2999-74-8; $CH_3MgBr$, 75-16-1; $Zn(CH_3)_2$, 544-97-8; $Cd(CH_3)_2$, 506-82-1; $B_5H_9$, 19624-22-7; boron-11, 14798-13-1.

**Supplementary Material Available**: listing of structure factor amplitudes (4 pages). Ordering information is given on any current masthead page.

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# Crystal and Molecular Structure of a Nickel(II) Iodide Complex of a Macrocyclic Tetradentate Ligand[1]

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The crystal structure of $Ni(C_{20}H_{22}N_2O_4)I_2$ has been determined by single-crystal x-ray diffraction using automatic diffractometer methods to collect three-dimensional data. The structure was solved using the Patterson heavy-atom technique and Fourier difference electron density maps and was refined by full-matrix least-squares methods to a conventional residual $R_F = 0.038$ and a weighted residual $R_w = 0.046$ based on 2686 reflections with $I_{obsd} \geq 3\sigma(I)$. The compound crystallizes in the orthorhombic space group $Pna2_1$ with cell dimensions of $a = 20.931$ (8) Å, $b = 14.221$ (3) Å, $c = 7.478$ (1) Å, and $V = 2226$ (1) Å$^3$. The density calculated assuming $Z = 4$ is 1.99 g cm$^{-3}$ as compared with a measured density of 2.00 g cm$^{-3}$. The structure consists of discrete molecules; the two iodine atoms lie in cis-coordination sites, the two oxygen atoms lie trans to one another, and the two nitrogens lie cis to one another in the inner coordination sphere. The Ni–I bond distances of 2.711 (1) and 2.757(1) Å are consistent with the calculated sum of the covalent radii for octahedral Ni(II) complexes.

## Introduction

Kluiber and Sasso[2a] have reported that bis(*N*-2-bromoethylsalicylaldiminato)nickel(II) reacts with sodium iodide in acetone to form a paramagnetic complex of empirical formula $Ni(C_{18}H_{18}N_2O_2)I_2$. The structure of this red material was reported by Johnston and Horrocks[2b] and shows the complex to be octahedral with the two iodides in trans configuration, the two oxygens in trans configuration, and the two nitrogens also in trans configuration. This tetradentate macrocyclic imine–ether complex is essentially planar with the iodine atoms above and below the plane.

Kluiber and Sasso[3] have also made nickel complexes with various substituents on the ligand. When a methoxy group is inserted in the para position to the ether oxygen in the salicylaldimine ligand and the nickel(II) complex is allowed to form at room temperature, a green compound crystallizes from acetone solution. If the ligand and metal are allowed to react in boiling acetone (56 °C), then two green crystalline modifications can be isolated from the solution: hexagonal crystals and rhombic crystals.

Elemental analysis of either modification yielded the following values. Anal. Calcd for $Ni(C_{20}H_{22}N_2O_4)I_2$: C, 36.02; H, 3.32; N, 4.20. Found: C, 36.15; H, 3.57; N, 4.01.

This study was undertaken to determine if the rhombic and hexagonal forms are the same material and also to determine the effect of the methoxy substituent of the ligand on the inner coordination sphere of the nickel.

This paper describes the crystal and molecular strucure of the rhombic form of $Ni(C_{20}H_{22}N_2O_4)I_2$. The structure of the hexagonal form is now under investigation and will hopefully appear shortly.

## Experimental Section

**Crystal Description and Preliminary Data.** The rhombic crystals were easily isolated from the mixture under a microscope. Preliminary Weissenberg and precession photographs indicated an orthorhombic system. Examination of the $0kl$, $1kl$, $hk0$, and $hk1$ layers taken with zirconium-filtered Mo K$\alpha$ radiation showed the following systematic extinctions: in $h0l$, $h = 2n + 1$; in $0kl$, $k + l = 2n + 1$. Two possible space groups are consistent with the observed extinctions: $Pna2_1$ and $Pnma$. The high percentage of data collected (72%) implied the noncentrosymmetric space group $Pna2_1$. Successful solution and refinement of the structure confirm this choice of space group.

A rhombic crystal with the dimensions 0.25 × 0.28 × 0.10 mm was mounted on the end of a glass fiber with epoxy cement. Accurate cell dimensions were determined on a Syntex $P2_1$ four-circle computer-controlled automatic diffractometer using graphite-monochromatized Mo K$\alpha$ radiation; 15 medium-angle reflections were accurately centered and least-squares refined to yield the following cell dimensions: $a = 20.931$ (8) Å, $b = 14.221$ (3) Å, $c = 7.478$ (1) Å, $V = 2226$ (1) Å$^3$. Standard deviations in the least significant digit, here and in the following tables, are given in parentheses. The density of the crystal was determined by the flotation method in a mixture of bromoform–chloroform and was found to be 2.00 (4) g cm$^{-3}$. The density calculated assuming $Z = 4$ is 1.99 g cm$^{-3}$. Assuming the crystal to be a cylinder of radius 0.015 cm with $\mu = 37.06$ cm$^{-1}$, the absorption correction factor for $0° < \theta < 30°$ ranges between 2.49 and 2.42, a change of less than 3%. Therefore absorption corrections were not deemed necessary.

**Data Collection and Reduction.** Intensity data were collected at 24 °C by the $\theta$–$2\theta$ scan technique out to a maximum of $2\theta = 60°$. Each scan covered a range of 0.65° below the $2\theta$ value calculated for K$\alpha_1$ and 0.65° above the $2\theta$ value calculated for K$\alpha_2$ for each reflection. The scan rate was varied according to the intensity accumulated during a preliminary 2-s peak count and ranged from 2.0 to 29.3° min$^{-1}$. Most of the observed data were collected in the 2.0–8.0° min$^{-1}$ range. Background measurements were made at the beginning and end of each scan with the counter stationary. The total time for background counts equaled the scan time and was equally distributed before and after the peak.

Four standard reflections were recorded every 46 reflections throughout the data collection and showed random variations of ±4% but no significant trends. A total of 2686 unique reflections $\geq 3\sigma(I)$ were collected out of a possible 3702. The intensities were calculated from

$$I_{hkl} = (P - (\mathrm{LB} + \mathrm{RB})) \times \mathrm{SR}$$

where $P$ is the peak count, LB is the left background count, RB is the right background count, and SR is the scan rate (degrees per minute). These intensities were corrected for any variations by computing decay factors on the basis of the four standard reflections. Fifteen peaks were rejected on the basis of a profile scan;[4] 2671 observed intensities were placed on an arbitrary scale by applying Lorentz and polarization corrections.[4] The polarization correction for the parallel–parallel mode Syntex $P2_1$ diffractometer was chosen assuming the monochromator crystal to be 50% mosaic and 50% perfect in character.[6] Initial standard deviations were calculated by

$$\sigma(I) = (P + \mathrm{LB} + \mathrm{RB})^{1/2} \times \mathrm{SR}$$

In order to incorporate a 3% instrumental error, $\sigma(F^2)$ was calculated from

$$\sigma(F^2) = 1/Lp(\sigma^2(I) + (0.03I)^2)^{1/2}$$

where $L$ and $p$ are the Lorentz and polarization factors, respectively. An estimate of the overall scale factor and overall temperature factor was obtained using Wilson's method.[4]

Table I. Fractional Atomic Coordinates for $Ni(C_{20}H_{22}N_2O_4)I_2$

| Atom | $x$ | $y$ | $z$ |
|---|---|---|---|
| I1 | 0.73157 (3) | 0.48523 (4) | 0.0[a] |
| I2 | 0.62500 (3) | 0.27206 (4) | 0.1510 (4) |
| Ni | 0.69764 (5) | 0.40238 (6) | 0.3203 (4) |
| O1 | 0.7900 (2) | 0.3310 (4) | 0.3304 (10) |
| O2 | 0.6115 (3) | 0.4788 (4) | 0.4255 (9) |
| O3 | 0.0229 (3) | 0.5051 (5) | 0.4547 (14) |
| O4 | 0.4070 (3) | 0.2886 (5) | 0.7255 (9) |
| N1 | 0.7387 (3) | 0.5147 (5) | 0.4419 (10) |
| N2 | 0.6870 (3) | 0.3288 (4) | 0.5562 (9) |
| C1 | 0.7974 (3) | 0.5298 (5) | 0.4740 (12) |
| C2 | 0.8517 (4) | 0.4673 (6) | 0.4297 (14) |
| C3 | 0.8479 (4) | 0.3753 (6) | 0.3619 (12) |
| C4 | 0.9048 (4) | 0.3279 (6) | 0.3208 (12) |
| C5 | 0.9644 (4) | 0.3685 (6) | 0.3497 (10) |
| C6 | 0.9679 (4) | 0.4584 (6) | 0.4185 (11) |
| C7 | 0.9120 (4) | 0.5073 (6) | 0.4572 (12) |
| C8 | 0.0809 (5) | 0.4528 (9) | 0.4642 (20) |
| C9 | 0.6932 (7) | 0.5897 (7) | 0.4745 (19) |
| C10 | 0.6325 (4) | 0.5499 (6) | 0.5521 (13) |
| C11 | 0.7793 (4) | 0.2492 (6) | 0.4450 (15) |
| C12 | 0.7452 (4) | 0.2827 (7) | 0.6130 (12) |
| C13 | 0.5725 (3) | 0.3494 (5) | 0.6067 (7) |
| C14 | 0.5606 (3) | 0.4274 (5) | 0.4980 (11) |
| C15 | 0.4990 (4) | 0.4572 (6) | 0.4664 (12) |
| C16 | 0.4472 (5) | 0.4804 (7) | 0.5437 (14) |
| C17 | 0.4594 (4) | 0.3306 (6) | 0.6535 (14) |
| C18 | 0.5212 (4) | 0.3022 (6) | 0.6848 (11) |
| C19 | 0.4159 (5) | 0.2068 (8) | 0.8291 (18) |
| C20 | 0.6373 (3) | 0.3128 (5) | 0.6475 (13) |
| HC1[b] | 0.8077 | 0.5926 | 0.5268 |
| HC4 | 0.9025 | 0.2635 | 0.2699 |
| HC5 | 0.0040 | 0.3317 | 0.3166 |
| HC7 | 0.9152 | 0.5732 | 0.5072 |
| HC15 | 0.4920 | 0.5138 | 0.3892 |
| HC16 | 0.4021 | 0.4272 | 0.5192 |
| HC18 | 0.5311 | 0.2448 | 0.7637 |
| HC20 | 0.6438 | 0.2739 | 0.7562 |
| H1C9 | 0.6842 | 0.6241 | 0.3621 |
| H2C9 | 0.7130 | 0.6354 | 0.5639 |
| H1C10 | 0.5995 | 0.6010 | 0.5651 |
| H2C10 | 0.6405 | 0.5213 | 0.6725 |
| H1C11 | 0.7525 | 0.2001 | 0.3869 |
| H2C11 | 0.8213 | 0.2202 | 0.4823 |
| H1C12 | 0.7735 | 0.3277 | 0.6777 |
| H2C12 | 0.7357 | 0.2275 | 0.6916 |

[a] Arbitrary coordinate. [b] Hydrogen positions were calculated and assigned isotropic temperature factors of 3.3 Å$^2$; HC1 is attached to C1.

**Structure Determination and Refinement.** The positional parameters of the two iodine and nickel atoms were easily obtained from a three-dimensional origin-removed sharpened Patterson–Fourier synthesis.[4] The coefficients used in this summation were obtained from

$$F_{hkl}^2(\text{modified}) = KF_{hkl}^2(\sum_i z_i^2/\sum_i f_i^2)\exp(2B\sin^2\theta_{hkl}/\lambda^2) - \sum_i z_i^2$$

where $K$ is the scale factor, $B$ is the overall temperature factor, $z_i$ and $f_i$ are the atomic number and scattering factor of the $i$th atom, respectively, and $\lambda$ is the wavelength.

A structure factor calculation,[4] based on the position of the three heavy atoms in the structure and using the 2671 observed intensities, led to a residual $R_{F^2} = 0.188$.[7] All of the remaining nonhydrogen light atoms were found in subsequent difference-Fourier syntheses.[4] The calculated residual $R_{F^2}$, with all nonhydrogen atoms included, was 0.124.[7] The positions of the 16 nonmethyl hydrogen atoms were calculated 1.0 Å from their respective carbon atoms.[4] These were kept fixed through all cycles of refinement and were arbitrarily assigned isotropic temperature factors of 3.3 Å$^2$. The structural parameters were refined by the full-matrix least-squares method,[4] initially minimizing the quantity

$$\sum_i w_i(||F_{o_i}|^2 - |F_{c_i}|^2|)^2$$

where $w_i$ is the weighting factor for each reflection, $F_o$ is the observed structure factor, and $F_c$ is the calculated structure factor. The weights

**Table II.** Thermal Parameters for $Ni(C_{20}H_{22}N_2O_4)I_2$[a]

| Atom | $10^4\beta_{11}$ | $10^4\beta_{22}$ | $10^4\beta_{33}$ | $10^4\beta_{12}$ | $10^4\beta_{13}$ | $10^4\beta_{23}$ |
|---|---|---|---|---|---|---|
| I1 | 18.1 (1) | 39.9 (2) | 113.5 (7) | −0.5 (1) | 1.0 (3) | 12.3 (5) |
| I2 | 23.6 (1) | 39.8 (2) | 111.0 (7) | −9.1 (2) | −0.7 (3) | −14.3 (5) |
| Ni | 13.0 (2) | 26.2 (4) | 89.5 (13) | 0.3 (2) | −3.0 (5) | −1.4 (7) |
| O1 | 16 (1) | 29 (2) | 143 (10) | 0 (1) | −3 (3) | 0 (5) |
| O2 | 14 (1) | 35 (3) | 108 (9) | 0 (2) | −6 (3) | 2 (4) |
| O3 | 15 (1) | 51 (3) | 323 (24) | −4 (2) | −4 (5) | −23 (8) |
| O4 | 16 (1) | 65 (3) | 202 (11) | −2 (2) | 8 (3) | −1 (6) |
| N1 | 15 (1) | 29 (3) | 109 (9) | 4 (2) | −8 (3) | −8 (5) |
| N2 | 14 (1) | 41 (3) | 120 (9) | 4 (1) | −2 (3) | 1 (4) |
| C1 | 15 (1) | 30 (3) | 173 (15) | −3 (2) | −2 (4) | −11 (5) |
| C2 | 13 (2) | 29 (4) | 116 (15) | 2 (2) | 0 (5) | 7 (7) |
| C3 | 12 (2) | 30 (4) | 97 (13) | −1 (2) | −8 (4) | 5 (6) |
| C4 | 16 (1) | 42 (3) | 129 (11) | −3 (2) | 6 (4) | −1 (6) |
| C5 | 16 (2) | 46 (3) | 120 (11) | −1 (2) | 5 (4) | −1 (6) |
| C6 | 14 (2) | 40 (3) | 161 (12) | −4 (2) | 0 (4) | −1 (6) |
| C7 | 15 (2) | 36 (3) | 171 (16) | −1 (2) | −7 (4) | −4 (6) |
| C8 | 13 (2) | 71 (7) | 228 (29) | −4 (3) | −6 (7) | −21 (13) |
| C9 | 15 (1) | 32 (3) | 180 (16) | 6 (2) | −10 (5) | −9 (6) |
| C10 | 18 (2) | 31 (3) | 149 (15) | 6 (2) | −5 (5) | −21 (6) |
| C11 | 15 (2) | 26 (4) | 135 (18) | 2 (2) | −1 (5) | 13 (7) |
| C12 | 15 (2) | 58 (5) | 126 (14) | 8 (2) | −7 (4) | 24 (7) |
| C13 | 17 (1) | 39 (3) | 66 (7) | 4 (2) | −4 (3) | −1 (4) |
| C14 | 14 (1) | 34 (3) | 91 (9) | 0 (2) | −2 (4) | 5 (6) |
| C15 | 17 (2) | 36 (3) | 122 (14) | 1 (2) | 0 (4) | 8 (6) |
| C16 | 16 (2) | 48 (4) | 175 (17) | 5 (2) | −6 (5) | 1 (7) |
| C17 | 15 (2) | 39 (3) | 117 (12) | 1 (2) | −5 (5) | −1 (7) |
| C18 | 14 (2) | 45 (4) | 102 (14) | 1 (2) | 1 (4) | 2 (6) |
| C19 | 20 (2) | 58 (5) | 190 (20) | −1 (3) | 12 (6) | 28 (10) |
| C20 | 19 (2) | 42 (3) | 110 (10) | 6 (2) | 3 (4) | 13 (6) |

[a] The anisotropic thermal ellipsoid is of the form $\exp(-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl))$.

were initially set equal to $1/\sigma_i^2(F_i^2)$.

The atomic scattering factors for all nonhydrogen atoms were taken from the Dirac–Slater calculations of Cromer and Waber.[8] The anomalous parts of the iodine and nickel scattering factors, as well as the scattering factors for hydrogen, were obtained from ref 9.

Further refinement was based on $F$;[4] a weighting scheme was chosen by an analysis of variance to make $|\Delta F|/\sigma$ independent of $|F_o|$.[4] This led to the following assignments for $\sigma(F_o)$

$$0 < |F_o| \leqslant 20.6 \qquad \sigma(F_o) = -0.0405|F_o| + 2.234$$

$$20.6 < |F_o| \leqslant 51.8 \qquad \sigma(F_o) = -0.006|F_o| + 1.412$$

$$|F_o| > 51.8 \qquad \sigma(F_o) = 0.0306|F_o| - 0.2004$$

With all nonhydrogen atoms allowed anisotropic thermal parameters, the refinement converged to values of the residuals $R_F = 0.038$ and $R_w = 0.046$.[7] The final error in an observation of unit weight, given by the error of fit function $[\sum_i w_i(\|F_{oi}| - |F_{ci}\|)^2/(n - m)]^{1/2}$, where $n$ is the number of observations and $m$ is the number of variable parameters, is 1.18.[4]

The observed and final calculated structure factor amplitudes, $|F_{oi}|$ and $|F_{ci}|$, are given in Appendix 1 (supplementary material). The initial scale factor converged to a value of 0.861. Table I lists the final parameters from the least-squares refinement, along with their standard deviations; the components of the anisotropic thermal tensors for the nonhydrogen atoms are given in Table II.

A final difference Fourier synthesis produced a general background of approximately 0.6 e Å$^{-3}$, with no peaks greater than 1.0 e Å$^{-3}$ which were found at the iodine sites.

## Description and Discussion of the Structure

The structure consists of discrete molecules of $Ni(C_{20}H_{22}N_2O_4)I_2$ as evidenced by the closest intermolecular distances of 2.50 Å between O4 (methoxy oxygen) and H1C9 and of 2.56 Å between O4 and H2C11. The configuration about the central nickel atom is found to be as follows: the two iodine atoms lie cis to one another, the two nitrogens lie cis to one another, and the two oxygen atoms of the salicylaldimine ligand lie trans to one another. Figure 1 is a stereoscopic representation of the molecule and indicates the labeling scheme. Tables III and IV provide bond distances and angles, respectively.

**Table III.** Bond Distances (Å) for $Ni(C_{20}H_{22}N_2O_4)I_2$

| | | | |
|---|---|---|---|
| Ni–I1 | 2.757 (1) | C4–C5 | 1.39 (1) |
| Ni–I2 | 2.711 (1) | C5–C6 | 1.38 (1) |
| Ni–O1 | 2.186 (5) | C6–C7 | 1.39 (1) |
| Ni–O2 | 2.248 (6) | C7–C2 | 1.40 (1) |
| Ni–N1 | 2.029 (7) | C6–O3 | 1.36 (1) |
| Ni–N2 | 2.063 (6) | O3–C8 | 1.43 (1) |
| O1–C3 | 1.39 (1) | C9–C10 | 1.51 (1) |
| O1–C11 | 1.46 (1) | C11–C12 | 1.52 (1) |
| O2–C10 | 1.45 (1) | C13–C20 | 1.48 (1) |
| O2–C14 | 1.40 (1) | C13–C14 | 1.40 (1) |
| N1–C1 | 1.27 (1) | C14–C15 | 1.38 (1) |
| N1–C9 | 1.45 (1) | C15–C16 | 1.41 (1) |
| N2–C20 | 1.26 (1) | C16–C17 | 1.40 (1) |
| N2–C12 | 1.45 (1) | C17–C18 | 1.37 (1) |
| C1–C2 | 1.47 (1) | C18–C13 | 1.39 (1) |
| C2–C3 | 1.40 (1) | C17–O4 | 1.36 (1) |
| C3–C4 | 1.40 (1) | O4–C19 | 1.41 (1) |

The Ni–I bond distances of 2.711 (1) and 2.757 (1) Å are consistent with the calculated sum of the covalent radii for octahedral Ni(II) complexes, 2.67 Å (octahedral Ni(II), 1.39 Å; I, 1.28 Å).[2b,10] This distance is significantly shorter than that found by Johnston and Horrocks[2b] (2.880 (1) Å) where the two iodines are trans to one another and occupy the apical positions in $Ni(C_{18}H_{18}N_2O_2)I_2$. The major differences between the structure under present investigation and that investigated by Johnston and Horrocks are as follows: (1) this compound is green; the latter is red; (2) the ligand of the former has methoxy groups para to the carbon atom bound to the oxygen of the salicylaldimine; the latter has phenyl rings; (3) this compound has iodines cis to one another, nitrogens cis to one another, and oxygens trans to one another; Johnston and Horrocks' compound has iodines, nitrogens, and oxygens all trans to one another, respectively;[2b] (4) this molecule possesses no crystallographic symmetry, whereas the latter has a twofold axis about the nickel atom.

Since the oxygens in this compound occupy apical positions and the iodines are cis to one another, the ether oxygen to Ni bond is weakened and thus long Ni–O bond lengths are found. Consequently, Ni–O distances of 2.186 (5) and 2.248 (6) Å

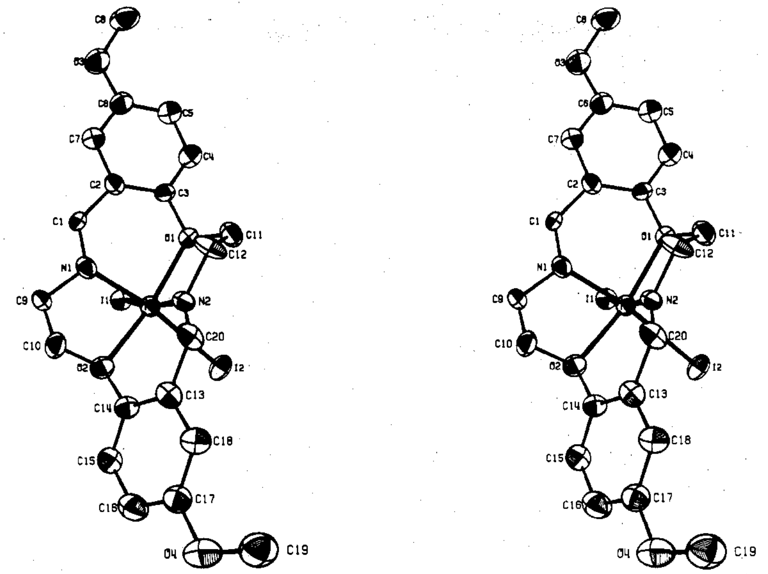

**Figure 1.** ORTEP[4] stereoscopic view depicting the molecular configuration of $Ni(C_{20}H_{22}N_2O_4)I_2$ with thermal ellipsoids representing 50% probability.

**Table IV.** Bond Angles (deg) for $Ni(C_{20}H_{22}N_2O_4)I_2$

| | | | |
|---|---|---|---|
| I1-Ni-I2 | 91.83 (4) | Ni-N2-C20 | 129.9 (5) |
| I1-Ni-O1 | 90.0 (2) | C12-N2-C20 | 116.9 (7) |
| I1-Ni-O2 | 107.6 (2) | N1-C9-C10 | 110.0 (6) |
| I1-Ni-N1 | 86.7 (2) | C9-C10-O2 | 105.4 (7) |
| I1-Ni-N2 | 170.2 (2) | N2-C12-C11 | 107.1 (7) |
| I2-Ni-O1 | 101.2 (1) | C12-C11-O1 | 107.9 (7) |
| I2-Ni-O2 | 92.5 (2) | O1-C3-C2 | 122.6 (8) |
| I2-Ni-N1 | 170.1 (2) | N1-C1-C2 | 126.9 (7) |
| I2-Ni-N2 | 89.6 (2) | O1-C3-C4 | 118.7 (7) |
| O1-Ni-O2 | 157.3 (3) | C1-C2-C3 | 126.6 (8) |
| O1-Ni-N1 | 88.6 (2) | C2-C3-C4 | 118.6 (8) |
| O1-Ni-N2 | 80.2 (2) | C3-C4-C5 | 121.8 (7) |
| O2-Ni-N1 | 78.6 (2) | C4-C5-C6 | 119.3 (7) |
| O2-Ni-N2 | 81.9 (2) | C5-C6-C7 | 119.7 (8) |
| N1-Ni-N2 | 93.5 (3) | C6-C7-C2 | 121.7 (8) |
| Ni-O1-C3 | 124.4 (5) | C7-C2-C3 | 118.8 (8) |
| Ni-O1-C11 | 104.7 (4) | C1-C2-C7 | 114.6 (7) |
| C3-O1-C11 | 113.2 (7) | C5-C6-O3 | 125.0 (7) |
| Ni-O2-C10 | 108.7 (5) | C7-C6-O3 | 115.3 (8) |
| Ni-O2-C14 | 119.5 (4) | C6-O3-C8 | 118.5 (8) |
| C10-O2-C14 | 109.7 (6) | O2-C14-C13 | 122.8 (6) |
| Ni-N1-C1 | 128.9 (6) | C14-C13-C20 | 123.4 (6) |
| Ni-N1-C9 | 112.1 (5) | C13-C20-N2 | 125.8 (7) |
| C1-N1-C9 | 118.6 (7) | C20-C13-C18 | 117.5 (6) |
| Ni-N2-C12 | 113.0 (5) | O2-C14-C15 | 118.8 (6) |
| C13-C14-C15 | 120.6 (7) | C18-C13-C14 | 119.1 (7) |
| C14-C15-C16 | 119.8 (8) | C16-C17-O4 | 115.6 (8) |
| C15-C16-C17 | 119.2 (8) | C18-C17-O4 | 124.2 (8) |
| C16-C17-C18 | 120.2 (8) | C17-O4-C19 | 118.2 (7) |
| C17-C18-C13 | 121.0 (8) | | |

are much longer than the sum of the covalent radii, 2.05 Å (octahedral Ni(II), 1.39 Å; O, 0.66 Å).[2b,10]

The Ni–N distances of 2.029 (7) and 2.063 (6) Å are in good agreement with that of high-spin octahedral bis(*N*-γ-dimethylaminopropylsalicylaldiminato)nickel(II), 2.05 Å.[11]

Strain in the macrocyclic coordination system is evidenced mainly in the puckering of the five-membered ring systems formed by O2–C10–C9–N1 and the Ni and by O1–C11–C12–N2 and the Ni. These two five-membered ring systems differ in spatial configuration (see Figure 1). This strain is evidenced also by the high degree of anisotropy found for the methylene carbons. This puckering, coupled with the fact that both methoxy carbon atoms lie on the same side of a plane defined by the two methoxy oxygens and the nickel atom (see Figure 1), destroys the possibility of a twofold axis through the nickel atom. The substituted phenyl rings are unremarkable, except in the fact that the bond distances exhibit very small deviations from the mean value of 1.39 (1) Å.

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**Registry No.** $Ni(C_{20}H_{22}N_2O_4)I_2$, 57719-37-6.

**Supplementary Material Available:** listing of structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

## References and Notes

(1) This compound crystallizes in two forms: green rhombic crystals and green hexagonal crystals. The structure of the hexagonal form is now under investigation.

(2) (a) R. W. Kluiber and G. Sasso, *Inorg. Chim. Acta*, **4**, 226 (1970); (b) D. L. Johnston and W. D. Horrocks, Jr., *Inorg. Chem.*, **10**, 687 (1971).

(3) Private communication.

(4) Computations were performed on an IBM 370-158 at the Center for Computer and Information Services, Rutgers, The State University of New Jersey, New Brunswick, N.J. Programs used were of local origin and also included the following: refinement of cell dimensions, Syntex program; data reduction and *Lp* correction, local modification of Glick's SYNCOR; Patterson function and Fourier syntheses, Zalkin's FORDAP; least squares of refinement and structure factor calculation, Busing, Martin, and Levy's ORFLS; hydrogen position calculating program, Shiono's GENATM; bond distance and angle program, ORFFE function and error program; analysis of variance program, NANOVA (J. S. Ricci, Jr., C. A. Eggers, and I. Bernal, *Inorg. Chim. Acta*, **6**, 97 (1972)); Johnson's ORTEP II thermal ellipsoid plotting program.

(5) "International Tables for X-Ray Crystallography", Vol. II, Kynoch Press, Birmingham, England, 1962, p 302.

(6) "Syntex XTL Manual", Syntex Analytical Instruments, Inc., Cupertino, Calif., 1973.

(7) $R_{F^2} = \sum \| F_o|^2 - |F_c|^2| / \Sigma |F_o|^2$; $R_F = \sum \| F_o| - |F_c\| / \Sigma |F_o|$; $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w |F_o|^2]^{1/2}$.

(8) D. T. Cromer and J. T. Waber, *Acta Crystallogr.*, **18**, 104 (1965).

(9) "International Tables for X-Ray Crystallography", Vol. III, Kynoch Press, Birmingham, England, 1962, pp 202, 215.

(10) L. Pauling, "The Nature of the Chemical Bond", 3d ed, Cornell University Press, Ithaca, N.Y., 1960, Tables 7-5, 7-13, 7-15.

(11) S. W. Hawkinson and E. B. Fleischer, *Inorg. Chem.*, **8**, 2402 (1969).