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5-Dimethylamino-8-methyl-2-quinolone

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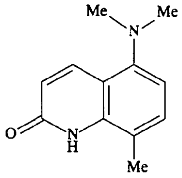
Abstract

The title compound, $C_{12}H_{14}N_2O$, is a new laser dye. The quinolone ring system is essentially planar. The hydrogen-bonding scheme involves N—H···O═C and C—H···N interactions.

Comment

Dye lasers are sources of coherent light that can be tuned over wide spectral regions from near infrared to ultraviolet. They are useful for the generation of ultrashort pulses and are also widely used in laser spectroscopy and photodynamic therapy (Schafer, 1977). The title compound, (1), is a laser dye, the laser performance of which is yet to be studied. Crystal structures of related compounds have been reported previously (Sudha, Subramanian, Sivaraman, Ramakrishnan, Steiner & Koellner, 1995; Sudha, Subramanian, Sivaraman, Sriraghavan & Steiner, 1995).



(1)

The conformation of (1) as found in the crystal structure is shown in Fig. 1. Bond lengths and angles in the quinolone ring system are normal (Sudha, Subramanian, Sivaraman, Ramakrishnan, Steiner & Koellner, 1995; Sudha, Subramanian, Sivaraman, Sriraghavan & Steiner, 1995). The angle between the best planes of the two rings is 4.5 (1)°. The angles C4—C10—C5 and C3—C2—O2 are greater than 120°, whereas C3—C2—N1 is smaller than 120°, as is commonly observed in quinolone derivatives (Chinnakali, Sivakumar, Natarajan, McGuire & Clearfield, 1991). The N11 atom of the

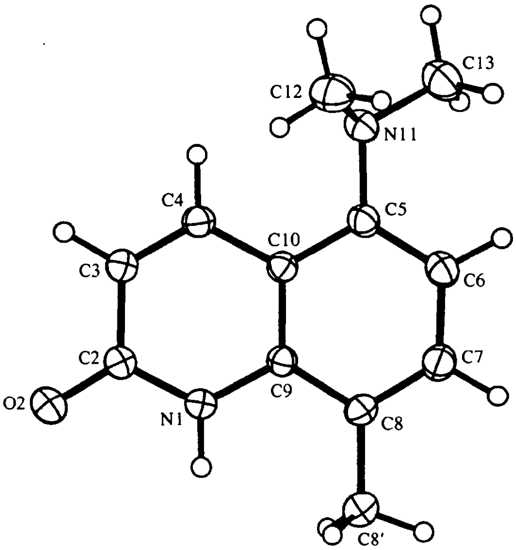


Fig. 1. The molecular structure and atom labelling of the title compound. Displacement ellipsoids are drawn at the 30% probability level.

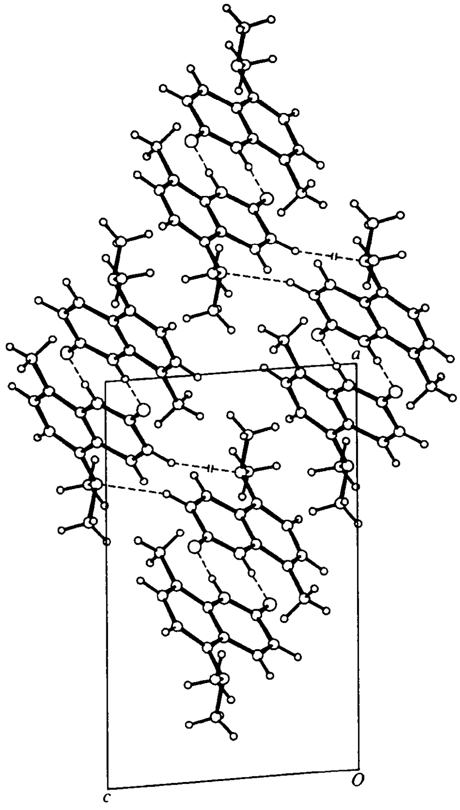


Fig. 2. Crystal-packing scheme and hydrogen-bonding pattern in the unit cell. Note that there are two distinct hydrogen-bond motifs: molecular dimers are joined by mutual N—H···O═C interactions and dimers related by the 2_1 axis are connected by C—H···N interactions. The screw around $[\frac{3}{4},y,\frac{3}{4}]$ is running in the −y direction (*i.e.* out of the plane of the paper) and that around $[\frac{5}{4},y,\frac{1}{4}]$ in the +y direction.

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dimethylamino group deviates by 0.041 (2) Å from the least-squares plane of the aromatic ring. The deviations of the C8′ and O2 atoms from the least-squares planes of the rings to which they are attached are marginal [0.006 (3) and 0.013 (3) Å, respectively]. The bisecting plane of the dimethylamino group forms a dihedral angle of 46.5 (1)° with the best plane of the aromatic ring.

The crystal packing scheme is shown in Fig. 2. Inversion-related molecules form planar dimers which are connected by a circular motif of mutual N—H···O═C hydrogen bonds [N···O($1-x$, $-y$, $1-z$) 2.881 (2) Å; for N—H normalized to 1.04 Å, H···O 1.87 Å and N—H···O 163°]. Analogous dimer arrangement is observed in related crystal structures (Sudha, Subramanian, Sivaraman, Ramakrishnan, Steiner & Koellner, 1995; Sudha, Subramanian, Sivaraman, Sriraghavan & Steiner, 1995; not explicitly mentioned there). The dimethylamino N11 lone pair accepts a weak C—H···N interaction from C3—H [C···N($\frac{3}{2}-x$, $-\frac{1}{2}+y$, $\frac{3}{2}-z$) 3.432 (3) Å; for C—H normalized to 1.09 Å, H···N 2.40 Å and C—H···N 158°]; this interaction connects dimers related by the twofold screw axis.

Experimental

The title compound, (1), was synthesized at and supplied by the University of Madras, India. 8-Methyl-2-quinolone on reaction with urea nitrate gave the 5-nitro derivative, which was then converted into 5-amino-8-methyl-2-quinolone. Further reaction with methyl iodide yielded compound (1). The crystals were grown by slow evaporation of a chloroform solution. The specimen used for data collection was cut from a crystal with dimensions 4 × 1 × 0.03 mm.

Crystal data

$C_{12}H_{14}N_2O$
$M_r = 202.25$
Monoclinic
$P2_1/n$
$a = 14.133\ (2)$ Å
$b = 8.642\ (2)$ Å
$c = 8.7698\ (11)$ Å
$\beta = 93.89\ (3)°$
$V = 1068.7\ (3)$ Å^3
$Z = 4$
$D_x = 1.257$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10.3–31.9°$
$\mu = 0.650$ mm^{-1}
$T = 293\ (2)$ K
Thin plate
0.50 × 0.40 × 0.03 mm
Colourless

Data collection

Enraf–Nonius Turbo-CAD-4 diffractometer
ω-2θ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.759$, $T_{\max} = 0.995$
1988 measured reflections
1578 independent reflections

1257 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 59.80°$
$h = -15 \rightarrow 15$
$k = -9 \rightarrow 1$
$l = -9 \rightarrow 1$
25 standard reflections frequency: 30 min intensity decay: 1.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0446$
$wR(F^2) = 0.1270$
$S = 1.095$
1578 reflections
153 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.068P)^2 + 0.2396P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.144$ e Å^{-3}
$\Delta\rho_{\min} = -0.177$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N1—C2	1.364 (2)	C3—C4	1.342 (3)
N1—C9	1.393 (2)	C5—N11	1.424 (2)
C2—O2	1.242 (2)	N11—C13	1.456 (3)
C2—C3	1.440 (3)	N11—C12	1.476 (3)
O2—C2—C3	123.1 (2)	C5—C10—C4	122.8 (2)
N1—C2—C3	115.8 (2)		
C6—C5—N11—C12	−110.7 (2)	C6—C5—N11—C13	19.9 (3)

H atoms were refined using a riding model (*SHELXL*93; Sheldrick, 1993), with methyl groups allowed to rotate and isotropic U allowed to vary (C—H 1.0 and N—H 0.95 Å).

Data collection: *CAD*-4 *Software* (Enraf–Nonus, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CAD*-4 *Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: JZ1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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