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Self-assembly and crystal structure of a novel hetero-octametallic molecular box (Chelate complexes, Part 23) ☆

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Abstract

Reaction of hexadentate ligand *m*-xylene-bis-(iminodiethanol) H_4L^2 (**4**) with lithium hydride and either copper(II) *acetate* or copper(II) *chloride* resulted in the formation of two completely different products. Starting from copper(II) *acetate* the green bis-dimetallic cyclophane $[Cu_4(H_2L^2)_2(OAc)_4]\cdot 2CH_2Cl_2$ (**5**) was formed. However, when **4** was allowed to react with copper(II) *chloride*, the novel hetero-octametallic molecular box $\{[Li_4(Cu_4O)(L^2)_2](H_2O)_2(EtOH)_2\}Cl_2$ (**6**) was isolated. Deep violet **6** can be described as two metallacrown ether sub-units linked together by a central μ_4-bridging O^{2-}-ion. The structures of all new compounds were determined unequivocally by single crystal X-ray analyses.

Keywords: Supramolecular chemistry; Copper; Self-assemblies; Coordination chemistry; Oligomers

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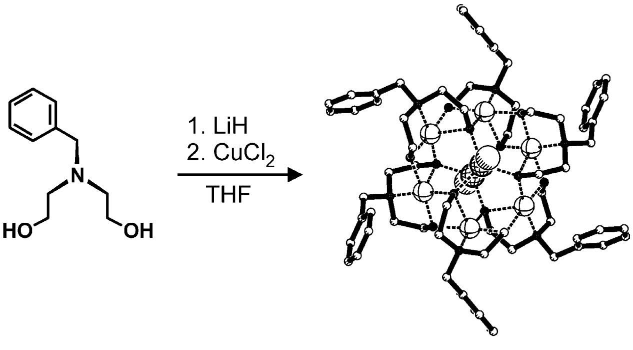
1. Introduction

Design and synthesis of supramolecular inorganic architectures exhibiting novel properties provide exciting new opportunities (for recent reviews, see [1–24]). In particular, oxo-bridged transition metal clusters proved to be valuable materials for versatile physical studies [25–44]. Recently, we reported on the self-assembly of six- and eight-membered iron-coronates as well as iron-coronands, accessible in one-pot reactions from iron(III) salts, N-substituted diethanolamines and alkali or alkaline earth bases [45–47]. Additionally, along these lines a series of hexanuclear copper wheels, forming exohedral bis-adducts with alkalimetal halides, successfully were prepared. The host–guest compound $[(LiCl)_2\cap Cu_6(L^1)_6]$ (**2**), for instance, was obtained with *N*-benzyliminodiethanol H_2L^1 (**1**), lithium hydride and copper(II) chloride and subsequent extraction of the crude precipitate with dimethyl sulfoxide (Scheme 1) [48,49].



Scheme 1. Synthesis of copper wheel **2**.

☆ Part 22: Ref. [59].

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2. Results and discussion

Unexpectedly, extraction of the aforementioned crude precipitate with nitromethane instead of DMSO produced dimetallic $[Cu_2(HL^1)_2Cl_2]$ (**3**) rather than copper wheel **2**, as was shown by an X-ray structure analysis (Scheme 2).

The linkage of two metal centers with two *mono*-diethanolamine ligands prompted us to generate bis-dimetallic cyclophanes with *bis*-diethanolamine ligands. Therefore we allowed to react hexadentate *m*-xylene-bis-(iminodiethanol) H_4L^2 (**4**) with lithium hydride and copper(II) acetate and isolated a green material **5** (Scheme 3).

For unambiguous characterization we carried out an X-ray crystallographic structure analysis of **5** (Fig. 1). According to this analysis, **5** is present in the crystal as bis-dimetallic cyclophane $[Cu_4(H_2L^2)_2(OAc)_4] \cdot 2CH_2Cl_2$. In **5**, each copper center is coordinated by one nitrogen, one hydroxyl and two μ_2-O donors of ligand $(H_2L^2)^{2-}$. The square pyramidal coordination sphere at each copper center is completed by an acetate donor. Additional hydrogen bonds are formed between the OH groups of ligand $(H_2L^2)^{2-}$ and the acetate co-ligand. Two methylene chloride solvent molecules are associated to the OH groups of $(H_2L^2)^{2-}$ across their hydrogen atoms (mean distance H–O: 2.09 and 2.33 Å) (Fig. 1).

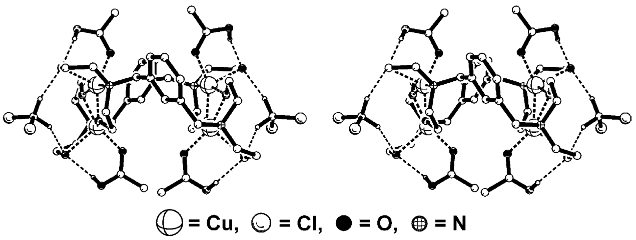
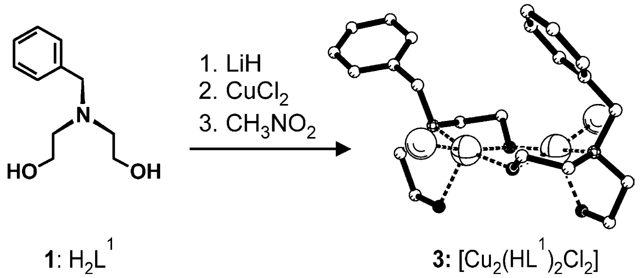
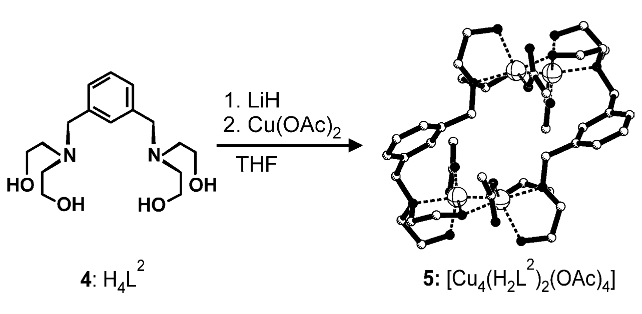


Fig. 1. Stereo view of $[Cu_4(H_2L^2)_2(OAc)_4] \cdot 2CH_2Cl_2$ (**5**). Hydrogen atoms are omitted, except those involved in hydrogen bonds.

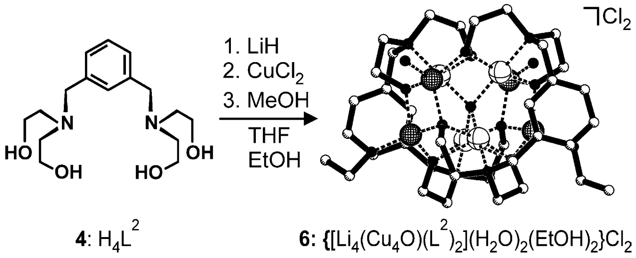
In contrast to **5**, available from H_4L^2 **4** and copper(II) *acetate*, with copper(II) *chloride* after work-up the totally different complex **6** was isolated. Deep violet **6** was identified on the basis of a single-crystal X-ray structure determination as the novel hetero-octametallic assembly $\{[Li_4(Cu_4O)(L^2)_2](H_2O)_2(EtOH)_2\}Cl_2$ (Scheme 4).



Scheme 2. Synthesis of dimetallic **3**.



Scheme 3. Synthesis of bis-dimetallic **5**.



Scheme 4. Synthesis of hetero-octametallic molecular box **6**.

Although **6** represents quite an elaborate system, its "building plan" is rather straightforward. Principally, **6** is composed of two metallacrown ether sub-units of general composition $[Cu_2Li_2(L^2)]^{2+}$ (Fig. 2, left). In this fragment, two lithium and two copper(II) cations are linked by four μ_2-oxygen donors to give an eight-membered ring. Putting two of these sub-units together and with concurrently placing an extra μ_4-bridging O^{2-}-anion in the center leads to the iconogram $[Li_4(Cu_4O)O_8]^{2+}$ (Fig. 2, right). This formal assembly results in a change of the μ_2-bridging oxygen donors of module $[Cu_2Li_2(L^2)]^{2+}$ into μ_3-bridging ones in $[Li_4(Cu_4O)O_8]^{2+}$.

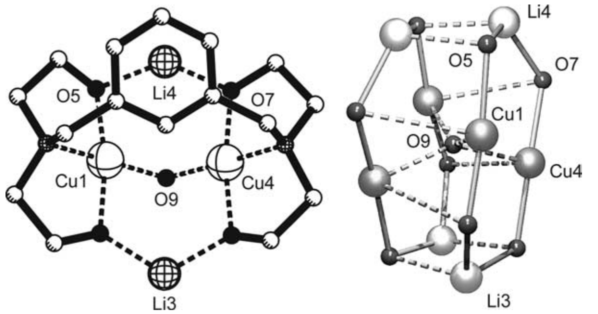


Fig. 2. (Left) Connectivity of sub-unit $[Cu_2Li_2(L^2)]^{2+}$ with the numbering of the metal and oxygen centers together with central μ_4-bridging O9. (Right) Schematic presentation of the linkage of the two sub-units across one central μ_4- and eight μ_3-O centers. For clarity only the metal and oxygen skeleton of the complex framework is depicted.

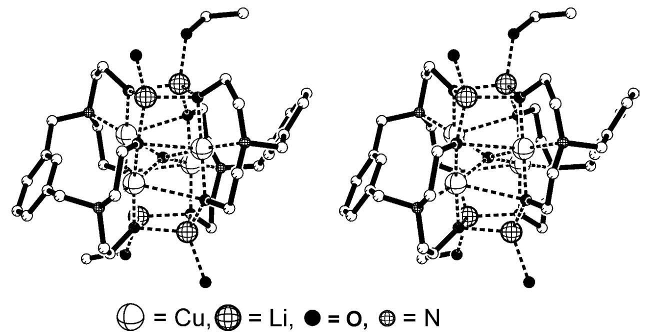


Fig. 3. Stereo view of dication $\{[Li_4(Cu_4O)(L^2)_2](H_2O)_2(EtOH)_2\}^{2+}$ of **6**. H atoms are not depicted for clarity.

The square pyramidal coordination sphere at each copper center comprises one nitrogen, one μ_4- and three μ_3-oxygen donors. The tetrahedral coordination sphere at the lithium centers is composed of three μ_3-oxygen donors and completed by aquo or ethanol ligands. The overall two positive charges of $\{[Li_4(Cu_4O)(L^2)_2](H_2O)_2(EtOH)_2\}^{2+}$ are compensated by two extra chloride anions which are not in close contact with the supramolecular core. Thus, the octametallic box $\{[Li_4(Cu_4O)(L^2)_2](H_2O)_2(EtOH)_2\}Cl_2$ (**6**) is formally constructed of an outer $[Li_4]$- and a central μ_4-oxocentered $[Cu_4O]$-tetrahedron, linked together by two $(L^2)^{4-}$ clamps. Contrary to classical coronates which scavenge cations, **6** encapsulates an O^{2-}-anion and thus may be considered as an example of an *inverse* crown ether sandwich complex (Fig. 3) [50–55].

3. Conclusion

The profound impact of different reaction conditions on the product formation in supramolecular chemistry was once again impressively confirmed. Contrary to green $[Cu_4(H_2L^2)_2(OAc)_4]$ (**5**), which was isolated when H_4L^2 (**4**) was reacted with copper(II) *acetate*, reaction of H_4L^2 (**4**) with copper(II) *chloride* yielded violet crystals of $\{[Li_4(Cu_4O)(L^2)_2](H_2O)_2(EtOH)_2\}Cl_2$ (**6**). More attention should therefore be paid to experimental details, since they strongly affect self-assembly processes.

4. Experimental

4.1. General techniques

Reagents and metal salts were used as obtained from Aldrich. Ligands **1** and **4** were prepared by standard procedures. IR spectra were recorded from KBr pellets on a Bruker IFS 25 spectrometer. FAB-MS spectra were recorded on a Micromass ZabSpec-E spectrometer. Elemental analyses were performed on a EA 1110 CHNS-Microautomat.

4.2. General method

To a suspension of 9 mmol (0.07 g) lithium hydride, in 60 mL anhydrous tetrahydrofuran 4 mmol (0.78 g) *N*-benzyliminodiethanol (**1**) (for **3**) or, respectively, 2 mmol (0.62 g) *m*-xylene-bis-(iminodiethanol) (**4**) (for **5** and **6**) were added. After stirring for 1 h at 20 °C, 4 mmol (0.54 g) copper(II) chloride (for **3** and **6**) or 4 mmol (0.80 g) $Cu(OAc)_2 \cdot H_2O$ (for **5**) were added. The reaction mixture was then stirred for one day at 20 °C. In the case of **3** a blue precipitate was subsequently collected on a glass frit. The solid was then extracted with 50 mL of hot nitromethane. After partial evaporation of the solvent diethyl ether was added and after standing for two days green crystals were obtained. In the case of **5** the green solution was filtrated and the solvent was then evaporated. The remaining green oil was extracted with 50 mL ethanol. The solvent was then evaporated and the remaining green solid was then re-crystallized from dichloromethane/diethyl ether. In the case of **6** a green precipitate was collected on a glass frit. The solid was then introduced into 50 mL methanol (H_2-generation!). After filtration of the deep violet solution the solvent was evaporated. The residue was then crystallized from $CHCl_3$/EtOH (95:5) by diffusion of diethyl ether. The microanalytical data for **3** and **6** deviate from theory due to crystal solvents and are not recorded here.

*4.2.1. $[Cu_2(HL^1)_2Cl_2]$ (**3**)*

3 $\cdot 1.5CH_3NO_2 \cdot Et_2O \cdot H_2O$: yield: 0.28 g (21%) green needles from nitromethane/diethyl ether (1:1) [56]; m.p. > 220 °C (decomp.), IR (KBr): $\tilde{\nu} = 3031, 2898$, and 2844 cm^{-1}, FAB-MS (3-nitrobenzyl alcohol-matrix): *m/z*: 1105 (100%) $[Cu_4Cl_2(HL^1)_4]^+$; 1070 $[Cu_4Cl(HL^1)_4]^+$.

*4.2.2. $[Cu_4(H_2L^2)_2(OAc)_4]$ (**5**)*

5 $\cdot 2CH_2Cl_2$: yield: 0.91 g (71%) green prismatic crystals from dichloromethane/diethyl ether (5:1). Correct microanalysis for $C_{42}H_{68}Cl_4Cu_4N_4O_{16}$: Calc.: C, 39.38; H, 5.35; N, 4.37. Found: C, 40.11; H, 5.20; N, 7.24%; m.p. > 220 °C (decomp.), IR (KBr): $\tilde{\nu} = 2922$, 2859, 1568, and 1419 cm^{-1}, FAB-MS (3-nitrobenzyl alcohol-matrix): *m/z* : 935 (85%) [**5**–3OAc]$^+$; 810 [**5**–4OAc–Cu]$^+$.

*4.2.3. $[Cu_4Li_4O(L^2)_2(H_2O)_2(EtOH)_2]Cl_2$ (**6**)*

6 $\cdot 5CHCl_3 \cdot 3.5H_2O$: yield: 0.80 g (45%) deep violet crystals from $CHCl_3$/EtOH and Et_2O (95:5) [56]; m.p. > 220 °C (decomp.), IR (KBr): $\tilde{\nu} = 2955$, 2852, and 1986 cm^{-1}, FAB-MS (3-nitrobenzyl alcohol-matrix): *m/z*: 949 (100%) $[Cu_4Li_4O(L^2)_2]Cl^+$; 914 $[Cu_4Li_4O(L^2)_2]^+$.

4.3. Single crystal X-ray structure analyses

Details for crystal data, data collection and refinement are given in Table 1. X-ray data for **3** were collected on a

Table 1
Details of X-ray structure determinations

	3	**5**	**6**
Formula	$2C_{22}H_{32}Cl_2Cu_2N_2O_4 \cdot Et_2O \cdot 1.5CH_3NO_2 \cdot H_2O$	$C_{40}H_{64}Cu_4N_4O_{16} \cdot 2CH_2Cl_2$	$C_{36}H_{64}Cl_2Cu_4Li_4N_4O_{13} \cdot 5CHCl_3 \cdot 3.5H_2O$
M_r	1356.71	1280.96	1773.63
Crystal size (mm)	0.30 × 0.30 × 0.20	0.64 × 0.50 × 0.30	0.40 × 0.16 × 0.16
Crystal system	triclinic	monoclinic	triclinic
Space group	$P\bar{1}$	$C2/c$	$P\bar{1}$
T (K)	173(2)	298(2)	183(2)
a (Å)	12.012(2)	17.299(6)	14.7576(6)
b (Å)	16.036(3)	14.156(5)	15.0453(6)
c (Å)	16.212(3)	23.022(7)	19.7266(8)
α (°)	81.30(3)	90	83.2120(10)
β (°)	89.51(3)	108.70(2)	73.1130(10)
γ (°)	82.92(3)	90	65.0230(10)
V (Å^3)	3063.3(11)	5340(3)	3799.1(3)
Z	2	4	2
$P_{calc.}$ (Mg m^{-3})	1.450	1.593	1.550
θ range (°)	4.08–27.45	1.87–27.00	1.08–27.03
Reflections collected	13 837	6781	36 364
Unique reflections	13 837	5835	16 168
[R_{int}]	0	0.0346	0.0347
Reflections observed [$I > 2\sigma(I)$]	9915	3117	10 718
Parameters	700	316	880
Final $R1[I > 2\sigma(I)]$	0.0978	0.0528	0.0609
$wR2$ (all data)	0.1754	0.1504	0.2040
Largest residuals (e Å^{-3})	1.172/−0.808	0.511/−0.932	1.631/−0.650

Nonius Kappa CCD area detector, for **5** on a Nicolet R3m/V, and for **6** on a Bruker AXS Smart using Mo Kα radiation ($\lambda = 0.71073$ Å). Semiempirical absorption corrections were employed. The structures were solved by direct methods with SHELXS-97 [57] and refined with full matrix least squares against F^2 with SHELXL-97 [58] (**3, 5**), respectively SHELXL-93 [58] (**6**).

5. Supplementary data

Crystallographic data (excluding structure factors) for the structures reported in this paper were deposited with the Cambridge Crystallographic Data Centre as supplementary publication Nos. CCDC-179212 (**3**), CCDC-179028 (**5**) and CCDC-181102 (**6**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44)1223-336-003; e-mail: deposit@ccdc.cam.ac.uk).

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