Mo(1)—O(5*a*)	2.341 (4)	N(1*a*)—N(2*a*)	1.403 (6)
Mo(1)—N(2*a*)	2.239 (4)	N(1*a*)—C(2*a*)	1.267 (8)
Mo(2)—O(1*b*)	1.716 (4)	N(1*b*)—N(2*b*)	1.413 (6)
Mo(2)—O(2*b*)	1.680 (4)	N(1*b*)—C(2*b*)	1.275 (8)
Mo(2)—O(3*b*)	2.030 (4)	N(2*a*)—C(3*a*)	1.275 (7)
Mo(2)—O(4*b*)	1.932 (4)	N(2*b*)—C(3*b*)	1.281 (7)
Mo(2)—O(5*b*)	2.327 (4)	C(1)—C(2*a*)	1.510 (9)
Mo(2)—N(2*b*)	2.228 (5)	C(1)—C(2*b*)	1.507 (9)
O(1*a*)—Mo(1)—O(2*a*)	105.0 (3)	O(2*b*)—Mo(2)—O(4*b*)	100.7 (2)
O(1*a*)—Mo(1)—O(3*a*)	97.9 (2)	O(2*b*)—Mo(2)—O(5*b*)	169.7 (2)
O(1*a*)—Mo(1)—O(4*a*)	98.1 (2)	O(2*b*)—Mo(2)—N(2*b*)	94.0 (2)
O(1*a*)—Mo(1)—O(5*a*)	171.3 (2)	O(3*b*)—Mo(2)—O(4*b*)	150.2 (2)
O(1*a*)—Mo(1)—N(2*a*)	93.0 (2)	O(3*b*)—Mo(2)—O(5*b*)	79.8 (1)
O(2*a*)—Mo(1)—O(3*a*)	95.0 (2)	O(3*b*)—Mo(2)—N(2*b*)	71.4 (1)
O(2*a*)—Mo(1)—O(4*a*)	106.1 (2)	O(4*b*)—Mo(2)—O(5*b*)	80.1 (2)
O(2*a*)—Mo(1)—O(5*a*)	83.7 (2)	O(4*b*)—Mo(2)—N(2*b*)	82.5 (2)
O(2*a*)—Mo(1)—N(2*a*)	159.2 (2)	O(5*b*)—Mo(2)—N(2*b*)	75.8 (1)
O(3*a*)—Mo(1)—O(4*a*)	149.1 (2)	N(2*a*)—N(1*a*)—C(2*a*)	110.0 (4)
O(3*a*)—Mo(1)—O(5*a*)	80.8 (2)	N(2*b*)—N(1*b*)—C(2*b*)	108.4 (5)
O(3*a*)—Mo(1)—N(2*a*)	71.7 (2)	N(1*a*)—N(2*a*)—C(3*a*)	116.6 (5)
O(4*a*)—Mo(1)—O(5*a*)	79.5 (2)	N(1*b*)—N(2*b*)—C(3*b*)	114.7 (4)
O(4*a*)—Mo(1)—N(2*a*)	81.4 (2)	C(2*a*)—C(1)—C(2*b*)	108.0 (6)
O(5*a*)—Mo(1)—N(2*a*)	78.5 (2)	O(3*a*)—C(2*a*)—N(1*a*)	124.3 (5)
O(1*b*)—Mo(2)—O(2*b*)	104.3 (2)	O(3*a*)—C(2*a*)—C(1)	116.4 (5)
O(1*b*)—Mo(2)—O(3*b*)	93.7 (2)	N(1*a*)—C(2*a*)—C(1)	119.4 (5)
O(1*b*)—Mo(2)—O(4*b*)	106.2 (2)	O(3*b*)—C(2*b*)—N(1*b*)	124.7 (6)
O(1*b*)—Mo(2)—O(5*b*)	85.2 (2)	O(3*b*)—C(2*b*)—C(1)	116.0 (6)
O(1*b*)—Mo(2)—N(2*b*)	157.6 (2)	N(1*b*)—C(2*b*)—C(1)	119.3 (6)
O(2*b*)—Mo(2)—O(3*b*)	95.4 (2)		

Table 3. *Selected interatomic distances* (Å) *in* (I) *and* (II)

	(I)	(II)
Mo(1)· · ·Mo(2)	7.370 (2)	6.370 (3)
S(1*a*)· · ·S(1*b*)	5.097 (3)	9.636 (3)
C(3*a*)· · ·C(3*b*)	7.37 (1)	8.294 (5)
O(3*a*)· · ·O(3*b*)	4.306 (7)	3.699 (1)

H atoms were located in difference Fourier syntheses and were included in the structure-factor calculations but their positions were not refined. Data collection was performed using CAD-4 diffractometer software (Enraf–Nonius, 1977). The structure was solved by direct methods using *MULTAN*11/82 (Main *et al.*, 1982) and refined by a full-matrix least squares using *SDP* (Frenz, 1978).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving H atoms and torsion angles have been deposited with the IUCr (Reference: BR1062). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Distorted Planar Palladium Complex with Maleonitriledithiolato and Triphenylphosphine Ligands, $[Pd(PPh_3)_2(mnt)]$

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Abstract

cis-[2,3-Disulfido-2-butenedinitrile(2−)-*S*,*S*′]bis(triphenylphosphine)palladium(II), $[Pd(C_4N_2S_2)(C_{18}H_{15}P)_2]$, is a distorted planar palladium complex. The *trans* P—Pd—S angles in the complex are 170.3 (1) and 165.4 (1)°. The distortion may be caused by intermolecular contacts between a cyano N atom and phenyl C atoms in the crystal.

Comment

Maleonitriledithiolate (mnt^{2-}) forms square-planar $[M(mnt)_2]^{2-}$ complexes with Group 10 atoms. These complexes oxidize to the corresponding monoanions in which the presence of unpaired electrons has been investigated and determined using ESR, XPS, IR spectroscopy and MO calculations (McCleverty, 1968; Eisenberg, 1970; Burns & McAuliffe, 1979). The bis(maleonitriledithiolato)palladium complexes which have been reported are mainly in the monoanion form, some examples being $K[Pd(mnt)_2].H_2O$, $(NH_4)[Pd(mnt)_2].H_2O$ (Hursthouse, Short, Clemenson & Underhil, 1989) and $(perylene)_2[Pd(mnt)_2]$ (Domingos *et al.*, 1988). A complex containing a dianion, $MV[Pd(mnt)_2]$ (MV = *N*,*N*′-dimethyl-4,4′-bipyridinium), has been reported recently (Lemke, Knoch & Kisch, 1993). However, only a few complexes of Group 10 elements which contain one maleonitriledithiolato ligand and two monodentate ligands have been reported. The structure of $[Pt(PPhMe_2)_2(mnt)]$ is an example (Fitzmaurice, Slawin, Williams, Woollins & Lindsay, 1990).

The title complex, (I), was obtained as a by-product during our studies of mixed-metal complexes of the general formula $[M(mnt)_2\{Ag(PR_3)_2\}_2]$ (M = Ni, Pd, Pt; R = Ph, ^{n}Bu). The related complex $[Pt(PPhMe_2)_2(mnt)]$ is square planar with *trans* P—Pt—S angles of nearly 180°. However, $[Pd(PPh_3)_2(mnt)]$ is considerably distorted from the ideal planar structure with *trans* P—Pd—S angles of 170.3 (1) and 165.4 (1)°. Since the

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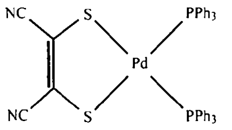
structure of [Pt(PPhMe$_2$)$_2$(mnt)] is not distorted and [Pd(PPh$_3$)$_2$(mnt)] has no appreciable intramolecular contacts, the distortion of [Pd(PPh$_3$)$_2$(mnt)] is thought to be caused by its crystal packing. Short intermolecular contacts in the structure are found between N2 and the phenyl C atoms on P1 at $\frac{1}{2}+x, -y, z-\frac{1}{2}$, with C6′···N2 3.26 (2), C12′···N2 3.28 (2) and C13′···N2 3.39 (2) Å. The deviation of N2 from the plane containing Pd1, S1, S2, C1 and C2 is 0.79 (1) Å, which is greater than that of N1 [0.48 (1) Å].



(I)

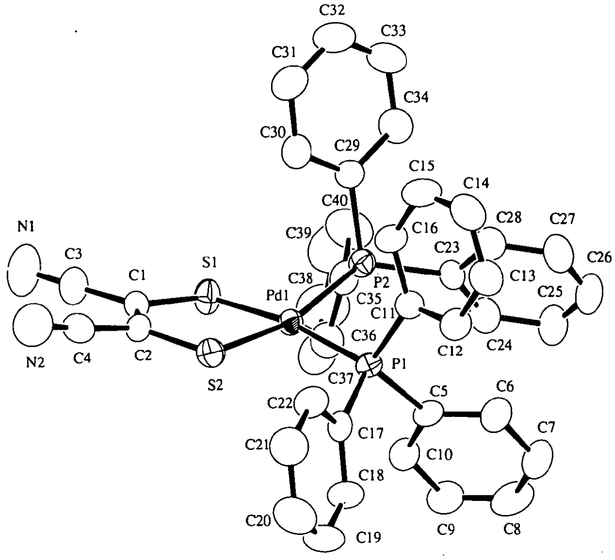


Fig. 1. The molecular structure of the title complex showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

The title complex was prepared from PdCl$_2$, Na$_2$mnt and PPh$_3$ in dichloromethane and recrystallized from the same solvent.

Crystal data

[Pd(C$_4$N$_2$S$_2$)(C$_{18}$H$_{15}$P)$_2$]
$M_r = 771.16$
Monoclinic
Pn
$a = 9.119$ (2) Å
$b = 11.039$ (2) Å
$c = 17.971$ (1) Å
$\beta = 101.07$ (1)°
$V = 1775.4$ (4) Å^3
$Z = 2$
$D_x = 1.442$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 12.5–15°$
$\mu = 0.762$ mm^{-1}
$T = 296$ K
Prismatic
0.3 × 0.2 × 0.2 mm
Red

Data collection

Rigaku AFC-5R diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5740 measured reflections
5430 independent reflections
4281 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.068$
$\theta_{\rm max} = 30.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 15$
$l = -25 \rightarrow 24$
3 standard reflections monitored every 150 reflections
intensity variation: 0.38%

Refinement

Refinement on F
$R = 0.046$
$wR = 0.057$
$S = 2.440$
4281 reflections
422 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + p^2(F_o)^2/4]$ where $p = 0.01$
$(\Delta/\sigma)_{\rm max} = 0.019$
$\Delta\rho_{\rm max} = 1.29$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.67$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm eq}$
Pd1	0.0000	0.19837 (6)	0.0000	0.0268 (1)
S1	0.1364 (3)	0.3627 (3)	−0.0305 (2)	0.0391 (8)
S2	0.0225 (3)	0.1104 (2)	−0.1133 (1)	0.0353 (8)
P1	−0.0966 (3)	0.0191 (2)	0.0345 (1)	0.0285 (6)
P2	−0.0666 (3)	0.3188 (2)	0.0948 (1)	0.0318 (8)
N1	0.266 (1)	0.494 (1)	−0.1964 (7)	0.082 (5)
N2	0.095 (2)	0.197 (1)	−0.3000 (7)	0.082 (6)
C1	0.150 (1)	0.3313 (9)	−0.1232 (5)	0.034 (3)
C2	0.100 (1)	0.2261 (10)	−0.1581 (6)	0.034 (3)
C3	0.216 (1)	0.422 (1)	−0.1639 (7)	0.048 (4)
C4	0.100 (1)	0.208 (1)	−0.2379 (7)	0.049 (4)
C5	−0.011 (1)	−0.0166 (9)	0.1321 (5)	0.032 (3)
C6	−0.090 (1)	−0.070 (1)	0.1825 (6)	0.045 (4)
C7	−0.012 (1)	−0.104 (1)	0.2538 (6)	0.055 (4)
C8	0.138 (1)	−0.083 (1)	0.2745 (6)	0.054 (4)
C9	0.218 (1)	−0.029 (1)	0.2264 (6)	0.045 (4)
C10	0.143 (1)	0.006 (1)	0.1560 (6)	0.042 (4)
C11	−0.300 (1)	0.0133 (9)	0.0189 (5)	0.033 (3)
C12	−0.374 (1)	−0.090 (1)	0.0336 (6)	0.041 (4)
C13	−0.527 (1)	−0.094 (1)	0.0212 (7)	0.052 (4)
C14	−0.606 (1)	0.005 (1)	−0.0083 (7)	0.057 (5)
C15	−0.536 (1)	0.106 (1)	−0.0246 (7)	0.055 (4)
C16	−0.381 (1)	0.112 (1)	−0.0116 (6)	0.040 (3)
C17	−0.054 (1)	−0.1164 (9)	−0.0156 (5)	0.032 (3)
C18	0.057 (1)	−0.198 (1)	0.0118 (6)	0.046 (4)
C19	0.085 (1)	−0.297 (1)	−0.0275 (7)	0.059 (4)
C20	0.000 (2)	−0.320 (1)	−0.0971 (8)	0.068 (5)
C21	−0.115 (1)	−0.241 (1)	−0.1267 (7)	0.056 (4)
C22	−0.143 (1)	−0.1407 (10)	−0.0869 (6)	0.045 (4)
C23	−0.136 (1)	0.2535 (10)	0.1753 (6)	0.035 (3)
C24	−0.042 (1)	0.239 (1)	0.2449 (7)	0.049 (4)
C25	−0.092 (2)	0.189 (1)	0.3053 (8)	0.058 (5)
C26	−0.238 (2)	0.153 (1)	0.2967 (8)	0.067 (5)
C27	−0.332 (1)	0.168 (1)	0.2291 (8)	0.057 (4)
C28	−0.281 (1)	0.218 (1)	0.1691 (6)	0.048 (4)
C29	−0.217 (1)	0.4200 (9)	0.0496 (6)	0.038 (3)
C30	−0.252 (1)	0.432 (1)	−0.0289 (6)	0.047 (4)
C31	−0.370 (1)	0.505 (1)	−0.0624 (7)	0.058 (4)
C32	−0.452 (2)	0.565 (1)	−0.0197 (8)	0.067 (5)
C33	−0.420 (2)	0.556 (1)	0.0566 (8)	0.065 (5)
C34	−0.305 (1)	0.484 (1)	0.0923 (7)	0.055 (4)
C35	0.089 (1)	0.4131 (10)	0.1416 (6)	0.039 (3)
C36	0.232 (1)	0.368 (1)	0.1574 (6)	0.050 (4)

C37	0.348 (1)	0.434 (1)	0.1961 (8)	0.064 (5)
C38	0.330 (2)	0.543 (2)	0.219 (1)	0.083 (6)
C39	0.199 (2)	0.592 (1)	0.204 (1)	0.100 (6)
C40	0.071 (2)	0.529 (1)	0.1654 (8)	0.071 (5)

Table 2. *Selected geometric parameters* (Å, °)

Pd1—S1	2.323 (3)	P2—C23	1.83 (1)
Pd1—S2	2.300 (3)	P2—C29	1.83 (1)
Pd1—P1	2.299 (3)	P2—C35	1.83 (1)
Pd1—P2	2.332 (3)	N1—C3	1.13 (1)
S1—C1	1.73 (1)	N2—C4	1.11 (2)
S2—C2	1.73 (1)	C1—C2	1.35 (1)
P1—C5	1.820 (9)	C1—C3	1.44 (1)
P1—C11	1.82 (1)	C2—C4	1.45 (2)
P1—C17	1.83 (1)		
S1—Pd1—S2	88.75 (10)	S2—Pd1—P1	88.83 (10)
S1—Pd1—P1	170.3 (1)	S2—Pd1—P2	165.36 (9)
S1—Pd1—P2	87.31 (10)	P1—Pd1—P2	97.28 (9)

H atoms were fixed at calculated positions with $U_{\rm iso}$ = 1.2 × $U_{\rm eq}$ of the C atoms to which they are attached. The weighting scheme used included a 'fudge factor' p based on multiple measurements for the three standard reflections. Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN TABLES*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1118). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dichloro[6-methyl-2-(2-pyridylmethyl)-pyridine-*N*,*N*′]copper(II)

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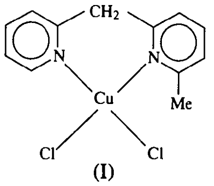
Abstract

The crystal structure of [$CuCl_2(C_{12}H_{12}N_2)$] is reported. The complex is monomeric, with the Cu atom coordinated to two N atoms and two Cl^- ions. The compound displays simple paramagnetic behaviour.

Comment

Copper(II) shows a great 'plasticity' in its coordination geometries, as expected for an ion with Jahn–Teller active ground states (Bersuker, 1984). This fact has permitted the magneto-structural studies of Cu^{II} complexes with heterocyclic amines. Continuing the characterization of complexes with di(2-pyridyl)methane ligands, we have prepared [$Cu(MeDPM)_2Cl_2$], (I), where MeDPM is 6-methyl-2-(2-pyridylmethyl)pyridine. While the chloro and bromo di(2-pyridyl)copper(II) complexes are dimeric in nature and show a ferromagnetically coupled ground state (Spodine, Manzur, Peña & Garland, 1986; Spodine *et al.*, 1993), the title complex is found to be monomeric, with simple paramagnetic behaviour.



(I)