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Ethyl 3-acetyl-4-(3-methoxyphenyl)-6-methyl-2-sulfanylidene-1,2,3,4-tetrahydropyrimidine-5-carboxylate

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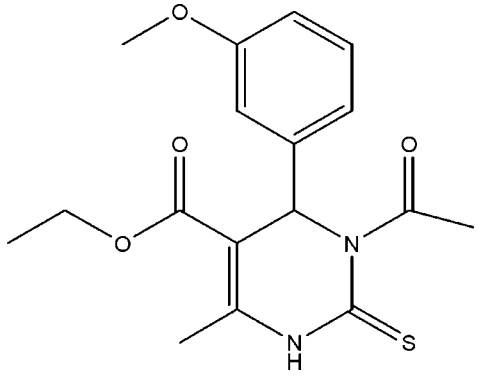
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.063; wR factor = 0.204; data-to-parameter ratio = 16.8.

In the title compound, $C_{17}H_{20}N_2O_4S$, the aryl ring is positioned perpendicular to the dihydropyrimidine ring, the dihedral angle between the ring planes being 77.48 (9)°. The carboxylate and methyl groups are in a *cis* conformation with respect to the C=C bond. The dihydropyrimidine ring adopts a twist-boat conformation. The crystal structure is stabilized by N—H···O and C—H···O interactions, the former resulting in molecular chains along the *b* axis and the latter forming inversion dimers.

Related literature

For the biological activity of dihydropyrimidines, see: Kappe (2000). For a related structure, see: Begum & Vasundhara (2009).



Experimental

Crystal data

$C_{17}H_{20}N_2O_4S$
M_r = 348.41
Monoclinic, $P2_1/n$
a = 11.515 (2) Å
b = 7.3687 (16) Å
c = 20.049 (4) Å
β = 90.960 (4)°
V = 1701.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 296 K
0.18 × 0.16 × 0.16 mm

Data collection

Bruker SMART APEX CCD detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.963, T_{max} = 0.967
10054 measured reflections
3707 independent reflections
2545 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.204
S = 1.03
3707 reflections
221 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.86	2.07	2.907 (3)	164
C3—H3B···O1^{ii}	0.96	2.60	3.393 (4)	140

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1996); 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: PV2529).

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supporting information

Acta Cryst. (2012). E**68**, o1555 [doi:10.1107/S1600536812017679]

Ethyl 3-acetyl-4-(3-methoxyphenyl)-6-methyl-2-sulfanylidene-1,2,3,4-tetrahydropyrimidine-5-carboxylate

Nikhath Fathima, H Nagarajaiah and Noor Shahina Begum

S1. Comment

Dihydropyrimidines have remarkable potency with antiviral, antitumor, antibacterial and anti-inflammatory activities, and are used as antihypertensive agents and calcium channel modulators (Kappe, 2000). In the structure of the title compound, the aryl ring substituted to chiral carbon atom (C4) is positioned axially to the dihydropyrimidine ring, whereas the carboxylate, methyl, acetyl and the thioxo groups are attached on the either sides of the ring. The dihedral angle between the planes of the aryl and dihydropyrimidine rings is 77.48 (9)°. The exocyclic ester at C5 adopts a *cis* orientation with respect to C5═C6 double bond. The central dihydropyrimidine ring is significantly puckered, assuming a conformation of twisted boat with the atoms N1 and C1 displaced from the mean plane of the remaining ring atoms (C4/C5/C6/N2) by 0.806 (4) Å and 0.517 (4) Å, respectively. The crystal structure is stabilized by intermolecular interactions N—H···O resulting in molecular chains along the crystallographic *b*-axis. The structure is further consolidated by intermolecular C—H···O interactions resulting in the formation of centrosymmetric dimers about inversion centers (Fig 2.) For a crystal structure related to the title compound, see: Begum & Vasundhara (2009).

S2. Experimental

4-(3-Methoxy-phenyl)-6-methyl-2-thioxo-1,2,3,4-tetrahydro-pyrimidine-5 carboxylic acid ethyl ester (3.06 g, 10 mmol) was mixed with acetic anhydride (20 ml) and refluxed for about 4 h. The reaction mixture was cooled and diluted by addition of water (20 ml). The solid separated, washed with water, filtered and dried (Yield: 1.92 g, 85% and mp 430-432 K). Pale yellow crystals of the title compound were obtained for diffraction by slow evaporation from a solution in chloroform.

S3. Refinement

The H atoms were placed at calculated positions in the riding model approximation with N—H = 0.86Å, C—H = 0.93, 0.96, 0.97 and 0.98 Å for aryl, methyl, methylene and methyne H-atoms respectively, with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) = 1.2U_{eq}(N/C).

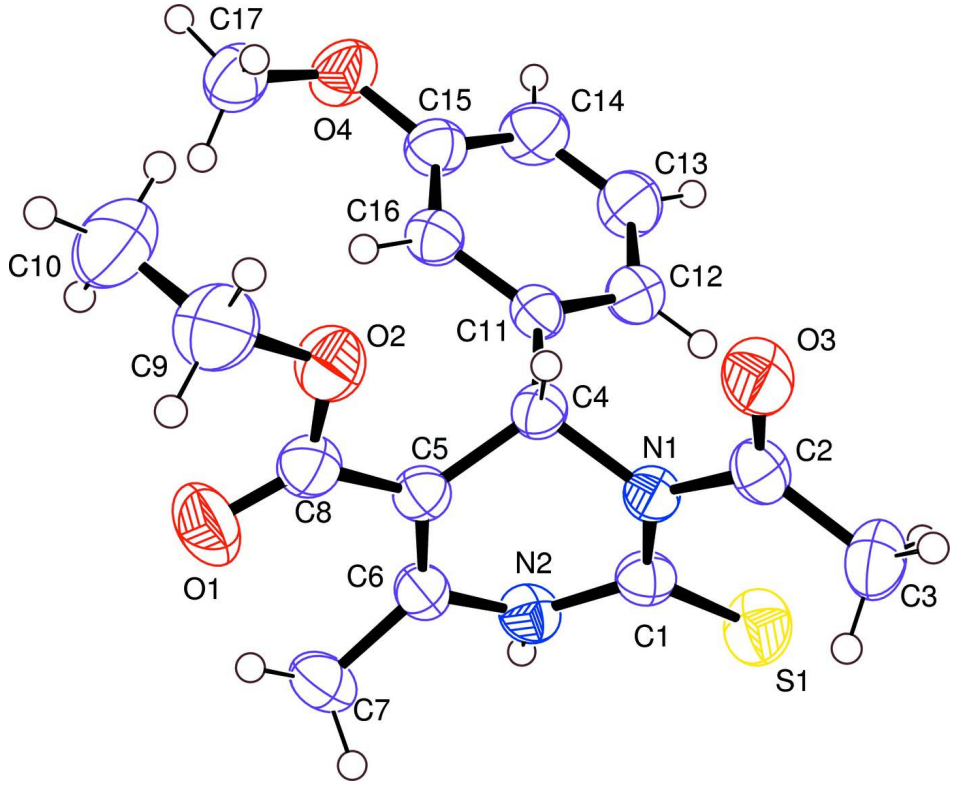

Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

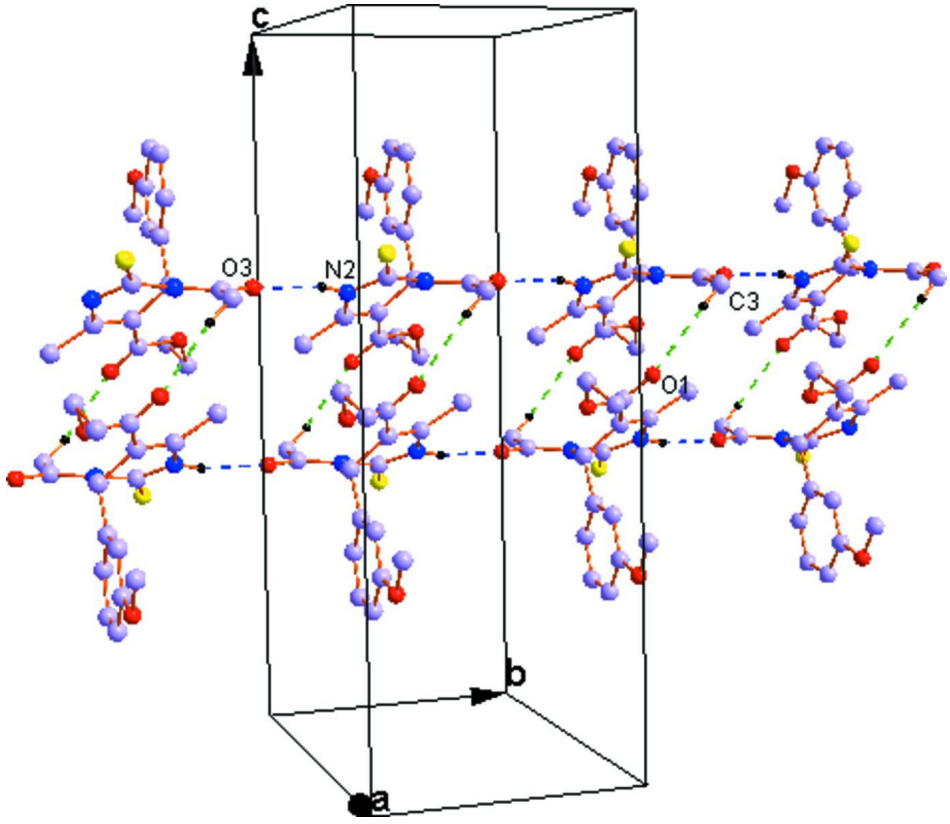

Figure 2

A view of the intermolecular hydrogen bonds (dotted lines) in the crystal structure of the title compound. H atoms non participating in H-bonding were ommitted for clarity.

Ethyl 3-acetyl-4-(3-methoxyphenyl)-6-methyl-2-sulfanylidene-1,2,3,4- tetrahydropyrimidine-5-carboxylate

Crystal data

$C_{17}H_{20}N_2O_4S$
$M_r = 348.41$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.515$ (2) Å
$b = 7.3687$ (16) Å
$c = 20.049$ (4) Å
$\beta = 90.960$ (4)°
$V = 1701.0$ (6) Å^3
$Z = 4$
$F(000) = 736$
$D_x = 1.360$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3707 reflections
$\theta = 2.0$–27.0°
$\mu = 0.21$ mm^{-1}
$T = 296$ K
Block, yellow
0.18 × 0.16 × 0.16 mm

Data collection

Bruker SMART APEX CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min} = 0.963$, $T_{max} = 0.967$
10054 measured reflections
3707 independent reflections
2545 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.0°$
$h = -12 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -23 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.204$
$S = 1.03$
3707 reflections
221 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1146P)^2 + 0.3834P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6452 (2)	0.0344 (3)	0.14742 (12)	0.0409 (6)
C2	0.6628 (2)	−0.3005 (3)	0.13502 (13)	0.0425 (6)
C3	0.7912 (2)	−0.2995 (4)	0.12735 (17)	0.0603 (8)
H3A	0.8280	−0.2751	0.1697	0.090*
H3B	0.8123	−0.2072	0.0960	0.090*
H3C	0.8162	−0.4157	0.1113	0.090*
C4	0.4706 (2)	−0.1548 (3)	0.13863 (12)	0.0362 (5)
H4	0.4507	−0.2765	0.1224	0.043*
C5	0.4207 (2)	−0.0197 (3)	0.08906 (11)	0.0377 (5)
C6	0.4725 (2)	0.1430 (3)	0.08596 (12)	0.0392 (6)
C7	0.4401 (3)	0.3040 (4)	0.04509 (14)	0.0525 (7)
H7A	0.3746	0.2748	0.0169	0.079*
H7B	0.5045	0.3383	0.0180	0.079*
H7C	0.4204	0.4029	0.0740	0.079*
C8	0.3207 (2)	−0.0652 (4)	0.04555 (13)	0.0485 (7)
C9	0.1922 (3)	−0.2962 (5)	0.00900 (18)	0.0734 (10)
H9A	0.1954	−0.4267	0.0031	0.088*
H9B	0.1998	−0.2402	−0.0345	0.088*
C10	0.0772 (3)	−0.2451 (7)	0.0375 (2)	0.0975 (14)
H10A	0.0713	−0.2945	0.0817	0.146*
H10B	0.0158	−0.2929	0.0098	0.146*
H10C	0.0709	−0.1153	0.0395	0.146*
C11	0.4234 (2)	−0.1375 (3)	0.20943 (12)	0.0380 (5)
C12	0.4961 (2)	−0.1400 (4)	0.26504 (13)	0.0481 (6)
H12	0.5763	−0.1422	0.2603	0.058*

C13	0.4480 (3)	−0.1390 (4)	0.32820 (14)	0.0586 (8)
H13	0.4968	−0.1423	0.3656	0.070*
C14	0.3303 (3)	−0.1334 (4)	0.33644 (14)	0.0575 (8)
H14	0.2998	−0.1332	0.3791	0.069*
C15	0.2568 (2)	−0.1281 (4)	0.28122 (14)	0.0480 (6)
C16	0.3032 (2)	−0.1295 (3)	0.21776 (13)	0.0428 (6)
H16	0.2540	−0.1251	0.1805	0.051*
C17	0.0614 (3)	−0.1270 (5)	0.23868 (15)	0.0603 (8)
H17A	0.0741	−0.0239	0.2105	0.090*
H17B	−0.0168	−0.1248	0.2544	0.090*
H17C	0.0737	−0.2365	0.2138	0.090*
O1	0.2712 (2)	0.0368 (4)	0.00752 (12)	0.0801 (8)
O2	0.28828 (17)	−0.2390 (3)	0.05194 (10)	0.0567 (5)
O3	0.60926 (17)	−0.4423 (3)	0.13563 (11)	0.0606 (6)
O4	0.14055 (17)	−0.1211 (3)	0.29420 (10)	0.0607 (6)
N1	0.59906 (16)	−0.1391 (3)	0.13862 (10)	0.0368 (5)
N2	0.57304 (19)	0.1684 (3)	0.12438 (11)	0.0434 (5)
H2	0.5913	0.2783	0.1345	0.052*
S1	0.76922 (7)	0.08775 (12)	0.18297 (5)	0.0651 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0441 (13)	0.0361 (13)	0.0426 (13)	−0.0028 (10)	0.0040 (10)	−0.0003 (10)
C2	0.0491 (14)	0.0370 (14)	0.0413 (13)	0.0064 (11)	−0.0005 (11)	−0.0020 (10)
C3	0.0470 (16)	0.0604 (19)	0.073 (2)	0.0127 (14)	−0.0017 (14)	−0.0011 (15)
C4	0.0344 (12)	0.0333 (12)	0.0408 (13)	−0.0016 (9)	0.0001 (10)	−0.0020 (9)
C5	0.0386 (12)	0.0397 (13)	0.0349 (12)	0.0014 (10)	0.0043 (10)	−0.0005 (10)
C6	0.0426 (13)	0.0398 (14)	0.0353 (12)	0.0066 (10)	0.0037 (10)	0.0016 (10)
C7	0.0661 (18)	0.0433 (15)	0.0484 (16)	0.0069 (13)	0.0059 (13)	0.0105 (12)
C8	0.0436 (14)	0.0605 (18)	0.0414 (14)	−0.0028 (13)	0.0010 (11)	0.0043 (12)
C9	0.073 (2)	0.080 (2)	0.066 (2)	−0.0215 (18)	−0.0174 (17)	−0.0050 (18)
C10	0.059 (2)	0.134 (4)	0.099 (3)	−0.022 (2)	−0.010 (2)	0.016 (3)
C11	0.0412 (13)	0.0338 (13)	0.0391 (13)	0.0014 (10)	−0.0008 (10)	0.0037 (10)
C12	0.0457 (14)	0.0522 (16)	0.0464 (15)	−0.0006 (12)	−0.0009 (11)	0.0042 (12)
C13	0.0581 (18)	0.077 (2)	0.0406 (15)	−0.0021 (15)	−0.0058 (12)	0.0020 (14)
C14	0.0617 (18)	0.072 (2)	0.0388 (15)	−0.0040 (15)	0.0076 (13)	−0.0009 (13)
C15	0.0484 (15)	0.0483 (16)	0.0476 (15)	0.0005 (12)	0.0083 (12)	0.0007 (12)
C16	0.0445 (14)	0.0444 (15)	0.0395 (13)	0.0005 (11)	0.0018 (11)	0.0033 (11)
C17	0.0472 (16)	0.075 (2)	0.0592 (18)	0.0029 (14)	0.0061 (13)	0.0026 (15)
O1	0.0780 (16)	0.0834 (17)	0.0776 (16)	−0.0116 (13)	−0.0360 (13)	0.0318 (13)
O2	0.0535 (12)	0.0591 (13)	0.0570 (12)	−0.0108 (9)	−0.0138 (9)	−0.0023 (9)
O3	0.0610 (13)	0.0308 (10)	0.0900 (17)	0.0025 (9)	−0.0015 (11)	−0.0033 (9)
O4	0.0478 (11)	0.0860 (16)	0.0487 (12)	−0.0003 (10)	0.0123 (9)	−0.0003 (10)
N1	0.0379 (11)	0.0323 (11)	0.0403 (11)	−0.0005 (8)	0.0005 (8)	−0.0009 (8)
N2	0.0506 (12)	0.0293 (11)	0.0504 (13)	−0.0006 (9)	0.0024 (10)	−0.0021 (9)
S1	0.0572 (5)	0.0578 (5)	0.0797 (6)	−0.0096 (4)	−0.0160 (4)	−0.0046 (4)

Geometric parameters (Å, º)

C1—N2	1.366 (3)	C9—C10	1.500 (6)
C1—N1	1.395 (3)	C9—H9A	0.9700
C1—S1	1.633 (3)	C9—H9B	0.9700
C2—O3	1.213 (3)	C10—H10A	0.9600
C2—N1	1.400 (3)	C10—H10B	0.9600
C2—C3	1.489 (4)	C10—H10C	0.9600
C3—H3A	0.9600	C11—C12	1.383 (4)
C3—H3B	0.9600	C11—C16	1.398 (3)
C3—H3C	0.9600	C12—C13	1.391 (4)
C4—N1	1.484 (3)	C12—H12	0.9300
C4—C5	1.513 (3)	C13—C14	1.369 (4)
C4—C11	1.534 (3)	C13—H13	0.9300
C4—H4	0.9800	C14—C15	1.383 (4)
C5—C6	1.341 (3)	C14—H14	0.9300
C5—C8	1.471 (3)	C15—O4	1.369 (3)
C6—N2	1.393 (3)	C15—C16	1.388 (4)
C6—C7	1.486 (4)	C16—H16	0.9300
C7—H7A	0.9600	C17—O4	1.427 (4)
C7—H7B	0.9600	C17—H17A	0.9600
C7—H7C	0.9600	C17—H17B	0.9600
C8—O1	1.207 (3)	C17—H17C	0.9600
C8—O2	1.341 (4)	N2—H2	0.8600
C9—O2	1.453 (4)		
N2—C1—N1	113.0 (2)	C9—C10—H10A	109.5
N2—C1—S1	119.74 (19)	C9—C10—H10B	109.5
N1—C1—S1	127.20 (19)	H10A—C10—H10B	109.5
O3—C2—N1	117.6 (2)	C9—C10—H10C	109.5
O3—C2—C3	120.7 (2)	H10A—C10—H10C	109.5
N1—C2—C3	121.6 (2)	H10B—C10—H10C	109.5
C2—C3—H3A	109.5	C12—C11—C16	119.4 (2)
C2—C3—H3B	109.5	C12—C11—C4	121.7 (2)
H3A—C3—H3B	109.5	C16—C11—C4	118.7 (2)
C2—C3—H3C	109.5	C11—C12—C13	119.3 (3)
H3A—C3—H3C	109.5	C11—C12—H12	120.4
H3B—C3—H3C	109.5	C13—C12—H12	120.4
N1—C4—C5	108.44 (18)	C14—C13—C12	121.4 (3)
N1—C4—C11	111.24 (19)	C14—C13—H13	119.3
C5—C4—C11	114.62 (19)	C12—C13—H13	119.3
N1—C4—H4	107.4	C13—C14—C15	119.9 (3)
C5—C4—H4	107.4	C13—C14—H14	120.1
C11—C4—H4	107.4	C15—C14—H14	120.1
C6—C5—C8	121.3 (2)	O4—C15—C14	115.9 (2)
C6—C5—C4	117.0 (2)	O4—C15—C16	124.6 (3)
C8—C5—C4	121.6 (2)	C14—C15—C16	119.6 (3)
C5—C6—N2	117.5 (2)	C15—C16—C11	120.5 (2)

C5—C6—C7	129.2 (2)	C15—C16—H16	119.8
N2—C6—C7	113.3 (2)	C11—C16—H16	119.8
C6—C7—H7A	109.5	O4—C17—H17A	109.5
C6—C7—H7B	109.5	O4—C17—H17B	109.5
H7A—C7—H7B	109.5	H17A—C17—H17B	109.5
C6—C7—H7C	109.5	O4—C17—H17C	109.5
H7A—C7—H7C	109.5	H17A—C17—H17C	109.5
H7B—C7—H7C	109.5	H17B—C17—H17C	109.5
O1—C8—O2	121.7 (3)	C8—O2—C9	115.5 (2)
O1—C8—C5	126.1 (3)	C15—O4—C17	117.6 (2)
O2—C8—C5	112.2 (2)	C1—N1—C2	125.9 (2)
O2—C9—C10	111.7 (3)	C1—N1—C4	116.71 (19)
O2—C9—H9A	109.3	C2—N1—C4	117.22 (19)
C10—C9—H9A	109.3	C1—N2—C6	125.8 (2)
O2—C9—H9B	109.3	C1—N2—H2	117.1
C10—C9—H9B	109.3	C6—N2—H2	117.1
H9A—C9—H9B	107.9		

N1—C4—C5—C6	−38.9 (3)	C12—C11—C16—C15	−1.4 (4)
C11—C4—C5—C6	86.0 (3)	C4—C11—C16—C15	175.0 (2)
N1—C4—C5—C8	140.3 (2)	O1—C8—O2—C9	1.5 (4)
C11—C4—C5—C8	−94.7 (3)	C5—C8—O2—C9	−178.2 (2)
C8—C5—C6—N2	−174.1 (2)	C10—C9—O2—C8	−82.3 (4)
C4—C5—C6—N2	5.2 (3)	C14—C15—O4—C17	−176.1 (2)
C8—C5—C6—C7	3.9 (4)	C16—C15—O4—C17	4.1 (4)
C4—C5—C6—C7	−176.8 (2)	N2—C1—N1—C2	156.5 (2)
C6—C5—C8—O1	−5.4 (4)	S1—C1—N1—C2	−25.5 (4)
C4—C5—C8—O1	175.4 (3)	N2—C1—N1—C4	−28.8 (3)
C6—C5—C8—O2	174.3 (2)	S1—C1—N1—C4	149.21 (19)
C4—C5—C8—O2	−4.9 (3)	O3—C2—N1—C1	171.0 (2)
N1—C4—C11—C12	−10.5 (3)	C3—C2—N1—C1	−12.0 (4)
C5—C4—C11—C12	−134.0 (2)	O3—C2—N1—C4	−3.7 (3)
N1—C4—C11—C16	173.2 (2)	C3—C2—N1—C4	173.4 (2)
C5—C4—C11—C16	49.7 (3)	C5—C4—N1—C1	52.0 (3)
C16—C11—C12—C13	1.6 (4)	C11—C4—N1—C1	−75.0 (2)
C4—C11—C12—C13	−174.7 (3)	C5—C4—N1—C2	−132.9 (2)
C11—C12—C13—C14	−0.8 (5)	C11—C4—N1—C2	100.2 (2)
C12—C13—C14—C15	−0.2 (5)	N1—C1—N2—C6	−10.4 (3)
C13—C14—C15—O4	−179.5 (3)	S1—C1—N2—C6	171.47 (19)
C13—C14—C15—C16	0.4 (4)	C5—C6—N2—C1	22.8 (4)
O4—C15—C16—C11	−179.8 (2)	C7—C6—N2—C1	−155.5 (2)
C14—C15—C16—C11	0.4 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.86	2.07	2.907 (3)	164

C3—H3*B*···O1^{ii}	0.96	2.60	3.393 (4)	140

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