Min Zhang, Zhong-Yan Li, Xian-You Yuan and Lin Yuan*

Crystal structure of 2-(4-bromophenyl)-2,3-dihydro-1H-perimidine, C₁₇H₁₃BrN₂

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.36 × 0.33 × 0.30 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.9 mm ⁻¹
Diffractometer, scan mode:	Bruker APEXII, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	24886, 2461, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2105
$N(\text{param})_{\text{refined}}$:	181
Programs:	Bruker programs [9], SHELX [10, 11]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.18042(3)	0.35089(2)	0.33392(2)	0.06296(13)
N1	0.2936(2)	0.68189(15)	0.57191(10)	0.0421(5)
H1	0.3410	0.6309	0.5783	0.051*
N2	0.09235(19)	0.74946(14)	0.53505(10)	0.0437(5)
H2	0.0534	0.7572	0.4975	0.052*
C1	0.2059(2)	0.82136(17)	0.62753(11)	0.0380(5)
C2	0.3044(2)	0.74847(17)	0.62398(11)	0.0375(5)
C3	0.4092(3)	0.74798(19)	0.66788(12)	0.0468(6)
H3	0.4737	0.6997	0.6658	0.056*
C4	0.4185(3)	0.8209(2)	0.71593(13)	0.0521(7)
H4	0.4884	0.8193	0.7462	0.062*
C5	0.3274(3)	0.8936(2)	0.71913(12)	0.0500(7)
H5	0.3371	0.9419	0.7508	0.060*
C6	0.2183(3)	0.89664(18)	0.67493(11)	0.0427(6)
C7	0.1232(3)	0.97190(19)	0.67458(13)	0.0510(7)
H7	0.1298	1.0219	0.7051	0.061*
C8	0.0222(3)	0.9715(2)	0.62974(14)	0.0541(7)
H8	−0.0393	1.0220	0.6300	0.065*
C9	0.0075(3)	0.89749(19)	0.58307(14)	0.0503(6)
H9	−0.0631	0.8989	0.5531	0.060*
C10	0.0979(2)	0.82290(17)	0.58178(12)	0.0408(5)
C11	0.1555(2)	0.65951(17)	0.55290(12)	0.0408(5)
H11	0.1087	0.6318	0.5911	0.049*
C12	0.1564(2)	0.58674(17)	0.49761(12)	0.0380(5)
C13	0.1791(2)	0.49116(18)	0.51340(12)	0.0447(6)
H13	0.1886	0.4737	0.5576	0.054*
C14	0.1880(3)	0.42121(18)	0.46551(13)	0.0458(6)
H14	0.2054	0.3573	0.4768	0.055*
C15	0.1706(2)	0.44749(18)	0.40048(12)	0.0423(6)
C16	0.1458(3)	0.54151(19)	0.38309(12)	0.0492(6)
H16	0.1326	0.5581	0.3390	0.059*
C17	0.1405(3)	0.61125(19)	0.43183(12)	0.0458(6)
H17	0.1260	0.6754	0.4203	0.055*

<https://doi.org/10.1515/ncrs-2018-0113>

Received March 20, 2018; accepted June 29, 2018; available online July 23, 2018

Abstract

C₁₇H₁₃BrN₂, orthorhombic, *Pbca* (no. 61), $a = 9.995(4)$ Å, $b = 13.845(5)$ Å, $c = 20.213(7)$ Å, $V = 2797.2(18)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0304$, $wR_{\text{ref}}(F^2) = 0.0784$, $T = 296(2)$ K.

CCDC no.: 1852427

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of naphthalene-1,8-diamine (1.58 g, 10 mmol) and 4-bromobenzaldehyde (1.85 g, 10 mmol), in anhydrous ethanol (50 mL) was refluxed at 353 K for about 2 h. Then the solvent was removed under reduced pressure. The resulting residue was purified by recrystallization from ethanol to obtain a crystal (yield 2.86 g, 88%).

*Corresponding author: Lin Yuan, College of Chemistry and Bioengineering, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, P.R. China, e-mail: yuanxianzhangmin@163.com

Min Zhang, Zhong-Yan Li and Xian-You Yuan: College of Chemistry and Bioengineering, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, P.R. China

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the hydrogen atoms were set to 1.2 $U_{\text{eq}}(\text{C})$.

Comment

Perimidines have drawn extensive examinations of many researchers for a long time, because they exhibit a diverse range of biological activities [1–3]. There are several preparative methods for the synthesis of perimidine derivatives [4–7]. Herein, we report on a novel perimidine compound which was synthesized and characterized by single-crystal X-ray diffraction.

In the structure, the bond angles of N(1)–C(11)–N(2), N(1)–C(11)–C(12) and N(2)–C(11)–C(12) are 107.17(19), 109.31(19) and 113.3(2) in turn, which indicates the sp³ hybridization state of C(11) atom and the new-formed three-membered heterocyclic ring is nonplanar. All bond lengths and angles are in the expected ranges [8].

Acknowledgements: This work was financially supported by the Natural Science Foundation of Hunan Province of China (2017JJ3093).

References

1. Bu, X.; Deady, L. W.; Finlay, G. J.; Baguley, B. C.; Denny, W. A.: Synthesis and cytotoxic activity of 7-oxo-7H-dibenz[*f,i*]isoquinoline and 7-oxo-7H-benzo[*e*]perimidine derivatives. *J. Med. Chem.* **44** (2001) 2004–2014.
2. Ye, J.; Sun, X. X.; Qiu, S. Y.; Hu, A. X.: Synthesis, crystal structure and fungicidal activity of N-(4-tert-butyl)-5-(1,2,4-triazol-1-yl)thiazol-2-yl) propionamide. *Chin. J. Struct. Chem.* **3** (2014) C429–C433.
3. Fu, C. R.; Pei, J.; Ning, Y.; Liu, M.; Shan, P. C.; Liu, J.; Li, Y. Q.; Hu, F. Z.; Zhu, Y. Q.; Yang, H. Z.; Zou, X. M.: Synthesis and insecticidal activities of novel pyrazole oxime ether derivatives with different substituted pyridyl rings. *Pest Manag. Sci.* **70** (2013) C1207–C1214.
4. Hendrickson, J. B.; Hussoin, M. S.: Seeking the ideal dehydrating reagent. *J. Org. Chem.* **52** (1987) 4137–4139.
5. Li, Z. Y.; Yuan, L.; Yuan, X. Y.; Zhang, M.: Crystal structure of 2-(4-fluorophenyl)-1,3-dimethyl-1H-perimidin-3-ium iodide, C₁₉H₁₆ClIN₂. *Z. Kristallogr. NCS.* **232** (2017) 821–822.
6. Vanden, E. J. J.; Delfosse, F.; Mayence, A.; Haverbeke, Y. V.: Old reagents, new results-aromatization of Hantzsch 1,4-dihydropyridines with manganese-dioxide and 2,3-dichloro-5,6-dicyano-1,4-benzoquinone. *Tetrahedron* **51** (1995) 5813–5818.
7. Deady, L. W.; Rodemann, T. J.: Synthesis of perimidine and fused perimidine derivatives from reaction of 1,8-naphthalenediamine with an iminoisocoumarin. *J. Heterocycl. Chem.* **35** (1998) 1417–1419.
8. Li, Z.-Y.; Zhang, M.; Yuan, X.-Y.; Yuan, L.: Crystal structure of 2-(4-chlorophenyl)-2,3-dihydro-1H-perimidine, C₁₇H₁₃ClN₂. *Z. Kristallogr. NCS* **232** (2018) 263–264.
9. Bruker. SAINT, APEX2 and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA (2009).
10. Sheldrick, G. M.: SHELXS-97: program for the solution of crystal structures. University of Göttingen, Germany (1997).
11. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.