symmetry

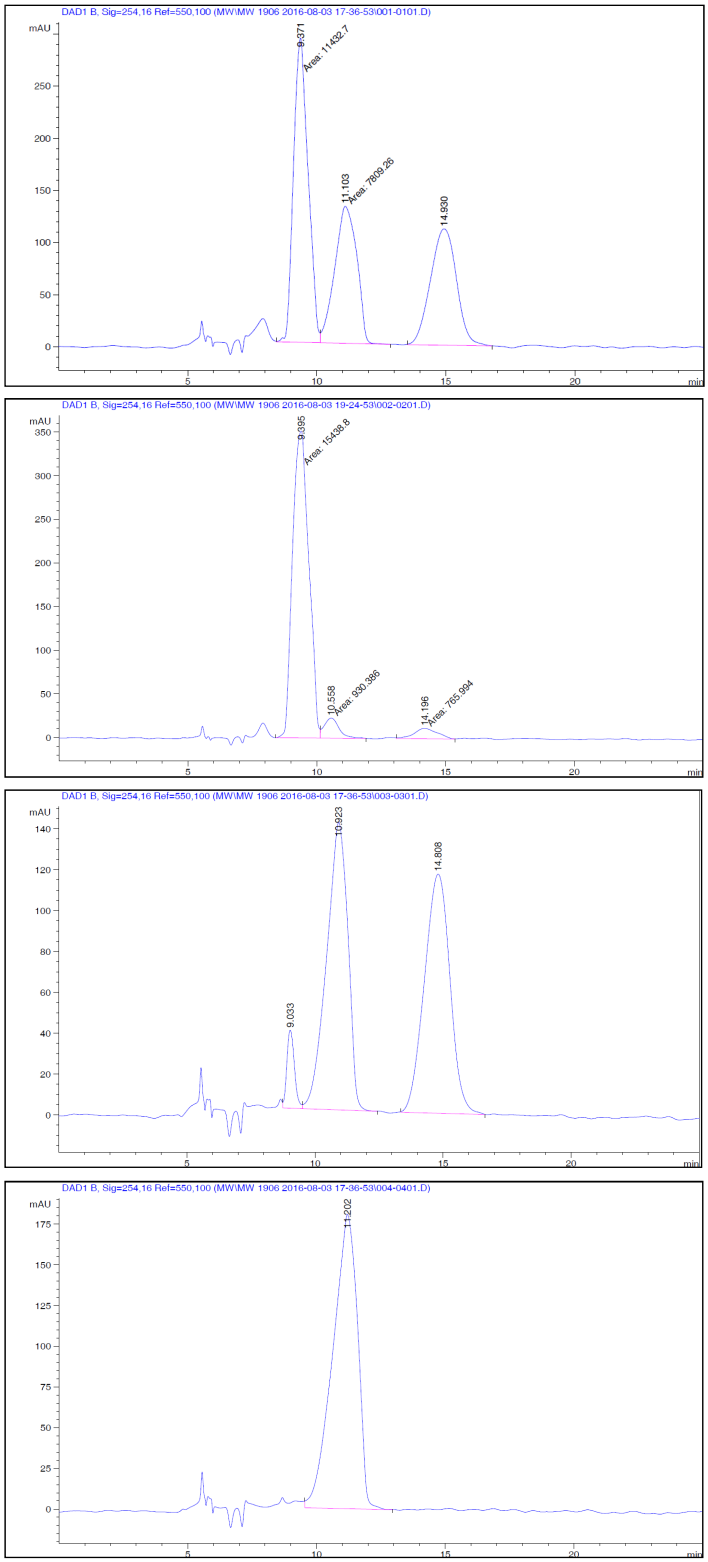
MDPI

Supplementary materials

Racemic and meso crystal structures of an axial-chiral spirobi-(dinaphthoazepin)ium salt: Emergence of an S_4-symmetric molecule

Philipp Honegger[1,*], Natalie Gajic[2], Alexander Prado-Roller[2] and Michael Widhalm[3]

[1] Institute of Computational Biological Chemistry, University of Vienna, Währinger Straße 17, 1090 Vienna, Austria
[2] Institute of Inorganic Chemistry, University of Vienna, Währinger Straße 42, 1090 Vienna, Austria
[3] Institute of Chemical Catalysis, University of Vienna, Währinger Straße 38, 1090 Vienna, Austria
* Correspondence: philipp.honegger@univie.ac.at



crude mixture

(*R*)(*S*)-**2** *meso*

(*R*)(*R*)/(*S*)(*S*)-**2** *racemate*

(*R*)(*R*)-**2** *enantiomer*§

Figure S1: HPLC Separation of Stereoisomers of **2***

* Chiralcel-ODH, MeOH (12.5%), EtOH (12.5%), Heptane (74.7%), TFA (0.3%), 0.5 mLmin^{-1}, 38 °C; samples: 0.3-0.4 mg dissolved in 0.3 mL DCM, 1 mL EtOH; 3-5 µL injected, integration at 254 nm.

§ authentical sample prepared from (*R*)-2,2'-bisbromomethyl-1,1'-binaphthyl.

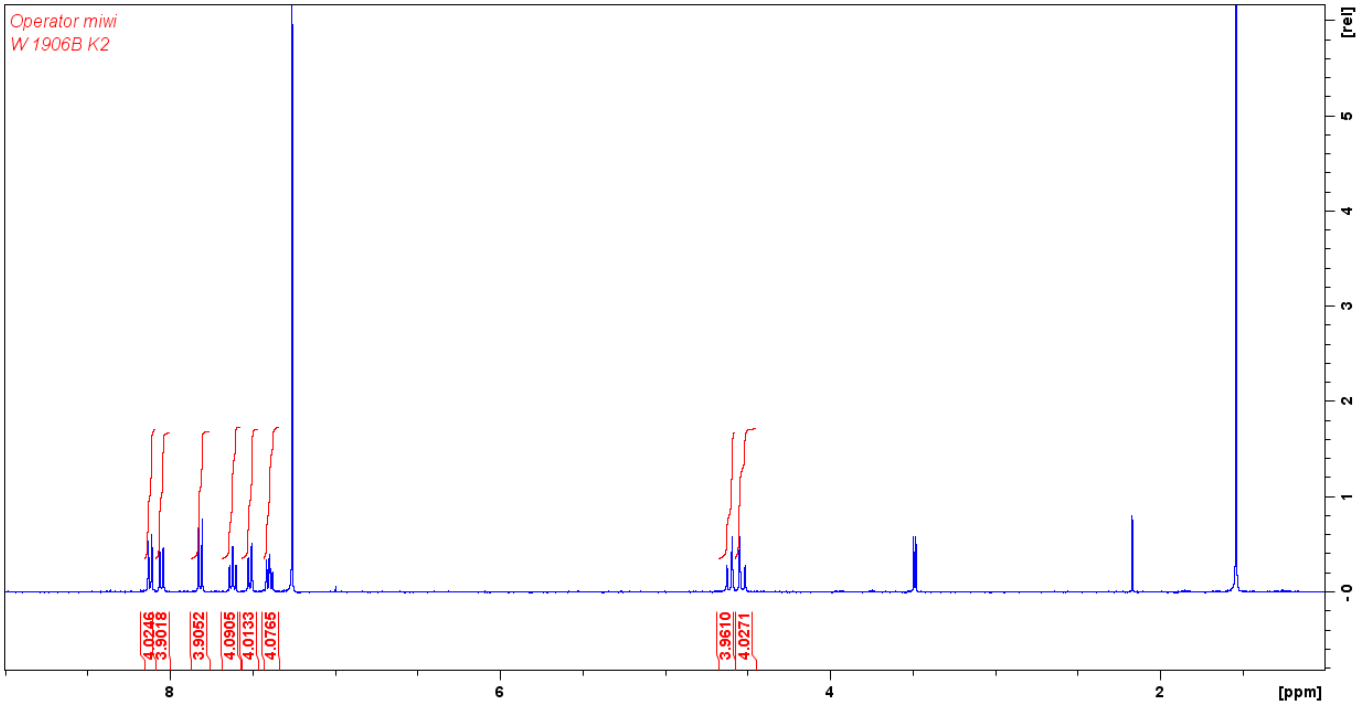


Figure S2: $(R,S)_{ax}$-**2** (^{1}H NMR in $CDCl_3$)

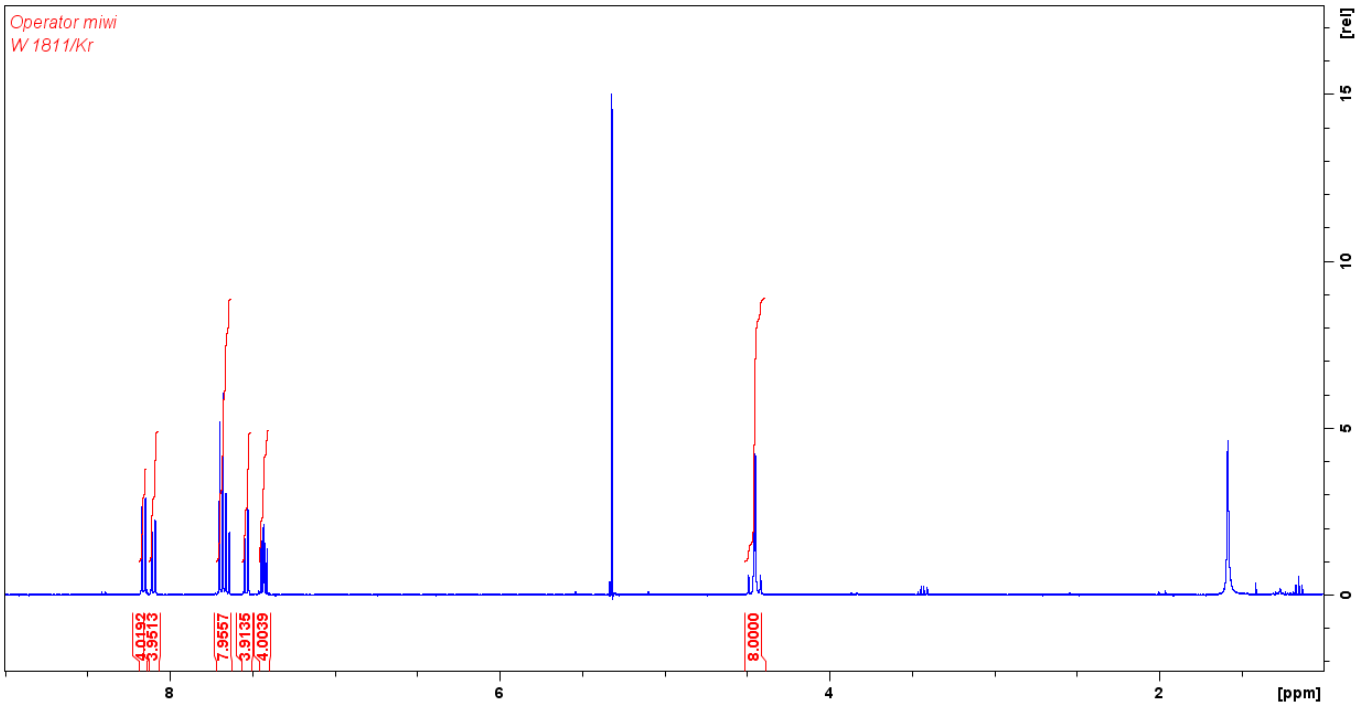


Figure S3: $(R,S)_{ax}$-**2** (^{1}H NMR in CD_2Cl_2)

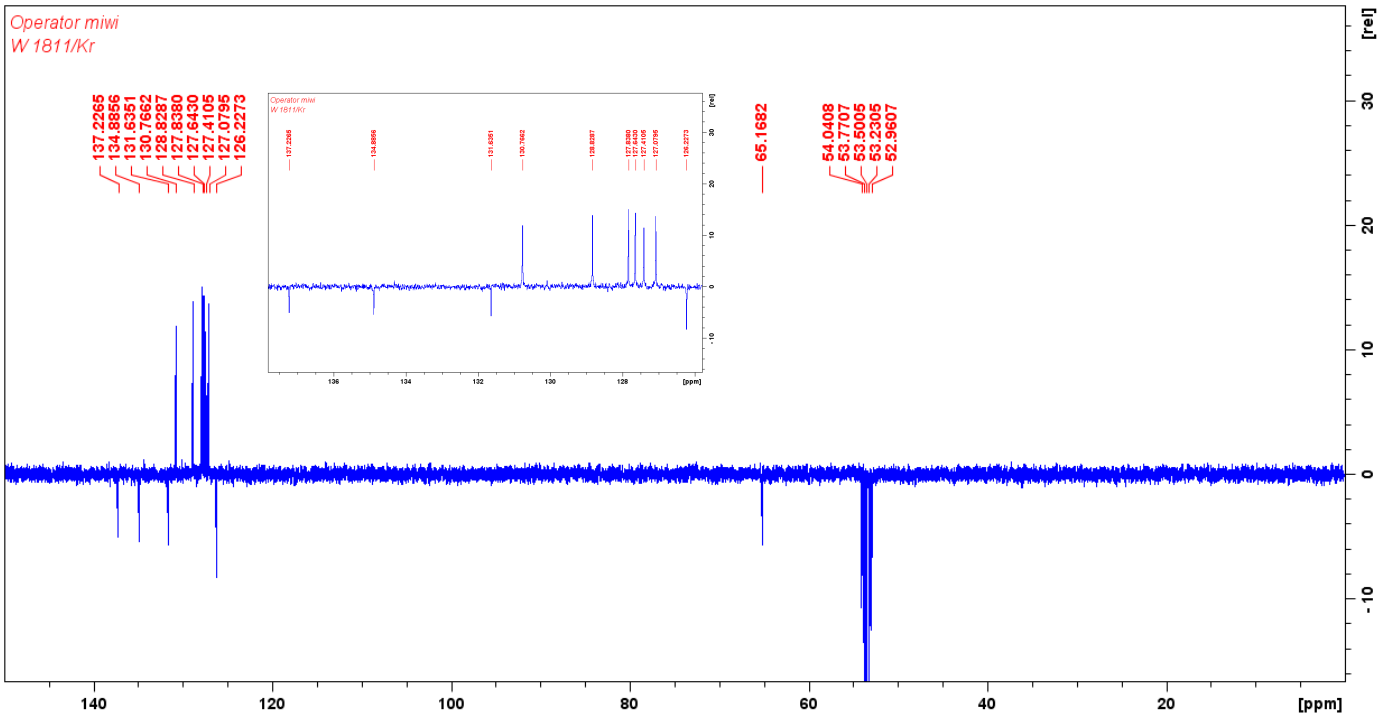


Figure S4: (*R*,*S*)$_{ax}$-**2** (^{13}C NMR in CD_2Cl_2)

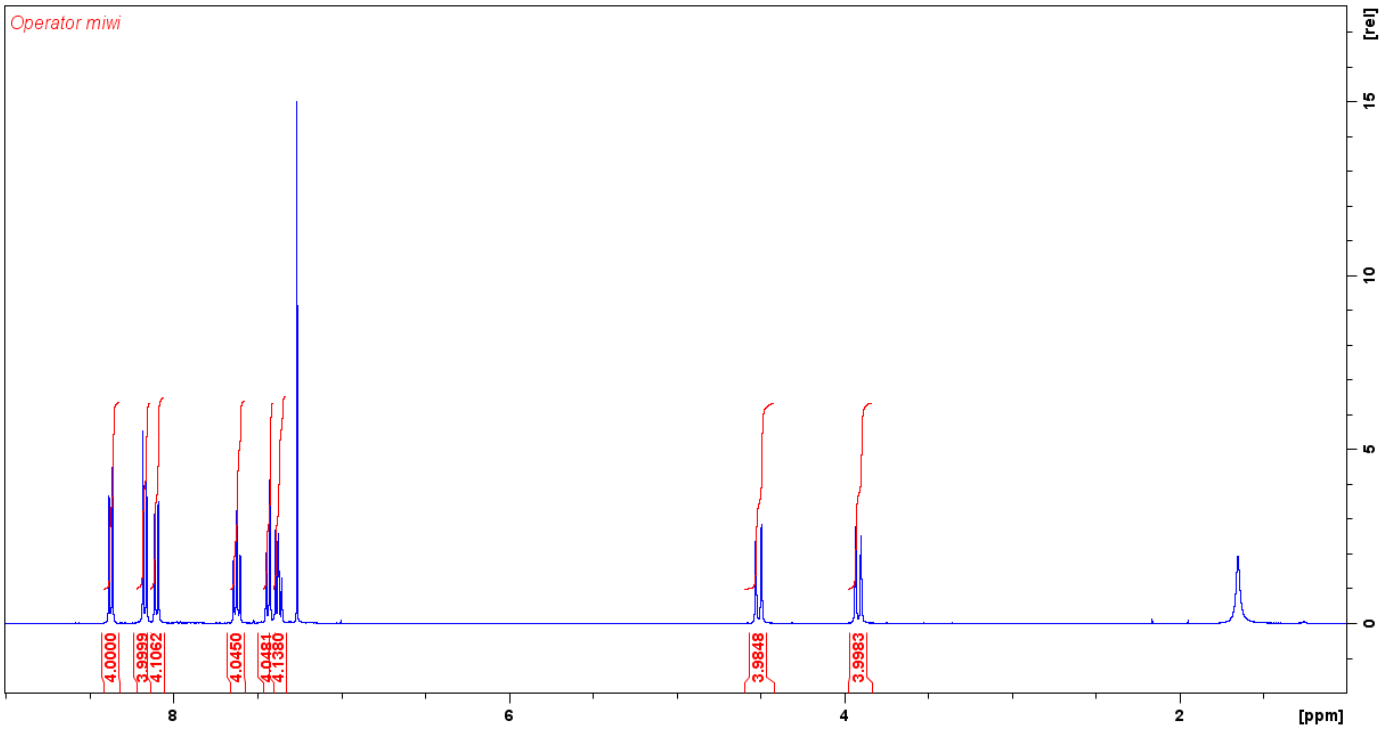


Figure S5: (*R*,*R*)$_{ax}$/(*S*,*S*)$_{ax}$-**2** (^{1}H NMR in $CDCl_3$)

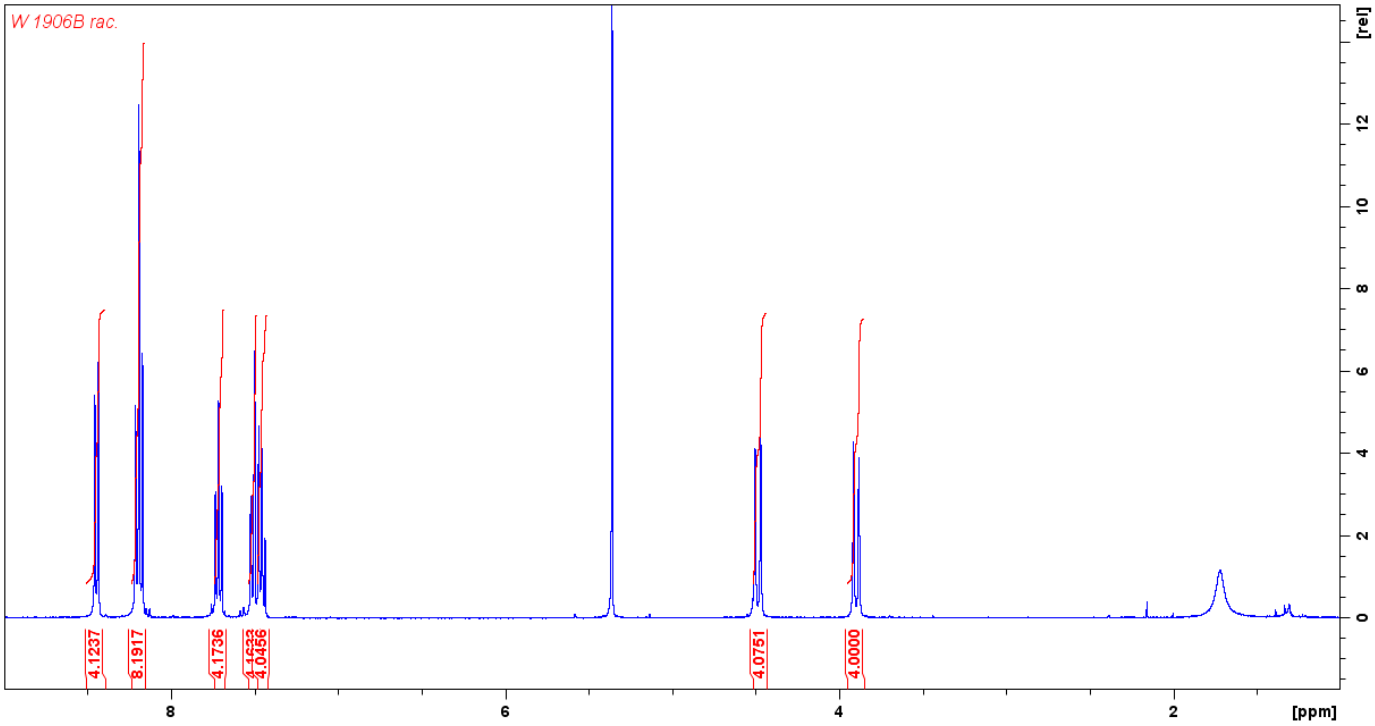


Figure S6: $(R,R)_{ax}/(S,S)_{ax}$-**2** (^{1}H NMR in CD_2Cl_2)

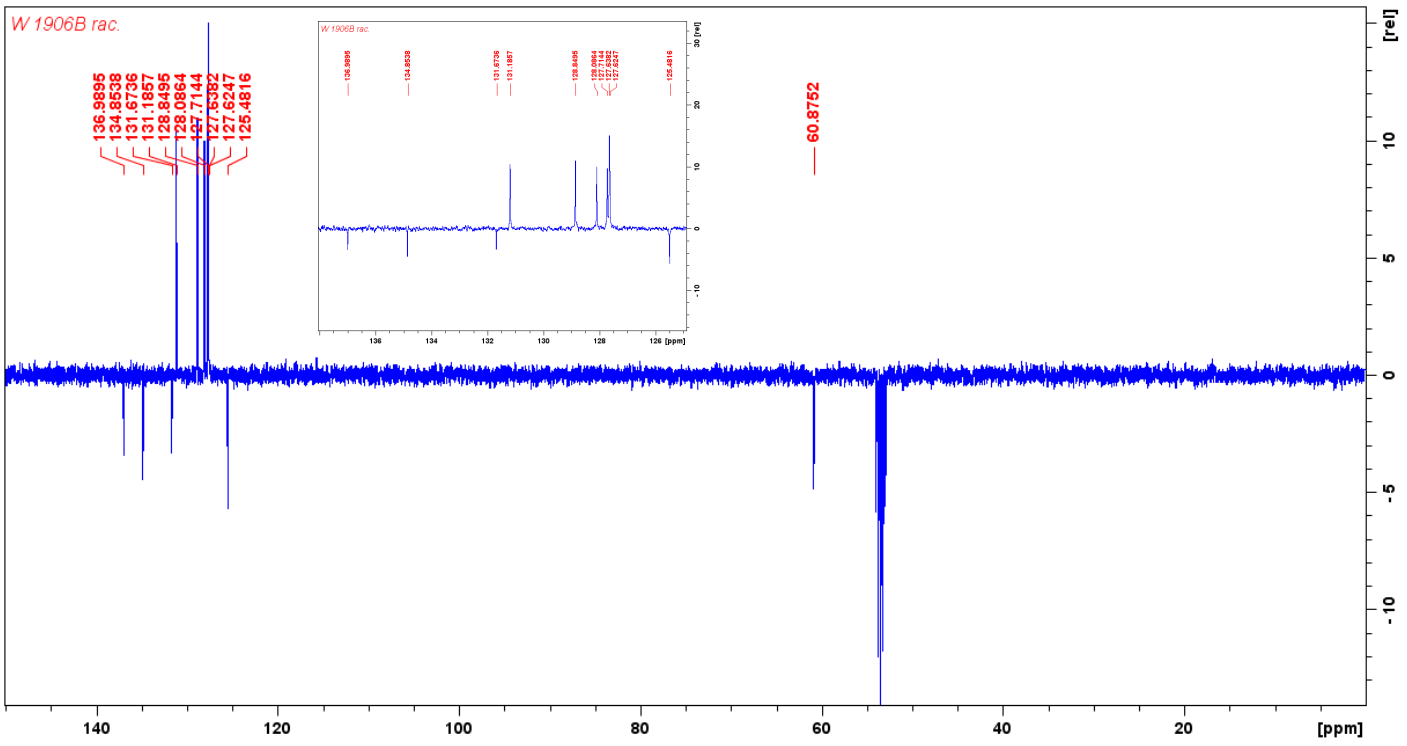


Figure S7: $(R,R)_{ax}/(S,S)_{ax}$-**2** (^{13}C NMR in CD_2Cl_2)

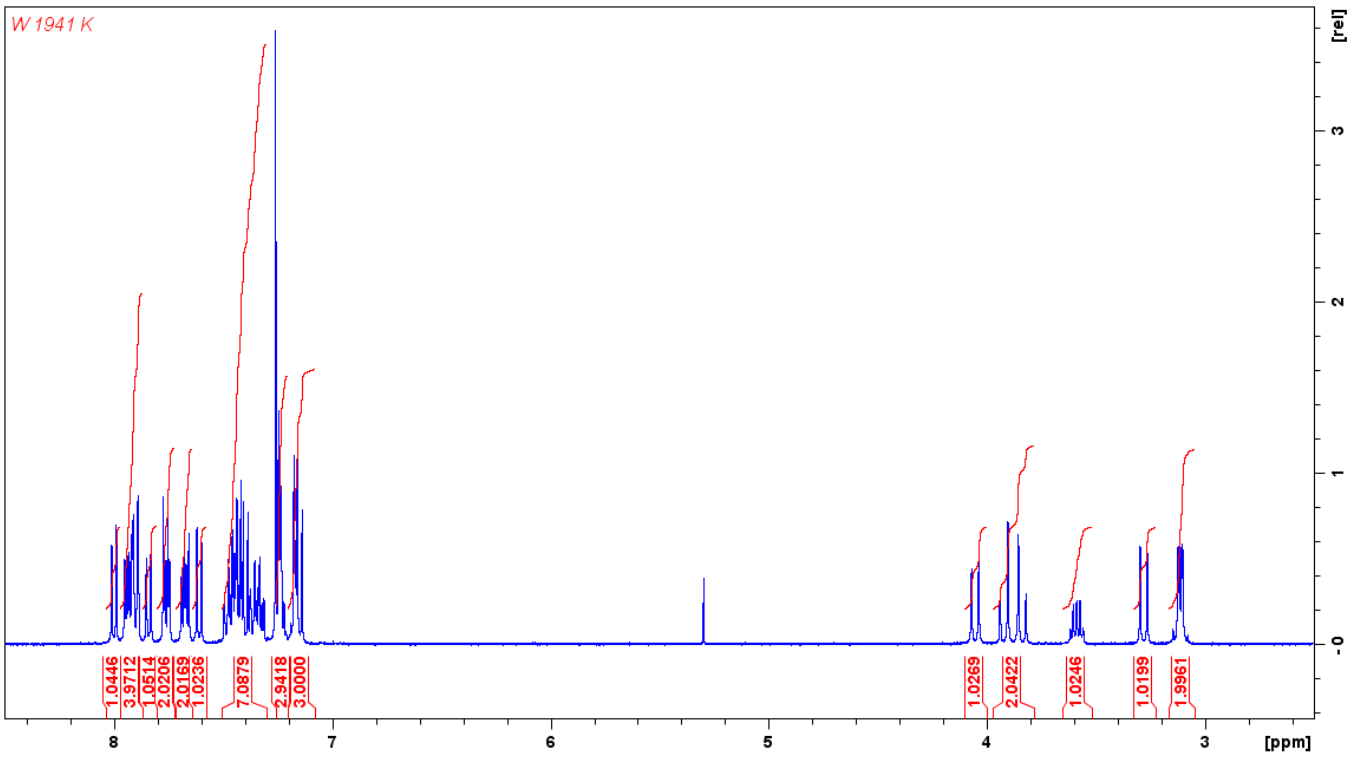


Figure S8: (*R*,*R*)$_{ax}$/(*S*,*S*)$_{ax}$-**4** (^{1}H NMR in $CDCl_3$)

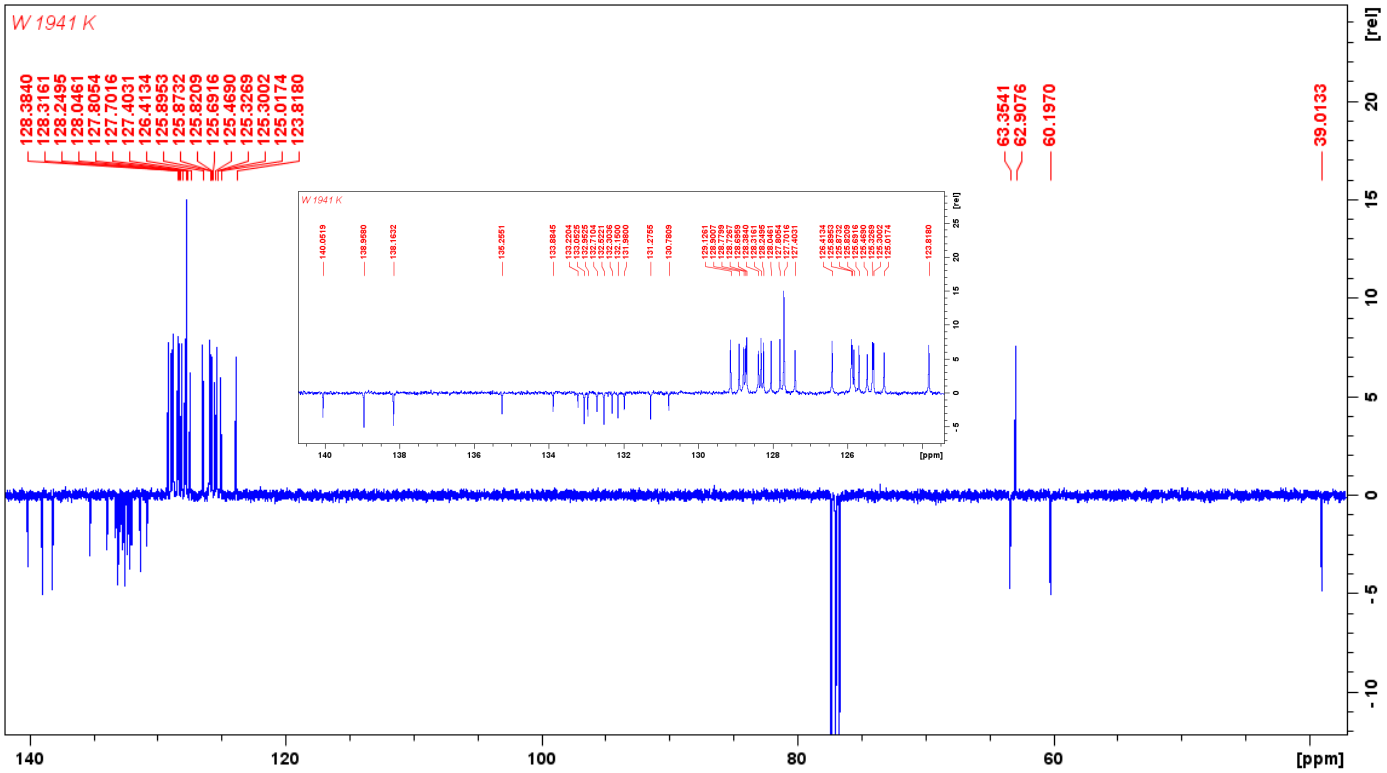


Figure S9: (*R*,*R*)$_{ax}$/(*S*,*S*)$_{ax}$-**4** (^{13}C NMR in $CDCl_3$)

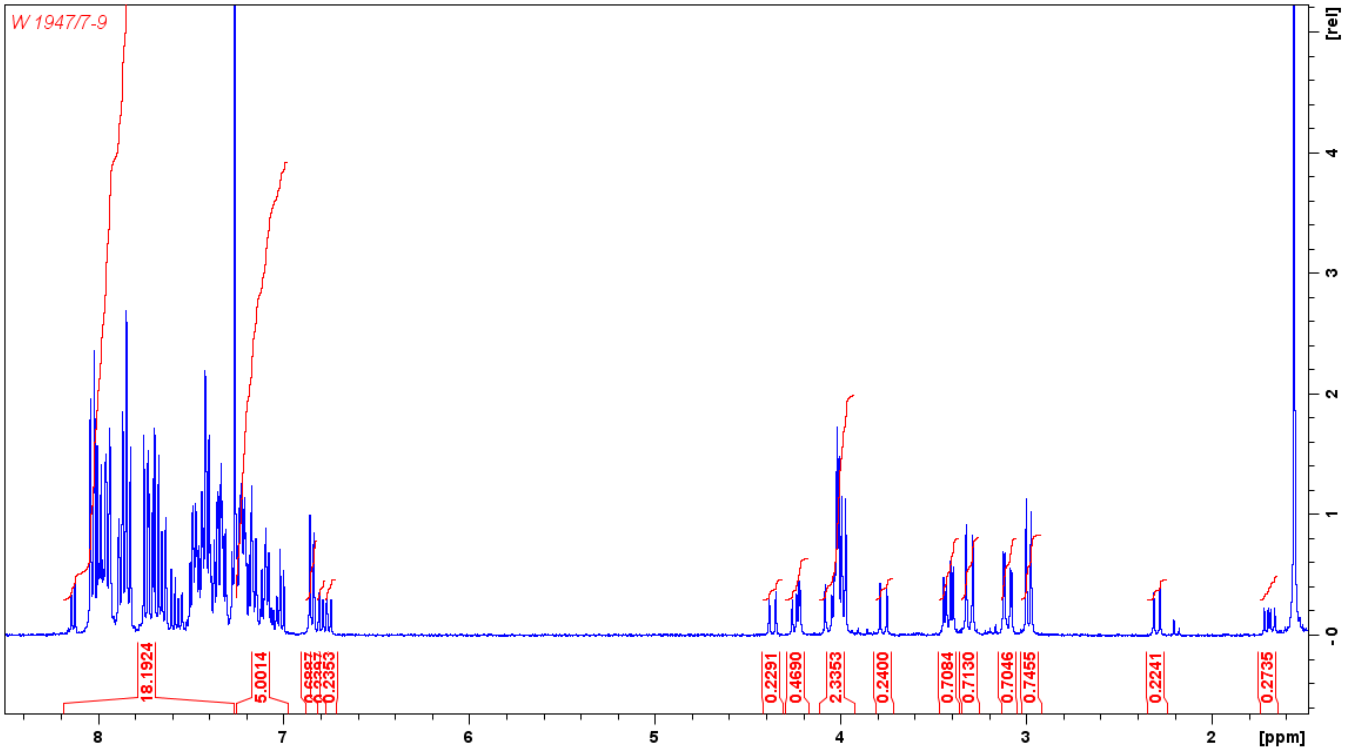


Figure S10: mixture of **5** and **6** (70:30) (^{1}H NMR in $CDCl_3$)

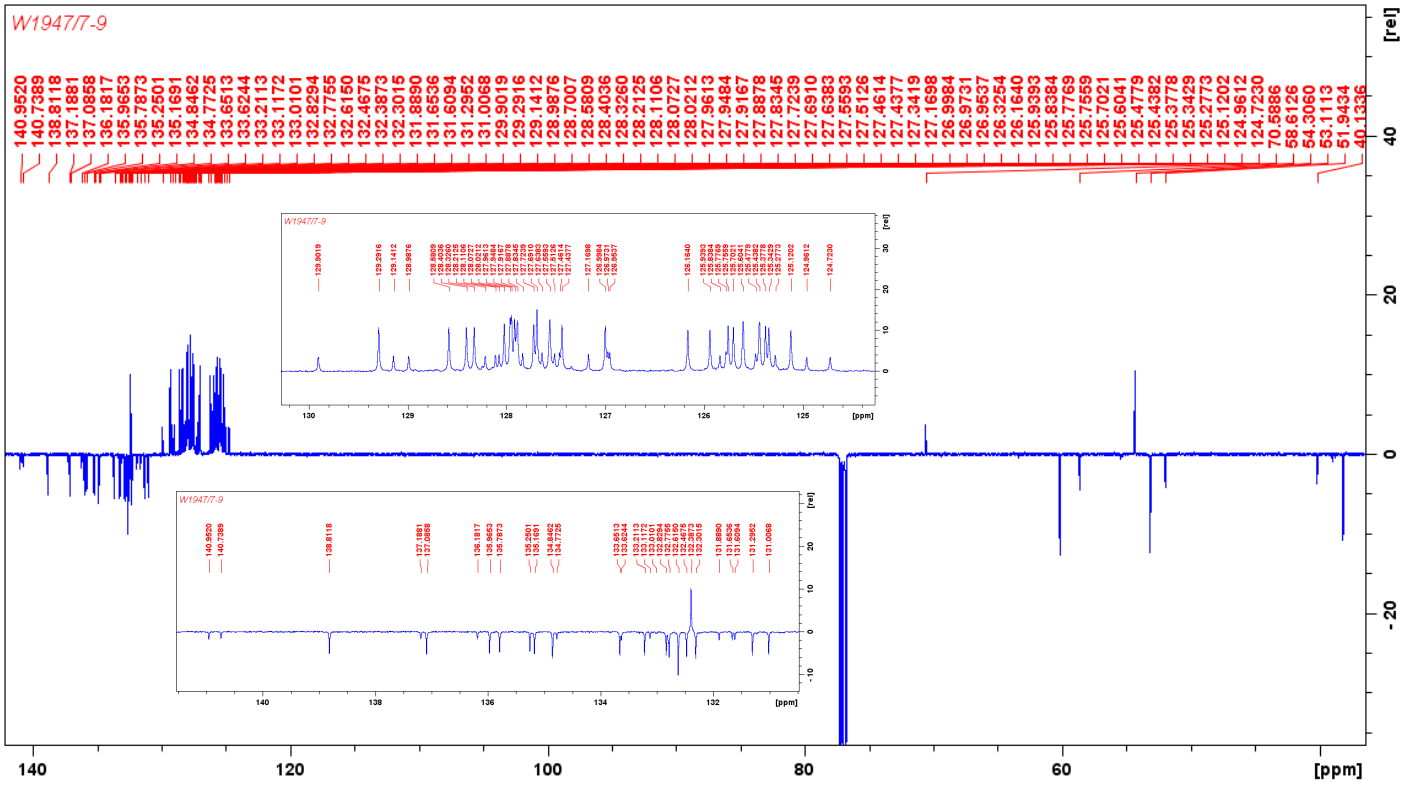


Figure S11: mixture of **5** and **6** (70:30) (^{1}H NMR in $CDCl_3$)

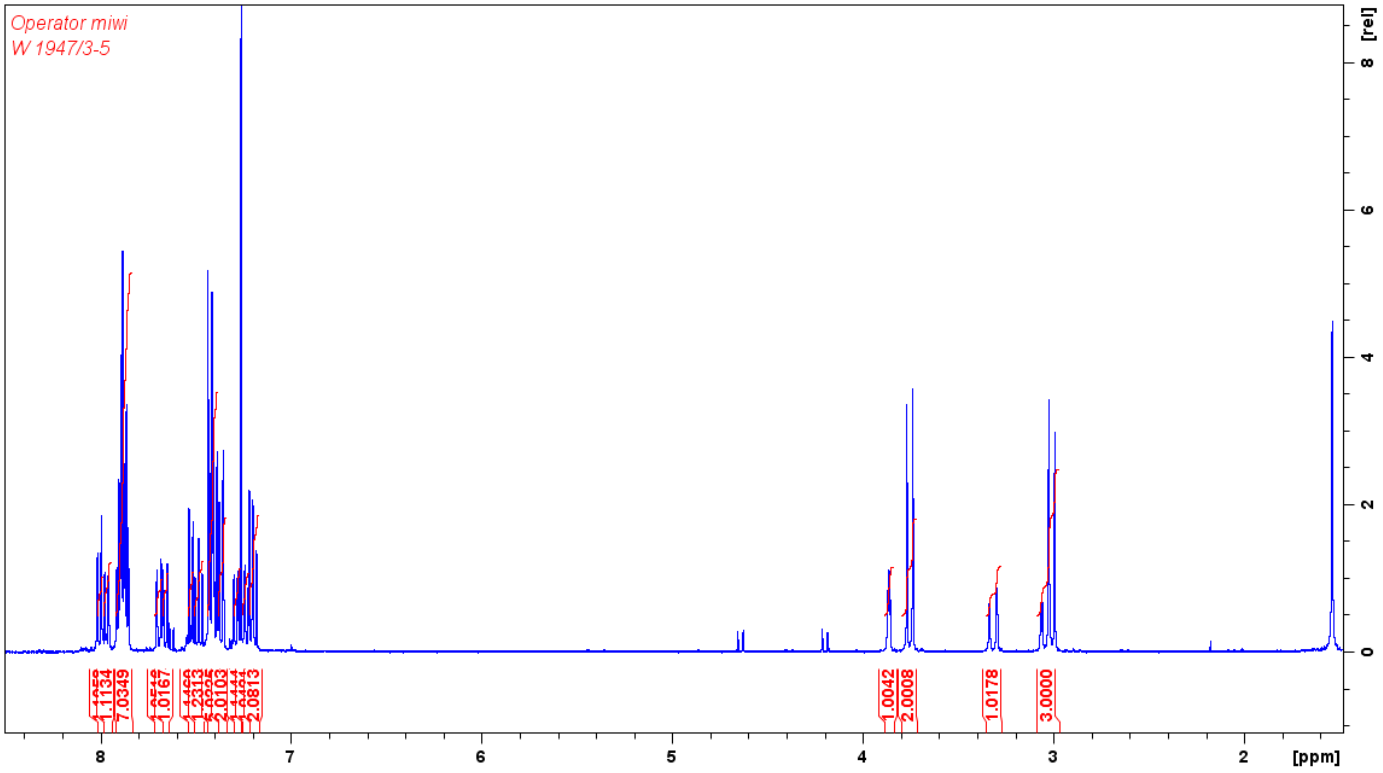


Figure S12: 7 (^{1}H NMR in $CDCl_3$)

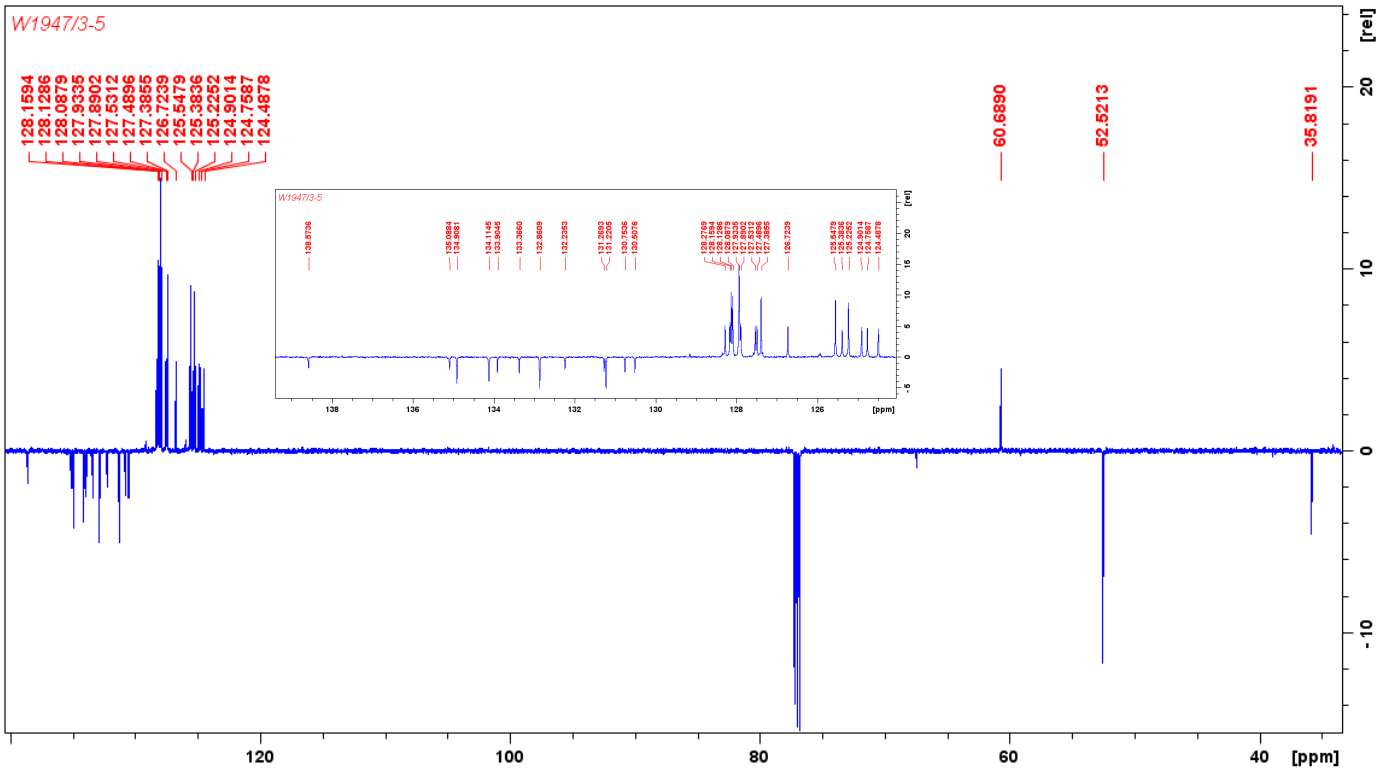


Figure S13: 7 (^{13}C NMR in $CDCl_3$)

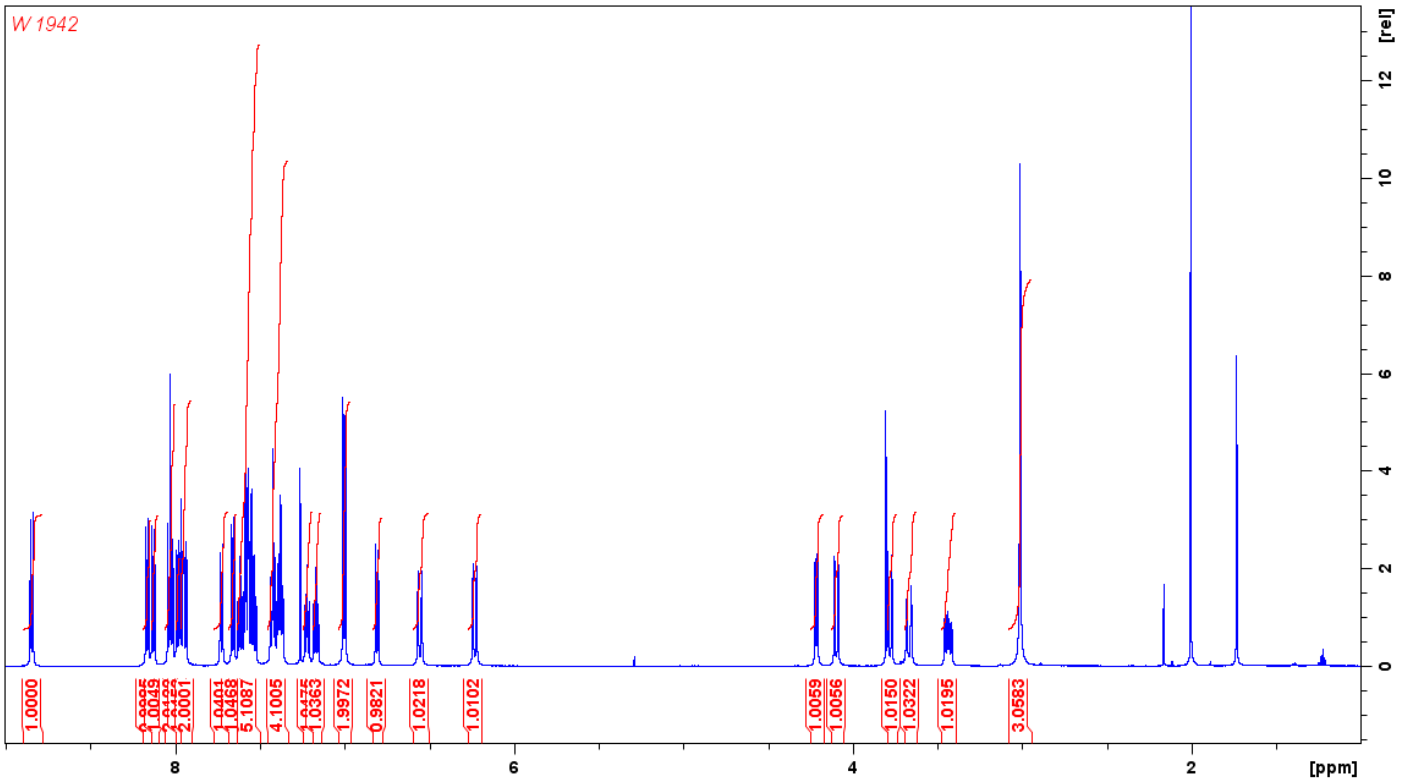


Figure S14: *N*-Methyl ammonium iodide of **4** (^{1}H NMR in $CDCl_3$)

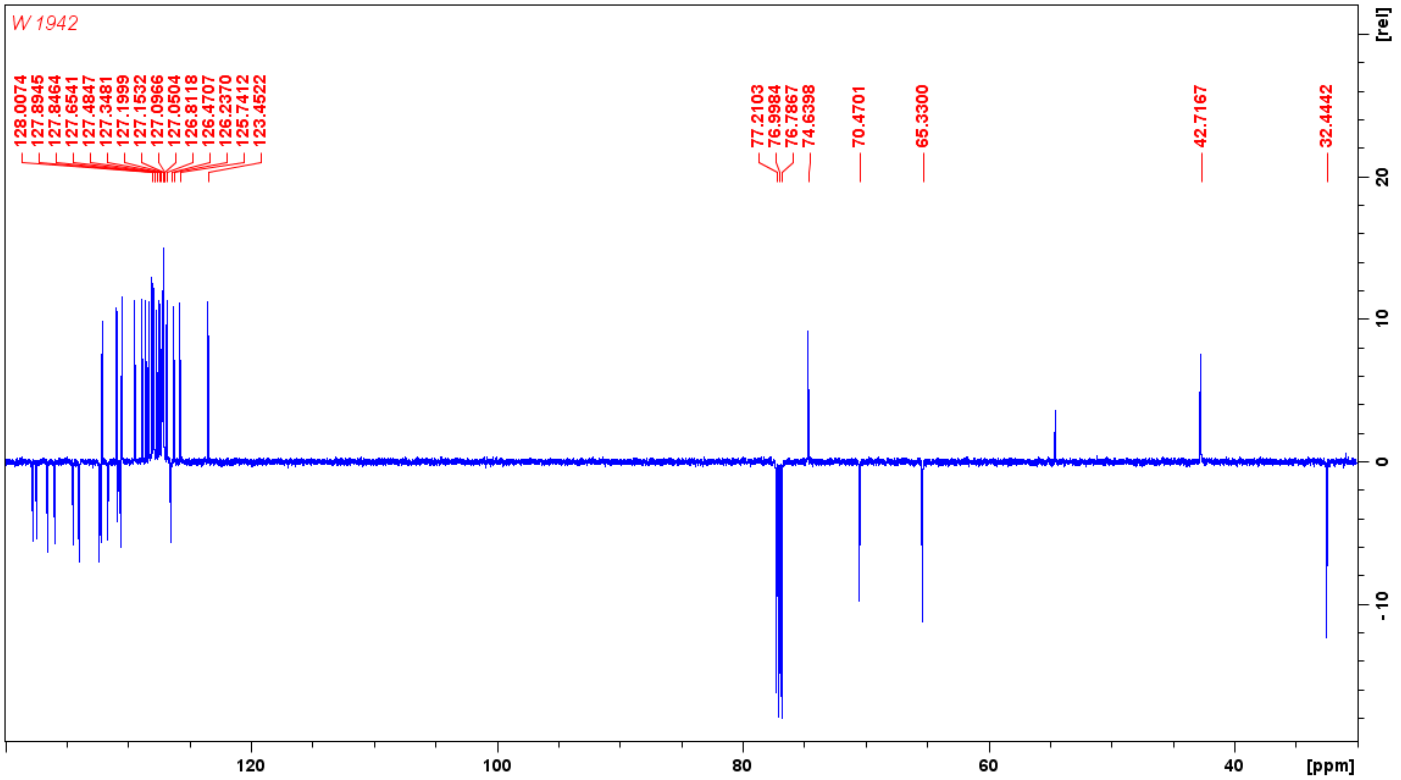


Figure S15: *N*-Methyl ammonium iodide of **4** (^{13}C NMR in $CDCl_3$)

Table S1: Experimental X-ray parameters and CCDC-Codes.

Sample	Machine	Source	Temp.	Detector Distance	Time/ Frame	#Frames	Frame width	CCDC
	Bruker		[K]	[mm]	[s]		[°]	
Meso2	D8	Mo	100	40	11.2	1846	0.400	2069313
Rac2	D8	Mo	100	30	20	2751	0.500	2069312
4	D8	Mo	100	25	8	919	0.700	2069311

Table S2: Short C-C contacts in *rac*-**2**.

Atom names	Distance [Å]
C2A—C39A	3.339
C3A—C39A	3.449
C3A—C40A	3.552
C4A—C40A	3.371
C4A—C41A	3.468
C4A—C34B	3.602
C4A—C36B	3.558
C5A—C40A	3.441
C5A—C36B	3.471
C6A—C36B	3.546
C6A—C37B	3.567
C36A—C4B	3.557
C36A—C5B	3.529
C36A—C6B	3.437
C37A—C6B	3.329
C37A—C7B	3.575
C36B—C4B	3.558
C36B—C5B	3.471
C36B—C6B	3.546
C39B—C2B	3.374
C39B—C3B	3.547
C40B—C3B	3.577
C40B—C4B	3.456
C40B—C5B	3.529
C41B—C4B	3.491

Table S3: Hydrogen bridges.

	Distance [Å]		**Angle [°]**	
meso-**2**	H1—Br1	2.387	O1—H1—Br2	165.66
	H2—Br2	2.403	O2—H2—Br2	171.09
rac-**2**	H45—Br1	2.435	C45—H45—Br1	175.02
	H46—Br1	2.479	C46—H46—Br1	171.49
	H48—Br2	2.453	C48—H48—Br2	167.84
	H49—Br2	2.506	C49—H49—Br2	160.28