An achiral form of the hexameric resorcin[4]arene capsule sustained by hydrogen bonding with alcohols

Supporting Information

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General Experimental Details:

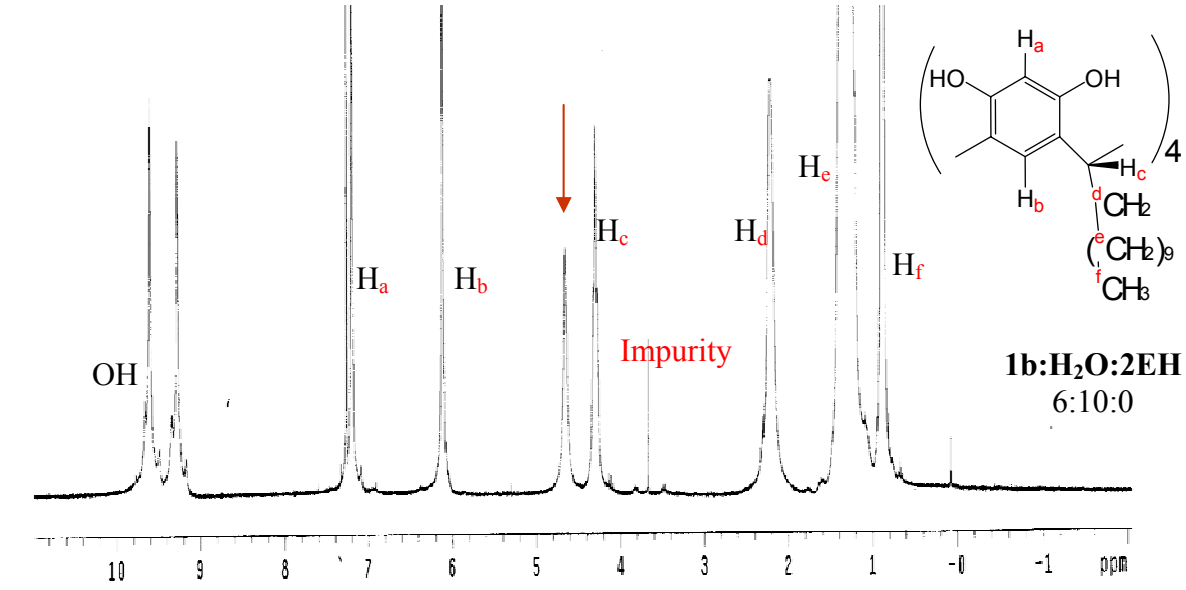
All reagents were obtained from Acros (Pittsburgh, PA) or Aldrich (Milwaukee, WI) and used without further purification unless otherwise stated. Deuterated chloroform was obtained from Cambridge Isotope Laboratories. All solvents employed in solution studies were dried over 4Å molecular sieves. Elemental analysis was carried out on a Perkin-Elmer PE2400 microanalyzer in our laboratory. Thermal Gravimetric Analysis was carried out using TA Instruments TGA 2050 under a constant stream of dry nitrogen gas. Single crystals and bulk samples of **3a**⊂3(**2EH**) were obtained by slow evaporation of a solution of **1a** in (±)-2-ethylhexanol under ambient conditions. Single crystal X-ray diffraction data (-100°C) were collected on a Siemens SMART 1000 CCD X-ray diffractometer. For further details, one should consult the CIF file.

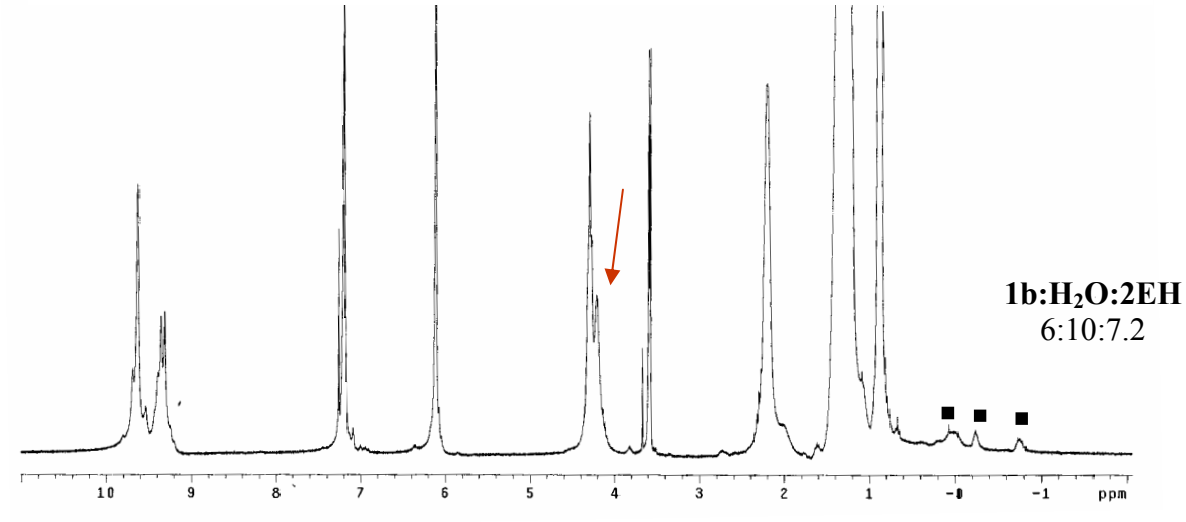
S1. NMR data

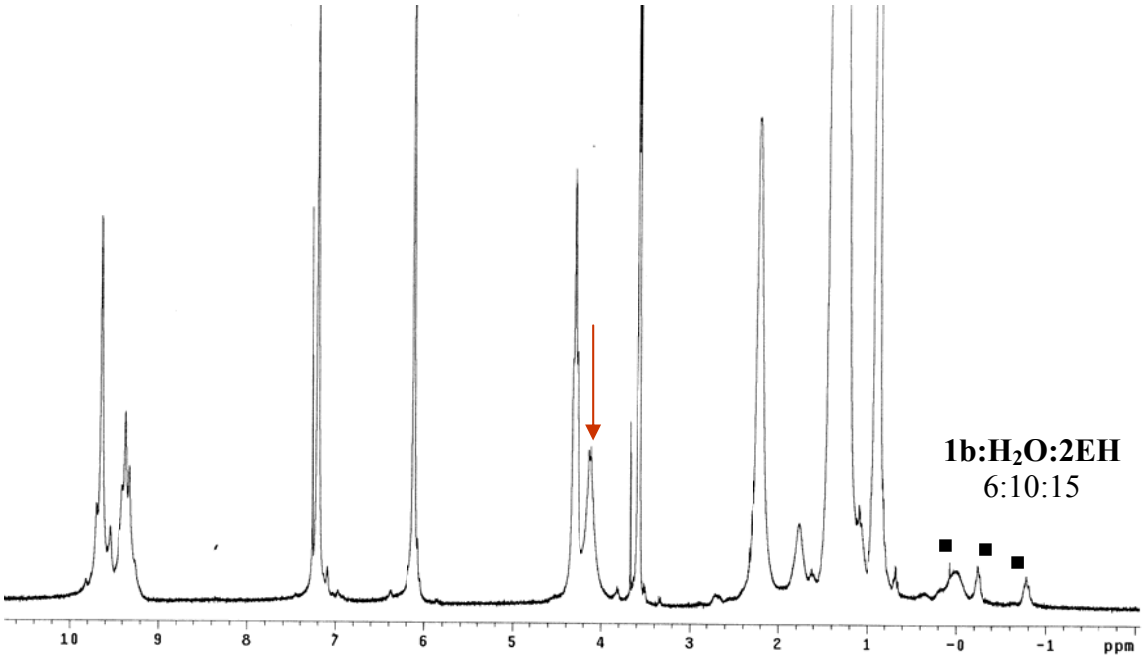
^{1}H NMR (300MHz) and ^{13}C NMR (75.5 MHz) spectra were recorded on a Varian Inova Spectrometer. All spectra were collected at 25˚C and referenced to the residual solvent signal. Proton spectra were obtained with a spectra width of 8000 Hz and a minimum of 64 scans.

27 mM stock solutions of **1b**, in $CDCl_3$ and $CHCl_3$, respectively, were prepared and portions were placed in NMR tubes. To these tubes were carefully syringed specific volumes of (±)-2-ethylhexanol and the accompanying spectral changes were immediately observed. These experiments are reproducible. Representative complete spectra are given in the following pages. The water peaks are indicated by red arrows, encapsulated (±)-2-ethyl-hexanol peaks are indicated by dark bullets, and encapsulated $CHCl_3$ peaks are indicated by red bullets.

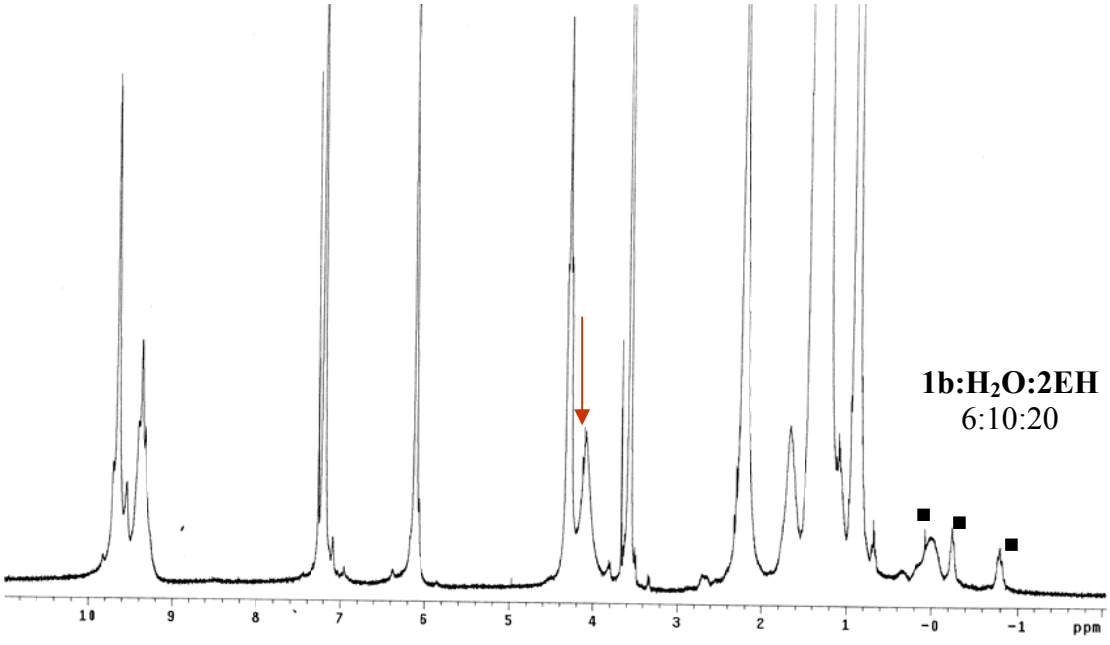
Addition of (±)-2EH to 1b in $CDCl_3$.



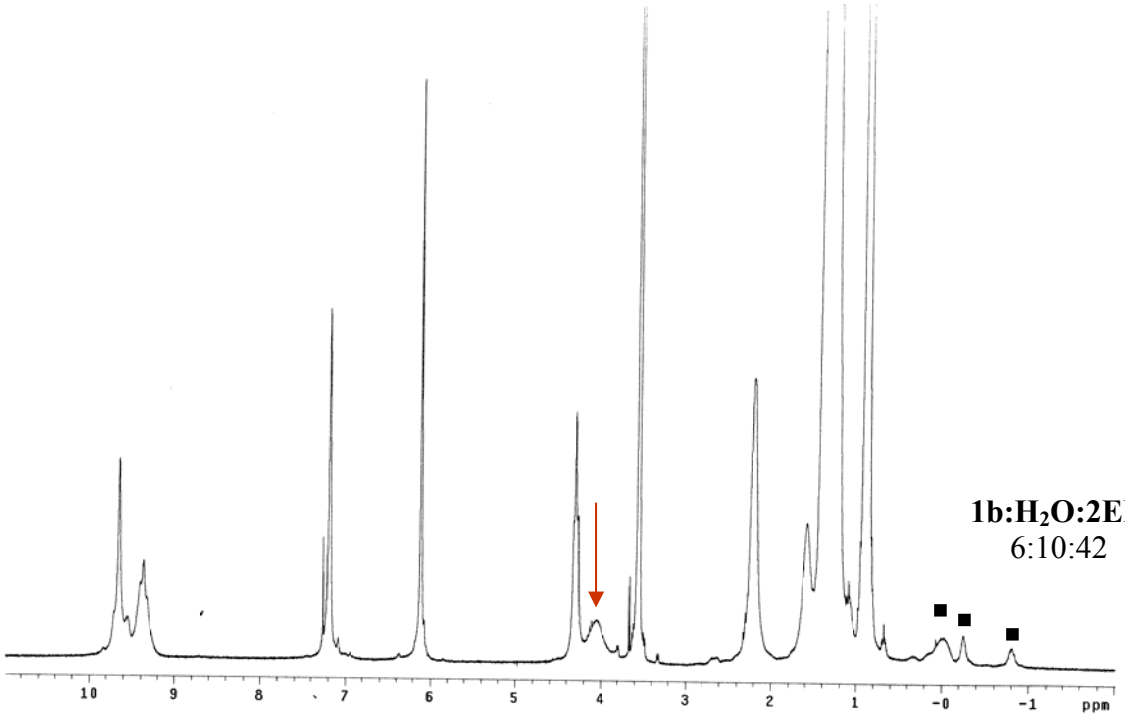




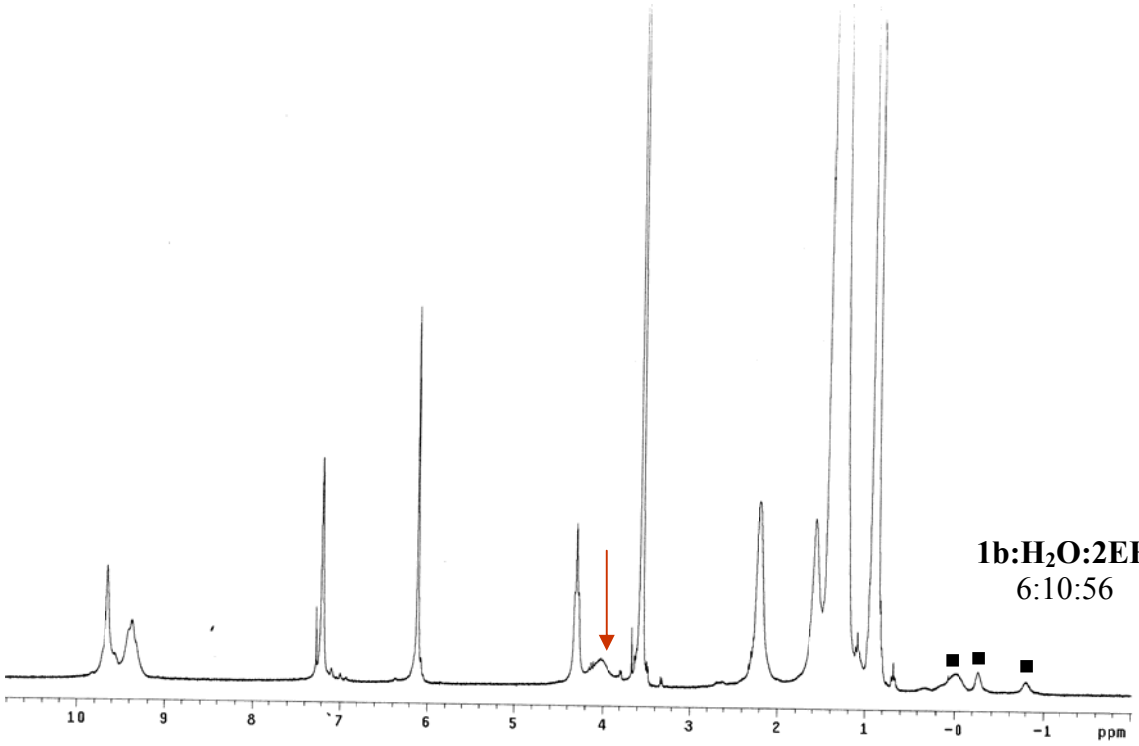
1b:H_2O:2EH
6:10:15



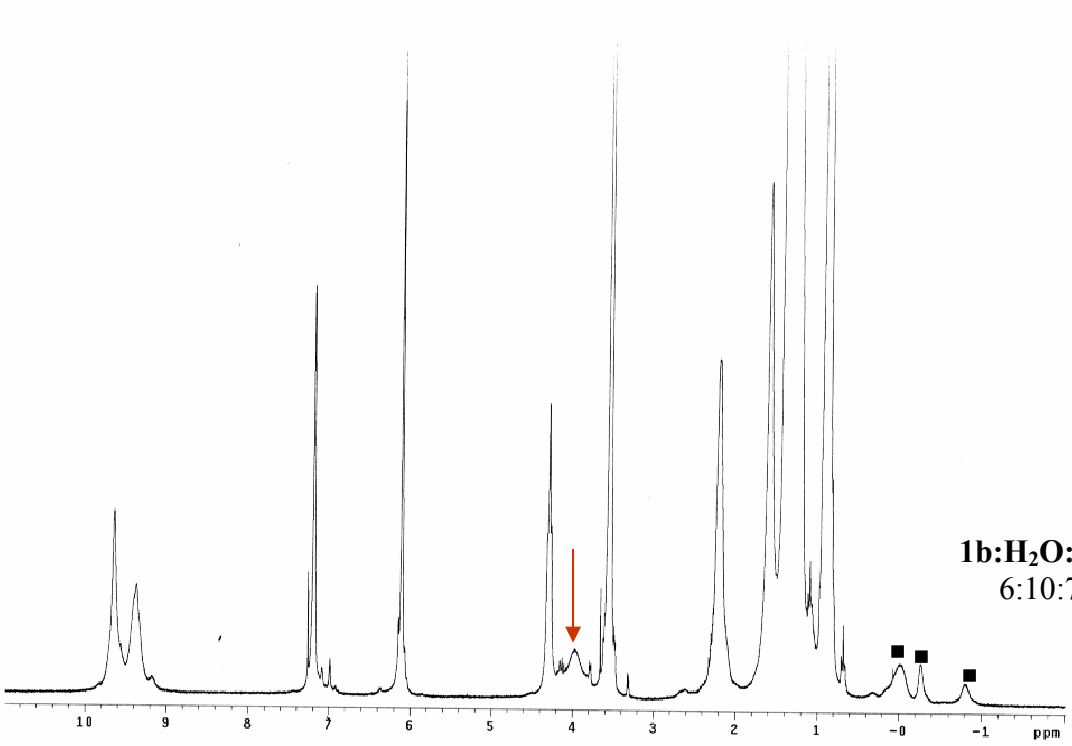
1b:H_2O:2EH
6:10:20



1b:H_2O:2EH
6:10:42

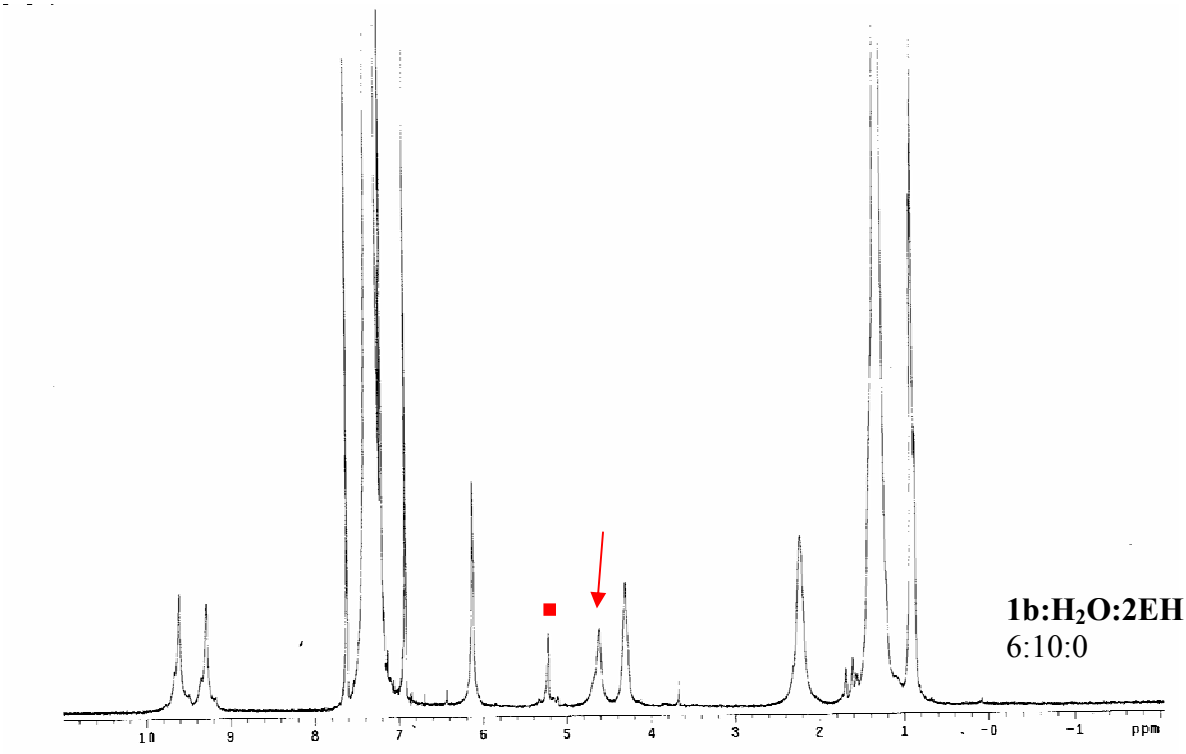


1b:H_2O:2EH
6:10:56

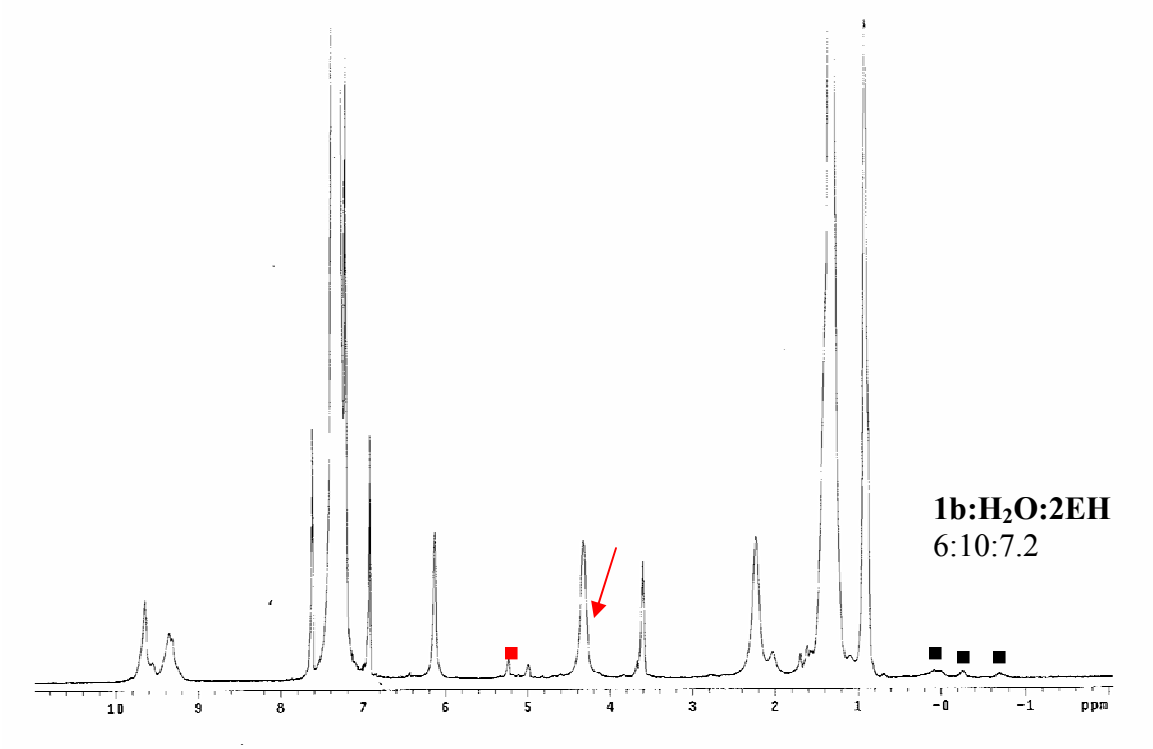


1b:H_2O:2EH
6:10:72

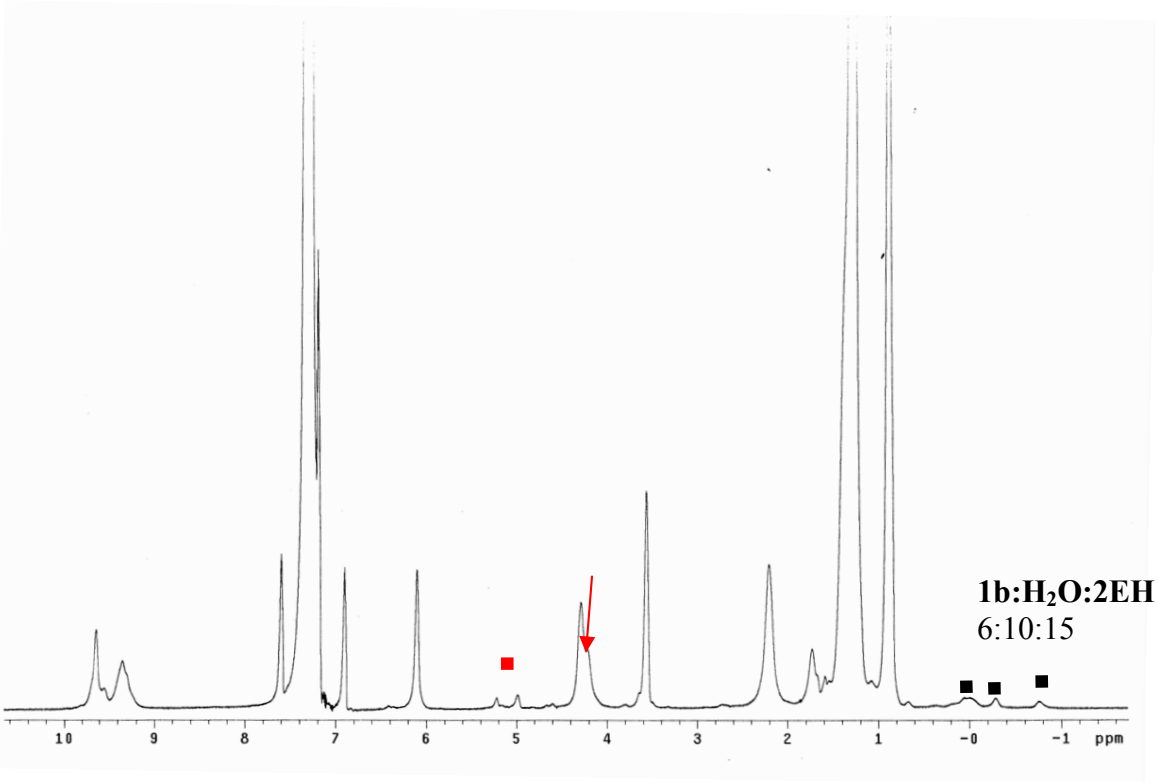
Addition of (±)-2EH **to** 1b **in $CHCl_3$.**



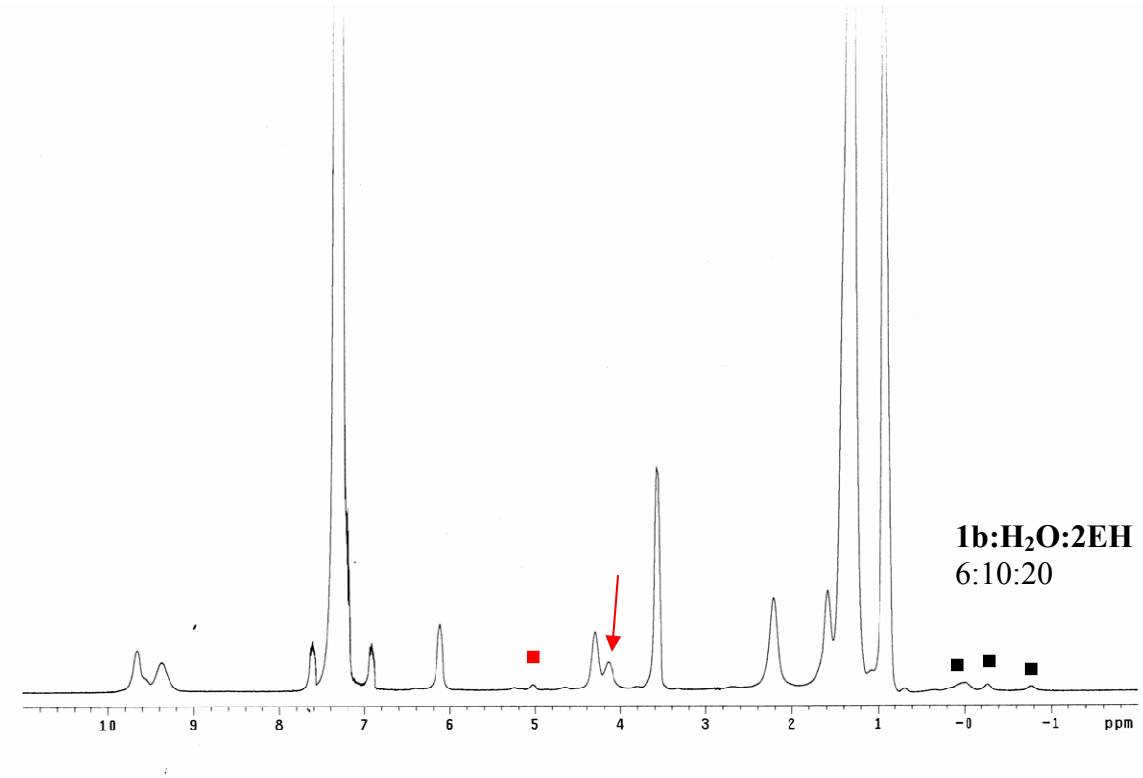
1b:H_2O:2EH
6:10:0



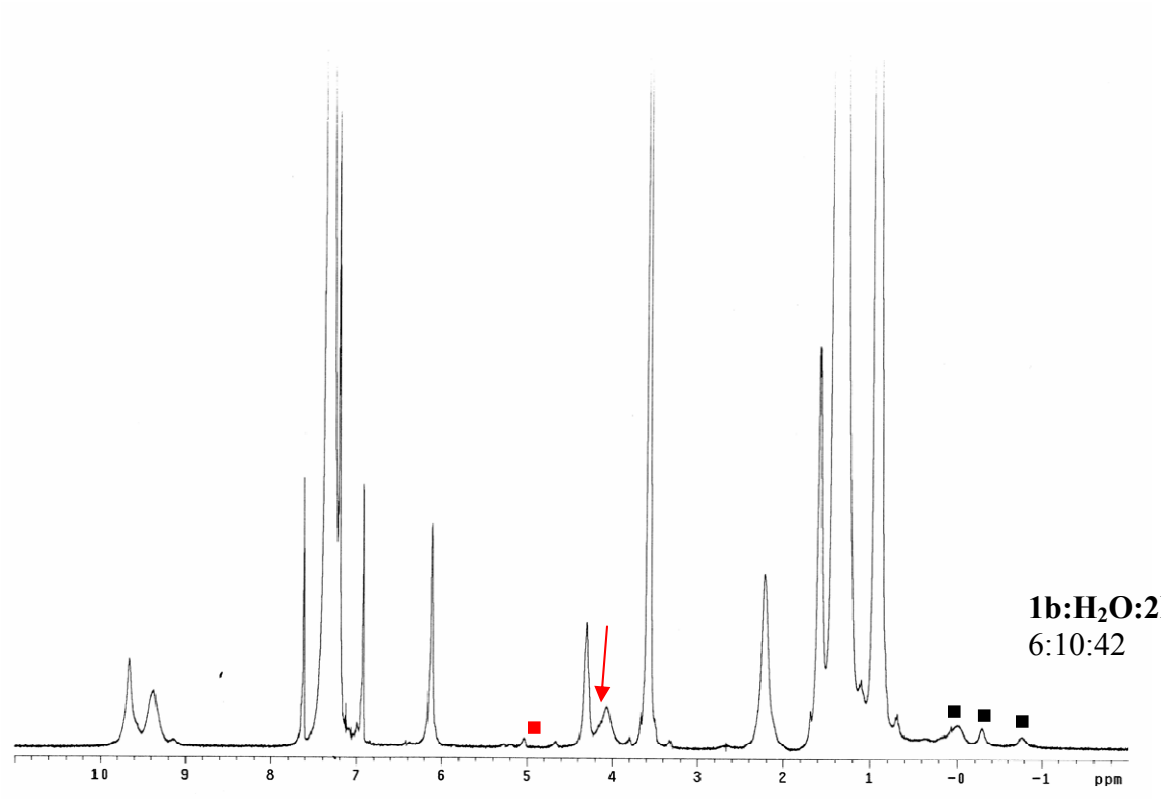
1b:H_2O:2EH
6:10:7.2



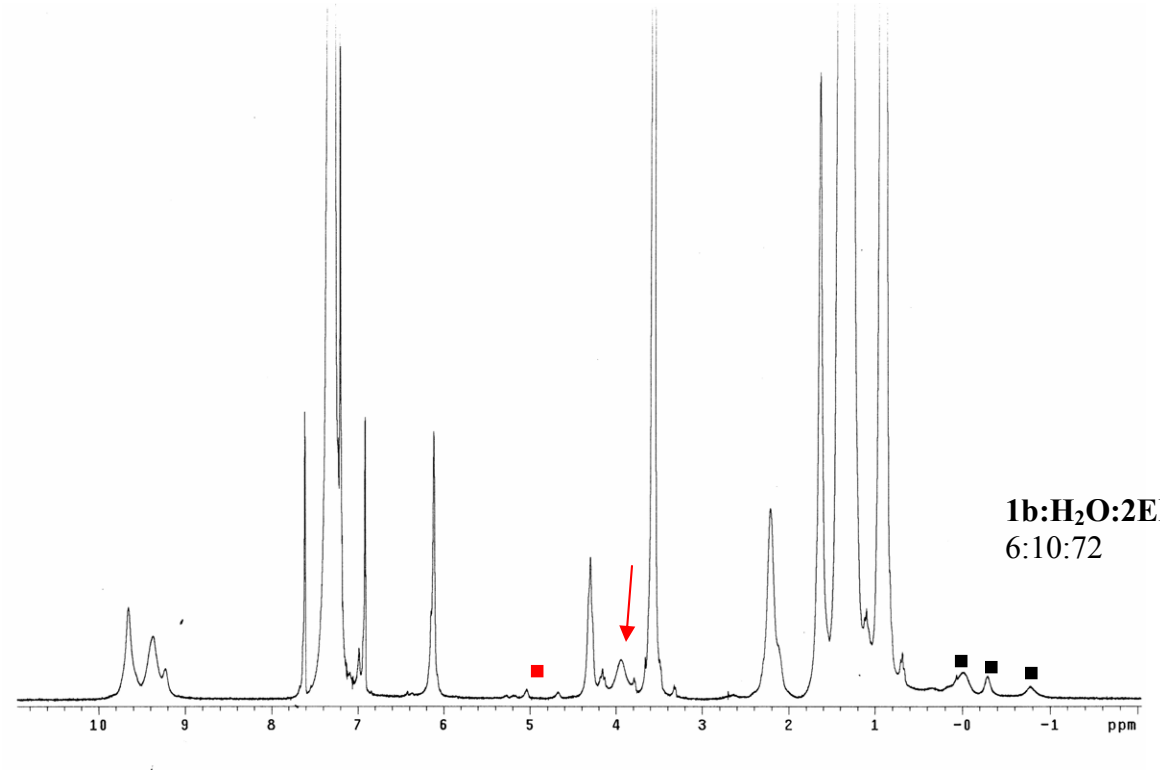
1b:H_2O:2EH
6:10:15



1b:H_2O:2EH
6:10:20

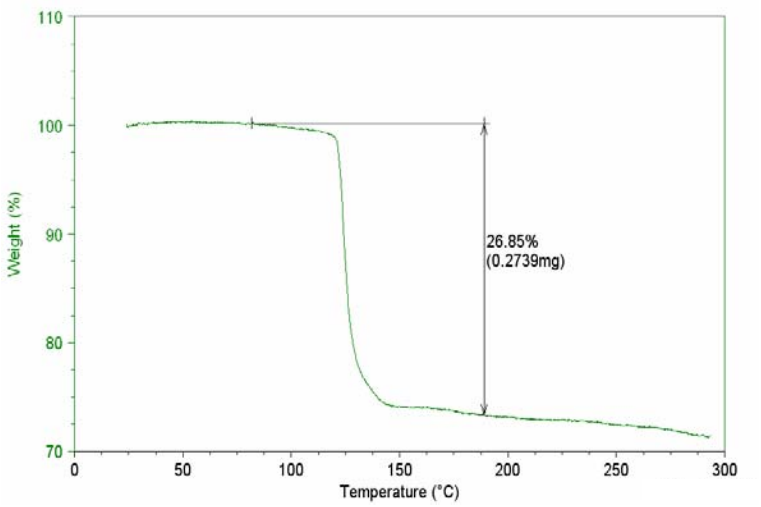


1b:H_2O:2EH
6:10:42



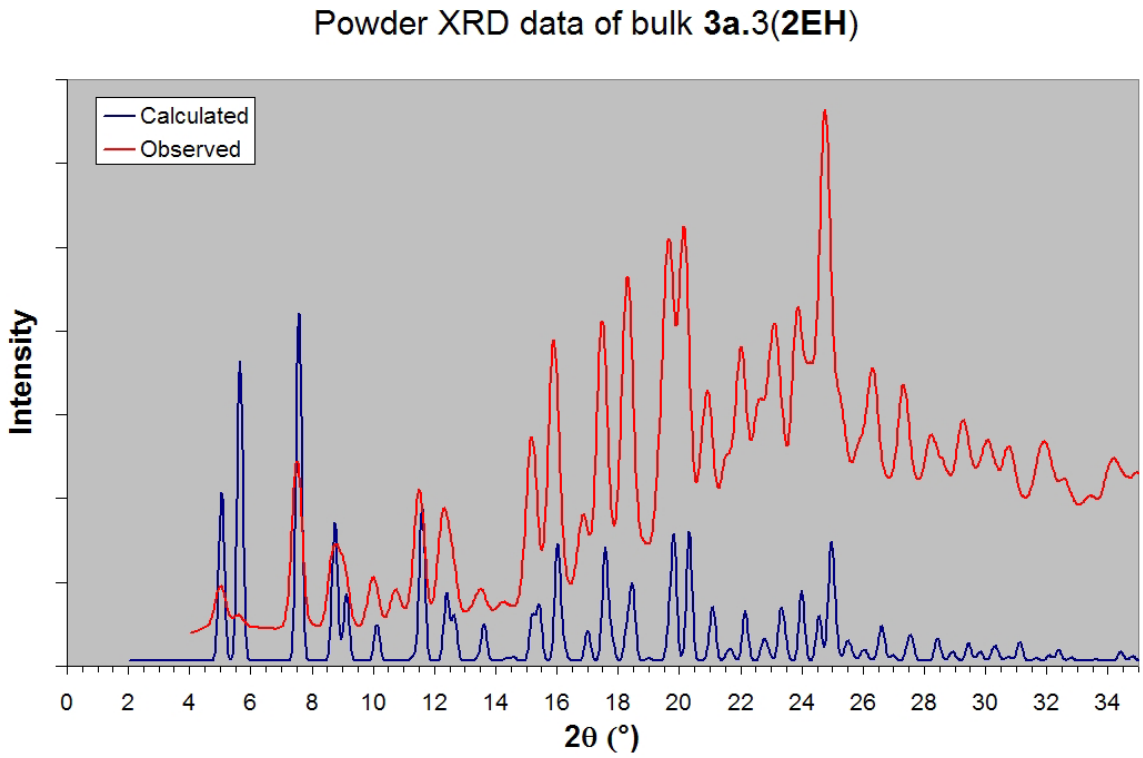
1b:H_2O:2EH
6:10:72

S2. Thermal Gravimetric Analysis



S3. Powder X-ray Diffraction

Powder X-ray diffraction experiments on bulk **3a**⊂3(**2EH**) were conducted on a Rigaku RAPID R-Axis X-ray diffractometer using Cu-Kα radiation. Data analysis and pattern indexing were performed using Jade 5.0 software. As shown below, the room temperature powder pattern of the bulk material compares favorably to the calculated pattern (arbitrarily broadened) derived from the low temperature single crystal data. These data confirm that the bulk material consists of **3a**⊂3(**2EH**). The observed powder pattern could be indexed to a hexagonal unit cell that is nearly identical (though slightly larger in volume due to temperature differences) to that obtained in the single crystal experiment. For details of the indexing results, the reader should consult the accompanying PowderXRDInexing.txt file.



Pattern Indexing Results:

Hexagonal, a = 35.10(4) Å, c = 18.44(2) Å, V = 19685 Å^3

Unit Cell from single crystal data:

Hexagonal, a = 34.9756(29) Å, c = 18.246(3) Å, V = 19330 Å^3