Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Cengiz Arıcı,[a]* Dinçer Ülkü,[a] M. Nawaz Tahir[a]† and Orhan Atakol[b]

[a]Department of Engineering Physics, Hacettepe University, Beytepe 06532, Ankara, Turkey, and [b]Department of Chemistry, Ankara University, Tandoğan 06100, Ankara, Turkey

† Present adress: Department of Physics, Government College, Jhang, Punjab, Pakistan.

Correspondence e-mail: arici@hacettepe.edu.tr

Key indicators

Single-crystal X-ray study
T = 301 K
Mean σ(C–C) = 0.005 Å
R factor = 0.032
wR factor = 0.085
Data-to-parameter ratio = 15.0

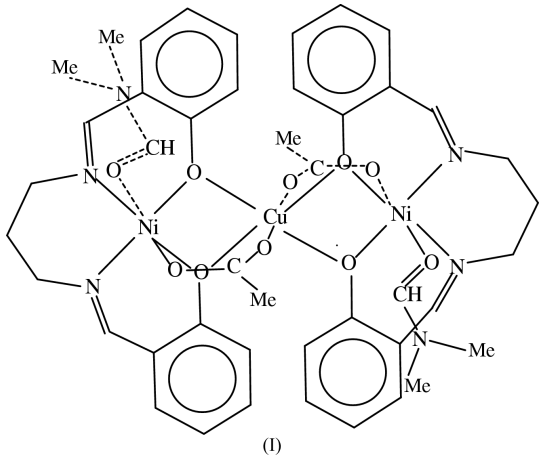
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $[CuNi_2(C_{17}H_{16}N_2O_2)_2(C_2H_3O_2)_2(C_3H_7NO)_2]$, contains a linear hetero-trinuclear complex with a central Cu^{II} ion located on an inversion center. The central copper(II) ion as well as the terminal nickel(II) ions have distorted octahedral coordination. Four O atoms from two bis(salicylidene)-1,3-propanediaminate ($SALPD^{2-}$, $C_{17}H_{16}N_2O_2^{2-}$) ligands and one O atom from each bridging acetate group constitute the octahedral coordination sphere around the Cu^{II} atom. The six nearest neigbors around the Ni^{II} atom are the two O and two N atoms of a $SALPD^{2-}$ ligand and one O atom each from an acetate and a dimethylformamide group. The Cu—Ni pairs are triply bridged *via* O atoms from $SALPD^{2-}$ ligands and acetate groups. The Cu···Ni distance is 3.0556 (5) Å. The structure is isomorphous with that of $[CdNi_2(C_{17}H_{16}N_2O_2)_2(C_2H_3O_2)_2(C_3H_7NO)_2]$.

Received 25 April 2001
Accepted 8 May 2001
Online 15 June 2001

Comment

Trinuclear linear homo- or heteronuclear complexes based on Schiff base ligands are of interest because of their magnetic super-exchange interactions between bridged metal ions (Fukuhara *et al.*, 1990). In these complexes, various combinations of metal ions in the central and terminal locations, as well as the μ-bridges, such as acetate or nitrite anions, are possible. Among the trinuclear complexes with similar formula reported from this laboratory previously (Ülkü *et al.*, 1997; Tahir *et al.*, 1998; Atakol *et al.*, 1999, and references therein), the title complex, (I), resembles $[CdNi_2(C_{17}H_{16}N_2O_2)_2(C_2H_3O_2)_2(C_3H_7NO)_2]$ (Ülkü *et al.*, 1997), (II), more than any other trinuclear complex. The crystal data indicate that (I) and (II) are isomorphous.



(I)

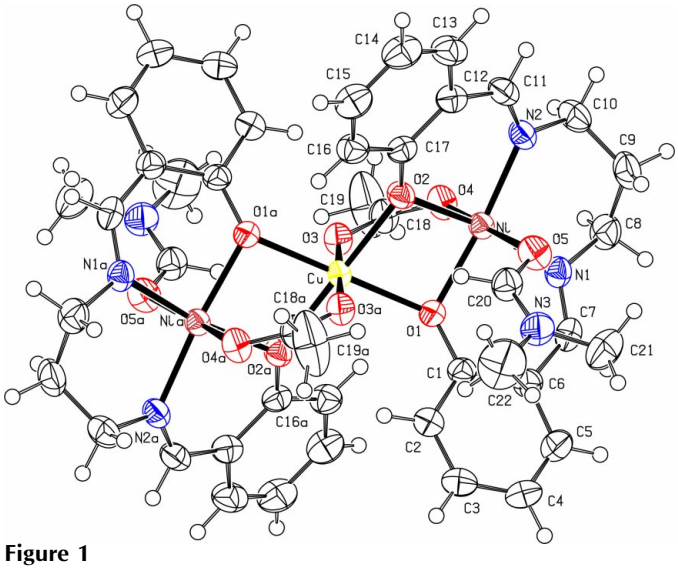


Figure 1
PLATON (Spek, 2000) drawing of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The central metal ions, *i.e.* Cu^{2+} in (I) and Cd^{2+} in (II), have irregular octahedral environments consisting of four bridging O atoms from two $SALPD^{2-}$ ligands [O1, O2, $O1^{i}$, $O2^{i}$; symmetry code: (i) $-x, -y, -z$; Fig. 1] and two O atoms from two bridging acetate groups (O3 and $O3^{i}$). The terminal metal ion, which is Ni^{2+} in both (I) and (II), has a distorted octahedral coordination also, involving two N and two O atoms from a $SALPD^{2-}$ ligand and one O atom each from an acetate and a dimethylformamide group. The Cu—O distances in (I) are all shorter than the corresponding Cd—O distances in (II). The corresponding Ni—O and Ni—N distances have almost the same values in these two complexes. As can be seen from Fig. 2, there is an intramolecular close contact between C20—H20 and O3 [H20···O3 2.54 Å, C20···O3 3.389 (4) Å and C20—H20···O3 148.7°].

Experimental

To a solution of *N*,*N*′-bis(salicylidene)-1,3-propanediamine (2.82 g, 0.1 mol) in hot ethanol (50 ml), 20% ammonia solution (10 ml) was added and the mixture was heated to boiling point. Then a solution of $NiCl_2{\cdot}6H_2O$ (2.38 g, 0.1 mol) 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 dimethylformamide and the temperature of the solution was increased to 383 K. Dropwise addition of $Cu(CH_3COO)_2{\cdot}H_2O$ (0.100 g, 0.5 mmol) dissolved in 20 ml hot ethanol resulted in the formation of crystals of (I) after 24 h.

Crystal data

$[CuNi_2(C_{17}H_{16}N_2O_2)_2(C_2H_3O_2)_2(C_3H_7NO)_2]$
$M_r = 1005.88$
Triclinic, $P\overline{1}$
$a = 9.5802\ (12)$ Å
$b = 10.6920\ (13)$ Å
$c = 12.2069\ (11)$ Å
$\alpha = 112.203\ (3)^\circ$
$\beta = 101.145\ (5)^\circ$
$\gamma = 90.376\ (3)^\circ$
$V = 1131.5\ (2)$ Å^3
$Z = 1$
$D_x = 1.476$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2.5$–25.7°
$\mu = 1.35$ mm^{-1}
$T = 301\ (2)$ K
Parallelepiped, blue green
0.25 × 0.20 × 0.15 mm

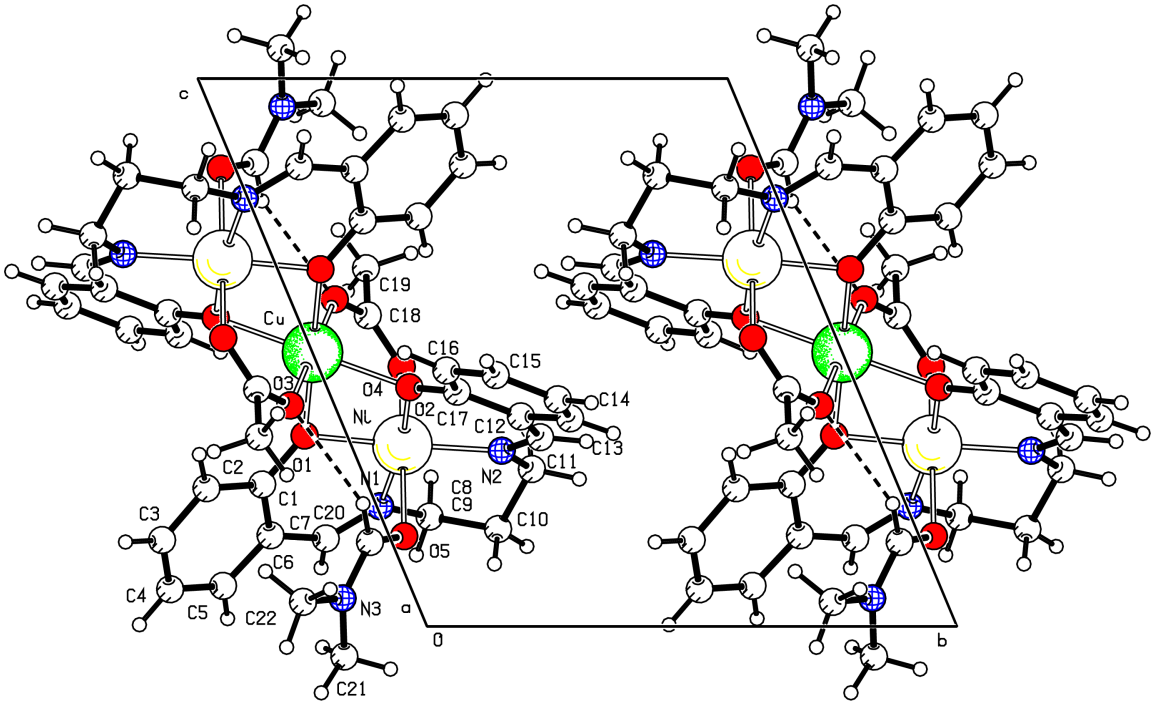


Figure 2
The molecules in the unit cell of (I). The broken lines are the C—H···O close contacts.

Data collection

CAD-4 *EXPRESS* diffractometer	R_{int} = 0.013
$\omega/2\theta$ scans	θ_{max} = 25.7°
Absorption correction: ψ scan (Fair, 1990)	$h = -11 \rightarrow 11$
T_{min} = 0.729, T_{max} = 0.823	$k = 0 \rightarrow 13$
4526 measured reflections	$l = -14 \rightarrow 13$
4282 independent reflections	3 standard reflections
3386 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: 0.1%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2 + 2.0204P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.085	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
S = 0.93	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
4282 reflections	
286 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu—Ni	3.0556 (5)	Ni—N2	2.012 (2)
Cu—O1	2.0705 (19)	Ni—O2	2.0082 (19)
Cu—O2	2.0782 (19)	Ni—O1	2.024 (2)
Cu—O3	2.082 (2)	Ni^{i}—O4	2.039 (2)
Ni—N1	2.030 (2)	Ni—O5	2.186 (2)
O1—Cu—O2	78.89 (8)	O1—Ni—N1	89.38 (9)
O1—Cu—O3	92.92 (8)	O2—Ni—O5	92.73 (9)
O2—Cu—O3	90.94 (8)	N2—Ni—O5	88.15 (9)
O2—Ni—N2	90.85 (9)	O1—Ni—O5	89.36 (8)
O2—Ni—O1	81.65 (8)	N1—Ni—O5	84.29 (9)
N2—Ni—O1	171.98 (9)	Ni—O1—Cu	96.55 (8)
O2—Ni—N1	170.60 (9)	Ni—O2—Cu	96.78 (8)
N2—Ni—N1	97.95 (10)		

Symmetry code: (i) $1 - x, -y, 1 - z$.

H atoms were placed geometrically with respect to their parent atoms and a riding model was used with $U_{eq}(H) = 1.2U_{eq}(C)$.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1993); cell refinement: *SHELXL*97 (Sheldrick, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97; molecular graphics: *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97.

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

References

Atakol, O., Arıcı, C., Tahir, M. N., Kurtaran, R. & Ülkü, D. (1999). *Anal. Sci.* **15**, 933–934.
Enraf–Nonius (1993). *CAD*-4 *EXPRESS*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN*. Enraf–Nonius, Delft,The Netherlands.
Fukuhara, C., Tsuneyoshi, K., Matsumoto, N., Kida, S., Mikuriya, M. & Mori, M. (1990). *J. Chem. Soc. Dalton Trans.* pp. 3473–3479.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (2000). *PLATON*. University of Utrecht, The Netherlands.
Tahir, M. N., Ülkü, D., Atakol, O. & Cakirer, O. (1998). *Acta Cryst.* C**54**, 468–470.
Ülkü, D., Tahir, M. N., Atakol, O. & Nazır, H. (1997). *Acta Cryst.* C**53**, 872–874.