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2,6-Dimethyl-3-nitro-8*H*-furo[2,3-*g*][1]benzopyran-8-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.069
wR factor = 0.168
Data-to-parameter ratio = 8.5

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

The maximum deviation from the mean plane of the furobenzopyranone skeleton of the title compound, $C_{13}H_9NO_5$, indicates a reasonably planar system. The other non-H atoms are nearly coplanar with this three-ring framework. Intra- and intermolecular C—H···O hydrogen bonds are observed. The crystal packing is determined by such contacts and the molecules are stacked in sheets with a spacing of 3.47 Å.

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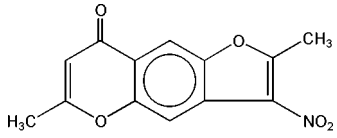
Comment

The title compound, (I), was obtained as an intermediate in the multistage synthesis of derivatives of 4-oxo-4*H*-1-benzopyran-7-carboxylic acid. These derivatives show low toxicity and analgesic and anti-inflammatory activity comparable to acetylsalicylic acid (Kossakowski & Zawadowski, 1995).



(I)

Fig. 1 shows a perspective view of the molecule. The furobenzopyranone system is essentially planar with no atomic deviation greater than 0.052 (3) Å from its least-squares plane (r.m.s. deviation 0.029 Å). Both the nitro group and atoms O14, C15 and C19 are found to be not markedly out of this plane. The deviations range from −0.134 (5) for C9 to 0.060 (5) Å for O14. All bond lengths and valence angles observed in the compound are in good agreement with the corresponding distances in related compounds (Léger *et al.*, 1981; Ajana *et al.*, 1987; El-Sayed *et al.*, 1988; Hariharan &

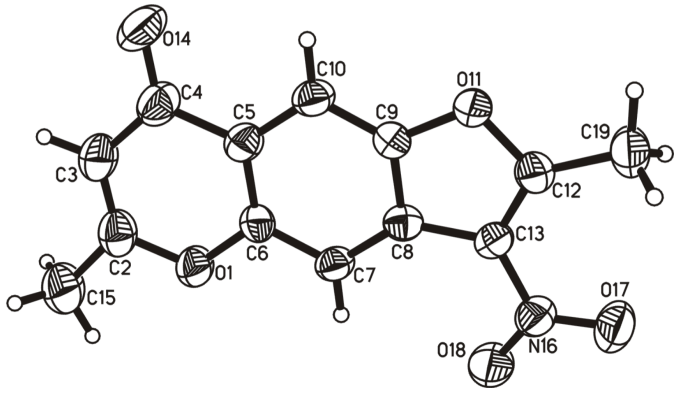


Figure 1
A view of the title compound together with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

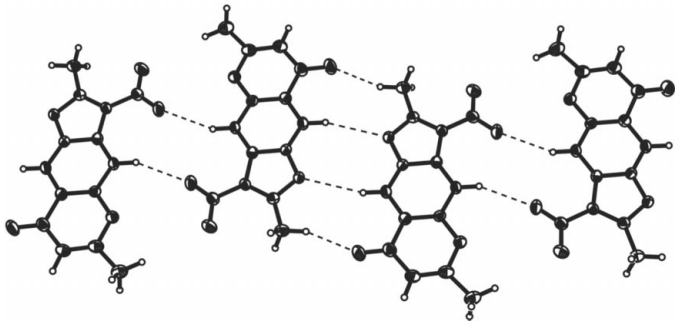


Figure 2
A view showing the interconnections within a sheet. Dashed lines indicate hydrogen bonds.

Rajan, 1990; Ginderow, 1991; Koh & Ng, 1993). The structure is stabilized by C—H···O-type contacts and stacking forces. The molecules connected through inversion centers are linked by C10—H10···O11, C7—H7···O18 and C19—H19*A*···O14 hydrogen bonds, forming sheets (Fig. 2) along the [120] and [1$\bar{2}$0] directions. The perpendicular distance between partly overlapping molecules from neighbouring parallel sheets is 3.47 Å. Cohesion between non-parallel sheets results also in C—H···O (C3—H3···O14 and C15—H15C···O17) interactions (Fig. 3). The geometric parameters of all intra- and intermolecular hydrogen bonds are given in Table 1.

Experimental

The synthesis of the title compound has been described elsewhere (Kossakowski & Zawadowski, 1995). Crystals were grown from acetic acid by slow evaporation.

Crystal data

$C_{13}H_9NO_5$
M_r = 259.21
Monoclinic, $P2_1/c$
a = 8.111 (2) Å
b = 5.732 (1) Å
c = 24.235 (5) Å
β = 93.51 (3)°
V = 1124.6 (4) Å^3
Z = 4
D_x = 1.531 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2228 reflections
θ = 2.5–21.2°
μ = 0.12 mm^{-1}
T = 293 (2) K
Prism, orange
0.60 × 0.25 × 0.03 mm

Data collection

Kuma KM-4 CCD diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4062 measured reflections
1463 independent reflections
1329 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 22.5°
$h = -8 \rightarrow 8$
$k = -4 \rightarrow 6$
$l = -26 \rightarrow 24$

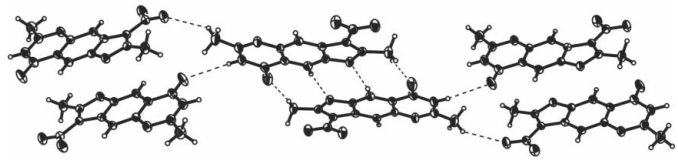


Figure 3
A view showing the interconnections between sheets

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.168
S = 1.13
1463 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 1.4018P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O18	0.93	2.53	2.985 (5)	110
C10—H10···O11^{i}	0.93	2.68	3.583 (4)	164
C7—H7···O18^{ii}	0.93	2.44	3.309 (5)	156
C3—H3···O14^{iii}	0.93	2.74	3.525 (5)	143
C19—H19*A*···O14^{i}	0.96	2.27	3.191 (5)	160
C15—H15*C*···O17^{iv}	0.96	2.65	3.391 (6)	134

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $-x, 3-y, 1-z$; (iii) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iv) $x, \frac{5}{2}-y, \frac{1}{2}+z$.

The H atoms were refined as riding and their U_{iso} values were set at 1.2 (1.5 for methyl groups) times U_{eq} of their carrier atoms.

Data collection: *CRYSALIS CCD* (Kuma, 1999); cell refinement: *CRYSALIS RED* (Kuma, 1999); data reduction: *CRYSALIS RED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *SHELXL*97.

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