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

Jaebum Lim,[a] Karolina Osowska,[a] Jacob A. Armitage,[b]
Benjamin R. Martin[b] and Ognjen Š. Miljanić*[a]
[a] Department of Chemistry, University of Houston
136 Fleming Building, Houston, TX 77204-5003
web: www.miljanicgroup.com ▪ email: miljanic@uh.edu ▪ phone: (832) 842-8827
[b] Department of Chemistry, Texas State University—San Marcos
601 University Drive, San Marcos, TX 78666

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 CH_2Cl_2. 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, $-7 \le h \le 8$, $-20 \le k \le 19$, $-24 \le l \le 23$. 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 $P\bar{1}$ (No. 2). The data were integrated using the Bruker SAINT software program and scaled using the SADABS 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

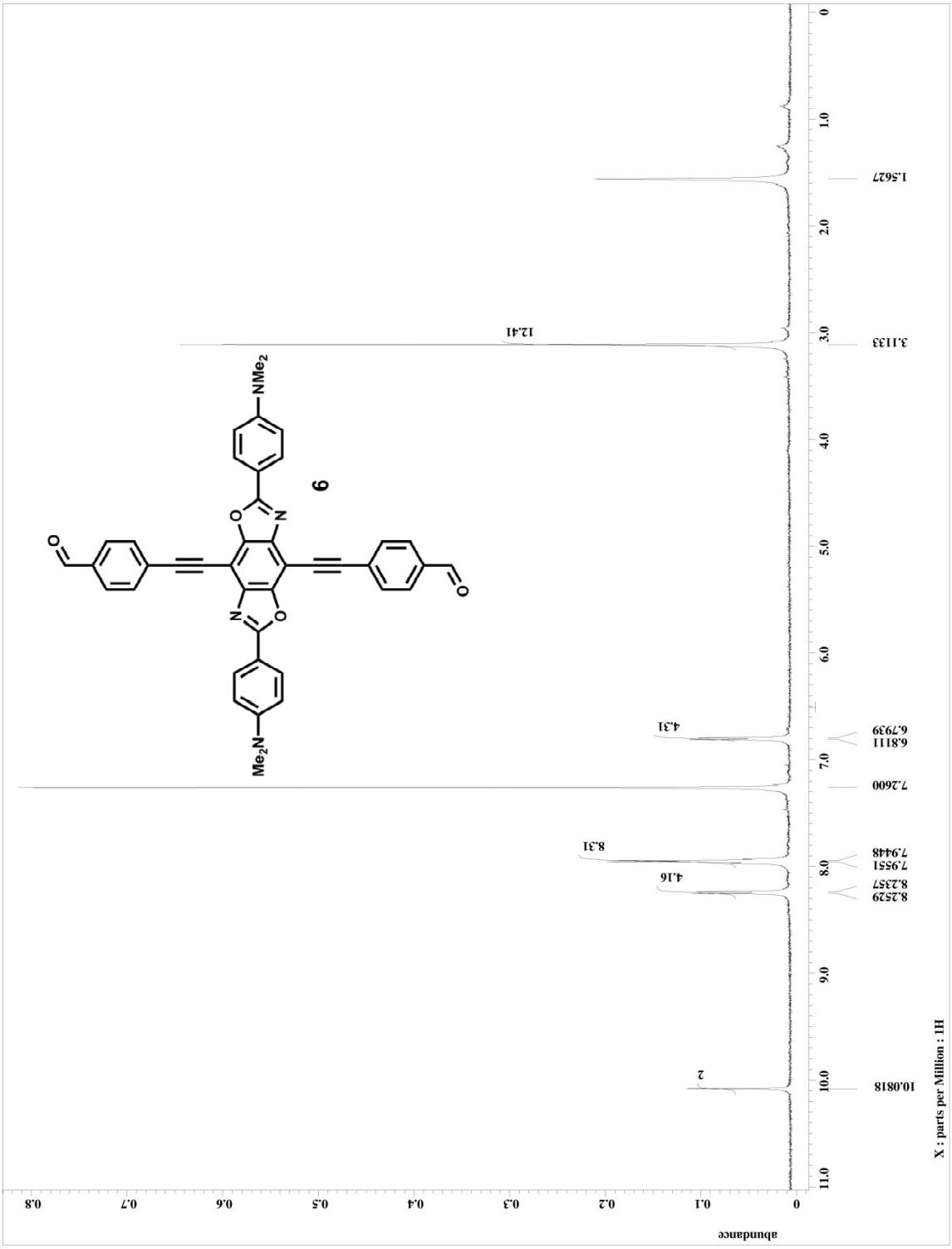
placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97. SQUEEZE was used to treat the unknown disordered solvent contribution in the crystal and has been noted in the CIF file.

References

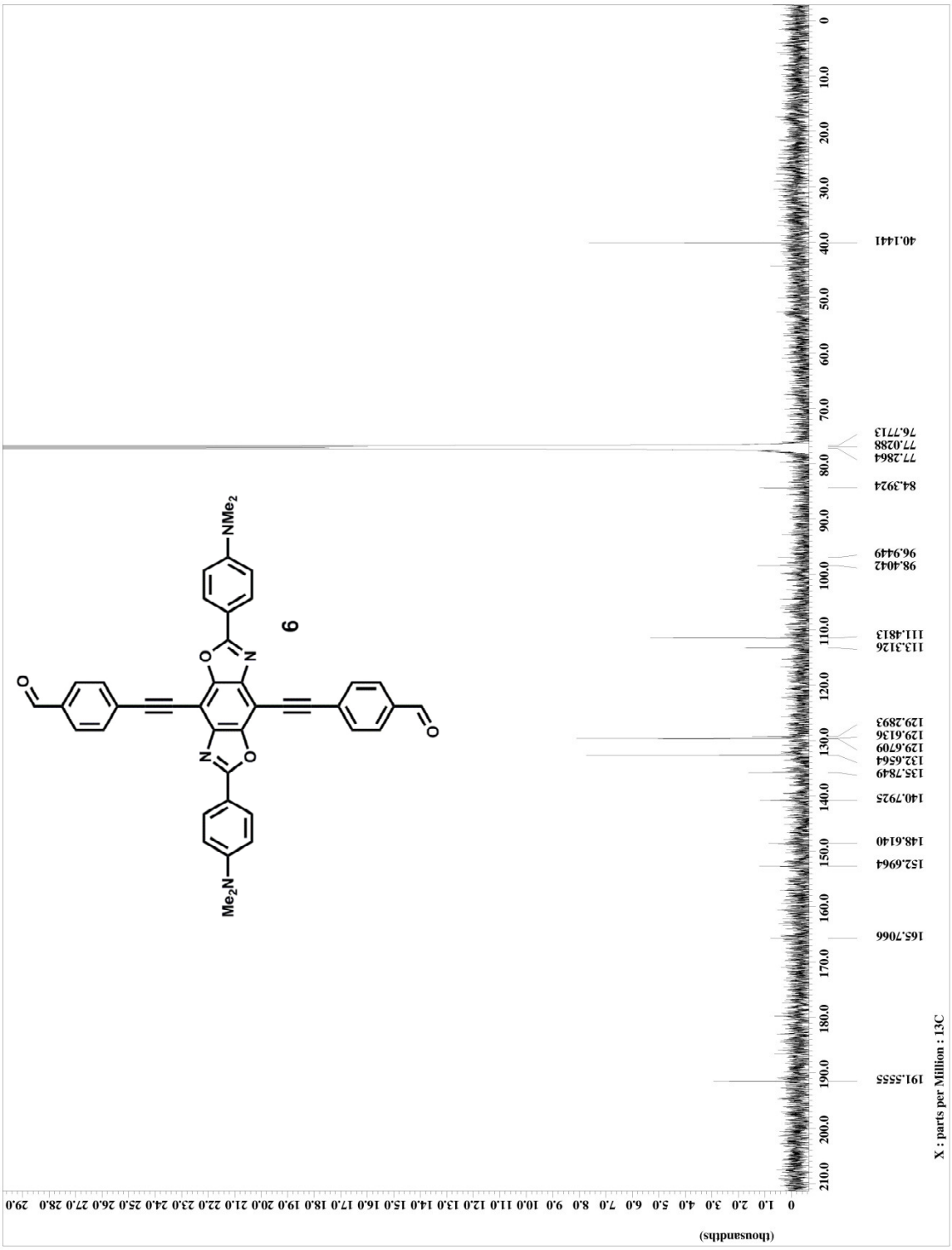
1 Rigaku, *Process-Auto: Automatic Data Acquisition and Processing Package for Imaging Plate and CCD Detectors,* Rigaku Corporation, Tokyo, Japan (2006).
2 T. Higashi, ABSCOR; Rigaku Corporation ,Tokyo, Japan (1995).
3 G. M. Sheldrick, *Acta Cryst.*, 2008, **A64**, 112.

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)