Inorganica Chimica Acta 548 (2023) 121365

Contents lists available at ScienceDirect

Inorganica Chimica Acta

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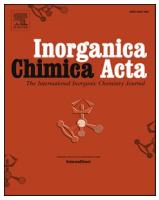


Novel hybrid phosphinite-theophylline ligands and their Pd(II) complexes. Synthesis, characterization and catalytic evaluation in Suzuki-Miyaura couplings

Edgar Marín-Carrillo [a], Hugo Valdés [b], Simón Hernández-Ortega [a], David Morales-Morales [a,*]

[a] Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Ciudad de México C.P. 04510, Mexico
[b] Institut de Química Computacional i Catàlisi, Departament de Química, Universitat de Girona, c/ Mª Aurèlia Capmany 69, 17003 Girona, Spain

ARTICLE INFO

Keywords:
Phosphinite Pd(II) complex
Pd complexes, Suzuki-Miyaura couplings
Xanthine
Theophylline
Microwaves
Aqueous C–C Cross-couplings
Catalysis

ABSTRACT

Two Pd(II) complexes bearing bidentated [R'$_2$(OR)P^P(OR)R'$_2$] (OR = 7-(2,3-dihydroxypropyl)theophylline and R' = Ph for (**1**) and R' = ^{i}Pr for (**2**)) ligands were synthesized and fully characterized, including the unequivocal determination of their molecular structures by single crystal X-ray diffraction analysis. Thus confirming the coordination of the phosphinite-theophylline ligand to the Pd(II) center in a chelate manner. Exploration of these compounds as catalysts in Suzuki-Miyaura Couplings revealed complex (**2**) to be the best of the pair of complexes in the formation of C—C bonds in the reactions of phenylboronic acid and a series of *para*-substituted bromobenzenes under mild reaction conditions (50 °C, 50 W, 5 min, and 0.05 mol % of catalyst), achieving yields of > 99 %.

1. Introduction

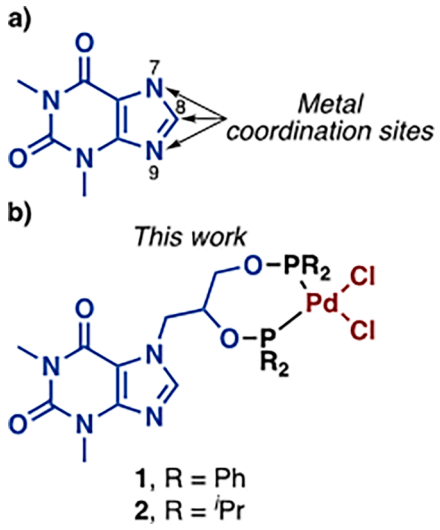
Theophylline, caffeine and theobromine, belong to a family of organic compounds called xanthines, which are present in common food products such as coffee, tea, commercial beverages, and cocoa [1]. In particular, theophylline is widely used for the treatment of respiratory system illnesses, and some other derivatives are used with other medical purposes such as treatment of Parkinson disease, heart failure and renal insufficiency [2]. The merge of xanthines with transition metals is also interesting for the design of complexes with potential use as anticancer and antibiotics agents [3], and more recently, to be used as catalysts for reactions such as the alkyne-azide cycloaddition, Glaser homocoupling and Suzuki-Miyaura couplings [4].

Theophylline has three potential coordination sites to form metal complexes (Scheme 1). First, the coordination through the nitrogen atoms N7 or N9 [5]. In 2014 our group described an efficient catalytic system based on xanthines for the Suzuki-Miyaura cross-coupling reaction of halopyridines in water [6]. The catalytic active species was formed after the coordination of the xanthine to the metal center, through the N9 atom. The catalytic reaction was carried out at 120 °C, irradiating with MW at 90 W for 10 min, and using 1 mol% of catalyst. Under these conditions, the C—C coupling products were obtained in yields up to 95 %. The second coordination mode of theophylline can be through the C8 carbon, forming a *N*-heterocyclic carbene (NHC) ligand [4c,7]. In 2011, Luo reported the synthesis of caffeine-based Pd(II) NHC-complexes and their use as catalysts in C—C cross-coupling reactions in water as solvent. The catalytic reaction was performed for 6 h at 65 °C, reaching up to 95 % yield [4c]. In 2017 Canovese and Paganelli designed a Pd(II) complex with a xanthine-based ligand, where the xanthine platform forms a NHC ligand [4g]. The complex catalyzes Suzuki-Miyaura couplings in 2 h at 60° or 80 °C, noteworthy the fact that they used water as solvent. Thus, xanthines are suitable platforms to prepare active species to catalyze cross-coupling reactions in water, although the direct coordination of the metal to the xanthine often produces water-insoluble metal complexes [5b].

Based on the above and following our continuous interest in the use of xanthines for the development of transition metal complexes and their applications as efficient catalysts, we hypothesize on merging a theophylline moiety with the well-known phosphinite motif in a single ligand to prepare a series of Pd(II) complexes, where the metal center is stabilized by the phosphinite ligands, leaving free the theophylline fragment which could enhance the solubility of the complexes in water and, consequently, their catalytic activity in the Suzuki-Miyaura reaction in water promoted by microwaves. In the last 20 years, phosphinites [8] have become important chemical motifs in the design of highly active catalysts since they form strong bonds with most transition metals

* Corresponding author.
E-mail address: damor@unam.mx (D. Morales-Morales).

https://doi.org/10.1016/j.ica.2022.121365
Received 6 September 2022; Received in revised form 28 November 2022; Accepted 23 December 2022
Available online 27 December 2022



Scheme 1. A) coordination sites of theophylline. b) complexes prepared in this work.

[9], while the Suzuki-Miyaura C—C cross-coupling reaction is probably one of the most important and widely used catalytic transformation for industrial and academic purposes, as it provides a simple method for the production of Csp^2-Csp^2 bonds from inexpensive starting materials *i.e.* aryl halides and boronic acids/esters [10].

2. Results and discussion

2.1. Synthesis and characterization of Pd(II) complexes based on phosphinite-theophylline.

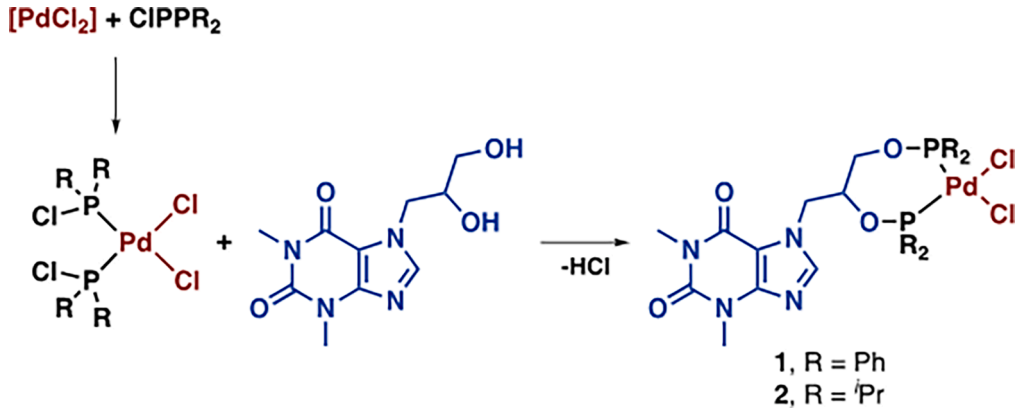
The synthesis of the Pd(II) complexes was carried out in a single pot by reacting $PdCl_2$ with the corresponding chlorophosphine in refluxing THF for 1 h. Then, 7-(2,3-dihydroxypropyl)theophylline was added to this reaction. The resulting reaction mixture was allowed to proceed for one hour, after which time a white solid containing the desired product was observed (Scheme 2). Complexes (**1**) and (**2**) were obtained in 87 % (**1**) and 95 % (**2**) yields, respectively. Both complexes were characterized by NMR spectroscopic techniques, mass spectrometry and elemental analyses.

The ^{1}H NMR spectra of the complexes showed the characteristic signals of the aliphatic protons of the theophylline *core* as singlets at upper field. For complex (**1**) these signals were shown at 3.21 and 2.50 ppm, while for complex (**2**) they were displayed at 3.59 and 3.39 ppm. In both complexes, the signal due to the NC*H*N fragment appears around 7.5 ppm. Other common protons exhibiting signals in the spectra are those corresponding to the aliphatic chain linking the theophylline *core* with the phosphinite fragments. These groups of signals are observed as multiplets between 4 ppm and 5 ppm. The major difference in the spectra of complex (**1**) and (**2**) is because the functional groups attached to phosphine atoms. In complex (**1**), the signals assigned to the phenyl group of the PPh_2 moiety are observed between 7.2 and 8.0 ppm. For complex (**2**), the signals due to the $-CH_3$ group of the P^iPr_2 fragment are observed as a set of doublets between 1.0 and 2.0 ppm. In a similar way, the signals produced by the –CH- fragments of the isopropyl functionality are shown as multiplets between 2.0 and 3.0 ppm. The latter should be expected due to the fact that the two P^iPr_2 fragments are not equivalent, so the isopropyl functionalities are found in a different chemical environment.

The $^{13}C\{^1H\}$ NMR spectra of the complexes showed the expected pattern by displaying the signals of the 7-(2,3-dihydroxypropyl) theophylline, and the corresponding signals of the PPh_2 and P^iPr_2 moieties. Complex (**1**) showed the signals of unsaturated carbons of the theophylline *core* at 105.9, 150.0 150.8, 151.4 and 154.2 ppm, and the signal of the $-CH_3$ groups at 27.5 and 29.4 ppm. The signals due to the 2,3-dihydroxypropyl moiety were observed at 47.1, 62.5 and 73.9 ppm. All the previously mentioned signals appeared in a similar chemical shift in the $^{13}C\{^1H\}$ NMR spectra of complex (**2**). The main difference between the two $^{13}C\{^1H\}$ NMR spectra was the signals due to the PPh_2 and P^iPr_2 moieties. For complex (**1**) the phenyl rings produced a set of signals due to the aromatic protons appearing at 125 – 135 ppm, while for complex (**2**) several signals in the region of 15 – 35 ppm were assigned to the isopropyl moiety (see SI for further details). The latter was expected since the isopropyl moieties are not chemically equivalent, and also due to the C—P coupling.

The $^{31}P\{^1H\}$ NMR spectra provided further information about the molecular structure of the complexes. The spectra of both complexes (**1**) and (**2**) displayed a typical AB pattern. The latter indicating that each phosphorus atom are coupled to palladium. Complex (**1**) showed two doublets at 119.4 and 119.0 ppm ($^2J_{P\text{-}P}$ = 40.4 Hz), while for complex (**2**) these signals were observed at 182.1 and 155.4 ppm ($^2J_{P\text{-}P}$ = 49.0 Hz). The coupling constant values are typical for this kind of systems [12]. In addition, the mass spectra of the complexes were measured in $DART^+$ mode, both spectra being very clean and exhibiting the molecular ions $[M-Cl]^+$ at 763 and 629 *m/z* for (**1**) and (**2**), respectively. Finally, elemental analyses were performed for samples of both compounds, with results agreeing with the proposed formulations.

The stability of the complexes was determined by $^{31}P\{^1H\}$ NMR. For this purpose, we separately prepared and kept under air a DMSO-d_6 and $CDCl_3$ solutions of both complexes (**1**) and (**2**), respectively. Then, we collected their NMR spectra after two days. To our delight we obtained the same spectrum for both complexes without any apparent change neither physically in the solution nor in the spectra (no extra signals were observed). The latter indicating that the complexes are stable in



Scheme 2. Synthesis of Pd(II) complexes including hybrid phosphinite-theophylline ligands.

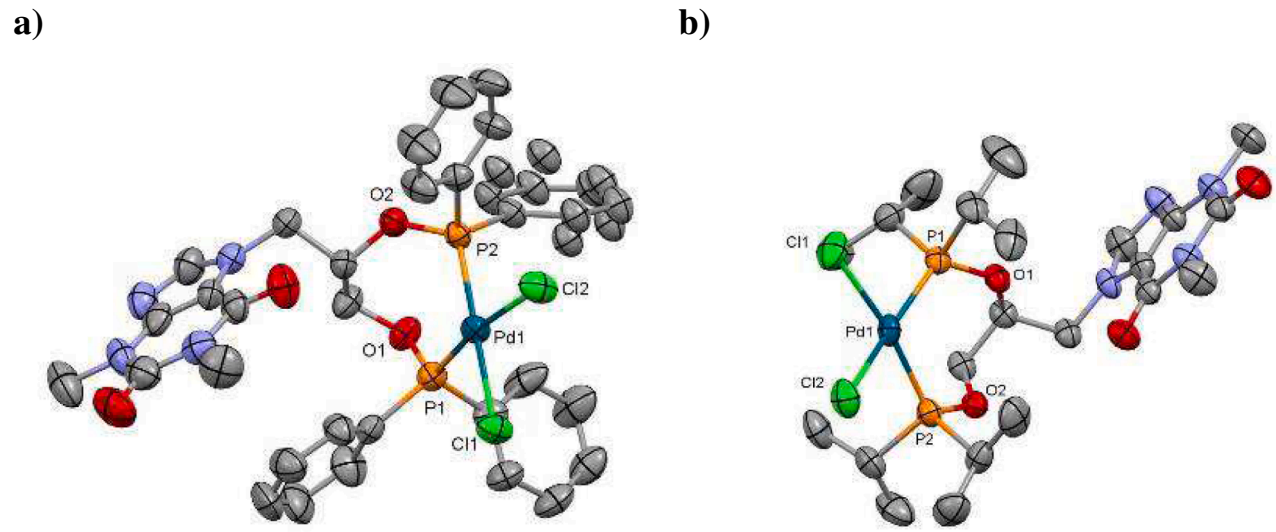


Fig. 1. Molecular structure of a) (**1**) and b) (**2**). The ellipsoids are represented at 50 % probability and hydrogen atoms have been omitted for clarity. *Selected bond lengths (Å):* (**1**) = Pd1-Cl1 2.3495(19), Pd1-Cl2 2.364(2), Pd1-P1 2.231(2), Pd1-P2 2.237(2). (**2**) = Pd1-Cl1 2.3709(19), Pd1-Cl2 2.3636(17), Pd1-P1 2.2541(17), Pd1-P2 2.2403(18). *Selected bond angles (°):* (**1**) = P1-Pd1-P2 94.90(8), P1-Pd1-Cl1 84.39(7), P2-Pd1-Cl1 178.94(9), P1-Pd1-Cl2 169.57(9), P2-Pd1-Cl2 88.98(8), Cl1-Pd1-Cl2 91.59(7). (**2**) = P1-Pd1-P2 100.43(7), P1-Pd1-Cl1 85.99(7), P2-Pd1-Cl1 170.97(4), P1-Pd1-Cl2 172.08(4), P2-Pd1-Cl2 82.68(7), Cl1-Pd1-Cl2 91.79(8).

solution and in the presence of air and moisture at least for 48 h. Also, the coordination of the phosphinites to Pd(II) reduces dramatically the strong tendency of the phosphinites to be oxidized and hydrolyzed under air. All our attempts to isolate and characterize the neat phosphinte ligands were unfruitful, observing the formation of several phosphine oxides and recovering the starting material 7-(2,3-dihydroxypropyl) theophylline.

Suitable crystals of both complexes for their analyses by single crystal X-ray diffraction techniques were obtained. The molecular structures of (**1**) and (**2**) are shown in Fig. 1. Complex (**1**) crystalized in a triclinic system with a P-1 space group, while complex (**2**) crystalized in a monoclinic system with a $P2_1/c$ space group (Table 1). Both complexes were isostructural; exhibiting the two phosphinite fragments coordinated to the Pd(II) center, and two chlorine atoms completing the coordination sphere of the metal. This coordination fashion generates a distorted square planar geometry around the Pd(II) center. The Pd-Cl lengths in both complexes (**1**) and (**2**) were very similar (2.4 Å), while the Pd-P lengths in complex (**1**) (~2.25 Å) were slightly longer than those observed in complex (**2**) (~2.36 Å). This slight difference is probably because alkyl-phosphines are better donors than aryl-phosphines.

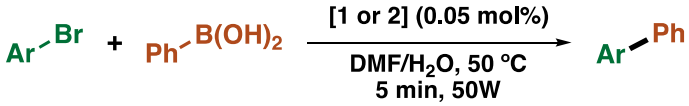
2.2. Catalytic evaluation of the Pd(II) complexes in the Suzuki-Miyaura cross-coupling reaction.

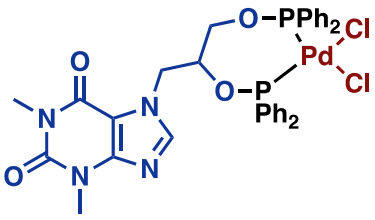
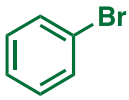
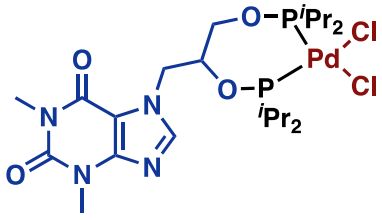
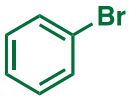
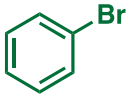
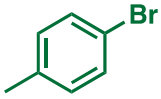
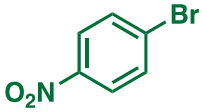
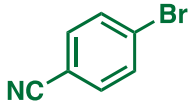
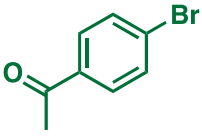
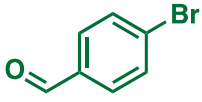
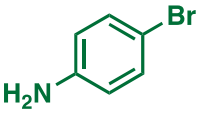
With the complexes on hand, we use them as catalysts in the Suzuki-Miyaura couplings of bromobenzene and phenylboronic acid using Na_2CO_3, DMF/H_2O (1:1), and 1 mol % of the corresponding complex. The reaction was heated at 110 °C and irradiated with microwaves at 150 W for 20 min. Under these reaction conditions, complex (**2**)

Table 1
Crystal data and structure refinement for (**1**) and (**2**).

	(**1**) (CCDC 2061459)	(**2**) (CCDC 2061460)
Empirical formula	$C_{34}H_{32}Cl_2N_4O_4P_2Pd$	$C_{22}H_{40}Cl_2N_4O_4P_2Pd$
Formula weight	799.87	663.82
Temperature	298(2) K	298(2)
Wavelength	0.71073 Å	0.71073 Å
Crystal system	Triclinic	Monoclinic
Space group	*P*-1	*P*21/*c*
Unit cell dimensions	a = 8.6202(10) Å α = 77.467(3)°	a = 9.212(6) Å α = 90°
	b = 10.4614(11) Å β = 87.278(3)°	b = 14.067(13) Å β = 92.92(5)°
	c = 19.891(2) Å γ = 76.982(3)°	c = 22.539(15) Å γ = 90°
Volume	1706.0(3) $Å^3$	2917(4)
Z	2	4
Density (calculated)	1.557 Mg/m^3	1.512
Absorption coefficient	0.839 mm^{-1}	0.963
F(000)	812.0	1368.0
Crystal size	0.208 × 0.085 × 0.060 mm	0.336 × 0.050 × 0.041
Theta range for data collection	4.976 to 50.618°	4.428 to 50.806
Index ranges	$-10\leq h\leq 10$, $-12\leq k\leq 12$, $-23\leq l\leq 23$	$-10\leq h\leq 11$, $-16\leq k\leq 15$, $-26\leq l\leq 27$
Reflections collected	13,453	30,832
Independent reflections	6130 [R(int) = 0.0940]	5339 [R(int) = 0.0551]
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Data / restraints / parameters	6130 / 174 / 472	5339 / 0 / 326
Goodness-of-fit on F^2	0.980	1.028
Final R indices [I > 2sigma(I)]	R1 = 0.0616, w*R*2 = 0.1135	R1 = 0.0419, w*R*2 = 0.0919
R indices (all data)	R1 = 0.1365, w*R*2 = 0.1454	R1 = 0.0711, w*R*2 = 0.1024
Largest diff. peak and hole	0.60 and −0.75 e.$Å^{-3}$	1.25 and −0.39 e.$Å^{-3}$

Table 2
Suzuki-Miyaura cross-coupling by complexes (**1**) and (**2**).[a]

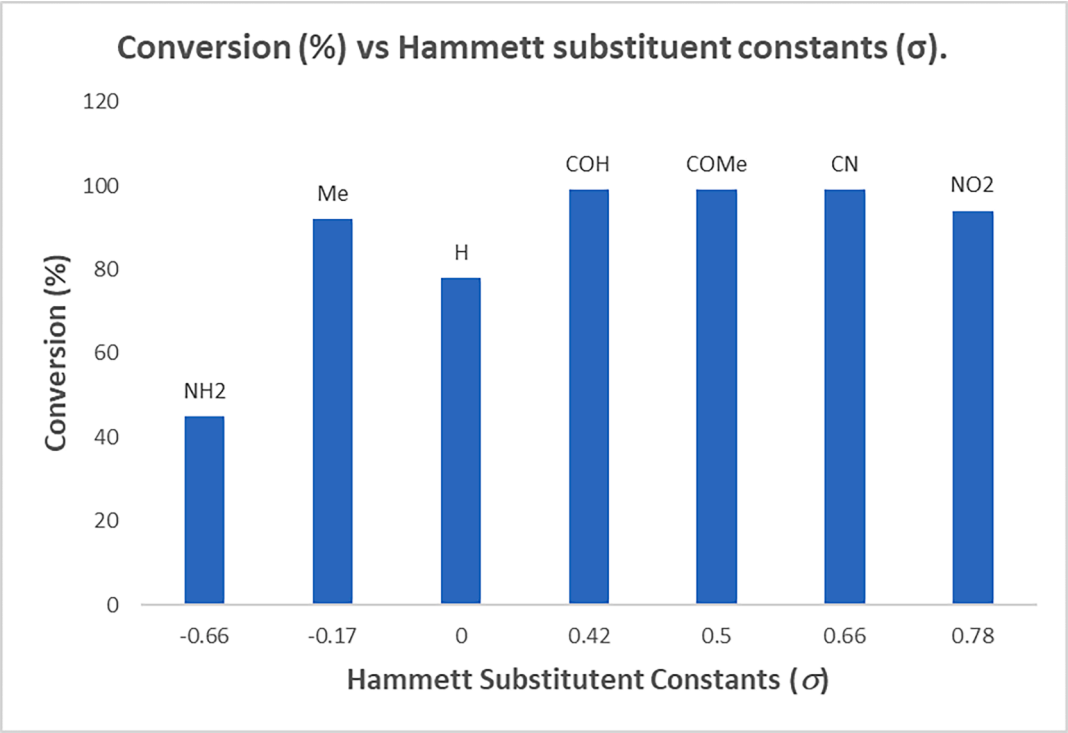


Entry	Complex	Ar-Br	Conversion (%)[b]
1	 **1**		80[c]
2	 **2**		>99[c]
3			78
4			92
5			94
6			>99
7			>99
8			>99
9			45

[a]General reaction conditions: Phenyl boronic acid (0.637 mmol), aryl bromide (0.637 mmol), Na_2CO_3 (0.637 mmol), catalyst (0.05 % mol), 5 mL of DMF/H_2O (1:1), 50 °C, 50 W, 5 min. [b]Conversions obtained by GC–MS are based on residual aryl bromide and the average of two runs. [c]Catalyst (1 % mol), 110 °C, 150 W, 20 min.

afforded > 99 % yield, while complex (**1**) only produced a yield of 80 % (Table 2). This behavior is most likely because the alkyl phosphinite ligands are better donors than the aryl-phosphinite ones. In addition, isopropyls have a higher steric hindrance than the phenyl fragments. The latter is important since bulky and strong donor ligands favor the oxidative addition and reductive elimination steps in cross-coupling processes [11].

With the purpose of optimizing the reaction conditions, we decreased the load of catalyst, time, temperature, and power of the microwave irradiation. Thus, we carried out the reaction using 0.05 mol % of complex (**2**), at 50 °C and irradiated by microwave at 50 W for 5 min. Under these new conditions the yield of the reaction between bromobenzene and phenylboronic acid yielded 78 % of biphenyl. To the best of your knowledge, the Suzuki-Miyaura couplings are traditionally catalyzed by using catalyst loadings often from 1 to 5 mol% [12]. Although there are some interesting reports using catalyst loadings in the range of ppm [13] and ppb [14], the reaction conditions are often harsher.



Graphic 1. Conversion (%) vs Hammett substituent constants (σ).

Motivated by this result, we explored the catalytic activity towards different *para*-substituted bromobenzenes under the above optimized conditions (Table 2). The results obtained suggested that using electron-withdrawing groups such as –CHO, –$COCH_3$, –CN, and –NO_2 (Graphic 1) produced higher yields up to > 99 %. Conversely, when using electron-donor substituents, such as –NH_2, the yield was reduced to only 45 % (Graphic 1). These results being in line with our previous reports, in the sense that substrates with electron-withdrawing groups promote this catalytic reaction. Trend that is better illustrated in the following graphic of Conversion (%) vs Hammett Substituent Constant (σ) [15] (Graphic 1).

3. Conclusions

In summary, we have synthesized and characterized two novel Pd(II) phosphinite complexes derived from theophylline. The molecular structures of (**1**) and (**2**) were unambiguously determined by single crystal X-ray diffraction analysis. Their structures show the phosphinite ligands to be coordinated to the metal center in a chelate manner, and two chlorine ligands completing the coordination sphere around the Pd (II) atom. Complex (**2**) was the most active catalyst in the Suzuki-Miyaura cross-coupling reactions. The catalytic reaction was performed under mild conditions (50 °C, 50 W, 5 min), using a low catalyst loading (0.05 mol %). Thus, this is a very attractive low-cost catalyst for the C—C cross-coupling reaction. Efforts aimed to test this catalytic system in other related cross coupling reactions, using different substrates and explore the further reactivity of these ligands with other metals also to produce NHC moieties are currently under development in our laboratory and will be disclosed in due time.

4. Experimental part

4.1. General

All chemical compounds were commercially obtained from Aldrich Chemical Co. and used as received without further purification. The ^{1}H, $^{13}C\{^{1}H\}$, $^{31}P\{^{1}H\}$ NMR spectra were recorded on a Bruker Ascend 500 spectrometer. Chemical shifts are reported in ppm down field of TMS using the residual signals in the solvent as internal standard. Elemental analyses were performed on a Perkin Elmer 240. CHNS analyses were performed in a Thermo Scientific Flash 2000 elemental analyzer, using a Mettler Toledo XP6 Automated-S Microbalance and sulfanilamide as standard (Thermo Scientific BN 217826, attained values N = 16.40 %, C = 41.91 %, H = 4.65 %, and S = 18.63 %; certified values N = 16.26 %, C = 41.81 %, H = 4.71 %, and S = 18.62 %). MS-DART experiments were recorded on a JEOL AccuTOF JMS-T100LC mass spectrometer. Melting points were carried out on Mel-Temp® Digital Melting Point Apparatus using open capillary tubes with a resolution of ± 1 °C and are reported without correction. Quantitative analyses by GC–MS were performed on an Agilent 6890 N GC with a 30.0 m DB-1MS capillary column coupled to an Agilent 5973 Inert Mass Selective detector.

4.2. General synthesis of the Pd(II) complexes.

A mixture of $PdCl_2$ (100 mg, 0.56 mmol) and the corresponding chlorophosphine (1.69 mmol) in THF was refluxed for 1 h. After this time, the solution was cooled to room temperature. Then, 7-(2,3-dihydroxypropyl)theophylline (143 mg, 0.56 mmol) was added in one portion and the solution was refluxed for 1 h. The resulting solid was filtered and washed several times with small portions of diethyl ether and hexane.

4.3. Synthesis of complex (1).

For the synthesis of (**1**), chlorodiphenylphosphine (373 mg, 1.69 mmol) was used. Yield: 392 mg (87 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 – 7.85 (m, 2H), 7.83 (s, 1H), 7.76 – 7.57 (m, 8H), 7.55 – 7.37 (m, 8H), 7.36 – 7.26 (m, 2H), 4.90 – 4.85 (m, 1H), 4.58 – 4.44 (m, 1H), 4.38 – 4.19 (m, 3H) 3.39 (s, 3H), 3.21 (s, 3H). $^{13}C\{^{1}H\}$ NMR (126 MHz, $CDCl_3$) δ 159.7, 156.0, 153.4, 148.15, 139 – 133 (m), 111.1, 79.1, 72.2, 71.5 (d, J = 5.8 Hz), 60.1, 34.6, 32.7. $^{31}P\{^{1}H\}$ NMR (202 MHz, $CDCl_3$) δ 124.1 (d, J = 40.5 Hz), 123.7 (d, J = 40.5 Hz). MS (DART): Calcd.

799.87; found *m/z* 763 $[M-Cl]^+$. Elem. Anal. Calcd. for $C_{34}H_{32}Cl_2N_4O_4P_2Pd$ C, 51.05; H, 4.03; N, 7.00;. Found: C, 51.02; H, 4.01; N, 6.88. Melting Point: 280 °C.

4.4. Synthesis of complex (2).

For the synthesis of (**2**), chlorodiisopropylphosphine (258 mg, 1.69 mmol) was used. Yield: 355 mg (95 %). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (s, 1H), 4.86 – 4.66 (m, 1H), 4.54 – 4.38 (m, 2H), 4.13 – 3.97 (m, 1H), 3.59 (s, 3H), 3.39 (s, 3H), 3.16 – 3.03 (m, 1H), 2.89 – 2.80 (m, 1H), 2.57 – 2.40 (m, 1H), 1.80 – 1.62 (m, 6H), 1.62 – 1.36 (m, 9H), 1.32 – 1.14 (m, 8H), 1.12 – 0.92 (m, 3H). ^{13}C{^{1}H} NMR (126 MHz, $CDCl_3$) δ 155.3, 151.4, 149.3, 142.0, 106.7, 74.3 (d, J = 8.9 Hz), 65.5 – 64.7 (m), 49.2 (d, J = 5.8 Hz), 33.5 (d, J = 39.6 Hz), 32.6 – 32.1 (m), 31.5 (d, J = 35.4 Hz), 30.0, 28.1, 25.4, 22.4, 20.9 (d, J = 21.2 Hz), 19.0 – 18.6 (m), 17.9 (d, J = 2.8 Hz), 17.5 (d, J = 4.4 Hz), 16.4 (d, J = 4.9 Hz). ^{31}P{^{1}H} NMR (202 MHz, $CDCl_3$) δ 182.1 (d, J = 49.1 Hz), 155.4 (d, J = 49.0 Hz). MS (DART): Calcd. 663.82; *m/z* 629 $[M-Cl]^+$. Elem. Anal. Calcd. for $C_{22}H_{40}Cl_2N_4O_4P_2Pd$: C, 39.80; H, 6.07; N, 8.44. Found: C, 40.03; H, 6.10; N, 8.49. Melting Point: 250 °C.

4.5. General procedure for the Suzuki-Miyaura cross-coupling.

A solution of phenyl boronic acid (0.637 mmol), aryl bromide (0.637 mmol), Na_2CO_3 (0.637 mmol), and the corresponding catalyst (0.05 % mol) in 5 mL of DMF/H_2O (1:1) was heated at 50 °C and irradiated with microwave (50 W). After the prescribed reaction time, the resulting reaction mixture was cooled at room temperature and the organic phase analyzed by gas chromatography (GC–MS).

4.6. Mercury drop experiments [16]

Following the above-described procedures; adding two drops of elemental Hg to the reaction mixture. After the prescribed reaction times, the solution was filtered and analyzed by GC–MS: no significant difference in conversion between these experiments and those in the absence of mercury was observed, indicating that heterogeneous Pd(0) is not involved.

4.7. Data collection and refinement for (1) and (2)

Colorless plates for complex **1** (CCDC 2061459) and complex **2** (CCDC 2061460), were grown independently from CH_2Cl_2/diethyl ether (liquid/liquid slow diffusion) and mounted on glass fibers, then complex **1** was placed on a Bruker Smart Apex II diffractometer and complex **2** mounted on Bruker D8 Venture \k-geometry diffractometer, with a Mo-target micro-focus X-ray source (λ = 0.71073 Å). The detector was placed at a distance of 5.0 cm from the crystals frames were collected with a scan width of 0.5 in ω and an exposure time of 10 s/frame. Frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm [17]. Non-systematic absences and intensity statistics were used in triclinic space group. The structures were solved using Patterson methods using SHELXS-2014/7 program [18]. The remaining atoms were located via a few cycles of least squares refinements and difference Fourier maps. Hydrogen atoms were input at calculated positions and allowed to ride on the atoms to which they are attached. Thermal parameters were refined for hydrogen atoms on the phenyl groups using a Ueq = 1.2 Å and a Ueq = 1.5 Å for methyl groups to precedent atom in all cases. For all complexes, the final cycle of refinement was carried out on all non-zero data using SHELXL-2014/7 [18]. Absorption correction was applied using SADABS program [19].

A phenyl ring on complex **1** was disordered and modeled in two major positions, using a site occupational factor (SOF). The ratio of SOF was 0.6/0.4 for phenyl atom disordered. 174 restraints were applied, 150 for SIMU and 24 for DELU, and 3 reflections were omitted.

CRediT authorship contribution statement

Edgar Marín-Carrillo: Investigation, Formal analysis, Writing – original draft. **Hugo Valdés:** Investigation, Formal analysis, Writing – original draft. **Simón Hernández-Ortega:** Investigation, Formal analysis, Writing – original draft. **David Morales-Morales:** Conceptualization, Methodology, Formal analysis, Validation, Writing – original draft, Writing – review & editing, Visualization, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgments

We would like to thank Dr. Francisco Javier Pérez Flores, Q. Eréndira García Ríos, M.Sc. Lucia del Carmen Márquez Alonso, M.Sc. Lucero Ríos Ruiz, M.Sc. Alejandra Núñez Pineda (CCIQS), Q. María de la Paz Orta Pérez, Q. Roció Patiño-Maya and Ph.D. Nuria Esturau Escofet for technical assistance. H.V. thanks CONACYT (CVU: 410706) and Generalitat de Catalunya (Beatriu de Pinós MSCA-Cofund 2019-BP-0080). E. M.-C. would like to thank CONACYT (No. de becario: 336120) for Ph.D. scholarship. The financial support of this research by PAPIIT-DGAPA-UNAM (PAPIIT IN210520) and CONACYT A1-S-33933 is gratefully acknowledged.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ica.2022.121365.

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