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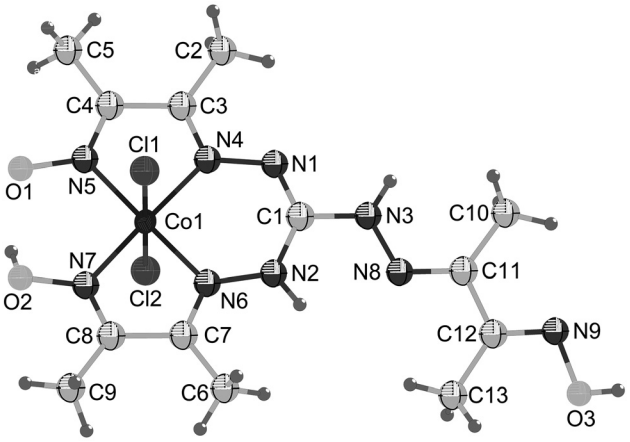
The crystal structure of dichlorido-[(*E*)-*N*′,*N*″-bis((2*E*,3*E*)-3-(hydroxyimino)butan-2-ylidene)-2-((*E*)-3-(hydroxyimino)butan-2-ylidene)hydrazine-1-carbohydrazonhydrazide-κ^4N^4]cobalt(II), $C_{13}H_{22}N_9O_3Cl_2Co$



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Abstract

$C_{13}H_{22}N_9O_3Cl_2Co$, monoclinic, $P2_1/n$ (no. 14), a = 7.7939(4) Å, b = 23.1095(11) Å, c = 11.7358(7) Å, β = 108.179(6)°, V = 2008.3 (2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0485, w$R_{ref}(F^2)$ = 0.1002, T = 293(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.10 × 0.05 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.16 mm^{-1}
Diffractometer, scan mode:	Multiwire proportional, φ and ω
θ_{max}, completeness:	25.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8592, 3743, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2,636
$N(param)_{refined}$:	261
Programs:	CrysAlis^{PRO},[1] Olex2,[2] SHELX[3,4]

1 Source of materials

A mixture of triaminoguanidine hydrochloride (10 mmol, 1.4 g), 2,3-butanedione monoxime (40 mmol, 4 g) were dissolved in methanol (50 mL) and stirred for 2 h at room temperature. Subsequently, reaction mixture was filtered, washed successively with water (10 mL) and methanol (10 mL). The residue was recrystallized in methanol so as to obtain the pure ligand compound (*E*)-*N*′,*N*″-bis((2*E*,3*E*)-3-(hydroxyimino) butan-2-ylidene)-2-((*E*)-3-(hydroxyimino)butan-2-ylidene)hydrazine-1-carbohydrazonhydrazide (L). Yield: 1.8 g (51 %, based on triaminoguanidine hydrochloride). The pure ligand L (1.4 mmol, 0.5 g) and cobalt chloride hexahydrate (5 mmol, 1.2 g) were dissolved in methanol (30 mL), and stired for 30 min. Next the mixture was filtered, and the filtrate was put aside for a few days under room temperature. And then the crystals of title compound were obtained. Yield: 0.3 g (43 %, based on ligand L). Anal. Calcd. for $C_{13}H_{22}N_9O_3Cl_2Co$ (%): C, 32.38; H, 4.60; N, 26.14; Cl, 14.70. Found: C, 32.25.; H, 4.61; N, 26.06; Cl, 14.51.

2 Experimental details

Crystallographic data collection and reduction were performed using the program CrysAlis^{PRO}.[1] Using Olex2,[2] the

structure was solved with the ShelXT[3] structure solution program and refined with the ShelXL[4] refinement package.

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

Atom	*X*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1552 (5)	0.96197 (16)	0.1822 (3)	0.0273 (8)
C2	−0.3700 (4)	0.94542 (16)	−0.0238 (3)	0.0337 (9)
H2B	−0.3170	0.9777	0.0264	0.051*
H2C	−0.4597	0.9278	0.0054	0.051*
H2D	−0.4254	0.9586	−0.1045	0.051*
C3	−0.2282 (4)	0.90265 (15)	−0.0216 (3)	0.0256 (8)
C4	−0.2642 (4)	0.84915 (16)	−0.0920 (3)	0.0271 (8)
C5	−0.4447 (4)	0.83051 (17)	−0.1711 (3)	0.0369 (10)
H5A	−0.4874	0.7988	−0.1343	0.055*
H5B	−0.4354	0.8184	−0.2472	0.055*
H5C	−0.5279	0.8622	−0.1827	0.055*
C6	0.6282 (4)	0.86120 (17)	0.2514 (3)	0.0378 (10)
H6A	0.6321	0.8502	0.3311	0.057*
H6B	0.6452	0.9023	0.2485	0.057*
H6C	0.7225	0.8416	0.2302	0.057*
C7	0.4500 (4)	0.84504 (16)	0.1654 (3)	0.0270 (8)
C8	0.4178 (5)	0.79082 (16)	0.0971 (3)	0.0303 (9)
C9	0.5564 (5)	0.74483 (17)	0.1109 (4)	0.0429 (10)
H9A	0.5145	0.7171	0.0473	0.064*
H9B	0.5770	0.7258	0.1867	0.064*
H9C	0.6670	0.7619	0.1074	0.064*
C10	0.2527 (5)	1.10877 (19)	0.3981 (4)	0.0528 (12)
H10A	0.1688	1.0878	0.4276	0.079*
H10B	0.1899	1.1255	0.3216	0.079*
H10C	0.3080	1.1389	0.4537	0.079*
C11	0.3944 (5)	1.06853 (17)	0.3850 (3)	0.0369 (10)
C12	0.5886 (5)	1.07928 (19)	0.4488 (4)	0.0443 (11)
C13	0.7323 (5)	1.0457 (2)	0.4196 (4)	0.0678 (15)
H13A	0.7510	1.0612	0.3485	0.102*
H13B	0.6961	1.0059	0.4063	0.102*
H13C	0.8426	1.0482	0.4852	0.102*
Cl1	0.02754 (12)	0.80243 (4)	0.17942 (8)	0.0384 (3)
Cl2	0.15298 (11)	0.89349 (4)	−0.12160 (8)	0.0364 (3)
Co1	0.09330 (6)	0.84768 (2)	0.03082 (4)	0.02527 (15)
N1	−0.0124 (4)	0.95627 (13)	0.1154 (2)	0.0289 (7)
N2	0.3072 (4)	0.92965 (13)	0.1957 (3)	0.0309 (7)
H2A	0.4090	0.9430	0.2403	0.037*
N3	0.1881 (4)	1.01059 (14)	0.2528 (3)	0.0362 (8)
H3	0.1011	1.0326	0.2566	0.043*
N4	−0.0590 (3)	0.90858 (12)	0.0435 (2)	0.0249 (7)
N5	−0.1192 (4)	0.81804 (13)	−0.0766 (3)	0.0311 (7)
N6	0.3053 (3)	0.87722 (13)	0.1419 (2)	0.0261 (7)
N7	0.2535 (4)	0.78679 (13)	0.0260 (3)	0.0302 (7)
N8	0.3642 (4)	1.02306 (14)	0.3176 (3)	0.0329 (7)
N9	0.6187 (5)	1.11998 (18)	0.5269 (3)	0.0608 (11)
O1	−0.1243 (3)	0.76766 (12)	−0.1305 (2)	0.0458 (7)
O2	0.2006 (3)	0.74037 (12)	−0.0432 (3)	0.0450 (7)
H2	0.0912	0.7418	−0.0761	0.067*
O3	0.8055 (4)	1.1279 (2)	0.5803 (3)	0.0900 (13)
H3A	0.8232	1.1533	0.6315	0.135*

Coordinates of hydrogen atoms were refined without any constraints or restraints. The U_{iso} values were set to be 1.5 U_{eq} of the carrier atom for methyl hydrogen atoms and 1.2 U_{eq} for aryl hydrogen atom.

3 Comment

As well-known, Schiff bases and their metal complexes possess good chemical and biological properties, which have been applied to many fields, such as analytical chemistry,[5–7] catalysis,[8–10] extraction metallurgy,[11] fluorescence,[12,13] magnetic properties,[14,15] medicine.[16–18] Moreover, many of cobalt complexes have been synthesized successfully in recent years, because of their good catalysis and magnetic performance.[19–21] Additionally, catalysis is becoming increasingly important in the field of new energy.[22] Herein, the ligand compound (*E*)-*N*′,*N*″-bis((2*E*,3*E*)-3-(hydroxyimino)butan-2-ylidene)-2-((*E*)-3-(hydroxyimino)butan-2-ylidene)hydrazine-1-carbohydrazonhydrazide (L) was synthesized by triaminoguanidine hydrochloride reacting with 2,3-butanedione monoxime.

The title compound crystallizes in the monoclinic crystal system with $P2_1/n$ space group. Its asymmetric unit contains one cobalt ion, one ligand molecule (L), and two chloride ions (the figure). The cobalt ion center shows a six-coordinated distorted octahedron geometry (CoN_4Cl_2) by four nitrogen atoms from ligand L and two chloride ions. The Co–N bond lengths are in the range of 1.871 (3) and 1.894 (3) Å. The Co–Cl bond lengths are in the range of 2.2260(10) and 2.2486(10) Å. The N–Co–N bond angles are in the range from 81.13 (13) to 178.70 (12)°. The Cl–Co–Cl bond angle is 178.69(4)°. The N–Co–Cl bond angles are in the range from 89.10 (9) to 91.46 (9)°. In the crystal structure, the title compound molecules are stacked by C–H···Cl interactions, C–H···O interactions and Van der Waals forces to extend into a three-dimensional supermolecular structure. All bond lengths and bond angles within the title structure are in the normal range.[23]

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