# organic compounds

# Methyl 4-hydroxy-2-methoxycarbonyl-methyl-1,1-dioxo-1,2-dihydro-1$\lambda^6$,2-benzothiazine-3-carboxylate[1]

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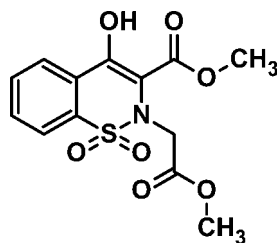Correspondence e-mail: mnachemist@hotmail.com

There are two independent molecules in the asymmetric unit of the title compound, $C_{13}H_{13}NO_7S$, which have almost identical geometries. The thiazine ring adopts a sofa conformation in both molecules and the molecular conformations are stabilized by intramolecular O—H···O hydrogen bonds. Intermolecular C—H···O hydrogen bonds stabilize the crystal packing.

## Related literature

For related structures, see; Arshad *et al.* (2009, 2010). For the synthesis, see; Arshad *et al.* (2011).



## Experimental

### Crystal data

| | |
|---|---|
| $C_{13}H_{13}NO_7S$ | Triclinic, $P\bar{1}$ |
| $M_r$ = 327.30 | $a$ = 8.9128 (14) Å |
| $b$ = 12.414 (2) Å | $Z$ = 4 |
| $c$ = 13.443 (2) Å | Mo $K\alpha$ radiation |
| $\alpha$ = 79.784 (2)° | $\mu$ = 0.27 mm$^{-1}$ |
| $\beta$ = 72.981 (3)° | $T$ = 100 K |
| $\gamma$ = 88.503 (3)° | 0.48 × 0.36 × 0.33 mm |
| $V$ = 1399.2 (4) Å$^3$ | |

### Data collection

| | |
|---|---|
| Siemens SMART diffractometer equipped with a Bruker APEXII detector | 17331 measured reflections |
| | 6923 independent reflections |
| | 6322 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2001) | $R_{int}$ = 0.018 |
| $T_{min}$ = 0.882, $T_{max}$ = 0.917 | |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.040 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.107 | |
| $S$ = 1.06 | |
| 6923 reflections | $\Delta\rho_{max}$ = 0.48 e Å$^{-3}$ |
| 407 parameters | $\Delta\rho_{min}$ = −0.42 e Å$^{-3}$ |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1$O$···O4 | 0.79 (3) | 1.87 (3) | 2.5754 (18) | 149 (3) |
| O8—H8$O$···O11 | 0.98 (3) | 1.68 (3) | 2.5623 (17) | 148 (3) |
| C13—H13$A$···O10$^i$ | 0.98 | 2.44 | 3.202 (2) | 134 |
| C2—H2···O11$^{ii}$ | 0.95 | 2.59 | 3.339 (2) | 136 |

Symmetry codes: (i) $-x, -y + 2, -z + 1$; (ii) $-x, -y + 1, -z + 1$.

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *X-SEED* (Barbour, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

## References

Arshad, M. N., Khan, I. U., Zia-ur-Rehman, M. & Shafiq, M. (2011). *Asian J. Chem.* **23**, 2801–2805.
Arshad, M. N., Zia-ur-Rehman, M. & Khan, I. U. (2009). *Acta Cryst.* E**65**, o3077.
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Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2001). *SADABS, APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# Methyl 4-hydroxy-2-methoxycarbonylmethyl-1,1-dioxo-1,2-dihydro-1$\lambda^6$,2-benzothiazine-3-carboxylate

**Muhammad Nadeem Arshad, Islam Ullah Khan, Muhammad Zia-ur-Rehman, Sheikh Asrar Ahmad and H. M. Rafique**

### S1. Comment

The crystal structure of title compound is being reported in continuation of our structural studies of various benzothiazines (Arshad *et al.*, 2009, 2010).

The title compound crystallizes with two independent molecules in the asymmetric unit (Fig. 1). In molecule A, the two fused rings are oriented at dihedral angle of 14.18 (7)° while in B this dihedral angle amounts to 18.40 (6)°. The root mean square deviation values for thiazine ring in both molecules are 0.1991Å and 0.2119Å respectively. The alkylated groups attached to the nitrogen atom in molecule A and B are inclined at almost perpendicular position with respect to the thiazine rings and given values are 85.81 (5)° and 88.12 (5)°. Both of the molecules are involved in intra and intermolecular hydrogen bondings. The intramolecular hydrogen bonding of O—H···O type forms six membered ring motif in each molecule. Other inter molecular C—H···O and O—H···O type hydrogen bonding interactions produce three dimensional network (Fig. 2 and Tab. 1).

### S2. Experimental

The synthesis of title compound is already reported (Arshad *et al.*, 2011). The compound was recrystallized under slow evaporation technique in ethylacetate.

### S3. Refinement

The C-H H-atoms were positioned with idealized geometry with C—H = 0.95 Å for aromatic, C—H = 0.99 Å for methylene & C—H = 0.98 Å for methyl groups and were refined using a riding model with $U_{iso}$(H) = 1.2 $U_{eq}$(C) for aromatic & methylene and $U_{iso}$(H) = 1.5 $U_{eq}$(C) for methyl groups.

The O—H H atoms were located in a difference map and refined isotropically.
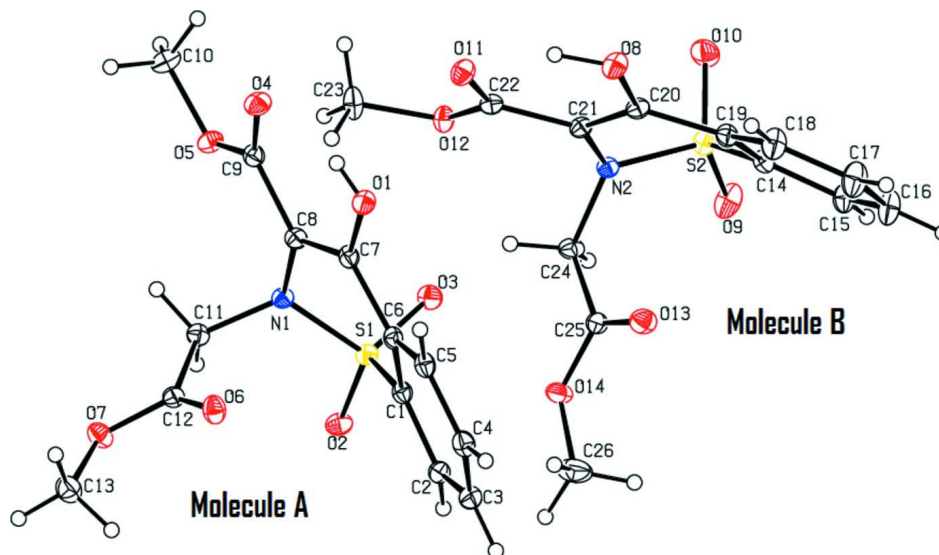
**Figure 1**

The *ORTEP* diagram of the title compound with displacement ellipsoids drawn at the 30% probability level.
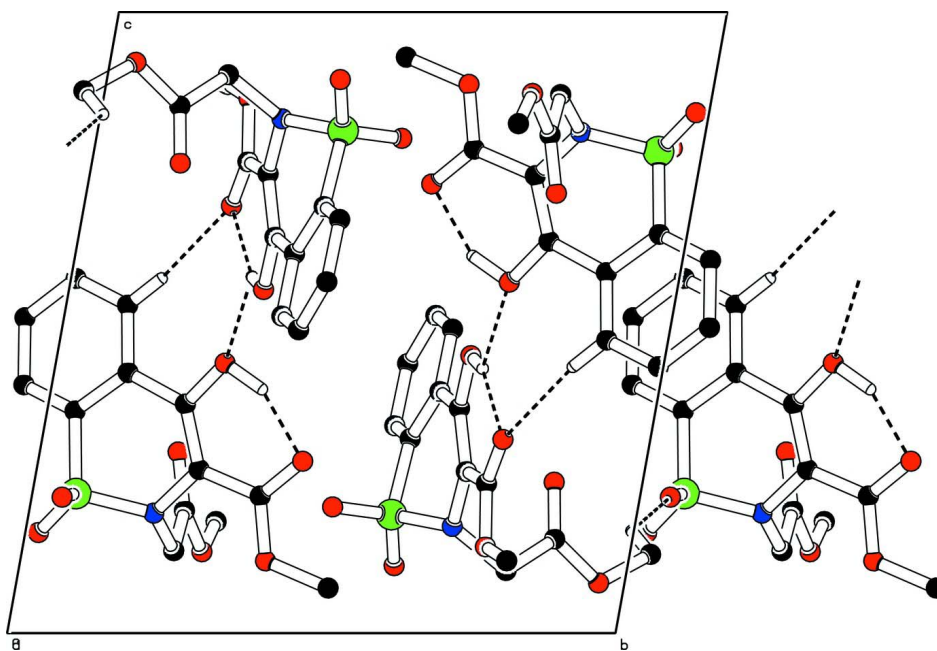


**Figure 2**

Unit cell packing of the title compound showing hydrogen bonding interaction as dashed lines.

**Methyl 4-hydroxy-2-methoxycarbonylmethyl-1,1-dioxo-1,2-dihydro- 1λ⁶,2-benzothiazine-3-carboxylate**

*Crystal data*

C₁₃H₁₃NO₇S

$M_r = 327.30$

Triclinic, $P\overline{1}$

Hall symbol: -P 1

$a = 8.9128$ (14) Å

$b = 12.414$ (2) Å

$c = 13.443$ (2) Å

$\alpha = 79.784$ (2)°

$\beta = 72.981$ (3)°

$\gamma = 88.503$ (3)°

$V = 1399.2$ (4) Å³

$Z = 4$

$F(000) = 680$
$D_x = 1.554$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9730 reflections
$\theta = 2.4$–28.9°

$\mu = 0.27$ mm$^{-1}$
$T = 100$ K
Blocks, colorless
$0.48 \times 0.36 \times 0.33$ mm

*Data collection*

Siemens SMART
   diffractometer equipped with a Bruker APEXII
   detector
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan
   (*SADABS*; Bruker, 2001)
$T_{min} = 0.882$, $T_{max} = 0.917$

17331 measured reflections
6923 independent reflections
6322 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 28.9°$, $\theta_{min} = 2.1°$
$h = -12 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -18 \rightarrow 17$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.107$
$S = 1.06$
6923 reflections
407 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0516P)^2 + 0.9585P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å$^{-3}$
$\Delta\rho_{min} = -0.42$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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$^2$ > 2sigma(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  | 0.16821 (4)  | 0.59653 (3)  | 0.19082 (3)  | 0.01849 (9)  |
| S2  | 0.52552 (5)  | 0.92829 (3)  | 0.77528 (3)  | 0.02196 (10) |
| O8  | 0.48743 (15) | 0.72159 (10) | 0.56519 (9)  | 0.0263 (3)   |
| O1  | 0.30206 (15) | 0.68027 (10) | 0.44663 (9)  | 0.0240 (2)   |
| O12 | 0.38864 (14) | 0.59665 (9)  | 0.88368 (9)  | 0.0225 (2)   |
| O4  | 0.56249 (14) | 0.75774 (10) | 0.31183 (9)  | 0.0267 (3)   |
| O7  | 0.12303 (14) | 0.95735 (10) | 0.08091 (9)  | 0.0269 (3)   |
| O11 | 0.39383 (14) | 0.56505 (10) | 0.72314 (9)  | 0.0259 (2)   |
| O14 | 0.89610 (14) | 0.70395 (10) | 0.86890 (10) | 0.0276 (3)   |
| O3  | 0.25344 (14) | 0.49769 (9)  | 0.20325 (9)  | 0.0245 (2)   |

| | | | |
|---|---|---|---|
| N1 | 0.29039 (15) | 0.70033 (10) | 0.17185 (10) | 0.0181 (2) |
| O13 | 0.86686 (14) | 0.77263 (11) | 0.70832 (10) | 0.0286 (3) |
| O2 | 0.09291 (14) | 0.61619 (10) | 0.10870 (9) | 0.0236 (2) |
| O5 | 0.59329 (13) | 0.76066 (10) | 0.13910 (9) | 0.0229 (2) |
| N2 | 0.53834 (15) | 0.79462 (11) | 0.80675 (10) | 0.0179 (2) |
| O9 | 0.59840 (17) | 0.97841 (11) | 0.83798 (10) | 0.0343 (3) |
| O6 | 0.06605 (15) | 0.85459 (10) | 0.24309 (9) | 0.0275 (3) |
| O10 | 0.36468 (15) | 0.94991 (11) | 0.78104 (11) | 0.0342 (3) |
| C19 | 0.61858 (19) | 0.87842 (13) | 0.58043 (12) | 0.0211 (3) |
| C11 | 0.27512 (18) | 0.80160 (13) | 0.10098 (12) | 0.0202 (3) |
| H11A | 0.2567 | 0.7827 | 0.0369 | 0.024* |
| H11B | 0.3751 | 0.8446 | 0.0785 | 0.024* |
| C3 | −0.2273 (2) | 0.57856 (14) | 0.43419 (13) | 0.0249 (3) |
| H3 | −0.3349 | 0.5583 | 0.4499 | 0.030* |
| C14 | 0.63731 (19) | 0.95418 (13) | 0.64225 (12) | 0.0203 (3) |
| C9 | 0.50907 (19) | 0.74369 (12) | 0.24092 (12) | 0.0201 (3) |
| C8 | 0.34682 (18) | 0.70707 (12) | 0.26034 (12) | 0.0189 (3) |
| C20 | 0.53123 (18) | 0.77482 (13) | 0.63114 (12) | 0.0200 (3) |
| C6 | 0.08927 (18) | 0.64003 (12) | 0.38905 (12) | 0.0188 (3) |
| C1 | 0.03184 (18) | 0.60509 (12) | 0.31319 (12) | 0.0187 (3) |
| C2 | −0.12533 (19) | 0.57587 (13) | 0.33394 (13) | 0.0215 (3) |
| H2 | −0.1625 | 0.5545 | 0.2807 | 0.026* |
| C15 | 0.7366 (2) | 1.04599 (13) | 0.60003 (14) | 0.0269 (3) |
| H15 | 0.7499 | 1.0952 | 0.6436 | 0.032* |
| C5 | −0.01566 (19) | 0.64217 (12) | 0.48944 (12) | 0.0216 (3) |
| H5 | 0.0204 | 0.6651 | 0.5424 | 0.026* |
| C22 | 0.42446 (18) | 0.62469 (13) | 0.77922 (12) | 0.0204 (3) |
| C24 | 0.64439 (19) | 0.75062 (14) | 0.86710 (12) | 0.0229 (3) |
| H24A | 0.6064 | 0.6760 | 0.9049 | 0.027* |
| H24B | 0.6399 | 0.7964 | 0.9211 | 0.027* |
| C21 | 0.50090 (17) | 0.73286 (12) | 0.73654 (12) | 0.0184 (3) |
| C18 | 0.6976 (2) | 0.89909 (15) | 0.47193 (13) | 0.0302 (4) |
| H18 | 0.6846 | 0.8501 | 0.4280 | 0.036* |
| C12 | 0.14236 (18) | 0.87217 (13) | 0.15144 (12) | 0.0207 (3) |
| C25 | 0.81345 (18) | 0.74493 (13) | 0.80252 (13) | 0.0209 (3) |
| C4 | −0.1721 (2) | 0.61080 (13) | 0.51154 (13) | 0.0246 (3) |
| H4 | −0.2422 | 0.6113 | 0.5800 | 0.029* |
| C23 | 0.3242 (2) | 0.48585 (14) | 0.92729 (14) | 0.0303 (4) |
| H23A | 0.2267 | 0.4773 | 0.9093 | 0.045* |
| H23B | 0.3021 | 0.4727 | 1.0043 | 0.045* |
| H23C | 0.4002 | 0.4330 | 0.8976 | 0.045* |
| C10 | 0.7541 (2) | 0.79979 (16) | 0.11653 (14) | 0.0290 (4) |
| H10A | 0.7569 | 0.8799 | 0.1072 | 0.043* |
| H10B | 0.8190 | 0.7768 | 0.0517 | 0.043* |
| H10C | 0.7949 | 0.7690 | 0.1755 | 0.043* |
| C7 | 0.25286 (19) | 0.67701 (12) | 0.36224 (12) | 0.0195 (3) |
| C17 | 0.7950 (3) | 0.99176 (17) | 0.42903 (14) | 0.0366 (4) |
| H17 | 0.8479 | 1.0059 | 0.3554 | 0.044* |

| C13 | 0.0082 (2) | 1.03446 (15) | 0.12689 (15) | 0.0309 (4) |
|---|---|---|---|---|
| H13A | −0.0919 | 0.9955 | 0.1652 | 0.046* |
| H13B | −0.0070 | 1.0915 | 0.0705 | 0.046* |
| H13C | 0.0464 | 1.0681 | 0.1759 | 0.046* |
| C16 | 0.8164 (3) | 1.06409 (15) | 0.49221 (15) | 0.0348 (4) |
| H16 | 0.8855 | 1.1260 | 0.4618 | 0.042* |
| C26 | 1.0610 (2) | 0.6897 (2) | 0.8203 (2) | 0.0437 (5) |
| H26A | 1.0733 | 0.6389 | 0.7705 | 0.066* |
| H26B | 1.1107 | 0.6600 | 0.8750 | 0.066* |
| H26C | 1.1108 | 0.7606 | 0.7823 | 0.066* |
| H1O | 0.389 (4) | 0.703 (3) | 0.425 (2) | 0.066* |
| H8O | 0.437 (4) | 0.653 (3) | 0.608 (2) | 0.066* |

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

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.01975 (18) | 0.01908 (18) | 0.01976 (18) | 0.00083 (13) | −0.00782 (14) | −0.00819 (13) |
| S2 | 0.0236 (2) | 0.02065 (19) | 0.02086 (19) | 0.00131 (14) | −0.00283 (14) | −0.00805 (14) |
| O8 | 0.0324 (6) | 0.0306 (6) | 0.0196 (5) | −0.0077 (5) | −0.0116 (5) | −0.0059 (5) |
| O1 | 0.0298 (6) | 0.0259 (6) | 0.0195 (5) | −0.0054 (5) | −0.0127 (5) | −0.0018 (4) |
| O12 | 0.0272 (6) | 0.0190 (5) | 0.0187 (5) | −0.0014 (4) | −0.0031 (4) | −0.0024 (4) |
| O4 | 0.0269 (6) | 0.0318 (6) | 0.0258 (6) | −0.0035 (5) | −0.0129 (5) | −0.0069 (5) |
| O7 | 0.0287 (6) | 0.0288 (6) | 0.0216 (5) | 0.0065 (5) | −0.0076 (5) | −0.0012 (5) |
| O11 | 0.0279 (6) | 0.0255 (6) | 0.0253 (6) | −0.0050 (5) | −0.0079 (5) | −0.0062 (5) |
| O14 | 0.0231 (6) | 0.0296 (6) | 0.0337 (6) | 0.0044 (5) | −0.0146 (5) | −0.0047 (5) |
| O3 | 0.0268 (6) | 0.0202 (5) | 0.0302 (6) | 0.0040 (4) | −0.0102 (5) | −0.0111 (5) |
| N1 | 0.0196 (6) | 0.0197 (6) | 0.0178 (6) | −0.0005 (5) | −0.0083 (5) | −0.0055 (5) |
| O13 | 0.0232 (6) | 0.0343 (7) | 0.0254 (6) | 0.0027 (5) | −0.0037 (5) | −0.0036 (5) |
| O2 | 0.0236 (6) | 0.0301 (6) | 0.0216 (5) | −0.0004 (4) | −0.0102 (4) | −0.0097 (5) |
| O5 | 0.0193 (5) | 0.0272 (6) | 0.0239 (5) | −0.0013 (4) | −0.0064 (4) | −0.0088 (4) |
| N2 | 0.0194 (6) | 0.0204 (6) | 0.0160 (6) | 0.0000 (5) | −0.0075 (5) | −0.0048 (5) |
| O9 | 0.0453 (8) | 0.0330 (7) | 0.0241 (6) | −0.0119 (6) | −0.0030 (5) | −0.0142 (5) |
| O6 | 0.0313 (6) | 0.0231 (6) | 0.0235 (6) | 0.0025 (5) | −0.0023 (5) | −0.0025 (4) |
| O10 | 0.0265 (6) | 0.0325 (7) | 0.0385 (7) | 0.0104 (5) | −0.0024 (5) | −0.0063 (6) |
| C19 | 0.0250 (8) | 0.0216 (7) | 0.0185 (7) | −0.0011 (6) | −0.0098 (6) | −0.0021 (6) |
| C11 | 0.0222 (7) | 0.0225 (7) | 0.0167 (7) | 0.0009 (6) | −0.0068 (6) | −0.0039 (5) |
| C3 | 0.0215 (8) | 0.0227 (8) | 0.0288 (8) | 0.0004 (6) | −0.0061 (6) | −0.0019 (6) |
| C14 | 0.0233 (7) | 0.0194 (7) | 0.0182 (7) | 0.0026 (6) | −0.0065 (6) | −0.0031 (5) |
| C9 | 0.0230 (7) | 0.0169 (7) | 0.0227 (7) | 0.0013 (5) | −0.0091 (6) | −0.0054 (5) |
| C8 | 0.0222 (7) | 0.0186 (7) | 0.0196 (7) | 0.0008 (5) | −0.0099 (6) | −0.0064 (5) |
| C20 | 0.0200 (7) | 0.0233 (7) | 0.0194 (7) | −0.0008 (6) | −0.0081 (6) | −0.0063 (6) |
| C6 | 0.0236 (7) | 0.0143 (6) | 0.0195 (7) | 0.0002 (5) | −0.0079 (6) | −0.0028 (5) |
| C1 | 0.0226 (7) | 0.0155 (6) | 0.0189 (7) | 0.0012 (5) | −0.0068 (6) | −0.0044 (5) |
| C2 | 0.0228 (7) | 0.0187 (7) | 0.0251 (7) | 0.0000 (6) | −0.0097 (6) | −0.0043 (6) |
| C15 | 0.0349 (9) | 0.0193 (8) | 0.0265 (8) | −0.0023 (6) | −0.0085 (7) | −0.0045 (6) |
| C5 | 0.0293 (8) | 0.0178 (7) | 0.0183 (7) | 0.0008 (6) | −0.0080 (6) | −0.0028 (5) |
| C22 | 0.0167 (7) | 0.0230 (7) | 0.0216 (7) | 0.0013 (5) | −0.0053 (6) | −0.0050 (6) |
| C24 | 0.0210 (7) | 0.0327 (8) | 0.0167 (7) | 0.0006 (6) | −0.0090 (6) | −0.0031 (6) |

| C21 | 0.0190 (7) | 0.0205 (7) | 0.0176 (7) | −0.0013 (5) | −0.0068 (5) | −0.0056 (5) |
| C18 | 0.0431 (10) | 0.0297 (9) | 0.0182 (7) | −0.0068 (7) | −0.0091 (7) | −0.0037 (6) |
| C12 | 0.0211 (7) | 0.0199 (7) | 0.0235 (7) | −0.0006 (6) | −0.0102 (6) | −0.0041 (6) |
| C25 | 0.0210 (7) | 0.0185 (7) | 0.0249 (7) | 0.0009 (5) | −0.0090 (6) | −0.0045 (6) |
| C4 | 0.0269 (8) | 0.0220 (8) | 0.0219 (7) | 0.0033 (6) | −0.0038 (6) | −0.0022 (6) |
| C23 | 0.0421 (10) | 0.0170 (8) | 0.0257 (8) | −0.0006 (7) | −0.0024 (7) | −0.0003 (6) |
| C10 | 0.0202 (8) | 0.0378 (10) | 0.0303 (9) | −0.0047 (7) | −0.0060 (6) | −0.0111 (7) |
| C7 | 0.0259 (8) | 0.0154 (7) | 0.0205 (7) | −0.0001 (5) | −0.0110 (6) | −0.0041 (5) |
| C17 | 0.0505 (12) | 0.0348 (10) | 0.0198 (8) | −0.0098 (8) | −0.0043 (8) | −0.0015 (7) |
| C13 | 0.0314 (9) | 0.0281 (9) | 0.0316 (9) | 0.0054 (7) | −0.0085 (7) | −0.0035 (7) |
| C16 | 0.0465 (11) | 0.0255 (9) | 0.0272 (9) | −0.0110 (8) | −0.0048 (8) | 0.0004 (7) |
| C26 | 0.0275 (10) | 0.0493 (12) | 0.0578 (13) | 0.0141 (9) | −0.0168 (9) | −0.0134 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O2 | 1.4347 (12) | C14—C15 | 1.391 (2) |
| S1—O3 | 1.4365 (12) | C9—O4 | 1.2226 (19) |
| S1—N1 | 1.6449 (13) | C9—C8 | 1.462 (2) |
| S1—C1 | 1.7548 (16) | C8—C7 | 1.371 (2) |
| S2—O9 | 1.4308 (13) | C20—O8 | 1.3413 (18) |
| S2—O10 | 1.4337 (14) | C20—C21 | 1.369 (2) |
| S2—N2 | 1.6477 (14) | C6—C1 | 1.401 (2) |
| S2—C14 | 1.7504 (16) | C6—C5 | 1.403 (2) |
| O8—C20 | 1.3413 (18) | C6—C7 | 1.461 (2) |
| O8—H8O | 0.98 (3) | C1—C2 | 1.391 (2) |
| O1—C7 | 1.3377 (18) | C2—H2 | 0.9500 |
| O1—H1O | 0.79 (3) | C15—C16 | 1.395 (2) |
| O12—C22 | 1.3285 (19) | C15—H15 | 0.9500 |
| O12—C23 | 1.457 (2) | C5—C4 | 1.389 (2) |
| O4—C9 | 1.2226 (19) | C5—H5 | 0.9500 |
| O7—C12 | 1.3330 (19) | C22—C21 | 1.464 (2) |
| O7—C13 | 1.461 (2) | C24—C25 | 1.510 (2) |
| O11—C22 | 1.2329 (19) | C24—H24A | 0.9900 |
| O14—C25 | 1.3423 (19) | C24—H24B | 0.9900 |
| O14—C26 | 1.443 (2) | C18—C17 | 1.391 (3) |
| N1—C8 | 1.4362 (18) | C18—H18 | 0.9500 |
| N1—C11 | 1.4658 (19) | C4—H4 | 0.9500 |
| O13—C25 | 1.204 (2) | C23—H23A | 0.9800 |
| O5—C9 | 1.3364 (19) | C23—H23B | 0.9800 |
| O5—C10 | 1.4535 (19) | C23—H23C | 0.9800 |
| N2—C21 | 1.4298 (18) | C10—H10A | 0.9800 |
| N2—C24 | 1.4543 (19) | C10—H10B | 0.9800 |
| O6—C12 | 1.204 (2) | C10—H10C | 0.9800 |
| C19—C18 | 1.402 (2) | C17—C16 | 1.392 (3) |
| C19—C14 | 1.403 (2) | C17—H17 | 0.9500 |
| C19—C20 | 1.468 (2) | C13—H13A | 0.9800 |
| C11—C12 | 1.521 (2) | C13—H13B | 0.9800 |
| C11—H11A | 0.9900 | C13—H13C | 0.9800 |

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|---|---|---|---|
| C11—H11B | 0.9900 | C16—H16 | 0.9500 |
| C3—C4 | 1.393 (2) | C26—H26A | 0.9800 |
| C3—C2 | 1.393 (2) | C26—H26B | 0.9800 |
| C3—H3 | 0.9500 | C26—H26C | 0.9800 |
| | | | |
| O2—S1—O3 | 118.97 (7) | C14—C15—H15 | 120.8 |
| O2—S1—N1 | 107.65 (7) | C16—C15—H15 | 120.8 |
| O3—S1—N1 | 107.63 (7) | C4—C5—C6 | 120.16 (15) |
| O2—S1—C1 | 110.07 (7) | C4—C5—H5 | 119.9 |
| O3—S1—C1 | 108.17 (7) | C6—C5—H5 | 119.9 |
| N1—S1—C1 | 103.18 (7) | O11—C22—O12 | 122.69 (14) |
| O9—S2—O10 | 119.43 (9) | O11—C22—C21 | 122.86 (14) |
| O9—S2—N2 | 107.81 (8) | O12—C22—C21 | 114.45 (13) |
| O10—S2—N2 | 107.01 (7) | N2—C24—C25 | 114.97 (13) |
| O9—S2—C14 | 110.62 (8) | N2—C24—H24A | 108.5 |
| O10—S2—C14 | 108.43 (8) | C25—C24—H24A | 108.5 |
| N2—S2—C14 | 102.09 (7) | N2—C24—H24B | 108.5 |
| C20—O8—H8O | 106.3 (17) | C25—C24—H24B | 108.5 |
| C7—O1—H1O | 107 (2) | H24A—C24—H24B | 107.5 |
| C22—O12—C23 | 115.47 (13) | C20—C21—N2 | 120.96 (13) |
| C12—O7—C13 | 113.54 (13) | C20—C21—C22 | 119.85 (13) |
| C25—O14—C26 | 115.71 (15) | N2—C21—C22 | 119.16 (13) |
| C8—N1—C11 | 118.88 (12) | C17—C18—C19 | 119.55 (16) |
| C8—N1—S1 | 114.54 (10) | C17—C18—H18 | 120.2 |
| C11—N1—S1 | 119.24 (10) | C19—C18—H18 | 120.2 |
| C9—O5—C10 | 115.65 (12) | O6—C12—O7 | 124.32 (15) |
| C21—N2—C24 | 119.88 (13) | O6—C12—C11 | 124.73 (14) |
| C21—N2—S2 | 114.97 (10) | O7—C12—C11 | 110.94 (13) |
| C24—N2—S2 | 119.48 (11) | O13—C25—O14 | 125.30 (15) |
| C18—C19—C14 | 118.56 (15) | O13—C25—C24 | 126.78 (14) |
| C18—C19—C20 | 121.16 (14) | O14—C25—C24 | 107.92 (13) |
| C14—C19—C20 | 119.98 (14) | C5—C4—C3 | 120.59 (15) |
| N1—C11—C12 | 113.26 (13) | C5—C4—H4 | 119.7 |
| N1—C11—H11A | 108.9 | C3—C4—H4 | 119.7 |
| C12—C11—H11A | 108.9 | O12—C23—H23A | 109.5 |
| N1—C11—H11B | 108.9 | O12—C23—H23B | 109.5 |
| C12—C11—H11B | 108.9 | H23A—C23—H23B | 109.5 |
| H11A—C11—H11B | 107.7 | O12—C23—H23C | 109.5 |
| C4—C3—C2 | 120.26 (15) | H23A—C23—H23C | 109.5 |
| C4—C3—H3 | 119.9 | H23B—C23—H23C | 109.5 |
| C2—C3—H3 | 119.9 | O5—C10—H10A | 109.5 |
| C15—C14—C19 | 122.05 (15) | O5—C10—H10B | 109.5 |
| C15—C14—S2 | 121.66 (12) | H10A—C10—H10B | 109.5 |
| C19—C14—S2 | 116.28 (12) | O5—C10—H10C | 109.5 |
| O4—C9—O5 | 123.11 (14) | H10A—C10—H10C | 109.5 |
| O4—C9—O5 | 123.11 (14) | H10B—C10—H10C | 109.5 |
| O4—C9—C8 | 122.76 (14) | O1—C7—C8 | 123.00 (14) |
| O4—C9—C8 | 122.76 (14) | O1—C7—C6 | 113.63 (13) |

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| O5—C9—C8 | 114.13 (13) | C8—C7—C6 | 123.36 (14) |
| C7—C8—N1 | 121.26 (14) | C18—C17—C16 | 121.10 (17) |
| C7—C8—C9 | 119.61 (13) | C18—C17—H17 | 119.4 |
| N1—C8—C9 | 119.10 (13) | C16—C17—H17 | 119.4 |
| O8—C20—C21 | 121.98 (14) | O7—C13—H13A | 109.5 |
| O8—C20—C21 | 121.98 (14) | O7—C13—H13B | 109.5 |
| O8—C20—C19 | 114.68 (13) | H13A—C13—H13B | 109.5 |
| O8—C20—C19 | 114.68 (13) | O7—C13—H13C | 109.5 |
| C21—C20—C19 | 123.29 (14) | H13A—C13—H13C | 109.5 |
| C1—C6—C5 | 118.23 (14) | H13B—C13—H13C | 109.5 |
| C1—C6—C7 | 120.43 (14) | C17—C16—C15 | 120.20 (17) |
| C5—C6—C7 | 121.30 (14) | C17—C16—H16 | 119.9 |
| C2—C1—C6 | 121.94 (14) | C15—C16—H16 | 119.9 |
| C2—C1—S1 | 120.99 (12) | O14—C26—H26A | 109.5 |
| C6—C1—S1 | 117.05 (12) | O14—C26—H26B | 109.5 |
| C1—C2—C3 | 118.78 (15) | H26A—C26—H26B | 109.5 |
| C1—C2—H2 | 120.6 | O14—C26—H26C | 109.5 |
| C3—C2—H2 | 120.6 | H26A—C26—H26C | 109.5 |
| C14—C15—C16 | 118.48 (16) | H26B—C26—H26C | 109.5 |
| | | | |
| O2—S1—N1—C8 | 165.62 (10) | N1—S1—C1—C2 | 145.82 (13) |
| O3—S1—N1—C8 | −65.00 (12) | O2—S1—C1—C6 | −150.26 (12) |
| C1—S1—N1—C8 | 49.24 (12) | O3—S1—C1—C6 | 78.24 (13) |
| O2—S1—N1—C11 | 15.84 (13) | N1—S1—C1—C6 | −35.61 (13) |
| O3—S1—N1—C11 | 145.22 (11) | C6—C1—C2—C3 | −1.9 (2) |
| C1—S1—N1—C11 | −100.54 (12) | S1—C1—C2—C3 | 176.63 (12) |
| O9—S2—N2—C21 | −167.55 (11) | C4—C3—C2—C1 | 0.5 (2) |
| O10—S2—N2—C21 | 62.81 (12) | C19—C14—C15—C16 | 1.7 (3) |
| C14—S2—N2—C21 | −50.99 (12) | S2—C14—C15—C16 | −176.97 (15) |
| O9—S2—N2—C24 | −14.11 (14) | C1—C6—C5—C4 | −0.4 (2) |
| O10—S2—N2—C24 | −143.74 (12) | C7—C6—C5—C4 | 177.35 (14) |
| C14—S2—N2—C24 | 102.45 (12) | C23—O12—C22—O11 | 5.7 (2) |
| C8—N1—C11—C12 | −70.27 (17) | C23—O12—C22—C21 | −175.20 (14) |
| S1—N1—C11—C12 | 78.20 (15) | C21—N2—C24—C25 | 69.53 (18) |
| C18—C19—C14—C15 | −2.8 (3) | S2—N2—C24—C25 | −82.61 (16) |
| C20—C19—C14—C15 | 170.92 (15) | O8—C20—C21—N2 | −175.35 (14) |
| C18—C19—C14—S2 | 175.91 (13) | O8—C20—C21—N2 | −175.35 (14) |
| C20—C19—C14—S2 | −10.3 (2) | C19—C20—C21—N2 | 7.1 (2) |
| O9—S2—C14—C15 | −25.92 (17) | O8—C20—C21—C22 | 2.7 (2) |
| O10—S2—C14—C15 | 106.82 (15) | O8—C20—C21—C22 | 2.7 (2) |
| N2—S2—C14—C15 | −140.43 (14) | C19—C20—C21—C22 | −174.91 (14) |
| O9—S2—C14—C19 | 155.31 (13) | C24—N2—C21—C20 | −121.58 (16) |
| O10—S2—C14—C19 | −71.94 (14) | S2—N2—C21—C20 | 31.75 (19) |
| N2—S2—C14—C19 | 40.81 (14) | C24—N2—C21—C22 | 60.39 (19) |
| O4—O4—C9—O5 | 0.00 (13) | S2—N2—C21—C22 | −146.28 (12) |
| O4—O4—C9—C8 | 0.00 (6) | O11—C22—C21—C20 | 4.7 (2) |
| C10—O5—C9—O4 | −1.3 (2) | O12—C22—C21—C20 | −174.38 (14) |
| C10—O5—C9—O4 | −1.3 (2) | O11—C22—C21—N2 | −177.28 (14) |

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| C10—O5—C9—C8 | 178.51 (14) | O12—C22—C21—N2 | 3.7 (2) |
| C11—N1—C8—C7 | 114.97 (16) | C14—C19—C18—C17 | 1.7 (3) |
| S1—N1—C8—C7 | −34.92 (18) | C20—C19—C18—C17 | −171.98 (18) |
| C11—N1—C8—C9 | −66.85 (18) | C13—O7—C12—O6 | 5.0 (2) |
| S1—N1—C8—C9 | 143.26 (12) | C13—O7—C12—C11 | −174.02 (13) |
| O4—C9—C8—C7 | −6.3 (2) | N1—C11—C12—O6 | 7.2 (2) |
| O4—C9—C8—C7 | −6.3 (2) | N1—C11—C12—O7 | −173.69 (12) |
| O5—C9—C8—C7 | 173.89 (13) | C26—O14—C25—O13 | −2.1 (2) |
| O4—C9—C8—N1 | 175.49 (14) | C26—O14—C25—C24 | 178.36 (15) |
| O4—C9—C8—N1 | 175.49 (14) | N2—C24—C25—O13 | −0.1 (2) |
| O5—C9—C8—N1 | −4.3 (2) | N2—C24—C25—O14 | 179.43 (13) |
| O8—O8—C20—C21 | 0.00 (15) | C6—C5—C4—C3 | −1.0 (2) |
| O8—O8—C20—C19 | 0.00 (19) | C2—C3—C4—C5 | 1.0 (2) |
| C18—C19—C20—O8 | −22.0 (2) | N1—C8—C7—O1 | 178.77 (13) |
| C14—C19—C20—O8 | 164.35 (14) | C9—C8—C7—O1 | 0.6 (2) |
| C18—C19—C20—O8 | −22.0 (2) | N1—C8—C7—C6 | −2.2 (2) |
| C14—C19—C20—O8 | 164.35 (14) | C9—C8—C7—C6 | 179.64 (14) |
| C18—C19—C20—C21 | 155.70 (17) | C1—C6—C7—O1 | −163.40 (14) |
| C14—C19—C20—C21 | −17.9 (2) | C5—C6—C7—O1 | 18.9 (2) |
| C5—C6—C1—C2 | 1.8 (2) | C1—C6—C7—C8 | 17.5 (2) |
| C7—C6—C1—C2 | −175.91 (14) | C5—C6—C7—C8 | −160.19 (15) |
| C5—C6—C1—S1 | −176.74 (11) | C19—C18—C17—C16 | 0.5 (3) |
| C7—C6—C1—S1 | 5.53 (19) | C18—C17—C16—C15 | −1.6 (3) |
| O2—S1—C1—C2 | 31.17 (15) | C14—C15—C16—C17 | 0.5 (3) |
| O3—S1—C1—C2 | −100.33 (14) | | |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| O1—H1O···O4 | 0.79 (3) | 1.87 (3) | 2.5754 (18) | 149 (3) |
| O8—H8O···O11 | 0.98 (3) | 1.68 (3) | 2.5623 (17) | 148 (3) |
| C13—H13A···O10[i] | 0.98 | 2.44 | 3.202 (2) | 134 |
| C2—H2···O11[ii] | 0.95 | 2.59 | 3.339 (2) | 136 |

Symmetry codes: (i) −x, −y+2, −z+1; (ii) −x, −y+1, −z+1.