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3-Ethyl-5-(4-oxochroman-3-ylmethylene)-1,3-imidazolidine-2,4-dione

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Key indicators

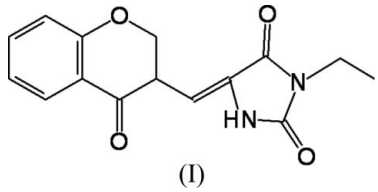
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.114
Data-to-parameter ratio = 15.4

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

The molecule of the title compound, $C_{15}H_{12}N_2O_4$, is planar, with a dihedral angle of 0.97 (4)° between the planes of the five-membered imidazolidine and benzopyran ring systems. The crystal structure is stabilized by C—H···O and N—H···O hydrogen-bonding interactions.

Comment

The prevalence of type 2 diabetes is increasing at a very fast rate. Type 2 diabetes (previously known as non-insulin-dependent diabetes) leads to complications such as blindness, end-stage renal failure, heart disease and amputation. The glitazones are a new class of antidiabetic drugs that act by improving sensitivity to insulin and are used in the treatment of type 2 diabetes (Bradley, 2002). Many natural and synthetic chromones have biological activities which make them of considerable pharmaceutical interest (Gotoda *et al.*, 1998; Wang *et al.*, 1999; Bozdağ-Dündar *et al.*, 2003; Yu *et al.*, 2003; Bozdağ-Dündar *et al.*, 2005). The title compound, (I), is an isostere of 2,4-thiazolidinedione and it was synthesized for antidiabetic activity. Initially, the chemical structure of (I) was evaluated by elementary analysis, and ^{1}H NMR, mass and IR spectroscopic techniques. The crystal structure analysis of (I) was undertaken to elucidate the molecular conformation.



(I)

The molecule of (I) contains a benzopyran ring system and an imidazolidine ring. The benzopyran ring system is almost planar (Fig. 1), and all the bond lengths and angles in the ring have normal values. In the benzopyran ring system, the O1—C6—C5 angle is widened to 121.19 (12)° and C8—C7—C5 is narrowed to 114.90 (12)° from the normal value of 120°. These angles were reported as 121.4 (2) and 114.4 (1)° in 3-methyl-5-(4-oxo-4*H*-chromen-3-yl-methylene)-1,3-thiazolidine-2,4-dione (Aslantaş *et al.*, 2006), 121.4 (2) and 114.9 (2)° in 3-(4-chorobenzyl)-5-(4-oxo-4*H*-chromen-3-yl-methylene)-1,3-thiazolidine-2,4-dione), (II) (Özgen *et al.*, 2005), and 121.2 (2) and 115.6 (2)° in morin (Cody & Luft, 1994). The five-membered imidazolidine ring is in a planar conformation, with a maximum deviation of 0.023 (2) Å for atom C13, and makes a dihedral angle of 0.97 (4)° with the benzopyran ring system. The group attached to N2 is slightly twisted, with torsion angles of 86.33 (2) and 178.1 (1)° for C13—N2—C14—C15 and C14—N2—C12—C11, respectively.

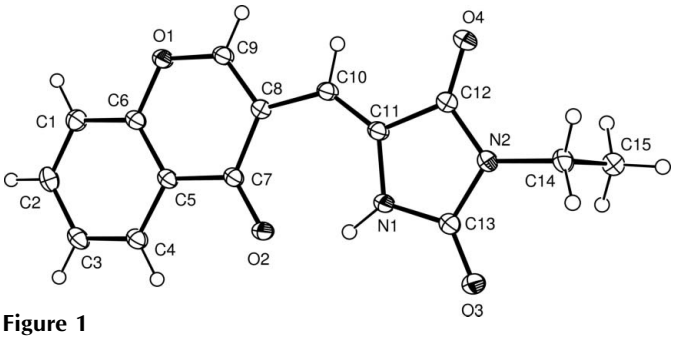


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids at the 40% probability level.

A short intramolecular hydrogen bond formed between O2 and N1 [1.86 (2) Å] is observed. The molecular structure is stabilized by C—H···O and N—H···O hydrogen-bonding interactions (Table 1).

Experimental

The chemical reagents used in the synthesis were purchased from E. Merck (Darmstadt, Germany), Fluka (Buchs, Switzerland) and Aldrich (Milwaukee, MI, USA). A mixture of chromone-3-carboxaldehyde (0.3 g, 1.72 mmol) and imidazolidine-2,4-dione (0.172 g, 1.72 mmol) was heated at 413–423 K in the presence of glacial acetic acid (1 ml) and sodium acetate (0.234 g, 1.72 mmol) for 2 h. The crude product was crystallized from dimethylformamide (DMF). 5-(4-Oxo-4*H*-chromen-3-yl methylene)-imidazolidine-2,4-dione (0.2 g, 0.78 mmol) and anhydrous sodium carbonate (0.166 g, 1.56 mmol) were dissolved in DMF (5 ml). Ethyl iodide (0.25 ml, 3.12 mmol) was added to this mixture and it was stirred at 673 K for 3 h. The reaction mixture was poured on to ice. The residue was filtered off. The filtrate was purified by column chromatography using silica gel 60 (230–400 mesh ASTM) as the adsorbent and petroleum ether–chloroform (1:1) as the eluant (yield 0.11 g, 49.6%; m.p: 2503 K). Spectroscopic analysis: IR (cm^{-1}) (γ pyrone CO): 1671; ^{1}H NMR (DMSO-d_6, 400 MHz, γ, p.p.m.): 1.11 (*t*, 3H, –CH_3), 3.48 (*q*, 2H, –CH_2–), 6.42 (*s*, 1H, ═CH), 7.52 (*ddd*, 1H, 6-H), 7.70 (*d*, 1H, $J_{8,7}$ = 8.80 Hz, 8-H), 7.84 (*ddd*, 1H, 7-H), 8.11 (*dd*, 1H, $J_{5,6}$ = 8.40 Hz, $J_{5,7}$ = 1.60 Hz, 5-H), 8.74 (*s*, 1H, 2-H), 10.53 (*s*, 1H, NH); ESMS[ES (+), m/*z*]: 285 (*M*+1). Calculated for $C_{15}H_{12}N_2O_4$: C 63.38, H 4.22, N 9.86%; found: C 63.42, H 4.48, N 9.79%

Crystal data

$C_{15}H_{12}N_2O_4$
M_r = 284.27
Triclinic, $P\overline{1}$
a = 5.3407 (4) Å
b = 10.4070 (8) Å
c = 12.0223 (9) Å
α = 106.953 (6)°
β = 101.066 (6)°
γ = 92.143 (6)°
V = 624.18 (9) Å^3
Z = 2
D_x = 1.513 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 295 (2) K
Prism, colourless
0.26 × 0.2 × 0.1 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.972, T_{max} = 0.989
9747 measured reflections
3131 independent reflections
2373 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 28.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.114
S = 1.03
3131 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2 + 0.095P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11···O2	0.93 (2)	1.86 (2)	2.6607 (17)	142.9 (18)
C10—H10···O4	0.93 (2)	2.674 (19)	2.995 (2)	101.0 (13)

The H atoms on C10 and N1 were located in difference maps and their coordinates and U_{iso} values were refined freely. In the final stages of refinement, the other H atoms were placed in geometrically idealized positions, with C—H distances of 0.93 Å (aromatic), 0.96 Å (methyl) or 0.97 Å (methylene). The U_{iso}(H) values were set equal to $1.2U_{eq}$(C) [$1.5U_{eq}$(C) for the methyl group].

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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