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The crystal structure of 2-(2-(2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-2-ium-1-yl)phenoxy)acetate, C₁₉H₁₈N₂O₃

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.09 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.74 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	73.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3187, 3187,
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2800
$N(\text{param})_{\text{refined}}$:	218
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

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Abstract

C₁₉H₁₈N₂O₃, monoclinic, $P2_1/c$ (no. 14), $a = 12.6187(7)$ Å, $b = 9.4445(5)$ Å, $c = 13.7424(8)$ Å, $\beta = 99.248(5)^\circ$, $V = 1616.49(16)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0793$, $wR_{\text{ref}}(F^2) = 0.2220$, $T = 100(10)$ 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.

Source of material

0.0901 g (2-Formylphenoxy)acetic acid (0.5 mmol), 0.0801 g tryptamin (0.5 mmol) and 0.020 g NaOH (0.5 mmol) were added to the solution of water-ethanol ($v:v = 1:1$) and the solution was heated up to 70 °C with stirring. After 1 h, 0.1098 g zinc(II) acetate dihydrate (0.5 mmol) was added.

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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	0.5085(2)	0.1878(3)	0.3942(2)	0.0192(7)
O2	0.5907(2)	0.2430(3)	0.2318(2)	0.0189(6)
O3	0.4614(2)	0.1269(3)	0.1337(2)	0.0204(7)
N1	0.7459(3)	0.4256(4)	0.6624(3)	0.0179(8)
H1	0.705240	0.374543	0.692848	0.021*
N2	0.6697(3)	0.4037(4)	0.3903(3)	0.0156(7)
H2A	0.636055	0.342872	0.346439	0.019*
H2B	0.634804	0.485823	0.381910	0.019*
C1	0.9546(3)	0.6802(5)	0.6557(4)	0.0217(9)
H1A	0.986453	0.724683	0.607410	0.026*
C2	0.9873(4)	0.7112(5)	0.7543(4)	0.0269(11)
H2	1.041296	0.777640	0.772051	0.032*
C3	0.9406(4)	0.6444(5)	0.8277(3)	0.0246(10)
H3	0.965523	0.665776	0.893496	0.029*
C4	0.8582(4)	0.5471(5)	0.8049(3)	0.0226(10)
H4	0.826641	0.503344	0.853666	0.027*
C5	0.8249(3)	0.5180(5)	0.7055(3)	0.0172(9)
C6	0.8723(3)	0.5801(5)	0.6298(3)	0.0175(9)
C7	0.8202(3)	0.5205(4)	0.5382(3)	0.0165(9)
C8	0.7441(3)	0.4299(4)	0.5619(3)	0.0159(8)
C9	0.6636(3)	0.3492(4)	0.4924(3)	0.0156(8)
H9	0.592164	0.371503	0.507574	0.019*
C10	0.8363(3)	0.5439(5)	0.4334(3)	0.0188(9)
H10A	0.912343	0.544360	0.429440	0.023*
H10B	0.806251	0.634536	0.409979	0.023*
C11	0.7805(3)	0.4249(5)	0.3698(3)	0.0197(9)
H11A	0.778131	0.447998	0.300706	0.024*
H11B	0.821106	0.337903	0.383305	0.024*

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C12	0.6766(3)	0.1898(4)	0.4944(3)	0.0166(9)
C13	0.7628(4)	0.1195(5)	0.5491(3)	0.0209(9)
H13	0.817328	0.171751	0.586250	0.025*
C14	0.7691(4)	−0.0271(5)	0.5496(3)	0.0231(10)
H14	0.827093	−0.072882	0.586909	0.028*
C15	0.6877(4)	−0.1045(5)	0.4938(3)	0.0245(10)
H15	0.691570	−0.202852	0.493881	0.029*
C16	0.6005(4)	−0.0379(5)	0.4378(3)	0.0214(9)
H16	0.546887	−0.090809	0.400016	0.026*
C17	0.5946(3)	0.1094(5)	0.4391(3)	0.0165(8)
C18	0.4487(3)	0.1378(5)	0.3038(3)	0.0220(10)
H18A	0.378025	0.181005	0.293769	0.026*
H18B	0.439335	0.036097	0.307890	0.026*
C19	0.5042(3)	0.1715(4)	0.2164(3)	0.0176(9)

The mixture continued stirring for 4 hours at 70 °C. After cooled to room temperature, the mixture was filtered. The colorless crystals of 2-(2-(2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-2-ium-1-yl)phenoxy)acetate were received through slow evaporation after 30 days.

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints. The hydrogen atoms were positioned geometrically (C–H = 0.93–0.97 Å and N–H = 0.86–0.89 Å). Their U_{iso} values were set to 1.2 U_{eq} of the parent atoms.

Comment

Many ylide compounds show widespread applications in organic chemistry [5–8]. In this paper, we report the synthesis of the side-product of the reaction of (2-formylphenoxy)acetic acid, tryptamin and Zn(II)ion. The new three-membered ring ylide compound, 2-(2-(2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-2-ium-1-yl)phenoxy)acetate, has been obtained. In the title compound, the C–N bond distances are 1.378(5) Å (N1–C8), 1.384(5) Å (N1–C5), 1.508(5) Å (N2–C9) and 1.484(5) Å (N2–C11), respectively. The C–O bond distances are 1.376(5) Å (O1–C17), 1.427(5) Å (O1–C18), 1.272(5) Å

(O2–C19) and 1.250(5) Å (O3–C19), respectively. The dihedral angles of ring 1 (C1–C2–C3–C4–C5–C6) and ring 2 (C5–C6–C7–C8–N1), ring 1 (C1–C2–C3–C4–C5–C6) and ring 3 (C7–C8–C9–N2–C11–C10), ring 2 (C7–C8–C9–N2–C11–C10) and ring 3 (C7–C8–C9–N2–C11–C10) are 1.4°, 5.8° and 4.4°, respectively, indicating that these rings are almost coplanar. However, the dihedral angles of ring 4 (C12–C13–C14–C15–C16–C17) with ring 1, ring 2 and ring 3 are 69.6°, 71.0° and 75.1°, respectively (see the figure). Geometric parameters are all in the expected ranges [9].

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