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A Novel One-Dimensional Copper(II) Complex of a Carboxymethylated Tricyclic Azacrown Ether Derivative Linked by Hydrogen Bonding

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A NOVEL ONE-DIMENSIONAL COPPER(II) COMPLEX OF A CARBOXYMETHYLATED TRICYCLIC AZACROWN ETHER DERIVATIVE LINKED BY HYDROGEN BONDING

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The complex $[\text{CuL}] \cdot \text{H}_2\text{O}$ ($\text{H}_2\text{L} = 4,13\text{-dioxo-}1,7,10,16\text{-tetraaza-}1,16:7,10\text{-bis(ethylene)-}9,18\text{-diacetic acid cyclo-octadecane-}8,17\text{-dione}$) has been synthesized and structurally characterized. It crystallizes in the triclinic system, space group $P1$ with $a = 9.0111(7)$, $b = 9.4371(7)$, $c = 14.3092(11)\text{\AA}$, $\alpha = 89.846(2)$, $\beta = 81.871(2)$, $\gamma = 65.616(2)^\circ$, $V = 1095.05(14)\text{\AA}^3$, and $Z = 1$. The structure of the complex consists of two different CuL coordination moieties in which six-coordinated copper ions each have different coordination environments. In both CuL moieties, copper ions are coordinated to two tertiary nitrogen and four oxygen atoms, two from the crown ether ring and two from the pendant carboxylate groups. These two different CuL moieties are linked alternately by water molecules to form an infinite one-dimensional chain structure through hydrogen bonding between the water molecule and the uncoordinated oxygen atom of the carboxyl group.

Keywords: Copper(II) complex; Tricyclic azacrown ether; One-dimensional chain; Crystal structure

INTRODUCTION

Macrocyclic ligands have long been employed for studies involving metal ion recognition [1]. The complexation properties of macrocyclic polyazapolycarboxylates have attracted considerable interest in recent years because the structural, equilibrium, and kinetic behaviour of their metal complexes differs considerably from that of linear analogues [2]. Some of these ligands have several potential applications, including their use as contrast agent for magnetic resonance imaging [3–5] and in the analytical separation of metal cations [6,7]. Stability constants for complexes of the first-row transition metal ions with some macrocyclic polyazapolycarboxylates are consonant with the Irving–Williams order of stability [8]. The high stability constant of the Cu^{2+} complex may be due to the special stabilization energy of its hexacoordinate complex resulting from Jahn–Teller distortion [9]. This series of ligands, having acetate groups as *N*-pendant arms, provides an opportunity to study the influence of the size of the macrocyclic ring and the number of donor atoms on the stability and selectivity of

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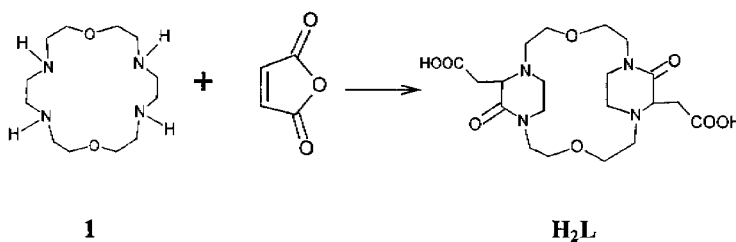
metal complexes [10]. Some X-ray crystal structure studies on copper complexes of polyazamacrocycles with functional carboxylate groups have been reported [11–13]. To investigate the effect of functional pendant arms on coordination behavior, we have prepared a copper complex with our newly-synthesized ligand, the carboxylic acid derivative of 1,10-dioxo-4,7,13,16-tetraazacyclooctadecane [14], and determined its crystal structure.

EXPERIMENTAL

Materials and Methods

All commercially available chemicals were of analytical grade and were used without further purification, except for toluene which was dried with sodium. C, H and N were determined using an Elementar vario EL instrument. Visible spectra in aqueous solution were recorded on a GBC Cintra 10e-UV-Visible spectrophotometer. IR spectra were recorded on a Nicolet-AVATAR 360 FT-IR spectrophotometer using KBr pellets in the 400–4000 cm^{-1} region. 1,10-Dioxo-4,7,13,16-tetraazacyclooctadecane (**1**) was prepared according to the literature [15,16].

Preparation of 4,13-Dioxo-1,7,10,16-tetraaza-1,16:7,10-bis(ethylene)-9,18-diacetic Acid Cyclooctadecane-8,17-dione (**H₂L**)



A solution of tetraazacrown ether (**1**) (0.26 g, 1 mmol) in dry toluene (20 cm^3) was added dropwise to a stirred solution of maleic anhydride (0.20 g, 2 mmol) in dry toluene (20 cm^3). A precipitate formed immediately and the heterogeneous mixture was stirred at room temperature for 7–8 h. The precipitate was collected by filtration, washed with acetone and dried. Yield 70%; m.p. 122–124°C. Elemental analysis and spectroscopic studies showed good agreement with the results of our previous work [14].

Preparation of the Complex

To a solution of the ligand **H₂L** (0.10 g, 0.2 mmol) in absolute ethanol (20 cm^3) was added with stirring a solution of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ (0.05 g, 0.2 mmol) in absolute ethanol (5 cm^3). Both ethanol solutions were dried with triethyl orthoformate for 5 h before mixing. A blue precipitate formed and the reaction mixture was refluxed for 4 h. The blue precipitate was filtered off, washed with CHCl_3 and dried. Some crystals suitable for X-ray determination were obtained on recrystallization from DMF. *Anal.* Calcd. for $\text{C}_{20}\text{H}_{32}\text{CuN}_4\text{O}_9$ ($M_r = 536.03$) (%): C, 44.82; H, 5.98; N, 10.46; found: C, 45.03; H, 5.31; N, 10.32.

TABLE I Crystal data and structure refinement details for $C_{40}H_{60}N_8O_{16}Cu_2 \cdot 2H_2O$

Empirical formula	$C_{40}H_{64}Cu_2N_8O_{18}$
Formula weight	1072.07
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, $P1$
Unit cell dimensions	$a = 9.0110(7)$ Å, $\alpha = 89.846(2)^\circ$ $b = 9.437(7)$ Å, $\beta = 81.872(2)^\circ$ $c = 14.3092(11)$ Å, $\gamma = 65.616(2)^\circ$
Volume	$1095.05(14)$ Å ³
Z, calculated density	1, 1.626 Mg/m ³
Absorption coefficient	1.059 mm ⁻¹
$F(000)$	562
Crystal size	$0.20 \times 0.10 \times 0.10$ mm
θ range for data collection	2.37 – 26.37°
Limiting indices	$-11 \leq h \leq 11$, $-11 \leq k \leq 11$, $0 \leq l \leq 17$
Reflection collected/unique	10513/4450 [$R(\text{int}) = 0.0467$]
Refinement method	Full-matrix least-squares on F^2
Goodness of fit on F^2	1.009
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0566$, $wR2 = 0.1184$
R indices (all data)	$R1 = 0.0919$, $wR2 = 0.1374$
Weighting scheme	$w = (\sigma^2(F_o^2) + (0.0592P)^2)^{-1}$; $P = (F_o^2 + 2F_c^2)/3$

X-ray Crystallography

A crystal of dimensions $0.20 \times 0.10 \times 0.10$ mm was selected for the X-ray diffraction study. Measurements were performed on a CCD area detector diffractometer at 296 K with graphite-monochromatized MoK α radiation ($\lambda = 0.71073$ Å). The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 using the SHELXS-97 and SHELXL-97 programs [17]. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were located by difference Fourier syntheses and refined isotropically. A summary of the crystallographic data and details of the structure refinement is given in Table I. Atomic coordinates and equivalent isotropic displacement parameters are listed in Table II.

RESULTS AND DISCUSSION

IR Spectra

A band at 1126 cm⁻¹ in the free ligand, assigned to ν (C–O–C), is shifted to 1113 cm⁻¹ in the complex, indicating the coordination of the ether oxygen atoms to the Cu²⁺ ion. No bands for free or coordinated nitrate were observed in the complex, implying that the carboxylic groups of the ligand are deprotonated on coordinating to Cu²⁺. Vibrations at 1571 and 1450 cm⁻¹ for deprotonated carboxylate anions in the free ligand were shifted to higher frequencies, 1611 and 1500 cm⁻¹ respectively, also showing the coordination of carboxylate ions to Cu²⁺.

Visible Absorption Spectra

Visible spectra of the copper complex in aqueous solution show an absorption maximum at 580 nm, in agreement with results for other six-coordinated copper(II) complexes with similar geometry [13]. The *trans* geometry of the two acetate side arms makes them favourable to bind metal ions in axial sites while retaining a strong

TABLE II Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (10^3 \AA^2) for $\text{C}_{40}\text{H}_{60}\text{N}_8\text{O}_{16}\text{Cu}_2 \cdot 2\text{H}_2\text{O}$

	x/a	y/b	z/c	U_{eq}
Cu(1)	5310(3)	145(3)	1606(2)	30(1)
Cu(2)	5310(3)	5143(3)	6598(2)	31(1)
C(1)	5168(16)	3679(18)	2125(12)	32(4)
C(2)	6694(16)	2568(17)	1408(11)	30(4)
C(3)	4912(19)	2760(20)	270(11)	43(4)
C(4)	3360(20)	3670(20)	964(16)	51(5)
C(5)	2190(20)	4660(20)	2661(14)	57(5)
C(6)	1850(20)	3270(20)	2952(14)	77(6)
C(7)	3161(19)	875(18)	3698(10)	37(3)
C(8)	3001(19)	-380(17)	3165(12)	34(4)
C(9)	5814(19)	-2468(14)	2906(11)	37(4)
C(10)	7350(20)	-3360(20)	2255(12)	41(4)
C(11)	4092(18)	-2352(18)	1793(11)	32(4)
C(12)	5550(20)	-3364(16)	1113(10)	32(4)
C(13)	2600(20)	-1386(19)	1347(12)	39(4)
C(14)	2710(20)	-200(20)	647(14)	48(5)
C(15)	8386(19)	-4365(15)	597(14)	49(5)
C(16)	8560(20)	-3270(20)	-53(14)	89(7)
C(17)	7670(30)	-640(20)	-322(13)	123(11)
C(18)	7600(20)	760(20)	63(13)	54(5)
C(19)	8069(15)	1733(18)	1991(11)	29(3)
C(20)	7800(20)	487(17)	2569(12)	34(4)
C(21)	2981(17)	7890(16)	7860(9)	24(3)
C(22)	1649(13)	8178(16)	7206(12)	32(4)
C(23)	4505(19)	8544(17)	6576(10)	37(4)
C(24)	3530(20)	8410(20)	5882(14)	45(5)
C(25)	940(20)	7980(20)	5736(15)	56(5)
C(26)	1190(20)	6420(20)	5654(13)	62(6)
C(27)	3150(20)	3750(20)	5589(16)	54(6)
C(28)	4903(16)	2770(20)	5277(13)	39(4)
C(29)	6182(17)	1719(17)	6680(13)	48(5)
C(30)	7270(20)	1831(18)	7376(12)	41(4)
C(31)	9034(19)	2107(15)	5940(10)	32(4)
C(32)	7745(18)	2292(18)	5353(13)	39(4)
C(33)	7860(20)	3403(18)	4588(10)	30(3)
C(34)	7660(20)	4975(15)	4936(13)	38(4)
C(35)	9710(20)	2208(19)	7531(10)	38(4)
C(36)	9505(18)	3958(19)	7487(15)	60(6)
C(37)	7598(19)	6450(20)	7557(15)	48(5)
C(38)	5830(20)	7460(20)	7890(16)	59(6)
C(39)	2840(20)	6930(20)	8638(13)	45(5)
C(40)	2997(14)	5342(17)	8351(11)	33(4)
N(1)	3647(17)	4033(16)	1895(10)	34(3)
N(2)	6185(12)	1598(15)	816(9)	27(3)
N(3)	4496(16)	-1377(15)	2455(8)	33(3)
N(4)	7021(16)	-3804(17)	1344(1)	41(4)
N(5)	1969(15)	8293(15)	6348(9)	29(3)
N(6)	4619(13)	7392(10)	7308(8)	26(3)
N(7)	8659(15)	2017(16)	6937(10)	39(3)
N(8)	6012(13)	2866(15)	5951(10)	39(4)
O(1)	5377(14)	4124(12)	2857(8)	44(3)
O(2)	5299(12)	-3784(15)	336(8)	45(3)
O(3)	7992(13)	-1819(12)	300(7)	63(3)
O(4)	3379(10)	2027(11)	3101(7)	48(2)
O(5)	8774(19)	-5(18)	3133(11)	74(5)
O(6)	6723(15)	33(13)	2465(9)	44(3)
O(7)	1734(15)	364(17)	100(11)	63(4)

(continued)

TABLE II Continued

	x/a	y/b	z/c	U_{eq}
O(8)	3934(16)	208(17)	676(9)	54(4)
O(9)	328(14)	8183(15)	7611(10)	55(4)
O(10)	10358(11)	2079(13)	5614(7)	35(3)
O(11)	2831(13)	5353(13)	5496(11)	56(4)
O(12)	7829(14)	4886(15)	7760(9)	48(3)
O(13)	2328(14)	4616(14)	8862(8)	46(3)
O(14)	3813(14)	4714(13)	7517(9)	43(3)
O(15)	8296(16)	5622(13)	4405(9)	54(4)
O(16)	6815(14)	5547(14)	5728(9)	41(3)
O(17)	7413(18)	8814(16)	4768(11)	59(4)
O(18)	3250(20)	1444(16)	8512(10)	63(4)

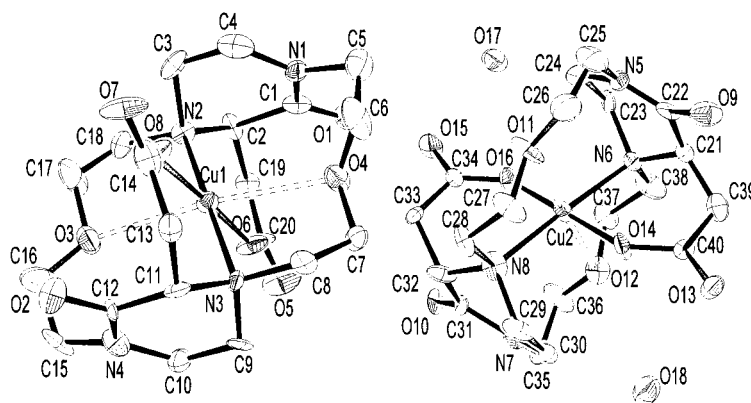


FIGURE 1 An ORTEP-3 drawing (35% probability ellipsoids) of the complex with atom numbering scheme.

ligand field in the plane of the ring. This agreement was verified by the position of the $d-d$ absorption band (mentioned above) and the crystal structure determination of the complex (see below).

Description of Structure

The structure of the copper complex is shown in Fig. 1, and selected bond lengths and angles are listed in Table III. X-ray structure analysis revealed that the structure consists of two different copper coordination moieties, CuL(1) and CuL(2), with a distance of 8.570 Å between Cu(1) and Cu(2) centres. In both CuL moieties, Cu(II) ions are six-coordinated with two nitrogen atoms and two oxygen atoms from the crown ether ring, and two oxygen atoms from the two pendant acetate groups in an *anti* fashion.

The two CuL moieties have different bond distances although they have the same coordination geometry. Distances in CuL(1) are 2.101(13), 2.165(13), 1.864(12), 1.928(12), 2.795(13), and 2.679(12) Å for Cu1–N2, Cu1–N3, Cu1–O6, Cu1–O8, Cu1–O3, and Cu1–O4, respectively. By contrast, the bond distances in CuL(2) are 2.160(9), 2.145(13), 1.913(12), 1.865(13), 2.854(12), and 2.930(13) Å for Cu2–N6, Cu2–N8, Cu2–O14, Cu2–O16, Cu2–O11, and Cu2–O12, respectively. It is worthy of note that the mean Cu–O distance for the acetate oxygen atoms is much shorter than those

TABLE III Selected bondlengths (Å) and angles (°) for $C_{40}H_{60}N_8O_{16}Cu_2 \cdot 2H_2O$

Cu(1)–O(6)	1.864(12)	Cu(1)–O(8)	1.928(12)
Cu(1)–N(2)	2.101(13)	Cu(1)–N(3)	2.165(13)
Cu(1)–O(3)	2.795(13)	Cu(1)–O(4)	2.679(13)
Cu(2)–O(14)	1.913(12)	Cu(2)–O(16)	1.865(13)
Cu(2)–N(6)	2.160(9)	Cu(2)–N(8)	2.145(13)
Cu(2)–O(11)	2.854(12)	Cu(2)–O(12)	2.930(13)
C(6)–O(4)	1.43(2)	C(7)–O(4)	1.440(18)
C(14)–O(7)	1.21(2)	C(14)–O(8)	1.32(2)
C(16)–O(3)	1.320(19)	C(17)–O(3)	1.38(2)
C(20)–O(5)	1.23(2)	C(20)–O(6)	1.236(19)
C(12)–O(2)	1.260(17)	C(22)–O(9)	1.246(17)
C(26)–O(11)	1.39(2)	C(27)–O(11)	1.42(2)
C(34)–O(15)	1.20(2)	C(34)–O(16)	1.25(2)
C(36)–O(12)	1.394(19)	C(37)–O(12)	1.44(2)
O(6)–Cu(1)–O(8)	177.4(8)	O(6)–Cu(1)–N(2)	88.6(5)
O(8)–Cu(1)–N(2)	90.4(5)	O(6)–Cu(1)–N(3)	89.0(5)
O(8)–Cu(1)–N(3)	92.0(5)	N(2)–Cu(1)–N(3)	177.5(6)
O(16)–Cu(2)–O(14)	178.3(8)	O(16)–Cu(2)–N(8)	92.8(5)
O(14)–Cu(2)–N(8)	87.5(5)	O(16)–Cu(2)–N(6)	88.7(5)
O(14)–Cu(2)–N(6)	90.9(5)	N(8)–Cu(2)–N(6)	177.5(7)
C(18)–N(2)–Cu(1)	114.2(10)	C(2)–N(2)–Cu(1)	113.2(9)
C(3)–N(2)–Cu(1)	113.6(8)	C(9)–N(3)–Cu(1)	111.2(9)
C(11)–N(3)–Cu(1)	107.4(9)	C(8)–N(3)–Cu(1)	108.6(9)
C(21)–N(6)–Cu(2)	109.1(8)	C(38)–N(6)–Cu(2)	114.4(9)
C(23)–N(6)–Cu(2)	108.3(7)	C(29)–N(8)–Cu(2)	110.5(10)
C(28)–N(8)–Cu(2)	114.9(9)	C(32)–N(8)–Cu(2)	110.2(8)
C(20)–O(6)–Cu(1)	139.4(11)	C(14)–O(8)–Cu(1)	134.8(13)
C(40)–O(14)–Cu(2)	134.4(10)	C(34)–O(16)–Cu(2)	136.6(10)

for the crown ether donors. This may be attributed to the low strain of the relevant six-membered chelate rings. Distances between copper ions and crown ether oxygen atoms are considerably longer than others, giving an elongated octahedral geometry. This may be due to extra strain arising from the bending of the ring and also the Jahn-Teller effect of $Cu^{2+}(d^9)$. This six-coordinated geometry contrasts with other octahedral copper(II) complexes in which the pendant carboxyl oxygen atoms occupy elongated axial sites [11–13].

The crown ether ring is twisted to meet requirements of coordination geometry. For CuL(1), the six donors of the crown ring, N1, N2, N3, N4, O3 and O4, are nearly coplanar (plane 1) with a mean deviation 0.154 Å from the least-squares plane. For CuL(2), the six donors N5, N6, N7, N8, O11 and O12, are perfectly coplanar (plane 2) with a mean deviation 0.017 Å. The dihedral angle of 88.6° between planes 1 and 2, shows that the two crown ring planes are nearly perpendicular to each other. The two copper centres lie in the crown ether planes, so that each Cu^{2+} ion is encapsulated by the crown ether ring and the two side-arms.

In addition, each CuL is linked to an adjacent moiety by hydrogen bonding between the uncoordinated oxygen atoms of the carboxyl groups and water molecules (Table IV). The H7 atom of the water molecule (indicated as O17) forms a hydrogen bond O17–H7···O15 with the O15 atom from CuL(2), while the other hydrogen atom H8 of the same water molecule forms another hydrogen bond O17–H8···O5ⁱ [symmetry code: $x, y+1, z$] with an adjacent moiety. The other water molecule (indicated as O18) forms two other hydrogen bonds O18–H6···O13 [O13 also from CuL(2)] and O18–H5···O7ⁱⁱ [symmetry code: $x, y, z+1$]. Thus, the water oxygen

TABLE IV Hydrogen bond parameters^a

$D-H \cdots A$	$H \cdots A$ (Å)	$D \cdots A$ (Å)	$D-H \cdots A$ (°)
O17-H7...O15	2.215	2.809	138.8
O17-H8...O5 ⁱ	1.676	2.919	168.4
O18-H5...O7 ⁱⁱ	2.172	2.886	166.4
O18-H6...O13	1.704	2.782	167.5

^aSymmetry code: ⁱ $x, y+1, z$; ⁱⁱ $x, y, z+1$.

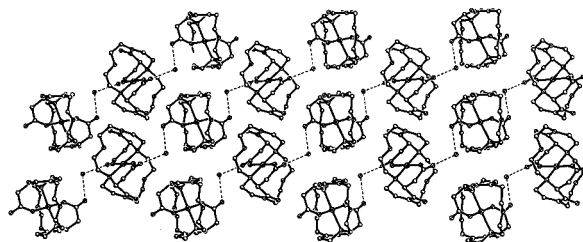


FIGURE 2 The one-dimensional chain coordination network linked by hydrogen bonding (viewed along the a axis; hydrogen atoms are omitted for clarity).

atoms O17 and O18 link the two moieties, CuL(1) and CuL(2), alternately through hydrogen bonding to form an infinite one-dimensional hydrogen-bond system (Fig. 2).

Acknowledgments

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