Critical role of weak [C–H···O] hydrogen bonds in the assembly of benzo[1,2-d:4,5-d’]bisoxazole cruciforms into supramolecular sheets

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Supporting Information

Data Collection Parameters for X-ray Diffraction

Compound 6. Single crystal X-ray diffraction measurements for compound 6 were performed using a Rigaku SCX-Mini diffractometer equipped with a Mo tube, SHINE optics, and a cryostat set at −50 °C. Crystals were mounted on glass fibers for measurement. Data collection and data integration were completed using Process-Auto.1 Absorption corrections were performed using ABSCOR.2 Solutions were generated by direct methods using SHELXS-97, and refined by full matrix least squares on $F^2$ using SHELXL-97.3 All non-hydrogen atoms were refined anisotropically, with the exception of disordered CH2Cl2. All aromatic hydrogen atoms were generated and refined using a riding model, and methyl hydrogens were identified using a Fourier search and refined using a riding model.

Compound 8. A yellow blade 0.12×0.05×0.02 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 10 seconds per frame using a scan width of 1.0°. Data collection was 96.6% complete to 67.00° in Θ. A total of 28025 reflections were collected covering the indices, $-7 \leq h \leq 8, -20 \leq k \leq 19, -24 \leq l \leq 23$. A total of 8696 reflections were found to be symmetry independent, with an $R_{int}$ of 0.0575. Indexing and unit cell refinement indicated a primitive, triclinic lattice. The space group was found to be $P\overline{1}$ (No. 2). The data were integrated using the Bruker SAINT software program and scaled using the SADABBS software program. Solution by direct methods (SIR-2008) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-97). All hydrogen atoms were...
placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97. SQUEEZE was used to treat the unknown disordered solvent contribution in the crystal and has been noted in the CIF file.

References

2. T. Higashi, ABSCOR; Rigaku Corporation, Tokyo, Japan (1995).
NMR Spectra of New Compounds

$^1$H NMR of Compound 6 (CDCl$_3$, 500 MHz)
$^{13}$C NMR of Compound 6 (CDCl$_3$, 125 MHz)
$^1$H NMR of Compound 8 (CDCl$_3$, 500 MHz)