Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cl	0.36753 (10)	0.3953 (2)	0.08929 (8)	0.0858 (10)
F1	0.24531 (18)	0.3210 (4)	0.20646 (18)	0.0754 (17)
F2	0.22348 (17)	0.3670 (4)	0.37220 (17)	0.079 (2)
F3	0.38091 (18)	0.3157 (4)	0.53851 (17)	0.081 (2)
F4	0.56387 (17)	0.2257 (4)	0.53708 (16)	0.0662 (18)
F5	0.58901 (16)	0.1856 (4)	0.37246 (15)	0.0635 (17)
F6	0.55941 (19)	0.2997 (4)	0.07093 (17)	0.082 (2)
F7	0.69580 (18)	0.0316 (4)	0.05978 (17)	0.090 (2)
F8	0.72461 (18)	−0.3485 (5)	0.15166 (18)	0.085 (2)
F9	0.61288 (16)	−0.4556 (4)	0.25379 (17)	0.0677 (17)
F10	0.47725 (16)	−0.1879 (3)	0.26776 (15)	0.0592 (16)
C1	0.4186 (3)	0.2534 (6)	0.2802 (3)	0.047 (3)
C2	0.3275 (3)	0.2990 (6)	0.2870 (3)	0.053 (3)
C3	0.3136 (3)	0.3211 (6)	0.3714 (3)	0.054 (3)
C4	0.3930 (3)	0.2952 (7)	0.4553 (3)	0.055 (3)
C5	0.4847 (3)	0.2513 (7)	0.4544 (3)	0.050 (3)
C6	0.4959 (3)	0.2299 (6)	0.3683 (3)	0.049 (3)
C7	0.5109 (3)	0.0706 (7)	0.1701 (3)	0.047 (3)
C8	0.5695 (3)	0.1131 (8)	0.1171 (3)	0.055 (3)
C9	0.6407 (3)	−0.0220 (9)	0.1106 (3)	0.059 (3)
C10	0.6555 (3)	−0.2134 (8)	0.1566 (3)	0.058 (3)
C11	0.5991 (3)	−0.2671 (7)	0.2095 (3)	0.050 (3)
C12	0.5297 (3)	−0.1242 (7)	0.2151 (3)	0.047 (3)
B	0.4354 (4)	0.2293 (8)	0.1840 (3)	0.054 (4)

Table 2. *Selected geometric parameters* (Å, °)

Cl—B	1.746 (5)	C1—B	1.566 (6)
F1—C2	1.351 (5)	C2—C3	1.367 (6)
F2—C3	1.339 (5)	C3—C4	1.363 (6)
F3—C4	1.337 (5)	C4—C5	1.358 (6)
F4—C5	1.349 (5)	C5—C6	1.376 (6)
F5—C6	1.353 (5)	C7—C8	1.392 (6)
F6—C8	1.347 (5)	C7—C12	1.380 (6)
F7—C9	1.340 (5)	C7—B	1.551 (7)
F8—C10	1.336 (5)	C8—C9	1.367 (7)
F9—C11	1.341 (5)	C9—C10	1.368 (8)
F10—C12	1.348 (4)	C10—C11	1.379 (6)
C1—C2	1.390 (6)	C11—C12	1.375 (6)
C1—C6	1.386 (6)		
C2—C1—C6	113.4 (4)	C12—C7—B	121.7 (4)
C2—C1—B	124.7 (4)	F6—C8—C7	119.6 (4)
C6—C1—B	121.9 (4)	F6—C8—C9	116.5 (4)
F1—C2—C1	119.5 (4)	C7—C8—C9	123.9 (4)
F1—C2—C3	116.1 (4)	F7—C9—C8	120.6 (5)
C1—C2—C3	124.3 (4)	F7—C9—C10	120.0 (4)
F2—C3—C2	120.9 (4)	C8—C9—C10	119.5 (4)
F2—C3—C4	120.0 (4)	F8—C10—C9	120.8 (4)
C2—C3—C4	119.1 (4)	F8—C10—C11	119.5 (5)
F3—C4—C3	120.0 (4)	C9—C10—C11	119.7 (4)
F3—C4—C5	120.0 (4)	F9—C11—C10	119.8 (4)
C3—C4—C5	120.0 (4)	F9—C11—C12	121.6 (4)
F4—C5—C4	120.6 (4)	C10—C11—C12	118.6 (4)
F4—C5—C6	120.1 (4)	F10—C12—C7	120.3 (4)
C4—C5—C6	119.4 (4)	F10—C12—C11	115.2 (4)
F5—C6—C1	119.8 (4)	C7—C12—C11	124.5 (4)
F5—C6—C5	116.3 (4)	Cl—B—C1	117.7 (3)
C1—C6—C5	123.8 (4)	Cl—B—C7	119.0 (3)
C8—C7—C12	113.8 (4)	C1—B—C7	123.3 (4)
C8—C7—B	124.3 (4)		

The structure was solved by direct methods. All non-H atoms were located *via E* map inspection and were refined anisotropically.

Data collection: *Enraf–Nonius CAD-4 Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* (Boer & Duisenberg, 1984); *CELDIM* (Enraf–Nonius, 1992). Data reduction: *NRCVAX94 DATRD2* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX94 SOLVER*. Program(s) used to refine structure: *NRCVAX94*. Molecular graphics: *NRCVAX94*; *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *NRCVAX94*.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BK1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Amino-8-methyl-2-quinolone Monohydrate

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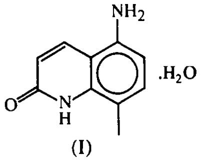
Abstract

The quinolone ring system in $C_{10}H_{10}N_2O.H_2O$ is essentially planar. The hydrogen-bonding scheme involves O—H···O, N—H···O, N—H···N and C—H···O interactions.

Comment

Since the observation of laser action from organic compounds, many classes of dyes have been shown to produce this action. Numerous new compounds have been synthesized and investigated in order to obtain high laser efficiency, wide tunability and photostability. With this goal in mind, the title compound, (I), which is a new laser dye, was synthesized. Fig. 1 shows a perspective view of the molecular geometry and the numbering scheme used.



(I)

The average C_{sp^2}—C_{sp^2} bond length is 1.390 (1) Å and the average angle involving these bonds is 120 (1)°. Bond lengths and valence angles in the quinolone ring are normal (Kido & Nakagawa, 1982) with C(3)—C(4) = 1.340 (3) Å 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°, whereas C(3)—C(2)—N(1) is less than 120°, as is commonly found in quinolone derivatives (Kido, Nakagawa, Fujiwara & Tomita, 1981; Kido & Nakagawa, 1982). The quinolone ring system is essentially planar (χ^2 = 14.10). The amino N atom N(11) at C(5) deviates by −0.065 (2) Å and the methyl atom C(8′) at C(8) deviates by −0.066 (4) Å from the least-squares plane through the aromatic ring. The molecular packing is illustrated in Fig. 2. The water molecule forms a hydrogen-bonded bridge between neighbouring molecules. The crystal structure is stabilized by intermolecular hydrogen bonds, the geometries of which are given in Table 3.

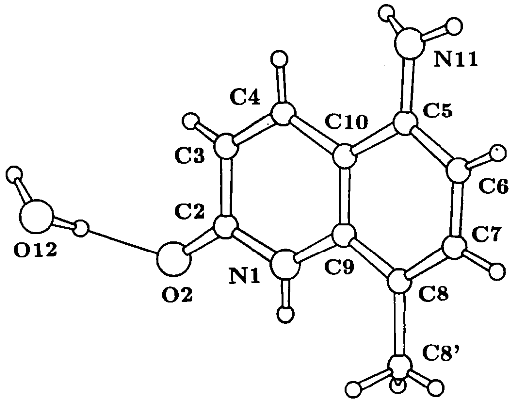


Fig. 1. Molecular structure and atomic numbering scheme of the title compound.

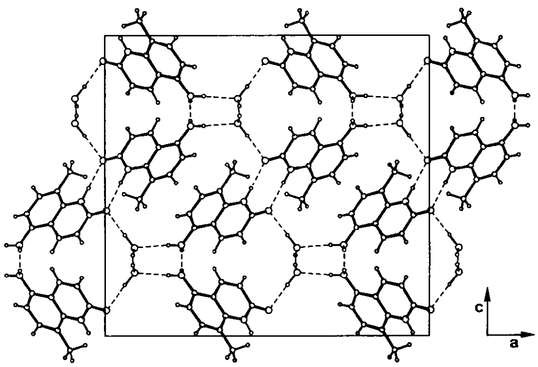


Fig. 2. Crystal packing and hydrogen-bonding pattern.

Experimental

8-Methyl-2-quinolone was nitrated with urea nitrate and reduced with iron and acetic acid to afford the title compound. The crystal density D_m was measured by flotation in water/KI.

Crystal data

$C_{10}H_{10}N_2O.H_2O$
M_r = 192
Orthorhombic
Pbca
a = 20.320 (3) Å
b = 18.618 (4) Å
c = 5.095 (2) Å
V = 1927.65 Å^3
Z = 8
D_x = 1.33 Mg m^{-3}
D_m = 1.32 Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.54184 Å
Cell parameters from 20 reflections
θ = 26–54°
μ = 0.73 mm^{-1}
T = 294 K
Needle
1.5 × 0.2 × 0.1 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–θ scans
Absorption correction: ψ scan
T_{min} = 0.72, T_{max} = 0.98
1725 measured reflections
1323 independent reflections
1308 observed reflections [$F > 3\sigma(F)$]

R_{int} = 0.027
θ_{max} = 60°
$h = 0 \rightarrow 22$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 5$
3 standard reflections monitored every 400 reflections
intensity decay: 11%

Refinement

Refinement on F
R = 0.0540
wR = 0.0620
S = 1.22
1308 reflections
175 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F) + 0.017460F^2]$

$(\Delta/\sigma)_{max}$ = 0.007
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
N(1)	0.5724 (1)	0.9449 (1)	−0.1780 (3)	3.0 (1)
C(2)	0.5476 (1)	0.8998 (1)	0.0058 (4)	3.1 (1)
O(2)	0.4951 (1)	0.9146 (1)	0.1244 (3)	3.9 (1)
C(3)	0.5849 (1)	0.8355 (1)	0.0537 (4)	3.4 (1)
C(4)	0.6412 (1)	0.8228 (1)	−0.0751 (4)	3.0 (1)
C(5)	0.7244 (1)	0.8580 (1)	−0.4155 (4)	3.0 (1)
C(6)	0.7399 (1)	0.9038 (1)	−0.6183 (4)	3.2 (1)
C(7)	0.7010 (1)	0.9627 (1)	−0.6728 (4)	3.4 (1)

C(8)	0.6459 (1)	0.9802 (1)	−0.5283 (4)	3.0 (1)
C(8′)	0.6071 (1)	1.0468 (1)	−0.5884 (5)	4.0 (1)
C(9)	0.6283 (1)	0.9327 (1)	−0.3284 (4)	2.8 (1)
C(10)	0.6656 (1)	0.8707 (1)	−0.2714 (4)	2.7 (1)
N(11)	0.7642 (1)	0.7992 (1)	−0.3583 (3)	3.4 (1)
O(12)	0.4087 (1)	0.7903 (1)	0.0940 (4)	5.3 (1)

Table 2. *Selected geometric parameters* (Å, °)

N(1)—C(2)	1.355 (3)	C(5)—C(10)	1.422 (3)
N(1)—C(9)	1.389 (3)	C(5)—N(11)	1.392 (3)
C(2)—O(2)	1.257 (3)	C(6)—C(7)	1.380 (3)
C(2)—C(3)	1.438 (3)	C(7)—C(8)	1.379 (3)
C(3)—C(4)	1.340 (3)	C(8)—C(8′)	1.501 (3)
C(4)—C(10)	1.429 (3)	C(8)—C(9)	1.396 (3)
C(5)—C(6)	1.376 (3)	C(9)—C(10)	1.411 (3)
C(2)—N(1)—C(9)	125.7 (2)	C(6)—C(7)—C(8)	123.1 (2)
N(1)—C(2)—C(3)	115.9 (2)	C(7)—C(8)—C(9)	116.6 (2)
N(1)—C(2)—O(2)	120.8 (2)	C(7)—C(8)—C(8′)	120.9 (2)
O(2)—C(2)—C(3)	123.3 (2)	C(8′)—C(8)—C(9)	122.5 (2)
C(2)—C(3)—C(4)	120.9 (2)	N(1)—C(9)—C(8)	120.6 (2)
C(3)—C(4)—C(10)	121.9 (2)	C(8)—C(9)—C(10)	122.1 (2)
C(10)—C(5)—N(11)	120.7 (2)	N(1)—C(9)—C(10)	117.4 (2)
C(6)—C(5)—N(11)	120.8 (2)	C(5)—C(10)—C(9)	118.8 (2)
C(6)—C(5)—C(10)	118.5 (2)	C(4)—C(10)—C(9)	117.9 (2)
C(5)—C(6)—C(7)	120.8 (2)	C(4)—C(10)—C(5)	123.3 (2)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O(12)—H(12*b*)···O(2)	0.99 (6)	1.93 (6)	2.909 (3)	170 (5)
C(8′)—H(8′*b*)···O(2^{i})	0.93 (6)	2.57 (6)	3.228 (3)	128 (5)
N(1)—H(1)···O(2^{i})	0.97 (6)	2.00 (6)	2.966 (3)	175 (5)
O(12)—H(12*a*)···O(12^{ii})	1.10 (7)	1.87 (6)	2.957 (3)	167 (5)
N(11)—H(11*b*)···N(11^{ii})	0.95 (6)	2.25 (6)	3.138 (2)	156 (5)
N(11)—H(11*a*)···O(12^{iii})	0.92 (6)	2.26 (6)	3.177 (3)	170 (5)

Symmetry codes: (i) $1 - x, 2 - y, -z$; (ii) $x, \frac{3}{2} - y, \frac{1}{2} + z$; (iii) $\frac{1}{2} + x, y, -\frac{1}{2} - z$.

Data collection, cell refinement and data reduction: *SDP* (Frenz, 1978); structure solution: *SHELXS*86 (Sheldrick, 1985); structure refinement: *SHELX*76 (Sheldrick, 1976); software used to prepare material for publication: *PARST* (Nardelli, 1983), *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1016). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2-Oxo-1,3-thiazolidin-3-yl)carbonylthioethylammonium Nitrate, $C_6H_{11}N_2O_2S_2^+.NO_3^-$

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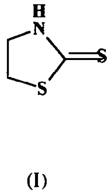
Abstract

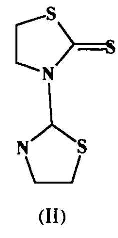
Addition of H_2O_2 to an aqueous solution of 1,3-thiazolidine-2-thione and $ZnCl_2$ produced $ZnSO_4.2H_2O$ and the title compound. The asymmetric unit of the latter consists of one NO_3^- anion and one $C_6H_{11}N_2O_2S_2^+$ cation. The thiazolidine ring has a C(2)-envelope conformation and the exocyclic ketonic and the carbonylic O atoms are *trans* related. The nitrate anions are linked to the cations through hydrogen bonds which involve the H atoms of the ammonium N atoms.

Comment

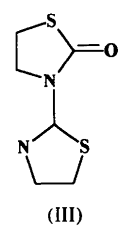
We have reported the syntheses and crystal structures of Pd^{II} (Kubiak & Głowiak, 1982), Cd^{II} (Kubiak & Głowiak, 1985) and Zn^{II} (Kubiak & Głowiak, 1987) complexes of 1,3-thiazolidine-2-thione [tzdtH, (I)], in which the ligand uses its exocyclic thione S atom in monodentate ligation to the metals. In a reaction with $FeCl_2.4H_2O$, the related unsaturated ligand thiazoline-2-thione (tztH) was oxidized to the *N*,*N*′-chelating 2,2′-dithiazolyl disulfide (ttzSSttz), giving $[Fe_2(ttzSSttz)_2Cl_4]$ (Raper, Miller, Głowiak & Kubiak, 1989). Similarly, the reaction of $ZnCl_2$/tzdtH in concentrated hydrochloric acid also produced an oxidation product, 2-(2-thioxo-1,3-thiazolidin-3-yl)-4,5-dihydro-1,3-thiazole [tzdtzS, (II)], which *S*,*N*-chelates to the metal in $[Zn(tzdtzS)Cl_3(H_2O)]$ (Kubiak & Głowiak, 1986). Oxidation of (I) by $CuCl_2.2H_2O$ also produced *S*,*N*-chelating tzdtzS in $[Cu(tzdtzS)Cl_2]_n$; with



(I)



(II)



(III)

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