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The crystal structure of (*E*)-2-((2-picolinoylhydrazono)methyl)phenoxy)acetic acid dihydrate, C₁₅H₁₇N₃O₆

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Abstract

C₁₅H₁₇N₃O₆, triclinic, $P\bar{1}$ (no. 2), $a = 7.2155(8)$ Å, $b = 10.7519(8)$ Å, $c = 10.8562(8)$ Å, $\alpha = 107.910(7)^\circ$, $\beta = 97.087(8)^\circ$, $\gamma = 100.409(7)^\circ$, $V = 773.69(12)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0500$, $wR_{\text{ref}}(F^2) = 0.1238$, $T = 150(1)$ 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.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.10 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5320, 2732, 0.042
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1836
$N(\text{param})_{\text{refined}}$:	224
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

Source of material

The title compound has been synthesized according to the following method: a mixture of 0.0895 g 2-formyl phenoxyacetic acid (0.5 mmol), 0.0685 g pyridine-2-carbohydrazide (0.5 mmol) and 0.020 g sodium hydroxide (0.5 mmol) were stirred and dissolved in 10 mL ethanol-water ($v:v = 1:1$). After complete dissolution, 0.0595 g calcium perchlorate hydrate (0.25 mmol) was added to the above solution. The reaction mixture was stirred for 3 h at 70 °C, cooled to room temperature and filtered. The colorless crystals of the title compound were obtained from the solution after 25 days.

Experimental details

The hydrogen atoms were positioned geometrically (C-H = 0.93–0.97 Å, N-H = 0.86 Å and O-H = 0.82–0.85 Å). Their U_{iso} values were set to $1.2U_{\text{eq}}$ or $1.5U_{\text{eq}}$ of the parent atoms.

Comment

Hydrazones and their metal complexes have found considerable applications in catalysis [5], and some show anticancer and antimicrobial activities [6, 7], or are used as electrochemical sensors [8]. Our research group has been devoted to the study of hydrazones [9–11].

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

Atom	x	y	z	U _{iso} */U _{eq}
O1	0.6069 (3)	0.83791 (15)	0.51295 (15)	0.0343 (5)
O2	0.8323 (2)	0.24274 (14)	0.43498 (14)	0.0276 (4)
O3	0.8027 (3)	0.11628 (15)	0.18016 (15)	0.0361 (5)
O4	0.9119 (3)	−0.05242 (15)	0.21748 (15)	0.0328 (5)
H4	0.895523	−0.082009	0.136762	0.049*
N1	0.5153 (3)	0.65522 (18)	0.17074 (18)	0.0302 (5)
N2	0.6282 (3)	0.62735 (17)	0.39873 (17)	0.0235 (5)
H2	0.606517	0.565728	0.322661	0.028*
N3	0.7051 (3)	0.60268 (18)	0.50855 (17)	0.0231 (5)
C1	0.5115 (3)	0.7574 (2)	0.2787 (2)	0.0249 (6)
C2	0.4448 (4)	0.8691 (2)	0.2737 (2)	0.0296 (6)
H2A	0.444782	0.937979	0.350667	0.036*
C3	0.3783 (4)	0.8755 (2)	0.1516 (3)	0.0345 (7)
H3	0.331582	0.948689	0.144631	0.041*
C4	0.3822 (4)	0.7720 (3)	0.0404 (3)	0.0379 (7)
H4A	0.339004	0.774163	−0.043100	0.046*
C5	0.4511 (4)	0.6653 (3)	0.0550 (2)	0.0371 (7)
H5	0.452859	0.595767	−0.020840	0.044*
C6	0.5864 (3)	0.7461 (2)	0.4083 (2)	0.0246 (6)
C7	0.7509 (3)	0.4901 (2)	0.4857 (2)	0.0226 (6)
H7	0.729395	0.432038	0.399153	0.027*
C8	0.8361 (3)	0.4496 (2)	0.5918 (2)	0.0221 (5)
C9	0.8820 (4)	0.5367 (2)	0.7218 (2)	0.0281 (6)
H9	0.855793	0.621496	0.741377	0.034*
C10	0.9652 (4)	0.4996 (2)	0.8219 (2)	0.0318 (6)
H10	0.995637	0.559081	0.908166	0.038*
C11	1.0035 (4)	0.3734 (2)	0.7935 (2)	0.0294 (6)
H11	1.059675	0.348053	0.861000	0.035*
C12	0.9590 (4)	0.2849 (2)	0.6659 (2)	0.0262 (6)
H12	0.984294	0.199923	0.647599	0.031*
C13	0.8768 (3)	0.3223 (2)	0.5652 (2)	0.0215 (5)
C14	0.8914 (4)	0.1193 (2)	0.4014 (2)	0.0267 (6)
H14A	0.816243	0.057163	0.434923	0.032*
H14B	1.025664	0.134507	0.439776	0.032*
C15	0.8618 (4)	0.0624 (2)	0.2544 (2)	0.0247 (6)
O5	0.8818 (3)	−0.1644 (2)	−0.03305 (16)	0.0407 (5)
H5A	0.979458	−0.145209	−0.065953	0.061*
H5B	0.780487	−0.180286	−0.089671	0.061*
O6	0.5732 (3)	−0.2216 (2)	−0.23826 (17)	0.0445 (5)
H6A	0.472136	−0.191659	−0.233432	0.067*
H6B	0.607254	−0.223954	−0.310932	0.067*

The title compound consists of one (*E*)-2-(2-((2-picolinoylhydrazono)methyl)phenoxy)acetic acid molecule and two lattice water molecules (the Figure). The bond distance of C7–N3 is 1.272(2) Å, which is shortest among those of C6–N2 (1.341(2) Å), C1–N1 (1.344(3) Å) and C5–N1 (1.328(3) Å), showing that the C7–N3 bond is a double bond [12, 13]. The bond distances of C6–O1 (1.226(3) Å) and C15–O3 (1.208(2) Å) are shorter than that of C15–O4 (1.307(2) Å), showing that the C6–O1 and C15–O3 bonds are also double bonds. The dihedral angle between the benzene ring (C8–C9–C10–C11–C12–C13) and pyridine ring (C1–C2–C3–C4–C5–N1) is 8.0°, indicating that the

whole hydrazone molecule is almost coplanar. In the crystal packing, the chain structure was formed by intermolecular O–H...O hydrogen bonds.

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