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Crystal structure of *rac-trans*-6,6'-((cyclohexane-1,2-diylbis(azanylylidene))bis(methanylylidene))bis(2-bromophenolato- $\kappa^4 N, N', O, O'$)-bis(methanol)cobalt(III) chloride, $C_{22}H_{25}Br_2Co_8N_2O_4Cl$

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Abstract

$C_{22}H_{25}Br_2Co_8N_2O_4Cl$, monoclinic, $P2_1/c$ (no. 14), $a = 7.8246(13)$ Å, $b = 23.695(4)$ Å, $c = 13.225(2)$ Å, $\beta = 95.987(17)^\circ$, $V = 2438.6(7)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0844$, $wR_{ref}(F^2) = 0.2192$, $T = 173(2)$ 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

All hydrogen atoms were placed in calculated positions using a riding model. Methyl C–H bonds were fixed at 0.97 Å, with $U_{iso}(H)$ set to 1.2 $U_{eq}(C)$, and were allowed to spin about the

Table 1: Data collection and handling.

Crystal:	Block
Size:	0.10 × 0.22 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.12 mm ^{−1}
Diffraction, scan mode:	Xcalibur, Eos, Gemini, ϕ and ω -scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	9593, 4290, 0.093
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1747
$N(param)_{refined}$:	297
Programs:	CrysAlis ^{PRO} [1], SHELX [2], OLEX2 [3], DIAMOND [4]

C–C bond. Aromatic C–H distances were set to 0.93 Å and their U_{iso} set to 1.2 times the U_{eq} of the parent atom.

Experimental details

All commercially available reagents were used as supplied. A mixture of 3-bromo-2-hydroxybenzaldehyde (0.094 mmol) and (+)-1,2-cyclohexanediol (0.047 mmol) was dissolved in 30 mL of methanol and stirred for 30 min at room temperature, then 0.1 g cobalt nitrate hexahydrate was added and afforded an orange solution. After this, the purple suspension was stirred at 60 °C for 3 h and filtrated. The purple crystals of the title compound slowly appears after 2 weeks.

Discussion

Cobalt-based complexes are of interest because of their special biological and catalytic activities [5–8]. In this field, a large number of Schiff-base ligands have been used [9, 10]. However related research about halogenated Schiff base cobalt complexes is still rare [11–13]. As a part of our current interest in Schiff base complexes, we report here a new Co(III) compound.

The title compound crystallizes in the monoclinic space group $P2_1/c$. Thus the unit cell contains the two racemic cationic monomer complexes $[Co((+)-3-brsalcy)(CH_3OH)_2]$ and $[Co((-)-3-brsalcy)(CH_3OH)_2]$. The central Co^{III} is chelated by the tetradentate Schiff-base ligand *via* N2O2 in the equatorial plane and two oxygen atoms at the axial sites from methanol ligands. The bond lengths of Co–O(N) are in the range of 1.860(6)–1.948(5) Å and bond angles of O(N)–Co–O vary from 86.0(3) to 179.5(3)°. In addition, a chloride anion act as counterions, by which the cationic monomers are linked

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} */U _{eq}
Br1	0.25032(17)	0.22523(5)	0.66695(11)	0.0913(5)
Br2	0.63496(16)	0.27004(5)	0.97420(10)	0.0827(4)
Co1	0.42329(14)	0.41407(5)	0.74650(10)	0.0442(4)
O1	0.3403(7)	0.3460(2)	0.6897(4)	0.0507(19)
O2	0.5032(7)	0.3715(3)	0.8599(5)	0.0535(19)
O3	0.2078(6)	0.4243(2)	0.8058(5)	0.0528(19)
H3	0.1290(18)	0.4504(7)	0.793(3)	0.079*
O4	0.6369(7)	0.4033(2)	0.6886(5)	0.059(2)
H4	0.714(2)	0.4290(6)	0.678(3)	0.089*
N1	0.3448(8)	0.4570(3)	0.6326(5)	0.042(2)
N2	0.5006(9)	0.4835(3)	0.8057(5)	0.046(2)
C1	0.2535(10)	0.3389(4)	0.6036(7)	0.046(3)
C2	0.1987(11)	0.2824(4)	0.5740(8)	0.055(3)
C3	0.1082(12)	0.2742(5)	0.4826(9)	0.068(4)
H3A	0.0700	0.2370	0.4656	0.082*
C4	0.0687(13)	0.3155(5)	0.4139(8)	0.067(3)
H4A	0.0087	0.3068	0.3497	0.081*
C5	0.1144(11)	0.3693(4)	0.4367(7)	0.055(3)
H5	0.0855	0.3986	0.3890	0.066*
C6	0.2061(11)	0.3818(4)	0.5327(7)	0.047(3)
C7	0.2527(10)	0.4398(4)	0.5509(7)	0.047(3)
H7	0.2143	0.4669	0.5007	0.056*
C8	0.4040(13)	0.5162(4)	0.6401(8)	0.065(3)
H8	0.5237	0.5160	0.6200	0.077*
C9	0.3093(13)	0.5598(4)	0.5754(8)	0.068(3)
H9A	0.1861	0.5582	0.5862	0.082*
H9B	0.3190	0.5510	0.5031	0.082*
C10	0.3759(15)	0.6196(4)	0.5979(8)	0.082(4)
H10A	0.4886	0.6241	0.5710	0.098*
H10B	0.2951	0.6470	0.5624	0.098*
C11	0.3950(14)	0.6320(5)	0.7075(8)	0.076(4)
H11A	0.2791	0.6354	0.7306	0.091*
H11B	0.4524	0.6691	0.7183	0.091*
C12	0.4940(12)	0.5894(4)	0.7718(8)	0.059(3)
H12A	0.4884	0.5983	0.8446	0.071*
H12B	0.6160	0.5902	0.7582	0.071*
C13	0.4196(13)	0.5322(4)	0.7481(8)	0.061(3)
H13	0.2992	0.5339	0.7669	0.073*
C14	0.6084(11)	0.4878(4)	0.8836(7)	0.046(3)
H14	0.6434	0.5249	0.9035	0.055*
C15	0.6837(11)	0.4421(4)	0.9455(7)	0.049(3)
C16	0.8172(12)	0.4548(4)	1.0252(7)	0.057(3)
H16	0.8536	0.4928	1.0368	0.068*
C17	0.8902(12)	0.4133(5)	1.0831(7)	0.061(3)
H17	0.9792	0.4222	1.1352	0.073*
C18	0.8391(13)	0.3582(5)	1.0688(7)	0.061(3)
H18	0.8929	0.3297	1.1114	0.073*
C19	0.7129(12)	0.3437(4)	0.9947(8)	0.056(3)
C20	0.6266(11)	0.3857(4)	0.9285(7)	0.051(3)
C21	0.1407(12)	0.3790(4)	0.8608(8)	0.081(4)
H21A	0.1200	0.3462	0.8160	0.122*
H21B	0.0325	0.3907	0.8857	0.122*
H21C	0.2237	0.3689	0.9187	0.122*
C22	0.7154(13)	0.3472(4)	0.6853(9)	0.088(4)
H22A	0.6353	0.3215	0.6466	0.132*
H22B	0.7416	0.3329	0.7547	0.132*
H22C	0.8217	0.3498	0.6524	0.132*
Cl1	0.0864(3)	0.51218(12)	0.2974(2)	0.0641(8)

by classical hydrogen bonds: [O3—H3···Cl1] ($\text{'} = -1 + x, y, z$): 2.961(6) Å, O4—H4···Cl1 Å: 2.941(7) Å].

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