

Supporting Information

Synthesis, Chiral Resolution and Enantiomers Absolute Configuration of 4-Nitropropranolol and 7-Nitropropranolol

Rosa Sparaco,¹ Antonia Scognamiglio,¹ Angela Corvino,¹ Giuseppe Caliendo,¹ Ferdinando Fiorino,¹ Elisa Magli,² Elisa Perissutti,¹ Vincenzo Santagada,¹ Beatrice Severino,¹ Paolo Luciano,¹ Marcello Casertano,¹ Anna Aiello,¹ Gilberto De Nucci,^{3,*} Francesco Frecentese¹

¹ *Department of Pharmacy, University of Naples Federico II, Via D. Montesano 49, Naples, Italy*

² *Department of Public Health, University of Naples Federico II, Via D. Montesano 49, Naples, Italy*

³ *Department of Pharmacology, Faculty of Medical Sciences, State University of Campinas (UNICAMP), São Paulo, Campinas, Brazil*

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Peak 1 - (+)-4-Nitropropranolol

Analysis results:

Item	Specification	Result
Appearance	Yellow powder	Compliant
Purity	LC-MS purity >95%	Compliant
Enantiomeric purity	HPLC 99%	Compliant

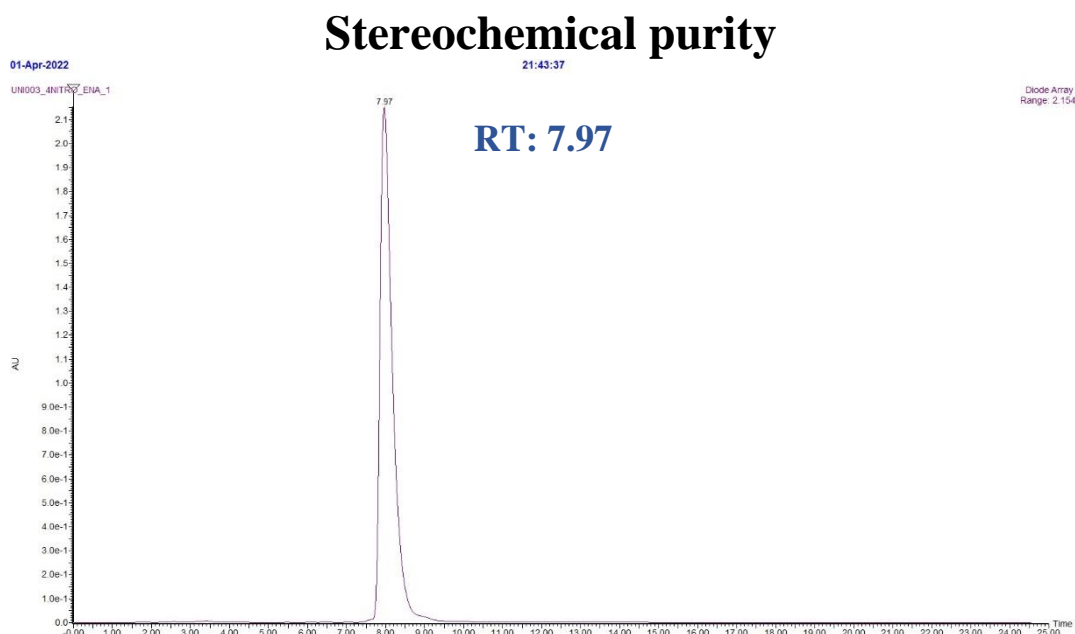
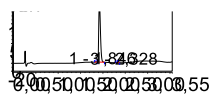


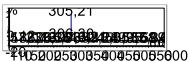
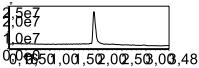
Figure S1 – chiral analytical HPLC: analysis of (+)-4-nitropropanolol

Sample:	4-Nitropropanolol – Peak 1	Flow:	1 mL/min
Column:	5 AMY-Coat -5 µm Phenomenex	Wavelength:	254/280 nm
Dimension:	150 x 4.6 mm	Mobile Phase:	N-Hexane-Isopropanol 86:14 (+0.1% DEA)

LC-MS Analysis - Peak 1 - (+)-4-Nitropropranolol



Peak Name	Retention Time min	Area mAU*min	Relative Area %
1	1.846	0.046	0,52
2	1.923	6.63	98.6
3	2.38	0.055	0.82



Peak 2 - (-)-4-Nitropropanolol

Analysis results:

Item	Specification	Result
Appearance	Yellow powder	Compliant
Purity	LC-MS purity >95%	Compliant
Enantiomeric purity	HPLC 99%	Compliant

Stereochemical purity

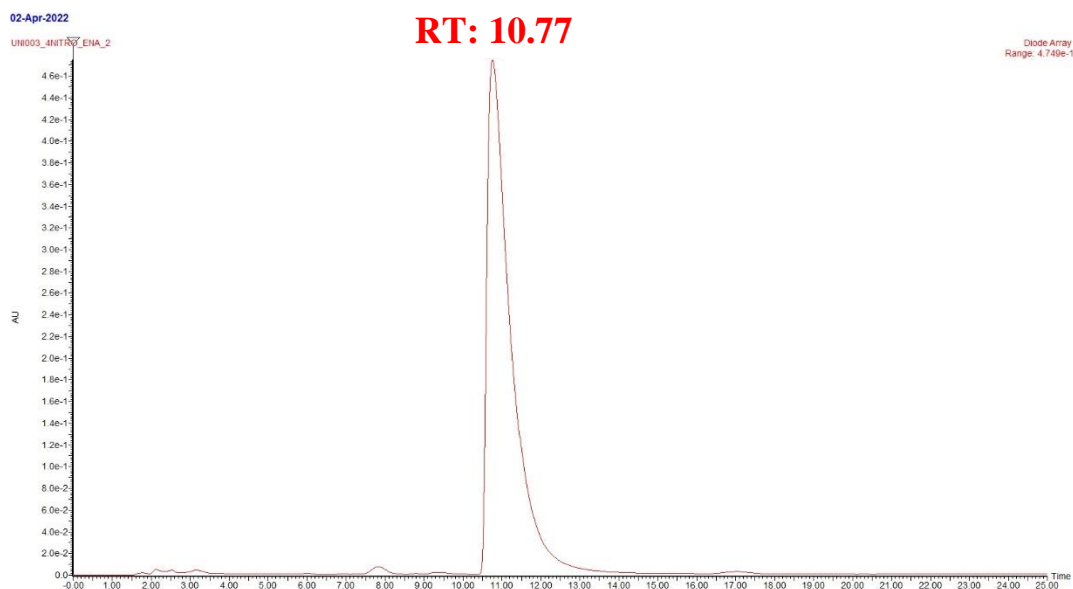
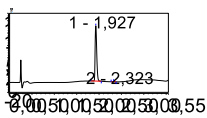


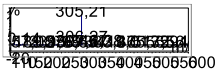
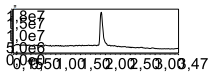
Figure S2 – chiral analytical HPLC: analysis of (*l*)-4-nitropropanolol

Sample:	4-Nitropropanolol – Peak 2	Flow:	1 mL/min
Column:	5 AMY-Coat -5 µm Phenomenex	Wavelength:	254/280 nm
Dimension:	150 x 4.6 mm	Mobile Phase:	N-Hexane-Isopropanol 86:14 (+0.1% DEA)

LC-MS Analysis



Peak Name	Retention Time min	Area mAU*min	Relative Area %
1	1.927	4.36	99.4
2	2.323	0.02	0.58



Peak 1 - (+)-7-Nitropropranolol

Analysis results:

Item	Specification	Result
Appearance	Yellow powder	Compliant
Purity	LC-MS purity >95%	Compliant
Enantiomeric purity	HPLC 99%	Compliant

Stereochemical purity

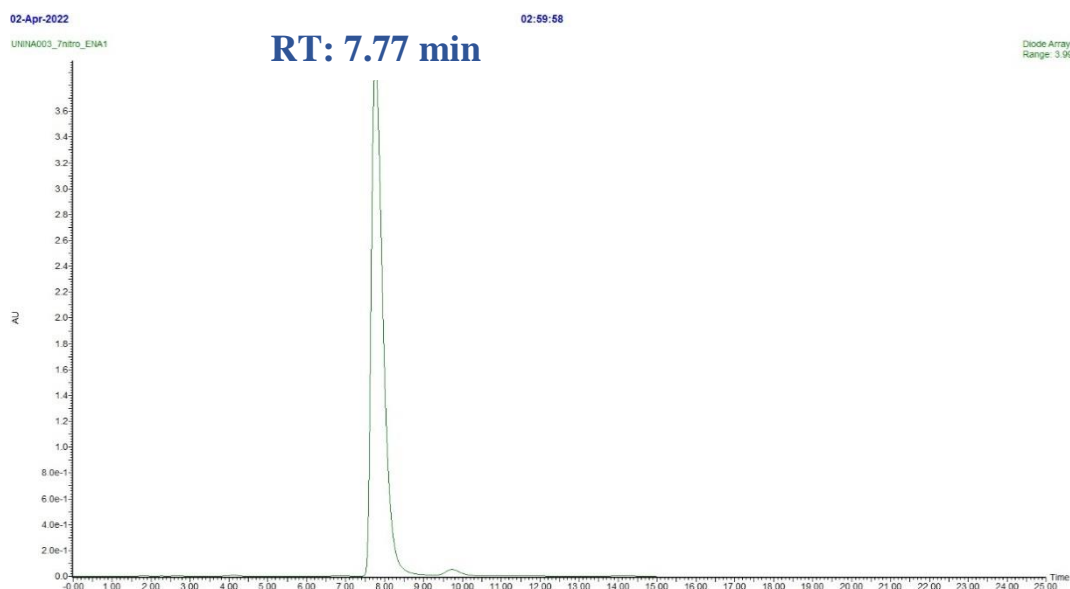
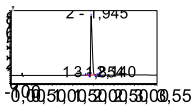


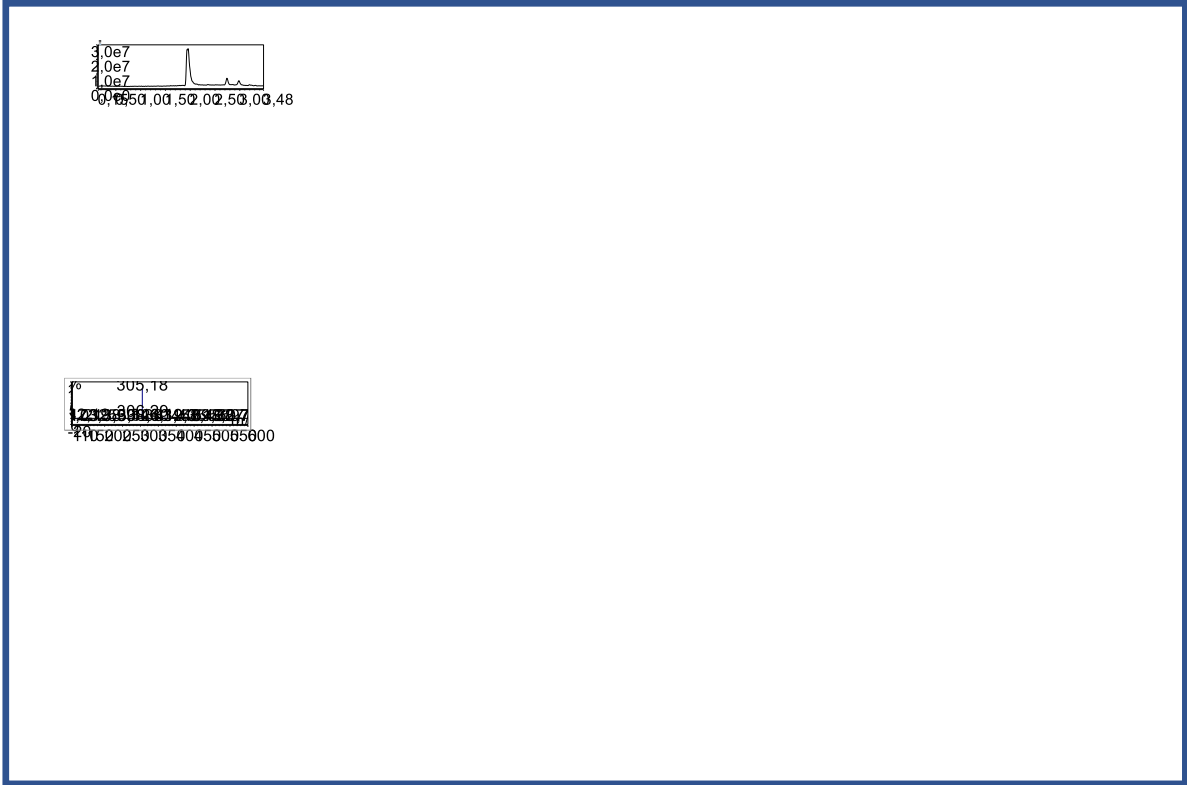
Figure S3 – chiral analytical HPLC: analysis of (d)-7-nitropropranolol

Sample:	7-Nitropropranolol – Peak 1	Flow:	1 mL/min
Column:	5 AMY-Coat -5 µm Phenomenex	Wavelength:	254/280 nm
Dimension:	150 x 4.6 mm	Mobile Phase:	N-Hexane-Isopropanol 86:14 (+0.1% DEA)

LC-MS Analysis



Peak Name	Retention Time min	Area mAU*min	Relative Area %
1	1.854	0.077	0.24
2	1.945	31.29	99.4
3	2.140	0.12	0.39



Peak 2 - (-)-7-Nitropropranolol

Analysis results:

Item	Specification	Result
Appearance	Yellow powder	Compliant
Purity	LC-MS purity >95%	Compliant
Enantiomeric purity	HPLC 99%	Compliant

Stereochemical purity

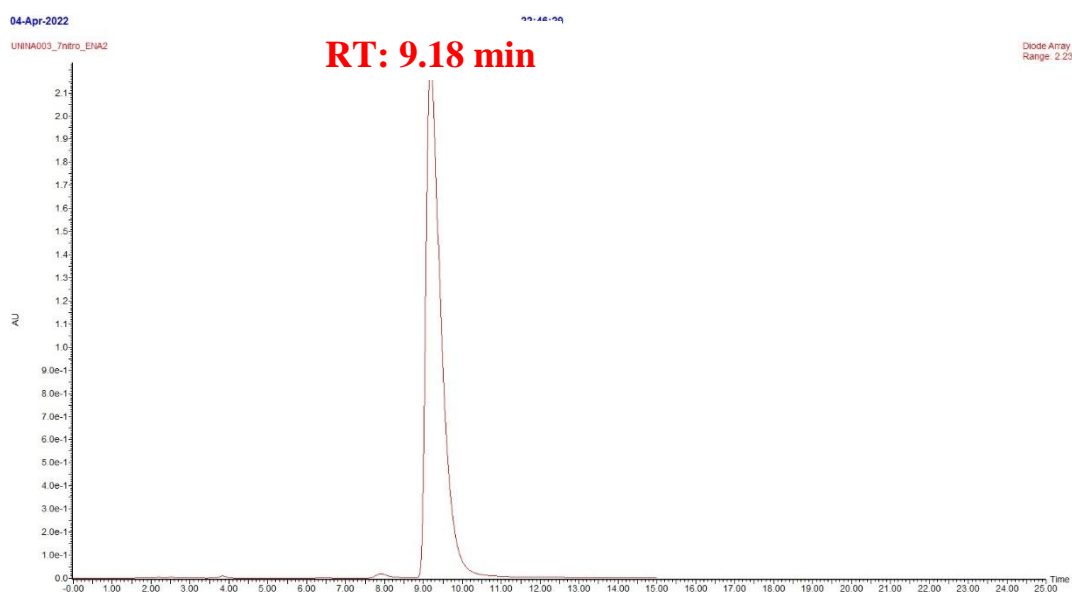
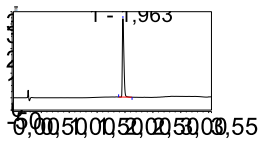


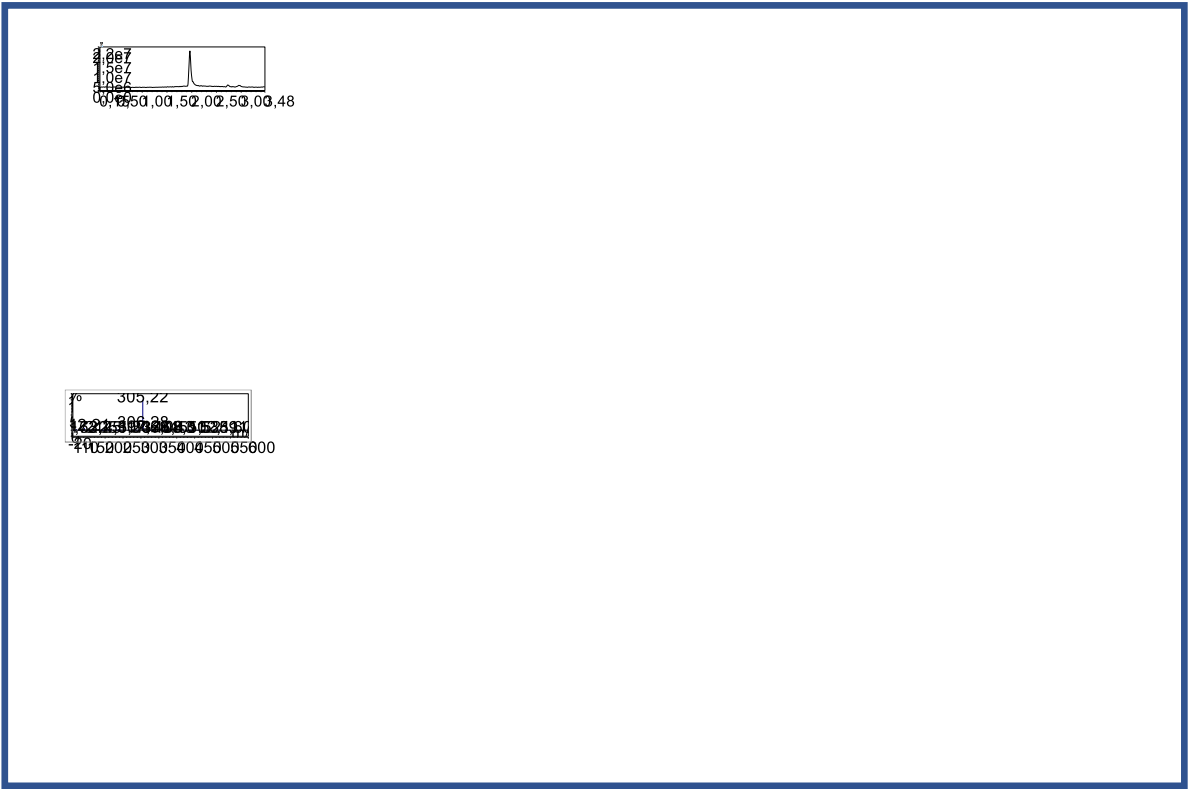
Figure S4 – chiral analytical HPLC: analysis of (1)-7-nitropropranolol

Sample:	7-Nitropropranolol – Peak 2	Flow:	1 mL/min
Column:	5 AMY-Coat -5 µm Phenomenex	Wavelength:	254/280 nm
Dimension:	150 x 4.6 mm	Mobile Phase:	N-Hexane-Isopropanol 86:14 (+0.1% DEA)

LC-MS Analysis



Peak Name	Retention Time min	Area mAU*min	Relative Area %
1	1.963	10.13	100



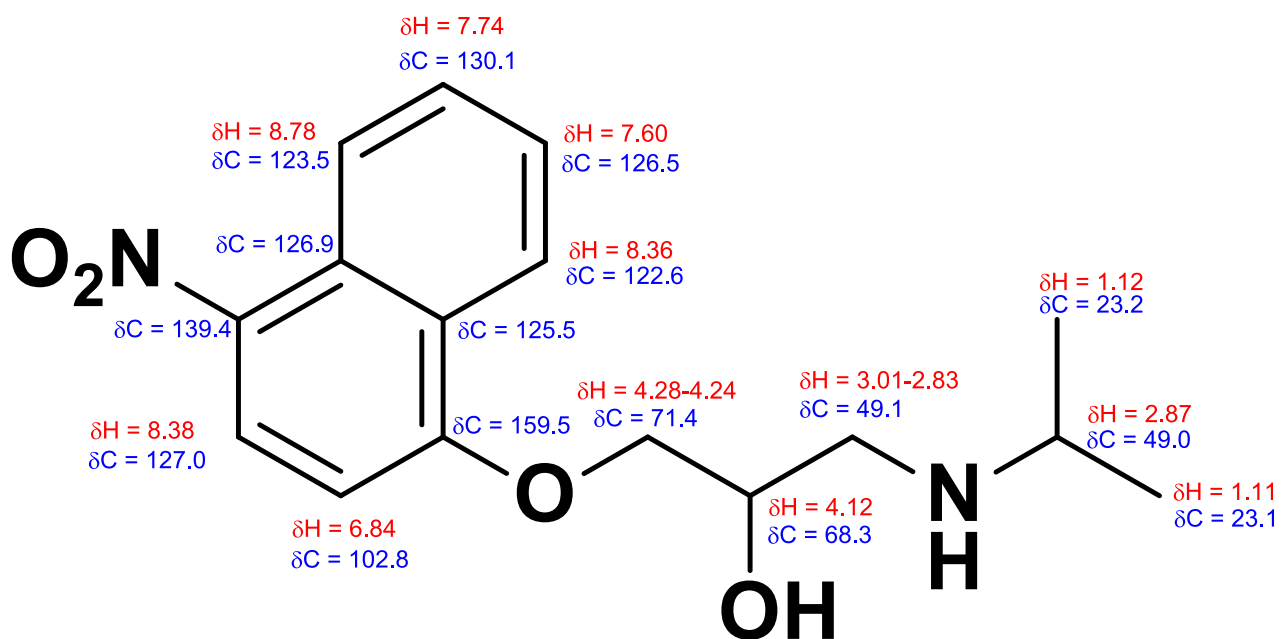


Figure S5 – NMR signals of 4-nitropropranolol **2a**. NMR data obtained from racemic mixture, (+)-enantiomer and (-)-enantiomer are the same.

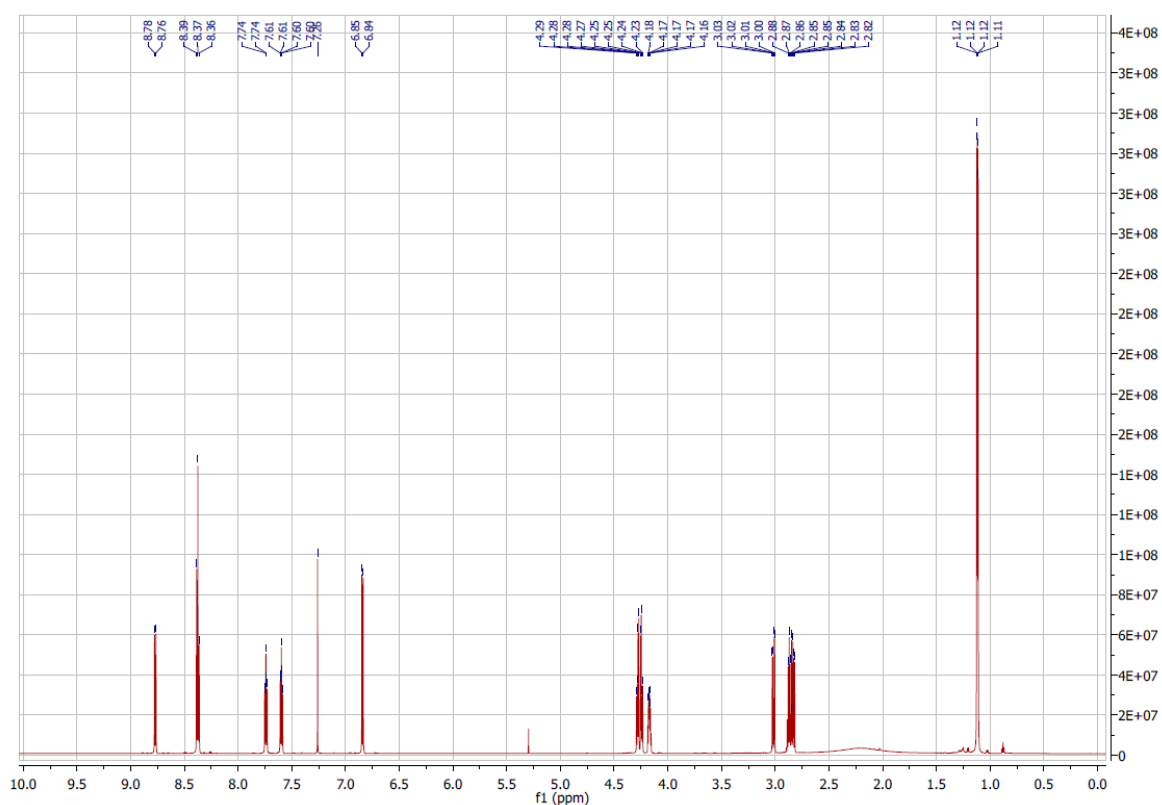


Figure S6 - ¹H-NMR 4-Nitropropanol (CDCl₃)

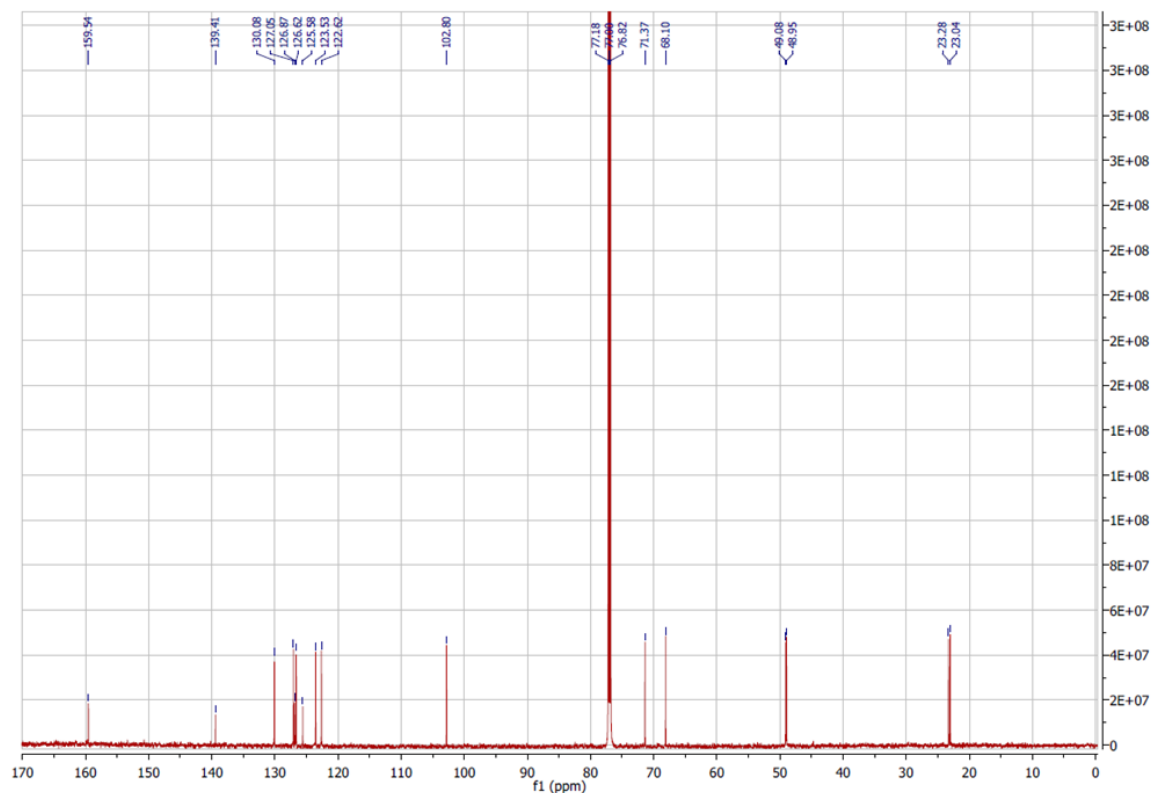


Figure S7 - ¹³C-NMR 4-Nitropropanol (CDCl₃)

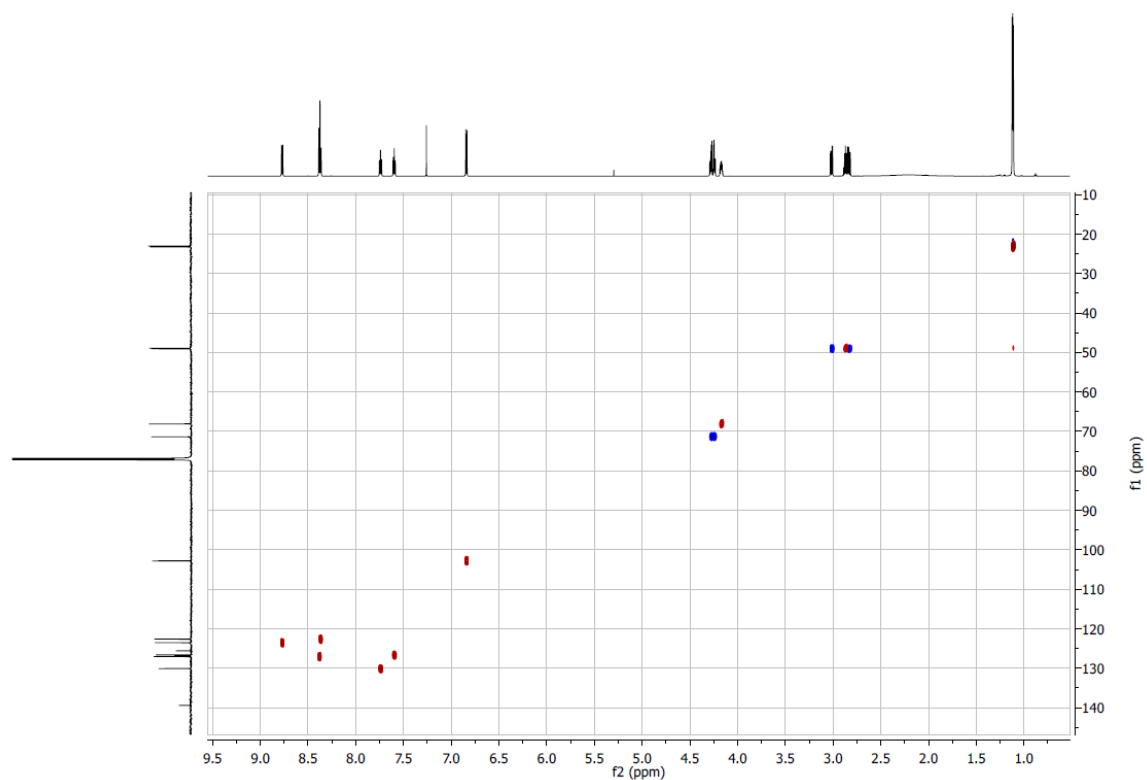


Figure S8 - HSQC 4-Nitropropanol

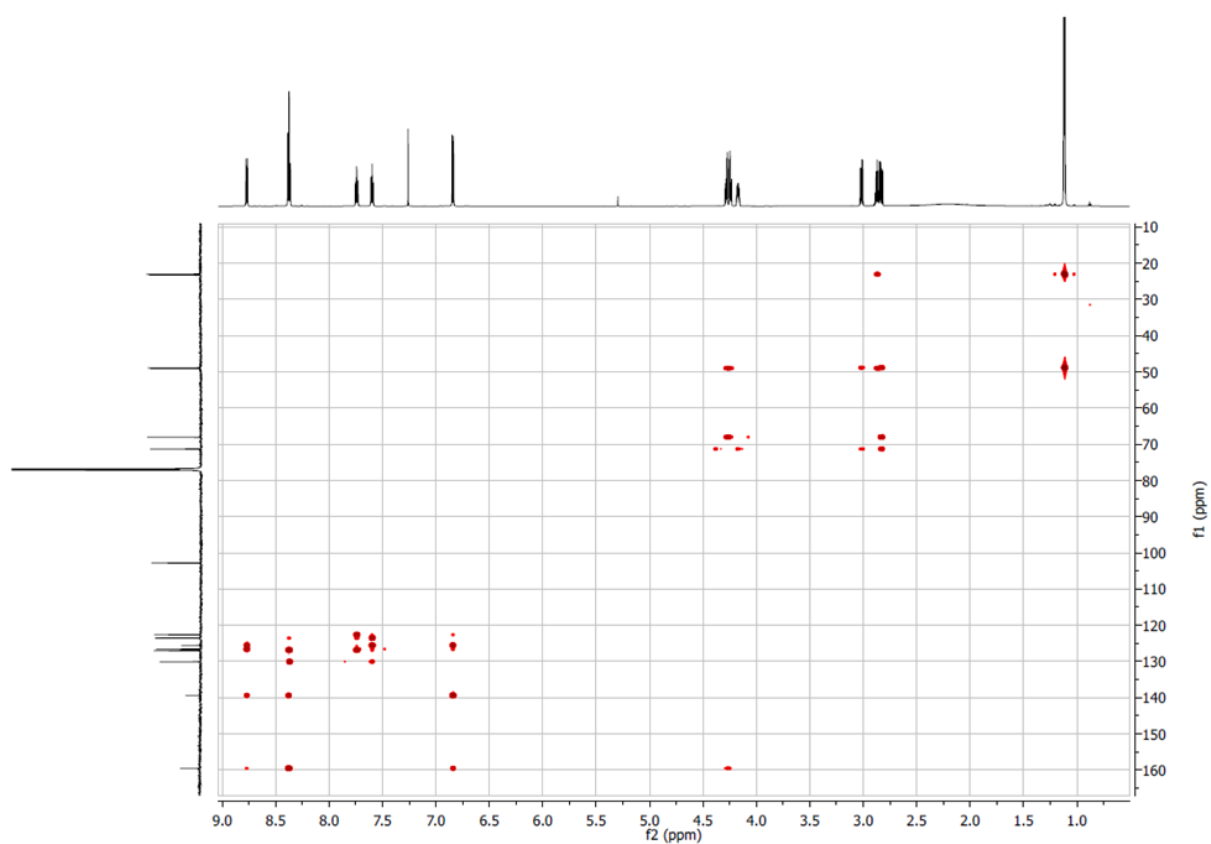


Figure S9 - HMBC 4-Nitropropanol

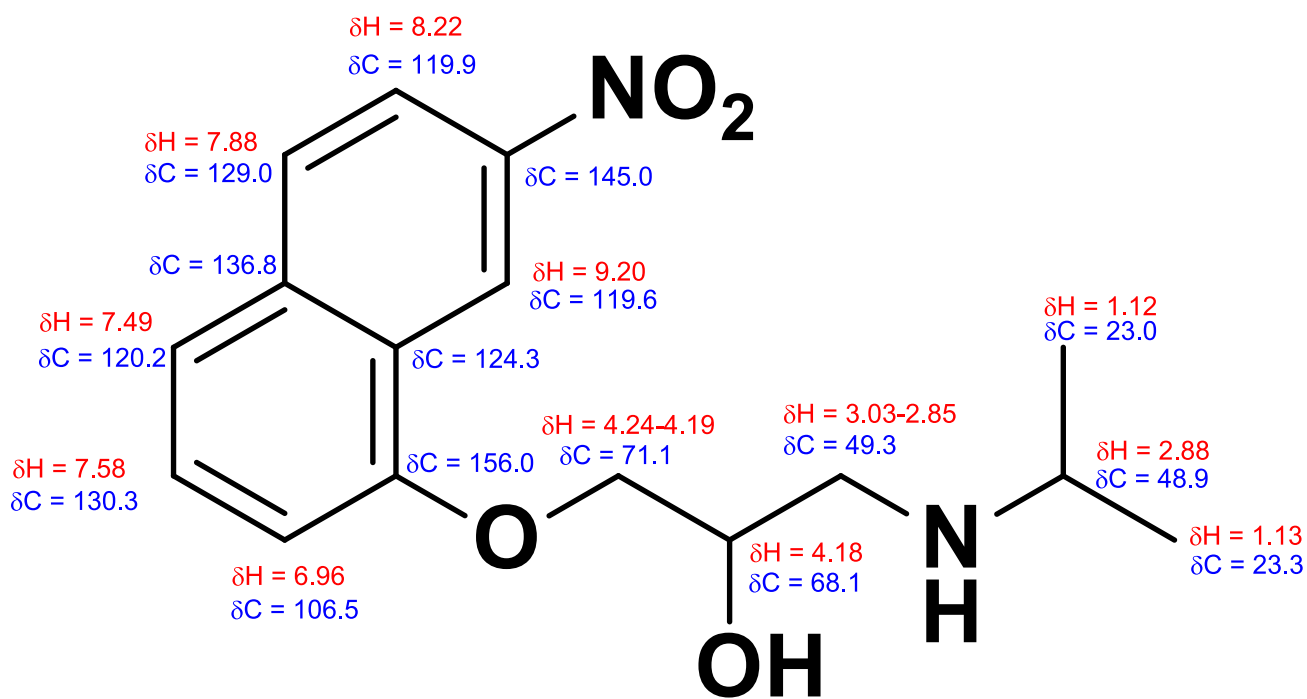


Figure S10 – NMR signals of 7-nitropropranolol **2b**. NMR data obtained from racemic mixture, (+)-enantiomer and (-)-enantiomer are the same.

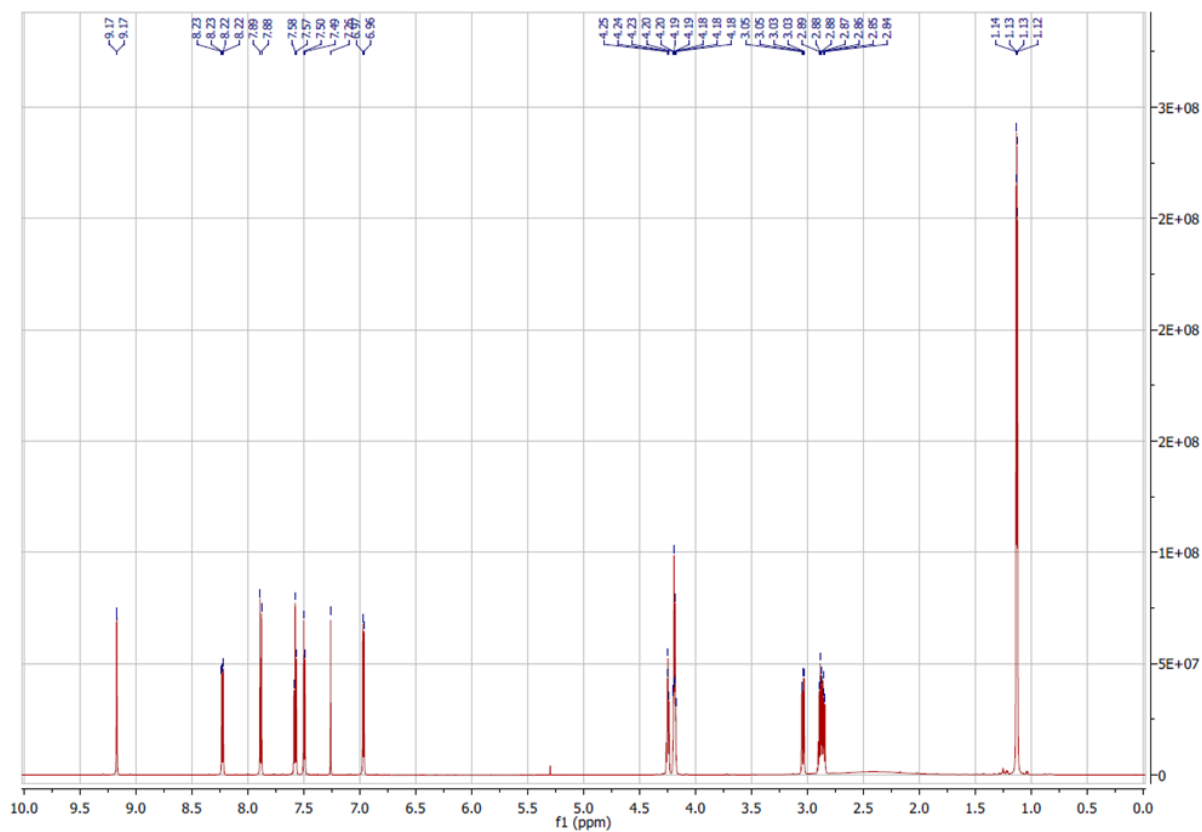


Figure S11 - ^1H -NMR 7-Nitropropanolol (CDCl_3)

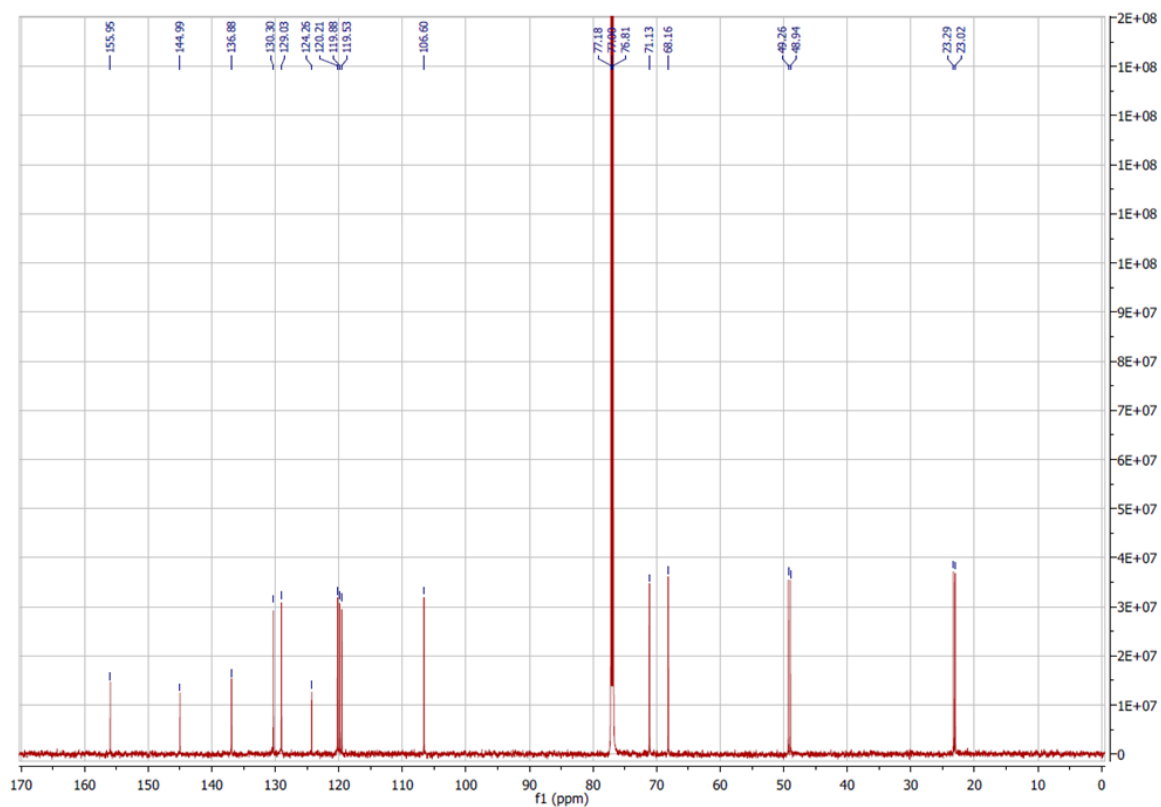


Figure S12 - ^{13}C -NMR 7-Nitropropanolol (CDCl_3)

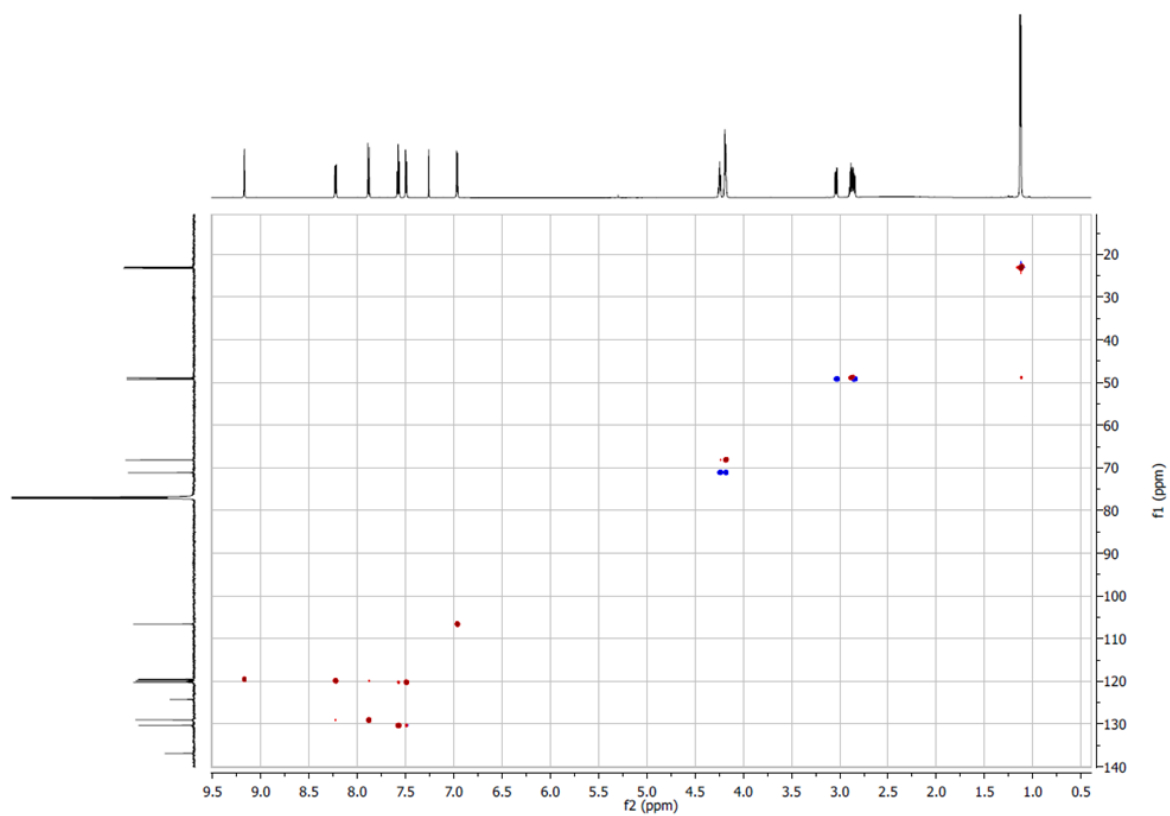


Figure S13 - HSQC 7-Nitropropranolol

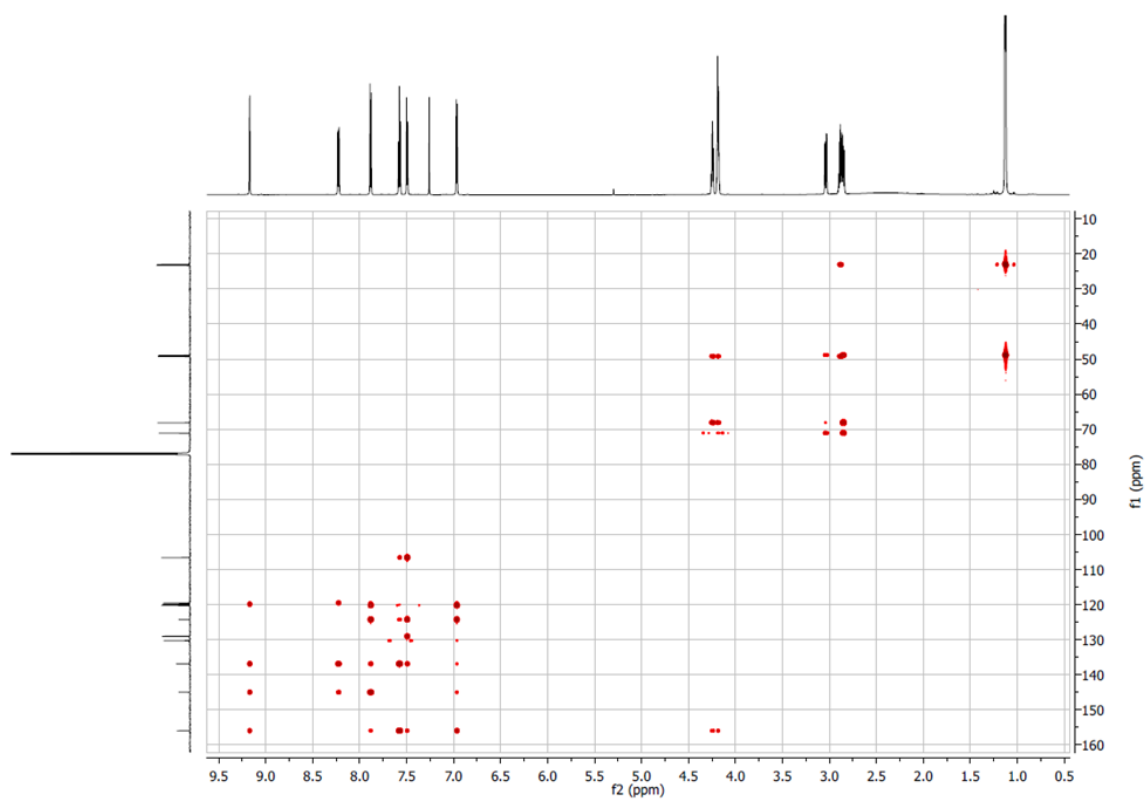


Figure S14 - HMBC 7-Nitropropranolol

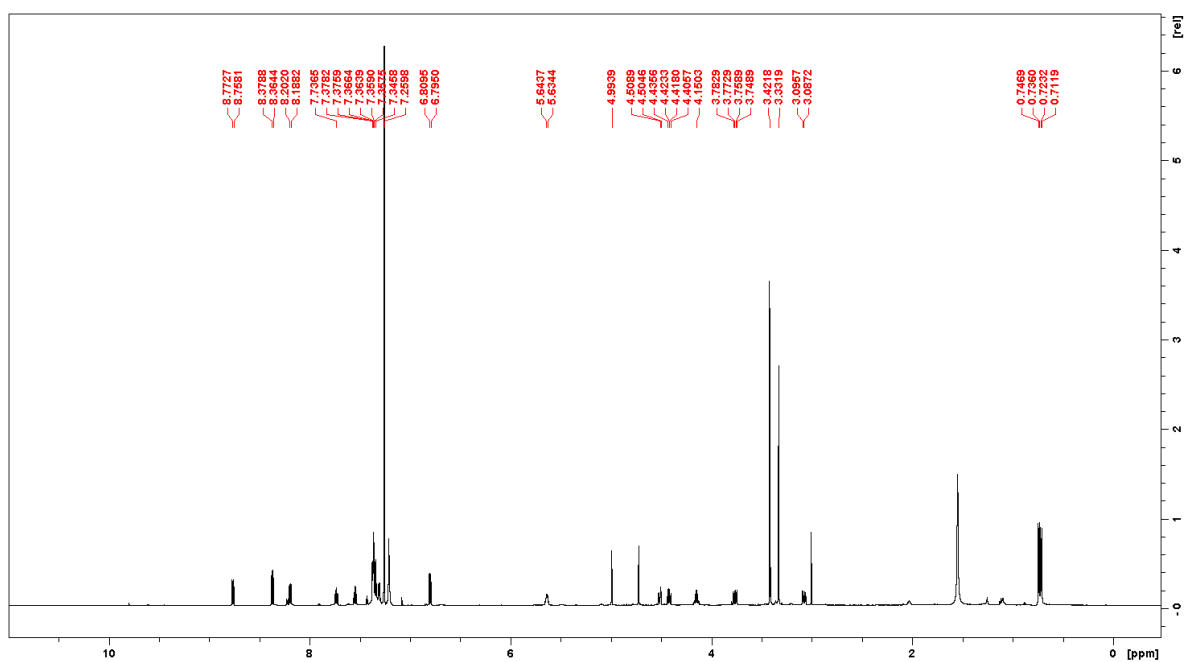


Figure S15 - ^1H -NMR of bis-(R)-MPA-(+)-4-NO₂-propranolol (CDCl₃)

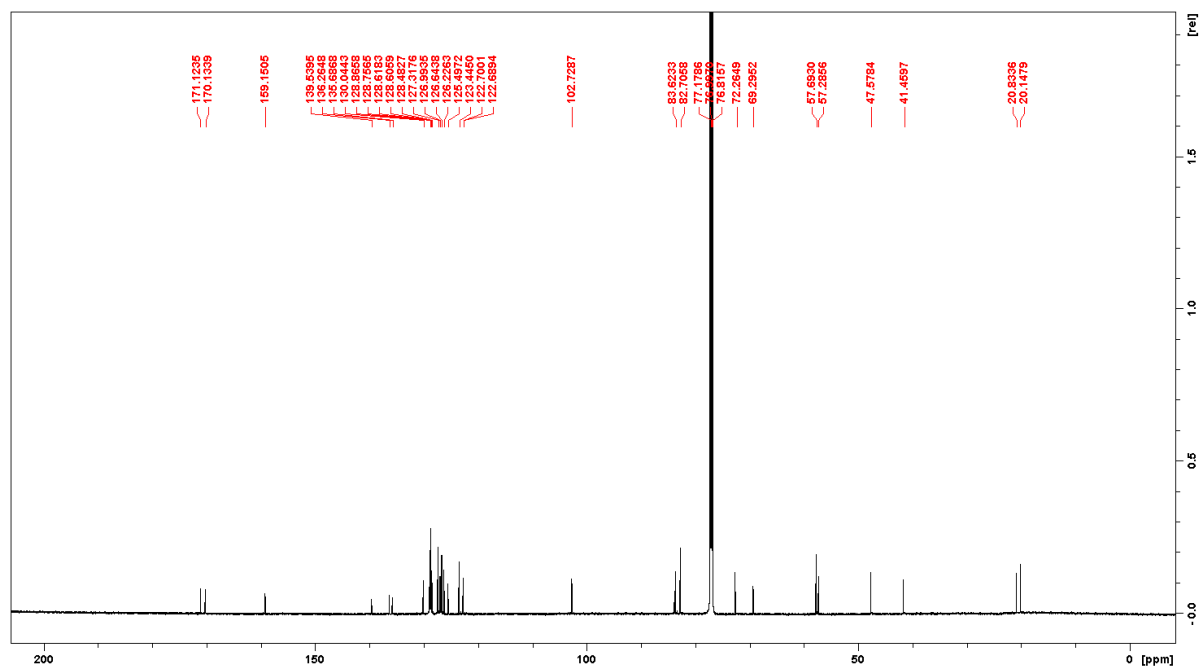


Figure S16 – ^{13}C -NMR of bis-(R)-MPA-(+)-4-NO₂-propranolol (CDCl₃)

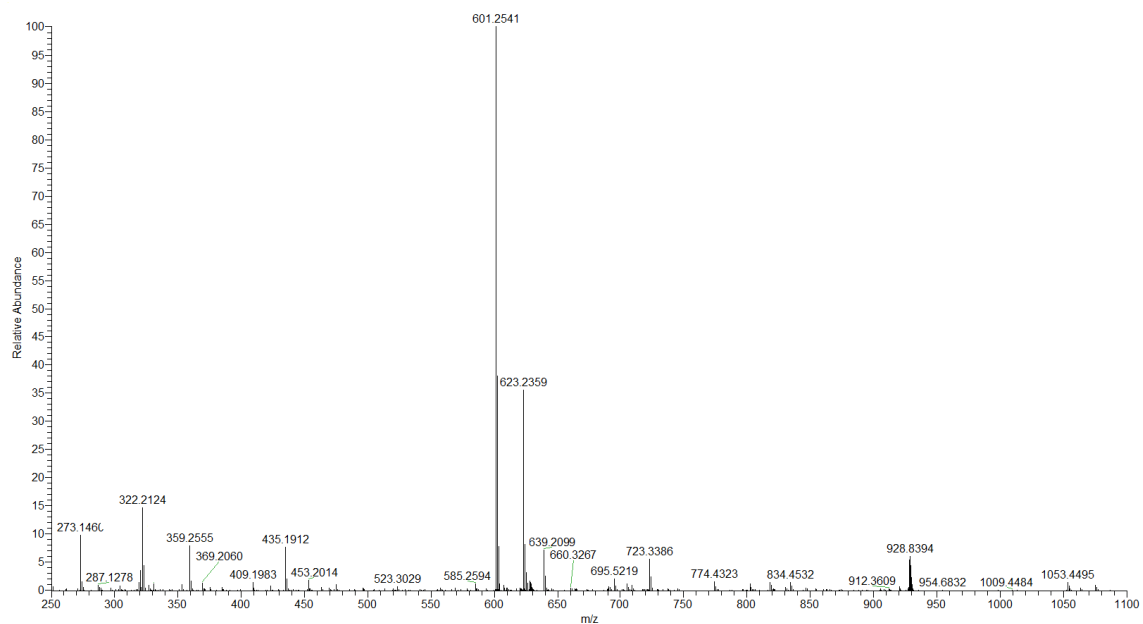


Figure S17 - HRMS spectrum of bis-(R)-MPA-(+)-4-NO₂-propanolol

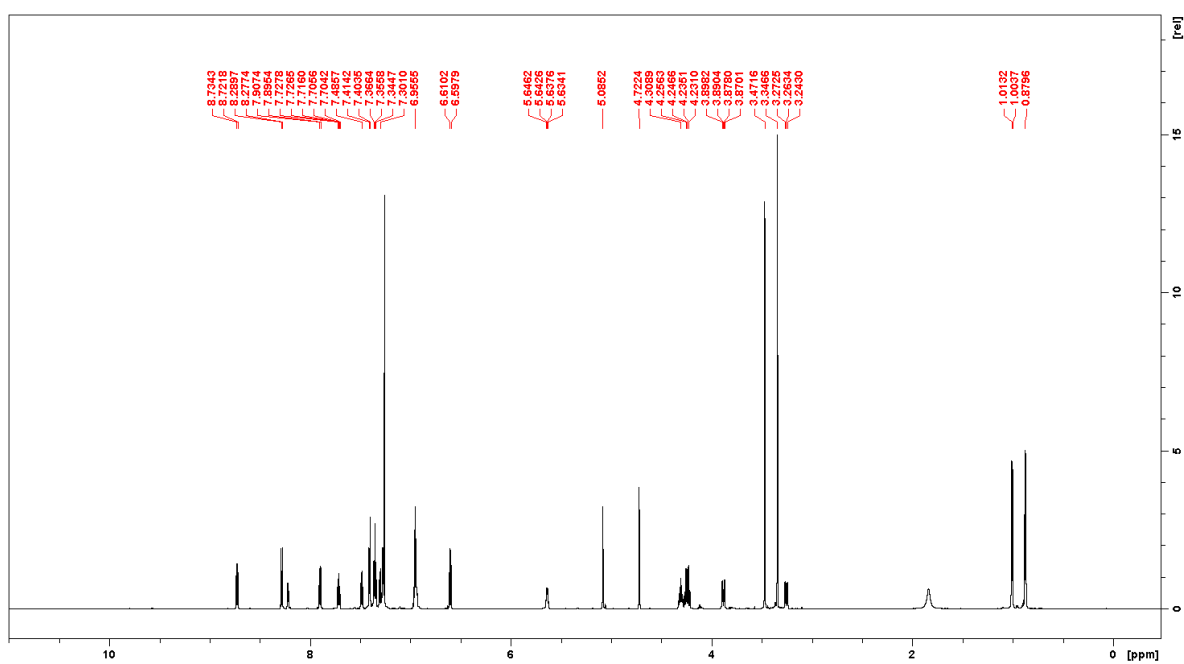


Figure S18 - ¹H-NMR of bis-(S)-MPA-(+)-4-NO₂-propanolol (CDCl₃)

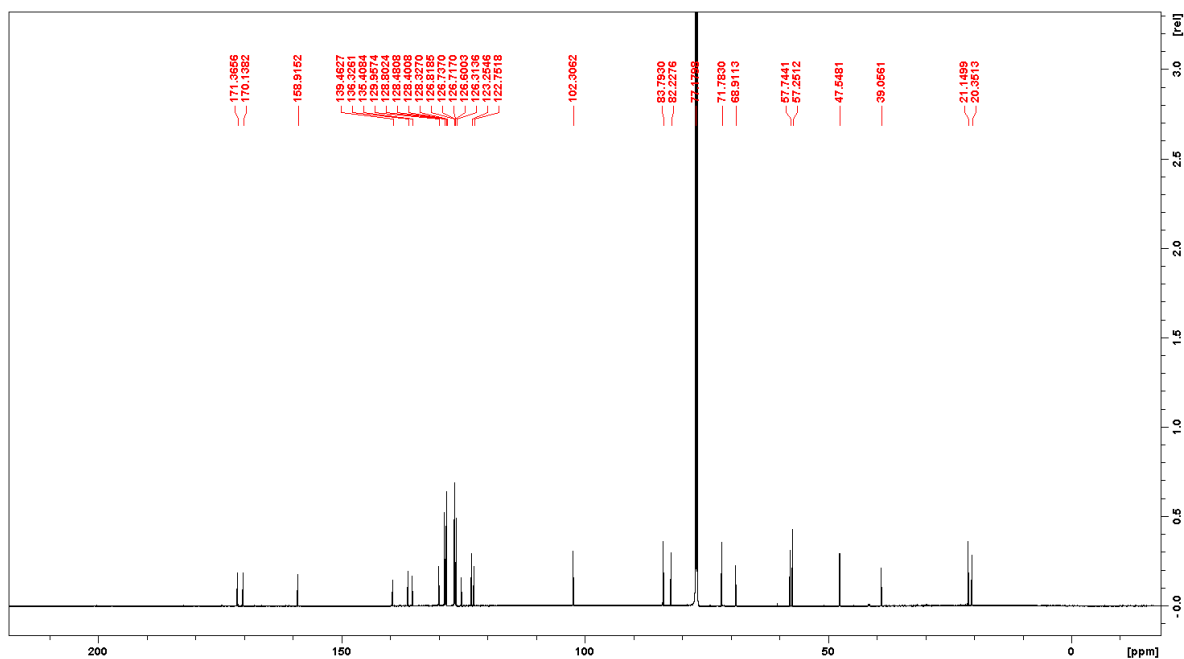


Figure S19 – ^{13}C -NMR of bis-(S)-MPA-(+)-4-NO₂-propranolol (CDCl₃)

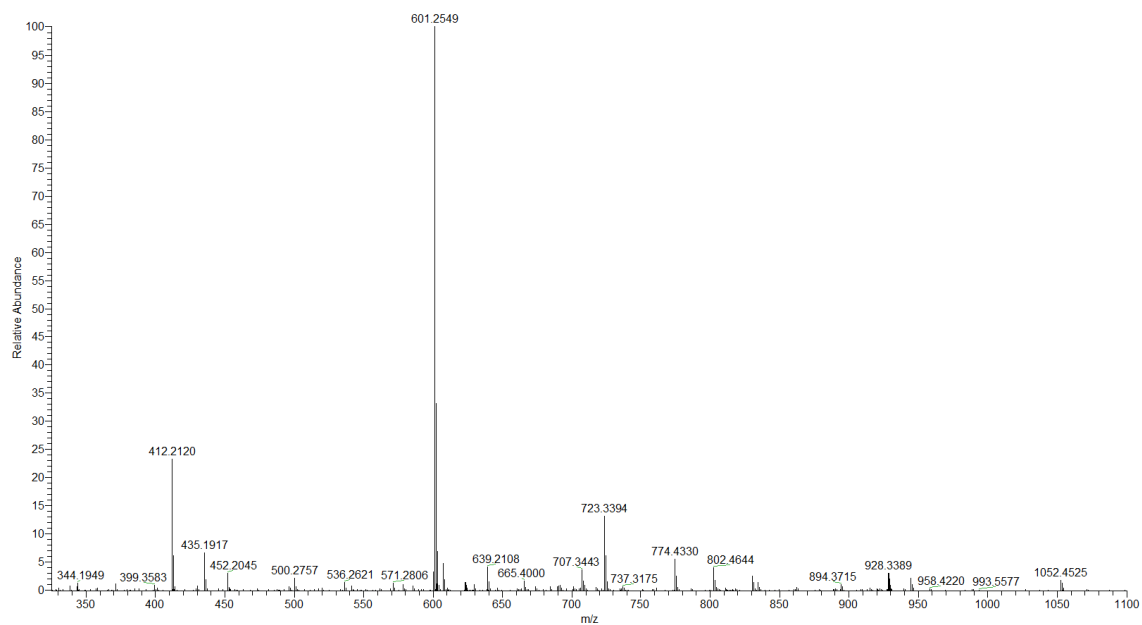


Figure S20 - HRMS spectrum of bis-(S)-MPA-(+)-4-NO₂-propranolol



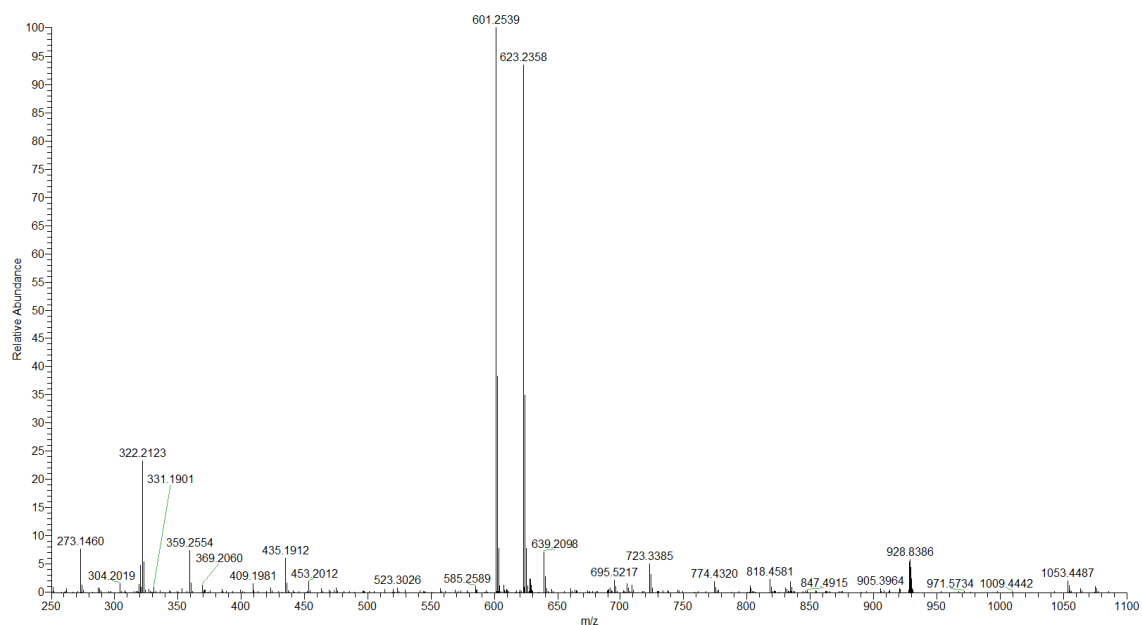


Figure S23 - HRMS spectrum of bis-(R)-MPA-(+)-7-NO₂-propanolol

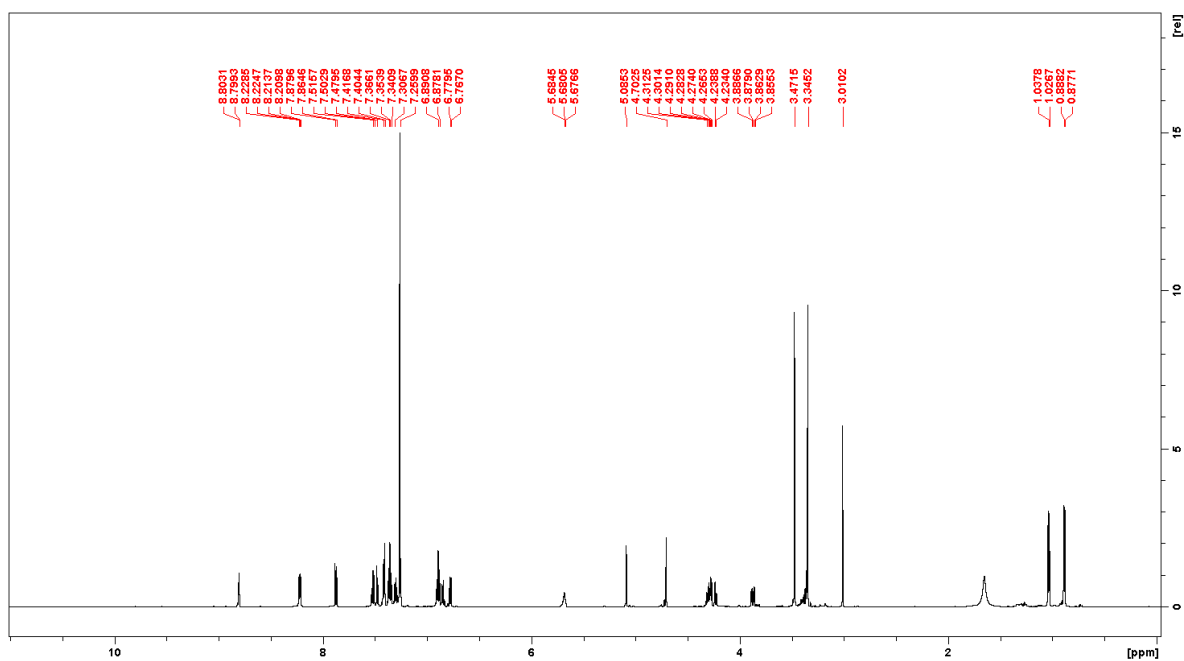


Figure S24 - ¹H-NMR of bis-(S)-MPA-(+)-7-NO₂-propanolol (CDCl₃)

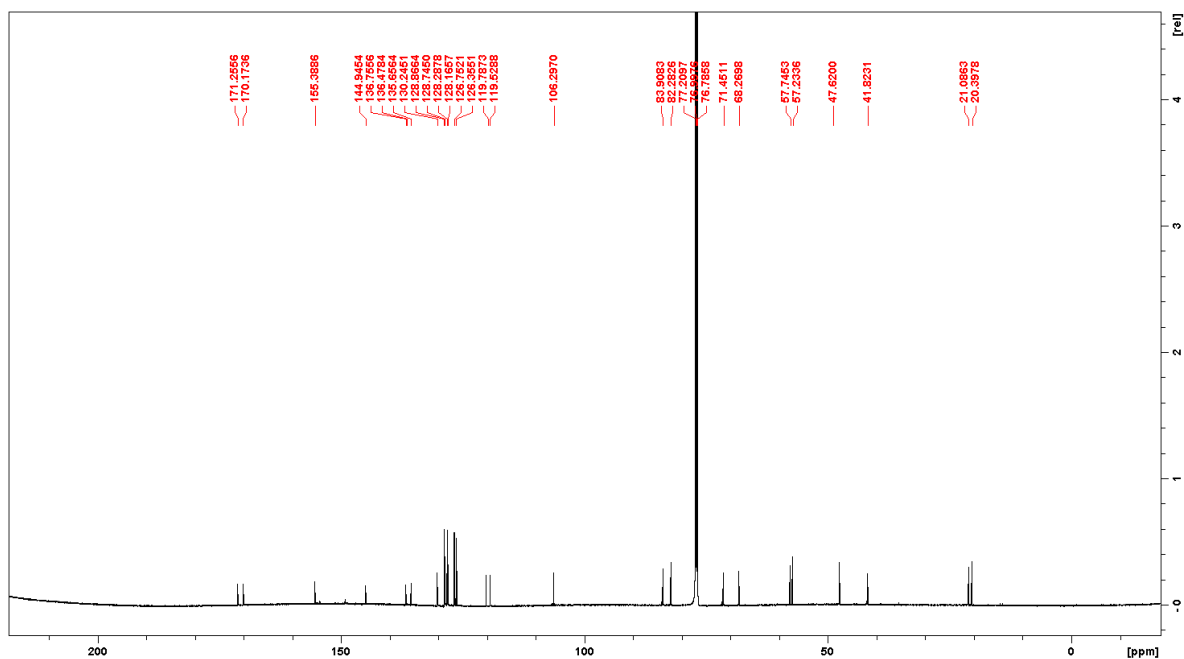


Figure S25 – ^{13}C -NMR of bis-(S)-MPA-(+)-7- NO_2 -propranolol (CDCl_3)

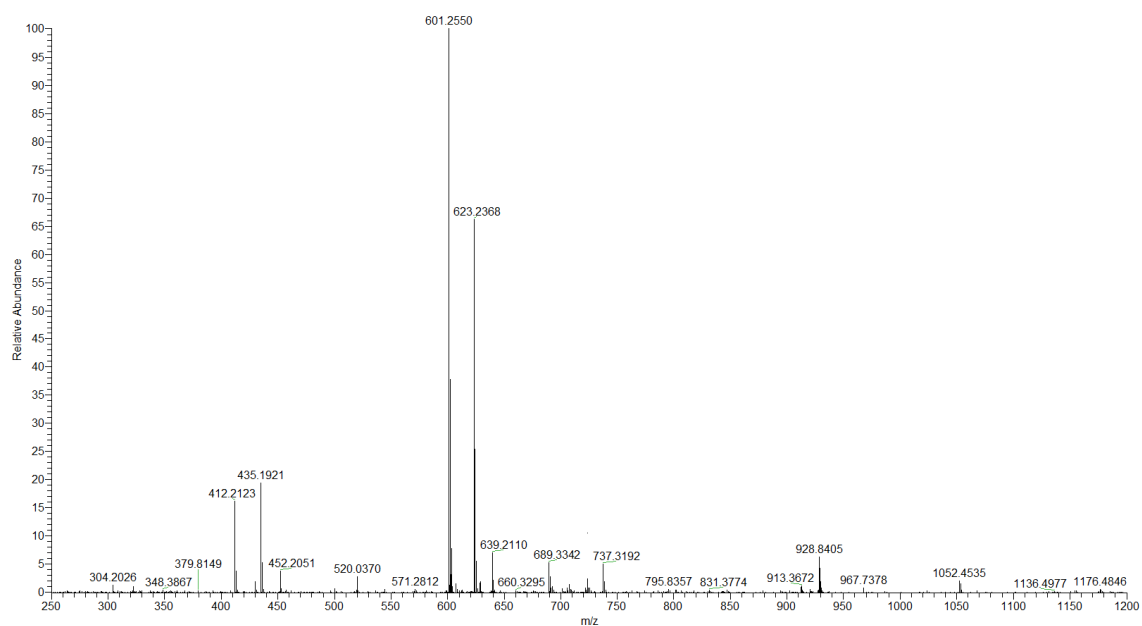


Figure S26 - HRMS spectrum of bis-(S)-MPA-(+)-7- NO_2 -propranolol

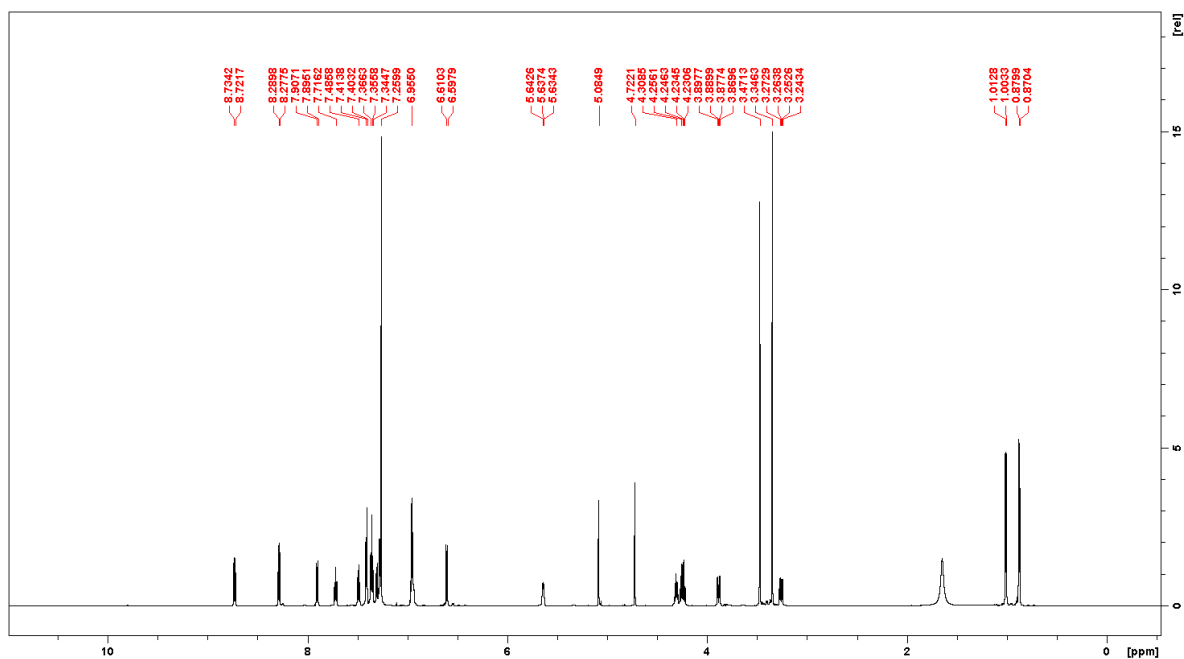


Figure S27 - ^1H -NMR of bis-(R)-MPA-(-)-4- NO_2 -propranolol (CDCl_3)

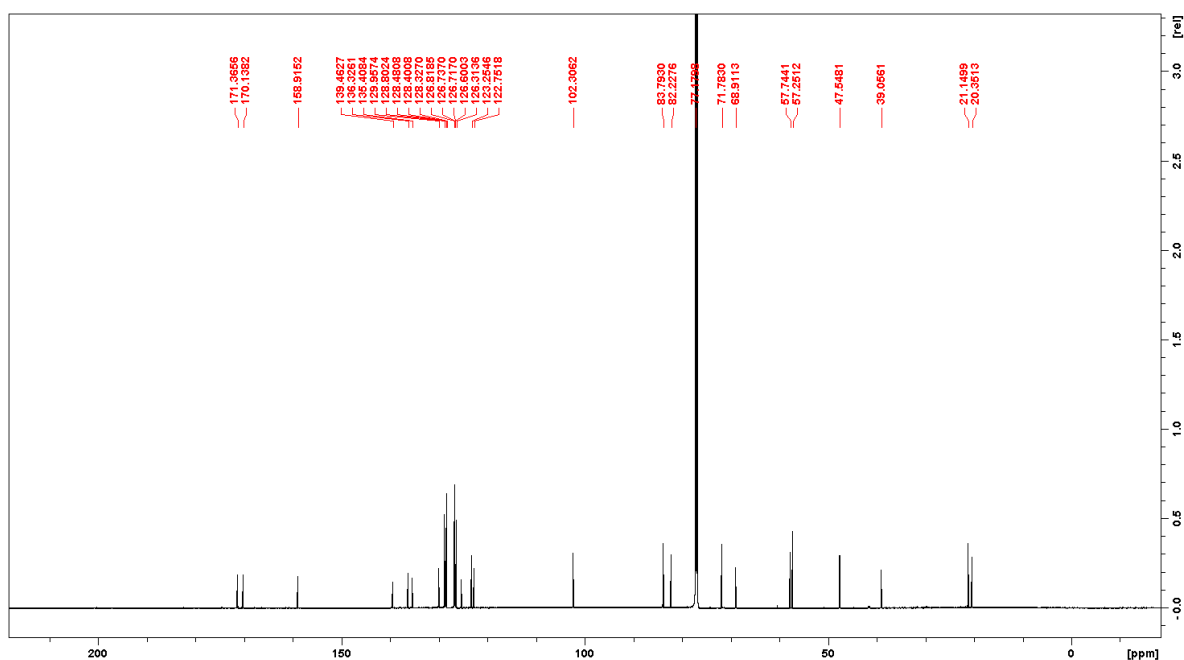


Figure S28 - ^{13}C -NMR of bis-(R)-MPA-(-)-4- NO_2 -propranolol (CDCl_3)

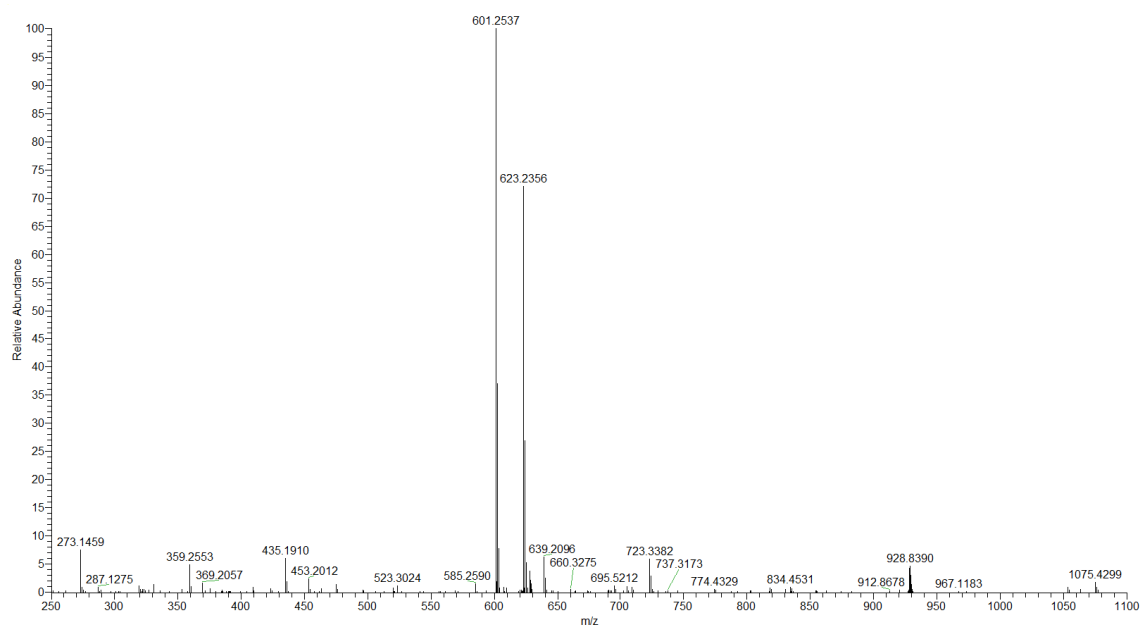


Figure S29 - HRMS spectrum of bis-(R)-MPA-(-)-4-NO₂-propanolol

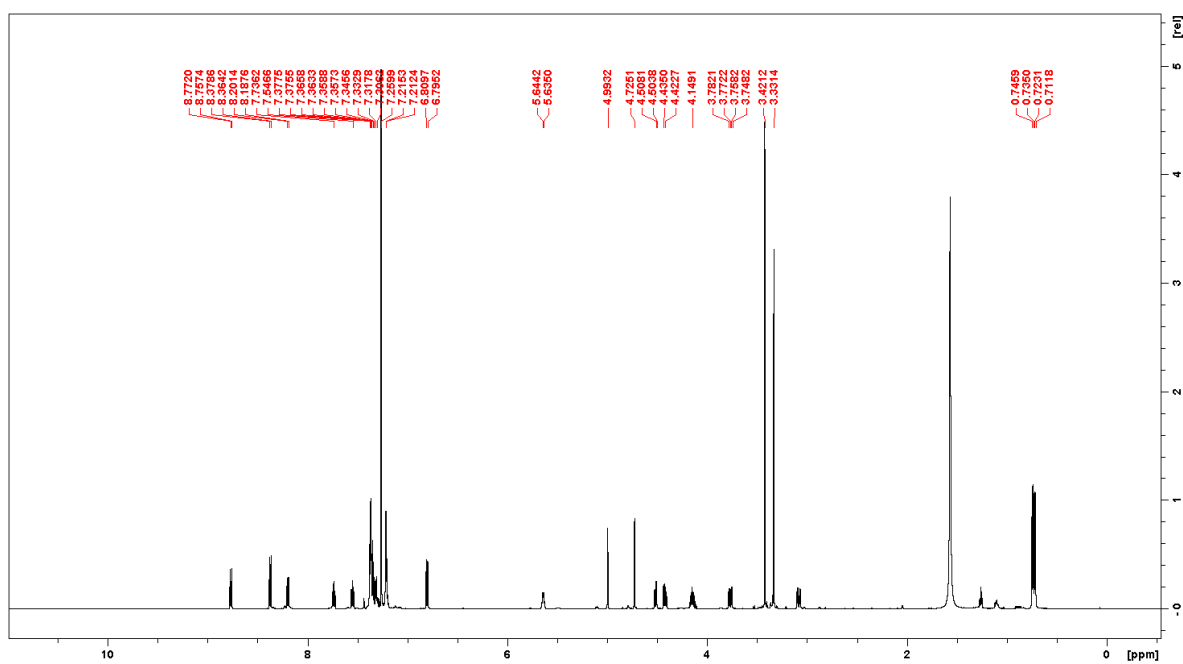


Figure S30 - ¹H-NMR of bis-(S)-MPA-(-)-4-NO₂-propanolol (CDCl₃)

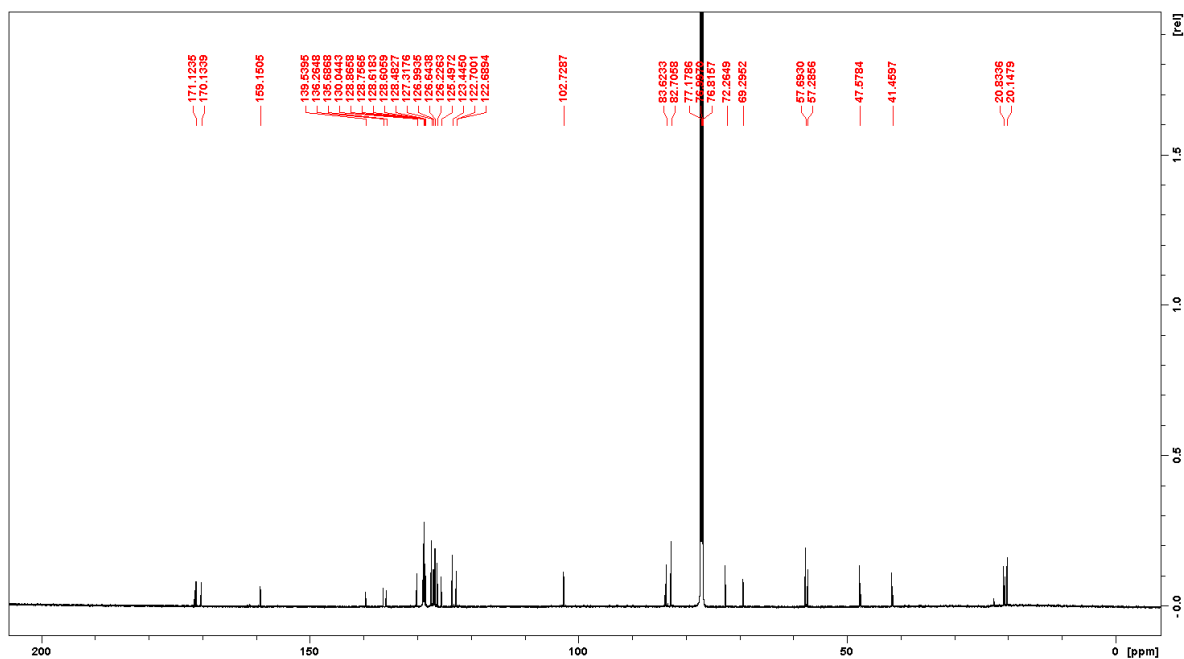


Figure S31 – ^{13}C -NMR of bis-(S)-MPA-(-)-4-NO₂-propranolol (CDCl₃)

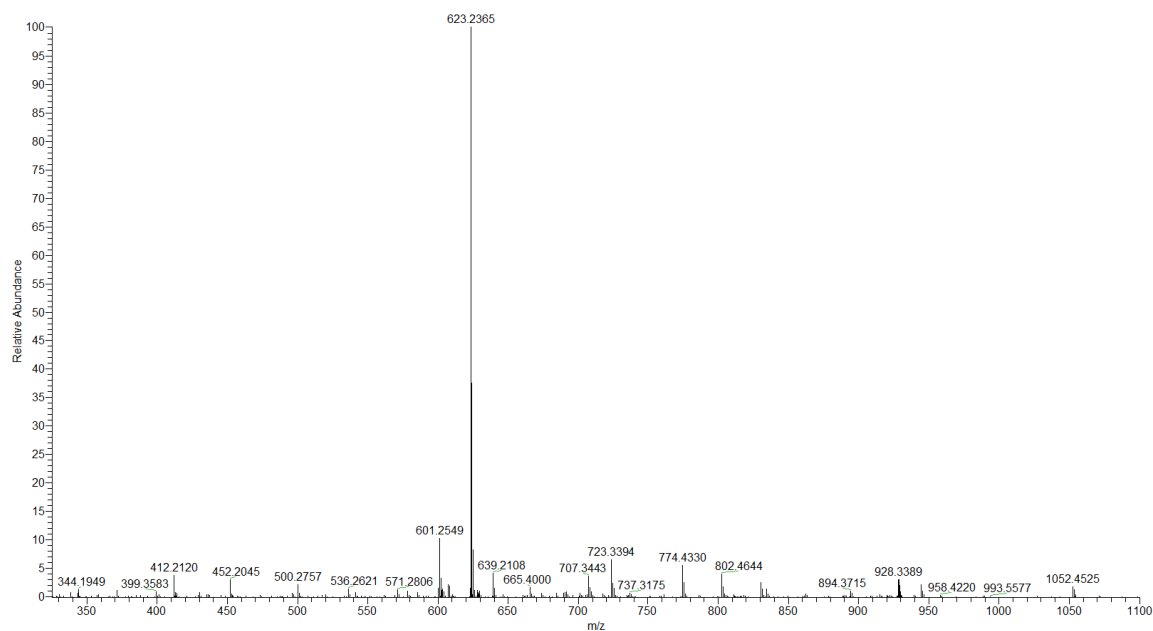


Figure S32 - HRMS spectrum of bis-(S)-MPA-(-)-4-NO₂-propranolol

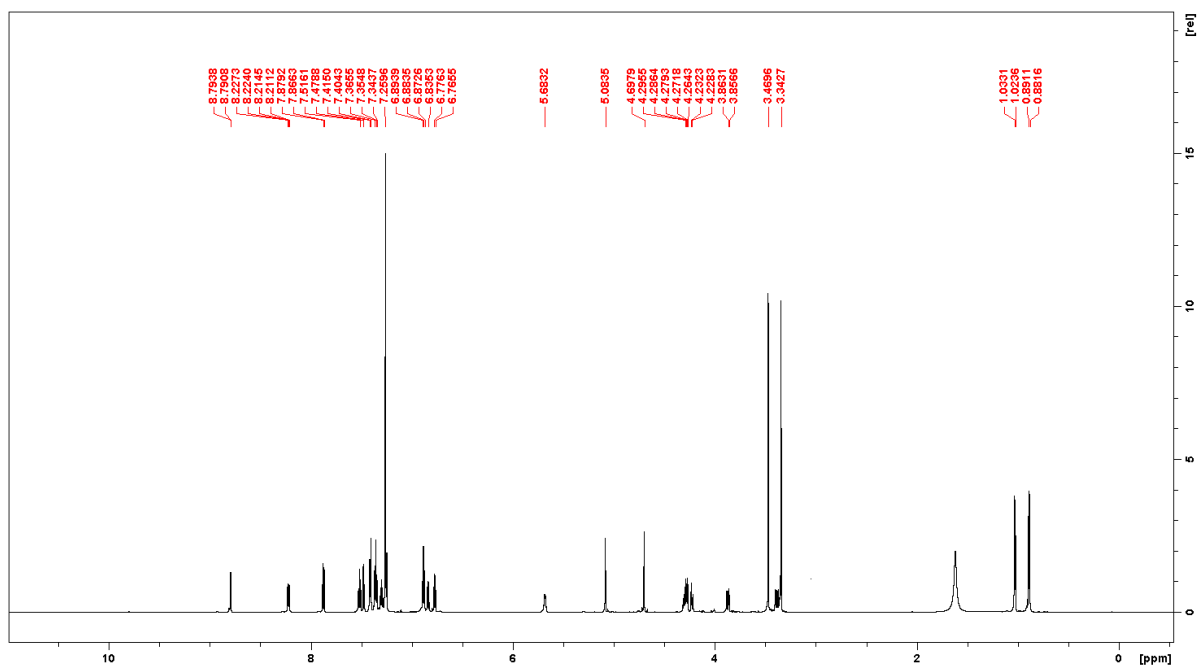


Figure S33 - ¹H-NMR of bis-(R)-MPA-(-)-7-NO₂-propanolol (CDCl₃)

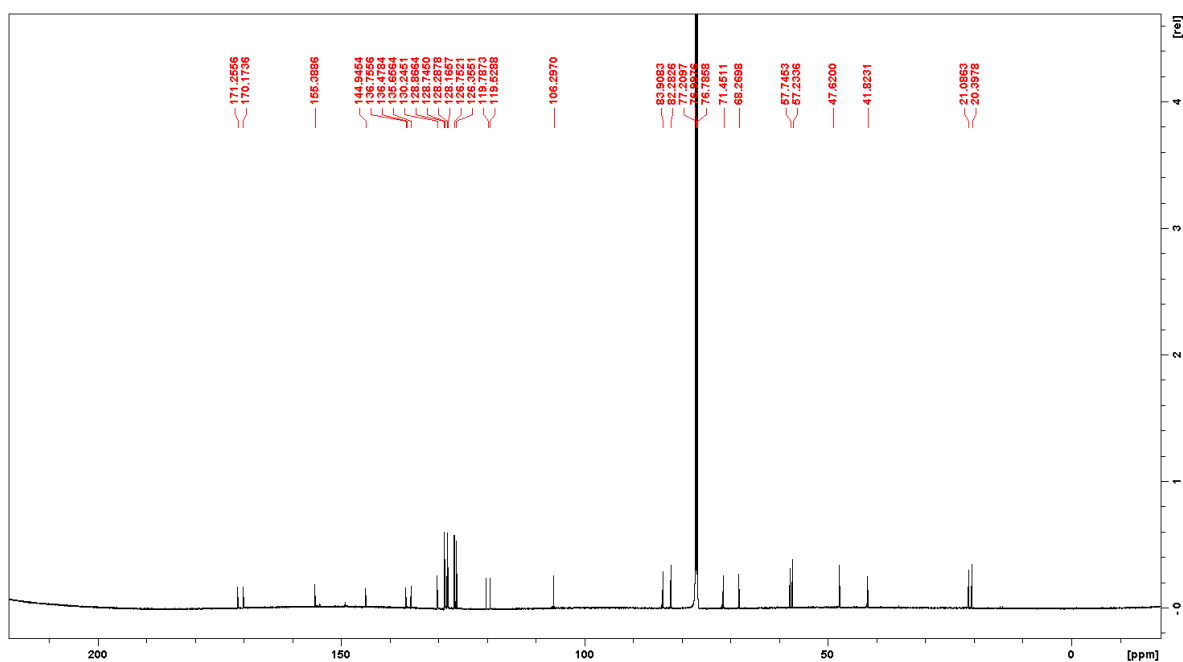


Figure S34 - ¹³C-NMR of bis-(R)-MPA-(-)-7-NO₂-propanolol (CDCl₃)

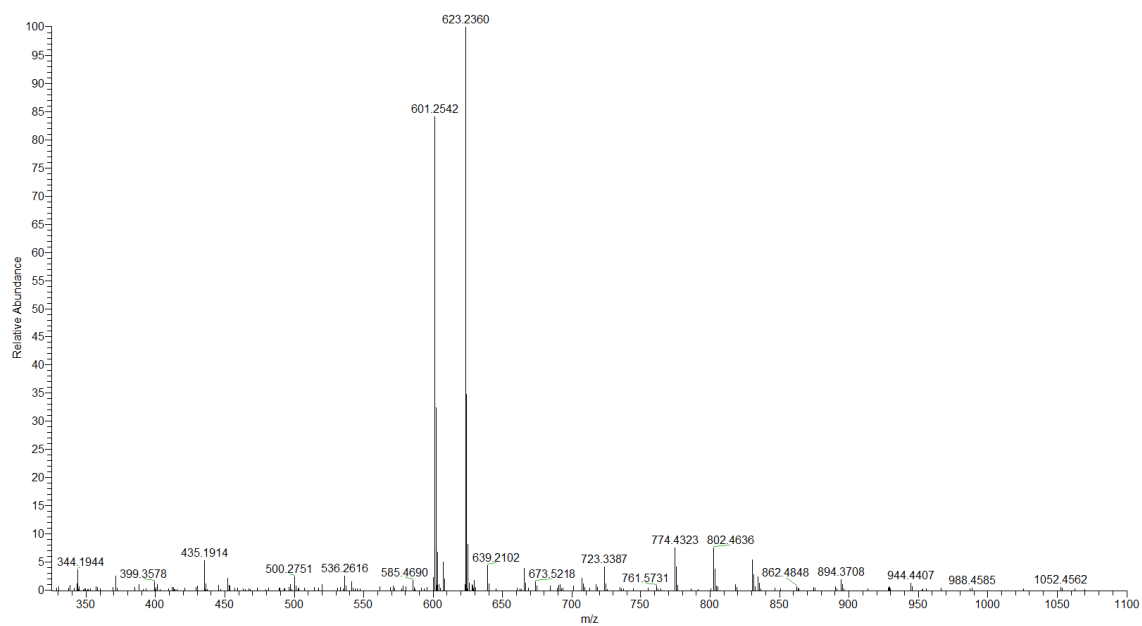


Figure S35 - HRMS spectrum of bis-(R)-MPA-(-)-7-NO₂-propranolol

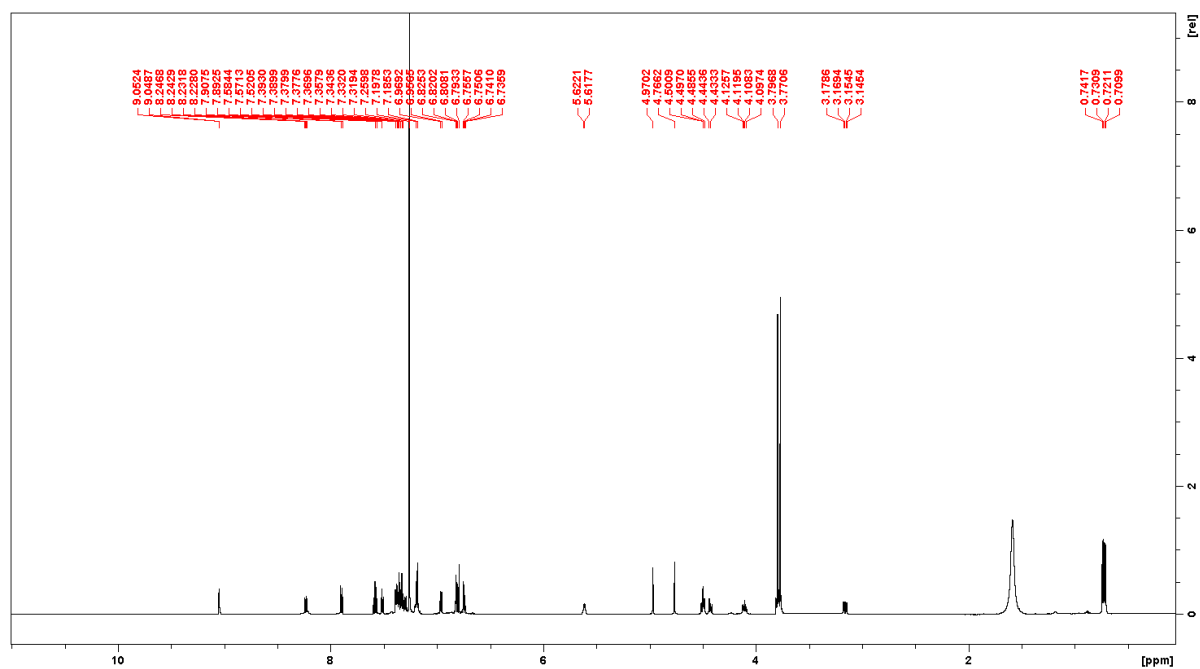


Figure S36 - ¹H-NMR of bis-(S)-MPA-(-)-7-NO₂-propranolol (CDCl₃)

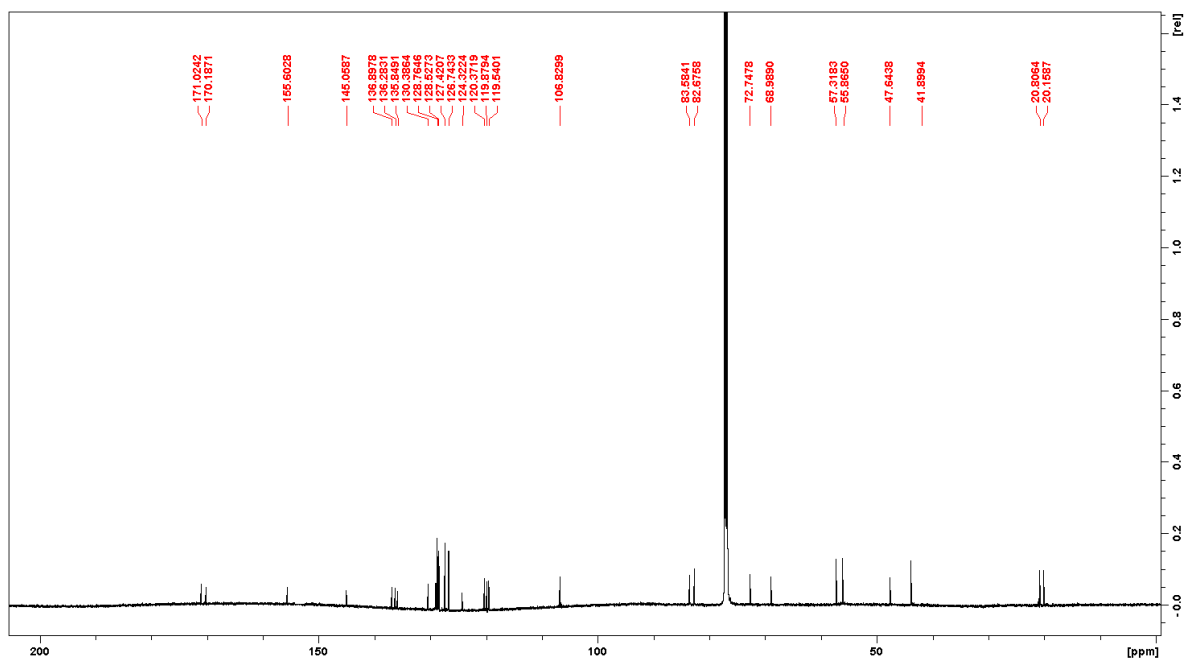


Figure S37 – ^{13}C -NMR of bis-(S)-MPA-(-)-7- NO_2 -propranolol (CDCl_3)

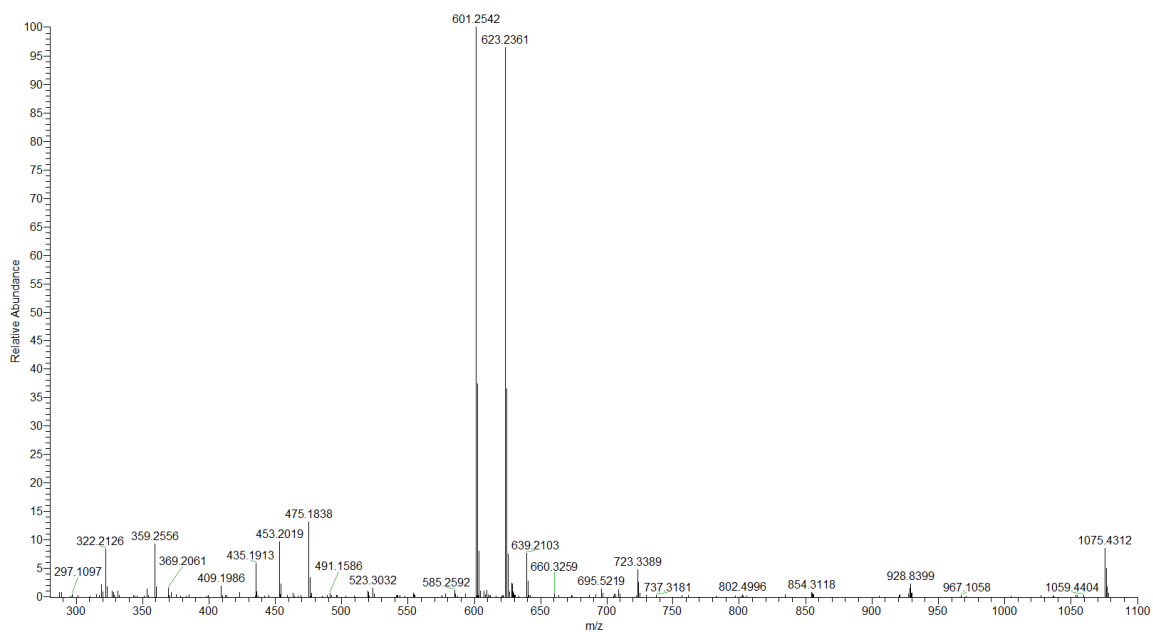


Figure S38 - HRMS spectrum of bis-(S)-MPA-(-)-7- NO_2 -propranolol