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1-Acetonyl-4-(2,5-dimethyl-4*H*-1,2,4-triazol-4-yl)-3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-one

Reşat Ustabaş,[a]* Ufuk Çoruh,[b] Kemal Sancak[c] and Esra Demirkan[c]

[a]Anatolian Trade High School, Merzifon 05300, Amasya, Turkey, [b]Department of Computer Education and Instructional Technology, Educational Faculty, Ondokuz Mayıs University, 55200 Atakum-Samsun, Turkey, and [c]Department of Chemistry, Faculty of Arts and Sciences, Karadeniz Teknik University, 61080 Trabzon, Turkey
Correspondence e-mail: rustabas@omu.edu.tr

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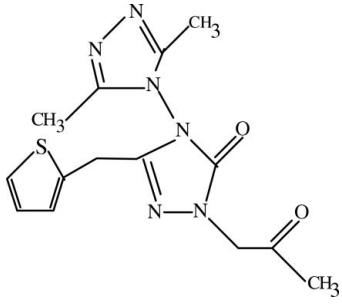
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.033; wR factor = 0.097; data-to-parameter ratio = 13.6.

The thiophene ring of the title compound, $C_{14}H_{16}N_6O_2S$, is disordered over two positions, with a site-occupancy ratio of approximately 5:4, corresponding to rotation of approximately 180° about the single C—C bond. Intermolecular C—H···N and C—H···O interactions stabilize the crystal structure.

Related literature

For related literature, see: Chai *et al.* (2003); Er-Rahimini & Mornet (1992); Ichikawa *et al.* (2001); Jenkins *et al.* (1989); Kim *et al.* (2003); Nakib *et al.* (1994); Sancak *et al.* (2005); Tsuda *et al.* (2004); Ueda (2003); Zhu *et al.* (2000); Çoruh *et al.* (2003).



Experimental

Crystal data

$C_{14}H_{16}N_6O_2S$
M_r = 332.39
Monoclinic, *Cc*
a = 21.1928 (16) Å
b = 9.6058 (6) Å
c = 8.3020 (7) Å
β = 106.696 (6)°
V = 1618.8 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 293 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
9238 measured reflections
3357 independent reflections
3185 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.097
S = 1.06
3357 reflections
246 parameters
179 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1644 Friedel pairs
Flack parameter: −0.02 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22B···N2^{i}	0.96	2.56	3.352 (3)	140
C41—H41A···N3^{ii}	0.97	2.32	3.287 (3)	178
C43—H43···O1^{iii}	0.93	2.49	3.414 (10)	174
C44′—H44′···O2^{iii}	0.93	2.46	3.162 (14)	132
C51—H51A···O2^{iv}	0.97	2.49	3.318 (2)	143

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (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: IS2184).

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supplementary materials

Acta Cryst. (2007). E**63**, o3443 [**doi:10.1107/S160053680703231X**]

1-Acetonyl-4-(2,5-dimethyl-4*H*-1,2,4-triazol-4-yl)-3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-one

R. Ustabas, U. Çoruh, K. Sancak and E. Demirkan

Comment

In a continuing search for pharmacologically active, 1,2,4-triazol and 1,2,4-triazol-5-one compounds, it has been found that most azole fungicides have been developed for diseases of cereal crops; examples include fluconazole (Ichikawa *et al.*, 2001), ravuconazole (Ueda, 2003) and posaconazole (Kim *et al.*, 2003). Moreover, 1,2,4-triazole derivatives have broad-spectrum biological effects, such as insecticidal (Tsuda *et al.*, 2004), herbicidal (Chai *et al.*, 2003), anticonvulsant (Er-Rahimini & Mornet, 1992), antitumor (Nakib *et al.*, 1994) and plant growth regulatory activities (Jenkins *et al.*, 1989).

The title compound, (I), consists of a triazole ring with an acetonyl group substituted at atom N5, a thienylmethyl group substituted at C4, a 1,2,4-triazole ring substituted at N4 atom and an oxo O atom at C3 (Fig. 1). The C1═N2 bond length, 1.298 (3) Å, is a little longer than some values reported in the literature [1.288 (3) Å in $C_{16}H_{28}N_6O_2$ Çoruh *et al.*, 2003) and 1.267 (2) Å in 4-(4-hydroxybenzylidenamino)-4*H*-1,2,4-triazole hemihydrate (Zhu *et al.*, 2000)]. In the central 1,2,4-triazole ring, atoms N2 and N3 have no substituents and the N2—N3 bond length, 1.398 (3) Å, is essentially identical to that [1.403 (8) Å] reported for a similar compound (Sancak *et al.*, 2005). Atom C4 has a trigonal configuration, the sums of the three bond angles around them being 359.99 (13)°.

The thiophene ring is disordered over two positions, corresponding to rotation of approximately 180° about the single C41—C42 bond, with a major-minor ratio of 55.9 (3):44.1 (3). The crystal structure of (I) is stabilized by two C—H···N and five C—H···O intermolecular hydrogen bonds (Table 1).

Experimental

4-(3,5-Dimethyl-4*H*-1,2,4-triazol-4-yl)-3-(2-thienyl methyl)-1*H*-1,2,4-triazol-5(4*H*)-one (0.001 mol) was refluxed with sodium metal (0.001 mol) in absolute ethanol (50 ml) for 1 h. Chloroacetone (0.001 mol) was added and the solution refluxed for 8 h. The resulting solution was filtered and then evaporated under reduced pressure. The solid residue was crystallized from absolute ethanol-diethylether (1:4) (yield 67%; m.p. 493–494 K).

Refinement

The thiophene ring is disordered over two positions about the C41—C42 bond, with major:minor ratio of 55.9 (3):44.1 (3). The geometry of these disordered components were restrained to be similar (SAME in *SHELXL*). The rigid bond and similar displacement parameter restraints (DELU and SIMU, respectively) were applied for the atoms involved. All H atoms were positioned geometrically [C—H = 0.93 (aromatic), 0.96 (methyl) and 0.97 Å (methylene)] and treated as riding on their parent atoms, with U_{iso}(H) = 1.13U_{eq}(aromatic C), 1.5U_{eq}(methyl C) and 1.2U_{eq}(methylene C).

Figures

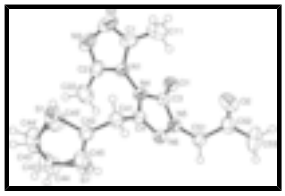


Fig. 1. An *ORTEP* drawing of (I), with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

1-acetonyl-4-(2,5-dimethyl-4*H*-\ 1,2,4-triazol-4-yl)-3-(2-thienylmethyl)-1*H*-1,2,4-triazol-5(4*H*)-one

Crystal data

$C_{14}H_{16}N_6O_2S$	F_{000} = 696
M_r = 332.39	D_x = 1.364 Mg m^{-3}
Monoclinic, *Cc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: C -2yc	Cell parameters from 3185 reflections
a = 21.1928 (16) Å	θ = 2.4–27.1º
b = 9.6058 (6) Å	μ = 0.22 mm^{-1}
c = 8.3020 (7) Å	*T* = 293 (2) K
β = 106.696 (6)º	Prism, colourless
V = 1618.8 (2) Å^3	0.30 × 0.20 × 0.15 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3185 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.039
Monochromator: graphite	θ_{max} = 27.1º
T = 293(2) K	θ_{min} = 2.4º
φ and ω scans	*h* = −27→27
Absorption correction: none	*k* = −12→12
9238 measured reflections	*l* = −10→10
3357 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.126P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.097	$(\Delta/\sigma)_{max} < 0.001$
S = 1.06	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
3357 reflections	$\Delta\rho_{min}$ = −0.18 e Å^{-3}

246 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
179 restraints	Extinction coefficient: 0.015 (3)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1644 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.02 (8)

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 $F^2 > 2\text{sigma}(F^2)$ 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)
O1	0.03169 (6)	0.79946 (12)	−0.49156 (15)	0.0472 (3)	
N1	0.13400 (7)	0.64873 (13)	−0.25347 (17)	0.0422 (3)	
N6	0.11847 (7)	0.99970 (13)	−0.14966 (18)	0.0393 (3)	
N5	0.07161 (7)	0.98340 (12)	−0.30460 (17)	0.0398 (3)	
C41	0.19981 (8)	0.84790 (17)	0.0412 (2)	0.0438 (3)	
H41A	0.1928	0.7556	0.0803	0.053*	
H41B	0.1976	0.9139	0.1278	0.053*	
C42	0.26718 (8)	0.85404 (16)	0.0173 (2)	0.0445 (3)	0.559 (3)
S1	0.32023 (8)	0.71922 (19)	0.0776 (3)	0.0634 (5)	0.559 (3)
C43	0.3784 (4)	0.8126 (10)	0.0046 (16)	0.0627 (17)	0.559 (3)
H43	0.4199	0.7770	0.0110	0.075*	0.559 (3)
C44	0.3585 (4)	0.9386 (11)	−0.0606 (16)	0.0633 (15)	0.559 (3)
H44	0.3831	1.0013	−0.1033	0.076*	0.559 (3)
C45	0.2952 (5)	0.9576 (8)	−0.0527 (14)	0.0614 (16)	0.559 (3)
H45	0.2720	1.0385	−0.0938	0.074*	0.559 (3)
C42'	0.26718 (8)	0.85404 (16)	0.0173 (2)	0.0445 (3)	0.441 (3)
S1'	0.29405 (13)	0.9970 (2)	−0.0653 (4)	0.0565 (5)	0.441 (3)
C43'	0.3687 (5)	0.9102 (13)	−0.047 (2)	0.0600 (18)	0.441 (3)
H43'	0.4027	0.9515	−0.0798	0.072*	0.441 (3)
C44'	0.3743 (7)	0.7795 (13)	0.018 (2)	0.068 (2)	0.441 (3)
H44'	0.4096	0.7185	0.0342	0.082*	0.441 (3)
C45'	0.3165 (5)	0.7580 (9)	0.0556 (15)	0.0654 (19)	0.441 (3)
H45'	0.3105	0.6757	0.1085	0.078*	0.441 (3)
C3	0.06799 (7)	0.85162 (14)	−0.36608 (19)	0.0381 (3)	
C52	−0.03960 (8)	1.0779 (2)	−0.3428 (2)	0.0517 (4)	
O2	−0.05226 (8)	0.97759 (18)	−0.2716 (2)	0.0688 (4)	
N4	0.11834 (6)	0.78629 (12)	−0.24189 (17)	0.0396 (3)	

C4	0.14634 (7)	0.87926 (14)	−0.11427 (19)	0.0372 (3)
C1	0.09964 (10)	0.53665 (16)	−0.2186 (2)	0.0487 (4)
N3	0.17852 (10)	0.46323 (18)	−0.3180 (3)	0.0668 (5)
N2	0.12657 (10)	0.42417 (16)	−0.2552 (2)	0.0648 (5)
C51	0.02666 (8)	1.09491 (16)	−0.3740 (2)	0.0442 (3)
H51A	0.0205	1.0996	−0.4943	0.053*
H51B	0.0460	1.1823	−0.3252	0.053*
C2	0.18161 (9)	0.59797 (19)	−0.3193 (2)	0.0506 (4)
C11	0.04103 (12)	0.5476 (2)	−0.1593 (3)	0.0646 (5)
H11A	0.0328	0.6437	−0.1408	0.097*
H11B	0.0482	0.4970	−0.0559	0.097*
H11C	0.0038	0.5093	−0.2423	0.097*
C53	−0.08696 (15)	1.1932 (4)	−0.4126 (5)	0.0988 (10)
H53A	−0.1278	1.1756	−0.3880	0.148*
H53B	−0.0946	1.1981	−0.5322	0.148*
H53C	−0.0688	1.2798	−0.3625	0.148*
C22	0.22523 (12)	0.6864 (3)	−0.3854 (3)	0.0695 (6)
H22A	0.2169	0.7825	−0.3674	0.104*
H22B	0.2169	0.6695	−0.5037	0.104*
H22C	0.2703	0.6647	−0.3283	0.104*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0443 (6)	0.0462 (6)	0.0463 (7)	−0.0034 (4)	0.0053 (5)	−0.0036 (4)
N1	0.0450 (6)	0.0316 (6)	0.0473 (7)	0.0032 (5)	0.0089 (5)	−0.0028 (5)
N6	0.0380 (6)	0.0364 (6)	0.0433 (7)	0.0000 (4)	0.0115 (5)	−0.0026 (5)
N5	0.0375 (5)	0.0363 (6)	0.0430 (7)	0.0038 (4)	0.0074 (5)	−0.0014 (5)
C41	0.0437 (7)	0.0436 (7)	0.0413 (9)	−0.0009 (6)	0.0078 (6)	−0.0003 (6)
C42	0.0420 (7)	0.0429 (7)	0.0433 (8)	0.0031 (6)	0.0038 (6)	−0.0019 (6)
S1	0.0519 (5)	0.0596 (9)	0.0741 (9)	0.0193 (6)	0.0109 (5)	0.0121 (6)
C43	0.0359 (17)	0.068 (4)	0.079 (3)	0.007 (2)	0.0082 (18)	−0.002 (3)
C44	0.049 (3)	0.064 (4)	0.074 (3)	0.005 (2)	0.013 (2)	0.002 (3)
C45	0.054 (2)	0.057 (3)	0.067 (3)	0.009 (3)	0.0088 (18)	0.003 (3)
C42'	0.0420 (7)	0.0429 (7)	0.0433 (8)	0.0031 (6)	0.0038 (6)	−0.0019 (6)
S1'	0.0475 (7)	0.0541 (11)	0.0683 (9)	−0.0008 (8)	0.0174 (6)	0.0090 (9)
C43'	0.043 (3)	0.060 (5)	0.078 (4)	0.010 (2)	0.018 (3)	0.008 (3)
C44'	0.053 (3)	0.062 (4)	0.084 (3)	0.013 (3)	0.010 (3)	0.012 (3)
C45'	0.062 (3)	0.053 (4)	0.072 (4)	0.013 (3)	0.004 (2)	0.011 (3)
C3	0.0361 (6)	0.0366 (6)	0.0416 (8)	−0.0014 (5)	0.0111 (5)	−0.0003 (6)
C52	0.0412 (8)	0.0618 (10)	0.0495 (10)	0.0039 (7)	0.0091 (7)	−0.0066 (8)
O2	0.0551 (7)	0.0827 (10)	0.0708 (11)	−0.0165 (7)	0.0218 (7)	−0.0037 (7)
N4	0.0405 (6)	0.0312 (5)	0.0443 (8)	0.0022 (4)	0.0074 (5)	−0.0042 (4)
C4	0.0357 (6)	0.0355 (7)	0.0413 (8)	−0.0022 (5)	0.0127 (5)	−0.0026 (5)
C1	0.0615 (10)	0.0363 (7)	0.0411 (9)	−0.0047 (6)	0.0031 (7)	0.0001 (6)
N3	0.0782 (11)	0.0488 (8)	0.0673 (11)	0.0169 (8)	0.0111 (9)	−0.0156 (7)
N2	0.0870 (12)	0.0369 (7)	0.0619 (11)	0.0018 (7)	0.0078 (9)	−0.0044 (6)
C51	0.0442 (7)	0.0380 (7)	0.0484 (9)	0.0064 (6)	0.0102 (6)	0.0020 (6)

C2	0.0495 (8)	0.0488 (8)	0.0485 (9)	0.0093 (7)	0.0062 (7)	−0.0115 (7)
C11	0.0683 (12)	0.0631 (11)	0.0615 (12)	−0.0164 (10)	0.0171 (10)	0.0037 (9)
C53	0.0668 (14)	0.112 (2)	0.116 (2)	0.0472 (15)	0.0246 (15)	0.0128 (17)
C22	0.0564 (10)	0.0831 (14)	0.0750 (15)	−0.0073 (9)	0.0284 (10)	−0.0232 (11)

Geometric parameters (Å, °)

O1—C3	1.2114 (19)	C44'—H44'	0.9300
N1—C2	1.368 (2)	C45'—H45'	0.9300
N1—N4	1.3726 (17)	C3—N4	1.4011 (19)
N1—C1	1.376 (2)	C52—O2	1.201 (3)
N6—C4	1.2939 (19)	C52—C53	1.495 (3)
N6—N5	1.3897 (19)	C52—C51	1.508 (2)
N5—C3	1.3587 (18)	N4—C4	1.3824 (19)
N5—C51	1.4392 (19)	C1—N2	1.298 (3)
C41—C4	1.483 (2)	C1—C11	1.465 (3)
C41—C42	1.498 (2)	N3—C2	1.296 (3)
C41—H41A	0.9700	N3—N2	1.398 (3)
C41—H41B	0.9700	C51—H51A	0.9700
C42—C45	1.371 (9)	C51—H51B	0.9700
C42—S1	1.694 (2)	C2—C22	1.473 (3)
S1—C43	1.766 (8)	C11—H11A	0.9600
C43—C44	1.343 (8)	C11—H11B	0.9600
C43—H43	0.9300	C11—H11C	0.9600
C44—C45	1.375 (13)	C53—H53A	0.9600
C44—H44	0.9300	C53—H53B	0.9600
C45—H45	0.9300	C53—H53C	0.9600
S1'—C43'	1.756 (10)	C22—H22A	0.9600
C43'—C44'	1.358 (10)	C22—H22B	0.9600
C43'—H43'	0.9300	C22—H22C	0.9600
C44'—C45'	1.362 (16)		
C2—N1—N4	126.30 (14)	N1—N4—C4	128.05 (12)
C2—N1—C1	107.52 (14)	N1—N4—C3	121.87 (12)
N4—N1—C1	125.81 (14)	C4—N4—C3	110.07 (12)
C4—N6—N5	105.52 (12)	N6—C4—N4	109.64 (13)
C3—N5—N6	113.63 (12)	N6—C4—C41	124.33 (14)
C3—N5—C51	125.70 (14)	N4—C4—C41	126.02 (13)
N6—N5—C51	120.09 (13)	N2—C1—N1	107.86 (18)
C4—C41—C42	113.30 (12)	N2—C1—C11	127.64 (18)
C4—C41—H41A	108.9	N1—C1—C11	124.42 (15)
C42—C41—H41A	108.9	C2—N3—N2	108.53 (15)
C4—C41—H41B	108.9	C1—N2—N3	108.08 (16)
C42—C41—H41B	108.9	N5—C51—C52	113.11 (14)
H41A—C41—H41B	107.7	N5—C51—H51A	109.0
C45—C42—C41	129.1 (4)	C52—C51—H51A	109.0
C45—C42—S1	110.4 (4)	N5—C51—H51B	109.0
C41—C42—S1	120.51 (14)	C52—C51—H51B	109.0
C42—S1—C43	88.8 (4)	H51A—C51—H51B	107.8
C44—C43—S1	115.3 (9)	N3—C2—N1	107.94 (18)

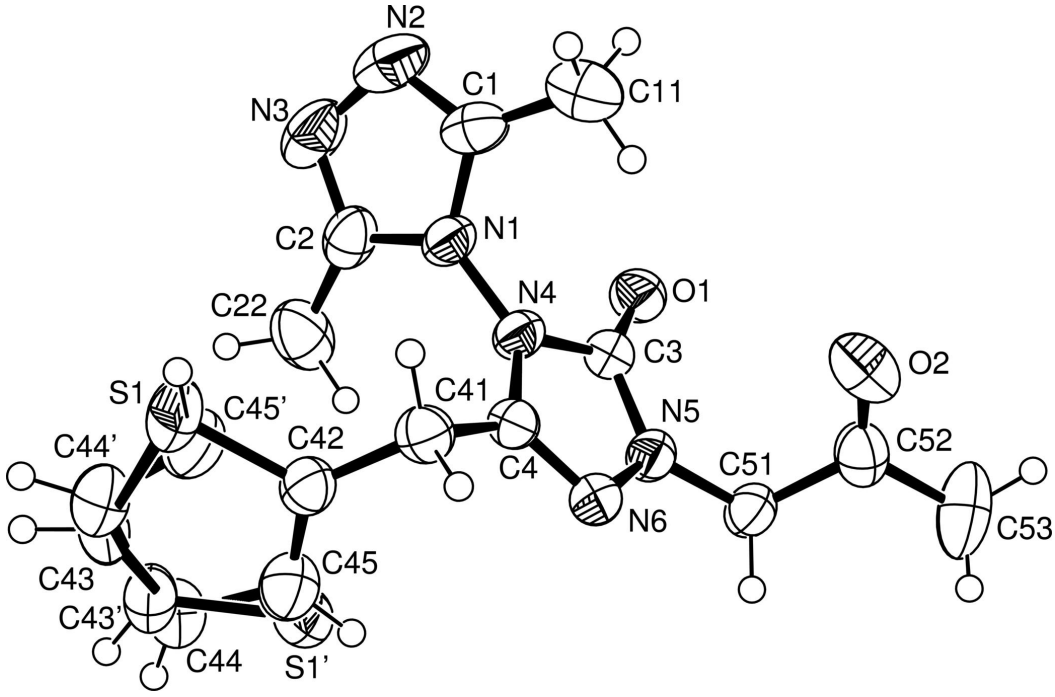
C44—C43—H43	122.3	N3—C2—C22	128.11 (17)
S1—C43—H43	122.3	N1—C2—C22	123.88 (17)
C43—C44—C45	107.3 (10)	C1—C11—H11A	109.5
C43—C44—H44	126.4	C1—C11—H11B	109.5
C45—C44—H44	126.4	H11A—C11—H11B	109.5
C42—C45—C44	118.2 (8)	C1—C11—H11C	109.5
C42—C45—H45	120.9	H11A—C11—H11C	109.5
C44—C45—H45	120.9	H11B—C11—H11C	109.5
C44'—C43'—S1'	116.6 (12)	C52—C53—H53A	109.5
C44'—C43'—H43'	121.7	C52—C53—H53B	109.5
S1'—C43'—H43'	121.7	H53A—C53—H53B	109.5
C43'—C44'—C45'	104.4 (13)	C52—C53—H53C	109.5
C43'—C44'—H44'	127.8	H53A—C53—H53C	109.5
C45'—C44'—H44'	127.8	H53B—C53—H53C	109.5
C44'—C45'—H45'	119.2	C2—C22—H22A	109.5
O1—C3—N5	131.43 (14)	C2—C22—H22B	109.5
O1—C3—N4	127.49 (13)	H22A—C22—H22B	109.5
N5—C3—N4	101.07 (12)	C2—C22—H22C	109.5
O2—C52—C53	124.4 (2)	H22A—C22—H22C	109.5
O2—C52—C51	121.49 (16)	H22B—C22—H22C	109.5
C53—C52—C51	114.1 (2)		
C4—N6—N5—C3	1.96 (16)	N5—N6—C4—C41	−179.87 (13)
C4—N6—N5—C51	173.77 (13)	N1—N4—C4—N6	179.59 (14)
C4—C41—C42—C45	50.9 (6)	C3—N4—C4—N6	−1.31 (16)
C4—C41—C42—S1	−128.07 (16)	N1—N4—C4—C41	−0.9 (2)
C45—C42—S1—C43	0.3 (7)	C3—N4—C4—C41	178.23 (13)
C41—C42—S1—C43	179.5 (4)	C42—C41—C4—N6	−97.73 (17)
C42—S1—C43—C44	0.3 (9)	C42—C41—C4—N4	82.80 (18)
S1—C43—C44—C45	−0.8 (13)	C2—N1—C1—N2	2.27 (19)
C41—C42—C45—C44	−180.0 (8)	N4—N1—C1—N2	175.59 (15)
S1—C42—C45—C44	−0.9 (12)	C2—N1—C1—C11	−174.74 (19)
C43—C44—C45—C42	1.1 (15)	N4—N1—C1—C11	−1.4 (3)
S1'—C43'—C44'—C45'	−1.8 (18)	N1—C1—N2—N3	−1.1 (2)
N6—N5—C3—O1	176.56 (14)	C11—C1—N2—N3	175.8 (2)
C51—N5—C3—O1	5.3 (3)	C2—N3—N2—C1	−0.5 (2)
N6—N5—C3—N4	−2.60 (16)	C3—N5—C51—C52	72.9 (2)
C51—N5—C3—N4	−173.87 (13)	N6—N5—C51—C52	−97.89 (16)
C2—N1—N4—C4	−87.1 (2)	O2—C52—C51—N5	−2.3 (2)
C1—N1—N4—C4	100.82 (19)	C53—C52—C51—N5	179.6 (2)
C2—N1—N4—C3	93.89 (19)	N2—N3—C2—N1	1.9 (2)
C1—N1—N4—C3	−78.2 (2)	N2—N3—C2—C22	−175.4 (2)
O1—C3—N4—N1	2.3 (2)	N4—N1—C2—N3	−175.84 (15)
N5—C3—N4—N1	−178.52 (13)	C1—N1—C2—N3	−2.57 (19)
O1—C3—N4—C4	−176.90 (14)	N4—N1—C2—C22	1.6 (3)
N5—C3—N4—C4	2.31 (15)	C1—N1—C2—C22	174.83 (19)
N5—N6—C4—N4	−0.33 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22B···N2^{i}	0.96	2.56	3.352 (3)	140
C41—H41A···N3^{ii}	0.97	2.32	3.287 (3)	178
C43—H43···O1^{iii}	0.93	2.49	3.414 (10)	174
C44'—H44'···O2^{iii}	0.93	2.46	3.162 (14)	132
C51—H51A···O2^{iv}	0.97	2.49	3.318 (2)	143

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

Fig. 1


N2
C1
C11
N3
C2
N1
C22
N4
O1
C3
O2
S1
C45'
C41
N5
C44'
C42
C4
C51
C52
N6
C53
C45
C43
C43'
C44
S1'