Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Poly[aquabis(μ-formato-κ²O:O′)(μ-pyrazine-κ²N:N′)nickel(II)]

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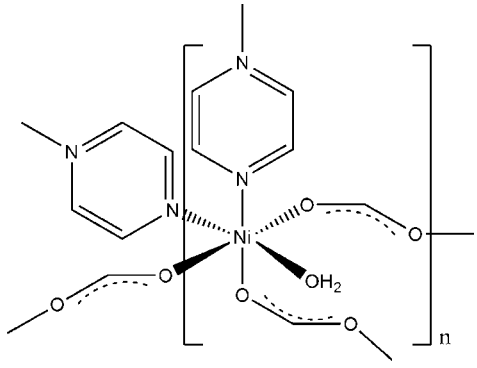
Received 23 March 2011; accepted 30 March 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.059; data-to-parameter ratio = 17.9.

In the title compound, $[Ni(CHO_2)_2(C_4H_4N_2)(H_2O)]$, the nickel(II) cations are coordinated by three *O*-bonded-formato anions, two *N*-bonded-pyrazine ligands and one water molecule in an octahedral coordination mode. The nickel(II) cations are connected by μ-1,3-bridging formato anions and *N*,*N*′-bridging pyrazine ligands into a three dimensional coordination network. The asymmetric unit consists of one nickel(II) cation, one water molecule and two crystallographically independent formato anions in general positions as well as two crystallographically independent pyrazine ligands, which are located on centers of inversion.

Related literature

For background of this work, see: Boeckmann & Näther (2010), Wriedt *et al.* (2009); Boeckmann *et al.* (2010). For a related structure, see: Manson *et al.* (2003). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$[Ni(CHO_2)_2(C_4H_4N_2)(H_2O)]$
M_r = 246.85
Monoclinic, $P2_1/c$
a = 7.8169 (4) Å
b = 7.0077 (3) Å
c = 15.6586 (7) Å
β = 98.971 (4)°
V = 847.26 (7) Å³
Z = 4
Mo $K\alpha$ radiation
μ = 2.29 mm⁻¹
T = 293 K
0.19 × 0.15 × 0.12 mm

Data collection

Stoe IPDS-2 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe, 2008)
T_{min} = 0.658, T_{max} = 0.770
15596 measured reflections
2291 independent reflections
2091 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.059
S = 1.10
2291 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å⁻³
$\Delta\rho_{min}$ = −0.30 e Å⁻³

Data collection: *X-AREA* (Stoe, 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); software used to prepare material for publication: *XP* in *SHELXTL* and *DIAMOND* (Brandenburg, 2011).

We gratefully acknowledge financial support by the DFG (project number NA 720/3-1) and the State of Schleswig-Holstein. We thank Professor Dr Bensch for access to his experimental facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5501).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Boeckmann, J. & Näther, C. (2010). *Dalton Trans.* **39**, 1119-1126.
Boeckmann, J., Wriedt, M. & Näther, C. (2010). *Eur. J. Inorg. Chem.* pp. 1820-1828.
Brandenburg, K. (2011). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Manson, J. L., Lecher, J. G., Gu, J., Geiser, U., Schlueter, J. A., Henning, R., Wang, X., Schultz, A. J., Koo, H.-J. & Whangbo, M.-H. (2003). *Dalton Trans.* pp. 2905–2911.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe (2008). *X-AREA*, *X-RED32* and *X-SHAPE*. Stoe & Cie, Darmstadt, Germany.
Wriedt, M., Jess, I. & Näther, C. (2009). *Eur. J. Inorg. Chem.* pp. 1406–1413.

supporting information

Acta Cryst. (2011). E**67**, m532 [doi:10.1107/S1600536811011913]

Poly[aquabis(*μ*-formato-κ^2O:O')(*μ*-pyrazine-κ^2N:N')nickel(II)]

Susanne Wöhlert, Mario Wriedt, Inke Jess and Christian Näther

S1. Comment

In our recent work on the synthesis, structures and properties of new coordination polymers based on paramagnetic transition metal, small-sized anions and *N*-donor ligands, we have shown that new ligand-deficient coordination polymers based on transition metal thiocyanates and selenocyanates can be prepared by thermal decomposition reactions (Wriedt, Jess & Näther, 2009 and Boeckmann & Näther, 2010). Later we have shown that also metal formates can be prepared by this route (Boeckmann, Wriedt & Näther, 2010). Within this project we tried to prepare new ligand-rich precursor compounds based on nickel(II) formate and pyrazine which resulted in the formation of the title compound that were identified by single crystal X-ray diffraction.

In the crystal structure of the title compound, each nickel(II) cation is coordinated by three bridging formato anions, two bridging pyrazine ligands and one water molecule (Fig. 1). The NiO_4N_2 octahedron is slightly distorted with Ni—OCHO distances between 2.0378 (11) Å and 2.0643 (12) Å and one Ni—OH_2 distance of 2.0420 (11) Å as well as two long Ni—N distances of 2.1066 (13) Å and 2.1171 (12) Å. The angles around the metal atoms range from 84.14 (5)° to 95.94 (5)° and from 173.18 (5)° to 179.19 (5)°. The nickel(II) cations are connected via *μ*-1,3 bridging formato anions into two dimensional $Ni(O_2CHO)_2$ layers that are further linked by the pyrazine ligands into a 3D coordination network (Fig. 2). The Ni—Ni distances between next neighboured Ni atoms ranges from 6.9770 (4) Å to 7.0689 (4) Å.

It must be noted that according to a search in the CCDC database (ConQuest Ver.1.12.) (Allen, 2002) compounds based on nickel(II) formate and pyrazine are unknown but with copper(II) formate one strcuture is reported (Manson *et al.*, 2003).

S2. Experimental

Nickel formate dihydrate [$Ni(CHO_2)_2.H_2O$] and pyrazine were obtained from Alfa Aesar. All chemicals were used without further purification. 0.25 mmol (46 mg) $Ni(CHO_2)_2.H_2O$ and 0.5 mmol (40 mg) pyrazine were reacted in 2 ml water. Light blue block-shaped single crystals of the title compound were obtained after a few days at room temperature.

S3. Refinement

The C-H H atoms were positioned with idealized geometry and were refined isotropic with $U_{iso}(H) = 1.2U_{eq}(C)$ and C—H distances of 0.93 Å using a riding model. The O-H H atoms were located in difference map, their bond lengths were set to 0.82 Å and afterwards they were refined isotropic with $U_{iso}(H) = 1.5U_{eq}(O)$ using a riding model.

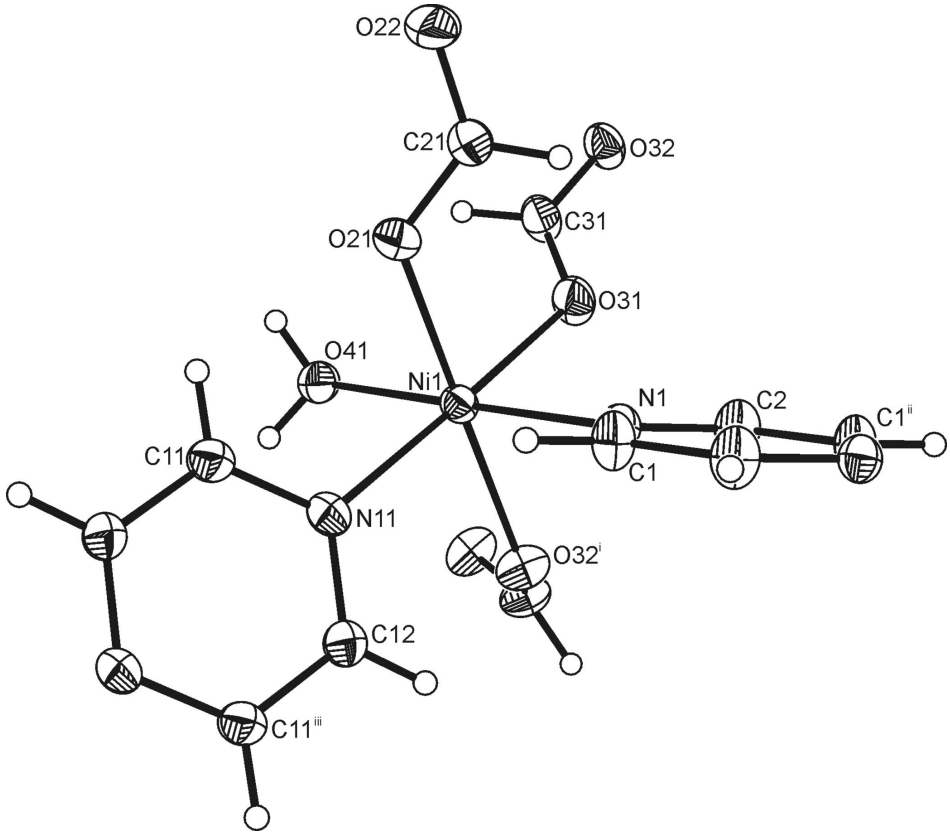


Figure 1

Crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50 % probability level. Symmetry codes: i = -x+1, y+1/2 i = -x+1,y+1/2,-z+3/2; ii = -x+1,-y,-z+1; iii = -x,-y+1,-z+1.

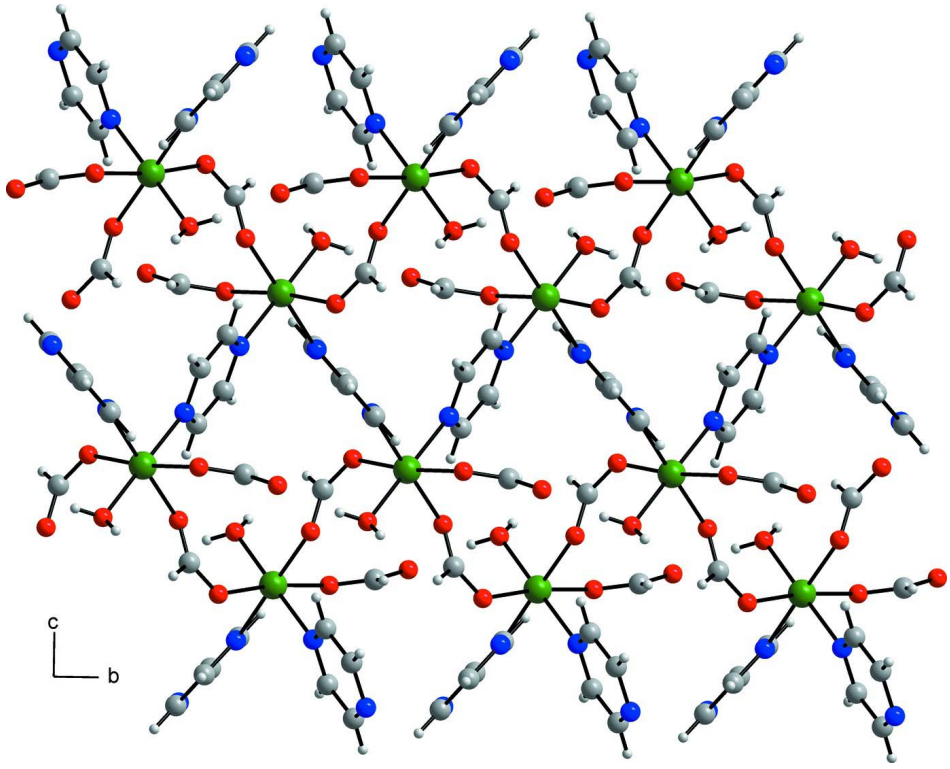


Figure 2

Crystal structure of the title compound with view along the crystallographic *a*-axis.

Poly[aquabis(*μ*-formato-*κ*^{2}*O*:*O*′)(*μ*- pyrazine-*κ*^{2}*N*:*N*′)nickel(II)]

Crystal data

$[Ni(CHO_2)_2(C_4H_4N_2)(H_2O)]$
$M_r = 246.85$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.8169$ (4) Å
$b = 7.0077$ (3) Å
$c = 15.6586$ (7) Å
$\beta = 98.971$ (4)°
$V = 847.26$ (7) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.935$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 15596 reflections
$\theta = 2.6$–29.3°
$\mu = 2.29$ mm^{-1}
$T = 293$ K
Block, light blue
0.19 × 0.15 × 0.12 mm

Data collection

Stoe IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe, 2008)
$T_{min} = 0.658$, $T_{max} = 0.770$
15596 measured reflections
2291 independent reflections
2091 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 29.3°$, $\theta_{min} = 2.6°$
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 9$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.059$
$S = 1.10$
2291 reflections
128 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
$w = 1/[\sigma^2(F_o^2) + (0.025P)^2 + 0.4622P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0158 (13)

Special details

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

Refinement. 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.30005 (2)	0.25657 (3)	0.649349 (11)	0.01731 (8)
N1	0.41659 (17)	0.10169 (18)	0.55756 (8)	0.0205 (2)
C1	0.3337 (2)	0.0453 (2)	0.48084 (10)	0.0250 (3)
H1	0.2169	0.0747	0.4654	0.030*
C2	0.5831 (2)	0.0558 (2)	0.57642 (10)	0.0257 (3)
H2	0.6452	0.0925	0.6294	0.031*
N11	0.12229 (17)	0.39364 (18)	0.55394 (8)	0.0209 (2)
C11	−0.0463 (2)	0.3831 (2)	0.55924 (11)	0.0241 (3)
H11	−0.0827	0.3027	0.6001	0.029*
C12	0.1684 (2)	0.5112 (2)	0.49450 (10)	0.0234 (3)
H12	0.2847	0.5224	0.4891	0.028*
O21	0.11734 (15)	0.04433 (16)	0.65066 (8)	0.0275 (3)
O22	0.0548 (2)	−0.25397 (17)	0.67969 (12)	0.0434 (4)
C21	0.1545 (2)	−0.1280 (2)	0.66185 (12)	0.0279 (3)
H21	0.2693	−0.1604	0.6602	0.042*
O31	0.46637 (15)	0.11818 (18)	0.74213 (8)	0.0282 (3)
O32	0.51992 (15)	−0.03704 (17)	0.86756 (7)	0.0260 (2)
C31	0.4364 (2)	0.0746 (2)	0.81445 (11)	0.0258 (3)
H31	0.3479	0.1347	0.8375	0.039*
O41	0.18980 (15)	0.40396 (16)	0.73925 (7)	0.0241 (2)
H1O	0.1126	0.3547	0.7617	0.036*
H2O	0.1500	0.5093	0.7246	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01857 (11)	0.01683 (11)	0.01653 (11)	0.00177 (7)	0.00272 (7)	0.00092 (7)
N1	0.0220 (6)	0.0203 (6)	0.0198 (6)	0.0012 (5)	0.0049 (5)	−0.0023 (5)
C1	0.0200 (7)	0.0306 (8)	0.0238 (7)	0.0045 (6)	0.0020 (6)	−0.0043 (6)
C2	0.0230 (7)	0.0321 (8)	0.0212 (7)	0.0019 (6)	0.0007 (6)	−0.0070 (6)
N11	0.0214 (6)	0.0199 (6)	0.0206 (6)	0.0019 (5)	0.0007 (5)	0.0018 (5)
C11	0.0233 (7)	0.0245 (7)	0.0243 (7)	0.0000 (6)	0.0030 (6)	0.0059 (6)
C12	0.0194 (7)	0.0258 (7)	0.0248 (7)	0.0013 (6)	0.0029 (6)	0.0035 (6)
O21	0.0261 (6)	0.0184 (5)	0.0390 (7)	−0.0001 (4)	0.0077 (5)	0.0045 (5)
O22	0.0477 (8)	0.0184 (6)	0.0720 (11)	0.0009 (5)	0.0340 (8)	0.0041 (6)
C21	0.0283 (8)	0.0206 (7)	0.0372 (9)	0.0027 (6)	0.0128 (7)	0.0011 (6)
O31	0.0273 (6)	0.0356 (6)	0.0218 (5)	0.0097 (5)	0.0043 (5)	0.0080 (5)
O32	0.0296 (6)	0.0276 (6)	0.0208 (5)	0.0089 (5)	0.0039 (4)	0.0040 (4)
C31	0.0286 (8)	0.0266 (8)	0.0227 (7)	0.0087 (6)	0.0054 (6)	0.0020 (6)
O41	0.0264 (6)	0.0208 (5)	0.0271 (6)	0.0015 (4)	0.0101 (4)	−0.0002 (4)

Geometric parameters (Å, º)

Ni1—O31	2.0378 (11)	C11—C12^{iii}	1.385 (2)
Ni1—O41	2.0420 (11)	C11—H11	0.9300
Ni1—O32^{i}	2.0636 (11)	C12—C11^{iii}	1.385 (2)
Ni1—O21	2.0643 (12)	C12—H12	0.9300
Ni1—N11	2.1066 (13)	O21—C21	1.2479 (19)
Ni1—N1	2.1171 (12)	O22—C21	1.238 (2)
N1—C2	1.328 (2)	C21—H21	0.9300
N1—C1	1.333 (2)	O31—C31	1.230 (2)
C1—C2^{ii}	1.382 (2)	O32—C31	1.2492 (19)
C1—H1	0.9300	O32—Ni1^{iv}	2.0636 (11)
C2—C1^{ii}	1.382 (2)	C31—H31	0.9299
C2—H2	0.9300	O41—H1O	0.8200
N11—C12	1.334 (2)	O41—H2O	0.8200
N11—C11	1.335 (2)		
O31—Ni1—O41	92.30 (5)	C1^{ii}—C2—H2	119.2
O31—Ni1—O32^{i}	93.04 (5)	C12—N11—C11	117.00 (13)
O41—Ni1—O32^{i}	95.94 (5)	C12—N11—Ni1	123.80 (11)
O31—Ni1—O21	90.89 (5)	C11—N11—Ni1	118.62 (10)
O41—Ni1—O21	89.46 (5)	N11—C11—C12^{iii}	121.75 (14)
O32^{i}—Ni1—O21	173.18 (5)	N11—C11—H11	119.1
O31—Ni1—N11	178.28 (5)	C12^{iii}—C11—H11	119.1
O41—Ni1—N11	87.46 (5)	N11—C12—C11^{iii}	121.25 (14)
O32^{i}—Ni1—N11	88.68 (5)	N11—C12—H12	119.4
O21—Ni1—N11	87.40 (5)	C11^{iii}—C12—H12	119.4
O31—Ni1—N1	86.89 (5)	C21—O21—Ni1	123.54 (11)
O41—Ni1—N1	179.19 (5)	O22—C21—O21	125.53 (16)
O32^{i}—Ni1—N1	84.14 (5)	O22—C21—H21	118.4

O21—Ni1—N1	90.52 (5)	O21—C21—H21	115.9
N11—Ni1—N1	93.35 (5)	C31—O31—Ni1	125.64 (11)
C2—N1—C1	116.76 (13)	C31—O32—Ni1^{iv}	130.50 (10)
C2—N1—Ni1	118.93 (11)	O31—C31—O32	127.86 (15)
C1—N1—Ni1	124.31 (10)	O31—C31—H31	120.4
N1—C1—C2^{ii}	121.65 (14)	O32—C31—H31	111.6
N1—C1—H1	119.2	Ni1—O41—H1O	120.0
C2^{ii}—C1—H1	119.2	Ni1—O41—H2O	116.4
N1—C2—C1^{ii}	121.59 (15)	H1O—O41—H2O	103.1
N1—C2—H2	119.2		

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