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Crystal structure of 2-(3,4-dimethylphenyl)-1,8-naphthyridine, C₁₆H₁₄N₂

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Abstract

C₁₆H₁₄N₂, orthorhombic, $P2_12_12_1$, $a = 6.449(8)$ Å, $b = 10.548(14)$ Å, $c = 17.84(2)$ Å, $V = 1213(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0406$, $wR_{\text{ref}}(F^2) = 0.0933$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Under N₂ atmosphere, t-BuOK (50 mol%), xantphos (4,5-bis(diphenylphosphino)-9,9-dimethylxanthene; 3 mol%), Ru₃(CO)₁₂ (1 mol%), (2-aminopyridin-3-yl)methanol (0.5 mmol), 1-(3,4-dimethylphenyl) ethan-1-ol (0.5 mmol) and

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.30 × 0.26 × 0.24 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10765, –, 0.058
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, –
$N(\text{param})_{\text{refined}}$:	165
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	–0.0273(2)	–0.80067(16)	–0.11350(10)	0.0559(4)
N2	0.0449(3)	–0.65154(17)	–0.20386(10)	0.0688(5)
C1	0.1687(4)	–1.2625(2)	0.11848(14)	0.0781(7)
H1A	0.1812	–1.2338	0.1693	0.117*
H1B	0.3028	–1.2861	0.0998	0.117*
H1C	0.0778	–1.3345	0.1166	0.117*
C2	0.0809(3)	–1.15737(18)	0.07076(12)	0.0597(5)
C3	–0.1196(3)	–1.11146(18)	0.08247(12)	0.0600(5)
C4	–0.1931(3)	–1.01494(18)	0.03748(12)	0.0587(5)
H4	–0.3259	–0.9840	0.0463	0.070*
C5	–0.0777(3)	–0.96187(18)	–0.02032(11)	0.0529(5)
C6	–0.1613(3)	–0.86172(18)	–0.07048(11)	0.0524(5)
C7	–0.1014(3)	–0.70983(18)	–0.16082(11)	0.0549(5)
C8	–0.3138(3)	–0.67853(18)	–0.16533(12)	0.0575(5)
C9	–0.3727(4)	–0.5812(2)	–0.21463(13)	0.0716(6)
H9	–0.5113	–0.5576	–0.2185	0.086*
C10	–0.2273(4)	–0.5219(2)	–0.25650(14)	0.0765(6)
H10	–0.2631	–0.4566	–0.2891	0.092*
C11	–0.0207(4)	–0.5620(2)	–0.24929(14)	0.0749(6)
H11	0.0778	–0.5220	–0.2791	0.090*
C12	–0.3765(3)	–0.8368(2)	–0.07332(13)	0.0647(6)
H12	–0.4666	–0.8824	–0.0428	0.078*
C13	–0.4521(3)	–0.7474(2)	–0.12003(14)	0.0680(6)
H13	–0.5939	–0.7315	–0.1222	0.082*
C14	0.1989(3)	–1.10362(19)	0.01422(12)	0.0638(6)
H14	0.3334	–1.1326	0.0064	0.077*
C15	0.1221(3)	–1.00794(19)	–0.03096(12)	0.0609(6)
H15	0.2047	–0.9742	–0.0687	0.073*
C16	–0.2536(4)	–1.1658(2)	0.14321(13)	0.0800(7)
H16A	–0.3844	–1.1223	0.1439	0.120*
H16B	–0.1860	–1.1555	0.1908	0.120*
H16C	–0.2764	–1.2543	0.1338	0.120*

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tert-amyl alcohol (1.0 mL) were introduced in a Schlenk tube (25 mL), successively. Then, the Schlenk tube was closed and the resulting mixture was stirred at 403 K for 5 h. After cooling to room temperature, the reaction mixture was concentrated removing the solvent under vacuum, and the residue was purified by preparative TLC on silica, eluting with petroleum ether (333–363 K): ethyl acetate (10:1, v/v) to give 2-(3,4-dimethylphenyl)-1,8-naphthyridine as colourless blocks.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses and included using riding models with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The methyl groups were idealized and refined using rigid groups allowed to rotate about the C–C bond (AFIX 137 option of the SHELX) [3].

Discussion

1,8-naphthyridine ring systems are attractive structural motifs, because of their wide distribution in bioactive molecules and pharmaceuticals [5, 6]. Hence, there is considerable interest in the development of effective methods for the synthesis of 1,8-naphthyridine and its analogues [7, 8].

However, the crystal structure of 2-(3,4-dimethylphenyl)-1,8-naphthyridine has not been reported before. Herein the crystal structure of the title compound is described to enrich the compendium of crystal structures of 1,8-naphthyridines. As in our previous study [9], the single-crystal structure verifies that all bond lengths and angles are in normal ranges.

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