



# Novel inorganic ionic compounds based on $\text{Re}_6$ chalcocyanide cluster complexes: synthesis and crystal structures of $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{S}_8(\text{CN})_6] \cdot 7\text{H}_2\text{O}$ , $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Se}_8(\text{CN})_6]$ and $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot \text{H}_2\text{O}$

Konstantin A. Brylev<sup>a</sup>, Nikolai G. Naumov<sup>a</sup>, Gabriel Peris<sup>b</sup>, Rosa Llusar<sup>c</sup>, Vladimir E. Fedorov<sup>a,\*</sup>

<sup>a</sup> *Nikolaev Institute of Inorganic Chemistry of Russian Academy of Sciences, 3 Acad. Lavrentiev Prospect, 630090 Novosibirsk, Russia*

<sup>b</sup> *Servei Central d'Instrumentació Científica, Campus Riu Sec, Universitat Jaume I, POB 224, 12071 Castello, Spain*

<sup>c</sup> *Departament de Ciències Experimentals, Campus Riu Sec, Universitat Jaume I, POB 224, 12071 Castello, Spain*

Received 5 May 2003; accepted 14 August 2003

## Abstract

Three new octahedral rhenium chalcocyanide cluster compounds  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{S}_8(\text{CN})_6] \cdot 7\text{H}_2\text{O}$  (**1**),  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Se}_8(\text{CN})_6]$  (**2**) and  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot \text{H}_2\text{O}$  (**3**) exhibiting ionic structures have been obtained by the diffusion of an ammonia solution of  $\text{KCs}_3[\text{Re}_6\text{S}_8(\text{CN})_6]$  (for **1**),  $\text{K}_4[\text{Re}_6\text{Se}_8(\text{CN})_6] \cdot 3.5\text{H}_2\text{O}$  (for **2**) or  $\text{Cs}_4[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot 2\text{H}_2\text{O}$  (for **3**) into a glycerol solution of  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  in the presence of trien (trien = triethylenetetramine). The compounds have been characterized by single-crystal X-ray diffraction. All three compounds contain a cationic complex  $[\text{CuNH}_3(\text{trien})]^{2+}$  which was not described previously. © 2003 Published by Elsevier Ltd.

**Keywords:** Rhenium; Clusters; Copper; Sulfur; Selenium; Tellurium

## 1. Introduction

The chemistry of octahedral rhenium cluster chalcocyanide anions  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-/-3-}$  ( $\text{Q} = \text{S}, \text{Se}, \text{Te}$ ) has been developed extensively in recent years [1–13]. A remarkable feature of these complexes is the ambidentate character of the outer cyano ligands. Interaction of these anions with aqua cations of different metals  $[\text{M}(\text{H}_2\text{O})_x]^{n+}$  may result in a partial or total replacement of their coordination water molecules by nitrogen atoms of the more nucleophilic CN ligand. Such interaction has the potential to yield polymeric structures with different dimensionalities (3D, 2D and 1D) where all or some of the CN ligands are coordinated to cations via  $\text{Re}-\text{C} \equiv \text{N}-\text{M}$  bridges.

It is known that specific complexation of cations plays an important role in the construction of molecular

architectures. Modification of the cation environment by blocking its coordination sites with ligands competing with nitrogen atoms of the CN groups can restrict its coordination possibilities. Partial blocking allows the adjustment of the dimensionality of these polymeric compounds (down to complexes with molecular structure). In the case of full cation blocking it is possible to prevent the formation of a polymeric structure in favor of ionic structures.

In this work we present the synthesis and structural study of three new ionic compounds  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{S}_8(\text{CN})_6] \cdot 7\text{H}_2\text{O}$  (**1**),  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Se}_8(\text{CN})_6]$  (**2**) and  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot \text{H}_2\text{O}$  (**3**) in which the  $\text{Cu}^{2+}$  cation is completely blocked from interaction with cyanide ligands of cluster anions. Information about the cationic complex  $[\text{CuNH}_3(\text{trien})]^{2+}$  in the Cambridge Structural Database has not been found, from which it is possible to conclude that this is the first example of a copper(II) complex with such ligand environment.

\* Corresponding author. Fax: +7-3832-344489.

E-mail address: [fed@che.nsk.su](mailto:fed@che.nsk.su) (V.E. Fedorov).

## 2. Experimental

Original cluster complexes  $\text{KCs}_3[\text{Re}_6\text{S}_8(\text{CN})_6]$  [14],  $\text{K}_4[\text{Re}_6\text{Se}_8(\text{CN})_6] \cdot 3.5\text{H}_2\text{O}$  [15] and  $\text{Cs}_4[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot 2\text{H}_2\text{O}$  [16] were prepared as previously described. The syntheses of title compounds were carried out in narrow diameter tubes. The IR spectra were recorded on a Bruker IFS-85 IR Fourier spectrometer (KBr tablets). Elemental analyses were carried out on a Carlo Erba 1106 instrument.

### 2.1. Synthesis of $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{S}_8(\text{CN})_6] \cdot 7\text{H}_2\text{O}$ (**1**)

Reaction was carried out by layering 1.0 mg (0.51  $\mu\text{mol}$ ) of  $\text{KCs}_3[\text{Re}_6\text{S}_8(\text{CN})_6]$  in 0.5 ml of aqueous ammonia solution on 3.5 mg (0.02 mmol) of  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  in 0.5 ml of glycerol in which 0.05 ml (0.33 mmol) of triethylenetetramine was added. After 1 week dark green crystals were obtained. Yield was quantitative based on Re. *Anal. Calc.* for  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{S}_8(\text{CN})_6] \cdot 7\text{H}_2\text{O}$ : C, 10.25; H, 2.68; N, 10.62. Found: C, 10.33; H, 2.54; N, 10.37%. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu(\text{CN}) = 2111$  and  $2126$ .

### 2.2. Synthesis of $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Se}_8(\text{CN})_6]$ (**2**)

In a similar manner dark red–brown crystals were obtained by layering 1.0 mg (0.47  $\mu\text{mol}$ ) of  $\text{K}_4[\text{Re}_6\text{Se}_8$

$(\text{CN})_6] \cdot 3.5\text{H}_2\text{O}$  in 0.5 ml of aqueous ammonia solution on 3.5 mg (0.02 mmol) of  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  in 0.5 ml of glycerol in which 0.05 ml (0.33 mmol) of triethylenetetramine was added. Yield was quantitative based on Re. *Anal. Calc.* for  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Se}_8(\text{CN})_6]$ : C, 9.17; H, 1.79; N, 9.50. Found: C, 9.41; H, 1.74; N, 9.42%. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu(\text{CN}) = 2080$  and  $2113$ .

### 2.3. Synthesis of $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot \text{H}_2\text{O}$ (**3**)

About 1.0 mg (0.35  $\mu\text{mol}$ ) of  $\text{Cs}_4[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot 2\text{H}_2\text{O}$  was employed in the procedure described for **1** and **2**. The yield of dark red–brown crystals was quantitative based on Re. *Anal. Calc.* for  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Te}_8(\text{CN})_6] \cdot \text{H}_2\text{O}$ : C, 7.82; H, 1.60; N, 8.10. Found: C, 7.82; H, 1.73; N, 7.81%. IR (KBr,  $\text{cm}^{-1}$ ):  $\nu(\text{CN}) = 2070$ ,  $2081$  and  $2090$ .

### 2.4. X-ray structural analysis

Crystals of **1**, **2** and **3** were mounted on the tip of a glass fibre with the use of grease. X-ray diffraction experiments were carried out on a Bruker SMART CCD diffractometer using Mo  $\text{K}\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at  $-50 \text{ }^\circ\text{C}$ . The data were collected with a frame width of  $0.3^\circ$  in  $\omega$  and a counting time of 25 s per frame for compounds **1** and **3** and 20 s per frame for compound **2**

Table 1

Crystal data and structure refinement for  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Q}_8(\text{CN})_6] \cdot n\text{H}_2\text{O}$ , Q = S (**1**), Se (**2**) and Te (**3**)

Compound	<b>1</b>	<b>2</b>	<b>3</b>
Empirical formula	$\text{C}_{18}\text{H}_{56}\text{Cu}_2\text{N}_{16}\text{O}_7\text{Re}_6\text{S}_8$	$\text{C}_{18}\text{H}_{42}\text{Cu}_2\text{N}_{16}\text{Re}_6\text{Se}_8$	$\text{C}_{18}\text{H}_{44}\text{Cu}_2\text{N}_{16}\text{ORe}_6\text{Te}_8$
Weight (g mol $^{-1}$ )	2109.55	2358.64	2765.77
Crystal system	monoclinic	triclinic	triclinic
<i>a</i> (Å)	11.189(4)	9.342(3)	9.747(3)
<i>b</i> (Å)	24.229(8)	11.190(3)	11.133(4)
<i>c</i> (Å)	17.997(6)	11.353(3)	11.625(4)
$\alpha$ (°)	90.00	68.130(6)	68.094(7)
$\beta$ (°)	91.822(8)	88.827(6)	88.757(8)
$\gamma$ (°)	90.00	81.697(6)	84.683(7)
<i>V</i> (Å $^3$ )	4876(8)	1089.2(5)	1165.2(7)
Space group ( <i>Z</i> )	$P2_1/n$ (4)	$P\bar{1}$ (1)	$P\bar{1}$ (1)
<i>D<sub>c</sub></i> (g cm $^{-3}$ )	2.873	3.596	3.941
Crystal size (mm)	0.310 × 0.270 × 0.150	0.418 × 0.288 × 0.275	0.187 × 0.125 × 0.079
$\mu(\text{Mo K}\alpha)$ (mm $^{-1}$ )	16.077	24.274	21.354
Ratio of minimum to maximum apparent transmission	0.45	0.15	0.51
$\theta$ range (°)	1.93–30.52	1.89–30.65	1.68–30.6
Reflections collected	39302	8692	9411
Unique reflections/ <i>R<sub>int</sub></i>	14 609/0.0764	6203/0.0390	6633/0.0359
Parameters refined	535	227	236
$R_1^a/wR_2^b$ ( $I > 4\sigma$ )	0.0518/0.1176	0.0498/0.1325	0.0428/0.1040
$R_1^a/wR_2^b$ (all data)	0.0818/0.1313	0.0615/0.1393	0.0591/0.1114
GOF $^c$	0.982	1.039	1.013
Maximum shift (esd)	0.153/0.004	0.002/0.000	0.001/0.000
Largest peak, hole (eÅ $^{-3}$ )	3.697/−1.688	5.832/−3.952	4.062/−1.889

$^a R_1 = \sum ||F_o| - |F_c|| / \sum F_o$ .

$^b wR_2 = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$ .

$^c \text{GOF} = [\sum w(F_o^2 - F_c^2)^2 / (n_{\text{obs}} - n_{\text{par}})]^{1/2}$ .

at a crystal-to-detector distance of 4 cm. The diffraction frames were integrated using the SAINT package and corrected for absorption with SADABS [17,18]. All structures were solved by direct methods and refined by the full-matrix method based on  $F^2$  using the SHELXTL software package [19]. The crystal parameters and basic information relating data collection and structure refinement for compounds **1**, **2** and **3** are summarized in Table 1.

### 3. Results and discussion

The reactions of  $M_4[Re_6Q_8(CN)_6]$  ( $M = K, Cs$ ;  $Q = S, Se, Te$ ) with metal salts in aqueous solutions lead to the formation of cyano bridged cluster complexes, in which the cations  $M^{n+}$  may be coordinated by one, two, three, four or six N atoms of CN ligands of the cluster anions [11,13]. The  $M-(OH_2)$  bonds in aqua complexes are weaker than  $M-(NC)$  bonds and hence ligand exchange occurs. However, some other N donor ligands (ammonia, ethylenediamine, etc.) by reason of their high nucleophilicity may compete with nitrogen of CN groups. Therefore, the addition of similar substances in the reaction solution results in formation of metal complexes with blocked coordination sites. Existence of partial or full blocked cations reduces the dimensionality or prevents the formation of polymeric structures.

It is known that effective blocking of cation coordination sites may be carried out by chelating polydentate ligands [13,20–22].

We now extend our investigations in this area to metal complexes with trien. Here we present three new cluster compounds, namely  $[CuNH_3(trien)]_2[Re_6S_8(CN)_6] \cdot 7H_2O$ ,  $[CuNH_3(trien)]_2[Re_6Se_8(CN)_6]$  and  $[CuNH_3(trien)]_2[Re_6Te_8(CN)_6] \cdot H_2O$ . All compounds were prepared under similar conditions by layering an aqueous ammonia solution of the corresponding cluster salt with a glycerol solution of  $CuCl_2$  in which triethylenetetramine was added.

All these compounds possess ionic structures, i.e. cations and anions are not covalently bonded to each other. The crystal structure of **1** consists of the packing of four cations and four anions in the unit cell connected though extended hydrogen bonds as shown in Fig. 1.

Compounds **2** and **3** are isomorphous with one cation and one anion per unit cell. In contrast with complex **1**, the  $Re_6Q_8$  cluster units in **2** and **3** are connected along the “a” axis through intermolecular  $Q-Q$  distances as show in Fig. 2 for the selenium and tellurium derivatives. The intercluster tellurium–tellurium distances are 3.68 Å while the equivalent selenium–selenium distances are 0.04 Å longer. The columns of clusters in **2** and **3** are connected through  $[CuNH_3(trien)]^{2+}$  cations via hydrogen bonds as represented in Fig. 3.

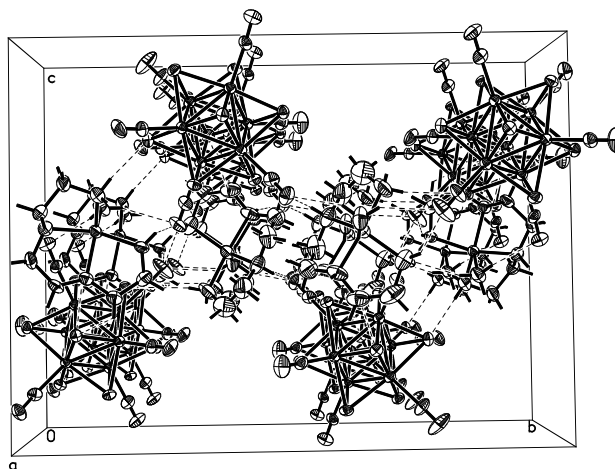


Fig. 1. Packing diagram for  $[CuNH_3(trien)]_2[Re_6S_8(CN)_6] \cdot 7H_2O$  (**1**) with part of the hydrogen bonding network.

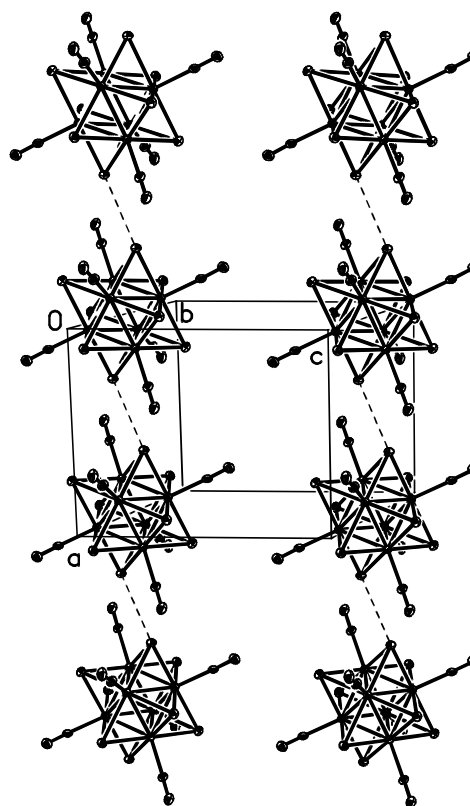


Fig. 2. Packing of the  $[Re_6Q_8(CN)_6]^{4-}$  anions along the “a” axis in structures (**2**) and (**3**). The dotted lines show the chalcogen...chalcogen contacts.

In all three compounds, the complex cation  $[CuNH_3(trien)]^{2+}$  has a square pyramid environment with four short bonds in the base, formed by the trien molecule, and one elongated bond which is formed by the ammonia molecule located in the vertex of the pyramid (see Fig. 4). Distances between copper and nitrogen atoms of trien vary from 2.015(9) to 2.079(10) Å in

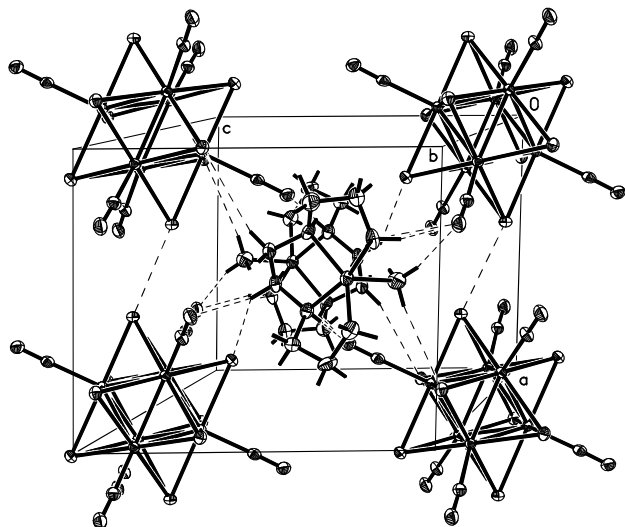


Fig. 3. Hydrogen bond scheme connecting columns of clusters through  $[\text{CuNH}_3(\text{trien})]^{2+}$  cations in structures (2) and (3).

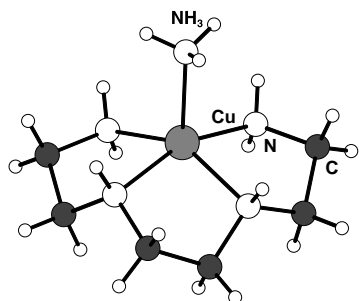


Fig. 4. A view of cationic complex  $[\text{CuNH}_3(\text{trien})]^{2+}$ .

**1**, from 2.028(8) to 2.053(8) Å in **2**, from 2.021(7) to 2.046(7) Å in **3**. Distances between copper and nitrogen atoms of ammonia are 2.118(9) and 2.150(8) Å in **1** (this compound has two crystallographically independent copper cations), 2.178(8) Å in **2**, 2.152(8) Å in **3**.

The geometry of the anion fragments  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  ( $\text{Q} = \text{S}, \text{Se}, \text{Te}$ ) found in the structures are similar to those in starting and related compounds. Table 2 presents a summary of relevant bond distances and angles for compounds **1**, **2** and **3**. The  $\text{Re}_6$  cluster is an almost ideal octahedron. The  $\text{Re}-\text{Re}$  bond distances vary from 2.5953(8) to 2.6071(8) Å in **1**, from 2.5253(10) to 2.6396(7) Å in **2**, from 2.6808(10) to 2.7044(9) Å in **3**. The chalcogen atoms are capping each triangular face of

Table 2

Selected averaged bond distances and angles for  $[\text{CuNH}_3(\text{trien})]_2[\text{Re}_6\text{Q}_8(\text{CN})_6] \cdot n\text{H}_2\text{O}$ ,  $\text{Q} = \text{S}$  (**1**),  $\text{Se}$  (**2**) and  $\text{Te}$  (**3**)

Bond	<b>1</b>	<b>2</b>	<b>3</b>
$\text{D}(\text{Re}-\text{Re})$ (Å)	2.601(3)	2.637(3)	2.686(6)
$\text{D}(\text{Re}-\text{Q})$ (Å)	2.496(5)	2.528(4)	2.697(5)
$\text{D}(\text{Re}-\text{C})$ (Å)	2.114(9)	2.104(10)	2.103(9)
$\text{Re}-\text{Q}-\text{Re}$ (°)	65.44(18)	62.89(12)	59.8(2)

the  $\text{Re}_6$  octahedron. The  $\text{Re}-\text{Q}$  bonds vary from 2.396(2) to 2.417(2) Å in **1**, from 2.5155(10) to 2.5338(11) Å in **2**, from 2.6841(9) to 2.7044(9) Å in **3**. Six cyanide ligands coordinate to rhenium atoms through carbon sticking out in pairs in a mutually perpendicular fashion.

An extensive system of weak hydrogen bonds joins the ionic fragments  $[\text{CuNH}_3(\text{trien})]^{2+}$  and  $[\text{Re}_6\text{Q}_8(\text{CN})_6]^{4-}$  to one another and to water molecules in the structures **1**–**3**. Shortest contacts of a given type include  $\text{N}-\text{H} \cdots \text{N}$  hydrogen bonds between  $\text{NH}_3$  and  $\text{CN}$  groups (3.16 Å in **1**; 3.30 Å in **2**; 3.35 Å in **3**),  $\text{N}-\text{H} \cdots \text{N}$  hydrogen bonds between  $\text{NH}_2$  and  $\text{CN}$  groups (3.01 Å in **1**; 2.92 Å in **2**; 2.94 Å in **3**),  $\text{N}-\text{H} \cdots \text{O}$  bonds between  $\text{NH}_3$  groups and  $\text{H}_2\text{O}$  molecules (3.08 Å in **1**),  $\text{N}-\text{H} \cdots \text{O}$  bonds between  $\text{NH}_2$  groups and  $\text{H}_2\text{O}$  molecules (2.91 Å in **1**), and  $\text{O}-\text{H} \cdots \text{N}$  bonds (2.99 in **1**, 3.07 Å in **3**) between  $\text{H}_2\text{O}$  molecules and  $\text{CN}$  groups.

A search through the Cambridge Structural Database shows that up to date only two trien and ammonia containing complexes have been reported, namely  $[\text{CoNH}_3(\text{trien})\text{Cl}](\text{NO}_3)_2$  [23] and  $[\text{CoNH}_3(\text{trien})\text{Cl}]\text{S}_5\text{O}_6$  [24]. Both compounds contain the cationic complex  $[\text{Co}(\text{NH}_3)(\text{trien})\text{Cl}]^{2+}$  where trien and ammonia are present in the coordination sphere of  $\text{Co}(\text{III})$  simultaneously. This ligand environment has not been described for other metals.

#### 4. Supplementary material

Supplementary data are available free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; Email: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk) or [www: http://www.ccdc.cam.ac.uk](http://www.ccdc.cam.ac.uk)) on request, quoting the deposition numbers CCDC 208414–208416.

#### Acknowledgements

This work was supported by INTAS (Grant INTAS-00-00689) and the Russian Foundation for Basic Research (Grants 02-03-32264 and 03-03-06114). We are most indebted to the ‘‘Servei Central d’Instrumentació Científica’’ (SCIC) of the University Jaume I for the use of X-ray facilities.

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