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N-{(2*S*)-3-Hydroxy-4-[(5-methyl-1,3,4-thiadiazol-2-yl)sulfanyl]-1-phenyl-2-butyl}-4-methylbenzenesulfonamide

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Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.016 Å; R factor = 0.078; wR factor = 0.206; data-to-parameter ratio = 8.1.

The thiadiazoyl and sulfonyl-benzene rings in the title compound, $C_{20}H_{23}N_3O_3S_3$, are aligned to the same side of the molecule, forming a twisted 'U' shape [dihedral angle = 77.6 (5)°]. The benzyl-benzene ring is orientated in the opposite direction from the molecule but projects approximately along the same axis as the other rings [dihedral angle between benzene rings = 28.2 (5)°] so that, overall, the molecule has a flattened shape. The hydroxy and amine groups are almost *syn* which enables the formation of intermolecular hydroxy-OH···N(thiadiazoyl) and amine-H···O(sulfonyl) hydrogen bonds leading to a supramolecular chain aligned along the *a* axis.

Related literature

For background to the use of amino alcohols in medicinal chemistry, see: Ferreira *et al.* (2009); de Oliveira *et al.* (2008); Brik & Wong (2003); Ghosh *et al.* (2001); Parikh *et al.* (2005); Andrews *et al.* (2006). For the anti-malarial activity of hydroxyethylpiperazines, see: Cunico, Gomes, Moreth *et al.* (2009). For the biological activity of hydroxyethylsulfonamides, see: Cunico *et al.* (2008, 2011); Cunico, Gomes, Facchinetti *et al.* (2009). For related structures, see: Cunico, Gomes, Harrison *et al.* (2009); Gomes *et al.* (2011).

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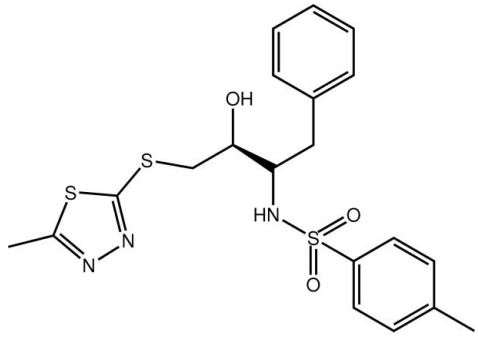

Experimental

Crystal data

$C_{20}H_{23}N_3O_3S_3$
M_r = 449.59
Orthorhombic, $P2_12_12_1$
a = 5.0420 (2) Å
b = 18.4840 (8) Å
c = 22.9650 (8) Å
V = 2140.25 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 120 K
0.14 × 0.02 × 0.02 mm

Data collection

Bruker–Nonius Roper CCD camera on κ-goniostat diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.438, T_{max} = 1.000
12594 measured reflections
2182 independent reflections
1538 reflections with $I > 2\sigma(I)$
R_{int} = 0.144

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.206
S = 1.29
2182 reflections
270 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Absolute structure: nd
Flack parameter: ?
Rogers parameter: ?

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···N1^{i}	0.84 (7)	2.11 (8)	2.860 (11)	148 (8)
N3—H3*N*···O3^{i}	0.88 (2)	2.05 (4)	2.902 (10)	163 (9)

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6366).

References

Andrews, K. T., Fairlie, D. P., Madala, P. K., Ray, J., Wyatt, D. M., Hilton, P. M., Melville, L. A., Beattie, L., Gardioner, D. L., Reid, R. C., Stoermer, M. J., Skinner-Adams, T., Berry, C. & McCarthy, J. S. (2006). *Antimicrob. Agents Chemother.* **50**, 639–648.

Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Brik, A. & Wong, C.-H. (2003). *Org. Biomol. Chem.* **1**, 5–14.

Cunico, W., Ferreira, M. L. G., Ferreira, T. G., Penido, C., Henriques, M. G. M. O., Krettli, L. G., Varotti, F. P. & Krettli, A. U. (2008). *Lett. Drug Des. Discov.* **5**, 178–181.

Cunico, W., Gomes, C. R. B., Facchinetti, V., Moreth, M., Penido, C., Henriques, M. G. M. O., Varotti, F. P., Krettli, L. G., Krettli, A. U., da Silva, F. S., Caffarena, E. R. & de Magalhães, C. S. (2009). *Eur. J. Med. Chem.* **44**, 3816–3820.

Cunico, W., Gomes, C. R. B., Ferreira, M. L. G., Ferreira, T. G., Cardinot, D., de Souza, M. V. N. & Lourenço, M. C. S. (2011). *Eur. J. Med. Chem.* **46**, 974–978.

Cunico, W., Gomes, C. R. B., Harrison, W. T. A., Moreth, M., Wardell, J. L. & Wardell, S. M. S. C. (2009). *Z. Kristallogr.* **224**, 461–470.

Cunico, W., Gomes, C. R. B., Moreth, M., Manhanini, D. P., Figueiredo, I. H., Penido, C., Henriques, M. G. M. O., Varotti, F. P. & Krettli, A. U. (2009). *Eur. J. Med. Chem.* **44**, 1363–1368.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Ferreira, M. L., Vasconcelos, T. R. A., de Carvalho, E. M., Lourenço, M. C. S., Wardell, S. M. S. V., Wardell, J. L., Ferreira, V. F. & de Souza, M. V. N. (2009). *Carbohydr. Res.* **344**, 2042–2047.

Ghosh, A. K., Bilcer, G. & Schiltz, G. (2001). *Synthesis*, pp. 2203–2229.

Gomes, C. R. B., Vasconcelos, T. R. A., Vellasco, W. T., Wardell, J. L., Wardell, S. M. S. V. & Tiekink, E. R. T. (2011). *Acta Cryst.* E**67**, o2313–o2314.

Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.

Oliveira, P. S. M. de, Ferreira, V. F., de Souza, M. V. N. & de Carvalho, E. M. (2008). *Quim. Nova*, **31**, 776–780.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

Parikh, S., Gut, J., Istvan, E., Goldberg, D. E., Havlir, D. V. & Rosenthal, P. J. (2005). *Antimicrob. Agents Chemother.* **49**, 2983–2985.

Sheldrick, G. M. (2007). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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N-{(2*S*)-3-Hydroxy-4-[(5-methyl-1,3,4-thiadiazol-2-yl)sulfanyl]-1-phenyl-2-butyl}-4-methylbenzenesulfonamide

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S1. Comment

Amino alcohols play versatile roles in medicinal chemistry (Ferreira *et al.*, 2009; de Oliveira *et al.*, 2008; Brik & Wong, 2003, Ghosh *et al.*, 2001; Parikh *et al.*, 2005; Andrews *et al.*, 2006). Some of us recently reported the anti-malarial activity of hydroxyethypiperazines against *Plasmodium falciparum* (Cunico, Gomes, Moreth *et al.*, 2009) and of hydroxyethylsulfonamide derivatives against *Plasmodium falciparum* (Cunico *et al.*, 2008; Cunico, Gomes, Facchinetti *et al.*, 2009), and mycobacterium tuberculosis H37Rv (Cunico *et al.*, 2011). In conjunction with these biological studies, crystal structure determinations of (2*R*,4*S*)-4-(arylmethyl)-1-(4-phenyl-3-amino-2-hydroxybutyl)-piperazine derivatives (Cunico, Gomes, Harrison *et al.*, 2009) and an example of a pyrimidyl derivative (Gomes *et al.*, 2011) have been carried out. In continuation of these structural studies, we now report the synthesis, Fig. 1, and structure of the title compound, (I).

In (I), Fig. 2, the thiadiazoyl and sulfonyl-benzene rings are orientated to the same side of the molecule but are not aligned in a parallel fashion as seen in the dihedral angle of 77.6 (5) ° formed between the rings. The benzyl-benzene is directed away from the rest of the twisted U-shaped molecule and forms a dihedral angle of 28.2 (5) ° with the sulfonyl-benzene ring. The key stereochemical feature of the molecule is the almost *syn* alignment of the hydroxyl and amine groups. This has an important consequence in the crystal packing.

As seen from Fig. 3, molecules assemble into a supramolecular chain *via* hydroxyl-OH···*N*(thiadiazoyl) and amine-*H*···*O*(sulfonyl) hydrogen bonds, Table 1. The chains are aligned along the *a* axis and assemble in the crystal structure without any specific interactions between them, Fig. 4.

S2. Experimental

Referring to Fig. 1, trifluoroacetic acid (1.5 ml, 20 mmol) was added to a solution of 2 (2 mmol), prepared from 1 and 5-methyl-1,3,4-thiadiazole-2(3*H*)-thione, in CH_2Cl_2 (6 ml). The mixture was stirred for 6 h, rotary evaporated to leave a residue, which was dissolved in EtOAc (20 ml), successively washed with 5% $NaHCO_3$ aqueous solution, water and brine, and dried over $MgSO_4$. The solvent was removed to afford the corresponding free amine, which was dissolved in EtOAc (10 mL) to which were added triethylamine (2.2 mmol) and *N*,*N*-dimethylformamide (0.2 mmol). The system was stirred for 30 minutes under nitrogen and *p*-toluenenesulfonyl chloride (2.0 mmol) was slowly added. The mixture was stirred for 8 h, successively washed with 5% HCl aqueous solution, water and brine, and dried over $MgSO_4$. The solvent was removed in high vacuum and the title product 3 was obtained in 68% yield after recrystallization from hexane. The crystals used in the structure determination were grown from EtOH solution. *M*.pt: 412–414 K. EI—MS (m/*z*) (%): 472.1 (*M*++Na, 80%). ^{1}H NMR [400.00 MHz, $CDCl_3$] δ: 7.42 (d, 2H, J = 8.4 Hz, $PhSO_2$); 7.11 (d, 2H, J = 8.0 Hz, $PhSO_2$);

7.07–6.99 (m, 5H, Ph); 3.84–3.82 (m, 1H, H2); 3.58 (dd, 1H, ^{1}J = 13.6 Hz, ^{2}J = 4.4 Hz; H1b); 3.61–3.55 (m, 1H, H3); 3.22 (dd, 1H, ^{1}J = 13.6 Hz, ^{2}J = 8.4 Hz, H1a); 2.92 (dd, 1H, ^{1}J = 14.0 Hz, ^{2}J = 4.4 Hz, H4b); 2.72 (s, 3H, CH_3(Het)); 2.59 (dd, ^{1}H, J = 14.0 Hz, ^{2}J = 8.4 Hz, H4a); 2.35 (s, 3H; CH_3) p.p.m.. ^{13}C NMR [100.0 MHz, $CDCl_3$] δ: 168.6; 167.9; 144.1; 139.9; 139.2; 130.7; 129.4; 127.8; 127.3; 115.2; 73.4; 60.6; 39.4; 37.1; 21.6; 15.4 p.p.m. IR (cm^{-1}; KBr pellets): ν_{max}: 3273 (OH); 3023 (NH); 1330, 1161 (O═S═O); 686 (C—S).

S3. Refinement

The C-bound H atoms were geometrically placed (C–H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C). The O– and N-bound H atoms were located from a difference map and refined with the distance restraints O–H = 0.84 ± 0.01 and N–H = 0.88±0.01 Å, and with U_{iso}(H) = zU_{eq}(carrier atom); z = 1.5 for O and z = 1.2 for N. The refinement of the Flack absolute sturcture was ambiguous [refined value = 0.24 (19)] and 1482 Friedel pairs were averaged in the final refinement and the absolute configuration was assigned on the basis of the chirality of the *L*-serine starting material. The small (0.02 *x* 0.02 *x* 0.14 mm) needle was weakly diffracting but it was not deemed necessary to secure a data set with synchrotron radiation. The poor nature of the sample is also reflected in the relatively high values of R_{int} and in the residuals. However, the structure has been determined unambiguously.

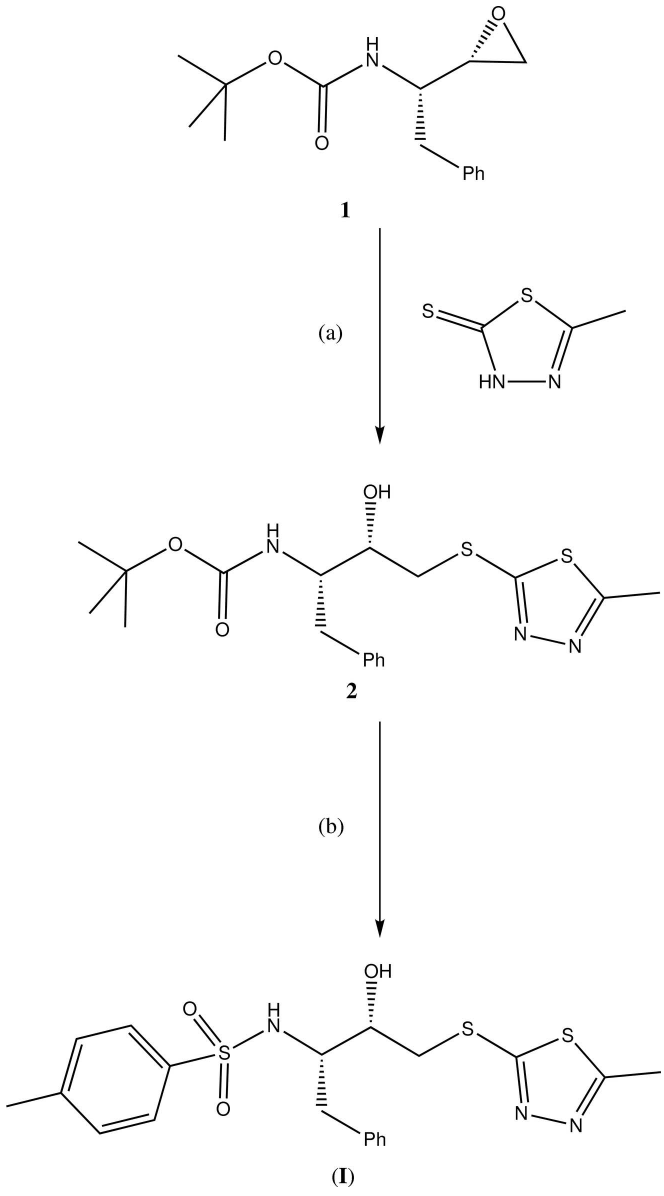

Figure 1

Synthesis. Reagents: (*a*) TFA/CH_2Cl_2 (1/3), RT, 6 h; (*b*) *p*-$MeC_6H_4SO_2Cl$, Et_3N, DMF, AcOEt, RT, 8 h.

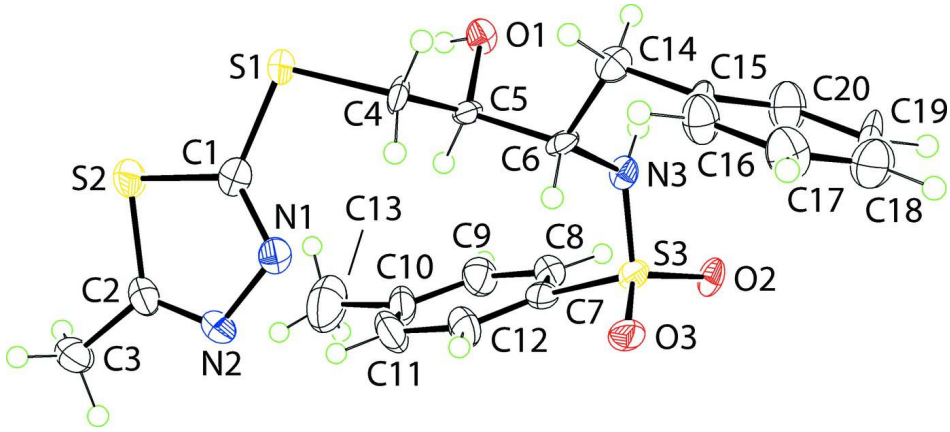

Figure 2

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

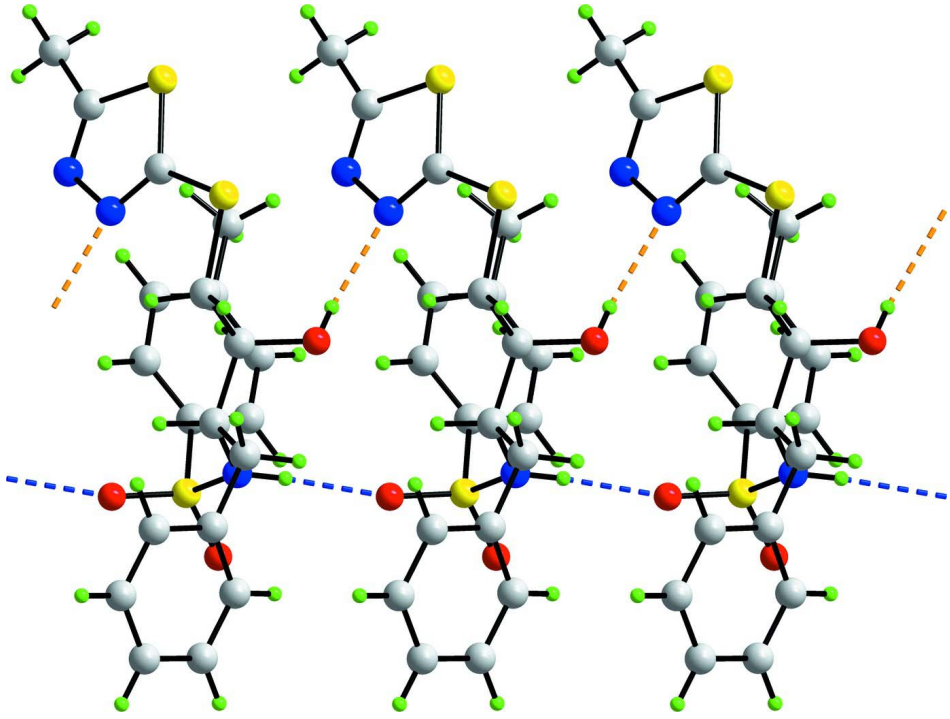

Figure 3

A view of the supramolecular chain along the *a* axis in (I). The O—H···N and N—H···O hydrogen bonding are shown as orange and blue dashed lines, respectively.

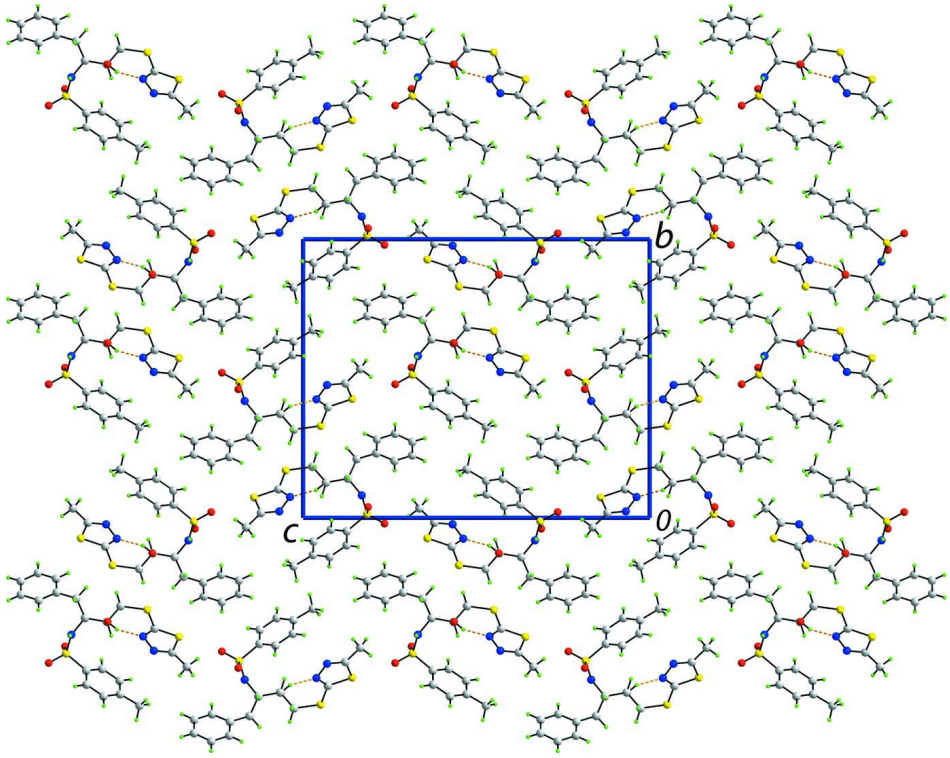

Figure 4

A view in projection down the *a* axis of the packing of supramolecular chains in (I). The The O—H···N hydrogen bonding is shown as orange dashed lines.

N-{(2*S*)-3-Hydroxy-4-[(5-methyl-1,3,4-thiadiazol-2-yl)sulfanyl]- 1-phenyl-2-butyl}-4-methylbenzenesulfonamide

Crystal data

$C_{20}H_{23}N_3O_3S_3$
$M_r = 449.59$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.0420$ (2) Å
$b = 18.4840$ (8) Å
$c = 22.9650$ (8) Å
$V = 2140.25$ (15) Å^3
$Z = 4$
$F(000) = 944$
$D_x = 1.395$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20956 reflections
$\theta = 2.9$–27.5°
$\mu = 0.37$ mm^{-1}
$T = 120$ K
Needle, colourless
0.14 × 0.02 × 0.02 mm

Data collection

Bruker–Nonius Roper CCD camera on κ-goniostat diffractometer
Radiation source: Bruker–Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ & ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
$T_{min} = 0.438$, $T_{max} = 1.000$
12594 measured reflections
2182 independent reflections
1538 reflections with $I > 2\sigma(I)$
$R_{int} = 0.144$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.4°$
$h = -4 \rightarrow 5$
$k = -20 \rightarrow 21$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.206$
$S = 1.29$
2182 reflections
270 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}
Absolute structure: nd

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.1474 (5)	0.17021 (13)	0.05038 (10)	0.0243 (6)
S2	0.3692 (5)	0.07316 (15)	0.14153 (11)	0.0324 (7)
S3	0.2827 (4)	0.01335 (13)	−0.18450 (11)	0.0222 (6)
O1	−0.1857 (12)	0.1270 (3)	−0.0645 (3)	0.0230 (15)
H1O	−0.23 (2)	0.097 (4)	−0.039 (3)	0.034*
O2	0.1680 (13)	−0.0208 (3)	−0.2346 (3)	0.0246 (15)
O3	0.5507 (12)	0.0390 (4)	−0.1872 (3)	0.0302 (17)
N1	0.5551 (15)	0.0748 (5)	0.0380 (4)	0.027 (2)
N2	0.7075 (16)	0.0234 (4)	0.0689 (4)	0.030 (2)
N3	0.0990 (15)	0.0809 (4)	−0.1688 (3)	0.0205 (18)
H3N	−0.074 (3)	0.078 (5)	−0.173 (4)	0.025*
C1	0.3774 (19)	0.1027 (5)	0.0713 (4)	0.023 (2)
C2	0.632 (2)	0.0174 (5)	0.1215 (4)	0.029 (2)
C3	0.757 (2)	−0.0339 (6)	0.1642 (5)	0.036 (3)
H3A	0.6187	−0.0634	0.1825	0.054*
H3B	0.8512	−0.0063	0.1943	0.054*
H3C	0.8827	−0.0655	0.1438	0.054*
C4	0.215 (2)	0.1833 (5)	−0.0267 (4)	0.024 (2)
H4A	0.1495	0.2316	−0.0384	0.028*
H4B	0.4099	0.1829	−0.0326	0.028*
C5	0.0909 (18)	0.1267 (5)	−0.0661 (4)	0.019 (2)
H5	0.1553	0.0778	−0.0539	0.023*
C6	0.1811 (18)	0.1406 (5)	−0.1295 (4)	0.020 (2)
H6	0.3791	0.1436	−0.1299	0.024*

C7	0.264 (2)	−0.0480 (5)	−0.1259 (4)	0.025 (2)
C8	0.058 (2)	−0.0988 (6)	−0.1256 (5)	0.032 (3)
H8	−0.0527	−0.1045	−0.1587	0.039*
C9	0.020 (2)	−0.1406 (6)	−0.0763 (5)	0.038 (3)
H9	−0.1215	−0.1746	−0.0750	0.046*
C10	0.189 (3)	−0.1329 (6)	−0.0285 (5)	0.043 (3)
C11	0.395 (3)	−0.0845 (6)	−0.0296 (5)	0.044 (3)
H11	0.5110	−0.0797	0.0029	0.052*
C12	0.431 (2)	−0.0421 (6)	−0.0799 (5)	0.033 (3)
H12	0.5749	−0.0090	−0.0816	0.040*
C13	0.135 (4)	−0.1739 (7)	0.0276 (6)	0.073 (5)
H13A	0.2824	−0.1666	0.0546	0.110*
H13B	0.1159	−0.2255	0.0190	0.110*
H13C	−0.0296	−0.1559	0.0452	0.110*
C14	0.071 (2)	0.2108 (5)	−0.1548 (5)	0.025 (2)
H14A	−0.1230	0.2056	−0.1598	0.031*
H14B	0.1017	0.2504	−0.1265	0.031*
C15	0.190 (2)	0.2317 (5)	−0.2115 (4)	0.022 (2)
C16	0.399 (2)	0.2813 (6)	−0.2132 (5)	0.034 (3)
H16	0.4615	0.3017	−0.1777	0.041*
C17	0.519 (2)	0.3018 (6)	−0.2654 (5)	0.038 (3)
H17	0.6593	0.3359	−0.2655	0.046*
C18	0.433 (2)	0.2729 (7)	−0.3151 (6)	0.045 (3)
H18	0.5146	0.2859	−0.3509	0.054*
C19	0.227 (3)	0.2243 (6)	−0.3145 (5)	0.047 (3)
H19	0.1661	0.2042	−0.3501	0.056*
C20	0.108 (3)	0.2043 (6)	−0.2634 (5)	0.039 (3)
H20	−0.0348	0.1707	−0.2642	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0275 (13)	0.0300 (13)	0.0153 (13)	0.0022 (11)	−0.0008 (10)	−0.0003 (10)
S2	0.0342 (15)	0.0430 (16)	0.0201 (14)	0.0050 (13)	0.0017 (11)	0.0089 (11)
S3	0.0178 (12)	0.0274 (14)	0.0213 (13)	−0.0001 (10)	0.0010 (9)	−0.0063 (10)
O1	0.019 (3)	0.029 (4)	0.021 (4)	−0.005 (3)	0.002 (3)	0.006 (3)
O2	0.027 (4)	0.032 (4)	0.015 (3)	−0.010 (3)	0.000 (3)	−0.008 (3)
O3	0.017 (3)	0.045 (4)	0.029 (4)	0.002 (3)	0.001 (3)	−0.006 (3)
N1	0.014 (4)	0.044 (5)	0.022 (5)	−0.007 (4)	0.004 (3)	0.004 (4)
N2	0.027 (5)	0.035 (5)	0.028 (5)	0.009 (4)	−0.004 (4)	0.004 (4)
N3	0.017 (4)	0.026 (4)	0.018 (4)	−0.007 (3)	−0.003 (3)	−0.005 (3)
C1	0.018 (5)	0.027 (5)	0.024 (6)	−0.013 (4)	0.002 (4)	−0.003 (4)
C2	0.033 (6)	0.031 (6)	0.024 (6)	0.001 (5)	−0.008 (5)	0.003 (5)
C3	0.032 (6)	0.041 (6)	0.035 (7)	0.009 (5)	−0.002 (5)	0.007 (5)
C4	0.036 (6)	0.026 (5)	0.009 (5)	0.000 (5)	0.001 (4)	−0.003 (4)
C5	0.022 (5)	0.018 (5)	0.018 (5)	0.008 (4)	0.008 (4)	0.004 (4)
C6	0.018 (5)	0.021 (5)	0.022 (5)	0.002 (4)	0.002 (4)	−0.012 (4)
C7	0.028 (5)	0.020 (5)	0.026 (6)	0.009 (4)	0.002 (4)	−0.001 (4)

C8	0.029 (6)	0.043 (7)	0.026 (6)	0.006 (5)	−0.002 (5)	0.002 (5)
C9	0.051 (7)	0.030 (6)	0.034 (8)	0.006 (5)	−0.003 (5)	0.003 (5)
C10	0.069 (9)	0.039 (6)	0.021 (7)	0.024 (7)	−0.011 (6)	−0.004 (5)
C11	0.054 (8)	0.042 (7)	0.035 (7)	0.015 (6)	−0.021 (6)	−0.001 (5)
C12	0.038 (6)	0.032 (6)	0.029 (7)	−0.001 (5)	−0.012 (5)	−0.003 (5)
C13	0.123 (14)	0.057 (9)	0.040 (9)	0.016 (10)	0.001 (9)	0.005 (7)
C14	0.030 (6)	0.019 (5)	0.027 (6)	−0.002 (4)	0.004 (4)	−0.002 (4)
C15	0.030 (6)	0.025 (5)	0.011 (5)	0.008 (4)	−0.001 (4)	−0.004 (4)
C16	0.033 (6)	0.042 (7)	0.027 (6)	−0.010 (5)	−0.003 (5)	0.006 (5)
C17	0.040 (7)	0.036 (7)	0.038 (8)	−0.011 (5)	−0.003 (5)	0.009 (6)
C18	0.048 (7)	0.050 (8)	0.036 (8)	0.003 (6)	0.012 (6)	0.012 (6)
C19	0.089 (10)	0.041 (7)	0.010 (6)	0.002 (7)	0.007 (6)	0.001 (5)
C20	0.048 (7)	0.039 (6)	0.030 (7)	−0.009 (5)	0.001 (6)	0.009 (5)

Geometric parameters (Å, º)

S1—C1	1.770 (10)	C7—C8	1.397 (14)
S1—C4	1.818 (9)	C8—C9	1.384 (16)
S2—C1	1.704 (10)	C8—H8	0.9500
S2—C2	1.742 (11)	C9—C10	1.397 (16)
S3—O3	1.433 (7)	C9—H9	0.9500
S3—O2	1.435 (6)	C10—C11	1.374 (18)
S3—N3	1.596 (8)	C10—C13	1.518 (18)
S3—C7	1.763 (10)	C11—C12	1.407 (16)
O1—C5	1.395 (11)	C11—H11	0.9500
O1—H1O	0.838 (11)	C12—H12	0.9500
N1—C1	1.285 (12)	C13—H13A	0.9800
N1—N2	1.414 (11)	C13—H13B	0.9800
N2—C2	1.271 (13)	C13—H13C	0.9800
N3—C6	1.485 (11)	C14—C15	1.484 (14)
N3—H3N	0.878 (11)	C14—H14A	0.9900
C2—C3	1.504 (14)	C14—H14B	0.9900
C3—H3A	0.9800	C15—C20	1.361 (15)
C3—H3B	0.9800	C15—C16	1.398 (14)
C3—H3C	0.9800	C16—C17	1.395 (16)
C4—C5	1.520 (13)	C16—H16	0.9500
C4—H4A	0.9900	C17—C18	1.332 (17)
C4—H4B	0.9900	C17—H17	0.9500
C5—C6	1.546 (13)	C18—C19	1.374 (17)
C5—H5	1.0000	C18—H18	0.9500
C6—C14	1.526 (13)	C19—C20	1.369 (16)
C6—H6	1.0000	C19—H19	0.9500
C7—C12	1.357 (15)	C20—H20	0.9500
C1—S1—C4	103.5 (5)	C8—C7—S3	118.4 (8)
C1—S2—C2	85.4 (5)	C9—C8—C7	118.9 (11)
O3—S3—O2	119.4 (4)	C9—C8—H8	120.6
O3—S3—N3	107.4 (4)	C7—C8—H8	120.6

O2—S3—N3	107.0 (4)	C8—C9—C10	120.0 (12)
O3—S3—C7	107.3 (5)	C8—C9—H9	120.0
O2—S3—C7	107.9 (4)	C10—C9—H9	120.0
N3—S3—C7	107.4 (4)	C11—C10—C9	120.9 (11)
C5—O1—H1O	105 (8)	C11—C10—C13	118.5 (12)
C1—N1—N2	110.5 (8)	C9—C10—C13	120.4 (14)
C2—N2—N1	111.9 (8)	C10—C11—C12	118.3 (10)
C6—N3—S3	123.9 (6)	C10—C11—H11	120.8
C6—N3—H3N	112 (6)	C12—C11—H11	120.8
S3—N3—H3N	121 (6)	C7—C12—C11	121.0 (10)
N1—C1—S2	116.9 (8)	C7—C12—H12	119.5
N1—C1—S1	125.3 (8)	C11—C12—H12	119.5
S2—C1—S1	117.8 (6)	C10—C13—H13A	109.5
N2—C2—C3	123.4 (10)	C10—C13—H13B	109.5
N2—C2—S2	115.3 (7)	H13A—C13—H13B	109.5
C3—C2—S2	121.3 (8)	C10—C13—H13C	109.5
C2—C3—H3A	109.5	H13A—C13—H13C	109.5
C2—C3—H3B	109.5	H13B—C13—H13C	109.5
H3A—C3—H3B	109.5	C15—C14—C6	114.1 (8)
C2—C3—H3C	109.5	C15—C14—H14A	108.7
H3A—C3—H3C	109.5	C6—C14—H14A	108.7
H3B—C3—H3C	109.5	C15—C14—H14B	108.7
C5—C4—S1	114.2 (7)	C6—C14—H14B	108.7
C5—C4—H4A	108.7	H14A—C14—H14B	107.6
S1—C4—H4A	108.7	C20—C15—C16	116.7 (9)
C5—C4—H4B	108.7	C20—C15—C14	123.3 (9)
S1—C4—H4B	108.7	C16—C15—C14	120.0 (9)
H4A—C4—H4B	107.6	C17—C16—C15	121.9 (10)
O1—C5—C4	113.2 (8)	C17—C16—H16	119.1
O1—C5—C6	108.6 (8)	C15—C16—H16	119.1
C4—C5—C6	109.0 (7)	C18—C17—C16	119.3 (11)
O1—C5—H5	108.7	C18—C17—H17	120.4
C4—C5—H5	108.7	C16—C17—H17	120.4
C6—C5—H5	108.7	C17—C18—C19	119.8 (12)
N3—C6—C14	107.5 (8)	C17—C18—H18	120.1
N3—C6—C5	111.4 (8)	C19—C18—H18	120.1
C14—C6—C5	113.1 (7)	C20—C19—C18	121.2 (12)
N3—C6—H6	108.2	C20—C19—H19	119.4
C14—C6—H6	108.2	C18—C19—H19	119.4
C5—C6—H6	108.2	C15—C20—C19	121.1 (11)
C12—C7—C8	120.7 (10)	C15—C20—H20	119.4
C12—C7—S3	120.7 (8)	C19—C20—H20	119.4

C1—N1—N2—C2	−0.1 (12)	O3—S3—C7—C8	−158.1 (8)
O3—S3—N3—C6	−35.0 (9)	O2—S3—C7—C8	−28.3 (9)
O2—S3—N3—C6	−164.3 (7)	N3—S3—C7—C8	86.7 (8)
C7—S3—N3—C6	80.1 (8)	C12—C7—C8—C9	3.3 (15)
N2—N1—C1—S2	0.5 (10)	S3—C7—C8—C9	−171.4 (8)

N2—N1—C1—S1	179.0 (6)	C7—C8—C9—C10	−1.5 (16)
C2—S2—C1—N1	−0.6 (8)	C8—C9—C10—C11	−0.5 (18)
C2—S2—C1—S1	−179.2 (6)	C8—C9—C10—C13	174.9 (11)
C4—S1—C1—N1	3.4 (9)	C9—C10—C11—C12	0.7 (17)
C4—S1—C1—S2	−178.2 (5)	C13—C10—C11—C12	−174.8 (11)
N1—N2—C2—C3	179.3 (9)	C8—C7—C12—C11	−3.1 (16)
N1—N2—C2—S2	−0.3 (11)	S3—C7—C12—C11	171.4 (8)
C1—S2—C2—N2	0.5 (8)	C10—C11—C12—C7	1.1 (17)
C1—S2—C2—C3	−179.1 (9)	N3—C6—C14—C15	−66.3 (10)
C1—S1—C4—C5	80.3 (8)	C5—C6—C14—C15	170.2 (8)
S1—C4—C5—O1	63.4 (9)	C6—C14—C15—C20	82.7 (12)
S1—C4—C5—C6	−175.7 (6)	C6—C14—C15—C16	−96.6 (11)
S3—N3—C6—C14	141.8 (7)	C20—C15—C16—C17	−0.1 (16)
S3—N3—C6—C5	−93.7 (9)	C14—C15—C16—C17	179.2 (10)
O1—C5—C6—N3	−64.8 (9)	C15—C16—C17—C18	−0.6 (17)
C4—C5—C6—N3	171.5 (7)	C16—C17—C18—C19	0.9 (18)
O1—C5—C6—C14	56.5 (10)	C17—C18—C19—C20	−0.5 (19)
C4—C5—C6—C14	−67.3 (10)	C16—C15—C20—C19	0.5 (16)
O3—S3—C7—C12	27.3 (10)	C14—C15—C20—C19	−178.8 (11)
O2—S3—C7—C12	157.1 (8)	C18—C19—C20—C15	−0.2 (19)
N3—S3—C7—C12	−87.9 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···N1^{i}	0.84 (7)	2.11 (8)	2.860 (11)	148 (8)
N3—H3*N*···O3^{i}	0.88 (2)	2.05 (4)	2.902 (10)	163 (9)

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