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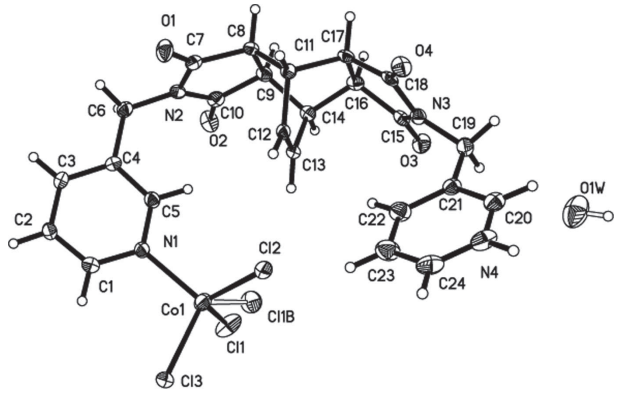
Crystal structure of 3-((1,3,5,7-tetraoxo-6-(pyridin-3-ylmethyl)-3,3a,4,4a,5,6,7,7a,8,8a-decahydro-4,8-ethenopyrrolo[3,4-*f*]isoindol-2(1*H*)-yl)methyl)pyridin-1-ium-κ*N*-trichloridocobalt(II) hemihydrate, $C_{24}H_{22}Cl_3CoN_4O_{4.5}$



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Abstract

$C_{24}H_{22}Cl_3CoN_4O_{4.5}$, monoclinic, $P2_1/c$ (no. 14), $a = 7.1852$ (3) Å, $b = 17.1435(7)$ Å, $c = 19.9264(9)$ Å, $\beta = 95.873(5)°$, $V = 2441.64(18)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0442$, $wR_{ref}(F^2) = 0.0859$, $T = 153$ K.

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The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A 5 mL CH_2Cl_2 solution of 2,6-bis(pyridin-3-ylmethyl) hexahydro-4,8-ethenopyrrolo-[3,4-*f*]isoindole-1,3,5,7-tetrone

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Table 1: Data collection and handling.

Crystal:	Blue blocks Size 0.20 x 0.15 x 0.10 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	10.7 cm^{-1}
Diffractometer, scan mode:	Bruker Frambo, φ and ω
$2\theta_{max}$, completeness:	54°, 98%,
$N(hkl)_{measured}$ $N(hkl)_{unique}$, R_{int}:	12329, 4688, 0.044
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2749
$N(param)_{refined}$:	349
Programs:	SHELX [6]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.37879(8)	0.66773(3)	0.09276(2)	0.03855(15)
Cl1[a]	0.1034(4)	0.72365(15)	0.12266(16)	0.0614(8)
Cl1B[a]	0.1710(4)	0.70009(16)	0.15984(12)	0.0522(7)
Cl2	0.6221(2)	0.64893(5)	0.16940(5)	0.0695(4)
Cl3	0.46732(15)	0.74979(5)	0.01306(4)	0.0444(3)
O1	0.5327(4)	0.30796(14)	0.14999(11)	0.0440(7)
O1W[a]	0.8763(10)	0.7221(4)	0.5519(4)	0.085(2)
H1B[a]	0.769(5)	0.739(6)	0.554(5)	0.127*
H1C[a]	0.956(9)	0.741(6)	0.580(5)	0.127*
O2	−0.0162(3)	0.41389(15)	0.19800(12)	0.0424(6)
O3	0.3761(3)	0.55960(14)	0.44023(11)	0.0391(6)
O4	0.9214(3)	0.44660(13)	0.39676(11)	0.0367(6)
N1	0.3058(4)	0.56605(15)	0.04431(13)	0.0273(6)
N2	0.2447(4)	0.36091(14)	0.15961(13)	0.0261(6)
N3	0.6688(4)	0.51875(15)	0.42007(13)	0.0275(7)
N4	1.1608(4)	0.70080(17)	0.39659(19)	0.0459(9)
H4A	1.2637	0.7232	0.4152	0.055*
C1	0.2885(4)	0.56121(19)	−0.02312(17)	0.0292(8)
H1A	0.3172	0.6057	−0.0486	0.035*
C2	0.2307(5)	0.49419(19)	−0.05678(17)	0.0314(9)
H2A	0.2142	0.4934	−0.1047	0.038*
C3	0.1972(5)	0.42863(19)	−0.02098(16)	0.0293(8)
H3A	0.1599	0.3817	−0.0438	0.035*

Table 2: (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C4	0.2179(4)	0.43106(17)	0.04912(16)	0.0249(8)
C5	0.2714(4)	0.50165(18)	0.07920(17)	0.0290(8)
H5A	0.2842	0.5045	0.1271	0.035*
C6	0.1781(5)	0.35888(18)	0.08818(16)	0.0310(8)
H6A	0.0412	0.3502	0.0837	0.037*
H6B	0.2361	0.3137	0.0674	0.037*
C7	0.4195(5)	0.33320(18)	0.18473(17)	0.0303(8)
C8	0.4384(5)	0.34102(18)	0.26054(15)	0.0273(8)
H8A	0.4546	0.2884	0.2820	0.033*
C9	0.2537(5)	0.37788(19)	0.27719(16)	0.0296(8)
H9A	0.1880	0.3423	0.3068	0.036*
C10	0.1393(5)	0.38768(19)	0.20960(17)	0.0300(8)
C11	0.6054(4)	0.39442(17)	0.28546(15)	0.0255(8)
H11A	0.7269	0.3728	0.2734	0.031*
C12	0.5669(5)	0.47365(18)	0.25547(15)	0.0257(8)
H12A	0.6509	0.4990	0.2287	0.031*
C13	0.4077(5)	0.50571(18)	0.26890(15)	0.0265(8)
H13A	0.3680	0.5555	0.2522	0.032*
C14	0.2944(4)	0.45742(18)	0.31260(15)	0.0261(8)
H14A	0.1765	0.4846	0.3218	0.031*
C15	0.4756(5)	0.51284(19)	0.41652(16)	0.0271(8)
C16	0.4189(4)	0.43892(18)	0.37832(16)	0.0280(8)
H16A	0.3508	0.4035	0.4073	0.034*
C17	0.6021(4)	0.40125(18)	0.36318(15)	0.0254(8)
H17A	0.6153	0.3485	0.3845	0.030*
C18	0.7535(5)	0.45457(19)	0.39376(15)	0.0281(8)
C19	0.7731(5)	0.5824(2)	0.45460(17)	0.0348(9)
H19A	0.8548	0.5610	0.4933	0.042*
H19B	0.6841	0.6191	0.4726	0.042*
C20	1.0543(5)	0.66204(19)	0.4361(2)	0.0374(9)
H20A	1.0903	0.6590	0.4833	0.045*
C21	0.8917(5)	0.62629(18)	0.40897(17)	0.0301(8)
C22	0.8447(5)	0.6345(2)	0.34083(19)	0.0422(10)
H22A	0.7314	0.6124	0.3207	0.051*
C23	0.9596(6)	0.6747(2)	0.3009(2)	0.0495(11)
H23A	0.9267	0.6791	0.2537	0.059*
C24	1.1185(6)	0.7072(2)	0.3300(2)	0.0488(11)
H24A	1.1997	0.7345	0.3034	0.059*

[a]Occupancy: 0.500;

(1 mmol, 0.43 g) was added to one leg of an H-shaped tube, and a 5 ml MeOH solution of $CoCl_2 \cdot 6H_2O$(1 mmol, 0.24 g) was added to the other leg of the tube. Blue single crystals were obtained after two weeks.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The H atoms of the water molecule were located on a difference Fourier map and refined with U_{iso}(H) set to 1.5U_{eq}(O).

For one of the chlorine positions a positional disorder was refined (*cf*. the figure).

Discussion

The terminal pyridyl derivatives have driven much attention for their novel topological structures and potential applications in the areas of luminescence, nonlinear optics, magnetism, catalysts and so on [1–3]. 2,6-Bis(pyridin-3-ylmethyl)hexahydro-4,8-ethenopyrrolo-[3,4-*f*]isoindole-1,3,5,7-tetrone (*3-pybtd*) has been used for the synthesis of complexes and have been reported by us earlier [4, 5]. The asymmetric unit of the title structure $[Co(C_{24}H_{21}N_4O_4)Cl_3]$. $0.5(H_2O)$ consists of Co^{2+}, one *3-pybtd* ligand, three chlorido ligands and one half of a water molecule. The Co(II) cation is coordinated by three chlorido ligands [Co—Cl bond lengths from 2.176(2) to 2.331(2) Å] and a N atom from *3-pybtd* ligand [Co-N = 2.035(2) Å] to complete a distored tetrahedral coordination enviroment (*cf*. the figure). The other pyridyl nitrogen atom N4 is protonated. The backbone of the structure is essentially *cis*-U conformation. The bond angles of C4—C6—N2 and C21—C19—N3 are 115.1(2) and 112.6(3)°, respectively. Each mononuclear complex is connected to adjacent complexes by N—H···Cl interactions.

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