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# *N*′-(3-Ethoxy-2-hydroxybenzylidene)-benzenesulfonohydrazide

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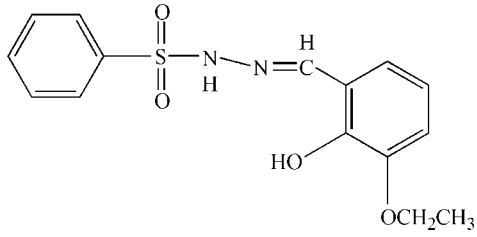
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.009 Å; $R$ factor = 0.074; $wR$ factor = 0.161; data-to-parameter ratio = 14.0.

There are two molecules in the asymmetric unit of the title compound, $C_{15}H_{16}N_2O_4S$, both of which are stabilized by an intramolecular O—H···N hydrogen bond. Intermolecular N—H···O hydrogen bonds lead to [101] chains of molecules in the crystal structure.

## Related literature

For related literature, see: Tai *et al.* (2003).



## Experimental

*Crystal data*

$C_{15}H_{16}N_2O_4S$
$M_r$ = 320.36
Monoclinic, $P2_1/c$
$a$ = 10.3149 (10) Å
$b$ = 39.935 (3) Å
$c$ = 7.9832 (8) Å
$\beta$ = 105.773 (2)°
$V$ = 3164.7 (5) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.22 mm$^{-1}$
$T$ = 298 (2) K
0.48 × 0.45 × 0.32 mm

*Data collection*

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min}$ = 0.900, $T_{max}$ = 0.932
16316 measured reflections
5576 independent reflections
3515 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.160
$S$ = 1.09
5576 reflections
399 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···N2 | 0.82 | 1.89 | 2.606 (5) | 145 |
| O5—H5···N4 | 0.82 | 1.87 | 2.591 (5) | 146 |
| N1—H1*A*···O5 | 0.90 | 2.14 | 2.948 (5) | 149 |
| N3—H3···O3$^{i}$ | 0.90 | 2.11 | 2.912 (5) | 147 |

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2710).

## References

Bruker (2000). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tai, X.-S., Yin, X.-H., Tan, M.-Y. & Li, Y.-Z. (2003). *Acta Cryst.* E**59**, o681–o682.

# supplementary materials

*Acta Cryst.* (2008). E**64**, o750 [ **doi:10.1107/S1600536808007988** ]

# *N*'-(3-Ethoxy-2-hydroxybenzylidene)benzenesulfonohydrazide

**X.-S. Tai, Y.-M. Feng and F.-Y. Kong**

## Comment

As part of our ongoing studies of aroylhydrazone ligands (Tai *et al.*, 2003), we now report the synthesis and structure of the title compound, (I), (Fig. 1).

There are two molecules in the asymmetric unit of (I), both of which are stabilised by an intramolecular O-H···N hydrogen bond (Table 1). Then, intermolecular N-H···O hydrogen bonds lead to [101] chains of molecules in the crystal.

## Experimental

3-Ethoxysalicylaldehyde (3 mmol) was added to a solution of benzenesulfonyl hydrazide (3 mmol) in 10 ml of 95% ethanol. The mixture was continuously stirred for 3 h at refluxing temperature, evaporating some ethanol, then, upon cooling, the solid product was collected by filtration and dried in vacuo (yield 78%). Colourless blocks of (I) were obtained by evaporation from a methanol solution after 3 days.

## Refinement

The H atoms were placed geometrically (C—H = 0.93–0.96 Å, N—H = 0.90 Å, O-H = 0.82Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C, N) or 1.5$U_{eq}$(methyl C, O).

## Figures

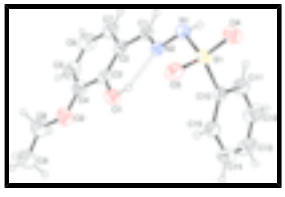


Fig. 1. The C1-containing molecule in (I) showing 30% displacement ellipsoids for the non-hydrogen atoms. The intramolecular hydrogen bond is indicated by a double-dashed line.

## N'-(3-Ethoxy-2-hydroxybenzylidene)benzenesulfonohydrazide

*Crystal data*

| | |
|---|---|
| $C_{15}H_{16}N_2O_4S$ | $F_{000}$ = 1344 |
| $M_r$ = 320.36 | $D_x$ = 1.345 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 3888 reflections |
| $a$ = 10.3149 (10) Å | θ = 2.3–22.4º |
| $b$ = 39.935 (3) Å | μ = 0.22 mm$^{-1}$ |

| | |
|---|---|
| $c$ = 7.9832 (8) Å | $T$ = 298 (2) K |
| β = 105.773 (2)º | Block, colourless |
| $V$ = 3164.7 (5) Å$^3$ | 0.48 × 0.45 × 0.32 mm |
| $Z$ = 8 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD diffractometer | 5576 independent reflections |
| Radiation source: fine-focus sealed tube | 3515 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.047 |
| $T$ = 298(2) K | $\theta_{max}$ = 25.0º |
| ω scans | $\theta_{min}$ = 2.0º |
| Absorption correction: multi-scan (SADABS; Bruker, 2000) | $h = -10 \rightarrow 12$ |
| $T_{min}$ = 0.900, $T_{max}$ = 0.932 | $k = -47 \rightarrow 47$ |
| 16316 measured reflections | $l = -8 \rightarrow 9$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.074 | H-atom parameters constrained |
| $wR(F^2)$ = 0.160 | $w = 1/[\sigma^2(F_o^2) + (0.0121P)^2 + 7.6148P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.09 | $(\Delta/\sigma)_{max} < 0.001$ |
| 5576 reflections | $\Delta\rho_{max}$ = 0.29 e Å$^{-3}$ |
| 399 parameters | $\Delta\rho_{min}$ = −0.41 e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.8862 (4) | 0.15474 (10) | 0.5062 (5) | 0.0505 (10) |

| | | | | |
|---|---|---|---|---|
| H1A | 0.8190 | 0.1591 | 0.4102 | 0.061* |
| N2 | 0.8858 (4) | 0.12091 (10) | 0.5385 (5) | 0.0457 (10) |
| N3 | 0.4135 (4) | 0.16102 (10) | −0.1714 (5) | 0.0555 (11) |
| H3 | 0.3271 | 0.1542 | −0.2033 | 0.067* |
| N4 | 0.4452 (4) | 0.16983 (10) | 0.0019 (5) | 0.0520 (10) |
| O1 | 1.0083 (3) | 0.06423 (9) | 0.6356 (5) | 0.0632 (10) |
| H1 | 1.0025 | 0.0839 | 0.6056 | 0.095* |
| O2 | 0.9949 (4) | 0.00231 (10) | 0.7310 (5) | 0.0795 (12) |
| O3 | 1.1327 (3) | 0.16038 (9) | 0.6246 (4) | 0.0632 (10) |
| O4 | 0.9990 (4) | 0.20311 (8) | 0.4304 (5) | 0.0704 (11) |
| O5 | 0.6248 (3) | 0.18032 (8) | 0.2945 (4) | 0.0590 (9) |
| H5 | 0.5955 | 0.1729 | 0.1956 | 0.088* |
| O6 | 0.6879 (4) | 0.20697 (10) | 0.6006 (5) | 0.0739 (11) |
| O7 | 0.6420 (3) | 0.13862 (10) | −0.1276 (5) | 0.0732 (11) |
| O8 | 0.4647 (3) | 0.12903 (9) | −0.4027 (4) | 0.0628 (10) |
| S1 | 1.02564 (12) | 0.16900 (3) | 0.47484 (16) | 0.0488 (3) |
| S2 | 0.50608 (12) | 0.13114 (4) | −0.21787 (17) | 0.0560 (4) |
| C1 | 0.7737 (5) | 0.10720 (13) | 0.5375 (6) | 0.0490 (12) |
| H1C | 0.6947 | 0.1197 | 0.5077 | 0.059* |
| C2 | 0.7698 (5) | 0.07232 (13) | 0.5827 (6) | 0.0490 (12) |
| C3 | 0.8843 (5) | 0.05248 (13) | 0.6323 (6) | 0.0524 (13) |
| C4 | 0.8766 (6) | 0.01893 (14) | 0.6801 (7) | 0.0643 (15) |
| C5 | 0.7519 (7) | 0.00549 (16) | 0.6748 (8) | 0.084 (2) |
| H5A | 0.7448 | −0.0169 | 0.7023 | 0.100* |
| C6 | 0.6378 (7) | 0.02532 (17) | 0.6289 (9) | 0.0834 (19) |
| H6 | 0.5550 | 0.0162 | 0.6301 | 0.100* |
| C7 | 0.6444 (5) | 0.05806 (15) | 0.5819 (7) | 0.0646 (15) |
| H7 | 0.5664 | 0.0709 | 0.5494 | 0.078* |
| C8 | 0.9923 (7) | −0.03169 (15) | 0.7840 (10) | 0.094 (2) |
| H8A | 0.9388 | −0.0451 | 0.6888 | 0.113* |
| H8B | 0.9529 | −0.0333 | 0.8811 | 0.113* |
| C9 | 1.1336 (8) | −0.04390 (16) | 0.8364 (10) | 0.106 (2) |
| H9A | 1.1737 | −0.0407 | 0.7422 | 0.158* |
| H9B | 1.1347 | −0.0673 | 0.8644 | 0.158* |
| H9C | 1.1839 | −0.0316 | 0.9365 | 0.158* |
| C10 | 1.0519 (4) | 0.14629 (11) | 0.2984 (6) | 0.0446 (11) |
| C11 | 0.9513 (5) | 0.14558 (15) | 0.1440 (7) | 0.0672 (16) |
| H11 | 0.8698 | 0.1564 | 0.1344 | 0.081* |
| C12 | 0.9733 (7) | 0.12869 (19) | 0.0053 (8) | 0.093 (2) |
| H12 | 0.9058 | 0.1279 | −0.0991 | 0.111* |
| C13 | 1.0929 (7) | 0.11296 (17) | 0.0181 (8) | 0.087 (2) |
| H13 | 1.1065 | 0.1014 | −0.0770 | 0.104* |
| C14 | 1.1931 (6) | 0.11413 (15) | 0.1711 (8) | 0.0729 (17) |
| H14 | 1.2752 | 0.1037 | 0.1791 | 0.087* |
| C15 | 1.1724 (5) | 0.13063 (13) | 0.3130 (7) | 0.0567 (13) |
| H15 | 1.2397 | 0.1311 | 0.4177 | 0.068* |
| C16 | 0.3577 (5) | 0.18495 (12) | 0.0593 (7) | 0.0536 (13) |
| H16 | 0.2712 | 0.1881 | −0.0130 | 0.064* |
| C17 | 0.3915 (5) | 0.19724 (11) | 0.2357 (7) | 0.0475 (12) |

| | | | | |
|---|---|---|---|---|
| C18 | 0.5232 (5) | 0.19556 (11) | 0.3449 (6) | 0.0487 (12) |
| C19 | 0.5557 (5) | 0.20971 (12) | 0.5107 (7) | 0.0543 (13) |
| C20 | 0.4553 (6) | 0.22539 (13) | 0.5660 (8) | 0.0709 (16) |
| H20 | 0.4764 | 0.2353 | 0.6754 | 0.085* |
| C21 | 0.3247 (6) | 0.22661 (14) | 0.4621 (9) | 0.0739 (17) |
| H21 | 0.2576 | 0.2365 | 0.5031 | 0.089* |
| C22 | 0.2943 (5) | 0.21326 (13) | 0.2996 (8) | 0.0651 (16) |
| H22 | 0.2064 | 0.2148 | 0.2290 | 0.078* |
| C23 | 0.7310 (7) | 0.22377 (17) | 0.7650 (9) | 0.094 (2) |
| H23A | 0.6864 | 0.2142 | 0.8464 | 0.113* |
| H23B | 0.7086 | 0.2474 | 0.7510 | 0.113* |
| C24 | 0.8777 (8) | 0.21949 (18) | 0.8307 (9) | 0.114 (3) |
| H24A | 0.8991 | 0.1960 | 0.8392 | 0.171* |
| H24B | 0.9086 | 0.2296 | 0.9437 | 0.171* |
| H24C | 0.9211 | 0.2300 | 0.7523 | 0.171* |
| C25 | 0.4610 (5) | 0.09376 (13) | −0.1326 (6) | 0.0530 (13) |
| C26 | 0.3513 (5) | 0.07603 (15) | −0.2264 (8) | 0.0721 (17) |
| H26 | 0.3004 | 0.0840 | −0.3335 | 0.087* |
| C27 | 0.3164 (6) | 0.04625 (16) | −0.1614 (10) | 0.0851 (19) |
| H27 | 0.2428 | 0.0340 | −0.2254 | 0.102* |
| C28 | 0.3898 (8) | 0.03494 (18) | −0.0040 (11) | 0.091 (2) |
| H28 | 0.3668 | 0.0149 | 0.0397 | 0.109* |
| C29 | 0.4971 (9) | 0.05293 (19) | 0.0901 (9) | 0.098 (2) |
| H29 | 0.5459 | 0.0452 | 0.1989 | 0.118* |
| C30 | 0.5346 (6) | 0.08246 (16) | 0.0265 (8) | 0.0758 (17) |
| H30 | 0.6088 | 0.0945 | 0.0908 | 0.091* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.034 (2) | 0.060 (3) | 0.053 (3) | 0.0022 (18) | 0.0051 (18) | 0.002 (2) |
| N2 | 0.042 (2) | 0.055 (3) | 0.040 (2) | −0.0004 (18) | 0.0097 (18) | 0.0001 (18) |
| N3 | 0.039 (2) | 0.063 (3) | 0.057 (3) | 0.003 (2) | 0.001 (2) | 0.000 (2) |
| N4 | 0.042 (2) | 0.056 (3) | 0.052 (3) | 0.002 (2) | 0.003 (2) | 0.001 (2) |
| O1 | 0.046 (2) | 0.064 (2) | 0.079 (3) | −0.0032 (17) | 0.0152 (18) | 0.0077 (19) |
| O2 | 0.084 (3) | 0.061 (2) | 0.094 (3) | −0.002 (2) | 0.023 (2) | 0.012 (2) |
| O3 | 0.0392 (18) | 0.097 (3) | 0.048 (2) | −0.0035 (18) | 0.0020 (16) | −0.0088 (19) |
| O4 | 0.069 (2) | 0.045 (2) | 0.100 (3) | −0.0005 (18) | 0.029 (2) | −0.001 (2) |
| O5 | 0.051 (2) | 0.071 (2) | 0.051 (2) | 0.0181 (17) | 0.0067 (17) | −0.0066 (17) |
| O6 | 0.075 (3) | 0.082 (3) | 0.057 (2) | 0.013 (2) | 0.005 (2) | −0.020 (2) |
| O7 | 0.0317 (18) | 0.101 (3) | 0.079 (3) | −0.0013 (19) | 0.0015 (18) | −0.004 (2) |
| O8 | 0.054 (2) | 0.087 (3) | 0.045 (2) | 0.0002 (19) | 0.0093 (16) | 0.0056 (18) |
| S1 | 0.0385 (6) | 0.0533 (8) | 0.0516 (8) | −0.0008 (6) | 0.0072 (6) | −0.0018 (6) |
| S2 | 0.0374 (7) | 0.0746 (9) | 0.0517 (8) | 0.0028 (6) | 0.0049 (6) | 0.0025 (7) |
| C1 | 0.037 (3) | 0.069 (3) | 0.041 (3) | 0.003 (2) | 0.009 (2) | 0.000 (2) |
| C2 | 0.045 (3) | 0.063 (3) | 0.039 (3) | −0.012 (2) | 0.012 (2) | −0.007 (2) |
| C3 | 0.055 (3) | 0.062 (3) | 0.043 (3) | −0.012 (3) | 0.018 (2) | −0.005 (2) |
| C4 | 0.073 (4) | 0.059 (4) | 0.063 (4) | −0.008 (3) | 0.021 (3) | 0.000 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.100 (5) | 0.069 (4) | 0.092 (5) | −0.036 (4) | 0.044 (4) | −0.010 (4) |
| C6 | 0.067 (4) | 0.094 (5) | 0.094 (5) | −0.036 (4) | 0.030 (4) | −0.016 (4) |
| C7 | 0.055 (3) | 0.078 (4) | 0.064 (4) | −0.017 (3) | 0.021 (3) | −0.009 (3) |
| C8 | 0.123 (6) | 0.057 (4) | 0.113 (6) | 0.000 (4) | 0.050 (5) | 0.006 (4) |
| C9 | 0.135 (7) | 0.071 (5) | 0.118 (6) | 0.023 (4) | 0.045 (5) | 0.008 (4) |
| C10 | 0.041 (3) | 0.049 (3) | 0.042 (3) | −0.004 (2) | 0.009 (2) | 0.009 (2) |
| C11 | 0.048 (3) | 0.092 (4) | 0.056 (4) | 0.006 (3) | 0.004 (3) | 0.000 (3) |
| C12 | 0.083 (5) | 0.140 (6) | 0.050 (4) | 0.000 (5) | 0.010 (3) | −0.020 (4) |
| C13 | 0.092 (5) | 0.111 (6) | 0.065 (4) | −0.007 (4) | 0.034 (4) | −0.023 (4) |
| C14 | 0.059 (4) | 0.079 (4) | 0.086 (5) | 0.003 (3) | 0.028 (3) | −0.008 (4) |
| C15 | 0.045 (3) | 0.070 (3) | 0.051 (3) | 0.002 (3) | 0.008 (2) | 0.003 (3) |
| C16 | 0.039 (3) | 0.045 (3) | 0.074 (4) | 0.003 (2) | 0.011 (3) | 0.011 (3) |
| C17 | 0.042 (3) | 0.037 (3) | 0.066 (3) | −0.001 (2) | 0.017 (2) | 0.004 (2) |
| C18 | 0.051 (3) | 0.040 (3) | 0.058 (3) | 0.008 (2) | 0.019 (3) | 0.003 (2) |
| C19 | 0.054 (3) | 0.045 (3) | 0.066 (4) | 0.001 (2) | 0.018 (3) | 0.000 (3) |
| C20 | 0.091 (5) | 0.054 (3) | 0.078 (4) | 0.002 (3) | 0.041 (4) | −0.011 (3) |
| C21 | 0.078 (4) | 0.056 (4) | 0.104 (5) | 0.000 (3) | 0.052 (4) | −0.012 (3) |
| C22 | 0.048 (3) | 0.050 (3) | 0.103 (5) | −0.001 (2) | 0.031 (3) | 0.004 (3) |
| C23 | 0.092 (5) | 0.099 (5) | 0.082 (5) | 0.009 (4) | 0.008 (4) | −0.036 (4) |
| C24 | 0.133 (7) | 0.104 (6) | 0.084 (5) | −0.003 (5) | −0.007 (5) | −0.032 (4) |
| C25 | 0.042 (3) | 0.068 (3) | 0.047 (3) | 0.017 (2) | 0.010 (2) | 0.006 (3) |
| C26 | 0.047 (3) | 0.084 (4) | 0.081 (4) | 0.001 (3) | 0.010 (3) | 0.023 (3) |
| C27 | 0.064 (4) | 0.079 (5) | 0.116 (6) | 0.002 (3) | 0.030 (4) | 0.018 (4) |
| C28 | 0.104 (6) | 0.077 (5) | 0.107 (6) | 0.024 (4) | 0.057 (5) | 0.024 (4) |
| C29 | 0.136 (7) | 0.093 (6) | 0.063 (4) | 0.038 (5) | 0.024 (5) | 0.024 (4) |
| C30 | 0.080 (4) | 0.077 (4) | 0.061 (4) | 0.016 (3) | 0.002 (3) | 0.000 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—N2 | 1.375 (5) | C10—C15 | 1.368 (6) |
| N1—S1 | 1.628 (4) | C10—C11 | 1.379 (6) |
| N1—H1A | 0.8996 | C11—C12 | 1.367 (8) |
| N2—C1 | 1.278 (5) | C11—H11 | 0.9300 |
| N3—N4 | 1.378 (5) | C12—C13 | 1.363 (8) |
| N3—S2 | 1.633 (4) | C12—H12 | 0.9300 |
| N3—H3 | 0.8998 | C13—C14 | 1.370 (8) |
| N4—C16 | 1.271 (6) | C13—H13 | 0.9300 |
| O1—C3 | 1.355 (5) | C14—C15 | 1.376 (7) |
| O1—H1 | 0.8187 | C14—H14 | 0.9300 |
| O2—C4 | 1.351 (6) | C15—H15 | 0.9300 |
| O2—C8 | 1.425 (6) | C16—C17 | 1.442 (7) |
| O3—S1 | 1.432 (3) | C16—H16 | 0.9300 |
| O4—S1 | 1.416 (3) | C17—C22 | 1.398 (7) |
| O5—C18 | 1.364 (5) | C17—C18 | 1.402 (6) |
| O5—H5 | 0.8202 | C18—C19 | 1.394 (7) |
| O6—C19 | 1.362 (6) | C19—C20 | 1.381 (7) |
| O6—C23 | 1.433 (6) | C20—C21 | 1.378 (8) |
| O7—S2 | 1.423 (3) | C20—H20 | 0.9300 |
| O8—S2 | 1.422 (3) | C21—C22 | 1.358 (8) |

| | | | |
|---|---|---|---|
| S1—C10 | 1.757 (5) | C21—H21 | 0.9300 |
| S2—C25 | 1.755 (5) | C22—H22 | 0.9300 |
| C1—C2 | 1.442 (7) | C23—C24 | 1.470 (9) |
| C1—H1C | 0.9300 | C23—H23A | 0.9700 |
| C2—C3 | 1.388 (7) | C23—H23B | 0.9700 |
| C2—C7 | 1.411 (6) | C24—H24A | 0.9600 |
| C3—C4 | 1.401 (7) | C24—H24B | 0.9600 |
| C4—C5 | 1.384 (8) | C24—H24C | 0.9600 |
| C5—C6 | 1.383 (9) | C25—C30 | 1.368 (7) |
| C5—H5A | 0.9300 | C25—C26 | 1.372 (7) |
| C6—C7 | 1.367 (8) | C26—C27 | 1.384 (8) |
| C6—H6 | 0.9300 | C26—H26 | 0.9300 |
| C7—H7 | 0.9300 | C27—C28 | 1.356 (9) |
| C8—C9 | 1.485 (9) | C27—H27 | 0.9300 |
| C8—H8A | 0.9700 | C28—C29 | 1.362 (9) |
| C8—H8B | 0.9700 | C28—H28 | 0.9300 |
| C9—H9A | 0.9600 | C29—C30 | 1.380 (9) |
| C9—H9B | 0.9600 | C29—H29 | 0.9300 |
| C9—H9C | 0.9600 | C30—H30 | 0.9300 |
| N2—N1—S1 | 114.8 (3) | C13—C12—C11 | 120.9 (6) |
| N2—N1—H1A | 108.0 | C13—C12—H12 | 119.6 |
| S1—N1—H1A | 108.0 | C11—C12—H12 | 119.6 |
| C1—N2—N1 | 118.0 (4) | C12—C13—C14 | 119.9 (6) |
| N4—N3—S2 | 114.9 (3) | C12—C13—H13 | 120.0 |
| N4—N3—H3 | 107.9 | C14—C13—H13 | 120.0 |
| S2—N3—H3 | 108.1 | C13—C14—C15 | 120.2 (6) |
| C16—N4—N3 | 119.0 (4) | C13—C14—H14 | 119.9 |
| C3—O1—H1 | 109.4 | C15—C14—H14 | 119.9 |
| C4—O2—C8 | 118.0 (5) | C10—C15—C14 | 119.3 (5) |
| C18—O5—H5 | 109.5 | C10—C15—H15 | 120.4 |
| C19—O6—C23 | 117.5 (4) | C14—C15—H15 | 120.4 |
| O4—S1—O3 | 119.6 (2) | N4—C16—C17 | 120.4 (4) |
| O4—S1—N1 | 104.8 (2) | N4—C16—H16 | 119.8 |
| O3—S1—N1 | 107.5 (2) | C17—C16—H16 | 119.8 |
| O4—S1—C10 | 110.9 (2) | C22—C17—C18 | 117.7 (5) |
| O3—S1—C10 | 107.1 (2) | C22—C17—C16 | 120.3 (5) |
| N1—S1—C10 | 106.1 (2) | C18—C17—C16 | 121.8 (4) |
| O8—S2—O7 | 121.0 (2) | O5—C18—C19 | 117.1 (4) |
| O8—S2—N3 | 104.5 (2) | O5—C18—C17 | 122.0 (4) |
| O7—S2—N3 | 106.7 (2) | C19—C18—C17 | 120.9 (5) |
| O8—S2—C25 | 108.4 (2) | O6—C19—C20 | 126.7 (5) |
| O7—S2—C25 | 108.1 (2) | O6—C19—C18 | 114.5 (4) |
| N3—S2—C25 | 107.5 (2) | C20—C19—C18 | 118.7 (5) |
| N2—C1—C2 | 120.0 (4) | C21—C20—C19 | 121.3 (6) |
| N2—C1—H1C | 120.0 | C21—C20—H20 | 119.4 |
| C2—C1—H1C | 120.0 | C19—C20—H20 | 119.4 |
| C3—C2—C7 | 118.6 (5) | C22—C21—C20 | 119.7 (5) |
| C3—C2—C1 | 122.9 (4) | C22—C21—H21 | 120.2 |
| C7—C2—C1 | 118.5 (5) | C20—C21—H21 | 120.2 |

| | | | |
|---|---|---|---|
| O1—C3—C2 | 122.2 (5) | C21—C22—C17 | 121.7 (5) |
| O1—C3—C4 | 116.7 (5) | C21—C22—H22 | 119.1 |
| C2—C3—C4 | 121.1 (5) | C17—C22—H22 | 119.1 |
| O2—C4—C5 | 125.1 (6) | O6—C23—C24 | 107.8 (5) |
| O2—C4—C3 | 116.0 (5) | O6—C23—H23A | 110.1 |
| C5—C4—C3 | 118.9 (6) | C24—C23—H23A | 110.1 |
| C6—C5—C4 | 120.2 (6) | O6—C23—H23B | 110.1 |
| C6—C5—H5A | 119.9 | C24—C23—H23B | 110.1 |
| C4—C5—H5A | 119.9 | H23A—C23—H23B | 108.5 |
| C7—C6—C5 | 121.2 (6) | C23—C24—H24A | 109.5 |
| C7—C6—H6 | 119.4 | C23—C24—H24B | 109.5 |
| C5—C6—H6 | 119.4 | H24A—C24—H24B | 109.5 |
| C6—C7—C2 | 119.9 (6) | C23—C24—H24C | 109.5 |
| C6—C7—H7 | 120.0 | H24A—C24—H24C | 109.5 |
| C2—C7—H7 | 120.0 | H24B—C24—H24C | 109.5 |
| O2—C8—C9 | 107.5 (5) | C30—C25—C26 | 120.3 (5) |
| O2—C8—H8A | 110.2 | C30—C25—S2 | 120.1 (4) |
| C9—C8—H8A | 110.2 | C26—C25—S2 | 119.6 (4) |
| O2—C8—H8B | 110.2 | C25—C26—C27 | 119.9 (6) |
| C9—C8—H8B | 110.2 | C25—C26—H26 | 120.0 |
| H8A—C8—H8B | 108.5 | C27—C26—H26 | 120.0 |
| C8—C9—H9A | 109.5 | C28—C27—C26 | 119.8 (7) |
| C8—C9—H9B | 109.5 | C28—C27—H27 | 120.1 |
| H9A—C9—H9B | 109.5 | C26—C27—H27 | 120.1 |
| C8—C9—H9C | 109.5 | C27—C28—C29 | 120.1 (7) |
| H9A—C9—H9C | 109.5 | C27—C28—H28 | 120.0 |
| H9B—C9—H9C | 109.5 | C29—C28—H28 | 120.0 |
| C15—C10—C11 | 120.8 (5) | C28—C29—C30 | 121.0 (6) |
| C15—C10—S1 | 120.1 (4) | C28—C29—H29 | 119.5 |
| C11—C10—S1 | 119.0 (4) | C30—C29—H29 | 119.5 |
| C12—C11—C10 | 118.9 (5) | C25—C30—C29 | 118.9 (6) |
| C12—C11—H11 | 120.5 | C25—C30—H30 | 120.6 |
| C10—C11—H11 | 120.5 | C29—C30—H30 | 120.6 |
| S1—N1—N2—C1 | −169.4 (3) | C12—C13—C14—C15 | 1.2 (10) |
| S2—N3—N4—C16 | −159.3 (4) | C11—C10—C15—C14 | 0.3 (8) |
| N2—N1—S1—O4 | 175.3 (3) | S1—C10—C15—C14 | −177.3 (4) |
| N2—N1—S1—O3 | −56.5 (4) | C13—C14—C15—C10 | −1.1 (9) |
| N2—N1—S1—C10 | 57.8 (4) | N3—N4—C16—C17 | −174.3 (4) |
| N4—N3—S2—O8 | −174.1 (3) | N4—C16—C17—C22 | −178.7 (4) |
| N4—N3—S2—O7 | −44.9 (4) | N4—C16—C17—C18 | 5.4 (7) |
| N4—N3—S2—C25 | 70.9 (4) | C22—C17—C18—O5 | 179.7 (4) |
| N1—N2—C1—C2 | −175.6 (4) | C16—C17—C18—O5 | −4.3 (7) |
| N2—C1—C2—C3 | 1.6 (7) | C22—C17—C18—C19 | −0.6 (7) |
| N2—C1—C2—C7 | 179.4 (4) | C16—C17—C18—C19 | 175.4 (4) |
| C7—C2—C3—O1 | 179.6 (4) | C23—O6—C19—C20 | −4.3 (8) |
| C1—C2—C3—O1 | −2.5 (7) | C23—O6—C19—C18 | 174.1 (5) |
| C7—C2—C3—C4 | 0.3 (7) | O5—C18—C19—O6 | 1.4 (6) |
| C1—C2—C3—C4 | 178.1 (5) | C17—C18—C19—O6 | −178.4 (4) |
| C8—O2—C4—C5 | −0.1 (9) | O5—C18—C19—C20 | 179.9 (4) |

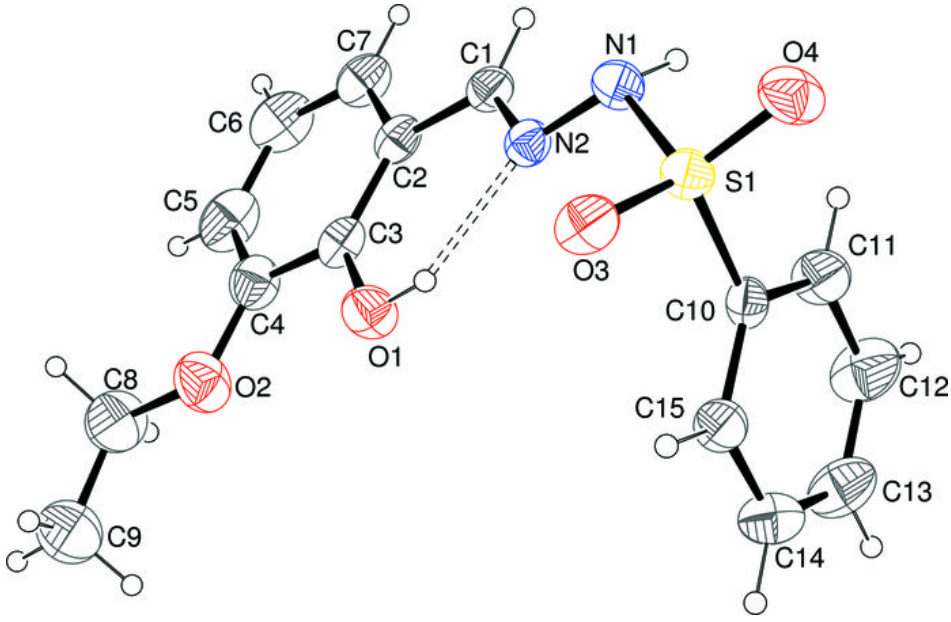
| | | | |
|---|---|---|---|
| C8—O2—C4—C3 | 178.8 (5) | C17—C18—C19—C20 | 0.1 (7) |
| O1—C3—C4—O2 | 2.5 (7) | O6—C19—C20—C21 | 179.7 (5) |
| C2—C3—C4—O2 | −178.1 (5) | C18—C19—C20—C21 | 1.4 (8) |
| O1—C3—C4—C5 | −178.5 (5) | C19—C20—C21—C22 | −2.5 (9) |
| C2—C3—C4—C5 | 0.9 (8) | C20—C21—C22—C17 | 2.1 (8) |
| O2—C4—C5—C6 | 176.7 (6) | C18—C17—C22—C21 | −0.6 (7) |
| C3—C4—C5—C6 | −2.2 (9) | C16—C17—C22—C21 | −176.6 (5) |
| C4—C5—C6—C7 | 2.4 (10) | C19—O6—C23—C24 | −175.7 (5) |
| C5—C6—C7—C2 | −1.1 (9) | O8—S2—C25—C30 | 151.3 (4) |
| C3—C2—C7—C6 | −0.2 (8) | O7—S2—C25—C30 | 18.6 (5) |
| C1—C2—C7—C6 | −178.1 (5) | N3—S2—C25—C30 | −96.3 (4) |
| C4—O2—C8—C9 | −178.9 (5) | O8—S2—C25—C26 | −28.9 (5) |
| O4—S1—C10—C15 | 119.0 (4) | O7—S2—C25—C26 | −161.7 (4) |
| O3—S1—C10—C15 | −13.1 (5) | N3—S2—C25—C26 | 83.5 (5) |
| N1—S1—C10—C15 | −127.7 (4) | C30—C25—C26—C27 | −1.3 (8) |
| O4—S1—C10—C11 | −58.7 (5) | S2—C25—C26—C27 | 179.0 (5) |
| O3—S1—C10—C11 | 169.2 (4) | C25—C26—C27—C28 | 0.9 (9) |
| N1—S1—C10—C11 | 54.6 (5) | C26—C27—C28—C29 | 0.3 (10) |
| C15—C10—C11—C12 | 0.5 (8) | C27—C28—C29—C30 | −1.2 (11) |
| S1—C10—C11—C12 | 178.2 (5) | C26—C25—C30—C29 | 0.4 (8) |
| C10—C11—C12—C13 | −0.5 (10) | S2—C25—C30—C29 | −179.8 (5) |
| C11—C12—C13—C14 | −0.4 (11) | C28—C29—C30—C25 | 0.8 (10) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N2 | 0.82 | 1.89 | 2.606 (5) | 145 |
| O5—H5···N4 | 0.82 | 1.87 | 2.591 (5) | 146 |
| N1—H1A···O5 | 0.90 | 2.14 | 2.948 (5) | 149 |
| N3—H3···O3$^{i}$ | 0.90 | 2.11 | 2.912 (5) | 147 |

Symmetry codes: (i) *x*−1, *y*, *z*−1.

**Fig. 1**


C7
C1
N1
O4
C6
N2
C2
S1
C5
C3
O3
C11
C4
C10
O1
C8
O2
C12
C15
C13
C9
C14