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Key indicators

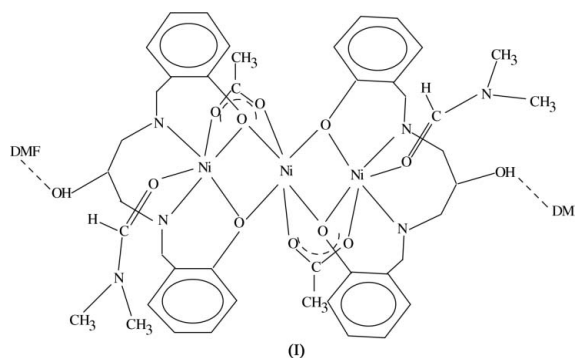
Single-crystal X-ray study
 $T = 300$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.034
 wR factor = 0.096
Data-to-parameter ratio = 15.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Di- μ -acetato-bis[μ -2-hydroxy- N,N' -bis(2-oxido-
benzyl)propane-1,3-diamine]bis(N,N' -dimethyl-
formamide)trinickel(II) N,N' -dimethylformamide
disolvate

The title compound, $[\text{Ni}_3(\text{C}_{17}\text{H}_{20}\text{N}_2\text{O}_2)_2(\text{C}_2\text{H}_3\text{O}_2)_2(\text{C}_3\text{H}_7\text{NO})_2] \cdot 2\text{C}_3\text{H}_7\text{NO}$, is a centrosymmetric trinuclear nickel(II) complex. The central and terminal nickel(II) atoms have distorted octahedral coordination, the central Ni^{II} atom by six O atoms, and the terminal Ni^{II} atoms by four O atoms and two N atoms each. The average Ni—O and Ni—N distances for the terminal Ni ions are 2.062 (17) and 2.082 (2) Å, respectively. The Ni···Ni distance is 3.035 (2) Å.

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Comment

Trinuclear linear homo- or heteronuclear complexes are of interest because of their ferroelectric and electronic properties (Lashgari *et al.*, 1998). Nickel(II) is also used as a spectroscopic probe in metal-replacement studies of other metalloenzyme systems (Cammack *et al.*, 1987; Blaszkak *et al.*, 1981). In these complexes, various combinations of metal ions in the central and terminal locations, as well as the μ -bridges, such as acetate and nitrite anions, are observed (Ülkü *et al.*, 1997; Atakol *et al.*, 1999; Yıldırım & Atakol, 2002; Arıcı *et al.*, 2005). Generally, homo- or heteronuclear complexes are formed with the types of Schiff bases used in this present study, and in some cases the alcohol OH group of this ligand is involved in coordination.



In the title compound, (I), the octahedrally coordinated central Ni atom is located at an inversion centre, with two *trans* positions occupied by O atoms from the two acetate groups. The coordination of each terminal Ni atom is distorted octahedral, comprising four bridging O and two N atoms (Fig. 1 and Table 1), with a tetradentate Schiff base ligand, attached *via* two O and two N atoms, as well as one O atom from the bridging acetate ligand and one O atom from a coordinated DMF (dimethylformamide) ligand. The terminal Ni atom lies 0.0525 (9) Å out of its equatorial plane (O1,O2,N1,N2). The Ni···Ni distance is 3.035 (2) Å and the Ni···Ni···Ni angle is linear by symmetry. The chelate ring (Ni1,N1,C8,C9,C10,N2) has a distorted chair conformation;

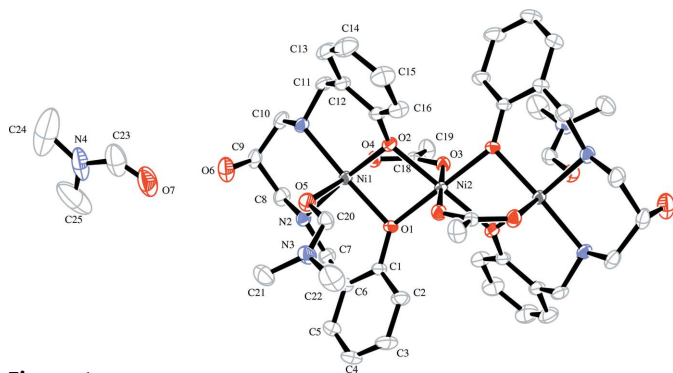


Figure 1

The molecular structure and atomic labelling scheme of the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. Unlabelled atoms are related to labelled atoms by $-x$, $-y$, $1 - z$.

the dihedral angles between the mean planes of (N1,N2,C8,C9) and each of (Ni1,N1,N2) and (C8,C9,C10) are 20.76 (15) and 46.16 (3) $^\circ$, respectively. The other two chelate rings approximate more nearly to boat forms. Hydrogen bonds are detailed in Table 2.

Experimental

The ligand was prepared in two steps. In the first step, the Schiff base was synthesized from 2-hydroxyacetophenone (4.88 g, 40 mmol) and 2-hydroxy-1,3-propanediamine (1.80 g, 20 mmol) in MeOH solution (40 ml). The mixture was left to stand in air. After 1 d, the crystalline yellow Schiff base was obtained. In the second step, the Schiff base (2.98 g, 10 mmol) was dissolved in cold MeOH (50 ml) and NaBH₄ was added slowly to this solution until it turned colourless. The reduced Schiff base precipitated after addition of excess water. The mixture was left to stand at 277 K for one day to yield crystals, which were filtered off. The crystals were allowed to dry in air. For the preparation of the title complex, the ligand (0.602 g, 2 mmol) was dissolved in hot DMF (50 ml), and solutions of NiCl₂·6H₂O (0.710 g, 3 mmol) in hot MeOH (25 ml) and of anhydrous CH₃COONa (0.492 g, 6 mmol) in hot water (5 ml) were added. The resulting blue crystals were filtered off and dried in air.

Crystal data

[Ni₃(C₁₇H₂₀N₂O₂)₂(C₂H₃O₂)₂·
(C₃H₇NO)₂·2C₃H₇NO]
M_r = 1187.24
Triclinic, *P*1̄
a = 10.373 (3) Å
b = 12.125 (1) Å
c = 12.863 (1) Å
 α = 107.99 (1) $^\circ$
 β = 107.99 (1) $^\circ$
 γ = 98.18 (1) $^\circ$
V = 1412.1 (5) Å³

Z = 1
D_x = 1.396 Mg m⁻³
Mo *K* α radiation
Cell parameters from 25
reflections
 θ = 1.8–26.0 $^\circ$
 μ = 1.06 mm⁻¹
T = 300 (2) K
Prism, blue
0.40 × 0.13 × 0.10 mm

Data collection

Enraf-Nonius CAD-4
diffractometer
 $\omega/2\theta$ scans
Absorption correction: ψ scan
 ψ scan (Fair, 1990)
T_{min} = 0.848, *T_{max}* = 0.900
5524 measured reflections
5524 independent reflections

4342 reflections with *I* > 2 σ (*I*)
R_{int} = 0.0
 θ_{\max} = 26.0 $^\circ$
h = -12 → 11
k = -14 → 14
l = 0 → 15

3 standard reflections
frequency: 120 min
intensity decay: 1.5%

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.034
wR(*F*²) = 0.096
S = 1.02
5524 reflections
356 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.5368P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.52 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$$

Table 1

Selected geometric parameters (Å, $^\circ$).

N1–Ni1	2.084 (2)	O2–Ni2	2.0374 (16)
N2–Ni1	2.080 (2)	O3–Ni2	2.1044 (16)
O1–Ni1	2.0339 (16)	O4–Ni1	2.0412 (17)
O1–Ni2	2.0453 (15)	O5–Ni1	2.1545 (18)
O2–Ni1	2.0189 (16)		
O2–Ni1–O1	81.90 (6)	N2–Ni1–N1	92.90 (9)
O2–Ni1–O4	93.19 (7)	O2–Ni1–O5	93.57 (7)
O1–Ni1–O4	90.77 (7)	O1–Ni1–O5	91.17 (7)
O2–Ni1–N2	173.05 (8)	O4–Ni1–O5	173.17 (7)
O1–Ni1–N2	92.44 (8)	N2–Ni1–O5	82.47 (9)
O4–Ni1–N2	90.90 (8)	N1–Ni1–O5	86.61 (8)
O2–Ni1–N1	92.57 (8)	O2–Ni2–O1	81.17 (6)
O1–Ni1–N1	173.91 (8)	O2–Ni2–O3	86.69 (7)
O4–Ni1–N1	92.09 (8)	O1–Ni2–O3	85.85 (7)

Table 2

Hydrogen-bond geometry (Å, $^\circ$).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N1–H1'...O6	0.81 (3)	2.39 (3)	2.765 (3)	109 (2)
N2–H2'...O6	0.81 (3)	2.49 (3)	2.836 (3)	107 (2)
O6–H6A...O7 ⁱ	1.11 (4)	1.57 (4)	2.660 (4)	168 (3)

Symmetry codes: (i) 1 – *x*, 1 – *y*, –*z*.

H atoms on N1, N2 and O6 were located in a difference Fourier map and refined isotropically. Other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with distances in the range 0.93–1.11 Å and with *U_{iso}*(H) = 1.3*U_{eq}*(C). It was noted that the DMF ligand has high displacement parameters, but multiple sites were not resolved.

Data collection: *CAD-4 EXPRESS* (Enraf-Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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