Crystal structure and Hirshfeld surface analysis of 3-methyl-4-oxo-\(N\)-phenyl-3,4-dihydroquinazoline-2-carbothioamide

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The asymmetric unit of the title compound, \(\text{C}_{16}\text{H}_{13}\text{N}_{3}\text{O}_{3}\text{S}\), comprises two molecules (A and B) with similar conformations that differ mainly in the orientation of the phenyl group relative to the rest of the molecule, as expressed by the \(\text{C}_{\text{thioamide}}-\text{N}_{\text{thioamide}}-\text{C}_{\text{phenyl}}-\text{C}_{\text{phenyl}}\) torsion angle of 49.3 (3)\(^\circ\) for molecule A and of 5.4 (3)\(^\circ\) for molecule B. In the crystal, two intermolecular N--H\(\cdots\)N hydrogen bonds lead to the formation of a dimer with \(R_{2}^{2}(10)\) graph-set notation. A Hirshfeld surface analysis revealed that H\(\cdots\)H interactions are the most important intermolecular interactions, contributing 40.9\% to the Hirshfeld surface.

1. Chemical context

Thioamides and their derivatives are important representatives of organic compounds containing a sulfur atom. The presence of bifunctional properties in thioamides, resulting from the presence of nitrogen and sulfur atoms, and their participation in reactions as electrophilic or nucleophilic reagents can lead to the formation of different heterocyclic compounds. Several review articles have been published on the syntheses, physico-chemical properties and applications of thioamides (Jagodziński, 2003; Belskaya et al., 2010; Koketsu & Ishihara, 2007; Krayushkin et al., 2004; Britsun et al., 2008).

One of the methods of choice for the synthesis of widely used thioamides is the Wilgerodt–Kindler reaction. As shown by previous studies, the Wilgerodt–Kindler reactions with 2-methylquinazoline-4-one went to the active methyl group in the position 2 and, accordingly, thioamides were synthesized in a series of quinazoline derivatives (Shakhidoyatov et al., 1997). Continuing our work in this direction, we have synthesized 2,3-dimethylquinazoline-4-one and studied the corresponding Wilgerodt–Kindler reactions.

During the reaction involving 2,3-dimethylquinazoline-4-one, sulfur, aniline, the solvent dimethyl sulfoxide and the catalyst sodium sulfide, the reaction went to the active methyl group in position 2 and new thioamides of a number of derivatives of quinazoline-4-one were obtained. The synthesis and crystal structure of 3-methyl-4-oxo-\(N\)-phenyl-3,4-dihydroquinazoline-2-carbothioamide, \(\text{C}_{16}\text{H}_{13}\text{N}_{3}\text{O}_{3}\text{S}\), is reported here. Relevant intermolecular contacts were quantified by using Hirshfeld surface analysis.
2. Structural commentary

The title compound crystallizes with two molecules, A and B, in the asymmetric unit (Fig. 1). In molecules A and B the orientations of the quinazoline ring system and the phenyl ring relative to the thioamide group differ, as shown by the values of the N3—C2—C10—S1 and C10—N11—C12—C13 torsion angles of 76.14 (19) and 49.3 (3)°, respectively, in molecule A and 83.78 (19) and 5.4 (3)°, respectively, in molecule B. As a result, there are differences in the intramolecular distances between the sulfur and hydrogen atoms in molecules A and B. In molecule A, the contacts S1A···H9AB and S1A···H13A are 2.873 and 2.897 Å whereas the corresponding distances in molecule B are 3.054 and 2.578 Å. The phenyl and pyrimidine rings in both molecules are essentially coplanar, with r.m.s. deviations of 0.0225 and 0.0119 Å for molecule A and B, respectively. Fig. 2 shows that the pyrimidine moieties of the molecules are almost superimposable.

3. Supramolecular features

In the crystal, molecules A and B form a dimer with an $R^2_2(10)$ ring motif through intermolecular N—H···N hydrogen bonds (Fig. 3, Table 1). In addition, molecule A interacts with molecule B by a C—H···π interaction (the C13A—H···Cg1 distance is 3.148 Å, Cg1 is the centroid of atoms C12B—C17B). Other weak C7A···H7A···O1B, C7A···H7A···O1B, C7B···H7B···O1A, C9A···H9AB···S1A and C13B···H13B···S1B hydrogen bonds link adjacent dimers, forming supramolecular layers expanding parallel to (010) (Fig. 4). The overall packing of molecules leads to the formation of narrow channels along
the $b$-axis direction, passing through nodes and the centre of the cell (Fig. 5).

4. Hirshfeld surface analysis
A Hirshfeld surface (HS) analysis (Spackman & Jayatilaka, 2009) was carried out using CrystalExplorer17.5 (Turner et al., 2017) to quantify and visualize intermolecular interactions in the crystal structure of the title compound. The HS mapped with $d_{\text{norm}}$ is represented in Fig. 6. The white surface indicates contacts with distances equal to the sum of van der Waals radii, and the red and blue colours indicate distances shorter or longer, respectively, than the van der Waals radii. The two-dimensional fingerprint plot for all contacts is depicted in Fig. 7a, and delineated into $\text{H}--\text{H}$, $\text{C}--\text{H}/\text{H}--\text{C}$, $\text{S}--\text{H}/\text{H}--\text{S}$, $\text{N}--\text{H}/\text{H}--\text{N}$, and $\text{O}--\text{H}/\text{H}--\text{O}$ contacts (Fig. 7b–f) whereby $\text{H}--\text{H}$ contacts are responsible for the largest contribution (40.9%) to the Hirshfeld surface. $\text{C}--\text{H}/\text{H}--\text{C}$ contribute 23.7%, $\text{S}--\text{H}/\text{H}--\text{S}$ contacts 10.7%, $\text{N}--\text{H}/\text{H}--\text{N}$ contacts 8.1% and $\text{O}--\text{H}/\text{H}--\text{O}$ contacts 7.0% to the total Hirshfeld surface. The contributions of further contacts are only minor and amount to $\text{C}--\text{C}$ (4.0%), $\text{S}--\text{C}/\text{C}--\text{S}$ (1.9%), $\text{N}--\text{C}/\text{C}--\text{N}$ (1.2%), $\text{S}--\text{C}/\text{C}--\text{S}$ (1.0%), $\text{O}--\text{N}$ (0.2%) and $\text{O}--\text{C}/\text{C}--\text{O}$ (0.1%).

5. Database survey
A search in the Cambridge Structural Database (CSD, version 5.41, update of January 2020; Groom et al., 2016) revealed six matches for molecules containing the 2,3-dimethylquinazolin-4(3$H$)-one moiety with a similar planar conformation as that in the title structure: AFOCIJ (Utayeva et al., 2013), HOCYED (Voitenko et al., 1999), MAHLOZ (Kotipalli et al., 2016), MUDHIE (Baglai et al., 2014), UTIDIM (Kundu et al., 2016) and XODZIB (Saifulukov et al., 2014). A search for the 2-methyl-N-phenylprop-2-enethioamide moiety gave six hits: ADEKUQ (Xiao & Jian, 2006), AGECIB (Skelton & Massi, 2018), GOFFOY (Li et al., 2014), GOXFUW (Li et al., 2016),...
6. Synthesis and crystallization

0.435 g (0.0025 mol) of 2,3-dimethylquinazoline-4-one, 0.465 g (0.005 mol) of aniline, 0.24 g (0.0075 mol) of sulfur, 0.05 g of sodium sulfide (Na₂S·9H₂O) and 4 ml of dimethyl sulfoxide were injected into a round-bottomed flask with a volume of 100 ml. Then the reaction flask was heated to 403 K for 6 h. After the end of the reaction, the flask was cooled and 40 ml of an aqueous sodium hydroxide solution were added. The resulting mixture was filtered, then added to a dilute solution of sulfuric acid (pH 6). The formed precipitate was filtered off and recrystallized in methanol. In total, 0.5 g (64.0%) of the product were obtained, m.p. 481–483 K.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were positioned geometrically, with C—H = 0.96 Å (for methylene H atoms) and C—H = 0.93 Å (for aromatic H atoms), and were refined with U( eq(H)) = 1.5U(eq(Cmethyl)) and 1.2U(eq(C)), respectively. H atoms bonded to nitrogen were located in a difference-Fourier map, and their positional and isotropic displacement parameters were freely refined.

Acknowledgements

The authors are grateful to the Institute of Bioorganic Chemistry, Academy Sciences of Uzbekistan, for providing laboratory facilities.

Funding information

The chemical part of the work was financially supported by a Georg Forster Research Fellowship for Experienced Researchers of the Alexander von Humboldt Foundation (AvH) to BE (UZB 1186936 GF-E).

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Table 2

| Crystal data | Chemical formula | C₆H₁₇N₃O₅S |
|--------------|-----------------|-------------|
| Crystal system, space group | Monoclinic, P2₁/n |
| Temperature (K) | 566 |
| a, b, c (Å) | 11.7685 (3), 16.3641 (3), 16.3798 (3) |
| β (°) | 110.646 (2) |
| V (Å³) | 2951.85 (11) |
| Z | 8 |
| Radiation type | Cu Kα |
| μ (mm⁻¹) | 1.96 |
| Crystal size (mm) | 0.25 × 0.23 × 0.20 |

Data collection

Diffractometer: XtaLAB Synergy, Single source at home/near, HyPix3000
Absorption correction: Multi-scan (CrysAlis PRO: Rigaku OD, 2020)

| Tmin, Tmax | 0.639, 1.000 |
| No. of measured, independent and observed [F > 2σ(F)] reflections | 16801, 5685, 4788 |
| R(int) | 0.022 |
| wR2 | 0.615 |

Refinement

| R[F² > 2σ(F²)], wR(F²), S | 0.042, 0.121, 1.06 |
| No. of reflections | 5685 |
| No. of parameters | 390 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.32, -0.43 |

Computer programs: CrysAlis PRO (Rigaku OD, 2020), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), PLATON (Spek, 2020), Mercury (Macrae et al., 2020) and pubICIF (Westrip, 2010).

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Computing details

Data collection: CrysAlis PRO (Rigaku OD, 2020); cell refinement: CrysAlis PRO (Rigaku OD, 2020); data reduction: CrysAlis PRO (Rigaku OD, 2020); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: PLATON (Spek, 2020), Mercury (Macrae et al., 2020); software used to prepare material for publication: publCIF (Westrip, 2010).

3-Methyl-4-oxo-N-phenyl-3,4-dihydroquinazoline-2-carbothioamide

Crystal data

C₁₆H₁₃N₃OS  
Mr = 295.35
Monoclinic, P2₁/n  
a = 11.7685 (3) Å
b = 16.3641 (3) Å
c = 16.3798 (3) Å
β = 110.646 (2)°
V = 2951.85 (11) Å³
Z = 8

F(000) = 1232  
Dₐ = 1.329 Mg m⁻³  
Cu Kα radiation, λ = 1.54184 Å

Cell parameters from 9141 reflections
θ = 2.7–71.1°
µ = 1.96 mm⁻¹
T = 566 K
Prismatic, yellow
0.25 × 0.23 × 0.20 mm

Data collection

XtaLAB Synergy, Single source at home/near, HyPix3000 diffractometer
Radiation source: micro-focus sealed X-ray tube
Detector resolution: 10.00000 pixels mm⁻¹
ω scans
Absorption correction: multi-scan
(CrysAlisPro; Rigaku OD, 2020)
Tmin = 0.639, Tmax = 1.000

16801 measured reflections
5685 independent reflections
4788 reflections with I > 2σ(I)

Refinement

Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.042
wR(F²) = 0.121
S = 1.06
5685 reflections
390 parameters

0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

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[https://doi.org/10.1107/S2056989021013116]
$w = \frac{1}{\sigma^2(F_o^2) + (0.0602P)^2 + 0.6163P}$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta\sigma)_{\text{max}} = 0.001$
$\Delta \rho_{\text{max}} = 0.32 \text{ e} \cdot \text{Å}^{-3}$

$\Delta \rho_{\text{min}} = -0.42 \text{ e} \cdot \text{Å}^{-3}$

Extinction correction: SHELXL (Sheldrick, 2015a), $F_c^* = kF_c[1+0.001xF_c^2/\sin(2\theta)]^{1/4}$
Extinction coefficient: 0.00124 (12)

**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 (Å²)**

| Atom | x   | y   | z   | $U_{iso}$ or $U_{eq}$ |
|------|-----|-----|-----|-----------------------|
| S1A  | 0.46771 (5) | 0.62649 (4) | 0.89046 (3) | 0.07206 (19) |
| O1A  | 0.69766 (15) | 0.59324 (9)  | 0.65155 (11) | 0.0755 (4)  |
| N1A  | 0.48752 (12) | 0.75527 (8)  | 0.72159 (9)  | 0.0456 (3)  |
| N1B  | 0.22774 (13) | 0.73699 (9)  | 0.57212 (9)  | 0.0474 (3)  |
| S1B  | 0.04219 (5)  | 0.86597 (6)  | 0.63642 (4)  | 0.1030 (3)  |
| O1B  | 0.30121 (15) | 0.90703 (8)  | 0.41700 (10) | 0.0690 (4)  |
| C2A  | 0.49005 (14) | 0.67801 (10) | 0.73853 (10) | 0.0430 (4)  |
| S3A  | 0.55988 (13) | 0.62176 (8)  | 0.71623 (10) | 0.0487 (3)  |
| N3B  | 0.24924 (13) | 0.87402 (8)  | 0.53417 (9)  | 0.0461 (3)  |
| C4A  | 0.63788 (17) | 0.64470 (11) | 0.67217 (12) | 0.0523 (4)  |
| C4B  | 0.28038 (16) | 0.85368 (11) | 0.46179 (11) | 0.0476 (4)  |
| C4A' | 0.63881 (16) | 0.73161 (11) | 0.65427 (12) | 0.0488 (4)  |
| C4B' | 0.28702 (15) | 0.76640 (10) | 0.44719 (10) | 0.0441 (4)  |
| C5A  | 0.7150 (2)   | 0.76334 (13) | 0.61289 (15) | 0.0663 (5)  |
| H5A  | 0.767186     | 0.728822     | 0.597752     | 0.080*      |
| C5B  | 0.31931 (18) | 0.73805 (12) | 0.37776 (12) | 0.0572 (5)  |
| H5B  | 0.336243     | 0.775028     | 0.340470     | 0.069*      |
| C6A  | 0.7128 (2)   | 0.84521 (14) | 0.59469 (16) | 0.0719 (6)  |
| H6A  | 0.763410     | 0.866124     | 0.567158     | 0.086*      |
| C6B  | 0.3260 (2)   | 0.65590 (14) | 0.36481 (14) | 0.0699 (6)  |
| H6B  | 0.347755     | 0.637057     | 0.318813     | 0.084*      |
| C7A  | 0.6353 (2)   | 0.89685 (13) | 0.61719 (15) | 0.0669 (5)  |
| H7A  | 0.633301     | 0.952178     | 0.603750     | 0.080*      |
| C7B  | 0.3002 (2)   | 0.60060 (13) | 0.42011 (16) | 0.0738 (6)  |
| H7B  | 0.304581     | 0.544836     | 0.410662     | 0.089*      |
| C8A  | 0.56154 (18) | 0.86710 (11) | 0.65916 (14) | 0.0570 (5)  |
| H8A  | 0.510698     | 0.902433     | 0.674732     | 0.068*      |
| C8B  | 0.2684 (2)   | 0.62723 (12) | 0.48872 (14) | 0.0644 (5)  |
| H8B  | 0.251967     | 0.589664     | 0.525684     | 0.077*      |
| C8A' | 0.56261 (15) | 0.78398 (10) | 0.67853 (11) | 0.0443 (4)  |
| C8B' | 0.26094 (16) | 0.71106 (10) | 0.50270 (11) | 0.0455 (4)  |
| C9A  | 0.5550 (2)   | 0.53429 (12) | 0.73431 (16) | 0.0712 (6)  |
| H9AA | 0.543485     | 0.503622     | 0.682028     | 0.107*      |
| H9AB | 0.488631     | 0.524014     | 0.754066     | 0.107*      |
H9AC  0.629728  0.517988  0.778655  0.107*
C9B   0.2411 (2)  0.96167 (12)  0.55107 (14)  0.0673 (5)
H9BA  0.308740  0.989779   0.544197   0.101*
H9BB  0.242392  0.969168   0.609523   0.101*
H9BC  0.166757  0.983288   0.510518   0.101*
C10A  0.40897 (15)  0.64886 (10)  0.78644 (11)  0.0464 (4)
C10B  0.18531 (16)  0.84207 (12)  0.65864 (11)  0.0520 (4)
N11A  0.29256 (13)  0.64598 (9)   0.73505 (9)   0.0459 (3)
N11B  0.27899 (14)  0.84235 (9)   0.73404 (9)   0.0458 (3)
C12A  0.28641 (16)  0.86210 (10)  0.82016 (10)  0.0460 (4)
C12B  0.17430 (18)  0.65831 (12)  0.83199 (13)  0.0579 (5)
H12A  0.231162  0.694273   0.868105   0.070*
H12B  0.1889 (2)  0.88031 (13)  0.84467 (13)  0.0615 (5)
C13A  0.10617  0.63823 (14)  0.84934 (16)  0.0687 (6)
C13B  0.2088 (2)  0.89785 (15)  0.93166 (14)  0.0733 (6)
H13A  0.143062  0.910500   0.948369   0.088*
H13B  0.106664  0.910500   0.948369   0.088*
C15A  0.0373110   0.58550 (15)  0.79631 (16)  0.0728 (6)
C15B  0.03230 (2)  0.89684 (15)  0.99301 (13)  0.0743 (6)
H15A  0.0335113   0.909440   1.050847   0.089*
H15B  0.0350499 (18)  0.55107 (15)  0.72556 (15)  0.0713 (6)
H16A  0.04194 (2)  0.87709 (17)  0.96852 (14)  0.0799 (7)
H16B  0.497078   0.875101   1.010346   0.096*
C17A  0.10845 (17)  0.57004 (13)  0.70712 (12)  0.0580 (5)
C17B  0.121368   0.546455   0.659469   0.070*
H17A  0.40270 (19)  0.86009 (14)  0.88256 (12)  0.0637 (5)
H17B  0.468896   0.847365   0.866451   0.076*

|   | U^21   | U^22   | U^23   | U^52   | U^53   | U^23   |
|---|--------|--------|--------|--------|--------|--------|
|S1A| 0.0501 (3) | 0.1132 (5) | 0.0526 (3) | −0.0013 (3) | 0.0178 (2) | 0.0220 (3) |
|O1A| 0.0852 (10) | 0.0582 (8) | 0.1095 (12) | 0.0080 (7) | 0.0668 (10) | −0.0077 (8) |
|N1A| 0.0427 (7) | 0.0479 (8) | 0.0540 (8) | 0.0015 (6) | 0.0267 (6) | 0.0025 (6) |
|N1B| 0.0546 (8) | 0.0508 (8) | 0.0435 (7) | 0.0033 (6) | 0.0257 (6) | 0.0007 (6) |
|S1B| 0.0536 (3) | 0.1936 (8) | 0.0591 (3) | 0.0382 (4) | 0.0165 (3) | −0.0226 (4) |
|O1B| 0.0907 (10) | 0.0565 (8) | 0.0774 (9) | −0.0040 (7) | 0.0513 (8) | 0.0100 (7) |
|C2A| 0.0387 (8) | 0.0476 (9) | 0.0469 (8) | −0.0015 (6) | 0.0201 (7) | 0.0002 (7) |
|C2B| 0.0423 (9) | 0.0539 (10) | 0.0380 (8) | 0.0038 (7) | 0.0152 (7) | −0.0023 (7) |
|N3A| 0.0499 (8) | 0.0422 (7) | 0.0625 (9) | −0.0013 (6) | 0.0304 (7) | −0.0013 (6) |
|N3B| 0.0495 (8) | 0.0443 (7) | 0.0464 (7) | −0.0005 (6) | 0.0195 (6) | −0.0044 (6) |
|C4A| 0.0521 (10) | 0.0519 (10) | 0.0633 (11) | −0.0020 (8) | 0.0333 (9) | −0.0068 (8) |
### Geometric parameters (Å, °)

|                  | S1A—C10A | O1A—C4A | N1A—C2A | N1A—C8A' | N1B—C2B | N1B—C8B' | S1B—C10B | O1B—C4B | C2A—N3A | C2A—C10A | C2B—N3B | C2B—C10B | N3A—C4A | N3A—C9A |
|------------------|----------|---------|---------|----------|---------|----------|----------|---------|---------|---------|---------|---------|---------|---------|
|                  | 1.6385 (17) | 1.219 (2) | 1.292 (2) | 1.392 (2) | 1.293 (2) | 1.392 (2) | 1.6401 (18) | 1.220 (2) | 1.367 (2) | 1.511 (2) | 1.368 (2) | 1.510 (2) | 1.404 (2) | 1.467 (2) |

|                  | C8B—C8B' | C8B—H8B | C9A—H9AA | C9A—H9AB | C9A—H9AC | C9B—H9BA | C9B—H9BB | C9B—H9BC | C10A—N11A | C10B—N11B | N11A—C12A | N11B—C12B | N11B—H11A | N11B—H11B |
|------------------|----------|---------|----------|----------|----------|----------|----------|---------|----------|----------|----------|----------|----------|----------|
|                  | 1.399 (2) | 0.9300  | 0.9600   | 0.9600   | 0.9600   | 0.9600   | 0.9600   | 0.9600   | 1.332 (2) | 1.334 (2) | 1.430 (2) | 1.420 (2) | 0.88 (2)  | 0.87 (2)  |
| Bond Length (Å) | Angle (°) |
|----------------|-----------|
| N3B—C4B        | 1.399 (2) |
| N3B—C9B        | 1.470 (2) |
| C4A—C4A'       | 1.453 (3) |
| C4B—C4B'       | 1.455 (2) |
| C4A'—C8A'      | 1.396 (2) |
| C4A'—C5A       | 1.401 (2) |
| C4B'—C8B'      | 1.392 (2) |
| C4B'—C5B       | 1.399 (2) |
| C5A—C6A        | 1.371 (3) |
| C5A—H5A        | 0.9300    |
| C5B—C6B        | 1.367 (3) |
| C5B—H5B        | 0.9300    |
| C6A—C7A        | 1.385 (3) |
| C6A—H6A        | 0.9300    |
| C6B—C7B        | 1.387 (3) |
| C6B—H6B        | 0.9300    |
| C7A—C8A        | 1.373 (3) |
| C7A—H7A        | 0.9300    |
| C7B—C8B        | 1.375 (3) |
| C7B—H7B        | 0.9300    |
| C8A—C8A'       | 1.396 (2) |
| C8A—H8A        | 0.9300    |
| C2A—N1A—C8A'  | 117.91 (14) |
| C2B—N1B—C8B'  | 117.44 (14) |
| N1A—C2A—N3A    | 124.89 (14) |
| N1A—C2A—C10A   | 116.71 (14) |
| N3A—C2A—C10A   | 118.39 (14) |
| N1B—C2B—N3B    | 125.33 (14) |
| N1B—C2B—C10B   | 116.84 (15) |
| N3B—C2B—C10B   | 117.83 (15) |
| C2A—N3A—C4A    | 121.39 (14) |
| C2A—N3A—C9A    | 122.23 (15) |
| C4A—N3A—C9A    | 116.35 (15) |
| C2B—N3B—C4B    | 121.15 (14) |
| C2B—N3B—C9B    | 122.32 (15) |
| C4B—N3B—C9B    | 116.47 (15) |
| O1A—C4A—N3A    | 120.23 (17) |
| O1A—C4A—C4A'   | 125.05 (17) |
| N3A—C4A—C4A'   | 114.72 (14) |
| O1B—C4B—N3B    | 120.52 (16) |
| O1B—C4B—C4B'   | 124.74 (16) |
| N3B—C4B—C4B'   | 114.73 (14) |
| C8A'—C4A'—C5A  | 119.71 (17) |
| C8A'—C4A'—C4A  | 119.49 (15) |
| C5A—C4A'—C4A   | 120.80 (16) |
| C8B'—C4B'—C5B  | 120.06 (16) |
| C8B'—C4B'—C4B  | 119.62 (14) |
| Bond                  | Angle (°)       | Bond                  | Angle (°)       |
|----------------------|-----------------|----------------------|-----------------|
| C5B—C4B′—C4B         | 120.33 (16)     | C13B—C12B—C17B       | 119.80 (16)     |
| C6A—C5A—C4A'        | 120.07 (19)     | C13B—C12B—N11B       | 125.03 (17)     |
| C6A—C5A—H5A         | 120.0           | C17B—C12B—N11B       | 115.15 (16)     |
| C4A'—C5A—H5A        | 120.0           | C14A—C13A—C12A       | 119.0 (2)       |
| C6B—C5B—C4B'        | 119.92 (18)     | C14A—C13A—H13A       | 120.5           |
| C6B—C5B—H5B         | 120.0           | C12A—C13A—H13A       | 120.5           |
| C4B'—C5B—H5B        | 120.0           | C12B—C13B—C14B       | 119.2 (2)       |
| C5A—C6A—C7A         | 120.17 (18)     | C12B—C13B—H13B       | 120.4           |
| C5A—C6A—H6A         | 119.9           | C14B—C13B—H13B       | 120.4           |
| C7A—C6A—H6A         | 119.9           | C15A—C14A—C13A       | 121.0 (2)       |
| C5B—C6B—C7B         | 120.16 (18)     | C15A—C14A—H14A       | 119.5           |
| C5B—C6B—H6B         | 119.9           | C13A—C14A—H14A       | 119.5           |
| C7B—C6B—H6B         | 119.9           | C15B—C14B—C13B       | 121.2 (2)       |
| C8A—C7A—C6A         | 120.58 (19)     | C15B—C14B—H14B       | 119.4           |
| C8A—C7A—H7A         | 119.7           | C13B—C14B—H14B       | 119.4           |
| C6A—C7A—H7A         | 119.7           | C14A—C15A—C16A       | 119.72 (19)     |
| C8B—C7B—C6B         | 120.80 (19)     | C14A—C15A—H15A       | 120.1           |
| C8B—C7B—H7B         | 119.6           | C16A—C15A—H15A       | 120.1           |
| C6B—C7B—H7B         | 119.6           | C14B—C15B—C16B       | 119.32 (19)     |
| C7A—C8A—C8A'        | 120.18 (18)     | C14B—C15B—H15B       | 120.3           |
| C7A—C8A—H8A         | 119.9           | C16B—C15B—H15B       | 120.3           |
| C8A'—C8A—H8A        | 119.9           | C15A—C16A—C17A       | 120.3 (2)       |
| C7B—C8B—C8B'        | 119.73 (19)     | C15A—C16A—H16A       | 119.9           |
| C7B—C8B—H8B         | 120.1           | C17A—C16A—H16A       | 119.9           |
| C8B'—C8B—H8B        | 120.1           | C15B—C16B—C17B       | 120.8 (2)       |
| N1A—C8A'—C4A'       | 121.56 (15)     | C15B—C16B—H16B       | 119.6           |
| N1A—C8A'—C8A'       | 119.17 (15)     | C17B—C16B—H16B       | 119.6           |
| C4A'—C8A'—C8A       | 119.26 (16)     | C12A—C17A—C16A       | 119.35 (19)     |
| C4B'—C8B'—N1B        | 121.68 (15)     | C12A—C17A—H17A       | 120.3           |
| C4B'—C8B'—C8B       | 119.33 (16)     | C16A—C17A—H17A       | 120.3           |
| N1B—C8B'—C8B        | 118.99 (16)     | C16B—C17B—C12B       | 119.6 (2)       |
| N3A—C9A—H9AA        | 109.5           | C16B—C17B—H17B       | 120.2           |
| N3A—C9A—H9AB        | 109.5           | C12B—C17B—H17B       | 120.2           |
| C8A'—N1A—C2A—N3A    | −1.0 (3)        | C4A—C4A'—C8A'—C8A    | −178.35 (17)    |
| C8A'—N1A—C2A—C10A   | 179.14 (14)     | C7A—C8A—C8A'—C8A    | 179.63 (18)     |
| C8B'—N1B—C2B—N3B    | −1.0 (3)        | C7A—C8A—C8A'—C4A    | −0.4 (3)        |
| C8B'—N1B—C2B—C10B   | 179.33 (14)     | C5B—C4B'—C8B'—N1B   | −179.59 (16)    |
| N1A—C2A—N3A—C4A     | 0.6 (3)         | C4B—C4B'—C8B'—N1B   | 0.4 (3)         |
| C10A—C2A—N3A—C4A    | −179.57 (16)    | C5B—C4B'—C8B'—C8B   | 0.6 (3)         |
| N1A—C2A—N3A—C9A     | −177.51 (18)    | C4B—C4B'—C8B'—C8B   | −179.50 (18)    |
| C10A—C2A—N3A—C9A    | 2.4 (3)         | C2B—N1B—C8B'—C8B'   | −0.5 (2)        |
| N1B—C2B—N3B—C4B     | 2.7 (3)         | C2B—N1B—C8B'—C8B    | 179.35 (18)     |
| C10B—C2B—N3B—C4B    | −177.67 (15)    | C7B—C8B—C8B'—C4B'   | −0.6 (3)        |
| N1B—C2B—N3B—C9B     | 179.73 (17)     | C7B—C8B—C8B'—N1B    | 179.5 (2)       |
| C10B—C2B—N3B—C9B    | −0.6 (2)        | N1A—C2A—C10A—N11A   | 75.1 (2)        |
| C2A—N3A—C4A—O1A     | −178.66 (18)    | N3A—C2A—C10A—N11A   | −104.80 (18)    |
| C9A—N3A—C4A—O1A     | −0.5 (3)        | N1A—C2A—C10A—S1A    | −103.97 (17)    |
C2A—N3A—C4A—C4A' 0.9 (3) N3A—C2A—C10A—S1A 76.14 (19)
C9A—N3A—C4A—C4A' 179.10 (17) N1B—C2B—C10B—N11B 83.6 (2)
C2B—N3B—C4B—O1B 1.2 (3) N3B—C2B—C10B—N11B −96.12 (19)
C9B—N3B—C4B—O1B −2.6 (2) N3B—C2B—C10B—S1B 83.78 (19)
C2B—N3B—C4B—C4B' −179.78 (16) C2A—C10A—N11A—C12A −177.47 (15)
C9B—N3B—C4B—C4B' −2.6 (2) N3B—C2B—C10B—S1B 83.78 (19)
C2B—N3B—C4B—C4B' −2.6 (2) N3B—C2B—C10B—S1B 83.78 (19)
C9B—N3B—C4B—C4B' −179.78 (16) C2A—C10A—N11A—C12A −177.47 (15)
O1A—C4A—C4A'—C8A' 177.6 (2) S1A—C10A—N11A—C12A 1.5 (3)
N3A—C4A—C4A'—C8A' 1.9 (3) C2B—C10B—N11B—C12B −179.09 (17)
O1A—C4A—C4A'—C5A −2.2 (3) S1B—C10B—N11B—C12B 1.0 (3)
N3A—C4A—C4A'—C5A 178.19 (18) C10A—N11A—C12A—C17A 1.5 (3)
O1B—C4B—C4B'—C8B' −179.87 (18) C10A—N11A—C12A—C17A 1.5 (3)
N3B—C4B—C4B'—C8B' −2.2 (3) S1B—C10B—N11B—C12B 1.0 (3)
O1B—C4B—C4B'—C5B 0.1 (3) S1B—C10B—N11B—C12B 1.0 (3)
N3B—C4B—C4B'—C5B 178.91 (16) C17A—C12A—C13A—C14A 0.8 (3)
C8A'—C4A'—C5A—C6A −1.4 (3) N11A—C12A—C13A—C14A 176.03 (17)
C4A'—C4A'—C5A—C6A 178.5 (2) C17B—C12B—C13B—C14B 1.1 (3)
C8B'—C4B'—C5B—C6B −0.4 (3) N11B—C12B—C13B—C14B 179.36 (19)
C4B'—C4B'—C5B—C6B 179.66 (18) C12A—C13A—C14A—C15A 0.3 (3)
C4A'—C5A—C6A—C7A 0.1 (4) C12B—C13B—C14B—C15B 0.4 (4)
C4B'—C5B—C6B—C7B 0.3 (3) C13A—C14A—C15A—C16A 1.0 (3)
C5A—C6A—C7A—C8A 1.1 (4) C13B—C14B—C15B—C16B 0.9 (4)
C5B—C6B—C7B—C8B −0.3 (4) C14A—C15A—C16A—C17A 0.6 (3)
C6A—C7A—C8A—C8A' −0.9 (3) C14B—C15B—C16B—C17B 1.5 (4)
C6B—C7B—C8B—C8B' 0.5 (4) C13A—C12A—C17A—C16A 1.2 (3)
C2A—N1A—C8A—C4A' −0.1 (2) N11A—C12A—C17A—C16A −175.84 (17)
C2A—N1A—C8A—C8A 179.82 (16) C15A—C16A—C17A—C12A −0.4 (3)
C5A—C4A'—C8A'—N1A −187.49 (18) C15B—C16B—C17B—C12B −0.8 (4)
C4A'—C4A'—C8A'—N1A 1.6 (3) C13B—C12B—C17B—C16B −0.5 (3)
C5A—C4A'—C8A'—C8A 1.5 (3) N11B—C12B—C17B—C16B −179.0 (2)

Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H   | H···A   | D···A   | D—H···A |
|-----------|-------|--------|--------|---------|
| N11A—H11A···N1B | 0.88 (2) | 2.05 (2) | 2.913 (2) | 166.7 (18) |
| N11B—H11B···N1A | 0.87 (2) | 2.04 (2) | 2.907 (2) | 171.6 (19) |
| C9A—H9A···S1A | 0.96 | 2.87 | 3.424 (2) | 118 |
| C13B—H13B···S1B | 0.93 | 2.58 | 3.243 (3) | 129 |
| C7A—H7A···O1B | 0.93 | 2.49 | 3.386 (3) | 162 |
| C7B—H7B···O1Aii | 0.93 | 2.47 | 3.385 (3) | 166 |

Symmetry codes: (i) −x+1, −y+2, −z+1; (ii) −x+1, −y+1, −z+1.