Supplementary data for:

Enhanced enantioselectivity of 3-methylcyclohexanone by mixed diol host compounds

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Experimental

Diffraction data for all three compounds were collected on a Bruker DUO APEX II diffractometer[1] using graphite-monochromated Mo-Kα radiation (λ = 0.7103 Å) at 173 (2) K controlled with an oxford Cryostream 700. The intensity data were collected by the standard phi and omega scan techniques, scaled and reduced using SAINT-Plus[2]. The space groups were determined from systematic absences by XPREP[3] and further justified by the refinement results. All three structures were solved using direct methods using SHELXS-97[4] and refined by full-matrix least squares with SHELXL-97[4], refining on F^2. X-Seed[5] was used as a graphical interface. In all cases, the hydrogen atoms bound to carbon atoms were placed at idealized positions and refined as riding atoms with U_{iso} (H) = 1.2 or 1.5 x U_{eq} (C).The hydroxyl hydrogen atoms were located in difference electron density maps and refined with bond length constraints according to a function of O-H versus OO distances based on neutron diffraction data[6].

For structure of **1**, C2A1 and C3A1, C2A2 and C3A2, C7B1 and C7B2 were refined with common isotropic displacement parameters. C1A, C4A1, C4A2, C5A, C6A, C7A1 and C7A2 were refined isotropically. Distances of O1-H1, O2-H2, H1-O1A and H2-O1B were restrained to 0.975(5), 0.965(5), 1.735(5) and 1.806(5) respectively. For the guest at site b, the site occupancy factors (s.o.f.) of atoms C7B1 and C7B2 were obtained by calculating a difference electron density map and assigning their values in proportion to their peak heights. These s.o.f. were subsequently fixed in the final refinement. For the guest at site a, the s.o.f. were refined freely.

For structure of **2**, distances of O1-H1, O2-H2, H1-O1A and H2-O1B were restrained to 0.975(5), 0.965(5), 1.751(5) and 1.836(5) respectively.

For structure of **3**, C2A, C2B, C3A, C3B, C5A, C5B, C6A, C6B, C7A and C7B were refined isotropically. Distance of O1-H1 was restrained to 0.960(5). The s.o.f. of the disordered guest were refined freely.

References

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