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trans-Diazido(1,8-dibenzyl-1,3,6,8,10,-13-hexaazacyclotetradecane)nickel(II)

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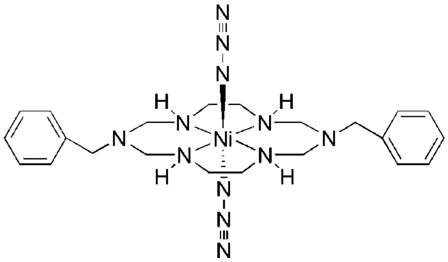
Key indicators: single-crystal X-ray study; *T* = 173 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.045; *wR* factor = 0.092; data-to-parameter ratio = 17.6.

In the centrosymmetric title compound, $[Ni(N_3)_2(C_{22}H_{34}N_6)]$, the Ni^{II} ion is coordinated by the four secondary N atoms of the macrocyclic ligand in a square-planar fashion with two N atoms of the azide ions in axial positions, resulting in a tetragonally distorted octahedron. An N—H⋯N hydrogen-bonding interaction between the secondary amine N atom of the macrocycle and an adjacent azide ion gives rise to a chain structure.

Related literature

For related literature, see: Hancock (1990); Jacquinot & Hauser (2003); Jung *et al.* (1989); Larionova *et al.* (2003); Min & Suh (2001); Liu *et al.* (2006); Tsuge *et al.* (2004).



Experimental

Crystal data

$[Ni(N_3)_2(C_{22}H_{34}N_6)]$
M_r = 525.32
Monoclinic, $P2_1/c$
a = 10.2150 (5) Å
b = 15.8337 (9) Å
c = 7.5477 (4) Å
β = 92.817 (1)°
V = 1219.30 (11) Å^3
Z = 2
Mo *K*α radiation
μ = 0.83 mm^{-1}
T = 173 (2) K
0.50 × 0.20 × 0.20 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.733, T_{max} = 0.847
7464 measured reflections
2820 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.092
S = 1.19
2820 reflections
160 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N3—H3⋯N6^{i}	0.93	2.24	3.145 (3)	163

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT* and *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2057).

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supplementary materials

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trans-Diazido(1,8-dibenzyl-1,3,6,8,10,13-hexaazacyclotetradecane)nickel(II)

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Comment

Coordination compounds with tetraaza macrocyclic ligands have been widely studied in the context of metalloenzymes and the construction of extended coordination polymers (Tsuge *et al.*, 2004; Larionova *et al.*, 2003). Especially, Ni^{II} macrocyclic complexes having vacant sites axially are good candidates for assembling novel multi-dimensional networks and catalysts for the reduction of carbon dioxide in which they can have unique properties (Min & Suh, 2001; Jacquinot *et al.*, 2003). Furthermore, the azide ion is a bifunctional ligand which can link to transition metal complexes, thus allowing for the assembly of polymeric compounds (Liu *et al.*, 2006). Therefore, complexes combined with azide ions can also be building blocks for extended network structured materials. Here, we report the synthesis and structure of Ni^{II} macrocyclic complex, *trans*-diazido(1,8-dibenzyl-1,3,6,8,10,13- hexaazacyclotetradecane)nickel(II), with two azide ions axially.

In the title compound, the coordination geometry around Ni^{II} ion is tetragonally elongated octahedron in which Ni^{II} ion is bonded to the four secondary amine N atoms of the macrocyclic ligand in the square-planar fashion and two N atoms from the azide ions at the axial sites as shown in Fig. 1. The average $Ni—N_{eq}$ and $Ni—N_{ax}$ bond distances are 2.072 (1) and 2.159 (1) Å, respectively. The axial Ni—N bond distance is longer than the equatorial Ni—N bond lengths, which can be attributed to the Jahn-Teller distortion of the Ni^{II} ion and/or the ring contraction of the macrocyclic ligand. Two N—N bond distances of the azide ion are not significant different even though one terminal nitrogen atom is coordinated to Ni^{II} ion, indicate that the azide ion is delocalized fully (N4—N5 = 1.189 (3) Å and N5—N6 = 1.171 (3) Å). The complex has an inversion center at the nickel atom and the azamacrocyclic ligand adopts thermodynamically the most stable *R,R,S,S* configuration (Hancock, 1990). All azide ions coordinating Ni^{II} ions axially are involved in hydrogen bonding interactions, which results to a rigid supramolecular one-dimensional chain propagating along the *c* axis (Fig. 2).

Experimental

The title compound is prepared as follows. To a DMF/H_2O (*v*/*v*; 1:1, 20 ml) solution of $[Ni(C_{22}H_{34}N_6)Cl_2]$ (0.20 g, 0.40 mmol) (Jung *et al.*, 1989) was added dropwise an aqueous solution (10 ml) containing NaN_3 (0.052 g, 0.80 mmol) at room temperature. The color of the solution turned from yellow to pale pink. The mixture solution was stirred for 1 h during which time a pink precipitate of formed which was collected by filtration, washed with methanol, and dried in air. Single crystals of the title compound suitable for X-ray crystallography were obtained by layering of the aqueous solution of NaN_3 on the DMF/H_2O solution of $[Ni(C_{22}H_{34}N_6)Cl_2]$ for several days.

Refinement

All H atoms in the title compound were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.95 (ring H atoms) or 0.99 (open chain H atoms) Å and N—H distance of 0.93 Å, and with U_{iso}(H) values of 1.2 times the equivalent anisotropic displacement parameters of the parent C and N atoms.

Figures

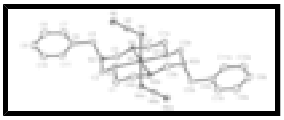

Fig. 1. Drawing of the molecular title compound at 50% probability. Atoms labeled with the suffix \`a' are at the symmetry position (-*x*+1, -*y*+2, -*z*).

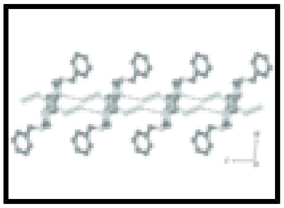

Fig. 2. Perspective view of the title compound showing a one-dimensional chain formed by N—H···N hydrogen-bonding interactions.

trans-Diazido(1,8-dibenzyl-1,3,6,8,10,13-hexaazacyclotetradecane)nickel(II)

Crystal data

$[Ni(N_3)_2(C_{22}H_{34}N_6)]$	$F_{000} = 556$
$M_r = 525.32$	$D_x = 1.431$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2820 reflections
a = 10.2150 (5) Å	θ = 2.0–28.3°
b = 15.8337 (9) Å	μ = 0.83 mm^{-1}
c = 7.5477 (4) Å	*T* = 173 (2) K
β = 92.817 (1)°	Block, violet
V = 1219.30 (11) Å^3	0.50 × 0.20 × 0.20 mm
Z = 2	

Data collection

Siemens SMART CCD diffractometer	2820 independent reflections
Radiation source: fine-focus sealed tube	2456 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
T = 173(2) K	$\theta_{max} = 28.3°$
φ and ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -13 \rightarrow 11$
$T_{min} = 0.733$, $T_{max} = 0.847$	$k = -17 \rightarrow 20$
7464 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained

$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 1.3575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.19$	$(\Delta/\sigma)_{max} < 0.001$
2820 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
160 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Ni1	0.5000	1.0000	0.0000	0.01702 (11)
N1	0.37796 (18)	0.89900 (12)	0.0535 (2)	0.0213 (4)
H1	0.3964	0.8821	0.1701	0.026*
N2	0.20050 (19)	0.99153 (13)	0.1463 (2)	0.0243 (4)
N3	0.38842 (18)	1.08789 (12)	0.1271 (2)	0.0204 (4)
H3	0.4085	1.0836	0.2483	0.024*
N4	0.6135 (2)	0.97898 (13)	0.2449 (2)	0.0254 (4)
N5	0.57874 (18)	0.93204 (12)	0.3563 (2)	0.0221 (4)
N6	0.5460 (2)	0.88500 (14)	0.4654 (3)	0.0315 (5)
C1	0.4175 (2)	0.83010 (15)	−0.0648 (3)	0.0268 (5)
H1A	0.3791	0.8395	−0.1862	0.032*
H1B	0.3852	0.7753	−0.0213	0.032*
C2	0.2360 (2)	0.92198 (15)	0.0349 (3)	0.0240 (5)
H2A	0.2139	0.9369	−0.0904	0.029*
H2B	0.1831	0.8720	0.0644	0.029*
C3	0.2447 (2)	1.07476 (15)	0.0969 (3)	0.0255 (5)
H3A	0.1979	1.1175	0.1658	0.031*
H3B	0.2208	1.0842	−0.0303	0.031*
C4	0.4333 (2)	1.17143 (15)	0.0675 (3)	0.0258 (5)
H4A	0.4050	1.2159	0.1495	0.031*
H4B	0.3948	1.1839	−0.0526	0.031*
C5	0.2105 (2)	0.97543 (16)	0.3375 (3)	0.0256 (5)
H5A	0.1964	1.0291	0.4012	0.031*
H5B	0.3002	0.9555	0.3709	0.031*
C6	0.1129 (2)	0.91069 (15)	0.3963 (3)	0.0230 (5)

C7	0.1481 (3)	0.85575 (17)	0.5339 (3)	0.0302 (5)
H7	0.2352	0.8566	0.5841	0.036*
C8	0.0580 (3)	0.79974 (17)	0.5991 (3)	0.0359 (6)
H8	0.0830	0.7633	0.6948	0.043*
C9	−0.0681 (3)	0.79699 (16)	0.5245 (3)	0.0322 (6)
H9	−0.1298	0.7582	0.5679	0.039*
C10	−0.1042 (2)	0.85094 (16)	0.3866 (3)	0.0288 (5)
H10	−0.1910	0.8491	0.3352	0.035*
C11	−0.0144 (2)	0.90777 (15)	0.3228 (3)	0.0254 (5)
H11	−0.0401	0.9448	0.2284	0.031*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0202 (2)	0.01814 (19)	0.01269 (18)	−0.00082 (17)	0.00037 (13)	0.00017 (16)
N1	0.0243 (10)	0.0257 (10)	0.0140 (8)	−0.0017 (8)	0.0009 (7)	−0.0004 (7)
N2	0.0238 (9)	0.0284 (11)	0.0210 (9)	−0.0012 (8)	0.0040 (7)	−0.0004 (8)
N3	0.0245 (10)	0.0224 (10)	0.0144 (8)	−0.0005 (8)	0.0011 (7)	0.0005 (7)
N4	0.0283 (10)	0.0301 (11)	0.0174 (9)	0.0000 (8)	−0.0017 (8)	0.0042 (8)
N5	0.0216 (10)	0.0257 (10)	0.0187 (9)	0.0035 (8)	−0.0029 (7)	−0.0055 (8)
N6	0.0390 (12)	0.0323 (12)	0.0232 (10)	−0.0042 (10)	0.0017 (9)	0.0044 (9)
C1	0.0291 (13)	0.0229 (12)	0.0287 (12)	−0.0041 (10)	0.0040 (9)	−0.0039 (9)
C2	0.0238 (11)	0.0306 (13)	0.0175 (10)	−0.0050 (10)	0.0010 (8)	−0.0025 (9)
C3	0.0245 (12)	0.0289 (13)	0.0230 (11)	0.0022 (10)	0.0005 (9)	0.0011 (9)
C4	0.0310 (13)	0.0199 (11)	0.0267 (12)	0.0019 (10)	0.0029 (9)	−0.0011 (9)
C5	0.0249 (12)	0.0330 (13)	0.0190 (11)	−0.0031 (10)	0.0019 (9)	−0.0041 (9)
C6	0.0263 (12)	0.0256 (12)	0.0174 (10)	0.0019 (10)	0.0038 (8)	−0.0053 (9)
C7	0.0308 (13)	0.0342 (14)	0.0249 (12)	0.0026 (11)	−0.0037 (10)	0.0015 (10)
C8	0.0493 (16)	0.0319 (14)	0.0263 (13)	0.0015 (12)	0.0006 (11)	0.0077 (10)
C9	0.0386 (15)	0.0278 (13)	0.0312 (13)	−0.0065 (11)	0.0124 (11)	−0.0010 (10)
C10	0.0225 (12)	0.0343 (14)	0.0300 (12)	0.0001 (10)	0.0060 (9)	−0.0056 (10)
C11	0.0238 (12)	0.0283 (12)	0.0243 (11)	0.0053 (10)	0.0033 (9)	0.0008 (9)

Geometric parameters (Å, °)

Ni1—N3	2.0650 (18)	C2—H2B	0.9900
Ni1—N3^{i}	2.0650 (18)	C3—H3A	0.9900
Ni1—N1	2.0795 (19)	C3—H3B	0.9900
Ni1—N1^{i}	2.0795 (19)	C4—C1^{i}	1.526 (3)
Ni1—N4^{i}	2.1592 (19)	C4—H4A	0.9900
Ni1—N4	2.1592 (19)	C4—H4B	0.9900
N1—C1	1.479 (3)	C5—C6	1.512 (3)
N1—C2	1.495 (3)	C5—H5A	0.9900
N1—H1	0.9300	C5—H5B	0.9900
N2—C2	1.443 (3)	C6—C7	1.389 (3)
N2—C3	1.448 (3)	C6—C11	1.389 (3)
N2—C5	1.464 (3)	C7—C8	1.385 (4)
N3—C4	1.477 (3)	C7—H7	0.9500

N3—C3	1.489 (3)	C8—C9	1.381 (4)
N3—H3	0.9300	C8—H8	0.9500
N4—N5	1.189 (3)	C9—C10	1.383 (4)
N5—N6	1.171 (3)	C9—H9	0.9500
C1—C4^{i}	1.526 (3)	C10—C11	1.388 (3)
C1—H1A	0.9900	C10—H10	0.9500
C1—H1B	0.9900	C11—H11	0.9500
C2—H2A	0.9900		

N3—Ni1—N3^{i}	180.0	N1—C2—H2A	108.8
N3—Ni1—N1	94.48 (7)	N2—C2—H2B	108.8
N3^{i}—Ni1—N1	85.52 (7)	N1—C2—H2B	108.8
N3—Ni1—N1^{i}	85.52 (7)	H2A—C2—H2B	107.7
N3^{i}—Ni1—N1^{i}	94.48 (7)	N2—C3—N3	113.92 (19)
N1—Ni1—N1^{i}	180.0	N2—C3—H3A	108.8
N3—Ni1—N4^{i}	90.49 (7)	N3—C3—H3A	108.8
N3^{i}—Ni1—N4^{i}	89.51 (7)	N2—C3—H3B	108.8
N1—Ni1—N4^{i}	89.03 (7)	N3—C3—H3B	108.8
N1^{i}—Ni1—N4^{i}	90.97 (7)	H3A—C3—H3B	107.7
N3—Ni1—N4	89.51 (7)	N3—C4—C1^{i}	108.36 (19)
N3^{i}—Ni1—N4	90.49 (7)	N3—C4—H4A	110.0
N1—Ni1—N4	90.97 (7)	C1^{i}—C4—H4A	110.0
N1^{i}—Ni1—N4	89.03 (7)	N3—C4—H4B	110.0
N4^{i}—Ni1—N4	180.00 (10)	C1^{i}—C4—H4B	110.0
C1—N1—C2	114.54 (17)	H4A—C4—H4B	108.4
C1—N1—Ni1	105.41 (13)	N2—C5—C6	113.11 (19)
C2—N1—Ni1	112.49 (14)	N2—C5—H5A	109.0
C1—N1—H1	108.1	C6—C5—H5A	109.0
C2—N1—H1	108.1	N2—C5—H5B	109.0
Ni1—N1—H1	108.1	C6—C5—H5B	109.0
C2—N2—C3	117.02 (18)	H5A—C5—H5B	107.8
C2—N2—C5	115.73 (19)	C7—C6—C11	118.8 (2)
C3—N2—C5	113.87 (19)	C7—C6—C5	119.6 (2)
C4—N3—C3	113.36 (18)	C11—C6—C5	121.5 (2)
C4—N3—Ni1	105.99 (13)	C8—C7—C6	121.0 (2)
C3—N3—Ni1	113.40 (14)	C8—C7—H7	119.5
C4—N3—H3	108.0	C6—C7—H7	119.5
C3—N3—H3	108.0	C9—C8—C7	119.9 (2)
Ni1—N3—H3	108.0	C9—C8—H8	120.1
N5—N4—Ni1	122.34 (16)	C7—C8—H8	120.1
N6—N5—N4	179.0 (2)	C8—C9—C10	119.7 (2)
N1—C1—C4^{i}	108.84 (19)	C8—C9—H9	120.1
N1—C1—H1A	109.9	C10—C9—H9	120.1
C4^{i}—C1—H1A	109.9	C9—C10—C11	120.4 (2)
N1—C1—H1B	109.9	C9—C10—H10	119.8
C4^{i}—C1—H1B	109.9	C11—C10—H10	119.8

H1A—C1—H1B	108.3	C10—C11—C6	120.3 (2)
N2—C2—N1	113.67 (18)	C10—C11—H11	119.9
N2—C2—H2A	108.8	C6—C11—H11	119.9
N3—Ni1—N1—C1	165.54 (14)	C3—N2—C2—N1	72.2 (3)
N3$^{\rm i}$—Ni1—N1—C1	−14.46 (14)	C5—N2—C2—N1	−66.4 (2)
N4$^{\rm i}$—Ni1—N1—C1	75.12 (14)	C1—N1—C2—N2	−177.66 (18)
N4—Ni1—N1—C1	−104.88 (14)	Ni1—N1—C2—N2	−57.3 (2)
N3—Ni1—N1—C2	40.06 (14)	C2—N2—C3—N3	−71.2 (3)
N3$^{\rm i}$—Ni1—N1—C2	−139.94 (14)	C5—N2—C3—N3	68.1 (2)
N4$^{\rm i}$—Ni1—N1—C2	−50.35 (14)	C4—N3—C3—N2	176.97 (18)
N4—Ni1—N1—C2	129.65 (14)	Ni1—N3—C3—N2	56.1 (2)
N1—Ni1—N3—C4	−164.63 (14)	C3—N3—C4—C1$^{\rm i}$	−166.97 (18)
N1$^{\rm i}$—Ni1—N3—C4	15.37 (14)	Ni1—N3—C4—C1$^{\rm i}$	−41.96 (19)
N4$^{\rm i}$—Ni1—N3—C4	−75.56 (14)	C2—N2—C5—C6	−66.6 (3)
N4—Ni1—N3—C4	104.44 (14)	C3—N2—C5—C6	153.5 (2)
N1—Ni1—N3—C3	−39.65 (15)	N2—C5—C6—C7	145.0 (2)
N1$^{\rm i}$—Ni1—N3—C3	140.35 (15)	N2—C5—C6—C11	−38.7 (3)
N4$^{\rm i}$—Ni1—N3—C3	49.42 (15)	C11—C6—C7—C8	−0.9 (4)
N4—Ni1—N3—C3	−130.58 (15)	C5—C6—C7—C8	175.5 (2)
N3—Ni1—N4—N5	83.63 (19)	C6—C7—C8—C9	1.2 (4)
N3$^{\rm i}$—Ni1—N4—N5	−96.37 (19)	C7—C8—C9—C10	−0.8 (4)
N1—Ni1—N4—N5	−10.84 (19)	C8—C9—C10—C11	0.0 (4)
N1$^{\rm i}$—Ni1—N4—N5	169.16 (19)	C9—C10—C11—C6	0.3 (4)
C2—N1—C1—C4$^{\rm i}$	165.51 (18)	C7—C6—C11—C10	0.1 (3)
Ni1—N1—C1—C4$^{\rm i}$	41.3 (2)	C5—C6—C11—C10	−176.2 (2)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N6$^{\rm ii}$	0.93	2.24	3.145 (3)	163

Symmetry codes: (ii) −*x*+1, −*y*+2, −*z*+1.

Fig. 1

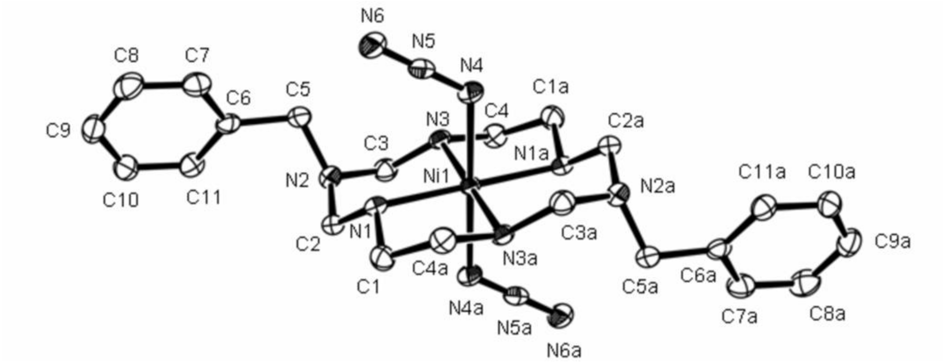
N6
N5
N4
C8
C7
C6
C5
C1a
C9
N3
C4
C2a
C3
N1a
N2
Ni1
N2a
C11a
C10a
C10
C11
C2
N1
C3a
C9a
C1
C4a
N3a
C5a
C6a
N4a
N5a
C7a
C8a
N6a

Fig. 2