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# *cis*-*N*-(2-Hydroxycyclohexyl)-*p*-toluenesulfonamide

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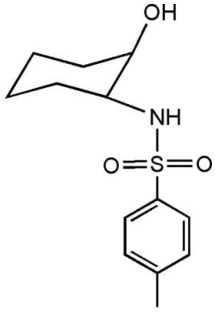
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.036; $wR$ factor = 0.111; data-to-parameter ratio = 18.7.

There are two symmetry-independent molecules in the asymmetric unit of the title compound, $C_{13}H_{19}NO_3S$. The cyclohexane rings in the two molecules adopt chair configurations. The hydroxy and amino groups on the cyclohexane ring assume axial and equatorial orientations, respectively, with respect to the plane of the ring. The crystal structure is stabilized by two intermolecular N—H···O and O—H···O hydrogen bonds from the two symmetry-independent molecules.

## Related literature

For related structures of $\beta$-amino alcohols, see: Bergmeier (2000); Krzemiński & Wojtczak (2005). For related structures of tosylamino compounds, see: Coote *et al.* (2008); Liu *et al.* (2005); Chinnakali *et al.* (2007); Nan & Xing (2006). For the synthesis of the title compound, see: Naiker *et al.* (2008).




## Experimental

### *Crystal data*

$C_{13}H_{19}NO_3S$
$M_r$ = 269.35
Triclinic, $P\overline{1}$
$a$ = 6.3031 (1) Å
$b$ = 12.8355 (2) Å
$c$ = 17.5367 (3) Å
$\alpha$ = 106.645 (1)°
$\beta$ = 93.971 (1)°
$\gamma$ = 100.047 (1)°
$V$ = 1327.75 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.24 mm$^{-1}$
$T$ = 173 K
0.51 × 0.31 × 0.25 mm

### *Data collection*

Bruker APEXII CCD diffractometer
18458 measured reflections
6423 independent reflections
4837 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.111
$S$ = 1.07
6423 reflections
343 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1*N*···O6$^{i}$ | 0.83 (2) | 2.00 (2) | 2.8255 (17) | 175.0 (18) |
| N2—H2*N*···O3$^{ii}$ | 0.82 (2) | 2.00 (2) | 2.8155 (18) | 173.1 (19) |
| O3—H3*O*···O5$^{iii}$ | 0.83 (2) | 1.93 (2) | 2.7489 (15) | 171 (2) |
| O6—H6*O*···O2$^{iv}$ | 0.83 (2) | 1.98 (2) | 2.8001 (15) | 169 (2) |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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, 1998); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

We wish to thank Dr Manuel Fernandes (University of the Witwatersrand) for the data collection, and the NRF, THRIP and the University of KwaZulu-Natal for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2131).

## References

Bergmeier, S. (2000). *Tetrahedron*, **56**, 2561–2576.
Brandenburg, K. (1998). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chinnakali, K., Poornachandran, M., Raghunathan, R. & Fun, H.-K. (2007). *Acta Cryst.* E**63**, o1030–o1031.
Coote, S. C., O'Brien, P. & Whitwood, A. C. (2008). *Org. Biomol. Chem.* **6**, 4299–4314.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Krzemiński, M. P. & Wojtczak, A. (2005). *Tetrahedron Lett.* **46**, 8299–8302.
Liu, Z., Fan, Y., Li, R., Zhou, B. & Wu, L. (2005). *Tetrahedron Lett.* **46**, 1023–1025.
Naiker, T., Datye, A. & Friedrich, H. B. (2008). *Appl. Catal. A*, **350**, 96–102.
Nan, Z.-H. & Xing, J.-D. (2006). *Acta Cryst.* E**62**, o1978–o1979.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supplementary materials

*Acta Cryst.* (2010). E**66**, o463 [ **doi:10.1107/S1600536810002151** ]

# *cis*-*N*-(2-Hydroxycyclohexyl)-*p*-toluenesulfonamide

**M. I. Fadlalla, H. B. Friedrich, G. E. M. Maguire and M. D. Bala**

## Comment

Molecules containing a β-amino alcohol system have been used as precursors for the synthesis of chiral ligands, aziridine and biologically active compounds (Bergmeier, 2000; Krzemiński & Wojtczak, 2005). As a part of study on this family of compounds, we report the crystal structure of the title compound (l) (Fig. 1).

The geometry of the benzenesulfonamide unit in (I) agrees with that for related structures (Chinnakali *et al.* 2007; Nan & Xing, 2006). The cyclohexane rings in the two molecules adopt the chair configuration. The hydroxy and amino groups on the cyclohexane ring respectively assume axial and equatorial orientations with respect to the plane of the ring. The crystal packing (Fig. 2) is stabilized by intermolecular N—H···O and O—H···O hydrogen bonds from the two neighbouring symmetry-independent molecules (Table 1).

## Experimental

The synthesis of the title compound was carried out using a modified literature method (Naiker *et al.* 2008) using a catalytic process. To a nitrogen saturated Schlenk tube, toluene (6 ml), water (172 µl) chloroamine-T (0.21 g, 0.956 mmol), cyclohexene (0.478 mmol) and catalyst (0.03 g) were added in that order. After the complete conversion of the starting material the catalyst was gravity filtered. The reaction mixture was washed with 15 ml of sodium sulfite (1 g in 15 ml of de-ionized water), followed by 15 ml of ethyl acetate. Then the aqueous layer was separated from the organic layer and washed further with 3 × 15 ml of ethyl acetate. The solvent was removed *in vacuo*, and the crude product was purified using preparative high pressure liquid chromatography to yield the title compound as a white solid. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in acetonitrile/water (1:1 v/v) at room temperature. (mp; 414–416 K) Spectroscopic analysis: $^{13}$C NMR (400 MHz, $CDCl_3$, δ, p.p.m): = 19.76 (s, 1 C), 21.54 (s, 2 C), 27.98 (s, 1 C), 31.46(s, 1 C), 55.10 (s, 1 C), 68.76 (s, 1 C), 126.97 (s, 2 C), 129.74 (s, 2 C), 137.98(s, 1 C), 143.39 (s, 143.39).. MS m/*z* –[fragment]–(%): 291.8 ($M$ + $Na^+$) calculated = 291.8 for $C_{13}H_{19}NO_3SNa^+$.

FT–IR ($cm^{-1}$): = 3414(*m*), (OH), 3137(*m*), (NH), 2938(w), 2849(w), 1598(*m*), (ar), 1059(*m*), (S=O).

## Refinement

All H-atoms were refined using a riding model, with C—H = 0.93 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for aromatic, C—H = 0.97 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for $CH_2$, C—H = 0.96 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C) for $CH_3$, N—H = 0.86 Å and $U_{iso}$(H) = 1.2$U_{eq}$(N) for NH, and O—H = 0.82 Å and $U_{iso}$(H) = 1.5$U_{eq}$(O) for OH.

## Figures

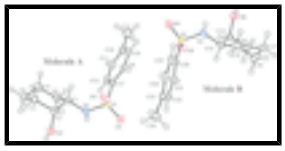


Fig. 1. The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50 % probability level. H atoms are presented as a small spheres of arbitrary radius.

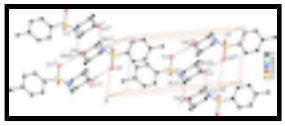


Fig. 2. N—H···O and O—H···O hydrogen bonding interactions (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) *x* - 1, *y* - 1, *z*; (ii) *x* + 1, *y* + 1, *z*; (iii) *x*, *y* - 1, *z*; (iv) *x*, *y* + 1, *z*; (v) *x* + 1, *y* + 1, *z*; (vi) *x* - 1, *y* - 1, *z*; (vii) *x*, *y* + 1, *z*; (viii) *x*, *y* - 1, *z*.]

## *cis*-*N*-(2-Hydroxycyclohexyl)-*p*-toluenesulfonamide cis-2-Tosylaminocyclohexanol

*Crystal data*

| | |
|---|---|
| $C_{13}H_{19}NO_3S$ | $Z = 4$ |
| $M_r = 269.35$ | $F(000) = 576$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.347$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Melting point = 414–416 K |
| $a = 6.3031$ (1) Å | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $b = 12.8355$ (2) Å | Cell parameters from 6946 reflections |
| $c = 17.5367$ (3) Å | $\theta = 2.4$–$28.3°$ |
| $\alpha = 106.645$ (1)° | $\mu = 0.24$ mm$^{-1}$ |
| $\beta = 93.971$ (1)° | $T = 173$ K |
| $\gamma = 100.047$ (1)° | Block, colourless |
| $V = 1327.75$ (4) Å$^3$ | 0.51 × 0.31 × 0.25 mm |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 4837 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.033$ |
| graphite | $\theta_{max} = 28.0°$, $\theta_{min} = 1.2°$ |
| φ and ω scans | $h = -8 \rightarrow 8$ |
| 18458 measured reflections | $k = -16 \rightarrow 16$ |
| 6423 independent reflections | $l = -23 \rightarrow 23$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.036$ | Hydrogen site location: difference Fourier map |
| $wR(F^2) = 0.111$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.07$ | $w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.0739P]$ where $P = (F_o^2 + 2F_c^2)/3$ |

| | |
|---|---|
| 6423 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 343 parameters | $\Delta\rho_{max} = 0.40$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.41$ e Å$^{-3}$ |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3149 (2) | 0.39725 (12) | 0.35318 (8) | 0.0230 (3) |
| H1 | 0.4155 | 0.4675 | 0.3860 | 0.028* |
| C2 | 0.4327 (2) | 0.30139 (12) | 0.34741 (9) | 0.0227 (3) |
| H2 | 0.5706 | 0.3156 | 0.3236 | 0.027* |
| C3 | 0.4842 (3) | 0.28788 (14) | 0.42986 (9) | 0.0303 (4) |
| H3A | 0.5896 | 0.3543 | 0.4639 | 0.036* |
| H3B | 0.5522 | 0.2225 | 0.4240 | 0.036* |
| C4 | 0.2795 (3) | 0.27272 (15) | 0.47095 (10) | 0.0344 (4) |
| H4A | 0.1800 | 0.2022 | 0.4399 | 0.041* |
| H4B | 0.3195 | 0.2684 | 0.5254 | 0.041* |
| C5 | 0.1644 (3) | 0.36911 (14) | 0.47692 (9) | 0.0337 (4) |
| H5A | 0.2583 | 0.4384 | 0.5127 | 0.040* |
| H5B | 0.0285 | 0.3557 | 0.5009 | 0.040* |
| C6 | 0.1107 (3) | 0.38351 (13) | 0.39456 (9) | 0.0266 (3) |
| H6A | 0.0034 | 0.3178 | 0.3607 | 0.032* |
| H6B | 0.0449 | 0.4497 | 0.4010 | 0.032* |
| C7 | 0.3445 (2) | 0.63056 (12) | 0.29981 (8) | 0.0212 (3) |
| C8 | 0.5061 (2) | 0.72537 (12) | 0.31511 (9) | 0.0238 (3) |
| H8 | 0.6421 | 0.7204 | 0.2956 | 0.029* |
| C9 | 0.4672 (3) | 0.82718 (12) | 0.35906 (9) | 0.0282 (3) |
| H9 | 0.5775 | 0.8920 | 0.3693 | 0.034* |
| C10 | 0.2692 (3) | 0.83612 (13) | 0.38840 (9) | 0.0290 (4) |
| C11 | 0.1103 (3) | 0.74045 (14) | 0.37164 (10) | 0.0310 (4) |
| H11 | −0.0257 | 0.7454 | 0.3911 | 0.037* |
| C12 | 0.1439 (3) | 0.63787 (13) | 0.32735 (9) | 0.0284 (3) |
| H12 | 0.0320 | 0.5735 | 0.3159 | 0.034* |
| C13 | 0.2270 (4) | 0.94682 (16) | 0.43611 (11) | 0.0459 (5) |
| H13A | 0.0900 | 0.9582 | 0.4130 | 0.069* |
| H13B | 0.3460 | 1.0061 | 0.4345 | 0.069* |

| | | | | |
|---|---|---|---|---|
| H13C | 0.2173 | 0.9480 | 0.4919 | 0.069* |
| N1 | 0.2596 (2) | 0.40722 (10) | 0.27298 (8) | 0.0243 (3) |
| O1 | 0.30664 (19) | 0.48034 (9) | 0.15979 (6) | 0.0319 (3) |
| O2 | 0.62179 (17) | 0.50634 (9) | 0.26023 (7) | 0.0322 (3) |
| O3 | 0.29042 (18) | 0.20373 (9) | 0.29512 (6) | 0.0262 (2) |
| S1 | 0.39239 (6) | 0.50145 (3) | 0.24179 (2) | 0.02302 (10) |
| H1N | 0.134 (3) | 0.3817 (15) | 0.2496 (11) | 0.039 (5)* |
| H3O | 0.364 (3) | 0.1556 (18) | 0.2813 (12) | 0.050 (6)* |
| C14 | 0.7984 (3) | 1.13220 (12) | 0.14293 (9) | 0.0260 (3) |
| H14 | 0.6960 | 1.0621 | 0.1109 | 0.031* |
| C15 | 0.6821 (2) | 1.22875 (12) | 0.14932 (8) | 0.0221 (3) |
| H15 | 0.5435 | 1.2142 | 0.1726 | 0.026* |
| C16 | 0.6321 (3) | 1.24306 (13) | 0.06696 (9) | 0.0275 (3) |
| H16A | 0.5649 | 1.3087 | 0.0729 | 0.033* |
| H16B | 0.5267 | 1.1769 | 0.0325 | 0.033* |
| C17 | 0.8384 (3) | 1.25814 (14) | 0.02681 (9) | 0.0311 (4) |
| H17A | 0.8013 | 1.2657 | −0.0269 | 0.037* |
| H17B | 0.9405 | 1.3268 | 0.0594 | 0.037* |
| C18 | 0.9460 (3) | 1.15881 (15) | 0.01851 (10) | 0.0382 (4) |
| H18A | 1.0803 | 1.1699 | −0.0068 | 0.046* |
| H18B | 0.8468 | 1.0909 | −0.0167 | 0.046* |
| C19 | 1.0017 (3) | 1.14389 (14) | 0.10043 (10) | 0.0322 (4) |
| H19A | 1.1115 | 1.2088 | 0.1339 | 0.039* |
| H19B | 1.0647 | 1.0768 | 0.0934 | 0.039* |
| C20 | 0.7614 (2) | 0.89890 (11) | 0.20252 (8) | 0.0214 (3) |
| C21 | 0.5911 (3) | 0.82342 (12) | 0.15054 (9) | 0.0253 (3) |
| H21 | 0.4570 | 0.8445 | 0.1409 | 0.030* |
| C22 | 0.6186 (3) | 0.71642 (13) | 0.11252 (9) | 0.0290 (3) |
| H22 | 0.5021 | 0.6643 | 0.0769 | 0.035* |
| C23 | 0.8145 (3) | 0.68457 (12) | 0.12593 (9) | 0.0276 (3) |
| C24 | 0.9834 (3) | 0.76236 (13) | 0.17751 (9) | 0.0276 (3) |
| H24 | 1.1182 | 0.7418 | 0.1867 | 0.033* |
| C25 | 0.9594 (3) | 0.86956 (13) | 0.21587 (9) | 0.0260 (3) |
| H25 | 1.0767 | 0.9221 | 0.2508 | 0.031* |
| C26 | 0.8384 (3) | 0.56743 (14) | 0.08535 (11) | 0.0414 (4) |
| H26A | 0.7230 | 0.5151 | 0.0976 | 0.062* |
| H26B | 0.9802 | 0.5569 | 0.1048 | 0.062* |
| H26C | 0.8275 | 0.5541 | 0.0272 | 0.062* |
| N2 | 0.8567 (2) | 1.12308 (11) | 0.22299 (8) | 0.0285 (3) |
| O4 | 0.81390 (19) | 1.05591 (9) | 0.33850 (6) | 0.0326 (3) |
| O5 | 0.49416 (18) | 1.02921 (9) | 0.23866 (7) | 0.0345 (3) |
| O6 | 0.82296 (18) | 1.32622 (9) | 0.20231 (6) | 0.0246 (2) |
| S2 | 0.72262 (6) | 1.03181 (3) | 0.25663 (2) | 0.02444 (11) |
| H2N | 0.981 (3) | 1.1520 (16) | 0.2453 (11) | 0.040 (6)* |
| H6O | 0.751 (4) | 1.3754 (19) | 0.2141 (13) | 0.061 (7)* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0245 (8) | 0.0189 (7) | 0.0237 (7) | 0.0036 (6) | −0.0012 (6) | 0.0050 (6) |
| C2 | 0.0177 (7) | 0.0233 (7) | 0.0270 (8) | 0.0037 (6) | 0.0014 (6) | 0.0080 (6) |
| C3 | 0.0286 (9) | 0.0345 (9) | 0.0307 (8) | 0.0111 (7) | −0.0005 (7) | 0.0128 (7) |
| C4 | 0.0389 (10) | 0.0442 (10) | 0.0277 (8) | 0.0158 (8) | 0.0068 (7) | 0.0177 (7) |
| C5 | 0.0375 (10) | 0.0382 (9) | 0.0257 (8) | 0.0127 (8) | 0.0057 (7) | 0.0068 (7) |
| C6 | 0.0293 (9) | 0.0256 (8) | 0.0260 (8) | 0.0117 (7) | 0.0049 (6) | 0.0054 (6) |
| C7 | 0.0221 (8) | 0.0212 (7) | 0.0237 (7) | 0.0071 (6) | 0.0046 (6) | 0.0099 (6) |
| C8 | 0.0237 (8) | 0.0256 (8) | 0.0240 (7) | 0.0051 (6) | 0.0049 (6) | 0.0099 (6) |
| C9 | 0.0337 (9) | 0.0227 (8) | 0.0275 (8) | 0.0039 (7) | 0.0026 (7) | 0.0080 (6) |
| C10 | 0.0394 (10) | 0.0282 (8) | 0.0226 (8) | 0.0152 (7) | 0.0027 (7) | 0.0080 (6) |
| C11 | 0.0286 (9) | 0.0383 (9) | 0.0344 (9) | 0.0170 (7) | 0.0114 (7) | 0.0164 (7) |
| C12 | 0.0235 (8) | 0.0280 (8) | 0.0382 (9) | 0.0074 (6) | 0.0069 (7) | 0.0153 (7) |
| C13 | 0.0564 (13) | 0.0389 (10) | 0.0413 (11) | 0.0240 (9) | 0.0051 (9) | 0.0021 (8) |
| N1 | 0.0210 (7) | 0.0224 (6) | 0.0285 (7) | −0.0005 (5) | −0.0031 (5) | 0.0106 (5) |
| O1 | 0.0401 (7) | 0.0300 (6) | 0.0254 (6) | 0.0060 (5) | 0.0061 (5) | 0.0087 (5) |
| O2 | 0.0210 (6) | 0.0244 (6) | 0.0502 (7) | 0.0075 (5) | 0.0079 (5) | 0.0073 (5) |
| O3 | 0.0234 (6) | 0.0204 (5) | 0.0322 (6) | 0.0070 (5) | 0.0023 (5) | 0.0026 (4) |
| S1 | 0.0216 (2) | 0.01999 (19) | 0.0284 (2) | 0.00532 (14) | 0.00508 (15) | 0.00776 (15) |
| C14 | 0.0291 (8) | 0.0184 (7) | 0.0274 (8) | 0.0021 (6) | −0.0020 (6) | 0.0051 (6) |
| C15 | 0.0173 (7) | 0.0232 (7) | 0.0245 (7) | 0.0023 (6) | 0.0009 (6) | 0.0069 (6) |
| C16 | 0.0258 (8) | 0.0306 (8) | 0.0256 (8) | 0.0063 (7) | −0.0013 (6) | 0.0084 (6) |
| C17 | 0.0329 (9) | 0.0383 (9) | 0.0236 (8) | 0.0084 (7) | 0.0055 (6) | 0.0106 (7) |
| C18 | 0.0422 (11) | 0.0415 (10) | 0.0282 (9) | 0.0140 (8) | 0.0095 (8) | 0.0022 (7) |
| C19 | 0.0356 (10) | 0.0278 (8) | 0.0343 (9) | 0.0167 (7) | 0.0075 (7) | 0.0045 (7) |
| C20 | 0.0232 (8) | 0.0187 (7) | 0.0243 (7) | 0.0055 (6) | 0.0056 (6) | 0.0085 (6) |
| C21 | 0.0230 (8) | 0.0252 (8) | 0.0289 (8) | 0.0071 (6) | 0.0026 (6) | 0.0090 (6) |
| C22 | 0.0328 (9) | 0.0240 (8) | 0.0274 (8) | 0.0046 (7) | 0.0011 (7) | 0.0048 (6) |
| C23 | 0.0380 (9) | 0.0250 (8) | 0.0261 (8) | 0.0130 (7) | 0.0150 (7) | 0.0112 (6) |
| C24 | 0.0256 (8) | 0.0324 (8) | 0.0333 (8) | 0.0140 (7) | 0.0107 (6) | 0.0167 (7) |
| C25 | 0.0219 (8) | 0.0279 (8) | 0.0302 (8) | 0.0050 (6) | 0.0033 (6) | 0.0119 (6) |
| C26 | 0.0559 (13) | 0.0276 (9) | 0.0450 (11) | 0.0177 (8) | 0.0194 (9) | 0.0092 (8) |
| N2 | 0.0258 (8) | 0.0223 (7) | 0.0366 (8) | −0.0015 (6) | −0.0052 (6) | 0.0133 (6) |
| O4 | 0.0412 (7) | 0.0254 (6) | 0.0288 (6) | 0.0038 (5) | 0.0057 (5) | 0.0059 (5) |
| O5 | 0.0235 (6) | 0.0230 (6) | 0.0539 (8) | 0.0081 (5) | 0.0060 (5) | 0.0047 (5) |
| O6 | 0.0234 (6) | 0.0209 (5) | 0.0268 (6) | 0.0066 (5) | 0.0010 (4) | 0.0023 (4) |
| S2 | 0.0242 (2) | 0.01775 (18) | 0.0308 (2) | 0.00465 (15) | 0.00403 (15) | 0.00618 (15) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.472 (2) | C14—N2 | 1.4690 (19) |
| C1—C2 | 1.528 (2) | C14—C15 | 1.528 (2) |
| C1—C6 | 1.529 (2) | C14—C19 | 1.532 (2) |
| C1—H1 | 1.0000 | C14—H14 | 1.0000 |
| C2—O3 | 1.4325 (17) | C15—O6 | 1.4316 (17) |
| C2—C3 | 1.525 (2) | C15—C16 | 1.526 (2) |

| | | | |
|---|---|---|---|
| C2—H2 | 1.0000 | C15—H15 | 1.0000 |
| C3—C4 | 1.531 (2) | C16—C17 | 1.530 (2) |
| C3—H3A | 0.9900 | C16—H16A | 0.9900 |
| C3—H3B | 0.9900 | C16—H16B | 0.9900 |
| C4—C5 | 1.522 (2) | C17—C18 | 1.522 (2) |
| C4—H4A | 0.9900 | C17—H17A | 0.9900 |
| C4—H4B | 0.9900 | C17—H17B | 0.9900 |
| C5—C6 | 1.530 (2) | C18—C19 | 1.528 (2) |
| C5—H5A | 0.9900 | C18—H18A | 0.9900 |
| C5—H5B | 0.9900 | C18—H18B | 0.9900 |
| C6—H6A | 0.9900 | C19—H19A | 0.9900 |
| C6—H6B | 0.9900 | C19—H19B | 0.9900 |
| C7—C8 | 1.390 (2) | C20—C21 | 1.385 (2) |
| C7—C12 | 1.393 (2) | C20—C25 | 1.389 (2) |
| C7—S1 | 1.7674 (14) | C20—S2 | 1.7665 (14) |
| C8—C9 | 1.387 (2) | C21—C22 | 1.391 (2) |
| C8—H8 | 0.9500 | C21—H21 | 0.9500 |
| C9—C10 | 1.393 (2) | C22—C23 | 1.392 (2) |
| C9—H9 | 0.9500 | C22—H22 | 0.9500 |
| C10—C11 | 1.386 (2) | C23—C24 | 1.388 (2) |
| C10—C13 | 1.507 (2) | C23—C26 | 1.507 (2) |
| C11—C12 | 1.384 (2) | C24—C25 | 1.387 (2) |
| C11—H11 | 0.9500 | C24—H24 | 0.9500 |
| C12—H12 | 0.9500 | C25—H25 | 0.9500 |
| C13—H13A | 0.9800 | C26—H26A | 0.9800 |
| C13—H13B | 0.9800 | C26—H26B | 0.9800 |
| C13—H13C | 0.9800 | C26—H26C | 0.9800 |
| N1—S1 | 1.5975 (13) | N2—S2 | 1.5982 (13) |
| N1—H1N | 0.83 (2) | N2—H2N | 0.82 (2) |
| O1—S1 | 1.4322 (11) | O4—S2 | 1.4343 (12) |
| O2—S1 | 1.4461 (11) | O5—S2 | 1.4452 (12) |
| O3—H3O | 0.83 (2) | O6—H6O | 0.83 (2) |
| N1—C1—C2 | 110.49 (11) | N2—C14—C15 | 110.28 (12) |
| N1—C1—C6 | 110.35 (12) | N2—C14—C19 | 109.93 (13) |
| C2—C1—C6 | 111.65 (12) | C15—C14—C19 | 112.15 (12) |
| N1—C1—H1 | 108.1 | N2—C14—H14 | 108.1 |
| C2—C1—H1 | 108.1 | C15—C14—H14 | 108.1 |
| C6—C1—H1 | 108.1 | C19—C14—H14 | 108.1 |
| O3—C2—C3 | 110.68 (12) | O6—C15—C16 | 110.75 (12) |
| O3—C2—C1 | 106.45 (11) | O6—C15—C14 | 107.06 (11) |
| C3—C2—C1 | 110.94 (12) | C16—C15—C14 | 110.71 (12) |
| O3—C2—H2 | 109.6 | O6—C15—H15 | 109.4 |
| C3—C2—H2 | 109.6 | C16—C15—H15 | 109.4 |
| C1—C2—H2 | 109.6 | C14—C15—H15 | 109.4 |
| C2—C3—C4 | 111.59 (13) | C15—C16—C17 | 111.19 (13) |
| C2—C3—H3A | 109.3 | C15—C16—H16A | 109.4 |
| C4—C3—H3A | 109.3 | C17—C16—H16A | 109.4 |
| C2—C3—H3B | 109.3 | C15—C16—H16B | 109.4 |
| C4—C3—H3B | 109.3 | C17—C16—H16B | 109.4 |

| | | | |
|---|---|---|---|
| H3A—C3—H3B | 108.0 | H16A—C16—H16B | 108.0 |
| C5—C4—C3 | 110.62 (14) | C18—C17—C16 | 110.11 (14) |
| C5—C4—H4A | 109.5 | C18—C17—H17A | 109.6 |
| C3—C4—H4A | 109.5 | C16—C17—H17A | 109.6 |
| C5—C4—H4B | 109.5 | C18—C17—H17B | 109.6 |
| C3—C4—H4B | 109.5 | C16—C17—H17B | 109.6 |
| H4A—C4—H4B | 108.1 | H17A—C17—H17B | 108.2 |
| C4—C5—C6 | 111.54 (13) | C17—C18—C19 | 110.83 (13) |
| C4—C5—H5A | 109.3 | C17—C18—H18A | 109.5 |
| C6—C5—H5A | 109.3 | C19—C18—H18A | 109.5 |
| C4—C5—H5B | 109.3 | C17—C18—H18B | 109.5 |
| C6—C5—H5B | 109.3 | C19—C18—H18B | 109.5 |
| H5A—C5—H5B | 108.0 | H18A—C18—H18B | 108.1 |
| C1—C6—C5 | 110.98 (13) | C18—C19—C14 | 110.71 (14) |
| C1—C6—H6A | 109.4 | C18—C19—H19A | 109.5 |
| C5—C6—H6A | 109.4 | C14—C19—H19A | 109.5 |
| C1—C6—H6B | 109.4 | C18—C19—H19B | 109.5 |
| C5—C6—H6B | 109.4 | C14—C19—H19B | 109.5 |
| H6A—C6—H6B | 108.0 | H19A—C19—H19B | 108.1 |
| C8—C7—C12 | 120.34 (14) | C21—C20—C25 | 120.81 (14) |
| C8—C7—S1 | 119.33 (11) | C21—C20—S2 | 119.75 (11) |
| C12—C7—S1 | 120.29 (11) | C25—C20—S2 | 119.36 (11) |
| C9—C8—C7 | 119.46 (14) | C20—C21—C22 | 119.29 (14) |
| C9—C8—H8 | 120.3 | C20—C21—H21 | 120.4 |
| C7—C8—H8 | 120.3 | C22—C21—H21 | 120.4 |
| C8—C9—C10 | 121.16 (15) | C21—C22—C23 | 120.93 (15) |
| C8—C9—H9 | 119.4 | C21—C22—H22 | 119.5 |
| C10—C9—H9 | 119.4 | C23—C22—H22 | 119.5 |
| C11—C10—C9 | 118.17 (14) | C24—C23—C22 | 118.55 (14) |
| C11—C10—C13 | 120.69 (16) | C24—C23—C26 | 121.60 (15) |
| C9—C10—C13 | 121.13 (16) | C22—C23—C26 | 119.84 (16) |
| C12—C11—C10 | 121.93 (15) | C25—C24—C23 | 121.41 (14) |
| C12—C11—H11 | 119.0 | C25—C24—H24 | 119.3 |
| C10—C11—H11 | 119.0 | C23—C24—H24 | 119.3 |
| C11—C12—C7 | 118.92 (15) | C24—C25—C20 | 119.00 (14) |
| C11—C12—H12 | 120.5 | C24—C25—H25 | 120.5 |
| C7—C12—H12 | 120.5 | C20—C25—H25 | 120.5 |
| C10—C13—H13A | 109.5 | C23—C26—H26A | 109.5 |
| C10—C13—H13B | 109.5 | C23—C26—H26B | 109.5 |
| H13A—C13—H13B | 109.5 | H26A—C26—H26B | 109.5 |
| C10—C13—H13C | 109.5 | C23—C26—H26C | 109.5 |
| H13A—C13—H13C | 109.5 | H26A—C26—H26C | 109.5 |
| H13B—C13—H13C | 109.5 | H26B—C26—H26C | 109.5 |
| C1—N1—S1 | 122.50 (10) | C14—N2—S2 | 122.76 (11) |
| C1—N1—H1N | 119.7 (13) | C14—N2—H2N | 117.8 (13) |
| S1—N1—H1N | 113.8 (13) | S2—N2—H2N | 116.7 (13) |
| C2—O3—H3O | 107.3 (15) | C15—O6—H6O | 107.3 (16) |
| O1—S1—O2 | 118.31 (7) | O4—S2—O5 | 119.25 (7) |
| O1—S1—N1 | 107.26 (7) | O4—S2—N2 | 106.51 (7) |

| | | | |
|---|---|---|---|
| O2—S1—N1 | 108.38 (7) | O5—S2—N2 | 107.95 (7) |
| O1—S1—C7 | 109.32 (7) | O4—S2—C20 | 108.22 (7) |
| O2—S1—C7 | 105.55 (7) | O5—S2—C20 | 105.34 (7) |
| N1—S1—C7 | 107.60 (7) | N2—S2—C20 | 109.34 (7) |
| N1—C1—C2—O3 | −57.65 (14) | N2—C14—C15—O6 | 56.00 (15) |
| C6—C1—C2—O3 | 65.57 (15) | C19—C14—C15—O6 | −66.87 (15) |
| N1—C1—C2—C3 | −178.14 (12) | N2—C14—C15—C16 | 176.81 (12) |
| C6—C1—C2—C3 | −54.92 (16) | C19—C14—C15—C16 | 53.94 (16) |
| O3—C2—C3—C4 | −62.41 (17) | O6—C15—C16—C17 | 62.91 (16) |
| C1—C2—C3—C4 | 55.53 (17) | C14—C15—C16—C17 | −55.68 (16) |
| C2—C3—C4—C5 | −56.05 (19) | C15—C16—C17—C18 | 58.13 (18) |
| C3—C4—C5—C6 | 55.88 (19) | C16—C17—C18—C19 | −58.33 (19) |
| N1—C1—C6—C5 | 178.09 (12) | C17—C18—C19—C14 | 56.40 (19) |
| C2—C1—C6—C5 | 54.80 (16) | N2—C14—C19—C18 | −177.42 (13) |
| C4—C5—C6—C1 | −55.44 (18) | C15—C14—C19—C18 | −54.35 (17) |
| C12—C7—C8—C9 | 0.9 (2) | C25—C20—C21—C22 | 1.2 (2) |
| S1—C7—C8—C9 | 178.53 (11) | S2—C20—C21—C22 | −175.50 (11) |
| C7—C8—C9—C10 | 0.2 (2) | C20—C21—C22—C23 | −0.1 (2) |
| C8—C9—C10—C11 | −0.8 (2) | C21—C22—C23—C24 | −0.7 (2) |
| C8—C9—C10—C13 | −179.95 (15) | C21—C22—C23—C26 | 178.69 (14) |
| C9—C10—C11—C12 | 0.2 (2) | C22—C23—C24—C25 | 0.6 (2) |
| C13—C10—C11—C12 | 179.39 (15) | C26—C23—C24—C25 | −178.82 (14) |
| C10—C11—C12—C7 | 0.9 (2) | C23—C24—C25—C20 | 0.4 (2) |
| C8—C7—C12—C11 | −1.5 (2) | C21—C20—C25—C24 | −1.3 (2) |
| S1—C7—C12—C11 | −179.05 (12) | S2—C20—C25—C24 | 175.38 (11) |
| C2—C1—N1—S1 | −103.58 (13) | C15—C14—N2—S2 | 102.29 (14) |
| C6—C1—N1—S1 | 132.45 (12) | C19—C14—N2—S2 | −133.55 (12) |
| C1—N1—S1—O1 | 175.08 (11) | C14—N2—S2—O4 | −174.30 (12) |
| C1—N1—S1—O2 | 46.28 (13) | C14—N2—S2—O5 | −45.13 (14) |
| C1—N1—S1—C7 | −67.40 (13) | C14—N2—S2—C20 | 68.98 (14) |
| C8—C7—S1—O1 | −95.15 (13) | C21—C20—S2—O4 | 134.13 (12) |
| C12—C7—S1—O1 | 82.45 (13) | C25—C20—S2—O4 | −42.57 (13) |
| C8—C7—S1—O2 | 33.11 (14) | C21—C20—S2—O5 | 5.55 (14) |
| C12—C7—S1—O2 | −149.29 (12) | C25—C20—S2—O5 | −171.15 (11) |
| C8—C7—S1—N1 | 148.68 (12) | C21—C20—S2—N2 | −110.23 (12) |
| C12—C7—S1—N1 | −33.72 (14) | C25—C20—S2—N2 | 73.07 (13) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1N···O6$^{i}$ | 0.83 (2) | 2.00 (2) | 2.8255 (17) | 175.0 (18) |
| N2—H2N···O3$^{ii}$ | 0.82 (2) | 2.00 (2) | 2.8155 (18) | 173.1 (19) |
| O3—H3O···O5$^{iii}$ | 0.83 (2) | 1.93 (2) | 2.7489 (15) | 171 (2) |
| O6—H6O···O2$^{iv}$ | 0.83 (2) | 1.98 (2) | 2.8001 (15) | 169 (2) |

Symmetry codes: (i) *x*−1, *y*−1, *z*; (ii) *x*+1, *y*+1, *z*; (iii) *x*, *y*−1, *z*; (iv) *x*, *y*+1, *z*.

**Fig. 1**

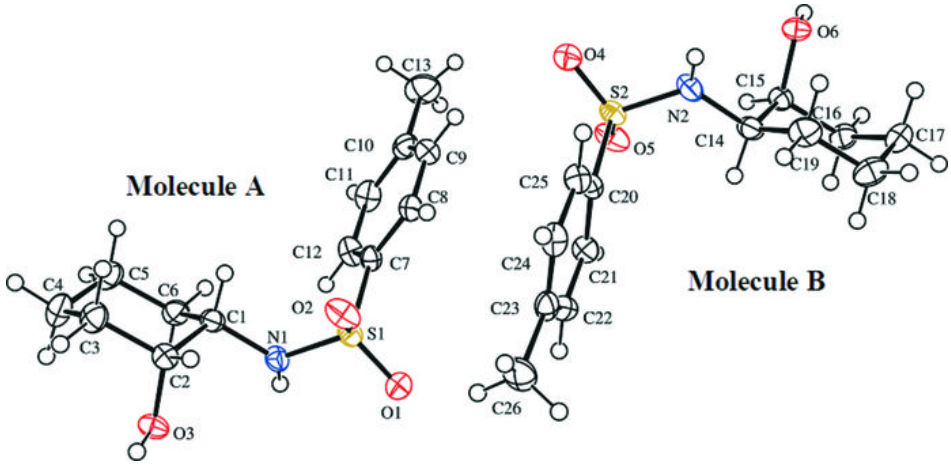
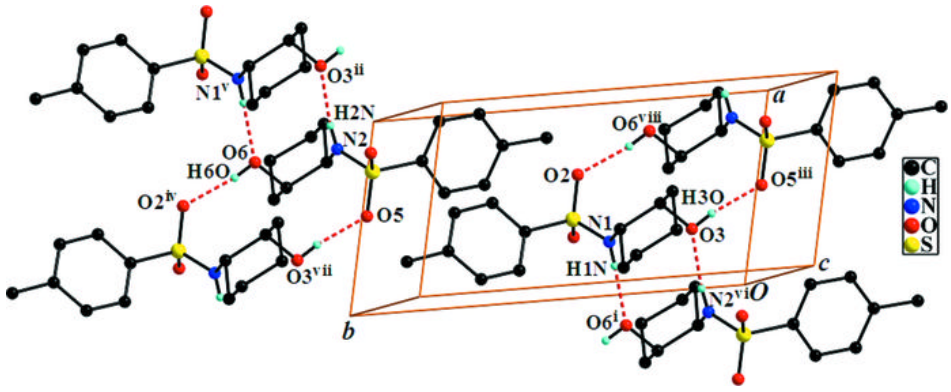

Molecule A
Molecule B
C13
C10
C9
C11
C8
C12
C7
O2
S1
N1
C4
C5
C6
C1
C3
C2
O3
O1
O4
S2
N2
O6
C15
C16
C17
C14
C19
C18
O5
C25
C20
C24
C21
C23
C22
C26

**Fig. 2**


O3ii
N1v
H2N
N2
O6
H6O
O2iv
O5
O3vii
O6viii
a
O2
O5iii
H3O
N1
O3
H1N
c
N2vi
O6i
b
C
H
N
O
S