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1′-*tert*-Butyl-2′-(2,2-dimethyl-4-oxochroman-6-carbonyl)benzohydrazide

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.056
wR factor = 0.138
Data-to-parameter ratio = 15.8

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, $C_{23}H_{26}N_2O_4$, the six-membered heterocyclic ring adopts a half-chair conformation. Intermolecular hydrogen bonds are present.

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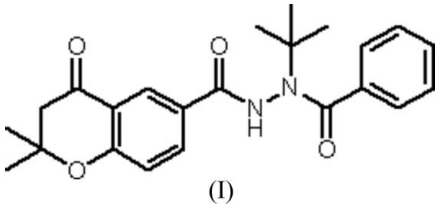
Comment

Dibenzoylhydrazines are well known as non-steroidal ecdysone agonists that have the potential to control lepidopteran pests while exerting only a low toxicity against non-target insects (Yoshihiro *et al.*, 2002). In addition, chroman derivatives also exhibit a wide spectrum of biological activity, including antiviral, anticancer and antibiotic properties (Cho *et al.*, 1997). The title compound, (I), which may be a new precursor for obtaining bioactive molecules, was designed and synthesized in our laboratory. In this paper, we present the X-ray crystallographic analysis of (I).



(I)

As shown in Fig. 1, the six-membered heterocyclic ring adopts a half-chair conformation. The puckering parameters (Cremer & Pople, 1975) corresponding to the sequence O2—C3—C4—C5—C6—C7 are Q = 0.456 (2) Å, Φ_2 = 129.6 (2)° and Θ_2 = 265.8 (4)°. The dihedral angle between the planes of the phenyl ring and the fused bicyclic ring system is 55.1 (2)°

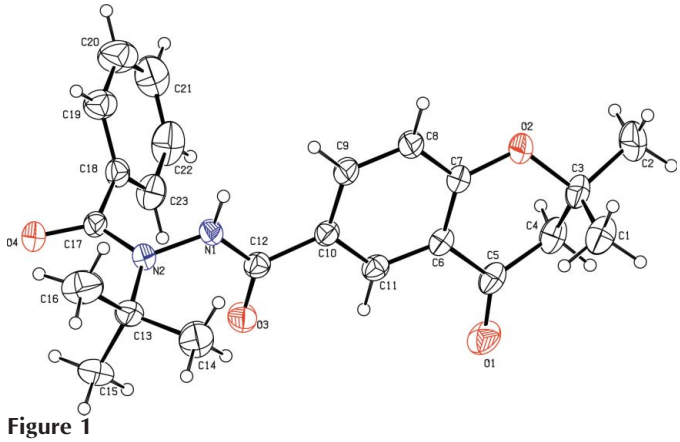


Figure 1
A view of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

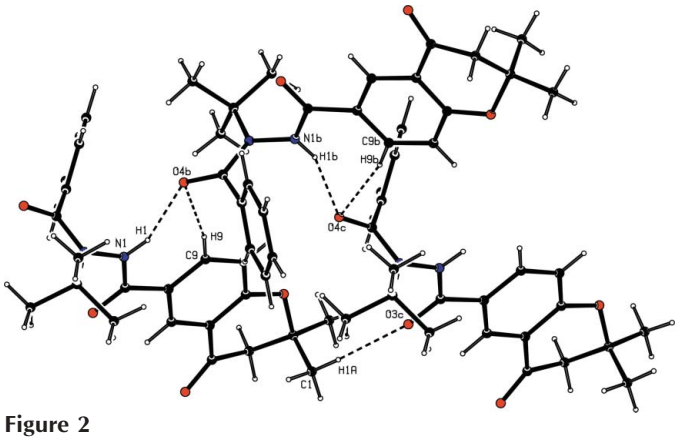


Figure 2
Hydrogen bonding in the crystal structure of (I). Hydrogen bonds are shown as dashed lines. [Symmetry codes: (*b*) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (*c*) $x, y-1, z$.]

(Fig. 1). The bond lengths and angles in the molecule are normal.

One intermolecular N—H···O hydrogen bond and two intermolecular C—H···O hydrogen bonds exist in the crystal structure (Table 1 and Fig. 2). Atoms N1 and C9 in the molecule act as donors, *via* atoms H1*B* and H9*B*, to atom O4 of an adjacent molecule (Table 1). As a result, a seven-membered ring is formed between the molecules (Fig. 3). In addition, atom C1 in the molecule acts as a donor, *via* atom H1*A*, to atom O3 of an adjacent molecule (Table 1). No π–π stacking interactions are observed in the crystal structure.

Experimental

A solution of 2,2-dimethyl-4-oxochroman-6-carboxylic acid *N′-tert*-butyl-hydrazide (1.5 mmol) in dichloromethane (10 ml) was added dropwise to a stirred mixture of benzoyl chloride (1.5 mmol), triethylamine (1.6 mmol) and dichloromethane (5 ml) in an ice bath. After the mixture had been stirred 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. The solvent was evaporated, and the residue was purified by column chromatography on silica gel using hexane/ethyl acetate (9:1, *v*/*v*) as eluant to afford (I) (yield 58%, m.p. 484 K). ^{1}H NMR ($CDCl_3$, 400 MHz, p.p.m.): 8.643 (*s*, 1H, N—H), 7.730 (*d*, 1H, C9—H), 7.702 (*d*, 1H, C11—H), 7.456 (*m*, 2H, C19—H, C23—H), 7.259 (*m*, 3H, C8—H, C20—H, C22—H), 6.884 (*m*, 1H, C21—H), 2.709 (*s*, 2H, C4—H), 1.644 (*m*, 9H, C14—H, C15—H, C16—H), 1.254 (*s*, 6H, C1—H, C2—H); MS (EI 70 eV) *m*/*z*(%): 394 (6), 339 (100), 321 (16), 203 (88), 147 (12),105 (39), 77 (27). Crystals suitable for an X-ray diffraction study were grown from methanol at 292 K.

Crystal data

$C_{23}H_{26}N_2O_4$
M_r = 394.46
Monoclinic, $P2_1/n$
a = 9.5123 (12) Å
b = 9.2752 (11) Å
c = 24.424 (3) Å
β = 95.967 (2)°
V = 2143.2 (5) Å^3
Z = 4
D_x = 1.222 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1949 reflections
θ = 2.4–21.4°
μ = 0.08 mm^{-1}
T = 292 (2) K
Block, colorless
0.30 × 0.20 × 0.10 mm

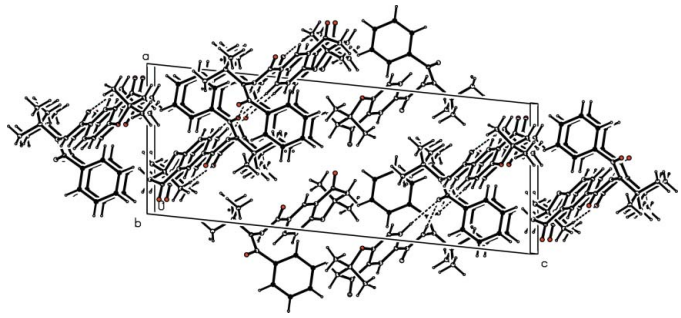


Figure 3
The molecular packing of (I) viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
16302 measured reflections
4217 independent reflections
2694 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 26.0°
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -30 \rightarrow 30$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.138
S = 1.03
4217 reflections
267 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 0.2557P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O3^{i}	0.96	2.54	3.500 (3)	177
N1—H1···O4^{ii}	0.86	2.09	2.903 (2)	157
C9—H9···O4^{ii}	0.93	2.49	3.410 (3)	168

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

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–0.97 Å, an N—H distance of 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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