Sr1—O11	2.686 (2)	C13—C14	1.501 (5)
Sr1—ON11	2.7331 (19)	O21—C23	1.255 (2)
Sr1—ON12	2.7881 (19)	O22—C23	1.251 (3)
Sr1—ON22^{ii}	2.813 (2)	C23—C24	1.504 (5)
Sr2—O11	2.5074 (15)	N1—ON12	1.236 (3)
Sr2—O22^{iii}	2.5238 (17)	N1—ON13	1.253 (3)
Sr2—O21	2.622 (2)	N1—ON11	1.260 (2)
Sr2—O*W*2	2.6373 (19)	N2—ON23	1.229 (3)
Sr2—ON11^{iv}	2.6557 (19)	N2—ON21	1.247 (3)
Sr2—ON21	2.6692 (19)	N2—ON22	1.248 (3)
O12—C13—O11	121.4 (2)	C24—C23—Sr2	169.2 (2)
O12—C13—C14	120.2 (2)	ON12—N1—ON13	122.5 (2)
O11—C13—C14	118.4 (3)	ON12—N1—ON11	119.2 (2)
C14—C13—Sr1	168.2 (2)	ON13—N1—ON11	118.3 (2)
O22—C23—O21	121.5 (3)	ON23—N2—ON21	118.5 (2)
O22—C23—C24	120.4 (2)	ON23—N2—ON22	121.8 (2)
O21—C23—C24	118.1 (2)	ON21—N2—ON22	119.7 (2)

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

The structure was solved using coordinates for the Sr atoms obtained from direct methods and subsequent full-matrix least-squares refinements on the basis of the results of the difference Fourier summations. All non-H atoms were refined anisotropically. All H atoms were located from difference maps and the H atoms were refined isotropically using an independent distance restraint for each water molecule and the CH_3 groups.

Data collection: *DIF*4 (Stoe & Cie, 1991*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991*b*). Program(s) used to solve structure: *SHELXS*96 (Sheldrick, 1996*b*). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996*a*). Molecular graphics: *ATOMS*3.2 (Dowty, 1995). Software used to prepare material for publication: *SHELXL*96.

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Bis{(μ-acetato)[μ-bis(salicylidene)-1,3-propanediaminato](*N*,*N*-dimethylformamide)nickel(II)}cadmium(II)†

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Abstract

The structure of the title compound, $[Cd(C_2H_3O_2)_2\{Ni(C_3H_7NO)(C_{17}H_{16}N_2O_2)\}_2]$, consists of a linear heterotrinuclear unit with a central Cd^{II} ion. Both this ion and the terminal Ni^{II} ions have pseudo-octahedral coordination. Each pair of metal ions is triply bridged *via* O atoms from *N*,*N'*-bis(salicylidene)-1,3-propanediaminato ($SALPD^{2-}$) ligands and acetate groups. The coordination around the Cd^{II} ion involves four O atoms from two $SALPD^{2-}$ and two acetate ligands. The coordination of the Ni^{II} ion is provided by the two O and two N atoms from a $SALPD^{2-}$ ligand and one O atom each from an acetate and a dimethylformamide ligand. The Cd···Ni distance is 3.2274 (5) Å.

Comment

Trinuclear metal complexes based on Schiff base ligands are of interest because of their magnetic super-exchange interactions between bridged metal ions. The synthesis and magnetic properties of trinuclear complexes with the general formulae $[M\mathrm{Cu}_2(\mathrm{CH_3CO_2})_2(\mathrm{SALPD})_2]$ (M = Mg, Mn, Co, Ni, Cu or Zn) and $[M\mathrm{Ni}_2(\mathrm{CH_3CO_2})_2(\mathrm{SALPD})_2]$ (M = Mg, Mn, Co, or Ni), where H_2SALPD represents *N*,*N'*-bis(salicylidene)-1,3-propanediamine, have been reported previously (Fukuhara *et al.*, 1990). From this group of compounds, only the structure of $[\mathrm{Zn}\{(\mathrm{CH_3CO_2})(\mathrm{SALPD})\mathrm{Cu}\}_2]$ has been determined by X-ray diffraction (Fukuhara *et al.*, 1990). Structural studies of the complexes formulated as $M_3(\mathrm{SALPN})_2(\mathrm{CH_3CO_2})_2$.2dmf [$M$ = Co, Fe; SALPN = *N*,*N'*-bis(salicylidene)-2,2-dimethylpropylenediamine; dmf = dimethylformamide] have also been reported (Gerli, Hagen & Marzilli, 1991). We describe

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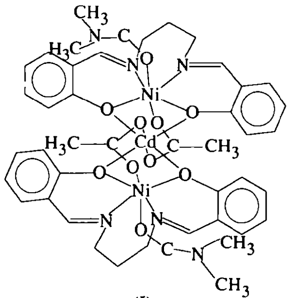
† Alternative name: bis(μ-acetato)-1:3κ^2*O*:*O'*;2:3κ^2*O*:*O'*-bis(*N*,*N*-dimethylformamide)-1κ*O*,2κ*O*-bis{μ-2,2'-[1,3-propanediylbis(nitrilomethylidyne)]diphenolato}-1κ^4*N*,*N'O*,*O'*:3κ^2*O*,*O'*;2κ^4*N*,*N'O*,*O'*:-3κ^2*O*,*O'*-cadmium(II)dinickel(II).

here a new linear heterometallic trinuclear complex, $[Cd\{(CH_3CO_2)(SALPD)Ni(dmf)\}_2]$, (I).



(I)

The structure consists of centrosymmetric trinuclear species with the central Cd ion occupying an inversion centre. The coordination around cadmium (Fig. 1) is a distorted octahedron involving four bridging O atoms from two SALPD^{2-} ligands in the equatorial plane and an O atom from each of the two bridging acetate groups in the apical positions. The two inversion-related Ni^{II} ions also have a pseudo-octahedral environment, with the four equatorial positions occupied by the two N and the two O atoms of the SALPD^{2-} ligand. The coordination sphere is completed by the O atom of the bridging acetate on one side and by a dmf ligand on the other. The dihedral angle between the equatorial planes of the neighbouring polyhedra (O1, O2, O1^i, O2^i and O1, O2, N1, N2) is 23.10 (7)° [symmetry code: (i) $-x$, $-y$, $-z$].

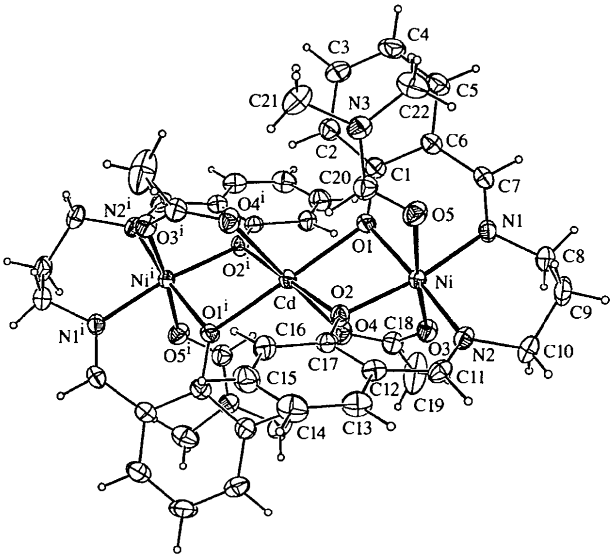


Fig. 1. The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are shown as small circles with arbitrary radii. [Symmetry code: (i) $-x$, $-y$, $-z$.]

The conformation of the SALPD^{2-} ligand resembles a flat shell. The dihedral angle between the best plane of the salicylaldimine residue (N1, C1–C7, O1) and the equatorial plane (N1, N2, O1, O2) around the Ni^{II} centre is 32.80 (7)°. The corresponding angle between the other salicylaldimine residue (N2, C11–C17, O2) and the equatorial plane around the Ni ion is 26.38 (6)°. In the Cd^{II} coordination sphere, the Cd—O(SALPD^{2-}) bond lengths [2.260 (1)–2.263 (2) Å] are shorter than the Cd—O(acetate) distances [2.293 (2) Å]. The six-membered chelate ring (Ni, N1, C8, C9, C10, N2) has a chair conformation. The distances of the two *para*-positioned chair atoms, Ni and C9, from the plane of the other four atoms are 0.4912 (2) and −0.727 (3) Å, respectively. The Ni—O bond lengths are in the range 2.012 (1)–2.190 (2) Å, the longest distance belonging to the Ni—O(dmf) bond. The Ni—N1 and Ni—N2 bond lengths are 2.039 (2) and 2.021 (2) Å, respectively. The Ni atom is 0.066 (2) Å out of the equatorial best plane defined by N1, O1, N2 and O2. The Cd···Ni distance is 3.2274 (5) Å. The coordination observed in (I) is similar to that reported by Gerli, Hagen & Marzilli (1991) in the homo-trinuclear species $M_3(SALPN)_2(CH_3CO_2)_2.2dmf$ (M_3 = Co_3 or Fe_3).

Experimental

To a solution of *N*,*N*′-bis(salicylidene)-1,3-propanediamine (0.282 g, 1 mmol) in hot ethanol (50 ml), 20% ammonia solution (10 ml) was added and the mixture was heated to boiling point. Then a solution of $NiCl_2.6H_2O$ (0.238 g, 1 mmol) in hot water (30 ml) was added and the resulting mixture set aside. After 2 h, the light-green Ni complex was filtered and dried at 423 K for 3 h. 0.339 g (1 mmol) of this complex was dissolved in 60 ml hot DMF and the temperature of the solution was increased to 383 K. Dropwise addition of $[Cd(CH_3COO)_2]$ (0.115 g, 0.5 mmol) dissolved in 20 ml hot ethanol resulted in the formation of green crystals after 24 h.

Crystal data

$[CdNi_2(C_2H_3O_2)_2(C_{17}H_{16}N_2O_2)_2(C_3H_7NO)_2]$
M_r = 1054.759
Triclinic
$P\bar{1}$
a = 9.616 (2) Å
b = 10.672 (2) Å
c = 12.344 (3) Å
α = 112.870 (2)°
β = 100.174 (3)°
γ = 90.257 (2)°
V = 1145.2 (6) Å^3
Z = 1
D_x = 1.529 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 17.90–18.09°
μ = 1.336 mm^{-1}
T = 294 K
Parallelepiped
0.25 × 0.20 × 0.15 mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer

3270 reflections with $I > 3\sigma(I)$

$\omega/2\theta$ scans
Absorption correction:
empirical *via* ψ scans
(Fair, 1990)
$T_{min} = 0.779$, $T_{max} = 0.817$
4097 measured reflections
3867 independent reflections

$R_{int} = 0.011$
$\theta_{max} = 24.66°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 0$
$l = -13 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: −0.4%

Refinement

Refinement on F
$R = 0.022$
$wR = 0.031$
$S = 1.16$
3170 reflections
286 parameters
H atoms riding
$w = 4F^2/[\sigma(I)^2 + (PF^2)^2]$,
if $F^2 <$ cutoff$[\sigma(I)^2 + (pF^2)^2]$, then the reflection is omitted
($p = 0.04$, cutoff = 3.0)

$(\Delta/\sigma)_{max} = 0.0001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.09$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Ni—Cd	3.2274 (5)	Ni—N1	2.039 (2)
Cd—O1	2.260 (1)	Ni—N2	2.021 (2)
Cd—O2	2.263 (2)	O1—C1	1.309 (2)
Cd—O4	2.293 (2)	O2—C17	1.319 (3)
Ni—O1	2.030 (2)	O3—C18	1.246 (3)
Ni—O2	2.012 (1)	O4—C18	1.257 (3)
Ni—O3	2.047 (2)	O5—C20	1.230 (3)
Ni—O5	2.190 (2)		
O1—Cd—O2	73.66 (5)	O5—Ni—N2	88.79 (7)
O1—Cd—O4	84.68 (5)	N1—Ni—N2	97.66 (7)
O2—Cd—O4	86.61 (6)	Cd—O1—Ni	97.44 (5)
O1—Ni—O2	84.26 (6)	Cd—O1—C1	134.0 (1)
O1—Ni—O3	92.88 (7)	Ni—O1—C1	124.1 (1)
O1—Ni—O5	87.98 (7)	Cd—O2—Ni	97.86 (6)
O1—Ni—N1	88.09 (7)	Cd—O2—C17	134.8 (1)
O1—Ni—N2	173.15 (7)	Ni—O2—C17	125.6 (1)
O2—Ni—O3	94.59 (6)	Ni—O3—C18	129.7 (1)
O2—Ni—O5	91.03 (6)	Cd—O4—C18	128.7 (2)
O2—Ni—N1	171.47 (6)	Ni—O5—C20	118.4 (2)
O2—Ni—N2	89.76 (6)	Ni—N1—C7	123.7 (2)
O3—Ni—O5	174.37 (5)	Ni—N1—C8	118.3 (1)
O3—Ni—N1	89.52 (6)	Ni—N2—C10	118.7 (1)
O3—Ni—N2	90.91 (7)	Ni—N2—C11	124.0 (2)
O5—Ni—N1	84.95 (6)		

All non-H atoms were refined with anisotropic displacement parameters. The H atoms were placed geometrically 0.95 Å from their parent C atoms with $U_{iso}(H) = 1.3U_{eq}(C)$. After refining the coordinates of methyl H atoms for a few cycles, a riding model was used for all H atoms.

Data collection: *CAD*-4 *EXPRESS* (Enraf-Nonius, 1994). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *MolEN* version of *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1135). Services for accessing these data are described at the back of the journal.

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Bis[*N*-(2,6-diisopropylphenyl)amido][*N*-(2,6-diisopropylphenyl)imido]bis(pyridine-*N*)-zirconium

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Abstract

The title compound, $[Zr(C_{12}H_{17}N)(C_{12}H_{18}N)_2(C_5H_5N)_2]$, contains a five-coordinate Zr atom with a distorted square-base pyramidal geometry comprising one *N*-(2,6-diisopropylphenyl)imido, two *N*-(2,6-diisopropylphenyl)amido and two pyridine ligands. There are two independent molecules in the asymmetric unit.

Comment

The chemistry of transition metal imido complexes continues to attract considerable interest (Wigley, 1994). Zirconium terminal imido complexes were first reported in 1988 (Walsh, Hollander & Bergman, 1988) and are still relatively rare species. We have been interested in developing Group 4–imido chemistry (Blake, Mountford, Nikonov & Swallow, 1996, and references therein) and during the course of our studies obtained crystals of the previously described (Arney, Bruck, Huber & Wigley, 1992) zirconium–imido complex $[Zr(N\text{-}2,6\text{-}C_6H_3{}^iPr_2)(NH\text{-}2,6\text{-}C_6H_3{}^iPr_2)_2(py)_2]$ (py = pyridine), (I).