Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# *rac*-2-Hydroxy-2,8-dimethyl-4-morpholinoethyl-1-thia-4-azaspiro[4.5]decan-3-one

**Mehmet Akkurt,[a]* Şerife Pınar Yalçın,[a] Nalan Terzioğlu Klip[b] and Orhan Büyükgüngör[c]**

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, İstanbul University, Beyazıt 34116, Istanbul, Turkey, and [c]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey
Correspondence e-mail: akkurt@erciyes.edu.tr

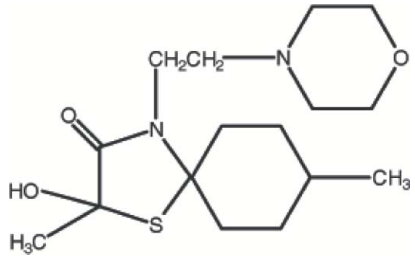
Received 16 July 2008; accepted 17 July 2008

Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.036; $wR$ factor = 0.094; data-to-parameter ratio = 18.6.

The title compound, $C_{16}H_{28}N_2O_3S$, is dimerized by inversion symmetry-related intermolecular O—H···N hydrogen bonding, forming an $R_2^2(16)$ motif. The dimers are also linked through intermolecular C—H···O hydrogen bonding. The compound is chiral with a stereogenic centre located in the thiazole ring, but in the crystal structure it forms a racemate. The thiazole ring has an envelope conformation, while the cyclohexane and morpholine rings adopt chair conformations.

## Related literature

For general background, see: Andres *et al.* (2000); Çapan *et al.* (1999); Srivastava *et al.* (2005). For related literature and bond-length data, see: Akkurt *et al.* (2007); Akkurt, Yalçın, Güzeldemirci *et al.* (2008); Akkurt, Yalçın, Klip *et al.* (2008). For ring conformation puckering parameters, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).




## Experimental

### *Crystal data*

$C_{16}H_{28}N_2O_3S$
$M_r$ = 328.47
Triclinic, $P\overline{1}$
$a$ = 8.0753 (4) Å
$b$ = 10.2002 (5) Å
$c$ = 11.8734 (6) Å
$\alpha$ = 82.467 (4)°
$\beta$ = 71.487 (4)°
$\gamma$ = 68.965 (4)°
$V$ = 865.44 (8) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.20 mm$^{-1}$
$T$ = 296 K
0.73 × 0.45 × 0.29 mm

### *Data collection*

Stoe IPDS-2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min}$ = 0.867, $T_{max}$ = 0.944
18220 measured reflections
3693 independent reflections
3319 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.093
$S$ = 1.04
3693 reflections
199 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···N2$^{i}$ | 0.82 | 2.00 | 2.8104 (14) | 169 |
| C7—H7$B$···S1 | 0.97 | 2.82 | 3.2235 (18) | 106 |
| C14—H14$B$···O1$^{ii}$ | 0.97 | 2.52 | 3.221 (2) | 129 |

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

The authors thank the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for use of the Stoe IPDS II diffractometer (purchased under grant No. F.279 of the University Research Fund). This work was supported by the Research Fund of İstanbul University (project Nos. 177/15012004 and UDP-730/05052006).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2184).

## References

Akkurt, M., Yalçın, Ş. P., Gürsoy, E., Güzeldemirci, N. U. & Büyükgüngör, O. (2007). *Acta Cryst.* E**63**, o3103.
Akkurt, M., Yalçın, Ş. P., Güzeldemirci, N. U. & Büyükgüngör, O. (2008). *Acta Cryst.* E**64**, o810–o811.
Akkurt, M., Yalçın, Ş. P., Klip, N. T. & Büyükgüngör, O. (2008). *Acta Cryst.* E**64**, o1572–o1573.
Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Andres, C. J., Bronson, J. J., D'Andrea, S. V., Deshpande, M. S., Falk, P. J., Grant-Young, K. A., Harte, W. E., Ho, H.-T., Misco, P. F., Robertson, J. G., Stock, D., Sun, Y. & Walsh, A. W. (2000). *Bioorg. Med. Chem. Lett.* **10**, 715–717.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Çapan, G., Ulusoy, N., Ergenç, N. & Kiraz, M. (1999). *Monatsh. Chem.* **130**, 1399–1407.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Srivastava, T., Gaikwad, A. K., Haq, W., Sinha, S. & Kati, S. B. (2005). *Arkivoc*, (**ii**), 120–130.
Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.

# supplementary materials

*Acta Cryst.* (2008). E**64**, o1574-o1575 [ **doi:10.1107/S1600536808022447** ]

# *rac*-2-Hydroxy-2,8-dimethyl-4-morpholinoethyl-1-thia-4-azaspiro[4.5]decan-3-one

**M. Akkurt, S.P. Yalçin, N. T. Klip and O. Büyükgüngör**

## Comment

Thiazolidinones and their spiroheterocyclic analogues have been reported to exhibit antibacterial (Andres *et al.*, 2000), antifungal (Çapan *et al.*, 1999) and antimycobacterial (Srivastava *et al.*, 2005) activity. In view of these observations, we synthesized the title spiro[4.5]decane derivative as a racemate and report its crystal structure.

In the title molecule (Fig. 1), the values of the geometric parameters are normal and comparable with those in the similar compound, 8-methyl-4-morpholinoethyl-1-thia- 4-azaspiro[4.5]decan-3-one (Akkurt, Yalçın, Klip *et al.*, 2008), which is a spiro[4.5]decane derivative.

The title compound is dimerized by inversion-symmetry related intermolecular O—H···N hydrogen bonds, forming an $R_2^2(16)$ motif (Bernstein *et al.*,1995) (Fig. 2). The dimers are interlinked through intermolecular C—H···O hydrogen bonds. The compound is chiral with a stereogenic centre C1, in the thiazole ring. As the structure is centrosymmetric, racemate occurs in the crystal. The thiazole ring (C1–C3/S1/N1) has an envelope conformation on S1 [puckering parameters (Cremer & Pople, 1975): Q(2) = 0.1885 (12) Å, φ(2) = 5.7 (4) °]. The cyclohexane and morpholine rings (C3–C8) and (C12–C15/N2/O2) adopt chair conformations [puckering parameters: $Q_T$ = 0.563 (2) Å, θ = 177.6 (2) °, φ = 58 (4) °, and $Q_T$ = 0.576 (2) Å, θ = 0.28 (15) °, φ = 340 (60) °, respectively].

The structure is stabilized by intramolecular C—H···S and intermolecular O—H···N and C—H···O hydrogen bonds (Table 1, Fig. 2).

## Experimental

A mixture of morpholinoethylamin (5 mmol), 4-methyl cyclohexanone (5 mmol) and α-mercaptopropionic acid (20 mmol) in dry benzene (20 ml) was refluxed for 18 h using a Dean-Stark water separator. Excess solvent was evaporated *in vacuo*. The residue was taken up in chloroform. The chloroform layer was triturated with saturated $NaHCO_3$ solution (2x) before drying over sodium sulfate and concentrated under reduced pressure to dryness. The crude product was triturated with diethyl ether several times and recrystallized from ethanol to yield racemic mixture as colourless prisms. IR (ν, $cm^{-1}$): 1678 (C=O). $^1$H-NMR (δ, DMSO-$d_6$, 400 MHz): 0.84 (3*H*, d, *J*=6.0 Hz, 8-$CH_3$), 0.96–1.03 (1*H*, m, cycl. CH), 1.15–1.24 (1*H*, m, cycl.CH), 1.32–1.42 (3*H*, m, cycl.CH), 1.51–1.52 (1*H*, m, cycl.CH), 1.66 (2*H*, d, *J*=12.8 Hz, cycl.CH), 1.79 (1*H*, dd, *J*=12.8, 2.8 Hz, cycl.CH), 1.95- 2.10 (3*H*, m, $SCHCH_3$), 2.33–2.44 (6*H*, m, morph.N—$CH_2$), 3.30–3.37 (3*H*, m, SCH and N—$CH_2$), 3.54 (4*H*, t, *J*=4.4 Hz, $OCH_2$). LC—MS (m/*z*): 313 (*M*+1).

### Refinement

H atoms were positioned geometrically, with C—H = 0.98–0.96 Å, O—H = 0.82 Å and constrained to ride on their parent atoms. The thermal parameter of H-atoms of methyl and hydroxyl groups was taken 1.5 times of the parent atom, whereas for all other H-atoms it was taken 1.2 times of their parent atoms.

### Figures

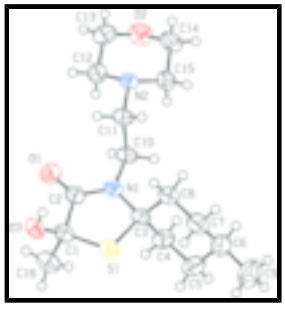


Fig. 1. The *ORTEP* diagram of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

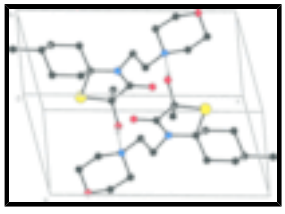


Fig. 2. View of the dimer generated by inversion symmetry related O—H···N hydrogen bonds, forming a $R_2^2(16)$ motif. For clarity, H atoms not involved in hydrogen bonds have been omitted.

### *rac*-2-Hydroxy-2,8-dimethyl-4-morpholinoethyl-1-thia-4- azaspiro[4.5]decan-3-one

*Crystal data*

| | |
|---|---|
| $C_{16}H_{28}N_2O_3S$ | $Z = 2$ |
| $M_r = 328.47$ | $F_{000} = 356$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.260$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 8.0753$ (4) Å | Cell parameters from 29023 reflections |
| $b = 10.2002$ (5) Å | $\theta = 2.1$–$27.4$º |
| $c = 11.8734$ (6) Å | $\mu = 0.20$ mm$^{-1}$ |
| $\alpha = 82.467$ (4)º | $T = 296$ K |
| $\beta = 71.487$ (4)º | Block, colourless |
| $\gamma = 68.965$ (4)º | 0.73 × 0.45 × 0.29 mm |
| $V = 865.44$ (8) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Stoe IPDS-2 diffractometer | 3693 independent reflections |
| Monochromator: plane graphite | 3319 reflections with $I > 2\sigma(I)$ |

| | |
|---|---|
| Detector resolution: 6.67 pixels $mm^{-1}$ | $R_{int}$ = 0.038 |
| $T$ = 296 K | $\theta_{max}$ = 26.9º |
| ω scans | $\theta_{min}$ = 2.1º |
| Absorption correction: integration (X-RED32; Stoe & Cie, 2002) | $h$ = −10→10 |
| $T_{min}$ = 0.867, $T_{max}$ = 0.944 | $k$ = −12→12 |
| 18220 measured reflections | $l$ = −15→15 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.036 | H-atom parameters constrained |
| $wR(F^2)$ = 0.093 | $w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.1524P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.04 | $(\Delta/\sigma)_{max} < 0.001$ |
| 3693 reflections | $\Delta\rho_{max}$ = 0.31 e $Å^{-3}$ |
| 199 parameters | $\Delta\rho_{min}$ = −0.21 e $Å^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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 | 0.33771 (4) | 0.24227 (4) | 0.79561 (3) | 0.0486 (1) |
| O1 | 0.45350 (15) | 0.25388 (12) | 0.45525 (9) | 0.0555 (3) |
| O2 | 1.23062 (15) | 0.29531 (12) | 0.15285 (9) | 0.0597 (3) |
| O3 | 0.11938 (12) | 0.32395 (10) | 0.65362 (9) | 0.0480 (3) |
| N1 | 0.59680 (14) | 0.23670 (11) | 0.59509 (9) | 0.0389 (3) |
| N2 | 0.90885 (14) | 0.39307 (11) | 0.34820 (9) | 0.0372 (3) |
| C1 | 0.28811 (17) | 0.22315 (13) | 0.65981 (12) | 0.0417 (4) |
| C2 | 0.45358 (17) | 0.24085 (13) | 0.55883 (11) | 0.0408 (3) |
| C3 | 0.58346 (16) | 0.22120 (13) | 0.72210 (10) | 0.0369 (3) |
| C4 | 0.7085 (2) | 0.07643 (14) | 0.75151 (12) | 0.0482 (4) |

| | | | | |
|---|---|---|---|---|
| C5 | 0.7034 (2) | 0.06187 (17) | 0.88162 (14) | 0.0583 (5) |
| C6 | 0.7495 (2) | 0.17691 (19) | 0.92078 (13) | 0.0587 (5) |
| C7 | 0.6200 (2) | 0.31982 (17) | 0.89350 (13) | 0.0541 (5) |
| C8 | 0.62959 (19) | 0.33651 (14) | 0.76215 (12) | 0.0445 (4) |
| C9 | 0.7389 (3) | 0.1587 (3) | 1.05180 (17) | 0.0881 (8) |
| C10 | 0.76951 (17) | 0.23709 (13) | 0.50474 (11) | 0.0413 (3) |
| C11 | 0.75792 (17) | 0.38482 (13) | 0.45390 (11) | 0.0408 (3) |
| C12 | 0.90146 (19) | 0.34106 (16) | 0.24137 (11) | 0.0471 (4) |
| C13 | 1.0540 (2) | 0.36400 (19) | 0.13593 (12) | 0.0584 (5) |
| C14 | 1.2384 (2) | 0.34737 (18) | 0.25562 (13) | 0.0554 (5) |
| C15 | 1.09318 (17) | 0.32359 (15) | 0.36461 (11) | 0.0432 (4) |
| C16 | 0.2750 (2) | 0.08031 (15) | 0.65024 (15) | 0.0561 (5) |
| H3 | 0.12210 | 0.40260 | 0.65820 | 0.0720* |
| H4A | 0.66870 | 0.00470 | 0.73310 | 0.0580* |
| H4B | 0.83500 | 0.06140 | 0.70220 | 0.0580* |
| H5A | 0.58070 | 0.06390 | 0.93020 | 0.0700* |
| H5B | 0.79130 | −0.02850 | 0.89480 | 0.0700* |
| H6 | 0.87690 | 0.16960 | 0.87490 | 0.0700* |
| H7A | 0.65370 | 0.39320 | 0.91510 | 0.0650* |
| H7B | 0.49370 | 0.33050 | 0.94100 | 0.0650* |
| H8A | 0.54300 | 0.42730 | 0.74850 | 0.0530* |
| H8B | 0.75330 | 0.33370 | 0.71510 | 0.0530* |
| H9A | 0.82270 | 0.06800 | 1.06540 | 0.1320* |
| H9B | 0.61470 | 0.16630 | 1.09830 | 0.1320* |
| H9C | 0.77270 | 0.23040 | 1.07430 | 0.1320* |
| H10A | 0.79540 | 0.17570 | 0.44090 | 0.0500* |
| H10B | 0.87110 | 0.20060 | 0.53970 | 0.0500* |
| H11A | 0.64170 | 0.42750 | 0.43460 | 0.0490* |
| H11B | 0.75450 | 0.44050 | 0.51540 | 0.0490* |
| H12A | 0.78170 | 0.39070 | 0.22810 | 0.0570* |
| H12B | 0.91750 | 0.24180 | 0.25140 | 0.0570* |
| H13A | 1.05010 | 0.32880 | 0.06510 | 0.0700* |
| H13B | 1.03320 | 0.46390 | 0.12390 | 0.0700* |
| H14A | 1.21920 | 0.44710 | 0.24480 | 0.0670* |
| H14B | 1.36030 | 0.30050 | 0.26640 | 0.0670* |
| H15A | 1.11570 | 0.22360 | 0.37830 | 0.0520* |
| H15B | 1.09970 | 0.36120 | 0.43340 | 0.0520* |
| H16A | 0.17160 | 0.06840 | 0.71300 | 0.0840* |
| H16B | 0.38720 | 0.00790 | 0.65670 | 0.0840* |
| H16C | 0.25790 | 0.07430 | 0.57490 | 0.0840* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0372 (2) | 0.0676 (2) | 0.0380 (2) | −0.0232 (2) | −0.0003 (1) | −0.0024 (1) |
| O1 | 0.0540 (6) | 0.0738 (7) | 0.0406 (5) | −0.0258 (5) | −0.0106 (4) | −0.0033 (4) |
| O2 | 0.0495 (6) | 0.0739 (7) | 0.0429 (5) | −0.0201 (5) | 0.0074 (4) | −0.0128 (5) |
| O3 | 0.0364 (5) | 0.0458 (5) | 0.0612 (6) | −0.0141 (4) | −0.0110 (4) | −0.0065 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| N1 | 0.0332 (5) | 0.0452 (6) | 0.0346 (5) | −0.0165 (4) | −0.0011 (4) | −0.0009 (4) |
| N2 | 0.0344 (5) | 0.0407 (5) | 0.0322 (5) | −0.0144 (4) | −0.0012 (4) | −0.0031 (4) |
| C1 | 0.0360 (6) | 0.0429 (6) | 0.0448 (7) | −0.0163 (5) | −0.0054 (5) | −0.0035 (5) |
| C2 | 0.0389 (6) | 0.0406 (6) | 0.0407 (6) | −0.0150 (5) | −0.0059 (5) | −0.0034 (5) |
| C3 | 0.0334 (5) | 0.0390 (6) | 0.0353 (6) | −0.0152 (5) | −0.0026 (4) | −0.0006 (4) |
| C4 | 0.0480 (7) | 0.0408 (7) | 0.0478 (7) | −0.0115 (6) | −0.0081 (6) | 0.0007 (5) |
| C5 | 0.0593 (9) | 0.0556 (8) | 0.0498 (8) | −0.0133 (7) | −0.0140 (7) | 0.0106 (6) |
| C6 | 0.0453 (7) | 0.0845 (11) | 0.0437 (7) | −0.0222 (7) | −0.0098 (6) | −0.0002 (7) |
| C7 | 0.0564 (8) | 0.0637 (9) | 0.0458 (7) | −0.0296 (7) | −0.0056 (6) | −0.0099 (6) |
| C8 | 0.0460 (7) | 0.0430 (7) | 0.0447 (7) | −0.0210 (5) | −0.0064 (5) | −0.0018 (5) |
| C9 | 0.0772 (13) | 0.1301 (19) | 0.0518 (10) | −0.0263 (12) | −0.0219 (9) | −0.0022 (10) |
| C10 | 0.0330 (6) | 0.0421 (6) | 0.0391 (6) | −0.0125 (5) | 0.0022 (5) | −0.0012 (5) |
| C11 | 0.0355 (6) | 0.0396 (6) | 0.0387 (6) | −0.0126 (5) | 0.0018 (5) | −0.0038 (5) |
| C12 | 0.0457 (7) | 0.0579 (8) | 0.0387 (6) | −0.0195 (6) | −0.0102 (5) | −0.0038 (5) |
| C13 | 0.0639 (9) | 0.0734 (10) | 0.0334 (7) | −0.0260 (8) | −0.0045 (6) | −0.0019 (6) |
| C14 | 0.0409 (7) | 0.0703 (9) | 0.0520 (8) | −0.0259 (7) | 0.0014 (6) | −0.0080 (7) |
| C15 | 0.0355 (6) | 0.0517 (7) | 0.0395 (6) | −0.0150 (5) | −0.0048 (5) | −0.0055 (5) |
| C16 | 0.0533 (8) | 0.0466 (8) | 0.0683 (9) | −0.0239 (6) | −0.0077 (7) | −0.0058 (6) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C1 | 1.8295 (14) | C4—H4B | 0.9700 |
| S1—C3 | 1.8410 (14) | C5—H5A | 0.9700 |
| O1—C2 | 1.2197 (16) | C5—H5B | 0.9700 |
| O2—C13 | 1.412 (2) | C6—H6 | 0.9800 |
| O2—C14 | 1.4204 (19) | C7—H7A | 0.9700 |
| O3—C1 | 1.3990 (17) | C7—H7B | 0.9700 |
| O3—H3 | 0.8200 | C8—H8A | 0.9700 |
| N1—C3 | 1.4688 (15) | C8—H8B | 0.9700 |
| N1—C10 | 1.4659 (18) | C9—H9A | 0.9600 |
| N1—C2 | 1.3420 (19) | C9—H9B | 0.9600 |
| N2—C12 | 1.4633 (17) | C9—H9C | 0.9600 |
| N2—C15 | 1.4651 (19) | C10—H10A | 0.9700 |
| N2—C11 | 1.4636 (17) | C10—H10B | 0.9700 |
| C1—C2 | 1.535 (2) | C11—H11A | 0.9700 |
| C1—C16 | 1.520 (2) | C11—H11B | 0.9700 |
| C3—C8 | 1.524 (2) | C12—H12A | 0.9700 |
| C3—C4 | 1.5298 (19) | C12—H12B | 0.9700 |
| C4—C5 | 1.521 (2) | C13—H13A | 0.9700 |
| C5—C6 | 1.516 (2) | C13—H13B | 0.9700 |
| C6—C9 | 1.521 (2) | C14—H14A | 0.9700 |
| C6—C7 | 1.523 (2) | C14—H14B | 0.9700 |
| C7—C8 | 1.527 (2) | C15—H15A | 0.9700 |
| C10—C11 | 1.5297 (18) | C15—H15B | 0.9700 |
| C12—C13 | 1.513 (2) | C16—H16A | 0.9600 |
| C14—C15 | 1.506 (2) | C16—H16B | 0.9600 |
| C4—H4A | 0.9700 | C16—H16C | 0.9600 |
| S1···N1 | 2.6146 (11) | H5B···H9A | 2.4800 |
| S1···H5A | 2.8900 | H6···H8B | 2.5600 |

| | | | |
|---|---|---|---|
| S1···H7B | 2.8200 | H6···H16A$^{v}$ | 2.5100 |
| O1···O3 | 2.8913 (15) | H7A···H9C | 2.5400 |
| O1···C11 | 3.187 (2) | H7A···H14A$^{vi}$ | 2.5400 |
| O1···C14$^{i}$ | 3.221 (2) | H7B···S1 | 2.8200 |
| O1···C15$^{i}$ | 3.230 (2) | H7B···O2$^{x}$ | 2.8100 |
| O2···N2 | 2.8364 (16) | H7B···H5A | 2.5600 |
| O3···C15$^{ii}$ | 3.3942 (17) | H7B···H9B | 2.5300 |
| O3···O1 | 2.8913 (15) | H8A···H12A$^{ii}$ | 2.5600 |
| O3···N2$^{ii}$ | 2.8104 (14) | H8B···O3$^{v}$ | 2.7800 |
| O1···H10A | 2.5400 | H8B···C10 | 2.7500 |
| O1···H11A | 2.6600 | H8B···C11 | 3.0700 |
| O1···H15B$^{i}$ | 2.7500 | H8B···H6 | 2.5600 |
| O1···H16C | 2.8000 | H8B···H10B | 2.3800 |
| O1···H14B$^{i}$ | 2.5200 | H8B···H11B | 2.4700 |
| O1···H16B$^{iii}$ | 2.8200 | H8B···H14A$^{vi}$ | 2.4400 |
| O2···H9B$^{iv}$ | 2.7900 | H9A···H5B | 2.4800 |
| O2···H7B$^{iv}$ | 2.8100 | H9B···O2$^{x}$ | 2.7900 |
| O3···H15B$^{i}$ | 2.6400 | H9B···C14$^{x}$ | 3.0700 |
| O3···H8B$^{i}$ | 2.7800 | H9B···H5A | 2.5000 |
| O3···H14A$^{ii}$ | 2.9100 | H9B···H7B | 2.5300 |
| N1···S1 | 2.6146 (11) | H9B···H14B$^{x}$ | 2.4800 |
| N2···O2 | 2.8364 (16) | H9C···C12$^{xi}$ | 3.0000 |
| N2···O3$^{ii}$ | 2.8104 (14) | H9C···H7A | 2.5400 |
| N2···H3$^{ii}$ | 2.0000 | H10A···O1 | 2.5400 |
| C2···C11$^{ii}$ | 3.5847 (18) | H10A···C12 | 2.8100 |
| C8···C11 | 3.4925 (18) | H10A···H12B | 2.2700 |
| C11···C8 | 3.4925 (18) | H10B···C4 | 2.8100 |
| C11···C2$^{ii}$ | 3.5847 (18) | H10B···C8 | 2.9100 |
| C11···O1 | 3.187 (2) | H10B···C15 | 2.7700 |
| C14···O1$^{v}$ | 3.221 (2) | H10B···H4B | 2.2500 |
| C15···O1$^{v}$ | 3.230 (2) | H10B···H8B | 2.3800 |
| C15···O3$^{ii}$ | 3.3942 (17) | H10B···H15A | 2.3300 |
| C2···H11A | 2.8300 | H11A···O1 | 2.6600 |
| C4···H10B | 2.8100 | H11A···C2 | 2.8300 |
| C8···H14A$^{vi}$ | 2.8600 | H11A···H12A | 2.3700 |
| C8···H10B | 2.9100 | H11A···C11$^{ii}$ | 3.0600 |
| C8···H11B | 2.9700 | H11A···H11A$^{ii}$ | 2.4000 |
| C10···H12B | 2.8600 | H11B···C8 | 2.9700 |
| C10···H4B | 2.8300 | H11B···H8B | 2.4700 |
| C10···H15A | 2.6800 | H11B···H15B | 2.5000 |
| C10···H8B | 2.7500 | H12A···H11A | 2.3700 |
| C11···H8B | 3.0700 | H12A···H8A$^{ii}$ | 2.5600 |
| C11···H3$^{ii}$ | 2.7100 | H12B···C10 | 2.8600 |

| | | | |
|---|---|---|---|
| C11···H11A$^{ii}$ | 3.0600 | H12B···H10A | 2.2700 |
| C12···H3$^{ii}$ | 2.9300 | H12B···H15A | 2.4700 |
| C12···H10A | 2.8100 | H13B···H14A | 2.3400 |
| C12···H9C$^{vii}$ | 3.0000 | H14A···H13B | 2.3400 |
| C14···H9B$^{iv}$ | 3.0700 | H14A···O3$^{ii}$ | 2.9100 |
| C14···H3$^{ii}$ | 3.0800 | H14A···C8$^{vi}$ | 2.8600 |
| C15···H10B | 2.7700 | H14A···H3$^{ii}$ | 2.5900 |
| C15···H3$^{ii}$ | 2.7400 | H14A···H7A$^{vi}$ | 2.5400 |
| H3···N2$^{ii}$ | 2.0000 | H14A···H8B$^{vi}$ | 2.4400 |
| H3···C11$^{ii}$ | 2.7100 | H14B···O1$^{v}$ | 2.5200 |
| H3···C12$^{ii}$ | 2.9300 | H14B···H9B$^{iv}$ | 2.4800 |
| H3···C14$^{ii}$ | 3.0800 | H15A···C10 | 2.6800 |
| H3···C15$^{ii}$ | 2.7400 | H15A···H10B | 2.3300 |
| H3···H14A$^{ii}$ | 2.5900 | H15A···H12B | 2.4700 |
| H4A···H15A$^{viii}$ | 2.5800 | H15A···H4A$^{viii}$ | 2.5800 |
| H4B···C10 | 2.8300 | H15B···O1$^{v}$ | 2.7500 |
| H4B···H10B | 2.2500 | H15B···O3$^{v}$ | 2.6400 |
| H5A···S1 | 2.8900 | H15B···H11B | 2.5000 |
| H5A···H7B | 2.5600 | H16A···H6$^{i}$ | 2.5100 |
| H5A···H9B | 2.5000 | H16B···O1$^{iii}$ | 2.8200 |
| H5A···H5A$^{ix}$ | 2.3300 | H16C···O1 | 2.8000 |
| C1—S1—C3 | 94.96 (6) | C6—C7—H7B | 109.00 |
| C13—O2—C14 | 109.74 (12) | C8—C7—H7A | 109.00 |
| C1—O3—H3 | 109.00 | C8—C7—H7B | 109.00 |
| C2—N1—C10 | 118.36 (10) | H7A—C7—H7B | 108.00 |
| C3—N1—C10 | 121.36 (11) | C3—C8—H8A | 109.00 |
| C2—N1—C3 | 120.12 (11) | C3—C8—H8B | 109.00 |
| C11—N2—C15 | 113.24 (10) | C7—C8—H8A | 109.00 |
| C12—N2—C15 | 109.83 (11) | C7—C8—H8B | 109.00 |
| C11—N2—C12 | 113.29 (11) | H8A—C8—H8B | 108.00 |
| S1—C1—C2 | 104.53 (10) | C6—C9—H9A | 109.00 |
| S1—C1—C16 | 113.45 (10) | C6—C9—H9B | 109.00 |
| S1—C1—O3 | 110.65 (9) | C6—C9—H9C | 109.00 |
| O3—C1—C16 | 107.00 (12) | H9A—C9—H9B | 110.00 |
| C2—C1—C16 | 108.91 (11) | H9A—C9—H9C | 109.00 |
| O3—C1—C2 | 112.40 (11) | H9B—C9—H9C | 109.00 |
| O1—C2—C1 | 121.93 (14) | N1—C10—H10A | 109.00 |
| N1—C2—C1 | 113.93 (11) | N1—C10—H10B | 109.00 |
| O1—C2—N1 | 124.12 (13) | C11—C10—H10A | 109.00 |
| S1—C3—N1 | 103.79 (9) | C11—C10—H10B | 109.00 |
| S1—C3—C8 | 109.34 (9) | H10A—C10—H10B | 108.00 |
| N1—C3—C4 | 111.50 (10) | N2—C11—H11A | 108.00 |
| S1—C3—C4 | 110.49 (9) | N2—C11—H11B | 108.00 |
| C4—C3—C8 | 110.34 (12) | C10—C11—H11A | 108.00 |
| N1—C3—C8 | 111.20 (10) | C10—C11—H11B | 108.00 |

| | | | |
|---|---|---|---|
| C3—C4—C5 | 111.95 (11) | H11A—C11—H11B | 107.00 |
| C4—C5—C6 | 112.88 (13) | N2—C12—H12A | 110.00 |
| C5—C6—C7 | 109.51 (14) | N2—C12—H12B | 110.00 |
| C7—C6—C9 | 111.87 (16) | C13—C12—H12A | 110.00 |
| C5—C6—C9 | 110.62 (16) | C13—C12—H12B | 110.00 |
| C6—C7—C8 | 111.54 (12) | H12A—C12—H12B | 108.00 |
| C3—C8—C7 | 111.69 (12) | O2—C13—H13A | 109.00 |
| N1—C10—C11 | 111.71 (11) | O2—C13—H13B | 109.00 |
| N2—C11—C10 | 115.84 (11) | C12—C13—H13A | 109.00 |
| N2—C12—C13 | 109.15 (13) | C12—C13—H13B | 109.00 |
| O2—C13—C12 | 111.53 (12) | H13A—C13—H13B | 108.00 |
| O2—C14—C15 | 111.11 (14) | O2—C14—H14A | 109.00 |
| N2—C15—C14 | 109.61 (11) | O2—C14—H14B | 109.00 |
| C3—C4—H4A | 109.00 | C15—C14—H14A | 109.00 |
| C3—C4—H4B | 109.00 | C15—C14—H14B | 109.00 |
| C5—C4—H4A | 109.00 | H14A—C14—H14B | 108.00 |
| C5—C4—H4B | 109.00 | N2—C15—H15A | 110.00 |
| H4A—C4—H4B | 108.00 | N2—C15—H15B | 110.00 |
| C4—C5—H5A | 109.00 | C14—C15—H15A | 110.00 |
| C4—C5—H5B | 109.00 | C14—C15—H15B | 110.00 |
| C6—C5—H5A | 109.00 | H15A—C15—H15B | 108.00 |
| C6—C5—H5B | 109.00 | C1—C16—H16A | 109.00 |
| H5A—C5—H5B | 108.00 | C1—C16—H16B | 109.00 |
| C5—C6—H6 | 108.00 | C1—C16—H16C | 109.00 |
| C7—C6—H6 | 108.00 | H16A—C16—H16B | 109.00 |
| C9—C6—H6 | 108.00 | H16A—C16—H16C | 109.00 |
| C6—C7—H7A | 109.00 | H16B—C16—H16C | 109.00 |
| C3—S1—C1—O3 | −136.07 (10) | C12—N2—C15—C14 | 56.88 (15) |
| C3—S1—C1—C2 | −14.85 (9) | C12—N2—C11—C10 | 71.48 (15) |
| C3—S1—C1—C16 | 103.66 (11) | S1—C1—C2—N1 | 12.26 (13) |
| C1—S1—C3—N1 | 13.84 (9) | O3—C1—C2—O1 | −49.39 (17) |
| C1—S1—C3—C4 | −105.81 (9) | O3—C1—C2—N1 | 132.32 (12) |
| C1—S1—C3—C8 | 132.57 (9) | C16—C1—C2—O1 | 69.00 (17) |
| C14—O2—C13—C12 | −59.69 (17) | C16—C1—C2—N1 | −109.30 (13) |
| C13—O2—C14—C15 | 59.60 (16) | S1—C1—C2—O1 | −169.45 (11) |
| C2—N1—C10—C11 | 79.30 (14) | N1—C3—C8—C7 | −178.85 (12) |
| C3—N1—C2—O1 | 179.78 (12) | C8—C3—C4—C5 | 52.97 (16) |
| C10—N1—C2—O1 | −4.83 (19) | S1—C3—C8—C7 | 67.13 (14) |
| C3—N1—C10—C11 | −105.38 (13) | N1—C3—C4—C5 | 177.06 (13) |
| C2—N1—C3—C8 | −126.75 (13) | C4—C3—C8—C7 | −54.59 (16) |
| C10—N1—C3—C8 | 58.01 (15) | S1—C3—C4—C5 | −68.06 (15) |
| C10—N1—C2—C1 | 173.42 (10) | C3—C4—C5—C6 | −54.55 (19) |
| C2—N1—C3—C4 | 109.65 (14) | C4—C5—C6—C7 | 55.23 (18) |
| C2—N1—C3—S1 | −9.30 (13) | C4—C5—C6—C9 | 178.97 (16) |
| C10—N1—C3—S1 | 175.45 (9) | C5—C6—C7—C8 | −56.25 (18) |
| C10—N1—C3—C4 | −65.59 (15) | C9—C6—C7—C8 | −179.25 (16) |
| C3—N1—C2—C1 | −1.97 (16) | C6—C7—C8—C3 | 57.30 (18) |
| C11—N2—C12—C13 | 175.83 (12) | N1—C10—C11—N2 | −169.01 (11) |
| C15—N2—C11—C10 | −54.44 (15) | N2—C12—C13—O2 | 58.47 (17) |

| | | | |
|---|---|---|---|
| C15—N2—C12—C13 | −56.45 (15) | O2—C14—C15—N2 | −58.52 (16) |
| C11—N2—C15—C14 | −175.38 (11) | | |

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*+1, *y*, *z*−1; (v) *x*+1, *y*, *z*; (vi) −*x*+2, −*y*+1, −*z*+1; (vii) *x*, *y*, *z*−1; (viii) −*x*+2, −*y*, −*z*+1; (ix) −*x*+1, −*y*, −*z*+2; (x) *x*−1, *y*, *z*+1; (xi) *x*, *y*, *z*+1.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···N2$^{ii}$ | 0.82 | 2.00 | 2.8104 (14) | 169 |
| C7—H7B···S1 | 0.97 | 2.82 | 3.2235 (18) | 106 |
| C14—H14B···O1$^{v}$ | 0.97 | 2.52 | 3.221 (2) | 129 |

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

**Fig. 1**

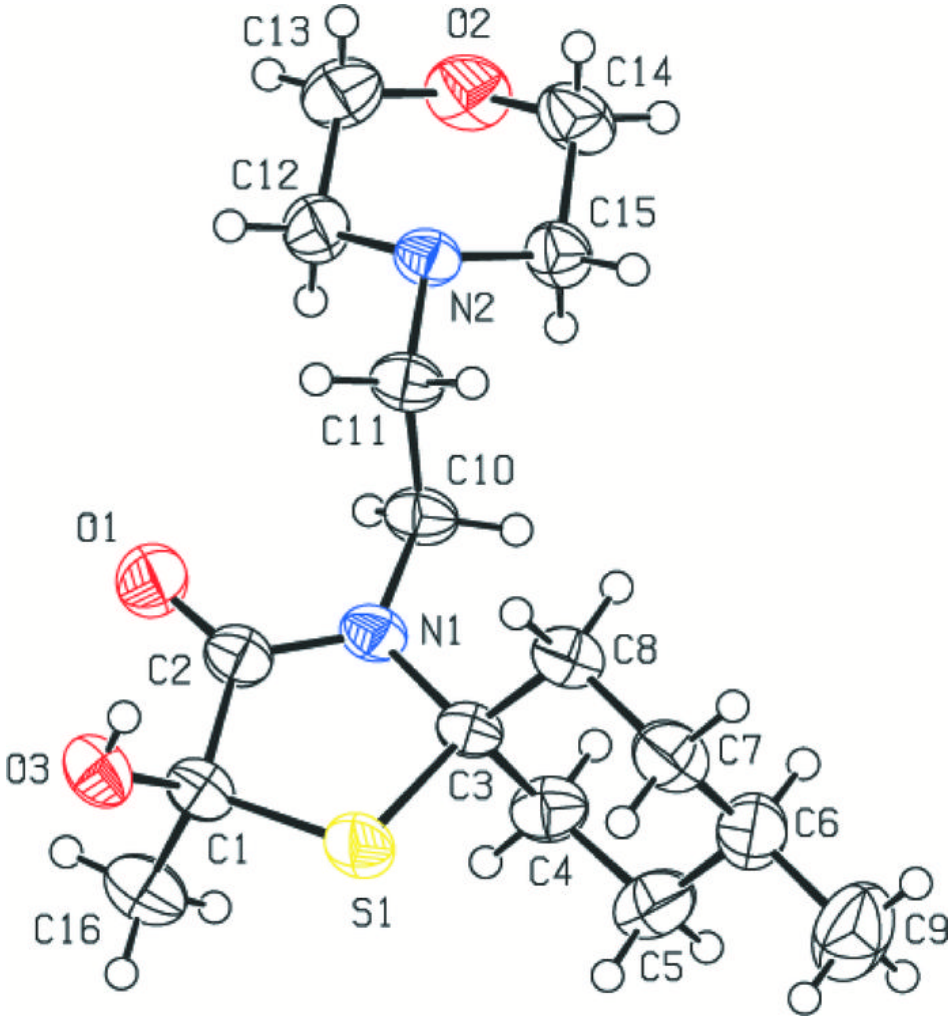

C13
O2
C14
C12
C15
N2
C11
C10
O1
N1
C8
C2
C7
O3
C3
C6
C1
C4
S1
C16
C5
C9

**Fig. 2**