Bis[μ-bis(diphenylphosphino)methane-κ²P:P]bis[(saccharinato-κN)-palladium(I)] dichloromethane solvate

William Henderson, Brian K. Nicholson and Dong C. Chung
Bis[μ-bis(diphenylphosphino)methane-κ²P:P’]-
bis[(saccharinate-κN)palladium(I)] dichloromethane
solvate

The dimeric palladium(I) saccharinate complex \([\text{Pd}_2(\text{sac})_2(\text{dppm})_2]\), has been characterized as its dichloromethane solvate, i.e. \([\text{Pd}_2(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_{25}\text{H}_{22}\text{P}_2)_2]\cdot\text{CH}_2\text{Cl}_2\). The complex features a Pd–Pd bond bridged by two dppm ligands, with the saccharinate ligands N-bonded trans to the Pd–Pd bond.

Comment

The structure determination of \([\text{Pd}_2(\text{sac})_2(\text{dppm})_2]\)·\text{CH}_2\text{Cl}_2, (I), the first for a Pd¹ amidate, shows that the saccharinate ligands have replaced the Cl groups in the starting material. The Pd–Pd distance of 2.5962 (6) Å is comparable to that in other dinuclear Pd¹ complexes, such as \([\text{Pd}_2(\text{NO}_3)_2(\text{dppm})_2]\) (2.594 Å; Maekawa et al., 1998a). The \text{Pd}_2(\text{dppm})_2 core has a twisted-boat conformation, as has been seen in other dinuclear complexes of this type. This twisting occurs in order to allow the dppm ligands to bridge a relatively short Pd–Pd bond (Maekawa et al., 1998b). The P1–Pd1–Pd2–P2 and P3–Pd1–Pd2–P4 torsion angles are both 51.0 (1)°. The planes of the two saccharinate ligands are approximately orthogonal to each of the \text{PdP}_2 planes, but are tilted so that the ligand planes make angles of 160 and 155° with the Pd1–N1 and Pd2–N2 vectors, respectively, allowing a degree of pyramidality at the amide N atoms. The two saccharinate ligands have a cisoid arrangement with respect to the Pd–Pd bond. The two Pd–N bonds [average 2.19 (2) Å] are longer than the Pt–N bond (trans to PPh₃) in cis-[PtCl²(sac)(PPh₃)] (2.064 (6) Å; Henderson et al., 1999), reflecting the high trans influence of the Pd–Pd bond.

Experimental

\([\text{Pd}_2(\text{sac})_2(\text{dppm})_2]\) was isolated in 68% yield from a reaction of \([\text{Pd}_2\text{Cl}_2(\text{dppm})_2]\) with sodium saccharinate hydrate in refluxing MeOH for 30 min. Crystals, as the dichloromethane solvate, were obtained from \text{CH}_2\text{Cl}_2–\text{Et}_2\text{O} solution. \(^{31}\text{P}\) NMR δ = 6.6 p.p.m.

Analysis found: C 54.0, H 3.8, N 1.9%; C₆₅H₅₄Cl₂N₂O₆P₄Pd₂S₂ requires: C 54.6, H 3.8, N 2.0%.

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Crystal data

\[\text{[Pd}_2(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_2\text{H}_2\text{P}_2)_2] \cdot \text{CH}_2\text{Cl}_2}\]

Mr = 1430.80
Monoclinic, \(P2_1/n\)

\(a = 13.819 (4) \text{ Å}\)
\(b = 22.550 (7) \text{ Å}\)
\(c = 20.230 (6) \text{ Å}\)
\(\beta = 108.53 (1)^\circ\)
\(V = 5977 (3) \text{ Å}^3\)

\(Z = 4\)

Data collection

Siemens CCD diffractometer
\(\omega\) multi-scans
Absorption correction: empirical (Blessing, 1995)

\(T_{\text{min}} = 0.611, T_{\text{max}} = 0.667\)
74 438 measured reflections
12 130 independent reflections

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Refinement

Refinement on \(F^2\)

\[R(F^2) = 0.068\]

\(S = 1.02\)

12 130 reflections
748 parameters

H-atoms were placed in calculated positions, with \(U_{\text{iso}}\) 1.2 times that of the carrier C atom, in the riding-model approximation.

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References