Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

9-(4-Fluorophenyl)-*N*-hydroxy-3,3,6,6-tetramethyl-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione monohydrate

Shujiang Tu,* Fang Fang and Songlei Zhu

Department of Chemistry, Xuzhou Normal University, Xuzhou 221116, People's Republic of China

Correspondence e-mail: laotu2001@263.net

Received 19 August 2004
Accepted 31 August 2004
Online 4 September 2004

Key indicators

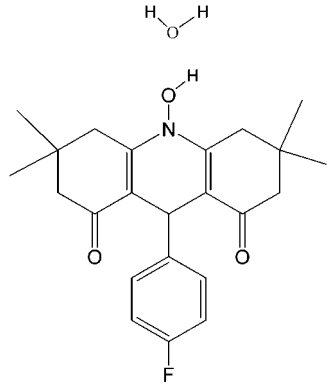
Single-crystal X-ray study
T = 288 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.102
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{23}H_{26}FNO_3\cdot H_2O$, has been synthesized by the reaction of hydroxylamine with 4-fluorobenzaldehyde and dimedone in ethylene glycol under microwave irradiation. X-ray analysis reveals that the dihydropyridine ring adopts a boat conformation.

Comment

Acridine have interesting chemical and physical properties, and immense utility in the pharmaceutical and dye industries. The discovery of acridines as antimalarial and antitumor agents has attracted the attention of organic chemists and thus led to intensive interest in the synthesis of several drugs based on acridine (Khurana *et al.*, 1990; Matsumoto *et al.*, 1983; Nakano *et al.*, 1982). Chemical modifications of the acridine ring system, such as the introduction of an aryl group on the N atom, cause laser activity (Murugan *et al.*, 1998). Recently, we achieved the introduction of a hydroxy group on the N atom. We report here the X-ray crystal structure of the title compound, (I).



(I)

In (I), the dihydropyridine ring adopts a boat conformation, with the atoms N and C7 deviating from the C8/C13/C19/C14 mean plane by 0.108 (2) and 0.240 (2) Å, respectively (Fig. 1). In the crystal structure there are O—H···O hydrogen bonds (Table 2). The crystal packing, shown in Fig. 2, reveals that the molecules are linked by hydrogen bonds involving the water molecule of crystallization (O4), forming a two-dimensional sheet parallel to the (100) plane.

Experimental

The title compound, (I), was prepared by the reaction of hydroxylamine (2 mmol) with 4-fluorobenzaldehyde (2 mmol) and dimedone (4 mmol) in ethylene glycol (2 ml) under microwave irradiation. The reaction was completed in 4 min with the yield of 90%. Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an ethanol solution (95%) (m.p. 506–507 K).

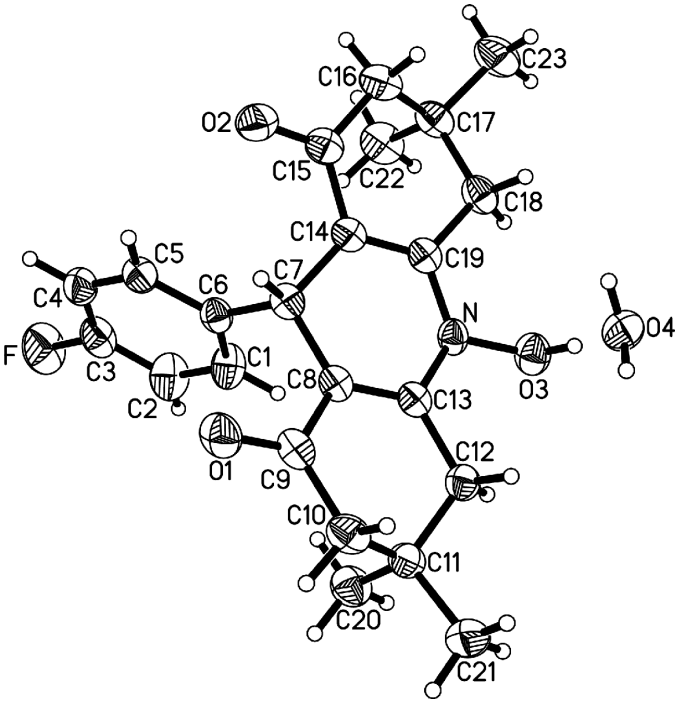


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

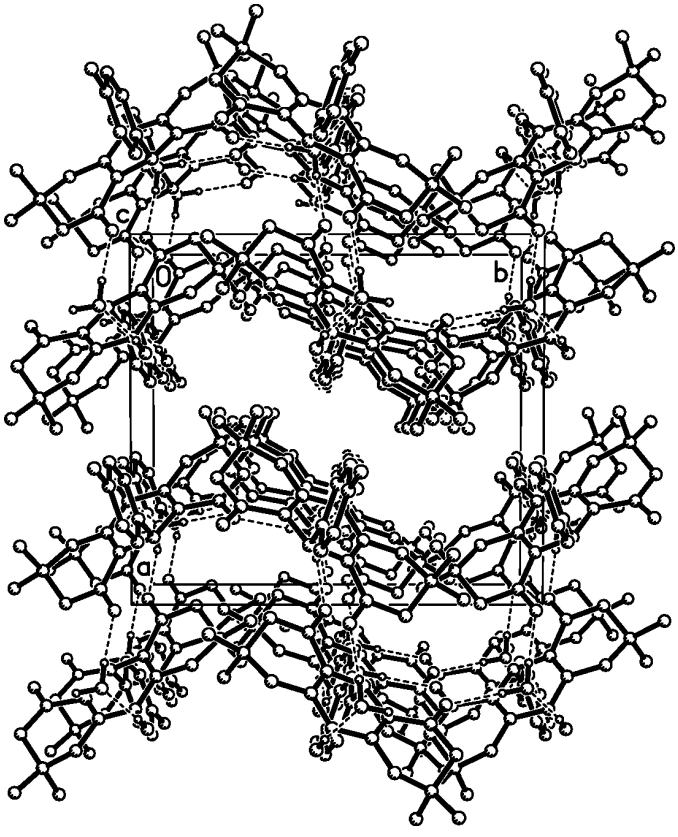


Figure 2
A molecular packing diagram for (I). Dashed lines indicate hydrogen bonds.

Crystal data

$C_{23}H_{26}FNO_3\cdot H_2O$	D_x = 1.246 Mg m^{-3}
M_r = 401.46	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 27 reflections
a = 12.638 (2) Å	θ = 3.5–14.9°
b = 14.039 (3) Å	μ = 0.09 mm^{-1}
c = 12.102 (2) Å	T = 288 (2) K
β = 94.60 (1)°	Block, yellow
V = 2140.2 (6) Å^3	0.58 × 0.58 × 0.40 mm
Z = 4	

Data collection

Siemens $P4$ diffractometer	θ_{max} = 25.0°
ω scans	$h = -15 \rightarrow 14$
Absorption correction: none	$k = -16 \rightarrow 0$
4291 measured reflections	$l = 0 \rightarrow 14$
3775 independent reflections	3 standard reflections
2220 reflections with $I > 2\sigma(I)$	every 97 reflections
R_{int} = 0.010	intensity decay: 2.9%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.102	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 0.91	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
3775 reflections	Extinction correction: *SHELXL*97
279 parameters	Extinction coefficient: 0.0083 (9)
H atoms: see below	

Table 1
Selected geometric parameters (Å, °).

O3—N	1.4033 (19)	C7—C14	1.518 (2)
N—C19	1.375 (2)	C8—C13	1.355 (2)
N—C13	1.387 (2)	C14—C19	1.351 (2)
C7—C8	1.505 (2)		
C19—N—C13	122.79 (15)	C13—C8—C7	122.65 (16)
C19—N—O3	118.20 (14)	C8—C13—N	119.06 (17)
C13—N—O3	117.36 (15)	C19—C14—C7	122.41 (17)
C8—C7—C14	109.78 (14)	C14—C19—N	119.38 (16)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3O···O4	0.858 (10)	1.82 (2)	2.610 (2)	152 (4)
O4—H4OA···O1^{i}	0.854 (10)	1.933 (10)	2.786 (2)	179 (3)
O4—H4OB···O2^{ii}	0.853 (10)	1.826 (11)	2.675 (2)	174 (4)

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

The hydroxyl and water H atoms were refined isotropically with a restrained O—H distance of 0.85 Å. The other H atoms were positioned geometrically and treated as riding, with C—H distances of 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(parent).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We thank the National Natural Science Foundation of China (No. 20372057) for financial support.

References

Khurana, J. M., Maikap, G. C. & Mehta, S. (1990). *Synthesis*, pp. 731–732.

Matsumoto, H., Arai, T., Takahashi, M., Ashizawa, T., Nakano, T. & Nagai, Y. (1983). *Bull. Chem. Soc. Jpn*, **56**, 3009–3014.
Murugan, P., Shanmmugasundaram, P., Ramakrishan, V. T., Venkatachalapathy, B., Srividya, N., Ramamurthy, P., Gunasekaran, K. & Velmurugan, D. (1998). *J. Chem. Soc. Perkin Trans.* 2, pp. 999–1003.
Nakano, T., Takahashi, M., Arai, T., Seki, S., Matsumoto, H. & Nagai, Y. (1982). *Chem. Lett.* pp. 613–616.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1994). *XSCANS*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.