Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(4-Chloro-2-phenyl-2*H*-chromen-3-ylmethylene)-*N*′-[4-(dimethylamino)benzoyl]hydrazide ethanol solvate

Aihua Nie, Sutapa Ghosh and Ziwei Huang*

The Burnham Institute for Medical Research, 10901 N. Torrey Pines Rd., La Jolla, CA 92037, USA

Correspondence e-mail: ziweihuang@burnham.org

Received 22 March 2006
Accepted 26 March 2006

The structure of the title compound, $C_{25}H_{22}ClN_3O_2\cdot C_2H_6O$, displays N—H···O, O—H···O, O—H···N, C—H···Cl, C—H···O hydrogen bonding.

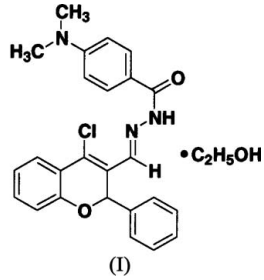
Key indicators

Single-crystal X-ray study
T = 208 K
Mean σ(C–C) = 0.004 Å
R factor = 0.061
wR factor = 0.159
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.

Comment

It has been demonstrated that flavanone and its derivatives have potential bioactivities against cancer (Senderowicz, 1999; Brueggemeier *et al.*, 2001; Bauvois *et al.*, 2003). By microwave-assisted synthesis we constructed a library of flavanone derivatives including the title compound, (I).



(I)

All the bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987). The structural data confirm that the plane of the phenyl ring (C11–C15) is perpendicular to the plane of the 2*H*-pyran ring and to the rest of the molecule [dihdral angle = 89.0 (6)°]. In the crystal structure, atoms (N2)H2A and O2 of the linker region form intermolecular hydrogen-bonding interactions with the ethanol solvent molecule (Table 1).

Experimental

For the preparation of the title compound, 4-chloro-2-phenyl-2*H*-chromene-3-carbaldehyde (27.1 mg, 0.1 mmol) and 4-dimethylaminobenzoic acid hydrazide (17.9 mg, 0.1 mmol) were dissolved in ethanol (1 ml). The solution was heated to 363 K for 30 min. by microwave irradiation. After cooling to room temperature, the resulting precipitate was collected by filtration and dried to afford the title compound as yellow crystals (yield 36.8 mg, 77.0%).

Crystal data

$C_{25}H_{22}ClN_3O_2\cdot C_2H_6O$
M_r = 477.97
Triclinic, $P\overline{1}$
a = 9.710 (2) Å
b = 10.668 (2) Å
c = 12.002 (3) Å
α = 78.409 (3)°
β = 88.988 (3)°
γ = 78.532 (3)°
V = 1193.3 (4) Å^3
Z = 2
D_x = 1.330 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5716 reflections
θ = 2.6–28.1°
μ = 0.20 mm^{-1}
T = 208 (2) K
Block, yellow
0.15 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.971, T_{max} = 0.981
12114 measured reflections
4200 independent reflections
3233 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 25.0°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.159
S = 1.06
4200 reflections
311 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0855P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3^{i}	0.87	2.20	2.907 (3)	138
O3—H3A···O2	0.83	2.01	2.805 (3)	160
O3—H3A···N1	0.83	2.58	3.170 (3)	129
C1—H1···Cl1	0.94	2.67	3.055 (3)	106
C10—H10···Cl1	0.94	2.65	3.032 (3)	105
C25—H25A···O2^{ii}	0.97	2.58	3.486 (4)	156

Symmetry codes: (i) $-x+2, -y, -z$; (ii) $-x+2, -y-1, -z$.

All H atoms were positioned geometrically with C—H = 0.94–0.99, N–H = 0.87 and O–H = 0.83Å, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C,N) for the CH, NH and CH_2 groups and 1.5U_{eq}(C,O) for CH_3 and OH groups].

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

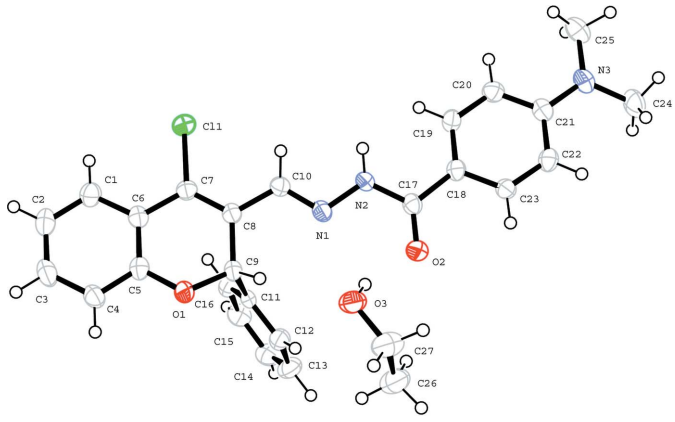


Figure 1
The structure of the title compound, (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

This work was supported by grants from the NIH and the ACS. We thank the X-ray Facility of the Chemistry Department of the University of California at San Diego for technical assistance.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Bauvois, B., Puiffe, M.-L., Bongui, J.-B., Paillat, S., Monneret, C. & Dauzonne, D. (2003). *J. Med. Chem.* **46**, 3900–3913.
Brueggemeier, R. W., Richards, J. A., Joomprabutra, S., Bhat, A. S. & Whetstone, J. L. (2001). *J. Ster. Biochem. Mol. Biol.* **79**, 75–84.
Bruker (2005). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Senderowicz, A. M. (1999). *Invest. New Drugs*, **17**, 313–320.
Sheldrick, G. M. (1997). *SADABS* and *SHELXL97*. University of Göttingen, Germany.