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Carbon–carbon and carbon–chlorine bond formation on reaction of iodine(III) reagents with the bis(alkynyl)palladium(II) motif, and structural chemistry of *trans*-Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] and *trans*-[PdCl(C≡C-*o*-Tol)(PMe$_2$Ph)$_2$]

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ABSTRACT

Trans-di(*ortho*-tolylethynyl)bis(dimethylphenylphosphine)palladium(II) reacts above −20 °C with the iodonium reagent IPhCl$_2$ to give predominantly *o*-Tol–C≡C–Cl, above 15 °C with IPh$_2$(OTf) (OTf = triflate) to give *o*-Tol–C≡C–Ph and (*o*-Tol–C≡C)$_2$ in ca. 3:1 ratio, and above 10 °C with IPh(C≡CR)(OTf) (R = Bu^t, SiMe$_3$) to give predominantly *o*-Tol–C≡C–C≡C–R and (*o*-Tol–C≡C)$_2$. ^{31}P NMR spectra provide evidence for detection of intermediates. The complexes *trans*-[Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] and *trans*-[PdCl(C≡C-*o*-Tol)(PMe$_2$Ph)$_2$] are obtained on reaction of *trans*-[PdCl$_2$(PMe$_2$Ph)$_2$] with Li(C≡C-*o*-Tol) and *o*-Tol-C≡CH/Et$_3$N, respectively, and have been characterised by X-ray crystallography.

1. Introduction

Alkynylpalladium(IV) species have been proposed as intermediates in stoichiometric [1] and catalytic [2–9] organic synthesis, but spectroscopic detection is limited to NMR studies of the formation of very unstable Pd^{IV}IMe$_2$(C≡CSiMe$_3$)(dmpe) (dmpe = 1,2-bis(dimethylphosphino)ethane) and Pd^{IV}(OTf)(O$_2$CPh)(C≡CSiMe$_3$)(NCN) (OTf = triflate, NCN = 2,6-(Me$_2$NCH$_2$)$_2$C$_6$H$_3$ 'pincer') at −50 °C and −80 °C, respectively [8]. These complexes were obtained using IPh(C≡CSiMe$_3$)(OTf) as a strong oxidising agent via elimination of PhI and formal transfer of 'C≡CSiMe$_3^+$' to Pd^{II}. Organopalladium(III) complexes have also been well characterised by X-ray crystallography, including mononuclear [9] and Pd^{III}–Pd^{III} bonded species [10,11]. In view of an earlier report proposing undetected mono(alkynyl)palladium(IV) and bis(alkynyl)palladium(IV) intermediates in complex reactions of *trans*-[PdCl$_2$(PBu$_3$)$_2$] with SnMe$_3$(C≡CR) (R = Me, Bu^t) [12], we have explored the interaction of a typical *trans*-[Pd(alkynyl)$_2$(phosphine)$_2$] reagent with IPh(C≡CR)(OTf)(R = Bu^t, SiMe$_3$) and other iodine(III) reagents (IPh$_2$(OTf), IPhCl$_2$) to explore potential higher oxidation state organopalladium chemistry.

2. Results and discussion

The reagent *trans*-[Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**) was used as a palladium(II) substrate in order to provide convenient NMR probes for the phosphine and alkynyl groups in both the aromatic and aliphatic regions, and presence of an alkynyl group different from IPh(C≡CR)(OTf) (R = Bu^t, SiMe$_3$) in order to probe potential selectivity in decomposition processes from intermediates. Complex **1** was obtained on reaction of *trans*-[PdCl$_2$(PMe$_2$Ph)$_2$] with Li(C≡C-*o*-Tol). In order to assist with NMR characterisation of some reaction products (see below), *trans*-[PdCl(C≡C-*o*-Tol)(PMe$_2$Ph)$_2$] (**2**) was obtained on reaction of *trans*-[PdCl$_2$(PMe$_2$Ph)$_2$] with *o*-Tol–C≡CH in triethylamine/acetone.

Complexes **1** and **2** formed as crystalline solids suitable for X-ray diffraction studies (Fig. 1) and have structural parameters similar to the closely related complexes *trans*-[Pd(C≡CPh)$_2$(PEt$_3$)$_2$] [13] and *trans*-[PdCl(C≡CPh)(PPh$_3$)$_2$] [14].

Complex **1** contains one half of a centrosymmetric molecule in the asymmetric unit, while **2** contains one molecule in the asymmetric unit, and complex **2** has disorder involving rotation about the Pd(1)–P(1) bond, modelled by two complementary refined occupancies for a methyl group and the phenyl substituent. For the phenyl ring, two orientations were noted that shared adjacent ortho and meta carbon atoms.

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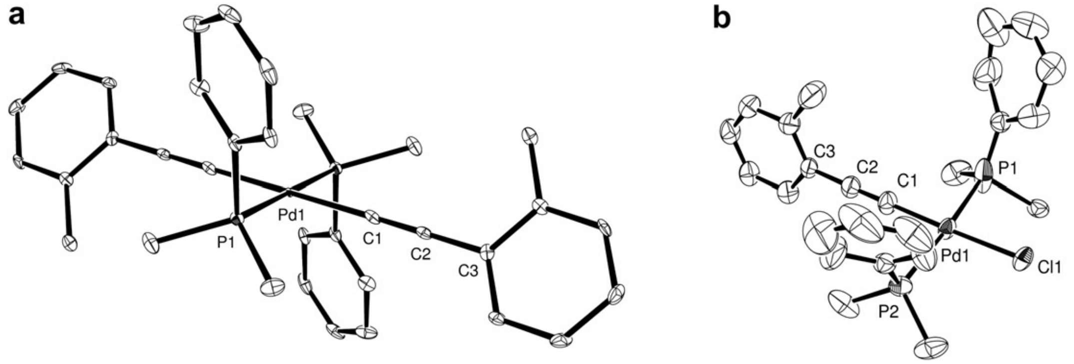


Fig. 1. ORTEP representations of the structures of (a) *trans*-[Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**), and (b) *trans*-[PdCl(C≡C-*o*-Tol)(PMe$_2$Ph)$_2$] (**2**). Displacement ellipsoids are drawn at the 50% probability level, and hydrogen atoms are omitted for clarity. Selected bond lengths (Å) and angles (deg) for **1**: Pd(1)–C(1) 2.006(3); Pd(1)–P(1) 2.2976(8); C(1)–C(2) 1.215(5) Å; C(1)–Pd(1)–P(1) 88.65(9), 91.35(9)°; **2**: Pd(1)–C(1) 1.955(5); Pd(1)–P(1,2) 2.2959(15), 2.3080(13); Pd(1)–Cl(1) 2.3536(12); C(1)–C(2) 1.197(7) Å; C(1)–Pd(1)–Cl(1) 178.11(15); C(1)–Pd(1)–P(1,2) 86.04(14), 87.72(14); Cl(1)–Pd(1)–P(1,2) 93.42(5), 92.96(5); P(1)–Pd(1)–P(2) 172.20(6); Pd(1)–C(1)–C(2) 179.0(5); C(1)–C(2)–C(3) 178.6(5) °.

The reactivity of *trans*-[Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**) toward iodonium reagents was monitored by ^{1}H and ^{31}P NMR spectroscopy, and organic products characterised by GCMS (Scheme 1). Reactions occurred with elimination of iodobenzene from iodonium reagents, and was least facile for IPh$_2$(OTf) (reaction above 15 °C) and most facile for IPhCl$_2$ (reaction above −20 °C). Colourless solutions of reagents are dark brown on completion of reaction and, for IPhCl$_2$ as oxidant, an initial bright red colouration occurs at −20 °C (visible spectra exhibit a broad shoulder at ca. 460 nm) and disappears during reaction as the dark brown colouration develops. ^{1}H NMR spectra during reactions were complex, showing low intensity resonances that arose and disappeared for reaction with IPh$_2$(OTf) (7.64 m and 2.05 s ppm), IPh(C≡CSiMe$_3$)(OTf) (8.13 s and 8.14 s ppm), and IPhCl$_2$ [8.25 and 8.26 ppm (singlets or doublet)]. ^{31}P NMR spectra showed new resonances that arose and disappeared as reaction concluded for reaction with IPh$_2$(OTf) (two resonances at −5.52 and −5.71 ppm, downfield from **1** (−6.58 ppm)), reaction with IPh(C≡CSiMe$_3$)(OTf) (one resonance at −8.10 ppm), and reaction with IPhCl$_2$ showed only the formation of *trans*-[PdCl(C≡C-*o*-Tol)(PMe$_2$Ph)$_2$] (**2**) at −7.17 ppm. On completion, reactions (i) and (ii) exhibit several ^{31}P resonances, consistent with the more complex decomposition pathways indicated by GCMS analyses (see below).

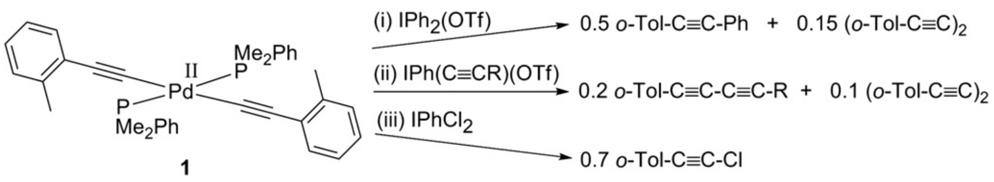
The major organic products, as indicated in Scheme 1, involve coupling of an *o*-Tol–C≡C group with a group anticipated to be transferred to Pd^{II} from the iodonium reagent, together with (*o*-Tol–C≡C)$_2$ for reactions of IPh$_2$(OTf) and IPh(C≡CR)(OTf) (R = Bu^t, SiMe$_3$). Based on the yield of *ortho*-tolylethynyl groups these organic products, and anticipated complementary Pd^{II} products, the overall yields are ca. 65% for reaction (i), ca. 35% for reaction (ii), and ca. 70% for reaction (iii). Reaction (iii) [**1** + IPhCl$_2$] gave traces of Ph–Ph and C$_6$H$_4$ICl; reaction (iii) [**1** + IPh(C≡CSiMe$_3$)(OTf)] gave a small quantity of (Me$_3$Si–C≡C)$_2$ (5%). Formation of Ph–Ph can be most likely attributed to oxidative addition of iodobenzene to Pd^0 decomposition products followed by exchange reactions between Pd^{II} centres and reductive elimination of biphenyl from 'Pd^{II}Ph$_2$' species. Similarly, for reaction (ii) [**1** + IPh(C≡CSiMe$_3$)(OTf)], (Me$_3$Si–C≡C)$_2$ may be formed from '(Me$_3$Si-C≡C)$_2$Pd^{II}' species formed after alkynyl group exchange [14,15] between '(Me$_3$Si–C≡C)Pd^{II}' decomposition product(s).

There is insufficient evidence to conclude the potential involvement of Pd^{III} or Pd^{IV} species such as 'Pd^{IV}(OTf)(C≡C-*o*-Tol)$_2$X(PMe$_2$Ph)$_2$' (X = Ph, C≡CR, Cl), followed by reductive elimination processes. Although spectroscopic data are insufficient to conclude that such intermediate(s) are present, they do indicate the formation of intermediates for reactions of IPh$_2$(OTf), IPh(C≡CSiMe$_3$)(OTf) and IPhCl$_2$, although ^{31}P NMR spectra exhibit resonances close to that expected of Pd^{II} species. The distinctive red colouration observed during reaction of IPhCl$_2$ may be indicative of Pd^{III}–Pd^{III} species, as reported complexes of this type are all red [10,11]. Well documented mononuclear Pd^{III} species are green and paramagnetic [9], and we observed no indication of paramagnetic shifts in NMR spectra.

The reactions reported here do appear to provide the first examples of alkynyl–alkynyl and alkynyl-halogen coupling from palladium centres in moderate to high yield in the presence of strong oxidising agents that are known to transfer 'R–C≡C$^+$', 'Ph$^+$' and 'Cl$^+$' electrophiles to Pd^{II} centres in other systems. The detection of C(*sp*)-halogen coupling reported here is complementary to earlier reports of C(*sp*^{2})-halogen coupling from isolated or detected Pd^{III} [11] and Pd^{IV} [27] complexes, and C(*sp*^{3})-halogen coupling from isolated or detected Pd^{III} [9] and Pd^{IV} complexes [28].

3. Experimental

Air-sensitive materials were handled under an argon atmosphere using standard Schlenk techniques. All solvents were dried and distilled by conventional methods prior to use. The reagent *trans*-[PdCl$_2$(PMe$_2$Ph)$_2$] was obtained using PdCl$_2$(SMe$_2$)$_2$ instead of PdCl$_2$ [16]; IPh(C≡CR)(OTf) (R = Bu^t, SiMe$_3$) [17], IPh$_2$(OTf) [18],



Scheme 1. Reactions of *trans*-[Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**) with iodonium reagents at 10 °C in acetone-*d*$_6$ (R = Bu^t, SiMe$_3$; OTf = triflate) illustrating major organic products; *trans*-[PdCl(C≡C-*o*-Tol)(PMe$_2$Ph)$_2$] is isolated from reaction (iii).

and $IPhCl_2$ [19] were prepared as reported. NMR spectra were recorded on a Varian Mercury Plus 300 MHz and Varian 400 Unity Inova spectrometers. ^1H NMR spectra were secondary-referenced to residual solvent signals. ^{31}P NMR spectra were externally referenced to 85% H_3PO_4.GCMS analyses were carried out on a Varian 3800 GC coupled to Varian 1200 triple quadrupole mass spectrometer in single quadrupole mode. A Varian 'Factor Four' VF-5 ms (30 m × 0.25 mm internal diameter and 0.25 micron film) column was used. Injections of 1 μL were made using a Varian CP-8400 autosampler and a Varian 1177 split/splitless injector at 220 °C with a split ratio of 30:1. The ion source was at 220 °C, and the transfer line at 290 °C. The carrier gas was helium at 1.2 mL min^{-1} using constant flow mode. The column oven was held at 50 °C for 2 min then ramped to 290 °C at 8 °C min^{-1}. The range from m/z 35 to 550 was scanned 3 times per sec. GCFID was carried out on a Varian 450-GC with 1177 split/splitless injector using similar conditions to the above, except that the carrier gas was nitrogen. The FID response factor for *n*-heptadecane was determined from a solution of known concentration and response factors for individual analyses were taken from the literature where known, e.g. iodobenzene [20]. For others the 'effective carbon number' concept and known negative effects on FID response [21–23] was employed to estimate corrections required for heteroatoms.

3.1. Synthesis of complexes

3.1.1. Trans-di(ortho-tolylethynyl)bis(dimethylphenylphosphine)palladium(II), trans-[Pd(C≡C-o-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**)

A suspension of $PdCl_2(PMe_2Ph)_2$ (0.512 g, 1.13 mmol) in dry diethyl ether (15 mL) was cooled to −70 °C for 15 min, and a freshly prepared cold solution of Li–C≡C-o-Tol (2.26 mmol) in diethyl ether (5 mL) in a Schlenk vessel was added dropwise over a period of 15 min with constant stirring. The reaction mixture was then allowed to rise slowly to 0 °C over ∼2 h, during which the colour of the suspension changed from light brown to milky white. The suspension was stirred at 10 °C for 5 min, cold water (1 mL) injected and the diethyl ether layer was collected. The water layer was further extracted with dichloromethane and, collected together with the diethyl ether layer, was dried over $MgSO_4$, filtered through Celite and dried under vacuum to obtain a white product which was further purified by recrystallisation from dichloromethane (0.54 g, 78%). Analysis (calc., C, 66.62; H, 5.92%): C, 66.41; H, 6.77. LSIMS m/z 612 $[M\text{-}Xl]^+$, $[^{12}C_{34}H_{36}{}^{31}P_2{}^{106}Pd]$. ^1H NMR (400 MHz, CD_2Cl_2) δ 7.92–7.90 (m, 4H P*Ph*), 7.45–7.43 (m, 6H P*Ph*), 7.16–6.98 (m, 8H, H $(3\text{–}6)_{Tolyl}$), 2.26 (s, 6H, $CH_{3(Tolyl)}$), 1.98 (s, 12H, PCH_3). ^{13}C NMR (75 MHz, CD_2Cl_2) δ 138.9, 131.9, 131.4, 130.2, 129.2, 128.6, 125.4, 125.3 (aryl); 115.8, 110.0 (*C*≡*C*); 21.2 $[CH_{3(Tolyl)}]$, 1.4 (PCH_3). ^{31}P NMR (162 MHz, CD_2Cl_2) δ −6.58.

3.1.2. Trans-chloro(ortho-tolylethynyl)bis(dimethylphenylphosphine)palladium(II), trans-[PdCl(C≡C-o-Tol)(PMe$_2$Ph)$_2$] (**2**)

In a Schlenk vessel under argon, *ortho*-tolylethyne (0.23 mL, 1.83 mmol) was added to a mixture of triethylamine (3 mL) and acetone (5 mL); after stirring for ∼5 min; to $PdCl_2(PMe_2Ph)_2$ (0.45 g, 1.014 mmol) was added. The reaction mixture was stirred at room temperature for 38 h, during which time the colour of the reaction mixture changed from brown to yellowish white. The solvent was evaporated under vacuum to obtain a sticky light yellow mass, which was dissolved in dichloromethane and filtered through Celite, and dichloromethane evaporated to dryness under vacuum. The product was washed with hexane followed by diethyl ether, dried overnight under vacuum, and collected as a white powder (0.37 g, 70%). Analysis (calc. C, 56.30; H, 5.48%): C, 57.32; H, 6.47. LSIMS m/z 532 $[M\text{-}Xl]^+$, $[^{12}C_{25}H_{29}{}^{35}Cl^{31}P_2{}^{106}Pd]$. ^1H NMR (300 MHz, CD_2Cl_2) δ 7.85–7.82 (m, 4H, P*Ph*), 7.46–7.44 (m, 6H. P*Ph*), 7.10–6.99 (m, 4H, tolyl), 2.20 (s, 3H, $PhCH_3$), 1.87 (bs, 12H, PCH_3). ^{13}C NMR (75 MHz, CD_2Cl_2) δ 131.8, 131.7, 131.4, 130.4, 129.2, 128.7, 125.8, 125.3 (aryl); 20.9 $[CH_{3(Tolyl)}]$, 8.7 (PCH_3). ^{31}P NMR (162 MHz, CD_2Cl_2) δ −7.17.

3.2. Studies of reactions of trans-[Pd(C≡C-o-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**) with iodonium reagents

A solution of iodonium reagent ($IPh(C{\equiv}CSiMe_3)(OTf)$, $IPh(C{\equiv}CBu^t)(OTf)$, $IPh_2(OTf)$, or $IPhCl_2$) (8.16×10^{-2} mmol) in dichloromethane-d_2 (0.5 mL) was added to a solution of *trans*-[Pd(C≡C-*o*-Tol)$_2$(PMe$_2$Ph)$_2$] (8.16×10^{-2} mmol) in dichloromethane-d_2 (0.5 mL) at −40 °C (slush bath). An aliquot was immediately transferred to a pre-cooled NMR tube and quickly inserted into the NMR probe which was pre-cooled - 40 °C. The temperature was raised slowly until reaction was evident, and this temperature maintained until completion of reaction.

3.3. Structural determinations

Data for **1** and **2** were collected at −173 °C for crystals mounted on a Hampton Scientific cryoloop at the MX1 beamline of the Australian Synchrotron ($\lambda = 0.77487$, 0.77506 for **1** and **2**, respectively) using Blue Ice software [24] and data reduced using XDS. The structures were solved by direct methods with SHELXS-97, refined using full-matrix least-squares routines against F^2 with SHELXL-97 [25], and visualised using X-SEED [26]. All non-hydrogen atoms were refined anisotropically using a riding model with fixed C–H distances of 0.95 Å (sp^2 C–H), 0.99 Å (CH_2), 0.98 Å (CH_3). The thermal parameters of all hydrogen atoms were estimated as $U_{iso}(H) = 1.2U_{eq}(C)$ except for CH_3 where $U_{iso}(H) = 1.5U_{eq}(C)$. Phosphine ligand disorder in **2** is described in Section 2.

3.3.1. Crystal data

trans-[Pd(C≡C-o-Tol)$_2$(PMe$_2$Ph)$_2$] (**1**): $C_{34}H_{36}P_2Pd$, $M = 612.97$, space group P-1, triclinic, $a = 7.5170(8)$, $b = 9.6070(4)$, $c = 10.8760(5)$ Å, $\alpha = 94.052(4)$, $\beta = 99.588(9)$, $\gamma = 108.341(4)$ °, V = 728.72(9) Å^3, $Z = 1$, $D_c = 1.397$ g cm^{-3}, specimen pale yellow prism, 0.05 mm × 0.02 mm × 0.02 mm, 7092 measured reflections, $R_{int} = 0.0309$, $R = 0.0491$ for 1879 observed data ((I) = 2σ(I)), wR = 0.0824, and GOOF = 1.098 for all 1896 unique data.

trans-[PdCl(C≡C-o-Tol)(PMe$_2$Ph)$_2$] (2): $C_{25}H_{29}ClP_2Pd$, $M = 533.27$, space group P-1, triclinic, $a = 9.270(2)$, $b = 9.2950(19)$, $c = 14.540(3)$ Å, $\alpha = 80.454(4)$, $\beta = 83.402(15)$, $\gamma = 86.165(2)$ °, V = 1225.8(5) Å^3, $Z = 2$, $D_c = 1.445$ g cm^{-3}, specimen yellow prism, 0.10 mm × 0.03 mm × 0.02 mm, 12681 measured reflections, $R_{int} = 0.0650$, $R = 0.0479$ for 3311 observed data [(I) = 2σ(I)], wR = 0.1176, and GOOF = 1.036 for all 3409 unique data.

Acknowledgements

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Appendix A. Supplementary material

CCDC numbers 800027 and 800028 contain the supplementary crystallographic data for this paper. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif.

Appendix A. Supplementary material

Visible spectra during reaction of **1** with $IPhCl_2$. Supplementary data related to this article can be found online at doi:10.1016/j.jorganchem.2011.01.018.

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