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# 2-[2-(2-Bromophenyl)-2-oxoethyl]-1$\lambda^6$,2-benzothiazole-1,1,3-trione

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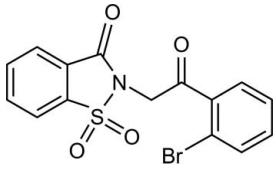
Key indicators: single-crystal X-ray study; $T$ = 123 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.050; $wR$ factor = 0.105; data-to-parameter ratio = 16.5.

The asymmetric unit of the title compound, $C_{15}H_{10}BrNO_4S$, contains two different conformers in which the benzisothiazole rings are essentially planar, with r.m.s. deviations of 0.012 and 0.017 Å. The mean planes of the benzene rings form dihedral angles 70.49 (13) and 72.79 (11)° with the benzisothiazole rings. The orientation of the Br atoms in the two conformers exhibit the most pronounced difference, with opposing orientations in the two molecules. The crystal structure is stabilized by $\pi$–$\pi$ interactions between the benzene rings of the benzisothiazole moieties of one molecule and bromobenzene rings of the other molecule, with distances between the ring centroids of 3.599 (3) and 3.620 (3) Å, respectively. The crystal packing is further consolidated by pairs of weak intermolecular C—H···O hydrogen bonds, which form inversion dimers.

## Related literature

For non-steroidal anti-inflammatory drugs (NSAIDs) and related compounds, see: Lombardino *et al.* (1971); Soler (1985); Carty *et al.* (1993); Turck *et al.* (1995); Blackham & Owen (1975); Singh *et al.* (2007); Vaccarino *et al.* (2007); Kapui *et al.* (2003). For related structures, see: Maliha *et al.* (2007); Siddiqui *et al.* (2007).




## Experimental

### Crystal data

$C_{15}H_{10}BrNO_4S$
$M_r$ = 380.21
Triclinic, $P\overline{1}$
$a$ = 7.574 (2) Å
$b$ = 13.903 (4) Å
$c$ = 14.814 (4) Å
$\alpha$ = 110.574 (15)°
$\beta$ = 96.936 (13)°
$\gamma$ = 93.640 (14)°
$V$ = 1440.3 (7) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 3.02 mm$^{-1}$
$T$ = 123 K
0.18 × 0.18 × 0.16 mm

### Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min}$ = 0.613, $T_{max}$ = 0.644
12284 measured reflections
6541 independent reflections
5268 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.105
$S$ = 1.12
6541 reflections
397 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.81 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.04 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C14—H14···O1$^{i}$ | 0.95 | 2.40 | 3.305 (5) | 159 |
| C17—H17···O5$^{ii}$ | 0.95 | 2.43 | 3.225 (5) | 141 |
| C27—H27···O7$^{iii}$ | 0.95 | 2.29 | 3.164 (5) | 153 |

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2412).

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# supporting information

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## 2-[2-(2-Bromophenyl)-2-oxoethyl]-1$\lambda^6$,2-benzothiazole-1,1,3-trione

**Nazia Sattar, Hamid Latif Siddiqui, Waseeq Ahmad Siddiqui, Muhammad Akram and Masood Parvez**

### S1. Comment

Oxicam, a class of non-steroidal anti-inflammatory drugs (NSAIDs) consists of 1,2-benzothiazine 1,1-dioxide derivatives which are found to be potent anti-inflammatory and analgesic agents, *e.g.*, Piroxicam (Lombardino *et al.*, 1971), Droxicam (Soler, 1985), Ampiroxicam (Carty *et al.*, 1993), Meloxicam (Turck *et al.*, 1995) and Sudoxicam (Blackham & Owen, 1975) are the recent members of this class currently in use in the international market. Various derivatives are known to be cyclooxygenase-2 (COX-2) inhibitors (Singh *et al.*, 2007), analgesic (Vaccarino *et al.*, 2007) and human leucocyte elastase (HLE) inhibitors (Kapui *et al.*, 2003). Earlier, we have reported the synthesis and crystal structures of some of the 1,2-benzothiazine derivatives (Maliha *et al.*, 2007; Siddiqui *et al.*, 2007). Herein, we report the synthesis and crystal structure of the title compound that has served as a precursor for the 1,2-benzothiazine derivative.

The asymmetric unit of the title compound contains two conformers (Fig. 1). In both molecules, the benzisothiazol rings S1/N1/C1–C7 and S2/N2/C16–C22 are essentially planar with rms deviations of fitted atoms being 0.017 Å and 0.012 Å, respectively, while the mean-planes of the benzene rings C10–C15 and C25–C30 form dihedral angles 70.49 (13)° and 72.79 (11)°, respectively, with the mean-planes of the benzisothiazol rings. The orientation of the Br atoms in the two conformers exhibit the most pronounced difference, with opposing orientations in the two molecules. The crystal structure is stabilized by $\pi$-$\pi$ interactions between benzene rings (C1–C6) of benzisothiazol moieties in one molecule and bromobenzene rings (C25–C30) in the other molecule with distances between the ring centroids being 3.599 (3) Å and 3.620 (3) Å, respectively. The crystal packing is further consolidated by weak intermolecular C—H···O hydrogen bonds. The molecule containing S1 forms centrosymmetric dimers *via* C14—H14···O1 hydrogen bonding interactions. The other molecule also forms centrosymmetric dimers *via* C17—H17···O5 hydrogen bonds; the dimers are further extended along the *b*-axis *via* C27—H27···O7 hydrogen bonds (Fig. 2 and Tab. 1).

The bond distances and angles in the title compound (Fig. 1) agree very well with the corresponding bond distances and angles reported in closely related compounds (Maliha *et al.*, 2007; Siddiqui *et al.*, 2007).

### S2. Experimental

A mixture of 2-bromo-1-(2-bromophenyl)ethanone (2.0 g, 7.2 mmol) and sodium saccharin (1.76 g, 8.6 mmol) in dimethylformamide (15 ml) was stirred at 383 K for 3 h under anhydrous conditions. On completion of the reaction (as indicated by TLC), the contents of the flask were poured into crushed ice. The precipitates formed were filtered, washed with water, ice-cold ethanol and dried to give the reddish brown title product (1.94 g, 71%). Crystals were grown by slow evaporation of a solution in EtOAc and $CHCl_3$ (1:1) at room temperature; m.p. 395–397 K.

### S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 and 0.99 Å, for aryl and methylene H-atoms, respectively. The $U_{iso}$(H) were allowed at 1.2$U_{eq}$(C).

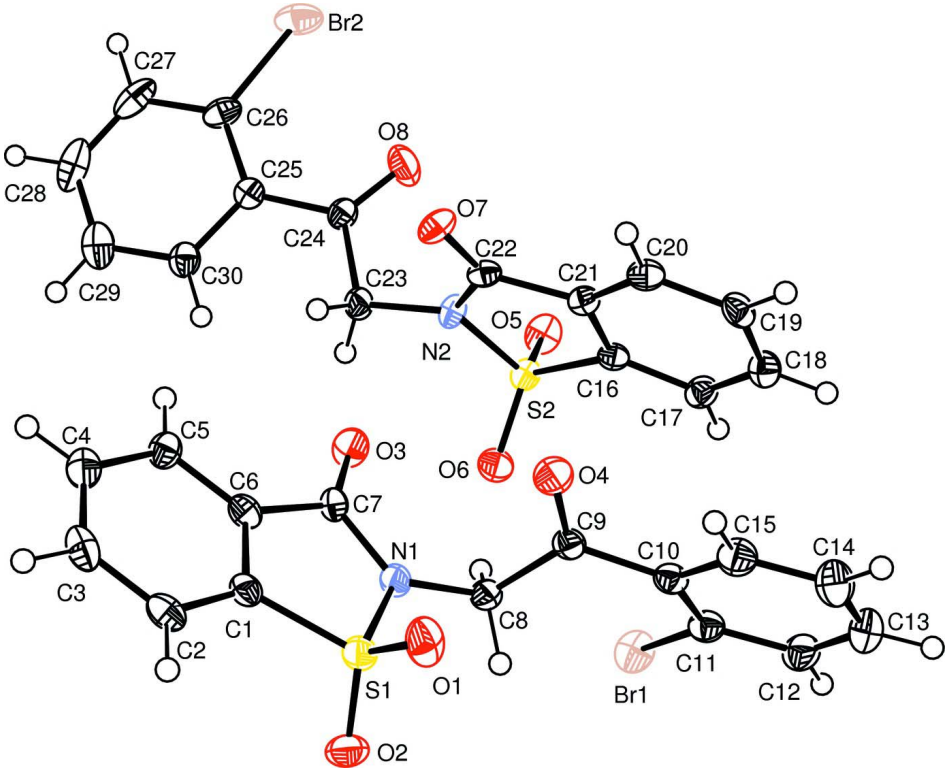


**Figure 1**

The asymmetric unit of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

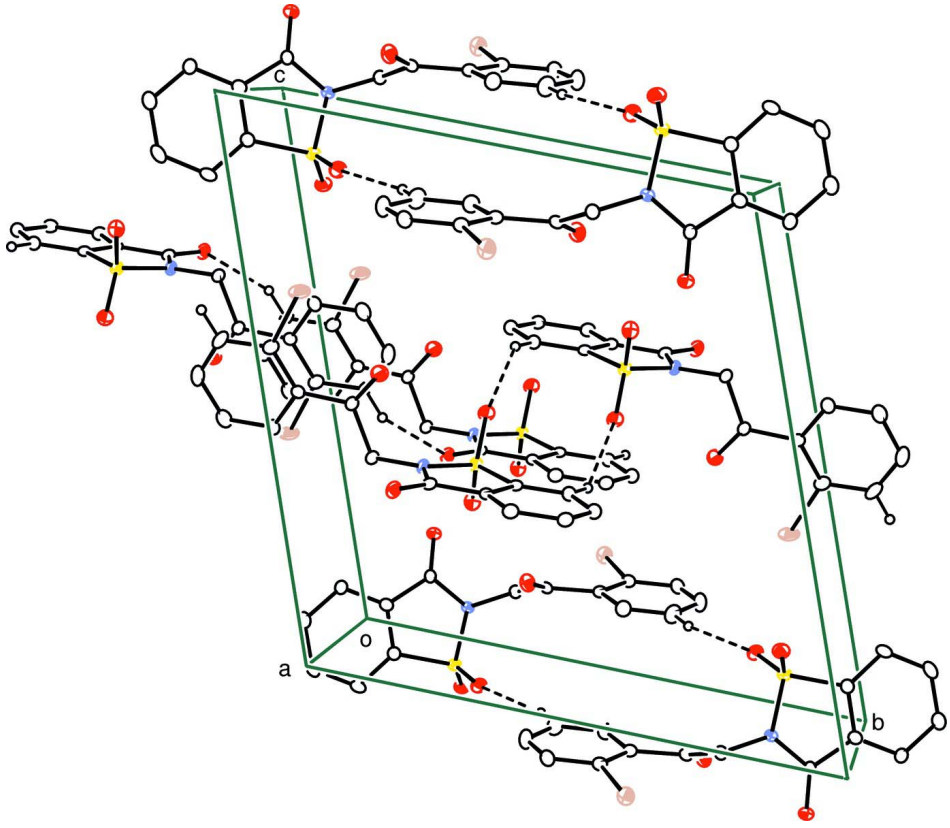


**Figure 2**

A view of the C—H···O hydrogen bonds (dotted lines) in the crystal structure of the title compound. H atoms not participating in hydrogen-bonding were omitted to enhance clarity.

### 2-[2-(2-Bromophenyl)-2-oxoethyl]-1$\lambda^6$,2-benzothiazole-1,1,3-trione

*Crystal data*

$C_{15}H_{10}BrNO_4S$
$M_r$ = 380.21
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a$ = 7.574 (2) Å
$b$ = 13.903 (4) Å
$c$ = 14.814 (4) Å
$\alpha$ = 110.574 (15)°
$\beta$ = 96.936 (13)°
$\gamma$ = 93.640 (14)°
$V$ = 1440.3 (7) Å$^3$
$Z$ = 4
$F(000)$ = 760
$D_x$ = 1.753 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 6425 reflections
$\theta$ = 1.0–27.5°
$\mu$ = 3.02 mm$^{-1}$
$T$ = 123 K
Prism, colorless
0.18 × 0.18 × 0.16 mm

*Data collection*

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{min}$ = 0.613, $T_{max}$ = 0.644
12284 measured reflections
6541 independent reflections
5268 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.5°
$h$ = −9→9
$k$ = −18→18
$l$ = −19→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.105$
$S = 1.12$
6541 reflections
397 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + 5.440P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.81$ e Å$^{-3}$
$\Delta\rho_{min} = -1.04$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 0.36505 (6) | 0.45967 (4) | 0.15933 (3) | 0.03241 (12) |
| Br2 | 0.91717 (7) | 0.09057 (4) | 0.66368 (3) | 0.03844 (13) |
| S1 | 0.67803 (14) | 0.13040 (8) | −0.09125 (7) | 0.0234 (2) |
| S2 | 0.60738 (13) | 0.34637 (7) | 0.39090 (7) | 0.02000 (19) |
| O1 | 0.8217 (4) | 0.1917 (2) | −0.1062 (2) | 0.0349 (7) |
| O2 | 0.5086 (4) | 0.1188 (2) | −0.1509 (2) | 0.0321 (7) |
| O3 | 0.6920 (4) | 0.1327 (2) | 0.1629 (2) | 0.0283 (6) |
| O4 | 0.9038 (4) | 0.3378 (2) | 0.1237 (2) | 0.0310 (7) |
| O5 | 0.5563 (4) | 0.3780 (2) | 0.4859 (2) | 0.0283 (6) |
| O6 | 0.4665 (4) | 0.3119 (2) | 0.3092 (2) | 0.0283 (6) |
| O7 | 1.0338 (4) | 0.2269 (2) | 0.3508 (2) | 0.0260 (6) |
| O8 | 0.8207 (5) | 0.2096 (2) | 0.5384 (2) | 0.0389 (8) |
| N1 | 0.6521 (5) | 0.1750 (2) | 0.0257 (2) | 0.0215 (7) |
| N2 | 0.7478 (4) | 0.2569 (2) | 0.3791 (2) | 0.0207 (7) |
| C1 | 0.7402 (5) | 0.0120 (3) | −0.0868 (3) | 0.0209 (8) |
| C2 | 0.7806 (5) | −0.0711 (3) | −0.1628 (3) | 0.0250 (8) |
| H2 | 0.7750 | −0.0703 | −0.2270 | 0.030* |
| C3 | 0.8297 (5) | −0.1558 (3) | −0.1406 (3) | 0.0277 (9) |
| H3 | 0.8576 | −0.2148 | −0.1908 | 0.033* |
| C4 | 0.8388 (5) | −0.1557 (3) | −0.0463 (3) | 0.0261 (9) |
| H4 | 0.8734 | −0.2146 | −0.0332 | 0.031* |
| C5 | 0.7983 (5) | −0.0711 (3) | 0.0290 (3) | 0.0230 (8) |
| H5 | 0.8054 | −0.0711 | 0.0934 | 0.028* |
| C6 | 0.7473 (5) | 0.0133 (3) | 0.0077 (3) | 0.0208 (8) |
| C7 | 0.6977 (5) | 0.1109 (3) | 0.0771 (3) | 0.0198 (8) |

| | | | | |
|---|---|---|---|---|
| C8 | 0.5961 (5) | 0.2769 (3) | 0.0709 (3) | 0.0213 (8) |
| H8A | 0.5346 | 0.2778 | 0.1264 | 0.026* |
| H8B | 0.5100 | 0.2916 | 0.0229 | 0.026* |
| C9 | 0.7555 (5) | 0.3606 (3) | 0.1071 (3) | 0.0205 (8) |
| C10 | 0.7307 (5) | 0.4707 (3) | 0.1184 (3) | 0.0215 (8) |
| C11 | 0.5795 (5) | 0.5213 (3) | 0.1368 (3) | 0.0234 (8) |
| C12 | 0.5781 (6) | 0.6246 (3) | 0.1462 (3) | 0.0305 (9) |
| H12 | 0.4728 | 0.6577 | 0.1583 | 0.037* |
| C13 | 0.7319 (7) | 0.6784 (4) | 0.1378 (4) | 0.0384 (11) |
| H13 | 0.7330 | 0.7492 | 0.1453 | 0.046* |
| C14 | 0.8831 (7) | 0.6296 (4) | 0.1187 (4) | 0.0386 (11) |
| H14 | 0.9878 | 0.6663 | 0.1118 | 0.046* |
| C15 | 0.8825 (6) | 0.5280 (3) | 0.1095 (3) | 0.0290 (9) |
| H15 | 0.9880 | 0.4953 | 0.0967 | 0.035* |
| C16 | 0.7715 (5) | 0.4356 (3) | 0.3809 (3) | 0.0181 (7) |
| C17 | 0.7503 (5) | 0.5350 (3) | 0.3834 (3) | 0.0229 (8) |
| H17 | 0.6394 | 0.5629 | 0.3918 | 0.028* |
| C18 | 0.8992 (5) | 0.5917 (3) | 0.3730 (3) | 0.0249 (8) |
| H18 | 0.8906 | 0.6597 | 0.3735 | 0.030* |
| C19 | 1.0615 (5) | 0.5497 (3) | 0.3619 (3) | 0.0232 (8) |
| H19 | 1.1622 | 0.5905 | 0.3564 | 0.028* |
| C20 | 1.0793 (5) | 0.4497 (3) | 0.3586 (3) | 0.0226 (8) |
| H20 | 1.1896 | 0.4213 | 0.3501 | 0.027* |
| C21 | 0.9302 (5) | 0.3926 (3) | 0.3681 (3) | 0.0181 (7) |
| C22 | 0.9183 (5) | 0.2841 (3) | 0.3645 (3) | 0.0197 (8) |
| C23 | 0.6895 (6) | 0.1524 (3) | 0.3720 (3) | 0.0224 (8) |
| H23A | 0.7401 | 0.1020 | 0.3186 | 0.027* |
| H23B | 0.5574 | 0.1393 | 0.3561 | 0.027* |
| C24 | 0.7505 (5) | 0.1370 (3) | 0.4683 (3) | 0.0204 (8) |
| C25 | 0.7189 (5) | 0.0296 (3) | 0.4674 (3) | 0.0196 (8) |
| C26 | 0.7853 (5) | −0.0009 (3) | 0.5443 (3) | 0.0255 (9) |
| C27 | 0.7537 (6) | −0.1035 (4) | 0.5369 (4) | 0.0343 (11) |
| H27 | 0.8041 | −0.1240 | 0.5883 | 0.041* |
| C28 | 0.6490 (7) | −0.1757 (4) | 0.4549 (4) | 0.0383 (12) |
| H28 | 0.6255 | −0.2452 | 0.4508 | 0.046* |
| C29 | 0.5792 (6) | −0.1468 (3) | 0.3795 (3) | 0.0335 (10) |
| H29 | 0.5064 | −0.1961 | 0.3236 | 0.040* |
| C30 | 0.6150 (5) | −0.0461 (3) | 0.3851 (3) | 0.0243 (8) |
| H30 | 0.5682 | −0.0275 | 0.3319 | 0.029* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Br1 | 0.0246 (2) | 0.0352 (2) | 0.0418 (3) | 0.01061 (18) | 0.01351 (19) | 0.0153 (2) |
| Br2 | 0.0388 (3) | 0.0569 (3) | 0.0219 (2) | 0.0144 (2) | 0.00103 (19) | 0.0167 (2) |
| S1 | 0.0299 (5) | 0.0228 (5) | 0.0171 (4) | 0.0017 (4) | 0.0034 (4) | 0.0071 (4) |
| S2 | 0.0177 (4) | 0.0206 (5) | 0.0245 (5) | 0.0040 (4) | 0.0070 (4) | 0.0101 (4) |
| O1 | 0.048 (2) | 0.0283 (16) | 0.0300 (16) | −0.0069 (14) | 0.0119 (15) | 0.0128 (14) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O2 | 0.0371 (18) | 0.0359 (17) | 0.0229 (15) | 0.0102 (14) | −0.0017 (13) | 0.0111 (13) |
| O3 | 0.0403 (18) | 0.0268 (15) | 0.0198 (14) | 0.0082 (13) | 0.0104 (13) | 0.0084 (12) |
| O4 | 0.0248 (16) | 0.0288 (16) | 0.0371 (17) | 0.0087 (13) | 0.0015 (13) | 0.0092 (14) |
| O5 | 0.0278 (16) | 0.0325 (16) | 0.0313 (16) | 0.0069 (13) | 0.0172 (13) | 0.0151 (13) |
| O6 | 0.0202 (14) | 0.0290 (16) | 0.0341 (16) | 0.0009 (12) | 0.0004 (12) | 0.0111 (13) |
| O7 | 0.0233 (15) | 0.0285 (15) | 0.0328 (16) | 0.0122 (12) | 0.0074 (12) | 0.0166 (13) |
| O8 | 0.059 (2) | 0.0251 (16) | 0.0254 (16) | −0.0119 (15) | −0.0055 (15) | 0.0068 (13) |
| N1 | 0.0300 (18) | 0.0185 (16) | 0.0148 (15) | 0.0028 (14) | 0.0034 (14) | 0.0044 (13) |
| N2 | 0.0239 (17) | 0.0175 (16) | 0.0249 (17) | 0.0046 (13) | 0.0076 (14) | 0.0110 (14) |
| C1 | 0.0184 (19) | 0.0199 (19) | 0.0218 (19) | −0.0026 (15) | −0.0003 (15) | 0.0061 (16) |
| C2 | 0.025 (2) | 0.028 (2) | 0.0169 (18) | −0.0004 (17) | 0.0037 (16) | 0.0019 (16) |
| C3 | 0.023 (2) | 0.021 (2) | 0.031 (2) | −0.0006 (16) | 0.0070 (17) | −0.0009 (17) |
| C4 | 0.0187 (19) | 0.022 (2) | 0.035 (2) | 0.0026 (16) | 0.0054 (17) | 0.0076 (18) |
| C5 | 0.023 (2) | 0.0214 (19) | 0.027 (2) | 0.0023 (16) | 0.0058 (16) | 0.0105 (17) |
| C6 | 0.0180 (19) | 0.0217 (19) | 0.0202 (18) | −0.0023 (15) | 0.0034 (15) | 0.0052 (16) |
| C7 | 0.0206 (19) | 0.0157 (18) | 0.0222 (19) | −0.0009 (14) | 0.0029 (15) | 0.0065 (15) |
| C8 | 0.0207 (19) | 0.0203 (19) | 0.0217 (19) | 0.0044 (15) | 0.0010 (15) | 0.0067 (16) |
| C9 | 0.024 (2) | 0.0204 (19) | 0.0158 (17) | 0.0051 (15) | 0.0028 (15) | 0.0039 (15) |
| C10 | 0.022 (2) | 0.024 (2) | 0.0174 (18) | 0.0013 (16) | 0.0015 (15) | 0.0072 (16) |
| C11 | 0.023 (2) | 0.027 (2) | 0.0204 (19) | 0.0049 (16) | 0.0032 (16) | 0.0079 (16) |
| C12 | 0.036 (2) | 0.026 (2) | 0.028 (2) | 0.0116 (19) | 0.0030 (19) | 0.0074 (18) |
| C13 | 0.046 (3) | 0.023 (2) | 0.045 (3) | 0.004 (2) | 0.001 (2) | 0.012 (2) |
| C14 | 0.036 (3) | 0.034 (3) | 0.047 (3) | −0.008 (2) | 0.003 (2) | 0.019 (2) |
| C15 | 0.023 (2) | 0.031 (2) | 0.033 (2) | 0.0036 (18) | 0.0031 (18) | 0.0122 (19) |
| C16 | 0.0201 (19) | 0.0215 (19) | 0.0129 (16) | 0.0022 (15) | 0.0024 (14) | 0.0064 (15) |
| C17 | 0.024 (2) | 0.023 (2) | 0.0229 (19) | 0.0068 (16) | 0.0046 (16) | 0.0084 (16) |
| C18 | 0.027 (2) | 0.022 (2) | 0.026 (2) | 0.0015 (16) | 0.0060 (17) | 0.0087 (17) |
| C19 | 0.022 (2) | 0.024 (2) | 0.0216 (19) | −0.0064 (16) | 0.0027 (16) | 0.0085 (16) |
| C20 | 0.0170 (19) | 0.029 (2) | 0.0229 (19) | 0.0040 (16) | 0.0035 (15) | 0.0101 (17) |
| C21 | 0.0193 (18) | 0.0208 (18) | 0.0149 (17) | 0.0044 (15) | 0.0045 (14) | 0.0066 (15) |
| C22 | 0.0201 (19) | 0.025 (2) | 0.0159 (17) | 0.0047 (15) | 0.0016 (15) | 0.0093 (15) |
| C23 | 0.029 (2) | 0.0188 (19) | 0.0212 (19) | −0.0011 (16) | 0.0027 (16) | 0.0097 (16) |
| C24 | 0.0211 (19) | 0.0204 (19) | 0.0224 (19) | 0.0027 (15) | 0.0052 (15) | 0.0102 (16) |
| C25 | 0.0195 (19) | 0.0214 (19) | 0.0191 (18) | 0.0043 (15) | 0.0069 (15) | 0.0070 (15) |
| C26 | 0.022 (2) | 0.035 (2) | 0.026 (2) | 0.0116 (17) | 0.0077 (16) | 0.0154 (18) |
| C27 | 0.038 (3) | 0.042 (3) | 0.043 (3) | 0.022 (2) | 0.021 (2) | 0.032 (2) |
| C28 | 0.047 (3) | 0.026 (2) | 0.056 (3) | 0.015 (2) | 0.033 (3) | 0.022 (2) |
| C29 | 0.037 (3) | 0.025 (2) | 0.038 (3) | 0.0022 (19) | 0.018 (2) | 0.007 (2) |
| C30 | 0.023 (2) | 0.022 (2) | 0.027 (2) | 0.0052 (16) | 0.0077 (17) | 0.0073 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Br1—C11 | 1.908 (4) | C10—C15 | 1.403 (6) |
| Br2—C26 | 1.890 (4) | C11—C12 | 1.394 (6) |
| S1—O1 | 1.426 (3) | C12—C13 | 1.387 (7) |
| S1—O2 | 1.433 (3) | C12—H12 | 0.9500 |
| S1—N1 | 1.664 (3) | C13—C14 | 1.377 (7) |
| S1—C1 | 1.762 (4) | C13—H13 | 0.9500 |

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| S2—O5 | 1.429 (3) | C14—C15 | 1.370 (6) |
| S2—O6 | 1.431 (3) | C14—H14 | 0.9500 |
| S2—N2 | 1.664 (3) | C15—H15 | 0.9500 |
| S2—C16 | 1.757 (4) | C16—C21 | 1.380 (5) |
| O3—C7 | 1.206 (5) | C16—C17 | 1.389 (5) |
| O4—C9 | 1.206 (5) | C17—C18 | 1.390 (5) |
| O7—C22 | 1.207 (4) | C17—H17 | 0.9500 |
| O8—C24 | 1.200 (5) | C18—C19 | 1.397 (6) |
| N1—C7 | 1.395 (5) | C18—H18 | 0.9500 |
| N1—C8 | 1.453 (5) | C19—C20 | 1.390 (5) |
| N2—C22 | 1.386 (5) | C19—H19 | 0.9500 |
| N2—C23 | 1.455 (5) | C20—C21 | 1.388 (5) |
| C1—C2 | 1.382 (5) | C20—H20 | 0.9500 |
| C1—C6 | 1.387 (5) | C21—C22 | 1.487 (5) |
| C2—C3 | 1.390 (6) | C23—C24 | 1.537 (5) |
| C2—H2 | 0.9500 | C23—H23A | 0.9900 |
| C3—C4 | 1.389 (6) | C23—H23B | 0.9900 |
| C3—H3 | 0.9500 | C24—C25 | 1.493 (5) |
| C4—C5 | 1.389 (6) | C25—C26 | 1.400 (5) |
| C4—H4 | 0.9500 | C25—C30 | 1.405 (6) |
| C5—C6 | 1.383 (5) | C26—C27 | 1.394 (6) |
| C5—H5 | 0.9500 | C27—C28 | 1.387 (7) |
| C6—C7 | 1.489 (5) | C27—H27 | 0.9500 |
| C8—C9 | 1.525 (5) | C28—C29 | 1.373 (7) |
| C8—H8A | 0.9900 | C28—H28 | 0.9500 |
| C8—H8B | 0.9900 | C29—C30 | 1.381 (6) |
| C9—C10 | 1.507 (5) | C29—H29 | 0.9500 |
| C10—C11 | 1.387 (5) | C30—H30 | 0.9500 |

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|---|---|---|---|
| O1—S1—O2 | 116.92 (19) | C14—C13—C12 | 120.2 (4) |
| O1—S1—N1 | 109.81 (18) | C14—C13—H13 | 119.9 |
| O2—S1—N1 | 109.41 (18) | C12—C13—H13 | 119.9 |
| O1—S1—C1 | 112.50 (19) | C15—C14—C13 | 119.9 (4) |
| O2—S1—C1 | 112.67 (18) | C15—C14—H14 | 120.1 |
| N1—S1—C1 | 92.85 (17) | C13—C14—H14 | 120.1 |
| O5—S2—O6 | 117.20 (18) | C14—C15—C10 | 122.0 (4) |
| O5—S2—N2 | 109.94 (17) | C14—C15—H15 | 119.0 |
| O6—S2—N2 | 109.76 (18) | C10—C15—H15 | 119.0 |
| O5—S2—C16 | 112.41 (18) | C21—C16—C17 | 123.2 (3) |
| O6—S2—C16 | 112.30 (17) | C21—C16—S2 | 110.1 (3) |
| N2—S2—C16 | 92.44 (17) | C17—C16—S2 | 126.7 (3) |
| C7—N1—C8 | 123.3 (3) | C16—C17—C18 | 116.7 (4) |
| C7—N1—S1 | 115.4 (3) | C16—C17—H17 | 121.7 |
| C8—N1—S1 | 121.2 (3) | C18—C17—H17 | 121.7 |
| C22—N2—C23 | 121.7 (3) | C17—C18—C19 | 120.7 (4) |
| C22—N2—S2 | 115.7 (3) | C17—C18—H18 | 119.7 |
| C23—N2—S2 | 122.2 (3) | C19—C18—H18 | 119.7 |
| C2—C1—C6 | 123.1 (4) | C20—C19—C18 | 121.7 (4) |

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| C2—C1—S1 | 127.2 (3) | C20—C19—H19 | 119.2 |
| C6—C1—S1 | 109.6 (3) | C18—C19—H19 | 119.2 |
| C1—C2—C3 | 116.5 (4) | C21—C20—C19 | 117.7 (4) |
| C1—C2—H2 | 121.7 | C21—C20—H20 | 121.1 |
| C3—C2—H2 | 121.7 | C19—C20—H20 | 121.1 |
| C4—C3—C2 | 121.3 (4) | C16—C21—C20 | 120.1 (3) |
| C4—C3—H3 | 119.4 | C16—C21—C22 | 113.3 (3) |
| C2—C3—H3 | 119.4 | C20—C21—C22 | 126.6 (3) |
| C3—C4—C5 | 121.1 (4) | O7—C22—N2 | 124.1 (4) |
| C3—C4—H4 | 119.5 | O7—C22—C21 | 127.4 (4) |
| C5—C4—H4 | 119.5 | N2—C22—C21 | 108.4 (3) |
| C6—C5—C4 | 118.3 (4) | N2—C23—C24 | 110.8 (3) |
| C6—C5—H5 | 120.9 | N2—C23—H23A | 109.5 |
| C4—C5—H5 | 120.9 | C24—C23—H23A | 109.5 |
| C5—C6—C1 | 119.7 (4) | N2—C23—H23B | 109.5 |
| C5—C6—C7 | 126.8 (4) | C24—C23—H23B | 109.5 |
| C1—C6—C7 | 113.5 (3) | H23A—C23—H23B | 108.1 |
| O3—C7—N1 | 124.0 (4) | O8—C24—C25 | 124.0 (4) |
| O3—C7—C6 | 127.5 (4) | O8—C24—C23 | 119.6 (3) |
| N1—C7—C6 | 108.4 (3) | C25—C24—C23 | 116.4 (3) |
| N1—C8—C9 | 111.4 (3) | C26—C25—C30 | 117.4 (4) |
| N1—C8—H8A | 109.4 | C26—C25—C24 | 124.0 (4) |
| C9—C8—H8A | 109.4 | C30—C25—C24 | 118.6 (3) |
| N1—C8—H8B | 109.4 | C27—C26—C25 | 120.6 (4) |
| C9—C8—H8B | 109.4 | C27—C26—Br2 | 115.6 (3) |
| H8A—C8—H8B | 108.0 | C25—C26—Br2 | 123.8 (3) |
| O4—C9—C10 | 119.6 (4) | C28—C27—C26 | 120.3 (4) |
| O4—C9—C8 | 119.7 (3) | C28—C27—H27 | 119.9 |
| C10—C9—C8 | 120.6 (3) | C26—C27—H27 | 119.9 |
| C11—C10—C15 | 117.1 (4) | C29—C28—C27 | 120.0 (4) |
| C11—C10—C9 | 128.4 (4) | C29—C28—H28 | 120.0 |
| C15—C10—C9 | 114.5 (4) | C27—C28—H28 | 120.0 |
| C10—C11—C12 | 121.6 (4) | C28—C29—C30 | 119.9 (4) |
| C10—C11—Br1 | 123.4 (3) | C28—C29—H29 | 120.0 |
| C12—C11—Br1 | 114.8 (3) | C30—C29—H29 | 120.0 |
| C13—C12—C11 | 119.3 (4) | C29—C30—C25 | 121.8 (4) |
| C13—C12—H12 | 120.4 | C29—C30—H30 | 119.1 |
| C11—C12—H12 | 120.4 | C25—C30—H30 | 119.1 |

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| O1—S1—N1—C7 | −111.1 (3) | Br1—C11—C12—C13 | 175.6 (3) |
| O2—S1—N1—C7 | 119.3 (3) | C11—C12—C13—C14 | 1.1 (7) |
| C1—S1—N1—C7 | 4.1 (3) | C12—C13—C14—C15 | −1.1 (7) |
| O1—S1—N1—C8 | 65.3 (3) | C13—C14—C15—C10 | 0.4 (7) |
| O2—S1—N1—C8 | −64.3 (3) | C11—C10—C15—C14 | 0.2 (6) |
| C1—S1—N1—C8 | −179.6 (3) | C9—C10—C15—C14 | −179.4 (4) |
| O5—S2—N2—C22 | −116.7 (3) | O5—S2—C16—C21 | 113.1 (3) |
| O6—S2—N2—C22 | 113.0 (3) | O6—S2—C16—C21 | −112.2 (3) |
| C16—S2—N2—C22 | −1.8 (3) | N2—S2—C16—C21 | 0.3 (3) |

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| O5—S2—N2—C23 | 70.9 (3) | O5—S2—C16—C17 | −67.8 (4) |
| O6—S2—N2—C23 | −59.4 (3) | O6—S2—C16—C17 | 66.9 (4) |
| C16—S2—N2—C23 | −174.2 (3) | N2—S2—C16—C17 | 179.4 (3) |
| O1—S1—C1—C2 | −69.0 (4) | C21—C16—C17—C18 | −0.4 (6) |
| O2—S1—C1—C2 | 65.8 (4) | S2—C16—C17—C18 | −179.4 (3) |
| N1—S1—C1—C2 | 178.2 (4) | C16—C17—C18—C19 | −0.8 (6) |
| O1—S1—C1—C6 | 109.9 (3) | C17—C18—C19—C20 | 1.5 (6) |
| O2—S1—C1—C6 | −115.4 (3) | C18—C19—C20—C21 | −0.9 (6) |
| N1—S1—C1—C6 | −2.9 (3) | C17—C16—C21—C20 | 1.0 (6) |
| C6—C1—C2—C3 | 0.1 (6) | S2—C16—C21—C20 | −179.9 (3) |
| S1—C1—C2—C3 | 178.8 (3) | C17—C16—C21—C22 | −178.0 (3) |
| C1—C2—C3—C4 | −0.5 (6) | S2—C16—C21—C22 | 1.1 (4) |
| C2—C3—C4—C5 | 0.3 (6) | C19—C20—C21—C16 | −0.3 (5) |
| C3—C4—C5—C6 | 0.4 (6) | C19—C20—C21—C22 | 178.6 (4) |
| C4—C5—C6—C1 | −0.8 (6) | C23—N2—C22—O7 | −4.5 (6) |
| C4—C5—C6—C7 | 179.7 (4) | S2—N2—C22—O7 | −176.9 (3) |
| C2—C1—C6—C5 | 0.6 (6) | C23—N2—C22—C21 | 175.0 (3) |
| S1—C1—C6—C5 | −178.3 (3) | S2—N2—C22—C21 | 2.6 (4) |
| C2—C1—C6—C7 | −179.9 (4) | C16—C21—C22—O7 | 177.2 (4) |
| S1—C1—C6—C7 | 1.3 (4) | C20—C21—C22—O7 | −1.7 (6) |
| C8—N1—C7—O3 | 1.4 (6) | C16—C21—C22—N2 | −2.3 (4) |
| S1—N1—C7—O3 | 177.7 (3) | C20—C21—C22—N2 | 178.8 (4) |
| C8—N1—C7—C6 | 179.8 (3) | C22—N2—C23—C24 | 83.4 (4) |
| S1—N1—C7—C6 | −3.9 (4) | S2—N2—C23—C24 | −104.7 (3) |
| C5—C6—C7—O3 | −0.7 (7) | N2—C23—C24—O8 | 6.4 (5) |
| C1—C6—C7—O3 | 179.8 (4) | N2—C23—C24—C25 | −172.9 (3) |
| C5—C6—C7—N1 | −179.0 (4) | O8—C24—C25—C26 | −7.6 (6) |
| C1—C6—C7—N1 | 1.5 (4) | C23—C24—C25—C26 | 171.7 (4) |
| C7—N1—C8—C9 | 90.7 (4) | O8—C24—C25—C30 | 171.8 (4) |
| S1—N1—C8—C9 | −85.4 (4) | C23—C24—C25—C30 | −8.9 (5) |
| N1—C8—C9—O4 | −22.3 (5) | C30—C25—C26—C27 | 2.2 (6) |
| N1—C8—C9—C10 | 155.1 (3) | C24—C25—C26—C27 | −178.4 (4) |
| O4—C9—C10—C11 | −155.3 (4) | C30—C25—C26—Br2 | −176.7 (3) |
| C8—C9—C10—C11 | 27.3 (6) | C24—C25—C26—Br2 | 2.7 (5) |
| O4—C9—C10—C15 | 24.3 (5) | C25—C26—C27—C28 | −2.9 (6) |
| C8—C9—C10—C15 | −153.1 (4) | Br2—C26—C27—C28 | 176.0 (3) |
| C15—C10—C11—C12 | −0.2 (6) | C26—C27—C28—C29 | 1.4 (6) |
| C9—C10—C11—C12 | 179.4 (4) | C27—C28—C29—C30 | 0.7 (6) |
| C15—C10—C11—Br1 | −176.0 (3) | C28—C29—C30—C25 | −1.4 (6) |
| C9—C10—C11—Br1 | 3.6 (6) | C26—C25—C30—C29 | −0.1 (6) |
| C10—C11—C12—C13 | −0.5 (6) | C24—C25—C30—C29 | −179.5 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C3—H3···O7$^{i}$ | 0.95 | 2.56 | 3.238 (5) | 129 |
| C14—H14···O1$^{ii}$ | 0.95 | 2.40 | 3.305 (5) | 159 |
| C17—H17···O5$^{iii}$ | 0.95 | 2.43 | 3.225 (5) | 141 |

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|---|---|---|---|---|
| C27—H27···O7$^{iv}$ | 0.95 | 2.29 | 3.164 (5) | 153 |
| C30—H30···O2$^{v}$ | 0.95 | 2.51 | 3.251 (5) | 135 |
| C8—H8*A*···Br1 | 0.99 | 2.82 | 3.165 (4) | 101 |
| C23—H23*A*···O3 | 0.99 | 2.48 | 3.014 (5) | 114 |

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