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Structures of the Ni^{II} Complexes of the Imine Ligands derived from 2,2-Diethoxyethylamine with Salicylaldehyde and 1,2- and 2,3-Hydroxynaphthaldehydes

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Abstract. (I): Bis[*N*-(2,2-diethoxyethyl)salicylideneaminato]nickel(II), [Ni(C₁₃H₁₈NO₃)₂], *M_r* = 531.3, monoclinic, *C*2/*c*, *a* = 32.244 (7), *b* = 5.520 (1), *c* = 26.742 (5) Å, β = 145.798 (11)°, *V* = 2675.50 Å³, *Z* = 4, *D_x* = 1.319 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 7.66 cm⁻¹, *F*(000) = 1128, *T* = 293 K, *R* = 0.069 for 1504 independent reflections. (II): Bis[*N*-(2,2-diethoxyethyl)-2-oxo-1-naphthylmethylideneaminato]nickel(II), [Ni(C₁₇H₂₀NO₃)₂], *M_r* = 631.4, monoclinic, *P*2₁/*c*, *a* = 13.493 (5), *b* = 6.014 (2), *c* = 19.783 (5) Å, β = 97.00 (2)°, *V* = 1593.36 Å³, *Z* = 2, *D_x* = 1.316 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 6.54 cm⁻¹, *F*(000) = 668, *T* = 293 K, *R* = 0.053 for 1674 independent reflections. (III): Bis[*N*-(2,2-diethoxyethyl)-3-oxo-2-naphthylmethylideneaminato]nickel(II), [Ni(C₁₇H₂₀NO₃)₂], *M_r* = 631.4, monoclinic, *C*2/*c*, *a* = 30.921 (2), *b* = 5.658 (4), *c* = 26.018 (2) Å, β = 134.87 (4)°, *V* = 3225.96 Å³, *Z* = 4, *D_x* = 1.300 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 6.46 cm⁻¹, *F*(000) = 1336, *T* = 293 K, *R* = 0.068 for 1648 independent reflections. The three compounds exhibit square-planar coordination around the metal atom. The chelate and aromatic ring(s) in each ligand are nearly planar in all three complexes with the *N*-diethoxyethyl groups at angles of 109–113° to the ring system. In compounds (I) to (III) there is a significant step at Ni between the two ligand planes. There are some small but significant differences between the Ni–N bond lengths in the two naphthaldehyde-derived complexes.

Introduction. The existence of electronic delocalization in metal complexes containing ligands from salicylaldehyde and naphthaldehyde was first proposed by Calvin & Wilson (1945) as an explanation for the stability order observed for a series of copper complexes. The stability order observed for the ligands acac (acetylacetonate), sal (salicylaldehyde) and 1,2- and 2,3-naphtha (naphtholate ion of hydroxynaphthaldehyde) was acac > 1,2-naphtha > sal > 2,3-naphtha and was related to the relative ease with which the different ligands allowed electronic delocalization to produce resonance structures like those shown in Fig. 1.

Resonance was later extensively used in enolate systems (Mehrotra, Bohra & Gaur, 1978) and electronic delocalization has also been suggested for the chelate ring in some imines derived from acetylacetonate (Hatfield & Bunger, 1966).

Metal imine complexes with ligands derived from salicylaldehyde, 2-hydroxy-1-naphthaldehyde and 3-hydroxy-2-naphthaldehyde could also be expected to show delocalization of electron density and the extent of this process might be reflected in some structural features. However, to our knowledge, there have been no reports on structural comparison between compounds of this type.

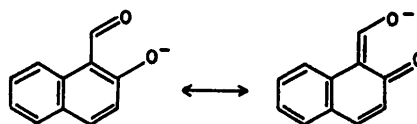


Fig. 1. Resonance structures of the 1,2-naphtha ligand.

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In this work we describe the structures of Ni^{II} complexes of the Schiff bases derived from salicylaldehyde, 2-hydroxy-1-naphthaldehyde and 3-hydroxy-2-naphthaldehyde with 2,2-diethoxyethylamine.

Experimental. Crystals by slow evaporation of THF–MeOH–CH₂Cl₂ solutions. Crystal dimensions (mm): 0.26 × 0.4 × 0.6, 0.12 × 0.42 × 0.60, 0.12 × 0.30 × 0.42. Cell dimensions from angular measurements of 25, 19 and 21 strong reflections in the range 3 < 2θ < 20°. Nicolet R3m four-circle diffractometer, graphite-monochromated Mo Kα radiation; ω scan with variable scan rate from 4 to 30° min⁻¹ and a scan width of 1.0°; 3 < 2θ < 45°; h 0→35, 15, 34; k 0→6, 7, 7; l -29, -22, -29→29, 22, 29. Two monitor reflections with constant intensity in each case. 1744, 2094 and 2091 measured reflections with 1504, 1674 and 1648 unique [I > 2.5σ(I)], R_{int} = 0.023, 0.015 and 0.023.

Lorentz and polarization corrections. Empirical absorption corrections based on ψ scans. Structure of (I) by Patterson heavy-atom method. Structure of (II) and (III) by direct methods and Fourier difference maps. In all three cases, metal atoms at centers of symmetry. Refinement by full-matrix least squares, anisotropic thermal parameters for all non-H atoms. H atoms in idealized positions, fixed U = 0.06 Å²; weighting scheme [σ²|F_o| + G|F_o|²]⁻¹, where σ is the estimated deviation based on counting statistics and G an adjustable variable; final G = 0.005, 0.002 and 0.004 respectively; maximum Δρ in final difference maps < 1.4 (peaks not close to any atom and not investigated further), 0.5 and 0.8 e Å⁻³; R = 0.069, 0.053 and 0.068; wR = 0.084, 0.056 and 0.076; (Δ/σ)_{max} = 0.062, 0.044 and 0.029; S = 1.363, 1.281 and 1.217. Solution, refinement and plots using SHELXTL (Sheldrick, 1981) on a Nova computer. Anomalous-scattering-factor values from *International Tables for X-ray Crystallography* (1974).

Discussion. Final positional parameters are shown in Table 1.* The molecular structures of complexes (I), (II) and (III), including atom-numbering schemes, are illustrated in Figs. 2, 3 and 4, respectively. Associated bond parameters for the three structures are presented in Table 2. The metal atoms display a square-planar coordination geometry in all three complexes. The chelate and aromatic rings are nearly planar with the N-diethoxyethyl groups almost perpendicular to the plane. The angle between the chelate and aromatic ring planes in (I) is 4.0°, while the angles between the

chelate ring and the aromatic skeleton are 8.5° in (II) and 4.5° in (III). A tilt is observed in all three compounds between the coordination plane and the two

Table 1. Atom coordinates (× 10⁴) and thermal parameters (Å² × 10³) of compounds (I), (II) and (III)

	$U_{eq} = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ tensor}).$			
	x	y	z	U_{eq}
(I)				
Ni	0	5000	0	37 (1)
N(1)	458 (2)	2247 (7)	142 (3)	37 (6)
O(1)	799 (2)	5606 (8)	1202 (3)	50 (5)
O(2)	910 (2)	2347 (8)	-475 (3)	55 (5)
O(3)	-378 (4)	1788 (15)	-1951 (5)	130 (12)
C(1)	1513 (3)	2069 (9)	1749 (3)	39 (6)
C(2)	1373 (3)	4220 (10)	1870 (4)	41 (7)
C(3)	1864 (4)	4929 (10)	2739 (4)	51 (9)
C(4)	2473 (3)	3541 (11)	3462 (4)	52 (7)
C(5)	2615 (3)	1393 (11)	3350 (4)	54 (8)
C(6)	2153 (3)	664 (11)	2527 (4)	48 (8)
C(7)	1068 (3)	1272 (10)	907 (3)	38 (7)
C(8)	116 (3)	992 (11)	-629 (4)	42 (7)
C(9)	170 (3)	2385 (13)	-1056 (4)	54 (7)
C(10)	1053 (4)	4000 (17)	-737 (5)	77 (11)
C(11)	1822 (4)	3507 (18)	-149 (5)	91 (12)
C(12)	-295 (7)	-312 (21)	-1958 (8)	156 (33)
C(13)	-985 (5)	-1222 (23)	-3049 (5)	118 (14)
(II)				
Ni	5000	10000	0	41 (1)
N(1)	4022 (3)	8582 (6)	468 (2)	44 (1)
O(1)	5059 (2)	12379 (5)	579 (2)	54 (1)
O(2)	1917 (2)	8897 (6)	-168 (2)	59 (1)
O(3)	2322 (3)	6045 (6)	-870 (2)	67 (1)
C(1)	4231 (3)	10938 (8)	1484 (2)	42 (1)
C(2)	4810 (3)	12520 (7)	1192 (2)	45 (2)
C(3)	5140 (4)	14449 (8)	1574 (3)	54 (2)
C(4)	4921 (4)	14722 (8)	2216 (3)	58 (2)
C(5)	4204 (4)	13419 (10)	3225 (2)	61 (2)
C(6)	3702 (4)	11877 (10)	3551 (3)	63 (2)
C(7)	3342 (4)	9981 (9)	3193 (2)	62 (2)
C(8)	3483 (4)	9656 (9)	2525 (2)	55 (2)
C(9)	4022 (8)	11219 (8)	2179 (2)	45 (2)
C(10)	4374 (3)	13135 (8)	2547 (2)	49 (2)
C(11)	3811 (3)	9146 (8)	1073 (2)	43 (1)
C(12)	3404 (3)	6802 (7)	121 (2)	45 (2)
C(13)	2686 (3)	7806 (8)	-447 (2)	49 (2)
C(14)	1754 (6)	11089 (12)	-343 (4)	108 (3)
C(15)	1079 (6)	12272 (11)	29 (3)	98 (3)
C(16)	1802 (6)	6696 (12)	-1499 (3)	100 (3)
C(17)	1743 (6)	4990 (11)	-1997 (3)	100 (3)
(III)				
Ni	0	10000	0	49 (1)
N(1)	424 (2)	7306 (7)	67 (2)	46 (3)
O(1)	649 (2)	10849 (7)	949 (2)	63 (3)
O(2)	840 (2)	7282 (7)	-686 (2)	65 (3)
O(3)	-203 (3)	6671 (15)	-1760 (3)	129 (7)
C(1)	1746 (2)	6010 (10)	1987 (3)	47 (4)
C(2)	1252 (2)	7310 (9)	1390 (3)	47 (4)
C(3)	1104 (3)	9540 (9)	1499 (3)	53 (5)
C(4)	1458 (3)	10319 (10)	2200 (3)	54 (5)
C(5)	2341 (3)	9820 (11)	3535 (3)	62 (5)
C(6)	2842 (3)	8558 (13)	4115 (4)	71 (6)
C(7)	2986 (3)	6394 (12)	3994 (3)	68 (5)
C(8)	2627 (3)	5552 (11)	3306 (3)	61 (5)
C(9)	2113 (2)	6818 (10)	2700 (3)	49 (5)
C(10)	1963 (2)	9025 (11)	2809 (3)	52 (5)
C(11)	917 (2)	6405 (10)	678 (3)	48 (4)
C(12)	174 (3)	5967 (11)	-581 (3)	53 (4)
C(13)	231 (3)	7320 (12)	-1036 (3)	60 (5)
C(14)	974 (3)	8834 (16)	-986 (4)	87 (7)
C(15)	1609 (3)	8466 (19)	-629 (4)	109 (8)
C(16)	-278 (7)	4511 (15)	-1922 (6)	174 (24)
C(17)	-755 (4)	3681 (22)	-2694 (5)	134 (10)

* Lists of structure factors, least-squares planes, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43004 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

parallel salen or naphthen planes, 6.8° in (I), 12.9° in (II) and 10.1° in (III). This conformation has been described as a 'stepped' conformation (Maslen & Waters, 1975) and has been observed for other nickel complexes (Stewart & Lingafelter, 1959; Bhatia, Bindlish, Saini & Jain, 1981). The two parallel salen or naphthen planes are stepped by *ca* 0.5 Å in (I), 0.94 Å in (II) and 0.68 Å in (III).

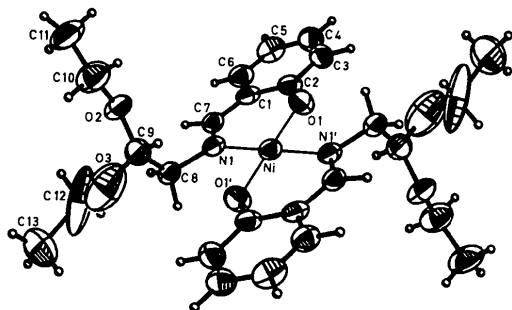


Fig. 2. The molecular structure of (I) showing the atom-numbering scheme adopted. The thermal ellipsoids show 50% probability.

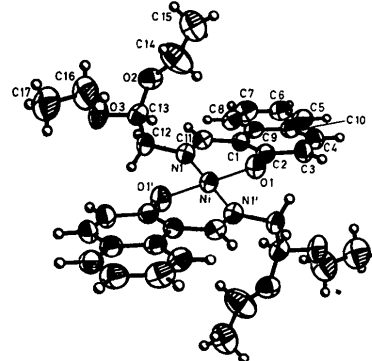


Fig. 3. The molecular structure of (II) showing the atom-numbering scheme adopted. The thermal ellipsoids show 50% probability.

Table 2. Bond lengths (Å) and angles (°) of compounds (I), (II) and (III)

	(I)	(II)	(III)		(I)	(II)	(III)
Ni—N(1)	1.926 (6)	1.904 (4)	1.937 (5)	Ni—O(1)	1.840 (4)	1.827 (3)	1.834 (3)
N(1)—C(7)	1.300 (5)	—	—	N(1)—C(8)	1.478 (11)	—	—
N(1)—C(11)	—	1.310 (6)	1.300 (5)	N(1)—C(12)	—	1.474 (6)	1.476 (9)
O(1)—C(2)	1.317 (6)	1.301 (5)	—	O(1)—C(3)	—	—	1.318 (6)
O(2)—C(9)	1.404 (10)	—	—	O(2)—C(10)	1.440 (17)	—	—
O(2)—C(13)	—	1.398 (6)	1.396 (9)	O(2)—C(14)	—	1.373 (8)	1.418 (14)
O(3)—C(9)	1.400 (11)	—	—	O(3)—C(12)	1.193 (16)	—	—
O(3)—C(13)	—	1.401 (6)	1.384 (8)	O(3)—C(16)	—	1.408 (7)	1.260 (12)
C(1)—C(2)	1.400 (11)	1.400 (6)	1.393 (6)	C(1)—C(6)	1.444 (6)	—	—
C(1)—C(7)	1.407 (11)	—	—	C(1)—C(9)	—	1.449 (6)	1.403 (9)
C(1)—C(11)	—	1.424 (6)	—	C(2)—C(3)	1.404 (11)	1.425 (6)	1.437 (9)
C(2)—C(11)	—	—	1.434 (9)	C(3)—C(4)	1.379 (7)	1.349 (7)	1.379 (9)
C(4)—C(5)	1.391 (12)	—	—	C(4)—C(10)	—	1.416 (7)	1.412 (7)
C(5)—C(6)	1.342 (11)	1.357 (8)	1.374 (8)	C(5)—C(10)	—	1.398 (7)	1.422 (9)
C(6)—C(7)	—	1.397 (8)	1.412 (12)	C(7)—C(8)	—	1.372 (7)	1.364 (10)
C(8)—C(9)	1.502 (16)	1.416 (7)	1.410 (7)	C(9)—C(10)	—	1.414 (7)	1.427 (10)
C(10)—C(11)	1.499 (16)	—	—	C(12)—C(13)	1.726 (17)	1.516 (6)	1.521 (13)
C(14)—C(15)	—	1.430 (11)	1.480 (14)	C(16)—C(17)	—	1.417 (9)	1.501 (14)
N(1)—Ni—O(1)	93.6 (2)	88.6 (1)	93.3 (2)	N(1)—Ni—N(1a)	180.0	180.0	180.0
O(1)—Ni—N(1a)	86.4 (2)	88.6 (1)	86.7 (2)	Ni—N(1)—C(7)	123.5 (6)	—	—
Ni—N(1)—C(8)	122.0 (3)	—	—	C(7)—N(1)—C(8)	114.4 (6)	—	—
Ni—N(1)—C(11)	—	124.8 (3)	123.6 (5)	Ni—N(1)—C(12)	—	119.0 (3)	121.7 (3)
C(11)—N(1)—C(12)	—	116.1 (4)	114.4 (5)	Ni—O(1)—C(2)	129.1 (5)	129.7 (3)	—
Ni—O(1)—C(3)	—	—	129.1 (4)	C(9)—O(2)—C(10)	112.7 (6)	—	—
C(9)—O(3)—C(12)	105.8 (9)	—	—	C(13)—O(2)—C(14)	—	117.0 (5)	114.4 (5)
C(2)—C(1)—C(6)	118.2 (7)	—	—	C(2)—C(1)—C(7)	122.6 (4)	—	—
C(13)—O(3)—C(16)	—	114.7 (4)	119.1 (8)	C(2)—C(1)—C(11)	—	118.8 (4)	—
C(2)—C(1)—C(9)	—	119.8 (4)	122.1 (6)	O(1)—C(2)—C(1)	122.7 (6)	124.2 (4)	—
C(6)—C(1)—C(7)	119.2 (7)	—	—	O(1)—C(3)—C(2)	—	—	121.6 (6)
O(1)—C(2)—C(3)	118.6 (7)	116.6 (4)	—	C(1)—C(2)—C(11)	—	—	118.5 (6)
O(1)—C(3)—C(4)	—	—	119.9 (6)	C(9)—C(1)—C(11)	—	121.3 (4)	—
C(3)—C(2)—C(11)	—	—	121.9 (4)	C(2)—C(3)—C(4)	121.0 (8)	120.5 (4)	118.4 (5)
C(1)—C(2)—C(3)	118.7 (5)	119.2 (4)	119.1 (8)	C(3)—C(4)—C(10)	—	122.8 (4)	122.4 (6)
C(3)—C(4)—C(5)	120.7 (7)	—	—	C(5)—C(6)—C(7)	—	118.7 (5)	119.7 (7)
C(4)—C(5)—C(6)	119.6 (5)	—	—	C(6)—C(7)—C(8)	—	121.5 (5)	120.2 (5)
C(6)—C(5)—C(10)	—	121.5 (5)	121.8 (7)	C(1)—C(6)—C(5)	121.7 (8)	—	—
C(7)—C(8)—C(9)	—	120.8 (5)	121.4 (7)	C(1)—C(9)—C(10)	—	119.5 (4)	118.3 (4)
C(1)—C(9)—C(8)	—	123.6 (4)	122.3 (6)	C(4)—C(10)—C(5)	—	121.2 (5)	123.3 (6)
C(8)—C(9)—C(10)	—	116.9 (4)	119.4 (6)	C(5)—C(10)—C(9)	—	120.6 (5)	117.5 (5)
C(4)—C(10)—C(9)	—	118.1 (4)	119.2 (6)	N(1)—C(8)—C(9)	113.5 (6)	—	—
N(1)—C(7)—C(1)	127.1 (7)	—	—	N(1)—C(12)—C(13)	—	109.3 (4)	112.1 (5)
N(1)—C(11)—C(10)	—	126.9 (4)	126.5 (6)	O(2)—C(9)—C(8)	108.0 (5)	—	—
O(2)—C(9)—O(3)	118.4 (11)	—	—	O(2)—C(13)—C(12)	—	109.4 (4)	109.1 (5)
O(2)—C(13)—O(3)	—	111.6 (4)	116.8 (9)	O(3)—C(13)—C(12)	—	106.7 (4)	113.0 (7)
O(3)—C(9)—C(8)	113.7 (8)	—	—	O(2)—C(14)—C(15)	—	116.1 (7)	110.0 (7)
O(2)—C(10)—C(11)	107.1 (7)	—	—	O(3)—C(16)—C(17)	—	113.1 (6)	121.8 (9)
O(3)—C(12)—C(13)	108.5 (9)	—	—				

especially between the 1,2- and 2,3-naphthaldehyde derivatives [1.904 (4) and 1.937 (5) Å, respectively]. Although similar differences have been described for Cu–N bond lengths in several copper–salicylaldimine complexes (Jain & Syal, 1973), those may be partly caused by the variation in the group bonded to the N atoms, while in (II) and (III), the same N-substituent is used.

The observed variation could then be ascribed in this case to the different positions of the coordinating groups within the naphthalene rings (1,2- or 2,3-). The 1,2-bond in naphthalene has been ascribed (Badger, 1969) a larger double-bond character than the 2,3-bond and this might favour a larger delocalization of the electron density in the 1,2-derivative which would then make compound (II) more stable than compound (III).

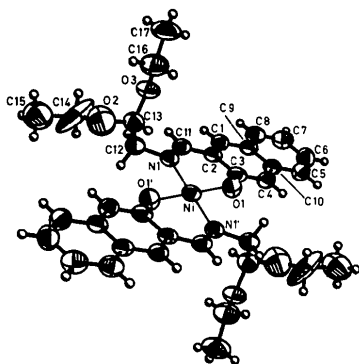


Fig. 4. The molecular structure of (III) showing the atom-numbering scheme adopted. The thermal ellipsoids show 50% probability.

The large vibrational amplitudes of the ethoxy groups in all of the compounds limited the precision of the structure determinations. In order to achieve a better understanding of the system, further studies on related compounds were undertaken and the results will be published elsewhere (Fernández, Rosales, Rubio, Salcedo, Toscano & Vela, in preparation).

We thank Mr Abelardo Cuellar for technical assistance.

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Structure of [*N,N'*-Bis(3-aminopropyl)-1,2-ethanediamine]perchloratocopper(II) Perchlorate Hemihydrate

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Abstract. [Cu(ClO₄)(C₈H₂₂N₄)]ClO₄·½H₂O, *M_r* = 445.74, orthorhombic, *Pbca*, *a* = 13.939 (3), *b* = 16.169 (6), *c* = 15.325 (5) Å, *V* = 3454 (6) Å³, *Z* = 8, *D_m* = 1.68 (8) (by flotation in CCl₄/hexane mixture), *D_x* = 1.714 Mg m⁻³, Mo *Kα*, λ = 0.71069 Å, μ =

1.669 mm⁻¹, *F*(000) = 1840, *T* = 296 (4) K, *R*(*F*) = 0.069 for 2105 observed reflections. The coordination about Cu^{II} is square pyramidal, with the tetraamine ligand equatorial and perchlorate ion axial. The two asymmetric nitrogens are of the same *R* or *S* configuration. The six-membered chelate rings exhibit a chair form and the five-membered ring takes a *gauche* form.

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