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Structures of (*N,N'*-Trimethylenedisalicylideneaminato)nickel(II) (1) and (*N,N'*-Trimethylenedisalicylideneaminato)copper(II) (2)

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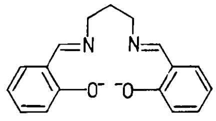
Abstract. (1) [Ni($C_{17}H_{16}N_2O_2$)], $M_r = 338{\cdot}8$, orthorhombic, $Bm2_1b$ (non-standard setting of No. 36 with $b{\rightarrow}c$ and $c{\rightarrow}b$), $a = 25{\cdot}080\,(11)$, $b = 6{\cdot}71\,(1)$, $c = 8{\cdot}82\,(1)$ Å, $U = 1484{\cdot}3$ Å^3, $Z = 4$, $D_m = 1{\cdot}52$, $D_x = 1{\cdot}52$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 13{\cdot}1$ cm^{-1}, $F(000) = 704$, $T = 298$ K, $R = 0{\cdot}055$ for 569 independent reflections. The structure consists of discrete molecules with imposed C_s symmetry. The Ni atom is in a square-planar environment [Ni—N 1·901 (4), Ni—O 1·845 (3) Å]. The molecules pack along y with Ni···Ni distances of 3·366 (1) Å. (2) [Cu($C_{17}H_{16}N_2O_2$)], $M_r = 343{\cdot}7$, orthorhombic, $Pn2_1a$ (non-standard setting of No. 33 with $b{\rightarrow}c$ and $c{\rightarrow}b$), $a = 11{\cdot}925\,(11)$, $b = 6{\cdot}890\,(6)$, $c = 17{\cdot}780\,(8)$ Å, $U = 1460{\cdot}9$ Å^3, $Z = 4$, $D_m = 1{\cdot}56\,(2)$, $D_x = 1{\cdot}56$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 15{\cdot}6$ cm^{-1}, $F(000) = 708$, $T = 298$ K, $R = 0{\cdot}058$ for 830 independent reflections. The structure consists of discrete molecules with bond lengths Cu—N 1·938 (11), 1·958 (12) and Cu—O 1·857 (9), 1·878 (11) Å. There is a distinct tetrahedral distortion to the essentially square-planar coordination sphere, the two Cu,N,O planes intersecting at 21·0 (1)°. The molecules pack along y with Cu···Cu distances of 3·445 (1) Å.

Introduction. There have been many crystal structure determinations of 1:1 complexes between Ni^{II} or Cu^{II} and planar tetradentate ligands with two O and two N donor atoms. The geometry of the immediate coordination sphere is usually planar in the case of Ni^{II}, but for Cu^{II} a tetrahedral distortion is often observed. An additional point of interest is the packing of these essentially planar molecules.

In order to investigate distortions in the geometry and the mode of packing, we have determined the structures of the complexes NiL^1 and CuL^1, where L^1 is *N,N'*-trimethylenedisalicylideneamine.



(L^1)

Experimental. The compounds were prepared following Holm (1960) and crystals of both compounds were obtained from ethanol as solvent. D_m measured by flotation in a carbon tetrachloride/legroin mixture. Needle crystals of approximate size 0·1 × 0·5 × 0·1 mm (1) and 0·1 × 0·5 × 0·1 mm (2) were mounted along **b** and intensities measured on a Stoe STADI-2 diffractometer equipped with a graphite monochromator. Cell dimensions measured from high-angle axial reflections. 918 (h 0–30, k 0–8, l 0–11, $2\theta_{\max} = 50°$) (1) and 1052 (h 0–14, k 0–7, l 0–20, $2\theta_{\max} = 50°$) (2) independent reflections measured by ω scans with a scan speed of 0·0333° s^{-1} and a background count of 20 s. Standard reflections $2k2$ measured for each layer every 20 measurements; no change in intensity observed. Of these intensity data 569 in (1) and 830 in (2) with $I > 2\sigma(I)$ were used in subsequent calculations. Neither absorption nor extinction corrections applied. Positions of the metal atoms in (1) and (2) obtained from the Patterson function and positions of the remaining non-hydrogen atoms obtained from Fourier maps. Atoms were refined anisotropically. H

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atoms fixed in trigonal and tetrahedral positions at 0·95 Å from the C atoms to which they are bonded. In (1) the final $R = 0{\cdot}055$ ($wR = 0{\cdot}060$, $S = 1{\cdot}75$). In view of the closeness of the y coordinates to 0·0 and the large U_{22} terms in comparison to the U_{11} and U_{33} terms, other space groups were considered; in particular the centrosymmetric *Bmmb* (No. 63) with the Ni atom at position 4(*c*). We refined in this space group with all ligand atoms apart from C(11) and C(12) on the mirror plane at $y = 0{\cdot}25$. R converged to 0·062 with 92 parameters (*cf.* 0·055 with 108 above). However, the thermal parameters and dimensions were similar to those obtained in the noncentrosymmetric space group and it seems likely that the true space group is $Bm2_1b$.

In (2) it proved difficult to refine the trimethylene linkage C(21), C(22), C(23) successfully and it seems probable that these atoms are somewhat disordered. In the refinement reported here, the N—C and C—C bond lengths were constrained to expected values. The final $R = 0{\cdot}058$ ($wR = 0{\cdot}067$, $S = 1{\cdot}53$). For both structures, calculations were carried out using the *SHELX*76 system (Sheldrick, 1976) at the University of Manchester Regional Computing Centre. Scattering factors and dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). For both refinements (on F) the weighting scheme was chosen to give similar values of $w\Delta^2$ over ranges of $\sin\theta/\lambda$ and F_o; $w = 1/[\sigma^2(F) + 0{\cdot}002F^2]$ where $\sigma(F)$ was taken from counting statistics. In final cycles of refinement no $\Delta/\sigma > 0{\cdot}1$. Difference Fourier maps showed no significant peaks [in (1) max. 1·32, min. −0·43 e Å^{-3}; in (2) max. 0·47, min. −0·43 e Å^{-3}].

Table 1. *Atomic coordinates* (×10⁴) *for* (1) *with estimated standard deviations in parentheses*

$$\bar{U} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$\bar{U}$(Å^2 × 10^3)
Ni(1)	0	0	−155 (1)	71 (1)
O(1)	465 (1)	97 (17)	−1775 (3)	85 (4)
N(1)	565 (2)	199 (16)	1274 (4)	76 (4)
C(1)	1070 (2)	154 (17)	929 (5)	76 (4)
C(2)	1301 (2)	25 (19)	−538 (5)	66 (4)
C(3)	985 (2)	1 (22)	−1830 (5)	73 (4)
C(10)	1240 (2)	162 (21)	−3247 (6)	104 (5)
C(11)	1786 (2)	−119 (21)	−3360 (7)	101 (5)
C(12)	2103 (2)	−151 (20)	−2066 (8)	94 (5)
C(13)	1862 (2)	−79 (19)	−697 (7)	91 (5)
C(14)	484 (3)	317 (21)	2928 (6)	108 (5)
C(15)	0	650 (18)	3456 (8)	89 (3)

Table 2. *Atomic coordinates* (×10⁴) *for* (2) *with estimated standard deviations in parentheses*

$$\bar{U} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$\bar{U}$(Å^2 × 10^3)
Cu(1)	64 (1)	0	58 (1)	58 (2)
N(1)	−1523 (9)	522 (15)	−62 (5)	43 (11)
O(1)	−4 (7)	152 (50)	1100 (5)	72 (17)
O(2)	1596 (9)	434 (23)	236 (6)	61 (13)
N(2)	284 (11)	−24 (36)	−1033 (7)	76 (17)
C(2)	−2118 (11)	471 (26)	561 (8)	60 (18)
C(3)	−1827 (12)	−358 (22)	1386 (9)	56 (15)
C(4)	−771 (10)	−498 (20)	1547 (8)	47 (13)
C(5)	−2765 (12)	−237 (28)	1898 (9)	70 (15)
C(6)	−2562 (14)	−294 (34)	2644 (10)	76 (24)
C(7)	−1526 (15)	−751 (27)	2874 (10)	96 (20)
C(8)	−573 (17)	−633 (31)	2301 (10)	65 (22)
C(11)	1288 (9)	−362 (20)	−1390 (7)	51 (12)
C(13)	2447 (13)	679 (30)	−280 (9)	94 (19)
C(12)	2301 (15)	−44 (38)	−1017 (9)	97 (22)
C(14)	3448 (12)	894 (27)	45 (8)	66 (14)
C(15)	4410 (17)	1152 (30)	−449 (9)	77 (22)
C(16)	4318 (13)	535 (28)	−1170 (11)	76 (21)
C(17)	3229 (15)	−107 (29)	−1521 (10)	127 (23)
C(21)	−2257 (17)	887 (26)	−686 (9)	68 (21)
C(22)	−1685 (24)	812 (66)	−1320 (15)	150 (54)
C(23)	−622 (26)	−332 (34)	−1536 (16)	129 (38)

Table 3. *Interatomic distances* (Å) *and angles* (°) *for* (1)

Ni coordination sphere

Ni(1)—O(1)	1·845 (3)	Ni(1)—N(1)	1·901 (4)
O(1)—Ni(1)—N(1)	92·31 (16)	O(1)—Ni(1)—O(1^i)	78·31 (13)
O(1)—Ni(1)—N(1^i)	169·01 (29)	N(1)—Ni(1)—N(1^i)	96·39 (18)

Remaining dimensions

O(1)—C(3)	1·309 (5)	C(3)—C(10)	1·408 (6)
N(1)—C(1)	1·303 (6)	C(10)—C(11)	1·384 (8)
N(1)—C(14)	1·475 (6)	C(11)—C(12)	1·391 (9)
C(1)—C(2)	1·420 (6)	C(12)—C(13)	1·351 (9)
C(2)—C(3)	1·388 (6)	C(14^i)—C(15)	1·309 (3)
C(2)—C(13)	1·415 (7)		
Ni(1)—O(1)—C(3)	131·1 (3)	O(1)—C(3)—C(10)	118·8 (4)
Ni(1)—N(1)—C(1)	124·6 (3)	C(2)—C(3)—C(10)	118·0 (4)
Ni(1)—N(1)—C(14)	123·8 (4)	C(3)—C(10)—C(11)	120·1 (5)
C(1)—N(1)—C(14)	111·5 (4)	C(10)—C(11)—C(12)	120·5 (5)
N(1)—C(1)—C(2)	127·6 (4)	C(11)—C(12)—C(13)	118·5 (5)
C(1)—C(2)—C(3)	121·1 (4)	C(2)—C(13)—C(12)	122·3 (5)
C(1)—C(2)—C(13)	119·9 (4)	N(1)—C(14)—C(15)	119·0 (6)
C(3)—C(2)—C(13)	119·0 (5)	C(14^i)—C(15)—C(14)	133·9 (8)
O(1)—C(3)—C(2)	122·6 (4)		

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

Table 4. *Interatomic distances* (Å) *and angles* (°) *for* (2)

Cu coordination sphere

Cu(1)—N(1)	1·938 (11)	Cu(1)—O(2)	1·878 (11)
Cu(1)—O(1)	1·857 (9)	Cu(1)—N(2)	1·958 (12)
N(1)—Cu(1)—O(1)	93·26 (46)	N(1)—Cu(1)—N(2)	91·31 (46)
N(1)—Cu(1)—O(2)	159·9 (5)	O(1)—Cu(1)—N(2)	174·1 (8)
O(1)—Cu(1)—O(2)	82·25 (41)	O(2)—Cu(1)—N(2)	92·2 (5)

Remaining dimensions

N(1)—C(2)	1·317 (17)	C(6)—C(7)	1·340 (22)
N(1)—C(21)	1·434 (18)	C(7)—C(8)	1·529 (23)
O(1)—C(4)	1·293 (17)	C(11)—C(12)	1·395 (21)
O(2)—C(13)	1·379 (16)	C(13)—C(12)	1·414 (26)
N(2)—C(11)	1·375 (16)	C(13)—C(14)	1·334 (22)
N(2)—C(23)	1·418 (30)	C(12)—C(17)	1·424 (21)
C(2)—C(3)	1·611 (20)	C(14)—C(15)	1·456 (22)
C(3)—C(4)	1·295 (18)	C(15)—C(16)	1·356 (25)
C(3)—C(5)	1·445 (19)	C(16)—C(17)	1·507 (25)
C(4)—C(8)	1·364 (21)	C(21)—C(22)	1·319 (25)
C(5)—C(6)	1·349 (23)	C(22)—C(23)	1·541 (29)
Cu(1)—N(1)—C(2)	115·4 (8)	C(6)—C(7)—C(8)	118·0 (18)
Cu(1)—N(1)—C(21)	135·5 (10)	C(4)—C(8)—C(7)	122·0 (16)
C(2)—N(1)—C(21)	109·1 (12)	N(2)—C(11)—C(12)	120·6 (13)
Cu(1)—O(1)—C(4)	128·7 (11)	O(2)—C(13)—C(12)	118·9 (16)
Cu(1)—O(2)—C(13)	128·5 (10)	O(2)—C(13)—C(14)	112·6 (14)
Cu(1)—N(2)—C(11)	125·1 (9)	C(12)—C(13)—C(14)	123·4 (13)
Cu(1)—N(2)—C(23)	121·6 (15)	C(11)—C(12)—C(13)	127·1 (14)
C(11)—N(2)—C(23)	110·3 (16)	C(11)—C(12)—C(17)	111·7 (16)
N(1)—C(2)—C(3)	131·2 (11)	C(13)—C(12)—C(17)	119·9 (18)
C(2)—C(3)—C(4)	116·0 (12)	C(13)—C(14)—C(15)	117·2 (13)
C(2)—C(3)—C(5)	112·7 (12)	C(14)—C(15)—C(16)	117·9 (16)
C(4)—C(3)—C(5)	128·1 (15)	C(15)—C(16)—C(17)	123·6 (16)
O(1)—C(4)—C(3)	121·7 (14)	C(12)—C(17)—C(16)	113·6 (18)
O(1)—C(4)—C(8)	120·4 (13)	N(1)—C(21)—C(22)	109·8 (19)
C(3)—C(4)—C(8)	113·0 (14)	C(21)—C(22)—C(23)	131·2 (28)
C(3)—C(5)—C(6)	118·7 (14)	N(2)—C(23)—C(22)	113·1 (23)
C(5)—C(6)—C(7)	118·2 (17)		

Discussion. Atomic coordinates for (1) and (2) are given in Tables 1 and 2, molecular dimensions in Tables 3 and 4.*

(1) and (2) form part of a series of four-coordinate structures with L^1 and the similar ligands *N,N'*-ethylenedisalicylideneamine (L^2), 2,2'-hydroxy-*N,N'*-trimethylenedinaphthalene-1-methanimine (L^3) and 2,2'-hydroxy-*N,N'*-ethylenedinaphthalene-1-methanimine (L^4). Details of four-coordinate structures of this type were abstracted from the Cambridge Structural Database (1984) and are listed in Table 5. The geometry of the coordination spheres is similar for the square-planar Ni structures. The Cu structures differ in the amount of tetrahedral distortion. There is also considerable variation in the mode of packing for these planar molecules.

The structure of (1) is shown in Fig. 1 together with the atomic numbering scheme. The molecule has crystallographically imposed C_s symmetry with the Ni atom on a mirror plane which is perpendicular to the general plane of the molecule. The molecules have the *cis* form as imposed by the geometry of the tetradentate ligand; the coordination of the metal atom is square planar, the angle between the two Ni,N,O planes being 8·9 (1)°. However, this distortion is a fold rather than a tetrahedral twist as the four donor atoms form a plane within experimental error. The Ni atom is 0·12 (1) Å from this plane. The two independent bond lengths are Ni—O 1·845 (3) and Ni—N 1·901 (4) Å.

The structure of (2) is shown in Fig. 2 together with the atomic numbering scheme. There is a considerable tetrahedral distortion of 21·0 (1)° between the two Cu,N,O planes. Such distortions are often found in Cu^{II} structures and have been variously ascribed to steric and/or electronic effects (Wei, 1972). This angle between planes can be compared to 1·7° in CuL^2 (Akhtar & Drew, 1982) and 43·8° in (α,α'-dimethyl-*N,N'*-trimethylenedisalicylideneaminato)copper(II) (Iida, Oonishi, Nakahara & Komiyama, 1970). The Cu—N distances [1·938 (11), 1·958 (12) Å] are longer than the Cu—O [1·857 (9), 1·878 (11) Å], this difference being similar to those observed in (1), CuL^2 and a number of other molecules listed in Table 5. In most Cu^{II} structures tetrahedral distortion has been ascribed to intramolecular distortion (Wei, 1972) but for these

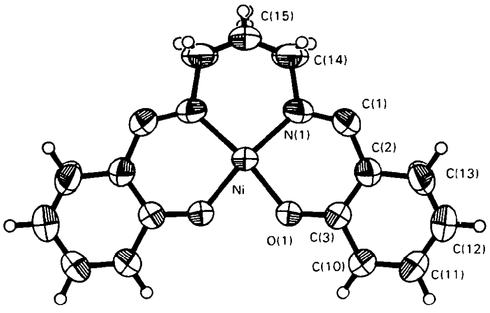

Fig. 1. The structure of (1) and atom numbering.

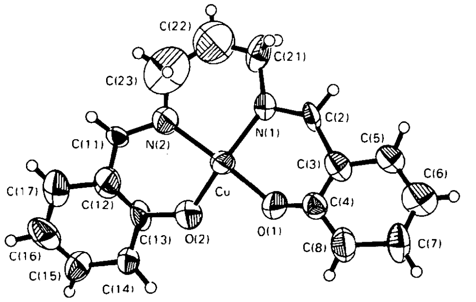

Fig. 2. The structure of (2) and atom numbering.

Table 5. *Four-coordinate structures containing L^1 and similar ligands*

Compound	Reference	Mean values M—N (Å)	M—O (Å)	$M\cdots M$ (Å)	Compound type
NiL^1	This work	1·901 (4)	1·845 (3)	3·366 (1)	Chain
CuL^1	This work	1·948 (15)	1·867 (15)	3·445 (1)	Chain
NiL^2	(1)	1·86	1·84	3·21	Dimer
CuL^2	(2)	2·01	1·97	3·25	Dimer (Cu···O 2·41 Å)
CuL^2.nitrophenol	(3)	1·94	1·90	3·52	Dimer
$CuL^2.CHCl_3$	(4)	1·905	1·940	3·35	Dimer (Cu···O 2·79 Å)
CuL^2.thiourea	(6)	1·931	1·890	4·15	Dimer (Cu···N 3·52 Å)
$NiL^2.NH_4BPh_4$	(7)	1·92	1·87	3·50	Dimer
$NiL^2.NaBPh_4$	(7)	1·848	1·860	3·38	Dimer
NiL^3	(8)	1·871	1·866	4·07	Chain
CuL^3	(8)	1·980	1·911	3·61	Dimer
NiL^4	(9) (5)	1·840	1·849	3·32	Dimer
CuL^4	(5)	1·925	1·951	3·30	Dimer

References: (1) Shkol'nikova, Yumal, Shugam & Voblikova (1970). (2) Hall & Waters (1960). (3) Baker, Hall & Waters (1970*a*). (4) Baker, Hall & Waters (1970*b*). (5) Frieburg, Reichert, Mekhers & Engelen (1980). (6) Ferrari, Fava & Pelizzi (1976). (7) Pahor *et al.* (1976). (8) Akhtar & Drew (1982). (9) Akhtar (1981).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42468 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

planar molecules we suspect that intramolecular rather than intermolecular steric forces may be responsible. After all a most striking distortion was noted in NiL^3 (Akhtar & Drew, 1982); here, although the metal coordination sphere was square planar, the structure was bowed with the two chelate Ni,O,C,C,C,N rings intersecting at an angle of 37·0 (1)° (*cf.* 6·1° in CuL^2). This distortion can only be due to intermolecular forces. Steric forces have more effect on Cu^{II} than on Ni^{II} coordination spheres and therefore the variation observed in (2) is presumably due to packing forces.

It remains to consider the packing of these types of planar molecules. Several types of bonding can be differentiated from Table 5. In several examples (those where an additional bond length is given in the table) two molecules pack together such that two weak axial $M\cdots O$ interactions are formed. With solvent molecules or ions in the unit cell, these can then interact with the metal or the donor N and O atoms and the packing becomes more complicated. However, in some of these, there are still some close $M\cdots M$ contacts. The more usual type of packing is when the molecules line up either in pairs or in a chain so that the $M\cdots M$ vector forms the closest contact and the vector is approximately perpendicular to the molecules. This arrangement is found in CuL^3, NiL^4, NiL^3 and also (1) and (2). However, in the former two, there are just two molecules packing in this way. In NiL^3 chains are formed with 4·07 Å between metal atoms. (1) and (2) are similar to each other but exceptional in that the $\cdots M\cdots M\cdots M\cdots$ distances are short at 3·366 (1) Å in (1) and 3·445 (1) Å in (2). This does not in our view represent a metal$\cdots$metal bond or even an interaction but rather an efficient way of packing the relatively planar molecules. However, against this it could be argued that the uniqueness of the packing may be indicative of such an interaction.

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Structure du Bis(benzènetétracarboxylate-1,2,4,5) de Pentaneptunyle(V) et de Triammonium à Sept Molécules d'Eau

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Abstract. $[NH_4]_3[NpO_2]_5[C_{10}H_2O_8]_2.7H_2O$, $M_r = 2025{\cdot}448$, monoclinic, $P2_1/c$, $a = 19{\cdot}315\ (4)$, $b = 19{\cdot}640\ (5)$, $c = 10{\cdot}295\ (3)$ Å, $\beta = 95{\cdot}27°$, $V = 3888{\cdot}86$ Å^3, $Z = 4$, $D_x = 3{\cdot}46$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 10·3 mm^{-1}, $F(000) = 3420{\cdot}84$, room temperature, $R(F) = 0{\cdot}039$, $wR = 0{\cdot}057$ for 9458

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