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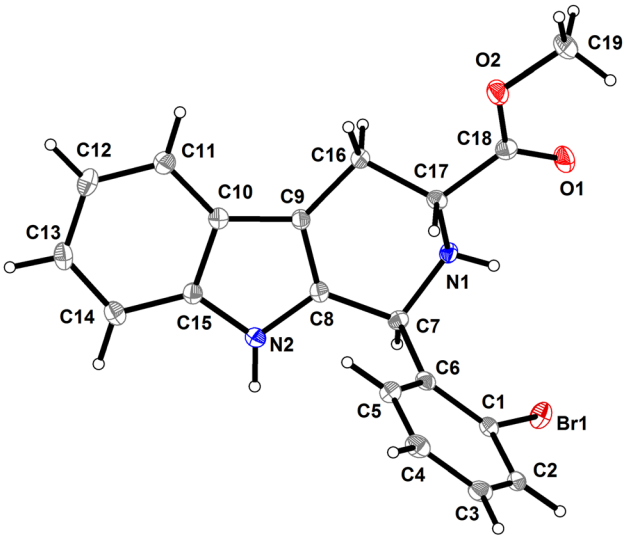
Crystal structure of methyl 1-(2-bromophenyl)-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indole-3-carboxylate, $C_{19}H_{17}BrN_2O_2$



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Abstract

$C_{19}H_{17}BrN_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 9.3764(3) Å, b = 9.7644(3) Å, c = 17.9519(6) Å, V = 1643.58(9) Å^3, Z = 4, $R_{gt}(F)$ = 0.0270 $wR_{ref}(F^2)$ = 0.0601, T = 170 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.12 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.52 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18,873, 3368, 0.054
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3033
$N(param)_{refined}$:	222
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

1 Source of materials

To a solution of D-tryptophan methyl ester hydrochloride (2.55 g, 10 mmol) in methanol (30 mL) was added 1-naphthaldehyde (2.21 g, 12 mmol). The mixture was refluxed for 3 h, until the TLC indicated the reaction was completed. The solvent was evaporated to dryness, yielding the crude product that was then purified to obtain a single crystal of high quality. For crystal growth, the crude product was dissolved in a minimal amount of hot ethanol 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

Tryptophan, an aromatic compound with a dual-ring indole side chain, is one of the rarest amino acids in the protein repertoire [5]. This article presents a novel crystal structure of a tryptophan derivative, specifically methyl 1-(2-bromophenyl)-2,3,4,9-tetrahydro-1H-pyrido[3,4-b] indole-3-carboxylate. Remarkably similar to the previously reported structures in the literature, its unique conformation potentially endows it with significant biological activity [6–10].

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.48962 (4)	−0.06258 (3)	0.40452 (2)	0.03152 (11)
C1	0.3626 (4)	−0.0225 (3)	0.48487 (18)	0.0217 (7)
C2	0.2428 (4)	−0.1026 (3)	0.49184 (19)	0.0259 (8)
H2A	0.225109	−0.174990	0.457739	0.031*
C3	0.1483 (4)	−0.0760 (4)	0.5494 (2)	0.0286 (8)
H3	0.064464	−0.129655	0.554727	0.034*
C4	0.1764 (4)	0.0289 (3)	0.5989 (2)	0.0285 (8)
H4	0.112349	0.046236	0.638814	0.034*
C5	0.2975 (4)	0.1089 (3)	0.5908 (2)	0.0250 (7)
H5	0.314705	0.181289	0.625072	0.030*
C6	0.3948 (4)	0.0852 (3)	0.53331 (18)	0.0205 (7)
C7	0.5301 (3)	0.1684 (3)	0.52481 (17)	0.0201 (7)
H7	0.540613	0.192241	0.470926	0.024*
C8	0.5267 (4)	0.2997 (3)	0.56827 (16)	0.0209 (7)
C9	0.6135 (4)	0.3321 (3)	0.62681 (18)	0.0203 (7)
C10	0.5731 (3)	0.4682 (3)	0.65009 (17)	0.0201 (7)
C11	0.6210 (4)	0.5602 (4)	0.70385 (18)	0.0257 (7)
H11	0.695132	0.535012	0.737230	0.031*
C12	0.5594 (4)	0.6883 (3)	0.7080 (2)	0.0301 (8)
H12	0.592022	0.751217	0.744656	0.036*
C13	0.4503 (4)	0.7275 (3)	0.6597 (2)	0.0297 (8)
H13	0.411710	0.817228	0.663384	0.036*
C14	0.3972 (4)	0.6389 (3)	0.6064 (2)	0.0269 (8)
H14	0.322267	0.665092	0.573753	0.032*
C15	0.4591 (3)	0.5087 (3)	0.60289 (18)	0.0210 (7)
C16	0.7226 (4)	0.2337 (3)	0.65598 (18)	0.0213 (7)
H16A	0.725344	0.236738	0.711089	0.026*
H16B	0.818428	0.257417	0.636733	0.026*
C17	0.6788 (3)	0.0906 (3)	0.62925 (17)	0.0195 (7)
H17	0.584995	0.067339	0.652519	0.023*
C18	0.7821 (4)	−0.0226 (3)	0.64673 (19)	0.0240 (8)
C19	0.9074 (5)	−0.1402 (4)	0.7410 (2)	0.0413 (11)
H19A	1.005399	−0.127375	0.723358	0.062*
H19B	0.867906	−0.224206	0.719389	0.062*
H19C	0.907304	−0.147629	0.795472	0.062*
N1	0.6597 (3)	0.0941 (3)	0.54795 (16)	0.0208 (6)
H1	0.656 (4)	0.014 (2)	0.5346 (18)	0.019 (9)*
N2	0.4326 (3)	0.4038 (3)	0.55344 (15)	0.0239 (6)
H2	0.366903	0.403691	0.518448	0.029*
O1	0.8221 (3)	−0.1057 (2)	0.60220 (14)	0.0336 (6)
O2	0.8210 (3)	−0.0239 (3)	0.71861 (13)	0.0364 (6)

In the titled compound, the carbon and nitrogen atoms within the indole moiety are nearly coplanar. The benzene ring in the benzylmethoxy segment is almost perpendicular to the indole plane, displaying a dihedral angle of 75.2°. The atoms in the six-membered piperidine ring are nonplanar, with a dihedral angle of 72.3° observed for C16–C17–N1–C7. The bond lengths, bond angles, and dihedral angles in the molecular structure fall within acceptable ranges and are consistent with similar structures previously reported [11–15].

Research ethics: Not applicable.

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

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

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Data availability: Not applicable.

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