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(4-Oxo-3,4-dihydroquinazolin-3-yl)-methyl piperidine-1-carbodithioate

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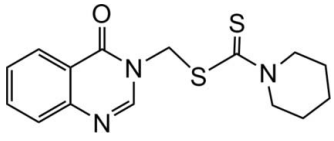
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.004 Å; R factor = 0.048; wR factor = 0.125; data-to-parameter ratio = 15.7.

In the title compound, $C_{15}H_{17}N_3OS_2$, the piperidine ring adopts a chair conformation. Weak intermolecular C—H···O hydrogen bonds link the molecules into chains along the c axis and further stability is provided by offset π–π stacking interactions [centroid separations = 3.73 (2)–3.78 (2) Å] involving the aromatic rings.

Related literature

For related literature, see: Liu *et al.* (2006); Sagara *et al.* (2006); Cao *et al.* (2005); Cremer & Pople (1975); Janiak (2000). For bond-length data, see: Allen *et al.* (1987). For a related structure, see: Jiang (2007).



Experimental

Crystal data

$C_{15}H_{17}N_3OS_2$
M_r = 319.44
Monoclinic, $P2_1/n$
a = 11.0217 (12) Å
b = 8.4199 (9) Å
c = 17.2294 (19) Å
β = 108.354 (2)°
V = 1517.6 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 292 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
8066 measured reflections
2982 independent reflections
2425 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.125
S = 1.06
2982 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···O1^{i}	0.97	2.56	3.375 (3)	142

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

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* (Bruker, 2001).

The author thanks Dr Meng Xiang-Gao for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2479).

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supplementary materials

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(4-Oxo-3,4-dihydroquinazolin-3-yl)methyl piperidine-1-carbodithioate

N.-F. She and W. Huang

Comment

Quinazolinone is a naturally occurring alkaloid as well as a core structural subunit in a growing class of bioactive natural products and synthetic compounds (Liu *et al.*, 2006). Furthermore, piperidine derivatives are found to possess pharmacological activities and can form an essential part of the molecular structure of important drugs (Sagara *et al.*, 2006). We report herein the crystal structure of the title dithiocarbamate derivative, (I).

In the molecule of (I), (Fig. 1) the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987) and are in accordance with the corresponding values in similar compounds (Jiang, 2007). The rings A (N3/C11–1 C5) are not planar having total puckering amplitude, Q_T of 0.567 (3) Å, and a chair conformation [φ = 198 (15)°, θ_2 = 0.013 (3) Å, θ_3 = −0.568 (3)Å (Cremer & Pople, 1975). Rings B (N1/N2/C6—C8) and C (C1—C6) are, of course, planar and they are also almost coplanar with a dihedral B/C angle of 1.38 (3)°.

In the crystal structure, weak intermolecular C—H···O hydrogen bonds (Table 1) link the molecules into chains along the *c* axis. Further stability is provided by offset π-π stacking interactions (Janiak, 2000), involving the rings; B and C. The adjacent C rings have a centroid-centroid distance of 3.73 (2) %A [symmetry code: 1 − *x*, 2 − *y*, − *z*], while rings B and C have a centroid-centroid distance of 3.78 (1) %A [symmetry codes: 1 − *x*, 2 − *y*, − *z*].

Experimental

The title compound was synthesized according to the literature method (Cao *et al.*, 2005) and blue blocks of (I) were obtained by slow evaporation of a dichloromethane solution at 283 K.

Refinement

The H atoms were positioned geometrically, with C—H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

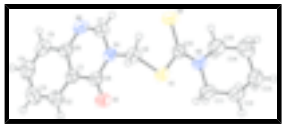

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms).

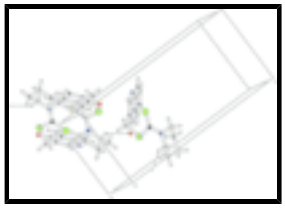

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

(4-Oxo-3,4-dihydroquinazolin-3-yl)methyl piperidine-1-carbodithioate

Crystal data

$C_{15}H_{17}N_3OS_2$	F_{000} = 672
M_r = 319.44	D_x = 1.398 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2499 reflections
a = 11.0217 (12) Å	θ = 2.5–24.4º
b = 8.4199 (9) Å	μ = 0.35 mm^{-1}
c = 17.2294 (19) Å	T = 292 (2) K
β = 108.354 (2)º	Block, blue
V = 1517.6 (3) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2425 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.025
Monochromator: graphite	θ_{max} = 26.0º
T = 292(2) K	θ_{min} = 2.0º
φ and ω scans	h = −13→11
Absorption correction: none	k = −10→8
8066 measured reflections	l = −21→21
2982 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
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H-atom parameters constrained
$wR(F^2)$ = 0.125	$w = 1/[\sigma^2(F_o^2) + (0.0622P)^2 + 0.3628P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
2982 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
190 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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}
C1	0.5285 (2)	1.0998 (2)	0.10061 (13)	0.0421 (5)
C2	0.4044 (2)	1.0422 (3)	0.08460 (15)	0.0544 (6)
H2	0.3890	0.9570	0.1145	0.065*
C3	0.3050 (3)	1.1106 (3)	0.02491 (17)	0.0647 (7)
H3	0.2222	1.0726	0.0146	0.078*
C4	0.3285 (3)	1.2365 (3)	−0.01997 (16)	0.0647 (7)
H4	0.2609	1.2825	−0.0605	0.078*
C5	0.4488 (3)	1.2942 (3)	−0.00588 (15)	0.0570 (6)
H5	0.4629	1.3781	−0.0371	0.068*
C6	0.5516 (2)	1.2276 (2)	0.05554 (13)	0.0458 (5)
C7	0.7630 (2)	1.2294 (3)	0.12735 (14)	0.0496 (5)
H7	0.8442	1.2730	0.1380	0.060*
C8	0.6342 (2)	1.0307 (2)	0.16534 (13)	0.0432 (5)
C9	0.8636 (2)	1.0460 (3)	0.23950 (13)	0.0505 (6)
H9A	0.8447	1.0497	0.2908	0.061*
H9B	0.9342	1.1181	0.2440	0.061*
C10	1.0075 (2)	0.8722 (3)	0.15837 (13)	0.0457 (5)
C11	1.0068 (3)	0.5750 (3)	0.15399 (18)	0.0640 (7)
H11A	0.9557	0.5205	0.1050	0.077*
H11B	0.9562	0.5846	0.1907	0.077*
C12	1.1246 (3)	0.4807 (3)	0.19436 (17)	0.0659 (7)
H12A	1.1005	0.3750	0.2065	0.079*
H12B	1.1718	0.5309	0.2457	0.079*
C13	1.2096 (3)	0.4680 (3)	0.14057 (19)	0.0718 (8)
H13A	1.1665	0.4067	0.0920	0.086*
H13B	1.2881	0.4135	0.1699	0.086*
C14	1.2404 (3)	0.6332 (4)	0.11589 (19)	0.0744 (8)
H14A	1.2932	0.6893	0.1638	0.089*
H14B	1.2881	0.6240	0.0775	0.089*
C15	1.1202 (3)	0.7259 (3)	0.07737 (17)	0.0717 (8)
H15A	1.1420	0.8325	0.0650	0.086*

H15B	1.0715	0.6757	0.0264	0.086*
N1	0.75210 (17)	1.1016 (2)	0.17428 (10)	0.0416 (4)
N2	0.6728 (2)	1.2941 (2)	0.07065 (12)	0.0533 (5)
N3	1.0413 (2)	0.7346 (2)	0.13230 (13)	0.0605 (6)
O1	0.62564 (16)	0.92246 (19)	0.21039 (10)	0.0564 (4)
S1	0.91313 (6)	0.84706 (7)	0.22454 (4)	0.0515 (2)
S2	1.04652 (7)	1.04974 (7)	0.13190 (4)	0.0617 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0517 (13)	0.0367 (11)	0.0415 (11)	0.0018 (9)	0.0197 (10)	−0.0077 (9)
C2	0.0577 (15)	0.0546 (15)	0.0563 (14)	−0.0054 (12)	0.0256 (12)	−0.0060 (11)
C3	0.0482 (15)	0.0742 (18)	0.0714 (18)	−0.0019 (13)	0.0183 (14)	−0.0148 (14)
C4	0.0623 (17)	0.0619 (17)	0.0593 (15)	0.0133 (13)	0.0039 (13)	−0.0063 (13)
C5	0.0705 (17)	0.0443 (13)	0.0500 (13)	0.0062 (12)	0.0100 (12)	0.0021 (11)
C6	0.0553 (14)	0.0376 (11)	0.0459 (12)	0.0003 (10)	0.0178 (11)	−0.0071 (9)
C7	0.0522 (14)	0.0387 (12)	0.0584 (14)	−0.0056 (10)	0.0180 (12)	−0.0015 (10)
C8	0.0555 (14)	0.0374 (11)	0.0426 (12)	0.0016 (10)	0.0236 (10)	−0.0040 (9)
C9	0.0562 (14)	0.0508 (14)	0.0442 (12)	0.0036 (11)	0.0156 (11)	−0.0019 (10)
C10	0.0446 (12)	0.0460 (13)	0.0453 (12)	0.0016 (10)	0.0124 (10)	0.0084 (10)
C11	0.0727 (18)	0.0443 (14)	0.0844 (19)	−0.0009 (12)	0.0384 (15)	0.0035 (13)
C12	0.081 (2)	0.0482 (15)	0.0720 (17)	0.0015 (13)	0.0291 (15)	0.0061 (12)
C13	0.0756 (19)	0.0622 (18)	0.0811 (19)	0.0133 (14)	0.0295 (16)	0.0005 (14)
C14	0.077 (2)	0.079 (2)	0.081 (2)	−0.0004 (15)	0.0442 (17)	−0.0045 (15)
C15	0.094 (2)	0.0644 (17)	0.0728 (18)	0.0065 (15)	0.0503 (17)	0.0104 (14)
N1	0.0496 (11)	0.0363 (9)	0.0404 (10)	0.0022 (8)	0.0164 (8)	−0.0007 (7)
N2	0.0625 (13)	0.0391 (10)	0.0564 (12)	−0.0029 (9)	0.0161 (10)	0.0072 (9)
N3	0.0738 (15)	0.0460 (12)	0.0757 (14)	0.0061 (10)	0.0435 (12)	0.0107 (10)
O1	0.0660 (11)	0.0528 (10)	0.0578 (10)	0.0023 (8)	0.0304 (9)	0.0149 (8)
S1	0.0585 (4)	0.0467 (4)	0.0552 (4)	0.0073 (3)	0.0264 (3)	0.0134 (3)
S2	0.0726 (5)	0.0445 (4)	0.0753 (5)	−0.0047 (3)	0.0339 (4)	0.0094 (3)

Geometric parameters (Å, °)

C1—C2	1.394 (3)	C9—H9B	0.9700
C1—C6	1.396 (3)	C10—N3	1.337 (3)
C1—C8	1.457 (3)	C10—S2	1.658 (2)
C2—C3	1.371 (4)	C10—S1	1.782 (2)
C2—H2	0.9300	C11—N3	1.477 (3)
C3—C4	1.384 (4)	C11—C12	1.493 (4)
C3—H3	0.9300	C11—H11A	0.9700
C4—C5	1.361 (4)	C11—H11B	0.9700
C4—H4	0.9300	C12—C13	1.514 (4)
C5—C6	1.401 (3)	C12—H12A	0.9700
C5—H5	0.9300	C12—H12B	0.9700
C6—N2	1.396 (3)	C13—C14	1.524 (4)
C7—N2	1.276 (3)	C13—H13A	0.9700
C7—N1	1.374 (3)	C13—H13B	0.9700

C7—H7	0.9300	C14—C15	1.500 (4)
C8—O1	1.220 (2)	C14—H14A	0.9700
C8—N1	1.394 (3)	C14—H14B	0.9700
C9—N1	1.457 (3)	C15—N3	1.475 (3)
C9—S1	1.805 (2)	C15—H15A	0.9700
C9—H9A	0.9700	C15—H15B	0.9700
C2—C1—C6	119.8 (2)	N3—C11—H11B	109.6
C2—C1—C8	120.7 (2)	C12—C11—H11B	109.6
C6—C1—C8	119.5 (2)	H11A—C11—H11B	108.1
C3—C2—C1	120.3 (2)	C11—C12—C13	111.5 (2)
C3—C2—H2	119.8	C11—C12—H12A	109.3
C1—C2—H2	119.8	C13—C12—H12A	109.3
C2—C3—C4	119.6 (2)	C11—C12—H12B	109.3
C2—C3—H3	120.2	C13—C12—H12B	109.3
C4—C3—H3	120.2	H12A—C12—H12B	108.0
C5—C4—C3	121.2 (2)	C12—C13—C14	110.0 (2)
C5—C4—H4	119.4	C12—C13—H13A	109.7
C3—C4—H4	119.4	C14—C13—H13A	109.7
C4—C5—C6	120.2 (2)	C12—C13—H13B	109.7
C4—C5—H5	119.9	C14—C13—H13B	109.7
C6—C5—H5	119.9	H13A—C13—H13B	108.2
N2—C6—C1	122.5 (2)	C15—C14—C13	110.8 (3)
N2—C6—C5	118.6 (2)	C15—C14—H14A	109.5
C1—C6—C5	118.9 (2)	C13—C14—H14A	109.5
N2—C7—N1	126.4 (2)	C15—C14—H14B	109.5
N2—C7—H7	116.8	C13—C14—H14B	109.5
N1—C7—H7	116.8	H14A—C14—H14B	108.1
O1—C8—N1	120.4 (2)	N3—C15—C14	111.0 (2)
O1—C8—C1	125.6 (2)	N3—C15—H15A	109.4
N1—C8—C1	113.98 (18)	C14—C15—H15A	109.4
N1—C9—S1	114.06 (15)	N3—C15—H15B	109.4
N1—C9—H9A	108.7	C14—C15—H15B	109.4
S1—C9—H9A	108.7	H15A—C15—H15B	108.0
N1—C9—H9B	108.7	C7—N1—C8	121.46 (19)
S1—C9—H9B	108.7	C7—N1—C9	119.77 (19)
H9A—C9—H9B	107.6	C8—N1—C9	118.58 (18)
N3—C10—S2	124.41 (17)	C7—N2—C6	116.2 (2)
N3—C10—S1	113.08 (16)	C10—N3—C15	122.7 (2)
S2—C10—S1	122.50 (14)	C10—N3—C11	125.7 (2)
N3—C11—C12	110.3 (2)	C15—N3—C11	111.6 (2)
N3—C11—H11A	109.6	C10—S1—C9	104.16 (11)
C12—C11—H11A	109.6		
C6—C1—C2—C3	−0.1 (3)	O1—C8—N1—C7	−176.3 (2)
C8—C1—C2—C3	178.4 (2)	C1—C8—N1—C7	2.5 (3)
C1—C2—C3—C4	0.6 (4)	O1—C8—N1—C9	−1.4 (3)
C2—C3—C4—C5	−0.1 (4)	C1—C8—N1—C9	177.51 (17)
C3—C4—C5—C6	−0.8 (4)	S1—C9—N1—C7	−116.52 (19)
C2—C1—C6—N2	177.7 (2)	S1—C9—N1—C8	68.4 (2)

C8—C1—C6—N2	−0.8 (3)	N1—C7—N2—C6	0.1 (3)
C2—C1—C6—C5	−0.7 (3)	C1—C6—N2—C7	1.4 (3)
C8—C1—C6—C5	−179.24 (19)	C5—C6—N2—C7	179.8 (2)
C4—C5—C6—N2	−177.3 (2)	S2—C10—N3—C15	1.1 (4)
C4—C5—C6—C1	1.2 (3)	S1—C10—N3—C15	−179.6 (2)
C2—C1—C8—O1	−0.8 (3)	S2—C10—N3—C11	−179.3 (2)
C6—C1—C8—O1	177.7 (2)	S1—C10—N3—C11	0.0 (3)
C2—C1—C8—N1	−179.60 (18)	C14—C15—N3—C10	121.4 (3)
C6—C1—C8—N1	−1.1 (3)	C14—C15—N3—C11	−58.3 (3)
N3—C11—C12—C13	−57.0 (3)	C12—C11—N3—C10	−121.2 (3)
C11—C12—C13—C14	54.9 (3)	C12—C11—N3—C15	58.4 (3)
C12—C13—C14—C15	−53.9 (3)	N3—C10—S1—C9	−172.68 (17)
C13—C14—C15—N3	55.9 (3)	S2—C10—S1—C9	6.57 (18)
N2—C7—N1—C8	−2.2 (3)	N1—C9—S1—C10	82.82 (18)
N2—C7—N1—C9	−177.1 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11B···O1^{i}	0.97	2.56	3.375 (3)	142

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

Fig. 1

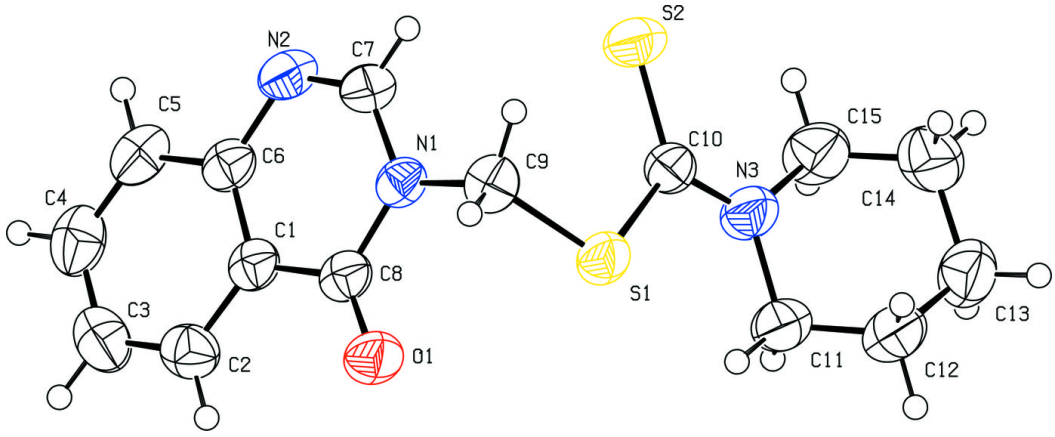

Fig. 2