

Self-Assembly of Fourteen Components into a Soluble, Neutral, Metalloprismatic Cage

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A novel one-pot synthesis of rhenium-based triangular prismatic cages has been accomplished in excellent yields by a self-assembly strategy. Reaction of $\text{Re}_2(\text{CO})_{10}$ with 2,4,6-tri-4-pyridyl-1,3,5-triazine (tpt) in the presence of an aliphatic alcohol (1-butanol, 1-octanol or 1-dodecanol) afforded molecular prisms, $[\{(\text{CO})_3\text{Re}(\mu_2\text{-OR})_2\text{Re}(\text{CO})_3\}_3(\mu_3\text{-tpt})_2]$ (**1**, R = C_4H_9 ; **2**, R = C_8H_{17} ; **3**, R = $\text{C}_{12}\text{H}_{25}$) by solvothermal methods. Fine tuning of the solubility is achieved by tailoring the

length of the alkyl chain of the alcohol. Compounds **1–3** reported here are the first Re-containing molecular prisms of the $\text{M}_6\text{L}_2\text{L}'_6$ type. In addition, they are the first neutral, prismatic cages without counterions that are highly air and moisture stable. Compounds **2** and **3** are soluble in most common organic solvents and they exhibit a large solvatochromism: the value of $\lambda_{\text{max}}^{\text{ab}}$ could be tuned over 125 nm simply by changing the polarity of the solvent.

Introduction

The fabrication of nanometer-scale macrocycles by metal-directed, self-assembled cage compounds is an important component of supramolecular chemistry.^[1] There is an upsurge of interest in this area due to the potential applications of these molecules in host-guest chemistry and molecular recognition.^[2] Since the construction of larger cages by conventional covalent synthetic procedures is a tedious process, self-assembly techniques have been employed by several groups to obtain the metal-directed assembly of cage compounds.^[3–5] The nanosized, spherical-cage Pd^{II} complexes constructed by the metal-induced self-assembly strategy using an exotridentate ligand 2,4,6-tri-4-pyridyl-1,3,5-triazine (tpt) exhibit remarkable thermodynamic stability with a large spherical void.^[6] Recently, we have initiated a systematic approach for the design of rhenium-containing, neutral, cage-like supramolecules to explore their applications. In this communication we report the synthesis and characterization of the prismatic cages of the type $\text{M}_6\text{L}_2\text{L}'_6$, $[\{(\text{CO})_3\text{Re}(\mu_2\text{-OR})_2\text{Re}(\text{CO})_3\}_3(\mu_3\text{-tpt})_2]$ (**1**, R = C_4H_9 ; **2**, R = C_8H_{17} ; **3**, R = $\text{C}_{12}\text{H}_{25}$) made from fourteen components, including the octahedrally coordinated Re metal center, the tpt ligand and an aliphatic alcohol. The advantage here is that the product is obtained in excellent yield in a one-pot synthesis by a self-assembly strategy using solvothermal methods, where the alcohol that is used as

solvent also serves as one of the reagents. It is noteworthy that the currently known prismatic supramolecules are either highly charged or encapsulated with counterions.^[1b,1–i,4d,5d] On the other hand, compounds **1–3** are the first neutral, prismatic cages without counterions that are highly air and moisture stable. Compounds **2** and **3** are soluble in most common organic solvents and they show a large solvatochromism.

Results and Discussion

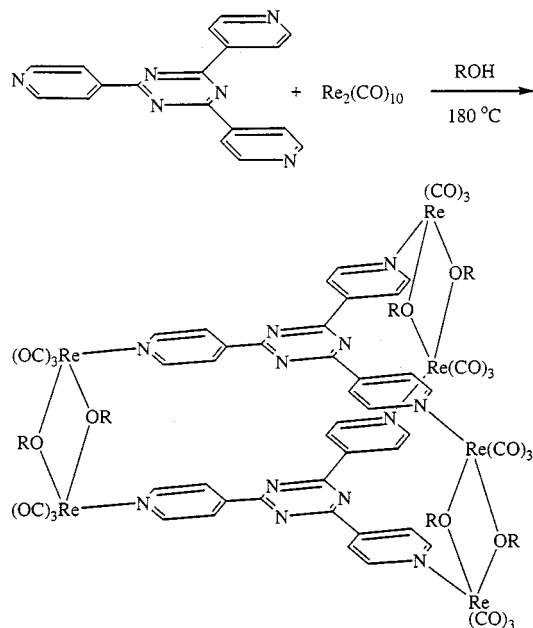
Treatment of $\text{Re}_2(\text{CO})_{10}$ with tpt in $\text{CH}_3(\text{CH}_2)_n\text{OH}$ ($n = 3, 7, 11$) using solvothermal methods at elevated temperatures leads to the formation of the thermodynamically stable cage structures **1**, **2**, and **3**, respectively, in excellent yields (Scheme 1). Compound **1** has been spectroscopically and crystallographically characterized. The ORTEP drawing of **1** (Figure 1) reveals a prismatic cage architecture, where two planar tpt ligands coordinate to six rhenium atoms, which are themselves connected by six butoxy bridges, thereby forming a triangular prismatic cage. Each rhenium atom occupies a corner of the triangular prism and is bonded to one nitrogen atom of the pyridyl group of the tpt ligand, two butoxy groups and three carbonyl groups, maintaining a distorted octahedral geometry. The maximum and minimum rhenium–rhenium distances in the molecule are 13.5 and 3.4 Å, respectively. The three pyridyl rings of the tpt moiety are canted slightly (ca. 15°) from the plane containing the triazine ring. Some groups of researchers have experienced difficulty in growing X-ray quality single crystals of metallomacromolecules containing two to twelve metal centers.^[7] In contrast, our synthetic strategy has led to the formation of single crystals of good quality. The electroneutrality of the cage compounds **1–3** mitigates

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interference of the counterions inside the cavity, which creates problem in most of the sensor studies. The extraordinary affinity of oxygen with rhenium is capitalized in this synthetic approach by utilizing oxo-bridges bearing long, hydrophobic tails. The ^1H and ^{13}C NMR spectral measurements and the FAB-MS data of the cage compounds are in agreement with the proposed structures.



Scheme 1

It has been a challenge to synthesize highly soluble multi-metal supramolecules and it is obvious that the solubility of such molecules is of paramount importance for their potential applications. Lees and co-workers have recently reported that complexes with more metal centers have lower solubility; a square complex with four metal components displayed lower solubility than a related triangular complex with three metal components.^[7b] Interestingly the solubility of compounds **1–3** has been improved enormously by changing the length of the alkyl moiety from butyl to octyl and then to dodecyl. An increase in the chain length of the alkoxy moiety increases the hydrophobicity of the cage, thereby increasing the solubility of the compound in organic solvents. Compounds **2** and **3** are readily soluble in CH_2Cl_2 , $(\text{CH}_3)_2\text{CO}$ and other common organic solvents, whereas **1** is much less soluble. Because of the greater solubility of compounds **2** and **3** they were used for further studies.

Compounds **2** and **3** display an identical CO stretching pattern in their infrared spectra which is similar to that observed for previously reported compounds.^[8] The UV/Vis spectra of **2** and **3** in CH_2Cl_2 show an intense, high-energy band at 243 nm and a broad low-energy band at 488 nm. In agreement with previous reports on related systems, the former absorption is assigned to the intraligand $\pi-\pi^*$ transition and the low energy absorption to a metal-to-ligand charge transfer (MLCT) transition.^[9] In addition, there is a shoulder at about 265 nm, which may be assigned

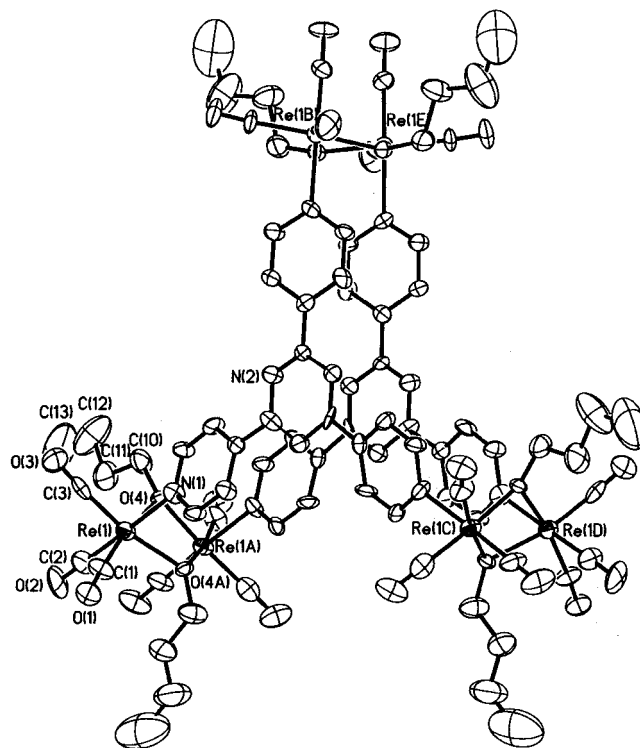


Figure 1. ORTEP diagram of $\{[(\text{CO})_3\text{Re}(\mu_2\text{-OC}_4\text{H}_9)_2\text{Re}(\text{CO})_3]_2(\mu_3\text{-tpt})_2\}$ (**1**); selected bond lengths (Å) and angles ($^\circ$): $\text{Re}(1)-\text{N}(1)$ 2.216(12), $\text{Re}(1)-\text{O}(4)$ 2.122(10), $\text{Re}(1)-\text{O}(4A)$ 2.144(9), $\text{Re}(1)-\text{C}(1)$ 1.98(2), $\text{Re}(1)-\text{C}(2)$ 1.91(2), $\text{Re}(1)-\text{C}(3)$ 1.89(2), $\text{O}(4)-\text{C}(10)$ 1.44(2); $\text{O}(4)-\text{Re}(1)-\text{N}(1)$ 83.1(4), $\text{O}(4A)-\text{Re}(1)-\text{N}(1)$ 82.0(4), $\text{Re}(1)-\text{O}(4)-\text{Re}(1A)$ 105.5(4), $\text{O}(4)-\text{Re}(1)-\text{O}(4A)$ 71.7(4), $\text{C}(10)-\text{O}(4)-\text{Re}(1)$ 124.8(10), $\text{C}(10)-\text{O}(4)-\text{Re}(1A)$ 122.6(10)

to another $\pi-\pi^*$ transition as suggested by Tapolsky et al.^[10] The interesting feature that deserves further attention is the observation of the MLCT at a comparatively long wavelength (488 nm), indicating the readily reducible nature of the ligand.^[11] We have recorded the absorption spectra of **2** and **3** in a large number of solvents and it is interesting to point out that they show substantial solvatochromism: the value of $\lambda_{\text{max}}^{\text{ab}}$ could be tuned over 125 nm, with a corresponding change in the color of the solution from yellow to red, simply by changing the polarity of the solvent ($\lambda_{\text{max}}^{\text{ab}}$ values for **2** in DMSO and CCl_4 are 424 and 549 nm, respectively).^[12]

In summary we have succeeded in the one-pot, high yield synthesis and characterization of neutral prismatic cages containing hexanuclear rhenium centers linked by six alkoxy bridges bearing lengthy alkyl chains. The presence of the lengthy alkyl chain dramatically increases the hydrophobic nature of the cage-like compounds and hence the solubility, which is a major problem in many of the supramolecular motifs. The interesting photophysical properties and solvatochromism of these supramolecular cage-like structures promise many applications. We are currently extending the metal-induced, self-assembly by solvothermal methods to the synthesis of molecular cages of new architecture from different sets of molecular components, and these results will be reported in a future publication.

Experimental Section

A suspension consisting of a mixture of $\text{Re}_2(\text{CO})_{10}$ (98 mg, 0.15 mmol) and tpt (32 mg, 0.10 mmol) in 1-butanol (10 mL) in a 30 mL Teflon flask was placed in a steel bomb. The bomb was placed in an oven maintained at 180 °C for 48 h and then cooled to 25 °C. Good quality, maroon-colored, single crystals of **1** were obtained. The crystals were separated by filtration and washed with 1-butanol. Compounds **2** and **3** were also synthesized by following a similar procedure. The solvent from the reaction mixture was removed by vacuum distillation and the residue was redissolved in CH_2Cl_2 and passed through a short silica gel column to get pure **2** and **3**. Yield: **1**, 109 mg, 81%; **2**, 124 mg, 82%; **3**, 142 mg, 85%.

[(CO)₃Re(μ₂-OC₄H₉)₂Re(CO)₃]₃(μ₃-tpt)₂] (1): $\text{C}_{78}\text{H}_{78}\text{N}_{12}\text{O}_{24}\text{Re}_6$ (2684.79): calcd. C 34.89, H 2.93, N 6.26; found C 35.06, H 2.94, N 6.40. – Crystal data ($\text{C}_{78}\text{H}_{78}\text{N}_{12}\text{O}_{24}\text{Re}_6$): Crystal dimensions 0.20 × 0.10 × 0.03 mm, rhombohedral, space group $R\bar{3}(\text{bar})c$, $a = b = 23.4486(9)$, $c = 27.6604(11)$ Å, $\alpha = \beta = 90^\circ$, $\gamma = 120^\circ$, $V = 13171.1(9)$ Å³, $Z = 6$, $T = 150(1)$ K, $\rho_{\text{calcd.}} = 2.031$ Mg/m³, $\mu = 8.316$ mm⁻¹, 9628 total reflections, 2539 independent reflections which were used in refinement. The structure was solved to $R1 = 0.0865$ and $wR2 = 0.1166$.

[(CO)₃Re(μ₂-OC₆H₁₇)₂Re(CO)₃]₃(μ₃-tpt)₂] (2): IR (CH_2Cl_2): $\nu_{\text{CO}} = 2022$ (s), 2009 (m), 1913 (m), 1890 (vs) cm⁻¹. – ¹H NMR (300 MHz, CDCl_3): $\delta = 8.72$ (d, $^3J = 6.5$ Hz, 12 H, H³), 8.24 (d, $^3J = 6.5$ Hz, 12 H, H²), 4.43 (t, $^3J = 8.0$ Hz, 12 H), 2.15 (m, 12 H), 1.49 (m, 36 H), 1.25 (m, 24 H), 0.94 (t, $^3J = 6.7$ Hz, 18 H). – ¹³C NMR (75 MHz, CDCl_3): $\delta = 197.2$ (s, CO), 170.1 (C, triazine), 153.5 (C³), 142.2 (C¹), 123.2 (C²), 82.9 (CH₂), 34.2 (CH₂), 31.9 (CH₂), 29.7 (CH₂), 29.4 (CH₂), 25.3 (CH₂), 22.7 (CH₂), 14.2 (CH₃). – UV/Vis (CH_2Cl_2): λ_{max} (nm) = 488 (MLCT), 243 (LIG). – $\text{C}_{108}\text{H}_{126}\text{N}_{12}\text{O}_{24}\text{Re}_6$ (3021.43): calcd. C 40.55, H 4.20, N 5.56; found C 40.56, H 4.23, N 5.53. – MS (FAB, ¹⁸⁷Re): $m/z = 3024$ [M⁺].

[(CO)₃Re(μ₂-OC₁₂H₂₅)₂Re(CO)₃]₃(μ₃-tpt)₂] (3): IR (CH_2Cl_2): $\nu_{\text{CO}} = 2022$ (s), 2009 (m), 1913 (m), 1890 (vs) cm⁻¹. – ¹H NMR (300 MHz, CDCl_3): $\delta = 8.72$ (d, $^3J = 6.5$ Hz, 12 H, H³), 8.24 (d, 12 H, H²), 4.42 (t, $^3J = 8.0$ Hz, 12 H), 2.15 (m, 12 H), 1.48 (m, 24 H), 1.29 (m, 84 H), 0.88 (t, $^3J = 6.2$ Hz, 18 H). – ¹³C NMR (75 MHz, CDCl_3): $\delta = 197.2$, 197.1 (s, 1:2 CO), 170.1 (C, triazine), 153.5 (C³), 142.1 (C¹), 123.2 (C²), 82.8 (CH₂), 34.1 (CH₂), 31.9 (CH₂), 31.5 (CH₂), 29.7 (2 CH₂), 29.6 (2 CH₂), 29.3 (CH₂), 25.2 (CH₂), 22.7 (CH₂), 14.1 (CH₃). – UV/Vis (CH_2Cl_2): λ_{max} (nm) = 488 (MLCT), 242 (LIG). – $\text{C}_{126}\text{H}_{174}\text{N}_{12}\text{O}_{24}\text{Re}_6$ (3358.08): calcd. C 44.99, H 5.22, N 5.00; found C 45.52, H 5.30, N 4.92. – MS (FAB, ¹⁸⁷Re): $m/z = 3360$ [M⁺].

Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-149282 and -149283 (1). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: (internat.) +44-1223/336-033; E-mail: deposit@ccdc.cam.ac.uk].

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