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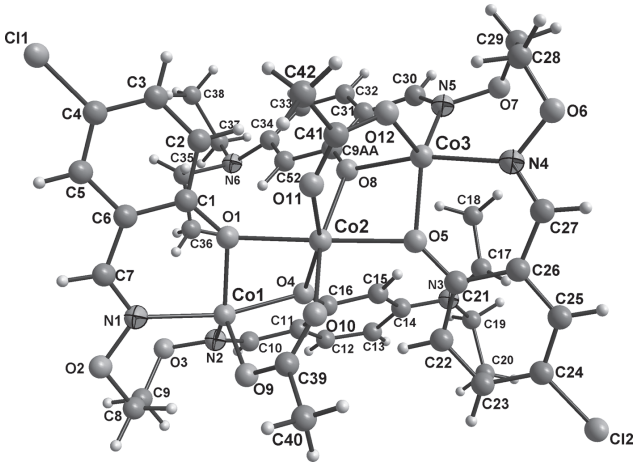
Crystal structure of bis(μ_2-acetato-κ^2O:O')-bis{μ_2-4-chloro-2-(8-(4-(diethylamino)-2-oxidophenyl)-3,6-dioxa-2,7-diazaocta-1,7-dien-1-yl)phenolato-κ^6O:O,N,N',O'':O''}tricobalt(II), $C_{44}H_{49}Cl_2Co_3N_6O_{12}$



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Abstract

$C_{44}H_{49}Cl_2Co_3N_6O_{12}$, Triclinic, $P\bar{1}$, $a = 9.7415(9)$ Å, $b = 13.6440(14)$ Å, $c = 17.5241(11)$ Å, $\alpha = 72.807(10)°$, $\beta = 83.019(9)°$, $\gamma = 72.246(9)°$, $V = 2450.6(5)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0700$, $wR_{ref}(F^2) = 0.1505$, $T = 291(2)$ 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.

Source of material

A methanol solution (3.0 mL) of cobalt acetate tetrahydrate (370.6 mg 0.015 mmol) was added to a acetone solution (2.0 mL) of H_2L 4-(diethylamino)-2-hydroxybenzaldehyde O-(2-(((5-chloro-2-hydroxybenzylidene)amino)oxy)ethyl) oxime (4.01 mg, 0.010 mmol) at room temperature, the color of the mixing solution turned brown immediately. After the mixture was stirred for 1 h, the mixture was filtered. The resulting filtrate was left undisturbed for about one week to form clear light brown block crystals. Yield: 78.5%. Elemental analysis-Anal. Calcd. for $C_{44}H_{49}Cl_2Co_3N_6O_{12}$: C, 47.97%; H, 4.48%; N, 7.63%. Found: C, 48.25%; H, 4.32%; N, 7.50%.

Table 1: Data collection and handling.

Crystal:	clear light brown needle
size:	0.13 × 0.08 × 0.07 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.18 mm^{-1}
Diffractometer, scan mode:	SuperNova ω
θ_{max}, completeness:	26.0°, 99.7%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	19011, 9620, 0.079
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4395
$N(param)_{refined}$:	657
Programs:	CrysAlis^{PRO} [17], SHELX [18]

Experimental details

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

Discussion

Salen and its derivatives are a very important versatile ligand in coordination chemistry as a kind of multidentate ligands [1–4], and their metal complexes [5–7] have a very wide range of applications, such as optical and magnetic materials [8–11], biological activities [12], supramolecular architectures [13–15].

The crystal structure shows that the title complex consists of three Co(II) atoms, two $(L)^{2-}$ units and two acetate ions. The coordination sphere of terminal Co1 or Co3 atom is completed by N_2O_2 donors of $(L)^{2-}$ unit and one oxygen atom of acetate. The sphere of central Co2 was completed by four phenolate oxygen atoms and two oxygen atoms of acetate ions, thus, constitute three octahedral geometries around three hexa-coordinated cobalt atoms, respectively. The two acetate ions coordinate to the three Co(II) atoms. The carbon, nitrogen and hydrogen atoms of one of the *N*,*N*′-diethylamine groups are disordered over two different positions (*cf*. Table 2). This disorder is similar to that for previously reported Salamo-type Ni complex [18].

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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}	Occ. (<1)
Co1	0.16016(8)	0.61281(8)	0.36431(5)	0.0527(3)	
Co2	0.34145(7)	0.48814(7)	0.26741(4)	0.0392(2)	
Co3	0.45802(8)	0.37486(7)	0.15535(5)	0.0413(2)	
Cl1	0.4109(2)	1.04789(18)	0.12451(13)	0.0951(8)	
Cl2	0.4638(3)	−0.10166(18)	0.44141(13)	0.1047(8)	
O1	0.2676(4)	0.6514(3)	0.2774(2)	0.0489(12)	
O2	0.1254(6)	0.7758(5)	0.4501(3)	0.0861(19)	
O3	−0.1255(5)	0.7404(5)	0.4059(3)	0.101(2)	
O4	0.1413(4)	0.5000(3)	0.3186(2)	0.0499(12)	
O5	0.4135(4)	0.3265(3)	0.2544(2)	0.0425(11)	
O6	0.6839(5)	0.1871(4)	0.1070(3)	0.0646(14)	
O7	0.4156(5)	0.3001(4)	0.0369(3)	0.0620(14)	
O8	0.2906(4)	0.5068(3)	0.1662(2)	0.0479(12)	
O9	0.2904(4)	0.5092(4)	0.4375(2)	0.0638(14)	
O10	0.4352(4)	0.4300(3)	0.3631(2)	0.0496(12)	
O11	0.5348(4)	0.5157(3)	0.2275(2)	0.0481(12)	
O12	0.6144(4)	0.4447(4)	0.1396(2)	0.0521(13)	
N1	0.1756(6)	0.7499(5)	0.3864(3)	0.0660(19)	
N2	−0.0509(6)	0.6431(5)	0.3867(3)	0.072(2)	
N3	−0.0438(7)	0.2409(6)	0.2753(4)	0.082(2)	
N4	0.5904(5)	0.2185(4)	0.1611(3)	0.0482(15)	
N5	0.3683(5)	0.3869(5)	0.0686(3)	0.0494(15)	
C1	0.3048(7)	0.7385(6)	0.2463(4)	0.0510(19)	
C2	0.3612(6)	0.7543(6)	0.1780(4)	0.0519(18)	
H2	0.3775	0.6997	0.1569	0.062*	
C3	0.3929(7)	0.8468(6)	0.1413(4)	0.0583(19)	
H3	0.4270	0.8549	0.0957	0.070*	
C4	0.3742(7)	0.9287(6)	0.1721(5)	0.067(2)	
C5	0.3233(8)	0.9156(7)	0.2389(5)	0.072(2)	
H5	0.3123	0.9702	0.2595	0.086*	
C6	0.2872(7)	0.8226(6)	0.2773(4)	0.057(2)	
C7	0.2270(8)	0.8223(6)	0.3457(4)	0.067(2)	
H7	0.2255	0.8799	0.3617	0.081*	
C8	0.0525(9)	0.7033(8)	0.4916(4)	0.086(3)	
H8A	0.0590	0.7003	0.5395	0.103*	
H8B	0.1018	0.6327	0.4860	0.103*	
C9	−0.1008(9)	0.7290(8)	0.4766(5)	0.107(4)	
H9A	−0.1397	0.6728	0.5062	0.128*	
H9B	−0.1531	0.7951	0.4880	0.128*	
C9AA	0.1834(6)	0.5668(5)	0.1257(3)	0.0430(16)	
C10	−0.1402(7)	0.5949(7)	0.3764(4)	0.074(3)	
H10	−0.2369	0.6222	0.3879	0.088*	
C11	−0.1056(7)	0.5075(7)	0.3499(4)	0.061(2)	
C12	−0.2158(7)	0.4602(8)	0.3511(4)	0.082(3)	
H12	−0.3067	0.4919	0.3677	0.098*	
C13	−0.1994(7)	0.3719(8)	0.3299(4)	0.081(3)	
H13	−0.2742	0.3407	0.3357	0.098*	
C14	−0.0646(7)	0.3280(7)	0.2983(4)	0.063(2)	
C15	0.0467(6)	0.3765(6)	0.2934(4)	0.0553(19)	
H15	0.1335	0.3506	0.2710	0.066*	
C16	0.0308(6)	0.4611(6)	0.3206(3)	0.0513(18)	
C17	0.0853(9)	0.2006(7)	0.2361(6)	0.089(3)	
H17	0.1633	0.1436	0.2552	0.107*	
C18	0.0777(10)	0.2601(9)	0.1635(6)	0.131(4)	
H18A	0.1608	0.2276	0.1386	0.196*	
H18B	0.0753	0.3327	0.1590	0.196*	

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
H18C	−0.0081	0.2594	0.1451	0.196*	
C19	−0.1507(9)	0.1783(9)	0.2901(6)	0.116(4)	
H19A	−0.1332	0.1348	0.2581	0.139*	
H19B	−0.2471	0.2271	0.2830	0.139*	
C20	−0.1417(14)	0.1049(12)	0.3652(7)	0.217(7)	
H20A	−0.1575	0.0385	0.3659	0.325*	
H20B	−0.2140	0.1397	0.3940	0.325*	
H20C	−0.0480	0.0911	0.3823	0.325*	
C21	0.4297(6)	0.2292(5)	0.2963(4)	0.0413(16)	
C22	0.3649(6)	0.2138(6)	0.3622(4)	0.0535(18)	
H22	0.3132	0.2736	0.3769	0.064*	
C23	0.3741(7)	0.1141(6)	0.4065(4)	0.065(2)	
H23	0.3285	0.1071	0.4500	0.079*	
C24	0.4508(8)	0.0259(6)	0.3860(4)	0.065(2)	
C25	0.5206(7)	0.0359(6)	0.3233(4)	0.058(2)	
H25	0.5747	−0.0256	0.3112	0.069*	
C26	0.5142(6)	0.1355(5)	0.2763(4)	0.0475(17)	
C27	0.5921(6)	0.1362(5)	0.2118(4)	0.0447(17)	
H27	0.6493	0.0706	0.2063	0.054*	
C28	0.6739(7)	0.2752(6)	0.0490(4)	0.066(2)	
H28A	0.7643	0.2640	0.0224	0.079*	
H28B	0.6611	0.3384	0.0645	0.079*	
C29	0.5525(8)	0.2962(6)	0.0024(4)	0.078(3)	
H29A	0.5453	0.3638	−0.0325	0.094*	
H29B	0.5764	0.2405	−0.0213	0.094*	
C30	0.2535(7)	0.4558(6)	0.0406(4)	0.0522(19)	
H30	0.2251	0.4467	0.0011	0.063*	
C31	0.1654(6)	0.5447(5)	0.0637(3)	0.0428(16)	
C32	0.0497(6)	0.6137(6)	0.0216(3)	0.0518(18)	
H32	0.0361	0.5986	−0.0185	0.062*	
C33	−0.0419(6)	0.7007(6)	0.0376(4)	0.062(2)	
H33	−0.1147	0.7454	0.0076	0.074*	
C34	−0.0284(6)	0.7244(6)	0.0989(4)	0.059(2)	
C39	0.4005(6)	0.4409(5)	0.4222(4)	0.0475(18)	
C40	0.4973(7)	0.3681(5)	0.4801(3)	0.063(2)	
H40A	0.5698	0.3144	0.4635	0.095*	
H40B	0.4412	0.3344	0.5170	0.095*	
H40C	0.5428	0.4091	0.4967	0.095*	
C41	0.6193(6)	0.5024(5)	0.1779(3)	0.0446(16)	
C42	0.7381(6)	0.5567(6)	0.1616(4)	0.058(2)	
H42A	0.7007	0.6295	0.1348	0.087*	
H42B	0.8148	0.5195	0.1357	0.087*	
H42C	0.7743	0.5556	0.2038	0.087*	
C52	0.0850(6)	0.6562(5)	0.1415(4)	0.0507(18)	
H52	0.0954	0.6711	0.1822	0.061*	
N6	−0.1392(16)	0.7951(13)	0.1280(9)	0.055(5)	0.50
C35	−0.082(2)	0.8600(16)	0.1661(10)	0.070(5)	0.50
H35A	0.0220	0.8369	0.1706	0.084*	0.50
H35B	−0.1171	0.9371	0.1480	0.084*	0.50
C36	−0.167(3)	0.813(2)	0.2319(12)	0.136(11)	0.50
H36A	−0.2640	0.8577	0.2316	0.204*	0.50
H36B	−0.1209	0.8083	0.2723	0.204*	0.50
H36C	−0.1675	0.7422	0.2322	0.204*	0.50
C37	−0.251(2)	0.8669(18)	0.0837(12)	0.077(7)	0.50
H37A	−0.3297	0.8986	0.1119	0.093*	0.50
H37B	−0.2867	0.8258	0.0616	0.093*	0.50

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C38	−0.210(3)	0.958(3)	0.0268(15)	0.110(9)	0.50
H38A	−0.1631	0.9317	−0.0115	0.165*	0.50
H38B	−0.1461	0.9844	0.0449	0.165*	0.50
H38C	−0.2958	1.0152	0.0115	0.165*	0.50
N6′	−0.1045(16)	0.8246(13)	0.1070(9)	0.055(5)	0.50
C35′	−0.139(2)	0.812(2)	0.1943(13)	0.078(6)	0.50
H35C	−0.2412	0.8359	0.2053	0.093*	0.50
H35D	−0.0994	0.7386	0.2218	0.093*	0.50
C36′	−0.061(2)	0.8835(16)	0.2029(14)	0.105(8)	0.50
H36D	0.0357	0.8641	0.1841	0.157*	0.50
H36E	−0.0582	0.8775	0.2511	0.157*	0.50
H36F	−0.1095	0.9559	0.1790	0.157*	0.50
C37′	−0.220(3)	0.899(2)	0.0632(13)	0.074(7)	0.50
H37C	−0.2910	0.9422	0.0890	0.089*	0.50
H37D	−0.2682	0.8614	0.0441	0.089*	0.50
C38′	−0.145(3)	0.969(3)	0.0056(14)	0.133(11)	0.50
H38D	−0.2157	1.0202	−0.0264	0.200*	0.50
H38E	−0.0738	0.9250	−0.0183	0.200*	0.50
H38F	−0.1002	1.0069	0.0255	0.200*	0.50

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