



Crystal structure of 1-hydroxy-2,2,6,6-tetramethylpiperidin-1-ium trifluoromethanesulfonate

Christian Godemann, Anke Spannenberg and Torsten Beweries*

Leibniz-Institut für Katalyse e.V. an der Universität Rostock, Albert-Einstein-Strasse 29a, 18059 Rostock, Germany. *Correspondence e-mail: torsten.beweries@catalysis.de

Received 26 October 2015; accepted 4 November 2015

Edited by C. Rizzoli, Università degli Studi di Parma, Italy

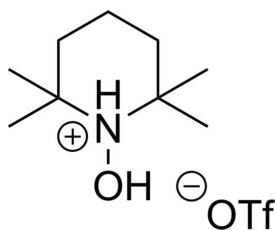
In the cation of the title salt, $\text{C}_9\text{H}_{20}\text{NO}^+\cdot\text{CF}_3\text{O}_3\text{S}^-$, the six-membered heterocyclic ring displays a chair conformation. In the crystal, centrosymmetric pairs of cations and anions are linked by $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds to form rings with a $R_4^4(14)$ graph-set motif.

Keywords: crystal structure; TEMPO; ammonium salt; triflate; hydrogen bonding.

CCDC reference: 1435030

1. Related literature

For molecular structures and discussions of related compounds, see: Jaitner & Wurst (1997); Spirk *et al.* (2010); Ananchenko *et al.* (2006); Percino *et al.* (2016). For the molecular structure of the neutral TEMPO-H compound, see: Mader *et al.* (2007); Giffin *et al.* (2011).



2. Experimental

2.1. Crystal data

$\text{C}_9\text{H}_{20}\text{NO}^+\cdot\text{CF}_3\text{O}_3\text{S}^-$
 $M_r = 307.33$
 Triclinic, $P\bar{1}$
 $a = 8.2824(2) \text{ \AA}$
 $b = 8.7656(2) \text{ \AA}$
 $c = 10.5703(3) \text{ \AA}$

$\alpha = 79.5417(7)^\circ$
 $\beta = 76.5159(7)^\circ$
 $\gamma = 75.5022(6)^\circ$
 $V = 716.28(3) \text{ \AA}^3$
 $Z = 2$

Mo $K\alpha$ radiation
 $\mu = 0.27 \text{ mm}^{-1}$
 $T = 150 \text{ K}$
 $0.55 \times 0.38 \times 0.34 \text{ mm}$

2.2. Data collection

Bruker APEXII CCD
 diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2014)
 $T_{\min} = 0.83$, $T_{\max} = 0.86$

22933 measured reflections
 3452 independent reflections
 3039 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.094$
 $S = 1.05$
 3452 reflections
 184 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|------------|-------------|-------------|---------------|
| $\text{O1}-\text{H1A}\cdots\text{O4}$ | 0.84 (2) | 1.78 (2) | 2.6163 (14) | 177 (2) |
| $\text{N1}-\text{H1B}\cdots\text{O3}^1$ | 0.875 (16) | 1.991 (16) | 2.8385 (14) | 163.0 (14) |

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL2014*.

Acknowledgements

Financial support by the BMBF (project 'Light2Hydrogen') is gratefully acknowledged.

Supporting information for this paper is available from the IUCr electronic archives (Reference: RZ5176).

References

- Ananchenko, G. S., Pojarova, M., Udachin, K. A., Leek, D. M., Coleman, A. W. & Ripmeester, J. A. (2006). *Chem. Commun.* pp. 386–388.
- Bruker (2013). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2014). *APEX2* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Giffin, N. A., Makramalla, M., Hendsbee, A. D., Robertson, K. N., Sherren, C., Pye, C. C., Masuda, J. D. & Clyburne, J. A. C. (2011). *Org. Biomol. Chem.* **9**, 3672–3680.
- Jaitner, P. & Wurst, K. (1997). *Inorg. Chim. Acta*, **255**, 95–98.
- Mader, E. A., Davidson, E. R. & Mayer, J. M. (2007). *J. Am. Chem. Soc.* **129**, 5153–5166.
- Percino, M. J., Cerón, M., Soriano-Moro, G., Pacheco, J. A., Castro, M. E., Chapela, V. M., Bonilla-Cruz, J. & Saldivar-Guerra, E. (2016). *J. Mol. Struct.* **1103**, 254–264.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Spirk, S., Belaj, F., Madl, T. & Pietschnig, R. (2010). *Eur. J. Inorg. Chem.* pp. 289–297.

supporting information

Acta Cryst. (2015). E71, o921 [https://doi.org/10.1107/S2056989015020897]

Crystal structure of 1-hydroxy-2,2,6,6-tetramethylpiperidin-1-ium trifluoromethanesulfonate

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S1. Synthesis and crystallization

An equimolar mixture of the titanocene(IV) triflate complex $[(\text{SiMe}_2\text{C}_5\text{Me}_4)_2\text{Ti}(\text{H}_2\text{O})(\text{OH})(\text{OTf})]$ (Godemann & Beweries, unpublished results) and 2,2,6,6-tetramethyl-1-hydroxypiperidine (TEMPO-H) in toluene was cooled to -78°C . After two weeks, the formation of colourless crystals of the title compound could be observed on slow evaporation of the solvent. Alternatively, layering a toluene solution of the same titanocene compound and TEMPO-H with *n*-hexane also resulted in the formation of colourless crystals of the title compound.

S2. Refinement

The H1A and H1B atoms were found from a difference Fourier map and refined freely. All other H atoms were placed in idealized positions with $d(\text{C}-\text{H}) = 0.99 \text{ \AA}$ (CH_2), 0.98 \AA (CH_3) and refined using a riding model with $U_{\text{iso}}(\text{H})$ fixed at $1.2 U_{\text{eq}}(\text{C})$ for CH_2 and $1.5 U_{\text{eq}}(\text{C})$ for CH_3 . A rotating model was used for the methyl groups.

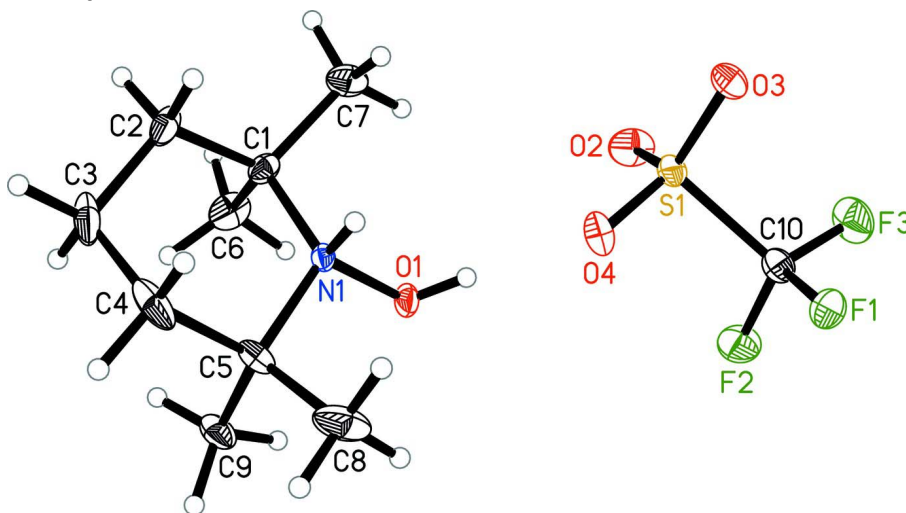


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

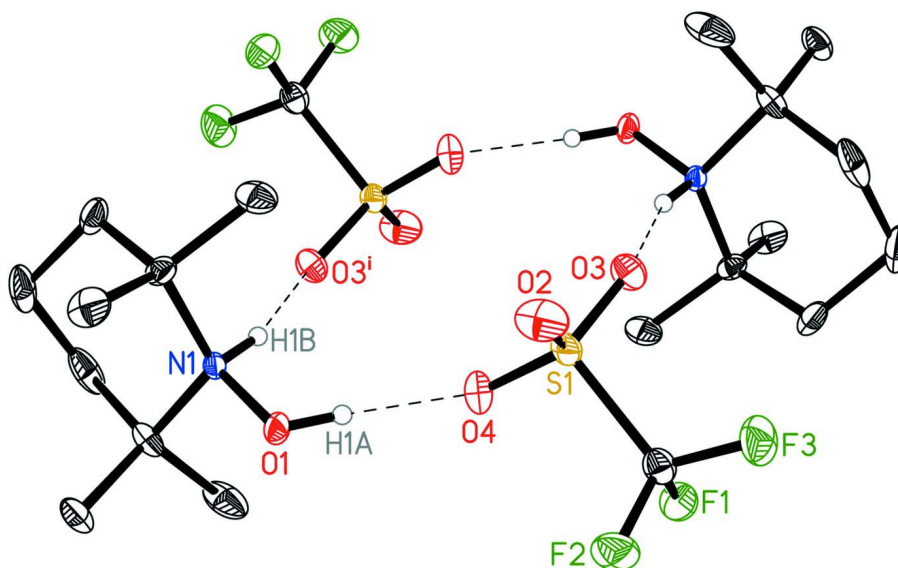


Figure 2

The hydrogen-bonding network (dashed lines) linking centrosymmetric pairs of cations and anions in the title compound. C-bound hydrogen atoms are omitted for clarity. Displacement ellipsoids are drawn at the 30% probability level.

1-Hydroxy-2,2,6,6-tetramethylpiperidin-1-ium trifluoromethanesulfonate

Crystal data

$\text{C}_9\text{H}_{20}\text{NO}^+\cdot\text{CF}_3\text{O}_3\text{S}^-$

$M_r = 307.33$

Triclinic, $P\bar{1}$

$a = 8.2824(2) \text{ \AA}$

$b = 8.7656(2) \text{ \AA}$

$c = 10.5703(3) \text{ \AA}$

$\alpha = 79.5417(7)^\circ$

$\beta = 76.5159(7)^\circ$

$\gamma = 75.5022(6)^\circ$

$V = 716.28(3) \text{ \AA}^3$

$Z = 2$

$F(000) = 324$

$D_x = 1.425 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9968 reflections

$\theta = 2.6\text{--}28.9^\circ$

$\mu = 0.27 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Prism, colourless

$0.55 \times 0.38 \times 0.34 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Detector resolution: $8.3333 \text{ pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2014)

$T_{\min} = 0.83$, $T_{\max} = 0.86$

22933 measured reflections

3452 independent reflections

3039 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 28.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.094$

$S = 1.05$

3452 reflections

184 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.2597P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$$

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.

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| C1 | 0.96872 (15) | 0.80108 (15) | 0.28534 (12) | 0.0251 (3) |
| C2 | 0.96590 (19) | 0.86192 (17) | 0.14106 (13) | 0.0371 (3) |
| H2A | 0.8640 | 0.9487 | 0.1352 | 0.045* |
| H2B | 1.0673 | 0.9072 | 0.1019 | 0.045* |
| C3 | 0.9637 (2) | 0.7356 (2) | 0.06137 (14) | 0.0503 (5) |
| H3A | 1.0691 | 0.6517 | 0.0611 | 0.060* |
| H3B | 0.9595 | 0.7834 | −0.0305 | 0.060* |
| C4 | 0.8101 (2) | 0.66311 (19) | 0.11958 (15) | 0.0456 (4) |
| H4A | 0.8100 | 0.5820 | 0.0655 | 0.055* |
| H4B | 0.7057 | 0.7472 | 0.1151 | 0.055* |
| C5 | 0.80421 (17) | 0.58576 (15) | 0.26184 (13) | 0.0287 (3) |
| C6 | 1.13968 (18) | 0.69450 (19) | 0.30494 (17) | 0.0396 (3) |
| H6A | 1.2234 | 0.7600 | 0.2937 | 0.059* |
| H6B | 1.1782 | 0.6194 | 0.2402 | 0.059* |
| H6C | 1.1273 | 0.6356 | 0.3936 | 0.059* |
| C7 | 0.92379 (19) | 0.93950 (17) | 0.36579 (16) | 0.0360 (3) |
| H7A | 0.9328 | 0.8983 | 0.4571 | 0.054* |
| H7B | 0.8072 | 0.9985 | 0.3625 | 0.054* |
| H7C | 1.0026 | 1.0105 | 0.3294 | 0.054* |
| C8 | 0.6305 (2) | 0.5497 (2) | 0.3250 (2) | 0.0511 (5) |
| H8A | 0.6018 | 0.4827 | 0.2724 | 0.077* |
| H8B | 0.5446 | 0.6494 | 0.3293 | 0.077* |
| H8C | 0.6334 | 0.4938 | 0.4138 | 0.077* |
| C9 | 0.94213 (19) | 0.43502 (16) | 0.27559 (15) | 0.0343 (3) |
| H9A | 0.9560 | 0.4085 | 0.3671 | 0.051* |
| H9B | 1.0498 | 0.4523 | 0.2192 | 0.051* |
| H9C | 0.9093 | 0.3473 | 0.2493 | 0.051* |
| C10 | 0.48030 (17) | 0.77783 (16) | 0.86989 (13) | 0.0301 (3) |
| F1 | 0.32733 (11) | 0.76163 (11) | 0.86237 (9) | 0.0403 (2) |
| F2 | 0.57731 (13) | 0.63247 (11) | 0.88758 (10) | 0.0488 (3) |
| F3 | 0.46049 (15) | 0.84651 (13) | 0.97606 (9) | 0.0522 (3) |
| N1 | 0.82282 (12) | 0.71302 (11) | 0.33670 (9) | 0.0180 (2) |
| O1 | 0.82554 (13) | 0.64554 (11) | 0.46877 (8) | 0.0285 (2) |
| O2 | 0.74038 (13) | 0.88719 (14) | 0.74267 (14) | 0.0495 (3) |
| O3 | 0.45998 (13) | 1.04716 (11) | 0.72367 (10) | 0.0352 (2) |
| O4 | 0.56749 (14) | 0.80767 (14) | 0.61969 (10) | 0.0425 (3) |

| | | | | |
|-----|-------------|-------------|-------------|--------------|
| S1 | 0.57433 (4) | 0.89340 (4) | 0.72262 (3) | 0.02752 (10) |
| H1A | 0.745 (3) | 0.700 (2) | 0.516 (2) | 0.050 (5)* |
| H1B | 0.730 (2) | 0.7873 (18) | 0.3356 (15) | 0.025 (4)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|---------------|---------------|
| C1 | 0.0187 (6) | 0.0249 (6) | 0.0285 (6) | −0.0058 (4) | −0.0015 (5) | 0.0019 (5) |
| C2 | 0.0327 (7) | 0.0331 (7) | 0.0270 (6) | 0.0054 (6) | 0.0074 (5) | 0.0086 (5) |
| C3 | 0.0644 (11) | 0.0464 (9) | 0.0167 (6) | 0.0215 (8) | 0.0011 (6) | −0.0030 (6) |
| C4 | 0.0617 (10) | 0.0393 (8) | 0.0359 (8) | 0.0188 (7) | −0.0286 (7) | −0.0221 (6) |
| C5 | 0.0290 (6) | 0.0228 (6) | 0.0368 (7) | 0.0021 (5) | −0.0114 (5) | −0.0145 (5) |
| C6 | 0.0203 (6) | 0.0402 (8) | 0.0531 (9) | −0.0038 (6) | −0.0086 (6) | 0.0050 (7) |
| C7 | 0.0354 (7) | 0.0303 (7) | 0.0484 (8) | −0.0133 (6) | −0.0121 (6) | −0.0068 (6) |
| C8 | 0.0323 (8) | 0.0330 (8) | 0.0957 (15) | −0.0088 (6) | −0.0148 (8) | −0.0230 (8) |
| C9 | 0.0393 (8) | 0.0223 (6) | 0.0381 (7) | 0.0055 (5) | −0.0090 (6) | −0.0112 (5) |
| C10 | 0.0286 (7) | 0.0293 (6) | 0.0315 (7) | −0.0007 (5) | −0.0078 (5) | −0.0068 (5) |
| F1 | 0.0280 (4) | 0.0431 (5) | 0.0489 (5) | −0.0120 (4) | 0.0008 (4) | −0.0084 (4) |
| F2 | 0.0473 (6) | 0.0309 (5) | 0.0588 (6) | 0.0034 (4) | −0.0127 (4) | 0.0043 (4) |
| F3 | 0.0693 (7) | 0.0580 (6) | 0.0319 (5) | −0.0098 (5) | −0.0128 (4) | −0.0148 (4) |
| N1 | 0.0177 (5) | 0.0180 (4) | 0.0160 (4) | −0.0009 (4) | −0.0013 (3) | −0.0031 (3) |
| O1 | 0.0368 (5) | 0.0252 (4) | 0.0151 (4) | 0.0002 (4) | 0.0018 (4) | −0.0004 (3) |
| O2 | 0.0206 (5) | 0.0412 (6) | 0.0850 (9) | −0.0062 (4) | −0.0104 (5) | −0.0052 (6) |
| O3 | 0.0290 (5) | 0.0264 (5) | 0.0432 (6) | 0.0058 (4) | −0.0055 (4) | −0.0057 (4) |
| O4 | 0.0435 (6) | 0.0459 (6) | 0.0332 (5) | −0.0042 (5) | 0.0055 (4) | −0.0172 (5) |
| S1 | 0.01852 (16) | 0.02389 (17) | 0.03577 (18) | 0.00056 (11) | −0.00008 (12) | −0.00683 (12) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|--------|-------------|--------|-------------|
| C1—C6 | 1.5247 (18) | C7—H7A | 0.9800 |
| C1—C2 | 1.5251 (18) | C7—H7B | 0.9800 |
| C1—C7 | 1.5279 (19) | C7—H7C | 0.9800 |
| C1—N1 | 1.5362 (15) | C8—H8A | 0.9800 |
| C2—C3 | 1.514 (2) | C8—H8B | 0.9800 |
| C2—H2A | 0.9900 | C8—H8C | 0.9800 |
| C2—H2B | 0.9900 | C9—H9A | 0.9800 |
| C3—C4 | 1.516 (3) | C9—H9B | 0.9800 |
| C3—H3A | 0.9900 | C9—H9C | 0.9800 |
| C3—H3B | 0.9900 | C10—F3 | 1.3262 (16) |
| C4—C5 | 1.528 (2) | C10—F1 | 1.3314 (16) |
| C4—H4A | 0.9900 | C10—F2 | 1.3323 (16) |
| C4—H4B | 0.9900 | C10—S1 | 1.8202 (15) |
| C5—C8 | 1.522 (2) | N1—O1 | 1.4168 (12) |
| C5—C9 | 1.5244 (17) | N1—H1B | 0.875 (16) |
| C5—N1 | 1.5354 (15) | O1—H1A | 0.84 (2) |
| C6—H6A | 0.9800 | O2—S1 | 1.4260 (11) |
| C6—H6B | 0.9800 | O3—S1 | 1.4406 (9) |
| C6—H6C | 0.9800 | O4—S1 | 1.4486 (11) |

| | | | |
|-------------|--------------|--------------|-------------|
| C6—C1—C2 | 112.20 (11) | C1—C7—H7B | 109.5 |
| C6—C1—C7 | 110.16 (12) | H7A—C7—H7B | 109.5 |
| C2—C1—C7 | 110.70 (11) | C1—C7—H7C | 109.5 |
| C6—C1—N1 | 111.77 (10) | H7A—C7—H7C | 109.5 |
| C2—C1—N1 | 106.75 (11) | H7B—C7—H7C | 109.5 |
| C7—C1—N1 | 104.98 (10) | C5—C8—H8A | 109.5 |
| C3—C2—C1 | 113.88 (12) | C5—C8—H8B | 109.5 |
| C3—C2—H2A | 108.8 | H8A—C8—H8B | 109.5 |
| C1—C2—H2A | 108.8 | C5—C8—H8C | 109.5 |
| C3—C2—H2B | 108.8 | H8A—C8—H8C | 109.5 |
| C1—C2—H2B | 108.8 | H8B—C8—H8C | 109.5 |
| H2A—C2—H2B | 107.7 | C5—C9—H9A | 109.5 |
| C2—C3—C4 | 109.99 (11) | C5—C9—H9B | 109.5 |
| C2—C3—H3A | 109.7 | H9A—C9—H9B | 109.5 |
| C4—C3—H3A | 109.7 | C5—C9—H9C | 109.5 |
| C2—C3—H3B | 109.7 | H9A—C9—H9C | 109.5 |
| C4—C3—H3B | 109.7 | H9B—C9—H9C | 109.5 |
| H3A—C3—H3B | 108.2 | F3—C10—F1 | 107.06 (11) |
| C3—C4—C5 | 113.93 (13) | F3—C10—F2 | 108.19 (12) |
| C3—C4—H4A | 108.8 | F1—C10—F2 | 107.48 (11) |
| C5—C4—H4A | 108.8 | F3—C10—S1 | 111.63 (10) |
| C3—C4—H4B | 108.8 | F1—C10—S1 | 111.42 (9) |
| C5—C4—H4B | 108.8 | F2—C10—S1 | 110.87 (10) |
| H4A—C4—H4B | 107.7 | O1—N1—C5 | 108.32 (9) |
| C8—C5—C9 | 109.71 (12) | O1—N1—C1 | 109.24 (9) |
| C8—C5—C4 | 111.57 (14) | C5—N1—C1 | 120.28 (9) |
| C9—C5—C4 | 112.76 (11) | O1—N1—H1B | 108.1 (10) |
| C8—C5—N1 | 105.18 (11) | C5—N1—H1B | 105.5 (10) |
| C9—C5—N1 | 111.54 (10) | C1—N1—H1B | 104.8 (10) |
| C4—C5—N1 | 105.78 (11) | N1—O1—H1A | 107.7 (13) |
| C1—C6—H6A | 109.5 | O2—S1—O3 | 115.62 (7) |
| C1—C6—H6B | 109.5 | O2—S1—O4 | 115.69 (7) |
| H6A—C6—H6B | 109.5 | O3—S1—O4 | 113.73 (7) |
| C1—C6—H6C | 109.5 | O2—S1—C10 | 103.77 (7) |
| H6A—C6—H6C | 109.5 | O3—S1—C10 | 103.23 (6) |
| H6B—C6—H6C | 109.5 | O4—S1—C10 | 102.32 (7) |
| C1—C7—H7A | 109.5 | | |
| C6—C1—C2—C3 | 71.46 (16) | C2—C1—N1—O1 | 176.56 (9) |
| C7—C1—C2—C3 | −165.02 (13) | C7—C1—N1—O1 | −65.88 (12) |
| N1—C1—C2—C3 | −51.30 (15) | C6—C1—N1—C5 | −72.58 (14) |
| C1—C2—C3—C4 | 58.13 (16) | C2—C1—N1—C5 | 50.45 (13) |
| C2—C3—C4—C5 | −59.31 (16) | C7—C1—N1—C5 | 168.01 (10) |
| C3—C4—C5—C8 | 166.79 (12) | F3—C10—S1—O2 | 65.11 (11) |
| C3—C4—C5—C9 | −69.21 (15) | F1—C10—S1—O2 | −175.25 (9) |
| C3—C4—C5—N1 | 52.95 (14) | F2—C10—S1—O2 | −55.59 (12) |
| C8—C5—N1—O1 | 64.27 (13) | F3—C10—S1—O3 | −55.88 (11) |

| | | | |
|-------------|--------------|--------------|--------------|
| C9—C5—N1—O1 | −54.59 (13) | F1—C10—S1—O3 | 63.76 (10) |
| C4—C5—N1—O1 | −177.53 (10) | F2—C10—S1—O3 | −176.58 (10) |
| C8—C5—N1—C1 | −169.19 (12) | F3—C10—S1—O4 | −174.21 (10) |
| C9—C5—N1—C1 | 71.95 (14) | F1—C10—S1—O4 | −54.57 (11) |
| C4—C5—N1—C1 | −50.99 (14) | F2—C10—S1—O4 | 65.09 (11) |
| C6—C1—N1—O1 | 53.53 (13) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---|-------------|---------------------|----------------------------|-------------------------------|
| O1—H1 <i>A</i> \cdots O4 | 0.84 (2) | 1.78 (2) | 2.6163 (14) | 177 (2) |
| N1—H1 <i>B</i> \cdots O3 ⁱ | 0.875 (16) | 1.991 (16) | 2.8385 (14) | 163.0 (14) |

Symmetry code: (i) $-x+1, -y+2, -z+1$.