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A tetranuclear nickel(II) cluster: bis[μ_3-2,6-bis(methylamino)pyridine(2−)-κ^4N^2:N^1,N^6:N^6]bis[μ_3-2,6-bis(methylamino)pyridine(1−)-κ^3N^1:N^2:N^2]-dichloridotetranickel(II)

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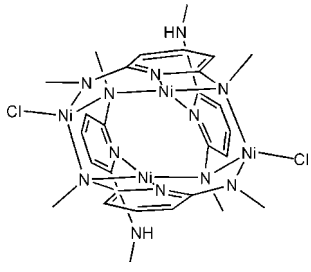
Key indicators: single-crystal X-ray study; T = 213 K; mean σ(C–C) = 0.007 Å; R factor = 0.042; wR factor = 0.094; data-to-parameter ratio = 14.2.

The title compound, $[Ni_4(C_7H_9N_3)_2(C_7H_{10}N_3)_2Cl_2]$, was obtained from a reaction in which 2,6-dimethylaminopyridine, in the presence of methyllithium used to deprotonate the organic ligand precursor, was added to $NiCl_2$ during an attempt to synthesize a linear trinuclear nickel chain. During this reaction, two equivalents of the amine were fully deprotonated, forming dianions, and the three N atoms coordinate to Ni centers. The other two equivalents of the amine were only partially deprotonated, giving monoanions in which only two N atoms coordinate to Ni centers. In this tetranuclear complex, two of the four Ni atoms have square-planar coordination and are bonded to four N donors, while the other two are in a distorted tetrahedral geometry, with three coordination sites occupied by N donors and the fourth by a Cl atom. The molecule is noncentrosymmetic but the crystal structure is racemic.

Related literature

For related structures, see: Clérac *et al.* (1999); Cotton *et al.* (2001, 2006).



Experimental

Crystal data

$[Ni_4(C_7H_9N_3)_2(C_7H_{10}N_3)_2Cl_2]$
M_r = 848.44
Monoclinic, $P2_1/c$
a = 12.710 (7) Å
b = 10.903 (6) Å
c = 24.729 (14) Å
β = 99.842 (11)°
V = 3376 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.40 mm^{-1}
T = 213 (2) K
0.38 × 0.10 × 0.10 mm

Data collection

Bruker SMART 1000 area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.463, T_{max} = 0.796
16834 measured reflections
5894 independent reflections
4026 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.094
S = 1.08
5894 reflections
415 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—N2	1.891 (4)	Ni3—N3	1.929 (4)
Ni1—N8	1.904 (4)	Ni3—N5	2.012 (4)
Ni1—N1	1.919 (4)	Ni3—N11	2.059 (4)
Ni1—N11	1.957 (4)	Ni3—Cl2	2.2492 (18)
Ni2—N4	1.945 (4)	Ni4—N6	1.885 (4)
Ni2—N1	2.000 (4)	Ni4—N10	1.913 (4)
Ni2—N9	2.056 (4)	Ni4—N5	1.915 (4)
Ni2—Cl1	2.2369 (17)	Ni4—N9	1.948 (4)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2066).

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supplementary materials

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A tetranuclear nickel(II) cluster: bis[μ_3-2,6-bis(methylamino)pyridine(2-)-$\kappa^4N^2:N^1,N^6:N^6$]bis[μ_3-2,6-bis(methylamino)pyridine(1-)-$\kappa^3N^1:N^2:N^2$]dichloridotetranickel(II)

F. A. Cotton, C. A. Murillo and Q. Wang

Comment

Molecules in the title complex, (I) (Fig. 1), are non-centrosymmetic but the crystals are racemic. During the reaction, two equivalents of the organic amine were fully deprotonated forming dianions and two equivalents were only partially deprotonated giving monoanions. In this tetranuclear complex, there are four divalent Ni atoms. Two of them, Ni1 and Ni4, are square planar and bonded to four N donors while the others, Ni2 and Ni3, are in a distorted tetrahedral geometry, with three coordination sites occupied by N donors and the fourth one by a Cl atom (Table 1). Even though there are a series of compounds having three Ni atoms forming a linear chain that have both four-coordinate and five-coordinate metal atoms (Clérac *et al.*, 1999; Cotton *et al.*, 2001, 2006), examples of clusters having four Ni atoms in a four-coordinate environment but two different geometries are rare.

Experimental

Compound (I) was obtained by adding 2 equivalents of CH_3Li (2 mmol) to deprotonate a THF solution (15 ml) of the organic amine, 2,6-dimethylaminopyridine (0.137 g, 1 mmol), at 195 K. This mixture was then transferred *via* cannula to solid, anhydrous $NiCl_2$ (0.130 g, 1 mmol). After stirring the mixture at room temperature for 5 h, the solvent was removed under vacuum. The brown solid was extracted with CH_2Cl_2 (10 ml). To this solution was added a layer of hexanes (30 ml). After a week at room temperature, large block-shaped, deep brown crystals were isolated.

Refinement

All H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.94 Å (CH) and 0.97 Å (CH_3), U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(C), and N—H = 0.87 Å, U_{iso}(H) = 1.2U_{eq}(N).

Figures

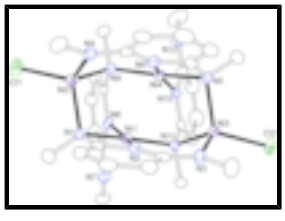

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 40% probability level. H atoms have been omitted for clarity.

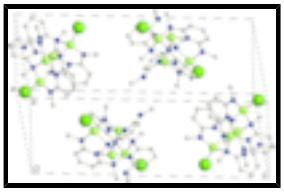

Fig. 2. The packing diagram of (I).

bis[μ_3-2,6-bis(methylamino)pyridine(2-)-$\kappa^4N^2:N^1,N^6:N^6$]bis[μ_3– 2,6-bis(methylamino)pyridine(1-)-$\kappa^3N^1:N^2:N^2$]dichloridotetranickel(II)

Crystal data

$[Ni_4(C_7H_9N_3)_2(C_7H_{10}N_3)_2Cl_2]$	F_{000} = 1744
M_r = 848.44	D_x = 1.669 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5894 reflections
a = 12.710 (7) Å	θ = 2.5–25.0º
b = 10.903 (6) Å	μ = 2.40 mm^{-1}
c = 24.729 (14) Å	T = 213 (2) K
β = 99.842 (11)º	Block, brown
V = 3376 (3) Å^3	0.38 × 0.10 × 0.10 mm
Z = 4	

Data collection

Bruker SMART 1000 area-detector diffractometer	5894 independent reflections
Radiation source: fine-focus sealed tube	4026 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.057
T = 213(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −15→15
T_{min} = 0.463, T_{max} = 0.796	k = −12→9
16834 measured reflections	l = −29→29

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.033P)^2 + 0.98P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.042	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.094	$\Delta\rho_{max}$ = 0.66 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.57 e Å^{-3}
5894 reflections	Extinction correction: none
415 parameters	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.85881 (5)	0.60891 (5)	0.15069 (2)	0.02005 (16)
Ni2	0.69244 (5)	0.44549 (6)	0.08446 (2)	0.02496 (17)
Ni3	0.78862 (5)	0.84198 (6)	0.22024 (2)	0.02465 (17)
Ni4	0.68609 (5)	0.74121 (6)	0.11001 (2)	0.02360 (16)
Cl1	0.69651 (12)	0.30291 (14)	0.01981 (6)	0.0501 (4)
Cl2	0.82948 (11)	1.02671 (12)	0.25863 (6)	0.0431 (4)
N9	0.7045 (3)	0.6199 (3)	0.05452 (14)	0.0229 (9)
N1	0.8208 (3)	0.4386 (4)	0.14373 (14)	0.0236 (9)
N6	0.5910 (3)	0.6543 (4)	0.14663 (15)	0.0275 (10)
N11	0.8911 (3)	0.7805 (3)	0.17005 (14)	0.0214 (9)
N8	0.8902 (3)	0.6215 (3)	0.07831 (14)	0.0211 (9)
N5	0.6492 (3)	0.8413 (4)	0.16761 (15)	0.0265 (10)
C12	0.7661 (4)	0.3467 (5)	0.22874 (18)	0.0266 (11)
H12A	0.7598	0.2651	0.2163	0.032*
C45	0.8823 (4)	0.8608 (4)	0.12564 (19)	0.0244 (11)
C35	0.8042 (4)	0.6318 (4)	0.03666 (18)	0.0211 (11)
N3	0.7878 (3)	0.7147 (4)	0.27437 (15)	0.0281 (10)
C41	0.7679 (4)	0.9352 (5)	0.0482 (2)	0.0342 (13)
C15	0.7818 (4)	0.5948 (5)	0.26349 (18)	0.0261 (12)
C31	0.9908 (4)	0.6291 (4)	0.06617 (18)	0.0224 (11)
C34	0.8177 (4)	0.6542 (4)	−0.01643 (18)	0.0271 (12)
H34A	0.7584	0.6600	−0.0448	0.033*
N7	1.0728 (3)	0.6098 (4)	0.10788 (15)	0.0282 (10)
H7A	1.0583	0.5847	0.1392	0.034*
N2	0.8066 (3)	0.5571 (4)	0.21415 (14)	0.0224 (9)
N10	0.7866 (3)	0.8578 (3)	0.09147 (15)	0.0247 (9)
C33	0.9199 (4)	0.6679 (5)	−0.02711 (19)	0.0313 (13)
H33A	0.9297	0.6859	−0.0631	0.038*
C1	0.8999 (4)	0.3439 (5)	0.1353 (2)	0.0347 (13)
H1A	0.9595	0.3461	0.1655	0.052*
H1B	0.9255	0.3600	0.1012	0.052*
H1C	0.8666	0.2637	0.1335	0.052*
C4	0.6031 (4)	0.9650 (5)	0.1564 (2)	0.0421 (15)
H4A	0.5362	0.9588	0.1310	0.063*
H4B	0.6525	1.0158	0.1405	0.063*
H4C	0.5902	1.0016	0.1905	0.063*
C8	0.9947 (3)	0.7864 (4)	0.20777 (19)	0.0271 (12)
H8A	1.0512	0.7592	0.1887	0.041*
H8B	0.9924	0.7338	0.2392	0.041*
H8C	1.0084	0.8702	0.2202	0.041*
C6	0.6110 (4)	0.6238 (5)	0.00933 (18)	0.0300 (12)
H6A	0.6078	0.7032	−0.0086	0.045*
H6B	0.5461	0.6107	0.0242	0.045*
H6C	0.6180	0.5600	−0.0172	0.045*
C21	0.5402 (4)	0.5453 (5)	0.14940 (18)	0.0263 (11)

C13	0.7460 (4)	0.3815 (5)	0.28017 (19)	0.0308 (13)
H13A	0.7270	0.3209	0.3037	0.037*
C32	1.0067 (4)	0.6562 (5)	0.01277 (18)	0.0291 (12)
H32A	1.0759	0.6660	0.0048	0.035*
C22	0.4607 (4)	0.5417 (5)	0.18400 (19)	0.0325 (13)
H22A	0.4211	0.4698	0.1866	0.039*
C42	0.8451 (5)	1.0147 (5)	0.0362 (2)	0.0456 (15)
H42A	0.8312	1.0669	0.0056	0.055*
C11	0.7955 (3)	0.4397 (5)	0.19779 (18)	0.0231 (11)
N4	0.5677 (3)	0.4520 (4)	0.12021 (15)	0.0271 (10)
C25	0.5747 (4)	0.7504 (5)	0.17780 (19)	0.0301 (12)
C14	0.7529 (4)	0.4998 (5)	0.29750 (19)	0.0312 (13)
H14A	0.7383	0.5192	0.3325	0.037*
C5	1.1831 (4)	0.6287 (5)	0.1031 (2)	0.0371 (14)
H5A	1.2283	0.6097	0.1378	0.056*
H5B	1.1937	0.7136	0.0936	0.056*
H5C	1.2016	0.5755	0.0747	0.056*
C2	0.7635 (5)	0.7497 (5)	0.32769 (19)	0.0450 (15)
H2A	0.7660	0.6777	0.3509	0.068*
H2B	0.6927	0.7856	0.3230	0.068*
H2C	0.8156	0.8092	0.3447	0.068*
C23	0.4429 (4)	0.6434 (6)	0.2131 (2)	0.0408 (15)
H23A	0.3897	0.6401	0.2352	0.049*
C44	0.9628 (4)	0.9405 (5)	0.1151 (2)	0.0347 (13)
H44A	1.0294	0.9425	0.1385	0.042*
C24	0.5000 (4)	0.7520 (5)	0.2116 (2)	0.0372 (14)
H24A	0.4881	0.8213	0.2323	0.045*
C7	0.6361 (6)	0.9856 (6)	−0.0350 (2)	0.084 (3)
H7B	0.5610	0.9703	−0.0486	0.126*
H7C	0.6786	0.9510	−0.0603	0.126*
H7D	0.6485	1.0733	−0.0319	0.126*
N12	0.6660 (4)	0.9295 (4)	0.01813 (18)	0.0474 (13)
H12B	0.6174	0.8900	0.0320	0.057*
C3	0.5172 (4)	0.3343 (5)	0.1267 (2)	0.0370 (13)
H3A	0.4560	0.3464	0.1448	0.055*
H3B	0.5680	0.2802	0.1487	0.055*
H3C	0.4938	0.2977	0.0909	0.055*
C43	0.9424 (5)	1.0160 (5)	0.0698 (2)	0.0478 (16)
H43A	0.9961	1.0690	0.0619	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0208 (3)	0.0221 (4)	0.0173 (3)	−0.0009 (3)	0.0035 (2)	−0.0001 (3)
Ni2	0.0246 (3)	0.0261 (4)	0.0238 (3)	−0.0024 (3)	0.0032 (3)	−0.0022 (3)
Ni3	0.0248 (3)	0.0252 (4)	0.0231 (3)	0.0019 (3)	0.0016 (3)	−0.0030 (3)
Ni4	0.0203 (3)	0.0264 (4)	0.0230 (3)	0.0010 (3)	0.0007 (3)	−0.0012 (3)
Cl1	0.0583 (9)	0.0478 (10)	0.0442 (8)	0.0032 (8)	0.0088 (7)	−0.0205 (7)

Cl2	0.0510 (9)	0.0275 (8)	0.0484 (8)	−0.0018 (7)	0.0018 (7)	−0.0116 (6)
N9	0.022 (2)	0.025 (2)	0.021 (2)	−0.002 (2)	0.0005 (17)	−0.0003 (17)
N1	0.023 (2)	0.026 (2)	0.022 (2)	−0.002 (2)	0.0049 (17)	−0.0030 (18)
N6	0.022 (2)	0.037 (3)	0.022 (2)	0.001 (2)	0.0009 (17)	−0.0017 (19)
N11	0.023 (2)	0.021 (2)	0.020 (2)	0.0011 (19)	0.0014 (16)	0.0014 (17)
N8	0.025 (2)	0.023 (2)	0.016 (2)	−0.0025 (19)	0.0048 (17)	−0.0027 (16)
N5	0.024 (2)	0.029 (2)	0.025 (2)	0.003 (2)	0.0012 (18)	−0.0068 (19)
C12	0.027 (3)	0.022 (3)	0.030 (3)	−0.005 (2)	0.000 (2)	0.007 (2)
C45	0.029 (3)	0.016 (3)	0.028 (3)	0.002 (2)	0.005 (2)	−0.002 (2)
C35	0.025 (3)	0.016 (3)	0.022 (2)	−0.001 (2)	0.004 (2)	−0.003 (2)
N3	0.035 (2)	0.029 (3)	0.023 (2)	0.002 (2)	0.0104 (19)	−0.0047 (19)
C41	0.046 (3)	0.023 (3)	0.031 (3)	0.004 (3)	0.001 (3)	0.005 (2)
C15	0.020 (2)	0.040 (4)	0.017 (2)	0.005 (3)	0.001 (2)	0.004 (2)
C31	0.026 (3)	0.019 (3)	0.024 (3)	−0.001 (2)	0.008 (2)	−0.001 (2)
C34	0.033 (3)	0.029 (3)	0.019 (3)	0.000 (3)	0.003 (2)	0.004 (2)
N7	0.018 (2)	0.043 (3)	0.025 (2)	−0.001 (2)	0.0056 (17)	0.002 (2)
N2	0.024 (2)	0.025 (2)	0.019 (2)	0.002 (2)	0.0069 (17)	0.0009 (18)
N10	0.027 (2)	0.019 (2)	0.027 (2)	0.005 (2)	−0.0003 (18)	0.0015 (18)
C33	0.041 (3)	0.035 (3)	0.020 (3)	−0.004 (3)	0.012 (2)	0.004 (2)
C1	0.034 (3)	0.038 (3)	0.034 (3)	0.008 (3)	0.009 (2)	−0.003 (3)
C4	0.042 (3)	0.043 (4)	0.037 (3)	0.015 (3)	−0.004 (3)	−0.006 (3)
C8	0.020 (2)	0.028 (3)	0.031 (3)	−0.001 (2)	−0.003 (2)	−0.002 (2)
C6	0.029 (3)	0.035 (3)	0.024 (3)	−0.003 (3)	−0.002 (2)	−0.001 (2)
C21	0.020 (3)	0.035 (3)	0.022 (3)	−0.003 (3)	−0.002 (2)	−0.001 (2)
C13	0.026 (3)	0.038 (4)	0.027 (3)	−0.003 (3)	0.000 (2)	0.017 (2)
C32	0.031 (3)	0.032 (3)	0.027 (3)	−0.004 (3)	0.012 (2)	0.000 (2)
C22	0.017 (3)	0.047 (4)	0.033 (3)	0.000 (3)	0.006 (2)	0.005 (3)
C42	0.070 (4)	0.030 (3)	0.036 (3)	−0.003 (3)	0.007 (3)	0.012 (3)
C11	0.013 (2)	0.033 (3)	0.022 (2)	0.006 (2)	−0.0010 (19)	−0.001 (2)
N4	0.024 (2)	0.028 (3)	0.029 (2)	−0.005 (2)	0.0031 (18)	0.000 (2)
C25	0.023 (3)	0.039 (3)	0.028 (3)	0.002 (3)	0.001 (2)	−0.002 (2)
C14	0.029 (3)	0.043 (4)	0.022 (3)	0.003 (3)	0.007 (2)	0.007 (2)
C5	0.024 (3)	0.055 (4)	0.032 (3)	−0.001 (3)	0.006 (2)	−0.002 (3)
C2	0.065 (4)	0.045 (4)	0.027 (3)	0.006 (3)	0.016 (3)	−0.003 (3)
C23	0.026 (3)	0.070 (4)	0.030 (3)	0.008 (3)	0.015 (2)	0.003 (3)
C44	0.029 (3)	0.031 (3)	0.045 (3)	−0.008 (3)	0.009 (2)	0.002 (3)
C24	0.025 (3)	0.052 (4)	0.036 (3)	0.008 (3)	0.007 (2)	−0.004 (3)
C7	0.127 (7)	0.061 (5)	0.043 (4)	−0.002 (5)	−0.045 (4)	0.020 (3)
N12	0.055 (3)	0.041 (3)	0.040 (3)	0.002 (3)	−0.012 (2)	0.013 (2)
C3	0.037 (3)	0.045 (4)	0.030 (3)	−0.010 (3)	0.007 (2)	0.001 (3)
C43	0.052 (4)	0.032 (4)	0.063 (4)	−0.017 (3)	0.022 (3)	0.006 (3)

Geometric parameters (Å, °)

Ni1—N2	1.891 (4)	N7—H7A	0.8700
Ni1—N8	1.904 (4)	N2—C11	1.343 (6)
Ni1—N1	1.919 (4)	C33—C32	1.354 (6)
Ni1—N11	1.957 (4)	C33—H33A	0.9400
Ni1—C11	2.393 (5)	C1—H1A	0.9700

Ni1—Ni4	2.6753 (14)	C1—H1B	0.9700
Ni2—N4	1.945 (4)	C1—H1C	0.9700
Ni2—N1	2.000 (4)	C4—H4A	0.9700
Ni2—N9	2.056 (4)	C4—H4B	0.9700
Ni2—Cl1	2.2369 (17)	C4—H4C	0.9700
Ni3—N3	1.929 (4)	C8—H8A	0.9700
Ni3—N5	2.012 (4)	C8—H8B	0.9700
Ni3—N11	2.059 (4)	C8—H8C	0.9700
Ni3—Cl2	2.2492 (18)	C6—H6A	0.9700
Ni4—N6	1.885 (4)	C6—H6B	0.9700
Ni4—N10	1.913 (4)	C6—H6C	0.9700
Ni4—N5	1.915 (4)	C21—N4	1.328 (6)
Ni4—N9	1.948 (4)	C21—C22	1.432 (6)
Ni4—C25	2.374 (5)	C13—C14	1.357 (7)
N9—C35	1.417 (5)	C13—H13A	0.9400
N9—C6	1.487 (5)	C32—H32A	0.9400
N1—C11	1.427 (5)	C22—C23	1.362 (7)
N1—C1	1.480 (6)	C22—H22A	0.9400
N6—C25	1.337 (6)	C42—C43	1.366 (8)
N6—C21	1.360 (6)	C42—H42A	0.9400
N11—C45	1.394 (6)	N4—C3	1.456 (6)
N11—C8	1.479 (5)	C25—C24	1.369 (6)
N8—C31	1.365 (5)	C14—H14A	0.9400
N8—C35	1.372 (5)	C5—H5A	0.9700
N5—C25	1.423 (6)	C5—H5B	0.9700
N5—C4	1.478 (6)	C5—H5C	0.9700
C12—C11	1.361 (6)	C2—H2A	0.9700
C12—C13	1.392 (6)	C2—H2B	0.9700
C12—H12A	0.9400	C2—H2C	0.9700
C45—N10	1.357 (6)	C23—C24	1.393 (7)
C45—C44	1.401 (6)	C23—H23A	0.9400
C35—C34	1.375 (6)	C44—C43	1.378 (7)
N3—C15	1.334 (6)	C44—H44A	0.9400
N3—C2	1.456 (5)	C24—H24A	0.9400
C41—N10	1.352 (6)	C7—N12	1.440 (7)
C41—N12	1.379 (6)	C7—H7B	0.9700
C41—C42	1.379 (7)	C7—H7C	0.9700
C15—N2	1.374 (5)	C7—H7D	0.9700
C15—C14	1.422 (6)	N12—H12B	0.8700
C31—N7	1.352 (6)	C3—H3A	0.9700
C31—C32	1.401 (6)	C3—H3B	0.9700
C34—C33	1.377 (6)	C3—H3C	0.9700
C34—H34A	0.9400	C43—H43A	0.9400
N7—C5	1.442 (5)		
N2—Ni1—N8	163.92 (17)	C41—N10—C45	119.7 (4)
N2—Ni1—N1	70.54 (16)	C41—N10—Ni4	124.7 (3)
N8—Ni1—N1	94.31 (15)	C45—N10—Ni4	115.5 (3)
N2—Ni1—N11	99.85 (16)	C32—C33—C34	121.8 (4)
N8—Ni1—N11	95.30 (15)	C32—C33—H33A	119.1

N1—Ni1—N11	170.38 (15)	C34—C33—H33A	119.1
N2—Ni1—C11	34.04 (15)	N1—C1—H1A	109.5
N8—Ni1—C11	130.90 (16)	N1—C1—H1B	109.5
N1—Ni1—C11	36.62 (15)	H1A—C1—H1B	109.5
N11—Ni1—C11	133.77 (15)	N1—C1—H1C	109.5
N2—Ni1—Ni4	95.25 (12)	H1A—C1—H1C	109.5
N8—Ni1—Ni4	84.14 (12)	H1B—C1—H1C	109.5
N1—Ni1—Ni4	108.08 (12)	N5—C4—H4A	109.5
N11—Ni1—Ni4	72.60 (11)	N5—C4—H4B	109.5
C11—Ni1—Ni4	106.04 (11)	H4A—C4—H4B	109.5
N4—Ni2—N1	107.15 (16)	N5—C4—H4C	109.5
N4—Ni2—N9	104.29 (16)	H4A—C4—H4C	109.5
N1—Ni2—N9	101.35 (15)	H4B—C4—H4C	109.5
N4—Ni2—Cl1	118.08 (13)	N11—C8—H8A	109.5
N1—Ni2—Cl1	112.46 (12)	N11—C8—H8B	109.5
N9—Ni2—Cl1	111.92 (12)	H8A—C8—H8B	109.5
N3—Ni3—N5	109.72 (17)	N11—C8—H8C	109.5
N3—Ni3—N11	105.25 (16)	H8A—C8—H8C	109.5
N5—Ni3—N11	100.78 (15)	H8B—C8—H8C	109.5
N3—Ni3—Cl2	112.30 (13)	N9—C6—H6A	109.5
N5—Ni3—Cl2	113.00 (12)	N9—C6—H6B	109.5
N11—Ni3—Cl2	114.95 (11)	H6A—C6—H6B	109.5
N6—Ni4—N10	163.47 (17)	N9—C6—H6C	109.5
N6—Ni4—N5	70.75 (17)	H6A—C6—H6C	109.5
N10—Ni4—N5	93.34 (17)	H6B—C6—H6C	109.5
N6—Ni4—N9	99.29 (17)	N4—C21—N6	118.1 (4)
N10—Ni4—N9	96.68 (16)	N4—C21—C22	125.6 (5)
N5—Ni4—N9	169.96 (16)	N6—C21—C22	116.3 (5)
N6—Ni4—C25	34.22 (17)	C14—C13—C12	122.4 (4)
N10—Ni4—C25	130.15 (17)	C14—C13—H13A	118.8
N5—Ni4—C25	36.81 (16)	C12—C13—H13A	118.8
N9—Ni4—C25	133.15 (17)	C33—C32—C31	118.4 (4)
N6—Ni4—Ni1	95.77 (13)	C33—C32—H32A	120.8
N10—Ni4—Ni1	84.81 (12)	C31—C32—H32A	120.8
N5—Ni4—Ni1	108.49 (12)	C23—C22—C21	119.5 (5)
N9—Ni4—Ni1	73.09 (11)	C23—C22—H22A	120.3
C25—Ni4—Ni1	107.90 (12)	C21—C22—H22A	120.3
C35—N9—C6	113.9 (3)	C43—C42—C41	118.7 (5)
C35—N9—Ni4	112.3 (3)	C43—C42—H42A	120.7
C6—N9—Ni4	109.5 (3)	C41—C42—H42A	120.7
C35—N9—Ni2	108.8 (3)	N2—C11—C12	124.5 (4)
C6—N9—Ni2	101.4 (3)	N2—C11—N1	105.2 (4)
Ni4—N9—Ni2	110.41 (17)	C12—C11—N1	130.4 (5)
C11—N1—C1	113.9 (4)	N2—C11—Ni1	52.0 (2)
C11—N1—Ni1	90.1 (3)	C12—C11—Ni1	174.7 (4)
C1—N1—Ni1	121.4 (3)	N1—C11—Ni1	53.3 (2)
C11—N1—Ni2	113.6 (3)	C21—N4—C3	117.0 (4)
C1—N1—Ni2	114.3 (3)	C21—N4—Ni2	125.9 (3)
Ni1—N1—Ni2	100.99 (18)	C3—N4—Ni2	115.3 (3)

C25—N6—C21	122.2 (4)	N6—C25—C24	123.7 (5)
C25—N6—Ni4	93.4 (3)	N6—C25—N5	105.7 (4)
C21—N6—Ni4	144.3 (3)	C24—C25—N5	130.7 (5)
C45—N11—C8	114.4 (4)	N6—C25—Ni4	52.4 (2)
C45—N11—Ni1	115.0 (3)	C24—C25—Ni4	172.7 (4)
C8—N11—Ni1	108.9 (3)	N5—C25—Ni4	53.8 (2)
C45—N11—Ni3	107.2 (3)	C13—C14—C15	121.0 (5)
C8—N11—Ni3	101.1 (3)	C13—C14—H14A	119.5
Ni1—N11—Ni3	109.33 (17)	C15—C14—H14A	119.5
C31—N8—C35	119.0 (4)	N7—C5—H5A	109.5
C31—N8—Ni1	124.4 (3)	N7—C5—H5B	109.5
C35—N8—Ni1	116.3 (3)	H5A—C5—H5B	109.5
C25—N5—C4	114.5 (4)	N7—C5—H5C	109.5
C25—N5—Ni4	89.4 (3)	H5A—C5—H5C	109.5
C4—N5—Ni4	121.4 (3)	H5B—C5—H5C	109.5
C25—N5—Ni3	115.3 (3)	N3—C2—H2A	109.5
C4—N5—Ni3	113.3 (3)	N3—C2—H2B	109.5
Ni4—N5—Ni3	100.52 (17)	H2A—C2—H2B	109.5
C11—C12—C13	114.9 (5)	N3—C2—H2C	109.5
C11—C12—H12A	122.5	H2A—C2—H2C	109.5
C13—C12—H12A	122.5	H2B—C2—H2C	109.5
N10—C45—N11	114.7 (4)	C22—C23—C24	122.9 (5)
N10—C45—C44	120.3 (4)	C22—C23—H23A	118.5
N11—C45—C44	125.0 (4)	C24—C23—H23A	118.5
N8—C35—C34	121.2 (4)	C43—C44—C45	118.6 (5)
N8—C35—N9	113.5 (4)	C43—C44—H44A	120.7
C34—C35—N9	125.3 (4)	C45—C44—H44A	120.7
C15—N3—C2	115.2 (4)	C25—C24—C23	115.1 (5)
C15—N3—Ni3	124.9 (3)	C25—C24—H24A	122.4
C2—N3—Ni3	117.8 (3)	C23—C24—H24A	122.4
N10—C41—N12	114.9 (5)	N12—C7—H7B	109.5
N10—C41—C42	121.7 (5)	N12—C7—H7C	109.5
N12—C41—C42	123.3 (5)	H7B—C7—H7C	109.5
N3—C15—N2	117.3 (4)	N12—C7—H7D	109.5
N3—C15—C14	127.3 (4)	H7B—C7—H7D	109.5
N2—C15—C14	115.4 (5)	H7C—C7—H7D	109.5
N7—C31—N8	116.9 (4)	C41—N12—C7	123.4 (5)
N7—C31—C32	122.4 (4)	C41—N12—H12B	118.3
N8—C31—C32	120.8 (4)	C7—N12—H12B	118.3
C35—C34—C33	118.6 (4)	N4—C3—H3A	109.5
C35—C34—H34A	120.7	N4—C3—H3B	109.5
C33—C34—H34A	120.7	H3A—C3—H3B	109.5
C31—N7—C5	123.3 (4)	N4—C3—H3C	109.5
C31—N7—H7A	118.4	H3A—C3—H3C	109.5
C5—N7—H7A	118.4	H3B—C3—H3C	109.5
C11—N2—C15	121.6 (4)	C42—C43—C44	120.8 (5)
C11—N2—Ni1	93.9 (3)	C42—C43—H43A	119.6
C15—N2—Ni1	144.4 (4)	C44—C43—H43A	119.6

Fig. 1

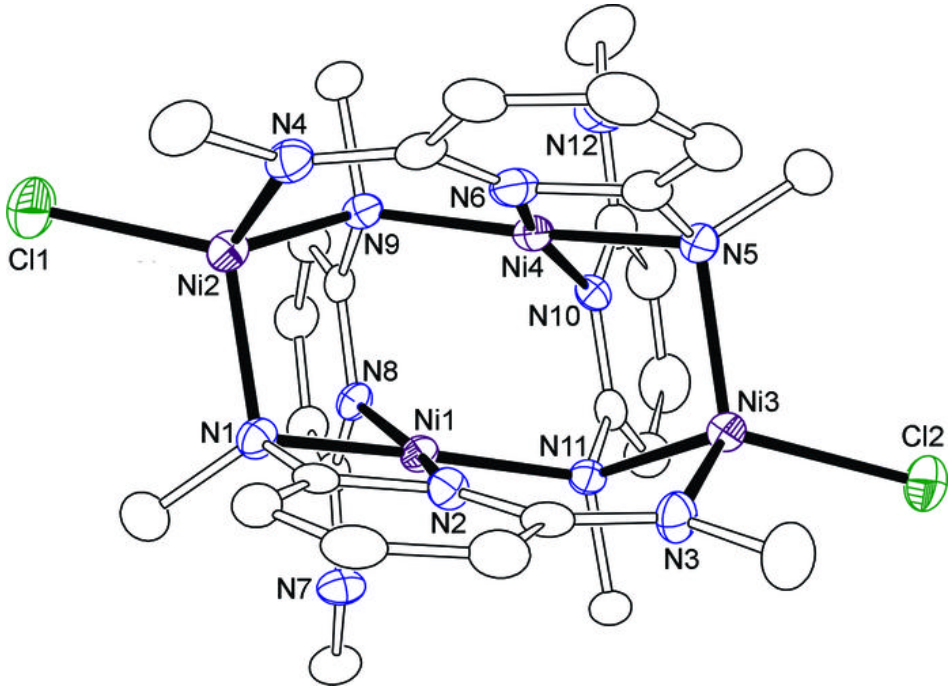

Fig. 2