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

# Ethyl 3-oxo-2-[(4-sulfamoylphenyl)-hydrazono]butyrate

**K. K. Upadhyay,[a]* Priyanka Rai,[b] Shalini Upadhyay[a] and M. Nethaji[c]**

[a]Department of Chemistry, Faculty of Science, Banaras Hindu University, Varanasi 221 005, India, [b]Department of Chemistry, Udai Pratap College (Autonomuos), Varanasi 221 002, India, and [c]Department of Inorganic and Physical Chemistry, Indian Institute of Sciences, Bangalore 560 012, India
Correspondence e-mail: drkaushalbhu@yahoo.co.in

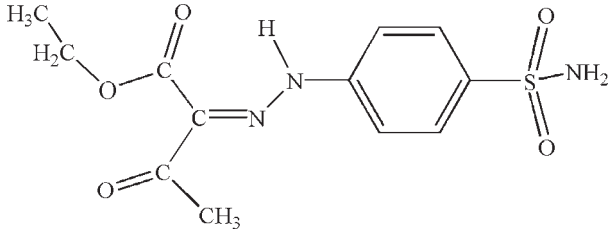
Received 10 August 2009; accepted 12 September 2009

Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.070; $wR$ factor = 0.222; data-to-parameter ratio = 16.4.

In the title compound, $C_{12}H_{15}N_3O_5S$, an intramolecular N—H···O hydrogen bond between the hydrazine unit and one of the carbonyl groups may influence the molecular conformation. In the crystal structure, intermolecular N—H···O hydrogen bonds, including one which is bifurcated, link the molecules into a two-dimensional network.

## Related literature

For background to sulfa drugs and their derivatives, see: Abbate *et al.* (2004); Badr (2008); Hanafy *et al.* (2007); Novinson *et al.* (1976); Supuran *et al.* (2003); Upadhyay *et al.* (2009); Zhong *et al.* (2007). For the synthesis of the title compound, see: Prakash & Gambhir (1964).



## Experimental

### Crystal data

$C_{12}H_{15}N_3O_5S$
$M_r$ = 313.33
Monoclinic, $P2_1/n$
$a$ = 7.490 (6) Å
$b$ = 14.819 (12) Å
$c$ = 12.689 (10) Å
$\beta$ = 95.219 (14)°
$V$ = 1402.6 (19) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.26 mm$^{-1}$
$T$ = 293 K
0.24 × 0.22 × 0.20 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.940, $T_{max}$ = 0.951
11777 measured reflections
3274 independent reflections
2177 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.052

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.222
$S$ = 0.87
3274 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.59 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O4 | 0.86 | 1.95 | 2.597 (5) | 131 |
| N3—H3$B$···O6$^{i}$ | 0.77 (5) | 2.33 (4) | 2.941 (6) | 137 (4) |
| N3—H3$B$···O5$^{i}$ | 0.77 (5) | 2.52 (5) | 3.208 (6) | 149 (5) |
| N3—H3$A$···O4$^{ii}$ | 0.85 (5) | 2.21 (5) | 3.003 (6) | 154 (4) |

Symmetry codes: (i) $-x, -y, -z+2$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; 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) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

KKU and SU are grateful to the CSIR, New Delhi, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2879).

## References

Abbate, F., Casini, A., Owa, T., Scozzafava, A. & Supuran, C. T. (2004). *Bioorg. Med. Chem. Lett.* **14**, 217–223.
Badr, E. E. (2008). *J. Disper. Sci. Technol.* **29**,1143–1149.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2008). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hanafy, A., Uno, J., Mitani, H., Kang, Y. & Mikami, Y. (2007). *Jpn J. Med. Mycol.* **48,** 47–50.
Novinson, T., Okabe, T., Robins, R. K. & Matthews, T. R. (1976). *J. Med. Chem.* **19**, 517–520.
Prakash, A. & Gambhir, I. R. (1964). *J. Indian Chem. Soc.* **41,** 133–136.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Supuran, C. T., Casini, A. & Scozzafava, A. (2003). *Med. Res. Rev.* **23**, 535–558.
Upadhyay, K. K., Upadhyay, S., Kumar, K. & Prasad, R. (2009). *J. Mol. Struct.* **927**, 60–68.
Zhong, Z., Chen, R., Xing, R., Chen, X., Liu, S., Guo, Z., Ji, X., Wang, L. & Li, P. (2007). *Carbohydr. Res.* **342**, 2390–2395.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o2499 [ **doi:10.1107/S1600536809036927** ]

# Ethyl 3-oxo-2-[(4-sulfamoylphenyl)hydrazono]butyrate

**K. K. Upadhyay, P. Rai, S. Upadhyay and M. Nethaji**

## Comment

Sulfadrugs and their derivatives have attracted much attention due to their wide spectrum of pharamaceutical (Abbate *et al.*, 2004; Badr, 2008; Supuran *et al.*, 2003) and biological applications (Hanafy *et al.*, 2007, Zhong *et al.*, 2007). Recently we reported details of a diazo derivative of sulfathiazole (Upadhyay *et al.*,2009) as a naked eye sensor for Hg(II) in DMSO. Although the title compound (I) has been reported in the literature (Prakash & Gambhir, 1964) its crystal structure determination has not been undertaken until now.

The molecular structure of the title compound is shown in Fig. 1. An intramolecular N—H···O hydrogen bond between the hydrazine unit and one carbonyl groups may influence the molecular conformation. In the crystal structure, intermolecular N—H···O hydrogen bonds, including one which which is bifurcated, link molecules in a two-dimensional network (see Fig. 2).

## Experimental

Compound (I) was synthesized using the literature procedure (Novinson *et al.*, 1976) as follows. Sulphanilamide (2 mmol, 344 mg) and sodium nitrite (~4 mmol, 300 mg) were dissolved separately in conc. HCl (2 ml) and distilled water (10 ml), respectively, followed by cooling on crushed ice. The cooled sodium nitrite solution was added to the sulphanilamide solution with constant stirring while maintaining the temperature. The resulting yellow solution was added to a mixture of ethyl aceto acetate (2 mmol, 0.25 ml) and sodium acetate (~37 mmol, 3 g) in distilled water (15 ml) with continuous stirring. The stirring was continued further for 2 h maintaining the temperature of the reaction vessel between 293–298 K. The resulting solids were filtered, washed with water, ethanol and finally, by diethyl ether. The crude product was recrystallized from a water–ethanol mixture (50% *v*/*v*) and dried *in vacuo*. Crystals were grown by layering a supersaturated solution of (I) in ethanol with diethylether and leaving for a few days.

Yield 76%. Spectroscopic anaylysis: $^{1}$HNMR (DMSO-$d_6$, TMS, δp.p.m.) 11.60 (1*H*, –HN—N=C<), 7.85–7.54 (m, 4H, Ar—H), 7.34 (s, 2H, $NH_2$),4.35–4.26 (2*H*, $CH_2$), 2.50–2.42(3*H*, $CH_3$ of $C_2H_5$), 1.33–1.26 (3*H*, $CH_3$). $^{13}$C NMR (DMSO-$d_6$, TMS, δ p.p.m.) 193.90 (>C=O),162.33 [C(OEt)=O], 145.16,138.31(C=C), 133.35, 127.36, 115.89, 114.76 (Ar—C), 61.37 (–$CH_2$), 25.28,13.83 (–$CH_3$).

## Refinement

H atoms were placed in calculated positions with C-H = 0.93 - 0.97Å and N-H = 0.86Å. They were included in the refinement in a riding-model approximation with $U_{iso}$(H) = 1.2$U_{eq}$(C,N) or 1.5$U_{eq}$(C) for methyl H atoms. The H atoms of the -$NH_2$ group were refined independently with isotropic displacement parameters.

## Figures

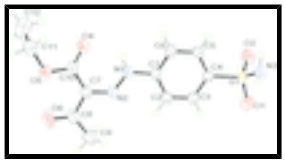


Fig. 1. The molecular structure of compound (I) showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

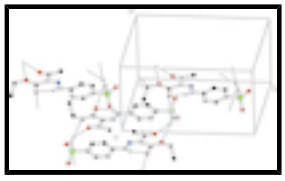


Fig. 2. Part of the crystal structure of (I) showing intramolecular and intermolecular hydrogen bonds as hashed lines.

## Ethyl 3-oxo-2-[(4-sulfamoylphenyl)hydrazono]butyrate

*Crystal data*

| | |
|---|---|
| $C_{12}H_{15}N_3O_5S$ | $F_{000}$ = 656 |
| $M_r$ = 313.33 | $D_x$ = 1.484 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Melting point: 398 K |
| Hall symbol: -P 2yn | Mo $K\alpha$ radiation, λ = 0.71073 Å |
| $a$ = 7.490 (6) Å | Cell parameters from 598 reflections |
| $b$ = 14.819 (12) Å | θ = 2.5–27.5º |
| $c$ = 12.689 (10) Å | μ = 0.26 mm$^{-1}$ |
| β = 95.219 (14)º | $T$ = 293 K |
| $V$ = 1402.6 (19) Å$^3$ | Rectangular, colourless |
| $Z$ = 4 | 0.24 × 0.22 × 0.20 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX diffractometer | 3274 independent reflections |
| Radiation source: fine-focus sealed tube | 2177 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.052 |
| Detector resolution: 0.3 pixels mm$^{-1}$ | $\theta_{max}$ = 28.2º |
| $T$ = 293 K | $\theta_{min}$ = 2.1º |
| ω scans | $h$ = −9→9 |
| Absorption correction: multi-scan (SADABS; Bruker, 2005) | $k$ = −17→19 |
| $T_{min}$ = 0.940, $T_{max}$ = 0.951 | $l$ = −16→16 |
| 11777 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full with fixed elements per cycle | Hydrogen site location: inferred from neighbouring sites |

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.070$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.222$ | $w = 1/[\sigma^2(F_o^2) + (0.1321P)^2 + 2.1941P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.87$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 3274 reflections | $\Delta\rho_{max} = 0.59$ e Å$^{-3}$ |
| 200 parameters | $\Delta\rho_{min} = -0.32$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.2438 (5) | 0.0661 (2) | 0.9456 (3) | 0.0391 (8) |
| C2 | 0.2126 (5) | 0.1343 (2) | 1.0159 (3) | 0.0429 (8) |
| H2 | 0.1983 | 0.1209 | 1.0862 | 0.051* |
| C3 | 0.2030 (5) | 0.2223 (2) | 0.9809 (3) | 0.0433 (8) |
| H3 | 0.1809 | 0.2684 | 1.0278 | 0.052* |
| C4 | 0.2258 (5) | 0.2424 (2) | 0.8772 (2) | 0.0372 (7) |
| C5 | 0.2609 (6) | 0.1745 (2) | 0.8081 (3) | 0.0518 (10) |
| H5 | 0.2790 | 0.1883 | 0.7384 | 0.062* |
| C6 | 0.2693 (6) | 0.0861 (2) | 0.8420 (3) | 0.0536 (10) |
| H6 | 0.2920 | 0.0401 | 0.7951 | 0.064* |
| C7 | 0.2498 (5) | −0.1296 (2) | 1.1080 (3) | 0.0400 (8) |
| C8 | 0.2482 (5) | −0.1381 (2) | 1.2239 (3) | 0.0451 (9) |
| C9 | 0.2452 (7) | −0.0539 (3) | 1.2876 (3) | 0.0659 (13) |
| H9A | 0.2411 | −0.0691 | 1.3609 | 0.099* |
| H9B | 0.1412 | −0.0190 | 1.2639 | 0.099* |
| H9C | 0.3513 | −0.0193 | 1.2791 | 0.099* |
| C10 | 0.2599 (5) | −0.2072 (2) | 1.0344 (3) | 0.0466 (9) |
| C11 | 0.2122 (7) | −0.3643 (3) | 0.9952 (4) | 0.0624 (12) |
| H11A | 0.1498 | −0.4147 | 1.0240 | 0.075* |
| H11B | 0.1458 | −0.3458 | 0.9296 | 0.075* |
| C12 | 0.3910 (8) | −0.3914 (3) | 0.9752 (5) | 0.0868 (17) |
| H12A | 0.4491 | −0.3427 | 0.9419 | 0.130* |
| H12B | 0.3847 | −0.4431 | 0.9294 | 0.130* |

| | | | | |
|---|---|---|---|---|
| H12C | 0.4583 | −0.4064 | 1.0409 | 0.130* |
| H3A | 0.042 (6) | 0.330 (3) | 0.688 (4) | 0.055 (13)* |
| H3B | −0.064 (6) | 0.334 (3) | 0.769 (4) | 0.049 (14)* |
| N1 | 0.2500 (4) | −0.02524 (19) | 0.9754 (2) | 0.0469 (8) |
| H1 | 0.2567 | −0.0667 | 0.9286 | 0.056* |
| N2 | 0.2454 (4) | −0.0464 (2) | 1.0745 (2) | 0.0417 (7) |
| N3 | 0.0211 (6) | 0.3532 (2) | 0.7471 (3) | 0.0481 (8) |
| O1 | 0.1619 (4) | 0.40903 (16) | 0.9163 (2) | 0.0565 (8) |
| O2 | 0.3416 (4) | 0.37652 (19) | 0.7692 (2) | 0.0581 (8) |
| O4 | 0.2924 (5) | −0.19684 (19) | 0.9443 (2) | 0.0711 (10) |
| O5 | 0.2233 (5) | −0.28696 (17) | 1.0729 (2) | 0.0620 (8) |
| O6 | 0.2547 (5) | −0.21135 (18) | 1.2667 (2) | 0.0703 (9) |
| S1 | 0.19753 (14) | 0.35341 (5) | 0.82925 (7) | 0.0425 (3) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.049 (2) | 0.0279 (16) | 0.0399 (17) | 0.0027 (14) | 0.0025 (15) | 0.0052 (13) |
| C2 | 0.062 (2) | 0.0381 (18) | 0.0278 (15) | 0.0020 (16) | 0.0020 (15) | 0.0040 (13) |
| C3 | 0.064 (2) | 0.0317 (17) | 0.0343 (16) | 0.0012 (15) | 0.0031 (15) | −0.0030 (13) |
| C4 | 0.052 (2) | 0.0285 (15) | 0.0305 (15) | −0.0009 (14) | 0.0022 (14) | 0.0018 (12) |
| C5 | 0.090 (3) | 0.0346 (18) | 0.0333 (17) | 0.0059 (19) | 0.0183 (18) | 0.0050 (14) |
| C6 | 0.094 (3) | 0.0294 (17) | 0.0392 (19) | 0.0098 (19) | 0.0148 (19) | 0.0008 (14) |
| C7 | 0.050 (2) | 0.0328 (17) | 0.0372 (17) | −0.0006 (14) | 0.0043 (15) | 0.0048 (13) |
| C8 | 0.055 (2) | 0.0417 (19) | 0.0383 (18) | −0.0101 (16) | 0.0017 (16) | 0.0054 (15) |
| C9 | 0.107 (4) | 0.049 (2) | 0.043 (2) | −0.015 (2) | 0.010 (2) | −0.0031 (18) |
| C10 | 0.064 (2) | 0.0351 (18) | 0.0416 (19) | 0.0099 (16) | 0.0103 (17) | 0.0076 (15) |
| C11 | 0.087 (3) | 0.043 (2) | 0.059 (3) | −0.006 (2) | 0.021 (2) | 0.0042 (18) |
| C12 | 0.091 (4) | 0.053 (3) | 0.112 (5) | 0.004 (3) | −0.009 (3) | 0.000 (3) |
| N1 | 0.073 (2) | 0.0307 (15) | 0.0374 (15) | 0.0042 (14) | 0.0051 (14) | 0.0063 (12) |
| N2 | 0.0537 (18) | 0.0352 (15) | 0.0361 (14) | −0.0017 (13) | 0.0032 (12) | 0.0083 (12) |
| N3 | 0.065 (2) | 0.0373 (17) | 0.0423 (18) | 0.0005 (16) | 0.0055 (16) | 0.0075 (14) |
| O1 | 0.088 (2) | 0.0294 (12) | 0.0521 (15) | −0.0011 (13) | 0.0082 (14) | −0.0060 (11) |
| O2 | 0.0708 (19) | 0.0462 (15) | 0.0591 (17) | −0.0080 (13) | 0.0152 (14) | 0.0121 (13) |
| O4 | 0.134 (3) | 0.0380 (15) | 0.0465 (15) | 0.0136 (16) | 0.0350 (18) | 0.0071 (12) |
| O5 | 0.113 (2) | 0.0307 (13) | 0.0460 (15) | 0.0019 (14) | 0.0270 (15) | 0.0046 (11) |
| O6 | 0.129 (3) | 0.0427 (16) | 0.0384 (14) | −0.0148 (17) | 0.0026 (16) | 0.0117 (12) |
| S1 | 0.0625 (6) | 0.0252 (4) | 0.0400 (5) | −0.0026 (4) | 0.0053 (4) | 0.0042 (3) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.379 (5) | C9—H9B | 0.9600 |
| C1—C2 | 1.382 (5) | C9—H9C | 0.9600 |
| C1—N1 | 1.404 (4) | C10—O4 | 1.200 (4) |
| C2—C3 | 1.378 (5) | C10—O5 | 1.317 (4) |
| C2—H2 | 0.9300 | C11—C12 | 1.443 (7) |
| C3—C4 | 1.375 (5) | C11—O5 | 1.509 (5) |
| C3—H3 | 0.9300 | C11—H11A | 0.9700 |
| C4—C5 | 1.376 (5) | C11—H11B | 0.9700 |

| | | | |
|---|---|---|---|
| C4—S1 | 1.760 (3) | C12—H12A | 0.9600 |
| C5—C6 | 1.379 (5) | C12—H12B | 0.9600 |
| C5—H5 | 0.9300 | C12—H12C | 0.9600 |
| C6—H6 | 0.9300 | N1—N2 | 1.300 (4) |
| C7—N2 | 1.304 (4) | N1—H1 | 0.8600 |
| C7—C8 | 1.477 (5) | N3—S1 | 1.607 (4) |
| C7—C10 | 1.488 (5) | N3—H3A | 0.85 (5) |
| C8—O6 | 1.213 (4) | N3—H3B | 0.77 (4) |
| C8—C9 | 1.488 (5) | O1—S1 | 1.423 (3) |
| C9—H9A | 0.9600 | O2—S1 | 1.419 (3) |
| C6—C1—C2 | 120.3 (3) | O4—C10—O5 | 122.4 (4) |
| C6—C1—N1 | 117.4 (3) | O4—C10—C7 | 121.7 (3) |
| C2—C1—N1 | 122.3 (3) | O5—C10—C7 | 115.9 (3) |
| C3—C2—C1 | 119.5 (3) | C12—C11—O5 | 109.3 (4) |
| C3—C2—H2 | 120.3 | C12—C11—H11A | 109.8 |
| C1—C2—H2 | 120.3 | O5—C11—H11A | 109.8 |
| C4—C3—C2 | 120.4 (3) | C12—C11—H11B | 109.8 |
| C4—C3—H3 | 119.8 | O5—C11—H11B | 109.8 |
| C2—C3—H3 | 119.8 | H11A—C11—H11B | 108.3 |
| C3—C4—C5 | 120.0 (3) | C11—C12—H12A | 109.5 |
| C3—C4—S1 | 120.8 (3) | C11—C12—H12B | 109.5 |
| C5—C4—S1 | 119.1 (3) | H12A—C12—H12B | 109.5 |
| C4—C5—C6 | 120.1 (3) | C11—C12—H12C | 109.5 |
| C4—C5—H5 | 119.9 | H12A—C12—H12C | 109.5 |
| C6—C5—H5 | 119.9 | H12B—C12—H12C | 109.5 |
| C1—C6—C5 | 119.7 (3) | N2—N1—C1 | 119.3 (3) |
| C1—C6—H6 | 120.1 | N2—N1—H1 | 120.3 |
| C5—C6—H6 | 120.1 | C1—N1—H1 | 120.3 |
| N2—C7—C8 | 113.7 (3) | N1—N2—C7 | 122.7 (3) |
| N2—C7—C10 | 121.9 (3) | S1—N3—H3A | 111 (3) |
| C8—C7—C10 | 124.4 (3) | S1—N3—H3B | 115 (3) |
| O6—C8—C7 | 121.2 (3) | H3A—N3—H3B | 113 (5) |
| O6—C8—C9 | 120.6 (3) | C10—O5—C11 | 116.1 (3) |
| C7—C8—C9 | 118.1 (3) | O2—S1—O1 | 118.86 (18) |
| C8—C9—H9A | 109.5 | O2—S1—N3 | 105.8 (2) |
| C8—C9—H9B | 109.5 | O1—S1—N3 | 107.7 (2) |
| H9A—C9—H9B | 109.5 | O2—S1—C4 | 109.76 (17) |
| C8—C9—H9C | 109.5 | O1—S1—C4 | 107.32 (16) |
| H9A—C9—H9C | 109.5 | N3—S1—C4 | 106.76 (17) |
| H9B—C9—H9C | 109.5 | | |
| C6—C1—C2—C3 | 1.5 (6) | N2—C7—C10—O5 | −165.3 (4) |
| N1—C1—C2—C3 | −178.1 (3) | C8—C7—C10—O5 | 16.2 (6) |
| C1—C2—C3—C4 | −0.6 (6) | C6—C1—N1—N2 | 172.9 (4) |
| C2—C3—C4—C5 | −0.9 (6) | C2—C1—N1—N2 | −7.5 (5) |
| C2—C3—C4—S1 | 175.3 (3) | C1—N1—N2—C7 | 179.7 (3) |
| C3—C4—C5—C6 | 1.5 (6) | C8—C7—N2—N1 | 178.4 (3) |
| S1—C4—C5—C6 | −174.8 (3) | C10—C7—N2—N1 | −0.2 (6) |
| C2—C1—C6—C5 | −1.0 (6) | O4—C10—O5—C11 | −4.0 (6) |

| | | | |
|---|---|---|---|
| N1—C1—C6—C5 | 178.7 (4) | C7—C10—O5—C11 | 173.1 (3) |
| C4—C5—C6—C1 | −0.5 (7) | C12—C11—O5—C10 | 77.1 (5) |
| N2—C7—C8—O6 | −178.8 (4) | C3—C4—S1—O2 | 133.9 (3) |
| C10—C7—C8—O6 | −0.3 (6) | C5—C4—S1—O2 | −49.9 (4) |
| N2—C7—C8—C9 | −1.0 (5) | C3—C4—S1—O1 | 3.4 (4) |
| C10—C7—C8—C9 | 177.5 (4) | C5—C4—S1—O1 | 179.6 (3) |
| N2—C7—C10—O4 | 11.8 (6) | C3—C4—S1—N3 | −111.9 (3) |
| C8—C7—C10—O4 | −166.7 (4) | C5—C4—S1—N3 | 64.3 (4) |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O4 | 0.86 | 1.95 | 2.597 (5) | 131 |
| N3—H3B···O6$^{i}$ | 0.77 (5) | 2.33 (4) | 2.941 (6) | 137 (4) |
| N3—H3B···O5$^{i}$ | 0.77 (5) | 2.52 (5) | 3.208 (6) | 149 (5) |
| N3—H3A···O4$^{ii}$ | 0.85 (5) | 2.21 (5) | 3.003 (6) | 154 (4) |

Symmetry codes: (i) −*x*, −*y*, −*z*+2; (ii) −*x*+1/2, *y*+1/2, −*z*+3/2.

**Fig. 1**

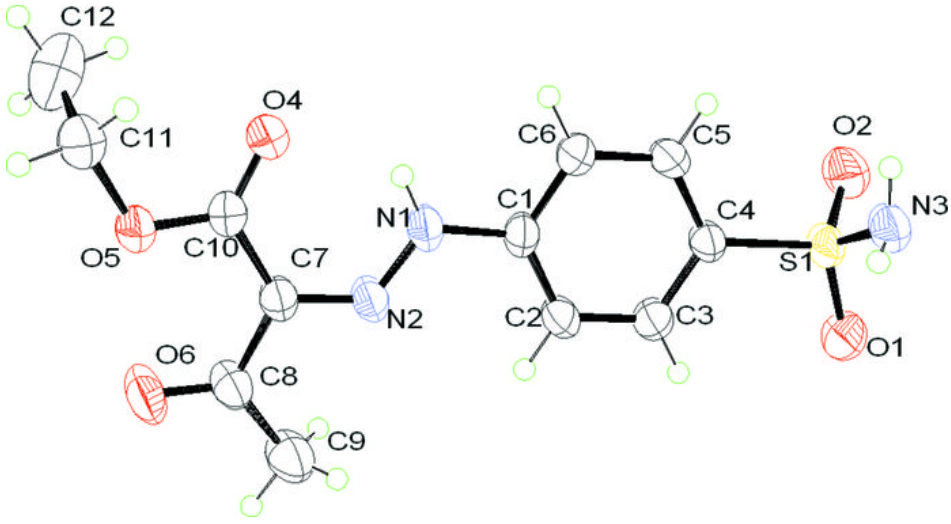


**Fig. 2**