

## 2-Phenylquinoline 1-oxide

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## Key indicators

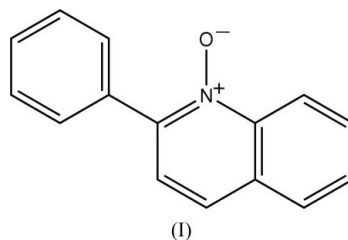
Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.041  
 $wR$  factor = 0.115  
Data-to-parameter ratio = 9.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{15}\text{H}_{11}\text{NO}$ , is not planar, as seen in the dihedral angle of  $41.92(12)^\circ$  formed between the quinoline and phenyl residues. This conformation allows the crystal structure to be stabilized by  $\text{C}-\text{H}\cdots\text{O}$  interactions. Zigzag chains along the  $a$  direction, mediated by  $\text{C}-\text{H}\cdots\text{O}$  interactions, may be discerned in the crystal structure and these stack along the  $c$  direction, again *via*  $\text{C}-\text{H}\cdots\text{O}$  interactions. Interactions of the type  $\text{C}-\text{H}\cdots\pi$  serve to stabilize the zigzag chains and to provide links between them.

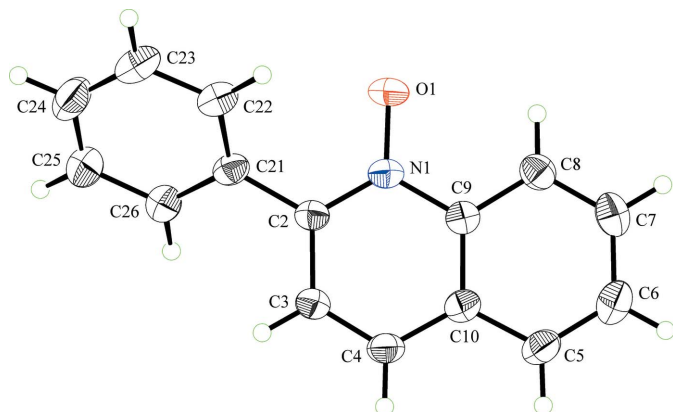
Received 3 August 2006  
Accepted 7 August 2006

## Comment

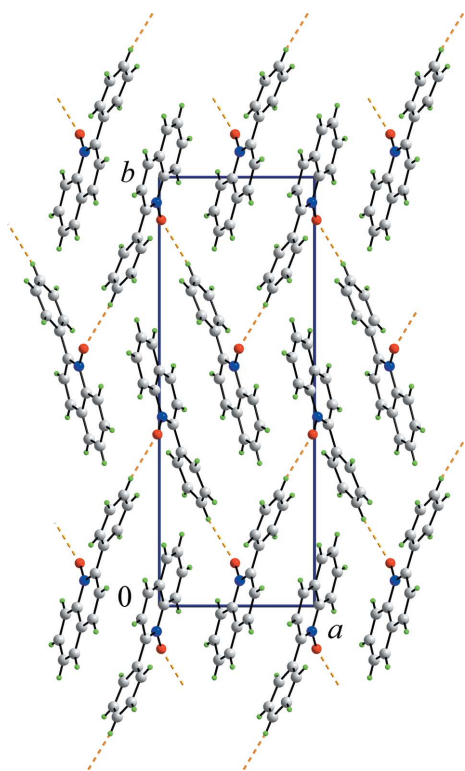
Quinolines substituted at the 2-position exhibit *in vivo* activity against *Leishmania infantum* and *Leishmania donovani* (Nakayama *et al.*, 2005). The title compound, (I), was synthesized as part of the generation of a series of 2-substituted quinolines and their corresponding *N*-oxides for testing as chemotherapeutic agents against this tropical parasite. This compound has been previously cocrystallized with 7,7,8,8-tetracyanoquinodimethane and its structure reported (Bocelli *et al.*, 1990). The molecule of (I) (Fig. 1) is not planar, as seen in the dihedral angle of  $41.92(12)^\circ$  formed between the least-squares planes of the quinoline and phenyl residues. The geometric parameters (Table 1) are as expected (Allen *et al.*, 1987). The observed conformation in (I) no doubt allows for the close approach of two H atoms, derived from two different molecules, to atom O1, leading to the formation of  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds. The first of these leads to the formation of



zigzag chains parallel to the  $a$  direction as shown in Fig. 2. The parameters associated with this association are  $\text{C}24-\text{H}24\cdots\text{O}1^i$  of 2.58 Å,  $\text{C}24\cdots\text{O}1^i$  of 3.471 (4) Å, with an angle at H24 of  $161^\circ$  [symmetry code: (i)  $-\frac{1}{2} + x, -\frac{1}{2} - y, 1 - z$ ]. Chains are stacked along the  $c$  direction *via*  $\text{C}26-\text{H}26\cdots\text{O}1^{ii}$  interactions so that  $\text{C}26-\text{H}\cdots\text{O}1^{ii}$  is 2.54 Å,  $\text{C}26\cdots\text{O}1^{ii}$  is 3.423 (4) Å and the angle at H26 is  $158^\circ$  [symmetry code: (ii)  $x, y, 1 + z$ ]. Additional stabilization in the crystal structure is afforded by  $\text{C}-\text{H}\cdots\pi$  interactions. Thus, the  $\text{N}1/\text{C}2-\text{C}4/\text{C}9/\text{C}10$  rings are linked so that the  $\text{C}3-\text{H}3\cdots$ [ring centroid of  $(\text{N}1/\text{C}2-\text{C}4/\text{C}9/\text{C}10)^i$ ] distance is 2.89 Å with an angle at H of  $126^\circ$ . As indicated by the angle at atom H3, this atom is more



**Figure 1**  
The molecular structure of (I) showing the crystallographic numbering scheme and displacement ellipsoids at the 35% probability level.



**Figure 2**  
View of the zigzag chains in (I) formed by C—H...O interactions (dashed lines). Colour code: O (red), N (blue), C (grey) & H (green).

closely associated with atoms C4, C9 and C10 of the ring. These interactions serve to provide links between the zigzag chains along the *b* direction. The phenyl rings are also connected *via* C—H... $\pi$  interactions so that the distance between C23—H23 and the ring centroid of (C21—C26)<sup>iii</sup> is 2.95 Å with an angle of 143° [(iii)  $-\frac{1}{2} - x, -y, \frac{1}{2} + z$ ]. These interactions provide stability within the zigzag chains.

## Experimental

The procedure involved the addition of phenyl Grignard to quinoline-*N*-oxide. This yielded both the expected 2-phenylquinoline and, unexpectedly, (I). It is not clear whether the *N*-oxide survives the reaction or if the product is re-oxidized subsequent to arylation.

Magnesium turnings (0.45 g, 18.4 mmol) were activated with a small crystal of iodine and bromobenzene (2.72 g, 17.2 mmol) in dry THF (5.7 ml) was added slowly over a period of 30 min under a nitrogen atmosphere. When most of the magnesium had dissolved, quinoline-*N*-oxide (0.5 g, 3.44 mmol) in dry THF (5.0 ml) was added over a period of 15 min and the reaction was refluxed for 1 h. After this period, the reaction was quenched with water and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 20 ml), and the combined organic phases washed with brine, dried (MgSO<sub>4</sub>) and condensed. The crude product was subjected to flash column chromatography on silica with a solvent gradient from neat *n*-hexane to *n*-hexane/EtOAc (4:1) to provide 2-phenylquinoline (289 mg, 41% yield) and 2-phenylquinoline-*N*-oxide (120 mg, 16% yield). The latter was crystallized from hexane/ethyl acetate (4:1) overnight at room temperature to provide pale-yellow block-shaped crystals of (I) [m.p. 315–316 K; literature 315–316 K (Endo *et al.*, 1981)].

### Crystal data

C <sub>15</sub> H <sub>11</sub> NO	Z = 4
<i>M<sub>r</sub></i> = 221.25	<i>D<sub>x</sub></i> = 1.339 Mg m <sup>-3</sup>
Orthorhombic, <i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	Mo <i>K</i> α radiation
<i>a</i> = 7.754 (3) Å	$\mu$ = 0.08 mm <sup>-1</sup>
<i>b</i> = 21.436 (6) Å	<i>T</i> = 293 (2) K
<i>c</i> = 6.603 (2) Å	Block, pale yellow
<i>V</i> = 1097.5 (6) Å <sup>3</sup>	0.50 × 0.30 × 0.20 mm

### Data collection

Rigaku AFC-7R diffractometer	<i>R</i> <sub>int</sub> = 0.011
$\omega$ scans	$\theta$ <sub>max</sub> = 27.5°
Absorption correction: none	3 standard reflections
1772 measured reflections	every 150 reflections
1486 independent reflections	intensity decay: 1.5%
996 reflections with <i>I</i> > 2σ( <i>I</i> )	

### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0354P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.115$	( $\Delta\sigma$ ) <sub>max</sub> < 0.001
<i>S</i> = 1.04	$\Delta\rho$ <sub>max</sub> = 0.16 e Å <sup>-3</sup>
1486 reflections	$\Delta\rho$ <sub>min</sub> = -0.23 e Å <sup>-3</sup>
154 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters (Å, °).

O1—N1	1.301 (3)	N1—C9	1.411 (3)
N1—C2	1.352 (3)		
O1—N1—C2	121.5 (2)	C2—N1—C9	120.6 (2)
O1—N1—C9	117.8 (2)		

H atoms were included in the riding-model approximation with distances C—H = 0.93 Å and with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C). In the absence of significant anomalous scattering effects, 67 Friedel pairs were averaged in the final refinement.

Data collection: *MSC/AFC7 Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC7 Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *TEXSAN for Windows*.

The authors thank the Australian Academy of Science for funding to allow DJY to visit UTSA.

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