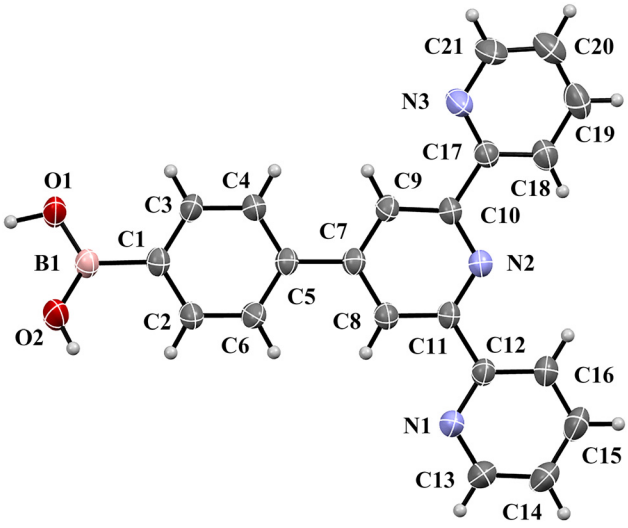
Wenjing Shi and Jun Wang*

The crystal structure of (4-([2,2′:6′,2″-terpyridin]-4′-yl)phenyl)boronic acid, $C_{21}H_{16}BN_3O_2$



https://doi.org/10.1515/ncrs-2025-0158
Received March 30, 2025; accepted May 7, 2025;
published online May 23, 2025

Abstract

$C_{21}H_{16}BN_3O_2$, orthorhombic, $Pna2_1$ (no. 33), a = 28.297(6) Å, b = 6.2978(13) Å, c = 9.6279(16) Å, V = 1715.8(6) Å^3, Z = 4, R_{gt} (F) = 0.0586, wR_{ref} (F^2) = 0.1643, T = 293 K.

CCDC no.: 2372374

The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

1 Source of materials

Dissolve 4-formylphenylboronic acid (1.5 g, 10 mmol) and 2-acetylpyridine (2.65 g, 22 mmol) in 50 mL anhydrous ethanol. With stirring, add NaOH (2.4 g, 60 mmol) and react for 10 h at room temperature. Then, add 37.5 mL ammonia solution and reflux for 20 h. After completion, filter under reduced pressure, wash with chloroform to get white product B–Tpy (4-([2,2′:6′,2″-terpyridin]-4′-yl)phenyl)boronic acid). Dissolve B–Tpy (15 mg, 0.04 mmol) in 10 mL methanol, add 5 mL DMF, seal with parafilm (pierce small holes), and evaporate slowly for 15 days to obtain colorless crystals.

Table 1: Data collection and handling.

Crystal:	Clear light colourless needle
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker Apex2, φ and ω scans
θ_{max}, completeness:	28.4°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	33,713, 4,212, 0.112
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3,485
$N(param)_{refined}$:	252
Programs:	Olex2,[1] Bruker,[2] SHELX,[3] Diamond[4]

2 Experimental details

Using Olex2,[1] the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, withthe d (C–H) = 0.97–0.99 Å, U_{iso} (H) = 1.2 times U_{eq} (C) andU_{iso} (H) = 1.5 times U_{eq}(O).

3 Comment

As a key molecular building block, B–Tpy functions as a versatile platform for designing multifunctional materials through metal-coordination-driven self-assembly.[5–7] This hybrid system uniquely integrates terpyridine's robust metal-binding capability with phenylboronic acid's hydrogen-bonding capacity, enabling precise control over supramolecular architectures via synergistic coordination and non-covalent interactions.[8] However, the lack of crystallographic data for B–Tpy hinders a comprehensive understanding of its aggregation behavior. Resolving its single-crystal structure is therefore critical to elucidating the structure-activity relationships of B–Tpy-based compounds.

***Corresponding author: Jun Wang**, School of Chemistry and Materials, Guizhou Normal University, Guiyang 550025, People's Republic of China, E-mail: wjc@gznu.edu.cn. https://orcid.org/0009-0007-8530-3025
Wenjing Shi, School of Chemistry and Materials, Guizhou Normal University, Guiyang 550025, People's Republic of China

There is one molecule in the asymmetric unit of the title crystal structure. In the crystal, the torsion angles of N1–C12–C11–C8, N3–C17–C10–C9, and C8–C7–C5–C6 are 7.85°, 15.54°, and −2.02°, respectively. The dihedral angles between the pyridine rings containing C11–C8, N1–C12, N3–C17, and the benzene ring containing C5–C6 are 10.65°, 15.54°, and 2.89°, respectively. These angles ensure that the aromatic rings in B–Tpy maintain small angles relative to each other while not being completely coplanar, providing a feasible basis for molecular aggregation. Additionally, in the crystal structure, N1 and N3 are arranged opposite to N2, and O1 and O2 connected to B1 extend to the left and right, respectively, establishing a structural foundation for hydrogen bonding within the crystal. All geometric parameters are in the expected ranges.[9,10]

Acknowledgements: This work was financially supported by the National Natural Science Foundation of China (grant No 22161012), and the Academic New Seedling Foundation Project of Guizhou Normal University (Grant No. Qianshixinmiao-[2022]07).

Conflict of interest: The authors declare no conflicts of interest regarding this article.

Research funding: Academic National Natural Science Foundation of China (grant No 22161012) and the Academic New Seedling Foundation Project of Guizhou Normal University (Grant No. Qianshixinmiao-[2022]07).

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

References

1. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschman, H. *J. Appl. Cryst.* **2009**, *421*, 339–341.
2. Bruker. *APEX2*, *SAINT* and *SADABS*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2012.
3. Sheldrick, G. M. Crystal Structure Refinement with *SHELXL*. *Acta Crystallogr.* **2015**, *C71*, 3–8.
4. Brandenburg, K. *DIAMOND. Visual Crystal Structure Information System. Ver. 4.0*; Crystal Impact: Bonn, Germany, 2015.
5. Li, Y.; Li, Z.; Wu, Y.; Wu, H.; Zhang, H.; Wu, T.; Yuan, C.; Xu, Y.; Zeng, B.; Dai, L. Carbon Particles co-doped with N, B and Fe from metal-organic Supramolecular Polymers for Boosted Oxygen Reduction Performance. *J. Power Sources* **2019**, *412*, 623–630.
6. Li, L.; Zhou, J.; Han, J.; Liu, D.; Qi, M.; Xu, J.; Yin, G.; Chen, T. Finel Manipulating Room Temperature Phosphorescence by Dynamic Lanthanide Coordination Toward multi-level Information Security. *Nat. Commun.* **2024**, *15*, 3846.
7. Wang, J. L.; Li, X.; Lu, X.; Hsieh, I. –H.; Cao, Y.; Moorefield, C. N.; Wesdemiotis, C.; Cheng, S. Z. D.; Newkome, G. R. Stoichiometric self-assembly of shape-persistent 2D Complexes: a Facile Route to a Symmetric Supramacromolecular Spoked Wheel. *J. Am. Chem. Soc.* **2011**, *133*, 11450–11453.
8. Guo, C.; Su, F.; Su, P.; Yu, X.; Li, X. Luminescent Terpyridine-based metallo-supramolecular Systems: from Design to Applications. *Sci. China Chem.* **2023**, *66*, 1940–1962.
9. Fornasari, L.; Mazzaro, R.; Boanini, E.; d'Agostino, S.; Bergamini, G.; Grepioni, F.; Braga, D. Self–Assembly and Exfoliation of a Molecular Solid Based on Cooperative B-N and Hydrogen Bonds. *Crys. Growth Des.* **2018**, *18*, 7259.
10. Sun, Q.; Tang, L.; Zhang, Z.; Zhang, K.; Xie, Z.; Chi, Z.; Zhang, H.; Yang, W. Bright NUV Mechanofluorescence from a Terpyridine-based Pure Organic Crystal. *Chem. Commun.* **2018**, *54*, 94.