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New octachlorodirhenate(III) salts: solid state manifestation for a certain conformational flexibility of the $[Re_2Cl_8]^{2-}$ ion

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Abstract

Glycinium and β-alaninium compounds $[GlyH]_2Re_2Cl_8 \cdot H_2O$ **1**, $[\beta\text{-AlaH}]_2Re_2Cl_8$ **2**, and $[GlyH]_4(Re_2Cl_8)Cl_2$ **3** were synthesized and characterized by X-ray diffraction. The $Re_2Cl_8^{2-}$ anion in structure **3** has a partially staggered conformation with unprecedentedly high rotational distortion (16.2°). The forced geometry is influenced by cooperative demands of four hydrogen bonds Cl···HO and a set of short van der Waals interactions. Consequent weakening of the δ-component results in a certain elongation of the quadruple Re–Re bond (2.2407(3) vs. 2.2374(8) Å for **2**).

Keywords: Rhenium; Quadruple bond; Octachlorodirhenate

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1. Introduction

Octachlorodirhenates(III) are paradigmatic species in the realm of transition metal clusters [1–3]. Unusually short metal–metal distance and fully eclipsed conformation of $Re_2Cl_8^{2-}$ were the important factors for discovery of a quadruple bonding over three decades ago [1,4]. Chemistry of the dirhenium(III) compounds is rich and versatile, they may retain a metal–metal bond in many reactions including substitution of the ligands and oxidation [3–6]. Some of these reactions led to compounds with a lower rhenium–rhenium bond order [1,7]. Two evident factors influencing the strength of the δ–δ bond in these species are interatomic distance M–M and rotational distortion of the M_2X_8 core from 0° (eclipsed) where the δ–δ overlap is maximal to 45° where the overlap becomes zero [1,2,8].

The $Re_2Cl_8^{2-}$ ion has a weak electrophilic character and the metal atoms may coordinate additional ligands at the axial position that is *trans*- to the Re–Re bond. This coordination effects elongation of the quadruple bond as was shown by examination of $Cs_2[Re_2Cl_8] \cdot H_2O$ [9] that comprises both the isolated $Re_2Cl_8^{2-}$ ions and the complex $[Re_2Cl_8(H_2O)_2]^{2-}$ groups (Δ(Re–Re) is approximately 0.02 Å). Thus the controllable binding of an axial ligand and control over conformation of $Re_2Cl_8^{2-}$ may be viewed as tools for control over the strength of the δ–δ bond in the ion. However, under many conditions the additional ligands easily substitute chlorine atoms giving a number of anionic and neutral products instead of $[Re_2Cl_8(L)_2]^{2-}$ associates [10]. In water solutions $Re_2Cl_8^{2-}$ ions themselves are stable only under very low pH values when the most common ligands exist as protonated cationic species and cannot provide formation of $[Re_2Cl_8(L)_2]^{2-}$ complexes. In this context we have examined amino acids that readily yield cations for generation of neutral $Kat_2[Re_2Cl_8]$ composites, but also retain donor character of a carbonyl group in highly acidic media. Interaction in this system is interesting also in the supramolecular sense as both counterparts possess a set of complementary binding sites for coordination and hydrogen bonding and presumably can associate in a very characteristic fashion. Herein we report preparation of the compounds based upon cationic glycine and β-alanine and the structure in which the $Re_2Cl_8^{2-}$ core has unprecedent-

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edly high rotational distortion influenced by the entire hydrogen bonding and packing forces.

2. Experimental

2.1. Synthesis

$[GlyH]_2Re_2Cl_8 \cdot H_2O$ **1** and $[GlyH]_4(Re_2Cl_8)Cl_2$ **3**. 8.0 g glycine was dissolved in 25 ml water and the solution was saturated at 0 °C with hydrogen chloride. Precipitated glycinium chloride was filtered off and 0.30 g $Re_2(AcO)_2Cl_4(H_2O)_2$ [11] was dissolved in the filtrate at 0 °C. The mixture was stored at −5 °C for 2 d after which large deep-blue crystals of **1** (0.19 g, 50%) were separated and dried over H_2SO_4. Complex **3** was prepared in a similar fashion: the resulting reaction mixture with $[GlyH]_2Re_2Cl_8 \cdot H_2O$ crystals was heated to 40 °C until total dissolution of the compound was observed. This was accompanied with a partial elimination of excess hydrogen chloride from the solution. The mixture was left overnight at −5 °C and brown crystals $[GlyH]_4(Re_2Cl_8)Cl_2$ **3** (0.21 g, 45%) were filtered and dried over H_2SO_4. *Anal.* Found for **1**: Re, 44.72; Cl, 34.07. Calc. for $C_4H_{14}Cl_8N_2O_5Re_2$: Re, 45.08; Cl, 34.33. Found for **3**: Re, 35.69; Cl, 34.12. Calc. for $C_8H_{24}Cl_{10}N_4O_8Re_2$: Re, 36.11; Cl, 34.38%.

[β-AlaH][Re_2Cl_8] **2**. 7.0 g β-Alanine was dissolved in 30 ml water and the solution was saturated at 0 °C with hydrogen chloride; 20 ml of such mixture was used as a solvent for 0.15 g $Re_2(AcO)_2Cl_4(H_2O)_2$. The resulting dark-blue mixture was left at −5 °C for 2 d after which large brown crystals of $[\beta\text{-AlaH}]_2Re_2Cl_8$ **2** were filtered and, without washing, dried over H_2SO_4. The yield was 0.17 g (92%). *Anal.* Found: Re, 44.17; Cl, 33.64. Calc. for $C_6H_{16}Cl_8N_2O_4Re_2$: Re, 44.53; Cl, 33.92%.

The compounds lose crystallinity in wet air. They are stable in dry air or under argon and are well soluble in 3 M HCl, solutions of hydrogen chloride in alcohols and immediately hydrolyze in water. The compounds are sparingly soluble in acidic solutions of the amino acid hydrochlorides and this was also used for convenient preparations starting with the perrhenate salts.

$[GlyH]_2Re_2Cl_8 \cdot H_2O$ **1**. 15 ml water, 1 ml 10 M hydrochloric acid, 20 g glycine, 7.0 g (27.2 mmol) $LiReO_4$ and 25.0 g (110.8 mmol) $SnCl_2 \cdot 2H_2O$ were placed in a flask and stirred under argon at 90–95 °C for 1.5 h. The resulting black mixture was diluted with 150 ml concentrated HCl and refluxed for 15 h under argon. The deep-blue solution was cooled to 0 °C and saturated with hydrogen chloride. After 2 d at −5 °C dark-blue crystals of **1** (9.8 g, 88%) were filtered and washed with 10 ml cold 10 M HCl. Salts including protonated α-alanine, α-aminobutyric acid and α-valine were prepared in the same way (yields 50–90%).

Quantitative analysis of rhenium and chlorine was performed by gravimetry, as nitron [4,5-dihydro-2,4-diphenyl-5-(phenylimino)-1H-1,2,4-triazolium] perrhenate and AgCl [12].

2.2. X-ray crystallography

The measurements were made using a Siemens SMART CCD diffractometer (graphite monochromated Mo Kα radiation, $\lambda = 0.71073$ Å, empirical absorption corrections using SADABS [13]). The structures were solved by direct methods and refined in the anisotropic approximation using SHELXS-86 and SHELXL-93 [14,15]. In structures **1** and **2** the hydrogen atoms were idealized and only water hydrogen atoms were refined. For **3** all hydrogen atoms were located and refined isotropically. The residual electron density map for **1** suggested disordering for one of two unique $Re_2Cl_8^{2-}$ ions in such a way that both the components retain the same positions of chlorine atoms, but differ in orientation of the Re_2 fragment inside the chlorine 'shell'. The contribution of the minor component was only 3.5%. This characteristic scheme is made possible by a cubic shape of the $Re_2Cl_8^{2-}$ ion and a lack of strong orientation interactions in the structure [1,16]. Structure **2** shows relatively high residual electron density within the $Re_2Cl_8^{2-}$ core (up to 2.4 e Å^{-3}) that reflects possible disorder. We were not successful to resolve it. All these electron density peaks were ghosts of the heavy rhenium atoms at the distances 0.7–1.5 Å. The crystal data are given in Table 1 and the principal bond lengths and angles are listed in Table 2. We note that structure **3** is to date the most precise determination of a Re–Re bond length for $Re_2Cl_8^{2-}$ species.

3. Results and discussion

Structures of $[GlyH]_2Re_2Cl_8 \cdot H_2O$ **1** and $[\beta\text{-AlaH}]_2Re_2Cl_8$ **2** reveal significant differences in the interaction between anionic and cationic counterparts due to the geometry factors. The functionality of the glycinium cation perfectly matches available axial binding sites of $[Re_2Cl_8]^{2-}$ and these moieties interact by means of hydrogen bonding and Re···O coordination (Fig. 1). There are two variants of such supramolecular ensembles: one of them is represented by the ionic pair $[GlyH]_2Re_2Cl_8$, while the second one effectively incorporates two additional water molecules by strong H-bonding with carboxylic OH groups: the O···O distances are 2.65 and 2.72 Å and the O–H···O angles are 160° and 174°. These water molecules somewhat stabilize the supramolecular pattern and the secondary Re···O interaction for the $[GlyH]_2(H_2O)_2Re_2Cl_8$ fragment appears to be slightly shorter (2.964 vs. 3.070 Å for

Table 1
Crystal data for $[GlyH]_2Re_2Cl_8\cdot H_2O$ **1**, $[\beta\text{-}AlaH]_2Re_2Cl_8$ **2**, and $[GlyH]_4(Re_2Cl_8)Cl_2$ **3**

	1	**2**	**3**
Empirical formula	$C_4H_{14}Cl_8N_2O_5Re_2$	$C_6H_{16}Cl_8N_2O_4Re_2$	$C_8H_{24}Cl_{10}N_4O_8Re_2$
Formula weight	826.17	836.21	1031.21
Temperature (K)	223(2)	223(2)	223(2)
Crystal system	triclinic	monoclinic	tetragonal
Space group	$P\bar{1}$	$C2/c$	$P4/n$
a (Å)	8.2132(5)	21.049(1)	11.7356(4)
b (Å)	9.6456(6)	7.7180(5)	11.7356(4)
c (Å)	12.8504(8)	13.2011(9)	9.7051(5)
α (°)	74.161(1)	90	90
β (°)	78.217(1)	116.397(1)	90
γ (°)	70.455(1)	90	90
V (Å^3)	915.76(10)	1921.0(2)	1336.63(9)
Z	2	4	2
$D_{calc.}$ (g cm^{-3})	2.996	2.891	2.562
μ(Mo Kα) (cm^{-1})	143.88	137.16	100.89
$2\theta_{max}$ (°)	52.0	52.0	52.5
No. reflections collected	5233	3716	7480
No. unique reflections (R_{int})	3510 (0.016)	1867 (0.046)	1355 (0.028)
Reflections observed [$I > 2\sigma(I)$]	3051	1389	1208
Data used/Parameters refined	3432/209	1783/101	1326/100
R_1, wR_2 (observed)	0.024, 0.061	0.054, 0.135	0.015, 0.033
R_1, wR_2 (all data)	0.029, 0.063	0.066, 0.141	0.018, 0.035
Largest difference peaks, (e Å^{-3})	1.45/−1.22	2.41/−2.18	0.73/−0.53

Table 2
Selected bond lengths (Å) and bond angles (°)

$[GlyH]_2Re_2Cl_8\cdot H_2O$ **1**, a: $-x, -y, -z$; b: $1-x, 1-y, 1-z$			
Re(1)–Re(1a)	2.2418(5)		
Re(1)–Cl(4)	2.3144(14)		
Re(1)–Cl(3)	2.336(2)	Re(1a)–Re(1)–Cl(1)	101.62(4)
Re(1)–Cl(2)	2.337(2)	Cl(4)–Re(1)–Cl(3)	88.36(6)
Re(1)–Cl(1)	2.343(2)	Cl(4)–Re(1)–Cl(1)	89.03(6)
Re(2)–Re(2b)	2.2306(5)	Re(2b)–Re(2)–Cl(5)	103.22(4)
Re(2)–Cl(8)	2.313(2)	Cl(7)–Re(2)–Cl(6)	87.18(5)
Re(2)–Cl(7)	2.333(1)	Cl(5)–Re(2)–Cl(6)	86.27(6)
Re(2)–Cl(5)	2.333(1)		
Re(2)–Cl(6)	2.341(2)		
$[\beta\text{-}AlaH]_2Re_2Cl_8$ **2**, a: $1-x, 1-y, -z$; b: $1-x, y, 0.5-z$			
Re(1)–Re(1a)	2.2374(8)	Re(1a)–Re(1)–Cl(1)	102.24(8)
Re(1)–Cl(1)	2.323(3)	Cl(1)–Re(1)–Cl(2)	88.19(9)
Re(1)–Cl(2)	2.324(2)	Cl(1)–Re(1)–Cl(4)	86.97(9)
Re(1)–Cl(3)	2.338(3)	Cl(2)–Re(1)–Cl(3)	88.07(10)
Re(1)–Cl(4)	2.331(2)	Re(1a)–Re(1)–Cl(1b)	174.37(5)
Re(1)–Cl(1b)	3.462(3)	Cl(4)–Re(1)–Cl(1b)	82.41(8)
$[GlyH]_4(Re_2Cl_8)Cl_2$ **3**, a: $y, 0.5-x, z$; $0.5-y, x, z$; c: $x, y, 1+z$			
Re(1)–Re(2)	2.2407(3)	Re(2)–Re(1)–Cl(2)	4 × 101.44(2)
Re(1)–Cl(2)	4 × 2.3290(7)	Re(1)–Re(2)–Cl(1)	4 × 102.32(2)
Re(2)–Cl(1)	4 × 2.3337(7)	Cl(2)–Re(1)–Cl(3)	4 × 78.56(2)
Re(1)–Cl(3)	3.425(2)	Cl(2a)–Re(1)–Cl(2)	87.746(8)
Re(2)–Cl(3c)	4.040(2)	Cl(1b)–Re(2)–Cl(1)	87.390(8)

$[GlyH]_2Re_2Cl_8$). According to DTA data the compound loses water only at 170 °C.

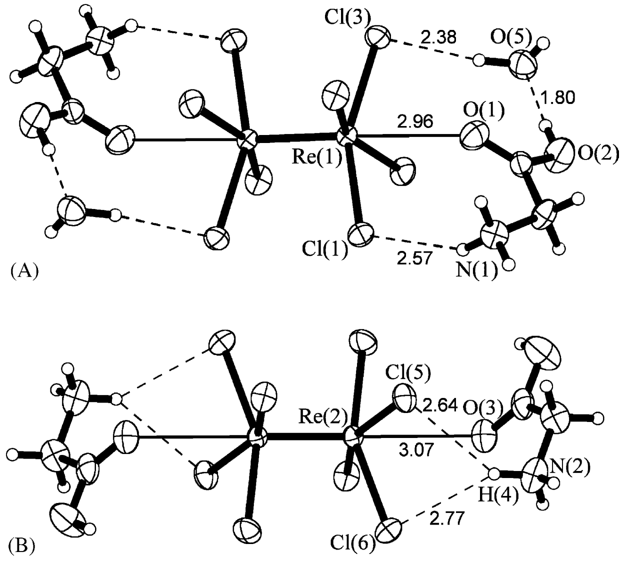

Fig. 1. Two sorts of octachlorodirhenate(III)–glycinium ensembles constituting structure **1**: $[GlyH]_2Re_2Cl_8(H_2O)_2$ (A) and $[GlyH]_2Re_2Cl_8$ (B) (50% probability ellipsoids; minor orientation of the disordered Re(2) atom is not shown). For the bifurcate hydrogen bond, B, the angles at the hydrogen atom are N(2)–H(4)···Cl(5) 142°, N(2)–H(4)···Cl(6) 141°.

Two binding sites (H_3N– and –COOH) in the frame of protonated β-alanine are too far separated and the cation evidently cannot afford the same supramolecular pattern as was found for the glycine analog. In **2** two cations dimerize by means of hydrogen bonding with formation of a characteristic carboxylate motif. Due to

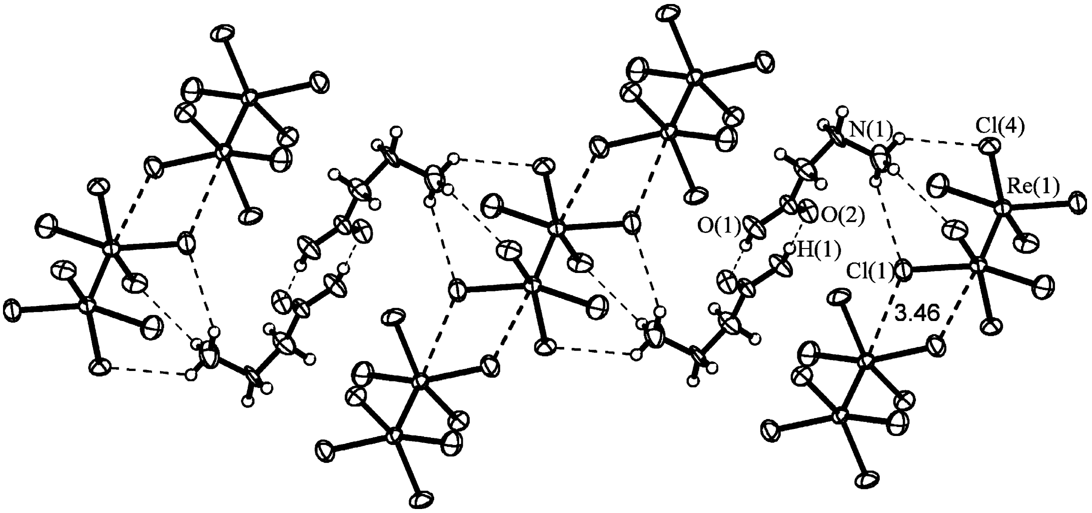

Fig. 2. Fragment of the structure [β-AlaH]$_2$Re$_2$Cl$_8$ **2** (50% probability ellipsoids) showing the infinite [Re$_2$Cl$_8^{2-}$]$_n$ stack and hydrogen bonded pattern based upon dimeric carboxylate motif (O···O 2.64 Å) and N–H···Cl interactions (N···Cl 3.29–3.59 Å).

the lack of coordinating groups in the structure, [Re$_2$Cl$_8$]$^{2-}$ aggregate by weak secondary Re···Cl interactions giving an infinite stack (Fig. 2). Dimeric cations connect these stacks into a three-dimensional structure: each H$_3$N group forms three hydrogen bonds with chlorine atoms of the same chlororhenate anion, one of the N–H···Cl interactions is bifurcated and extends the structure in a third direction. In both structures **1** and **2** the [Re$_2$Cl$_8$]$^{2-}$ ions are centrosymmetric and hence adopt a typical fully eclipsed conformation.

Structure [GlyH]$_4$(Re$_2$Cl$_8$)Cl$_2$ **3** is rather different. It displays no characteristic motifs for Re$_2$Cl$_8^{2-}$ interaction with the organic portion and is essentially dominated by specific functions of additional chloride anions. The resulting pattern has several very salient features. Four glycinium cations associate through effective hydrogen bonding N–H···O (Table 3) forming a 'supramolecular crown' that possesses four NH binding sites and incorporates a chloride anion inside the hole (Fig. 3). All four H-bond donating NH groups are related by a four-fold axis and are situated in the same plane. Thus the chlorine atom has quite an unusual square planar environment deviating from the plane of the corresponding hydrogen atoms by only 0.24 Å. This binding scheme evidently is not favorable and the chlorine atom displays high anisotropy of thermal motion in an orthogonal direction, see Fig. 3 (the second independent chloride anion in the structure has

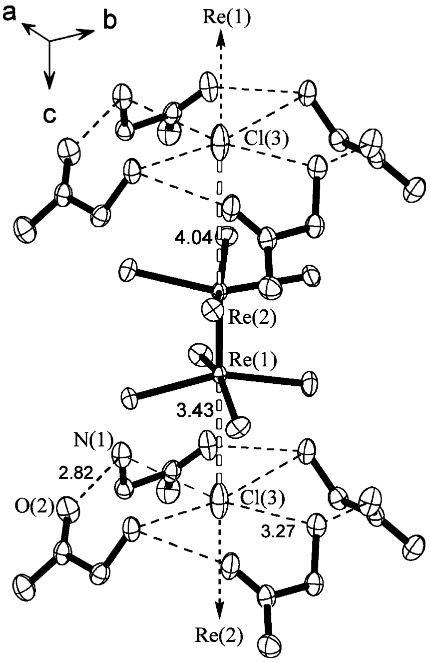

Fig. 3. Fragment of [GlyH]$_4$(Re$_2$Cl$_8$)Cl$_2$ **3** structure (50% probability ellipsoids) that shows how four glycinium cations associate with the formation of a 'supramolecular crown' enclathrating a chloride anion. Note that chlorine atoms adopt a unique square planar H-bonded pattern. Re$_2$Cl$_8^{2-}$ ions are situated between Cl(GlyH)$_4$ fragments.

Table 3
Geometry of hydrogen bonding in the structure [GlyH]$_4$(Re$_2$Cl$_8$)Cl$_2$ **3**

Donor (D)	H atom	Acceptor (A)	D–H (Å)	D···A (Å)	H···A (Å)	∠D–H···A (°)
O(1)	H(1)	Cl(1)	0.76(5)	3.339(3)	2.69(5)	145(4)
N(1)	H(2)	O(2) $(1+y, 0.5-x, z)$	0.85(4)	2.823(4)	2.03(4)	155(4)
N(1)	H(3)	Cl(3) $(1-x, -y, 1-z)$	0.97(5)	3.268(3)	2.32(5)	168(3)
N(1)	H(4)	Cl(4)	0.90(5)	3.210(3)	2.42(5)	147(4)

a typical distorted tetrahedral H-bond environment). The [Cl(GlyH)$_4$] ensembles in the lattice alternate with Re_2Cl_8 ions and together they form an infinite chain with long axial Re···Cl contacts: 3.42 and 4.04 Å. The surrounding of the $Re_2Cl_8^{2-}$ ion is spectacular: it is located between two [Cl(GlyH)$_4$] fragments and also appears inside a larger supramolecular cycle involving eight GlyH cations and four chlorine anions (Fig. 4). The four chlorine atoms at the Re(2) atom are not involved in hydrogen bonding, but have in dense van der Waals contacts with oxygen and nitrogen atoms of neighboring cations (3.46–3.65 Å). At the same time the four chlorine atoms at Re(1) form H-bonds with carboxylic groups, Cl···O are 4 × 3.34 Å (Table 3).

It seems likely that the orientation of the two $ReCl_4$ fragments constituting the $Re_2Cl_8^{2-}$ ion is influenced by cooperative demands of the hydrogen bonds and short van der Waals interactions and conformation of the cluster is adaptable to the requirements of the crystal environment. As a result, the conformation of the $Re_2Cl_8^{2-}$ ion is partially staggered with a Cl–Re–Re–Cl torsion angle at 16.18(3)°. The parameter clearly characterizes the rotational distortion as the ion is situated on a four-fold axis. This observation is worth noting as such a distortion decreases the overlap of d_{xy} orbitals and lowers the energy of the Re–Re δ–bond as a function of cos 2χ. This rotation in structure **3** is the largest of documented structures and presents a proof for a certain conformational flexibility of the dirhenium cluster. The only example for appreciable distortion of the $Re_2Cl_8^{2-}$ ion (by approximately 12°) was found for a disordered structure [17]. In fact the typical values are within a 0–2° range as was observed for numerous salts including alkali metals, substituted ammonium and phosphonium and more complex cations [1,18–22], and the present structural feature is unique. According to theoretical considerations, under the 16.2° rotation observed for **3**, the δ-component of the quadruple bond loses 15% energy that is approximately 1–2% of the entire energy of the Re–Re bond [1,8]. The bond distance in **3**, 2.2407(3) Å, is one of the longest for $Re_2Cl_8^{2-}$ compounds without significant axial interactions (2.211–2.234 Å). In the structure **2** the rhenium atoms maintain comparable weak axial Re···Cl contacts, but the Re–Re bond is slightly shorter (2.2374(8) Å).

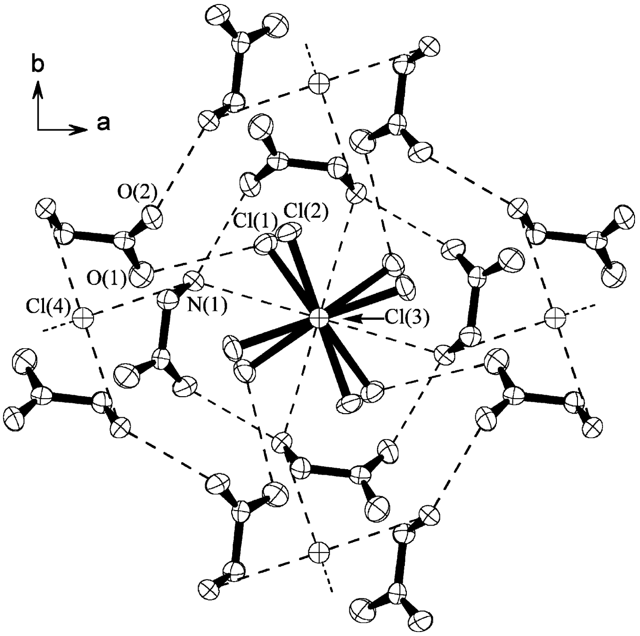

Fig. 4. View of the [GlyH]$_4$(Re_2Cl_8)Cl_2 **3** structure down the direction of Re–Re bonds (rhenium atoms are behind Cl(3) and are hidden) showing the glycinium shell around $Re_2Cl_8^{2-}$ ions and how cooperation of four hydrogen bonds OH···Cl facilitates rotational distortion of chlororhenate.

We suppose that the elongation of the metal–metal bond is a consequence of the weakening of the δ-component under rotational distortion of the ion. It is perhaps counterintuitive that relatively weak OH···Cl hydrogen bonding can influence the strength of even the weakest covalent bond of the δ–δ type. We are currently seeking further examples of molecular and lattice clathrates incorporating $Re_2Cl_8^{2-}$ ions that adopt forced geometry.

4. Supplementary data

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-197503-197505 (compounds **1**–**3**). 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 or www.http://www.ccdc.cam.ac.uk).

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