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Structure of Bis(diphenylboron-dimethylglyoximato)nickel(II)*

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Abstract. [Ni($C_{32}H_{32}B_2N_4O_4$)], M_r = 616·96, triclinic, $P\bar{1}$, a = 8·385 (2), b = 14·068 (3), c = 14·234 (3) Å, α = 75·20 (2), β = 72·74 (2), γ = 72·53 (2)°, V = 1503·8 Å^3, Z = 2, D_m = 1·36, D_x = 1·37 g cm^{-3}, Mo $K\alpha$ radiation (λ = 0·71069 Å), μ = 6·91 cm^{-1}, $F(000)$ = 1148, room temperature, R = 0·042 for 4366 observed reflections. The macrocyclic ligand has a pronounced saddle-shaped conformation. In this four-coordinate complex, the coordination geometry around the Ni^{II} atom is a distorted square pyramid and the Ni^{II} atom deviates from the coordination plane by 0·14 Å as is found in five-coordinate square pyramidal Ni^{II} complexes.

Introduction. Ordinarily the coordination geometry around the Ni^{II} atom is square planar in four-coordinate low-spin complexes, but larger deviations from exact coplanarity may occur in unsymmetrical complexes (Wells, 1984). A number of papers have reported larger tetrahedral distortion owing to steric interference between the ligands (Braun & Lingafelter, 1967; Cotton, DeBoer & Pipal, 1970). The present work describes a novel square-pyramidal distortion in a four-coordination Ni^{II} complex.

Experimental. The complex was prepared by mixing stoichiometric amounts of $NiCl_2.6H_2O$, dimethylglyoxime and sodium tetraphenylboron in a chloroform–ethanol solution. The solution was refluxed for 12 h. After the reaction mixture had cooled to room temperature, the resulting white precipitate was separated by filtration, then reddish crystals suitable for X-ray analysis were formed by evaporation at 343 K. D_m was determined by flotation (CCl_4–C_6H_6). A well shaped crystal with dimensions 0·20 × 0·30 × 0·28 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. Unit-cell parameters were determined by least-squares fit of 2θ values of 25 higher-order reflections ($20 \leq 2\theta \leq 28°$). Intensity data were collected by the ω–2θ scan using graphite-monochromated Mo $K\alpha$ radiation, the scan width was $\Delta\omega$

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* {Bis-μ-[2,3-butanedionedioximato(2−)-O,O']-bis(diphenylborio-N,N',N'',N''')}nickel(II).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters of non*-H *atoms with e.s.d.'s in parentheses*

	x	y	z	B_{eq}^*(Å^2)
Ni	0·21255 (6)	0·25005 (4)	0·25001 (3)	3·31
O(1)	0·3187 (3)	0·0553 (2)	0·1928 (2)	4·48
O(2)	0·1649 (3)	0·4452 (2)	0·1244 (2)	4·47
O(3)	0·0667 (3)	0·4451 (2)	0·3070 (2)	4·00
O(4)	0·2348 (3)	0·0548 (2)	0·3755 (2)	4·02
N(1)	0·2845 (4)	0·1573 (2)	0·1634 (2)	3·75
N(2)	0·2094 (4)	0·3432 (2)	0·1313 (2)	3·93
N(3)	0·1055 (4)	0·3432 (2)	0·3364 (2)	3·75
N(4)	0·1841 (4)	0·1568 (2)	0·3687 (2)	3·85
C(1)	0·2779 (5)	0·1954 (3)	0·0716 (3)	4·27
C(2)	0·2339 (5)	0·3065 (3)	0·0516 (3)	4·28
C(3)	0·0452 (5)	0·3049 (3)	0·4283 (3)	4·06
C(4)	0·0918 (5)	0·1938 (3)	0·4484 (3)	4·09
C(11)	0·3143 (7)	0·1315 (4)	−0·0055 (3)	6·57
C(21)	0·2175 (7)	0·3699 (3)	−0·0474 (3)	6·59
C(31)	−0·0604 (6)	0·3681 (4)	0·5055 (3)	5·28
C(41)	0·0399 (6)	0·1306 (4)	0·5482 (6)	6·15
C(101)	0·4211 (5)	−0·1018 (3)	0·3142 (3)	4·10
C(102)	0·3760 (6)	−0·1598 (3)	0·2635 (3)	5·85
C(103)	0·4143 (7)	−0·2650 (4)	0·2888 (4)	7·57
C(104)	0·4964 (7)	−0·3144 (3)	0·3641 (4)	7·04
C(105)	0·5402 (7)	−0·2589 (4)	0·4162 (4)	8·76
C(106)	0·5042 (6)	−0·1542 (3)	0·3899 (3)	7·07
C(107)	0·5463 (4)	0·0627 (3)	0·2767 (3)	4·11
C(108)	0·6756 (5)	0·0616 (3)	0·1886 (3)	4·99
C(109)	0·8271 (6)	0·0875 (4)	0·1801 (4)	6·49
C(110)	0·8526 (6)	0·1163 (4)	0·2596 (4)	9·07
C(111)	0·7257 (6)	0·1202 (4)	0·3458 (4)	8·67
C(112)	0·5754 (5)	0·0936 (3)	0·3545 (3)	6·17
C(201)	0·1330 (5)	0·6018 (3)	0·1863 (3)	4·43
C(202)	0·2404 (6)	0·6543 (3)	0·1100 (3)	5·80
C(203)	0·1974 (7)	0·7592 (4)	0·0841 (4)	8·28
C(204)	0·0463 (7)	0·8144 (4)	0·1358 (4)	8·38
C(205)	−0·0621 (7)	0·7653 (4)	0·2112 (4)	6·12
C(206)	−0·0200 (5)	0·6600 (3)	0·2360 (3)	5·07
C(207)	0·3857 (5)	0·4378 (3)	0·2235 (3)	4·31
C(208)	0·5240 (5)	0·4067 (3)	0·1456 (3)	5·46
C(209)	0·6923 (6)	0·3796 (4)	0·1536 (4)	6·73
C(210)	0·7289 (6)	0·3834 (4)	0·2407 (4)	8·66
C(211)	0·5951 (7)	0·4122 (4)	0·3196 (4)	8·78
C(212)	0·4264 (5)	0·4385 (3)	0·3113 (3)	6·00
B(1)	0·3832 (5)	0·0198 (3)	0·2891 (3)	4·00
B(2)	0·1917 (5)	0·4806 (3)	0·2115 (3)	3·96

* As defined by Hamilton (1959).

$= (0{\cdot}80 + 0{\cdot}35\tan\theta)^\circ$, and scan rate was variable from 2·0 to 5·5° $\min^{-1}$. A total of 6147 independent reflections were measured with 2θ up to 54° and index range $h = 0$ to 18, $k = -18$ to 18, $l = -18$ to 18. 4366 observed reflections [$|F_o| \geq 3\sigma(F_o)$, $\sin\theta/\lambda \leq 0{\cdot}62$ Å^{-1}] were employed in the refinement. Three standard reflections were measured every hour, no intensity variation being observed. Correction for Lorentz–polarization effects were applied, but no absorption or extinction corrections were carried out.

The structure was solved by the Patterson heavy-atom method followed by Fourier syntheses, and refined by block-diagonal least squares using the *HBLS*-V program (Ashida, 1979). $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1$. The H atoms of the phenyl rings were placed 1·08 Å from the bonded C atoms with a fixed isotropic thermal parameter $B =$ 4·0 Å^2. The H atoms of the methyl groups were located on a difference Fourier map reasonably clearly and refined isotropically. The number of reflections per refined parameter is 4366/437 = 9·99. $R = 0{\cdot}042$, $wR = 0{\cdot}046$, $S = 1{\cdot}01$, $(\Delta/\sigma)_{\max} = 0{\cdot}69$ for non-H atoms. $(\Delta\rho)_{\max} = 0{\cdot}5$, $(\Delta\rho)_{\min} = -0{\cdot}8$ e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All computations were performed on a Honeywell DPS8 computer at the Computation Center, Zhejiang University.

Discussion. The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.* An *ORTEP* drawing of the molecule together with the atomic numbering scheme is shown in Fig. 1.

It is interesting to note that the molecule has approximate $C2$ symmetry around the Ni atom (see Table 1) and the Ni atoms occupy the approximate special positions (x, $\frac{1}{4}$, $\frac{1}{4}$) and ($1 - x$, $\frac{3}{4}$, $\frac{3}{4}$) in the cell. Thus, there are the following four approximate equivalent positions in the unit cell: (1) x, y, z; (2) $-x, -y, -z$; (3) $x', \frac{1}{2} - y, \frac{1}{2} - z$; (4) $-x', \frac{1}{2} + y, \frac{1}{2} + z$ (where no certain relationship exists between x and x'). They result in an approximate systematic absence $k + l = 2n + 1$ for $0kl$ reflections in the triclinic system.

The remarkable structural feature of the complex is that the ligand has a pronounced saddle-shaped conformation. The four N atoms of the macrocyclic ligand make up a highly planar base with the four O atoms, but the two B atoms are directed out of the N_4 coordination plane by 0·769 and 0·771 Å, respectively, in the same direction, while both dimethylglyoxime fragments (N1—N2—O1—O2—C1—C2—C11—C21 and N3—N4—O3—O4—C3—C4—C31—C41) are essentially planar but bent down from the N_4 plane with a dihedral angle of about 27° between the two least-squares planes of the dimethylglyoxime fragments. This conformation contrasts

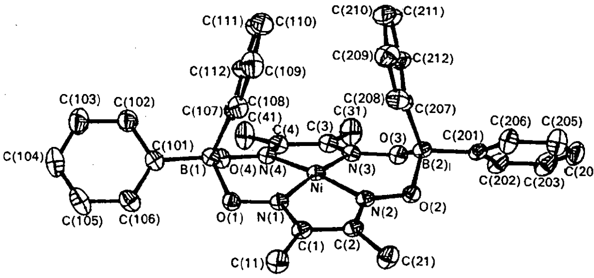

Fig. 1. *ORTEP* (Johnson, 1974) drawing of the molecule with atomic numbering scheme. Non-H atoms are thermal ellipsoids drawn at the 30% probability level, and H atoms are omitted for clarity.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and selected least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52788 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and angles* (°) *for non*-H *atoms with e.s.d.'s in parentheses*

Ni—N(1)	1·870 (3)	Ni—N(2)	1·857 (3)
Ni—N(3)	1·870 (3)	Ni—N(4)	1·857 (3)
O(1)—N(1)	1·354 (4)	O(1)—B(1)	1·533 (5)
O(2)—N(2)	1·355 (4)	O(2)—B(2)	1·544 (5)
O(3)—N(3)	1·352 (4)	O(3)—B(2)	1·522 (5)
O(4)—N(4)	1·356 (4)	O(4)—B(1)	1·528 (5)
N(1)—C(1)	1·290 (5)	N(2)—C(2)	1·301 (5)
N(3)—C(3)	1·291 (5)	N(4)—C(4)	1·302 (5)
C(1)—C(2)	1·466 (6)	C(3)—C(4)	1·467 (6)
C(1)—C(11)	1·498 (7)	C(2)—C(21)	1·483 (7)
C(3)—C(31)	1·494 (7)	C(4)—C(41)	1·490 (7)
B(1)—C(101)	1·607 (6)	B(1)—C(107)	1·601 (6)
B(2)—C(201)	1·602 (6)	B(2)—C(207)	1·601 (6)
C(101)—C(102)	1·406 (6)	C(101)—C(106)	1·386 (7)
C(102)—C(103)	1·391 (8)	C(103)—C(104)	1·370 (8)
C(104)—C(105)	1·387 (8)	C(105)—C(106)	1·385 (8)
C(107)—C(108)	1·393 (6)	C(107)—C(112)	1·394 (6)
C(108)—C(109)	1·387 (7)	C(109)—C(110)	1·386 (8)
C(110)—C(111)	1·366 (8)	C(111)—C(112)	1·382 (7)
C(201)—C(202)	1·395 (6)	C(201)—C(206)	1·394 (6)
C(202)—C(203)	1·388 (8)	C(203)—C(204)	1·378 (8)
C(204)—C(205)	1·369 (8)	C(205)—C(206)	1·392 (7)
C(207)—C(208)	1·395 (6)	C(207)—C(212)	1·392 (6)
C(208)—C(209)	1·379 (7)	C(209)—C(210)	1·379 (8)
C(210)—C(211)	1·377 (8)	C(211)—C(212)	1·384 (7)

N(1)—Ni—N(2)	82·3 (1)	N(1)—Ni—N(3)	171·0 (1)
N(1)—Ni—N(4)	96·9 (1)	N(2)—Ni—N(3)	96·8 (1)
N(2)—Ni—N(4)	171·8 (1)	N(3)—Ni—N(4)	82·6 (1)
N(1)—O(1)—B(1)	112·6 (3)	N(2)—O(2)—B(2)	113·9 (3)
N(3)—O(3)—B(2)	112·7 (3)	N(4)—O(4)—B(1)	113·8 (3)
Ni—N(1)—O(1)	124·6 (2)	Ni—N(1)—C(1)	115·8 (3)
O(1)—N(1)—C(1)	118·9 (3)	Ni—N(2)—O(2)	124·8 (2)
Ni—N(2)—C(2)	116·2 (3)	O(2)—N(2)—C(2)	118·6 (3)
Ni—N(3)—O(3)	124·8 (2)	Ni—N(3)—C(3)	115·5 (3)
O(3)—N(3)—C(3)	119·0 (3)	Ni—N(4)—O(4)	124·8 (2)
Ni—N(4)—C(4)	116·1 (3)	O(4)—N(4)—C(4)	118·7 (3)
N(1)—C(1)—C(2)	112·8 (4)	N(1)—C(1)—C(11)	122·8 (4)
C(2)—C(1)—C(11)	124·4 (4)	N(2)—C(2)—C(1)	112·0 (4)
N(2)—C(2)—C(21)	123·8 (4)	C(1)—C(2)—C(21)	124·2 (4)
N(3)—C(3)—C(4)	113·0 (4)	N(3)—C(3)—C(31)	123·0 (4)
C(4)—C(3)—C(31)	123·9 (4)	N(4)—C(4)—C(3)	112·0 (4)
N(4)—C(4)—C(41)	124·0 (4)	C(3)—C(4)—C(41)	124·0 (4)
O(1)—B(1)—O(4)	108·4 (3)	O(1)—B(1)—C(101)	107·3 (3)
O(1)—B(1)—C(107)	110·3 (3)	O(4)—B(1)—C(101)	104·6 (3)
O(4)—B(1)—C(107)	111·9 (3)	C(101)—B(1)—C(107)	113·9 (3)
O(2)—B(2)—O(3)	108·1 (3)	O(2)—B(2)—C(201)	104·6 (3)
O(2)—B(2)—C(207)	111·9 (3)	O(3)—B(2)—C(201)	107·6 (3)
O(3)—B(2)—C(207)	110·6 (3)	C(201)—B(2)—C(207)	113·8 (3)
B(1)—C(101)—C(102)	124·1 (4)	B(1)—C(101)—C(106)	118·9 (4)
C(102)—C(101)—C(106)	117·0 (4)	C(101)—C(102)—C(103)	120·9 (4)
C(102)—C(103)—C(104)	120·6 (5)	C(103)—C(104)—C(105)	119·7 (5)
C(104)—C(105)—C(106)	119·6 (5)	C(101)—C(106)—C(105)	122·2 (5)
B(1)—C(107)—C(108)	120·3 (4)	B(1)—C(107)—C(112)	122·8 (4)
C(108)—C(107)—C(112)	116·6 (4)	C(107)—C(108)—C(109)	121·5 (4)
C(108)—C(109)—C(110)	120·4 (5)	C(109)—C(110)—C(111)	119·1 (6)
C(110)—C(111)—C(112)	120·5 (5)	C(107)—C(112)—C(111)	122·0 (5)
B(2)—C(201)—C(202)	118·9 (4)	B(2)—C(201)—C(206)	124·4 (4)
C(202)—C(201)—C(206)	116·7 (4)	C(201)—C(202)—C(203)	121·9 (5)
C(202)—C(203)—C(204)	119·8 (5)	C(203)—C(204)—C(205)	119·8 (6)
C(204)—C(205)—C(206)	120·3 (5)	C(201)—C(206)—C(205)	121·5 (4)
B(2)—C(207)—C(208)	122·9 (4)	B(2)—C(207)—C(212)	120·5 (4)
C(208)—C(207)—C(212)	116·3 (4)	C(207)—C(208)—C(209)	122·3 (4)
C(208)—C(209)—C(210)	120·1 (5)	C(209)—C(210)—C(211)	119·1 (6)
C(210)—C(211)—C(212)	120·5 (5)	C(207)—C(212)—C(211)	121·7 (4)

with the five-coordinate $\mathrm{Ni^{II}}$ complex (Stephens & Vagg, 1981) and the six-coordinate $\mathrm{Fe^{II}}$ complex (Jansen & Verhage, 1982), both of which have macrocyclic ligands similar to the title complex.

Another notable feature of the title complex is that the $\mathrm{Ni^{II}}$ atom deviates from the N_4 coordination plane by 0·14 Å in the same direction as the B atoms. Hence the coordination geometry around the Ni atom is a distorted square pyramid as in five-coordinate $\mathrm{Ni^{II}}$ complexes (Stephens & Vagg, 1981; Anderson, 1981).

This apical displacement seems to be a consequence of the flexing of the dimethylglyoxime fragments. The fact that the lone electron pairs of the N atoms do not point towards the center of the N_4 coordination plane but are directed slightly out of this plane results in the displacement of the Ni atom towards one side of the plane in order to maximize the overlap of the ligand and metal orbitals.

The bond distances and angles are listed in Table 2. All of the intramolecular bond distances and angles fall into the expected range. The $\mathrm{Ni^{II}}$—N distances agree well with the average distance of 1·86 Å for square planar nickel(II) complexes (Tomlinson, 1981), although the Ni atom departs significantly from the coordination plane in the title complex.

The crystal structure projected along the *a** axis is given in Fig. 2. All intermolecular atomic contacts are considered to be normal van der Waals distances, the shortest non-H atomic contact being 3·670 (8) Å between C(41) and C(111) (1 − *x*, −*y*, 1 − *z*) atoms.

The remaining question is why the dimethylglyoxime planes are bent down from the N_4 plane. The distances between the B and C atoms bonded to N atoms fall in the range 3·579 (6) to 3·627 (6) Å. They are significantly longer than values of 3·50 Å in the corresponding $\mathrm{Ni^{II}}$ complex (Stephens & Vagg, 1981) and 3·52 Å in the $\mathrm{Co^{II}}$ complex (Bakac, Brynildson & Espenson, 1986). In those complexes the glyoxime planes are not bent from the coordination plane in the opposite direction to the B atoms. Therefore, it seems that no unusual steric interference exists within the molecule of the title complex. The intermolecular distances between the methyl C atoms and the non-H atoms of neighboring molecules are longer than 3·60 Å, and this also suggests that there is no steric repulsion between the dimethylglyoxime groups and neighboring molecules.

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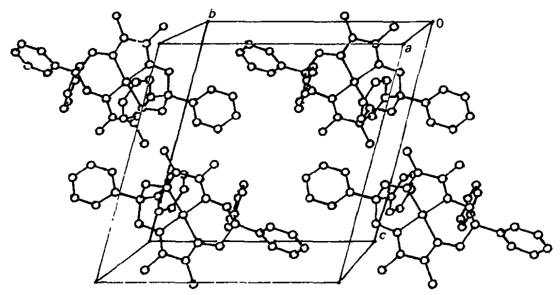

Fig. 2. Crystal structure projected along the *a** axis. H atoms are omitted for clarity.

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Structure of L-1-Benzyl-4-hydroxymethyl-2-azetidinone

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Abstract. $C_{11}H_{13}NO_2$, $M_r = 191{\cdot}22$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}183\,(1)$, $b = 6{\cdot}364\,(1)$, $c = 21{\cdot}79\,(1)$ Å, $V = 996{\cdot}07\,(1)$ Å^3, $Z = 4$, $D_m = 1{\cdot}25$, $D_x = 1{\cdot}275$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 5{\cdot}2$ cm^{-1}, $F(000) = 408$, $T = 298$ K. Final $R = 0{\cdot}045$ for 1164 observed reflections. In the β-lactam system, the amide group is almost planar with bond distances 1·334 (4) and 1·220 (4) Å for C—N and C═O, respectively. These structural results are consistent with those of other biologically inactive β-lactams.

Introduction. In those β-lactams having antibiotic activity, Sweet & Dahl (1970) have pointed out that the lactam N atom has pyramidal character. Increased activity was also correlated with decreased electron delocalization in the amide group as shown by lengthening of the C—N bond and shortening of the C═O bond. The crystal-structure determination now reported for L-1-benzyl-4-hydroxymethyl-2-azetidinone (Fig. 1) shows molecular features for this biologically inactive β-lactam which help to confirm the Sweet & Dahl hypothesis.

Experimental. A sample of the title compound was provided by C. H. Lee. Colorless rectangular crystals were grown from dichloromethane solution. The crystal density was measured by flotation in benzene/carbon tetrachloride. A crystal measuring 0·3 × 0·3 × 0·5 mm was used for data collection with an Enraf–Nonius CAD-4 diffractometer using Nb-filtered Mo $K\alpha$ radiation. Unit-cell parameters were determined from 25 reflections having $30 < 2\theta < 40°$. Intensities were measured for 1720 reflections with $(\sin\theta)/\lambda < 0{\cdot}70$ Å^{-1}, indices in the range $0 \le h \le 10$, $0 \le k \le 8$ and $0 \le l \le 30$ using $\omega/2\theta$ scans with scan width $\Delta\omega = (0{\cdot}7 + 0{\cdot}35\tan\theta)°$. Three monitor reflections showed deviations less than 3% from their average intensities. No absorption corrections were applied. There were 1164 reflections with $|F_o| > 3\sigma(F_o)$ which were used in the structure refinement.

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