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4-Methyl-7-phenylsulfonamido-2*H*-1-benzopyran-2-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.124
Data-to-parameter ratio = 12.5

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

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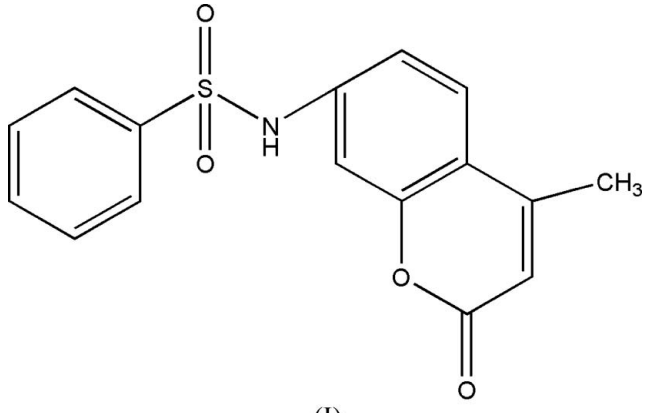
In the title compound, $C_{12}H_{11}NO_3$, the molecules have two intramolecular C—H···O hydrogen bonds that define an $S(6)S(5)$ pattern. The molecules are linked by N—H···O hydrogen bonds into a centrosymmetric dimer. These dimers are linked by further C—H···O hydrogen bonds, so forming a [100] ribbon of $R_2^2(8)R_4^4(18)[R_4^4(20)]$ rings, a $[1\bar{1}1]$ chain of alternating $R_2^2(8)$ and $R_2^2(22)$ rings, and an [010] complex chain of $R_4^2(18)$ rings. The combination of the [100] ribbon, the $[1\bar{1}1]$ chain and the [010] chain results in the formation of a three-dimensional network structure.

Comment

Aminocoumarin derivatives are known to be a category of important fluorogenic dyes (Lemieux *et al.*, 2003; Zhao *et al.*, 2004). Some aminocoumarin derivatives show high activity as antiplatelet agents (Roma *et al.*, 2003). We recently reported the molecular structure of an aminocoumarin (Yang *et al.*, 2006). Here, we report the molecular structure of the title aminocoumarin derivative, (I).



(I)

In the molecule of (I), the molecule adopts a similar 'vault' conformation, and two intramolecular C—H···O hydrogen bonds define an $S(6)S(5)$ pattern (García-Báez *et al.*, 2002) (Fig. 1 and Table 1). The dihedral angle between the six-membered ring O3/S1/N1/C7/C6/H1 and the five-membered ring O3/S1/C11/C12/H12 is 80.28 (9)°. The coumarin unit and the phenyl ring (C11–C16) make a dihedral angle of 75.21 (8)°. The geometric parameters for (I) are normal (Allen *et al.*, 1987).

In the crystal structure of (I), the molecules are linked by a pair of N—H···O hydrogen bonds into a centrosymmetric dimer of $R_2^2(8)$ ring motif (Bernstein *et al.*, 1995), centred at $(\frac{1}{2}, 1, 1)$ (Fig. 2 and Table 1).

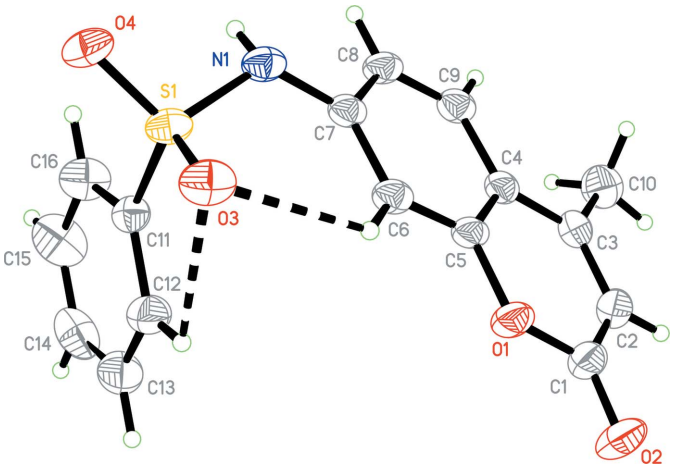


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme and intramolecular hydrogen-bonded $S(5)S(6)$ rings. Displacement ellipsoids are drawn at the 30% probability level. Dashed lines indicate hydrogen bonds.

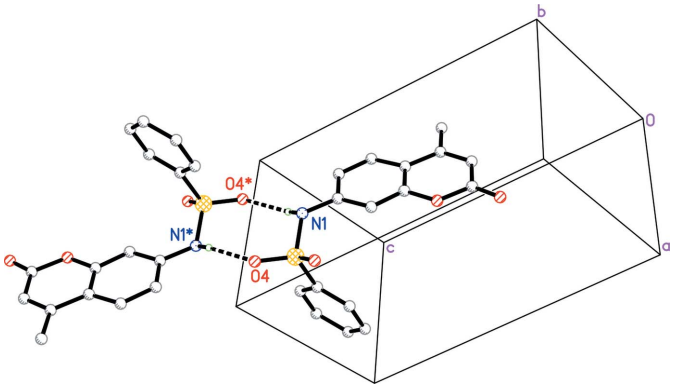


Figure 2
Part of the crystal structure of (I), showing the formation of a dimer of $R_2^2(8)$ ring motif. For clarity, H atoms not involved in the motif shown have been omitted. Dashed lines indicate hydrogen bonds·[Symmetry code: (*) $1-x, 2-y, 2-z$].

These dimers are the basic building block in the formation of three one-dimensional substructures. Atom C9 in the molecule at (x, y, z) acts as hydrogen-bond donor to atom O3 in the molecule at $(-1+x, y, z)$, so forming a centrosymmetric $R_4^4(18)$ ring centred at (0, 1, 1). These dimers are linked by C—H···O hydrogen bonds into a ribbon of $R_2^2(8)R_4^4(18)[R_4^4(20)]$ rings (García-Báez *et al.*, 2002) along the [100] direction (Fig. 3 and Table 1). Atom C12 in the molecule at (x, y, z) acts as hydrogen-bond donor to atom O2 in the molecule at $(2-x, 1-y, 1-z)$; propagation of the C—H···O interaction by inversion, generates a centrosymmetric $R_2^2(22)$ ring centred at $(1, \frac{1}{2}, \frac{1}{2})$, and these dimers are linked into a chain of alternating $R_2^2(8)$ and $R_2^2(22)$ rings along the $[1\bar{1}1]$ direction (Fig. 4). Atom C14 in the molecules at (x, y, z) and $(1-x, 1-y, 2-z)$ act as hydrogen-bond donors to atoms O4 in the molecules at $(x, -1+y, z)$ and $(1-x, 2-y, 2-z)$, respectively, linking adjacent dimers into a complex [010] chain of rings and forming a centrosymmetric $R_4^2(18)$ ring centred at $(\frac{1}{2}, \frac{3}{2}, 1)$ (Bernstein *et al.*, 1995), also linking the dimers into a complex

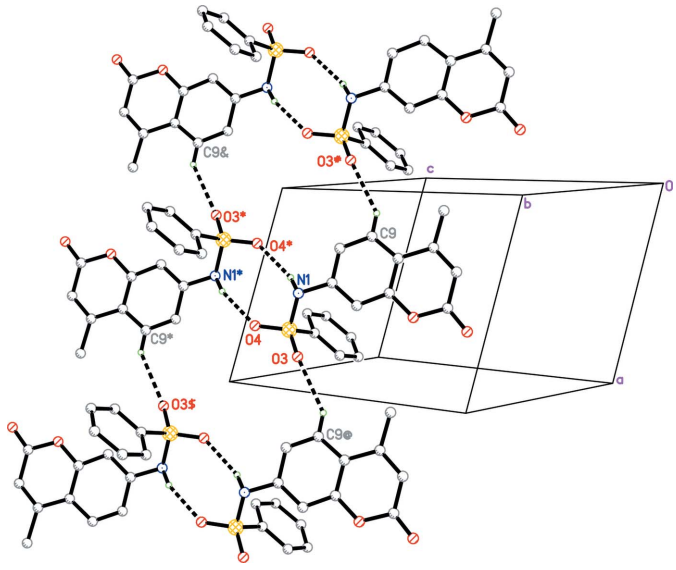


Figure 3
Part of the crystal structure of (I), showing the formation of a ribbon of $R_2^2(8)R_4^4(18)[R_4^4(20)]$ rings along the [100] direction. For clarity, H atoms not involved in the motif shown have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (*) $1-x, 2-y, 2-z$; (#) $-1+x, y, z$; (&) $-x, 2-y, 2-z$; ($) $2-x, 2-y, 2-z$; (@) $1+x, y, z$].

[010] chain of rings (Fig. 5). The combination of the [010] chain, the $[1\bar{1}1]$ chain and the [100] ribbon results in the formation of a three-dimensional network structure.

Experimental

To a solution containing 7-amino-4-methylcoumarin (1.75 g, 10 mmol) and anhydrous pyridine (10 ml), a solution of phenylsulfonyl chloride (11 mmol) was slowly added at 278–283 K with stirring over a period of 30 min. The reaction mixture was then stirred continuously for 12 h at room temperature (298–300 K) and then poured into ice–water (200 ml). The solid obtained was filtered off, washed with water and dried at room temperature. Yellow crystals of (I) suitable for X-ray structure analysis were obtained by evaporation of an ethanol solution of the product over a period of one week (m.p. 518–519 K).

Crystal data

$C_{16}H_{13}NO_4S$
$M_r = 315.33$
Triclinic, $P\bar{1}$
$a = 8.040$ (3) Å
$b = 8.193$ (3) Å
$c = 11.656$ (5) Å
$\alpha = 83.246$ (5)°
$\beta = 85.316$ (5)°
$\gamma = 70.287$ (4)°
$V = 717.0$ (5) Å^3
$Z = 2$
$D_x = 1.461$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.24$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.42 × 0.25 × 0.21 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.905$, $T_{max} = 0.951$
3735 measured reflections
2493 independent reflections
1853 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 25.0°$

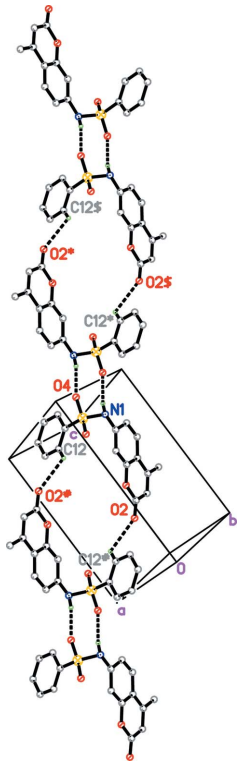


Figure 4
Part of the crystal structure of (I), showing the formation of a chain of alternating $R_2^2(8)$ and $R_2^2(22)$ rings along the [1$\bar{1}$1] direction. For clarity, H atoms not involved in the motif shown have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (*) $1-x, 2-y, 2-z$; (#) $2-x, 1-y, 1-z$; (&) $1+x, -1+y, -1+z$; ($) $-1+x, 1+y, 1+z$; (@) $-x, 3-y, 3-z$].

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.2626P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.124$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.34$ e Å^{-3}
2493 reflections	
200 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3	0.93	2.46	3.099 (4)	126
N1—H1···O4^{i}	0.90	2.07	2.923 (3)	157
C9—H9···O3^{ii}	0.93	2.51	3.287 (3)	141
C12—H12···O2^{iii}	0.93	2.72	3.278 (4)	120
C14—H14···O4^{iv}	0.93	2.57	3.336 (4)	140

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

All H atoms were positioned geometrically and refined as riding on their parent atoms, with C—H = 0.96 Å and U_{iso}(H)= 1.5U_{eq}(C) for methyl H atoms, N—H = 0.90 Å and U_{iso}(H)= 1.2U_{eq}(C) for amino H atoms, and C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

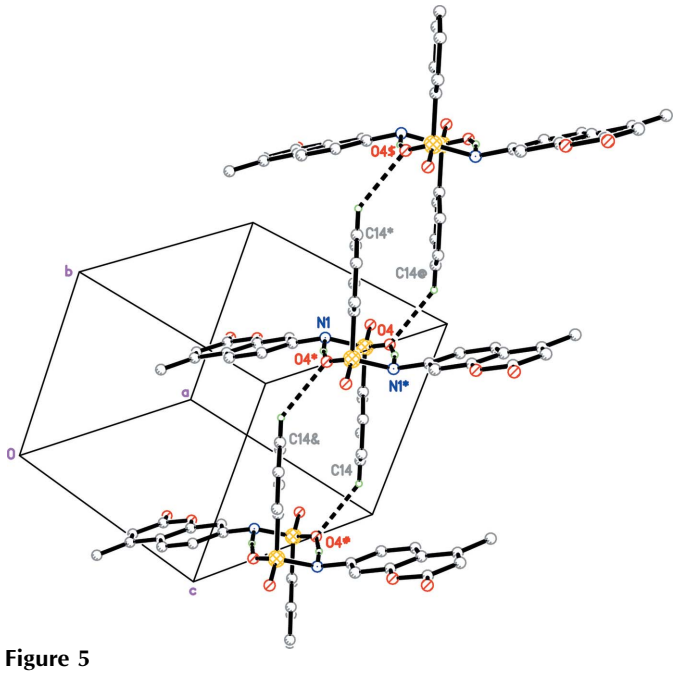


Figure 5
Part of the crystal structure of (I), showing the formation of a chain of hydrogen-bonded dimers along [010]. For clarity, H atoms not involved in the motif shown have been omitted. Dashed lines indicate hydrogen bonds. [Symmetry codes: (*) $1-x, 2-y, 2-z$; (#) $x, -1+y, z$; (&) $1-x, 1-y, 2-z$; ($) $1-x, 3-y, 2-z$; (@) $x, 1+y, z$].

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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