

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1-(4-Methoxyphenylsulfonyl)-5-methyl-5-phenylimidazolidine-2,4-dione

Abid Hussain,^a Shahid Hameed^{a*} and Helen Stoeckli-Evans^b^aDepartment of Chemistry, Quaid-I-Azam University, Islamabad 45320, Pakistan, and ^bInstitute of Physics, University of Neuchâtel, rue Emile-Argand 11, CH-2009 Neuchâtel, Switzerland

Correspondence e-mail: shameed@qau.edu.pk

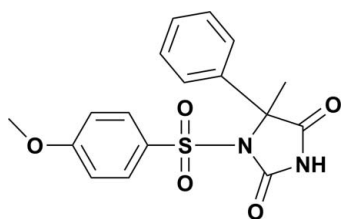
Received 29 April 2009; accepted 29 April 2009

Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.029; wR factor = 0.058; data-to-parameter ratio = 13.8.

The title compound, $\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_5\text{S}$, crystallized in the chiral monoclinic space group $P2_1$, with two enantiomeric molecules (A and B) in the asymmetric unit. It is composed of a methylimidazolidine-2,4-dione unit substituted with a phenyl group and a 4-methoxyphenylsulfonyl group. The benzene ring mean planes are inclined to one another by 22.20 (14)° in molecule A and by 15.82 (13)° in molecule B . In the crystal structure, the A and B molecules are linked by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming centrosymmetric dimers. A number of $\text{C}-\text{H}\cdots\text{O}$ interactions are also present in the crystal structure, leading to the formation of a three-dimensional network.

Related literature

For the applications of imidazolidine-2,4-diones, see: Thenmozhiyal *et al.* (2004); Zhang *et al.* (2004). For the activity of sulfonylimidazolidine-2,4-diones, see: Kashif, Ahmad & Hameed (2008); Ahmad *et al.* (2000, 2002); Murakami *et al.* (1997). For related crystal structures, see: Hussain *et al.* (2009); Kashif, Hussain *et al.* (2008).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_5\text{S}$
 $M_r = 360.38$
 Monoclinic, $P2_1$

$a = 6.2314$ (6) Å
 $b = 17.5694$ (12) Å
 $c = 15.5892$ (16) Å

$\beta = 99.373$ (12)°
 $V = 1683.9$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.22$ mm⁻¹
 $T = 173$ K
 $0.38 \times 0.30 \times 0.19$ mm

Data collection

Stoe IPDS diffractometer
 Absorption correction: none
 13521 measured reflections

6394 independent reflections
 4309 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.058$
 $S = 0.81$
 6394 reflections
 465 parameters
 3 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³
 Absolute structure: Flack (1983), 2987 Friedel pairs
 Flack parameter: 0.06 (5)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2N}\cdots\text{O8}^{\text{i}}$	0.91 (2)	1.95 (2)	2.851 (3)	171 (2)
$\text{N4}-\text{H4N}\cdots\text{O3}^{\text{ii}}$	0.92 (2)	1.89 (2)	2.800 (3)	171 (2)
$\text{C5}-\text{H5}\cdots\text{O2}^{\text{i}}$	0.95	2.36	3.218 (3)	150
$\text{C10}-\text{H10A}\cdots\text{O8}$	0.98	2.56	3.437 (3)	149
$\text{C10}-\text{H10B}\cdots\text{O2}$	0.98	2.45	3.055 (3)	120
$\text{C12}-\text{H12}\cdots\text{O2}$	0.95	2.52	2.905 (3)	104
$\text{C13}-\text{H13}\cdots\text{O9}^{\text{iii}}$	0.95	2.43	3.287 (3)	150
$\text{C22}-\text{H22}\cdots\text{O7}^{\text{ii}}$	0.95	2.42	3.351 (3)	165
$\text{C24}-\text{H24}\cdots\text{O8}^{\text{iv}}$	0.95	2.56	3.492 (3)	166
$\text{C27}-\text{H27A}\cdots\text{O7}$	0.98	2.52	3.126 (4)	120
$\text{C29}-\text{H29}\cdots\text{O7}$	0.95	2.54	2.916 (3)	104
$\text{C30}-\text{H30}\cdots\text{O4}^{\text{v}}$	0.95	2.38	3.159 (3)	138
$\text{C34}-\text{H34}\cdots\text{O8}$	0.95	2.56	3.291 (3)	134

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

Data collection: *EXPOSE* in *IPDS-I* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS-I*; data reduction: *INTEGRATE* in *IPDS-I*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2941).

References

- Ahmad, I., Hameed, S., Duddeck, H., Lenzen, S., Rustenbeck, I. & Ahmad, R. (2002). *Z. Naturforsch. Teil B*, **57**, 349–354.
 Ahmad, R., Jabeen, R., Zia-ul-Haq, M., Nadeem, H., Duddeck, H. & Verspohl, E. J. (2000). *Z. Naturforsch. Teil B* **55**, 203–207.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Hussain, A., Hameed, S. & Stoeckli-Evans, H. (2009). *Acta Cryst.* **E65**, o858–o859.
 Kashif, M. K., Ahmad, I. & Hameed, S. (2008). *ARKIVOC*, **xvi**, 311–317.
 Kashif, M. K., Hussain, A., Khawar Rauf, M., Ebihara, M. & Hameed, S. (2008). *Acta Cryst.* **E64**, o444.
 Murakami, N., Ohta, M., Kato, K., Nakayama, K., Mizota, M., Miwa, I. & Okuda, J. (1997). *Arzneim. Forsch.* **47**, 1222–1225.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
 Stoe & Cie (2000). *IPDS-I*. Stoe & Cie, Darmstadt, Germany.

Thenmozhiyal, J. C., Wong, P. T.-H. & Chui, W.-K. (2004). *J. Med. Chem.* **47**, 1527–1535.

Zhang, X., Allan, G. F., Sbriscia, T., Linton, O., Lundeen, S. G. & Sui, Z. (2004). *Bioorg. Med. Chem. Lett.* **14**, 6107–6111.

supplementary materials

Acta Cryst. (2009). E65, o1207-o1208 [doi:10.1107/S1600536809016092]

1-(4-Methoxyphenylsulfonyl)-5-methyl-5-phenylimidazolidine-2,4-dione

A. Hussain, S. Hameed and H. Stoeckli-Evans

Comment

Imidazolidine-2,4-diones have found applications as androgen receptor antagonists (Zhang *et al.*, 2004) and possess strong anticonvulsant activity (Thenmozhiyal *et al.*, 2004). Sulfonyl derivatives of imidazolidine-2,4-diones on the other hand, are finding utility as inhibitors of aldose reductase (Murakami *et al.*, 1997). The potential of this class of compounds as hypoglycemic agents has already been reported from our laboratory (Kashif, Ahmad & Hameed, 2008; Ahmad *et al.*, 2002, 2000). In continuation of our work on sulfonyl cyclic ureas (Hussain *et al.*, 2009; Kashif, Hussain *et al.*, 2008), the title compound was synthesized taking an imidazolidine-2,4-dione as the scaffold.

The molecular structure of the title compound is illustrated in Fig. 1, and full geometrical details are available in the archived CIF. It crystallized in the chiral monoclinic space group $P2_1$ with two enantiomeric molecules (A and B) in the asymmetric unit. It is composed of a methylimidazolidine-2,4-dione moiety substituted with a phenyl group and a 4-methoxyphenylsulfonyl group. The bond distances and angles of the two independent molecules are very similar to those observed in 1-(4-Chlorophenylsulfonyl)-5-(4-fluorophenyl)-5-methylimidazolidine-2,4-dione (Hussain *et al.*, 2009), which also crystallized with two independent molecules per asymmetric unit but in the centrosymmetric triclinic space group P-1.

Both molecules A and B are U-shaped with slightly different conformations, as can be seen in the auto-fit view, Fig. 2 (Spek, 2009). The best fit was obtained for inverted molecule B on molecule A (25 non-H atoms) with the weighted and unit weight r.m.s. fits being 0.687 and 0.460 Å, respectively. The benzene ring mean planes are inclined to one another by 22.20 (14)° in molecule A and 15.82 (13)° in molecule B. This is different to the situation in the compound mentioned above where the same angles are 6.07 (8)° and 8.67 (8)°, for molecules A and B, respectively (Hussain *et al.*, 2009).

In the crystal structure of the title compound the A and B molecules are linked by N—H···O hydrogen bonds to form a dimer-like arrangement (Fig. 3 and Table 1). There are also a number of C—H···O interactions present in the crystal structure, which leads to the formation of a three-dimensional network (Fig. 4 and Table 1).

Experimental

5-Methyl-5-phenylimidazolidine-2,4-dione (4.8 mmol) in CH_2Cl_2 was stirred with triethyl amine (4.8 mmol) and catalytic amounts of DMAP. 4-Methoxybenzene sulfonyl chloride (5.8 mmol) in CH_2Cl_2 was added dropwise. The reaction mixture was stirred continually at 298 K until the reaction was complete (monitored by TLC). The reaction mixture was then diluted with 1 N HCl and extracted with CH_2Cl_2 (3 × 25 ml). The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. Crystallization of the residue in ethyl acetate afforded colourless rod-like crystals of the title compound, suitable for X-ray analysis.

Refinement

The NH H-atoms were located in difference Fourier maps and freely refined. The H-atoms bonded to C were included in calculated positions [$C-H = 0.95 - 0.98 \text{ \AA}$] and treated as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$.

Figures

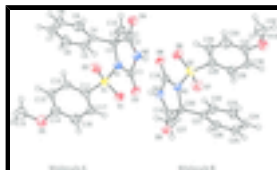


Fig. 1. A view of the molecular structure of the two independent molecules of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

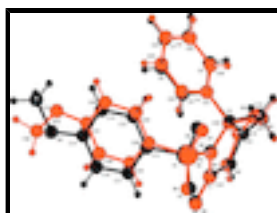


Fig. 2. An auto-fit view (Spek, 2009) of the two independent molecules (A black; B red) in the title compound.

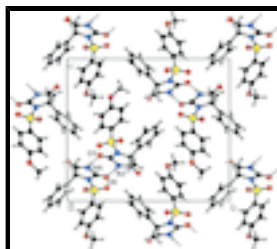


Fig. 3. A view along the a axis of the crystal packing in the title compound, showing the $N-H\cdots O$ hydrogen bonds which link the two independent molecules (A and B) to form a dimer-like arrangement (see Table 1 for details).

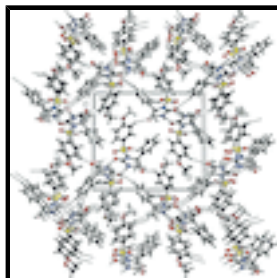


Fig. 4. A perspective view along the a axis of the crystal packing in the title compound showing the $N-H\cdots O$ and $C-H\cdots O$ hydrogen bonds, which link the molecules to form a three-dimensional network (see Table 1 for details).

1-(4-Methoxyphenylsulfonyl)-5-methyl-5-phenylimidazolidine-2,4-dione

Crystal data

$C_{17}H_{16}N_2O_5S$

$M_r = 360.38$

Monoclinic, $P2_1$

Hall symbol: $P\ 2y_b$

$a = 6.2314 (6) \text{ \AA}$

$b = 17.5694 (12) \text{ \AA}$

$F_{000} = 752$

$D_x = 1.421 \text{ Mg m}^{-3}$

Melting point = 471–473 K

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 8000 reflections

$\theta = 2.6\text{--}26.0^\circ$

$c = 15.5892 (16) \text{ \AA}$
 $\beta = 99.373 (12)^\circ$
 $V = 1683.9 (3) \text{ \AA}^3$
 $Z = 4$

$\mu = 0.22 \text{ mm}^{-1}$
 $T = 173 \text{ K}$
 Rod, colourless
 $0.38 \times 0.30 \times 0.19 \text{ mm}$

Data collection

Stoe IPDS diffractometer
 Radiation source: fine-focus sealed tube
 Monochromator: graphite
 $T = 173 \text{ K}$
 φ rotation scans
 Absorption correction: none
 13521 measured reflections
 6394 independent reflections

4309 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\text{max}} = 26.1^\circ$
 $\theta_{\text{min}} = 2.7^\circ$
 $h = -7 \rightarrow 7$
 $k = -20 \rightarrow 21$
 $l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.058$
 $S = 0.81$
 6394 reflections
 465 parameters
 3 restraints
 Primary atom site location: structure-invariant direct methods
 Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.024P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick, 2008),
 $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0042 (4)
 Absolute structure: Flack (1983), 2987 Friedel pairs
 Flack parameter: 0.06 (5)

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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.

supplementary materials

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.49288 (9)	0.27479 (4)	0.41621 (4)	0.0322 (2)
O1	0.5317 (3)	0.19861 (10)	0.39102 (11)	0.0374 (6)
O2	0.6619 (3)	0.33014 (11)	0.42511 (11)	0.0414 (6)
O3	0.0791 (3)	0.20334 (11)	0.32063 (11)	0.0388 (6)
O4	-0.0034 (3)	0.43832 (11)	0.19756 (12)	0.0497 (7)
O5	0.0979 (3)	0.26824 (12)	0.73090 (12)	0.0622 (8)
N1	0.3022 (3)	0.30995 (11)	0.33861 (12)	0.0273 (7)
N2	-0.0122 (3)	0.31655 (12)	0.24927 (13)	0.0340 (8)
C1	0.1190 (4)	0.26883 (16)	0.30444 (15)	0.0299 (8)
C2	0.0728 (4)	0.38756 (16)	0.24475 (17)	0.0330 (9)
C3	0.2862 (4)	0.39125 (14)	0.30845 (15)	0.0285 (8)
C4	0.2584 (4)	0.44517 (15)	0.38246 (15)	0.0289 (8)
C5	0.0795 (4)	0.43753 (17)	0.42345 (17)	0.0409 (10)
C6	0.0477 (5)	0.48556 (18)	0.49017 (19)	0.0480 (10)
C7	0.1927 (5)	0.54235 (18)	0.51559 (19)	0.0519 (11)
C8	0.3705 (6)	0.55014 (19)	0.4763 (2)	0.0616 (11)
C9	0.4043 (5)	0.50230 (18)	0.40945 (18)	0.0483 (10)
C10	0.4663 (4)	0.41178 (16)	0.25751 (18)	0.0419 (10)
C11	0.3765 (4)	0.27369 (15)	0.51029 (15)	0.0303 (8)
C12	0.4167 (4)	0.33274 (15)	0.56842 (16)	0.0395 (9)
C13	0.3248 (5)	0.33334 (16)	0.64261 (17)	0.0447 (10)
C14	0.1936 (4)	0.27364 (18)	0.65885 (16)	0.0438 (10)
C15	0.1345 (7)	0.32823 (19)	0.7938 (2)	0.0826 (16)
C16	0.1511 (4)	0.21413 (17)	0.60013 (18)	0.0453 (10)
C17	0.2418 (4)	0.21460 (15)	0.52550 (16)	0.0360 (9)
S2	0.17941 (9)	0.16666 (4)	0.07688 (4)	0.0343 (2)
O6	0.1557 (3)	0.24522 (10)	0.09316 (11)	0.0443 (7)
O7	0.0040 (3)	0.11541 (11)	0.07948 (12)	0.0434 (6)
O8	0.5996 (3)	0.24274 (10)	0.16846 (11)	0.0382 (6)
O9	0.7248 (3)	0.00425 (10)	0.27716 (11)	0.0390 (6)
O10	0.5175 (3)	0.11964 (11)	-0.24698 (12)	0.0523 (7)
N3	0.3788 (3)	0.13476 (12)	0.15370 (13)	0.0285 (7)
N4	0.7059 (3)	0.12965 (12)	0.23550 (12)	0.0295 (7)
C18	0.5652 (4)	0.17648 (16)	0.18420 (15)	0.0290 (8)
C19	0.6295 (4)	0.05719 (16)	0.23806 (15)	0.0300 (8)
C20	0.4033 (4)	0.05427 (15)	0.18445 (15)	0.0286 (8)
C21	0.4028 (4)	-0.00248 (14)	0.10977 (15)	0.0284 (8)
C22	0.5752 (4)	-0.00181 (16)	0.06382 (15)	0.0354 (9)
C23	0.5797 (4)	-0.05236 (18)	-0.00387 (17)	0.0442 (10)
C24	0.4127 (5)	-0.10347 (16)	-0.02589 (17)	0.0466 (10)
C25	0.2398 (5)	-0.10367 (18)	0.01867 (19)	0.0488 (11)
C26	0.2350 (4)	-0.05315 (16)	0.08642 (17)	0.0390 (9)
C27	0.2452 (5)	0.03531 (17)	0.24687 (19)	0.0424 (10)
C28	0.2780 (4)	0.15486 (15)	-0.02036 (16)	0.0318 (8)
C29	0.1934 (4)	0.09875 (15)	-0.07862 (16)	0.0338 (8)

C30	0.2755 (4)	0.08912 (15)	-0.15410 (16)	0.0366 (9)
C31	0.4463 (4)	0.13473 (15)	-0.17102 (16)	0.0375 (9)
C32	0.6969 (5)	0.1640 (2)	-0.26728 (19)	0.0633 (13)
C33	0.5329 (4)	0.18981 (17)	-0.11248 (17)	0.0487 (10)
C34	0.4462 (4)	0.19955 (17)	-0.03783 (17)	0.0453 (10)
H2N	-0.143 (3)	0.2979 (16)	0.2234 (17)	0.057 (9)*
H5	-0.02360	0.39850	0.40540	0.0490*
H6	-0.07520	0.47900	0.51840	0.0580*
H7	0.16960	0.57630	0.56060	0.0620*
H8	0.47330	0.58910	0.49500	0.0740*
H9	0.52860	0.50890	0.38210	0.0580*
H10A	0.47220	0.37370	0.21200	0.0630*
H10B	0.60600	0.41290	0.29690	0.0630*
H10C	0.43690	0.46200	0.23070	0.0630*
H12	0.50880	0.37340	0.55720	0.0470*
H13	0.35120	0.37450	0.68240	0.0540*
H15A	0.29050	0.33220	0.81610	0.1240*
H15B	0.08230	0.37640	0.76630	0.1240*
H15C	0.05580	0.31710	0.84180	0.1240*
H16	0.05990	0.17330	0.61140	0.0540*
H17	0.21190	0.17440	0.48450	0.0430*
H4N	0.835 (3)	0.1490 (15)	0.2635 (16)	0.053 (9)*
H22	0.69070	0.03350	0.07890	0.0420*
H23	0.69830	-0.05180	-0.03520	0.0530*
H24	0.41700	-0.13860	-0.07190	0.0560*
H25	0.12350	-0.13850	0.00300	0.0590*
H26	0.11500	-0.05340	0.11700	0.0470*
H27A	0.09600	0.03590	0.21510	0.0640*
H27B	0.25970	0.07320	0.29360	0.0640*
H27C	0.27880	-0.01530	0.27190	0.0640*
H29	0.07880	0.06710	-0.06620	0.0410*
H30	0.21600	0.05140	-0.19480	0.0440*
H32A	0.82400	0.15630	-0.22210	0.0950*
H32B	0.65720	0.21800	-0.27000	0.0950*
H32C	0.73170	0.14780	-0.32360	0.0950*
H33	0.65070	0.22050	-0.12370	0.0580*
H34	0.50350	0.23790	0.00250	0.0540*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0318 (3)	0.0308 (4)	0.0350 (3)	0.0037 (3)	0.0081 (3)	0.0046 (3)
O1	0.0456 (10)	0.0279 (11)	0.0406 (10)	0.0154 (8)	0.0127 (8)	0.0030 (8)
O2	0.0309 (9)	0.0458 (13)	0.0475 (11)	-0.0090 (9)	0.0062 (8)	0.0103 (10)
O3	0.0469 (10)	0.0229 (12)	0.0444 (11)	-0.0020 (8)	0.0006 (8)	-0.0035 (9)
O4	0.0650 (12)	0.0332 (12)	0.0448 (11)	0.0118 (10)	-0.0093 (9)	0.0037 (10)
O5	0.0954 (15)	0.0495 (14)	0.0531 (12)	0.0007 (12)	0.0464 (11)	-0.0022 (11)
N1	0.0297 (10)	0.0221 (13)	0.0300 (11)	0.0007 (8)	0.0046 (9)	-0.0009 (9)

supplementary materials

N2	0.0392 (13)	0.0240 (14)	0.0355 (12)	0.0028 (10)	-0.0037 (10)	-0.0047 (10)
C1	0.0356 (13)	0.0231 (16)	0.0305 (13)	0.0035 (13)	0.0038 (10)	-0.0040 (12)
C2	0.0422 (15)	0.0260 (17)	0.0308 (15)	0.0074 (12)	0.0060 (12)	-0.0041 (12)
C3	0.0352 (13)	0.0192 (15)	0.0315 (14)	0.0022 (10)	0.0068 (11)	0.0020 (11)
C4	0.0350 (13)	0.0224 (15)	0.0289 (13)	0.0001 (11)	0.0043 (11)	0.0009 (11)
C5	0.0381 (15)	0.0392 (19)	0.0462 (17)	-0.0060 (13)	0.0092 (13)	-0.0137 (14)
C6	0.0500 (17)	0.055 (2)	0.0410 (16)	0.0091 (15)	0.0136 (13)	-0.0104 (14)
C7	0.083 (2)	0.035 (2)	0.0374 (16)	0.0077 (16)	0.0091 (16)	-0.0062 (14)
C8	0.091 (2)	0.046 (2)	0.0474 (19)	-0.0311 (18)	0.0097 (18)	-0.0166 (16)
C9	0.0581 (17)	0.041 (2)	0.0479 (17)	-0.0172 (15)	0.0152 (14)	-0.0016 (15)
C10	0.0473 (16)	0.0388 (19)	0.0435 (16)	0.0055 (13)	0.0187 (13)	0.0089 (13)
C11	0.0329 (12)	0.0241 (15)	0.0346 (13)	-0.0004 (12)	0.0078 (10)	0.0022 (12)
C12	0.0529 (16)	0.0253 (17)	0.0408 (15)	-0.0063 (12)	0.0091 (13)	0.0031 (13)
C13	0.0721 (19)	0.0257 (17)	0.0390 (15)	-0.0021 (14)	0.0175 (14)	-0.0040 (12)
C14	0.0571 (17)	0.0390 (18)	0.0404 (15)	0.0067 (15)	0.0234 (13)	0.0032 (14)
C15	0.154 (4)	0.048 (2)	0.060 (2)	0.012 (2)	0.060 (2)	-0.0070 (18)
C16	0.0553 (17)	0.0359 (19)	0.0496 (17)	-0.0069 (13)	0.0235 (13)	-0.0008 (14)
C17	0.0407 (14)	0.0306 (17)	0.0384 (14)	-0.0018 (12)	0.0117 (11)	-0.0037 (12)
S2	0.0317 (3)	0.0336 (4)	0.0382 (4)	0.0030 (3)	0.0073 (3)	-0.0010 (3)
O6	0.0518 (11)	0.0318 (13)	0.0487 (11)	0.0145 (9)	0.0062 (9)	-0.0040 (9)
O7	0.0269 (9)	0.0545 (13)	0.0501 (11)	-0.0038 (9)	0.0101 (8)	0.0012 (10)
O8	0.0482 (10)	0.0232 (12)	0.0417 (11)	-0.0054 (8)	0.0026 (8)	-0.0007 (8)
O9	0.0500 (10)	0.0287 (12)	0.0367 (10)	0.0005 (9)	0.0024 (8)	0.0032 (9)
O10	0.0612 (12)	0.0570 (15)	0.0427 (11)	-0.0077 (10)	0.0202 (9)	-0.0060 (10)
N3	0.0325 (10)	0.0199 (13)	0.0335 (11)	-0.0013 (8)	0.0064 (9)	-0.0012 (9)
N4	0.0337 (12)	0.0226 (13)	0.0320 (12)	-0.0019 (10)	0.0050 (9)	-0.0015 (9)
C18	0.0330 (13)	0.0269 (17)	0.0284 (13)	0.0023 (12)	0.0085 (10)	-0.0048 (12)
C19	0.0430 (15)	0.0258 (16)	0.0231 (13)	-0.0004 (12)	0.0109 (11)	-0.0014 (11)
C20	0.0325 (13)	0.0228 (15)	0.0325 (14)	-0.0023 (10)	0.0114 (11)	-0.0041 (11)
C21	0.0323 (13)	0.0204 (15)	0.0324 (14)	-0.0014 (11)	0.0050 (11)	-0.0017 (11)
C22	0.0345 (13)	0.0394 (18)	0.0334 (14)	-0.0009 (12)	0.0090 (11)	-0.0068 (12)
C23	0.0547 (17)	0.0437 (19)	0.0353 (15)	0.0077 (15)	0.0105 (13)	-0.0057 (14)
C24	0.077 (2)	0.0272 (18)	0.0341 (15)	0.0070 (15)	0.0050 (15)	-0.0063 (13)
C25	0.0674 (19)	0.0307 (19)	0.0450 (17)	-0.0152 (14)	-0.0007 (15)	-0.0053 (14)
C26	0.0448 (15)	0.0308 (17)	0.0421 (16)	-0.0109 (13)	0.0088 (12)	-0.0017 (13)
C27	0.0479 (16)	0.0352 (19)	0.0494 (18)	-0.0030 (13)	0.0242 (14)	-0.0004 (13)
C28	0.0326 (12)	0.0291 (16)	0.0331 (13)	0.0000 (11)	0.0037 (10)	0.0007 (11)
C29	0.0328 (13)	0.0308 (16)	0.0371 (14)	-0.0056 (11)	0.0033 (11)	0.0006 (12)
C30	0.0426 (15)	0.0297 (17)	0.0360 (15)	-0.0056 (12)	0.0023 (12)	-0.0042 (12)
C31	0.0429 (14)	0.0403 (18)	0.0303 (14)	-0.0028 (13)	0.0088 (11)	0.0005 (12)
C32	0.0559 (18)	0.088 (3)	0.0501 (17)	-0.0130 (19)	0.0212 (14)	0.0026 (18)
C33	0.0520 (16)	0.055 (2)	0.0409 (16)	-0.0264 (14)	0.0131 (13)	-0.0059 (14)
C34	0.0553 (17)	0.0441 (19)	0.0362 (15)	-0.0192 (14)	0.0069 (13)	-0.0085 (13)

Geometric parameters (Å, °)

S1—O1	1.4266 (19)	C7—H7	0.9500
S1—O2	1.424 (2)	C8—H8	0.9500
S1—N1	1.670 (2)	C9—H9	0.9500

S1—C11	1.739 (2)	C10—H10B	0.9800
S2—N3	1.676 (2)	C10—H10A	0.9800
S2—O6	1.4155 (19)	C10—H10C	0.9800
S2—O7	1.422 (2)	C12—H12	0.9500
S2—C28	1.738 (3)	C13—H13	0.9500
O3—C1	1.212 (3)	C15—H15B	0.9800
O4—C2	1.203 (3)	C15—H15A	0.9800
O5—C14	1.358 (3)	C15—H15C	0.9800
O5—C15	1.432 (4)	C16—H16	0.9500
O8—C18	1.216 (3)	C17—H17	0.9500
O9—C19	1.213 (3)	C19—C20	1.518 (3)
O10—C31	1.357 (3)	C20—C27	1.530 (4)
O10—C32	1.440 (4)	C20—C21	1.532 (3)
N1—C1	1.382 (3)	C21—C22	1.385 (3)
N1—C3	1.502 (3)	C21—C26	1.376 (4)
N2—C1	1.372 (3)	C22—C23	1.383 (4)
N2—C2	1.362 (3)	C23—C24	1.375 (4)
N2—H2N	0.91 (2)	C24—C25	1.374 (4)
N3—C20	1.493 (3)	C25—C26	1.384 (4)
N3—C18	1.390 (3)	C28—C34	1.372 (4)
N4—C18	1.363 (3)	C28—C29	1.385 (4)
N4—C19	1.362 (3)	C29—C30	1.368 (4)
N4—H4N	0.92 (2)	C30—C31	1.391 (4)
C2—C3	1.526 (4)	C31—C33	1.378 (4)
C3—C4	1.524 (3)	C33—C34	1.371 (4)
C3—C10	1.520 (4)	C22—H22	0.9500
C4—C9	1.374 (4)	C23—H23	0.9500
C4—C5	1.379 (4)	C24—H24	0.9500
C5—C6	1.379 (4)	C25—H25	0.9500
C6—C7	1.361 (4)	C26—H26	0.9500
C7—C8	1.357 (5)	C27—H27A	0.9800
C8—C9	1.381 (4)	C27—H27B	0.9800
C11—C17	1.380 (4)	C27—H27C	0.9800
C11—C12	1.373 (4)	C29—H29	0.9500
C12—C13	1.372 (4)	C30—H30	0.9500
C13—C14	1.378 (4)	C32—H32A	0.9800
C14—C16	1.387 (4)	C32—H32B	0.9800
C16—C17	1.374 (4)	C32—H32C	0.9800
C5—H5	0.9500	C33—H33	0.9500
C6—H6	0.9500	C34—H34	0.9500
O1—S1—O2	120.78 (12)	C13—C12—H12	120.00
O1—S1—N1	106.27 (10)	C14—C13—H13	120.00
O1—S1—C11	109.54 (12)	C12—C13—H13	120.00
O2—S1—N1	104.23 (11)	O5—C15—H15B	109.00
O2—S1—C11	109.21 (12)	O5—C15—H15A	109.00
N1—S1—C11	105.65 (11)	H15A—C15—H15B	109.00
O7—S2—N3	104.59 (11)	H15A—C15—H15C	110.00
O7—S2—C28	109.38 (12)	O5—C15—H15C	109.00
N3—S2—C28	104.90 (11)	H15B—C15—H15C	110.00

supplementary materials

O6—S2—C28	109.55 (12)	C14—C16—H16	120.00
O6—S2—O7	120.61 (12)	C17—C16—H16	120.00
O6—S2—N3	106.56 (11)	C16—C17—H17	120.00
C14—O5—C15	118.2 (2)	C11—C17—H17	120.00
C31—O10—C32	117.5 (2)	N3—C18—N4	107.7 (2)
S1—N1—C1	122.10 (17)	O8—C18—N3	126.9 (2)
S1—N1—C3	125.57 (16)	O8—C18—N4	125.4 (2)
C1—N1—C3	111.58 (19)	O9—C19—C20	125.8 (2)
C1—N2—C2	113.3 (2)	O9—C19—N4	125.9 (2)
C2—N2—H2N	129.4 (18)	N4—C19—C20	108.3 (2)
C1—N2—H2N	117.2 (18)	N3—C20—C21	112.64 (19)
S2—N3—C18	123.31 (18)	N3—C20—C19	100.5 (2)
S2—N3—C20	124.85 (16)	C19—C20—C27	107.1 (2)
C18—N3—C20	110.71 (19)	C21—C20—C27	114.8 (2)
C18—N4—C19	112.8 (2)	N3—C20—C27	111.6 (2)
C18—N4—H4N	119.1 (16)	C19—C20—C21	109.1 (2)
C19—N4—H4N	128.1 (16)	C22—C21—C26	119.3 (2)
N1—C1—N2	107.0 (2)	C20—C21—C22	118.8 (2)
O3—C1—N1	126.9 (2)	C20—C21—C26	122.0 (2)
O3—C1—N2	126.1 (2)	C21—C22—C23	120.2 (2)
O4—C2—C3	125.6 (2)	C22—C23—C24	120.1 (2)
O4—C2—N2	126.3 (2)	C23—C24—C25	120.0 (3)
N2—C2—C3	108.1 (2)	C24—C25—C26	120.0 (3)
N1—C3—C10	111.9 (2)	C21—C26—C25	120.5 (2)
N1—C3—C4	111.35 (19)	C29—C28—C34	119.9 (2)
N1—C3—C2	99.77 (19)	S2—C28—C29	120.4 (2)
C2—C3—C4	108.9 (2)	S2—C28—C34	119.7 (2)
C2—C3—C10	108.0 (2)	C28—C29—C30	119.8 (2)
C4—C3—C10	115.6 (2)	C29—C30—C31	119.8 (2)
C5—C4—C9	118.3 (2)	O10—C31—C33	124.5 (2)
C3—C4—C9	122.3 (2)	O10—C31—C30	115.0 (2)
C3—C4—C5	119.4 (2)	C30—C31—C33	120.5 (2)
C4—C5—C6	121.1 (3)	C31—C33—C34	118.9 (2)
C5—C6—C7	119.9 (3)	C28—C34—C33	121.1 (3)
C6—C7—C8	119.6 (3)	C21—C22—H22	120.00
C7—C8—C9	121.0 (3)	C23—C22—H22	120.00
C4—C9—C8	120.1 (3)	C22—C23—H23	120.00
S1—C11—C17	120.15 (19)	C24—C23—H23	120.00
S1—C11—C12	119.5 (2)	C23—C24—H24	120.00
C12—C11—C17	120.4 (2)	C25—C24—H24	120.00
C11—C12—C13	120.4 (2)	C24—C25—H25	120.00
C12—C13—C14	119.4 (3)	C26—C25—H25	120.00
O5—C14—C16	115.5 (3)	C21—C26—H26	120.00
C13—C14—C16	120.5 (2)	C25—C26—H26	120.00
O5—C14—C13	124.0 (3)	C20—C27—H27A	109.00
C14—C16—C17	119.6 (3)	C20—C27—H27B	110.00
C11—C17—C16	119.7 (2)	C20—C27—H27C	109.00
C4—C5—H5	119.00	H27A—C27—H27B	109.00
C6—C5—H5	119.00	H27A—C27—H27C	110.00

C5—C6—H6	120.00	H27B—C27—H27C	109.00
C7—C6—H6	120.00	C28—C29—H29	120.00
C6—C7—H7	120.00	C30—C29—H29	120.00
C8—C7—H7	120.00	C29—C30—H30	120.00
C7—C8—H8	120.00	C31—C30—H30	120.00
C9—C8—H8	119.00	O10—C32—H32A	109.00
C4—C9—H9	120.00	O10—C32—H32B	110.00
C8—C9—H9	120.00	O10—C32—H32C	109.00
H10A—C10—H10B	109.00	H32A—C32—H32B	109.00
H10A—C10—H10C	109.00	H32A—C32—H32C	109.00
C3—C10—H10C	109.00	H32B—C32—H32C	109.00
C3—C10—H10B	109.00	C31—C33—H33	121.00
C3—C10—H10A	110.00	C34—C33—H33	121.00
H10B—C10—H10C	110.00	C28—C34—H34	119.00
C11—C12—H12	120.00	C33—C34—H34	119.00
O1—S1—N1—C1	44.6 (2)	N2—C2—C3—N1	4.8 (2)
O1—S1—N1—C3	-146.18 (18)	O4—C2—C3—N1	-173.7 (2)
O2—S1—N1—C1	173.23 (18)	O4—C2—C3—C4	69.6 (3)
O2—S1—N1—C3	-17.6 (2)	O4—C2—C3—C10	-56.8 (3)
C11—S1—N1—C1	-71.7 (2)	C10—C3—C4—C9	-8.8 (4)
C11—S1—N1—C3	97.5 (2)	N1—C3—C4—C9	120.3 (3)
O1—S1—C11—C12	149.2 (2)	C2—C3—C4—C5	48.5 (3)
O1—S1—C11—C17	-31.9 (2)	N1—C3—C4—C5	-60.6 (3)
O2—S1—C11—C12	14.9 (2)	C10—C3—C4—C5	170.3 (2)
O2—S1—C11—C17	-166.3 (2)	C2—C3—C4—C9	-130.6 (3)
N1—S1—C11—C12	-96.7 (2)	C9—C4—C5—C6	-0.4 (4)
N1—S1—C11—C17	82.2 (2)	C3—C4—C5—C6	-179.5 (2)
O6—S2—N3—C18	-38.6 (2)	C3—C4—C9—C8	179.4 (3)
O6—S2—N3—C20	154.74 (18)	C5—C4—C9—C8	0.3 (4)
O7—S2—N3—C18	-167.37 (19)	C4—C5—C6—C7	1.0 (4)
O7—S2—N3—C20	26.0 (2)	C5—C6—C7—C8	-1.5 (5)
C28—S2—N3—C18	77.5 (2)	C6—C7—C8—C9	1.5 (5)
C28—S2—N3—C20	-89.1 (2)	C7—C8—C9—C4	-0.8 (5)
O6—S2—C28—C29	-137.8 (2)	S1—C11—C12—C13	179.4 (2)
O6—S2—C28—C34	44.6 (3)	C17—C11—C12—C13	0.5 (4)
O7—S2—C28—C29	-3.6 (3)	S1—C11—C17—C16	179.8 (2)
O7—S2—C28—C34	178.9 (2)	C12—C11—C17—C16	-1.4 (4)
N3—S2—C28—C29	108.1 (2)	C11—C12—C13—C14	0.8 (4)
N3—S2—C28—C34	-69.4 (2)	C12—C13—C14—O5	178.9 (3)
C15—O5—C14—C13	0.2 (4)	C12—C13—C14—C16	-1.2 (4)
C15—O5—C14—C16	-179.6 (3)	O5—C14—C16—C17	-179.7 (2)
C32—O10—C31—C30	-178.8 (2)	C13—C14—C16—C17	0.4 (4)
C32—O10—C31—C33	0.5 (4)	C14—C16—C17—C11	0.9 (4)
S1—N1—C3—C2	-175.22 (16)	O9—C19—C20—N3	-179.8 (2)
S1—N1—C1—O3	-5.0 (4)	O9—C19—C20—C21	-61.3 (3)
S1—N1—C1—N2	174.13 (15)	O9—C19—C20—C27	63.6 (3)
C3—N1—C1—O3	-175.6 (2)	N4—C19—C20—N3	0.9 (2)
C3—N1—C1—N2	3.6 (3)	N4—C19—C20—C21	119.4 (2)
C1—N1—C3—C2	-5.0 (2)	N4—C19—C20—C27	-115.8 (2)

supplementary materials

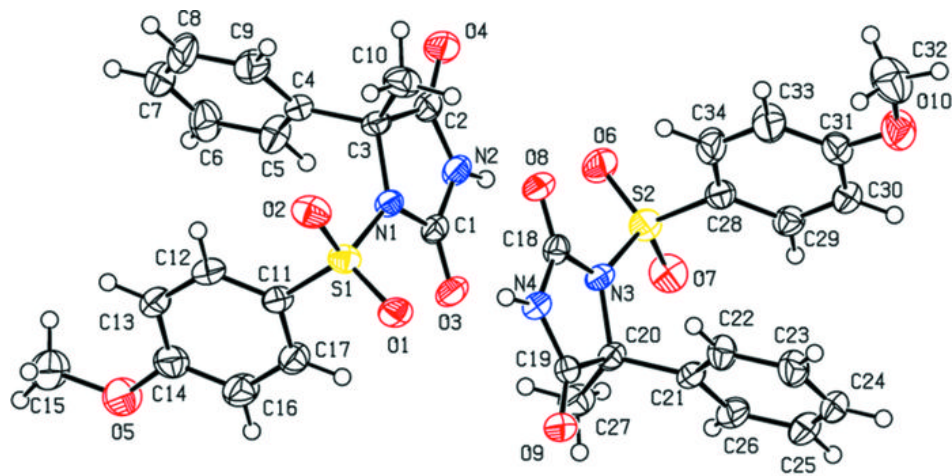
S1—N1—C3—C4	-60.3 (3)	N3—C20—C21—C22	67.3 (3)
S1—N1—C3—C10	70.7 (2)	N3—C20—C21—C26	-111.8 (3)
C1—N1—C3—C4	109.9 (2)	C19—C20—C21—C22	-43.4 (3)
C1—N1—C3—C10	-119.1 (2)	C19—C20—C21—C26	137.6 (2)
C1—N2—C2—O4	175.3 (3)	C27—C20—C21—C22	-163.5 (2)
C2—N2—C1—O3	179.0 (2)	C27—C20—C21—C26	17.4 (3)
C2—N2—C1—N1	-0.2 (3)	C20—C21—C22—C23	179.9 (2)
C1—N2—C2—C3	-3.2 (3)	C26—C21—C22—C23	-1.0 (4)
S2—N3—C18—O8	9.1 (4)	C20—C21—C26—C25	-180.0 (3)
S2—N3—C18—N4	-170.73 (16)	C22—C21—C26—C25	1.0 (4)
C20—N3—C18—N4	-2.4 (3)	C21—C22—C23—C24	0.1 (4)
S2—N3—C20—C19	169.04 (16)	C22—C23—C24—C25	0.8 (4)
S2—N3—C20—C21	53.1 (3)	C23—C24—C25—C26	-0.9 (4)
S2—N3—C20—C27	-77.7 (2)	C24—C25—C26—C21	-0.1 (4)
C18—N3—C20—C19	0.9 (2)	S2—C28—C29—C30	-178.7 (2)
C18—N3—C20—C21	-115.0 (2)	C34—C28—C29—C30	-1.2 (4)
C18—N3—C20—C27	114.2 (2)	S2—C28—C34—C33	177.6 (2)
C20—N3—C18—O8	177.4 (2)	C29—C28—C34—C33	0.1 (4)
C19—N4—C18—N3	3.1 (3)	C28—C29—C30—C31	1.3 (4)
C18—N4—C19—O9	178.2 (2)	C29—C30—C31—O10	179.0 (2)
C18—N4—C19—C20	-2.5 (3)	C29—C30—C31—C33	-0.3 (4)
C19—N4—C18—O8	-176.8 (2)	O10—C31—C33—C34	180.0 (2)
N2—C2—C3—C4	-112.0 (2)	C30—C31—C33—C34	-0.8 (4)
N2—C2—C3—C10	121.7 (2)	C31—C33—C34—C28	0.9 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2N \cdots O8 ⁱ	0.91 (2)	1.95 (2)	2.851 (3)	171 (2)
N4—H4N \cdots O3 ⁱⁱ	0.92 (2)	1.89 (2)	2.800 (3)	171 (2)
C5—H5 \cdots O2 ⁱ	0.95	2.36	3.218 (3)	150
C10—H10A \cdots O8	0.98	2.56	3.437 (3)	149
C10—H10B \cdots O2	0.98	2.45	3.055 (3)	120
C12—H12 \cdots O2	0.95	2.52	2.905 (3)	104
C13—H13 \cdots O9 ⁱⁱⁱ	0.95	2.43	3.287 (3)	150
C22—H22 \cdots O7 ⁱⁱ	0.95	2.42	3.351 (3)	165
C24—H24 \cdots O8 ^{iv}	0.95	2.56	3.492 (3)	166
C27—H27A \cdots O7	0.98	2.52	3.126 (4)	120
C29—H29 \cdots O7	0.95	2.54	2.916 (3)	104
C30—H30 \cdots O4 ^v	0.95	2.38	3.159 (3)	138
C34—H34 \cdots O8	0.95	2.56	3.291 (3)	134

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

Fig. 1



Molecule A

Molecule B

Fig. 2

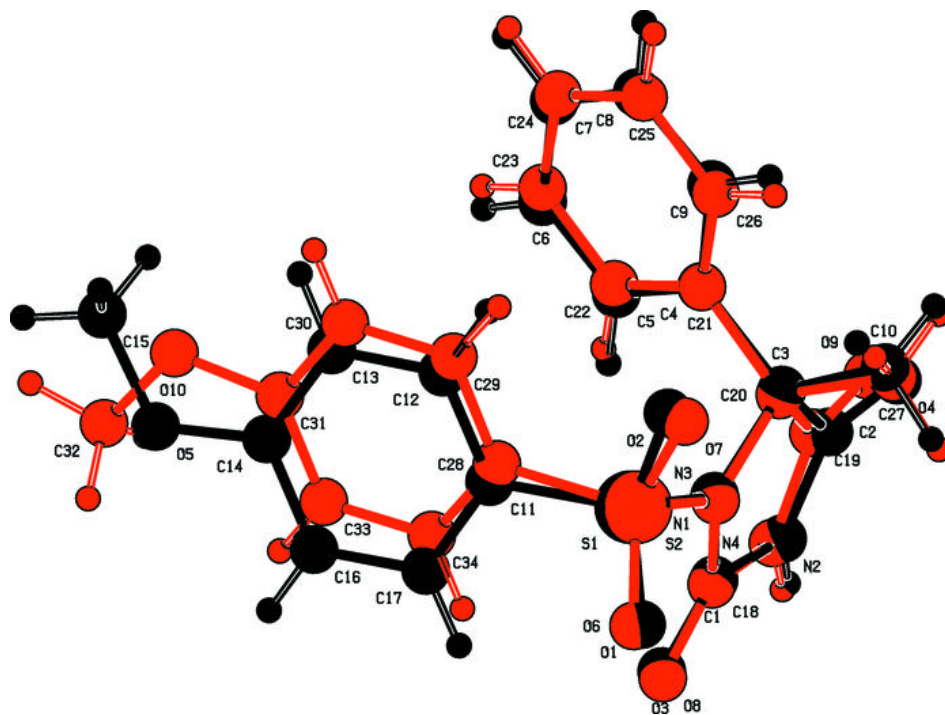


Fig. 3

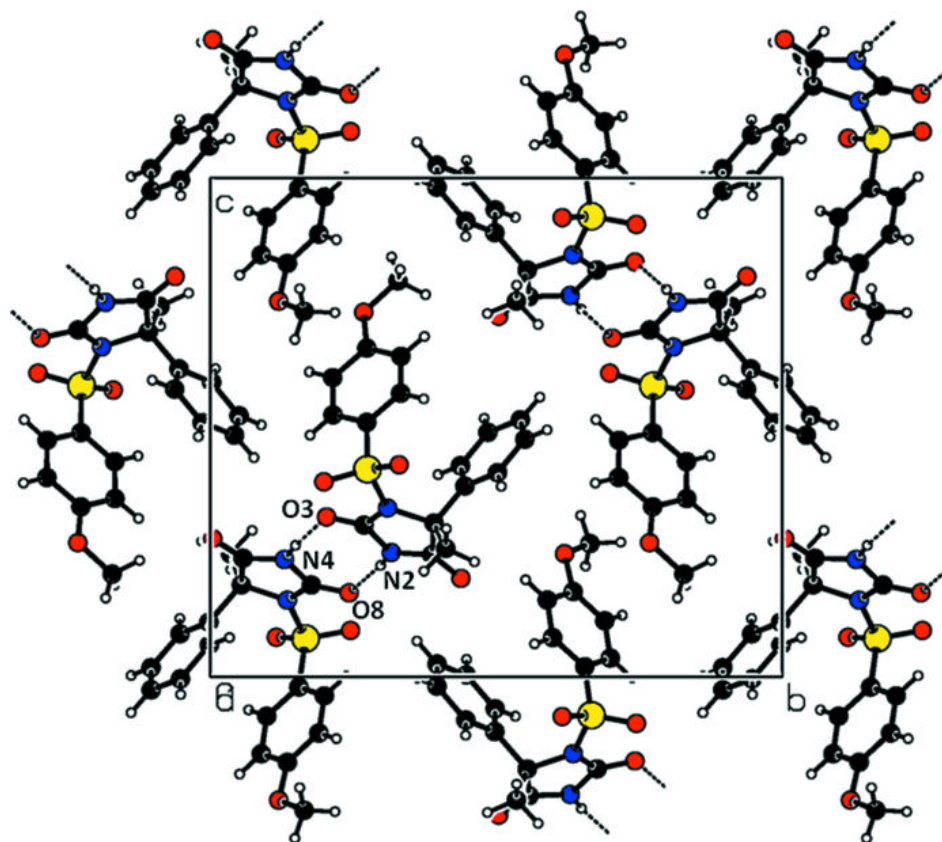


Fig. 4

