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The crystal structure of 6-hydroxy-5H-pyrrolo[3,4-b]pyridine-5,7(6H)-dione monohydrate, $C_7H_6N_2O_4$

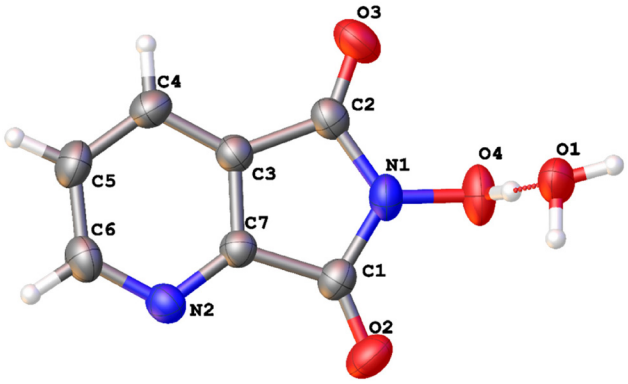

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Bruker Apex2, φ and ω scan
θ_{max}, completeness:	27.6°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12460, 1,776, 0.020
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1573
$N(param)_{refined}$:	130
Programs:	Olex2,[1] SHELX,[2,3] Bruker[4]

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Abstract

$C_7H_6N_2O_4$, monoclinic, $P2_1/c$ (no. 14), a = 8.1416(3) Å, b = 10.2220(3) Å, c = 9.2212(3) Å, β = 91.7600(10)°, V = 767.06(4) $Å^3$, Z = 15, $R_{gt}(F)$ = 0.1022, $wR_{ref}(F^2)$ = 0.1077, T = 296.15 K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

1 Source of materials

All chemicals were purchased from commercial sources and used as received without further purification, and 2,3-pyridinedicarboxylic anhydride was synthesized by our laboratory. Under N_2, anhydrous sodium acetate (3.956 g, 48.2 mmol) and hydroxylamine hydrochloride (3.956 g, 57.1 mmol) were dissolved in acetic acid (25 mL). Then, the above mixture solution was refluxed for 5 min. The mixture was filtered to remove precipitated NaCl. And then, 2,3-pyridinedicarboxylic anhydride (3.956 g, 26.6 mmol) was added to the filtrate, and the mixture was refluxed for 25 min. After completion, the mixture was filtered and concentrated to a saturated solution under vacuum, a yellow precipitate is formed. The precipitate was collected by filtration, washed with diethyl ether. The crystals were obtained by recrystallization in the cooled acetonitrile solution. 1H NMR (400 MHz, DMSO-d_6) δ: 11.04 (s, 1H) 8.94 (m, 1H), 8.24 (m, 1H), 7.77 (m, 1H). 13C NMR (101 MHz, DMSO-d_6) δ: 162.66, 154.58, 149.01, 130.96, 128.03, 125.32.

2 Experimental details

Olex2[1] software and the SHELXL[2] refinement were used to solve and refine the crystal structure. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Organic nitrogen hydroxy compounds (N–OH), as a novel class of organic catalysts, with *N*-hydroxyphthalimide (NHPI) being the most representative, have recently been

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employed as an effective system for C–H bonds activation via hydrogen extraction.[5–9] The molecule features the unique properties of the N–OH function which is different from the previously proposed systems.[10,11] Thus, organic nitrogen hydroxy compounds have attracted increasing interest from both the academic and industrial communities over the past decade.[12] These compounds primarily achieve classical C–H bond oxidation reactions through the homolytic cleavage of the hydroxyl bond in N–OH to form N-oxyl radical.[13–15] As a result, this kind of compound has a wide range of applications in carbon-carbon, carbon-nitrogen, and carbon-silicon bond formation reactions.[12] With the continuous in-depth research on the synthesis and application of organic nitrogen hydroxy compounds, exploring the structural characteristics of these compounds is of great significance for understanding their structure-activity relationships.

However, reports on the crystal structures of organic nitrogen hydroxy compounds remain limited. To date, only two classes of symmetrical organic nitrogen hydroxyl compounds have been reported, i.e., HPPDO[16] and DNHPI.[17]

The molecular structure comprises at least two distinct planes: the pyridine ring plane, the pyrrole ring plane. In the crystal, the torsion angles of the nitrogen hydroxyl group (angle N(1)–O(4)H) is 106.8°. The O–H bond length in the nitrogen hydroxyl group is 0.95 Å, which is longer than the O–H bond length in water molecules (0.85 Å). Additionally, the hydrogen in the nitrogen hydroxyl group can form hydrogen bonds with water molecules, which are present. All geometric parameters fall within expected ranges.[16,17]

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