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Note

First microwave-assisted synthesis of an electron-rich phosphane and its coordination chemistry with platinum(II) and palladium(II)

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ABSTRACT

The P–O ligand 3-(di(2-methoxyphenyl)phosphanyl)propionic acid (**HL**) was synthesized by a microwave-assisted reaction of a secondary phosphane. The coordination of **HL** to Pt^{II} yielded the neutral mononuclear complex *trans*-[PtCl(κ^2-*P,O*-**L**)(κ-P-**HL**)] (**1**), while the reaction of PdClMe(η^4-COD) (COD = 1,4-cyclooctadiene) with **HL** in the presence of NEt_3 gave the anionic Pd^{II} compound of the formula ($HNEt_3$)[PdClMe(κ^2-*P,O*-**L**)] (**2**). Upon crystallization of the latter compound the neutral chloride-bridged dimetallic compound *cis*-[Pd(μ-Cl)Me(**HL**)]$_2$ (**3**) was obtained. **HL**, **1** and **3**·CH_2Cl_2 have been characterized by single crystal X-ray structure analyses.

1. Introduction

The organometallic chemistry of functionalized phosphanes modified with an additional (non-phosphane) donor group has revealed a great deal of applications in homogeneous catalysis [1]. Among these non-phosphane donors, sulfonate [2] and carboxylate [3] groups have gained particular interest in the Ni(P-O)-catalyzed olefin oligomerization (SHOP) [4] as well as in the Pd(P-O)-catalyzed non-strictly CO olefin copolymerization reaction [2]. In particular, the presence of the 2-methoxyphenyl moiety in the ligand scaffold has shown to confer high stability to the ligand against phosphane oxide formation, which is the most encountered deactivation process occurring in metal-phosphane catalyzed reactions [5]. In order to contribute to the field of synthesis of new phosphanyl-carboxylate ligands, we report here a microwave-assisted synthesis of 3-(di(2-methoxyphenyl)phosphanyl)propionic acid (**HL**) and its coordination chemistry with Pt^{II} and Pd^{II}.

2. Experimental

2.1. Methods and materials

All synthetic reactions and manipulations were carried out under an argon atmosphere by using standard Schlenk techniques. Reagents were used as received from Aldrich, unless stated otherwise. Bis-(di(2-methoxy)phenyl)phosphane [6] and [PdClMe(η^4-COD)] [7] were prepared according to literature methods. The microwave synthesis was carried out with an Anton Paar Synthos 3000 (1400 W unpulsed microwave) dual magnetron system, with an operation volume of 60 mL and reaction vessels of PTFE–TFM. Deuterated solvents for routine NMR measurements were dried with activated molecular sieves. ^1H, $^{13}C\{^1H\}$ and $^{31}P\{^1H\}$ NMR spectra were obtained with a Bruker Avance DRX-400 spectrometer acquiring spectra at 400.13, 100.62 and 161.98 MHz, respectively. Chemical shifts (δ) are reported in ppm relative to TMS (^1H and ^{13}C NMR spectra) or 85% H_3PO_4. IR spectra were acquired on a Nicolet 5700 ATR FT-IR spectrometer. Microanalyses were performed using a Carlo-Erba Model 1106 elemental analyzer. FAB mass spectrometric measurements were carried out on a Finnigan MAT-95 spectrometer, using 3-nitrobenzylalcohol (NOBA) as matrix.

2.2. Synthesis of *HL*

Deaerated ethyl 3-chloropropionate (12 mL, 88.0 mmol) was added to a teflon vessel, which was sealed after

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bis-(di(2-methoxy)phenyl)phosphane (200.3 mg, 0.813 mmol) had been added. The reaction vessel was heated by microwave irradiation at 100 °C for 2 h. Afterwards, deaerated water (50 mL) was added to the reaction mixture. The water phase was separated, concentrated to dryness and the viscous residue was dissolved in deaerated EtOH (20 mL). Then $NaOCH_3$ (7.0 g, 130.1 mmol) was added to the reaction solution, which was stirred at 80 °C for 4 h. The reaction solvent was then completely removed and the crude residue was dissolved in deaerated water (20 mL) and HCl was added at room temperature, causing the precipitation of the desired product as an off-white product, which was then separated from solution by filtration and dried by vacuum. Yield 106.2 mg (41%). Mp 145–148 °C. *Anal.* Calc. for $C_{17}H_{19}O_4P$ (318.29): C, 64.15; H, 6.02. Found: C, 63.99; H, 6.04%. ^{1}H NMR (DMSO-d_6, 21 °C): δ 2.21–2.22 (s, 4H, CH_2CH_2COO), 3.73 (s, 6H, OCH_3), 6.91–7.37 (m, 8H, Ar-*H*), 12.17 (s, 1H, COOH). ^{13}C{^{1}H} NMR (DMSO-d_6, 21 °C): δ 19.78 (d, $^2J_{PC}$ = 12.6 Hz, CH_2CO_2H), 31.31 ($^1J_{PC}$ = 18.6 Hz, CH_2P), 55.96 (s, OCH_3), 111.30 (s, Ar-*C*), 121.30 (s, Ar-*C*), 125.03 (d, $^1J_{PC}$ = 17.1 Hz, *ipso*-Ar-*C*), 130.66 (s, Ar-*C*), 132.42 (d, $^2J_{PC}$ = 5.2 Hz, Ar-*C*), 161.40 (d, $^2J_{PC}$ = 13.4 Hz, Ar-*C*), 174.57 (d, $^3J_{PC}$ = 13.9 Hz, CO_2H). ^{31}P{^{1}H} NMR (DMSO-d_6, 21 °C): δ −33.97 (s). IR (ν, cm^{-1}) 1695 (CO_2H). MS (FAB+) *m*/*z*: 318.11 (M^+).

2.3. Synthesis of trans-[PtCl(κ^2−P,O-**L**)(κ-P-**HL**)] (**1**)

In a Schlenk tube **HL** (100.0 mg, 0.314 mmol) was suspended in water (20 mL) and on addition of KOH (17.6 mg, 0.314 mmol), the suspension became a clear solution. To this latter solution a solution of $K_2PtCl_4{\cdot}4H_20$ (76.5 mg, 0.157 mmol) in water (20 mL) was added under stirring, which was continued at room temperature for 2 h. Afterwards, the solvent was completely removed obtaining the crude off-white product, which was suspended in a small amount of MeOH (2 mL), filtered off and then dried under vacuum. Yield 98.4 mg (72%). Mp 204 °C. *Anal.* Calc. for $C_{34}H_{37}ClO_8P_2Pt$ (866.11): C, 47.15; H, 4.31. Found: C, 47.01; H, 4.14%. ^{1}H NMR (DMF-d_7, 21 °C): δ 2.25 (m, 2H, CH_2), 2.53 (m, 2H, CH_2), 2.93 (m, 4H, CH_2), 3.85 (s, 6H, OCH_3), 3.92 (s, 6H, OCH_3), 6.99–7.91 (m, 16H, Ar-*H*). ^{13}C{^{1}H} NMR (DMF-d_7, 21 °C): δ 19.78 (s, CH_2CO_2), 22.64 (d, $^2J_{PC}$ = 28.3 Hz, CH_2P), 55.49 (s, OCH_3), 55.80 (s, OCH_3), 111.22–161.46 (Ar-*C*), 174.39 (s, CO_2H), 176.71 (s, CO_2^-). ^{31}P{^{1}H} NMR (DMF-d_7, 21 °C): δ 14.74 (d, $^2J_{PP}$ = 467.0 Hz, $^1J_{PtP}$ = 2793.0 Hz, $P(CO_2H)$), 16.86 (d, $^2J_{PP}$ = 467.0 Hz, $^1J_{PtP}$ = 2782.0 Hz, $P(CO_2^-)$). ^{195}Pt NMR (DMF-d_7, 21 °C): δ −3461.16 (t, $^1J_{PtP}$ = 2799.0 Hz). IR (ν, cm^{-1}) 1695 (CO_2H), 1671 (CO_2^-). MS (FAB+) *m*/*z*: 867.3 ($M+H^+$), 830.4 ($M-Cl^-$).

2.4. Synthesis of ($HNEt_3$) [PdClMe(κ^2-P,O-**L**)] (**2**)

In a Schlenk tube **HL** (50.0 mg, 0.157 mmol) was dissolved in deaerated CH_2Cl_2 (10 mL). Afterwards, NEt_3 (5.0 mL) was added to the solution and allowed to stir for 1 h. Then [PdClMe(η^4-COD)] (30.1 mg, 0.157 mmol) was added to the latter solution, which turned slightly yellow. After a reaction time of half an hour the solvent was removed completely and the resulting brownish solid was washed with *n*-hexane and dried under vacuum. Yield 45.8 mg (51%). Mp 109 °C (decomposition). *Anal.* Calc. for $C_{24}H_{37}ClNO_4Pd$ (576.40): C, 52.88; H, 6.79. Found: C, 52.73; H, 6.59%. ^{1}H NMR (CD_2Cl_2, 21 °C): δ 0.08 (d, $^3J_{PH}$ = 2.8 Hz, 3H, PdCH_3), 1.25 (t, $^3J_{HH}$ = 7.2 Hz, 9H, CH_3CH_2), 2.42 (dm, $^2J_{PC}$ = 29.6 Hz, 2H, CH_2CO_2), 2.70 (m, 2H, CH_2P), 3.21 (q, $^3J_{HH}$ = 7.2 Hz, 6H, CH_3CH_2), 3.86 (s, 6H, OCH_3), 7.00–7.73 (m, 8H, Ar-*H*). ^{13}C{^{1}H} NMR (CD_2Cl_2, 21 °C): δ −6.43 (s, PdCH_3), 8.44 (s, CH_3CH_2), 22.27 (d, $^1J_{PC}$ = 32.14 Hz, CH_2P), 33.27 (s, CH_2CO_2), 45.07 (s, CH_3CH_2), 55.51 (s, OCH_3), 111.05 (d, $^3J_{PC}$ = 4.1 Hz, Ar-*C*), 118.10 (d, $^1J_{PC}$ = 17.2 Hz, *ipso*-Ar-*C*), 120.34 (d, $^2J_{PC}$ = 11.9 Hz, Ar-*C*), 132.61 (s, Ar-*C*), 160.38 (s, Ar-*C*), 179.26 (s, CO_2^-). ^{31}P{^{1}H} NMR (CD_2Cl_2, 21 °C): δ 37.66 (s). IR (ν, cm^{-1}) 1585 (CO_2^-). MS (FAB+) *m*/*z*: 439.0 (M^+–$HNEt_3$–Cl).

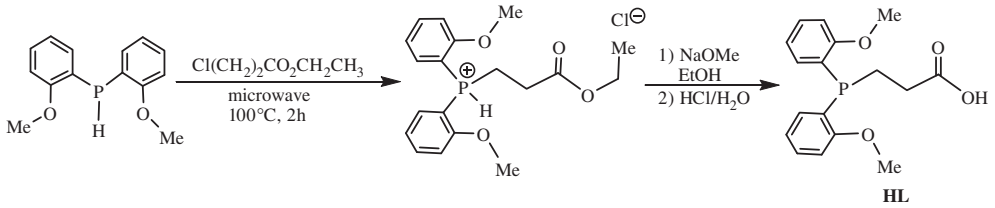
2.5. X-ray crystal structure determinations

Single crystals of **HL**, suitable for an X-ray structure analysis, were obtained by slow evaporation of a 1:1 (v/v) water–EtOH solution of **HL** on air, while single crystals of **1** were obtained from a corresponding water-acetic acid-acetone solution. Single crystals

Table 1
Crystallographic data and structure refinement details for compounds **HL**, **1** and **3**·CH_2Cl_2.[a]

	HL	**1**	**3**·CH_2Cl_2
Empirical formula	$C_{17}H_{19}O_4P$	$C_{34}H_{37}ClO_8P_2Pt$	$C_{37}H_{44}Cl_4O_8P_2Pd_2$
Formula weight	318.29	866.11	1033.26
a (Å)	12.9634(4)	12.4658(2)	19.1988(3)
b (Å)	8.1575(6)	16.1336(2)	13.9774(2)
c (Å)	15.3186(8)	17.3074(3)	17.2253(2)
α (°)			
β (°)	98.790(3)	94.6333(8)	93.7793(7)
γ (°)			
V (Å^3)	1600.90(15)	3469.46(9)	4612.35(11)
Z	4	4	4
D_{calc} (Mg/m^3)	1.321	1.658	1.488
Absorption coefficient (mm^{-1})	0.187	4.263	1.124
F(0 0 0)	672	1720	2080
Θ Range for data collection (°)	1.59–27.50	1.64–27.47	1.80–27.48
Limiting indices	$-16 \leqslant h \leqslant 16$	$-15 \leqslant h \leqslant 16$	$-24 \leqslant h \leqslant 24$
	$-10 \leqslant k \leqslant 10$	$-20 \leqslant k \leqslant 20$	$-18 \leqslant k \leqslant 18$
	$-19 \leqslant l \leqslant 19$	$-22 \leqslant l \leqslant 22$	$-22 \leqslant l \leqslant 22$
Reflections collected	9060	30 073	39 158
Independent reflections (R_{int})	3672 (0.0376)	7954 (0.0230)	10 569 (0.0380)
Data/restraints/parameters	3638/0/203	7912/0/420	10 459/17/499
Goodness-of-fit on F^2	1.033	1.030	1.026
Final *R* indices [$I > 2\sigma(I)$] R_1, wR_2	0.0372, 0.0950	0.0253, 0.0609	0.0371, 0.0881
R indices (all data) R_1, wR_2	0.0460, 0.1130	0.0321, 0.0718	0.0465, 0.1052
Largest difference in peak and hole (e Å^{-3})	0.200 and −0.210	1.260 and −0.910	0.974 and −0.570

[a] Temperature, 243(2) K; crystal shape, prism; crystal size, 0.25 × 0.15 × 0.10 (**HL**, **1**), 0.45 × 0.20 × 0.03 (**3**·CH_2Cl_2); crystal colour, colourless; crystal system, monoclinic; space group, $P2_1/c$; absorption corrections, multi-scan; refinement method, full-matrix least-squares on F^2.



Scheme 1.

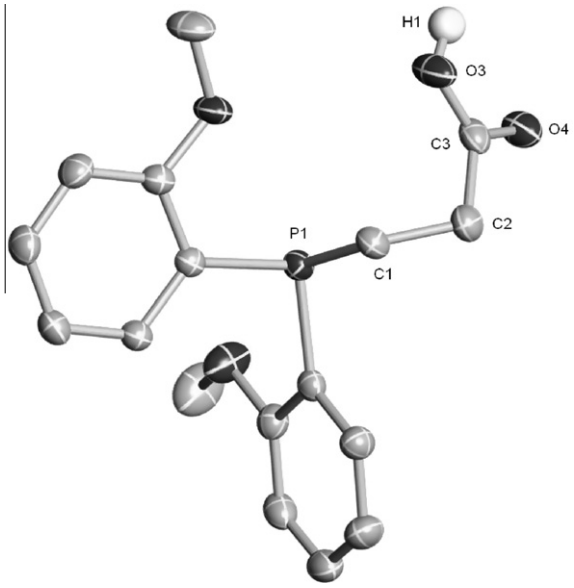


Fig. 1. ORTEP-plot of **HL**. Hydrogen atoms, except for the carboxylic group H(1), are omitted for clarity and thermal ellipsoids are shown at the 30% probability level.

of **3**·CH_2Cl_2, were obtained by a reverse gas-phase diffusion, where **3**, dissolved in CH_2Cl_2, was exposed to *n*-hexane at room temperature under light exclusion. Crystallographic data and structure refinement details for all three compounds are summarized in Table 1. Diffraction data were collected on a Nonius Kappa CCD diffractometer using φ–ω-scans and graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å). Cell refinement, data reduction and empirical absorption correction were carried out with the Denzo and Scalepack programs [8a]. All structure determination calculations were performed with SHELXTL NT V6.1 including SHELXS-97 and SHELXL-97 [8b]. Final refinements on F^2 were carried out applying anisotropic thermal parameters for all non-hydrogen atoms, while all hydrogen atoms were included in the refinement using a riding model with isotropic *U* values depending on the U_{eq} of the adjacent non-hydrogen atoms, except for the carboxylic acid hydrogen atoms in **3**·CH_2Cl_2, which were refined isotropically with fixed *U*.

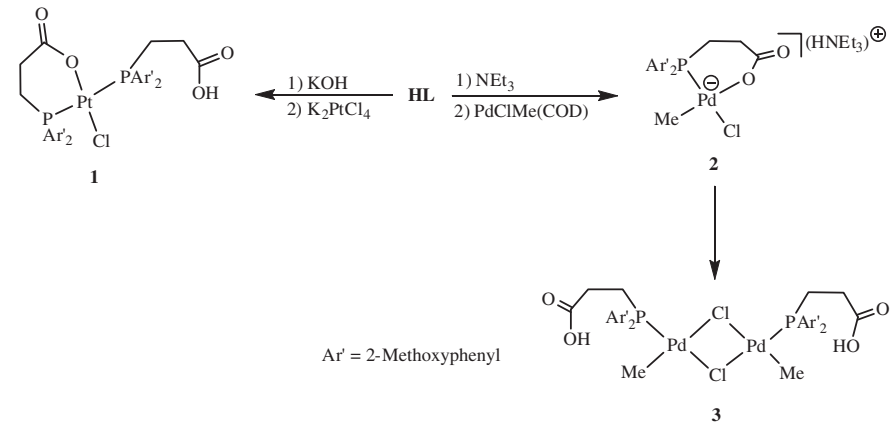
3. Results and discussion

The new P–O ligand **HL** was obtained by a microwave-assisted reaction of bis(di(2-methoxy)phenyl)phosphane in neat ethyl-3-chloropropionate, followed by a hydrolysis step and protonation reaction of the resulting sodium salt with HCl as shown in Scheme 1.

The microwave-assisted conversion of bis(di(2-methoxy)-phenyl)phosphane (i.e. secondary phosphane) into a most likely phosphonium intermediate, characterized by a $^{31}P\{^1H\}$ NMR singlet centred at 28.71 ppm in EtOH, is so far unique [9]. Upon hydrolysis of the phosphonium intermediate, giving **NaL** and successive protonation of the latter compound with HCl in water gave **HL** after work up with 41% yield. Alternative synthetic protocols such as a Michael reaction between bis(di(2-methoxy)phenyl)phosphane and ethyl acrylate [10] did not give the desired product, while the thermal reaction between 3-chloropropionic acid and the *in situ* formed Na-salt of bis(di(2-methoxy)phenyl)phosphane in a THF/DMSO solvent mixture gave **HL** with only 8% yield. A broad ^1H NMR singlet centered at 12.17 ppm (DMSO-d_6) is proof of the presence of the carboxylic acid functionality and a ^{13}C NMR doublet at 174.57 ppm ($^3J_{PC}$ = 13.9 Hz) is in agreement with a P(1)C(1)C(2)C(3) dihedral angle of 70.4(2)° [10], as found in the crystal structure of **HL** (Fig. 1).

The reaction of **KL** with $K_2PtCl_4·4H_2O$ in a 2:1 stoichiometric ratio gave **1** with 72% yield (Scheme 2).

The *trans* stereochemistry of the phosphorus atoms in **1** and the concomitant presence of the κ^2-*P*,*O* and κ-P coordination mode of $\mathbf{L}^-$ and **HL**, respectively, is unambiguously confirmed by a single



Scheme 2.

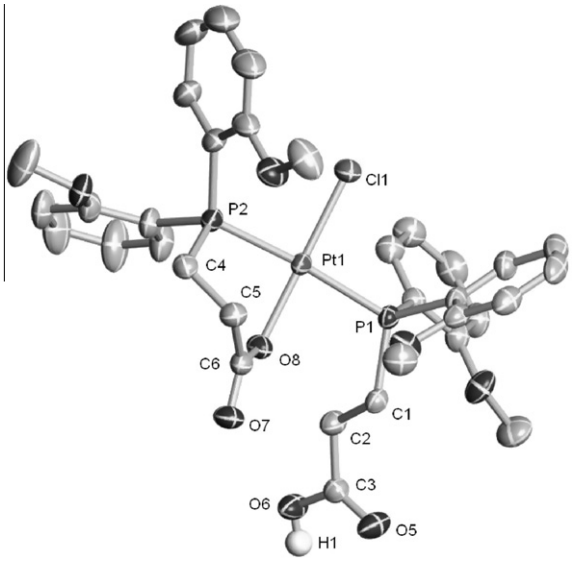


Fig. 2. ORTEP-plot of **1**. Hydrogen atoms, except for the carboxylic acid hydrogen atom H(1), are omitted for clarity and thermal ellipsoids are shown at the 30% probability level.

Table 2
Selected bond distances [Å] and angles [°] for **1** and **3**·CH_2Cl_2.

	1	**3**·CH_2Cl_2
Pt(1)–P(1)	2.3319(10)	
Pt(1)–P(2)	2.2892(10)	
Pt(1)–Cl(1)	2.2830(11)	
Pt(1)–O(8)	2.044(3)	
Pd(1)–Cl(1)		2.4131(10)
Pd(1)–Cl(2)		2.4672(10)
Pd(2)–Cl(1)		2.4114(11)
Pd(2)–Cl(2)		2.4513(11)
Pd(1)–P(1)		2.2208(10)
Pd(2)–P(2)		2.2182(11)
Pd(1)–C(10)		2.044(4)
Pd(2)–C(20)		2.038(4)
Cl(1)–Pt(1)–O(8)	177.93(8)	
P(1)–Pt(1)–P(2)	175.03(3)	
P(2)–Pt(1)–O(8)	86.21(8)	
Cl(1)–Pd(1)–Cl(2)		84.44(4)
Cl(1)–Pd(2)–Cl(2)		84.82(4)
Pd(1)–Cl(1)–Pd(2)		87.81(4)
Pd(1)–Cl(2)–Pd(2)		85.72(3)
P(1)–Pd(1)–C(10)		87.87(12)
P(2)–Pd(2)–C(20)		85.69(12)
P(1)–Pd(1)–Cl(1)		175.68(4)
P(2)–Pd(2)–Cl(1)		174.48(4)

crystal X-ray structure analysis (Fig. 2). Selected bond distances and angles are compiled in Table 2.

The coordination of $\mathbf{L}^-$ to Pt^{II} led to the formation of a six-membered heteroatom ring, showing a bite angle of 86.21(8)°, which is comparable to that found for bis(2-methoxyphenyl)phosphanylethylsulphonate (P-SO_3^-) in [Pd(P-SO_3)$_2$] of 85.30(12) and 84.44(13)° [2]. The $^{31}P\{^1H\}$ NMR spectrum of **1**, acquired in dry DMF-d_7, proved that the relative stereochemistry, found for the solid state, is maintained also in solution. Accordingly, both phosphorus nuclei are part of an AB spin system characterized by $^2J_{PP}$ of 476.0 Hz [11]. $^{13}C\{^1H\}$ NMR and IR data are in agreement with the concomitant presence of a coordinating carboxylate moiety and a free carboxylic acid functionality.

The reaction of [PdClMe(η^4-COD)] with [(**HNEt$_3$)L**] in an 1:1 stoichiometric ratio gave the anionic Pd^{II}-compound **2** with 51% yield (Scheme 2). The *cis* coordination of the methyl group to palladium with respect to the phosphorus donor atom is proved by a

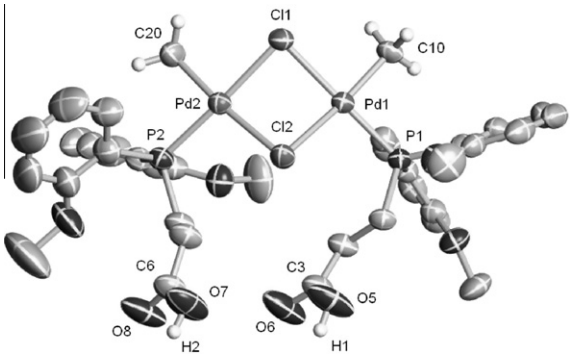


Fig. 3. ORTEP-plot of **3**·CH_2Cl_2. The solvent molecule and hydrogen atoms, except for the carboxylic acid hydrogen atoms H(1) and H(2), are omitted for clarity and thermal ellipsoids are shown at the 30% probability level.

characteristic $^3J_{PH}$ of 2.8 Hz [2,12] for PdCH_3, while the κ^2-*P,O*-coordination of $\mathbf{L}^-$ to palladium is confirmed by the ^{13}C NMR singlet at 179.26 ppm. Upon crystallization of **2**, in the presence of traces of water, **3**·CH_2Cl_2 was obtained in only a small amount (i.e. 10%). The latter compound proved to be insoluble in all commonly used organic solvents. An ORTEP-plot of **3**·CH_2Cl_2 is shown in Fig. 3 and selected bond distances and angles are reported in Table 2.

The partial conversion of **2** into **3** may be rationalized by a water mediated protonation of the coordinating carboxylic oxygen atom in **2**, yielding NEt_3 and the mononuclear T-shaped Pd^{II} species [PdClMe(**HL**)] [13], which subsequently dimerizes and crystallizes yielding **3**·CH_2Cl_2. The regioselectivity of this latter dimerization reaction is object of a further study. Both **HL** ligands in **3**·CH_2Cl_2 are *cis*-coordinated with respect to the central $Pd_2(\mu\text{-}Cl)_2$ core, which shows a folding angle of 136.50(4)°.

4. Conclusions

The new P–O ligand **HL** was synthesized by means of a microwave-assisted reaction involving for the first time a secondary phosphane. The coordination of **HL** to Pt^{II} and Pd^{II} has been confirmed by multinuclear NMR-, IR-, MS-spectroscopy and single crystal X-ray structure analysis. Compound **1** is a rare example of a Pt^{II} compound showing both metal coordination modes (i.e. κ-*P* and κ^2-*P,O*) of a P–O ligand.

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Appendix A. Supplementary material

CCDC 811119, 811120 and 811121 contain the supplementary crystallographic data for **HL**, **1** and **3**·CH_2Cl_2, respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2011.05.020.

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