

N*-(8-Quinolyl)-*o*-phenylenediamine*Tarimala Seshadri, Ulrich Flörke* and Gerald Henkel**

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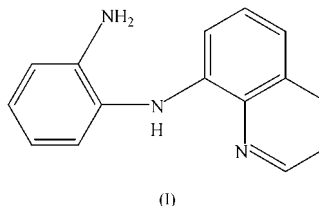
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Key indicatorsSingle-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.043
 wR factor = 0.107
Data-to-parameter ratio = 9.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The crystal packing of the title compound, $\text{C}_{15}\text{H}_{13}\text{N}_3$, is determined by intermolecular $\text{N}-\text{H}\cdots\text{N}$ interactions, resulting in parallel rows of molecules along [100].

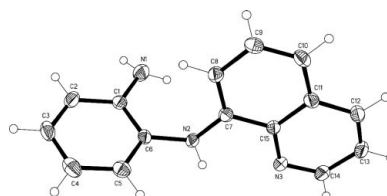
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CommentStudies related to the complex-forming properties of 8-aminoquinoline and its derivatives are very rare. Although *N*-(8-quinolyl)-*o*-phenylenediamine (8-Q-phen) is a very interesting tridentate ligand which was synthesized by Jensen & Nielsen (1964), none of its donating properties have been explored, except for a Ni^{II} complex (Okada *et al.*, 2001). A Pt complex with *o*-dimethylaminophenyl(8-quinolyl)amine as ligand has been studied by Peters *et al.* (2001). The primary amino group in the ligand 8-Q-phen can undergo several substitution reactions and a variety of Schiff base derivatives can be prepared to obtain hitherto unknown hexa- and octadentate ligands which provide access to multinuclear metal complexes.

In the present study, we report the crystal structure of the parent title compound, (I). In (I) (Fig. 1), the planes of the substituted phenyl ring C1–C6 and the quinolyl system N3/C7–C15 are almost perpendicular, with a dihedral angle of $76.95(6)^\circ$; the torsion angle C7–N2–C6–C5 is $-95.6(2)^\circ$. The C6–N2–C7 angle at sp^3 atom N2 is $121.4(2)^\circ$ and the bond lengths N1–C1 [$1.381(3)$ Å], C1–C6 [$1.406(3)$ Å], C6–N2 [$1.424(3)$ Å] and N2–C7 [$1.385(2)$ Å] lie in the expected ranges, similar to those of phenylethylaminoquinoline (Lynch & McClenaghan, 2001) and diphenylphosphido-methylaminoquinoline (Clarke *et al.*, 2002). Intramolecular hydrogen bonds are formed: N2–H2 \cdots N3 with $\text{H}\cdots\text{N} = 2.25(2)$ Å and $\text{N}-\text{H}\cdots\text{N} = 106(2)^\circ$, and N1–H1A \cdots N2 with $\text{H}\cdots\text{N} = 2.43(2)$ Å and $\text{N}-\text{H}\cdots\text{N} = 100(2)^\circ$.

**Figure 1**

The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

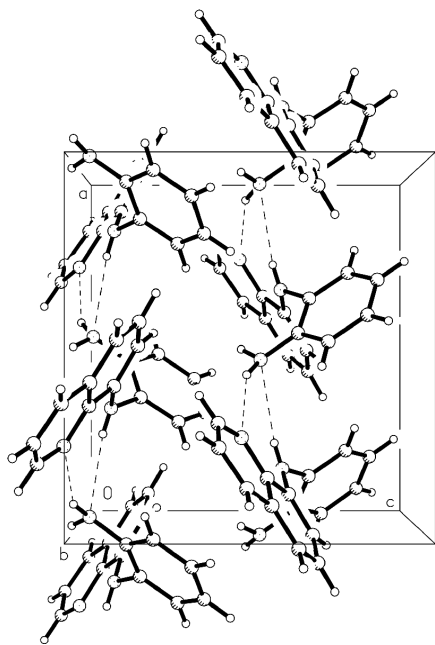


Figure 2
Crystal packing of (I), viewed along [010], with the hydrogen-bonding pattern indicated by dashed lines.

The crystal packing (Fig. 2) shows two intermolecular H \cdots N interactions [N1—H1A \cdots N2 ($x - \frac{1}{2}, 1 - y, z$) with H \cdots N = 2.23 (2) Å and N—H \cdots N = 151 (2)°, and N2—H2 \cdots N1 ($x + \frac{1}{2}, 1 - y, z$) with H \cdots N = 2.29 (3) Å and N—H \cdots N = 164 (2)°] which link the molecules in parallel rows along [100]. These values are normalized for N—H = 1.03 Å, whereas the values in Table 2 are those obtained in the refinement and uncorrected.

Studies are now in progress to examine the coordination properties of this ligand as well as its Schiff bases.

Experimental

Compound (I) was prepared by following the modified Bucherer reaction method of Jensen & Nielsen (1964). Crystals suitable for diffraction analysis were grown by recrystallization from acetonitrile. ^1H NMR (500 MHz, CDCl_3 , δ): 8.82 (*d*, 1H, 4 Hz), 8.13 (*d*, 1H, 8.3 Hz), 7.67 (*s*, 1H), 7.46–7.10 (*m*, 4H), 6.91–6.81 (*m*, 3H), 3.93 (*s*, 2H); IR (KBr, ν [cm^{-1}]): 3444 (*s*), 3326 (*s*), 1616 (*s*), 1569, 1498, 819, 789 (*s*), 756.

Crystal data

$\text{C}_{15}\text{H}_{13}\text{N}_3$
 $M_r = 235.28$
Orthorhombic, $Pca2_1$
 $a = 10.7227$ (13) Å
 $b = 11.0789$ (13) Å
 $c = 10.1268$ (12) Å
 $V = 1203.0$ (2) Å 3
 $Z = 4$
 $D_x = 1.299$ Mg m $^{-3}$

Mo $K\alpha$ radiation
Cell parameters from 1682 reflections
 $\theta = 2.6$ – 23.2°
 $\mu = 0.08$ mm $^{-1}$
 $T = 120$ (2) K
Plate, colorless
 $0.50 \times 0.45 \times 0.02$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.941$, $T_{\max} = 0.989$
7244 measured reflections

1584 independent reflections
1409 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.059$
 $\theta_{\max} = 28.3^\circ$
 $h = -8 \rightarrow 14$
 $k = -14 \rightarrow 13$
 $l = -12 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.107$
 $S = 1.11$
1584 reflections
174 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0655P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28$ e Å $^{-3}$
 $\Delta\rho_{\min} = -0.20$ e Å $^{-3}$

Table 1
Hydrogen-bonding geometry (Å, °).

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
N1—H1A \cdots N2	0.86 (3)	2.47 (3)	2.806 (3)	104 (3)
N2—H2 \cdots N3	0.81 (4)	2.32 (3)	2.719 (2)	111 (2)
N1—H1B \cdots N3 ⁱ	0.97 (3)	2.29 (3)	3.175 (3)	150 (2)
N2—H2 \cdots N1 ⁱⁱ	0.81 (4)	2.51 (4)	3.298 (3)	166 (3)

Symmetry codes: (i) $x - \frac{1}{2}, 1 - y, z$; (ii) $\frac{1}{2} + x, 1 - y, z$.

H atoms attached to C were placed at calculated positions, riding on their carrier atoms, with isotropic displacement parameters $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Positional and isotropic displacement parameters of H atoms bonded to N atoms were refined freely; a common U_{iso} value was refined for atoms H1A and H1B. The title compound crystallizes in the non-centrosymmetric space group $Pca2_1$; however, in the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Accordingly, the Friedel pairs were merged.

Data collection: SMART (Bruker, 2002); cell refinement: SMART; data reduction: SAINT (Bruker, 2002); program(s) used to solve structure: SHELXTL (Bruker, 2002); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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