

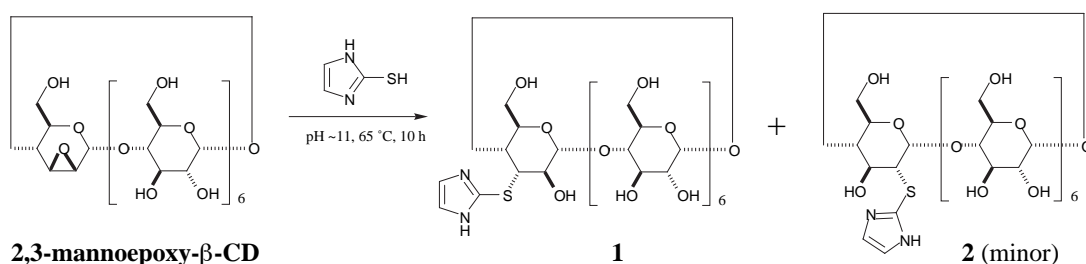
## Crystal structure of mono[3-(2-imidazolylthio)]-*altro*- $\beta$ -cyclodextrin: elliptical distortion of the cavity and unique ‘yin-yang’ stacking

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Compound **1** was prepared from the reaction of the 2,3-mannoepoxy- $\beta$ -cyclodextrin and 2-mercaptoimidazole.



2,3-Mannoepoxy- $\beta$ -cyclodextrin **1** (200 mg, 0.18 mmol) and 2-mercaptoimidazole (27 mg, 0.27 mmol) were dissolved in water (1 mL). After being adjusted to ca. pH 11, the solution was stirred at 65 °C for 10 h. The reaction mixture was then neutralized, filtered through a cellulose acetate membrane (3.0  $\mu$ m), and chromatographed on a reversed-phase Lobar column (Merck LiChroprep® RP-18, Size B). Elution of the column with a gradient from water to 30% aqueous methanol solution afforded the 3<sup>A</sup>-deoxy-3<sup>A</sup>-(2-imidazolylthio)-*altro*- $\beta$ -cyclodextrin (150 mg, 69%). <sup>1</sup>H-NMR:  $\delta$  7.05 (s, 2H, imidazole), 5.07 (d,  $J$  = 3.9 Hz, 1H, H1), 5.04 (d,  $J$  = 3.9 Hz, 1H, H1), 5.03 (d,  $J$  = 3.9 Hz, 1H, H1), 4.99 (d,  $J$  = 3.7 Hz, 1H, H1), 4.96 (d,  $J$  = 3.7 Hz, 1H, H1), 4.95 (d,  $J$  = 3.7 Hz, 1H, H1), 4.89 (d,  $J$  = 6.0 Hz, 1H, H1A), 4.33 (br., 4.04 (br., 1H, H4A), 4.00~3.46 (m, 40 H, the other protons). <sup>13</sup>C-NMR:  $\delta$  137.9 and 124.5 (imidazole); 102.9 (1A), 102.6, 102.4, 102.1, 101.9 and 100.9 (1); 81.6, 81.4, 81.0 and 79.1 (4); 79.0 (4A), ~77.0 (5A), 74.2, 74.0, 73.8, 73.7, 73.3, 72.7, 72.5 and 72.1 (5, 3, 2); 70.6 (2A), 61.4, 61.0 and 60.8 (6), 59.8 (6A), 51.8 (3A). FAB-MAS:  $m/z$  1217 (M + H). The single crystal suitable for X-ray diffraction measurement was obtained by placing the NMR sample solution in a refrigerator.

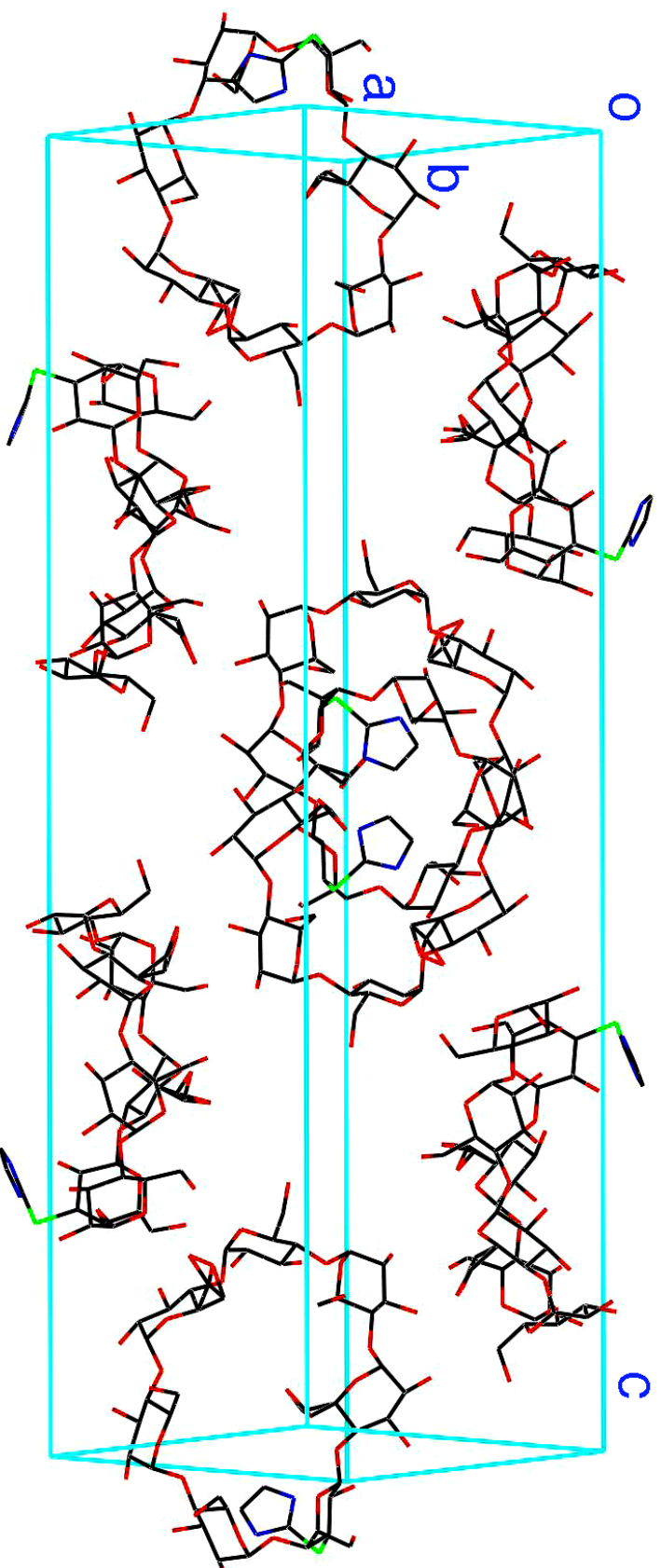


Fig. 3 Packing diagram of **1**, crystal water molecules and hydrogen atoms are omitted for clarity.