



Bond lengths and angles observed in this structure agree well with those found in related structures (Lu *et al.*, 1996; Fun, Lu, Duan, Tian, You, Guo & Gong, 1997). The molecule is in the keto tautomeric form; the dihedral angle between the planes of the phenyl and pyridinyl rings is 11.47(7)<sup>o</sup> and the planes of these two rings form angles of 13.94(9) and 2.48(9)<sup>o</sup>, respectively, with the central hydrazone moiety.

In the crystal, the inversion-related molecules are stacked along the *a* direction with N1···C13(−*x* + 1, −*y*, −*z* + 1) [3.432(3) Å] being the shortest contact involving the pyridine and benzene ring atoms, indicating significant π–π interactions. The stacked molecules are linked by an infinite O···H—O—H···O—H···O cooperative hydrogen-bond network involving the water molecules and carbonyl-O atom to form a column-like structure. The adjacent columns are linked by O2W—H···N1 hydrogen bonds to form a supramolecular structure (Fig 2). O1W is involved in four hydrogen bonds, and O2W in five, since O2W makes a bifurcated hydrogen bond to O1<sup>ii</sup> and N3<sup>ii</sup> (details in Table 2).

## Experimental

The synthesis of the compound was carried out by reaction of *o*-chlorobenzaldehyde and nicotinoylhydrazine in ethanol solution under reflux for 3 h. Single crystals were obtained by recrystallization from ethanol.

### Crystal data

C<sub>13</sub>H<sub>10</sub>ClN<sub>3</sub>O·2H<sub>2</sub>O

*M<sub>r</sub>* = 295.72

Triclinic

*P* $\bar{1}$

*a* = 7.7970(6) Å

*b* = 9.4921(7) Å

*c* = 9.7074(7) Å

α = 89.506(2)<sup>o</sup>

β = 74.124(2)<sup>o</sup>

γ = 79.242(2)<sup>o</sup>

*V* = 678.21(9) Å<sup>3</sup>

*Z* = 2

*D<sub>x</sub>* = 1.448 Mg m<sup>−3</sup>

*D<sub>m</sub>* not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 2848 reflections

θ = 3.00–30.00<sup>o</sup>

μ = 0.293 mm<sup>−1</sup>

*T* = 293(2) K

Parallelepiped

0.46 × 0.26 × 0.14 mm

Colourless

### Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction:

empirical using *SADABS* (Sheldrick, 1996)

*T<sub>min</sub>* = 0.877, *T<sub>max</sub>* = 0.960

5309 measured reflections

3793 independent reflections

2710 reflections with

*I* > 2σ(*I*)

*R<sub>int</sub>* = 0.020

θ<sub>max</sub> = 29.99<sup>o</sup>

*h* = −10 → 10

*k* = −13 → 13

*l* = 0 → 13

### Refinement

Refinement on *F*<sup>2</sup>

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.054

*wR*(*F*<sup>2</sup>) = 0.151

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.299 e Å<sup>−3</sup>

Δρ<sub>min</sub> = −0.344 e Å<sup>−3</sup>

*S* = 1.043

3793 reflections

237 parameters

H atoms: see text

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.071*P*)<sup>2</sup> + 0.1826*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

Extinction correction: none

Scattering factors from

*International Tables for*

*Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

C1—C13	1.7393 (19)	N2—C6	1.352 (3)
O1—C6	1.227 (2)	N2—N3	1.382 (2)
N1—C1	1.333 (3)	N3—C7	1.274 (2)
N1—C5	1.335 (3)		
C1—N1—C5	117.00 (19)	O1—C6—N2	122.44 (18)
C6—N2—N3	118.34 (16)	O1—C6—C4	121.11 (17)
C7—N3—C2	115.45 (17)	N3—C7—C8	120.27 (17)
N1—C1—N2	123.5 (2)		
C6—N2—N3—C7	−175.7 (2)	C3—C4—C6—N2	−4.5 (3)
N3—N2—C6—O1	0.7 (3)	N2—N3—C7—C8	177.5 (2)
N3—N2—C6—C4	−179.0 (2)	N3—C7—C8—C9	8.3 (3)
C5—C4—C6—O1	−5.1 (3)		

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

<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>
N2—H1N2···O1W	0.88 (3)	2.07 (3)	2.932 (3)	168 (3)
C3—H3···O1W	0.91 (3)	2.42 (3)	3.303 (3)	163 (2)
O1W—H1W1···O2W	0.76 (4)	1.96 (4)	2.719 (3)	174 (4)
O1W—H2W1···O1 <sup>i</sup>	0.79 (4)	2.27 (4)	2.992 (3)	152 (4)
O2W—H2W2···O1 <sup>ii</sup>	0.79 (4)	2.34 (4)	3.051 (3)	151 (4)
O2W—H2W2···N3 <sup>ii</sup>	0.79 (4)	2.54 (4)	3.177 (3)	139 (4)
O2W—H1W2···N1 <sup>iii</sup>	0.78 (4)	2.12 (4)	2.898 (3)	176 (4)

Symmetry codes: (i) 1 − *x*, −*y*, 1 − *z*; (ii) 2 − *x*, −*y*, 1 − *z*; (iii) 1 + *x*, *y* − 1, *z*.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180<sup>o</sup>) for the crystal and each exposure of 30 s covered 0.3<sup>o</sup> in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35<sup>o</sup>. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating thirty initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible.

The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996a). Cell refinement: *SAINT* (Siemens, 1996b). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1996c). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1232). Services for accessing these data are described at the back of the journal.

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#### 4-Methyl-7,7a,13a,14-tetrahydrobenzo[e]-pyrano[2',3':5,6]naphtho[2,3-b][1,4]dioxin-2-one

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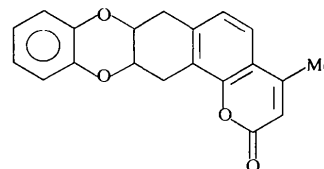
## Abstract

In the title molecule, C<sub>20</sub>H<sub>16</sub>O<sub>4</sub>, the coumarin moiety is planar and both the tetrahydrobenzene and the dioxin rings adopt a half-chair conformation. The mean planes through the tetrahydrobenzene and dioxin rings form a dihedral angle of 72.8 (1)°.

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## Comment

The majority of DNA monointercalating antitumour drugs have a common general structure, comprising a tri- or tetracyclic chromophore to which are attached one or two flexible side chains bearing cationic charges (Palmer *et al.*, 1988). Recently it has been reported that a series of substituted dibenzo[1,4]dioxins show remarkable activity against wild-type P388 leukaemia *in vitro* and *in vivo* (Lee *et al.*, 1992). Because of their antitumour activity and ecotoxicity, different substituted dibenzo[1,4]dioxins have been synthesized and the crystal structure determination of one of them, (I), is reported here.



(I)

Bond lengths and bond angles in the coumarin moiety and C—O distances in the dioxin ring are comparable with reported values (Kumar *et al.*, 1997; Chinnakali *et al.*, 1998; Rissanen *et al.*, 1987). The tetrahydrobenzene ring adopts a half-chair conformation with asymmetry parameter  $\Delta C_2(C8—C7) = 0.045$  (2) (Nardelli, 1983). The dioxin ring also adopts a half-chair conformation with C13 and C14 deviating from the O17—C18—C23—O24 plane by  $-0.436$  (4) and  $0.318$  (4) Å, re-

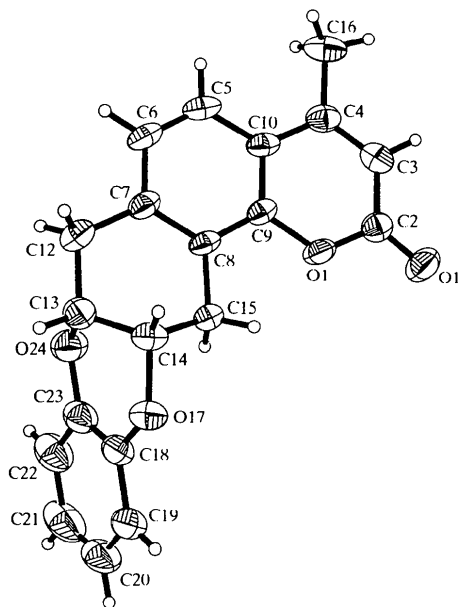


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.