Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-[1-Hydroxy-3-(methylsulfanylmethylsulfinyl)-propan-2-yl]-2-oxo-2*H*-chromene-3-carboxamide

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Received 9 January 2006
Accepted 18 January 2006

In the title compound, $C_{15}H_{17}NO_5S_2$, an analogue of the antibiotic sparsomycin, the chiral S atom is in an *R* configuration and the chiral C atom is in an *S* configuration. Molecules translated by one unit along the *b* axis are linked into chains by intermolecular O—H···O and C—H···O hydrogen bonds. Adjacent screw-related chains are interlinked *via* C—H···O hydrogen-bonding interactions.

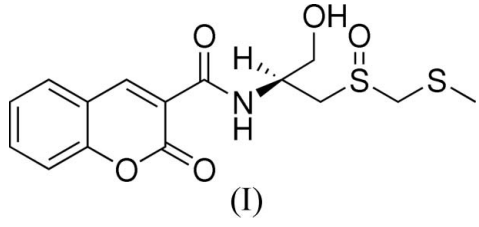
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.029
wR factor = 0.072
Data-to-parameter ratio = 12.7

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

Comment

Sparsomycin, a metabolite of *Streptomyces sparsogenes* or *Streptomyces cuspidosporus* (Argoudelis & Herr, 1962), exhibits broad-spectrum antibiotic activity against a variety of Gram-negative and Gram-positive bacteria, and shows potent antitumour activity (Goldberg, 1974). In the molecule, there are two chiral centres, *viz.* the chiral C atom and the S atom of the sulfoxide group (Ottenheijm *et al.*, 1981). A structure–activity relationship study showed that the configuration of the molecule plays an important role in its biological acitivity (Liskamp & Clostee, 1984; Lin & Dubois, 1977). Here, we report the crystal structure of the title compound, (I), which is an analogue of sparsomycin with high antibacterial activity.



(I)

The molecule of (I) (Fig. 1) contains a coumarin ring system and a monooxodithiaacetal group. The chiral S atom of the sulfoxide group is in an *R* configuration and the chiral C atom is in an *S* configuration. Bond lengths and angles (Table 1) fall into the normal ranges for such organic compounds (Ottenheijm *et al.*, 1981). The dihedral angle between the S1/S2/C11–C14 and N1/O1–O3/C1–C10 planes is 75.43 (5)°.

An intramolecular N1—H1*A*···O2 hydrogen bond is present in the molecular structure of (I) (Table 2.). Molecules translated by one unit along the *b* axis are linked into chains by a combination of O—H···O and C—H···O intermolecular hydrogen bonds (Fig. 2). Adjacent screw-related chains are interlinked through intermolecular C—H···O hydrogen-bonding interactions involving the H atoms attached to atoms C12 and C14 (Fig. 3).

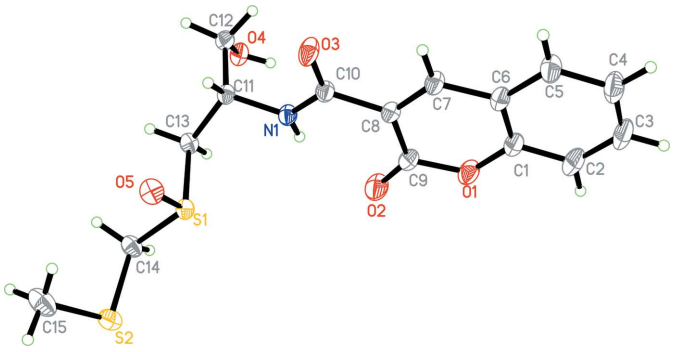


Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

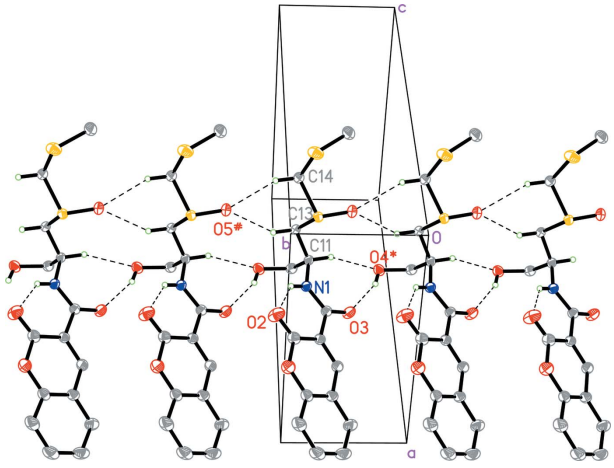


Figure 2
Part of the crystal structure of (I), showing the formation of a chain along [010]. Only H atoms involved in the hydrogen bonding (dashed lines) are shown. Atoms marked with an asterisk (*) or a hash (#) are at the symmetry positions $(x, -1 + y, z)$ and $(x, 1 + y, z)$, respectively.

Experimental

A solution of monooxodithiaacetal amine (0.5 mmol) was added to an *N*,*N*-dimethylformamide (DMF) solution (5 ml) of coumarinic acid (0.55 mmol), *N*,*N*′-dicyclohexylcarbodiimide (0.55 mmol) and 1-hydroxy-1*H*-benzotriazole (0.5 mmol). The reaction mixture was stirred for 24 h at room temperature. Compound (I) was obtained by flash chromatographic purification. Crystals of (I) suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in dichloromethane and methanol (15:1 *v*/*v*) (m.p. 428–429 K). Analysis, found: C 50.42, H 4.50, N 3.81%; $C_{15}H_{17}NO_5S_2$ requires: C 50.69, H 4.82, N 3.94%.

Crystal data

$C_{15}H_{17}NO_5S_2$
M_r = 355.42
Monoclinic, $P2_1$
a = 10.3234 (17) Å
b = 5.2159 (9) Å
c = 15.448 (3) Å
β = 98.341 (2)°
V = 823.0 (3) Å^3
Z = 2
D_x = 1.434 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2819 reflections
θ = 2.6–26.4°
μ = 0.35 mm^{-1}
T = 294 (2) K
Block, colourless
0.38 × 0.22 × 0.18 mm

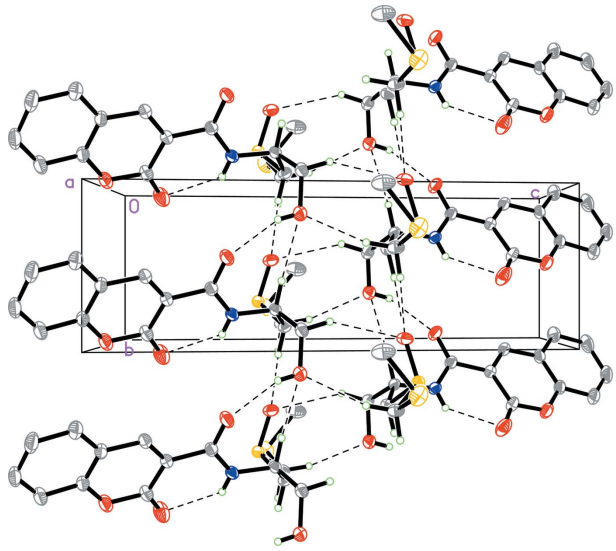


Figure 3
The crystal packing of (I), viewed down the *a* axis. Only H atoms involved in the hydrogen bonding (dashed lines) are shown.

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.816, T_{max} = 0.939
4696 measured reflections
2740 independent reflections
2521 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 26.4°
$h = -10 \rightarrow 12$
$k = -6 \rightarrow 5$
$l = -19 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.072
S = 1.09
2740 reflections
215 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0372P)^2 + 0.0863P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack (1983), with 859 Friedel pairs
Flack parameter: 0.07 (7)

Table 1
Selected geometric parameters (Å, °).

S1—C13	1.805 (2)	S2—C15	1.796 (3)
S1—C14	1.805 (2)	S2—C14	1.797 (2)
O5—S1—C13	106.79 (10)	C13—S1—C14	95.62 (10)
O5—S1—C14	107.42 (10)	C15—S2—C14	100.56 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	0.78 (3)	2.12 (2)	2.719 (2)	134 (2)
O4—H4···O3^{i}	0.77 (3)	2.08 (3)	2.811 (2)	158 (3)
C13—H13A···O5^{i}	0.97	2.40	3.258 (3)	147
C14—H14A···O4^{ii}	0.97	2.51	3.420 (3)	156
C14—H14B···O5^{i}	0.97	2.41	3.291 (3)	151
C11—H11···O4^{iii}	0.98	2.53	3.426 (3)	152

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

Hydroxyl and amino H atoms were located in a difference map and their positional parameters were refined. H atoms attached to C atoms were placed in idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å. U_{iso}(H) values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxyl and methyl H atoms, and $1.2U_{eq}$ for the remaining H atoms. A rotating-group refinement was used for the methyl group.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

The authors thank the National Natural Science Foundation of China (grant No. 20432010) and the Natural Science Foundation of Tianjin (grant No. 013616311) for financial support.

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