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# A compound of a novel tetraaza-macrocycle with trinuclear tetracyanonickelate-bridged cations

**Neil F. Curtis[a]* and Horst Puschmann[b]**

[a]School of Chemical and Physical Sciences, Victoria University of Wellington, Box 600, Wellington, New Zealand, and [b]Durham University Chemical Crystallography Group, Durham DH1 3LE, England
Correspondence e-mail: neil.curtis@vuw.ac.nz

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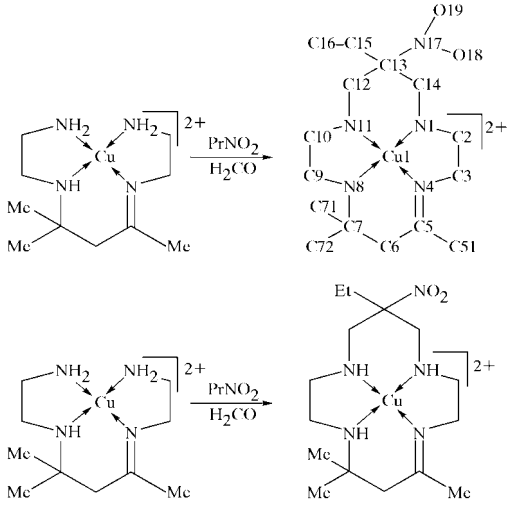
The cation of the title compound, $[Cu(L)]^{2+}$, is formed by Michael condensation of (4,6,6-trimethyl-3,7-diazanon-3-ene-1,9-diamine)copper(II) with methanal and nitropropane. This cation forms a tetracyanonickelate(II) compound, the unit cell of which contains two centrosymmetric tetracyanonickelate(II)-bridged trinuclear cations, namely diaqua-1,3$\kappa^2O$-di-$\mu$-cyano-1:2$\kappa^2C$:$N$;1:3$\kappa^2C$:$N$-dicyano-1$\kappa^2C$-bis(13-ethyl-5,7,7-trimethyl-13-nitro-1,4,8,11-tetraazacyclotetradec-4-ene)-2$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$;3$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$-dicopper(II)-nickel(II) di-$\mu$-cyano-1:2$\kappa^2C$:$N$;1:3$\kappa^2C$:$N$-dicyano-1$\kappa^2C$-bis(13-ethyl-5,7,7-trimethyl-13-nitro-1,4,8,11-tetraazacyclotetradec-4-ene)-2$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$;3$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$-dicopper(II)-nickel(II) bis[tetracyanonickelate(II)] octahydrate, $[Cu_2Ni(CN)_4(C_{15}H_{31}N_5O_2)_2(H_2O)_2][Cu_2Ni(CN)_4(C_{15}H_{31}N_5O_2)_2][Ni(CN)_4]_2\cdot 8H_2O$. One cation, $[(L)Cu–NC–Ni(CN)_2–CN–Cu(L)]^{2+}$, has an axially coordinated bridging $[Ni(CN)_4]^{2-}$ ion, with a Cu—N distance of 2.226 (3) Å and a Cu—N—C angle of 168.2 (3)°. The other cation, $[(H_2O)(L)Cu–NC–Ni(CN)_2–CN–Cu(L)(OH_2)]^{2+}$, has water axially coordinated *trans* to a weakly bound bridging $[Ni(CN)_4]^{2-}$ ion, with a Cu—O distance of 2.396 (3) Å, a Cu—N distance of 2.677 (4) Å, an O—Cu—N angle of 168.7 (1)° and a Cu—N—C angle of 137.7 (3)°. These cations, plus independent $[Ni(CN)_4]^{2-}$ ions and water molecules, are linked into a hydrogen-bonded network. All $[Ni(CN)_4]^{2-}$ ions are on centres of symmetry.

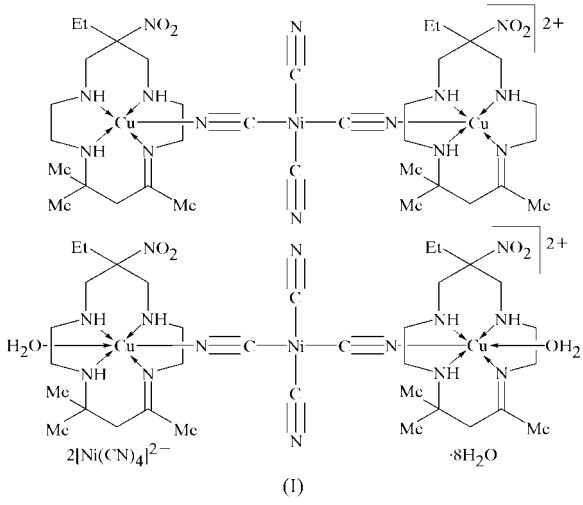
## Comment

Michael condensations of (polyamine)metal complexes with methanal and nitroalkanes form nitroalkyl-substituted cyclic amine complexes (Lawrance, Lye *et al.*, 1993; Lawrance, Maeder *et al.*, 1993; Comba *et al.*, 1986), such as 6-methyl-6-nitro-1,4,8,11-tetraazacyclotetradecane)copper(II), which is formed from (3,7-diazanonane-1,9-diamine)metal compounds, methanal and nitroethane (Comba *et al.*, 1988*a*,*b*). The (tetraaza-macrocycle)copper(II) cation, $[Cu(L)]^{2+}$, present in the title compound, formed by an analogous reaction of (4,6,6-trimethyl-3,7-diazanon-3-ene-1,9-diamine)copper(II) with methanal and nitropropane, differs by the presence of the imine function, the introduction of the 5,7,7-trimethyl substituents and the substitution of a 6-ethyl substituent for 6-methyl.




Structures of a number of methyl/nitro-substituted aza-macrocycle compounds obtained by reaction of (amine)metal compounds with nitroethane and methanal have been reported, but this is the first for an ethyl/nitro-substituted analogue derived from nitropropane.

The structures of many compounds of (amine)metal cations with cyanometallate anions have been reported, often with oligo- or polymeric structures with bridging cyanometallate ions (Cernak *et al.*, 2002).




(I)

The title compound, (I), which crystallizes from aqueous solutions containing $[Ni(CN)_4]^{2-}$ and $[Cu(L)]^{2+}$, has the formal composition $[Cu(L)][Ni(CN)_4]\cdot 2.5H_2O$, but has two structurally distinct centrosymmetric tetracyanonickelate(II)-bridged (aza-macrocycle)copper(II) trinuclear cations, two independent tetracyanonickelate(II) anions, and one coordinated and four uncoordinated water molecules (see Fig. 1 and Table 1).

Atom Cu1*A* is in a square-planar coordination environment formed by the three secondary amine atoms, *viz*. N1*A*, N8*A* and N11*A*, and imine atom N4*A* of macrocycle $L^a$, with atom

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N55 of the $[Ni5(CN)_4]^{2-}$ tetracyanonickelate(II) ion coordinated axially; the result is a centrosymmetric trinuclear cation, $[(L^a)Cu–NC–Ni(CN)_2–CN–Cu(L^a)]^{2+}$, with a Cu···Cu separation of 10.426 (5) Å (see Fig. 2).

Atom Cu1*B* is in a square-planar coordination environment formed by the four N atoms, *viz*. N1*B*, N4*B*, N8*B* and N11*B*, of macrocycle $L^b$, with weaker axial interactions with water atom O10 and atom N65 of the $[Ni6(CN)_4]^{2-}$ ion forming a weakly bound centrosymmetric trinuclear cation, $[(H_2O)(L^b)Cu–NC–Ni(CN)_2–CN–Cu(L^b)(OH_2)]^{2+}$, with a Cu···Cu separation of 10.599 (5) Å (see Fig. 3).

For the two (aza-macrocycle)copper(II) cations, the Cu—$N_{ring}$ distances are similar (with the Cu—$N_{imine}$ distance *ca* 0.03 Å shorter than the mean Cu—$N_{amine}$ distance), the configuration is the same (1*S*,8*R*,11*R*; Spek, 2002) and the conformations are similar. The nitro group and the C72 methyl component of the gem-dimethyl group are axially oriented on the same side of the $N_4$ macrocycle coordination plane as the N1—H1 and N11—H11 groups, with the N8—H8 group and the axial ligand (N55 for Cu1*A* and O10 for Cu1*B*) on the other side. The $N_4$ plane is less tetrahedrally twisted and the Cu atom is further displaced from this plane for the $[Cu1A(L^a)]^{2+}$ ion [±0.017 (2) and 0.246 (2) Å] than for the $[Cu1B(L^b)]^{2+}$ ion [±0.067 (2) and 0.088 (2) Å]; these planes are inclined at 30.9 (2)°. The C15 methylene substituents of both macrocycles are equatorially oriented, with the terminal methyl group, C16*A*, of the $[Cu(L^a)]^{2+}$ ion further equatorially extended and closer to atom O18*A*, while the C16*B* group is axially oriented on the same side as axial water ligand O10.

The coordinated isocyano atom N55 is close to the square-pyramidal axis of the $[Cu1A(L^a)]^{2+}$ ion, with $N_{ring}$—Cu1*A*—N55 angles of between 95.1 (1) and 99.3 (1)°. The non-bridging N56—C56—Ni5 group is approximately aligned with the C7*A*···C14*A* axis [N56···Ni5···Cu1*A*—C7*A* = −1.4 (2)°]. The ion is tilted with respect to the $N_4$ coordination plane so that the N8*A*···N56 distance [6.250 (5) Å] is longer than the N1*A*···N56($-x$, $2-y$, $-z$) distance [4.998 (5) Å].

For $[Cu1B(L^b)]^{2+}$, the coordinated water O and isocyano N atoms are displaced from the square-bipyramidal axis, with $N_{ring}$—Cu1*B*—O10 angles of 87.1 (1)–101.8 (1)° and $N_{ring}$—Cu1*B*—N65 angles of 81.9 (1)–96.8 (1)°.

The dimensions of the coordinated and non-coordinated $[Ni(CN)_4]^{2-}$ ions, all centrosymmetric, do not differ significantly. The two tetracyanonickelate(II) anions including atoms Ni7 and Ni8, and the water molecules including atoms O11, O12, O13 and O14 have no direct interaction with the copper(II) cations, though all are linked into a hydrogen-bonding network (see Table 2).

Chain polymeric structures are present for bis(ethane-1,2-diamine)copper(II) tetracyanonickelate(II), [–Cu(en)$_2$–NC–Ni(CN)$_2$–CN–Cu(en)$_2$–] (Luo *et al.*, 2000; Lokaj *et al.*, 1991),

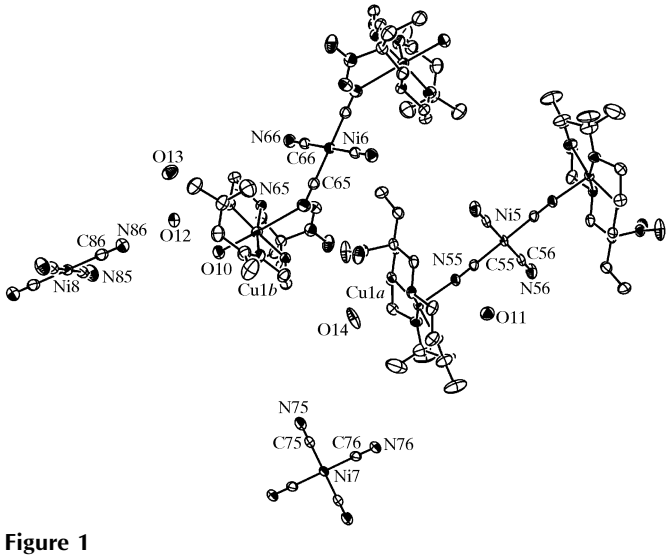


**Figure 1**
The structure of the title compound, drawn with displacement ellipsoids at the 50% probability level for non-H atoms, showing the asymmetric unit (labelled atoms and atoms of associated macrocycles), with additional atoms generated by symmetry operations to complete the tetracyanonickelate(II) anions and trinuclear cations.

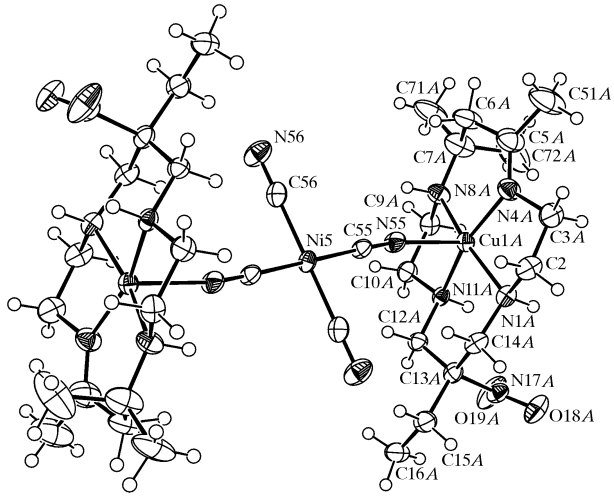


**Figure 2**
The $[(L^a)Cu–NC–Ni(CN)_2–CN–Cu(L^a)]^{2+}$ cation, drawn with displacement ellipsoids at the 50% probability level.

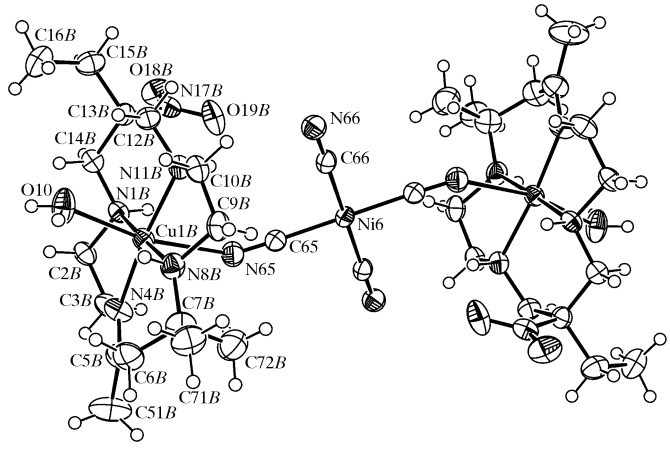


**Figure 3**
The $[(H_2O)(L^a)Cu–NC–Ni(CN)_2–CN–Cu(L^b)(OH_2)]^{2+}$ cation, drawn with displacement ellipsoids at the 50% probability level.

and for analogous cyanometallate compounds of other (tetraamine)copper(II) cations, including the *meso*-(5,5,-7,12,12,14-hexamethyl-1,4,8,11-tetraazacyclotetradecane)-copper(II), $[Cu(L^1)]^{2+}$, compounds with $[Fe(CN)_6]^{3-}$ (Zou *et al.*, 1998) and $[Cr(CN)_6]^{3-}$ (El Fallah *et al.*, 2001). The $[Ni(CN)_4]^{2-}$ compounds formed by $[Ni(L^1)]^{2+}$ (Gainsford & Curtis, 1984) and (3,10-diethyl-1,3,5,8,10,12-hexaazacyclotetradecane)nickel(II) (Kou *et al.*, 2000) have similar structures, but with the Ni—$N_{cyano}$ distances longer than Cu—$N_{cyano}$. The two faces of these (aza-macrocycle)metal(II) cations are equivalent, favouring the symmetrical structures observed. The two faces of the $[Cu(L)]^{2+}$ cation are inherently different, the configuration observed having the axial nitro and methyl groups on the same side, which minimizes the interaction with an axial substituent coordinated on the other side. For the $[Cu(L^a)]^{2+}$ ion, the isocyano N atom is coordinated on this less congested side, while for the $[Cu(L^b)]^{2+}$ ion, water is bound on this side and the isocyano group is bound more weakly on the other side.

## Experimental

Aqua(13-ethyl-5,7,7-trimethyl-13-nitro-1,4,8,11-tetraazacyclotetradec-4-ene)copper(II) bis(perchlorate), $[Cu(L)(H_2O)](ClO_4)_2$, was prepared by condensation of (4,6,6-trimethyl-3,7-diazanon-3-ene-1,9-diamine)copper(II) perchlorate (Blight & Curtis, 1962; Curtis, 1972; Curtis *et al.*, 2003), methanal and nitropropane in water, with $NaHCO_3$ as base. The mauve-coloured tetracyanonickelate(II) compound precipitated when aqueous solutions containing $[Ni(CN)_4]^{2-}$ and $[Cu(L)]^{2+}$ were mixed. The sparingly soluble compound was recrystallized by evaporation of an aqueous solution.

*Crystal data*

$[Cu_2Ni(CN)_4(C_{15}H_{31}N_5O_2)_2(H_2O)_2][Cu_2Ni(CN)_4(C_{15}H_{31}N_5O_2)_2][Ni(CN)_4]_2\cdot 8H_2O$
$M_r = 2339.23$
Triclinic, $P\overline{1}$
$a = 11.7497$ (2) Å
$b = 14.0540$ (3) Å
$c = 17.9014$ (4) Å
$\alpha = 70.154$ (1)°
$\beta = 78.165$ (1)°
$\gamma = 81.290$ (1)°
$V = 2710.6$ (1) Å$^3$
$Z = 1$
$D_x = 1.431$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 7862 reflections
$\theta = 2.3$–$29.3$°
$\mu = 1.52$ mm$^{-1}$
$T = 120$ (2) K
Plate, purple
0.35 × 0.35 × 0.08 mm

*Data collection*

Bruker SMART 1K CCD area detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.538$, $T_{max} = 0.885$
$R_{int} = 0.047$ before correction
22 241 measured reflections
14 372 independent reflections
9857 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 29.1$°
$h = -15 \rightarrow 15$
$k = -19 \rightarrow 19$
$l = -24 \rightarrow 23$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.162$
$S = 1.05$
14 372 reflections
666 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 5.1175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 1.41$ e Å$^{-3}$
$\Delta\rho_{min} = -0.73$ e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| Cu1*A*—N4*A* | 1.999 (4) | Cu1*B*—N8*B* | 2.018 (4) |
| Cu1*A*—N11*A* | 2.013 (3) | Cu1*B*—N1*B* | 2.020 (3) |
| Cu1*A*—N8*A* | 2.031 (3) | Cu1*B*—N11*B* | 2.026 (4) |
| Cu1*A*—N1*A* | 2.041 (3) | Cu1*B*—O10 | 2.396 (3) |
| Cu1*A*—N55 | 2.226 (3) | Cu1*B*—N65 | 2.677 (4) |
| N4*A*—C5*A* | 1.304 (6) | N4*B*—C5*B* | 1.304 (6) |
| Cu1*B*—N4*B* | 1.986 (4) | | |
| N4*A*—Cu1*A*—N11*A* | 164.6 (2) | N4*B*—Cu1*B*—N11*B* | 170.7 (2) |
| N4*A*—Cu1*A*—N8*A* | 95.6 (2) | N8*B*—Cu1*B*—N11*B* | 86.9 (2) |
| N11*A*—Cu1*A*—N8*A* | 85.9 (1) | N1*B*—Cu1*B*—N11*B* | 91.0 (2) |
| N4*A*—Cu1*A*—N1*A* | 85.4 (2) | N4*B*—Cu1*B*—O10 | 101.6 (2) |
| N11*A*—Cu1*A*—N1*A* | 89.7 (1) | N8*B*—Cu1*B*—O10 | 87.0 (2) |
| N8*A*—Cu1*A*—N1*A* | 166.8 (2) | N1*B*—Cu1*B*—O10 | 93.9 (1) |
| N4*A*—Cu1*A*—N55 | 99.9 (2) | N11*B*—Cu1*B*—O10 | 87.3 (2) |
| N11*A*—Cu1*A*—N55 | 95.1 (1) | N4*B*—Cu1*B*—N65 | 88.6 (2) |
| N8*A*—Cu1*A*—N55 | 96.7 (2) | N8*B*—Cu1*B*—N65 | 96.8 (2) |
| N1*A*—Cu1*A*—N55 | 96.1 (1) | N1*B*—Cu1*B*—N65 | 81.9 (1) |
| C55—N55—Cu1*A* | 168.2 (3) | N11*B*—Cu1*B*—N65 | 82.2 (1) |
| N4*B*—Cu1*B*—N8*B* | 95.9 (2) | O10—Cu1*B*—N65 | 168.7 (1) |
| N4*B*—Cu1*B*—N1*B* | 86.0 (2) | C65—Ni6—C66 | 89.1 (2) |
| N8*B*—Cu1*B*—N1*B* | 177.7 (2) | C65—N65—Cu1*B* | 137.7 (3) |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*A*—H1*A*···N85$^{i}$ | 0.91 | 2.26 | 3.124 (5) | 159 |
| N11*A*—H11*A*···O14 | 0.91 | 2.11 | 2.869 (5) | 140 |
| N11*A*—H11*A*···O19*A* | 0.91 | 2.41 | 2.978 (5) | 121 |
| N1*B*—H1*B*···O19*A* | 0.91 | 2.12 | 2.937 (5) | 149 |
| N8*B*—H8*B*···O12 | 0.91 | 2.13 | 3.041 (5) | 174 |
| N8*B*—H8*B*···O10 | 0.91 | 2.58 | 3.052 (5) | 113 |
| N11*B*—H11*B*···O19*B* | 0.91 | 2.29 | 2.874 (5) | 122 |
| O10—H10*E*···N75$^{ii}$ | 0.84 (5) | 1.92 (5) | 2.745 (6) | 168 (5) |
| O10—H10*F*···O12 | 0.85 (5) | 1.93 (5) | 2.702 (5) | 151 (5) |
| O11—H11*E*···N66$^{iii}$ | 0.84 (3) | 2.04 (4) | 2.870 (5) | 170 (6) |
| O11—H11*F*···N56 | 0.83 (5) | 2.28 (5) | 3.106 (5) | 172 (5) |
| O12—H12*F*···N86 | 0.83 (4) | 1.97 (4) | 2.788 (5) | 168 (6) |
| O12—H12*E*···O13 | 0.84 (4) | 1.88 (3) | 2.702 (5) | 171 (6) |
| O13—H13*E*···O11$^{iv}$ | 0.84 (4) | 1.94 (4) | 2.733 (5) | 158 (6) |
| O13—H13*F*···N56$^{iii}$ | 0.83 (4) | 2.15 (4) | 2.973 (6) | 170 (5) |
| O14—H14*E*···N76$^{ii}$ | 0.82 (4) | 2.03 (4) | 2.847 (5) | 174 (7) |
| O14—H14*F*···N85$^{i}$ | 0.82 (5) | 2.10 (5) | 2.872 (6) | 159 (6) |

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

C- and N-bound H atoms were placed in calculated positions and treated as riding. Water H atoms were located from difference syntheses, and their positions were refined with restrained O—H distances [0.82 (2) Å] and H—O—H angles [H···H = 1.35 (2) Å].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP*-3.2 (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1674). Services for accessing these data are described at the back of the journal.

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# supporting information

*Acta Cryst.* (2004). C**60**, m410–m413 [doi:10.1107/S0108270104015483]

# A compound of a novel tetraaza-macrocycle with trinuclear tetracyanonickelate-bridged cations

**Neil F. Curtis and Horst Puschmann**

## Computing details

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3.2* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

**di-$\mu$-cyano-1:2$\kappa^2C$:$N$;1:3$\kappa^2C$:$N$-tetracyano-1$\kappa^4N$-bis(13-ethyl-5,7,7- trimethyl-13-nitro-1,4,8,11-tetraazacyclotetradec-4-ene)- 2$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$;3$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$-dicopper(II)nickel(II) diaqua-1,3$\kappa^2O$-di-$\mu$-cyano-1:2$\kappa^2C$:$N$;1:3$\kappa^2C$:$N$-tetracyano-1$\kappa^4N$- bis(13-ethyl-5,7,7-trimethyl-13-nitro-1,4,8,11-tetraazacyclotetradec-4-ene)- 2$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$;3$\kappa^4N^1$,$N^4$,$N^8$,$N^{11}$-dicopper(II)nickel(II) bis[tetracyanonickelate(II)] octahydrate**

*Crystal data*

| | |
|---|---|
| $[Cu_2Ni(CN)_4(C_{15}H_{31}N_5O_2)_2(H_2O)_2]$ $[Cu_2Ni(CN)_4(C_{15}H_{31}N_5O_2)_2]$ $[Ni(CN)_4]_2\cdot 8H_2O$ | $V$ = 2710.6 (1) Å$^3$ |
| $M_r$ = 2339.23 | $Z$ = 1 |
| Triclinic, $P\bar{1}$ | $F(000)$ = 1220 |
| Hall symbol: -P 1 | $D_x$ = 1.431 Mg m$^{-3}$ |
| $a$ = 11.7497 (2) Å | Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å |
| $b$ = 14.0540 (3) Å | Cell parameters from 7862 reflections |
| $c$ = 17.9014 (4) Å | $\theta$ = 2.3–29.3° |
| $\alpha$ = 70.154 (1)° | $\mu$ = 1.52 mm$^{-1}$ |
| $\beta$ = 78.165 (1)° | $T$ = 120 K |
| $\gamma$ = 81.290 (1)° | Plate, purple |
| | 0.35 × 0.35 × 0.08 mm |

*Data collection*

| | |
|---|---|
| SMART 1K CCD area detector diffractometer | $T_{min}$ = 0.538, $T_{max}$ = 0.885 |
| Radiation source: fine-focus sealed tube | 22241 measured reflections |
| Graphite monochromator | 14372 independent reflections |
| Detector resolution: 8 pixels mm$^{-1}$ | 9857 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int}$ = 0.034 |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2001) R(int)=0.0470 before correction | $\theta_{max}$ = 29.1°, $\theta_{min}$ = 1.2° |
| | $h$ = −15→15 |
| | $k$ = −19→19 |
| | $l$ = −24→23 |

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.162$
$S = 1.05$
14372 reflections
666 parameters
15 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2 + 5.1175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 1.41$ e Å$^{-3}$
$\Delta\rho_{min} = -0.73$ e Å$^{-3}$

*Special details*

**Experimental**. The data collection nominally covered full sphere of reciprocal space, by a combination of 5 sets of $\omega$ scans; each set at different $\varphi$ and/or $2\theta$ angles and each scan (10 sec exposure) covering 0.3° in $\omega$. Crystal to detector distance 4.95 cm.

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cu1A | 0.51585 (4) | 0.65895 (3) | 0.23611 (3) | 0.02303 (11) |
| N1A | 0.3381 (3) | 0.6770 (2) | 0.2623 (2) | 0.0259 (7) |
| H1A | 0.3127 | 0.6170 | 0.2967 | 0.031* |
| N4A | 0.5137 (3) | 0.7137 (3) | 0.3259 (2) | 0.0355 (8) |
| N8A | 0.6877 (3) | 0.6078 (3) | 0.2270 (2) | 0.0316 (8) |
| H8A | 0.7206 | 0.6564 | 0.1829 | 0.038* |
| N11A | 0.5031 (3) | 0.5711 (2) | 0.17031 (19) | 0.0232 (6) |
| H11A | 0.4750 | 0.5125 | 0.2054 | 0.028* |
| N17A | 0.2472 (3) | 0.5344 (3) | 0.1980 (2) | 0.0303 (7) |
| C2A | 0.3118 (4) | 0.7529 (3) | 0.3059 (3) | 0.0360 (10) |
| H2A | 0.2310 | 0.7525 | 0.3324 | 0.043* |
| H2B | 0.3238 | 0.8204 | 0.2682 | 0.043* |
| C3A | 0.3928 (4) | 0.7256 (4) | 0.3681 (3) | 0.0400 (11) |
| H3A | 0.3850 | 0.7788 | 0.3923 | 0.048* |
| H3B | 0.3732 | 0.6627 | 0.4103 | 0.048* |
| C5A | 0.6043 (5) | 0.7166 (4) | 0.3564 (3) | 0.0481 (12) |
| C6A | 0.7245 (4) | 0.7033 (4) | 0.3093 (3) | 0.0468 (12) |
| H6A | 0.7818 | 0.7063 | 0.3404 | 0.056* |
| H6B | 0.7331 | 0.7602 | 0.2599 | 0.056* |
| C7A | 0.7534 (5) | 0.6051 (5) | 0.2876 (4) | 0.0521 (14) |
| C9A | 0.7008 (4) | 0.5189 (3) | 0.1980 (3) | 0.0316 (9) |

| | | | | |
|---|---|---|---|---|
| H9A | 0.6769 | 0.4591 | 0.2422 | 0.038* |
| H9B | 0.7817 | 0.5055 | 0.1755 | 0.038* |
| C10A | 0.6247 (3) | 0.5438 (3) | 0.1343 (3) | 0.0313 (9) |
| H10A | 0.6526 | 0.6003 | 0.0883 | 0.038* |
| H10B | 0.6266 | 0.4856 | 0.1164 | 0.038* |
| C12A | 0.4256 (3) | 0.6120 (3) | 0.1093 (2) | 0.0272 (8) |
| H12A | 0.4295 | 0.5636 | 0.0808 | 0.033* |
| H12B | 0.4544 | 0.6741 | 0.0704 | 0.033* |
| C13A | 0.2975 (3) | 0.6348 (3) | 0.1435 (2) | 0.0272 (8) |
| C14A | 0.2764 (3) | 0.7086 (3) | 0.1926 (3) | 0.0293 (9) |
| H14A | 0.3002 | 0.7743 | 0.1571 | 0.035* |
| H14B | 0.1933 | 0.7172 | 0.2117 | 0.035* |
| C51A | 0.5990 (6) | 0.7505 (6) | 0.4274 (4) | 0.074 (2) |
| H51A | 0.5207 | 0.7493 | 0.4566 | 0.111* |
| H51B | 0.6222 | 0.8183 | 0.4096 | 0.111* |
| H51C | 0.6508 | 0.7057 | 0.4619 | 0.111* |
| C71A | 0.8848 (5) | 0.5850 (6) | 0.2652 (4) | 0.0654 (18) |
| H71A | 0.9014 | 0.5263 | 0.2474 | 0.098* |
| H71B | 0.9211 | 0.5730 | 0.3114 | 0.098* |
| H71C | 0.9148 | 0.6428 | 0.2227 | 0.098* |
| C72A | 0.7108 (8) | 0.5134 (6) | 0.3658 (4) | 0.090 (3) |
| H72A | 0.6275 | 0.5225 | 0.3805 | 0.136* |
| H72B | 0.7473 | 0.5122 | 0.4095 | 0.136* |
| H72C | 0.7319 | 0.4504 | 0.3545 | 0.136* |
| C15A | 0.2225 (4) | 0.6784 (3) | 0.0753 (3) | 0.0347 (9) |
| H15A | 0.1409 | 0.6809 | 0.0998 | 0.042* |
| H15B | 0.2407 | 0.7477 | 0.0457 | 0.042* |
| C16A | 0.2383 (4) | 0.6202 (4) | 0.0155 (3) | 0.0398 (11) |
| H16A | 0.3143 | 0.6283 | −0.0169 | 0.060* |
| H16B | 0.1798 | 0.6460 | −0.0185 | 0.060* |
| H16C | 0.2308 | 0.5494 | 0.0442 | 0.060* |
| O18A | 0.1574 (3) | 0.5370 (3) | 0.2445 (2) | 0.0469 (9) |
| O19A | 0.3001 (3) | 0.4555 (2) | 0.1910 (3) | 0.0598 (11) |
| Ni5 | 0.5000 | 1.0000 | 0.0000 | 0.02336 (15) |
| N55 | 0.5351 (3) | 0.8009 (2) | 0.1318 (2) | 0.0311 (8) |
| N56 | 0.7477 (3) | 1.0469 (3) | −0.0123 (3) | 0.0437 (10) |
| C55 | 0.5245 (3) | 0.8761 (3) | 0.0810 (2) | 0.0258 (8) |
| C56 | 0.6528 (4) | 1.0292 (3) | −0.0074 (2) | 0.0297 (9) |
| Cu1B | 0.09397 (4) | 0.18678 (4) | 0.25244 (3) | 0.02937 (13) |
| N1B | 0.2299 (3) | 0.2477 (3) | 0.2668 (2) | 0.0289 (7) |
| H1B | 0.2285 | 0.3131 | 0.2334 | 0.035* |
| N4B | 0.0107 (4) | 0.2139 (3) | 0.3526 (2) | 0.0388 (9) |
| N8B | −0.0399 (3) | 0.1284 (3) | 0.2332 (2) | 0.0373 (8) |
| H8B | −0.0212 | 0.0602 | 0.2525 | 0.045* |
| N11B | 0.1758 (3) | 0.1839 (3) | 0.1419 (2) | 0.0300 (7) |
| H11B | 0.1701 | 0.2487 | 0.1077 | 0.036* |
| C2B | 0.2048 (4) | 0.2517 (3) | 0.3513 (3) | 0.0341 (9) |
| H2C | 0.2504 | 0.3009 | 0.3559 | 0.041* |

| | | | | |
|---|---|---|---|---|
| H2D | 0.2258 | 0.1858 | 0.3884 | 0.041* |
| C3B | 0.0763 (4) | 0.2817 (4) | 0.3717 (3) | 0.0417 (11) |
| H3C | 0.0558 | 0.2752 | 0.4285 | 0.050* |
| H3D | 0.0575 | 0.3518 | 0.3406 | 0.050* |
| C5B | −0.0948 (4) | 0.1919 (4) | 0.3884 (3) | 0.0422 (11) |
| C6B | −0.1509 (5) | 0.1167 (4) | 0.3657 (3) | 0.0489 (13) |
| H6C | −0.2295 | 0.1106 | 0.3960 | 0.059* |
| H6D | −0.1077 | 0.0508 | 0.3834 | 0.059* |
| C7B | −0.1583 (4) | 0.1412 (4) | 0.2763 (3) | 0.0456 (12) |
| C9B | −0.0222 (4) | 0.1486 (4) | 0.1444 (3) | 0.0387 (10) |
| H9C | −0.0692 | 0.1068 | 0.1318 | 0.046* |
| H9D | −0.0449 | 0.2193 | 0.1173 | 0.046* |
| C10B | 0.1040 (4) | 0.1237 (3) | 0.1175 (3) | 0.0339 (9) |
| H10C | 0.1196 | 0.1394 | 0.0595 | 0.041* |
| H10D | 0.1247 | 0.0517 | 0.1417 | 0.041* |
| C12B | 0.2997 (4) | 0.1465 (3) | 0.1334 (3) | 0.0336 (9) |
| H12C | 0.3280 | 0.1557 | 0.0767 | 0.040* |
| H12D | 0.3051 | 0.0740 | 0.1615 | 0.040* |
| C13B | 0.3825 (4) | 0.1929 (3) | 0.1630 (3) | 0.0313 (9) |
| C14B | 0.3471 (4) | 0.1995 (3) | 0.2486 (3) | 0.0316 (9) |
| H14C | 0.3524 | 0.1312 | 0.2864 | 0.038* |
| H14D | 0.4032 | 0.2368 | 0.2576 | 0.038* |
| C15B | 0.5068 (4) | 0.1368 (4) | 0.1545 (3) | 0.0447 (11) |
| H15F | 0.5320 | 0.1397 | 0.0987 | 0.054* |
| H15G | 0.5603 | 0.1725 | 0.1677 | 0.054* |
| C16B | 0.5147 (5) | 0.0253 (4) | 0.2079 (4) | 0.0500 (13) |
| H16D | 0.4573 | −0.0094 | 0.1986 | 0.075* |
| H16E | 0.5005 | 0.0220 | 0.2635 | 0.075* |
| H16F | 0.5912 | −0.0063 | 0.1951 | 0.075* |
| C51B | −0.1626 (5) | 0.2302 (5) | 0.4549 (4) | 0.0696 (19) |
| H51D | −0.1264 | 0.2860 | 0.4566 | 0.104* |
| H51E | −0.1637 | 0.1766 | 0.5054 | 0.104* |
| H51F | −0.2412 | 0.2524 | 0.4452 | 0.104* |
| C71B | −0.2370 (5) | 0.0675 (4) | 0.2707 (4) | 0.0538 (14) |
| H71D | −0.2042 | −0.0010 | 0.2926 | 0.081* |
| H71E | −0.2430 | 0.0806 | 0.2154 | 0.081* |
| H71F | −0.3132 | 0.0765 | 0.3008 | 0.081* |
| C72B | −0.2114 (5) | 0.2517 (4) | 0.2413 (4) | 0.0613 (15) |
| H72D | −0.2231 | 0.2635 | 0.1873 | 0.092* |
| H72E | −0.1590 | 0.2981 | 0.2411 | 0.092* |
| H72F | −0.2849 | 0.2621 | 0.2739 | 0.092* |
| N17B | 0.3986 (3) | 0.3035 (3) | 0.1073 (2) | 0.0364 (8) |
| O19B | 0.3262 (4) | 0.3450 (3) | 0.0628 (2) | 0.0553 (10) |
| O18B | 0.4805 (3) | 0.3460 (3) | 0.1104 (2) | 0.0518 (9) |
| O10 | 0.1759 (3) | 0.0153 (3) | 0.3044 (2) | 0.0500 (9) |
| H10E | 0.215 (4) | −0.006 (4) | 0.342 (3) | 0.060* |
| H10F | 0.127 (4) | −0.029 (3) | 0.317 (3) | 0.060* |
| Ni6 | 0.0000 | 0.5000 | 0.0000 | 0.02634 (16) |

| | | | | |
|---|---|---|---|---|
| N65 | 0.0374 (4) | 0.3807 (3) | 0.1702 (2) | 0.0395 (9) |
| N66 | 0.1233 (3) | 0.3209 (3) | −0.0505 (2) | 0.0380 (9) |
| C65 | 0.0219 (4) | 0.4272 (3) | 0.1060 (3) | 0.0296 (8) |
| C66 | 0.0769 (4) | 0.3893 (3) | −0.0320 (3) | 0.0300 (9) |
| Ni7 | 1.5000 | 0.0000 | 0.5000 | 0.02809 (17) |
| N75 | 1.3252 (5) | −0.0362 (4) | 0.4144 (3) | 0.0727 (17) |
| N76 | 1.5201 (4) | 0.2129 (3) | 0.3831 (2) | 0.0404 (9) |
| C75 | 1.3917 (5) | −0.0218 (3) | 0.4474 (3) | 0.0455 (12) |
| C76 | 1.5120 (4) | 0.1317 (3) | 0.4271 (3) | 0.0335 (9) |
| Ni8 | 0.0000 | −0.5000 | 0.5000 | 0.03084 (18) |
| N85 | 0.2488 (4) | −0.5019 (3) | 0.4135 (3) | 0.0525 (11) |
| N86 | −0.0354 (4) | −0.2762 (3) | 0.4102 (3) | 0.0512 (11) |
| C85 | 0.1545 (4) | −0.5016 (4) | 0.4466 (3) | 0.0377 (10) |
| C86 | −0.0221 (4) | −0.3605 (4) | 0.4439 (3) | 0.0381 (10) |
| O11 | 0.8374 (3) | 0.8482 (3) | 0.1108 (2) | 0.0472 (8) |
| H11E | 0.841 (5) | 0.797 (2) | 0.096 (3) | 0.057* |
| H11F | 0.808 (5) | 0.899 (3) | 0.078 (3) | 0.057* |
| O12 | 0.0259 (4) | −0.0996 (3) | 0.2857 (2) | 0.0486 (9) |
| H12E | 0.025 (5) | −0.108 (4) | 0.2417 (17) | 0.058* |
| H12F | −0.001 (5) | −0.147 (3) | 0.325 (2) | 0.058* |
| O13 | 0.0452 (3) | −0.1146 (3) | 0.1370 (2) | 0.0549 (10) |
| H13E | −0.015 (3) | −0.113 (5) | 0.118 (3) | 0.066* |
| H13F | 0.098 (3) | −0.096 (5) | 0.098 (2) | 0.066* |
| O14 | 0.4609 (4) | 0.4265 (3) | 0.3287 (3) | 0.0643 (12) |
| H14E | 0.482 (5) | 0.3659 (19) | 0.345 (4) | 0.077* |
| H14F | 0.395 (3) | 0.433 (4) | 0.353 (4) | 0.077* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cu1A | 0.0241 (2) | 0.0199 (2) | 0.0228 (2) | 0.00072 (17) | −0.00397 (18) | −0.00506 (17) |
| N1A | 0.0251 (16) | 0.0198 (15) | 0.0314 (17) | −0.0009 (12) | −0.0009 (13) | −0.0089 (13) |
| N4A | 0.034 (2) | 0.044 (2) | 0.0325 (19) | −0.0004 (16) | −0.0062 (16) | −0.0184 (17) |
| N8A | 0.0308 (18) | 0.0321 (18) | 0.0337 (19) | 0.0069 (14) | −0.0137 (15) | −0.0121 (15) |
| N11A | 0.0218 (15) | 0.0192 (15) | 0.0245 (16) | −0.0018 (12) | −0.0025 (12) | −0.0024 (12) |
| N17A | 0.0271 (18) | 0.0245 (17) | 0.0378 (19) | −0.0071 (14) | −0.0061 (15) | −0.0055 (14) |
| C2A | 0.029 (2) | 0.034 (2) | 0.050 (3) | −0.0006 (17) | −0.0005 (19) | −0.024 (2) |
| C3A | 0.036 (2) | 0.048 (3) | 0.043 (3) | −0.005 (2) | 0.003 (2) | −0.030 (2) |
| C5A | 0.046 (3) | 0.062 (3) | 0.048 (3) | 0.001 (2) | −0.010 (2) | −0.034 (3) |
| C6A | 0.037 (3) | 0.069 (3) | 0.050 (3) | 0.005 (2) | −0.018 (2) | −0.037 (3) |
| C7A | 0.041 (3) | 0.069 (4) | 0.059 (3) | 0.018 (3) | −0.019 (2) | −0.040 (3) |
| C9A | 0.028 (2) | 0.030 (2) | 0.032 (2) | 0.0047 (16) | −0.0019 (17) | −0.0081 (17) |
| C10A | 0.024 (2) | 0.037 (2) | 0.032 (2) | 0.0014 (17) | −0.0007 (16) | −0.0148 (18) |
| C12A | 0.029 (2) | 0.0234 (19) | 0.029 (2) | −0.0060 (15) | −0.0076 (16) | −0.0044 (15) |
| C13A | 0.028 (2) | 0.0204 (18) | 0.030 (2) | −0.0044 (15) | −0.0038 (16) | −0.0032 (15) |
| C14A | 0.0238 (19) | 0.0200 (18) | 0.042 (2) | −0.0008 (15) | −0.0061 (17) | −0.0075 (17) |
| C51A | 0.062 (4) | 0.117 (6) | 0.072 (4) | 0.016 (4) | −0.024 (3) | −0.070 (4) |
| C71A | 0.040 (3) | 0.112 (5) | 0.064 (4) | 0.029 (3) | −0.030 (3) | −0.056 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C72A | 0.129 (7) | 0.085 (5) | 0.054 (4) | 0.035 (5) | −0.038 (4) | −0.023 (4) |
| C15A | 0.033 (2) | 0.031 (2) | 0.038 (2) | −0.0042 (17) | −0.0109 (18) | −0.0038 (18) |
| C16A | 0.037 (2) | 0.045 (3) | 0.042 (3) | −0.008 (2) | −0.014 (2) | −0.013 (2) |
| O18A | 0.0382 (18) | 0.0399 (18) | 0.059 (2) | −0.0161 (15) | 0.0117 (16) | −0.0170 (16) |
| O19A | 0.045 (2) | 0.0256 (17) | 0.095 (3) | −0.0104 (15) | 0.013 (2) | −0.0122 (18) |
| Ni5 | 0.0273 (4) | 0.0142 (3) | 0.0243 (3) | −0.0034 (3) | −0.0017 (3) | −0.0014 (3) |
| N55 | 0.0341 (19) | 0.0213 (16) | 0.0354 (19) | −0.0087 (14) | −0.0093 (15) | −0.0012 (14) |
| N56 | 0.035 (2) | 0.0290 (19) | 0.061 (3) | −0.0063 (16) | −0.0079 (19) | −0.0048 (18) |
| C55 | 0.0242 (19) | 0.0217 (18) | 0.031 (2) | −0.0058 (14) | −0.0044 (16) | −0.0058 (16) |
| C56 | 0.038 (2) | 0.0155 (18) | 0.030 (2) | −0.0008 (16) | −0.0045 (17) | −0.0006 (15) |
| Cu1B | 0.0320 (3) | 0.0313 (3) | 0.0269 (3) | −0.0058 (2) | −0.0053 (2) | −0.0105 (2) |
| N1B | 0.0351 (19) | 0.0270 (17) | 0.0238 (17) | −0.0070 (14) | −0.0025 (14) | −0.0065 (14) |
| N4B | 0.044 (2) | 0.042 (2) | 0.034 (2) | −0.0169 (18) | 0.0029 (17) | −0.0170 (17) |
| N8B | 0.034 (2) | 0.041 (2) | 0.039 (2) | −0.0061 (16) | −0.0104 (16) | −0.0108 (17) |
| N11B | 0.0363 (19) | 0.0259 (17) | 0.0303 (18) | −0.0030 (14) | −0.0091 (15) | −0.0103 (14) |
| C2B | 0.045 (3) | 0.034 (2) | 0.026 (2) | −0.0075 (19) | −0.0052 (18) | −0.0121 (17) |
| C3B | 0.049 (3) | 0.047 (3) | 0.034 (2) | −0.019 (2) | 0.001 (2) | −0.018 (2) |
| C5B | 0.040 (3) | 0.047 (3) | 0.040 (3) | −0.015 (2) | 0.001 (2) | −0.014 (2) |
| C6B | 0.043 (3) | 0.050 (3) | 0.050 (3) | −0.021 (2) | 0.001 (2) | −0.009 (2) |
| C7B | 0.041 (3) | 0.046 (3) | 0.053 (3) | −0.014 (2) | −0.004 (2) | −0.019 (2) |
| C9B | 0.044 (3) | 0.038 (2) | 0.039 (2) | −0.005 (2) | −0.018 (2) | −0.012 (2) |
| C10B | 0.046 (3) | 0.030 (2) | 0.031 (2) | −0.0064 (18) | −0.0110 (19) | −0.0116 (17) |
| C12B | 0.034 (2) | 0.032 (2) | 0.037 (2) | −0.0022 (17) | −0.0046 (18) | −0.0150 (18) |
| C13B | 0.034 (2) | 0.027 (2) | 0.031 (2) | −0.0003 (17) | −0.0060 (17) | −0.0075 (17) |
| C14B | 0.035 (2) | 0.030 (2) | 0.031 (2) | −0.0065 (17) | −0.0058 (18) | −0.0102 (17) |
| C15B | 0.035 (2) | 0.050 (3) | 0.055 (3) | −0.004 (2) | −0.006 (2) | −0.027 (2) |
| C16B | 0.047 (3) | 0.043 (3) | 0.068 (4) | 0.004 (2) | −0.025 (3) | −0.022 (3) |
| C51B | 0.053 (4) | 0.087 (5) | 0.078 (4) | −0.030 (3) | 0.023 (3) | −0.047 (4) |
| C71B | 0.040 (3) | 0.061 (3) | 0.069 (4) | −0.013 (2) | −0.008 (3) | −0.027 (3) |
| C72B | 0.054 (3) | 0.056 (4) | 0.067 (4) | 0.004 (3) | −0.010 (3) | −0.014 (3) |
| N17B | 0.038 (2) | 0.038 (2) | 0.0310 (19) | −0.0087 (17) | 0.0029 (16) | −0.0114 (16) |
| O19B | 0.080 (3) | 0.048 (2) | 0.0366 (19) | −0.0211 (19) | −0.0215 (19) | 0.0027 (16) |
| O18B | 0.0379 (19) | 0.044 (2) | 0.071 (3) | −0.0199 (16) | 0.0027 (17) | −0.0153 (18) |
| O10 | 0.057 (2) | 0.0339 (18) | 0.058 (2) | −0.0118 (16) | −0.0290 (19) | 0.0016 (16) |
| Ni6 | 0.0239 (4) | 0.0255 (4) | 0.0292 (4) | −0.0016 (3) | −0.0070 (3) | −0.0069 (3) |
| N65 | 0.045 (2) | 0.037 (2) | 0.036 (2) | 0.0007 (17) | −0.0105 (18) | −0.0109 (17) |
| N66 | 0.041 (2) | 0.033 (2) | 0.039 (2) | −0.0010 (17) | −0.0054 (17) | −0.0119 (17) |
| C65 | 0.030 (2) | 0.028 (2) | 0.031 (2) | −0.0019 (16) | −0.0059 (17) | −0.0112 (17) |
| C66 | 0.028 (2) | 0.029 (2) | 0.032 (2) | −0.0040 (16) | −0.0084 (17) | −0.0055 (17) |
| Ni7 | 0.0347 (4) | 0.0229 (4) | 0.0236 (4) | −0.0003 (3) | −0.0062 (3) | −0.0037 (3) |
| N75 | 0.103 (4) | 0.044 (3) | 0.070 (3) | −0.027 (3) | −0.055 (3) | 0.015 (2) |
| N76 | 0.051 (2) | 0.032 (2) | 0.034 (2) | −0.0030 (17) | −0.0105 (18) | −0.0021 (16) |
| C75 | 0.067 (3) | 0.027 (2) | 0.040 (3) | −0.010 (2) | −0.024 (2) | 0.0038 (19) |
| C76 | 0.036 (2) | 0.035 (2) | 0.030 (2) | 0.0003 (18) | −0.0120 (18) | −0.0080 (18) |
| Ni8 | 0.0329 (4) | 0.0360 (4) | 0.0239 (4) | −0.0116 (3) | −0.0039 (3) | −0.0067 (3) |
| N85 | 0.041 (2) | 0.052 (3) | 0.051 (3) | −0.008 (2) | 0.007 (2) | −0.006 (2) |
| N86 | 0.069 (3) | 0.042 (2) | 0.041 (2) | −0.014 (2) | −0.013 (2) | −0.005 (2) |
| C85 | 0.041 (3) | 0.038 (2) | 0.031 (2) | −0.007 (2) | −0.007 (2) | −0.0045 (19) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C86 | 0.041 (3) | 0.045 (3) | 0.031 (2) | −0.012 (2) | −0.0067 (19) | −0.012 (2) |
| O11 | 0.045 (2) | 0.047 (2) | 0.054 (2) | 0.0014 (17) | −0.0155 (17) | −0.0196 (18) |
| O12 | 0.064 (2) | 0.0332 (18) | 0.049 (2) | −0.0175 (16) | −0.0165 (19) | −0.0036 (16) |
| O13 | 0.044 (2) | 0.076 (3) | 0.052 (2) | −0.021 (2) | −0.0031 (17) | −0.025 (2) |
| O14 | 0.068 (3) | 0.0320 (19) | 0.056 (2) | 0.0013 (18) | 0.021 (2) | 0.0120 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cu1A—N4A | 1.999 (4) | N8B—C7B | 1.464 (6) |
| Cu1A—N11A | 2.013 (3) | N8B—C9B | 1.492 (6) |
| Cu1A—N8A | 2.031 (3) | N8B—H8B | 0.9100 |
| Cu1A—N1A | 2.041 (3) | N11B—C12B | 1.467 (5) |
| Cu1A—N55 | 2.226 (3) | N11B—C10B | 1.494 (5) |
| N1A—C14A | 1.476 (5) | N11B—H11B | 0.9100 |
| N1A—C2A | 1.486 (5) | C2B—C3B | 1.509 (7) |
| N1A—H1A | 0.9100 | C2B—H2C | 0.9700 |
| N4A—C5A | 1.304 (6) | C2B—H2D | 0.9700 |
| N4A—C3A | 1.480 (6) | C3B—H3C | 0.9700 |
| N8A—C7A | 1.443 (6) | C3B—H3D | 0.9700 |
| N8A—C9A | 1.484 (5) | C5B—C51B | 1.503 (7) |
| N8A—H8A | 0.9100 | C5B—C6B | 1.525 (7) |
| N11A—C12A | 1.476 (5) | C6B—C7B | 1.538 (7) |
| N11A—C10A | 1.492 (5) | C6B—H6C | 0.9700 |
| N11A—H11A | 0.9100 | C6B—H6D | 0.9700 |
| N17A—O18A | 1.208 (5) | C7B—C71B | 1.528 (7) |
| N17A—O19A | 1.218 (5) | C7B—C72B | 1.550 (8) |
| N17A—C13A | 1.541 (5) | C9B—C10B | 1.489 (6) |
| C2A—C3A | 1.526 (7) | C9B—H9C | 0.9700 |
| C2A—H2A | 0.9700 | C9B—H9D | 0.9700 |
| C2A—H2B | 0.9700 | C10B—H10C | 0.9700 |
| C3A—H3A | 0.9700 | C10B—H10D | 0.9700 |
| C3A—H3B | 0.9700 | C12B—C13B | 1.514 (6) |
| C5A—C51A | 1.489 (7) | C12B—H12C | 0.9700 |
| C5A—C6A | 1.510 (7) | C12B—H12D | 0.9700 |
| C6A—C7A | 1.529 (7) | C13B—C14B | 1.534 (6) |
| C6A—H6A | 0.9700 | C13B—N17B | 1.550 (5) |
| C6A—H6B | 0.9700 | C13B—C15B | 1.555 (6) |
| C7A—C71A | 1.522 (7) | C14B—H14C | 0.9700 |
| C7A—C72A | 1.598 (10) | C14B—H14D | 0.9700 |
| C9A—C10A | 1.508 (6) | C15B—C16B | 1.534 (7) |
| C9A—H9A | 0.9700 | C15B—H15F | 0.9700 |
| C9A—H9B | 0.9700 | C15B—H15G | 0.9700 |
| C10A—H10A | 0.9700 | C16B—H16D | 0.9600 |
| C10A—H10B | 0.9700 | C16B—H16E | 0.9600 |
| C12A—C13A | 1.534 (6) | C16B—H16F | 0.9600 |
| C12A—H12A | 0.9700 | C51B—H51D | 0.9600 |
| C12A—H12B | 0.9700 | C51B—H51E | 0.9600 |
| C13A—C14A | 1.537 (6) | C51B—H51F | 0.9600 |

| | | | |
|---|---|---|---|
| C13A—C15A | 1.553 (6) | C71B—H71D | 0.9600 |
| C14A—H14A | 0.9700 | C71B—H71E | 0.9600 |
| C14A—H14B | 0.9700 | C71B—H71F | 0.9600 |
| C51A—H51A | 0.9600 | C72B—H72D | 0.9600 |
| C51A—H51B | 0.9600 | C72B—H72E | 0.9600 |
| C51A—H51C | 0.9600 | C72B—H72F | 0.9600 |
| C71A—H71A | 0.9600 | N17B—O18B | 1.227 (5) |
| C71A—H71B | 0.9600 | N17B—O19B | 1.229 (5) |
| C71A—H71C | 0.9600 | O10—H10E | 0.839 (19) |
| C72A—H72A | 0.9600 | O10—H10F | 0.853 (19) |
| C72A—H72B | 0.9600 | Ni6—C65$^{ii}$ | 1.876 (4) |
| C72A—H72C | 0.9600 | Ni6—C65 | 1.876 (4) |
| C15A—C16A | 1.522 (7) | Ni6—C66$^{ii}$ | 1.879 (4) |
| C15A—H15A | 0.9700 | Ni6—C66 | 1.879 (4) |
| C15A—H15B | 0.9700 | N65—C65 | 1.149 (5) |
| C16A—H16A | 0.9600 | N66—C66 | 1.147 (5) |
| C16A—H16B | 0.9600 | Ni7—C75 | 1.846 (5) |
| C16A—H16C | 0.9600 | Ni7—C75$^{iii}$ | 1.846 (5) |
| Ni5—C56 | 1.871 (4) | Ni7—C76$^{iii}$ | 1.874 (4) |
| Ni5—C56$^{i}$ | 1.871 (4) | Ni7—C76 | 1.874 (4) |
| Ni5—C55 | 1.877 (4) | N75—C75 | 1.148 (7) |
| Ni5—C55$^{i}$ | 1.877 (4) | N76—C76 | 1.150 (5) |
| N55—C55 | 1.146 (5) | Ni8—C85$^{iv}$ | 1.870 (5) |
| N56—C56 | 1.159 (6) | Ni8—C85 | 1.871 (5) |
| Cu1B—N4B | 1.986 (4) | Ni8—C86$^{iv}$ | 1.880 (5) |
| Cu1B—N8B | 2.018 (4) | Ni8—C86 | 1.880 (5) |
| Cu1B—N1B | 2.020 (3) | N85—C85 | 1.145 (6) |
| Cu1B—N11B | 2.026 (4) | N86—C86 | 1.136 (6) |
| Cu1B—O10 | 2.396 (3) | O11—H11F | 0.832 (19) |
| Cu1B—N65 | 2.677 (4) | O12—H12E | 0.835 (19) |
| N1B—C14B | 1.465 (5) | O12—H12F | 0.832 (19) |
| N1B—C2B | 1.500 (5) | O13—H13E | 0.837 (19) |
| N1B—H1B | 0.9100 | O13—H13F | 0.831 (19) |
| N4B—C5B | 1.304 (6) | O14—H14E | 0.818 (19) |
| N4B—C3B | 1.474 (6) | O14—H14F | 0.815 (19) |

| | | | |
|---|---|---|---|
| N4A—Cu1A—N11A | 164.6 (2) | O10—Cu1B—N65 | 168.7 (1) |
| N4A—Cu1A—N8A | 95.6 (2) | C14B—N1B—C2B | 111.4 (3) |
| N11A—Cu1A—N8A | 85.9 (1) | C14B—N1B—Cu1B | 117.4 (3) |
| N4A—Cu1A—N1A | 85.4 (2) | C2B—N1B—Cu1B | 106.6 (3) |
| N11A—Cu1A—N1A | 89.7 (1) | C14B—N1B—H1B | 107.0 |
| N8A—Cu1A—N1A | 166.8 (2) | C2B—N1B—H1B | 107.0 |
| N4A—Cu1A—N55 | 99.9 (2) | Cu1B—N1B—H1B | 107.0 |
| N11A—Cu1A—N55 | 95.1 (1) | C5B—N4B—C3B | 123.4 (4) |
| N8A—Cu1A—N55 | 96.7 (2) | C5B—N4B—Cu1B | 126.2 (3) |
| N1A—Cu1A—N55 | 96.1 (1) | C3B—N4B—Cu1B | 109.3 (3) |
| C14A—N1A—C2A | 110.9 (3) | C7B—N8B—C9B | 118.9 (4) |
| C14A—N1A—Cu1A | 115.6 (2) | C7B—N8B—Cu1B | 120.9 (3) |

| | | | |
|---|---|---|---|
| C2A—N1A—Cu1A | 105.4 (2) | C9B—N8B—Cu1B | 105.4 (3) |
| C14A—N1A—H1A | 108.2 | C7B—N8B—H8B | 102.9 |
| C2A—N1A—H1A | 108.2 | C9B—N8B—H8B | 102.9 |
| Cu1A—N1A—H1A | 108.2 | Cu1B—N8B—H8B | 102.9 |
| C5A—N4A—C3A | 122.2 (4) | C12B—N11B—C10B | 112.0 (3) |
| C5A—N4A—Cu1A | 125.7 (3) | C12B—N11B—Cu1B | 117.5 (3) |
| C3A—N4A—Cu1A | 110.3 (3) | C10B—N11B—Cu1B | 105.3 (3) |
| C7A—N8A—C9A | 119.2 (4) | C12B—N11B—H11B | 107.2 |
| C7A—N8A—Cu1A | 120.2 (3) | C10B—N11B—H11B | 107.2 |
| C9A—N8A—Cu1A | 106.8 (3) | Cu1B—N11B—H11B | 107.2 |
| C7A—N8A—H8A | 102.5 | N1B—C2B—C3B | 108.6 (4) |
| C9A—N8A—H8A | 102.5 | N1B—C2B—H2C | 110.0 |
| Cu1A—N8A—H8A | 102.5 | C3B—C2B—H2C | 110.0 |
| C12A—N11A—C10A | 112.0 (3) | N1B—C2B—H2D | 110.0 |
| C12A—N11A—Cu1A | 117.1 (2) | C3B—C2B—H2D | 110.0 |
| C10A—N11A—Cu1A | 106.6 (2) | H2C—C2B—H2D | 108.4 |
| C12A—N11A—H11A | 106.9 | N4B—C3B—C2B | 108.0 (4) |
| C10A—N11A—H11A | 106.9 | N4B—C3B—H3C | 110.1 |
| Cu1A—N11A—H11A | 106.9 | C2B—C3B—H3C | 110.1 |
| O18A—N17A—O19A | 123.1 (4) | N4B—C3B—H3D | 110.1 |
| O18A—N17A—C13A | 119.2 (3) | C2B—C3B—H3D | 110.1 |
| O19A—N17A—C13A | 117.8 (3) | H3C—C3B—H3D | 108.4 |
| N1A—C2A—C3A | 108.5 (3) | N4B—C5B—C51B | 124.4 (5) |
| N1A—C2A—H2A | 110.0 | N4B—C5B—C6B | 118.3 (4) |
| C3A—C2A—H2A | 110.0 | C51B—C5B—C6B | 117.2 (4) |
| N1A—C2A—H2B | 110.0 | C5B—C6B—C7B | 117.2 (4) |
| C3A—C2A—H2B | 110.0 | C5B—C6B—H6C | 108.0 |
| H2A—C2A—H2B | 108.4 | C7B—C6B—H6C | 108.0 |
| N4A—C3A—C2A | 107.6 (4) | C5B—C6B—H6D | 108.0 |
| N4A—C3A—H3A | 110.2 | C7B—C6B—H6D | 108.0 |
| C2A—C3A—H3A | 110.2 | H6C—C6B—H6D | 107.2 |
| N4A—C3A—H3B | 110.2 | N8B—C7B—C71B | 111.5 (4) |
| C2A—C3A—H3B | 110.2 | N8B—C7B—C6B | 107.9 (4) |
| H3A—C3A—H3B | 108.5 | C71B—C7B—C6B | 107.5 (4) |
| N4A—C5A—C51A | 124.5 (5) | N8B—C7B—C72B | 109.8 (4) |
| N4A—C5A—C6A | 118.6 (4) | C71B—C7B—C72B | 109.5 (5) |
| C51A—C5A—C6A | 116.0 (5) | C6B—C7B—C72B | 110.5 (5) |
| C5A—C6A—C7A | 115.9 (5) | C10B—C9B—N8B | 107.4 (4) |
| C5A—C6A—H6A | 108.3 | C10B—C9B—H9C | 110.2 |
| C7A—C6A—H6A | 108.3 | N8B—C9B—H9C | 110.2 |
| C5A—C6A—H6B | 108.3 | C10B—C9B—H9D | 110.2 |
| C7A—C6A—H6B | 108.3 | N8B—C9B—H9D | 110.2 |
| H6A—C6A—H6B | 107.4 | H9C—C9B—H9D | 108.5 |
| N8A—C7A—C71A | 114.4 (4) | C9B—C10B—N11B | 109.7 (4) |
| N8A—C7A—C6A | 110.2 (4) | C9B—C10B—H10C | 109.7 |
| C71A—C7A—C6A | 110.3 (5) | N11B—C10B—H10C | 109.7 |
| N8A—C7A—C72A | 106.4 (5) | C9B—C10B—H10D | 109.7 |
| C71A—C7A—C72A | 107.3 (5) | N11B—C10B—H10D | 109.7 |

| | | | |
|---|---|---|---|
| C6A—C7A—C72A | 108.0 (5) | H10C—C10B—H10D | 108.2 |
| N8A—C9A—C10A | 107.5 (3) | N11B—C12B—C13B | 118.5 (4) |
| N8A—C9A—H9A | 110.2 | N11B—C12B—H12C | 107.7 |
| C10A—C9A—H9A | 110.2 | C13B—C12B—H12C | 107.7 |
| N8A—C9A—H9B | 110.2 | N11B—C12B—H12D | 107.7 |
| C10A—C9A—H9B | 110.2 | C13B—C12B—H12D | 107.7 |
| H9A—C9A—H9B | 108.5 | H12C—C12B—H12D | 107.1 |
| N11A—C10A—C9A | 108.0 (3) | C12B—C13B—C14B | 117.0 (4) |
| N11A—C10A—H10A | 110.1 | C12B—C13B—N17B | 109.8 (3) |
| C9A—C10A—H10A | 110.1 | C14B—C13B—N17B | 105.6 (3) |
| N11A—C10A—H10B | 110.1 | C12B—C13B—C15B | 110.0 (4) |
| C9A—C10A—H10B | 110.1 | C14B—C13B—C15B | 109.3 (4) |
| H10A—C10A—H10B | 108.4 | N17B—C13B—C15B | 104.3 (3) |
| N11A—C12A—C13A | 114.4 (3) | N1B—C14B—C13B | 115.7 (3) |
| N11A—C12A—H12A | 108.7 | N1B—C14B—H14C | 108.4 |
| C13A—C12A—H12A | 108.7 | C13B—C14B—H14C | 108.4 |
| N11A—C12A—H12B | 108.7 | N1B—C14B—H14D | 108.4 |
| C13A—C12A—H12B | 108.7 | C13B—C14B—H14D | 108.4 |
| H12A—C12A—H12B | 107.6 | H14C—C14B—H14D | 107.4 |
| C12A—C13A—C14A | 115.2 (3) | C16B—C15B—C13B | 114.2 (4) |
| C12A—C13A—N17A | 108.9 (3) | C16B—C15B—H15F | 108.7 |
| C14A—C13A—N17A | 108.1 (3) | C13B—C15B—H15F | 108.7 |
| C12A—C13A—C15A | 111.0 (3) | C16B—C15B—H15G | 108.7 |
| C14A—C13A—C15A | 107.7 (3) | C13B—C15B—H15G | 108.7 |
| N17A—C13A—C15A | 105.6 (3) | H15F—C15B—H15G | 107.6 |
| N1A—C14A—C13A | 115.0 (3) | C15B—C16B—H16D | 109.5 |
| N1A—C14A—H14A | 108.5 | C15B—C16B—H16E | 109.5 |
| C13A—C14A—H14A | 108.5 | H16D—C16B—H16E | 109.5 |
| N1A—C14A—H14B | 108.5 | C15B—C16B—H16F | 109.5 |
| C13A—C14A—H14B | 108.5 | H16D—C16B—H16F | 109.5 |
| H14A—C14A—H14B | 107.5 | H16E—C16B—H16F | 109.5 |
| C5A—C51A—H51A | 109.5 | C5B—C51B—H51D | 109.5 |
| C5A—C51A—H51B | 109.5 | C5B—C51B—H51E | 109.5 |
| H51A—C51A—H51B | 109.5 | H51D—C51B—H51E | 109.5 |
| C5A—C51A—H51C | 109.5 | C5B—C51B—H51F | 109.5 |
| H51A—C51A—H51C | 109.5 | H51D—C51B—H51F | 109.5 |
| H51B—C51A—H51C | 109.5 | H51E—C51B—H51F | 109.5 |
| C7A—C71A—H71A | 109.5 | C7B—C71B—H71D | 109.5 |
| C7A—C71A—H71B | 109.5 | C7B—C71B—H71E | 109.5 |
| H71A—C71A—H71B | 109.5 | H71D—C71B—H71E | 109.5 |
| C7A—C71A—H71C | 109.5 | C7B—C71B—H71F | 109.5 |
| H71A—C71A—H71C | 109.5 | H71D—C71B—H71F | 109.5 |
| H71B—C71A—H71C | 109.5 | H71E—C71B—H71F | 109.5 |
| C7A—C72A—H72A | 109.5 | C7B—C72B—H72D | 109.5 |
| C7A—C72A—H72B | 109.5 | C7B—C72B—H72E | 109.5 |
| H72A—C72A—H72B | 109.5 | H72D—C72B—H72E | 109.5 |
| C7A—C72A—H72C | 109.5 | C7B—C72B—H72F | 109.5 |
| H72A—C72A—H72C | 109.5 | H72D—C72B—H72F | 109.5 |

| | | | |
|---|---|---|---|
| H72B—C72A—H72C | 109.5 | H72E—C72B—H72F | 109.5 |
| C16A—C15A—C13A | 115.9 (4) | O18B—N17B—O19B | 123.1 (4) |
| C16A—C15A—H15A | 108.3 | O18B—N17B—C13B | 119.3 (4) |
| C13A—C15A—H15A | 108.3 | O19B—N17B—C13B | 117.6 (4) |
| C16A—C15A—H15B | 108.3 | Cu1B—O10—H10E | 125 (4) |
| C13A—C15A—H15B | 108.3 | Cu1B—O10—H10F | 114 (4) |
| H15A—C15A—H15B | 107.4 | H10E—O10—H10F | 103 (4) |
| C15A—C16A—H16A | 109.5 | C65$^{ii}$—Ni6—C65 | 180.0 |
| C15A—C16A—H16B | 109.5 | C65$^{ii}$—Ni6—C66$^{ii}$ | 89.14 (18) |
| H16A—C16A—H16B | 109.5 | C65—Ni6—C66$^{ii}$ | 90.86 (18) |
| C15A—C16A—H16C | 109.5 | C65$^{ii}$—Ni6—C66 | 90.86 (18) |
| H16A—C16A—H16C | 109.5 | C65—Ni6—C66 | 89.14 (18) |
| H16B—C16A—H16C | 109.5 | C66$^{ii}$—Ni6—C66 | 180.00 (14) |
| C56—Ni5—C56$^{i}$ | 179.999 (1) | C65—N65—Cu1B | 137.7 (3) |
| C56—Ni5—C55 | 90.63 (16) | N65—C65—Ni6 | 178.1 (4) |
| C56$^{i}$—Ni5—C55 | 89.37 (16) | N66—C66—Ni6 | 179.1 (4) |
| C56—Ni5—C55$^{i}$ | 89.37 (16) | C75—Ni7—C75$^{iii}$ | 180.000 (1) |
| C56$^{i}$—Ni5—C55$^{i}$ | 90.63 (16) | C75—Ni7—C76$^{iii}$ | 89.64 (19) |
| C55—Ni5—C55$^{i}$ | 179.999 (1) | C75$^{iii}$—Ni7—C76$^{iii}$ | 90.36 (19) |
| C55—N55—Cu1A | 168.2 (3) | C75—Ni7—C76 | 90.36 (19) |
| N55—C55—Ni5 | 177.4 (4) | C75$^{iii}$—Ni7—C76 | 89.64 (19) |
| N56—C56—Ni5 | 179.6 (5) | C76$^{iii}$—Ni7—C76 | 179.998 (3) |
| N4B—Cu1B—N8B | 95.9 (2) | N75—C75—Ni7 | 179.2 (6) |
| N4B—Cu1B—N1B | 86.0 (2) | N76—C76—Ni7 | 179.0 (4) |
| N8B—Cu1B—N1B | 177.7 (2) | C85$^{iv}$—Ni8—C85 | 179.999 (2) |
| N4B—Cu1B—N11B | 170.7 (2) | C85$^{iv}$—Ni8—C86$^{iv}$ | 89.5 (2) |
| N8B—Cu1B—N11B | 86.9 (2) | C85—Ni8—C86$^{iv}$ | 90.5 (2) |
| N1B—Cu1B—N11B | 91.0 (2) | C85$^{iv}$—Ni8—C86 | 90.5 (2) |
| N4B—Cu1B—O10 | 101.6 (2) | C85—Ni8—C86 | 89.5 (2) |
| N8B—Cu1B—O10 | 87.0 (2) | C86$^{iv}$—Ni8—C86 | 180.0 |
| N1B—Cu1B—O10 | 93.9 (1) | N85—C85—Ni8 | 179.4 (5) |
| N11B—Cu1B—O10 | 87.3 (2) | N86—C86—Ni8 | 179.8 (5) |
| N4B—Cu1B—N65 | 88.6 (2) | H11E—O11—H11F | 109 (4) |
| N8B—Cu1B—N65 | 96.8 (2) | H12E—O12—H12F | 112 (4) |
| N1B—Cu1B—N65 | 81.9 (1) | H13E—O13—H13F | 106 (4) |
| N11B—Cu1B—N65 | 82.2 (1) | H14E—O14—H14F | 105 (4) |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*A*—H1*A*···N85$^{v}$ | 0.91 | 2.26 | 3.124 (5) | 159 |
| N11*A*—H11*A*···O14 | 0.91 | 2.11 | 2.869 (5) | 140 |
| N11*A*—H11*A*···O19*A* | 0.91 | 2.41 | 2.978 (5) | 121 |
| N1*B*—H1*B*···O19*A* | 0.91 | 2.12 | 2.937 (5) | 149 |
| N8*B*—H8*B*···O12 | 0.91 | 2.13 | 3.041 (5) | 174 |
| N8*B*—H8*B*···O10 | 0.91 | 2.58 | 3.052 (5) | 113 |

| | | | | |
|---|---|---|---|---|
| N11*B*—H11*B*···O19*B* | 0.91 | 2.29 | 2.874 (5) | 122 |
| O10—H10*E*···N75$^{vi}$ | 0.84 (5) | 1.92 (5) | 2.745 (6) | 168 (5) |
| O10—H10*F*···O12 | 0.85 (5) | 1.93 (5) | 2.702 (5) | 151 (5) |
| O11—H11*E*···N66$^{vii}$ | 0.84 (3) | 2.04 (4) | 2.870 (5) | 170 (6) |
| O11—H11*F*···N56 | 0.83 (5) | 2.28 (5) | 3.106 (5) | 172 (5) |
| O12—H12*F*···N86 | 0.83 (4) | 1.97 (4) | 2.788 (5) | 168 (6) |
| O12—H12*E*···O13 | 0.84 (4) | 1.88 (3) | 2.702 (5) | 171 (6) |
| O13—H13*E*···O11$^{viii}$ | 0.84 (4) | 1.94 (4) | 2.733 (5) | 158 (6) |
| O13—H13*F*···N56$^{vii}$ | 0.83 (4) | 2.15 (4) | 2.973 (6) | 170 (5) |
| O14—H14*E*···N76$^{vi}$ | 0.82 (4) | 2.03 (4) | 2.847 (5) | 174 (7) |
| O14—H14*F*···N85$^{v}$ | 0.82 (5) | 2.10 (5) | 2.872 (6) | 159 (6) |

Symmetry codes: (v) $x, y+1, z$; (vi) $x-1, y, z$; (vii) $-x+1, -y+1, -z$; (viii) $x-1, y-1, z$.