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Structure of Dichlorobis(*N*-isopropylideneaniline)palladium(II)

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Abstract. $[PdCl_2(C_9H_{11}N)_2]$, $M_r = 443{\cdot}7$, monoclinic, $P2_1/n$, $a = 8{\cdot}5651\,(3)$, $b = 12{\cdot}0982\,(5)$, $c = 9{\cdot}2466\,(4)$ Å, $\beta = 93{\cdot}916\,(5)°$, $V = 955{\cdot}92$ Å^3, $Z = 2$, $D_x = 1{\cdot}541$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}24$ mm^{-1}, $F(000) = 448$, $T = 293$ K, $R = 0{\cdot}023$ for 1421 unique observed reflections. Pd, on an inversion centre, is square-planar coordinated by two *trans*-Cl and two *trans*-imine ligands: Pd—Cl = 2·303 (1), Pd—N = 2·024 (2) Å, N—Pd—Cl = 89·9 (1)°, N=C = 1·273 (4) Å. The ligand plane [$CN{=}CC_2$, r.m.s. $\Delta = 0{\cdot}014\,(2)$ Å] is essentially perpendicular to the coordination plane [$PdCl_2N_2$, exactly planar; dihedral angle between planes =89·9 (1)°] and to the plane of the phenyl substituent [C_6, r.m.s. $\Delta = 0{\cdot}006\,(2)$ Å; dihedral angle = 91·4 (1)°].

Experimental. K_2PdCl_4 (1·25 mmol) reacts with diphenylformamidine (1·25 mmol) in a 2:5 methanol/water mixture (70 ml) at the reflux temperature to produce a yellow powder identified by elemental analysis as $[(C_6H_4NHCHNC_6H_5)PdCl]_n$. Attempts to grow crystals of this very sparingly soluble complex from acetone over three months under a nitrogen atmosphere at room temperature produced yellow crystals of the present complex in small yield; palladium metal, free *N*-(isopropylidene)aniline and N-phenylformamide were also formed. The reaction may be rationalized in terms of initial hydrolysis of the formamidino group by traces of water in the acetone, causing cleavage of the C=N bond to form coordinated formamide and aniline ligands. The latter would undergo a standard condensation reaction with acetone to form *N*-(isopropylidine)aniline.

Crystal size 0·2 × 0·25 × 0·4 mm, Siemens AED2 diffractometer, cell parameters from 2θ values of 32 reflections ($20 < 2\theta < 25°$). Intensity measurements in ω/θ scan mode, scan width 0·51° below α_1 to 0·51° above α_2, scan time = 14–56 s, $2\theta_{\max} = 50°$, h −10→0, k 0→14, l −10→10, no significant variation in three standard reflections, semi-empirical absorption correction, transmission 0·577–0·615, extinction insignificant. 1680 reflections measured, all unique, 1421 with $F < 4\sigma(F)$. Patterson synthesis confirms Pd on centre of symmetry and locates Cl, other atoms from difference syntheses, blocked-cascade refinement on F, $w = 1/\sigma^2(F)$, anisotropic thermal parameters for all non-H atoms, H atoms constrained [C—H = 0·96 Å, H—C—H = 109·5° in rigid methyl groups, aromatic H on ring angle external bisectors, $U(\mathrm{H}) = 1{\cdot}2U_{\mathrm{eq}}(\mathrm{C})$]. 113 parameters, $R = 0{\cdot}023$, $wR = 0{\cdot}026$, max. $\Delta/\sigma = 0{\cdot}056$, mean = 0·016, slope of normal probability plot = 1·78, max $\Delta\rho = +0{\cdot}37$, min. = $-0{\cdot}23$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* programs (Sheldrick, 1985).

Fig. 1 gives a view of the molecule with atomic numbering scheme. Table 1 gives the atomic co-

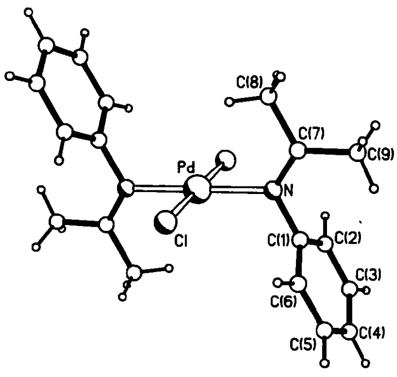

Fig. 1. Molecular structure, showing the labelling scheme.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$)

$U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq}
Pd	5000	5000	5000	353 (1)
Cl	6334 (1)	6546 (1)	4313 (1)	644 (3)
N	3000 (2)	5614 (2)	4020 (2)	387 (7)
C(1)	2726 (3)	5285 (2)	2528 (3)	415 (9)
C(2)	1861 (3)	4350 (3)	2187 (3)	579 (11)
C(3)	1603 (4)	4036 (3)	752 (4)	664 (13)
C(4)	2235 (4)	4625 (3)	−320 (4)	708 (13)
C(5)	3083 (6)	5540 (4)	34 (4)	973 (18)
C(6)	3358 (5)	5874 (3)	1461 (3)	775 (14)
C(7)	2032 (3)	6230 (2)	4626 (3)	469 (9)
C(8)	2321 (4)	6519 (3)	6196 (3)	624 (12)
C(9)	590 (4)	6705 (3)	3850 (4)	712 (13)

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

Pd—Cl	2·303 (1)	Pd—N	2·024 (2)
N—C(1)	1·439 (4)	N—C(7)	1·273 (4)
C(1)—C(2)	1·377 (4)	C(1)—C(6)	1·360 (5)
C(2)—C(3)	1·384 (5)	C(3)—C(4)	1·363 (5)
C(4)—C(5)	1·351 (6)	C(5)—C(6)	1·385 (5)
C(7)—C(8)	1·497 (4)	C(7)—C(9)	1·499 (4)
Cl—Pd—N	89·9 (1)	Pd—N—C(1)	113·9 (2)
Pd—N—C(7)	125·1 (2)	C(1)—N—C(7)	121·1 (2)
N—C(1)—C(2)	119·8 (3)	N—C(1)—C(6)	120·2 (3)
C(2)—C(1)—C(6)	119·9 (3)	C(1)—C(2)—C(3)	119·6 (3)
C(2)—C(3)—C(4)	120·5 (3)	C(3)—C(4)—C(5)	119·1 (3)
C(4)—C(5)—C(6)	121·6 (4)	C(1)—C(6)—C(5)	119·2 (3)
N—C(7)—C(8)	119·6 (3)	N—C(7)—C(9)	123·5 (3)
C(8)—C(7)—C(9)	116·8 (3)		

ordinates and U_{eq} values for the non-hydrogen atoms, and Table 2 lists selected bond lengths and angles.*

Related literature. Similar square-planar Pd complexes with related ligands have been reported by Anderson & Einstein (1978), Einstein & Field (1979), Keijsper, van der Poel, Polm, van Koten, Vrieze, Seignette, Varenhorst & Stam (1983) and, most closely related, Kuz'mina & Struchkov (1979).

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* 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 43733 (9 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 (Benzylideneacetone)tricarbonyliron (Monoclinic Form)

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Abstract. Tricarbonyl(η^4-1-phenyl-1-buten-3-one)iron, $[Fe(CO)_3(C_{10}H_{10}O)]$, $M_r = 286·05$, monoclinic, $P2_1/a$, $a = 10·4439$ (16), $b = 12·7485$ (23), $c = 10·6642$ (16) Å, $\beta = 116·330$ (13)°, $V = 1272·6$ Å^3, $Z = 4$, $D_x = 1·493$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 11·84$ cm^{-1}, $F(000) = 584$, room temperature, final $R = 0·0255$ for 1869 independent observed reflections. The molecular structure is essentially the same as that in the orthorhombic form [Hubener, Kuhr & Weiss (1981). *Cryst. Struct. Commun.* **10**, 1451–1455]. The phenyl group makes an angle of 24 (1)° to the rest of the ligand.

Experimental. Crystallization by cooling a solution (hexane:CH_2Cl_2, 10:1) gave needles (*Pbca*) and plates; yellow plate 0·3 × 0·3 × 0·1 mm selected; D_m not measured. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from refinement of 25 reflections in range $10·7 < \theta < 13·7°$; 4739 reflection intensities measured by $\omega/2\theta$ scan, scan width 0·8° +0·35° tanθ, θ 1→25°, h −12→12, k 0→15, l 0→12 and some equivalents. Empirical absorption correction 0·906–1·167; correction for decay of two standard reflections by about 15%. 2241 unique reflections, $R_{int} = 0·016$, 1869 with

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