Preparation of potentially porous, chiral organometallic materials through spontaneous resolution of pincer palladium conformers

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Experimental part

Synthesis of compounds **4**,[1] **5** and **6**[2] were according to published procedures.

Recrystallisations**:**

PCPPdCl (4):

Hexagonal crystals:

Single crystals were obtained by slow evaporation from pentane, hexane, cyclohexane or ethanol.

Monoclinic crystals:

4 (10 mg) was dissolved in 1 ml EtOH under heating, 60 µl 1M hydrochloric acid was added. Single crystals were obtained by slow evaporation.

Orthorhombic crystals:

4 (10 mg) was dissolved in 1 ml EtOH under heating, 60 µl of concentrated hydrochloric acid was added. Single crystals were obtained by slow evaporation.

PCPPdBr (5):

Hexagonal crystals:

Single crystals were obtained by slow evaporation of pentane, hexane or cyclohexane.

Orthorhombic crystals:

Fd2d: Crystals of non-solvated **5** were prepared upon attempted co-crystallisation of **5** with Br_2. A hexane solution (10 mL) of 1.80 mL of Br_2 was added to another hexane solution (5 mL) of **5** (20 mg, 0.035 mmol). Upon addition, the brownish solid was formed and was separated by filtration. Colourless crystals suitable for X-ray analyses were obtained after recrystallisation of the crude product from ethanol.

Pbca: **5** (10 mg) was dissolved in 1 ml EtOH under heating, 60 µl of concentrated hydrobromic acid was added. Single crystals were obtained by slow evaporation.

PCPPdI (6):

Single crystals were obtained by slow evaporation from methanol and heptane, respectively.

Single crystal X-ray diffraction

For **5**·Hept, data were collected on an Oxford Diffraction Xcalibur2 diffractometer with a Sapphire 3 CCD detector using graphite-monochromated MoK_α radiation (λ = 0.71073 Å) at 295 K. *CrysAlis* programs[3] were used for data collection and processing. The intensities were corrected for absorption using the multi-scan absorption correction method.[4] For **5**·BuBr-2, data were collected on Bruker-Nonius Kappa Apex II diffractometer using graphite-monochromated CuK_α radiation (λ = 1.54184 Å) at 295 K. For all other structures data were collected on the same instrument using graphite-monochromated MoK_α radiation (λ = 0.71073 Å) at 123 K. *COLLECT*[5] software was used for the data collection and *DENZO-SMN*[6] for the data processing. The structures were solved using direct methods with following programs: *SIR2002*[7] for all PCPPdCl solvates, **5**·BuBr-1, **5**·BuBr-2, **5**·DiBrOct, $\mathbf{4}_m$, $\mathbf{4}_o$, $\mathbf{5}_{oF}$, $\mathbf{5}_{oP}$ and **6**, *SIR97*[8] for **5**·Hex-1, **5**·Hex-2, **5**·CyHex, **5**·Hept, **5**·Lim, **5**·Hex·A, **5**·EtOH and *SHELXS97*[9] for $\mathbf{5}_m$. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares calculations based on F^2. All hydrogen atoms were included in calculated positions as riding atoms, with *SHELXL97*[9] defaults. Structures of **4**·Pen, **4**·PE·A-1, **4**·PE·A-3, **4**·PE·B, **5**·Hex·A, **5**·BuBr-1 and **5**·EtOH contain solvent accessible voids with small amount of solvent molecule(s) used for recrystallisation or other guest molecules. As they could not be modeled satisfactorily the data were treated with the *Squeeze* routine in *PLATON*[10] and the amounts of those heavily disordered molecules were estimated based on the electron density count from the *Squeeze* (see Tables S6 and S7). *PLATON* and *MERCURY*[11] programs were used for structure analysis and drawings preparation. The CCDC deposition numbers 913145 – 913161 and 927659 - 927663 contain the supplementary crystallographic data for this paper. These data

can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Conventional and temperature-dependent X-ray powder diffraction

The X–ray powder diffraction (XPD) data of halides **4**, **5** (including a few thermogravimetric residues heated at ~180 °C), **5**·BuBr and **6** were all measured by PANalytical X´Pert PRO diffractometer in Bragg–Brentano geometry using the step–scan technique and a Johansson monochromator to produce pure Cu $K_{\alpha 1}$ radiation (1.5406 Å; 45kV, 30mA). Small sample quantities were prepared on a silicon-made zero–background holder using petrolatum jelly as an adhesive. The data were collected from a spinning sample by a X´Celerator detector using continuous scanning mode in the 2θ range of 3–70° with a step size of 0.033° (or 0.017°) and counting times of 120 s (or 240 s) per step. Programmable divergence slit (PDS) was used in automatic mode to set the irradiated length on sample to 8 mm together with a fixed 10 mm incident beam mask. Soller slits of 0.02° rad were used on incident and diffracted beam sides together with anti–scatter slits of 4° and 8.7 mm, respectively. The data processing was performed by the X´pert HighScore Plus 2.2d program and the simulated (XPD) patterns were generated by the program *MERCURY* from the CIFs of the single crystal structures presented in this study.

Temperature dependent (*in situ*) XPD analysis of **5** was performed with the same instrument using an Anton Paar TTK450 low-temperature chamber capable of analysing samples from -193 to 450 °C. A sample was heated from room temperature to 240°C with a heating rate of 10 °C/min under static air atmosphere. Data collection were carried out isothermally at selected temperatures (80, 120, 150, 180, 210 and 240 °C) including two to three short consecutive measurements at each temperature. The instrumental parameters varied from conventional settings as follows: irradiated length 6mm, anti-scatter slit on diffracted beam side 6.6 mm, measured 2θ-range 4-60° and counting time of each consecutive pattern was 50 s per step.

Thermogravimetry

Thermal decomposition paths of PCPPdBr (**5**), PCPPdBr 1-bromobutane solvate (**5**·BuBr) and PCPPdI (**6**) powders were obtained with a PerkinElmer TGA 7 thermogravimetric analyser. In the case of **5**·BuBr**,** TG samples were prepared by crushing a few single crystals used in the single crystal structure determination. Measurements were carried out in an open platinum pan under nitrogen atmosphere (flow rate of 50 ml/min) with a heating rate of 10 °C/min in a temperature range of 25 – 700 °C. For **5,** a few replicate measurements were carried out under air atmosphere using the same measurement conditions. Temperature calibration of the analyser was made using Curie-point calibration technique (Alumel, Ni, Perkalloy, Fe). The weight balance was calibrated by measuring the standard weight of 50 mg at room temperature. The sample weights used in the measurements were about 2 – 4 mg. The decomposition temperature (T_d) was obtained as an extrapolated onset.

Description of PCPPdBr solvate structures

Very small electron density was observed in structures when cyclohexane (**5**·CyHex), heptene (**5**·Hep) and limonene (**5**·Lim) were used for recrystallisation. Addition of perfluorinated 1,8-diiodooctane into solution of hexane resulted in much higher electron density in the channels (**5**·Hex·A) as a clear indication that the additional reagent is present in the channels. As in **4**, the atoms of solvent molecules and additional reagent are completely disordered in the channels.

Butyl bromide was also used as a crystallisation solvent. Data were collected for two crystals from the same batch and they differ significantly in the residual electron density. For the second crystal, **5**·BuBr-2 *Squeeze* was not used in the refinement as residual density was very small. Similar behavior, *viz*. significant difference in the electron density, was observed in the case of PCPPdCl solvates **4**·PE·A-1, **4**·PE·A-2 and **4**·PE·A-3 where three crystals from the same batch were measured. For **5**·BuBr-2, as well as for **5**·Hept, data were collected at room temperature, and therefore, the total potential solvent area is larger and represents 17.2 % and 17.0 %, respectively, of the unit cell. A small electron density was observed also in a structure obtained by

recrystallisation of **5** from dibromooctane (**5**·DiBrOct), which means that a very small amount of solvent exists in the channels.

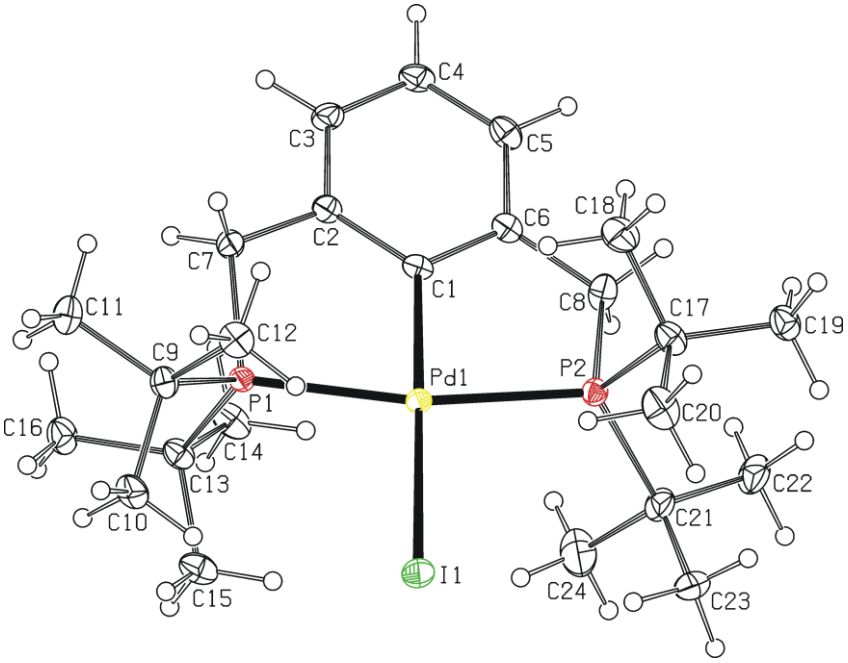

Figure S1. A molecular structure of **6**, with the atom-numbering scheme. Displacement ellipsoids for nonhydrogen atoms are drawn at the 30 % probability level.

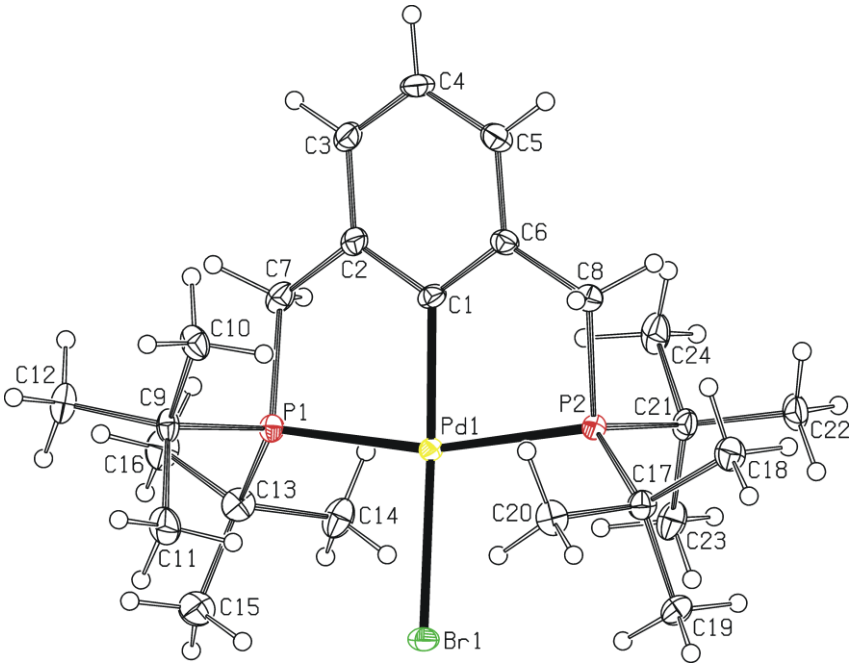

Figure S2. A molecular structure of $\mathbf{5}_{oP}$, with the atom-numbering scheme. Displacement ellipsoids for nonhydrogen atoms are drawn at the 30 % probability level.

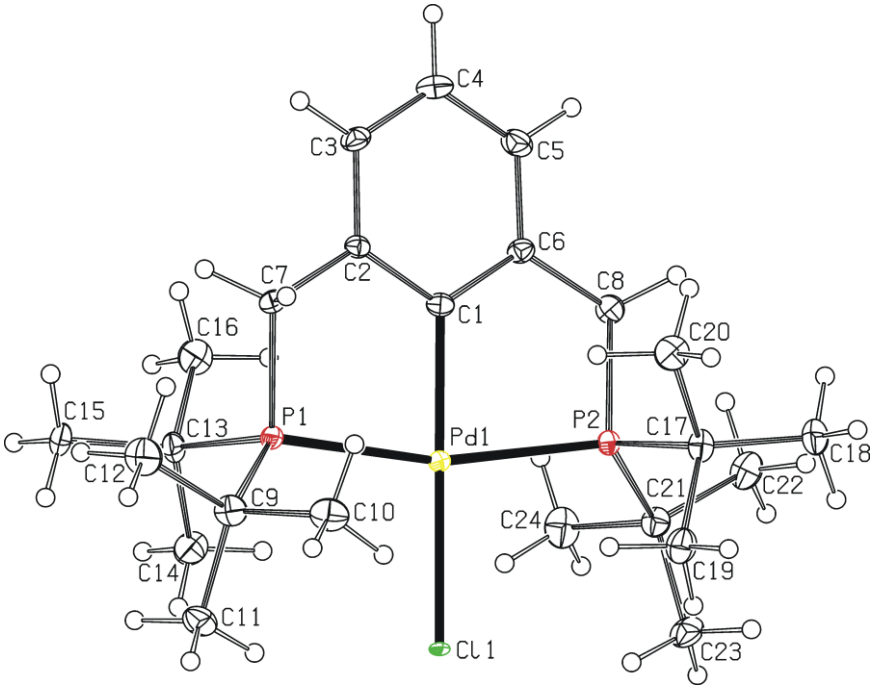

Figure S3. A molecular structure of $\mathbf{4}_{o}$, with the atom-numbering scheme. Displacement ellipsoids for nonhydrogen atoms are drawn at the 30 % probability level.

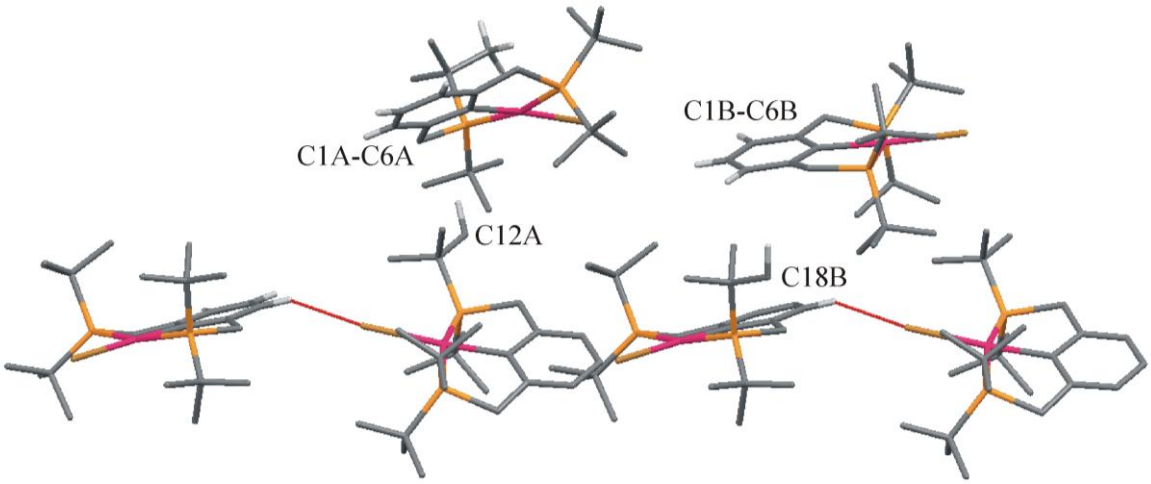

Figure S4. Part of the crystal structure of **5m**, showing C−H···Br hydrogen bond and C−H···π interactions. Hydrogen atoms not included in these interactions have been omitted for clarity.

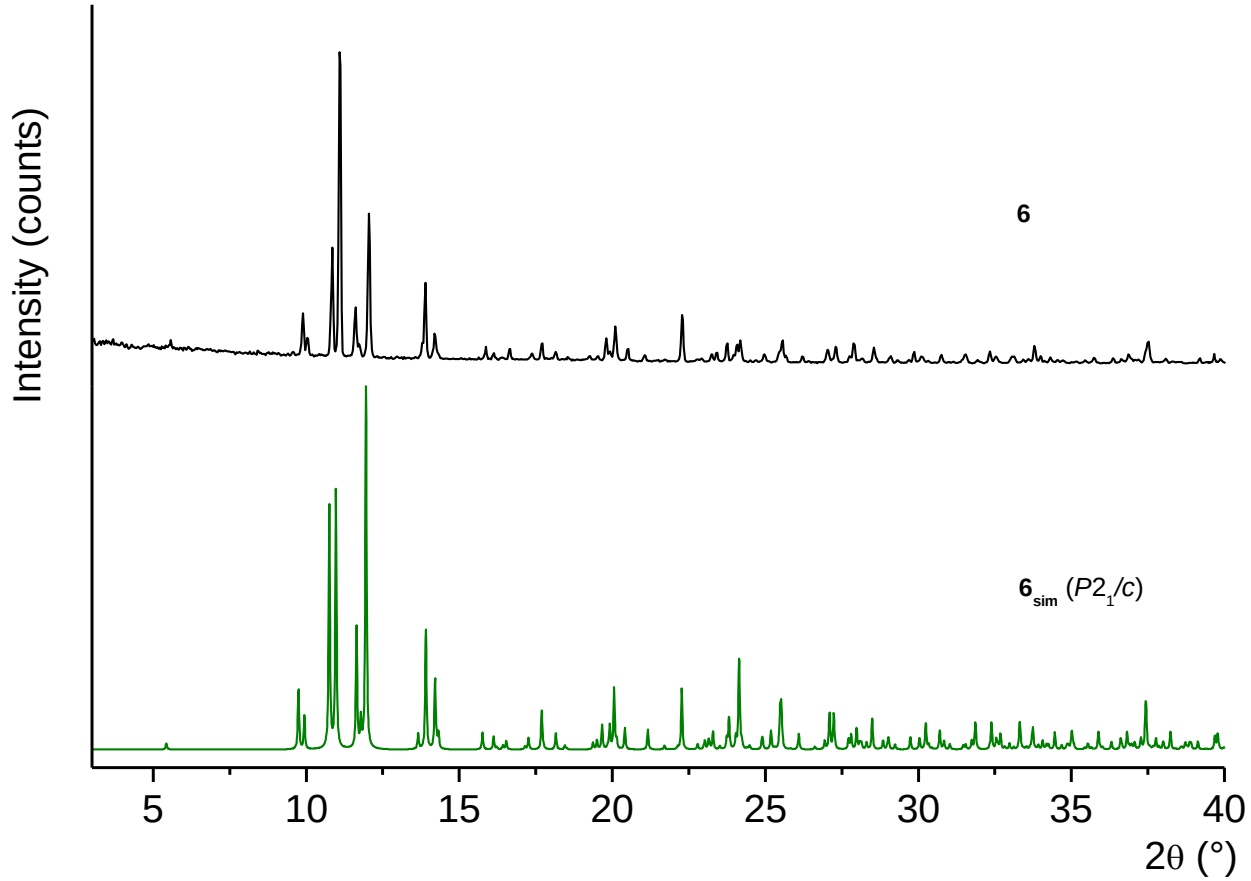

Figure S5. Experimental and simulated XRD patterns of **6**.

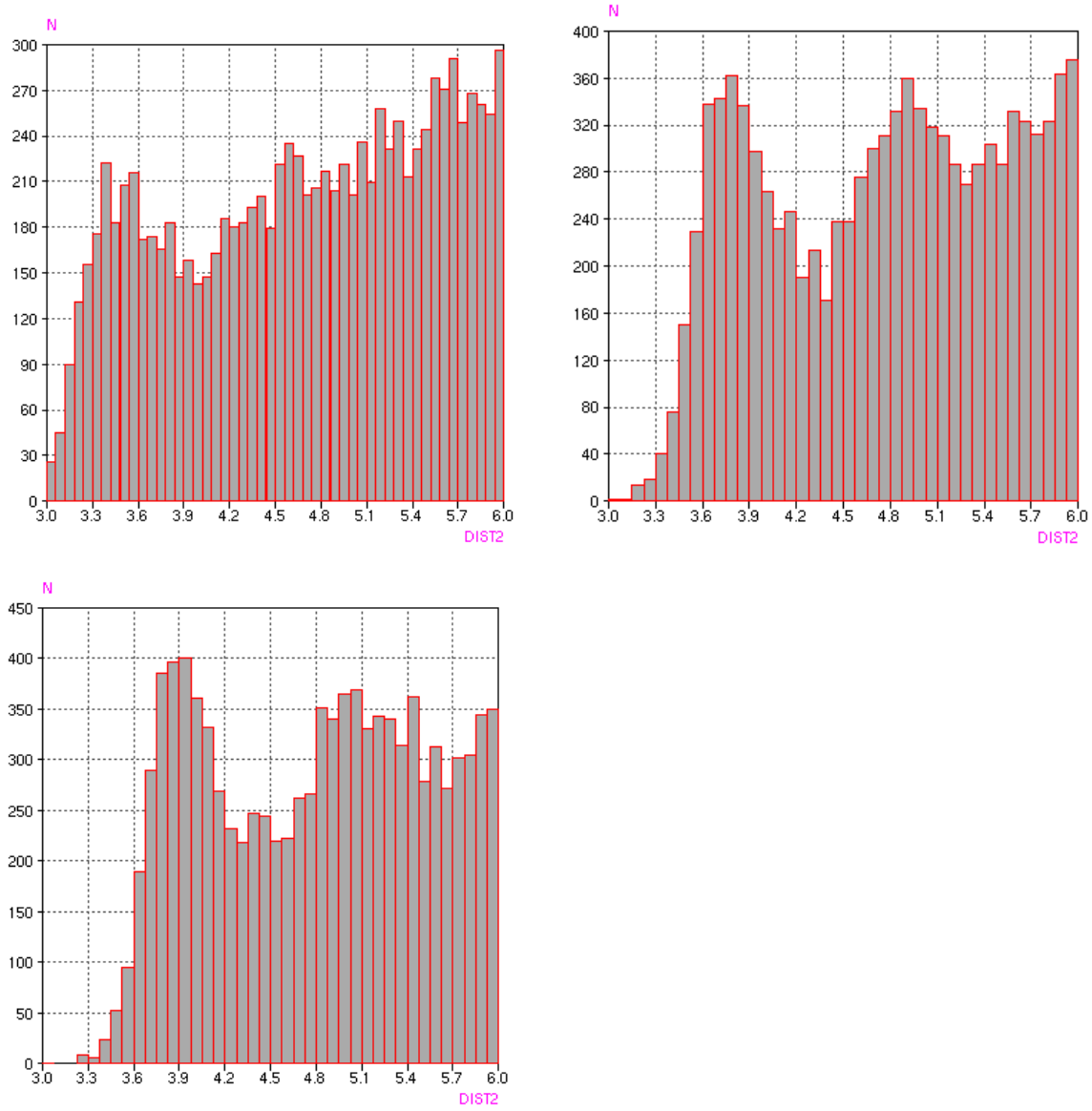

Figure S6. CSD search for M(NO_2)...H-C, Pd-Cl...H-C, Pd-Br...H-C. Optimal angles were found by selecting the peak hits and analysing the angles in another histogram.

Table S1. Maximum electron density, total potential solvent area volume and values of the torsion angle defining twist of the phenyl ring with respect to the P–Pd–P plane for PCPPdCl solvates

Structure Space group	Solvent	Additional reagent	Max. el. density / eA^{-3}	Void vol. / Å^3	P1–C2 –C2a –P1a torsion angle / ^{o}
4·Pen *P* 6_5 2 2	Pentane	----	1.184, 0.461 [a]	714	27.9(2)
4·PE·A-1 *P* 6_1 2 2	Petroleum-ether	$C_8F_{16}I_2$	2.180, 0.484 [a]	711	-27.6(3)
4·PE·A-2 *P* 6_1 2 2	Petroleum-ether	$C_8F_{16}I_2$	0.653 [b]	709	-28.0(5)
4·PE·A-3 *P* 6_5 2 2	Petroleum-ether	$C_8F_{16}I_2$	1.883, 0.434 [a]	710	27.8(4)
4·PE·B *P* 6_5 2 2	Petroleum-ether	$C_4F_8I_2$	1.259, 0.417 [a]	710	27.5(3)
4·PE·C *P* 6_1 2 2	Petroleum-ether	$C_6F_4I_2$	0.501 [b]	713	-28.4(3)

[a] before and after *Squeeze* was applied in structure refinement

[b] due to low electron density *Squeeze* was not used in the refinement

Table S2. Maximum electron density, total potential solvent area volume and values of the torsion angle defining twist of the phenyl ring with respect to the P–Pd–P plane for PCPPdBr solvates

Structure Space group	Solvent	Additional reagent	Max. el. density / eA^{-3}	Void vol. / $Å^3$	P1–C2 –C2a –P1a torsion angle / ^{o}
5·Hex-1 *P* 6_5 2 2	Hexane	----	0.559 [b]	742	27.1(4)
5·Hex-2 *P* 6_1 2 2	Hexane	----	0.509 [b]	746	-26.6(3)
5·CyHex *P* 6_1 2 2	Cyclo-hexane	----	0.745 [b]	748	-26.6(3)
5·Hept [c] *P* 6_5 2 2	Heptane	----	0.616 [b]	788	26.7(5)
5·Lim *P* 6_1 2 2	Limonene	----	0.680 [b]	749	-26.3(4)
5·Hex·A *P* 6_1 2 2	Hexane	$C_8F_{16}I_2$	1.760, 0.213 [a]	740	-26.4(2)
5·BuBr-1 *P* 6_5 2 2	Butyl-bromide	----	3.322, 0.241 [a]	737	26.5(3)
5·BuBr-2 [c] *P* 6_1 2 2	Butyl-bromide	----	0.769 [b]	800	-27.4(4)
5·DiBrOct *P* 6_1 2 2	Dibromo-octane	----	0.801 [b]	733	-26.7(7)
5·EtOH *P* 6_1 2 2	Ethanol	----	0.991, 0.412 [a]	742	-25.4(5)

[a] before and after *Squeeze* was applied in structure refinement

[b] due to low electron density *Squeeze* was not used in the refinement

[c] data were collected at room temperature

Table S3. Total potential solvent area volume, percentage of the volume per volume of the unit cell and values of the torsion angle defining twist of the phenyl ring with respect to the P–Pd–P plane for complexes **4 – 6**

Structure Space group	Void vol. / Å^3 % of unit cell	P1–C2 –C6 –P2 (or P1–C2 –C2a –P1a) torsion angle / ^{o}
4$_m$ *P* 2$_1$/*c*	112.2 4.2	± 23.11(11)
4$_o$ *P* 2$_1$ 2$_1$ 2$_1$	28.9 1.1	- 20.4(3)
5$_{oF}$ *F d* 2 *d*	-----	18.6(3)
5$_{oP}$ P b c a	147 2.7	± 25.3(3)
5$_m$ *P* 2$_1$	----- 	-17.9(3) +8.1(3)
6 *P* 2$_1$/*c*	43 1.6	± 14.1(2)

Table S4. Intermolecular hydrogen-bonding and C−H···π interactions geometry for complexes **4 – 6**

	D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (^{o})	Symmetry codes
4$_o$	C8−H8B···Cl1	0.99	2.72	3.576(4)	146	–x, –1/2+y,
4$_m$	C12−H12C···Cl1	0.98	2.82	3.739(2)	157	x, 1/2–y,
5$_{oP}$	C10−H10A···Br1	0.98	2.87	3.796(5)	159	3/2–x, –1/2+y,
5$_m$	C5B−H5B···Br1A	0.95	2.91	3.752(5)	148	2–x, –1/2+y, 1–z
	C12A−H12C···*Cg*1 [a]	0.98	2.96	3.941(6)	177	2–x, 1/2+y, –z
	C18B−H18D···Cg2 [a]	0.98	2.96	3.935(6)	176	1–x, –1/2+y, 1–z
6	C11−H11A···*Cg*3 [b]	0.98	3.00	3.921(5)	157	2–x, 1/2+y,

[a] *Cg*1 and *Cg*2 are the centroids of the C1A–C6A and C1B–C6B rings, respectively.

[b] *Cg*3 is the C1–C6 ring centroid.

Table S5. Thermal properties of compounds **5**, **5**·BuBr and **6**

Compound	Δwt-% (T) (%, (°C))*	Decomp. T_d (°C)
5 (under air)	3.16 (135) 22.2 (226)	263
5 (under N_2)	3.25 (135) 24.52 (220)	254
5·BuBr	9.26 (133)	254
6	1.02 (133)	253

* Weight loss with its temperature onset

Table S6. X-ray crystallographic data for PCPPdCl solvate structures

	4·Pen	**4**·PE·A-1	**4**·PE·A-2	**4**·PE·A-3	**4**·PE·B	**4**·PE·C
Formula	$C_{24}H_{43}ClP_2Pd \cdot 1.25\ C_5H_{12}$	$C_{24}H_{43}ClP_2Pd \cdot 0.15\ C_8F_{16}I_2$	$C_{24}H_{43}ClP_2Pd$	$C_{24}H_{43}ClP_2Pd \cdot 0.06\ C_8F_{16}I_2$	$C_{24}H_{43}ClP_2Pd \cdot 0.06\ C_4F_8I_2$	$C_{24}H_{43}ClP_2Pd$
Formula weight	625.56	633.46	535.37	574.61	562.60	535.37
T / K	123	123	123	123	123	123
Crystal system	hexagonal	hexagonal	hexagonal	hexagonal	hexagonal	hexagonal
Space group	$P\ 6_5\ 2\ 2$	$P\ 6_1\ 2\ 2$	$P\ 6_1\ 2\ 2$	$P\ 6_5\ 2\ 2$	$P\ 6_5\ 2\ 2$	$P\ 6_1\ 2\ 2$
Unit cell dimensions						
a, b / Å	16.3781(3)	16.3363(4)	16.3541(3)	16.3507(4)	16.3334(3)	16.3660(2)
c / Å	19.1666(2)	19.1594(5)	19.1791(5)	19.1646(6)	19.1661(3)	19.1996(3)
V / Å^3	4452.48(12)	4428.12(19)	4442.34(16)	4437.1(2)	4428.10(13)	4453.56(10)
Z	6	6	6	6	6	6
$D_{calc.}$ / g cm^{-3}	1.400	1.425	1.201	1.290	1.266	1.198
Absorption coef. μ / mm^{-1}	0.841	1.168	0.832	0.966	0.964	0.830
θ range / °	1.44 - 25	1.44 - 25	3.27 - 25	3.58 - 25	1.79 - 25	3.57 - 25
Collected reflections No.	23407	33461	11386	25075	33105	19539
Independent refl. No. / $R_{Int.}$	2625 / 0.0427	2606 / 0.0515	2610 / 0.0680	2609 / 0.0670	2606 / 0.0770	2616 / 0.0592
Reflections No. $I \geq 2\sigma(I)$	2506	2475	2264	2384	2401	2394
Data / Restraints / Param.	2625 / 0 / 135	2606 / 0 / 135	2610 / 0 / 135	2609 / 0 / 135	2606 / 0 / 135	2616 / 0 /135
Goodness-of-fit on F^2, S	0.967	0.994	0.981	0.982	0.982	1.001
R [$I \geq 2\sigma(I)$] / R [all data]	0.0257 / 0.0276	0.0279 / 0.0303	0.0446 / 0.0575	0.0373 / 0.0433	0.0359 / 0.0412	0.0326 / 0.0378
wR [$I \geq 2\sigma(I)$] / wR [all data]	0.0763 / 0.0775	0.0943 / 0.0957	0.1299 / 0.1429	0.0949 / 0.0970	0.1011 / 0.1046	0.1013 / 0.1054
Flack parameter, x	0.11(4)	0.07(5)	0.13(8)	0.12(6)	0.11(6)	0.08(5)
Max. / min. el. dens. / e Å^{-3}	0.461 / -0.232	0.484 / -0.300	0.653 / -0.776	0.434 / -0.379	0.417 / -0.307	0.501 / -0.463
CCDC Number	913145	913146	927659	927660	927661	927662

Table S7. X-ray crystallographic data for PCPPdBr solvate structures

	5·Hex-1	**5**·Hex-2	**5**·CyHex	**5**·Hept	**5**·Lim
Formula	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$
Formula weight	579.83	579.83	579.83	579.83	579.83
T / K	123	123	123	293	123
Crystal system	hexagonal	hexagonal	hexagonal	hexagonal	hexagonal
Space group	$P\,6_5\,2\,2$	$P\,6_1\,2\,2$	$P\,6_1\,2\,2$	$P\,6_5\,2\,2$	$P\,6_1\,2\,2$
Unit cell dimensions					
a, b / Å	16.4751(4)	16.4782(2)	16.5002(3)	16.5019(5)	16.4706(3)
c / Å	19.2531(3)	19.2576(3)	19.1805(2)	19.6449(6)	19.2727(4)
V / Å^3	4525.71(17)	4528.48(10)	4522.40(13)	4632.8(2)	4527.85(15)
Z	6	6	6	6	6
$D_{calc.}$ / g cm^{-3}	1.276	1.276	1.277	1.247	1.276
Absorption coef. μ / mm^{-1}	2.053	2.051	2.054	2.005	2.052
θ range / °	1.78 - 24.99	1.78 - 25	2.56 - 24.96	2.47 - 24.98	1.78 - 25
Collected reflections No.	30287	20617	17157	29522	25237
Independent refl. No. / $R_{Int.}$	2668 / 0.0990	2670 / 0.0424	2654 / 0.0457	2733 / 0.0756	2666 /
Reflections No. $I \geq 2\sigma(I)$	2309	2509	2489	1971	2428
Data / Restraints / Param.	2668 / 0 / 135	2670 / 0 / 135	2654 / 0 / 135	2733 / 0 / 135	2666 / 0 / 135
Goodness-of-fit on F^2, S	0.990	1.006	1.005	1.008	1.007
R [$I \geq 2\sigma(I)$] / R [all data]	0.0398 / 0.0513	0.0258 / 0.0293	0.0286 / 0.0344	0.0384 / 0.0637	0.0356 / 0.0423
wR [$I \geq 2\sigma(I)$] / wR [all data]	0.0997 / 0.1078	0.0881 / 0.0969	0.0901 / 0.1019	0.1076 / 0.1186	0.1138 / 0.1237
Flack parameter, x	0.11(2)	0.064(15)	0.035(16)	0.02(2)	0.07(2)
Max. / min. el. dens. / e Å^{-3}	0.559 / -0.404	0.510 / -0.298	0.722 / -0.691	0.616 / -0.264	0.683 / -0.436
CCDC Number	913147	913148	913149	913150	913151

Table S7. Cont.

	5·Hex·A	**5**·BuBr-1	**5**·BuBr-2	**5**·DiBrOct	**5**·EtOH
Formula	$C_{24}H_{43}BrP_2Pd \cdot 0.2\ C_8F_{16}I_2$	$C_{24}H_{43}BrP_2Pd \cdot 0.2\ C_4H_9Br$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd \cdot C_2H_6O$
Formula weight	710.61	607.24	579.83	579.83	625.90
T / K	123	123	295	123	123
Crystal system	hexagonal	hexagonal	hexagonal	hexagonal	hexagonal
Space group	$P\ 6_1\ 2\ 2$	$P\ 6_5\ 2\ 2$	$P\ 6_1\ 2\ 2$	$P\ 6_1\ 2\ 2$	$P\ 6_1\ 2\ 2$
Unit cell dimensions					
a, b / Å	16.46320(10)	16.4373(2)	16.5479(2)	16.4359(7)	16.4222(6)
c / Å	19.2709(2)	19.2522(2)	19.6062(4)	19.2850(10)	19.3446(4)
V / Å^3	4523.36(6)	4504.76(9)	4649.54(12)	4511.7(4)	4518.1(3)
Z	6	6	6	6	6
$D_{calc.}$ / g cm^{-3}	1.565	1.343	1.242	1.280	1.380
Absorption coef. μ / mm^{-1}	2.488	2.330	7.349	2.059	2.064
θ range / °	1.43 - 24.99	2.69 - 24.98	3.08 - 63.33	1.78 - 25	1.78 - 24.99
Collected reflections No.	30820	28031	64368	20745	13039
Independent refl. No. / $R_{Int.}$	2667 / 0.0532	2649 / 0.0535	2541 / 0.0600	2658 / 0.1179	2657 / 0.0882
Reflections No. $I \geq 2\sigma(I)$	2549	2427	2391	2074	2042
Data / Restraints/Param.	2667 / 0 / 135	2649 / 0 / 135	2541 / 0 / 136	2658 / 0 / 135	2657 / 0 / 135
Goodness-of-fit on F^2, S	0.981	0.999	0.996	1.005	1.019
R [$I \geq 2\sigma(I)$] / R [all data]	0.0224 / 0.0246	0.0286 / 0.0334	0.0362 / 0.0385	0.0554 / 0.0832	0.0558 / 0.0813
wR [$I \geq 2\sigma(I)$] / wR [all data]	0.0540 / 0.0548	0.0800 / 0.0821	0.1140 / 0.1163	0.1439 / 0.1643	0.1037 / 0.1121
Flack parameter, x	0.111(11)	0.090(14)	0.015(15)	0.08(3)	0.09(2)
Max. / min. el. dens. / e Å^{-3}	0.213 / -0.207	0.241 / -0.369	0.769 / -0.282	0.801 / -0.560	0.412 / -0.306
CCDC Number	927663	913152	913153	913154	913155

Table S8. X-ray crystallographic data for complexes **4 - 6**

	4_m	**4_o**	**5_{oF}**	**5_{oP}**	**5_m**	**6**
Formula	$C_{24}H_{43}ClP_2Pd$	$C_{24}H_{43}ClP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}BrP_2Pd$	$C_{24}H_{43}IP_2Pd$
Formula weight	535.37	535.37	579.83	579.83	579.83	626.82
T / K	123	123	123	123	123	123
Crystal system	monoclinic	orthorhombic	orthorhombic	orthorhombic	monoclinic	monoclinic
Space group	$P\,2_1/c$	$P\,2_1\,2_1\,2_1$	F d 2 d	P b c a	$P\,2_1$	$P\,2_1/c$
Unit cell dimensions						
a / Å	11.7371(2)	12.9992(4)	12.8097(3)	15.3928(4)	15.1219(3)	16.3407(6)
b / Å	14.9785(2)	13.8548(4)	17.9649(5)	15.5649(4)	11.6115(4)	10.8505(5)
c / Å	15.6887(2)	14.6295(3)	22.9892(6)	22.4687(6)	16.1272(3)	15.4033(6)
V / Å^3	2680.17(7)	2634.79(12)	5290.4(2)	5383.2(2)	2627.32(12)	2669.91(19)
Z	4	4	8	8	4	4
$D_{calc.}$ / g cm^{-3}	1.327	1.35	1.456	1.431	1.466	1.559
Absorption coef. μ / mm^{-1}	0.919	0.935	2.341	2.301	2.357	1.980
θ range / °	3.68 - 25	2.56 - 24.99	2.14 - 25	1.81 - 25	2.36 - 25	2.74 - 25
Collected reflections No.	20988	9818	4730	28514	27122	11776
Independent refl. No. / $R_{Int.}$	4707 / 0.0271	4565 / 0.0276	1718 / 0.0378	4743 / 0.0581	9016 / 0.0451	4680 / 0.0357
Reflections No. $I \geq 2\sigma(I)$	4257	4219	1650	3943	8242	4137
Data / Restraints /Param.	4707 / 0 / 265	4565 / 0 / 265	1718 / 1 / 135	4743 / 0 / 265	9016 / 1 / 530	4680 / 0 / 265
Goodness-of-fit on F^2, S	0.996	0.945	1.032	1.004	1.066	0.967
R [$I \geq 2\sigma(I)$] / R [all data]	0.0195 / 0.0235	0.0333 / 0.0376	0.0284 / 0.0304	0.0339 / 0.0480	0.0323 / 0.0390	0.0337 / 0.0405
wR [$I \geq 2\sigma(I)$] / wR [all data]	0.0475 / 0.0498	0.0765 / 0.0788	0.0838 / 0.0862	0.1011 / 0.1186	0.0617 / 0.0638	0.0780 / 0.0815
Flack parameter, x	---	0.10(4)	0.098(14)	---	0.233(6)	---
Max. / min. el. dens. / e Å^{-3}	0.337 / -0.335	0.882 / -0.398	0.416 / -0.391	0.571 / -0.675	0.433 / -0.387	1.225 / -0.865
CCDC Number	913156	913157	913158	913159	913160	913161

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