Crystal structure of diaqua-*trans*-dicinnamato-dihexamethylene-tetraminecobalt(II) dihydrate, $Co(C_6H_{12}N_4)_2(C_9H_7O_2)_2(H_2O)_2 \cdot 2H_2O$

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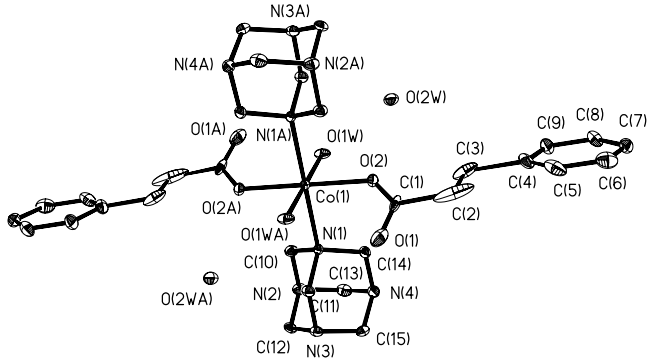

Abstract

$C_{30}H_{46}CoN_8O_8$, monoclinic, $P12_1/c1$ (No. 14), a = 12.779(3) Å, b = 10.938(2) Å, c = 12.218(2) Å, β = 97.56(3)°, V = 1693.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.085, $wR_{ref}(F^2)$ = 0.201, T = 293 K.

Source of material

All reagents and solvents were used as obtained without further purification. C, H and N elemental analyses were performed on a Perkin-Elmer elemental analyzer. Cobalt cinnamate dihydrate (1 mmol, 389 mg) and hexamethylenetetramine (1 mmol, 140 mg) were dissolved in ammonium solution (20 ml), and stirred for ca. 10 min to obtain a clear purple solution. After keeping the solution in air for two weeks with the ammonium gas escaping, large violet crystals were formed. This product was collected, washed three times with water, and dried in a vacuum desiccator using $CaCl_2$ (yield 66%). Elemental analysis: found – C, 50.88%; H, 6.66%; N, 15.70%; calc. for $C_{30}H_{46}CoN_8O_8$ – C, 51.06%; H, 6.57%; N, 15.88%.

Experimental details

The relative large R values are perhaps a result of a deficient absorption correction because the form of the crystal was irregular. Residual electron density: +0.843 e·Å^3, – 0.792 e·Å^3.

All H atoms were placed in geometrically idealised positions and constrained to ride on their parent atoms with N—H and C—H distances of 0.90 Å and 0.96 Å, respectively, and the U_{iso} values for H atoms were fixed at 0.080. Although the C(2) atom was a little disordered (U_{eq} = 0.184(8)) we do not try to split it.

Discussion

Each of the title discrete complex consists of a diaqua-dicinnamato-dihexamethylenetetraminecobalt(II) coordination dication and two lattice water molecules. The central cobalt(II) atom is in a octahedral environment and is coordinated by two nitrogen atoms from two hexamethylenetetramine molecules and four oxygen atoms, two of which from two water molecules and the rest from two cinnamate anions. The Co—O bond lengths are 2.051(4) Å and 2.086(3) Å, demonstrating that the Co(1) atom is di-valent. The Co—N bond length 2.280(4) Å is also in the normal range. The three diagonal angles of the Co(II) polyhedron are all 180°, and the other angles around Co(1) atom are from 85.4(1)° to 94.6(1)°, indicating a slightly distorted octahedron CoO_4N_2. The cinnamate anion in this complex is a unidentate ligand and is in the *trans*-conformation. A great number of hydrogen bonds connect the complex along *ac*-plane to form a two-dimensional network.

Table 1. Data collection and handling.

Crystal:	purple, irregular fragment, size 0.40 × 0.40 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.67 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, 625 images, $\Delta\varphi$ = 0.6°
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7438, 3327
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2402
$N(param)_{refined}$:	215
Programs:	SHELXTL [1], SADABS [2]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	–0.3257	0.3205	–0.0069	0.08
H(3A)	4*e*	–0.2225	0.2359	–0.1673	0.08
H(5A)	4*e*	–0.4703	0.3871	–0.0633	0.08
H(6A)	4*e*	–0.5926	0.5240	–0.1570	0.08
H(7A)	4*e*	–0.5696	0.5925	–0.3342	0.08
H(8A)	4*e*	–0.4289	0.5152	–0.4168	0.08
H(9A)	4*e*	–0.3097	0.3795	–0.3221	0.08
H(10A)	4*e*	0.2229	0.0895	–0.0660	0.08
H(10B)	4*e*	0.2504	0.0462	0.0555	0.08
H(11A)	4*e*	0.1537	0.1248	0.1972	0.08
H(11B)	4*e*	0.0654	0.2215	0.1677	0.08
H(12A)	4*e*	0.3343	0.1818	0.1921	0.08
H(12B)	4*e*	0.3615	0.3143	0.1599	0.08
H(13A)	4*e*	0.3206	0.3816	–0.0239	0.08
H(13B)	4*e*	0.2641	0.2947	–0.1134	0.08
H(14A)	4*e*	0.0220	0.2892	–0.0203	0.08

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14B)	4*e*	0.0848	0.2391	–0.1112	0.08
H(15A)	4*e*	0.1079	0.4214	0.1206	0.08
H(15B)	4*e*	0.2245	0.4604	0.1184	0.08
H(1WA)	4*e*	0.0880	0.0495	–0.1764	0.08
H(1WB)	4*e*	0.0122	–0.0498	–0.2075	0.08
H(2WB)	4*e*	–0.0764	0.1069	–0.3693	0.08
H(2WA)	4*e*	–0.1487	0.0023	–0.3674	0.08

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)	2*a*	0	0	0	0.0352(5)	0.0262(5)	0.0277(5)	–0.0015(4)	–0.0017(3)	0.0019(4)
O(1)	4*e*	–0.1505(4)	0.2064(4)	0.1092(5)	0.075(3)	0.051(3)	0.121(4)	0.007(3)	0.026(3)	–0.015(3)
O(2)	4*e*	–0.1186(3)	0.1217(3)	–0.0509(3)	0.046(2)	0.033(2)	0.072(3)	0.001(2)	–0.016(2)	0.005(2)
N(1)	4*e*	0.1207(3)	0.1534(3)	0.0338(3)	0.035(2)	0.027(2)	0.030(2)	–0.002(2)	0.000(2)	0.004(2)
N(2)	4*e*	0.3054(3)	0.2123(4)	0.0303(4)	0.035(2)	0.052(3)	0.060(3)	–0.010(2)	0.009(2)	–0.008(2)
N(3)	4*e*	0.2098(3)	0.2915(4)	0.1745(3)	0.047(3)	0.042(3)	0.035(2)	–0.014(2)	0.002(2)	–0.003(2)
N(4)	4*e*	0.1657(3)	0.3627(4)	–0.0140(4)	0.049(3)	0.038(3)	0.050(3)	–0.015(2)	–0.003(2)	0.012(2)
C(1)	4*e*	–0.1681(4)	0.1926(6)	0.0073(8)	0.021(3)	0.022(3)	0.161(8)	0.000(2)	–0.006(4)	0.006(4)
C(2)	4*e*	–0.274(1)	0.279(1)	–0.044(1)	0.26(2)	0.14(1)	0.19(1)	–0.15(1)	0.18(1)	–0.12(1)
C(3)	4*e*	–0.2725(7)	0.2790(7)	–0.1295(8)	0.137(7)	0.050(4)	0.109(7)	–0.041(5)	0.079(6)	–0.030(4)
C(4)	4*e*	–0.3778(5)	0.3730(6)	–0.1839(6)	0.051(4)	0.063(5)	0.091(5)	–0.027(4)	–0.029(4)	0.028(4)
C(5)	4*e*	–0.4615(7)	0.4144(8)	–0.1362(6)	0.091(6)	0.106(7)	0.055(4)	–0.053(5)	–0.018(4)	0.026(4)
C(6)	4*e*	–0.5338(6)	0.4954(8)	–0.1913(7)	0.062(4)	0.096(6)	0.089(6)	–0.029(4)	0.020(4)	–0.014(5)
C(7)	4*e*	–0.5215(5)	0.5349(6)	–0.2955(6)	0.048(4)	0.049(4)	0.086(5)	–0.003(3)	–0.011(3)	0.006(3)
C(8)	4*e*	–0.4381(5)	0.4903(6)	–0.3433(5)	0.055(4)	0.085(5)	0.056(4)	–0.002(4)	–0.010(3)	0.010(4)
C(9)	4*e*	–0.3676(5)	0.4104(6)	–0.2875(6)	0.052(4)	0.068(5)	0.073(5)	0.005(3)	–0.012(3)	0.008(4)
C(10)	4*e*	0.2274(4)	0.1149(5)	0.0097(5)	0.034(3)	0.044(3)	0.056(3)	–0.004(2)	0.004(2)	–0.009(3)
C(11)	4*e*	0.1326(4)	0.1936(5)	0.1506(4)	0.049(3)	0.044(3)	0.031(3)	–0.011(3)	0.003(2)	0.003(2)
C(12)	4*e*	0.3106(4)	0.2498(5)	0.1456(4)	0.043(3)	0.048(3)	0.052(3)	–0.014(3)	–0.017(3)	0.000(3)
C(13)	4*e*	0.2694(4)	0.3173(6)	–0.0370(5)	0.057(4)	0.063(4)	0.043(3)	–0.026(3)	0.010(3)	0.004(3)
C(14)	4*e*	0.0902(4)	0.2618(5)	–0.0348(4)	0.048(3)	0.037(3)	0.041(3)	–0.009(2)	–0.006(2)	0.011(2)
C(15)	4*e*	0.1754(4)	0.3942(5)	0.1036(4)	0.057(3)	0.033(3)	0.055(3)	–0.010(3)	0.000(3)	–0.007(3)
O(1W)	4*e*	0.0416(3)	–0.0002(3)	–0.1595(3)	0.061(2)	0.049(2)	0.041(2)	–0.028(2)	0.010(2)	–0.007(2)
O(2W)	4*e*	–0.1021(4)	0.0496(4)	–0.3342(3)	0.146(5)	0.044(3)	0.058(3)	–0.012(3)	–0.037(3)	0.001(2)

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