organic papers

N'-tert-Butyl-4-chloro-N'-(4-oxo-4H-chromen-3-ylcarbonyl)benzohydrazide 0.65-hydrate

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Key indicators

Single-crystal X-ray study
$T = 292$ K
Mean σ(C–C) = 0.004 Å
H-atom completeness 94%
Disorder in solvent or counterion
R factor = 0.055
wR factor = 0.182
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.

In the crystal structure of the title compound, $C_{21}H_{19}ClN_2O_4 \cdot 0.65H_2O$, the dihedral angle formed by the bicyclic benzopyran system and the chlorobenzene ring is 55.1 (2)°. In the crystal structure, molecules are linked into molecular tapes through weak C—H···O hydrogen bonds.

Comment

Dibenzoylhydrazines are non-steroidal moulting hormone agonists that have insecticidal activity, while exerting only a low toxicity against non-target insects (Nakagawa *et al.*, 2005). In addition, chromone derivatives are of interest as active natural products, because of their wide spectrum of biological activity, including antibacterial, antifungal, anti-allergic and anti-inflammatory activities (Kim *et al.*, 2004). We have recently focused on the structural modification of the chromone backbone by introduction of dibenzoylhydrazine substituents at the 3-position. The title compound, (I), was investigated as a potential bioactive molecule.
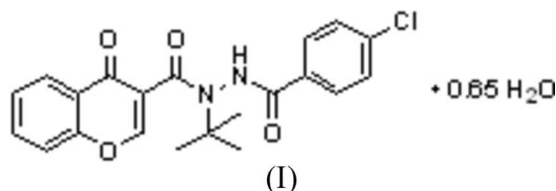
A view of (I) is shown in Fig. 1, and selected bond lengths and angles are listed in Table 1. Atoms C1–C6 in the benzopyran ring system are essentially coplanar (r.m.s. deviation 0.006 Å) and the dihedral angle between this plane and that of atoms C16–C21 (r.m.s. deviation 0.011 Å) of the chlorobenzene ring is 55.1 (2)°.

In the crystal structure, molecules of (I) are linked by weak C—H···O hydrogen bonds to form extended molecular tapes in the *a* axis direction. There are no obvious interactions between adjacent chains.

Experimental

A solution of 4-chlorobenzoic acid N'-tert-butylhydrazide (1.5 mmol) in dichloromethane (10 ml) was added dropwise to a stirred mixture of chromone-3-carbonyl chloride (1.5 mmol), triethylamine (1.6 mmol) and dichloromethane (5 ml) in an ice bath. After stirring the mixture at room temperature for 3 h, ethyl acetate (30 ml) was added to the reaction mixture. The organic layer was separated and washed successively with water (15 ml) and brine (15 ml), and then dried over anhydrous sodium sulfate. After evaporating the solvent, the residue was purified by column chromatography on silica gel using hexane–ethyl acetate (9:1 *v/v*) as eluent to afford (I) (yield
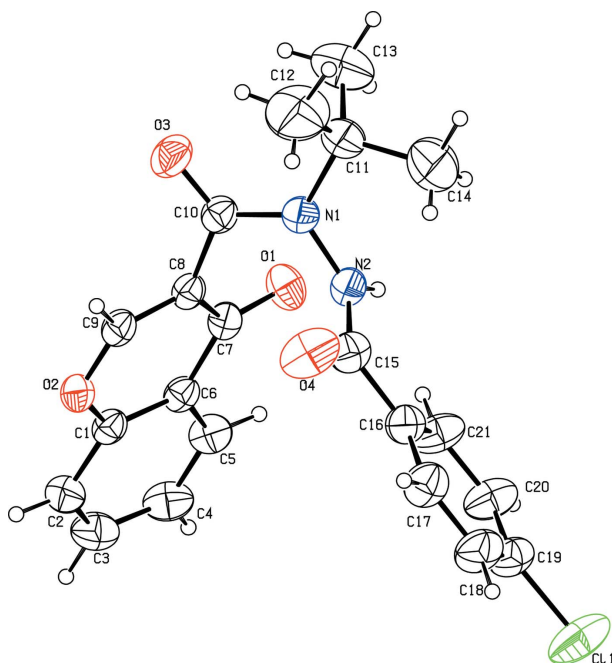
Figure 1
A view of (I), showing 50% probability displacement ellipsoids, with H atoms represented by circles of arbitrary size. The partial-occupancy solvent water molecule is not shown.

58%, m.p. 496 K). Spectroscopic analysis: ^{1}H NMR (CDCl$_3$, 400 MHz, δ, p.p.m.): 11.541 (s, 1H, N—H), 9.142 (s, 1H, C9—H), 8.264 (d, 1H, C5—H), 7.653 (m, 2H, C16—H, C17—H), 7.570 (m, 2H, C3—H, C4—H), 7.402 (d, 1H, C2—H), 7.257 (m, 2H, C18—H, C20—H), 1.648 (m, 9H, C12—H, C13—H, C14—H). MS (EI 70 eV) m/z (%): 398.1 (41), 341.9 (37), 206.3 (100), 153.6 (31), 104.7 (71), 103.6 (64), 105 (39), 76.7 (49), 49.4 (46). Crystals of (I) suitable for X-ray diffraction study were grown from methanol at 292 K.

Crystal data

$C_{21}H_{19}ClN_2O_4\cdot0.65H_2O$	$Z = 2$
$M_r = 409.23$	$D_x = 1.307$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.7913$ (11) Å	Cell parameters from 4083
$b = 11.7863$ (18) Å	reflections
$c = 13.799$ (2) Å	$\theta = 2.6$–27.0°
$\alpha = 75.463$ (2)°	$\mu = 0.22$ mm^{-1}
$\beta = 81.619$ (3)°	$T = 292$ (2) K
$\gamma = 77.108$ (3)°	Block, colourless
$V = 1037.5$ (3) Å^3	$0.30 \times 0.20 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	2910 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.027$
Absorption correction: none	$\theta_{max} = 25.0$°
7452 measured reflections	$h = -7 \rightarrow 8$
3610 independent reflections	$k = -14 \rightarrow 14$
	$l = -16 \rightarrow 16$

Refinement

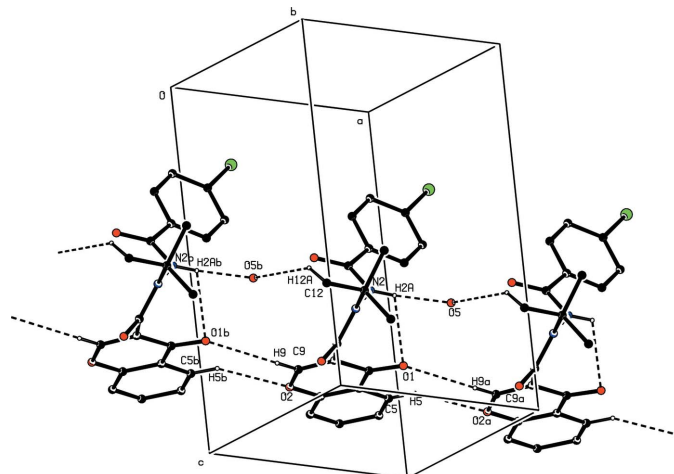|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1051P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$+ 0.222P]$
$wR(F^2) = 0.182$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
3610 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
267 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
H-atom parameters constrained	Extinction correction: *SHELXL97* (Sheldrick, 1997)
	Extinction coefficient: 0.032 (7)

Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (a) $x + 1$, y, z; (b) $x - 1$, y, z.]

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$A\cdots$O5^i	0.96	2.47	3.259 (8)	140
C9—H9$\cdots$O1^i	0.93	2.56	3.403 (3)	152
C5—H5$\cdots$O2^{ii}	0.93	2.54	3.406 (3)	155
N2—H2$A\cdots$O1	0.86	2.47	2.898 (3)	112
N2—H2$A\cdots$O5	0.86	1.98	2.761 (4)	150

Symmetry codes: (i) $x - 1$, y, z; (ii) $x + 1$, y, z.

The H atoms bonded to the partial-occupancy water O atom were neither located nor included in the refinement. All other H atoms were positioned geometrically and treated as riding, with N—H = 0.86 Å and C—H = 0.93 or 0.96 Å. U_{iso}(H) values were set equal to xU_{eq}(carrier atom), where $x = 1.2$ for Csp^2 and $x = 1.5$ for N and methyl C.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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