within 0·04 Å]. The dihedral angle between the equatorial CuO_4 plane and the chelate plane is 158·3°. Bond lengths and bond angles in the ligand adopt values typical for acetylacetonates [C—C(C)=O fragment] (Lingafelter & Braun, 1966) and phosphonic acid derivatives $[O=P(O)_2-C$ fragment] (Naumov & Vilkov, 1986). The angles in the $P(C)O_3$ tetrahedron vary from 98·0 (6) [O(3a)—P—O(3b)] to 116·2 (5)° [O(2)—P—O(3a)]. The conformation of both propoxy groups is different and follows the requirements of molecular packing. Torsion angles P—O—C—C [O—C—C—C] are 158 (2), −172 (1) [−20 (3), −180 (2)°] for branches a and b respectively.

The cyano group with a C(4)—N bond length of 1·15 (1) Å has triple-bond character. Consideration of the relatively short Cu···N distance, and the geometry of the cyano groups within the copper coordination sphere [angle Cu···N≡C $(-x, \frac{1}{2}+y, \frac{1}{2}-z) =$ 149·8 (9)°], indicates that π-electron delocalization of C≡N is effectively compensated by σ-electron transfer

from Cu to N. In this structure the ligand is tridentate and performs a chelate-bridging role (Shkolnikova & Poray-Koshits, 1982).

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Structures of Chloro(glycinato)(1,10-phenanthroline)copper(II) Monohydrate (I) and Aqua(1,10-phenanthroline)(L-phenylalaninato)copper(II) Nitrate Monohydrate (II)

By X. Solans

Dpto Cristalografía, Mineralogía y Depósitos Minerales, Universidad de Barcelona, Martí y Franqués, 08020 Barcelona, Spain

L. Ruiz-Ramírez, A. Martinez and L. Gasque

Dpto Química Inorgánica, Facultad de Química, Universidad Nacional Autónoma de México, CP, 04510 Mexico DF

and J. L. Briansó

Dpto Geologia, Universidad Autónoma de Barcelona, 08193 Bellaterra, Spain

Abstract. (I) $[CuCl(C_2H_4NO_2)(C_{12}H_8N_2)].H_2O$, $M_r = 371·28$, orthorhombic, $P2_12_12_1$, $a = 6·795$ (3), $b = 12·496$ (4), $c = 17·273$ (5) Å, $V = 1467$ (1) Å³, $D_x = 1·680$ Mg m⁻³, $Z = 4$, $F(000) = 756$, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu(Mo K\alpha) = 1·742$ mm⁻¹. Room temperature. Final $R = 0·046$ for 1302 unique observed reflections. (II) $[Cu(C_9H_{10}NO_2)(C_{12}H_8N_2)(H_2O)]NO_3.H_2O$, $M_r = 505·98$, monoclinic, $P2_1$, $a = 5·782$ (2), $b = 20·700$ (6), $c = 9·355$ (3) Å, $\beta = 97·58$ (2)°, $V = 1110$ (1) Å³, $D_x = 1·514$ Mg m⁻³, $Z = 2$, $F(000) = 522$, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu(Mo\ K\alpha) = 1·076$ mm⁻¹. Room temperature. Final $R = 0·069$ for 1929 unique observed reflections. The Cu ion displays distorted square-pyramidal coordination in both (I) and (II), with the chlorine atom (I) or the water molecule (II) in the apical position. The Cu—N bond lengths alter according to the electronegative character of the *trans* atom. The conformations of the five-membered chelate rings appear to depend on H bonding and van der Waals interactions.

Introduction. Interest in mixed-ligand chelate complexes has been clearly established in the last few years (Griesser & Sigel, 1970). A series of compounds with formula $M(N-N)(O-N)$ $(M = Cu$, N—N = 1,10-phenanthroline, bipyridine, substituted 1,10-phenan-

throline or bipyridine, and O—N = amino acid) has been synthesized and characterized, with stability constants determined potentiometrically (Ruiz-Ramírez, Martínez & Gasque, 1988). The crystal structures of two compounds of this series, (I) and (II), are presented here with a discussion of the effect of the amino-acid ligands on the environment of the metal ion.

Experimental. A similar process was used for data collection in both structures. Dark prismatic crystals [0·1 × 0·1 × 0·15 mm for (I) and 0·1 × 0·1 × 0·2 mm for (II)], Philips PW1100 diffractometer, Mo$K\alpha$, graphite monochromator, cell parameters from 25 reflections ($4 \le \theta \le 12°$), ω-scan technique, scan width 1°, scan speed 0·03° s^{-1}, 1373 independent reflections with $\theta \le 27°$; 1302 with $I \ge 2\cdot5\sigma(I)$ in (I); hkl range: 0 to 8; 0 to 14; and 0 to 20. 2423 independent with $\theta \le 25°$; 1929 with $I \ge 2\cdot5\sigma(I)$ in (II); hkl range: −6 to 6; 0 to 24; and 0 to 11. Three standard reflections measured every 2 h, no significant intensity decay, Lp correction, absorption ignored.

Both structures were solved by direct methods (*MULTAN;* Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Isotropic and aniso-tropic full-matrix least-squares refinement (*SHELX76;* Sheldrick, 1976), $\sum w||F_o| - |F_c||^2$ minimized, $w = [\sigma^2(F_o) + k|F_o|^2]^{-1}$, where $k = 0\cdot0004$ in (I) and 0·0 in (II), scattering factors and anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974). Ten H of (I) and all H of (II) were located from a difference synthesis and the remaining four H atoms of (I) were placed in computed positions, all H were refined with an overall isotropic temperature factor with the remaining atoms anisotropic. The four computed H atoms of (I) were constrained with C—H = 1·08 Å. Final R (wR) were 0·046 (0·052) for 1302 reflections in (I) and 0·069 (0·095) for 1929 reflections in (II). Max. $\Delta/\sigma = -0\cdot14$ in z of H(C3) in (I) and 0·25 in y of H(N8) in (II), max. and min. peaks in final $\Delta\rho$ map 0·4 and −0·3 e Å^{-3}, respectively, in both structures.

Discussion. Figs. 1 and 2 show views of the molecules with the numbering of atoms for structures (I) and (II), respectively. Final coordinates and selected bond lengths and angles are listed in Tables 1 and 2.* In both structures the Cu atom displays a distorted square-pyramidal coordination, with the phenanthroline and amino-acid ligands in the basal plane and a Cl atom in (I), or a water molecule in (II), in the apical site. The average Cu—O and Cu—N(amino acid) bond lengths are 1·936 (9) and 2·006 (8) Å, while the Cu—N-

(phenanthroline) bond length alters according to the electronegative character of the *trans* ligand: The Cu—N bond lengths *trans* to O are 0·033 (8) Å [average value from (I) and (II)] longer than those *trans* to N. This fact was also observed in (1,10-phenanthroline)(salicylaldehydato)copper(II) nitrate (Solans, Ruiz-Ramírez, Gasque & Briansó, 1987) where comparable values are 1·991 (3) and 2·005 (3) Å, respectively. The Cu—N(phenanthroline) distances are shorter than those observed in the *trans*-bis(1,10-phenanthroline)copper(II) complex (Boys, Escobar & Martínez-Carreras, 1981) [average value 2·05 (1) Å]. The Cu—O(W1) bond length in (II), 2·213 (10) Å, is intermediate between those observed for Cu—ONO$_2$-(apical) in nitratobis(2,9-dimethyl-1,10-phenanthro-line)copper(II) trichloroacetate trichloroacetic acid solvate where Cu—O = 2·15 (4) Å (Van Meerssche, Germain, Declercq & Wilputte-Steinert, 1981) and the value of 2·402 (3) Å in (1,10-phenanthroline)(sali-cylaldehydato)copper(II) nitrate (Solans, Ruiz-Ramírez, Gasque & Briansó, 1987). The Cu—Cl-(apical) bond length [2·546 (2) Å] in (I) is intermediate when compared with the following values: 2·233 (1) observed in di-μ-chloro-bis[chloro(1,2-cyclohexanedi-one dioxime-N,N')copper(II)] (Mégnamisi-Bélombé & Endres, 1983); 2·463 (1) in dichloro(2,6-diacetyl-pyridinedioxime)copper(II) dihydrate (Nicholson, Petersen & McCormick, 1982) and 2·565 (2) in dichloro(2,2':6',2''-terpyridyl)copper(II) monohydrate

Fig. 1. View of (I) with atom numbering.
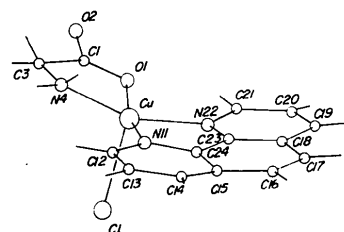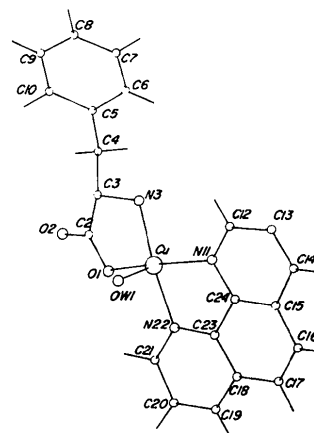

Fig. 2. View of (II) with atom numbering.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44607 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates* $[\times 10^4; Cu \times 10^5$ *in* (I)]

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	$B_{eq}(\text{Å}^2)$
(I)				
Cu	92001 (15)	51095 (7)	41420 (5)	2·96 (4)
Cl	9317 (4)	4218 (2)	8992 (1)	3·68 (8)
OW	5546 (14)	1498 (7)	9223 (6)	5·90 (44)
O(1)	9718 (10)	4572 (5)	3105 (4)	4·26 (29)
C(1)	10538 (13)	5221 (8)	2644 (5)	3·80 (37)
O(2)	10928 (12)	5023 (6)	1962 (3)	5·52 (34)
C(3)	11002 (16)	6330 (8)	2949 (5)	4·18 (40)
N(4)	10837 (13)	6364 (6)	3807 (4)	3·29 (29)
N(11)	8705 (10)	3610 (6)	4509 (4)	3·02 (29)
C(12)	8601 (12)	2722 (7)	4088 (6)	3·51 (36)
C(13)	8467 (13)	1692 (8)	4430 (7)	4·20 (44)
C(14)	8440 (14)	1609 (7)	5216 (6)	3·76 (41)
C(15)	8529 (12)	2545 (7)	5683 (5)	3·15 (35)
C(16)	8498 (15)	2532 (8)	6510 (6)	4·07 (42)
C(17)	8689 (15)	3468 (9)	6913 (6)	4·38 (45)
C(18)	8929 (13)	4479 (7)	6541 (5)	3·28 (34)
C(19)	9235 (17)	5449 (8)	6905 (5)	3·84 (38)
C(20)	9544 (15)	6370 (7)	6493 (5)	3·80 (40)
C(21)	9573 (14)	6309 (7)	5676 (5)	3·36 (37)
N(22)	9263 (11)	5396 (5)	5299 (3)	2·82 (25)
C(23)	8936 (12)	4480 (6)	5710 (4)	2·71 (30)
C(24)	8696 (11)	3514 (7)	5294 (5)	2·77 (33)
(II)				
Cu	2066 (2)	6582	4027 (1)	3·08 (6)
O(1)	−553 (12)	6489 (4)	5087 (9)	3·26 (31)
C(2)	−671 (20)	6899 (6)	6137 (10)	3·30 (39)
O(2)	−2301 (15)	6934 (6)	6842 (11)	4·92 (43)
C(3)	1451 (19)	7307 (5)	6554 (12)	3·10 (37)
N(3)	2930 (16)	7320 (5)	5378 (9)	3·11 (34)
C(4)	790 (19)	7988 (8)	7020 (13)	4·23 (50)
C(5)	2703 (20)	8331 (6)	7888 (11)	3·42 (43)
C(6)	4567 (26)	8653 (5)	7298 (17)	4·46 (53)
C(7)	6354 (28)	8947 (8)	8186 (22)	5·44 (72)
C(8)	6359 (25)	8984 (8)	9642 (24)	5·76 (74)
C(9)	4729 (32)	8664 (6)	10338 (17)	5·70 (64)
C(10)	2924 (27)	8354 (8)	9389 (21)	5·57 (67)
N(11)	728 (19)	5960 (4)	2555 (10)	3·36 (37)
C(12)	−1058 (24)	5575 (7)	2556 (16)	4·47 (54)
C(13)	−1945 (25)	5165 (8)	1392 (17)	4·74 (59)
C(14)	−919 (28)	5230 (7)	151 (20)	4·49 (57)
C(15)	1003 (24)	5597 (7)	75 (19)	4·52 (56)
C(16)	2303 (31)	5659 (9)	−1122 (14)	5·10 (68)
C(17)	4056 (27)	6067 (6)	−1156 (10)	3·77 (45)
C(18)	4816 (20)	6491 (5)	89 (9)	3·12 (39)
C(19)	6691 (21)	6913 (8)	182 (12)	4·42 (52)
C(20)	7198 (21)	7277 (6)	1386 (15)	3·85 (46)
C(21)	5842 (18)	7195 (6)	2552 (11)	3·23 (40)
N(22)	4094 (14)	6814 (6)	2515 (7)	3·16 (34)
C(23)	3525 (21)	6434 (6)	1318 (15)	3·88 (50)
C(24)	1764 (20)	5989 (6)	1321 (13)	3·49 (43)
O(W1)	4675 (17)	5892 (5)	5084 (11)	4·62 (38)
O(W2)	4402 (40)	4698 (8)	6177 (29)	10·23 (119)
N(31)	10480 (33)	8770 (10)	3337 (17)	6·61 (85)
O(31)	10417 (39)	9345 (8)	3023 (28)	4·94 (88)
O(32)	8579 (29)	8516 (9)	3572 (17)	7·56 (77)
O(33)	12258 (42)	8475 (12)	3397 (19)	5·97 (103)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

(I)

Cl–Cu	2·546 (2)		C(15)–C(14)	1·422 (13)
O(1)–Cu	1·945 (6)		C(16)–C(15)	1·429 (14)
N(4)–Cu	2·007 (7)		C(24)–C(15)	1·389 (11)
N(11)–Cu	2·007 (7)		C(17)–C(16)	1·367 (14)
N(22)–Cu	2·031 (6)		C(18)–C(17)	1·427 (12)
C(1)–O(1)	1·265 (11)		C(19)–C(18)	1·381 (12)
O(2)–C(1)	1·233 (10)		C(23)–C(18)	1·437 (11)
C(3)–C(1)	1·516 (13)		C(20)–C(19)	1·369 (13)
N(4)–C(3)	1·487 (10)		C(21)–C(20)	1·415 (12)
C(12)–N(11)	1·329 (11)		N(22)–C(21)	1·330 (10)
C(24)–N(11)	1·361 (10)		C(23)–N(22)	1·365 (9)
C(13)–C(12)	1·419 (13)		C(24)–C(23)	1·414 (11)
C(14)–C(13)	1·361 (15)			
O(1)–Cu–Cl	100·9 (2)		C(15)–C(14)–C(13)	120·1 (9)
N(4)–Cu–Cl	103·4 (2)		C(16)–C(15)–C(14)	123·9 (8)
N(4)–Cu–O(1)	84·5 (3)		C(24)–C(15)–C(14)	116·5 (8)
N(11)–Cu–Cl	100·6 (2)		C(24)–C(15)–C(16)	119·6 (8)
N(11)–Cu–O(1)	89·9 (3)		C(17)–C(16)–C(15)	119·9 (8)
N(11)–Cu–N(4)	156·0 (3)		C(18)–C(17)–C(16)	122·6 (9)
N(22)–Cu–Cl	93·5 (2)		C(19)–C(18)–C(17)	126·1 (8)
N(22)–Cu–O(1)	164·4 (3)		C(23)–C(18)–C(17)	116·8 (8)
N(22)–Cu–N(4)	97·7 (3)		C(23)–C(18)–C(19)	117·0 (7)
N(22)–Cu–N(11)	81·8 (3)		C(20)–C(19)–C(18)	121·6 (8)
C(1)–O(1)–Cu	116·0 (6)		C(21)–C(20)–C(19)	118·4 (8)
O(2)–C(1)–O(1)	124·6 (9)		N(22)–C(21)–C(20)	122·2 (8)
C(3)–C(1)–O(1)	117·4 (7)		C(21)–N(22)–Cu	129·5 (5)
C(3)–C(1)–O(2)	118·0 (9)		C(23)–N(22)–Cu	111·1 (5)
N(4)–C(3)–C(1)	110·8 (7)		C(23)–N(22)–C(21)	119·4 (6)
C(3)–N(4)–Cu	107·9 (6)		N(22)–C(23)–C(18)	121·4 (7)
C(12)–N(11)–Cu	128·0 (6)		C(24)–C(23)–C(18)	120·4 (7)
C(24)–N(11)–Cu	113·4 (5)		C(24)–C(23)–N(22)	118·1 (6)
C(24)–N(11)–C(12)	118·1 (7)		C(15)–C(24)–N(11)	124·0 (8)
C(13)–C(12)–N(11)	122·2 (9)		C(23)–C(24)–N(11)	115·5 (7)
C(14)–C(13)–C(12)	119·1 (10)		C(23)–C(24)–C(15)	120·5 (7)

(II)

O(1)–Cu	1·927 (6)		C(24)–N(11)	1·370 (17)
N(3)–Cu	2·004 (9)		C(13)–C(12)	1·422 (18)
N(11)–Cu	1·968 (9)		C(14)–C(13)	1·378 (26)
N(22)–Cu	2·010 (9)		C(15)–C(14)	1·356 (21)
O(W1)–Cu	2·213 (10)		C(16)–C(15)	1·435 (22)
C(2)–O(1)	1·306 (15)		C(24)–C(15)	1·439 (17)
O(2)–C(2)	1·222 (16)		C(17)–C(16)	1·322 (24)
C(3)–C(2)	1·498 (14)		C(18)–C(17)	1·478 (15)
N(3)–C(3)	1·480 (12)		C(19)–C(18)	1·386 (20)
C(4)–C(3)	1·538 (17)		C(23)–C(18)	1·456 (14)
C(5)–C(4)	1·467 (17)		C(20)–C(19)	1·356 (20)
C(6)–C(5)	1·438 (18)		C(21)–C(20)	1·435 (16)
C(10)–C(5)	1·393 (21)		N(22)–C(21)	1·279 (16)
C(7)–C(6)	1·378 (22)		C(23)–N(22)	1·372 (16)
C(8)–C(7)	1·364 (29)		C(24)–C(23)	1·374 (18)
C(9)–C(8)	1·383 (27)		O(31)–N(31)	1·224 (28)
C(10)–C(9)	1·430 (20)		O(32)–N(31)	1·264 (21)
C(12)–N(11)	1·304 (17)		O(33)–N(31)	1·190 (27)
N(3)–Cu–O(1)	84·2 (3)		C(24)–N(11)–Cu	112·7 (8)
N(11)–Cu–O(1)	91·8 (4)		C(24)–N(11)–C(12)	117·5 (10)
N(11)–Cu–N(3)	169·4 (4)		C(13)–C(12)–N(11)	124·7 (14)
N(22)–Cu–O(1)	163·1 (3)		C(14)–C(13)–C(12)	115·8 (13)
N(22)–Cu–N(3)	98·2 (4)		C(15)–C(14)–C(13)	123·1 (14)
N(22)–Cu–N(11)	82·9 (4)		C(16)–C(15)–C(14)	127·4 (15)
O(W1)–Cu–O(1)	103·9 (3)		C(24)–C(15)–C(14)	116·0 (15)
O(W1)–Cu–N(3)	96·4 (4)		C(24)–C(15)–C(16)	116·5 (14)
O(W1)–Cu–N(11)	94·1 (4)		C(17)–C(16)–C(15)	123·9 (14)
O(W1)–Cu–N(22)	92·4 (3)		C(18)–C(17)–C(16)	120·6 (11)
C(2)–O(1)–Cu	116·4 (6)		C(19)–C(18)–C(17)	124·9 (9)
O(2)–C(2)–O(1)	124·4 (10)		C(23)–C(18)–C(17)	116·4 (11)
C(3)–C(2)–O(1)	116·1 (10)		C(23)–C(18)–C(19)	118·6 (10)
C(3)–C(2)–O(2)	119·3 (11)		C(20)–C(19)–C(18)	119·0 (9)
N(3)–C(3)–C(2)	110·5 (9)		C(21)–C(20)–C(19)	119·1 (10)
C(4)–C(3)–C(2)	111·4 (9)		N(22)–C(21)–C(20)	124·1 (10)
C(4)–C(3)–N(3)	112·5 (8)		C(21)–N(22)–Cu	131·3 (7)
C(3)–N(3)–Cu	109·6 (6)		C(23)–N(22)–Cu	109·7 (7)
C(5)–C(4)–C(3)	113·8 (9)		C(23)–N(22)–C(21)	118·7 (9)
C(6)–C(5)–C(4)	124·1 (10)		N(22)–C(23)–C(18)	120·3 (10)
C(10)–C(5)–C(4)	122·5 (13)		C(24)–C(23)–C(18)	120·9 (11)
C(10)–C(5)–C(6)	113·4 (13)		C(24)–C(23)–N(22)	118·8 (10)
C(7)–C(6)–C(5)	120·9 (15)		C(15)–C(24)–N(11)	122·5 (12)
C(8)–C(7)–C(6)	122·0 (16)		C(23)–C(24)–N(11)	115·9 (9)
C(9)–C(8)–C(7)	122·1 (14)		C(23)–C(24)–C(15)	121·5 (12)
C(10)–C(9)–C(8)	114·2 (16)		O(32)–N(31)–O(31)	116·6 (22)
C(9)–C(10)–C(5)	127·1 (18)		O(33)–N(31)–O(31)	120·5 (20)
C(12)–N(11)–Cu	129·6 (9)		O(33)–N(31)–O(32)	122·9 (22)

(Rojo, Vlasse & Beltran-Porter, 1983); 2·647 (1) in chlorodiiodo[1-(2-pyridylazo)-2-naphtholate]copper-(II) (Desiraju, Luss & Smith, 1978); 2·698 (3) in dichloro(dimethylglyoxime)copper(II) (Svedung, 1969) and 2·734 (4) Å in dichloro(N,N'-dimethylethylenedi-amine)copper(II) (Phelps, Goodman & Hodgson, 1976). The loss of planarity of the Cu atom with respect to the basal plane [deviation of Cu from mean plane = 0·334 (5) in (I) and 0·230 (6) Å in (II)] and the lengthening of the Cu–apical bond may possibly be explained by a Jahn–Teller effect.

The Cu–N–C–C–N(phenanthroline) five-membered rings are planar [largest deviation

0·016 (7) Å at C(23) of (I)]. The Cu—O—C—C—N(amino acid) five-membered chelate rings have an envelope conformation with N(4) 0·326 (7) Å out of plane in (I) and C(3) 0·256 (9) Å out of plane in (II). These variations in conformation are assigned to packing forces. In compound (I) O(2) and N(4) are hydrogen bonded to the hydrate water molecule [lengths: $OW \cdots O(2^i)$ 2·805 (8) and $OW \cdots N(4^{ii})$ 3·15 (2) Å; symmetry code: (i) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (ii) $\frac{3}{2}-x$, $1-y, \frac{1}{2}+z$], while in (II) only $O(W1)$ (apical position) is strongly hydrogen bonded to $O(W2)$ [$O(W2) \cdots O(W1^i)$ 2·69 (1) Å; (i) x, y, z].

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Structure of {N-[2-(2-Aminoethylamino)ethyl]salicylideneaminato-O,N,N',N''}nickel(II) Perchlorate

BY J. PODLAHOVÁ, K. KNÍŽEK AND J. LOUB*

Department of Inorganic Chemistry, Charles University, Hlavova 8/2030, 128 40 Praha 2, Czechoslovakia

AND J. HAŠEK

Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, Heyrovského nám. 2, 162 06 Praha 6, Czechoslovakia

Abstract. [Ni($C_{11}H_{16}N_3O$)]ClO$_4$, $M_r = 364·43$, monoclinic, $P2_1/c$, $a = 8·930$ (4), $b = 13·391$ (5), $c = 11·902$ (6) Å, $\beta = 98·34$ (4)°, $V = 1408$ (1) Å³, $Z = 4$, $D_m = 1·74$ (2), $D_x = 1·718$ (1) Mg m⁻³, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1·59$ mm⁻¹, $F(000) = 752$, $T = 295$ K, $R = 0·078$ for 2498 unique observed reflections. The structure consists of [Ni($C_{11}H_{16}N_3O$)]⁺ complex cations and perchlorate anions. The coordination polyhedron around Ni is a distorted square with the tetradentate $C_{11}H_{16}N_3O^-$ ligand (saden) bonded to the metal through one O and three N atoms. The perchlorate anion is disordered.

Introduction. The study of the title compound was undertaken as part of an investigation of complexes with unsymmetrical tetradentate Schiff bases (Haber, 1986).

Experimental. The crystals were prepared by crystallization from a mixture of nickel(II) perchlorate, salicylaldehyde and diethylenetriamine from aqueous ethanol (Haber, 1986). Orange crystals were obtained, stable in air and to X-rays. Density determined pycnometrically. A sphere-shaped crystal of $r = 0·15$ mm was used for the measurements. Syntex $P2_1$ diffractometer, graphite monochromator, 20 reflections with 2θ from 8·86 to 20·66° for measuring lattice

* To whom correspondence should be addressed.