Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(dicyanamido-κN)tetrakis(pyridazine-κN)nickel(II)

Susanne Wöhlert,[a] Mario Wriedt,[b] Inke Jess[a] and Christian Näther[a]*

[a]Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany, and [b]Departement of Chemistry, Texas A&M University, College Station, Texas 77843, USA
Correspondence e-mail: swoehlert@ac.uni-kiel.de

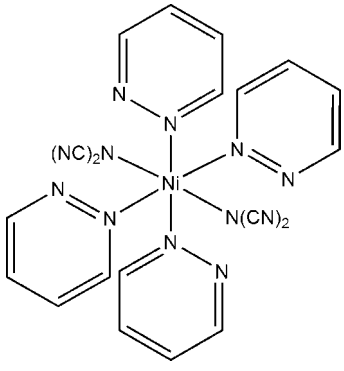
Received 23 April 2012; accepted 24 April 2012

Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.006 Å; R factor = 0.048; wR factor = 0.097; data-to-parameter ratio = 13.3.

Reaction of nickel(II) chloride with sodium dicyanamide and pyridazine leads to single crystals of the title compound, $[Ni\{N(CN)_2\}_2(C_4H_4N_2)_4]$, in which the Ni^{II} cation is octahedrally coordinated by two dicyanamide anions and four pyridazine ligands into a discrete complex that is located on a center of inversion.

Related literature

For the synthesis, structures and properties of dicyanamide coordination compounds, see: Wriedt & Näther (2011).



Experimental

Crystal data

$[Ni(C_2N_3)_2(C_4H_4N_2)_4]$
M_r = 511.18
Triclinic, $P\overline{1}$
a = 8.1796 (12) Å
b = 8.4125 (12) Å
c = 8.9643 (11) Å
α = 81.364 (16)°
β = 66.027 (15)°
γ = 84.879 (17)°
V = 556.97 (13) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.91 mm^{-1}
T = 170 K
0.10 × 0.08 × 0.06 mm

Data collection

Stoe IPDS-1 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.783, T_{max} = 0.927
4159 measured reflections
2142 independent reflections
1582 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.097
S = 1.01
2142 reflections
161 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2008); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2011).; software used to prepare material for publication: *XCIF* in *SHELXTL*.

We gratefully acknowledge financial support by the DFG (project No. NA 720/3–1) and the State of Schleswig–Holstein. We thank Professor Dr Wolfgang Bensch for access to his experimental facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5900).

References

Brandenburg, K. (2011). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (2008). *X-AREA*, *X-RED32* and *X-SHAPE*. Stoe & Cie, Darmstadt, Germany.
Wriedt, M. & Näther, C. (2011). *Dalton Trans.* **40**, 886–898.

supplementary materials

Acta Cryst. (2012). E**68**, m696 [doi:10.1107/S1600536812018363]

Bis(dicyanamido-κN)tetrakis(pyridazine-κN)nickel(II)

Susanne Wöhlert, Mario Wriedt, Inke Jess and Christian Näther

Comment

Recently we have reported on the synthesis and characterization of paramagnetic transition metal complexes with dicyanamide as anion (Wriedt & Näther, 2011). As a part of our ongoing study in this field the crystal structure of the title compound was determined. The asymmetric unit of the title compound consits of one nickel(II) cation which is located on a center of inversion as well as one dicyanamide anion and two pyridazine ligands both in general position (Fig. 1). In the crystal structure discrete complexes are formed, in which each nickel(II) cation is coordinated by two terminal coordinated dicyanamide anions and four pyridazine ligands in a slightly distorted octahedral geometry. The Ni—N distances are in the range of 2.058 (3) Å to 2.147 (3) Å with the longer distances to the pyridazine ligands. The shortest intermolecular Ni···Ni distance amounts to 8.1796 Å.

Experimental

Nickel(II) chloride hexahydrate ($NiCl_2x6H_2O$), sodium dicyanamide ($NaN(CN)_2$) and pyridazine were obtained from Alfa Aesar. All chemicals were used without further purification. 0.125 mmol (29.7 mg) $NiCl_2x6H_2O$, 0.25 mmol (22.3 mg) $NaN(CN)_2$ were reacted in 1.5 ml pyridazine. Green single crystals of the title compound were obtained after one week.

Refinement

All H atoms were located in difference map but were positioned with idealized geometry and were refined isotropically with $U_{eq}(H) = 1.2\ U_{eq}(C)$ of the parent atom using a riding model with C—H = 0.95 Å.

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2008); cell refinement: *X-AREA* (Stoe & Cie, 2008); data reduction: *X-AREA* (Stoe & Cie, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2011).; software used to prepare material for publication: XCIF in *SHELXTL* (Sheldrick, 2008).

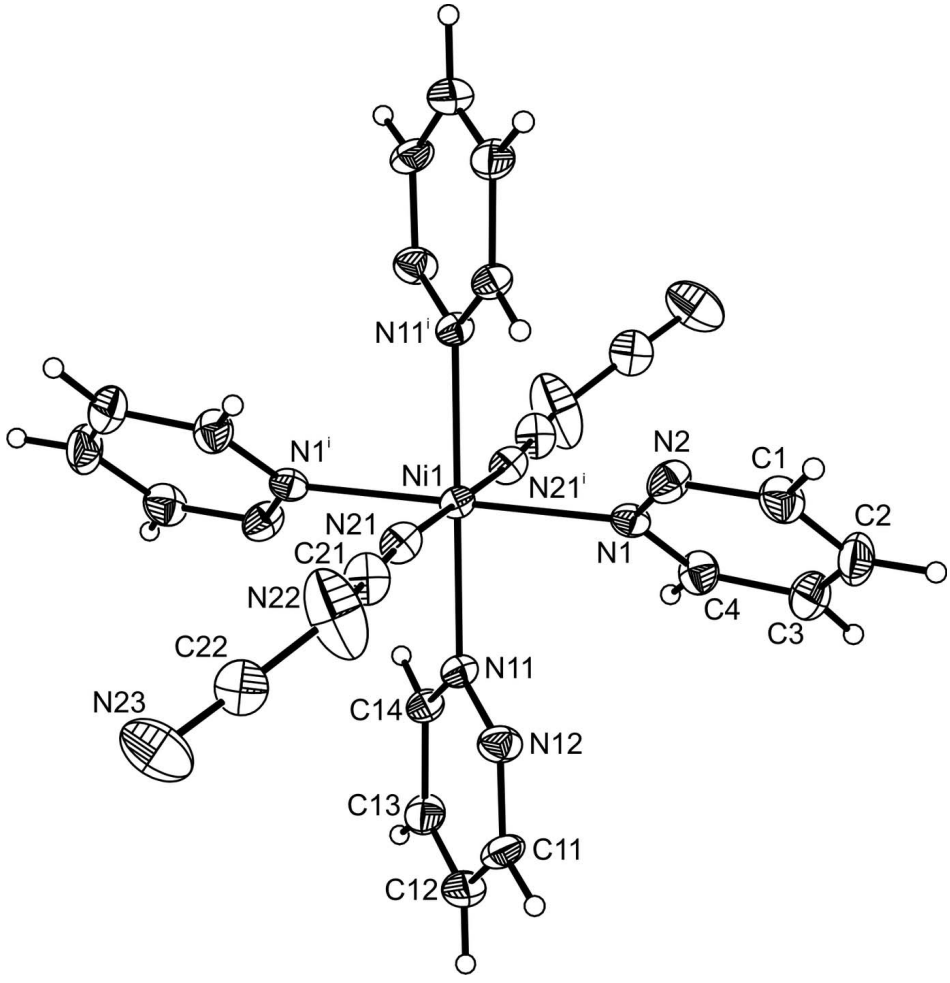


Figure 1

Crystal structure of the title compund with labelling and displacement ellipsoids drawn at the 50% probability level. Symmetry code: i = -*x*, -*y* + 1, -*z* + 1.

Bis(dicyanamido-*κN*)tetrakis(pyridazine-*κN*)nickel(II)

Crystal data

$[Ni(C_2N_3)_2(C_4H_4N_2)_4]$
M_r = 511.18
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 8.1796 (12) Å
b = 8.4125 (12) Å
c = 8.9643 (11) Å
α = 81.364 (16)°
β = 66.027 (15)°
γ = 84.879 (17)°
V = 556.97 (13) Å^3
Z = 1
F(000) = 262
D_x = 1.524 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 4159 reflections
θ = 2.5–26.0°
μ = 0.91 mm^{-1}
T = 170 K
Block, green
0.10 × 0.08 × 0.06 mm

Data collection

Stoe IPDS-1 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi scan
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.783, T_{max} = 0.927
4159 measured reflections
2142 independent reflections
1582 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 26.0°, θ_{min} = 2.5°
h = −10→10
k = −10→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.097
S = 1.01
2142 reflections
161 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0348P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.025 (4)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.0000	0.5000	0.5000	0.0161 (2)
N1	0.1088 (4)	0.7246 (3)	0.4952 (4)	0.0190 (6)
N2	0.0726 (4)	0.7802 (4)	0.6384 (4)	0.0249 (7)
C1	0.1491 (5)	0.9151 (5)	0.6353 (5)	0.0295 (9)
H1	0.1204	0.9557	0.7366	0.035*
C2	0.2687 (6)	0.9998 (5)	0.4919 (6)	0.0336 (10)
H2	0.3228	1.0941	0.4945	0.040*
C3	0.3045 (6)	0.9406 (5)	0.3470 (5)	0.0333 (9)
H3	0.3853	0.9920	0.2449	0.040*
C4	0.2185 (5)	0.8030 (4)	0.3549 (5)	0.0254 (8)
H4	0.2391	0.7628	0.2550	0.031*
N11	0.2622 (4)	0.4283 (3)	0.3430 (4)	0.0184 (6)
N12	0.3917 (4)	0.4485 (4)	0.3946 (4)	0.0250 (7)
C11	0.5593 (5)	0.4051 (5)	0.3036 (5)	0.0264 (8)
H11	0.6503	0.4227	0.3390	0.032*

C12	0.6091 (5)	0.3350 (5)	0.1588 (5)	0.0295 (9)
H12	0.7298	0.3030	0.0984	0.035*
C13	0.4777 (5)	0.3144 (4)	0.1079 (5)	0.0273 (8)
H13	0.5035	0.2669	0.0107	0.033*
C14	0.3019 (4)	0.3658 (4)	0.2037 (4)	0.0201 (7)
H14	0.2092	0.3556	0.1680	0.024*
N21	0.0470 (4)	0.4009 (4)	0.7037 (4)	0.0223 (7)
C21	0.0896 (5)	0.3404 (4)	0.8065 (5)	0.0263 (8)
N22	0.1310 (7)	0.2874 (5)	0.9320 (5)	0.0581 (13)
C22	0.1948 (5)	0.1418 (5)	0.9515 (5)	0.0300 (9)
N23	0.2467 (6)	0.0160 (5)	0.9879 (5)	0.0480 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0151 (4)	0.0195 (4)	0.0144 (4)	−0.0010 (3)	−0.0066 (3)	−0.0020 (3)
N1	0.0177 (14)	0.0215 (15)	0.0193 (16)	0.0027 (12)	−0.0090 (12)	−0.0047 (12)
N2	0.0227 (16)	0.0314 (17)	0.0223 (17)	−0.0058 (13)	−0.0082 (13)	−0.0083 (13)
C1	0.032 (2)	0.029 (2)	0.033 (2)	0.0018 (17)	−0.0151 (18)	−0.0137 (17)
C2	0.037 (2)	0.0228 (19)	0.047 (3)	−0.0085 (17)	−0.021 (2)	−0.0029 (18)
C3	0.033 (2)	0.028 (2)	0.032 (2)	−0.0079 (17)	−0.0069 (18)	0.0039 (17)
C4	0.029 (2)	0.0245 (18)	0.021 (2)	−0.0027 (16)	−0.0087 (16)	0.0008 (15)
N11	0.0157 (15)	0.0224 (15)	0.0174 (16)	−0.0012 (12)	−0.0062 (12)	−0.0043 (12)
N12	0.0211 (16)	0.0290 (17)	0.0291 (18)	0.0023 (13)	−0.0131 (14)	−0.0083 (14)
C11	0.0145 (17)	0.032 (2)	0.033 (2)	−0.0030 (15)	−0.0085 (16)	−0.0050 (17)
C12	0.0207 (19)	0.030 (2)	0.031 (2)	0.0018 (16)	−0.0031 (16)	−0.0047 (17)
C13	0.0249 (19)	0.028 (2)	0.021 (2)	−0.0026 (16)	0.0003 (15)	−0.0065 (16)
C14	0.0172 (17)	0.0242 (18)	0.0162 (19)	−0.0016 (14)	−0.0036 (14)	−0.0025 (14)
N21	0.0219 (16)	0.0263 (16)	0.0191 (18)	−0.0035 (13)	−0.0079 (14)	−0.0034 (13)
C21	0.035 (2)	0.028 (2)	0.018 (2)	−0.0030 (16)	−0.0129 (17)	−0.0034 (15)
N22	0.113 (4)	0.037 (2)	0.053 (3)	0.007 (2)	−0.065 (3)	−0.0054 (19)
C22	0.037 (2)	0.035 (2)	0.024 (2)	−0.0021 (19)	−0.0190 (18)	−0.0022 (17)
N23	0.059 (3)	0.050 (2)	0.043 (2)	0.020 (2)	−0.030 (2)	−0.0134 (19)

Geometric parameters (Å, º)

Ni1—N21	2.058 (3)	C4—H4	0.9500
Ni1—N21^{i}	2.058 (3)	N11—C14	1.333 (4)
Ni1—N11^{i}	2.125 (3)	N11—N12	1.349 (4)
Ni1—N11	2.125 (3)	N12—C11	1.330 (5)
Ni1—N1^{i}	2.147 (3)	C11—C12	1.399 (5)
Ni1—N1	2.147 (3)	C11—H11	0.9500
N1—C4	1.327 (5)	C12—C13	1.359 (5)
N1—N2	1.342 (4)	C12—H12	0.9500
N2—C1	1.336 (5)	C13—C14	1.410 (5)
C1—C2	1.394 (6)	C13—H13	0.9500
C1—H1	0.9500	C14—H14	0.9500
C2—C3	1.372 (6)	N21—C21	1.148 (5)
C2—H2	0.9500	C21—N22	1.308 (5)
C3—C4	1.385 (5)	N22—C22	1.304 (6)

C3—H3	0.9500	C22—N23	1.155 (6)
N21—Ni1—N21^{i}	180.00 (8)	C2—C3—H3	121.2
N21—Ni1—N11^{i}	89.44 (11)	C4—C3—H3	121.2
N21^{i}—Ni1—N11^{i}	90.56 (11)	N1—C4—C3	123.2 (3)
N21—Ni1—N11	90.56 (11)	N1—C4—H4	118.4
N21^{i}—Ni1—N11	89.44 (11)	C3—C4—H4	118.4
N11^{i}—Ni1—N11	180.0	C14—N11—N12	120.5 (3)
N21—Ni1—N1^{i}	88.23 (11)	C14—N11—Ni1	124.5 (2)
N21^{i}—Ni1—N1^{i}	91.77 (11)	N12—N11—Ni1	115.0 (2)
N11^{i}—Ni1—N1^{i}	87.48 (11)	C11—N12—N11	118.6 (3)
N11—Ni1—N1^{i}	92.52 (10)	N12—C11—C12	123.7 (3)
N21—Ni1—N1	91.77 (11)	N12—C11—H11	118.1
N21^{i}—Ni1—N1	88.23 (11)	C12—C11—H11	118.1
N11^{i}—Ni1—N1	92.52 (10)	C13—C12—C11	117.3 (3)
N11—Ni1—N1	87.48 (11)	C13—C12—H12	121.3
N1^{i}—Ni1—N1	180.0	C11—C12—H12	121.3
C4—N1—N2	120.1 (3)	C12—C13—C14	117.9 (3)
C4—N1—Ni1	121.1 (2)	C12—C13—H13	121.0
N2—N1—Ni1	118.7 (2)	C14—C13—H13	121.0
C1—N2—N1	118.4 (3)	N11—C14—C13	121.9 (3)
N2—C1—C2	123.8 (3)	N11—C14—H14	119.1
N2—C1—H1	118.1	C13—C14—H14	119.1
C2—C1—H1	118.1	C21—N21—Ni1	173.1 (3)
C3—C2—C1	116.9 (3)	N21—C21—N22	173.1 (4)
C3—C2—H2	121.6	C22—N22—C21	122.1 (4)
C1—C2—H2	121.6	N23—C22—N22	171.8 (4)
C2—C3—C4	117.6 (4)		

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