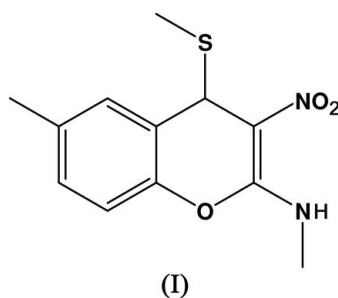


N*,6-Dimethyl-4-(methylsulfanyl)-3-nitro-4*H*-chromen-2-amine*D. Gayathri,^a D. Velmurugan,^{a*}
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Key indicatorsSingle-crystal X-ray study
T = 293 K
Mean $\sigma(C-C)$ = 0.002 Å
R factor = 0.039
wR factor = 0.121
Data-to-parameter ratio = 17.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound, C₁₂H₁₄N₂O₃S, the pyran ring adopts an envelope conformation. An N—H···O hydrogen bond generates an *S*(6) ring motif and intermolecular C—H···O interactions generate a centrosymmetric *R*₂²(14) dimer.Received 5 April 2006
Accepted 15 April 2006**Comment**Chromene derivatives have recently received considerable attention owing to their synthetic and pharmaceutical importance, and they are frequently found in naturally occurring heterocycles, many of which exhibit biological activity. These derivatives inhibit bacterial lipopolysaccharide (LPS)-stimulated production of TNF- α from human peripheral blood mononuclear cells (PBMC). Additionally, these compounds inhibit NF- κ B-mediated transcription activation (Cheng *et al.*, 2003).The *C*_{sp²}—N, C—S and N—O bond lengths in the title compound, (I) (Table 1), deviate slightly from the corresponding mean values of 1.353 (7), 1.819 (19) and 1.218 (13) Å, respectively (Allen *et al.*, 1987). The bond angles around atom C3 show deviation from the ideal tetrahedral value. The bond lengths and angles are comparable to those observed for *N*-benzyl-*N*-[4-(methylsulfanyl)-3-nitro-4*H*-chromen-2-yl]amine (Bhaskaran *et al.*, 2006).The pyran ring adopts an envelope conformation, with atom C3 deviating from the O1/C1/C2/C4/C5 plane by 0.222 (2) Å. The asymmetry parameter (Nardelli, 1983) $\Delta C_s(C3)$ is 0.7 (2)°, and the puckering parameters (Cremer & Pople, 1975) *Q*, θ and φ are 0.164 (1) Å, 103.6 (5)° and 358.8 (5)°, respectively. The dihedral angle between the O1/C1/C2/C4/C5 and C4—C9 planes is 5.05 (6)°. The nitro and methylsulfanyl groups are nearly coplanar with the chromene system (Fig. 1).The molecular structure is stabilized by an intramolecular N1—H1···O2 hydrogen-bonding interaction, which generates an *S*(6) ring motif (Bernstein *et al.*, 1995). The crystal packing is stabilized by C—H···O intermolecular interactions

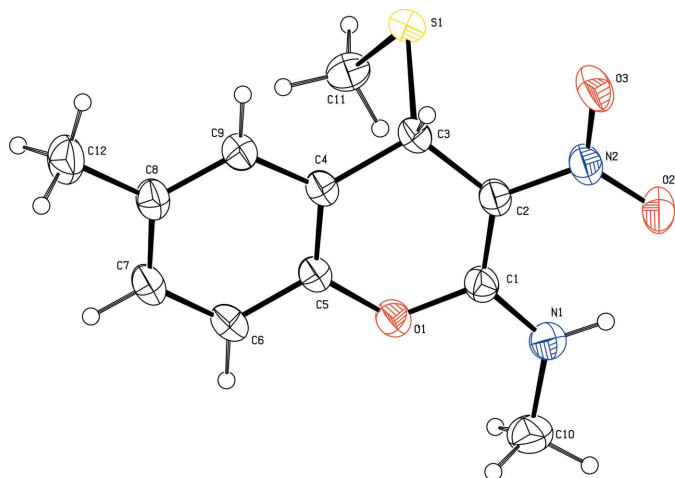


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids.

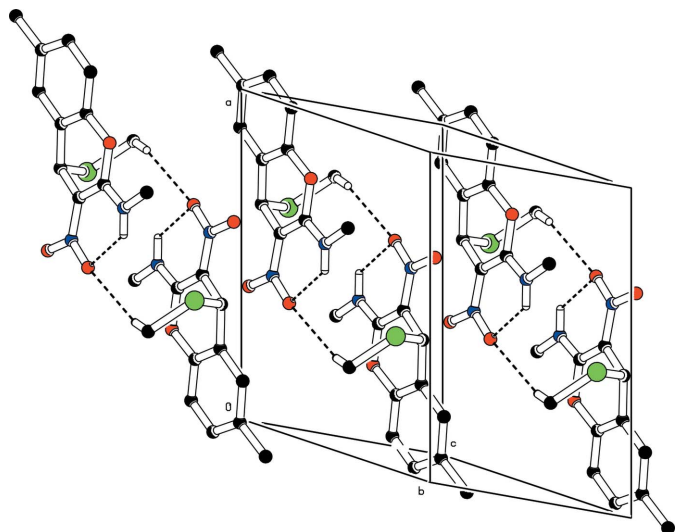


Figure 2
The crystal packing of (I). For clarity, H atoms not involved in the hydrogen-bonding interactions have been omitted. Hydrogen bonds are shown as dashed lines.

(Table 2), which generate a centrosymmetric $R_2^2(14)$ dimer centred at $(\frac{1}{2}, 0, \frac{1}{2})$.

Experimental

To a mixture of 5-methyl-2-hydroxybenzaldehyde (27 mmol) and 60% NaH (40 mmol) in dry tetrahydrofuran (7 ml), *N*-methyl-*N*-[(*E*)-1-(methylsulfanyl)-2-nitro-1-ethenyl]amine (27 mmol) was added under a nitrogen atmosphere. The reaction mixture was stirred thoroughly for 1 h and then refluxed for 8 h. Unreacted NaH was quenched with a few drops of acetic acid. The reaction mixture was diluted with dichloromethane (50 ml), and washed with a 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 (7:3 v/v) solution.

Crystal data

$C_{12}H_{14}N_2O_3S$
 $M_r = 266.31$
Monoclinic, $P2_1/c$
 $a = 10.5558$ (5) Å
 $b = 17.5031$ (8) Å
 $c = 6.9415$ (3) Å
 $\beta = 101.863$ (1)°
 $V = 1255.11$ (10) Å³

$Z = 4$
 $D_x = 1.409$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 293$ (2) K
Block, pale yellow
0.22 × 0.22 × 0.19 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
Absorption correction: none
14189 measured reflections

2962 independent reflections
2716 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.019$
 $\theta_{max} = 28.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.121$
 $S = 1.07$
2962 reflections
166 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0707P)^2 + 0.2498P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.004$
 $\Delta\rho_{max} = 0.31$ e Å⁻³
 $\Delta\rho_{min} = -0.16$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C1–N1	1.317 (2)	C10–N1	1.449 (2)
C1–O1	1.341 (2)	C11–S1	1.793 (2)
C2–N2	1.380 (2)	N2–O3	1.243 (2)
C3–S1	1.862 (1)	N2–O2	1.264 (2)
C5–O1	1.395 (2)		
N2–C2–C3	116.5 (1)	C8–C9–C4	121.9 (1)
C1–C2–C3	122.8 (1)	C1–N1–C10	124.8 (1)
C5–C4–C9	117.4 (1)	O3–N2–O2	120.5 (1)
C9–C4–C3	122.3 (1)	O3–N2–C2	118.6 (1)
C4–C5–O1	122.5 (1)	C11–S1–C3	100.9 (1)
C6–C5–O1	115.3 (1)		
O1–C1–C2–N2	176.6 (1)	C3–C2–N2–O3	3.9 (2)
N2–C2–C3–S1	70.6 (1)	C1–C2–N2–O2	2.8 (2)
O1–C1–N1–C10	–5.3 (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 O2	0.86	1.99	2.611 (2)	128
C11–H11A \cdots O2 ⁱ	0.96	2.44	3.243 (2)	141

Symmetry code: (i) $-x + 1, -y, -z + 1$.

H atoms were placed in calculated positions, with an N–H distance of 0.86 Å and C–H distances in the range 0.93–0.98 Å. The $U_{iso}(H)$ values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; 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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