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Synthesis, characterization and catalytic activity in Suzuki-Miura and Mizoroki-Heck coupling reactions of trans-dichloro bis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex

Abstract. In this research palladium (II) chloride, PdCl₂, reacts with (4'-bromobiphenyl-4-yl) diphenylphosphine compound, Br(C₆H₄)₂P(C₆H₅)₂, to give trans-PdCl₂L₂ [L: Br(C₆H₄)₂P(C₆H₅)₂] complex. trans-dichloro bis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex has been characterized by elemental analysis, FT-IR, ¹H, ³¹P and ¹³C NMR. The catalytic performance of this complex has been rigorously evaluated and compared in the context of Suzuki-Miyaura and Mizoroki-Heck coupling reactions. It is noteworthy that under specific optimal conditions, including solvent type, base type, base quantity, catalyst quantity, and temperature, this complex serves as an efficient and suitable catalyst for the synthesis of biphenyl aryl halide derivatives. The resulting yield in these processes strongly supports the efficacy of this complex as a catalyst.

Key words: Phosphine, palladium, NMR analysis, Suzuki&Heck, coupling reactions, aryl halides.

Introduction

The ability to tune both the electronic and steric properties of phosphine ligands lie behind their importance in the development of effective homogeneous and heterogeneous catalysts, thus in recent years, the synthesis of phosphine ligands has attracted the attention of many chemical researchers due to their structure, unique characteristics and reactivity as well as the chemistry of their metal complexes. Organic chemistry that inspires the design of new phosphines with various structures and tuning their properties includes studies of the reactivity of phosphines, especially those based on the attack of a nucleophile on the carbon atom of an electrophilic substrate [1].

The utilization of an extensive array of trivalent phosphorus ligands in the realm of transition metal catalysis persists as a pivotal driver in the realm of conventional P-C-bonded phosphines. Concurrently, the domain of organocatalytic processes finds itself in a rapid state of proliferation within the annals of organic chemistry. This, in turn, serves as the well-spring of inspiration for the conception of novel phosphines, each possessing an assortment of structural attributes, inviting the meticulous refinement of their intrinsic properties [2]. What is certain tri-

phenylphosphine with IUPAC name: triphenylphosphane is a common organophosphorus compound with the formula P(C₆H₅)₃, that is frequently abbreviated as PPh₃ or Ph₃P. It is widely used in organic and organometallic compound synthesis because it is an effective reducing agent as well as a neutral ligand. This ligand binds well to most transition metals, especially those in the middle and late transition metals of groups 7–10. In terms of steric bulk [3].

Transition metal-catalyzed cross-coupling reactions of organic electrophiles with organometallic reagents represent one of the most important ways for the formation of carbon-carbon and carbon-heteroatom bonds, respectively [4]. A variety of organometallic reagents and organic halides, including aryl, alkyl, allyl, alkenyl and alkynyl groups, can be applied [5]. The advantages of cross-couplings over classical organic reactions are the easily accessible and inexpensive starting materials, versatile coupling methods, high productivity and selectivity as well as the tolerance concerning various functional groups and shorter reaction sequences [6].

In addition, C-C cross-coupling reactions are useful tools for the synthesis of complex structures, fine chemicals and natural products. However, the success of these transformations is highly dependent on the applied transition metal catalyst [7].

Generally, phosphines are widely used as a ligand for nickel or palladium catalysts in cross-coupling reactions. They are also effective ligands for rhodium, iridium and gold catalysts and used in catalytic reactions such as hydrogenation and cyclization reactions. The ability to fine-tune the reaction conditions (temperature, solvents, ligands, bases and other additives) of palladium catalysts makes palladium catalysis an extremely versatile tool in organic chemical synthesis [8].

As catalysts, typically late transition metals (*e. g.* Ni, Pd, Cu) are used and especially palladium attracted much attention during the last 50 years [9]. The advantages of palladium compared to other metals are its *non*-toxicity, its insensitivity toward oxygen and moisture and its tolerance toward numerous functional groups [10]. So, in our project, Pd is one of the most efficient metal elements in the field of catalytic processes and is commonly used as one of the most powerful tools for the formation of carbon-carbon bonds in the synthesis of organometallic compounds. Therefore, palladium-catalysed C-C cross-coupling reactions, especially the Suzuki and Heck reactions, have played an important role in chemical research at both industrial and laboratory scales. The Suzuki reaction is important to organic chemistry because it forms carbon-carbon bonds allowing the synthesization of various organic molecules and the Heck reaction, generally defined as the substitution of a vinylic hydrogen with an aryl, vinyl, or benzyl group, is widely regarded as one of the premier synthetic tools for the construction of new C-C bonds [11-15].

In this research paper, our investigation revolved around the synthesis of a novel phosphino Pd(II) complex, denoted as PdCl₂L₂ [L: Br(C₆H₄)₂P(C₆H₅)₂]. What is evident in this process is the use of cheap and available raw materials, performing organic reactions in shorter times compared to previous methods, and observing the principles of green chemistry and atomic economy. We thoroughly characterized this complex utilizing a variety of analytical techniques, including FT-IR, ¹H, ³¹P and ¹³C NMR spectroscopy. Furthermore, we explored the catalytic performance of the synthesized complex in carbon-carbon coupling reactions, specifically focusing on the Suzuki and Heck reactions.

Materials and methods

The present investigation employed chemicals and reagents sourced from Merck, Aldrich, and Fluka. Melting points were determined using the

IA9100 device by Electrothermal (England). The FT-IR spectra were acquired using a Bruker Vertex 70, utilizing potassium bromide pellets. NMR spectra were obtained on a 400 MHz Bruker spectrometer in CDCl₃ or DMSO as the solvent at room temperature. TLC was performed utilizing Merck silica gel 60 F254 plates that were pre-coated with a thickness of 0.25 mm.

Synthesis of (4'-bromobiphenyl-4-yl)diphenylphosphine, Br(C₆H₄)₂P(C₆H₅)₂, ligand: 10 mmol of 4,4'-dibromo-1,1'-biphenyl was dissolved in 190 mL of dry diethyl ether under a nitrogen atmosphere. Then, 10 mmol of n-butyl lithium was slowly added to the solution at -10°C, and the mixture was stirred for 2 h. In the following, the mixture was stirred for 1 h at room temperature. Afterward, 10 mmol of chlorodiphenylphosphine was added to the solution at -10°C, and the mixture was stirred for 9 h. Following this, the mixture was stirred for a period of 2 hours at room temperature. Upon the completion of the stirring process, dehydrated diethyl ether was added to the mixture. The product (4'-bromobiphenyl-4-yl)diphenylphosphine was subsequently isolated and underwent purification using dichloromethane. This process yielded 84.69% of the product in the form of a white powder, as shown in Scheme 1. **¹H NMR:** (250 MHz, CDCl₃, ppm) δ: 7.88 – 7.65 (m, 1H), 7.65 – 7.52 (m, 1H), 7.52 – 7.30 (m, 1H). **¹³C NMR:** (63 MHz, CDCl₃, ppm) δ: 143.43, 143.38 – 131.82 (m), 131.81 – 128.45 (m), 26.95 (d, J = 12.3 Hz), 122.59. **³¹P NMR:** (101 MHz, CDCl₃, ppm) δ: 28.99, -5.87 (d, J = 6.5 Hz). (Figure 1). **FT-IR (ν, cm⁻¹):** 3468, 3417, 2924, 2857, 1629, 1498, 1459, 1379, 1107, 1061, 990, 806, 622, 538, 496.

Synthesis of trans-dichlorobis (4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex as catalyst: 100 mg of (4'-bromobiphenyl-4-yl)diphenylphosphine was dissolved in 2 mL of dichloromethane. Then, 21.2 mg palladium(II) chloride, 0.5 mL acetone and 5 mL dichloromethane were added to the solution and the mixture were stirred at room temperature for 24 h. The separated solid was filtered off and washed with 2 mL methanol and 60 mL dichloromethane to give dichlorobis (4'- bromobiphenyl-4-yl)diphenylphosphine palladium(II) as a yellow powder [16, 19-22]. Yield: 63% (Scheme 2). **¹H NMR:** (300 MHz, CDCl₃) δ 7.75 (ddt, J = 15.4, 10.2, 4.4 Hz, 1H), 7.65 – 7.52 (m, 8H), 7.52 – 7.36 (m, 15H), 7.33 (s, 1H), 1.49 – 1.24 (m, 4H), 0.92 (q, J = 7.2 Hz, 2H). **¹³C NMR:** (75 MHz, CDCl₃) δ: 141.44, 138.58, 135.08 (d, J = 6.2 Hz), 134.62 (t, J = 6.2 Hz), 131.58, 130.23, 129.05, 128.78 – 126.93 (m), 126.02, 121.83, 77.01,

76.59, 76.16. $^{31}\text{P-NMR}$: (121 MHz, CDCl_3) δ : 32.42, 29.01, 23.00 (d, $J = 3.1$ Hz). (Figure 2). **FT-IR**(ν , cm^{-1}): 3440, 3052, 2924, 2856, 1706, 1661, 1588, 1471, 1429, 1378, 1185, 1094, 995, 844, 806, 741, 691, 518.

General method for Suzuki cross-coupling reaction: In a typical method, a mixture of aryl halide (0.25 mmol), phenylboronic acid (0.30 mmol), Potassium carbonate (0.375 mmol), 0.0012 g of dichloro bis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex as catalyst and 2 mL dimethylformamide as solvent was heated at 80°C using an oil bath for a suitable time. The reaction progress was monitored using TLC (n-hexane). After removing the solvent, an adequate amount of ethyl acetate was added to the mixture and poured into the separatory funnel containing distilled water. After separating the organic phase, the ethyl acetate was allowed to drain from the reaction vessel.

General method for Heck cross-coupling reaction: In a typical method, a mixture of aryl halide (0.25 mmol), *n*-butyl acrylate (0.30 mmol), Potassium carbonate (0.375 mmol), 0.0012 g of dichlorobis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex as catalyst and 2 mL dimethylformamide as solvent was heated at 80°C using an oil bath. The reaction was monitored by

employing TLC with a mobile phase consisting of a 9:1 combination of n-hexane and ethyl acetate. After the reaction was completed, the reaction mixture was allowed to cool to room temperature. Following removing the solvent, an appropriate amount of ethyl acetate was added to the mixture and poured into the Separatory funnel containing hot distilled water. After separating the organic phase, the ethyl acetate was allowed to drain from the reaction vessel.

Results and discussion

NMR analysis For the (4'-bromobiphenyl-4-yl)diphenylphosphine ligand. In the $^{31}\text{P-NMR}$ signal, Phosphorus resonance has appeared as a distinct single signal in the -5.87 ppm region. The position of the obtained signal compared to the signal of phosphorus (related to the free chlorodiphenylphosphine ligand with $\delta = -6$ ppm) indicates the formation of a new bond between the phosphorus atom with the carbon atom, which has caused the phosphorus signal to shift to a down field by reducing the electron density on the phosphorus atom. The signal seen in 28.99 ppm is related to (4'-bromobiphenyl-4-yl)diphenylphosphine oxide. The data show that the desired organic ligand has been synthesized with a high percentage of purity (95%) (Figure 1) [19, 20].

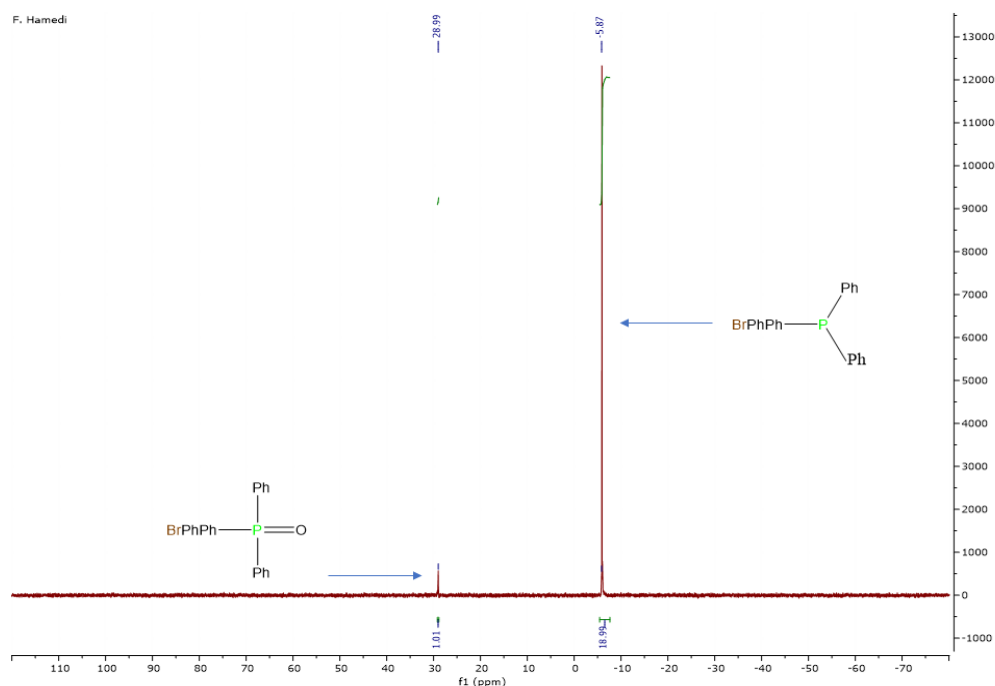


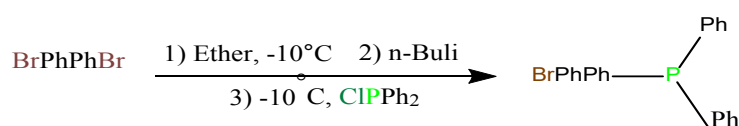
Figure 1 – ^{31}P NMR spectra of a (4'-bromobiphenyl-4-yl)diphenylphosphine ligand

In the ^1H NMR spectrum, the peak in the $\delta = 7.26$ ppm region is related to the CDCl_3 solvent. In this spectrum, hydrogens of aromatic rings have appeared as multiple peaks in the $\delta = 7.772\text{--}7.399$ ppm region (Scheme 1).

NMR analysis For the trans-dichlorobis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex. The ^{31}P NMR signals indicated a mixture of trans- PdCl_2L_2 , (4'-bromobiphenyl-4-yl)diphenylphosphine oxide and cis- PdCl_2L_2 ($\delta =$

32.42, 29.01, 23.00 ppm) respectively. It is clear that the signal corresponding to (4'-bromobiphenyl-4-yl)diphenylphosphine oxide remained unchanged, indicating that this compound did not react with PdCl_2L_2 (Figure 2) [16-23].

Based on the literature, an equilibrium mixture of the the trans and cis (trans/cis: 10/1) show that the phosphine ligands are predominantly (91%) coordinate in a trans configuration about the ion center (Scheme 2).



Scheme 1 – Synthesis of (4'-bromobiphenyl-4-yl)diphenylphosphine ligand

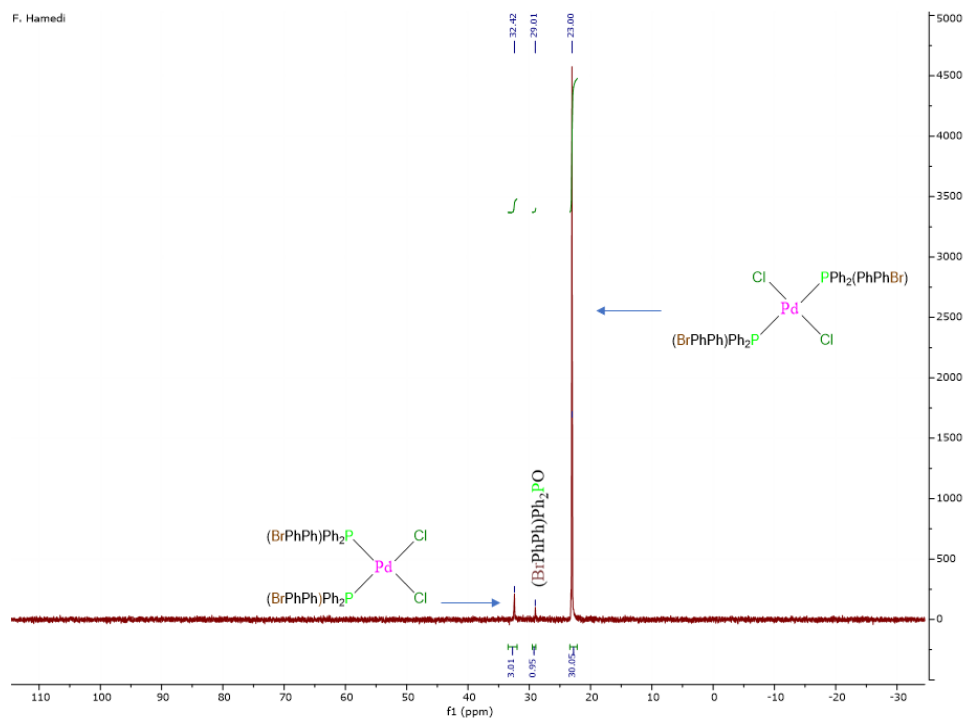
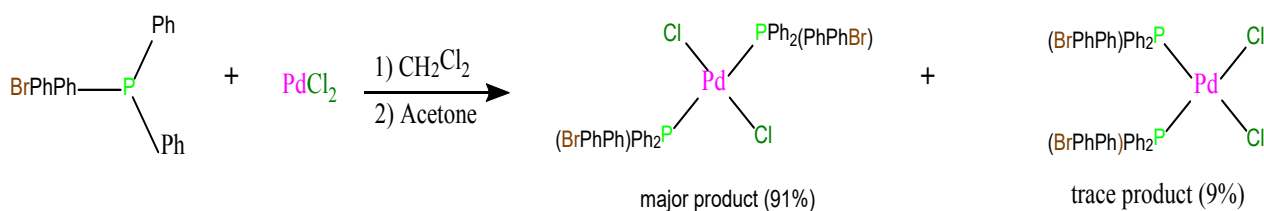


Figure 2 – ^{31}P -NMR spectra of dichlorobis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) as catalyst



Scheme 2 – Synthesis of trans-dichlorobis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex

FT-IR analysis for the (4'-bromobiphenyl-4-yl)diphenylphosphine ligand:

Aromatic compounds have characteristic infrared bands in five separate regions of the mid-infrared spectrum. The vibrational frequencies corresponding to the stretching of the C-H bonds in aromatic compounds are seen in the spectral region between 3100 and 3000 cm^{-1} (in this paper: 2924 cm^{-1}). This difference makes it possible to separate these bands from those due to aliphatic C-H groups, which appear at frequencies below the threshold of 3000 cm^{-1} . In the 2000-1700 cm^{-1} spectral region, a series of complex combination and overtone bands appear, showing a correspondence between the configuration of these overtone bands and the pattern of substitutions on the benzene ring (in this paper: not found). Skeletal vibrations, representing C=C stretching, absorb in the 1650 – 1430 cm^{-1} range (in this paper: 1629 and 1498 cm^{-1}). The C-H bending bands appear in the regions 1275 – 1000 cm^{-1} (in-plane bending) (in this paper: 1107, 1061 and 990 cm^{-1}), and 900 – 690 cm^{-1} (out-of-plane bending) (in this paper: 806 cm^{-1}). The vibrations associated with out-of-plane bending in aromatic compounds give rise to robust and distinctive bands, the intensity of which corresponds to the number of hydrogen atoms in the ring. Consequently, these bands can be used to elucidate the pattern of substitutions on the aromatic ring. The absorption regions for C-X stretching (X = F, Cl, Br or I) in organic halogen compounds observed in the 800 – 400 cm^{-1} range (in this paper: 622, 538 and 494 cm^{-1}). The absorption regions for aromatic P-C stretching appear in the 1450 – 1430 cm^{-1} range (in this paper: 1459 cm^{-1}) [24, 25].

FT-IR analysis for the trans-dichlorobis(4'-bromobiphenyl-4-yl)diphenylphosphine palladium(II) complex: Based on what was said above: The C-H stretching bands of aromatic compounds appeared in the 3052 cm^{-1} , the overtone bands observed in the 1706 cm^{-1} , the C=C stretching appear in the 1588 and 1471 cm^{-1} , the C-H bending bands appeared in the 1185 and 1094 cm^{-1} , the out-of-plane bending observed in the 691 cm^{-1} ($\nu_{\text{p-C}}$, vs), the C-X stretching appear in the 691 cm^{-1} and the aromatic P-C stretching appeared in the 1429 cm^{-1} ($\nu_{\text{p-Ph}}$, s) [24-26].

Catalytic study-Suzuki cross-coupling reactions:

The catalytic activity of the Pd complex was evaluated in the C-C coupling reactions of aryl halides with phenylboronic acid. Initial experiments were carried out to investigate the interaction between iodobenzene and phenylboronic acid. The aim was

to fine-tune the reaction parameters, which included choice of solvent, choice of base, amount of catalyst and temperature.

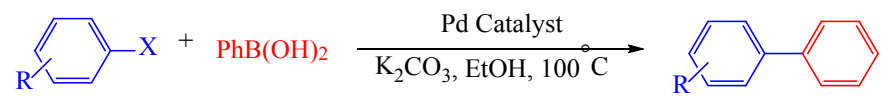
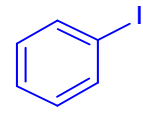
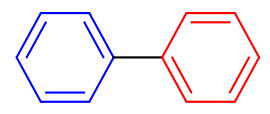
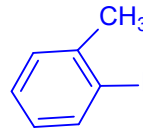
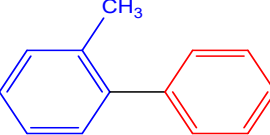
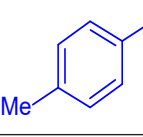
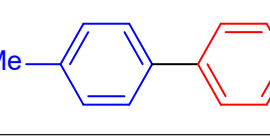
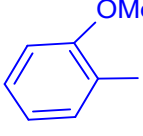
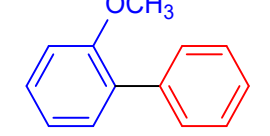
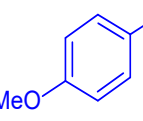
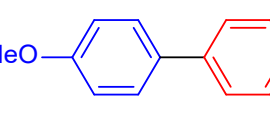
The results are presented in Table 1. During the preliminary refinement phase, we focused on evaluating the effect of a protic solvent on the reaction. Consequently, the prototypical reaction was carried out in a number of solvents, namely DMF, EtOH, i-PrOH and PEG. Among the range of non-aqueous solvents investigated, EtOH emerged as the frontrunner, providing superior yields of the coupled product (Table 1, entries 1-4). For the next stage, various inorganic bases were selected for investigation. Notable among these were K_2CO_3 , Na_2CO_3 , KOH and NaOH, each chosen to assess their influence on the desired reaction (Table 1, entries 5-8). As can be seen from the tabulated data, K_2CO_3 proved to be the optimum base for the reaction, giving the highest yield. In the third step, the reactions were carried out at different temperatures (as listed in Table 1, entries 9-11). In particular, the reduction in reaction temperature correlated with a decrease in yield, accompanied by an intrinsic increase in reaction time. Consequently, the optimum temperature was found to be 100°C, which provided a suitable optimum temperature. In the next stage, attention shifted to the amount of catalyst used. As predicted, the degree of variation in catalyst dosage had a pronounced effect on catalyst performance. Remarkably, an optimal amount was found to be 0.0007g of Pd complex, a revelation underlined by the data in Table 1 (entries 12-13). Conversely, a decrease in catalyst amount resulted in correspondingly lower yields, confirming the catalytic importance (Table 1, entry 4). As described in Table 4, the yield of biphenyl (Table 1, entry 10) could not be significantly increased by increasing the amount of catalyst to 0.0020 g. Therefore, from an economic point of view, 0.0007g of catalyst was identified as the optimum catalyst loading.

The optimized conditions are: 0.0518g of K_2CO_3 as base, 0.0007g of Pd complex as catalyst at 100°C in EtOH as solvent. Following the optimization of the experimental parameters, we embarked on an evaluation of the catalytic efficacy of the palladium complex towards a range of aryl halides. The results of these evaluations are summarized in Table 2. Various aryl iodides (Table 2, entries 1-5), bromides (Table 2, entries 6-7) and chlorides (Table 2, entries 8-9) were involved in successful reactions with phenylboronic acid in this study. In each case, the resulting yields ranged from good to excellent yields.

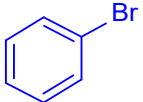
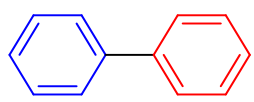
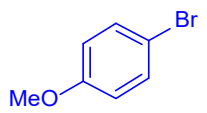
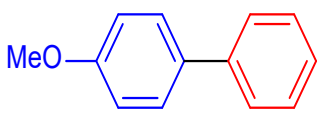
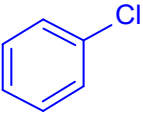
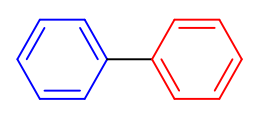
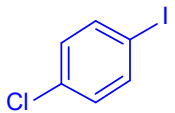
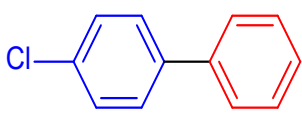
Table 1 – Optimization of reaction conditions for Carbon-Carbon cross coupling of iodobenzene with PhB(OH)₂

Entry	Catalyst (g)	Base	Base amount (g)	Solvent	θ (°C)	Time (min)	Yield ^a (%)
1	0.0012	K ₂ CO ₃	0.0518	DMF	80	40	45
2	0.0012	K ₂ CO ₃	0.0518	EtOH	80	40	90
3	0.0012	K ₂ CO ₃	0.0518	PEG	80	40	50
4	0.0012	K ₂ CO ₃	0.0518	i-PrOH	80	40	60
5	0.0012	K ₂ CO ₃	0.0518	EtOH	80	40	90
6	0.0012	Na ₂ CO ₃	0.0397	EtOH	80	40	70
7	0.0012	KOH	0.0210	EtOH	80	40	80
8	0.0012	NaOH	0.0149	EtOH	80	40	75
9	0.0012	K ₂ CO ₃	0.0518	EtOH	60	40	65
10	0.0012	K ₂ CO ₃	0.0518	EtOH	80	40	90
11	0.0012	K ₂ CO ₃	0.0518	EtOH	100	40	95
12	0.0007	K ₂ CO ₃	0.0518	EtOH	100	40	95
13	0.0020	K ₂ CO ₃	0.0518	EtOH	100	40	92

Table 2 – The optimal conditions for the Suzuki C-C coupling of aryl halides

						
Entry	Aryl halide	Product	Time (min)	Yield (%) ^b	Melting point (°C)	
					Measured	literature
1			40	95	68	[27]
2			27	90	Pale yellow liquid	[27]
3			25	93	45-47	[28]
4			30	90	Pale yellow liquid	[29]
5			20	96	86-88	[28]

Continuation of the table

6			40	90	68	[27]
7			30	89	86-88	[28]
8			50	85	68	[27]
9			70	75	80	[30]

Catalytic study-Heck cross-coupling reactions: Following an investigation of the Suzuki reaction with various aryl halides using a Pd complex as a catalyst, our aim was to evaluate the catalytic efficacy of the aforementioned catalyst in Heck coupling reactions.

Therefore, we studied the effect of non-aqueous protic solvents, inorganic bases, different amounts of optimum base, different amounts of catalyst and various temperature on the model reaction involving iodobenzene and n-butyl acrylate catalyzed by a Pd complex (Table 3). In the initial phase of optimization, a comprehensive investigation was carried out to measure the influence of various solvents, including DMF, EtOH, i-PrOH and PEG. In particular, polyethylene glycol (PEG) demonstrated superior solvent properties when compared to the other solvents investigated. Furthermore, the use of polyethylene glycol (PEG) as a solvent resulted in a significant increase in production efficiency, reaching a remarkable maximum of 95%. (Table

3, entry 1-4). The next stage was to explore the implications of using different bases in the context of the Heck reaction carried out in the PEG solvent. The selection included K_2CO_3 , Na_2CO_3 , NaOH and KOH, each of which was carefully evaluated (Table 3, entries 5-8). Based on the data presented in Table 3, K_2CO_3 was identified as the optimal base of choice. Moving on to the third step, a meticulous exploration of different amounts of the optimal base, ranging from 0.0518 g to 0.1036 g, was carried out for this reaction. In particular, 0.1036 g of K_2CO_3 was found to be the optimal dosage (Table 3, entries 9-11). The next step involved a meticulous examination of different amounts of catalyst, ranging from 0.0007g to 0.0020g, in search of the optimum performance of the reaction. Notably, 0.0012 g of catalyst appeared to be the optimal choice (Table 3, entries 12-14). In the fifth progression, a substantial augmentation in the reaction's progress was obtained by increasing the temperature from 80 to 120°C (Table 3, entries 15-17).

Table 3 – Optimizing conditions for the C-C coupling of iodobenzene with n-butyl acrylate

Entry	Catalyst (g)	Base	Base amount (g)	Solvent	θ (°C)	Time (min)	Yield ^a (%)
1	0.0012	K_2CO_3	0.0518	DMF	80	20	25
2	0.0012	K_2CO_3	0.0518	EtOH	80	20	35
3	0.0012	K_2CO_3	0.0518	PEG	80	20	95
4	0.0012	K_2CO_3	0.0518	i-PrOH	80	20	20
5	0.0012	K_2CO_3	0.0518	PEG	80	20	95
6	0.0012	Na_2CO_3	0.0397	PEG	80	20	50

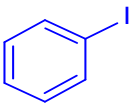
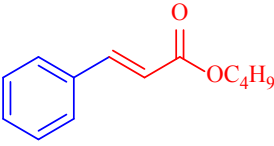
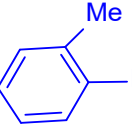
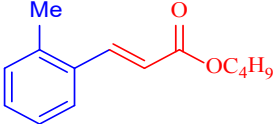
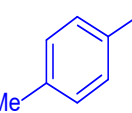
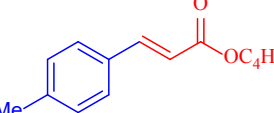
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Entry	Catalyst (g)	Base	Base amount (g)	Solvent	θ (°C)	Time (min)	Yield ^a (%)
7	0.0012	KOH	0.0210	PEG	80	20	45
8	0.0012	NaOH	0.0149	PEG	80	20	70
9	0.0012	K ₂ CO ₃	0.0518	PEG	80	20	90
10	0.0012	K ₂ CO ₃	0.0691	PEG	80	16	91
11	0.0012	K ₂ CO ₃	0.1036	PEG	80	10	95
12	0.0007	K ₂ CO ₃	0.1036	PEG	80	20	85
13	0.0012	K ₂ CO ₃	0.1036	PEG	80	10	95
14	0.0020	K ₂ CO ₃	0.1036	PEG	80	8	95
15	0.0012	K ₂ CO ₃	0.1036	PEG	80	10	95
16	0.0012	K ₂ CO ₃	0.1036	PEG	100	7	96
17	0.0012	K ₂ CO ₃	0.1036	PEG	120	5	98

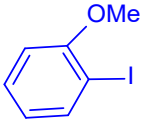
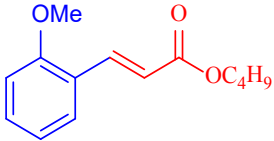
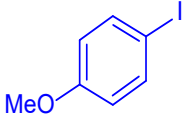
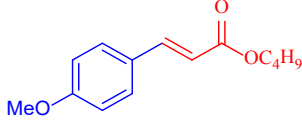
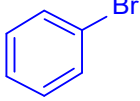
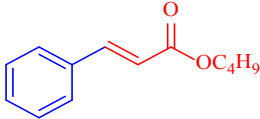
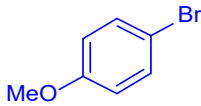
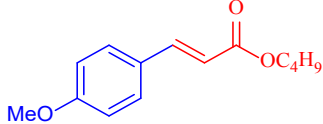
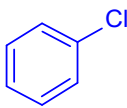
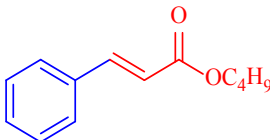
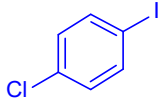
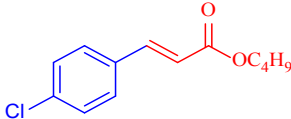
The reaction with activated aryl halides was easily carried out at 120°C. Therefore, the reaction temperature was maintained at 120°C, which was determined to be the optimum temperature. As Table 3 reveals, the optimal conditions involved using a Pd complex (0.0012 g) within a PEG medium, with 0.1036 g of K₂CO₃ as the base, all operating

at 120°C. Following the fine-tuning of these reaction parameters, we expanded the capabilities of this catalytic system to facilitate the reaction of n-butyl acrylate with a diverse range of aryl halides, encompassing both electron-donating and electron-withdrawing groups. The outcomes under these optimized conditions are presented in Table 4.

Table 4 – Heck C-C coupling of aryl halides under ideal conditions

Entry	Aryl halide	Product	Time (min)	Yield (%) ^b	Melting point (°C) Measured literature
1			5	98	Pale yellow liquid [27]
2			19	89	Pale yellow liquid [28]
3			6	90	Pale yellow liquid [28]

Continuation of the table

4			40	87	Pale yellow liquid [31]
5			20	95	Pale yellow liquid [32]
6			25	90	Pale yellow liquid [32]
7			35	85	Pale yellow liquid [31]
8			85	80	Pale yellow liquid [29]
9			25	80	Pale yellow liquid [23]

In all cases, we obtained high yields, ranging from good to excellent.

Conclusion

In this study, we prepared the trans-Pd(II) ion catalyst and systematically characterized its structure using NMR and FT-IR. Furthermore, the trans-Pd(II) ion complex was employed as a highly efficient and precisely defined catalyst for the facilitation of Suzuki and Heck cross-coupling reactions involving various aryl halides, leading to the production of corresponding products with yields ranging from

moderate to excellent. Notably, the catalyst exhibited remarkable activity.

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Conflict of interest

All authors are aware of the article's content and declare no conflict of interest.

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