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[*N*,*N*′-Ethylenebis(salicylideneiminato)]-nickel(II) dimethylformamide solvate

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The title compound, $[Ni(C_{16}H_{14}N_2O_2)]\cdot C_3H_7NO$, crystallizes with one Ni(salen) molecule [salen is *N*,*N*′-ethylenebis(salicylideneiminate)] and one dimethylformamide molecule in the asymmetric unit. The molecular structure is similar to that of the solvent-free compound, known from the literature. In the crystal structure, the nearly planar molecules are stacked to form polymeric chains in the crystallographic *b* direction. The crystal structure has pseudo-translational symmetry (superstructure).

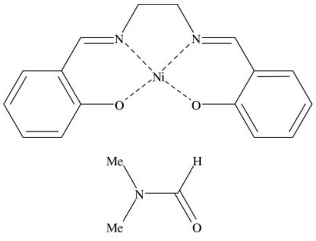
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.027
wR factor = 0.080
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The dimethylformamide (DMF) solvate of [*N*,*N*′-ethylenebis(salicylideneiminato)]nickel(II), (I), was obtained by recrystallization of solvent-free Ni(salen) from DMF.



(I)

The molecular structure of (I) (Fig. 1) has an approximate non-crystallographic twofold symmetry. The Ni atom is in a square-planar environment with an angle sum of 360°. The N—Ni—O angles in the six-membered chelate rings are both

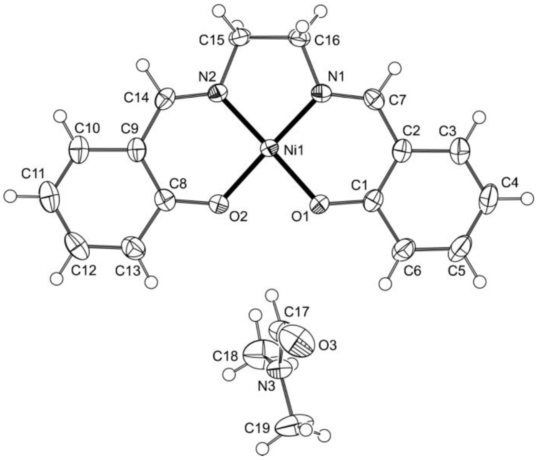


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

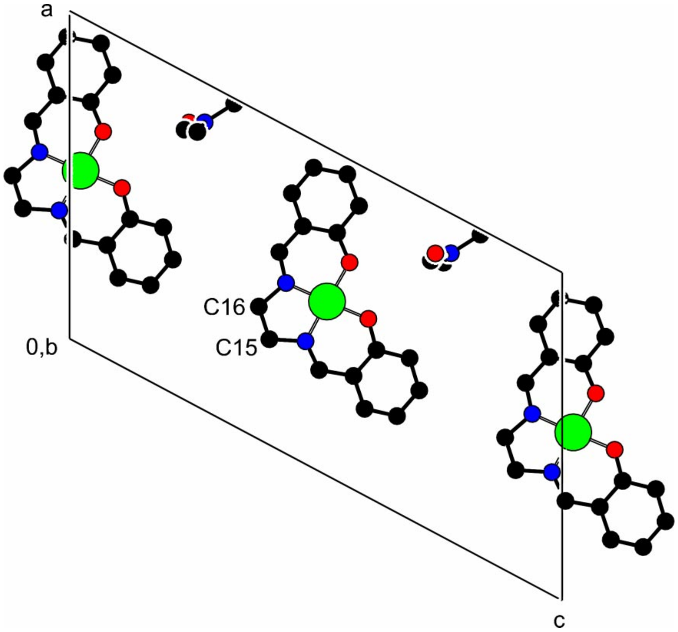


Figure 2
The packing of compound (I) in the crystal structure, viewed along the crystallographic *b* axis. Only the molecules with approximately the same *y* value are shown. The pseudo-translational symmetry is broken by the orientation of the C15—C16 bridge and by the arrangement of the DMF solvent molecules. (Green denotes Ni atoms, red O atoms, blue N atoms and black C atoms.)

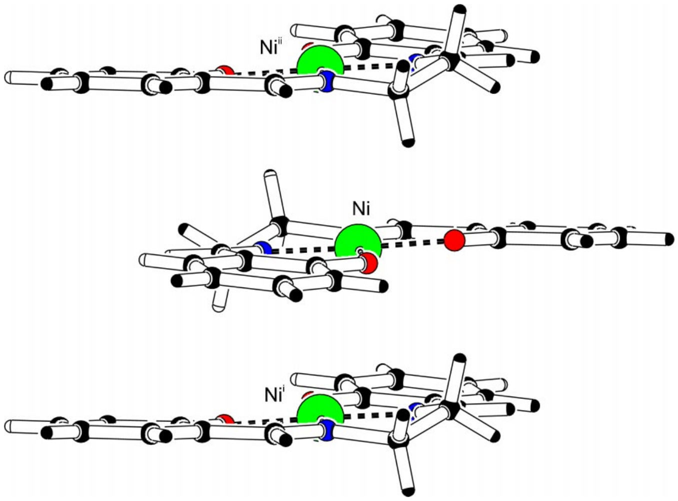


Figure 3
Stacking of the Ni(salen) molecules in the crystallographic *b* direction [symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii): $1 - x, 2 - y, 1 - z$].

94.94 (5)°, and thereby larger than the five-membered chelate N1—Ni1—N2 angle of 86.50 (6)° and the O1—Ni1—O2 angle of 83.69 (5)°. The average Ni—N and Ni—O distances of 1.8467 and 1.8474 Å, respectively, are equal within standard uncertainties. The molecule is slightly bent, with a dihedral angle of 7.13 (8)° between the benzene rings. No disorder of the C15—C16 ethylene bridge is observed. The overall molecular structure is comparable with that of the solvent-free structure, known from the literature (Montgomery & Morosin, 1961; Shkol'nikova *et al.*, 1970; Gaetani Manfredotti & Guastini, 1983; DiMauro & Kozlowski, 2002).

The structure of (I) has pseudo-translational symmetry in the crystallographic *c* direction. This symmetry is only broken by the orientation of the C15—C16 bridge and by the arrangement of the DMF solvent molecules (Fig. 2). This pseudo-symmetry is also observed in reciprocal space: reflections *hkl* with $l = 2n$ have an average intensity of 1336.4, and for those with $l = 2n + 1$ the average intensity is 139.6, based on calculated structure factors. The average of the normalized structure factors for the sublattice with $l = 2n$ is $<E^2> = 1.706$, while for the superlattice, $<E^2> = 0.232$. As expected (Cascarano *et al.*, 1985), the cumulative $N(z)$ probability distribution shows hypercentric behaviour.

In the solvent-free crystal structure, the Ni(salen) molecules form centrosymmetric dimers by stacking of the nearly planar molecules, with a short intermolecular Ni···Ni distance of 3.1802 (6) Å (DiMauro & Kozlowski, 2002). From a quantum-chemical point of view, this can be explained by an interaction of the d_{z^2} orbitals of the Ni^{2+} ions (Aullón *et al.*, 1998). In the DMF solvate, (I), of the present communication, the Ni(salen) molecules are stacked into polymers with intermolecular Ni···Ni distances of 3.3901 (3) and 3.5513 (3) Å (Fig. 3). The solvent molecules are arranged between these polymeric chains.

Experimental

The solvent-free Ni(salen) complex was heated in dimethylformamide until a saturated solution was obtained. After filtration, the solution was allowed to cool. The title complex, (I), crystallized as red needles, which are elongated along the *b* axis.

Crystal data

$[Ni(C_{16}H_{14}N_2O_2)]\cdot C_3H_7NO$
$M_r = 398.10$
Monoclinic, $P2_1/c$
$a = 13.3866$ (2) Å
$b = 6.6690$ (1) Å
$c = 22.7332$ (4) Å
$\beta = 118.0383$ (7)°
$V = 1791.31$ (5) Å^3
$Z = 4$

$D_x = 1.476$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 57 238 reflections
$\theta = 1.0$–27.5°
$\mu = 1.11$ mm^{-1}
$T = 150$ (2) K
Needle, red
0.58 × 0.08 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.83$, $T_{\max} = 0.94$
36 002 measured reflections

4132 independent reflections
3209 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.057$
$\theta_{\max} = 27.5°$
$h = -17 \rightarrow 17$
$k = -8 \rightarrow 8$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.080$
$S = 1.07$
4132 reflections
249 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.3546P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.8445 (11)	C3—C4	1.368 (3)
Ni1—N2	1.8451 (13)	C4—C5	1.398 (3)
Ni1—N1	1.8483 (13)	C5—C6	1.379 (3)
Ni1—O2	1.8503 (11)	C8—C13	1.414 (2)
Ni1—Ni1^{i}	3.3901 (3)	C8—C9	1.418 (2)
Ni1—Ni1^{ii}	3.5513 (3)	C9—C10	1.416 (2)
O1—C1	1.317 (2)	C9—C14	1.425 (2)
O2—C8	1.3143 (18)	C10—C11	1.369 (3)
N1—C7	1.295 (2)	C11—C12	1.393 (3)
N1—C16	1.4762 (19)	C12—C13	1.378 (2)
N2—C14	1.296 (2)	C15—C16	1.514 (2)
N2—C15	1.478 (2)	O3—C17	1.218 (2)
C1—C2	1.413 (2)	N3—C17	1.330 (2)
C1—C6	1.416 (2)	N3—C19	1.441 (2)
C2—C3	1.418 (2)	N3—C18	1.447 (2)
C2—C7	1.432 (2)		
O1—Ni1—N2	177.53 (5)	C3—C4—C5	119.23 (17)
O1—Ni1—N1	94.94 (5)	C6—C5—C4	121.33 (18)
N2—Ni1—N1	86.50 (6)	C5—C6—C1	120.47 (18)
O1—Ni1—O2	83.69 (5)	N1—C7—C2	125.30 (15)
N2—Ni1—O2	94.94 (5)	O2—C8—C13	118.57 (15)
N1—Ni1—O2	177.62 (5)	O2—C8—C9	123.62 (14)
C1—O1—Ni1	127.37 (11)	C13—C8—C9	117.80 (15)
C8—O2—Ni1	127.35 (10)	C10—C9—C8	119.46 (16)
C7—N1—C16	118.36 (14)	C10—C9—C14	118.78 (16)
C7—N1—Ni1	126.54 (12)	C8—C9—C14	121.61 (14)
C16—N1—Ni1	115.07 (10)	C11—C10—C9	121.55 (17)
C14—N2—C15	118.32 (14)	C10—C11—C12	118.82 (16)
C14—N2—Ni1	126.56 (12)	C13—C12—C11	121.55 (17)
C15—N2—Ni1	115.04 (10)	C12—C13—C8	120.78 (17)
O1—C1—C2	123.80 (14)	N2—C14—C9	125.24 (15)
O1—C1—C6	117.99 (15)	N2—C15—C16	108.12 (12)
C2—C1—C6	118.22 (15)	N1—C16—C15	108.52 (12)
C1—C2—C3	119.63 (16)	C17—N3—C19	121.29 (16)
C1—C2—C7	121.45 (15)	C17—N3—C18	121.56 (17)
C3—C2—C7	118.92 (16)	C19—N3—C18	117.12 (15)
C4—C3—C2	121.12 (18)	O3—C17—N3	125.35 (19)
C16—N1—C7—C2	174.74 (14)	N2—C15—C16—N1	27.36 (16)
C15—N2—C14—C9	170.98 (14)		

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, 2-y, 1-z$.

Atoms H7, H14 and H17 were refined freely with isotropic displacement parameters. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.99–1.00 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl H atoms and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for all other H atoms.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *HKL*2000 (Otwinowski & Minor, 1997); data reduction: *HKL*2000 and *SORTAV* (Blessing, 1997); program(s) used to solve structure: *DIRDIF*99 (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

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