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Note

First structurally characterized triazinyl palladium complexes via oxidative addition of cyanuric chloride derivatives

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ABSTRACT

The reaction of cyanuric chloride and two disubstituted derivatives with $[Pd(PPh_3)_4]$ is described, leading to the *trans*-triazinyl complexes $[(PPh_3)_2Pd(C_3N_3R_2)Cl]$ **2a**–**c** (R = Ph (**a**), *t*-Bu (**b**), Cl (**c**)) in good to excellent yield via oxidative addition. The complexes were fully characterized including X-ray structure determination.

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1. Introduction

Derivatives of 1,3,5-triazine have found numerous applications in chemistry ranging from building blocks in supramolecular chemistry [1] to the use as herbicides [2]. Triazines are also used in medicinal chemistry [3], catalysis [4] and polymer chemistry [5]. Cheap and easily available cyanuric chloride is a convenient source to get into 1,3,5-triazine chemistry due to the easy displacement of the three chlorine atoms by a wide range of nucleophilic reagents. Furthermore, the three chlorine atoms can be subsequently addressed by carefully controlling the reaction temperature to get mono- $(0 \circ C)$, di- $(25 \circ C)$ or three-substituted (65 $\circ C)$ compounds [1].

Some C-metallated triazine derivatives are known in the literature including main group metals like Mg [6] and some transition metals. Reports about the nucleophilic substitution of C-bonded halide by carbonyl metallates date back to the mid-1960s [7]. To the best of our knowledge there are only two reports about the oxidative addition of triazine derivatives to transition metal fragments: the reaction of s-triazine with Mo(PMe₃)₆, described by Parkin and coworkers, proceeds under C–H activation to a η^2 (CN) molybdenum hydride complex [8] while the oxidative addition of C–F bonds of a couple of fluorotriazins to low-valent titanium compounds has been reported by Beckhaus and coworkers [9]. No triazinyl palladium complexes have been synthesized so far although a theoretical study has been published lately [10].

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Recently, we reported a synthesis of the first pyrimidine based Nheterocyclic carbene complex by oxidative addition of an N-alkyl-2-halopyrimidinium cation to $[Pd(PPh_3)_4]$ [11]. Related oxidative addition reactions for other ring systems have been reported earlier [12]. This work inspired us to explore whether oxidative additions of cyanuric chloride derivatives to Pd(0) proceed analogously and preliminary results of this study are reported in this paper.

2. Results and discussion

Diphenyltriazine **1a** was synthesized by treatment of cyanuric chloride with 2 equiv. of Ph–Mg–Cl [13]. The di(*tert*-butyl) derivative **1b** was prepared by a copper-catalyzed cross-coupling reaction with *t*-BuMgCl reported by Hintermann et al. [14]. All triazine compounds reacted with [Pd(PPh₃)₄] smoothly in THF at ambient temperature over night to afford the complexes **2a–c** as shown in Scheme 1. All palladium complexes were isolated as white or yellow powders in good to excellent yield after workup and fully characterized by spectroscopic methods and elemental analysis. The ³¹P{¹H}-NMR spectra show only one signal and indicate the *trans* orientation of the triphenylphosphine ligands. A *cis*-isomer is not observed in the ³¹P{¹H}-NMR spectrum and indicates a fast reaction, where the thermodynamically more stable *trans*-complex is formed rapidly.

The formation of the *trans*-complexes is also evident from the ¹³C{¹H}-NMR spectrum. The C2 atom of the triazine moiety shows a triplet structure with ${}^{2}J_{CP} = 8.2$ Hz (**2a**), 8.2 Hz (**2b**), 8.7 Hz (**2c**). Due to a strong virtual coupling between the two phosphorus nuclei the phenyl C-atoms of the triphenylphosphine ligands also





Scheme 1. Synthesis of the triazinyl palladium complexes 2a-c.

show a CP coupling (ipso: ${}^{1/3}J_{CP} = 22.7$ Hz (**2a**), 22.5 Hz (**2b**), 24.0 Hz (**2c**); ortho: ${}^{2/4}J_{CP} = 6.3$ Hz (**2a**), 6.3 Hz (**2b**), 6.4 Hz (**2c**); meta: ${}^{3/5}J_{CP} = 5.2$ Hz (**2a**), 5.1 Hz (**2b**), 5.2 Hz (**2c**)) to the two phosphorus nuclei. The effect of a strong virtual PP coupling has been demonstrated before by Nelson and Cary [15].

Single crystals suitable for X-ray diffraction were obtained by slow diffusion of *n*-hexane into a saturated solution of **2a** and **2c** in dichloromethane at room temperature. The molecular structures of the *trans*-complexes **2a** and **2c** are shown in Figs. 1 and 2, respectively, together with some representative geometric parameters. They are the first structurally characterized Pd-triazinyl complexes.

The structure shows the Pd atom of **2a** in a slightly distorted square-planar environment with *trans* angles for C1–Pd1–Cl1 of 172.49(5)° and P1–Pd1–P2 of 167.366(16)°, respectively, and *cis* angles for C1-Pd1-P of 97.198(18)° for P1 and 90.569(18)° for P2. The coordination plane around the Pd atom forms a dihedral angle of 74.5° with the triazine ring plane. While the Pd–P and Pd–Cl bond lengths fall in the range observed for (PPh₃)₂PdCl complexes with other aryl groups, the Pd1–C1 bond of 198.52(16) pm is slightly shorter than for the aforementioned derivatives [16].

In contrast to the diphenyltriazinyl derivative **2a**, the structure of the dichloro compound **2c** shows two independent molecules in the chosen asymmetric unit of the crystal structure. Both Pd atoms



Fig. 1. ORTEP representation of complex **2a** (ellipsoids drawn at 50% probability level; H atoms are omitted for clarity). Selected distances (pm) and angles (deg): Pd1–Cl1 240.65(5), Pd1–P1 236.44(5), Pd1–P2 235.50(5), Pd1–C1 198.52(16), C1–Pd1–Cl1 172.49(5), P1–Pd1–P2 167.366(16), C11–Pd1–P1 97.198(18), C1–Pd1–P2 90.569(18), C1–Pd1–P1 86.93(5), Cl1–Pd1–P2 90.569(18).

are in a slightly distorted square-planar environment with *trans* angles for C1–Pd1–Cl1 177.31(8)° and P1–Pd1–P2 177.55(3)° for Pd1 and Cl4–Pd2–C40 175.25(8)° and P3–Pd2–P4 173.64(3)° for Pd2. The two independent molecules feature almost identical bond distances and angles. The most notable difference is found for the interplanar angle between the coordination plane around palladium and the triazine ring plane, which is found to be 86.5° and 73.3°, respectively. The Pd–C bonds of 196.30(2) pm and 197.30(2) pm are even a bit shorter than in complex **2a** and are among the shortest Pd–C bonds observed for related (PPh₃)₂ClPd-aryl complexes, including the N-methylpyridinium derived carbene complexes described recently by Raubenheimer and Schuster [16f], and Raubenheimer and Herrmann [12d], respectively.

3. Conclusion

Oxidative additions of chlorotriazines to $[Pd(PPh_3)_4]$ were found to proceed straightforwardly to give the square-planar *trans*-(PPh_3)₂PdCl-triazinyl complexes **2a**–**c** in good yield. X-ray crystal structure determinations for complexes **2a** and **2c** revealed geometrical parameters very close to related derivatives, the comparatively short Pd–C bonds being noteworthy exceptions.

4. Experimental section

4.1. General

All manipulations were performed under an inert gas atmosphere of dry nitrogen by using standard vacuum line and Schlenk techniques. Glassware was dried at 120 °C in an oven for at least 12 h. THF was distilled from sodium/benzophenone and *n*-hexane from sodium. All solvents were stored under an atmosphere of nitrogen. NMR spectra were recorded on Bruker Avance DRX200 or Bruker Avance DRX500 spectrometers. Chemical shifts are reported in ppm (δ) compared to TMS (¹H and ¹³C) using the residual peak of deuterated solvents as internal standard (¹H: CDCl₃ 7.26 ppm; ¹³C: CDCl₃ 77.0 ppm) and ³¹P{¹H} spectra to external H₃PO₄ (85%). Coupling constants (*J*) are quoted in Hz. Elemental analyses were performed by the institute of pharmaceutical chemistry at the Heinrich-Heine-Universität Düsseldorf on a Perkin Elmer CHN 2400 series II. For MS data a Thermo Finnigan Trace DSQ (EI) and a Bruker Ultrafelx I TOF (MALDI) were used.

4.2. Starting materials and reagents

2-Chloro-4,6-diphenyl-1,3,5-triazine [13] and 2-chloro-4,6-di*tert*-butyl-1,3,5-triazine [14] were prepared according to literature procedures. Cyanuric chloride was purchased from Acros Organics and used without further purification.

4.3. General procedure for complexation

500 mg of $[Pd(PPh_3)_4]$ and 1.05 equiv. of **1a**–**c** were dissolved in 15 ml of THF and the resulting yellow solution was stirred over night at ambient temperature. After removal of the solvent, the crude product was suspended in 10 ml *n*-hexane and stirred over night to remove the PPh₃. The product was isolated by filtration and washed with *n*-hexane (3 × 5 ml) and dried in *vacuo*.

4.3.1. Chloro-(2,4-diphenyl)-1,3,5-triazinyl-bis-triphenylphosphanpalladium(II) (**2a**)

Obtained as a white powder in 75% yield. ¹H-NMR (500 MHz in CDCl₃): δ = 7.13–7.46 (m, 30H, -PPh₃), 7.68–8.11 (m, 10H, Ph_{Triazinyl}) ppm; ¹³C{¹H}-NMR (126 MHz in CDCl₃): δ = 127.93 (t, ^{3/5}J_{CP} = 5.2 Hz, C _{meta}, PPh₃), 127.99 (s, C _{ortho} Ph), 128.49 (s, C _{meta}



Fig. 2. ORTEP representation of complex **2c** (ellipsoids drawn at 50% probability level; H atoms are omitted for clarity). Selected distances (pm) and angles (deg): Pd1–Cl1 236.76(7), Pd1–P1 233.78(7), Pd1–P2 233.00(7), Pd1–C1 196.30(2), C1–Pd1–Cl1 177.31(8), P1–Pd1–P2 177.55(3), Cl1–Pd1–P2 88.15(2), C1–Pd1–P2 89.41(7), C1–Pd1–P1 90.97(7), Cl1–Pd1–P1 91.41(2); Pd2–Cl4 237.56(8), Pd2–P3 235.69(7), Pd2–P4 234.57(7), Pd2–C40 197.30(2), Cl4–Pd2–C40 175.25(8), P3–Pd2–P4 173.64(3), Cl4–Pd2–P3 93.18(3), Cl4–Pd2–P4 89.91(3), C40–Pd2–P3 89.38(7), C40–Pd2–P4 87.94(7).

Ph), 130.79 (t, ${}^{1/3}J_{CP} = 22.7$ Hz, C $_{ipso}$ PPh₃), 131.14 (s, C $_{para}$ PPh₃), 134.61 (t, ${}^{2/4}J_{CP} = 6.3$ Hz, C $_{ortho}$ PPh₃), 136.47 (s, C $_{ipso}$ Ph), 165.15 (s, C–Ph), 206.88 (t, ${}^{2}J_{CP} = 8.2$ Hz, C $_{ipso}$ C–Pd) ppm; ${}^{31}P{}^{1}H$ -NMR (81 MHz in CDCl₃): $\delta = 19.87$ (s) ppm; MS (MALDI): m/z (%) = 862.2 (M⁺-Cl⁻, 30%), 600.0 (M⁺-Cl⁻-PPh₃, 50%); elemental analysis calcd. (%) for C₅₁H₄₀ClN₃P₂Pd (898.72): C 68.16, H 4.49, N 4.68; found: C 65.09, H 4.44, N 4.48.

4.3.2. Chloro-(2,4-di-tert-butyl)-1,3,5-triazinyl-bis-triphenylphosphan-palladium(II) (**2b**)

Obtained as a pale yellow powder in 85% yield. ¹H-NMR (500 MHz in CDCl₃): $\delta = 0.86$ (s, 18H, C(CH₃)₃), 7.17–7.20 (m, 12H, –PPh₃), 7.25–7.26 (m, 6H, –PPh₃), 7.54–7.58 (m, 12H, –PPh₃) ppm; ¹³C{¹H}-NMR (126 MHz in CDCl₃): $\delta = 28.61$ (s, C(<u>CH₃)₃</u>), 38.26 (s, C(CH₃)₃), 128.00 (t, ^{3/5}*J*_{CP} = 5.1 Hz, <u>C</u> meta–Ph), 129.85 (s, <u>C</u> para–Ph), 131.47 (t, ^{1/3}*J*_{CP} Hz = 22.5 Hz, <u>C</u> ipso–Ph), 134.56 (t, ^{2/4}*J*_{CP} = 6.3 Hz, <u>C</u> ortho–Ph), 179.12 (s, <u>C</u> meta–Triazinyl), 207.10 (t, ^{2/}*J*_{CP} = 8.2 Hz, C ipso <u>C</u>–Pd) ppm; ³¹P{¹H}-NMR (81 MHz in CDCl₃): $\delta = 19.62$ (s) ppm; MS (MALDI): *m/z* (%) = 822.2 (M⁺–Cl⁻, 60%), 560.1 (M⁺–Cl⁻–PPh₃, 100%); elemental analysis *calcd.* (%) for C₄₇H₄₈ClN₃P₂Pd (858.74): C 65.74, H 5.63, N 4.89; *found*: C 65.85, H 5.52, N 4.65.

4.3.3. Chloro-(2,4-dichloro)-1,3,5-triazinyl-bis-triphenylphosphan-palladium(II) (**2c**)

Obtained as a yellow powder in 95% yield. ¹H-NMR (500 MHz in CDCl₃): δ = 7.34–7.37 (m, 12H, –PPh₃), 7.40–7.43 (m, 6H, –PPh₃), 7.68–7.72 (m, 12H, -PPh₃) ppm; ¹³C{¹H}-NMR (126 MHz in CDCl₃): δ = 128.28 (t, ^{3/5}J_{CP} = 5.2 Hz, <u>C</u> meta-Ph), 129.79 (t, ^{1/3}J_{CP} Hz = 24.0 Hz, <u>C</u> ipso-Ph), 130.69 (s, <u>C</u> para-Ph), 134.47 (t, ^{2/4}J_{CP} = 6.4 Hz, <u>C</u> ortho-Ph), 164.52 (s, <u>C</u> meta-Triazinyl), 211.75 (t, ²J_{CP} = 8.7 Hz, C ipso <u>C</u>-Pd) ppm; ³¹P{¹H}-NMR (81 MHz in CDCl₃): δ = 20.81 (s) ppm; MS (MALDI): *m/z* (%) = 779.9 (M⁺-Cl⁻, 45%), 517.8 (M⁺-Cl⁻-PPh₃, 35%); elemental analysis *calcd*. (%) for C₃₉H₃₀Cl₃N₃P₂Pd (815.41): C 57.45, H 3.71, N 5.15; *found*: C 57.52, H 3.78, N 4.86.

5. Crystal structure determinations

Crystals of compounds $C_{51}H_{40}ClN_3P_2Pd(2a)$ and $C_{39}H_{30}Cl_3N_3P_2Pd(2c)$ suitable for X-ray study were selected by means of a polarization

microscope and investigated on an STOE Imaging Plate Diffraction System and an Oxford Diffraction Excalibur Diffractometer, respectively, using graphite monochromatized MoK α radiation ($\lambda = 0.71073$ Å). Unit cell parameters were determined by leastsquares refinements on the positions of 8000 and 84 265 reflections, respectively. For **2a** the anorthic crystal system is consistent

Table 1 Details of the crystal structure determinations.

Compound	2a	2c
CCDC number	833624	833625
Empirical formula	C ₅₁ H ₄₀ ClN ₃ P ₂ Pd	$C_{39}H_{30}Cl_3N_3P_2Pd$
Formla weight	898.65	815.35
Temperature	291(2) K	291(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	triclinic	Monoclinic
Space group	PĪ	$P2_1/c$
Unit cell	a = 9.3352(6) Å	a = 26.1701(6) Å
dimensions	b = 11.2620(8) Å	b = 11.29530(10) Å
	c = 21.4646(14) Å	c = 25.5013(6) Å
	$\alpha = 97.069(8)^{\circ}$	$lpha=90^{\circ}$
	$\beta = 101.217(7)^{\circ}$	$eta=100.297(2)^\circ$
	$\gamma = 93.088(8)^{\circ}$	$\gamma=90^{\circ}$
Volume	2189.7(3) Å ³	7416.8(3) Å ³
Z	2	8
Density (calculated)	1.363 mg/m ³	1.460 mg/m ³
Absorption coefficient	0.596 mm^{-1}	0.835 mm^{-1}
F(000)	920	3296
Crystal size	$0.2 \times 0.2 \times 0.2 \text{ mm}^3$	$0.54\times0.23\times0.21\ mm^3$
Theta range for	2.49-25.00°	2.97–25.00°
data collection		
Index ranges	-10 < = h < = 11,	-31 < = h < = 31,
	-13 < = k < = 13,	-13 < = k < = 11,
	-25 < = l < = 25	-30 < = l < = 30
Reflections collected	28751	54559
Independent reflections	7526 [R(int) = 0.0312]	13 038 [R(int) = 0.0481]
Refinement method	Full-matrix	Full-matrix least-squares
	least-squares	on F^2
	on F ²	
Data/restraints/parameters	7526/0/523	13038/0/865
Goodness-of-fit on F ²	0.978	1.094
Final <i>R</i> indices $[I > 2\sigma(I)]$	$R_1 = 0.0241$,	$R_1 = 0.0325$, w $R_2 = 0.0673$
	$wR_2 = 0.0637$	
R indices (all data)	$R_1 = 0.0278,$	$R_1 = 0.0359$, w $R_2 = 0.0685$
	$wR_2 = 0.0647$	
Largest diff. peak and hole	0.572 and -0.202 e.Å ⁻³	0.404 and -0.390 e.Å ⁻³

with space group types *P* 1 and $P\overline{1}$. In accordance with E-statistics significantly better results were observed with a structural model in the centrosymmetric type *P*1 in the course of structure refinement. Space group type no. 14 was uniquely determined in the case 2c. Corrections for Lorentz and polarization effects and in the case of 2c multi-scan absorption corrections were applied. The structures were solved by direct methods [17] and subsequent ΔF -syntheses. Approximate positions of all the hydrogen atoms were found in different stages of converging refinements by full-matrix leastsquares calculations on F^2 [18]. With idealised bonds lengths and angles assumed for all the CH groups of both compounds, the riding model was applied for the corresponding H atoms and their isotropic displacement parameters were constrained to 120% of the equivalent isotropic displacement parameters of the parent carbon atoms. Details can be found in Table 1. CCDC-833624 (2a) and CCDC-833625 (2c) contain the supplementary crystallographic data (excluding structure factors) 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.

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