

Supporting information

High-temperature solvent-free synthesis of low-molecular-weight
organogelators consisting of starch-derived 1,5-anhydro-D-
glucitol coupled with fatty acids

Shiro Komba* and Rika Iwaura

Food Research Institute, National Agriculture and Food Research Organization,
2-1-12, Kannondai, Tsukuba, Ibaraki 305-8642, Japan.

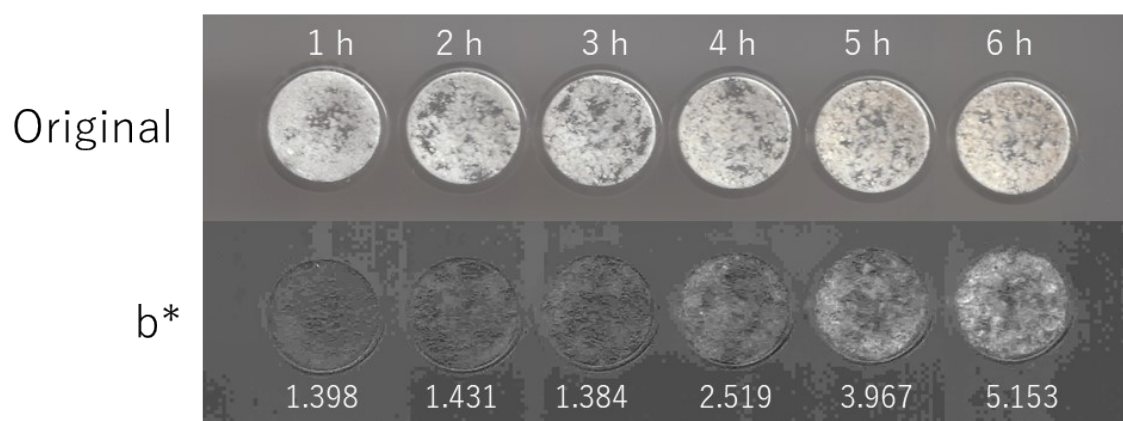


Fig. 1s Scan image and converted b^* stack image for measuring yellowness (whiteness) shown in (C) of Fig. 2.

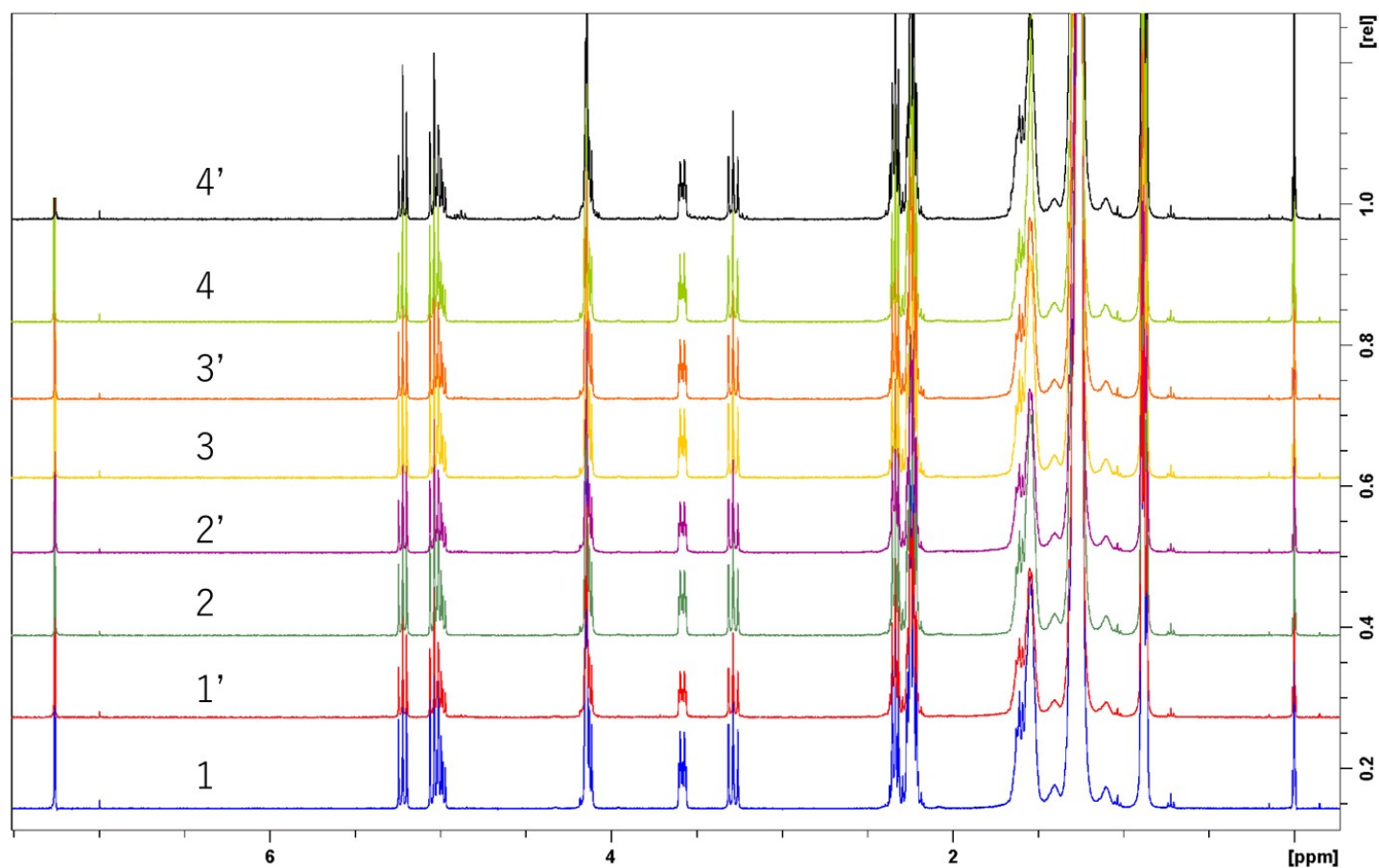


Fig. 2s $^1\text{H-NMR}$ spectra of each sample of C16AG in CDCl_3 ($\text{Me}_4\text{Si} = 0$ ppm). 1: K_2CO_3 , primary crystal (Table 1, entry 1), 1': K_2CO_3 , secondary crystal (Table 1, entry 1'), 2: NaOH , primary crystal (Table 1, entry 2), 2': NaOH , secondary crystal (Table 1, entry 2'), 3: KOH , primary crystal (Table 1, entry 3), 3': KOH , secondary crystal (Table 1, entry 3'), 4: noncatalyst, primary crystal (Table 1, entry 4), 4': noncatalyst, secondary crystal (Table 1, entry 4').

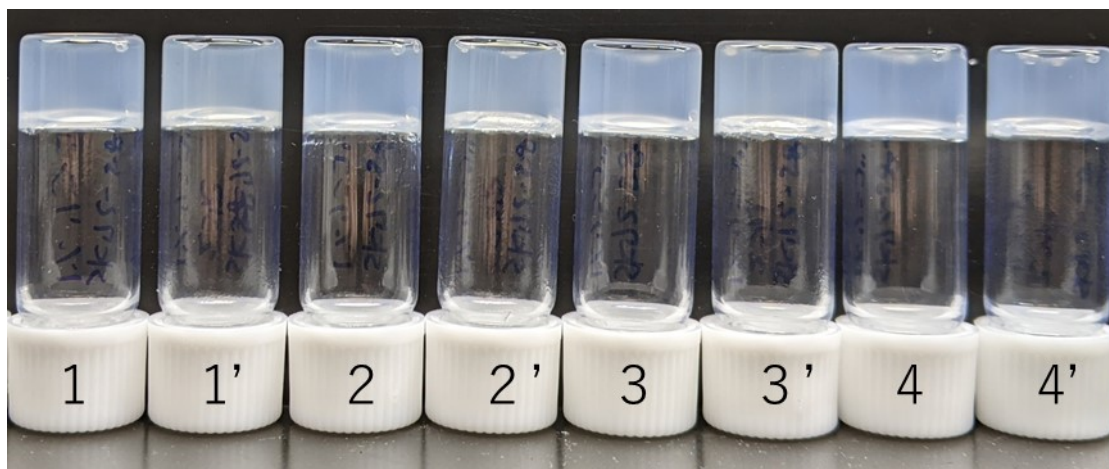


Fig. 3s Appearance of gels (1 wt % gel of each sample in diisostearyl malate) in each entry shown in Table 1. None of the gels flowed down when inverted.

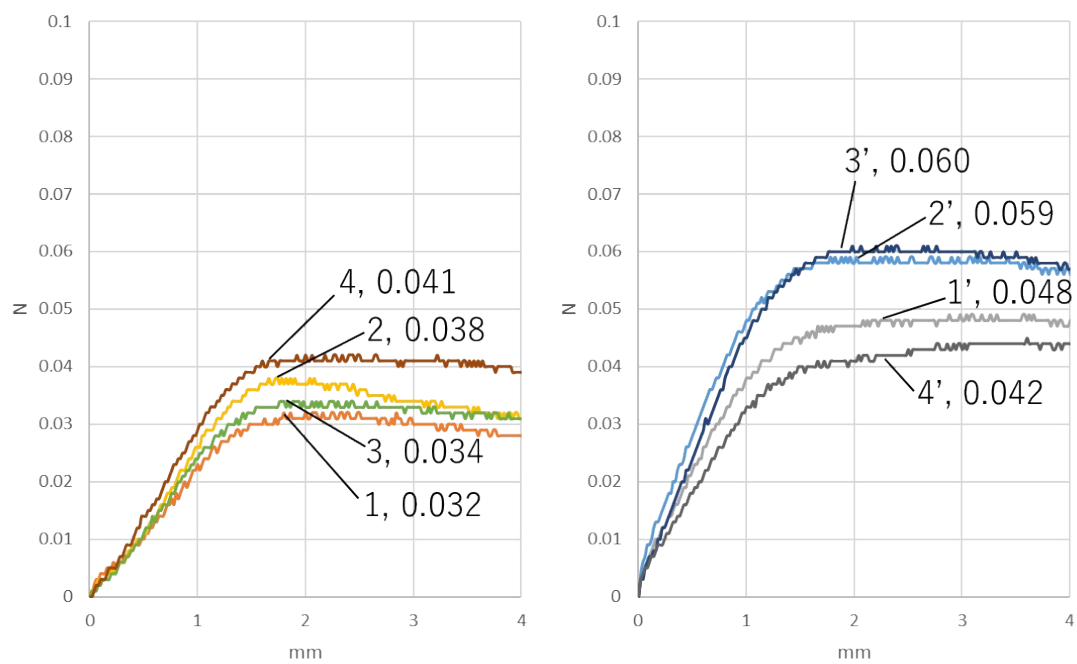


Fig. 4s Gel hardness measurement results for each entry are shown in Table 1.

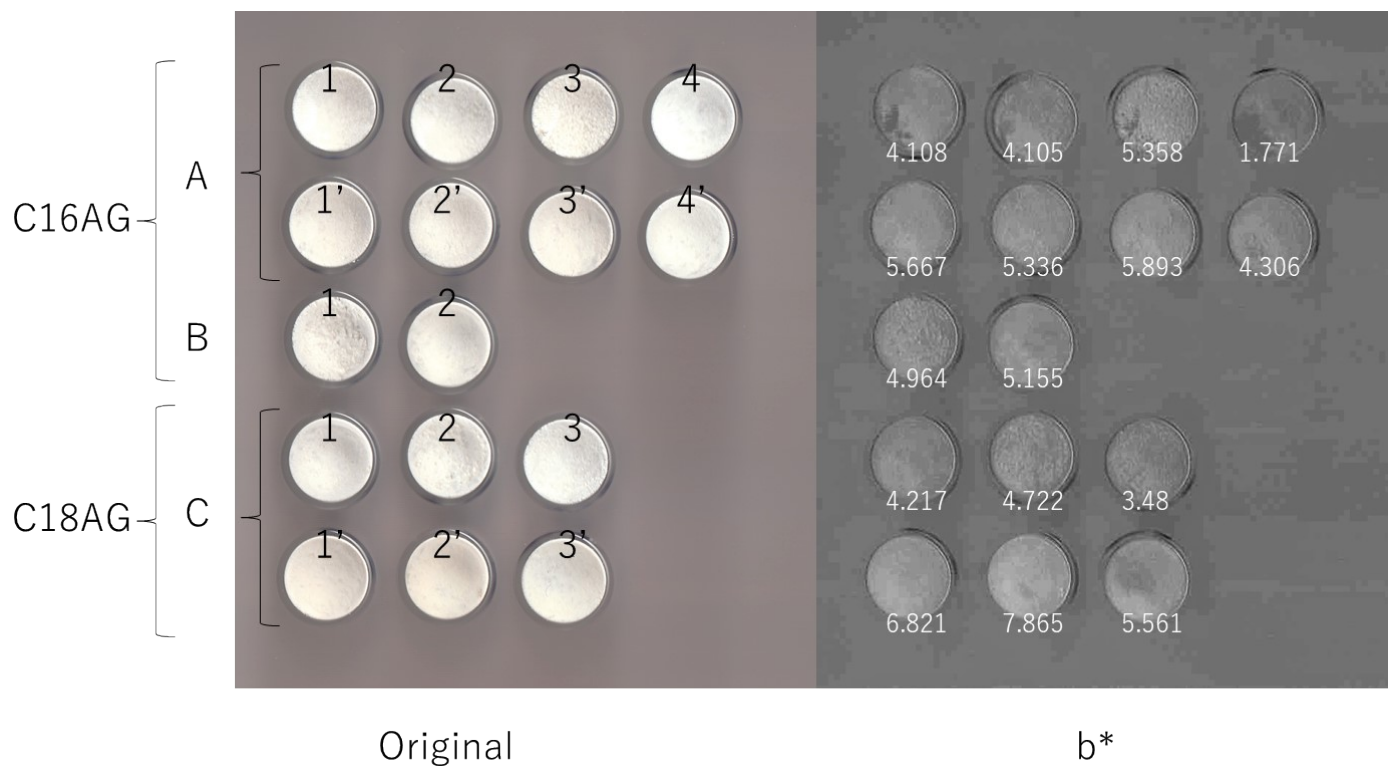


Fig. 5s The scan image and converted b* stack image of each sample. A: Scan image and converted b* stack image for measuring yellowness (whiteness) shown in Table 1. B: Scan image and converted b* stack image for measuring the yellowness (whiteness) of the compounds shown in Fig. 4. C: Scan image and converted b* stack image for measuring yellowness (whiteness) shown in Table 2.

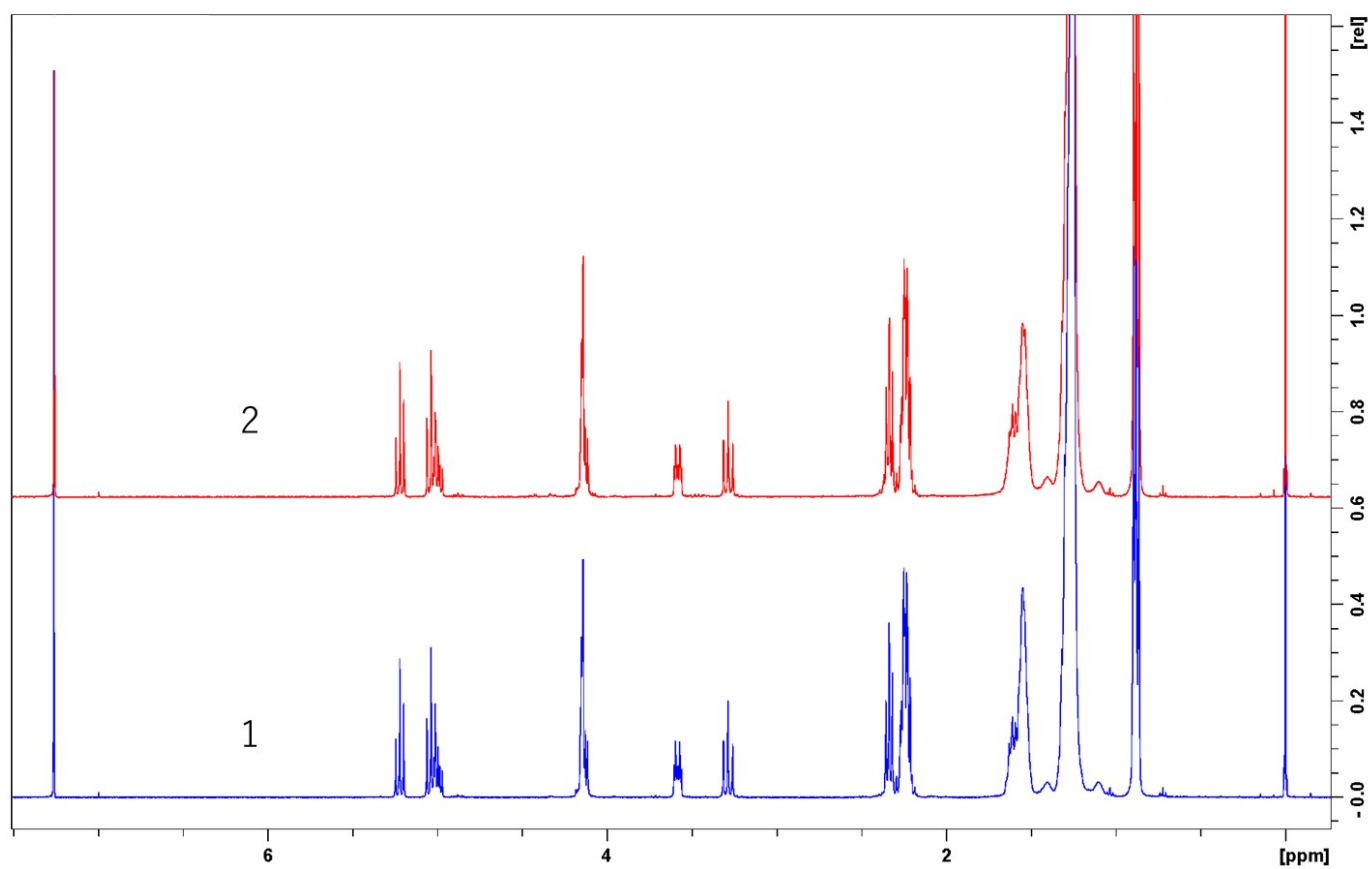


Fig. 6s $^1\text{H-NMR}$ spectra of each sample of C16AG in CDCl_3 ($\text{Me}_4\text{Si} = 0 \text{ ppm}$). 1: K_2CO_3 , (Fig. 4, entry 1), 2: NaOH , (Fig. 4, entry 2).

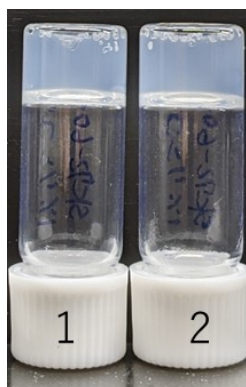


Fig. 7s Appearance of gels (1 wt % gel of each sample in diisostearyl malate) in both entries shown in Fig. 4. Both gels did not flow down when inverted.

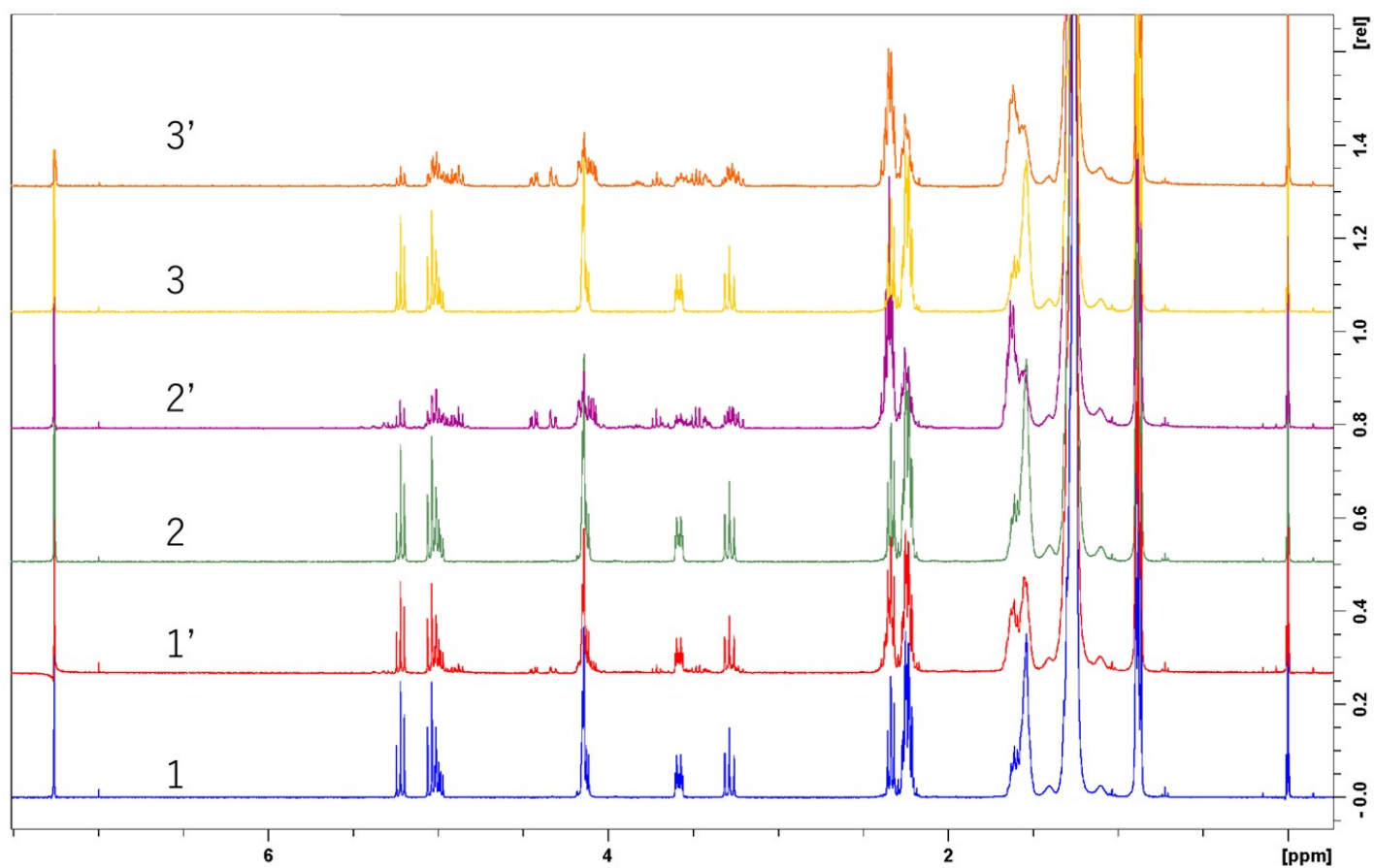


Fig. 8s ^1H -NMR spectra of each sample of C18AG in CDCl_3 ($\text{Me}_4\text{Si} = 0$ ppm). 1: K_2CO_3 , primary crystal (Table 2, entry 1), 1': K_2CO_3 , secondary crystal (Table 2, entry 1'), 2: NaOH , primary crystal (Table 2, entry 2), 2': NaOH , secondary crystal (Table 2, entry 2'), 3: noncatalyst, primary crystal (Table 2, entry 3), 3': noncatalyst, secondary crystal (Table 2, entry 3').

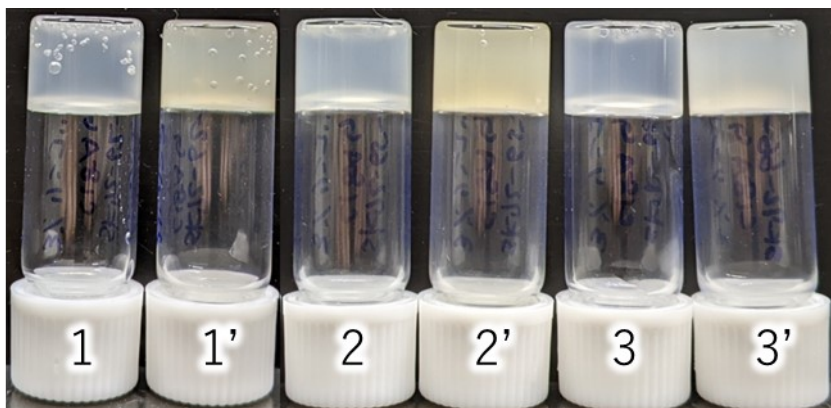


Fig. 9s Appearance of the gels (3 wt % gel of each sample in diisostearyl malate) in each entry shown in Table 2. None of the gels flowed down when inverted.

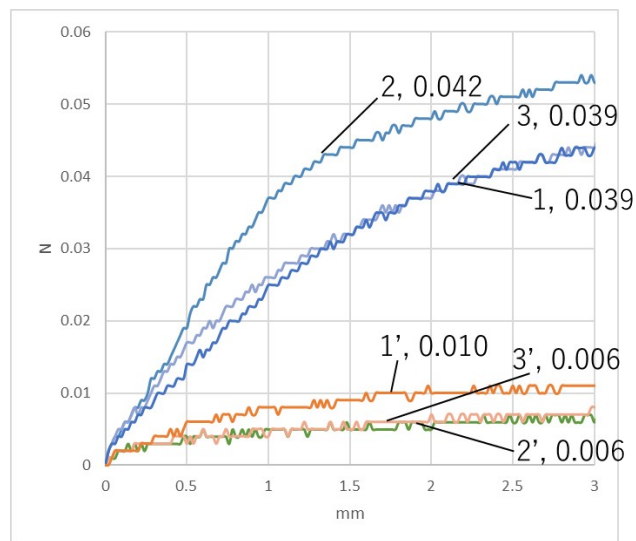


Fig. 10s Gel hardness measurement results for each entry are shown in Table 2.

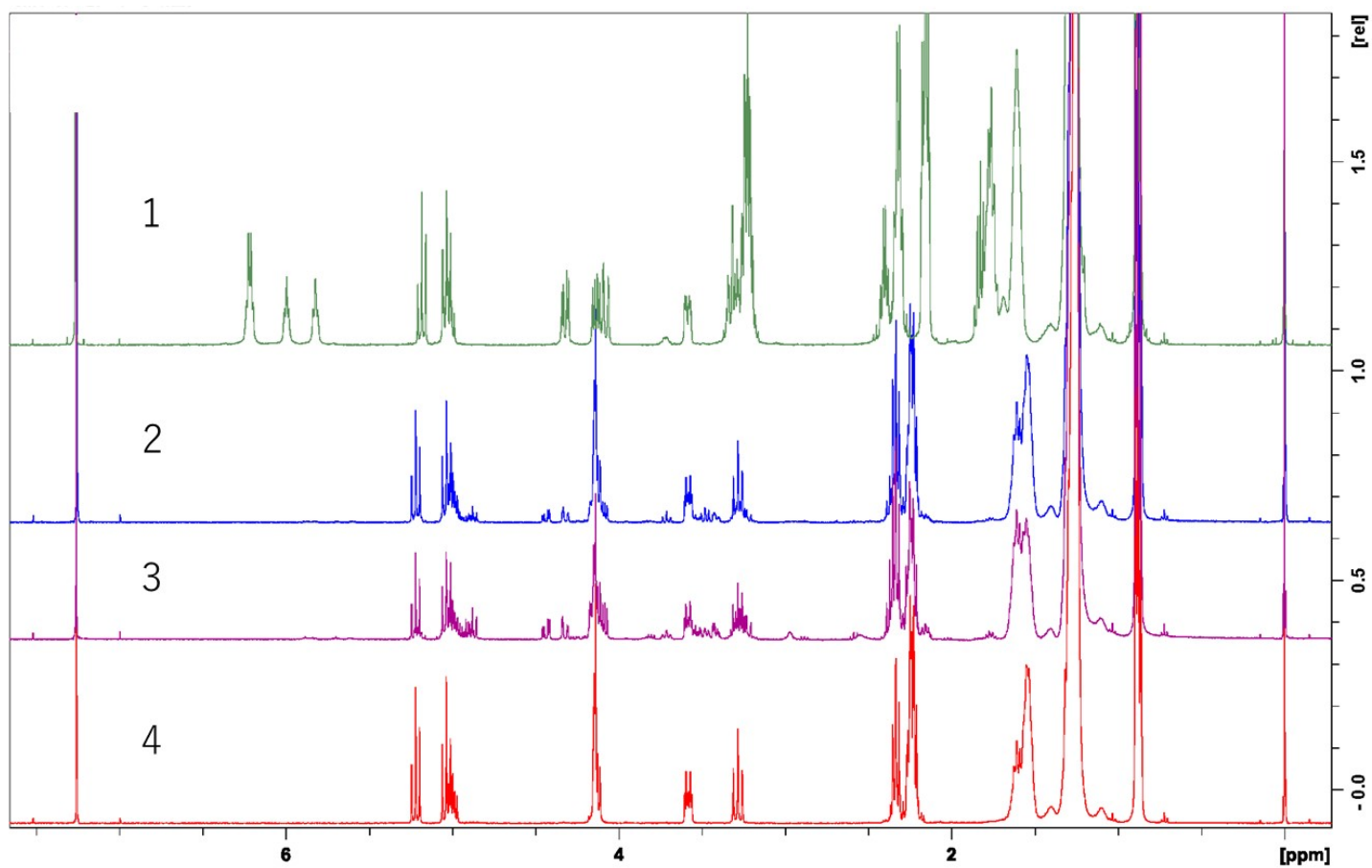


Fig. 11s $^1\text{H-NMR}$ spectra of each sample in CDCl_3 ($\text{Me}_4\text{Si} = 0 \text{ ppm}$). 1: $\text{C}_{16}\text{GABA-AG}$ standard, 2: Reaction products of 1,5-AG and C_{16}GABA by thermal synthesis (with base catalyst), 3: Reaction products of 1,5-AG and C_{16}GABA by thermal synthesis (without base catalyst), 4: C_{16}AG standard. In both entries 2 and 3, C_{16}AG was synthesized instead of $\text{C}_{16}\text{GABA-AG}$.

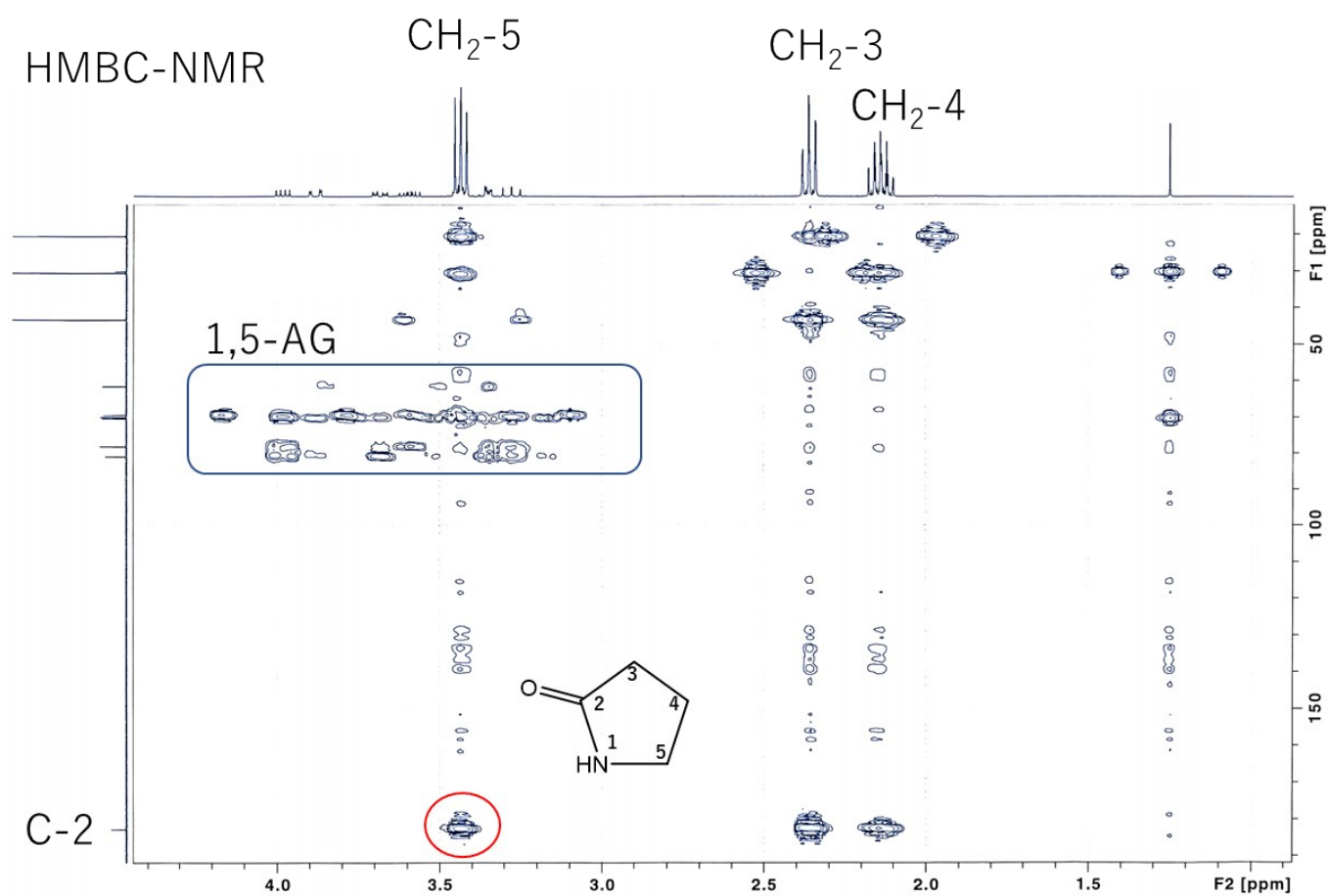


Fig. 12s HMBC-NMR spectra of the reaction product of 1,5-AG and GABA by thermal synthesis in D₂O (*t*BuOH = 1.25 ppm, 30.29 ppm). The cross peaks of CH₂-5 and C-2 indicate intramolecular cyclization.