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Original article

Synthesis and characterization of palladium complexes in combination with Phosphonium salts of 1,3-bis (chloro-methyl)-*benzene* and benzyl bromide by using stereoscopy methods

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ABSTRACT

Phosphonium salts have the general formula $R_4P^+X^-$ and are derived from the sub-ion phosphonium tetrahedral(pH_4^+), such as phosphonium iodide $[p(CH_3)_4^+]^-$. Generally, phosphonium is attributed to an organic derivative, such as tetra-phenyl phosphonium chloride and tetra tetra-methyl phosphonium iodide. In this project, phosphonium compounds $[C_6H_5(CH_2pph_3Br)]$, $\{C_6H_4$ -1- (CH_2Cl) -3- $(CH_2pph_3Cl)\}$ and palladium complexes, $[C_6H_5(CH_2pph_3)]_2[pd_2cl_2Br_2]$, $[C_6H_4$ -1- $CH_2Cl)$ -3-$(CH_2pph_3)]_2$ $[pd_2Cl_6]$ were prepared and characterized. Phosphonium salts was prepared from the reaction of 1,3-bis (chloro-methyl)-*benzene* and tri-phenyl phosphine. Then, through the reaction between phosphonium compounds and $Na_2[pd_2Cl_6]$ final complexes were prepared. Next phosphonium salt was prepared from the reaction between benzyl bromide and tri-phenyl phosphine. Then, final complexes were prepared through a reaction between phosphonium compounds and $Na_2[pd_2Cl_6]$.

1. Introduction

Alkyl tri- phenyl Phosphonium salts are used to prepare Wittig reagents in Wittig reaction, widely. Such salts are made easily by using tri-phenyl phosphine reaction with an Alkyl halide. If the alkyl, methyl or primary group alkyl have not space prevention, the reaction well done. But usually react with secondary alkyl halides is a weak reaction. Tertiary alkyl groups can’t form Aylyd. Phosphonium salt is a stable compound which often is achieved from ethanol, by recrystallization (Hertly, 1994). In recent years, Phosphonium salts are of great interest due to their structure, properties and reactivity.

Aylyd reaction with aldehydes or ketones, which is one of the most common methods for the formation of region specific alkenes, was first reported in 1953 by George Wittig. This reaction is a key step to produce a large number of natural and biological products. The Main reagent in the Wittig process is prepared through the deprotonated Phosphonium salt, which has been achieved through four coordination of phosphine with a combination of Hollow. The main advantage of Wittig reaction is it’s stereo selectivity. Stereochemistry depends on several factors. Phosphonium salt structure is The presence of metal cations and laboratory conditions (Moussaousi and Said, 2006).

Wittig reaction in addition to done applied research, has broad applications to produce pharmaceutical and industrial materials. Wittig, to start the reaction, react an alkyl halide with a phosphine to obtain four coordinate kation phosphine. To produce a neutral compound, which is called P aylyd, this cation is then deprotonated by a strong base such as butyl lithium. Aylyd negatively charged carbon, acts as nucleophile and attacks to carbonyl Aldehyde group or ketone. Protective and corrosion inhibitor metals play an important role in industry. Most of protectors and inhibitors are organic compounds which have hetero atoms such as phosphorus, nitrogen, sulfur and oxygen. These compounds and their derivatives are corrosion inhibitors for iron and steel. Many of these corrosion inhibitors and protective have hazardous effects on the environment. So achieving effective and non-toxic organic inhibitors for iron and steel is a crucial point. Suitable inhibitors for the environment should have biodegradability properties and should not be destructive for environment. Phosphonium compounds in Acidic solutions are used as protective for most metals (Bhrara et al., 2008).

Four coordinated Phosphonium compounds have been identified in many systems as factors inhibiting the growth of bacteria, which are highly adapted to the environment (Incomopoulou et al., 2005). The benefits of these compounds including very low toxicity, rapid degradation in the environment (via hydrolysis, oxidation, light degradation and biodegradation), and destructive or non-destructive effects on the environment (Aldridge et al., 2007). For example, Tri phenyl phosphonium bromide in 0/5 M sulfuric acid solution is known as a corrosion inhibitor for iron and steel, and has been proven by Galvanostatic and Potentiostatic measurements and using FT-IR spectroscopy and images from electron microscopy and quantum computing (Kenawy et al., 2006). Also, some reports provided about the preparation of biologically active polymers using ammonium groups and Four coordinated Phosphonium, which have medical use (Cieniecka-Roslonkiewicz et al., 2005).

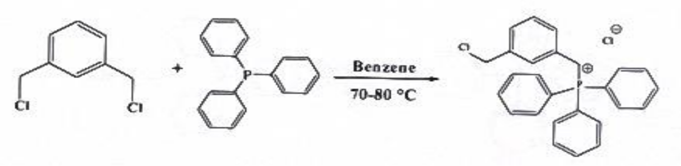
Production of ionic liquids is another application of phosphonium salts, which the liquids are organic salts which are in liquid state at low temperatures (less than 100 °C) (Tseng et al., 2007) and has advantages such as wide temperature range of liquid output, high thermal and chemical stability, strong solvents and very low vapor pressure in the synthesis of organic compounds, chemical catalysts, different separation methods and the role of new electrolytes in the manufacture of solar and fuel cells (Sons and Gong, 2002). In recent years, many studies have been done on Four coordinated Phosphonium systems and the new composition of the phosphonium known as ionic liquids and the use of phosphonium salts is more than ever (Wang and Tian, 2007).

In recent years, important polymer humidity sensors are built. Different types of monomers sensitive to moisture have been used to build the humidity sensors, including cationic or anionic salt. Phosphonium salts due to the simplicity and easy preparation and high stability under different conditions are used, in a very large extent, for the manufacture of polymer humidity sensors. Most recently, tri-phenyl phosphonium chloride salts have been prepared and used for the production waterproof humidity sensors. You can also use Tri-butyl phosphonium chloride, as monomer, to prepare the sensors (Terada and Kouchi, 2006). Phosphonium salts successfully have been used as a catalyst for many types of organic reactions such that it can be notedis simple phosphonium salt catalytic activity as benzyl chloride phosphonium Alkoxysilyl cyano reaction of the ketones (Nandurkar and Bhanage, 2008) and the use of phosphonium salt as the catalyst Lewis acid without metal, which has been rapid growth in the field of organic catalysts (Terada and Kouchi, 2006).

2. Experimental method

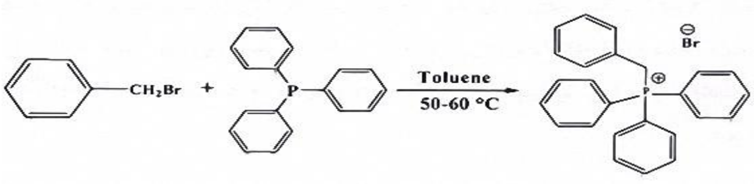
2.1. The compound procurement process $[C_6H_5(CH_2pph_3Br)]$

In this project, in a flask of 200 ml, 5.25 g (0 m mole) of 1, 3-bis (Chloromethyl) benzene were dissolved in 50 ml of benzene, to prepare the compound (3 (Chloromethyl) benzyl) phenyl phosphonium chloride $\{C_6H_4 - 1 -$ $CH2cl–3–CH2pph3Cl$. Then 7.86 g (30 m mole) phenyl phosphine were dissolved in 50 ml of benzene, separately, and then was added to the flask. According to the following reaction, the reaction mixture was stirred for 48 hours at a temperature of 80-70 °C. After this period, we clear solution, a white precipitate was obtained, the precipitate washed with diethyl ether and then dried in desiccator. The compound decomposes at temperatures above 334 °C. The resulting sediment weight is 7.007 and the reaction efficiency is 53%.



The compound procurement process $[C_6H_4 - 1 - (CH_2Cl) - 3 - (CH_2pph_3Cl)]$.

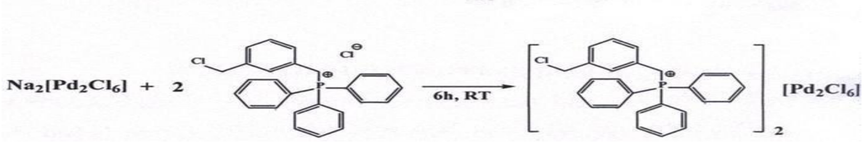
To prepare the benzyl tri-phenyl phosphonium bromide compound, in a flask of 100 ml, 5.24 g (20 m mole) of tri-phenyl phosphine were dissolved in 30 ml of toluene. Then 3.24 g (20 m mole) of benzyl bromide liquid were added to flask, little by little, According to the following reaction, the reaction mixture is stirred for 48 hours at a temperature of 50-60 °C. After this period, we clear solution, a white precipitate is obtained, the precipitate washed with diethyl ether and then dried in desiccator. The compound had a melting point of 293 °C and obtained sediment weight was 8.114, and the reaction efficiency is 94%.



Procurement of palladium complexes.

2.2. Procurement of $[C_6H_4 - 1 - (CH_2Cl) - 3 - (CH_2pph_3)]_2[pd_2cl_6]$ complex

At this stage, 0.0266 g (0.15 m mole) palladium chloride shed in a 20 ml tube hose, 0.00877 g (0.15 m mole) of sodium chloride are added to it. Then 10 ml methanol is added and until a clear solution is achieved stirred for 18 hours at room temperature (Nandurkar and Bhanage, 2008). Then 0.065 g (0.015 m mole) phosphonium compound, are added to the clear solution which immediately began to precipitate out, According to the following scheme, and the reaction mixture is stirred for 6 hours at room temperature. After that time we straightened deposits, and the obtained sediment washed with methanol and diethyl ether and then dissolved in dichloromethane to be pure. This compound has a melting point of 266 ℃, the resulting sediment weight was 0.079 g and the reaction efficiency is 42%.

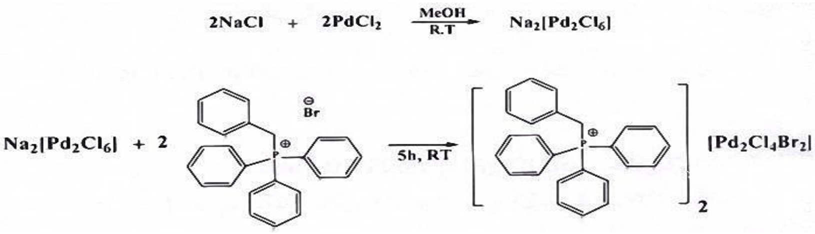


Layout (2-1) complex formation process $[C_6H_4 - 1 - (CH_2Cl) - 3 - (CH_2pph_3)]_2[pd_2cl_6]$.

2.3. Procurement of $[C_6H_5(CH_2PPH_3Br)]_2[pd_2Cl_4Br_2]$ complex

First, 0.055 g (0.31 m mole) of palladium chloride is poured in a 20 ml tube hoses, and then 0.018 g (0.31 m mole) of Sodium chloride is added to. Then 10 ml of methanol is added and to obtain a clear solution is stirred for

18 hours at room temperature (Nandurkar and Bhanage, 2008). Then we add the 0.134 g (0.31 m mole) of phosphonium complex to the transparent solution which immediately began to precipitate out (According to the following scheme). The reaction mixture was stirred for 5 hours at room temperature. After this time filtered deposits and the resulting precipitate washed with methanol and diethyl ether, and then, an order to purify, dissolved in dichloromethane. This compound has a melting point of 262 ℃, the resulting sediment weight is 0.206 g and the reaction efficiency is 54%.



Layout (2-2) $[C_6H_5(CH_2PPH_3Br)]_2[pd_2Cl_4Br_2]$ formation process.

3. Results and discussion

In order to verify the obtained compounds, NMR ranges include HNMR, CNMR, PNMR were measured by FT-NMR400 MH2, and infrared spectra were measured by FT-IR, BOMMEN model and using noujol oil and KBR tablet and mass spectra were measured by the Agilent technology (HP). The outcome of which is as follows:

1 spectra: Shows $\{C_6H_4 - 1 - (CH_2cl) - 3 - (CH_2PPh_3Cl)\}$ composition

In this spectra, the resonance of phosphorus appeared in $\delta = 23.66\,ppm$ as a identified unique peak, the peak position compared with the peak of phosphorus regarding free ligand of phenyl phosphine which appear in $\delta = 4.8\,ppm$, which represents a phosphorus atom is formed new bond with carbon atom, which by reducing electron density on the phosphorus atom, causes phosphorus peak shift to lower field. Also, the peak for similar compounds has appeared in the $\delta = 24.22\,ppm$ (Naghipour et al., 2008). These peaks represent formation of the desired compound. The uniqueness of obtained peak phosphorus without a second peak in the spectra represents a high purity compound prepared.

Spectra 1

Spectra 2

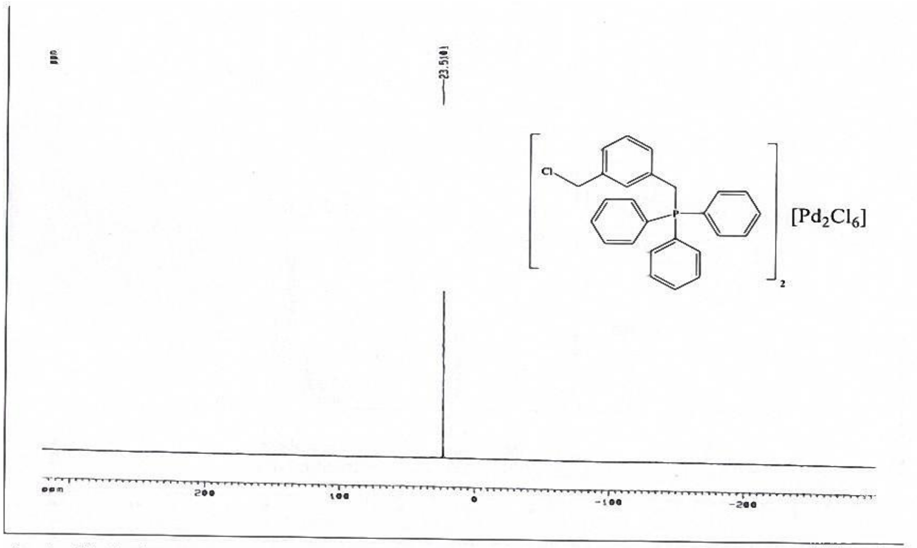
HNMR show the $\{C_6H_4 - 1 - (CH_2cl) - 3 - (CH_2PPh_3Cl)\}$ compound

In this spectra, a unique peak at $\delta = 42.27\,ppm$ correspond to methyl group hydrogen attached to the chlorine, and a double peak in $\delta = 5.48\,ppm$ correspond to attached methyl group hydrogen to the phosphorus, which has $2j_{p-h} = 15.01\,Hz$ coupling constant, and multiple peaks in the $\delta = 6 - 95 - \frac{7}{72}\,ppm$ correspond to the aromatic hydrogen in the compound, have appeared. Summary of results of the spectra are presented in the below Table.

Peak shape	**Number of protons**	$\delta(ppm)$	**Type of proton**
A unique peak	2	4.27	CH_2Cl
A double peak	2	5.48	CH_3P
Multiple peaks	19	6.95-7.72	$3C_6H_5$, C_6H_4

HNMR shows the $[C_6H_4 - 1 - (CH_2cl) - 3 - (CH_2PPh_3)]_2[pd_2Cl_6]$ compound

In this spectra, the phosphorus resonance has appeared in the form of a specified unique peak at $\delta = 23.51\,ppm$. The peak position, compared to free ligand of tri phenyl phosphine, has been transferred to lower field. But the phosphorus peak position in the compound, compared to phosphonium compound used to form the compound, had not much movement. So it can be concluded that the obtained combination is a counter ion and makes the palladium compound be stable. The unique shape of phosphorus obtained peak, without other peaks in the spectra, indicating that the produced compound has a high purity.



Spectra 13: $^{31}P\{^1H\}$ NMR $[C_6H_4\text{-}1\text{-}(CH_2Cl)\text{-}3\text{-}(CH_2PPh_3)]_2[Pd_2Cl_6]$ compound in DMSO-d^6 solvent

Spectra 13

Spectra 14

HNMR show the $[C_6H_4 - 1 - (CH_2cl) - 3 - (CH_2PPh_3)]_2[pd_2Cl_6]$ compound

In this spectra a unique peak at $\delta = 2.49\,ppm$ which correspond to the dimethyl sulfoxide solvent, and a unique peak at $\delta = 3.27\,ppm$ which correspond to the methanol solvent, and a unique peak in $\delta = 4.28\,ppm$ correspond to the hydrogen of methyl group attached to chlorine, and a double peak at $\delta = 5.14\,ppm$ correspond to the hydrogen of methyl group attached to phosphorus, which has $2j_{p-h} = 15.01\,Hz$, and multiple peaks at $\delta = 6.97 - 7.94\,ppm$ correspond to the existing aromatic hydrogen in combination, is appeared. Summary of results are presented in the following Table.

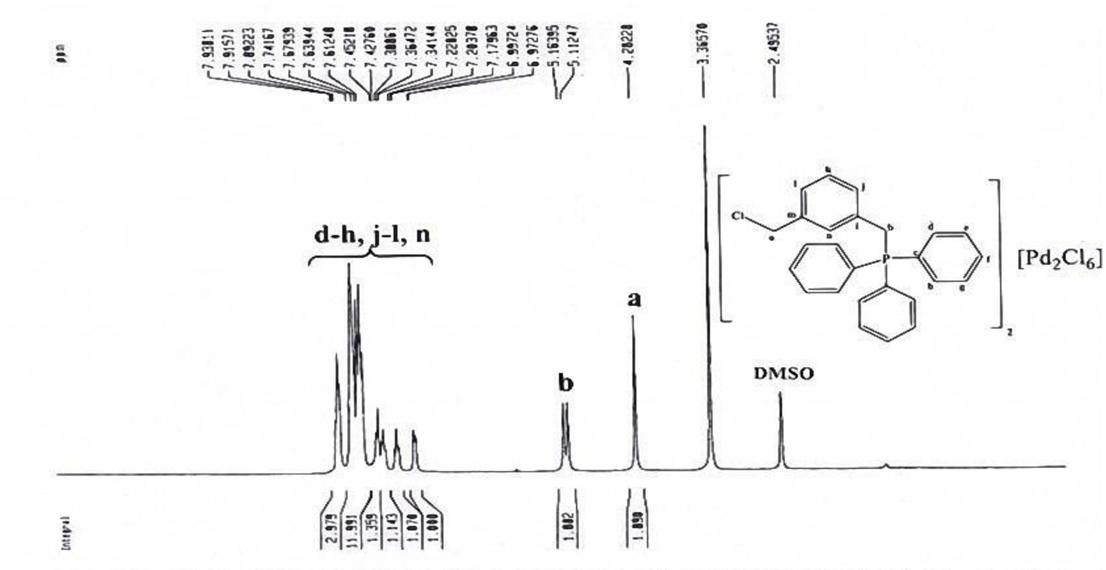
Peak shape	Number of protons	$\delta(ppm)$	Type of proton
A unique peak	2	4.28	CH_2Cl
A double peak	2	5.14	CH_2P
Multiple peaks	19	6.97-7.94	$3C_6H_5$, C_6H_4

Spectra 15

C$\{H\}\ NMR$ shows the $[C_6H_4 - 1 - (CH_2cl) - 3 - (CH_2PPh_3)]_2[pd_2Cl_6]$ compound

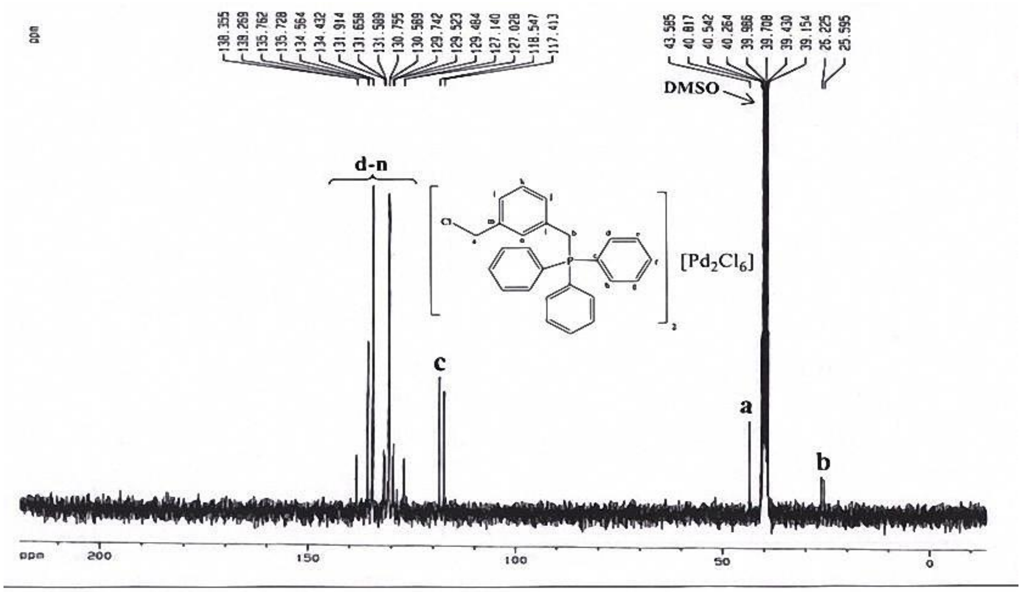
In this spectra a double peak at $\delta = 25.90\,ppm$ correspond to the carbon of methyl group attached to the phosphorus, which has $1j_{p-h} = 47.55\,Hz$, and existing peaks at $\delta = 39.15 - 40.82\,ppm$ correspond to the dimethyl sulfoxide solvent and A unique peak at $\delta = 43.58\,ppm$ carbon of the methyl group attached to the chlorine, and a double peak at $\delta = 117.98\,ppm$ which has $1j_{p-h} = 86.04\,Hz$, correspond to the phenyl carbon which attached to the phosphorus, also, the existing peaks at $\delta = 127.04 - 138.35\,ppm$, correspond to Carbon of the benzene ring Phenyl in the compound. Summary of results are presented in the Table below.

Peak shape	$\delta(ppm)$	Carbon type
A double peak	25.90	CH_2P
A unique peak	43.58	CH_2Cl
A double peak	117.98	P-C
Multiple peaks	127.03-138.35	Aromatic



Spectra 14: ^{1}H NMR [C_6H_4-1-(CH_2Cl)-3-(CH_2PPh_3)]$_2$ [Pd_2Cl_6] compound DMSO-d^6 , solvent

Spectra 14



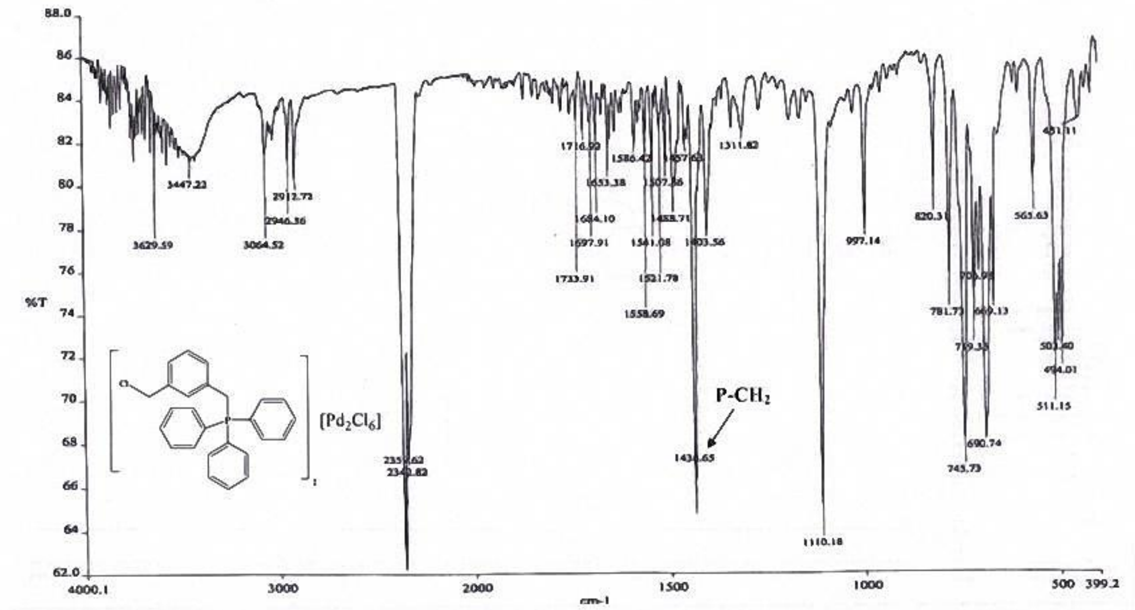
Spectra 15: ^{13}C{^{1}H} NMR , [C_6H_4-1-(CH_2Cl)-3-(CH_2PPh_3)]$_2$ [Pd_2Cl_6] compound in DMSO-d^6 , solvent

Spectra 15

Spectra 16

IR shows the $[C_6H_4 - 1 - (CH_2cl) - 3 - (CH_2PPh_3)]_2[pd_2Cl_6]$ compound

In the spectrum, absorption of bending vibration C-H, two rings of substituted single ring substituent of phenyl from 600-90 cm^{-1} are observed. As well as absorption in 3.64 cm^{-1} area is related to C-H stretching vibration in phenyl rings. Absorptions in 1457-1653 cm^{-1} area are related to $C = C$vibrations in phenyl rings. And strong absorption in the region in 1437 cm^{-1} is also related to vibrations in P-CH_2 bonds and absorption related to Carbon-phosphorus bond vibrations in phenyl.



Spectra 16: (KBr) $[C_6H_4\text{-}1\text{-}(CH_2Cl)\text{-}3\text{-}(CH_2PPh_3)]_2\,[Pd_2Cl_6]$ compound

Spectra 16

The results of elemental analysis of this compound based on $C_{52}H_{46}CL_8p_2pd_2$ empirical formulas is given in Table 1.

Table 1

Elemental analysis data of $[C_6H_4-1-(CH_2cl)-3-(CH_2PPh_3)]_2[pd_2Cl_6]$ compound.

$C_{52}H_{46}CL_8p_2pd_2$	%C	%H	%N
Experimental	51.20	4.00	-
Theoretical	51.16	3.97	-

Checking the structure of $[C_6H_5(CH_2PPh_3Br)]_2[pd_2Cl_6Br_2]$ compound

21 Spectra

$p\{1H\}\,NMR$ shows the $[C_6H_4-1-(CH_2cl)-3-(CH_2PPh_3)]_2[pd_2Cl_6]$ compound

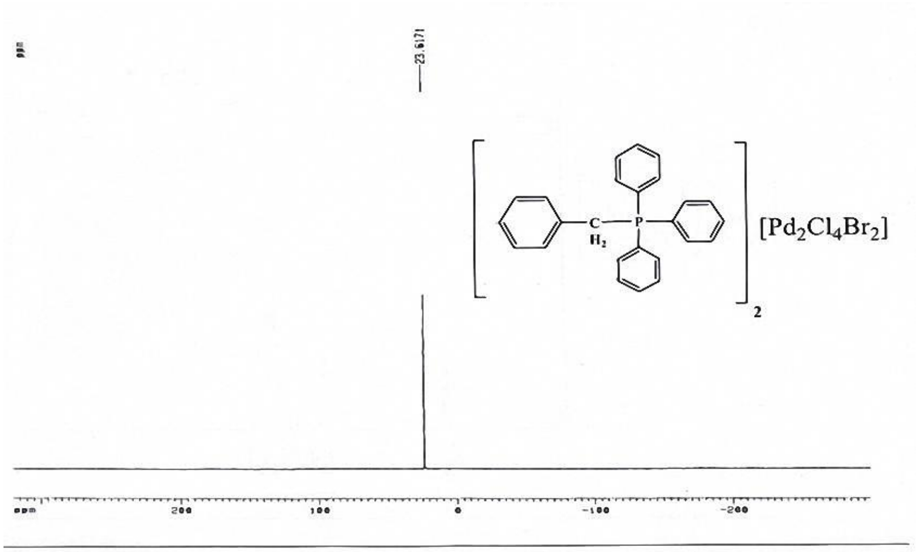
In the spectrum, Phosphorus resonance in the form of an identified unique peak appeared in $\delta = 23.62\,ppm$ area. Peak location, compared with free ligand in phenyl phosphine, has been transferred to lower field. But the Phosphorus peak position, compared with the phosphonium compound which used to formation of this compound, has not significant displacement. Thus it can be concluded that the obtained combination is a counter ion and causes the palladium compound be stable. The unique shape of obtained phosphorus peak without any other peaks represents the high purity of prepared compound.

Spectra 22

HNMR shows the $[C_6H_5(CH_2PPh_3Br)]_2[pd_2Cl_6Br_2]$ compound

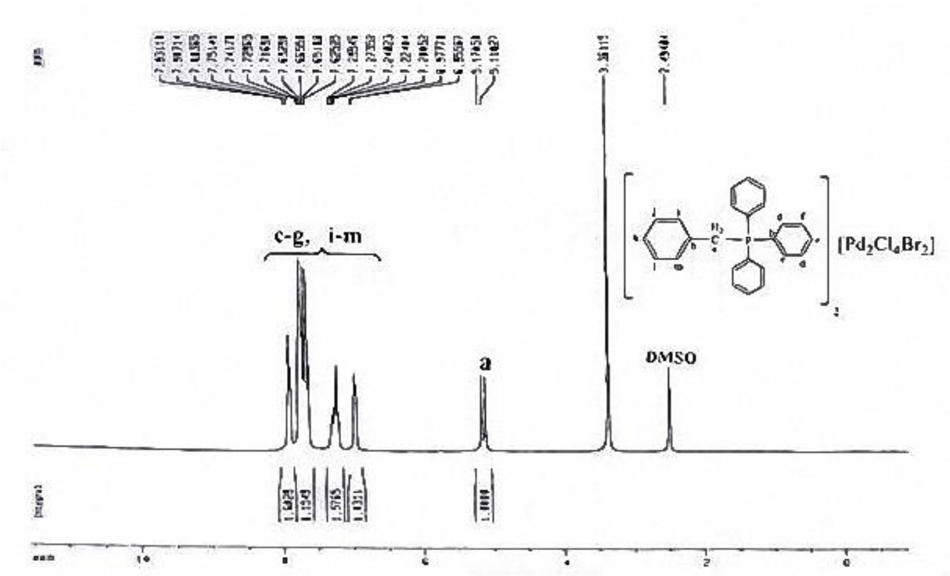
In the spectra, a unique peak was achieved at $\delta = 2.49\,ppm$ which was related to dimethyl sulfoxide solvent, a unique peak at $\delta = 3.36\,ppm$, related to methanol solvent, and a double peak at $\delta = 5.14\,ppm$, related to the hydrogen of a methyl group attached to the phosphorus, which has $2j_{p\text{-}H}$ =015.01 Hz, and multiple peaks related to hydrogen aromatic ring at $\delta = 6.95 - 7.93\,ppm$. Summary of results are presented in the Table below.

Peak shape	Number of protons	$\delta(ppm)$	Type of proton
A double peak	2	5.14	CH_2
Multiple peaks	20	6.95-7.93	$4C_6H_5$



Spectra 21: $^{31}P\{^1H\}$ NMR $[C_6H_5(CH_2PPh_3)]_2[Pd_2Cl_4Br_2]$ compound in $CDCl_3$ solvent

Spectra 21



Spectra 22

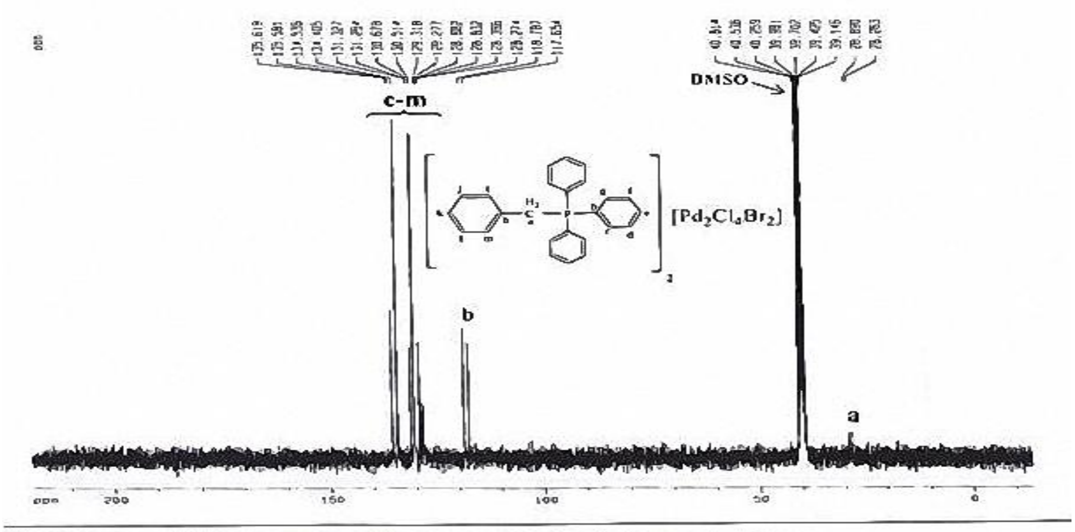
Spectra 23

13C [1H] NMR shows the $[C_6H_5(CH_2PPh_3Br)]_2[pd_2Cl_6Br_2]$ compound

In the spectra, a double peak is related to the carbon on Methyl group which attached to phosphorus at $\delta = 28.57\,ppm$ with 2j$_{p\text{-}C}$47.55 Hz, and existing peaks at $\delta = 39.15 - 40.81\,ppm$ are related to dimethyl sulfoxide, and a double peak at $\delta = 118.22\,ppm$ with 1j$_{p\text{-}}$C=86.04 Hz is related to the carbon of benzene ring and the Phenyl in the compound. Summary of results are presented in the Table 2.

Table 2
CNMR data for $[C_6H_5(CH_2PPh_3Br)]_2[pd_2Cl_6Br_2]$.

Peak shape	$\delta(ppm)$	Carbon type
A double peak	28.57	CH_2P
A double peak	118.22	P-C
Multiple peaks	128.27-135.62	Aromatic rings

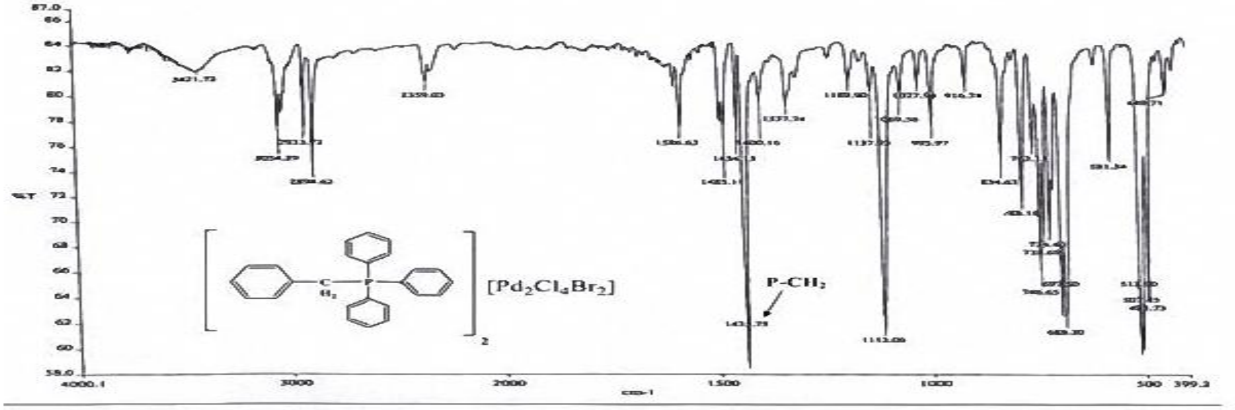


Spectra 23: $^{13}C\{^1H\}$ NMR $[C_6H_5(CH_2PPh_3)]_2[Pd_2Cl_6Br_2]$ compound in DMSo-d^6 solvent

Spectra 23

24 Spectra: IR shows the $[C_6H_5(CH_2PPh_3Br)]_2[pd_2Cl_6Br_2]$ compound

In the spectra, absorption of C-H bending vibration in single substituent ring are observed in 600-900 cm^{-1}, as well as, absorption in the region of 3054 cm^{-1} is related to C-H stretching vibration in phenyl rings. Absorptions in 1454-1600 cm^{-1} are related to $C = C$ vibrations in phenyl rings. The strong absorption at 1436 cm^{-1} is related to p-ch$_2$ bond. And the absorption of phosphorus bond with the carbon in phenyl can be seen in 995.97 cm^{-1}.



Spectra 24: (KBr) $[C_6H_5(CH_2PPh_3)]_2[Pd_2Cl_6Br_2]$ compound

24 Spectra

The results of elemental analysis of this compound based on $C_{50}H_{44}CL_4Br_2p_2pd_2$ empirical formulas is given in Table below.

$C_{50}H_{44}CL_4Br_2p_2pd_2$	%C	%H	%N
Experimental	51.20	4.00	-
Theoretical	51.16	3.97	-

4. Conclusion

In this study, synthesis of Palladium complexes using phosphonium salts, it's numerous applications, including as a catalyst in the Heck reaction, Suzuki reaction and Hydrogenation of carbon, isomer of terminal olefins and switching the *** group in vinyl ethers in organic chemistry, has been investigated.

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