Table 3. *Comparison of some non-bonded distances* (Å) *and angles* (°) *in the tetramers of* (p-$CH_3OC_6H_4)_2TeCl_2$ (*A*), (p-$BrC_6H_4)(C_6H_5)TeCl_2$ (*B*) *and* $C_{12}H_8OTeCl_2$ (*C*)

	Step	*A*	*B*	*C*
Te$\cdots$Te'	Vertical*	4·678 (1)	4·609 (2)	4·594 (3)
	Central	4·472 (1)	4·720 (2)	4·793 (3)
TeClTe'	Vertical	99·1 (1)	83·0 (2)	98·9 (1)
	Central	78·6 (1)	82·7 (2)	88·0 (1)
ClTeCl'	Vertical	80·9 (1)	96·8 (2)	80·9 (1)
	Central	101·4 (1)	97·3 (2)	92·0 (1)

* Averaged for upper and lower step.

[O(4)C(47)] in molecule 2, thus minimizing the repulsions. Distortions in the central step formed by Te(1')$\cdots$Cl(4)$\cdots$Te(1)$\cdots$Cl(4') also appear to be designed to minimize repulsion. Thus in *C*, this central step approximates to a rectangle with the Cl$\cdots$Te$\cdots$Cl angle being 92·0 (1)° and the Te$\cdots$Te distance across the step being 4·793 (3) Å (Table 3). The shortest Te$\cdots$Te distance in this compound is in fact between molecules 1 and 2 and averages 4·594 (3) Å. In *B*, where the alternating packing of substituted and non-substituted rings presumably helps alleviate the repulsion between substituents, there is some distortion around the central rectangle so that the Cl$\cdots$Te$\cdots$Cl angle is now 97·3 (2)° with the Te$\cdots$Te distance slightly reduced to 4·720 (2) Å. However, in *A*, where methoxy groups on molecules 1 and 2 are in fairly close proximity, the distortion is larger so that the Cl$\cdots$Te$\cdots$Cl angle is now 101·4 (1)° and the Te$\cdots$Te distance reduced to 4·472 (1) Å. This has the effect of elongating the vertical steps so that the Te(1)$\cdots$Te(2) distances are now longer [on average 4·678 (1) Å] and the methoxy groups slightly further apart. In $TeCl_4$ (Buss & Krebs, 1971) in which the tetramer has all four Te atoms in the alternate vertices of an approximate cube, the Te$\cdots$Te distance is 4·309 (1) Å, considerably shorter than the shortest distance in *A*.

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[2,2-Di(2-pyridyl)-1,3-dioxolane-*N*,*N*′]dichloropalladium(II), $[PdCl_2(C_{13}H_{12}N_2O_2)]$

By George R. Newkome,* Vinod K. Gupta, Kevin J. Theriot, James C. Ewing, Steven P. Wicelinski, William R. Huie, Frank R. Fronczek and Steven F. Watkins

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803, *USA*

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Abstract. $M_r = 405{\cdot}6$, monoclinic, $P2_1/c$, $a = 9{\cdot}767\,(1)$, $b = 19{\cdot}224\,(3)$, $c = 15{\cdot}351\,(3)$ Å, $\beta = 90{\cdot}34\,(1)^\circ$, $V = 2882\,(2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}869\,(1)$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 16{\cdot}45$ cm^{-1}, $F(000) = 1600$, $T = 296$ K, $R = 0{\cdot}031$ for 4502 observations (of 5078 unique data). The title molecule consists of a central Pd^{II} atom bonded to two pyridine nitrogens and two chlorine atoms. The coordination geometry of Pd is square planar with slight tetrahedral distortion. The asymmetric unit

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* To whom correspondence should be addressed.

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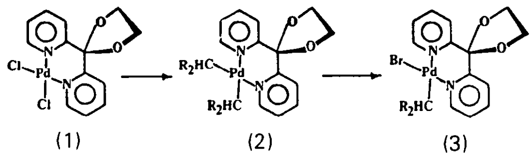
contains two distinct molecules which differ from each other in the conformation of the acetal rings and distortions in the coordination sphere. Both exhibit a close intramolecular contact between Pd and a single acetal oxygen.

Introduction. The title compound (1) was prepared as precursor to a novel series of (malonate ester-*C*) palladium(II) complexes (2) and (3) (Newkome, Gupta, Fronczek & Taylor, 1983). The structural analysis was undertaken to provide a model for a carbanion ligand exchange pathway (Newkome, Gupta, Taylor & Fronczek, 1984), and as an exercise in a laboratory course in crystallography. Similar palladium(II) complexes containing five-membered chelate rings have shown acute deviations from coplanarity of the pyridine ligand and the coordination plane as well as other deviations from ideality (Newkome, Fronczek, Gupta, Puckett, Pantaleo & Kiefer, 1982).



R=CO$_2$Me or CO$_2$Et

Experimental. Equimolar mixture of 2,2-di(2-pyridyl)-1,3-dioxolane (Newkome, Sauer & McClure, 1973) and PdCl$_2$ in CH$_3$CN at 298 K for 3 h gave (95%) orange crystalline precipitate (1):* m.p. 578 K (decomposed); ^{1}H NMR(CDCl$_3$) δ 4·40 (*m*, CH$_2$, 4H), 7·41 (*ddd*, 5-pyH, $J_{5,6} = 4{\cdot}9$, $J_{5,4} = 7{\cdot}3$, $J_{5,3} = 1{\cdot}2$ Hz, 2 H), 7·90 (*m*, 3,4-pyH, 4 H), 9·27 (*dd*, 6-pyH, $J_{6,5} = 4{\cdot}9$, $J_{6,4} = 1{\cdot}2$Hz, 2 H); IR(CsI) 1590, 1454, 1436 cm^{-1}. Rectangular prismatic crystal, by evaporation from CH$_3$CN, cleaved to 0·32 × 0·24 × 0·12 mm. Enraf–Nonius CAD-4 diffractometer with graphite monochromator and Mo $K\alpha$ radiation. Cell dimensions from least-squares fit of setting angles of 25 reflections, $13 < \theta < 14°$. Systematic absences $h0l$: l odd and $0k0$: k odd. Data collection by ω–2θ scans. Three standard reflections (400, 080, 006), no significant variation in intensities. Absorption corrections using ψ scans of reflections near $\chi = 90°$, min. relative transmission factor 75·58%. $0 \le h \le 11$, $0 \le k \le 22$, and $-18 \le l \le 18$, 5563 reflections ($2 < \theta < 25°$) measured. Equivalent data averaged ($R_{\rm int} = 0{\cdot}030$), 5078 unique data. 4502 reflections with $I > 3\sigma(I)$ used in structure solution, by Patterson and difference Fourier syntheses. Refinement by full-matrix least squares (based on F) with $w = [\sigma^2(F) + 0{\cdot}002218F^2]^{-1}$, non-hydrogen atoms anisotropic. *SHELX* (Sheldrick, 1976) and locally generated programs used; scattering factors of Cromer & Mann (1968), anomalous coefficients of Cromer & Liberman (1970). Hydrogen atoms from difference maps following anisotropic refinement as peaks of density 0·52–0·67 e Å^{-3} and constrained to 'ride' 1·08 Å from bonded carbon, with individually refined isotropic thermal parameters. Convergence achieved (max. $\Delta/\sigma = 0{\cdot}24$, av. $\Delta/\sigma = 0{\cdot}029$) with $R = 0{\cdot}031$, $R_w = 0{\cdot}044$ for all data and 385 parameters. Max. residual peak in final difference synthesis 0·38 e Å^{-3}. Coordinates of non-hydrogen atoms and their equivalent isotropic thermal parameters are given in Table 1.*

Table 1. *Fractional atomic positional parameters* (×10^4) *and equivalent isotropic temperature factors* (Å^2 ×10^3)

$U_{\rm eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	$U_{\rm eq}$
Pd(1)	−106·3 (4)	5629·4 (2)	2799·9 (2)	32·0 (1)
Cl(1)	531 (2)	4562 (1)	2277 (1)	62 (1)
Cl(2)	2129 (1)	5974 (1)	2910 (1)	51·7 (5)
N(1)	−742 (4)	6560 (2)	3276 (3)	34 (1)
N(2)	−2108 (4)	5371 (2)	2703 (3)	36 (1)
O(1)	−1389 (4)	5460 (2)	4549 (2)	45 (1)
O(2)	−3425 (4)	6006 (2)	4748 (3)	55 (1)
C(1)	−206 (5)	7153 (3)	2986 (4)	45 (2)
C(2)	−646 (6)	7793 (3)	3270 (4)	59 (2)
C(3)	−1642 (6)	7820 (3)	3898 (5)	61 (2)
C(4)	−2194 (5)	7213 (3)	4202 (4)	48 (2)
C(5)	−1749 (5)	6581 (3)	3872 (3)	33 (1)
C(6)	−2391 (5)	5895 (3)	4146 (3)	37 (2)
C(7)	−1466 (8)	5613 (4)	5468 (4)	75 (3)
C(8)	−2902 (8)	5851 (5)	5594 (4)	87 (3)
C(9)	−2983 (5)	5518 (2)	3348 (3)	37 (2)
C(10)	−4353 (6)	5371 (3)	3280 (4)	52 (2)
C(11)	−4857 (7)	5057 (3)	2528 (4)	61 (2)
C(12)	−3965 (6)	4912 (3)	1871 (4)	51 (2)
C(13)	−2603 (6)	5066 (3)	1970 (3)	46 (2)
Pd(2)	4829·7 (4)	1701·7 (2)	3994·1 (2)	29·6 (1)
Cl(3)	2641 (1)	2011 (1)	3633 (1)	53·6 (5)
Cl(4)	4026 (1)	986 (1)	5074 (1)	44·5 (4)
N(3)	6809 (4)	1450 (2)	4283 (2)	31 (1)
N(4)	5596 (4)	2320 (2)	3034 (2)	34 (1)
O(3)	8441 (4)	3136 (2)	4149 (3)	49 (1)
O(4)	6357 (4)	2884 (2)	4722 (2)	42 (1)
C(14)	7179 (5)	783 (3)	4429 (3)	41 (2)
C(15)	8530 (6)	590 (3)	4558 (4)	50 (2)
C(16)	9513 (6)	1092 (3)	4553 (4)	56 (2)
C(17)	9161 (5)	1778 (3)	4406 (3)	44 (2)
C(18)	7786 (5)	1938 (2)	4273 (3)	32 (1)
C(19)	7330 (5)	2683 (2)	4086 (3)	34 (1)
C(20)	8143 (8)	3659 (5)	4754 (7)	102 (4)
C(21)	6642 (7)	3583 (4)	4919 (6)	78 (3)
C(22)	6692 (5)	2726 (2)	3177 (3)	35 (1)
C(23)	7239 (6)	3147 (3)	2536 (4)	46 (2)
C(24)	6632 (7)	3141 (3)	1724 (3)	57 (2)
C(25)	5509 (7)	2728 (3)	1570 (4)	56 (2)
C(26)	5003 (6)	2328 (3)	2240 (3)	46 (2)

Discussion. Two independent molecules *A* and *B* are contained in the unit cell. In both, the palladium atoms attain the expected square-planar coordination, being bonded to two chlorine atoms and two nitrogen atoms

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* Analytical data are within acceptable limits.

* Lists of structure factors, coordinates of hydrogen atoms, bond lengths, bond angles, deviations from least-squares planes and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39445 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Pd(1)—Cl(1)	2·290 (2)	N(1)—Pd(1)—N(2)	86·8 (2)
Pd(1)—Cl(2)	2·287 (1)	N(1)—Pd(1)—Cl(1)	177·8 (5)
Pd(2)—Cl(3)	2·285 (1)	N(1)—Pd(1)—Cl(2)	90·7 (1)
Pd(2)—Cl(4)	2·297 (1)	N(2)—Pd(1)—Cl(1)	91·1 (1)
Pd(1)—N(1)	2·030 (4)	N(2)—Pd(1)—Cl(2)	177·3 (5)
Pd(1)—N(2)	2·022 (4)	Cl(1)—Pd(1)—Cl(2)	91·40 (6)
Pd(2)—N(3)	2·039 (4)		
Pd(2)—N(4)	2·039 (4)	N(3)—Pd(2)—N(4)	86·8 (2)
		N(3)—Pd(2)—Cl(3)	177·9 (9)
		N(3)—Pd(2)—Cl(4)	91·6 (1)
		N(4)—Pd(2)—Cl(3)	91·1 (1)
		N(4)—Pd(2)—Cl(4)	178·3 (3)
		Cl(3)—Pd(2)—Cl(4)	90·45 (5)

Table 3. *Selected torsion angles* (°)

Molecule *A*		
Chelate ring	Pd(1)—N(1)—C(5)—C(6)	−1·5 (6)
	N(1)—C(5)—C(6)—C(9)	−57·8 (5)
	C(5)—C(6)—C(9)—N(2)	58·3 (6)
	C(6)—C(9)—N(2)—Pd(1)	0·7 (6)
	C(9)—N(2)—Pd(1)—N(1)	−43·7 (4)
	N(2)—Pd(1)—N(1)—C(5)	44·0 (4)
Acetal ring	C(6)—O(1)—C(7)—C(8)	26·9 (6)
	O(1)—C(7)—C(8)—O(2)	−15·7 (7)
	C(7)—C(8)—O(2)—C(6)	−1·8 (7)
	C(8)—O(2)—C(6)—O(1)	19·1 (6)
	O(2)—C(6)—O(1)—C(7)	−29·1 (5)
Molecule *B*		
Chelate ring	Pd(2)—N(3)—C(18)—C(19)	3·2 (5)
	N(3)—C(18)—C(19)—C(22)	−62·1 (5)
	C(18)—C(19)—C(22)—N(4)	59·2 (5)
	C(19)—C(22)—N(4)—Pd(2)	1·1 (6)
	C(22)—N(4)—Pd(2)—N(3)	−43·3 (4)
	N(4)—Pd(2)—N(3)—C(18)	40·7 (3)
Acetal ring	C(19)—O(3)—C(20)—C(21)	−12·0 (7)
	O(3)—C(20)—C(21)—O(4)	25·0 (8)
	C(20)—C(21)—O(4)—C(19)	−28·4 (7)
	C(21)—O(4)—C(19)—O(3)	21·8 (5)
	O(4)—C(19)—O(3)—C(20)	−5·4 (6)

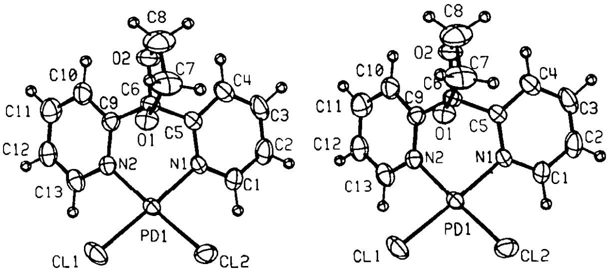

Fig. 1. Stereopair of molecule *A*, illustrating the numbering scheme. Thermal ellipsoids are drawn at the 42% probability level.

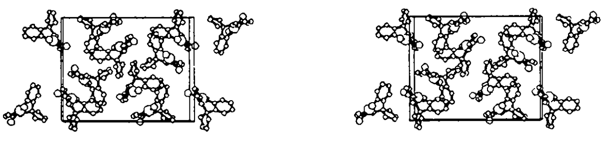

Fig. 2. Crystal packing diagram. *b* axis is horizontal, *c* axis is vertical. Atoms are represented by circles reflecting their radii.

of the dipyridyl acetal ligand. A stereoview of *A* with atomic numbering scheme is given in Fig. 1; *B* is numbered analogously. Selected bond lengths and angles of both are listed in Table 2. The four Pd—N bond lengths do not differ significantly from the average [2·032 (2) Å], nor do the Pd—Cl bond lengths of *A* differ from the average [2·290 (1) Å, see Table 2]. The two Pd—Cl distances in *B* do differ significantly from the average as well as from each other. However, the average bond distances in the coordination sphere of Pd in both *A* and *B* are in reasonable agreement with those found in similar square-planar palladium(II) complexes (Newkome *et al.*, 1982). Values of 2·296 (3) and 2·301 (3) Å for Pd—Cl and 2·014 (6) and 2·032 (6) Å for Pd—N have been determined in the closely related dichloro[dihydroxydi(2-pyridyl)methane]palladium(II) (4) (Annibale *et al.*, 1981). The chelating N—Pd—N angle of 86·8 (2)° in *A* and *B* agrees with the value of 87·1 (2)° reported for (4).

In both *A* and *B*, the six-membered chelate ring assumes a boat conformation (see Table 3). The coordination geometry around Pd(1) (in *A*) is tetrahedrally distorted square planar. The deviation of Cl(1) from the Pd(1)—N(1)—N(2) plane is 0·030 (1) Å and that of Cl(2) is −0·038 (1) Å. In *B*, Pd(2), N(2), N(3), Cl(4) all lie in a plane within experimental error, and Cl(3) is 0·030 (1) Å out of that plane. Each of the four pyridine rings is planar, and they produce intramolecular dihedral angles of 64·5 (3) and 63·4 (3)° in *A* and *B*, respectively. These angles differ significantly from the value of 73° found in (2) and 67·3° in (3) (Newkome, Gupta, Fronczek & Taylor, 1983) but agree well with a value of 65·5° in (4) (Annibale *et al.*, 1981). The acetal rings of the two molecules differ significantly (see Table 3); both adopt an envelope conformation, but the envelope 'flap' in *B* is a carbon atom [C(21)], whereas, in *A*, oxygen atom O(1) occupies that unique conformational position. The Pd(1)···O(1) and Pd(2)···O(4) distances [2·987 (4) and 2·935 (3) Å, respectively] are close to the minimum expected van der Waals distance of 3·0 Å. This contact is also observed in (4), in which the Pd···O distance is 2·824 (6) Å (Annibale *et al.*, 1981). A crystal packing diagram of the unit cell is depicted in Fig. 2; no intermolecular contacts less than 3·5 Å between non-hydrogen atoms exist.

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Acta Cryst. (1984). C**40**, 1355–1357

Structure de (Diméthyl-5,5 Tétraphényl-1,2,8,9 Tétraaza-3,4,6,7 Nonatétraène-1,3,6,8 Diolato-1,9-*O,O′,N,N′*)nickel(II), $Ni(C_{31}H_{26}N_4O_2)$

Par F. Abraham, J. M. Capon, G. Nowogrocki et S. Sueur

Equipe de Cristallochimie et Physicochimie du Solide, ERA–CNRS 1004, *Université de Valenciennes et Ecole Nationale Supérieure de Chimie de Lille, BP* 108, 59652 *Villeneuve d'Ascq CEDEX, France*

et C. Brémard

LASIR, CNRS, Université de Lille I, 59655 *Villeneuve d'Ascq CEDEX, France*

(*Reçu le* 5 *janvier* 1984, *accepté le* 25 *avril* 1984)

Abstract. $M_r = 545{\cdot}3$, monoclinic, $P2_1/c$, $a = 12{\cdot}63\,(1)$, $b = 13{\cdot}00\,(2)$, $c = 19{\cdot}44\,(2)$ Å, $\beta = 121{\cdot}47\,(8)°$, $V = 2723$ Å^3, $Z = 4$, $D_m = 1{\cdot}31\,(3)$, $D_x = 1{\cdot}33$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 7{\cdot}49$ cm^{-1}, $F(000) = 1128$, room temperature, $R = 0{\cdot}042$, $R_w = 0{\cdot}043$ for 1947 unique diffractometer data. The macrocycle is coordinated to the Ni atom as a tetradentate ligand with a *cis* N_2O_2 donor in a nearly square-planar geometry. The Ni atom is displaced 0·024 (1) Å from the N_2O_2 mean plane. The atoms of the five-membered monoaza and triaza chelate rings and the six-membered diazachelate ring adopt a nearly coplanar conformation.

Introduction. La réaction template entre le benzilmonohydrazone et l'acétone en présence d'ions Ni^{2+} conduit à la formation d'un complexe macrocyclique (Kerwin & Melson, 1972) noté NiMMK. Les auteurs ont tenté d'élucider les mécanismes réactionnels qui conduisent à la formation *in situ* d'un tel macrocycle (Bonfoey & Melson, 1975). La réactivité des groupements carbonyles du macrocycle vis à vis d'amines a également été étudiée (Melson, 1974; Kerwin & Melson, 1973; Funke & Melson, 1975; Nafisi-Movaghar & Melson, 1975; Shalhoub, Reider & Melson, 1982). Les hypothèses structurales découlent d'études spectroscopiques infrarouge et RMN ^{1}H (Melson, 1974) mais aucune structure radiocristallographique permettant d'établir définitivement la structure moléculaire des composés n'existe actuellement, ni pour NiMMK, ni pour les dérivés issus de sa condensation avec les amines. Or, la condensation entre des complexes carbonylés et des hydrazones est une voie d'accès à des composés polymétalliques.

Partie expérimentale. Synthèses de NiMMK réalisées suivant la méthode de Kerwin & Melson (1972) conduisent systématiquement à des cristaux maclés inutilisables pour une étude radiocristallographique. Cristaux de bonne qualité obtenus en maintenant le mélange réactionnel: benzilmonohydrazone, acétone, acétate de nickel, dans l'éthanol à 273 K pendant environ 6 mois; microanalyse C,N,H et résultats spectroscopiques identiques à ceux effectués sur les cristaux maclés. Paramètres de maille à partir de 25 réflexions, cristal prismatique, 0,110 × 0,190 × 0,235 mm, masse volumique mesurée par flottation, diffractomètre Philips PW 1100, 4004 réflexions avec $2 < \theta < 23°$, $-12 \leq h \leq 0$, $0 \leq k \leq 14$, $-17 \leq l \leq 17$, dont 2052 avec $I > 3\sigma(I)$, 1947 réflexions indépendantes, 3 réflexions de référence (113, $\bar{3}\bar{1}2$, $\bar{3}12$), mesurées toutes les deux heures sans variation significative d'intensité; correction de Lorentz–polarisation, pas de correction d'absorption; position de l'atome de nickel déduite de la fonction de Patterson, positions des atomes non-hydrogène déduites d'affinements et synthèses de Fourier différences successives; affinement par moindres carrés (basé sur F), matrice entière *SFLS*5 (Prewitt, 1966); paramètres affinés x, y, z, β_{ij}; H localisés par synthèse de Fourier différence, B fixés à B_{eq} des atomes de carbone auxquels ils sont reliés; cycles alternés: affinement x, y, z, β_{ij} des atomes

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