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N-[(3-Methyl-5-phenoxy-1-phenylpyrazol-4-yl)carbonyl]-N'-(5-propyl-1,3,4-thiadiazol-2-yl)thiourea

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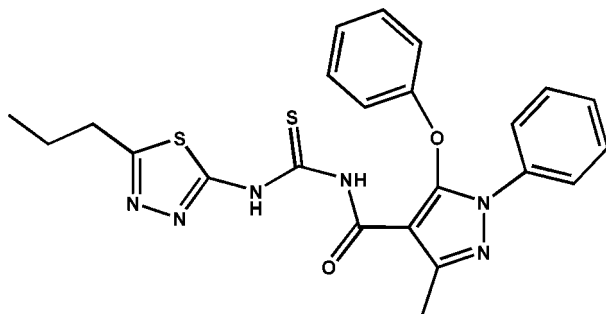
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Key indicators: single-crystal X-ray study; $T = 153$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; disorder in main residue; R factor = 0.043; wR factor = 0.165; data-to-parameter ratio = 16.3.

In the crystal structure of the title compound, $\text{C}_{23}\text{H}_{22}\text{N}_6\text{O}_2\text{S}_2$, there are two intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. The propyl chain is disordered over two sites, with occupancy factors of 0.639 (5) and 0.361 (5).

Related literature

For pharmacological and biological properties, see: Ranise *et al.* (2003); Akbas *et al.* (2005); Daidone *et al.* (2004); Park *et al.* (2005); Thomasco *et al.* (2003); Foroumadi *et al.* (2002); Supuran & Scozzafava (2000).



Experimental

Crystal data

$\text{C}_{23}\text{H}_{22}\text{N}_6\text{O}_2\text{S}_2$
 $M_r = 478.58$
 Triclinic, $P\bar{1}$
 $a = 8.4489$ (3) Å
 $b = 9.9099$ (4) Å

$c = 14.3492$ (5) Å
 $\alpha = 86.089$ (1)°
 $\beta = 74.048$ (1)°
 $\gamma = 81.591$ (1)°
 $V = 1142.25$ (7) Å³

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.27$ mm⁻¹

$T = 153$ (2) K
 $0.32 \times 0.15 \times 0.13$ mm

Data collection

Rigaku R-Axis SPIDER
 diffractometer
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.919$, $T_{\max} = 0.966$

11326 measured reflections
 5171 independent reflections
 3376 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.165$
 $S = 1.05$
 5171 reflections

317 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.36$ e Å⁻³
 $\Delta\rho_{\min} = -0.54$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N3}-\text{H3A}\cdots\text{O1}$	0.88	2.18	2.917 (2)	141
$\text{N4}-\text{H4A}\cdots\text{O2}$	0.88	1.89	2.623 (2)	139

Data collection: *RAPID-AUTO* (Rigaku 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2066).

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supplementary materials

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N-[(3-Methyl-5-phenoxy-1-phenylpyrazol-4-yl)carbonyl]-*N'*-(5-propyl-1,3,4-thiadiazol-2-yl)thiourea

Y.-R. Sun, G. Liu, C.-J. Liu and Y.-P. Li

Comment

In previous papers, aroylthioureas were reported to be endowed with various and interesting pharmacological properties (Ranize *et al.*, 2003). Compounds including pyrazole ring are known to possess several biological properties, such as antiseptis, antileukosis, antitumor (Akbas *et al.*, 2005; Daidone *et al.*, 2004; Park *et al.*, 2005). The 1,3,4-thiadiazoles also have widespread biological activity, such as antibacterial, antitubercular, antineoplastic activities (Thomasco *et al.*, 2003; Foroumadi *et al.*, 2002; Supuran & Scozzafava, 2000). Due to identical molecular including many heterocyclic nucleus can attain to effective superimposition of biological activity, we designed and synthesized *N*-(2-propyl-1,3,4-thiodiazol-5-yl)-*N'*-(1-phenyl-3-methyl-5- phenoxy)pyrazol-4-yl)-carbonylthiourea.

The molecule of the title complex (Fig. 1) has two intramolecular hydrogen bonds, which were formed between N3—H3A and O1 and between N4—H4A and O2, which lead to the formation of two six-membered closed loop. Creation of these (pseudo) rings is crucial for the molecular conformations, because it prevents free rotation within the central carbonylthiourea moiety and locks its atoms in a nearly planar arrangement.

Experimental

A mixture of 1-phenyl-3-methyl-5-phenoxy)pyrazole-4-isothiocyanate (1 mmol) and 5-propyl-2-amino-1,3,4-thiodiazole (1 mmol) in absolute acetonitrile was refluxed for 10 h at about 354–364 K, then the product was decanted from the hot solution in a funnel, and dried at room temperature for a yield 25.1% (0.12 g), m.p. 461–463 K. Block-like single-crystal of compound (I) was grown from solution of ethanol by slow evaporation.

Refinement

All H atoms were found in difference electron maps and were subsequently refined in the riding-model approximation with C—H = 0.95–0.99 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$; N—H = 0.88 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

It is disordered in the propyl moiety of molecule. The propyl chain was split into two chains and the restraints applied to the two chains and the two chains can share the same C21 atom. The ratio of occupancy factors is 0.639 (5)/0.361.

Figures

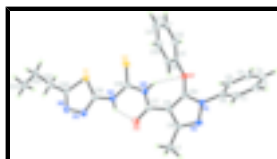


Fig. 1. The molecule structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The H atoms are shown as spheres of arbitrary radius. The intramolecular H bond are marked as dashed lines. Only major fragment of propyl chain are drawn.

supplementary materials

N-[(3-Methyl-5-phenoxy-1-phenylpyrazol-4-yl)carbonyl]-*N'*-(5-propyl-1,3,4-thiadiazol-2-yl)thiourea

Crystal data

$C_{23}H_{22}N_6O_2S_2$	$Z = 2$
$M_r = 478.58$	$F_{000} = 500$
Triclinic, $P\bar{1}$	$D_x = 1.391 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Melting point: 461-463 K
$a = 8.4489 (3) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.9099 (4) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$c = 14.3492 (5) \text{ \AA}$	Cell parameters from 7971 reflections
$\alpha = 86.089 (1)^\circ$	$\theta = 3.1\text{--}27.5^\circ$
$\beta = 74.048 (1)^\circ$	$\mu = 0.27 \text{ mm}^{-1}$
$\gamma = 81.591 (1)^\circ$	$T = 153 (2) \text{ K}$
$V = 1142.25 (7) \text{ \AA}^3$	Block, yellow
	$0.32 \times 0.15 \times 0.13 \text{ mm}$

Data collection

Rigaku R-Axis Spider diffractometer	5171 independent reflections
Radiation source: fine-focus sealed tube	3376 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.024$
$T = 153(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
ω scans	$\theta_{\text{min}} = 3.1^\circ$
Absorption correction: empirical (using intensity measurements) (ABSCOR; Higashi, 1995)	$h = -10 \rightarrow 10$
$T_{\text{min}} = 0.919$, $T_{\text{max}} = 0.966$	$k = -12 \rightarrow 12$
11326 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.165$	$w = 1/[\sigma^2(F_o^2) + (0.1061P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
5171 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
317 parameters	$\Delta\rho_{\text{max}} = 0.36 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.54 \text{ e \AA}^{-3}$
	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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
S1	0.56160 (7)	0.15893 (6)	0.37164 (4)	0.04016 (19)	
S2	0.56697 (9)	0.18955 (7)	0.58112 (5)	0.0511 (2)	
O1	0.72651 (18)	0.43251 (15)	0.78717 (10)	0.0330 (3)	
O2	0.7995 (2)	0.57769 (18)	0.48360 (11)	0.0438 (4)	
N1	0.8356 (2)	0.63699 (19)	0.78887 (13)	0.0328 (4)	
N2	0.8911 (2)	0.74382 (19)	0.72761 (13)	0.0360 (4)	
N3	0.6887 (2)	0.4151 (2)	0.59251 (13)	0.0346 (4)	
H3A	0.6652	0.3916	0.6546	0.042*	
N4	0.6649 (2)	0.3756 (2)	0.44130 (13)	0.0357 (4)	
H4A	0.7085	0.4522	0.4261	0.043*	
N5	0.6442 (3)	0.3835 (2)	0.28420 (14)	0.0439 (5)	
N6	0.6035 (3)	0.3101 (2)	0.21699 (14)	0.0480 (5)	
C1	0.9603 (3)	0.6775 (3)	0.91597 (18)	0.0408 (5)	
H1A	1.0540	0.7037	0.8681	0.049*	
C2	0.9530 (3)	0.6782 (3)	1.01344 (19)	0.0467 (6)	
H2B	1.0420	0.7061	1.0326	0.056*	
C3	0.8177 (3)	0.6388 (3)	1.08327 (18)	0.0452 (6)	
H3B	0.8146	0.6389	1.1500	0.054*	
C4	0.6870 (3)	0.5994 (3)	1.05608 (17)	0.0418 (6)	
H4B	0.5946	0.5714	1.1041	0.050*	
C5	0.6905 (3)	0.6005 (2)	0.95854 (16)	0.0364 (5)	
H5B	0.5996	0.5761	0.9395	0.044*	
C6	0.8294 (3)	0.6380 (2)	0.88941 (16)	0.0336 (5)	
C7	0.9194 (3)	0.8149 (3)	0.55833 (18)	0.0433 (6)	
H7A	0.9635	0.8908	0.5786	0.065*	
H7B	0.8206	0.8502	0.5366	0.065*	
H7C	1.0040	0.7683	0.5050	0.065*	
C8	0.8741 (3)	0.7167 (2)	0.64179 (16)	0.0354 (5)	
C9	0.8061 (3)	0.5921 (2)	0.64604 (15)	0.0325 (5)	
C10	0.7862 (3)	0.5469 (2)	0.74127 (16)	0.0320 (5)	
C11	0.8364 (3)	0.3093 (2)	0.77255 (14)	0.0283 (4)	
C12	0.7675 (3)	0.1955 (3)	0.81626 (19)	0.0417 (6)	
H12A	0.6567	0.2036	0.8563	0.050*	

supplementary materials

C13	0.8630 (3)	0.0697 (3)	0.8006 (2)	0.0516 (7)	
H13A	0.8178	-0.0100	0.8302	0.062*	
C14	1.0242 (3)	0.0589 (3)	0.7422 (2)	0.0530 (7)	
H14A	1.0888	-0.0282	0.7308	0.064*	
C15	1.0906 (3)	0.1736 (3)	0.70074 (19)	0.0473 (6)	
H15A	1.2016	0.1657	0.6609	0.057*	
C16	0.9973 (3)	0.3014 (3)	0.71634 (16)	0.0370 (5)	
H16A	1.0438	0.3814	0.6887	0.044*	
C17	0.7669 (3)	0.5307 (2)	0.56726 (16)	0.0329 (5)	
C18	0.6421 (3)	0.3310 (2)	0.53439 (16)	0.0335 (5)	
C19	0.6281 (3)	0.3158 (2)	0.36680 (16)	0.0351 (5)	
C20	0.5602 (3)	0.1932 (3)	0.25151 (17)	0.0428 (6)	
C21	0.5096 (4)	0.0933 (3)	0.1944 (2)	0.0555 (7)	
H21A	0.4153	0.0522	0.2383	0.067*	
H21B	0.4675	0.1455	0.1428	0.067*	
C22	0.6339 (5)	-0.0172 (4)	0.1491 (3)	0.0465 (11)	0.639 (5)
H22A	0.5827	-0.0767	0.1163	0.056*	0.639 (5)
H22B	0.6754	-0.0730	0.1995	0.056*	0.639 (5)
C23	0.7787 (7)	0.0383 (5)	0.0753 (3)	0.0568 (13)	0.639 (5)
H23A	0.8616	-0.0378	0.0465	0.085*	0.639 (5)
H23B	0.8293	0.0972	0.1077	0.085*	0.639 (5)
H23C	0.7381	0.0912	0.0243	0.085*	0.639 (5)
C22'	0.6510 (12)	0.0828 (9)	0.0931 (5)	0.053 (2)	0.361 (5)
H22C	0.6563	0.1730	0.0591	0.064*	0.361 (5)
H22D	0.7611	0.0490	0.1038	0.064*	0.361 (5)
C23'	0.6019 (12)	-0.0149 (10)	0.0362 (6)	0.064 (3)	0.361 (5)
H23D	0.6845	-0.0268	-0.0267	0.096*	0.361 (5)
H23E	0.4931	0.0205	0.0260	0.096*	0.361 (5)
H23F	0.5954	-0.1029	0.0715	0.096*	0.361 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0411 (3)	0.0325 (3)	0.0431 (3)	-0.0066 (2)	-0.0042 (3)	-0.0007 (2)
S2	0.0611 (4)	0.0503 (4)	0.0486 (4)	-0.0317 (3)	-0.0178 (3)	0.0171 (3)
O1	0.0335 (8)	0.0240 (8)	0.0338 (8)	-0.0074 (6)	0.0049 (6)	0.0028 (6)
O2	0.0581 (11)	0.0374 (10)	0.0327 (8)	-0.0179 (8)	-0.0026 (8)	0.0059 (7)
N1	0.0345 (9)	0.0273 (10)	0.0335 (9)	-0.0100 (8)	-0.0016 (8)	0.0017 (8)
N2	0.0399 (10)	0.0260 (10)	0.0380 (10)	-0.0109 (8)	-0.0015 (8)	0.0055 (8)
N3	0.0350 (10)	0.0361 (11)	0.0306 (9)	-0.0125 (8)	-0.0027 (8)	0.0046 (8)
N4	0.0368 (10)	0.0328 (11)	0.0341 (9)	-0.0113 (8)	-0.0009 (8)	0.0025 (8)
N5	0.0511 (12)	0.0401 (12)	0.0344 (10)	-0.0093 (10)	0.0008 (9)	-0.0035 (9)
N6	0.0606 (14)	0.0455 (14)	0.0335 (10)	-0.0092 (11)	-0.0020 (10)	-0.0095 (9)
C1	0.0337 (12)	0.0384 (14)	0.0479 (13)	-0.0099 (10)	-0.0050 (11)	0.0007 (11)
C2	0.0472 (14)	0.0464 (16)	0.0520 (15)	-0.0117 (12)	-0.0202 (12)	-0.0003 (12)
C3	0.0545 (15)	0.0401 (15)	0.0429 (13)	-0.0073 (12)	-0.0157 (12)	-0.0015 (11)
C4	0.0441 (13)	0.0412 (14)	0.0363 (12)	-0.0096 (11)	-0.0024 (11)	-0.0015 (10)
C5	0.0322 (11)	0.0381 (13)	0.0357 (11)	-0.0091 (10)	-0.0016 (9)	-0.0014 (10)

C6	0.0359 (11)	0.0248 (11)	0.0356 (11)	-0.0041 (9)	-0.0029 (9)	0.0021 (9)
C7	0.0466 (13)	0.0353 (14)	0.0425 (13)	-0.0110 (11)	-0.0026 (11)	0.0099 (10)
C8	0.0352 (11)	0.0273 (12)	0.0376 (11)	-0.0055 (9)	0.0004 (10)	0.0014 (9)
C9	0.0313 (11)	0.0259 (11)	0.0332 (11)	-0.0043 (9)	0.0029 (9)	0.0013 (9)
C10	0.0296 (10)	0.0242 (11)	0.0367 (11)	-0.0054 (8)	0.0007 (9)	0.0005 (9)
C11	0.0316 (10)	0.0262 (11)	0.0272 (9)	-0.0060 (8)	-0.0065 (8)	-0.0029 (8)
C12	0.0357 (12)	0.0302 (13)	0.0578 (15)	-0.0106 (10)	-0.0077 (11)	0.0013 (11)
C13	0.0462 (14)	0.0266 (13)	0.084 (2)	-0.0097 (11)	-0.0190 (14)	0.0010 (13)
C14	0.0469 (15)	0.0330 (14)	0.080 (2)	0.0041 (12)	-0.0211 (14)	-0.0101 (13)
C15	0.0344 (12)	0.0494 (16)	0.0518 (14)	0.0023 (11)	-0.0049 (11)	-0.0035 (12)
C16	0.0361 (12)	0.0368 (13)	0.0359 (11)	-0.0075 (10)	-0.0057 (10)	0.0041 (10)
C17	0.0287 (10)	0.0288 (11)	0.0342 (11)	-0.0025 (9)	0.0028 (9)	-0.0005 (9)
C18	0.0266 (10)	0.0362 (13)	0.0346 (11)	-0.0069 (9)	-0.0024 (9)	0.0031 (9)
C19	0.0318 (11)	0.0286 (12)	0.0372 (11)	-0.0041 (9)	0.0035 (9)	-0.0013 (9)
C20	0.0478 (14)	0.0350 (14)	0.0384 (12)	-0.0033 (11)	0.0005 (11)	-0.0074 (10)
C21	0.0705 (19)	0.0468 (17)	0.0462 (15)	-0.0094 (14)	-0.0078 (14)	-0.0112 (13)
C22	0.051 (2)	0.038 (2)	0.054 (2)	-0.0081 (18)	-0.019 (2)	-0.0067 (19)
C23	0.063 (3)	0.052 (3)	0.046 (2)	-0.013 (2)	0.006 (2)	-0.015 (2)
C22'	0.064 (6)	0.050 (5)	0.042 (4)	-0.015 (4)	-0.004 (4)	-0.013 (4)
C23'	0.093 (6)	0.064 (6)	0.042 (4)	-0.034 (5)	-0.017 (4)	-0.006 (4)

Geometric parameters (Å, °)

S1—C19	1.719 (2)	C7—H7C	0.9800
S1—C20	1.737 (2)	C8—C9	1.427 (3)
S2—C18	1.648 (2)	C9—C10	1.381 (3)
O1—C10	1.362 (3)	C9—C17	1.455 (3)
O1—C11	1.411 (3)	C11—C16	1.371 (3)
O2—C17	1.230 (3)	C11—C12	1.378 (3)
N1—C10	1.337 (3)	C12—C13	1.378 (4)
N1—N2	1.380 (2)	C12—H12A	0.9500
N1—C6	1.430 (3)	C13—C14	1.382 (4)
N2—C8	1.328 (3)	C13—H13A	0.9500
N3—C17	1.383 (3)	C14—C15	1.368 (4)
N3—C18	1.384 (3)	C14—H14A	0.9500
N3—H3A	0.8800	C15—C16	1.387 (3)
N4—C18	1.349 (3)	C15—H15A	0.9500
N4—C19	1.384 (3)	C16—H16A	0.9500
N4—H4A	0.8800	C20—C21	1.503 (4)
N5—C19	1.305 (3)	C21—C22	1.453 (5)
N5—N6	1.389 (3)	C21—C22'	1.607 (8)
N6—C20	1.292 (3)	C21—H21A	0.9900
C1—C6	1.379 (3)	C21—H21B	0.9900
C1—C2	1.383 (3)	C22—C23	1.525 (6)
C1—H1A	0.9500	C22—H22A	0.9900
C2—C3	1.382 (4)	C22—H22B	0.9900
C2—H2B	0.9500	C23—H23A	0.9800
C3—C4	1.381 (3)	C23—H23B	0.9800
C3—H3B	0.9500	C23—H23C	0.9800

supplementary materials

C4—C5	1.391 (3)	C22'—C23'	1.479 (10)
C4—H4B	0.9500	C22'—H22C	0.9900
C5—C6	1.392 (3)	C22'—H22D	0.9900
C5—H5B	0.9500	C23'—H23D	0.9800
C7—C8	1.492 (3)	C23'—H23E	0.9800
C7—H7A	0.9800	C23'—H23F	0.9800
C7—H7B	0.9800		
C19—S1—C20	85.87 (11)	C15—C14—C13	120.0 (2)
C10—O1—C11	116.95 (16)	C15—C14—H14A	120.0
C10—N1—N2	110.63 (17)	C13—C14—H14A	120.0
C10—N1—C6	129.28 (19)	C14—C15—C16	120.6 (2)
N2—N1—C6	120.00 (17)	C14—C15—H15A	119.7
C8—N2—N1	105.57 (18)	C16—C15—H15A	119.7
C17—N3—C18	129.42 (18)	C11—C16—C15	118.4 (2)
C17—N3—H3A	115.3	C11—C16—H16A	120.8
C18—N3—H3A	115.3	C15—C16—H16A	120.8
C18—N4—C19	128.0 (2)	O2—C17—N3	121.6 (2)
C18—N4—H4A	116.0	O2—C17—C9	122.9 (2)
C19—N4—H4A	116.0	N3—C17—C9	115.49 (19)
C19—N5—N6	111.0 (2)	N4—C18—N3	114.41 (19)
C20—N6—N5	112.6 (2)	N4—C18—S2	125.85 (18)
C6—C1—C2	118.9 (2)	N3—C18—S2	119.73 (16)
C6—C1—H1A	120.5	N5—C19—N4	117.9 (2)
C2—C1—H1A	120.5	N5—C19—S1	115.74 (18)
C3—C2—C1	120.8 (2)	N4—C19—S1	126.40 (17)
C3—C2—H2B	119.6	N6—C20—C21	123.7 (2)
C1—C2—H2B	119.6	N6—C20—S1	114.75 (18)
C4—C3—C2	120.0 (2)	C21—C20—S1	121.5 (2)
C4—C3—H3B	120.0	C22—C21—C20	118.1 (3)
C2—C3—H3B	120.0	C22—C21—C22'	47.1 (4)
C3—C4—C5	120.1 (2)	C20—C21—C22'	104.9 (3)
C3—C4—H4B	119.9	C22—C21—H21A	107.8
C5—C4—H4B	119.9	C20—C21—H21A	107.8
C4—C5—C6	118.9 (2)	C22'—C21—H21A	146.2
C4—C5—H5B	120.6	C22—C21—H21B	107.8
C6—C5—H5B	120.6	C20—C21—H21B	107.8
C1—C6—C5	121.3 (2)	C22'—C21—H21B	69.9
C1—C6—N1	119.4 (2)	H21A—C21—H21B	107.1
C5—C6—N1	119.32 (19)	C21—C22—C23	110.9 (4)
C8—C7—H7A	109.5	C21—C22—H22A	109.5
C8—C7—H7B	109.5	C23—C22—H22A	109.5
H7A—C7—H7B	109.5	C21—C22—H22B	109.5
C8—C7—H7C	109.5	C23—C22—H22B	109.5
H7A—C7—H7C	109.5	H22A—C22—H22B	108.0
H7B—C7—H7C	109.5	C22—C23—H23A	109.5
N2—C8—C9	111.13 (19)	C22—C23—H23B	109.5
N2—C8—C7	119.6 (2)	H23A—C23—H23B	109.5
C9—C8—C7	129.2 (2)	C22—C23—H23C	109.5
C10—C9—C8	103.68 (19)	H23A—C23—H23C	109.5

C10—C9—C17	129.3 (2)	H23B—C23—H23C	109.5
C8—C9—C17	127.0 (2)	C23'—C22'—C21	105.3 (6)
N1—C10—O1	121.07 (19)	C23'—C22'—H22C	110.7
N1—C10—C9	108.98 (19)	C21—C22'—H22C	110.7
O1—C10—C9	130.0 (2)	C23'—C22'—H22D	110.7
C16—C11—C12	122.1 (2)	C21—C22'—H22D	110.7
C16—C11—O1	123.2 (2)	H22C—C22'—H22D	108.8
C12—C11—O1	114.70 (18)	C22'—C23'—H23D	109.5
C11—C12—C13	118.6 (2)	C22'—C23'—H23E	109.5
C11—C12—H12A	120.7	H23D—C23'—H23E	109.5
C13—C12—H12A	120.7	C22'—C23'—H23F	109.5
C12—C13—C14	120.3 (2)	H23D—C23'—H23F	109.5
C12—C13—H13A	119.8	H23E—C23'—H23F	109.5
C14—C13—H13A	119.8		
C10—N1—N2—C8	0.1 (2)	C11—C12—C13—C14	-0.1 (4)
C6—N1—N2—C8	-176.87 (19)	C12—C13—C14—C15	1.1 (4)
C19—N5—N6—C20	-0.2 (3)	C13—C14—C15—C16	-0.4 (4)
C6—C1—C2—C3	0.6 (4)	C12—C11—C16—C15	2.3 (3)
C1—C2—C3—C4	-0.6 (4)	O1—C11—C16—C15	-175.1 (2)
C2—C3—C4—C5	-0.6 (4)	C14—C15—C16—C11	-1.3 (4)
C3—C4—C5—C6	1.9 (4)	C18—N3—C17—O2	3.3 (4)
C2—C1—C6—C5	0.6 (4)	C18—N3—C17—C9	-177.0 (2)
C2—C1—C6—N1	179.9 (2)	C10—C9—C17—O2	-175.8 (2)
C4—C5—C6—C1	-1.9 (4)	C8—C9—C17—O2	5.0 (4)
C4—C5—C6—N1	178.8 (2)	C10—C9—C17—N3	4.6 (3)
C10—N1—C6—C1	140.6 (2)	C8—C9—C17—N3	-174.7 (2)
N2—N1—C6—C1	-43.1 (3)	C19—N4—C18—N3	-178.9 (2)
C10—N1—C6—C5	-40.1 (3)	C19—N4—C18—S2	1.5 (3)
N2—N1—C6—C5	136.3 (2)	C17—N3—C18—N4	-5.9 (3)
N1—N2—C8—C9	0.4 (2)	C17—N3—C18—S2	173.78 (18)
N1—N2—C8—C7	178.47 (19)	N6—N5—C19—N4	179.78 (19)
N2—C8—C9—C10	-0.7 (2)	N6—N5—C19—S1	-0.4 (3)
C7—C8—C9—C10	-178.6 (2)	C18—N4—C19—N5	173.0 (2)
N2—C8—C9—C17	178.7 (2)	C18—N4—C19—S1	-6.8 (3)
C7—C8—C9—C17	0.9 (4)	C20—S1—C19—N5	0.63 (19)
N2—N1—C10—O1	179.90 (18)	C20—S1—C19—N4	-179.6 (2)
C6—N1—C10—O1	-3.5 (3)	N5—N6—C20—C21	179.9 (2)
N2—N1—C10—C9	-0.6 (2)	N5—N6—C20—S1	0.7 (3)
C6—N1—C10—C9	176.1 (2)	C19—S1—C20—N6	-0.7 (2)
C11—O1—C10—N1	-101.1 (2)	C19—S1—C20—C21	-179.9 (2)
C11—O1—C10—C9	79.5 (3)	N6—C20—C21—C22	97.3 (4)
C8—C9—C10—N1	0.7 (2)	S1—C20—C21—C22	-83.6 (3)
C17—C9—C10—N1	-178.7 (2)	N6—C20—C21—C22'	48.3 (5)
C8—C9—C10—O1	-179.8 (2)	S1—C20—C21—C22'	-132.6 (4)
C17—C9—C10—O1	0.8 (4)	C20—C21—C22—C23	-61.3 (4)
C10—O1—C11—C16	0.9 (3)	C22'—C21—C22—C23	23.1 (5)
C10—O1—C11—C12	-176.69 (18)	C22—C21—C22'—C23'	65.6 (6)
C16—C11—C12—C13	-1.7 (4)	C20—C21—C22'—C23'	-179.7 (6)
O1—C11—C12—C13	175.9 (2)		

supplementary materials

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N3—H3A···O1	0.88	2.18	2.917 (2)	141
N4—H4A···O2	0.88	1.89	2.623 (2)	139

Fig. 1

