2-phenylquinoline 1-oxide

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Published
2006

Journal Title
Acta crystallographica. Section E, Structure reports online

DOI
https://doi.org/10.1107/S1600536806030935

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2-Phenylquinoline 1-oxide

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

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 co-crystallized 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)° 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 C—H···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 C24–H24···O1i of 2.58 Å, C24···O1i of 3.471 (4) Å, with an angle at H24 of 161° [symmetry code: (i) x−1/2, y, 1−z]. Chains are stacked along the c direction via C26···O1ii interactions so that C26–H···O1ii is 2.54 Å, C26···O1ii is 3.423 (4) Å and the angle at H26 is 158° [symmetry code: (ii) x, y, 1+z]. Additional stabilization in the crystal structure is afforded by C—H···π interactions. Thus, the N1/C2–C4/C9/C10 rings are linked so that the C3–H···[ring centroid of (N1/C2–C4/C9/C10)] distance is 2.89 Å with an angle at H of 126°. As indicated by the angle at atom H3, this atom is more
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 CH2Cl2 (3 × 20 ml), and the combined organic phases washed with brine, dried (MgSO4) 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

\[ \text{C}_{15} \text{H}_{11} \text{NO} \]

\[ M_r = 221.25 \]

Orthorhombic, \( P2_1\overline{2}2_1 \)

\[ a = 7.754 (3) \text{ Å} \]

\[ b = 21.436 (6) \text{ Å} \]

\[ c = 6.603 (2) \text{ Å} \]

\[ V = 1097.5 (6) \text{ Å}^3 \]

\[ Z = 4 \]

\[ D_x = 1.339 \text{ Mg m}^{-3} \]

Mo Ka radiation

\[ \mu = 0.08 \text{ mm}^{-1} \]

\[ T = 293 (2) \text{ K} \]

Block, pale yellow

\[ 0.50 \times 0.30 \times 0.20 \text{ mm} \]

Data collection

Rigaku AFC-7R diffractometer

\( \omega \) scans

Absorption correction: none

1772 measured reflections

1486 independent reflections

996 reflections with \( I > 2\sigma(I) \)

Refinement

Refinement on \( F^2 \)

\[ R[F^2 > 2\sigma(F^2)] = 0.041 \]

\[ wR^2(F) = 0.115 \]

\( S = 1.04 \)

1486 reflections

154 parameters

H-atom parameters constrained

H atoms were included in the riding-model approximation with distances C–H = 0.93 Å and with \( U_{eq}(H) = 1.2U_{eq}(C) \). 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: ORTEPIII (Johnson, 1976) and DIAMOND (Crystal Impact, 2006); software used to prepare material for publication: TEXSAN for Windows.

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.

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 \( \text{via C—H· · ·π} \) interactions so that the distance between C23—H23 and the ring centroid of (C21–C26)\( ^{\text{II}} \) is 2.95 Å with an angle of 143° [\( (\text{iii}) \, -\frac{1}{2} + x, -y, \frac{1}{2} + z \)]. These interactions provide stability within the zigzag chains.
The authors thank the Australian Academy of Science for funding to allow DJY to visit UTSA.

References