Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(Dibutylamino)-3-(4-fluorophenyl)-5,6,7,8-tetrahydro-7-methyl-6,8-diphenylpyridine[3′,4′:2,3]thieno[5,4-*d*]pyrimidin-4(3*H*)-one

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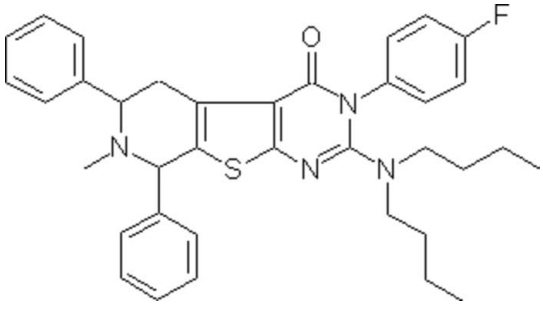
Received 15 January 2008; accepted 25 January 2008

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.076; wR factor = 0.178; data-to-parameter ratio = 15.1.

In the crystal structure of the title compound, $C_{36}H_{39}FN_4OS$, the two fused rings of the thienopyrimidine system are coplanar. The 4-fluorophenyl ring is twisted with respect to the heterocyclic pyrimidinone ring by 67.21 (14)°. The piperidine ring shows a half-chair conformation. One of the *n*-butyl chains is disordered equally over two sites. The crystal packing is stabilized by C—H···O hydrogen bonds.

Related literature

The preparation and biological activity are described by Walter (1999*a*,*b*). For related literature, see: Ding *et al.* (2004). For the crystal structures of other fused pyrimidinone derivatives, see: Hu *et al.* (2006, 2007).



Experimental

Crystal data

$C_{36}H_{39}FN_4OS$
M_r = 594.77
Monoclinic, $P2_1/c$
a = 13.723 (4) Å
b = 9.836 (3) Å
c = 24.5496 (15) Å
β = 101.342 (2)°
V = 3249.0 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 294 (2) K
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.973, T_{max} = 0.986
32225 measured reflections
6359 independent reflections
4457 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.178
S = 1.10
6359 reflections
420 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^{i}	0.93	2.47	3.304 (6)	149

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors gratefully acknowledge financial support of this work by the Key Science Research Project of Hubei Provincial Department of Education (grant No. D200724001) and the Science Research Project of Yunyang Medical College (grant No. 2006QDJ16).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2671).

References

Bruker (2001). *SMART* (Version 5.628), *SAINT-Plus* (Version 6.45) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Ding, M. W., Xu, S. Z. & Zhao, J. F. (2004). *J. Org. Chem.* **69**, 8366–8371.
Hu, Y.-G., Li, G.-H. & Zhou, M.-H. (2007). *Acta Cryst.* E**63**, o1836–o1838.
Hu, Y.-G., Zheng, A.-H. & Li, G.-H. (2006). *Acta Cryst.* E**62**, o1457–o1459.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Walter, H. (1999*a*). PCT Int. Appl. No. 44.
Walter, H. (1999*b*). PCT Int. Appl. No. 89.

supporting information

Acta Cryst. (2008). E**64**, o535 [doi:10.1107/S1600536808002845]

2-(Dibutylamino)-3-(4-fluorophenyl)-5,6,7,8-tetrahydro-7-methyl-6,8-diphenylpyridine[3′,4′:2,3]thieno[5,4-*d*]pyrimidin-4(3*H*)-one

Guo-ping Zeng, Qing Li and Yang-gen Hu

S1. Comment

The derivatives of heterocycles containing the thienopyrimidine system, which are well known bioisosteres of quinazolines, are of great importance because of their remarkable biological properties (Walter, 1999*a*; Walter, 1999*b*; Ding *et al.*, 2004). Recently, we have focused on the synthesis of the fused heterocycle systems containing thienopyrimidine *via* aza-Wittig reaction at room temperature. Some X-ray crystal structures of fused pyrimidinone derivatives have been reported (Hu *et al.*, 2006; 2007). The title compound (Fig. 1) may be used as a precursor for obtaining bioactive molecules. The two fused rings of the thienopyrimidine ring system are coplanar, making a dihedral angle of 0.36 (13)°. The *p*-fluorophenyl ring is twisted with respect to pyrimidinone ring by 67.21 (14)°. The piperidine ring shows a half-chair conformation [φ =25.9 (4)° and θ = 49.9 (3)°, puckering Amplitude = 0.515 (3) Å]. One of the *n*-butyl chains is disordered over two sites. The crystal packing is stabilized by C—H···O hydrogen bonds interactions (Table 1).

S2. Experimental

To a solution of ethyl 2-((*p*-flurophenylimino)methyleneamino)-4,5,6,7-tetrahydro-6-methyl- 5,7-diphenylthieno[2,3-*c*]pyridine-3-carboxylate (3 mmol) in dichloromethane (5 ml) was added dibutylamine (3 mmol). After stirring the reaction mixture for 1 h, the solvent was removed and anhydrous ethanol (10 ml) with several drops of EtONa in EtOH was added. The mixture was stirred for 4 h at room temperature. The solution was concentrated under reduced pressure and the residue was recrystallized from dichloromethane and ethanol (*v*/*v* = 1:1) to give the title compound in a yield of 80%. Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from a mixed solvent of ethanol and dichloromethane (1:3 *v*/*v*) at room temperature.

S3. Refinement

All H-atoms were positioned with idealized geometry and refined using a riding model with U_{iso}(H)= 1.5U_{eq}(C) for methyl H atoms and U_{iso}(H) =1.2U_{eq}(C) for all other H atoms and with C—H ranging from 0.93° to 0.98 Å. The methyl groups were allowed to rotate but not to tip. Three atoms of one *n*-butyl chains are disordered over two sites with site occupation factors of 0.540 (12) and 0.460 (12).

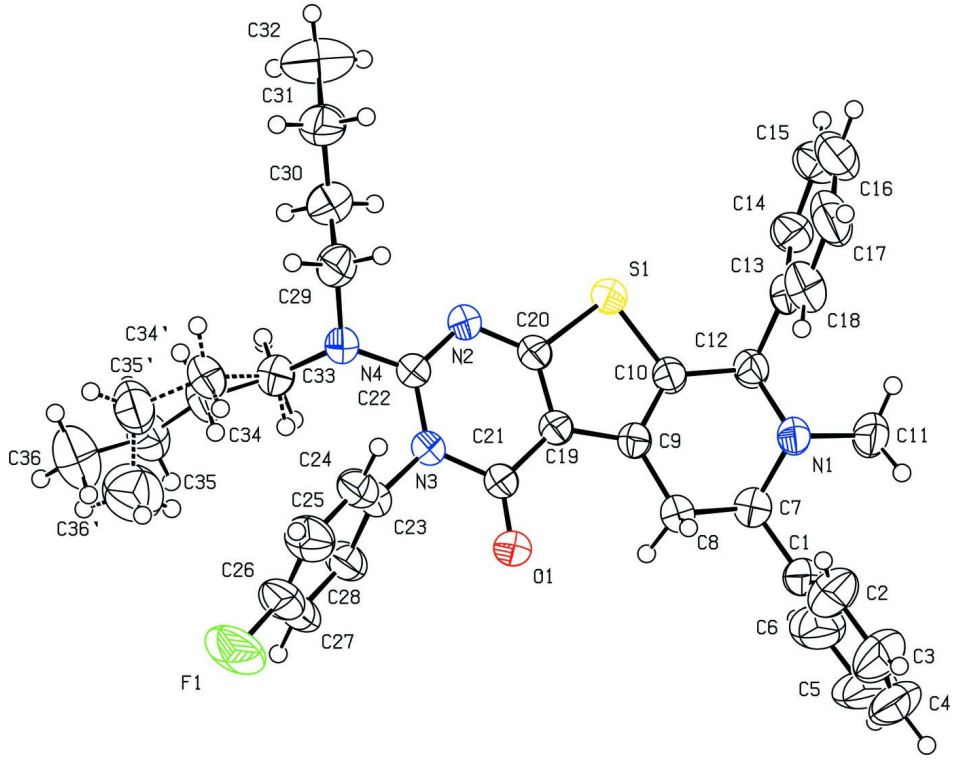


Figure 1

Molecular structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level

2-(Dibutylamino)-3-(4-fluorophenyl)-5,6,7,8-tetrahydro-7-methyl-6,8- diphenylpyridine[3′,4′:2,3]thieno[5,4-*d*]pyrimidin-4(3*H*)-one

Crystal data

$C_{36}H_{39}FN_4OS$
$M_r = 594.77$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.723\ (4)$ Å
$b = 9.836\ (3)$ Å
$c = 24.5496\ (15)$ Å
$\beta = 101.342\ (2)°$
$V = 3249.0\ (14)$ Å^3
$Z = 4$
$F(000) = 1264$
$D_x = 1.216$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4792 reflections
$\theta = 2.2–21.1°$
$\mu = 0.14$ mm^{-1}
$T = 294$ K
Block, colorless
0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.973$, $T_{max} = 0.986$
32225 measured reflections
6359 independent reflections
4457 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.0°$
$h = -16\rightarrow16$
$k = -12\rightarrow12$
$l = -29\rightarrow30$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.178$
$S = 1.10$
6359 reflections
420 parameters
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
$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 1.2986P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.3447 (2)	0.2687 (3)	0.22128 (13)	0.0579 (8)	
C2	0.3770 (3)	0.3694 (5)	0.19044 (17)	0.0870 (12)	
H2	0.3466	0.4543	0.1877	0.104*	
C3	0.4545 (4)	0.3456 (7)	0.1635 (2)	0.1172 (18)	
H3	0.4759	0.4152	0.1431	0.141*	
C4	0.4996 (4)	0.2237 (9)	0.1661 (3)	0.136 (3)	
H4	0.5511	0.2084	0.1472	0.163*	
C5	0.4692 (4)	0.1243 (7)	0.1963 (3)	0.131 (2)	
H5	0.5001	0.0399	0.1982	0.158*	
C6	0.3933 (3)	0.1448 (4)	0.22453 (19)	0.0905 (13)	
H6	0.3745	0.0750	0.2459	0.109*	
C7	0.2588 (2)	0.2901 (3)	0.25038 (12)	0.0510 (7)	
H7	0.2445	0.2034	0.2668	0.061*	
C8	0.2858 (2)	0.3941 (3)	0.29718 (11)	0.0510 (7)	
H8A	0.3355	0.3555	0.3267	0.061*	
H8B	0.3143	0.4739	0.2832	0.061*	
C9	0.19730 (18)	0.4348 (3)	0.31993 (10)	0.0411 (6)	
C10	0.10476 (18)	0.4041 (3)	0.29181 (11)	0.0427 (6)	
C11	0.1498 (2)	0.2452 (4)	0.16164 (14)	0.0767 (11)	
H11A	0.1524	0.1519	0.1733	0.115*	
H11B	0.1998	0.2612	0.1400	0.115*	
H11C	0.0854	0.2645	0.1396	0.115*	
C12	0.08097 (19)	0.3292 (3)	0.23755 (11)	0.0474 (7)	
H12	0.0669	0.2341	0.2450	0.057*	
C13	−0.0112 (2)	0.3913 (3)	0.20114 (11)	0.0503 (7)	

C14	−0.1018 (2)	0.3286 (4)	0.19700 (14)	0.0644 (9)	
H14	−0.1059	0.2453	0.2143	0.077*	
C15	−0.1881 (3)	0.3895 (5)	0.16690 (18)	0.0912 (13)	
H15	−0.2494	0.3472	0.1643	0.109*	
C16	−0.1813 (3)	0.5119 (5)	0.14142 (17)	0.0936 (14)	
H16	−0.2383	0.5533	0.1217	0.112*	
C17	−0.0902 (3)	0.5741 (4)	0.14485 (14)	0.0851 (12)	
H17	−0.0860	0.6567	0.1270	0.102*	
C18	−0.0060 (3)	0.5145 (4)	0.17451 (13)	0.0715 (10)	
H18	0.0552	0.5571	0.1768	0.086*	
C19	0.19517 (18)	0.5079 (3)	0.37045 (10)	0.0398 (6)	
C20	0.10039 (18)	0.5317 (3)	0.37838 (10)	0.0421 (6)	
C21	0.27613 (19)	0.5607 (3)	0.41048 (11)	0.0416 (6)	
C22	0.14679 (18)	0.6441 (3)	0.45949 (11)	0.0438 (7)	
C23	0.32616 (19)	0.6976 (3)	0.49318 (11)	0.0434 (6)	
C24	0.3317 (2)	0.8363 (3)	0.49281 (12)	0.0539 (8)	
H24	0.2826	0.8865	0.4699	0.065*	
C25	0.4101 (2)	0.9018 (4)	0.52640 (15)	0.0714 (10)	
H25	0.4140	0.9962	0.5271	0.086*	
C26	0.4817 (2)	0.8244 (4)	0.55861 (15)	0.0740 (10)	
C27	0.4788 (2)	0.6871 (4)	0.55940 (14)	0.0716 (10)	
H27	0.5289	0.6376	0.5819	0.086*	
C28	0.3998 (2)	0.6217 (3)	0.52609 (12)	0.0581 (8)	
H28	0.3962	0.5273	0.5258	0.070*	
C29	0.0333 (2)	0.7957 (3)	0.49500 (14)	0.0594 (8)	
H29A	0.0426	0.8720	0.5205	0.071*	
H29B	0.0223	0.8328	0.4577	0.071*	
C30	−0.0583 (2)	0.7227 (4)	0.50169 (15)	0.0665 (9)	
H30A	−0.0484	0.6841	0.5387	0.080*	
H30B	−0.0704	0.6484	0.4752	0.080*	
C31	−0.1489 (2)	0.8153 (4)	0.49285 (15)	0.0700 (10)	
H31A	−0.1369	0.8882	0.5199	0.084*	
H31B	−0.1571	0.8559	0.4562	0.084*	
C32	−0.2429 (3)	0.7447 (5)	0.4977 (2)	0.1140 (17)	
H32A	−0.2572	0.6750	0.4699	0.171*	
H32B	−0.2965	0.8092	0.4925	0.171*	
H32C	−0.2357	0.7043	0.5339	0.171*	
C33	0.1645 (2)	0.6617 (4)	0.56025 (12)	0.0669 (9)	
H33A	0.1087	0.6153	0.5709	0.080*	0.540 (12)
H33B	0.2126	0.5920	0.5563	0.080*	0.540 (12)
H33C	0.2243	0.6084	0.5614	0.080*	0.460 (12)
H33D	0.1146	0.6048	0.5719	0.080*	0.460 (12)
C34	0.2095 (12)	0.7431 (12)	0.6075 (4)	0.066 (3)	0.540 (12)
H34A	0.1596	0.8048	0.6163	0.079*	0.540 (12)
H34B	0.2616	0.7980	0.5971	0.079*	0.540 (12)
C35	0.2531 (8)	0.6640 (10)	0.6590 (3)	0.068 (3)	0.540 (12)
H35A	0.2014	0.6101	0.6704	0.082*	0.540 (12)
H35B	0.3035	0.6023	0.6509	0.082*	0.540 (12)

C36	0.2984 (9)	0.7568 (10)	0.7053 (4)	0.104 (4)	0.540 (12)
H36A	0.3532	0.8046	0.6952	0.156*	0.540 (12)
H36B	0.3216	0.7044	0.7383	0.156*	0.540 (12)
H36C	0.2494	0.8210	0.7121	0.156*	0.540 (12)
C35′	0.2274 (7)	0.7482 (16)	0.6599 (3)	0.084 (4)	0.460 (12)
H35C	0.2191	0.8236	0.6840	0.101*	0.460 (12)
H35D	0.1883	0.6727	0.6692	0.101*	0.460 (12)
C36′	0.3346 (8)	0.7077 (14)	0.6708 (7)	0.118 (5)	0.460 (12)
H36D	0.3423	0.6263	0.6505	0.177*	0.460 (12)
H36E	0.3570	0.6914	0.7098	0.177*	0.460 (12)
H36F	0.3733	0.7794	0.6591	0.177*	0.460 (12)
C34′	0.1879 (13)	0.7894 (15)	0.6001 (5)	0.073 (4)	0.460 (12)
H34C	0.2365	0.8465	0.5873	0.088*	0.460 (12)
H34D	0.1277	0.8424	0.5982	0.088*	0.460 (12)
F1	0.56013 (15)	0.8889 (3)	0.59104 (11)	0.1141 (9)	
N1	0.16778 (16)	0.3340 (3)	0.21066 (9)	0.0490 (6)	
N2	0.07372 (15)	0.5982 (2)	0.42205 (9)	0.0460 (6)	
N3	0.24634 (15)	0.6284 (2)	0.45583 (9)	0.0425 (5)	
N4	0.12771 (17)	0.7164 (3)	0.50414 (9)	0.0543 (6)	
O1	0.36369 (14)	0.5545 (2)	0.40787 (8)	0.0573 (6)	
S1	0.01216 (5)	0.46324 (8)	0.32512 (3)	0.0508 (2)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0445 (16)	0.074 (2)	0.0542 (19)	0.0018 (15)	0.0070 (14)	−0.0218 (17)
C2	0.079 (3)	0.107 (3)	0.085 (3)	−0.001 (2)	0.041 (2)	0.002 (2)
C3	0.087 (3)	0.182 (6)	0.097 (3)	−0.014 (4)	0.054 (3)	−0.018 (4)
C4	0.056 (3)	0.235 (8)	0.125 (5)	−0.007 (4)	0.039 (3)	−0.087 (5)
C5	0.061 (3)	0.156 (6)	0.174 (6)	0.032 (3)	0.016 (3)	−0.081 (5)
C6	0.067 (2)	0.089 (3)	0.112 (3)	0.014 (2)	0.008 (2)	−0.025 (2)
C7	0.0510 (16)	0.0564 (18)	0.0459 (17)	0.0009 (14)	0.0106 (13)	−0.0015 (14)
C8	0.0448 (15)	0.068 (2)	0.0388 (15)	0.0030 (14)	0.0046 (12)	−0.0073 (14)
C9	0.0400 (14)	0.0488 (16)	0.0347 (14)	−0.0002 (12)	0.0081 (11)	0.0020 (12)
C10	0.0399 (14)	0.0525 (17)	0.0355 (14)	−0.0058 (12)	0.0066 (11)	−0.0016 (12)
C11	0.058 (2)	0.119 (3)	0.055 (2)	−0.014 (2)	0.0159 (16)	−0.035 (2)
C12	0.0450 (15)	0.0556 (18)	0.0411 (16)	−0.0080 (13)	0.0071 (12)	−0.0064 (13)
C13	0.0488 (16)	0.067 (2)	0.0336 (15)	−0.0044 (14)	0.0038 (12)	−0.0170 (14)
C14	0.0482 (18)	0.077 (2)	0.066 (2)	−0.0068 (16)	0.0060 (15)	−0.0212 (18)
C15	0.053 (2)	0.120 (4)	0.094 (3)	−0.001 (2)	−0.002 (2)	−0.039 (3)
C16	0.084 (3)	0.113 (4)	0.068 (3)	0.033 (3)	−0.023 (2)	−0.029 (3)
C17	0.102 (3)	0.091 (3)	0.051 (2)	0.012 (2)	−0.012 (2)	−0.0048 (19)
C18	0.080 (2)	0.080 (3)	0.0490 (19)	−0.0041 (19)	−0.0023 (17)	0.0017 (18)
C19	0.0375 (14)	0.0448 (15)	0.0364 (14)	−0.0001 (11)	0.0058 (11)	0.0009 (12)
C20	0.0377 (14)	0.0516 (17)	0.0358 (14)	0.0007 (12)	0.0042 (11)	0.0012 (12)
C21	0.0390 (15)	0.0494 (17)	0.0355 (14)	0.0040 (12)	0.0051 (11)	0.0017 (12)
C22	0.0379 (14)	0.0559 (17)	0.0366 (15)	0.0056 (12)	0.0051 (11)	−0.0015 (13)
C23	0.0402 (14)	0.0524 (18)	0.0357 (14)	0.0009 (12)	0.0029 (11)	−0.0049 (12)

C24	0.0487 (16)	0.059 (2)	0.0503 (17)	0.0029 (14)	0.0008 (13)	0.0050 (15)
C25	0.064 (2)	0.061 (2)	0.085 (3)	−0.0114 (17)	0.0048 (18)	−0.0115 (19)
C26	0.0468 (18)	0.093 (3)	0.075 (2)	−0.0105 (18)	−0.0055 (16)	−0.026 (2)
C27	0.0550 (19)	0.083 (3)	0.065 (2)	0.0176 (17)	−0.0177 (16)	−0.0154 (19)
C28	0.0512 (17)	0.0601 (19)	0.0563 (19)	0.0085 (15)	−0.0060 (14)	−0.0049 (15)
C29	0.0628 (19)	0.058 (2)	0.0566 (19)	0.0073 (16)	0.0109 (15)	−0.0101 (16)
C30	0.062 (2)	0.069 (2)	0.071 (2)	0.0080 (17)	0.0174 (17)	−0.0034 (18)
C31	0.061 (2)	0.080 (2)	0.068 (2)	0.0180 (18)	0.0116 (17)	0.0058 (19)
C32	0.068 (3)	0.108 (4)	0.170 (5)	0.010 (2)	0.032 (3)	0.019 (3)
C33	0.0541 (18)	0.104 (3)	0.0436 (18)	0.0043 (18)	0.0125 (14)	−0.0012 (18)
C34	0.075 (8)	0.078 (6)	0.038 (5)	0.001 (5)	−0.003 (4)	0.002 (4)
C35	0.071 (6)	0.074 (6)	0.057 (4)	−0.016 (4)	0.006 (4)	−0.001 (4)
C36	0.123 (8)	0.112 (8)	0.068 (6)	−0.016 (6)	−0.004 (5)	−0.002 (5)
C35′	0.084 (7)	0.113 (11)	0.053 (6)	−0.016 (7)	0.005 (5)	−0.010 (6)
C36′	0.113 (10)	0.119 (10)	0.115 (12)	0.018 (8)	0.005 (8)	0.003 (8)
C34′	0.060 (7)	0.113 (11)	0.042 (6)	0.007 (7)	−0.003 (4)	−0.001 (7)
F1	0.0640 (13)	0.128 (2)	0.132 (2)	−0.0187 (13)	−0.0241 (13)	−0.0511 (17)
N1	0.0426 (13)	0.0682 (16)	0.0365 (12)	−0.0072 (11)	0.0083 (10)	−0.0095 (11)
N2	0.0389 (12)	0.0603 (15)	0.0380 (13)	0.0010 (11)	0.0055 (10)	−0.0082 (11)
N3	0.0376 (12)	0.0517 (14)	0.0365 (12)	0.0029 (10)	0.0028 (9)	−0.0032 (10)
N4	0.0461 (13)	0.0767 (18)	0.0388 (13)	0.0114 (12)	0.0047 (10)	−0.0105 (12)
O1	0.0381 (11)	0.0840 (15)	0.0489 (12)	0.0018 (10)	0.0061 (8)	−0.0122 (11)
S1	0.0361 (4)	0.0717 (5)	0.0441 (4)	−0.0058 (3)	0.0064 (3)	−0.0110 (4)

Geometric parameters (Å, º)

C1—C2	1.373 (5)	C23—C28	1.382 (4)
C1—C6	1.385 (5)	C23—N3	1.452 (3)
C1—C7	1.509 (4)	C24—C25	1.380 (4)
C2—C3	1.378 (6)	C24—H24	0.9300
C2—H2	0.9300	C25—C26	1.366 (5)
C3—C4	1.345 (8)	C25—H25	0.9300
C3—H3	0.9300	C26—C27	1.352 (5)
C4—C5	1.343 (8)	C26—F1	1.363 (3)
C4—H4	0.9300	C27—C28	1.381 (4)
C5—C6	1.374 (7)	C27—H27	0.9300
C5—H5	0.9300	C28—H28	0.9300
C6—H6	0.9300	C29—C30	1.485 (4)
C7—N1	1.489 (3)	C29—N4	1.491 (4)
C7—C8	1.528 (4)	C29—H29A	0.9700
C7—H7	0.9800	C29—H29B	0.9700
C8—C9	1.488 (4)	C30—C31	1.521 (4)
C8—H8A	0.9700	C30—H30A	0.9700
C8—H8B	0.9700	C30—H30B	0.9700
C9—C10	1.355 (3)	C31—C32	1.491 (5)
C9—C19	1.439 (4)	C31—H31A	0.9700
C10—C12	1.501 (4)	C31—H31B	0.9700
C10—S1	1.741 (3)	C32—H32A	0.9600

C11—N1	1.468 (4)	C32—H32B	0.9600
C11—H11A	0.9600	C32—H32C	0.9600
C11—H11B	0.9600	C33—C34	1.444 (9)
C11—H11C	0.9600	C33—N4	1.473 (4)
C12—N1	1.472 (3)	C33—C34′	1.585 (12)
C12—C13	1.525 (4)	C33—H33A	0.9700
C12—H12	0.9800	C33—H33B	0.9700
C13—C14	1.374 (4)	C33—H33C	0.9700
C13—C18	1.385 (5)	C33—H33D	0.9700
C14—C15	1.401 (5)	C34—C35	1.506 (10)
C14—H14	0.9300	C34—H34A	0.9700
C15—C16	1.368 (6)	C34—H34B	0.9700
C15—H15	0.9300	C35—C36	1.494 (10)
C16—C17	1.379 (6)	C35—H35A	0.9700
C16—H16	0.9300	C35—H35B	0.9700
C17—C18	1.371 (5)	C36—H36A	0.9600
C17—H17	0.9300	C36—H36B	0.9600
C18—H18	0.9300	C36—H36C	0.9600
C19—C20	1.373 (3)	C35′—C36′	1.496 (12)
C19—C21	1.428 (3)	C35′—C34′	1.517 (11)
C20—N2	1.366 (3)	C35′—H35C	0.9700
C20—S1	1.734 (3)	C35′—H35D	0.9700
C21—O1	1.217 (3)	C36′—H36D	0.9600
C21—N3	1.424 (3)	C36′—H36E	0.9600
C22—N2	1.301 (3)	C36′—H36F	0.9600
C22—N4	1.375 (3)	C34′—H34C	0.9700
C22—N3	1.395 (3)	C34′—H34D	0.9700
C23—C24	1.366 (4)		

C2—C1—C6	117.6 (4)	C27—C26—F1	118.6 (3)
C2—C1—C7	121.9 (3)	C27—C26—C25	123.0 (3)
C6—C1—C7	120.5 (3)	F1—C26—C25	118.3 (4)
C1—C2—C3	120.4 (5)	C26—C27—C28	118.6 (3)
C1—C2—H2	119.8	C26—C27—H27	120.7
C3—C2—H2	119.8	C28—C27—H27	120.7
C4—C3—C2	121.4 (6)	C27—C28—C23	119.6 (3)
C4—C3—H3	119.3	C27—C28—H28	120.2
C2—C3—H3	119.3	C23—C28—H28	120.2
C5—C4—C3	118.9 (5)	C30—C29—N4	117.4 (3)
C5—C4—H4	120.5	C30—C29—H29A	108.0
C3—C4—H4	120.5	N4—C29—H29A	108.0
C4—C5—C6	121.4 (6)	C30—C29—H29B	108.0
C4—C5—H5	119.3	N4—C29—H29B	108.0
C6—C5—H5	119.3	H29A—C29—H29B	107.2
C5—C6—C1	120.3 (5)	C29—C30—C31	112.4 (3)
C5—C6—H6	119.9	C29—C30—H30A	109.1
C1—C6—H6	119.9	C31—C30—H30A	109.1
N1—C7—C1	111.3 (2)	C29—C30—H30B	109.1

N1—C7—C8	110.3 (2)	C31—C30—H30B	109.1
C1—C7—C8	110.6 (2)	H30A—C30—H30B	107.9
N1—C7—H7	108.2	C32—C31—C30	113.9 (3)
C1—C7—H7	108.2	C32—C31—H31A	108.8
C8—C7—H7	108.2	C30—C31—H31A	108.8
C9—C8—C7	111.5 (2)	C32—C31—H31B	108.8
C9—C8—H8A	109.3	C30—C31—H31B	108.8
C7—C8—H8A	109.3	H31A—C31—H31B	107.7
C9—C8—H8B	109.3	C31—C32—H32A	109.5
C7—C8—H8B	109.3	C31—C32—H32B	109.5
H8A—C8—H8B	108.0	H32A—C32—H32B	109.5
C10—C9—C19	112.0 (2)	C31—C32—H32C	109.5
C10—C9—C8	120.1 (2)	H32A—C32—H32C	109.5
C19—C9—C8	127.9 (2)	H32B—C32—H32C	109.5
C9—C10—C12	125.4 (2)	C34—C33—N4	124.2 (6)
C9—C10—S1	112.6 (2)	N4—C33—C34′	106.1 (6)
C12—C10—S1	121.97 (19)	C34—C33—H33A	106.3
N1—C11—H11A	109.5	N4—C33—H33A	106.3
N1—C11—H11B	109.5	C34—C33—H33B	106.3
H11A—C11—H11B	109.5	N4—C33—H33B	106.3
N1—C11—H11C	109.5	H33A—C33—H33B	106.4
H11A—C11—H11C	109.5	N4—C33—H33C	110.9
H11B—C11—H11C	109.5	C34′—C33—H33C	110.1
N1—C12—C10	109.2 (2)	N4—C33—H33D	110.8
N1—C12—C13	112.0 (2)	C34′—C33—H33D	110.2
C10—C12—C13	109.5 (2)	H33C—C33—H33D	108.8
N1—C12—H12	108.7	C33—C34—C35	115.1 (8)
C10—C12—H12	108.7	C33—C34—H34A	108.5
C13—C12—H12	108.7	C35—C34—H34A	108.5
C14—C13—C18	119.1 (3)	C33—C34—H34B	108.5
C14—C13—C12	119.9 (3)	C35—C34—H34B	108.5
C18—C13—C12	120.9 (3)	H34A—C34—H34B	107.5
C13—C14—C15	120.4 (4)	C36—C35—C34	111.1 (7)
C13—C14—H14	119.8	C36—C35—H35A	109.4
C15—C14—H14	119.8	C34—C35—H35A	109.4
C16—C15—C14	119.3 (4)	C36—C35—H35B	109.4
C16—C15—H15	120.3	C34—C35—H35B	109.4
C14—C15—H15	120.3	H35A—C35—H35B	108.0
C15—C16—C17	120.4 (4)	C36′—C35′—C34′	113.4 (10)
C15—C16—H16	119.8	C36′—C35′—H35C	108.9
C17—C16—H16	119.8	C34′—C35′—H35C	108.9
C18—C17—C16	120.1 (4)	C36′—C35′—H35D	108.9
C18—C17—H17	120.0	C34′—C35′—H35D	108.9
C16—C17—H17	120.0	H35C—C35′—H35D	107.7
C17—C18—C13	120.6 (4)	C35′—C36′—H36D	109.5
C17—C18—H18	119.7	C35′—C36′—H36E	109.5
C13—C18—H18	119.7	H36D—C36′—H36E	109.5
C20—C19—C21	118.1 (2)	C35′—C36′—H36F	109.5

C20—C19—C9	112.8 (2)	H36D—C36′—H36F	109.5
C21—C19—C9	129.0 (2)	H36E—C36′—H36F	109.5
N2—C20—C19	126.9 (2)	C35′—C34′—C33	112.1 (9)
N2—C20—S1	121.52 (19)	C35′—C34′—H34C	109.2
C19—C20—S1	111.6 (2)	C33—C34′—H34C	109.2
O1—C21—N3	120.2 (2)	C35′—C34′—H34D	109.2
O1—C21—C19	126.0 (2)	C33—C34′—H34D	109.2
N3—C21—C19	113.8 (2)	H34C—C34′—H34D	107.9
N2—C22—N4	120.1 (2)	C11—N1—C12	109.2 (2)
N2—C22—N3	123.0 (2)	C11—N1—C7	110.4 (2)
N4—C22—N3	116.8 (2)	C12—N1—C7	110.2 (2)
C24—C23—C28	120.5 (3)	C22—N2—C20	115.6 (2)
C24—C23—N3	120.0 (2)	C22—N3—C21	122.5 (2)
C28—C23—N3	119.4 (3)	C22—N3—C23	122.3 (2)
C23—C24—C25	120.0 (3)	C21—N3—C23	114.6 (2)
C23—C24—H24	120.0	C22—N4—C33	118.2 (2)
C25—C24—H24	120.0	C22—N4—C29	116.5 (2)
C26—C25—C24	118.3 (3)	C33—N4—C29	117.4 (2)
C26—C25—H25	120.9	C20—S1—C10	91.03 (12)
C24—C25—H25	120.9		

C6—C1—C2—C3	0.7 (6)	C24—C25—C26—F1	178.8 (3)
C7—C1—C2—C3	−178.7 (4)	F1—C26—C27—C28	−179.4 (3)
C1—C2—C3—C4	0.7 (7)	C25—C26—C27—C28	0.1 (6)
C2—C3—C4—C5	−1.0 (9)	C26—C27—C28—C23	−0.2 (5)
C3—C4—C5—C6	0.0 (9)	C24—C23—C28—C27	0.9 (5)
C4—C5—C6—C1	1.5 (8)	N3—C23—C28—C27	176.4 (3)
C2—C1—C6—C5	−1.8 (5)	N4—C29—C30—C31	178.4 (3)
C7—C1—C6—C5	177.7 (4)	C29—C30—C31—C32	178.4 (4)
C2—C1—C7—N1	56.1 (4)	N4—C33—C34—C35	−172.7 (8)
C6—C1—C7—N1	−123.3 (3)	C34′—C33—C34—C35	157 (5)
C2—C1—C7—C8	−66.9 (4)	C33—C34—C35—C36	179.6 (12)
C6—C1—C7—C8	113.7 (3)	C36′—C35′—C34′—C33	78.0 (18)
N1—C7—C8—C9	46.5 (3)	C34—C33—C34′—C35′	−25 (3)
C1—C7—C8—C9	170.1 (3)	N4—C33—C34′—C35′	−179.7 (10)
C7—C8—C9—C10	−14.1 (4)	C10—C12—N1—C11	172.2 (3)
C7—C8—C9—C19	166.6 (3)	C13—C12—N1—C11	−66.3 (3)
C19—C9—C10—C12	179.6 (2)	C10—C12—N1—C7	50.8 (3)
C8—C9—C10—C12	0.3 (4)	C13—C12—N1—C7	172.3 (2)
C19—C9—C10—S1	0.3 (3)	C1—C7—N1—C11	48.7 (3)
C8—C9—C10—S1	−179.0 (2)	C8—C7—N1—C11	171.9 (3)
C9—C10—C12—N1	−18.6 (4)	C1—C7—N1—C12	169.4 (2)
S1—C10—C12—N1	160.7 (2)	C8—C7—N1—C12	−67.5 (3)
C9—C10—C12—C13	−141.6 (3)	N4—C22—N2—C20	177.8 (2)
S1—C10—C12—C13	37.6 (3)	N3—C22—N2—C20	0.5 (4)
N1—C12—C13—C14	137.7 (3)	C19—C20—N2—C22	0.6 (4)
C10—C12—C13—C14	−100.9 (3)	S1—C20—N2—C22	179.5 (2)
N1—C12—C13—C18	−45.9 (4)	N2—C22—N3—C21	0.1 (4)

C10—C12—C13—C18	75.4 (3)	N4—C22—N3—C21	−177.3 (2)
C18—C13—C14—C15	−1.0 (5)	N2—C22—N3—C23	170.3 (3)
C12—C13—C14—C15	175.4 (3)	N4—C22—N3—C23	−7.1 (4)
C13—C14—C15—C16	0.3 (5)	O1—C21—N3—C22	176.8 (3)
C14—C15—C16—C17	0.6 (6)	C19—C21—N3—C22	−1.6 (4)
C15—C16—C17—C18	−0.9 (6)	O1—C21—N3—C23	5.9 (4)
C16—C17—C18—C13	0.2 (5)	C19—C21—N3—C23	−172.5 (2)
C14—C13—C18—C17	0.7 (5)	C24—C23—N3—C22	−64.1 (4)
C12—C13—C18—C17	−175.6 (3)	C28—C23—N3—C22	120.4 (3)
C10—C9—C19—C20	−0.8 (3)	C24—C23—N3—C21	106.9 (3)
C8—C9—C19—C20	178.5 (3)	C28—C23—N3—C21	−68.6 (3)
C10—C9—C19—C21	−178.4 (3)	N2—C22—N4—C33	119.2 (3)
C8—C9—C19—C21	0.9 (5)	N3—C22—N4—C33	−63.3 (4)
C21—C19—C20—N2	−2.2 (4)	N2—C22—N4—C29	−29.2 (4)
C9—C19—C20—N2	180.0 (3)	N3—C22—N4—C29	148.3 (3)
C21—C19—C20—S1	178.8 (2)	C34—C33—N4—C22	138.2 (9)
C9—C19—C20—S1	1.0 (3)	C34′—C33—N4—C22	148.7 (8)
C20—C19—C21—O1	−175.9 (3)	C34—C33—N4—C29	−73.7 (9)
C9—C19—C21—O1	1.5 (5)	C34′—C33—N4—C29	−63.2 (8)
C20—C19—C21—N3	2.5 (4)	C30—C29—N4—C22	85.0 (4)
C9—C19—C21—N3	179.9 (2)	C30—C29—N4—C33	−63.7 (4)
C28—C23—C24—C25	−1.5 (5)	N2—C20—S1—C10	−179.7 (2)
N3—C23—C24—C25	−177.0 (3)	C19—C20—S1—C10	−0.7 (2)
C23—C24—C25—C26	1.3 (5)	C9—C10—S1—C20	0.2 (2)
C24—C25—C26—C27	−0.6 (6)	C12—C10—S1—C20	−179.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.93	2.47	3.304 (6)	149

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+1/2.