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The crystal structure of 1,1'-bisisoquinoline, $C_{18}H_{12}N_2$

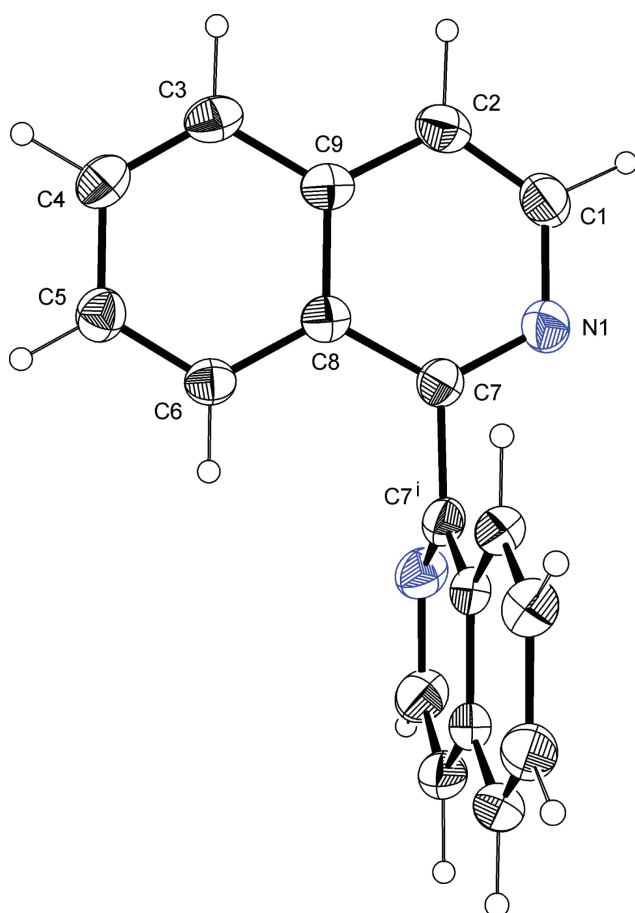


Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	$0.64 \times 0.58 \times 0.32$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.8 cm^{-1}
Diffractometer, scan mode:	STOE IPDS 2, ω scans
$2\theta_{\text{max}}$, completeness:	50° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8390, 1164, 0.055
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 927
$N(\text{param})_{\text{refined}}$:	116
Programs:	SHELX [1], WinGX [2], DIAMOND [3], PLATON [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.18787(9)	0.82972(9)	0.16368(10)	0.0612(4)
H1	0.2126(10)	0.8789(10)	0.2112(10)	0.071(4)*
C2	0.24871(9)	0.77946(8)	0.10507(10)	0.0553(3)
H2	0.3242(9)	0.7896(9)	0.1085(9)	0.058(3)*
C3	0.26831(9)	0.65245(8)	−0.02158(9)	0.0530(3)
H3	0.3387(10)	0.6630(9)	−0.0206(8)	0.059(3)*
C4	0.22713(9)	0.58602(9)	−0.08111(9)	0.0551(3)
H4	0.2675(10)	0.5459(10)	−0.1238(10)	0.068(4)*
C5	0.12673(9)	0.57268(9)	−0.08217(9)	0.0524(3)
H5	0.0986(9)	0.5240(9)	−0.1231(10)	0.058(3)*
C6	0.06914(9)	0.62691(8)	−0.02349(8)	0.0466(3)
H6	0.0012(9)	0.6174(9)	−0.0235(8)	0.052(3)*
C7	0.05365(8)	0.75473(7)	0.10355(8)	0.0455(3)
C8	0.10969(7)	0.69667(7)	0.03968(8)	0.0420(3)
C9	0.21067(7)	0.70946(7)	0.04073(8)	0.0457(3)
N1	0.09037(7)	0.81878(7)	0.16407(8)	0.0574(3)

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Abstract

$C_{18}H_{12}N_2$, tetragonal, $I4_1/a$ (no. 88), $a = 13.8885(6)$ Å, $c = 13.6718(6)$ Å, $V = 2637.2(3)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0295$, $wR_{\text{ref}}(F^2) = 0.0854$, $T = 210$ K.

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The molecular structure of the title compound 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.

Source of materials

The title compound has been obtained by nickel-catalyzed C–C coupling of 1-chlorisoquinoline [5], which was synthesized by chlorination of isoquinoline-*N*-oxid with POCl₃ [6].

The *N*-oxid was prepared by reaction of isoquinoline with H₂O₂ [7]. Yellow crystals (Mp. 162–163 °C) suitable for single-crystal X-ray diffraction were obtained from diethyl ether by slow evaporation within 3 days.

Experimental details

Coordinates of hydrogen atoms were refined without any constraints or restraints.

Comment

The title compound is a well-known ligand in the coordination chemistry [8–13]. In most complexes 1,1'-bisisoquinoline acts as bidentate ligand, but can also occur as bridging ligand in binuclear complexes. The latter ones are also observed in luminescent silver(I) complexes [14], whereas in gold(I) complexes both types of complexes are formed [15]. Optically active complexes with high chiral recognition can be formed by reaction of 1,1'-bisisoquinoline with chiral palladium complexes [16]. Pd(II) and Ni(II) complexes of 1,1'-bisisoquinolines can be used as catalysts for Suzuki and Heck reactions [17]. 1,1'-Bisisoquinolinium salts exhibit chemiluminescence on addition of hydrogen peroxide in alkaline solution [18].

The title structure shows C₂ symmetry. The asymmetric unit contains half a molecule. Both quinoline moieties are related by a $\bar{4}$ axis, located between the bridging atoms (C7 and C7') running along the crystallographic *c* direction. The quinoline ring system is nearly full planar with a maximal deviation of the best plane of 0.012(1) Å (C1). The length of the C7–C7' bond is 1.496(2) Å. The main structural feature is the position of the quinoline subunits being almost perpendicular to each other [88.54(1)°], which is caused by the transannular steric effect of the hydrogen atoms in the 8-position (labelled here with H6). For enhancing the transannular steric hindrance a series of 8,8'-dialkylsubstituted 1,1-bisisoquinolines were synthesized, whereby dihedral angles between both isoquinoline moieties of 77.68° (Me) and 86.27° (Et) can be observed [19]. Introducing ethoxy substituents in the 4,4'-positions leads to a dihedral angle of 66.20° [20]. During complexation reaction the isoquinoline subunits can be forced into a more planar position depending on the requirements of the metal centre [13]. In presence of other suitable ligands, which also can complete the coordination sphere the twisted bisisoquinoline structure is only slightly changed and bisisoquinoline often acts as bridging ligand and binuclear complexes are formed [16]. The crystal packing is characterized by a large number of π – π interactions. Furthermore, C–H... π interactions are observed [C4–H4...C3–C6/C8/C9 ring].

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