

*(c) Organisation de la structure*

Le cristal de méthyl-naphtohydroquinone est formé d'empilements des molécules parallèles de direction [010]; la distance entre plans est de 3,55 Å l'inclinaison sur l'axe de la pile est de 38°: le recouvrement moléculaire, s'il est notable, n'intéresse pas tout le noyau naphthalène mais se fait entre cycles benzéniques et noyaux phénols.

C'est un phénomène très voisin de celui mis en évidence dans la naphtohydroquinone; dans le dérivé substitué le recouvrement est modifié de manière à mettre en superposition atome d'oxygène et groupement méthyle d'une part, atome d'oxygène et carbone hybride  $sp^2$  d'autre part (Fig. 5). Cette configuration pourrait expliquer l'intervalle plus important observé entre plans moléculaires (0,07 Å).

Les liaisons hydrogène O-H... relie, autour des axes hélicoïdaux, deux empilements pour créer des feuillets moléculaires parallèles au plan (001). Les molécules font entre elles un angle de 75° (Fig. 6).

Aucune autre liaison forte n'existe pour assurer la cohésion entre feuillets parallèles distants de  $c/2$  (Fig. 7); ceux-ci sont arrangés suivant un close-packing classique pour lequel chaque colonne est entouré de six colonnes identiques.

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## The Crystal and Molecular Structure of Diaquobis-(2,2'-biimidazole)nickel(II) Dinitrate, $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$

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The crystal and molecular structure of diaquobis-(2,2'-biimidazole)nickel(II) dinitrate,  $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$ , was determined by single-crystal X-ray diffraction techniques. This compound crystallizes in the monoclinic system with  $a = 10.3925 \pm 0.0004$  Å,  $b = 9.6284 \pm 0.0004$  Å,  $c = 9.9615 \pm 0.0003$  Å,  $\beta = 96.308 \pm 0.003^\circ$ , space group  $P2_1/n$ ,  $\rho_c = 1.63$  g. cm $^{-3}$ , and  $Z = 2$ . Three-dimensional data (3151 reflections) were used and the structure solved by the heavy atom method. The  $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$  molecule is centrosymmetric with the nickel atom at the center of an octahedron formed by two water oxygen atoms and four nitrogen atoms. The four coordinating nitrogen atoms, two from each biimidazole molecule, form a parallelogram (3.193 × 2.711 Å) which is nearly a rectangle. The structure consists of these complex cations linked to one another by nitrate anions which are hydrogen bonded to the coordinated water molecules, and to the N-H groups in the biimidazole molecules. All hydrogen atoms in this structure were located in a difference map. Final refinement of this model by three-dimensional anisotropic least-squares analysis resulted in an  $R$  value of 5.2%.

**Introduction**

As part of a program in the investigation of the relationship between the detailed crystallographic environment and the spectra of transition element ions the complex, diaquobis-(2,2'-biimidazole)nickel(II) dinitrate, was prepared. From the chemical composition and preliminary unit cell data, it appeared likely that the nickel ion resides in a tetragonal or lower symmetry crystalline field. The presence of water in this complex, moreover, was taken as an indication that hydrogen bonding was present in the structure. The

structure determination of  $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$  was undertaken to provide the structural information required for an analysis of the polarized electronic absorption spectrum and to elaborate the details of the hydrogen bonding network.

**Experimental**

Crystals of  $Ni(C_6H_6N_4)_2(H_2O)_2(NO_3)_2$  were grown by evaporation of an aqueous solution of  $Ni(NO_3)_2$  and 2,2'-biimidazole. The data for the space group determination and the preliminary cell dimensions were ob-

tained from zero and upper level precession photographs. The observed systematic extinctions led to the assignment of the crystal to the monoclinic space group  $P2_1/n$ .

Prior to the determination of precise unit-cell dimensions, a single crystal ( $\sim 0.4 \times 0.3 \times 0.2$  mm) was mounted on a diffractometer and a rapid survey made of all reflections in the  $2\theta$  region greater than  $120^\circ$  with Cu  $K\alpha$  ( $\lambda = 1.54051$ ) radiation. Seventeen strong reflections were selected for  $2\theta$  measurement. The cell parameters were then refined by least-squares analysis to obtain optimum agreement between observed and calculated  $2\theta$  angles. Assuming two molecules of  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{H}_2\text{O})_2(\text{NO}_3)_2$  per unit cell and using the refined cell parameters, the X-ray density was calculated to be  $1.630 \text{ g.cm}^{-3}$ . The density obtained by flotation was  $1.68 \text{ g.cm}^{-3}$ . A summary of the crystal data is given in Table 1.

Table 1. *Crystal data for*  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{HOH})_2(\text{NO}_3)_2$

$a = 10.3925 \pm 0.0004 \text{ \AA}$	Space group $P2_1/n$
$b = 9.6284 \pm 0.0004$	$Z = 2$
$c = 9.9615 \pm 0.0003$	$\rho_o = 1.68 \text{ g cm}^{-3}$
$\beta = 96.308 \pm 0.003^\circ$	$\rho_c = 1.630$
Systematic extinctions	
$h0l$	$h+l=2n+1$
$0k0$	$k=2n+1$

The errors in the cell parameters are the standard deviations obtained from a least-squares refinement of these parameters using seventeen experimental  $2\theta$  values. The  $2\theta$  measurements were taken at  $23 \pm 2^\circ\text{C}$ .

For the structure determination, the stationary-crystal stationary-counter method was employed. The crystal was oriented with the  $b$  axis coincident with the  $\varphi$  axis. The intensities of all 3151 unique reflections (2165 observed, 986 unobserved) within  $\sin \theta/\lambda = 0.724$  were measured using molybdenum radiation and a  $\beta$  filter of  $0.025$  mm thick niobium. Approximately 400 reflections equivalent to those in the above set were measured and it was found that equivalent pairs agreed within counter error. The data thus obtained were corrected for Lorentz and polarization factors. As the linear absorption coefficient for crystalline  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{H}_2\text{O})_2(\text{NO}_3)_2$  is only  $10.5 \text{ cm}^{-1}$ , no absorption correction was applied. Additional details on the method of data collection are given in a previous paper (Reimann, Mighell, & Mauer, 1967).

#### Determination of structure

The chemical composition, density, unit-cell volume, and space group require that the nickel ion lie on a center of symmetry. This conclusion is confirmed by the fact that the observed reflections with an even  $(h+k+l)$  were greater, on the average, than those with odd  $(h+k+l)$ .

To determine the structure, an electron density map was calculated using reflections for which the

$F_c$  based upon the nickel atom position satisfied the condition:

$$\frac{1}{2}|F_o| < F_c < 3/2|F_o|.$$

As nickel contributes to reflections only if  $(h+k+l)$  is even, two images of the structure related by a mirror normal to  $\mathbf{b}$  appeared in the electron density map. An internally consistent image was identified in this map based upon the assumptions of octahedral coordination about the nickel atom and of the planar configuration of the biimidazole molecule. Another Fourier synthesis was then calculated with phases determined from the chosen image. The mirror image structure present in the initial electron density map was thus obliterated and the resulting map contained a chemically reasonable structure.

#### Refinement

Full-matrix isotropic least-squares refinement on all atoms except hydrogen was carried out on the tentative model. In this refinement scattering factors for neutral nickel, carbon, nitrogen, hydrogen and singly negative oxygen were used and these were obtained from *International Tables for X-ray Crystallography* (1962). The quantity minimized in this refinement was  $\sum w(|F_o| - |F_c|)^2$  with the weighting scheme  $w=1$  for  $|F_o| < 35$  and  $w=35/|F_o|$  for  $|F_o| > 35$ . Reflections for which the net number of counts observed did not exceed zero by at least twice the standard deviation were designated as unobserved, and assigned a value for the net number of counts equal to one standard deviation. These 986 unobserved reflections were given zero weight in the refinement unless the value of  $F_c$  exceeded that of  $F_o$ . Those reflections for which this occurred were given unit weight. As a result of this refinement the agreement factor,  $R$ , where  $R = \sum (|F_o| - |F_c|) / \sum F_o$  based upon 2165 observed reflections refined to a value of 0.099.

The model was next refined by full-matrix anisotropic least-squares analysis and the  $R$  value decreased to 0.064. A difference  $(F_o - F_c)$  synthesis was then calculated and this map contained well defined peaks outside the periphery of the biimidazole rings and near the coordinated oxygen atoms. Bond angle analysis indicated that these peaks are consistent with hydrogen atom positions. Bond length calculations showed that the average C-H, N-H and O-H distances were  $0.96 \text{ \AA}$ ,  $0.84 \text{ \AA}$  and  $0.75 \text{ \AA}$  respectively. Although these distances are shorter than the accepted inter-nuclear separations, they are consistent with X-ray bond distances involving hydrogen atoms; e.g. Brown (1967), Marsh (1959). Including the hydrogen atoms in the final structure factor calculation reduced the  $R$  value from 0.064 to 0.052. For this calculation each hydrogen atom was assigned an isotropic temperature factor of 4.5. The final atomic coordinates, anisotropic thermal parameter and structure factors are presented in Tables 2, 3 and 4 respectively.

Table 2. Atomic coordinates\*

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>
Ni	0.5	0.5	0.5
O(1)	0.31801 (23)	0.43983 (24)	0.55393 (23)
O(2)	0.31232 (36)	0.18766 (33)	0.72223 (27)
O(3)	0.35130 (27)	0.36334 (28)	0.84930 (27)
O(4)	0.35373 (32)	0.15832 (32)	0.93547 (27)
N(1)	0.40924 (26)	0.56615 (28)	0.31128 (26)
N(2)	0.35612 (28)	0.50783 (35)	0.09800 (25)
N(3)	0.46857 (30)	0.21324 (31)	0.19424 (29)
N(4)	0.50687 (27)	0.31509 (27)	0.39273 (26)
N(5)	0.33891 (30)	0.23627 (33)	0.83471 (30)
C(1)	0.35548 (35)	0.67919 (38)	0.24190 (36)
C(2)	0.32214 (37)	0.64400 (44)	0.11050 (38)
C(3)	0.40814 (30)	0.46529 (34)	0.22124 (30)
C(4)	0.45982 (31)	0.33166 (32)	0.26413 (31)
C(5)	0.52222 (40)	0.11414 (39)	0.28181 (42)
C(6)	0.54461 (37)	0.17799 (36)	0.40416 (38)
H(1)	0.3374 (44)	0.7730 (50)	0.2826 (45)
H(2)	0.2883 (45)	0.6960 (47)	0.0378 (45)
H(3)	0.3413 (45)	0.4576 (47)	0.0334 (75)
H(4)	0.4433 (45)	0.2022 (48)	0.1134 (45)
H(5)	0.5343 (44)	0.0212 (48)	0.2632 (46)
H(6)	0.5836 (44)	0.1428 (49)	0.4899 (44)
H(7)	0.3278 (45)	0.4003 (52)	0.6127 (46)
H(8)	0.2862 (46)	0.5131 (51)	0.5719 (46)

\* The numbers in parentheses are standard deviations in the last significant figures.

### Discussion

The only crystallographic constraint on the  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{H}_2\text{O})_2(\text{NO}_3)_2$  molecule is that it must have a center of symmetry. The asymmetric unit, therefore, comprises half a nickel ion, one biimidazole molecule, one water molecule and one nitrate anion. The crystal structure consists of discrete, centrosymmetric complex cations,  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{H}_2\text{O})_2^{2+}$ , joined by hydrogen bonding *via* their O-H and N-H groups to the nitrate anions. This cation with the unique coordination distances and angles is shown in Fig. 1. The packing arrangement and the distances between atoms connected by hydrogen bonds are shown in Fig. 2.

### The basal plane

In order to describe the details of the structure of  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{H}_2\text{O})_2(\text{NO}_3)_2$ , we will utilize the rigorous plane defined by the coordinating nitrogen atoms [N(1)N(1')N(4)N(4')] as a reference plane. This plane, which passes through the nickel ion, hereafter will be referred to as the basal plane. The structure of  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{H}_2\text{O})_2(\text{NO}_3)_2$  may now be considered in terms of the orientation of the various groups (water, nitrate, biimidazole) with respect to the basal plane (Table 5).

### The coordinated water molecules

The water molecules lie above and below the basal plane. The nickel oxygen direction is virtually normal to the basal plane making an angle of  $88.6^\circ$  with it. The plane of the water molecule was found to make an

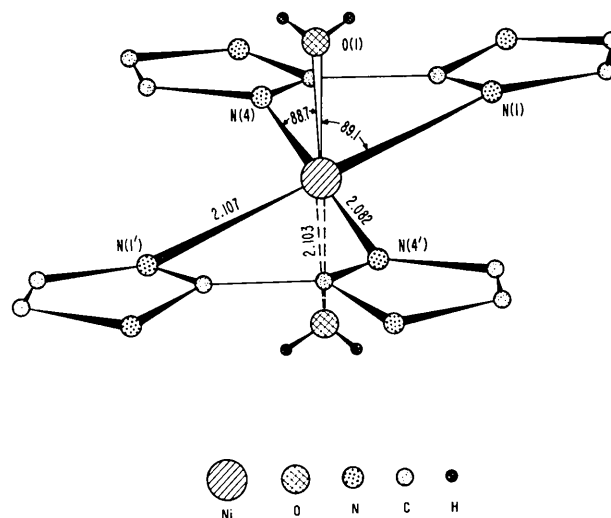


Fig. 1. The  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{HOH})_2^{2+}$  cation. Unique bond distances and angles are indicated.

Table 3. Anisotropic thermal parameters\*†

	$10^5\beta_{11}$	$10^5\beta_{22}$	$10^5\beta_{33}$	$10^5\beta_{12}$	$10^5\beta_{13}$	$10^5\beta_{23}$
Ni	705 (5)	628 (5)	590 (5)	92 (5)	42 (4)	-15 (5)
O(1)	890 (23)	850 (26)	944 (26)	85 (20)	129 (20)	101 (21)
O(2)	2435 (51)	1516 (44)	880 (28)	246 (38)	-275 (30)	-268 (29)
O(3)	1254 (32)	947 (30)	1236 (33)	-154 (25)	262 (26)	-68 (25)
O(4)	1963 (43)	1536 (40)	1030 (30)	-752 (33)	-379 (28)	394 (28)
N(1)	782 (25)	819 (30)	771 (27)	134 (22)	67 (20)	38 (23)
N(2)	935 (27)	1256 (37)	699 (24)	-13 (30)	-61 (20)	59 (29)
N(3)	1033 (32)	951 (33)	919 (31)	-28 (26)	119 (25)	-288 (26)
N(4)	859 (27)	739 (28)	787 (27)	38 (22)	87 (21)	-58 (22)
N(5)	1066 (32)	1046 (36)	841 (31)	-157 (27)	38 (25)	24 (27)
C(1)	903 (35)	1009 (40)	1001 (38)	175 (30)	88 (29)	135 (32)
C(2)	949 (38)	1363 (50)	966 (39)	74 (35)	-50 (30)	307 (36)
C(3)	672 (27)	997 (37)	670 (28)	-65 (24)	73 (22)	20 (25)
C(4)	740 (30)	764 (33)	763 (30)	-18 (24)	110 (24)	-131 (25)
C(5)	1174 (43)	868 (39)	1384 (49)	156 (33)	162 (36)	-227 (36)
C(6)	978 (37)	815 (36)	1167 (41)	161 (30)	60 (31)	-59 (31)

\* The general anisotropic temperature factor has the form  $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$ .

† The numbers in parentheses are standard deviations in the last significant figures.

Table 4. Observed and calculated structure factors

The columns are  $I$ ,  $10F_o$  and  $10F_c$ , respectively. Unobserved reflections are indicated with a \*.

Table with multiple columns of numerical data representing structure factors. Includes headers for columns and various sub-headers for different reflections (e.g., 001, 100, 110, etc.).

STRUCTURE OF DIAQUOBIS(2,2'-BIIMIDAZOLE)NICKEL(II) DINITRATE

Table 4 (cont.)

Table with multiple columns of numerical data, organized into sections labeled with coordinates like 7,1-L, 8,1-L, 9,1-L, etc. Each section contains several rows of values, some with negative signs, representing structural parameters.

angle of  $29^\circ$  with the basal plane. This angle, assuming tetrahedral coordination about the water molecule, would be  $35.26^\circ$ . Thus, with respect to the oxygen atom, the nickel atom and the two hydrogen atoms occupy, very nearly, three apices of a tetrahedron.

The hydrogen atoms of the coordinated water molecules are involved in hydrogen bonding with two ni-

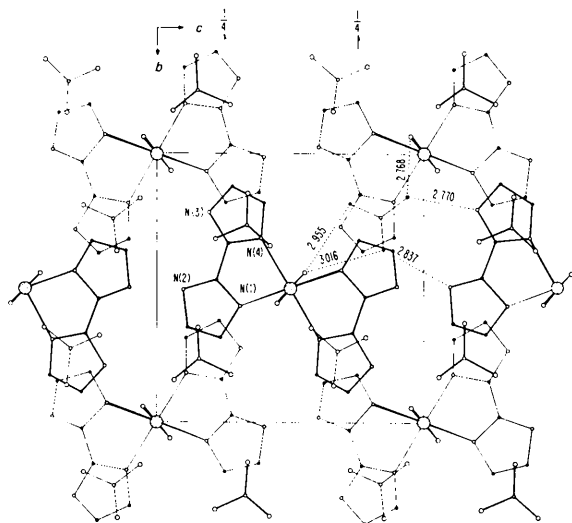


Fig. 2. Projection of the contents of the unit cell for  $\text{Ni}(\text{C}_6\text{H}_6\text{N}_4)_2(\text{HOH})_2(\text{NO}_3)_2$  down the  $a$  axis. The hydrogen bonding network in relation to the overall structure is shown by dotted lines.

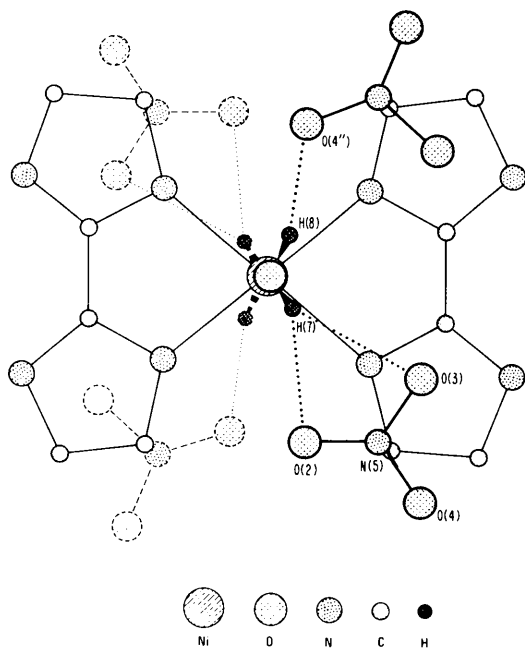


Fig. 3. Orientation of the nitrate groups with respect to a single cation. Hydrogen bonding between the coordinated water molecules and the nitrate groups is indicated by dotted lines.

Table 5. Angles defining the orientation of the least squares planes of the indicated groups with respect to the basal plane.\*

Group	Angle
Nitrate ( $\text{NO}_3^-$ )	$22.4^\circ$
Nitrate ( $\text{NO}_3^-$ )'	$14.9$
Coordinated water	$28.6$
Biimidazole†	
Imidazole ring 1 [C(1), C(2), C(3), N(1), N(2)]	$3.6$
Imidazole ring 2 [C(4), C(5), C(6), N(3), N(4)]	$3.6$

\* Plane defined by nitrogen atoms coordinated to the nickel ion.

† Ring 1 and ring 2 in biimidazole make an angle of  $1.18^\circ$  with each other.

trate groups. One hydrogen atom, H(8) is bonded to an oxygen atom, O(4), in one nitrate group while the other hydrogen atom, H(7) is involved in a bifurcated hydrogen bond with O(2) and O(3) in a second nitrate group. This situation is depicted in detail in Figs. 3 and 4. These Figures include the hydrogen atom positions which were obtained from the above mentioned difference synthesis.

#### The biimidazole molecule

The biimidazole molecule consists of two imidazole rings: ring 1 [N(1), N(2), C(1), C(2), C(3)] and ring 2 [N(3), N(4), C(4), C(5), C(6)] bonded through C(3)–C(4). The atoms in each ring were fitted to a plane by least-squares analysis. Both rings were found to be planar (Table 6) to well within one standard deviation of the atoms normal to the calculated planes. Ring 1 and ring 2 make angles of  $3.57^\circ$  and  $3.58^\circ$ , respectively, with the basal plane. These two rings are nearly coplanar making an angle of  $1.18^\circ$  with each other. The hydrogen atoms were found to lie in the imidazole planes to within two standard deviations of the hydrogen atom positions (Table 6).

Table 6. Distances from the ring atoms to the least-squares planes of the imidazole rings in biimidazole\*. These planes were calculated through carbon and nitrogen atom positions, but not hydrogen positions.

Imidazole ring 1		Imidazole ring 2	
	Distance (Å)		Distance (Å)
N(1)	0.0013	C(4)	0.0058
N(2)	-0.0004	C(5)	-0.0017
C(1)	-0.0016	C(6)	0.0051
C(2)	0.0013	N(3)	-0.0067
C(3)	-0.0006	N(4)	-0.0026
H(1)	-0.049	H(4)	-0.017
H(2)	0.040	H(5)	-0.055
H(3)	-0.075	H(6)	0.017

\* The equation of the plane in direct space is given by  $PX + QY + RZ = S$ . For ring 1,  $P=9.7173$ ,  $Q=2.7031$ ,  $R=3.1677$ , and  $S=4.5224$ . For ring 2,  $P=9.7803$ ,  $Q=2.5146$ ,  $R=3.1569$ , and  $S=4.5032$ .

The crystallographically independent imidazole rings which comprise the biimidazole molecules were found to be nearly superimposable. Bond distances (average

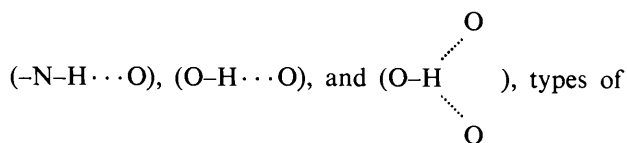
$\sigma=0.0045 \text{ \AA}$ ) and angles are given in Fig.5. The position of the nickel atom is included in this Figure to emphasize the fact that the exterior angles,  $N(3)-C(4)-C(3)(130.4^\circ)$ ,  $C(4)-C(3)-N(2)(129.8^\circ)$ ,  $N(4)-C(4)-C(3)(118.6^\circ)$ , and  $C(4)-C(3)-N(1)(118.7^\circ)$  are undoubtedly modified by coordination. The importance of resonance in the bonding description of the biimidazole molecule can be inferred from the near coplanarity of the two imidazole rings, from the short C-C single bond which connects these rings and from the relatively narrow range of observed bond distances. The shortest observed distance in each ring is, however, between atoms involved in the classical (C-N) double bond.

#### The nitrate anion and hydrogen bonding

Although the bond angles in the nitrate group are essentially equal, the N-O bond distances vary significantly, from 1.218 to 1.249 Å. Least-squares plane analysis showed the nitrate group to be planar to well within experimental error.

The orientation of the nitrate groups with respect to a single cation is shown in Fig. 3 and Table 5. In Fig. 3 the nitrate groups above the basal plane are related through the center of symmetry to those below. This Figure shows that four nitrate groups are involved in hydrogen bonding with the two coordinated water molecules within a single cation and that the planes of these nitrate groups are roughly parallel to the basal plane. The two nitrate groups labeled by N(5) and O(4'') make angles of  $22.4^\circ$  and  $14.9^\circ$ , respectively, with the basal plane.

In addition to being hydrogen bonded to water, each nitrate group is also hydrogen bonded to the two N-H groups in a single coordinated biimidazole molecule (Figs. 2, 4). Each nitrate anion, then, is associated with three different cations *via* three distinct



hydrogen bond. Altogether five hydrogen bonds are formed by each nitrate anion.

This involvement in a unique hydrogen bonding situation by each of the oxygen atoms of the nitrate group, moreover, is reflected in the marked variation in the N-O distances in the nitrate group. The oxygen atom involved in the longest of the N-O (1.249 Å) distances participates in two single hydrogen bonds while the oxygen atom involved in the shortest N-O (1.218 Å) distance participates in only half of a bifurcated hydrogen bond.

We would like to acknowledge that most of the computer calculations on this structure were performed with the X-ray 63 system of programs developed at the University of Maryland Computer Science

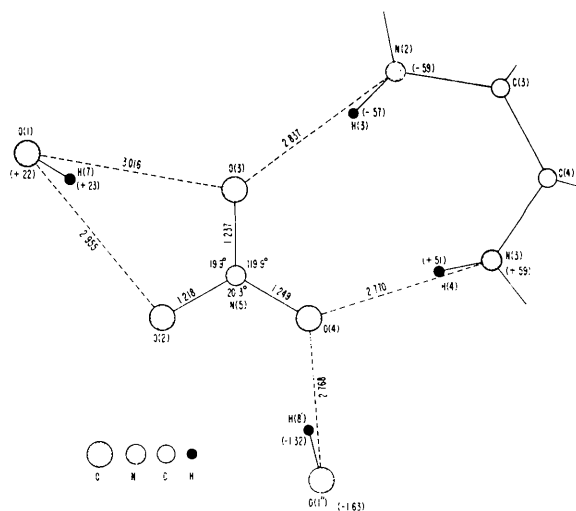


Fig. 4. The nitrate anion and hydrogen bonding. The numbers in parentheses give the distance in ångströms which a given atom lies above or below the plane of the nitrate group.

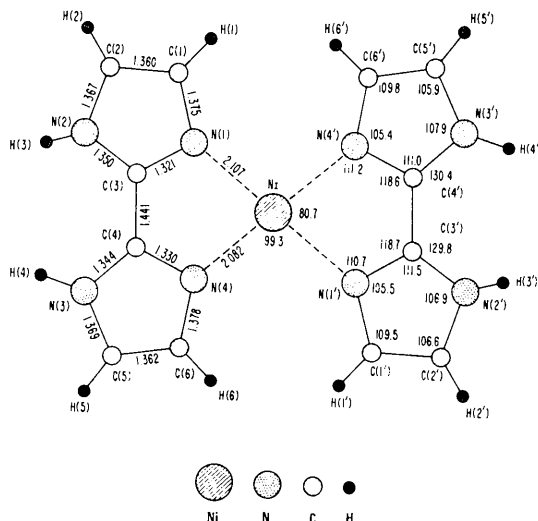


Fig. 5. The biimidazole molecule. Unique bond distances and angles are indicated. The average standard deviations in bond lengths are as follows: Ni-O,  $\pm 0.0025 \text{ \AA}$ ; Ni-N,  $\pm 0.0025 \text{ \AA}$ ; N-C,  $\pm 0.004 \text{ \AA}$ ; and C-C,  $\pm 0.005 \text{ \AA}$ . The average standard deviation in the bond angles is  $0.35^\circ$ .

Center (1964) and the University of Washington. Programs used in calculating bond distances and angles and in drawing the Figures were written at the National Bureau of Standards.

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