Crystal structure of diaqua-di(2-methoxyacetato)cobalt(II), $C_6H_{14}CoO_8$

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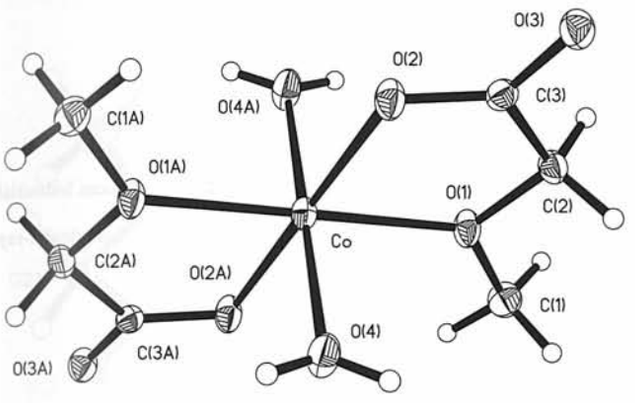

Abstract

$C_6H_{14}CoO_8$, monoclinic, $P12_1/n1$ (No. 14), $a = 6.985(1)$ Å, $b = 10.102(2)$ Å, $c = 7.132(1)$ Å, $\beta = 97.15(1)°$, $V = 499.3$ Å^3, $Z = 2$, $R_{gt}(F) = 0.028$, $wR_{ref}(F^2) = 0.072$, $T = 292$ K.

Source of material

Reagents and solvents used were of commercially available quality. A solution of acid (maaH) (2 mmol, 180 mg) in methanol (10 mL) was added to a stirred solution of $CoCl_2 \cdot 6H_2O$ (1 mmol, 238 mg) in water (10 mL). The resulting solution was filtered and washed with water and methanol after staying still in air for about 3 days. Large pink prism crystals of $Co(maa)_2(H_2O)_2$ were obtained almost quantitatively (yield 95%). Elemental analysis: found – C, 26.31%; H, 5.22%; calc. for $C_6H_{14}O_8Co$ – C, 26.39%; H, 5.17%.

Experimental details

The two H atoms attached to the coordination water molecules were located from difference Fourier maps and refined isotropically, mainly because these hydrogen atoms were able to rotate around the O4–Co1 axis.

Discussion

The C—H and C—C bond activations of carbohydrates brought about by bare transition metal ions are of fundamental interest in various areas of chemical research [1]. The oxidative addition and reductive elimination of ligands are two of the most fundamental steps in reactions occurring at metal centers in catalytic processes. The discovery of metal-containing coordination complexes that are able to bind molecular oxygen reversibly was made by Werner over a century ago [2]. Since that time a wide range of transition metal complexes have been found to have similar behavior. Chelating ligands of carboxylates, amines and imines have been studied extensively to understand the thermodynamics and kinetics of oxygen binding in biological model systems. In investigating the catalysis interaction of Co(II) complexes to the carbohydrate oxygenations by alkylperoxide, we isolated a new Co(II) complex with 2-methoxyacetate.

The title complex is an electronically neutral one. One metal atom, two ligand anions and two coordination water molecules constitute each simplest structural unit. In the unit, each metal atom is six-coordinated by six oxygen atoms, two of which come from the two coordinated water and the rest four from the two ligands, forming a slightly distorted octahedron. Each ligand in the complex is bidentate with one ether oxygen atom and one oxygen from the carboxylate. All the carboxylates are unidentate ligands. O1, O2, C2, C3, O1A, O2A, C2A and C3A constitute a large plane. The metal atom and its four coordinated oxygen atoms are in the plane with the two water molecules occupy the apical positions. All oxygen atoms from the carboxylates and the water molecules contribute to the formation of the hydrogen bonds. Every structural unit is linked by strong H-bonds to four neighboring structure units. The strong hydrogen bonds link the above structure units along z-axis to form a 1D chain. In each 1D are parallel each other at a short distance of 3.454(1) Å. Two different chains are alternatively arranged at the angle of 66.6°. The hydrogen bonds link the chains into a plane, and then link the planes into a 3D structure.

Table 1. Data collection and handling.

Crystal:	pink prism, size 0.40 × 0.56 × 0.56 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.41 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1186, 980
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 866
$N(param)_{refined}$:	80
Programs:	SHELXTL [3], SHELXTL-plus [4]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.2601	–0.0428	0.5037	0.041
H(1B)	4*e*	0.1239	0.0736	0.4267	0.041
H(1C)	4*e*	0.3468	0.0841	0.4199	0.041
H(2A)	4*e*	0.3914	–0.1870	0.2616	0.031
H(2B)	4*e*	0.4931	–0.0555	0.2100	0.031
H(0A)	4*e*	0.097(4)	0.207(3)	–0.173(3)	0.05(1)
H(0B)	4*e*	0.253(2)	0.186(3)	–0.042(4)	0.045(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}
Co	2*a*	0	0	0	0.0194(3)	0.0274(3)	0.0194(3)	0.0015(2)	–0.0009(2)	0.0009(2)
O(1)	4*e*	0.2154(2)	–0.0339(2)	0.2274(2)	0.0245(9)	0.042(1)	0.0195(9)	0.0082(8)	–0.0026(7)	–0.0061(8)
O(2)	4*e*	0.1918(2)	–0.1159(2)	–0.1216(2)	0.0250(9)	0.042(1)	0.0220(8)	0.0061(8)	–0.0023(7)	–0.0037(8)
O(3)	4*e*	0.4827(2)	–0.2077(2)	–0.0846(2)	0.0252(9)	0.038(1)	0.0296(9)	0.0046(8)	0.0022(7)	–0.0058(8)
O(4)	4*e*	0.1397(3)	0.1702(2)	–0.0747(3)	0.026(1)	0.041(1)	0.036(1)	–0.0076(9)	–0.0043(9)	0.0122(9)
C(1)	4*e*	0.2384(4)	0.0250(3)	0.4091(4)	0.038(2)	0.039(2)	0.025(1)	0.004(1)	–0.001(1)	–0.006(1)
C(2)	4*e*	0.3767(3)	–0.1080(3)	0.1836(3)	0.026(1)	0.028(1)	0.024(1)	0.006(1)	–0.002(1)	–0.000(1)
C(3)	4*e*	0.3474(3)	–0.1469(2)	–0.0240(3)	0.024(1)	0.022(1)	0.024(1)	–0.003(1)	0.001(1)	0.000(1)

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