Crystal structure of bis(2,2′-bipyridyl-*N*,*N*′)carbonatocobalt(III) iodide pentahydrate, $[Co(C_{10}H_8N_2)_2(CO_3)][I] \cdot 5H_2O$

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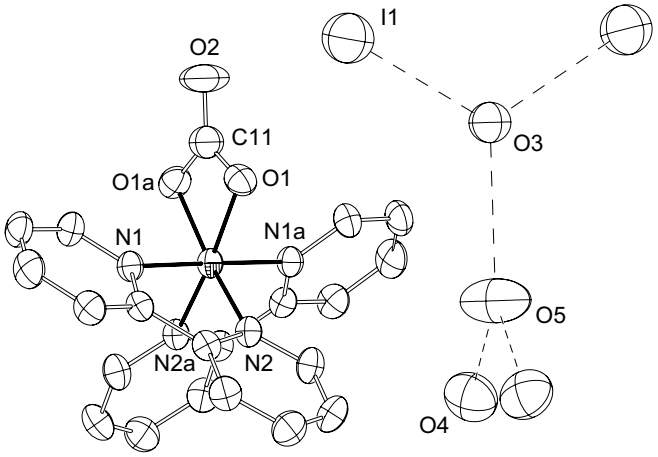

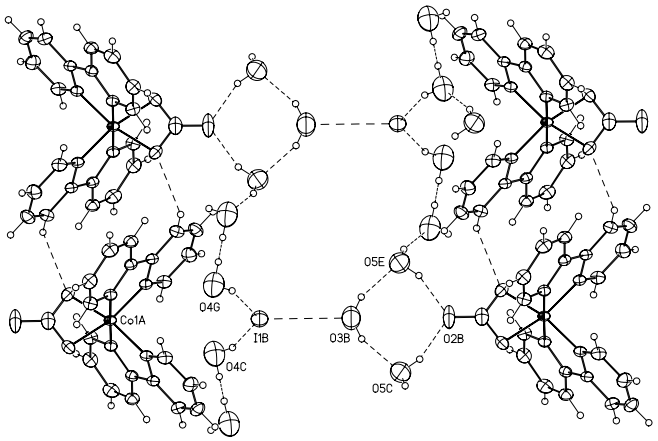

Abstract

$C_{21}H_{26}CoIN_4O_8$, monoclinic, *C*12/*c*1 (no. 15), *a* = 11.0277(5) Å, *b* = 16.0127(7) Å, *c* = 14.4886(7) Å, β = 103.055(2)°, *V* = 2492.3 Å^3, *Z* = 4, $R_{gt}(F)$ = 0.049, $wR_{ref}(F^2)$ = 0.151, *T* = 293 K.

Source of material

Freshly prepared $CoCO_3$ (0.12 g, 1.01 mmol), 2,2′-bipyridine (0.08 g, 0.51 mmol), 2-iodo-benzoic acid (0.22 g, 0.89 mmol), 15 mL CH_3OH/H_2O (1:2, *v*/*v*) were mixed and stirred for ca. 3 h. Subsequently, the resulting suspension was heated in a 23 ml Teflon-lined stainless steel autoclave at 423 K for 5 day and 373 K for 3 days. After the autoclave was cooled to room temperature, the solid was filtered off. The resulting orange filtrate was allowed to stand at room temperature and slow evaporation for a week afforded orange block-shaped crystals.

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Elemental analysis – found: C, 39.57 %; H, 3.80 %; N, 8.52 %; calc. for $C_{21}H_{26}CoIN_4O_8$: C, 38.87 %; H, 4.01 %; N, 8.64 %.

Experimental details

H atoms bound to C atoms were placed on calculated positions with *d*(C—H) = 0.93 Å, and refined using the riding rigid-body approximation and $U_{iso} = 1.2U_{eq}$(C). H atoms at O atoms were located in Fourier difference maps, refined with an O—H distance restraint of 0.85 Å and U_{iso}(H) = 1.5 U_{eq}(O) and then fixed.

Discussion

The crystal structure of the title compound is similar to those of $[Co(C_{10}H_8N_2)_2(CO_3)]NO_3 \cdot 5H_2O$ [1], $[Co(C_{10}H_8N_2)_2(NO_3)](NO_3)(OH) \cdot 4H_2O$ [2], $[Co(2,2'\text{-bpy})_2(NO_3)]NO_3 \cdot 5H_2O$ [3], $[Co(2,2'\text{-bpy})_2(CO_3)](HCO_3) \cdot 5H_2O$ [4]. It consists of the monovalent $[Co(2,2'\text{-bpy})_2(CO_3)]^+$ complex cations, iodide anions and crystal water molecules (figure, top). Within the $[Co(2,2'\text{-bpy})_2(CO_3)]^+$, the Co atoms are each coordinated by four N atoms from two bidentate chelating 2,2′-bipyridyl ligands and two O atoms from one bidentate chelating carbonate anions ligands, to complete significantly distorted CoN_4O_2 octahedra environment with *d*(Co—N) = 1.926(3) Å (2×), 1.934(3) Å (2×) and *d*(Co—O) = 1.898(3) Å (2×), the two $[Co(2,2'\text{-bpy})_2(CO_3)]^+$ complex cations are interlinked by hydrogen bonds and π-π stacking interactions into 1D supramolecular chains along the [100] direction, but the interplanar distance of 3.642 Å indicates that the intercationic π-π stacking interactions between the neighboring 2,2′-bpy planes are very weak (figure, bottom). Moreover, there are hydrogen bonds and weak C–H···O interactions between the coordinating carbonato groups, non-coordinating iodide anions, crystal water molecules and 2,2′-bipyridyl H atoms with *d*(O···O) = 2.669 Å – 2.720 Å, ∠O–H···O = 154.0° – 179.3°, *d*(O4···I1^i) = 3.557 Å, ∠O4–H4A···I1^i = 150.1° (i: *x*+½,*y*–½,*z*), *d*(C7···O1^{ii}) = 3.537 Å, ∠C7–H7···O1^{ii} = 124.42° (ii: –1–*x*,*y*,–½–*z*). Through these hydrogen bonds and intercationic π-π stacking interactions the structure constituents are interlinked to a 3D network (figure, bottom).

Table 1. Data collection and handling.

Crystal:	orange block, size 0.10 × 0.15 × 0.29 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	19.80 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD II, φ/ω
$2\theta_{max}$:	56.6°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8185, 3027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2135
$N(param)_{refined}$:	162
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	8*g*	0.0196	–0.0736	0.2015	0.232
H(4A)	8*g*	0.2957	–0.2757	0.1192	0.206
H(4B)	8*g*	0.2516	–0.2447	0.0320	0.206
H(5A)	8*g*	0.0497	–0.2070	0.1562	0.180
H(5B)	8*g*	0.1174	–0.1727	0.1070	0.180
H(1)	8*g*	–0.2451	–0.0830	–0.1878	0.055
H(2)	8*g*	–0.0928	–0.0849	–0.0496	0.062
H(3)	8*g*	–0.1178	–0.0051	0.0777	0.068
H(4)	8*g*	–0.2940	0.0761	0.0647	0.057
H(7)	8*g*	–0.4646	0.1589	0.0314	0.059
H(8)	8*g*	–0.6491	0.2336	–0.0147	0.069
H(9)	8*g*	–0.7805	0.2064	–0.1596	0.068
H(10)	8*g*	–0.7225	0.1093	–0.2588	0.057

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}
Co(1)	4*e*	–½	–0.00549(4)	–¼	0.0313(3)	0.0418(4)	0.0249(3)	0	0.0033(2)	0
N(1)	8*g*	–0.3582(2)	–0.0046(2)	–0.1442(2)	0.033(1)	0.044(2)	0.030(1)	–0.000(1)	0.002(1)	0.000(1)
N(2)	8*g*	–0.5631(3)	0.0773(2)	–0.1757(2)	0.033(1)	0.045(2)	0.029(1)	0.001(1)	0.006(1)	0.001(1)
I(1)	4*e*	0	0.16134(3)	¼	0.0845(4)	0.0783(4)	0.0885(4)	0	0.0402(3)	0
O(1)	8*g*	–0.5572(3)	–0.1027(2)	–0.1980(2)	0.050(2)	0.048(2)	0.044(1)	–0.007(1)	0.005(1)	0.009(1)
O(2)	4*e*	–½	–0.2246(3)	–¼	0.119(5)	0.036(3)	0.134(5)	0	0.005(4)	0
O(3)	4*e*	0	–0.0499(5)	¼	0.24(1)	0.093(5)	0.106(6)	0	–0.014(6)	0
O(4)	8*g*	0.2536(5)	–0.2339(3)	0.0916(4)	0.128(4)	0.120(4)	0.145(5)	–0.003(3)	–0.009(4)	0.009(4)
O(5)	8*g*	0.0477(6)	–0.1625(3)	0.1225(4)	0.135(5)	0.118(4)	0.100(4)	0.017(3)	0.008(3)	0.012(3)
C(1)	8*g*	–0.2551(3)	–0.0510(3)	–0.1365(3)	0.036(2)	0.055(2)	0.044(2)	0.006(2)	0.005(2)	–0.000(2)
C(2)	8*g*	–0.1636(4)	–0.0523(3)	–0.0540(3)	0.037(2)	0.060(3)	0.054(2)	0.007(2)	–0.000(2)	0.007(2)
C(3)	8*g*	–0.1785(4)	–0.0047(3)	0.0216(3)	0.047(2)	0.073(3)	0.039(2)	–0.001(2)	–0.011(2)	0.007(2)
C(4)	8*g*	–0.2832(4)	0.0436(3)	0.0140(3)	0.049(2)	0.057(2)	0.032(2)	–0.003(2)	–0.001(2)	–0.000(2)
C(5)	8*g*	–0.3723(3)	0.0431(2)	–0.0702(2)	0.036(2)	0.044(2)	0.031(2)	–0.005(1)	0.006(1)	0.002(1)
C(6)	8*g*	–0.4875(3)	0.0923(2)	–0.0894(2)	0.039(2)	0.044(2)	0.029(2)	–0.003(1)	0.009(1)	0.001(1)
C(7)	8*g*	–0.5174(4)	0.1498(3)	–0.0275(3)	0.050(2)	0.057(2)	0.040(2)	–0.002(2)	0.013(2)	–0.008(2)
C(8)	8*g*	–0.6277(4)	0.1936(3)	–0.0548(3)	0.058(3)	0.059(3)	0.060(3)	0.007(2)	0.025(2)	–0.011(2)
C(9)	8*g*	–0.7053(4)	0.1781(3)	–0.1410(4)	0.048(2)	0.063(3)	0.062(3)	0.018(2)	0.017(2)	0.000(2)
C(10)	8*g*	–0.6699(3)	0.1196(3)	–0.2001(3)	0.037(2)	0.062(3)	0.041(2)	0.006(2)	0.004(2)	0.002(2)
C(11)	4*e*	–½	–0.1488(4)	–¼	0.056(4)	0.049(4)	0.063(4)	0	–0.018(3)	0

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