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

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The dimeric palladium(I) saccharinate complex [$Pd_2(sac)_2$-$(dppm)_2$], has been characterized as its dichloromethane solvate, *i.e.* [$Pd_2(C_7H_4NO_3S)_2(C_{25}H_{22}P_2)_2$]·$CH_2Cl_2$. The complex features a Pd—Pd bond bridged by two dppm ligands, with the saccharinate ligands N-bonded *trans* to the Pd—Pd bond.

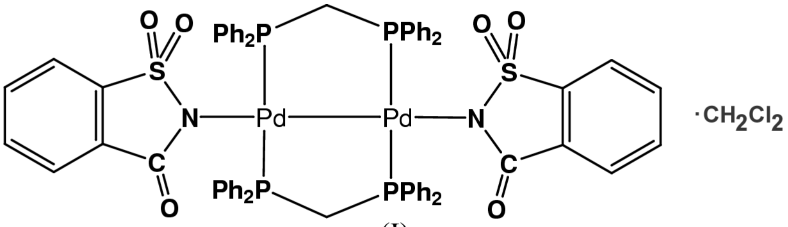
Key indicators

Single-crystal X-ray study
T = 168 K
Mean σ(C–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.

Comment

The structure determination of [$Pd_2(sac)_2(dppm)_2$]·CH_2Cl_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 [$Pd_2(NO_3)_2(dppm)_2$] (2.594 Å; Maekawa *et al.*, 1998*a*). The $Pd_2(dppm)_2$ 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 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 PPh_3) in *cis*-[$PtCl(sac)(PPh_3)_2$] [2.064 (6) Å; Henderson *et al.*, 1999], reflecting the high *trans* influence of the Pd—Pd bond.



(I)

Experimental

[$Pd_2(sac)_2(dppm)_2$] was isolated in 68% yield from a reaction of [$Pd_2Cl_2(dppm)_2$] with sodium saccharinate hydrate in refluxing MeOH for 30 min. Crystals, as the dichloromethane solvate, were obtained from CH_2Cl_2–Et_2O solution. ^{31}P NMR δ −6.6 p.p.m. Analysis found: C 54.0, H 3.8, N 1.9%; $C_{65}H_{54}Cl_2N_2O_6P_4Pd_2S_2$ requires: C 54.6, H 3.8, N 2.0%.

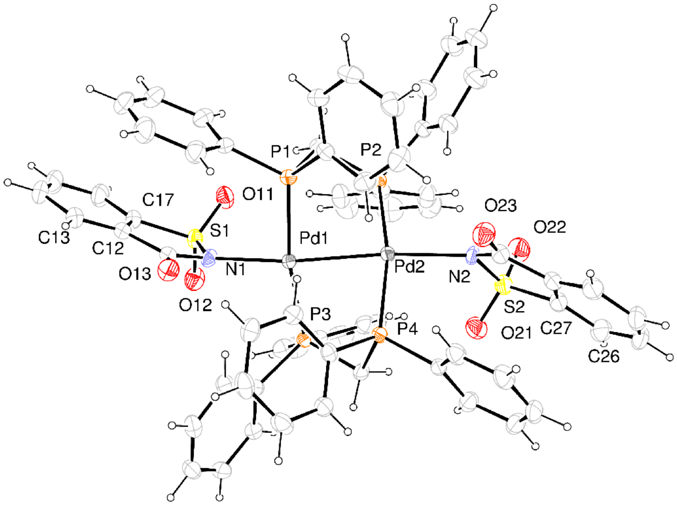


Figure 1
The structure of (I), drawn at the 50% probability level (Farrugia, 1997). The CH_2Cl_2 solvent molecule has been omitted.

Crystal data

$[Pd_2(C_7H_4NO_3S)_2(C_{25}H_{22}P_2)_2]\cdot CH_2Cl_2$
$M_r = 1430.80$
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) Å^3
$Z = 4$

$D_x = 1.590$ 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 × 0.50 × 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 $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 26.4°$
$h = -17 \rightarrow 17$
$k = -28 \rightarrow 27$
$l = -21 \rightarrow 25$

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

$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 6.6496P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.56$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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