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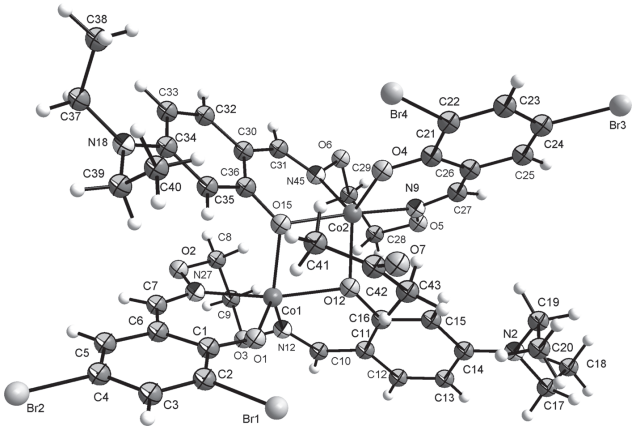
Crystal structure of bis{μ_2-2,4-dichloro-6-(8-(4-(diethylamino)-2-oxidophenyl)-3,6-dioxa-2,7-diazaocta-1,7-dien-1-yl)phenolato-$\kappa^5O{:}O,N,N',O'$}dicobalt(II) acetone solvate, $C_{43}H_{48}Br_4Co_2N_6O_9$



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Abstract

$C_{43}H_{48}Br_4Co_2N_6O_9$, triclinic, $P\bar{1}$, $a = 11.9298(7)$ Å, $b = 12.8850(9)$ Å, $c = 17.5241(11)$ Å, $\alpha = 84.854(6)°$, $\beta = 85.181(5)°$, $\gamma = 65.796(6)°$, $V = 2443.7(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0556$, $wR_{ref}(F^2) = 0.1053$, $T = 293.51(10)$ 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 (373.5 mg, 0.015 mmol) was added to a acetone solution (2.0 mL) of H_2L: 4-(diethylamino)-2-hydroxybenzaldehyde *O*-(2-(((3,5-dichloro-2-hydroxybenzylidene)amino)oxy)ethyl) oxime (3.96 mg, 0.010 mmol) at room temperature. The color of the solution turned brown immediately. After the mixture was stirred for 1 h, the mixture was filtered. The resulting filtrate was left undisturbed for about two weeks to form clear light brown block crystals. Yield: 58%. Elemental analysis-Anal. Calcd. for $C_{43}H_{48}Br_4Co_2N_6O_9$: C, 41.98%; H, 3.93%; N, 6.83%. Found: C, 42.18%; H, 3.91%; N, 6.80%.

Table 1: Data collection and handling.

Crystal:	Clear light brown block
Size:	0.24 × 0.25 × 0.23 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	4.01 mm^{-1}
Diffractometer, scan mode:	SuperNova ω
θ_{max}, completeness:	26.0°, 99.0%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17199, 9533, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5688
$N(param)_{refined}$:	583
Programs:	CrysAlis^{PRO} [18], OLEX2 [19], SHELX [20]

Experimental details

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

Discussion

Salen-type ligands and their derivatives have a significant position in the field of modern coordination chemistry as a kind of N_2O_2 tetradentate ligands [1–5]. They are capable of forming stable metal complexes [6–9], which may have unique properties and various structures, for example, they can show biological activity [10], form supramolecular architectures [11–13], or may construct optical and magnetic materials [14–16], and so on.

The crystal structure of the title compound is only built up by the $C_{43}H_{48}Br_4Co_2N_6O_9$ complex and one acetone molecule. The complex consists of two Co(II) atoms and two completely deprotonated L^{2-} units resulting in an asymmetrical binuclear Co(II) complex. The penta-coordinated Co(II) atom (Co1 or Co2) lies in the N_2O_2 coordination sphere (O1, O12, N12 and N27 or O4, O15, N9 and N45), and coordinates to another penolato oxygen atom (O12 or O15). The Co(II) atoms of the title compound can be described as trigonal bipyramidally coordinated with axial donor atoms (O12 and N27 or O15 and N5). The title compound has a "stepped" conformation as observed in the dimers of [{Cu(Brsalamo)}$_2$] [17]. The dimer is bridged through phenolato oxygen

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.11660(5)	0.53464(7)	0.82380(4)	0.0741(2)
Br2	0.14949(7)	0.33564(8)	0.54579(4)	0.1003(3)
Br3	0.04000(6)	1.35544(6)	0.98651(4)	0.0871(3)
Br4	0.06071(5)	1.13914(6)	0.71779(4)	0.0797(3)
Co1	0.48537(5)	0.60019(6)	0.75071(3)	0.03218(18)
Co2	0.45666(5)	0.84565(6)	0.77939(4)	0.03416(19)
O1	0.3538(2)	0.5482(3)	0.75941(17)	0.0377(9)
O2	0.6552(3)	0.5051(3)	0.60445(18)	0.0451(10)
O3	0.7421(3)	0.4144(3)	0.75681(19)	0.0443(10)
O4	0.3042(2)	0.9781(3)	0.77439(17)	0.0406(9)
O5	0.6294(3)	0.8767(3)	0.8882(2)	0.0581(12)
O6	0.6658(3)	0.9084(4)	0.7194(2)	0.0579(11)
O12	0.4615(2)	0.7046(3)	0.84035(16)	0.0334(8)
O15	0.4275(2)	0.7545(3)	0.69725(16)	0.0313(8)
N2	0.4074(4)	0.7756(4)	1.1071(2)	0.0503(13)
N9	0.5092(3)	0.9196(4)	0.8618(2)	0.0407(11)
N12	0.6491(3)	0.5053(3)	0.7980(2)	0.0369(11)
N18	0.2528(4)	0.7718(4)	0.4591(2)	0.0480(12)
N27	0.5465(3)	0.5128(3)	0.6482(2)	0.0356(10)
N45	0.5864(3)	0.8577(4)	0.6998(2)	0.0407(11)
C1	0.3133(4)	0.5021(4)	0.7111(3)	0.0325(12)
C2	0.2028(4)	0.4874(5)	0.7286(3)	0.0457(15)
C3	0.1563(5)	0.4378(5)	0.6810(3)	0.0569(17)
H3	0.0839	0.4287	0.6953	0.068*
C4	0.2176(5)	0.4015(5)	0.6117(3)	0.0548(16)
C5	0.3246(4)	0.4132(5)	0.5908(3)	0.0456(14)
H5	0.3651	0.3886	0.5439	0.055*
C6	0.3742(4)	0.4621(4)	0.6396(3)	0.0322(12)
C7	0.4897(4)	0.4680(4)	0.6132(3)	0.0363(13)
H7	0.5255	0.4364	0.5669	0.044*
C8	0.7256(4)	0.5469(5)	0.6441(3)	0.0457(15)
H8A	0.6699	0.6138	0.6701	0.055*
H8B	0.7767	0.5707	0.6069	0.055*
C9	0.8064(4)	0.4623(5)	0.7018(3)	0.0489(16)
H9A	0.8703	0.4010	0.6749	0.059*
H9B	0.8461	0.4993	0.7289	0.059*
C10	0.6736(4)	0.4957(4)	0.8699(3)	0.0403(13)
H10	0.7453	0.4350	0.8850	0.048*
C11	0.6014(4)	0.5690(4)	0.9271(3)	0.0352(13)
C12	0.6397(4)	0.5416(5)	1.0033(3)	0.0491(15)
H12	0.7098	0.4759	1.0136	0.059*
C13	0.5774(4)	0.6080(5)	1.0618(3)	0.0468(15)
H13	0.6053	0.5870	1.1112	0.056*
C14	0.4713(4)	0.7082(5)	1.0485(3)	0.0420(14)
C15	0.4340(4)	0.7387(4)	0.9723(3)	0.0362(13)
H15	0.3653	0.8056	0.9620	0.043*
C16	0.4977(4)	0.6709(4)	0.9125(3)	0.0330(12)
C17	0.4323(5)	0.7383(5)	1.1874(3)	0.0541(16)
H17A	0.4712	0.6557	1.1921	0.065*
H17B	0.3550	0.7623	1.2176	0.065*
C18	0.5145(5)	0.7854(5)	1.2196(3)	0.0684(18)
H18A	0.5918	0.7607	1.1905	0.103*
H18B	0.5282	0.7582	1.2722	0.103*
H18C	0.4755	0.8672	1.2164	0.103*
C19	0.2947(5)	0.8772(5)	1.0937(3)	0.0596(18)
H19A	0.3040	0.9147	1.0445	0.072*
H19B	0.2822	0.9297	1.1331	0.072*
C20	0.1828(5)	0.8499(6)	1.0941(4)	0.081(2)
H20A	0.1942	0.7987	1.0547	0.122*
H20B	0.1113	0.9189	1.0848	0.122*
H20C	0.1719	0.8146	1.1432	0.122*
C21	0.2544(4)	1.0614(4)	0.8202(3)	0.0390(13)
C22	0.1361(4)	1.1458(5)	0.8067(3)	0.0478(15)
C23	0.0733(5)	1.2325(5)	0.8548(3)	0.0534(16)
H23	−0.0054	1.2858	0.8442	0.064*
C24	0.1289(5)	1.2388(5)	0.9187(3)	0.0537(16)
C25	0.2469(5)	1.1639(5)	0.9325(3)	0.0508(15)
H25	0.2846	1.1719	0.9747	0.061*
C26	0.3116(4)	1.0752(4)	0.8839(3)	0.0386(13)
C27	0.4382(4)	1.0072(5)	0.8981(3)	0.0432(14)
H27	0.4720	1.0282	0.9368	0.052*
C28	0.7113(4)	0.7881(5)	0.8421(3)	0.0564(17)
H28A	0.6694	0.7417	0.8297	0.068*
H28B	0.7811	0.7397	0.8717	0.068*
C29	0.7581(5)	0.8306(6)	0.7684(3)	0.0640(19)
H29A	0.8096	0.8676	0.7812	0.077*
H29B	0.8097	0.7652	0.7397	0.077*
C30	0.4984(4)	0.8353(4)	0.5857(3)	0.0362(13)
C31	0.5784(4)	0.8665(4)	0.6260(3)	0.0411(13)
H31	0.6288	0.8956	0.5968	0.049*
C32	0.4921(4)	0.8593(5)	0.5060(3)	0.0472(15)
H32	0.5445	0.8898	0.4808	0.057*
C33	0.4125(4)	0.8398(5)	0.4643(3)	0.0460(15)
H33	0.4106	0.8577	0.4117	0.055*
C34	0.3328(4)	0.7925(5)	0.5005(3)	0.0398(14)
C35	0.3415(4)	0.7632(4)	0.5796(3)	0.0349(12)
H35	0.2927	0.7281	0.6039	0.042*
C36	0.4213(4)	0.7854(4)	0.6224(3)	0.0311(12)
C37	0.2414(5)	0.8050(5)	0.3772(3)	0.0559(17)
H37A	0.2024	0.7627	0.3550	0.067*
H37B	0.3232	0.7833	0.3528	0.067*
C38	0.1686(5)	0.9301(6)	0.3594(4)	0.082(2)
H38A	0.1686	0.9459	0.3048	0.123*
H38B	0.2052	0.9730	0.3821	0.123*
H38C	0.0855	0.9513	0.3798	0.123*
C39	0.1673(5)	0.7229(6)	0.4956(3)	0.070(2)
H39A	0.1463	0.6845	0.4575	0.084*
H39B	0.2087	0.6663	0.5357	0.084*
C40	0.0542(6)	0.8081(7)	0.5286(4)	0.109(3)
H40A	0.0057	0.8568	0.4882	0.164*
H40B	0.0744	0.8530	0.5615	0.164*
H40C	0.0083	0.7707	0.5578	0.164*
O7	−0.0526(4)	0.8951(5)	0.8385(3)	0.125(2)
C41	0.1093(5)	0.8234(6)	0.7462(4)	0.091(2)
H41A	0.0468	0.8249	0.7145	0.137*
H41B	0.1754	0.7489	0.7473	0.137*
H41C	0.1402	0.8788	0.7257	0.137*
C42	0.0567(5)	0.8504(6)	0.8244(4)	0.074(2)
C43	0.1389(6)	0.8254(8)	0.8871(4)	0.125(3)
H43A	0.0914	0.8389	0.9351	0.188*
H43B	0.1829	0.8736	0.8803	0.188*
H43C	0.1963	0.7470	0.8872	0.188*

atoms (O12 and O15) across the crystallographic center of asymmetry (Co1—O15 = 1.989(3) Å, Co1—O12 = 2.085(3) Å, Co2—O12 = 2.006(3) Å, Co2—O15 = 2.073(3) Å, Co2—Co1 = 3.121(4) Å, Co2—O12—Co1 = 99.41(13)°, Co2—O15—Co1 = 100.41(13)°, O12—Co1—O15 = 77.70(12)° and O12—Co2—O15 = 77.59(12)°).

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