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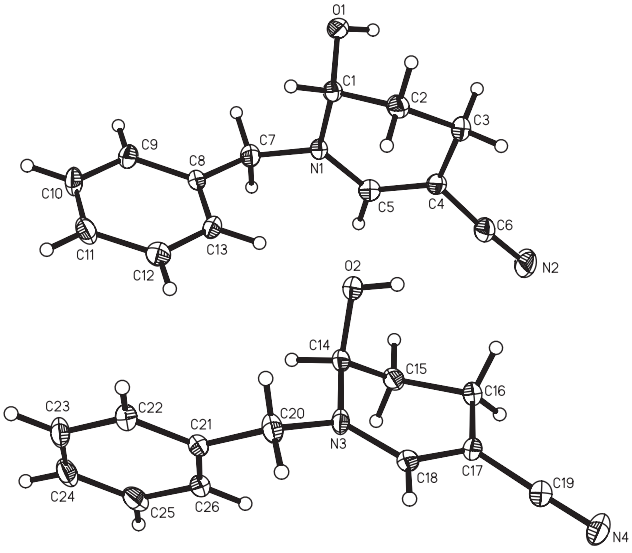
Crystal structure of 1-benzyl-6-hydroxy-1,4,5,6-tetrahydropyridine-3-carbonitrile, $C_{13}H_{14}N_2O$



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Abstract

$C_{13}H_{14}N_2O$, monoclinic, $P2_1/c$ (no. 14), $a = 9.933(2)$ Å, $b = 22.120(6)$ Å, $c = 10.487(3)$ Å, $\beta = 90.160(7)°$, $V = 2304.2(10)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0452$, $wR_{ref}(F^2) = 0.0808$, $T = 293$ K.

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

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Table 1: Data collection and handling.

Crystal:	Colourless prism Size 0.22 × 0.16 × 0.14 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.8 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	55.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10566, 5314, 0.045
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4278
$N(param)_{refined}$:	292
Programs:	SHELX [7], Platon [8], Bruker programs [9]

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

Atom	x	y	z	U_{iso}*/U_{eq}
O1	0.02948(10)	0.10207(4)	0.11240(15)	0.0423(4)
H1	0.0251	0.1323	0.0674	0.063*
N1	0.25880(12)	0.08562(5)	0.06535(16)	0.0335(4)
N2	0.47598(15)	0.27269(6)	−0.0328(2)	0.0600(6)
C1	0.16171(15)	0.09687(6)	0.1644(2)	0.0341(4)
H1A	0.1617	0.0613	0.2201	0.041*
C2	0.20403(16)	0.15062(6)	0.2461(2)	0.0363(4)
H2A	0.2839	0.1400	0.2946	0.044*
H2B	0.1328	0.1600	0.3061	0.044*
C3	0.23351(16)	0.20657(6)	0.1644(2)	0.0359(4)
H3A	0.1504	0.2222	0.1284	0.043*
H3B	0.2738	0.2380	0.2167	0.043*
C4	0.32733(14)	0.18915(6)	0.06064(19)	0.0325(4)
C6	0.41190(16)	0.23385(6)	0.0064(2)	0.0435(5)
C7	0.26475(15)	0.02442(6)	0.0132(2)	0.0369(5)
H7A	0.3186	0.0248	−0.0638	0.044*
H7B	0.1745	0.0116	−0.0100	0.044*
C8	0.32431(14)	−0.02088(6)	0.10621(18)	0.0289(4)
C9	0.27866(16)	−0.08053(6)	0.1045(2)	0.0367(5)
H9	0.2098	−0.0920	0.0493	0.044*
C10	0.33686(18)	−0.12276(6)	0.1861(2)	0.0424(5)
H10	0.3066	−0.1625	0.1841	0.051*
C11	0.43769(17)	−0.10720(7)	0.2693(2)	0.0409(5)
H11	0.4766	−0.1362	0.3220	0.049*
C12	0.48094(16)	−0.04761(7)	0.2739(2)	0.0424(5)
H12	0.5477	−0.0362	0.3314	0.051*
C13	0.42422(15)	−0.00517(6)	0.1925(2)	0.0383(5)
H13	0.4539	0.0347	0.1959	0.046*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
O2	0.54885(10)	0.11088(4)	0.36762(14)	0.0396(3)
H2	0.5431	0.1457	0.3942	0.059*
N3	0.78378(13)	0.10053(5)	0.40665(17)	0.0344(4)
N4	0.96678(15)	0.29821(6)	0.44811(19)	0.0493(5)
C14	0.67625(15)	0.10210(6)	0.31167(19)	0.0324(4)
H14	0.6743	0.0622	0.2713	0.039*
C15	0.70930(15)	0.14720(6)	0.20766(19)	0.0318(4)
H15A	0.6347	0.1493	0.1479	0.038*
H15B	0.7881	0.1335	0.1615	0.038*
C16	0.73652(15)	0.21051(6)	0.26224(19)	0.0334(4)
H16A	0.7748	0.2362	0.1968	0.040*
H16B	0.6528	0.2286	0.2906	0.040*
C17	0.83274(14)	0.20523(6)	0.37244(19)	0.0318(4)
C18	0.85038(14)	0.15228(6)	0.43636(19)	0.0322(4)
H18	0.9112	0.1515	0.5039	0.039*
C19	0.90812(15)	0.25621(7)	0.4151(2)	0.0378(5)
C20	0.79362(17)	0.04784(6)	0.4903(2)	0.0388(5)
H20A	0.7092	0.0430	0.5355	0.047*
H20B	0.8635	0.0550	0.5533	0.047*
C21	0.82497(14)	−0.01034(6)	0.41974(19)	0.0300(4)
C22	0.77542(15)	−0.06441(6)	0.4689(2)	0.0349(4)
H22	0.7239	−0.0641	0.5428	0.042*
C23	0.80265(18)	−0.11878(6)	0.4079(2)	0.0415(5)
H23	0.7692	−0.1547	0.4410	0.050*
C24	0.87789(17)	−0.11962(7)	0.2999(2)	0.0395(5)
H24	0.8955	−0.1561	0.2593	0.047*
C25	0.92858(16)	−0.06626(7)	0.2501(2)	0.0408(5)
H25	0.9804	−0.0667	0.1764	0.049*
C26	0.90077(15)	−0.01157(6)	0.3123(2)	0.0347(5)
H26	0.9347	0.0244	0.2794	0.042*
C5	0.33446(14)	0.13107(6)	0.0181(2)	0.0341(4)
H5	0.3947	0.1222	−0.0470	0.041*

Source of material

The title compound was synthesized by 1-benzyl-3-cyano-1,4-dihydropyridine under irradiation of UV-light in methanol solution. After approximately 24 h, followed by thin-layer chromatography, the residue was purified by flash chromatography using ethyl acetate-petroleum ether (1:10 v/v). After concentration in vacuo, colorless solids were collected and dried, and recrystallized from THF. After 3 days colorless crystals suitable for X-ray diffraction were obtained.

Experimental details

All H atoms were placed in idealized positions (C—H = 0.93–0.98 Å, O—H = 0.82 Å) and refined as riding atoms. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for oxygen H atoms and $1.2U_{eq}$ for the remaining H atoms. Pseudo-merohedral twinning in the crystal was identified [TwinRotMat [8]; twin law −1 0 0, 0 −1 0, 0 0 1] giving a final twin ratio of 0.500(1)/0.500(1).

Discussion

Tetrahydropyridines are a class of interesting compounds because they play important roles in synthetic, therapeutic, and bioorganic chemistry [1–6]. In order to search for new tetrahydropyridines, the title compound was synthesized and its crystal structure determined.

The asymmetric unit of the title compound consists of two independent and conformationally similar molecules. All bond lengths and angles are in the expected ranges. The tetrahydro-pyridine ring adopts a half-chair conformation. In the crystal, O—H···N hydrogen bonds generate chains along the *a*-axis direction.

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