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# Methyl (2*Z*)-2-({*N*-[2-(hydroxymethyl)-phenyl]-4-methylbenzenesulfonamido}-methyl)-3-phenylprop-2-enoate

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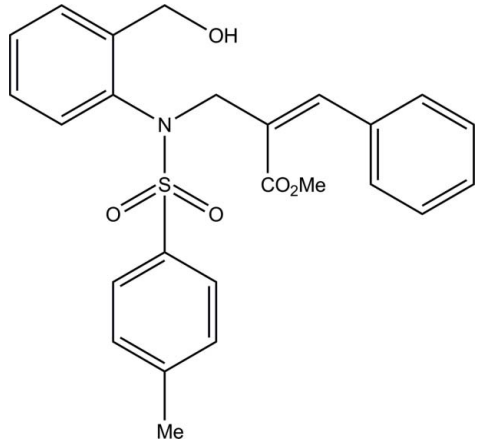
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.042; $wR$ factor = 0.127; data-to-parameter ratio = 24.0.

In the title compound, $C_{25}H_{25}NO_5S$, the O atom of the hydroxy group is disordered over two positions, with occupancies of 0.820 (2) and 0.180 (2). The sulfonyl-bound benzene ring forms dihedral angles of 31.8 (1) and 60.7 (1)°, respectively, with the hydroxymethylbenzene and phenyl rings. The molecular conformation is stabilized by an intramolecular O—H···O hydrogen bond, generating an $S$(8) ring motif. The crystal packing is stabilized by intermolecular C—H···O hydrogen bonds and C—H···$\pi$ interactions.

## Related literature

For background to the pharmacological uses of sulfonamides, see: Korolkovas (1988); Mandell & Sande (1992). For resonance effects of acrylate, see: Merlino (1971); Varghese *et al.* (1986). For related structures, see: Madhanraj *et al.* (2011); Aziz-ur-Rehman *et al.* (2010). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).




## Experimental

### Crystal data

$C_{25}H_{25}NO_5S$
$M_r$ = 451.52
Triclinic, $P\overline{1}$
$a$ = 7.9528 (3) Å
$b$ = 9.5396 (3) Å
$c$ = 15.3299 (5) Å
$\alpha$ = 88.253 (2)°
$\beta$ = 83.571 (1)°
$\gamma$ = 76.215 (2)°
$V$ = 1122.42 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.18 mm$^{-1}$
$T$ = 293 K
0.23 × 0.21 × 0.16 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.959, $T_{max}$ = 0.971
25861 measured reflections
7132 independent reflections
5255 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.127
$S$ = 1.05
7132 reflections
297 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

*Cg*1 and *Cg*2 are the centroids of the C18–C23 and C8–C13 benzene rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1*A*—H1*A*···O2 | 0.82 | 2.23 | 2.958 (2) | 147 |
| C9—H9···*Cg*1 | 0.93 | 2.80 | 3.545 (2) | 138 |
| C5—H5···O4$^{i}$ | 0.93 | 2.54 | 3.429 (2) | 160 |
| C14—H14*C*···O2$^{ii}$ | 0.96 | 2.55 | 3.359 (2) | 143 |
| C12—H12···*Cg*1$^{iii}$ | 0.93 | 2.72 | 3.506 (2) | 143 |
| C20—H20···*Cg*2$^{iv}$ | 0.93 | 2.92 | 3.593 (2) | 130 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia (1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5779).

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# supplementary materials

*Acta Cryst.* (2012). E**68**, o418-o419 [ **doi:10.1107/S1600536812000864** ]

# Methyl (2*Z*)-2-({*N*-[2-(hydroxymethyl)phenyl]-4-methylbenzenesulfonamido}methyl)-3-phenylprop-2-enoate

**R. Madhanraj, S. Murugavel, D. Kannan and M. Bakthadoss**

## Comment

Sulfonamide drugs are widely used for the treatment of certain infections caused by Gram-positive and Gram-negative microorganisms, some fungi, and certain protozoa (Korolkovas, 1988, Mandell & Sande, 1992). In view of this biological importance, the crystal structure of the title compound has been determined and the results are presented here.

Fig. 1. shows a displacement ellipsoid plot of (I), with the atom numbering scheme. The O1 atom of the hydroxyl group is disordered over two positions with occupancies 0.820 (2):0.180 (2). The S1 atom shows a distorted tetrahedral geometry, with O2—S1—O3[120.0 (1)°] and N1—S1—C8[108.6 (1)°] angles deviating from ideal tetrahedral values. The significant difference in length of the C24—O5 = 1.329 (2) Å and C25—O5 = 1.442 (2) Å bonds is attributed to a partial contribution from the $O^-$–C = $O^+$–C resonance structure of the O4═C24—O5—C25 group (Merlino, 1971). This feature, commonly observed in the carboxylic ester group of the substituents in various compounds gives average values of 1.340 Å and 1.447 Å respectively for these bonds (Varghese *et al.,* 1986). The sum of bond angles around N1 (346°) indicates that N1 is in $sp^2$ hybridization. The sulfonyl-bound benzene (C8–C13) ring forms dihedral angles of 31.8 (1)° and 60.7 (1)°, respectively, with the hydroxymethyl benzene (C1–C6) and benzene (C18–C23) rings. The dihedral angle between hydroxymethyl benzene and benzene rings is 41.2 (1)°. The geometric parameters of the title molecule agrees well with those reported for similar structures (Madhanraj *et al.,* 2011, Aziz-ur-Rehman *et al.,* 2010).

The molecule is stabilized by an intramolecular O1A-H1A···O2 hydrogen bond generating an S(8) ring motif (Bernstein *et al.,* 1995) and an intramolecular C-H···π interaction between a sulfonyl–bound benzene H atom and a benzene (C18–C23) ring with a C9—H9···Cg1 seperation of 2.80 Å (Table 1; Cg1 is the centroid of the C18–C23 benzene ring). The crystal packing is stabilized by intermolecular C—H···O hydrogen bonds. The molecules at *x, y, z* and *1-x, 1-y, 1-z* are linked by C5—H5···O4 hydrogen bonds into cyclic centrosymmetric $R_2^2(18)$ dimers (Fig. 2). These dimers are linked by C14—H14C···O2 hydrogen bonds forming supramolecular tapes running along the [100] directions (Fig. 3). The crystal packing is further stabilized by C—H···π interactions, the first one between a sulfonyl–bound benzene H atom and the benzene ring (C18–C23) of an adjacent molecule, with a C12—H12···Cg1$^{iii}$ seperation of 2.72 Å and the second one between a benzene H atom and the benzene ring (C8–C13) of a neighbouring molecule, with a C20—H20···Cg2$^{iv}$ seperation of 2.92 Å ( Table 1 and Fig. 4; Cg1 and Cg2 are the centroids of the (C18–C23) benzene and (C8–C13) benzene rings, respectively. symmetry code as in Fig. 3).

## Experimental

A solution of *N*-(2-(hydroxymethyl)phenyl)-(4-methylbenzene)sulfonamide (1 mmol, 0.277 g) and potassium carbonate (1.5 mmol, 0.207 g) in acetonitrile solvent was stirred for 15 minutes at room temperature. To this solution, (*z*)-methyl-2-(bromomethyl)-3-phenylprop-2-enoate (1.2 mmol, 0.304 g) was added dropwise till the addition is complete. After the completion of the reaction, as indicated by TLC, acetonitile was evaporated. ETOAc (15 ml) and water (15 ml) were added to the crude

mass. The organic layer was dried over anhydrous sodium sulfate. Removal of solvent led to the crude product, which was purified through pad of silica gel (100-200 mesh) using ethylacetate and hexanes (1:9) as solvents. The pure title compound was obtained as a colourless solid (0.435 g, 96% yield). Recrystallization was carried out using ethylacetate as solvent.

### Refinement

Atom O1 is disordered over two positions (O1A/O1B) with refined occpancies of 0.820 (2) and 0.180 (2). The C—O distances of the disordered hydroxyl group were restrained to 1.40 Å. All the H atoms were positioned geometrically, (C–H = 0.93–0.97 Å and O—H = 0.82 Å) constrained to ride on their parent atom, with $U_{iso}$(H) =1.5$U_{eq}$ for methyl H atoms and 1.2$U_{eq}$(C) for other H atoms.

### Figures

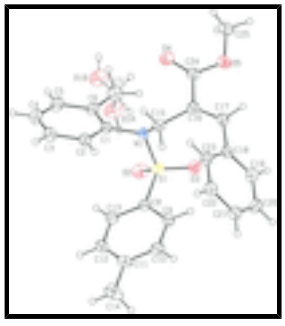


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids. H atoms are presented as a small spheres of arbitrary radius. The disordered component is shown.

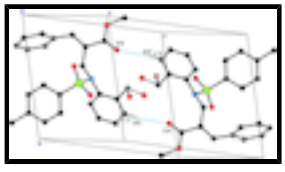


Fig. 2. Part of the crystal structure of the title compound showing C—H···O intermolecular hydrogen bonds (dotted lines) generating $R^2_2(18)$ centrosymmetric dimer. [Symmetry code: (i) *1-x, 1-y, 1-z*].

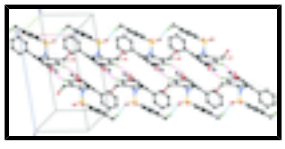


Fig. 3. Supramolecular tape formation in (I) whereby centrosymmetrc $R_2^2(18)$ dimeric aggregates sustained by C—H···O (magenta dashed lines) contacts are linked *via* C—H···O contacts (cyan dashed lines) along [1 0 0].

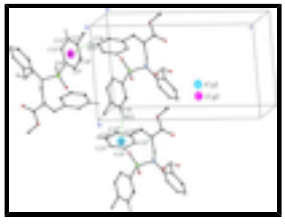


Fig. 4. A view of the C—H···π interactions, in the crystal structure of the title compound. Cg1 and Cg2 are the centroids of the (C18–C23) benzene and (C8–C13) benzene rings, respectively. [Symmetry code: (iii) *x, 1+y, z*; (iv) *-x, 1-y, -z*.]

### Methyl (2*Z*)-2-({*N*-[2-(hydroxymethyl)phenyl]-4- methylbenzenesulfonamido}methyl)-3-phenylprop-2-enoate

*Crystal data*

| | |
|---|---|
| $C_{25}H_{25}NO_5S$ | $Z = 2$ |
| $M_r = 451.52$ | $F(000) = 476$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.336$ Mg m$^{-3}$ |

| | |
|---|---|
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 7.9528$ (3) Å | Cell parameters from 7307 reflections |
| $b = 9.5396$ (3) Å | $\theta = 1.3–31.3°$ |
| $c = 15.3299$ (5) Å | $\mu = 0.18$ mm$^{-1}$ |
| $\alpha = 88.253$ (2)° | $T = 293$ K |
| $\beta = 83.571$ (1)° | Block, colourless |
| $\gamma = 76.215$ (2)° | 0.23 × 0.21 × 0.16 mm |
| $V = 1122.42$ (7) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 7132 independent reflections |
| Radiation source: fine-focus sealed tube | 5255 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.027$ |
| Detector resolution: 10.0 pixels mm$^{-1}$ | $\theta_{max} = 31.3°$, $\theta_{min} = 1.3°$ |
| ω scans | $h = -11\rightarrow11$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) | $k = -13\rightarrow13$ |
| $T_{min} = 0.959$, $T_{max} = 0.971$ | $l = -21\rightarrow22$ |
| 25861 measured reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.127$ | H-atom parameters constrained |
| $S = 1.05$ | $w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.1756P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 7132 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 297 parameters | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| 2 restraints | $\Delta\rho_{min} = -0.31$ 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 $F^2 > 2$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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.20025 (16) | 0.58467 (13) | 0.36181 (8) | 0.0327 (2) | |
| C2 | 0.03176 (18) | 0.65951 (14) | 0.39145 (9) | 0.0401 (3) | |
| H2 | −0.0629 | 0.6363 | 0.3699 | 0.048* | |
| C3 | 0.0045 (2) | 0.76791 (16) | 0.45268 (10) | 0.0511 (4) | |
| H3 | −0.1082 | 0.8175 | 0.4725 | 0.061* | |
| C4 | 0.1448 (3) | 0.80219 (17) | 0.48427 (11) | 0.0575 (4) | |
| H4 | 0.1271 | 0.8762 | 0.5248 | 0.069* | |
| C5 | 0.3121 (2) | 0.72685 (16) | 0.45592 (10) | 0.0515 (4) | |
| H5 | 0.4056 | 0.7507 | 0.4783 | 0.062* | |
| C6 | 0.34408 (18) | 0.61594 (14) | 0.39460 (9) | 0.0393 (3) | |
| C8 | 0.04825 (17) | 0.61769 (13) | 0.16515 (8) | 0.0343 (3) | |
| C9 | −0.05349 (19) | 0.55105 (14) | 0.12048 (9) | 0.0410 (3) | |
| H9 | −0.0160 | 0.4536 | 0.1064 | 0.049* | |
| C10 | −0.2116 (2) | 0.63118 (16) | 0.09707 (10) | 0.0469 (3) | |
| H10 | −0.2801 | 0.5867 | 0.0672 | 0.056* | |
| C11 | −0.26927 (19) | 0.77612 (15) | 0.11741 (10) | 0.0443 (3) | |
| C12 | −0.1653 (2) | 0.84054 (14) | 0.16182 (10) | 0.0460 (3) | |
| H12 | −0.2029 | 0.9380 | 0.1758 | 0.055* | |
| C13 | −0.0071 (2) | 0.76351 (14) | 0.18575 (9) | 0.0423 (3) | |
| H13 | 0.0615 | 0.8085 | 0.2152 | 0.051* | |
| C14 | −0.4407 (2) | 0.8624 (2) | 0.09128 (14) | 0.0681 (5) | |
| H14A | −0.4480 | 0.8451 | 0.0306 | 0.102* | |
| H14B | −0.4493 | 0.9632 | 0.0997 | 0.102* | |
| H14C | −0.5343 | 0.8338 | 0.1268 | 0.102* | |
| C15 | 0.11357 (16) | 0.36554 (12) | 0.31857 (8) | 0.0339 (2) | |
| H15A | 0.0152 | 0.3908 | 0.2843 | 0.041* | |
| H15B | 0.0683 | 0.3736 | 0.3801 | 0.041* | |
| C16 | 0.20914 (16) | 0.21190 (13) | 0.29938 (8) | 0.0350 (3) | |
| C17 | 0.16244 (17) | 0.12583 (14) | 0.24467 (9) | 0.0393 (3) | |
| H17 | 0.2324 | 0.0328 | 0.2378 | 0.047* | |
| C18 | 0.01129 (17) | 0.16300 (13) | 0.19390 (9) | 0.0364 (3) | |
| C19 | 0.0349 (2) | 0.14001 (16) | 0.10405 (10) | 0.0459 (3) | |
| H19 | 0.1452 | 0.0979 | 0.0768 | 0.055* | |
| C20 | −0.1044 (2) | 0.17926 (17) | 0.05454 (10) | 0.0530 (4) | |
| H20 | −0.0870 | 0.1655 | −0.0059 | 0.064* | |
| C21 | −0.2684 (2) | 0.23862 (17) | 0.09474 (11) | 0.0528 (4) | |
| H21 | −0.3616 | 0.2661 | 0.0613 | 0.063* | |
| C22 | −0.29490 (19) | 0.25740 (16) | 0.18413 (11) | 0.0482 (3) | |
| H22 | −0.4065 | 0.2948 | 0.2114 | 0.058* | |
| C23 | −0.15595 (18) | 0.22084 (15) | 0.23353 (9) | 0.0403 (3) | |
| H23 | −0.1744 | 0.2350 | 0.2939 | 0.048* | |
| C24 | 0.36113 (17) | 0.15603 (14) | 0.34979 (9) | 0.0385 (3) | |
| C25 | 0.6052 (2) | −0.03467 (18) | 0.36773 (12) | 0.0552 (4) | |
| H25A | 0.6903 | 0.0223 | 0.3604 | 0.083* | |
| H25B | 0.6568 | −0.1303 | 0.3459 | 0.083* | |

| | | | | | |
|---|---|---|---|---|---|
| H25C | 0.5648 | −0.0397 | 0.4289 | 0.083* | |
| N1 | 0.22546 (13) | 0.47007 (10) | 0.29838 (7) | 0.0317 (2) | |
| O2 | 0.36596 (14) | 0.61308 (12) | 0.18924 (7) | 0.0517 (3) | |
| O3 | 0.29601 (13) | 0.38840 (11) | 0.14434 (6) | 0.0463 (2) | |
| O4 | 0.39010 (15) | 0.21719 (12) | 0.41165 (8) | 0.0565 (3) | |
| O5 | 0.46033 (14) | 0.03080 (11) | 0.31967 (7) | 0.0522 (3) | |
| S1 | 0.24992 (4) | 0.51837 (3) | 0.19400 (2) | 0.03568 (9) | |
| C7 | 0.52803 (19) | 0.53288 (18) | 0.36886 (11) | 0.0509 (4) | |
| H7A | 0.5850 | 0.5067 | 0.4217 | 0.061* | 0.820 (2) |
| H7B | 0.5248 | 0.4441 | 0.3405 | 0.061* | 0.820 (2) |
| H7C | 0.5370 | 0.4333 | 0.3870 | 0.061* | 0.180 (2) |
| H7D | 0.5488 | 0.5337 | 0.3053 | 0.061* | 0.180 (2) |
| O1A | 0.62756 (19) | 0.60607 (18) | 0.31313 (10) | 0.0630 (4) | 0.820 (2) |
| H1A | 0.5817 | 0.6274 | 0.2677 | 0.095* | 0.820 (2) |
| O1B | 0.6587 (8) | 0.5806 (8) | 0.4002 (4) | 0.0630 (4) | 0.180 (2) |
| H1B | 0.6916 | 0.5313 | 0.4426 | 0.095* | 0.180 (2) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0352 (6) | 0.0318 (5) | 0.0329 (6) | −0.0103 (5) | −0.0073 (5) | 0.0012 (4) |
| C2 | 0.0375 (7) | 0.0411 (7) | 0.0410 (7) | −0.0077 (5) | −0.0050 (5) | −0.0004 (5) |
| C3 | 0.0522 (9) | 0.0453 (7) | 0.0490 (8) | 0.0005 (7) | −0.0006 (7) | −0.0060 (6) |
| C4 | 0.0780 (12) | 0.0469 (8) | 0.0479 (9) | −0.0131 (8) | −0.0075 (8) | −0.0130 (7) |
| C5 | 0.0619 (10) | 0.0499 (8) | 0.0506 (8) | −0.0235 (7) | −0.0166 (7) | −0.0052 (6) |
| C6 | 0.0404 (7) | 0.0410 (6) | 0.0412 (7) | −0.0157 (6) | −0.0117 (5) | 0.0007 (5) |
| C8 | 0.0372 (6) | 0.0345 (6) | 0.0325 (6) | −0.0101 (5) | −0.0069 (5) | 0.0034 (5) |
| C9 | 0.0461 (7) | 0.0339 (6) | 0.0444 (7) | −0.0090 (5) | −0.0124 (6) | −0.0010 (5) |
| C10 | 0.0471 (8) | 0.0449 (7) | 0.0533 (8) | −0.0144 (6) | −0.0185 (6) | 0.0033 (6) |
| C11 | 0.0422 (7) | 0.0425 (7) | 0.0469 (8) | −0.0079 (6) | −0.0077 (6) | 0.0134 (6) |
| C12 | 0.0571 (9) | 0.0318 (6) | 0.0468 (8) | −0.0062 (6) | −0.0066 (6) | 0.0045 (5) |
| C13 | 0.0543 (8) | 0.0351 (6) | 0.0408 (7) | −0.0139 (6) | −0.0114 (6) | 0.0013 (5) |
| C14 | 0.0516 (10) | 0.0588 (10) | 0.0901 (14) | −0.0034 (8) | −0.0194 (9) | 0.0235 (9) |
| C15 | 0.0310 (6) | 0.0329 (6) | 0.0398 (6) | −0.0107 (5) | −0.0060 (5) | 0.0000 (5) |
| C16 | 0.0316 (6) | 0.0341 (6) | 0.0408 (6) | −0.0093 (5) | −0.0079 (5) | 0.0025 (5) |
| C17 | 0.0337 (6) | 0.0343 (6) | 0.0502 (8) | −0.0065 (5) | −0.0083 (5) | −0.0041 (5) |
| C18 | 0.0352 (6) | 0.0316 (6) | 0.0449 (7) | −0.0099 (5) | −0.0096 (5) | −0.0053 (5) |
| C19 | 0.0446 (8) | 0.0464 (7) | 0.0468 (8) | −0.0111 (6) | −0.0021 (6) | −0.0097 (6) |
| C20 | 0.0671 (10) | 0.0552 (8) | 0.0417 (8) | −0.0198 (8) | −0.0144 (7) | −0.0038 (6) |
| C21 | 0.0543 (9) | 0.0470 (8) | 0.0627 (10) | −0.0127 (7) | −0.0288 (8) | 0.0006 (7) |
| C22 | 0.0341 (7) | 0.0471 (7) | 0.0645 (9) | −0.0079 (6) | −0.0120 (6) | −0.0089 (7) |
| C23 | 0.0368 (7) | 0.0434 (7) | 0.0430 (7) | −0.0122 (5) | −0.0061 (5) | −0.0076 (5) |
| C24 | 0.0355 (6) | 0.0372 (6) | 0.0448 (7) | −0.0107 (5) | −0.0099 (5) | 0.0052 (5) |
| C25 | 0.0428 (8) | 0.0558 (9) | 0.0626 (10) | 0.0017 (7) | −0.0180 (7) | 0.0089 (7) |
| N1 | 0.0315 (5) | 0.0334 (5) | 0.0332 (5) | −0.0115 (4) | −0.0074 (4) | −0.0002 (4) |
| O2 | 0.0446 (6) | 0.0671 (7) | 0.0514 (6) | −0.0299 (5) | −0.0065 (5) | 0.0116 (5) |
| O3 | 0.0406 (5) | 0.0528 (6) | 0.0407 (5) | −0.0009 (4) | −0.0032 (4) | −0.0082 (4) |
| O4 | 0.0584 (7) | 0.0547 (6) | 0.0587 (7) | −0.0063 (5) | −0.0291 (5) | −0.0066 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O5 | 0.0452 (6) | 0.0466 (5) | 0.0611 (7) | 0.0038 (5) | −0.0211 (5) | −0.0034 (5) |
| S1 | 0.03119 (16) | 0.04236 (17) | 0.03454 (16) | −0.01077 (12) | −0.00391 (11) | 0.00105 (12) |
| C7 | 0.0365 (7) | 0.0594 (9) | 0.0609 (9) | −0.0160 (7) | −0.0129 (6) | −0.0015 (7) |
| O1A | 0.0481 (8) | 0.0859 (10) | 0.0618 (9) | −0.0297 (7) | −0.0067 (6) | 0.0066 (8) |
| O1B | 0.0481 (8) | 0.0859 (10) | 0.0618 (9) | −0.0297 (7) | −0.0067 (6) | 0.0066 (8) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.3926 (18) | C16—C24 | 1.4897 (17) |
| C1—C6 | 1.3966 (17) | C17—C18 | 1.4717 (18) |
| C1—N1 | 1.4484 (15) | C17—H17 | 0.9300 |
| C2—C3 | 1.380 (2) | C18—C19 | 1.3863 (19) |
| C2—H2 | 0.9300 | C18—C23 | 1.3900 (19) |
| C3—C4 | 1.375 (2) | C19—C20 | 1.385 (2) |
| C3—H3 | 0.9300 | C19—H19 | 0.9300 |
| C4—C5 | 1.381 (2) | C20—C21 | 1.375 (2) |
| C4—H4 | 0.9300 | C20—H20 | 0.9300 |
| C5—C6 | 1.395 (2) | C21—C22 | 1.374 (2) |
| C5—H5 | 0.9300 | C21—H21 | 0.9300 |
| C6—C7 | 1.504 (2) | C22—C23 | 1.3799 (19) |
| C8—C9 | 1.3869 (18) | C22—H22 | 0.9300 |
| C8—C13 | 1.3901 (18) | C23—H23 | 0.9300 |
| C8—S1 | 1.7535 (13) | C24—O4 | 1.1998 (17) |
| C9—C10 | 1.3859 (19) | C24—O5 | 1.3288 (16) |
| C9—H9 | 0.9300 | C25—O5 | 1.4419 (17) |
| C10—C11 | 1.383 (2) | C25—H25A | 0.9600 |
| C10—H10 | 0.9300 | C25—H25B | 0.9600 |
| C11—C12 | 1.385 (2) | C25—H25C | 0.9600 |
| C11—C14 | 1.505 (2) | N1—S1 | 1.6548 (10) |
| C12—C13 | 1.379 (2) | O2—S1 | 1.4323 (10) |
| C12—H12 | 0.9300 | O3—S1 | 1.4247 (10) |
| C13—H13 | 0.9300 | C7—O1B | 1.367 (4) |
| C14—H14A | 0.9600 | C7—O1A | 1.385 (2) |
| C14—H14B | 0.9600 | C7—H7A | 0.9700 |
| C14—H14C | 0.9600 | C7—H7B | 0.9700 |
| C15—N1 | 1.4915 (15) | C7—H7C | 0.9700 |
| C15—C16 | 1.5025 (17) | C7—H7D | 0.9700 |
| C15—H15A | 0.9700 | O1A—H7D | 1.0542 |
| C15—H15B | 0.9700 | O1A—H1A | 0.8200 |
| C16—C17 | 1.3315 (18) | O1B—H1B | 0.8200 |
| C2—C1—C6 | 120.74 (12) | C20—C19—H19 | 119.8 |
| C2—C1—N1 | 119.33 (11) | C18—C19—H19 | 119.8 |
| C6—C1—N1 | 119.89 (11) | C21—C20—C19 | 120.00 (14) |
| C3—C2—C1 | 120.35 (13) | C21—C20—H20 | 120.0 |
| C3—C2—H2 | 119.8 | C19—C20—H20 | 120.0 |
| C1—C2—H2 | 119.8 | C22—C21—C20 | 120.15 (14) |
| C4—C3—C2 | 119.66 (14) | C22—C21—H21 | 119.9 |
| C4—C3—H3 | 120.2 | C20—C21—H21 | 119.9 |
| C2—C3—H3 | 120.2 | C21—C22—C23 | 120.04 (14) |

| | | | |
|---|---|---|---|
| C3—C4—C5 | 120.15 (14) | C21—C22—H22 | 120.0 |
| C3—C4—H4 | 119.9 | C23—C22—H22 | 120.0 |
| C5—C4—H4 | 119.9 | C22—C23—C18 | 120.64 (13) |
| C4—C5—C6 | 121.63 (15) | C22—C23—H23 | 119.7 |
| C4—C5—H5 | 119.2 | C18—C23—H23 | 119.7 |
| C6—C5—H5 | 119.2 | O4—C24—O5 | 123.50 (12) |
| C5—C6—C1 | 117.44 (13) | O4—C24—C16 | 123.47 (12) |
| C5—C6—C7 | 119.57 (13) | O5—C24—C16 | 113.03 (11) |
| C1—C6—C7 | 122.96 (12) | O5—C25—H25A | 109.5 |
| C9—C8—C13 | 120.41 (12) | O5—C25—H25B | 109.5 |
| C9—C8—S1 | 119.85 (10) | H25A—C25—H25B | 109.5 |
| C13—C8—S1 | 119.73 (10) | O5—C25—H25C | 109.5 |
| C10—C9—C8 | 119.29 (12) | H25A—C25—H25C | 109.5 |
| C10—C9—H9 | 120.4 | H25B—C25—H25C | 109.5 |
| C8—C9—H9 | 120.4 | C1—N1—C15 | 115.15 (10) |
| C11—C10—C9 | 121.12 (13) | C1—N1—S1 | 115.85 (8) |
| C11—C10—H10 | 119.4 | C15—N1—S1 | 115.24 (8) |
| C9—C10—H10 | 119.4 | C24—O5—C25 | 116.57 (12) |
| C10—C11—C12 | 118.59 (13) | O3—S1—O2 | 119.99 (7) |
| C10—C11—C14 | 120.71 (14) | O3—S1—N1 | 106.47 (6) |
| C12—C11—C14 | 120.69 (14) | O2—S1—N1 | 105.76 (6) |
| C13—C12—C11 | 121.53 (13) | O3—S1—C8 | 107.61 (6) |
| C13—C12—H12 | 119.2 | O2—S1—C8 | 107.99 (6) |
| C11—C12—H12 | 119.2 | N1—S1—C8 | 108.60 (6) |
| C12—C13—C8 | 119.05 (13) | O1B—C7—O1A | 60.6 (3) |
| C12—C13—H13 | 120.5 | O1B—C7—C6 | 117.5 (3) |
| C8—C13—H13 | 120.5 | O1A—C7—C6 | 114.78 (14) |
| C11—C14—H14A | 109.5 | O1B—C7—H7A | 49.6 |
| C11—C14—H14B | 109.5 | O1A—C7—H7A | 108.6 |
| H14A—C14—H14B | 109.5 | C6—C7—H7A | 108.6 |
| C11—C14—H14C | 109.5 | O1B—C7—H7B | 132.9 |
| H14A—C14—H14C | 109.5 | O1A—C7—H7B | 108.6 |
| H14B—C14—H14C | 109.5 | C6—C7—H7B | 108.6 |
| N1—C15—C16 | 112.99 (10) | H7A—C7—H7B | 107.5 |
| N1—C15—H15A | 109.0 | O1B—C7—H7C | 108.7 |
| C16—C15—H15A | 109.0 | O1A—C7—H7C | 135.8 |
| N1—C15—H15B | 109.0 | C6—C7—H7C | 108.0 |
| C16—C15—H15B | 109.0 | H7A—C7—H7C | 65.9 |
| H15A—C15—H15B | 107.8 | H7B—C7—H7C | 44.2 |
| C17—C16—C24 | 120.28 (12) | O1B—C7—H7D | 107.1 |
| C17—C16—C15 | 124.55 (11) | O1A—C7—H7D | 49.4 |
| C24—C16—C15 | 115.12 (11) | C6—C7—H7D | 107.9 |
| C16—C17—C18 | 126.57 (12) | H7A—C7—H7D | 143.1 |
| C16—C17—H17 | 116.7 | H7B—C7—H7D | 64.9 |
| C18—C17—H17 | 116.7 | H7C—C7—H7D | 107.2 |
| C19—C18—C23 | 118.62 (12) | C7—O1A—H7D | 44.3 |
| C19—C18—C17 | 119.55 (12) | C7—O1A—H1A | 109.5 |
| C23—C18—C17 | 121.83 (12) | H7D—O1A—H1A | 71.9 |
| C20—C19—C18 | 120.50 (14) | C7—O1B—H1B | 109.5 |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 1.2 (2) | C21—C22—C23—C18 | −0.9 (2) |
| N1—C1—C2—C3 | 179.20 (12) | C19—C18—C23—C22 | −1.3 (2) |
| C1—C2—C3—C4 | 0.2 (2) | C17—C18—C23—C22 | 178.71 (13) |
| C2—C3—C4—C5 | −1.1 (3) | C17—C16—C24—O4 | −165.13 (15) |
| C3—C4—C5—C6 | 0.6 (3) | C15—C16—C24—O4 | 12.34 (19) |
| C4—C5—C6—C1 | 0.8 (2) | C17—C16—C24—O5 | 14.26 (19) |
| C4—C5—C6—C7 | −177.31 (15) | C15—C16—C24—O5 | −168.28 (11) |
| C2—C1—C6—C5 | −1.67 (19) | C2—C1—N1—C15 | −46.81 (15) |
| N1—C1—C6—C5 | −179.64 (12) | C6—C1—N1—C15 | 131.19 (12) |
| C2—C1—C6—C7 | 176.36 (13) | C2—C1—N1—S1 | 91.87 (12) |
| N1—C1—C6—C7 | −1.61 (19) | C6—C1—N1—S1 | −90.13 (12) |
| C13—C8—C9—C10 | −0.4 (2) | C16—C15—N1—C1 | −139.33 (11) |
| S1—C8—C9—C10 | −179.19 (11) | C16—C15—N1—S1 | 81.74 (11) |
| C8—C9—C10—C11 | 0.1 (2) | O4—C24—O5—C25 | 2.6 (2) |
| C9—C10—C11—C12 | 0.1 (2) | C16—C24—O5—C25 | −176.78 (12) |
| C9—C10—C11—C14 | 179.67 (15) | C1—N1—S1—O3 | 172.29 (8) |
| C10—C11—C12—C13 | 0.0 (2) | C15—N1—S1—O3 | −49.07 (10) |
| C14—C11—C12—C13 | −179.51 (15) | C1—N1—S1—O2 | 43.60 (10) |
| C11—C12—C13—C8 | −0.4 (2) | C15—N1—S1—O2 | −177.76 (8) |
| C9—C8—C13—C12 | 0.6 (2) | C1—N1—S1—C8 | −72.09 (10) |
| S1—C8—C13—C12 | 179.34 (11) | C15—N1—S1—C8 | 66.55 (9) |
| N1—C15—C16—C17 | −122.13 (14) | C9—C8—S1—O3 | 15.29 (13) |
| N1—C15—C16—C24 | 60.53 (14) | C13—C8—S1—O3 | −163.49 (11) |
| C24—C16—C17—C18 | 176.84 (13) | C9—C8—S1—O2 | 146.17 (11) |
| C15—C16—C17—C18 | −0.4 (2) | C13—C8—S1—O2 | −32.61 (13) |
| C16—C17—C18—C19 | 127.54 (16) | C9—C8—S1—N1 | −99.59 (12) |
| C16—C17—C18—C23 | −52.5 (2) | C13—C8—S1—N1 | 81.63 (12) |
| C23—C18—C19—C20 | 2.5 (2) | C5—C6—C7—O1B | −6.8 (4) |
| C17—C18—C19—C20 | −177.55 (13) | C1—C6—C7—O1B | 175.2 (4) |
| C18—C19—C20—C21 | −1.4 (2) | C5—C6—C7—O1A | −75.15 (19) |
| C19—C20—C21—C22 | −0.8 (2) | C1—C6—C7—O1A | 106.87 (16) |
| C20—C21—C22—C23 | 2.0 (2) | | |

*Hydrogen-bond geometry (Å, °)*

Cg1 and Cg2 are the centroids of the C18–C23 and C8–C13 benzene rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1A—H1A···O2 | 0.82 | 2.23 | 2.958 (2) | 147. |
| C9—H9···Cg1 | 0.93 | 2.80 | 3.545 (2) | 138. |
| C5—H5···O4$^{i}$ | 0.93 | 2.54 | 3.429 (2) | 160. |
| C14—H14C···O2$^{ii}$ | 0.96 | 2.55 | 3.359 (2) | 143. |
| C12—H12···Cg1$^{iii}$ | 0.93 | 2.72 | 3.506 (2) | 143. |
| C20—H20···Cg2$^{iv}$ | 0.93 | 2.92 | 3.593 (2) | 130. |

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

**Fig. 1**

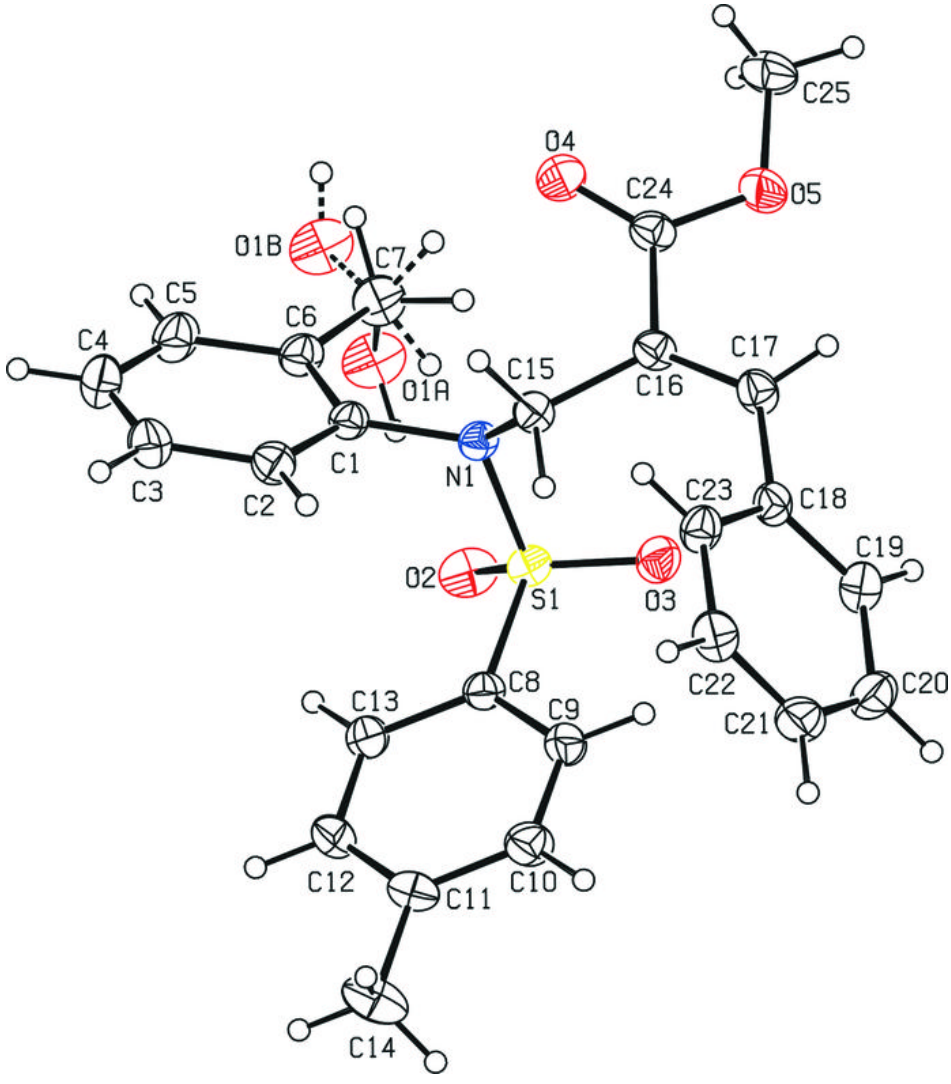

C25
O4
C24
O5
O1B
C7
C5
C6
C4
O1A
C15
C16
C17
C1
N1
C3
C2
C23
C18
C19
O2
S1
O3
C22
C20
C8
C13
C9
C21
C12
C11
C10
C14

**Fig. 2**

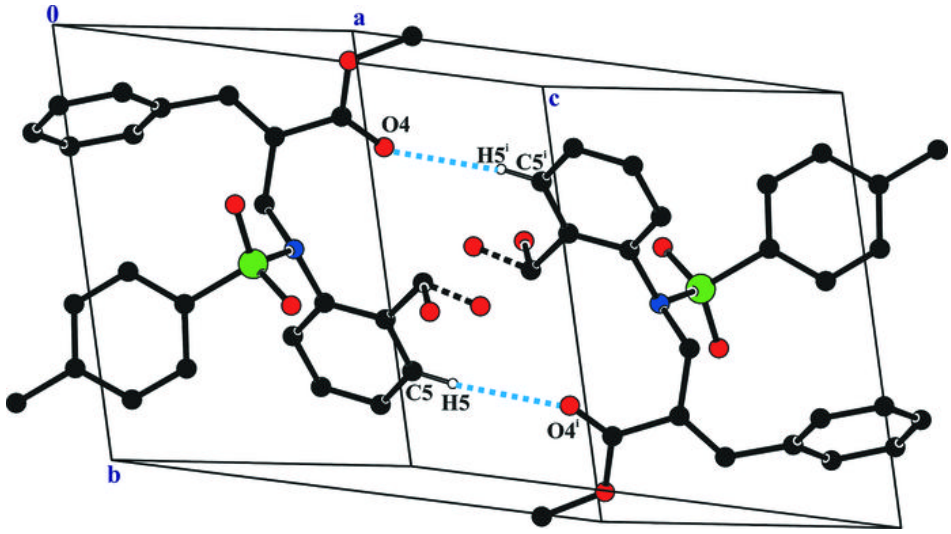


Fig. 3

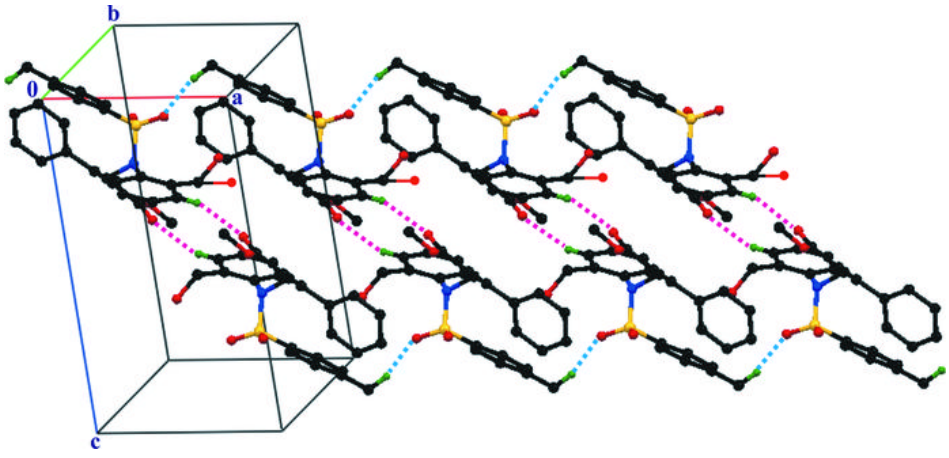
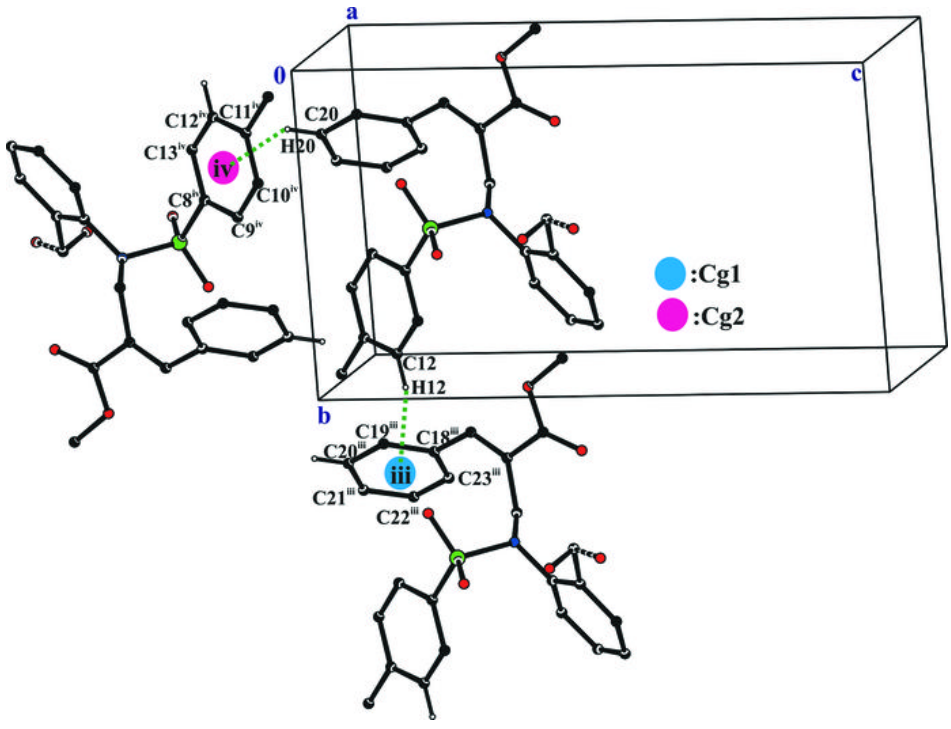


**Fig. 4**


a
0
c
b
C12iv
C11iv
C13iv
C8iv
C9iv
C10iv
C20
H20
iv
:Cg1
:Cg2
C12
H12
C19iii
C18iii
C20iii
C23iii
C21iii
C22iii
iii