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Synthesis and structure of ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl)sulfanyl]acetate

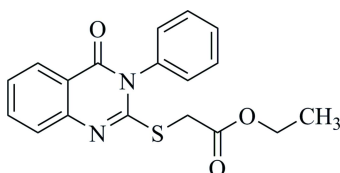
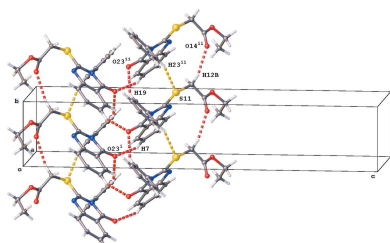
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The title compound, C₁₈H₁₆N₂O₃S, was synthesized by reaction of 2-mercapto-3-phenylquinazolin-4(3*H*)-one with ethyl chloroacetate. The quinazoline ring forms a dihedral angle of 86.83 (5)° with the phenyl ring. The terminal methyl group is disordered by a rotation of about 60° in a 0.531 (13):0.469 (13) ratio. In the crystal, C—H···O hydrogen-bonding interactions result in the formation of columns running in the [010] direction. Two parallel columns further interact by C—H···O hydrogen bonds. The most important contributions to the surface contacts are from H···H (48.4%), C···H/H···C (21.5%) and O···H/H···O (18.7%) interactions, as concluded from a Hirshfeld analysis.

1. Chemical context

Hybrid derivatives, where quinazolin-4-one is incorporated with different heterocycles, possess a variety of biological effects including anticancer (Khalil *et al.*, 2003; Gursoy & Karal, 2003; Gawad *et al.*, 2010; Elfekki *et al.*, 2014; Alanazi *et al.*, 2016; El-Sayed *et al.*, 2017; Nguyen *et al.*, 2019), anti-convulsant (El-Azab *et al.*, 2013) and antimicrobial (Pandey *et al.*, 2009; Al-Khuzai & Al-Majidi, 2014; Al-Majidi & Al-Khuzai, 2015; Lv *et al.*, 2018; Godhani *et al.*, 2016) activities. Some derivatives of 2-mercapto-3-(4-methoxyphenyl)quinazolin-4(3*H*)-one containing the thiazolidine-4-one moiety have been found to have good antituberculosis activity (Godhani *et al.*, 2016). In addition, many amide and *N*-substituted hydrazide compounds derived from 2-mercapto-3-phenylquinazolin-4-one have been demonstrated to have valuable biological activities such as antitumor (Al-Suwaidan *et al.*, 2016, 2017; Mohamed *et al.*, 2016), anti-convulsant (El-Helby & Wahab, 2003) and antibacterial (Lfta *et al.*, 2016) activity. The capacity to increase the HDL cholesterol activity of some *N*-substituted compounds containing a quinazolin-4-one moiety has also been investigated (Deshmukh & Dhongade, 2004).



Ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl)sulfanyl]acetate is an intermediate compound in the synthesis



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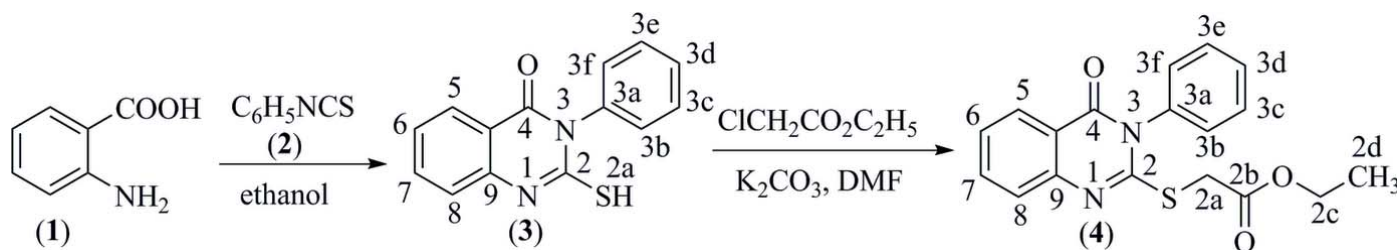


Figure 1
Reaction scheme for the synthesis of the title compound (4).

process of both *N*-substituted and heterocyclic compounds containing a quinazolin-4-one moiety. The synthesis and properties of ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl)thio]acetate have therefore attracted much attention.

As shown in Fig. 1, 2-mercapto-3-phenylquinazolin-4(3*H*)-one (**3**) was obtained by the reaction of anthranilic acid (**1**) and phenyl isothiocyanate (**2**) (Nguyen *et al.*, 2019). The IR spectrum of (**3**) shows the stretching vibrations of N–H (3217 and 3134 cm^{-1}) and C=O (1659 cm^{-1}) bonds, indicating that (**3**) exists in the thione form (Al-Majidi & Al-Khuzai, 2015). In the ^1H NMR spectrum, besides signals of nine protons in the aromatic area, there is a singlet signal with the intensity of 1H at δ 13.05 ppm attributed to the proton of the thiol group. In an alkaline medium, (**3**) exists in the thiolate form and reacts easily with ethyl chloroacetate to yield (**4**). In the IR spectrum of (**4**), the disappearance of the NH stretching and the presence of a strong C=O absorption at 1732 cm^{-1} indicate the existence of an ester compound. In the ^1H NMR spectrum of (**4**), the signal at δ 13.05 ppm disappears and three new signals in the aliphatic area [singlet signal at δ 3.99 (2H), quartet signal at δ 4.15 (2H) and triplet signal at δ 1.23 ppm (3H)] are consistent with the presence of the $-\text{CH}_2\text{COOCH}_2\text{CH}_3$ moiety in (**4**).

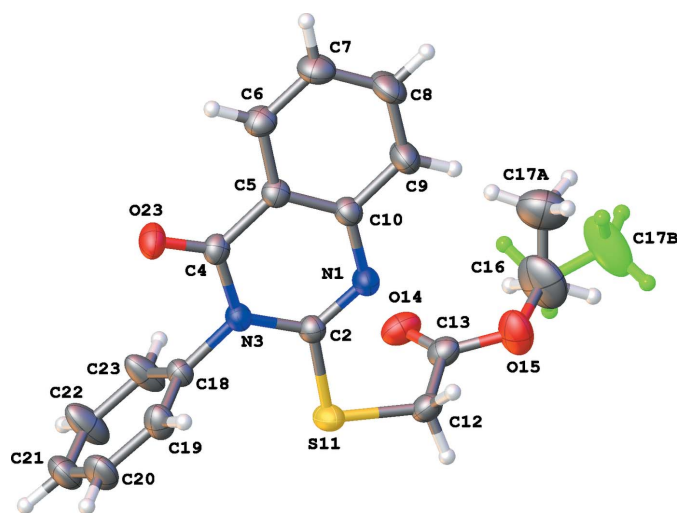


Figure 2
The molecular structure of the title compound, showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. Methyl group C17*B* [occupancy 0.469 (13)] is shown in green.

As no X-ray crystallographic information is available for this ester, we have determined the crystal structure by single-crystal X-ray diffraction and a Hirshfeld surface analysis has been performed to gain further insight into the intermolecular interactions.

2. Structural commentary

The title compound crystallizes in the space group $P2_1/n$ with four molecules in the unit cell. The asymmetric unit of the title compound is illustrated in Fig. 2. The C17 methyl group is disordered over two orientations by a rotation of about 60° about the O15–C16 bond in a 0.531 (13):0.469 (13) ratio. The quinazoline ring system is almost planar (r.m.s. deviation = 0.0207 Å). The angle between the two fused six-membered rings is $2.05 (9)^\circ$. The substituents S11, C18 and O23 deviating by $-0.0951 (17)$, $-0.140 (2)$ and $0.108 (2)$ Å, respectively, from the best plane through the quinazoline ring system. This plane makes an angle of $86.83 (5)^\circ$ with the plane of the C18–C23 phenyl ring (r.m.s. deviation = 0.0052 Å). The dihedral angle between the best planes through the acetate atoms (C12, C13, O14 and O15) and the quinazoline ring system is $75.21 (5)^\circ$. A short intramolecular C16–H16*B*···O14 contact is observed [C16–H16*B* = 0.97 Å, H16*B*···O14 = 2.28 Å, C16···O14 = 2.655 (4) Å, C16–H16*B*···O14 = 102°].

Theoretically, compound (**3**) may exist in the thione form, namely 3-phenyl-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one. Therefore, it could react with ethyl chloroacetate to give ethyl 2-(4-oxo-3-phenyl-2-thioxo-3,4-dihydroquinazolin-1(2*H*)-yl)acetate as illustrated in Fig. 3. However, our current structure determination indicates that the final product is ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl)sulfanyl]acetate (**4**), which proves that in the alkaline environment, (**3**) converts into the thiolate form and then reacts with ethyl chloroacetate to yield the title compound (**4**).

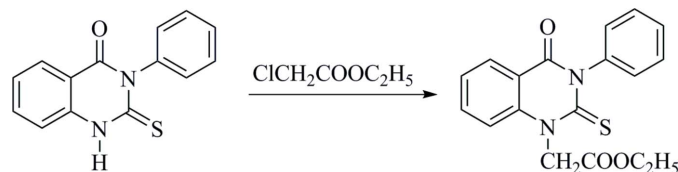


Figure 3
Reaction scheme for the thione tautomer of (**3**) with ethyl chloroacetate resulting in ethyl 2-(4-oxo-3-phenyl-2-thioxo-3,4-dihydroquinazolin-1(2*H*)-yl)acetate as reaction product.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C7-H7\cdots O23^i$	0.93	2.59	3.452 (3)	155
$C12-H12B\cdots O14^{ii}$	0.97	2.42	3.311 (3)	153
$C19-H19\cdots O23^{ii}$	0.93	2.41	3.236 (2)	148

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

3. Supramolecular features and Hirshfeld surface analysis

The crystal packing is mainly characterized by $C-H\cdots O$ hydrogen-bonding interactions (Table 1, Figs. 4 and 5). Columns running in the [010] direction are formed by $C12-H12B\cdots O14^{ii}$ and $C19-H19\cdots O23^{ii}$ interactions, which results also in a short $S11\cdots H23^{ii}$ contact of 3.020 Å [symmetry code: (ii) $x, y + 1, z$]. Two parallel columns interact via $C7-H7\cdots O23^i$ hydrogen-bonding interactions [symmetry

code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$]. No voids, $C-H\cdots\pi$ interactions or $\pi-\pi$ stackings are observed in the crystal packing.

In order to gain further insight into the intermolecular interactions, a Hirshfeld surface and two-dimensional fingerprint plots were calculated using *CrystalExplorer* (Turner *et al.*, 2017). The Hirshfeld surface mapped over d_{norm} (Fig. 6) shows the expected bright-red spots near atoms O14, O23, H7, H12B and H19 involved in the $C-H\cdots O$ hydrogen-bonding interactions described above. In addition, the faint-red spots near atoms S11 and O14 indicate a short $S\cdots O$ contact [3.2128 (16) Å]. Small faint-red spots appear near atoms H8 and H17E are due to a short $H8\cdots H17E$ contact (2.352 Å). The $S11\cdots H23$ contact mentioned is only visible as a white spot, while a white region above the C18–C23 phenyl ring is present because of the proximity of atom H20. The distance between H20 and the centroid of this phenyl ring of 3.204 Å, however, is too long for a $C-H\cdots\pi$ interaction. The fingerprint plots (Fig. 7) illustrate that the largest contributions to the Hirshfeld surface come from $H\cdots H$ contacts (48.4%), followed by significant contributions by reciprocal $C\cdots H$ /

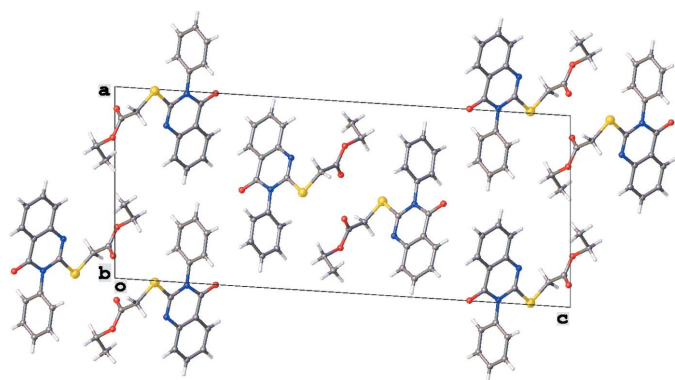


Figure 4
View of the crystal packing of the title compound along the [010] direction. Only the major component of the disordered C17 methyl group is shown.

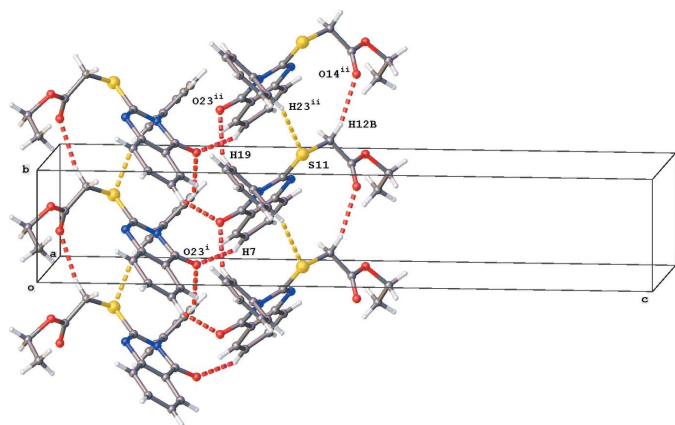


Figure 5
Partial crystal packing of the title compound showing two parallel columns running in the [010] direction. Intermolecular $C-H\cdots O$ interactions are shown as red dashed lines (see Table 1 for details), $C-H\cdots S$ interactions as yellow dashed lines. Only the major component of the disordered C17 methyl group is shown.

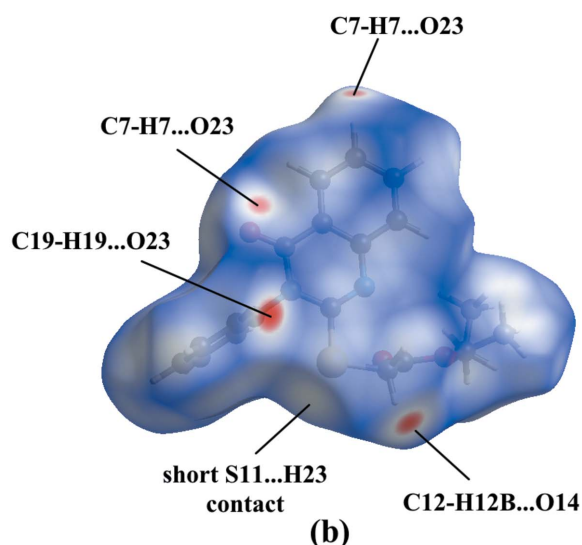
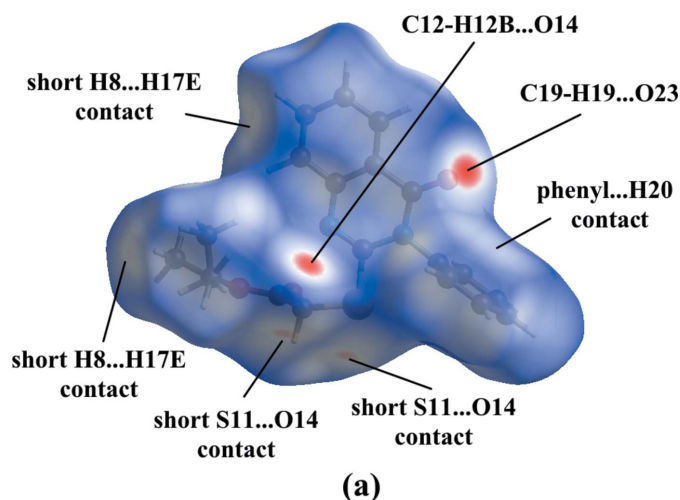


Figure 6
The Hirshfeld surface of (4) mapped over d_{norm} for the title compound in the range -0.2419 to 1.2857 a.u.

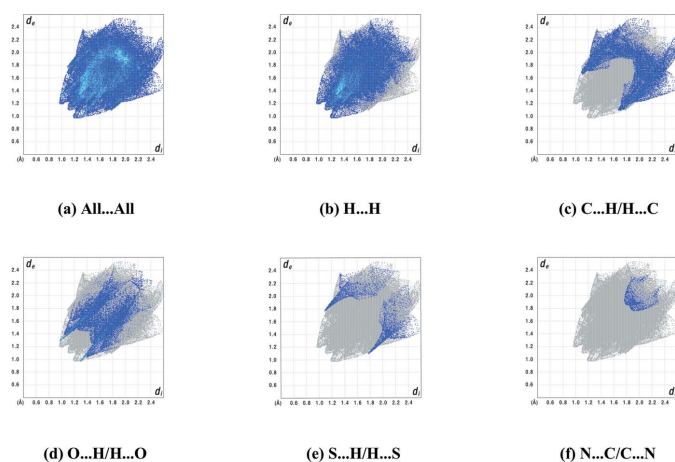


Figure 7

Full two-dimensional fingerprint plots for the title compound, showing (a) all interactions, and delineated into (b) H...H, (c) C...H/H...C, (d) O...H/H...O, (e) S...H/H...S and (f) N...C/C...N interactions. The d_i and d_e values are the closest internal and external distances (in Å) from a given point on the Hirshfeld surface.

H...C (21.5%) and O...H/H...O (18.7%) contacts. Smaller contributions are from S...H/H...S (4.0%), N...C/C...N (1.6%), C...C (1.6%), C...S/S...C (1.4%), N...H/H...N (1.3%), S...O/O...S (1.0%), N...S/S...N (1.0%) and O...O contacts (0.1%).

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.41, update of November 2019; Groom *et al.*, 2016) for 4-oxo-3,4-dihydroquinazoline gave 645 hits, of which 141 have a phenyl group at position N3 and 27 have a sulfur atom at position C2. A combination of both substitutions (without a link between the two) results in a set of 10 hits, which was used for further analysis. The dihedral angle between the least-squares planes through the quinazoline and phenyl rings varies between 71.99° (CSD refcode MUDGID; Saeed *et al.*, 2014) and 86.46° (CSD refcode GUWDIM; Rimaz *et al.*, 2009) with an average of 81.63°. The dihedral angle does not depend on eventual *ortho* substitution of the phenyl ring, as illustrated by the structures MUDGID (71.99°) and MUDNAC (85.90°; Saeed *et al.*, 2014), which both have an *o*-toluidine substituent at position N3. The almost perpendicular mutual orientation of both rings is also observed for the title compound.

5. Synthesis and crystallization

Anthranilic acid, phenyl isothiocyanate and ethyl chloroacetate were purchased from Acros and used without purification. Melting points were measured in open capillary tubes on a Gallenkamp melting point apparatus. IR spectra (ν , cm^{-1}) were recorded on FTIR-8400S-SHIMADZU spectrometer using KBr pellets. The NMR spectra were recorded on a Bruker Avance III spectrometer (500 MHz for ^1H NMR) using residual solvent DMSO- d_6 signals as internal reference.

The spin-spin coupling constants (J) are given in Hz. Peak multiplicity is reported as *s* (singlet), *d* (doublet), *dd* (doublet-doublet), *t* (triplet), *q* (quartet), *m* (multiplet). The synthetic protocol for title compound (**4**) is shown in Fig. 1 (Nguyen *et al.*, 2019).

Synthesis of 2-mercapto-3-phenylquinazolin-4-one (**3**):

Phenyl isothiocyanate (**2**) (0.1 mol) was added to the solution of anthranilic acid (**1**) (0.1 mol) and triethylamine (3.0 mL) in absolute ethanol (200 mL). The reaction mixture then was refluxed for 4 h. After cooling to room temperature, the reaction mixture was poured into cold water. The resulting solid was filtered and recrystallized from a mixture of DMF and water, then washed with cold ethanol to give the product (**3**). M.p. 569 K; yield 80%. IR (KBr, cm^{-1}): 3217, 3134 (N—H), 3028 (C—H aromatic), 1659 (C=O), 1618, 1524, 1485 (C=N, C=C aromatic). ^1H NMR [Bruker XL-500, 500 MHz, d_6 -DMSO, δ (ppm), J (Hz)]: 13.05 (*s*, 1H, H^{2a}), 7.96 (*d*, 1H, $^3J = 8.0$ Hz, H^5), 7.80 (*dd*, 1H, $^3J_1 = ^3J_2 = 8.0$ Hz, H^7), 7.50–7.40 (*m*, 3H, $\text{H}^{8,3c,3e}$), 7.42 (*dd*, 1H, $^3J_1 = ^3J_2 = 7.5$ Hz, H^6), 7.36 (*dd*, 1H, $^3J_1 = ^3J_2 = 7.5$ Hz, H^{3d}), 7.29 (*d*, 2H, $^3J = 7.5$ Hz, $\text{H}^{3b,3f}$).

Synthesis of ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl) sulfanyl]acetate (**4**):

A mixture of (**3**) (20 mmol) and anhydrous potassium carbonate (20 mmol) in dry DMF (30 mL) was stirred for 30 min, ethyl chloroacetate (20 mmol) was then added. After refluxing for 5 h, the reaction mixture was cooled to room temperature and poured into ice-cold water. The white precipitate was filtered off and recrystallized from ethanol to afford crystals of (**4**). Colourless crystals, m.p. 485 K, yield 65%. IR (KBr, cm^{-1}): 3059 (C—H aromatic), 2976, 2906 (C—H aliphatic), 1732 (C=O ester), 1680 (C=O ketone), 1607, 1598, 1468 (C=N, C=C aromatic). ^1H NMR [Bruker XL-500, 500 MHz, d_6 -DMSO, δ (ppm), J (Hz)]: 8.09 (*d*, 1H, $^3J = 8.0$ Hz, H^5), 7.84 (*d*, 1H, $^3J = 7.5$ Hz, H^8), 7.61–7.48 (*m*, 7H, $\text{H}^{6,7,3b,3c,3d,3e,3f}$), 4.15 (*q*, 2H, $^3J = 7.0$ Hz, H^{2c}), 3.99 (*s*, 2H, H^{2a}), 1.23 (*t*, 3H, $^3J = 7.0$ Hz, H^{2d}).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The methyl group C17 is disordered over two positions with population parameters 0.531 (13) and 0.469 (13)]. The H atoms were placed in idealized positions and included as riding contributions with $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}$ or $1.5U_{\text{eq}}$ of the parent atoms, with C—H distances of 0.93 (aromatic), 0.97 (CH_2) and 0.96 Å (CH_3). In the final cycles of refinement, two outliers were omitted.

Funding information

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Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₈ H ₁₆ N ₂ O ₃ S
<i>M_r</i>	340.39
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.8865 (6), 5.1298 (3), 28.2942 (14)
β (°)	93.667 (4)
<i>V</i> (Å ³)	1721.72 (16)
<i>Z</i>	4
Radiation type	Mo Kα
μ (mm ⁻¹)	0.21
Crystal size (mm)	0.5 × 0.15 × 0.15
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, single source at offset/far, Eos
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2018)
<i>T_{min}</i> , <i>T_{max}</i>	0.715, 1.000
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	18522, 3533, 2875
<i>R_{int}</i>	0.024
(sin θ/λ) _{max} (Å ⁻¹)	0.625
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.043, 0.111, 1.08
No. of reflections	3533
No. of parameters	229
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.15, -0.22

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b) and *OLEX2* (Dolomanov *et al.*, 2009).

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supporting information

Acta Cryst. (2020). E76, 668-672 [https://doi.org/10.1107/S2056989020005071]

Synthesis and structure of ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl)sulfanyl]acetate

Cong Nguyen Tien, Quang Nguyen Tan, Dung Pham Duc, Phuong Tran Hoang, Dat Nguyen Dang, Luong Truong Minh and Luc Van Meervelt

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018); cell refinement: *CrysAlis PRO* (Rigaku OD, 2018); data reduction: *CrysAlis PRO* (Rigaku OD, 2018); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009); software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009).

Ethyl 2-[(4-oxo-3-phenyl-3,4-dihydroquinazolin-2-yl)sulfanyl]acetate

Crystal data

$C_{18}H_{16}N_2O_3S$

$M_r = 340.39$

Monoclinic, $P2_1/n$

$a = 11.8865$ (6) Å

$b = 5.1298$ (3) Å

$c = 28.2942$ (14) Å

$\beta = 93.667$ (4)°

$V = 1721.72$ (16) Å³

$Z = 4$

$F(000) = 712$

$D_x = 1.313$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7343 reflections

$\theta = 2.9$ – 26.9 °

$\mu = 0.21$ mm⁻¹

$T = 293$ K

Needle, colourless

$0.5 \times 0.15 \times 0.15$ mm

Data collection

Rigaku Oxford Diffraction SuperNova, Single source at offset/far, Eos diffractometer

Radiation source: micro-focus sealed X-ray tube, SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 15.9631 pixels mm⁻¹

ω scans

Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)

$T_{\min} = 0.715$, $T_{\max} = 1.000$

18522 measured reflections

3533 independent reflections

2875 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 2.7$ °

$h = -14 \rightarrow 14$

$k = -6 \rightarrow 6$

$l = -35 \rightarrow 35$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.111$

$S = 1.08$

3533 reflections

229 parameters

0 restraints

Primary atom site location: dual

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.5306P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.14 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.68674 (11)	0.7908 (3)	0.38689 (5)	0.0521 (4)	
C2	0.58088 (13)	0.8340 (3)	0.37790 (6)	0.0453 (4)	
N3	0.51456 (10)	0.7160 (3)	0.34201 (5)	0.0438 (3)	
C4	0.55985 (14)	0.5351 (3)	0.31132 (6)	0.0456 (4)	
C5	0.67846 (13)	0.4761 (3)	0.32214 (6)	0.0450 (4)	
C6	0.73246 (16)	0.2867 (4)	0.29607 (7)	0.0577 (5)	
H6	0.692325	0.197199	0.271885	0.069*	
C7	0.84377 (17)	0.2334 (4)	0.30609 (8)	0.0702 (6)	
H7	0.879740	0.107617	0.288799	0.084*	
C8	0.90293 (17)	0.3672 (5)	0.34210 (9)	0.0794 (7)	
H8	0.979020	0.331142	0.348609	0.095*	
C9	0.85182 (16)	0.5518 (5)	0.36837 (8)	0.0709 (6)	
H9	0.893080	0.639601	0.392451	0.085*	
C10	0.73754 (14)	0.6078 (4)	0.35893 (6)	0.0489 (4)	
S11	0.50921 (4)	1.05531 (10)	0.41245 (2)	0.06194 (18)	
C12	0.62397 (16)	1.1534 (4)	0.45253 (7)	0.0564 (5)	
H12A	0.686170	1.209133	0.434376	0.068*	
H12B	0.600486	1.301892	0.470701	0.068*	
C13	0.66465 (17)	0.9421 (4)	0.48617 (7)	0.0563 (5)	
O14	0.61472 (13)	0.7513 (3)	0.49620 (5)	0.0728 (4)	
O15	0.76681 (14)	1.0046 (3)	0.50483 (6)	0.0907 (5)	
C16	0.8156 (3)	0.8266 (9)	0.54064 (14)	0.1386 (14)	
H16A	0.856216	0.924744	0.565576	0.166*	0.531 (13)
H16B	0.756086	0.729319	0.554614	0.166*	0.531 (13)
H16C	0.780435	0.857679	0.570144	0.166*	0.469 (13)
H16D	0.798109	0.649159	0.530875	0.166*	0.469 (13)
C17A	0.8872 (9)	0.6589 (19)	0.5203 (4)	0.129 (4)	0.531 (13)
H17A	0.844520	0.538073	0.500469	0.194*	0.531 (13)
H17B	0.930264	0.565109	0.544649	0.194*	0.531 (13)
H17C	0.937310	0.755551	0.501607	0.194*	0.531 (13)
C17B	0.9293 (6)	0.850 (3)	0.5485 (4)	0.152 (7)	0.469 (13)
H17D	0.964924	0.820026	0.519528	0.228*	0.469 (13)
H17E	0.955908	0.724381	0.571772	0.228*	0.469 (13)
H17F	0.947221	1.022473	0.559888	0.228*	0.469 (13)
C18	0.39382 (13)	0.7640 (3)	0.33627 (6)	0.0454 (4)	
C19	0.35129 (17)	0.9418 (4)	0.30424 (7)	0.0620 (5)	

H19	0.399266	1.040013	0.286589	0.074*
C20	0.23496 (18)	0.9749 (5)	0.29822 (9)	0.0763 (6)
H20	0.205352	1.096917	0.276528	0.092*
C21	0.16481 (17)	0.8324 (5)	0.32340 (9)	0.0773 (6)
H21	0.087168	0.852870	0.318649	0.093*
C22	0.20844 (17)	0.6590 (6)	0.35575 (11)	0.0981 (9)
H22	0.160210	0.563733	0.373781	0.118*
O23	0.50149 (10)	0.4422 (3)	0.27855 (5)	0.0628 (4)
C23	0.32353 (16)	0.6215 (5)	0.36235 (9)	0.0804 (7)
H23	0.352638	0.500489	0.384337	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0442 (8)	0.0581 (9)	0.0531 (9)	0.0025 (7)	-0.0027 (6)	-0.0094 (7)
C2	0.0450 (9)	0.0470 (9)	0.0436 (9)	0.0019 (7)	0.0000 (7)	-0.0009 (7)
N3	0.0390 (7)	0.0495 (8)	0.0429 (7)	-0.0008 (6)	0.0014 (6)	-0.0007 (6)
C4	0.0448 (9)	0.0500 (10)	0.0426 (9)	-0.0079 (7)	0.0059 (7)	-0.0003 (7)
C5	0.0436 (8)	0.0490 (9)	0.0432 (9)	-0.0020 (7)	0.0087 (7)	0.0011 (7)
C6	0.0592 (11)	0.0621 (12)	0.0530 (10)	0.0015 (9)	0.0133 (9)	-0.0057 (9)
C7	0.0640 (12)	0.0763 (14)	0.0722 (14)	0.0156 (11)	0.0198 (10)	-0.0067 (11)
C8	0.0448 (10)	0.1025 (18)	0.0911 (16)	0.0192 (11)	0.0054 (10)	-0.0125 (14)
C9	0.0454 (10)	0.0877 (15)	0.0784 (14)	0.0082 (10)	-0.0050 (9)	-0.0172 (12)
C10	0.0415 (9)	0.0551 (10)	0.0503 (10)	0.0012 (8)	0.0051 (7)	-0.0006 (8)
S11	0.0562 (3)	0.0648 (3)	0.0638 (3)	0.0166 (2)	-0.0040 (2)	-0.0173 (2)
C12	0.0662 (11)	0.0442 (10)	0.0583 (11)	0.0019 (9)	-0.0007 (9)	-0.0083 (8)
C13	0.0682 (12)	0.0521 (11)	0.0486 (10)	0.0027 (9)	0.0039 (9)	-0.0064 (9)
O14	0.0954 (11)	0.0510 (8)	0.0736 (10)	-0.0013 (8)	0.0189 (8)	-0.0009 (7)
O15	0.0849 (11)	0.0986 (12)	0.0845 (11)	-0.0084 (9)	-0.0259 (9)	0.0233 (10)
C16	0.130 (3)	0.164 (3)	0.115 (3)	0.017 (3)	-0.037 (2)	0.058 (3)
C17A	0.131 (7)	0.112 (6)	0.143 (7)	0.033 (5)	0.006 (5)	0.020 (5)
C17B	0.093 (5)	0.221 (15)	0.137 (9)	0.000 (6)	-0.030 (5)	0.083 (10)
C18	0.0390 (8)	0.0472 (9)	0.0495 (9)	0.0018 (7)	-0.0019 (7)	0.0005 (7)
C19	0.0591 (11)	0.0629 (12)	0.0625 (12)	-0.0047 (9)	-0.0084 (9)	0.0135 (10)
C20	0.0663 (13)	0.0759 (14)	0.0830 (15)	0.0141 (11)	-0.0229 (12)	0.0171 (12)
C21	0.0445 (10)	0.0862 (16)	0.0997 (17)	0.0125 (11)	-0.0074 (11)	0.0044 (14)
C22	0.0431 (11)	0.113 (2)	0.139 (2)	0.0074 (12)	0.0148 (13)	0.0576 (19)
O23	0.0525 (7)	0.0796 (9)	0.0558 (8)	-0.0115 (7)	0.0002 (6)	-0.0174 (7)
C23	0.0441 (10)	0.0888 (16)	0.1085 (18)	0.0097 (10)	0.0063 (11)	0.0494 (14)

Geometric parameters (Å, °)

N1—C2	1.287 (2)	S11—C12	1.7896 (19)
N1—C10	1.390 (2)	C12—H12A	0.9700
C17Aa—H17A	0.9600	C12—H12B	0.9700
C17Aa—H17B	0.9600	C12—C13	1.502 (3)
C17Aa—H17C	0.9600	C13—O14	1.188 (2)
C17Bb—H17D	0.9600	C13—O15	1.332 (2)

C17Bb—H17E	0.9600	O15—C16	1.456 (3)
C17Bb—H17F	0.9600	C16—H16A	0.9700
C2—N3	1.384 (2)	C16—H16B	0.9700
C2—S11	1.7541 (17)	C16—H16C	0.9700
N3—C4	1.402 (2)	C16—H16D	0.9700
N3—C18	1.4550 (19)	C16—C17A	1.363 (8)
C4—C5	1.455 (2)	C16—C17B	1.361 (9)
C4—O23	1.219 (2)	C18—C19	1.360 (2)
C5—C6	1.400 (2)	C18—C23	1.362 (3)
C5—C10	1.393 (2)	C19—H19	0.9300
C6—H6	0.9300	C19—C20	1.393 (3)
C6—C7	1.363 (3)	C20—H20	0.9300
C7—H7	0.9300	C20—C21	1.346 (3)
C7—C8	1.383 (3)	C21—H21	0.9300
C8—H8	0.9300	C21—C22	1.356 (3)
C8—C9	1.370 (3)	C22—H22	0.9300
C9—H9	0.9300	C22—C23	1.382 (3)
C9—C10	1.397 (2)	C23—H23	0.9300
C2—N1—C10	117.28 (15)	H16Aa—C16—H16B	108.2
N1—C2—N3	124.99 (15)	C17Bb—C16—H16C	108.8
N1—C2—S11	120.31 (13)	C17Bb—C16—H16D	108.8
H17Aa—C17Aa—H17B	109.5	H16Cb—C16—H16D	107.7
H17Aa—C17Aa—H17C	109.5	H12A—C12—H12B	107.7
N3—C2—S11	114.70 (11)	C13—C12—S11	113.59 (13)
C2—N3—C4	121.38 (13)	C13—C12—H12A	108.8
C2—N3—C18	121.27 (13)	C13—C12—H12B	108.8
C4—N3—C18	117.26 (13)	O14—C13—C12	126.89 (19)
N3—C4—C5	114.37 (14)	O14—C13—O15	124.07 (19)
O23—C4—N3	120.51 (15)	O15—C13—C12	109.01 (17)
H17Ba—C17Aa—H17C	109.5	C13—O15—C16	115.9 (2)
H17Db—C17Bb—H17E	109.5	O15—C16—H16A	109.8
H17Db—C17Bb—H17F	109.5	O15—C16—H16B	109.8
H17Eb—C17Bb—H17F	109.5	O15—C16—H16C	108.8
O23—C4—C5	125.12 (16)	O15—C16—H16D	108.8
C6—C5—C4	120.27 (16)	C16—C17Aa—H17A	109.5
C10—C5—C4	119.46 (15)	C19—C18—N3	120.65 (16)
C10—C5—C6	120.27 (16)	C16—C17Aa—H17B	109.5
C5—C6—H6	120.0	C19—C18—C23	120.39 (17)
C7—C6—C5	120.09 (19)	C23—C18—N3	118.92 (15)
C7—C6—H6	120.0	C16—C17Aa—H17C	109.5
C6—C7—H7	120.2	C16—C17Bb—H17D	109.5
C6—C7—C8	119.64 (19)	C18—C19—H19	120.4
C8—C7—H7	120.2	C18—C19—C20	119.14 (19)
C7—C8—H8	119.3	C16—C17Bb—H17E	109.5
C9—C8—C7	121.39 (19)	C16—C17Bb—H17F	109.5
C9—C8—H8	119.3	C20—C19—H19	120.4
C8—C9—H9	120.1	C19—C20—H20	119.6

C8—C9—C10	119.9 (2)	C21—C20—C19	120.9 (2)
C10—C9—H9	120.1	C21—C20—H20	119.6
N1—C10—C5	122.43 (15)	C20—C21—H21	120.3
N1—C10—C9	118.83 (17)	C20—C21—C22	119.37 (19)
C17Aa—C16—O15	109.5 (5)	C22—C21—H21	120.3
C5—C10—C9	118.73 (17)	C21—C22—H22	119.5
C2—S11—C12	99.06 (8)	C21—C22—C23	121.0 (2)
C17Bb—C16—O15	113.9 (5)	C23—C22—H22	119.5
S11—C12—H12A	108.8	C18—C23—C22	119.24 (19)
C17Aa—C16—H16A	109.8	C18—C23—H23	120.4
S11—C12—H12B	108.8	C22—C23—H23	120.4
C17Aa—C16—H16B	109.8		
N1—C2—N3—C4	-0.6 (3)	C7—C8—C9—C10	0.0 (4)
N1—C2—N3—C18	176.02 (16)	C8—C9—C10—N1	178.3 (2)
N1—C2—S11—C12	0.18 (17)	C8—C9—C10—C5	-1.0 (3)
C2—N1—C10—C5	0.7 (3)	C10—N1—C2—N3	-1.3 (3)
C2—N1—C10—C9	-178.59 (18)	C10—N1—C2—S11	178.30 (13)
C2—N3—C4—C5	2.9 (2)	C10—C5—C6—C7	-1.1 (3)
C2—N3—C4—O23	-176.56 (16)	S11—C2—N3—C4	179.77 (12)
C2—N3—C18—C19	97.9 (2)	S11—C2—N3—C18	-3.6 (2)
C2—N3—C18—C23	-84.4 (2)	S11—C12—C13—O14	-19.1 (3)
C2—S11—C12—C13	-68.94 (15)	S11—C12—C13—O15	162.70 (14)
N3—C2—S11—C12	179.82 (13)	C12—C13—O15—C16	176.3 (3)
N3—C4—C5—C6	176.06 (15)	O14—C13—O15—C16	-1.9 (4)
N3—C4—C5—C10	-3.3 (2)	C13—O15—C16—C17Bb	161.5 (9)
N3—C18—C19—C20	177.27 (18)	C13—O15—C16—C17Aa	97.7 (7)
N3—C18—C23—C22	-177.5 (2)	C18—N3—C4—C5	-173.88 (14)
C4—N3—C18—C19	-85.4 (2)	C18—N3—C4—O23	6.7 (2)
C4—N3—C18—C23	92.4 (2)	C18—C19—C20—C21	-0.4 (4)
C4—C5—C6—C7	179.52 (18)	C19—C18—C23—C22	0.3 (4)
C4—C5—C10—N1	1.7 (3)	C19—C20—C21—C22	1.5 (4)
C4—C5—C10—C9	-179.01 (18)	C20—C21—C22—C23	-1.7 (5)
C5—C6—C7—C8	0.0 (3)	C21—C22—C23—C18	0.8 (5)
C6—C5—C10—N1	-177.70 (16)	O23—C4—C5—C6	-4.5 (3)
C6—C5—C10—C9	1.6 (3)	O23—C4—C5—C10	176.07 (17)
C6—C7—C8—C9	0.6 (4)	C23—C18—C19—C20	-0.5 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C7—H7 \cdots O23 ⁱ	0.93	2.59	3.452 (3)	155
C12—H12B \cdots O14 ⁱⁱ	0.97	2.42	3.311 (3)	153
C19—H19 \cdots O23 ⁱⁱ	0.93	2.41	3.236 (2)	148

Symmetry codes: (i) $-x+3/2, y-1/2, -z+1/2$; (ii) $x, y+1, z$.