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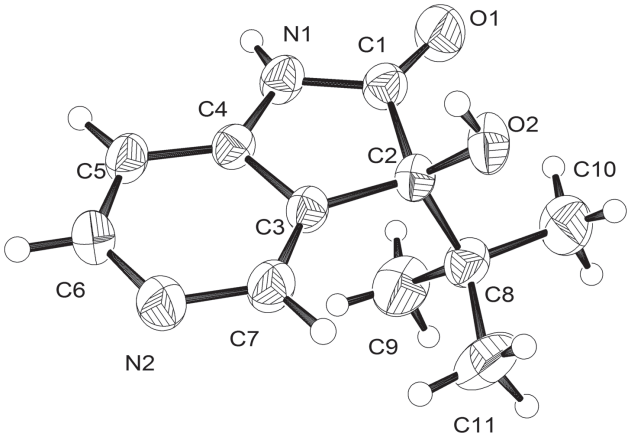
# Crystal structure of 3-*tert*-butyl-3-hydroxy-1,3-dihydro-2*H*-pyrrolo[3,2-*c*]pyridin-2-one, $C_{11}H_{14}N_2O_2$




DOI 10.1515/ncrs-2015-0276
Received December 1, 2015; accepted March 1, 2016; available online April 2, 2016

## 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.

**CCDC no.:** 1456766

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) |
| $\mu$: | 7.17 cm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, Dual, Cu at zero, Atlas, $\omega$ 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 1.2$U_{eq}$(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 1.5$U_{eq}$(C/O).

**Acknowledgements:** The authors extend their appreciation to the Deanship of Scientific Research at King Saud University for its funding for this research through the research group project RGP-VPP-239 and to Cardiff University for continued support.

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