Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Isopropyl-2-morpholino-5,6,7,8-tetrahydrobenzothieno[2,3-*d*]pyrimidin-4(3*H*)-one

Xiao-Hua Zeng, Lan-Hua Zhao, Hong Luo and Ji-Yin Long*

Department of Medicinal Chemistry, Yunyang Medical College, Shiyan 442000, People's Republic of China
Correspondence e-mail: long_jiyin@yahoo.com.cn

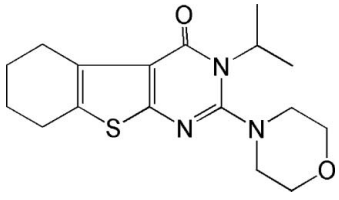
Received 2 April 2007; accepted 8 May 2007

Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.051; wR factor = 0.143; data-to-parameter ratio = 16.9.

In the title compound, $C_{17}H_{23}N_3O_2S$, the central thienopyrimidine ring system is essentially planar. The cyclohexene ring, in which the four CH_2 groups are disordered in a 3:1 ratio, adopts a half-chair conformation and the morpholine ring is in a standard chair conformation. The molecular structure is stabilized by intramolecular C—H···O and C—H···N hydrogen bonds. In the crystal packing, intermolecular C—H···O hydrogen-bonding interactions link the molecules into chains along the *b* axis.

Related literature

For related literature, see: Ding *et al.* (2004); Zeng *et al.* (2006).



Experimental

Crystal data

$C_{17}H_{23}N_3O_2S$
M_r = 333.44
Monoclinic, $C2/c$
a = 25.8379 (12) Å
b = 8.0874 (6) Å
c = 20.5316 (10) Å
β = 128.8570 (10)°
V = 3340.9 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 297 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
9964 measured reflections
3637 independent reflections
2623 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 0.99
3637 reflections
215 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13*C*···O1	0.96	2.52	3.079 (3)	117
C12—H12*A*···O1	0.96	2.32	2.882 (2)	117
C11—H11···N3	0.98	2.28	2.802 (2)	112
C14—H14*B*···O1^{i}	0.97	2.47	3.246 (2)	137
C12—H12*C*···O1^{i}	0.96	2.53	3.483 (3)	170

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

The authors gratefully acknowledge financial support of this work by the Natural Science Foundation of Hubei Province (grant No. 2006ABB016) and the School Science Foundation of Yunyang Medical College.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2136).

References

Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ding, M. W., Xu, S. Z. & Zhao, J. F. (2004). *J. Org. Chem.* **69**, 8366–8371.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zeng, X.-H., Wang, H.-M., Cui, Z.-P., Ding, M.-W. & He, H.-W. (2006). *Acta Cryst.* E**62**, o228–o229.

  doi:10.1107/S1600536807022672

supplementary materials

Acta Cryst. (2007). E**63**, o3004 [**doi:10.1107/S1600536807022672**]

3-Isopropyl-2-morpholino-5,6,7,8-tetrahydrobenzothieno[2,3-*d*]pyrimidin-4(3*H*)-one

X.-H. Zeng, L.-H. Zhao, H. Luo and J.-Y. Long

Comment

Pyrimidine derivatives are attracting the increasing attention of the synthetic community because of the important role played by such systems in many natural products, antibiotics and drugs (Ding *et al.*, 2004). In recent years, we have been engaged in the preparation of derivatives of heterocycles *via* the aza-Wittig reaction. The title compound, (I), was synthesized and structurally characterized in this context.

In the fused heterobicyclic ring of the molecule, bond lengths and angles (Table 1) are similar to those observed in closely related structures (Zeng *et al.*, 2006). All ring atoms in thienopyrimidine system are essentially coplanar. The cyclohexane ring adopts a half-chair conformation, while the pyrimidinone ring is in standard chair conformation.

The crystal packing is stabilized by intra- and intermolecular hydrogen bonding interactions (Table 2). Atoms C12 and C14, as hydrogen-bond donors, both link to the same acceptor atom, O1, forming chains along the *b* axis (Fig. 2).

The C3 carbon atom of the cyclohexane ring is disordered over two positions, with refined site occupancies of 0.743 (10) and 0.257 (10) for the major and minor components respectively.

Experimental

To a solution of iminophosphorane (*a*) (1.45 g, 3 mmol) in anhydrous dichloromethane (15 ml), isopropyl isocyanate (3 mmol) was added under dry N_2 at room temperature. After the reaction mixture was left unstirred for 48 h at room temperature, the solvent was removed under reduced pressure and ether/petroleum ether (1:2 *v*/*v*, 20 ml) was added to precipitate triphenylphosphine oxide. After filtration the solvent was removed to give carbodiimide (*b*), which was used directly without further purification. To the solution of carbodiimide (15 ml), morpholine (3 mmol) was added. After the mixture was stirred for 6 h, the solvent was removed and anhydrous ethanol (10 ml) containing several drops of EtONa in EtOH was added. The mixture was stirred for 12hr at room temperature. The solution was condensed and the residue was recrystallized from ethanol to give the title compound (I), in a yield of 81% (m.p. 455 K). Anal. Calcd. for $C_{17}H_{23}N_3O_2S$: C, 61.23; H, 6.95; N, 12.60. Found: C, 61.07; H,7.04; N, 12.46 Crystals suitable for single-crystal X-ray analysis were obtained on slow evaporation of a hexane/dichloromethane solution (1:3 *v*/*v*) at room temperature.

Refinement

All H atoms were located in difference Fourier maps and refined as riding, with C—H = 0.96 (CH_3) or 0.97 Å (CH_2), and with U_{iso}(H) values of 1.2U_{eq}(C), or 1.5U_{eq}(C) for the methyl groups. The C3 carbon atom of the cyclohexane ring is disordered over two positions, with refined site occupancies of 0.743 (10) and 0.257 (10) for the major and minor components respectively. The major component was refined anisotropically whereas the minor component was refined isotropically. The C—C bond lengths involving the disordered C3 atom were restrained to 1.48 (1) Å.

Figures

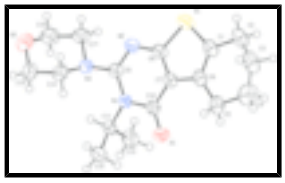


Fig. 1. A view of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at 30% probability level. H atoms are represented by circles of arbitrary size. Only the major component of the disordered C3 atom is shown.

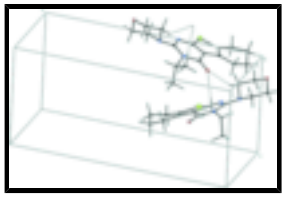


Fig. 2. A part of the crystal structure of (I), showing the formation of hydrogen bonds (dashed lines). Only the major conformation of the disordered cyclohexane ring is shown.

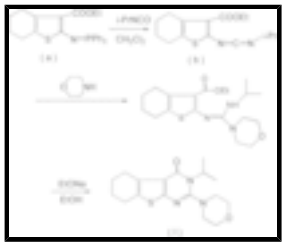


Fig. 3. The preparation of (I).

3-Isopropyl-2-morpholino-5,6,7,8-tetrahydrobenzothieno[2,3-*d*]pyrimidin-4(3*H*)-one

Crystal data

$C_{17}H_{23}N_3O_2S$	F_{000} = 1424
M_r = 333.44	D_x = 1.326 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2844 reflections
a = 25.8379 (12) Å	θ = 0.00–0.00º
b = 8.0874 (6) Å	μ = 0.21 mm^{-1}
c = 20.5316 (10) Å	*T* = 297 (2) K
β = 128.8570 (10)º	Block, colourless
V = 3340.9 (3) Å^3	0.30 × 0.20 × 0.20 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	2623 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.049
Monochromator: graphite	θ_{max} = 27.0º
T = 297(2) K	θ_{min} = 2.0º
φ and ω scans	*h* = −32→23
Absorption correction: none	*k* = −10→8
9964 measured reflections	*l* = −20→25

3637 independent reflections

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.143$	$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} < 0.001$
3637 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
215 parameters	$\Delta\rho_{min} = -0.24$ e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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)
C1	0.29131 (10)	0.5253 (3)	0.53820 (12)	0.0461 (5)	
C2	0.33982 (11)	0.5318 (3)	0.63169 (12)	0.0599 (6)	
H2A	0.3285	0.6226	0.6515	0.072*	0.743 (10)
H2B	0.3373	0.4299	0.6545	0.072*	0.743 (10)
H2C	0.3172	0.5666	0.6532	0.072*	0.257 (10)
H2D	0.3582	0.4225	0.6537	0.072*	0.257 (10)
C3	0.40839 (16)	0.5543 (7)	0.66108 (19)	0.0707 (15)	0.743 (10)
H3A	0.4360	0.5934	0.7184	0.085*	0.743 (10)
H3B	0.4254	0.4473	0.6612	0.085*	0.743 (10)
C4	0.41539 (13)	0.6684 (4)	0.61222 (16)	0.0922 (10)	
H4A	0.4607	0.6601	0.6321	0.111*	0.743 (10)
H4B	0.4097	0.7797	0.6245	0.111*	0.743 (10)
H4C	0.4520	0.5921	0.6346	0.111*	0.257 (10)
H4D	0.4339	0.7788	0.6237	0.111*	0.257 (10)
C5	0.36933 (10)	0.6484 (3)	0.51824 (12)	0.0508 (5)	
H5A	0.3623	0.7553	0.4924	0.061*	

H5B	0.3899	0.5762	0.5026	0.061*	
C6	0.30353 (9)	0.5775 (2)	0.48628 (11)	0.0400 (4)	
C7	0.24678 (9)	0.5506 (2)	0.39978 (11)	0.0357 (4)	
C8	0.19488 (9)	0.4765 (2)	0.39055 (11)	0.0402 (4)	
C9	0.12880 (9)	0.4679 (2)	0.25010 (11)	0.0357 (4)	
C10	0.23668 (9)	0.6041 (2)	0.32569 (11)	0.0363 (4)	
C11	0.15229 (9)	0.6443 (2)	0.17066 (11)	0.0379 (4)	
H11	0.1091	0.5967	0.1250	0.045*	
C12	0.19807 (11)	0.6124 (3)	0.14938 (13)	0.0533 (5)	
H12A	0.2397	0.6673	0.1894	0.080*	
H12B	0.1779	0.6540	0.0944	0.080*	
H12C	0.2055	0.4956	0.1509	0.080*	
C13	0.14058 (11)	0.8258 (2)	0.17408 (13)	0.0537 (6)	
H13A	0.1081	0.8383	0.1823	0.081*	
H13B	0.1247	0.8777	0.1225	0.081*	
H13C	0.1815	0.8769	0.2196	0.081*	
C14	0.08494 (9)	0.2799 (2)	0.13577 (12)	0.0456 (5)	
H14A	0.0984	0.1811	0.1697	0.055*	
H14B	0.1208	0.3114	0.1351	0.055*	
C15	0.02315 (10)	0.2451 (3)	0.04815 (14)	0.0559 (6)	
H15A	0.0119	0.3419	0.0136	0.067*	
H15B	0.0316	0.1545	0.0251	0.067*	
C16	−0.04365 (10)	0.3330 (3)	0.08135 (14)	0.0597 (6)	
H16A	−0.0808	0.3027	0.0796	0.072*	
H16B	−0.0557	0.4325	0.0482	0.072*	
C17	0.01619 (9)	0.3681 (3)	0.17061 (13)	0.0503 (5)	
H17A	0.0064	0.4572	0.1930	0.060*	
H17B	0.0278	0.2706	0.2048	0.060*	
C3'	0.3938 (5)	0.6511 (16)	0.6589 (6)	0.057 (3)*	0.257 (10)
H3'1	0.3794	0.7591	0.6624	0.068*	0.257 (10)
H3'2	0.4321	0.6209	0.7153	0.068*	0.257 (10)
N1	0.17404 (7)	0.56023 (17)	0.24993 (9)	0.0339 (3)	
N2	0.13614 (8)	0.4282 (2)	0.31659 (10)	0.0430 (4)	
N3	0.07162 (7)	0.41505 (18)	0.17194 (9)	0.0370 (4)	
O1	0.27571 (7)	0.68684 (18)	0.32496 (8)	0.0513 (4)	
O2	−0.03148 (8)	0.2038 (2)	0.04583 (10)	0.0647 (5)	
S1	0.21228 (3)	0.44392 (8)	0.48531 (3)	0.0554 (2)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0452 (12)	0.0542 (12)	0.0342 (10)	0.0086 (9)	0.0226 (9)	0.0028 (9)
C2	0.0643 (15)	0.0727 (15)	0.0326 (11)	0.0115 (12)	0.0255 (11)	0.0046 (10)
C3	0.053 (2)	0.099 (4)	0.0334 (16)	0.0118 (19)	0.0144 (15)	0.0000 (17)
C4	0.0549 (16)	0.131 (3)	0.0466 (14)	−0.0233 (16)	0.0105 (13)	0.0043 (16)
C5	0.0391 (11)	0.0591 (13)	0.0406 (11)	−0.0013 (9)	0.0185 (9)	−0.0060 (9)
C6	0.0381 (10)	0.0445 (10)	0.0319 (9)	0.0068 (8)	0.0193 (8)	0.0005 (8)
C7	0.0351 (10)	0.0403 (10)	0.0308 (9)	0.0037 (8)	0.0202 (8)	0.0007 (7)

C8	0.0397 (10)	0.0488 (11)	0.0337 (10)	0.0037 (8)	0.0238 (9)	0.0051 (8)
C9	0.0309 (9)	0.0392 (10)	0.0365 (10)	0.0021 (7)	0.0209 (8)	0.0026 (8)
C10	0.0335 (9)	0.0410 (10)	0.0335 (9)	−0.0007 (8)	0.0205 (8)	−0.0020 (8)
C11	0.0387 (10)	0.0445 (10)	0.0271 (9)	−0.0030 (8)	0.0190 (8)	0.0005 (7)
C12	0.0579 (13)	0.0694 (14)	0.0437 (11)	0.0005 (11)	0.0372 (11)	0.0024 (10)
C13	0.0650 (15)	0.0458 (12)	0.0430 (12)	0.0059 (10)	0.0304 (11)	0.0058 (9)
C14	0.0391 (11)	0.0442 (11)	0.0506 (12)	−0.0021 (8)	0.0267 (10)	−0.0067 (9)
C15	0.0489 (13)	0.0605 (13)	0.0537 (13)	−0.0130 (10)	0.0299 (11)	−0.0156 (10)
C16	0.0316 (11)	0.0896 (17)	0.0511 (13)	−0.0049 (11)	0.0226 (10)	0.0066 (12)
C17	0.0344 (11)	0.0707 (14)	0.0484 (12)	−0.0034 (10)	0.0272 (10)	0.0022 (10)
N1	0.0326 (8)	0.0405 (8)	0.0282 (7)	−0.0021 (6)	0.0188 (6)	0.0004 (6)
N2	0.0382 (9)	0.0552 (10)	0.0371 (9)	−0.0031 (7)	0.0243 (8)	0.0032 (7)
N3	0.0297 (8)	0.0454 (8)	0.0358 (8)	−0.0029 (7)	0.0204 (7)	−0.0027 (7)
O1	0.0419 (8)	0.0699 (10)	0.0402 (8)	−0.0162 (7)	0.0248 (7)	−0.0002 (6)
O2	0.0477 (9)	0.0755 (11)	0.0594 (10)	−0.0232 (8)	0.0281 (8)	−0.0135 (8)
S1	0.0508 (4)	0.0828 (4)	0.0367 (3)	−0.0002 (3)	0.0294 (3)	0.0086 (3)

Geometric parameters (Å, °)

C1—C6	1.355 (3)	C10—O1	1.218 (2)
C1—C2	1.496 (3)	C10—N1	1.416 (2)
C1—S1	1.732 (2)	C11—N1	1.510 (2)
C2—C3	1.481 (4)	C11—C13	1.510 (3)
C2—C3'	1.487 (7)	C11—C12	1.518 (3)
C2—H2A	0.9700	C11—H11	0.9800
C2—H2B	0.9700	C12—H12A	0.9600
C2—H2C	0.9700	C12—H12B	0.9600
C2—H2D	0.9700	C12—H12C	0.9600
C3—C4	1.455 (4)	C13—H13A	0.9600
C3—H3A	0.9700	C13—H13B	0.9600
C3—H3B	0.9700	C13—H13C	0.9600
C4—C3'	1.386 (7)	C14—N3	1.478 (2)
C4—C5	1.511 (3)	C14—C15	1.503 (3)
C4—H4A	0.9700	C14—H14A	0.9700
C4—H4B	0.9700	C14—H14B	0.9700
C4—H4C	0.9700	C15—O2	1.422 (3)
C4—H4D	0.9700	C15—H15A	0.9700
C5—C6	1.500 (3)	C15—H15B	0.9700
C5—H5A	0.9700	C16—O2	1.417 (3)
C5—H5B	0.9700	C16—C17	1.508 (3)
C6—C7	1.443 (2)	C16—H16A	0.9700
C7—C8	1.369 (3)	C16—H16B	0.9700
C7—C10	1.439 (2)	C17—N3	1.465 (2)
C8—N2	1.368 (2)	C17—H17A	0.9700
C8—S1	1.7204 (19)	C17—H17B	0.9700
C9—N2	1.295 (2)	C3'—H3'1	0.9700
C9—N1	1.389 (2)	C3'—H3'2	0.9700
C9—N3	1.402 (2)		
C6—C1—C2	125.3 (2)	N1—C9—N3	116.44 (15)

C6—C1—S1	112.96 (15)	O1—C10—N1	120.56 (16)
C2—C1—S1	121.78 (17)	O1—C10—C7	125.15 (16)
C3—C2—C1	110.6 (2)	N1—C10—C7	114.20 (15)
C3'—C2—C1	109.8 (4)	N1—C11—C13	109.73 (15)
C3—C2—H2A	109.5	N1—C11—C12	113.80 (15)
C3'—C2—H2A	79.0	C13—C11—C12	113.18 (17)
C1—C2—H2A	109.5	N1—C11—H11	106.5
C3—C2—H2B	109.5	C13—C11—H11	106.5
C3'—C2—H2B	134.5	C12—C11—H11	106.5
C1—C2—H2B	109.5	C11—C12—H12A	109.5
H2A—C2—H2B	108.1	C11—C12—H12B	109.5
C3—C2—H2C	133.4	H12A—C12—H12B	109.5
C3'—C2—H2C	109.3	C11—C12—H12C	109.5
C1—C2—H2C	109.6	H12A—C12—H12C	109.5
H2B—C2—H2C	77.5	H12B—C12—H12C	109.5
C3'—C2—H2D	110.3	C11—C13—H13A	109.5
C1—C2—H2D	109.7	C11—C13—H13B	109.5
H2A—C2—H2D	133.0	H13A—C13—H13B	109.5
H2C—C2—H2D	108.1	C11—C13—H13C	109.5
C4—C3—C2	115.6 (3)	H13A—C13—H13C	109.5
C4—C3—H3A	108.4	H13B—C13—H13C	109.5
C2—C3—H3A	108.4	N3—C14—C15	109.40 (16)
C4—C3—H3B	108.4	N3—C14—H14A	109.8
C2—C3—H3B	108.4	C15—C14—H14A	109.8
H3A—C3—H3B	107.5	N3—C14—H14B	109.8
C2—C3—H4C	145.5	C15—C14—H14B	109.8
H3A—C3—H4C	102.5	H14A—C14—H14B	108.2
H3B—C3—H4C	75.5	O2—C15—C14	111.75 (18)
C3'—C4—C5	122.4 (4)	O2—C15—H15A	109.3
C3—C4—C5	118.3 (3)	C14—C15—H15A	109.3
C3'—C4—H4A	127.2	O2—C15—H15B	109.3
C3—C4—H4A	107.7	C14—C15—H15B	109.3
C5—C4—H4A	107.7	H15A—C15—H15B	107.9
C3—C4—H4B	107.7	O2—C16—C17	112.02 (18)
C5—C4—H4B	107.7	O2—C16—H16A	109.2
H4A—C4—H4B	107.1	C17—C16—H16A	109.2
C3'—C4—H4C	107.6	O2—C16—H16B	109.2
C5—C4—H4C	106.9	C17—C16—H16B	109.2
H4B—C4—H4C	136.7	H16A—C16—H16B	107.9
C3'—C4—H4D	106.1	N3—C17—C16	108.44 (17)
C3—C4—H4D	132.3	N3—C17—H17A	110.0
C5—C4—H4D	106.4	C16—C17—H17A	110.0
H4C—C4—H4D	106.5	N3—C17—H17B	110.0
C6—C5—C4	111.6 (2)	C16—C17—H17B	110.0
C6—C5—H5A	109.3	H17A—C17—H17B	108.4
C4—C5—H5A	109.3	C4—C3'—C2	119.7 (6)
C6—C5—H5B	109.3	C4—C3'—H3'1	107.4
C4—C5—H5B	109.3	C2—C3'—H3'1	107.4
H5A—C5—H5B	108.0	C4—C3'—H3'2	107.4

C1—C6—C7	111.27 (18)	C2—C3'—H3'2	107.4
C1—C6—C5	122.30 (18)	H3'1—C3'—H3'2	106.9
C7—C6—C5	126.40 (18)	C9—N1—C10	121.04 (15)
C8—C7—C10	118.22 (16)	C9—N1—C11	120.01 (14)
C8—C7—C6	112.76 (17)	C10—N1—C11	117.85 (14)
C10—C7—C6	128.74 (17)	C9—N2—C8	115.22 (16)
N2—C8—C7	125.95 (17)	C9—N3—C17	114.59 (15)
N2—C8—S1	122.18 (14)	C9—N3—C14	112.97 (14)
C7—C8—S1	111.85 (14)	C17—N3—C14	109.38 (15)
N2—C9—N1	124.68 (16)	C16—O2—C15	110.21 (16)
N2—C9—N3	118.89 (16)	C8—S1—C1	91.12 (9)
C6—C1—C2—C3	−17.1 (4)	C3—C2—C3'—C4	63.4 (9)
S1—C1—C2—C3	161.6 (3)	C1—C2—C3'—C4	−34.4 (12)
C6—C1—C2—C3'	18.7 (6)	N2—C9—N1—C10	6.9 (3)
S1—C1—C2—C3'	−162.5 (6)	N3—C9—N1—C10	−172.92 (15)
C3'—C2—C3—C4	−55.1 (6)	N2—C9—N1—C11	−160.82 (17)
C1—C2—C3—C4	39.9 (5)	N3—C9—N1—C11	19.4 (2)
C2—C3—C4—C3'	58.0 (6)	O1—C10—N1—C9	−179.11 (16)
C2—C3—C4—C5	−49.1 (5)	C7—C10—N1—C9	−2.3 (2)
C3'—C4—C5—C6	−10.9 (8)	O1—C10—N1—C11	−11.1 (2)
C3—C4—C5—C6	29.6 (4)	C7—C10—N1—C11	165.67 (15)
C2—C1—C6—C7	178.74 (18)	C13—C11—N1—C9	101.35 (19)
S1—C1—C6—C7	−0.1 (2)	C12—C11—N1—C9	−130.68 (17)
C2—C1—C6—C5	0.4 (3)	C13—C11—N1—C10	−66.8 (2)
S1—C1—C6—C5	−178.42 (15)	C12—C11—N1—C10	61.2 (2)
C4—C5—C6—C1	−5.6 (3)	N1—C9—N2—C8	−3.3 (3)
C4—C5—C6—C7	176.3 (2)	N3—C9—N2—C8	176.48 (15)
C1—C6—C7—C8	−1.2 (2)	C7—C8—N2—C9	−4.9 (3)
C5—C6—C7—C8	177.02 (18)	S1—C8—N2—C9	176.89 (14)
C1—C6—C7—C10	172.61 (18)	N2—C9—N3—C17	19.5 (2)
C5—C6—C7—C10	−9.2 (3)	N1—C9—N3—C17	−160.67 (16)
C10—C7—C8—N2	9.0 (3)	N2—C9—N3—C14	−106.7 (2)
C6—C7—C8—N2	−176.43 (17)	N1—C9—N3—C14	73.2 (2)
C10—C7—C8—S1	−172.55 (13)	C16—C17—N3—C9	173.71 (17)
C6—C7—C8—S1	2.0 (2)	C16—C17—N3—C14	−58.3 (2)
C8—C7—C10—O1	171.77 (18)	C15—C14—N3—C9	−173.17 (16)
C6—C7—C10—O1	−1.8 (3)	C15—C14—N3—C17	57.9 (2)
C8—C7—C10—N1	−4.9 (2)	C17—C16—O2—C15	−58.6 (2)
C6—C7—C10—N1	−178.39 (16)	C14—C15—O2—C16	57.4 (2)
N3—C14—C15—O2	−57.5 (2)	N2—C8—S1—C1	176.76 (17)
O2—C16—C17—N3	59.4 (2)	C7—C8—S1—C1	−1.72 (15)
C3—C4—C3'—C2	−61.3 (8)	C6—C1—S1—C8	1.03 (16)
C5—C4—C3'—C2	32.8 (14)	C2—C1—S1—C8	−177.87 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13C···O1	0.96	2.52	3.079 (3)	117
C12—H12A···O1	0.96	2.32	2.882 (2)	117

C11—H11···N3	0.98	2.28	2.802 (2)	112
C14—H14B···O1^{i}	0.97	2.47	3.246 (2)	137
C12—H12C···O1^{i}	0.96	2.53	3.483 (3)	170

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

Fig. 1

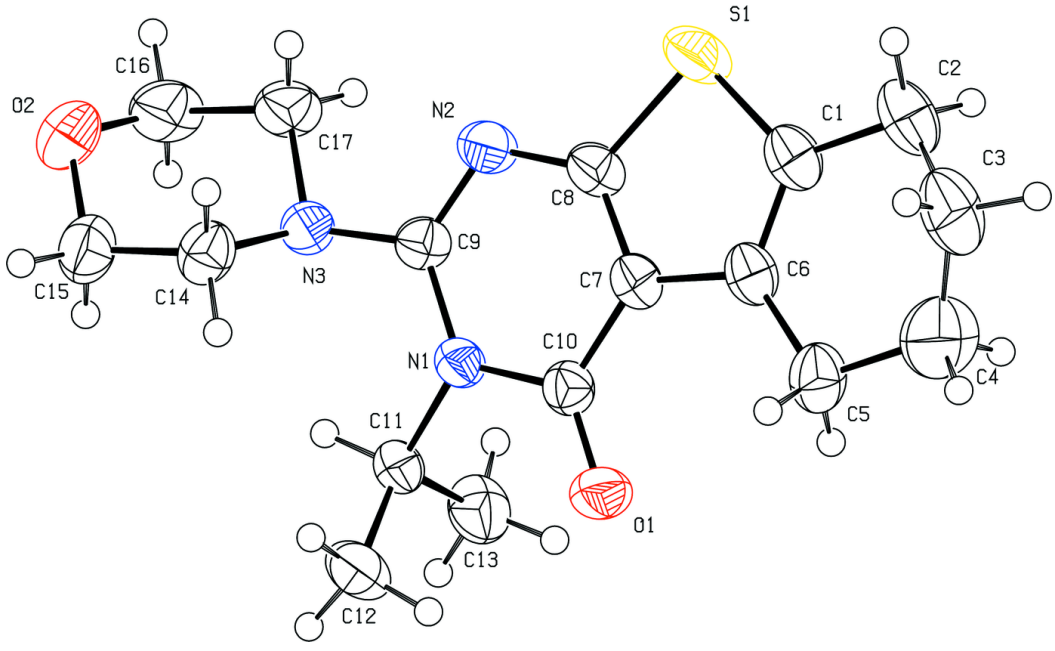

S1
C16
O2
C17
N2
C1
C2
C3
C8
C9
N3
C15
C14
C7
C6
C10
N1
C4
C11
C5
O1
C13
C12

Fig. 2

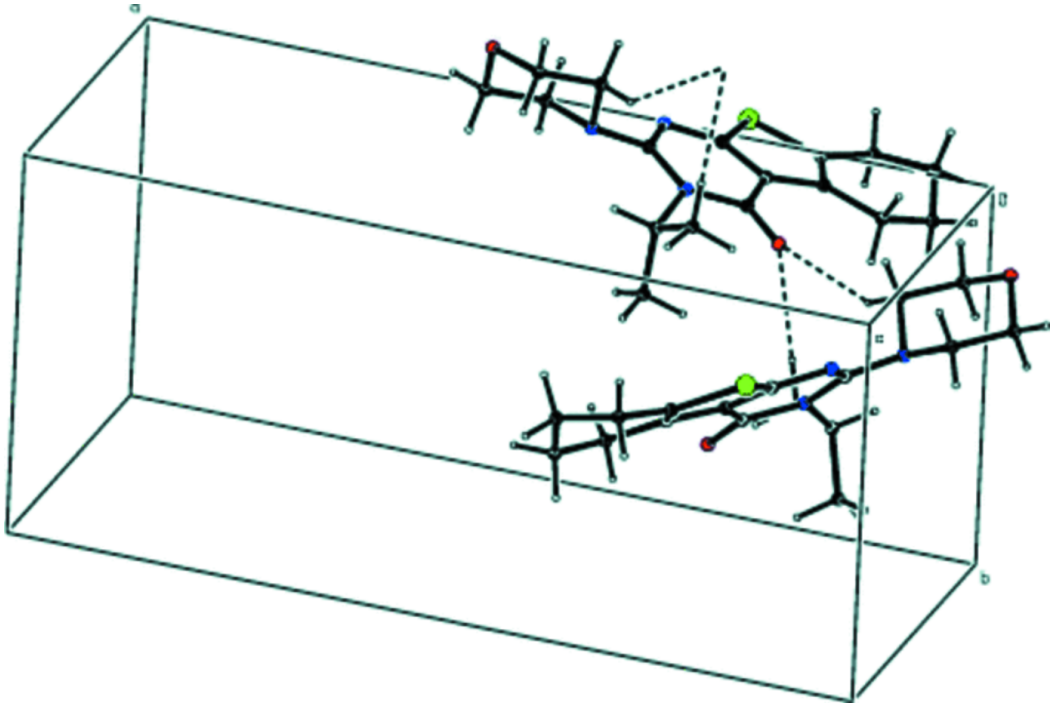
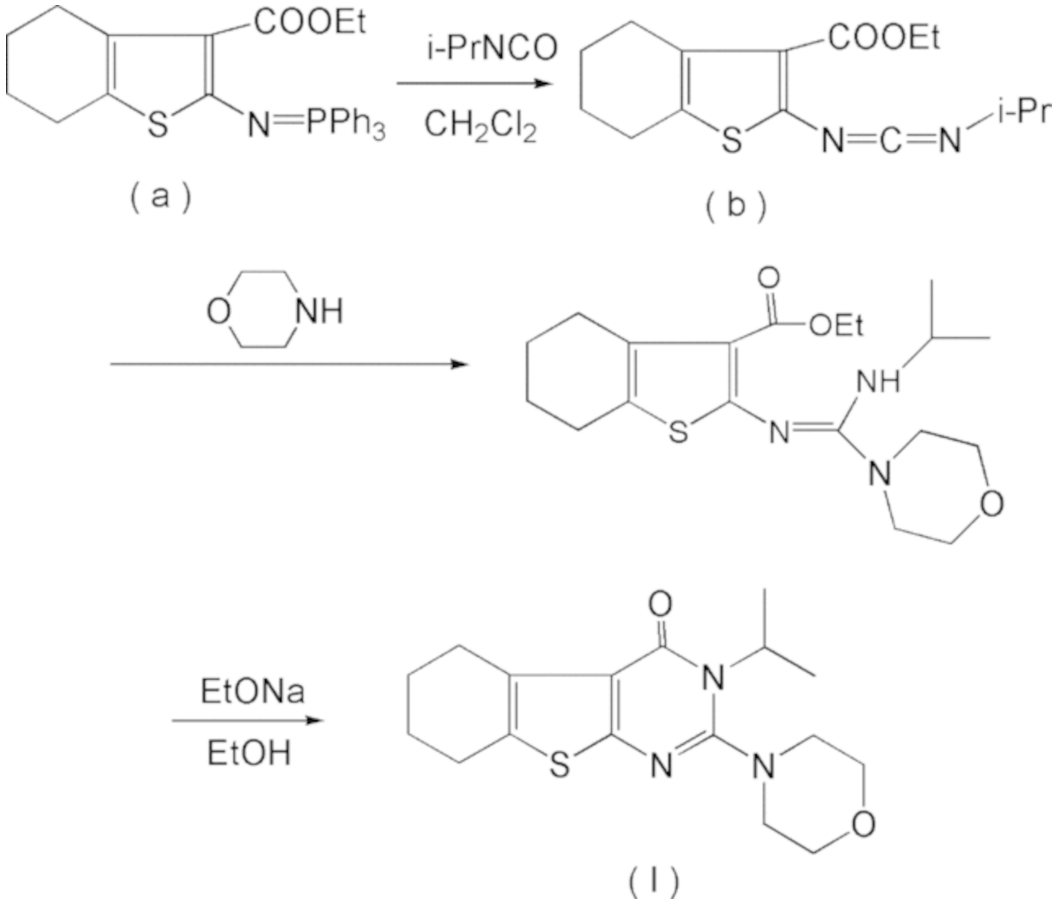


Fig. 3


COOEt
i-PrNCO
CH2Cl2
S
N=PPh3
(a)
COOEt
S
N=C=N
i-Pr
(b)
O
NH
O
OEt
NH
S
N
N
O
EtONa
EtOH
O
N
S
N
N
O
(I)