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Acta Cryst. (1997). **C53**, 1862–1864

5,6,8-Trichloro-4-phenyl-3,4-dihydroquinolin-2(1H)-one

R. THIRUMURUGAN,^a S. SHANMUGA SUNDARA RAJ,^a
 P. MURUGAN,^b V. T. RAMAKRISHNAN^b AND
 K. K. CHACKO^a

^aDepartment of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and ^bDepartment of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India. E-mail: crystal@giasmd01.vsnl.net.in

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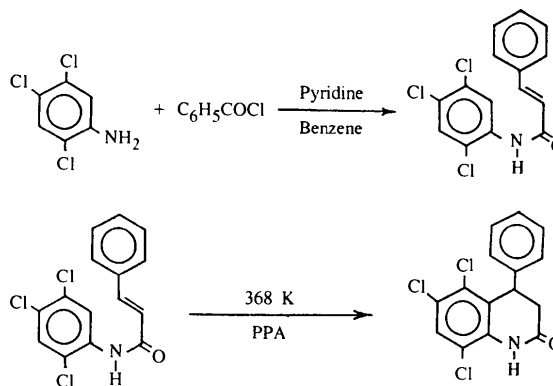
Abstract

In the quinolone ring system of the title compound, C₁₅H₁₀Cl₃NO, the pyridone moiety assumes a slightly twisted boat conformation. The plane of the benzene ring is twisted at an angle of 11.9(1)° with respect to the pyridone ring, indicating the slight non-planarity of the system. The molecules are stabilized by N—H···O-type hydrogen bonds in addition to van der Waals interactions.

Comment

The quinolones are a new class of antibiotics with therapeutic properties and potential attributable to their broad spectrum of antimicrobial activity (Fitton, 1992; McCarter, Mazens Sullivan & Bartlet, 1992). Quinolone antibacterial agents have been shown to be inhibitors of DNA gyrase, a bacterial topoisomerase II enzyme. Quinolones possess good activity *in vivo* against both

systemic tumour and subcutaneously implanted human tumour xenografts (Chu *et al.*, 1992). In the treatment of asthma, urinary infection and tuberculosis, quinolones play an important role (Dahl & Haahela, 1992; Reeves, Bywater, Holt & White, 1984).



An *ORTEPII* (Johnson, 1976) plot of the molecule with 50% probability displacement ellipsoids and the atomic numbering is shown in Fig. 1. When compared with the N—C_{yp} single-bond distance of 1.452 Å (Ammon, Mazzochi, Regan & Colicelli, 1979), the bond distances N1—C2 [1.394(4) Å] and N1—C9 [1.397(4) Å] are shorter, exhibiting the partial double-bond character (Chinnakali, Sivakumar, Natarajan, McGuire & Clearfield, 1991; Kido & Nakagawa, 1982). The C2—O1 bond length is 1.213(5) Å, indicative of a double bond, which is a unique feature in quinolone compounds (Baenzler & Wawzonek, 1984).

In the quinolone ring system, the pyridone moiety assumes a slightly twisted boat conformation. This kind of feature is uncommon in quinolone ring systems.

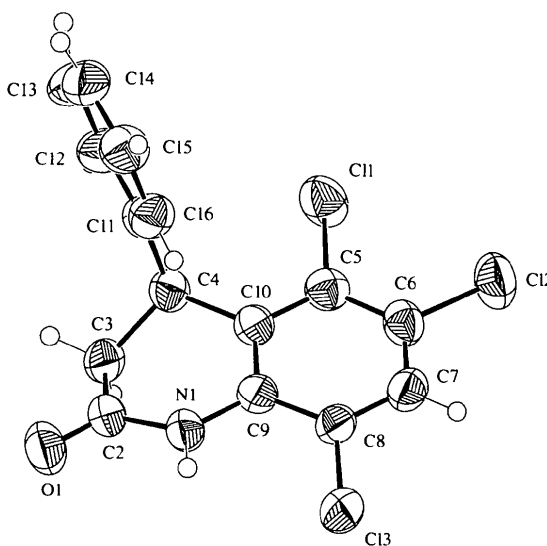


Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title molecule showing 50% probability displacement ellipsoids.

Usually the atoms in a quinolone ring system lie almost in the same plane (Sudha, 1996). The plane of the benzene ring is twisted at an angle of 11.9(1)° with respect to the pyridine ring, indicating the slight non-planarity of the system.

The phenyl ring also lies approximately on the plane of the quinolone ring system. The twist angle is only 82.9(2)°. The C—Cl bond lengths of 1.719(4), 1.738(4) and 1.724(3) Å are comparable with the literature value (Allen *et al.*, 1987).

The molecules are stabilized by N—H...O-type hydrogen bonds in addition to van der Waals interactions. The N1—H1...O1(−x, −y + 1, −z + 1) interaction is fairly linear as can be evidenced from the subtending angle of 165(4)° [N1—H1 0.89(5), N1...O1 2.980(3) and H1...O1 2.11(5) Å]. The intermolecular C—H...O hydrogen bonding observed between C15 and O1(x, y − 1, z) has dimensions C15—H15 0.91(4), C15...O1 3.652(5), H15...O1 2.79(4) Å and C15—H15...O1 159(3)°. The molecules are packed in aggregate fashion and extend in the c direction.

Experimental

The title compound was synthesized (see scheme) according to the method of Manimaran & Ramakrishnan (1979). To a solution of 2,4,5-trichloroaniline and pyridine in dry benzene, cinnamoyl chloride in dry benzene was added dropwise with stirring. After 1 h, the separated cinnamanilide derivative was filtered and dried. This was mixed with polyphosphoric acid (PPA) and heated at 368 K for 8 h. Crushed ice was added to the reaction mixture and the separated solid was filtered, dried and purified by chromatography over a column of silica gel; yield 58%.

Crystal data

C₁₅H₁₀Cl₃NO
M_r = 326.61
 Monoclinic
*P*2₁/*c*
a = 8.455(3) Å
b = 7.798(3) Å
c = 21.508(5) Å
 β = 91.37(2)°
V = 1417.7(8) Å³
Z = 4
D_x = 1.530 Mg m^{−3}
D_m not measured

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 ψ scan (North, Phillips
 & Mathews, 1968)
T_{min} = 0.222, *T_{max}* = 0.352
 2876 measured reflections
 2681 independent reflections

Cu K α radiation
 λ = 1.54178 Å
 Cell parameters from 25
 reflections
 θ = 15–25°
 μ = 0.5799 mm^{−1}
T = 293(2) K
 Prism
 0.30 × 0.24 × 0.18 mm
 Transparent

2456 reflections with
 $I > 2\sigma(I)$
R_{int} = 0.090
 θ_{\max} = 70°
h = 0 → 9
k = 0 → 9
l = −26 → 26
 3 standard reflections
 frequency: 120 min
 intensity decay: < 1.5%

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.073$
 $wR(F^2) = 0.220$
S = 1.080
 2658 reflections
 210 parameters
 All H atoms refined
 $w = 1/[\sigma^2(F_o^2) + (0.1583P)^2 + 0.5506P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = -0.550$
 $\Delta\rho_{\max} = 0.689 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.390 \text{ e \AA}^{-3}$
 Extinction correction:
 SHELXL93 (Sheldrick,
 1993)
 Extinction coefficient:
 0.0645
 Scattering factors from
 International Tables for
 Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

C11—C5	1.719(4)	C3—C4	1.555(5)
C12—C6	1.738(4)	C4—C10	1.535(5)
C13—C8	1.724(3)	C5—C10	1.382(5)
O1—C2	1.213(5)	C5—C6	1.402(5)
N1—C2	1.394(4)	C6—C7	1.359(5)
N1—C9	1.397(4)	C7—C8	1.369(5)
N1—H1	0.89(5)	C8—C9	1.407(5)
C2—C3	1.478(5)	C9—C10	1.386(5)
C2—N1—C9	123.9(3)	C5—C6—C12	121.3(3)
O1—C2—N1	121.9(3)	C6—C7—C8	118.2(3)
O1—C2—C3	122.2(3)	C7—C8—C9	122.8(3)
N1—C2—C3	115.8(3)	C7—C8—C13	117.8(3)
C2—C3—C4	114.7(3)	C9—C8—C13	119.4(3)
C10—C4—C3	109.6(3)	C10—C9—N1	120.6(3)
C10—C5—C6	121.0(3)	C10—C9—C8	118.4(3)
C10—C5—C11	119.3(3)	N1—C9—C8	121.0(3)
C6—C5—C11	119.7(3)	C5—C10—C9	118.9(3)
C7—C6—C5	120.6(3)	C5—C10—C4	121.8(3)
C7—C6—C12	118.1(3)	C9—C10—C4	119.3(3)

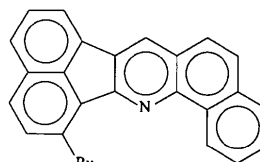
Data collection: *SDP* (Frenz, 1978). Cell refinement: *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1227). Services for accessing these data are described at the back of the journal.

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(I)

An ellipsoid plot of (I) with the atom-numbering scheme is shown in Fig. 1. The bond lengths and angles observed in this structure are normal. The aromatic ring system is planar with a dihedral angle of only 1.78 (3)° between the planes of the benzoquinoline and naphthalene moieties. The butyl group is perpendicular to the plane of the rest of the molecule. The N atom is involved in short intramolecular contacts with C3 [2.818 (2) Å] and C24 [3.225 (2) Å]. The crystal structure is stabilized by van der Waals interactions.

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6-*n*-Butylacenaphtho[1,2-*b*]benzo[*h*]-quinoline

JAYANTA KUMAR RAY,^a BIDHAN CHANDRA ROY,^a
A. CHAKRABORTY,^a KANDASAMY CHINNAKALI^{b†} AND
HOONG-KUN FUN^b

^aDepartment of Chemistry, Indian Institute of Technology, Kharagpur 721 302, India, and ^bX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: hkfun@usm.my

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Abstract

The molecule of the title compound, C₂₇H₂₁N, is planar except for the extended *n*-butyl chain which is perpendicular to the molecular plane. The crystal structure is stabilized by van der Waals interactions.

Comment

Butylation of polycyclic azaarenes (PAA) improves their solubility in common organic solvents and thus enhances their potential as substrates for molecular recognition. They are relatively rigid planar hosts for metal ions and organic molecules (Bell & Firestone, 1986). In an attempt to synthesize monoalkylated PAA with a butyl group exclusively in the semi-bay region of the acenaphthylene moiety, which is highly influenced by the position of the heteroatoms, we have developed a new route involving simple alkylolithiums (Ray, Roy & Kar, 1996). As part of studies on the synthesis, characterization and bioactivity of polycyclic aromatic hydrocarbon derivatives, the structure of the title compound, (I), has been determined.

† On leave from: Department of Physics, Anna University, Chennai 600 025, India.

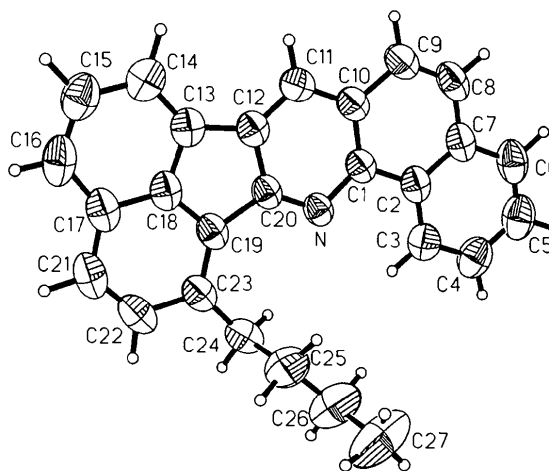


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Single crystals of the title compound were obtained by slow evaporation from a chloroform–tetrachloromethane mixture.

Crystal data

C₂₇H₂₁N
M_r = 359.45
Monoclinic
P2₁/c
a = 19.289 (4) Å
b = 6.0580 (10) Å
c = 16.792 (3) Å
β = 98.230 (10)°
V = 1942.0 (6) Å³
Z = 4
D_x = 1.229 Mg m⁻³
D_m not measured

Mo Kα radiation
λ = 0.71073 Å
Cell parameters from 43 reflections
θ = 5.3–12.6°
μ = 0.071 mm⁻¹
T = 293 (2) K
Rectangular slab cut from bigger crystal
0.98 × 0.54 × 0.36 mm
Yellow