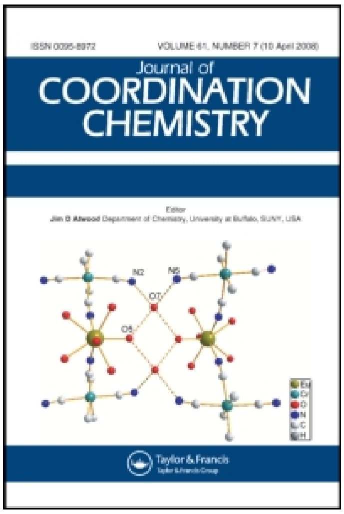

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New $Ni^{II}Cu^{II}Ni^{II}$ and $Ni^{II}Co^{II}Ni^{II}$ trinuclear complexes with macrocyclic oxamido ligands

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New trinuclear complexes $[Cu(NiL^1)_2(C_2H_5OH)_2](ClO_4)_2$ (**1**) and $[Co(NiL^2)_2(H_2O)_2](ClO_4)_2 \cdot 2(H_2O)$ (**2**) were prepared using macrocyclic complex ligands with excess $Cu(ClO_4)_2 \cdot 6(H_2O)$ and $Co(ClO_4)_2 \cdot 6(H_2O)$, respectively, and characterized by X-ray crystallography. L^1 and L^2 denote the dianions of diethyl 5,6,7,8,15,16-hexahydro-6,7-dioxodibenzo[1,4,8,11]tetraazacyclotetradecine-13,18-dicarboxylate and diethyl 5,6,7,8,15,16-hexahydro-15-methyl-6,7-dioxodibenzo[1,4,8,11]tetraazacyclotetradecine-13,18-dicarboxylate, respectively. In each complex, Ni(II) has the same distorted N4 square planar coordination. In **1**, Cu(II) resides in a distorted octahedral O6 coordination environment, and the trinuclear cations are linked by oxamido–carbonyl interactions and $\pi \cdots \pi$ interactions involving unclosed π-systems to form 1-D supramolecular chains. In **2**, Co(II) also resides in a distorted octahedral O6 coordination environment, while the trinuclear cations are linked by O–H $\cdots$ O hydrogen bonds and $\pi \cdots \pi$ interactions involving unclosed π-systems resulting in 1-D supramolecular chains.

Keywords: Synthesis; Crystal structure; Intermolecular interaction; Trinuclear complex; Macrocyclic ligand

1. Introduction

Design and synthesis of polynuclear complexes have been fertile areas of research in supramolecular chemistry [1–4], bioinorganic chemistry [5–7], materials chemistry, and catalysis [8–12]. One strategy to synthesize polynuclear complexes is the "complex as ligand" approach [13–17]. In this article, two similar mononuclear complexes of macrocyclic oxamido Schiff bases, NiL^1 and NiL^2 (scheme 1), were used as "complex ligands" to synthesize two new trinuclear complexes, $[Cu(NiL^1)_2(C_2H_5OH)_2](ClO_4)_2$ (**1**) and $[Co(NiL^2)_2(H_2O)_2](ClO_4)_2 \cdot 2(H_2O)$ (**2**). L^1 and L^2 denote the dianions of diethyl 5,6,7,8,15,16-hexahydro-6,7-dioxodibenzo[1,4,8,11]tetraazacyclotetradecine-13, 18-dicarboxylate and diethyl 5,6,7,8,15,16-hexahydro-15-methyl-6,7-dioxodibenzo[1,4, 8,11]tetraazacyclotetradecine-13,18-dicarboxylate, respectively. These ligands can be involved in rich non-covalent interactions, including oxamido–carbonyl interactions [15, 16], $\pi \cdots \pi$ stacking and hydrogen-bonding [15–18], and therefore could extend molecular units into supramolecular networks.

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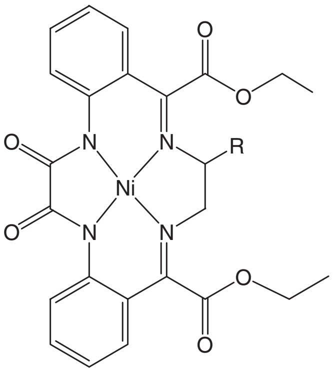

Scheme 1. NiL^1: R = H; NiL^2: R = CH_3.

2. *Experimental*

All starting chemicals were of reagent grade and used as purchased. The macrocyclic complex ligands NiL^1 and NiL^2 were prepared by the literature methods [18, 19]. IR spectra were recorded as KBr pellets on a BIO-RAD 3000 infrared spectrophotometer from 4000 to 400 cm^{-1}. Elemental analyses were performed on a Perkin-Elmer 240 analyzer.

Caution! Although we have not encountered any problems, it should be kept in mind that perchlorate salts of metal complexes are potentially explosive and should be handled with care.

2.1. *Synthesis of* $[Cu(NiL^1)_2(C_2H_5OH)_2](ClO_4)_2$ *(1)*

The mixture of NiL^1 (0.096 mmol), $Cu(ClO_4)_2 \cdot 6H_2O$ (0.288 mmol) and C_2H_5OH (30 mL) was stirred at 70°C for 10 min. The resulting solution was then cooled to room temperature and filtered. The filtrate was evaporated slowly at room temperature for 28 days, and orange crystals were obtained. Yield: 31.1%. Anal. Calcd for $C_{52}H_{56}Cl_2CuN_8Ni_2O_{22}$: C, 44.71%; H, 4.04%; N, 8.02%. Found: C, 44.58%; H, 4.14%; N, 7.95%. IR (KBr, cm^{-1}): 3507(br), 1736(s), 1620(m), 1603(s), 1575(s), 1558(s), 1485(s, w), 1444(m), 1344(s), 1218(s), 1116(s), 1090(s), 749(w), 623(w).

2.2. *Synthesis of* $[Co(NiL^2)_2(H_2O)_2](ClO_4)_2 \cdot 2(H_2O)$ *(2)*

The mixture of NiL^2 (0.097 mmol), $Co(ClO_4)_2 \cdot 6H_2O$ (0.195 mmol), and C_2H_5OH (30 mL) was stirred at 70°C for 10 min. The resulting solution was then cooled to room temperature and filtered. The filtrate was stored at room temperature for a month and red crystals were obtained. Yield: 41.0%. Anal. Calcd for $C_{50}H_{56}Cl_2CoN_8Ni_2O_{24}$: C, 42.89%; H, 4.03%; N, 8.00%. Found: C, 43.00%; H, 4.11%; N, 8.05%. IR (KBr, cm^{-1}): 3525(br), 1732(s), 1602(s), 1582(s), 1560(s), 1486(s, w), 1445(m), 1348(s), 1220(s), 1121(s), 1089(s), 1047(s), 751(w), 625(w).

2.3. *X-ray structure determinations*

X-ray intensities were measured at 293 K with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) on a Rigaku RAXIS RAPIS IP diffractometer for **1** and on a SMART APEX II CCD area detector for **2**. Crystal structures were solved using direct methods and successive Fourier difference syntheses (SHELXS-97), and refined using the full-matrix least-squares method on F^2 with anisotropic displacement parameters for all non-hydrogen atoms (SHELXL-97) [20]. Hydrogens were added theoretically and refined with riding model positional parameters and fixed isotropic displacement parameters. The crystal parameters, data collections and refinements for **1** and **2** are given in table 1. Selected bond lengths and angles are listed in table 2 for **1** and table 3 for **2**.

3. Results and discussion

3.1. *Syntheses*

Complexes **1** and **2** were prepared in the presence of considerable excess of metal salts ($Cu(ClO_4)_2 \cdot 6H_2O$ for **1** and $Co(ClO_4)_2 \cdot 6H_2O$ for **2**. When the theoretical molar ratio

Table 1. Crystal data and structure refinements for **1** and **2**.

Compound	**1**	**2**
Emperical formula	$C_{52}H_{56}Cl_2CuN_8Ni_2O_{22}$	$C_{50}H_{56}Cl_2CoN_8Ni_2O_{24}$
M_r	1396.88	1400.24
Color and habit	Orange, block	Red, block
Crystal system	Monoclinic	Triclinic
Space group	$P21/c$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	10.944(2)	10.8580(9)
b	12.663(3)	10.9433(9)
c	21.381(4)	13.8651(11)
α		69.1940(10)
β	101.73(3)	83.4280(10)
γ		82.3290(10)
Volume (Å^3), Z	2901.3(10), 2	1522.3(2), 1
Calculated density (g cm^{-3})	1.597	1.527
Absorption coefficient (mm^{-1})	1.184	1.055
$F(000)$	1434	721
Crystal size (mm^3)	$0.71 \times 0.19 \times 0.12$	$0.20 \times 0.14 \times 0.12$
θ range for data collection (°)	3.22–27.51	1.90–25.03
Limiting indices	$-14 \le h \le 4$, $-16 \le k \le 16$, $-27 \le l \le 27$	$-12 \le h \le 12$, $-13 \le k \le 13$, $-11 \le l \le 16$
Reflections collected	23,627	8350
Unique reflections	6487 [R(int) = 0.0675]	5317 [R(int) = 0.0331]
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Refinement methods	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	6487/25/404	5317/83/442
Goodness-of-fit on F^2	1.018	0.914
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0611$, $wR_2 = 0.1676$	$R_1 = 0.0607$, $wR_2 = 0.1794$
R indices (all data)	$R_1 = 0.0887$, $wR_2 = 0.1969$	$R_1 = 0.1045$, $wR_2 = 0.1909$
Largest difference peak and hole (e Å^{-3})	0.655, −0.605	0.691, −0.391

Table 2. Selected bond lengths (Å) and angles (°) for **1**.

Cu–O(1)	1.973(3)	N(2)–C(1)	1.333(5)
Cu–O(2)	2.233(3)	N(2)–C(10)	1.413(5)
Cu–O(3)	2.055(4)	N(3)–C(5)	1.279(5)
Ni(1)–N(1)	1.886(3)	N(3)–C(6)	1.479(6)
Ni(1)–N(2)	1.869(3)	N(4)–C(8)	1.293(5)
Ni(1)–N(3)	1.873(3)	N(4)–C(7)	1.482(6)
Ni(1)–N(4)	1.865(3)	C(1)–O(2)	1.236(5)
N(1)–C(2)	1.326(5)	C(2)–O(1)	1.255(5)
N(1)–C(3)	1.410(5)	C(1)–C(2)	1.528(6)
O(1)–Cu–O(2)	79.05(12)	N(4)–Ni(1)–N(3)	87.24(15)
O(1)–Cu–O(3)	90.95(15)	C(1)–N(2)–C(10)	121.9(3)
O(2)–Cu–O(3)	86.93(15)	C(1)–N(2)–Ni(1)	111.4(3)
O(1)–Cu–O(1)^i	180.00(0)	C(10)–N(2)–Ni(1)	126.7(3)
O(1)–Cu–O(2)^i	100.95(12)	C(2)–N(1)–C(3)	121.7(3)
O(1)–Cu–O(3)^i	89.05(15)	C(2)–N(1)–Ni(1)	109.3(3)
O(2)–Cu–O(2)^i	180.00(0)	C(3)–N(1)–Ni(1)	128.2(3)
O2–Cu–O(3)^i	93.07(15)	C(5)–N(3)–C(6)	119.1(4)
O3–Cu–O(3)^i	180.00(0)	C(5)–N(3)–Ni(1)	128.4(3)
N(2)–Ni(1)–N(1)	86.97(14)	C(6)–N(3)–Ni(1)	112.4(3)
N(2)–Ni(1)–N(3)	178.21(15)	C(7)–N(4)–Ni(1)	110.3(3)
N(3)–Ni(1)–N(1)	93.62(15)	C(8)–N(4)–C(7)	121.1(4)
N(4)–Ni(1)–N(1)	173.81(15)	C(8)–N(4)–Ni(1)	128.5(3)
N(4)–Ni(1)–N(2)	92.36(15)		

Symmetry code ^{i}: $1-x$, $-y$, $-z$.

Table 3. Selected bond lengths (Å) and angles (°) for **2**.

Co(1)–O(1)	2.088(4)	N(2)–C(2)	1.325(7)
Co(1)–O(2)	2.084(4)	N(2)–C(3)	1.403(8)
Co(1)–O(7)	2.082(5)	N(3)–C(9)	1.286(8)
Ni(1)–N(1)	1.889(5)	N(3)–C(13)	1.533(9)
Ni(1)–N(2)	1.878(5)	N(4)–C(14)	1.484(9)
Ni(1)–N(3)	1.866(6)	N(4)–C(15)	1.272(8)
Ni(1)–N(4)	1.878(5)	O(1)–C(1)	1.246(7)
N(1)–C(1)	1.331(7)	O(2)–C(2)	1.247(6)
N(1)–C(24)	1.384(8)	C(1)–C(2)	1.509(9)
N(2)–Ni(1)–N(1)	86.6(2)	O(7)–Co(1)–O(7)^i	180.0(3)
N(2)–Ni(1)–N(4)	179.0(2)	C(1)–N(1)–C(24)	121.3(5)
N(3)–Ni(1)–N(1)	175.8(2)	C(1)–N(1)–Ni(1)	109.2(4)
N(3)–Ni(1)–N(2)	92.7(2)	C(24)–N(1)–Ni(1)	127.3(4)
N(3)–Ni(1)–N(4)	87.8(2)	C(2)–N(2)–C(3)	121.5(5)
N(4)–Ni(1)–N(1)	93.0(2)	C(2)–N(2)–Ni(1)	110.3(4)
O(1)–Co(1)–O(2)	78.80(16)	C(3)–N(2)–Ni(1)	126.9(4)
O(1)–Co(1)–O(7)	88.55(18)	C(9)–N(3)–C(13)	119.8(6)
O(1)–Co(1)–O(1)^i	180.0(2)	C(9)–N(3)–Ni(1)	130.0(5)
O(1)–Co(1)–O(7)^i	91.45(18)	C(13)–N(3)–Ni(1)	110.1(5)
O(1)–Co(1)–O(2)^i	101.20(16)	C(15)–N(4)–C(14)	119.2(6)
O(2)–Co(1)–O(7)	88.69(18)	C(15)–N(4)–Ni(1)	130.2(4)
O(2)–Co(1)–O(2)^i	180.0(0)	C(14)–N(4)–Ni(1)	110.6(5)
O(2)–Co(1)–O(7)^i	91.31(18)		

Symmetry code ^{i}: $-x+1$, $-y-1$, $-z+1$.

2 : 1 (complex ligand to salt) was adopted, only crystals of NiL^1 or NiL^2 were obtained, as observed in the preparation of other multinuclear complexes having NiL^i-like complex ligands [15, 16, 21]. Although the role of the excess metal salts is still not clear, the strategy is effective in achieving stable solutions and crystals of the products.

3.2. *Crystal structures*

Complex **1** crystallizes with the centrosymmetric trinuclear complex dication $[Cu(NiL^1)_2(C_2H_5OH)_2]^{2+}$ (figure 1a) and two perchlorate ions, and **2** crystallizes with the centrosymmetric trinuclear complex dication $[Co(NiL^2)_2(H_2O)_2]^{2+}$ (figure 1b), two disordered perchlorate ions and two disordered water molecules. The oxamido group from each of the two macrocyclic ligands in a trinuclear complex dication bridges M(II) (Cu(II) for **1** and Co(II) for **2** and the corresponding Ni(II)). Nickel resides in distorted N4 square-planar environment with short Ni–N bond lengths (1.865–1.889 Å, tables 2 and 3). Deviations of the four donors and the Ni center from their mean plane are in the ranges −0.071 to 0.072 Å in **1** and −0.047 to 0.036 Å in **2**. Both Cu(II) and Co(II) have distorted octahedral O6 coordination with four O donors from two oxamido groups of two macrocyclic ligands and two C_2H_5OH ligands or two H_2O ligands. The Cu–O distances are in the range 1.973–2.233 Å, from the Jahn–Teller effect, whereas the Co–O

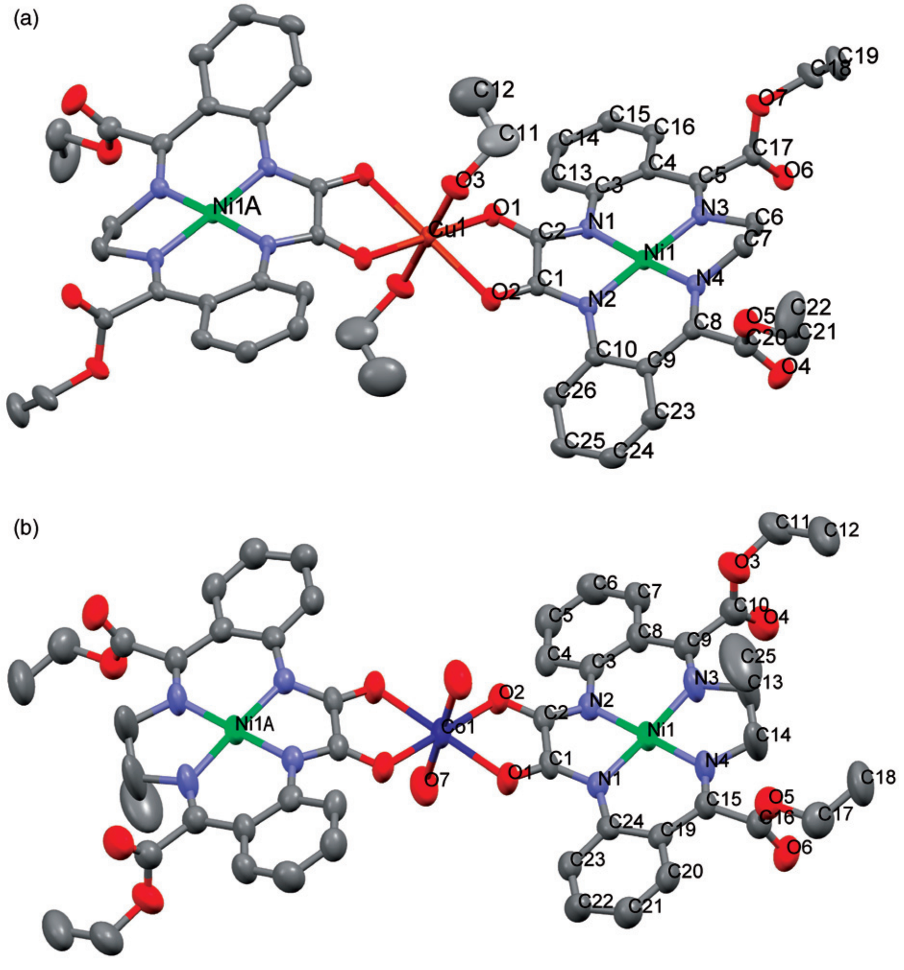

Figure 1. (a) Structures of $[Cu(NiL^1)_2(C_2H_5OH)_2]^{2+}$ in **1** and (b) $[Co(NiL^2)_2(H_2O)_2]^{2+}$ in **2**.

Table 4. Contact distances (Å) between neighboring trinuclear cations for **1**.

O(6)$\cdots$C(1)^i	3.048(5)	C(10)$\cdots$C(16)^i	3.537(6)
O(6)$\cdots$C(2)^i	3.191(5)	N(1)$\cdots$C(5)^i	3.786(7)
Ni(1)$\cdots$C(4)^i	3.650(4)	N(2)$\cdots$C(16)^i	3.687(6)
Ni(1)$\cdots$C(16)^i	3.692(4)	N(3)$\cdots$C(3)^i	3.752(7)
C(8)$\cdots$C(15)^i	3.331(6)	N(4)$\cdots$C(14)^i	3.696(7)
C(9)$\cdots$C(15)^i	3.329(7)	N(4)$\cdots$C(15)^i	3.597(6)

Symmetry code ^{i}: $1-x$, $1-y$, $-z$.

Table 5. Contact distances (Å) between neighboring trinuclear cations for **2**.

O(6)$\cdots$O(7)^i	2.804(7)	C(4)$\cdots$C(20)^i	3.351(10)
Ni(1)$\cdots$N(1)^i	3.800(5)	C(4)$\cdots$C(21)^i	3.503(11)
Ni(1)$\cdots$C(23)^i	3.787(8)	C(8)$\cdots$C(21)^i	3.577(10)
Ni(1)$\cdots$C(24)^i	3.684(7)	C(8)$\cdots$C(22)^i	3.425(10)
C(3)$\cdots$C(20)^i	3.560(10)	N(2)$\cdots$C(19)^i	3.708(9)
C(3)$\cdots$C(21)^i	3.523(10)	N(3)$\cdots$C(23)^i	3.644(9)

Symmetry code ^{i}: $1-x, -y, 1-z$.

distances vary narrowly between 2.082 and 2.088 Å. The intramolecular Ni$\cdots$Ni distances are 10.399 Å for **1** and 10.452 Å for **2**. The intramolecular Cu$\cdots$Ni and Co$\cdots$Ni distances are 5.199 and 5.226 Å, respectively. The three metals in each trinuclear dication are linear. The N–C bonds (1.325–1.333 Å) in the oxamido groups are not only longer than the N=C double bonds (N(3)–C(5) and N(4)–C(8) in **1**, N(3)–C(9) and N(4)–C(15) in **2**: 1.272–1.293 Å), but also shorter than the N–C single bonds (N(3)–C(6), N(4)–C(7) in **1** and N(3)–C(13), N(4)–C(14) in **2**: 1.479–1.533 Å). The sum of the three bond angles around each amidate nitrogen is close to 360° (357.8°–360.0°). These data reveal sp^2-hybridized character of the amidate nitrogens and delocalization of the π-electrons on these atoms and the carbonyls. The C–C bonds in both oxamido groups (C(1)–C(2) = 1.528 Å in **1** and C(1)–C(2) = 1.509 Å in **2**) are longer than normal single bonds indicating that each oxamido group is divided into two π-systems, each of which includes a carbonyl and an amidate nitrogen. The C=N groups of the Schiff bases have not been involved in conjugated systems because the C–C and C–N bonds around them have normal single bond lengths.

In **1** and **2**, each NiL^i ($i = 1$ or 2) fragment of a trinuclear cation overlaps with a NiL^i fragment of a neighboring trinuclear cation in close distances (tables 4 and 5). Some of the atom-to-atom distances between two overlapping NiL^i fragments are in the range of $\pi\cdots\pi$ interactions (<3.80 Å) [18, 22]. These $\pi\cdots\pi$ interactions involve unclosed π-systems (see the above paragraph) and belong to the same type as found between the NiL^i-like mononuclear and polynuclear complexes [15–18]. In **1**, the oxygen of a carbonyl O(6) from each of the two overlapping NiL^1 fragments contacts C(1) and C(2) (the two carbons of the oxamido group) from the other of the two overlapping fragments with distances as short as 3.048(5) and 3.191(5) Å, which are less than the sum of the van der Waals radii of oxygen and carbon (3.22 Å) [23]. Such short O$\cdots$C contacts have also been found in other polynuclear complexes of NiL^i-like complex ligands and was termed oxamido–carbonyl interaction [15, 16]. The $\pi\cdots\pi$ interactions and oxamido–carbonyl interactions link the trinuclear cations to form 1-D

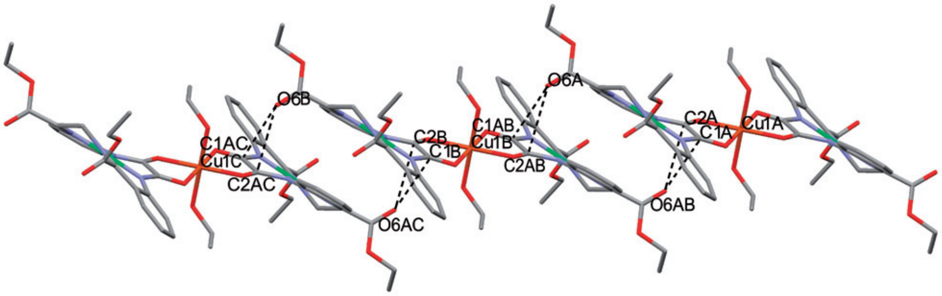

Figure 2. Supramolecular 1-D chain of trinuclear cations in **1**.
The dotted lines show the intermolecular contacts between the ester carbonyl oxygen atoms and the carbon atoms of the oxamido groups.

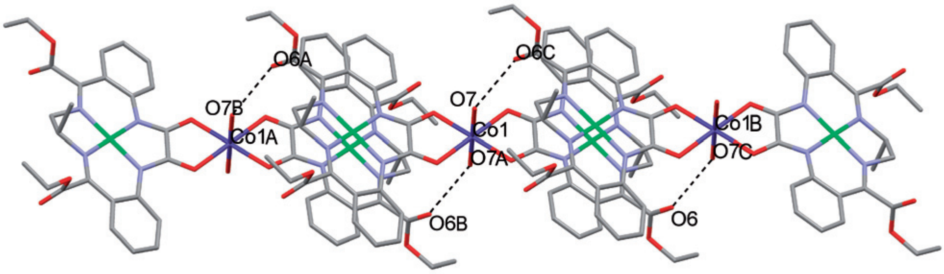

Figure 3. Supramolecular 1-D chain of the trinuclear cations in **2**.
The dotted lines show the intermolecular hydrogen bonds between the ester carbonyl oxygen atoms and the H_2O ligands.

supramolecular chains in crystals of **1** (figure 2). In **2**, there exist no such short $O\cdots C$ contacts. However, the distance between O(6) in one of the two ester carbonyls of each NiL^2 fragment and O(7) from a H_2O ligand of a neighboring trinuclear dication is as short as 2.804 Å, and therefore $O–H\cdots O$ hydrogen bonds are formed ($O(7)\cdots O(6)^i$ 2.804(7) Å, $H(7A)\cdots O(6)^i$ 2.132 Å, $O(7)–H(7A)\cdots O(6)^i$ 135.50°. Symmetry code ^{i}: $1-x$, $-y$, $1-z$). The $\pi\cdots\pi$ interactions and $O–H\cdots O$ hydrogen bonds link the trinuclear cations into 1-D supramolecular chains in **2** (figure 3). It is likely that oxamido–carbonyl interactions and $O–H\cdots O$ hydrogen bonds are competitive, and occur in **1** and **2**, respectively, owing to the weaker donor ability of CH_3CH_2OH than H_2O for hydrogen bonding.

In **1**, C_2H_5OH forms one $O–H\cdots O$ hydrogen bond with a perchlorate ($O(3)\cdots O(9)$ 2.722(9) Å). Except the $O(7)–H(A)\cdots O(6)$ mentioned in the last paragraph, other $O–H\cdots O$ hydrogen bonds also exist in **2** ($O(7)\cdots O(9')^i$ 2.731(15) Å, $H(7B)\cdots O(9')^i$ 1.898 Å, $O(7)–H(7B)\cdots O(9')^i$ 165.16°; $O(7)\cdots O(11)^i$ 2.778(17) Å, $H(7B)\cdots O(11)^i$ 2.210 Å, $O(7)\text{-}H(7B)\cdots O(11)^i$ 123.99°. Symmetry code ^{i}: x, $-1+y$, z).

The bands at 1620 and 1603 cm^{-1} in the IR spectrum of **1** and that at 1603 cm^{-1} in the spectrum of **2** can be assigned to the absorption of the oxamido C=O groups that coordinate to metal [17]; two bands in this area in the IR spectrum of **1** implies that the environment of the two oxamido C=O groups are different. The band at 1620 cm^{-1} should be attributed to the shorter C=O bond (C(1)–O(2) 1.236(5) Å) involving the longer Cu–O bond (Cu–O(2) 2.233(3) Å) and the band at 1603 cm^{-1} to the longer oxamido C=O bond (C(2)–O(1) 1.255(5) Å) involving the shorter Cu–O bond (Cu–O(1) 1.973(3) Å). Bands at 1736 and 1732 cm^{-1} in **1** and **2**, respectively, are attributed to $\nu_{(C=O)}$ (ester). The broad bands at *ca* 1090 cm^{-1} for **1** and 1089 cm^{-1} for **2** are attributed to ClO_4^-.

4. Conclusions

Excess metal salts were needed in the preparation of single crystals of the two new trinuclear complexes. This strategy may find applications in preparing other quasistable solutions and crystalline complexes. Compared with "non-complex ligands" [24–28], the stable mononuclear macrocyclic complexes used in this article are easy to offer heteronuclear complexes. A notable characteristic of the trinuclear complexes formed from this type of complex ligands is the effectiveness of the special $\pi \cdots \pi$ interactions in organizing the trinuclear complex cations into 1-D supramolecular chains with oxamido–carbonyl interactions or O–H $\cdots$ O hydrogen bonds. Competition between oxamido–carbonyl interactions and O–H $\cdots$ O hydrogen bonds was observed in formation of 1-D supramolecular chains.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 684639 for **1** and 684640 for **2**. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.uk or http://www.ccdc.cam.ac.uk).

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