Deng-Ze Wu*

The crystal structure of 3-methoxyphenanthridin-6(5*H*)-one, C₁₄H₁₁NO₂

Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	0.25 × 0.21 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25.1°, >98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7224, 1856, 0.023
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1655
$N(\text{param})_{\text{refined}}$:	155
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], DIAMOND [4]

<https://doi.org/10.1515/ncrs-2019-0222>

Received March 25, 2019; accepted April 17, 2019; available online May 13, 2019

Abstract

C₁₄H₁₁NO₂, monoclinic, $P2_1/c$ (no. 14), $a = 12.7731(19)$ Å, $b = 5.4767(9)$ Å, $c = 17.3616(18)$ Å, $\beta = 119.747(7)^\circ$, $V = 1054.5(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0562$, $wR_{\text{ref}}(F^2) = 0.1907$, $T = 298(2)$ K.

CCDC no.: 1903701

The molecular structure of the title crystal structure is shown in the figure (atoms are drawn with arbitrary radii). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was obtained according to the literature method [5]. Single crystal suitable for X-ray diffraction were grown during slow evaporation of a mixture of ethyl acetate and petroleum ether.

Experimental details

H atoms bonded were positioned geometrically and refined using a riding model, with C–H = 0.93/0.96/0.97 Å and N–H = 0.86 Å with $U_{\text{iso}} = 1.2$ times $U_{\text{eq}}(\text{C})$ and 1.2 times $U_{\text{eq}}(\text{N})$.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.65335(18)	1.4052(4)	1.04173(12)	0.0309(5)
O2	0.10937(18)	0.8896(4)	0.70427(14)	0.0388(6)
N1	0.4843(2)	1.2381(4)	0.93005(14)	0.0260(5)
H1	0.442630	1.351950	0.936183	0.031*
C1	0.6166(3)	0.8733(5)	0.90424(18)	0.0268(6)
C2	0.6881(3)	0.6964(5)	0.89365(19)	0.0313(7)
H2	0.651512	0.576088	0.850592	0.038*
C3	0.8122(3)	0.6991(6)	0.9465(2)	0.0386(7)
H3	0.858171	0.580135	0.938842	0.046*
C4	0.8693(3)	0.8789(6)	1.0114(2)	0.0392(8)
H4	0.952796	0.880146	1.046757	0.047*
C5	0.8002(3)	1.0541(6)	1.0222(2)	0.0357(7)
H5	0.837667	1.174536	1.065098	0.043*
C6	0.6747(3)	1.0533(5)	0.96955(18)	0.0274(6)
C7	0.6054(2)	1.2450(5)	0.98415(17)	0.0257(6)
C8	0.4217(2)	1.0626(5)	0.86554(17)	0.0242(6)
C9	0.4851(2)	0.8749(5)	0.85166(17)	0.0247(6)
C10	0.4162(3)	0.6988(5)	0.78704(18)	0.0281(6)
H10	0.455697	0.572087	0.776251	0.034*
C11	0.2929(3)	0.7086(5)	0.73962(19)	0.0304(6)
H11	0.249726	0.589542	0.697434	0.036*
C12	0.2325(2)	0.8977(5)	0.75488(18)	0.0294(6)
C13	0.2963(2)	1.0768(5)	0.81720(18)	0.0270(6)
H13	0.255981	1.204584	0.826609	0.032*
C14	0.0437(3)	1.0860(6)	0.7145(3)	0.0467(9)
H14A	0.063324	1.096460	0.775388	0.070*
H14B	−0.041188	1.057347	0.677702	0.070*
H14C	0.064933	1.236345	0.697203	0.070*

*Corresponding author: Deng-Ze Wu, College of Chemistry and Materials Engineering, Wenzhou University, Chashan University Town, Wenzhou Zhejiang Province 325035, P.R. China, e-mail: wudengze@wzu.edu.cn

Discussion

In recent years, phenanthridinone has been found to be a key core unit in some natural products with important biological

activities [6–8] and drug molecules for the treatment of nerve diseases [9–11]. Thus, the synthesis and crystal structures of various phenanthridinone derivatives have attracted great attention [5, 12].

There is one molecule in the asymmetric unit. In the title compound, the two benzo rings and the pyridin-2(1*H*)-one ring are in the same plane, indicating the compound is an almost planar molecule. This planar parent structure is also present in the previously reported phenanthridinone derivatives [5, 13]. The molecules of the title compound in the same layer are connected by N(1)–H(1)···O(1) hydrogen bonds (donor-acceptor distance: 2.829 Å), which is in agreement with that in previously reported structure of phenanthridone [13]. The molecules in the next layers are stabilized by two kinds of aromatic C–H··· π (2.805 and 3.441 Å) and π – π stacking interaction with a distance of 3.686 Å between the pyridin-2(1*H*)-one moieties.

Acknowledgements: I acknowledge the financial support from Wenzhou University for the publication fee.

References

1. Oxford Diffraction Ltd, CrysAlis^{PRO}, Abingdon, Oxfordshire, England (2006).
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. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Ver. 4.0. Crystal Impact, Bonn, Germany (2015).
5. Saha, R.; Sekar, G.: Stable Pd-nanoparticles catalyzed domino CAH activation/CAN bond formation strategy: An access to phenanthridinones. *J. Catal.* **366** (2018) 176–188.
6. Sarikaya, B. B.; Zencir, S.; Somer, U. N.; Kaya, G. I.; Onur, M. A.; Bastida, J.; Berenyi, A.; Zupko, I.; Topcu, Z.: The effects of arolycoridine and narciprimine on tumor cell killing and topoisomerase activity. *Rec. Nat. Prod.* **6** (2012) 381–385.
7. Chakthong, S.; Ampaprom, R.; Inparn, S.; Phetkul, U.; Chusri, S.; Limsuwan, S.; Voravuthikunchai, S. P.: New alkylamide from the stems of zanthoxylum nitidum. *Nat. Prod. Res.* **33** (2019) 153–161.
8. Wurz, G.; Hofer, O.; Greger, H.: Structure and synthesis of phenaglydon, a new quinolone derived phenanthridine alkaloid from *Glycosmis cyanocarpa*. *Nat. Prod. Lett.* **3** (1993) 177–182.
9. Ruchelman, A. L.; Houghton, P. J.; Zhou, N.; Liu, A.; Liu, L. F.; LaVoie, E. J.: 5-(2-Aminoethyl)dibenzo[*c,h*][1,6]naphthyridin-6-ones: variation of *n*-alkyl substituents modulates sensitivity to efflux transporters associated with multidrug resistance. *J. Med. Chem.* **48** (2005) 792–804.
10. Lin, S. Y.; Liu, C. L.; Chang, Y. M.; Zhao, J.; Perlman, S.; Hou, M. H.: Structural basis for the identification of the *N*-terminal domain of coronavirus nucleocapsid protein as an antiviral target. *J. Med. Chem.* **57** (2014) 2247–2257.
11. Haikarainen, T.; Narwal, M.; Joensuu, P.; Lehtio, L.: Evaluation and structural basis for the inhibition of tankyrases by PARP inhibitors. *Med. Chem. Lett.* **5** (2014) 18–22.
12. Harayama, T.; Akamatsu, H.; Okamura, K.; Miyagoe, T.; Akiyama, T.; Abe, H.; Takeuchi, Y.: Synthesis of trisphaeridine and norchelerythrine through palladiumcatalyzed aryl-aryl coupling reaction. *J. Chem. Soc. Perkin Trans.* **1** (2001) 523–528.
13. Sen, D. K.: Crystal and molecular structure of phenanthridone. *Acta Crystallogr.* **B26** (1970) 1629–1630.