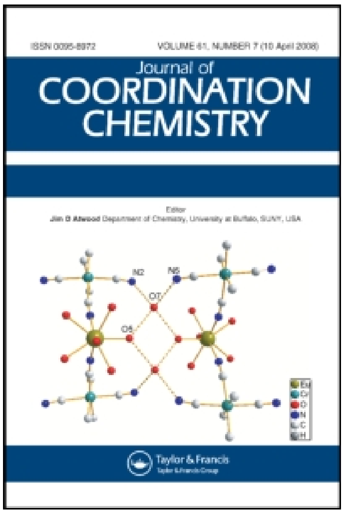

**Journal of Coordination Chemistry**
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

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**To cite this Article** Venkatasubramanian, K. and Suresh, E.(1993) 'THE CRYSTAL AND MOLECULAR STRUCTURE OF DIAQUABIS(L-LYSINO)NICKEL(II) DICHLORIDE MONOHYDRATE', Journal of Coordination Chemistry, 29: 3, 225 — 232

**To link to this Article: DOI:** 10.1080/00958979308045669

**URL:** http://dx.doi.org/10.1080/00958979308045669

J. Coord. Chem. 1993, Vol. 29, pp. 225–232
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# THE CRYSTAL AND MOLECULAR STRUCTURE OF DIAQUABIS(*L*-LYSINO)NICKEL(II) DICHLORIDE MONOHYDRATE

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*(Received 27 July 1992; in final form 23 October 1992)*

The complex, diaquabis(*L*-lysino)nickel(II) dichloride monohydrate $[Ni(C_6N_2O_2H_{15})_2(H_2O)_2]Cl_2.H_2O$ crystallizes in the triclinic space group $P1$, with $Z=1$. Cell dimensions are $a=9.802(1)$, $b=7.118(1)$, $c=8.533(1)$Å, $\alpha=79.47(1)$, $\beta=112.27(1)$, $\gamma=94.48(1)°$. The structure was solved by the heavy atom method and was refined to an $R$ of 0.054 for 3689 observed reflections. The analysis shows that the Ni(II) ion has a distorted octahedral geometry. The α-amino nitrogens and hydroxyl oxygens of lysine coordinate to Ni(II) to form a square plane at distances between 2.025(2)Å and 2.100(2)Å. Water molecules approach the nickel apically at distances of 2.144(4) and 2.153(3)Å, respectively. Chlorines lie outside the octahedron. Both lysine ligands have different conformations. The structure is connected by an elaborate network of hydrogen bonds and electrostatic attractions.

KEY WORDS: Nickel, lysine, conformations, crystal structure.

## INTRODUCTION

Studies of interactions of small biomolecules with metal ions have assumed great importance because they model compounds for understanding the activities of biomacromolecules. Of these, emphasis has been placed upon metal-aminoacid (peptide) and metal-nucleoside (nucleotide) complexes in recent times. Among the former, complexes with lysine are of interest as lysine is an essential aminoacid. A systematic study of metal-lysine interactions has been initiated.[1] The present work describes the synthesis and structural characterization of diaquabis(*L*-lysino)nickel(II) dichloride monohydrate.

## EXPERIMENTAL

$Na_2CO_3$ and $NiCl_2.6H_2O$ was from B.D.H. Limited, while *L*-lysine monohydrochloride was obtained from Sigma, USA.

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### *Synthesis of Ni(L-lys)$_2$Cl$_2$.3H$_2$O*

To a refluxing solution of $NiCl_2.6H_2O$ (1 mmol, 0.238 g) was added a solution of sodium carbonate (1 mmol, 0.106 g). The resulting precipitate was filtered. To the precipitate was added a solution of *L*-lysine monohydrochloride (2 mmol, 0.3653 g). The appearance of a greenish blue colour indicated the formation of the complex. The solution was reduced to one-third of its volume in a water bath and kept for crystallization. Greenish-blue, plate-like crystals were obtained on slow evaporation. The composition of the crystals were ascertained by C, H, N analysis.

### *X-ray structure determination*

A greenish-blue plate of dimensions 0.32 × 0.32 × 0.10 mm was used for intensity data collection. Accurate cell dimensions were obtained by a least-squares fit of 25 arbitrarily chosen reflections within the $2\theta$ range 34°–36°. Preliminary investigations have shown the space group to be $P1$ or $P\bar{1}$. Since the compound is optically active the space group is uniquely determined to be $P1$. Intensity data were collected on an Enraf-Nonius CAD4 diffractometer using graphite-monochromatized Mo-$K_\alpha$ radiation in the $2\theta$ range 2°–60°. Decay and orientation of the crystal throughout the period of data collection were monitored using two sets of three control reflections. Using four moderately strong reflections near $\chi = 90°$, absorption corrections were applied empirically.[2] Relevant crystallographic data are given in Table 1.

**Table 1** Crystallographic data

| | |
|---|---|
| | Diaquabis(*L*-lysine)nickel(II) dichloride monohydrate |
| Formula | $[Ni(C_6N_2O_2H_{15})_2(H_2O)_2]Cl_2.H_2O$ |
| Molecular weight | 524.04 |
| System | Triclinic |
| Space group | $P1$ |
| Cell dimensions | |
| $a$ | 9.802(1)Å |
| $b$ | 7.118(1)Å |
| $c$ | 8.533(1)Å |
| $\alpha$ | 79.47(1)° |
| $\beta$ | 112.27(1)° |
| $\gamma$ | 94.48(1)° |
| $V$ | 541.6(4)Å$^3$ |
| $Z$ | 1 |
| $D_{calc}$ | 1.607 g cm$^{-3}$ |
| $F(000)$ | 258 |
| Equipment | Enraf-Nonius CAD4 Diffractometer |
| Data Collection | $\omega/2\theta$ mode |
| Range | 2°–60° |
| Radiation | Graphite monochromatized Mo-$K_\alpha$ ($\lambda = 0.71073$Å) |
| Zone | $+h, \pm k, \pm l$ |
| Total reflections measured | 4769 |
| observed | ($I > 3\sigma(I)$): 3689 |
| $\mu$ | 12.205 cm$^{-1}$ (for Mo-$K_\alpha$) |
| Transmission factors | Min. 54.36 |
| | Max. 99.67 |
| | Av. 73.09 |
| $R$ | =0.054 |
| $R_w$ | =0.036 |

The structure was solved by the heavy-atom method. An origin-removed, three-dimensional, sharpened Patterson map gave the positions of two chlorines, with respect to nickel, which was placed at the origin. A difference Fourier map, after 3 cycles of isotropic full-matrix refinement of the nickel and chlorine atoms, yielded the positions of all the non-hydrogen atoms. Hydrogen atoms were either generated using stereochemical constraints or located in a difference Fourier map, after complete convergence of the anisotropic refinement of non-hydrogen atoms, and were given isotropic thermal parameters based on the atom to which they connect. A mixed-mode refinement, refining all the non-hydrogen atoms anisotropically, while keeping the hydrogens fixed using a Dunitz-Seiler weighting scheme[3] by full-matrix methods, resulted in convergence at an $R$ value of 0.054 ($R_w = 0.038$) for 3689 observed reflections; the shift to error ratio at this stage was less than 0.02. The scattering factors for nickel, chlorine, oxygen, nitrogen and carbon were from Vol. IV of the *International Tables for Crystallography*,[4] while the scattering factors of hydrogen were from Stewart *et al.*[5] All the computations were carried out using the SDP package of programs[6] available on the PDP-11/73 systems.

## RESULTS AND DISCUSSION

An Ortep[7] diagram of the cationic part of the molecule, along with the numbering scheme is shown in Figure 1. The final positional and equivalent isotropic thermal parameters of non-hydrogen atoms are shown in Table 2, while Table 3 lists the bond lengths and angles in the molecule; these tables also carry information about the estimated standard deviations, which are only slightly underestimated because of the use of full-matrix methods. The Ni(II) ion has a distorted octahedral geometry. The coordinating valencies of the basal square are provided by the hydroxyl oxygens of the carboxyl group (2.025(2) and 2.030(1)Å) and the nitrogens of the α-amino group (2.100(2) and 2.075(3)Å). These values agree with the observation that metal-nitrogen bonds are expected to be around 0.05Å longer than metal-oxygen bonds, based on considerations of atomic radii. The Ni-N and Ni-O bonds in the square base compare

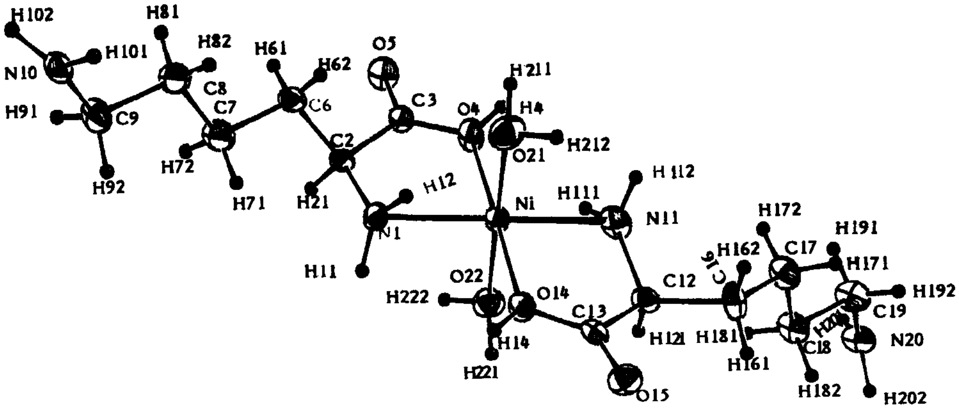

Figure 1 An ORTEP view of the diaquabis(*L*-lysino)nickel(II) cation.

**Table 2** Positional and equivalent isotropic thermal parameters for non-hydrogen atoms and their estimated standard devitions

| Atom | $x/a$ | $y/b$ | $z/c$ | $B$(Åa$^2$)* |
|---|---|---|---|---|
| Ni | 0.000 | 0.000 | 0.000 | 1.428(2) |
| Cl1 | 0.21644(8) | 0.1450(1) | 0.6006(1) | 2.91(1) |
| Cl2 | 0.8106(1) | 0.8465(1) | 0.4071(1) | 3.16(1) |
| O4 | −0.0050(2) | 0.2526(2) | −0.1563(2) | 2.11(2) |
| O5 | 0.0849(3) | 0.5431(2) | −0.1782(2) | 2.45(2) |
| O14 | 0.0125(2) | −0.2521(2) | 0.1606(2) | 1.77(2) |
| O15 | −0.0917(2) | −0.5427(3) | 0.1741(2) | 2.19(2) |
| O21 | 0.1588(3) | −0.1069(3) | −0.0779(3) | 2.62(3) |
| O22 | −0.1592(2) | 0.0987(3) | 0.0837(3) | 2.36(3) |
| O23 | 0.5630(4) | 0.1049(9) | 0.7583(6) | 5.49(8) |
| N1 | 0.1703(2) | 0.1456(3) | 0.1699(2) | 1.76(2) |
| N10 | 0.8763(2) | 0.2774(4) | 0.4832(2) | 1.95(2) |
| N11 | −0.1722(3) | −0.1398(3) | −0.1669(2) | 2.20(3) |
| N20 | −0.8421(2) | −0.2792(4) | −0.4739(2) | 2.15(3) |
| C2 | 0.1901(2) | 0.3389(3) | 0.0902(2) | 1.74(2) |
| C3 | 0.0824(2) | 0.3897(2) | −0.0939(2) | 1.50(2) |
| C6 | 0.3485(2) | 0.3702(4) | 0.0956(2) | 2.05(3) |
| C7 | 0.4597(2) | 0.3602(5) | 0.2762(3) | 2.37(3) |
| C8 | 0.6178(2) | 0.3445(4) | 0.2854(3) | 2.25(3) |
| C9 | 0.7296(2) | 0.3352(4) | 0.4668(3) | 2.14(3) |
| C12 | −0.2227(2) | −0.3001(3) | −0.0617(2) | 1.70(2) |
| C13 | −0.0919(2) | −0.3742(3) | 0.1046(2) | 1.56(2) |
| C16 | −0.3041(2) | −0.4641(3) | −0.1604(3) | 2.11(3) |
| C17 | −0.4356(2) | −0.4058(4) | −0.3250(3) | 2.21(3) |
| C18 | −0.5761(2) | −0.3626(4) | −0.3018(2) | 2.12(3) |
| C19 | −0.7021(2) | −0.3390(4) | −0.4795(3) | 2.38(3) |

*Given in the form of the isotropic equivalent displacement parameter defined as: $(4/3)*[a^2*B(1,1)+b^2*B(2,2)+c^2*B(3,3)+ab(\cos\ \text{gamma})*B(1,2)+ac(\cos\ \text{beta})*B(1,3)+bc(\cos\ \text{alpha})*B(2,3)]$.

well with their counterparts in other nickel-aminoacid complexes.[8-10] The five atoms O4, O14, N1, N11 and Ni, forming square plane, are planar to within 0.026(3)Å. While one of the five-membered rings formed by chelation NiN1C2C3O4 (B) is planar to within 0.029(2)Å, the other is skewed. It can be considered as a shallow half-boat, wherein atoms C13 and C12 are displaced by −0.508(2) and −0.141(2)Å from the mean plane defined by Ni-N11-O14 (C). Water molcules approach the apical positions at distances of 2.144(4) and 2.153(4)Å, respectively, to complete the octahedron. The distortion in the octahedron is reflected in the *trans* angles, 178.06(6) to 178.52(9)°, and the *cis* angles whose range is 80.28(6) to 99.29(7)°. This distortion can be attributed to steric effects.

Lysine occurs in the free protonated form though there is a slight change in C—O distances on complexation. The C=O bonds are elongated by 0.01 to 0.03Å on complexation, while C—OH bonds are shortened by the same amount. An inspection of the angles around the carboxylate group (122.5(1), 121.6(1) and 115.7(1)°, and 124.3(1), 119.4(1) and 116.2(1)°, respectively for lysine ligands 1 and 2) indicate the presence of the protonated group. It should be mentioned here that the smallest CCO angles involve the oxygens of the hydroxyl groups, which are involved in hydrogen bonding. Such a situation has been found in other metal-aminoacid complexes,

**Table 3** Selected bond distances (Ångstroms) and angles (degrees) in the molecule

| Atom 1 | Atom 2 | Distance | Atom 1 | Atom 2 | Distance | Atom 1 | Atom 2 | Distance |
|---|---|---|---|---|---|---|---|---|
| Ni | O4 | 2.025(2) | O14 | C13 | 1.293(2) | C6 | C7 | 1.506(2) |
| Ni | O14 | 2.030(1) | O15 | C13 | 1.237(2) | C7 | C8 | 1.534(3) |
| Ni | O21 | 2.144(4) | N1 | C2 | 1.453(3) | C8 | C9 | 1.513(3) |
| Ni | O22 | 2.153(3) | N10 | C9 | 1.475(4) | C12 | C13 | 1.546(2) |
| Ni | N1 | 2.100(2) | N11 | C12 | 1.493(3) | C12 | C16 | 1.553(3) |
| Ni | N11 | 2.074(3) | N20 | C19 | 1.488(3) | C16 | C17 | 1.518(2) |
| O4 | C3 | 1.298(2) | C2 | C3 | 1.520(2) | C17 | C18 | 1.525(3) |
| O5 | C3 | 1.197(3) | C2 | C6 | 1.535(3) | C18 | C19 | 1.541(3) |

| Atom 1 | Atom 2 | Atom 3 | Angle | Atom 1 | Atom 2 | Atom 3 | Angle | Atom 1 | Atom 2 | Atom 3 | Angle |
|---|---|---|---|---|---|---|---|---|---|---|---|
| O4 | Ni | O14 | 178.08(6) | O22 | Ni | N11 | 87.55(9) | C6 | C7 | C8 | 112.1(3) |
| O4 | Ni | O21 | 90.53(9) | N1 | Ni | N11 | 178.52(9) | C7 | C8 | C9 | 112.0(3) |
| O4 | Ni | O22 | 91.34(8) | Ni | O4 | C3 | 118.8(2) | N10 | C9 | C8 | 112.3(3) |
| O4 | Ni | N1 | 80.38(6) | Ni | O14 | C13 | 115.7(1) | N11 | C12 | C13 | 109.9(1) |
| O4 | Ni | N11 | 99.29(7) | Ni | N1 | C2 | 111.68(9) | N11 | C12 | C16 | 114.3(3) |
| O14 | Ni | O21 | 88.41(8) | Ni | N11 | C12 | 107.8(2) | C13 | C12 | C16 | 112.0(2) |
| O14 | Ni | O22 | 89.74(7) | N1 | C2 | C3 | 113.4(1) | O14 | C13 | O15 | 124.3(1) |
| O14 | Ni | N1 | 98.02(6) | N1 | C2 | C6 | 110.7(2) | O14 | C13 | C12 | 116.2(1) |
| O14 | Ni | N11 | 82.35(6) | C3 | C2 | C6 | 109.4(1) | O15 | C13 | C12 | 119.4(2) |
| O21 | Ni | O22 | 178.15(8) | O4 | C3 | O5 | 122.5(1) | C12 | C16 | C17 | 116.0(2) |
| O21 | Ni | N1 | 89.27(8) | O4 | C3 | C2 | 115.7(1) | C16 | C17 | C18 | 115.6(2) |
| O21 | Ni | N11 | 92.2(1) | O5 | C3 | C2 | 121.6(2) | C17 | C18 | C19 | 107.9(2) |
| O22 | Ni | N1 | 91.02(8) | C2 | C6 | C7 | 112.2(2) | N20 | C19 | C18 | 112.3(2) |

Numbers in parentheses are estimated standard deviations.

some examples being Pd(*D*,*L*-ethionine)$Cl_2$,[11] Pt(*D*,*L*-ethionine)$Cl_2$,[12] Cu(*D*,*L*-lysine)$_2Cl_2.2H_2O$[1] and $PtCl_6$(lysine).[13] The bond lengths and angles found for lysine agree with those found for their counterparts in other cases. It should be mentioned here, that in the structure of [bis(*L*-lysine)Cu(II)dichloride] dihydrate,[14] the zwitterionic form of lysine has a protonated amino group instead of a protonated carboxylate.

The aliphatic chains C6-C7-C8-C9-N10 (D) and C16-C17-C18-C19-N20 (E) deviate considerably from planarity, the maximum deviation of any of the constitutent atoms from the mean plane(s) being 0.101(2) and 0.072(2)Å, respectively. The dihedral angle between D and the plane of the carboxyl group C2-C3-O4-O5 is 97.6(2)°, while that between E and the carboxyl group C12-C13-O14-O15 is 96.9(2)°, respectively, showing that the aliphatic chains are oriented perpendicular to the carboxyl groups. While the amino nitrogen N1 connected to C2-C3-O4-O5 is coplanar with the carboxyl group (deviation of −0.024Å), N11 is displaced by 0.651Å from the mean plane defined by C12-C13-O14-O15. Such deviations have also been noted in the structure of [bis(*L*-lysine)Cu(II)dichloride] dihydrate, wherein the deviations noted for the corresponding nitrogen atoms are −0.876 and 0.264Å, respectively. Atoms C3 and N1 deviate from the plane C6-C7-C8-C9-N10 by −0.176 and −1.911Å, respectively, and C13 and N11 from C16-C17-C18-C19-N20 by 0.984 and 2.438Å. The corresponding deviations in [bis(lysine)Cu(II)dichloride] dihydrate are 0.255Å and −0.756Å for lysine moiety 1 and −0.109Å and 1.154Å for lysine moiety 2. The differences in the values of the deviations can be attributed to the differences in the conformations of lysine in these complexes.

The conformation of lysine is defined by six dihedral angles. Of these, $\psi_1$ and $\psi_2$ define the angles that are governed by the disposition of oxygen with respect to amino nitrogen, whether it is *cis* or *trans*. These values are expected to be in the range of 0 and 180° ideally, though large variations from the ideal values occur, due to the conformational flexibility of lysine because of steric factors. Angles $\psi_1$ and $\psi_2$ for molcules 1 and 2 in our complex are −3.7, −179.4 and 27.1, −152.1°, respectively.

**Table 4** Torsion angles defining the aliphatic chain in *L*-lysine derivatives

| Compound/complex | $\chi_1$ | $\chi_2$ | $\chi_3$ | $\chi_4$ | Reference |
|---|---|---|---|---|---|
| $PtCl_6$*L*-lysine | −179.1 | 168.1 | 172.2 | −73.1 | 13 |
| Cu(*L*-lys)$_2Cl_2.2H_2O$ | −65.7 | 166.7 | 179.8 | 167.3 | 14 |
| | −54.9 | −76.4 | −170.9 | 174.9 | 14 |
| *L*-lysine hydrochloride monohydrate | −56.4 | −176.0 | −171.1 | −179.2 | 15 |
| *L*-lysine succinate (form 1) | −173.7 | 172.5 | 176.5 | −178.9 | 17 |
| *L*-lysine succinate 1 (form 2) | −165.6 | −174.4 | −172.8 | −171.6 | 17 |
| *L*-lysine succinate 2 (form 2) | −165.5 | −173.1 | −175.4 | −170.8 | 17 |
| *L*-lysine succinate 3 (form 2) | 72.2 | −174.4 | −172.8 | −179.3 | 17 |
| *L*-lysine succinate 4 (form 2) | 172.0 | −153.4 | −61.5 | −179.2 | 17 |
| *L*-lysine-*L*-aspartate | −67.7 | 179.6 | 165.5 | 161.2 | 18 |
| *L*-lysine-pantothonate | −79.0 | −175.1 | −175.1 | −177.9 | 19 |
| *L*-lysine-*D*-aspartate | −62.8 | 175.2 | 168.5 | 173.2 | 20 |
| *L*-lysine-*D*-glutomate 1 | 63 | −152.0 | −170.0 | −170.0 | 20 |
| *L*-lysine-*D*-glutamate 2 | 68.0 | −157.0 | −176.0 | −172.0 | 20 |
| *L*-lysine acetate | 63.9 | 178.2 | 167.4 | 175.6 | 21 |
| *L*-lysine sulphate | 53.6 | −179.6 | 176.3 | −73.3 | 22 |

This can be compared with those found in the Cu-*L*-lysine complex (−11.6, 169.6 and 34.1 and 20.5°), the Cu-*D*,*L*-lysine complex (−11.6, 166.6°), *D*,*L*-lysine monohydrochloride (−34.5, 150.7°)[15] and *L*-lysine monohydrochloride (−162, 20°).[16]

The other four dihedral angles, $\chi_1$, $\chi_2$, $\chi_3$ and $\chi_4$, define the conformation of the aliphatic chain. These angles are ideally constricted to the values of 180° (*trans*-conformation, *t*) or −60 and 60° (*gauche*-conformations, $g^+$ anf $g^-$). Various values near these are found in lysine complexes and are shown in Table 4.[17–22] It can be seen that the conformation of lysine moiety 1 (*g-t-t-t*) is similar to that found in other *L*-lysine complexes. Lysine moiety 2 has, however, a different conformation, (*g-g-t-t*), a conformation so far not found in any lysine compound and this may be due to steric factors.

### *Molecular packing and crystal structure*

A perspective view of the molecular packing along the *b* axis is shown in Figure 2. Unlike complexes of lysine with copper chloride, the chlorines are not in the coordination sphere; instead, water occupies the apical positions. The structure is connected by a network of inter- and intramolecular hydrogen bonds and electrostatic attractions between $[Ni(II)(L\text{-}lys)_2.2H_2O]^+$ and $Cl^-$ ions. There are 16 hydrogens available and all of them take part in hydrogen bonding. The hydrogen bonding details within the crystal structure are given in Table 5. Thirteen bonds have an angle varying from 126.9 to 178.8°. Three contacts, two involving bifurcated hydrogen bonds, have values from 101.6 to 111.8°. The H-bond N10-H102-O1 subtends an angle of only 101.6° and may or may not be significant.

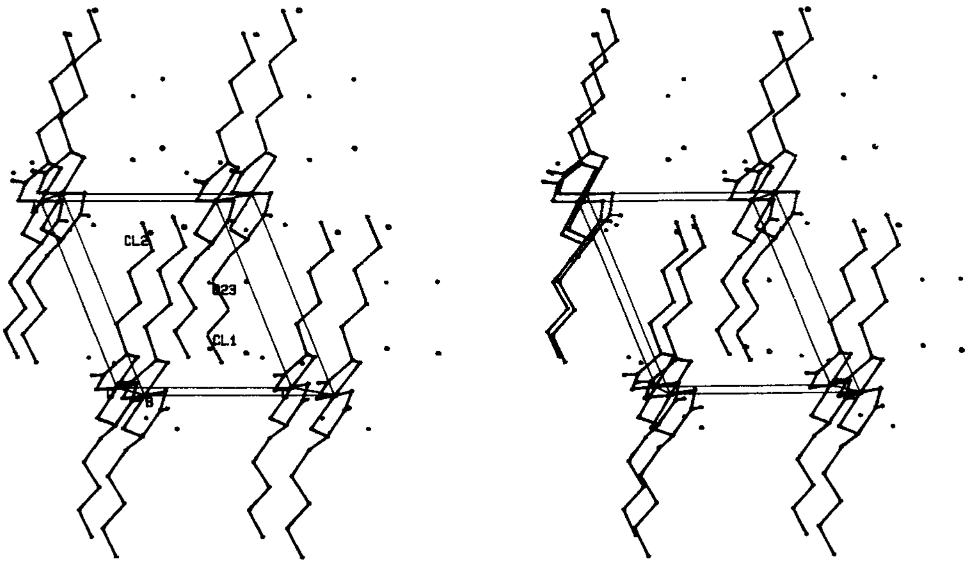

**Figure 2** Molecular packing viewed down the *b* axis.

**Table 5** Hydrogen bond data for diaquabis(*L*-lysine)nickel(II) chloride

| A | H | B | A-B | A-H | H-B | A-H-B | Symmetry of B |
|---|---|---|---|---|---|---|---|
| O4 | H4 | N10 | 2.823 | 0.954 | 1.980 | 146.1 | *x*−1, *y*, *z*−1 |
| O14 | H14 | N20 | 2.702 | 0.944 | 2.144 | 133.2 | 1+x, y, 1+z |
| O21 | H211 | Cl1 | 3.206 | 0.859 | 2.348 | 175.4 | *x*, *y*, *z*−1 |
| O21 | H212 | O5 | 2.737 | 0.841 | 1.897 | 177.7 | *x*, *y*−1, *z* |
| O22 | H221 | Cl2 | 3.107 | 0.919 | 2.244 | 156.1 | *x*−1, *y*−1, *z* |
| O22 | H222 | O15 | 2.760 | 0.945 | 1.817 | 175.4 | *x*, 1+*y*, *z* |
| O23 | H231 | Cl1 | 3.159 | 1.057 | 2.101 | 178.8 | *x*, *y*, *z* |
| O23 | H232 | N11 | 3.037 | 1.071 | 2.257 | 128.0 | 1+*x*, *y*, 1+*z* |
| N1 | H12 | O21 | 2.982 | 0.924 | 2.124 | 1.538 | *x*, *y*, *z* |
| N1 | H11 | Cl1 | 3.258 | 0.946 | 2.674 | 150.5 | *x*, *y*, *z* |
| N10 | H101 | Cl2 | 3.217 | 0.984 | 2.304 | 153.5 | *x*, *y*−1, *z* |
| N10 | H102 | O15 | 2.832 | 0.970 | 2.473 | 101.6 | 1+*x*, 1+*y*, *z* |
| N11 | H111 | O4 | 3.124 | 1.153 | 2.401 | 118.8 | *x*, *y*, *z* |
| N11 | H111 | O22 | 2.925 | 1.153 | 2.419 | 104.3 | *x*, *y*, *z* |
| N11 | H112 | Cl2 | 3.540 | 0.980 | 2.784 | 140.3 | *x*−1, *y*−1, *z*−1 |
| N20 | H201 | Cl1 | 3.163 | 0.863 | 2.567 | 126.9 | *x*−1, *y*, *z*−1 |
| N20 | H202 | O5 | 2.922 | 0.987 | 1.981 | 158.5 | *x*−1, *y*−1, *z* |

## *Supplementary Data*

Full lists of H atom positions, anisotropic thermal parameters and lists of observed and calculated structure factors are available from K.V.

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