Long-Sheng Zou* and Xian-You Yuan

Crystal structure of 2-(4-bromophenyl)-1,3-dimethyl-1*H*-perimidin-3-ium iodide, C₁₉H₁₆BrIN₂

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.38 × 0.34 × 0.30 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.12 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	26.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18407, 3616, 0.018
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3262
$N(\text{param})_{\text{refined}}$:	210
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.84550(2)	0.18172(2)	0.13134(2)	0.04970(6)
Br1	0.22252(4)	0.48680(2)	0.41402(2)	0.05925(8)
N1	0.3063(2)	0.12110(10)	0.33821(13)	0.0393(4)
C1	0.2763(3)	0.00601(11)	0.42360(17)	0.0384(4)
N2	0.2413(2)	0.11962(9)	0.51234(13)	0.0370(4)
C2	0.3030(3)	0.04431(12)	0.33151(16)	0.0404(5)
C3	0.3262(4)	0.00870(15)	0.2400(2)	0.0585(6)
H3	0.341254	0.034414	0.179190	0.070*
C4	0.3269(4)	−0.06717(16)	0.2397(2)	0.0682(8)
H4	0.343311	−0.091385	0.177785	0.082*
C5	0.3045(3)	−0.10600(14)	0.3264(3)	0.0627(7)
H5	0.306707	−0.156236	0.323528	0.075*
C6	0.2775(3)	−0.07069(12)	0.4223(2)	0.0475(5)
C7	0.2479(3)	−0.10779(14)	0.5153(2)	0.0585(7)
H7	0.245956	−0.158066	0.515796	0.070*
C8	0.2222(4)	−0.07086(15)	0.6040(2)	0.0604(7)
H8	0.204279	−0.096365	0.664623	0.072*
C9	0.2222(3)	0.00526(14)	0.60629(19)	0.0509(5)
H9	0.205370	0.029781	0.667916	0.061*
C10	0.2471(3)	0.04273(11)	0.51670(16)	0.0368(4)
C11	0.1932(3)	0.15803(13)	0.60708(18)	0.0492(5)
H11A	0.292674	0.154709	0.658566	0.074*
H11B	0.086279	0.136439	0.633363	0.074*
H11C	0.169251	0.207932	0.590976	0.074*
C12	0.2706(3)	0.15538(11)	0.42504(15)	0.0358(4)
C13	0.3585(4)	0.16118(14)	0.24536(19)	0.0542(6)
H13A	0.261443	0.158328	0.192315	0.081*
H13B	0.467201	0.140338	0.220183	0.081*
H13C	0.380738	0.210909	0.263312	0.081*

<https://doi.org/10.1515/ncrs-2018-0301>

Received August 9, 2018; accepted October 8, 2018; available online October 17, 2018

Abstract

C₁₉H₁₆BrIN₂, monoclinic, $P2_1/c$ (no. 14), $a = 7.3409(17)$ Å, $b = 18.494(4)$ Å, $c = 12.905(3)$ Å, $\beta = 93.372(3)^\circ$, $V = 1749.0(7)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0205$, $wR_{\text{ref}}(F^2) = 0.0512$, $T = 296(2)$ K.

CCDC no.: 1871996

The asymmetric unit of the title 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.

*Corresponding author: Long-Sheng Zou, Department of Building Environment and Energy Engineering, Guilin University of Aerospace Technology, Guilin Guangxi 541004, P.R. China, e-mail: yuanxianzhangmin@163.com

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

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C14	0.2615(3)	0.23594(11)	0.42463(16)	0.0393(4)
C15	0.1098(3)	0.26924(13)	0.37634(18)	0.0478(5)
H15	0.015816	0.241578	0.345338	0.057*
C16	0.0995(3)	0.34381(13)	0.37474(19)	0.0493(5)
H16	−0.002394	0.366569	0.343402	0.059*
C17	0.2400(3)	0.38436(12)	0.41947(17)	0.0439(5)
C18	0.3919(3)	0.35184(13)	0.46772(18)	0.0483(5)
H18	0.486208	0.379742	0.497796	0.058*
C19	0.4014(3)	0.27727(13)	0.47059(18)	0.0476(5)
H19	0.502114	0.254705	0.503527	0.057*

Source of material

Iodomethane (3.02 g, 0.021 mol) was added dropwise to a solution of 2-(4-bromophenyl)-1-methyl-1*H*-perimidine (3.37 g, 0.01 mol) in dimethylformamide (20 mL) at 373 K. The mixture was heated for 8 h, giving a yellow precipitate. After cooling, the yellow solid was filtered, washed with anhydrous ethanol and dried under vacuum to give the desired salt 2-(4-bromophenyl)-1,3-dimethyl-1*H*-perimidin-3-ium iodide (3.35 g, 70%). Light yellow needle crystals were obtained by recrystallized from anhydrous ethanol.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The methyl groups were idealized and refined using rigid groups allowed to rotate about the N–C bond (AFIX 137 option of the SHELXL program). The *U*_{iso} values of the hydrogen atoms of methyl groups were set to 1.5*U*_{eq}(C) and those of all other hydrogen atoms were set to 1.2*U*_{eq}(C).

Discussion

Perimidines have drawn extensive examinations for a long time, because they exhibit a diverse range of biological activities [4–6]. There are several preparative methods for the synthesis of perimidine derivatives [7–12]. And some structures are already reported [7, 8, 10]. Herein, a novel perimidine compound was synthesized and characterized by single-crystal X-ray diffraction.

In the title structure, the bond angles of N(1)–C(12)–N(2), N(1)–C(12)–C(14) and N(2)–C(12)–C(14) are 121.72(19),

119.03(18) and 119.25(18) in turn, which indicates the sp² hybridization state of C12 and the new-formed heterocyclic ring is almost planar. Geometric parameters are in perfect accord with those in similar structure [7, 8, 10].

Acknowledgements: This work was financially supported by the Research Foundation for PhD of Guilin University of Aerospace Technology.

References

1. Bruker: APEX3, SAINT-Plus, XPREP. Bruker AXS Inc., Madison, WI, USA (2016).
2. Sheldrick, G. M.: SHELXT – Integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
4. Bu, X.; Deady, L. W.; Finlay, G. J.; Baguley, B. C.; Denny, W. A.: Synthesis and cytotoxic activity of 7-oxo-7*H*-dibenz[*f,i*]isoquinoline and 7-oxo-7*H*-benzo[*e*]perimidine derivatives. *J. Med. Chem.* **44** (2001) 2004–2014.
5. Farghaly, T. A.; Mahmoud, H. K.: Synthesis, zautomeric structures, and antitumor activity of new perimidines. *Arch. Pharm.* **346** (2013) 392–402.
6. 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) 1207–C1214.
7. Yuan, L.; Li, Z. Y.; Yuan, X. Y.; Zhang, M.; Liu, F.: Crystal structure of 1,3-dimethyl-2-phenyl-1*H*-perimidin-3-ium iodide, C₁₉H₁₇IN₂. *Z. Kristallogr. NCS.* **232** (2017) 541–543.
8. Zhang, M.; Yuan, L.; Yuan, X. Y.; Li, Z. Y.: Crystal structure of 4-(1,3-dimethyl-2,3-dihydro-1*H*-perimidin-2-yl)benzonitrile, C₂₀H₁₇N₃. *Z. Kristallogr. NCS.* **232** (2017) 267–268.
9. Hendrickson, J. B.; Hussoin, M. S.: Seeking the ideal dehydrating reagent. *J. Org. Chem.* **52** (1987) 4137–4139.
10. Li, Z. Y.; Yuan, L.; Yuan, X. Y.; Zhang, M.: Crystal structure of 2-(4-chlorophenyl)-1,3-dimethyl-1*H*-perimidin-3-ium iodide, C₁₉H₁₆ClIN₂. *Z. Kristallogr. NCS.* **232** (2017) 821–822.
11. 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.
12. Deady, L. W.; Rodemann, T. J.: Synthesis of perimidine and fused perimidine derivatives from reaction of 1,8-naphthalenediamine with an iminoisocoumarin. *Heterocycl. Chem.* **35** (1998) 1417–1419.