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Electronic Supplementary Information

Solvent inclusion in the structural voids of form II carbamazepine: single-crystal X-ray diffraction, NMR spectroscopy and Hirshfeld surface analysis

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Supplementary crystal data. Diffraction data were collected on a Oxford Diffraction Xcalibur S diffractometer at 100(2) K using Mo–Kα radiation. The structure was solved by direct methods using the program SIR92.[1] Data were merged with SORTAV[2] and a θ cut off of 29° was applied. Full-matrix least-squares structure refinement against F^2 was performed using the program CRYSTALS.[3] All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in idealised positions. Hydrogen atoms attached to the nitrogen atom were located on a difference Fourier map. Subsequent to location, the position of all hydrogen atoms was regularised using the X-ray data subject to geometry restraints. Hydrogen atoms were subsequently allowed to ride on their parent atom. Hydrogen atoms for the THF moiety were omitted but taken into account for calculation of density, *F*(000), etc..

1. A. Altomare, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, M. C. Burla, G. Polidori and M. Camalli, *J. Appl. Cryst.*, 1994, **27**, 435.
2. R. H. Blessing, *J. Appl. Cryst.*, 1997, **30**, 421.
3. P. W. Betteridge, J. R. Carruthers, R. I. Cooper, K. Prout and D. J. Watkin, *J. Appl. Cryst.*, 2003, **36**, 1487.

Supplementary disorder data. Two peaks of conspicuous electron density near the high-symmetry site were located on a difference Fourier map. Based on their heights and separations these were assigned to O and C atoms, with the O atom on a $\overline{3}$ axis. The X-ray data is consistent with a THF molecule disordered about the *b*3 Wyckoff position (0,0,½) giving partial occupancy of ½ for the O atom and ⅔ for the C atom. Each site has a refined occupancy of *ca.* 0.6, later confirmed by NMR. During the final refinement occupancies of the O and C atoms were fixed to 0.3 and 0.4, respectively, whilst refining anisotropic thermal parameters, thus giving rise to the solvate stoichiometry CBZ: THF 1 : 0.1. It was not possible to satisfactorily model a complete THF molecule as a rigid group due to the large parameter shifts at this high symmetry site.