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Bis[bis(diethylamino)glyoximato]nickel(II), $C_{20}H_{42}N_8NiO_4$

BY L. A. NUVAN* AND J. L. BRIANSÓ

Departamento de Cristalografía y Mineralogía, Universidad Autónoma de Barcelona, Bellaterra (Barcelona), Spain

X. SOLANS AND M. FONT-ALTABA

Departamento de Crystalografía y Mineralogía, Universidad de Barcelona, Gran Via 585, *Barcelona*-7, *Spain*

AND M. DE MATHEUS

Departamento de Química, Universidad Nacional de Colombia, Bogotá, Colombia

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Abstract. $M_r = 517{\cdot}3$, monoclinic, space group $P2_1/a$, $a = 15{\cdot}760\ (2)$, $b = 11{\cdot}792\ (3)$, $c = 7{\cdot}297\ (1)$ Å, $\beta = 99{\cdot}10\ (1)°$, $V = 1339{\cdot}0\ (7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}28$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}765$ mm^{-1}, $F(000) = 556$. Final $R = 0{\cdot}068$ for 1809 observed reflections. The Ni atom is located on an inversion centre, and displays a square-planar coordination. The bis(diaminoglyoximato)nickel(II) moiety is nearly planar, and the displacement of ethyl groups from this plane is a result of steric effects.

Introduction. Synthesis of compounds with possible metal···metal or metal···ligand contacts is presently being carried out by the Department of Chemistry of the National University of Colombia. In order to elucidate the configuration of the title compound, X-ray analysis was performed.

Experimental. Red elongated prisms, Philips PW-1100 diffractometer, Mo $K\alpha$, graphite monochromator, ω-scan technique, 1819 independent with $\theta \leq 25°$, 1809 with $I \geq 2{\cdot}5\sigma(I)$, Lp correction, absorption ignored; direct methods (*MULTAN* 80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), anisotropic full matrix (*SHELX* 76; Sheldrick, 1976), anomalous scattering factors for all atoms (*International Tables for X-ray Crystallography*, 1974), H (from ΔF synthesis) overall isotropic temperature factor, final $R = 0{\cdot}068$, $R_w = 0{\cdot}077$, $w = [\sigma^2(F) + 0{\cdot}0026\,|F|^2]^{-1}$; Digital VAX-750.

Discussion. Atomic parameters are given in Table 1;† atom numbering, bond distances and angles in Fig. 1.

The crystal structure consists of discrete molecules linked by van der Waals interactions in layers parallel to (001). The shortest Ni···Ni and Ni···N(Et)$_2$ lengths are 7·30 and 6·11 Å. The shortest intramolecular distance Ni···H(C43^i) (where $i = x,y,z + 1$) [3·04 (8) Å] is comparable to Ni···N [3·478 (3)] obtained by Endres (1979) in bis(oxamide oxi-

* This paper reports part of the research work undertaken to satisfy the requirements for this author's Master Thesis. Present address: Departamento de Química, Universidad Francisco de Paula, Santander, Colombia.

† Lists of structure factors, anisotropic thermal parameters and a complete list of distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38225 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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mato)nickel(II) dihydrate, which justified the red colour of the crystal according to Endres, Keller, Lehmann, Poveda, Rupp & van de Sand (1977).

The Ni atoms are located on inversion centres, and display a square-planar coordination. The bis-(diaminoglyoximato)nickel moiety is nearly planar, with N(5) displaying the largest displacement from atom to mean plane of −0·05 (1) Å. There is no significant difference in the molecular geometry of this moiety and those observed in the literature (Table 2). The C(3)–N(31) and C(4)–N(41) bond distances [mean value 1·381 (6) Å] are shorter than the typical C–N (=1·47 Å) distance (*International Tables for X-ray Crystallography*, 1968). This partial double-bond character produces a tendency for the diethyl group to lie in the plane of the bis(diaminoglyoximato)nickel moiety, in spite of steric effects. The torsion angles N(2)–C(3)–N(31)–C(34), C(4)–C(3)–N(31)–O(32), N(5)–C(4)–N(41)–C(42) and C(3)–C(4)–N(41)–C(44) are equal to −33·7 (6), −58·6 (6), −39·8 (6) and −65·2 (6)°, respectively, and the shortest C···C intramolecular distance, C(32)···C(44), is 3·492 (8) Å, which produces the largest deviation of these ethyl groups from planarity.

Table 1. *Final atomic coordinates* ($\times 10^4$ *for non-*H *atoms;* $\times 10^3$ *for* H *atoms) and isotropic temperature coefficients*

B_{iso} for all H atoms is 0·13 (1) Å^2.

	x	y	z	B^*_{eq}(Å^2)
Ni	10000	0	10000	3·50 (4)
O(1)	11811 (3)	−283 (4)	10821 (7)	5·1 (2)
N(2)	11151 (3)	307 (4)	9867 (7)	3·9 (2)
C(3)	11295 (3)	1035 (5)	8618 (8)	3·9 (2)
C(4)	10498 (3)	1533 (5)	7618 (8)	4·0 (2)
N(5)	9817 (3)	1092 (4)	8104 (7)	4·0 (2)
O(6)	9033 (2)	1495 (4)	7444 (6)	5·2 (2)
N(31)	12092 (3)	1381 (5)	8281 (8)	5·2 (2)
C(32)	12299 (5)	1278 (8)	6383 (11)	6·3 (3)
C(33)	12640 (8)	152 (11)	5992 (18)	8·9 (6)
C(34)	12811 (4)	1471 (8)	9797 (14)	6·7 (3)
C(35)	12594 (6)	2165 (12)	11346 (14)	8·0 (5)
N(41)	10506 (3)	2379 (5)	6310 (7)	5·2 (2)
C(42)	9849 (5)	2338 (14)	4573 (13)	9·5 (6)
C(43)	10156 (10)	2479 (12)	2859 (18)	11·2 (7)
C(44)	10878 (6)	3467 (8)	6995 (15)	7·6 (5)
C(45)	10301 (11)	4150 (12)	7965 (28)	11·6 (7)
H(O1)	838 (7)	68 (9)	781 (16)	
H(C32)	1177 (7)	116 (8)	571 (16)	
H(C32)′	1277 (7)	208 (9)	643 (15)	
H(C33)	1323 (8)	50 (10)	680 (17)	
H(C33)′	1290 (8)	16 (8)	472 (22)	
H(C33)″	1243 (7)	−44 (10)	640 (16)	
H(C34)	1323 (7)	198 (9)	900 (18)	
H(C34)′	1302 (7)	93 (10)	1010 (16)	
H(C35)	1232 (8)	266 (10)	1107 (18)	
H(C35)′	1194 (8)	182 (9)	1194 (16)	
H(C35)″	1307 (8)	213 (10)	1222 (16)	
H(C42)	954 (7)	294 (9)	443 (14)	
H(C42)′	971 (7)	84 (10)	440 (18)	
H(C43)	949 (7)	237 (9)	153 (16)	
H(C43)′	1059 (8)	199 (10)	254 (16)	
H(C44)	1149 (7)	334 (9)	764 (15)	
H(C44)′	1101 (7)	415 (10)	583 (17)	
H(C45)	1066 (9)	493 (10)	858 (19)	
H(C45)′	1055 (9)	361 (11)	930 (17)	
H(C45)″	971 (8)	394 (10)	691 (16)	

* $B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

Table 2. *Comparison of bis(diaminoglyoximato)nickel-moiety geometry values; distances in* Å, *angles in* °

	This paper		Literature*	
	Mean value	Range	Mean value	Range
Ni N	1·873 (4)	1·867 (4)–1·879 (4)	1·87 (2)	1·85 (1)–1·90 (2)
N O	1·344 (5)	1·339 (5)–1·350 (6)	1·34 (2)	1·316 (7)–1·391 (5)
N–C	1·294 (6)	1·291 (6)–1·298 (6)	1·30 (3)	1·290 (7)–1·314 (5)
C–C	1·472 (7)	–	1·48 (4)	1·435 (7)–1·54 (3)
C–*R*	1·381 (6)	1·380 (6)–1·383 (6)	1·336 (6)	1·333 (6)–1·339 (5)
O···O	2·439 (4)	–	2·46 (4)	2·40 (2)–2·567 (4)
O–H	1·18 (8)	–	–	–
H···O	1·47 (10)	–	–	–
N Ni N	82·3 (2)	–	83 (1)	82·2 (2)–84 (1)
Ni–N–O	122·7 (3)	122·2 (3)–123·3 (3)	124 (2)	122·9–127·3 (3)
Ni–N–C	116·2 (3)	116·1 (3)–116·4 (3)	116 (2)	114 (2)–117·6 (3)

* Bowers, Banks & Jacobson (1972); Calleri, Ferraris & Viterbo (1967); Endres (1979); Endres, Keller, Moroni & Weiss (1975); Leichert & Weiss (1975); Murmann & Schlemper (1967); Stephens & Vagg (1977); and Williams, Wohlauer & Rundle (1959).

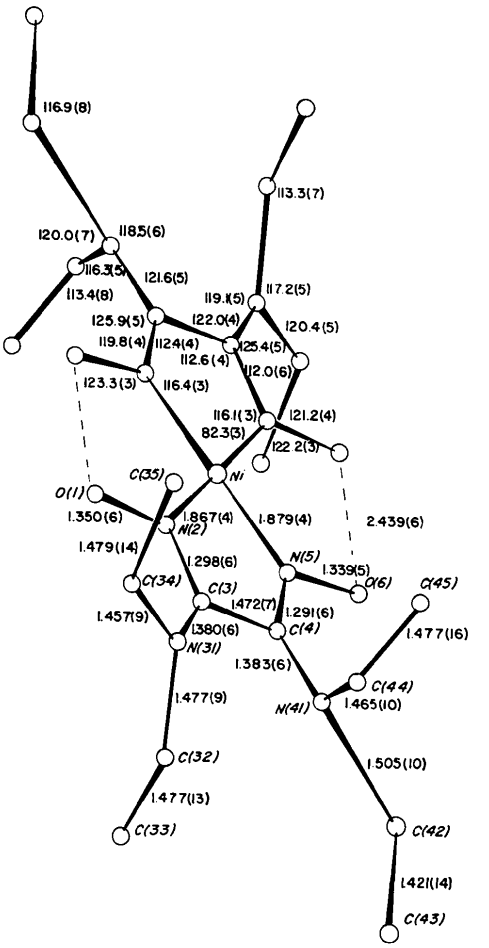

Fig. 1. View of the molecule with the numbering of atoms, bond distances (in Å) and angles (°).

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