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Crystal structure of 2-tert-butyl-1*H*-imidazo[4,5-*b*]pyridine, C₁₀H₁₃N₃

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Abstract

C₁₀H₁₃N₃, orthorhombic, *Pbca* (no. 14), $a = 9.4795(7)$ Å, $b = 10.7585(10)$ Å, $c = 18.8783(13)$ Å, $V = 1925.3(3)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0702$, $wR_{\text{ref}}(F^2) = 0.1959$, $T = 296(2)$ K.

CCDC no.: 1904458

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

A 25 mL Schlenk tube was charged with 2,3-diiodopyridine (1.0 mmol), pivalamidine (1.2 mmol), Cs₂CO₃ (2.0 mmol), CuI (5 mol%) in 2 mL of toluene step by step. The reaction mixture was stirred at 110 °C for 24 h. Upon the reaction completion (monitored by TLC), the mixture was cooled to room temperature and the reaction mixture was quenched with 2 mL of water, extracted with EtOAc (5 mL), washed with brine. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, concentrated *in vacuo* and the residue was purified by chromatography on silica gel, using hexane/EtOAc (5:1) as the eluent, to give the 2-tert-butyl-1*H*-imidazo[4,5-*b*]pyridine

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.13 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ^{−1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.0°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6333, 1662, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1444
$N(\text{param})_{\text{refined}}$:	121
Programs:	Bruker [1], SHELX [2]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.7971(3)	0.8055(3)	0.58882(12)	0.0186(6)
C2	0.9136(3)	0.8633(3)	0.55913(13)	0.0223(6)
H2	0.917007	0.948488	0.551061	0.027*
C3	1.0251(3)	0.7848(3)	0.54226(13)	0.0231(7)
H3A	1.106510	0.817659	0.522176	0.028*
C4	1.0164(3)	0.6580(3)	0.55498(14)	0.0232(7)
H4	1.093461	0.609194	0.542507	0.028*
C5	0.7965(3)	0.6769(2)	0.60008(12)	0.0182(6)
C6	0.5986(3)	0.7443(2)	0.63739(14)	0.0199(6)
C7	0.4534(3)	0.7524(2)	0.67084(15)	0.0244(7)
C8	0.3651(3)	0.8510(4)	0.6336(2)	0.0506(11)
H8A	0.408000	0.931057	0.640146	0.076*
H8B	0.271797	0.851740	0.653377	0.076*
H8C	0.359937	0.832437	0.583979	0.076*
C9	0.4712(4)	0.7846(6)	0.7471(2)	0.0861(19)
H9A	0.530180	0.723758	0.769589	0.129*
H9B	0.380559	0.785827	0.769838	0.129*
H9C	0.514201	0.865123	0.751120	0.129*
C10	0.3781(4)	0.6290(4)	0.6605(4)	0.088(2)
H10A	0.371103	0.610967	0.610845	0.132*
H10B	0.285157	0.633787	0.680589	0.132*
H10C	0.430315	0.564180	0.683652	0.132*
N1	0.9047(2)	0.6008(2)	0.58401(12)	0.0237(6)
N2	0.6710(2)	0.6396(2)	0.63076(12)	0.0225(6)
N3	0.6686(2)	0.8462(2)	0.61340(11)	0.0189(5)
H3	0.638318	0.921508	0.613565	0.023*

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as a yellow solid. The solid dissolve in chloroform and heated to 60 °C, resulting in a colourless solution. Crystals of the title compound were obtained by slow evaporation within 3 days.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

Substituted imidazopyridine derivatives are privileged organic compounds for their diverse biological and therapeutic activities, which may be related to their structural similarity to purines derivatives, and as the important building blocks of DNA/RNA and the basic heterocyclic structure found in numerous alkaloids [3]. The imidazopyridine derivatives have also found utility in advanced materials [4] and marketed drug formulations and drug candidates [5]. Hence, the development of rapid and efficient methods for construction of imidazopyridine skeleton has stimulated considerable interest [6]. However, the crystal structure of 2-tert-butyl-1*H*-imidazo[4,5-*b*]pyridine has not been reported until now. Herein the crystal structure of the title compound is described to enrich the related crystal structures of 2-tert-butyl-1*H*-imidazo[4,5-*b*]pyridine. Finally it should not keep unmentioned that, amidines are moieties found in current antiviral and antithrombotic drugs presumably because of their unique structural properties [7]. We have previously reported the novel and practical method for synthesis of heterocyclic compounds such as 2-acetylthienopyridine [8], thienopyridines [9], polysubstituted haloimidazoles [10], 5-iminopyrrolones [11] and benzo[*b*]thiophenes [12].

As in our previous study [13, 14], the title compound, built up by the C₁₀H₁₃N₃ molecules (*cf.* the figure), has been synthesized. The single crystal structure verifies that all bond lengths are in normal ranges.

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