Table 1. *Atomic fractional coordinates and equivalent isotropic displacement parameters* (Å²)

	x	y	z	U_{eq}*
Pt	0·06238 (3)	0·06724 (2)	0·11786 (5)	0·048
Cl(1)	0·0384 (3)	0·1553 (2)	−0·0853 (5)	0·099
Cl(2)	−0·0218 (2)	0·1278 (2)	0·2922 (5)	0·083
S(1)	0·0869 (2)	−0·0176 (2)	0·3166 (4)	0·051
S(2)	0·1400 (2)	0·0067 (2)	−0·0553 (4)	0·072
F(1)	0·1314 (5)	−0·1887 (5)	0·3129 (13)	0·111
F(2)	0·1950 (6)	−0·1271 (6)	0·4884 (14)	0·133
F(3)	0·2509 (5)	−0·1715 (5)	0·2752 (15)	0·137
C(1)	0·1263 (9)	0·0324 (8)	0·4886 (17)	0·087
C(2)	0·2279 (9)	0·0551 (9)	−0·0540 (2)	0·101
C(3)	0·1728 (6)	−0·0628 (7)	0·2383 (16)	0·062
C(4)	0·1865 (11)	−0·1406 (11)	0·3234 (26)	0·109
C(5)	0·1641 (8)	−0·0767 (8)	0·0587 (18)	0·080

* U_{eq} is the mean latent root of the anisotropic vibration tensor.

Table 2. *Bond lengths* (Å) *and interbond angles* (°)

Pt—Cl(1)	2·303 (4)	Pt—Cl(2)	2·308 (4)
Pt—S(1)	2·241 (3)	Pt—S(2)	2·231 (4)
S(1)—C(1)	1·791 (14)	S(1)—C(3)	1·819 (12)
S(2)—C(2)	1·761 (17)	S(2)—C(5)	1·784 (15)
F(1)—C(4)	1·288 (20)	F(2)—C(4)	1·372 (23)
F(3)—C(4)	1·314 (19)	C(3)—C(4)	1·551 (18)
C(3)—C(5)	1·490 (19)		
Cl(1)—Pt—Cl(2)	90·9 (2)	Cl(1)—Pt—S(1)	179·3 (4)
Cl(1)—Pt—S(2)	88·7 (2)	Cl(2)—Pt—S(1)	89·2 (2)
Cl(2)—Pt—S(2)	177·9 (2)	S(1)—Pt—S(2)	91·2 (2)
Pt—S(1)—C(1)	108·2 (5)	Pt—S(1)—C(3)	101·3 (4)
C(1)—S(1)—C(3)	99·7 (7)	Pt—S(2)—C(2)	107·6 (6)
Pt—S(2)—C(5)	102·0 (5)	C(2)—S(2)—C(5)	100·7 (8)
S(1)—C(3)—C(4)	110·9 (10)	S(1)—C(3)—C(5)	109·3 (9)
C(4)—C(3)—C(5)	108·0 (14)	F(1)—C(4)—F(2)	105·1 (14)
F(1)—C(4)—F(3)	110·8 (18)	F(1)—C(4)—C(3)	115·6 (15)
F(2)—C(4)—F(3)	105·7 (16)	F(2)—C(4)—C(3)	107·6 (17)
F(3)—C(4)—C(3)	111·4 (13)	S(2)—C(5)—C(3)	113·6 (10)

The Pt—S and Pt—Cl bond lengths in Table 2 may be compared with corresponding values in other *cis*-$PtCl_2L$ complexes: Pt—S 2·231 (1), 2·244 (2) Å, Pt—Cl 2·324 (2), 2·316 (2) Å, L = MeSCH(CF_3)CH(CF_3)-SMe (Hunter, Muir & Sharp, 1984); Pt—S 2·240 (3), 2·270 (3) Å, Pt—Cl 2·306 (3), 2·299 (3) Å, L = MeSCF_2CH_2SMe (Cano, Leal, Quintana & Torrens, 1984); Pt—S 2·239 (3), 2·260 (3) Å, Pt—Cl 2·295 (3), 2·290 (4) Å, L = $CF_3SCH_2CH_2SCF_3$ (Manojlović-Muir *et al.*, 1977). Evidently, the Pt—S bond lengths in these complexes are insensitive to the pattern of substitution at the ring S or C atoms. The Pt—Cl(*trans* to SCH_3) distances are 0·01–0·03 Å longer than the Pt—Cl(*trans* to SCF_3) distances, implying a stronger *trans* influence for SCH_3 than for SCF_3, but the effect is small and only barely significant statistically.

We thank SERC for a studentship (to WNH) and for support in purchasing the diffractometer and computer.

References

Cano, O., Leal, J., Quintana, P. & Torrens, H. (1984). *Inorg. Chim. Acta*, **89**, L9–L10.

Cross, R. J., Rycroft, D. S., Sharp, D. W. A. & Torrens, H. (1980). *J. Chem. Soc. Dalton Trans.* pp. 2434–2441.

Hunter, W. N., Muir, K. W. & Sharp, D. W. A. (1984). *Acta Cryst.* C**40**, 37–39.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99 and 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Mallinson, P. R. & Muir, K. W. (1985). *J. Appl. Cryst.* **18**, 51–53.

Manojlović-Muir, Lj., Muir, K. W. & Solomun, T. (1977). *Inorg. Chim. Acta*, **22**, 69–73.

Torrens, H. (1977). PhD thesis, Univ. of Glasgow.

Acta Cryst. (1985). C**41**, 1752–1755

ab,cf-Bis[4-(2-aminoethyl)imidazole-N^3,N^8]-*de*-bis(isothiocyanato)nickel(II)

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(*Received* 14 *February* 1985; *accepted* 9 *August* 1985)

Abstract. [$Ni(NCS)_2(C_5H_9N_3)_2$], M_r = 397·17, monoclinic, $P2_1/c$, a = 8·4903 (5), b = 14·224 (1), c = 14·5746 (9) Å, β = 93·248 (5)°, V = 1757·3 (2) Å³, Z = 4, D_m = 1·50 (1), D_x = 1·5125 (1) Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 2·80 mm^{-1}, F(000) = 816, T = 293 K, R = 0·0309 for 2349 observed reflections. The six N atoms coordinated to Ni^{2+} form a distorted octahedron. Both histamine ligands are coordinated to Ni^{2+} *via* their N^{π} atoms and amino groups of the side chains. The structure contains a three-dimensional

network of hydrogen bonds, with the N$^{\tau}$—H and amino groups as donors and the S atoms of the NCS$^-$ ions as acceptors.

Introduction. The additive action of endopeptidases and exopeptidases on proteins in a living organism results in a mixture of amino acids. These amino acids are partly enzymatically decarboxylated into amines, some of which possess important physiological activity. Histamine, which is the product of enzymatic decarboxylation of histidine, is one of those amines. It is present in mammalian blood and tissues, and is a powerful vasodilator and stimulator of gastric secretion (Kowalewski, Chmura & Shier, 1969; Hess, 1968). Histamine is also interesting from a chemical point of view since it may exist in two tautomeric forms N$^{\pi}$—H and N$^{\tau}$—H.[*] Containing three N atoms, histamine can act as a mono-, bi- or tridentate ligand. The present study has been undertaken to determine the tautomeric form, the mode of complexation and the hydrogen-bond network in the title structure.

Experimental. Crystals from water, density by flotation method, 15 reflections with $37 < 2\theta < 50°$ used for measuring lattice parameters, 2766 reflections up to $2\theta = 115°$ ($h\ 0\rightarrow10$, $k\ 0\rightarrow16$, $l\ -16\rightarrow16$) measured on a Syntex $P2_1$ diffractometer for $0{\cdot}27 \times 0{\cdot}28 \times 0{\cdot}25$ mm crystal using Cu $K\alpha$ radiation, graphite monochromator, 2629 unique reflections, $R_{\text{int}} = 0{\cdot}0399$, 2353 observed reflections with $I \geq 1{\cdot}96\sigma(I)$, no significant intensity variations for two standard reflections, no absorption corrections, four reflections ($21\bar{1}$, 100, 002, 200) omitted because of extinction, structure solved by Patterson method, $\sum(\Delta F)^2$ minimized by anisotropic full-matrix least-squares refinement, unit weights, H-atom parameters found from $\Delta\rho$ map and refined isotropically except for the fixed C(13)—H(131), H(132) group, final $R = 0{\cdot}0309$ and $S = 0{\cdot}9932$ for 2349 reflections, $(\Delta/\sigma)_{\text{max}}$ in final refinement cycle 0·28, $|\Delta\rho|_{\text{max}} = 0{\cdot}26$ e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used: *SHELX*76 (Sheldrick, 1976), *PLUTO*78 (Motherwell & Clegg, 1978) and those described by Jaskólski (1982), RIAD-32 computer.

Discussion. The atomic parameters, and bond lengths and angles are given in Tables 1 and 2.[†] Fig. 1 shows the atom-numbering scheme of the molecule.

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{\text{eq}} = (U_{11} \times U_{22} \times U_{33})^{1/3}$.

	x	y	z	U_{eq}
Ni	0·15620 (6)	0·10598 (3)	0·27550 (3)	0·0355 (2)
N(1)	0·3874 (3)	0·0852 (2)	0·2307 (2)	0·044 (1)
C(2)	0·4239 (4)	0·1161 (3)	0·1380 (3)	0·053 (1)
C(3)	0·3190 (4)	0·0671 (3)	0·0659 (3)	0·057 (1)
C(4)	0·1526 (4)	0·1029 (2)	0·0603 (2)	0·046 (1)
C(5)	0·0573 (5)	0·1246 (3)	−0·0143 (3)	0·055 (2)
N(6)	−0·0833 (4)	0·1526 (2)	0·0163 (2)	0·050 (1)
C(7)	−0·0715 (4)	0·1481 (2)	0·1080 (2)	0·043 (1)
N(8)	0·0690 (3)	0·1189 (2)	0·1385 (2)	0·0381 (9)
N(11)	0·0879 (4)	−0·0368 (2)	0·2652 (2)	0·045 (1)
C(12)	0·0803 (5)	−0·0958 (3)	0·3482 (3)	0·063 (2)
C(13)	0·2370 (5)	−0·0954 (3)	0·4029 (3)	0·067 (2)
C(14)	0·2646 (4)	−0·0049 (3)	0·4530 (2)	0·052 (1)
C(15)	0·3083 (5)	0·0088 (3)	0·5419 (3)	0·064 (2)
N(16)	0·3197 (4)	0·1031 (3)	0·5543 (2)	0·062 (1)
C(17)	0·2814 (5)	0·1449 (3)	0·4738 (3)	0·058 (1)
N(18)	0·2471 (3)	0·0818 (2)	0·4097 (2)	0·0438 (9)
N(21)	−0·0649 (3)	0·1451 (2)	0·3208 (2)	0·048 (1)
C(22)	−0·1714 (4)	0·1918 (2)	0·3319 (2)	0·040 (1)
S(23)	−0·3262 (1)	0·25958 (7)	0·34597 (7)	0·0554 (3)
N(31)	0·2202 (3)	0·2468 (2)	0·2823 (2)	0·047 (1)
C(32)	0·2653 (3)	0·3228 (2)	0·2858 (2)	0·035 (1)
S(33)	0·3236 (1)	0·43229 (6)	0·29088 (7)	0·0536 (3)

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

Ni—N(1)	2·125 (3)	C(7)—N(8)	1·316 (4)
Ni—N(8)	2·098 (3)	N(11)—C(12)	1·476 (5)
Ni—N(11)	2·115 (3)	C(12)—C(13)	1·511 (5)
Ni—N(18)	2·090 (3)	C(13)—C(14)	1·492 (5)
Ni—N(21)	2·100 (3)	C(14)—C(15)	1·342 (5)
Ni—N(31)	2·077 (3)	C(14)—N(18)	1·389 (4)
N(1)—C(2)	1·470 (5)	C(15)—N(16)	1·356 (6)
C(2)—C(3)	1·509 (5)	N(16)—C(17)	1·338 (5)
C(3)—C(4)	1·499 (5)	C(17)—N(18)	1·317 (4)
C(4)—C(5)	1·354 (5)	N(21)—C(22)	1·141 (4)
C(4)—N(8)	1·395 (4)	C(22)—S(23)	1·652 (4)
C(5)—N(6)	1·357 (5)	N(31)—C(32)	1·147 (4)
N(6)—C(7)	1·335 (4)	C(32)—S(33)	1·635 (4)

N(8)—Ni—N(1)	90·1 (1)	C(7)—N(6)—C(5)	107·3 (3)
N(11)—Ni—N(1)	95·7 (1)	N(8)—C(7)—N(6)	111·6 (3)
N(11)—Ni—N(8)	86·3 (1)	C(4)—N(8)—Ni	126·5 (2)
N(18)—Ni—N(1)	88·1 (1)	C(7)—N(8)—Ni	127·8 (2)
N(18)—Ni—N(8)	175·4 (1)	C(7)—N(8)—C(4)	105·6 (3)
N(18)—Ni—N(11)	89·6 (1)	C(12)—N(11)—Ni	120·8 (3)
N(21)—Ni—N(1)	172·6 (1)	C(13)—C(12)—N(11)	110·6 (3)
N(21)—Ni—N(8)	90·1 (1)	C(14)—C(13)—C(12)	111·8 (3)
N(21)—Ni—N(11)	91·7 (1)	C(15)—C(14)—C(13)	128·7 (4)
N(21)—Ni—N(18)	92·2 (1)	N(18)—C(14)—C(13)	122·2 (3)
N(31)—Ni—N(1)	84·5 (1)	N(18)—C(14)—C(15)	109·0 (3)
N(31)—Ni—N(8)	92·2 (1)	N(16)—C(15)—C(14)	106·6 (4)
N(31)—Ni—N(11)	178·5 (1)	C(17)—N(16)—C(15)	108·1 (4)
N(31)—Ni—N(18)	91·9 (1)	N(18)—C(17)—N(16)	110·7 (4)
N(31)—Ni—N(21)	88·0 (1)	C(14)—N(18)—Ni	126·5 (2)
C(2)—N(1)—Ni	119·2 (2)	C(17)—N(18)—Ni	127·4 (2)
C(3)—C(2)—N(1)	110·8 (3)	C(17)—N(18)—C(14)	105·6 (3)
C(4)—C(3)—C(2)	113·5 (3)	C(22)—N(21)—Ni	158·2 (3)
C(5)—C(4)—C(3)	129·8 (3)	S(23)—C(22)—N(21)	179·0 (3)
N(8)—C(4)—C(3)	122·2 (3)	C(32)—N(31)—Ni	175·6 (3)
N(8)—C(4)—C(5)	108·1 (3)	S(33)—C(32)—N(31)	178·1 (3)
N(6)—C(5)—C(4)	107·4 (3)		

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[*] Following the IUPAC–IUB Commission on Biochemical Nomenclature (1970) recommendations for histidine, the imidazole N atom nearer the side chain is designated N$^{\pi}$ and the farther one N$^{\tau}$ (Black & Ganellin, 1974).

[†] Lists of structure factors, anisotropic thermal parameters, H-atom parameters, details of the hydrogen bonding and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42424 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The biological activity of histamine (hm) is connected with the N^{τ}—H form (H_1-receptor) (Durrant, Ganellin & Parsons, 1975). In contrast to results found in $[Ni(NCS)_4(Hhm^+)_2]$ (Wojtczak, Jaskólski & Kosturkiewicz, 1983), in the present compound histamine exists in the N^{τ}—H active form, which is stabilized by its bidentate coordination.

The imidazole (im) bond lengths and angles are comparable to those found in $[Pd(hm)_2]Cl_2$ (Dahan, 1976) and $[Cu(ClO_4)_2(hm)_2]$ (Bonnet & Jeannin, 1970*a*) and determine the histamine form as 4-(2-aminoethyl)imidazole.

The imidazole rings in both histamine ligands are planar (χ^2 3·97 and 2·27). The dihedral angle between their best planes is 106·43°.

Bond lengths and angles in the side chains are similar to those found in histamine base (Bonnet & Ibers, 1973) and in the complexes mentioned above.

In the present structure histamine exists in the +synclinal form. Torsion angles C(4)—C(3)—C(2)—N(1) and C(5)—C(4)—C(3)—C(2) in the two histamine ligands are 74·0 (4), 135·0 (4)° and 74·5 (4), 129·7 (4)° respectively. These angles are significantly different from those [69·4 (4) and −92·1 (4)°] found in $[Ni(NCS)_4(Hhm^+)_2]$ (Wojtczak *et al.*, 1983).

Both histamine molecules act as bidentate ligands coordinated to Ni^{2+} *via* the N^{π} atoms of the imidazole rings and the amino groups of the side chains. The coordination sphere (distorted octahedron) is completed by two NCS^- ions. The Ni—N(CS) [2·100 (3), 2·077 (3) Å] and Ni—N(im) [2·098 (3), 2·090 (3) Å] distances are similar to those found in $[Ni(NCS)_4(Hhm^+)_2]$ (Wojtczak *et al.*, 1983). The Ni—N(amino) distances [2·125 (3), 2·115 (3) Å] are a little longer than those found in $[Ni(ClO_4)(hm)_2(H_2O)]ClO_4$ and a little shorter than those found in $[Ni(hm)_3](ClO_4)_2$ (Bonnet & Jeannin, 1970*b*, 1972).

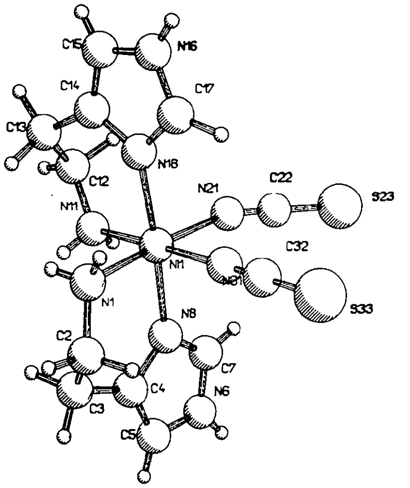

Fig. 1. View of the molecule, showing the atomic numbering.

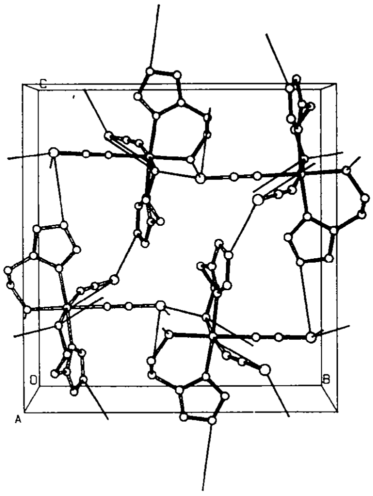

Fig. 2. Perspective view of the crystal packing of $[Ni(NCS)_2(C_5H_9N_3)_2]$. Hydrogen bonds are indicated by thin lines.

The complex molecule possesses an approximately pseudo-crystallographic twofold axis situated on the bisectrix of the N(31)—Ni—N(21) angle.

The structure contains a three-dimensional network of hydrogen bonds (Fig. 2), with the N^{τ}—H and amino groups as donors and the S atoms of the NCS^- ions as acceptors. The S(23) atom participates in two non-equivalent hydrogen bonds [H···S 3·05 (4), 2·53 (4) Å] and in consequence the Ni—N(21)—C(22) angle [158·2 (3)°] deviates significantly from 180°. S(33) participates in three similar hydrogen bonds [H···S 2·67 (4), 2·78 (4) and 2·91 (5) Å]. The Ni—N(31)—C(32) angle is 178·6 (3)°.

The authors thank Dr J. Wojtczak for suggesting the problem and providing the compound.

References

BLACK, J. W. & GANELLIN, C. R. (1974). *Experientia*, **30**, 111–113.

BONNET, J. J. & IBERS, A. (1973). *J. Am. Chem. Soc.* **95**, 4829–4833.

BONNET, J. J. & JEANNIN, Y. (1970*a*). *Acta Cryst.* B**26**, 318–326.

BONNET, J. J. & JEANNIN, Y. (1970*b*). *Bull. Soc. Fr. Minéral. Cristallogr.* **93**, 287–299.

BONNET, J. J. & JEANNIN, Y. (1972). *Bull. Soc. Fr. Minéral. Cristallogr.* **95**, 61–67.

DAHAN, F. (1976). *Acta Cryst.* B**32**, 2472–2475.

DURRANT, G. J., GANELLIN, C. R. & PARSONS, M. E. (1975). *J. Med. Chem.* **18**, 905–909.

HESS, H. J. (1968). *Annu. Rep. Med. Chem.* pp. 56–66.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

IUPAC–IUB Commission on Biochemical Nomenclature (1970). *J. Mol. Biol.* **52**, 1–17.
Jaskólski, M. (1982). *Collected Abstracts of the Fourth Symposium on Organic Crystal Chemistry*, Poznań, September 1982, edited by Z. Kałuski, pp. 70–71. A. Mickiewicz Univ., Poznań.
Kowalewski, K., Chmura, G. & Shier, J. (1969). *Am. J. Dig. Dis.* **14**, 788–796.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
Wojtczak, A., Jaskólski, M. & Kosturkiewicz, Z. (1983). *Acta Cryst.* C**39**, 545–547.

Acta Cryst. (1985). C**41**, 1755–1758

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

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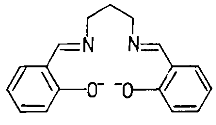
(*Received* 26 *March* 1985; *accepted* 10 *September* 1985)

Abstract. (1) [Ni($C_{17}H_{16}N_2O_2$)], $M_r = 338\cdot8$, orthorhombic, $Bm2_1b$ (non-standard setting of No. 36 with $b\rightarrow c$ and $c\rightarrow b$), $a = 25\cdot080\,(11)$, $b = 6\cdot71\,(1)$, $c = 8\cdot82\,(1)$ Å, $U = 1484\cdot3$ Å^3, $Z = 4$, $D_m = 1\cdot52$, $D_x = 1\cdot52$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 13\cdot1$ cm^{-1}, $F(000) = 704$, $T = 298$ K, $R = 0\cdot055$ 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($C_{17}H_{16}N_2O_2$)], $M_r = 343\cdot7$, orthorhombic, $Pn2_1a$ (non-standard setting of No. 33 with $b\rightarrow c$ and $c\rightarrow b$), $a = 11\cdot925\,(11)$, $b = 6\cdot890\,(6)$, $c = 17\cdot780\,(8)$ Å, $U = 1460\cdot9$ Å^3, $Z = 4$, $D_m = 1\cdot56\,(2)$, $D_x = 1\cdot56$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 15\cdot6$ cm^{-1}, $F(000) = 708$, $T = 298$ K, $R = 0\cdot058$ 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

0108-2701/85/121755-04$01.50