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X-ray Evidence of CN Bridging in Bimetallic Complexes Based on $[M(CN)_8]^{4-}$ (M = Mo, W). The Crystal Structure of $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$

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Introduction

Cyano-bridged systems based on hexacyanometalates[1] and heptacyanomolybdate(III)[2] have been extensively employed as building blocks for constructing polynuclear assemblies because of their applications in electron-transfer processes, in molecular electronics, and as molecular magnets. The alternative octacyanometalate redox couples, offering eight cyano ligands potentially able to form highly structured networks of desired properties, have attracted some attention in recent years.[3] The three-dimensional coordination polymers $[(Me_3Sn)_4M(CN)_8]_n$, based on octacyanomolybdate(IV) and -tungstate(IV) building units with organotin cations $(R_3Sn)^{3+}$, have provided the first structural proof of cyano-bridge formation by octacyanides.[4]

The cyano bridge provides moderate coupling of d-metal centers via σ-donor and π^*-acceptor bonding. The square antiprismatic or dodecahedral limiting geometries[5] and 4d or 5d valence orbitals of octacyanides of Mo(IV) or W(IV) coupled with 3d orbitals of other metal centers represent novel features in the field of supramolecular chemistry. To achieve the construction of d−d clusters of limited nuclearity based on $[M(CN)_8]^{4-}$ precursors, we have employed *cis*-$[Mn(bpy)_2Cl_2]$ by attaching the cyanometalate moieties as ligands at labile Mn(II) coordination sites. The labile Cl^- ligands in the hexacoordinate manganese(II) center leave two cis-vacant coordination sites, which implies that the cluster will probably grow by linking metal centers thorough double cyano bridges.

We report here the synthesis and vibrational characteristics of bimetalic hexanuclear $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[M(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 14H_2O$ (M = Mo or W) and the single-crystal X-ray structure of the Mn_4Mo_2 cluster $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[M(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$, representing a variety of accessible oxidation states due to the redox character of the centers involved.

Experimental Section

Materials. $K_4[Mo(CN)_8]\cdot 2H_2O$, $K_4[W(CN)_8]\cdot 2H_2O$, and $[Mn(bpy)_2Cl_2]\cdot 2H_2O\cdot EtOH$ were made according to literature procedures.[6−8] All other chemicals were purchased from Aldrich and used as received.

Physical Measurements. UV−vis reflectance spectra were measured as pellets in $BaSO_4$ vs $BaSO_4$ as reference (Shimadzu UV 2101PC equipped with ISR-260 attachment). IR spectra were measured as KBr pellets on a Bruker IFS 48 spectrometer. Thermogravimetric (TG) and differential thermal analysis (DTA) were performed on a Mettler thermoanalyzer TGA/SDTA 851, under argon or in air with a heating rate of 5 °C/min. The H_2O contents were obtained by vacuum dehydration over P_4O_{10} at room temperature.

Synthesis of $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 14H_2O$ (1). To a solution of $K_4[Mo(CN)_8]\cdot 2H_2O$ (0.50 g, 1.0 mmol) in water (30 mL, natural pH) was added $[Mn(bpy)_2Cl_2]\cdot 2H_2O\cdot EtOH$ (2.0 g, 2.0 mmol) in water (30 mL), which gave immediately the precipitation of a light-yellow product (**1**). The salt was washed three times with water and then with ethanol and dried in air (1.95 g, 84%).

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Table 1. Crystallographic Data and Structure Refinement for $\{[Mn(bpy)_2]_2(\mu\text{-}CN)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$

empirical formula	$C_{96}H_{80}N_{32}O_8Mn_4Mo_2$
fw	2221.56
temp, K	180(2)
λ, Å	0.710 73
cryst syst	monoclinic
space group	$P2_1/n$
a, Å	13.9804(10)
b, Å	22.8924(12)
c, Å	15.5757(12)
β, deg	91.710(3)
V, Å^3	4982.7(6)
ρ, g cm^{-3}	1.481
Z	4
μ, mm^{-1}	0.808
final R indices [$I > 2\sigma(I)$]^a	R1 = 0.0644 wR2 = 0.1635
R indices (for all data)^a	R1 = 0.1257 wR2 = 0.1333

^{a} $R = \sum||F_o| - |F_c||/\sum|F_o|$. $R_w = [(\sum w(|F_o| - |F_c|)^2/\sum wF_o^2)]^{1/2}$.

Anal. Calcd for $C_{96}H_{92}N_{32}Mn_4Mo_2O_{14}$: C, 49.49; H, 3.95; N, 19.25; H_2O, 10.83. Found: C, 49.80; H, 3.65; N, 19.28; H_2O, 9.44. The anhydrous complex and H_2O contents were obtained by vacuum dehydration over P_4O_{10} at room temperature.

Synthesis of $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$ (1a). The procedure was analogous to that for **1** except that the crystals were grown in an H-tube from solutions of reagents of the same concentration as in the synthesis of **1**.

Synthesis of $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[W(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 14H_2O$ (2). The procedure was analogous to that of **1**; $K_4[W(CN)_8]\cdot 2H_2O$ (0.58 g, 1.0 mmol) was used (2.20 g, 91%). Anal. Calcd for $C_{96}H_{92}N_{32}Mn_4W_2O_{14}$: C, 45.98; H, 3.67; N, 17.88; H_2O, 10.43. Found: C, 46.04; H, 3.14; N, 17.69; H_2O, 10.03.

X-ray Crystallographic Studies. The crystallographic data of **1a** are summarized in Table 1. Data were collected using a Siemens SMART CCD area detector diffractometer. Absorption corrections were applied[9] using SADABS. The structure was solved by direct methods and refined[10] for all data using SHELXL 97.

Results and Discussion

Synthesis and Chemical Properties. While studying systematically the possibility of binding $[M(CN)_8]^{n-}$ ions (M = Mo or W) to other metal centers, we prepared and structurally characterized the hexanuclear μ-cyano complexes on the basis of octacyanometalates(IV) and $[Mn(bpy)_2]^{2+}$ moieties. The reaction of $[M(CN)_8]^{4-}$ complex with labile *cis*-$[Mn(bpy)_2Cl_2]$[7] in aqueous solution affords a yellow polycrystalline species of the general formula $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[M(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 14H_2O$, where M = Mo (**1**) or W (**2**), bpy = 2,2′-bipyridine. Thermogravimetric measurements of $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[M(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 14H_2O$ show that the dehydration process starts at room temperature without any DTA or TG maximum and ends at ca. 100 °C. The analogous synthesis of **1** in an H-tube, affording single crystals suitable for X-ray structure determination, yields $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$ (**1a**). All compounds are insoluble in water and organic solvents.

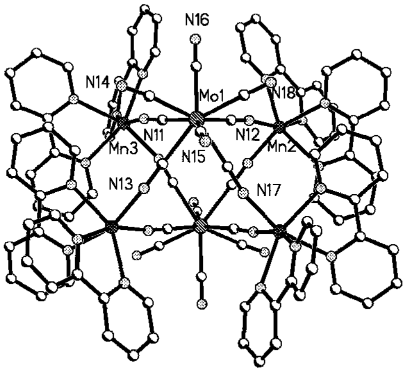

Figure 1. Molecular structure of **1a**. Hydrogen atoms have been omitted for clarity.

Table 2. Selected Bond Lengths (Å) and Angles (deg) for $\{[Mn(bpy)_2]_2(\mu\text{-}CN)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$

Mo(1)−C(11)	2.163(7)	C(11)−N(11)	1.151(8)
Mo(1)−C(12)	2.176(7)	C(12)−N(12)	1.161(8)
Mo(1)−C(13)	2.149(7)	C(13)−N(13)	1.144(8)
Mo(1)−C(14)	2.155(7)	C(14)−N(14)	1.153(8)
Mo(1)−C(15)	2.172(8)	C(15)−N(15)	1.155(9)
Mo(1)−C(16)	2.151(8)	C(16)−N(16)	1.163(9)
Mo(1)−C(17)	2.149(7)	C(17)−N(17)	1.151(8)
Mo(1)−C(18)	2.153(7)	C(18)−N(18)	1.164(8)
Mn(2)−N(12)	2.179(5)	Mn(3)−N(11)	2.156(6)
Mn(2)−N(13)	2.142(6)	Mn(3)−N(17)	2.130(6)
C(13)−Mo(1)−C(17)	70.3(2)	C(16)−Mo(1)−C(14)	75.3(2)
C(16)−Mo(1)−C(18)	74.9(2)	C(13)−Mo(1)−C(11)	71.9(2)
C(18)−Mo(1)−C(15)	69.9(3)	C(14)−Mo(1)−C(15)	74.7(3)
C(17)−Mo(1)−C(12)	72.3(2)	C(11)−Mo(1)−C(12)	76.7(2)
N(12)−Mn(2)−N(13)	91.2(2)	C(12)−N(12)−Mn(2)	172.8(6)
C(13)−N(13)−Mn(2)	153.3(6)	N(11)−Mn(3)−N(17)	90.4(2)
C(11)−N(11)−Mn(3)	162.5(6)	C(17)−N(17)−Mn(3)	155.7(6)

The electronic solid-state reflectance spectra of **1**, **1a**, and **2** show the superposition of characteristic bands of the reagents at 249, 300, and 410 (broad) nm for the Mo analogue (the spectra of **1** and **1a** are identical) and at 249, 300, and 415 (broad) nm for the W analogue. No additional bands that could be attributed to metal-to-metal charge-transfer transitions were observed.

Structure of 1a. The molecular structure of $\{[Mn(bpy)_2]_2(\mu\text{-}CN)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$ is shown in Figure 1, and selected bond lengths and angles are collected in Table 2. The molecule is centrosymmetric and contains four Mn centers connected by two octacyanomolybdate(IV) units via eight cyano bridges. The Mn atoms are in a rectangular array (Mn−Mn distances of 5.611(2) and 6.455(1) Å), and the Mo atoms are located at ±3.273(1) Å from the Mn plane; the vector joining Mo1 to its symmetry-equivalent atom is almost orthogonal to the Mn plane (e.g., Mo1−centroid−Mn2 = 92.44(2)°). The Mn atoms are hexacoordinate, having a distorted octahedral geometry with two bpy and two *cis* cyano bridges. The bond lengths and angles in the $[Mn(bpy)_2]^{2+}$ moiety are practically identical to those found in $[Mn(bpy)_2Cl_2]\cdot 2H_2O\cdot EtOH$.[8] Since the $[Mo(CN)_8]^{4-}$ ion is thermally stable and kinetically inert, we postulate the Mn−NC−Mo assembly in the structure. Each Mo atom has four bridging and four terminal cyano ligands arranged in a square antiprismatic arrangement. The mean Mo−C and C−N distances are 2.159 and 1.155 Å, respectively, and the Mo−C−N units are almost linear (maximun deviation of 6°); within experimental error there are no dimensional differences between the mean values for the bridging and terminal cyano ligands. The Mo−C and Mo−N bond distances are in the range typical for other octacyanomolybdates(IV) with different cations.[11] The asymmetric unit contains four oxygen atoms (i.e., eight per formula unit) that are clearly associated with water molecules, but unfortunately, the hydrogen atoms could not be located. Short O···O and O···N separations (about

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(9) Sheldrick, G. M. *SADABS, Program for Absorption Corrections*; University of Göttingen: Göttingen, Germany, 1996.

(10) Sheldrick, G. M. *SHELXL 97, Program for Crystal Structure Refinement*; University of Göttingen: Göttingen, Germany, 1997.

2.8 Å), however, are indicative of hydrogen bonding; specifically the nonbridging N14 and N18 atoms of cyano ligands are involved in these interactions. The structure of the Mn_4Mo_2 cluster can be compared with the three-dimensional coordination polymers $[(Me_3Sn)_4M(CN)_8]$.[4] The structure of these contain square antiprismatic $[Mo(CN)_8]^{4-}$ units with Mo−C and C−N bond lengths practically the same within experimental error. However, all eight cyano ligands in $[(Me_3Sn)_4M(CN)_8]$ are involved in the formation of bent cyano bridges (which seems to be a characteristic feature of octacyanides).

The solid-state vibrational spectrum of the $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[M(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}$ unit is found to be essentially independent of the content of water molecules in the ν(CN) stretching region. Complex **1**, as well as its anhydrous form, exhibits bands at 2069w, 2110vs, 2119vs 2138vs, and 2150m (IR) and at 2077w, 2084sh, 2124vs and 2147vs (R), which are attributed to these vibrations, whereas ν(CN) stretching modes for **2** are observed at 2108vs, 2124sh, and 2147m (IR) and at 2062vw, 2077vw, 2118vs, and 2145vs (R). Generally the region and pattern of the ν(CN) bands are similar to those observed for octacyanometalates(IV).[3b,11b,12,13] For simple ionic compounds of octacyanomolybdate(IV), the ν(CN) bands (IR) are in the range 2060−2160 cm^{-1},[11b,13,14] whereas in cyano-bridged systems the range 2085−2141 cm^{-1} has been found.[3b] A relative insensitivity of ν(CN) stretching bands to the type of bonding, bridging or terminal, is thus observed. This is in agreement with the IR spectra of $[(Me_3Sn)_4M(CN)_8]$, where ν(CN) bands at 2143 and 2105 cm^{-1} (Mo) and at 2141 and 2105 cm^{-1} (W) were observed.[4] The very small shift of ν (CN) in cyano-bridged complexes is, however, to be expected, considering the opposing effects of $d\pi-\pi^*$(CN) back-bonding from the C-bonded Mo and N-bonded Mn coupled with the kinematic effect, when the parent frequency is relatively high and the metal bridged does not form a donor−acceptor system.[15]

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Supporting Information Available: X-ray crystallographic files in CIF format for the structure determination of $\{[Mn(bpy)_2]_2(\mu\text{-}NC)_2[Mo(CN)_6]_2(\mu\text{-}CN)_2[Mn(bpy)_2]_2\}\cdot 8H_2O$. This material is available free of charge via the Internet at http://pubs.acs.org.

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