Pd colloid-catalyzed methoxycarbonylation of iodobenzene in ionic liquids

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Dedicated with best wishes to Professor Józef J. Ziółkowski in recognition of his inspirational spirit.

Abstract

A Pd colloid prepared by reduction of the $[Pd(\eta^3\text{-allyl})_2Cl]_2$ precursor with Vcp_2 (vanadocene) in presence of polyvinylpyrrolidone (PVP) as a protecting polymer was found to be an active and stable catalyst of methoxycarbonylation of iodobenzene carried out in an ionic liquid (IL) medium of [bmim]X and/or [bumepy]X (bmim = 1-methyl-3-butyl imidazolium cation, bumepy = 1-butyl-4-methyl pyridinium cation, X = Cl, BF_4, PF_6) or in the presence of ammonium salts of $[R_4N]X$ type (R = ^nBu, Et, Me; X = Cl, Br, I). The yield of benzoic acid methyl ester produced in methoxycarbonylation of iodobenzene in ionic liquids depends on their structure: those with the pyridinium cation are much more effective than those with the imidazolium one. At the optimal concentration of methanol, the yield of methoxycarbonylation reaction catalyzed by Pd colloid decreases in the order: $[^nBu_4N]Br > [^nBu_4N]I > [^nBu_4N]Cl > [bumepy]PF_6 > [Et_4N]Br > [bumepy]Cl > [bumepy]BF_4 > [Et_4N]Cl > [bmim]PF_6 > [bmim]BF_4 > [bmim]Cl$.
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1. Introduction

Metal colloids have recently received increasing interest from many research groups which have applied metal nanoparticles in catalytic reactions [1–10]. It has been demonstrated that colloids of such transition metals as palladium, rhodium and ruthenium exhibit attractive catalytic properties and may successfully compete as catalysts with traditional organometallic species [1–10].

Metal colloids have till now been effectively used first of all in catalytic hydrogenation of olefins and aromatics [6,11–14], but there are also examples of their application in carbon–carbon coupling in the Heck [2,4] and Suzuki [1,3,8] reactions. The essential parameter of metal colloids that characterizes their high catalytic ability is a high surface-to-volume ratio. A high value of that factor is an indicator of potentially better access of substrates to the catalytically active centers compared with heterogeneous catalysts. Catalytic systems based on metal colloids are relatively simple and do not require the use of complicated modifying ligands. However, metal colloids may undergo aggregation during the reaction leading to loss of catalytic activity. The size distribution of palladium nanoparticles after recycling in the Suzuki reaction has been studied recently [1]. Careful selection of the reaction medium, for example, the addition of ammonium salts, may prevent aggregation probably via electrostatic interactions and stabilize the size of metal colloid nanoparticles sufficient for catalysis. We have recently found that in carbonylation with $PdCl_2(cod)$ as the catalyst precursor Pd colloids are formed in situ and their catalytic activity increases when ammonium salts are added [15].

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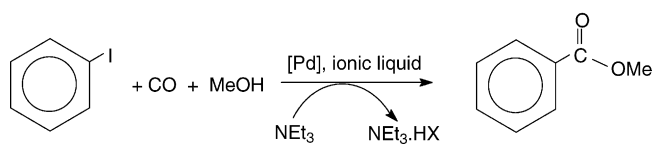
Scheme 1.

In this paper we present studies on methoxycarbonylation reaction of iodobenzene catalyzed by Pd colloid in the ionic liquid media (Scheme 1).

Ionic liquids, characterized by very low vapor pressure, have recently been frequently used as solvents for catalytic reactions replacing less environmentally friendly organic solvents [16–20]. A palladium catalyst with phosphane ligands was applied in carbonylation in imidazolium ionic liquids [16]. However, interactions of metal colloids with ionic liquids are not understood well, and according to our best knowledge this paper presents the first examples of the design of an active and stable catalytic system for carbonylation reaction composed of palladium colloid and an ionic liquid.

2. Results and discussion

The Pd colloid that was used in this work as a catalyst for methoxycarbonylation of iodobenzene was prepared by reduction of the $[Pd(\eta^3\text{-allyl})_2Cl]_2$ complex with Vcp_2 (vanadocene) in presence of polyvinylpyrrolidone (PVP) as a protecting polymer and characterized with transmission electron microscope (TEM) and wide angle X-rays scattering (WAXS) methods [13]. This colloid catalyzes hydrogenation of nitrobenzene to aniline [13]. Preliminary studies of its catalytic activity in methoxycarbonylation of iodobenzene demonstrated rather limited activity with a 26% yield of the product, benzoic acid methyl ester. Further studies were conducted in ionic liquids (IL) containing imidazolium (bmim = 1-methyl-3-butyl imidazolium) or pyridinium (bumepy = 1-butyl-4-methyl pyridinium) cations with anions such as Cl^-, BF_4^- and PF_6^-. Ionic liquids were used in amounts (in moles) comparable to the amount of iodobenzene, and in all reactions the system was biphasic with the colloid dissolved in the ionic liquids (bottom layer).

It was also demonstrated that the ionic liquid itself (e.g. [bumepy]BF$_4$) did not show any catalytic activity.

The introduction of an ionic liquid with the [bumepy] cation caused an increase in the yield of the product compared to the yield of the reaction carried out in methanol only (Table 1, entry 1).

The maximum yield of 83% was obtained after 3 h of reaction conducted in [bumepy]PF$_6$. Slightly lower yields of ester, 60–66%, were obtained for ionic liquids with the [bumepy] cation and BF$_4^-$ or Cl$^-$ anions (entries 10, 11). On the other hand, ionic liquids with the bmim cation were found to be inhibitors and only with [bmim]PF$_6$ 35% of ester was ob-

Table 1

Effect of ionic liquids (IL) on the yield of methoxycarbonylation reaction of iodobenzene

Entry	IL	$p(CO)$ (atm)	Time (h)	Yield of ester (%)
1	–	10	4	26
2[a]	[mokt]BF$_4$	5	3	0
3[b]	[mokt]BF$_4$	5	3	1
4[a]	[bmim]Cl	5	3	8
5[b]	[bmim]Cl	5	3	0
6[a]	[bmim]BF$_4$	5	3	18
7[b]	[bmim]BF$_4$	5	3	8
8[a]	[bmim]PF$_6$	5	3	35
9[b]	[bmim]PF$_6$	5	3	0
10[a]	[bumepy]Cl	5	3	66
11[a]	[bumepy]BF$_4$	5	3	60
12[b]	[bumepy]BF$_4$	5	3	34
13[b]	[bumepy]BF$_4$	5	4	36
14[b]	[bumepy]BF$_4$	10	3	36
15[b]	[bumepy]BF$_4$	10	4	36
16[a]	[bumepy]PF$_6$	5	3	83

Reaction conditions: [MeOH]:[PhI] = 2.8, [MeOH]:[IL] = 2.7, [IL] = 9.11 × 10^{-3} mol, 1.0 cm^3 (8.93 × 10^{-3} mol) PhI; 3.0 cm^3 (2.15 × 10^{-2} mol) NEt$_3$; 0.64 cm^3 (4.6 × 10^{-3} mol) mesitylene; 90 °C.

[a] 2 × 10^{-3} M (1.17 × 10^{-5} mol) [Pd].

[b] 1.2 × 10^{-3} M (1.17 × 10^{-5} mol) [Pd], $V = 10$ cm^3, 2.5 cm^3 (2.35 × 10^{-2} mol) toluene.

tained (entry 8), a little more than with methanol only. In the case of [mokt]BF$_4$ (mokt = 1-methyl-3-octyloimidazolium), methoxycarbonylation was not observed at all (entry 2). Considering the effect of anions, best results were obtained with the PF$_6^-$ anion (Table 1).

Positive effect of pyridinium salts was confirmed in methoxycarbonylation reactions carried out in the presence of toluene as a cosolvent. Under similar reaction conditions (see note under Table 1) but at a twice lower concentration of the catalyst, the reaction yield dropped to ca. 36%. Again

Table 2

Effect of methanol and [IL] concentrations on the yield of methoxycarbonylation reaction of iodobenzene in [bumepy]BF$_4$ solution

Entry	[MeOH]:[Pd]	[MeOH]:[IL]	[PhI]:[IL]	Yield of ester (%)
17	1000	1.3	1	50
18	1900	2.7	1	60
19	3100	4.1	1	60
20	2500	3.5	1.3	65
21	3500	4.9	1.3	78
22	4500	6.6	1.3	82
23	2600	4.9	1.8	63
24	4400	8.9	1.8	73
25	5700	11.2	1.8	50
26	2600	7.1	2.5	44
27	2800	9.9	3.5	43
28[a]	2100	2.7	1	31
29[b]	2100	2.7	1	21

Reaction conditions: 1.17 × 10^{-5} mol [Pd]; 1.25–1.91 M [Pd]; 1.0 cm^3 (8.93 × 10^{-3} mol) PhI; 3.0 cm^3 (2.15 × 10^{-2} mol) NEt$_3$; 0.64 cm^3 (4.6 × 10^{-3} mol) mesitylene; 90 °C; 5 atm CO; 3 h.

[a] Experiment started after 1 h stirring of Pd colloid with NEt$_3$ and [bumepy]BF$_4$.

[b] Experiment started after 1 h stirring of Pd colloid with MeOH and [bumepy]BF$_4$.

Table 3
Effect of methanol and Pd colloid concentrations of methoxycarbonylation of iodobenzene at the presence of toluene and [bumepy]BF$_4$

Entry	[Pd] $\times 10^3$ M	[MeOH]:[Pd]	[MeOH]:[bumepy]BF$_4$	Yield of ester (%)	Toluene (cm^3)
30	1.19	1000	1.3	26	3.10
31	1.19	2100	2.7	36	2.55
32	1.20	2600	3.4	43	2.25
33	0.49	5100	2.7	10	2.55
34	0.63	4000	2.7	22	2.55
35	1.21	2000	2.7	36	2.55
36	1.56	1600	2.7	41	2.55
37	2.19	1100	2.7	52	2.55
38	2.24	1100	2.7	57	2.55
39[a]	1.23	1000	1.3	38	1.10
40[a]	1.19	2100	2.7	55	0.56

Reaction conditions: 1.17×10^{-5} mol [Pd]; 1.0 cm^3 (8.94×10^{-3} mol) PhI; 3.0 cm^3 (2.15×10^{-2} mol) NEt$_3$; 0.64 cm^3 (4.6×10^{-3} mol) mesitylene; 1.80 cm^3 (9.11×10^{-3} mol) [bumepy]BF$_4$; $V = 10$ cm^3, 90 °C; 10 atm CO, 4 h.

[a] $V = 8$ cm^3, 5 atm CO, 3 h.

ionic liquids with the bmim cation showed a rather inhibiting effect on the reaction course (entries 3, 5, 7).

A very small effect of pressure on the yield of ester was noted and similar results were obtained at 5 and 10 atm. This may suggest that solubility of carbon monoxide in the reaction medium is not a yield-determining factor. Also prolongation of the reaction time over 3 h did not impact on the yield of ester. This observation could suggest deactivation of the catalyst; however, further experiments showed that the catalyst separated from the post-reaction mixture was still active. From the data collected in Table 2, one may conclude that [bumepy]BF$_4$ concentration is the yield-determining factor. In a test experiment, nearly the same amounts of Pd colloid and different amounts of [bumepy]BF$_4$ (from 9.1×10^{-3} to 2.6×10^{-3} mol) were used. As a consequence of methanol concentration variations, the [MeOH]:[IL] ratio also changed from 1.3 to 11.2. The best results, up to 82% of ester, were obtained at 5.0×10^{-3} to 6.9×10^{-3} mol of [bumepy]BF$_4$ (Table 2, entry 22). Further increase of IL amounts caused a drop in the yield of the product. The small effect of methanol concentration was noted in reactions performed at [PhI]:[IL] ratio equal to 1.3 and 1.8, where the yield of ester increased with the methanol amount increase (entries 22–25).

To clarify possible effects of colloid solubility, mixtures of Pd colloid with NEt$_3$ and [bumepy]BF$_4$ as well as Pd colloid with MeOH and [bumepy]BF$_4$ were stirred for 1 h before addition of other reaction components (Table 2, entries 28, 29). However, such pretreatment only caused a reaction yield decrease. The best results were always obtained when all reactants were added simultaneously to the autoclave.

The effect of methanol and palladium concentration was clearly demonstrated in a series of reaction experiments conducted at constant volume ($V = 8$ or 10 cm^3) (Table 3). To ensure the most comparable reaction conditions (first of all a constant concentration of the catalyst), depending on the amounts of methanol used, sufficient amounts of toluene (from 0.09 to 3.1 cm^3) were added to bring the total volume to $V = 8$ or 10 cm^3. Both series of reactions (in 8 and

10 cm^3) showed that a positive effect of methanol addition was observed up to the [MeOH]:[bumepyBF$_4$] ratio of ca. 3. The presence of toluene in the system generally caused a decrease in the reaction yield compared with systems in which only methanol was used. The effect of Pd colloid concentration on the yield of methoxycarbonylation is shown in Table 3. As could be expected, the yield of ester increased with an increase in Pd colloid concentration from 10% at [Pd] 4.9×10^{-4} M to 57% at [Pd] 2.24×10^{-4} M (entries 33–38).

To sum up the above-discussed results, one may conclude that at optimum reaction conditions, the reaction practically stopped when it reached 55–60% yield of ester and in spite of unreacted iodobenzene in reaction mixture did not proceed further. It appears, however, that a modification of the reaction procedure by constantly adding back the consumed amount of methanol led to some increase in substrate conversion and a higher yield of the product. So, when methanol was introduced to the reactor at the rate of 1.25×10^{-2} mol/h, the yield of ester could reach even 84% in spite of a decrease in the Pd concentration (Table 4). This experiment showed that the yield of carbonylation can be increased if methanol is added gradually in small portions instead of one big portion at the beginning of the reaction. This experiment additionally proved that Pd colloid does not undergo deactivation. The observed retardation of reaction over time is most probably caused by unfavorable phase composition making the catalyst–reagent contacts difficult.

Table 4
The yield of ester in methoxycarbonylation reaction of iodobenzene carried out with addition of 0.5 cm^3 (1.25×10^{-2} mol) of methanol/h

Entry	Time (h)	Yield of ester (%)
41	1	10
41.1	2	40
41.2	3	71
41.3	4	84

Reaction conditions: 1.17×10^{-5} mol [Pd]; 1.0 cm^3 (8.93×10^{-3} mol) PhI; 3.0 cm^3 (2.15×10^{-2} mol) NEt$_3$; 9.11×10^{-3} mol [bumepyBF$_4$]; 0.64 cm^3 (4.6×10^{-3} mol) mesitylene; p(CO) 5 atm, 90 °C.

Table 5
Methoxycarbonylation of iodobenzene with repeated use of catalyst

Entry	$[Pd] \times 10^3$ M	[MeOH]:[Pd]	[MeOH]:[IL]	$p(CO)$ (atm)	Time (h)	Yield of ester (%)
42, 1 cycle	1.91	900	1.3	5	3	50
42, 2 cycle	1.91	900	1.3	5	3	49
43, 1 cycle	1.70	1000	1.3	5	4	50
43, 2 cycle	1.70	1000	1.3	5	3	38
43, 3 cycle	1.70	1000	1.3	5	4	34
44, 1 cycle	1.70	1000	1.3	10	4	50
44, 2 cycle	1.59	2100	2.7	10	4	83
44, 3 cycle	1.59	2100	2.7	10	4	83
44, 4 cycle	1.59	2100	2.7	10	4	73
45, 1 cycle	1.74	1900	2.7	5	3	60
45, 2 cycle	1.74	1900	2.7	5	3	50
45, 3 cycle	1.74	1900	2.7	5	3	55
45, 4 cycle	1.74	1900	2.7	5	3	47
45, 5 cycle	1.74	1900	2.7	5	3	41
46[a], 1 cycle	1.77	1900	2.7	5	3	66
46[a], 2 cycle	1.77	1900	2.7	5	3	43
46[a], 3 cycle	1.77	1900	2.7	5	3	28

Reaction conditions: $1.0 \, cm^3$ $(8.94 \times 10^{-3}$ mol) PhI; $3.0 \, cm^3$ $(2.15 \times 10^{-2}$ mol) NEt_3; [bumepy]BF_4, [bumepy]PF_6 $(9.11 \times 10^{-3}$ mol); $0.64 \, cm^3$ $(4.6 \times 10^{-3}$ mol) mesitylene; $90 \,°C$.

[a] Reactions in [bumepy]PF_6.

What is an important attribute of any catalytic system, besides high productivity and selectivity, is stability of the catalyst and its recycling. Application of ionic liquids as reaction media makes it possible to separate the catalyst from the reaction products relatively easily by simple extraction with diethyl ether. It was analytically proved (using the ICP method) that the diethyl ether extract did not contain measurable amounts of palladium. Table 5 presents results of several experiments involving the recycling of Pd colloid dissolved in [bumepy]BF_4 or [bumepy]PF_6.

It was found that the Pd colloid was active in the second cycle, although conversion of iodobenzene in the first cycle did not reach 100%, which suggested some deactivation of the catalyst. The best results in experiments with repeated use of the catalyst were obtained in reactions in which in the first cycle the [MeOH]:[bumepyBF$_4$] ratio was 1.4 and in the second cycle the amount of methanol was doubled to [MeOH]:[bumepyBF$_4$] = 2.8 (Table 5, entry 44). The effect was similar to that observed in the experiment with repeated addition of methanol to the reaction mixture. In experiments in which five cycles of the reaction were conducted at [MeOH]:[bumepyBF$_4$] = 2.8, a decrease in reaction yield was observed from 60% in the first cycle to 41% in the fifth cycle (Table 5). In addition, the catalyst separated after each reaction was not deactivated when stored in air.

When the ionic liquid [bumepy]PF_6 was used, the yield of ester was initially higher (66%) but in the third cycle dropped to as low as 28% (entry 46). This may be explained by an effect of low stability of PF_6^- ions, which may undergo decomposition [21,22].

The methoxycarbonylation reactions were also performed with the addition of ammonium salts instead of ionic liquids in amounts similar to the amount of iodobenzene. The results

Table 6
Methoxycarbonylation of iodobenzene carried out in ammonium salts medium

Entry	Salt	Yield of ester (%)
47	[Bu$_4$N]Br	98
47a[a], 1 cycle	[Bu$_4$N]Br	72
47a[a], 2 cycle	[Bu$_4$N]Br	35
47a[a], 3 cycle	[Bu$_4$N]Br	14
48	[Bu$_4$N]I	95
49	[Bu$_4$N]Cl	88
50	[Et$_4$N]Br	68
51	[Et$_4$N]Cl	38
52	[Me$_4$N]Cl	0

Reaction conditions: 1.17×10^{-5} mol [Pd]; $1.0 \, cm^3$ $(8.94 \times 10^{-3}$ mol) PhI; $1.0 \, cm^3$ $(2.47 \times 10^{-2}$ mol) MeOH; $3.0 \, cm^3$ $(2.15 \times 10^{-2}$ mol) NEt_3; $0.64 \, cm^3$ $(4.6 \times 10^{-3}$ mol) mesitylene; [[R$_4$N]X] 9.11×10^{-3} mol; [[R$_4$N]X]:[Pd] = 779; [MeOH]:[salt] = 2.7; $90 \,°C$, $p(CO)$ 5 atm, reaction time 3 h.

[a] Reaction time 2 h.

presented in Table 6 show an extremely strong effect of ammonium salts on the reaction yield. The best results, yield of 88–98%, were obtained for catalytic systems with [^{n}BuN]$_4$X (X = Cl, Br, I). The use of [Et$_4$N]Cl is less effective (the yield of ester is 38–68%). No methoxycarbonylation reaction was observed in the system with [Me$_4$N]Cl. With recycled catalyst (Table 6, entries 47a) used consecutive decrease of catalytic activity was observed, more significant than in a case of ionic liquids.

3. Conclusions

Pd colloid stabilized with PVP demonstrated very attractive properties as a catalyst of methoxycarbonylation but only at carefully selected reaction conditions. Two reaction pa-
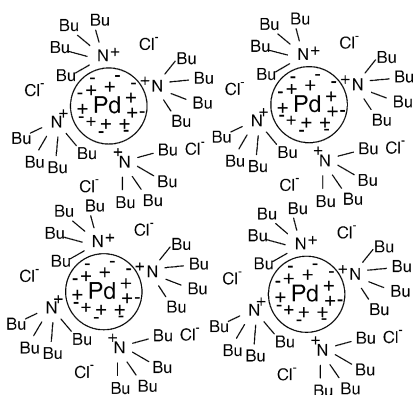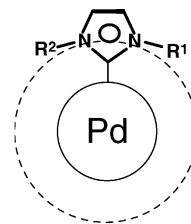
Scheme 2.

rameters are particularly important. First, the structure of the ionic liquid or ammonium salt used and, secondly, the concentration of methanol.

In terms of the yield of the methoxycarbonylation product, the effectiveness of the ionic liquids used decreased in the following order: $[^{n}Bu_4N]Br$ (98%) > $[^{n}Bu_4N]I$ (95%) > $[^{n}Bu_4N]Cl$ (88%) > [bumepy]PF_6 (83%) > $[Et_4N]Br$ (68%) > [bumepy]Cl (67%) > [bumepy]BF_4 (60%) > $[Et_4N]$Cl (38%) > [bmim]PF_6 (35%) > [bmim]BF_4 (18%) > [bmim]Cl (8%). Such an order can be related to the ability of salts to stabilize the initial or in situ formed forms of Pd colloid during the catalytic process. Interactions between the metal colloid and the ionic liquid are usually assumed to be electrostatic ones, depending on the formation of a double layer of ions surrounding the metal colloid crystallite, which prevents metal atom aggregation to bigger, inactive forms (Scheme 2).

Methoxycarbonylation reaction starts with the activation of the iodobenzene molecule via oxidative addition, leading to the formation of Ph–Pd–I fragments on the Pd colloid surface. XPS studies confirmed the formation of oxidized forms of palladium when Pd colloid was refluxed with an excess of PhBr [23]. Further interaction with an ammonium salt ($[^{n}Bu_4N]Br$) produced the $PdBr_4^{2-}$ anion [23]. Similar interactions can be proposed for systems containing Pd colloid and an ionic liquid.

It is worth noting that ionic liquids of the [bumepy]X type (X = PF_6, Cl, BF_4) were found to be much better than the [bmim]X type (X = PF_6, Cl, BF_4). This may be explained in terms of different reactivity of both cations, [bumepy] and [bmim], towards the Pd colloid. There are well-known literature examples of palladium carbene complexes formed in reaction with imidazolium salts [24–26]. In these carbene

Scheme 4.

complexes, a neutral imidazole ligand is coordinated to palladium (Scheme 3).

A similar reaction proceeding with C–H bond breaking is not possible in the case of the pyridinium cation. Formation of stable carbenes on the surface of Pd colloid may be competitive for coordination of aryl halide, the substrate of carbonylation, and therefore may inhibit the reaction (Scheme 4).

In fact, all ionic liquids with imidazolium cations (except [bmim]PF_6) were found to be inhibitors of carbonylation reaction.

4. Experimental

Palladium colloid was obtained according to [13].

Methanol, Et_3N and diethyl ether were purified using standard procedures [27]. Iodobenzene and mesitylene (POCh, Gliwice, Poland) were used without purification. Ionic liquids were purchased from Fluka and used as obtained.

4.1. Catalytic reactions

The reactions were carried out in a $130\,cm^3$ thermostated steel autoclave with magnetic stirring. Reagents: $1.0\,cm^3$ (8.93×10^{-3} mol) PhI, $3.0\,cm^3$ (2.15×10^{-2} mol) NEt_3, $0.64\,cm^3$ (4.6×10^{-3} mol) mesitylene as internal standard, methanol and an ionic liquid or ammonium salt (9.11×10^{-3} mol) were introduced to the autoclave in an N_2 atmosphere. Next, the N_2 atmosphere was replaced by CO. The reaction was carried out at $90\,°C$ for 3 or 4 h. Afterwards, the autoclave was cooled down and organic products were separated by extraction with diethyl ether (3 times with $3\,cm^3$) and GC–MS analyzed (Hewlett Packard 8452A).

Scheme 3.

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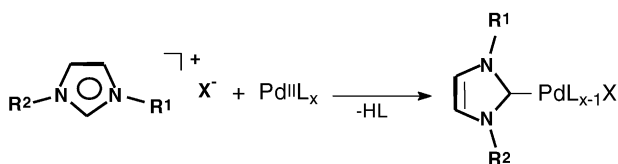
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