

**Reactions of R<sub>2</sub>P–P(SiMe<sub>3</sub>)Li with [(R'<sub>3</sub>P)<sub>2</sub>PtCl<sub>2</sub>]. A General and Efficient Entry to Phosphanylphosphinidene Complexes of Platinum. Syntheses and Structures of [(\eta<sup>2</sup>-P=P<sup>i</sup>Pr<sub>2</sub>)Pt(*p*-Tol<sub>3</sub>P)<sub>2</sub>], [(\eta<sup>2</sup>-P=P<sup>t</sup>Bu<sub>2</sub>)Pt(*p*-Tol<sub>3</sub>P)<sub>2</sub>], [<{(\eta^2-P=P(N^iPr\_2)\_2)}Pt(*p*-Tol<sub>3</sub>P)<sub>2</sub>] and [<{Et<sub>2</sub>PhP})<sub>2</sub>Pt}<sub>2</sub>P<sub>2</sub>]**

Wioleta Domańska-Babul<sup>a</sup>, Jarosław Chojnacki<sup>a</sup>, Eberhard Matern<sup>b</sup>, Jerzy Pikies<sup>a\*</sup>

<sup>a</sup> Faculty of Chemistry, Department of Inorganic Chemistry, Gdańsk University of Technology,  
G. Narutowicza St. 11/12. PL-80-952 Gdańsk, Poland

<sup>b</sup> Institut für Anorganische Chemie, Universität Karlsruhe (TH), D-76128 Karlsruhe, Germany

**Supplementary materials**

---

\* Prof. Dr. Jerzy Pikies

e-mail [pikies@chem.pg.gda.pl](mailto:pikies@chem.pg.gda.pl)

Tel: + 48 58 3472622

**Table 2.** Crystal data and structure refinement for  $[\{\eta^2\text{-P}=\text{P}^i\text{Pr}_2\}\text{Pt}(p\text{-Tol}_3\text{P})_2\cdot\text{toluene}]$  (**4·toluene**),  $[\{\eta^2\text{-P}=\text{P}^t\text{Bu}_2\}\text{Pt}(p\text{-Tol}_3\text{P})_2\cdot\text{toluene}]$  (**6·toluene**),  $[\{\eta^2\text{-P}=\text{P}(\text{N}^i\text{Pr}_2)_2\}\text{Pt}(p\text{-Tol}_3\text{P})_2\cdot\text{toluene}]$  (**13·toluene**) and  $[(\text{Ph}_2\text{EtP})_2\text{Pt}]_2\text{P}_2$  (**14**)

Identification code	<b>4·toluene</b>	<b>6·toluene</b>	<b>13·toluene</b>	<b>14</b>
Empirical formula	C55 H64 P4 Pt	C57 H68 P4 Pt	C61 H78 N2 P4 Pt	C56 H60 P6 Pt2
Formula weight	1044.03	1072.08	1158.22	1309.04
Temperature	120(2) K	120(2) K	100(2) K	120(2) K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system	Triclinic	Triclinic	Monoclinic	Monoclinic
Space group	$P\bar{1}$	$P\bar{1}$	$P 2_1/c$	$P 2_1/c$
Unit cell dimensions	$a = 13.2476(5)$ Å $b = 13.3322(5)$ Å $c = 15.2573(6)$ Å $\alpha = 74.503(3)^\circ$ $\beta = 77.636(3)^\circ$ $\gamma = 75.331(4)^\circ$	$a = 13.3097(8)$ Å $b = 13.4111(10)$ Å $c = 15.3900(12)$ Å $\alpha = 77.834(6)^\circ$ $\beta = 77.293(6)^\circ$ $\gamma = 76.611(6)^\circ$	$a = 15.336(3)$ Å $b = 24.002(5)$ Å $c = 18.974(7)$ Å $\alpha = 90^\circ$ $\beta = 125.737(19)^\circ$ $\gamma = 90^\circ$	$a = 19.5441(16)$ Å $b = 9.6862(10)$ Å $c = 29.7907(17)$ Å $\alpha = 90^\circ$ $\beta = 107.751(9)^\circ$ $\gamma = 90^\circ$
Volume	2480.93(16) Å <sup>3</sup>	2569.7(3) Å <sup>3</sup>	5669(3) Å <sup>3</sup>	5371.1(8) Å <sup>3</sup>
Z	2	2	4	4
Density (calculated)	1.398 Mg/m <sup>3</sup>	1.386 Mg/m <sup>3</sup>	1.357 Mg/m <sup>3</sup>	1.619 Mg/m <sup>3</sup>
Absorption coefficient	2.992 mm <sup>-1</sup>	2.890 mm <sup>-1</sup>	2.627 mm <sup>-1</sup>	5.417 mm <sup>-1</sup>
F(000)	1064	1096	2384	2568
Crystal size	0.16 x 0.12 x 0.06 mm <sup>3</sup>	0.21 x 0.12 x 0.03 mm <sup>3</sup>	0.28 x 0.26 x 0.21 mm <sup>3</sup>	0.2 x 0.07 x 0.01 mm <sup>3</sup>
Theta range for data collection	2.09 to 25.10°	2.11 to 26.00°	2.74 to 27.50°	2.22 to 25.50°
Index ranges	-15≤h≤15, -15≤k≤15,	-15≤h≤16, -15≤k≤16,	-19≤h≤17, -31≤k≤30,	-23≤h≤23, -11≤k≤11,

	-15<=l<=18	-13<=l<=18	-24<=l<=24	-36<=l<=31
Reflections collected	17416	18854	41630	27697
Independent reflections	8813 [R(int) = 0.0341]	10040 [R(int) = 0.0746]	13012 [R(int) = 0.0396]	9542 [R(int) = 0.1484]
Completeness to theta = 25.50°	99.8 %	99.4 %	99.9 %	95.3 %
Absorption correction	Analytical	Analytical	Analytical	Analytical
Max. and min. transmission	0.815 and 0.673	0.861 and 0.662	0.614 and 0.53879	0.981 and 0.427
Refinement method	Full-matrix least-squares on F <sup>2</sup>			
Data / restraints / parameters	8813 / 13 / 517	10040 / 0 / 572	13012 / 12 / 622	9542 / 54 / 581
Goodness-of-fit on F <sup>2</sup>	1.042	0.990	1.108	1.140
Final R indices [I>2sigma(I)]	R1 = 0.0376, wR2 = 0.0924	R1 = 0.0494, wR2 = 0.1131	R1 = 0.0346, wR2 = 0.0734	R1 = 0.1227, wR2 = 0.2875
R indices (all data)	R1 = 0.0528, wR2 = 0.1040	R1 = 0.0786, wR2 = 0.1276	R1 = 0.0396, wR2 = 0.0752	R1 = 0.1777, wR2 = 0.3115
Largest diff. peak and hole	1.781 and -0.855 e·Å <sup>-3</sup>	2.563 and -1.373 e·Å <sup>-3</sup>	3.246 and -0.937 e·Å <sup>-3</sup>	4.351 and -4.018 e·Å <sup>-3</sup>