Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(1*S**,2*S**)-(+)-2-Amino-1-[4-(methylsulfanyl)-phenyl]propane-1,3-diol

Brian Samas,* Dainius Macikenas and Anthony C Blackburn

Pharmaceutical Sciences, Pfizer Global R&D, 2800 Plymouth Road, Ann Arbor, MI 48105, USA

Correspondence e-mail: brian.samas@pfizer.com

Received 1 July 2005
Accepted 29 July 2005
Online 6 August 2005

Key indicators

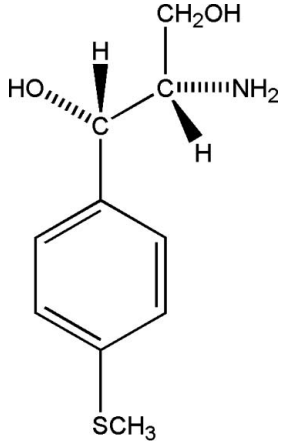
Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.004 Å
R factor = 0.056
wR factor = 0.133
Data-to-parameter ratio = 18.6

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

The title compound, $C_{10}H_{15}NO_2S$, commonly referred to as (1*S**,2*S**)-(+)-thiomicamine, forms molecular sheets parallel to the *a* and *b* axes. Within these sheets, the molecules are connected by strong O—H···O and O—H···N hydrogen bonds. The methylsulfanyl groups protrude from this network and are interwoven between adjacent sheets.

Comment

As part of a study of pharmaceutical counterions, the crystal structure of (1*S**,2*S**)-(+)-thiomicamine, (I), at 299 K was solved and refined. The asymmetric unit consists of one molecule (Fig. 1). Two-dimensional hydrogen-bond networks (Fig. 2) extend along the *a* and *b* axes. The methylsulfanyl groups protrude from these networks and are interwoven between these hydrogen-bonded sheets. H atoms involved in hydrogen bonding are listed in Table 1. Two weak intramolecular hydrogen bonds form five-membered rings: *viz*. N1—H99*A*···O1 and and N1—H99*B*···O2. Although both of these hydrogen-bond angles are necessarily small, both protons approach the O-atom acceptor lone pairs, despite no conformational restriction to do so.



(I)

Experimental

A sample of (I), labeled (1*S*,2*S*)-(+)-thiomicamine, was obtained from Sigma–Aldrich; it was dissolved in acetonitrile at a concentration of approximately 1 mg ml^{-1} and placed in a chamber with an antisolvent (methyl *tert*-butyl ether) to allow for vapor diffusion (all reagents purchased from Sigma–Aldrich). After several days, the sample was removed from the antisolvent chamber and the remaining solvent was allowed to evaporate to dryness. A crystal of (I) was removed and mounted on a glass fiber for data collection.

© 2005 International Union of Crystallography
Printed in Great Britain – all rights reserved

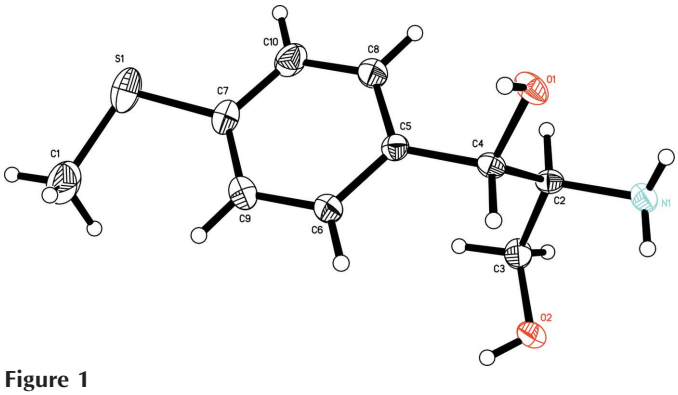


Figure 1
View of the molecule of (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size.

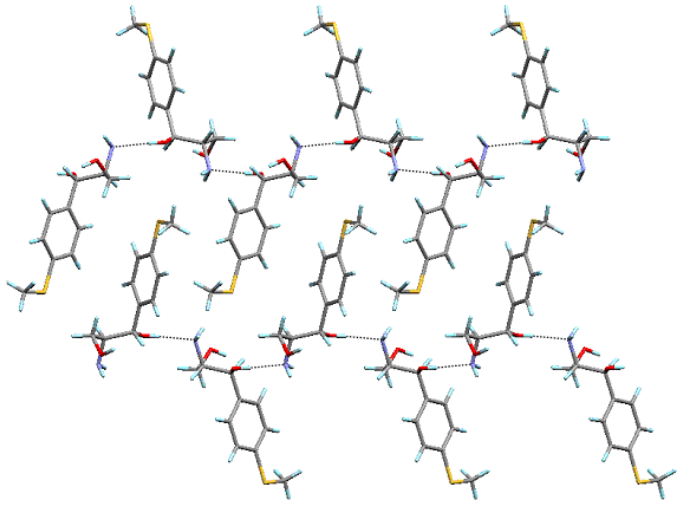


Figure 2
Intermolecular hydrogen bonding (dashed lines) of (I), viewed along the *a* axis, showing the stacking of the two-dimensional networks.

Crystal data

$C_{10}H_{15}NO_2S$
M_r = 213.29
Orthorhombic, $P2_12_12_1$
a = 5.8044 (9) Å
b = 9.5929 (15) Å
c = 20.075 (3) Å
V = 1117.8 (3) Å^3
Z = 4
D_x = 1.267 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3109 reflections
θ = 2.0–28.3°
μ = 0.27 mm^{-1}
T = 299 (2) K
Column, colorless
0.50 × 0.05 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.879, T_{max} = 0.987
18448 measured reflections
2700 independent reflections
2056 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.3°
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.133
S = 1.04
2700 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.3094P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1094 Friedel pairs
Flack parameter: 0.15 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H99A···O1	0.82 (3)	2.50 (3)	2.826 (3)	105 (3)
N1—H99B···O2	0.85 (3)	2.38 (3)	2.768 (3)	108 (2)
O2—H98A···O1^{i}	0.81 (4)	1.88 (4)	2.669 (3)	164 (4)
N1—H99A···O2^{ii}	0.82 (3)	2.49 (3)	3.234 (3)	151 (3)
O1—H98B···N1^{iii}	0.79 (3)	1.92 (3)	2.703 (3)	171 (3)

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

H atoms covalently bonded to heteroatoms were located in a Fourier map and their positions were refined freely with isotropic displacement parameters. The remaining H atoms were placed in idealized positions and refined with riding constraints, with C—distances in the range 0.93–0.98 Å and with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C). The Flack (1983) parameter was 0.15 (14), giving an inconclusive indication of the absolute configuration. This is on the edge of confidence for a refined Flack parameter (Flack & Bernardinelli, 2000), so this determination definitively establishes only the relative configurations of the asymmetric atoms. For this reason, they are labeled with an asterisk. However, the final structure agrees with the absolute stereospecific label used by the supplier.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXTL* (Bruker, 2003).

References

Bruker (2003). *SMART* for WNT/2000 (Version 5.630), *SAINT-Plus* (Version 6.45) and *SHELXTL* (Version 6.14). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* B**58**, 389–397.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Flack, H. D. & Bernardinelli, G. (2000). *J. Appl. Cryst.* **33**, 1143–1148.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2002). *SADABS*. Version 2.02. Bruker AXS Inc., Madison, Wisconsin, USA.