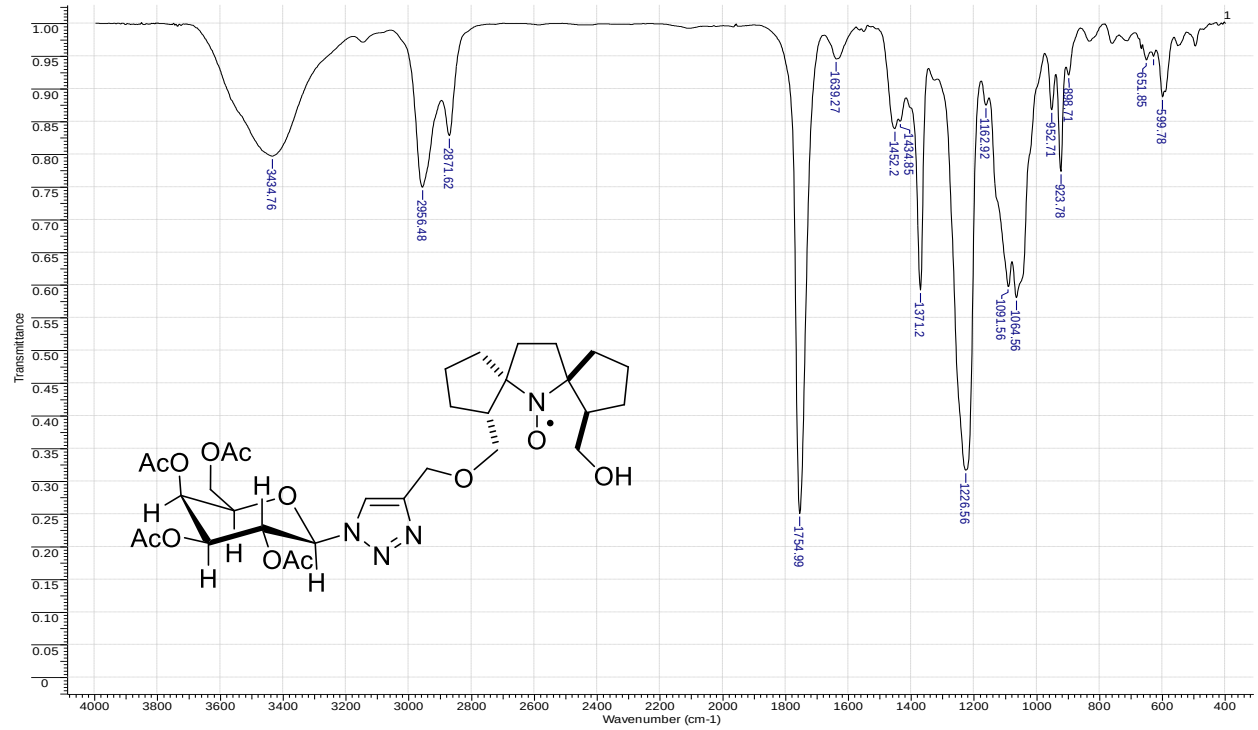


**Fig. S19.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl-8-({(1-[2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl]-1*H*-1,2,3-triazol-4-yl)methoxy}methyl)-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**32**) (KBr)

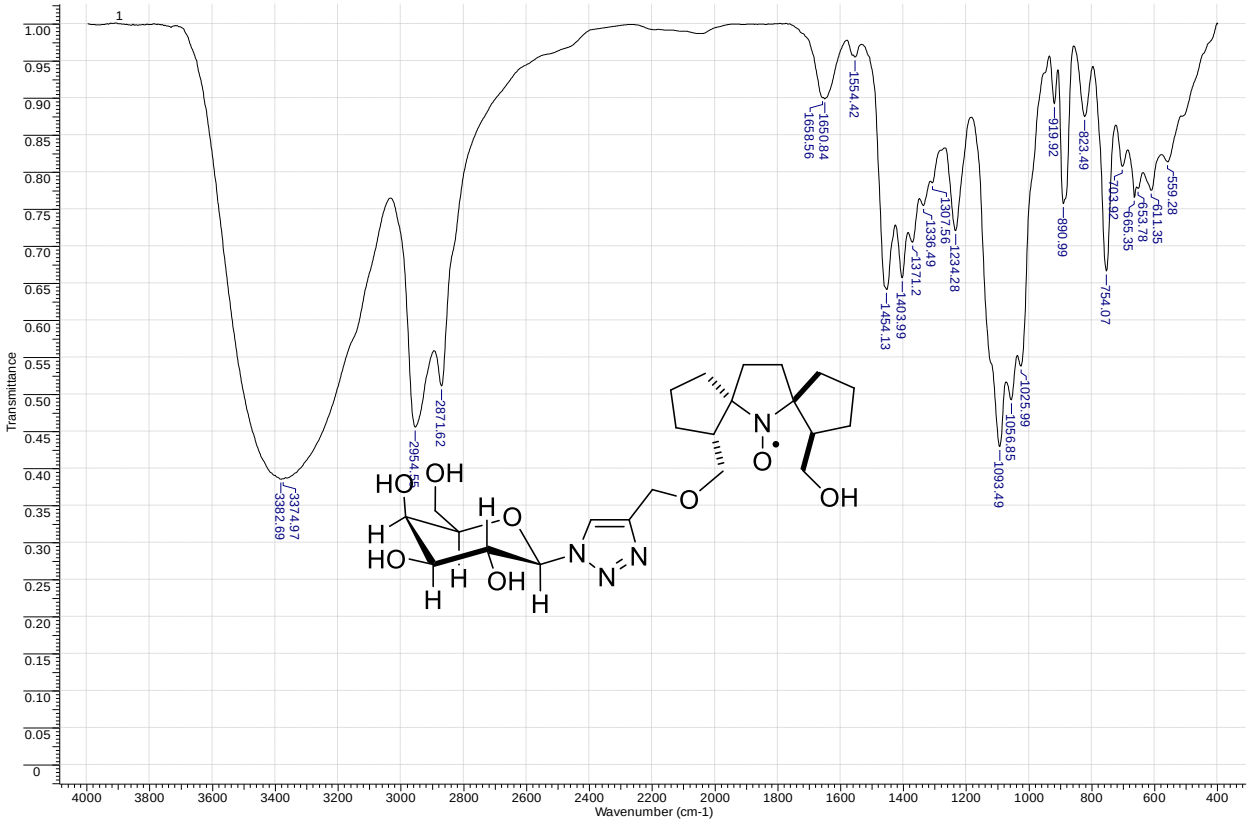


**Fig. S20.** IR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-({(1-[β-D-galactopyranosyl]-1*H*-1,2,3-triazol-4-yl)methoxy}methyl)-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**33**) (KBr)

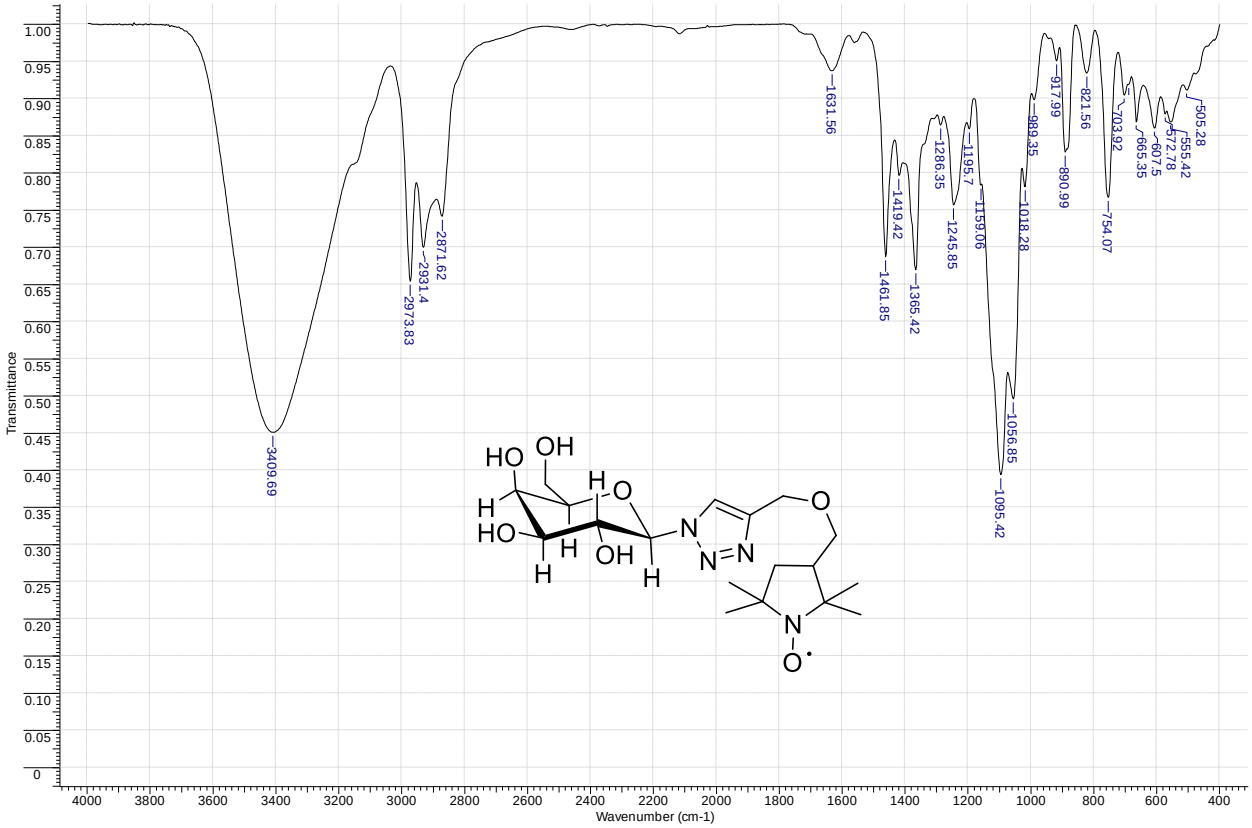


**Fig. S21.** IR spectrum of 2,2,5,5-Tetramethyl-3-(((1-(β-D-galactopyranosyl)-1*H*-1,2,3-triazol-4-yl)methoxy)methyl)pyrrolidine-1-oxyl (**35**) (KBr)

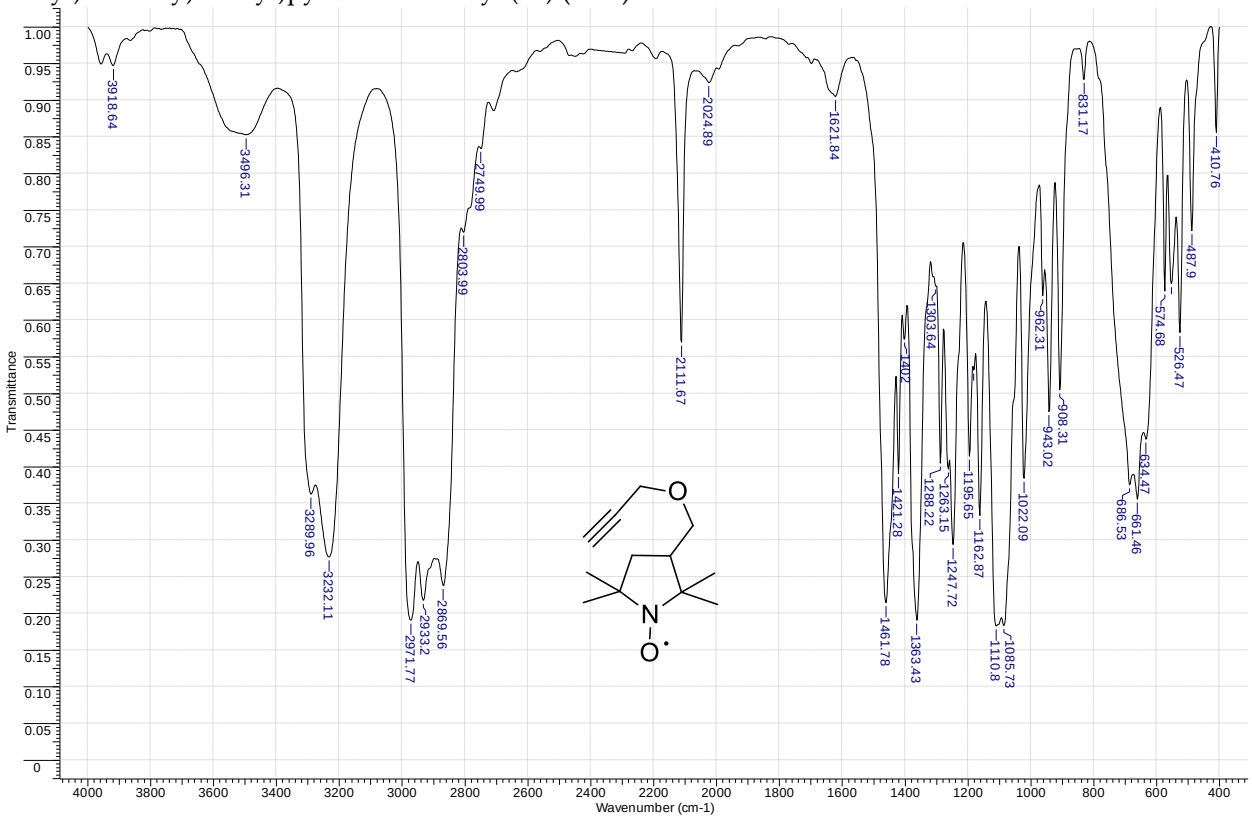


**Fig. S22.** IR spectrum of 2,2,5,5-Tetramethyl-3-((prop-2-ynyloxy)methyl)pyrrolidine-1-oxyl (**37**) (KBr)

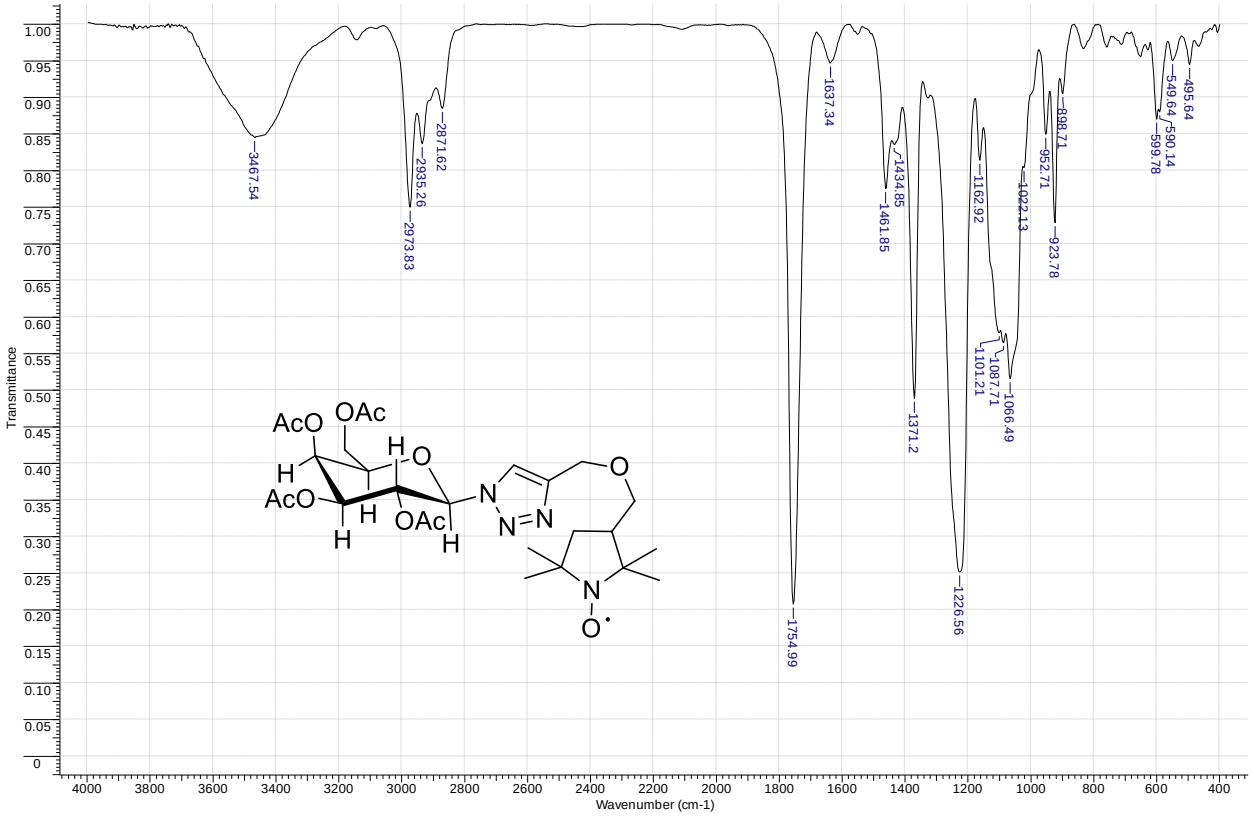


**Fig. S23.** IR spectrum of 2,2,5,5-Tetramethyl-3-(((1-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl)-1*H*-1,2,3-triazol-4-yl)methoxy)methyl)pyrrolidine-1-oxyl (**38**) (KBr)

## 3. UV spectral data

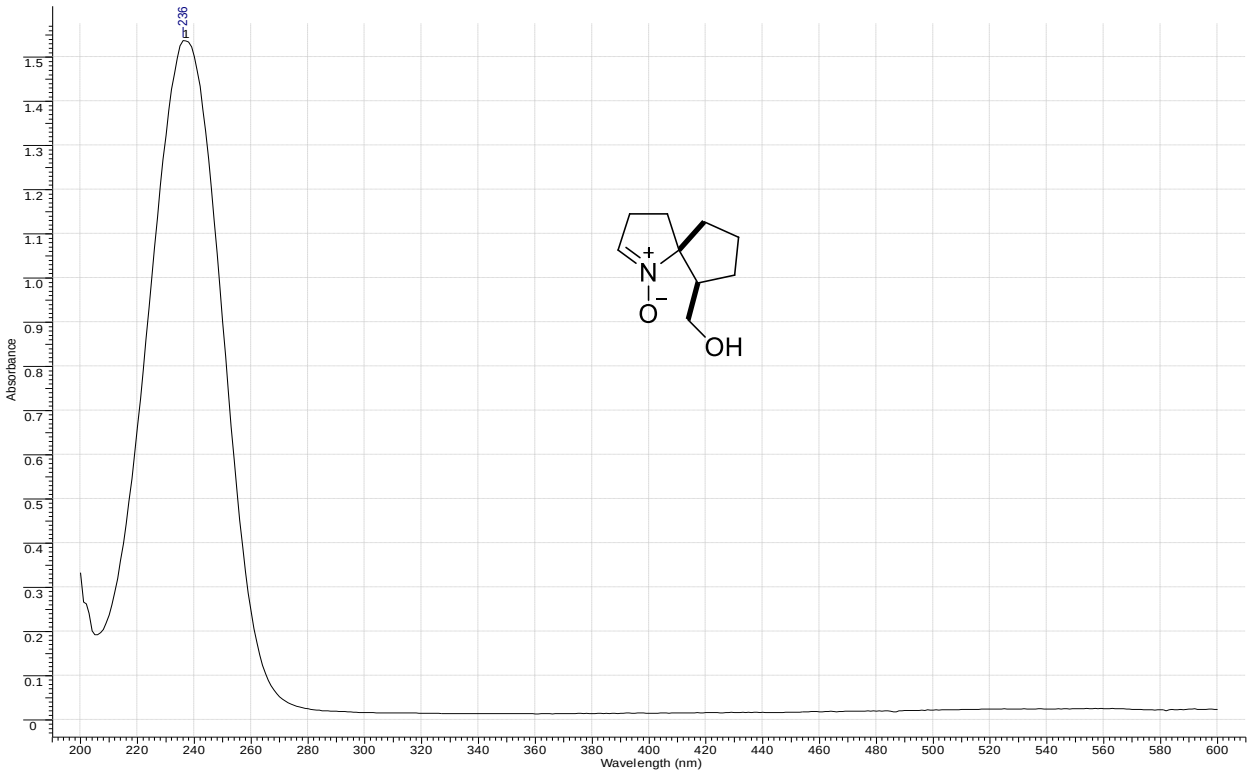


**Fig. S24.** UV spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-1-azaspiro[4.4]non-1-en-6-yl]methanol (**9**) in EtOH (0.429mg/25ml, L=2cm)

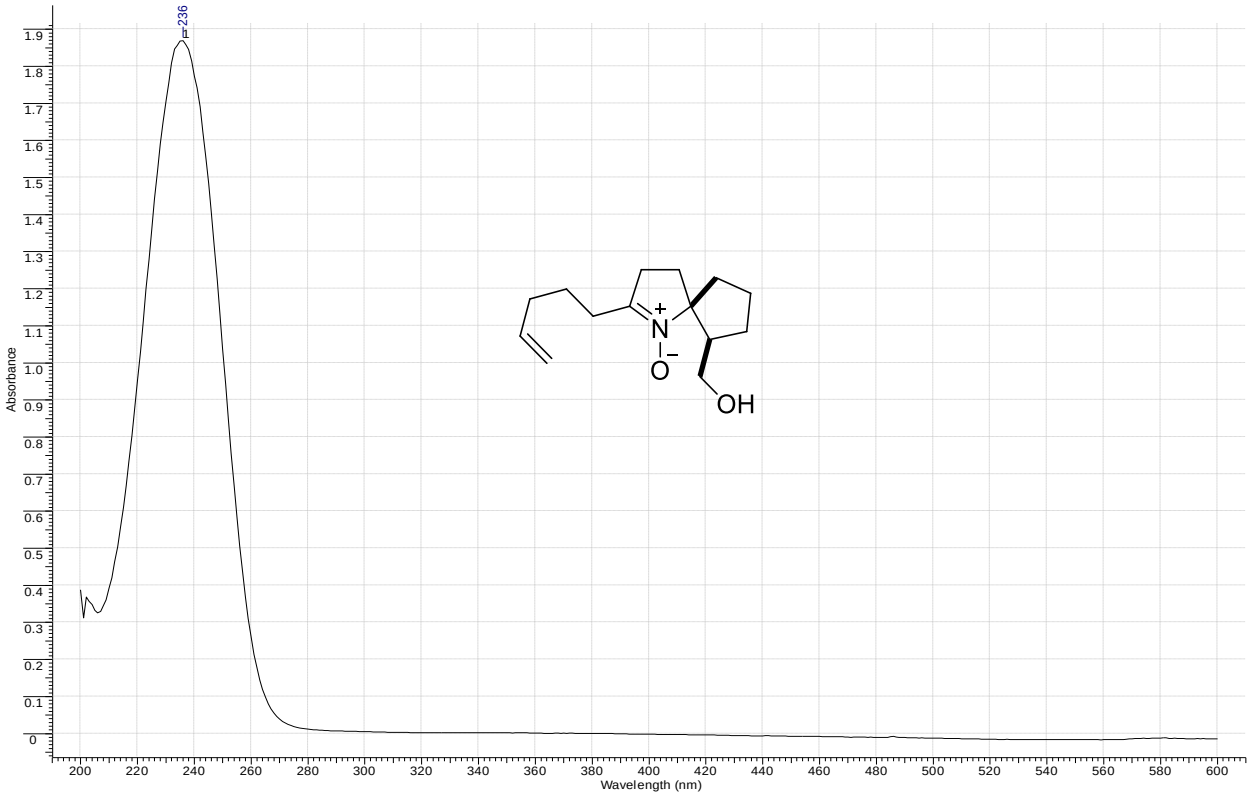


**Fig. S25.** UV spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-2-pent-4-en-1-yl-1-azaspiro[4.4]non-1-en-6-yl]methanol (**12**) in EtOH (0.596mg/25ml, L=2cm)

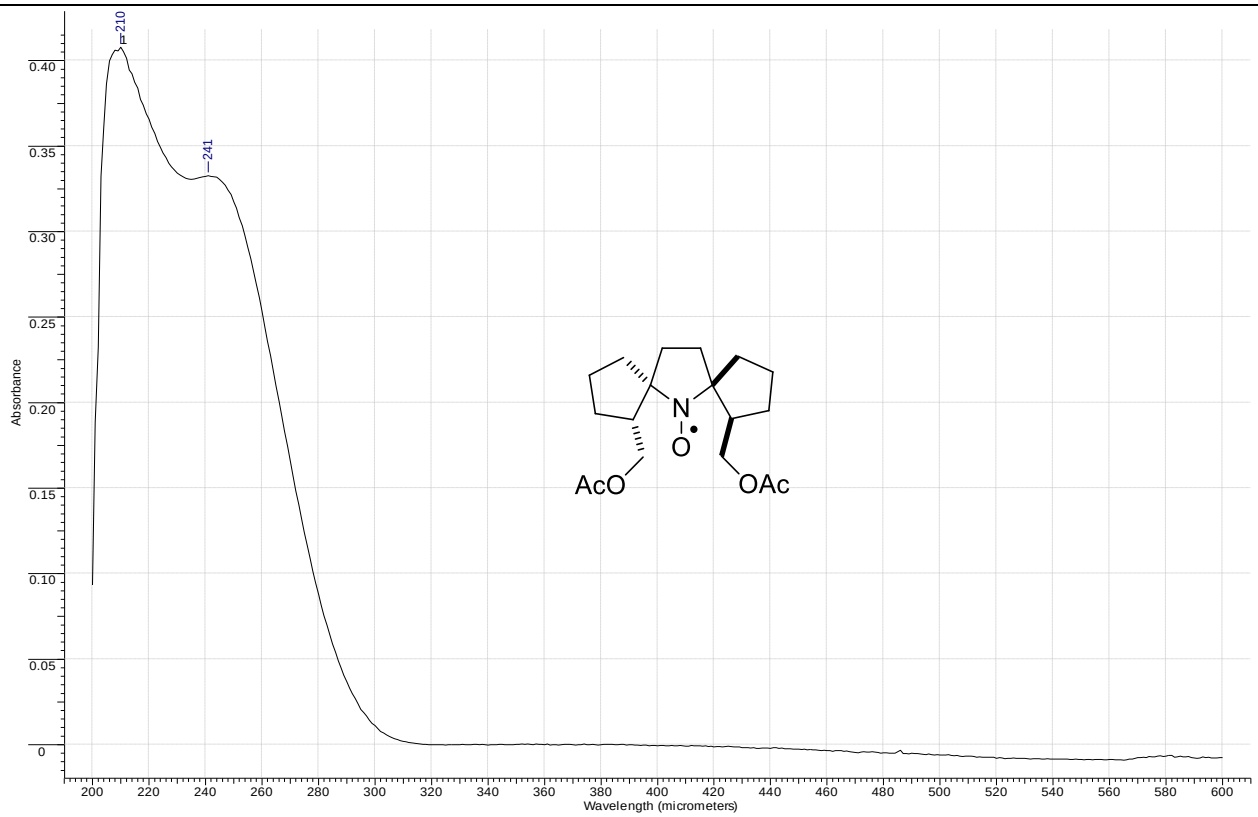


**Fig. S26.** UV spectrum of (1*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(acetyloxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**17**) in EtOH (0.848mg/25ml, L=2cm)

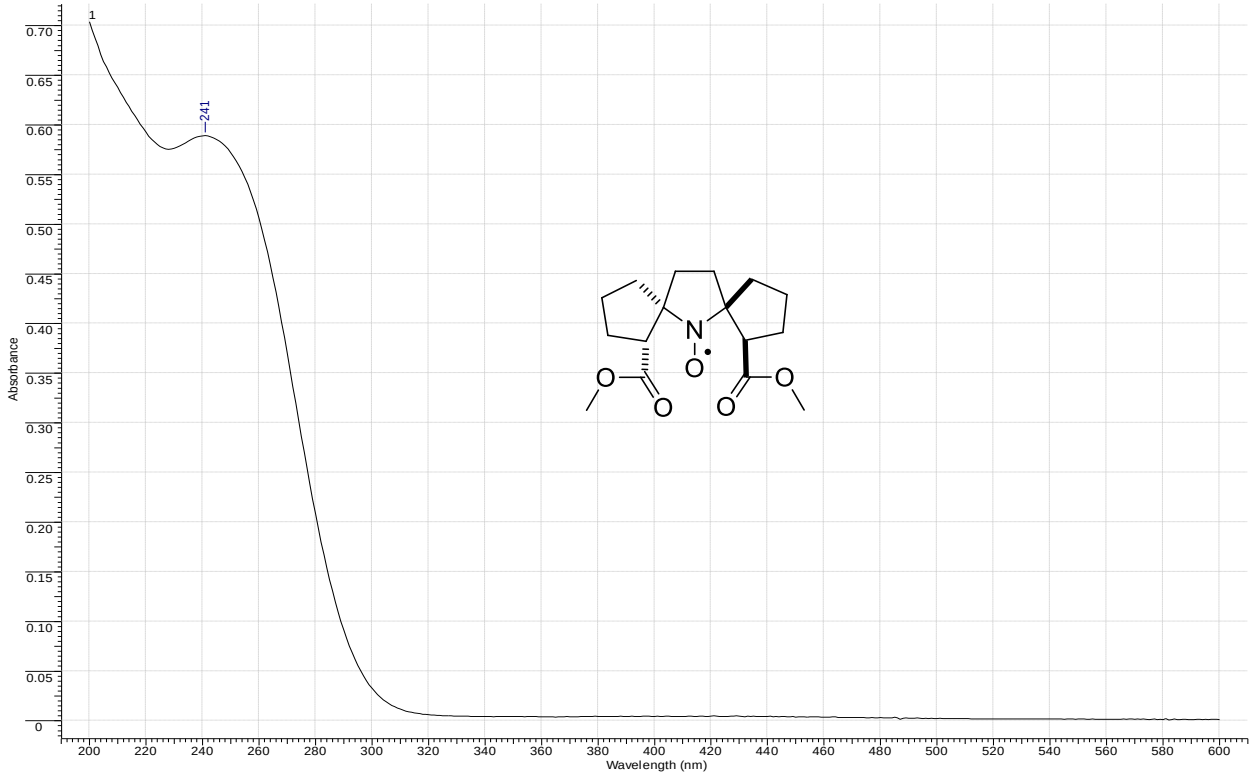


**Fig. S27.** UV spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[Methoxycarbonyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**22**) in EtOH (4.985mg/25ml, L=0.5cm)

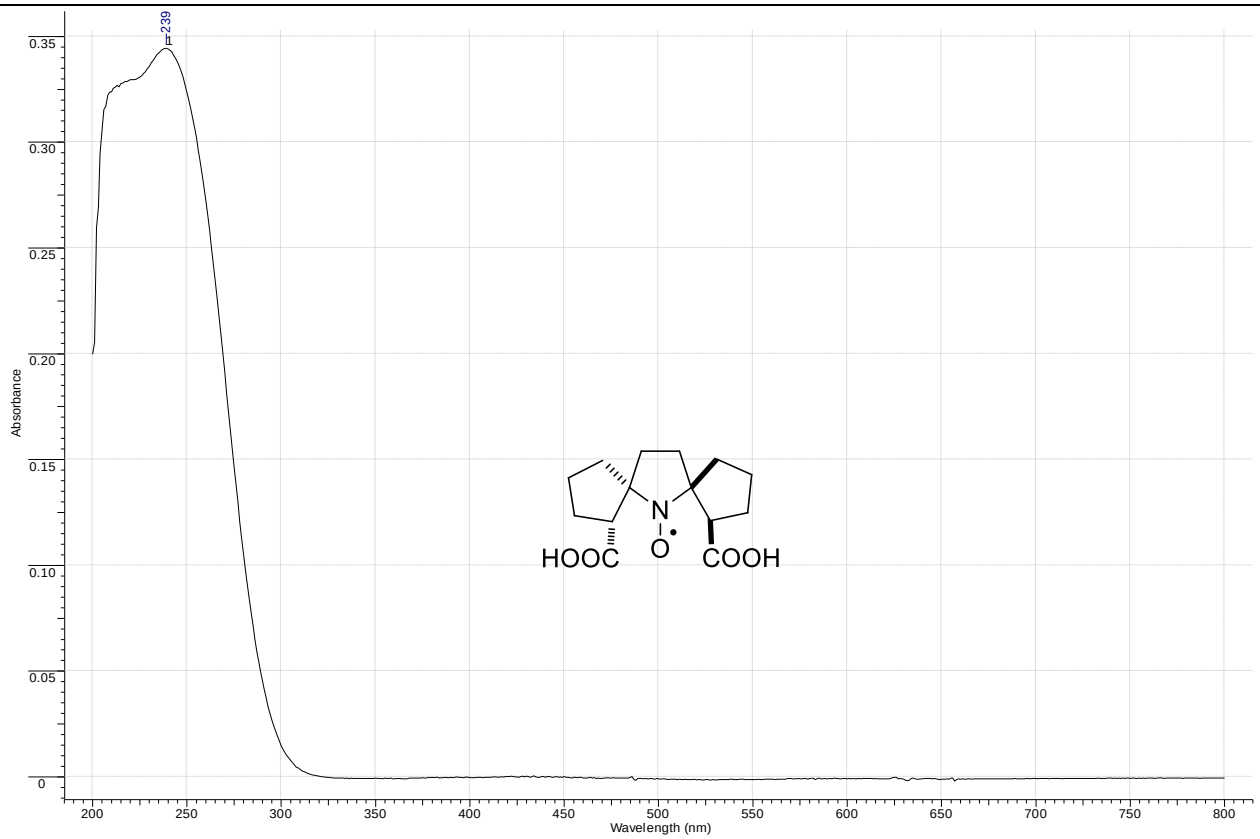


**Fig. S28.** UV spectrum of 1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Dicarboxy-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**23a**) in EtOH (0.708mg/25ml, L=2cm)

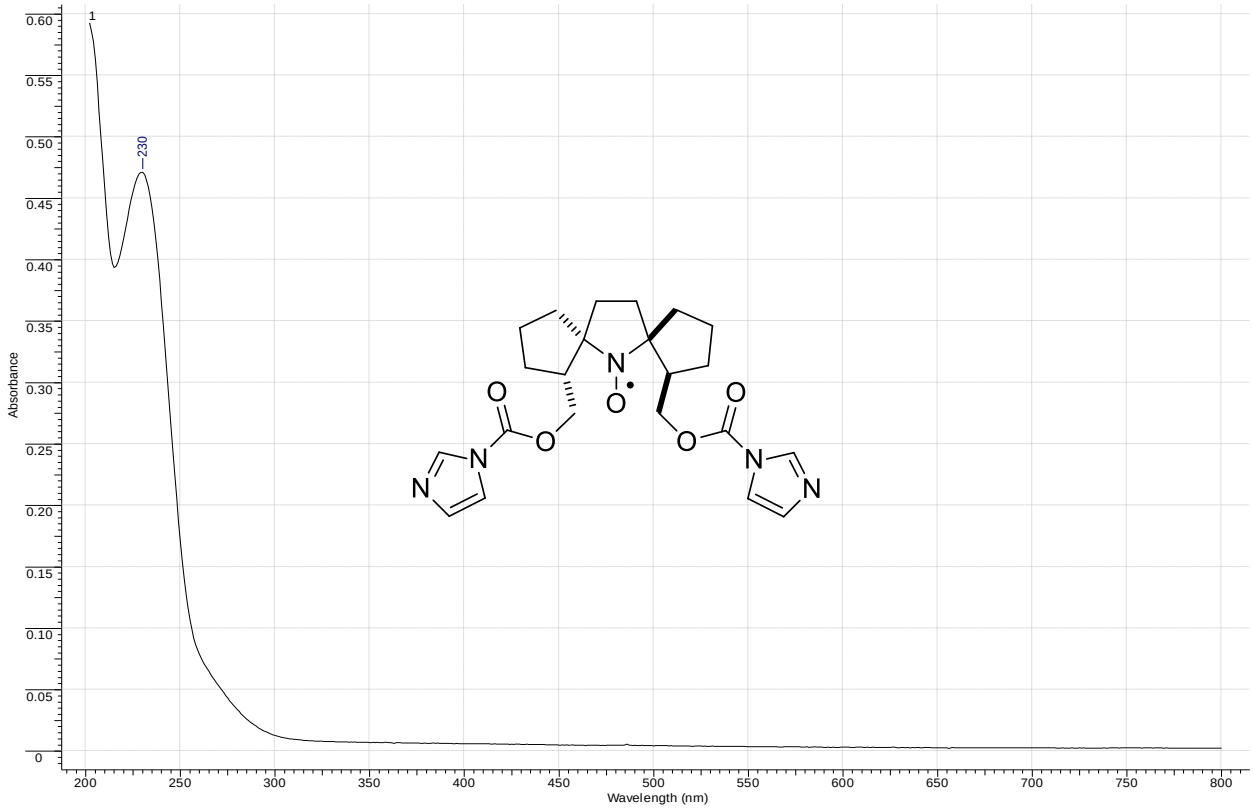


**Fig. S29.** UV spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[({1*H*-imidazol-1-ylcarbonyl}oxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**26**) in EtOH (1.150mg/25ml, L=0.5cm)

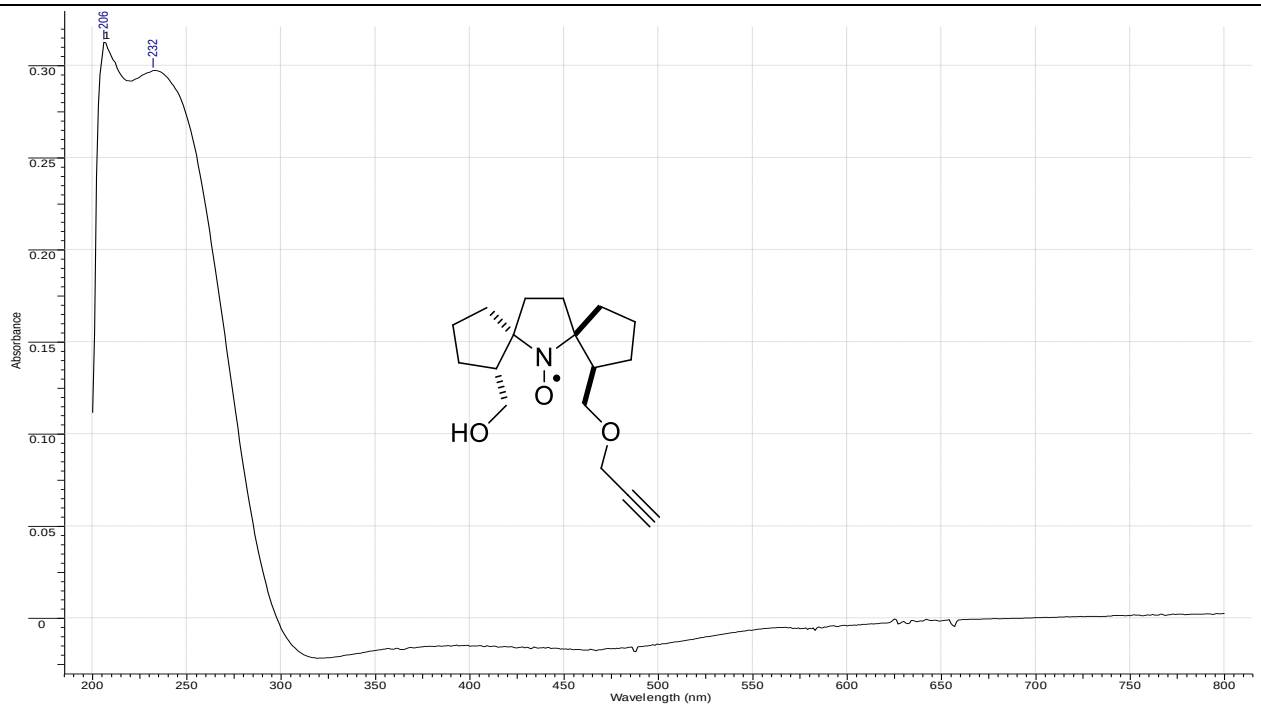


**Fig. S30.** UV spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-[(prop-2-yn-1-yloxy)methyl]-6-azadispiro[4.1.4.2]tridecane-6-oxyl (**31**) in EtOH (0.740mg/25ml, L=2cm)

## 4. $^{1}$H and $^{13}$C NMR spectral data

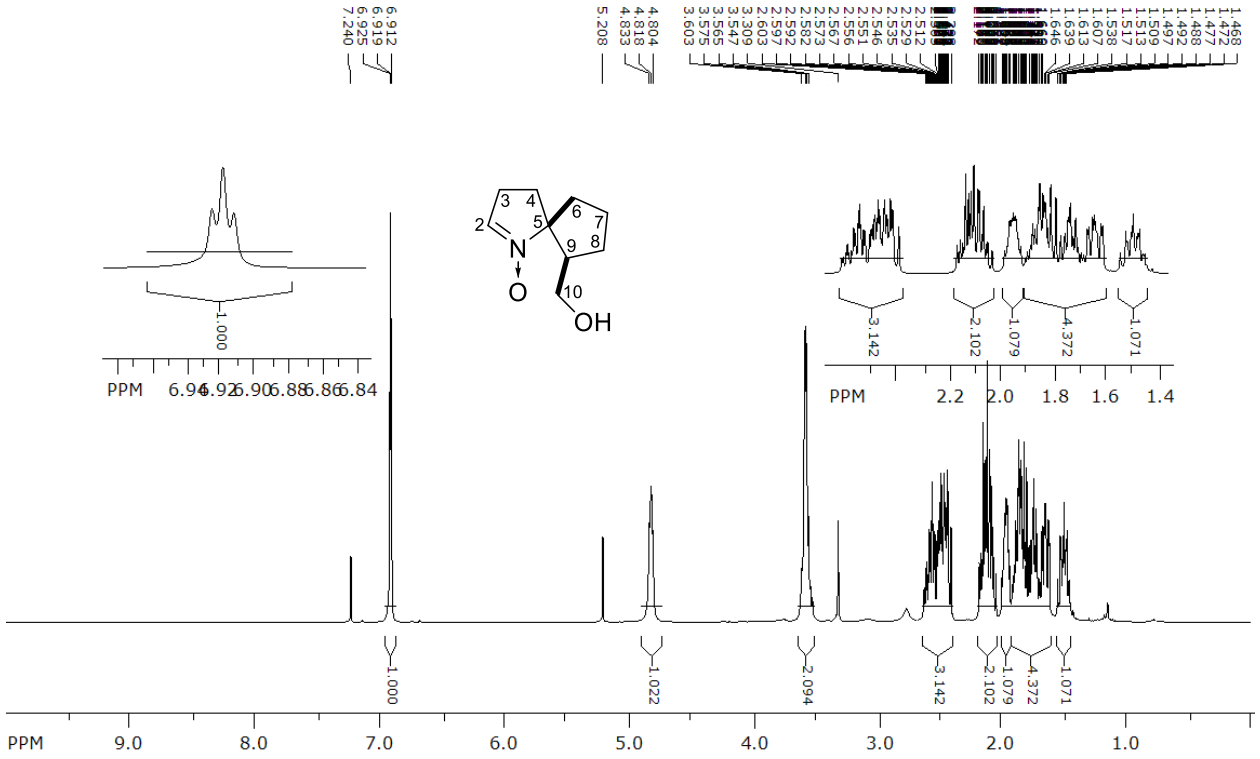


**Fig. S31.** $^{1}$H NMR spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-1-azaspiro[4.4]non-1-en-6-yl]methanol (**9**) in $CDCl_3$ at 400 MHz

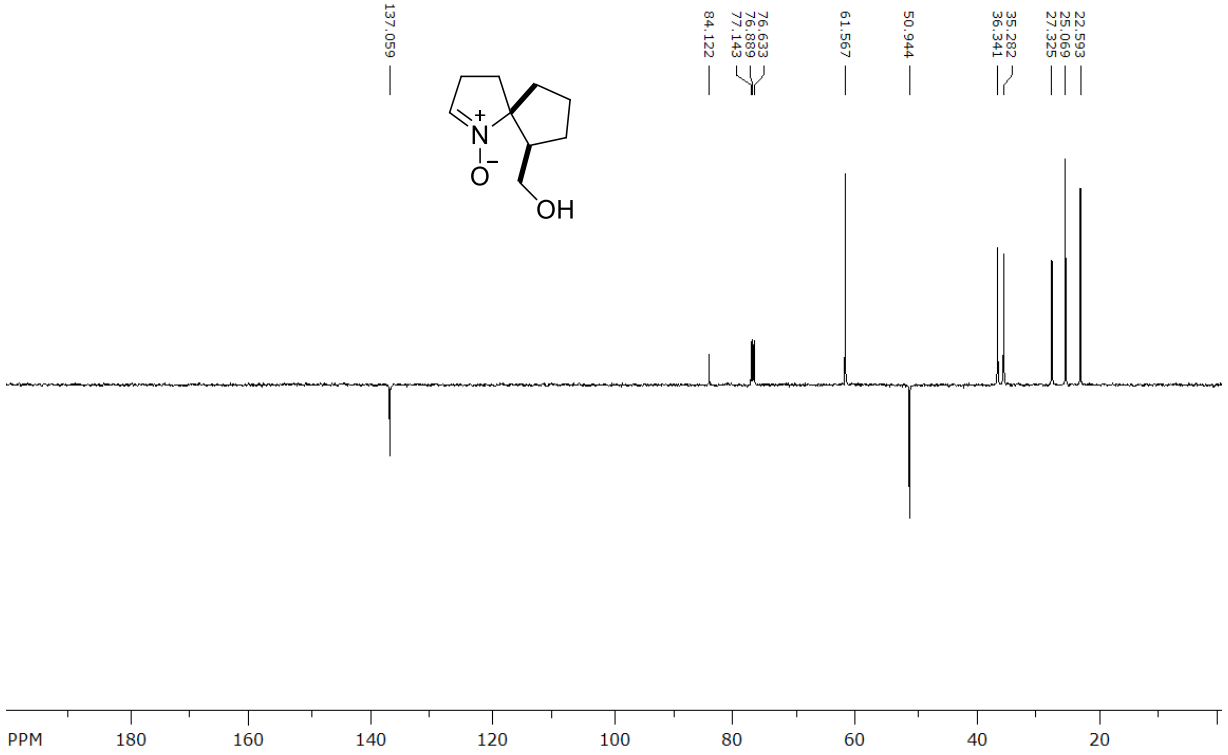


**Fig. S32.** $^{13}$C NMR spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-1-azaspiro[4.4]non-1-en-6-yl]methanol (**9**) in $CDCl_3$ at 125 MHz

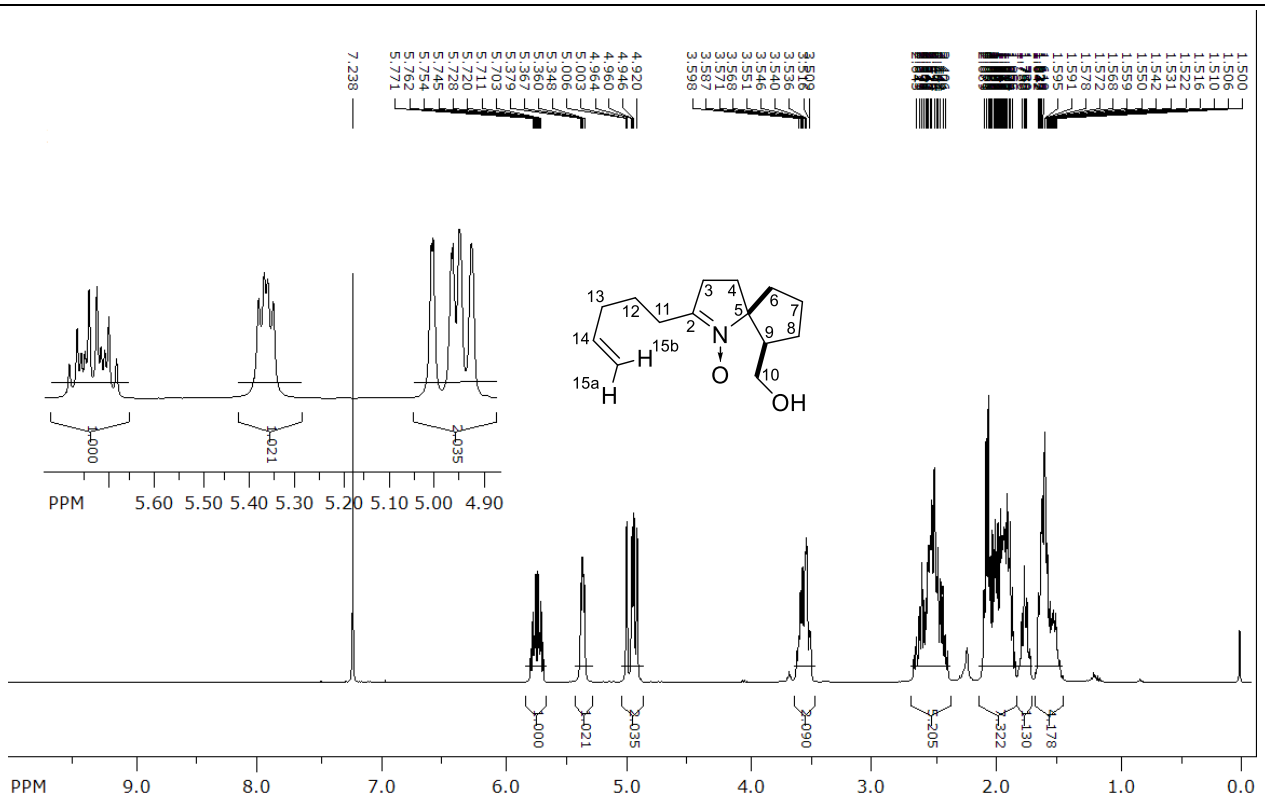


**Fig. S33.** [1]H NMR spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-2-(pent-4-en-1-yl)-1-azaspiro[4.4]non-1-en-6-yl]methanol (**12**) in $CDCl_3$ at 400 MHz

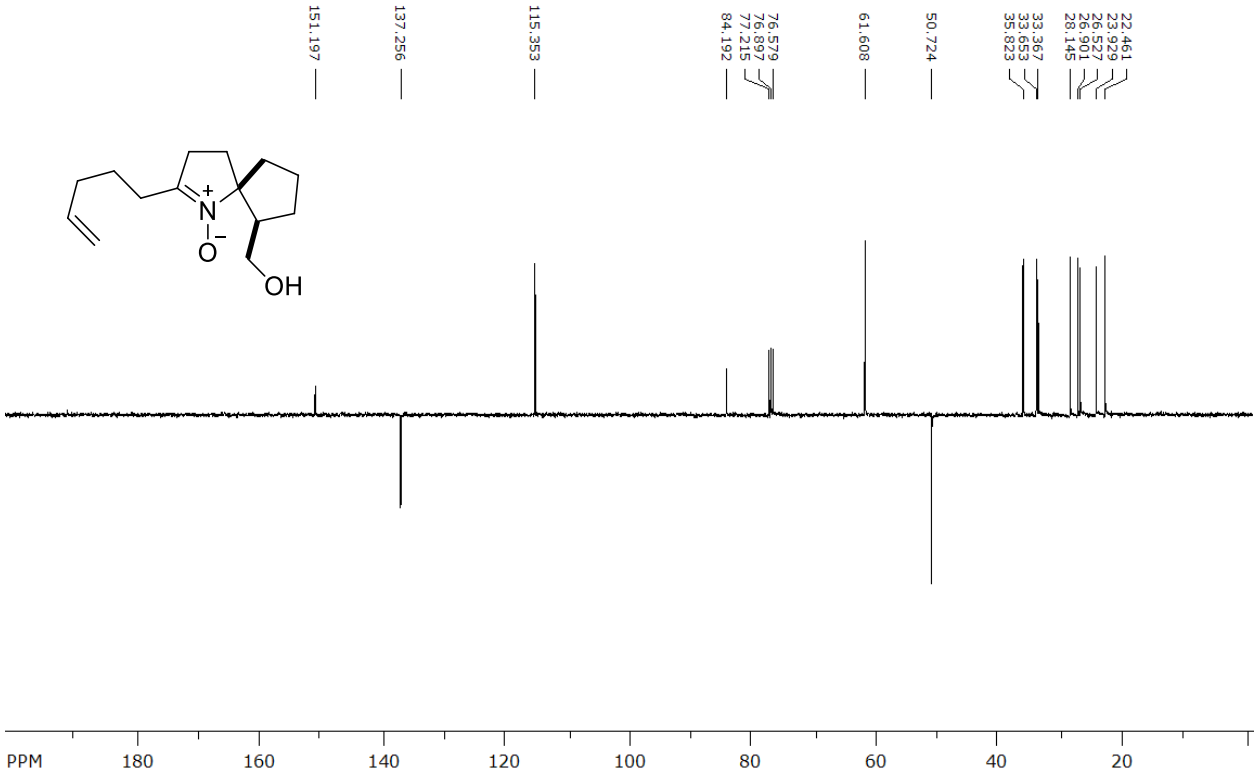


**Fig. S34.** [13]C NMR spectrum of [(5*R*(*S*),6*R*(*S*))-1-Oxido-2-(pent-4-en-1-yl)-1-azaspiro[4.4]non-1-en-6-yl]methanol (**12**) in $CDCl_3$ at 100 MHz

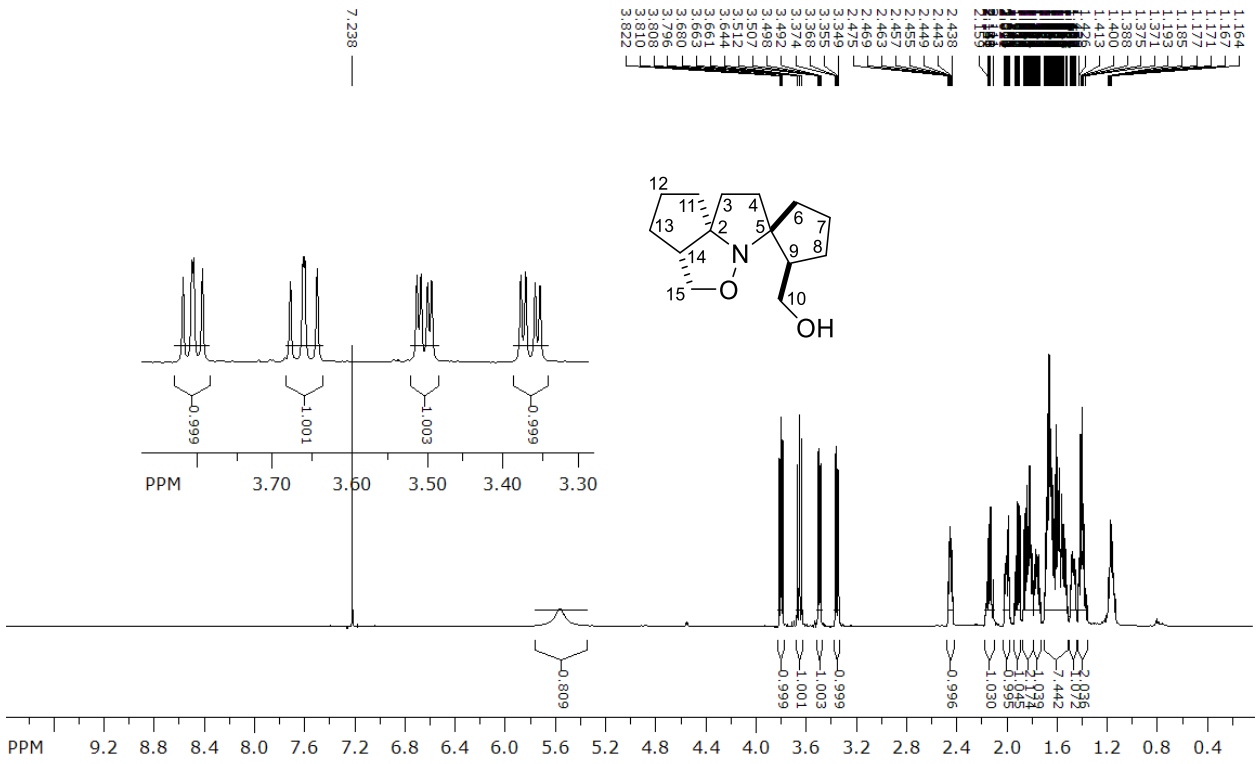


**Fig. S35.** [1]H NMR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*)-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) in $CDCl_3$ at 600 MHz

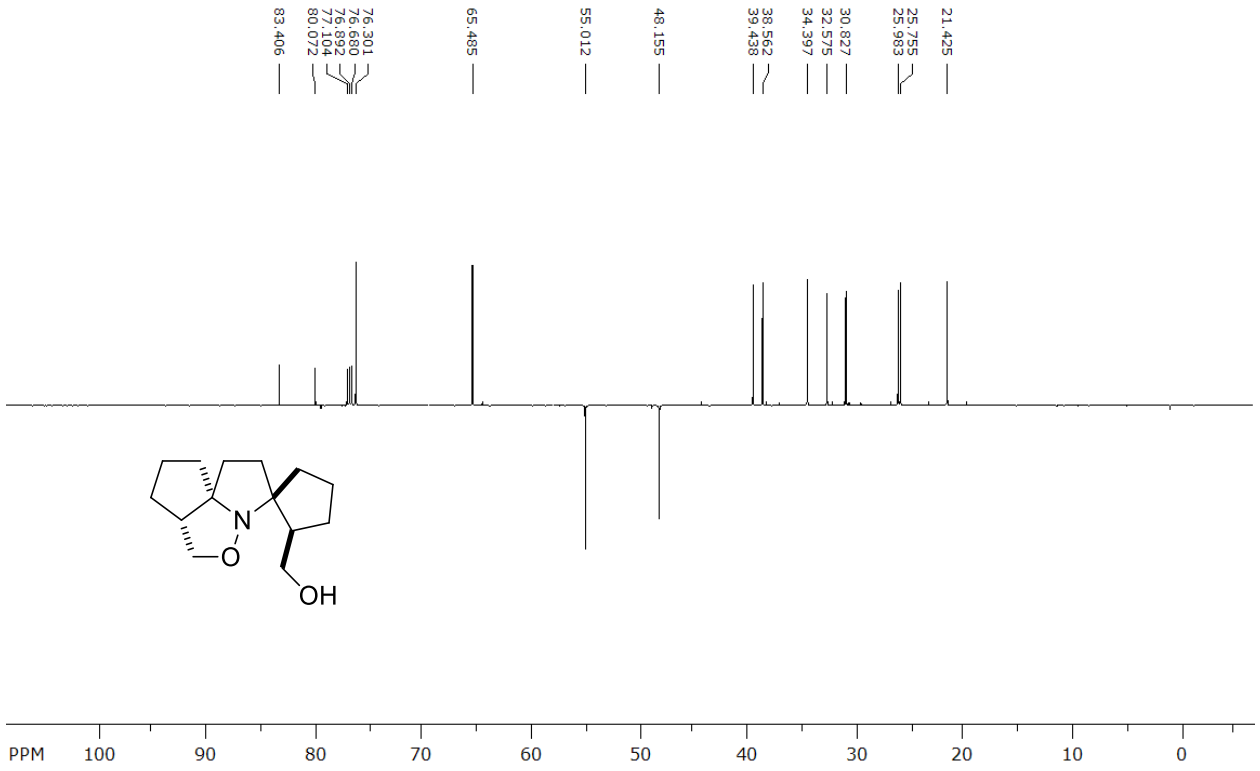


**Fig. S36.** [13]C NMR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*)-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) in $CDCl_3$ at 150 MHz

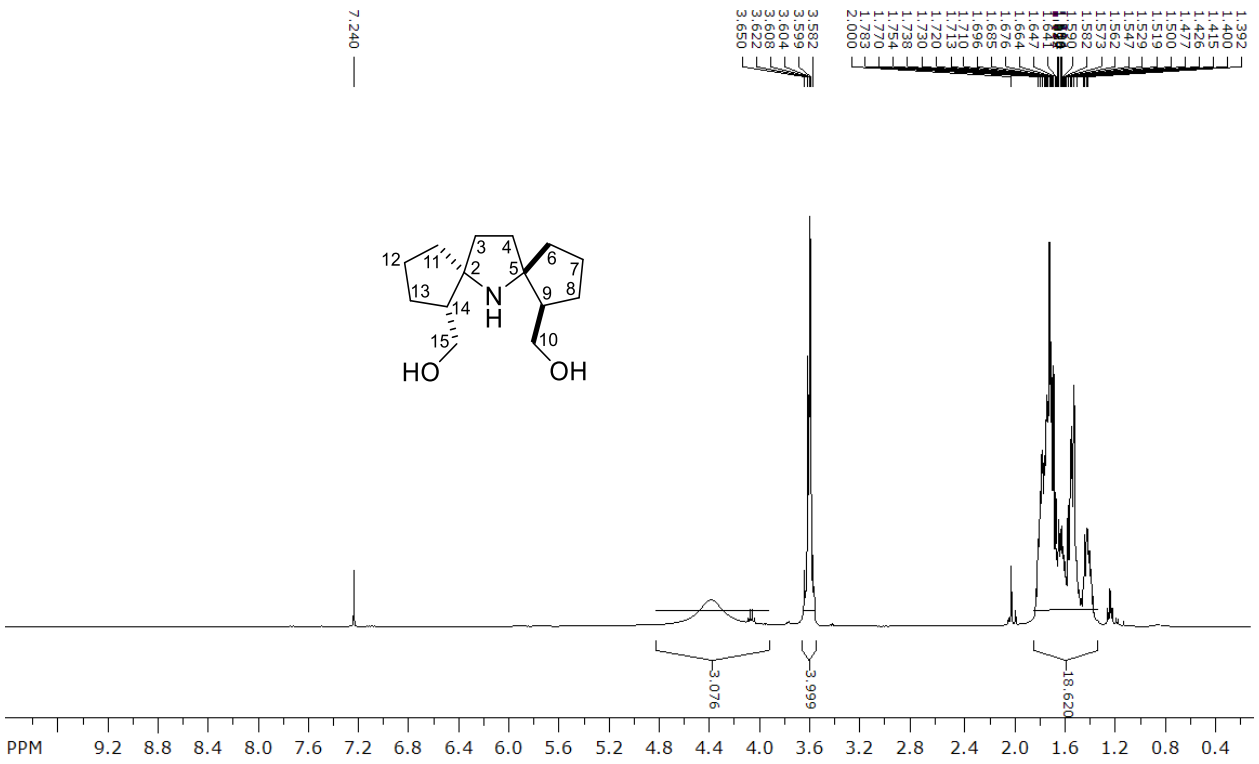


**Fig. S37.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-diyldimethanol (**14**) in $CDCl_3$ at 400 MHz

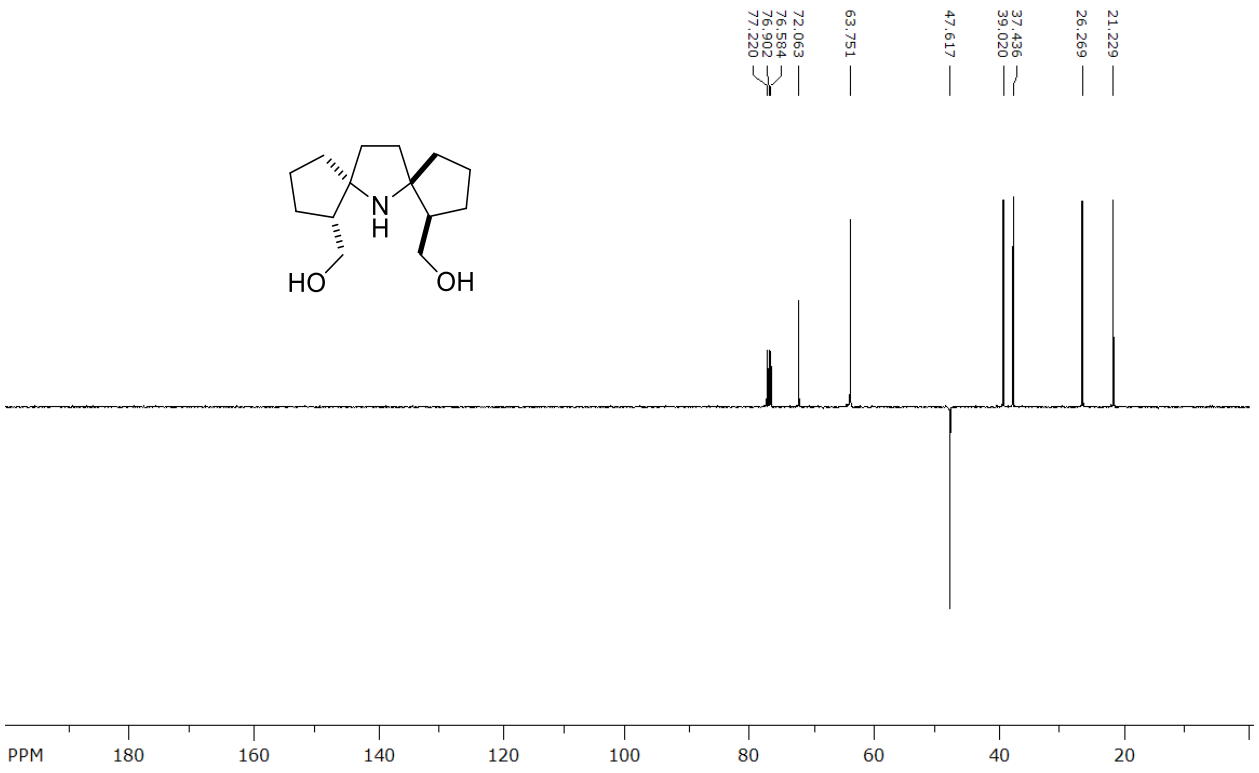


**Fig. S38.** $^{13}$C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-diyldimethanol (**14**) in $CDCl_3$ at 100 MHz

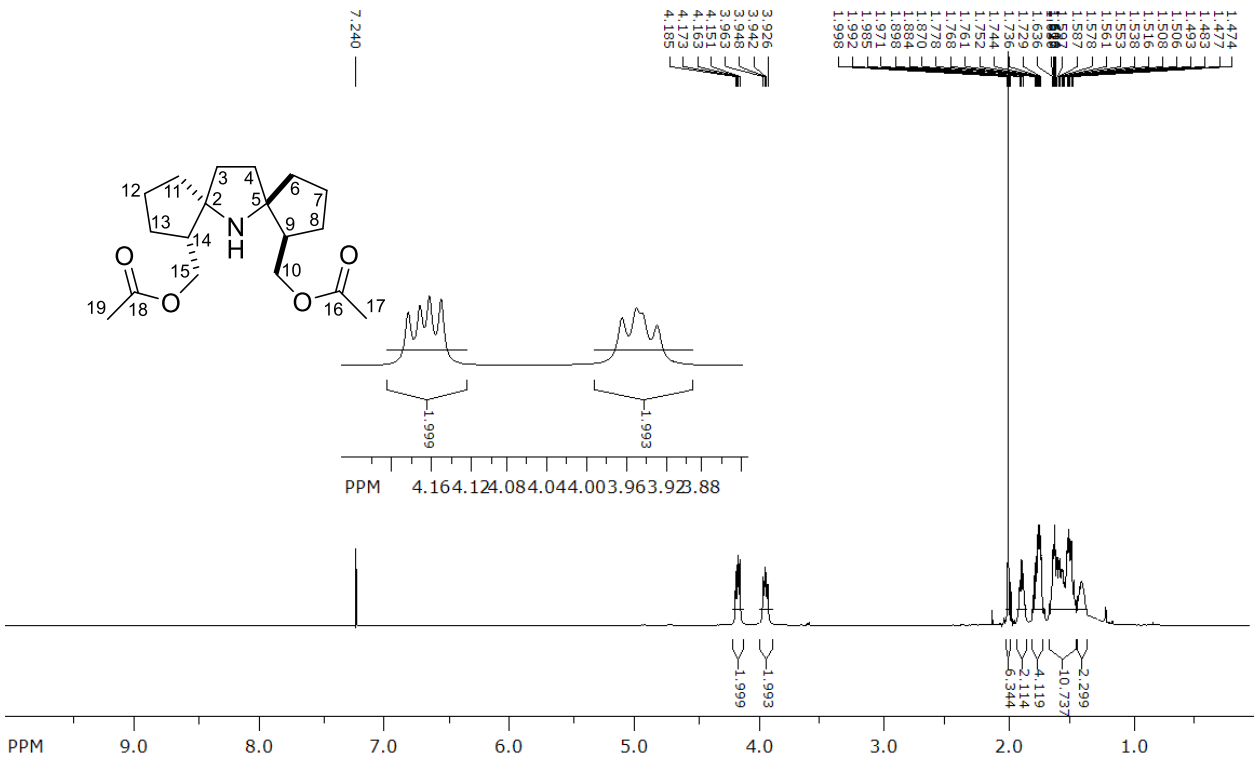


**Fig. S39.** [1]H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-diyldi(methylene) diacetate (**16**) in $CDCl_3$ at 500 MHz

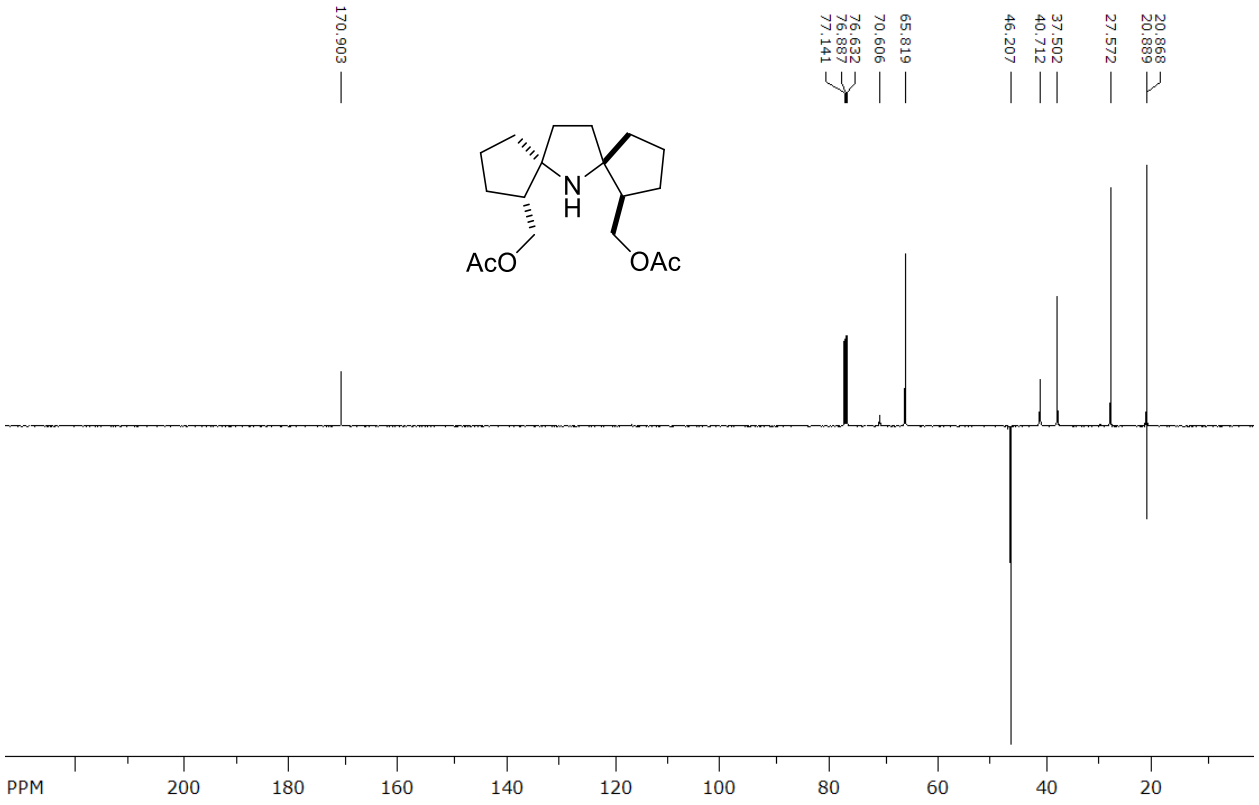


**Fig. S40.** [13]C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-diyldi(methylene) diacetate (**16**) in $CDCl_3$ at 125 MHz

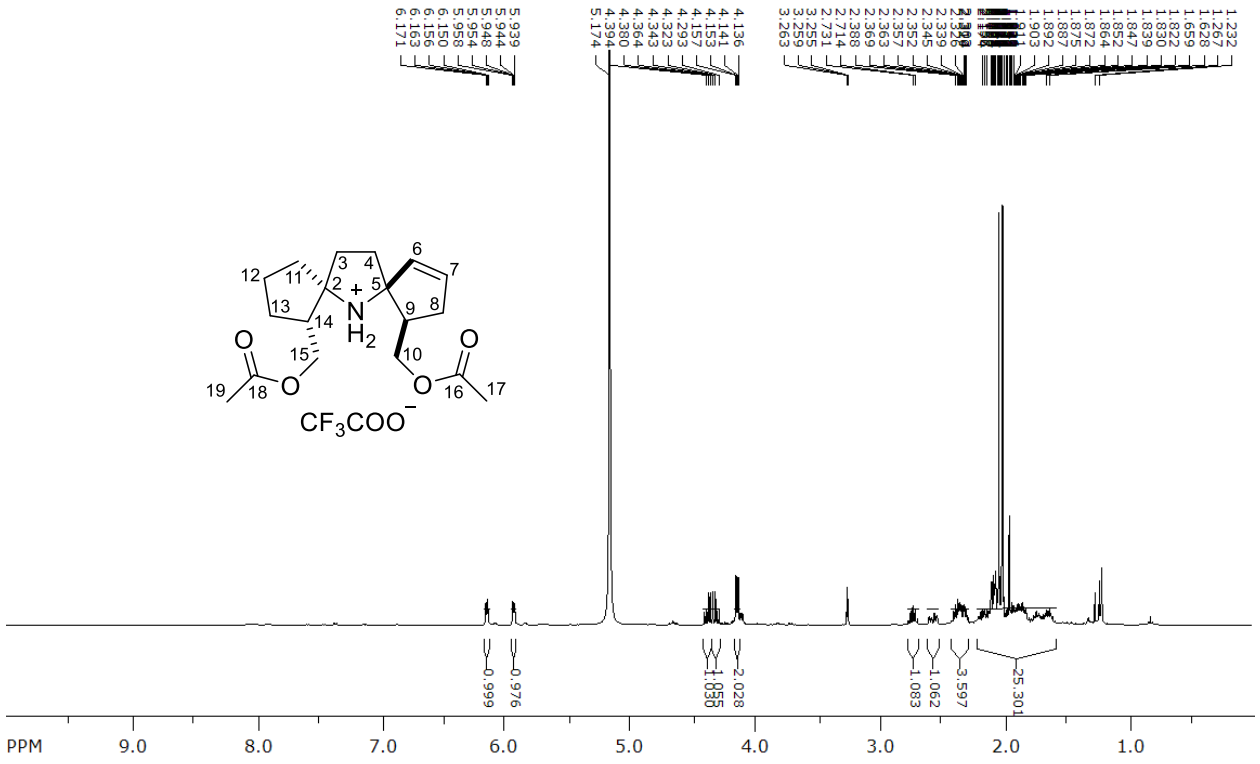


**Fig. S41.** [1]H NMR spectrum of (4*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-4,8-Bis[(acetyloxy)methyl]-6-azoniadispiro[4.1.4.2]tridec-1-ene trifluoroacetate (**19**) in $CD_3OD$+$CF_3COOH$ at 400 MHz

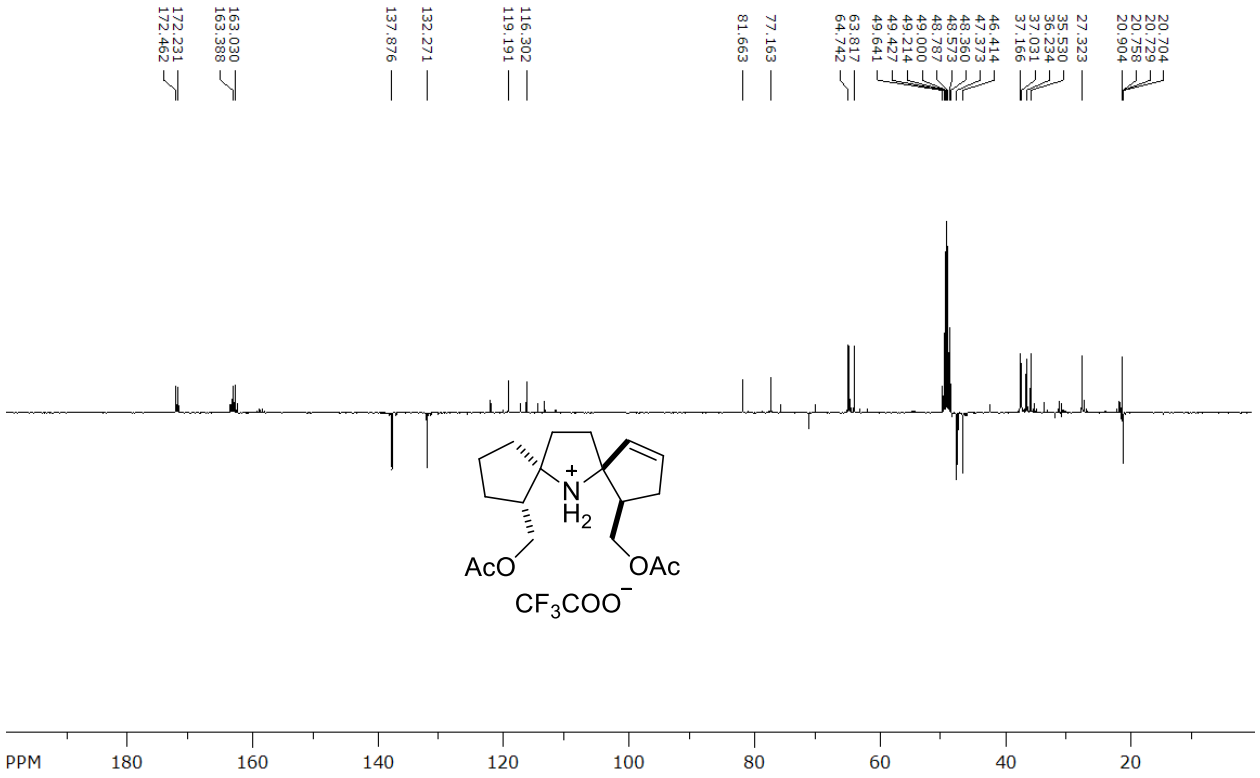


**Fig. S42.** [13]C NMR spectrum of (4*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-4,8-Bis[(acetyloxy)methyl]-6-azoniadispiro[4.1.4.2]tridec-1-ene trifluoroacetate (**19**) in $CD_3OD$+$CF_3COOH$ at 100 MHz

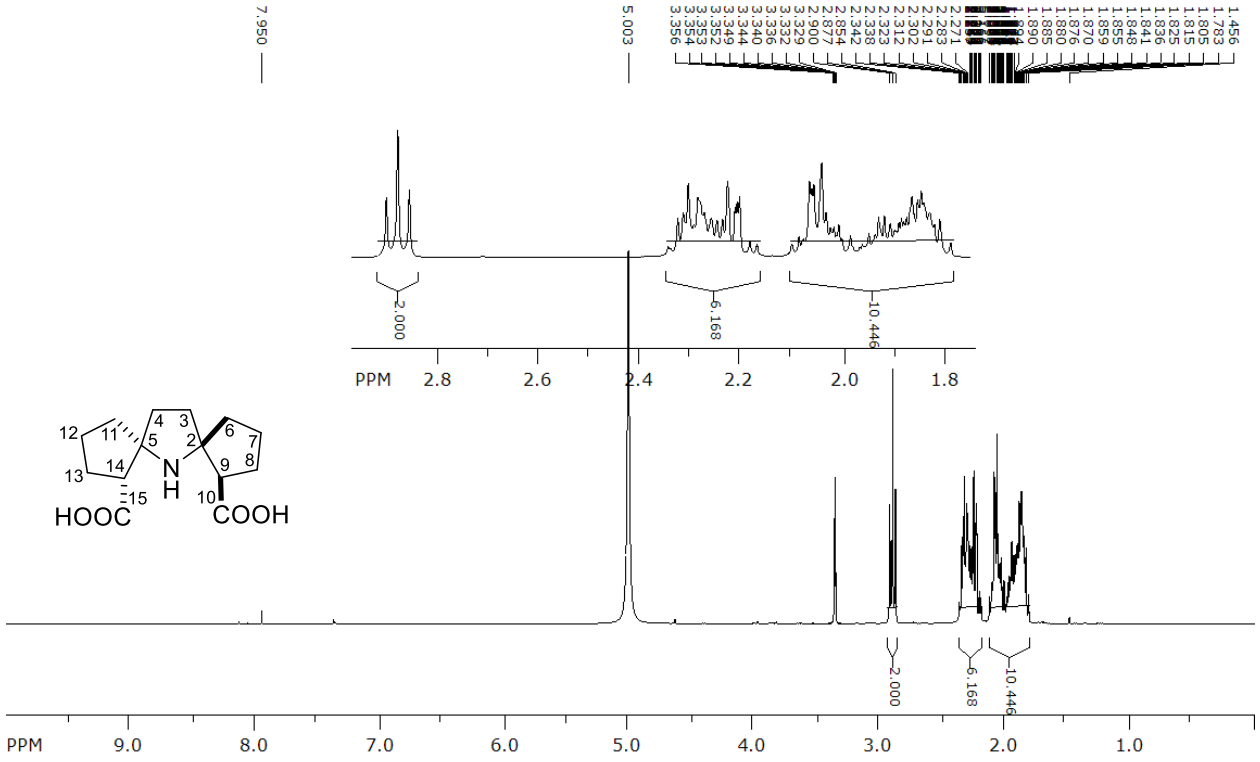


**Fig. S43.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-Azadisporo[4.1.4.2]tridecan-1,8-dicarboxylic acid (**20**) in $CD_3OD$ at 400 MHz

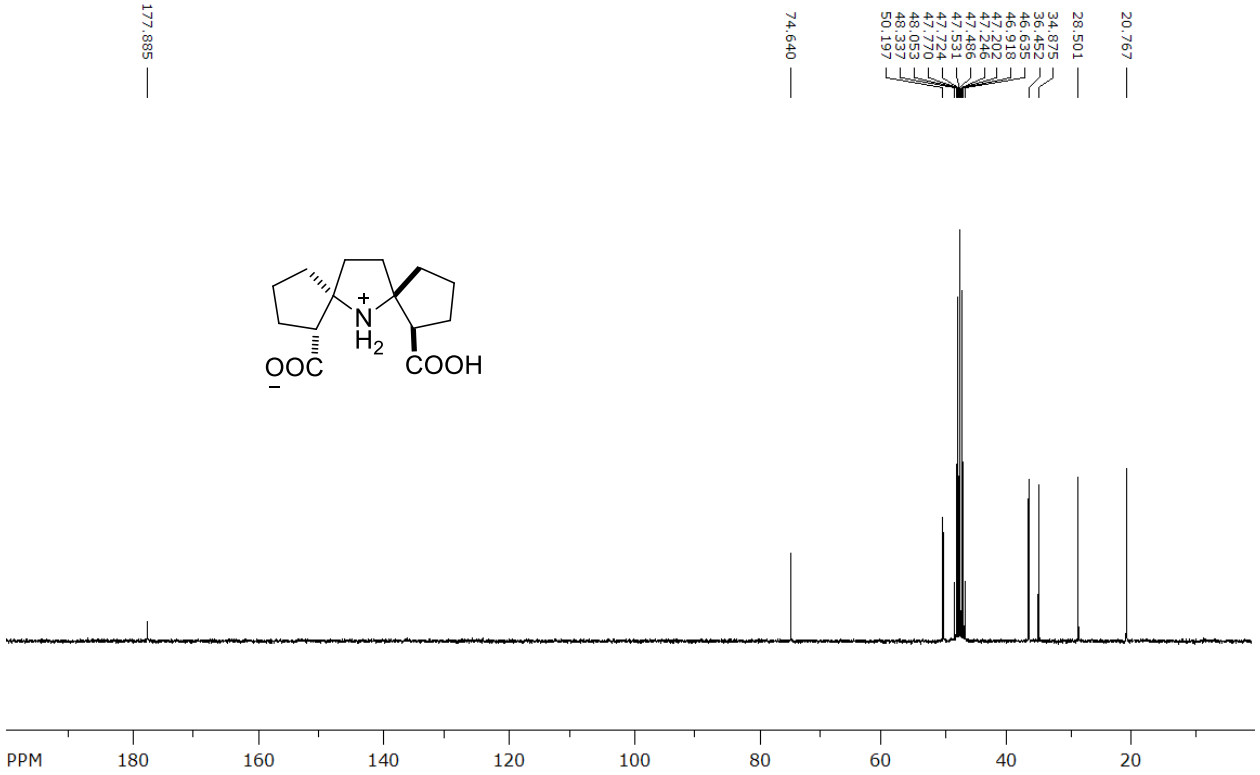


**Fig. S44.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-Azadisporo[4.1.4.2]tridecan-1,8-dicarboxylic acid (**20**) in $CD_3OD$ at 75 MHz

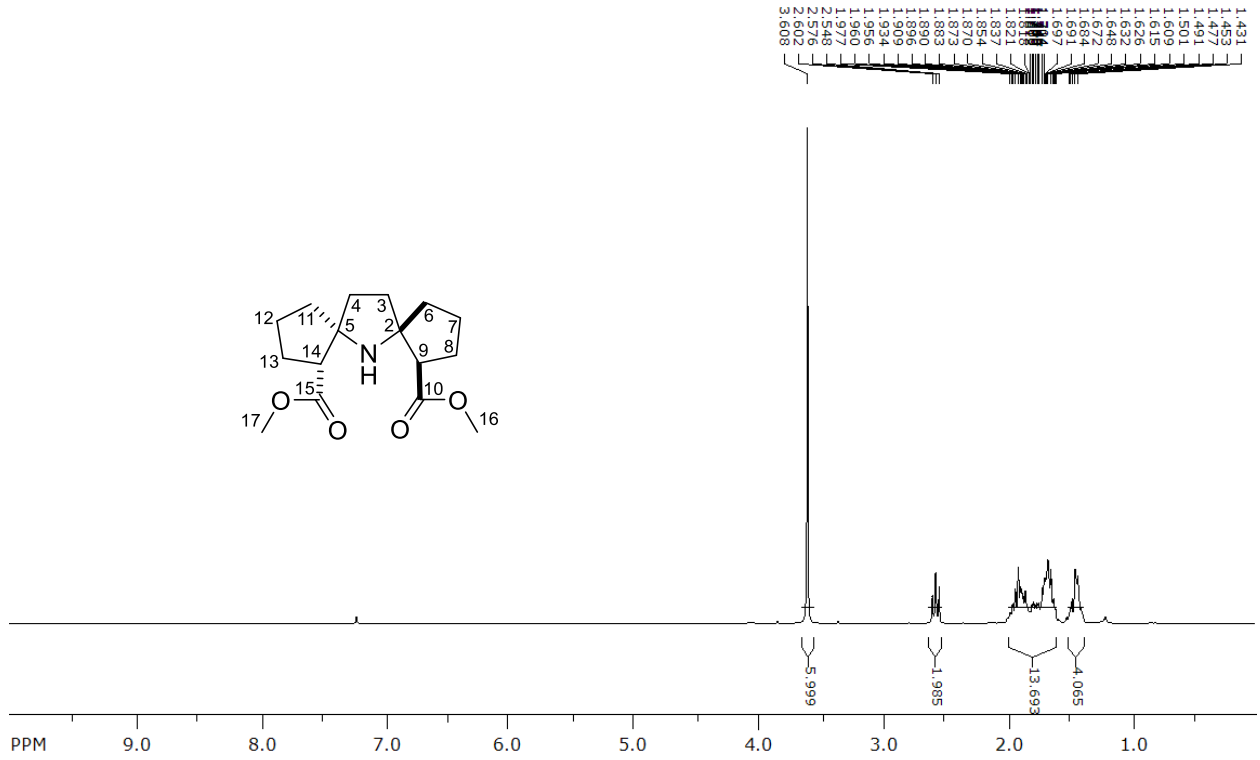


**Fig. S45.** [1]H NMR spectrum of Dimethyl (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-dicarboxylate (**21**) in $CDCl_3$ at 300 MHz

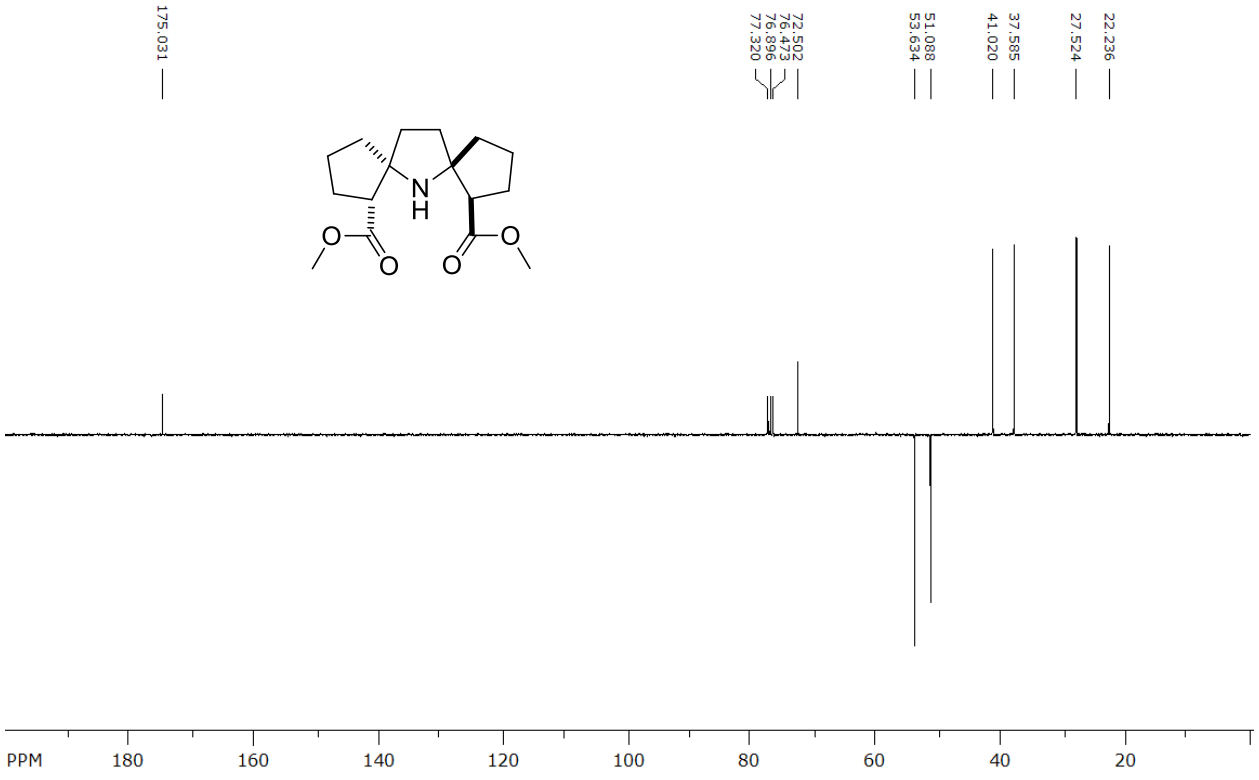


**Fig. S46.** [1]H NMR spectrum of Dimethyl (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-6-azadispiro[4.1.4.2]tridecan-1,8-dicarboxylate (**21**) in $CDCl_3$ at 75 MHz

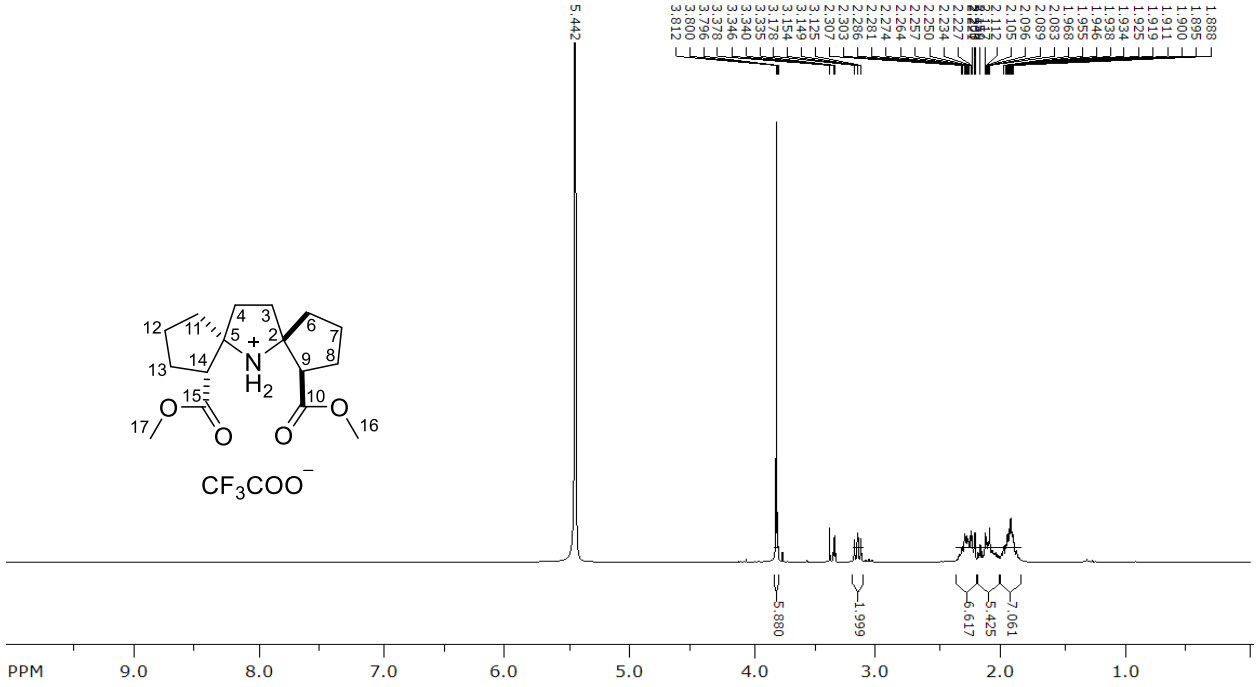


**Fig. S47.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis(methoxycarbonyl)-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**22-red**) in $CD_3OD$+$CF_3COOH$ at 300 MHz

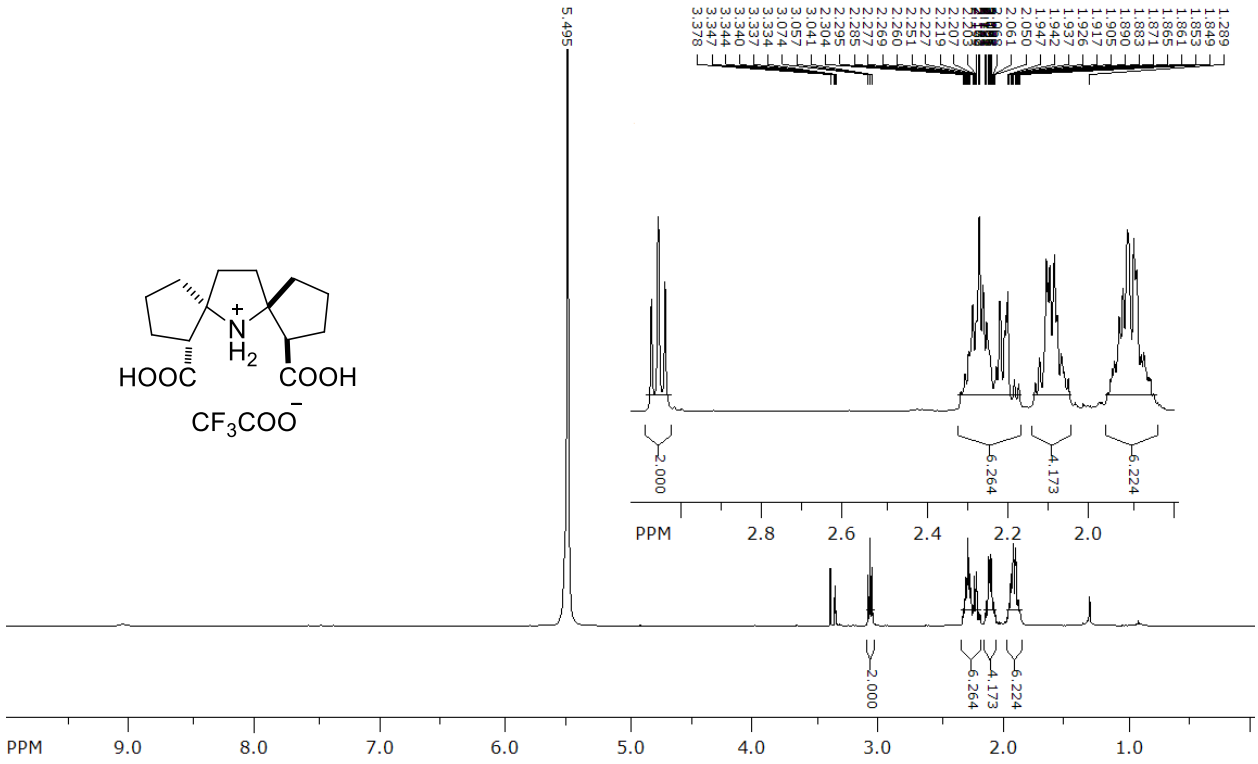


**Fig. S48.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Dicarboxy-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**23a-red**) in $CD_3OD$+$CF_3COOH$ at 500 MHz

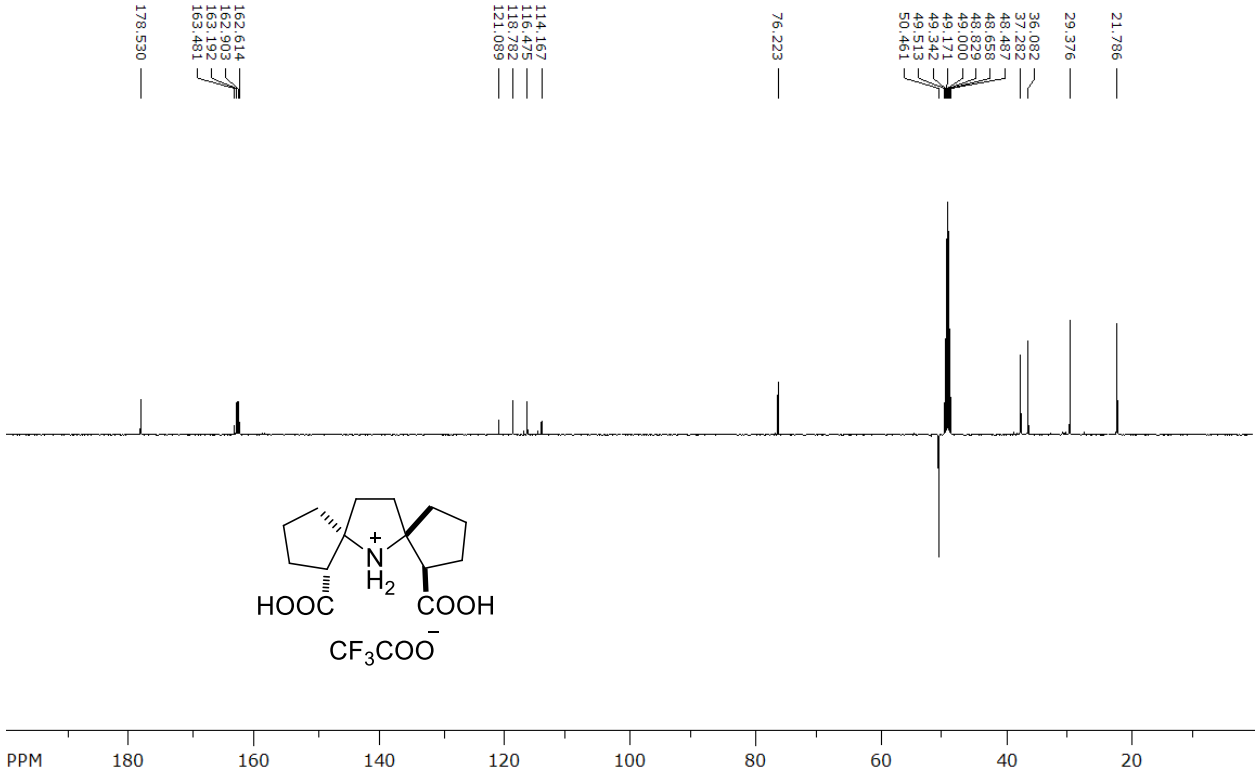


**Fig. S49.** $^{13}$C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Dicarboxy-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**23a-red**) in $CD_3OD$+$CF_3COOH$ at 125 MHz

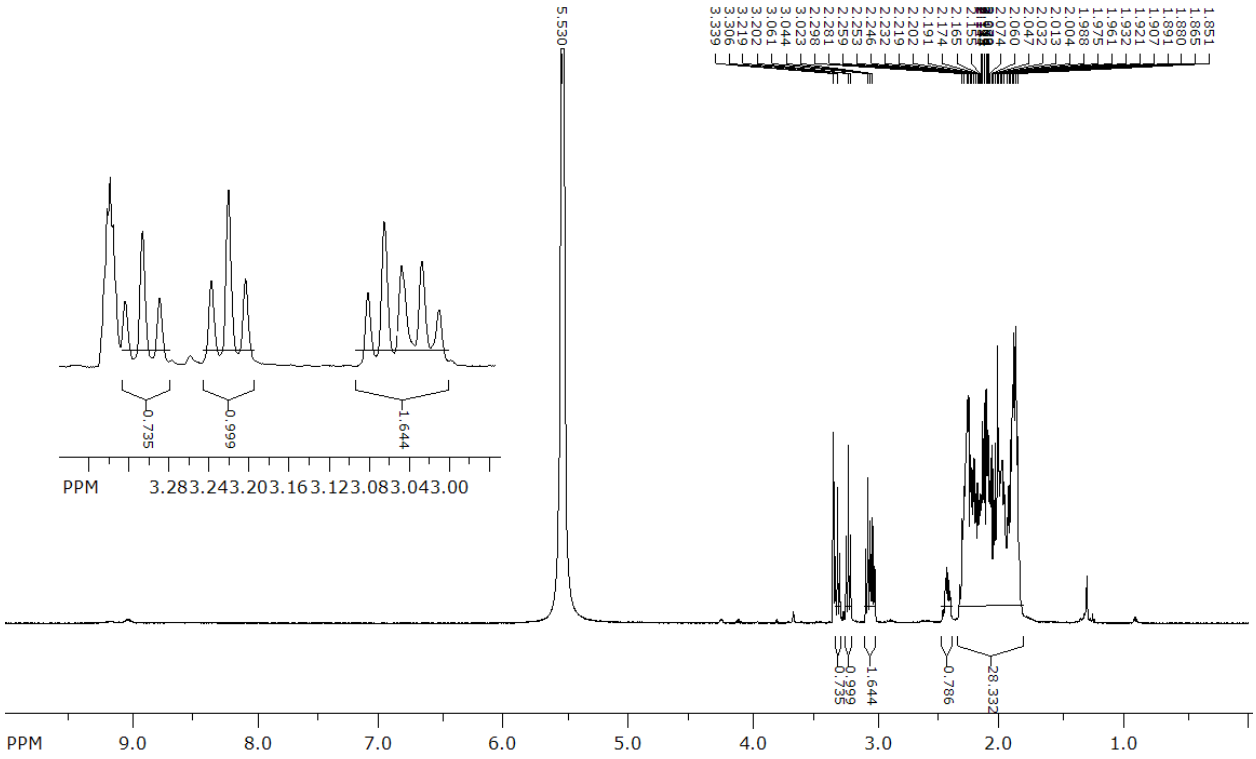


**Fig. S50.** $^{1}$H NMR spectrum of isomeric mixture of 1,8-Dicarboxy-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetates (**23a+23b+23c-red**) in $CD_3OD$+$CF_3COOH$ at 500 MHz

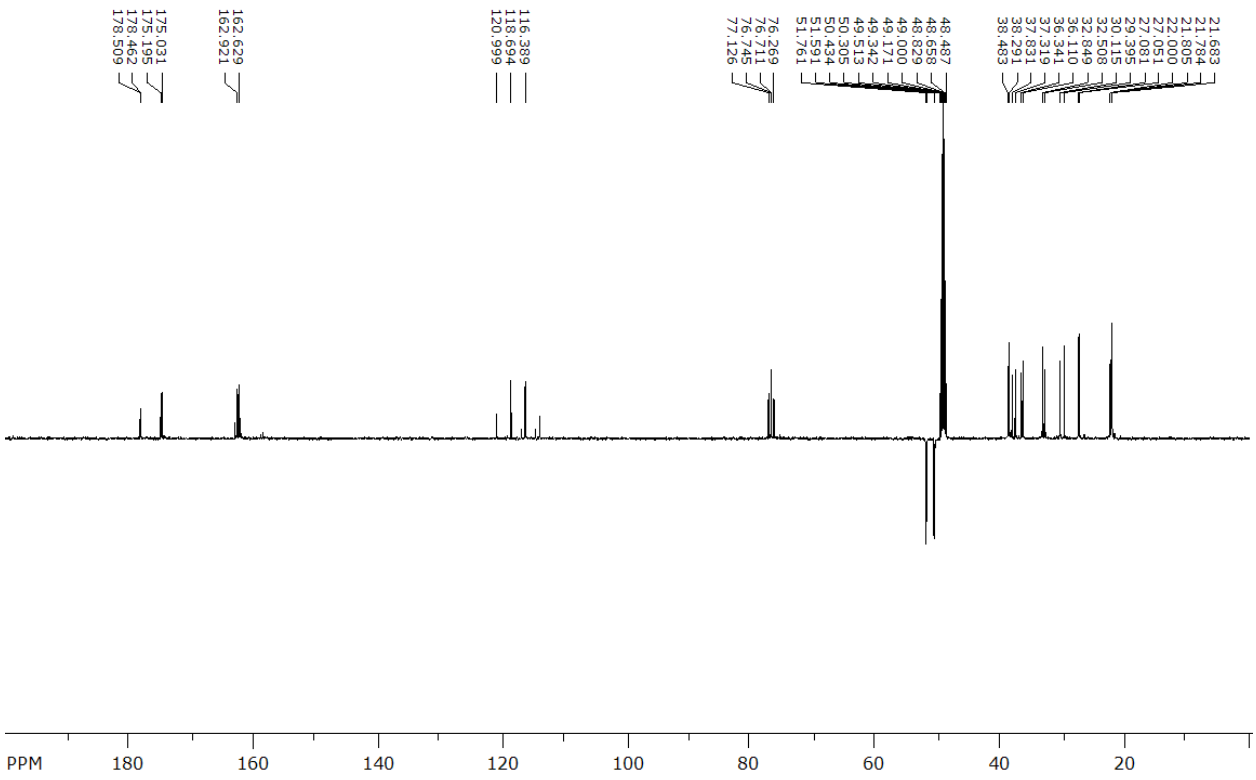


**Fig. S51.** $^{13}C$ NMR spectrum of isomeric mixture of 1,8-Dicarboxy-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetates (**23a+23b+23c-red**) in $CD_3OD+CF_3COOH$ at 125 MHz

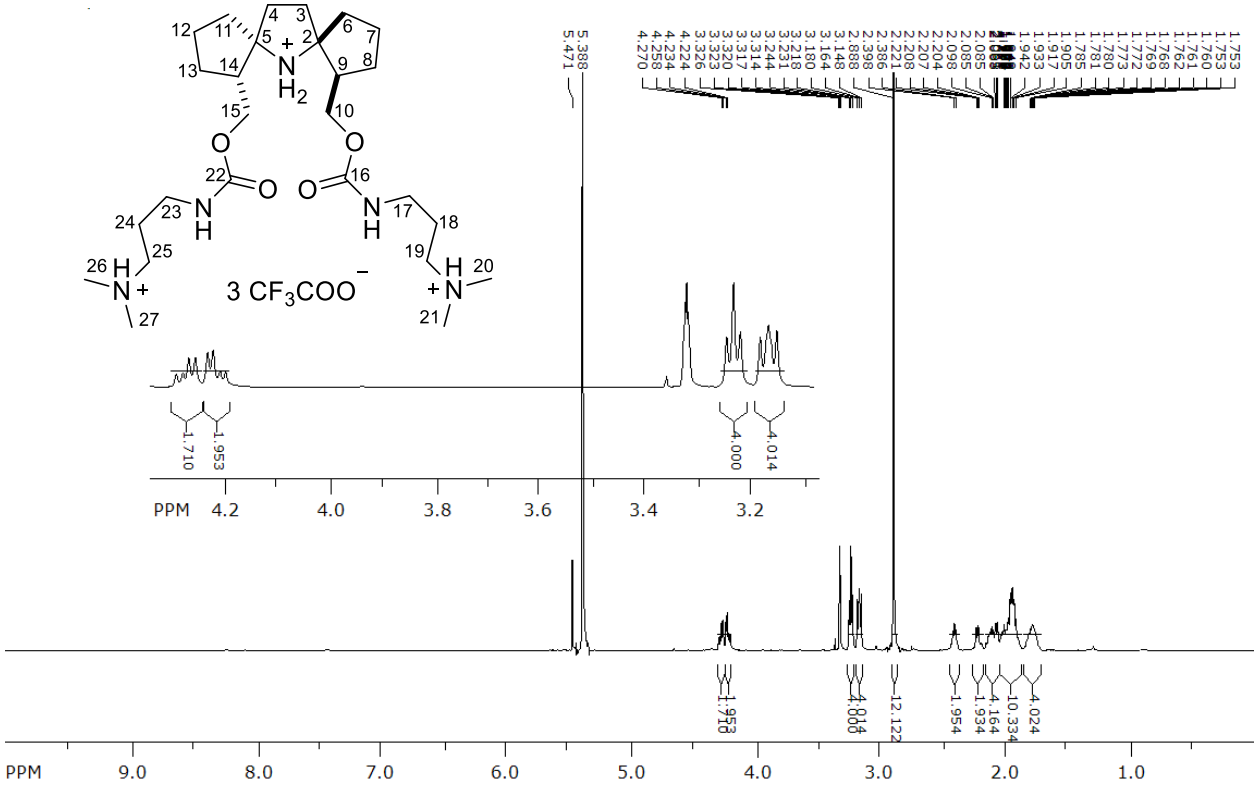


**Fig. S52.** $^{1}H$ NMR spectrum of (1R(S),5R(S),7R(S),8R(S))-1,8-Bis[(([{3-(dimethylamino)propyl}amino]carbonyl)oxy)methyl]-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**27-red**) in $CD_3OD+CF_3COOD$ at 500 MHz

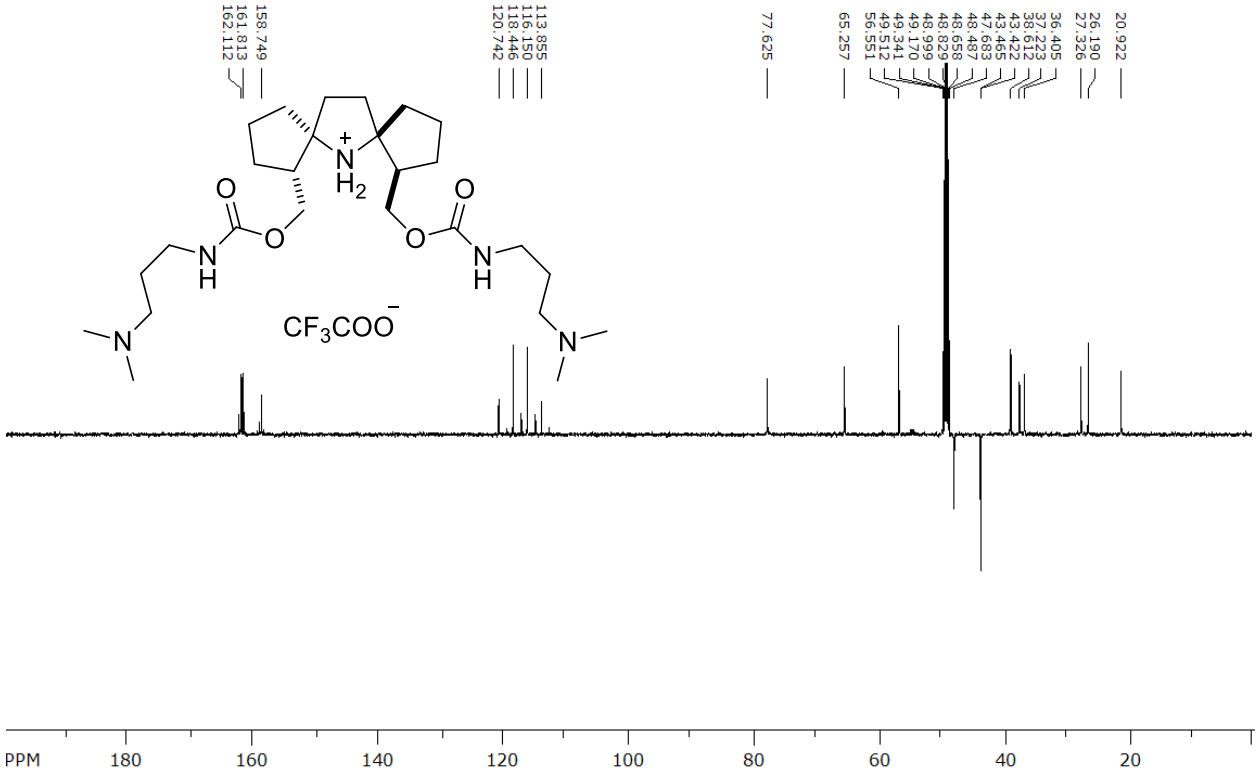


**Fig. S53.** $^{13}$C NMR spectrum of (1R(S),5R(S),7R(S),8R(S))-1,8-Bis[(([{3-(dimethylamino)propyl}amino]carbonyl)oxy)methyl]-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**27-red**) in $CD_3OD$+$CF_3COOD$ at 125 MHz

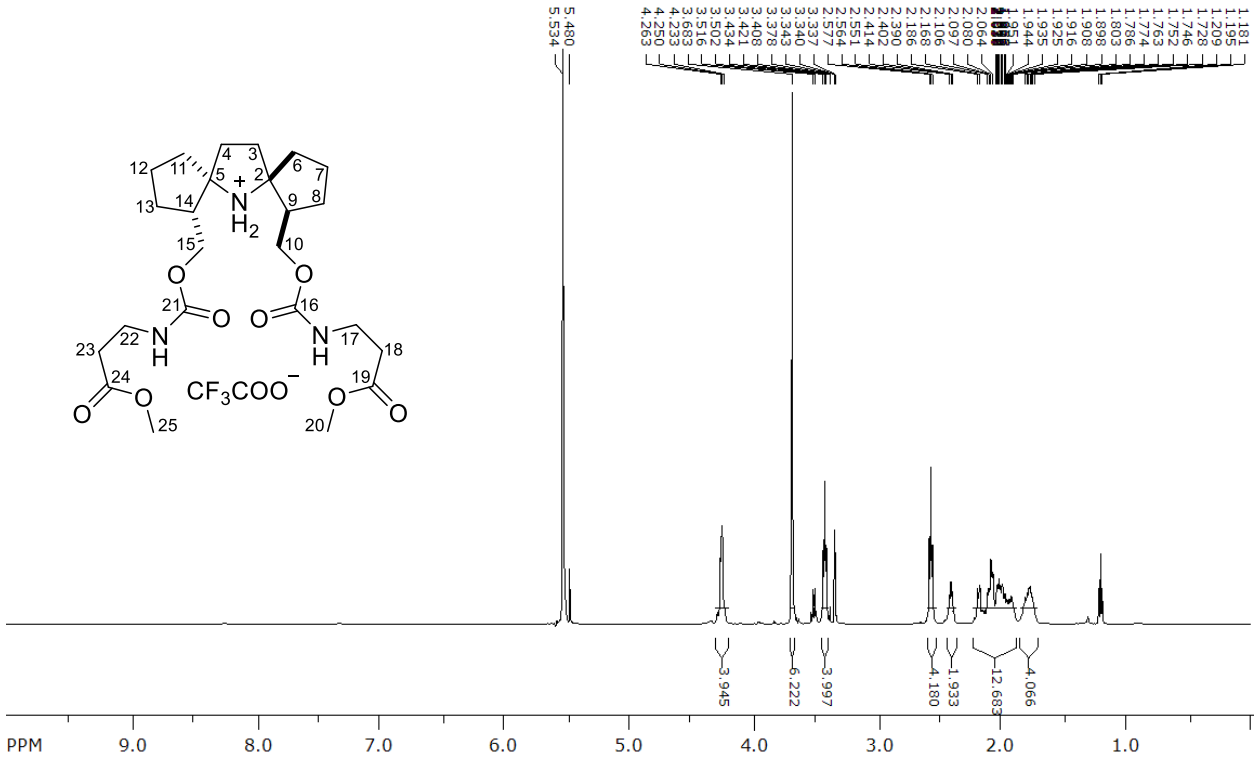


**Fig. S54.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(([{3-methoxy-3-oxopropyl}amino]carbonyl)oxy)methyl]-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**28-red**) in $CD_3OD$+$CF_3COOD$ at 500 MHz

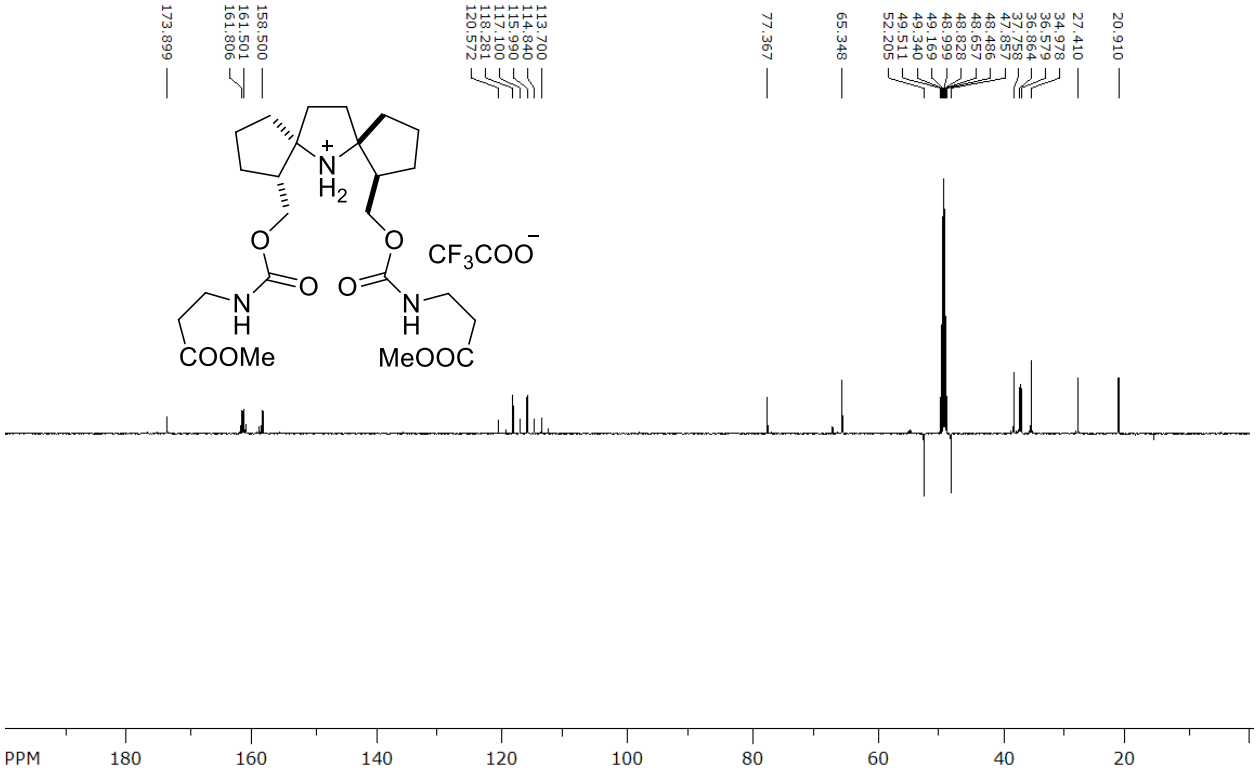


**Fig. S55.** $^{13}$C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(([{3-methoxy-3-oxopropyl}amino]carbonyl)oxy)methyl]-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**28-red**) in $CD_3OD$+$CF_3COOD$ at 125 MHz

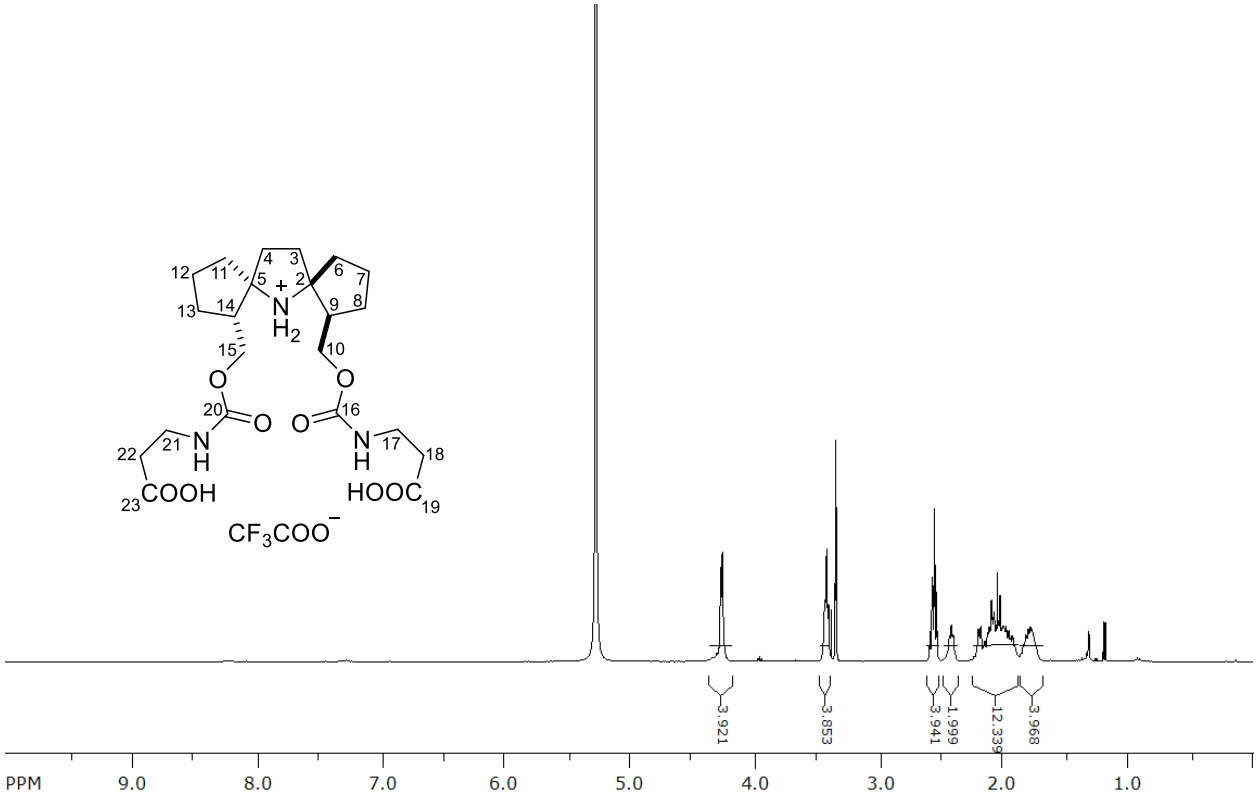


**Fig. S56.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(([{2-carboxyethyl}amino]carbonyl)oxy)methyl]-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**29-red**) in $CD_3OD$+$CF_3COOD$ at 400 MHz

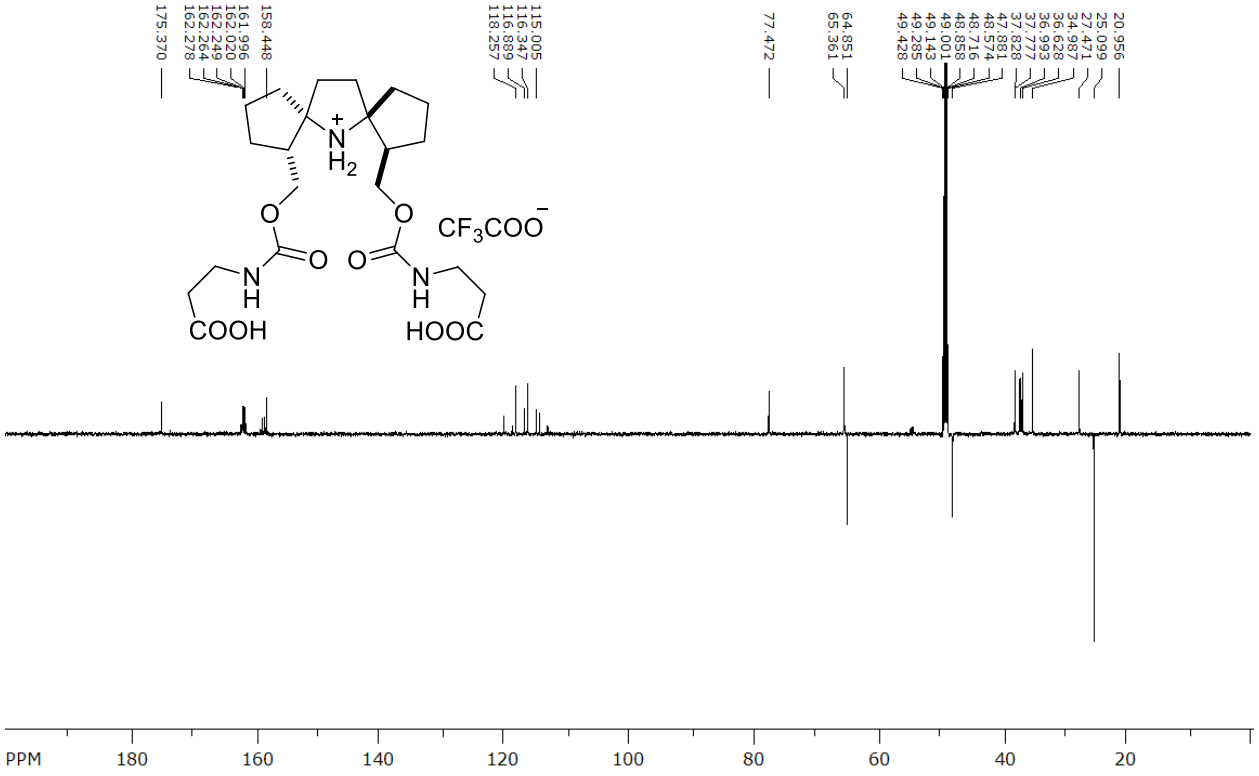


**Fig. S57.** $^{13}$C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1,8-Bis[(([{2-carboxyethyl}amino]carbonyl)oxy)methyl]-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**29-red**) in $CD_3OD$+$CF_3COOD$ at 150 MHz

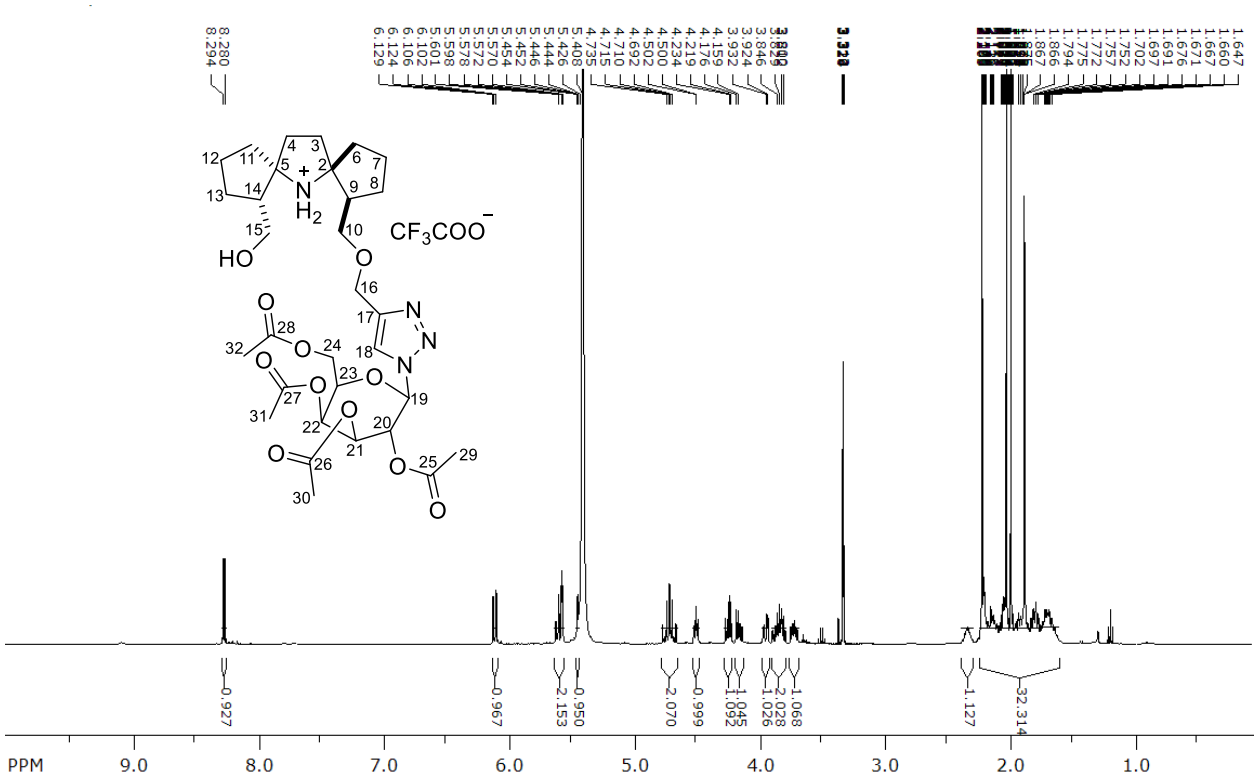


**Fig. S58.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-({(1-[2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl]-1*H*-1,2,3-triazol-4-yl)methoxy}methyl)-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**32-red**) in $CD_3OD$+$CF_3COOH$ at 400 MHz

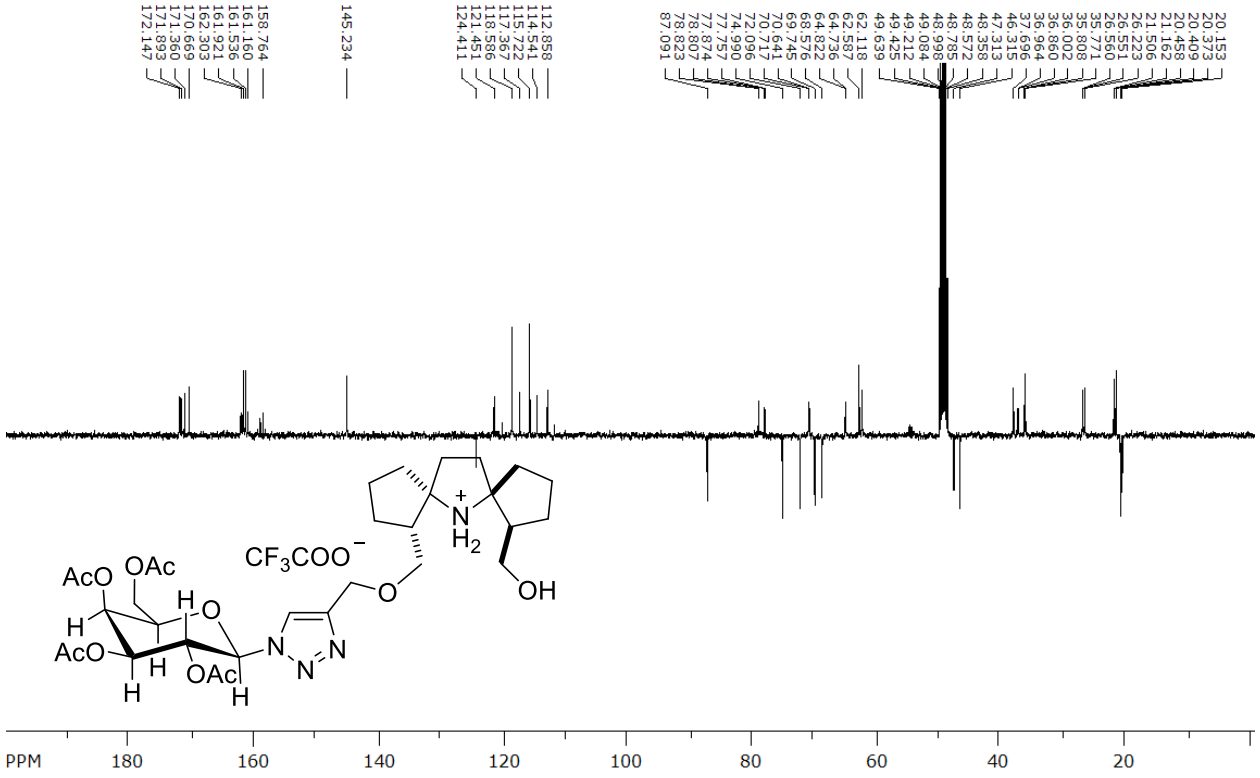


**Fig. S59.** $^{13}$C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-({(1-[2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl]-1*H*-1,2,3-triazol-4-yl)methoxy}methyl)-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**32-red**) in $CD_3OD$+$CF_3COOD$ at 100 MHz

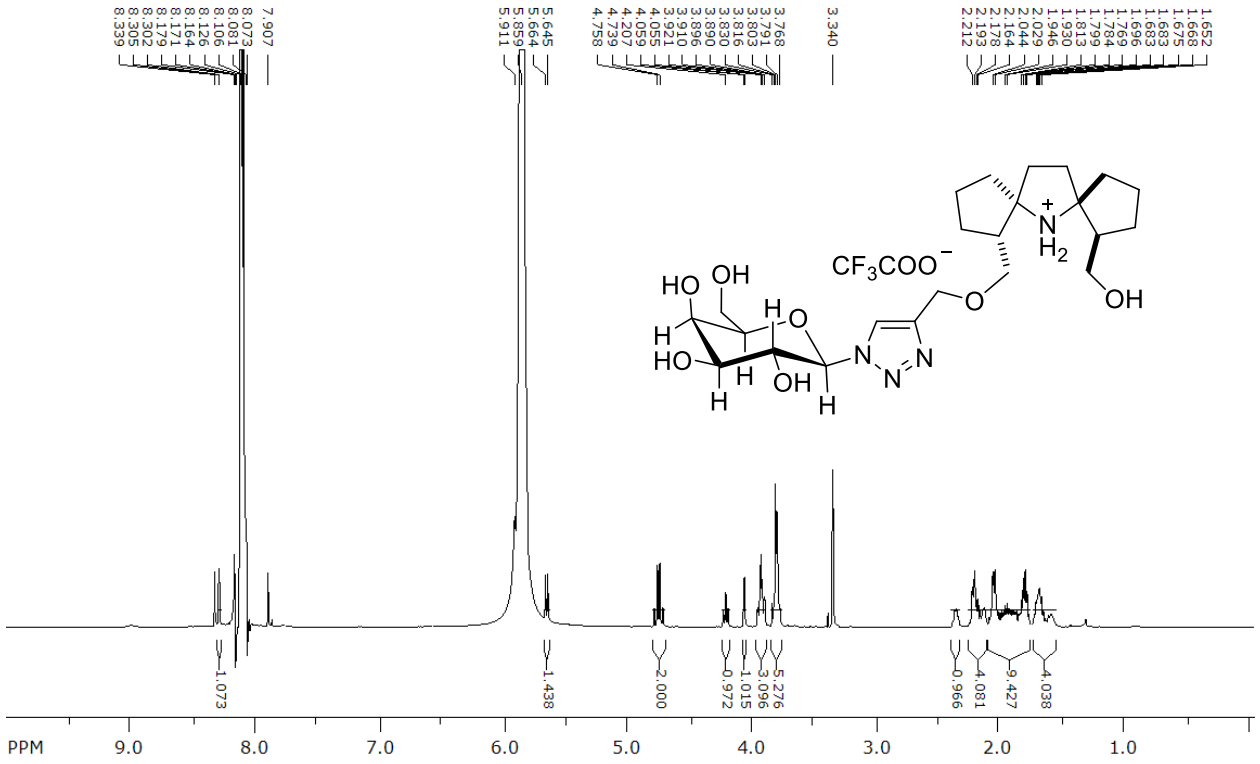


**Fig. S60.** $^{1}$H NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-({(1-[β-D-galactopyranosyl]-1*H*-1,2,3-triazol-4-yl)methoxy}methyl)-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**33-red)** in $CD_3OD$+HCOOH+HOOCCOOH at 500 MHz

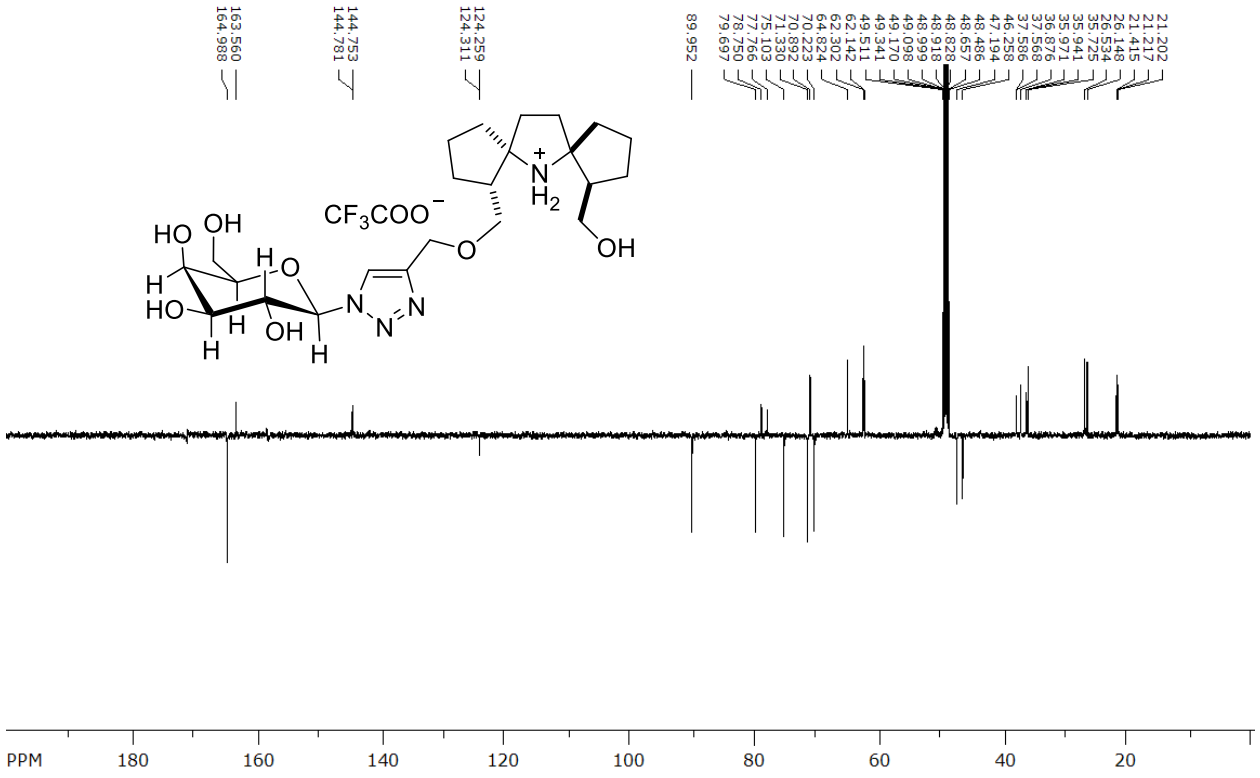


**Fig. S61.** $^{13}$C NMR spectrum of (1*R*(*S*),5*R*(*S*),7*R*(*S*),8*R*(*S*))-1-(Hydroxymethyl)-8-({(1-[β-D-galactopyranosyl]-1*H*-1,2,3-triazol-4-yl)methoxy}methyl)-6-azoniadispiro[4.1.4.2]tridecane trifluoroacetate (**33-red)** in $CD_3OD$+HCOOH+HOOCCOOH at 125 MHz

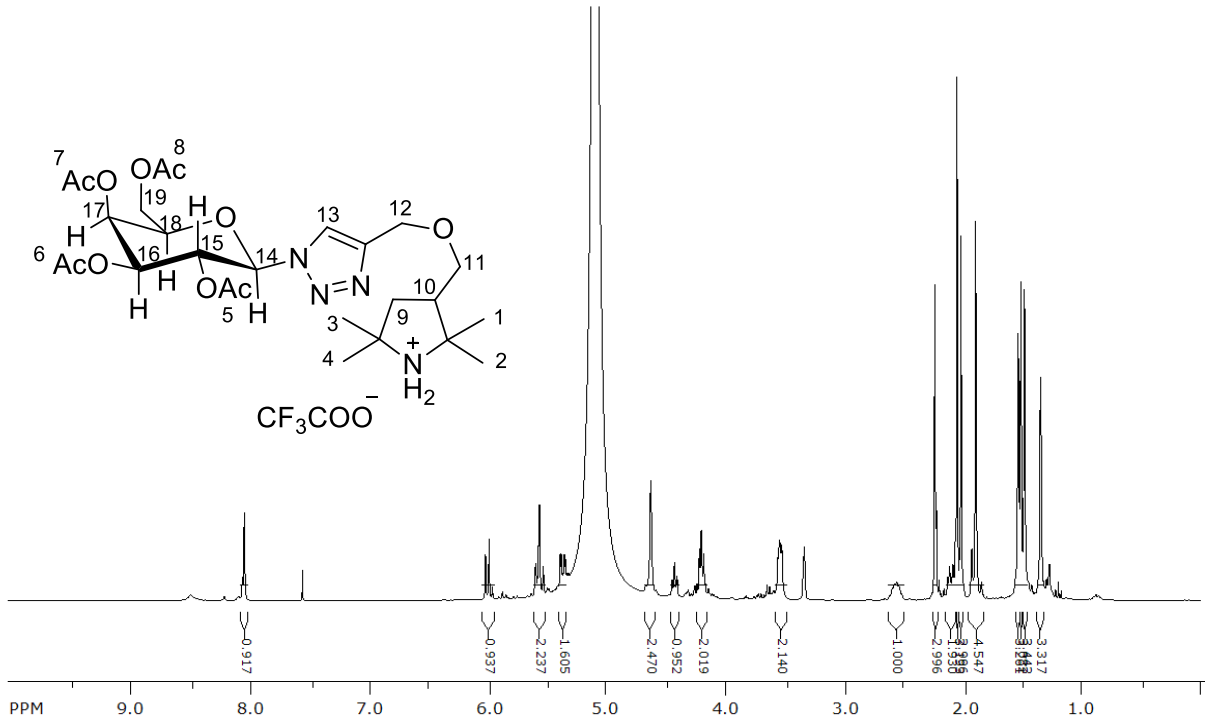


**Fig. S62.** $^1$H NMR spectrum of 2,2,5,5-Tetramethyl-3-(((1-(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyl)-1*H*-1,2,3-triazol-4-yl)methoxy)methyl)pyrrolidinium trifluoroacetate (**38-red)** in $CD_3OD$+$CF_3COOH$ at 300 MHz

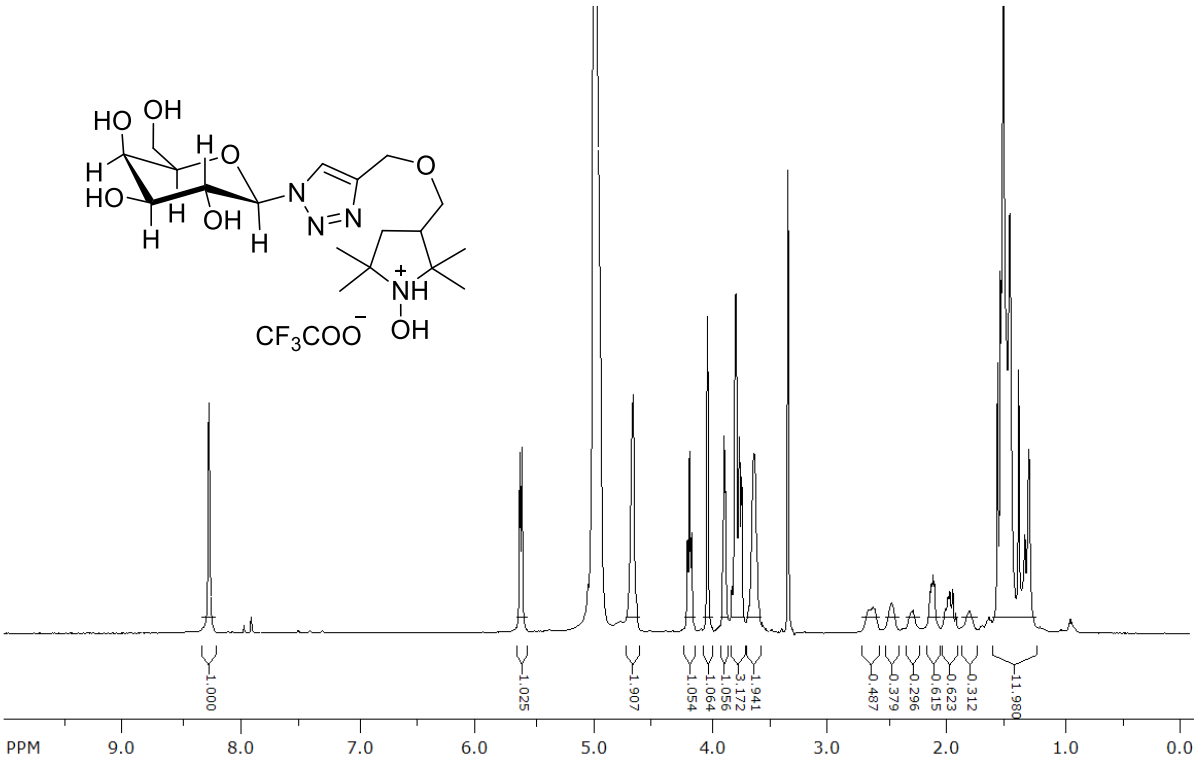


**Fig. S63.** $^{1}$H NMR spectrum of 1-Hydroxy-2,2,5,5-tetramethyl-3-(((1-(β-D-galactopyranosyl)-1*H*-1,2,3-triazol-4-yl)methoxy)methyl)pyrrolidinium trifluoroacetate (**35-red)** in $CD_3OD$+$CF_3COOH$ at 500 MHz

## 5. 2D NMR spectral data

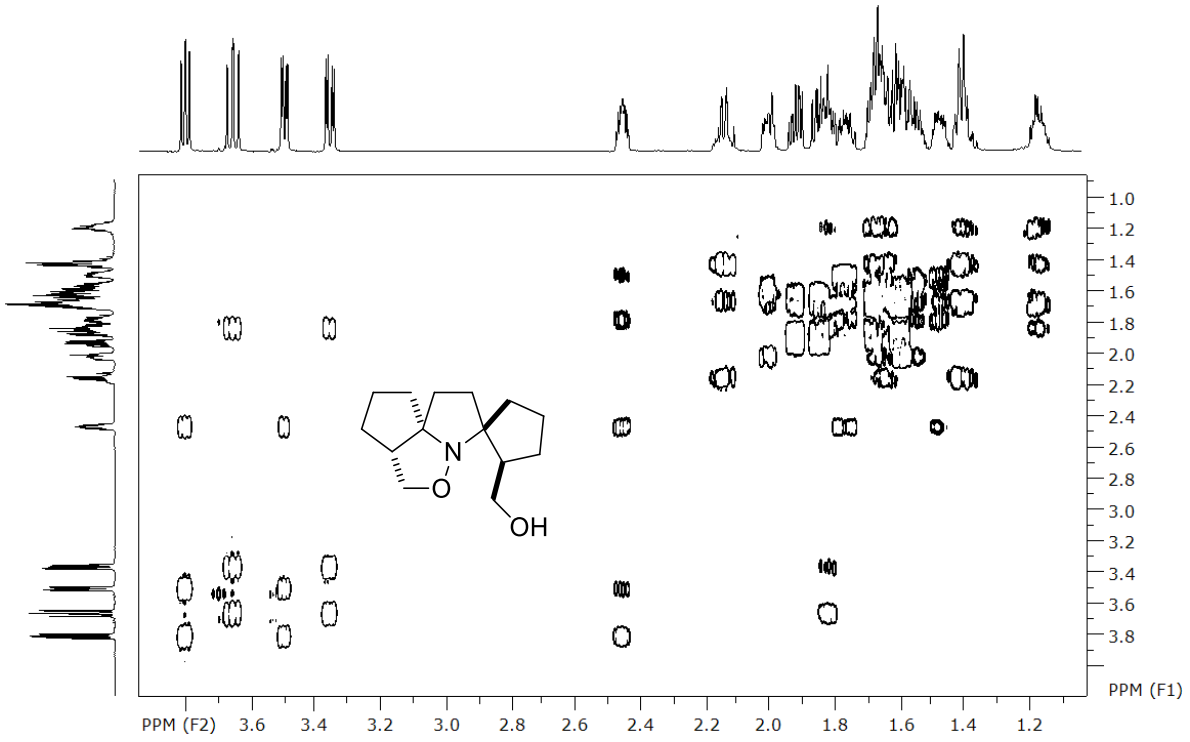


**Fig. S64.** $^{1}$H-$^{1}$H COSY NMR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*)-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) in $CDCl_3$.

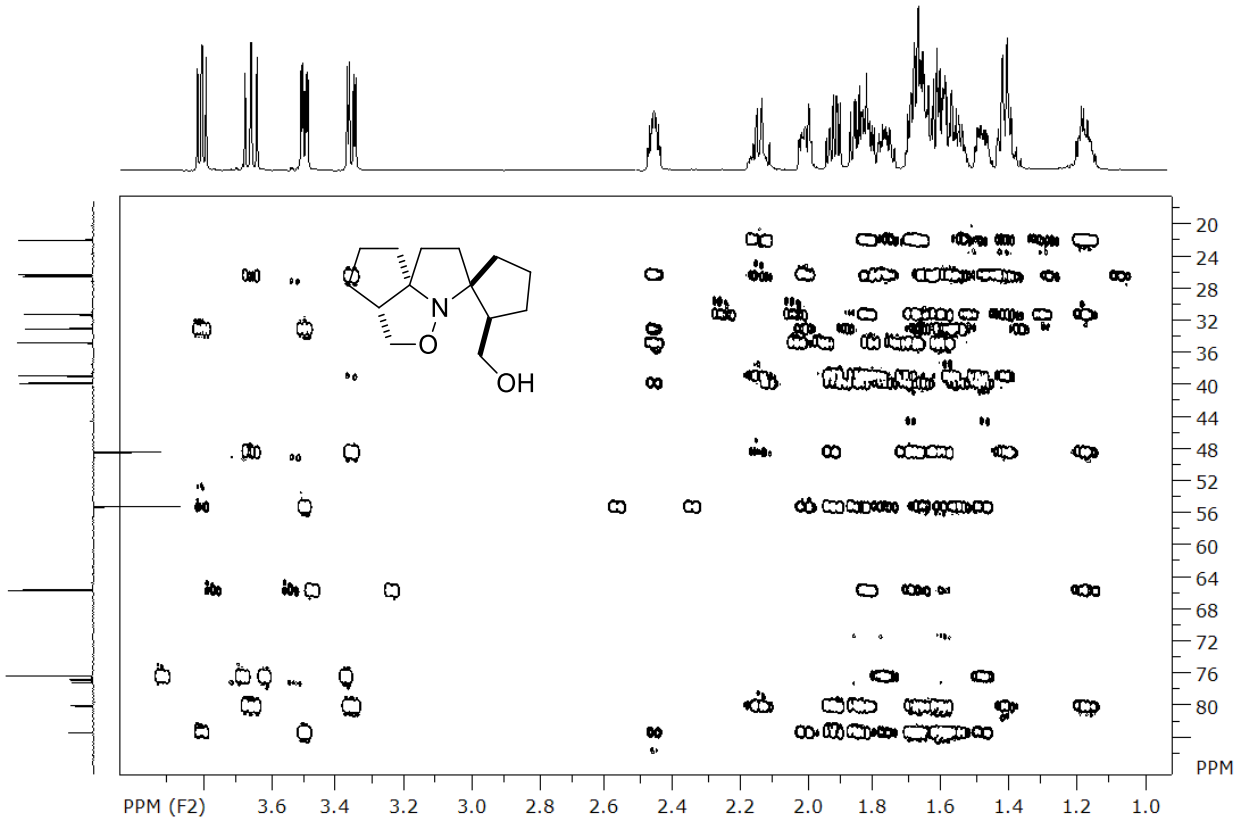


**Fig. S65.** $^{1}$H-$^{13}$C HMBC NMR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*)-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) in $CDCl_3$.

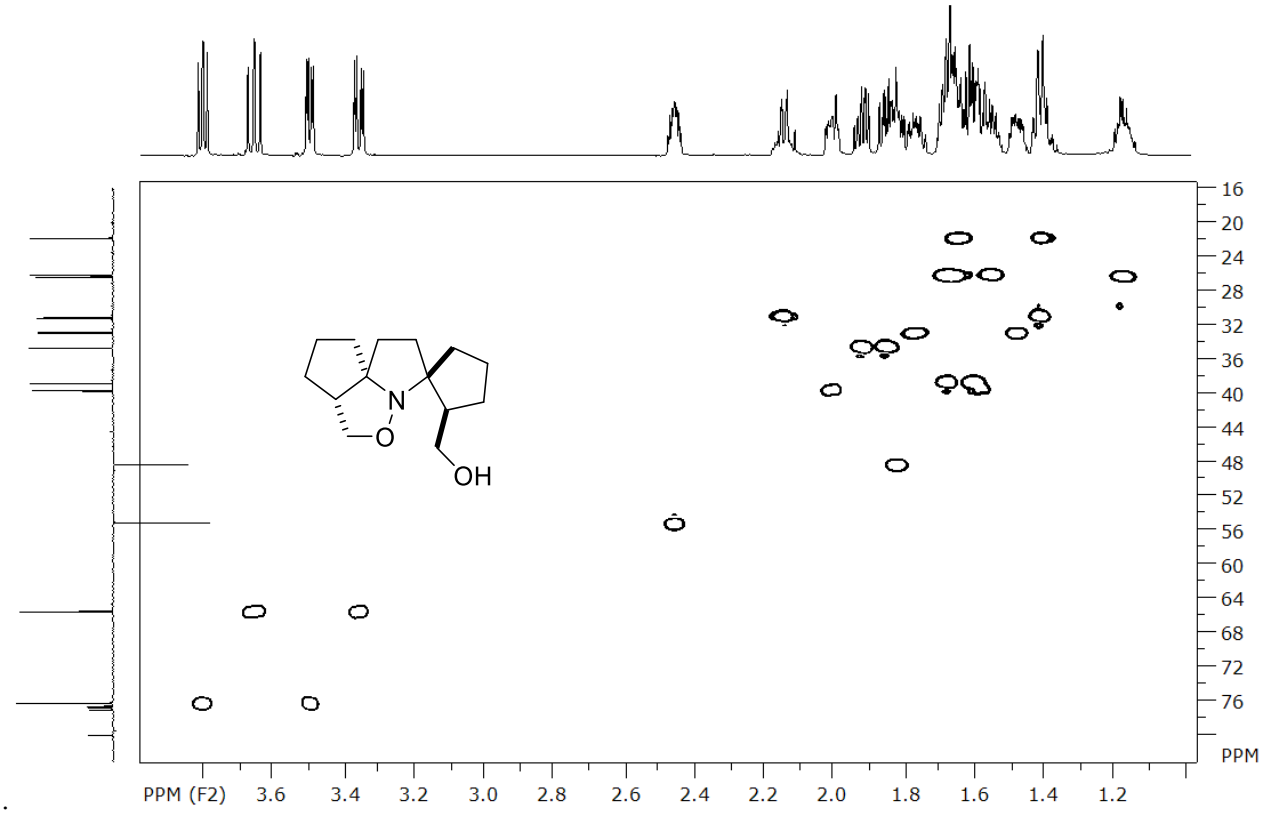


**Fig. S66.** $^{1}$H-$^{13}$C HSQC NMR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*)-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) in $CDCl_3$.

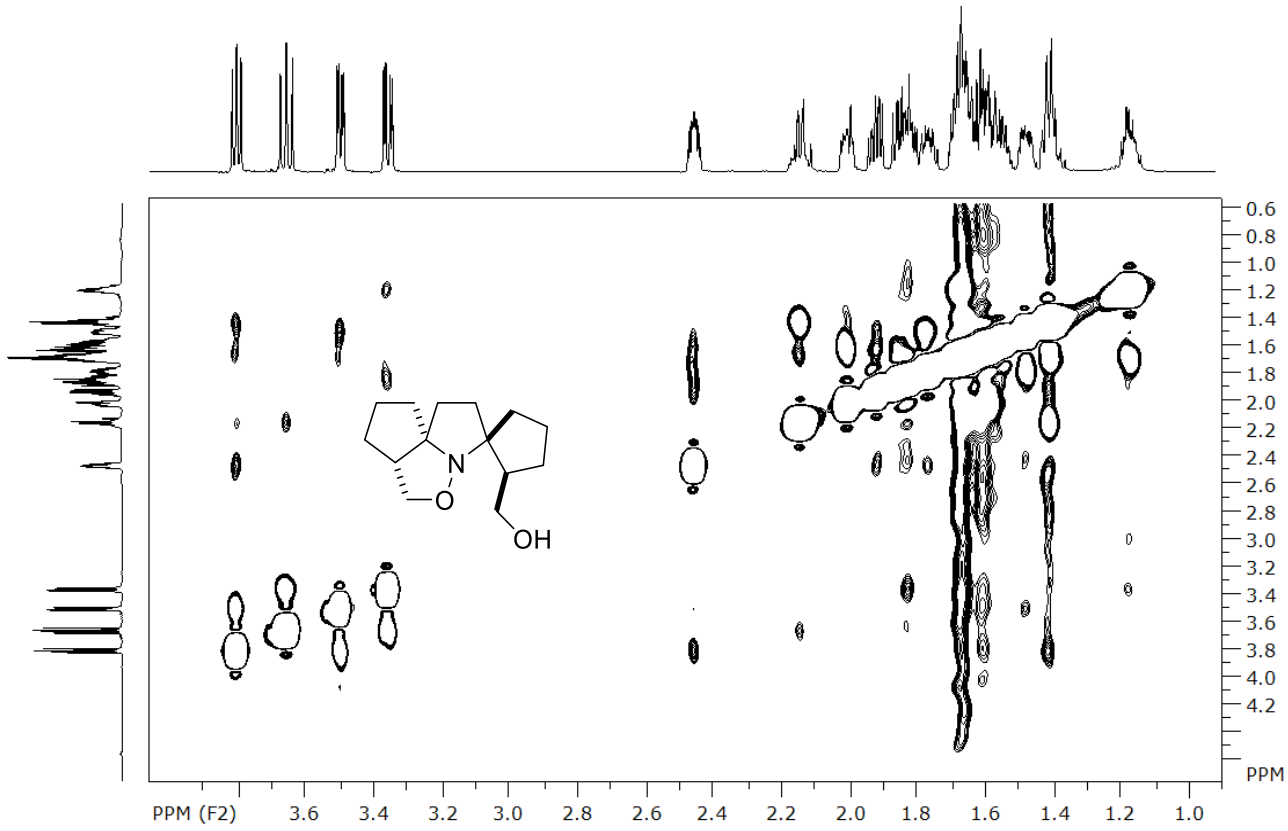


**Fig. S67.** $^{1}$H-$^{1}$H NOESY NMR spectrum of (1*R*(*S*),2*R*(*S*),6a′*R*(*S*),9a′*R*(*S*)-Hexahydro-6′*H*-spiro[cyclopentan-1,3′-cyclopenta[*c*]pyrrolo[1,2-*b*]isoxazol]-2-ylmethanol (**13**) in $CDCl_3$.

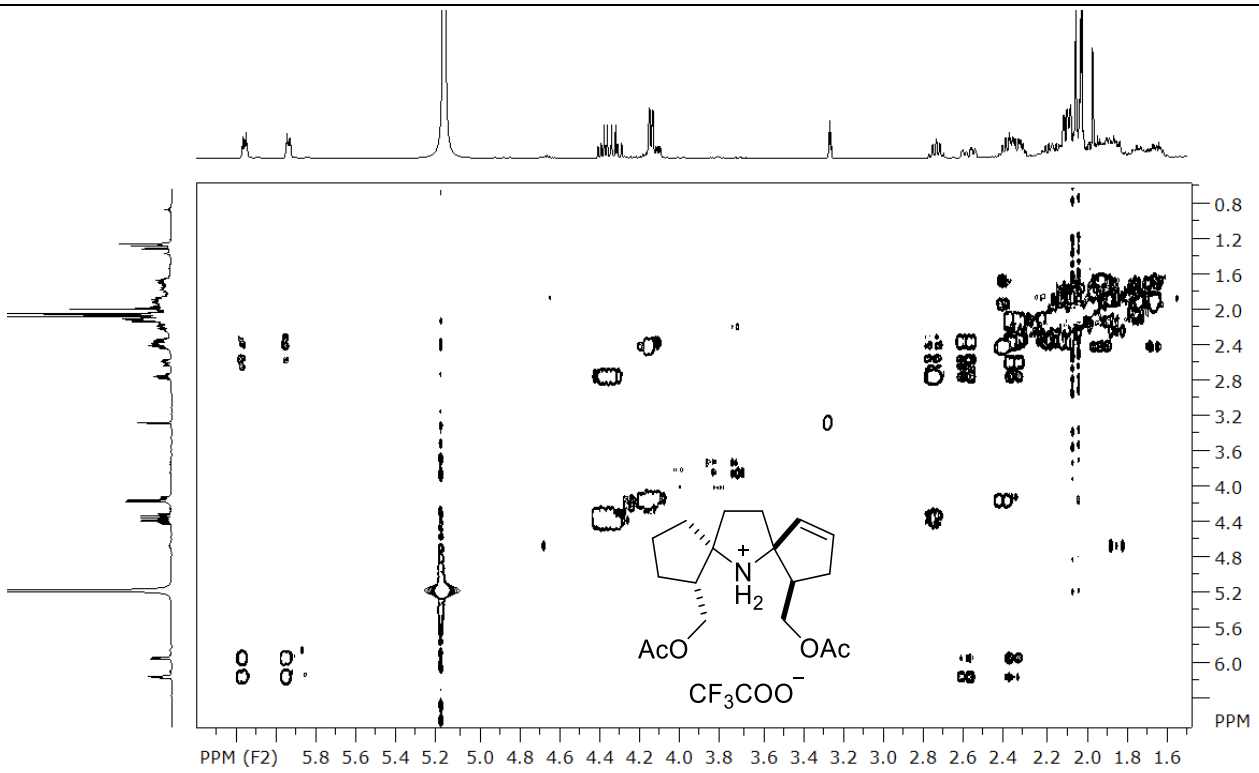


**Fig. S68.** $^{1}$H-$^{1}$H COSY NMR spectrum of (4*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-4,8-Bis[(acetyloxy)methyl]-6-azoniadispiro[4.1.4.2]tridec-1-ene trifluoroacetate (**19**) in $CD_3OD$+$CF_3COOH$.

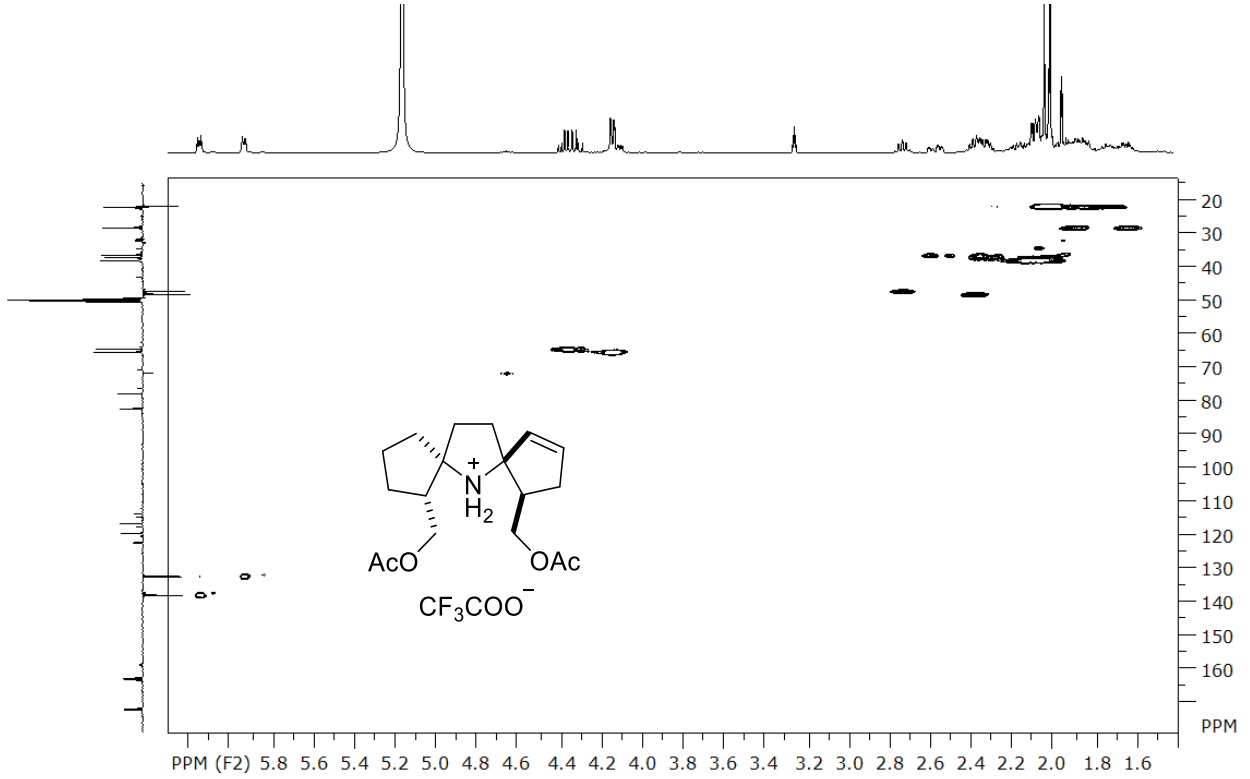


**Fig. S69.** $^{1}$H-$^{13}$C HSQC NMR spectrum of (4*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-4,8-Bis[(acetyloxy)methyl]-6-azoniadispiro[4.1.4.2]tridec-1-ene trifluoroacetate (**19**) in $CD_3OD$+$CF_3COOH$.

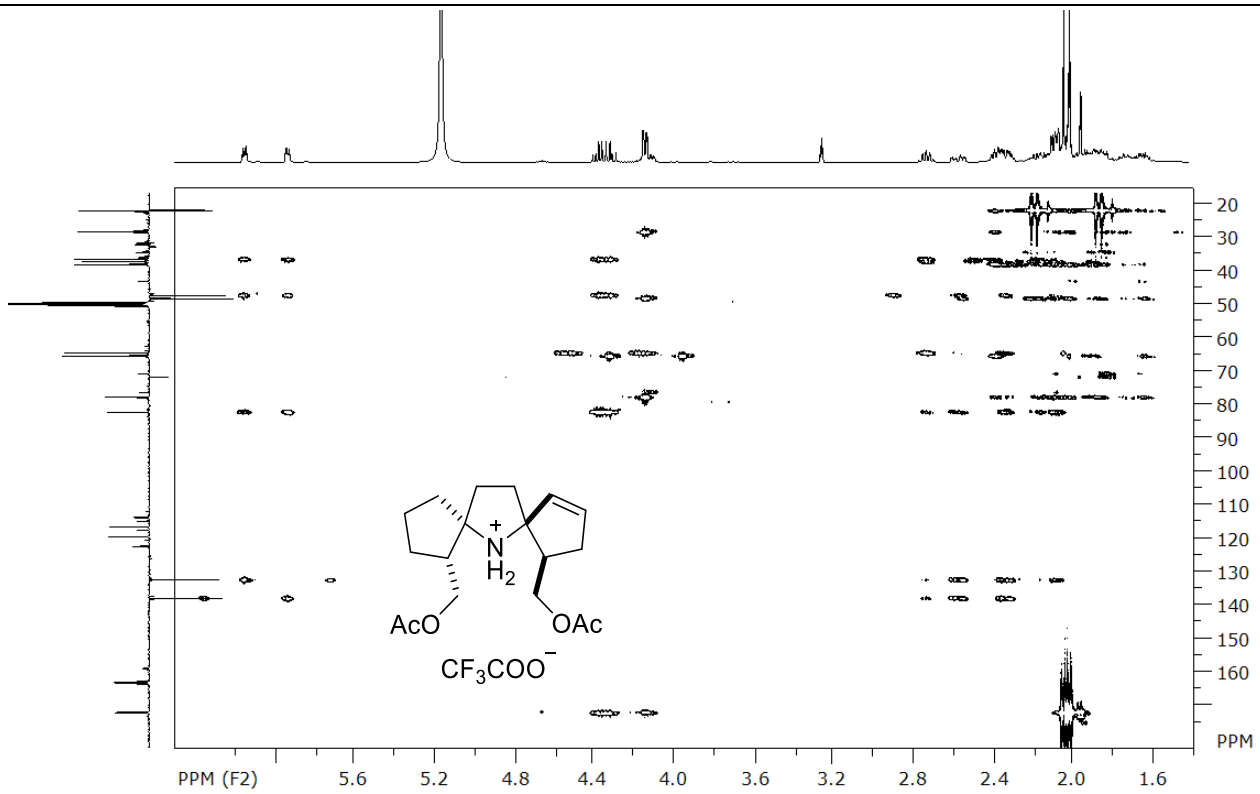


**Fig. S70.** $^{1}$H-$^{13}$C HMBC NMR spectrum of (4*R*(*S*),5*S*(*R*),7*R*(*S*),8*R*(*S*))-4,8-Bis[(acetyloxy)methyl]-6-azoniadispiro[4.1.4.2]tridec-1-ene trifluoroacetate (**19**) in $CD_3OD$+$CF_3COOH$.

## 6. EPR measurements.

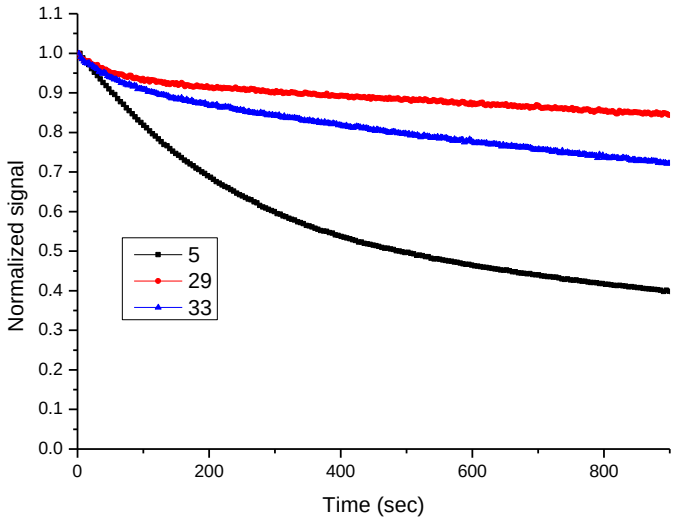


**Fig. S71**. The kinetics of decrease of EPR signal of nitroxides **5**, **29** and **33** in solution of ascorbic acid (100mM) and glutathione (5mM). These curves were fitted with exponential decay function to calculate the first order reaction rate constants, which were later used for calculation second order reaction rate constants.

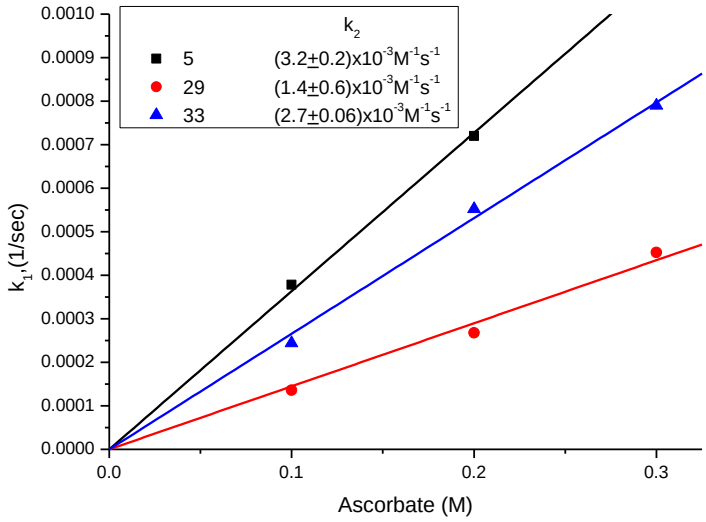


**Fig. S72**. The dependence of first order reaction rate constants versus ascorbic acid concentration of the radicals **5**, **29** and **33**. Data were fitted with linear dependence starting from zero value. The calculated slopes correspond to second order reaction rate constants.

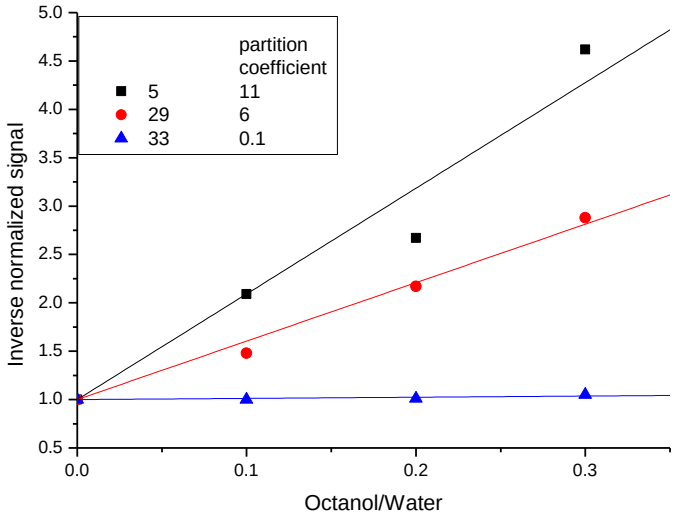


**Fig. S73**. Data for calculation of partition coefficients of the radicals **5**, **29** and **33**. The inverse value of the relative decrease of EPR signal (inverse normalized value) of the radical in the water were plotted versus octanol/water ratio. Data were fitted with linear dependence starting from one unit value. The absolute value of this slope corresponds to partition coefficient.

**Table S1.** Experimental details of X-ray diffraction experiments for compounds investigated.

| | **5** | **9** | **14** | **17** |
|---|---|---|---|---|
| Crystal data | | | | |
| Chemical formula | $C_{14}H_{24}NO_3$ | $C_9H_{15}NO_2$ | $C_{14}H_{25}NO_2$ | $C_{18}H_{28}NO_5$ |
| $M_r$ | 254.34 | 169.22 | 239.35 | 338.41 |
| Crystal system, space group | Triclinic, $P\bar{1}$ | Monoclinic, $P2_1/c$ | Monoclinic, $C2/c$ | Tetragonal, $P4_32_12$ |
| Temperature (K) | 299 | 296 | 296 | 296 |
| $a$, $b$, $c$ (Å) | 6.5386(6), 18.1055(17), 19.912(2) | 5.913(5), 11.952(11), 12.654(12) | 25.596(3), 8.3639(7), 25.239(2) | 6.9123(3), 6.9123(3), 37.383(2) |
| α, β, γ (°) | 114.383(4), 91.155(4), 96.370(4) | 90, 103.27(2), 90 | 90, 90.021(6), 90 | 90, 90, 90 |
| $V$ (Å$^3$) | 2128.1(4) | 870.4(13) | 5403.2(9) | 1786.2(2) |
| $Z$ | 6 | 4 | 16 | 4 |
| $F$(000) | 834 | 368 | 2112 | 732 |
| μ (mm$^{-1}$) | 0.08 | 0.09 | 0.08 | 0.09 |
| Crystal size (mm) | 1.00 × 0.42 × 0.20 | 1.00 × 0.38 × 0.25 | 1.00 × 0.80 × 0.15 | 1.00 × 0.79 × 0.60 |
| | | | | |
| Data collection | | | | |
| $T_{min}$, $T_{max}$ | 0.799, 0.862 | 0.742, 0.875 | 0.763, 0.862 | 0.798, 0.863 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 52841, 7588, 5736 | 6851, 1977, 1609 | 32116, 4879, 2916 | 30703, 3320, 3035 |
| $R_{int}$ | 0.052 | 0.055 | 0.043 | 0.037 |
| θ values (°) | $\theta_{max}$ = 25.1, $\theta_{min}$ = 1.1 | $\theta_{max}$ = 27.5, $\theta_{min}$ = 2.4 | $\theta_{max}$ = 25.3, $\theta_{min}$ = 2.3 | $\theta_{max}$ = 35.0, $\theta_{min}$ = 3.0 |
| Range of $h$, $k$, $l$ | $h$ = -7→7, $k$ = -21→21, $l$ = -23→23 | $h$ = -7→5, $k$ = -14→15, $l$ = -16→16 | $h$ = -30→30, $k$ = -10→10, $l$ = -30→30 | $h$ = -10→9, $k$ = -11→11, $l$ = -56→48 |
| | | | | |
| Refinement | | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.076, 0.241, 1.06 | 0.056, 0.184, 1.06 | 0.093, 0.294, 1.04 | 0.060, 0.164, 1.21 |
| No. of reflections | 7588 | 1977 | 4879 | 3320 |
| No. of parameters | 493 | 110 | 322 | 111 |
| No. of restraints | 12 | 0 | 3 | 0 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.45, -0.48 | 0.46, -0.34 | 0.47, -0.32 | 0.30, -0.20 |
| Absolute structure parameter | – | – | – | 0.52 (16) |

| | 20 | 22 | 23a | 26 |
|---|---|---|---|---|
| Crystal data | | | | |
| Chemical formula | $C_{14}H_{21}NO_4$ | $C_{16}H_{24}NO_5$ | $C_{14}H_{20}NO_5$ | $C_{22}H_{28}N_5O_5$ |
| $M_r$ | 267.32 | 310.36 | 282.31 | 442.49 |
| Crystal system, space group | Triclinic, $P^-1$ | Orthorhombic, $P2_12_12$ | Monoclinic, $P2_1/n$ | Monoclinic, $C2/c$ |
| Temperature (K) | 296 | 296 | 296 | 299 |
| $a$, $b$, $c$ (Å) | 8.4272(7), 8.9015(7), 9.3829(8) | 10.9663(7), 11.8601(7), 6.3522(4) | 9.4026(5), 11.9073(5), 12.6526(6) | 12.6524(14), 10.5198(11), 16.8648(17) |
| α, β, γ (°) | 83.367(3), 83.990(4), 66.215(3) | 90, 90, 90 | 90, 98.191(2), 90 | 90, 103.860(4), 90 |
| $V$ (Å$^3$) | 638.45(9) | 826.18(9) | 1402.13(12) | 2179.4(4) |
| $Z$ | 2 | 2 | 4 | 4 |
| $F$(000) | 288 | 334 | 604 | 940 |
| μ (mm$^{-1}$) | 0.10 | 0.09 | 0.10 | 0.10 |
| Crystal size (mm) | 0.48 × 0.12 × 0.09 | 0.54 × 0.20 × 0.10 | 0.63 × 0.50 × 0.44 | 0.60 × 0.19 × 0.16 |
| | | | | |
| Data collection | | | | |
| $T_{min}$, $T_{max}$ | 0.816, 0.862 | 0.882, 0.958 | 0.869, 0.914 | 0.926, 0.958 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15091, 2265, 1802 | 6026, 1455, 1303 | 22682, 3231, 2477 | 14914, 2385, 2035 |
| $R_{int}$ | 0.041 | 0.058 | 0.037 | 0.040 |
| θ values (°) | $\theta_{max}$ = 25.2, $\theta_{min}$ = 2.2 | $\theta_{max}$ = 25.0, $\theta_{min}$ = 3.6 | $\theta_{max}$ = 27.5, $\theta_{min}$ = 2.4 | $\theta_{max}$ = 27.0, $\theta_{min}$ = 2.5 |
| Range of $h$, $k$, $l$ | $h$ = -10→10, $k$ = -10→10, $l$ = -11→11 | $h$ = -12→13, $k$ = -11→14, $l$ = -7→7 | $h$ = -12→12, $k$ = -15→15, $l$ = -16→15 | $h$ = -15→16, $k$ = -13→13, $l$ = -21→15 |
| | | | | |
| Refinement | | | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.075, 0.198, 1.04 | 0.082, 0.250, 1.02 | 0.060, 0.192, 1.01 | 0.041, 0.119, 1.07 |
| No. of reflections | 2265 | 1455 | 3231 | 2385 |
| No. of parameters | 174 | 139 | 209 | 165 |
| No. of restraints | 0 | 47 | 9 | 0 |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.37, -0.29 | 0.54, -0.32 | 0.50, -0.41 | 0.22, -0.14 |
| Absolute structure parameter | – | -0.4 (8) | – | – |