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2-[2-(Methylamino)-3-nitro-4*H*-chromen-4-yl]-phenyl acetate

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In the title compound, $C_{18}H_{16}N_2O_5$, the pyran ring adopts a flattened envelope conformation. In the crystal structure, N—H···O and C—H···O hydrogen bonds generate centrosymmetric dimers with $R_2^2(12)$ and $R_2^2(20)$ ring motifs, respectively. In addition, *C*(8) chains generated by C—H···O hydrogen bonds are observed along the *b* axis.

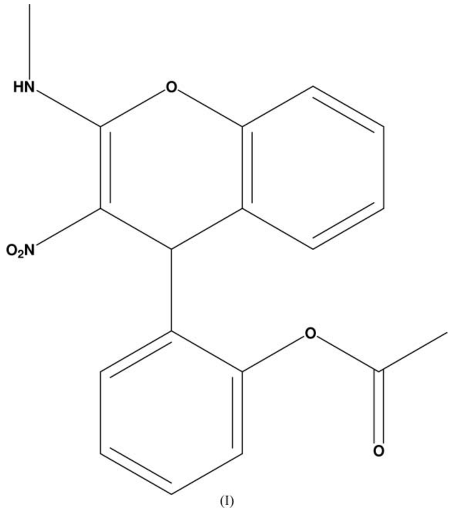
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.125
Data-to-parameter ratio = 16.5

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

Comment

Chromenes have been widely employed as important intermediates in the synthesis of natural products and medicinal agents (Wang & Finn, 2000). They have been found to possess anti-inflammatory and anti-allergic properties, and these derivatives can also yield a new family of potassium-channel activating drugs (Atwal et al., 1995; Salamon et al., 2002). In view of the above importance, the structural study of the title compound, (I), was undertaken



(I)

Bond lengths and angles in (I) (Fig. 1) are comparable to those observed for *N*,6-dimethyl-4-(methylsulfanyl)-3-nitro-4*H*-chromen-2-amine (Gayathri *et al.*, 2006). The Csp^2—N and N—O bond lengths (Table 1) deviate slightly from the corresponding mean values of 1.353 (7) and 1.218 (13) Å, respectively (Allen *et al.*, 1987). The sum of the bond angles around N2 (360.0°) indicates sp^2-hybridization.

The pyran ring adopts a flattened envelope conformation, with atom C7 deviating from the O1/C1/C6/C8/C9 plane by 0.092 (2) Å. The puckering parameters (Cremer & Pople, 1975) Q, θ and φ are 0.065 (1) Å, 108.6 (9)° and 6.2 (9)°, respectively. The C1–C6 and C11–C16 benzene rings form dihedral angles of 0.97 (7) and 86.20 (3)°, respectively, with the O1/C1/C6/C8/C9 plane.

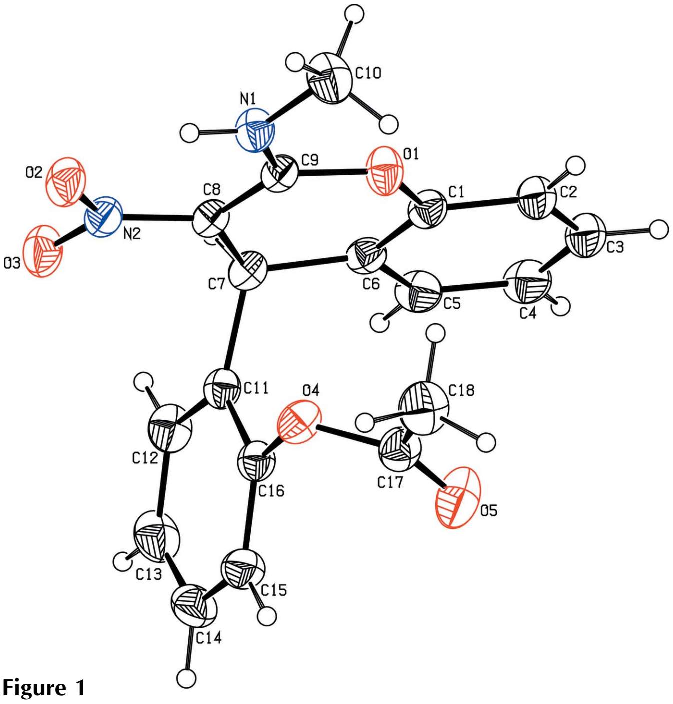


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

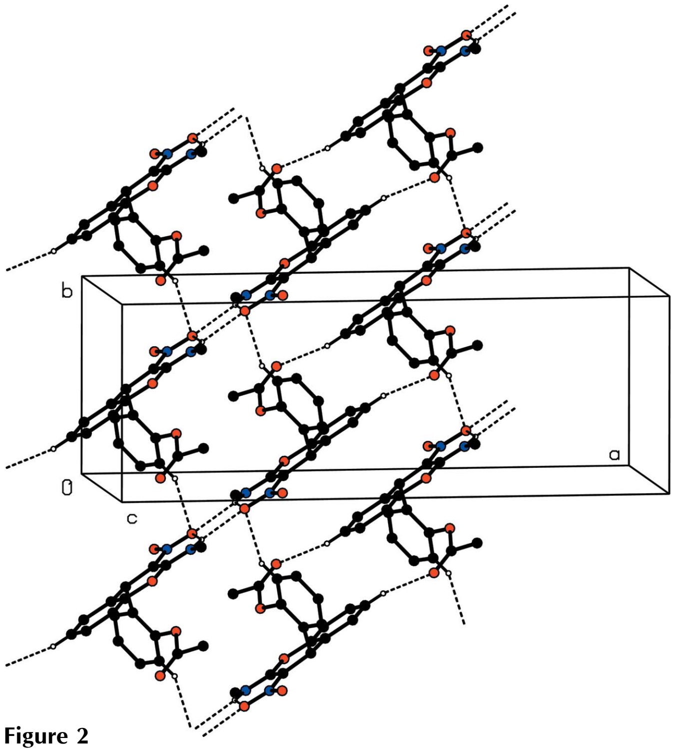


Figure 2
Part of the crystal packing of (I), viewed approximately along the *c* axis. For clarity, H atoms not involved in the hydrogen bonds have been omitted. Hydrogen bonds are shown as dashed lines.

The molecular structure is stabilized by an N1—H1···O2 intramolecular hydrogen-bonding interaction, which generates an *S*(6) ring motif. The crystal packing is stabilized by N1—H1···O2^{i} and C4—H4···O5^{ii} hydrogen bonds (Table 2), which generate centrosymmetric dimers with $R_2^2(12)$ and $R_2^2(20)$ ring motifs, respectively. In addition, the C15—H15···O2^{iii} interactions generate a *C*(8) chain along the *b* axis. Symmetry codes are given in Table 2.

Experimental

To a well stirred mixture of 2-[2-(methylamino)-3-nitro-4*H*-4-chromenyl]phenol (1.6 mmol) and acetic anhydride (5.1 mmol), pyridine (0.2 mmol) was added at room temperature. The reaction mixture was stirred thoroughly for 16 h. Unreacted pyridine was quenched with 0.01 *N* HCl (60 ml). The reaction mixture was diluted with dichloromethane (35 ml), and the organic layer was washed with water and then with brine. Evaporation of the solvent afforded compound (I) as a pale-yellow solid. Single crystals of (I) were grown by slow evaporation of a hexane–dichloromethane (8:2 *v*/*v*) solution.

Crystal data

$C_{18}H_{16}N_2O_5$	$V = 3193.7$ (3) Å^3
$M_r = 340.33$	$Z = 8$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 23.7412$ (13) Å	$\mu = 0.11$ mm^{-1}
$b = 8.3846$ (5) Å	$T = 293$ (2) K
$c = 16.1457$ (9) Å	0.26 × 0.25 × 0.23 mm
$\beta = 96.440$ (1)°	

Data collection

Bruker SMART APEX area-detector diffractometer	3756 independent reflections
Absorption correction: none	3193 reflections with $I > 2\sigma(I)$
17564 measured reflections	$R_{\rm int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	228 parameters
$wR(F^2) = 0.125$	H-atom parameters constrained
$S = 1.05$	$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
3756 reflections	$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.392 (1)	C10—N1	1.451 (2)
C9—N1	1.310 (2)	N2—O3	1.246 (1)
C9—O1	1.341 (1)	N2—O2	1.271 (1)
C9—N1—C10	124.7 (1)	C17—O4—C16	119.8 (1)
C9—O1—C1	121.1 (1)		
O1—C9—N1—C10	4.6 (2)	O5—C17—O4—C16	7.0 (2)
C8—C9—N1—C10	−175.5 (1)	C18—C17—O4—C16	−173.8 (1)
C9—C8—N2—O3	175.4 (1)	C11—C16—O4—C17	−122.0 (1)
C9—C8—N2—O2	−5.4 (2)	C15—C16—O4—C17	62.1 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	1.97	2.591 (1)	129
N1—H1···O2^{i}	0.86	2.25	2.966 (1)	140
C4—H4···O5^{ii}	0.93	2.55	3.427 (2)	157
C15—H15···O2^{iii}	0.93	2.47	3.323 (2)	152

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

H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.86 Å, C—H = 0.93–0.98 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(methyl C) or 1.2$U_{\rm eq}$(CN).

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Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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