I(1)—Cd(1)—I(2)	109.4 (1)	I(1)—Cd(1)—I(3)	109.9 (1)
I(2)—Cd(1)—I(3)	110.2 (1)	I(1)—Cd(1)—I(4)	106.3 (1)
I(2)—Cd(1)—I(4)	108.1 (1)	I(3)—Cd(1)—I(4)	112.8 (1)
C(1)—N(1)—C(2)	124 (2)	C(5)—N(2)—C(6)	124 (2)
N(1)—C(1)—C(3)	117 (2)	N(1)—C(1)—C(2^{i})	117 (2)
C(3)—C(1)—C(2^{i})	126 (2)	N(1)—C(2)—C(4)	117 (2)
N(1)—C(2)—C(1^{i})	119 (2)	C(4)—C(2)—C(1^{i})	124 (2)
N(2)—C(5)—C(7)	120 (2)	N(2)—C(5)—C(6^{ii})	118 (2)
C(7)—C(5)—C(6^{ii})	122 (2)	N(2)—C(6)—C(8)	117 (2)
N(2)—C(6)—C(5^{ii})	118 (2)	C(8)—C(6)—C(5^{ii})	125 (2)
N(1)···O(1)	2.684 (7)	O(1)···O(2)	2.834 (7)
O(2)···O(3)	2.833 (7)	N(2)···O(3)	2.676 (7)

Symmetry codes: (i) $2 - x, 1 - y, 1 - z$; (ii) $-x, 1 - y, -z$.

The structures were solved by direct methods and refined by full-matrix least squares. Corrections were applied for Lorentz and polarization effects, and real and imaginary anomalous dispersion (Cromer, 1974). Structure solution, refinement and the calculation of derived results were performed by using *SHELXTL* (Sheldrick, 1985). All non-H atoms were refined anisotropically except for the C atoms of (1); H atoms were not located. For (1), the asymmetric unit contains one diprotonated tetramethylpyrazinium cation and one CdI_4^{2-} anion, each occupying general positions in the cell. For (2), the asymmetric unit contains two unique cations, each situated about inversion centers [cation *A* at $(1\,\frac{1}{2}\,\frac{1}{2})$; cation *B* at $(0\,\frac{1}{2}\,0)$], three water molecules and a CdI_4^{2-} anion, all occupying general positions.

Lists of structure factors and anisotropic displacement parameters, along with a packing diagram of compound (2), have been deposited with the IUCr (Reference: HR1003). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Forms of (5,5-Dimethyl-4,7-diazadecane-1,10-diamine)nickel(II) Diperchlorate

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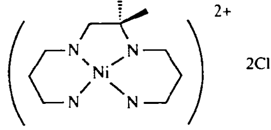
Abstract

The title Ni^{II} complex, $[Ni(C_{10}H_{26}N_4)](ClO_4)_2$, crystallizes in yellow and orange forms. In both crystals, the stereochemistry and conformation of the complex cations are identical. Small differences arise from packing effects, differences in the orientations of the perchlorate groups and hydrogen bonding. The Ni^{II} atom has square-planar coordination with the four tetraamine N atoms in equatorial positions. In each complex, the two chiral N-atom centres are in the (4*RS*,7*RS*) configuration. The tetraamine binds to the Ni^{II} atom in a relatively strain-free planar manner; the central six-membered ring exhibits a stable chair conformation and the two terminal five-membered rings adopt stable skew forms.

Comment

The crystal structures of open-chain tetraamine complexes containing secondary amine groups have been studied extensively. Anichini, Fabbrizzi, Paoletti & Clay (1977) pointed out that tetraaza complexes of Ni^{II} exist in equilibrium as a mixture of blue (paramagnetic, octahedral) and yellow (diamagnetic, square planar) forms. However, complexes containing methyl groups on the C(5) atom (see Fig. 1 for numbering) of the tetraamine ligand have received very little attention in the solid state. In this study, the structures of two different crystal forms, yellow and orange, of (5,5-dimethyl-4,7-diazadecane-1,10-diamine)nickel(II) diperchlorate, (I), have been determined.



(I)

In both the yellow and the orange crystals, the Ni^{II} complexes are four-coordinate and have identical stereochemistry and conformation. The geometry about the Ni^{II} ions is square planar with slight tetrahedral distortion. In both complexes, the four N atoms are planar to within ±0.10 Å. The Ni—N distances span a very narrow range and are close to the estimated value of 1.95 (4) Å for four-coordinate Ni^{II} complexes (Lu, Chung & Ashida, 1991). The amine H(N2) and H(N3) atoms are *trans* to the plane formed by the four N atoms. The two asymmetric N atoms are of the same *R* or *S* configuration. Both six-membered rings are in stable chair conformations and the central five-membered ring has a skew form. The torsion angle data (Table 2) show that the conformation of the ligand is very similar in both crystals. Small differences arise from packing effects, differences in the orientations of the perchlorate groups and hydrogen bonding. The shortest Ni···O distance in the yellow isomer is 2.795 (9) Å, while in the orange isomer it is 3.426 (6) Å (Fig. 1). In each crystal, one perchlorate anion is disordered.

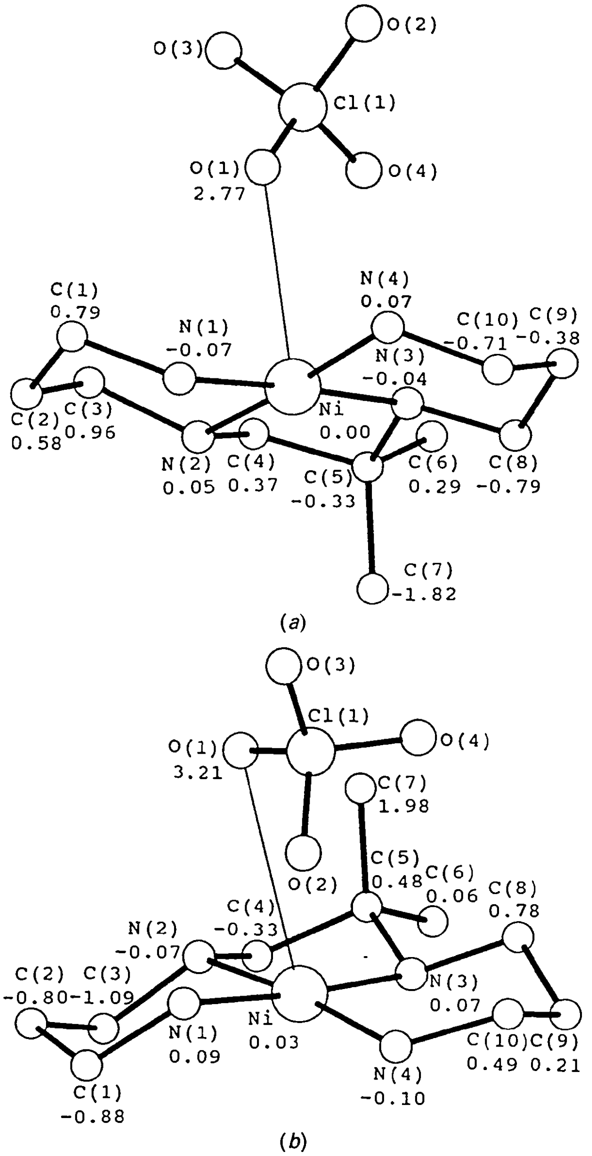


Fig. 1. Perspective views of the Ni^{II} complex, with the atom-numbering scheme, showing (*a*) the yellow isomer and (*b*) the orange isomer, excluding the H atoms and one of the perchlorate anions. The displacements of the atoms from the best plane formed by the four N atoms coordinated to the Ni^{II} ion are indicated; the e.s.d.'s are less than 0.01 Å. The shortest Ni···O distance is shown by a thin line.

Experimental

The ligand was synthesized according to the method of Lu, Shan, Chao & Chung (1987). A solution of $[Ni(ClO_4)_2].6H_2O$ (7.4 g, 0.02 mol) in methanol (100 ml) was added dropwise to a solution of 5,5-dimethyl-4,7-diazadecane-1,10-diamine (3.76 g, 0.02 mol) in ethanol (100 ml). The colour of the solution changed rapidly from light red to a brownish colour and the solution was stirred continuously for 3 h on a steam bath. A small amount of $Ni(OH)_2$ formed and was filtered off. The brownish filtrate was evaporated to dryness. Single crystals of the yellow and orange forms were obtained from the same aqueous solution by slow evaporation.

Yellow isomer

Crystal data

$[Ni(C_{10}H_{26}N_4)](ClO_4)_2$
$M_r = 459.95$
Monoclinic
$P2_1/n$
$a = 9.995$ (4) Å
$b = 14.143$ (3) Å
$c = 13.171$ (4) Å
$\beta = 91.96$ (3)°
$V = 1861$ (1) Å^3
$Z = 4$
$D_x = 1.642$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 6.53$–12.66°
$\mu = 1.38$ mm^{-1}
$T = 298$ (3) K
Parallelepiped
0.34 × 0.31 × 0.28 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
$T_{min} = 0.777$, $T_{max} = 0.999$
2591 measured reflections
2428 independent reflections

1869 observed reflections [$I \geq 1.5\sigma(I)$]
$R_{int} = 0.023$
$\theta_{max} = 22.5°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 15$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: ±1%

Refinement

Refinement on F
$R = 0.062$
$wR = 0.083$
$S = 1.57$
1869 reflections
253 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + 0.002F_o^2]$
$(\Delta/\sigma)_{max} = 0.083$
$\Delta\rho_{max} = 1.52$ (11) e Å^{-3}
$\Delta\rho_{min} = -0.47$ (11) e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Orange isomer

Crystal data

$[Ni(C_{10}H_{26}N_4)](ClO_4)_2$
M_r = 459.95
Monoclinic
$P2_1/n$
a = 9.605 (2) Å
b = 15.309 (6) Å
c = 13.010 (2) Å
β = 105.64 (2)°
V = 1842.2 (9) Å^3
Z = 4
D_x = 1.658 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 5.55–15.82°
μ = 1.39 mm^{-1}
T = 298 (3) K
Rhombic bipyramid
0.34 × 0.31 × 0.19 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical (North, Phillips & Mathews, 1968)
T_{min} = 0.881, T_{max} = 0.999
2531 measured reflections
2411 independent reflections

2073 observed reflections [$I \geq 1.5\sigma(I)$]
R_{int} = 0.018
θ_{max} = 22.5°
$h = -10 \rightarrow 9$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 60 min
intensity decay: ±1%

Refinement

Refinement on F
R = 0.049
wR = 0.066
S = 1.27
2073 reflections
244 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + 0.002F_o^2]$
$(\Delta/\sigma)_{max}$ = 0.063
$\Delta\rho_{max}$ = 0.74 (9) e Å^{-3}
$\Delta\rho_{min}$ = −0.35 (9) e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	Occupancy	x	y	z	B_{eq}
Yellow isomer					
Ni	1.00	0.24616 (7)	0.37640 (6)	0.23361 (6)	2.83 (4)
Cl(1)	1.00	0.27316 (22)	0.62562 (12)	0.13354 (16)	5.07 (9)
Cl(2)	1.00	0.32529 (20)	0.38175 (13)	0.61602 (4)	4.48 (8)
O(1)	1.00	0.2431 (12)	0.5733 (6)	0.2153 (7)	12.7 (7)
O(2)	1.00	0.3805 (8)	0.6848 (6)	0.1612 (8)	11.2 (6)
O(3)	1.00	0.1723 (12)	0.6897 (10)	0.1092 (12)	19.9 (11)
O(4)	1.00	0.3057 (15)	0.5684 (6)	0.0570 (7)	15.1 (8)
O(5)	1.00	0.1901 (8)	0.3702 (6)	0.6111 (9)	11.1 (6)
O(6)	0.85	0.3581 (11)	0.4387 (6)	0.7018 (6)	8.6 (5)
O(6′)	0.15	0.448 (6)	0.376 (5)	0.683 (3)	11 (4)
O(7)	0.50	0.3956 (19)	0.4044 (21)	0.5337 (15)	9.3 (14)
O(7′)	0.50	0.3244 (25)	0.4552 (18)	0.5415 (15)	10.6 (13)
O(8)	0.75	0.3724 (12)	0.2874 (7)	0.6453 (12)	10.5 (7)
O(8′)	0.25	0.373 (5)	0.3171 (23)	0.552 (3)	13.2 (27)
N(1)	1.00	0.1559 (7)	0.3903 (5)	0.3597 (5)	5.1 (3)
N(2)	1.00	0.0823 (6)	0.3549 (4)	0.1577 (4)	3.61 (25)
N(3)	1.00	0.3355 (5)	0.3547 (4)	0.1073 (4)	3.34 (22)
N(4)	1.00	0.4109 (6)	0.4063 (5)	0.3067 (4)	4.9 (3)
C(1)	1.00	0.0284 (9)	0.4444 (6)	0.3614 (6)	5.5 (4)
C(2)	1.00	−0.0730 (8)	0.4081 (7)	0.2860 (7)	5.5 (4)
C(3)	1.00	−0.0360 (7)	0.4176 (6)	0.1806 (6)	5.1 (4)
C(4)	1.00	0.1075 (8)	0.3586 (5)	0.0482 (5)	4.5 (3)
C(5)	1.00	0.2395 (8)	0.3128 (5)	0.0293 (5)	4.2 (3)
C(6)	1.00	0.2810 (9)	0.3390 (7)	−0.0786 (5)	5.7 (4)
C(7)	1.00	0.2279 (9)	0.2063 (6)	0.0433 (6)	5.7 (4)
C(8)	1.00	0.4664 (8)	0.3093 (6)	0.1103 (7)	5.8 (4)
C(9)	1.00	0.5681 (8)	0.3585 (7)	0.1769 (8)	6.1 (5)
C(10)	1.00	0.5326 (8)	0.3536 (7)	0.2867 (7)	5.9 (4)
Orange isomer					
Ni	1.00	0.65931 (6)	0.70320 (4)	0.13588 (4)	2.66 (3)
Cl(1)	1.00	0.34707 (16)	0.64811 (9)	−0.12278 (10)	4.84 (7)
Cl(2)	1.00	0.25741 (15)	0.90354 (9)	0.15593 (12)	5.01 (7)
O(1)	1.00	0.4243 (16)	0.5821 (13)	−0.0562 (14)	8.4 (13)
O(2)	1.00	0.3045 (7)	0.7124 (3)	−0.0594 (5)	8.6 (3)
O(3)	1.00	0.2299 (18)	0.6107 (14)	−0.1944 (5)	13.2 (4)
O(4)	1.00	0.4335 (19)	0.6896 (14)	−0.1803 (15)	11.3 (14)
O(5)	1.00	0.3282 (18)	0.8349 (14)	0.2215 (15)	10.8 (14)
O(6)	1.00	0.2652 (17)	0.9783 (13)	0.2211 (15)	10.1 (14)
O(7)	0.85	0.1127 (17)	0.8924 (16)	0.1093 (18)	12.4 (16)
O(7′)	0.15	0.160 (15)	0.857 (3)	0.216 (13)	9.9 (25)
O(8)	0.75	0.3361 (11)	0.9284 (6)	0.0828 (8)	10.9 (6)
O(8′)	0.25	0.2595 (24)	0.8564 (21)	0.0710 (15)	9.4 (16)
N(1)	1.00	0.4803 (4)	0.6769 (3)	0.1711 (4)	4.39 (20)
N(2)	1.00	0.7416 (4)	0.59513 (22)	0.1991 (3)	3.20 (17)
N(3)	1.00	0.8372 (4)	0.72179 (22)	0.0953 (3)	3.07 (16)
N(4)	1.00	0.5859 (5)	0.8164 (3)	0.0887 (4)	4.53 (21)
C(1)	1.00	0.4868 (6)	0.6476 (6)	0.2802 (4)	4.7 (3)
C(2)	1.00	0.5722 (6)	0.5663 (4)	0.3098 (5)	5.1 (3)
C(3)	1.00	0.7282 (6)	0.5772 (3)	0.3085 (4)	4.47 (24)
C(4)	1.00	0.8970 (5)	0.5942 (3)	0.2019 (4)	3.69 (22)
C(5)	1.00	0.9132 (5)	0.6342 (3)	0.0990 (4)	3.48 (21)
C(6)	1.00	1.0728 (5)	0.6470 (4)	0.1085 (5)	5.1 (3)
C(7)	1.00	0.8400 (6)	0.5799 (4)	0.0021 (5)	4.9 (3)
C(8)	1.00	0.8314 (7)	0.7719 (4)	−0.0017 (5)	5.1 (3)
C(9)	1.00	0.7727 (7)	0.8620 (3)	0.0051 (5)	5.6 (3)
C(10)	1.00	0.6180 (6)	0.8631 (4)	0.0004 (5)	4.8 (3)

Table 2. *Selected geometric parameters* (Å, °)

	Yellow isomer	Orange isomer
Ni—N(1)	1.926 (6)	1.938 (4)
Ni—N(2)	1.914 (6)	1.920 (4)
Ni—N(3)	1.940 (5)	1.940 (4)
Ni—N(4)	1.926 (6)	1.909 (4)
N(1)—C(1)	1.49 (1)	1.474 (7)
N(2)—C(3)	1.52 (1)	1.488 (6)
N(2)—C(4)	1.474 (9)	1.483 (6)
N(3)—C(5)	1.503 (8)	1.521 (6)
N(3)—C(8)	1.46 (1)	1.465 (6)
N(4)—C(10)	1.46 (1)	1.455 (7)
C(1)—C(2)	1.49 (1)	1.483 (8)
C(2)—C(3)	1.45 (1)	1.512 (8)
C(4)—C(5)	1.50 (1)	1.518 (7)
C(5)—C(6)	1.54 (1)	1.518 (7)
C(5)—C(7)	1.52 (1)	1.516 (7)
C(8)—C(9)	1.49 (1)	1.502 (8)
C(9)—C(10)	1.50 (1)	1.471 (8)
N(1)—Ni—N(2)	92.8 (3)	90.5 (2)
N(1)—Ni—N(3)	176.8 (3)	176.0 (2)
N(1)—Ni—N(4)	87.9 (3)	89.4 (2)
N(2)—Ni—N(3)	86.4 (2)	87.3 (2)
N(2)—Ni—N(4)	176.3 (3)	173.5 (2)
N(3)—Ni—N(4)	93.0 (2)	93.2 (2)
Ni—N(1)—C(1)	119.5 (8)	118.8 (3)
Ni—N(2)—C(3)	117.5 (5)	115.9 (3)
Ni—N(2)—C(4)	109.5 (4)	108.3 (3)
C(3)—N(2)—C(4)	109.5 (5)	108.7 (4)
Ni—N(3)—C(5)	110.3 (4)	108.2 (3)
Ni—N(3)—C(8)	119.0 (5)	118.7 (3)
C(5)—N(3)—C(8)	113.4 (6)	113.3 (4)
Ni—N(4)—C(10)	120.1 (5)	124.2 (3)
N(1)—C(1)—C(2)	112.2 (6)	112.5 (4)
C(1)—C(2)—C(3)	114.6 (7)	112.6 (4)
N(2)—C(3)—C(2)	111.2 (6)	111.9 (4)
N(2)—C(4)—C(5)	109.2 (6)	108.4 (4)
N(3)—C(5)—C(4)	105.1 (5)	102.8 (3)
N(3)—C(5)—C(6)	110.5 (6)	110.8 (4)
N(3)—C(5)—C(7)	110.9 (6)	110.2 (4)
C(4)—C(5)—C(6)	108.4 (7)	108.8 (4)
C(4)—C(5)—C(7)	109.6 (6)	112.2 (4)
C(6)—C(5)—C(7)	112.0 (6)	111.7 (4)

N(3)—C(8)—C(9)	113.8 (7)	111.0 (4)
C(8)—C(9)—C(10)	111.5 (7)	113.6 (5)
N(4)—C(10)—C(9)	112.1 (6)	112.8 (4)
N(1)—C(1)—C(2)—C(3)	−63.9 (6)	61.5 (4)
C(1)—C(2)—C(3)—N(2)	66.8 (6)	−65.0 (4)
C(4)—N(2)—C(3)—C(2)	173.9 (9)	−173.2 (5)
C(3)—N(2)—C(4)—C(5)	168.7 (8)	−166.0 (5)
N(2)—C(4)—C(5)—N(3)	−47.6 (4)	53.5 (3)
C(8)—N(3)—C(5)—C(4)	172.0 (8)	−176.9 (5)
C(5)—N(3)—C(8)—C(9)	172.0 (9)	−172.8 (5)
N(3)—C(8)—C(9)—C(10)	66.3 (7)	−69.6 (4)
C(8)—C(9)—C(10)—N(4)	−66.2 (7)	61.9 (4)

For both structures, *NRCVAX* (Gabe, Le Page, White & Lee, 1987) was used for all calculations.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and possible hydrogen bonds have been deposited with the IUCr (Reference: AS1127). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Di-μ-chloro-bis[chloro(tributylphosphine)palladium]

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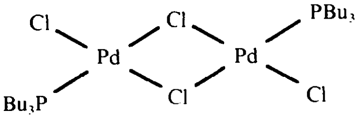
Abstract

The structure of *trans*-di-μ-chloro-bis[chloro(tributylphosphine)palladium], $[Pd_2Cl_4(C_{12}H_{27}P)_2]$, is planar with an inversion centre between the metal atoms. Bond lengths are typical of related species.

Comment

The title complex (I) was prepared (Hartley, 1970) as a precursor to palladium complexes of diaziridines. The structures of a range of related platinum complexes have been determined. In $[Pt_2(\mu\text{-}Cl)_2Cl_2(PEt_3)_2]$ (Blake, Gould, Marr, Rankin & Schröder, 1989) the terminal metal–chlorine bonds were 2.282 (3) Å in length, compared with 2.270 (1) Å here. Bonds between the Pt atoms and bridging halogens were 2.318 (3) and 2.431 (1) Å (the longer bond being for the halogen *trans* to the Cl atoms, as would be expected from the *trans* effect), compared with 2.314 (1) and 2.439 (1) Å here. Angles at the Pt and Cl atoms were also similar. In $[PdPt(\mu\text{-}Cl)_2Cl_2(PEt_3)_2]$ (Clark, Ferguson, Jain & Parvez, 1985) the two metals were disordered and the complex possessed a centre of symmetry. Related complexes which have been structurally characterized include the platinum derivative of di-*tert*-



(I)

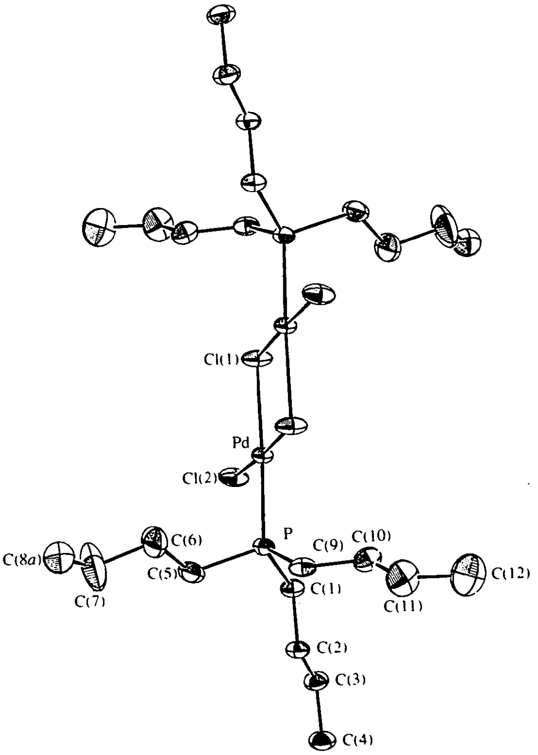


Fig. 1. Molecular structure and numbering scheme (*ORTEP*; Johnson, 1965). The displacement ellipsoids are drawn at the 50% probability level.