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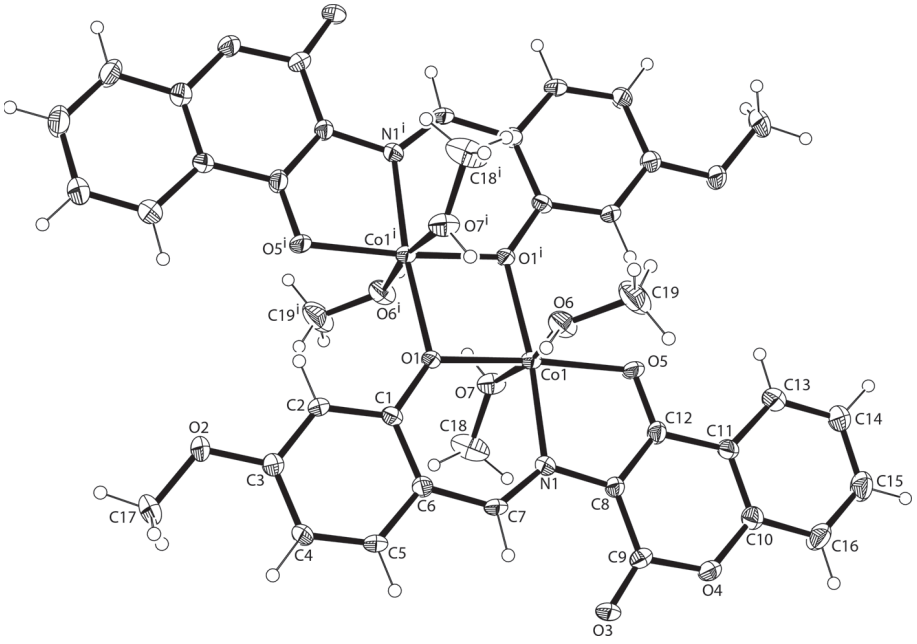
Crystal structure of tetrakis(methanol-κ*O*)bis{3-((4-methoxy-2-oxidobenzylidene)amino)-2-oxo-2*H*-chromen-4-olato-κ^3*O*,*N*,*O*′} bicobalt(II), $C_{38}H_{38}Co_2N_2O_{14}$



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Abstract

$C_{38}H_{38}Co_2N_2O_{14}$, monoclinic, $P2_1/n$ (no. 14), $a = 9.7459(5)$ Å, $b = 7.9026(7)$ Å, $c = 24.0622(15)$ Å, $\beta = 100.260(5)°$, $Z = 2$, $V = 1823.6(2)$ Å^3, $R_{gt}(F) = 0.0576$, $wR_{ref}(F^2) = 0.1201$, $T = 293(2)$ K.

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

Table 1: Data collection and handling.

Crystal:	Block, dark brown
Size:	0.34 × 0.31 × 0.14 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.98 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	26°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6511, 3575, 0.059
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2288
$N(param)_{refined}$:	262
Programs:	Bruker programs [1], SHELX [2]

Source of material

Synthesis of the ligand HL: The ligand was synthesized according to a method reported previously [3, 4]. To an ethanol solution (5 mL) of 2-hydroxy-4-methoxybenzaldehyde (152.2 mg, 1 mmol) was added an ethanol solution (5 mL) of 3-amino-4-hydroxy-2*H*-chromen-2-one (177.2 mg, 1 mmol). The solution was stirred under reflux conditions at 434 K for 6 h. After cooling to room temperature, the precipitate was filtered and washed with ethanol and hexane. The product was dried under vacuum obtaining a yellow solid (yield 71.8%, m.p. 460–461 K). Anal. calcd. For $C_{17}H_{13}NO_5$ (%): C, 65.59; H, 4.21; N, 4.50. Found (%): C, 66.47; H, 4.13; N, 4.85.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Co1	0.61056(5)	0.39372(8)	0.47557(2)	0.0303(2)
O1	0.6012(2)	0.5407(4)	0.54338(10)	0.0292(7)
O2	0.7196(3)	0.7569(5)	0.72685(12)	0.0488(9)
O3	1.1047(3)	0.2039(4)	0.51123(11)	0.0388(8)
O4	1.0502(3)	0.0907(4)	0.42672(12)	0.0380(8)
O5	0.6488(3)	0.2636(4)	0.40732(11)	0.0397(9)
O6	0.6628(3)	0.6404(4)	0.43577(12)	0.0423(9)
H6	0.727(3)	0.706(3)	0.4536(8)	0.063*
O7	0.5442(3)	0.1843(4)	0.51906(13)	0.0447(9)
H7	0.4611(15)	0.186(4)	0.5268(14)	0.067*
N1	0.8197(3)	0.3452(5)	0.50275(13)	0.0290(9)
C1	0.7005(4)	0.5610(5)	0.58934(16)	0.0256(10)
C2	0.6689(4)	0.6497(6)	0.63508(16)	0.0293(10)
H2	0.5801	0.6953	0.6327	0.035*
C3	0.7654(4)	0.6727(6)	0.68439(17)	0.0329(11)
C4	0.9002(4)	0.6084(6)	0.68777(17)	0.0366(12)
H4	0.9670	0.6248	0.7201	0.044*
C5	0.9319(4)	0.5217(6)	0.64313(17)	0.0342(11)
H5	1.0220	0.4798	0.6459	0.041*
C6	0.8368(4)	0.4913(6)	0.59292(16)	0.0270(10)
C7	0.8900(4)	0.3949(6)	0.55073(17)	0.0308(11)
H7A	0.9839	0.3655	0.5587	0.037*
C8	0.8733(4)	0.2476(6)	0.46262(16)	0.0271(10)
C9	1.0129(4)	0.1860(6)	0.46979(17)	0.0287(10)
C10	0.9592(4)	0.0560(6)	0.37721(18)	0.0334(11)
C11	0.8228(4)	0.1158(6)	0.36909(17)	0.0296(10)
C12	0.7759(4)	0.2119(6)	0.41354(16)	0.0296(10)
C13	0.7363(4)	0.0790(6)	0.31712(18)	0.0410(12)
H13	0.6445	0.1170	0.3101	0.049*
C14	0.7868(5)	−0.0123(7)	0.27683(19)	0.0467(14)
H14	0.7293	−0.0354	0.2425	0.056*
C15	0.9229(5)	−0.0707(7)	0.2868(2)	0.0507(14)
H15	0.9558	−0.1336	0.2593	0.061*
C16	1.0099(5)	−0.0365(7)	0.3370(2)	0.0492(14)
H16	1.1014	−0.0753	0.3436	0.059*
C17	0.8102(5)	0.7603(7)	0.78035(17)	0.0519(14)
H17A	0.8894	0.8305	0.7780	0.078*
H17B	0.8411	0.6475	0.7908	0.078*
H17C	0.7611	0.8052	0.8083	0.078*
C18	0.6281(5)	0.0846(7)	0.5613(2)	0.0687(18)
H18A	0.7012	0.0315	0.5456	0.103*
H18B	0.5715	−0.0007	0.5744	0.103*
H18C	0.6683	0.1557	0.5923	0.103*
C19	0.6654(5)	0.6589(8)	0.37690(19)	0.0690(18)
H19A	0.5868	0.6012	0.3554	0.103*
H19B	0.7500	0.6112	0.3686	0.103*
H19C	0.6610	0.7769	0.3672	0.103*

Synthesis of the cobalt(II) complex: To a solution of the aforementioned organic ligand (4.4 mg, 0.014 mmol) in 5 mL dichloromethane was added a few drops of triethylamine solution, and the mixture was added dropwise to a methanol solution (4 mL) of cobalt acetate tetrahydrate (12.5 mg, 0.05 mmol) at room temperature. The mixing solution turned to red-brown immediately and the filtrate was allowed to stand at room temperature for about two weeks. Dark brown block single crystals were obtained. Elemental Analysis-Anal. calcd. for $C_{38}H_{38}Co_2N_2O_{14}$: C, 52.79; H, 4.43; N, 3.24. Found(%): C, 52.81; H, 4.35; N, 3.36.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Azomethine group (—C=N—) containing compounds, typically known as Schiff bases, have been synthesized via condensation of primary amines with active carbonyls, as a class of ligands and are known to coordinate with metal ions through the azomethine nitrogen atom [5, 6]. And they can form all kinds of stable metal complexes [7–10], which are considered more and more important because their novelty structural, topological and widely using in the fields of biochemistry [11, 12], magnetic properties [13, 14], photophysical properties [15, 16], molecular recognition [17], supramolecular architectures [18]. Herein, a new cobalt(II) complex with coumarin-Schiff base ligand ((*E*)-4-hydroxy-3-((2-hydroxy-4-methoxybenzylidene) amino)-2*H*-chromen-2-one) is reported.

The single crystal structure of the title Co(II) complex reveals a symmetric dinuclear structure. It crystallizes in the monoclinic space group $P2_1/n$, and consists of two Co(II) ions, two tridentate ligand units L^{2-}, and four coordinated methanol ligands. The Co(II) center (Co1 or Co1a) is coordinated by one N1 and two O (O5, O1) atoms from a tridentate ligand unit, and two methanol molecules to obtain two [CoL(MeOH)$_2$] symmetry dependent moieties. The [CoL(MeOH)$_2$] moieties are bridged via the μ_2-phenolate oxygen atom (O1) to form a binuclear structure. The coordination geometry of the Co(II) center can be described as a slightly distorted octahedron.

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