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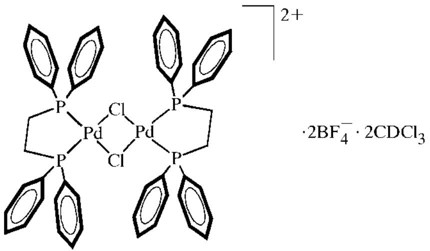
The title compound was formed upon slow evaporation of a solution of the solvated dicationic complex bis(acetonitrile)-bis[1,2-bis(diphenyl-phosphino)ethane]palladium(II) bis-(tetrafluoroborate) in deuterated chloroform. The dinucleur palladium complex forms triclinic crystals and there is an inversion center between the Pd atoms. Compared to the corresponding monomeric compound, the Cl—Pd—Cl angles decrease upon briding from 94.19 (7) to 86.96 (4)°.

Comment

Solvated dicationic palladium(II) complexes bearing bidentate phosphine ligands have been successfully used as catalysts for alternating co-polymerization of alkenes with carbon monoxide (Abu-Surrah, Eckert *et al.*, 1996; Abu-Surrah, Wursche *et al.*, 1996) and for polymerization of norbornene to poly(2,3-bicyclo[2.2.1]hept-2-ene) (Abu-Surrah & Rieger, 1998). For both polymerization processes, halogenated solvents such as chloroform and dichloromethane have been used as polymerization media.

In the course of our work on homo- and copolymerization reactions of alkenes with palladium(II) catalysts (Abu-Surrah & Rieger, 1999), we found that the title compound, (I), results from the catalyst precursor bis(acetonitrile)bis[1,2-bis(diphenylphosphino)ethane]palladium(II) bis(tetrafluoroborate), [Pd(dppe)(NCCH$_3$)$_2$](BF$_4$)$_2$ (Xu *et al.*, 1993), *via* recombination of the palladium cation with the chloride groups of the solvent (CDCl$_3$). The resulting compound is inactive towards polymerization. Such deactivation through dimer formation was also reported in metallocene catalysis (Hackmann & Rieger, 1997).



(I)

The dinuclear palladium complex, [Pd$_2$(dppe)$_2$Cl$_2$]-(BF$_4$)$_2$.2CDCl$_3$ forms triclinic crystals with space group $P\bar{1}$ and there is an inversion center between the Pd atoms. The Pd—Cl bonds in the title compound [2.4077 (11) and 2.4100 (11) Å] are longer than in the monomeric compound [Pd(dppe)Cl$_2$].2CH$_2$Cl$_2$ (Steffen & Palenik, 1976; 2.361 (2) and 2.357 (2) Å]. In addition, the Cl—Pd—Cl angles decrease upon bridging from 94.19 (7) to 86.96 (4)°.

Experimental

The compound [Pd$_2$(dppe)$_2$Cl$_2$)](BF$_4$)$_2$.2CDCl$_3$ was isolated by slow evaporation of a solution of bis(acetonitrile)bis[1,2-bis(diphenylphosphino)ethane]palladium(II) bis(tetrafluoroborate) in CDCl$_3$. Yellow crystals suitable for X-ray analysis were formed.

Crystal data

[Pd$_2$Cl$_2$(C$_{26}$H$_{24}$P$_2$)$_2$](BF$_4$)$_2$·2CDCl$_3$
M_r = 1494.85
Triclinic, $P\bar{1}$
a = 10.8680 (14) Å
b = 12.5889 (16) Å
c = 13.835 (2) Å
α = 114.432 (13)°
β = 109.881 (13)°
γ = 95.870 (14)°
V = 1554.0 (4) Å^3

Z = 1
D_x = 1.597 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from all reflections
θ = 1.86–25.95°
μ = 1.085 mm^{-1}
T = 293 (2) K
Prism, light yellow
0.18 × 0.16 × 0.12 mm

Data collection

Stoe–IPDS image-plate diffractometer
Oscillation scans
18439 measured reflections
5632 independent reflections
4418 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.0389
$\theta_{\rm max}$ = 25.95°
$h = -13 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.114$
S = 1.001
5632 reflections
352 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0750P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.002
$\Delta\rho_{\rm max}$ = 0.93 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.65 e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

Pd1—P2	2.2335 (11)	Pd1—Cl1^i	2.4077 (11)
Pd1—P1	2.2407 (11)	Pd1—Cl1	2.4100 (11)
P2—Pd1—P1	85.05 (4)	P2—Pd1—Cl1	92.66 (4)
P2—Pd1—Cl1^i	176.76 (4)	P1—Pd1—Cl1	175.30 (5)
P1—Pd1—Cl1^i	95.55 (4)	Cl1^i—Pd1—Cl1	86.96 (4)

Symmetry code: (i) $-x, -y, -z$.

The structure was solved by direct methods using *XMY* (Debaerdemaeker, 1993). The positions of the H atoms were calculated geometrically and their displacement factors ($U_{\rm iso}$) were set to $1.2U_{\rm eq}$ of the parent atom. The $CDCl_3$ molecule contains deuterium which was refined as hydrogen but the formula weight is calculated using the molar mass of deuterium. In the final refinement, the atoms of the anion and the solvent molecule were fixed with *DFIX* and ISOR parameters.

Data collection: *IPDS* (Stoe & Cie, 1997); cell refinement: *IPDS* (Stoe & Cie, 1997); data reduction: *IPDS* (Stoe & Cie, 1997); program(s) used to solve structure: *XMY*93 (Debaerdemaeker, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick,1990); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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