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Crystal structure of catena-poly[aqua-(μ_2 -1,7-dicarba-closo-dodecaborane-1,7-dicarboxylato- $\kappa^2 O:O'$)-(1,10-phenanthroline- $\kappa^2 N,N'$)copper(II)], $C_{16}H_{20}B_{10}CuN_2O_5$

$V = 2307.4(4) \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.0583$, $wR_{\text{ref}}(F^2) = 0.1320$, $T = 298(2) \text{ K}$ [1–3].

CCDC no.: 1881270

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	$0.12 \times 0.10 \times 0.08 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	0.98 mm^{-1}
Diffractometer, scan mode:	CCD, φ and ω
θ_{max} , completeness:	25.0° , $>99\%$
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13596, 4058, 0.108
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1934
$N(\text{param})_{\text{refined}}$:	307
Programs:	Bruker [1], SHELX [2, 3]

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

Solvents, which were employed to prepare the title complexes, were dried with appropriate drying agents and distilled under nitrogen prior to use. Dichloromethane was directly used without further purification. The 1,7-dihydroxycarbonyl-1,7-dicarba-closo-dodecaborane was prepared according to the literature [4].

$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (34.1 mg, 0.2 mmol) was added to a suspension of 1,7-(COOH)₂-1,7- $\text{C}_2\text{B}_{10}\text{H}_{10}$ (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. Crystals were obtained by slow evaporation of dichloromethane-*n*-hexane (34.4 mg, 20.9%). m.p. 188–189 K. FTIR (KBr) ν (cm^{-1}): 2980, 2960, 2920, 2865 (C–H); 2610, 2600, 2582, 2552 (B–H).

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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{B})$, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

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Abstract

$C_{16}H_{20}B_{10}CuN_2O_5$, monoclinic, $P2_1/n$ (no. 14), $a = 7.4252(8) \text{ \AA}$, $b = 13.5409(14) \text{ \AA}$, $c = 23.020(3) \text{ \AA}$, $\beta = 94.4880(10)^\circ$,

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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}
Cu1	0.62348(9)	0.89743(4)	0.85985(3)	0.0483(2)
N1	0.6945(6)	1.0407(3)	0.8666(2)	0.0507(12)
N2	0.6744(5)	0.9041(3)	0.94820(17)	0.0435(11)
O1	0.5328(5)	0.7641(3)	0.86750(15)	0.0582(11)
O2	0.6874(6)	0.6861(3)	0.8024(2)	0.0846(14)
O3	0.5140(5)	0.9178(2)	0.78020(15)	0.0541(10)
O4	0.2428(6)	0.9233(3)	0.81478(17)	0.0697(12)
O5	0.8944(5)	0.8470(3)	0.83441(16)	0.0665(11)
H5C	0.8643	0.7960	0.8140	0.080*
H5D	0.9777	0.8777	0.8188	0.080*
B1	0.2359(10)	0.6169(5)	0.8675(3)	0.066(2)
H1	0.1835	0.6877	0.8825	0.079*
B2	0.4098(11)	0.5534(5)	0.9091(3)	0.070(2)
H2	0.4693	0.5833	0.9507	0.084*
B3	0.5420(10)	0.4888(5)	0.8592(3)	0.061(2)
H3	0.6879	0.4772	0.8690	0.073*
B4	0.4492(9)	0.5130(4)	0.7883(3)	0.0509(17)
H4	0.5342	0.5162	0.7513	0.061*
B5	0.2604(9)	0.5933(5)	0.7931(3)	0.0521(17)
H5	0.2231	0.6489	0.7594	0.062*
B6	0.0943(10)	0.5282(6)	0.8287(3)	0.065(2)
H6	−0.0510	0.5401	0.8180	0.078*
B7	0.1891(12)	0.5027(6)	0.9012(3)	0.087(3)
H7	0.1043	0.4986	0.9382	0.105*
B8	0.3792(12)	0.4236(6)	0.8961(3)	0.078(3)
H8	0.4187	0.3685	0.9299	0.094*
B9	0.4017(9)	0.4005(5)	0.8214(3)	0.0591(19)
H9	0.4539	0.3300	0.8059	0.071*
B10	0.1837(10)	0.4083(5)	0.8470(3)	0.069(2)
H10	0.0954	0.3433	0.8482	0.083*
C1	0.4442(7)	0.6005(4)	0.8412(2)	0.0467(13)
C2	0.2351(7)	0.4685(4)	0.7846(2)	0.0461(14)
C3	0.5689(8)	0.6912(4)	0.8365(3)	0.0536(15)
C4	0.3452(9)	0.9326(4)	0.7753(2)	0.0501(15)
C5	0.7106(8)	1.1078(4)	0.8245(2)	0.0565(15)
H5A	0.6839	1.0891	0.7860	0.068*
C6	0.7662(8)	1.2053(4)	0.8366(3)	0.0631(17)
H17	0.7779	1.2494	0.8062	0.076*
C7	0.8031(8)	1.2355(4)	0.8924(3)	0.0636(17)
H11	0.8364	1.3007	0.9002	0.076*
C8	0.7908(7)	1.1677(4)	0.9389(3)	0.0518(15)
C9	0.7375(7)	1.0702(4)	0.9232(2)	0.0432(13)
C10	0.7264(7)	0.9956(4)	0.9673(2)	0.0433(13)
C11	0.7683(7)	1.0192(4)	1.0262(2)	0.0479(14)
C12	0.8195(8)	1.1186(5)	1.0415(3)	0.0623(17)
H12	0.8451	1.1351	1.0805	0.075*
C13	0.8312(8)	1.1893(4)	0.9997(3)	0.0597(16)
H13	0.8662	1.2529	1.0108	0.072*
C14	0.7563(8)	0.9423(5)	1.0667(2)	0.0601(17)
H14	0.7832	0.9538	1.1063	0.072*
C15	0.7042(8)	0.8499(4)	1.0473(3)	0.0613(17)
H15	0.6971	0.7984	1.0737	0.074*
C16	0.6621(8)	0.8334(4)	0.9881(3)	0.0546(15)
H16	0.6240	0.7708	0.9759	0.066*

Comment

The polyhedral boron clusters “carboranes” have extensive applications in fundamental chemistry and applications as pharmaceuticals [5–8]. A series of amine-carboxyboranes and polyhedral hydroborate salts were tested for their anti-neoplastic-cytotoxic activity. Several carboxylate derivatives of Cu based on 1,2- or 1,7-dicarba-closo-dodecaborane have also been reported, in which the carborane cage is linked to the carboxylic moiety via a boron or carbon atom. Currently, the best known and most investigated carboranes are the three 12- vertex ortho, meta, and para isomers C₂B₁₀H₁₂, dicarbo-closo-dodecaboranes. Here, we report the copper coordination polymer with the dicarboxylate of 1,7-dihydroxycarbonyl-1,7-dicarba-closo-dodecaborane [9]. The structure is similar with the reference [4].

The figure depicts a perspective of a part of structure unit of the complex. The environment of the Cu atom is five coordinated, in which two positions are occupied by the two oxygen atoms of the ligand carborane, one position is occupied by the oxygen atom of the water molecule, and the other two position are occupied by the two nitrogen atoms of the 1,10-phenanthroline. The bond lengths of Cu1–O1, Cu1–O3 and Cu1–O5 are 1.941 Å, 1.966 Å and 2.245 Å, respectively. The bond lengths of Cu1–N1 and Cu1–N2 are 2.013 Å and 2.041 Å, respectively.

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