

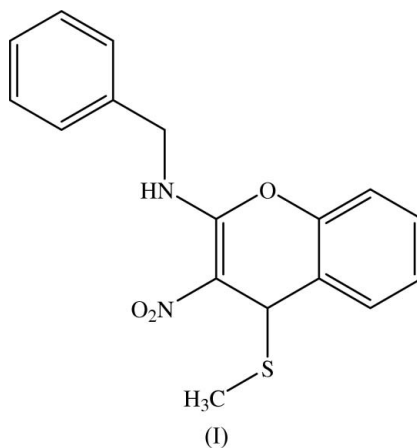
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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(C-C) = 0.003$ Å
 R factor = 0.045
 wR factor = 0.138
Data-to-parameter ratio = 18.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.***N*-Benzyl-*N*-[4-(methylsulfanyl)-3-nitro-4*H*-chromen-2-yl]amine**In the title compound, $C_{17}H_{16}N_2O_3S$, the pyran ring adopts a flattened envelope conformation. $N-H \cdots O$ hydrogen bonds generate a centrosymmetric $R_2^2(12)$ dimer. The dimers translated by a unit cell along the c axis are linked *via* $C-H \cdots S$ hydrogen bonds to form a $C(6)$ chain.

Comment

Chromenes (2*H*-1-benzopyran derivatives) are frequently found in naturally occurring heterocycles, many of which exhibit biological activity (Bowers *et al.*, 1976). They have been widely employed as important intermediates in the synthesis of natural products and medicinal agents (Wang & Finn, 2000). Chromene derivatives can 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.The $N-O$, $C-S$ and $C_{sp^2}-N$ bond lengths (Table 1) deviate slightly from the corresponding mean values of 1.218 (13), 1.819 (19) and 1.353 (7) Å, respectively (Allen *et al.*, 1987). The difference in the $C-O$ bond lengths in the pyran ring is an observation common to the chromene system. The $C5-C6-C7$, $C6-C5-C10$ and $C6-C7-C8$ angles deviate from 120° , which may be due to the fusion of the benzene ring ($C5-C10$) with the non-planar pyran ring. The $C4-S1-C18$ angle of $101.1(1)^\circ$ is comparable with that reported for a diazapentacyclo compound (Bhaskaran *et al.*, 2003).The pyran ring adopts a flattened envelope conformation, with atom C4 deviating from the $O1/C2/C3/C5/C6$ plane by $0.229(2)$ Å. The asymmetry parameter (Nardelli, 1995) $\Delta C_s(C4)$ is $0.7(2)^\circ$, and the puckering parameters (Cremer & Pople, 1975) Q , θ and φ are $0.175(1)$ Å, $80.7(5)^\circ$ and

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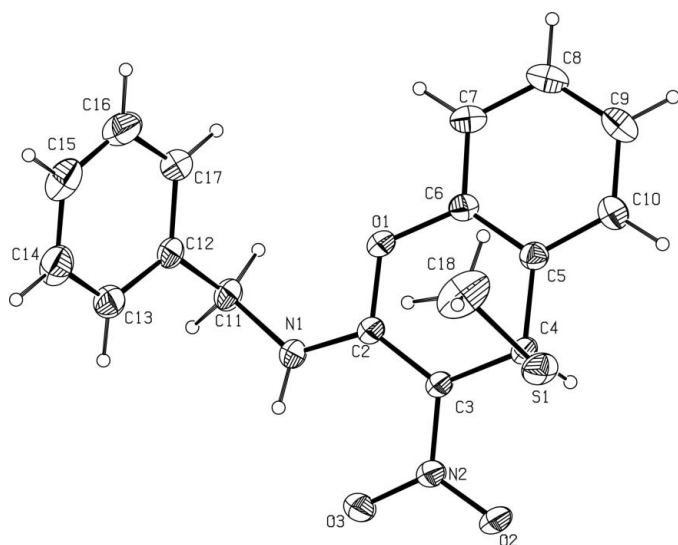


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

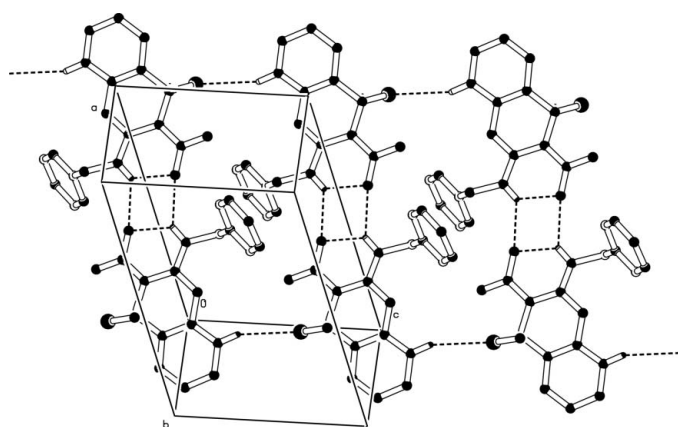


Figure 2
The crystal packing of (I). For clarity, H atoms not involved in the hydrogen-bonding interactions have been omitted. Hydrogen bonds are shown as dashed lines.

178.8 (5)°, respectively. The small puckering may be attributed to the resonance in the pyran ring. The O—N—C—C torsion angles (Table 1) indicate that the nitro group is twisted slightly away from the chromene system (Fig. 1). The dihedral angle between the O1/C2/C3/C5—C10 and C12—C17 planes is 60.63 (6)°.

In the molecular structure, an intramolecular N1—H1ⁱ⋯O3 hydrogen bond generates an S(6) ring motif (Bernstein *et al.*, 1995). The crystal packing is stabilized by N—H⋯O and C—H⋯S intermolecular hydrogen bonds (Table 2). The N1—H1ⁱ⋯O3ⁱ and N1ⁱ—H1ⁱ⋯O3 (symmetry code as in Table 2) hydrogen bonds generate a centrosymmetric R₂²(12) dimer centred at (½, 0, 1). The dimers translated by a unit cell along the *c* axis are linked *via* C7—H7⋯S1ⁱⁱ hydrogen bonds to form a C(6) chain (Fig. 2).

Experimental

To a mixture of 2-hydroxybenzaldehyde (27 mmol) and 60% NaH (40 mmol) in dry tetrahydrofuran (THF, 10 ml) *N*-benzyl-*N*-[(*E*)-1-methylsulfanyl]-2-nitro-1-ethenyl]amine (27 mmol) was added under a nitrogen atmosphere. The reaction mixture was stirred thoroughly for about 1 h and then refluxed for 7 h. Unreacted NaH was quenched with a few drops of acetic acid. The reaction mixture was diluted with dichloromethane (35 ml) and washed with water and brine solution. Evaporation of the solvent and column chromatography (silica gel, hexane and ethyl acetate) afforded compound (I) as a pale-yellow solid. Single crystals were grown by slow evaporation of a hexane–ethyl acetate (8:2 *v/v*) solution.

Crystal data

C₁₇H₁₆N₂O₃S
M_r = 328.38
 Monoclinic, *P*2₁/*c*
a = 10.6411 (7) Å
b = 19.6795 (12) Å
c = 8.2268 (5) Å
 β = 109.795 (1)°
V = 1620.99 (18) Å³
Z = 4

D_x = 1.346 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 9200 reflections
 θ = 2.0–28.0°
 μ = 0.22 mm⁻¹
T = 293 (2) K
 Block, pale yellow
 0.21 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area detector
 diffractometer
 ω scans
 Absorption correction: none
 18458 measured reflections
 3844 independent reflections

3140 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.022
 θ_{\max} = 28.0°
h = -13 → 14
k = -25 → 25
l = -10 → 10

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.045
wR (*F*²) = 0.138
S = 1.03
 3844 reflections
 209 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0853P)^2 + 0.1762P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.14 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

N2—O2	1.243 (2)	C4—S1	1.843 (1)
N2—O3	1.256 (2)	S1—C18	1.774 (2)
N2—C3	1.383 (2)	C3—C2	1.394 (2)
N1—C2	1.314 (2)	C2—O1	1.342 (2)
N1—C11	1.468 (2)	O1—C6	1.393 (2)
C4—C3	1.487 (2)	C11—C12	1.501 (2)
C4—C5	1.501 (2)		
O2—N2—O3	120.4 (1)	C5—C6—C7	123.1 (1)
O2—N2—C3	119.1 (1)	C5—C6—O1	122.1 (1)
O3—N2—C3	120.6 (1)	C6—C5—C10	117.3 (1)
C3—C4—C5	110.9 (1)	C9—C10—C5	121.0 (2)
C18—S1—C4	101.1 (1)	C6—C7—C8	118.1 (2)
C2—C3—C4	122.9 (1)	C13—C12—C17	118.5 (2)
O1—C2—C3	120.3 (1)	C14—C13—C12	121.5 (2)
C2—O1—C6	120.3 (1)		
O2—N2—C3—C2	172.60 (13)	C4—C3—C2—O1	3.2 (2)
O3—N2—C3—C2	-6.9 (2)	C3—C2—O1—C6	11.1 (2)
O2—N2—C3—C4	-5.0 (2)	C2—O1—C6—C5	-11.7 (2)
O3—N2—C3—C4	175.48 (13)	O1—C6—C5—C4	-2.4 (2)
C5—C4—C3—C2	-15.3 (2)	C3—C4—C5—C6	14.6 (2)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O3$	0.86	2.00	2.600 (2)	126
$N1-H1\cdots O3^i$	0.86	2.31	2.989 (2)	136
$C7-H7\cdots S1^{ii}$	0.93	2.87	3.743 (2)	157

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

H atoms were positioned geometrically and were treated as riding on their parent C atoms, with $N-H = 0.86$ Å and $C-H = 0.93-0.98$ Å, and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms or $1.2U_{eq}(C)$ for other H atoms.

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: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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