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# Crystal structure, Hirshfeld surface analysis and energy framework study of 6-formyl-7,8,9,11-tetrahydro-5*H*-pyrido[2,1-*b*]quinazolin-11-one

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**Supporting information**: this article has supporting information at journals.iucr.org/e

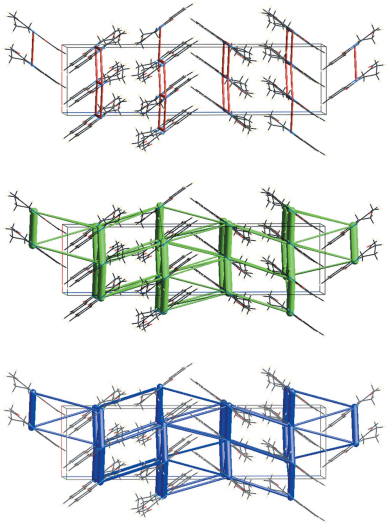
At 100 K, the title compound, $C_{13}H_{12}N_2O_2$, crystallizes in the orthorhombic space group $Pna2_1$ with two very similar molecules in the asymmetric unit. An intramolecular N—H···O hydrogen bond leads to an $S(6)$ graph-set motif in each of the molecules. Intermolecular $\pi$–$\pi$ stacking and C=O···$\pi$ interactions involving the aldehyde O atoms link molecules into stacks parallel to [100]. A Hirshfeld surface analysis indicates that the most important contributions to the crystal packing stem from H···H (49.4%) and H···O/O···H (21.5%) interactions. Energy framework calculations reveal a significant contribution of dispersion energy. The crystal studied was refined as a two-component inversion twin.

## 1. Chemical context

Two major aspects contribute to the interest in modified structural analogues of quinazoline alkaloids. On the one hand, they are attractive targets for the development of methods in organic synthesis; reactions sufficiently general to target a wide range of derivatives of a given lead structure should be easy to carry out and warrant high yields. On the other hand, substituted quinazolines allow the study of structure–property relationships with respect to their biological activities (Shakhidoyatov, 1988; Shakhidoyatov & Elmuradov, 2014).

The quinazoline alkaloid 7,8,9,11-tetrahydro-5*H*-pyrido[2,1-*b*]quinazolin-11-one (mackinazolinone alkaloid) was first isolated from the plant Mackinlaya subulata Philipson (Fitzgerald *et al.*, 1966). A simple method for the synthesis of mackinazolinone *via* condensation of anthranilic acid with $\delta$-valerolactam promoted the use of this compound as a synthon for chemical transformations (Shakhidoyatov *et al.*, 1976; Oripov *et al.*, 1979).

The title compound, 6-formyl-7,8,9,11-tetrahydro-5*H*-pyrido[2,1-*b*]quinazolin-11-one (**1**) (Fig. 1), does react with primary amines (Zhurakulov & Vinogradova, 2015, 2016), but does not react with pseudoephedrine or 1-(phenyl)-6,7-dimethoxy-1,2,3,4-tetrahydroisoquinoline in a range of solvents with different polarities such as acetonitrile, chloroform, ethanol, trifluoroacetic acid, acetic acid, benzene, DMF



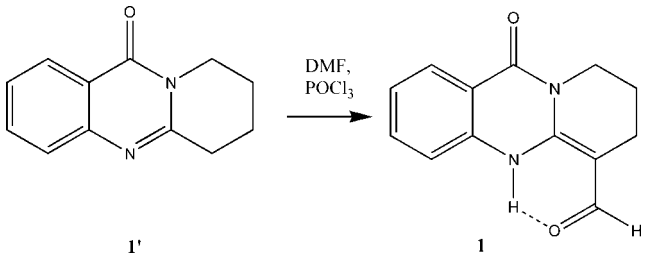
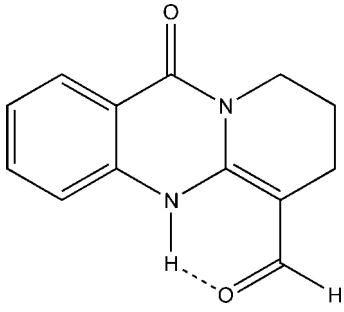



**Figure 1**
Chemical scheme showing the synthesis of the title compound.

or dioxane. The existence of several tautomeric forms for compound (**1**) may be the reason for this selectivity towards primary amines.

Based on $^1$H NMR data and quantum-chemical calculations, Zhurakulov *et al.* (2016) confirmed that the tautomer with the intramolecular hydrogen bond represents the energetically favourable form. In order to establish the tautomeric form of (**1**) in the solid state, we studied its molecular and crystal structure. We also report the analysis of the Hirshfeld surface and the energy framework of crystalline (**1**).




## 2. Structural commentary

The asymmetric unit of the title compound contains two molecules *A* and *B* (Fig. 2). They are almost superimposable, with an r.m.s. of 0.023 Å (Spek, 2020); an overlay of *A* and *B* is depicted in the supporting information (Fig. S1). In contrast to the quinazolinone moiety, the alkyl ring is not planar. The maximum deviation from the least-squares plane through each of the molecules is encountered for the atoms C2*A* and C2*B* and amounts to 0.515 (3) and 0.521 (3) Å, respectively. The almost coplanar arrangement of the aldehyde group and the pyrimidine ring in either molecule *A* and *B* enables an intramolecular N—H···O interaction (Table 1) and formation of an *S*(6) graph-set motif.

Molecules of (**1**) stack into columns parallel to [100] in an equidistant series of coplanar moieties; the independent molecules *A* and *B* segregate into different stacks (Fig. 3). The intra-stack arrangement does obviously not correspond to translation but involves the *a* glide plane with its mirror component along [010]. The carbonyl groups in subsequent molecules of a stack are therefore oriented alternately in the positive and negative direction of the crystallographic *b* axis, and the same arrangement can be expected for their dipole moments. Although no 'real' translation relates consecutive molecules along [100], the rather regular arrangement of essentially planar objects at half a lattice parameter is reflected in moderate pseudosymmetry in reciprocal space: reflection intensities $I_{hkl}$ are stronger for even indices *h* than for odd ones, with a ratio $I_{hkl}$, $h = 2n$: $I_{hkl}$, $h = (2n + 1)$ of 1.5.

Compound (**1**) crystallizes in the non-centrosymmetric achiral space group $Pna2_1$, and its absolute structure deserves a comment. The absolute structure is linked to the direction of the polar screw axis along [001]. In the absence of heavy atoms, resonant scattering in (**1**) is minor, with *Friedif* (Flack & Shmueli, 2007) of 28. We have recently investigated a case of similar low resonant scattering in a Sohnke group, where the absolute structure could be linked to the absolute configuration of the target molecule, and chemical and spectroscopic information could help (Wang & Englert, 2019). As might be expected, the commonly used indicators for diffraction-based assignment of the absolute structure of (**1**) were associated with rather large standard uncertainties: the Flack parameter (Flack, 1983) refined to 0.51 (7), and similar results were obtained for Parsons' quotient method [0.52 (5); Parsons *et al.*, 2013] and Hooft's Bayesian analysis [0.51 (5); Hooft *et al.*, 2010]. All of these indicators suggest that the specimen used for the diffraction experiment was a twin. Refinement converged for a volume ratio of 0.7 (2):0.3 (2) for the twin domains.

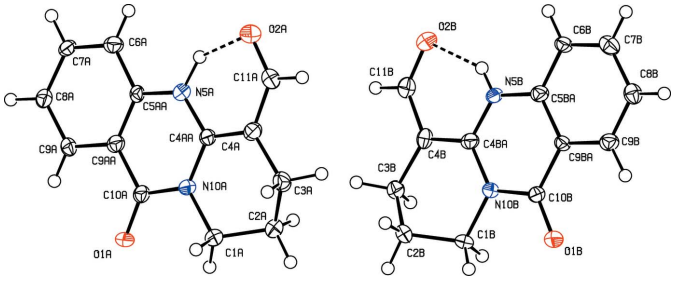



**Figure 2**
The asymmetric unit of (**1**) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular N—H···O hydrogen bond forming an *S*(6) ring motif is shown with dashed lines.

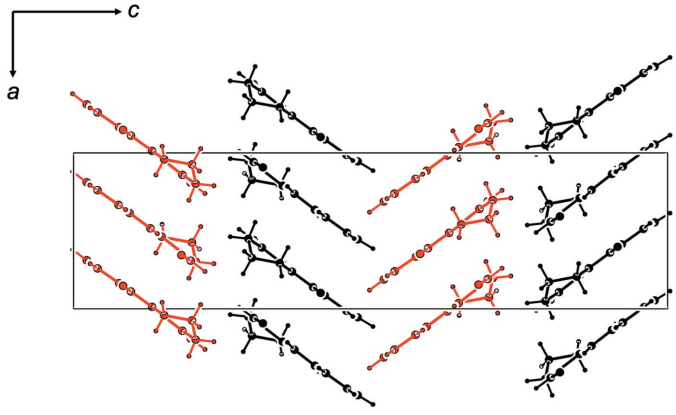



**Figure 3**
Packing in a view along [010]; the independent molecules *A* (black) and *B* (red) stack into separate columns of equidistant molecules along [100].

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N5$A$—H5$A$···O2$A$ | 0.93 (3) | 1.77 (3) | 2.592 (3) | 146 (3) |
| N5$B$—H5$B$···O2$B$ | 0.90 (3) | 1.82 (3) | 2.582 (3) | 141 (3) |
| C1$A$—H1$A$2···O1$A^{i}$ | 0.99 | 2.57 | 3.535 (3) | 164 |
| C6$A$—H6$A$···O1$A^{ii}$ | 0.95 | 2.39 | 3.230 (4) | 147 |
| C6$B$—H6$B$···O1$B^{ii}$ | 0.95 | 2.40 | 3.239 (4) | 148 |
| C8$A$—H8$A$···O1$B^{iii}$ | 0.95 | 2.60 | 3.469 (4) | 153 |
| C1$B$—H1$B$2···O1$B^{iv}$ | 0.99 | 2.59 | 3.550 (3) | 164 |

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

## 3. Supramolecular features

Consecutive molecules in each column along [100] interact *via* $\pi$–$\pi$ stacking and C=O···$\pi$ contacts (Fig. 4). $\pi$–$\pi$ stacking interactions occur between pyrimidine (*Cg*1, *Cg*7) and benzene (*Cg*3, *Cg*9) rings and involve contact distances of *Cg*1···*Cg*3($-\frac{1}{2}+x, \frac{3}{2}-y, z$) = 3.5154 (18) Å (slippage 0.954 Å) and of *Cg*7···*Cg*9($-\frac{1}{2}+x, \frac{3}{2}-y, z$) = 3.5159 (19) Å (slippage 1.054 Å).

Molecules within each $\pi$-stacked column additionally interact *via* C=O···$\pi$ contacts; they amount to C11$A$=O2$A$···*Cg*1($x+\frac{1}{2}, -y+\frac{3}{2}, z$) = 3.212 (2) Å and C11$B$=O2$B$···*Cg*7($x-\frac{1}{2}, -y+\frac{3}{2}, z$) = 3.215 (2) Å. Perpendicular to the stacking direction, non-classical C—H···O hydrogen bonds (Table 1) link the columns along [001] (Fig. 4) and thus form layers parallel to (010).

## 4. Hirshfeld surface analysis

In order to visualize intermolecular interactions in (**1**), the Hirshfeld surface (HS) (Spackman & Jayatilaka, 2009) was analysed and the associated two-dimensional fingerprint plots (McKinnon *et al.*, 2007) calculated with *Crystal Explorer 17* (Turner *et al.*, 2017). The HS mapped with $d_{norm}$ is represented in Fig. 5. White surface areas indicate contacts with distances equal to the sum of van der Waals radii, whereas red and blue colours denote distances shorter (*e.g.* due to hydrogen bonds) or longer than the sum of the van der Waals radii, respectively.

The two-dimensional fingerprint plot for all contacts is depicted in Fig. 6*a*. H···H contacts are responsible for the largest contribution (49.4%) to the Hirshfeld surface (Fig. 6*b*). Besides these contacts, H···O/O···H (21.5%), H···C/C···H (14.9%), C···C (6.7%) and N···C/C···N (4.0%) interactions contribute significantly to the total Hirshfeld surface; their decomposed fingerprint plots are shown in Fig. 6*c*–*f*. The contributions of further contacts are only minor and amount to N···O/O···N (1.4%), C···O/O···C (1.4%), N···H/H···N (0.5%) and O···O (0.1%).

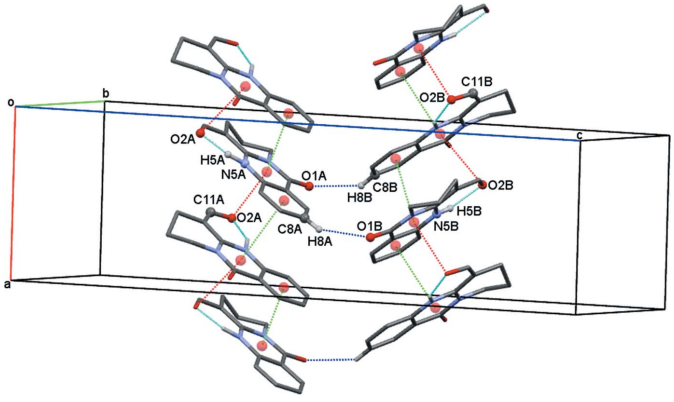



**Figure 4**
Crystal packing of (**1**) in a view along [100]. Intramolecular N—H···O hydrogen bonds are shown as light-blue and intermolecular C—H···O hydrogen bonds as dark-blue dashed lines. Dashed red lines denote contacts C=O···*Cg*1 and C=O···*Cg*7 (slippage 1.676 Å for both), and dashed light-green lines *Cg*1···*Cg*3 and *Cg*7···*Cg*9 contacts. *Cg*3, *Cg*9, *Cg*1 and *Cg*7 correspond to the ring centroids C6*A*–C9*A*/C9*AA*/C5*AA*, C6*B*–C9*B*/C9*BA*/C5*BA*, N5*A*/C4*AA*/N10*A*/C10*A*/C9*AA*/C5*AA* and N5*B*/C4*BA*/N10*B*/C10*B*/C9*BA*/C5*BA*, respectively. For clarity, only H atoms H5*A*, H8*A*, H5*B* and H8*B* are shown.

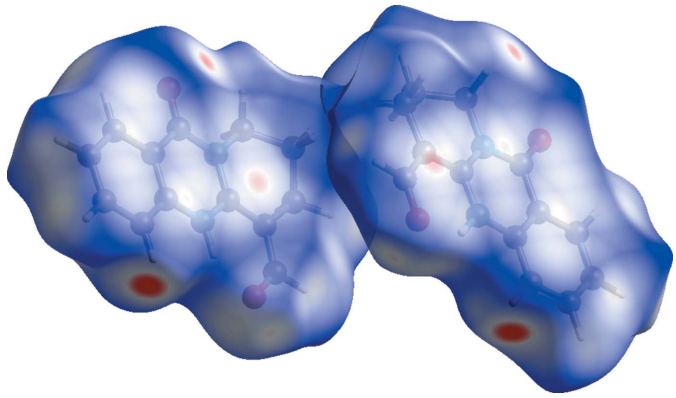


**Figure 5**
Three-dimensional Hirshfeld surface of the title compound plotted over $d_{norm}$ in the range −0.2446 to 1.1709 a.u.

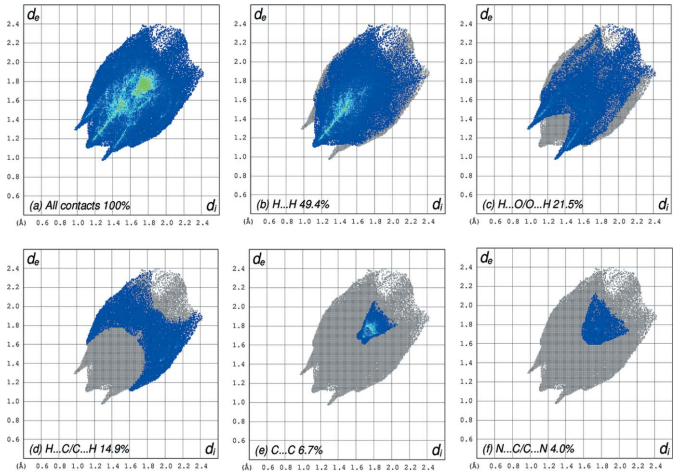



**Figure 6**
Hirshfeld fingerprint plots for (*a*) all contacts and decomposed into (*b*) H···H, (*c*) H···O/O···H, (*d*) H···C/C···H, (*e*) C···C and (*f*) N···C/C···N contacts. $d_i$ and $d_e$ denote the closest internal and external distances (in Å) from a point on the surface.

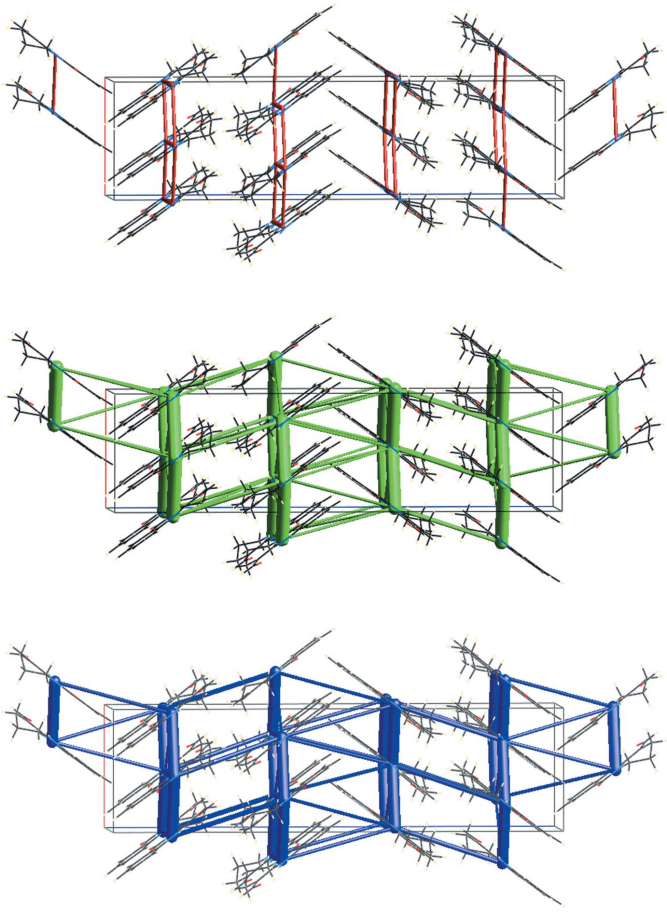


**Figure 7**
Energy frameworks for the electrostatic (red, top) and dispersion (green, middle) components and the total interaction energy (blue, bottom). Cylinder radii are proportional to the corresponding energy; a scale factor of 80 and a cut-off value of 10 kJ mol$^{-1}$ have been used.

## 5. Interaction energy calculations

Intermolecular interaction energies were calculated using the CE–HF/3-21G energy model available in *Crystal Explorer 17* (Turner *et al.*, 2017). The total intermolecular energy ($E_{tot}$) is the sum of electrostatic ($E_{elec}$), polarization ($E_{pol}$), dispersion ($E_{dis}$) and exchange-repulsion ($E_{rep}$) energies (Turner *et al.*, 2015) with scale factors of 1.019, 0.651, 0.901 and 0.811, respectively (Mackenzie *et al.*, 2017). According to these calculations, the major contribution of −306.5 kJ mol$^{-1}$ is due to dispersion interactions (Fig. 7). The other energy components have values of −91.5 kJ mol$^{-1}$, −37.6 kJ mol$^{-1}$ and 155.7 kJ mol$^{-1}$ for the $E_{elec}$, $E_{pol}$ and $E_{rep}$ energies, respectively. The total interaction energy resulting from these four components amounts to −267.1 kJ mol$^{-1}$.

## 6. Database survey

A search in the Cambridge Structural Database (CSD, version 5.41, update January 2020; Groom *et al.*, 2016) revealed six matches for molecules containing the 3-methyl-2-(propan-2-ylidene)-2,3-dihydroquinazolin-4(1*H*)-one moiety with a similar planar conformation to that in the title structure: 3-(2-methylphenyl)-2-(2-oxophenylethyl)-4(3*H*)-quinazolinone (FABWUA10; Duke & Codding, 1993), 3-(2-chlorophenyl)-2-[2-oxo-2-(4-pyridyl)ethyl]-4(3*H*)-quinazolinone (FABXAH10; Duke & Codding, 1993), 2-[2-oxo-2-(4-pyridyl)ethyl]-3-phenyl-4(3*H*)quinazolinone (FABXEL10; Duke & Codding, 1993), 3-(2-methylphenyl)-2-[2-oxo-2-(4-pyridyl)-ethyl]-4(3*H*)-quinazolinone (HADLAZ; Duke & Codding, 1993), 3-(4-chlorophenyl)-2-[2-oxo-2-(4-pyridyl)ethyl]-4(3*H*)-quinazolinone (HADLED; Duke & Codding, 1993) and (*E*)-2-[2-oxo-2-(thiophen-2-yl)ethylidene]-3-phenyl-2,3-dihydroquinazolin-4(1*H*)-one (SATJOP; Narra *et al.*, 2017). A search for the 2-amino-1,4,5,6-tetrahydropyridine-3-carbaldehyde moiety gave one hit with similar conformation: 1-methyl-2-(methylamino)-1,4,5,6-tetrahydropyridine-3-carbaldehyde (MFHPYM10; Horváth *et al.*, 1983). Similar to in (**1**), all compounds mentioned above exist as the enamine tautomer in the crystalline state, and their intramolecular N—H···O hydrogen bond between the ethanone and the amine N atom results in an *S*(6) graph set motif.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{13}H_{12}N_2O_2$ |
| $M_r$ | 228.25 |
| Crystal system, space group | Orthorhombic, $Pna2_1$ |
| Temperature (K) | 100 |
| $a$, $b$, $c$ (Å) | 8.284 (2), 8.006 (2), 31.637 (6) |
| $V$ (Å$^3$) | 2098.2 (8) |
| $Z$ | 8 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.81 |
| Crystal size (mm) | 0.40 × 0.22 × 0.07 |
| | |
| Data collection | |
| Diffractometer | Stoe Stadivari goniometer, Dectris Pilatus 200K area detector |
| Absorption correction | Multi-scan (*LANA*; Koziskova *et al.*, 2016) |
| $T_{min}$, $T_{max}$ | 0.261, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15842, 3629, 3409 |
| $R_{int}$ | 0.013 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.619 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.036, 0.103, 1.03 |
| No. of reflections | 3629 |
| No. of parameters | 316 |
| No. of restraints | 2 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.36, −0.24 |
| Absolute structure | Refined as an inversion twin. |
| Absolute structure parameter | 0.3 (2) |

Computer programs: *X-AREA* (Stoe & Cie, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *publCIF* (Westrip, 2010).

## 7. Synthesis and crystallization

Compound (**1**) was synthesized according to the method of Oripov *et al.* (1979). Yield 12.55 g, 91%; m.p. 474–476 K (after crystallization from hexane), $R_f$ 0.78 ($C_6H_6$: MeOH 4:1). A

detailed report on the synthesis of (**1**) and its characterization by $^1$H NMR is available in Zhurakulov *et al.* (2017). Crystals suitable for X-ray diffraction were obtained from a methanol solution by slow evaporation of the solvent at room temperature.

## 8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms attached to C were positioned geometrically, with C—H = 0.95 Å (for aromatic), 0.95 Å (for the aldehyde H atom), 0.99 Å (for methylene H atoms) and were refined with $U_{iso}$(H) = 1.2$U_{eq}$(C). The enamine H atoms H5*A* and H5*B* were refined with a common isotropic displacement parameter; N—H distances were restrained to similarity.

## Acknowledgements

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## Funding information

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# supporting information

*Acta Cryst.* (2021). E**77**, 47-51 [https://doi.org/10.1107/S2056989020016059]

# Crystal structure, Hirshfeld surface analysis and energy framework study of 6-formyl-7,8,9,11-tetrahydro-5*H*-pyrido[2,1-*b*]quinazolin-11-one

**Akmal Tojiboev, Sherzod Zhurakulov, Valentina Vinogradova, Ulli Englert and Ruimin Wang**

## Computing details

Data collection: *X-AREA* (Stoe & Cie, 2017); cell refinement: *X-AREA* (Stoe & Cie, 2017); data reduction: *X-AREA* (Stoe & Cie, 2017); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020); software used to prepare material for publication: *publCIF* (Westrip, 2010).

### 6-Formyl-7,8,9,11-tetrahydro-5*H*-pyrido[2,1-*b*]quinazolin-11-one

*Crystal data*

$C_{13}H_{12}N_2O_2$
$M_r = 228.25$
Orthorhombic, $Pna2_1$
$a = 8.284$ (2) Å
$b = 8.006$ (2) Å
$c = 31.637$ (6) Å
$V = 2098.2$ (8) Å$^3$
$Z = 8$
$F(000) = 960$
$D_x = 1.445$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54186$ Å
Cell parameters from 17372 reflections
$\theta = 5.6$–$73.9°$
$\mu = 0.81$ mm$^{-1}$
$T = 100$ K
Plate, brown
0.40 × 0.22 × 0.07 mm

*Data collection*

Stoe Stadivari goniometer, Dectris Pilatus 200K area detector diffractometer
Radiation source: XENOCS microsource
rotation method, $\omega$ scans
Absorption correction: multi-scan (*LANA*; Koziskova *et al.*, 2016)
$T_{min} = 0.261$, $T_{max} = 1.000$
15842 measured reflections
3629 independent reflections
3409 reflections with $I > 2\sigma(I)$
$R_{int} = 0.013$
$\theta_{max} = 72.8°$, $\theta_{min} = 5.6°$
$h = -7 \rightarrow 10$
$k = -7 \rightarrow 9$
$l = -35 \rightarrow 38$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.103$
$S = 1.03$
3629 reflections
316 parameters
2 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0861P)^2 + 0.1201P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.36$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ e Å$^{-3}$
Extinction correction: SHELXL-2018/3 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0008 (2)

Absolute structure: Refined as an inversion twin.
Absolute structure parameter: 0.3 (2)

*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.
**Refinement**. Refined as a two-component inversion twin

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1A | 0.6021 (2) | 0.3190 (2) | 0.91582 (6) | 0.0238 (4) |
| O2A | 0.9070 (2) | 0.9397 (2) | 0.81869 (6) | 0.0273 (4) |
| N10A | 0.7388 (2) | 0.4746 (2) | 0.86753 (6) | 0.0188 (4) |
| N5A | 0.7374 (4) | 0.7667 (3) | 0.87182 (9) | 0.0205 (6) |
| H5A | 0.785 (4) | 0.861 (3) | 0.8601 (12) | 0.031 (5)* |
| C10A | 0.6394 (3) | 0.4572 (3) | 0.90289 (7) | 0.0193 (4) |
| C4AA | 0.7900 (3) | 0.6267 (3) | 0.85180 (7) | 0.0183 (5) |
| C5AA | 0.6354 (3) | 0.7651 (3) | 0.90621 (10) | 0.0156 (6) |
| C9AA | 0.5867 (3) | 0.6135 (3) | 0.92277 (9) | 0.0199 (6) |
| C9A | 0.4876 (4) | 0.6078 (3) | 0.95915 (9) | 0.0198 (6) |
| H9A | 0.450928 | 0.504197 | 0.970188 | 0.024* |
| C8A | 0.4454 (4) | 0.7578 (3) | 0.97834 (12) | 0.0208 (7) |
| H8A | 0.386242 | 0.756350 | 1.004070 | 0.025* |
| C7A | 0.4889 (4) | 0.9111 (4) | 0.96020 (9) | 0.0201 (5) |
| H7A | 0.450755 | 1.012023 | 0.972419 | 0.024* |
| C6A | 0.5870 (3) | 0.9174 (4) | 0.92456 (9) | 0.0221 (5) |
| H6A | 0.620410 | 1.021297 | 0.912959 | 0.027* |
| C4A | 0.8927 (3) | 0.6410 (3) | 0.81695 (8) | 0.0219 (5) |
| C11A | 0.9422 (3) | 0.7999 (4) | 0.80294 (8) | 0.0236 (5) |
| H11A | 1.010208 | 0.801898 | 0.778758 | 0.028* |
| C3A | 0.9495 (3) | 0.4844 (3) | 0.79423 (7) | 0.0251 (5) |
| H3A1 | 1.055458 | 0.449174 | 0.805650 | 0.030* |
| H3A2 | 0.962786 | 0.508158 | 0.763718 | 0.030* |
| C2A | 0.8267 (3) | 0.3449 (3) | 0.80027 (8) | 0.0261 (5) |
| H2A1 | 0.725049 | 0.374290 | 0.785613 | 0.031* |
| H2A2 | 0.868683 | 0.240345 | 0.787679 | 0.031* |
| C1A | 0.7936 (3) | 0.3181 (3) | 0.84685 (7) | 0.0234 (5) |
| H1A1 | 0.709624 | 0.231135 | 0.850210 | 0.028* |
| H1A2 | 0.893143 | 0.277918 | 0.860881 | 0.028* |
| O1B | 0.6478 (2) | 0.3186 (2) | 0.58347 (6) | 0.0246 (4) |
| O2B | 0.3447 (2) | 0.9370 (2) | 0.68172 (6) | 0.0260 (4) |
| N10B | 0.5127 (2) | 0.4739 (2) | 0.63232 (6) | 0.0182 (4) |
| N5B | 0.5164 (4) | 0.7649 (2) | 0.62934 (9) | 0.0175 (5) |
| H5B | 0.482 (4) | 0.861 (3) | 0.6411 (12) | 0.031 (5)* |
| C10B | 0.6112 (3) | 0.4574 (3) | 0.59674 (7) | 0.0188 (4) |

| | | | | |
|---|---|---|---|---|
| C4BA | 0.4630 (3) | 0.6258 (3) | 0.64828 (8) | 0.0186 (5) |
| C5BA | 0.6149 (4) | 0.7667 (3) | 0.59430 (11) | 0.0206 (7) |
| C9BA | 0.6647 (3) | 0.6125 (3) | 0.57672 (8) | 0.0166 (5) |
| C9B | 0.7593 (4) | 0.6123 (3) | 0.54056 (10) | 0.0214 (6) |
| H9B | 0.788639 | 0.508758 | 0.528072 | 0.026* |
| C8B | 0.8117 (4) | 0.7591 (3) | 0.52233 (13) | 0.0232 (8) |
| H8B | 0.880004 | 0.757998 | 0.498217 | 0.028* |
| C7B | 0.7611 (4) | 0.9097 (4) | 0.54040 (10) | 0.0244 (6) |
| H7B | 0.793649 | 1.011674 | 0.527607 | 0.029* |
| C6B | 0.6666 (3) | 0.9156 (4) | 0.57585 (9) | 0.0196 (5) |
| H6B | 0.636423 | 1.020004 | 0.587748 | 0.023* |
| C4B | 0.3593 (3) | 0.6378 (3) | 0.68323 (8) | 0.0205 (5) |
| C11B | 0.3090 (3) | 0.7967 (4) | 0.69727 (8) | 0.0234 (4) |
| H11B | 0.239986 | 0.798223 | 0.721255 | 0.028* |
| C3B | 0.3026 (3) | 0.4828 (3) | 0.70566 (7) | 0.0238 (5) |
| H3B1 | 0.196540 | 0.447861 | 0.694240 | 0.029* |
| H3B2 | 0.289541 | 0.506169 | 0.736197 | 0.029* |
| C2B | 0.4255 (3) | 0.3433 (3) | 0.69938 (8) | 0.0254 (5) |
| H2B1 | 0.527309 | 0.372514 | 0.714023 | 0.030* |
| H2B2 | 0.383741 | 0.238507 | 0.711861 | 0.030* |
| C1B | 0.4583 (3) | 0.3175 (3) | 0.65270 (7) | 0.0226 (5) |
| H1B1 | 0.542250 | 0.230628 | 0.649173 | 0.027* |
| H1B2 | 0.358647 | 0.277673 | 0.638669 | 0.027* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0349 (8) | 0.0136 (9) | 0.0228 (8) | −0.0017 (6) | 0.0023 (6) | 0.0010 (5) |
| O2A | 0.0321 (9) | 0.0214 (9) | 0.0282 (10) | −0.0033 (7) | −0.0001 (7) | −0.0003 (8) |
| N10A | 0.0238 (8) | 0.0142 (9) | 0.0185 (9) | 0.0011 (7) | −0.0015 (7) | −0.0010 (7) |
| N5A | 0.0249 (14) | 0.0170 (11) | 0.0196 (13) | −0.0012 (7) | −0.0001 (11) | −0.0019 (7) |
| C10A | 0.0225 (10) | 0.0184 (11) | 0.0170 (10) | 0.0010 (7) | −0.0047 (8) | −0.0013 (7) |
| C4AA | 0.0207 (10) | 0.0172 (12) | 0.0171 (11) | 0.0013 (8) | −0.0046 (8) | 0.0005 (8) |
| C5AA | 0.0155 (14) | 0.0179 (12) | 0.0133 (12) | 0.0008 (7) | −0.0040 (10) | 0.0000 (8) |
| C9AA | 0.0217 (12) | 0.0192 (15) | 0.0189 (12) | 0.0017 (8) | −0.0050 (11) | −0.0019 (10) |
| C9A | 0.0225 (12) | 0.0216 (14) | 0.0153 (11) | 0.0016 (9) | −0.0026 (9) | 0.0015 (9) |
| C8A | 0.0249 (17) | 0.0211 (16) | 0.0165 (17) | 0.0007 (8) | −0.0021 (11) | −0.0021 (7) |
| C7A | 0.0251 (12) | 0.0166 (12) | 0.0185 (12) | 0.0035 (10) | −0.0036 (10) | −0.0042 (10) |
| C6A | 0.0246 (12) | 0.0179 (13) | 0.0237 (12) | 0.0008 (10) | −0.0052 (11) | 0.0000 (10) |
| C4A | 0.0239 (12) | 0.0204 (12) | 0.0214 (12) | 0.0025 (10) | −0.0023 (9) | −0.0030 (10) |
| C11A | 0.0248 (11) | 0.0254 (13) | 0.0205 (12) | −0.0006 (11) | 0.0001 (9) | 0.0032 (11) |
| C3A | 0.0299 (11) | 0.0247 (12) | 0.0208 (12) | 0.0057 (9) | 0.0040 (8) | 0.0022 (9) |
| C2A | 0.0370 (12) | 0.0216 (10) | 0.0197 (12) | 0.0022 (9) | 0.0011 (8) | −0.0028 (8) |
| C1A | 0.0317 (11) | 0.0180 (13) | 0.0204 (13) | 0.0027 (9) | 0.0008 (9) | −0.0022 (8) |
| O1B | 0.0348 (8) | 0.0165 (9) | 0.0226 (8) | 0.0016 (6) | 0.0030 (6) | −0.0001 (5) |
| O2B | 0.0311 (8) | 0.0213 (8) | 0.0256 (9) | 0.0023 (8) | 0.0014 (6) | −0.0046 (7) |
| N10B | 0.0237 (9) | 0.0141 (9) | 0.0169 (8) | −0.0007 (7) | −0.0017 (7) | 0.0009 (7) |
| N5B | 0.0209 (12) | 0.0146 (11) | 0.0170 (12) | 0.0002 (7) | −0.0024 (10) | −0.0021 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10B | 0.0228 (10) | 0.0159 (10) | 0.0176 (10) | 0.0004 (8) | −0.0030 (8) | −0.0006 (7) |
| C4BA | 0.0213 (10) | 0.0150 (12) | 0.0194 (11) | 0.0006 (7) | −0.0065 (8) | −0.0005 (8) |
| C5BA | 0.0243 (17) | 0.0169 (13) | 0.0205 (15) | 0.0008 (8) | −0.0056 (12) | −0.0005 (9) |
| C9BA | 0.0197 (11) | 0.0139 (14) | 0.0163 (11) | −0.0004 (8) | −0.0030 (10) | 0.0017 (8) |
| C9B | 0.0228 (12) | 0.0178 (13) | 0.0235 (13) | 0.0027 (9) | −0.0029 (10) | −0.0008 (10) |
| C8B | 0.0207 (16) | 0.0280 (17) | 0.0209 (19) | −0.0026 (8) | −0.0001 (11) | 0.0022 (8) |
| C7B | 0.0235 (13) | 0.0231 (13) | 0.0265 (14) | −0.0021 (11) | −0.0043 (11) | 0.0065 (11) |
| C6B | 0.0238 (12) | 0.0144 (12) | 0.0205 (12) | 0.0001 (9) | −0.0038 (10) | 0.0025 (9) |
| C4B | 0.0200 (11) | 0.0262 (13) | 0.0154 (10) | −0.0002 (9) | −0.0031 (8) | −0.0023 (9) |
| C11B | 0.0240 (11) | 0.0261 (12) | 0.0201 (12) | 0.0003 (10) | −0.0019 (8) | −0.0017 (11) |
| C3B | 0.0314 (11) | 0.0215 (11) | 0.0185 (11) | −0.0044 (9) | 0.0031 (8) | 0.0004 (8) |
| C2B | 0.0380 (12) | 0.0210 (10) | 0.0170 (11) | −0.0012 (9) | 0.0015 (9) | 0.0035 (7) |
| C1B | 0.0330 (11) | 0.0129 (12) | 0.0220 (12) | −0.0020 (8) | 0.0005 (9) | 0.0021 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1A—C10A | 1.220 (3) | O1B—C10B | 1.226 (3) |
| O2A—C11A | 1.259 (4) | O2B—C11B | 1.262 (4) |
| N10A—C4AA | 1.382 (3) | N10B—C4BA | 1.380 (3) |
| N10A—C10A | 1.396 (3) | N10B—C10B | 1.397 (3) |
| N10A—C1A | 1.485 (3) | N10B—C1B | 1.478 (3) |
| N5A—C4AA | 1.359 (3) | N5B—C4BA | 1.340 (3) |
| N5A—C5AA | 1.377 (4) | N5B—C5BA | 1.377 (5) |
| N5A—H5A | 0.93 (2) | N5B—H5B | 0.90 (2) |
| C10A—C9AA | 1.467 (3) | C10B—C9BA | 1.463 (3) |
| C4AA—C4A | 1.397 (4) | C4BA—C4B | 1.404 (4) |
| C5AA—C9AA | 1.382 (4) | C5BA—C6B | 1.395 (4) |
| C5AA—C6A | 1.409 (4) | C5BA—C9BA | 1.415 (4) |
| C9AA—C9A | 1.414 (4) | C9BA—C9B | 1.386 (4) |
| C9A—C8A | 1.390 (4) | C9B—C8B | 1.380 (4) |
| C9A—H9A | 0.9500 | C9B—H9B | 0.9500 |
| C8A—C7A | 1.402 (4) | C8B—C7B | 1.398 (4) |
| C8A—H8A | 0.9500 | C8B—H8B | 0.9500 |
| C7A—C6A | 1.391 (4) | C7B—C6B | 1.369 (4) |
| C7A—H7A | 0.9500 | C7B—H7B | 0.9500 |
| C6A—H6A | 0.9500 | C6B—H6B | 0.9500 |
| C4A—C11A | 1.409 (4) | C4B—C11B | 1.410 (4) |
| C4A—C3A | 1.520 (3) | C4B—C3B | 1.505 (3) |
| C11A—H11A | 0.9500 | C11B—H11B | 0.9500 |
| C3A—C2A | 1.523 (3) | C3B—C2B | 1.524 (3) |
| C3A—H3A1 | 0.9900 | C3B—H3B1 | 0.9900 |
| C3A—H3A2 | 0.9900 | C3B—H3B2 | 0.9900 |
| C2A—C1A | 1.514 (3) | C2B—C1B | 1.516 (3) |
| C2A—H2A1 | 0.9900 | C2B—H2B1 | 0.9900 |
| C2A—H2A2 | 0.9900 | C2B—H2B2 | 0.9900 |
| C1A—H1A1 | 0.9900 | C1B—H1B1 | 0.9900 |
| C1A—H1A2 | 0.9900 | C1B—H1B2 | 0.9900 |

| | | | |
|---|---|---|---|
| C4AA—N10A—C10A | 123.88 (18) | C4BA—N10B—C10B | 123.5 (2) |
| C4AA—N10A—C1A | 119.42 (18) | C4BA—N10B—C1B | 119.72 (19) |
| C10A—N10A—C1A | 116.69 (17) | C10B—N10B—C1B | 116.73 (18) |
| C4AA—N5A—C5AA | 123.9 (2) | C4BA—N5B—C5BA | 124.3 (2) |
| C4AA—N5A—H5A | 110 (2) | C4BA—N5B—H5B | 115 (3) |
| C5AA—N5A—H5A | 126 (2) | C5BA—N5B—H5B | 121 (3) |
| O1A—C10A—N10A | 120.6 (2) | O1B—C10B—N10B | 120.4 (2) |
| O1A—C10A—C9AA | 123.7 (2) | O1B—C10B—C9BA | 123.1 (2) |
| N10A—C10A—C9AA | 115.7 (2) | N10B—C10B—C9BA | 116.5 (2) |
| N5A—C4AA—N10A | 117.4 (2) | N5B—C4BA—N10B | 118.1 (2) |
| N5A—C4AA—C4A | 119.7 (2) | N5B—C4BA—C4B | 119.8 (2) |
| N10A—C4AA—C4A | 122.9 (2) | N10B—C4BA—C4B | 122.1 (2) |
| N5A—C5AA—C9AA | 119.1 (2) | N5B—C5BA—C6B | 121.8 (3) |
| N5A—C5AA—C6A | 119.5 (2) | N5B—C5BA—C9BA | 118.7 (2) |
| C9AA—C5AA—C6A | 121.4 (3) | C6B—C5BA—C9BA | 119.4 (3) |
| C5AA—C9AA—C9A | 120.4 (2) | C9B—C9BA—C5BA | 119.3 (2) |
| C5AA—C9AA—C10A | 120.0 (2) | C9B—C9BA—C10B | 121.8 (2) |
| C9A—C9AA—C10A | 119.6 (2) | C5BA—C9BA—C10B | 118.8 (2) |
| C8A—C9A—C9AA | 118.3 (3) | C8B—C9B—C9BA | 121.5 (3) |
| C8A—C9A—H9A | 120.9 | C8B—C9B—H9B | 119.3 |
| C9AA—C9A—H9A | 120.9 | C9BA—C9B—H9B | 119.3 |
| C9A—C8A—C7A | 120.8 (3) | C9B—C8B—C7B | 118.0 (4) |
| C9A—C8A—H8A | 119.6 | C9B—C8B—H8B | 121.0 |
| C7A—C8A—H8A | 119.6 | C7B—C8B—H8B | 121.0 |
| C6A—C7A—C8A | 120.9 (3) | C6B—C7B—C8B | 122.4 (3) |
| C6A—C7A—H7A | 119.5 | C6B—C7B—H7B | 118.8 |
| C8A—C7A—H7A | 119.5 | C8B—C7B—H7B | 118.8 |
| C7A—C6A—C5AA | 117.9 (3) | C7B—C6B—C5BA | 119.3 (3) |
| C7A—C6A—H6A | 121.0 | C7B—C6B—H6B | 120.4 |
| C5AA—C6A—H6A | 121.0 | C5BA—C6B—H6B | 120.4 |
| C4AA—C4A—C11A | 120.0 (2) | C4BA—C4B—C11B | 119.4 (2) |
| C4AA—C4A—C3A | 119.6 (2) | C4BA—C4B—C3B | 120.4 (2) |
| C11A—C4A—C3A | 120.4 (3) | C11B—C4B—C3B | 120.2 (2) |
| O2A—C11A—C4A | 127.6 (2) | O2B—C11B—C4B | 127.6 (2) |
| O2A—C11A—H11A | 116.2 | O2B—C11B—H11B | 116.2 |
| C4A—C11A—H11A | 116.2 | C4B—C11B—H11B | 116.2 |
| C4A—C3A—C2A | 109.80 (19) | C4B—C3B—C2B | 109.50 (19) |
| C4A—C3A—H3A1 | 109.7 | C4B—C3B—H3B1 | 109.8 |
| C2A—C3A—H3A1 | 109.7 | C2B—C3B—H3B1 | 109.8 |
| C4A—C3A—H3A2 | 109.7 | C4B—C3B—H3B2 | 109.8 |
| C2A—C3A—H3A2 | 109.7 | C2B—C3B—H3B2 | 109.8 |
| H3A1—C3A—H3A2 | 108.2 | H3B1—C3B—H3B2 | 108.2 |
| C1A—C2A—C3A | 110.31 (19) | C1B—C2B—C3B | 110.27 (19) |
| C1A—C2A—H2A1 | 109.6 | C1B—C2B—H2B1 | 109.6 |
| C3A—C2A—H2A1 | 109.6 | C3B—C2B—H2B1 | 109.6 |
| C1A—C2A—H2A2 | 109.6 | C1B—C2B—H2B2 | 109.6 |
| C3A—C2A—H2A2 | 109.6 | C3B—C2B—H2B2 | 109.6 |
| H2A1—C2A—H2A2 | 108.1 | H2B1—C2B—H2B2 | 108.1 |

| | | | |
|---|---|---|---|
| N10A—C1A—C2A | 111.38 (18) | N10B—C1B—C2B | 111.35 (18) |
| N10A—C1A—H1A1 | 109.4 | N10B—C1B—H1B1 | 109.4 |
| C2A—C1A—H1A1 | 109.4 | C2B—C1B—H1B1 | 109.4 |
| N10A—C1A—H1A2 | 109.4 | N10B—C1B—H1B2 | 109.4 |
| C2A—C1A—H1A2 | 109.4 | C2B—C1B—H1B2 | 109.4 |
| H1A1—C1A—H1A2 | 108.0 | H1B1—C1B—H1B2 | 108.0 |
| | | | |
| C4AA—N10A—C10A—O1A | −178.06 (19) | C4BA—N10B—C10B—O1B | −178.11 (19) |
| C1A—N10A—C10A—O1A | 0.6 (3) | C1B—N10B—C10B—O1B | 0.6 (3) |
| C4AA—N10A—C10A—C9AA | 1.4 (3) | C4BA—N10B—C10B—C9BA | 1.3 (3) |
| C1A—N10A—C10A—C9AA | −179.96 (19) | C1B—N10B—C10B—C9BA | −179.99 (19) |
| C5AA—N5A—C4AA—N10A | −0.8 (4) | C5BA—N5B—C4BA—N10B | 1.5 (4) |
| C5AA—N5A—C4AA—C4A | 179.6 (3) | C5BA—N5B—C4BA—C4B | −178.3 (3) |
| C10A—N10A—C4AA—N5A | −1.1 (3) | C10B—N10B—C4BA—N5B | −2.1 (3) |
| C1A—N10A—C4AA—N5A | −179.7 (2) | C1B—N10B—C4BA—N5B | 179.2 (2) |
| C10A—N10A—C4AA—C4A | 178.4 (2) | C10B—N10B—C4BA—C4B | 177.7 (2) |
| C1A—N10A—C4AA—C4A | −0.2 (3) | C1B—N10B—C4BA—C4B | −1.0 (3) |
| C4AA—N5A—C5AA—C9AA | 2.4 (4) | C4BA—N5B—C5BA—C6B | 179.9 (3) |
| C4AA—N5A—C5AA—C6A | −179.6 (3) | C4BA—N5B—C5BA—C9BA | −0.1 (5) |
| N5A—C5AA—C9AA—C9A | 177.3 (3) | N5B—C5BA—C9BA—C9B | 177.7 (3) |
| C6A—C5AA—C9AA—C9A | −0.7 (5) | C6B—C5BA—C9BA—C9B | −2.3 (5) |
| N5A—C5AA—C9AA—C10A | −2.0 (4) | N5B—C5BA—C9BA—C10B | −0.7 (4) |
| C6A—C5AA—C9AA—C10A | −179.9 (2) | C6B—C5BA—C9BA—C10B | 179.2 (2) |
| O1A—C10A—C9AA—C5AA | 179.6 (2) | O1B—C10B—C9BA—C9B | 1.1 (4) |
| N10A—C10A—C9AA—C5AA | 0.2 (3) | N10B—C10B—C9BA—C9B | −178.3 (2) |
| O1A—C10A—C9AA—C9A | 0.4 (4) | O1B—C10B—C9BA—C5BA | 179.5 (2) |
| N10A—C10A—C9AA—C9A | −179.1 (2) | N10B—C10B—C9BA—C5BA | 0.2 (3) |
| C5AA—C9AA—C9A—C8A | −2.0 (4) | C5BA—C9BA—C9B—C8B | 2.8 (5) |
| C10A—C9AA—C9A—C8A | 177.3 (3) | C10B—C9BA—C9B—C8B | −178.8 (3) |
| C9AA—C9A—C8A—C7A | 5.1 (5) | C9BA—C9B—C8B—C7B | −2.6 (6) |
| C9A—C8A—C7A—C6A | −5.5 (5) | C9B—C8B—C7B—C6B | 1.9 (6) |
| C8A—C7A—C6A—C5AA | 2.7 (4) | C8B—C7B—C6B—C5BA | −1.5 (5) |
| N5A—C5AA—C6A—C7A | −177.7 (3) | N5B—C5BA—C6B—C7B | −178.4 (3) |
| C9AA—C5AA—C6A—C7A | 0.3 (5) | C9BA—C5BA—C6B—C7B | 1.7 (5) |
| N5A—C4AA—C4A—C11A | 0.5 (4) | N5B—C4BA—C4B—C11B | 1.7 (4) |
| N10A—C4AA—C4A—C11A | −179.0 (2) | N10B—C4BA—C4B—C11B | −178.1 (2) |
| N5A—C4AA—C4A—C3A | −179.2 (2) | N5B—C4BA—C4B—C3B | −178.1 (2) |
| N10A—C4AA—C4A—C3A | 1.3 (3) | N10B—C4BA—C4B—C3B | 2.1 (3) |
| C4AA—C4A—C11A—O2A | 1.3 (4) | C4BA—C4B—C11B—O2B | 0.8 (4) |
| C3A—C4A—C11A—O2A | −179.1 (2) | C3B—C4B—C11B—O2B | −179.4 (2) |
| C4AA—C4A—C3A—C2A | 26.3 (3) | C4BA—C4B—C3B—C2B | 26.0 (3) |
| C11A—C4A—C3A—C2A | −153.4 (2) | C11B—C4B—C3B—C2B | −153.9 (2) |
| C4A—C3A—C2A—C1A | −54.1 (3) | C4B—C3B—C2B—C1B | −54.1 (3) |
| C4AA—N10A—C1A—C2A | −28.9 (3) | C4BA—N10B—C1B—C2B | −28.5 (3) |
| C10A—N10A—C1A—C2A | 152.38 (19) | C10B—N10B—C1B—C2B | 152.68 (19) |
| C3A—C2A—C1A—N10A | 56.2 (2) | C3B—C2B—C1B—N10B | 56.2 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N5*A*—H5*A*···O2*A* | 0.93 (3) | 1.77 (3) | 2.592 (3) | 146 (3) |
| N5*B*—H5*B*···O2*B* | 0.90 (3) | 1.82 (3) | 2.582 (3) | 141 (3) |
| C1*A*—H1*A*2···O1*A*$^{i}$ | 0.99 | 2.57 | 3.535 (3) | 164 |
| C6*A*—H6*A*···O1*A*$^{ii}$ | 0.95 | 2.39 | 3.230 (4) | 147 |
| C6*B*—H6*B*···O1*B*$^{ii}$ | 0.95 | 2.40 | 3.239 (4) | 148 |
| C8*A*—H8*A*···O1*B*$^{iii}$ | 0.95 | 2.60 | 3.469 (4) | 153 |
| C1*B*—H1*B*2···O1*B*$^{iv}$ | 0.99 | 2.59 | 3.550 (3) | 164 |

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