4-Oxo-3,4-dihydroquinazolin-3-yl-methyl N,N-diisopropylcarbamodithioate

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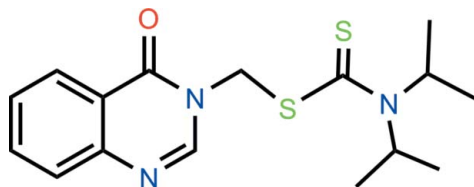
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In the title compound, $C_{16}H_{21}N_3OS_2$, the dihedral angle between the aromatic rings is 3.60 (3)°. An intermolecular C—H⋯O hydrogen bond links the molecules into chains along the c axis.

Related literature

For related literature, see: Liu *et al.* (2006); Cao *et al.* (2005). For bond-length data, see: Allen *et al.* (1987). For related structures, see: She & Huang (2007); Jiang (2007).

Experimental

Crystal data

$C_{16}H_{21}N_3OS_2$	V = 1684.3 (3) Å^3
M_r = 335.48	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 12.9585 (12) Å	μ = 0.32 mm^{-1}
b = 8.8470 (8) Å	T = 292 (2) K
c = 14.9378 (15) Å	$0.20 \times 0.10 \times 0.06$ mm
β = 100.422 (2)°	

Data collection

Bruker SMART 4K CCD diffractometer	3615 independent reflections
Absorption correction: none	2740 reflections with $I > 2\sigma(I)$
9077 measured reflections	R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.129	
S = 1.04	
3615 reflections	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
209 parameters	$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C8—H8⋯O1^i	0.93	2.57	3.103 (3)	117

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* (Bruker, 2001).

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doi:10.1107/S160053680704932X

supplementary materials

4-Oxo-3,4-dihydroquinazolin-3-ylmethyl *N,N*-diisopropylcarbamodithioate

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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, dithiocarbamate derivatives are found to possess pharmacological activities and generally form an essential part of the molecular structure of important drugs (Cao *et al.*, 2005). 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 (She & Huang, 2007). Rings A (N1/N2/C1/C6—C8) and B (C1—C6) are, of course, planar and they are also almost coplanar with a dihedral angle of A/B = 3.60 (3)°.

In the crystal, a weak intermolecular C—H···O hydrogen bond (Table 1, Fig. 2) link the molecules into chains along the *c* axis.

Experimental

The title compound was synthesized according to the literature method (She & Huang, 2007). Colourless blocks of (I) were obtained by slow evaporation of the dichloromethane solution at 283 K.

Refinement

The positions of H12 and H14 were freely refined with $U_{iso}(H) = 1.2U_{eq}(C)$. The other H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).
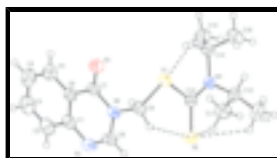
Figures

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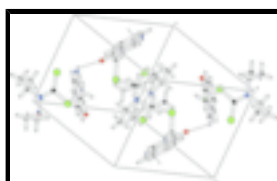

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

supplementary materials

4-Oxo-3,4-dihydroquinazolin-3-ylmethyl *N,N*-diisopropylcarbamodithioate

Crystal data

$C_{16}H_{21}N_3OS_2$	$F_{000} = 712$
$M_r = 335.48$	$D_x = 1.323$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3183 reflections
$a = 12.9585$ (12) Å	$\theta = 2.3–23.8º$
$b = 8.8470$ (8) Å	$\mu = 0.32$ mm^{-1}
$c = 14.9378$ (15) Å	$T = 292$ (2) K
$\beta = 100.422$ (2)º	Block, colorless
$V = 1684.3$ (3) Å^3	$0.20 \times 0.10 \times 0.06$ mm
$Z = 4$	

Data collection

Bruker SMART 4K CCD diffractometer	2740 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.033$
Monochromator: graphite	$\theta_{max} = 27.0º$
$T = 292(2)$ K	$\theta_{min} = 1.9º$
φ and ω scans	$h = -16\rightarrow15$
Absorption correction: none	$k = -11\rightarrow9$
9077 measured reflections	$l = -18\rightarrow19$
3615 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
$R[F^2 > 2\sigma(F^2)] = 0.050$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.129$	$w = 1/[\sigma^2(F_o^2) + (0.0646P)^2 + 0.1059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3615 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.21$ 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, convention-al 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 calculat-ing 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*	$U_{iso}*/U_{eq}$
S1	0.10518 (4)	0.08161 (6)	0.22859 (4)	0.04484 (19)
S2	−0.07121 (5)	0.30153 (7)	0.17179 (5)	0.0584 (2)
N3	−0.08186 (13)	0.0036 (2)	0.14073 (11)	0.0388 (4)
N2	0.21525 (13)	0.31472 (19)	0.17370 (11)	0.0401 (4)
C10	−0.02751 (15)	0.1253 (2)	0.17468 (13)	0.0368 (5)
O1	0.33747 (13)	0.1290 (2)	0.20134 (11)	0.0583 (5)
C9	0.16744 (17)	0.2629 (3)	0.24957 (14)	0.0444 (5)
H9A	0.1157	0.3365	0.2608	0.053*
H9B	0.2211	0.2568	0.3039	0.053*
C1	0.29368 (18)	0.4198 (3)	0.02963 (15)	0.0469 (6)
C6	0.34002 (17)	0.2922 (3)	0.07510 (14)	0.0444 (5)
N1	0.21223 (16)	0.4984 (2)	0.05789 (13)	0.0503 (5)
C7	0.30154 (16)	0.2364 (3)	0.15455 (15)	0.0437 (5)
C14	−0.03619 (17)	−0.1516 (3)	0.14633 (15)	0.0448 (5)
H14	0.0382 (19)	−0.142 (3)	0.1735 (15)	0.054*
C8	0.17786 (18)	0.4423 (2)	0.12571 (15)	0.0450 (5)
H8	0.1223	0.4924	0.1444	0.054*
C12	−0.19555 (16)	0.0091 (3)	0.09710 (16)	0.0474 (6)
H12	−0.2154 (18)	−0.096 (3)	0.0815 (16)	0.057*
C5	0.42100 (19)	0.2173 (3)	0.04318 (18)	0.0596 (7)
H5	0.4525	0.1331	0.0740	0.071*
C15	−0.0415 (2)	−0.2205 (3)	0.05282 (18)	0.0653 (7)
H15A	−0.1134	−0.2398	0.0262	0.098*
H15B	−0.0029	−0.3137	0.0581	0.098*
H15C	−0.0116	−0.1516	0.0149	0.098*
C2	0.3287 (2)	0.4689 (3)	−0.04888 (17)	0.0630 (7)
H2	0.2981	0.5531	−0.0803	0.076*
C11	−0.21454 (19)	0.0946 (3)	0.00728 (16)	0.0607 (7)
H11A	−0.1963	0.1991	0.0182	0.091*
H11B	−0.2872	0.0869	−0.0203	0.091*
H11C	−0.1720	0.0519	−0.0327	0.091*
C3	0.4073 (2)	0.3937 (4)	−0.07935 (19)	0.0735 (8)
H3	0.4300	0.4272	−0.1315	0.088*
C4	0.4538 (2)	0.2679 (4)	−0.0335 (2)	0.0732 (8)
H4	0.5076	0.2178	−0.0550	0.088*
C16	−0.0861 (2)	−0.2510 (3)	0.20894 (19)	0.0701 (8)
H16A	−0.0771	−0.2055	0.2682	0.105*
H16B	−0.0533	−0.3486	0.2135	0.105*

H16C	−0.1596	−0.2618	0.1849	0.105*
C13	−0.26576 (19)	0.0594 (4)	0.1623 (2)	0.0725 (8)
H13A	−0.2492	0.0023	0.2177	0.109*
H13B	−0.3378	0.0428	0.1350	0.109*
H13C	−0.2548	0.1650	0.1755	0.109*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0407 (3)	0.0363 (3)	0.0540 (3)	−0.0030 (2)	−0.0009 (2)	0.0056 (2)
S2	0.0552 (4)	0.0392 (4)	0.0779 (5)	0.0094 (3)	0.0045 (3)	−0.0070 (3)
N3	0.0366 (9)	0.0397 (11)	0.0398 (9)	−0.0017 (8)	0.0056 (7)	−0.0011 (8)
N2	0.0446 (10)	0.0334 (10)	0.0410 (9)	−0.0048 (8)	0.0040 (7)	−0.0009 (7)
C10	0.0390 (10)	0.0387 (12)	0.0337 (10)	0.0014 (9)	0.0094 (8)	0.0010 (9)
O1	0.0542 (10)	0.0527 (11)	0.0664 (10)	0.0071 (8)	0.0069 (8)	0.0199 (9)
C9	0.0506 (12)	0.0410 (13)	0.0396 (11)	−0.0099 (10)	0.0026 (9)	−0.0034 (9)
C1	0.0521 (13)	0.0430 (14)	0.0428 (12)	−0.0119 (11)	0.0010 (10)	−0.0012 (10)
C6	0.0432 (12)	0.0413 (13)	0.0473 (12)	−0.0065 (10)	0.0047 (9)	−0.0018 (10)
N1	0.0608 (12)	0.0395 (11)	0.0491 (11)	−0.0002 (9)	0.0063 (9)	0.0029 (9)
C7	0.0418 (12)	0.0384 (13)	0.0478 (12)	−0.0056 (10)	−0.0004 (9)	0.0008 (10)
C14	0.0417 (12)	0.0342 (13)	0.0574 (13)	−0.0005 (10)	0.0061 (10)	−0.0009 (10)
C8	0.0511 (12)	0.0326 (12)	0.0487 (12)	0.0007 (10)	0.0024 (10)	−0.0043 (10)
C12	0.0364 (11)	0.0519 (15)	0.0532 (13)	0.0005 (10)	0.0061 (9)	−0.0017 (11)
C5	0.0516 (14)	0.0583 (17)	0.0687 (16)	−0.0011 (12)	0.0106 (12)	−0.0026 (13)
C15	0.0670 (16)	0.0579 (17)	0.0738 (17)	0.0029 (13)	0.0198 (13)	−0.0197 (13)
C2	0.0730 (18)	0.0618 (18)	0.0541 (15)	−0.0097 (14)	0.0110 (13)	0.0089 (13)
C11	0.0556 (14)	0.0644 (18)	0.0564 (14)	0.0026 (13)	−0.0052 (11)	0.0039 (12)
C3	0.079 (2)	0.087 (2)	0.0579 (16)	−0.0156 (18)	0.0217 (14)	0.0035 (15)
C4	0.0619 (17)	0.088 (2)	0.0757 (19)	−0.0038 (16)	0.0275 (15)	−0.0124 (17)
C16	0.085 (2)	0.0526 (17)	0.0726 (17)	−0.0013 (14)	0.0151 (15)	0.0173 (14)
C13	0.0442 (14)	0.094 (2)	0.0831 (19)	0.0049 (15)	0.0225 (13)	−0.0084 (16)

Geometric parameters (Å, °)

S1—C9	1.798 (2)	C12—C13	1.515 (3)
S1—C10	1.804 (2)	C12—C11	1.521 (3)
S2—C10	1.657 (2)	C12—H12	0.98 (2)
N3—C10	1.335 (3)	C5—C4	1.368 (4)
N3—C14	1.492 (3)	C5—H5	0.9300
N3—C12	1.500 (3)	C15—H15A	0.9600
N2—C8	1.377 (3)	C15—H15B	0.9600
N2—C7	1.389 (3)	C15—H15C	0.9600
N2—C9	1.460 (3)	C2—C3	1.362 (4)
O1—C7	1.221 (3)	C2—H2	0.9300
C9—H9A	0.9700	C11—H11A	0.9600
C9—H9B	0.9700	C11—H11B	0.9600
C1—N1	1.392 (3)	C11—H11C	0.9600
C1—C6	1.396 (3)	C3—C4	1.387 (4)
C1—C2	1.401 (3)	C3—H3	0.9300

C6—C5	1.396 (3)	C4—H4	0.9300
C6—C7	1.454 (3)	C16—H16A	0.9600
N1—C8	1.278 (3)	C16—H16B	0.9600
C14—C16	1.511 (3)	C16—H16C	0.9600
C14—C15	1.514 (3)	C13—H13A	0.9600
C14—H14	0.98 (2)	C13—H13B	0.9600
C8—H8	0.9300	C13—H13C	0.9600
C9—S1—C10	104.37 (10)	C13—C12—H12	105.5 (14)
C10—N3—C14	122.98 (17)	C11—C12—H12	105.5 (14)
C10—N3—C12	123.22 (18)	C4—C5—C6	119.8 (3)
C14—N3—C12	113.76 (17)	C4—C5—H5	120.1
C8—N2—C7	121.50 (19)	C6—C5—H5	120.1
C8—N2—C9	120.15 (19)	C14—C15—H15A	109.5
C7—N2—C9	118.28 (18)	C14—C15—H15B	109.5
N3—C10—S2	126.48 (16)	H15A—C15—H15B	109.5
N3—C10—S1	113.05 (15)	C14—C15—H15C	109.5
S2—C10—S1	120.47 (12)	H15A—C15—H15C	109.5
N2—C9—S1	112.62 (14)	H15B—C15—H15C	109.5
N2—C9—H9A	109.1	C3—C2—C1	120.3 (3)
S1—C9—H9A	109.1	C3—C2—H2	119.9
N2—C9—H9B	109.1	C1—C2—H2	119.9
S1—C9—H9B	109.1	C12—C11—H11A	109.5
H9A—C9—H9B	107.8	C12—C11—H11B	109.5
N1—C1—C6	122.7 (2)	H11A—C11—H11B	109.5
N1—C1—C2	118.5 (2)	C12—C11—H11C	109.5
C6—C1—C2	118.8 (2)	H11A—C11—H11C	109.5
C5—C6—C1	120.1 (2)	H11B—C11—H11C	109.5
C5—C6—C7	120.5 (2)	C2—C3—C4	120.8 (3)
C1—C6—C7	119.4 (2)	C2—C3—H3	119.6
C8—N1—C1	116.0 (2)	C4—C3—H3	119.6
O1—C7—N2	120.6 (2)	C5—C4—C3	120.2 (3)
O1—C7—C6	125.4 (2)	C5—C4—H4	119.9
N2—C7—C6	113.97 (19)	C3—C4—H4	119.9
N3—C14—C16	111.1 (2)	C14—C16—H16A	109.5
N3—C14—C15	111.55 (19)	C14—C16—H16B	109.5
C16—C14—C15	112.6 (2)	H16A—C16—H16B	109.5
N3—C14—H14	107.1 (14)	C14—C16—H16C	109.5
C16—C14—H14	107.1 (14)	H16A—C16—H16C	109.5
C15—C14—H14	107.1 (13)	H16B—C16—H16C	109.5
N1—C8—N2	126.2 (2)	C12—C13—H13A	109.5
N1—C8—H8	116.9	C12—C13—H13B	109.5
N2—C8—H8	116.9	H13A—C13—H13B	109.5
N3—C12—C13	112.78 (19)	C12—C13—H13C	109.5
N3—C12—C11	112.89 (19)	H13A—C13—H13C	109.5
C13—C12—C11	113.6 (2)	H13B—C13—H13C	109.5
N3—C12—H12	105.5 (14)		
C14—N3—C10—S2	180.00 (16)	C5—C6—C7—N2	−174.88 (19)
C12—N3—C10—S2	−2.7 (3)	C1—C6—C7—N2	3.7 (3)

C14—N3—C10—S1	−0.7 (2)	C10—N3—C14—C16	112.0 (2)
C12—N3—C10—S1	176.61 (15)	C12—N3—C14—C16	−65.5 (2)
C9—S1—C10—N3	170.00 (14)	C10—N3—C14—C15	−121.5 (2)
C9—S1—C10—S2	−10.62 (15)	C12—N3—C14—C15	61.0 (2)
C8—N2—C9—S1	115.45 (18)	C1—N1—C8—N2	1.4 (3)
C7—N2—C9—S1	−67.5 (2)	C7—N2—C8—N1	3.6 (3)
C10—S1—C9—N2	−89.67 (17)	C9—N2—C8—N1	−179.47 (19)
N1—C1—C6—C5	179.5 (2)	C10—N3—C12—C13	−62.5 (3)
C2—C1—C6—C5	1.1 (3)	C14—N3—C12—C13	115.0 (2)
N1—C1—C6—C7	0.9 (3)	C10—N3—C12—C11	68.0 (3)
C2—C1—C6—C7	−177.5 (2)	C14—N3—C12—C11	−114.5 (2)
C6—C1—N1—C8	−3.6 (3)	C1—C6—C5—C4	−0.9 (4)
C2—C1—N1—C8	174.8 (2)	C7—C6—C5—C4	177.7 (2)
C8—N2—C7—O1	175.52 (19)	N1—C1—C2—C3	−179.2 (2)
C9—N2—C7—O1	−1.5 (3)	C6—C1—C2—C3	−0.7 (4)
C8—N2—C7—C6	−5.9 (3)	C1—C2—C3—C4	0.0 (4)
C9—N2—C7—C6	177.09 (17)	C6—C5—C4—C3	0.3 (4)
C5—C6—C7—O1	3.6 (3)	C2—C3—C4—C5	0.2 (4)
C1—C6—C7—O1	−177.8 (2)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1[i]	0.93	2.57	3.103 (3)	117

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

Fig. 1

Fig. 2