Water co-encapsulation in an inverted molecular capsule

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NMR experimental part

All NMR spectra were recorded on 500 MHz Bruker spectrometer equipped with BTO2000 thermocouple. After sample filtration the spectra were taken immediately or the samples with water were kept over a drop of aqueous phase. For each guests the following set of samples was checked:
1$_2$+ **guest** without water
1$_2$+ **guest** in the presence of water
1$_2$+ **guest** + crotonic acid without water
1$_2$+ **guest** + crotonic acid in the presence of water
1$_2$+ *rac*-**guest** without water
1$_2$+ *rac*-**guest** in the presence of water
1$_2$+ *rac*-**guest** + crotonic acid without water
1$_2$+ *rac*-**guest** + crotonic acid in the presence of water

For the chiral recognition experiments crotonic acid was necessary to reach thermodynamic equilibrium during 10 day in heterogeneous samples.

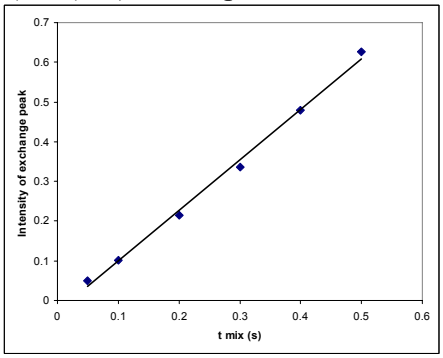
The EXSY spectra were recorded at 303 K on Bruker 500 MHz spectrometer with a phase sensitive NOESY pulse sequence supplied with the Bruker software. Spectra were measured for various mixing times. The integration of the 2D spectra was preformed using SpinWorks. Exchange rates were calculated according to following equations (ref. 1, 2):

$$k = \frac{1}{\tau_m} \ln \frac{r+1}{r-1}$$

$$r = \frac{I_{AA} + I_{BB}}{I_{AB} + I_{BA}}$$

$$k = k_1 + k_{-1}$$

Example of data:
(**1**$_2$⊃(*S*-**6**)$_1$ in the presence of water)



1. C. L. Perrin and T. J. Dwyer, *Chem. Rev.*, 1990, **90**, 935–967.
2. *„Dynamic NMR studies of supramolecular complexes"* Pons M., Millet O., *Progress In Nuclear Magnetic Resonance Spectroscopy, 38* , **2001,** 267-324.

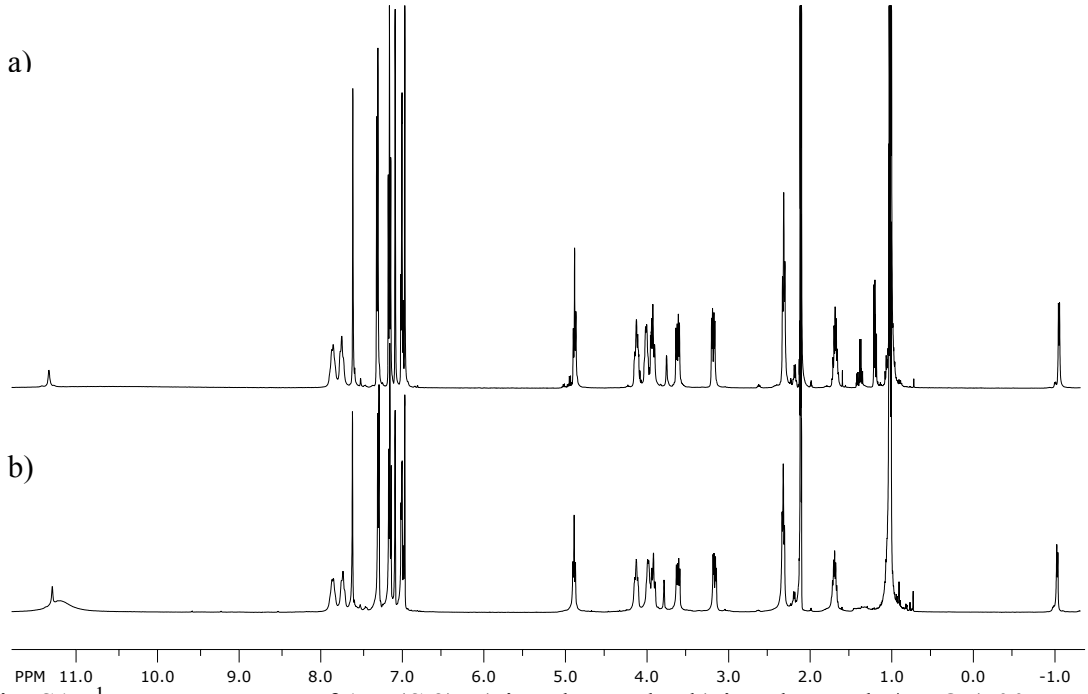


Fig. S1. ^{1}H NMR spectra of $\mathbf{1}_2$ +(*S*-**2**) a) in toluene-d_8 ; b) in toluene-d_8 / H_2O (500MHz, 303K)

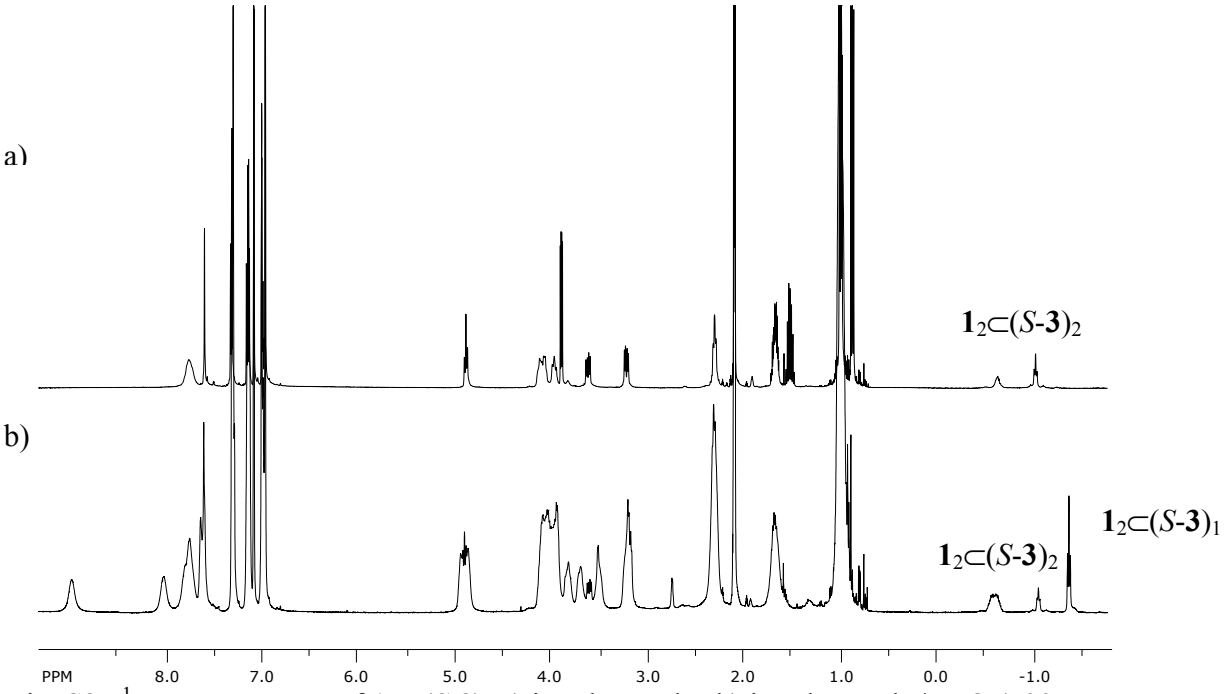


Fig. S2. ^{1}H NMR spectra of $\mathbf{1}_2$ +(*S*-**3**) a) in toluene-d_8 ; b) in toluene-d_8 / H_2O (500MHz, 303K).

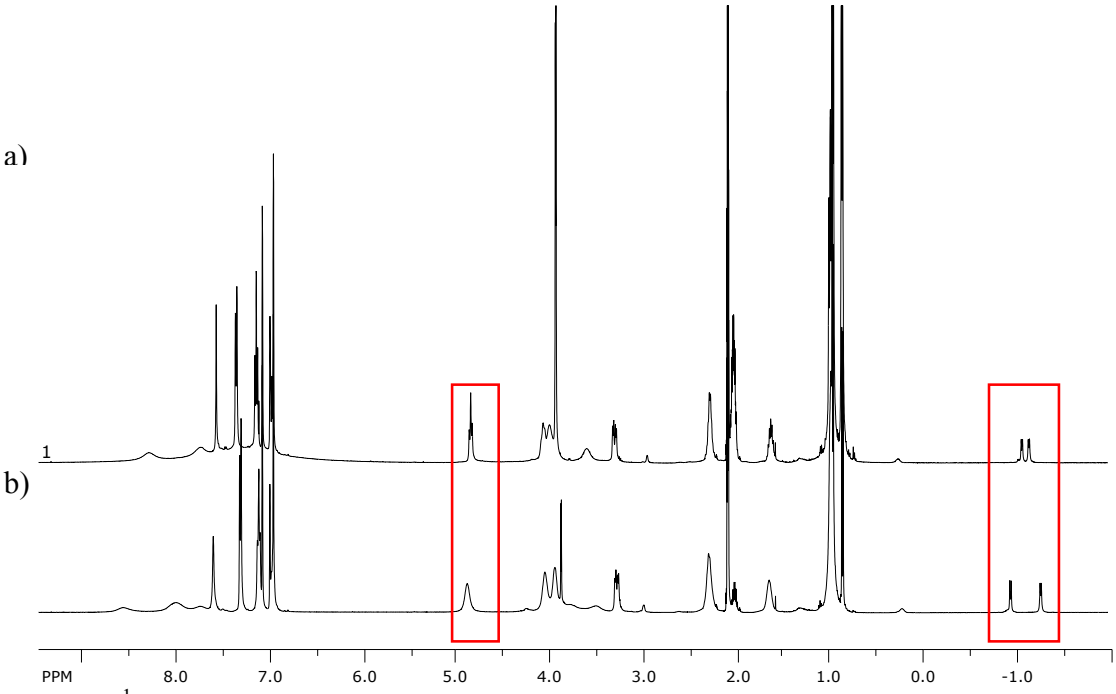


Fig. S3. ^{1}H NMR spectra of $\mathbf{1}_2\subset(S\text{-}\mathbf{4})_1$ a) in toluene-d_8 ; b) in toluene-d_8 / H_2O (500MHz, 303K)

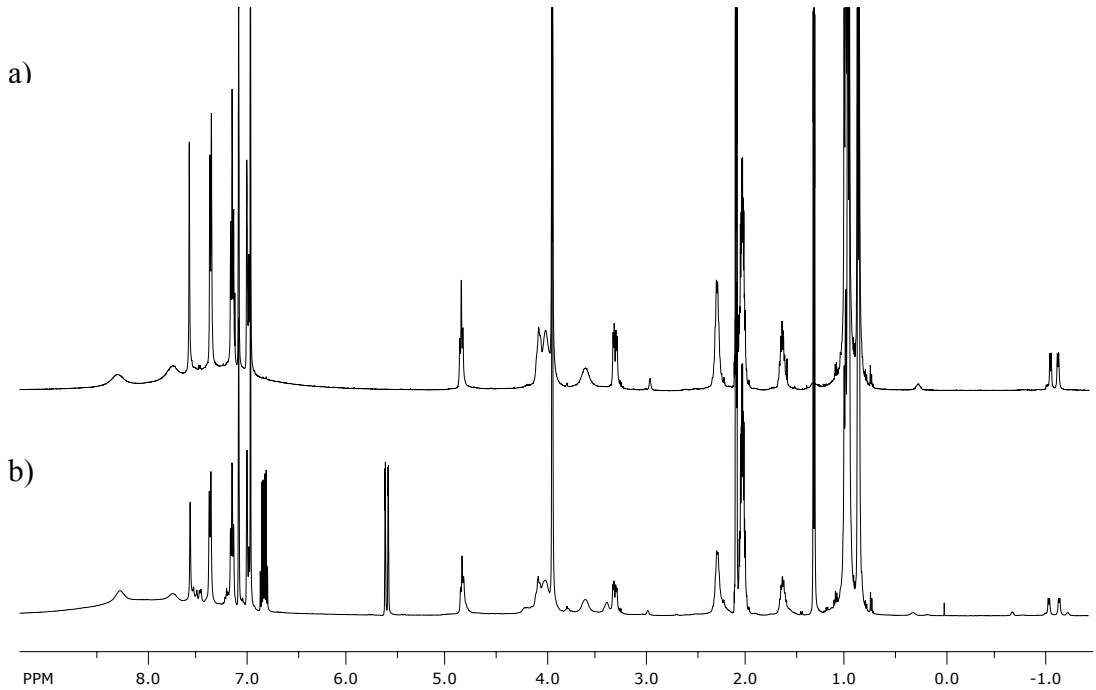


Fig. S4. Control ^{1}H NMR spectra of $\mathbf{1}_2\subset(S\text{-}\mathbf{4})_1$ a) in toluene-d_8; b) +crotonic acid in toluene-d_8 (500MHz, 303K)

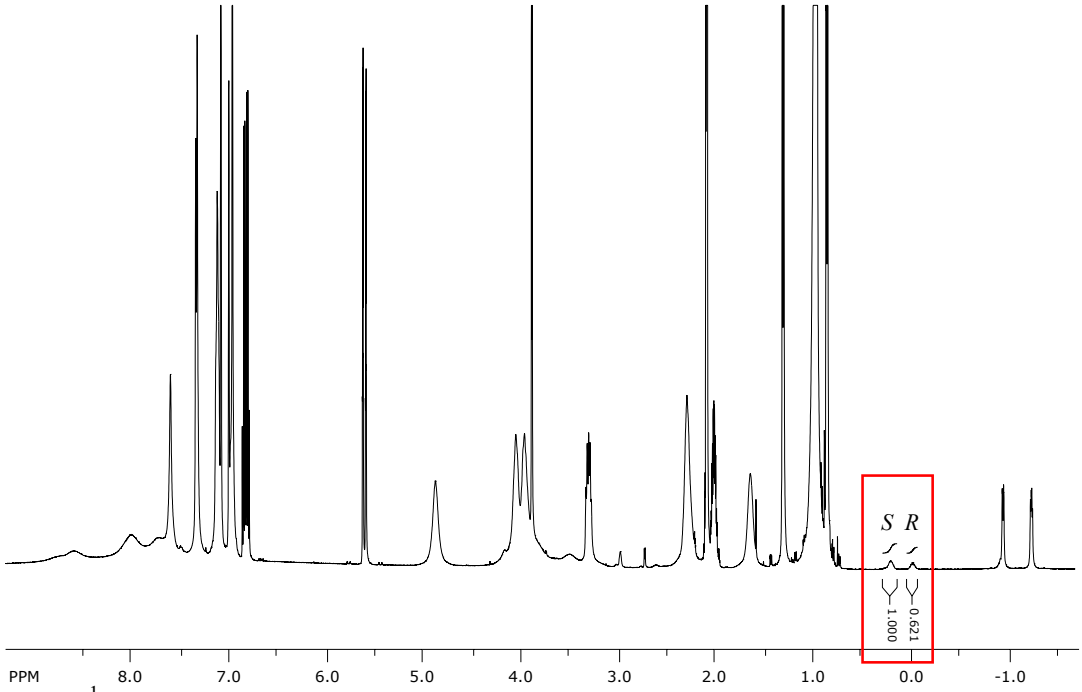


Fig. S5. ^{1}H NMR spectrum of $\mathbf{1}_2$ + rac-**4** in toluene-d_8 / H_2O +crotonic acid (500MHz, 303K)

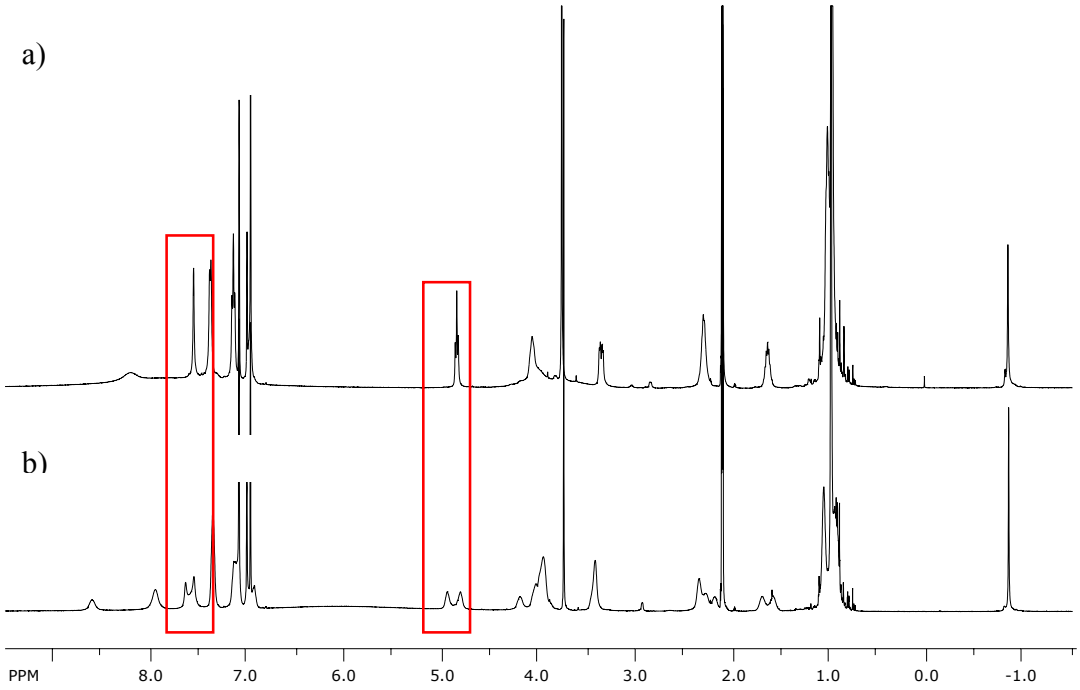


Fig. S6. ^{1}H NMR spectra of $\mathbf{1}_2\subset(S\text{-}\mathbf{5})_1$ a) in toluene-d_8 / H_2O; b) in toluene-d_8 (500MHz, 303K)

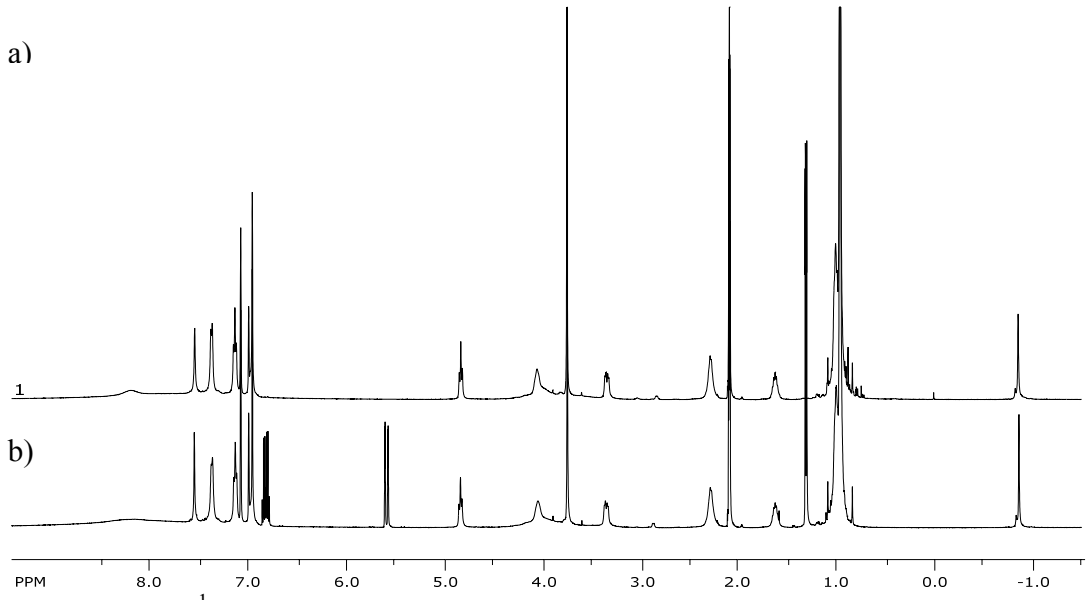


Fig. S7. Control ^{1}H NMR spectra of $\mathbf{1}_2\subset(\mathbf{5})_1$ a) in toluene-d_8; b) +crotonic acid in toluene-d_8 (500MHz, 303K)

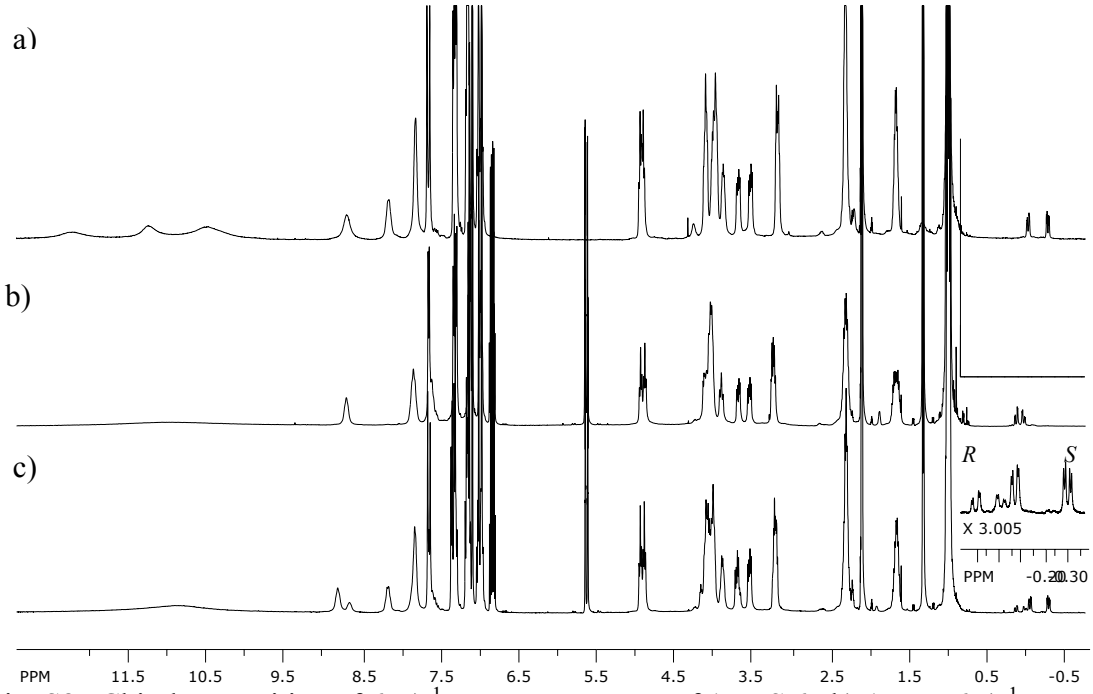


Fig. S8. Chiral recognition of **6**. a) ^{1}H NMR spectrum of $\mathbf{1}_2$ + *S*-**6** , b) $\mathbf{1}_2$ + *R*-**6** a) ^{1}H NMR spectrum of $\mathbf{1}_2$ + rac-**6** (toluene-d_8 / H_2O, crotonic acid, 500MHz, 303K).

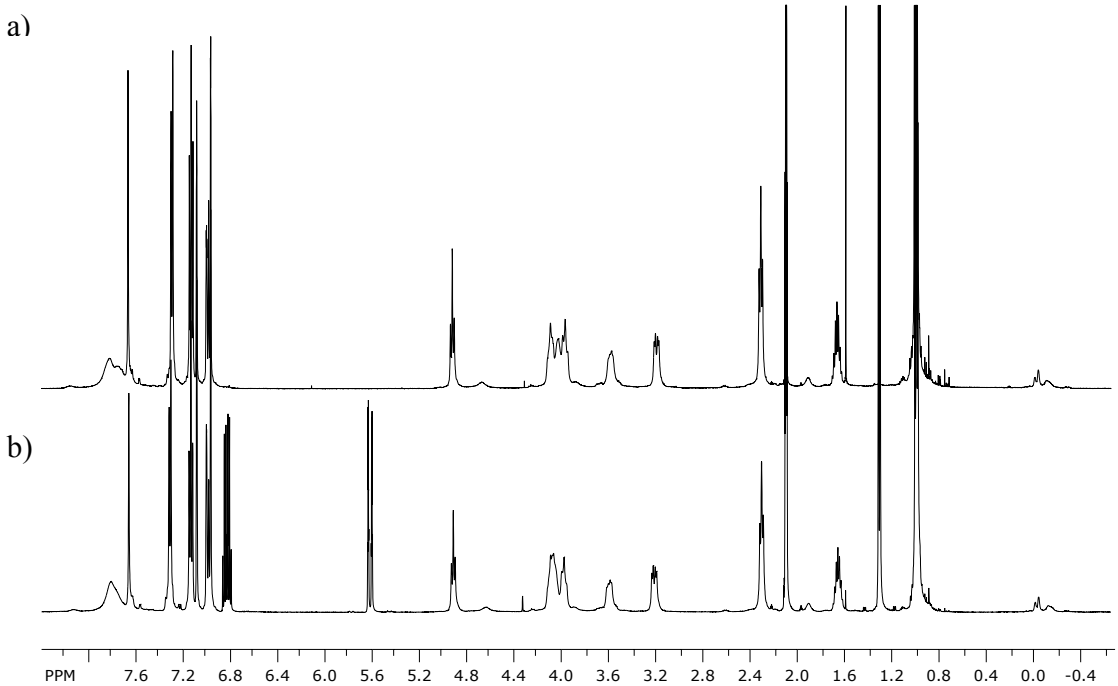


Fig. S9. Control ^{1}H NMR spectra of $\mathbf{1}_2\subset(S\text{-}\mathbf{6})_1$ a) in toluene-d_8; b) +crotonic acid in toluene-d_8 (500MHz, 303K)

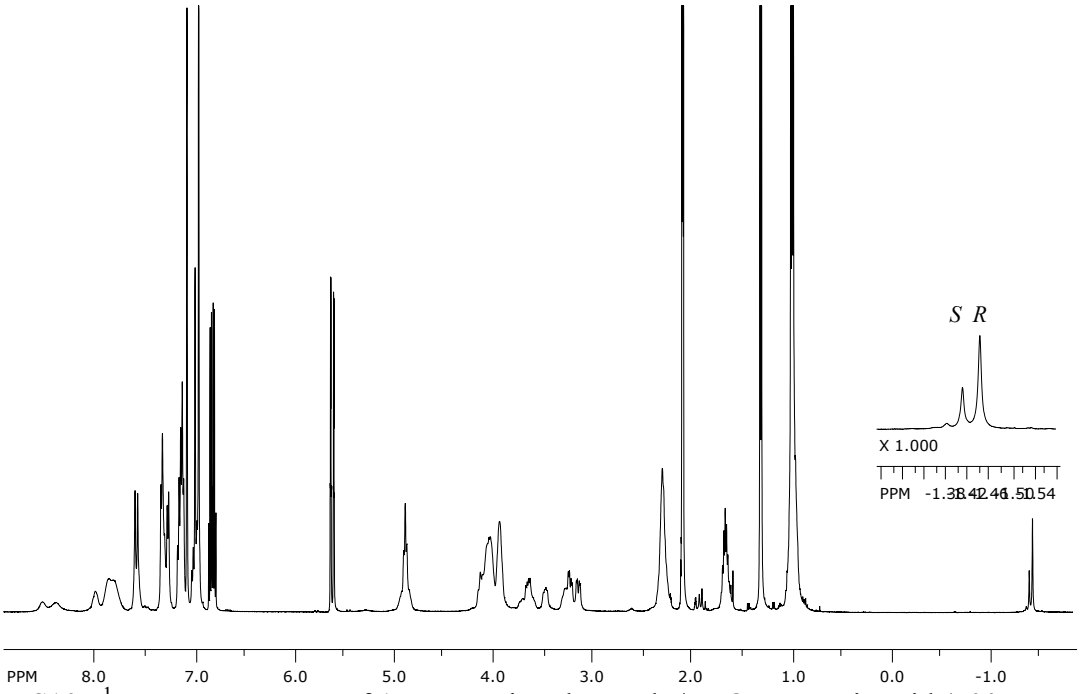


Fig. S10. ^{1}H NMR spectrum of $\mathbf{1}_2$ + rac-**7** in toluene-d_8 / H_2O +crotonic acid (500MHz, 303K)