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The *Pbca* Polymorph of Dichloro(η^4-1,5-cyclooctadiene)palladium(II)

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Abstract

The Pd—C bond length range is 2.189 (2)–2.221 (2) Å, the Pd—Cl lengths are 2.3065 (6) and 2.3072 (6) Å, and the Cl—Pd—Cl angle is 91.84 (2)°. The cyclooctadiene ring is in the twist-boat conformation, with C═C bond lengths of 1.382 (3) and 1.385 (3) Å.

Comment

The published structure of (1,5-cod)$PdCl_2$ (cod = cyclooctadiene) has space group $P2_12_12_1$, and the crystals obtained from CH_2Cl_2 are described as both needles and octahedra (Rettig, Wing & Wiger, 1981) or only as 'un cristal allongé suivant l'axe *c*...' (the 6.876 Å axis) (Benchekroun, Herpin, Julia & Saussine, 1977). Our crystallizations from CH_2Cl_2 yielded needles, octahedra and prisms with rhombic cross sections. In order to clarify the uncertainty, we measured the unit-cell dimensions of all three morphological types. We found the needles to have space group $P2_12_12_1$, with dimensions $a = 6.874$ (2), $b = 12.293$ (1), $c = 10.972$ (2) Å at 296 K, in good agreement with the published structure. We found that the rhombic prism and the octahedron are both a second polymorph, of space group *Pbca*, identical to that described by Howells (1973). A rhombic prism yielded unit-cell dimensions $a = 11.8266$ (9), $b = 11.9875$ (12), $c = 13.0812$ (9) Å at 295 K. We have carried out a full structure determination using a flattened octahedron.

The determination reported here represents an increase in precision over the $P2_12_12_1$ form by a factor of 2–3, and the agreement between the two molecular structures is good, including the conformation of the cod ring. The root-mean-square deviation between the two sets of eight endocyclic torsion angles is 6.8°, and the largest individual deviation is only 10°, for C2—C3—C4—C5. All four Pd—C bond distances of the *Pbca* form agree with those of the $P2_12_12_1$ form within experimental error. While the $P2_12_12_1$ form exhibited a difference in Pd—Cl distances of marginal significance, our determination has equal Pd—Cl distances.

The Cl—Pd—Cl angle is slightly larger in the *Pbca* form; 91.84 (2) *versus* 90.31 (5)°. This angle has a value of 94.02 (5)° in dichloro(norbornadiene)palladium(II) (Baenziger, Richards & Doyle, 1965), 91.11 (3)° in dichloro[(1,2,5,6-η)-cyclooctatetraene]palladium(II) (Baenziger, Goebel, Berg & Doyle, 1978), 91.9° in dichloro(1,4-cyclooctadiene)palladium(II) (Rettig *et al.*, 1981) and 90.1° in dichloro(1,5-cyclononadiene)palladium(II) (Rettig *et al.*, 1981).

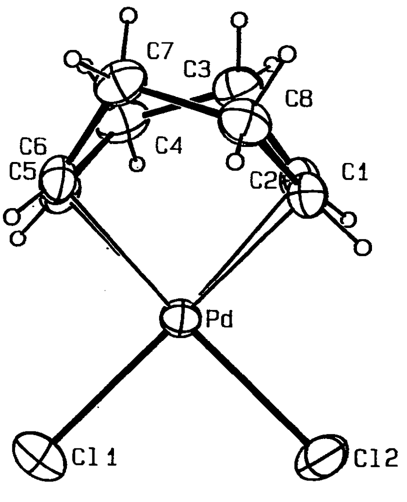

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the title compound, with thermal ellipsoids drawn at the 40% probability level.

Experimental

Crystal data

$[PdCl_2(C_8H_{12})]$
$M_r = 285.5$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Orthorhombic
Pbca
$a = 11.8271\ (4)$ Å
$b = 11.9869\ (4)$ Å
$c = 13.0821\ (5)$ Å
$V = 1854.7\ (2)$ Å^3
$Z = 8$
$D_x = 2.045$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 11\text{-}14°$
$\mu = 2.49$ mm^{-1}
$T = 295$ K
Flattened octahedron
0.33 × 0.32 × 0.18 mm
Amber

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: empirical
$T_{\min} = 0.933$, $T_{\max} = 0.998$
4544 measured reflections
4065 independent reflections

3105 observed reflections $[I > 3\sigma(I)]$
$\theta_{\max} = 35°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 21$
3 standard reflections frequency: 166.7 min intensity variation: <1%

Refinement

Refinement on F
Final $R = 0.022$
$wR = 0.029$
$S = 1.455$
3105 reflections
149 parameters
All H-atom parameters refined
$w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$
$(\Delta/\sigma)_{\max} = 0.04$

$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $g = 2.86\ (8) \times 10^{-7}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$B_{\rm eq}$
Pd	0.35479 (1)	0.60963 (1)	0.69883 (1)	2.029 (2)
Cl1	0.34127 (5)	0.54235 (5)	0.86362 (4)	3.76 (1)
Cl2	0.48296 (4)	0.47290 (4)	0.65451 (5)	3.58 (1)
C1	0.4148 (2)	0.7088 (2)	0.5692 (1)	2.54 (3)
C2	0.3195 (2)	0.6521 (2)	0.5371 (1)	2.58 (3)
C3	0.2024 (2)	0.7012 (2)	0.5266 (2)	3.06 (4)
C4	0.1298 (2)	0.6852 (2)	0.6222 (2)	3.13 (4)
C5	0.1932 (2)	0.6972 (2)	0.7201 (2)	2.80 (3)
C6	0.2789 (2)	0.7728 (2)	0.7406 (1)	2.71 (3)
C7	0.3192 (2)	0.8626 (2)	0.6690 (2)	2.85 (3)
C8	0.4216 (2)	0.8262 (2)	0.6071 (2)	2.84 (3)

Table 2. *Geometric parameters* (Å, °)

Pd—Cl1	2.3072 (6)	C1—C8	1.494 (3)
Pd—Cl2	2.3065 (6)	C2—C3	1.511 (3)
Pd—C1	2.189 (2)	C3—C4	1.528 (3)
Pd—C2	2.216 (2)	C4—C5	1.492 (3)
Pd—C5	2.198 (2)	C5—C6	1.385 (3)
Pd—C6	2.221 (2)	C6—C7	1.504 (3)
C1—C2	1.382 (3)	C7—C8	1.520 (3)
Cl1—Pd—Cl2	91.84 (2)	C4—C5—C6	126.6 (2)
C2—C1—C8	127.4 (2)	C5—C6—C7	125.5 (2)
C1—C2—C3	125.7 (2)	C6—C7—C8	112.3 (2)
C2—C3—C4	113.0 (2)	C1—C8—C7	113.8 (2)
C3—C4—C5	114.1 (2)		
C8—C1—C2—C3	4.8 (3)	C3—C4—C5—C6	37.9 (3)
C2—C1—C8—C7	35.3 (3)	C4—C5—C6—C7	4.0 (3)
C1—C2—C3—C4	−93.3 (3)	C5—C6—C7—C8	−95.4 (3)
C2—C3—C4—C5	38.0 (3)	C6—C7—C8—C1	41.1 (3)

Programs used include *MolEN* (Fair, 1990) and *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71258 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1058]

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Bis(μ-benzoato-κO:$\kappa O'$)-bis[(benzoato-$\kappa^2 O,O'$)(N,N-dimethylformamide-κO)-dioxouranium(VI)]

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Abstract

The action of dimethylformamide on diaqua(benzoato)chlorodioxouranium(VI), in the presence of β-cyclodextrin, yields the title compound. The crystal structure of this new complex reveals that the linear uranyl is equatorially surrounded by five O

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