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The Crystal and Molecular Structure of Bis(glycinato)bis(imidazole)nickel(II)

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The structure of a mixed-ligand complex, bis(glycinato)bis(imidazole)nickel(II), has been determined from three-dimensional counter data. The crystals are monoclinic with $a = 10.57$ (1), $b = 8.83$ (1), $c = 16.17$ (2) Å, $\beta = 99.0$ (1)°, $Z = 4$ and the space group is $P2_1/c$. The positional and anisotropic thermal parameters have been refined by least-squares methods. The final residual R is 0.049 for 2618 independent reflexions (479 unobservably weak), and the positional parameters of the light atoms have a mean e.s.d. of 0.004 Å. The complex is octahedral and the configuration around the metal ion is *cis*-O(carboxyl), *cis*-N(amino), *cis*-N(imidazole). The mean metal-ligand bond lengths are Ni-O(carboxyl) = 2.09, Ni-N(amino) = 2.10, and Ni-N(imidazole) = 2.07 Å.

Introduction

The structure of bis(glycinato)bis(imidazole)nickel(II), Ni(Gly)₂(ImH)₂, is inherently interesting as an example of a mixed-ligand complex, and more specifically as a model for the type of interaction that may occur between a metal ion and donor groups widely separated along a protein chain.

Among the mixed-ligand complexes whose preparations and spectra have been reported are copper(II) complexes of imidazole and glycylglycine (Driver & Walker, 1968), and cadmium(II) and nickel(II) complexes of peptides, amino acids and imidazole (Rao & Li, 1966). X-ray crystal structure analyses of glycylglycinato bis(imidazole)copper(II) perchlorate, glycylglycinatoaquoimidazolecopper(II) sesquihydrate and diglycylglycinatoaquoimidazolecopper(II) monohydrate have been made (Bell, Freeman, Wood, Driver & Walker, 1969). The only previous structure analysis of a mixed-ligand complex containing amino-acid ligands is that of L-histidinato-L-threoninatoaquocopper(II) hydrate (Freeman, Guss, Healy, Martin, Nockolds & Sarkar, 1969).

Experimental

Crystal data

Ni(Gly)₂(ImH)₂, prepared by the method of Rao & Li (1966), forms large blue-purple crystals. The unit cell

is monoclinic with $a = 10.57$ (1), $b = 8.83$ (1), $c = 16.17$ (2) Å, $\beta = 99.0$ (1)°, $U = 1491$ (3) Å³, $D_m = 1.54$ (2) g.cm⁻³ (by flotation in a mixture of chloroform¹ and 1,2-dibromoethane), $D_x = 1.528$ g.cm⁻³ for C₁₀H₁₆NiN₆O₄, $Z = 4$ with F.W. 342.8, and $\mu(\text{Cu } K\alpha) = 20.8$ cm⁻¹. The space group is $P2_1/c$ (No. 14) from systematic absences ($0k0$ absent for $k = 2n + 1$, $h0l$ absent for $l = 2n + 1$).

The reciprocal cell constants a^* , b^* and c^* were calculated from precise measurements of high-angle reflexions from two crystal specimens, using an equi-inclination diffractometer with Cu $K\alpha$ radiation [$\lambda(\text{Cu } K\alpha_1) = 1.5405$, $\lambda(\text{Cu } K\alpha_2) = 1.5443$ Å]. Adequate accuracy (confirmed by comparing the calculated and experimental directions of other reflexions) was obtained by measuring the Bragg angle θ of one reflection along each reciprocal axis. The angle β^* was fitted to the differences between the φ (crystal rotation) angles of all the $h00$ and $00l$ reflexions. The probable errors in the resulting unit-cell dimensions were derived directly from estimates of the errors in θ and φ .

X-ray data collection

The data were collected using one crystal of dimensions 0.13 × 0.08 × 0.25 mm, mounted successively about the [0,1,0] and [0,0,1] directions. The intensity measurements were made with a fully automatic Supper diffractometer (Freeman, Guss, Nockolds, Page & Webster, 1970), and the control parameters for the scan-range and scan-speed calculations as defined in the cited reference were: $\Delta\lambda = 0.007$ Å, $X = 0.6^\circ$, $\varphi_m = 1.2^\circ$, $P = 0.001$, $\delta\mu = 0.05^\circ$, $\varphi'_{\max} = 0.25$ deg. sec⁻¹, $\varphi'_{\min} = 0.04$ deg. sec⁻¹, and $R_e = 2\%$.

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Table 1. The observed and calculated structure factors for bis(glycinato)bis(imidazole)nickel(II)

The absolute values of F_obs, F_calc, and S (the estimated standard deviations of the observed structure factors) have been multiplied by five.

A large table with multiple columns of numerical data representing structure factors and standard deviations. The table is organized into several sections, with some rows marked with 'L13', 'L14', 'L15', and 'L16'. The data points are arranged in a grid-like format across the page.

Table 1 (cont.)

UNREFINED				REFINED				UNREFINED				REFINED				UNREFINED				REFINED				UNREFINED				REFINED				UNREFINED				REFINED																																															
H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c	H	K	F _o	F _c																																								
L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0	L4	0	0	0
0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08	0	0	1.24	1.08

Table 2. Atomic positional parameters† in fractional coordinates for bis(glycinato)bis(imidazole)nickel(II), with anisotropic temperature factors‡ for the non-hydrogen atoms and isotropic temperature factors for the hydrogen atoms

Atom	10^4z_x	10^4z_y	10^4z_z	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$	Atom	10^3z_x	10^3z_y	10^3z_z	B
N1	2104(.6)	1880(.7)	3778(.4)	47(.5)	53(.8)	19(.2)	3(.6)	7(.3)	6(.4)	H(1)	-8(.4)	261(.5)	394(.3)	4(.1)
O(1)	2609(.2)	1148(.3)	5032(1)	57(2)	98(4)	231(1)	7(2)	9(1)	11(1)	H(2)	-9(.4)	99(.5)	400(.2)	3(.1)
O(2)	1838(.3)	725(.3)	6184(1)	78(3)	117(4)	21(1)	22(5)	10(1)	7(2)	H(3)	48(4)	314(5)	526(2)	4(1)
O(3)	1757(.3)	-397(.3)	3420(2)	85(3)	62(3)	27(1)	-1(3)	11(1)	0(1)	H(4)	-19(4)	159(4)	531(2)	2(1)
O(4)	1337(.3)	-1889(.3)	2301(2)	80(3)	97(4)	39(1)	-11(3)	1(2)	-19(2)	H(5)	46(.5)	225(.5)	350(3)	3(.1)
N(2)	249(.3)	1882(.4)	4156(2)	65(3)	81(4)	24(1)	0(3)	4(2)	11(2)	H(6)	159(4)	290(.5)	226(2)	2(1)
N(3)	1535(.3)	2161(.3)	2507(2)	67(3)	75(.5)	27(1)	1(3)	10(2)	10(2)	H(7)	190(5)	59(.5)	158(3)	4(.1)
N(4)	5896(.3)	902(.4)	3526(2)	51(3)	82(.5)	31(1)	5(3)	12(2)	9(2)	H(8)	236(4)	76(4)	191(2)	2(1)
N(5)	2381(3)	4163(4)	4086(2)	65(3)	74(4)	38(1)	2(3)	6(2)	-10(2)	H(9)	469(4)	-13(5)	415(3)	4(.1)
N(6)	2378(3)	6647(4)	4059(2)	102(4)	62(.5)	45(2)	3(3)	13(2)	6(2)	H(10)	664(5)	34(6)	360(3)	5(.1)
C(1)	470(4)	1949(5)	5070(2)	66(4)	95(5)	24(1)	22(4)	13(2)	4(1)	H(11)	639(5)	246(5)	272(3)	5(.1)
C(2)	1740(4)	1211(4)	5449(2)	61(4)	61(5)	24(2)	7(3)	7(2)	-0(2)	H(12)	409(5)	348(6)	282(3)	6(.1)
C(3)	1542(4)	745(5)	2072(2)	64(4)	103(4)	21(1)	6(4)	8(2)	-2(2)	H(13)	190(4)	745(5)	313(2)	3(.1)
C(4)	1534(3)	-624(5)	2636(2)	41(3)	87(5)	29(1)	-2(3)	8(1)	-8(2)	H(14)	217(4)	745(5)	365(2)	2(.1)
C(5)	4815(5)	688(5)	3801(2)	61(4)	89(5)	29(2)	13(4)	5(2)	5(2)	H(15)	338(5)	693(4)	516(3)	6(.1)
C(6)	5720(5)	2133(6)	2992(3)	73(4)	172(9)	53(2)	-1(5)	31(3)	26(4)	H(16)	337(5)	465(6)	521(3)	6(.1)
C(7)	4541(5)	2829(5)	3723(3)	77(4)	136(7)	55(2)	12(5)	24(3)	39(3)					
C(8)	2017(4)	5382(4)	3644(3)	70(4)	81(4)	36(2)	2(4)	6(2)	2(2)					
C(9)	3005(6)	6238(5)	4813(3)	212(9)	91(7)	43(2)	-29(6)	-10(4)	-12(3)					
C(10)	3917(6)	4711(5)	4829(3)	163(7)	93(7)	36(2)	-12(6)	-10(3)	-1(3)					

† Numbers in parentheses are estimated standard deviations in the units of the least significant digit of the preceding number.

‡ The form of the anisotropic temperature parameter is: $\exp(-h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})$.

For each reflexion with integrated peak count P and background counts B_1 and B_2 , the intensity $I(hkl)$ was given by $I(hkl) = P - (B_1 + B_2)$ and its standard deviation by $\sigma[I(hkl)] = (P + B_1 + B_2)^{1/2}$. Standard Lorentz-polarization corrections were applied to give a set of corrected structure amplitudes $F(hkl)$ and standard deviations $\sigma[F(hkl)]$. Absorption corrections were made by the method of Coppens, Leiserowitz & Rabinovitch (1965) using a $(4 \times 8 \times 4)$ grid parallel to \mathbf{a} , \mathbf{b} and \mathbf{c} respectively.

The sets of data recorded about the two rotation axes were brought to a single scale by the least-squares method of Rae (1965), in which a relative scale factor is found for the reflexions from each reciprocal lattice layer. The values of the scale factors did not differ significantly from one another and did not vary systematically with the equi-inclination angle μ , indicating that both the counter apertures and scan ranges had been correctly chosen (Freeman *et al.*, 1970).

New weights, replacing the inverses of the original

statistical standard deviations, were assigned to the structure amplitudes, using a modification of the method described by Abrahams & Reddy (1965). The total variance $V_T[F(h)]$ of a structure factor $F(h)$ was assumed to be the sum of two terms, $\sigma^2[F(h)]$ and $V_S[F(h)]$. The variance $\sigma^2[F(h)]$ represents the random error of a single measurement as given by counting statistics, while $V_S[F(h)]$ is the contribution of systematic errors. Strictly speaking, $V_S[F(h)]$ is the isotropic systematic error: there are also anisotropic errors which are even harder to estimate. For reflexions which are measured more than once, the differences between the measurements provide an estimate of the total variance:

$$V_T[F(h)] = \sum_{i=1}^R |F(h)_i - \bar{F}(h)|^2 / (R - 1),$$

where $\bar{F}(h)$ is the mean of R independent measurements $F(h)_i$. For an individual observation

$$V_S[F(h)] = V_T[\bar{F}(h)] - \sigma^2[F(h)_i].$$

The majority of reflexions are measured only once, so that $V_T[\bar{F}(h)]$ cannot be estimated for them by this direct method. In the present work it was assumed that estimates of *average* systematic errors, which were valid for the entire data set, could be derived from the multiply observed reflexions alone. The values of $V_T[\bar{F}(h)]$ and $\sigma^2[F(h)]$, each averaged in ranges of $\bar{F}(h)$, were plotted against $\bar{F}(h)$ for the multiply observed reflexions. The difference between the two curves, representing $V_S[F(h)]$ as a function of $\bar{F}(h)$, was approximated by a polynomial,

$$V_S[\bar{F}(h)] = l + m|\bar{F}| + n|\bar{F}|^2 + p|\bar{F}|^3.$$

The total variance of each structure factor was then expressed as the sum of the random error $\sigma^2[F(h)]$ and the *average* systematic error $V_S[\bar{F}(h)]$. The values of l , m , n and p were 9.39×10^{-2} , -6.46×10^{-2} , 2.00×10^{-4} and 5.20×10^{-6} respectively.

Solution and refinement

The structure was solved using unsharpened Patterson and standard Fourier syntheses. In refinement, the

function $\sum w(|F_o| - s|F_c|)^2$ was minimized by full-matrix least squares using the program *ORFLS* (Busing, Martin & Levy, 1962). Four refinement cycles, in which the hydrogen atom parameters were excluded from the structure factor calculations, and in which the thermal motions of the atoms were represented isotropically, resulted in a residual $R = \sum ||F_o| - s|F_c|| / \sum |F_o|$ of 0.09. These parameters were then used to calculate a $(F_o - F_c)$ synthesis from which the coordinates of all the hydrogen atoms were determined. The inclusion of 64 additional parameters for the 16 hydrogen atoms was significant at the 95% confidence level (Hamilton, 1964), causing the weighted residual $R_w = [\sum w(|F_o| - s|F_c|)^2 / \sum wF_o^2]^{1/2}$ to decrease from 0.092 to 0.081. After five more cycles in which anisotropic thermal parameters were assigned to the non-hydrogen atoms, the residuals R and R_w , converged to 0.049 and 0.047 respectively. In the final cycle in which a parameter was varied, the shift was not more than 0.1 standard deviation for a non-hydrogen atom and not more than 1.0 standard deviation for a hydrogen atom. A final $(F_o - F_c)$ synthesis showed no excursions of magnitude greater than $0.2 \text{ e.}\text{\AA}^{-3}$.

The atomic scattering factors used were those for Ni^{2+} , C, O and N (Cromer & Waber, 1965) and H (Stewart, Davidson & Simpson, 1965). The real part of the correction for anomalous dispersion, $-3.1e$ (*International Tables for X-ray Crystallography*, 1962), was added to the Ni^{2+} values over the entire range of $\sin \theta$. The observed and calculated structure factors are listed in Table 1 and the final positional and thermal parameters in Table 2.

Description of the structure

A stereoscopic view of one formula unit is shown in Fig. 1. The two glycine molecules act as bidentate lig-

Table 3. *Dimensions of bis(glycinato)bis(imidazole)nickel(II)*

(a) Bond lengths

Ni—N(1)	2.102 (4) Å	Ni—N(2)	2.104 (4) Å
Ni—O(1)	2.071 (3)	Ni—O(3)	2.108 (3)
Ni—N(3)	2.060 (4)	Ni—N(5)	2.086 (3)
N(1)—C(1)	1.460 (5)	N(2)—C(3)	1.468 (5)
C(1)—C(2)	1.531 (5)	C(3)—C(4)	1.516 (6)
C(2)—O(1)	1.255 (4)	C(4)—O(3)	1.269 (5)
C(2)—O(2)	1.253 (4)	C(4)—O(4)	1.245 (5)
N(3)—C(5)	1.314 (5)	N(5)—C(8)	1.316 (5)
C(5)—N(4)	1.331 (5)	C(8)—N(6)	1.328 (5)
N(4)—C(6)	1.349 (6)	N(6)—C(9)	1.343 (7)
C(6)—C(7)	1.337 (7)	C(9)—C(10)	1.349 (7)
C(7)—N(3)	1.366 (5)	C(10)—N(5)	1.370 (6)

Table 3 (*cont.*)

(b) Bond angles

Ni—N(1)—C(1)	108.3 (3)°	Ni—N(2)—C(3)	107.7 (2)°
N(1)—C(1)—C(2)	110.8 (3)	N(2)—C(3)—C(4)	112.0 (3)
C(1)—C(2)—O(1)	117.6 (3)	C(3)—C(4)—O(3)	117.2 (3)
C(1)—C(2)—O(2)	117.5 (3)	C(3)—C(4)—O(4)	118.1 (3)
O(1)—C(2)—O(2)	124.9 (3)	O(3)—C(4)—O(4)	124.7 (4)
C(2)—O(1)—Ni	115.7 (2)	C(4)—O(3)—Ni	115.0 (2)
Ni—N(3)—C(5)	127.8 (3)	Ni—N(5)—C(8)	130.0 (3)
N(3)—C(5)—N(4)	111.6 (4)	N(5)—C(8)—N(6)	112.1 (4)
C(5)—N(4)—C(6)	107.4 (4)	C(8)—N(6)—C(9)	107.1 (4)
N(4)—C(6)—C(7)	106.2 (4)	N(6)—C(9)—C(10)	106.7 (5)
C(6)—C(7)—N(3)	110.4 (4)	C(9)—C(10)—N(5)	109.5 (5)
C(7)—N(3)—C(5)	104.5 (4)	C(10)—N(5)—C(8)	104.5 (4)
C(7)—N(3)—Ni	127.7 (3)	C(10)—N(5)—Ni	125.6 (3)
N(1)—Ni—O(1)	80.4 (2)	O(1)—Ni—N(5)	93.9 (1)
N(1)—Ni—N(2)	93.0 (2)	N(2)—Ni—O(3)	79.6 (1)
N(1)—Ni—O(3)	87.3 (1)	N(2)—Ni—N(3)	92.8 (2)
N(1)—Ni—N(3)	173.3 (1)	N(2)—Ni—N(5)	97.9 (1)
N(1)—Ni—N(5)	91.7 (1)	O(3)—Ni—N(3)	90.4 (1)
O(1)—Ni—N(2)	166.7 (1)	O(3)—Ni—N(5)	177.3 (1)
O(1)—Ni—O(3)	88.5 (1)	N(3)—Ni—N(5)	90.9 (1)
O(1)—Ni—N(3)	93.3 (2)		

Table 4. Bond lengths and angles involving hydrogen atoms

(a) Bond lengths			
N(1)—H(1)	0.81 (5) Å		
N(1)—H(2)	0.90 (4)		
C(1)—H(3)	1.01 (4)		
C(1)—H(4)	0.92 (4)		
N(2)—H(5)	0.93 (5)		
N(2)—H(6)	0.83 (4)		
C(3)—H(7)	0.91 (5)		
C(3)—H(8)	0.94 (4)		
C(5)—H(9)	0.94 (5)		
N(4)—H(10)	0.92 (5)		
C(6)—H(11)	0.94 (5)		
C(7)—H(12)	0.92 (5)		
C(8)—H(13)	0.88 (4)		
N(6)—H(14)	0.80 (4)		
C(9)—H(15)	0.88 (5)		
C(10)—H(16)	0.89 (5)		
(b) Bond angles			
Ni—N(1)—H(1)	107 (3)°	H(7)—C(3)—N(2)	115 (3)°
Ni—N(1)—H(2)	108 (2)	H(8)—C(3)—C(4)	106 (2)
H(1)—N(1)—C(1)	113 (3)	N(3)—C(5)—H(9)	125 (3)
H(2)—N(1)—C(1)	107 (2)	N(4)—C(5)—H(9)	123 (3)
H(1)—N(1)—H(2)	113 (4)	C(5)—N(4)—H(10)	128 (3)
H(3)—C(1)—H(4)	100 (3)	C(6)—N(4)—H(10)	125 (3)
N(1)—C(1)—H(4)	116 (2)	N(4)—C(6)—H(11)	120 (3)
C(2)—C(1)—H(4)	112 (2)	C(7)—C(6)—H(11)	134 (3)
H(3)—C(1)—N(1)	110 (2)	C(6)—C(7)—H(12)	134 (3)
H(3)—C(1)—C(2)	108 (2)	N(3)—C(7)—H(12)	116 (3)
Ni—N(2)—H(5)	105 (3)	N(5)—C(8)—H(13)	122 (3)
Ni—N(2)—H(6)	117 (3)	N(6)—C(8)—H(13)	126 (3)
H(5)—N(2)—C(3)	107 (3)	C(8)—N(6)—H(14)	120 (3)
H(6)—N(2)—C(3)	110 (3)	C(9)—N(6)—H(14)	133 (3)
H(5)—N(2)—H(6)	109 (4)	N(6)—C(9)—H(15)	120 (3)
H(7)—C(3)—H(8)	104 (4)	C(10)—C(9)—H(15)	133 (3)
N(2)—C(3)—H(8)	109 (2)	C(9)—C(10)—H(16)	133 (4)
C(4)—C(3)—H(7)	110 (3)	N(5)—C(10)—H(16)	118 (4)

ands and the two imidazole molecules as unidentate ligands, thus providing two oxygen and four nitrogen donor atoms per nickel ion. Each pair of chemically equivalent donor atoms occupies two adjacent corners of the coordination octahedron, so that the complex may be named systematically *cis, cis*-bis(glycinato)-*cis*-bis(imidazole)nickel(II). The *cis, cis* configuration of the glycine residues has not been reported previously for an octahedral metal amino acid complex (Freeman, 1967). The two chemically equivalent pairs of ligands are not related by crystallographic symmetry.

The dimensions of the complex molecule are given in Tables 3 and 4.* The bond-lengths and angles of the

* In Tables 3, 4 and 6, the numbers in parentheses are estimated standard deviations in the units of the least significant digit of the preceding number.

glycinate and imidazole residues are not significantly different from those found in the free ligands (Marsh, 1958; Martinez-Carrera, 1966). The torsional angles about the carbon-carbon bonds in the two chelate rings (calculated as the angles between the NCC and CCOO planes) are 23.8 and -20.7° , compared with 26.5° in diaquobis(glycinato)nickel(II) (Freeman & Guss, 1968). Variations of this order of magnitude are common among the conformations of amino-acid chelate rings (Freeman, 1967). The imidazole rings are planar (Table 5, planes 4 and 5). The nickel atom lies 0.057 Å away from one imidazole plane and a negligible distance (0.003 Å) from the other. The first imidazole molecule lies in a plane (plane 4) which roughly bisects the angle O(1)—Ni—O(3) (planes 1 and 2), while the other imidazole molecule (plane 5) is almost coplanar with four nickel-ligand bonds (plane 3). Space filling models show that the rotations of the imidazole groups about the Ni—N bonds are restricted to a few degrees.

The arrangement of the complexes in the unit cell is illustrated in Fig. 2. Each molecule forms ten hydrogen bonds to seven symmetry-related molecules (Table 6 and Fig. 3). With the exception of H(2), all the hydrogen atoms which are attached to nitrogen atoms are involved in hydrogen bonds. There are no unusually short non-bonded contacts.

The average nickel-ligand bond lengths (Table 3) are

Table 5. Deviations of atoms from planes of best fit

(a) Least-squares planes					
Equations of planes $AX + BY + CZ + D = 0$, where X, Y, Z are orthogonal coordinates defined by					
$X = ax - cz \cos \beta$					
$Y = by$					
$Z = cz \sin \beta$					
Plane	Description	A	B	C	D
1	Four donors	-0.0845	-0.9759	-0.2011	2.8082
2	Four donors	-0.2239	0.2739	-0.9353	5.5076
3	Four donors	0.9790	-0.0705	-0.1913	0.0997
4	Imidazole	-0.2249	-0.5805	-0.7826	6.0279
5	Imidazole	0.9286	-0.0074	-0.3711	1.0737

(b) Angles between planes of donor atoms and imidazole molecules

Donor plane	Imidazole plane	
	4	5
1	42.0°	89.8°
2	51.4	82.1
3	91.7	11.3

Table 5 (cont.)

(c) Deviations from the planes

Plane	Deviations of atoms							
1	N(1)	-0.085 Å	N(2)	0.076 Å	N(3)	-0.077 Å	O(1)	0.086 Å
2	N(1)	-0.080	N(3)	-0.080	N(5)	0.078	O(3)	0.081
3	N(2)	-0.045	N(5)	0.039	O(1)	-0.043	O(3)	0.048
4	N(3)	0.005	C(5)	-0.004	N(4)	0.000	C(6)	0.003
5	N(5)	-0.003	C(8)	0.000	N(6)	0.002	C(9)	-0.004
							C(7)	-0.005
							C(10)	0.004

Table 6. *Hydrogen bonds in bis(glycinato)bis(imidazole)nickel(II)*

(a) Hydrogen bond lengths

Bond (N-H...O)	Equivalent bond (O...H-N)	Estimated standard deviations:		
		d_{N-H}	$d_{H...O}$	$d_{N...O}$
N(1)-H(1)····O(4 ⁱ)	O(4)····H(1 ^v)-N(1 ^v)	0.81 Å	2.26 Å	2.901 Å
N(2)-H(6)····O(2 ⁱⁱ)	O(2)····H(6 ^{vi})-N(2 ^{vi})	0.83	2.17	2.951
N(2)-H(5)····O(4 ⁱ)	O(4)····H(5 ^v)-N(2 ^v)	0.93	2.11	3.006
N(4)-H(10)····O(2 ⁱⁱⁱ)	O(2)····H(10 ⁱⁱⁱ)-N(4 ⁱⁱⁱ)	0.92	1.85	2.772
N(6)-H(14)····O(3 ^{iv})	O(3)····H(14 ^{iv})-N(6 ^{iv})	0.80	2.05	2.845

(b) Bond angles at hydrogen-bonded atoms

Ni—N(1)····O(4 ⁱ)	102.4 (2)°	C(9)—N(6)····O(3 ^{iv})	129.0 (3)°
C(1)—N(1)····O(4 ⁱ)	140.7 (3)	C(2)—O(2)····N(2 ^{vi})	118.3 (2)
Ni—N(2)····O(2 ⁱⁱ)	134.2 (2)	C(2)—O(2)····N(4 ⁱⁱⁱ)	107.3 (2)
Ni—N(2)····O(4 ⁱ)	99.1 (2)	N(2 ^{vi})····O(2)····N(4 ⁱⁱⁱ)	116.0 (2)
C(3)—N(2)····O(2 ⁱⁱ)	97.6 (2)	Ni—O(3)····N(6 ^{iv})	139.0 (2)
C(3)—N(2)····O(4 ⁱ)	120.1 (3)	C(4)—O(3)····N(6 ^{iv})	102.4 (2)
O(2 ⁱⁱ)····N(2)····O(4 ⁱ)	100.2 (1)	C(4)—O(4)····N(1 ^v)	136.6 (3)
C(5)—N(4)····O(2 ⁱⁱⁱ)	128.5 (3)	C(4)—O(4)····N(2 ^v)	107.5 (2)
C(6)—N(4)····O(2 ⁱⁱⁱ)	124.1 (3)	N(1 ^v)····O(4)····N(2 ^v)	62.2 (3)
C(8)—N(6)····O(3 ^{iv})	123.8 (3)		

Table 6 (cont.)

(c) Bond angles at hydrogen atoms in hydrogen bonds

N(1)-H(1)····O(4 ⁱ)	137 (4)°
N(2)-H(5)····O(4 ⁱ)	161 (4)
N(2)-H(6)····O(2 ⁱⁱ)	156 (4)
N(4)-H(10)····O(2 ⁱⁱⁱ)	179 (4)
N(6)-H(14)····O(3 ^{iv})	174 (4)

Code for superscripts:

	Symmetry position			Symmetry position	
none	x ,	y ,	z	iv	x , $1+y$, z
i	$-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$	v	$-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$
ii	x ,	$\frac{1}{2}-y$,	$-\frac{1}{2}+z$	vi	x , $\frac{1}{2}-y$, $\frac{1}{2}+z$
iii	$1-x$,	$-y$,	$1-z$	vii	x , $-1+y$, z

Ni-N(amino) = 2.103 (4), Ni-O(carboxyl) = 2.09 (2) and Ni-N(imidazole) = 2.07 (1) Å respectively. These values lie within three e.s.d.'s of the corresponding bond lengths in diaquobis(glycinato)nickel(II) (Freeman & Guss, *loc. cit.*) and hexakis(imidazole)nickel(II) dinitrate (Santoro, Mighell, Zocchi & Reimann, 1969). The individual lengths of the crystallographically independent Ni-O(carboxyl) and Ni-N(imidazole) bonds, however, differ by amounts which would be significant even if the stated e.s.d.'s had been grossly underestimated. We are unable to suggest a plausible explanation for the difference of 0.03 Å between the lengths of Ni-O(1) and Ni-O(3): The only obvious difference between the environments of the two O(carboxyl) atoms

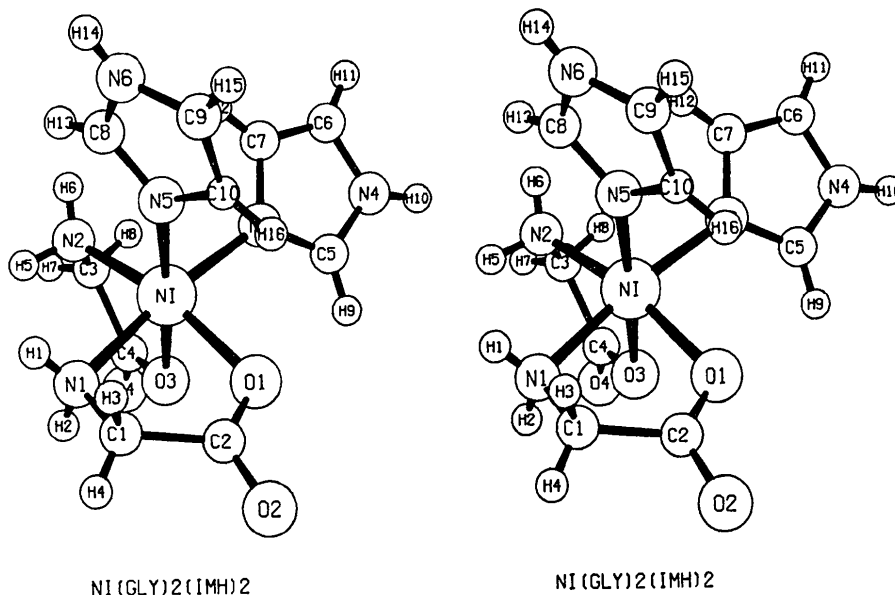


Fig. 1. A stereoscopic view of a molecule of bis(glycinato)bis(imidazole)nickel(II).

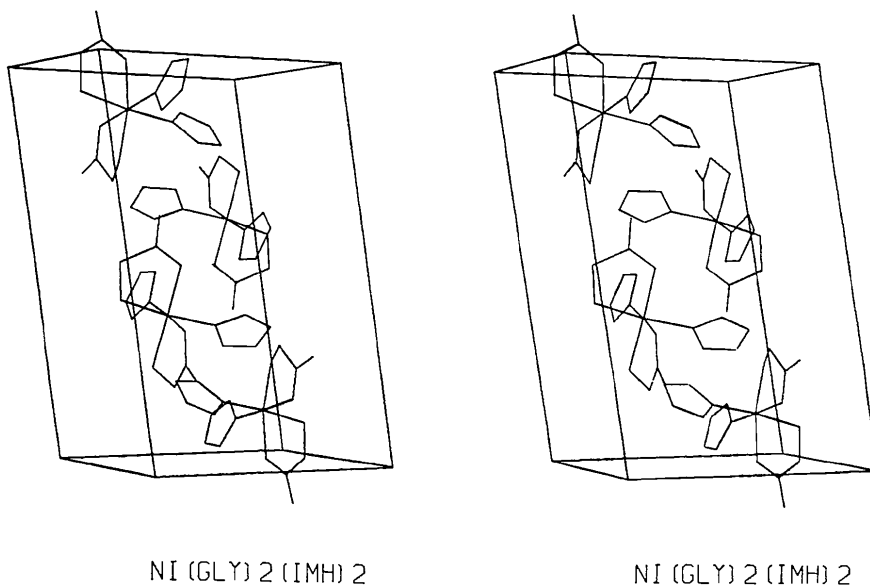


Fig. 2. A stereoscopic view of a unit cell of bis(glycinato)bis(imidazole)nickel(II). The direction of c is upwards, a left to right, and b away from the observer.

is that O(3) is involved in a hydrogen bond. The fact that the bond Ni-N(5) is longer than Ni-N(3) may be due to the difference between the orientations of the two imidazole molecules with respect to the rest of the coordination octahedron (see above). The imidazole molecule which includes N(5) makes two non-bonded intra-complex contacts [$H(16)\cdots O(1)=2.69$, $H(13)\cdots N(2)=2.96$ Å] which are shorter than the shortest intra-complex contacts of the other imidazole molecule [$H(9)\cdots O(1)=3.00$ Å, $H(12)\cdots N(2)=3.11$ Å].

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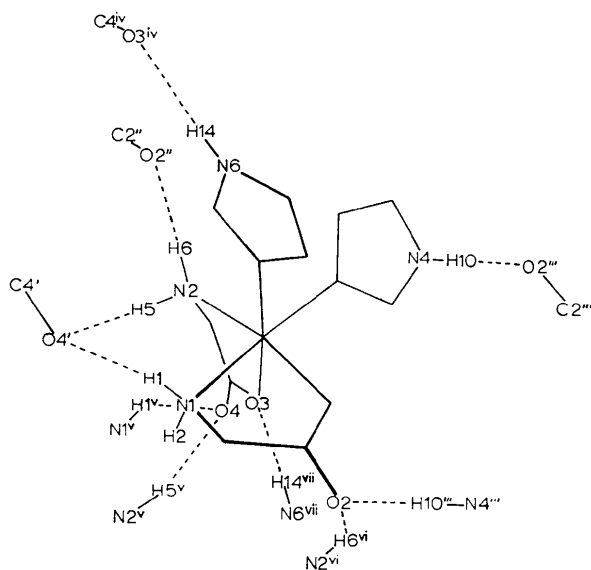


Fig. 3. Schematic diagram of hydrogen bonds formed by one complex molecule.