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Crystal structure of bis(hydroxydi(pyridin-2-yl) methanolato- $\kappa^3 N,N'O$)cobalt(III) 7,7,8,8-tetracyanoquinodimethane, $C_{34}H_{22}CoN_8O_4$

Table 1: Data collection and handling.

Crystal:	Purple block
Size:	0.13 × 0.12 × 0.09 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.64 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6587, 2720, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2505
$N(\text{param})_{\text{refined}}$:	215
Programs:	Bruker programs [1], SHELX [2]

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Abstract

$C_{34}H_{22}CoN_8O_4$, triclinic, $P\bar{1}$ (no. 2), $a = 6.8423(10)$ Å, $b = 8.8919(13)$ Å, $c = 12.6319(18)$ Å, $\alpha = 104.743(2)^\circ$, $\beta = 94.606(2)^\circ$, $\gamma = 90.934(2)^\circ$, $V = 740.30(19)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0369$, $wR_{\text{ref}}(F^2) = 0.1179$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of materials

A solution of $Co(ClO_4)_2 \cdot 6H_2O$ (18.2 mg, 0.05 mmol) in 1 mL DMF was slowly added to a solution of bis(2-pyridyl)ketone (9.4 mg, 0.05 mmol) and triethylamine (10 μ L) in 1 mL MeCN with stirring. After stirring for 15 min, the resulting mixture

was carefully layered on a solution of LiTCNQ (TCNQ = 7,7,8,8-tetracyanoquinodimethane) (11.2 mg, 0.5 mmol) in MeOH (1 mL) in a 5 dram vial. X-ray quality purple crystals were separated after two days, in a yield of 20% (based on Co).

Experimental details

Crystal data, data collection and structure refinement details are summarized in Table 1. H atoms were included in the refinement at calculated positions and treated as riding with their U_{iso} values set to 1.2–1.5 U_{eq} of the parent atoms.

Comment

Rational design and construction of cobalt complexes has garnered remarkable attention in the field of molecular materials owing to their potential applications in biochemistry, catalysis, electrochemistry, hydrogen production and magnetism [3–5].

The asymmetric unit of the title crystal structure is shown in the figure. The title structure is made up of one $[Co(HL)_2]$ cation and one TCNQ anion each sitting on an inversion centre. The central metal Co(III) ion is sixfold coordinated by two oxygen atoms and four nitrogen atoms from two HL^- ligands. The cation of the title complex is same as our previous report [6]. In title complex, the bond length of Co–O is 1.8809(15) Å, and the Co–N bond distances are in the range of 1.9214(19)–1.9255(18) Å, which are in excellent agreement with those in other Co(III) complexes [7]. In the crystal

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N1	−0.0021(4)	0.6945(4)	0.2267(2)	0.0663(8)
N2	−0.0519(4)	0.2043(4)	0.0481(3)	0.0669(8)
N3	0.5314(3)	0.2203(2)	0.56445(15)	0.0281(4)
N4	0.4917(3)	0.0437(2)	0.35808(15)	0.0280(4)
O1	0.7733(2)	0.01420(18)	0.49409(14)	0.0308(4)
O2	0.9850(2)	0.1865(2)	0.44204(18)	0.0446(5)
H2	1.0591	0.1256	0.4621	0.067*
C1	0.0628(4)	0.5909(4)	0.1667(2)	0.0495(7)
C2	0.1446(4)	0.4646(4)	0.0934(2)	0.0422(6)
C3	0.0359(4)	0.3213(4)	0.0691(2)	0.0479(7)
C4	0.3219(4)	0.4808(3)	0.0465(2)	0.0357(5)
C5	0.4295(4)	0.6258(3)	0.0739(2)	0.0396(6)
H5	0.3821	0.7103	0.1237	0.047*
C6	0.4006(4)	0.3560(3)	−0.0292(2)	0.0392(6)
H6	0.3346	0.2593	−0.0489	0.047*
C7	0.4188(4)	0.3133(3)	0.6338(2)	0.0361(5)
H7	0.3060	0.2724	0.6541	0.043*
C8	0.4700(5)	0.4696(3)	0.6750(2)	0.0470(7)
H8	0.3932	0.5339	0.7237	0.056*
C9	0.6358(5)	0.5287(3)	0.6430(2)	0.0490(7)
H9	0.6708	0.6339	0.6699	0.059*
C10	0.7505(4)	0.4338(3)	0.5715(2)	0.0419(6)
H10	0.8623	0.4734	0.5492	0.050*
C11	0.6948(3)	0.2768(3)	0.53321(19)	0.0309(5)
C12	0.7934(3)	0.1464(3)	0.4539(2)	0.0318(5)
C13	0.6633(3)	0.1120(3)	0.34610(19)	0.0307(5)
C14	0.7017(4)	0.1423(3)	0.2482(2)	0.0405(6)
H14	0.8222	0.1860	0.2400	0.049*
C15	0.5549(5)	0.1056(3)	0.1613(2)	0.0473(7)
H15	0.5766	0.1251	0.0942	0.057*
C16	0.3782(5)	0.0406(3)	0.1752(2)	0.0448(6)
H16	0.2788	0.0182	0.1182	0.054*
C17	0.3495(4)	0.0087(3)	0.2749(2)	0.0341(5)
H17	0.2311	−0.0370	0.2843	0.041*
Co1	0.5000	0.0000	0.5000	0.02453(16)

structure, the cations are interlinked via centrosymmetric O—H···O hydrogen bonds and π – π interactions to form a 3D supramolecular structure. TCNQ is linked to four cations via C—H(arom)···N(TCNQ) hydrogen bonds.

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