& DiGiacomo, 1981; Dines, Cooksey, Griffith & Lane, 1983; Johnson, Jacobsen, Brody & Lewandowski, 1984), but no divalent metal phosphonates based on the layered $Mg(HPO_4).3H_2O$ (Abbona, Boistelle & Hauser, 1979) structure have been reported. Our finding that these compounds can be prepared on surfaces as thin multilayer films (Lee, Kepley, Hong, Cao & Mallouk, 1987) motivated a systematic study of the reactions of soluble Mg, Ca and Mn salts with alkyl- and aryl-phosphoric acids (Cao, Lee, Lynch & Mallouk, 1987) in which crystalline layered compounds of formula $M(O_3PR).H_2O$ and $M(HO_3PR)_2$ were found.

Funding for this project was provided by the Texas Advanced Technology Research Program, the Robert A. Welch Foundation, and the National Institutes of Health (GM 31077).

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Acta Cryst. (1988). C**44**, 367–369

Structure of a Complex of Palladium(II) Chloride and 5-Phenoxy-1-phenyltetrazole Co-crystallized with 1,2-Dichloroethane Solvent

By Derek J. Chadwick, Robert A. W. Johnstone and Peter J. Price

The Robert Robinson Laboratories, Department of Organic Chemistry, Liverpool University,
PO Box 147, *Liverpool L*69 3*BX, England*

and Marjorie M. Harding

The Donnan Laboratories, Department of Inorganic, Physical and Industrial Chemistry, Liverpool University,
PO Box 147, *Liverpool L*69 3*BX, England*

(*Received* 22 *July* 1987; *accepted* 9 *October* 1987)

Abstract. $[PdCl_2(C_{13}H_{10}N_4O)_2].C_2H_4Cl_2$, $M_r = 752\cdot75$, monoclinic, $P2_1/c$, $a = 10\cdot151\ (2)$, $b = 9\cdot695\ (3)$, $c = 17\cdot186\ (4)$ Å, $\beta = 104\cdot86\ (2)^\circ$, $V = 1703\ (1)$ Å^3, $Z = 2$, $D_m = 1\cdot55$, $D_x = 1\cdot57$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 41$ cm^{-1}, $F(000) = 780$, $T = 293$ K, $R = 0\cdot058$ for 2207 unique observed reflections with $F > 1\cdot5\sigma(F)$. The complex crystallizes with a molecule of 1,2-dichloroethane solvent. Each Pd atom is bound to two *trans* Cl atoms and, coordinatively, to the N(3) atom in each of the two tetrazolyl rings to form a square-planar structure. The Pd—N(3) bonds lie in the planes of the coordinated tetrazolyl rings.

Experimental. Compound obtained by addition of 5-phenoxy-1-phenyltetrazole to solution of bis(benzonitrile)palladium(II) chloride in dichloromethane. Crystals obtained by slow diffusion of pentane into a solution of this complex in 1,2-dichloroethane. Cuboid *ca* 0·30 mm in edge mounted in glass capillary. Density measured by flotation in a mixture of tetrachloromethane and hexane. Intensities measured by SERC service with an Enraf–Nonius CAD-4 diffractometer and ω–2θ scans. Unit cell determined from least-squares analysis of angle data for 25 reflections with $6 < \theta < 19^\circ$. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1},

0108-2701/88/020367-03$03.00

Table 1. *Fractional atomic coordinates, mean isotropic thermal parameters, and their e.s.d.'s*

$\bar{U} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U or $\bar{U}$(Å^2)
Pd(1)	0·0000	0·0000	0·0000	0·0370 (4)
Cl(1)	0·2119 (2)	−0·0227 (2)	0·0842 (1)	0·0760 (13)
N(1)	0·1259 (5)	0·3066 (4)	−0·1223 (3)	0·0392 (24)
N(2)	0·0565 (5)	0·2591 (4)	−0·0709 (3)	0·0456 (27)
N(3)	0·0748 (5)	0·1270 (4)	−0·0691 (3)	0·0412 (25)
N(4)	0·1533 (5)	0·0832 (4)	−0·1168 (3)	0·0449 (26)
C(5)	0·1841 (6)	0·1971 (5)	−0·1489 (3)	0·0440 (30)
O(6)	0·2602 (5)	0·2130 (4)	−0·2005 (3)	0·0658 (28)
C(7)	0·3034 (6)	0·0890 (5)	0·2309 (4)	0·0476 (32)
C(8)	0·2273 (7)	0·0416 (8)	−0·3021 (4)	0·0575 (38)
C(9)	0·2698 (9)	−0·0794 (7)	−0·3334 (5)	0·0716 (47)
C(10)	0·3886 (8)	−0·1411 (7)	−0·2931 (5)	0·0733 (47)
C(11)	0·4626 (7)	−0·0927 (7)	−0·2211 (5)	0·0711 (48)
C(12)	0·4205 (7)	0·0257 (7)	−0·1873 (5)	0·0599 (41)
C(13)	0·1320 (5)	0·4513 (5)	−0·1378 (3)	0·0388 (28)
C(14)	0·1303 (7)	0·5406 (6)	−0·0745 (4)	0·0529 (36)
C(15)	0·1377 (8)	0·6812 (6)	−0·0892 (4)	0·0605 (40)
C(16)	0·1487 (8)	0·7292 (6)	−0·1633 (5)	0·0663 (43)
C(17)	0·1502 (7)	0·6387 (6)	−0·2238 (4)	0·0592 (38)
C(18)	0·1428 (7)	0·4965 (5)	−0·2120 (4)	0·0484 (34)
Cl(2)	0·4277 (6)	0·2977 (6)	0·0175 (4)	0·1701 (30)
C(19)	0·4976 (26)	0·4863 (19)	0·0322 (12)	0·1502 (77)

Table 2. *Bond distances* (Å), *bond angles* (°), *and their e.s.d.'s* (*not including* H *atoms*)

Pd(1)	Cl(1)	2·274 (2)	Pd(1)	N(3)	1·991 (4)
N(1)	N(2)	1·345 (7)	N(1)	C(5)	1·350 (7)
N(1)	C(13)	1·432 (7)	N(2)	N(3)	1·293 (6)
N(3)	N(4)	1·350 (6)	N(4)	C(5)	1·308 (7)
C(5)	O(6)	1·326 (7)	O(6)	C(7)	1·424 (6)
C(7)	C(8)	1·349 (6)	C(7)	C(12)	1·376 (9)
C(8)	C(9)	1·404 (11)	C(9)	C(10)	1·365 (11)
C(10)	C(11)	1·356 (10)	C(11)	C(12)	1·402 (10)
C(13)	C(14)	1·394 (8)	C(13)	C(18)	1·379 (8)
C(14)	C(15)	1·392 (9)	C(15)	C(16)	1·387 (10)
C(16)	C(17)	1·364 (9)	C(17)	C(18)	1·398 (7)
Cl(2)	C(19)	1·954 (20)			

Cl(1)	Pd(1)	N(3)	89·8 (1)	N(2)	N(1)	C(5)	107·6 (4)
N(2)	N(1)	C(13)	120·8 (4)	C(5)	N(1)	C(13)	131·6 (5)
N(1)	N(2)	N(3)	105·0 (4)	Pd(1)	N(3)	N(2)	123·5 (3)
Pd(1)	N(3)	N(4)	122·8 (3)	N(2)	N(3)	N(4)	113·6 (4)
N(3)	N(4)	C(5)	103·5 (4)	N(1)	C(5)	N(4)	110·4 (5)
N(1)	C(5)	O(6)	121·0 (5)	N(4)	C(5)	O(6)	128·6 (5)
C(5)	O(6)	C(7)	115·7 (4)	O(6)	C(7)	C(8)	117·5 (6)
O(6)	C(7)	C(12)	118·7 (6)	C(8)	C(7)	C(12)	123·8 (6)
C(7)	C(8)	C(9)	118·1 (6)	C(8)	C(9)	C(10)	119·3 (7)
C(9)	C(10)	C(11)	121·5 (7)	C(10)	C(11)	C(12)	120·3 (7)
C(7)	C(12)	C(11)	116·9 (7)	N(1)	C(13)	C(14)	117·0 (5)
N(1)	C(13)	C(18)	120·0 (5)	C(14)	C(13)	C(18)	123·0 (5)
C(13)	C(14)	C(15)	117·0 (6)	C(14)	C(15)	C(16)	121·1 (6)
C(15)	C(16)	C(17)	120·2 (5)	C(16)	C(17)	C(18)	120·8 (6)
C(13)	C(18)	C(17)	117·9 (6)				

$0 < h < 12$, $0 < k < 11$, $-20 < l < 20$; empirical absorption correction applied, transmission factors 1·000–0·880. Three standard reflections (608, $75\bar{5}$, $31\bar{3}$) varied ±5%; linear-drift correction applied. 3030 reflections measured, 2778 unique ($R_{\text{int}} = 0{\cdot}024$), 571 reflections with $F < 1{\cdot}5\sigma(F)$ considered unobserved. Solved from Fourier synthesis with *SHELX*76 (Sheldrick, 1976) assuming Pd at origin. Full-matrix least squares on F values with *SHELX*76. Non-H atoms refined anisotropically, H atoms calculated geometrically and allowed to 'ride' on associated heavy atoms with a common isotropic temperature factor. Intermediate difference synthesis revealed presence of a molecule of 1,2-dichloroethane solvent apparently subject to some disorder: positional and isotropic vibration parameters of Cl and C atoms and site-occupancy factor (final value 0·82) also refined for a total of 188 variables. $R = 0{\cdot}058$, $wR = 0{\cdot}064$ where weight$=4{\cdot}6917/[\sigma^2(F) + 0{\cdot}00093F^2]$. Final $(\Delta/\sigma)_{\max} < 0{\cdot}002$, $\Delta\rho_{\max} = 1{\cdot}34$ and $\Delta\rho_{\min} = -0{\cdot}85$ e Å^{-3} on final difference map in solvent region, but elsewhere $< 0{\cdot}6$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom parameters are listed in Table 1 and bond lengths and angles in Table 2. The molecule and numbering scheme are shown in Fig. 1.

Related literature. Other than for closely similar types of tetrazolato/metal complexes, there seems little uniformity in either the arrangement of ligands around the central metal or in the point of attachment of the tetrazolato ring. Thus $ZnCl_2$ forms a tetrahedral complex with 1-methyltetrazole in which the N(4) atom is coordinated to zinc (Baenziger & Schultz, 1971). Other complexes are square planar or octahedral (Ansell, 1973; Kreutzer, Weis, Boehme, Kemmerich, Beck, Spencer & Mason, 1972) or tetrahedral (Gaughan, Bowman & Dori, 1972) and involve either

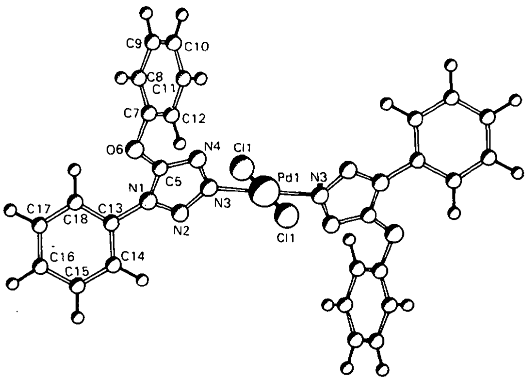

Fig. 1. A view of the molecule drawn with *PLUTO* (Motherwell & Clegg, 1978).

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* Lists of structure factors, H-atom coordinates and isotropic temperature factors, and anisotropic temperature factors for non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44439 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

coordinate or σ bonds to the N(1, 2 or 4) atoms; the square-planar or octahedral arrangement around the central metal can change simply with change of anion (Bowers & Popov, 1968). The square-planar complex reported here, coordinated through the N(3) atom, appears to be yet another variation similar to the σ complex formed with palladium bis(5-phenyltetrazolate) and triphenylphosphine (Kreutzer *et al.*, 1972).

PJP is indebted to the SERC for a research studentship.

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Acta Cryst. (1988). C**44**, 369–370

Structure of Bis(tetraphenylphosphonium) Oxothiotetra(thiocyanato-*N*)tungstate(VI) Methyl Cyanide Solvate

By C. Potvin* and J. M. Manoli

Laboratoire de Cinétique Chimique, Université Pierre et Marie Curie, 1 *rue Guy de la Brosse,* 75005 *Paris, France*

S. Marzak

Laboratoire de Chimie des Polymères Inorganiques, Université Pierre et Marie Curie, 8 *rue Cuvier,* 75252 *Paris CEDEX* 05, *France*

and F. Secheresse

Laboratoire de Chimie des Métaux de Transition, Université Pierre et Marie Curie, 8 *rue Cuvier,* 75252 *Paris CEDEX* 05, *France*

(*Received* 27 *May* 1987; *accepted* 9 *October* 1987)

Abstract. $[P(C_6H_5)_4]_2[WOS(NCS)_4].CH_3CN$, $M_r = 1184{\cdot}1$, triclinic, $P\bar{1}$, $a = 13{\cdot}414\,(6)$, $b = 19{\cdot}84\,(2)$, $c = 11{\cdot}962\,(7)$ Å, $\alpha = 107{\cdot}34\,(5)$, $\beta = 117{\cdot}95\,(4)$, $\gamma = 75{\cdot}06\,(3)°$, $V = 2644\,(6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}48$ Mg m^{-3}, $T = 293$ K, Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å), $F(000) = 1187$, $\mu = 23{\cdot}5$ cm^{-1}, $R = 0{\cdot}050$ for 9320 reflections. The environment of W^{VI} is an approximate octahedron with S and O atoms *cis*-orientated and four N-bonded NCS groups. The W—N bonds to the NCS groups *trans* to the S or O atoms are significantly longer than the other two. The W—N—C and N—C—S angles are all linear within 10° except for the W—N(2)—C(2)—S(2) moiety. The only short intermolecular contact is the S(3)···S(3) distance [3·509 (4) Å].

Experimental. Triclinic prism (0·40 × 0·32 × 0·30 mm) obtained at 273 K from the reaction of $[P(C_6H_5)_4]_2WS_4$ and AgSCN in CH_3CN. Philips PW 1100 diffractometer, graphite monochromator, cell parameters from 25 reflections ($7° < \theta < 14°$). Intensities were collected by a flying step scan technique, scan width 1·4°, scan speed 0·02 s in steps of 0·02° (θ); 11 803 independent reflections with $3° < \theta < 29°$ ($\pm h$, $\pm k$, $+l$; $h_{\max} = 16$, $k_{\max} = 26$, $l_{\max} = 13$); 9320 with $F > 6\sigma(F)$. Three standard reflections were measured every hour, no significant intensity decay was observed; Lp correction; absorption ignored. Structure solution by Patterson and difference syntheses. Refinements on F for 359 parameters (*SHELX76*, Sheldrick 1976); anisotropic thermal parameters for W, S, P, O and N, C for the anion; isotropic for C and N atoms of the cation and solvate; phenyl H on external C—C—C

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* To whom correspondence should be addressed.

0108-2701/88/020369-02$03.00