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# Tuning the dimensionality of H bonded networks using different chalcogen atoms in $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$ (Q = S, Se) clusters

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**Abstract** Two novel compounds based on  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  (Q = S, Se) and small H bond donor *bis*-amidinium dication of 2,2'-methylenediimidazolium (Cat)  $(\text{Cat})_2[\text{Re}_6\text{S}_8(\text{CN})_6]\cdot 2\text{H}_2\text{O}$  (**1**) and  $(\text{Cat})_2[\text{Re}_6\text{Se}_8(\text{CN})_6]\cdot 2\text{H}_2\text{O}$  (**2**) were synthesized and structurally characterized. Depending of the chalcogen atom (S or Se) in the cluster anion, the dimensionality of the obtained (2D and 3D) network and the nature of the H bond pattern change.

**Keywords:** rhenium cluster, dications, H bonds, amidinium, charge-assisted hydrogen bonding

## Introduction

The design of molecular networks [1-3] is based on the principles of self-recognition of complementary units, leading to fragments assembly, then to crystallization process. Using weak interactions leads to compounds presenting properties not encountered for other compounds like coordination polymers. For example, using halogen bonds may lead to interesting properties [4, 5], while the use of hydrogen bonds has encountered a large success during the last decades, with the formation of HOFs [6, 7]. But in hybrid hydrogen bonded networks, ionic hydrogen bonding [8] in combination with electrostatic interactions (charge-assisted hydrogen bonding, CAHB) [9], allows the formation of robust molecular networks with reliable recognition patterns. To illustrate the formation of hydrogen bond-based hybrid networks, the possibility of mastering the connectivity between the chosen starting components, as well as the packing of the formed entities in the crystalline phase has been illustrated, for example through the use of discrete cyanometallate H bond acceptors with organic cationic H bond donor compounds [10]. Hexacyanoferrate(II) or Hexacyanoferrate(III), assembled with cationic N Hydrogen bond donors bisamidinium derivatives leads to robust hydrogen bonded networks [11-14]. Using

$[M(CN)_2]^-$  (M=Ag or Au) anions, the reported compounds present luminescent properties [15, 16]. Other organometallic H bond acceptors can be used, and along this line, metallic clusters [17] appears to be candidate of choice due to their large size and rigid geometry. In a similar way to what was observed with cyanometallates, the cyanide metal clusters  $[M_6Q_8(CN)_6]^{n-}$  (M = Re, Mo, Q = S, Se, Te) have been used for the formation of extended coordination networks, “super Prussian blue” [18-21]. H bonded compounds have been also reported, and in addition, the formed networks present electronic and photophysical properties [22-25].

In this work, we intend to explore the formation of H bonded networks using a small H bond donor cationic species (cation of 2,2'-methylenediimidazolium,  $Cat^{2+}$ ), when combined with rhenium cyano clusters  $[Re_6Q_8(CN)_6]^{4-}$  (Q = S, Se) and the study of the influence of the chalcogen atom on the dimensionality of the obtained network and the nature of the H bond pattern.

## Experimental Part

FT-IR spectra were recorded on a Perkin Elmer ATR spectrometer.

Elemental analyses were performed by the Service de Micro-analyses de la Federation de Recherche Chimie of the University de Strasbourg.

### Crystallography

For **1** and **2**, the data were collected at 173(2) K on a Bruker APEX8 CCD diffractometer equipped with an Oxford Cryosystem liquid N<sub>2</sub> device, using graphite-monochromated MoK $\alpha$  ( $\lambda = 0.71073$  Å) radiation. The structure was solved using the program SHELXS-97 [26]. The refinement and all further calculations were carried out using SHELXL-2014 [27]. The H-atoms were included in calculated positions and treated as riding atoms using SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-squares on F<sup>2</sup>. A semi-empirical absorption correction was applied using SADABS in APEX2 [28].

CCDC Numbers for **1**: 2305609 and **2**: 2305608. Crystal data and structure refinement parameters are listed in Table 1.

### Synthesis

(Cat)Cl<sub>2</sub> was obtained as reported [29]. Cs<sub>4</sub>[Re<sub>6</sub>S<sub>8</sub>(CN)<sub>6</sub>] $\cdot$ 3H<sub>2</sub>O and Cs<sub>4</sub>[Re<sub>6</sub>Se<sub>8</sub>(CN)<sub>6</sub>] $\cdot$ 3H<sub>2</sub>O were obtained by recrystallization from concentrated aqueous solution of Cs<sub>3</sub>K[Re<sub>6</sub>S<sub>8</sub>(CN)<sub>6</sub>] $\cdot$ 2H<sub>2</sub>O [30] and K<sub>4</sub>[Re<sub>6</sub>Se<sub>8</sub>(CN)<sub>6</sub>] $\cdot$ 3.5H<sub>2</sub>O [31] with twenty-fold excess of CsCl.

Compounds (Cat)<sub>2</sub>[Re<sub>6</sub>S<sub>8</sub>(CN)<sub>6</sub>] $\cdot$ 2H<sub>2</sub>O (**1**) and (Cat)<sub>2</sub>[Re<sub>6</sub>Se<sub>8</sub>(CN)<sub>6</sub>] $\cdot$ 2H<sub>2</sub>O (**2**) were obtained using the same method: a 2 ml of water solution of organic (Cat)Cl<sub>2</sub> (C = 7.5 mM) was layered

in a thin tube on 3 ml of water solution of  $\text{Cs}_4[\text{Re}_6\text{Q}_8(\text{CN})_6]\cdot 3\text{H}_2\text{O}$  ( $\text{Q} = \text{S}$  or  $\text{Se}$ ) ( $\text{C} = 0.5$  mM) with addition of one drop of concentrated  $\text{HCl}$ . Yellow crystals suitable for X-ray diffraction were grown directly on the tube walls after few days. Yield 2–3 mg, ~ 40–50%.

**1:** CHN calculated for  $(\text{C}_9\text{N}_4\text{H}_{18})_2[\text{Re}_6\text{S}_8(\text{CN})_6]\cdot 2\text{H}_2\text{O}$  C 14.93, H 2.09, N 10.16; found C 14.91 H 12.05, N 10.17. EDS Re : S 6 : 7.8. IR  $\nu(\text{CN})$   $2110\text{ cm}^{-1}$ , also present all bands, corresponding to  $\text{Cat}^{2+}$ .

**2:** CHN calculated for  $(\text{C}_9\text{N}_4\text{H}_{18})_2[\text{Re}_6\text{Se}_8(\text{CN})_6]\cdot 2\text{H}_2\text{O}$  C 12.50, H 1.75, N 8.50; found C 12.48, H 1.73, N 8.48. EDS Re : Se 6 : 8.1. IR  $\nu(\text{CN})$   $2108, 2102\text{ cm}^{-1}$ , also present all bands, corresponding to  $\text{Cat}^{2+}$ .

## Results and discussion

The chemical structure of the cyanide metal clusters  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  ( $\text{Q} = \text{S}$  or  $\text{Se}$ ) [32] is displayed in Figure 1, together with the one of  $\text{Cat}^{2+}$  [29]. The octahedral  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  ( $\text{Q} = \text{S}$  or  $\text{Se}$ ) anions present six pendant terminal  $-\text{C}\equiv\text{N}$  moieties, orthogonally disposed at the vertices of the octahedral  $\text{Re}_6$  cluster (Figure 1a, b). As already reported in previous crystallographic studies, average distances between the nitrogen atoms of  $-\text{C}\equiv\text{N}$  groups within cluster are around  $7\text{ \AA}$  for  $-\text{C}\equiv\text{N}$  groups in *cis* position and  $10\text{ \AA}$  for the groups in *trans* position (Figure 1a, b). Compared to the parent flat bisamidinium cations [10],  $\text{Cat}^{2+}$  differs by its propensity to bent due to the short methylene spacer between the amidinium moieties, and the average distances between the N atoms of linked amidinium rings are  $3.15$  and  $4.02\text{ \AA}$  in the flat model (Figure 1c). This feature appears unfavorable for the regular dihapto recognition mode through H bond between the donors and acceptors.

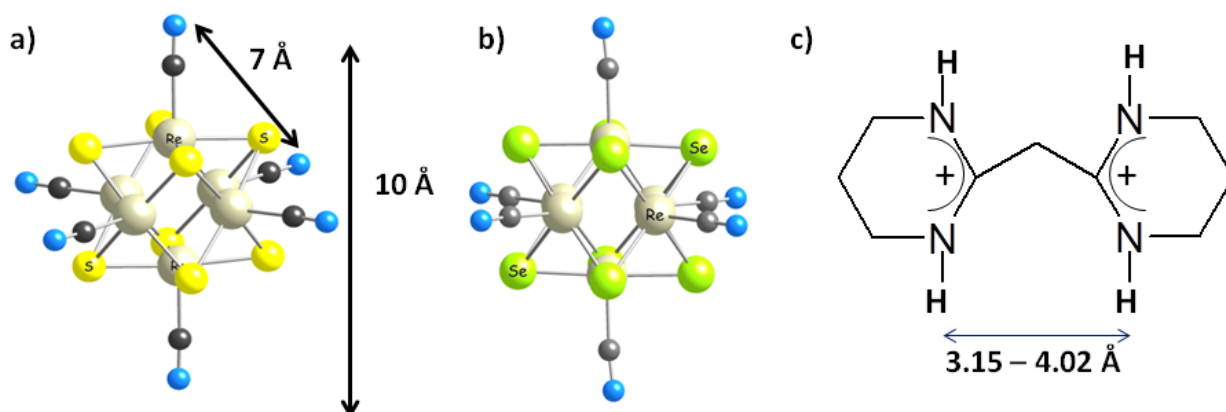


Figure 1. The individual components used for the formation of the H-bonded molecular networks, and their metrics a) Cluster anion  $[\text{Re}_6\text{S}_8(\text{CN})_6]^{4-}$ , b) Cluster anion  $[\text{Re}_6\text{Se}_8(\text{CN})_6]^{4-}$  and c) the organic cation  $\text{Cat}^{2+}$ .

## Structural study

Both **1** and **2** crystallize in the monoclinic  $P 2_1/n$  space group (see crystallographic Table 1) and contain two organic cations  $Cat^{2+}$  and one inorganic cluster anions  $[Re_6Q_8(CN)_6]^{4-}$  ( $Q = S$  or  $Se$ ), and two water molecules in the unit cell. Selected bond lengths are listed in Table 2 for **1** and **2**. In both compounds, the distances found in the  $[Re_6Q_8(CN)_6]^{4-}$  ( $Q = S$  or  $Se$ ) anions are in accordance with what has already been observed for those compounds [21] and are not discussed there. The structures will be described below. Both compounds are stable on air during long time. Compounds are not soluble in pure water, but can be soluble in alkaline solutions.

**Table 1.** Crystal data and structure refinement parameters for **1** and **2**, measured at 173K

Identification code	<b>1</b>	<b>2</b>
<b>Chemical formula</b>	$C_{24}H_{40}N_{14}O_2Re_6S_8$ ' $Re_6S_8C_6N_6$ , $2(C_9H_{18}N_4)$ , $2(H_2O)$ '	$C_{24}H_{40}N_{14}O_2Re_6Se_8$ ' $Re_6Se_8C_6N_6$ , $2(C_9H_{18}N_4)$ , $2(H_2O)$ '
<b>Formula weight g/mol</b>	1930.38	2305.58
<b>Temperature K</b>	173(2)	173(2)
<b>Wavelength Å</b>	0.71073	0.71073
<b>Crystal size mm</b>	0.030 x 0.030 x 0.040	0.030 x 0.040 x 0.040
<b>Color</b>	yellow	yellow
<b>Crystal system</b>	monoclinic	monoclinic
<b>Space group</b>	$P 2_1/n$	$P 2_1/n$
<b>Unit cell dimensions Å</b>	a = 10.0287(5) b = 11.5055(6) c = 18.4970(8) $\beta = 103.3^\circ$	a = 10.3092(3) b = 19.3074(5) c = 11.3566(3) $\beta = 97.9^\circ$
<b>Volume Å<sup>3</sup></b>	2076.83(17)	2238.99(11)
<b>Z</b>	2	2
<b>Density (calculated) g/cm<sup>3</sup></b>	3.087	3.420
<b>Absorption coefficient mm<sup>-1</sup></b>	17.863	22.700
<b>F(000)</b>	1752	2040
<b>Theta range for data collection °</b>	2.10 to 30.09	2.10 to 30.07
<b>Index ranges</b>	$-14 \leq h \leq 14$ $-16 \leq k \leq 16$ $-22 \leq l \leq 25$	$-12 \leq h \leq 13$ $-27 \leq k \leq 27$ $-16 \leq l \leq 16$
<b>Reflections collected</b>	22907	115474
<b>Independent reflections</b>	5859 [R(int) = 0.0386]	6361 [R(int) = 0.0397]
<b>Max. and min. transmission</b>	0.6190 and 0.5360	0.5510 and 0.4680
<b>Data / restraints / parameters</b>	5859 / 3 / 250	6361 / 3 / 250
<b>Goodness-of-fit on F<sup>2</sup></b>	1.003	1.006
$\Delta/\sigma_{max}$	0.001	0.005
<b>Final R indices</b>	5041 data; $I > 2\sigma(I)$ $R_1 = 0.0229$ , $wR_2 = 0.0400$ all data $R_1 = 0.0307$ , $wR_2 = 0.0418$	5550 data; $I > 2\sigma(I)$ $R_1 = 0.0159$ , $wR_2 = 0.0356$ all data $R_1 = 0.0231$ , $wR_2 = 0.0382$
<b>Largest diff. peak and hole eÅ<sup>-3</sup></b>	0.982 and -1.036	0.763 and -1.085
<b>R.M.S. deviation from mean eÅ<sup>-3</sup></b>	0.221	0.169

## Structure of compound 1

**1** is described as a 2D H-bonded system (figure 2a). Each  $[\text{Re}_6\text{S}_8(\text{CN})_6]^{4-}$  anionic cluster is surrounded by four cations oriented in two directions of space and each of the cations is surrounded by two anions, so that the anion : cation ratio is equal to 1 : 2 as suggested by the charge balance of both ions (figure 2b, c). In the dication, there are two opposite nitrogen atoms from amidinium moieties that are not involved in H bonds. Each of the  $\text{C}\equiv\text{N}$  moieties of  $[\text{Re}_6\text{S}_8(\text{CN})_6]^{4-}$  are connected through H bonds in the basal plane with  $\text{N}\dots\text{N}$  distances of 3.047(5) and 3.080(5) Å, thus forming a corrugated 2D H-bonded network and the two apical  $\text{C}\equiv\text{N}$  moieties of  $[\text{Re}_6\text{S}_8(\text{CN})_6]^{4-}$  form H bonds with a water molecule at  $\text{N}\dots\text{O}$  distance of 2.819(6) Å, pointing at the surface of the corrugated plane. Thus, water molecules are located between layers and linked with another layer with  $\text{O}\dots\text{N}$  distance 3.157(5) Å. The  $\text{Cat}^{2+}$  dication, due to the  $\text{sp}^3$  character of the carbon linker between the amidinium moieties, is bent so that the layers are relatively thick (*ca* 8Å). As already mentioned, the recognition mode through H bond between the donors and acceptors is different from dihapto, due to the flexibility of  $\text{Cat}^{2+}$ , and in the present case, the recognition mode is bis-monohapto.

The 2D layers are stacked along the *a* axis, without interactions between them (figure 2d).

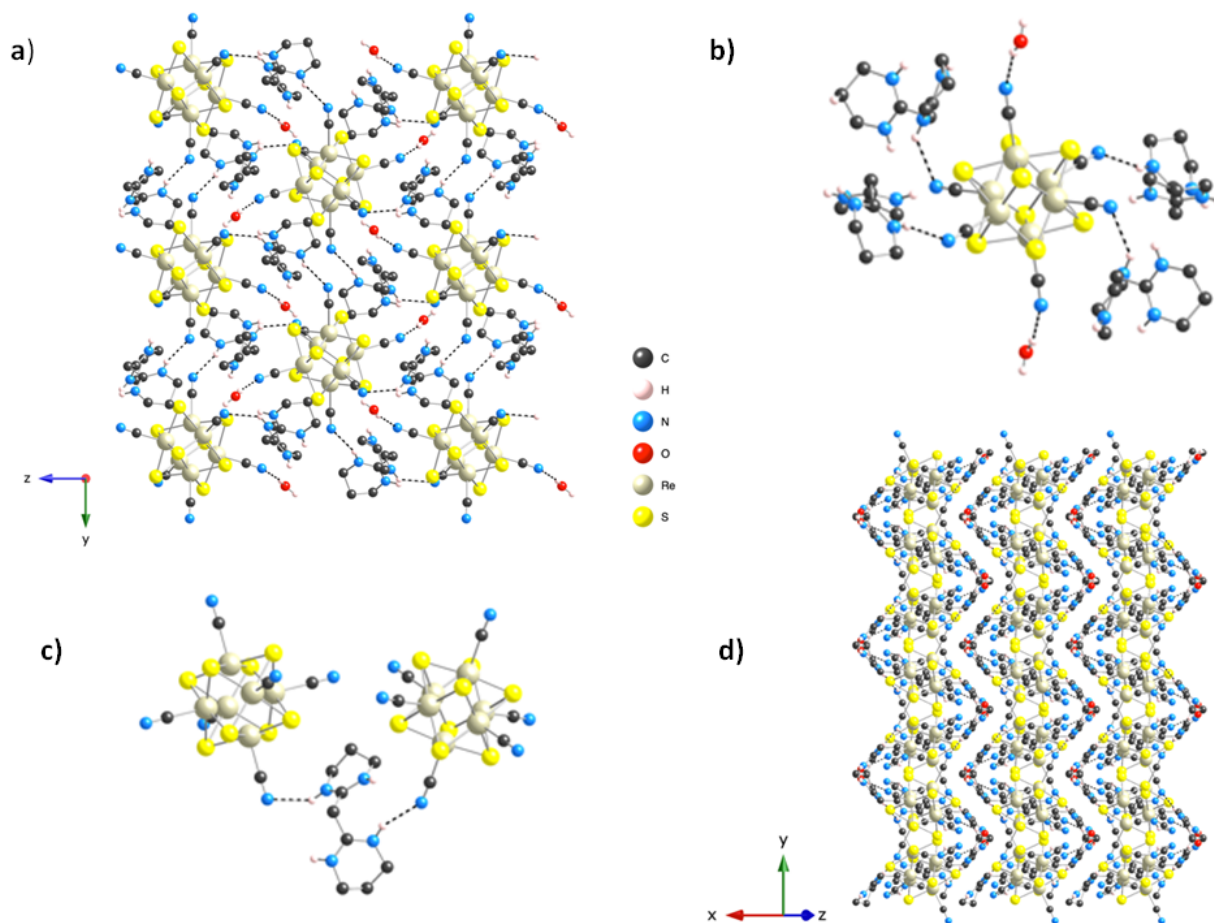


Figure 2. For compound **1**, (a) The layered structure in the *yOz* plane; (b) the surrounding around the  $[\text{Re}_6\text{S}_8(\text{CN})_6]^{4-}$  anions, (c) surrounding around  $\text{Cat}^{2+}$  dication; (d) the packing of the formed

layers along the a axis. Black dashed lines are representing hydrogen bonds between cluster and organic cation/water.

Table 2. Selected bond length, Å min–max, average

	<b>1</b>	<b>2</b>
Re–Re	2.5951(2) – 2.6051(2) 2.6014(2)	2.624(1) – 2.631(1) 2.628 (1)
Re–Q (Q = S or Se)	2.3965(10) – 2.4136(10) 2.408(1)	2.5077(3) – 2.5314(3) 2.5215(3)
Re–C	2.111(4) – 2.129(4) 2.119(4)	2.101(3) – 2.107(3) 2.104(3)
N...N	3.047(5) 3.080(5)	2.901(4) 2.918(4)
N...O	2.819(6)	2.760(4)

### Structure of compound 2

In compound **2**, each  $[\text{Re}_6\text{Se}_8(\text{CN})_6]^{4-}$  anionic cluster is surrounded by six cations oriented in the three directions of space and each of the  $\text{Cat}^{2+}$  dication is surrounded by three anions so that the anion : cation ratio is equal to 1 : 2 (figures 3b, c). In the dication, there is one nitrogen atom from amidinium moieties that is not involved in H bonds with  $\text{C}\equiv\text{N}$  moieties, but with a molecule of water, with a short N...O distance of 2.760(4) Å. The coordinated anions form thus, through H-bonds, a 3D H bonded network (figure 3a) with N...N distances (between amidinium and cyano) equal to 2.901(4), 2.918(4) and 2.960(4) Å. The recognition mode through H bond between the  $\text{Cat}^{2+}$  donors and  $[\text{Re}_6\text{Se}_8(\text{CN})_6]^{4-}$  acceptors is tris-monohapto.

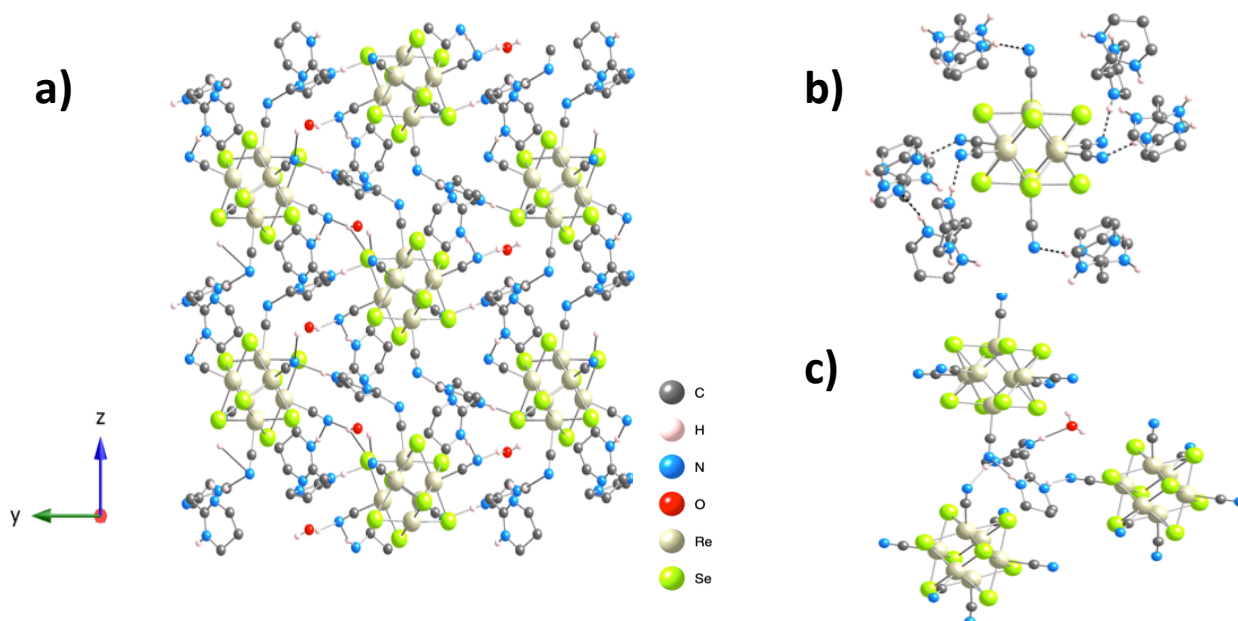


Figure 3. For compound **2**, (a) The 3D H bonded structure in the yOz plane, (b) the surrounding of the  $[\text{Re}_6\text{Se}_8(\text{CN})_6]^{4-}$  anions and (c)  $\text{Cat}^{2+}$  dication. Black dashed lines are representing hydrogen bonds between cluster and organic cation/water.

## Conclusion

We illustrated the possibility of forming hydrogen bonded compounds through the recognition of the amidinium H donor atoms with the cyano appended  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  (Q = S or Se) cluster building blocks. Depending on the nature of Q, the H bonded networks display either a 2D (Q=S) or a 3D dimensionality (Q=Se). The difference of dimensionality can be explained by the different recognition mode with the  $\text{C}\equiv\text{N}$  moieties appended on the anionic cluster with the H bond donor cation. The presence of Se atoms in  $[\text{Re}_6\text{Se}_8(\text{CN})_6]^{4-}$  allows the recognition of the six  $\text{C}\equiv\text{N}$  moieties, probably due to an increase of the electronic density as attested by shorter N-N distances. Whereas for  $[\text{Re}_6\text{S}_8(\text{CN})_6]^{4-}$ , only 4 of the 6  $\text{C}\equiv\text{N}$  moieties are involved in recognition with  $\text{Cat}^{2+}$  dication, and the N-N distances are longer attesting a weaker charge-assisted hydrogen bond between them.

These results are interesting for the control of the networks connectivity by using  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  (Q = S or Se) involved in different number of hydrogen bonds.

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