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*Acta Cryst.* (1997). **C53**, 1232–1234

### Bis[*N*-( $\beta$ -naphthyl)-2-oxy-1-naphthaldiminato]nickel(II)

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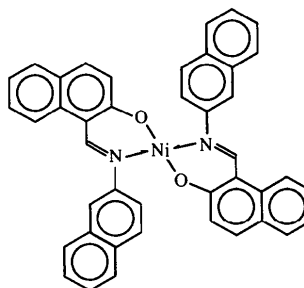
(Received 11 April 1997; accepted 12 May 1997)

## Abstract

There is a remarkable stereochemical difference between the free ligand and that coordinated to the Ni atom in the title compound {systematic name: bis[1-(2-naphthyliminomethyl)-2-naphtholato-*N,O*]nickel(II),  $[Ni(C_{21}H_{14}NO)_2]$ }. Upon coordination, the orientation of the *N*-( $\beta$ -naphthyl)-2-oxy-1-naphthaldimine ligand (which is almost planar in the solid state when uncomplexed) is changed so that the plane of the *N*- $\beta$ -naphthyl substituent is almost normal to the plane of the naphthaldiminato moiety. Two bidentate ligands coordinate to the Ni atom (which lies on an inversion centre) with their O and N atoms in a (necessarily) *trans* arrangement and forming a stepped conformation with a mean chelate-plane vertical separation of 0.639 (2) Å. Main dimensions are Ni—O 1.820 (2), Ni—N 1.901 (2) Å and O—Ni—N 91.75 (7)°.

## Comment

We are currently studying the variation of stereochemistry as a function of ligand structure in a series of complexes with the same metal atom. The crystal structure of the title compound, (I), was determined in order to ascertain the conformational perturbations of the ligand upon coordination.



(I)

The Ni atom of compound (I) (Fig. 1) lies on an inversion centre and is coordinated to two bidentate Schiff base ligands in a characteristic square-planar manner with the coordinating O and N atoms necessarily in a *trans* arrangement (Fig. 1). The Ni<sup>II</sup> square-planar geometry has normal dimensions, with Ni—O1 1.820 (2), Ni—N1 1.901 (2) Å and O1—Ni—N1 91.75 (7)°. Delocalization of the chelate-ring  $\pi$ -electrons accounts for both the O1—C12 [1.300 (3) Å] and N1—C1 [1.298 (3) Å] bond lengths being intermediate between single- and double-bond values (Allen *et al.*, 1987). Bond lengths in the naphthalene moieties are mostly as expected; the shortening of the C13—C14 bond length [to 1.347 (4) Å] is consistent with a quinoid effect (Exelby & Grinter, 1965).

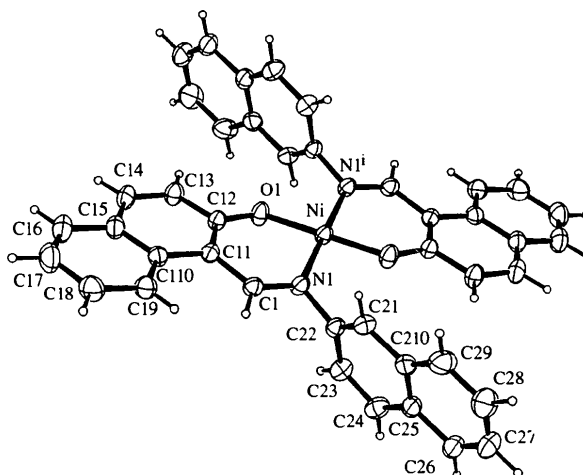


Fig. 1. ORTEP (Johnson, 1976) view of the title molecule showing 30% probability displacement ellipsoids. H atoms are plotted as circles of arbitrary size. [Symmetry code: (i)  $-x, -y, -z$ ].

In the solid state, molecule (I) adopts a stepped conformation quantified by the perpendicular separation between the mean planes of opposite chelate rings and this distance is 0.639 (2) Å (Holm & O'Connor, 1971). With only one exception (Kamenar, Kaitner, Ferguson & Waters, 1990), all *N*-substituted salicylaldimine and naphthaldimine bis-bidentate Ni<sup>II</sup> complexes show stepped conformations (Elerman, Kabak & Tahir, 1996; Kaitner, Meštrović & Pavlović, 1997; Hökelek, Gündüz, Hayvali & Kiliç, 1995*a,b*; Fernandez-G. *et al.*, 1987; Fernandez-G., Rosales, Toscano & Tapia, 1986). The N1...O1 bite distance is increased upon coordination from 2.529 (4) Å in the free ligand to 2.672 (2) Å in (I) with concomitant small increases in the chelate-ring valence angles (details are in Table 1).

A feature of complex (I) is the orientation of the bulky *N*-β-naphthyl substituent on the ligand. The stereochemistry of the free ligand in the solid is almost planar (Gavranić, Kaitner & Meštrović, 1996), but upon coordination to the Ni atom, the ligand conformation is changed so that the plane of the *N*-β-naphthyl substituent is almost normal to the plane of the naphthaldiminato moiety [C1—N1—C22—C21 torsion angle -83.7 (2)°].

## Experimental

The title compound was obtained as a green precipitate from the template condensation of an equimolar mixture of 2-hydroxy-1-naphthaldehyde, β-naphthylamine and NiCl<sub>2</sub>·6H<sub>2</sub>O. Suitable crystals for our X-ray analysis were obtained by liquid diffusion (chloroform:*n*-hexane 1:5).

### Crystal data

[Ni(C<sub>21</sub>H<sub>14</sub>NO)<sub>2</sub>]  
*M<sub>r</sub>* = 651.41  
 Monoclinic  
*P*2<sub>1</sub>/c  
*a* = 9.941 (2) Å  
*b* = 12.084 (3) Å  
*c* = 14.001 (3) Å  
 β = 110.14 (2)°  
*V* = 1579.1 (6) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.370 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

### Data collection

Phillips PW1100 diffractometer  
 ω scan  
 Absorption correction:  
 ψ scans (North, Phillips & Mathews, 1968)  
*T<sub>min</sub>* = 0.683, *T<sub>max</sub>* = 0.735  
 4774 measured reflections  
 4598 independent reflections

Mo Kα radiation  
 λ = 0.71069 Å  
 Cell parameters from 25 reflections  
 θ = 12–20°  
 μ = 0.66 mm<sup>-1</sup>  
*T* = 293 K  
 Prism  
 0.75 × 0.44 × 0.43 mm  
 Dark green

2578 reflections with  
*I<sub>net</sub>* > 2σ(*I<sub>net</sub>*)  
*R<sub>int</sub>* = 0.033  
 θ<sub>max</sub> = 30°  
*h* = -14 → 13  
*k* = 0 → 17  
*l* = 0 → 19  
 3 standard reflections  
 frequency: 90 min  
 intensity decay: 2.0%

### Refinement

Refinement on *F*  
*R* = 0.034  
*wR* = 0.054  
*S* = 1.05  
 2578 reflections  
 214 parameters  
 H atoms constrained  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*) + 0.002*F<sub>o</sub>*<sup>2</sup>]

(Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.32 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.24 e Å<sup>-3</sup>  
 Extinction correction: none  
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (Å, °)

Ni—O1	1.820 (2)	N1—C22	1.446 (3)
Ni—N1	1.901 (2)	C1—C11	1.425 (3)
O1—C12	1.300 (3)	C11—C12	1.399 (3)
N1—C1	1.298 (3)	C13—C14	1.347 (4)
O1—Ni—N1	91.75 (7)	C1—N1—C22	116.1 (2)
Ni—O1—C12	130.0 (1)	N1—C1—C11	126.2 (2)
Ni—N1—C1	125.5 (1)	C1—C11—C12	119.2 (2)
Ni—N1—C22	118.4 (1)	O1—C12—C11	124.4 (2)

H atoms were allowed for in the refinement as riding atoms with *d*(C—H) = 0.95 Å. All H atoms were included in the structure-factor calculations with isotropic displacement parameters set at 1.2*U<sub>eq</sub>* of the atom to which they were attached.

Data collection: *DIF4* (Stoe & Cie, 1991*a*). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1991*b*). Program(s) used to solve structure: *NRCVAX SOLVER* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES*.

The Research Grant to BK by the Ministry of Science and Technology, the Republic of Croatia is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1334). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1997). **C53**, 1234–1236

### A New Pentachlorotellurate(IV): *catena*-Poly[hexakis(acetonitrile)aluminium tris-[tetrachlorotellurate(IV)- $\mu$ -chloro] acetonitrile]

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(Received 16 October 1996; accepted 26 March 1997)

#### Abstract

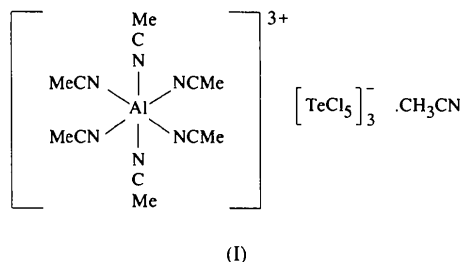
In the title compound, [Al(CH<sub>3</sub>CN)<sub>6</sub>][TeCl<sub>5</sub>]<sub>3</sub>.CH<sub>3</sub>CN, the polymeric anions form infinite helical chains from apex-sharing distorted octahedral TeCl<sub>6</sub> units. These anionic chains are stabilized by [Al(CH<sub>3</sub>CN)<sub>6</sub>]<sup>3+</sup> counterions.

#### Comment

From tetra- to hexahalide, all tellurium(IV) halide structures are based on octahedrally or pseudo-octahedrally coordinated tellurium (Hazell, 1966; Adams & Lock, 1967; Collins & Webster, 1972; Krebs, Bonmann & Gretenkord, 1992). TeCl<sub>4</sub> has been described as TeCl<sub>3</sub>Cl<sup>-</sup>, with Te and Cl atoms arranged within a three-dimensional cubic array to form distorted Te-centred octahedra displaying three short and three long Te—Cl bonds (Buss & Krebs, 1971). This structural arrangement has also been encountered in TeCl<sub>3</sub>X moieties with X = AlCl<sub>4</sub> (Krebs, Buss & Altena, 1971), SbF<sub>6</sub>, AsF<sub>6</sub> (Christian, Collins, Gillespie & Sawyer, 1985), ClO<sub>4</sub> (Favier, 1991), MoCl<sub>4</sub> (Beck, 1991), etc. On the other hand, A<sub>2</sub>TeCl<sub>6</sub> salts (where A = K, Rb or NH<sub>4</sub>) contain discrete and regular octahedral hexa-

chlorotellurate(IV) anions (Webster & Collins, 1973; Armstrong, Dufort & Powell, 1991).

Between the three-dimensional condensed tellurium tetrachlorides and the discrete molecular species of tellurium hexachloride, intermediate polymeric moieties are formed, depending on the number of Cl atoms per Te atom. In [H<sub>9</sub>O<sub>4</sub>]<sub>n</sub>[Te<sub>2</sub>Cl<sub>9</sub>]<sub>n</sub>, with a Cl/Te ratio of 4.5, the Te<sub>2</sub>Cl<sub>10</sub> dimeric units (two TeCl<sub>6</sub> octahedra condensed by edge-sharing) polymerize into infinite [Te<sub>2</sub>Cl<sub>9</sub>]<sub>n</sub><sup>n-</sup> zigzag chains through Te—Cl—Te bridges (Krebs, Bonmann & Gretenkord, 1992). [PCL<sub>4</sub>][TeCl<sub>5</sub>] contains distorted octahedral TeCl<sub>6</sub> units fused by corner-sharing within an infinite [TeCl<sub>5</sub>]<sub>n</sub><sup>n-</sup> chain (Collins & Webster, 1972; Krebs, Buss & Berger, 1973). This assertion is corroborated by the structure of the title compound, (I), which mainly consists of an infinite pseudo-helical anionic chain of [TeCl<sub>5</sub>]<sub>n</sub><sup>n-</sup> stabilized by *n*/3 discrete [Al(CH<sub>3</sub>CN)<sub>6</sub>]<sup>3+</sup> units.



The structure also contains free molecules of acetonitrile. The helical unit comprises three non-equivalent Te atoms in distorted octahedral environments. Each

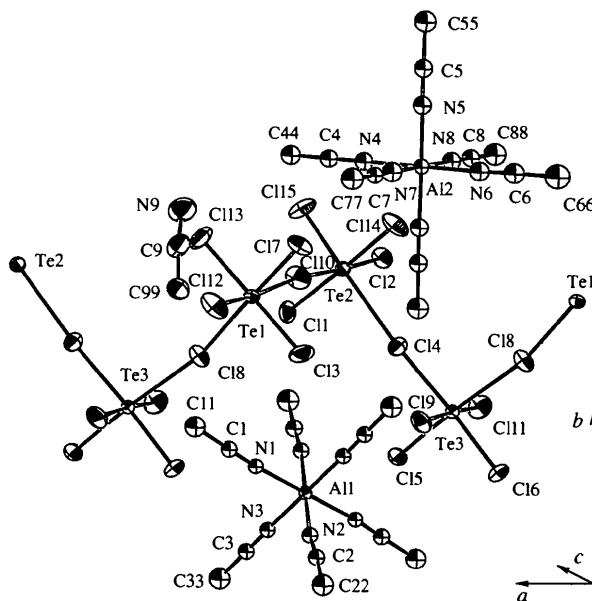


Fig. 1. The molecular structure of the title compound. H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level.