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Crystal structure of diaqua-bis(1,10-phenanthroline- κ^2N,N')-bis(1,7-dicarba-closo-dodecaborane-1,7-dicarboxylato- $\kappa^3O,O':O''$) dicobalt(II) — ethanol (1/1), $C_{34}H_{46}B_{20}Co_2N_4O_{11}$

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.40 × 0.31 × 0.16 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.75 mm ⁻¹
Diffractometer, scan mode:	SMART area detector, φ and ω
$\theta_{\max}$, completeness:	25.0°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6034, 4157, 0.018
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3319
$N(\text{param})_{\text{refined}}$:	337
Programs:	Bruker [1], SHELX [2, 3]

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Abstract

$C_{34}H_{46}B_{20}Co_2N_4O_{11}$, triclinic, $P\bar{1}$, $a = 10.372(11)$ Å, $b = 11.920(12)$ Å, $c = 12.141(13)$ Å, $\alpha = 61.136(11)^\circ$, $\beta = 85.699(10)^\circ$, $\gamma = 67.497(11)^\circ$, $V = 1203(2)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0410$, $wR_{\text{ref}}(F^2) = 0.1065$, $T = 298(2)$ K.

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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

Solvents, which were employed to prepare the title complex, were dried with appropriate drying agents and distilled

under dinitrogen prior to use. Dichloromethane for preparation was directly used without further purification. The 1,7-dihydroxycarbonyl-1,7-dicarba-closo-dodecaborane was prepared according to the literature method [4]. $Co(OAc)_2 \cdot 4H_2O$ (49.8 mg, 0.2 mmol) was added to a suspension of 1,7-(COOH)₂-1,7-C₂B₁₀H₁₀ (0.10 mmol) and 1,10-phenanthroline (37.0 mg, 0.2 mmol) in chloromethane (10 mL). The mixture was stirred for 6 h at room temperature under the protection of dry nitrogen. Then a colourless solution formed. Single crystals were obtained by slow evaporation of a dichloromethane-*n*-hexane solution. (32.4 mg, 22.9%). m.p. 198–205 K. FTIR (KBr) ν (cm⁻¹): 2981, 2961, 2922, 2863; 2613, 2602, 2581, 2553.

Experimental details

All H atoms were placed geometrically and treated as riding on their parent atoms, with B–H 1.10, C–H 0.96, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(B)$, $U_{\text{iso}}(H) = 1.5U_{\text{eq}}(C)$.

Comment

The coordination chemistry of functionalized carboranes is one of the most dynamic areas of modern research. These boron clusters have several properties, such as three-dimensional aromaticity, chemical inertness, thermal stability, and a hydrophobic character. The best known and most investigated carboranes are the three 12-vertex ortho, meta, and para isomers C₂B₁₀H₁₂, dicarbo-closo-dodecaboranes [5, 6]. The acidity of the terminal C–H bonds and the specific reactivity of the B–H groups in these boron clusters can be

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Co1	0.20159(4)	0.32482(4)	0.21053(4)	0.03287(14)
N1	0.2664(2)	0.1721(3)	0.4015(2)	0.0342(6)
N2	0.1107(2)	0.1861(3)	0.2255(2)	0.0346(6)
O1	0.2987(2)	0.4415(2)	0.2216(2)	0.0446(5)
O2	0.1234(2)	0.6470(3)	0.1675(3)	0.0596(7)
O3	0.6177(2)	0.7653(2)	−0.13833(18)	0.0415(5)
O4	0.7917(2)	0.5849(2)	0.00526(19)	0.0428(5)
O5	0.0091(2)	0.4720(2)	0.1948(2)	0.0434(5)
H5A	0.0363	0.5383	0.1766	0.075(14)*
H5B	−0.0653	0.5011	0.1461	0.063(12)*
O6	−0.0273(15)	0.6780(12)	0.3823(10)	0.187(5)
H6	0.0169	0.6531	0.3336	0.281*
C1	0.3437(3)	0.1666(4)	0.4883(3)	0.0446(8)
H1	0.3688	0.2413	0.4656	0.054*
C2	0.3884(4)	0.0550(4)	0.6106(3)	0.0553(9)
H2	0.4424	0.0553	0.6682	0.066*
C3	0.3524(4)	−0.0560(4)	0.6464(3)	0.0537(9)
H3	0.3821	−0.1319	0.7282	0.064*
C4	0.2701(3)	−0.0542(3)	0.5582(3)	0.0399(7)
C5	0.2298(3)	0.0625(3)	0.4366(3)	0.0308(6)
C6	0.1464(3)	0.0698(3)	0.3418(3)	0.0314(6)
C7	0.1047(3)	−0.0401(3)	0.3725(3)	0.0381(7)
C8	0.1469(4)	−0.1575(3)	0.4972(3)	0.0473(8)
H8	0.1198	−0.2305	0.5179	0.057*
C9	0.2256(4)	−0.1647(3)	0.5862(3)	0.0500(8)
H9	0.2514	−0.2425	0.6670	0.060*
C10	0.0226(3)	−0.0258(4)	0.2758(4)	0.0494(8)
H10	−0.0067	−0.0961	0.2912	0.059*
C11	−0.0138(4)	0.0906(4)	0.1600(4)	0.0542(9)
H11	−0.0685	0.1007	0.0959	0.065*
C12	0.0314(3)	0.1950(4)	0.1376(3)	0.0481(8)
H12	0.0051	0.2745	0.0579	0.058*
C13	0.2486(3)	0.5671(3)	0.1925(3)	0.0337(7)
C14	0.6689(3)	0.6764(3)	−0.0259(3)	0.0314(6)
C15	0.026(2)	0.5697(17)	0.5090(13)	0.172(7)
H15A	−0.0388	0.5914	0.5648	0.207*
H15B	0.1155	0.5654	0.5323	0.207*
C16	0.0445(17)	0.4326(15)	0.5285(12)	0.136(6)
H16A	0.1111	0.3607	0.6030	0.204*
H16B	0.0789	0.4243	0.4561	0.204*
H16C	−0.0442	0.4233	0.5393	0.204*
C17	0.3597(3)	0.6264(3)	0.1859(3)	0.0280(6)
C18	0.5745(3)	0.6800(3)	0.0760(3)	0.0282(6)
B1	0.4015(3)	0.7151(3)	0.0403(3)	0.0293(7)
H1A	0.3548	0.7337	−0.0485	0.035*
B2	0.4485(4)	0.8410(4)	0.0452(3)	0.0367(8)
H2A	0.4343	0.9406	−0.0404	0.044*
B3	0.5963(4)	0.7488(4)	0.1657(3)	0.0373(8)
H3A	0.6771	0.7902	0.1575	0.045*
B4	0.6426(3)	0.5663(4)	0.2329(3)	0.0326(7)
H4	0.7524	0.4903	0.2678	0.039*
B5	0.5228(3)	0.5449(3)	0.1561(3)	0.0293(7)
H5	0.5546	0.4540	0.1419	0.035*
B6	0.3046(4)	0.8044(4)	0.1204(3)	0.0359(8)
H6A	0.1948	0.8802	0.0849	0.043*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
B7	0.4251(4)	0.8255(4)	0.1975(4)	0.0445(9)
H7	0.3939	0.9165	0.2114	0.053*
B8	0.5445(4)	0.6560(4)	0.3136(4)	0.0430(9)
H8A	0.5905	0.6372	0.4027	0.052*
B9	0.4980(3)	0.5300(4)	0.3075(3)	0.0347(8)
H9A	0.5119	0.4296	0.3920	0.042*
B10	0.3640(4)	0.6896(4)	0.2854(3)	0.0388(8)
H10A	0.2919	0.6916	0.3562	0.047*

used to selectively functionalize both vertices, thereby generating a series of building blocks to be used in synthesis of various kinds of supermolecular materials. The structure is similar with the one in reference [7].

The compound crystallizes with one formula unit in the unit cell. The figure depicts the complex showing the atom numbering. The environment of the Cu atom is six coordinated, in which three positions are occupied by the three oxygen atoms of the carborate, one position is occupied by the oxygen atom of water molecule, and the other two positions are occupied by two nitrogen atoms. The bond lengths of Co1—O1, Co1—O3, Co1—O4 and Co1—O5 are 2.059 Å, 2.135 Å, 2.310 Å and 2.038 Å, respectively. The bond lengths of Co1—N1 and Co1—N2 are 2.089 Å and 2.129 Å, respectively.

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