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# Crystal structures of 1-(4-chlorophenyl)-4-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carboxylic acid and 4-(4-methoxyphenyl)-1-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile

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**Keywords:** crystal structure; cyclohexene ring; dihydropyridine ring; hexahydroquinoline ring; dimer.

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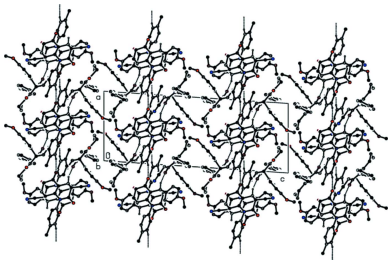
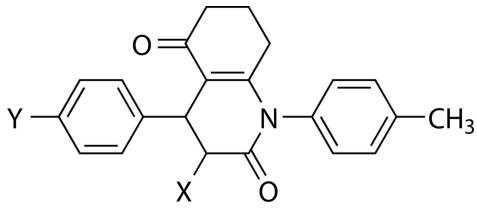
In the title compounds $C_{23}H_{21}ClN_2O_3$ [**I**, namely 1-(4-chlorophenyl)-4-(4-methylphenyl)-3,8-dioxo-1,2,5,6,7,8-hexahydroquine-3-carboxylic acid] and $C_{24}H_{22}N_2O_3$ [**II**, namely 4-(4-methoxyphenyl)-1-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile], each of the cyclohexene and dihydropyridine rings of the 1,2,5,6,7,8-hexahydroquinoline moieties adopts a twisted-boat conformation. The asymmetric units of both compounds **I** and **II** consist of two independent molecules (*A* and *B*). In **II***A*, three carbon atoms of the cyclohexene ring are disordered over two sets of sites in a 0.670 (11):0.330 (11) occupancy ratio. In the crystal of **I**, molecules are linked through classical N—H···O hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ ring motif and with their molecular planes parallel to the crystallographic (020) plane. Non-classical C—H···O hydrogen-bonding interactions connect the dimers, resulting in a three-dimensional network. In the crystal of **II**, molecules are linked by C—H···N, C—H···O and C—H···$\pi$ interactions, forming a three-dimensional network.

## 1. Chemical context

Quinoline and its derivatives have for some time attracted the attention of both synthetic and biological chemists as a result of their diverse chemical and pharmacological properties (Kumar *et al.*, 2009). There are a number of natural products bearing the quinoline skeleton that are used as a medicine or employed as lead molecule for the development of new and potent therapeutics (Venkat Reddy *et al.*, 2009). Quinoline derivatives fused with various heterocycles have already demonstrated potent anticancer activity (Afzal *et al.*, 2015). In addition, it has been found that various quinoline compounds show anti-tuberculosis (TB) activity (Muscia *et al.*, 2014), anti-inflammatory activity (Psomas & Kessissoglou, 2013), anti-convulsant effects (Guo *et al.*, 2009), and anti-malarial parasite effects (Abdel-Gawad *et al.*, 2005). Furthermore, quinolones have been proved to be very effective in many antimicrobial and antioxidant investigations (Praveen *et al.*, 2010). In this context, we report herein the crystal structures of two derivatives of hexahydroquinoline.






I: X = C(O)NH$_2$; Y = Cl
II: X = CN; Y = OCH$_3$

## 2. Structural commentary

Compound **I** crystallizes in the orthorhombic space group *Pbca* with $Z$ = 16, while compound **II** crystallizes in the monoclinic space group $P2_1/c$ with $Z$ = 8. The asymmetric units of both compounds (**I** and **II**) each comprise two molecules (*A* and *B*). As shown in Figs. 1 and 2, the cyclohexene (C4–C9) and dihydropyridine (N1/C1–C4/C9) rings of the 1,2,5,6,7,8-hexahydroquinoline moieties (N1/C1–C9) each adopt a twisted-boat conformation. The puckering parameters (Cremer & Pople, 1975) of the cyclohexene rings are $Q_T$ = 0.441 (3) Å, $\theta$ = 123.3 (3)°, and $\varphi$ = 1.3 (3)° for **I***A*, $Q_T$ = 0.450 (2) Å, $\theta$ = 122.0 (3)°, and $\varphi$ = 4.3 (3)° for **I***B*, $Q_T$ = 0.352 (8) Å, $\theta$ = 60.7 (11)°, and $\varphi$ = 188.1 (13)° for **II***A* (major component of the disorder), and $Q_T$ = 0.446 (2) Å, $\theta$ = 123.5 (3)°, and $\varphi$ = 355.2 (3)° for **II***B*. The puckering parameters of the dihydropyridine rings are $Q_T$ = 0.4929 (18) Å, $\theta$ = 64.2 (2)°, and $\varphi$ = 150.6 (2)° for **I***A*, $Q_T$ = 0.4529 (18) Å, $\theta$ = 61.1 (2)°, and $\varphi$ = 139.9 (3)° for **I***B*, $Q_T$ = 0.422 (2) Å, $\theta$ = 58.9 (3)°, and $\varphi$ = 138.4 (3)° for **II***A* and $Q_T$ = 0.437 (2) Å, $\theta$ = 62.5 (3)°, and $\varphi$ = 142.1 (3)° for **II***B*.

## 3. Supramolecular features

In the crystal of **I**, two molecules are linked by a pair of intermolecular N—H···O hydrogen bonds with an $R_2^2(8)$ ring motif (Bernstein *et al.*, 1995; Table 1), forming an inversion dimer (Fig. 3). These assemble into a three-dimensional network *via* C—H···O interactions. In the crystal of **II**, molecules are linked by non-classical C—H···O, C—H···N and C—H···$\pi$ interactions, resulting in a three-dimensional network (Table 2 and Figs. 4 and 5). In the crystals of the two compounds (**I** and **II**) $\pi$-$\pi$-stacking interactions are not observed despite the presence of two aromatic rings in every molecule.

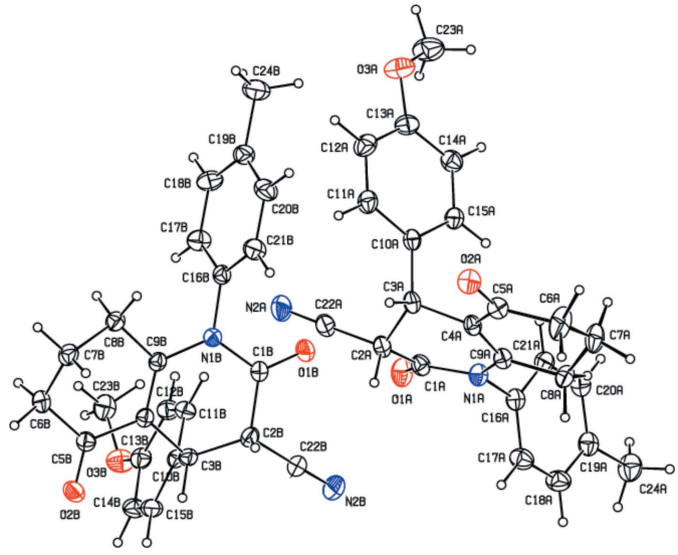



**Figure 2**
The molecular structure of **II** with the atom-numbering scheme and with displacement ellipsoids drawn at the 20% probability level.

## 4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.40, February 2019; Groom *et al.*, 2016) for the 4-phenyl-2,3,4,6,7,8-hexahydroquinolin-5(1*H*)-one moiety resulted in six closely related hits, *viz.* 2-amino-4-(4-chloro-

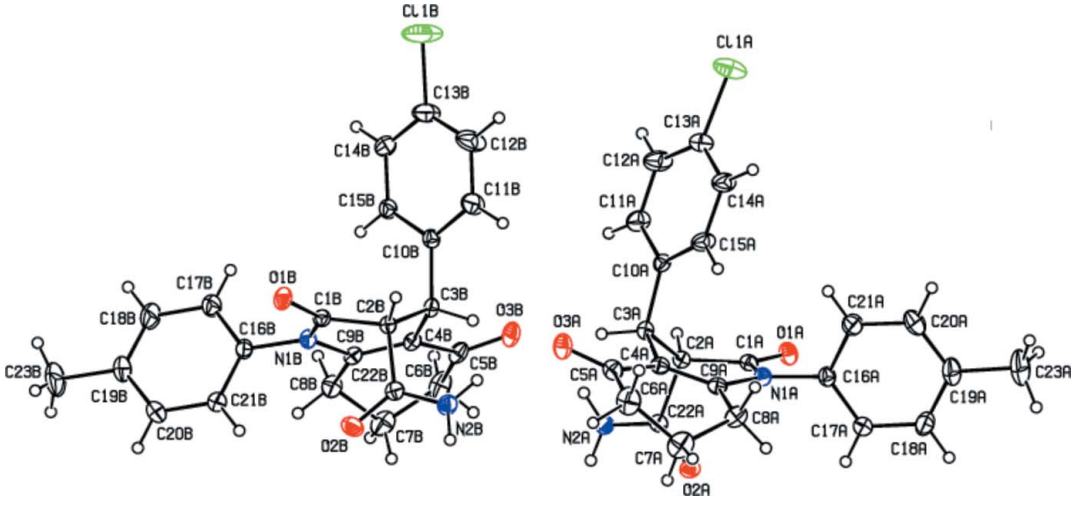



**Figure 1**
The molecular structure of **I** with the atom-numbering scheme and with displacement ellipsoids drawn at the 30% probability level.

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2*A*—H2*A*2···O2*A*$^{i}$ | 0.88 (2) | 2.12 (3) | 2.979 (2) | 164 (2) |
| N2*B*—H2*B*1···O2*A*$^{ii}$ | 0.86 (2) | 2.26 (2) | 3.055 (2) | 154 (2) |
| N2*A*—H2*A*1···O3*B* | 0.86 (2) | 2.06 (2) | 2.865 (2) | 156 (2) |
| N2*B*—H2*B*2···O3*A* | 0.85 (3) | 2.04 (3) | 2.847 (2) | 159 (2) |
| C8*A*—H8*AB*···O2*B*$^{ii}$ | 0.97 | 2.50 | 3.392 (2) | 153 |
| C6*B*—H6*BB*···O1*B*$^{iii}$ | 0.97 | 2.57 | 3.430 (2) | 148 |
| C14*A*—H14*A*···O1*A*$^{iv}$ | 0.93 | 2.54 | 3.393 (3) | 153 |
| C17*B*—H17*B*···O1*B*$^{v}$ | 0.93 | 2.52 | 3.424 (3) | 164 |

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

**Table 2**
Hydrogen-bond geometry (Å, °) for **II**.

*Cg*4 and *Cg*9 are the centroids of the C10*A*–C15*A* and C16*B*–C21*B* rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C21*A*—H21*A*···N2*B*$^{i}$ | 0.95 | 2.65 | 3.258 (4) | 123 |
| C14*B*—H14*B*···O3*A*$^{ii}$ | 0.95 | 2.65 | 3.417 (3) | 139 |
| C3*A*—H3*A*···O1*B* | 1.00 | 2.37 | 3.171 (2) | 136 |
| C23*B*—H23*E*···*Cg*9$^{iii}$ | 0.98 | 2.93 | 3.868 (3) | 160 |
| C23*B*—H23*F*···*Cg*4$^{iv}$ | 0.98 | 2.74 | 3.710 (3) | 169 |

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

phenyl)-1-(4-methylphenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (HUYVUU; Mohamed *et al.*, 2015), methyl-2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (TEJQII; Morales *et al.*, 1996), 3-acetyl-2,7,7-trimethyl-4-phenyl-1,4,5,6,7,8-hexahydro-5-quinolone (TEJQOO; Morales *et al.*, 1996), 4-(4-chlorophenyl)-8-methyl-2-oxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (AZOWAO; Asiri *et al.*, 2011), 8-methyl-2-oxo-4-(thiophen-2-yl)-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile

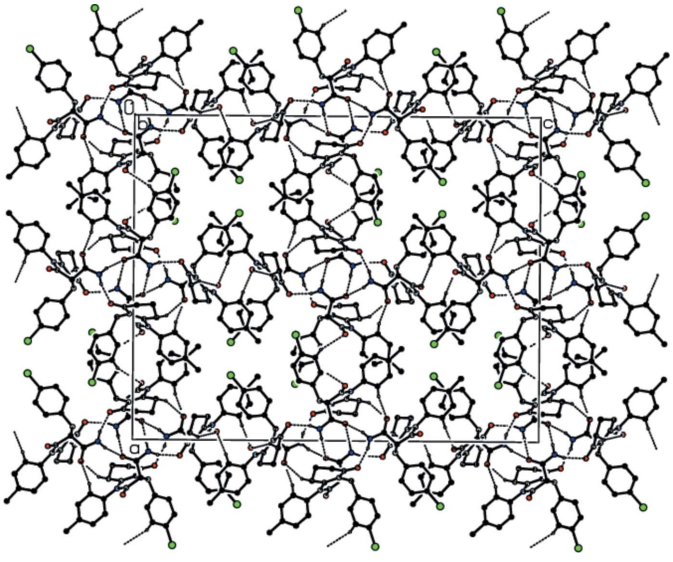


**Figure 3**
The crystal packing of **I** viewed down the *b* axis showing intermolecular hydrogen bonds as dashed lines.

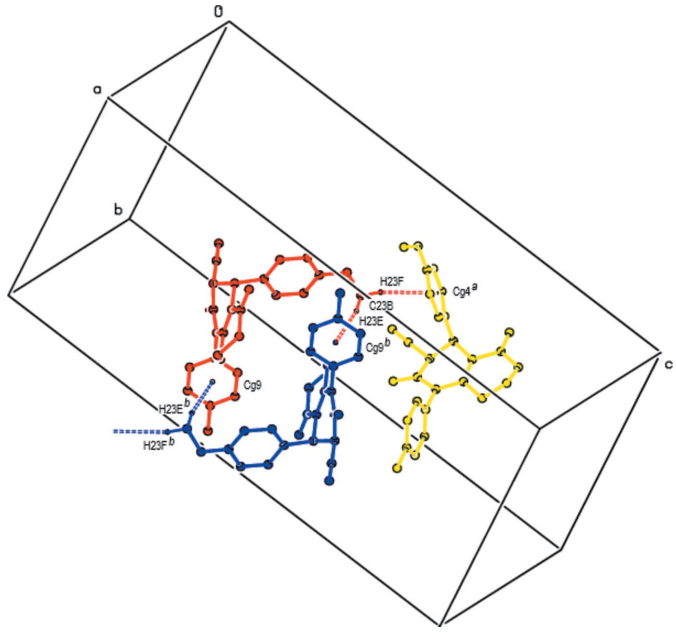



**Figure 4**
A view of the C—H···$\pi$ interactions in **II** shown as dashed lines. [Symmetry codes: (*a*) $1-x, 1-y, 1-z$; (*b*) $2-x, 1-y, 1-z$].

(XECCAL; Asiri *et al.*, 2012) and ethyl-2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (XAYVEA; Kurbanova *et al.*, 2012).

In compounds **I** and **II**, the observed bond lengths and bond angles are in good agreement with the reported experimental values as found in the structures of HUYVUU, TEJQII, TEJQOO, AZOWAO, XECCAL and XAYVEA. The metrical parameters are, hence, unremarkable.

The angles between the planes of the two benzene rings in **I** and **II** are 52.64 (11)° for **I***A*, 33.78 (12)° for **I***B*, 21.80 (11)° for **II***A* and 19.39 (11)° for **II***B*, respectively. These angles are notably distinct, even for the two independent molecules in each structure. They are all also significantly larger than the value of 11.52 (7)° found in HUYVUU (the only other example with two benzene rings amongst the related struc-

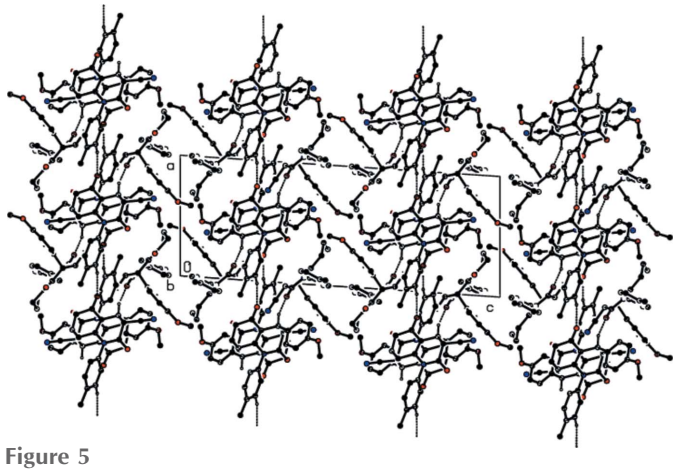


**Figure 5**
The crystal packing of **II** viewed down the *b* axis showing the intermolecular hydrogen bonds as dashed lines.

Table 3
Experimental details.

| | **II** | **II** |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{23}H_{21}ClN_2O_3$ | $C_{24}H_{22}N_2O_3$ |
| $M_r$ | 408.87 | 386.43 |
| Crystal system, space group | Orthorhombic, *Pbca* | Monoclinic, $P2_1/c$ |
| Temperature (K) | 173 | 173 |
| *a*, *b*, *c* (Å) | 27.9446 (4), 8.4311 (1), 35.0101 (5) | 10.3486 (2), 13.9969 (3), 27.5353 (5) |
| $\alpha$, $\beta$, $\gamma$ (°) | 90, 90, 90 | 90, 93.797 (2), 90 |
| *V* (Å$^3$) | 8248.51 (19) | 3979.69 (14) |
| *Z* | 16 | 8 |
| Radiation type | Cu *K*$\alpha$ | Cu *K*$\alpha$ |
| $\mu$ (mm$^{-1}$) | 1.86 | 0.69 |
| Crystal size (mm) | 0.16 × 0.10 × 0.05 | 0.12 × 0.08 × 0.04 |
| Data collection | | |
| Diffractometer | Rigaku Oxford Diffraction Xcalibur, Eos, Gemini | Rigaku Oxford Diffraction Xcalibur, Eos, Gemini |
| Absorption correction | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015) | Multi-scan (*CrysAlis PRO*; Rigaku OD, 2015) |
| $T_{min}$, $T_{max}$ | 0.614, 1.000 | 0.945, 1.000 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 64509, 7983, 6950 | 15107, 7579, 5380 |
| $R_{int}$ | 0.043 | 0.027 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.615 | 0.615 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.050, 0.132, 1.04 | 0.049, 0.139, 1.04 |
| No. of reflections | 7983 | 7579 |
| No. of parameters | 541 | 555 |
| No. of restraints | 0 | 36 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.66, −0.55 | 0.25, −0.16 |

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

tures). Intermolecular interactions can be weaker or stronger based on the presence or absence or difference of functional groups and the molecular environment, depending on the crystal system, which all affect the molecular conformation. The observed difference in the angles between the two benzene rings may be attributed to these factors.

## 5. Synthesis and crystallization

***1-(4-Chlorophenyl)-4-(4-methylphenyl)-3,8-dioxo-1,2,5,6,-7,8-hexahydroquine-3-carboxlic acid*, I**

A solution of 2-amino-4-(4-chlorophenyl)-1-(4-methylphenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (2.0 g, 0.0051 mol) in conc. $H_2SO_4$ (20 mL) was stirred for 4 h at room temperature. Then the reaction mixture was poured into ice-cold water. The formed precipitate was collected, filtered off, washed with water and recrystallized from ethanol as pale-yellow crystals; yield 73%, m.p. 518 K.

***4-(4-Methoxyphenyl)-1-(4-methyl-phenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile*, II**

To a solution of 1,3-cyclohexanedione (3.36 g, 0.03 mol) and *p*-toluidine (3.21 g, 0.03 mol) in ethanol (40 ml), a catalytic amount of triethylamine was added and the mixture was heated under reflux for 3 h. Ethyl-(2*Z*)-2-cyano-3-(4-methoxyphenyl)acrylate (6.93 g, 0.03 mol) was added to the reaction mixture while refluxing for another 3 h. The reaction mixture was then cooled to room temperature. The precipitate that formed was filtered off, dried and recrystallized from ethanol solution as orange crystals; yield 67%, m.p. 525 K.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For **I**, the hydrogen atoms of the $NH_2$ group were found in the difference-Fourier map and refined freely. All C-bound H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C-methyl). For **II**, all H atoms were positioned geometrically (C—H = 0.95–1.00 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C-methyl). For **II**, twenty reflections (4 15 10, 3 15 13, 3 14 16, 3 16 7, 3 16 8, 1 3 8, 2 3 0, $\bar{2}$ 16 3, $\bar{2}$ 4 10, 2 15 11, 0 14 8, 10 7 13, 1 16 11, 2 1 5, 3 16 4, $\bar{8}$ 13 7, $\bar{1}$ 16 4, 0 15 3, 1 16 10, $\bar{2}$ 16 11) were omitted as clear outlier data. In **II***A*, atoms C6*A*, C7*A* and C8*A* of the cyclohexane ring are disordered over two sets of sites in a 0.670 (11):0.330 (11) occupancy ratio. The coordinates and the $U^{ij}$ components of the C6*A*, C7*A*, C8*A* and the C6*AA*, C7*AA* and C8*AA* atoms were restrained using SADI and SIMU instructions.

*K* values, which are large only for weak reflections with an $F_c/F_{cmax}$ ratio less than 0.005 and less than 0.015 for **I** and **II**, respectively, were observed as 2.713 for **I** and 5.559 for **II**.

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# supporting information

*Acta Cryst.* (2021). E**77**, 351-355 [https://doi.org/10.1107/S2056989021002140]

# Crystal structures of 1-(4-chlorophenyl)-4-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carboxylic acid and 4-(4-methoxyphenyl)-1-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile

**Omyma A. Abd Allah, Manpreet Kaur, Mehmet Akkurt, Shaaban K. Mohamed, Asmaa H. A. Tamam, Sahar M. I. Elgarhy and Jerry P. Jasinski**

## Computing details

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

### 1-(4-Chlorophenyl)-4-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carboxylic acid (I)

*Crystal data*

$C_{23}H_{21}ClN_2O_3$
$M_r$ = 408.87
Orthorhombic, *Pbca*
$a$ = 27.9446 (4) Å
$b$ = 8.4311 (1) Å
$c$ = 35.0101 (5) Å
$V$ = 8248.51 (19) Å$^3$
$Z$ = 16
$F(000)$ = 3424
$D_x$ = 1.317 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 21280 reflections
$\theta$ = 4.0–71.5°
$\mu$ = 1.86 mm$^{-1}$
$T$ = 173 K
Prism, pale yellow
0.16 × 0.10 × 0.05 mm

*Data collection*

Rigaku Oxford Diffraction Xcalibur, Eos, Gemini diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2015)
$T_{min}$ = 0.614, $T_{max}$ = 1.000
64509 measured reflections
7983 independent reflections
6950 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043
$\theta_{max}$ = 71.5°, $\theta_{min}$ = 4.1°
$h$ = −29→34
$k$ = −10→6
$l$ = −42→42

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.132

$S$ = 1.04
7983 reflections
541 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 5.5774P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.66$ e Å$^{-3}$
$\Delta\rho_{min} = -0.55$ e Å$^{-3}$

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1A | 0.82611 (2) | 0.31975 (8) | 0.40005 (2) | 0.0615 (2) |
| O1A | 0.67114 (5) | 0.01077 (15) | 0.52217 (4) | 0.0322 (3) |
| O2A | 0.55412 (5) | 0.09204 (16) | 0.52328 (3) | 0.0296 (3) |
| O3A | 0.58223 (5) | 0.60354 (17) | 0.44478 (4) | 0.0410 (3) |
| N1A | 0.65291 (5) | 0.26677 (17) | 0.53641 (4) | 0.0243 (3) |
| N2A | 0.53741 (6) | 0.0761 (2) | 0.46051 (5) | 0.0317 (4) |
| C1A | 0.64987 (6) | 0.1309 (2) | 0.51400 (5) | 0.0238 (3) |
| C2A | 0.61843 (6) | 0.1434 (2) | 0.47878 (5) | 0.0226 (3) |
| H2A | 0.6294 | 0.0641 | 0.4603 | 0.027* |
| C3A | 0.62358 (6) | 0.3088 (2) | 0.46017 (5) | 0.0240 (3) |
| H3AA | 0.5998 | 0.3194 | 0.4397 | 0.029* |
| C4A | 0.61353 (6) | 0.4304 (2) | 0.49046 (5) | 0.0249 (4) |
| C5A | 0.59223 (7) | 0.5801 (2) | 0.47852 (6) | 0.0312 (4) |
| C6A | 0.58550 (10) | 0.7071 (3) | 0.50809 (7) | 0.0469 (5) |
| H6AA | 0.6119 | 0.7813 | 0.5065 | 0.056* |
| H6AB | 0.5563 | 0.7649 | 0.5025 | 0.056* |
| C7A | 0.58272 (10) | 0.6436 (3) | 0.54805 (7) | 0.0498 (6) |
| H7AA | 0.5526 | 0.5879 | 0.5513 | 0.060* |
| H7AB | 0.5832 | 0.7316 | 0.5659 | 0.060* |
| C8A | 0.62403 (8) | 0.5307 (2) | 0.55737 (5) | 0.0340 (4) |
| H8AA | 0.6535 | 0.5905 | 0.5596 | 0.041* |
| H8AB | 0.6180 | 0.4790 | 0.5817 | 0.041* |
| C9A | 0.62920 (6) | 0.4077 (2) | 0.52666 (5) | 0.0241 (3) |
| C10A | 0.67368 (6) | 0.3265 (2) | 0.44312 (5) | 0.0253 (4) |
| C11A | 0.68482 (8) | 0.2534 (3) | 0.40888 (6) | 0.0398 (5) |
| H11A | 0.6608 | 0.2039 | 0.3949 | 0.048* |
| C12A | 0.73127 (9) | 0.2530 (3) | 0.39512 (7) | 0.0476 (6) |
| H12A | 0.7384 | 0.2024 | 0.3722 | 0.057* |
| C13A | 0.76677 (7) | 0.3277 (2) | 0.41543 (6) | 0.0368 (4) |
| C14A | 0.75649 (8) | 0.4059 (3) | 0.44857 (6) | 0.0434 (5) |
| H14A | 0.7805 | 0.4579 | 0.4621 | 0.052* |
| C15A | 0.70993 (8) | 0.4069 (3) | 0.46177 (6) | 0.0395 (5) |

| | | | | |
|---|---|---|---|---|
| H15A | 0.7027 | 0.4632 | 0.4839 | 0.047* |
| C16A | 0.68490 (6) | 0.2579 (2) | 0.56903 (5) | 0.0268 (4) |
| C17A | 0.67007 (7) | 0.1859 (2) | 0.60237 (5) | 0.0341 (4) |
| H17A | 0.6388 | 0.1494 | 0.6049 | 0.041* |
| C18A | 0.70240 (8) | 0.1686 (3) | 0.63213 (6) | 0.0440 (5) |
| H18A | 0.6923 | 0.1219 | 0.6548 | 0.053* |
| C19A | 0.74938 (9) | 0.2194 (3) | 0.62881 (6) | 0.0452 (5) |
| C20A | 0.76314 (8) | 0.2944 (3) | 0.59536 (7) | 0.0448 (5) |
| H20A | 0.7942 | 0.3326 | 0.5930 | 0.054* |
| C21A | 0.73132 (7) | 0.3134 (3) | 0.56536 (6) | 0.0375 (4) |
| H21A | 0.7411 | 0.3630 | 0.5429 | 0.045* |
| C22A | 0.56659 (6) | 0.10306 (19) | 0.48956 (5) | 0.0228 (3) |
| C23A | 0.78526 (12) | 0.1889 (5) | 0.66044 (9) | 0.0773 (10) |
| H23A | 0.7691 | 0.1890 | 0.6846 | 0.116* |
| H23B | 0.8092 | 0.2707 | 0.6602 | 0.116* |
| H23C | 0.8002 | 0.0878 | 0.6565 | 0.116* |
| Cl1B | 0.69954 (3) | 0.51675 (14) | 0.24096 (2) | 0.0960 (4) |
| O1B | 0.47607 (5) | 0.76223 (15) | 0.29218 (4) | 0.0334 (3) |
| O2B | 0.43042 (5) | 0.68918 (19) | 0.37411 (4) | 0.0401 (3) |
| O3B | 0.54926 (5) | 0.18004 (16) | 0.38319 (4) | 0.0376 (3) |
| N1B | 0.45537 (5) | 0.50593 (17) | 0.30323 (4) | 0.0246 (3) |
| N2B | 0.49180 (7) | 0.7000 (2) | 0.41537 (5) | 0.0335 (4) |
| C1B | 0.47972 (6) | 0.6448 (2) | 0.31185 (5) | 0.0240 (3) |
| C2B | 0.50872 (6) | 0.64104 (19) | 0.34849 (5) | 0.0219 (3) |
| H2B | 0.5323 | 0.7270 | 0.3476 | 0.026* |
| C3B | 0.53578 (6) | 0.4824 (2) | 0.35275 (5) | 0.0228 (3) |
| H3BA | 0.5485 | 0.4757 | 0.3788 | 0.027* |
| C4B | 0.50099 (6) | 0.3490 (2) | 0.34697 (5) | 0.0238 (3) |
| C5B | 0.51222 (7) | 0.1969 (2) | 0.36457 (5) | 0.0303 (4) |
| C6B | 0.47826 (9) | 0.0613 (2) | 0.35856 (6) | 0.0448 (5) |
| H6BA | 0.4766 | −0.0005 | 0.3819 | 0.054* |
| H6BB | 0.4909 | −0.0069 | 0.3387 | 0.054* |
| C7B | 0.42824 (9) | 0.1114 (3) | 0.34759 (6) | 0.0432 (5) |
| H7BA | 0.4126 | 0.1589 | 0.3695 | 0.052* |
| H7BB | 0.4099 | 0.0188 | 0.3401 | 0.052* |
| C8B | 0.42911 (7) | 0.2300 (2) | 0.31484 (6) | 0.0345 (4) |
| H8BA | 0.4389 | 0.1766 | 0.2916 | 0.041* |
| H8BB | 0.3972 | 0.2720 | 0.3109 | 0.041* |
| C9B | 0.46301 (6) | 0.3640 (2) | 0.32309 (5) | 0.0247 (3) |
| C10B | 0.57756 (6) | 0.4789 (2) | 0.32494 (5) | 0.0255 (4) |
| C11B | 0.62165 (7) | 0.5355 (3) | 0.33652 (6) | 0.0432 (5) |
| H11B | 0.6260 | 0.5660 | 0.3618 | 0.052* |
| C12B | 0.65946 (9) | 0.5476 (4) | 0.31115 (8) | 0.0608 (8) |
| H12B | 0.6890 | 0.5862 | 0.3192 | 0.073* |
| C13B | 0.65256 (8) | 0.5013 (3) | 0.27362 (7) | 0.0505 (6) |
| C14B | 0.60972 (8) | 0.4409 (3) | 0.26148 (6) | 0.0376 (4) |
| H14B | 0.6058 | 0.4080 | 0.2363 | 0.045* |
| C15B | 0.57242 (7) | 0.4297 (2) | 0.28716 (5) | 0.0296 (4) |

| | | | | |
|---|---|---|---|---|
| H15B | 0.5433 | 0.3884 | 0.2791 | 0.035* |
| C16B | 0.41761 (6) | 0.5176 (2) | 0.27496 (5) | 0.0280 (4) |
| C17B | 0.42624 (8) | 0.4736 (3) | 0.23774 (6) | 0.0410 (5) |
| H17B | 0.4561 | 0.4357 | 0.2305 | 0.049* |
| C18B | 0.38942 (9) | 0.4867 (4) | 0.21121 (6) | 0.0557 (7) |
| H18B | 0.3950 | 0.4580 | 0.1859 | 0.067* |
| C19B | 0.34492 (9) | 0.5414 (4) | 0.22167 (7) | 0.0554 (7) |
| C20B | 0.33726 (7) | 0.5846 (3) | 0.25939 (6) | 0.0449 (5) |
| H20B | 0.3073 | 0.6209 | 0.2668 | 0.054* |
| C21B | 0.37358 (7) | 0.5744 (3) | 0.28612 (6) | 0.0344 (4) |
| H21B | 0.3683 | 0.6055 | 0.3113 | 0.041* |
| C22B | 0.47312 (6) | 0.6765 (2) | 0.38092 (5) | 0.0262 (4) |
| C23B | 0.30543 (12) | 0.5582 (7) | 0.19237 (10) | 0.1053 (16) |
| H23D | 0.2808 | 0.4815 | 0.1975 | 0.158* |
| H23E | 0.3183 | 0.5407 | 0.1673 | 0.158* |
| H23F | 0.2921 | 0.6630 | 0.1938 | 0.158* |
| H2A2 | 0.5079 (9) | 0.045 (3) | 0.4652 (6) | 0.031 (6)* |
| H2B1 | 0.4729 (9) | 0.732 (3) | 0.4330 (7) | 0.037 (6)* |
| H2A1 | 0.5470 (9) | 0.085 (3) | 0.4374 (7) | 0.038 (6)* |
| H2B2 | 0.5214 (10) | 0.689 (3) | 0.4200 (7) | 0.043 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1A | 0.0364 (3) | 0.0663 (4) | 0.0816 (5) | −0.0009 (3) | 0.0225 (3) | −0.0174 (3) |
| O1A | 0.0331 (7) | 0.0261 (7) | 0.0375 (7) | 0.0074 (5) | −0.0058 (5) | −0.0021 (5) |
| O2A | 0.0288 (6) | 0.0351 (7) | 0.0251 (6) | −0.0052 (5) | 0.0032 (5) | −0.0055 (5) |
| O3A | 0.0462 (8) | 0.0361 (8) | 0.0406 (8) | 0.0084 (6) | −0.0093 (6) | 0.0078 (6) |
| N1A | 0.0246 (7) | 0.0238 (7) | 0.0246 (7) | 0.0008 (6) | −0.0018 (6) | −0.0010 (6) |
| N2A | 0.0252 (8) | 0.0441 (10) | 0.0259 (8) | −0.0092 (7) | −0.0025 (6) | 0.0044 (7) |
| C1A | 0.0209 (8) | 0.0230 (8) | 0.0276 (8) | −0.0008 (7) | 0.0020 (6) | 0.0006 (7) |
| C2A | 0.0230 (8) | 0.0207 (8) | 0.0240 (8) | 0.0002 (6) | 0.0005 (6) | −0.0021 (6) |
| C3A | 0.0257 (8) | 0.0230 (8) | 0.0233 (8) | −0.0006 (7) | −0.0023 (6) | 0.0007 (6) |
| C4A | 0.0243 (8) | 0.0215 (8) | 0.0288 (9) | −0.0019 (7) | 0.0021 (7) | 0.0003 (7) |
| C5A | 0.0297 (9) | 0.0249 (9) | 0.0391 (10) | 0.0003 (7) | −0.0009 (8) | 0.0030 (8) |
| C6A | 0.0615 (15) | 0.0252 (10) | 0.0542 (13) | 0.0112 (10) | −0.0046 (11) | −0.0017 (9) |
| C7A | 0.0705 (16) | 0.0353 (11) | 0.0437 (12) | 0.0156 (11) | 0.0062 (11) | −0.0097 (9) |
| C8A | 0.0460 (11) | 0.0265 (9) | 0.0294 (9) | −0.0005 (8) | 0.0033 (8) | −0.0046 (7) |
| C9A | 0.0236 (8) | 0.0205 (8) | 0.0283 (8) | −0.0028 (6) | 0.0028 (7) | −0.0003 (7) |
| C10A | 0.0281 (9) | 0.0250 (8) | 0.0229 (8) | −0.0013 (7) | 0.0002 (7) | 0.0032 (6) |
| C11A | 0.0408 (11) | 0.0482 (12) | 0.0305 (10) | −0.0114 (10) | 0.0021 (8) | −0.0116 (9) |
| C12A | 0.0474 (13) | 0.0529 (13) | 0.0423 (12) | −0.0075 (11) | 0.0142 (10) | −0.0205 (10) |
| C13A | 0.0316 (10) | 0.0366 (10) | 0.0423 (11) | −0.0015 (8) | 0.0097 (8) | −0.0003 (8) |
| C14A | 0.0335 (11) | 0.0631 (14) | 0.0336 (10) | −0.0162 (10) | 0.0034 (8) | −0.0070 (10) |
| C15A | 0.0371 (11) | 0.0538 (13) | 0.0275 (9) | −0.0129 (9) | 0.0060 (8) | −0.0105 (9) |
| C16A | 0.0271 (9) | 0.0274 (9) | 0.0257 (8) | 0.0025 (7) | −0.0035 (7) | −0.0027 (7) |
| C17A | 0.0283 (9) | 0.0425 (11) | 0.0314 (9) | 0.0042 (8) | 0.0018 (7) | 0.0034 (8) |
| C18A | 0.0493 (12) | 0.0545 (13) | 0.0282 (10) | 0.0092 (10) | −0.0039 (9) | 0.0052 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C19A | 0.0477 (12) | 0.0477 (13) | 0.0402 (11) | 0.0084 (10) | −0.0178 (10) | −0.0065 (10) |
| C20A | 0.0309 (10) | 0.0487 (13) | 0.0548 (13) | −0.0066 (9) | −0.0126 (9) | −0.0066 (10) |
| C21A | 0.0344 (10) | 0.0422 (11) | 0.0360 (10) | −0.0079 (9) | −0.0036 (8) | 0.0021 (9) |
| C22A | 0.0242 (8) | 0.0162 (7) | 0.0279 (8) | 0.0010 (6) | 0.0011 (7) | −0.0010 (6) |
| C23A | 0.0704 (19) | 0.097 (2) | 0.0647 (18) | 0.0068 (17) | −0.0403 (16) | 0.0008 (17) |
| Cl1B | 0.0615 (4) | 0.1578 (9) | 0.0687 (5) | −0.0455 (5) | 0.0382 (4) | −0.0341 (5) |
| O1B | 0.0397 (7) | 0.0276 (7) | 0.0327 (7) | −0.0015 (6) | −0.0062 (6) | 0.0098 (5) |
| O2B | 0.0275 (7) | 0.0539 (9) | 0.0388 (7) | 0.0046 (6) | 0.0030 (6) | −0.0066 (7) |
| O3B | 0.0484 (8) | 0.0321 (7) | 0.0322 (7) | 0.0089 (6) | −0.0075 (6) | 0.0075 (6) |
| N1B | 0.0254 (7) | 0.0244 (7) | 0.0241 (7) | −0.0002 (6) | −0.0039 (6) | 0.0020 (6) |
| N2B | 0.0365 (9) | 0.0389 (9) | 0.0251 (8) | 0.0098 (7) | 0.0016 (7) | −0.0039 (7) |
| C1B | 0.0247 (8) | 0.0236 (8) | 0.0238 (8) | 0.0017 (7) | 0.0013 (6) | 0.0018 (7) |
| C2B | 0.0234 (8) | 0.0186 (8) | 0.0236 (8) | 0.0000 (6) | −0.0015 (6) | 0.0001 (6) |
| C3B | 0.0274 (8) | 0.0218 (8) | 0.0192 (7) | 0.0037 (7) | −0.0041 (6) | −0.0011 (6) |
| C4B | 0.0306 (9) | 0.0204 (8) | 0.0204 (8) | 0.0023 (7) | 0.0010 (6) | 0.0000 (6) |
| C5B | 0.0455 (11) | 0.0231 (9) | 0.0222 (8) | 0.0049 (8) | 0.0009 (8) | 0.0004 (7) |
| C6B | 0.0723 (16) | 0.0214 (9) | 0.0407 (11) | −0.0050 (10) | −0.0097 (11) | 0.0064 (8) |
| C7B | 0.0568 (14) | 0.0321 (10) | 0.0406 (11) | −0.0195 (10) | −0.0031 (10) | 0.0044 (9) |
| C8B | 0.0390 (10) | 0.0299 (10) | 0.0347 (10) | −0.0085 (8) | −0.0055 (8) | 0.0011 (8) |
| C9B | 0.0301 (9) | 0.0219 (8) | 0.0220 (8) | 0.0003 (7) | 0.0022 (7) | 0.0001 (6) |
| C10B | 0.0260 (8) | 0.0245 (8) | 0.0258 (8) | 0.0036 (7) | −0.0020 (7) | −0.0021 (7) |
| C11B | 0.0329 (10) | 0.0629 (14) | 0.0338 (10) | −0.0063 (10) | −0.0015 (8) | −0.0183 (10) |
| C12B | 0.0325 (11) | 0.093 (2) | 0.0563 (15) | −0.0212 (13) | 0.0061 (10) | −0.0279 (14) |
| C13B | 0.0378 (12) | 0.0696 (16) | 0.0441 (12) | −0.0095 (11) | 0.0146 (10) | −0.0107 (11) |
| C14B | 0.0399 (11) | 0.0463 (11) | 0.0267 (9) | 0.0032 (9) | 0.0028 (8) | −0.0048 (8) |
| C15B | 0.0283 (9) | 0.0348 (10) | 0.0256 (9) | 0.0016 (8) | −0.0032 (7) | −0.0033 (7) |
| C16B | 0.0280 (9) | 0.0304 (9) | 0.0257 (9) | −0.0013 (7) | −0.0049 (7) | 0.0034 (7) |
| C17B | 0.0364 (11) | 0.0569 (13) | 0.0296 (10) | 0.0075 (10) | −0.0037 (8) | −0.0046 (9) |
| C18B | 0.0534 (14) | 0.0867 (19) | 0.0269 (10) | 0.0080 (13) | −0.0111 (10) | −0.0080 (11) |
| C19B | 0.0399 (12) | 0.089 (2) | 0.0375 (12) | 0.0031 (13) | −0.0149 (10) | 0.0056 (12) |
| C20B | 0.0261 (10) | 0.0658 (15) | 0.0429 (12) | 0.0036 (10) | −0.0041 (8) | 0.0047 (10) |
| C21B | 0.0305 (10) | 0.0441 (11) | 0.0287 (9) | −0.0003 (8) | −0.0008 (7) | 0.0028 (8) |
| C22B | 0.0301 (9) | 0.0193 (8) | 0.0291 (9) | 0.0029 (7) | 0.0021 (7) | 0.0013 (6) |
| C23B | 0.0598 (19) | 0.202 (5) | 0.0542 (18) | 0.023 (3) | −0.0332 (15) | −0.004 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1A—C13A | 1.745 (2) | Cl1B—C13B | 1.746 (2) |
| O1A—C1A | 1.209 (2) | O1B—C1B | 1.210 (2) |
| O2A—C22A | 1.234 (2) | O2B—C22B | 1.221 (2) |
| O3A—C5A | 1.230 (2) | O3B—C5B | 1.232 (2) |
| N1A—C1A | 1.391 (2) | N1B—C1B | 1.387 (2) |
| N1A—C9A | 1.403 (2) | N1B—C9B | 1.400 (2) |
| N1A—C16A | 1.452 (2) | N1B—C16B | 1.450 (2) |
| N2A—C22A | 1.323 (2) | N2B—C22B | 1.329 (2) |
| N2A—H2A2 | 0.88 (2) | N2B—H2B1 | 0.85 (3) |
| N2A—H2A1 | 0.86 (3) | N2B—H2B2 | 0.85 (3) |
| C1A—C2A | 1.518 (2) | C1B—C2B | 1.518 (2) |

| | | | |
|---|---|---|---|
| C2A—C22A | 1.535 (2) | C2B—C22B | 1.539 (2) |
| C2A—C3A | 1.546 (2) | C2B—C3B | 1.544 (2) |
| C2A—H2A | 0.9800 | C2B—H2B | 0.9800 |
| C3A—C4A | 1.502 (2) | C3B—C4B | 1.500 (2) |
| C3A—C10A | 1.529 (2) | C3B—C10B | 1.521 (2) |
| C3A—H3AA | 0.9800 | C3B—H3BA | 0.9800 |
| C4A—C9A | 1.355 (3) | C4B—C9B | 1.357 (2) |
| C4A—C5A | 1.456 (3) | C4B—C5B | 1.457 (2) |
| C5A—C6A | 1.501 (3) | C5B—C6B | 1.500 (3) |
| C6A—C7A | 1.500 (3) | C6B—C7B | 1.510 (3) |
| C6A—H6AA | 0.9700 | C6B—H6BA | 0.9700 |
| C6A—H6AB | 0.9700 | C6B—H6BB | 0.9700 |
| C7A—C8A | 1.532 (3) | C7B—C8B | 1.522 (3) |
| C7A—H7AA | 0.9700 | C7B—H7BA | 0.9700 |
| C7A—H7AB | 0.9700 | C7B—H7BB | 0.9700 |
| C8A—C9A | 1.500 (2) | C8B—C9B | 1.503 (3) |
| C8A—H8AA | 0.9700 | C8B—H8BA | 0.9700 |
| C8A—H8AB | 0.9700 | C8B—H8BB | 0.9700 |
| C10A—C15A | 1.383 (3) | C10B—C11B | 1.382 (3) |
| C10A—C11A | 1.383 (3) | C10B—C15B | 1.394 (2) |
| C11A—C12A | 1.385 (3) | C11B—C12B | 1.384 (3) |
| C11A—H11A | 0.9300 | C11B—H11B | 0.9300 |
| C12A—C13A | 1.373 (3) | C12B—C13B | 1.384 (3) |
| C12A—H12A | 0.9300 | C12B—H12B | 0.9300 |
| C13A—C14A | 1.365 (3) | C13B—C14B | 1.369 (3) |
| C14A—C15A | 1.381 (3) | C14B—C15B | 1.380 (3) |
| C14A—H14A | 0.9300 | C14B—H14B | 0.9300 |
| C15A—H15A | 0.9300 | C15B—H15B | 0.9300 |
| C16A—C17A | 1.379 (3) | C16B—C17B | 1.376 (3) |
| C16A—C21A | 1.385 (3) | C16B—C21B | 1.377 (3) |
| C17A—C18A | 1.387 (3) | C17B—C18B | 1.391 (3) |
| C17A—H17A | 0.9300 | C17B—H17B | 0.9300 |
| C18A—C19A | 1.386 (4) | C18B—C19B | 1.376 (4) |
| C18A—H18A | 0.9300 | C18B—H18B | 0.9300 |
| C19A—C20A | 1.385 (3) | C19B—C20B | 1.386 (3) |
| C19A—C23A | 1.516 (3) | C19B—C23B | 1.513 (3) |
| C20A—C21A | 1.386 (3) | C20B—C21B | 1.383 (3) |
| C20A—H20A | 0.9300 | C20B—H20B | 0.9300 |
| C21A—H21A | 0.9300 | C21B—H21B | 0.9300 |
| C23A—H23A | 0.9600 | C23B—H23D | 0.9600 |
| C23A—H23B | 0.9600 | C23B—H23E | 0.9600 |
| C23A—H23C | 0.9600 | C23B—H23F | 0.9600 |

| | | | |
|---|---|---|---|
| C1A—N1A—C9A | 122.11 (14) | C1B—N1B—C9B | 122.57 (14) |
| C1A—N1A—C16A | 116.03 (14) | C1B—N1B—C16B | 116.61 (14) |
| C9A—N1A—C16A | 121.72 (14) | C9B—N1B—C16B | 120.53 (14) |
| C22A—N2A—H2A2 | 118.9 (14) | C22B—N2B—H2B1 | 117.2 (16) |
| C22A—N2A—H2A1 | 121.2 (16) | C22B—N2B—H2B2 | 122.7 (17) |

| | | | |
|---|---|---|---|
| H2A2—N2A—H2A1 | 120 (2) | H2B1—N2B—H2B2 | 120 (2) |
| O1A—C1A—N1A | 121.78 (16) | O1B—C1B—N1B | 121.69 (16) |
| O1A—C1A—C2A | 122.32 (16) | O1B—C1B—C2B | 122.89 (16) |
| N1A—C1A—C2A | 115.90 (14) | N1B—C1B—C2B | 115.34 (14) |
| C1A—C2A—C22A | 109.34 (14) | C1B—C2B—C22B | 105.92 (14) |
| C1A—C2A—C3A | 110.54 (14) | C1B—C2B—C3B | 111.19 (13) |
| C22A—C2A—C3A | 113.03 (14) | C22B—C2B—C3B | 114.42 (13) |
| C1A—C2A—H2A | 107.9 | C1B—C2B—H2B | 108.4 |
| C22A—C2A—H2A | 107.9 | C22B—C2B—H2B | 108.4 |
| C3A—C2A—H2A | 107.9 | C3B—C2B—H2B | 108.4 |
| C4A—C3A—C10A | 112.35 (14) | C4B—C3B—C10B | 113.37 (14) |
| C4A—C3A—C2A | 107.50 (14) | C4B—C3B—C2B | 108.59 (14) |
| C10A—C3A—C2A | 109.75 (14) | C10B—C3B—C2B | 109.32 (14) |
| C4A—C3A—H3AA | 109.1 | C4B—C3B—H3BA | 108.5 |
| C10A—C3A—H3AA | 109.1 | C10B—C3B—H3BA | 108.5 |
| C2A—C3A—H3AA | 109.1 | C2B—C3B—H3BA | 108.5 |
| C9A—C4A—C5A | 121.53 (16) | C9B—C4B—C5B | 120.72 (16) |
| C9A—C4A—C3A | 120.26 (16) | C9B—C4B—C3B | 121.35 (15) |
| C5A—C4A—C3A | 117.73 (16) | C5B—C4B—C3B | 117.59 (15) |
| O3A—C5A—C4A | 120.51 (18) | O3B—C5B—C4B | 120.41 (17) |
| O3A—C5A—C6A | 121.28 (18) | O3B—C5B—C6B | 121.21 (17) |
| C4A—C5A—C6A | 118.12 (17) | C4B—C5B—C6B | 118.37 (17) |
| C7A—C6A—C5A | 113.28 (18) | C5B—C6B—C7B | 114.10 (17) |
| C7A—C6A—H6AA | 108.9 | C5B—C6B—H6BA | 108.7 |
| C5A—C6A—H6AA | 108.9 | C7B—C6B—H6BA | 108.7 |
| C7A—C6A—H6AB | 108.9 | C5B—C6B—H6BB | 108.7 |
| C5A—C6A—H6AB | 108.9 | C7B—C6B—H6BB | 108.7 |
| H6AA—C6A—H6AB | 107.7 | H6BA—C6B—H6BB | 107.6 |
| C6A—C7A—C8A | 112.44 (19) | C6B—C7B—C8B | 111.15 (18) |
| C6A—C7A—H7AA | 109.1 | C6B—C7B—H7BA | 109.4 |
| C8A—C7A—H7AA | 109.1 | C8B—C7B—H7BA | 109.4 |
| C6A—C7A—H7AB | 109.1 | C6B—C7B—H7BB | 109.4 |
| C8A—C7A—H7AB | 109.1 | C8B—C7B—H7BB | 109.4 |
| H7AA—C7A—H7AB | 107.8 | H7BA—C7B—H7BB | 108.0 |
| C9A—C8A—C7A | 110.44 (17) | C9B—C8B—C7B | 111.05 (16) |
| C9A—C8A—H8AA | 109.6 | C9B—C8B—H8BA | 109.4 |
| C7A—C8A—H8AA | 109.6 | C7B—C8B—H8BA | 109.4 |
| C9A—C8A—H8AB | 109.6 | C9B—C8B—H8BB | 109.4 |
| C7A—C8A—H8AB | 109.6 | C7B—C8B—H8BB | 109.4 |
| H8AA—C8A—H8AB | 108.1 | H8BA—C8B—H8BB | 108.0 |
| C4A—C9A—N1A | 120.00 (15) | C4B—C9B—N1B | 120.31 (16) |
| C4A—C9A—C8A | 122.81 (16) | C4B—C9B—C8B | 122.79 (16) |
| N1A—C9A—C8A | 117.15 (15) | N1B—C9B—C8B | 116.81 (15) |
| C15A—C10A—C11A | 117.57 (17) | C11B—C10B—C15B | 118.23 (17) |
| C15A—C10A—C3A | 122.30 (16) | C11B—C10B—C3B | 119.34 (16) |
| C11A—C10A—C3A | 120.04 (16) | C15B—C10B—C3B | 122.30 (16) |
| C10A—C11A—C12A | 120.90 (19) | C10B—C11B—C12B | 121.18 (19) |
| C10A—C11A—H11A | 119.5 | C10B—C11B—H11B | 119.4 |

| | | | |
|---|---|---|---|
| C12A—C11A—H11A | 119.5 | C12B—C11B—H11B | 119.4 |
| C13A—C12A—C11A | 119.72 (19) | C11B—C12B—C13B | 118.8 (2) |
| C13A—C12A—H12A | 120.1 | C11B—C12B—H12B | 120.6 |
| C11A—C12A—H12A | 120.1 | C13B—C12B—H12B | 120.6 |
| C14A—C13A—C12A | 120.63 (19) | C14B—C13B—C12B | 121.4 (2) |
| C14A—C13A—Cl1A | 118.75 (17) | C14B—C13B—Cl1B | 118.84 (18) |
| C12A—C13A—Cl1A | 120.60 (16) | C12B—C13B—Cl1B | 119.73 (18) |
| C13A—C14A—C15A | 119.05 (19) | C13B—C14B—C15B | 118.95 (19) |
| C13A—C14A—H14A | 120.5 | C13B—C14B—H14B | 120.5 |
| C15A—C14A—H14A | 120.5 | C15B—C14B—H14B | 120.5 |
| C14A—C15A—C10A | 121.97 (19) | C14B—C15B—C10B | 121.35 (18) |
| C14A—C15A—H15A | 119.0 | C14B—C15B—H15B | 119.3 |
| C10A—C15A—H15A | 119.0 | C10B—C15B—H15B | 119.3 |
| C17A—C16A—C21A | 120.58 (17) | C17B—C16B—C21B | 121.26 (18) |
| C17A—C16A—N1A | 120.22 (16) | C17B—C16B—N1B | 120.03 (17) |
| C21A—C16A—N1A | 119.08 (16) | C21B—C16B—N1B | 118.71 (16) |
| C16A—C17A—C18A | 119.10 (19) | C16B—C17B—C18B | 118.8 (2) |
| C16A—C17A—H17A | 120.4 | C16B—C17B—H17B | 120.6 |
| C18A—C17A—H17A | 120.4 | C18B—C17B—H17B | 120.6 |
| C19A—C18A—C17A | 121.4 (2) | C19B—C18B—C17B | 121.2 (2) |
| C19A—C18A—H18A | 119.3 | C19B—C18B—H18B | 119.4 |
| C17A—C18A—H18A | 119.3 | C17B—C18B—H18B | 119.4 |
| C20A—C19A—C18A | 118.36 (19) | C18B—C19B—C20B | 118.8 (2) |
| C20A—C19A—C23A | 120.7 (2) | C18B—C19B—C23B | 120.7 (3) |
| C18A—C19A—C23A | 120.9 (2) | C20B—C19B—C23B | 120.6 (3) |
| C19A—C20A—C21A | 121.0 (2) | C21B—C20B—C19B | 121.0 (2) |
| C19A—C20A—H20A | 119.5 | C21B—C20B—H20B | 119.5 |
| C21A—C20A—H20A | 119.5 | C19B—C20B—H20B | 119.5 |
| C16A—C21A—C20A | 119.4 (2) | C16B—C21B—C20B | 119.03 (19) |
| C16A—C21A—H21A | 120.3 | C16B—C21B—H21B | 120.5 |
| C20A—C21A—H21A | 120.3 | C20B—C21B—H21B | 120.5 |
| O2A—C22A—N2A | 123.25 (16) | O2B—C22B—N2B | 123.20 (17) |
| O2A—C22A—C2A | 121.21 (15) | O2B—C22B—C2B | 120.30 (16) |
| N2A—C22A—C2A | 115.49 (15) | N2B—C22B—C2B | 116.41 (16) |
| C19A—C23A—H23A | 109.5 | C19B—C23B—H23D | 109.5 |
| C19A—C23A—H23B | 109.5 | C19B—C23B—H23E | 109.5 |
| H23A—C23A—H23B | 109.5 | H23D—C23B—H23E | 109.5 |
| C19A—C23A—H23C | 109.5 | C19B—C23B—H23F | 109.5 |
| H23A—C23A—H23C | 109.5 | H23D—C23B—H23F | 109.5 |
| H23B—C23A—H23C | 109.5 | H23E—C23B—H23F | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2*A*—H2*A*2···O2*A*$^{i}$ | 0.88 (2) | 2.12 (3) | 2.979 (2) | 164 (2) |
| N2*B*—H2*B*1···O2*A*$^{ii}$ | 0.86 (2) | 2.26 (2) | 3.055 (2) | 154 (2) |
| N2*A*—H2*A*1···O3*B* | 0.86 (2) | 2.06 (2) | 2.865 (2) | 156 (2) |
| N2*B*—H2*B*2···O3*A* | 0.85 (3) | 2.04 (3) | 2.847 (2) | 159 (2) |

| | | | | |
|---|---|---|---|---|
| C3*A*—H3*AA*···O3*A* | 0.98 | 2.45 | 2.793 (2) | 100 |
| C8*A*—H8*AB*···O2*B*$^{ii}$ | 0.97 | 2.50 | 3.392 (2) | 153 |
| C6*B*—H6*BB*···O1*B*$^{iii}$ | 0.97 | 2.57 | 3.430 (2) | 148 |
| C14*A*—H14*A*···O1*A*$^{iv}$ | 0.93 | 2.54 | 3.393 (3) | 153 |
| C17*A*—H17*A*···O2*B*$^{ii}$ | 0.93 | 2.48 | 3.110 (2) | 125 |
| C17*B*—H17*B*···O1*B*$^{v}$ | 0.93 | 2.52 | 3.424 (3) | 164 |

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

### 4-(4-Methoxyphenyl)-1-(4-methylphenyl)-2,5-dioxo-1,2,5,6,7,8-hexahydroquinoline-3-carbonitrile (II)

*Crystal data*

$C_{24}H_{22}N_2O_3$
$M_r$ = 386.43
Monoclinic, $P2_1/c$
$a$ = 10.3486 (2) Å
$b$ = 13.9969 (3) Å
$c$ = 27.5353 (5) Å
$\beta$ = 93.797 (2)°
$V$ = 3979.69 (14) Å$^3$
$Z$ = 8
$F(000)$ = 1632
$D_x$ = 1.290 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54184 Å
Cell parameters from 3849 reflections
$\theta$ = 4.3–70.8°
$\mu$ = 0.69 mm$^{-1}$
$T$ = 173 K
Irregular, orange
0.12 × 0.08 × 0.04 mm

*Data collection*

Rigaku Oxford Diffraction Xcalibur, Eos, Gemini diffractometer
Detector resolution: 16.0416 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2015)
$T_{min}$ = 0.945, $T_{max}$ = 1.000
15107 measured reflections
7579 independent reflections
5380 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 71.4°, $\theta_{min}$ = 3.5°
$h$ = −9→12
$k$ = −10→16
$l$ = −26→33

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.139
$S$ = 1.03
7579 reflections
555 parameters
36 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0628P)^2 + 0.1965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

*Special details*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1A | 0.36261 (17) | 0.53971 (14) | 0.33466 (6) | 0.0704 (5) | |
| O2A | 0.78343 (15) | 0.78301 (13) | 0.24000 (6) | 0.0635 (4) | |

| | | | | | |
|---|---|---|---|---|---|
| O3A | 0.5360 (2) | 0.99458 (16) | 0.43140 (8) | 0.0866 (6) | |
| N1A | 0.40024 (16) | 0.60665 (14) | 0.26190 (6) | 0.0490 (4) | |
| N2A | 0.6257 (3) | 0.56408 (18) | 0.41665 (8) | 0.0773 (6) | |
| C1A | 0.4369 (2) | 0.57641 (15) | 0.30847 (7) | 0.0502 (5) | |
| C2A | 0.5814 (2) | 0.58943 (16) | 0.32306 (7) | 0.0499 (5) | |
| H2A | 0.628554 | 0.537522 | 0.306612 | 0.060* | |
| C3A | 0.63700 (18) | 0.68516 (15) | 0.30610 (7) | 0.0462 (4) | |
| H3A | 0.733419 | 0.678557 | 0.307960 | 0.055* | |
| C4A | 0.59327 (19) | 0.69818 (15) | 0.25308 (7) | 0.0440 (4) | |
| C5A | 0.6773 (2) | 0.75536 (16) | 0.22368 (8) | 0.0509 (5) | |
| C6A | 0.6240 (7) | 0.7794 (6) | 0.1737 (2) | 0.093 (3) | 0.670 (11) |
| H6A1 | 0.647523 | 0.846564 | 0.167295 | 0.112* | 0.670 (11) |
| H6A2 | 0.669856 | 0.739274 | 0.150722 | 0.112* | 0.670 (11) |
| C7A | 0.4928 (5) | 0.7699 (5) | 0.16172 (18) | 0.0727 (19) | 0.670 (11) |
| H7A1 | 0.478234 | 0.768970 | 0.125825 | 0.087* | 0.670 (11) |
| H7A2 | 0.447588 | 0.826567 | 0.173844 | 0.087* | 0.670 (11) |
| C8A | 0.4343 (10) | 0.6823 (10) | 0.1818 (3) | 0.058 (3) | 0.670 (11) |
| H8A1 | 0.454564 | 0.627035 | 0.161223 | 0.070* | 0.670 (11) |
| H8A2 | 0.338993 | 0.689814 | 0.180160 | 0.070* | 0.670 (11) |
| C6AA | 0.6417 (13) | 0.7776 (12) | 0.1706 (4) | 0.081 (6) | 0.330 (11) |
| H6A3 | 0.609006 | 0.843920 | 0.167569 | 0.097* | 0.330 (11) |
| H6A4 | 0.719692 | 0.772369 | 0.151825 | 0.097* | 0.330 (11) |
| C7AA | 0.5411 (9) | 0.7106 (9) | 0.1504 (2) | 0.062 (3) | 0.330 (11) |
| H7A3 | 0.587817 | 0.655632 | 0.137269 | 0.074* | 0.330 (11) |
| H7A4 | 0.496696 | 0.743252 | 0.122131 | 0.074* | 0.330 (11) |
| C8AA | 0.4327 (18) | 0.668 (2) | 0.1804 (5) | 0.054 (5) | 0.330 (11) |
| H8A3 | 0.354750 | 0.708882 | 0.177077 | 0.064* | 0.330 (11) |
| H8A4 | 0.408875 | 0.603398 | 0.167990 | 0.064* | 0.330 (11) |
| C9A | 0.48142 (19) | 0.66155 (15) | 0.23353 (7) | 0.0454 (4) | |
| C10A | 0.60548 (19) | 0.76995 (15) | 0.33746 (7) | 0.0453 (4) | |
| C11A | 0.6939 (2) | 0.79817 (18) | 0.37505 (8) | 0.0580 (5) | |
| H11A | 0.773514 | 0.764736 | 0.380089 | 0.070* | |
| C12A | 0.6683 (2) | 0.8734 (2) | 0.40504 (9) | 0.0664 (6) | |
| H12A | 0.730490 | 0.891710 | 0.430239 | 0.080* | |
| C13A | 0.5529 (2) | 0.92254 (17) | 0.39877 (9) | 0.0594 (6) | |
| C14A | 0.4648 (2) | 0.89723 (17) | 0.36131 (9) | 0.0588 (5) | |
| H14A | 0.385742 | 0.931387 | 0.356279 | 0.071* | |
| C15A | 0.4919 (2) | 0.82139 (17) | 0.33077 (8) | 0.0528 (5) | |
| H15A | 0.430961 | 0.804786 | 0.304833 | 0.063* | |
| C16A | 0.2677 (2) | 0.58533 (16) | 0.24393 (7) | 0.0478 (5) | |
| C17A | 0.2392 (2) | 0.50222 (18) | 0.21912 (10) | 0.0633 (6) | |
| H17A | 0.305254 | 0.456910 | 0.214024 | 0.076* | |
| C18A | 0.1124 (3) | 0.48517 (19) | 0.20156 (10) | 0.0689 (7) | |
| H18A | 0.092331 | 0.427678 | 0.184344 | 0.083* | |
| C19A | 0.0151 (2) | 0.55004 (18) | 0.20862 (8) | 0.0574 (5) | |
| C20A | 0.0455 (2) | 0.63227 (18) | 0.23423 (8) | 0.0568 (5) | |
| H20A | −0.020731 | 0.677108 | 0.239908 | 0.068* | |
| C21A | 0.1721 (2) | 0.65056 (16) | 0.25189 (7) | 0.0521 (5) | |

| | | | | |
|---|---|---|---|---|
| H21A | 0.192371 | 0.707754 | 0.269352 | 0.062* |
| C22A | 0.6045 (2) | 0.57437 (17) | 0.37597 (8) | 0.0578 (5) |
| C23A | 0.4110 (3) | 1.0295 (3) | 0.43679 (13) | 0.0962 (10) |
| H23A | 0.378518 | 1.060752 | 0.406571 | 0.144* |
| H23B | 0.413342 | 1.075712 | 0.463558 | 0.144* |
| H23C | 0.353668 | 0.976341 | 0.444030 | 0.144* |
| C24A | −0.1230 (3) | 0.5333 (2) | 0.18813 (11) | 0.0803 (8) |
| H24A | −0.183518 | 0.561316 | 0.210066 | 0.120* |
| H24B | −0.139106 | 0.464541 | 0.185086 | 0.120* |
| H24C | −0.135458 | 0.563409 | 0.156018 | 0.120* |
| O1B | 0.88611 (15) | 0.55891 (11) | 0.33264 (6) | 0.0597 (4) |
| O2B | 1.17002 (16) | 0.19933 (11) | 0.42029 (6) | 0.0623 (4) |
| O3B | 0.61297 (18) | 0.20781 (13) | 0.49274 (7) | 0.0735 (5) |
| N1B | 1.05243 (16) | 0.52114 (12) | 0.38701 (6) | 0.0442 (4) |
| N2B | 0.7586 (3) | 0.37087 (19) | 0.27435 (9) | 0.0866 (8) |
| C1B | 0.96338 (19) | 0.50067 (15) | 0.34912 (7) | 0.0445 (4) |
| C2B | 0.9722 (2) | 0.40016 (16) | 0.32852 (7) | 0.0473 (5) |
| H2B | 1.045583 | 0.399447 | 0.306669 | 0.057* |
| C3B | 0.99990 (19) | 0.32262 (14) | 0.36769 (7) | 0.0445 (4) |
| H3B | 1.028406 | 0.263711 | 0.350848 | 0.053* |
| C4B | 1.11285 (18) | 0.35722 (14) | 0.40066 (6) | 0.0409 (4) |
| C5B | 1.19280 (19) | 0.28396 (15) | 0.42658 (7) | 0.0459 (4) |
| C6B | 1.3012 (2) | 0.31731 (17) | 0.46160 (8) | 0.0566 (5) |
| H6BA | 1.372469 | 0.270099 | 0.462105 | 0.068* |
| H6BB | 1.269539 | 0.320138 | 0.494725 | 0.068* |
| C7B | 1.3531 (2) | 0.41394 (17) | 0.44876 (8) | 0.0546 (5) |
| H7BA | 1.398831 | 0.408795 | 0.418402 | 0.066* |
| H7BB | 1.416456 | 0.435436 | 0.475017 | 0.066* |
| C8B | 1.2457 (2) | 0.48714 (16) | 0.44186 (7) | 0.0496 (5) |
| H8BA | 1.213782 | 0.503444 | 0.473927 | 0.060* |
| H8BB | 1.280653 | 0.546139 | 0.427828 | 0.060* |
| C9B | 1.13513 (18) | 0.45094 (14) | 0.40896 (6) | 0.0409 (4) |
| C10B | 0.88665 (18) | 0.29520 (14) | 0.39738 (7) | 0.0438 (4) |
| C11B | 0.8175 (2) | 0.36257 (15) | 0.42189 (8) | 0.0490 (5) |
| H11B | 0.834148 | 0.428504 | 0.417023 | 0.059* |
| C12B | 0.7244 (2) | 0.33612 (16) | 0.45338 (8) | 0.0529 (5) |
| H12B | 0.677947 | 0.383529 | 0.469731 | 0.064* |
| C13B | 0.6998 (2) | 0.24062 (16) | 0.46077 (8) | 0.0524 (5) |
| C14B | 0.7626 (2) | 0.17243 (16) | 0.43470 (9) | 0.0596 (6) |
| H14B | 0.742172 | 0.106745 | 0.438278 | 0.072* |
| C15B | 0.8550 (2) | 0.19959 (16) | 0.40346 (8) | 0.0552 (5) |
| H15B | 0.897639 | 0.152078 | 0.385788 | 0.066* |
| C16B | 1.05339 (19) | 0.61852 (14) | 0.40490 (7) | 0.0439 (4) |
| C17B | 1.0002 (2) | 0.64075 (17) | 0.44792 (8) | 0.0591 (5) |
| H17B | 0.964595 | 0.591822 | 0.466862 | 0.071* |
| C18B | 0.9988 (3) | 0.73479 (19) | 0.46353 (9) | 0.0659 (6) |
| H18B | 0.963120 | 0.749395 | 0.493561 | 0.079* |
| C19B | 1.0478 (2) | 0.80763 (17) | 0.43663 (9) | 0.0593 (6) |

| | | | | |
|---|---|---|---|---|
| C20B | 1.1019 (3) | 0.78322 (18) | 0.39393 (10) | 0.0700 (7) |
| H20B | 1.137513 | 0.831935 | 0.374852 | 0.084* |
| C21B | 1.1056 (3) | 0.68946 (17) | 0.37820 (8) | 0.0617 (6) |
| H21B | 1.144360 | 0.674336 | 0.348844 | 0.074* |
| C22B | 0.8523 (2) | 0.38295 (17) | 0.29799 (8) | 0.0599 (6) |
| C23B | 0.5890 (3) | 0.2675 (2) | 0.53236 (9) | 0.0676 (6) |
| H23D | 0.541756 | 0.324436 | 0.520484 | 0.101* |
| H23E | 0.671532 | 0.286706 | 0.549044 | 0.101* |
| H23F | 0.537152 | 0.232836 | 0.555109 | 0.101* |
| C24B | 1.0432 (3) | 0.9097 (2) | 0.45374 (13) | 0.0892 (9) |
| H24D | 0.989531 | 0.913847 | 0.481671 | 0.134* |
| H24E | 1.006030 | 0.950037 | 0.427298 | 0.134* |
| H24F | 1.131171 | 0.931536 | 0.463385 | 0.134* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0649 (10) | 0.0884 (13) | 0.0588 (9) | −0.0090 (9) | 0.0108 (8) | 0.0254 (9) |
| O2A | 0.0504 (9) | 0.0733 (11) | 0.0676 (10) | −0.0076 (8) | 0.0095 (7) | 0.0054 (8) |
| O3A | 0.0840 (13) | 0.0771 (13) | 0.0989 (14) | −0.0122 (10) | 0.0082 (11) | −0.0350 (11) |
| N1A | 0.0440 (9) | 0.0576 (10) | 0.0458 (9) | −0.0023 (7) | 0.0052 (7) | 0.0081 (7) |
| N2A | 0.1003 (18) | 0.0772 (15) | 0.0541 (12) | 0.0104 (13) | 0.0028 (11) | 0.0156 (10) |
| C1A | 0.0551 (12) | 0.0493 (11) | 0.0472 (10) | 0.0038 (9) | 0.0099 (9) | 0.0094 (9) |
| C2A | 0.0532 (12) | 0.0510 (12) | 0.0457 (10) | 0.0118 (9) | 0.0045 (9) | 0.0089 (9) |
| C3A | 0.0378 (9) | 0.0527 (11) | 0.0482 (10) | 0.0077 (8) | 0.0025 (8) | 0.0105 (9) |
| C4A | 0.0429 (10) | 0.0475 (11) | 0.0425 (10) | 0.0094 (8) | 0.0077 (8) | 0.0061 (8) |
| C5A | 0.0464 (11) | 0.0552 (12) | 0.0522 (11) | 0.0016 (9) | 0.0115 (9) | 0.0026 (9) |
| C6A | 0.095 (5) | 0.129 (6) | 0.055 (4) | −0.062 (4) | −0.002 (3) | 0.049 (4) |
| C7A | 0.069 (3) | 0.097 (4) | 0.051 (2) | −0.014 (3) | −0.0021 (19) | 0.029 (2) |
| C8A | 0.058 (4) | 0.069 (5) | 0.046 (4) | −0.003 (3) | −0.009 (3) | 0.002 (2) |
| C6AA | 0.065 (7) | 0.122 (12) | 0.058 (8) | 0.023 (7) | 0.014 (6) | −0.023 (7) |
| C7AA | 0.066 (5) | 0.084 (7) | 0.035 (3) | −0.013 (5) | 0.008 (3) | 0.006 (3) |
| C8AA | 0.052 (8) | 0.075 (9) | 0.037 (7) | −0.018 (6) | 0.024 (6) | 0.010 (5) |
| C9A | 0.0445 (10) | 0.0503 (11) | 0.0421 (10) | 0.0058 (8) | 0.0088 (8) | 0.0049 (8) |
| C10A | 0.0411 (10) | 0.0502 (11) | 0.0445 (10) | −0.0005 (8) | 0.0026 (8) | 0.0100 (8) |
| C11A | 0.0454 (11) | 0.0657 (14) | 0.0616 (13) | 0.0010 (10) | −0.0051 (10) | 0.0044 (11) |
| C12A | 0.0593 (14) | 0.0718 (16) | 0.0664 (14) | −0.0142 (12) | −0.0085 (11) | −0.0072 (12) |
| C13A | 0.0596 (13) | 0.0534 (13) | 0.0660 (13) | −0.0135 (10) | 0.0094 (11) | −0.0063 (10) |
| C14A | 0.0508 (12) | 0.0572 (13) | 0.0686 (14) | 0.0063 (10) | 0.0046 (10) | 0.0008 (11) |
| C15A | 0.0466 (11) | 0.0570 (12) | 0.0538 (11) | 0.0030 (9) | −0.0048 (9) | 0.0006 (9) |
| C16A | 0.0449 (10) | 0.0540 (12) | 0.0451 (10) | −0.0013 (9) | 0.0082 (8) | 0.0076 (9) |
| C17A | 0.0542 (13) | 0.0550 (13) | 0.0811 (16) | 0.0032 (10) | 0.0071 (11) | −0.0048 (12) |
| C18A | 0.0643 (15) | 0.0568 (14) | 0.0847 (17) | −0.0078 (11) | −0.0007 (13) | −0.0096 (12) |
| C19A | 0.0530 (12) | 0.0643 (14) | 0.0552 (12) | −0.0051 (10) | 0.0048 (10) | 0.0117 (10) |
| C20A | 0.0523 (12) | 0.0664 (14) | 0.0523 (11) | 0.0096 (10) | 0.0085 (9) | 0.0076 (10) |
| C21A | 0.0571 (12) | 0.0535 (12) | 0.0459 (10) | 0.0008 (10) | 0.0055 (9) | 0.0016 (9) |
| C22A | 0.0637 (14) | 0.0543 (13) | 0.0555 (13) | 0.0103 (10) | 0.0039 (10) | 0.0096 (10) |
| C23A | 0.090 (2) | 0.094 (2) | 0.106 (2) | 0.0119 (18) | 0.0168 (18) | −0.0366 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C24A | 0.0576 (15) | 0.095 (2) | 0.0873 (19) | −0.0087 (14) | −0.0062 (13) | 0.0090 (16) |
| O1B | 0.0548 (9) | 0.0560 (9) | 0.0661 (9) | 0.0093 (7) | −0.0118 (7) | 0.0051 (7) |
| O2B | 0.0607 (9) | 0.0479 (9) | 0.0773 (10) | 0.0092 (7) | −0.0046 (8) | 0.0018 (7) |
| O3B | 0.0720 (11) | 0.0662 (11) | 0.0845 (12) | −0.0222 (9) | 0.0214 (9) | −0.0025 (9) |
| N1B | 0.0465 (9) | 0.0438 (9) | 0.0420 (8) | 0.0044 (7) | 0.0004 (7) | −0.0016 (7) |
| N2B | 0.0919 (17) | 0.0799 (16) | 0.0825 (15) | −0.0069 (13) | −0.0364 (14) | −0.0028 (12) |
| C1B | 0.0426 (10) | 0.0496 (11) | 0.0412 (9) | 0.0033 (8) | 0.0011 (8) | 0.0045 (8) |
| C2B | 0.0483 (11) | 0.0565 (12) | 0.0367 (9) | 0.0033 (9) | −0.0003 (8) | −0.0037 (8) |
| C3B | 0.0472 (10) | 0.0443 (10) | 0.0418 (9) | 0.0072 (8) | 0.0007 (8) | −0.0078 (8) |
| C4B | 0.0386 (9) | 0.0482 (11) | 0.0361 (9) | 0.0049 (8) | 0.0049 (7) | −0.0014 (8) |
| C5B | 0.0437 (10) | 0.0489 (12) | 0.0453 (10) | 0.0067 (8) | 0.0062 (8) | −0.0010 (8) |
| C6B | 0.0526 (12) | 0.0586 (13) | 0.0572 (12) | 0.0100 (10) | −0.0076 (10) | 0.0048 (10) |
| C7B | 0.0434 (11) | 0.0633 (14) | 0.0558 (11) | 0.0018 (9) | −0.0065 (9) | −0.0036 (10) |
| C8B | 0.0485 (11) | 0.0517 (12) | 0.0478 (10) | 0.0006 (9) | −0.0023 (9) | −0.0035 (9) |
| C9B | 0.0402 (9) | 0.0487 (11) | 0.0342 (8) | 0.0048 (8) | 0.0051 (7) | −0.0017 (7) |
| C10B | 0.0409 (9) | 0.0455 (10) | 0.0439 (9) | 0.0006 (8) | −0.0058 (8) | −0.0040 (8) |
| C11B | 0.0501 (11) | 0.0409 (10) | 0.0562 (11) | −0.0020 (8) | 0.0052 (9) | −0.0017 (9) |
| C12B | 0.0498 (11) | 0.0520 (12) | 0.0575 (12) | −0.0008 (9) | 0.0070 (9) | −0.0061 (9) |
| C13B | 0.0458 (11) | 0.0551 (12) | 0.0557 (11) | −0.0101 (9) | −0.0001 (9) | 0.0018 (9) |
| C14B | 0.0625 (13) | 0.0432 (11) | 0.0726 (14) | −0.0109 (10) | 0.0000 (11) | −0.0026 (10) |
| C15B | 0.0570 (13) | 0.0439 (11) | 0.0641 (13) | 0.0013 (9) | −0.0010 (10) | −0.0111 (9) |
| C16B | 0.0420 (10) | 0.0467 (11) | 0.0428 (9) | 0.0054 (8) | 0.0006 (8) | 0.0003 (8) |
| C17B | 0.0657 (14) | 0.0561 (13) | 0.0573 (12) | −0.0059 (11) | 0.0186 (10) | −0.0036 (10) |
| C18B | 0.0691 (15) | 0.0678 (16) | 0.0625 (14) | 0.0010 (12) | 0.0173 (12) | −0.0212 (12) |
| C19B | 0.0551 (12) | 0.0513 (13) | 0.0697 (14) | 0.0069 (10) | −0.0090 (11) | −0.0095 (11) |
| C20B | 0.0912 (19) | 0.0468 (13) | 0.0725 (15) | −0.0048 (12) | 0.0084 (14) | 0.0094 (11) |
| C21B | 0.0829 (16) | 0.0534 (13) | 0.0507 (12) | 0.0017 (12) | 0.0184 (11) | 0.0030 (10) |
| C22B | 0.0708 (15) | 0.0574 (13) | 0.0497 (11) | 0.0034 (11) | −0.0101 (11) | −0.0021 (10) |
| C23B | 0.0629 (14) | 0.0806 (17) | 0.0601 (13) | −0.0064 (13) | 0.0093 (11) | 0.0109 (12) |
| C24B | 0.093 (2) | 0.0580 (16) | 0.114 (2) | 0.0077 (15) | −0.0134 (18) | −0.0221 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1A—C1A | 1.204 (3) | C23A—H23B | 0.9800 |
| O2A—C5A | 1.222 (3) | C23A—H23C | 0.9800 |
| O3A—C13A | 1.369 (3) | C24A—H24A | 0.9800 |
| O3A—C23A | 1.400 (4) | C24A—H24B | 0.9800 |
| N1A—C1A | 1.380 (3) | C24A—H24C | 0.9800 |
| N1A—C9A | 1.412 (3) | O1B—C1B | 1.209 (2) |
| N1A—C16A | 1.458 (3) | O2B—C5B | 1.218 (3) |
| N2A—C22A | 1.136 (3) | O3B—C13B | 1.378 (3) |
| C1A—C2A | 1.533 (3) | O3B—C23B | 1.409 (3) |
| C2A—C22A | 1.476 (3) | N1B—C1B | 1.376 (3) |
| C2A—C3A | 1.543 (3) | N1B—C9B | 1.412 (3) |
| C2A—H2A | 1.0000 | N1B—C16B | 1.449 (3) |
| C3A—C4A | 1.510 (3) | N2B—C22B | 1.145 (3) |
| C3A—C10A | 1.516 (3) | C1B—C2B | 1.522 (3) |
| C3A—H3A | 1.0000 | C2B—C22B | 1.471 (3) |

| | | | |
|---|---|---|---|
| C4A—C9A | 1.345 (3) | C2B—C3B | 1.544 (3) |
| C4A—C5A | 1.465 (3) | C2B—H2B | 1.0000 |
| C5A—C6A | 1.487 (6) | C3B—C4B | 1.511 (3) |
| C5A—C6AA | 1.515 (13) | C3B—C10B | 1.522 (3) |
| C6A—C7A | 1.382 (7) | C3B—H3B | 1.0000 |
| C6A—H6A1 | 0.9900 | C4B—C9B | 1.349 (3) |
| C6A—H6A2 | 0.9900 | C4B—C5B | 1.472 (3) |
| C7A—C8A | 1.490 (10) | C5B—C6B | 1.504 (3) |
| C7A—H7A1 | 0.9900 | C6B—C7B | 1.506 (3) |
| C7A—H7A2 | 0.9900 | C6B—H6BA | 0.9900 |
| C8A—C9A | 1.503 (7) | C6B—H6BB | 0.9900 |
| C8A—H8A1 | 0.9900 | C7B—C8B | 1.514 (3) |
| C8A—H8A2 | 0.9900 | C7B—H7BA | 0.9900 |
| C6AA—C7AA | 1.481 (13) | C7B—H7BB | 0.9900 |
| C6AA—H6A3 | 0.9900 | C8B—C9B | 1.500 (3) |
| C6AA—H6A4 | 0.9900 | C8B—H8BA | 0.9900 |
| C7AA—C8AA | 1.556 (13) | C8B—H8BB | 0.9900 |
| C7AA—H7A3 | 0.9900 | C10B—C11B | 1.386 (3) |
| C7AA—H7A4 | 0.9900 | C10B—C15B | 1.391 (3) |
| C8AA—C9A | 1.518 (14) | C11B—C12B | 1.388 (3) |
| C8AA—H8A3 | 0.9900 | C11B—H11B | 0.9500 |
| C8AA—H8A4 | 0.9900 | C12B—C13B | 1.378 (3) |
| C10A—C15A | 1.380 (3) | C12B—H12B | 0.9500 |
| C10A—C11A | 1.392 (3) | C13B—C14B | 1.382 (3) |
| C11A—C12A | 1.375 (4) | C14B—C15B | 1.381 (3) |
| C11A—H11A | 0.9500 | C14B—H14B | 0.9500 |
| C12A—C13A | 1.379 (4) | C15B—H15B | 0.9500 |
| C12A—H12A | 0.9500 | C16B—C21B | 1.368 (3) |
| C13A—C14A | 1.377 (3) | C16B—C17B | 1.374 (3) |
| C14A—C15A | 1.394 (3) | C17B—C18B | 1.385 (3) |
| C14A—H14A | 0.9500 | C17B—H17B | 0.9500 |
| C15A—H15A | 0.9500 | C18B—C19B | 1.376 (4) |
| C16A—C17A | 1.371 (3) | C18B—H18B | 0.9500 |
| C16A—C21A | 1.374 (3) | C19B—C20B | 1.378 (4) |
| C17A—C18A | 1.388 (4) | C19B—C24B | 1.506 (3) |
| C17A—H17A | 0.9500 | C20B—C21B | 1.383 (4) |
| C18A—C19A | 1.380 (4) | C20B—H20B | 0.9500 |
| C18A—H18A | 0.9500 | C21B—H21B | 0.9500 |
| C19A—C20A | 1.375 (4) | C23B—H23D | 0.9800 |
| C19A—C24A | 1.519 (3) | C23B—H23E | 0.9800 |
| C20A—C21A | 1.391 (3) | C23B—H23F | 0.9800 |
| C20A—H20A | 0.9500 | C24B—H24D | 0.9800 |
| C21A—H21A | 0.9500 | C24B—H24E | 0.9800 |
| C23A—H23A | 0.9800 | C24B—H24F | 0.9800 |
| | | | |
| C13A—O3A—C23A | 119.0 (2) | O3A—C23A—H23B | 109.5 |
| C1A—N1A—C9A | 122.95 (18) | H23A—C23A—H23B | 109.5 |
| C1A—N1A—C16A | 116.59 (17) | O3A—C23A—H23C | 109.5 |

| | | | |
|---|---|---|---|
| C9A—N1A—C16A | 120.30 (16) | H23A—C23A—H23C | 109.5 |
| O1A—C1A—N1A | 122.7 (2) | H23B—C23A—H23C | 109.5 |
| O1A—C1A—C2A | 123.00 (19) | C19A—C24A—H24A | 109.5 |
| N1A—C1A—C2A | 114.25 (17) | C19A—C24A—H24B | 109.5 |
| C22A—C2A—C1A | 109.44 (18) | H24A—C24A—H24B | 109.5 |
| C22A—C2A—C3A | 112.57 (19) | C19A—C24A—H24C | 109.5 |
| C1A—C2A—C3A | 113.61 (16) | H24A—C24A—H24C | 109.5 |
| C22A—C2A—H2A | 106.9 | H24B—C24A—H24C | 109.5 |
| C1A—C2A—H2A | 106.9 | C13B—O3B—C23B | 116.91 (19) |
| C3A—C2A—H2A | 106.9 | C1B—N1B—C9B | 122.42 (17) |
| C4A—C3A—C10A | 113.21 (16) | C1B—N1B—C16B | 116.28 (16) |
| C4A—C3A—C2A | 107.67 (17) | C9B—N1B—C16B | 121.24 (15) |
| C10A—C3A—C2A | 114.14 (17) | O1B—C1B—N1B | 122.5 (2) |
| C4A—C3A—H3A | 107.1 | O1B—C1B—C2B | 122.54 (18) |
| C10A—C3A—H3A | 107.1 | N1B—C1B—C2B | 114.93 (16) |
| C2A—C3A—H3A | 107.1 | C22B—C2B—C1B | 107.18 (18) |
| C9A—C4A—C5A | 120.91 (18) | C22B—C2B—C3B | 113.26 (19) |
| C9A—C4A—C3A | 122.37 (18) | C1B—C2B—C3B | 113.74 (15) |
| C5A—C4A—C3A | 116.70 (18) | C22B—C2B—H2B | 107.5 |
| O2A—C5A—C4A | 121.5 (2) | C1B—C2B—H2B | 107.5 |
| O2A—C5A—C6A | 122.6 (3) | C3B—C2B—H2B | 107.5 |
| C4A—C5A—C6A | 116.0 (3) | C4B—C3B—C10B | 110.53 (15) |
| O2A—C5A—C6AA | 116.4 (6) | C4B—C3B—C2B | 107.16 (17) |
| C4A—C5A—C6AA | 122.0 (6) | C10B—C3B—C2B | 116.00 (16) |
| C7A—C6A—C5A | 119.8 (5) | C4B—C3B—H3B | 107.6 |
| C7A—C6A—H6A1 | 107.4 | C10B—C3B—H3B | 107.6 |
| C5A—C6A—H6A1 | 107.4 | C2B—C3B—H3B | 107.6 |
| C7A—C6A—H6A2 | 107.4 | C9B—C4B—C5B | 120.73 (17) |
| C5A—C6A—H6A2 | 107.4 | C9B—C4B—C3B | 122.01 (17) |
| H6A1—C6A—H6A2 | 106.9 | C5B—C4B—C3B | 117.10 (18) |
| C6A—C7A—C8A | 114.0 (6) | O2B—C5B—C4B | 120.75 (19) |
| C6A—C7A—H7A1 | 108.7 | O2B—C5B—C6B | 121.47 (19) |
| C8A—C7A—H7A1 | 108.7 | C4B—C5B—C6B | 117.77 (18) |
| C6A—C7A—H7A2 | 108.7 | C5B—C6B—C7B | 113.01 (18) |
| C8A—C7A—H7A2 | 108.7 | C5B—C6B—H6BA | 109.0 |
| H7A1—C7A—H7A2 | 107.6 | C7B—C6B—H6BA | 109.0 |
| C7A—C8A—C9A | 113.4 (7) | C5B—C6B—H6BB | 109.0 |
| C7A—C8A—H8A1 | 108.9 | C7B—C6B—H6BB | 109.0 |
| C9A—C8A—H8A1 | 108.9 | H6BA—C6B—H6BB | 107.8 |
| C7A—C8A—H8A2 | 108.9 | C6B—C7B—C8B | 111.51 (18) |
| C9A—C8A—H8A2 | 108.9 | C6B—C7B—H7BA | 109.3 |
| H8A1—C8A—H8A2 | 107.7 | C8B—C7B—H7BA | 109.3 |
| C7AA—C6AA—C5A | 110.6 (11) | C6B—C7B—H7BB | 109.3 |
| C7AA—C6AA—H6A3 | 109.5 | C8B—C7B—H7BB | 109.3 |
| C5A—C6AA—H6A3 | 109.5 | H7BA—C7B—H7BB | 108.0 |
| C7AA—C6AA—H6A4 | 109.5 | C9B—C8B—C7B | 111.81 (18) |
| C5A—C6AA—H6A4 | 109.5 | C9B—C8B—H8BA | 109.3 |
| H6A3—C6AA—H6A4 | 108.1 | C7B—C8B—H8BA | 109.3 |

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|---|---|---|---|
| C6AA—C7AA—C8AA | 123.9 (12) | C9B—C8B—H8BB | 109.3 |
| C6AA—C7AA—H7A3 | 106.4 | C7B—C8B—H8BB | 109.3 |
| C8AA—C7AA—H7A3 | 106.4 | H8BA—C8B—H8BB | 107.9 |
| C6AA—C7AA—H7A4 | 106.4 | C4B—C9B—N1B | 120.81 (17) |
| C8AA—C7AA—H7A4 | 106.4 | C4B—C9B—C8B | 123.09 (18) |
| H7A3—C7AA—H7A4 | 106.4 | N1B—C9B—C8B | 116.10 (18) |
| C9A—C8AA—C7AA | 109.2 (12) | C11B—C10B—C15B | 117.5 (2) |
| C9A—C8AA—H8A3 | 109.8 | C11B—C10B—C3B | 122.08 (18) |
| C7AA—C8AA—H8A3 | 109.8 | C15B—C10B—C3B | 120.23 (19) |
| C9A—C8AA—H8A4 | 109.8 | C10B—C11B—C12B | 121.7 (2) |
| C7AA—C8AA—H8A4 | 109.8 | C10B—C11B—H11B | 119.2 |
| H8A3—C8AA—H8A4 | 108.3 | C12B—C11B—H11B | 119.2 |
| C4A—C9A—N1A | 120.92 (18) | C13B—C12B—C11B | 119.6 (2) |
| C4A—C9A—C8A | 121.5 (4) | C13B—C12B—H12B | 120.2 |
| N1A—C9A—C8A | 117.5 (4) | C11B—C12B—H12B | 120.2 |
| C4A—C9A—C8AA | 125.8 (7) | O3B—C13B—C12B | 123.5 (2) |
| N1A—C9A—C8AA | 113.1 (7) | O3B—C13B—C14B | 116.8 (2) |
| C15A—C10A—C11A | 117.6 (2) | C12B—C13B—C14B | 119.7 (2) |
| C15A—C10A—C3A | 123.09 (18) | C15B—C14B—C13B | 120.1 (2) |
| C11A—C10A—C3A | 119.35 (18) | C15B—C14B—H14B | 119.9 |
| C12A—C11A—C10A | 121.4 (2) | C13B—C14B—H14B | 119.9 |
| C12A—C11A—H11A | 119.3 | C14B—C15B—C10B | 121.2 (2) |
| C10A—C11A—H11A | 119.3 | C14B—C15B—H15B | 119.4 |
| C11A—C12A—C13A | 120.4 (2) | C10B—C15B—H15B | 119.4 |
| C11A—C12A—H12A | 119.8 | C21B—C16B—C17B | 119.6 (2) |
| C13A—C12A—H12A | 119.8 | C21B—C16B—N1B | 119.62 (19) |
| O3A—C13A—C14A | 125.0 (2) | C17B—C16B—N1B | 120.78 (19) |
| O3A—C13A—C12A | 115.6 (2) | C16B—C17B—C18B | 119.7 (2) |
| C14A—C13A—C12A | 119.4 (2) | C16B—C17B—H17B | 120.1 |
| C13A—C14A—C15A | 119.9 (2) | C18B—C17B—H17B | 120.1 |
| C13A—C14A—H14A | 120.1 | C19B—C18B—C17B | 121.7 (2) |
| C15A—C14A—H14A | 120.1 | C19B—C18B—H18B | 119.2 |
| C10A—C15A—C14A | 121.4 (2) | C17B—C18B—H18B | 119.2 |
| C10A—C15A—H15A | 119.3 | C18B—C19B—C20B | 117.4 (2) |
| C14A—C15A—H15A | 119.3 | C18B—C19B—C24B | 120.9 (3) |
| C17A—C16A—C21A | 120.6 (2) | C20B—C19B—C24B | 121.7 (3) |
| C17A—C16A—N1A | 120.6 (2) | C19B—C20B—C21B | 121.6 (2) |
| C21A—C16A—N1A | 118.7 (2) | C19B—C20B—H20B | 119.2 |
| C16A—C17A—C18A | 119.1 (2) | C21B—C20B—H20B | 119.2 |
| C16A—C17A—H17A | 120.4 | C16B—C21B—C20B | 120.0 (2) |
| C18A—C17A—H17A | 120.4 | C16B—C21B—H21B | 120.0 |
| C19A—C18A—C17A | 121.3 (2) | C20B—C21B—H21B | 120.0 |
| C19A—C18A—H18A | 119.3 | N2B—C22B—C2B | 179.0 (3) |
| C17A—C18A—H18A | 119.3 | O3B—C23B—H23D | 109.5 |
| C20A—C19A—C18A | 118.6 (2) | O3B—C23B—H23E | 109.5 |
| C20A—C19A—C24A | 119.8 (2) | H23D—C23B—H23E | 109.5 |
| C18A—C19A—C24A | 121.6 (2) | O3B—C23B—H23F | 109.5 |
| C19A—C20A—C21A | 120.8 (2) | H23D—C23B—H23F | 109.5 |

| | | | |
|---|---|---|---|
| C19A—C20A—H20A | 119.6 | H23E—C23B—H23F | 109.5 |
| C21A—C20A—H20A | 119.6 | C19B—C24B—H24D | 109.5 |
| C16A—C21A—C20A | 119.5 (2) | C19B—C24B—H24E | 109.5 |
| C16A—C21A—H21A | 120.2 | H24D—C24B—H24E | 109.5 |
| C20A—C21A—H21A | 120.2 | C19B—C24B—H24F | 109.5 |
| N2A—C22A—C2A | 178.0 (3) | H24D—C24B—H24F | 109.5 |
| O3A—C23A—H23A | 109.5 | H24E—C24B—H24F | 109.5 |

*Hydrogen-bond geometry (Å, º)*

*Cg*4 and *Cg*9 are the centroids of the C10*A*–C15*A* and C16*B*–C21*B* rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C21*A*—H21*A*···N2*B*$^{i}$ | 0.95 | 2.65 | 3.258 (4) | 123 |
| C14*B*—H14*B*···O3*A*$^{ii}$ | 0.95 | 2.65 | 3.417 (3) | 139 |
| C3*A*—H3*A*···O1*B* | 1.00 | 2.37 | 3.171 (2) | 136 |
| C23*B*—H23*E*···*Cg*9$^{iii}$ | 0.98 | 2.93 | 3.868 (3) | 160 |
| C23*B*—H23*F*···*Cg*4$^{iv}$ | 0.98 | 2.74 | 3.710 (3) | 169 |

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