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Crystal structure of 3-methyl-2,6-bis(4-methyl-1,3-thiazol-5-yl)piperidin-4-one

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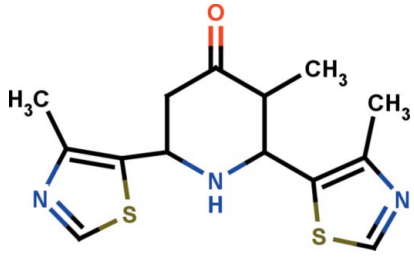
In the title compound, $C_{14}H_{17}N_3OS_2$, the central piperidinone ring adopts a chair conformation and the thiazole rings are inclined to its mean plane by 80.16 (12) and 67.15 (12)°. The O atom and methyl group C atom deviate significantly from the mean plane of the central piperidinone ring, by 0.8138 (2) and 0.3175 (2) Å, respectively. The dihedral angle between the thiazole rings is 51.88 (13)°. In the crystal, molecules are linked *via* C—H···O hydrogen bonds, forming zigzag *C*(10) chains running parallel to [001].

Keywords: crystal structure; thiazole; piperidine; zigzag chains.

CCDC reference: 1020191

1. Related literature

For biological and pharmaceutical applications of piperidinones and thiazoles, see: Ganellin & Spickett (1965). For the synthesis of substituted piperidin-4-ones and their derivatives, see: Noller & Baliah (1948). For related structures, see: Gayathri *et al.* (2008); Nithya *et al.* (2009).



2. Experimental

2.1. Crystal data

$C_{14}H_{17}N_3OS_2$
M_r = 307.43
Orthorhombic, *Pbca*
a = 11.389 (5) Å
b = 12.660 (5) Å
c = 21.667 (5) Å
V = 3124 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 296 K
0.30 × 0.25 × 0.20 mm

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.903, T_{max} = 0.934
19116 measured reflections
3762 independent reflections
2539 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.150
S = 1.00
3762 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···N2^{i}	0.93	2.49	3.365 (4)	157

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2768).

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supporting information

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Crystal structure of 3-methyl-2,6-bis(4-methyl-1,3-thiazol-5-yl)piperidin-4-one

A. Manimaran, K. Sethusankar, S. Ganesan and S. Ananthan

S1. Experimental

4-methyl-5-formyl thiazole (0.20 mol), 2-butanone (0.10 mol) and ammonium acetate (0.10 mol) were dissolved in 80 ml of distilled ethanol and heated over a boiling water bath with stirring for 8–10 h. Hydrochloric acid in isopropyl alcohol was added and the compound was filtered off as the hydrochloride salt under a nitrogen atmosphere. The compound was neutralized and extracted with dichloromethane. The dichloromethane layer was concentrated and crystals of the title compound were obtained by slow evaporation of a solution in ethanol.

S2. Refinement

The H atoms were localized from difference electron density maps. During refinement they were treated as riding atoms: N-H = 0.86 Å, C—H = 0.93 - 0.98 Å with U_{iso}(H) = 1.5*U*eq(C-methyl) and = 1.2U_{eq}(N,C) for other H atoms.

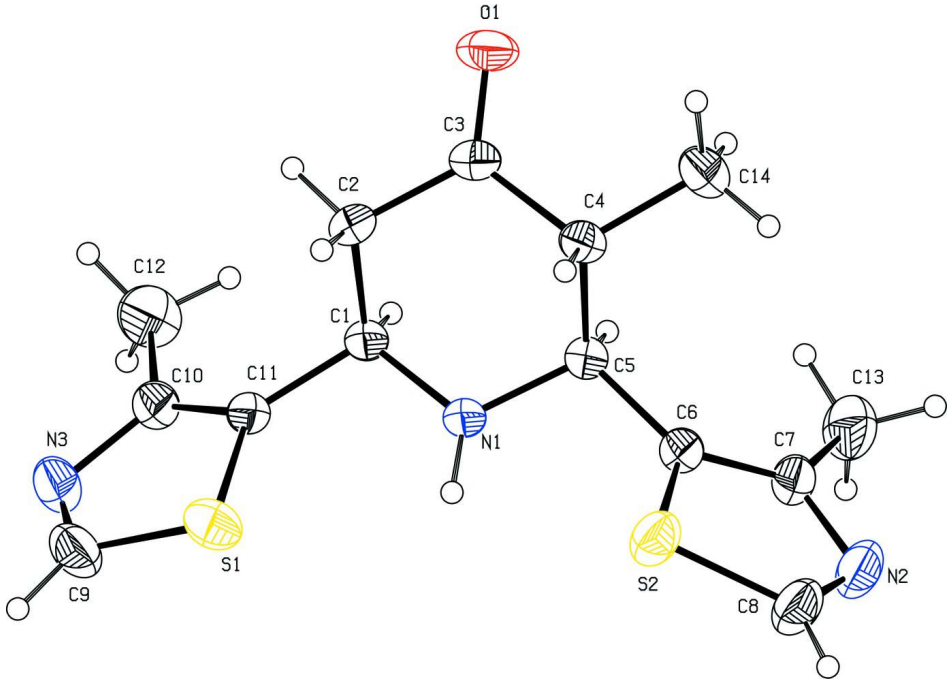

Figure 1

The molecular structure of the title molecular, with atom labelling. The displacement ellipsoids are drawn at the 30% probability level.

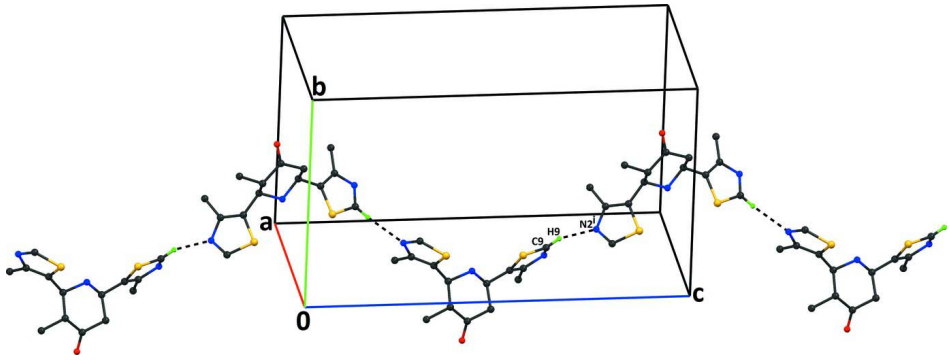

Figure 2

Part of the crystal packing of the title compound viewed along the *b* axis. Hydrogen bonds are shown as dashed lines; see Table 1 for details.

3-Methyl-2,6-bis(4-methyl-1,3-thiazol-5-yl)piperidin-4-one

Crystal data

$C_{14}H_{17}N_3OS_2$
M_r = 307.43
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 11.389 (5) Å
b = 12.660 (5) Å
c = 21.667 (5) Å
V = 3124 (2) Å^3
Z = 8
F(000) = 1296
D_x = 1.307 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 2539 reflections
θ = 2.6–28.4°
μ = 0.34 mm^{-1}
T = 296 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.903, T_{max} = 0.934
19116 measured reflections
3762 independent reflections
2539 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 28.4°, θ_{min} = 2.6°
h = −15→15
k = −16→14
l = −27→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.150
S = 1.00
3762 reflections
184 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
$w = 1/[\sigma^2(F_o^2) + (0.073P)^2 + 1.5978P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2579 (2)	−0.01628 (17)	0.49282 (10)	0.0398 (5)
H1	0.3299	−0.0482	0.4765	0.048*
C2	0.1630 (2)	−0.10230 (18)	0.49779 (11)	0.0494 (6)
H2A	0.1914	−0.1595	0.5235	0.059*
H2B	0.0933	−0.0730	0.5170	0.059*
C3	0.1325 (2)	−0.14405 (17)	0.43518 (11)	0.0447 (5)
C4	0.1030 (2)	−0.06171 (17)	0.38720 (11)	0.0423 (5)
H4	0.0300	−0.0275	0.4003	0.051*
C5	0.19957 (19)	0.02385 (17)	0.38724 (10)	0.0393 (5)
H5	0.2730	−0.0079	0.3727	0.047*
C6	0.1691 (2)	0.11454 (18)	0.34598 (11)	0.0435 (5)
C7	0.2132 (2)	0.1418 (2)	0.29028 (11)	0.0514 (6)
C8	0.0832 (3)	0.2696 (2)	0.29990 (14)	0.0647 (8)
H8	0.0403	0.3295	0.2895	0.078*
C9	0.2859 (3)	0.1374 (3)	0.64598 (14)	0.0684 (8)
H9	0.2725	0.1849	0.6780	0.082*
C10	0.3678 (2)	0.0071 (2)	0.59575 (11)	0.0492 (6)
C11	0.2849 (2)	0.03318 (17)	0.55366 (10)	0.0404 (5)
C12	0.4537 (3)	−0.0816 (3)	0.59110 (16)	0.0757 (9)
H12A	0.4317	−0.1368	0.6192	0.114*
H12B	0.5307	−0.0564	0.6014	0.114*
H12C	0.4538	−0.1087	0.5497	0.114*
C13	0.3096 (3)	0.0875 (3)	0.25606 (15)	0.0752 (9)
H13A	0.3797	0.1294	0.2583	0.113*
H13B	0.2871	0.0787	0.2137	0.113*
H13C	0.3239	0.0196	0.2742	0.113*
C14	0.0807 (3)	−0.1090 (2)	0.32444 (12)	0.0630 (7)
H14A	0.1513	−0.1420	0.3096	0.095*
H14B	0.0573	−0.0544	0.2963	0.095*
H14C	0.0195	−0.1609	0.3274	0.095*
N1	0.21742 (16)	0.06327 (14)	0.44979 (8)	0.0402 (4)
H1A	0.2050	0.1279	0.4602	0.048*
N2	0.1627 (2)	0.2306 (2)	0.26420 (11)	0.0649 (6)
N3	0.3680 (2)	0.0677 (2)	0.64846 (10)	0.0656 (6)
O1	0.13353 (18)	−0.23686 (14)	0.42350 (9)	0.0653 (5)

S1	0.20268 (6)	0.13746 (6)	0.58011 (3)	0.0607 (2)
S2	0.06218 (6)	0.20349 (6)	0.36761 (3)	0.0582 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0485 (12)	0.0321 (11)	0.0387 (12)	0.0025 (9)	−0.0018 (9)	−0.0007 (9)
C2	0.0679 (15)	0.0339 (11)	0.0464 (14)	−0.0065 (11)	−0.0023 (11)	0.0072 (10)
C3	0.0467 (12)	0.0337 (12)	0.0537 (14)	−0.0044 (9)	0.0014 (10)	−0.0022 (10)
C4	0.0451 (11)	0.0376 (11)	0.0443 (13)	−0.0024 (9)	−0.0015 (10)	−0.0042 (9)
C5	0.0441 (11)	0.0364 (11)	0.0375 (12)	−0.0004 (9)	−0.0007 (9)	−0.0003 (9)
C6	0.0492 (12)	0.0417 (12)	0.0397 (12)	−0.0049 (10)	−0.0033 (10)	0.0007 (9)
C7	0.0618 (15)	0.0530 (14)	0.0393 (14)	−0.0086 (12)	−0.0001 (11)	0.0052 (11)
C8	0.0773 (18)	0.0585 (17)	0.0583 (18)	0.0041 (14)	−0.0084 (15)	0.0211 (13)
C9	0.088 (2)	0.0675 (19)	0.0494 (17)	−0.0164 (17)	0.0147 (14)	−0.0199 (14)
C10	0.0536 (13)	0.0494 (14)	0.0446 (14)	−0.0061 (11)	−0.0038 (11)	−0.0004 (10)
C11	0.0491 (12)	0.0345 (11)	0.0377 (12)	−0.0008 (9)	0.0028 (9)	−0.0007 (9)
C12	0.0685 (18)	0.074 (2)	0.085 (2)	0.0149 (16)	−0.0254 (16)	−0.0040 (17)
C13	0.085 (2)	0.084 (2)	0.0569 (18)	−0.0011 (17)	0.0187 (15)	0.0051 (16)
C14	0.0793 (18)	0.0601 (17)	0.0496 (16)	−0.0130 (14)	−0.0044 (13)	−0.0115 (13)
N1	0.0541 (11)	0.0291 (9)	0.0376 (10)	0.0013 (8)	−0.0033 (8)	−0.0005 (7)
N2	0.0783 (15)	0.0663 (15)	0.0502 (14)	−0.0056 (13)	−0.0050 (12)	0.0214 (11)
N3	0.0822 (17)	0.0697 (16)	0.0449 (13)	−0.0143 (14)	−0.0069 (12)	−0.0080 (11)
O1	0.0847 (14)	0.0328 (9)	0.0784 (14)	−0.0010 (9)	−0.0094 (11)	−0.0062 (8)
S1	0.0656 (4)	0.0514 (4)	0.0651 (5)	0.0086 (3)	0.0065 (3)	−0.0160 (3)
S2	0.0670 (4)	0.0539 (4)	0.0538 (4)	0.0132 (3)	0.0032 (3)	0.0126 (3)

Geometric parameters (Å, º)

C1—N1	1.448 (3)	C8—S2	1.706 (3)
C1—C11	1.491 (3)	C8—H8	0.9300
C1—C2	1.538 (3)	C9—N3	1.287 (4)
C1—H1	0.9800	C9—S1	1.713 (3)
C2—C3	1.497 (3)	C9—H9	0.9300
C2—H2A	0.9700	C10—C11	1.354 (3)
C2—H2B	0.9700	C10—N3	1.376 (3)
C3—O1	1.202 (3)	C10—C12	1.493 (4)
C3—C4	1.510 (3)	C11—S1	1.717 (2)
C4—C14	1.507 (3)	C12—H12A	0.9600
C4—C5	1.543 (3)	C12—H12B	0.9600
C4—H4	0.9800	C12—H12C	0.9600
C5—N1	1.458 (3)	C13—H13A	0.9600
C5—C6	1.496 (3)	C13—H13B	0.9600
C5—H5	0.9800	C13—H13C	0.9600
C6—C7	1.352 (3)	C14—H14A	0.9600
C6—S2	1.723 (3)	C14—H14B	0.9600
C7—N2	1.384 (3)	C14—H14C	0.9600
C7—C13	1.492 (4)	N1—H1A	0.8600

C8—N2	1.289 (4)		
N1—C1—C11	110.04 (18)	S2—C8—H8	122.4
N1—C1—C2	108.27 (18)	N3—C9—S1	115.9 (2)
C11—C1—C2	112.34 (19)	N3—C9—H9	122.0
N1—C1—H1	108.7	S1—C9—H9	122.0
C11—C1—H1	108.7	C11—C10—N3	115.1 (2)
C2—C1—H1	108.7	C11—C10—C12	126.5 (2)
C3—C2—C1	110.46 (19)	N3—C10—C12	118.3 (2)
C3—C2—H2A	109.6	C10—C11—C1	129.5 (2)
C1—C2—H2A	109.6	C10—C11—S1	110.05 (18)
C3—C2—H2B	109.6	C1—C11—S1	120.38 (17)
C1—C2—H2B	109.6	C10—C12—H12A	109.5
H2A—C2—H2B	108.1	C10—C12—H12B	109.5
O1—C3—C2	122.3 (2)	H12A—C12—H12B	109.5
O1—C3—C4	122.1 (2)	C10—C12—H12C	109.5
C2—C3—C4	115.58 (19)	H12A—C12—H12C	109.5
C14—C4—C3	112.6 (2)	H12B—C12—H12C	109.5
C14—C4—C5	113.6 (2)	C7—C13—H13A	109.5
C3—C4—C5	109.01 (18)	C7—C13—H13B	109.5
C14—C4—H4	107.1	H13A—C13—H13B	109.5
C3—C4—H4	107.1	C7—C13—H13C	109.5
C5—C4—H4	107.1	H13A—C13—H13C	109.5
N1—C5—C6	108.96 (18)	H13B—C13—H13C	109.5
N1—C5—C4	109.88 (18)	C4—C14—H14A	109.5
C6—C5—C4	111.90 (18)	C4—C14—H14B	109.5
N1—C5—H5	108.7	H14A—C14—H14B	109.5
C6—C5—H5	108.7	C4—C14—H14C	109.5
C4—C5—H5	108.7	H14A—C14—H14C	109.5
C7—C6—C5	130.0 (2)	H14B—C14—H14C	109.5
C7—C6—S2	109.81 (19)	C1—N1—C5	113.88 (17)
C5—C6—S2	120.19 (17)	C1—N1—H1A	123.1
C6—C7—N2	114.6 (2)	C5—N1—H1A	123.1
C6—C7—C13	126.8 (3)	C8—N2—C7	111.0 (2)
N2—C7—C13	118.5 (2)	C9—N3—C10	110.3 (2)
N2—C8—S2	115.3 (2)	C9—S1—C11	88.64 (14)
N2—C8—H8	122.4	C8—S2—C6	89.28 (14)
N1—C1—C2—C3	54.1 (2)	N3—C10—C11—S1	−0.1 (3)
C11—C1—C2—C3	175.84 (19)	C12—C10—C11—S1	177.6 (2)
C1—C2—C3—O1	127.6 (3)	N1—C1—C11—C10	−147.0 (2)
C1—C2—C3—C4	−51.0 (3)	C2—C1—C11—C10	92.3 (3)
O1—C3—C4—C14	−2.3 (3)	N1—C1—C11—S1	34.6 (3)
C2—C3—C4—C14	176.4 (2)	C2—C1—C11—S1	−86.1 (2)
O1—C3—C4—C5	−129.2 (2)	C11—C1—N1—C5	174.46 (18)
C2—C3—C4—C5	49.5 (3)	C2—C1—N1—C5	−62.4 (2)
C14—C4—C5—N1	−178.6 (2)	C6—C5—N1—C1	−174.67 (18)
C3—C4—C5—N1	−52.2 (2)	C4—C5—N1—C1	62.4 (2)

C14—C4—C5—C6	60.2 (3)	S2—C8—N2—C7	0.0 (3)
C3—C4—C5—C6	−173.35 (19)	C6—C7—N2—C8	0.6 (3)
N1—C5—C6—C7	131.6 (3)	C13—C7—N2—C8	−178.5 (3)
C4—C5—C6—C7	−106.6 (3)	S1—C9—N3—C10	−0.7 (3)
N1—C5—C6—S2	−48.5 (2)	C11—C10—N3—C9	0.5 (3)
C4—C5—C6—S2	73.2 (2)	C12—C10—N3—C9	−177.4 (3)
C5—C6—C7—N2	178.9 (2)	N3—C9—S1—C11	0.6 (2)
S2—C6—C7—N2	−0.9 (3)	C10—C11—S1—C9	−0.28 (19)
C5—C6—C7—C13	−2.1 (4)	C1—C11—S1—C9	178.4 (2)
S2—C6—C7—C13	178.0 (2)	N2—C8—S2—C6	−0.5 (3)
N3—C10—C11—C1	−178.6 (2)	C7—C6—S2—C8	0.8 (2)
C12—C10—C11—C1	−0.9 (4)	C5—C6—S2—C8	−179.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···N2^{i}	0.93	2.49	3.365 (4)	157

Symmetry code: (i) x, $-y+1/2$, $z+1/2$.