Crystal structure of diaqua-bis(pyridine)- μ_2 -squarato(1,3)cobalt(II), $\text{Co}(\text{C}_4\text{O}_4)(\text{C}_5\text{H}_5\text{N})_2(\text{H}_2\text{O})_2$

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Abstract

$\text{C}_{14}\text{H}_{14}\text{N}_2\text{CoO}_6$, orthorhombic, $Pbca$ (no. 61), $a = 11.325(2)$ Å, $b = 7.959(1)$ Å, $c = 16.664(3)$ Å, $V = 1502.0$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.023$, $wR_{\text{ref}}(F^2) = 0.060$, $T = 293$ K.

Source of material

Single crystals suitable for X-ray analysis have been obtained by mixing 7.5 ml of 0.2 M cobalt(II) nitrate solution with 7.5 ml sodium metasilicate solution (1.06 g/ml, pH = 6.0). 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. set gel. Within one week well developed red crystals are formed on the gel-solution interface.

Experimental details

During structure refinement, H atoms bound to the coordinated water molecule O3, were located directly from the difference Fourier synthesis and refined isotropically. The remaining H atoms (pyridine) were geometrically set as idealized aromatic C–H groups using a riding model.

Discussion

Recently we reported about the crystal growth and structure determination of *catena*-diaquabis(pyridine)squarato(1,3)-nickel(II) [1]. Now we have been able to obtain the cobalt ana-

logue, which is not isotypic to the formerly described nickel or copper compounds [2]. The coordination polyhedron about cobalt in diaquabis(pyridine)squarato(1,3)cobalt(II) is a distorted octahedron as in the cases of the respective nickel(II) or copper(II) complexes. Each cobalt atom is *trans*-connected to two molecules of pyridine ($d(\text{Co1}—\text{N1}) = 2.165$ Å), two molecules of water ($d(\text{Co1}—\text{O3}) = 2.051$ Å) and two squarate-oxygen atoms ($d(\text{Co1}—\text{O1}) = 2.115$ Å). The crystal structure is based on cobalt(II)- μ_2 -squarato chains which run parallel to the *b* axis. Additional stability in the *ab* layer is provided by the hydrogen bonds originating from the metal-water centres. One hydrogen bond ($\text{O2} \cdots \text{H3a}—\text{O3}$) is located between two parallel strands and the second one ($\text{O2} \cdots \text{H3b}—\text{O3}$) within the chain. The structure of the analogous nickel complex [1] differs from the title compound in the arrangement of the squarate-planes between two neighbouring polymer chains.

Table 1. Data collection and handling.

Crystal:	red prism, size $0.19 \times 0.27 \times 0.35$ mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	11.75 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS, φ
$2\theta_{\text{max}}$:	51.82°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	10864, 1380
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1128
$N(\text{param})_{\text{refined}}$:	114
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(3B)	8c	0.166(2)	0.184(3)	0.439(1)	0.051(6)
H(3A)	8c	0.214(2)	0.016(3)	0.440(1)	0.045(6)
H(3)	8c	−0.2205	0.1881	0.4383	0.042
H(6)	8c	−0.0360	0.0943	0.1993	0.068
H(7)	8c	0.0447	0.0058	0.3179	0.044
H(4)	8c	−0.3089	0.2800	0.3221	0.064
H(5)	8c	−0.2150	0.2342	0.1997	0.075

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Co(1)	4b	0	0	½	0.0195(2)	0.0097(2)	0.0222(2)	0.0011(1)	0.0024(1)	0.0017(1)
O(2)	8c	0.14623(9)	0.3963(1)	0.41887(6)	0.0233(5)	0.0147(6)	0.0317(6)	0.0002(4)	0.0074(4)	−0.0004(4)
O(3)	8c	0.1611(1)	0.0793(2)	0.45843(8)	0.0239(6)	0.0151(7)	0.0523(8)	0.0035(5)	0.0110(5)	0.0072(6)
O(1)	8c	−0.03906(9)	0.2377(1)	0.55007(6)	0.0311(5)	0.0105(6)	0.0257(5)	−0.0006(5)	0.0056(4)	0.0015(4)
N(1)	8c	−0.0795(1)	0.0884(2)	0.38967(7)	0.0302(7)	0.0175(8)	0.0246(6)	0.0003(6)	−0.0008(5)	0.0005(5)
C(2)	8c	0.0654(1)	0.4528(2)	0.46320(8)	0.0177(7)	0.0131(8)	0.0217(7)	−0.0005(5)	−0.0025(6)	−0.0012(6)
C(1)	8c	−0.0170(1)	0.3800(2)	0.52189(8)	0.0199(7)	0.0138(9)	0.0196(7)	−0.0008(6)	−0.0023(5)	0.0007(6)
C(3)	8c	−0.1830(2)	0.1689(2)	0.3895(1)	0.038(1)	0.028(1)	0.0384(9)	0.0083(7)	−0.0089(8)	−0.0016(7)
C(6)	8c	−0.0753(2)	0.1147(3)	0.2474(1)	0.078(2)	0.067(2)	0.0234(9)	−0.027(1)	−0.004(1)	0.0011(9)
C(7)	8c	−0.0273(2)	0.0622(3)	0.3189(1)	0.042(1)	0.039(1)	0.0289(9)	−0.0090(9)	0.0039(7)	−0.0048(8)
C(4)	8c	−0.2366(2)	0.2248(3)	0.3200(1)	0.063(1)	0.036(1)	0.062(1)	0.011(1)	−0.034(1)	0.002(1)
C(5)	8c	−0.1812(3)	0.1974(3)	0.2475(1)	0.095(2)	0.051(2)	0.043(1)	−0.020(1)	−0.040(1)	0.016(1)

References

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