

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

N-[4-Acetyl-5-(3-methoxyphenyl)-4,5-dihydro-1,3,4-thiadiazol-2-yl]acetamide

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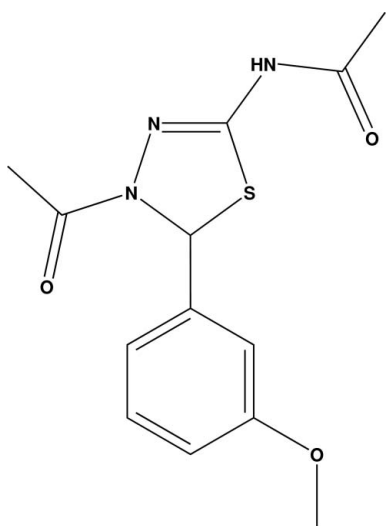
Received 29 September 2008; accepted 6 October 2008

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.042; wR factor = 0.109; data-to-parameter ratio = 20.7.

The title compound, $\text{C}_{13}\text{H}_{15}\text{N}_3\text{O}_3\text{S}$, crystallizes with two molecules in the asymmetric unit. The thiadiazole rings in both the molecules adopt an envelope conformation. The crystal packing is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For biological activities of thiadiazole derivatives, see: Balasubramanian *et al.* (2004); Li *et al.* (2001); Radwan *et al.* (2007); Supuran *et al.* (2001). For ring conformational analysis, see: Cremer & Pople (1975); Nardelli (1983).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{15}\text{N}_3\text{O}_3\text{S}$
 $M_r = 293.34$
Monoclinic, $P2_1$

$a = 11.3790$ (4) Å
 $b = 10.5993$ (3) Å
 $c = 11.9596$ (2) Å

$\beta = 108.225$ (2)°
 $V = 1370.08$ (7) Å³
 $Z = 4$
Mo $K\alpha$ radiation

$\mu = 0.25$ mm⁻¹
 $T = 293$ (2) K
 $0.30 \times 0.20 \times 0.16$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 1999)
 $T_{\min} = 0.930$, $T_{\max} = 0.962$

16660 measured reflections
7595 independent reflections
5635 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.109$
 $S = 1.03$
7595 reflections
367 parameters
1 restraint

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³
Absolute structure: Flack (1983), with 3584 Friedel pairs
Flack parameter: 0.07 (7)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N3}-\text{H3A}\cdots\text{O5}^i$ | 0.86 | 1.95 | 2.801 (3) | 171 |
| $\text{N6}-\text{H6A}\cdots\text{O2}^{ii}$ | 0.86 | 1.96 | 2.799 (3) | 164 |
| $\text{C25}-\text{H25C}\cdots\text{O2}^{ii}$ | 0.96 | 2.37 | 3.238 (4) | 150 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINTE* (Bruker, 2004); data reduction: *SAINTE*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

This research work was supported by the second stage of the BK21 Program, Republic of Korea. DV acknowledges financial support from the University Grants Commission (UGC-SAP) and the Department of Science and Technology (DST-FIST), Government of India, for providing facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2802).

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supplementary materials

Acta Cryst. (2008). E64, o2096 [doi:10.1107/S1600536808032108]

N-[4-Acetyl-5-(3-methoxyphenyl)-4,5-dihydro-1,3,4-thiadiazol-2-yl]acetamide

G. Aridoss, S. Amirthaganesan, D. Velmurugan, S. H. Kim and Y. T. Jeong

Comment

Nitrogen heterocycles are one of the most important classes of biologically active compounds. Suitably substituted 1,3,4-thiadiazoles have attracted great attention owing to their broad spectrum of biological activities in the areas of medicine which includes antimicrobial, antituberculosis, anesthetic, antithrombotic, anticonvulsant, antihypertensive, anti-inflammatory and antiulcer activities (Balasubramanian *et al.*, 2004; Li *et al.*, 2001; Radwan *et al.* 2007; Supuran *et al.*, 2001). Their action depends directly on the type and location of polar substituents on the heterocyclic ring. In general, pharmacological effect of potential drugs depends sensitively and solely on the stereochemistry and ring conformations. Thus, by keeping in view the promising biological potency of 1,3,4-thiadiazoles and variously substituted 1,3,4-thiadiazole frameworks, we have carried out the crystal structure determination of the title compound.

The title compound crystallizes with two molecules in the asymmetric unit. The sum of the angles at N1 (359.9 (6)°) and N4 (360.0 (6)°) are in accordance with sp^2 hybridization. The torsion angles around C6—C1—O1—C13 [0.0 (4)°] and C19—C14—O4—C26 [-1.8 (4)°] indicates the coplanarity of the methoxy groups with the corresponding phenyl rings (C1—C6) and (C14—C19), respectively. The thiadiazole ring in both the molecules adopt envelope conformation with atoms C7 and C20 deviating by 0.395 (3) and 0.350 (3) Å, respectively, from the mean plane of the remaining atoms. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for the thiadiazole rings C10—S2—C7—N1—N2 and C20—S2—C23—N5—N4 are $q_2 = 0.245$ (2), 0.217 (2) Å, $\varphi = 36.4$ (6), 215.4 (6)° and $\Delta_s(C_7) = 6.9$ (2), $\Delta_s(C_{20}) = 5.4$ (2), respectively. N—H···O and C—H···O intermolecular interactions stabilize the crystal packing (Table 1).

Experimental

The title compound was obtained by applying the method of Balasubramanian *et al.* (2004). 3-Methoxybenzaldehyde thiosemicarbazone obtained by the reaction of 3-Methoxybenzaldehyde and thiosemicarbazide was refluxed with excess of freshly distilled acetic anhydride on a water bath for about 7 h. After the completion of reaction, the excess of acetic anhydride was distilled off under reduced pressure and the obtained crude mass was purified by column chromatography (benzene–ethylacetate 5:1 v/v). Crystals were obtained from the solution of freshly distilled ethanol by slow evaporation at room temperature. ¹H NMR (DMSO-*d*₆, p.p.m.): 10.90 (s, 1H, amide NH); 7.36–6.76 (m, 5H, aromatic and ring methine protons); 3.78 (s, 3H, OCH₃); 2.38 (s, 3H, –COCH₃); 2.28 (s, 3H, –COCH₃).

Refinement

All H-atoms were refined using a riding model with $d(C-H) = 0.93$ Å, $U_{iso} = 1.2U_{eq}$ (C) for aromatic, 0.98 Å, $U_{iso} = 1.2U_{eq}$ (C) for CH, 0.96 Å, $U_{iso} = 1.5U_{eq}$ (C) for CH₃ and 0.86 Å, $U_{iso} = 1.2U_{eq}$ (N) for NH atoms. The methyl groups were allowed to rotate but not to tip.

Figures

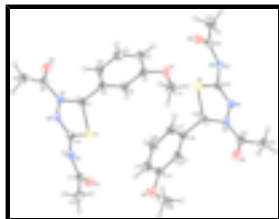


Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

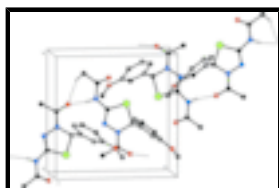


Fig. 2. The molecular packing of the title compound. For clarity, hydrogen atoms which are not involved in hydrogen bonding were omitted.

N-[4-Acetyl-5-(3-methoxyphenyl)-4,5-dihydro-1,3,4-thiadiazol-2-yl]acetamide

Crystal data

$C_{13}H_{15}N_3O_3S$

$M_r = 293.34$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 11.3790$ (4) Å

$b = 10.5993$ (3) Å

$c = 11.9596$ (2) Å

$\beta = 108.225$ (2)°

$V = 1370.08$ (7) Å³

$Z = 4$

$F_{000} = 616$

$D_x = 1.422$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5353 reflections

$\theta = 1.8$ – 29.6 °

$\mu = 0.25$ mm⁻¹

$T = 293$ (2) K

Prism, colourless

$0.30 \times 0.20 \times 0.16$ mm

Data collection

Bruker Kappa-APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

ω and φ scans

Absorption correction: multi-scan
(SADABS; Bruker, 1999)

$T_{\min} = 0.930$, $T_{\max} = 0.962$

16660 measured reflections

7595 independent reflections

5635 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 29.6$ °

$\theta_{\min} = 1.8$ °

$h = -15 \rightarrow 15$

$k = -14 \rightarrow 14$

$l = -14 \rightarrow 16$

Refinement

Refinement on F^2

Hydrogen site location: inferred from neighbouring sites

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.042$$

$$wR(F^2) = 0.109$$

$$S = 1.03$$

7595 reflections

367 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.2666P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Absolute structure: Flack (1983), with 3584 Friedel pairs

Flack parameter: 0.07 (7)

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|------------|------------|----------------------------------|
| C1 | 0.5869 (2) | 0.7772 (2) | 0.4618 (2) | 0.0397 (5) |
| C2 | 0.6405 (3) | 0.8511 (3) | 0.3955 (3) | 0.0498 (8) |
| H2 | 0.6027 | 0.8595 | 0.3148 | 0.060* |
| C3 | 0.7499 (3) | 0.9119 (3) | 0.4499 (2) | 0.0538 (7) |
| H3 | 0.7862 | 0.9615 | 0.4055 | 0.065* |
| C4 | 0.8069 (2) | 0.9005 (3) | 0.5697 (2) | 0.0442 (6) |
| H4 | 0.8808 | 0.9428 | 0.6055 | 0.053* |
| C5 | 0.7547 (2) | 0.8268 (2) | 0.6360 (2) | 0.0309 (5) |
| C6 | 0.64253 (18) | 0.7665 (2) | 0.5812 (2) | 0.0350 (5) |
| H6 | 0.6051 | 0.7186 | 0.6258 | 0.042* |
| C7 | 0.8123 (2) | 0.8112 (3) | 0.7677 (2) | 0.0324 (6) |
| H7 | 0.7546 | 0.8419 | 0.8076 | 0.039* |
| C8 | 0.9343 (2) | 1.0017 (3) | 0.8382 (2) | 0.0363 (6) |
| C9 | 1.0571 (2) | 1.0645 (3) | 0.8708 (3) | 0.0441 (7) |
| H9A | 1.0464 | 1.1541 | 0.8742 | 0.066* |
| H9B | 1.0969 | 1.0455 | 0.8129 | 0.066* |
| H9C | 1.1074 | 1.0346 | 0.9464 | 0.066* |
| C10 | 1.0096 (2) | 0.6892 (3) | 0.8300 (2) | 0.0315 (6) |
| C11 | 1.0879 (3) | 0.4753 (3) | 0.8707 (2) | 0.0390 (6) |
| C12 | 1.1970 (3) | 0.3942 (3) | 0.8773 (3) | 0.0569 (8) |

supplementary materials

| | | | | |
|------|--------------|-------------|--------------|--------------|
| H12A | 1.1920 | 0.3658 | 0.7997 | 0.085* |
| H12B | 1.1975 | 0.3226 | 0.9267 | 0.085* |
| H12C | 1.2716 | 0.4420 | 0.9098 | 0.085* |
| C13 | 0.4198 (2) | 0.6425 (3) | 0.4616 (3) | 0.0548 (7) |
| H13A | 0.3971 | 0.6925 | 0.5186 | 0.082* |
| H13B | 0.4748 | 0.5763 | 0.5011 | 0.082* |
| H13C | 0.3468 | 0.6062 | 0.4074 | 0.082* |
| C14 | 0.8900 (2) | 0.2827 (3) | 0.5333 (2) | 0.0431 (6) |
| C15 | 0.8399 (3) | 0.3437 (3) | 0.6089 (3) | 0.0499 (8) |
| H15 | 0.8734 | 0.3319 | 0.6897 | 0.060* |
| C16 | 0.7400 (3) | 0.4223 (3) | 0.5648 (2) | 0.0524 (7) |
| H16 | 0.7053 | 0.4623 | 0.6162 | 0.063* |
| C17 | 0.6906 (2) | 0.4425 (2) | 0.4454 (2) | 0.0420 (6) |
| H17 | 0.6236 | 0.4967 | 0.4164 | 0.050* |
| C18 | 0.7410 (2) | 0.3820 (3) | 0.3691 (2) | 0.0313 (5) |
| C19 | 0.84171 (19) | 0.3018 (2) | 0.4136 (2) | 0.0365 (5) |
| H19 | 0.8764 | 0.2611 | 0.3626 | 0.044* |
| C20 | 0.6864 (2) | 0.3966 (3) | 0.2384 (2) | 0.0316 (5) |
| H20 | 0.7459 | 0.3662 | 0.2005 | 0.038* |
| C21 | 0.5698 (2) | 0.2018 (3) | 0.1704 (2) | 0.0352 (6) |
| C22 | 0.4489 (2) | 0.1337 (3) | 0.1313 (3) | 0.0457 (7) |
| H22A | 0.4280 | 0.1056 | 0.1990 | 0.069* |
| H22B | 0.4554 | 0.0621 | 0.0844 | 0.069* |
| H22C | 0.3857 | 0.1895 | 0.0855 | 0.069* |
| C23 | 0.4851 (2) | 0.5119 (3) | 0.1630 (2) | 0.0307 (5) |
| C24 | 0.4060 (2) | 0.7251 (3) | 0.1174 (2) | 0.0371 (6) |
| C25 | 0.2952 (2) | 0.8055 (3) | 0.1067 (3) | 0.0520 (8) |
| H25A | 0.2616 | 0.8347 | 0.0271 | 0.078* |
| H25B | 0.3190 | 0.8766 | 0.1587 | 0.078* |
| H25C | 0.2340 | 0.7569 | 0.1275 | 0.078* |
| C26 | 1.0400 (3) | 0.1360 (3) | 0.5113 (3) | 0.0692 (9) |
| H26A | 1.0753 | 0.1935 | 0.4686 | 0.104* |
| H26B | 0.9772 | 0.0863 | 0.4568 | 0.104* |
| H26C | 1.1035 | 0.0814 | 0.5586 | 0.104* |
| N1 | 0.92957 (17) | 0.8777 (2) | 0.81336 (18) | 0.0319 (5) |
| N2 | 1.03611 (17) | 0.8057 (2) | 0.8278 (2) | 0.0339 (5) |
| N3 | 1.10017 (18) | 0.5994 (2) | 0.8432 (2) | 0.0383 (5) |
| H3A | 1.1689 | 0.6224 | 0.8336 | 0.046* |
| N4 | 0.57049 (17) | 0.3249 (2) | 0.1927 (2) | 0.0332 (5) |
| N5 | 0.46128 (17) | 0.3956 (2) | 0.16790 (19) | 0.0330 (5) |
| N6 | 0.39184 (18) | 0.6002 (2) | 0.1401 (2) | 0.0357 (5) |
| H6A | 0.3199 | 0.5754 | 0.1399 | 0.043* |
| O1 | 0.47967 (16) | 0.7197 (2) | 0.39944 (16) | 0.0550 (5) |
| O2 | 0.83930 (16) | 1.0588 (2) | 0.8297 (2) | 0.0525 (5) |
| O3 | 0.99404 (17) | 0.4378 (2) | 0.88764 (19) | 0.0514 (5) |
| O4 | 0.98715 (18) | 0.2046 (2) | 0.58456 (19) | 0.0676 (6) |
| O5 | 0.66898 (16) | 0.1464 (2) | 0.18601 (19) | 0.0513 (5) |
| O6 | 0.50089 (16) | 0.7653 (2) | 0.10697 (18) | 0.0487 (5) |
| S1 | 0.85560 (5) | 0.64828 (6) | 0.81229 (6) | 0.03785 (17) |

S2 0.63984 (5) 0.55696 (6) 0.18841 (6) 0.03857 (17)

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0346 (10) | 0.0384 (13) | 0.0415 (13) | 0.0002 (9) | 0.0053 (10) | -0.0048 (10) |
| C2 | 0.0547 (16) | 0.054 (2) | 0.0347 (15) | -0.0045 (14) | 0.0053 (13) | 0.0004 (13) |
| C3 | 0.0597 (15) | 0.0617 (19) | 0.0407 (15) | -0.0191 (14) | 0.0166 (12) | 0.0057 (13) |
| C4 | 0.0404 (12) | 0.0500 (15) | 0.0426 (14) | -0.0140 (11) | 0.0136 (11) | -0.0016 (12) |
| C5 | 0.0285 (10) | 0.0297 (14) | 0.0349 (13) | 0.0004 (9) | 0.0102 (10) | -0.0034 (11) |
| C6 | 0.0299 (9) | 0.0337 (12) | 0.0405 (12) | -0.0024 (8) | 0.0099 (9) | -0.0001 (10) |
| C7 | 0.0236 (10) | 0.0341 (14) | 0.0378 (14) | -0.0022 (10) | 0.0071 (10) | -0.0053 (12) |
| C8 | 0.0347 (12) | 0.0371 (16) | 0.0382 (15) | 0.0007 (11) | 0.0129 (11) | -0.0028 (12) |
| C9 | 0.0377 (12) | 0.0392 (16) | 0.0552 (18) | -0.0079 (13) | 0.0142 (12) | -0.0115 (15) |
| C10 | 0.0253 (10) | 0.0370 (16) | 0.0324 (13) | -0.0024 (10) | 0.0093 (9) | -0.0015 (11) |
| C11 | 0.0412 (13) | 0.0371 (16) | 0.0370 (15) | -0.0039 (12) | 0.0096 (11) | -0.0004 (12) |
| C12 | 0.0521 (16) | 0.0326 (16) | 0.086 (2) | 0.0014 (14) | 0.0209 (16) | 0.0069 (17) |
| C13 | 0.0384 (12) | 0.0568 (17) | 0.0667 (18) | -0.0103 (12) | 0.0129 (12) | -0.0108 (15) |
| C14 | 0.0331 (10) | 0.0456 (15) | 0.0455 (14) | -0.0001 (10) | 0.0048 (10) | 0.0045 (11) |
| C15 | 0.0554 (16) | 0.055 (2) | 0.0351 (16) | -0.0062 (14) | 0.0080 (13) | -0.0022 (13) |
| C16 | 0.0600 (15) | 0.0552 (18) | 0.0441 (15) | 0.0019 (13) | 0.0192 (13) | -0.0117 (13) |
| C17 | 0.0406 (11) | 0.0386 (13) | 0.0458 (14) | 0.0050 (10) | 0.0119 (11) | -0.0053 (11) |
| C18 | 0.0251 (10) | 0.0291 (14) | 0.0374 (14) | -0.0036 (9) | 0.0066 (10) | -0.0007 (11) |
| C19 | 0.0301 (9) | 0.0380 (12) | 0.0417 (13) | 0.0036 (9) | 0.0118 (9) | 0.0018 (11) |
| C20 | 0.0237 (10) | 0.0339 (14) | 0.0383 (14) | -0.0015 (10) | 0.0112 (10) | -0.0012 (12) |
| C21 | 0.0325 (12) | 0.0351 (15) | 0.0398 (15) | -0.0032 (11) | 0.0141 (10) | -0.0071 (12) |
| C22 | 0.0401 (13) | 0.0432 (18) | 0.0551 (18) | -0.0099 (13) | 0.0167 (12) | -0.0118 (15) |
| C23 | 0.0268 (10) | 0.0343 (15) | 0.0289 (13) | -0.0005 (10) | 0.0056 (9) | 0.0035 (10) |
| C24 | 0.0371 (13) | 0.0330 (15) | 0.0358 (14) | -0.0013 (11) | 0.0036 (11) | 0.0008 (12) |
| C25 | 0.0428 (14) | 0.0347 (16) | 0.074 (2) | 0.0065 (12) | 0.0111 (14) | 0.0046 (16) |
| C26 | 0.0557 (16) | 0.066 (2) | 0.088 (2) | 0.0258 (16) | 0.0253 (16) | 0.0328 (19) |
| N1 | 0.0219 (9) | 0.0343 (13) | 0.0381 (12) | -0.0010 (8) | 0.0074 (8) | -0.0054 (10) |
| N2 | 0.0238 (9) | 0.0348 (13) | 0.0425 (13) | 0.0014 (9) | 0.0096 (9) | -0.0019 (10) |
| N3 | 0.0308 (10) | 0.0327 (13) | 0.0541 (14) | -0.0009 (8) | 0.0171 (9) | 0.0045 (10) |
| N4 | 0.0234 (9) | 0.0334 (13) | 0.0407 (13) | -0.0005 (9) | 0.0069 (8) | -0.0048 (10) |
| N5 | 0.0257 (9) | 0.0332 (13) | 0.0388 (12) | 0.0007 (9) | 0.0083 (8) | -0.0038 (10) |
| N6 | 0.0272 (9) | 0.0322 (13) | 0.0461 (12) | -0.0002 (8) | 0.0089 (8) | 0.0034 (10) |
| O1 | 0.0433 (9) | 0.0665 (13) | 0.0459 (10) | -0.0145 (9) | 0.0006 (8) | -0.0070 (10) |
| O2 | 0.0383 (9) | 0.0388 (12) | 0.0845 (15) | 0.0039 (10) | 0.0252 (10) | -0.0079 (12) |
| O3 | 0.0465 (11) | 0.0472 (14) | 0.0617 (13) | -0.0102 (10) | 0.0187 (10) | 0.0077 (11) |
| O4 | 0.0572 (11) | 0.0840 (16) | 0.0567 (12) | 0.0303 (11) | 0.0110 (10) | 0.0231 (12) |
| O5 | 0.0363 (9) | 0.0405 (12) | 0.0795 (14) | 0.0048 (10) | 0.0216 (9) | -0.0079 (12) |
| O6 | 0.0431 (10) | 0.0430 (13) | 0.0617 (13) | -0.0049 (9) | 0.0187 (9) | 0.0097 (11) |
| S1 | 0.0281 (3) | 0.0403 (4) | 0.0434 (4) | -0.0051 (3) | 0.0087 (2) | 0.0057 (3) |
| S2 | 0.0264 (3) | 0.0371 (4) | 0.0505 (4) | -0.0023 (3) | 0.0096 (3) | 0.0090 (3) |

Geometric parameters (Å, °)

C1—O1 1.359 (3) C14—C19 1.377 (3)

supplementary materials

| | | | |
|----------|-----------|-------------|-------------|
| C1—C6 | 1.374 (3) | C15—C16 | 1.374 (4) |
| C1—C2 | 1.384 (4) | C15—H15 | 0.9300 |
| C2—C3 | 1.372 (4) | C16—C17 | 1.378 (4) |
| C2—H2 | 0.9300 | C16—H16 | 0.9300 |
| C3—C4 | 1.380 (4) | C17—C18 | 1.378 (3) |
| C3—H3 | 0.9300 | C17—H17 | 0.9300 |
| C4—C5 | 1.374 (3) | C18—C19 | 1.392 (3) |
| C4—H4 | 0.9300 | C18—C20 | 1.499 (3) |
| C5—C6 | 1.394 (3) | C19—H19 | 0.9300 |
| C5—C7 | 1.514 (4) | C20—N4 | 1.471 (3) |
| C6—H6 | 0.9300 | C20—S2 | 1.824 (3) |
| C7—N1 | 1.456 (3) | C20—H20 | 0.9800 |
| C7—S1 | 1.829 (3) | C21—O5 | 1.234 (3) |
| C7—H7 | 0.9800 | C21—N4 | 1.332 (4) |
| C8—O2 | 1.215 (3) | C21—C22 | 1.493 (3) |
| C8—N1 | 1.345 (4) | C22—H22A | 0.9600 |
| C8—C9 | 1.486 (4) | C22—H22B | 0.9600 |
| C9—H9A | 0.9600 | C22—H22C | 0.9600 |
| C9—H9B | 0.9600 | C23—N5 | 1.267 (4) |
| C9—H9C | 0.9600 | C23—N6 | 1.377 (3) |
| C10—N2 | 1.274 (3) | C23—S2 | 1.757 (2) |
| C10—N3 | 1.375 (3) | C24—N6 | 1.370 (4) |
| C10—S1 | 1.753 (2) | C24—C25 | 1.493 (4) |
| C11—O3 | 1.215 (3) | C25—H25A | 0.9600 |
| C11—N3 | 1.373 (4) | C25—H25B | 0.9600 |
| C11—C12 | 1.492 (4) | C25—H25C | 0.9600 |
| C12—H12A | 0.9600 | C26—O4 | 1.410 (4) |
| C12—H12B | 0.9600 | C26—H26A | 0.9600 |
| C12—H12C | 0.9600 | C26—H26B | 0.9600 |
| C13—O1 | 1.416 (3) | C26—H26C | 0.9600 |
| C13—H13A | 0.9600 | N1—N2 | 1.397 (3) |
| C13—H13B | 0.9600 | N3—H3A | 0.8600 |
| C13—H13C | 0.9600 | N4—N5 | 1.401 (3) |
| C14—O4 | 1.364 (3) | N6—H6A | 0.8600 |
| C14—C15 | 1.373 (4) | | |
| O1—C1—C6 | 125.1 (2) | C16—C17—C18 | 119.6 (2) |
| O1—C1—C2 | 114.9 (2) | C16—C17—H17 | 120.2 |
| C6—C1—C2 | 120.0 (2) | C18—C17—H17 | 120.2 |
| C3—C2—C1 | 119.4 (3) | C17—C18—C19 | 119.6 (2) |
| C3—C2—H2 | 120.3 | C17—C18—C20 | 121.3 (2) |
| C1—C2—H2 | 120.3 | C19—C18—C20 | 119.0 (2) |
| C2—C3—C4 | 120.9 (3) | C14—C19—C18 | 120.0 (2) |
| C2—C3—H3 | 119.6 | C14—C19—H19 | 120.0 |
| C4—C3—H3 | 119.6 | C18—C19—H19 | 120.0 |
| C5—C4—C3 | 120.1 (2) | N4—C20—C18 | 111.4 (2) |
| C5—C4—H4 | 120.0 | N4—C20—S2 | 103.01 (16) |
| C3—C4—H4 | 120.0 | C18—C20—S2 | 114.9 (2) |
| C4—C5—C6 | 119.2 (2) | N4—C20—H20 | 109.1 |
| C4—C5—C7 | 122.5 (2) | C18—C20—H20 | 109.1 |

| | | | |
|---------------|-------------|---------------|-------------|
| C6—C5—C7 | 118.3 (2) | S2—C20—H20 | 109.1 |
| C1—C6—C5 | 120.5 (2) | O5—C21—N4 | 119.2 (2) |
| C1—C6—H6 | 119.8 | O5—C21—C22 | 121.8 (3) |
| C5—C6—H6 | 119.8 | N4—C21—C22 | 118.9 (2) |
| N1—C7—C5 | 112.6 (2) | C21—C22—H22A | 109.5 |
| N1—C7—S1 | 102.46 (16) | C21—C22—H22B | 109.5 |
| C5—C7—S1 | 113.33 (19) | H22A—C22—H22B | 109.5 |
| N1—C7—H7 | 109.4 | C21—C22—H22C | 109.5 |
| C5—C7—H7 | 109.4 | H22A—C22—H22C | 109.5 |
| S1—C7—H7 | 109.4 | H22B—C22—H22C | 109.5 |
| O2—C8—N1 | 119.7 (3) | N5—C23—N6 | 120.6 (2) |
| O2—C8—C9 | 122.5 (3) | N5—C23—S2 | 118.25 (19) |
| N1—C8—C9 | 117.7 (2) | N6—C23—S2 | 121.2 (2) |
| C8—C9—H9A | 109.5 | O6—C24—N6 | 121.8 (3) |
| C8—C9—H9B | 109.5 | O6—C24—C25 | 123.3 (3) |
| H9A—C9—H9B | 109.5 | N6—C24—C25 | 114.9 (2) |
| C8—C9—H9C | 109.5 | C24—C25—H25A | 109.5 |
| H9A—C9—H9C | 109.5 | C24—C25—H25B | 109.5 |
| H9B—C9—H9C | 109.5 | H25A—C25—H25B | 109.5 |
| N2—C10—N3 | 120.0 (2) | C24—C25—H25C | 109.5 |
| N2—C10—S1 | 118.12 (19) | H25A—C25—H25C | 109.5 |
| N3—C10—S1 | 121.8 (2) | H25B—C25—H25C | 109.5 |
| O3—C11—N3 | 120.9 (3) | O4—C26—H26A | 109.5 |
| O3—C11—C12 | 124.3 (3) | O4—C26—H26B | 109.5 |
| N3—C11—C12 | 114.8 (3) | H26A—C26—H26B | 109.5 |
| C11—C12—H12A | 109.5 | O4—C26—H26C | 109.5 |
| C11—C12—H12B | 109.5 | H26A—C26—H26C | 109.5 |
| H12A—C12—H12B | 109.5 | H26B—C26—H26C | 109.5 |
| C11—C12—H12C | 109.5 | C8—N1—N2 | 122.2 (2) |
| H12A—C12—H12C | 109.5 | C8—N1—C7 | 121.6 (2) |
| H12B—C12—H12C | 109.5 | N2—N1—C7 | 116.1 (2) |
| O1—C13—H13A | 109.5 | C10—N2—N1 | 109.3 (2) |
| O1—C13—H13B | 109.5 | C11—N3—C10 | 124.2 (2) |
| H13A—C13—H13B | 109.5 | C11—N3—H3A | 117.9 |
| O1—C13—H13C | 109.5 | C10—N3—H3A | 117.9 |
| H13A—C13—H13C | 109.5 | C21—N4—N5 | 122.2 (2) |
| H13B—C13—H13C | 109.5 | C21—N4—C20 | 122.0 (2) |
| O4—C14—C15 | 115.8 (2) | N5—N4—C20 | 115.8 (2) |
| O4—C14—C19 | 124.1 (2) | C23—N5—N4 | 110.0 (2) |
| C15—C14—C19 | 120.2 (2) | C24—N6—C23 | 124.4 (2) |
| C14—C15—C16 | 119.7 (3) | C24—N6—H6A | 117.8 |
| C14—C15—H15 | 120.1 | C23—N6—H6A | 117.8 |
| C16—C15—H15 | 120.1 | C1—O1—C13 | 117.9 (2) |
| C15—C16—C17 | 120.9 (3) | C14—O4—C26 | 118.5 (2) |
| C15—C16—H16 | 119.6 | C10—S1—C7 | 88.3 (1) |
| C17—C16—H16 | 119.6 | C23—S2—C20 | 88.6 (1) |
| O1—C1—C2—C3 | -179.1 (3) | N3—C10—N2—N1 | 179.8 (2) |
| C6—C1—C2—C3 | 0.8 (4) | S1—C10—N2—N1 | 1.3 (3) |
| C1—C2—C3—C4 | -0.1 (5) | C8—N1—N2—C10 | 162.3 (3) |

supplementary materials

| | | | |
|-----------------|------------|----------------|--------------|
| C2—C3—C4—C5 | 0.5 (5) | C7—N1—N2—C10 | -18.8 (3) |
| C3—C4—C5—C6 | -1.4 (4) | O3—C11—N3—C10 | -1.5 (4) |
| C3—C4—C5—C7 | -179.6 (3) | C12—C11—N3—C10 | 178.4 (3) |
| O1—C1—C6—C5 | 178.2 (2) | N2—C10—N3—C11 | 166.0 (3) |
| C2—C1—C6—C5 | -1.8 (4) | S1—C10—N3—C11 | -15.5 (4) |
| C4—C5—C6—C1 | 2.1 (4) | O5—C21—N4—N5 | 174.7 (2) |
| C7—C5—C6—C1 | -179.6 (2) | C22—C21—N4—N5 | -7.5 (4) |
| C4—C5—C7—N1 | -2.5 (4) | O5—C21—N4—C20 | -1.6 (4) |
| C6—C5—C7—N1 | 179.3 (2) | C22—C21—N4—C20 | 176.2 (2) |
| C4—C5—C7—S1 | -118.2 (2) | C18—C20—N4—C21 | -81.9 (3) |
| C6—C5—C7—S1 | 63.5 (3) | S2—C20—N4—C21 | 154.4 (2) |
| O4—C14—C15—C16 | -178.6 (3) | C18—C20—N4—N5 | 101.6 (3) |
| C19—C14—C15—C16 | 1.1 (4) | S2—C20—N4—N5 | -22.2 (3) |
| C14—C15—C16—C17 | -1.1 (5) | N6—C23—N5—N4 | -179.8 (2) |
| C15—C16—C17—C18 | 0.8 (4) | S2—C23—N5—N4 | -0.8 (3) |
| C16—C17—C18—C19 | -0.4 (4) | C21—N4—N5—C23 | -160.5 (3) |
| C16—C17—C18—C20 | 177.1 (3) | C20—N4—N5—C23 | 16.0 (3) |
| O4—C14—C19—C18 | 179.0 (2) | O6—C24—N6—C23 | 5.9 (4) |
| C15—C14—C19—C18 | -0.7 (4) | C25—C24—N6—C23 | -174.3 (2) |
| C17—C18—C19—C14 | 0.4 (4) | N5—C23—N6—C24 | -170.8 (3) |
| C20—C18—C19—C14 | -177.2 (2) | S2—C23—N6—C24 | 10.3 (4) |
| C17—C18—C20—N4 | -74.4 (3) | C6—C1—O1—C13 | 0.0 (4) |
| C19—C18—C20—N4 | 103.1 (2) | C2—C1—O1—C13 | 180.0 (2) |
| C17—C18—C20—S2 | 42.2 (3) | C15—C14—O4—C26 | 177.9 (3) |
| C19—C18—C20—S2 | -140.2 (2) | C19—C14—O4—C26 | -1.8 (4) |
| O2—C8—N1—N2 | -176.4 (2) | N2—C10—S1—C7 | 11.6 (2) |
| C9—C8—N1—N2 | 6.0 (4) | N3—C10—S1—C7 | -166.9 (2) |
| O2—C8—N1—C7 | 4.7 (4) | N1—C7—S1—C10 | -18.96 (18) |
| C9—C8—N1—C7 | -172.9 (2) | C5—C7—S1—C10 | 102.70 (18) |
| C5—C7—N1—C8 | 82.3 (3) | N5—C23—S2—C20 | -10.6 (2) |
| S1—C7—N1—C8 | -155.5 (2) | N6—C23—S2—C20 | 168.4 (2) |
| C5—C7—N1—N2 | -96.6 (3) | N4—C20—S2—C23 | 16.77 (18) |
| S1—C7—N1—N2 | 25.5 (3) | C18—C20—S2—C23 | -104.62 (18) |

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| N3—H3A \cdots O5 ⁱ | 0.86 | 1.95 | 2.801 (3) | 171 |
| N6—H6A \cdots O2 ⁱⁱ | 0.86 | 1.96 | 2.799 (3) | 164 |
| C25—H25C \cdots O2 ⁱⁱ | 0.96 | 2.37 | 3.238 (4) | 150 |

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

Fig. 2

