Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Ni(1)—S(1)	2·224 (4)	C(3)—C(31)	1·479 (22)
Ni(1)—S(2)	2·219 (4)	C(3)—C(32)	1·453 (23)
Ni(1)—S(3)	2·124 (4)	C(1S)—C(2S)	1·359 (21)
Ni(1)—S(4)	2·111 (4)	C(1S)—C(4)	1·61 (3)
S(1)—C(1)	1·711 (14)	C(2S)—C(3S)	1·385 (21)
S(2)—C(1)	1·713 (14)	C(3S)—C(5)	1·502 (20)
S(3)—C(3S)	1·654 (15)	C(4)—F(1)	1·29 (3)
S(4)—C(1S)	1·696 (15)	C(4)—F(2)	1·35 (3)
C(1)—N(1)	1·337 (18)	C(4)—F(3)	1·35 (3)
N(1)—C(2)	1·508 (22)	C(4)—F(1′)	1·30 (3)
N(1)—C(3)	1·463 (21)	C(4)—F(2′)	1·28 (4)
C(2)—C(21)	1·42 (3)	C(4)—F(3′)	1·29 (4)
C(2)—C(22)	1·417 (25)		

S(1)—Ni(1)—S(2)	77·52 (14)	C(31)—C(3)—C(32)	116·3 (14)
S(1)—Ni(1)—S(3)	168·98 (15)	S(4)—C(1S)—C(2S)	130·1 (12)
S(1)—Ni(1)—S(4)	89·31 (14)	S(4)—C(1S)—C(4)	111·4 (11)
S(2)—Ni(1)—S(3)	91·46 (14)	C(2S)—C(1S)—C(4)	118·6 (14)
S(2)—Ni(1)—S(4)	166·78 (15)	C(1S)—C(2S)—C(3S)	130·1 (14)
S(3)—Ni(1)—S(4)	101·71 (15)	S(3)—C(3S)—C(2S)	127·3 (12)
Ni(1)—S(1)—C(1)	86·9 (5)	S(3)—C(3S)—C(5)	117·4 (10)
Ni(1)—S(2)—C(1)	87·0 (5)	C(2S)—C(3S)—C(5)	115·3 (13)
Ni(1)—S(3)—C(3S)	116·8 (5)	C(1S)—C(4)—F(1)	113·5 (16)
Ni(1)—S(4)—C(1S)	114·0 (5)	C(1S)—C(4)—F(2)	109·4 (15)
S(1)—C(1)—S(2)	108·6 (7)	C(1S)—C(4)—F(3)	105·5 (16)
S(1)—C(1)—N(1)	122·9 (10)	C(1S)—C(4)—F(1′)	113·5 (18)
S(2)—C(1)—N(1)	128·4 (11)	C(1S)—C(4)—F(2′)	108·0 (21)
C(1)—N(1)—C(2)	120·7 (13)	C(1S)—C(4)—F(3′)	101·9 (19)
C(1)—N(1)—C(3)	121·2 (13)	F(1)—C(4)—F(2)	111·0 (18)
C(2)—N(1)—C(3)	118·0 (13)	F(1)—C(4)—F(3)	110·2 (18)
N(1)—C(2)—C(21)	111·0 (15)	F(2)—C(4)—F(3)	107·0 (18)
N(1)—C(2)—C(22)	112·6 (15)	F(1′)—C(4)—F(2′)	103·6 (24)
C(21)—C(2)—C(22)	119·4 (16)	F(1′)—C(4)—F(3′)	111·4 (23)
N(1)—C(3)—C(31)	115·6 (13)	F(2′)—C(4)—F(3′)	118·8 (26)
N(1)—C(3)—C(32)	116·1 (14)		

$= 0{\cdot}0831$, $0{\cdot}0662$, $S = 1{\cdot}150$ for 188 parameters, $(\Delta/\sigma)_{max}$ in final cycle 0·05, max. and min. residues in final difference Fourier synthesis 0·53, $-0{\cdot}43$ e Å^{-3} respectively. The weighting scheme, $w^{-1} = \sigma^2(F) + 0{\cdot}000225F^2$, gave satisfactory agreement analysis. Scattering factors were inlaid (Sheldrick, 1976) except for Ni (Cromer & Mann, 1968).

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, while selected bond lengths and angles appear in Table 2.* The atom-numbering scheme for the molecule is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

We thank the SERC for support.

* Lists of structure factors, torsion angles, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51909 (11 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 (*tert*-Butylisonitrile)(chloro)(1-methyl-2-phenylimidazolato-$C^{2'},N^3$)-palladium(II) Dichloromethane Solvate at 213 K

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Abstract. $[Pd(C_5H_9N)(C_{10}H_9N_2)Cl].CH_2Cl_2$, $M_r = 934{\cdot}23$, monoclinic, $P2_1/c$, $a = 13{\cdot}689$ (3), $b = 24{\cdot}432$ (2), $c = 12{\cdot}111$ (2) Å, $\beta = 107{\cdot}57$ (3)°, $V = 3861{\cdot}6$ Å^3, $Z = 8$, $D_x = 1{\cdot}607$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 13{\cdot}6$ cm^{-1}, $F(000) = 1872$, $T = 213$ K. The final R value is 0·034 for 3177 significant

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Table 1. *Data-collection and structure-refinement parameters*

Crystal shape	Prism
Diffractometer used	CAD-4, Enraf–Nonius
Method of intensity measurement	$\theta/2\theta$
No. and θ range (°) of reflections for lattice parameters	25; 10–24
Absorption coeficient μ (cm^{-1})	13·690
Method used for absorption correction	*DIFABS* (Walker & Stuart, 1983)
Minimum absorption correction	0·729
Maximum absorption correction	1·376
Average absorption correction	1·003
Maximum value of $(\sin\theta)/\lambda$ reached in intensity measurement ($Å^{-1}$)	0·53
Range of h, k and l	$-14\rightarrow14$, $0\rightarrow25$, $0\rightarrow12$
Standard reflections	$6\bar{3}3$, $5\bar{1}2$
Interval, standard reflections measured	2 h, no intensity variation
Total No. of reflections measured; θ range (°)	4798; 22 (947 unobserved reflections, 141 systematic absences included)
No. of observed reflections	3173 with $I > 3\sigma(I)$
Method used to solve structure	Patterson
Use of F or F^2 in LS refinement	F
Method of locating H atoms	H(C) calculated in idealized positions with d(C—H) = 0·95 Å, included in structure-factor calculation
Weighting scheme	$1/\sigma^2$
Parameters refined	416
Value of R	0·034
Value of wR	0·038
Ratio of max. LS shift to e.s.d. (Δ/σ)	0·003
Max. height in final ΔF map (e $Å^{-3}$)	0·427
Error in an observation of unit weight	3·304
Secondary-extinction coefficient	9·45 (1) × 10^{-9} (Zachariasen, 1963)
Sources of atomic scattering factors	*International Tables for X-ray Crystallography* (1974)
Computer used	MicroVAX II
Programs used	*VAXSDP*, version 3.0 (1986) (Frenz, 1978)

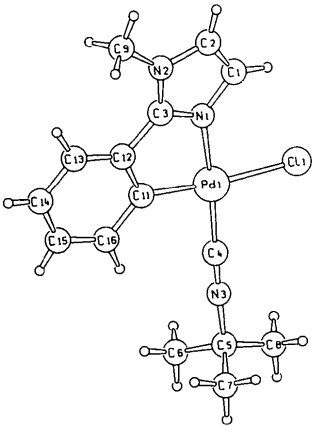

Fig. 1. View of molecule *A* illustrating the atom labeling.

[$I > 3\sigma(I)$] reflections. The Pd atom has a square-planar environment and is coordinated to the N and *ortho*-phenyl C atoms of the chelating 2-phenylimidazole group, the isonitrile C atom, and the Cl atom. In the resulting five-membered metallacycle, the average N—Pd—C bond angle is 81·1 (3)°.

Table 2. *Positional parameters and equivalent isotropic thermal parameters U_{eq} (Å^2) with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Molecule *A*				
Pd1	0·96956 (4)	0·13146 (2)	0·25720 (4)	0·0264 (3)
Cl1	0·8376 (2)	0·1564 (1)	0·0862 (2)	0·046 (1)
N1	0·8702 (4)	0·0942 (3)	0·3279 (4)	0·027 (4)
N2	0·8361 (4)	0·0586 (2)	0·4774 (5)	0·027 (3)
N3	1·1316 (5)	0·1925 (3)	0·1752 (5)	0·036 (4)
C1	0·7655 (6)	0·0880 (3)	0·2989 (6)	0·032 (5)
C2	0·7441 (6)	0·0656 (3)	0·3911 (6)	0·033 (5)
C3	0·9100 (5)	0·0767 (3)	0·4344 (5)	0·024 (4)
C4	1·0725 (5)	0·1695 (3)	0·2065 (5)	0·031 (5)
C5	1·2067 (6)	0·2220 (3)	0·1332 (6)	0·036 (5)
C6	1·2942 (7)	0·1833 (4)	0·1425 (9)	0·076 (7)
C7	1·2448 (8)	0·2702 (4)	0·2128 (8)	0·069 (6)
C8	1·1535 (7)	0·2406 (5)	0·0119 (7)	0·066 (8)
C9	0·8469 (6)	0·0348 (3)	0·5912 (6)	0·039 (5)
C11	1·0683 (5)	0·1083 (3)	0·4084 (5)	0·024 (4)
C12	1·0212 (5)	0·0818 (3)	0·4841 (6)	0·024 (4)
C13	1·0795 (6)	0·0626 (3)	0·5921 (6)	0·029 (4)
C14	1·1858 (6)	0·0687 (3)	0·6282 (6)	0·037 (5)
C15	1·2308 (6)	0·0949 (3)	0·5553 (6)	0·035 (5)
C16	1·1746 (6)	0·1143 (3)	0·4477 (6)	0·031 (5)
Molecule *B*				
Pd2	0·59162 (4)	0·33349 (2)	0·09713 (4)	0·0241 (3)
Cl2	0·4161 (1)	0·31711 (9)	−0·0083 (2)	0·040 (1)
N21	0·6411 (4)	0·3102 (2)	−0·0359 (4)	0·024 (3)
N22	0·7661 (4)	0·2840 (2)	−0·1010 (4)	0·026 (3)
N23	0·5403 (5)	0·3659 (3)	0·3191 (5)	0·031 (4)
C21	0·6002 (6)	0·2929 (3)	−0·1481 (6)	0·033 (5)
C22	0·6770 (6)	0·2764 (3)	−0·1898 (6)	0·034 (5)
C23	0·7427 (5)	0·3040 (3)	−0·0096 (5)	0·024 (4)
C24	0·5600 (5)	0·3544 (3)	0·2357 (6)	0·028 (4)
C25	0·5175 (6)	0·3811 (3)	0·4248 (5)	0·032 (5)
C26	0·5874 (6)	0·4292 (4)	0·4786 (6)	0·042 (5)
C27	0·5401 (6)	0·3321 (4)	0·5044 (6)	0·044 (5)
C28	0·4061 (7)	0·3985 (5)	0·3919 (7)	0·061 (8)
C29	0·8682 (6)	0·2680 (3)	−0·1073 (6)	0·039 (5)
C211	0·7449 (5)	0·3422 (3)	0·1750 (5)	0·025 (4)
C212	0·8028 (5)	0·3220 (3)	0·1057 (5)	0·021 (4)
C213	0·9108 (6)	0·3227 (3)	0·1469 (6)	0·029 (5)
C214	0·9585 (6)	0·3444 (3)	0·2568 (6)	0·038 (5)
C215	0·9023 (6)	0·3637 (3)	0·3244 (6)	0·035 (5)
C216	0·7950 (6)	0·3617 (3)	0·2840 (6)	0·033 (4)
Solvent				
C30	0·4115 (8)	0·0531 (4)	0·3415 (8)	0·081 (7)
Cl31	0·4688 (2)	0·0716 (1)	0·2367 (2)	0·073 (2)
Cl32	0·4096 (2)	−0·0180 (1)	0·3563 (3)	0·094 (2)
C40	0·1601 (9)	0·5062 (5)	0·533 (1)	0·097 (8)
Cl41	0·1179 (3)	0·4688 (1)	0·6318 (3)	0·106 (2)
Cl42	0·1700 (3)	0·4670 (2)	0·4199 (3)	0·133 (3)

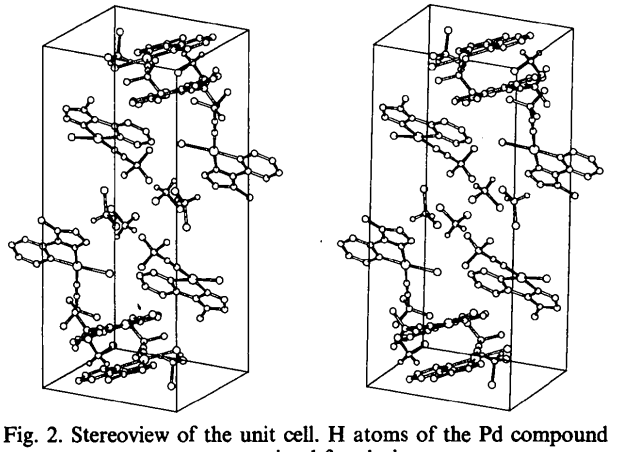

Fig. 2. Stereoview of the unit cell. H atoms of the Pd compound are omitted for clarity.

Table 3. *Selected distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Molecule *A*		Molecule *B*	
Pd1—Cl1	2·380 (2)	Pd2—Cl2	2·389 (2)
Pd1—N1	2·028 (6)	Pd2—N21	2·009 (7)
Pd1—C4	1·937 (9)	Pd2—C24	1·924 (8)
Pd1—C11	2·002 (6)	Pd2—C211	2·036 (6)
N1—C1	1·378 (9)	N21—C21	1·370 (8)
N1—C3	1·311 (8)	N21—C23	1·339 (9)
N2—C2	1·384 (8)	N22—C22	1·373 (8)
N2—C3	1·34 (2)	N22—C23	1·33 (1)
N2—C9	1·461 (9)	N22—C29	1·48 (2)
N3—C4	1·14 (2)	N23—C24	1·16 (1)
N3—C5	1·47 (2)	N23—C25	1·45 (1)
C1—C2	1·35 (1)	C21—C22	1·36 (1)
C3—C12	1·465 (9)	C23—C212	1·458 (8)
Cl1—Pd1—N1	93·0 (1)	C12—Pd2—N21	93·0 (2)
Cl1—Pd1—C4	92·1 (2)	C12—Pd2—C24	93·2 (3)
Cl1—Pd1—C11	173·7 (2)	C12—Pd2—C211	174·0 (2)
N1—Pd1—C4	173·8 (2)	N21—Pd2—C24	173·4 (2)
N1—Pd1—C11	80·8 (3)	N21—Pd2—C211	81·4 (2)
C4—Pd1—C11	94·1 (3)	C24—Pd2—C211	92·3 (3)
Pd1—N1—C1	136·1 (4)	Pd2—N21—C21	138·3 (5)
Pd1—N1—C3	115·4 (5)	Pd2—N21—C23	114·8 (4)
C1—N1—C3	107·9 (6)	C21—N21—C23	106·6 (7)
C2—N2—C3	106·6 (6)	C22—N22—C23	108·7 (7)
C2—N2—C9	124·8 (6)	C22—N22—C29	123·7 (6)
C3—N2—C9	128·5 (5)	C23—N22—C29	127·5 (5)
C4—N3—C5	179·2 (6)	C24—N23—C25	178·9 (7)
N1—C1—C2	107·6 (6)	N21—C21—C22	109·2 (6)
N2—C2—C1	107·4 (7)	N22—C22—C21	105·8 (6)
N1—C3—N2	110·6 (6)	N21—C23—N22	109·7 (5)
N1—C3—C12	116·7 (7)	N21—C23—C212	116·0 (6)
N2—C3—C12	132·7 (6)	N22—C23—C212	134·2 (7)
Pd1—C4—N3	178·8 (6)	Pd2—C24—N23	178·7 (7)
Solvent			
Cl31—C30	1·75 (1)	Cl41—C40	1·73 (1)
Cl32—C30	1·75 (1)	Cl42—C40	1·71 (1)

Experimental. A colorless single crystal of approximate dimensions 0·10 × 0·15 × 0·15 mm was sealed in a capillary. Because the crystals decompose at room temperature all X-ray investigations were performed at 213 K. The systematic absences pointed to space group $P2_1/c$, and this space group was used for all further calculations. In the final full-matrix least-squares refinement all non-H atoms were assigned anisotropic thermal parameters. More details of the intensity data collection, structure solution and refinement are listed in Table 1. Final atomic coordinates are given in Table 2, selected distances and angles in Table 3.* The two independent molecules *A* and *B* in the unit cell have the same structural features. A *SCHAKAL* (Keller, 1988) plot of molecule *A* is shown in Fig. 1, a stereoview of the unit cell in Fig. 2.

Related literature. The structure determination is part of our studies of cyclometallated complexes derived from Schiff-base ligands (Gayoso, Alonso, Vila, Rivero, Hiller & Strähle, 1988; Hiller, Castiñeiras, Vila, Suarez, Pereira & Gayoso, 1986; Pereira, Vila, Gayoso, Gayoso, Hiller & Strähle, 1988; Suarez, Vila, Gayoso, Gayoso, Hiller, Castiñeiras & Strähle, 1986).

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* Lists of structure factors, H-atom positions, bond distances and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51883 (23 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 3β-Hydroxy-16α-methyl-5-pregnen-20-one

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Abstract. $C_{22}H_{34}O_2$, $M_r = 330{\cdot}52$, triclinic, $P\bar{1}$, $a = 6{\cdot}3650\ (5)$, $b = 12{\cdot}900\ (1)$, $c = 6{\cdot}3365\ (5)$ Å, $\alpha = 96{\cdot}488\ (9)$, $\beta = 107{\cdot}361\ (9)$, $\gamma = 98{\cdot}843\ (8)°$, $V = 483{\cdot}66$ Å^3, $Z = 1$, $D_x = 1{\cdot}135$ g cm^3, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}655$ cm^{-1}, $F(000) = 182$, $T = 298$ K, $R = 0{\cdot}043$ for 2587 unique observed reflec-

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