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Selective head-to-tail coupling of methyl phenylpropynoate providing palladacycles with bidentate N-ligands

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Dedicated to Professor Dr. G. van Koten, in friendship and in recognition of his many outstanding contributions to organometallic chemistry.

Abstract

The complexes (1,3-dicarbomethoxy-2,4-diphenylbuta-1,3-dien-1,4-diyl)(*N*,*N*)palladium(II), where *N*,*N* = 2,2′-bipyridyl (**1**), tetramethylethelenediamine (**2**), 1,10-phenanthroline (**3**) have been obtained by reaction of $Pd(dba)_2$ with the respective bidentate N-ligand and two equivalents of methyl phenylpropynoate via a completely regioselective head-to-tail coupling of the asymmetric acetylenes. Such regioselectivity, especially in conjunction with the high yield, is very unusual in the formation of palladacycles and has so far only been observed for head-to-head or tail to tail coupling. The compounds **1**–**3** have been characterized by elemental analyses, NMR spectra and single crystal X-ray diffraction studies for **2** and **3**. The X-ray crystal structures reveal pseudo square planar metal centers, the palladacycles and chelate rings are essentially planar.

Keywords: Palladacyclopentadiene; Regioselectivity; Bidentate N-ligands; Methyl phenylpropynoate; Crystal structure

1. Introduction

Metallacyclic compounds have attracted much recent attention, especially those formed from unsaturated ligands such as alkenes and alkynes. Their interest stems principally from studies concerning the mechanism of transition metal catalyzed oligomerization reactions.

The synthesis of palladacyclopentadienes bearing electron withdrawing substituents has been reported previously [1]. Contrary to other metals, where usually no metallacycles have been observed [2,3], palladium permits mechanistic investigations due to the lower reactivity of palladium (η^2-alkyne) complexes and palladacycles that have been observed in this and in previous cases [4]. These compounds were found to be intermediates in the Pd(0)-catalyzed cyclotrimerization of acetylenes [5], dimerization-carbostannylation of alkynes [6] and catalytic conversion of alkynes to conjugated (*Z*,*Z*)-dienes (Scheme 1) [7].

In many instances, dissymmetric alkynes have been employed, which give up to three isomeric metallacycles (Scheme 2) and two isomeric benzene derivatives as the trimerization products [8,9]. The catalytic three-component synthesis of conjugated (*Z*,*Z*)-dienes appeared to be limited to the use of symmetric (and electron-deficient) alkynes; all attempts employing dissymmetric alkynes were unsuccessful [4].

The regiochemical outcome of the reactions involving dissymmetric alkynes is difficult to predict a priori, due to the two possible, often rather similar, orientations of the incoming alkyne during the insertion into the

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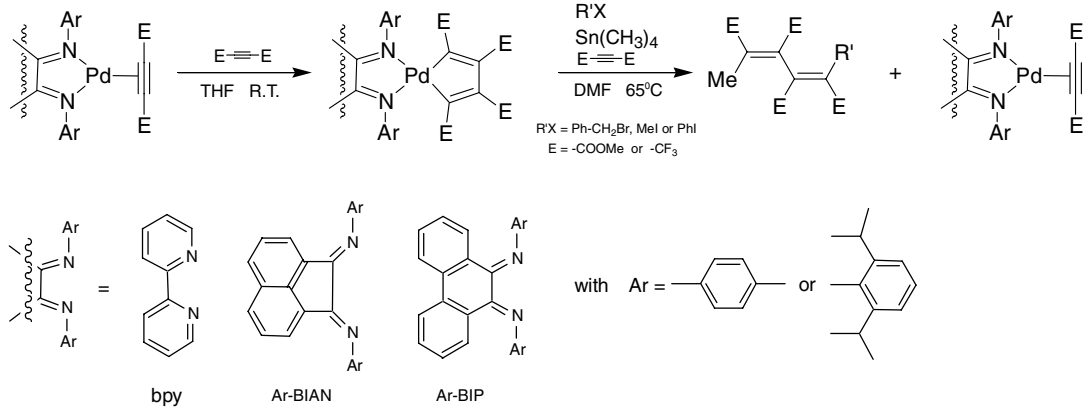
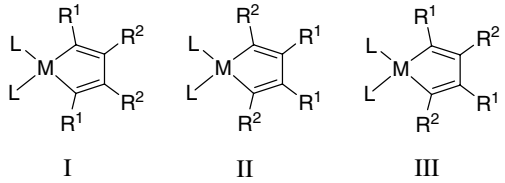


Scheme 1.



Scheme 2.

M(η^2-alkyne) likely intermediate. We endeavoured to study the feasibility and selectivity, if any, of the formation of palladacyclopentadienes starting from dissymmetric alkynes. In this contribution, we describe our results of the regioselective formation of head-to-tail palladacycles involving methyl phenylpropynoate (Ph—C≡C—CO_2CH_3).

2. Experimental

2.1. General procedures

All reactions were performed in Schlenck tubes under nitrogen unless otherwise specified. Solvents were dried and distilled under nitrogen prior to use: diethyl ether, *n*-hexane and tetrahydrofuran over sodium, dichloromethane over calcium hydride.

All NMR spectra were recorded at 298 K. The ^{1}H and ^{13}C {^{1}H} NMR were recorded on a Bruker AMX 300 spectrometer (300.13 and 75.18 MHz, respectively). The chemical shifts are referenced to TMS via the residual solvent peaks. In the cases of **1** and **3** the assignments of the ^{1}H NMR and ^{13}C {^{1}H} NMR signals were made with the aid of COSY and HETCOR. Chemical shifts (δ) and coupling constants (J) are expressed in ppm and in Hertz (Hz), respectively.

The starting materials $Pd(dba)_2$ [10] (dba = dibenzylidenacetone), 4-$MeOC_6H_4$-bian [11,12] were prepared according to the literature procedures. Methyl phenylpropynoate, dimethyl-2-butynedioate (dmbd), 2,2′-bipyridyl,1,10-phenanthroline were obtained commercially and were used without further purification. *N*,*N*-tetramethylethylenediamine (tmeda) was obtained commercially and was distilled prior to use.

2.2. Synthesis of pallada-2,4-bis(carbomethoxy)-3,5-diphenylcyclopentadiene-bipyridyl (*1*)

A suspension of $Pd(dba)_2$ (200 mg, 0.35 mmol), 2,2′-bipyridyl was stirred at room temperature for 10 min in THF (20 mL). Then methyl phenylpropynoate (134 μL, 0.90 mmol) was added to the solution. After 2 h, the solvent of the resulting orange solution was removed in vacuo and the product was washed with diethyl ether (4 × 50 mL). The orange product was dissolved in dichloromethane and filtered over celite filter aid in order to remove the metallic palladium. The solvent was again removed in vacuo, yielding 145 mg (0.25 mmol, 72%) of yellow orange product.

1. *Anal*. Calc. for $C_{30}H_{24}N_2O_4Pd$: C, 61.81; H, 4.15; N, 4.81. Found: C, 61.64; H, 4.22; N, 4.88%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.80 (d, 1H, H6, $^3J = 5.5$), 7.99 (m, 2H, H4 and H6′), 7.95 (d, 1H, H3, $^3J = 8.0$), 7.82 (dd, 1H, H5′, $^3J = 7.6$, $^3J = 7.5$), 7.50 (dd, 1H, H5, $^3J = 5.3$), 7.41 (dd, 2H, $H_{o'}$, $^3J = 7.0$), 7.35 (dd, 2H, H_o, $^3J = 7.0$), 7.27–7.16 (m, 6H, $H_{m,m'}$ and $H_{p,p'}$), 7.02 (dd, 1H, H4′, $^3J = 6.5$), 6.98 (d, 1H, H3′, $^3J = 5.0$), 3.42 (s, 3H, OCH_3), 3.17 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$, 75.48 MHz): δ 177.1 (*C*O), 175.5 (*C*O′), 166.5 (C_β), 156.9 ($C_{\alpha'}$), 155.4 and 155.2 (2C, C2 and C2′), 151.9 (C3), 151.6 (C6′), 149.0 ($C_{\beta'}$), 147.5 ($C_{i'}$), 146.9 (C_α), 140.1 (C_i), 139.3 (C6), 138.8 (C5), 128.2, 128.0, 127.8 and 127.6 (4 × 2C, $2C_o$, $2C_{o'}$, $2C_m$ and $2C_{m'}$), 126.5 and 126.4 (2C, C_p and $C_{p'}$), 126.0 (C5′), 125.6 (C4), 122.4 and 122.0 (2C, C3′ and C4′), 51.0 and 50.8 (2C, O*C*H_3 and O*C*H_3').

2.3. Synthesis of pallada-2,4-bis(carbomethoxy)-3, 5-diphenylcyclopentadiene-tmeda (2)

The procedure was the same as for **1**: $Pd(dba)_2$ (200 mg, 0.35 mmol), tmeda (60 μL, 0.40 mmol) and methyl phenylpropynoate (140 μL, 0.95 mmol) gave **2** after 3 h reaction and washing with diethyl ether (2 × 50 mL) 85 mg (0.16 mmol, 45%) of yellow product.

2. *Anal*. Calc. for $C_{26}H_{32}N_2O_4Pd \cdot 0.5CH_2Cl_2$: C, 54.38; H, 5.68; N, 4.79. Found: C, 54.79; H, 5.62; N, 5.19%.

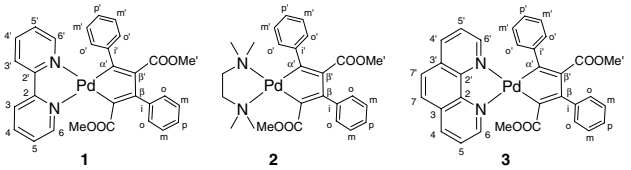
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.17 (m, 10H, ar), 3.32 (s, 3H, OCH_3), 2.98 (s, 3H, OCH_3), 2.59 (s, 6H, 2 × CH_3), 2.43 (s, 4H, 2 × CH_2), 1.95 (s, 6H, 2 × CH_3). ^{13}C NMR ($CDCl_3$, 75.48 MHz): δ 176.2 (*C*O), 173.0 (*C*O′), 166.5 (C_β), 155.8 ($C_{\alpha'}$), 148.5 (C_α), 148.3 ($C_{\beta'}$), 148.0 ($C_{i'}$), 140.4 (C_i), 127.7 (8C, 2 C_o, 2$C_{o'}$, 2 C_m and 2 $C_{m'}$), 126.3 and 125.6 (2C, C_p and $C_{p'}$), 62.0 and 61.3 (2C, *C*H_2 and *C*H'_2), 51.0 and 50.5 (2C, O*C*H_3 and O*C*H'_3), 48.8 and 48.7 (4C, 2 × *C*H_3 and 2 × *C*H'_3).

2.4. Synthesis of pallada-2,4-bis(carbomethoxy)-3, 5-diphenylcyclopentadiene-phenanthroline (3)

The procedure was the same as for **1**: $Pd(dba)_2$ (307 mg, 0.53 mmol), 1,10-phenanthroline (96 mg, 0.53 mmol) and phenyl methylpropynoate (200 μL, 1.36 mmol) gave **3** after 1.5 h reaction and washing with diethyl ether (2 × 50 mL) 195 mg (0.32 mmol, 61%) of yellow product.

3. *Anal*. Calc. for $C_{32}H_{24}N_2O_4Pd$: C, 63.32; H, 3.99; N, 4.62. Found: C, 63.26; H, 4.08; N, 4.55%.

^{1}H NMR ($CDCl_3$, 500 MHz): δ 9.10 (d, 1H, H6, $^3J = 5.0$), 8.45 (d, 1H, H4, $^3J = 8.0$), 8.29 (d, 1H, H6′, $^3J = 8.0$), 7.88 (s, 2H, H7 and H7′), 7.80 (dd, 1H, H5, $^3J = 7.8$, $^3J = 5.3$), 7.41 (d, 2H, $H_{o'}$, $^3J = 6.5$), 7.39 (d, 2H, H_o, $^3J = 7.0$), 7.30 (dd, 1H, H5′, $^3J = 7.8$, $^3J = 5.3$), 7.27–7.15 (m, 6H, $H_{m,m'}$ and $H_{p,p'}$), 7.08 (d, 1H, H4′, $^3J = 5.0$), 3.46 (s, 3H, OCH_3), 3.18 (s, 3H, OCH_3). ^{13}C NMR ($CDCl_3$, 75.48 MHz): δ 177.0 (*C*O), 175.2 (*C*O′), 166.4 (C_β), 156.7 ($C_{\alpha'}$), 152.0 (C4′), 151.6 (C6), 149.0 ($C_{\beta'}$), 147.8 ($C_{i'}$), 147.1 (C_α), 146.2 (2C, C2 and C2′), 140.2 (C_i), 138.4 (C4), 137.9 (C6′), 130.0 and 129.7 (2C, C3 and C3′), 130.9–125.9 (14C, 2 C_o, 2 $C_{o'}$, 2 C_m 2 $C_{m'}$, C_p, C'_p, C5, C5′, C7, and C7′), 51.0 and 50.8 (2C, O*C*H_3 and O*C*H'_3).



2.5. X-ray crystallographic structure determination of 2

Reflections were collected on a Kappa CCD diffractometer using Mo Kα graphite-monochromated radiation (λ = 0.71073 Å). The structure was solved using direct methods and refined against |*F*|. Hydrogen atoms were introduced as fixed contributors. For all computations, the Nonius OpenMoleN package was used [13].

2.6. X-ray crystallographic structure determination of 3

A crystal of **3** with dimensions 0.15 × 0.30 × 0.35 mm approximately was used for data collection on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Cu Kα radiation and ω–2θ scan. A total of 5350 unique reflections was measured within the range $-14 \leqslant h \leqslant 14$, $-15 \leqslant k \leqslant 0$, $0 \leqslant l \leqslant 22$. Of these, 4605 were above the significance level of $4\sigma(F_{obs})$ and were treated as observed. The range of $(\sin\theta)/\lambda$ was 0.049–0.626 Å ($4.3 \leqslant \theta \leqslant 74.7°$). Two reference reflections ($[\bar{3}\,\bar{1}\,1]$, $[1\,0\,3]$) were measured hourly and showed no decrease during the 91 h collecting time. Unit-cell parameters were refined by a least squares fitting procedure using 23 reflections with $40.21 \leqslant 2\theta \leqslant 44.69$. Corrections for Lorentz and polarization effects were applied. Absorption correction was performed with the program PLATON [14] following the method of North et al. [15] using Ψ-scans of five reflections, with coefficients in the range 0.673–0.984. The structure was solved by the PATTY option of the DIRDIF-99 program system [16].

The hydrogen atoms were calculated and kept fixed at their calculated positions with $U = 0.1$ Å^2. Full-matrix least-squares refinement on *F*, anisotropic for the non-hydrogen atoms and isotropic for the hydrogen atoms, converged to $R = 0.080$, $R_w = 0.091$, $(\Delta/\sigma)_{max} = 0.03$, $S = 1.04$. A weighting scheme $w = [13 + 0.01 * (\sigma(F_{obs}))^2 + 0.01/(\sigma(F_{obs}))]^{-1}$ was used. The secondary isotropic extinction coefficient [17,18] refined to $g = 1794(117)$. A final difference Fourier map revealed a residual electron density between −3.57 and 2.23 e Å^{-3} in the vicinity of the heavy atom and O2 and C26. Scattering factors were taken from Cromer and Mann [19,20]. The anomalous scattering of Pd was taken into account [21]. All calculations were performed with XTAL3.7 [22], unless stated otherwise.

Atoms O2 and C26 are highly anisotropic compared to the other atoms. This is maybe due to positional disorder in that OCH_3 moiety; no attempts were made to quantify this disorder.

3. Results and discussion

3.1. Synthesis and identification

Reaction of $Pd(dba)_2$ with the respective bidentate *N*,*N* ligand: tmeda, 2,2′-bipyridyl or 1,10-phenanthroline in the presence of 2 equivalents of methyl phenylpropynoate, gave the pure compounds **1**–**3** as unique regioisomers. The palladacycles **1**–**3** were synthesized in good yield (35–70%) via a completely regioselective

head-to-tail coupling of the two dissymmetric alkynes involved (Scheme 3). The typical reaction of $Pd(dba)_2$ with 2,2′-bipyridyl and methyl phenylpropynoate leads to reproducible results. The new compounds are air-stable solids which are very soluble in chloroform and dichloromethane. They have been analyzed by elemental analysis or mass spectrometry and by ^1H and ^{13}C NMR spectroscopy in solution. The formation of compounds **1**–**3** was usually instantaneous. In none of the cases studied could π-acetylenic intermediates be observed, which is probably due to the weak π-acceptor character of the ancillary ligands. It has been reported that $(L)_2Pd^0(\eta^2$-alkyne) complexes are more stable with increasing π-accepting capacity of the ligands L [23,24].

The most characteristic chemical shifts of all complexes are the ones that correspond to the methoxycarbonyl groups. They can act as a probe for the geometry of compounds **1**–**3**, i.e., symmetric complexes (of types **I** and **II**) show one peak for the methoxycarbonyls and the asymmetric one (type **III**) shows two resonances for the methoxycarbonyls. In the present cases, two signals due to the methoxycarbonyl groups are observed around 3.4 ppm in the ^1H NMR spectra. A low-frequency shift of about 0.4 ppm due to anisotropic shielding by the phenyl groups of the metallacycle is observed relatively to the pallada-2,3,4,5-tetrakis(carbomethoxy)-cyclopentadienes [25]. Therefore, we wondered whether it was a 50/50 mixture of complexes of types **I** and **II** or only the type **III**. The structure of compounds **1**–**3** was established on the basis of the anisochronicity of the protons located on two inequivalent halves of the N,N-ligand, which show mutual coupling. Furthermore, we observe only one set of resonances for each complex, proving that we are dealing with a single compound (type **III**) in each case and not with a mixture of regioisomers **I** and **II**.

X-ray crystal structure determinations were performed on pallada-2,4-bis(carbomethoxy)-3,5-bis(phenyl)-cyclopentadiene-tmeda **2** and its phenanthroline analogue **3**, which corroborated that their molecular structures are of type **III**. In most of the cases, metallacyclopentadienes similar to the ones presented are supposed to be intermediates in cyclotrimerization process, but just a few of them have been isolated and characterized so far.

3.2. X-ray crystal structures of *2* and *3*

The adopted numbering schemes of the molecular structures of **2** and **3** are depicted in Figs. 1 and 2, selected bond distances, bond angles and torsion angles have been compiled in Table 1.

Both complexes **2** and **3** are characterized by a distorted square-planar coordination around the metal center. The palladacycle itself and the chelate ring are essentially planar as indicated in **3** by the small deviations from the least-squares planes, which are 0.01(1) and 0.02(1) Å for C16 and C13, respectively. The distortion from square planarity is reflected by the dihedral angle between these planes of 21.6(3)°. It is normal for palladacyclopentadiene complexes, e.g., for (bpy)pallada-2,3,4,5-tetrakis(carbomethoxy)-2,4-cyclopentadiene and its phenyl-bip analogue [4] it was 15.6(4)° and 27.1(4)°, respectively. This is caused by an intermediate interaction of the phenanthroline, as compared to the bpy and phenyl-bip, with the ester group and the phenyl group on the palladacycle. The bite angle N12–Pd–N1

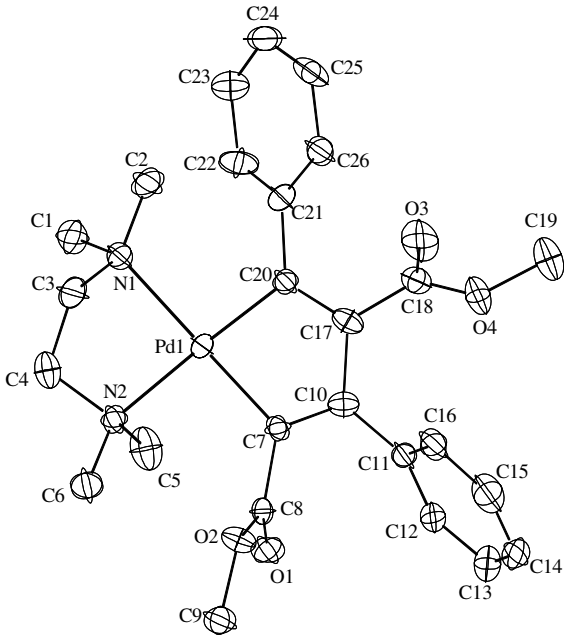
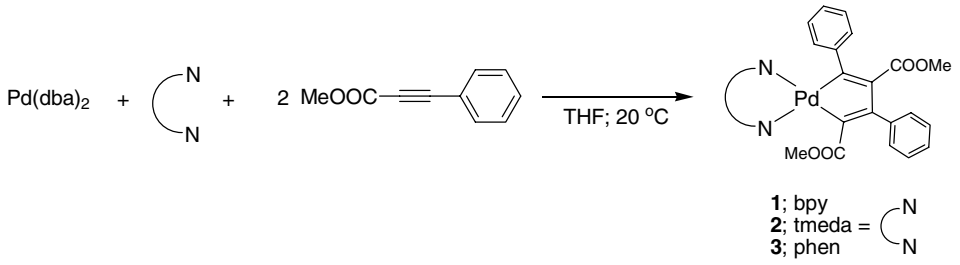


Fig. 1. Crystal structure of **2**. Ellipsoids are at the 30% probability level; hydrogen atoms are omitted for clarity.



Scheme 3.

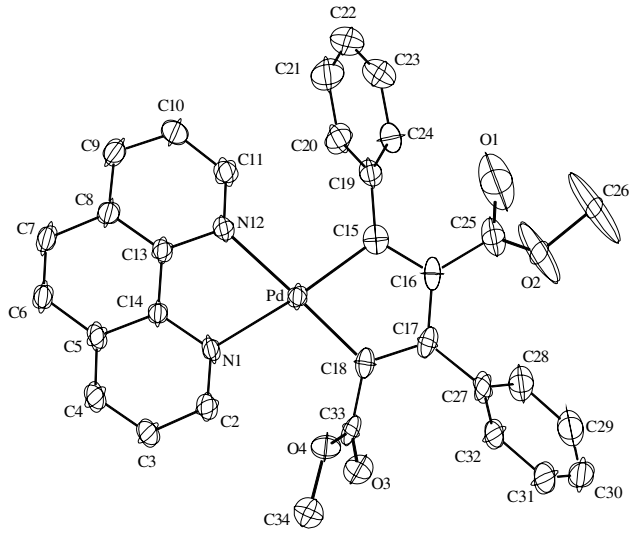


Fig. 2. Crystal structure of **3**. Ellipsoids are at the 30% probability level; hydrogen atoms are omitted for clarity.

Table 1
Crystal data and structure refinement for compounds **2** and **3**

	2	**3**
Empirical formula	$2[C_{26}H_{32}N_2O_4Pd]\cdot CH_2Cl_2$	$C_{32}H_{24}N_2O_4Pd$
Formula weight (g mol^{-1})	585.44	606.96
Temperature (K)	173	295
Crystal size (nm)	$0.12\times0.08\times0.08$	$0.35\times0.30\times0.15$
Wavelength (Å)	0.71073	1.54180
Crystal system	monoclinic	monoclinic
Space group	$P1c1$	$P2_1/n$
a (Å)	13.8537(1)	11.3922(8)
b (Å)	10.9551(1)	12.4860(6)
c (Å)	35.4177(2)	18.318(4)
β (°)	98.797(5)	90.57(2)
Volume (Å^3)	5312.06(7)	2605.5(6)
Z	2	4
Number of data measured	16264	5350
Number of data	11432 with $I>3\sigma(I)$	4605 with $F>4\sigma(F)$
Number of variables	1241	353
Goodness-of-fit on F	1.213	1.04
R	0.037	0.080
wR_2	0.062	0.091

of 78.1(3)° is somewhat smaller than the similar bite angle in the palladium bis(methoxycarbonyl) coordination compound $[Pd(phen)(CO_2CH_3)_2]$ [26] (82.5(3)°), due to a larger steric encumbrance of the palladacycle compared to the two mutually *cis* carbomethoxy groups (see Table 2).

3.3. Regioselectivity

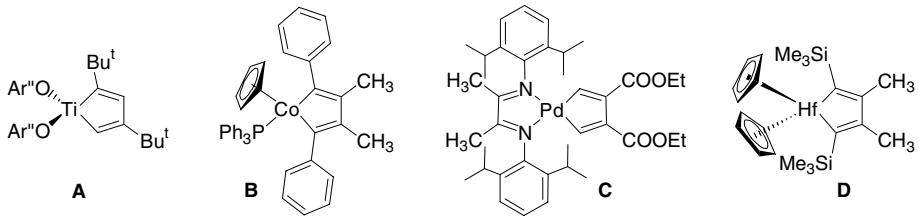
To the best of our knowledge there are only a few examples in the literature of comparable regioselectivity

Table 2
Selected bond lengths (Å), bond angles (°) and torsion angles (°) for **2** and **3**

2		**3**	
Atoms	*Distance*	*Atoms*	*Distance*
Pd1–N1	2.227(1)	Pd–N12	2.143(8)
Pd1–N2	2.229(6)	Pd–N1	2.194(7)
Pd1–C20	2.041(7)	Pd–C15	2.037(9)
Pd1–C7	2.032(7)	Pd–C18	1.997(9)
C3–C4	1.49(1)	C13–C14	1.427(12)
C20–C17	1.36(1)	C15–C16	1.390(13)
C17–C10	1.47(1)	C16–C17	1.454(13)
C10–C7	1.36(1)	C17–C18	1.386(13)
Atoms	*Bond angle*	*Atoms*	*Bond angle*
N1–Pd1–N2	80.9(2)	N12–Pd–N1	78.1(3)
C20–Pd1–C7	79.3(3)	C15–Pd–C18	80.8(4)
N1–Pd1–C7	179.5(3)	N12–Pd–C18	172.3(3)
N1–Pd1–C20	100.8(3)	N12–Pd–C15	101.3(3)
N2–Pd1–C7	98.9(3)	N1–Pd–C18	102.2(3)
N2–Pd1–C20	177.9(3)	N1–Pd–C15	162.0(3)
Pd1–N1–C3	104.4(5)	Pd–N12–C13	113.3(6)
Pd1–N2–C4	107.0(5)	Pd–N1–C14	111.8(6)
Pd1–C20–C17	115.0(5)	Pd–C15–C16	112.7(6)
Pd1–C7–C10	116.7(5)	Pd–C18–C17	117.0(7)
C20–C17–C10	115.9(6)	C15–C16–C17	117.2(8)
C7–C10–C17	113.0(6)	C18–C17–C16	112.3(8)
Atoms	*Torsion angle*	*Atoms*	*Torsion angle*
N1–C3–C4–N2	−58.1(7)	N12–C13–C14–N1	−2.3(13)
C20–C17–C10–C7	2.7(9)	C15–C16–C17–C18	1.5(12)
O3–C18–C17–C10	−120.3(9)	O1–C25–C16–C17	−153.0(14)
O1–C8–C7–C10	−105.3(8)	O3–C33–C18–C17	65.9(13)

in metallacyclopentadiene formation involving unsymmetric alkynes. Some bis(aryloxy)titanacyclopentadiene complexes (Scheme 4; **A**) exhibit such regioselectivity since only the head-to-tail coupled regioisomer is formed [27]. A [cobalt(η^5-cyclopentadienyl)(triphenylphosphine) (acetylene)] complex **B** [8] yields one regioisomer for the homocoupling product of 1-phenylprop-1-yne, but it is important to notice that the homocoupling occurs via a tail to tail coupling. However, with this cobalt complex, no regioselectivity was observed for methyl phenylpropynoate as the alkyne.

In the same way, the selective formation of a symmetrically substituted hafnacyclopentadiene (**D**), which presents the same regioisomer (tail to tail coupling), has been reported [28]. Concerning the formation of palladacycles from unsymmetric alkynes, selective head-to-tail coupling is very unusual; only head to head or tail to tail coupling has been observed. One example involving a symmetric palladacyclopentadiene supported by *N*-aryldiazadienes, regioselectively formed in low yield, via a head to head coupling due to the steric hindrance of the isopropyl group of the *N*-aryldiazadienes ligand, has been reported (**C**) [5]. The regioselectivity of the cyclization process in such cases appears to be governed by the steric factors of the substituents of the alkyne, rather than by electronic factors. The regioselectivity



Scheme 4.

in case of **1**–**3**, which has been obtained by head-to-tail coupling, has never been observed before.

It is clear that we did not observe any product(s) that stem from a homogeneous catalytic cyclotrimerization. Previously, apart from the case of dimethylbutynedioate [29], this has never been observed for palladium(0) either, except in the case of intramolecular process [30] or cyclotrimerization of arynes [31]. In all of those cases, only cyclo-*co*trimerization has been observed, by introducing a third alkyne that is different from the one present in the palladacycle, but such a reaction never proceeded under catalytic conditions [1a,5].

The integrity of compounds **1**–**3** in the presence of other alkynes [27] and their reactions with organic halides and organotin reagents will be the subject of future investigations.

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