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# Bis(azido-$\kappa N$)bis(quinolin-8-amine-$\kappa^2 N,N'$)iron(II) monohydrate

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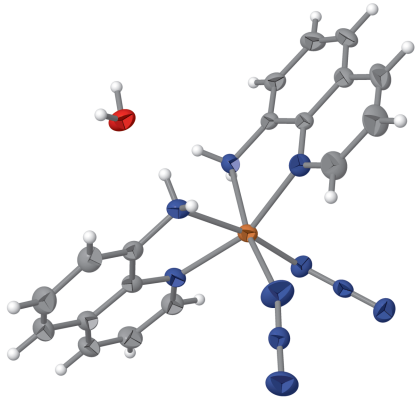
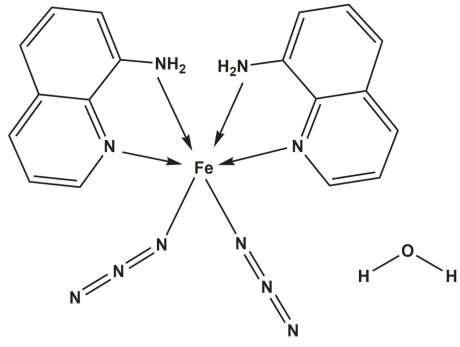
**Structural data:** full structural data are available from iucrdata.iucr.org

In the hydrated title complex, $[Fe(N_3)_2(AQ)_2]\cdot H_2O$ (AQ is 8-aminoquinoline, $C_9H_9N_2$), the $Fe^{II}$ ion is coordinated in a distorted octahedral manner by two neutral, chelating AQ ligands and two anionic, monodentate azide ($N_3^-$) ions in a *syn*,*cis*-configuration. From the two AQ ligands, the pyridyl N atoms are opposite to each other and the amino groups *trans* to the azide ligands. Distortion results from different Fe—N bond lengths [2.112 (2)–2.231 (2) Å] and $\langle$(N—Fe—N)$_{cis}$ [75.25 (6)–99.91 (7)°] and $\langle$(N—Fe—N)$_{trans}$ [159.98 (7)–170.62 (7)°] bond angles. The water molecule acts as the acceptor of hydrogen bonds with the $NH_2$ groups of both AQ-ligands in one and the same molecule, and as donor to the $\gamma$-N and $\alpha$-N atoms of the azido ligands of two adjacent iron complexes. In addition, both terminal N atoms of the azido ligands are involved in hydrogen bonds with $NH_2$ groups in neighboring iron complexes, so that the hydrogen-bonding pattern leads to a rod-like arrangement of the molecules in the *b*-axis direction.

3D view



Chemical scheme




## Structure description

Pseudohalide compounds derived from transition-metal ions are of great interest from the perspective of their magnetic properties, rich molecular architectures and for their topologies (Setifi, Ghazzali *et al.*, 2016, Setifi *et al.*, 2018, 2022; Merabet *et al.*, 2022). One of the pseudohalide ligands that has received much attention in the last decade is the azide [$N_3^-$] ion, partly due to its ability to produce a wide variety of coordination compounds with different nuclearities ranging from simple mononuclear to polynuclear species (Escuer & Aromi, 2006; Benamara *et al.*, 2021; Merabet *et al.*, 2023).

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Fe1—N1 | 2.142 (2) | Fe1—N4 | 2.112 (2) |
| N1—N2 | 1.159 (3) | N4—N5 | 1.202 (2) |
| N2—N3 | 1.166 (3) | N5—N6 | 1.156 (3) |
| | | | |
| N1—N2—N3 | 177.3 (3) | N4—N5—N6 | 179.2 (2) |
| N2—N1—Fe1 | 157.4 (2) | N5—N4—Fe1 | 122.3 (2) |

Up to now, mononuclear, octahedral iron(II) bis-azido complexes with bidentate Lewis bases $LB_{NN}$ having the general composition $Fe^{II}(LB_{NN})_2(N_3)_2$, are only known for $LB_{NN}$ = 1,10-phenanthroline (Miao *et al.* 2006), 4-amino-3,5-bis(2-pyridyl)-1,2,4-triazole (Setifi *et al.* 2021), and quinolin-8-amine (Setifi, Moon *et al.*, 2016). Very recently, this class of compounds was expanded by hydrates with the monohydrate $Fe(LB_{NN})_2(N_3)_2{\cdot}H_2O$ where $LB_{NN}$ = 2,2-dipyridylamine (Setifi *et al.*, 2024).

Here we report on the monohydrate of the quinolin-8-amine complex, $[Fe(N_3)_2(AQ)_2]{\cdot}H_2O$, revealing for the first time that hydrated as well as unhydrated forms of a specific azido iron(II) complex may exist and that the azido ligands in such complexes may have different orientations relative to each other. The compound was prepared under solvothermal conditions and its structure is described.

The title compound crystallizes in the orthorhombic space group *Pbcn* with eight formula units in the unit cell. The asymmetric unit therefore consists of one iron(II) complex and one water molecule both with all atoms in general positions (Fig. 1). The overall composition of the complex corresponds to $Fe^{II}(N_3)_2(AQ)_2$ with two neutral, chelating Lewis base molecules AQ = 8-aminoquinoline, and two monodentate azide ions, $N_3^-$, in a *cis* position. From the two AQ ligands, the pyridyl N atoms are *trans* and the two amino groups *cis* to each other.

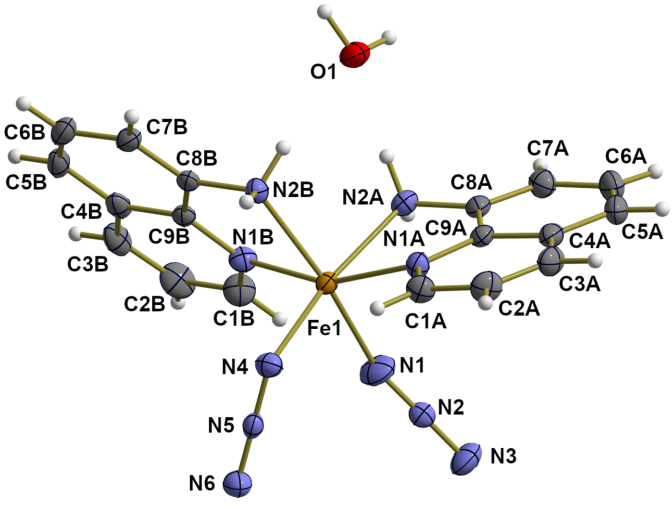



**Figure 1**
Displacement ellipsoid plot of the asymmetric unit of the title compound $Fe^{II}(LB_{NN})_2(N_3)_2{\cdot}H_2O$ ($LB_{NN}$ = AQ) showing the atom numbering. With the exception of the hydrogen atoms, which are shown as spheres of arbitrary radius, all atoms are drawn with displacement ellipsoids at the 40% probability level.

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N4$^{i}$ | 0.96 | 1.88 | 2.838 (3) | 175 |
| O1—H2···N3$^{ii}$ | 0.96 | 1.85 | 2.807 (3) | 174 |
| N2*A*—H11···N6$^{ii}$ | 0.89 | 2.17 | 3.052 (3) | 170 |
| N2*A*—H12···O1 | 0.89 | 2.28 | 3.086 (3) | 151 |
| N2*B*—H21···N3$^{iii}$ | 0.89 | 2.35 | 3.206 (3) | 160 |
| N2*B*—H22···O1 | 0.89 | 2.17 | 3.037 (2) | 164 |

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

The $Fe^{II}$ atom exhibits as usually a slightly distorted octahedral $\{FeN_6\}$ coordination (Table 1, Fig. 2). In contrast to the unhydrated compound (Setifi, Moon *et al.*, 2016), the two azido ligands have a *syn* orientation with an angle between them of 54.6 (6)°. A similar orientation was previously found in the triazane complex. Distortion results from different Fe—N bond lengths [$d$(Fe—$N_{azido}$) = 2.112 (2)/2.142 (2) Å, < $d$(Fe—$N_{AQ}$) = 2.177 (2)–2.231 (2) Å] and different bond angles [⟨($N_{Amine}$—Fe—$N_{Quinoline}$)$_{cis}$ = 75.25 (6)/76.06 (7)°, ⟨($N_{Amine}$—Fe—$N_{Azide}$)$_{cis}$ = 91.37 (7)/87.40 (8)°, ⟨($N_{Quinoline}$—Fe—$N_{Azide}$)$_{cis}$ = 92.83 (8)–99.91 (7)°, ⟨(N—Fe—N)$_{trans}$ = 170.62 (7)/159.98 (7)/168.94 (8)°].

Both azido ligands are slightly bent [177.3 (3)/179.2 (2)°] with N—N bond lengths [1.159 (3)–1.202 (2) Å] typical for formal N=N double bonds with the longer one to the metal-coordinating N atom. They are different to some extend because of different coordination modes: in the first azido ligand (N1–N3) the metal-coordinated nitrogen atom is additionally involved in a hydrogen bond (Table 2) to a hydrogen atom of the water molecule and the terminal

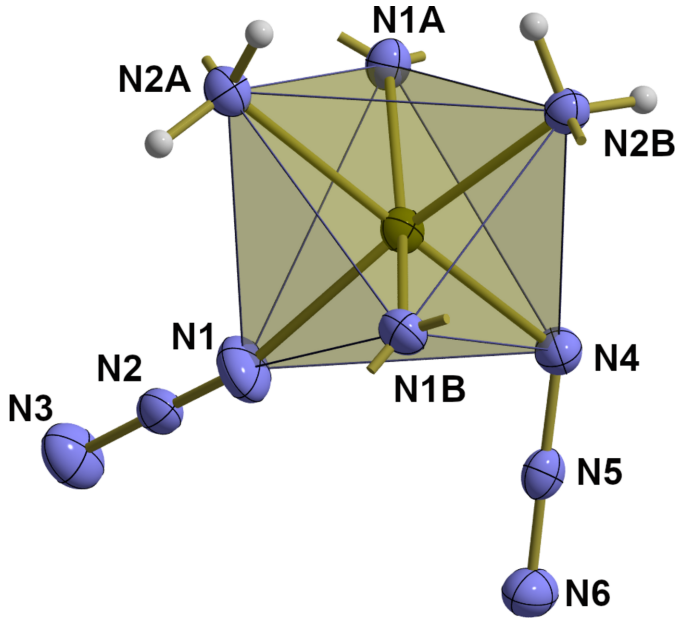



**Figure 2**
The $\{FeN_6\}$ octahedron in polyhedral representation, showing the *syn* orientation of both azido ligands. With the exception of the hydrogen atoms, which are shown as spheres of arbitrary radius, all atoms are drawn with displacement ellipsoids at the 40% probability level. The position of the carbon atoms attached to the nitrogen atoms of the ligands are indicated as shortened sticks.

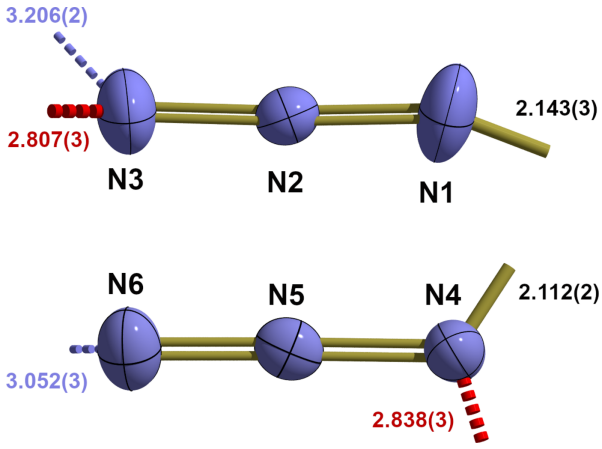



**Figure 3**
Displacement ellipsoid plot showing the two azido ligands in the iron(II) complex of the title compound in detail, with selected bond lengths (Å), hydrogen bonds [dashed, shortened sticks, $d(D\cdots A)$ in Å, O—H···N = red, N—H···N = blue] and dative bonds (shortened sticks) to the central iron atom. With the exception of the hydrogen atoms, which are shown as spheres of arbitrary radius, all atoms are drawn with displacement ellipsoids at the 40% probability level.

nitrogen atom in a hydrogen bond to a $NH_2$ group of AQ(B), while in the second azido ligand (N4–N6) the terminal nitrogen atom N6 is involved in two hydrogen bonds, one to a hydrogen atom of a the water molecule and second one to the hydrogen atom of a $NH_2$ group of AQ(A) (Fig. 3).

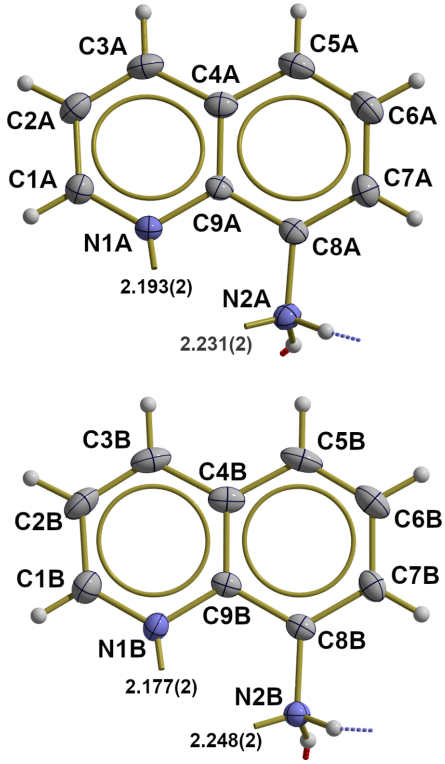



**Figure 4**
Displacement ellipsoid plot models showing the two 8-aminoquinoline ligand molecules in the iron(II) complex of the title compound in detail, with selected bond lengths (Å), bond angles (°), and hydrogen bonds (O—H···N as red, N—H···N as blue dashed lines). With the exception of the hydrogen atoms, which are shown as spheres of arbitrary radius, all atoms are drawn with displacement ellipsoids at the 40% probability level.

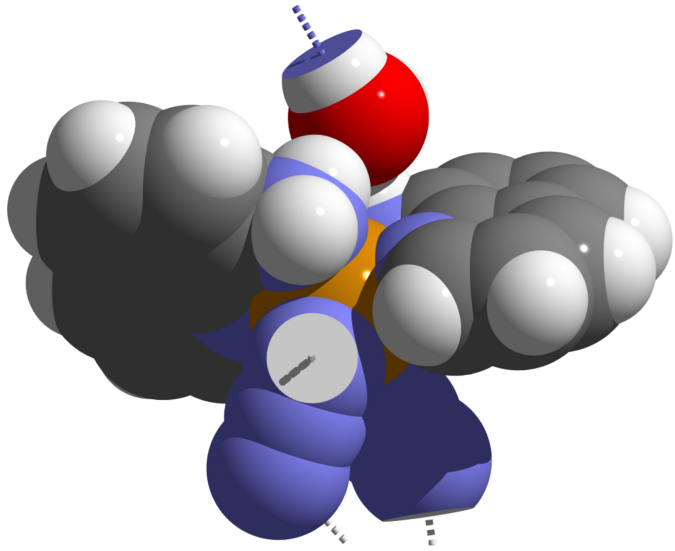


**Figure 5**
Space-filling model of one $[Fe^{II}(N_3)_2(AQ)_2]$ complex molecule and a water molecule visualizing the hydrogen-bonding scheme (dashed lines). Atoms are drawn as single-colored or truncated, two-colored spheres according to their van der Waals radii and cut-offs based on the intersection of the two spheres with cut-off faces showing the color of the interpenetrating atom. Atom colors and van der Waals radii (Å) are as follows: H = white/1.10, C = gray/1.70, N = blue/1.55, O = red/1.52/ and Fe = orange/2.00.

N—C and C—C bond lengths and angles in the quinoline ring systems of the two ligands (labeled with suffixes *A* and *B*; Fig. 4) are comparable to those of the pure AQ molecule (van Meervelt *et al.*, 1997) or the AQ molecules in the unhydrated $Fe^{II}$ complex (Setifi, Moon *et al.*, 2016) as are the bond lengths and angles of the attached $NH_2$ groups. Both amine groups act as hydrogen donors in hydrogen bonds to the oxygen atom O1 of the water molecule and to the terminal nitrogen atoms of the azide ligands: N2*A* to the N6 atom of the second azide ion and N2*B* to N3 of the first one. The water molecule also acts as a hydrogen donor in hydrogen bonds to the terminal nitrogen atom N3 of the first azide ligand and to the iron coordinated nitrogen atom N4 of the second one. Numerical details of the hydrogen bonds are summarized in Table 2 and visualized in Fig. 5.

In the crystal, the complex molecules are arranged in columns parallel to the *b*-axis *via* $N_{azide}\cdots H—O$ hydrogen bonds while the $O\cdots H_2—N$ hydrogen bonds act as bridges between the two amine groups of one and the same $Fe(N_3)_2$-$(AQ)_2$-molecule (Fig. 6).

## Synthesis and crystallization

The title compound was prepared solvothermally from a mixture of iron(II) bis(tetrafluoroborate) hexahydrate (34 mg, 0.1 mmol), 8-aminoquinoline (29 mg, 0.2 mmol) and sodium azide (13 mg, 0.2 mmol) in a mixture of water/ethanol (4:1 *v*/*v*, 25 ml). This mixture was sealed in a Teflon-lined autoclave and

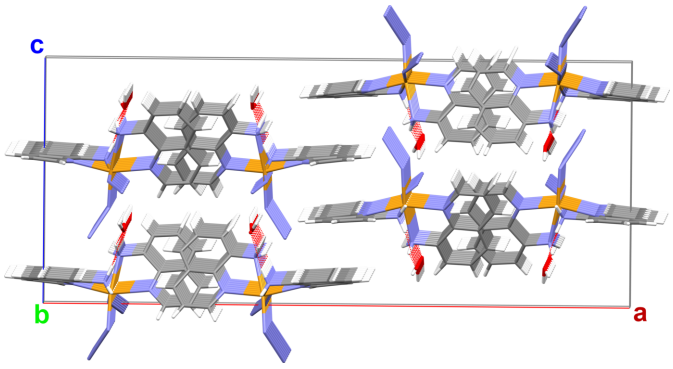



**Figure 6**
Stick model of the crystal packing down the crystallographic *b* axis. Color code: N = blue, H = white, C = gray, O = red, Fe = orange. O···$H_2$—N hydrogen bonds between the two amine groups of each molecule are shown with red dashed lines. The O—H···$N_{Azide}$ hydrogen bonds linking the molecules into columns parallel to the *b* axis are omitted for clarity.

held at 400 K for 2 d, and then cooled to ambient temperature at a rate of 10 K $h^{-1}$ to give the product in form of red plates (yield 36%).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $[Fe(N_3)_2(C_9H_8N_2)_2]\cdot H_2O$ |
| $M_r$ | 446.27 |
| Crystal system, space group | Orthorhombic, *Pbcn* |
| Temperature (K) | 293 |
| *a*, *b*, *c* (Å) | 32.5164 (15), 8.8531 (5), 13.5952 (6) |
| *V* (Å$^3$) | 3913.7 (3) |
| *Z* | 8 |
| Radiation type | Mo *Kα* |
| $\mu$ (mm$^{-1}$) | 0.81 |
| Crystal size (mm) | 0.27 × 0.11 × 0.05 |
| | |
| Data collection | |
| Diffractometer | Bruker APEXII CCD |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.789, 0.913 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 20186, 5690, 3470 |
| $R_{int}$ | 0.050 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.703 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.047, 0.100, 0.93 |
| No. of reflections | 5690 |
| No. of parameters | 273 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.50, −0.46 |

Computer programs: *APEX2* and *SAINT-Plus* (Bruker, 2019), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg, 2006), *Mercury* (Macrae *et al.*, 2020) and *publCIF* (Westrip, 2010).

## Acknowledgements

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# full crystallographic data

*IUCrData* (2025). **10**, x250235 [https://doi.org/10.1107/S2414314625002354]

## Bis(azido-$\kappa N$)bis(quinolin-8-amine-$\kappa^2 N,N'$)iron(II) monohydrate

**Fatima Setifi, Zouaoui Setifi, Hans Reuter, Mohammad Hadi Al-Douh and Abderezak Addala**

**Bis(azido-$\kappa N$)bis(quinolin-8-amine-$\kappa^2 N,N'$)iron(II) monohydrate**

*Crystal data*

$[Fe(N_3)_2(C_9H_8N_2)_2]\cdot H_2O$
$M_r$ = 446.27
Orthorhombic, *Pbcn*
$a$ = 32.5164 (15) Å
$b$ = 8.8531 (5) Å
$c$ = 13.5952 (6) Å
$V$ = 3913.7 (3) Å$^3$
$Z$ = 8
$F(000)$ = 1840
$D_x$ = 1.515 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 5258 reflections
$\theta$ = 2.4–28.5°
$\mu$ = 0.81 mm$^{-1}$
$T$ = 293 K
Plate, red
0.27 × 0.11 × 0.05 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: sealed X-ray tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.789, $T_{max}$ = 0.913
20186 measured reflections
5690 independent reflections
3470 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.050
$\theta_{max}$ = 30.0°, $\theta_{min}$ = 2.8°
$h$ = −45→45
$k$ = −12→8
$l$ = −19→13

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.100
$S$ = 0.93
5690 reflections
273 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.46 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.

**Refinement**. The positions of all H atoms were clearly identified in difference Fourier syntheses. Those of the organic ligands were refined with calculated positions (–CH– = 0.93 Å, –$NH_2$– = 0.89 Å) and isotropic displacement parameters depending on the equivalent isotropic temperature factor of the parent atoms. The position of the H atom of the water molecule were refined with fixed O—H distances of 0.96 Å and a bond angle of 104.95° before they were fixed and allowed to ride on the parent O atom with isotropic displacement parameters.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Fe1 | 0.38035 (2) | 0.21817 (4) | 0.56455 (2) | 0.02331 (9) |
| O1 | 0.35856 (5) | 0.4514 (2) | 0.82039 (12) | 0.0403 (4) |
| H1 | 0.3663 | 0.5464 | 0.8490 | 0.046 (8)* |
| H2 | 0.3573 | 0.3822 | 0.8747 | 0.080 (11)* |
| N1 | 0.38357 (7) | −0.0156 (3) | 0.52450 (17) | 0.0498 (6) |
| N2 | 0.37150 (5) | −0.1342 (2) | 0.50298 (13) | 0.0265 (4) |
| N3 | 0.36072 (7) | −0.2560 (2) | 0.48292 (19) | 0.0475 (6) |
| N4 | 0.38371 (6) | 0.2776 (2) | 0.41435 (13) | 0.0298 (4) |
| N5 | 0.40034 (5) | 0.1988 (2) | 0.35437 (13) | 0.0259 (4) |
| N6 | 0.41626 (6) | 0.1243 (2) | 0.29588 (15) | 0.0382 (5) |
| N1A | 0.31339 (5) | 0.2393 (2) | 0.57831 (13) | 0.0268 (4) |
| C1A | 0.28792 (7) | 0.3007 (3) | 0.51410 (17) | 0.0325 (5) |
| H1A | 0.2989 | 0.3567 | 0.4626 | 0.039* |
| C2A | 0.24486 (7) | 0.2857 (3) | 0.51992 (18) | 0.0383 (6) |
| H2A | 0.2280 | 0.3338 | 0.4744 | 0.046* |
| C3A | 0.22820 (7) | 0.2004 (3) | 0.59249 (18) | 0.0363 (6) |
| H3A | 0.1998 | 0.1876 | 0.5960 | 0.044* |
| C4A | 0.25394 (7) | 0.1311 (3) | 0.66260 (16) | 0.0301 (5) |
| C5A | 0.23947 (7) | 0.0367 (3) | 0.73857 (18) | 0.0374 (6) |
| H5A | 0.2115 | 0.0178 | 0.7448 | 0.045* |
| C6A | 0.26660 (8) | −0.0267 (3) | 0.80300 (19) | 0.0401 (6) |
| H6A | 0.2570 | −0.0923 | 0.8512 | 0.048* |
| C7A | 0.30896 (7) | 0.0055 (3) | 0.79768 (17) | 0.0333 (5) |
| H7A | 0.3268 | −0.0367 | 0.8434 | 0.040* |
| C8A | 0.32400 (6) | 0.0983 (2) | 0.72585 (15) | 0.0258 (5) |
| C9A | 0.29669 (6) | 0.1584 (2) | 0.65421 (15) | 0.0243 (5) |
| N2A | 0.36646 (5) | 0.1420 (2) | 0.71743 (13) | 0.0271 (4) |
| H11 | 0.3825 | 0.0640 | 0.7328 | 0.033* |
| H12 | 0.3718 | 0.2166 | 0.7595 | 0.033* |
| N1B | 0.44530 (5) | 0.2273 (2) | 0.60288 (14) | 0.0283 (4) |
| C1B | 0.47090 (7) | 0.1127 (3) | 0.60427 (19) | 0.0395 (6) |
| H1B | 0.4602 | 0.0158 | 0.5973 | 0.047* |
| C2B | 0.51386 (7) | 0.1293 (3) | 0.61585 (19) | 0.0441 (7) |
| H2B | 0.5308 | 0.0446 | 0.6181 | 0.053* |
| C3B | 0.53032 (7) | 0.2701 (3) | 0.62365 (18) | 0.0380 (6) |
| H3B | 0.5586 | 0.2825 | 0.6297 | 0.046* |
| C4B | 0.50415 (7) | 0.3966 (3) | 0.62245 (15) | 0.0298 (5) |
| C5B | 0.51836 (7) | 0.5466 (3) | 0.62898 (17) | 0.0377 (6) |
| H5B | 0.5464 | 0.5656 | 0.6338 | 0.045* |

| | | | | |
|---|---|---|---|---|
| C6B | 0.49145 (8) | 0.6632 (3) | 0.62832 (17) | 0.0392 (6) |
| H6B | 0.5013 | 0.7617 | 0.6319 | 0.047* |
| C7B | 0.44891 (7) | 0.6373 (3) | 0.62234 (16) | 0.0302 (5) |
| H7B | 0.4308 | 0.7187 | 0.6230 | 0.036* |
| C8B | 0.43379 (6) | 0.4934 (3) | 0.61555 (14) | 0.0238 (5) |
| C9B | 0.46122 (6) | 0.3697 (3) | 0.61423 (14) | 0.0236 (5) |
| N2B | 0.39027 (5) | 0.4594 (2) | 0.61037 (13) | 0.0251 (4) |
| H21 | 0.3782 | 0.5207 | 0.5672 | 0.030* |
| H22 | 0.3788 | 0.4749 | 0.6689 | 0.030* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Fe1 | 0.02297 (14) | 0.02148 (16) | 0.02548 (15) | −0.00273 (14) | −0.00115 (13) | −0.00175 (15) |
| O1 | 0.0536 (11) | 0.0335 (10) | 0.0338 (10) | −0.0087 (8) | −0.0002 (8) | −0.0067 (9) |
| N1 | 0.0737 (16) | 0.0235 (12) | 0.0521 (14) | −0.0043 (12) | −0.0006 (12) | −0.0056 (11) |
| N2 | 0.0255 (9) | 0.0261 (12) | 0.0279 (10) | 0.0029 (8) | 0.0000 (7) | 0.0004 (9) |
| N3 | 0.0475 (13) | 0.0320 (14) | 0.0631 (16) | −0.0089 (11) | −0.0041 (12) | −0.0109 (12) |
| N4 | 0.0368 (10) | 0.0269 (10) | 0.0257 (10) | 0.0016 (9) | 0.0013 (8) | −0.0001 (9) |
| N5 | 0.0241 (9) | 0.0263 (11) | 0.0273 (10) | −0.0077 (8) | −0.0012 (8) | 0.0022 (9) |
| N6 | 0.0432 (12) | 0.0336 (13) | 0.0377 (12) | −0.0038 (10) | 0.0089 (9) | −0.0066 (10) |
| N1A | 0.0256 (9) | 0.0272 (11) | 0.0276 (10) | −0.0035 (8) | −0.0023 (7) | 0.0017 (8) |
| C1A | 0.0333 (12) | 0.0334 (15) | 0.0308 (12) | 0.0009 (11) | −0.0029 (10) | 0.0028 (11) |
| C2A | 0.0309 (12) | 0.0454 (16) | 0.0386 (13) | 0.0035 (12) | −0.0093 (10) | 0.0010 (13) |
| C3A | 0.0225 (11) | 0.0443 (17) | 0.0420 (14) | −0.0027 (11) | −0.0023 (9) | −0.0069 (13) |
| C4A | 0.0279 (11) | 0.0295 (14) | 0.0328 (12) | −0.0033 (10) | 0.0015 (10) | −0.0077 (11) |
| C5A | 0.0322 (13) | 0.0398 (17) | 0.0401 (14) | −0.0115 (12) | 0.0090 (11) | −0.0048 (12) |
| C6A | 0.0456 (14) | 0.0380 (17) | 0.0369 (14) | −0.0093 (12) | 0.0102 (12) | 0.0035 (13) |
| C7A | 0.0385 (13) | 0.0283 (13) | 0.0332 (13) | 0.0006 (11) | 0.0025 (10) | 0.0028 (12) |
| C8A | 0.0291 (11) | 0.0220 (12) | 0.0263 (11) | −0.0025 (9) | 0.0018 (9) | −0.0045 (10) |
| C9A | 0.0266 (11) | 0.0195 (11) | 0.0269 (11) | −0.0031 (9) | −0.0005 (9) | −0.0031 (10) |
| N2A | 0.0273 (9) | 0.0244 (11) | 0.0295 (10) | −0.0010 (8) | −0.0048 (7) | −0.0021 (9) |
| N1B | 0.0283 (9) | 0.0254 (10) | 0.0312 (10) | 0.0048 (9) | −0.0004 (8) | −0.0008 (9) |
| C1B | 0.0365 (14) | 0.0353 (16) | 0.0467 (15) | 0.0071 (12) | 0.0001 (11) | 0.0025 (13) |
| C2B | 0.0319 (14) | 0.0491 (19) | 0.0513 (16) | 0.0178 (13) | −0.0006 (12) | 0.0036 (15) |
| C3B | 0.0214 (11) | 0.0588 (18) | 0.0337 (13) | 0.0030 (12) | −0.0001 (9) | 0.0034 (13) |
| C4B | 0.0250 (11) | 0.0462 (16) | 0.0183 (11) | −0.0046 (11) | −0.0018 (8) | 0.0017 (11) |
| C5B | 0.0281 (12) | 0.0561 (18) | 0.0288 (13) | −0.0189 (12) | −0.0016 (10) | 0.0007 (12) |
| C6B | 0.0449 (15) | 0.0380 (15) | 0.0349 (14) | −0.0177 (13) | −0.0025 (11) | −0.0014 (12) |
| C7B | 0.0382 (13) | 0.0256 (13) | 0.0268 (12) | −0.0056 (11) | −0.0002 (9) | −0.0026 (11) |
| C8B | 0.0258 (11) | 0.0287 (12) | 0.0170 (10) | −0.0054 (9) | 0.0011 (8) | −0.0012 (9) |
| C9B | 0.0236 (10) | 0.0299 (13) | 0.0174 (10) | −0.0044 (10) | −0.0009 (8) | −0.0002 (10) |
| N2B | 0.0230 (9) | 0.0261 (11) | 0.0260 (10) | −0.0005 (8) | 0.0006 (7) | 0.0005 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Fe1—N1 | 2.142 (2) | C7A—C8A | 1.367 (3) |
| Fe1—N1B | 2.177 (2) | C7A—H7A | 0.9300 |

| | | | |
|---|---|---|---|
| Fe1—N1A | 2.193 (2) | C8A—C9A | 1.421 (3) |
| Fe1—N2A | 2.231 (2) | C8A—N2A | 1.438 (3) |
| Fe1—N2B | 2.248 (2) | N2A—H11 | 0.8900 |
| O1—H1 | 0.9600 | N2A—H12 | 0.8900 |
| O1—H2 | 0.9600 | N1B—C1B | 1.313 (3) |
| N1—N2 | 1.159 (3) | N1B—C9B | 1.372 (3) |
| N2—N3 | 1.166 (3) | C1B—C2B | 1.413 (3) |
| Fe1—N4 | 2.112 (2) | C1B—H1B | 0.9300 |
| N4—N5 | 1.202 (2) | C2B—C3B | 1.361 (4) |
| N5—N6 | 1.156 (3) | C2B—H2B | 0.9300 |
| N1A—C1A | 1.321 (3) | C3B—C4B | 1.407 (3) |
| N1A—C9A | 1.368 (3) | C3B—H3B | 0.9300 |
| C1A—C2A | 1.409 (3) | C4B—C5B | 1.409 (3) |
| C1A—H1A | 0.9300 | C4B—C9B | 1.421 (3) |
| C2A—C3A | 1.356 (3) | C5B—C6B | 1.353 (4) |
| C2A—H2A | 0.9300 | C5B—H5B | 0.9300 |
| C3A—C4A | 1.409 (3) | C6B—C7B | 1.405 (3) |
| C3A—H3A | 0.9300 | C6B—H6B | 0.9300 |
| C4A—C5A | 1.409 (3) | C7B—C8B | 1.369 (3) |
| C4A—C9A | 1.415 (3) | C7B—H7B | 0.9300 |
| C5A—C6A | 1.364 (3) | C8B—C9B | 1.412 (3) |
| C5A—H5A | 0.9300 | C8B—N2B | 1.449 (2) |
| C6A—C7A | 1.409 (3) | N2B—H21 | 0.8900 |
| C6A—H6A | 0.9300 | N2B—H22 | 0.8900 |

| | | | |
|---|---|---|---|
| N4—Fe1—N1 | 89.57 (8) | C9A—C8A—N2A | 116.40 (19) |
| N4—Fe1—N1B | 99.91 (7) | N1A—C9A—C4A | 122.67 (19) |
| N1—Fe1—N1B | 92.82 (8) | N1A—C9A—C8A | 117.69 (18) |
| N4—Fe1—N1A | 96.47 (7) | C4A—C9A—C8A | 119.6 (2) |
| N1—Fe1—N1A | 98.77 (8) | C8A—N2A—Fe1 | 110.46 (12) |
| N1B—Fe1—N1A | 159.98 (7) | C8A—N2A—H11 | 109.6 |
| N4—Fe1—N2A | 170.62 (7) | Fe1—N2A—H11 | 109.6 |
| N1—Fe1—N2A | 87.41 (8) | C8A—N2A—H12 | 109.6 |
| N1B—Fe1—N2A | 89.12 (7) | Fe1—N2A—H12 | 109.6 |
| N1A—Fe1—N2A | 75.25 (6) | H11—N2A—H12 | 108.1 |
| N4—Fe1—N2B | 91.37 (7) | C1B—N1B—C9B | 118.0 (2) |
| N1—Fe1—N2B | 168.84 (8) | C1B—N1B—Fe1 | 126.16 (17) |
| N1B—Fe1—N2B | 76.06 (7) | C9B—N1B—Fe1 | 115.29 (14) |
| N1A—Fe1—N2B | 92.18 (6) | N1B—C1B—C2B | 123.2 (3) |
| N2A—Fe1—N2B | 93.32 (7) | N1B—C1B—H1B | 118.4 |
| H1—O1—H2 | 105.0 | C2B—C1B—H1B | 118.4 |
| N1—N2—N3 | 177.3 (3) | C3B—C2B—C1B | 119.5 (2) |
| N2—N1—Fe1 | 157.4 (2) | C3B—C2B—H2B | 120.2 |
| N4—N5—N6 | 179.2 (2) | C1B—C2B—H2B | 120.2 |
| N5—N4—Fe1 | 122.3 (2) | C2B—C3B—C4B | 119.4 (2) |
| C1A—N1A—C9A | 117.74 (19) | C2B—C3B—H3B | 120.3 |
| C1A—N1A—Fe1 | 126.96 (16) | C4B—C3B—H3B | 120.3 |
| C9A—N1A—Fe1 | 114.43 (13) | C3B—C4B—C5B | 123.5 (2) |

| | | | |
|---|---|---|---|
| N1A—C1A—C2A | 123.2 (2) | C3B—C4B—C9B | 117.5 (2) |
| N1A—C1A—H1A | 118.4 | C5B—C4B—C9B | 119.0 (2) |
| C2A—C1A—H1A | 118.4 | C6B—C5B—C4B | 120.4 (2) |
| C3A—C2A—C1A | 119.4 (2) | C6B—C5B—H5B | 119.8 |
| C3A—C2A—H2A | 120.3 | C4B—C5B—H5B | 119.8 |
| C1A—C2A—H2A | 120.3 | C5B—C6B—C7B | 120.9 (2) |
| C2A—C3A—C4A | 119.8 (2) | C5B—C6B—H6B | 119.6 |
| C2A—C3A—H3A | 120.1 | C7B—C6B—H6B | 119.6 |
| C4A—C3A—H3A | 120.1 | C8B—C7B—C6B | 120.6 (2) |
| C5A—C4A—C3A | 123.7 (2) | C8B—C7B—H7B | 119.7 |
| C5A—C4A—C9A | 119.2 (2) | C6B—C7B—H7B | 119.7 |
| C3A—C4A—C9A | 117.0 (2) | C7B—C8B—C9B | 119.7 (2) |
| C6A—C5A—C4A | 119.9 (2) | C7B—C8B—N2B | 123.2 (2) |
| C6A—C5A—H5A | 120.0 | C9B—C8B—N2B | 117.08 (19) |
| C4A—C5A—H5A | 120.0 | N1B—C9B—C8B | 118.40 (18) |
| C5A—C6A—C7A | 121.1 (2) | N1B—C9B—C4B | 122.3 (2) |
| C5A—C6A—H6A | 119.5 | C8B—C9B—C4B | 119.3 (2) |
| C7A—C6A—H6A | 119.5 | C8B—N2B—Fe1 | 110.56 (13) |
| C8A—C7A—C6A | 120.6 (2) | C8B—N2B—H21 | 109.5 |
| C8A—C7A—H7A | 119.7 | Fe1—N2B—H21 | 109.5 |
| C6A—C7A—H7A | 119.7 | C8B—N2B—H22 | 109.5 |
| C7A—C8A—C9A | 119.4 (2) | Fe1—N2B—H22 | 109.5 |
| C7A—C8A—N2A | 124.2 (2) | H21—N2B—H22 | 108.1 |

| | | | |
|---|---|---|---|
| C9A—N1A—C1A—C2A | −0.6 (3) | C9B—N1B—C1B—C2B | −0.6 (4) |
| Fe1—N1A—C1A—C2A | 168.09 (18) | Fe1—N1B—C1B—C2B | 170.49 (18) |
| N1A—C1A—C2A—C3A | −2.2 (4) | N1B—C1B—C2B—C3B | −1.7 (4) |
| C1A—C2A—C3A—C4A | 1.6 (4) | C1B—C2B—C3B—C4B | 1.5 (4) |
| C2A—C3A—C4A—C5A | −177.9 (2) | C2B—C3B—C4B—C5B | −179.3 (2) |
| C2A—C3A—C4A—C9A | 1.5 (3) | C2B—C3B—C4B—C9B | 0.8 (3) |
| C3A—C4A—C5A—C6A | 179.5 (2) | C3B—C4B—C5B—C6B | −179.4 (2) |
| C9A—C4A—C5A—C6A | 0.1 (3) | C9B—C4B—C5B—C6B | 0.6 (3) |
| C4A—C5A—C6A—C7A | 2.8 (4) | C4B—C5B—C6B—C7B | 0.8 (4) |
| C5A—C6A—C7A—C8A | −1.8 (4) | C5B—C6B—C7B—C8B | −1.0 (3) |
| C6A—C7A—C8A—C9A | −2.1 (3) | C6B—C7B—C8B—C9B | −0.2 (3) |
| C6A—C7A—C8A—N2A | 177.4 (2) | C6B—C7B—C8B—N2B | 179.2 (2) |
| C1A—N1A—C9A—C4A | 4.0 (3) | C1B—N1B—C9B—C8B | −178.3 (2) |
| Fe1—N1A—C9A—C4A | −166.08 (17) | Fe1—N1B—C9B—C8B | 9.6 (2) |
| C1A—N1A—C9A—C8A | −177.1 (2) | C1B—N1B—C9B—C4B | 3.1 (3) |
| Fe1—N1A—C9A—C8A | 12.9 (2) | Fe1—N1B—C9B—C4B | −168.97 (15) |
| C5A—C4A—C9A—N1A | 175.0 (2) | C7B—C8B—C9B—N1B | −177.09 (19) |
| C3A—C4A—C9A—N1A | −4.5 (3) | N2B—C8B—C9B—N1B | 3.4 (3) |
| C5A—C4A—C9A—C8A | −3.9 (3) | C7B—C8B—C9B—C4B | 1.6 (3) |
| C3A—C4A—C9A—C8A | 176.6 (2) | N2B—C8B—C9B—C4B | −177.91 (17) |
| C7A—C8A—C9A—N1A | −174.1 (2) | C3B—C4B—C9B—N1B | −3.2 (3) |
| N2A—C8A—C9A—N1A | 6.4 (3) | C5B—C4B—C9B—N1B | 176.8 (2) |
| C7A—C8A—C9A—C4A | 4.9 (3) | C3B—C4B—C9B—C8B | 178.22 (19) |
| N2A—C8A—C9A—C4A | −174.59 (19) | C5B—C4B—C9B—C8B | −1.7 (3) |

| | | | |
|---|---|---|---|
| C7A—C8A—N2A—Fe1 | 158.94 (18) | C7B—C8B—N2B—Fe1 | 166.65 (17) |
| C9A—C8A—N2A—Fe1 | −21.6 (2) | C9B—C8B—N2B—Fe1 | −13.9 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N4$^{i}$ | 0.96 | 1.88 | 2.838 (3) | 175 |
| O1—H2···N3$^{ii}$ | 0.96 | 1.85 | 2.807 (3) | 174 |
| N2*A*—H11···N6$^{ii}$ | 0.89 | 2.17 | 3.052 (3) | 170 |
| N2*A*—H12···O1 | 0.89 | 2.28 | 3.086 (3) | 151 |
| N2*B*—H21···N3$^{iii}$ | 0.89 | 2.35 | 3.206 (3) | 160 |
| N2*B*—H22···O1 | 0.89 | 2.17 | 3.037 (2) | 164 |

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