

Bis[μ_2 -*N,N'*-bis(2-oxidobenzyl)propane-1,3-diamine]-1 κ^4 *O,N,N',O':2\kappa^2*O,O':-*2 κ^2 *O,O':3\kappa^4*O,N,N',O'-bis(N,N'-dimethylformamide)-1\kappa O,3\kappa O-di-\mu_2*-acetato-1:2 κ^2 *O:O';2:3\kappa^2*O:O'-dinickel(II)zinc(II)****

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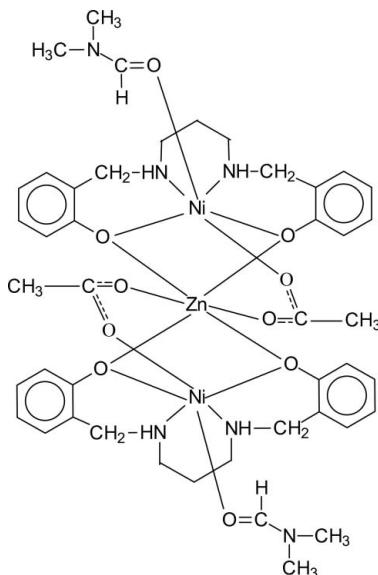
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(C-C) = 0.010$ Å;
 R factor = 0.056; wR factor = 0.176; data-to-parameter ratio = 14.1.

The molecule of the title compound, $[Ni_2Zn(C_{17}H_{20}N_2O_2)_2 \cdot (C_2H_3O_2)_2(C_3H_7NO)_2]$, contains a linear hetero-trinuclear arrangement with a central Zn^{II} ion located on an inversion centre. The $Zn \cdots Ni$ pairs are triply bridged via O atoms of $SALPD^{2-}$ [N,N' -bis(salicylidene)-1,3-propanediamine] and acetate ligands. The central Zn^{II} ion is in a distorted octahedral coordination environment formed by four O atoms of two $SALPD^{2-}$ ligands in the equatorial plane and two O atoms of two symmetry-related acetate ligands in the axial positions. The terminal Ni^{II} ions, related by an inversion centre, also have distorted octahedral coordination environments formed by two O and two N atoms of $SALPD^{2-}$ ligands in the equatorial plane; the axial positions are occupied by O atoms of dimethylformamide and acetate ligands, which are *trans* with respect to the terminal Ni atoms. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds.

Related literature

For general background, see: Aneetha *et al.* (1999); Reglinski *et al.* (2006); Fukuhara *et al.* (1990). For related literature, see: Ülkü *et al.* (1997); Tahir *et al.* (1998); Atakol *et al.* (1999); Ülkü *et al.* (1999); Ülkü *et al.* (2001); Arıcı *et al.* (2001); Tatar & Atakol (2002); Tatar *et al.* (2007).



Experimental

Crystal data

$[Ni_2Zn(C_{17}H_{20}N_2O_2)_2 \cdot (C_2H_3O_2)_2(C_3H_7NO)_2]$	$\beta = 92.124 (13)^\circ$
$M_r = 1015.75$	$V = 2318.5 (7) \text{ \AA}^3$
Monoclinic, $P2_1/n$	$Z = 2$
$a = 10.3035 (13) \text{ \AA}$	$Cu K\alpha$ radiation
$b = 17.894 (4) \text{ \AA}$	$\mu = 2.06 \text{ mm}^{-1}$
$c = 12.584 (2) \text{ \AA}$	$T = 298 (2) \text{ K}$
	$0.3 \times 0.1 \times 0.1 \text{ mm}$

Data collection

Enraf-Nonius TurboCAD-4 diffractometer	4017 independent reflections
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	2381 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.781$, $T_{\max} = 0.814$	$R_{\text{int}} = 0.033$
4017 measured reflections	3 standard reflections
	frequency: 120 min
	intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$	285 parameters
$wR(F^2) = 0.176$	H-atom parameters not refined
$S = 1.03$	$\Delta\rho_{\max} = 0.32 \text{ e \AA}^{-3}$
4017 reflections	$\Delta\rho_{\min} = -0.85 \text{ e \AA}^{-3}$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C2-H2A \cdots O3^i$	0.93	2.51	3.211 (7)	133
$C16-H16 \cdots O3^i$	0.93	2.55	3.241 (8)	132
$C20-H20 \cdots O3^i$	0.93	2.50	3.325 (8)	149

Symmetry code: (i) $-x, -y, -z$.

Table 2Coordination properties (\AA , $^\circ$) of similar trinuclear complexes.. M_c is the central metal ion and M_t the terminal metal ion.

Complex	$M_c \cdots M_t$	$M_c\text{--O}$	$\text{O}\text{--}M_c\text{--O}$	$M_t\text{--O/N}$	$\text{O/N}\text{--}M_t\text{--O/N}$
(I)	3.0705 (11)	2.064 (4)	80.76 (15)	2.038 (4)	81.99 (15)
		2.121 (4)	99.24 (15)	2.168 (4)	93.85 (16)
(II)	3.0520 (8)	2.098 (3)	77.5 (1)	1.975 (4)	80.3 (1)
		2.124 (4)	102.5 (1)	2.071 (4)	104.9 (2)
(III)	3.0017 (6)	2.055 (2)	76.04 (9)	1.941 (2)	81.5 (1)
		2.188 (2)	93.74 (9)	2.355 (3)	97.0 (1)
(IV)	Not given	2.083 (6)	79.0 (2)	1.995 (5)	82.1 (2)
		2.118 (5)	84.5 (2)	2.179 (6)	97.8 (3)
(V)	3.043 (2)	2.024 (3)	79.4 (1)	2.010 (3)	79.4 (1)
		2.098 (3)	87.1 (1)	2.254 (3)	96.2 (2)
(VI)	2.9967 (4)	2.048 (2)	78.70 (8)	2.003 (2)	80.88 (8)
		2.103 (2)	85.86 (9)	2.152 (2)	98.5 (1)
(VII)	3.0556 (5)	2.0705 (19)	78.89 (8)	2.0082 (19)	81.65 (8)
		2.082 (2)	92.92 (8)	2.186 (2)	97.95 (10)
(VIII)	3.0601 (6)	2.052 (2)	80.96 (9)	2.029 (2)	81.79 (9)
		2.102 (2)	86.57 (9)	2.165 (2)	96.95 (10)
(IX)	3.0857 (14)	2.075 (3)	80.34 (12)	2.037 (3)	81.85 (12)
		2.160 (3)	99.66 (12)	2.147 (3)	93.54 (13)

Notes: (II) $[\text{Zn}|\text{Zn}(\text{SALPD}^{2-})(\text{acetato})]_2$ (Ülkü *et al.*, 2001); (III) $[\text{Zn}|\text{Cu}(\text{SALPD}^{2-})(\text{nitrato})]_2$ (Ülkü *et al.*, 1999); (IV) $[\text{Co}|\text{Ni}(\text{SALPD}^{2-})(\text{nitrato})(\text{DMF})]_2$ (Atakol *et al.*, 1999); (V) $[\text{Ni}|\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{DMSO})]_2$ (Ülkü *et al.*, 1997); (VI) $[\text{Cu}|\text{Ni}(\text{SALPD}^{2-})(\text{nitrato})(\text{DMF})]_2$ (Tahir *et al.*, 1998); (VII) $[\text{Cu}|\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{DMF})]_2$ (Arici *et al.*, 2001); (VIII) $[\text{Ni}|\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{DMF})]_2$ (Tatar & Atakol, 2002); (IX) $[\text{Ni}|\text{Ni}(\text{DMLH}_2)(\text{formato})(\text{DMF})]_2$, where $\text{DMLH}_2 = (\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}_2)$ (Tatar *et al.*, 2007).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2314).

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Acta Cryst. (2007). E63, m2424-m2425 [doi:10.1107/S160053680704144X]

**Bis[μ_2 -*N,N'*-bis(2-oxidobenzyl)propane-1,3-diamine]-
 $1\text{H}^4O,N,N',O':2\text{H}^2O,O';2\text{H}^2O,O':3\text{H}^4O,N,N',O'$ -bis(*N,N'*-dimethylformamide)- $1\text{H}O,3\text{H}O$ -di- μ_2 -
 acetato- $1:2\text{H}^2O:O';2:3\text{H}^2O:O'$ -dinickel(II)zinc(II)**

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Comment

As it is well known, Schiff bases are easily reduced in alcoholic media with the presence of NaBH_4 giving secondary amines. The ONNO phenol amines are obtained by the reduction of ONNO type Schiff bases (Aneetha *et al.*, 1999; Reglinski *et al.*, 2006). Bis-*N,N'*(2-salicylidene)-1,3-propanediamine is a Schiff base ligand tending to give polynuclear complexes [Fukuhara *et al.*, 1990], and its reduction results in the formation of bis-*N,N'*(2-hydroxybenzyl)-1,3-propanediamine. In its complexes, various combinations of metal ions in the central and terminal locations, as well as the μ -bridges, such as acetato, formato (CHO_2), nitrato (NO_3) or nitrito (NO_2) anions are possible. Oxygen-bridged polynuclear complexes of transition series based on Schiff base ligands with similar formula have been the subject of much interest in our laboratory Ülkü *et al.*, 1997; Tahir *et al.*, 1998; Atakol *et al.*, 1999; Ülkü *et al.*, 1999; Ülkü *et al.*, 2001; Arıcı *et al.*, 2001; Tatar & Atakol, 2002; Tatar *et al.*, 2007). The structure determination of the title compound, (I), a hetero-trinuclear $[\text{Zn}\{\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{dmf})\}_2]$ complex [where SALPD $^{2-}$ is *N,N'*-bis(salicylidene)-1,3-propanediaminato and dmf is dimethylformamide], was undertaken in order to determine the ligands properties and also to compare the results obtained with those reported previously.

The molecule of the title compound, (I), contains a linear hetero-trinuclear arrangement with a central Zn^{II} ion located on an inversion centre and two terminal Ni^{II} ions related by an inversion centre (Fig. 1). Four O atoms of two SALPD $^{2-}$ ligands in the equatorial plane and two O atoms of two acetate ligands located at the axial positions constitute the distorted octahedral coordination sphere around the Zn atom. The terminal Ni^{II} ions also have distorted octahedral coordination environments formed by two O and two N atoms of SALPD $^{2-}$ ligands in the equatorial plane. The Ni atom is 0.0548 (9) Å away from the equatorial plane. The dihedral angle between (O1/Ni/O2) and (N1/Ni/N2) planes is 4.22 (14) $^\circ$. The axial positions are occupied by O atoms of dmf and acetato ligands, in which they are *trans* about the terminal Ni atoms. The dihedral angle between (O1/Zn/O2) and (O1/Ni/O2) planes is 19.44 (13) $^\circ$. The overall result is three edge shared octahedrons, in which the closest Zn···Ni distance is 3.0702 (11) Å.

The coordination geometry about the central metal ion ($M_c = \text{Zn, Ni, Cu, Co}$) and the terminal metal ions ($M_t = \text{Ni, Zn, Cu}$) are very similar to those found for the corresponding complexes, in which the metal ions or type of μ -bridges are replaced. M_c ions of these complexes retain the distorted octahedral coordination. If there is a solvent molecule (*e.g.* dmf, DMSO: dimethylsulfoxide) in the coordination sphere of the terminal metal ion, its coordination will be six-coordinated polyhedron and the solvent molecule will be coordinated to the metal ion with longest coordination bond. If there is no solvent molecule in the coordination sphere of the terminal metal ion, its coordination will be five-coordinated polyhedron. A comparison of the properties of coordination bond length ranges (M_c —O and M_t —O/N), bond angle ranges (O— M_c —O and O/N— M_t —O/N) and M_c ··· M_t distances are given in Table 2, for the similar oxygen-bridged trinuclear complexes reported previously. The crystal structure may be stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

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Experimental

The bis-*N,N'*(salicylidene)-1,3-propanedimine Schiff base was prepared through the condensation reaction of 1,3-propane-diamine and salicylaldehyde in EtOH and it was reduced with NaBH₄ in MeOH until the solution totally colorless. The phenolic amine ligand was precipitated with the addition of the excess of ice. The complex was prepared with template method, since it was very cumbersome to isolate mononuclear bis-*N,N'*(2-oxybenzyl)-1,3-propanediaminato -nickel(II) complex. Bis-*N,N'*(2-hydroxybenzyl)-1,3-propanediamine (568 mg, 2 mmol) was dissolved in hot dmf (50 ml). NiCl₂·6H₂O solution (475 mg, 2 mmol) in hot methanol (20 ml) and ET₃N (0.5 ml) were added to it and the mixture was stirred for 10 min. Then, a solution of Zn(CH₃COO)₂·2H₂O (220 mg, 1 mmol) in hot MeOH (10 ml) was added and the resulting mixture was kept on the bench for 2–3 d. The blue crystals were filtered off, and dried on air (yield; 440 mg, 44%).

Refinement

H atoms were positioned geometrically, with N—H = 0.91 Å (for NH) and C—H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C},\text{N})$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

Figures

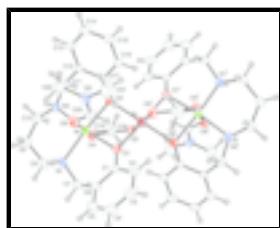


Fig. 1. The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 25% probability level [symmetry code: (i) $-x, -y, -z$].

Bis[μ_2 -*N,N'*-bis(2-oxidobenzyl)propane-1,3-diamine]- 1 κ^4 O,*N,N'*,O $^{\prime}$:2 κ^2 O,O $^{\prime}$; 2 κ^2 O,O $^{\prime}$:3 κ^4 O,*N,N'*,O $^{\prime}$ - bis(*N,N'*-dimethylformamide)-1 κ O,3 κ O-di- μ_2 - formato-1:2 κ^2 O:O $^{\prime}$;2:3 κ^2 O:O $^{\prime}$ - dinickel(II)zinc(II)

Crystal data

[Ni ₂ Zn(C ₁₇ H ₂₀ N ₂ O ₂) ₂ (C ₂ H ₃ O ₂) ₂ (C ₃ H ₇ NO) ₂]	$F_{000} = 1064$
$M_r = 1015.75$	$D_x = 1.444 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Cu $K\alpha$ radiation
Hall symbol: -P 2yn	$\lambda = 1.54184 \text{ \AA}$
$a = 10.3035 (13) \text{ \AA}$	Cell parameters from 15 reflections
$b = 17.894 (4) \text{ \AA}$	$\theta = 21.2\text{--}23.6^\circ$
$c = 12.584 (2) \text{ \AA}$	$\mu = 2.06 \text{ mm}^{-1}$
$\beta = 92.124 (13)^\circ$	$T = 298 (2) \text{ K}$
$V = 2318.5 (7) \text{ \AA}^3$	Needle, blue
$Z = 2$	$0.3 \times 0.1 \times 0.1 \text{ mm}$

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	$R_{\text{int}} = 0.033$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 74.2^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 4.3^\circ$
non-profiled ω scans	$h = -12 \rightarrow 0$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$k = 0 \rightarrow 22$
$T_{\text{min}} = 0.781$, $T_{\text{max}} = 0.814$	$l = -15 \rightarrow 15$
4319 measured reflections	3 standard reflections
4017 independent reflections	every 120 min
2381 reflections with $I > 2\sigma(I)$	intensity decay: 1%

Refinement

Refinement on F^2	Hydrogen site location: geomt
Least-squares matrix: full	H-atom parameters not refined
$R[F^2 > 2\sigma(F^2)] = 0.056$	$w = 1/[\sigma^2(F_o^2) + (0.0755P)^2 + 2.4395P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.176$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.32 \text{ e \AA}^{-3}$
4017 reflections	$\Delta\rho_{\text{min}} = -0.85 \text{ e \AA}^{-3}$
285 parameters	Extinction correction: ?
Primary atom site location: structure-invariant direct methods	Extinction coefficient: ?
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn	0	0	0	0.0520 (4)
Ni	0.03300 (9)	0.14110 (5)	-0.13520 (7)	0.0397 (3)
O1	0.0849 (4)	0.0312 (2)	-0.1398 (3)	0.0419 (9)
O2	-0.0889 (3)	0.1023 (2)	-0.0233 (3)	0.0400 (9)

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O3	0.1493 (4)	0.0619 (2)	0.0801 (3)	0.0434 (10)
O4	0.1688 (4)	0.1660 (2)	-0.0187 (3)	0.0469 (10)
O5	-0.1190 (4)	0.1291 (2)	-0.2579 (3)	0.0506 (11)
N1	0.1567 (5)	0.1698 (3)	-0.2571 (4)	0.0488 (13)
H1	0.1066	0.173	-0.318	0.059*
N2	-0.0410 (4)	0.2498 (3)	-0.1248 (4)	0.0438 (12)
H2	-0.0984	0.2555	-0.1809	0.053*
N3	-0.3129 (5)	0.0750 (3)	-0.2948 (4)	0.0551 (14)
C1	0.1100 (5)	-0.0014 (4)	-0.2319 (5)	0.0452 (14)
C2	0.0630 (6)	-0.0711 (4)	-0.2601 (6)	0.0602 (18)
H2A	0.0167	-0.0984	-0.2113	0.072*
C3	0.0832 (8)	-0.1012 (5)	-0.3592 (7)	0.081 (3)
H3	0.0491	-0.1478	-0.3773	0.098*
C4	0.1544 (10)	-0.0616 (6)	-0.4316 (6)	0.092 (3)
H4	0.1664	-0.0805	-0.4994	0.111*
C5	0.2067 (9)	0.0058 (6)	-0.4019 (6)	0.087 (3)
H5	0.2571	0.0313	-0.4498	0.104*
C6	0.1877 (6)	0.0369 (4)	-0.3051 (5)	0.0560 (17)
C7	0.2506 (6)	0.1092 (4)	-0.2724 (6)	0.064 (2)
H7A	0.3009	0.1016	-0.2066	0.077*
H7B	0.3102	0.1242	-0.3264	0.077*
C8	0.2227 (6)	0.2431 (4)	-0.2424 (5)	0.0617 (19)
H8A	0.2788	0.2516	-0.3014	0.074*
H8B	0.2769	0.2415	-0.1777	0.074*
C9	0.1284 (6)	0.3073 (4)	-0.2357 (5)	0.0598 (19)
H9A	0.0656	0.3037	-0.2948	0.072*
H9B	0.1757	0.3537	-0.2435	0.072*
C10	0.0561 (6)	0.3104 (3)	-0.1333 (5)	0.0559 (17)
H10A	0.1181	0.307	-0.0737	0.067*
H10B	0.0123	0.3582	-0.129	0.067*
C11	-0.1156 (6)	0.2582 (3)	-0.0284 (5)	0.0502 (16)
H11A	-0.1456	0.3094	-0.0236	0.06*
H11B	-0.0592	0.2482	0.0333	0.06*
C12	-0.2309 (5)	0.2065 (3)	-0.0269 (4)	0.0417 (14)
C13	-0.3568 (6)	0.2341 (4)	-0.0311 (5)	0.0596 (18)
H13	-0.3703	0.2853	-0.038	0.072*
C14	-0.4622 (6)	0.1873 (4)	-0.0254 (6)	0.065 (2)
H14	-0.5461	0.2065	-0.0297	0.078*
C15	-0.4417 (6)	0.1110 (4)	-0.0130 (5)	0.0599 (18)
H15	-0.5125	0.0792	-0.0075	0.072*
C16	-0.3172 (6)	0.0817 (4)	-0.0088 (5)	0.0505 (16)
H16	-0.305	0.0306	0.0012	0.061*
C17	-0.2101 (5)	0.1283 (3)	-0.0194 (4)	0.0402 (13)
C18	0.1879 (5)	0.1269 (4)	0.0641 (5)	0.0441 (14)
C19	0.2672 (7)	0.1630 (4)	0.1520 (5)	0.0622 (19)
H19A	0.2914	0.2125	0.1309	0.093*
H19B	0.3441	0.1339	0.1668	0.093*
H19C	0.2171	0.1658	0.2147	0.093*
C20	-0.1918 (6)	0.0743 (4)	-0.2577 (5)	0.0530 (17)

H20	-0.1591	0.0298	-0.2296	0.064*
C21	-0.3956 (9)	0.0099 (5)	-0.2891 (8)	0.105 (3)
H21A	-0.48	0.0215	-0.3196	0.158*
H21B	-0.4035	-0.0044	-0.2161	0.158*
H21C	-0.3583	-0.0305	-0.3278	0.158*
C22	-0.3717 (7)	0.1418 (4)	-0.3325 (7)	0.093 (3)
H22A	-0.4598	0.1317	-0.3558	0.14*
H22B	-0.3244	0.1608	-0.391	0.14*
H22C	-0.3709	0.1781	-0.2764	0.14*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn	0.0468 (7)	0.0557 (8)	0.0534 (7)	-0.0001 (6)	0.0004 (5)	0.0038 (6)
Ni	0.0329 (5)	0.0467 (6)	0.0395 (5)	-0.0020 (4)	0.0006 (4)	0.0042 (5)
O1	0.039 (2)	0.046 (2)	0.041 (2)	-0.0007 (18)	0.0041 (17)	-0.0011 (18)
O2	0.033 (2)	0.043 (2)	0.044 (2)	0.0033 (17)	0.0021 (17)	0.0048 (18)
O3	0.043 (2)	0.040 (2)	0.047 (2)	-0.0032 (19)	-0.0082 (18)	0.0009 (19)
O4	0.042 (2)	0.053 (3)	0.046 (2)	-0.0076 (19)	-0.0054 (18)	0.009 (2)
O5	0.044 (2)	0.060 (3)	0.047 (2)	-0.007 (2)	-0.0066 (19)	0.005 (2)
N1	0.040 (3)	0.068 (3)	0.039 (3)	-0.003 (3)	0.001 (2)	0.010 (3)
N2	0.040 (3)	0.047 (3)	0.044 (3)	-0.004 (2)	-0.002 (2)	0.006 (2)
N3	0.044 (3)	0.059 (4)	0.061 (3)	-0.006 (3)	-0.017 (3)	0.013 (3)
C1	0.035 (3)	0.054 (4)	0.046 (3)	0.011 (3)	0.000 (3)	-0.007 (3)
C2	0.047 (4)	0.070 (5)	0.064 (4)	0.009 (3)	0.003 (3)	-0.025 (4)
C3	0.065 (5)	0.089 (6)	0.089 (6)	0.025 (5)	-0.011 (5)	-0.039 (5)
C4	0.106 (7)	0.121 (8)	0.050 (5)	0.041 (7)	0.012 (5)	-0.021 (5)
C5	0.092 (6)	0.110 (8)	0.060 (5)	0.023 (6)	0.024 (4)	0.002 (5)
C6	0.046 (4)	0.078 (5)	0.044 (4)	0.019 (4)	0.008 (3)	0.004 (3)
C7	0.045 (4)	0.083 (5)	0.065 (5)	0.004 (4)	0.020 (3)	0.024 (4)
C8	0.047 (4)	0.079 (5)	0.058 (4)	-0.016 (4)	-0.001 (3)	0.022 (4)
C9	0.053 (4)	0.070 (5)	0.057 (4)	-0.017 (4)	-0.004 (3)	0.019 (4)
C10	0.057 (4)	0.044 (4)	0.065 (4)	-0.011 (3)	-0.008 (3)	0.005 (3)
C11	0.054 (4)	0.043 (4)	0.053 (4)	0.005 (3)	-0.003 (3)	-0.001 (3)
C12	0.035 (3)	0.047 (4)	0.043 (3)	0.007 (3)	0.002 (3)	0.003 (3)
C13	0.052 (4)	0.063 (5)	0.065 (4)	0.017 (3)	0.010 (3)	0.012 (4)
C14	0.038 (4)	0.090 (6)	0.068 (5)	0.016 (4)	0.007 (3)	0.006 (4)
C15	0.035 (3)	0.078 (5)	0.068 (5)	-0.003 (3)	0.011 (3)	-0.005 (4)
C16	0.035 (3)	0.053 (4)	0.064 (4)	-0.002 (3)	0.008 (3)	-0.003 (3)
C17	0.032 (3)	0.053 (4)	0.036 (3)	0.003 (3)	0.005 (2)	0.000 (3)
C18	0.032 (3)	0.054 (4)	0.047 (3)	0.003 (3)	-0.002 (2)	-0.003 (3)
C19	0.067 (5)	0.060 (4)	0.058 (4)	-0.014 (4)	-0.013 (4)	0.001 (3)
C20	0.046 (4)	0.063 (4)	0.049 (4)	0.012 (3)	-0.015 (3)	0.006 (3)
C21	0.087 (6)	0.088 (6)	0.137	-0.028 (5)	-0.043 (6)	0.025 (6)
C22	0.057 (5)	0.084 (6)	0.137 (8)	-0.003 (5)	-0.017 (5)	0.036 (6)

supplementary materials

Geometric parameters (\AA , $^\circ$)

Zn—O1 ⁱ	2.070 (4)	C5—H5	0.93
Zn—O1	2.070 (4)	C6—C7	1.497 (9)
Zn—O2	2.064 (4)	C7—H7A	0.97
Zn—O2 ⁱ	2.064 (4)	C7—H7B	0.97
Zn—O3 ⁱ	2.121 (4)	C8—C9	1.510 (9)
Zn—O3	2.121 (4)	C8—H8A	0.97
Zn—Ni ⁱ	3.0705 (11)	C8—H8B	0.97
Zn—Ni	3.0705 (11)	C9—C10	1.513 (9)
Ni—O1	2.039 (4)	C9—H9A	0.97
Ni—O2	2.043 (4)	C9—H9B	0.97
Ni—O4	2.038 (4)	C10—H10A	0.97
Ni—O5	2.168 (4)	C10—H10B	0.97
Ni—N1	2.094 (5)	C11—H11A	0.97
Ni—N2	2.095 (5)	C11—H11B	0.97
O1—C1	1.331 (7)	C12—C11	1.505 (8)
O2—C17	1.334 (6)	C12—C13	1.387 (8)
O3—C18	1.248 (7)	C13—C14	1.375 (9)
O4—C18	1.264 (7)	C13—H13	0.93
O5—C20	1.234 (7)	C14—H14	0.93
N1—C7	1.470 (8)	C15—C14	1.389 (9)
N1—C8	1.486 (8)	C15—H15	0.93
N1—H1	0.91	C16—C15	1.386 (8)
N2—C10	1.482 (7)	C16—H16	0.93
N2—C11	1.468 (7)	C17—C16	1.394 (8)
N2—H2	0.91	C17—C12	1.420 (8)
N3—C20	1.316 (7)	C18—C19	1.498 (8)
N3—C21	1.446 (9)	C19—H19A	0.96
N3—C22	1.414 (8)	C19—H19B	0.96
C1—C2	1.380 (8)	C19—H19C	0.96
C1—C6	1.419 (9)	C20—H20	0.93
C2—C3	1.382 (9)	C21—H21A	0.96
C2—H2A	0.93	C21—H21B	0.96
C3—C4	1.386 (12)	C21—H21C	0.96
C3—H3	0.93	C22—H22A	0.96
C4—H4	0.93	C22—H22B	0.96
C5—C4	1.367 (12)	C22—H22C	0.96
C5—C6	1.361 (10)		
O2—Zn—O2 ⁱ	180.0 (3)	C4—C5—H5	118.7
O2—Zn—O1 ⁱ	99.24 (15)	N2—C11—C12	112.7 (5)
O2 ⁱ —Zn—O1 ⁱ	80.76 (15)	N2—C11—H11A	109.1
O2—Zn—O1	80.76 (15)	C12—C11—H11A	109.1
O2 ⁱ —Zn—O1	99.24 (15)	N2—C11—H11B	109.1
O1 ⁱ —Zn—O1	180.0 (3)	C12—C11—H11B	109.1
O2—Zn—O3 ⁱ	94.90 (15)	H11A—C11—H11B	107.8

O2 ⁱ —Zn—O3 ⁱ	85.10 (15)	N1—C8—C9	112.7 (5)
O1 ⁱ —Zn—O3 ⁱ	86.64 (14)	N1—C8—H8A	109
O1—Zn—O3 ⁱ	93.36 (14)	C9—C8—H8A	109
O2—Zn—O3	85.10 (15)	N1—C8—H8B	109
O2 ⁱ —Zn—O3	94.90 (15)	C9—C8—H8B	109
O1 ⁱ —Zn—O3	93.36 (14)	H8A—C8—H8B	107.8
O1—Zn—O3	86.64 (14)	C8—C9—C10	114.4 (5)
O3 ⁱ —Zn—O3	180.0 (3)	C8—C9—H9A	108.7
O2—Zn—Ni ⁱ	138.64 (10)	C10—C9—H9A	108.7
O2 ⁱ —Zn—Ni ⁱ	41.36 (10)	C8—C9—H9B	108.7
O1 ⁱ —Zn—Ni ⁱ	41.27 (11)	C10—C9—H9B	108.7
O1—Zn—Ni ⁱ	138.73 (11)	H9A—C9—H9B	107.6
O3 ⁱ —Zn—Ni ⁱ	74.97 (10)	C5—C6—C1	119.1 (8)
O3—Zn—Ni ⁱ	105.03 (10)	C5—C6—C7	121.7 (7)
O2—Zn—Ni	41.36 (10)	C1—C6—C7	119.2 (6)
O2 ⁱ —Zn—Ni	138.64 (10)	N1—C7—C6	113.1 (5)
O1 ⁱ —Zn—Ni	138.73 (11)	N1—C7—H7A	109
O1—Zn—Ni	41.27 (11)	C6—C7—H7A	109
O3 ⁱ —Zn—Ni	105.03 (10)	N1—C7—H7B	109
O3—Zn—Ni	74.97 (10)	C6—C7—H7B	109
Ni ⁱ —Zn—Ni	180.00 (3)	H7A—C7—H7B	107.8
O4—Ni—O1	93.30 (16)	C18—O4—Ni	123.6 (4)
O4—Ni—O2	90.10 (15)	C18—O3—Zn	129.8 (4)
O1—Ni—O2	81.99 (15)	C20—N3—C22	121.0 (6)
O4—Ni—N1	93.04 (18)	C20—N3—C21	121.9 (6)
O1—Ni—N1	92.76 (18)	C22—N3—C21	116.8 (6)
O2—Ni—N1	174.03 (18)	O3—C18—O4	127.5 (6)
O4—Ni—N2	89.64 (18)	O3—C18—C19	116.8 (5)
O1—Ni—N2	173.45 (17)	O4—C18—C19	115.8 (6)
O2—Ni—N2	92.17 (17)	C16—C15—C14	120.9 (6)
N1—Ni—N2	92.9 (2)	C16—C15—H15	119.5
O4—Ni—O5	172.84 (17)	C14—C15—H15	119.5
O1—Ni—O5	93.85 (16)	O5—C20—N3	124.2 (6)
O2—Ni—O5	90.62 (15)	O5—C20—H20	117.9
N1—Ni—O5	86.90 (17)	N3—C20—H20	117.9
N2—Ni—O5	83.21 (17)	C14—C13—C12	121.4 (7)
O4—Ni—Zn	82.44 (11)	C14—C13—H13	119.3
O1—Ni—Zn	42.04 (10)	C12—C13—H13	119.3
O2—Ni—Zn	41.86 (10)	N2—C10—C9	112.9 (5)
N1—Ni—Zn	133.64 (15)	N2—C10—H10A	109
N2—Ni—Zn	132.91 (14)	C9—C10—H10A	109
O5—Ni—Zn	102.77 (12)	N2—C10—H10B	109
C20—O5—Ni	120.1 (4)	C9—C10—H10B	109
C17—O2—Ni	120.5 (3)	H10A—C10—H10B	107.8
C17—O2—Zn	135.6 (4)	C1—C2—C3	121.4 (7)
Ni—O2—Zn	96.77 (15)	C1—C2—H2A	119.3

supplementary materials

C1—O1—Ni	120.4 (4)	C3—C2—H2A	119.3
C1—O1—Zn	136.4 (4)	N3—C22—H22A	109.5
Ni—O1—Zn	96.69 (16)	N3—C22—H22B	109.5
C7—N1—C8	111.5 (5)	H22A—C22—H22B	109.5
C7—N1—Ni	109.7 (4)	N3—C22—H22C	109.5
C8—N1—Ni	114.4 (4)	H22A—C22—H22C	109.5
C7—N1—H1	106.9	H22B—C22—H22C	109.5
C8—N1—H1	106.9	C13—C14—C15	119.1 (6)
Ni—N1—H1	106.9	C13—C14—H14	120.4
C11—N2—C10	111.1 (5)	C15—C14—H14	120.4
C11—N2—Ni	110.5 (3)	C2—C3—C4	119.7 (8)
C10—N2—Ni	115.3 (4)	C2—C3—H3	120.1
C11—N2—H2	106.5	C4—C3—H3	120.1
C10—N2—H2	106.5	N3—C21—H21A	109.5
Ni—N2—H2	106.5	N3—C21—H21B	109.5
O2—C17—C16	122.7 (5)	H21A—C21—H21B	109.5
O2—C17—C12	118.7 (5)	N3—C21—H21C	109.5
C16—C17—C12	118.6 (5)	H21A—C21—H21C	109.5
O1—C1—C2	122.8 (6)	H21B—C21—H21C	109.5
O1—C1—C6	119.1 (6)	C5—C4—C3	119.0 (8)
C2—C1—C6	118.1 (6)	C5—C4—H4	120.5
C13—C12—C17	119.5 (6)	C3—C4—H4	120.5
C13—C12—C11	121.2 (6)	C18—C19—H19A	109.5
C17—C12—C11	119.3 (5)	C18—C19—H19B	109.5
C15—C16—C17	120.3 (6)	H19A—C19—H19B	109.5
C15—C16—H16	119.8	C18—C19—H19C	109.5
C17—C16—H16	119.8	H19A—C19—H19C	109.5
C6—C5—C4	122.5 (9)	H19B—C19—H19C	109.5
C6—C5—H5	118.7		
O2—Zn—Ni—O4	98.7 (2)	N2—Ni—N1—C7	167.5 (4)
O2 ⁱ —Zn—Ni—O4	-81.3 (2)	O5—Ni—N1—C7	-109.4 (4)
O1 ⁱ —Zn—Ni—O4	76.47 (19)	Zn—Ni—N1—C7	-4.7 (5)
O1—Zn—Ni—O4	-103.53 (19)	O4—Ni—N1—C8	-48.4 (4)
O3 ⁱ —Zn—Ni—O4	178.82 (16)	O1—Ni—N1—C8	-141.9 (4)
O3—Zn—Ni—O4	-1.18 (16)	N2—Ni—N1—C8	41.4 (4)
O2—Zn—Ni—O1	-157.8 (2)	O5—Ni—N1—C8	124.4 (4)
O2 ⁱ —Zn—Ni—O1	22.2 (2)	Zn—Ni—N1—C8	-130.9 (4)
O1 ⁱ —Zn—Ni—O1	180	O4—Ni—N2—C11	-74.7 (4)
O3 ⁱ —Zn—Ni—O1	-77.64 (19)	O2—Ni—N2—C11	15.4 (4)
O3—Zn—Ni—O1	102.36 (19)	N1—Ni—N2—C11	-167.7 (4)
O2 ⁱ —Zn—Ni—O2	180	O5—Ni—N2—C11	105.8 (4)
O1 ⁱ —Zn—Ni—O2	-22.2 (2)	Zn—Ni—N2—C11	4.6 (5)
O1—Zn—Ni—O2	157.8 (2)	O4—Ni—N2—C10	52.3 (4)
O3 ⁱ —Zn—Ni—O2	80.15 (19)	O2—Ni—N2—C10	142.4 (4)
O3—Zn—Ni—O2	-99.85 (19)	N1—Ni—N2—C10	-40.7 (4)
O2—Zn—Ni—N1	-174.3 (2)	O5—Ni—N2—C10	-127.2 (4)
O2 ⁱ —Zn—Ni—N1	5.7 (2)	Zn—Ni—N2—C10	131.7 (4)

O1 ⁱ —Zn—Ni—N1	163.4 (2)	Ni—O2—C17—C16	134.1 (5)
O1—Zn—Ni—N1	−16.6 (2)	Zn—O2—C17—C16	−8.7 (8)
O3 ⁱ —Zn—Ni—N1	−94.2 (2)	Ni—O2—C17—C12	−45.6 (6)
O3—Zn—Ni—N1	85.8 (2)	Zn—O2—C17—C12	171.6 (4)
O2—Zn—Ni—N2	16.3 (2)	Ni—O1—C1—C2	−135.3 (5)
O2 ⁱ —Zn—Ni—N2	−163.7 (2)	Zn—O1—C1—C2	9.0 (9)
O1 ⁱ —Zn—Ni—N2	−5.9 (2)	Ni—O1—C1—C6	44.3 (7)
O1—Zn—Ni—N2	174.1 (2)	Zn—O1—C1—C6	−171.4 (4)
O3 ⁱ —Zn—Ni—N2	96.4 (2)	O2—C17—C12—C13	175.2 (5)
O3—Zn—Ni—N2	−83.6 (2)	C16—C17—C12—C13	−4.5 (8)
O2—Zn—Ni—O5	−76.33 (19)	O2—C17—C12—C11	−5.8 (8)
O2 ⁱ —Zn—Ni—O5	103.67 (19)	C16—C17—C12—C11	174.5 (5)
O1 ⁱ —Zn—Ni—O5	−98.5 (2)	O2—C17—C16—C15	−175.2 (6)
O1—Zn—Ni—O5	81.5 (2)	C12—C17—C16—C15	4.4 (9)
O3 ⁱ —Zn—Ni—O5	3.82 (16)	C10—N2—C11—C12	169.4 (5)
O3—Zn—Ni—O5	−176.18 (16)	Ni—N2—C11—C12	−61.3 (5)
O1—Ni—O5—C20	42.3 (5)	C13—C12—C11—N2	−114.2 (6)
O2—Ni—O5—C20	−39.7 (5)	C17—C12—C11—N2	66.8 (7)
N1—Ni—O5—C20	134.8 (5)	C7—N1—C8—C9	175.1 (5)
N2—Ni—O5—C20	−131.8 (5)	Ni—N1—C8—C9	−59.6 (6)
Zn—Ni—O5—C20	0.7 (5)	N1—C8—C9—C10	72.0 (7)
O4—Ni—O2—C17	126.7 (4)	C4—C5—C6—C1	−0.5 (12)
O1—Ni—O2—C17	−140.0 (4)	C4—C5—C6—C7	177.3 (8)
N2—Ni—O2—C17	37.0 (4)	O1—C1—C6—C5	−175.9 (6)
O5—Ni—O2—C17	−46.2 (4)	C2—C1—C6—C5	3.7 (9)
Zn—Ni—O2—C17	−154.8 (5)	O1—C1—C6—C7	6.3 (9)
O4—Ni—O2—Zn	−78.51 (17)	C2—C1—C6—C7	−174.1 (6)
O1—Ni—O2—Zn	14.81 (15)	C8—N1—C7—C6	−170.6 (5)
N2—Ni—O2—Zn	−168.16 (17)	Ni—N1—C7—C6	61.6 (6)
O5—Ni—O2—Zn	108.61 (16)	C5—C6—C7—N1	115.0 (7)
O1 ⁱ —Zn—O2—C17	−46.3 (5)	C1—C6—C7—N1	−67.2 (8)
O1—Zn—O2—C17	133.7 (5)	O1—Ni—O4—C18	−46.3 (5)
O3 ⁱ —Zn—O2—C17	41.1 (5)	O2—Ni—O4—C18	35.7 (5)
O3—Zn—O2—C17	−138.9 (5)	N1—Ni—O4—C18	−139.2 (5)
Ni ⁱ —Zn—O2—C17	−31.6 (6)	N2—Ni—O4—C18	127.9 (5)
Ni—Zn—O2—C17	148.4 (6)	Zn—Ni—O4—C18	−5.6 (4)
O1 ⁱ —Zn—O2—Ni	165.37 (14)	O2—Zn—O3—C18	−31.5 (5)
O1—Zn—O2—Ni	−14.63 (14)	O2 ⁱ —Zn—O3—C18	148.5 (5)
O3 ⁱ —Zn—O2—Ni	−107.25 (16)	O1 ⁱ —Zn—O3—C18	−130.5 (5)
O3—Zn—O2—Ni	72.75 (16)	O1—Zn—O3—C18	49.5 (5)
Ni ⁱ —Zn—O2—Ni	180	Ni ⁱ —Zn—O3—C18	−170.7 (5)
O4—Ni—O1—C1	−129.0 (4)	Ni—Zn—O3—C18	9.3 (5)
O2—Ni—O1—C1	141.3 (4)	Zn—O3—C18—O4	−18.1 (9)
N1—Ni—O1—C1	−35.8 (4)	Zn—O3—C18—C19	162.7 (4)
O5—Ni—O1—C1	51.2 (4)	Ni—O4—C18—O3	15.1 (9)
Zn—Ni—O1—C1	156.1 (5)	Ni—O4—C18—C19	−165.7 (4)

supplementary materials

O4—Ni—O1—Zn	74.88 (16)	C17—C16—C15—C14	−1.5 (10)
O2—Ni—O1—Zn	−14.76 (14)	Ni—O5—C20—N3	150.1 (5)
N1—Ni—O1—Zn	168.08 (17)	C22—N3—C20—O5	−4.1 (11)
O5—Ni—O1—Zn	−104.84 (16)	C21—N3—C20—O5	−177.7 (7)
O2—Zn—O1—C1	−134.9 (5)	C17—C12—C13—C14	1.7 (10)
O2 ⁱ —Zn—O1—C1	45.1 (5)	C11—C12—C13—C14	−177.3 (6)
O3 ⁱ —Zn—O1—C1	−40.5 (5)	C11—N2—C10—C9	−175.7 (5)
O3—Zn—O1—C1	139.5 (5)	Ni—N2—C10—C9	57.6 (6)
Ni ⁱ —Zn—O1—C1	30.5 (6)	C8—C9—C10—N2	−70.7 (7)
Ni—Zn—O1—C1	−149.5 (6)	O1—C1—C2—C3	175.4 (6)
O2—Zn—O1—Ni	14.66 (14)	C6—C1—C2—C3	−4.2 (9)
O2 ⁱ —Zn—O1—Ni	−165.34 (14)	C12—C13—C14—C15	1.2 (11)
O3 ⁱ —Zn—O1—Ni	109.08 (16)	C16—C15—C14—C13	−1.4 (11)
O3—Zn—O1—Ni	−70.92 (16)	C1—C2—C3—C4	1.4 (11)
Ni ⁱ —Zn—O1—Ni	180	C6—C5—C4—C3	−2.4 (14)
O4—Ni—N1—C7	77.7 (4)	C2—C3—C4—C5	2.0 (13)
O1—Ni—N1—C7	−15.7 (4)		

Symmetry codes: (i) $-x, -y, -z$.

Hydrogen-bond geometry (\AA , °)

$D\cdots H$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
C2—H2A···O3 ⁱⁱ	0.93	2.51	3.211 (7)	133
C16—H16···O3 ⁱⁱ	0.93	2.55	3.241 (8)	132
C20—H20···O3 ⁱⁱ	0.93	2.50	3.325 (8)	149

Symmetry codes: (ii) $-x, -y, -z$.

?

Complex	$M_c\cdots M_t$	$M_c\cdots O$	$O\cdots M_c$	$M_t\cdots O/N$	$O/N\cdots M_t\cdots O/N$
(I)	3.0705 (11)	2.064 (4)	80.76 (15)	2.038 (4)	81.99 (15)
		2.121 (4)	99.24 (15)	2.168 (4)	93.85 (16)
(II)	3.0520 (8)	2.098 (3)	77.5 (1)	1.975 (4)	80.3 (1)
		2.124 (4)	102.5 (1)	2.071 (4)	104.9 (2)
(III)	3.0017 (6)	2.055 (2)	76.04 (9)	1.941 (2)	81.5 (1)
		2.188 (2)	93.74 (9)	2.355 (3)	97.0 (1)
(IV)	Not_given	2.083 (6)	79.0 (2)	1.995 (5)	82.1 (2)
		2.118 (5)	84.5 (2)	2.179 (6)	97.8 (3)
(V)	3.043 (2)	2.024 (3)	79.4 (1)	2.010 (3)	79.4 (1)
		2.098 (3)	87.1 (1)	2.254 (3)	96.2 (2)
(VI)	2.9967 (4)	2.048 (2)	78.70 (8)	2.003 (2)	80.88 (8)
		2.103 (2)	85.86 (9)	2.152 (2)	98.5 (1)
(VII)	3.0556 (5)	2.0705 (19)	78.89 (8)	2.0082 (19)	81.65 (8)
		2.082 (2)	92.92 (8)	2.186 (2)	97.95 (10)
(VIII)	3.0601 (6)	2.052 (2)	80.96 (9)	2.029 (2)	81.79 (9)
		2.102 (2)	86.57 (9)	2.165 (2)	96.95 (10)
(IX)	3.0857 (14)	2.075 (3)	80.34 (12)	2.037 (3)	81.85 (12)

supplementary materials

2.160 (3) 99.66 (12) 2.147 (3) 93.54 (13)

Notes: (II) $[\text{Zn}\{\text{Zn}(\text{SALPD}^{2-})(\text{acetato})\}_2]$ (Ülkü *et al.*, 2001); (III) $[\text{Zn}\{\text{Cu}(\text{SALPD}^{2-})(\text{nitrato})\}_2]$ (Ülkü *et al.*, 1999); (IV) $[\text{Co}\{\text{Ni}(\text{SALPD}^{2-})(\text{nitrito})(\text{DMF})\}_2]$ (Atakol *et al.*, 1999); (V) $[\text{Ni}\{\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{DMSO})\}_2]$ (Ülkü *et al.*, 1997); (VI) $[\text{Cu}\{\text{Ni}(\text{SALPD}^{2-})(\text{nitrito})(\text{DMF})\}_2]$ (Tahir *et al.*, 1998); (VII) $[\text{Cu}\{\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{DMF})\}_2]$ (Ariç *et al.*, 2001); (VIII) $[\text{Ni}\{\text{Ni}(\text{SALPD}^{2-})(\text{acetato})(\text{DMF})\}_2]$ (Tatar & Atakol, 2002); (IX) $[\text{Ni}\{\text{Ni}(\text{DMLH}_2)(\text{formato})(\text{DMF})\}_2]$, where $\text{DMLH}_2 = (\text{C}_{19}\text{H}_{24}\text{N}_2\text{O}_2)$ (Tatar *et al.*, 2007).

supplementary materials

Fig. 1

