

Table 2. Selected geometric parameters (Å, °)

Br—C(1)	1.96 (1)	C(3)—N	1.13 (2)
C(1)—C(2)	1.527 (8)	C(2)—C(2 ⁱⁱⁱ)	1.514 (9)
C(1)—C(3)	1.50 (2)		
Br—C(1)—C(2)	109.6 (2)	C(1)—C(3)—N	178 (1)
Br—C(1)—C(3)	105.2 (7)	C(1)—C(2)—C(2 ⁱⁱⁱ)	111.2 (5)
C(2)—C(1)—C(3)	110.2 (3)	C(2)—C(1)—C(2 ⁱⁱ)	111.7 (4)
D—H...A	H...A	D...A	D—H...A
C(2)—H(2a)...Br ^v	3.07 (1)	4.049 (8)	150.2 (5)
C(2)—H(2e)...Br ^v	3.043 (8)	3.994 (6)	146.3 (3)

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

After an anisotropic refinement of the displacement parameters, the H-atom positions were obtained from a Fourier synthesis. Further refinements were performed with the H atoms riding on bound C atoms.

Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure: *SHELXL76* (Sheldrick, 1976). Software used to prepare material for publication: *MoIEN* (Fair, 1990), *PARST* (Nardelli, 1983), *ORTEPII* (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: CR1127). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Aihara, A., Kitazawa, C. & Iwasaki, F. (1968). *Bull. Chem. Soc. Jpn.* **41**, 1034.
- Augsburger, J. D., Dykstyra, C. E. & Zwier, T. S. (1992). *J. Phys. Chem.* **96**, 7252–7257.
- Barón, M. (1988). *J. Mol. Struct.* **172**, 355–367.
- Barón, M. (1991). *J. Mol. Struct.* **248**, 275–280.
- Barón, M., De Zenobi, E. L. & Davidson, M. (1975). *J. Mol. Struct.* **24**, 432–438.
- Bucourt, R. & Hainaut, D. (1965). *Soc. Chim.* **5**, 1366–1378.
- Donati, D., Fusi, S., Ponticelli, F. & Fiorenza, M. (1989). *Acta Cryst.* **C45**, 317–319.
- Echeverria, G., Punte, G., Rivero, B. E. & Barón, M. (1995). *Acta Cryst.* **C51**, 1020–1023.
- Fair, C. K. (1990). *MoIEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Kozima, K. & Yoshimo, T. (1953). *J. Am. Chem. Soc.* **75**, 166–171.
- Le Févre, C. G. & Le Févre, R. J. W. (1956). *J. Chem. Soc. pp.* 3549–3563.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Sheldrick, G. M. (1976). *SHELXL76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
- Suzuki, S., Green, P. G., Bumgarner, R. E., Dasgupta, S., Goddard, W. A. III & Balke, G. A. (1992). *Science*, **257**, 942–945.
- Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

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5-(Isopropylamino)-8-methyl-2-quinolone

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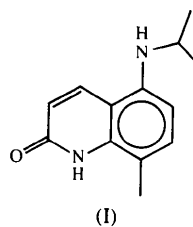
(Received 27 June 1994; accepted 7 November 1994)

Abstract

The title compound, C₁₃H₁₆N₂O, is a new laser dye. The quinolone ring system is essentially planar. The methyl and the amino groups at C(5) and C(8) are in equatorial positions with respect to the ring. The isopropyl group is inclined from the plane of the aromatic ring. N—H...O and weak C—H...O hydrogen bonds are formed.

Comment

This study was undertaken as part of our program on the structural aspects of quinolone laser dyes. The laser performance of the title compound, (I), is yet to be studied, but the laser emission of similar compounds is around $\lambda = 410$ nm (Hammond, Fletcher, Henry & Atkins, 1975; Hammond *et al.*, 1976).



Bond lengths and valence angles in the quinolone ring are normal (Chinnakali, Sivakumar, Natarajan, McGuire & Clearfield, 1991); the C(3)—C(4) bond length of 1.350 (6) Å is indicative of a localized double bond. The angles C(4)—C(10)—C(5) and C(3)—C(2)—O(2) are greater than 120°, while C(3)—C(2)—N(1) is less than 120°, as is commonly found in quinolone derivatives (Kido, Nakagawa, Fujiwara & Tomita, 1981). The C(2)—O(2) bond length is 1.259 (5) Å, indicative of a double bond, which is a unique value in quinolone derivatives (Chinnakali *et al.*, 1991). The C(8)—C(8')

bond length of 1.501 (6) Å agrees with the analogous bond lengths in similar compounds (Fronczek, Dupré, Ravi Kumar & Maverick, 1991). The quinolone ring is planar ($\chi^2 = 14.10$). The N(11) atom of the amino group at C(5) deviates by -0.025 (3) Å and the methyl C(8') atom at C(8) deviates by $+0.023$ (8) Å from the mean plane of the ring. Fig. 2 shows the packing of the molecule in the unit cell. The crystal structure is stabilized by a number of intermolecular hydrogen bonds (Table 2).

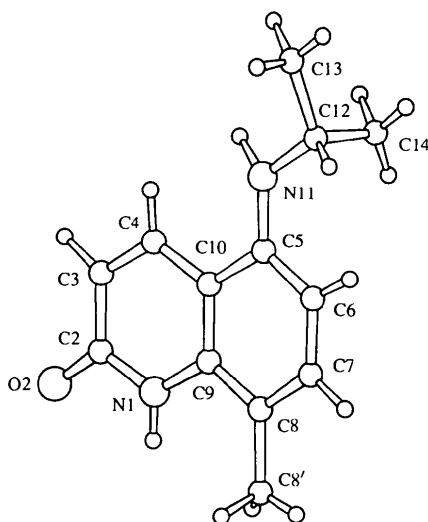


Fig. 1. Molecular structure and atomic numbering scheme.

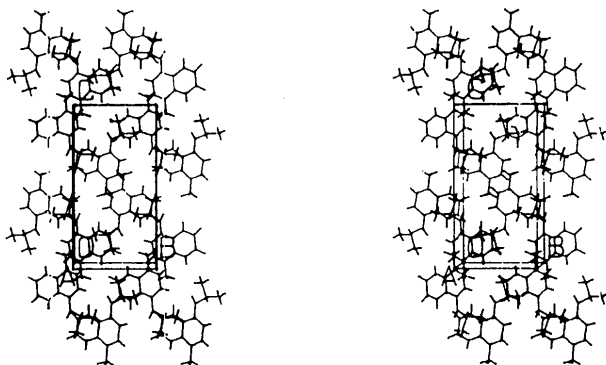


Fig. 2. Stereoview of the molecular packing.

Experimental

Alkylation of 5-amino-8-methylquinol-2-one with isopropyl bromide at 433 K in an autoclave afforded the title compound. The density D_m was measured by flotation in KI and water.

Crystal data

$C_{13}H_{16}N_2O$
 $M_r = 216$

Cu $K\alpha$ radiation
 $\lambda = 1.54184$ Å

Monoclinic

$P2_1/c$

$a = 8.478$ (3) Å

$b = 8.386$ (3) Å

$c = 16.719$ (7) Å

$\beta = 92.73$ (3)°

$V = 1187.3$ (8) Å³

$Z = 4$

$D_x = 1.20$ Mg m⁻³

$D_m = 1.21$ Mg m⁻³

Cell parameters from 22

reflections

$\theta = 10$ – 22°

$\mu = 0.584$ mm⁻¹

$T = 294$ K

Needle

$0.6 \times 0.5 \times 0.5$ mm

Colourless

Data collection

Enraf–Nonius CAD-4
diffractometer

$\omega/2\theta$ scans

Absorption correction:
none

1979 measured reflections

1674 independent reflections

1614 observed reflections

$[F > 3\sigma(F)]$

$R_{int} = 0.047$

$\theta_{max} = 60^\circ$

$h = -9 \rightarrow 9$

$k = 0 \rightarrow 9$

$l = 0 \rightarrow 18$

3 standard reflections

frequency: 60 min

intensity decay: <2%

Refinement

Refinement on F

$R = 0.0747$

$wR = 0.089$

1614 reflections

209 parameters

H atoms refined isotropically

$w = 0.5895/[\sigma^2(F)$

$+ 0.006344F^2]$

$(\Delta/\sigma)_{max} = 0.009$

$\Delta\rho_{max} = 0.326$ e Å⁻³

$\Delta\rho_{min} = -0.282$ e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{eq} = (1/3)\sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
N(1)	1.0958 (4)	-0.1625 (4)	0.4296 (2)	3.6 (1)
C(2)	1.0412 (4)	-0.0441 (5)	0.3796 (2)	3.8 (1)
O(2)	0.9629 (3)	0.0697 (3)	0.4067 (2)	4.8 (1)
C(3)	1.0764 (5)	-0.0594 (5)	0.2977 (2)	4.0 (1)
C(4)	1.1552 (5)	-0.1880 (5)	0.2719 (2)	3.9 (1)
C(5)	1.2969 (4)	-0.4459 (5)	0.2975 (2)	3.7 (1)
C(6)	1.3518 (5)	-0.5544 (5)	0.3553 (3)	4.6 (1)
C(7)	1.3193 (5)	-0.5347 (5)	0.4344 (3)	4.9 (1)
C(8)	1.2326 (5)	-0.4085 (5)	0.4630 (2)	4.2 (1)
C(8')	1.1998 (10)	-0.3937 (8)	0.5501 (3)	5.8 (2)
C(9)	1.1795 (4)	-0.2962 (4)	0.4056 (2)	3.5 (1)
C(10)	1.2094 (4)	-0.3115 (4)	0.3249 (2)	3.4 (1)
N(11)	1.3283 (4)	-0.4567 (4)	0.2180 (2)	4.3 (1)
C(12)	1.4146 (5)	-0.5906 (6)	0.1855 (3)	5.1 (1)
C(13)	1.3174 (9)	-0.7444 (8)	0.1811 (5)	7.2 (2)
C(14)	1.4688 (10)	-0.5420 (12)	0.1042 (4)	7.8 (3)

Table 2. Selected geometric parameters (Å, °)

N(1)—C(2)	1.365 (5)	C(6)—C(7)	1.374 (7)
N(1)—C(9)	1.396 (5)	C(7)—C(8)	1.387 (6)
C(2)—O(2)	1.259 (5)	C(8)—C(8')	1.501 (6)
C(2)—C(3)	1.421 (5)	C(8)—C(9)	1.403 (5)
C(3)—C(4)	1.350 (6)	C(9)—C(10)	1.391 (5)
C(4)—C(10)	1.425 (5)	N(11)—C(12)	1.459 (6)
C(5)—C(6)	1.391 (6)	C(12)—C(13)	1.530 (9)
C(5)—C(10)	1.436 (5)	C(12)—C(14)	1.511 (9)
C(5)—N(11)	1.371 (5)		
C(2)—N(1)—C(9)	124.8 (3)	C(7)—C(8)—C(8')	121.6 (4)
N(1)—C(2)—C(3)	116.4 (4)	C(8')—C(8)—C(9)	122.7 (4)
N(1)—C(2)—O(2)	120.0 (3)	N(1)—C(9)—C(8)	119.6 (3)
O(2)—C(2)—C(3)	123.6 (4)	C(8)—C(9)—C(10)	122.3 (3)

C(2)—C(3)—C(4)	120.7 (3)	N(1)—C(9)—C(10)	118.1 (3)	
C(3)—C(4)—C(10)	122.1 (3)	C(5)—C(10)—C(9)	120.2 (3)	
C(10)—C(5)—N(11)	119.2 (3)	C(4)—C(10)—C(9)	117.9 (3)	
C(6)—C(5)—N(11)	123.8 (4)	C(4)—C(10)—C(5)	121.9 (3)	
C(6)—C(5)—C(10)	116.9 (3)	C(5)—N(11)—C(12)	122.4 (4)	
C(5)—C(6)—C(7)	121.0 (4)	N(11)—C(12)—C(14)	107.9 (5)	
C(6)—C(7)—C(8)	123.9 (4)	N(11)—C(12)—C(13)	112.8 (4)	
C(7)—C(8)—C(9)	115.8 (4)	C(13)—C(12)—C(14)	111.7 (5)	
<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N(1)—H(1)...O(2 ⁱ)	0.87 (4)	2.06 (4)	2.911 (5)	167 (3)
C(8 ⁱ)—H(8 ⁱ <i>b</i>)...O(2 ⁱ)	1.15 (5)	2.74 (5)	3.147 (8)	100 (3)
C(8 ⁱ)—H(8 ⁱ <i>a</i>)...O(2 ⁱ)	0.87 (7)	2.69 (7)	3.147 (8)	114 (3)
N(11)—H(11)...O(2 ⁱⁱ)	1.06 (3)	2.19 (3)	3.162 (5)	151 (3)

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

The structure was solved by direct methods. The initial *R* index for the model was 0.32. After a few cycles of full-matrix least-squares refinement of non-H atoms, the *R* index dropped to 0.17. The H atoms were located from a difference Fourier map and refined isotropically.

Data collection: Enraf-Nonius CAD-4 diffractometer software. Structure solution: *SHELXS86* (Sheldrick, 1985). Structure refinement: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEPII* (Johnson, 1976). Preparation of material for publication: *PARST* (Nardelli, 1983).

KS thanks the Deutscher Akademischer Austauschdienst for support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances involving H atoms and least-squares-planes data have been deposited with the IUCr (Reference: KA1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Chinnakali, K., Sivakumar, K., Natarajan, S., McGuire, N. K. & Clearfield, A. (1991). *Acta Cryst.* **C47**, 561–563.
- Fronczek, F. R., Dupré, M. D., Ravi Kumar & Maverick, A. W. (1991). *Acta Cryst.* **C47**, 456–457.
- Hammond, P. R., Fletcher, A. N., Bliss, D. E., Henry, R. A., Atkins, R. L. & Moore, D. W. (1976). *Appl. Phys.* **9**, 67–68.
- Hammond, P. R., Fletcher, A. N., Henry, R. A. & Atkins, R. L. (1975). *Appl. Phys.* **8**, 311–315.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Kido, M., Nakagawa, K., Fujiwara, T. & Tomita, K. (1981). *Chem. Pharm. Bull.* **29**, 2109–2115.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

ADDENDA AND ERRATA

Acta Cryst. (1995). **C51**, 1028

Structure of tris(pyridine)mercury(II) bis(trifluoroacetate). Erratum. By JOAN HALFPENNY, *Department of Chemistry and Physics, Nottingham Trent University, Clifton Lane, Nottingham NG11 8NS, England*, and R. W. H. SMALL, *Department of Chemistry, The University, Lancaster LA1 4YA, England*

(Received 27 February 1995)

Abstract

In the paper by Halfpenny & Small [*Acta Cryst.* (1978), **B34**, 3758–3760] the coordinates of the atom N(1) are stated incorrectly. The correct values of the coordinates are

$$x = 0.312 (2), y = 0.075 (1) \text{ and } z = 0.082 (3).$$

All relevant information is given in the *Abstract*.