Zellvolumen als auch die thermische Zersetzung zu $KAlF_4.0{,}5H_2O$ einfach zu erklären:*

$$(H_3O)_2KAlF_6 \rightarrow KAlF_4.0{,}5H_2O + 1{,}5H_2O + 2HF.$$

Wir danken der Deutschen Forschungsgemeinschaft und dem Fonds der Chemischen Industrie für ihre Unterstützung.

*_Anmerkung bei der Korrektur:_ Inzwischen wurde dies auch durch Bentrup & Kolditz (1989) klargestellt.

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Coordination Mode of 4-Pyridinecarbaldehyde Phenylhydrazone. Structure of Dichloro(4-pyridinecarbaldehyde phenylhydrazone)(tri-_n_-propylphosphine)palladium(II)

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Abstract. $[PdCl_2(C_9H_{21}P)(C_{12}H_{11}N_3)]$, $M_r = 534{\cdot}8$, monoclinic, $P2_1/c$, $a = 8{\cdot}049$ (1), $b = 25{\cdot}104$ (3), $c = 12{\cdot}251$ (2) Å, $\beta = 91{\cdot}02$ (2)°, $V = 2475{\cdot}1$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}435$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 1{\cdot}03$ mm^{-1}, $F(000) = 1096$, $T = 298$ K, final $R = 0{\cdot}044$, $wR = 0{\cdot}047$ for 2145 independent reflections [$I > 3\sigma(I)$]. The title complex was prepared by reaction of 4-pyridinecarbaldehyde phenylhydrazone (pap) with the dichloro-bridged complex $[Pd_2(PPr^n_3)_2Cl_4]$ in dichloromethane. $[Pd(PPr^n_3)(pap)Cl_2]$ has a square-planar geometry with the two chlorides _trans_ to each other. The hydrazone coordinates through the pyridine N atom in a _syn_ configuration instead of through the generally preferred N-iminic site.

Introduction. The capability of hydrazones to behave as nitrogen-donor ligands with respect to transition metals is well established (Cockburn, Howe, Keating, Johnson & Lewis, 1973). In the hydrazones derived from aldehydes $R_1R_2C{=}N^1{-}N^2HR_3$ both iminic and aminic N atoms (N^1 and N^2) are potential donor sites (Stapfer, D'Andrea & Herber, 1972; Stapfer & D'Andrea, 1971; Bresciani-Pahor, Calligaris, Delise, Randaccio, Maresca & Natile, 1976; Nolte & Singleton, 1974; Natile, Gasparrini, Misiti & Perego, 1977), though coordination through N^1 is generally observed. When R_1 or R_2 are groups bearing donor atoms the hydrazone can exhibit at least one more coordination mode. In addition, these hydrazones may adopt _syn_ or _anti_ configurations. In this paper we report the structure of the complex _trans_-$[Pd(PPr^n_3)(pap)Cl_2]$ where the pap ligand (4-pyridinecarbaldehyde phenylhydrazone) is a potentially multidentate ligand. This compound was obtained by the cleavage of the chloro-bridged complex $[Pd_2(PPr^n_3)_2Cl_4]$ with the hydrazone. Its IR

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spectrum (Nujol mull), showing only one Pd—Cl stretching frequency at 350 cm^{-1}, suggests that the two chlorides are *trans* to each other (Adams, 1967) but does not give any indication of the bonding mode of the pap ligand. On the other hand preliminary data on the photoisomerization (Guglielmo, Giuffrida, Cusumano, Campagna & Ricevuto, 1988) of the coordinated hydrazone from the *syn* to the *anti* form suggest that the pap ligand is coordinated through the pyridine N atom. There is very little difference in the quantum yields for the photoisomerization of the free and coordinated hydrazone. Clearly coordination to palladium through the iminic or aminic N atom would hamper the process. In order to gain conclusive evidence on the geometry of the complex we have determined the crystal and molecular structure by X-ray analysis. Our results show that the complex has a square-planar geometry with the two Cl atoms *trans* to each other and that the hydrazone is indeed coordinated through the pyridine N atom in the *syn* configuration.

Experimental. The ligand pap was prepared by condensation of 4-pyridinecarbaldehyde with phenylhydrazine in methanol in the presence of acetic acid and crystallized from ethanol. The *trans*-[Pd(PPr^n_3)(pap)Cl_2] complex was obtained by the cleavage of the dichloro-bridged [$Pd_2(PPr^n_3)_2Cl_4$] complex with the pap ligand in a molar ratio 1:2 in acetone–chloroform. After evaporation of the solvent, the complex was crystallized from methanol–acetone.

The IR spectra in the range 250–4000 cm^{-1} were recorded as Nujol mulls on a Perkin–Elmer 783 spectrometer. Single crystals suitable for X-ray analysis were crystallized from a methanol solution; a yellow prism with approximate dimensions 0·20 × 0·20 × 0·15 mm was used for intensity-data collection. Accurate unit-cell dimensions and crystal orientation matrices were obtained from least-squares refinement of 2θ, ω, χ and ψ values of 20 strong reflections in the range $14 < 2\theta < 25°$. Siemens–Stoe four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ mode, $2\theta_{max} = 50°$ ($-10 \le h \le 10$, $0 \le k \le 30$, $-15 \le l \le 0$). Three standard reflections ($13\bar{2}$, $21\bar{3}$ and $\bar{3}\bar{3}4$) monitored every 3600 s showed no significant intensity variation over the total exposure time. Lorentz and polarization corrections were applied to the intensity data but no correction for absorption was considered.

The structure was solved using standard Patterson methods, successive least-squares refinements (on F) and difference Fourier maps. All non-H atoms were refined anisotropically, while H atoms were added at calculated positions and included in the structure-factor calculations with a common thermal parameter ($U = 0·08$ Å^2). Of 3980 measured independent reflections, 2145 having $I > 3\sigma(I)$ were used to refine 265 parameters to final residuals of $R = 0·044$ and $wR = 0·047$, $w = 1·3799/[\sigma^2(F_o) + 0·00070F_o^2]$, $S = 1·146$, $(\Delta/\sigma)_{max} < 0·7$, largest peak in final difference map 0·47 e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed with *SHELX76* (Sheldrick, 1976) and *PARST* (Nardelli, 1983) systems of programs on an IBM 4341 computer at the 'Centro di Calcolo dell' università di Messina'. The refined structure was plotted using *ORTEP* (Johnson, 1976).*

Discussion. Atomic positional parameters for non-H atoms are listed in Table 1. The molecular structure is depicted in Fig. 1 together with the atomic labelling scheme. Bond distances and angles are given in Table 2.

The weighted mean plane through the four donor atoms shows a lack of planarity with N(1) and P(1) deviating by 0·128 (7) and 0·015 (3) Å, respectively. Cl(1) and Cl(2) are −0·016 (3) and −0·016 (3) Å on the opposite side of the plane, indicating a slight distortion towards the tetrahedral configuration around the Pd atom, which lies 0·049 (1) Å out of this plane. While the two *trans*-Pd—Cl bond distances [mean value 2·288 (5) Å] are in good agreement with the values found in various square-planar complexes of palladium (Albinati, Anklin, Ganazzoli, Ruegg & Pregosin, 1987), the Cl—Pd—Cl bond angle deviates from linearity [176·4 (5)°]. The Pd—P(1) distance of 2·232 (2) Å is similar to that found in *trans*-[$PdCl_2$(2-NH_2-3-Mepy)(PEt_3)] (Albinati, Arz & Pregosin, 1987); in PPr^n_3 the structural features are as usual, as well as the large thermal parameters or disorder of the propyl chains. The Pd atom completes its coordination sphere by binding the pyridine N atom of the ligand (pap), in contrast to the generally preferred N-iminic or the rarely observed N-aminic (Galli, Gasparrini, Maresca, Natile & Palmieri, 1983) coordination. The Pd—N(1) bond distance of 2·115 (6) Å is relatively long (because of the large *trans* influence of PPr^n_3) and is less than that found in the cited complex (Albinati, Arz & Pregosin, 1987), 2·155 (5) Å. Steric and electronic factors could both cause such a difference.

The pyridine ring (which is planar) makes an angle of 56·1 (1)° with the coordination plane. Pyridine-type ligands normally adopt the best orientation for $p\pi$–$d\pi$ bonding between metal d orbitals and the ligand π-system; in the present compound the long

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52220 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for non-*H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Pd(1)	571 (1)	1321 (1)	1956 (1)	62·0 (1)
Cl(1)	2465 (3)	1061 (1)	3271 (2)	92·6 (1)
Cl(2)	−1354 (3)	1525 (1)	631 (2)	113·0 (1)
P(1)	−962 (3)	1728 (1)	3208 (2)	80·3 (1)
C(1)	220 (12)	2000 (4)	4380 (7)	99·6 (4)
C(2)	1513 (15)	2374 (4)	4085 (10)	114·3 (5)
C(3)	2626 (16)	2488 (6)	5070 (13)	163·2 (7)
C(4)	−2173 (17)	2308 (5)	2663 (10)	172·0 (8)
C(5)	−3100 (27)	2602 (9)	3151 (22)	267·2 (17)
C(6)	−4165 (18)	3003 (6)	2570 (16)	183·1 (9)
C(7)	−2458 (19)	1205 (6)	3741 (13)	192·0 (9)
C(8)	−3023 (29)	1157 (10)	4822 (13)	312·7 (18)
C(9)	−4014 (15)	694 (6)	4926 (15)	176·2 (8)
N(1)	2061 (8)	951 (3)	770 (5)	64·3 (2)
C(10)	1522 (10)	576 (4)	105 (7)	75·6 (2)
C(11)	2496 (11)	330 (3)	−662 (7)	74·4 (3)
C(12)	4127 (10)	493 (3)	−782 (6)	66·1 (3)
C(13)	4688 (10)	887 (3)	−91 (7)	67·4 (3)
C(14)	3644 (10)	1101 (3)	676 (7)	68·6 (3)
C(15)	5220 (12)	255 (3)	−1587 (6)	72·6 (3)
N(2)	6625 (8)	469 (3)	−1761 (5)	67·9 (2)
N(3)	7622 (9)	241 (3)	−2475 (5)	77·1 (3)
C(16)	9083 (10)	500 (4)	−2758 (6)	65·1 (3)
C(17)	9527 (12)	983 (4)	−2312 (8)	87·1 (4)
C(18)	10983 (15)	1222 (4)	−2627 (9)	100·0 (4)
C(19)	11988 (13)	995 (5)	−3380 (9)	102·5 (5)
C(20)	11577 (12)	523 (5)	−3801 (8)	93·8 (4)
C(21)	10115 (12)	273 (4)	−3521 (7)	81·9 (4)

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

Pd(1)—Cl(1)	2·294 (2)	Pd(1)—Cl(2)	2·283 (3)
Pd(1)—P(1)	2·232 (2)	Pd(1)—N(1)	2·115 (6)
P(1)—C(1)	1·840 (7)	P(1)—C(4)	1·870 (8)
P(1)—C(7)	1·904 (9)	C(1)—C(2)	1·453 (13)
C(2)—C(3)	1·516 (16)	C(4)—C(5)	1·213 (22)
C(5)—C(6)	1·496 (24)	C(7)—C(8)	1·414 (9)
C(8)—C(9)	1·417 (25)	N(1)—C(10)	1·315 (10)
N(1)—C(14)	1·336 (9)	C(10)—C(11)	1·380 (11)
C(11)—C(12)	1·385 (10)	C(12)—C(13)	1·373 (10)
C(13)—C(14)	1·380 (10)	C(12)—C(15)	1·461 (10)
N(2)—C(15)	1·274 (9)	N(2)—N(3)	1·328 (8)
N(3)—C(16)	1·394 (10)	C(16)—C(17)	1·375 (12)
C(17)—C(18)	1·377 (12)	C(18)—C(19)	1·363 (13)
C(19)—C(20)	1·334 (13)	C(20)—C(21)	1·382 (12)
C(21)—C(16)	1·384 (10)		
Cl(1)—Pd(1)—Cl(2)	176·4 (1)	Cl(1)—Pd(1)—P(1)	91·0 (1)
Cl(1)—Pd(1)—N(1)	88·8 (2)	Cl(2)—Pd(1)—P(1)	90·5 (1)
Cl(2)—Pd(1)—N(1)	89·8 (2)	P(1)—Pd(1)—N(1)	178·7 (2)
Pd(1)—N(1)—C(14)	119·3 (5)	Pd(1)—N(1)—C(10)	123·8 (5)
C(14)—N(1)—C(10)	116·9 (6)	N(1)—C(14)—C(13)	122·9 (7)
C(14)—C(13)—C(12)	120·2 (7)	C(13)—C(12)—C(11)	116·6 (7)
C(12)—C(11)—C(10)	119·4 (8)	C(11)—C(10)—N(1)	123·9 (7)
C(13)—C(12)—C(15)	121·1 (7)	C(11)—C(12)—C(15)	122·3 (8)
C(12)—C(15)—N(2)	119·1 (8)	C(15)—N(2)—N(3)	118·5 (7)
N(2)—N(3)—C(16)	119·0 (7)	N(3)—C(16)—C(17)	121·8 (7)
N(3)—C(16)—C(21)	119·6 (8)	C(16)—C(17)—C(18)	119·2 (8)
C(17)—C(18)—C(19)	121·8 (9)	C(18)—C)19)—C(20)	119·1 (9)
C(19)—C(20)—C(21)	121·0 (9)	C(20)—C(21)—C(16)	120·3 (9)
C(21)—C(16)—C(17)	118·5 (8)	Pd(1)—P(1)—C(1)	114·9 (3)
Pd(1)—P(1)—C(4)	113·7 (4)	Pd(1)—P(1)—C(7)	106·3 (4)
P(1)—C(1)—C(2)	114·1 (8)	C(1)—C(2)—C(3)	109·8 (10)
P(1)—C(4)—C(5)	128·2 (16)	C(4)—C(5)—C(6)	121·8 (24)
P(1)—C(7)—C(8)	126·6 (15)	C(7)—C(8)—C(9)	110·2 (18)
C(4)—P(1)—C(1)	104·3 (5)	C(7)—P(1)—C(1)	108·0 (6)
C(7)—P(1)—C(4)	109·3 (8)		

Pd—N bond distance indicates that such an interaction is not present and the observed twist is essentially due to the molecular packing.

As usual, the phenylhydrazone group adopts a *syn* configuration (Willey & Drew, 1983), the bulkier phenyl and pyridine groups lying on opposite sides of the C—N double bond. The pyridine and phenyl rings make angles of 11·5 (5) and 6·6 (5)°, respectively, with the C=N—N fragment. In a series of investigations (Drew, Vickery & Willey, 1984) it was established that the dimensions of the C(sp^2)—NH—N=C fragment are very dependent upon the presence or absence of an intramolecular hydrogen bond; these distances for compounds with intramolecular hydrogen bonding are: 1·40 Å for C(sp^2)—NH, 1·31 Å for HN—N and 1·31 Å for N=C, and 1·36, 1·39 and 1·28 Å, respectively, for compounds without hydrogen bonding. In the present compound, where an intermolecular hydrogen bond is present, the corresponding bond lengths

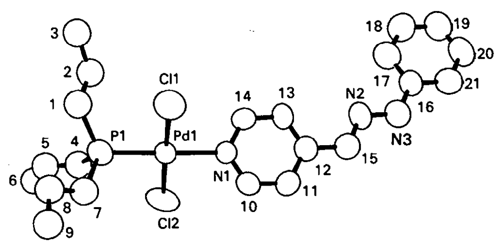

Fig. 1. *ORTEP* drawing (Johnson, 1976) of the molecular structure of the title compound showing the atom labelling. Thermal ellipsoids are drawn at the 40% probability level.

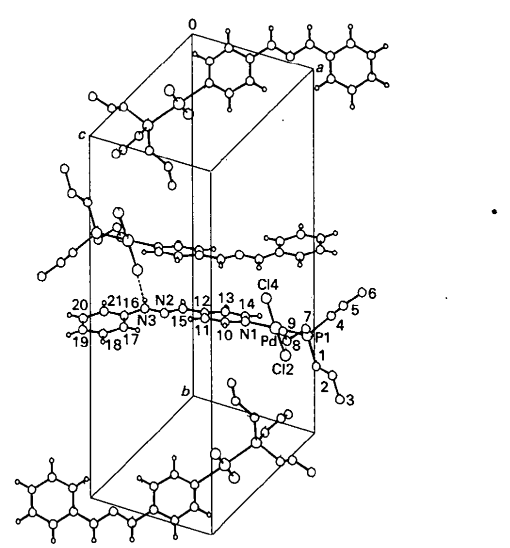

Fig. 2. *PLUTO* drawing (Motherwell & Clegg, 1978) of the molecular packing.

are 1·394 (10) Å for N(3)—C(16), 1·328 (8) Å for N(2)—N(3) and 1·274 (9) Å for C(15)=N(2).

The molecular packing is mainly determined by an intermolecular hydrogen bond between Cl(1) $(1-x, -y, -z)$ and N(3) [3·409 (8) Å] of two molecules related by an inversion centre. There is also a contact (< 3·65 Å) between the C=N—N fragment and the pyridine ring $(1-x, -y, -z)$ [C(12)···C(15) 3·49 (1), C(13)···C(15) 3·53 (1), C(10)···N(2) 3·62 (1) Å] and such an arrangement, depicted in Fig. 2, may be responsible for the pyridine twist.

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Structure of Di-μ-methoxo-bis[dichlorodimethoxomolybdenum(V)]*

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Abstract. $[Mo_2Cl_4(OCH_3)_6]$, $M_r = 519.9$, monoclinic, $P2_1/n$, $a = 12.029$ (2), $b = 9.615$ (2), $c = 7.240$ (1) Å, $\beta = 95.60$ (1)°, $V = 833.38$ Å^3, $Z = 2$, $D_x = 2.074$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 17.77$ mm^{-1}, $F(000) = 508$, final $R = 0.056$ for 1118 reflections. The dimeric molecules, located on crystallographic inversion centres, consist of two edge-sharing octahedra. Each Mo atom is coordinated by a pair of *cis* terminal Cl atoms [2·391 (2) and 2·393 (2) Å], a pair of *trans* terminal methoxo O atoms [1·811 (5) and 1·801 (6) Å], and a pair of *cis* bridging methoxo O atoms [2·027 (5) and 2·029 (5) Å]. The Mo–Mo distance is 2·733 (1) Å.

Introduction. The crystal structure analysis of the title complex forms part of our research on Mo^V complexes with O- and N-donor ligands. The attempts to prepare such complexes with different ligands, *e.g.* methionine or morpholine in methanol as solvent, always resulted in the binuclear chloro-(methoxo) complex $Mo_2Cl_4(OCH_3)_6$ (Kamenar, Korpar-Čolig, Penavić & Cindrić, 1988*b*). It was of interest to determine the crystal structure of this complex in order to establish the type of bridging between the two Mo atoms, the strength of the Mo^V–Mo^V interaction, as well as to compare our findings with earlier works (Cotton, 1987).

Experimental. In the attempt to prepare the complex of Mo^V with methionine, by mixing $MoCl_5$ with methionine in dry methanol, the title compound was isolated in low yield as a first-reaction product. Deep-red-brown crystals sensitive to air and moisture were separated by filtration in a dry box under dry nitrogen. Alternatively, the same complex can be obtained by the procedure given by Funk, Hesselbarth & Schmeil (1962).

* Reported at the 11th European Crystallographic Meeting, Vienna 1988, but with the space group assigned as $P2_1/c$ (Kamenar, Korpar-Čolig, Penavić & Cindrić, 1988*a*).

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