liés à Pt(1) forment un quadrilatère qui, compte tenu des incertitudes, est assimilable à un carré. En effet, les distances Pt(1)—Cl(1) et Pt(1)—Cl(2) sont pratiquement égales [2,305 (2) et 2,306 (1) Å] et l'angle Cl(1)—Pt(1)—Cl(2) mesure 89,86 (5)°. L'environnement de Pt(2) comprend deux atomes de chlore et quatre atomes d'azote appartenant à deux molécules de propanediamine-1,3. Ces six atomes réalisent un octaèdre déformé. Les liaisons Pt(2)—Cl(3), Pt(2)—N(1) et Pt(2)—N(5) mesurent respectivement 2,302 (2), 2,076 (4) et 2,065 (4) Å. Les valeurs des angles Cl(3)—Pt(2)—N(1) [87,0 (1)°] et Cl(3)—Pt(2)—N(5) [86,6 (1)°] montrent que la droite Cl(3)—Pt(2)—Cl(3^i) [(i) $1-x$, $-y$, $1-z$] n'est pas perpendiculaire au plan P(I) du parallélogramme N(1)N(5)N(1^i)N(5^i). Dans ce dernier, l'angle N(1)—Pt(2)—N(5) vaut 87,5 (2)°.

On remarque que les longueurs des liaisons Pt^{II}—Cl de l'anion et Pt^{IV}—Cl du cation sont très voisines et qu'elles sont comparables à celles des liaisons Pt—Cl présentes dans le tétrachlorure tétrachloroplatinate(II) de bis(diéthylènetriammonium) [2,306 (1) et 2,293 (1) Å] (Britten & Lock, 1979) ou dans le chlorure de *trans*-dichlorobis(propanediamine-1,3)platine(IV) tétrahydrate [2,304 (1) Å] (Delafontaine, Toffoli, Khodadad & Rodier, 1988). De même, les distances Pt(2)—N(1) et Pt(2)—N(5) sont du même ordre que leurs homologues dans $[Pt(NH_3)_4][PtBr_2(NH_3)_4](HSO_4)_4$ [2,056 (6) et 2,061 (6) Å] (Tanaka, Tsujikawa, Toriumi & Ito, 1982).

Comme dans le chlorure de *trans*-dichlorobis(propanediamine-1,3)platine(IV) tétrahydrate déjà cité, le cycle formé par l'atome de platine et l'une des molécules d'amine présente la conformation chaise. Pt(2) et C(3) sont situés de part et d'autre du plan moyen P(II) défini par N(1), C(2), C(4) et N(5); le premier en est distant de 0,973 (1) Å, le second de 0,665 (6) Å. Les liaisons N—C et C—C contenues dans ce cycle ont des longueurs normales. N(1)N(5) et C(2)C(4) sont sensiblement perpendiculaires au plan Cl(3)Pt(2)C(3) [plan P(III)], car les angles que font les plans P(I), P(II) et C(2)C(3)C(4) avec P(III) valent respectivement 90,3 (1), 90,2 (1) et 90,5 (2)°. En outre, les distances à P(III) de N(1) et de N(5) ou celles de C(2) et de C(4) sont égales aux incertitudes près. Donc, P(III) est approximativement plan de symétrie pour le cation.

Plusieurs liaisons hydrogène N—H···Cl participent à la cohésion de la structure. Leurs longueurs et leurs angles sont rapportés dans le Tableau 2.

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Structures of Bis(piperidine-1-dithiocarbamato)nickel(II) and Bis(piperidine-1-dithiocarbamato)copper(II)

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(*Received* 28 *September* 1987; *accepted* 5 *April* 1988)

Abstract. $[Ni(C_6H_{10}NS_2)_2]$, $M_r = 378\cdot7$, monoclinic, $P2_1/c$, $a = 6\cdot122$ (2), $b = 8\cdot537$ (1), $c = 15\cdot156$ (3) Å, $\beta = 95\cdot86$ (2)°, $V = 788$ Å^3, $Z = 2$, $D_m = 1\cdot60$, $D_x = 1\cdot595$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6\cdot34$ mm^{-1}, $F(000) = 392\cdot0$, room temperature, $R = 0\cdot065$, $wR = 0\cdot062$ for 1201 unique reflections. $[Cu(C_6H_{10}NS_2)_2]$, $M_r = 383\cdot5$, monoclinic, $P2_1/c$, $a = 6\cdot159$ (1), $b = 8\cdot567$ (1), $c = 15\cdot264$ (2) Å, $\beta = 95\cdot52$ (1)°, $V = 802$ Å^3, $Z = 2$, $D_m = 1\cdot60$, $D_x = 1\cdot60$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 6\cdot41$ mm^{-1}, $F(000) = 394$, room temperature, $R = 0\cdot078$, $wR = 0\cdot073$ for 1207 unique reflections. The nickel and

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copper complexes are isomorphous with the central metal atom lying on an inversion centre and exhibiting a pseudo square-planar configuration around it. The piperidine ring has a chair conformation.

Introduction. 1,1-Dithio-acids are formed by the reaction of carbon disulfide with various nucleophiles. Metal ions readily react with these dithio-acids to yield complexes in which the two S atoms are bonded to the same metal atom, thus forming a four-membered chelate ring. The chemistry of complexes of Ni and Cu with dithiocarbamates has been extensively studied (Coucouvanis, 1979). Several X-ray crystallographic structural determinations of Ni^{II} and Cu^{II} dithiocarbamates have been reported in recent years. Thus structures of $[Ni(R_1R_2dtc)_2]$ with $R_1 = R_2 = CH_3$ (Peyronel & Pegnedoli, 1967), $R_1 = R_2 = H$ (Gasparri, Nardelli & Villa, 1967), $R_1 = R_2 = C_2H_5$ (Coucouvanis, 1979), $R_1 = R_2 = C_2H_4OH$ (Ramalingam, Radha, Aravamudan, Mahadevan, Subrahmanyam & Seshasayee, 1984), $R_1 = CH_3$, $R_2 = C_6H_5$ (Martin, Newman, Robinson & White, 1972), $R_1R_2 = (CH_2)_4$ (Raston & White, 1974), $R_1R_2 = (CH_2)_6$ (Agre, Shugan & Rukhadze, 1968) and of $[Cu(R_1R_2dtc)_2]$ with $R_1 = R_2 = CH_3$ (Einstein & Field, 1974), $R_1 = R_2 = C_4H_9$ (Boyd, Mitra, Raston, Rowbottom & White, 1981), $R_1R_2 = (CH_2)_4$ (Newman, Raston & White, 1973), $R_1 = CH_3$, $R_2 = C_6H_5$ (Martin, Newman, Robinson & White, 1972), $R_1 = R_2 = C_2H_4OH$ (Radha, Seshasayee, Radha, Aravamudan & Subrahmanyam, 1985) have been determined. The dtc ligand in most of its complexes with transition-metal ions adopts a delocalized form.

The present work consists of the crystal structure analysis of two dithiocarbamate complexes, one of Ni^{II} and one of Cu^{II}. The substituent on the N atom is a piperidine ring in both cases.

Experimental. Good single crystals of both complexes were obtained by the following method: 4 mmol of sodium piperidine dithiocarbamate dissolved in 50 ml of water was added to 50 ml of an aqueous solution containing 2 mmol of nickel nitrate or copper nitrate. The Ni and Cu complexes precipitated as green and black powders, respectively. The precipitates were washed with water and dried. Single crystals were obtained on slow evaporation at 303 K using dichloromethane as solvent. The density was measured by the method of flotation.

Crystals $0{\cdot}625 \times 0{\cdot}150 \times 0{\cdot}075$ mm for Ni complex (*A*) and $0{\cdot}645 \times 0{\cdot}150 \times 0{\cdot}075$ mm for Cu complex (*B*) were mounted on a CAD-4 diffractometer. Unit-cell dimensions obtained from 25 high-angle reflections in the range $40° < \theta < 64°$ for *A* and $25° < \theta < 55°$ for *B*. Graphite-monochromated Cu $K\alpha$ radiation was employed to collect data in ω–2θ scan mode. 1818 and 1842 reflections were collected in the interval $2° < \theta < 70°$ for *A* and *B* (*h*: 0 to 7, *k*: 0 to 10, *l*: −18 to 18 for *A* and *B*). Two standard reflections (013, 020 for *A* and $\bar{2}1\bar{4}$, $\bar{2}4\bar{6}$ for *B*) showed intensity variations less than 5%. The scan width and aperture width for complexes *A* and *B* were $(0{\cdot}75 + 0{\cdot}45\tan\theta)$, $(2{\cdot}75 + 1{\cdot}25\tan\theta)$ mm and $(1{\cdot}00 + 0{\cdot}16\tan\theta)$, $(1{\cdot}00 + 0{\cdot}5\tan\theta)°$, respectively. 1201 unique reflections with $I > 3\sigma(I)$ for *A* and 1207 unique reflections with $I > 3\sigma(I)$ for *B* were used in structure solution. $R_{\rm int}$ for *A* and *B* are respectively 0·03 and 0·09. Patterson and difference Fourier gaps gave the structure. Program *SHELX*76 (Sheldrick, 1976) was used. Final refinement with 124 parameters gave $R = 0{\cdot}065$ for *A* and 0·078 for *B* and $wR = 0{\cdot}062$ and 0·073 for *A* and *B* respectively; $w = 1/[\sigma^2(F_o) + 0{\cdot}0124|F_o|^2]$ for *A* and $w = 1/[\sigma^2(F_o) + 0{\cdot}0197|F_o|^2]$ for *B*. Four of the ten H atoms for *A* and nine H atoms for *B* were obtained from difference Fourier maps and the remainder were fixed geometrically. The final difference Fourier maps were featureless for both *A* and *B*. Maximum and average shift/e.s.d. in atomic parameters of non-hydrogen atoms are 0·47, 0·29 and 0·52, 0·38, respectively, for *A* and *B*. Atomic scattering factors for non-hydrogen atoms from Cromer & Mann (1968) and anomalous-dispersion corrections for all non-hydrogen atoms from Cromer & Liberman (1970). H scattering factors from Stewart, Davidson & Simpson (1965). Absorption correction was applied to the intensity data on the basis of the crystal dimensions using the *ABSC* subroutine of the *SHELX*76 program. The maximum and minimum transmission factors are 0·6398, 0·1047 for *A* and 0·6474, 0·1551 for *B*.

Discussion. The fractional coordinates of all atoms for *A* and *B* with their equivalent isotropic thermal parameters are given in Table 1. *ORTEP* plots of molecules *A* and *B* are shown in Figs. 1 and 2. The relevant bond lengths and bond angles are given in Table 2.*

Molecules *A* and *B* are monomeric and isostructural. This is the second instance of isostructurality in Ni^{II} and Cu^{II} dithiocarbamates, the first being Ni/Cu-$(S_2CNMePh)_2$ (Martin, Newman, Robinson & White, 1972). In both *A* and *B* the central metal atom lies at an inversion centre and exhibits a pseudo square-planar configuration.

The metal atom is bonded to four S atoms from the two piperidine dithiocarbamate ligands. The planarity of the molecules is not limited to MS_4 (*M*: Ni or Cu) but includes the C—N bond as well. The maximum

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44913 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (× 10^4 Å^2 *for* Ni, Cu, *and* S *atoms and* × 10^3 Å^2 *for others*) *with estimated standard deviations in parentheses*

$$U_{eq} = \tfrac{1}{3}\sum_{i=1}^{3} U_{ii}.$$

	x	y	z	U_{eq}
Ni	1·0	0·0	1·0	396 (6)
*Cu	1·0	0·0	1·0	338 (10)
S(1)	0·6838 (1)	−0·0154 (1)	0·9164 (1)	470 (6)
*S(1)	0·6744 (3)	−0·0168 (2)	0·9128 (1)	496 (11)
S(2)	1·0913 (1)	−0·1534 (1)	0·8934 (1)	462 (6)
*S(2)	1·0863 (3)	−0·1602 (2)	0·8886 (1)	490 (11)
C(1)	0·8279 (6)	−0·1229 (4)	0·8460 (2)	40 (2)
*C(1)	0·822 (10)	−0·125 (8)	0·844 (4)	41 (3)
N(1)	0·7494 (5)	−0·1701 (4)	0·7655 (2)	45 (2)
*N(1)	0·744 (9)	−0·173 (6)	0·765 (4)	45 (3)
C(2)	0·8897 (7)	−0·2456 (5)	0·7041 (3)	55 (2)
*C(2)	0·881 (13)	−0·251 (10)	0·704 (4)	55 (5)
C(3)	0·9197 (7)	−0·1335 (6)	0·6290 (3)	56 (2)
*C(3)	0·913 (12)	−0·138 (10)	0·628 (5)	59 (4)
C(4)	0·7018 (8)	−0·0798 (6)	0·5828 (3)	59 (3)
*C(4)	0·697 (13)	−0·084 (11)	0·583 (4)	61 (5)
C(5)	0·5557 (7)	−0·0106 (5)	0·6493 (3)	53 (2)
*C(5)	0·555 (13)	−0·014 (8)	0·649 (6)	59 (4)
C(6)	0·5314 (6)	−0·1272 (5)	0·7241 (3)	51 (2)
*C(6)	0·528 (11)	−0·128 (10)	0·724 (4)	52 (4)

* Molecule *B*.

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

	Molecule *A* (Ni)	Molecule *B* (Cu)
M—S(1)	2·207 (1)	2·304 (2)
M—S(2)	2·196 (1)	2·288 (2)
S(1)—C(1)	1·718 (3)	1·724 (7)
S(2)—C(1)	1·717 (4)	1·728 (7)
C(1)—N(1)	1·327 (5)	1·316 (8)
N(1)—C(2)	1·478 (4)	1·466 (9)
C(2)—C(3)	1·512 (6)	1·521 (11)
C(3)—C(4)	1·513 (6)	1·499 (11)
C(4)—C(5)	1·533 (6)	1·538 (10)
C(5)—C(6)	1·528 (6)	1·550 (11)
C(6)—N(1)	1·462 (5)	1·472 (9)
S(1)—*M*—S(2)	79·4 (0)	77·6 (1)
M—S(1)—C(1)	85·0 (1)	84·4 (2)
M—S(2)—C(1)	85·3 (1)	84·8 (2)
S(1)—C(1)—S(2)	110·0 (2)	112·8 (4)
S(1)—C(1)—N(1)	124·9 (3)	123·6 (5)
S(2)—C(1)—N(1)	125·1 (3)	123·5 (5)
C(1)—N(1)—C(2)	122·2 (3)	123·1 (6)
C(1)—N(1)—C(6)	123·2 (3)	123·2 (6)
C(2)—N(1)—C(6)	113·7 (3)	112·9 (5)
N(1)—C(2)—C(3)	108·8 (3)	110·0 (7)
C(2)—C(3)—C(4)	111·8 (4)	111·0 (6)
C(3)—C(4)—C(5)	111·0 (3)	111·2 (6)
C(4)—C(5)—C(6)	110·1 (3)	110·0 (6)
C(5)—C(6)—N(1)	109·1 (3)	107·7 (6)

deviation from planarity is 0·060 (2) Å for *A* and 0·07 (1) Å for *B*. The ligand acts as an equal bidentate ligand with both *M*—S bond lengths nearly equal. The intraligand S(1)···S(2) distances of 2·813 (6) Å for *A* and 2·876 (1) Å for *B* are less than the interligand S(1)···S′(2) distances of 3·387 (1) Å for *A* and 3·580 (1) Å for *B*. The bite and non-bite S—*M*—S angles are 79·46 (2), 100·62 (2)° for *A* and 77·6 (2), 102·6 (2)° for *B*. The deviation from an exact square-planar coordination with dsp^2 hybridization of the valence orbitals of the metal atom is due to the small bite of the dithiocarbamate ligand.

The S—C bond lengths of 1·717 (4) Å for *A* and 1·727 (2) Å for *B* are shorter than the single-bond length of 1·81 Å and slightly longer than the double-bond length of 1·69 Å. This indicates partial S—C double-bond character. The C—N bond lengths of 1·327 (5) and 1·315 (4) Å have high double-bond character, a further confirmation of delocalization and partial double-bond character. The partial double-bond characters of the S—C and C—N bonds indicate that there are minor adjustments of these bonds and hence the delocalized form results. This is also supported by the planarity of the S_2CN moiety. The bond angles around N(1), C(1) indicate sp^2 hybridization.

The C—N bond lengths in the piperidine ring are in the range of single-bond lengths. The bond parameters around the C atoms in the piperidine ring are nearly in agreement with sp^3 hybridization.

The conformation of the piperidine ring can be best described as a chair for both molecules *A* and *B*. The torsion angles of the rings are alternately positive and negative with average magnitudes of 56·0 (4) and 57·3 (7)° for *A* and *B*, respectively (Table 3).

There are no short metal–metal interactions in *A* and *B*; short intermolecular contacts Ni···H(6) 2·930, S(2)···H(1) 2·864, S(2)···C(5) 3·831, S(2)···C(4) 3·859 and S(2)···C(2) 3·788 Å in molecule *A* and S(2)···C(2) 3·815, S(2)···C(4) 3·881, S(2)···C(5) 3·819 Å in *B* aid in crystal packing.

In Cu^{II} dithiocarbamate complexes, dimer formation occurs in the solid state and the structures of Ni and Cu analogues are not isomorphous, the only reported

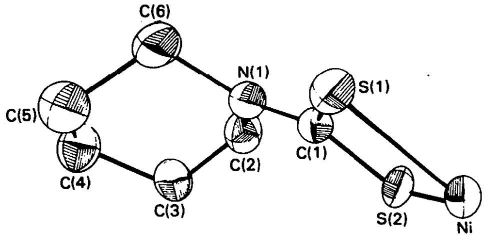

Fig. 1. An *ORTEP* plot of molecule *A*.

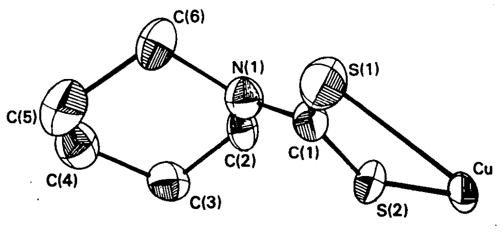

Fig. 2. An *ORTEP* plot of molecule *B*.

Table 3. *Torsion angles (°) involving the piperidine ring*

	Molecule *A*	Molecule *B*
N(1)–C(2)–C(3)–C(4)	−55·2 (5)	−55·5 (8)
C(2)–C(3)–C(4)–C(5)	54·1 (5)	53·7 (8)
C(3)–C(4)–C(5)–C(6)	−53·9 (5)	−54·9 (8)
C(4)–C(5)–C(6)–N(1)	56·0 (4)	57·1 (7)
C(5)–C(6)–N(1)–C(2)	−60·8 (4)	−61·6 (7)
C(6)–N(1)–C(2)–C(3)	56·0 (4)	61·1 (7)

exception (other than the present work) being that of $Cu(S_2CNMePh)_2$, where the dimer formation is prevented by the steric hindrance of the phenyl rings.

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Acta Cryst. (1988). C**44**, 1381–1383

[Di(azathien)-1-yl-S^1,N^4]bis(triethylphosphine)platinum(II) Hexafluorophosphate

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(*Received* 5 *November* 1987; *accepted* 22 *April* 1988)

Abstract. $[Pt(HN_2S_2)(C_6H_{15}P)_2][PF_6]$, M_r = 669·51, monoclinic, $P2_1/n$, a = 8·716 (3), b = 20·335 (10), c = 13·590 (5) Å, β = 99·47 (3)°, V = 2376 Å^3, Z = 4, D_x = 1·88 Mg m^{-3}, Cu $K\alpha$ radiation, λ = 1·54178 Å, μ = 15·22 mm^{-1}, $F(000)$ = 1304, room temperature, R = 0·042 for 2176 observed reflections with $|F_o| > 3\sigma(|F_o|)$. The structure of $[Pt(PEt_3)(S_2N_2H)][PF_6]$ consists of channels of cations and anions. The cation has normal square-planar geometry with Pt—P distances of 2·320 (3) and 2·270 (3), Pt–S 2·282 (3), Pt–N 2·022 (9) Å. The S—Pt—N and P—Pt—P angles are 83·5 (2) and 97·5 (1)°, respectively. The PtS_2N_2 ring contains two short [1·570 (9), 1·536 (10) Å] and one long [1·659 (10) Å] S—N bonds. The interplanar separations between adjacent cations are 4·06 (2) and 4·22 (2) Å.

Introduction. Several metal—sulfur—nitrogen compounds containing phosphine ligands have been reported (Kelly & Woollins, 1986; Jones, Kelly, Williams & Woollins, 1988). One particularly interesting feature of complexes of the type $[Pt(PR_3)_2(S_2N_2H)]X$ ($X = PF_6$, BF_4, Cl) is that in the solid state the cations and anions pack into channels. The dependence of the interplanar separation between adjacent cations in $[Pt(PMe_2Ph)_2(S_2N_2H)]X$, (2), on the nature of the anion has been investigated (Jones *et al.*, 1988) but the effect of the nature of the phosphine is poorly understood. Here we report on the structure of $[Pt(PEt_3)_2(S_2N_2H)][PF_6]$, (1), in comparison with (2).

Experimental. Single crystals of (1), suitable for X-ray crystallography, were grown at room temperature from dichloromethane/hexane and had m.p. 549 K. Crystal size 0·10 × 0·12 × 0·22 mm. Refined unit-cell parameters obtained from setting angles of 12 reflections with 8·7 ≤ θ ≤ 23·9°. Nicolet $R3m$ diffractometer. 2441 independent reflections (θ ≤ 50°) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 2176 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −8/8, k 0/20, l 0/13; two check reflections measured every 50 reflections, net count constant; Lorentz and polarization corrections, analytical absorption correction (face indexed crystal), max. and min. transmission factors 0·37 and 0·16. Structure solved by heavy-atom method; non-hydrogen atoms refined anisotropically; PF_6 anion refined as a rigid body; N(1) hydrogen atom refined isotropically; positions of

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