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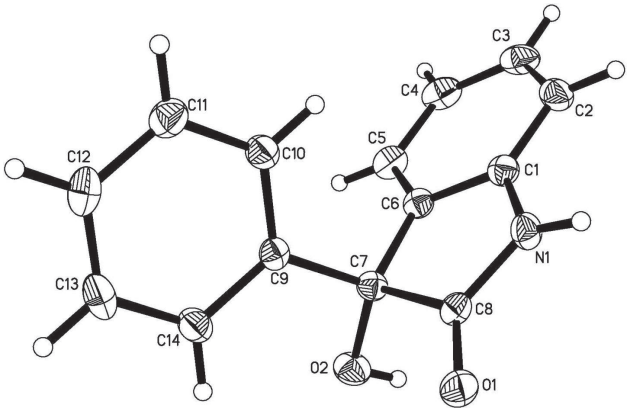
Crystal structure of 3-hydroxy-3-phenyl-1,3-dihydro-2*H*-indol-2-one, $C_{14}H_{11}NO_2$



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Abstract

$C_{14}H_{11}NO_2$, orthorhombic, *Pbca*, $a = 10.3868(9)$ Å, $b = 7.7695(7)$ Å, $c = 26.576(2)$ Å, $V = 2144.7(3)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0348$, $wR_{ref}(F^2) = 0.0911$, $T = 298(2)$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.

Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.15×0.21×0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.94 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II diffractometer, φ and ω scans
$2\theta_{max}$:	50.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8413, 1989
$N(param)_{refined}$:	155
Programs:	SHELX [2], PLATON [3], Bruker programs [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.9486	0.8544	0.5093	0.041
H(2)	8*c*	1.2527	0.7014	0.6056	0.062
H(2A)	8*c*	0.8293	0.5414	0.5051	0.047
H(3)	8*c*	0.8133	0.2808	0.5483	0.054
H(4)	8*c*	0.9230	0.2354	0.6217	0.054
H(5)	8*c*	1.0494	0.4517	0.6565	0.046
H(10)	8*c*	0.8408	0.8086	0.6417	0.041
H(11)	8*c*	0.7396	0.9731	0.7023	0.051
H(12)	8*c*	0.8588	1.1512	0.7539	0.055
H(13)	8*c*	1.0791	1.1649	0.7446	0.056
H(14)	8*c*	1.1820	1.0003	0.6842	0.044

Source of material

The title product was obtained by a known method [1]. Under a nitrogen atmosphere, indolin-2-one (0.65 mmol), NHC-Pd(II)-Im complex (1.0 mol%), potassium tertiary butoxide (4.0 equiv), toluene (1.0 mL) and chlorobenzene (0.5 mmol) were successively added. The mixture was stirred vigorously at 100°C for 12 h. Then the mixture was cooled to room temperature and stirred under air atmosphere for another 3 h. The solvent was removed under reduced pressure and the residue was purifed by flash column chromatography to give the pure product in 72% yield. Crystal suitable for X-ray diffraction was grown in a mixture of ethyl acetate and petroleum ether.

Experimental details

All hydrogen atoms were introduced using the HFIX command in the SHELXL program [2]. The N—H distance was restrained to 0.86 Å with U_{iso} values to be $1.2U_{eq}$(N). The O—H distance was restrained to 0.82 Å with U_{iso} values to be $1.5U_{eq}$(O). All other aromatic C—H distances were restrained to 0.93 Å with U_{iso} values to be $1.2U_{eq}$(C).

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

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	8*c*	0.9816(1)	0.8123(1)	0.53632(4)	0.0410(6)	0.0339(6)	0.0269(5)	−0.0005(5)	−0.0048(4)	0.0040(4)
O(1)	8*c*	1.12093(9)	1.0324(1)	0.55566(3)	0.0464(6)	0.0338(5)	0.0365(5)	−0.0073(4)	0.0026(4)	0.0054(4)
O(2)	8*c*	1.21913(8)	0.7669(1)	0.62590(4)	0.0277(5)	0.0461(6)	0.0493(6)	0.0050(4)	−0.0047(4)	−0.0034(4)
C(1)	8*c*	0.9519(1)	0.6478(2)	0.55575(5)	0.0295(6)	0.0297(6)	0.0328(6)	0.0023(5)	0.0032(5)	−0.0035(5)
C(2)	8*c*	0.8738(1)	0.5231(2)	0.53504(5)	0.0330(7)	0.0437(8)	0.0420(7)	−0.0007(6)	−0.0006(6)	−0.0126(6)
C(3)	8*c*	0.8648(1)	0.3682(2)	0.56115(6)	0.0379(8)	0.0344(7)	0.064(1)	−0.0080(6)	0.0114(7)	−0.0153(7)
C(4)	8*c*	0.9301(2)	0.3410(2)	0.60551(6)	0.0456(8)	0.0271(7)	0.0620(9)	−0.0006(6)	0.0142(7)	0.0018(6)
C(5)	8*c*	1.0064(1)	0.4691(2)	0.62627(5)	0.0400(7)	0.0316(7)	0.0432(8)	0.0049(6)	0.0045(6)	0.0050(5)
C(6)	8*c*	1.0166(1)	0.6232(2)	0.60078(5)	0.0287(6)	0.0270(6)	0.0324(6)	0.0035(5)	0.0034(5)	−0.0018(5)
C(7)	8*c*	1.0876(1)	0.7867(2)	0.61411(5)	0.0268(6)	0.0288(6)	0.0298(6)	0.0006(5)	−0.0019(5)	0.0019(5)
C(8)	8*c*	1.0678(1)	0.8948(2)	0.56539(4)	0.0305(6)	0.0291(6)	0.0281(6)	0.0020(5)	0.0049(5)	−0.0003(5)
C(9)	8*c*	1.0221(1)	0.8866(1)	0.65653(4)	0.0333(6)	0.0255(6)	0.0244(6)	0.0004(5)	−0.0021(5)	0.0049(5)
C(10)	8*c*	0.8894(1)	0.8800(2)	0.66238(5)	0.0338(7)	0.0345(7)	0.0353(7)	−0.0014(5)	−0.0015(5)	−0.0038(5)
C(11)	8*c*	0.8286(1)	0.9787(2)	0.69867(5)	0.0401(7)	0.0428(8)	0.0437(7)	0.0039(6)	0.0070(6)	−0.0030(6)
C(12)	8*c*	0.8996(2)	1.0850(2)	0.72946(5)	0.062(1)	0.0419(8)	0.0335(7)	0.0080(7)	0.0051(7)	−0.0062(6)
C(13)	8*c*	1.0311(2)	1.0928(2)	0.72386(5)	0.062(1)	0.0417(8)	0.0360(7)	−0.0043(7)	−0.0092(7)	−0.0091(6)
C(14)	8*c*	1.0930(1)	0.9942(2)	0.68762(5)	0.0377(7)	0.0387(7)	0.0346(7)	−0.0054(6)	−0.0057(5)	−0.0004(5)

Discussion

3-Substituted 3-hydroxyoxindoles are attractive compounds in organic synthesis because they are frequently found in many alkaloids, some natural substances and in pharmaceutically and biologically active compounds [5–7]. The bond lengths and angles derived from the title structure are within normal ranges. Adjacent molecules are connected by O—H···O hydrogen bonds [3.0925(13) Å], and N—H···O hydrogen bonds [2.9267(14) Å], which may stabilize the crystal structure.

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