Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

# A new solvate of afatinib, a specific inhibitor of the ErbB family of tyrosine kinases

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Received 6 February 2017
Accepted 15 February 2017

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

**Keywords:** crystal structure; pseudo-symmetry; pseudo-inversion center; ErbB tyrosine kinase inhibitor; hydrogen bonding.

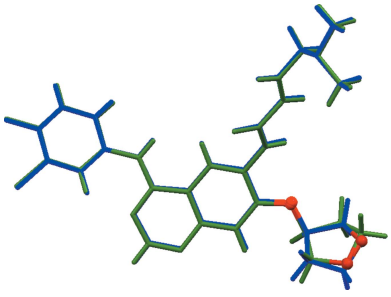
**CCDC reference**: 1532940

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

Afatinib (systematic name: *N*-{4-(3-chloro-4-fluoroanilino)-7-[(tetrahydrofuran-3-yl)oxy]quinazolin-6-yl}-4-(dimethylamino)but-2-enamide), is a specific inhibitor of the ErbB family of tyrosine kinases. The free base form crystallizes from acetonitrile as a mixed water–acetonitrile solvent, $C_{24}H_{25}ClFN_5O_3\cdot 0.25C_2H_3N\cdot 2H_2O$. It crystallizes with two independent molecules (*A* and *B*) in the asymmetric unit of the chiral space group $P42_12$, but exhibits close to perfect pseudo-inversion symmetry, emulating $P4/ncc$ that relates the two molecules to each other. Exact inversion symmetry is however broken by swapping of oxygen and $CH_2$ moieties of the outer tetrahydrofuranyl substituents of the two independent molecules. This can, in turn, be traced back to C—H···N and C—H···O interactions of the acetonitrile solvent molecules with the tetrahydrofuran oxygen and $CH_2$ units. In the crystal, neighboring molecules are connected *via* N—H···O hydrogen bonds between the secondary amine and the amide keto O atom. Additional hydrogen bonds are formed through the water solvent molecules, which are engaged in O—H···O and O—H···N hydrogen bonds connecting to the dimethylamino N atom, the amide keto O atom, and one of the quinazoline N atoms of a neighboring molecule, leading to an intricate three-dimensional hydrogen-bonded superstructure. There are two types of channels stretching along the direction of the *c* axis; one along the fourfold rotational axis, occupied by acetonitrile solvent molecules situated on that axis, and parallel channels which are not occupied by any solvent.

## 1. Chemical context

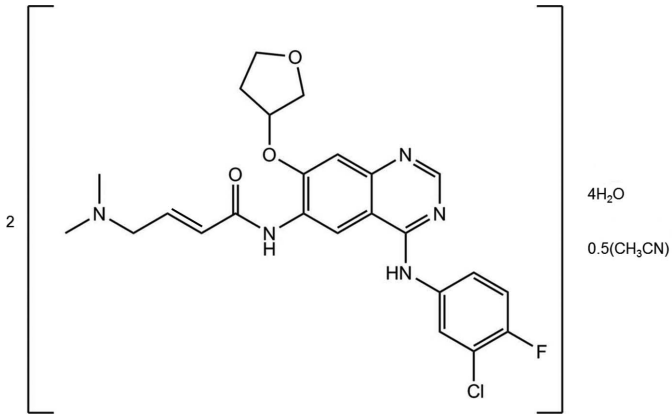
Afatinib is an orally administered antitumor drug used for the treatment of patients with metastatic nonsmall cell lung carcinoma (Keating, 2014). This drug is an irreversible specific inhibitor of ErbB family of tyrosine kinases, comprising EGFR (ErbB1), HER2 (ErbB2), and HER4 (ErbB4) (Hirsh, 2011; Keating, 2014). It is marketed as a dimaleate salt (Giotrif, Boehringer–Ingelheim Pharma GmbH, Ingelheim, Germany) and is reported to present polymorphism as a free base and in its salt forms, as well as an ethanol solvate (Gidwani *et al.*, 2012; Jiadeng, 2016). However, to the best of our knowledge no single-crystal structure has been described so far for any of the reported crystal forms. Herein, we describe an acetonitrile–water solvent structure of afatinib free base obtained *via* vapor diffusion experiments.



## 2. Structural commentary

Crystals of free base afatinib were obtained from an acetonitrile solution through vapor diffusion of hexanes. The

compound crystallized in a tetragonal setting, space group $P42_12$, as a mixed water–acetonitrile solvent with two molecules of water and one-quarter molecule of acetonitrile per formula unit of afatinib (Fig. 1). Two crystallographically independent molecules (*A* and *B*) of afatinib are present, and both exhibit disorder of its tetrahydrofuran-3-yloxy units, with a disorder ratio of 0.718 (9):0.282 (9) for molecule *A* and 0.787 (5):0.213 (5) for molecule *B*. The type of disorder differs slightly between the two molecules (see Fig. 2 and §5, *Refinement*).




The two independent molecules (*A* and *B*) are related by a pseudo-inversion center with close to centrosymmetric *P*4/*ncc* symmetry. After inversion, the two molecules are nearly superimposable with only very minor deviations for the

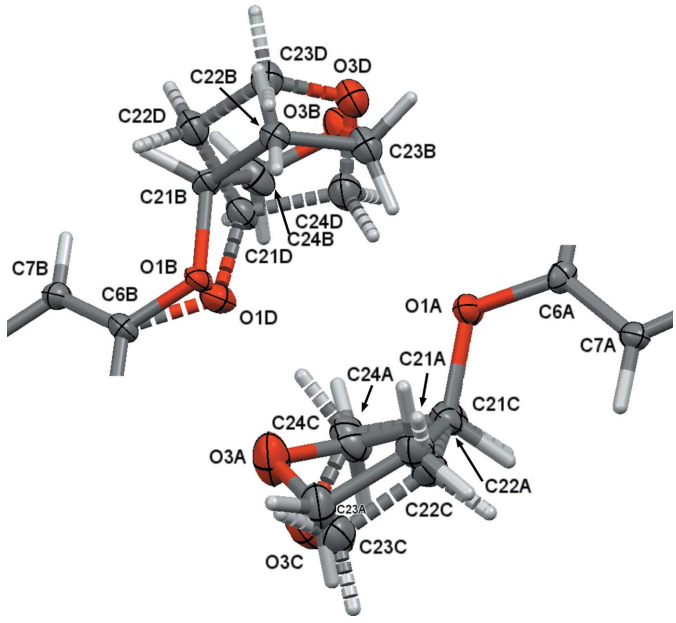



**Figure 2**
View of the two disordered tetrahydrofuran-3-yloxy moieties, with 50% probability displacement ellipsoids. Dashed lines indicate minor disordered moieties *C* and *D*.

aromatic core, the chlorofluoroaniline substituent and the (dimethylamino)but-2-enamide unit (see Fig. 3 for the molecular overlay). Focusing only on the two major moieties exact inversion symmetry is broken solely by the positions of the tetrahydrofuran (THF) O atoms, which are swapped with a

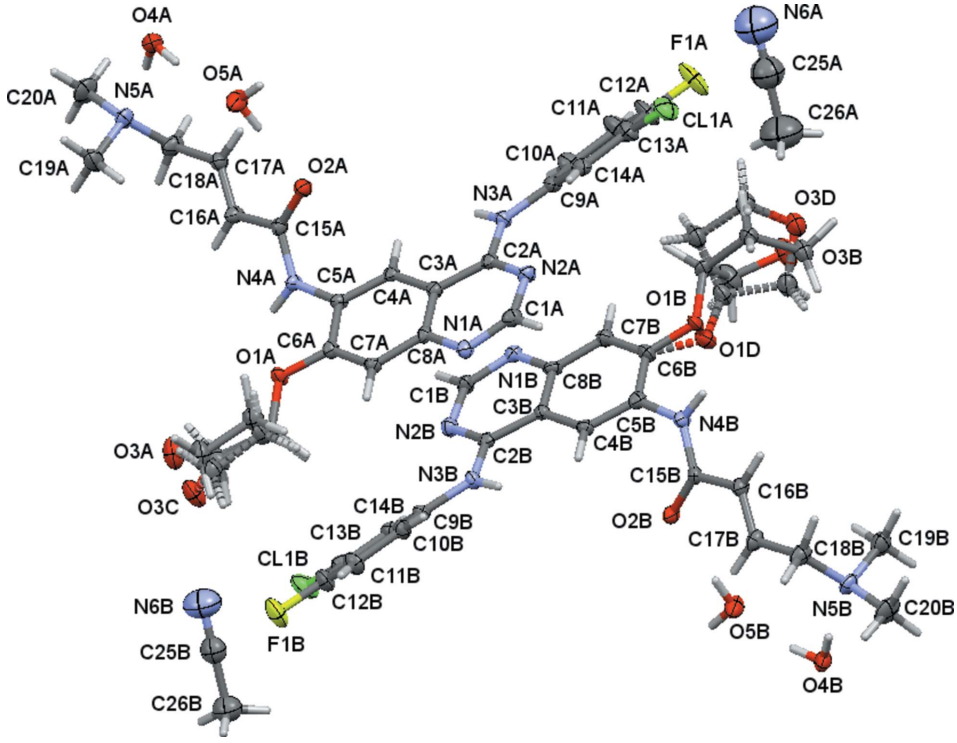



**Figure 1**
The molecular structure of the title compound, showing the atom labeling. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate minor disordered moieties *C* and *D* of the tetrahydrofuran-3-yloxy units. C-atom labels of the disordered moieties have been omitted for clarity.

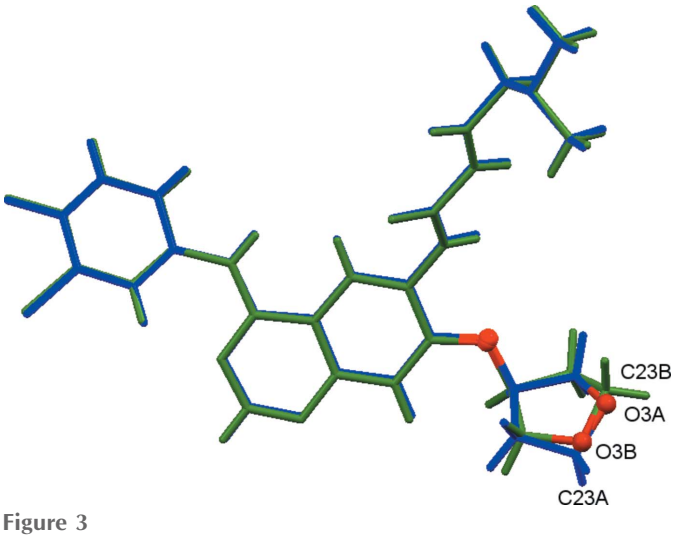



**Figure 3**
Least-squares overlay of molecule *A* (blue) on inverted molecule *B* (green). O atoms of the tetrahydrofuran-3-yloxy units are shown as red spheres to illustrate the absence of inversion symmetry in the title structure. The r.m.s. deviation is 0.6892 Å.

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4*A*—H4*A*1···O4*B*$^{i}$ | 0.88 | 1.94 | 2.804 (5) | 166 |
| N3*A*—H3*A*···O2*A*$^{ii}$ | 0.88 | 2.22 | 3.088 (5) | 167 |
| O4*A*—H4*C*···N5*A* | 0.85 (5) | 1.97 (5) | 2.816 (6) | 173 (4) |
| O4*A*—H4*D*···O5*A* | 0.86 (5) | 1.91 (5) | 2.759 (6) | 169 (5) |
| O5*A*—H5*C*···O2*A* | 0.85 (3) | 2.00 (4) | 2.844 (4) | 174 (5) |
| O5*A*—H5*D*···N1*B*$^{ii}$ | 0.83 (5) | 1.98 (5) | 2.775 (6) | 161 (5) |
| C4*A*—H4*A*···O2*A*$^{ii}$ | 0.95 | 2.31 | 3.246 (6) | 168 |
| C16*A*—H16*A*···O4*B*$^{i}$ | 0.95 | 2.41 | 3.183 (6) | 139 |
| C22*A*—H22*A*···O3*A*$^{iii}$ | 0.99 | 2.40 | 3.353 (13) | 162 |
| C24*A*—H24*A*···O5*B*$^{i}$ | 0.99 | 2.50 | 3.388 (14) | 150 |
| N4*B*—H4*B*1···O4*A*$^{iv}$ | 0.88 | 1.96 | 2.820 (5) | 167 |
| N3*B*—H3*B*···O2*B*$^{v}$ | 0.88 | 2.26 | 3.123 (5) | 166 |
| O4*B*—H4*E*···N5*B* | 0.87 (5) | 1.95 (5) | 2.811 (5) | 175 (6) |
| O4*B*—H4*F*···O5*B* | 0.86 (4) | 1.91 (4) | 2.756 (5) | 169 (4) |
| O5*B*—H5*E*···O2*B* | 0.85 (4) | 1.98 (3) | 2.828 (4) | 173 (5) |
| O5*B*—H5*F*···N1*A*$^{v}$ | 0.86 (4) | 1.95 (4) | 2.794 (5) | 169 (5) |
| C4*B*—H4*B*···O2*B*$^{v}$ | 0.95 | 2.34 | 3.280 (6) | 169 |
| C16*B*—H16*B*···O4*A*$^{iv}$ | 0.95 | 2.42 | 3.197 (6) | 139 |
| C22*B*—H22*D*···O5*A*$^{iv}$ | 0.99 | 2.43 | 3.160 (8) | 130 |
| C24*B*—H24*D*···O3*B*$^{vi}$ | 0.99 | 2.57 | 3.455 (9) | 149 |

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

$CH_2$ group between the two molecules. Associated with the different positions of O and $CH_2$ moieties, and possibly providing an explanation for this observation, is an ordering of the acetonitrile solvent molecules. Acetonitrile molecules related by pseudo-inversion do not as expected point in opposite directions, but are co-parallel with each other. The $CH_3CN$ molecules are located in channels surrounded by the tetrahydrofuranyl units, and they interact with molecules *A* and *B* in opposite ways. The methyl ends of both $CH_3CN$ molecules form C—H···O hydrogen bonds with the O atoms of the major moieties of molecule *B*, capping a tetramer of THF units on both sides. The nitrogen ends of the acetonitrile units, on the other hand, act as acceptors of weak C—H···N hydrogen bonds (Fig. 4 and Table 1). The methyl and nitrogen ends of the linear molecules are related by the pseudo-inversion operation. The different polarity of the two ends, one an hydrogen-bond donor, the other an hydrogen-bond acceptor, can thus be seen as an immediate cause for the swapping of oxygen and the $CH_2$ groups, which are also hydrogen-bond donors and acceptors, so that an attractive rather than repulsive interaction is maintained. Exact inversion symmetry is also broken by the different disorder patterns for the tetrahydrofuran-3-yloxy units substituents (see §5, *Refinement*, and Fig. 2 for details). The absence of inversion symmetry is further evidenced by the value of the absolute structure parameter for racemic twinning, which refined to 0.02 (1), indicating a chiral or noncentrosymmetric space group incompatible with centrosymmetric *P*4/*ncc* symmetry.

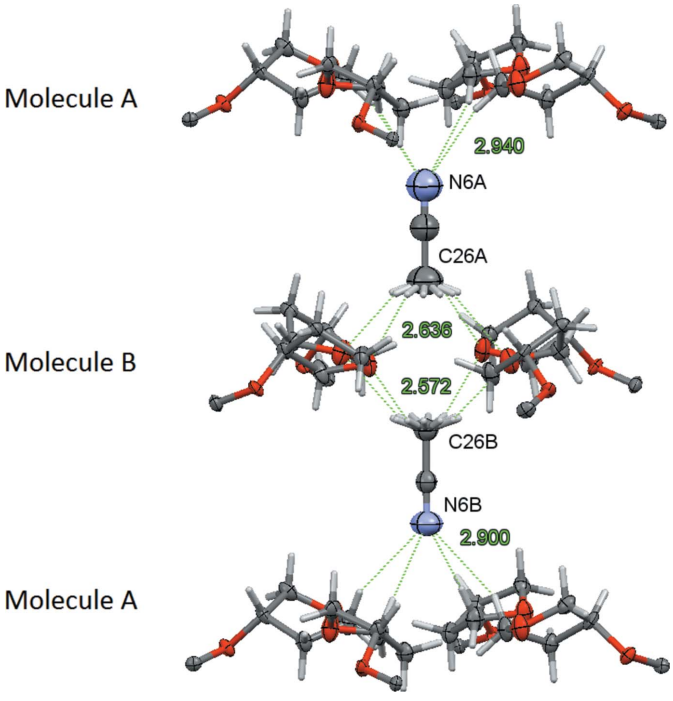



**Figure 4**
View of the interactions of the acetonitrile solvent molecules with the tetrahydrofuranyl units of molecules *A* and *B*. The remaining sections of molecules *A* and *B* have been omitted for clarity. Acetonitrile molecules are located on a fourfold rotation axis hence and the H atoms are fourfold disordered. C—H···N and C—H···O hydrogen bonds are indicated as green dashed lines and the H···N and H···O distances (Å) are given (see Table 1).

Bond lengths and angles in both molecules are unexceptional and in the expected ranges. The central quinazoline cores of the molecules are nearly planar, with maximum deviations of 0.073 (5) Å for atoms C5*A* and C5*B* in molecules *A* and *B*, respectively. The but-2-enamide units are all-*trans* and also nearly planar (r.m.s. deviations are 0.046 Å for molecule *A* and 0.042 Å for molecule *B*, for all non-H atoms including the directly neighboring quinazoline C atom). Their mean planes are inclined to the quinazoline ring by 47.8 (2) and 47.3 (2)° in molecules *A* and *B*, respectively. The chlorofluoroaniline rings are also twisted out of the mean plane of

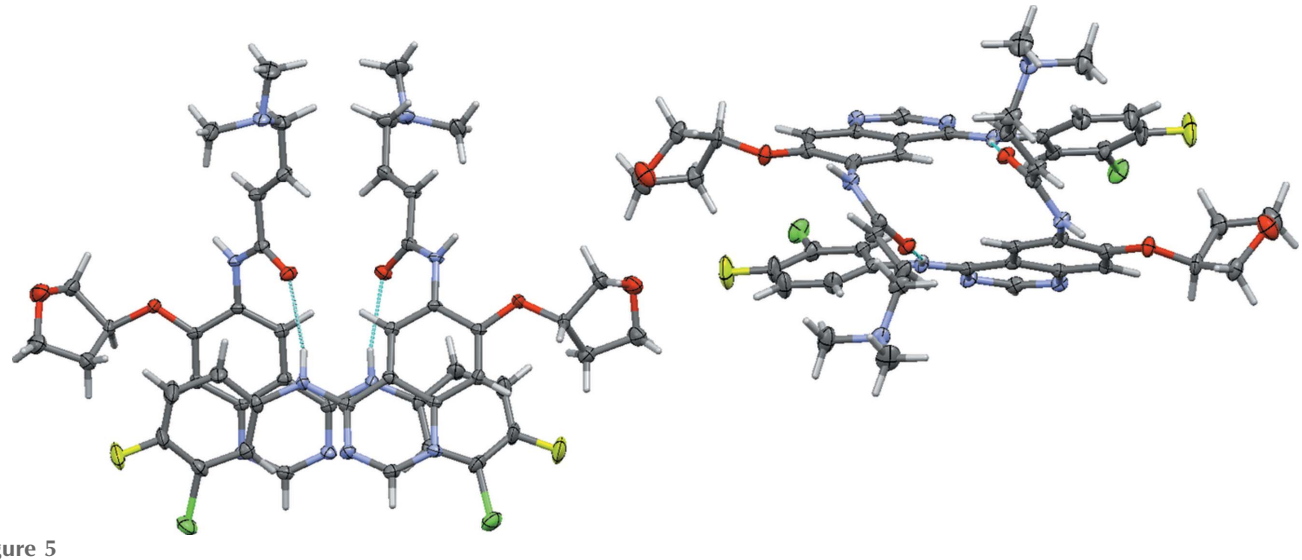


**Figure 5**
Hydrogen-bonded twofold rotation dimer of *A* molecules, in two oblique views.

the quinazoline ring by 36.6 (2) and 36.9 (1)° for molecules *A* and *B*, respectively.

The simulated powder pattern of the acetonitrile–water solvate reported here does not agree with any of the free base forms of afatinib A–D reported in the literature (Gidwani *et al.*, 2012).

## 3. Supramolecular features

In the crystal of the title compound, neighboring molecules are connected *via* N—H···O hydrogen bonds between the secondary amine and the amide keto O atom (see Table 1 for details). Molecules are connected through pairwise hydrogen

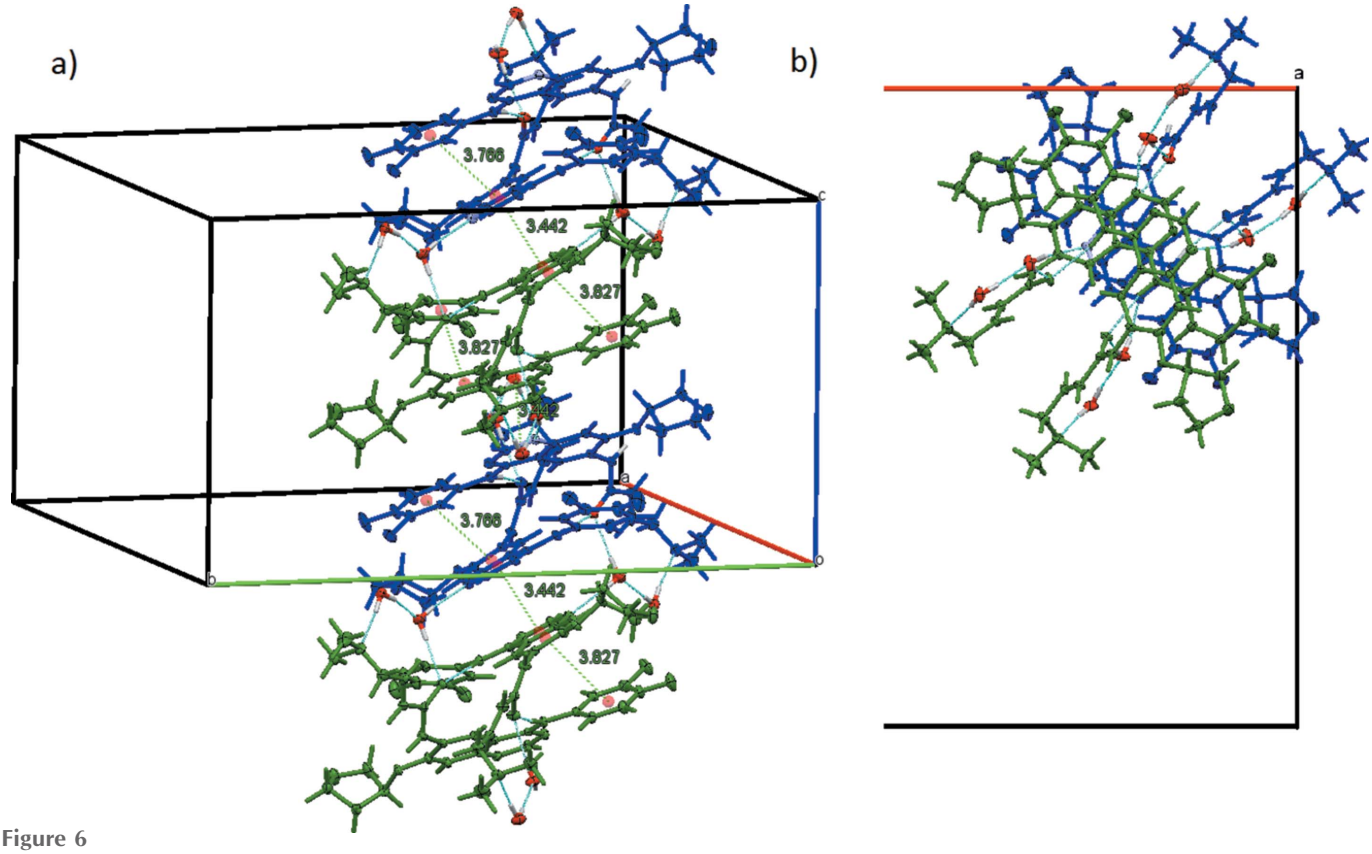



**Figure 6**
Stacks along the *c*-axis direction connected through hydrogen bonds (blue dashed lines) and $\pi$–$\pi$ stacking interactions (green dashed lines). (*a*) Side view with distances (Å) between ring centroids (red spheres) involved in $\pi$–$\pi$ stacking. (*b*) Top view down the *c* axis. Colour code: molecule *A* blue, molecule *B* green.

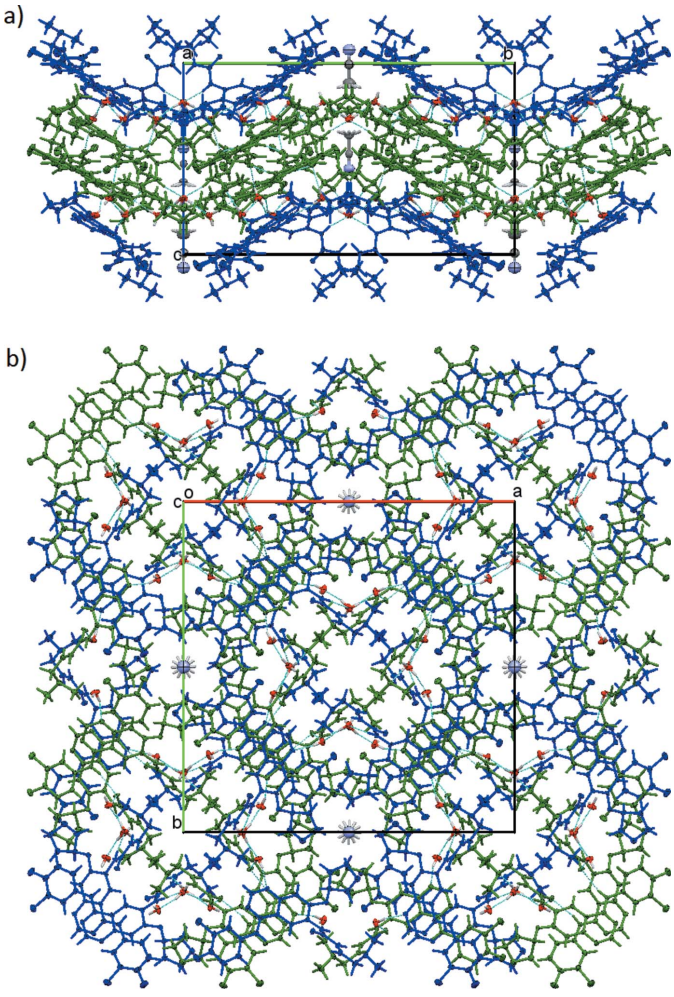



**Figure 7**
Crystal packing viewed along the *a* axis, showing the interdigitation of parallel stacks along the *c*-axis direction (see Fig. 4) and the hydrogen bonds (dashed lines) connecting them, as well as acetonitrile occupied and empty channels at the *A*- and *B*-faces and the center of the unit cell, respectively.

bonds, graph-set motif $R_2^2 18$, to their symmetry-related counterparts, creating twofold rotation symmetric dimers of *A* and *B* molecules, respectively (Fig. 5). Individual *A*–*A* and *B*–*B* dimers are arranged in infinite stacks along the *c*-axis direction through $\pi$-stacking interactions between the quinazoline units, and through weaker and more tilted $\pi$-stacking interactions between the annulated and the fluorochloro benzene rings (Fig. 6). Individual stacking interactions between the quinazoline units are across the pseudo-inversion centers, forming close to centrosymmetric pairs of *A*–*B* dimers, with an interplanar angle between quinazoline units of only 1.24 (11)°. The centroid to centroid distance between the pyrimidine rings of the *A* and *B* molecules is 3.442 (2) Å, the perpendicular distances between the two rings are 3.3144 (18) and 3.3113 (17) Å, with a slippage between the rings of 0.937 Å. The distance between annulated and chlorofluorobenzene rings is at 3.827 (3) Å substantially larger, and the slippage is 1.506 Å. The stacks created along the *c*-axis direction are further stabilized *via* hydrogen bonds involving the solvent water molecules. The O5 water molecule hydrogen bonds to the N1 atom of the quinazoline unit, connecting every second molecule in the infinite stacks, and the O4 water molecule hydrogen bonds to the dimethylamine N atom and water molecule O5, thus bridging the two ends of the dimethylaminobut-2-enamide units, giving the stacks additional support and stiffness (see Table 1 for details).

The four parallel stacks within each unit cell are interdigitating with each other, and are also connected through another hydrogen bond facilitated by the water molecule, which acts as a hydrogen-bonding acceptor for the amide N—H group. Additional weaker C—H···N and C—H···O interactions (Table 1) also contribute to the structure and lattice stability (see Table 1 for details). In combination, these interactions lead to an intricate three-dimensional superstructure facilitated by hydrogen bonding, $\pi$-stacking and interdigitation of molecule side arms (Fig. 7). In between the connected infinite stacks there remains some void space in the form of channels that stretch along the *c*-axis direction. Two different types of channels are found: channels along the fourfold rotational axis that are occupied by the acetonitrile solvent molecules, with the solvent molecules situated on that axis, and another set of parallel channels that stretch directly along the *c*-axis direction and are not occupied by any solvent.

## 4. Synthesis and crystallization

High-purity afatinib free base (>99%) was acquired from LC Labs and high-purity solvents (acetonitrile and hexanes) were procured from Sigma–Aldrich (Sigma–Aldrich, St Louis, MO, USA). Crystals suitable for single X-ray diffraction studies were obtained by vapor diffusion (Spingler *et al.*, 2012), where afatinib free base was solubilized in acetonitrile and exposed to vapor of hexanes in a closed system.

## 5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The structure exhibits pseudo-inversion symmetry emulating the space group *P*4/*ncc*, with two independent molecules, indicated by label suffixes *A* and *B*, in the asymmetric unit that are nearly related by a pseudo-inversion center. Exact inversion symmetry is not realized, as evidenced by the BASF value for racemic twinning, which refined to 0.02 (1). Exact inversion symmetry is broken by flipping of the THF ring, exchanging O and $CH_2$ units, and by a different disorder pattern for the tetrahydrofuranyl substituents of the two independent molecules.

For the first molecule, suffix *A*, the two disordered moieties differ mostly by the position of the tetrahydrofuranyl O atom, which forms the flap of the THF's envelope conformation, and is bent to opposite sides for the two moieties. The ether O atom and the THF C atoms are only slightly shifted between the two disordered moieties. For molecule *B*, the disorder is more pronounced and extends to the ether oxygen atom. The THF ring is mirror imaged between the two disordered units,

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $2C_{24}H_{25}ClFN_5O_3{\cdot}0.5C_2H_3N{\cdot}4H_2O$ |
| $M_r$ | 1064.47 |
| Crystal system, space group | Tetragonal, $P42_12$ |
| Temperature (K) | 100 |
| $a$, $c$ (Å) | 26.2427 (4), 15.1639 (3) |
| $V$ (Å$^3$) | 10443.1 (4) |
| $Z$ | 8 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 1.75 |
| Crystal size (mm) | 0.35 × 0.15 × 0.11 |
| | |
| Data collection | |
| Diffractometer | Rigaku Rapid II curved image plate |
| Absorption correction | Multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997) |
| $T_{min}$, $T_{max}$ | 0.681, 0.831 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 50059, 9958, 7679 |
| $R_{int}$ | 0.056 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.617 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.049, 0.122, 1.03 |
| No. of reflections | 9958 |
| No. of parameters | 797 |
| No. of restraints | 414 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.30, −0.40 |
| Absolute structure | Flack $x$ determined using 2765 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013) |
| Absolute structure parameter | 0.02 (1) |

Computer programs: *HKL-3000* (Otwinowski & Minor, 1997), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *SHELXLE* (Hübschle *et al.*, 2011), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

swapping the positions of the O atom with that of a methylene group and shifting the two units against each other.

All four THF moieties were restrained to have similar geometries (SAME commands of *SHELXL2016*; Sheldrick, 2015), and $U_{ij}$ components of the anisotropic displacement parameters were restrained to be similar for disordered atoms closer to each other than 1.7 Å (SIMU commands of *SHELXL2016*; Sheldrick, 2015). The occupancy ratio refined to 0.718 (9):0.282 (9) for moieties *A* and *C*, and to 0.787 (5):0.213 (5) for moieties *B* and *D*.

The water H atoms were located in difference Fourier maps and refined with a distance restraint of O—H = 0.84 (2) Å, and C- and N-bound H atoms were placed in calculated positions and treated as riding, with C—H = 0.95–0.99 Å and N—H = 0.88 Å, and with $U_{iso}$(H) = 1.5$U_{eq}$(O,C-methyl) and 1.2$U_{eq}$(C,N) for other H atoms. Acetonitrile molecules are located on fourfold axes and the H atoms are fourfold disordered by symmetry.

## Acknowledgements

The authors acknowledge funding for this work provided by the Sao Paulo Research Foundation (FAPESP) (grant Nos. 2015/15456-5 and 2015/05685-7).

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# supporting information

*Acta Cryst.* (2017). E**73**, 417-422 [https://doi.org/10.1107/S2056989017002626]

## A new solvate of afatinib, a specific inhibitor of the ErbB family of tyrosine kinases

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### Computing details

Data collection: *HKL-3000* (Otwinowski & Minor, 1997); cell refinement: *HKL-3000* (Otwinowski & Minor, 1997); data reduction: *HKL-3000* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015) and *SHELXLE* (Hübschle *et al.*, 2011); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

***N*-{4-(3-Chloro-4-fluoroanilino)-7-[(tetrahydrofuran-3-yl)oxy]quinazolin-6-yl}-4-(dimethylamino)but-2-enamide–acetonitrile–water (2/1/8)**

*Crystal data*

$2C_{24}H_{25}ClFN_5O_3\cdot0.5C_2H_3N\cdot4H_2O$
$M_r$ = 1064.47
Tetragonal, $P42_12$
$a$ = 26.2427 (4) Å
$c$ = 15.1639 (3) Å
$V$ = 10443.1 (4) Å$^3$
$Z$ = 8
$F(000)$ = 4472
$D_x$ = 1.354 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 50059 reflections
$\theta$ = 3.4–72.1°
$\mu$ = 1.75 mm$^{-1}$
$T$ = 100 K
Needle, colorless
0.35 × 0.15 × 0.11 mm

*Data collection*

Rigaku Rapid II curved image plate diffractometer
Radiation source: microfocus X-ray tube
Laterally graded multilayer (Goebel) mirror monochromator
$\omega$ scans
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)
$T_{min}$ = 0.681, $T_{max}$ = 0.831
50059 measured reflections
9958 independent reflections
7679 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.056
$\theta_{max}$ = 72.1°, $\theta_{min}$ = 3.4°
$h$ = −32→29
$k$ = −26→32
$l$ = −17→18

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.122
$S$ = 1.03
9958 reflections
797 parameters
414 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 7.7055P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{min} = -0.40$ e Å$^{-3}$

Extinction correction: (SHELXL2016; Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00033 (4)
Absolute structure: Flack *x* determined using 2765 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.02 (1)

*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**. The structure exhibits pseudo-inversion symmetry emulating the space group P4/ncc. Exact inversion symmetry is not realized (BASF value for racemic twinning 0.020 (10)).
Two tetrahydrofuran rings (related by pseudo-inversion) are disordered in differing ways. For one the ring is inverted at the oxygen. For the other the ring is mirror imaged swapping the position of the oxygen atom. All four moieties were restrained to have similar geometries, and Uij components of ADPs were restrained to be similar for disordered atoms closer to each other than 1.7 Angstrom. Occupancy ratios refined to 0.718 (9) to 0.282 (9) for moieties A and C, and to 0.787 (5) to 0.213 (5) for moieties B and D.
Water H atom O-H bond lengths were restrained to 0.84 (2) Angstrom.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Cl1A | 0.54599 (6) | 0.23028 (6) | 0.07047 (8) | 0.0360 (4) | |
| F1A | 0.53997 (14) | 0.11926 (14) | 0.0581 (2) | 0.0539 (11) | |
| N1A | 0.75215 (15) | 0.32939 (16) | −0.2041 (2) | 0.0181 (9) | |
| N2A | 0.69450 (16) | 0.27266 (17) | −0.1303 (2) | 0.0203 (10) | |
| N3A | 0.72103 (16) | 0.19492 (16) | −0.0728 (2) | 0.0184 (10) | |
| H3A | 0.746818 | 0.173820 | −0.066170 | 0.022* | |
| C1A | 0.7073 (2) | 0.3151 (2) | −0.1742 (3) | 0.0201 (12) | |
| H1A | 0.680162 | 0.338100 | −0.185363 | 0.024* | |
| C2A | 0.7321 (2) | 0.24006 (19) | −0.1137 (3) | 0.0168 (11) | |
| C3A | 0.78385 (19) | 0.25115 (19) | −0.1379 (3) | 0.0170 (12) | |
| C4A | 0.82582 (19) | 0.22136 (19) | −0.1152 (3) | 0.0153 (11) | |
| H4A | 0.820572 | 0.190478 | −0.083829 | 0.018* | |
| N4A | 0.91692 (16) | 0.20715 (16) | −0.1080 (2) | 0.0166 (10) | |
| H4A1 | 0.941274 | 0.200066 | −0.145975 | 0.020* | |
| C5A | 0.87443 (19) | 0.2355 (2) | −0.1371 (3) | 0.0165 (11) | |
| N5A | 1.04992 (17) | 0.12854 (18) | 0.1606 (3) | 0.0254 (11) | |
| C6A | 0.88205 (19) | 0.2798 (2) | −0.1896 (3) | 0.0166 (11) | |
| C7A | 0.84157 (19) | 0.31025 (19) | −0.2122 (3) | 0.0170 (11) | |
| H7A | 0.846941 | 0.340077 | −0.246439 | 0.020* | |
| C8A | 0.79197 (19) | 0.2972 (2) | −0.1844 (2) | 0.0148 (11) | |
| C9A | 0.6739 (2) | 0.1778 (2) | −0.0399 (3) | 0.0202 (12) | |
| C10A | 0.6657 (2) | 0.1251 (2) | −0.0379 (3) | 0.0286 (13) | |
| H10A | 0.691093 | 0.102591 | −0.059468 | 0.034* | |
| C11A | 0.6204 (3) | 0.1056 (2) | −0.0044 (4) | 0.0401 (16) | |

| | | | | | |
|---|---|---|---|---|---|
| H11A | 0.614608 | 0.069887 | −0.003718 | 0.048* | |
| C12A | 0.5843 (2) | 0.1383 (2) | 0.0276 (4) | 0.0350 (15) | |
| C13A | 0.5921 (2) | 0.1902 (2) | 0.0279 (3) | 0.0271 (13) | |
| C14A | 0.6371 (2) | 0.2107 (2) | −0.0059 (3) | 0.0218 (12) | |
| H14A | 0.642506 | 0.246471 | −0.005889 | 0.026* | |
| O2A | 0.89023 (13) | 0.20073 (14) | 0.03517 (19) | 0.0215 (8) | |
| C15A | 0.92164 (19) | 0.1904 (2) | −0.0238 (3) | 0.0185 (12) | |
| C16A | 0.96642 (19) | 0.1581 (2) | −0.0071 (3) | 0.0193 (12) | |
| H16A | 0.990825 | 0.153465 | −0.052699 | 0.023* | |
| C17A | 0.9736 (2) | 0.1354 (2) | 0.0697 (3) | 0.0226 (13) | |
| H17A | 0.948054 | 0.139789 | 0.113545 | 0.027* | |
| C18A | 1.0186 (2) | 0.1035 (2) | 0.0928 (3) | 0.0273 (14) | |
| H18A | 1.006874 | 0.069948 | 0.114942 | 0.033* | |
| H18B | 1.039407 | 0.097553 | 0.039344 | 0.033* | |
| C19A | 1.0761 (2) | 0.1733 (2) | 0.1246 (4) | 0.0365 (15) | |
| H19A | 1.101101 | 0.162373 | 0.080490 | 0.055* | |
| H19B | 1.093584 | 0.191439 | 0.172349 | 0.055* | |
| H19C | 1.051144 | 0.196023 | 0.096980 | 0.055* | |
| C20A | 1.0865 (2) | 0.0933 (2) | 0.1990 (4) | 0.0406 (15) | |
| H20A | 1.109638 | 0.081026 | 0.152897 | 0.061* | |
| H20B | 1.068379 | 0.064377 | 0.225073 | 0.061* | |
| H20C | 1.106198 | 0.110843 | 0.244765 | 0.061* | |
| O1A | 0.93145 (13) | 0.28774 (13) | −0.2136 (2) | 0.0206 (8) | |
| C21A | 0.9410 (6) | 0.3289 (5) | −0.2758 (17) | 0.022 (2) | 0.718 (9) |
| H21A | 0.915222 | 0.328831 | −0.324386 | 0.027* | 0.718 (9) |
| C22A | 0.9429 (4) | 0.3805 (5) | −0.2302 (6) | 0.028 (2) | 0.718 (9) |
| H22A | 0.913750 | 0.402123 | −0.248009 | 0.034* | 0.718 (9) |
| H22B | 0.942657 | 0.376599 | −0.165226 | 0.034* | 0.718 (9) |
| C23A | 0.9922 (4) | 0.4027 (4) | −0.2612 (6) | 0.035 (2) | 0.718 (9) |
| H23A | 0.987716 | 0.420478 | −0.318291 | 0.042* | 0.718 (9) |
| H23B | 1.005922 | 0.427143 | −0.217496 | 0.042* | 0.718 (9) |
| O3A | 1.0252 (2) | 0.3599 (2) | −0.2706 (4) | 0.0374 (16) | 0.718 (9) |
| C24A | 0.9951 (6) | 0.3219 (4) | −0.3124 (14) | 0.028 (2) | 0.718 (9) |
| H24A | 1.008122 | 0.287436 | −0.298384 | 0.034* | 0.718 (9) |
| H24B | 0.995381 | 0.326476 | −0.377212 | 0.034* | 0.718 (9) |
| C21C | 0.9418 (16) | 0.3249 (13) | −0.281 (5) | 0.024 (3) | 0.282 (9) |
| H21C | 0.919160 | 0.318416 | −0.332591 | 0.029* | 0.282 (9) |
| C22C | 0.9382 (9) | 0.3802 (12) | −0.2548 (16) | 0.026 (3) | 0.282 (9) |
| H22E | 0.907338 | 0.395998 | −0.280376 | 0.031* | 0.282 (9) |
| H22F | 0.936849 | 0.383724 | −0.189820 | 0.031* | 0.282 (9) |
| C23C | 0.9853 (9) | 0.4050 (8) | −0.2906 (15) | 0.032 (3) | 0.282 (9) |
| H23E | 0.975859 | 0.433007 | −0.331212 | 0.039* | 0.282 (9) |
| H23F | 1.005744 | 0.419462 | −0.241821 | 0.039* | 0.282 (9) |
| O3C | 1.0136 (5) | 0.3679 (5) | −0.3358 (10) | 0.037 (3) | 0.282 (9) |
| C24C | 0.9974 (16) | 0.3185 (9) | −0.309 (4) | 0.029 (3) | 0.282 (9) |
| H24E | 1.018320 | 0.306284 | −0.258484 | 0.035* | 0.282 (9) |
| H24F | 1.000348 | 0.293884 | −0.357681 | 0.035* | 0.282 (9) |
| O4A | 0.99752 (15) | 0.18082 (17) | 0.2950 (2) | 0.0274 (9) | |

| | | | | | |
|---|---|---|---|---|---|
| H4C | 1.011 (2) | 0.164 (2) | 0.253 (3) | 0.041* | |
| H4D | 0.9765 (17) | 0.2009 (18) | 0.269 (3) | 0.041* | |
| O5A | 0.92301 (15) | 0.23351 (18) | 0.2047 (2) | 0.0360 (10) | |
| H5C | 0.912 (2) | 0.226 (2) | 0.154 (2) | 0.054* | |
| H5D | 0.8972 (16) | 0.246 (2) | 0.228 (4) | 0.054* | |
| N6A | 0.500000 | 0.000000 | 0.0706 (10) | 0.100 (5) | |
| C25A | 0.500000 | 0.000000 | −0.0068 (10) | 0.065 (5) | |
| C26A | 0.500000 | 0.000000 | −0.1044 (10) | 0.096 (6) | |
| H26A | 0.470993 | −0.018658 | −0.125464 | 0.144* | 0.25 |
| H26B | 0.530662 | −0.015792 | −0.125464 | 0.144* | 0.25 |
| H26C | 0.498346 | 0.034450 | −0.125464 | 0.144* | 0.25 |
| Cl1B | 0.95744 (6) | 0.26892 (6) | −0.55731 (8) | 0.0386 (4) | |
| F1B | 0.96334 (12) | 0.37984 (13) | −0.5399 (2) | 0.0437 (9) | |
| N1B | 0.74716 (16) | 0.16513 (17) | −0.3001 (2) | 0.0194 (10) | |
| N2B | 0.80584 (16) | 0.22306 (17) | −0.3705 (2) | 0.0202 (10) | |
| N3B | 0.77931 (16) | 0.30174 (16) | −0.4234 (2) | 0.0167 (9) | |
| H3B | 0.753408 | 0.322621 | −0.430670 | 0.020* | |
| C1B | 0.7923 (2) | 0.1797 (2) | −0.3279 (3) | 0.0211 (12) | |
| H1B | 0.819331 | 0.156553 | −0.316512 | 0.025* | |
| C2B | 0.7685 (2) | 0.25621 (19) | −0.3862 (3) | 0.0167 (11) | |
| C3B | 0.71662 (19) | 0.24443 (19) | −0.3633 (3) | 0.0143 (11) | |
| C4B | 0.67449 (19) | 0.27542 (19) | −0.3855 (3) | 0.0170 (11) | |
| H4B | 0.679762 | 0.306750 | −0.415510 | 0.020* | |
| N4B | 0.58392 (16) | 0.28961 (16) | −0.3930 (2) | 0.0197 (10) | |
| H4B1 | 0.559066 | 0.295893 | −0.355643 | 0.024* | |
| C5B | 0.6261 (2) | 0.2604 (2) | −0.3638 (3) | 0.0175 (11) | |
| N5B | 0.45204 (17) | 0.37462 (18) | −0.6582 (2) | 0.0253 (11) | |
| C6B | 0.6177 (2) | 0.2159 (2) | −0.3129 (3) | 0.0223 (12) | |
| C7B | 0.6581 (2) | 0.1847 (2) | −0.2923 (3) | 0.0204 (12) | |
| H7B | 0.652526 | 0.154174 | −0.260097 | 0.024* | |
| C8B | 0.7077 (2) | 0.1982 (2) | −0.3191 (3) | 0.0183 (12) | |
| C9B | 0.8275 (2) | 0.3199 (2) | −0.4520 (3) | 0.0176 (11) | |
| C10B | 0.8359 (2) | 0.3723 (2) | −0.4483 (3) | 0.0208 (11) | |
| H10B | 0.810418 | 0.394157 | −0.424751 | 0.025* | |
| C11B | 0.8815 (2) | 0.3928 (2) | −0.4789 (3) | 0.0274 (13) | |
| H11B | 0.887333 | 0.428497 | −0.476902 | 0.033* | |
| C12B | 0.9179 (2) | 0.3606 (2) | −0.5119 (3) | 0.0285 (13) | |
| C13B | 0.9105 (2) | 0.3082 (2) | −0.5159 (3) | 0.0243 (12) | |
| C14B | 0.8650 (2) | 0.2880 (2) | −0.4860 (3) | 0.0214 (12) | |
| H14B | 0.859325 | 0.252258 | −0.488762 | 0.026* | |
| O2B | 0.61156 (13) | 0.29856 (14) | −0.53473 (19) | 0.0209 (8) | |
| C15B | 0.58026 (19) | 0.3081 (2) | −0.4757 (3) | 0.0170 (11) | |
| C16B | 0.53511 (19) | 0.3408 (2) | −0.4920 (3) | 0.0202 (12) | |
| H16B | 0.510440 | 0.344796 | −0.446629 | 0.024* | |
| C17B | 0.5283 (2) | 0.36465 (19) | −0.5681 (3) | 0.0206 (12) | |
| H17B | 0.553618 | 0.360544 | −0.612326 | 0.025* | |
| C18B | 0.4836 (2) | 0.3974 (2) | −0.5888 (3) | 0.0272 (14) | |
| H18C | 0.462781 | 0.402146 | −0.534975 | 0.033* | |

| | | | | | |
|---|---|---|---|---|---|
| H18D | 0.495562 | 0.431380 | −0.608348 | 0.033* | |
| C19B | 0.4253 (2) | 0.3290 (2) | −0.6251 (3) | 0.0325 (14) | |
| H19D | 0.405129 | 0.313979 | −0.672842 | 0.049* | |
| H19E | 0.402741 | 0.338744 | −0.576490 | 0.049* | |
| H19F | 0.450346 | 0.304135 | −0.604111 | 0.049* | |
| C20B | 0.4154 (2) | 0.4119 (2) | −0.6936 (4) | 0.0407 (15) | |
| H20D | 0.396854 | 0.396662 | −0.743090 | 0.061* | |
| H20E | 0.433746 | 0.442196 | −0.713900 | 0.061* | |
| H20F | 0.391252 | 0.421513 | −0.647204 | 0.061* | |
| O1B | 0.56935 (19) | 0.2103 (2) | −0.2827 (3) | 0.0193 (12) | 0.787 (5) |
| C21B | 0.5595 (2) | 0.1713 (3) | −0.2175 (4) | 0.0215 (14) | 0.787 (5) |
| H21B | 0.588006 | 0.168742 | −0.173961 | 0.026* | 0.787 (5) |
| C22B | 0.5094 (2) | 0.1847 (3) | −0.1725 (4) | 0.0226 (14) | 0.787 (5) |
| H22C | 0.509418 | 0.173841 | −0.109982 | 0.027* | 0.787 (5) |
| H22D | 0.502461 | 0.221710 | −0.175706 | 0.027* | 0.787 (5) |
| C23B | 0.4716 (3) | 0.1550 (3) | −0.2253 (5) | 0.0273 (15) | 0.787 (5) |
| H23C | 0.440105 | 0.148926 | −0.190939 | 0.033* | 0.787 (5) |
| H23D | 0.462678 | 0.173237 | −0.280321 | 0.033* | 0.787 (5) |
| O3B | 0.49710 (19) | 0.10754 (19) | −0.2447 (3) | 0.0357 (13) | 0.787 (5) |
| C24B | 0.5490 (3) | 0.1204 (3) | −0.2617 (5) | 0.0354 (17) | 0.787 (5) |
| H24C | 0.555014 | 0.122991 | −0.325968 | 0.042* | 0.787 (5) |
| H24D | 0.571903 | 0.093880 | −0.237446 | 0.042* | 0.787 (5) |
| O1D | 0.5663 (7) | 0.1987 (7) | −0.3159 (11) | 0.025 (4) | 0.213 (5) |
| C21D | 0.5525 (7) | 0.1519 (9) | −0.2718 (12) | 0.026 (3) | 0.213 (5) |
| H21E | 0.570895 | 0.122722 | −0.299762 | 0.032* | 0.213 (5) |
| C22D | 0.5634 (7) | 0.1525 (10) | −0.1741 (12) | 0.030 (3) | 0.213 (5) |
| H22G | 0.586501 | 0.180850 | −0.158334 | 0.036* | 0.213 (5) |
| H22H | 0.578862 | 0.119913 | −0.154763 | 0.036* | 0.213 (5) |
| C23D | 0.5114 (7) | 0.1598 (9) | −0.1336 (12) | 0.029 (3) | 0.213 (5) |
| H23G | 0.510087 | 0.144714 | −0.073751 | 0.035* | 0.213 (5) |
| H23H | 0.502886 | 0.196451 | −0.129409 | 0.035* | 0.213 (5) |
| O3D | 0.4770 (6) | 0.1344 (8) | −0.1910 (11) | 0.032 (3) | 0.213 (5) |
| C24D | 0.4952 (7) | 0.1431 (10) | −0.2782 (12) | 0.030 (3) | 0.213 (5) |
| H24G | 0.478318 | 0.173350 | −0.304025 | 0.036* | 0.213 (5) |
| H24H | 0.487987 | 0.113276 | −0.316080 | 0.036* | 0.213 (5) |
| O4B | 0.50270 (15) | 0.32327 (17) | −0.7952 (2) | 0.0268 (9) | |
| H4E | 0.489 (2) | 0.340 (2) | −0.752 (3) | 0.040* | |
| H4F | 0.5294 (15) | 0.3086 (19) | −0.774 (3) | 0.040* | |
| O5B | 0.58067 (15) | 0.27400 (17) | −0.7083 (2) | 0.0304 (9) | |
| H5E | 0.589 (2) | 0.284 (2) | −0.657 (2) | 0.046* | |
| H5F | 0.6065 (16) | 0.262 (2) | −0.736 (3) | 0.046* | |
| N6B | 1.000000 | 0.500000 | −0.4494 (7) | 0.073 (4) | |
| C25B | 1.000000 | 0.500000 | −0.5259 (8) | 0.039 (3) | |
| C26B | 1.000000 | 0.500000 | −0.6212 (7) | 0.046 (3) | |
| H26D | 1.029352 | 0.481890 | −0.642284 | 0.069* | 0.25 |
| H26E | 1.001008 | 0.534475 | −0.642284 | 0.069* | 0.25 |
| H26F | 0.969640 | 0.483635 | −0.642284 | 0.069* | 0.25 |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1A | 0.0283 (8) | 0.0446 (9) | 0.0350 (6) | 0.0075 (7) | 0.0128 (6) | 0.0002 (6) |
| F1A | 0.040 (2) | 0.046 (2) | 0.076 (2) | −0.0179 (18) | 0.0315 (19) | 0.0005 (19) |
| N1A | 0.016 (2) | 0.020 (2) | 0.0188 (17) | 0.0039 (18) | 0.0012 (16) | 0.0001 (17) |
| N2A | 0.019 (3) | 0.022 (3) | 0.0201 (18) | 0.004 (2) | −0.0011 (17) | 0.0057 (18) |
| N3A | 0.015 (2) | 0.020 (2) | 0.0203 (17) | 0.001 (2) | 0.0018 (17) | 0.0050 (17) |
| C1A | 0.023 (3) | 0.019 (3) | 0.018 (2) | 0.005 (2) | 0.000 (2) | 0.000 (2) |
| C2A | 0.017 (3) | 0.020 (3) | 0.0126 (18) | 0.002 (2) | −0.0013 (19) | −0.0014 (18) |
| C3A | 0.017 (3) | 0.019 (3) | 0.015 (2) | 0.002 (2) | 0.0008 (19) | 0.0001 (18) |
| C4A | 0.017 (3) | 0.018 (3) | 0.0109 (19) | 0.001 (2) | −0.0007 (18) | −0.0006 (18) |
| N4A | 0.014 (2) | 0.020 (2) | 0.0154 (17) | 0.0072 (19) | 0.0011 (16) | 0.0038 (16) |
| C5A | 0.017 (3) | 0.017 (3) | 0.0150 (19) | 0.007 (2) | 0.0028 (18) | 0.0031 (19) |
| N5A | 0.020 (3) | 0.029 (3) | 0.028 (2) | 0.006 (2) | −0.0035 (19) | 0.009 (2) |
| C6A | 0.015 (3) | 0.017 (3) | 0.018 (2) | 0.004 (2) | 0.0012 (19) | −0.0011 (19) |
| C7A | 0.019 (3) | 0.016 (3) | 0.017 (2) | −0.002 (2) | −0.0002 (19) | 0.0025 (18) |
| C8A | 0.013 (3) | 0.016 (3) | 0.0152 (19) | 0.003 (2) | −0.0012 (18) | −0.0030 (18) |
| C9A | 0.019 (3) | 0.026 (3) | 0.0152 (19) | −0.004 (2) | 0.001 (2) | 0.002 (2) |
| C10A | 0.032 (3) | 0.020 (3) | 0.034 (2) | 0.001 (3) | 0.010 (2) | −0.005 (2) |
| C11A | 0.044 (4) | 0.025 (4) | 0.051 (3) | −0.012 (3) | 0.019 (3) | −0.003 (3) |
| C12A | 0.027 (3) | 0.034 (4) | 0.044 (3) | −0.010 (3) | 0.018 (3) | 0.003 (3) |
| C13A | 0.019 (3) | 0.037 (4) | 0.025 (2) | 0.001 (3) | 0.007 (2) | 0.001 (2) |
| C14A | 0.023 (3) | 0.026 (3) | 0.016 (2) | −0.001 (2) | 0.001 (2) | 0.001 (2) |
| O2A | 0.018 (2) | 0.028 (2) | 0.0180 (14) | 0.0041 (17) | 0.0020 (14) | 0.0017 (15) |
| C15A | 0.015 (3) | 0.020 (3) | 0.020 (2) | 0.001 (2) | −0.001 (2) | 0.001 (2) |
| C16A | 0.020 (3) | 0.023 (3) | 0.0157 (19) | 0.008 (2) | 0.001 (2) | 0.001 (2) |
| C17A | 0.017 (3) | 0.030 (3) | 0.021 (2) | 0.004 (3) | 0.002 (2) | 0.004 (2) |
| C18A | 0.027 (3) | 0.031 (4) | 0.024 (2) | 0.011 (3) | −0.003 (2) | 0.005 (2) |
| C19A | 0.021 (3) | 0.047 (4) | 0.041 (3) | 0.000 (3) | 0.002 (3) | 0.019 (3) |
| C20A | 0.033 (4) | 0.044 (4) | 0.045 (3) | 0.009 (3) | −0.010 (3) | 0.017 (3) |
| O1A | 0.0143 (19) | 0.022 (2) | 0.0254 (16) | 0.0020 (16) | 0.0037 (14) | 0.0069 (15) |
| C21A | 0.017 (4) | 0.023 (4) | 0.027 (4) | −0.002 (3) | 0.006 (3) | 0.009 (4) |
| C22A | 0.027 (4) | 0.022 (4) | 0.035 (4) | 0.000 (3) | 0.000 (3) | 0.003 (4) |
| C23A | 0.023 (4) | 0.032 (4) | 0.050 (5) | −0.003 (3) | −0.002 (4) | −0.002 (4) |
| O3A | 0.023 (3) | 0.031 (3) | 0.058 (3) | −0.001 (2) | −0.001 (2) | −0.006 (3) |
| C24A | 0.023 (4) | 0.030 (4) | 0.031 (4) | 0.003 (3) | 0.007 (3) | 0.006 (3) |
| C21C | 0.022 (6) | 0.021 (6) | 0.028 (6) | 0.002 (6) | 0.005 (6) | 0.005 (6) |
| C22C | 0.021 (6) | 0.022 (6) | 0.035 (7) | 0.005 (5) | 0.000 (6) | 0.009 (6) |
| C23C | 0.024 (6) | 0.030 (6) | 0.043 (7) | 0.002 (5) | −0.001 (6) | 0.006 (6) |
| O3C | 0.025 (5) | 0.035 (5) | 0.050 (5) | −0.003 (5) | 0.012 (5) | 0.004 (5) |
| C24C | 0.022 (6) | 0.030 (6) | 0.035 (6) | 0.000 (6) | 0.008 (6) | 0.004 (6) |
| O4A | 0.024 (2) | 0.036 (3) | 0.0221 (16) | −0.0015 (18) | −0.0012 (17) | 0.0049 (16) |
| O5A | 0.025 (2) | 0.061 (3) | 0.0220 (17) | 0.013 (2) | 0.0019 (16) | −0.0072 (19) |
| N6A | 0.108 (9) | 0.108 (9) | 0.084 (10) | 0.000 | 0.000 | 0.000 |
| C25A | 0.068 (8) | 0.068 (8) | 0.060 (9) | 0.000 | 0.000 | 0.000 |
| C26A | 0.110 (10) | 0.110 (10) | 0.067 (10) | 0.000 | 0.000 | 0.000 |
| Cl1B | 0.0316 (8) | 0.0372 (9) | 0.0470 (7) | 0.0096 (7) | 0.0191 (6) | 0.0103 (6) |

| | | | | | | |
|---|---|---|---|---|---|---|
| F1B | 0.026 (2) | 0.044 (2) | 0.0611 (19) | −0.0100 (16) | 0.0132 (16) | 0.0082 (17) |
| N1B | 0.018 (3) | 0.022 (2) | 0.0182 (17) | 0.0003 (19) | −0.0031 (16) | 0.0021 (17) |
| N2B | 0.016 (2) | 0.021 (3) | 0.0237 (19) | 0.0019 (19) | 0.0009 (17) | 0.0036 (18) |
| N3B | 0.014 (2) | 0.015 (2) | 0.0209 (17) | −0.0010 (19) | 0.0018 (16) | 0.0016 (17) |
| C1B | 0.016 (3) | 0.022 (3) | 0.025 (2) | 0.003 (2) | −0.002 (2) | 0.007 (2) |
| C2B | 0.018 (3) | 0.019 (3) | 0.0125 (18) | 0.001 (2) | −0.0006 (19) | −0.0004 (18) |
| C3B | 0.016 (3) | 0.014 (3) | 0.013 (2) | 0.000 (2) | −0.0005 (18) | −0.0001 (17) |
| C4B | 0.018 (3) | 0.016 (3) | 0.017 (2) | −0.001 (2) | −0.0005 (19) | 0.0025 (19) |
| N4B | 0.015 (2) | 0.028 (3) | 0.0164 (17) | 0.005 (2) | 0.0022 (17) | 0.0016 (17) |
| C5B | 0.019 (3) | 0.018 (3) | 0.015 (2) | 0.004 (2) | −0.0008 (19) | −0.0008 (19) |
| N5B | 0.022 (3) | 0.031 (3) | 0.0231 (19) | 0.007 (2) | −0.0067 (19) | 0.0055 (19) |
| C6B | 0.018 (3) | 0.026 (3) | 0.023 (2) | −0.005 (2) | 0.003 (2) | 0.005 (2) |
| C7B | 0.019 (3) | 0.022 (3) | 0.020 (2) | 0.001 (2) | 0.002 (2) | 0.005 (2) |
| C8B | 0.021 (3) | 0.019 (3) | 0.0145 (19) | 0.004 (2) | 0.0005 (19) | 0.0056 (19) |
| C9B | 0.018 (3) | 0.018 (3) | 0.017 (2) | −0.002 (2) | 0.0013 (19) | 0.0031 (19) |
| C10B | 0.019 (3) | 0.020 (3) | 0.024 (2) | −0.003 (2) | −0.0003 (19) | 0.0019 (19) |
| C11B | 0.029 (3) | 0.020 (3) | 0.033 (2) | −0.007 (2) | 0.003 (2) | 0.003 (2) |
| C12B | 0.023 (3) | 0.033 (4) | 0.030 (2) | −0.009 (3) | 0.004 (2) | 0.004 (2) |
| C13B | 0.026 (3) | 0.023 (3) | 0.024 (2) | 0.005 (2) | 0.003 (2) | 0.005 (2) |
| C14B | 0.024 (3) | 0.020 (3) | 0.020 (2) | −0.001 (2) | 0.001 (2) | 0.002 (2) |
| O2B | 0.015 (2) | 0.030 (2) | 0.0173 (14) | 0.0087 (16) | 0.0007 (14) | −0.0002 (15) |
| C15B | 0.012 (3) | 0.020 (3) | 0.019 (2) | −0.001 (2) | −0.0009 (19) | 0.000 (2) |
| C16B | 0.016 (3) | 0.024 (3) | 0.020 (2) | 0.004 (2) | 0.001 (2) | 0.000 (2) |
| C17B | 0.020 (3) | 0.019 (3) | 0.023 (2) | 0.004 (2) | −0.005 (2) | −0.003 (2) |
| C18B | 0.027 (3) | 0.030 (4) | 0.024 (2) | 0.011 (3) | −0.003 (2) | −0.001 (2) |
| C19B | 0.024 (3) | 0.036 (4) | 0.038 (3) | 0.002 (3) | −0.007 (3) | 0.009 (3) |
| C20B | 0.037 (4) | 0.043 (4) | 0.042 (3) | 0.016 (3) | −0.014 (3) | 0.009 (3) |
| O1B | 0.013 (2) | 0.023 (3) | 0.022 (3) | 0.001 (2) | 0.006 (2) | 0.008 (2) |
| C21B | 0.019 (3) | 0.022 (3) | 0.024 (3) | 0.002 (3) | −0.002 (2) | 0.010 (3) |
| C22B | 0.020 (3) | 0.026 (3) | 0.022 (3) | −0.002 (3) | 0.000 (2) | 0.005 (2) |
| C23B | 0.023 (3) | 0.028 (4) | 0.032 (3) | −0.004 (3) | 0.003 (3) | −0.003 (3) |
| O3B | 0.029 (3) | 0.025 (3) | 0.053 (3) | −0.006 (2) | 0.008 (2) | −0.004 (2) |
| C24B | 0.026 (4) | 0.033 (4) | 0.047 (3) | 0.002 (3) | 0.014 (3) | 0.000 (3) |
| O1D | 0.025 (6) | 0.023 (7) | 0.027 (7) | 0.001 (6) | 0.007 (6) | 0.006 (6) |
| C21D | 0.019 (5) | 0.027 (5) | 0.033 (5) | −0.003 (5) | 0.005 (5) | 0.005 (5) |
| C22D | 0.023 (6) | 0.033 (6) | 0.035 (6) | −0.005 (6) | 0.000 (6) | 0.006 (6) |
| C23D | 0.024 (6) | 0.032 (6) | 0.031 (6) | −0.003 (6) | −0.004 (6) | 0.001 (6) |
| O3D | 0.024 (6) | 0.036 (6) | 0.036 (6) | −0.005 (5) | 0.001 (5) | 0.003 (5) |
| C24D | 0.022 (6) | 0.031 (6) | 0.037 (6) | −0.001 (5) | −0.002 (5) | 0.005 (5) |
| O4B | 0.019 (2) | 0.039 (3) | 0.0230 (16) | −0.0001 (18) | −0.0041 (17) | −0.0031 (16) |
| O5B | 0.021 (2) | 0.047 (3) | 0.0224 (17) | 0.0058 (19) | −0.0002 (15) | −0.0103 (17) |
| N6B | 0.086 (7) | 0.086 (7) | 0.048 (7) | 0.000 | 0.000 | 0.000 |
| C25B | 0.037 (5) | 0.037 (5) | 0.043 (6) | 0.000 | 0.000 | 0.000 |
| C26B | 0.050 (5) | 0.050 (5) | 0.039 (6) | 0.000 | 0.000 | 0.000 |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1A—C13A | 1.729 (6) | F1B—C12B | 1.362 (6) |
| F1A—C12A | 1.349 (6) | N1B—C1B | 1.315 (6) |
| N1A—C1A | 1.315 (6) | N1B—C8B | 1.380 (6) |
| N1A—C8A | 1.377 (6) | N2B—C2B | 1.332 (6) |
| N2A—C2A | 1.329 (6) | N2B—C1B | 1.355 (6) |
| N2A—C1A | 1.340 (6) | N3B—C2B | 1.351 (6) |
| N3A—C2A | 1.368 (6) | N3B—C9B | 1.420 (6) |
| N3A—C9A | 1.407 (6) | N3B—H3B | 0.8800 |
| N3A—H3A | 0.8800 | C1B—H1B | 0.9500 |
| C1A—H1A | 0.9500 | C2B—C3B | 1.439 (7) |
| C2A—C3A | 1.437 (7) | C3B—C8B | 1.407 (6) |
| C3A—C4A | 1.394 (6) | C3B—C4B | 1.413 (7) |
| C3A—C8A | 1.415 (6) | C4B—C5B | 1.369 (7) |
| C4A—C5A | 1.369 (6) | C4B—H4B | 0.9500 |
| C4A—H4A | 0.9500 | N4B—C15B | 1.348 (5) |
| N4A—C15A | 1.356 (5) | N4B—C5B | 1.417 (6) |
| N4A—C5A | 1.411 (6) | N4B—H4B1 | 0.8800 |
| N4A—H4A1 | 0.8800 | C5B—C6B | 1.418 (7) |
| C5A—C6A | 1.424 (6) | N5B—C18B | 1.466 (6) |
| N5A—C20A | 1.454 (7) | N5B—C20B | 1.472 (7) |
| N5A—C19A | 1.467 (6) | N5B—C19B | 1.474 (6) |
| N5A—C18A | 1.472 (6) | C6B—O1B | 1.358 (7) |
| C6A—O1A | 1.363 (5) | C6B—C7B | 1.375 (7) |
| C6A—C7A | 1.373 (6) | C6B—O1D | 1.424 (17) |
| C7A—C8A | 1.411 (7) | C7B—C8B | 1.410 (7) |
| C7A—H7A | 0.9500 | C7B—H7B | 0.9500 |
| C9A—C14A | 1.396 (7) | C9B—C14B | 1.390 (7) |
| C9A—C10A | 1.399 (7) | C9B—C10B | 1.396 (7) |
| C10A—C11A | 1.389 (8) | C10B—C11B | 1.391 (7) |
| C10A—H10A | 0.9500 | C10B—H10B | 0.9500 |
| C11A—C12A | 1.367 (8) | C11B—C12B | 1.370 (7) |
| C11A—H11A | 0.9500 | C11B—H11B | 0.9500 |
| C12A—C13A | 1.378 (8) | C12B—C13B | 1.391 (8) |
| C13A—C14A | 1.393 (7) | C13B—C14B | 1.383 (7) |
| C14A—H14A | 0.9500 | C14B—H14B | 0.9500 |
| O2A—C15A | 1.246 (6) | O2B—C15B | 1.241 (5) |
| C15A—C16A | 1.471 (7) | C15B—C16B | 1.483 (7) |
| C16A—C17A | 1.322 (6) | C16B—C17B | 1.325 (6) |
| C16A—H16A | 0.9500 | C16B—H16B | 0.9500 |
| C17A—C18A | 1.491 (7) | C17B—C18B | 1.489 (7) |
| C17A—H17A | 0.9500 | C17B—H17B | 0.9500 |
| C18A—H18A | 0.9900 | C18B—H18C | 0.9900 |
| C18A—H18B | 0.9900 | C18B—H18D | 0.9900 |
| C19A—H19A | 0.9800 | C19B—H19D | 0.9800 |
| C19A—H19B | 0.9800 | C19B—H19E | 0.9800 |
| C19A—H19C | 0.9800 | C19B—H19F | 0.9800 |

| | | | |
|---|---|---|---|
| C20A—H20A | 0.9800 | C20B—H20D | 0.9800 |
| C20A—H20B | 0.9800 | C20B—H20E | 0.9800 |
| C20A—H20C | 0.9800 | C20B—H20F | 0.9800 |
| O1A—C21C | 1.43 (6) | O1B—C21B | 1.445 (7) |
| O1A—C21A | 1.456 (19) | C21B—C24B | 1.520 (9) |
| C21A—C22A | 1.522 (12) | C21B—C22B | 1.521 (8) |
| C21A—C24A | 1.534 (10) | C21B—H21B | 1.0000 |
| C21A—H21A | 1.0000 | C22B—C23B | 1.495 (8) |
| C22A—C23A | 1.495 (10) | C22B—H22C | 0.9900 |
| C22A—H22A | 0.9900 | C22B—H22D | 0.9900 |
| C22A—H22B | 0.9900 | C23B—O3B | 1.444 (8) |
| C23A—O3A | 1.426 (9) | C23B—H23C | 0.9900 |
| C23A—H23A | 0.9900 | C23B—H23D | 0.9900 |
| C23A—H23B | 0.9900 | O3B—C24B | 1.427 (8) |
| O3A—C24A | 1.423 (15) | C24B—H24C | 0.9900 |
| C24A—H24A | 0.9900 | C24B—H24D | 0.9900 |
| C24A—H24B | 0.9900 | O1D—C21D | 1.445 (19) |
| C21C—C22C | 1.51 (2) | C21D—C22D | 1.508 (19) |
| C21C—C24C | 1.53 (2) | C21D—C24D | 1.524 (19) |
| C21C—H21C | 1.0000 | C21D—H21E | 1.0000 |
| C22C—C23C | 1.497 (19) | C22D—C23D | 1.509 (19) |
| C22C—H22E | 0.9900 | C22D—H22G | 0.9900 |
| C22C—H22F | 0.9900 | C22D—H22H | 0.9900 |
| C23C—O3C | 1.403 (18) | C23D—O3D | 1.420 (18) |
| C23C—H23E | 0.9900 | C23D—H23G | 0.9900 |
| C23C—H23F | 0.9900 | C23D—H23H | 0.9900 |
| O3C—C24C | 1.43 (2) | O3D—C24D | 1.424 (18) |
| C24C—H24E | 0.9900 | C24D—H24G | 0.9900 |
| C24C—H24F | 0.9900 | C24D—H24H | 0.9900 |
| O4A—H4C | 0.87 (3) | O4B—H4E | 0.87 (3) |
| O4A—H4D | 0.86 (3) | O4B—H4F | 0.86 (3) |
| O5A—H5C | 0.84 (3) | O5B—H5E | 0.85 (3) |
| O5A—H5D | 0.84 (3) | O5B—H5F | 0.85 (3) |
| N6A—C25A | 1.174 (17) | N6B—C25B | 1.161 (14) |
| C25A—C26A | 1.48 (2) | C25B—C26B | 1.444 (14) |
| C26A—H26A | 0.9600 | C26B—H26D | 0.9600 |
| C26A—H26B | 0.9600 | C26B—H26E | 0.9600 |
| C26A—H26C | 0.9600 | C26B—H26F | 0.9600 |
| C26A—H26A$^{i}$ | 0.9600 | C26B—H26D$^{iv}$ | 0.9600 |
| C26A—H26A$^{ii}$ | 0.9600 | C26B—H26D$^{v}$ | 0.9600 |
| C26A—H26A$^{iii}$ | 0.9600 | C26B—H26D$^{vi}$ | 0.9600 |
| C26A—H26B$^{i}$ | 0.9600 | C26B—H26E$^{iv}$ | 0.9600 |
| C26A—H26B$^{ii}$ | 0.9600 | C26B—H26E$^{v}$ | 0.9600 |
| C26A—H26B$^{iii}$ | 0.9600 | C26B—H26E$^{vi}$ | 0.9600 |
| C26A—H26C$^{i}$ | 0.9599 | C26B—H26F$^{iv}$ | 0.9599 |
| C26A—H26C$^{ii}$ | 0.9599 | C26B—H26F$^{v}$ | 0.9599 |
| C26A—H26C$^{iii}$ | 0.9599 | C26B—H26F$^{vi}$ | 0.9599 |
| Cl1B—C13B | 1.725 (5) | | |

| | | | |
|---|---|---|---|
| C1A—N1A—C8A | 115.3 (4) | C2B—N2B—C1B | 116.1 (4) |
| C2A—N2A—C1A | 116.2 (4) | C2B—N3B—C9B | 127.7 (5) |
| C2A—N3A—C9A | 128.6 (5) | C2B—N3B—H3B | 116.2 |
| C2A—N3A—H3A | 115.7 | C9B—N3B—H3B | 116.2 |
| C9A—N3A—H3A | 115.7 | N1B—C1B—N2B | 129.3 (5) |
| N1A—C1A—N2A | 129.3 (5) | N1B—C1B—H1B | 115.3 |
| N1A—C1A—H1A | 115.3 | N2B—C1B—H1B | 115.3 |
| N2A—C1A—H1A | 115.3 | N2B—C2B—N3B | 119.8 (5) |
| N2A—C2A—N3A | 119.1 (5) | N2B—C2B—C3B | 120.9 (5) |
| N2A—C2A—C3A | 121.5 (4) | N3B—C2B—C3B | 119.3 (5) |
| N3A—C2A—C3A | 119.4 (5) | C8B—C3B—C4B | 118.7 (5) |
| C4A—C3A—C8A | 118.9 (5) | C8B—C3B—C2B | 117.2 (5) |
| C4A—C3A—C2A | 124.8 (5) | C4B—C3B—C2B | 124.0 (4) |
| C8A—C3A—C2A | 116.3 (4) | C5B—C4B—C3B | 120.2 (5) |
| C5A—C4A—C3A | 121.6 (5) | C5B—C4B—H4B | 119.9 |
| C5A—C4A—H4A | 119.2 | C3B—C4B—H4B | 119.9 |
| C3A—C4A—H4A | 119.2 | C15B—N4B—C5B | 122.8 (4) |
| C15A—N4A—C5A | 122.5 (4) | C15B—N4B—H4B1 | 118.6 |
| C15A—N4A—H4A1 | 118.7 | C5B—N4B—H4B1 | 118.6 |
| C5A—N4A—H4A1 | 118.7 | C4B—C5B—N4B | 119.6 (5) |
| C4A—C5A—N4A | 121.2 (4) | C4B—C5B—C6B | 120.7 (5) |
| C4A—C5A—C6A | 119.2 (5) | N4B—C5B—C6B | 119.6 (5) |
| N4A—C5A—C6A | 119.6 (4) | C18B—N5B—C20B | 111.0 (4) |
| C20A—N5A—C19A | 110.4 (4) | C18B—N5B—C19B | 110.8 (4) |
| C20A—N5A—C18A | 111.4 (4) | C20B—N5B—C19B | 110.6 (4) |
| C19A—N5A—C18A | 111.1 (4) | O1B—C6B—C7B | 125.4 (5) |
| O1A—C6A—C7A | 125.5 (5) | O1B—C6B—C5B | 114.8 (5) |
| O1A—C6A—C5A | 114.1 (4) | C7B—C6B—C5B | 119.7 (5) |
| C7A—C6A—C5A | 120.4 (5) | C7B—C6B—O1D | 123.2 (9) |
| C6A—C7A—C8A | 119.9 (5) | C5B—C6B—O1D | 113.0 (9) |
| C6A—C7A—H7A | 120.1 | C6B—C7B—C8B | 119.8 (5) |
| C8A—C7A—H7A | 120.1 | C6B—C7B—H7B | 120.1 |
| N1A—C8A—C7A | 119.1 (4) | C8B—C7B—H7B | 120.1 |
| N1A—C8A—C3A | 121.2 (5) | N1B—C8B—C3B | 121.2 (5) |
| C7A—C8A—C3A | 119.7 (5) | N1B—C8B—C7B | 118.4 (4) |
| C14A—C9A—C10A | 119.7 (5) | C3B—C8B—C7B | 120.4 (5) |
| C14A—C9A—N3A | 122.8 (5) | C14B—C9B—C10B | 119.8 (5) |
| C10A—C9A—N3A | 117.4 (5) | C14B—C9B—N3B | 122.8 (5) |
| C11A—C10A—C9A | 120.3 (5) | C10B—C9B—N3B | 117.3 (5) |
| C11A—C10A—H10A | 119.8 | C11B—C10B—C9B | 120.2 (5) |
| C9A—C10A—H10A | 119.8 | C11B—C10B—H10B | 119.9 |
| C12A—C11A—C10A | 119.4 (6) | C9B—C10B—H10B | 119.9 |
| C12A—C11A—H11A | 120.3 | C12B—C11B—C10B | 119.0 (5) |
| C10A—C11A—H11A | 120.3 | C12B—C11B—H11B | 120.5 |
| F1A—C12A—C11A | 119.2 (6) | C10B—C11B—H11B | 120.5 |
| F1A—C12A—C13A | 119.6 (5) | F1B—C12B—C11B | 119.7 (5) |
| C11A—C12A—C13A | 121.3 (5) | F1B—C12B—C13B | 118.4 (5) |

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| C12A—C13A—C14A | 120.4 (5) | C11B—C12B—C13B | 121.8 (5) |
| C12A—C13A—Cl1A | 119.9 (4) | C14B—C13B—C12B | 119.1 (5) |
| C14A—C13A—Cl1A | 119.7 (4) | C14B—C13B—Cl1B | 120.5 (4) |
| C13A—C14A—C9A | 118.9 (5) | C12B—C13B—Cl1B | 120.4 (4) |
| C13A—C14A—H14A | 120.5 | C13B—C14B—C9B | 120.1 (5) |
| C9A—C14A—H14A | 120.5 | C13B—C14B—H14B | 120.0 |
| O2A—C15A—N4A | 123.0 (5) | C9B—C14B—H14B | 120.0 |
| O2A—C15A—C16A | 122.0 (4) | O2B—C15B—N4B | 123.4 (5) |
| N4A—C15A—C16A | 114.9 (4) | O2B—C15B—C16B | 121.7 (4) |
| C17A—C16A—C15A | 121.6 (5) | N4B—C15B—C16B | 114.9 (4) |
| C17A—C16A—H16A | 119.2 | C17B—C16B—C15B | 121.7 (4) |
| C15A—C16A—H16A | 119.2 | C17B—C16B—H16B | 119.2 |
| C16A—C17A—C18A | 124.9 (5) | C15B—C16B—H16B | 119.2 |
| C16A—C17A—H17A | 117.5 | C16B—C17B—C18B | 124.2 (5) |
| C18A—C17A—H17A | 117.5 | C16B—C17B—H17B | 117.9 |
| N5A—C18A—C17A | 110.8 (5) | C18B—C17B—H17B | 117.9 |
| N5A—C18A—H18A | 109.5 | N5B—C18B—C17B | 111.1 (4) |
| C17A—C18A—H18A | 109.5 | N5B—C18B—H18C | 109.4 |
| N5A—C18A—H18B | 109.5 | C17B—C18B—H18C | 109.4 |
| C17A—C18A—H18B | 109.5 | N5B—C18B—H18D | 109.4 |
| H18A—C18A—H18B | 108.1 | C17B—C18B—H18D | 109.4 |
| N5A—C19A—H19A | 109.5 | H18C—C18B—H18D | 108.0 |
| N5A—C19A—H19B | 109.5 | N5B—C19B—H19D | 109.5 |
| H19A—C19A—H19B | 109.5 | N5B—C19B—H19E | 109.5 |
| N5A—C19A—H19C | 109.5 | H19D—C19B—H19E | 109.5 |
| H19A—C19A—H19C | 109.5 | N5B—C19B—H19F | 109.5 |
| H19B—C19A—H19C | 109.5 | H19D—C19B—H19F | 109.5 |
| N5A—C20A—H20A | 109.5 | H19E—C19B—H19F | 109.5 |
| N5A—C20A—H20B | 109.5 | N5B—C20B—H20D | 109.5 |
| H20A—C20A—H20B | 109.5 | N5B—C20B—H20E | 109.5 |
| N5A—C20A—H20C | 109.5 | H20D—C20B—H20E | 109.5 |
| H20A—C20A—H20C | 109.5 | N5B—C20B—H20F | 109.5 |
| H20B—C20A—H20C | 109.5 | H20D—C20B—H20F | 109.5 |
| C6A—O1A—C21C | 118.2 (19) | H20E—C20B—H20F | 109.5 |
| C6A—O1A—C21A | 116.7 (8) | C6B—O1B—C21B | 118.4 (5) |
| O1A—C21A—C22A | 111.8 (16) | O1B—C21B—C24B | 110.6 (6) |
| O1A—C21A—C24A | 107.7 (13) | O1B—C21B—C22B | 107.4 (5) |
| C22A—C21A—C24A | 103.9 (9) | C24B—C21B—C22B | 104.1 (5) |
| O1A—C21A—H21A | 111.0 | O1B—C21B—H21B | 111.5 |
| C22A—C21A—H21A | 111.0 | C24B—C21B—H21B | 111.5 |
| C24A—C21A—H21A | 111.0 | C22B—C21B—H21B | 111.5 |
| C23A—C22A—C21A | 103.4 (7) | C23B—C22B—C21B | 102.3 (5) |
| C23A—C22A—H22A | 111.1 | C23B—C22B—H22C | 111.3 |
| C21A—C22A—H22A | 111.1 | C21B—C22B—H22C | 111.3 |
| C23A—C22A—H22B | 111.1 | C23B—C22B—H22D | 111.3 |
| C21A—C22A—H22B | 111.1 | C21B—C22B—H22D | 111.3 |
| H22A—C22A—H22B | 109.0 | H22C—C22B—H22D | 109.2 |
| O3A—C23A—C22A | 104.5 (7) | O3B—C23B—C22B | 104.5 (5) |

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| O3A—C23A—H23A | 110.8 | O3B—C23B—H23C | 110.8 |
| C22A—C23A—H23A | 110.8 | C22B—C23B—H23C | 110.8 |
| O3A—C23A—H23B | 110.8 | O3B—C23B—H23D | 110.8 |
| C22A—C23A—H23B | 110.8 | C22B—C23B—H23D | 110.8 |
| H23A—C23A—H23B | 108.9 | H23C—C23B—H23D | 108.9 |
| C24A—O3A—C23A | 105.0 (8) | C24B—O3B—C23B | 106.0 (5) |
| O3A—C24A—C21A | 105.6 (11) | O3B—C24B—C21B | 107.5 (5) |
| O3A—C24A—H24A | 110.6 | O3B—C24B—H24C | 110.2 |
| C21A—C24A—H24A | 110.6 | C21B—C24B—H24C | 110.2 |
| O3A—C24A—H24B | 110.6 | O3B—C24B—H24D | 110.2 |
| C21A—C24A—H24B | 110.6 | C21B—C24B—H24D | 110.2 |
| H24A—C24A—H24B | 108.7 | H24C—C24B—H24D | 108.5 |
| O1A—C21C—C22C | 117 (4) | C6B—O1D—C21D | 119.5 (15) |
| O1A—C21C—C24C | 108 (4) | O1D—C21D—C22D | 113.5 (19) |
| C22C—C21C—C24C | 103.6 (18) | O1D—C21D—C24D | 110.3 (17) |
| O1A—C21C—H21C | 109.4 | C22D—C21D—C24D | 104.5 (13) |
| C22C—C21C—H21C | 109.4 | O1D—C21D—H21E | 109.4 |
| C24C—C21C—H21C | 109.4 | C22D—C21D—H21E | 109.4 |
| C23C—C22C—C21C | 105.9 (16) | C24D—C21D—H21E | 109.4 |
| C23C—C22C—H22E | 110.6 | C21D—C22D—C23D | 103.3 (14) |
| C21C—C22C—H22E | 110.6 | C21D—C22D—H22G | 111.1 |
| C23C—C22C—H22F | 110.6 | C23D—C22D—H22G | 111.1 |
| C21C—C22C—H22F | 110.6 | C21D—C22D—H22H | 111.1 |
| H22E—C22C—H22F | 108.7 | C23D—C22D—H22H | 111.1 |
| O3C—C23C—C22C | 108.2 (15) | H22G—C22D—H22H | 109.1 |
| O3C—C23C—H23E | 110.0 | O3D—C23D—C22D | 105.4 (14) |
| C22C—C23C—H23E | 110.0 | O3D—C23D—H23G | 110.7 |
| O3C—C23C—H23F | 110.0 | C22D—C23D—H23G | 110.7 |
| C22C—C23C—H23F | 110.0 | O3D—C23D—H23H | 110.7 |
| H23E—C23C—H23F | 108.4 | C22D—C23D—H23H | 110.7 |
| C23C—O3C—C24C | 109.3 (17) | H23G—C23D—H23H | 108.8 |
| O3C—C24C—C21C | 105 (2) | C23D—O3D—C24D | 106.3 (14) |
| O3C—C24C—H24E | 110.7 | O3D—C24D—C21D | 107.2 (14) |
| C21C—C24C—H24E | 110.7 | O3D—C24D—H24G | 110.3 |
| O3C—C24C—H24F | 110.7 | C21D—C24D—H24G | 110.3 |
| C21C—C24C—H24F | 110.7 | O3D—C24D—H24H | 110.3 |
| H24E—C24C—H24F | 108.8 | C21D—C24D—H24H | 110.3 |
| H4C—O4A—H4D | 104 (5) | H24G—C24D—H24H | 108.5 |
| H5C—O5A—H5D | 101 (6) | H4E—O4B—H4F | 107 (5) |
| N6A—C25A—C26A | 180.0 | H5E—O5B—H5F | 110 (5) |
| C25A—C26A—H26A | 109.5 | N6B—C25B—C26B | 180.0 |
| C25A—C26A—H26B | 109.5 | C25B—C26B—H26D | 109.5 |
| H26A—C26A—H26B | 109.5 | C25B—C26B—H26E | 109.5 |
| C25A—C26A—H26C | 109.5 | H26D—C26B—H26E | 109.5 |
| H26A—C26A—H26C | 109.5 | C25B—C26B—H26F | 109.5 |
| H26B—C26A—H26C | 109.5 | H26D—C26B—H26F | 109.5 |
| C25A—C26A—H26A$^{i}$ | 109.471 (1) | H26E—C26B—H26F | 109.5 |
| C25A—C26A—H26A$^{ii}$ | 109.5 | C25B—C26B—H26D$^{iv}$ | 109.469 (1) |

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| C25A—C26A—H26A$^{iii}$ | 109.471 (1) | C25B—C26B—H26D$^{v}$ | 109.469 (1) |
| C25A—C26A—H26B$^{i}$ | 109.5 | C25B—C26B—H26D$^{vi}$ | 109.469 (2) |
| H26A$^{i}$—C26A—H26B$^{i}$ | 109.5 | C25B—C26B—H26E$^{iv}$ | 109.467 (2) |
| C25A—C26A—H26B$^{ii}$ | 109.471 (1) | H26D$^{iv}$—C26B—H26E$^{iv}$ | 109.5 |
| H26A$^{ii}$—C26A—H26B$^{ii}$ | 109.5 | C25B—C26B—H26E$^{v}$ | 109.467 (1) |
| C25A—C26A—H26B$^{iii}$ | 109.471 (1) | H26D$^{v}$—C26B—H26E$^{v}$ | 109.5 |
| H26A$^{iii}$—C26A—H26B$^{iii}$ | 109.5 | C25B—C26B—H26E$^{vi}$ | 109.467 (1) |
| C25A—C26A—H26C$^{i}$ | 109.473 (1) | H26D$^{vi}$—C26B—H26E$^{vi}$ | 109.5 |
| H26A$^{i}$—C26A—H26C$^{i}$ | 109.5 | C25B—C26B—H26F$^{iv}$ | 109.470 (1) |
| H26B$^{i}$—C26A—H26C$^{i}$ | 109.5 | H26D$^{iv}$—C26B—H26F$^{iv}$ | 109.5 |
| C25A—C26A—H26C$^{ii}$ | 109.5 | H26E$^{iv}$—C26B—H26F$^{iv}$ | 109.5 |
| H26A$^{ii}$—C26A—H26C$^{ii}$ | 109.5 | C25B—C26B—H26F$^{v}$ | 109.470 (2) |
| H26B$^{ii}$—C26A—H26C$^{ii}$ | 109.5 | H26D$^{v}$—C26B—H26F$^{v}$ | 109.5 |
| C25A—C26A—H26C$^{iii}$ | 109.5 | H26E$^{v}$—C26B—H26F$^{v}$ | 109.5 |
| H26A$^{iii}$—C26A—H26C$^{iii}$ | 109.5 | C25B—C26B—H26F$^{vi}$ | 109.470 (1) |
| H26B$^{iii}$—C26A—H26C$^{iii}$ | 109.5 | H26D$^{vi}$—C26B—H26F$^{vi}$ | 109.5 |
| C1B—N1B—C8B | 115.2 (4) | H26E$^{vi}$—C26B—H26F$^{vi}$ | 109.5 |

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| C8A—N1A—C1A—N2A | −3.0 (7) | C1B—N2B—C2B—N3B | 175.9 (4) |
| C2A—N2A—C1A—N1A | 0.3 (7) | C1B—N2B—C2B—C3B | −3.6 (6) |
| C1A—N2A—C2A—N3A | −176.9 (4) | C9B—N3B—C2B—N2B | 2.0 (7) |
| C1A—N2A—C2A—C3A | 3.0 (6) | C9B—N3B—C2B—C3B | −178.6 (4) |
| C9A—N3A—C2A—N2A | −3.6 (7) | N2B—C2B—C3B—C8B | 3.9 (6) |
| C9A—N3A—C2A—C3A | 176.5 (4) | N3B—C2B—C3B—C8B | −175.6 (4) |
| N2A—C2A—C3A—C4A | 173.4 (4) | N2B—C2B—C3B—C4B | −173.7 (4) |
| N3A—C2A—C3A—C4A | −6.6 (7) | N3B—C2B—C3B—C4B | 6.9 (6) |
| N2A—C2A—C3A—C8A | −3.4 (6) | C8B—C3B—C4B—C5B | 0.2 (6) |
| N3A—C2A—C3A—C8A | 176.5 (4) | C2B—C3B—C4B—C5B | 177.7 (4) |
| C8A—C3A—C4A—C5A | −0.2 (7) | C3B—C4B—C5B—N4B | −174.6 (4) |
| C2A—C3A—C4A—C5A | −177.0 (4) | C3B—C4B—C5B—C6B | 5.2 (7) |
| C3A—C4A—C5A—N4A | 175.0 (4) | C15B—N4B—C5B—C4B | 43.7 (7) |
| C3A—C4A—C5A—C6A | −4.5 (7) | C15B—N4B—C5B—C6B | −136.0 (5) |
| C15A—N4A—C5A—C4A | −45.2 (7) | C4B—C5B—C6B—O1B | 168.8 (5) |
| C15A—N4A—C5A—C6A | 134.4 (5) | N4B—C5B—C6B—O1B | −11.5 (7) |
| C4A—C5A—C6A—O1A | −174.7 (4) | C4B—C5B—C6B—C7B | −6.5 (7) |
| N4A—C5A—C6A—O1A | 5.8 (6) | N4B—C5B—C6B—C7B | 173.2 (4) |
| C4A—C5A—C6A—C7A | 5.1 (7) | C4B—C5B—C6B—O1D | −164.5 (9) |
| N4A—C5A—C6A—C7A | −174.4 (4) | N4B—C5B—C6B—O1D | 15.2 (10) |
| O1A—C6A—C7A—C8A | 178.8 (4) | O1B—C6B—C7B—C8B | −172.3 (5) |
| C5A—C6A—C7A—C8A | −1.0 (7) | C5B—C6B—C7B—C8B | 2.4 (7) |
| C1A—N1A—C8A—C7A | −178.5 (4) | O1D—C6B—C7B—C8B | 158.1 (9) |
| C1A—N1A—C8A—C3A | 2.3 (6) | C1B—N1B—C8B—C3B | −1.4 (6) |
| C6A—C7A—C8A—N1A | 177.1 (4) | C1B—N1B—C8B—C7B | 179.2 (4) |
| C6A—C7A—C8A—C3A | −3.8 (6) | C4B—C3B—C8B—N1B | 176.5 (4) |
| C4A—C3A—C8A—N1A | −176.4 (4) | C2B—C3B—C8B—N1B | −1.2 (6) |
| C2A—C3A—C8A—N1A | 0.6 (6) | C4B—C3B—C8B—C7B | −4.2 (6) |
| C4A—C3A—C8A—C7A | 4.4 (6) | C2B—C3B—C8B—C7B | 178.1 (4) |
| C2A—C3A—C8A—C7A | −178.6 (4) | C6B—C7B—C8B—N1B | −177.8 (4) |

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| C2A—N3A—C9A—C14A | −31.5 (7) |
| C2A—N3A—C9A—C10A | 151.7 (5) |
| C14A—C9A—C10A—C11A | 1.7 (8) |
| N3A—C9A—C10A—C11A | 178.6 (5) |
| C9A—C10A—C11A—C12A | −0.8 (9) |
| C10A—C11A—C12A—F1A | 178.5 (5) |
| C10A—C11A—C12A—C13A | −0.5 (9) |
| F1A—C12A—C13A—C14A | −178.0 (5) |
| C11A—C12A—C13A—C14A | 1.0 (9) |
| F1A—C12A—C13A—Cl1A | 2.0 (8) |
| C11A—C12A—C13A—Cl1A | −179.0 (5) |
| C12A—C13A—C14A—C9A | −0.1 (7) |
| Cl1A—C13A—C14A—C9A | 179.9 (4) |
| C10A—C9A—C14A—C13A | −1.2 (7) |
| N3A—C9A—C14A—C13A | −177.9 (4) |
| C5A—N4A—C15A—O2A | −3.5 (8) |
| C5A—N4A—C15A—C16A | 175.4 (4) |
| O2A—C15A—C16A—C17A | 5.0 (8) |
| N4A—C15A—C16A—C17A | −173.9 (5) |
| C15A—C16A—C17A—C18A | −178.0 (5) |
| C20A—N5A—C18A—C17A | 166.2 (4) |
| C19A—N5A—C18A—C17A | −70.3 (5) |
| C16A—C17A—C18A—N5A | 112.2 (6) |
| C7A—C6A—O1A—C21C | −12 (2) |
| C5A—C6A—O1A—C21C | 167 (2) |
| C7A—C6A—O1A—C21A | −6.8 (11) |
| C5A—C6A—O1A—C21A | 173.0 (10) |
| C6A—O1A—C21A—C22A | 81.6 (14) |
| C6A—O1A—C21A—C24A | −164.8 (10) |
| O1A—C21A—C22A—C23A | 128.0 (11) |
| C24A—C21A—C22A—C23A | 12.1 (18) |
| C21A—C22A—C23A—O3A | −33.6 (13) |
| C22A—C23A—O3A—C24A | 43.4 (10) |
| C23A—O3A—C24A—C21A | −35.0 (15) |
| O1A—C21A—C24A—O3A | −105.5 (13) |
| C22A—C21A—C24A—O3A | 13.2 (19) |
| C6A—O1A—C21C—C22C | 75 (4) |
| C6A—O1A—C21C—C24C | −168 (2) |
| O1A—C21C—C22C—C23C | 133 (3) |
| C24C—C21C—C22C—C23C | 15 (5) |
| C21C—C22C—C23C—O3C | 1 (4) |
| C22C—C23C—O3C—C24C | −19 (3) |
| C23C—O3C—C24C—C21C | 28 (5) |
| O1A—C21C—C24C—O3C | −150 (4) |
| C22C—C21C—C24C—O3C | −26 (6) |
| C6B—C7B—C8B—C3B | 2.9 (7) |
| C2B—N3B—C9B—C14B | 32.6 (7) |
| C2B—N3B—C9B—C10B | −149.8 (4) |
| C14B—C9B—C10B—C11B | 0.4 (7) |
| N3B—C9B—C10B—C11B | −177.3 (4) |
| C9B—C10B—C11B—C12B | −0.5 (7) |
| C10B—C11B—C12B—F1B | −178.2 (4) |
| C10B—C11B—C12B—C13B | 0.2 (8) |
| F1B—C12B—C13B—C14B | 178.7 (4) |
| C11B—C12B—C13B—C14B | 0.3 (8) |
| F1B—C12B—C13B—Cl1B | −1.2 (7) |
| C11B—C12B—C13B—Cl1B | −179.6 (4) |
| C12B—C13B—C14B—C9B | −0.4 (7) |
| Cl1B—C13B—C14B—C9B | 179.5 (4) |
| C10B—C9B—C14B—C13B | 0.1 (7) |
| N3B—C9B—C14B—C13B | 177.6 (4) |
| C5B—N4B—C15B—O2B | 4.9 (8) |
| C5B—N4B—C15B—C16B | −175.8 (4) |
| O2B—C15B—C16B—C17B | −5.6 (8) |
| N4B—C15B—C16B—C17B | 175.2 (5) |
| C15B—C16B—C17B—C18B | 179.2 (5) |
| C20B—N5B—C18B—C17B | −166.4 (5) |
| C19B—N5B—C18B—C17B | 70.3 (6) |
| C16B—C17B—C18B—N5B | −113.4 (6) |
| C7B—C6B—O1B—C21B | 6.0 (8) |
| C5B—C6B—O1B—C21B | −168.9 (5) |
| C6B—O1B—C21B—C24B | −86.5 (7) |
| C6B—O1B—C21B—C22B | 160.5 (5) |
| O1B—C21B—C22B—C23B | 93.5 (6) |
| C24B—C21B—C22B—C23B | −23.8 (7) |
| C21B—C22B—C23B—O3B | 38.2 (6) |
| C22B—C23B—O3B—C24B | −38.4 (7) |
| C23B—O3B—C24B—C21B | 22.5 (7) |
| O1B—C21B—C24B—O3B | −113.5 (6) |
| C22B—C21B—C24B—O3B | 1.6 (7) |
| C7B—C6B—O1D—C21D | 19 (2) |
| C5B—C6B—O1D—C21D | 176.1 (14) |
| C6B—O1D—C21D—C22D | 59 (2) |
| C6B—O1D—C21D—C24D | 175.9 (15) |
| O1D—C21D—C22D—C23D | 104 (2) |
| C24D—C21D—C22D—C23D | −16 (2) |
| C21D—C22D—C23D—O3D | 33 (2) |
| C22D—C23D—O3D—C24D | −37 (2) |
| C23D—O3D—C24D—C21D | 27 (3) |
| O1D—C21D—C24D—O3D | −128 (2) |

| | | | |
|---|---|---|---|
| C8B—N1B—C1B—N2B | 1.9 (7) | C22D—C21D—C24D—O3D | −5 (3) |
| C2B—N2B—C1B—N1B | 0.7 (7) | | |

Symmetry codes: (i) −*x*+1, −*y*, *z*; (ii) −*y*+1/2, *x*−1/2, *z*; (iii) *y*+1/2, −*x*+1/2, *z*; (iv) −*x*+2, −*y*+1, *z*; (v) −*y*+3/2, *x*−1/2, *z*; (vi) *y*+1/2, −*x*+3/2, *z*.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N4*A*—H4*A*1···O4*B*$^{vii}$ | 0.88 | 1.94 | 2.804 (5) | 166 |
| N3*A*—H3*A*···O2*A*$^{viii}$ | 0.88 | 2.22 | 3.088 (5) | 167 |
| O4*A*—H4*C*···N5*A* | 0.85 (5) | 1.97 (5) | 2.816 (6) | 173 (4) |
| O4*A*—H4*D*···O5*A* | 0.86 (5) | 1.91 (5) | 2.759 (6) | 169 (5) |
| O5*A*—H5*C*···O2*A* | 0.85 (3) | 2.00 (4) | 2.844 (4) | 174 (5) |
| O5*A*—H5*D*···N1*B*$^{viii}$ | 0.83 (5) | 1.98 (5) | 2.775 (6) | 161 (5) |
| C4*A*—H4*A*···O2*A*$^{viii}$ | 0.95 | 2.31 | 3.246 (6) | 168 |
| C16*A*—H16*A*···O4*B*$^{vii}$ | 0.95 | 2.41 | 3.183 (6) | 139 |
| C22*A*—H22*A*···O3*A*$^{vi}$ | 0.99 | 2.40 | 3.353 (13) | 162 |
| C24*A*—H24*A*···O5*B*$^{vii}$ | 0.99 | 2.50 | 3.388 (14) | 150 |
| N4*B*—H4*B*1···O4*A*$^{ix}$ | 0.88 | 1.96 | 2.820 (5) | 167 |
| N3*B*—H3*B*···O2*B*$^{x}$ | 0.88 | 2.26 | 3.123 (5) | 166 |
| O4*B*—H4*E*···N5*B* | 0.87 (5) | 1.95 (5) | 2.811 (5) | 175 (6) |
| O4*B*—H4*F*···O5*B* | 0.86 (4) | 1.91 (4) | 2.756 (5) | 169 (4) |
| O5*B*—H5*E*···O2*B* | 0.85 (4) | 1.98 (3) | 2.828 (4) | 173 (5) |
| O5*B*—H5*F*···N1*A*$^{x}$ | 0.86 (4) | 1.95 (4) | 2.794 (5) | 169 (5) |
| C4*B*—H4*B*···O2*B*$^{x}$ | 0.95 | 2.34 | 3.280 (6) | 169 |
| C16*B*—H16*B*···O4*A*$^{ix}$ | 0.95 | 2.42 | 3.197 (6) | 139 |
| C22*B*—H22*D*···O5*A*$^{ix}$ | 0.99 | 2.43 | 3.160 (8) | 130 |
| C24*B*—H24*D*···O3*B*$^{iii}$ | 0.99 | 2.57 | 3.455 (9) | 149 |

Symmetry codes: (iii) *y*+1/2, −*x*+1/2, *z*; (vi) *y*+1/2, −*x*+3/2, *z*; (vii) *x*+1/2, −*y*+1/2, −*z*−1; (viii) −*y*+1, −*x*+1, −*z*; (ix) *x*−1/2, −*y*+1/2, −*z*; (x) −*y*+1, −*x*+1, −*z*−1.