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Bis[μ -bis(diphenylphosphino)methane- $\kappa^2 P:P'$]bis[(saccharinato- κN)-palladium(I)] dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 168 K
 Mean $\sigma(\text{C}-\text{C})$ = 0.004 Å
R factor = 0.025
wR factor = 0.068
 Data-to-parameter ratio = 16.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Bis[μ -bis(diphenylphosphino)methane- $\kappa^2 P:P'$]-bis[(saccharinato- 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.

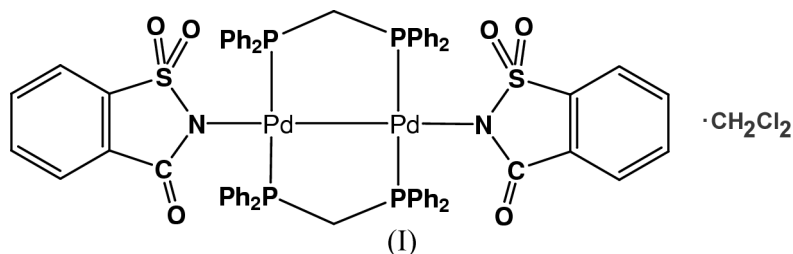
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Comment

The structure determination of $[\text{Pd}_2(\text{sac})_2(\text{dppm})_2]\cdot\text{CH}_2\text{Cl}_2$, (I), the first for a Pd^I 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 Pd^I complexes, such as $[\text{Pd}_2(\text{NO}_3)_2(\text{dppm})_2]$ (2.594 Å; Maekawa *et al.*, 1998*a*). 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.*, 1998*b*). 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 PdP₂ 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*-[PtCl(sac)(PPh₃)₂] [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 CH_2Cl_2 – Et_2O solution. ³¹P NMR δ –6.6 p.p.m. Analysis found: C 54.0, H 3.8, N 1.9%; $\text{C}_{65}\text{H}_{54}\text{Cl}_2\text{N}_2\text{O}_6\text{P}_4\text{Pd}_2\text{S}_2$ requires: C 54.6, H 3.8, N 2.0%.

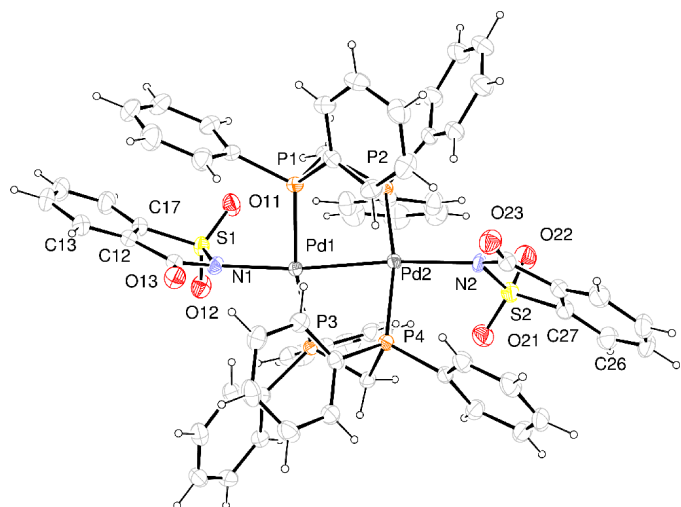


Figure 1
The structure of (I), drawn at the 50% probability level (Farrugia, 1997). The CH_2Cl_2 solvent molecule has been omitted.

Crystal data

$[\text{Pd}_2(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_{25}\text{H}_{22}\text{P}_2)_2] \cdot \text{CH}_2\text{Cl}_2$
 $M_r = 1430.80$
 Monoclinic, $P2_1/n$
 $a = 13.819$ (4) Å
 $b = 22.550$ (7) Å
 $c = 20.230$ (6) Å
 $\beta = 108.53$ (1)°
 $V = 5977$ (3) Å³
 $Z = 4$

$D_x = 1.590$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5200 reflections
 $\theta = 2$ –26°
 $\mu = 0.92$ mm⁻¹
 $T = 168$ (2) K
 Block, orange
 $0.55 \times 0.50 \times 0.44$ mm

Data collection

Siemens CCD diffractometer
 ω multi-scans
 Absorption correction: empirical (Blessing, 1995)
 $T_{\min} = 0.611$, $T_{\max} = 0.667$
 74 438 measured reflections
 12 130 independent reflections

10 579 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\text{max}} = 26.4^\circ$
 $h = -17 \rightarrow 17$
 $k = -28 \rightarrow 27$
 $l = -21 \rightarrow 25$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.068$
 $S = 1.02$
 12 130 reflections
 748 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 6.6496P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} = 0.002$$

$$\Delta\rho_{\text{max}} = 0.56 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.66 \text{ e \AA}^{-3}$$

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

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SADABS* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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