

ELECTRONIC SUPPLEMENTARY INFORMATION (ESI)

**Glycosyl squaramide: a new class of supramolecular gelators**

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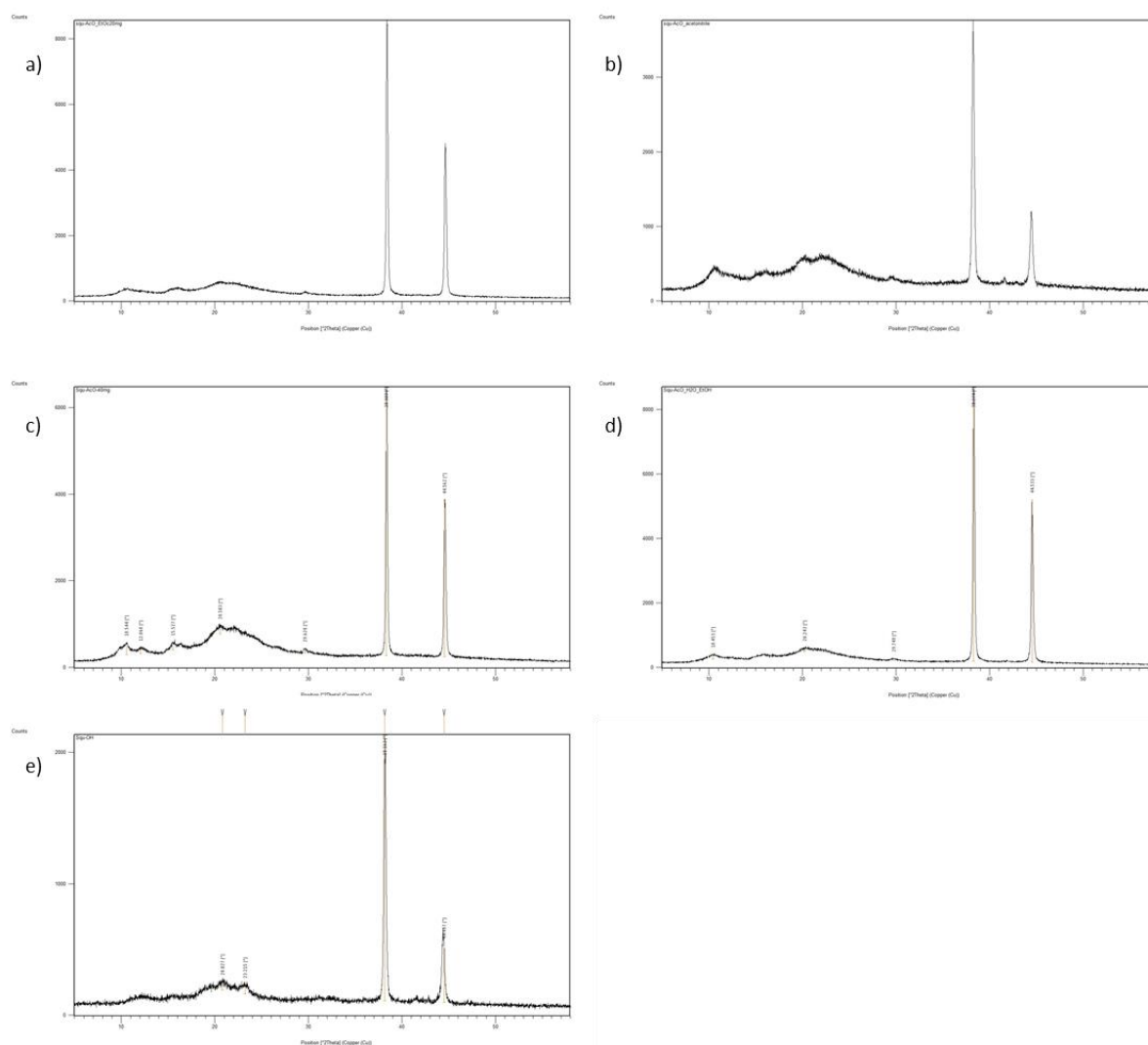
**Figure ESI3.** <sup>1</sup>H-NMR spectra of **5** in (D<sub>3</sub>)-acetonitrile at increasing concentrations.

**Figure ESI4.** FTIR-ATR spectra of **6** at 0.1 w/v % with EtOH:H<sub>2</sub>O (1:1)

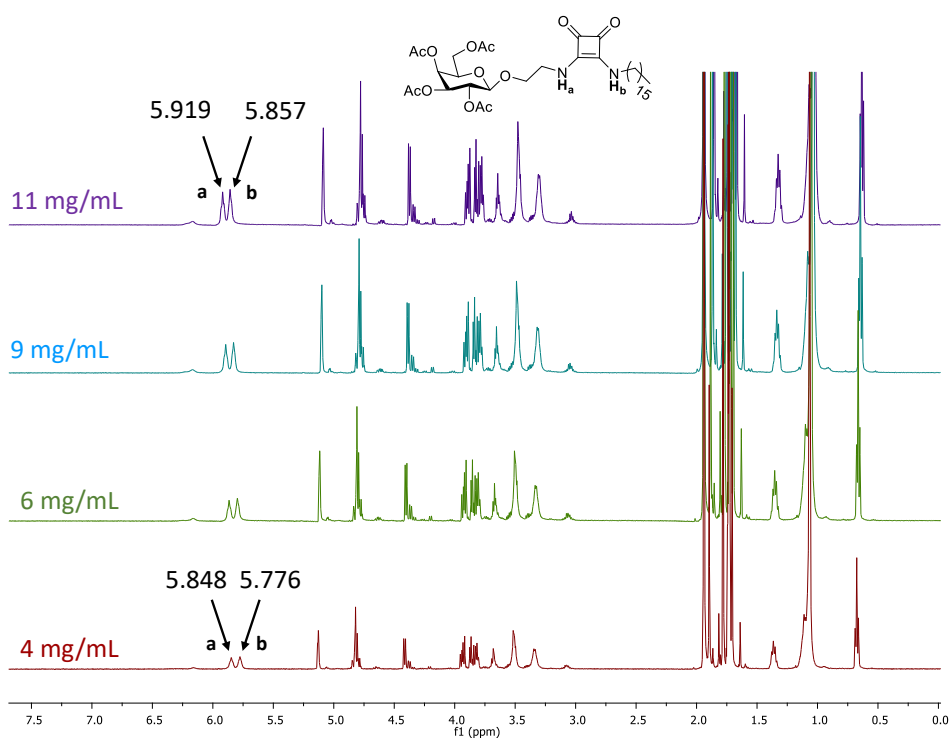
**Figure ESI5-ESI14.** <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra of compounds **1-3**, **5** and **6**.



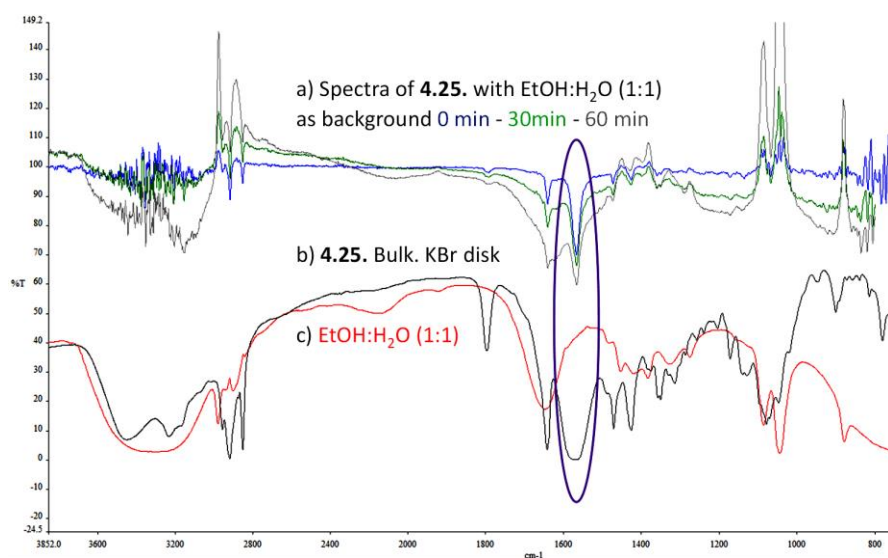
**Figure ESI1.** Physical appearance of the gel formed by **6** in EtOH:H<sub>2</sub>O (1:1) 15 months after formulation.



**Figure ESI2.** XRD diffractograms of glycosquaramides xerogels: a) **5** in EtOAc; b) **5** in MeCN; c) **5** in EtOH; d) **5** in EtOH:H<sub>2</sub>O (1:1); e) **6** in EtOH:H<sub>2</sub>O (1:1). All gels were prepared at CGC. X-ray powder patterns were recorded on an Inel Equinox 3000 powder diffractometer, fitted with a curved position sensitive detector (calibrated using Y<sub>2</sub>O<sub>3</sub>) between 5 and 60 ° (2 $\theta$ ) using Cu K $\alpha$  radiation ( $\lambda = 1.54178 \text{ \AA}$ ). Settings: X-ray tube: 35 kV, 25 mA; Scan time: 1200 s. The peaks at 38.2 and 44.3 are from the Al sample holder. Because of the small sample sizes, the floor of the sample holder was not completely covered.



**Figure ES13.**  $^1\text{H-NMR}$  spectra of **5** in  $(\text{D}_3)$ -acetonitrile at increasing concentrations. The solutions were prepared in a NMR tube and heated to aid the solubility of the gelator in the solvent. Spectra were measured at 300 K (27 °C), which is the standard operation temperature of the NMR spectrometer.



**Figure ES14.** a) FTIR-ATR spectra of **4.25**. 0.1 w/v % with EtOH:H<sub>2</sub>O (1:1) as background from 0 – 60 min; b) **4.25**. Bulk state (KBr disk) and c) FTIR-ATR spectra of EtOH:H<sub>2</sub>O (1:1).

Figure ES15.  $^1\text{H}$  NMR spectrum of 3,4-di(prop-2-yn-1-ylamino)cyclobut-3-ene-1,2-dione **1**

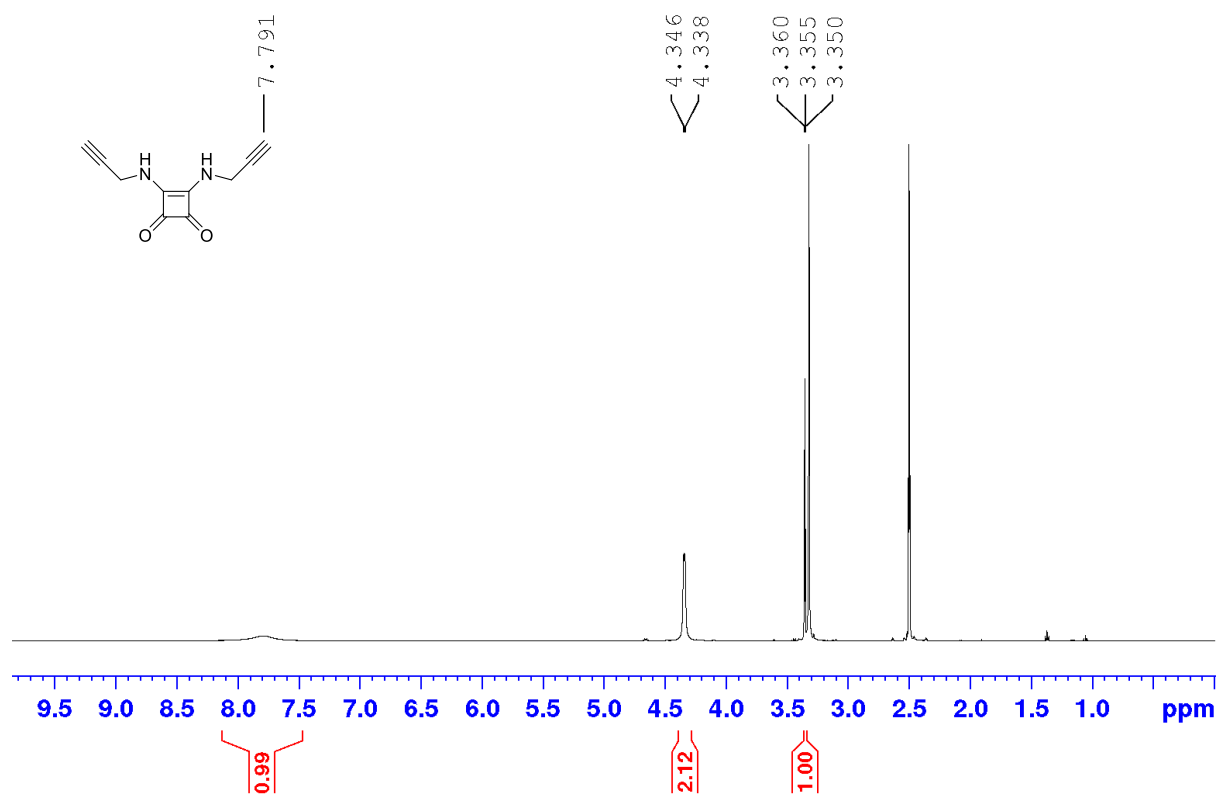
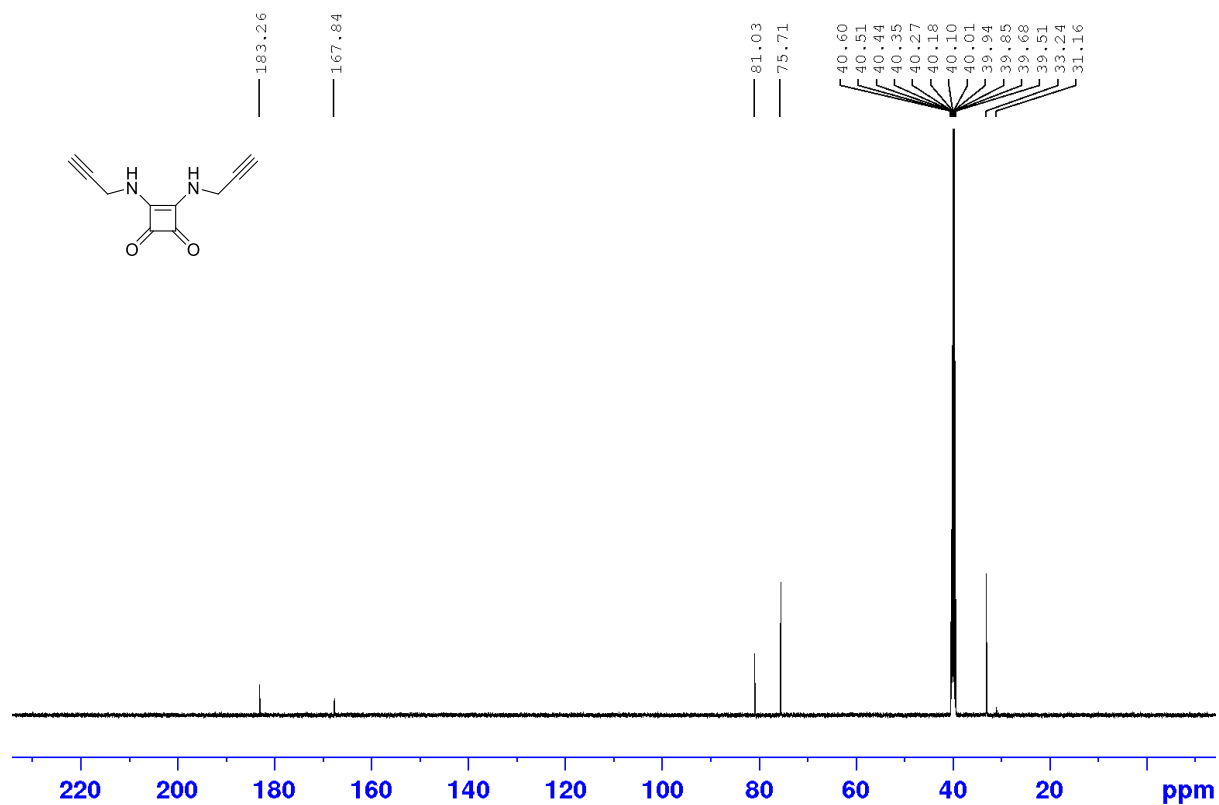
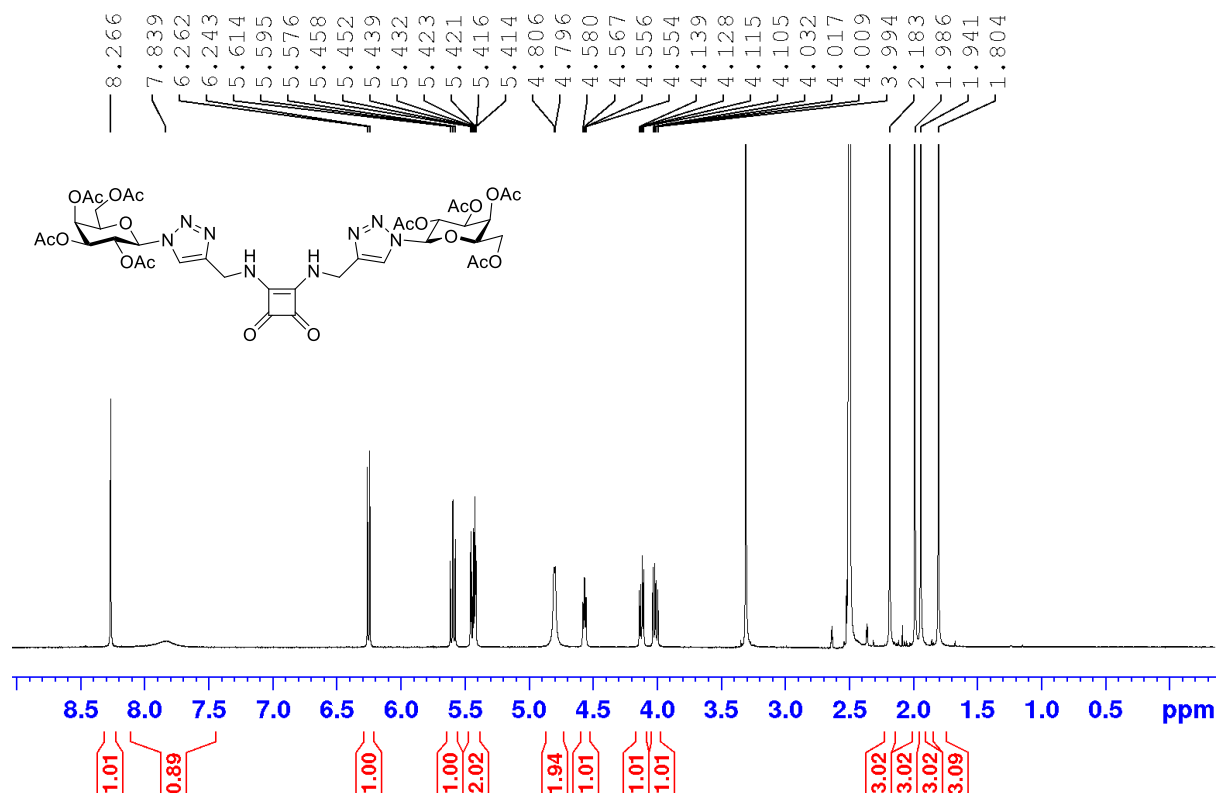


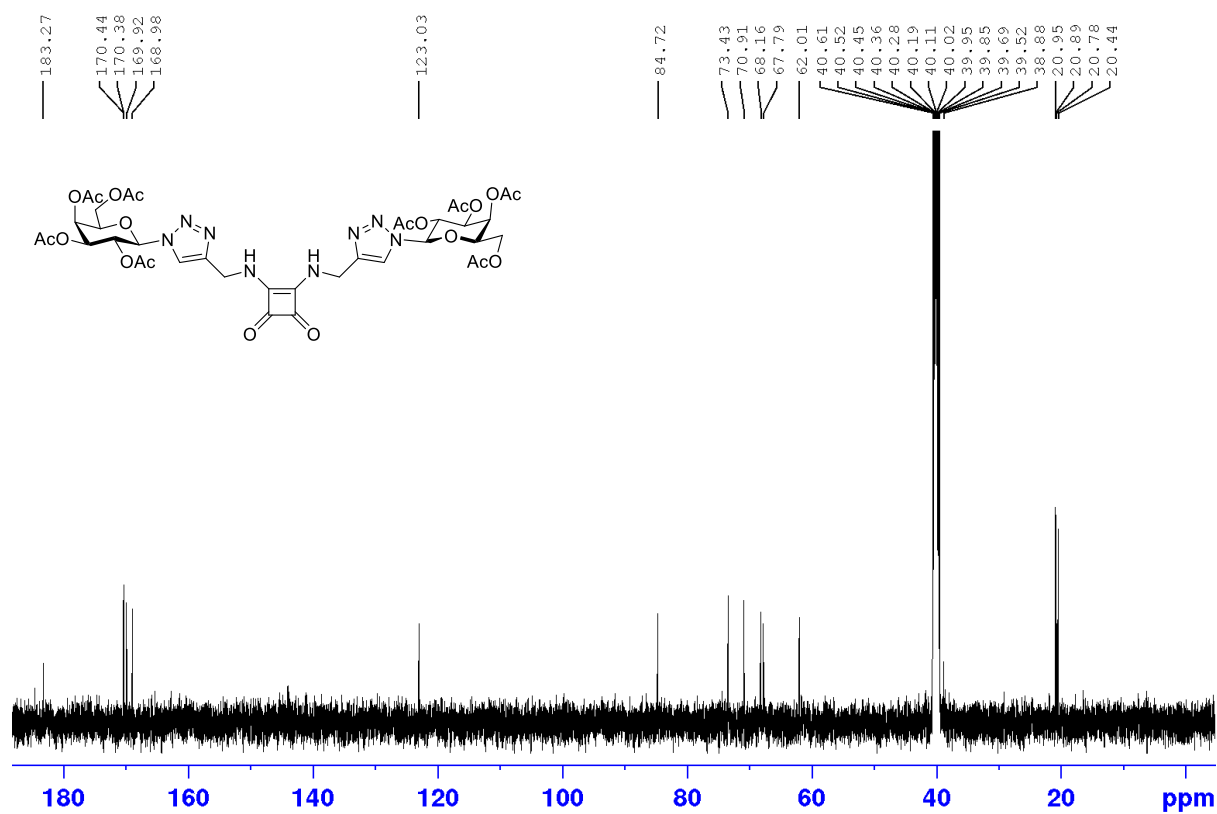
Figure ES16.  $^{13}\text{C}$  NMR spectrum of 3,4-di(prop-2-yn-1-ylamino)cyclobut-3-ene-1,2-dione **1**



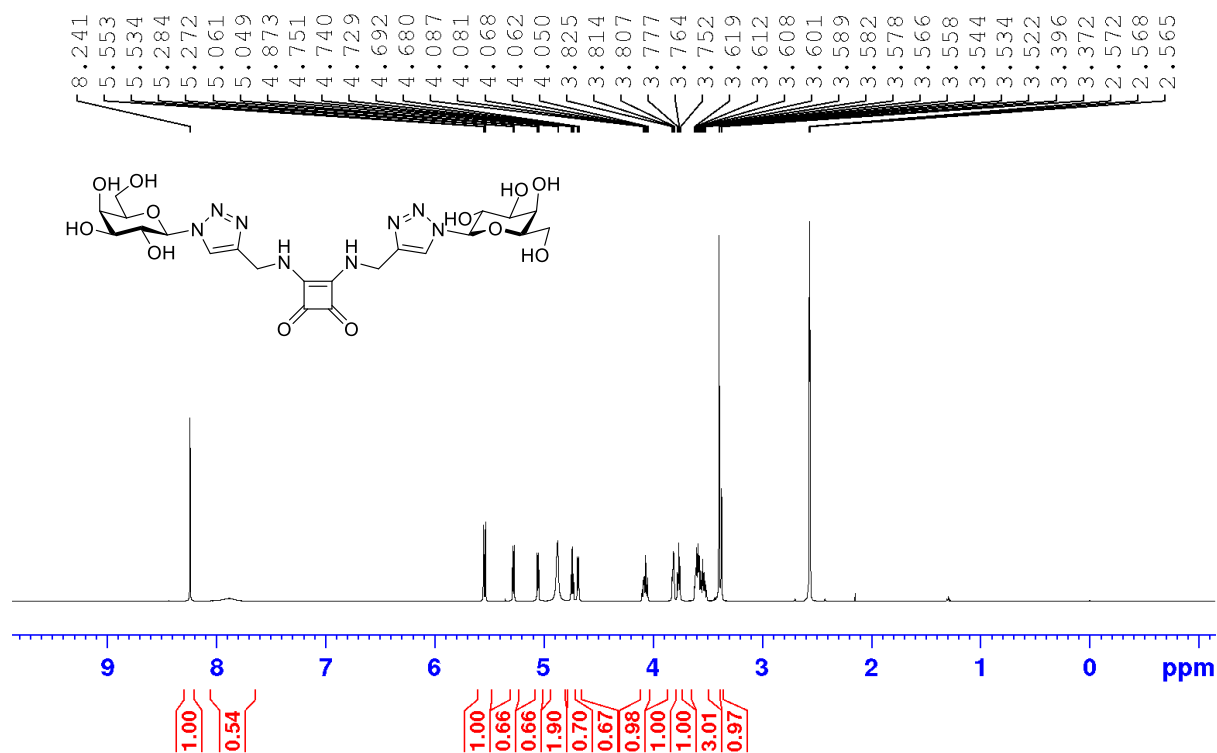
**Figure ES17.**  $^1\text{H}$  NMR spectrum of 3,4-di(2,3,4,6-tetra-*O*-acetyl- $\beta$ -*D*-galactopyranosyl-1,2,3-triazol-4-ylmethylamino)cyclobut-3-ene-1,2-dione **2**



**Figure ES18.**  $^{13}\text{C}$  NMR spectrum of 3,4-di(2,3,4,6-tetra-*O*-acetyl- $\beta$ -*D*-galactopyranosyl-1,2,3-triazol-4-ylmethylamino)cyclobut-3-ene-1,2-dione **2**



**Figure ES19.**  $^1\text{H}$  NMR spectrum of 3,4-di( $\beta$ -D-galactopyranosyl-1,2,3-triazol-4-ylmethylamino)cyclobut-3-ene-1,2-dione **3**



**Figure ES10.**  $^{13}\text{C}$  NMR spectrum of 3,4-di( $\beta$ -D-galactopyranosyl-1,2,3-triazol-4-ylmethylamino)cyclobut-3-ene-1,2-dione **3**

