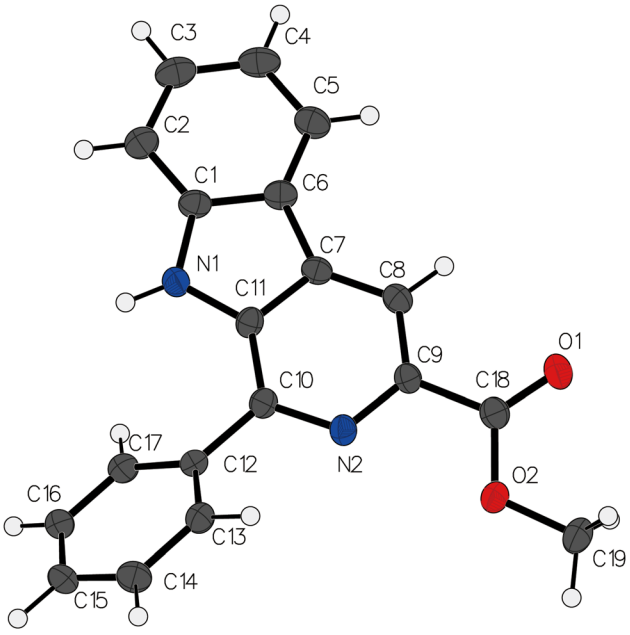
Hangzhou Tian, Geyu Chen, Sen Chen, Wenqiang Tang and Bin Liu*

Crystal structure of methyl 1-phenyl-9*H*-pyrido[3,4-*b*]indole-3-carboxylate, $C_{19}H_{14}N_2O_2$



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Abstract

$C_{19}H_{14}N_2O_2$, monoclinic, $P2_1/n$ (no. 14), a = 11.3111(5) Å, b = 7.2906(3) Å, c = 18.0875(10) Å, β = 102.935(2)° V = 1,453.73(12) Å^3, Z = 4, $R_{gt}(F)$ = 0.0545, $wR_{ref}(F^2)$ = 0.1389, T = 150 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

***Corresponding author: Bin Liu**, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China, E-mail: lb125lb@163.com. https://orcid.org/0000-0003-2852-7739
Hangzhou Tian, Geyu Chen, Sen Chen and Wenqiang Tang, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China. https://orcid.org/0000-0001-5150-4549 (W. Tang)

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.16 × 0.08 × 0.06 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
θ_{max}, completeness:	26.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10,087, 2,812, 0.085
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1,853
$N(param)_{refined}$:	209
Programs:	Bruker[1], SHELX[2,3], Olex2[4]

1 Source of materials

To a solution of 3-chloro-1-phenyl-9*H*-pyrido[3,4-*b*]indole (2.78 g, 10 mmol) in methanol (25 mL) were added 1,1′-bis(diphenylphosphino)ferrocene-palladium(II) dichloride dichloromethane complex (408 mg, 0.5 mmol) and triethylamine (3.0 g, 30 mmol). Then, carbon monoxide gas (5 atm) was introduced into the reaction system and stirred at 60 °C for 3 h, until the TLC indicated the reaction was completed. The solvent was evaporated using a rotary evaporator, yielding the crude product that was purified. For crystal growth, the crude product was dissolved in a minimal amount of hot methanol and slowly cooled to room temperature.

2 Experimental details

Hydrogen atoms were assigned isotropic displacement factors: U_{iso}(H) = 1.2 times U_{eq}(C). All hydrogen atoms were refined as being bonded to their respective parent atoms.

3 Comment

Pyrido[3,4-*b*]indoles, which consist of a fused indole ring with a pyridine component, have been discovered to disrupt DNA replication and transcription, evoke strong neuropharmacological effects, and exhibit excellent cytotoxicity against cancer cells, fungi, and bacteria.[5] Numerous crystal structures of pyrido[3,4-*b*]indole derivatives have been reported in the literature.[6–12] In this paper, we report a crystal structure of a pyrido[3,4-*b*]indole derivative and provide a detailed

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1410 (2)	0.6852 (3)	0.54911 (13)	0.0240 (6)
C2	0.0573 (2)	0.7632 (3)	0.48928 (14)	0.0309 (6)
H2	−0.022128	0.794584	0.494122	0.037*
C3	0.0942 (2)	0.7933 (3)	0.42256 (14)	0.0333 (7)
H3	0.039024	0.847512	0.380913	0.040*
C4	0.2103 (3)	0.7464 (3)	0.41453 (14)	0.0339 (7)
H4	0.232535	0.767712	0.367628	0.041*
C5	0.2933 (2)	0.6691 (3)	0.47434 (14)	0.0293 (6)
H5	0.372599	0.638181	0.469100	0.035*
C6	0.2584 (2)	0.6374 (3)	0.54257 (13)	0.0241 (6)
C7	0.3204 (2)	0.5697 (3)	0.61665 (13)	0.0221 (5)
C8	0.4372 (2)	0.5100 (3)	0.64865 (13)	0.0234 (6)
H8	0.497626	0.503812	0.619758	0.028*
C9	0.4620 (2)	0.4600 (3)	0.72446 (13)	0.0215 (5)
C10	0.2671 (2)	0.5224 (3)	0.73927 (13)	0.0214 (5)
C11	0.2353 (2)	0.5765 (3)	0.66288 (13)	0.0214 (5)
C12	0.1810 (2)	0.5238 (3)	0.79032 (13)	0.0214 (5)
C13	0.2208 (2)	0.5743 (3)	0.86635 (13)	0.0258 (6)
H13	0.302957	0.609362	0.885050	0.031*
C14	0.1418 (2)	0.5739 (4)	0.91458 (14)	0.0314 (6)
H14	0.169979	0.608292	0.966195	0.038*
C15	0.0217 (2)	0.5235 (3)	0.88795 (14)	0.0309 (6)
H15	−0.032702	0.524463	0.921050	0.037*
C16	−0.0187 (2)	0.4717 (3)	0.81268 (14)	0.0294 (6)
H16	−0.100834	0.436256	0.794324	0.035*
C17	0.0601 (2)	0.4715 (3)	0.76440 (14)	0.0260 (6)
H17	0.031772	0.435366	0.713019	0.031*
C18	0.5854 (2)	0.3917 (3)	0.76081 (14)	0.0245 (6)
C19	0.7064 (2)	0.2410 (4)	0.86703 (15)	0.0338 (7)
H19A	0.736619	0.157553	0.833004	0.051*
H19B	0.698172	0.174387	0.912670	0.051*
H19C	0.763623	0.342675	0.881388	0.051*
N1	0.12679 (17)	0.6445 (3)	0.62153 (11)	0.0250 (5)
H1	0.060068	0.659505	0.638212	0.030*
N2	0.38040 (17)	0.4664 (3)	0.76895 (11)	0.0227 (5)
O1	0.67253 (15)	0.4035 (3)	0.73280 (10)	0.0345 (5)
O2	0.58916 (14)	0.3127 (2)	0.82863 (9)	0.0264 (4)

discussion, thereby offering a foundation for drug development based on the reported crystal structure.

In the crystal structure of the titled compound, the skeleton atoms of the pyrido[3,4-*b*]indole moiety are almost coplanar, forming a conjugated structure consistent with many previously reported crystal structures of pyrido[3,4-*b*]indole derivatives.[13–19] Additionally, the atoms in the aliphatic group within this crystal structure are also nearly coplanar with the skeleton atoms of the pyrido[3,4-*b*]indole moiety, with the dihedral angle C8–C9–C18–O1 being only 12.5(4)°. The benzene ring in the crystal structure is significantly twisted relative to the pyrido[3,4-*b*]indole moiety, with a dihedral angle of 39.5°. Furthermore, no significant intermolecular hydrogen bonds were observed in the crystal structure.

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

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