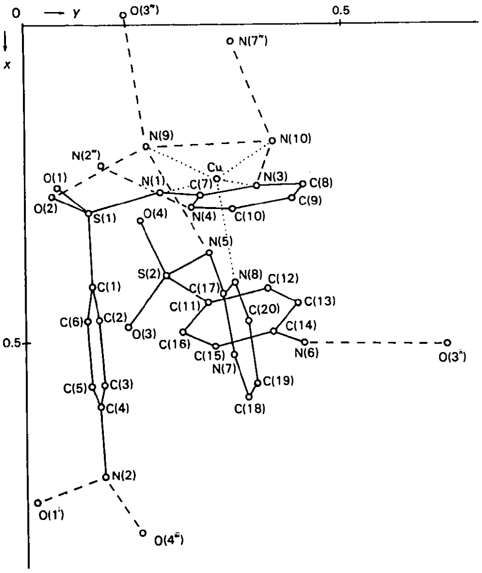

Fig. 1. Arrangement of molecule in (001) projection of part of unit-cell contents, showing atom numbering, copper coordination (dotted lines) and H-bonding system (dashed lines). Symmetry code: (i) $\frac{1}{2}+x, y, \frac{1}{2}-z$; (ii) $1-x, \frac{1}{2}+y, 1-z$; (iii) $\frac{1}{2}+x, y, 1\frac{1}{2}-z$; (iv) $x-\frac{1}{2}, y, 1\frac{1}{2}-z$; (v) $1-x, \frac{1}{2}+y, -z$.

Tables for X-ray Crystallography (1974); $R = 0.045$, $wR = 0.080$, $w^{1/2} = 1/F_o$.

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1,* and selected bond lengths and interbond angles are listed in Table 2.

The Cu atom is 5-coordinated in a distorted square pyramid, with N(1), N(3), N(9) and N(10) at distances 2·071 (13), 2·479 (18), 2·083 (11) and 1·894 (10) Å, respectively, in an irregular square, with a mean N—Cu—N angle, subtended by adjacent N atoms, of 87·6 (4)°. Atom N(8) at 2·377 (10) Å is at the apex with a mean N(8)—Cu—N angle of 100·0 (4)°. This 5-coordination in the Cu complex compares with the nearly regular tetrahedral coordination in the Zn complex (Brown, Cook & Sengier, 1985) where only N(1), N(8), N(9) and N(10) are used, N(3) being outside the sphere of coordination.

The intramolecular bond lengths and angles all agree well with the values found in this class of compound as well as in sulfamethazine (Basak, Mazumdar & Chaudhuri, 1983). The four organic molecular rings are each effectively planar with r.m.s. deviations all < 0·006 Å. The dihedral angles between the phenyl and diazine rings of each molecule are +94·17 (5) and +91·76 (5)°. There are eight probable hydrogen bonds of lengths between 2·837 (16) and 3·251 (21) Å, but as the electron density on difference maps around the N atoms was somewhat diffuse there is no certainty about these. The likely H-bond arrangement (Fig. 1) shows some differences from that in the Zn complex, and this may give rise to the differences in the unit-cell dimensions.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, phenyl-ring bonds and angles, mean planes and H bonds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44303 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Dichloro(4,7-diphenyl-1,10-phenanthroline)palladium(II)

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Abstract. $[PdCl_2(C_{24}H_{16}N_2)]$, $M_r = 509·4$, monoclinic, $P2_1/n$, $a = 15·354$ (2), $b = 17·551$ (3), $c = 15·665$ (1) Å, $\beta = 105·33$ (1)°, $V = 4071$ Å^3, $Z = 8$, $D_x = 1·66$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 11·73$ cm^{-1}, $F(000) = 2032$, $T = 293$ K, final $R = 0·035$ for 6324 observed reflections [$I > 2\sigma(I)$]. In both independent molecules a planar, bidentate 1,10-phenanthroline ligand is chelated to a $PdCl_2$ entity. The

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coordination geometry of the Pd atom is distorted square planar, whereby two Cl atoms and the N atoms of the bidentate ligand occupy the four coordination sites.

Introduction. The sample under investigation was kindly supplied by Professor Dr Chen Ying-Wu and Mr Pu Bao-Shan of Lanzhou Institute of Chemical Physics, Academia Sinica.

The coordination chemistry of *cis*-substituted palladium and platinum compounds has recently gained interest due to antitumor properties of certain aminehalide derivatives. A single-crystal X-ray diffraction study of the title compound was therefore undertaken not only to establish its chemical constitution but also to detect possible structural differences to similar compounds (see Table 3) – dichloro(2,9-dimethyl-1,10-phenanthroline)zinc(II) (V) (Preston & Kennard, 1969*a*), aquadichloro(2,9-dimethyl-1,10-phenanthroline)copper(II) (VII) (Preston & Kennard, 1969*c*), di-μ-chloro-*sym-trans*-dichlorobis(2,9-dimethyl-1,10-phenanthroline)dinickel(II)–chloroform (1/2) (I) (Preston & Kennard 1969*b*), di-μ-chloro-dichlorobis(2,9-dimethyl-1,10-phenanthroline)dinickel(II) (II) and di-μ-bromo-dibromobis(2,9-dimethyl-1,10-phenanthroline)dinickel(II) (II′) (Butcher & Sinn, 1977), (2,9-dimethyl-1,10-phenanthroline)diiodonickel(II) (III) (Butcher, O'Connor & Sinn, 1979), dichloro(1,10-phenanthroline)zinc (IV) (Reimann, Block & Perloff, 1966), trichloro(dimethylformamide)(1,10-phenanthroline)chromium(III) (VI) (Broomhead, Evans, Grumley & Sterns, 1977), *cis*-(2,3-butanediamine)-dichloropalladium(II) (VIII) (Ito, Marumo & Saito, 1971), (2,9-dimethyl-1,10-phenanthroline)dinitropalladium(II) (IX) (Fridrichsons, Mathieson & Power, 1971), and *cis*-dichloro(ethylenediamine)platinum(II) and -palladium(II) (X) (Iball, MacDougall & Scrimgeour, 1975).

Whereas complexes of 1,10-phenanthroline with various metals show a close-packed arrangement of ligands around the metal, the coordination of low-spin (d^8) $\mathrm{Pd^{II}}$ complexes with few exceptions is square planar (Maitlis, Espinet & Russell, 1982). In addition, few of the $\mathrm{Pd^{II}}$ halogen compounds are ionic in the solid state (Wiesner & Lingafelter, 1966). In all cases, the 1,10-phenanthroline moiety is essentially planar, as in the uncomplexed form (Nishigaki, Yoshioka & Nakatsu, 1978), and the metal atom is displaced slightly from the ligand coordination plane.

Experimental. Intensity data of an orange crystal (0·24 × 0·17 × 0·38 mm) of dichloro(4,7-diphenyl-1,10-phenanthroline)palladium(II), mounted in a glass capillary, were measured on an Enraf–Nonius CAD-4 diffractometer with graphite monochromator and were corrected for Lorentz, polarization and absorption effects [transmission 0·71–0·83 (Coppens, Leiserowitz & Rabinovich, 1965)]. Accurate cell dimensions were derived from a least-squares fit of 2θ setting angles for 75 automatically centered reflections ($9\cdot2 < \theta < 22\cdot5°$). The compound crystallizes in the monoclinic space group $P2_1/n$, with systematic absences $h0l$ for $h + l = 2n + 1$ and $0k0$ for $k = 2n + 1$. One set of intensities (h $-19\rightarrow19$, k $0\rightarrow22$, l $0\rightarrow20$) was collected by θ–2θ-scan techniques to give 9515 reflections with θ values between 1·00 and 27·44°, maximum value of $(\sin\theta)/\lambda = 0\cdot65$ Å^{-1}. Within this set, 9150 unique reflections were found ($R_{\rm int} = 0\cdot013$). 6324 reflections were considered observed by the criterion $I > 2\sigma(I)$. The standard reflections (12,0,0, 0,10,6, 008) showed no significant variation during the data collection.

The structure was solved by the heavy-atom method, which yielded the positions of both independent Pd atoms. The positions of four Cl atoms were obtained from an additional Fourier map and the remaining non-H atoms were located in a subsequent map. A difference Fourier map revealed the locations of all H atoms, which were included in the final cycles of the refinement, where the function minimized was $\sum w(F_o - F_c)^2$ with $w = 1/\sigma^2(F_o)$ (651 refined parameters, H atoms isotropic), $R = 0\cdot035$ and $wR = 0\cdot041$. Error of fit = 2·08, final average shift/e.s.d. of the coordinates 0·10, max. shift/e.s.d. 0·73, maximum residual electron density 0·78 e Å^{-3}, minimum $-0\cdot5$ e Å^{-3}. A correction for the effects of anomalous dispersion for Pd and Cl was included in the structure-factor calculations. Relativistic Hartree–Fock scattering factors (Cromer & Waber, 1965) for Pd, Cl, N, C and the best spherical scattering factors for the H atoms (Stewart, Davidson & Simpson, 1965) were used throughout the refinement. Atomic scattering curves were taken from *International Tables for X-ray Crystallography* (1974). Computer programs used in this investigation include modified versions of the *DATAP* data-reduction program (Coppens *et al.*, 1965), *SHELX*76 (Sheldrick, 1976) for crystal-structure solution and refinement, a modified version of *ORFLS* least-squares program for full-matrix refinement (Busing, Martin & Levy, 1962), *DAESD* program for distances and angles (Davis & Harris, 1970), *XANADU* molecular geometry program (Roberts & Sheldrick, 1974) and *ORTEP* thermal-ellipsoid plotting program (Johnson, 1976).

Discussion. Final atomic parameters with equivalent temperature factors are given in Table 1, averaged bond distances and angles over the two independent molecules in Table 2.* A perspective drawing of one of the

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* Lists of structure factors, anisotropic thermal parameters, and a table of bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44289 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic thermal parameters* (Å²)

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

	x	y	z	U_{eq}/U_{iso}
Pd(1)	0·1625 (1)	0·0331 (1)	0·0546 (1)	0·037
Cl(1)	0·2210 (1)	0·0005 (1)	0·1994 (1)	0·056
Cl(2)	0·2349 (1)	0·1478 (1)	0·0783 (1)	0·057
N(1)	0·1068 (2)	0·0526 (2)	−0·0763 (2)	0·039
N(2)	0·1002 (2)	−0·0691 (2)	0·0208 (2)	0·035
C(1)	0·1096 (3)	0·1151 (3)	−0·1232 (3)	0·050
C(2)	0·0675 (3)	0·1196 (3)	−0·2125 (3)	0·055
C(3)	0·0219 (3)	0·0590 (2)	−0·2597 (3)	0·043
C(4)	0·0227 (2)	−0·0106 (2)	−0·2114 (2)	0·038
C(5)	−0·0129 (3)	−0·0816 (2)	−0·2506 (3)	0·041
C(6)	−0·0109 (3)	−0·1452 (2)	−0·2024 (3)	0·041
C(7)	0·0247 (2)	−0·1451 (2)	−0·1082 (2)	0·036
C(8)	0·0278 (2)	−0·2092 (2)	−0·0529 (3)	0·039
C(9)	0·0644 (3)	−0·1992 (3)	0·0370 (3)	0·048
C(10)	0·0993 (3)	−0·1290 (2)	0·0720 (3)	0·045
C(11)	0·0618 (2)	−0·0764 (2)	−0·0682 (2)	0·035
C(12)	0·0628 (2)	−0·0095 (2)	−0·1202 (2)	0·035
C(13)	−0·0249 (3)	0·0670 (2)	−0·3550 (3)	0·043
C(14)	−0·1174 (3)	0·0494 (3)	−0·3872 (3)	0·059
C(15)	−0·1604 (4)	0·0605 (3)	−0·4745 (4)	0·063
C(16)	−0·1148 (4)	0·0875 (3)	−0·5327 (3)	0·063
C(17)	−0·0243 (4)	0·1041 (3)	−0·5023 (3)	0·057
C(18)	0·0193 (3)	0·0943 (2)	−0·4145 (3)	0·049
C(19)	−0·0072 (3)	−0·2859 (2)	−0·0881 (2)	0·040
C(20)	−0·0985 (3)	−0·2983 (2)	−0·1233 (3)	0·046
C(21)	−0·1303 (3)	−0·3703 (3)	−0·1496 (3)	0·056
C(22)	−0·0718 (4)	−0·4301 (3)	−0·1436 (3)	0·061
C(23)	0·0195 (4)	−0·4178 (3)	−0·1098 (4)	0·067
C(24)	0·0513 (3)	−0·3467 (3)	−0·0828 (3)	0·056
H(1)	0·144 (3)	0·155 (2)	−0·085 (3)	0·055
H(2)	0·070 (3)	0·165 (2)	−0·241 (3)	0·051
H(5)	−0·035 (3)	−0·087 (2)	−0·311 (3)	0·044
H(6)	−0·032 (3)	−0·187 (2)	−0·227 (3)	0·040
H(9)	0·069 (3)	−0·234 (2)	0·069 (3)	0·046
H(10)	0·117 (3)	−0·117 (2)	0·138 (3)	0·048
H(14)	−0·149 (3)	0·037 (2)	−0·348 (3)	0·057
H(15)	−0·220 (3)	0·057 (2)	−0·493 (3)	0·053
H(16)	−0·138 (3)	0·097 (2)	−0·590 (3)	0·062
H(17)	0·009 (3)	0·121 (3)	−0·544 (3)	0·066
H(18)	0·073 (3)	0·106 (2)	−0·399 (3)	0·048
H(20)	−0·138 (3)	−0·260 (2)	−0·130 (3)	0·046
H(21)	−0·191 (3)	−0·381 (2)	−0·170 (3)	0·061
H(22)	−0·094 (3)	−0·480 (2)	−0·159 (3)	0·053
H(23)	0·064 (3)	−0·452 (2)	−0·101 (3)	0·051
H(24)	0·106 (3)	−0·342 (2)	−0·064 (3)	0·054
Pd(1*A*)	0·8280 (1)	0·4012 (1)	0·3262 (1)	0·046
Cl(1*A*)	0·7588 (1)	0·4444 (1)	0·1873 (1)	0·073
Cl(2*A*)	0·8531 (1)	0·2839 (1)	0·2721 (1)	0·079
N(1*A*)	0·8952 (2)	0·3757 (2)	0·4537 (2)	0·043
N(2*A*)	0·8125 (2)	0·5017 (2)	0·3849 (2)	0·043
C(1*A*)	0·9326 (3)	0·3104 (3)	0·4864 (3)	0·055
C(2*A*)	0·9797 (3)	0·3013 (3)	0·5744 (3)	0·052
C(3*A*)	0·9902 (3)	0·3618 (2)	0·6329 (3)	0·043
C(4*A*)	0·9537 (2)	0·4336 (2)	0·5981 (2)	·0·037
C(5*A*)	0·9645 (3)	0·5035 (2)	0·6473 (3)	0·038
C(6*A*)	0·9227 (3)	0·5683 (2)	0·6127 (3)	0·038
C(7*A*)	0·8640 (2)	0·5708 (2)	0·5243 (2)	0·037
C(8*A*)	0·8109 (3)	0·6358 (2)	0·4850 (3)	0·041
C(9*A*)	0·7632 (3)	0·6283 (3)	0·3981 (3)	0·047
C(10*A*)	0·7650 (3)	0·5633 (3)	0·3487 (3)	0·046
C(11*A*)	0·8596 (2)	0·5054 (2)	0·4723 (2)	0·037
C(12*A*)	0·9047 (3)	0·4368 (2)	0·5084 (3)	0·040
C(13*A*)	1·0363 (3)	0·3515 (2)	0·7285 (3)	0·045
C(14*A*)	0·9966 (4)	0·3757 (3)	0·7930 (3)	0·061
C(15*A*)	1·0407 (5)	0·3669 (3)	0·8813 (3)	0·076
C(16*A*)	1·1244 (5)	0·3339 (3)	0·9066 (3)	0·075
C(17*A*)	1·1646 (4)	0·3088 (3)	0·8435 (3)	0·064
C(18*A*)	1·1205 (3)	0·3171 (2)	0·7552 (3)	0·049
C(19*A*)	0·8051 (2)	0·7072 (2)	0·5343 (3)	0·041
C(20*A*)	0·7942 (3)	0·7068 (3)	0·6197 (3)	0·049
C(21*A*)	0·7813 (3)	0·7737 (3)	0·6608 (3)	0·060
C(22*A*)	0·7803 (3)	0·8430 (3)	0·6166 (4)	0·063
C(23*A*)	0·7932 (3)	0·8448 (3)	0·5338 (4)	0·059
C(24*A*)	0·8042 (3)	0·7775 (3)	0·4922 (3)	0·049
H(1*A*)	0·924 (3)	0·270 (3)	0·450 (3)	0·064
H(2*A*)	0·998 (2)	0·252 (2)	0·598 (3)	0·040
H(5*A*)	1·003 (3)	0·504 (2)	0·702 (3)	0·043
H(6*A*)	0·935 (3)	0·610 (2)	0·640 (3)	0·039
H(9*A*)	0·743 (3)	0·661 (2)	0·375 (3)	0·045
H(10*A*)	0·733 (3)	0·557 (2)	0·282 (3)	0·053
H(14*A*)	0·938 (3)	0·394 (2)	0·778 (3)	0·049
H(15*A*)	1·017 (3)	0·376 (3)	0·914 (3)	0·049
H(16*A*)	1·155 (3)	0·329 (2)	0·971 (3)	0·060
H(17*A*)	1·218 (3)	0·282 (3)	0·865 (3)	0·069
H(18*A*)	1·153 (3)	0·293 (2)	0·706 (3)	0·053
H(20*A*)	0·795 (3)	0·661 (2)	0·646 (3)	0·047
H(21*A*)	0·772 (3)	0·768 (2)	0·725 (3)	0·057
H(22*A*)	0·772 (3)	0·882 (3)	0·646 (3)	0·062
H(23*A*)	0·790 (3)	0·895 (2)	0·500 (3)	0·057
H(24*A*)	0·811 (2)	0·783 (2)	0·433 (3)	0·043

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

Pd—Cl(1)	2·283 (1)	C(4)—C(5)	1·434 (6)
Pd—Cl(2)	2·29 (1)	C(4)—C(12)	1·404 (8)
Pd—N(1)	2·036 (6)	C(5)—C(6)	1·346 (6)
Pd—N(2)	2·035 (3)	C(6)—C(7)	1·437 (6)
N(1)—C(1)	1·326 (6)	C(7)—C(8)	1·43 (2)
N(1)—C(12)	1·363 (9)	C(7)—C(11)	1·404 (6)
N(2)—C(10)	1·33 (1)	C(8)—C(9)	1·377 (9)
N(2)—C(11)	1·370 (5)	C(8)—C(19)	1·494 (8)
C(1)—C(2)	1·383 (7)	C(9)—C(10)	1·390 (8)
C(2)—C(3)	1·380 (6)	C(11)—C(12)	1·431 (6)
C(3)—C(4)	1·432 (6)	C(13)—C(18) Ph ring	1·38 (1)
C(3)—C(13)	1·486 (6)	C(19)—C(24) Ph ring	1·38 (1)

Cl(1)—Pd—Cl(2)	91·2 (6)	C(5)—C(4)—C(12)	117·5 (3)
Cl(1)—Pd—N(1)	174 (1)	C(4)—C(5)—C(6)	122·1 (4)
Cl(1)—Pd—N(2)	93·8 (4)	C(5)—C(6)—C(7)	121·5 (4)
Cl(2)—Pd—N(1)	94·2 (1)	C(6)—C(7)—C(8)	125·0 (4)
Cl(2)—Pd—N(2)	174·5 (6)	C(6)—C(7)—C(11)	117·3 (3)
N(1)—Pd—N(2)	80·8 (2)	C(8)—C(7)—C(11)	117·7 (3)
Pd—N(1)—C(1)	129·0 (4)	C(7)—C(8)—C(9)	116·9 (9)
Pd—N(1)—C(12)	113·0 (3)	C(7)—C(8)—C(19)	123·0 (5)
C(1)—N(1)—C(12)	118·0 (6)	C(9)—C(8)—C(19)	120·2 (4)
Pd—N(2)—C(10)	128·5 (3)	C(8)—C(9)—C(10)	122 (1)
Pd—N(2)—C(11)	113·4 (2)	N(2)—C(10)—C(9)	121·4 (6)
C(10)—N(2)—C(11)	118·1 (4)	N(2)—C(11)—C(7)	123·4 (4)
N(1)—C(1)—C(2)	122·6 (6)	N(2)—C(11)—C(12)	115·7 (4)
C(1)—C(2)—C(3)	122 (1)	C(7)—C(11)—C(12)	121·0 (4)
C(2)—C(3)—C(4)	116·8 (8)	N(1)—C(12)—C(4)	123·1 (5)
C(2)—C(3)—C(13)	120·8 (4)	N(1)—C(12)—C(11)	116·8 (5)
C(4)—C(3)—C(13)	122 (1)	C(4)—C(12)—C(11)	120·2 (4)
C(3)—C(4)—C(5)	124·6 (4)	C(13)—C(18) Ph ring	120 (1)
C(3)—C(4)—C(12)	117·9 (3)	C(19)—C(24) Ph ring	120 (1)

two independent molecules and its thermal ellipsoids is shown in Fig. 1; Fig. 2 gives the graphic representation of the contents of the unit cell. The two independent [$PdCl_2(C_{24}H_{16}N_2)$] molecules in the asymmetric unit show no substantial differences in their geometrical data, except for the orientation of the two phenyl rings relative to the phenanthroline plane. The corresponding interplanar angles between the phenyl rings and the phenanthroline plane are 56 and 67° in molecule (1) and 49 and 41° in molecule (2), respectively. The coordination of the Pd atoms is approximately square planar. The angle between the plane defined by the unit Cl—Pd—Cl and the phenanthroline plane is 9·8° in molecule (1) and 11·0° in molecule (2). The Cl atoms [av. distance: Pd—Cl 2·287 (5) Å] as well as the N

Table 3. *A comparison of selected bond lengths* (Å) *and bond angles* (°) *in* $[\mathrm{PdCl_2(C_{24}H_{16}N_2)}]$ *and similar compounds*

Compound	Bond lengths				Bond angles			
	M–N(1)	*M*–N(2)	*M*–*X*(1)*	*M*–*X*(2)	N(1)–*M*–N(2)	*X*(1)–*M*–*X*(2)	N–*M*–*X*(1)	N–*M*–*X*(2)
$[\mathrm{PdCl_2(C_{24}H_{16}N_2)}]$†	2·031 (3)	2·037 (3)	2·282 (1)	2·283 (1)	80·9 (1)	90·7 (1)	94·1 (1)	94·2 (1)
	2·040 (3)	2·033 (3)	2·284 (1)	2·297 (1)	80·6 (1)	91·6 (1)	93·5 (1)	94·2 (1)
$[\mathrm{Ni_2Cl_4(C_{14}H_{12}N_2)_2}].\mathrm{2CHCl_3}$ (I)	2·069 (10)	2·060 (10)	2·394 (3)	2·316 (3)	81·7 (4)	162·2 (1)	91·8 (3)	100·3 (3)
$[\mathrm{Ni_2Cl_4(C_{14}H_{12}N_2)_2}]$ (II)	2·033 (2)	2·049 (2)	2·378 (1)	2·307 (1)	82·00 (7)	91·51 (2)	121·65 (5)	91·51 (5)
$[\mathrm{Ni_2Br_4(C_{14}H_{12}N_2)_2}]$ (II′)	2·021 (2)	2·034 (2)	2·468 (1)	2·458 (1)	82·44 (8)	94·28 (1)	125·61 (6)	92·81 (6)
$[\mathrm{NiI_2(C_{14}H_{12}N_2)}]$ (III)	1·987 (3)	2·005 (3)	2·546 (1)	2·530 (1)	83·8 (1)	126·75 (2)	103·94 (7)	112·40 (8)
$[\mathrm{ZnCl_2(C_{12}H_8N_2)}]$ (IV)	2·050 (7)	2·072 (7)	2·198 (3)	2·207 (3)	80·4 (3)	114·7 (1)	108·7 (3)	108·2 (3)
$[\mathrm{ZnCl_2(C_{14}H_{12}N_2)}]$ (V)	2·059 (11)	2·074 (9)	2·204 (3)	2·219 (3)	81·5 (3)	120·3 (1)	111·6 (2)	112·7 (2)
$[\mathrm{CrCl_3(C_{12}H_8N_2)(C_3H_7NO)}]$ (VI)	2·10 (2)	2·09 (2)	2·27 (1)	2·34 (1)	76·7 (7)	94·6 (2)	96·2 (6)	85·8 (6)
$[\mathrm{CuCl_2(C_{14}H_{12}N_2)(H_2O)}]$ (VII)	1·982 (9)	2·236 (11)	2·303 (4)	2·320 (4)	79·7 (4)	155·6 (2)	86·6 (4)	103·7 (3)
$[\mathrm{PdCl_2(C_4H_{12}N_2)}]$ (VIII)	2·028 (6)	2·030 (6)	2·318 (2)	2·316 (2)	83·6 (2)	95·3 (1)	90·6 (2)	90·6 (2)
$[\mathrm{Pd(NO_2)_2(C_{14}H_{12}N_2)}]$ (IX)	2·09	2·09	1·98	2·02	81	79	102	98
$[\mathrm{PdCl_2(C_2H_8N_2)}]$ (X)	1·978 (12)	—	2·309 (3)	—	83 (1)	95·3 (3)	90·8 (3)	—

* *M* = metal atom; *X* = –Cl, $-\mathrm{NO_2}$, –Br, –I.

† This work (two independent molecules); see *Introduction* for other compound references.

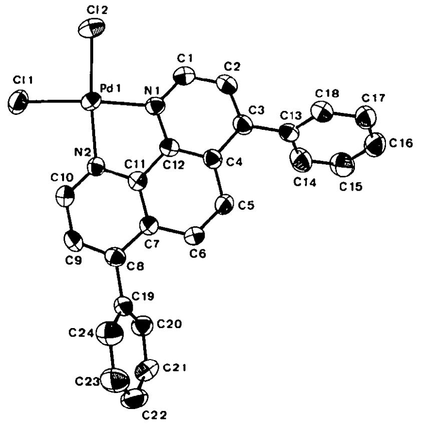

Fig. 1. Perspective drawing of $[\mathrm{PdCl_2(C_{24}H_{16}N_2)}]$ (molecule 1).

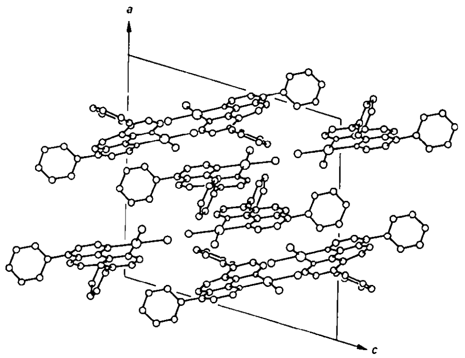

Fig. 2. Contents of the unit cell of $[\mathrm{PdCl_2(C_{24}H_{16}N_2)}]$ projected on the *ac* plane.

atoms [av. distance: Pd–N 2·036 (6) Å] are nearly equidistant from the Pd atoms.

The phenanthroline ligand itself (Nishigaki *et al.*, 1978), in addition to its coordination complexes with various metals including palladium (Fridrichsons *et al.*, 1971; Zagorodnikov, Khodashova, Vargaftik, Moiseev & Porai-Koshits, 1985; Rund & Hazell, 1980), has been investigated extensively. The geometry of the ligand and the Pd–N distances are in good agreement with known values. The title compound, however, shows the shortest Pd–Cl distances established so far. Selected bond lengths and bond angles of this and some related compounds are compared in Table 3.

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Structure of 2-Methyl-2-(2-pyridyl)propyl-*S*,*S*-dimethylsulfonium Hexafluorophosphate: an Intramolecular Non-Bonding S···N Interaction

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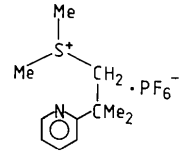
Abstract. $C_{11}H_{18}NS^+.PF_6^-$, $M_r = 341{\cdot}29$, orthorhombic, $Pc2_1n$, $a = 17{\cdot}725\,(1)$, $b = 10{\cdot}580\,(1)$, $c = 8{\cdot}314\,(1)$ Å, $V = 1559{\cdot}2\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}454$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha_1) = 1{\cdot}5401$ Å, $\mu = 3{\cdot}295$ mm^{-1}, $F(000) = 704$, $T = 296$ K, $R = 0{\cdot}051$ for 1248 observed reflexions. A weak intramolecular interaction is observed between S and N with a length of 2·834 Å. This is longer than that of the typical N—S—N sulfurane but shorter than the van der Waals contact. The torsion angles N—C(2)—C(Me$_2$)—C(H$_2$) and C(2)—C(Me$_2$)—C(H$_2$)—S are 4·4 (6) and −60·6 (5)°, respectively, so that the conformation about the exocyclic chain is favourable for such an interaction.

Introduction. A variety of organic σ-sulfuranes has been synthesized and the structures have been determined by X-ray analysis (Baenziger, Buckles, Maner & Simpson, 1969; Paul, Martin & Perozzi, 1972; Kálmán, Sasvári & Kapvits, 1973; Perozzi, Martin & Paul, 1974; Adzima, Chiang, Paul & Martin, 1978). Every compound bears electron-withdrawing apical groups due to the electron-rich and polarizable nature of the apical three-centre four-electron bond. A series of *S*-substituted *N*-methyl-6,7-dihydro-5*H*,12*H*$^+$-dibenzo[*b*,*g*][1,5]thiazocinium salts was synthesized and their structures were determined by X-ray analysis (Iwasaki & Akiba, 1985). From the features of these structures these compounds were concluded to be a new type of σ-sulfurane with a strong transannular S···N interaction. The compound with $X_{\mathrm{ap}} = \mathrm{Me}$ is the first example of a σ-alkylsulfurane with an apical alkyl group despite the electron-donating character of an alkyl group. The stability of such a structure may be attributed to ring formation. In order to investigate the S···N interaction for the noncyclic compound the structure determination of the title compound was carried out.



Experimental. Pale yellow prisms, 0·60 × 0·30 × 0·10 mm, unit-cell parameters by least squares from 25 reflexions ($65 < 2\theta < 75°$), Rigaku AFC4 diffractometer, graphite monochromated, $2 \leq 2\theta \leq 130°$, $h = 0\rightarrow20$, $k = 0\rightarrow12$, $l = 0\rightarrow9$, ω–2θ scan, scan range $\Delta\omega = 1{\cdot}2° + 0{\cdot}5°\tan\theta$, scan speed 4° min^{-1} in 2θ, three reflexions monitored (040, 402, $\bar{3}02$) every 50 reflexions, variations within 2%. 1531 independent reflexions measured, 1248 observed, $|F_o| \geq 3\sigma(F)$. Two heavy atoms S and P were obtained by *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Other non-H atoms were obtained from successive Fourier maps. Disordered PF_6^- ion with the occupancy factors estimated from the peak heights of a D map. H atoms were found from the D map. Block-diagonal least squares with anisotropic temperature factors for non-H and isotropic ones for H. $\sum w(|F_c| - k^{-1}|F_o|)^2$ was minimized. $w = 1/(0{\cdot}7194 + 0{\cdot}0297|F_o| + 0{\cdot}0006|F_o|^2)$. Three strong reflexions were omitted from the final refinement. $\Delta\rho(\mathrm{max.}) = 0{\cdot}32$, $\Delta/\sigma(\mathrm{max.}) = 0{\cdot}14$. $R = 0{\cdot}049$ (0·051 for 1248

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