Excision of the $\{Mo_6Se_8\}$ Cluster Core from a Chevrel Phase: Synthesis and Properties of the First Molybdenum Octahedral Cluster Selenocyanide Anions $[Mo_6Se_8(CN)_6]^{7-}$ and $[Mo_6Se_8(CN)_6]^{6-}$

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Abstract: The synthesis of new molybdenum cluster selenocyanide anionic complexes $[Mo_6Se_8(CN)_6]^{7-}$ and $[Mo_6Se_8(CN)_6]^{6-}$ is reported. The $[Mo_6Se_8(CN)_6]^{7-}$ ion was obtained by excision of the cluster core $\{Mo_6Se_8\}$ from a Chevrel phase in the reaction of Mo_6Se_8 with KCN at 650 °C; the $[Mo_6Se_8(CN)_6]^{6-}$ ion is formed by oxidation of $[Mo_6Se_8(CN)_6]^{7-}$. New cluster salts $K_7[Mo_6Se_8(CN)_6] \cdot 8H_2O$ (**1**) and $(Me_4N)_4K_2[Mo_6Se_8(CN)_6] \cdot 10H_2O$ (**2**) were isolated and their crystal structures were solved. Compound **1** crystallizes in the cubic space group *Fm*$\bar{3}$*m* ($a = 15.552(2)$ Å, $Z = 4$, $V = 3761.5(8)$ Å^3), compound **2** crystallizes in the triclinic space group $P\bar{1}$ ($a = 11.706(2)$, $b = 11.749(2)$, $c = 12.459(2)$ Å, $\alpha = 72.25(1)$, $\beta = 77.51(1)$, $\gamma = 63.04(1)°$, $Z = 1$, $V = 1448.5(4)$ Å^3). Compound **1** is paramagnetic due to an availability of 21 electrons per Mo_6 cluster; cyclic voltammetry reveals a quasi-reversible transition $[Mo_6Se_8(CN)_6]^{7-} \rightleftharpoons [Mo_6Se_8(CN)_6]^{6-}$, $E_{1/2} = 0.63$ V.

Keywords: cluster compounds · chalcogens · Chevrel phase · cyanides · molybdenum

Introduction

Hexanuclear molybdenum cluster complexes have attracted attention because of their relationships to higher dimensional systems such as superconducting Chevrel phases. In most cases the complexes with $\{Mo_6X_8\}$ (X = S, Se) cluster cores were prepared from molecular precursors by using the reductive dimerization of trinuclear fragments Mo_3X_4[1, 2] or ligand exchange in the cluster core of octahedral cluster complexes such as Mo_6Cl_{12}.[3] Excision of cluster fragments from preformed solid-state cluster compounds is one of the major synthetic methods for the preparation of molecular cluster compounds.[4, 5] Recently we discovered that the molten cyanides (NaCN or KCN) are the excellent reagents for excision of cluster cores from solids with polymeric framework structures at moderate (≈600 °C) temperatures.[6–9] Using this method a series of new chalcocyanide complexes containing $[Re_6X_8(CN)_6]^{4-}$ (X = S, Se, Te) cluster anions has been synthesized and characterized structurally.[10–17] Here we present the first example of excision of the $\{Mo_6Se_8\}$ cluster core from the parent Mo_6Se_8 solid (Chevrel-type phase) by reaction of Mo_6Se_8 with molten KCN. This results in the formation of soluble $[Mo_6Se_8(CN)_6]^{7-}$/$[Mo_6Se_8(CN)_6]^{6-}$ complexes. Structures and some properties of the first molybdenum octahedral selenocyanide cluster compounds $K_7[Mo_6Se_8(CN)_6] \cdot 8H_2O$ (**1**) and $(Me_4N)_4K_2[Mo_6Se_8(CN)_6] \cdot 10H_2O$ (**2**) are also reported.

Results and Discussion

Interaction of the Mo_6Se_8-containing neutral $\{Mo_6Se_8\}^0$ cluster core with molten potassium cyanide results in the formation of the deep blue anion $[Mo_6Se_8(CN)_6]^{7-}$ with a negatively charged $\{Mo_6Se_8\}^-$ cluster core. The presence of a negatively charged $\{Mo_6Se_8\}^-$ cluster core in $K_7[Mo_6Se_8(CN)_6] \cdot 8H_2O$ was in agreement with chemical analysis, structural,[18] and magnetic data. In aqueous solution the anion $[Mo_6Se_8(CN)_6]^{7-}$ is not stable for long periods and transforms readily to give the brown-colored oxidized form $[Mo_6Se_8(CN)_6]^{6-}$, which was isolated as salt **2**. Facile oxidation of the deep blue solution of $[Mo_6Se_8(CN)_6]^{7-}$ in air was confirmed by electrochemical measurements which indicated a one-electron quasi-reversible oxidation wave at 0.63 V.

The cluster anions of both compounds are shown in Figure 1. In compound **1** the $[Mo_6Se_8(CN)_6]^{7-}$ ion has the ideal O_h point symmetry. In the case of **2** the $[Mo_6Se_8(CN)_6]^{6-}$ ion has the site symmetry C_s and, therefore, the cluster is

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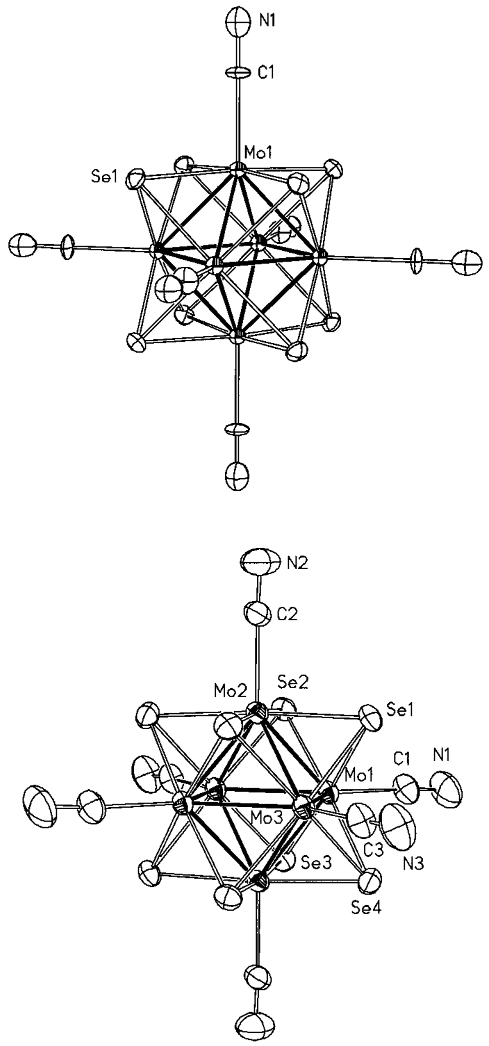

Figure 1. Cluster anions in the compounds **1** (top) and **2** (bottom); 50% probability ellipsoids.

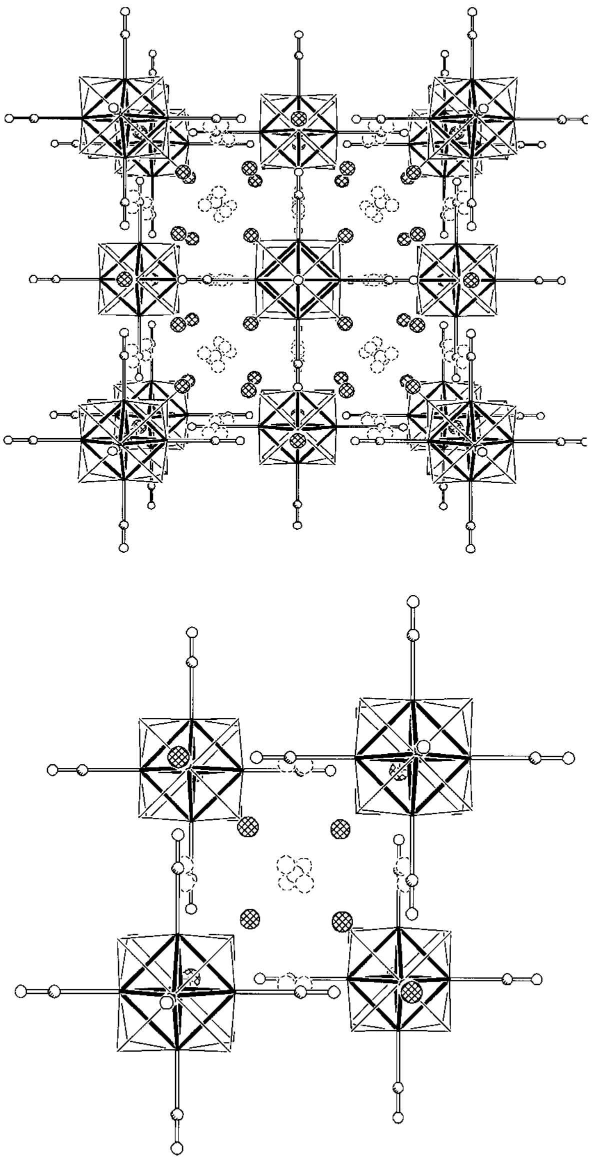

Figure 2. Unit cell (top) and one octant (bottom) in the crystal structure of **1** (*yz* projection). Circles of Mo and Se atoms are omitted for clarity. K^+ ions are shown as cross-shaded circles. The oxygen atoms O1 and O2 of water molecules are shown as empty and dashed circles, respectively.

slightly distorted. In **1** the cluster anions adopt cubic close packing (Figure 2 top). The distance between the centers of the cluster anions is 10.997 Å. There are two different cation positions denoted K1 and K2. K1 lies in the "octahedral interstitial" site of close packing, and K2 forms the tetrahedron within the "tetrahedral interstitial" site (Figure 2 bottom). Both cation positions are partially occupied. There are two different oxygen atom positions denoted O1 and O2 for the water molecules; O1 lies in the center of K_4 tetrahedron, and O2 is disordered over two positions around (1/2 3/4 3/4).

In the case of **2**, the centers of cluster anions form one layered hexagonal packing motif (Figure 3); the distances between the centers of these anions within the layer ranges between 11.706 and 12.262 Å and that between the layers is 12.459 Å. Potassium cations are coordinated by water molecules and are located between layers. The Me_4N^+ ions are located both within as well as between layers.

It is interesting to compare the geometry of cluster cores in **1** and **2** with other compounds based on the $\{Mo_6Se_8\}$ unit.

Abstract in Russian:

В работе сообщается о синтезе новых халькоцианидных кластерных анионных комплексах молибдена $[Mo_6Se_8(CN)_6]^{7-}$и $[Mo_6Se_8(CN)_6]^{6-}$. Анион $[Mo_6Se_8(CN)_6]^{7-}$ образуется в результате реакции вырезания при взаимодействии фазы Шевреля Mo_6Se_8 с KCN при 650°C, при окислении он превращается в $[Mo_6Se_8(CN)_6]^{6-}$. Получены соли состава $K_7[Mo_6Se_8(CN)_6]\cdot 8H_2O$ (**1**) и $(Me_4N)_4K_2[Mo_6Se_8(CN)_6]\cdot 10H_2O$ (**2**), методом рентгеноструктурного анализа определена их кристаллическая структура. Соединение **1** кристаллизуется в кубической сингонии ($Fm\bar{3}m$), a=15.552(2)Å, Z=4, V=3761.5(8)Å^3, соединение **2** кристаллизуется в триклинной сингонии ($P\bar{1}$), a=11.706(2) Å, b=11.749(2)Å, c= 12.459(2)Å, α=72.25(1)°, β=77.51(1)°, γ=63.04(1)°, Z=1, V=1448.5(4)Å^3. Соединение **1** с 21 валентным электроном на Mo_6 кластер имеет парамагнитный характер, циклическая вольтамперометрия показывает квазиобратимый переход $[Mo_6Se_8(CN)_6]^{7-} \rightleftarrows [Mo_6Se_8(CN)_6]^{6-}$, $E_{1/2}$= 0.63V.

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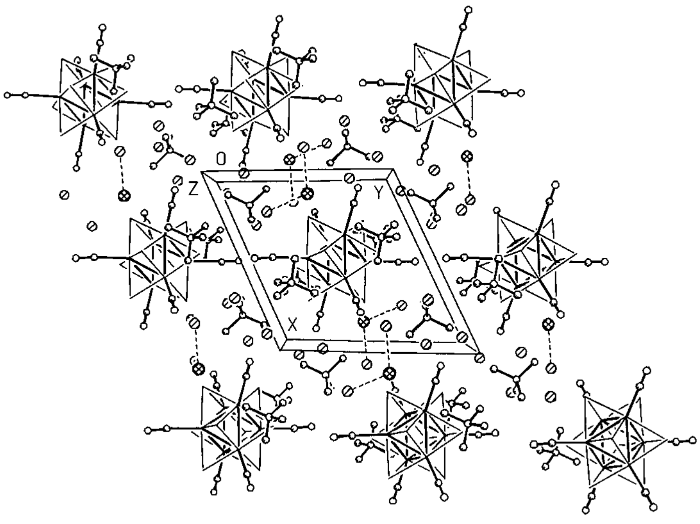

Figure 3. Crystal packing in the structure of **2** (projection along *c* axis). Circles of Mo and Se atoms are omitted for clarity. K^+ cations are shown as cross-shaded circles. Water molecules are shown as crossed out circles.

Details of some of the bond lengths in structurally characterized Chevrel-type compounds $M_xMo_6Se_8$ are given in Table 1.

It is well known that the Mo_6 octahedron in the polymeric Mo_6Se_8 phase with a $\{Mo_6Se_8\}^0$ cluster core is distorted in such a manner that there are six short and six long Mo–Mo distances. Compound **2**, which is built up of isolated $[Mo_6Se_8(CN)_6]^{6-}$ molecular clusters, formally contains the same zero-charged core $\{Mo_6Se_8\}^0$. The molecular character of the structure of **2**, however, dramatically changes the geometry of the cluster core: the Mo_6 octahedron in **2** becomes much more symmetrical (Δ(Mo–Mo) = 0.021 instead of 0.151 Å as in the Mo_6Se_8 phase) and the average intracluster Mo–Mo distance becomes shorter.

The same situation occurs between derivative Chevrel phases $M_xMo_6Se_8$ and compound **1**. They all contain the formally negatively charged $\{Mo_6Se_8\}^{n-}$ core. The $[Mo_6Se_8(CN)_6]^{7-}$ ion in **1** has an ideal octahedral core, whereas in the Chevrel phases $M_xMo_6Se_8$ the cluster cores are more or less distorted. The distinction between **1** and **2**, however, is much less; the difference between average Mo–Mo distances is only 0.011 Å. At this point it should be mentioned that it is known that the removal of bonding electrons from clusters in these compounds is expected to weaken the Mo–Mo bonds. This rule is also applicable here: both in molecular complexes and in a series of polymeric compounds, the Mo–Mo bonds in Mo_6 clusters increase in length with increasing valence electron concentration. However, the situation is exactly the opposite of that in $[Mo_6Se_8(PEt_3)_6]^0/[Mo_6Se_8(PEt_3)_6]^-$ complexes, in which the Mo–Mo distances in the negatively charged cluster core become longer:[2] average Mo–Mo = 2.703 Å in $[Mo_6Se_8(PEt_3)_6]$, and average Mo–Mo = 2.714 Å in $[PPN][Mo_6Se_8(PEt_3)_6]$.

In the structure of **1** one of the oxygen atoms (O2) appeared to be disordered between two symmetrically equivalent positions at a distance of 0.88. Thus, the occupancy of O2 was set to 50%.

As is clear from the above-mentioned data, the high-temperature reaction of polymeric hexanuclear molybdenum selenide Mo_6Se_8 with molten KCN leads to the formation of the octahedral cluster selenocyanide anion $[Mo_6Se_8(CN)_6]^{7-}$. This result regarding the excision of the cluster core $\{Mo_6Se_8\}$ from the structure of the polymeric precursor is similar to that obtained by reacting KCN with polymeric $[Re_6Te_{15}]$[10, 11] and $[Re_6X_8Br_2]$[9, 17] (X = S, Se); however, whereas rhenium cluster cores $\{Re_6X_8\}$ containing 24 electrons per Re_6 cluster do not change their electronic states on going from polymers to molecular complexes, the reaction of $[Mo_6Se_8]$ (20 electrons per Mo_6 cluster) with KCN generates the negatively charged cluster core $\{Mo_6Se_8\}^-$ with 21 electrons per Mo_6 cluster. This is particularly interesting because the resulting cluster selenocyanide anion $[Mo_6Se_8(CN)_6]^{7-}$ has paramagnetic properties.

In aqueous solution the anion $[Mo_6Se_8(CN)_6]^{7-}$ is not stable for long periods and transforms readily into the oxidized form $[Mo_6Se_8(CN)_6]^{6-}$. The electrochemical behavior of **1** has been studied in 0.1 M Na_2SO_4 aqueous solution. The cyclic voltammogram indicated a one-electron quasi-reversible oxidation wave at 0.63 V (Figure 4) that is attributed to the $[Mo_6Se_8(CN)_6]^{7-} \rightleftharpoons [Mo_6Se_8(CN)_6]^{6-}$ equilibrium. Further oxidation at 1.4 V is irreversible.

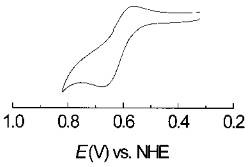

Figure 4. Cyclic voltammogram of **1**.

The measurement of the temperature dependence of the magnetic susceptibility showed that the compound **1** is paramagnetic over the temperature range (2–300 K) because the $[Mo_6Se_8(CN)_6]^{7-}$ ion has one unpaired spin due to an odd number of cluster valence electrons (21 e/Mo_6). The magnetic properties observed could not be explained by using the Curie–Weiss model and it is necessary to include a Van Vleck

Table 1. Bond lengths in related structurally characterized Chevrel-type compounds $M_xMo_6Se_8$.

Compound	Core charge	Mo–Mo [Å]	Ref.
Mo_6Se_8	0	2.685(2)–2.836(2) (av 2.761)	19
$(Me_4N)_4K_2[Mo_6Se_8(CN)_6]\cdot 10\,H_2O$	0	2.700(2)–2.721(2) (av 2.711)	this work
$K_7[Mo_6Se_8(CN)_6]\cdot 8\,H_2O$	−1	2.700(3)	this work
$AgMo_6Se_8$	−1	2.701–2.776 (av 2.738)	20
$Ni_{0.67}Mo_6Se_8$	−1.33	2.649(2)–2.818(2) (av 2.730)	21
$PbMo_6Se_8$	−2	2.696(1)–2.735(1) (av 2.716)	22

independent temperature term. Similar magnetic properties were observed earlier for paramagnetic $Nb_6X_{12}^{n+}$ cluster compounds.[23]

Extended Hückel calculations for cluster anion $[Mo_6Se_8(CN)_6]^{6-}$ were performed by using the CACAO program.[24] The bond lengths and angles in the crystal structure of **2** were employed to model the anion. HOMO as well as LUMO bands are formed mainly by 4d Mo–Mo bonding orbitals and the contribution of Se, C, and N orbitals is very small. In the $[Mo_6Se_8(CN)_6]^{6-}$ ion all the energy levels are occupied up to −10.8 eV, and the energy gap between the HOMO and LUMO bands is about 0.59 eV. This explains why the $[Mo_6Se_8(CN)_6]^{6-}$ cluster anion in which all the electrons are paired (20 e per Mo_6 cluster) was found to be diamagnetic, whereas the $[Mo_6Se_8(CN)_6]^{7-}$ ion, which in effect contains one unpaired electron (21 e per Mo_6 cluster), is paramagnetic. The LUMO has bonding orbital character as supported by the fact that Mo–Mo distances in $[Mo_6Se_8(CN)_6]^{7-}$ cluster are shorter than those in $[Mo_6Se_8(CN)_6]^{6-}$.

In conclusion, the interaction of the Chevrel phase Mo_6Se_8 with molten KCN results in the formation of the highly charged paramagnetic $[Mo_6Se_8(CN)_6]^{7-}$ ion.

Experimental Section

1: Mo_6Se_8 (1.10 g, 0.91 mmol) and KCN (1.10 g, 16.9 mmol) were combined in a fused-silica tube that was evacuated and sealed. The tube was heated to 650 °C for 12 h, held at this temperature for 36 h, and then cooled to room temperature at 20 °C h^{-1}. The reaction mixture was washed with cool water (400 mL) to give a dark blue solution. The resulting solution was concentrated to 20 mL under heating, and then methanol (40 mL) was added to this solution. The dark blue suspension was filtered and the precipate was washed with methanol (20 mL) and diethyl ether (20 mL), and dried under vacuum. Yield: 0.91 g (56 %). Elemental analysis (%) calcd for $C_6H_{16}N_6O_8K_7Mo_6Se_8$ (1781.24): K 15.36, Mo 32.32; found: K 14.87, Mo 32.10; IR (KBr): $\tilde{\nu} = 2080$ cm^{-1} (CN). UV/Vis (H_2O): λ_{max} (ε) = 599 sh (2590), 560 (3040), 320 sh nm (11 600 mol^{-1}dm^{3}cm^{-1}). Differential thermogravimetry (DTG) analysis: one mass loss step (8.2 %) with the maximum in differential thermal analysis at about 105 °C which corresponds to the loss of eight water molecules (calcd 8.09 %). Cyclic voltammetry (scan rates: 20–200 mV s^{-1}, scanning interval −0.5 to 1.5 V vs. NHE, 0.1 M Na_2SO_4 aqueous solution): $E_{1/2} = 0.63$ V. Single crystals for X-ray structural analysis were obtained by slow evaporation of saturated solution of **1** at about 80 °C in the presence of KCN excess.

2: A blue solution of **1** (0.1 g) in H_2O (10 mL) was loaded at room temperature for 12 h under air. During this time the blue color of solution changed to brown. Me_4NCl (0.1 g) and KCN (0.02 g) were added to the brown solution. The resulting solution was refluxed for 10 min and cooled. The solution was slowly evaporated at room temperature to a volume of 1 mL. The precipitated dark brown crystals of **2** were filtered and dried under air. IR (KBr): $\tilde{\nu} = 2098$ cm^{-1} (CN); UV/Vis: λ_{max} (ε) = 455 nm (5006 mol^{-1} dm^3 cm^{-1}).

X-ray structural analyses: Structural data were collected by standard techniques at room temperature on Enraf Nonius CAD4 (**1**) and Siemens P4 (**2**) ($Mo_{K\alpha}$ radiation ($\lambda = 0.7107$ Å), $\theta/2\theta$ scan mode) diffractometers. Data were corrected for absorption with azimutal scans. The structures were solved by direct methods and were refined by the least-squares method against F^2. All non-hydrogen atoms were refined anisotropically taking into account all independent reflections. Hydrogen atoms of Me_4N^+ ions were refined with a riding model, those in water molecules were not located. All calculations were carried out on an IBM PC using the SHELX-97 program package.[25]

1: dark blue cube, crystal dimensions 0.14 × 0.14 × 0.14 mm^3, cubic, space group $Fm\bar{3}m$, $Z = 4$, $a = 15.552(2)$ Å, $V = 3761.5(8)$ Å^3, $\rho_{calcd} = 3.145$ g cm^{-3}, $\mu = 10.494$ mm^{-1}, 999 measured reflections, 311 independent, $R(F) = 0.0255$ and $R_w(F^2) = 0.0533$ for 132 reflection with $F_{hkl} \geq 4\sigma(F_{hkl})$.

2: dark brown plate, crystal dimensions 0.08 × 0.18 × 0.26 mm^3, triclinic, space group $P\bar{1}$, $Z = 1$, $a = 11.706(2)$, $b = 11.749(2)$, $c = 12.459(2)$ Å, $\alpha = 72.25(1)$, $\beta = 77.51(1)$, $\gamma = 63.04(1)°$, $V = 1448.5(4)$ Å^3, $\rho_{calcd} = 2.199$g cm^{-3}, $\mu = 6.476$ mm^{-1}, 5336 measured reflections, 5081 independent, $R(F) = 0.0622$ and $R_w(F^2) = 0.1429$ for 3106 reflection with $F_{hkl} \geq 4\sigma(F_{hkl})$.

Further details of the crystal structure investigation of **1** can be obtained from Fachinformationszentrum Karlsruhe, 76344 Eggenstein-Leopoldshafen, Germany (fax: +49-7247-808-666; e-mail: crysdata@fiz-karlsruhe.de) on quoting the depository number CSD-410778. Crystallographic data (excluding structure factors) for the crystal structure of **2** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 118276. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223 336-033; e-mail: deposit@ccdc.cam.ac.uk).

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[18] First stage refinement of **1** was carried out assuming full occupancy for both crystallographically independent K atom positions according to the rather unexpected formula "$K_9[Mo_6Se_8(CN)_6]$". But in this model K^+ ions had large isotropic thermal parameters. Furthermore, the occupancies of both K atoms were reduced and refined without any restraints; this led to a reduction in the *R* value. The resulting total number of K^+ ions in the unit cell was very close to 28 (about 28.5, 1.125 K per one anion). This value corresponds to chemical analysis data. Therefore, at present time there is no evidence for the existence of $K_x[Mo_6Se_8(CN)_6]$ compounds with $x > 7$. Therefore we concluded

that "$K_{7.125}$" is a result of inaccuracies associated with the refinement of the K^+ site occupancies from diffraction data (possibly due to the well-known correlation between occupancy and displacement parameters). Therefore the final refinement of **1** was carried out with the following K^+ occupancy restraints: occ.(K(1)) + 8 × occ.(K(2)) = 7.00(1). This gives the "K_7" formula and insignificant variations in the final residuals.
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