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Studies on Macrocyclic Complexes Derived from *vic*-Dioximes.
X. The Crystal Structure of the 1:1 Adduct of Benzylamine with Bis(difluoroborondimethylglyoximato)nickel(II)

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The 1:1 adduct of benzylamine with bis(difluoroborondimethylglyoximato)nickel(II) is orthorhombic, space group Pbcn, *with* a = *19.563(12),* b = *10.087(7),* c = *21.719(11) Å, Z = 8. The structure was refined to* R = *0.077 for 1661 photographic reflexions. The complex is dimeric [Ni···Ni 3.873(3) Å] with the two monomer units related by a crystallographic two-fold axis. Each Ni atom has a square-based pyramidal environment formed from four N atoms of the closely planar macrocycle [average, Ni–N 1.859(9) Å, N–Ni–N 82.3(4) and 96.0(4)°] and the benzylamine N atom [Ni–N 2.286(11) Å]. The Ni atom is displaced 0.23 Å out of the N_4 equatorial plane towards the apical N atom. The perpendicular distance between the two macrocyclic planes of the dimer is 3.30 Å and the C···O intermolecular contacts in the dimer are 3.24–3.37 Å.*

Introduction

Bis(difluoroborondimethylglyoximato) nickel(II), $Ni(dmgBF_2)_2$, forms brightly coloured diamagnetic 1:1 adducts with monodentate bases [1]. Aniline forms such an adduct and its crystal structure [2] shows the dimeric nature of the parent [3] to be retained on coordination of the base in axial positions. The phenyl rings of the ligands are oriented with respect to the macrocyclic moieties to suggest the presence of π-bonding between these groups. Such an interaction has been proposed for related cobaloximes [4, 5].

The ability of the planar gloximate residue to bond in this manner with suitable aromatic molecules has been demonstrated in the structures of the π-sandwich complexes with benzimidazole [6] and anthracene [7].

Like aniline, benzylamine has the potential to both coordinate and to π-bond to the nickel macrocycle. Its 1:1 adduct was prepared and its crystal structure undertaken to investigate the mode of interaction of the base.

Experimental

Crystal Data

$C_{15}H_{21}B_2F_4N_5O_4Ni$, $M_r = 491.7$, Orthorhombic, $a = 19.563(12)$, $b = 10.087(7)$, $c = 21.719(11)$ Å, $U = 4285.9$ Å^3, $Z = 8$, $D_c = 1.524$ Mg m^{-3}, F(000) = 2016, μ(Cu-K_α) = 1.88 mm^{-1}. Systematic absences: $0kl$ if $k \neq 2n$, $h0l$ if $l \neq 2n$ and $hk0$ if $h + k \neq 2n$; space group *Pbcn* (No. 60).

The complex crystallises as deep-red needles with *b* parallel to the needle direction. Cell parameters were determined from precession photographs using Mo-K_α radiation. Intensities were estimated visually from equi-inclination Weissenberg photographs using Cu-K_α radiation for the layers 0–6 about [100] and [010]. They were corrected for Lorentz and polarisation effects but not for absorption or extinction. The structure factors were placed on a common scale by internal correlation and 1661 non-zero unique reflexions were obtained. Scattering factors were taken from 'International Tables for X-Ray Crystallography' [8]. All calculations were carried out on a UNIVAC 1106 computer with programmes written by F.S.S..

The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations in which the function minimised was $\Sigma w\Delta^2$. The weight for each reflexion, initially unity, was finally given by $w = (4.4 + 0.16|F_o| + 0.0016|F_o|^2)^{-1}$. When the maximum shift in any parameter was of the order of its estimated standard deviation a Fourier difference map was calculated. This map showed the approximate positions of all the hydrogen atoms. The positions of these atoms were optimised assuming C–H and N–H to be 1.0 Å and they were included in subsequent calculations but at no time were their parameters refined. Refinement was

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*Part IX: F. S. Stephens and R. S. Vagg, *Inorg. Chim. Acta, 52,* 245 (1981).

continued with anisotropic thermal parameters for all non-hydrogen atoms and terminated when the maximum shift in any parameter was $<0.1\sigma$. The final value for R was 0.077 and for $R' \{=(\Sigma w\Delta^2/\Sigma w|F_o|^2)^{1/2}\}$ was 0.105. A final Fourier difference map showed no features $>|0.6|e\text{Å}^{-3}$. Final atomic parameters are given in Tables I–III. A list of observed and calculated structure factors has been deposited with the Editor.

TABLE I. Final Atomic Coordinates (fractional $\times 10^4$) for Non-hydrogen Atoms with Estimated Standard Deviations in Parentheses.

	x	*y*	*z*
Ni	809.4(8)	497.4(15)	1986.9(6)
N(11)	659(4)	2304(8)	1966(4)
N(12)	143(4)	527(10)	1372(3)
N(21)	1285(4)	514(10)	2715(3)
N(22)	776(5)	−1345(9)	2137(4)
N	1698(6)	607(12)	1306(6)
C(11)	221(6)	2774(12)	1561(5)
C(12)	−72(6)	1664(13)	1200(4)
C(21)	1410(5)	−727(14)	2965(5)
C(22)	1113(5)	−1767(10)	2608(5)
C(M11)	37(7)	4176(12)	1467(6)
C(M12)	−573(7)	1894(13)	671(5)
C(M21)	1794(6)	−852(13)	3571(5)
C(M22)	1194(7)	−3189(13)	2785(6)
O(11)	941(4)	3222(7)	2349(4)
O(12)	−122(4)	−593(8)	1099(3)
O(21)	1492(4)	1565(9)	3035(3)
O(22)	446(4)	−2239(7)	1770(3)
B(1)	1575(9)	2817(17)	2665(7)
B(2)	305(7)	−1814(17)	1118(6)
F(11)	2083(4)	2618(7)	2236(4)
F(12)	1704(5)	3789(8)	3083(4)
F(21)	889(3)	−1636(7)	801(3)
F(22)	−91(3)	−2804(6)	879(3)
C	2202(9)	−242(18)	1405(8)
C(1)	2680(9)	−357(21)	784(10)
C(2)	3278(9)	301(20)	723(8)
C(3)	3714(8)	39(21)	256(10)
C(4)	3521(10)	−897(25)	−167(9)
C(5)	2921(12)	−1484(26)	−193(10)
C(6)	2456(10)	−1141(22)	293(11)

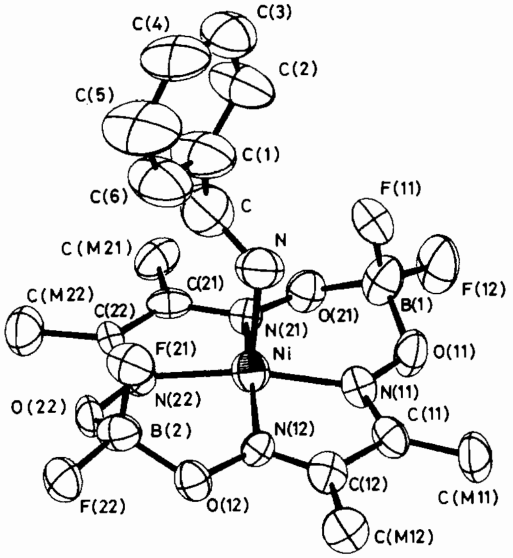

Fig. 1. A perspective drawing of the Ni(dmgBF$_2$)$_2$(NH$_2$CH$_2$-C$_6$H$_5$) moiety with the atom labelling scheme. Thermal ellipsoids are scaled to include 35% probability.

TABLE II. Final Anisotropic Thermal Parameters ($\times 10^4$) in the Form $\exp\text{-}(h^2b_{11} + k^2b_{22} + l^2b_{33} + 2hkb_{12} + 2hlb_{13} + 2klb_{23})$ with Estimated Standard Deviations in Parentheses.

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
Ni	39.5(5)	122.1(22)	26.3(4)	−1.4(9)	−0.1(4)	−2.2(7)
N(11)	39(3)	105(11)	15(2)	−9(5)	−2(2)	−1(4)
N(12)	28(3)	94(11)	20(2)	9(5)	0(2)	−4(4)
N(21)	30(2)	93(11)	19(2)	2(5)	0(2)	−1(5)
N(22)	33(3)	99(11)	28(2)	3(5)	8(2)	9(5)
N	37(3)	195(18)	53(4)	−4(7)	3(3)	−27(7)
C(11)	39(4)	111(16)	23(3)	−13(7)	1(3)	−17(5)
C(12)	41(4)	137(16)	19(2)	0(8)	9(3)	10(6)
C(21)	21(3)	174(21)	23(2)	3(6)	7(2)	−3(7)
C(22)	27(3)	63(12)	34(3)	−7(6)	−1(3)	6(6)
C(M11)	57(5)	100(16)	42(4)	−7(8)	−8(4)	16(6)
C(M12)	48(4)	182(20)	27(3)	−3(8)	−7(3)	6(6)
C(M21)	39(4)	178(19)	30(3)	24(7)	1(3)	16(6)
C(M22)	56(5)	129(19)	45(4)	7(8)	6(4)	19(7)
O(11)	51(3)	93(10)	41(2)	14(5)	−13(2)	−15(4)
O(12)	38(2)	117(10)	25(2)	−9(5)	−4(2)	−4(4)
O(21)	40(3)	132(11)	26(2)	7(5)	−5(2)	1(4)

(continued on facing page)

TABLE II. *(continued)*

O(22)	39(2)	66(8)	33(2)	−7(4)	−4(2)	−3(4)
B(1)	54(6)	142(24)	28(4)	19(10)	−9(4)	−17(8)
B(2)	30(4)	166(24)	31(4)	−2(9)	−2(3)	−24(8)
F(11)	48(3)	143(10)	47(2)	−25(4)	−1(2)	−1(4)
F(12)	77(4)	146(11)	61(3)	6(5)	−33(3)	−38(5)
F(21)	41(2)	188(11)	34(2)	−9(4)	8(2)	−35(4)
F(22)	46(2)	124(9)	43(2)	−9(4)	−10(2)	−23(4)
C	62(7)	245(30)	53(6)	−13(12)	−12(5)	25(10)
C(1)	50(6)	345(40)	68(7)	−12(13)	16(5)	−74(14)
C(2)	50(6)	350(38)	56(6)	−51(12)	9(5)	−48(12)
C(3)	49(6)	272(31)	56(6)	−10(11)	13(5)	18(11)
C(4)	46(6)	411(45)	52(6)	11(13)	22(5)	−28(13)
C(5)	69(9)	515(56)	79(9)	2(18)	33(7)	−120(18)
C(6)	57(7)	336(37)	87(8)	−27(13)	31(7)	−68(16)

Discussion

The crystal structure consists of molecular dimers on which the space group imposes $2(C_2)$ symmetry relating the two $[Ni(dmgBF_2)_2(NH_2CH_2C_6H_5)]$ monomer units. Figure 1 shows a perspective drawing [9] of the monomer unit together with the atom labelling scheme. The bond lengths and angles are given in Table IV. The packing of the molecular dimers is shown in Fig. 2 and closest intramolecular contacts are listed in Table Va.

The dimeric nature of the planar parent $Ni(dmgBF_2)_2$ [3] is retained upon reaction with benzylamine which coordinates to each nickel atom in an axial position forming a square-based pyramidal arrangement. The molecular dimensions within the $[Ni(dmgBF_2)_2]_2$ unit are in accord with those in the parent [3] with the exception that the Ni···Ni separation has increased to 3.873(3) Å and that each nickel atom is displaced by 0.23 Å from its N_4-plane (Table VI, plane 1) towards the apical N atom. A comparison of structural data for N-base adducts of $Ni(dmgBF_2)_2$ is given in Table VII.

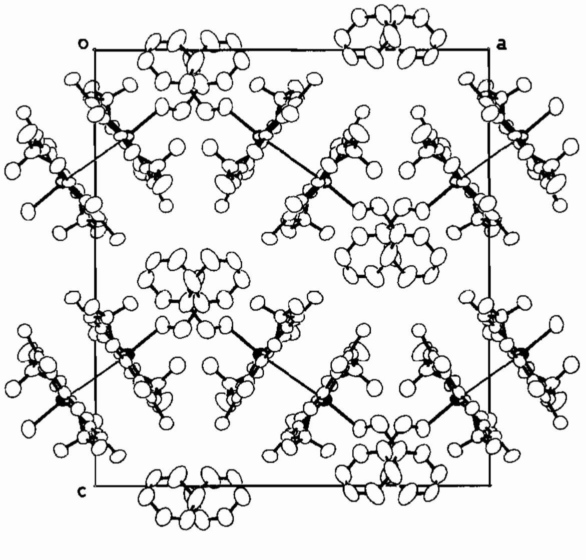

Fig. 2. The packing of the dimer units in the unit cell.

TABLE III. Hydrogen Atomic Parameters (Coordinates: fractional, $\times 10^3$)[a].

	x	y	z
H(111)	−38	438	172
H(112)	42	478	158
H(113)	−8	435	101
H(121)	−77	283	73
H(122)	−96	126	69
H(123)	−34	185	27
H(211)	206	0	360
H(212)	147	−90	393
H(213)	210	−163	359
H(221)	115	−363	237
H(222)	165	−340	299
H(223)	82	−351	308
H(N1)	188	155	132
H(N2)	150	47	88
H(C1)	248	8	177
H(C2)	198	−112	154
H(2)	340	98	104
H(3)	417	52	20
H(4)	386	−114	−48
H(5)	281	−218	−49
H(6)	195	−123	26

[a]For all H atoms $B = 7.0$ Å^2.

The bis-glyoximato fragment is closely planar (Table VI, plane 2) and the perpendicular distance between the planes of these two macrocyclic fragments in the dimer is 3.30 Å. This distance is similar to that found in related compounds in which the $[Ni(dmgBF_2)_2]_2$ dimer unit is present [2, 3, 6, 10] and together with the close interatomic contacts between the moieties, listed in Table Vb, is indicative of a π–π interaction between macrocyclic moieties.

TABLE IV. Bond Lengths and Angles with Estimated Standard Deviations in Parentheses.

a) Distances (Å)[a]			
	Ni···Ni^{I}	3.873(3)	
Ni–N	2.286(11)		
Ni–N(11)	1.846(9)	Ni–N(21)	1.835(8)
Ni–N(12)	1.867(8)	Ni–N(22)	1.888(9)
N(11)–C(11)	1.317(13)	N(21)–C(21)	1.386(13)
N(12)–C(12)	1.277(12)	N(22)–C(22)	1.288(12)
N(11)–O(11)	1.362(10)	N(21)–O(21)	1.331(10)
N(12)–O(12)	1.376(10)	N(22)–O(22)	1.366(11)
C(11)–C(12)	1.482(15)	C(21)–C(22)	1.428(14)
C(11)–C(M11)	1.474(15)	C(21)–C(M21)	1.521(14)
C(12)–C(M12)	1.528(15)	C(22)–C(M22)	1.493(15)
B(1)–O(11)	1.475(16)	B(2)–O(12)	1.488(16)
B(1)–O(21)	1.507(18)	B(2)–O(22)	1.505(15)
B(1)–F(11)	1.376(17)	B(2)–F(21)	1.344(14)
B(1)–F(12)	1.360(16)	B(2)–F(22)	1.367(15)
N–C	1.323(18)	C(3)–C(4)	1.369(23)
C–C(1)	1.647(24)	C(4)–C(5)	1.316(26)
C(1)–C(2)	1.351(22)	C(5)–C(6)	1.435(22)
C(2)–C(3)	1.352(22)	C(6)–C(1)	1.398(23)
b) Angles (°)[a]			
N(11)–Ni–N(12)	81.7(4)	N(21)–Ni–N(22)	82.9(4)
N(11)–Ni–N(21)	95.3(4)	N(12)–Ni–N(22)	96.6(4)
N(11)–Ni–N(22)	166.0(4)	N(12)–Ni–N(21)	166.1(3)
N(11)–Ni–N	93.3(4)	N(21)–Ni–N	99.9(4)
N(12)–Ni–N	93.9(4)	N(22)–Ni–N	100.7(4)
Ni–N(11)–C(11)	118.4(7)	Ni–N(21)–C(21)	114.8(7)
Ni–N(11)–O(11)	126.3(7)	Ni–N(21)–O(21)	127.7(7)
C(11)–N(11)–O(11)	115.3(9)	C(21)–N(21)–O(21)	117.4(8)
Ni–N(12)–C(12)	116.9(8)	Ni–N(22)–C(22)	116.4(8)
Ni–N(12)–O(12)	123.9(7)	Ni–N(22)–O(22)	124.4(7)
C(12)–N(12)–O(12)	119.2(9)	C(22)–N(22)–O(22)	119.2(9)
N(11)–C(11)–C(12)	109.5(10)	N(21)–C(21)–C(22)	112.3(10)
N(11)–C(11)–C(M11)	126.7(10)	N(21)–C(21)–C(M21)	120.1(10)
C(12)–C(11)–C(M11)	123.9(11)	C(22)–C(21)–C(M21)	127.6(12)
N(12)–C(12)–C(11)	113.4(11)	N(22)–C(22)–C(21)	113.3(10)
N(12)–C(12)–C(M12)	124.5(11)	N(22)–C(22)–C(M22)	125.1(10)
C(11)–C(12)–C(M12)	122.1(11)	C(21)–C(22)–C(M22)	121.6(11)
N(11)–O(11)–B(1)	115.9(9)	N(21)–O(21)–B(1)	114.9(8)
N(12)–O(12)–B(2)	117.2(8)	N(22)–O(22)–B(2)	116.7(8)
O(11)–B(1)–O(21)	113.0(13)	O(12)–B(2)–O(22)	111.4(10)
O(11)–B(1)–F(11)	109.4(10)	O(12)–B(2)–F(21)	110.6(12)
O(11)–B(1)–F(12)	105.4(12)	O(12)–B(2)–F(22)	106.0(10)
O(21)–B(1)–F(11)	108.5(12)	O(22)–B(2)–F(21)	111.3(10)
O(21)–B(1)–F(12)	105.6(11)	O(22)–B(2)–F(22)	104.7(12)
F(11)–B(1)–F(12)	115.0(14)	F(21)–B(2)–F(22)	112.6(11)
Ni–N–C	115.4(12)	C(1)–C(2)–C(3)	121.5(17)
N–C–C(1)	109.5(14)	C(2)–C(3)–C(4)	117.7(15)
C–C(1)–C(2)	122.5(17)	C(3)–C(4)–C(5)	125.7(16)
C–C(1)–C(6)	119.1(15)	C(4)–C(5)–C(6)	115.2(18)
C(6)–C(1)–C(2)	118.4(17)	C(5)–C(6)–C(1)	119.9(17)

[a] Roman numeral superscript I refers to the equivalent position −*x*, *y*, ½ − *z*, relative to that at *x*, *y*, *z*.

Similar to other N-base adducts [2, 10] the macrocycle has a *cis* conformation of the BF_2 groups in which the separation of the axial F atoms is increased by $\sim$0.3 Å from that observed in the parent [3] to accommodate the axial coordination of the base. The molecular geometry of the present complex closely parallels that of the 4,4′-bipyridine adduct [10] but is markedly in contrast with that of the aniline

TABLE V. Intra- and Inter-Dimer Contact Distances^a $\leqslant 3.5$ Å with Estimated Standard Deviations in Parentheses.

(a) Intra-dimer Distances (Å)

F(12)····C(M21^{II})	3.14(1)	F(11)····C^{II}	3.14(2)
F(12)····C(M22^{III})	3.27(2)	F(22)····C(4^{VI})	3.39(2)
F(21)····C(M12^{IV})	3.27(1)	F(21)····C(2^{VII})	3.50(2)
F(22)···C(M11^{V})	3.31(1)		

b) Inter-dimer Distances (Å)

C(12)···O(21^{I})	3.24(1)	C(M11)···O(11^{I})	3.35(2)	N(22)···N(22^{I})	3.42(1)
C(21)···O(12^{I})	3.24(1)	C(M12)···O(21^{I})	3.35(1)	N(12)···N(21^{I})	3.43(1)
C(11)···O(11^{I})	3.31(1)	C(M21)···O(12^{I})	3.36(1)	N(11)···N(11^{I})	3.47(1)
C(22)···O(22^{I})	3.37(1)	C(M22)···O(22^{I})	3.49(2)	N(22)···O(22^{I})	3.49(1)

^{a}For Roman numeral superscripts see footnote Table IV, and II $\frac{1}{2} - x, \frac{1}{2} + y, z$; III $x, 1 + y, z$; IV $-x, -y, -z$; V $x, y - 1, z$; VI $x - \frac{1}{2}, -\frac{1}{2} - y, -z$; VII $\frac{1}{2} - x, y - \frac{1}{2}, z$.

TABLE VI. Least-squares Planes Data.

(a) Least-squares planes and their equations given by $lX + mY + nZ - p = 0$. Deviations (Å) of relevant atoms from the planes are given in square brackets.

	l	m	n	p
Plane (1): N(11), N(12), N(22), N(21) [N(11), N(22) 0.003; N(12), N(21) −0.003; Ni −0.23; N −2.50]	−0.7937	0.0118	0.6081	1.5990
Plane (2): O(11), N(11), C(11), C(12), N(12), O(12), O(21), N (21), C(21), C(22), N(22), O(22) [O(11), O(22) −0.01; N(11) −0.08, C(11) 0.08; C(12), C(21) 0.06; N(12) −0.05; O(12) < |0.01|; O(21) 0.02; N(21) −0.07; C(22) 0.05; N(22) −0.04; Ni −0.29; N −2.57]	−0.7971	0.0013	0.6038	1.6306
Plane (3): C(1)–C(6) [C(1) 0.077; C(2) −0.031, C(3) −0.032; C(4) 0.047; C(5) 0.001; C(6) −0.062; C 0.33; N −0.82; Ni −0.64]	0.3944	−0.7528	0.5270	3.1596
Plane (4): B(1), F(11), F(12)	0.4313	−0.6701	0.6041	2.9209
Plane (5): B(2), F(21), F(22)	0.4596	−0.6637	0.5901	2.9213
Plane (6): N(11), C(11), C(12), N(12) [N(11) −0.006; C(11) 0.010; C(12) −0.011; N(12) 0.007; Ni −0.07; C(M11) 0.04; C(M12) −0.07; O(11), O(12) 0.07]	−0.7421	−0.0697	0.6666	1.7350
Plane (7): N(21), C(21), C(22), N(22) [N(21) −0.005; C(21) 0.008; C(22) −0.008; N(22) 0.005; Ni −0.10; C(M21) 0.10; C(M22) −0.03; O(21) 0.11; O(22) 0.04]	−0.8313	0.0675	0.5517	1.2019

(b) Dihedral angles (°) between relevant planes.

2:3	89.8	1:4	89.0	6:7	11.5
4:3	6.8	1:5	90.8		
5:3	7.3	4:5	89.7		

adduct [2]. In particular the plane of the phenyl ring (Table VI, plane 3) is perpendicular to the macrocyclic plane (plane 2) and has dihedral angles of 7° with each of the BF_2 planes (planes 4 and 5). Thus in the present stucture the phenyl ring does not adopt a position in which a π-interaction with a macrocyclic group is possible. The longer Ni–N(*amine*) bonds observed in the aniline adduct [2], their inclined nature, and the change to an M–L–M stacking arrangement [11], must each occur so as to accommodate π-bonding between the phenyl ring of the aniline and the macrocycle. In contrast the intermolecular

TABLE VII. Comparison of Structural Data for N-base Adducts of $Ni(dmgBF_2)_2$. Distances are in Å.

	$[Ni(dmgBF_2)_2]_2$	$[Ni(dmgBF_2)_2(NH_2C_6H_5)]_2$	$\{[Ni(dmgBF_2)_2]_2(4,4'\text{-bipy})\}$	$[Ni(dmgBF_2)_2(NH_2CH_2C_6H_5)]_2$
Ref.	[3]	[2]	[10]	[present work]
$Ni\cdots Ni$	3.207(6)	3.654(2)	3.909(3)	3.873(3)
$Ni-N_{eq}$[a]	1.87(1)	1.871(1)	1.85(1)	1.859(9)
$Ni-N_{ax}$[a]		2.724(9), 2.602(10)	2.35(1)	2.286(11)
ΔNi[b]	0.03, 0.01	0.12, 0.14	0.20	0.23
$F_{ax}\cdots F_{ax}$	5.46, 5.50	5.76, 5.80	5.92	5.79
$N_{ax}\cdots F_{ax}$		3.05–3.08	2.96	2.96, 2.97
$C\cdots O$	3.13–3.17	3.15–3.25	3.24–3.34	3.24–3.37
Dihedral Angle NCCN/NCCN(°)	6.9, 0.8	10.2, 10.3	15.8	11.5
Stacking arrangement [11]	M–M	M–L–M	M–M	M–M

[a] eq = equatorial; ax = axial. [b] ΔNi is the distance of the Ni atom from the N_4 square plane.

bonding forces in the benzylamine adduct reported here must be represented solely by the short Ni–N (*amine*) distance of 2.286(11) Å. A parallel orientation of the phenyl ring to the glyoxímate plane as well would place this ring over the methyl groups of the macrocycle thus preventing any significant π-bonding. The *cis*-arrangement of the axial F atoms, enforced by the dimeric nature of the chelate, would not allow any of the other previously observed orientations which are suggestive of π-bonding [5].

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