

the five crystallographically distinct Na⁺ atoms all have sixfold regular octahedral geometries.

The strongest hydrogen bonding found in this compound involves water molecule H(1)—O(1)—H(2). H(1) forms a hydrogen bond to O(3) with an O—O distance of 2.876 Å, while H(2) forms a hydrogen bond to N(2) with an O—N distance of 2.899 Å. The other two water molecules do not participate in strong hydrogen bonding, the only exception being H(4), which is bonded to N(3) (O—N distance = 2.985 Å). The atoms H(3), H(5), and H(6) all participate in weaker hydrogen bonding of the bifurcated type as shown in Table 3(D).

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The Crystal and Molecular Structure of Bis(difluoroborondimethylglyoximato)nickel(II)

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The structure of the title compound has been refined to *R* 0.093 for 964 photographic reflexions. The monoclinic unit cell, space group *C2/c*, has *a* = 20.815 (40), *b* = 7.860 (18), *c* = 19.545 (40) Å, β = 110.8 (1)°, *Z* = 8. The complex consists of molecular dimers showing a weak Ni—Ni interaction of length 3.207 (6) Å; the space group imposes 2 (*C*₂) symmetry on these units. The dimethylglyoxime residues and the Ni atom in each formula unit are coplanar and the BF₂ groups are displaced out of the plane and away from the other formula moiety. The environment about each Ni is distorted square-planar with average Ni—N = 1.87 (1) Å and N—Ni—N = 83.5 (8) and 96.5 (8)°. Each planar formula unit is rotated some 92° relative to the other in the dimer around the Ni—Ni direction.

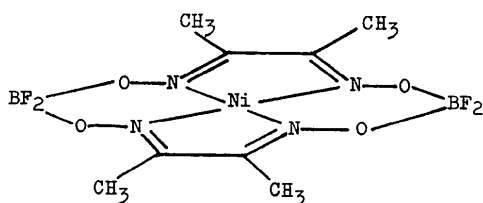
Introduction

Much of the current interest in transition-metal chelates of substituted glyoximes is centred on their use as chemical models for biological processes such as vitamin B₁₂ (Brown, 1973; Pratt & Craig, 1973; Schrauzer, 1976) and oxygen transport (McLendon &

Martell, 1976) functions, their activity in biological test systems (Carlsson, Charlson & Watton, 1974; Takamiya, 1960) and their semiconducting properties in the solid state (Thomas & Underhill, 1969, 1972; Underhill, Watkins & Pethig, 1973). These chelates appear to be quite stable, both thermodynamically and chemically, although those of Co demonstrate an

affinity for further reaction at the metal centre (McLendon & Martell, 1976; Schrauzer, 1976) and those of the Group VIIIb metals demonstrate an ability to form mixed-valence compounds (Gleizes, Marks & Ibers, 1975; Thomas & Underhill, 1972; Underhill, Watkins & Pethig, 1973). In each case, however, the glyoxime moieties remain planar and inert.

Schrauzer (1962) first proposed that the reaction of BF_3 with bis(dimethylglyoximate)nickel(II) resulted in the substitution of the two protons involved in $\text{O}\cdots\text{O}$ bridging by BF_2 units to yield the macrocyclic title compound (I). Unlike its parent glyoximate this yellow diamagnetic species forms adducts with monodentate ligands which, however, retain diamagnetic character. We have studied a series of these complexes with several glyoxime substituents and various nitrogenous monodentate and bidentate bases (Stephens, Vagg & Watton, 1977). This paper describes the determination of the structure of (I).



Experimental

The complex was prepared by a similar method to that of Schrauzer (1962). Recrystallization from acetone yielded long yellow needle crystals.

Crystal data

$\text{C}_8\text{H}_{12}\text{B}_2\text{F}_4\text{N}_4\text{NiO}_4$, $M_r = 384.5$, monoclinic, $a = 20.815$ (40), $b = 7.860$ (18), $c = 19.545$ (40) Å, $\beta = 110.8$ (1)°, $U = 2989.3$ Å³, $D_m = 1.70$ (by flotation), $Z = 8$, $D_c = 1.709$ g cm⁻³, $F(000) = 1552$. Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 13.5$ cm⁻¹, for cell dimensions and intensity measurements. Systematic absences: hkl if $h + k \neq 2n$, $h0l$ if $l \neq 2n$. Space group Cc or $C2/c$.

Cell parameters were determined from precession photographs with Mo $K\alpha$ radiation. Intensities were estimated visually from precession photographs for the layers 0–2 about [100], 0–2 about [010] and 0–5 about [001]. Each reflexion was corrected for Lorentz and polarization effects but not for absorption or extinction. 1206 non-zero reflexions were recorded which, after correlation, yielded 964 unique reflexions on a common scale.

The scattering factors were taken from *International Tables for X-ray Crystallography* (1962). All calculations were carried out on a Univac 1106 computer with programs written by FSS.

Structure determination

The approximate positions of all atoms except B and F were found from a Patterson synthesis, which also indicated the centrosymmetric space group $C2/c$; this was confirmed subsequently by the successful refinement of the structure. Fourier syntheses located the remaining non-hydrogen atoms.

The structure was refined by full-matrix least squares; the function minimized was $\Sigma w\Delta^2$. The weight for each reflexion was initially unity; in the final refinement $w = (1.0 + 0.1|F_o|)^{-1}$. For this latter weighting scheme the average values of $w\Delta^2$ for ranges of increasing $|F_o|$ were almost constant. Reflexions for which $|F_o| < 0.25|F_o|$ were omitted.

Initially positional and individual isotropic thermal parameters were refined. A difference synthesis revealed the H atoms of the methyl groups. Refinement was continued with H atoms included, but their parameters were not refined.

Final refinement included anisotropic thermal parameters for all non-hydrogen atoms, and was

Table 1. Final atomic coordinates (fractional; $\times 10^4$ for non-hydrogen atoms, $\times 10^3$ for hydrogen atoms) with estimated standard deviations in parentheses

	x	y	z
Ni(1)	0	396 (5)	2500
Ni(2)	0	4477 (6)	2500
F(11)	1531 (6)	-87 (20)	1413 (6)
F(12)	951 (6)	-2038 (18)	1832 (8)
F(21)	1594 (6)	4928 (19)	4708 (6)
F(22)	979 (6)	6889 (18)	3883 (8)
O(11)	435 (6)	479 (17)	1259 (8)
O(12)	1364 (7)	510 (17)	2452 (7)
O(21)	1375 (7)	4393 (17)	3511 (9)
O(22)	467 (6)	4427 (16)	4053 (8)
N(11)	-94 (6)	353 (16)	1513 (8)
N(12)	961 (8)	361 (20)	2875 (7)
N(21)	959 (7)	4427 (20)	2806 (13)
N(22)	-54 (9)	4495 (19)	3425 (12)
C(11)	716 (9)	466 (23)	3945 (12)
C(12)	1243 (8)	434 (23)	3592 (12)
C(21)	1223 (9)	4509 (27)	2325 (13)
C(22)	676 (11)	4438 (26)	1552 (12)
C(M11)	900 (10)	460 (30)	4767 (12)
C(M12)	2013 (10)	469 (30)	4015 (12)
C(M21)	1960 (10)	4396 (34)	2421 (12)
C(M22)	873 (12)	4376 (31)	914 (14)
B(1)	1070 (12)	-266 (41)	1788 (16)
B(2)	1113 (13)	5161 (36)	4050 (16)
H(111)	125	15	484
H(112)	106	163	480
H(113)	44	40	498
H(121)	213	92	362
H(122)	202	88	444
H(123)	215	-49	392
H(211)	218	494	282
H(212)	202	325	247
H(213)	230	463	193
H(221)	114	542	78
H(222)	106	335	100
H(223)	50	440	42

terminated when the maximum shift in any parameter was $<0.1\sigma$. The final cycle included 961 reflexions. The final R , based on 964 reflexions, was 0.093 and $R' = (\sum w\Delta^2 / \sum w|F_o|^2)^{1/2}$ was 0.120. A final difference synthesis showed the maximum positive electron densities of $0.7 e \text{ \AA}^{-3}$ to be located about the Ni atoms.

Final atomic coordinates are given in Table 1.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32632 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

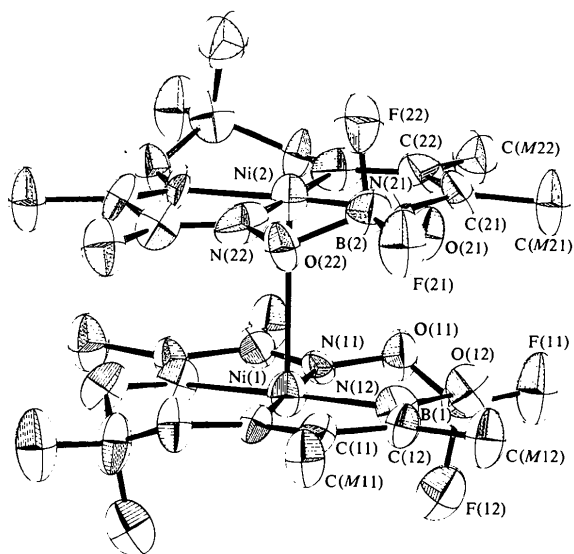


Fig. 1. A perspective drawing of the dimeric molecular unit and the labelling of the atoms. Thermal ellipsoids are scaled to include 35% probability.

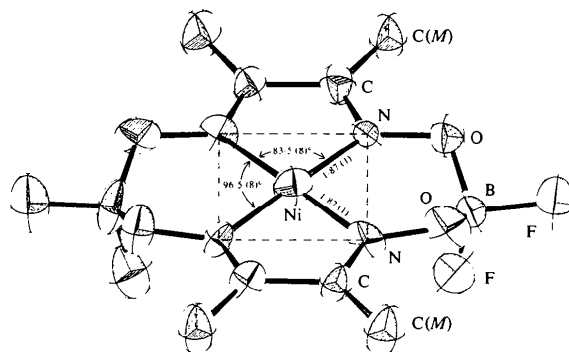


Fig. 2. A perspective drawing of the formula unit showing the square-planar nickel coordination. Thermal ellipsoids are scaled to include 35% probability.

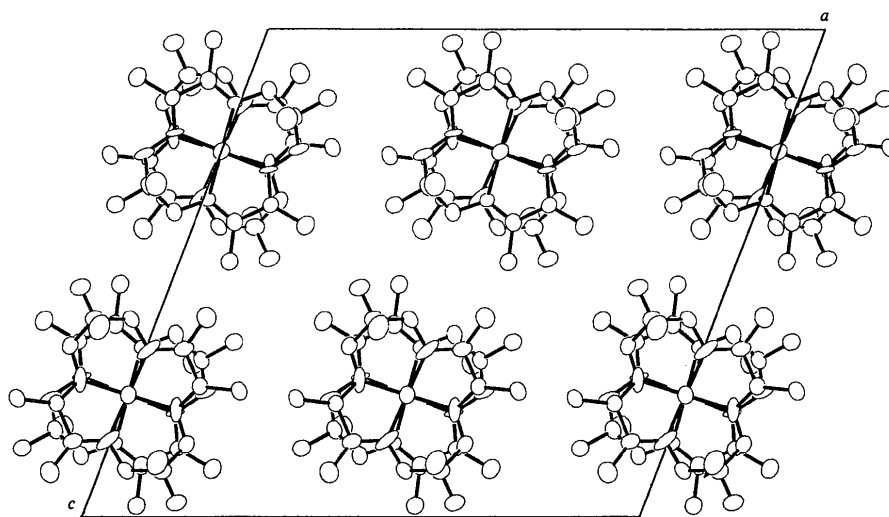


Fig. 3. The packing of the molecules in the crystal.

Discussion

In the original report of the isolation of the title complex, Schrauzer (1962), employing X-ray powder photography and assuming an orthorhombic space group, concluded that the compound contained no Ni–Ni bonding. This present work shows the assumption and conclusion to be incorrect.

The structure of the complex consists of molecular dimers on which the space group imposes 2 (C_2) symmetry. The dimeric unit with the labelling of the atoms is shown in Fig. 1, and the molecular geometry (Johnson, 1965) in Fig. 2. The packing is given by the (010) projection (Fig. 3). The dimethylglyoxime residues and the Ni atom in each formula unit are coplanar [planes (1) and (2), Table 2]; the BF_2 groups are displaced out of the planes and away from the other formula moiety. Each formula unit is rotated some 92°

Table 2. *Least-squares planes and their equations given by $lX' + mY' + nZ' - p = 0$ where X' , Y' and Z' are orthogonal coordinates related to X , Y and Z by $X' = X \sin \beta$, $Y' = Y$, $Z' = Z + X \cos \beta$*

Deviations (Å) of the most relevant atoms from the planes are given in square brackets. Atoms marked with a prime are related to those at x, y, z by the twofold axis at $0, y, \frac{1}{4}$.

	<i>l</i>	<i>m</i>	<i>n</i>	<i>p</i>
Plane (1): Ni(1), N(11'), N(12), C(11), C(12) [Ni(1) 0.03, N(11') -0.04, N(12) -0.03, C(11) 0.03, C(12) 0.01, C(M11) -0.01, C(M12) 0.01, O(11') 0.06, O(12) 0.10, O(11) 0.13, B(1) -0.48]	-0.0135	0.9998	-0.0160	0.2043
Plane (2): Ni(2), N(21), N(22'), C(21), C(22) [Ni(2) 0.01, N(21) -0.03, N(22') 0.01, C(21) 0.04, C(22) -0.03, C(M21) -0.05, C(M22) -0.08, O(22') -0.05, O(21) -0.05, O(22) -0.02, B(2) 0.56]	0.0039	1.0000	0.0036	3.5307
Plane (3): Ni(1), N(11), N(12), N(11'), N(12') [Ni(1) 0.025, N(11)N(11')-0.010, N(12)N(12')-0.003]	0.0000	1.0000	0.0000	0.2866
Plane (4): Ni(2), N(21), N(22), N(21'), N(22') [Ni(2) 0.010, N(21)N(21') -0.029, N(22)N(22') 0.024]	0.0000	1.0000	0.0000	3.5085
Plane (5): N(11'), O(11'), Ni(1), C(11) [N(11') -0.06; O(11'), Ni(1), C(11) 0.02]	0.0000	0.9998	-0.0213	0.1919
Plane (6): N(12), O(12), Ni(1), C(12) [N(12) -0.05, O(12) 0.02, Ni(1) 0.01, C(12) 0.02]	-0.0217	0.9995	0.0233	0.4112
Plane (7): C(11), N(11'), C(12), C(M11) [C(11) 0.03; N(11'), C(12), C(M11), -0.01]	-0.0327	0.9993	-0.0170	0.1670
Plane (8): C(12), N(12), C(11), C(M12) [C(12) 0.002; N(12), C(11), C(M12) -0.001]	-0.0139	0.9991	-0.0391	0.0663
Plane (9): N(21), O(21), Ni(2), C(21) [N(21) -0.018, O(21) 0.007, Ni(2) 0.005, C(21) 0.007]	0.0110	0.9991	0.0400	3.7061
Plane (10): N(22'), O(22'), Ni(2), C(22) [N(22') 0.03, O(22'), Ni(2), C(22) -0.01]	-0.0014	0.9999	-0.0129	3.4634
Plane (11): C(21), N(21), C(22), C(M21) [C(21) 0.05, N(21) -0.02, C(22) -0.01, C(M21) -0.02]	0.0122	0.9999	-0.0015	3.5146
Plane (12): C(22), N(22'), C(21), C(M22) [C(22) 0.011, N(22') 0.004, C(21) 0.003, C(M22) 0.004]	0.0062	0.9990	-0.0441	3.3920

Table 3. *Bond lengths and angles with estimated standard deviations in parentheses*

Atoms marked with a prime are related to those at x, y, z by the two-fold axis at $0, y, \frac{1}{4}$.

(a) Distances (Å)

	Ni(1)···Ni(2)	3.207 (6)	
Ni(1)—N(11)	1.87 (2)	Ni(2)—N(21)	1.87 (1)
Ni(1)—N(12)	1.87 (1)	Ni(2)—N(22)	1.85 (2)
N(11)—O(11)	1.36 (2)	N(21)—O(21)	1.34 (2)
N(12)—O(12)	1.38 (2)	N(22)—O(22)	1.32 (2)
N(11)—C(11')	1.29 (3)	N(21)—C(21)	1.25 (3)
N(12)—C(12)	1.31 (2)	N(22)—C(22')	1.31 (3)
C(11)—C(12)	1.49 (3)	C(21)—C(22)	1.54 (3)
C(11)—C(M11)	1.51 (3)	C(21)—C(M21)	1.48 (3)
C(12)—C(M12)	1.52 (2)	C(22)—C(M22)	1.45 (3)
B(1)—O(11)	1.48 (3)	B(2)—O(21)	1.48 (3)
B(1)—O(12)	1.37 (3)	B(2)—O(22)	1.47 (3)
B(1)—F(11)	1.41 (3)	B(2)—F(21)	1.33 (3)
B(1)—F(12)	1.42 (3)	B(2)—F(22)	1.40 (3)

Table 3 (cont.)

(b) Angles (°)			
N(11)—Ni(1)—Ni(2)	91.1 (6)	N(21)—Ni(2)—Ni(1)	88.8 (5)
N(12)—Ni(1)—Ni(2)	90.9 (6)	N(22)—Ni(2)—Ni(1)	90.4 (5)
N(11)—Ni(1)—N(11')	177.9 (6)	N(21)—Ni(2)—N(21')	177.6 (5)
N(12)—Ni(1)—N(12')	178.3 (6)	N(22)—Ni(2)—N(22')	179.1 (5)
N(11)—Ni(1)—N(12)	96.3 (6)	N(21)—Ni(2)—N(22)	83.3 (9)
N(11)—Ni(1)—N(12')	83.7 (6)	N(21)—Ni(2)—N(22')	96.7 (9)
Ni(1)—N(11)—O(11)	125 (1)	Ni(2)—N(21)—O(21)	124 (1)
Ni(1)—N(11)—C(11')	115 (1)	Ni(2)—N(21)—C(21)	118 (2)
O(11)—N(11)—C(11')	119 (1)	O(21)—N(21)—C(21)	119 (1)
Ni(1)—N(12)—O(12)	124 (1)	Ni(2)—N(22)—O(22)	126 (1)
Ni(1)—N(12)—C(12)	115 (1)	Ni(2)—N(22)—C(22')	116 (1)
O(12)—N(12)—C(12)	120 (1)	O(22)—N(22)—C(22')	118 (1)
N(11)—O(11)—B(1)	111 (2)	N(21)—O(21)—B(2)	117 (1)
N(12)—O(12)—B(1)	112 (1)	N(22)—O(22)—B(2)	116 (2)
N(11')—C(11)—C(12)	113 (1)	N(21)—C(21)—C(22)	112 (2)
N(11')—C(11)—C(M11)	124 (1)	N(21)—C(21)—C(M21)	128 (2)
C(12)—C(11)—C(M11)	123 (1)	C(22)—C(21)—C(M21)	120 (2)
N(12)—C(12)—C(11)	112 (1)	N(22')—C(22)—C(21)	111 (1)
N(12)—C(12)—C(M12)	124 (2)	N(22')—C(22)—C(M22)	128 (2)
C(11)—C(12)—C(M12)	124 (2)	C(21)—C(22)—C(M22)	121 (2)
O(11)—B(1)—O(12)	118 (2)	O(21)—B(2)—O(22)	114 (2)
F(11)—B(1)—F(12)	107 (2)	F(21)—B(2)—F(22)	112 (2)
O(11)—B(1)—F(11)	102 (2)	O(21)—B(2)—F(21)	107 (2)
O(11)—B(1)—F(12)	107 (2)	O(21)—B(2)—F(22)	109 (2)
O(12)—B(1)—F(11)	107 (2)	O(22)—B(2)—F(21)	109 (2)
O(12)—B(1)—F(12)	114 (2)	O(22)—B(2)—F(22)	106 (2)

Table 4. Average molecular dimensions in dioxime structures: distances in Å and angles in degrees

Compound*	gH ₂		dmgH ₂		Ni(gH ₂)		Ni(dmgH ₂)	Ni(emgH ₂)	Ni(bcdH ₂)	Ni(bcdH ₂) 0.5I	Ni(dmg- BF ₂) ₂
	(1)	(2)	(3)†	(4)	(5)	(6)	(7)	(8)	(9)	(10)	
Ni—N				1.874 (4)	1.869 (4)	1.85 (1)	1.862 (4)	1.868 (4)	1.90 (2)	1.87 (1)	
N—O	1.397 (5)	1.38 (‡)	1.312 (11)	1.346 (5)	1.341 (11)	1.35 (2)	1.347 (7)	1.316 (7)	1.33 (3)	1.35 (2)	
N—C	1.284 (5)	1.27 (‡)	1.253 (11)	1.290 (7)	1.300 (15)	1.29 (3)	1.298 (6)	1.311 (8)	1.31 (4)	1.29 (3)	
C—C	1.449 (6)	1.44 (‡)	1.562 (18)	1.435 (7)	1.438 (15)	1.54 (3)	1.487 (8)	1.445 (8)	1.50 (4)	1.51 (3)	
C—R		1.53 (‡)	1.479 (15)			1.51 (3)	1.503 (7)	1.426 (8)	1.43 (4)	1.49 (3)	
O...O				2.453 (6)	2.445 (13)	2.40 (2)	2.454 (5)	2.480 (6)	2.43 (3)	2.46 (2)	
N—Ni—N				82.2 (2)	82.4 (4)	83.1 (‡)	82.5 (2)	83.6 (2)	84 (1)	83.5 (8)	
Ni—N—O				123.2 (3)	123.3 (8)	122.9 (‡)	123.9 (3)	125.3 (4)	124 (2)	124 (1)	
Ni—N—C				115.3 (3)	115.6 (7)	117.4 (‡)	116.2 (3)	‡	114 (2)	116 (1)	
O—N—C	111.1 (3)	113.9 (‡)	111.4 (9)	121.4 (4)	121.0 (9)	119.7 (‡)	119.8 (4)	‡	‡	119 (1)	
N—C—C	117.7 (3)	115.1 (‡)	113.8 (9)	113.5 (5)	113.1 (9)	111.5 (‡)	112.4 (4)	113.4 (5)	114 (2)	112 (1)	
N—C—R		124.6 (‡)	126.0 (10)			124.5 (‡)	123.3 (5)	127.2 (6)	126 (3)	126 (2)	
C—C—R		120.4 (‡)	120.0 (9)			124.0 (‡)	124.2 (5)	119.4 (6)	120 (3)	121 (2)	

References: (1) Calleri, Ferraris & Viterbo (1966). (2) Merritt & Lanterman (1952). (3) Hamilton (1961). (4) Calleri, Ferraris & Viterbo (1967). (5) Murmann & Schlemper (1967). (6) Williams, Wohler & Rundle (1959). (7) Bowers, Banks & Jacobson (1972). (8) Leichert & Weiss (1975). (9) Endres, Keller, Moroni & Weiss (1975). (10) This work.

* gH₂ = glyoxime; dmgH₂ = dimethylglyoxime; emgH₂ = ethylmethylglyoxime; bcdH₂ = 1,2-benzoquinone dioxime.

† Neutron diffraction study.

‡ Data not given.

relative to the other around the Ni—Ni direction. The molecules are thus packed so that the Ni atoms are aligned along **b** with a Ni...Ni separation between dimers of 4.653 (9) Å and within each dimer of 3.207 (6) Å. This latter value represents a weak but significant interaction (Thomas & Underhill, 1972; Banks & Barnum, 1958).

The bond distances and angles are given in Table 3. The environment about each Ni atom is distorted

square-planar [planes (3) and (4), Table 2] with average Ni—N 1.87 (1) Å and N—Ni—N = 83.5 (8) and 96.5 (8)°. There is no significant difference in the molecular geometry of each formula unit. The average dimensions in the complex are given in Table 4, where they are compared with those of other glyoxime structures together with two recently reported Ni complexes of 1,2-benzoquinone dioxime. It may be seen that in all the Ni—glyoxime structures the coordination

about the metal atoms is very similar, with average Ni-N = 1.86 (1) Å and average N-Ni-N = 82.7 (6)°. In addition, substitution by the BF₂ group for the proton yields no significant change from the average O...O interatomic distance of 2.44 (3) Å. The molecular geometry is unaffected by the BF₂ substitution, and the packing is very similar to that in bis(dimethylglyoximato)nickel(II) (Williams, Wohlauer & Rundle, 1959). A major difference between the two structures, however, is the severing of the Ni...Ni chain of the parent (Ni...Ni, 3.25 Å) into dimers with a 0.04 Å decrease in the Ni-Ni distance. The Ni...Ni distances in nine other substituted glyoxime Ni^{II} chelates (Banks & Barnum, 1958) range from 3.2 to 3.6 Å, although each displays the metal-metal chain typical of glyoximate chelates of the Group VIIIb metals which is thought to be the reason for their semi-conducting properties (Thomas & Underhill, 1969, 1972; Underhill, Watkins & Pethig, 1973). Indeed the steric requirements of the bulky BF₂ groups in the title compound would not allow a chain-like structure to form, although a close association of pairs of molecular units is maintained with the two displaced BF₂ groups of each unit adopting a *cis* configuration to the molecular plane.

The N and C atoms of the glyoxime residues are trigonally planar [planes (5)-(12), Table 2] and the angles subtended at the B atoms, average 109 (2)°, indicate their expected tetrahedral covalency. The average N-O-B angle of 114 (2)° is slightly larger than the regular tetrahedral angle, but this may be a consequence of the nature of the molecular stacking.

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