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Isolated-palladium complexes for catalyzed telomerization of butadiene with methanol in the presence of water

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ABSTRACT

In order to study the performance of organometallic complexes in the telomerization of butadiene with methanol in aqueous medium, we synthesized and characterised hydrosoluble palladium complexes. $[(\pi\text{-allyl})Pd(TPPTS)_2]^+Cl^-$ complex exhibited strong stability as no degradation was observed after storage at room temperature under air atmosphere for weeks. TON's up to 36 000 were achieved at 50 °C.

1. Introduction

Because of increasing environmental constraints, the effective implementation of the principles of the green chemistry is an essential issue. It implies the involvement of catalytic reactions allowing a maximal use of atoms. The telomerization process which allows the synthesis of functionalized octadienyl substrate with a 100% atom efficiency, fulfils most of these conditions (Fig. 1) [1–3]. It merely involves palladium catalysts, and readily available 1,3-butadiene and alcohols or water reagents.

Indeed, the telomerization reaction has been considered to be a useful industrial reaction and was applied to the preparation of 2,7-octadien-1-ol [4]. Recently, very efficient complexes bearing either phosphine or carbene ligands were reported and exhibited very high activity for the telomerization of methanol with butadiene [5]. Beller et al. reported the use of mono(phosphane)-diallylether-palladium complex ($L = PPh_3$) which is highly active in the telomerization of butadiene with MeOH [6]. More recently, the same group described the preparation of industrially viable catalyst system that catalyzed the telomerization of 1,3-butadiene with primary alcohols and phenol derivatives [7,8]. The catalysts are carbene-divinyldisiloxane-palladium complexes bearing a single carbene ligand and two labile olefin moieties.

Moreover, this transformation was applied to polyols such as ethylene glycol [9,10], glycerol [11–13], mono or polysaccharides in aqueous or organic conditions [14–17]. The group of Weckhuysen described the use of new highly active catalysts for the telomerization of crude glycerol with butadiene. The highest activity was achieved with the tris(*ortho*-methoxyphenyl)phosphine-based complex [18].

For these polyol substrates, the development of water-soluble complexes was shown as an alternative. Kuntz reported the first applications of butadiene telomerization with several nucleophiles such as methanol, phenol, acetic acid or diethylamine in aqueous media [19,20]. One of the most efficient hydrosoluble complexes is based on the TPPTS ligand (tris(*m*-sulfonato-phenyl) phosphine trisodium salt, Fig. 1). This ligand allows the formation of highly hydrosoluble complexes that were used industrially in the field of hydroformylation [21]. Water-soluble catalyst prepared *in situ* from $Pd(OAc)_2$ and TPPTS generated a zero-valent palladium species that was shown to be an efficient catalyst for C–C coupling [22]. Behr developed the TPPTS/Pd catalyzed telomerization of butadiene with glycols either in monophasic or biphasic conditions [11].

The efficient etherification of mono and disaccharide nucleophiles *via* telomerization reaction catalyzed by Pd/TPPTS complexes has been described [17,23,24]. We applied also this catalytic system for the preparation of hydrophobic starch [25,26]. Basset et al. reported the telomerization of butadiene with methanol in ionic liquids as solvents using either PPh_3 or sulfonated phosphine (TPPMS, TPPDS or TPPTS, Fig. 1) as ligands [27]. All these previous works suffered some significant limitations such as the use of at least 3 (up to

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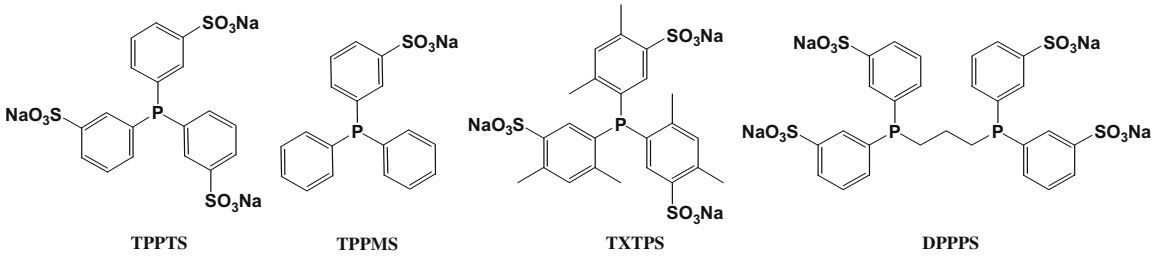


Fig. 1. Structure of the hydrosoluble ligands.

5) equivalents of TPPTS/Pd. Also in many cases, the synthesized complexes were sensitive to degradation. In order to optimize the reaction, we studied the preparation and the use of TPPTS/Pd-based catalysts in telomerization of butadiene with alcohols such as methanol or starch in the presence of water [25]. $Pd(OAc)_2$ and $Pd(acac)_2$ are standard palladium precursors for the preparation of active complexes for the telomerization of butadiene with alcohols. It was shown that in the presence of three equivalents of sulfonated phosphine ligand, active species have been obtained. In this paper, we aimed to reduce the ligand/metal ratio because TPPTS is an expensive ligand. Also, the complex has to be stable in order to be handled without controlled atmosphere. The performance of phosphine-based complexes for the telomerization of methanol with butadiene in the presence of water has been studied.

2. Experimental section

All complexes were prepared under nitrogen using standard Schlenck techniques. The solvents were degassed prior to use. Proton and phosphorous NMR were recorded on a Bruker A 250 MHz.

2.1. Synthesis of $[(\pi\text{-allyl})Pd(TPPTS)_2]^+Cl^-$ complex

About 5 ml of deionized water was added in a 10 mL Schlenk tube with a magnetic stirrer, and was deoxygenated by vacuum-nitrogen cycles under fast stirring. 0.464 mmol (270 mg) TPPTS (containing 4–5% OTPPTS) were added and the resulting yellow solution was deoxygenated by vacuum-nitrogen cycle with fast stirring. After complete dissolution of TPPTS, 0.116 mmol (42.7 mg) of $[\pi\text{-allylPdCl}]_2$ were added and the resulting solution was deoxygenated by vacuum-nitrogen cycle with fast stirring. After 30 min of stirring, the palladium dimer was fully solubilized and the solution turned dark orange. The catalyst was used directly in telomerization reaction. For characterisation purpose, the water was evaporated at 40 °C at 20 mbar and further drying under vacuum overnight afforded 302 mg (0.225 mmol, >95%) of a slightly brown solid. NMR data are in accordance with literature [28]. ^{31}P NMR (D_2O) ppm: 24.6 $[(\pi\text{-allyl})Pd(TPPTS)_2]^+Cl^-$; 34.5 residual OTPPPTS; ^1H NMR (D_2O) ppm: 3.61 (m, 2H), 4.29–4.32 (d, J = 7.5 Hz, 2H), 6.04 (m, 1H), 7.42 (dd, J = 7.5 Hz, J = 18 Hz, 12H), 7.72 (d, J = 2.5 Hz, 6H), 7.83–7.86 (d, J = 7.5 Hz, 6H).

2.2. Synthesis of $[(\pi\text{-allyl})Pd(TPPMS)_2]^+Cl^-$ complex

In a 50 ml flask, 459 mg of TPPMS (1.26 mmol, 2.1 eq) were dissolved in a 10 ml/5 ml THF/water mixture. The resulting solution was deoxygenated by flushing through nitrogen for 15 min. About 110 mg of $[(\pi\text{-allyl})PdCl]_2$ (0.3 mmol, 0.5 eq) were then added and the resulting solution was stirred at room temperature under nitrogen for 4 h. The solution turned from yellow to orange. THF and water were evaporated under vacuum to yield 462 mg (0.504 mmol, 84%) of a yellow solid. ^{31}P (D_2O) ppm: 23.55 ($[(\pi\text{-allyl})Pd(PAr_3)_2]Cl$), NMR ^1H (D_2O) ppm: 3.39 (broad m, $2H_{anti}$), 3.78 (large m, 2H), 5.80 (broad m, 1H), 6.94 et 7.05 (M, 24H), 7.73 (large M, 4 H).

2.3. Methanol telomerization

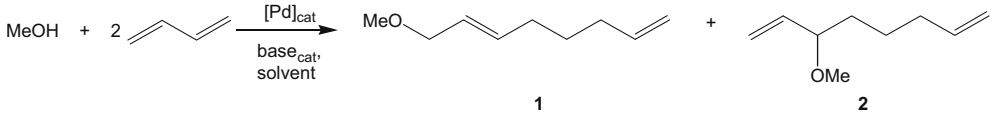
All the catalytic reactions were performed in a stainless steel autoclave equipped with a mechanical stirrer and a heating jacket. In a typical reaction, degassed methanol (22.5 mL, 0.55 mol), H_2O (22.5 mL) and NaOH (222 mg, 5.5 mmol) were introduced in the autoclave. The previously synthesized catalyst $[(\pi\text{-allyl})Pd(TPPTS)_2]^+Cl^-$ (0.005 mol%) was then added under nitrogen. The autoclave was cooled to −30 °C, and the desired volume of butadiene was introduced from a graduated burette (typically 23 mL, 0.28 mol). The reactor was then slowly heated to the desired temperature (50 °C), the pressure increased up to 6 bar. After the desired reaction time, the autoclave was cooled to room temperature, the excess butadiene was eliminated and the autoclave was purged with argon. After separation, the aqueous layer was extracted with heptane (10 mL). The combined organic phases were dried over $MgSO_4$ and after evaporation of the solvent, 9.7 g (69 mmol; 51% yield) of a slightly yellow liquid were obtained. The selectivity of the reaction was determined by GC analysis according to literature procedure [7].

3. Results and discussion

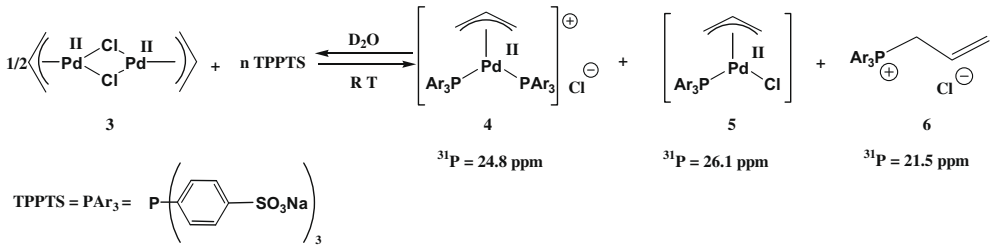
It is well established that during the preparation of hydrosoluble palladium(0) complex from palladium(II) salt such as $Pd(OAc)_2$, one phosphine ligand acts as the reducing agent and is oxidized to phosphine oxide [22]. In order to limit the amount of ligand essential to get a highly active and stable catalyst, we studied the preparation of complexes starting from palladium(II) precursor. Our choice was based on the $[(\pi\text{-allyl})PdCl]_2$ precursor (Scheme 2). Recently, the direct use of allyl alcohol in Tsuji–Trost reaction was reported in the presence of $[(\pi\text{-allyl})(TPPTS)_nPd]$ species which were not precisely characterised [29]. Kuntz et col. previously reported the use of $[(\pi\text{-allyl})Pd(TPPTS)_2]Cl$ complex in the allylation of guaiacol [30]. This complex was formed *in situ* from $Pd(TPPTS)_3$ and allylic alcohol in alkaline media. However, in highly basic media, some degradation of the system appears with formation of metallic palladium particles. The same group showed that palladium zero complex was formed quantitatively and instantaneously by the addition of NaOH [31]. Some allylic palladium complexes were also shown to be efficient in the telomerization of butadiene or isoprene with alcohols in organic medium.

The nature of the organometallic species formed with variable amount of hydrosoluble ligands was evaluated (Scheme 2).

The NMR study of the complexes prepared from $[(\pi\text{-allyl})PdCl]_2$ precursor and TPPTS ligand was performed using from 1 to 4 equivalents of ligand per metal in D_2O. Whatever the ratio, a small signal corresponding to OTPPTS (δ_P = 34.5 ppm) was observed, because of the presence of oxide in the initial sample of TPPTS (Table 1 and Fig. 3).



Scheme 1. Telomerization of methanol with butadiene to form ether.



Scheme 2. Preparation of active TPPTS-Pd complexes for telomerization reaction.

Table 1
^{31}P and ^{1}H NMR analysis of Pd/TPPTS complexes in D_2O.

Entry	P/Pd molar ratio	δ ^{31}P NMR (relative integration)[a]	Relative integration ^{1}H NMR (%)		
			3	**4**	**5**
1	1	24.8 (39%); 26.0 (55%); 34.5 (6%)	16	25	59
2	1.7	24.8 (68%); 26.1 (23%); 34.5 (9%)	<1	65	35
3	1.9	24.8 (67%); 26.1 (25%); 34.5 (8%)	<1	84	16
4	2.01	21.6 (4%); 24.8 (87%); 34.5 (9%)	<1	<1	100
5	4	20.8 (50%, broad); 21.5 (22%; 24.8 (11%); 34.5 (16%)	Broad signals		

[a] δ ^{31}P = 34.5 ppm corresponds to O=PAr_3 present in the starting material (6%).

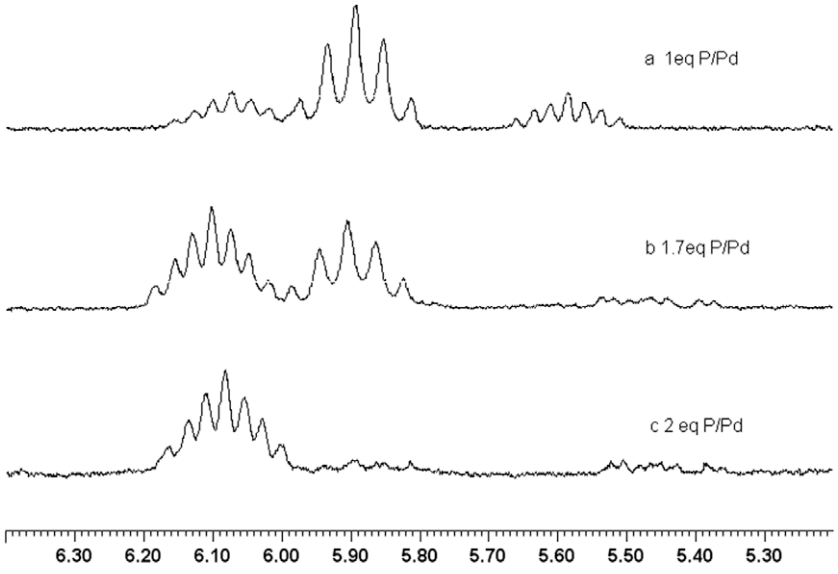


Fig. 2. Evolution of ^{1}H NMR with TPPTS/palladium ratio.

In the presence of one equivalent of ligand per palladium, the reaction was not complete because an equilibrium was established between two different complexes bearing phosphine ligand (δ_{P1} = 24.8 ppm; δ_{P2} = 26.1 ppm, Fig. 3) together with a residual amount of the initial dimeric palladium complex **3** (Scheme 2). According to literature data δ_{P1} = 24.8 ppm is attributed to the cationic complex [(π-allyl)Pd(TPPTS)$_2$]Cl **4** bearing two ligands [28]. The second complex formed during this reaction involves phosphine ligand (δ_{P2} = 26.1 ppm) and an allylic moiety (Fig. 2). It can be attributed to the neutral complex **5** bearing one phosphine ligand [32]. ^{1}H NMR allowed quantifying these three species to 16%, 25% and 59% for complexes **3**, **4** and **5**, respectively (Fig. 2). It has to be mentioned that under similar conditions, the mixing of dimer [(π-allyl)PdCl]$_2$ **3** with one equivalent of PPh_3 yields to the formation of a single complex [(σ-allyl)Pd(PPh_3)Cl] (δ_P = 22.8 ppm, $CDCl_3$). The different behaviour observed with TPPTS ligand can be attributed either to the different reactivity of the ligand or to the influence of the solvent.

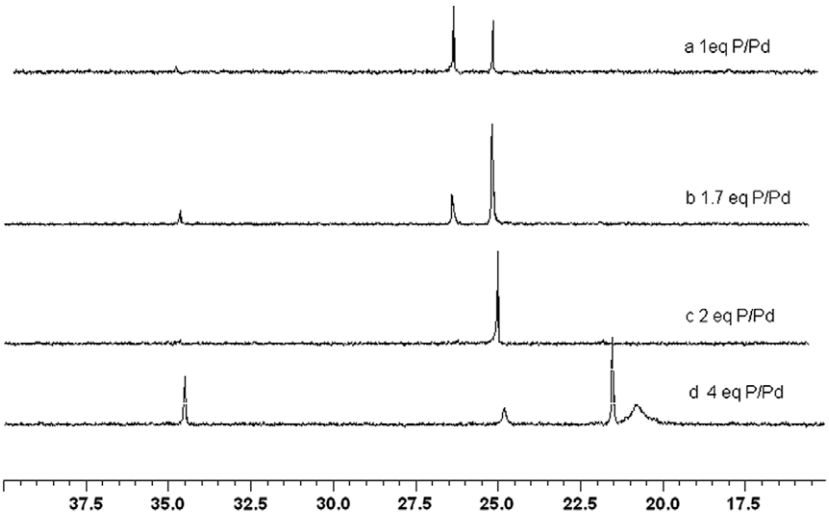


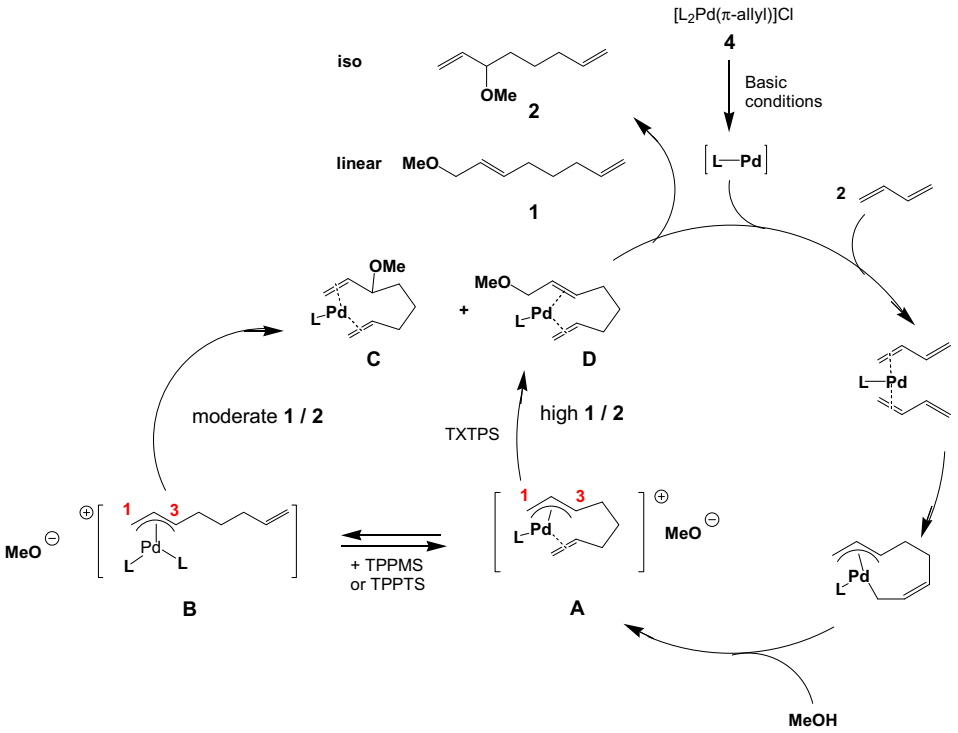
Fig. 3. Evolution of ^{31}P NMR with TPPTS/palladium ratio.

Increasing the P/Pd ratio up to 2, drives to the disappearance of the starting material as observed in ^{1}H NMR (entry 4, Table 1). Simultaneously, the ratio **4**/**5** increases as well, and in the presence of 2 equivalents of ligand per metal, the cationic complex **4** is the exclusive product (Figs. 2 and 3).

The use of a large excess of ligand (P/Pd = 4) has a dramatic influence on the complexes formed in solution. Under these specific conditions, the presence of phosphonium **6** (δ_{P3} = 21.5 ppm, Fig. 3) is clearly identified and is attributed to the reaction of ligand in excess with the cationic complex **4** as previously reported. A broad signal at δ_P = 20.8 ppm was also analyzed that could be assigned to [Pd(TPPTS)$_n$] [31].

The cationic complex **4** prepared on a 3 g scale, exhibited a strong stability as no degradation was detected (visual observation and NMR analysis) after storage at RT under air atmosphere for weeks. It is supposed that under telomerization conditions (basic conditions), active species are formed (Scheme 3) [31].

This complex was further used for the telomerization of butadiene with methanol. Other allylic palladium complexes were prepared *in situ* starting from different hydrosoluble phosphine ligands such as commercially available TPPMS and TXTPS, as well as DPPPS (sulfonated form of the DPPP) (Fig. 1) [33]. A ratio P/Pd = 2 was chosen for each complex. All these complexes, isolated or prepared *in situ*, were evaluated in the telomerization of palladium with methanol as nucleophile at 50 °C in the presence of water (MeOH/H_2O = 1/1) for 24 h (Scheme 1). For comparison, we examined the reaction in the presence of *in situ* prepared $Pd(OAc)_2$/3 TPPTS system for comparison.



Scheme 3. Mechanism of the telomerization reaction.

Table 2
Influence of the nature of catalyst on conversion and selectivity.[a]

Entry	Catalytic system	Yield (**1+2**) (%)	**1** / **2** ratio	TON	TOF (h^{-1})
1	[π-allylPdCl]$_2$	0			
2	Pd(OAc)$_2$/TPPTS (1/3)	43	93/7	8600	358
3	[π-allylPdCl]$_2$/TPPTS (1/2)	58	89/11	12 200	508
4	[(π-allyl)Pd(TPPTS)$_2$]Cl	50	95/5	10 000	417
5	[(π-allyl)Pd(TPPTS)$_2$]Cl[b]	38	96/4	**36 000**	1500
6	[π-allylPdCl]$_2$/TPPMS (1/2)	54	95/5	10 800	450
7	[π-allylPdCl]$_2$/TXTPS (1/2)	45	**98/2**	9000	375
8	[π-allylPdCl]$_2$/DPPPS (1/1)	20	96/4	4000	167

[a] Reaction conditions [Pd]$_{but}$% = 0.005%, Butadiene/MeOH/H_2O = 1/1/1 (vol), [NaOH] = 2 mol%, 50 °C, 24 h.
[b] [Pd]$_{but}$% = 0.001%.

The reaction was performed in the presence of an excess of nucleophile relative to the diene (n_{but}/n_{MeOH} = 1/2), so the yield is based on butadiene consumption (Table 2). Whatever the reaction conditions, negligible amounts of octatriene and vinylcyclohexene were detected (<1%).

No conversion was observed in the presence of the dimeric [π-allylPdCl]$_2$ complex without ligand (entry 1). After 24 h reaction, 43% of butadiene were converted in the presence of the *in situ* Pd(OAc)$_2$/TPPTS (1/3) catalyst (entry 2). Under identical conditions, the catalyst prepared *in situ* from the [π-allylPdCl]$_2$ precursor (Pd/L = 1/2) yielded 58% of octadienyl ethers corresponding to a TON = 12 200 (entry 3). Similar result was achieved in the presence of isolated cationic complex [(π-allyl)Pd(TPPTS)$_2$]Cl **4** (entry 4). Beller et al. reported the use of Pd(OAc)$_2$/3H-TPPTS in the presence of triethylamine as base; in that case they obtained 5% conversion at 90 °C for 2.5 h [34]. In this study, the complex prepared from only two equivalents of hydrosoluble phosphine exhibited slightly higher reactivity in comparison with the previous Pd(OAc)$_2$/TPPTS (1/3) complex. This is a remarkable result considering the cost of the TPPTS ligand. Even with a substrate:catalyst ratio of 100 000:1 a significant conversion was achieved (entry 5, TON = 36 000).

The nature of the sulfonated ligand had a significant impact on the reactivity or selectivity of the complex. The diphosphine ligand DPPPS exhibited lower activity (entry 8). This result correlates with Sbrana's report who found that the activity of palladium complexes chelated to DPPP was lower than that of monophosphine palladium complexes [35]. This was attributed to the formation of a six-membered ring intermediate which is more stable and less active. Similarly Moreno–Manas reported that highly stable macrocylic palladium complex is inactive in the telomerization of butadiene with MeOH [36]. Such ligands are not well adapted for this reaction. Next, we tested alternative sulfonated monophosphine such as TPPMS and TXTPS ligands (Fig. 1). As shown in Table 2, the nature of sulfonated monophosphine has a minor influence on the yield of telomerization products (entries 3, 6 and 7). Using standard reaction conditions, 9000 up to 12 000 TON's were achieved. As far as the *n*/iso ratio is concerned, all these complexes yielded an excellent regioselectivity towards the linear ether (higher than 89/11). The significant n/iso selectivity achieved with the TXTPS ligand must be underlined (**1/2** = 98/2, entry 7). The regioselectivity of the telomerization reaction can be correlated mainly to steric and electronic effects. Considering π-allyl palladium systems, the formation of linear ether is favoured by steric constraints while electronic effects increase the formation of branched products. It is supposed that the main reason for the predominant formation of the linear product is the formation of a trigonal planar (1,6-diene) palladium **D** complex which is more stable compared to the (1,7-diene) palladium complex **C** (Scheme 3). Considering the TXTPS ligand, due to the presence of methyl group, its π-acceptor behaviour decreases while the steric hindrance increases [37]. This favours the attack at the C_1 center which is less electron-rich in that case. Moreover, due to steric hindrance, the formation of the (bis-diphosphine) Pd (η^3-octadienyl) species **B**, which increases the formation of branched product, is disfavoured. Both contributions enhanced significantly the linear/branched ratio in a similar level than observed with carbene ligands [7]. Such impact of *o*-substituted phosphine on the regioselectivity of the butadiene telomerization with alcohols was previously reported in different reaction conditions. On the other hand, the formation of intermediate **B** bearing two ligands is more suitable with TPPTS or TPPMS. In that case, the attack at C_3 center is electronically favourable and a lower **1/2** ratio is attained.

These complexes exhibited high activity even in the presence of water, so they are suitable for starch telomerization and the study of this transformation is under progress [38].

In conclusion, hydrosoluble palladium complex bearing two phosphine ligands can be prepared on a large scale and isolated in the cationic form. This stable complex is suitable for the telomerization of butadiene with methanol in the presence of water. High TON's up to 36 000 are obtained. Several hydrosoluble monophosphine ligands were evaluated and the highest linear/iso regioselectivity is achieved with the TXPTS one. Further works are under progress to synthesize new stable catalyst precursors, and to apply them to the telomerization of butadiene with different nucleophiles.

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