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Supporting information for article:

Methyl β-lactoside [methyl β-D-galactopyranosyl-(1→4)-β-D-gluco-pyranoside] monohydrate: a solvomorphism study

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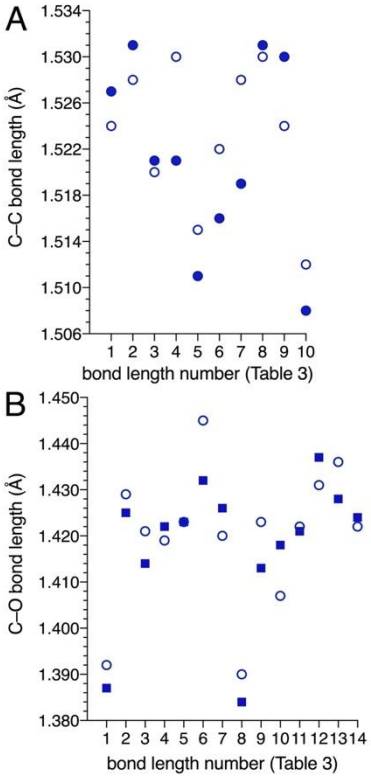


Figure S1. Plots of C–C (A) and C–O (B) bond lengths in (I) (open symbols) and (II) (closed symbols). Bond length numbers were taken from Table 3 in the text.

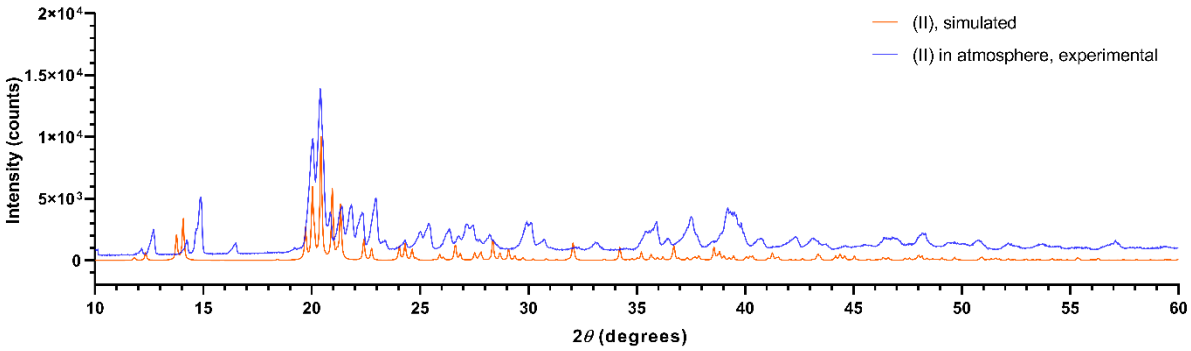


Figure S2. PXRD diffractogram of (II) after exposure to the atmosphere. The simulated PXRD diffractogram of (II) is also shown.

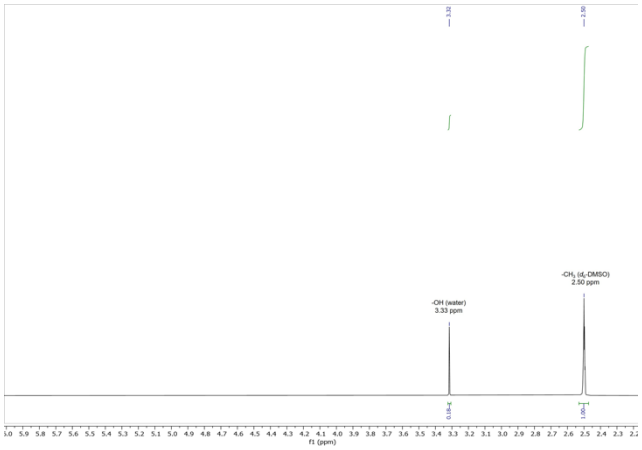
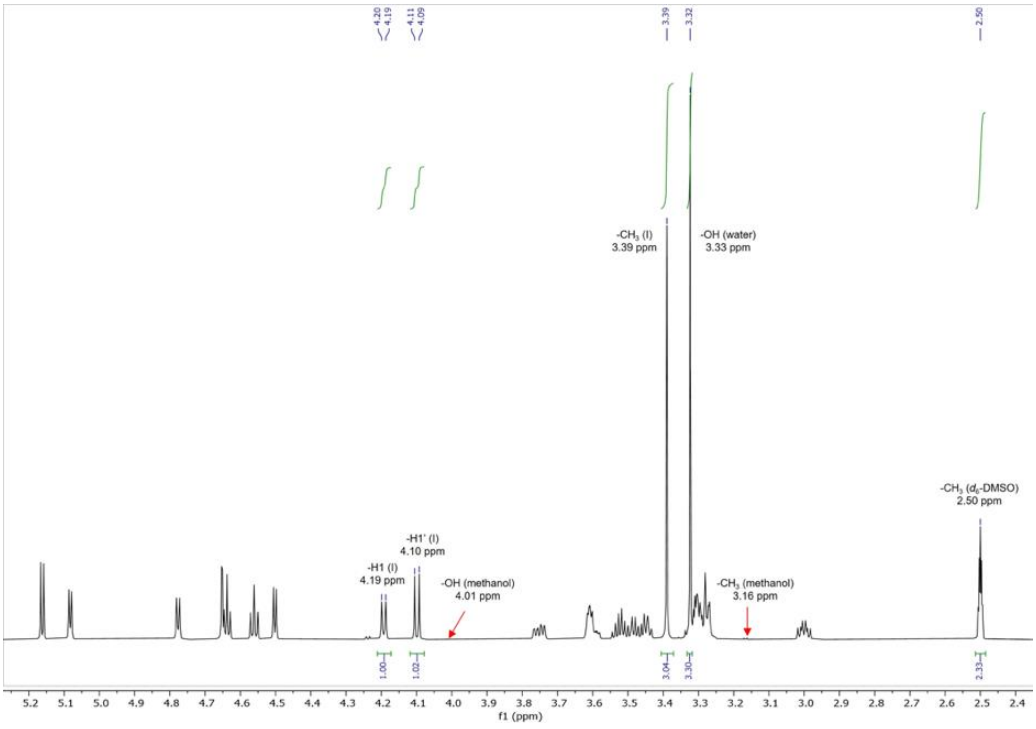


Figure 3a

Figure S3. Solvent newly extracted ampoule, was (600 MHz) to water in the solvent integration of the value of 0.18 methyl signals of set at a value of DMSO-d_6, which was from a sealed glass analyzed by ^{1}H NMR estimate the amount of (Figure 3a). Signal water signal gave a relative to that of the DMSO-d_6, which was 1.00.

The ^{1}H NMR spectrum of (I) (600 MHz; Figure S3b) revealed no methanol in the sample, indicating near quantitative conversion of (II) to (I) after exposure of (II) to the atmosphere for 4 days. Integration of the water signal gave a value of 3.30, and whereas that of the methyl signals of DMSO-d_6 gave a value of 2.33, relative to the integrals of the H1 or H1’ signals, which were set at 1.00. Based on the observations made on neat DMSO-d_6, the contribution to the water signal from solvent is estimated as 2.33 x 0.18 ≈ 0.42, so the remaining water integral, computed as 3.30 – 0.42 = 2.88, derives from the sample of (I). The expected remaining integral is 2.0. The integration difference (0.88) is due to overlap of the water signal with those from other hydrogens in (I) and absorbance of water from the atmosphere during sample preparation.

Figure 3b


4.20
4.19
4.11
4.09
3.39
3.32
2.50
-CH3 (I)
3.39 ppm
-OH (water)
3.33 ppm
-H1 (I)
4.19 ppm
-H1' (I)
4.10 ppm
-OH (methanol)
4.01 ppm
-CH3 (methanol)
3.16 ppm
-CH3 (d6-DMSO)
2.50 ppm
1.00
1.02
3.04
3.30
2.33
f1 (ppm)

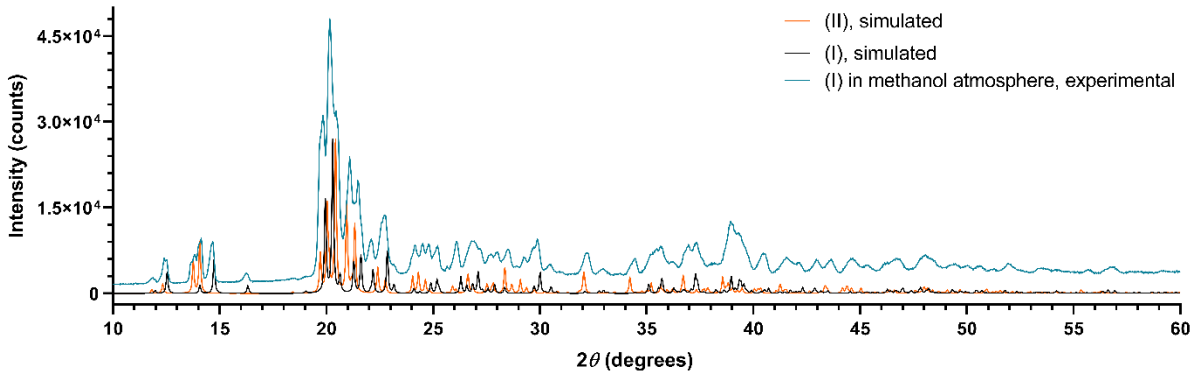


Figure S4. PXRD diffractogram of (I) after exposure to a methanol atmosphere. Simulated PXRD diffractograms of (I) and (II) are also shown.

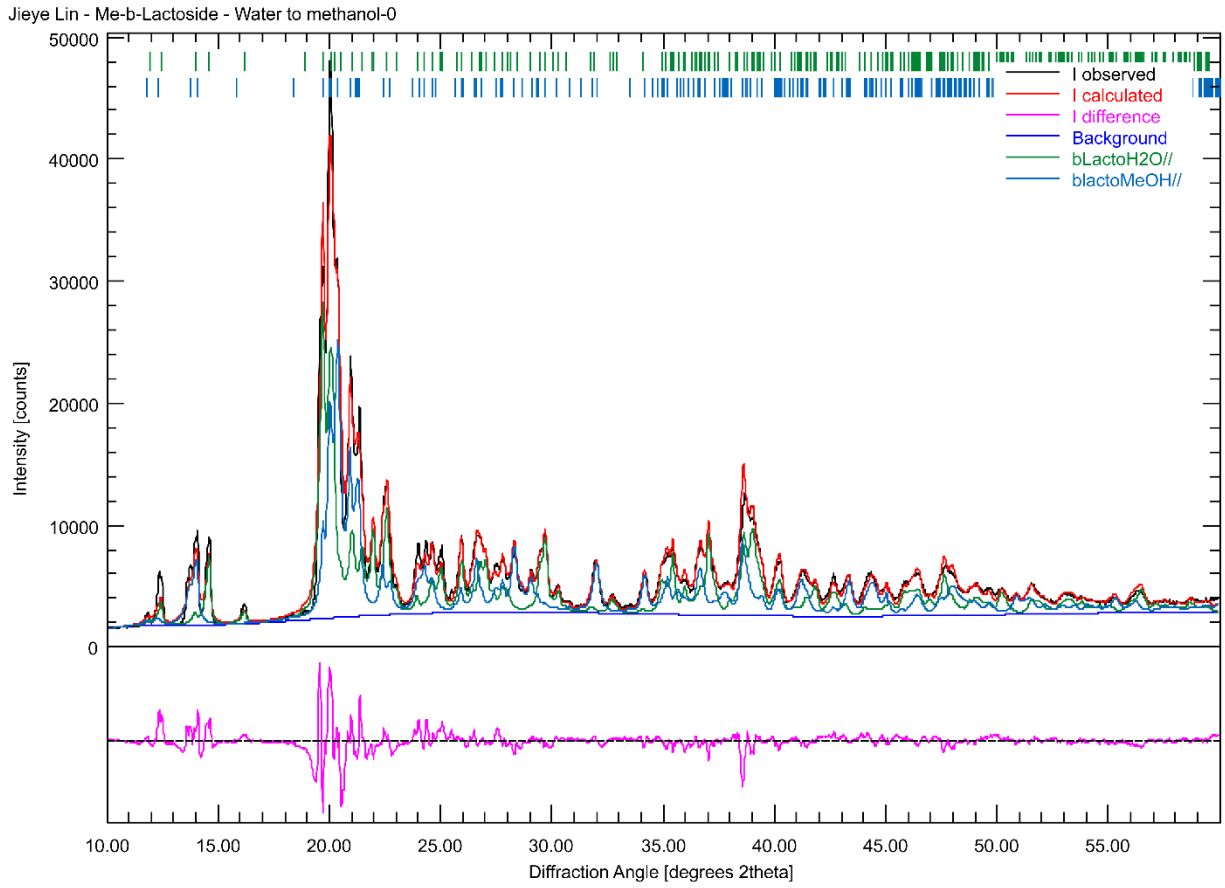


Figure S5. Rietveld refinement of (I) and (II).

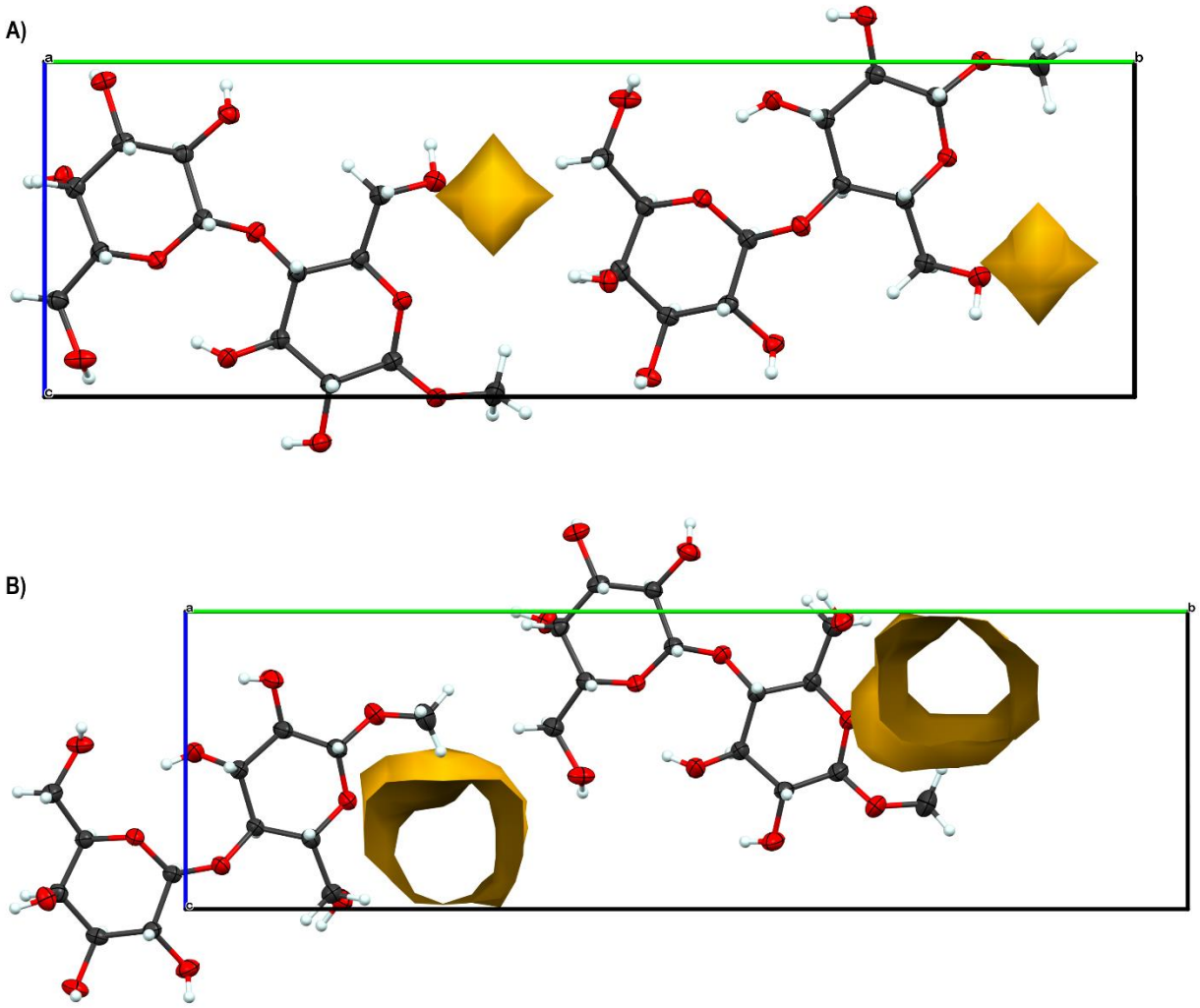


Figure S6. Voids in crystalline lattices of (I) (A) and (II) (B), viewed along the *a*-axis. The probe radii of (I) and (II) are 1.0 Å and 1.2 Å, respectively.

Table S1. Rietveld refinement statistics of (I) and (II).

Rwp	8.24
Rexp	1.41
Chi^2	34.152

GoF	5.844
H_2O : MeOH ratio	0.498(6) : 0.502(6)