Crystal structure of diaqua-bis(pyridine)-μ_2-squarato(1,3)-nickel(II), $Ni(C_4O_4)(C_5H_5N)_2(H_2O)_2$

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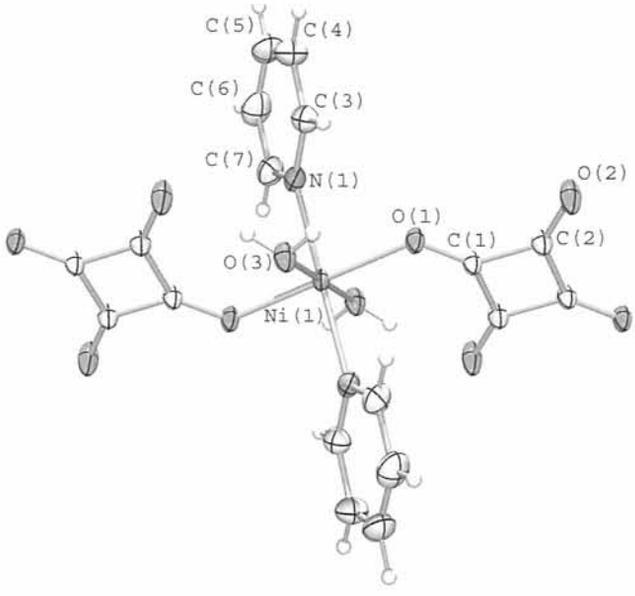

Abstract

$C_{14}H_{14}N_2NiO_6$, orthorhombic, *Pbca* (No. 61), $a = 7.914(1)$ Å, $b = 12.020(2)$ Å, $c = 15.718(3)$ Å, $V = 1495.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.026$, $wR_{ref}(F^2) = 0.062$, $T = 293$ K.

Source of material

Single crystals suitable for X-ray analysis have been obtained by mixing 7.5 ml of 0.2 M nickel(II) nitrate solution with 7.5 ml sodium metasilicate solution (1.06 g/ml, pH 5.0). After three days the gelling process is finished and a solution, containing 0.05 M sodium-squarate and 0.1 M pyridine, was placed over the set gel. Within one week well developed blue crystals are formed on the gel-solution interface.

Discussion

The coordination polyhedron of nickel in diaquabis(pyridine)-squarato(1,3)nickel(II) is best described as an distorted octahedron. Each nickel atom is *trans*-connected to two molecules of pyridine (d(Ni1—N1) = 2.103 Å), two molecules water (d(Ni1—O3) = 2.090 Å) and two squarate-oxygen atoms (d(Ni1—O1) = 2.079 Å). The squarate anion acts as a bridging ligand so that one-dimensional chains of trans-squarato bridged nickel(II) ions are built. These chains run parallel to the a axis. The water molecules are stabilized by building two different hydrogen bonds with two squarate-oxygen atoms. One hydrogen bond (O2···H3a–O3) is located between two parallel strands and one hydrogen bond (O2···H3b–O3) is located within the chain. During structure refinement, H atoms bound to the coordinated water molecule O3 were localized directly from the difference Fourier synthesis. The remaining H atoms (pyridine) were geometrically set as idealized aromatic C–H groups using a riding model.

Table 1. Data collection and handling.

Crystal:	blue prism, size 0.12 × 0.23 × 0.46 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.31 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, φ
$2\theta_{max}$:	51.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10831, 1407
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1009
$N(param)_{refined}$:	114
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(7)	8c	–0.1599	0.5854	0.1634	0.041
H(3)	8c	0.0076	0.2885	0.1077	0.039
H(6)	8c	–0.2240	0.5305	0.2994	0.054
H(4)	8c	–0.0546	0.2270	0.2423	0.052
H(5)	8c	–0.1665	0.3488	0.3410	0.055
H(3A)	8c	0.016(4)	0.697(2)	0.050(2)	0.047(8)
H(3B)	8c	0.175(4)	0.665(2)	0.031(2)	0.058(9)

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	4*a*	0	1/2	0	0.0129(2)	0.0161(2)	0.0228(2)	0.0001(1)	–0.0001(1)	0.0004(1)
O(1)	8*c*	–0.2351(2)	0.5747(1)	–0.01453(8)	0.0153(8)	0.0172(6)	0.0373(8)	–0.0014(5)	–0.0005(6)	0.0028(5)
O(3)	8*c*	0.0840(2)	0.6483(1)	0.05499(9)	0.0204(9)	0.0192(7)	0.0365(8)	0.0014(6)	–0.0020(7)	–0.0027(6)
O(2)	8*c*	–0.6122(2)	0.6739(1)	–0.0179(1)	0.0183(9)	0.0161(7)	0.073(1)	0.0013(6)	0.0005(7)	0.0048(6)
N(1)	8*c*	–0.0687(2)	0.4438(1)	0.1219(1)	0.022(1)	0.0264(9)	0.0267(8)	–0.0017(8)	0.0019(7)	0.0008(6)
C(7)	8*c*	–0.1374(3)	0.5125(2)	0.1795(1)	0.033(1)	0.035(1)	0.034(1)	0.0004(9)	0.0059(9)	–0.0019(9)
C(3)	8*c*	–0.0392(3)	0.3380(2)	0.1467(1)	0.034(1)	0.031(1)	0.033(1)	0.0015(9)	0.0001(9)	0.0045(9)
C(6)	8*c*	–0.1763(4)	0.4803(2)	0.2614(2)	0.043(2)	0.061(2)	0.031(1)	–0.001(1)	0.010(1)	–0.007(1)
C(4)	8*c*	–0.0758(4)	0.3007(2)	0.2275(2)	0.048(2)	0.042(1)	0.041(1)	–0.001(1)	–0.001(1)	0.015(1)
C(5)	8*c*	–0.1433(3)	0.3724(2)	0.2859(1)	0.045(2)	0.067(2)	0.027(1)	–0.006(1)	0.004(1)	0.011(1)
C(1)	8*c*	–0.3791(3)	0.5327(1)	–0.0062(1)	0.016(1)	0.0156(8)	0.027(1)	0.0002(7)	–0.0017(8)	0.0002(7)
C(2)	8*c*	–0.5499(3)	0.5783(2)	–0.0086(1)	0.018(1)	0.0180(8)	0.030(1)	0.0000(7)	–0.0014(8)	0.0000(7)

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