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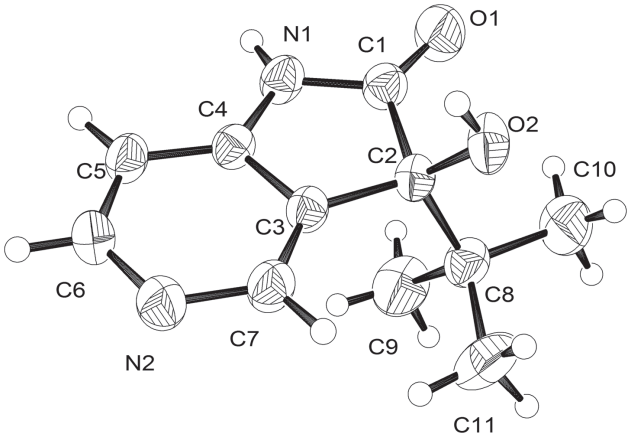
Crystal structure of 3-*tert*-butyl-3-hydroxy-1,3-dihydro-2*H*-pyrrolo[3,2-*c*]pyridin-2-one, $C_{11}H_{14}N_2O_2$



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Abstract

$C_{11}H_{14}N_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.5411(2)$ Å, $b = 11.5148(2)$ Å, $c = 12.5370(2)$ Å, $V = 1088.64(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0301$, $wR_{ref}(F^2) = 0.0826$, $T = 296$ K.

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The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared as previously reported *via* double lithiation of 4-pivaloylaminopyridine with *n*-BuLi (2.1 mole equivalents) in anhydrous THF at 0°C for 3 h followed by a reaction with carbon monoxide. The crude product obtained was purified by crystallization from ethyl acetate to give the title compound in 65% yield as colourless crystals [1].

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.081×0.135×0.298 mm
Wavelength:	Cu$K\alpha$ radiation (1.54184 Å)
μ:	7.17 cm^{-1}
Diffractometer, scan mode:	SuperNova, Dual, Cu at zero, Atlas, ω scans
$2\theta_{max}$:	148.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3668, 2123
$N(param)_{refined}$:	141
Programs:	CrysAlis^{PRO} [5], SHELX [6], WinGX [7], ChemDraw [8]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(5)	4*a*	0.1302	1.0553	0.0934	0.0480
H(6)	4*a*	0.3050	1.0206	−0.0539	0.0520
H(7)	4*a*	0.3790	0.6972	0.02510	0.0500
H(9A)	4*a*	0.5447	0.7417	0.3790	0.1000
H(9B)	4*a*	0.3600	0.8025	0.3922	0.1000
H(9C)	4*a*	0.4705	0.8209	0.2877	0.1000
H(10A)	4*a*	0.3884	0.5571	0.4235	0.0970
H(10B)	4*a*	0.2357	0.5112	0.3498	0.0970
H(10C)	4*a*	0.2024	0.6179	0.4249	0.0970
H(11A)	4*a*	0.5359	0.6524	0.1737	0.1030
H(11B)	4*a*	0.4211	0.5392	0.1812	0.1030
H(11C)	4*a*	0.5708	0.5625	0.2653	0.1030
H(1)	4*a*	0.0055	0.9476	0.2712	0.0480
H(2)	4*a*	0.0242	0.6203	0.1404	0.0660

Discussion

Synthesis of azaindoles is of great interest due to their growing biological applications [2]. The asymmetric unit consists of one molecule of $C_{11}H_{14}N_2O_2$. In the crystal structure, the hydroxyl group accepts one N—H···O hydrogen bond from a neighbouring molecule (N1—H1···O2 angle = 169.63°,

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Table 3: Fractional atomic coordinate and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*a*	0.0581(3)	0.7795(2)	0.2890(1)	0.0419(9)	0.0402(9)	0.0364(8)	0.0029(8)	0.0009(8)	0.0024(8)
C(2)	4*a*	0.1868(2)	0.7014(1)	0.2231(1)	0.0392(9)	0.0281(8)	0.0367(8)	−0.0005(7)	−0.0012(7)	0.0017(6)
C(3)	4*a*	0.2355(2)	0.7836(1)	0.1328(1)	0.0358(8)	0.0278(7)	0.0363(8)	0.0001(7)	−0.0026(7)	0.0000(7)
C(4)	4*a*	0.1578(2)	0.8916(1)	0.1517(1)	0.0356(8)	0.0300(8)	0.0354(8)	0.0007(7)	−0.0030(7)	−0.0021(6)
C(5)	4*a*	0.1812(3)	0.9829(2)	0.0818(2)	0.0428(9)	0.0275(8)	0.049(1)	0.0017(7)	−0.0024(9)	0.0034(7)
C(6)	4*a*	0.2847(3)	0.9599(2)	−0.0064(2)	0.046(1)	0.0376(9)	0.047(1)	−0.0022(8)	0.0019(9)	0.0118(8)
C(7)	4*a*	0.3312(3)	0.7698(2)	0.0404(2)	0.048(1)	0.0336(9)	0.0426(9)	0.0069(8)	0.0063(9)	0.0004(7)
C(8)	4*a*	0.3488(3)	0.6613(2)	0.2921(2)	0.0434(9)	0.0393(9)	0.044(1)	0.0023(8)	−0.0085(8)	0.0025(7)
C(9)	4*a*	0.4394(4)	0.7663(2)	0.3424(2)	0.062(1)	0.060(1)	0.078(2)	−0.004(1)	−0.027(1)	−0.004(1)
C(10)	4*a*	0.2882(4)	0.5793(2)	0.3808(2)	0.062(1)	0.071(2)	0.061(1)	0.000(1)	−0.017(1)	0.027(1)
C(11)	4*a*	0.4814(4)	0.5980(3)	0.2216(2)	0.067(2)	0.073(2)	0.067(2)	0.034(1)	−0.006(1)	0.004(1)
N(1)	4*a*	0.0594(2)	0.8886(1)	0.2445(1)	0.0487(8)	0.0339(7)	0.0385(8)	0.0088(7)	0.0043(7)	−0.0017(6)
N(2)	4*a*	0.3582(2)	0.8572(2)	−0.0288(1)	0.0509(9)	0.0437(9)	0.0443(9)	0.0044(7)	0.0099(8)	0.0072(7)
O(1)	4*a*	−0.0295(2)	0.7513(1)	0.3652(1)	0.071(1)	0.060(1)	0.0525(8)	0.0116(8)	0.0230(8)	0.0138(7)
O(2)	4*a*	0.0957(2)	0.6015(1)	0.1865(1)	0.0555(8)	0.0306(6)	0.0455(7)	−0.0087(6)	−0.0133(6)	0.0068(5)

N1···O2 distance = 2.851 Å) and donates one O—H···N bond to another molecule (O2—H2···N2 angle = 172.48°, O2···N2 distance = 2.710 Å) leading to a 3-D network. An extremely weak C—H···O contact is also observed (C5—H5···O1 angle = 173.13°, C···O distance = 3.362 Å) resulting in $R^2_2(9)$ graph set descriptor for this motif [3] between two molecules. The shortest C—H···O contact in the related 3-isopropyl-1-methyl-1,3-dihydro-2*H*-pyrrolo[3,2-*c*]pyridin-2-one [4] involves a tertiary C atom (C—H···O angle = 144.18° and C···O distance = 3.292 Å).

Experimental details

H atoms were placed in calculated positions and refined using a riding model, with U_{iso}(H) set to 1.2U_{eq}(C) and C—H and N—H distance of 0.93 and 0.86 Å respectively. The exceptions were the methyl (C—H = 0.96 Å) and hydroxyl (O—H = 0.86 Å) groups which were allowed to rotate around the C—C bond (HFIX 137 in SHELX [6]) and C—O bond (HFIX 147), with U_{iso}(H) set to 1.5U_{eq}(C/O).

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