

Salahaddin University
College of Education
Chemistry Department



Recent review of some metal complexes with tertiary phosphine ligands

Research Project

Submitted to the department of in (Chemistry) partial fulfillment of the requirements for the degree of **BSc.** in (Chemistry)

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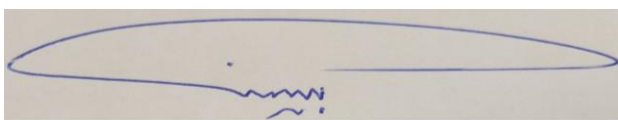
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2023-2024

Supervisor recommendation

I am the student's supervisor, **Lana Masood Abdulkreem** . I support that the student has completed all the requirements for submitting the research drawn entitled (Recent review of some metal complexes with tertiary phosphine ligands) according to the numbered administrative order 3/1/5/1972 on 9th oct. 2022 in accordance with the instructions of Salahaddin university quality assurance and it is ready for discussion.



Lec. Beriwan Muhamad Hamad Ameen

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Research project lecturer

Dr. Dler D. Kurda

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THANKS AGAIN TO ALL WHO SUPPORTED

Abstract

My research is about the study of recent review of some metal complexes with tertiary phosphine ligands. Phosphine ligands have been intensively used in coordination chemistry because of their electron releasing capacity. Phosphine ligands usually attached to the metal center through the pair of valence electrons on the phosphorus. In addition, my research consists of some metal complexes of tertiarydiphosphines. Also in my research, show some structure of diphosphines. Another part of my research is about literature review of some metal complexes of tertiary diphosphines ligand.

Keyword: tertiary diphosphines, metal complexes.

1. Introduction

1.1 Coordination chemistry

Is the branch of inorganic chemistry that deals with the study of coordination compounds is called coordination chemistry (Satya *et al.*, 2002). Coordination compound or complex is formed when a ligand attached to an acceptor by mean of alone-pair of electron , coordination compound contains complex ion or molecule (Singh et al., 2011). In coordination complex a central atom or ion is coordination by one or more molecules or ion (ligand) which act as lewis base forming (coordination bond) with the central atom or ion .the latter act as a lewis acid atom in the ligand that are directly bonded to the central atom or ion are donor atom. The simple yet distinctive concept of the coordinate bond (also sometimes called a dative bond) lies at the core of coordination chemistry. Molecular structure, in its simplest sense, is interpreted in terms of covalent bonds formed through shared pairs of electrons. The coordinate bond, however, arises not through the sharing of electrons, one from each of two partner atoms, as occurs in a standard covalent bond, but from the donation of a pair of electrons from an orbital on one atom (a lone pair) to occupy an empty orbital on what will become its partner atom (Lawrance, 2013). The chemistry of the transition elements is very largely concerned with the chemistry of coordination compounds. Coordination compounds or, as they are perhaps more usually called, complexes play a very important part in our lives today; the study of them has contributed greatly to our understanding of the chemical bond and of inorganic chemistry as a whole. The number of possible coordination compounds in chemistry is almost infinite. Present-day research workers prepare many new complexes every week; most of these will be of academic interest only, but just a few will be of considerable economic importance as well. For many years

complexes were regarded as of interest to the theoretical and inorganic chemist only, but now they are playing vital roles in analytical chemistry, in the synthesis of organic chemicals, in polymerization processes, and in our understanding of biological processes. Perhaps the most important are the naturally occurring complexes such as chlorophyll, the magnesium complex that is important in plant photosynthesis, and hemoglobin, the iron complex that is a carrier of oxygen in blood (Nicholls, 1974).

1.2 Transition Metal

The transition metals include the three series of elements that are positioned between the first two groups and the last six groups in the periodic table. These series have as their general characteristic that a set of *d* orbitals is being filled in progressing from one element to the next. The first series, usually referred to as the first-row transition metals, involves filling the *3d* orbitals (Gelatt Jr et al., 1983). The second- and third-row transition metals correspond to those in which the *4d* and *5d* orbitals are being filled. Because a set of *d* orbitals can hold a maximum of 10 electrons, there are 10 elements in each series. The groups containing the transition metals are sometimes designated as the — B groups or as groups 3 through 10. In the latter case, the groups are usually denoted as 1 through 18 in going across the periodic table. The elements of the first transition series are those for which the *3d* electron shell contains between one and nine electrons. Copper is included because, although its outer electronic configuration is $3d^{10} 4s^1$, it has the $3d^9$ configuration in its commonly occurring +2 oxidation state. Zinc is not normally considered a transition element since in both the element and its compounds the *3d* electron shell remains filled. It therefore does not show the characteristic properties of colored compounds and Para magnetism shown by the other elements in at

least one of their oxidation states. Scandium is included by the definition, but so far only the +3 oxidation state has been established with certainty. Its compounds are thus diamagnetic with no color from d—d transitions, and its chemistry thus resembles that of aluminum rather than that of the other transition elements (Nicholls, 1974).

1.3 Tertiary Phosphine Ligands

Phosphine ligands have been intensively used in coordination chemistry because of their electron releasing capacity (ALI et al., 2019) The phosphine ligands can be organized based on the type of substituents bonded to the phosphorus atom (Lagasse et al., 2000). Bisphosphines are class of bidentate ligand that contain two phosphine atoms joined by a bridge (ALI et al., 2019). Phosphines ligands usually attached to the metal center through the pair of valence electrons on the phosphorus (Kraaijkamp et al., 1983). Phosphine ligands are usually strong σ -donor ligands and only weak π -acceptors (Scharf and Gessner, 2017). This impact can be expanded with electron-donating groups in the rest R, although electron with-drawing groups in 'R' favor the π -acceptor back bonding (Daura-Oller et al., 2003). Phosphines are ubiquitous ligands, with a wealth of history and rich metal coordination chemistry. Much of modern day homogeneous transition metal catalysis is underpinned by phosphine ligands and metal–phosphine coordination (Orton et al., 2021). So common are phosphine ligands in transition metal catalysts that their use has become second nature to many researchers in the field. Chemists have long known that the reactivity and behaviour of phosphines is governed by their substituents. For good catalytic activity in cross-coupling reactions, phosphines typically need to be both basic (electron-rich) and sufficiently sterically encumbered (as defined by a cone angle of the substituents around

the phosphorus atom) (Surry, 2011). The ability to tune both the electronic and steric properties of phosphine ligands lies behind their importance in the development of effective homogeneous catalysts (Barber et al., 2020). Some typical phosphine ligands are shown in Fig. (1.1).

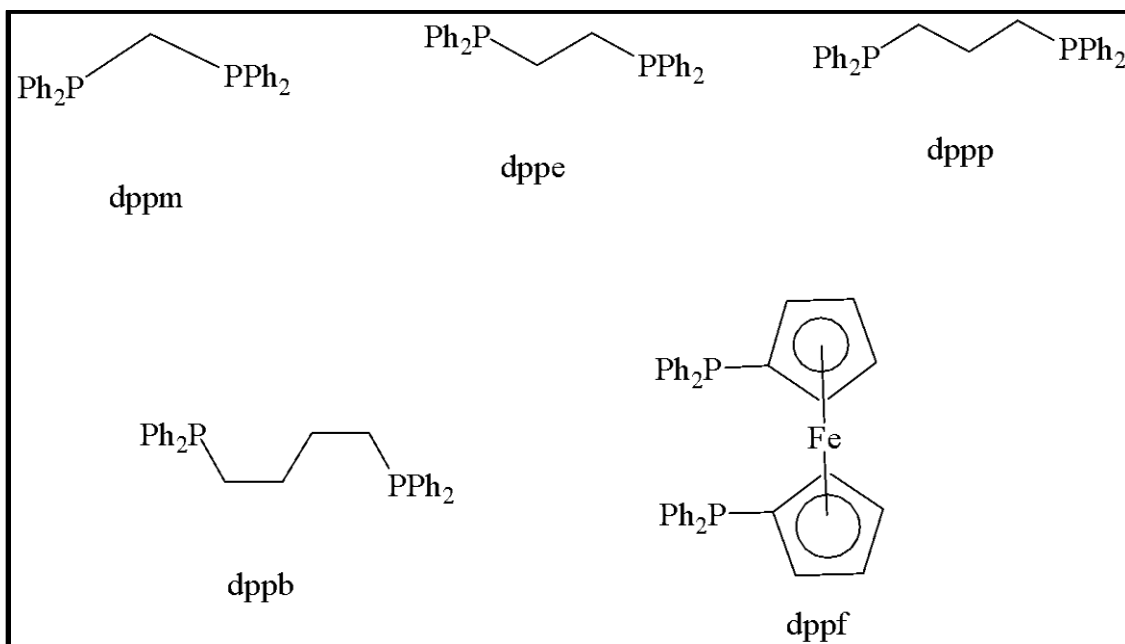


Fig. (1.1): Some typical phosphine ligands

1.4 Complexes of tertiary diphosphine

Tertiary phosphines are amongst the most commonly encountered ligands in transition metal complexes. There are examples of complexes of PR₃ ligands with virtually every transition metal (Berners-Price and Sadler, 2005). Phosphine ligands which were commonly powerful σ -donor ligands and only feeble π -acceptors. Those solitary combine of the phosphorus is subject to the σ – donation towards palaver orbitals. The back- bonding donation take place starting filled metal orbitals to palaver orbitals of the ligands; this indicates

that back-donation arises the d-orbitals of metal into the σ^* - orbitals to the phosphorus substance (Cevik, 2017)

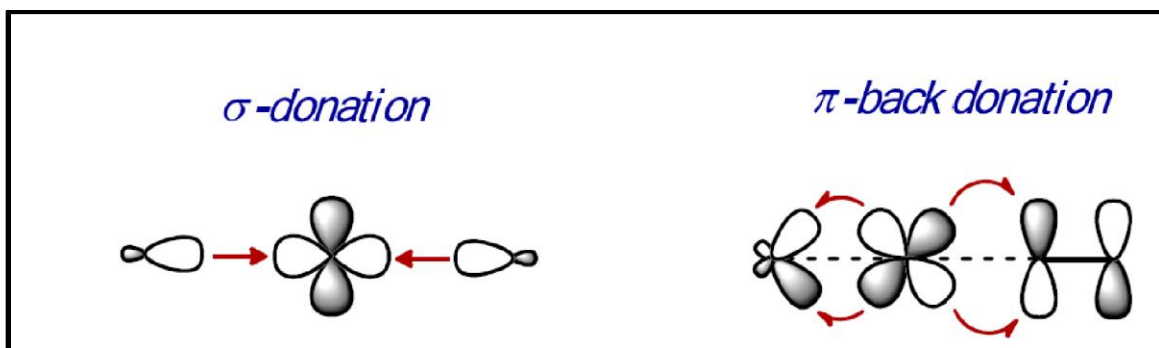


Fig. (1.2): σ -giver and π -back contribution interactions in phosphine ligands. On the left side, strong σ – contribution takes place, and on the right side a weak π –back-donation to the M–L bond

On the different side, phosphines What's more phosphine metal bringing complexes compound were already vivaciously mulled over outstanding to their far reaching employments Concerning illustration antitumor Also mitigating medications (Shaheen et al., 2008a). The π -acceptor facility of phosphine ligands is associated to the electronegativity of a side chain on atoms of phosphorus; the high electronegative the substituents are enhanced the π -acceptor facility of the phosphine (Adams, 1998).

Complexes with bis-tertiary phosphine ligands are poorly investigated in the literature (Lobana et al., 2000). Tertiary phosphine complexes of silver(I) of the type $[\text{AgXL}_n]$ (L = tertiary phosphine, PR_3 (R = alkyl, aryl or H); $n = 1-4$; X = coordinating or non-coordinating anion) were first prepared in 1937 by Mann et al (Meijboom et al., 2009). Previous periodic reviews have reported on the coordination chemistry of silver (Cortez and Raptis, 1997) and Polymers (Khlobystov et al., 2001) containing nitrogen-donor ligands. In addition, the chemistry of silver scorpionates (Pettinari et al., 2004) and silver

carboxylates (Grodzicki et al., 2005) The versatile diphosphine ligands ‘dppx’ are particularly appropriate for the synthesis of low-valent metal complexes. Bis(diphenylphosphino)methane (dppm) and 1,2- bis(diphenylphosphino) ethane (dppe) are widely used in inorganic chemistry, dppm affording dinuclear metal complexes in μ -dppm form, while dppe affords both chelate and bridged complexes. Complexes containing the $M_2(dppm)_2$ structural unit have been intensively investigated. Although a key feature of the chemistry of this class of complexes is the relatively stable and quasi-planar M_2P_4 framework where two metal atoms are connected by a pair of bridging dppm ligands, it is well known that a variety of structural possibilities can occur when low-valent metals are bonded to a bidentate phosphine and a labile ligand such as nitrate or carboxylate (Meijboom et al., 2009).

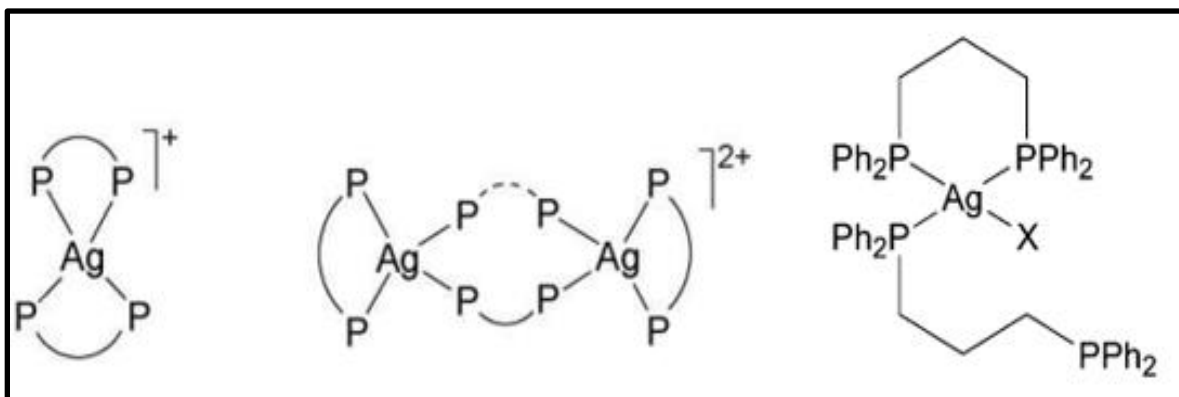


Fig. (1.3): The structures of silver complexes with some tertiary diphosphines

2. Literature review for complexes of tertiary diphosphines

In 2004, Amin O. H. et al. synthesized The complexes $[ML_2A_2]$ or $[ML_2B]$ where $M = Pd$ or Pt , $L = 5$ -phenyl-1,3,4-oxadiazole-2-thione ion, $A =$ tertiary monophos-phines and $B =$ tertiary diphosphines have been used effectively to prepare bimetallic complexes of the type $[A_2M(\mu-L)_2M Cl_2]$ or $[BM(\mu-L)_2M Cl_2]$, where $M = Co, Pd$ or $SnCl_2$. The prepared complexes were characterized by elemental analysis, magnetic susceptibility, IR and UV–Vis spectral data. (Amin et al., 2004)

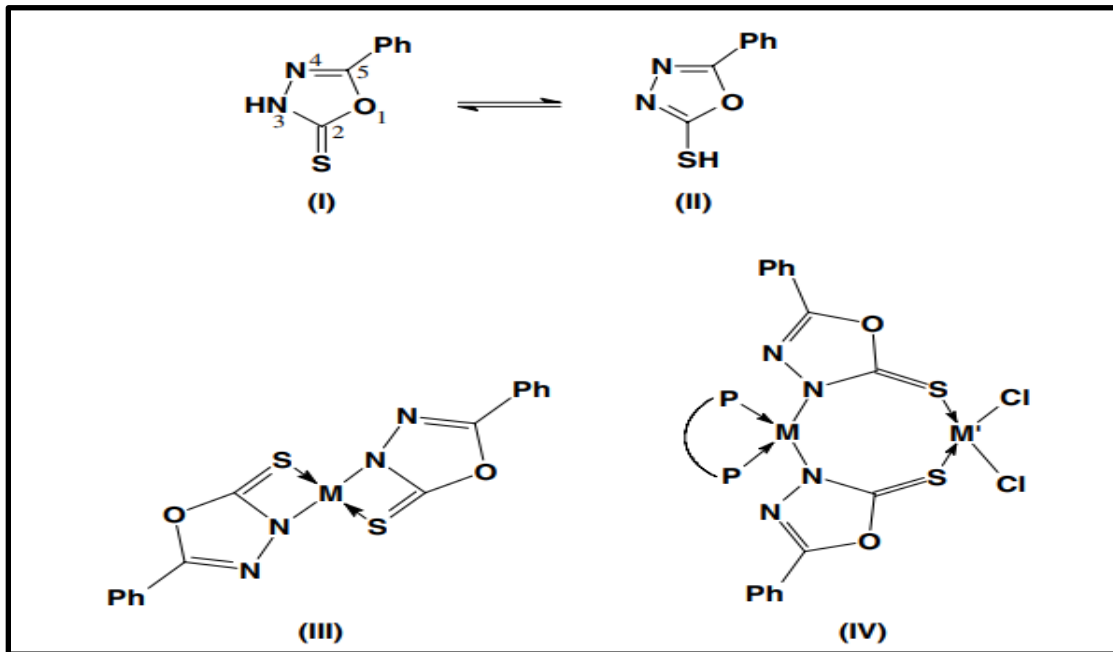


Fig. (2.1): The ligand 5-phenyl-1,3,4-oxadiazole-2-thione (LH), its tautomeric forms (I) and (II) and the suggested structure of its bis(chelated) complexes (III) and the bridging bimetallic complexes (IV)

In 2006, Asma M. coworkers synthesized A number of palladium(II) complexes with Ph₃P and aromatic amines as ligands having the general formula [(Ph₃P)Pd(Ph₃P)(NH₂R)Cl₂] {where R = Ph (1), m-ClC₆H₄ (2), p-ClC₆H₅ (3)} and [Pd(CH₃NHPh)Cl₂] (4), have been synthesized and characterized by elemental analysis, IR, ¹H and ¹³C-NMR. The X-ray crystal structure of [(Ph₃P)Pd(m-ClC₆H₄NH₂)Cl₂]. CH₂ Cl₂ (2) shows a distorted square planar environment around the Pd(II) ion with the P-Pd-N and Cl(1)-Pd-Cl(2) bond angles of 174.88(5) and 176.77(2) respectively. The complexes were screened for enzyme inhibition activity against glucuronides and found to be active (Asma et al., 2006)

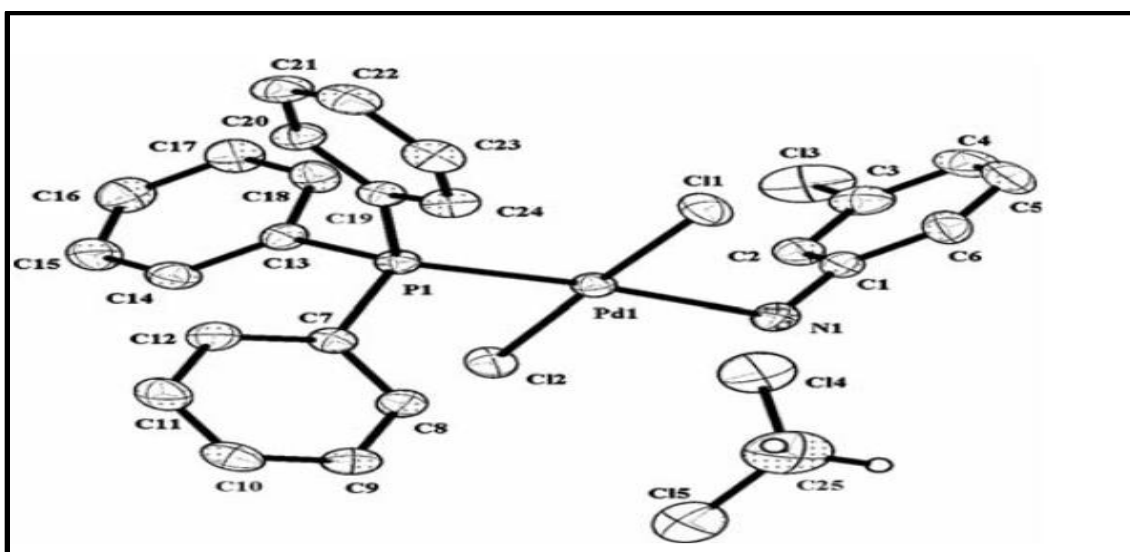


Fig. (2.2): Crystal structure of compound (2), showing the atom-numbering scheme. The thermal ellipsoids are drawn at the 30 % probability level.

In 2008, Shaheen F. et al. synthesized The new four-coordinated mononuclear palladium(II) complexes with chelating heterocyclic thiolates and tertiary phosphines with general formula $[Pd(L)_nCl(R'R_2P)]$ ($L = R_3P = PPh_3$, $n = 1, 2$). These compounds were characterized by elemental analysis, FT-IR and multinuclear (1H , ^{13}C and ^{31}P) NMR. The X-ray diffraction study of non-ionic compound 5 showed that the thiolate acts as unidentate and that the chelating ($-N,S$) ligand adopts a slightly distorted square planar geometry around the palladium atom (Shaheen et al., 2008b)

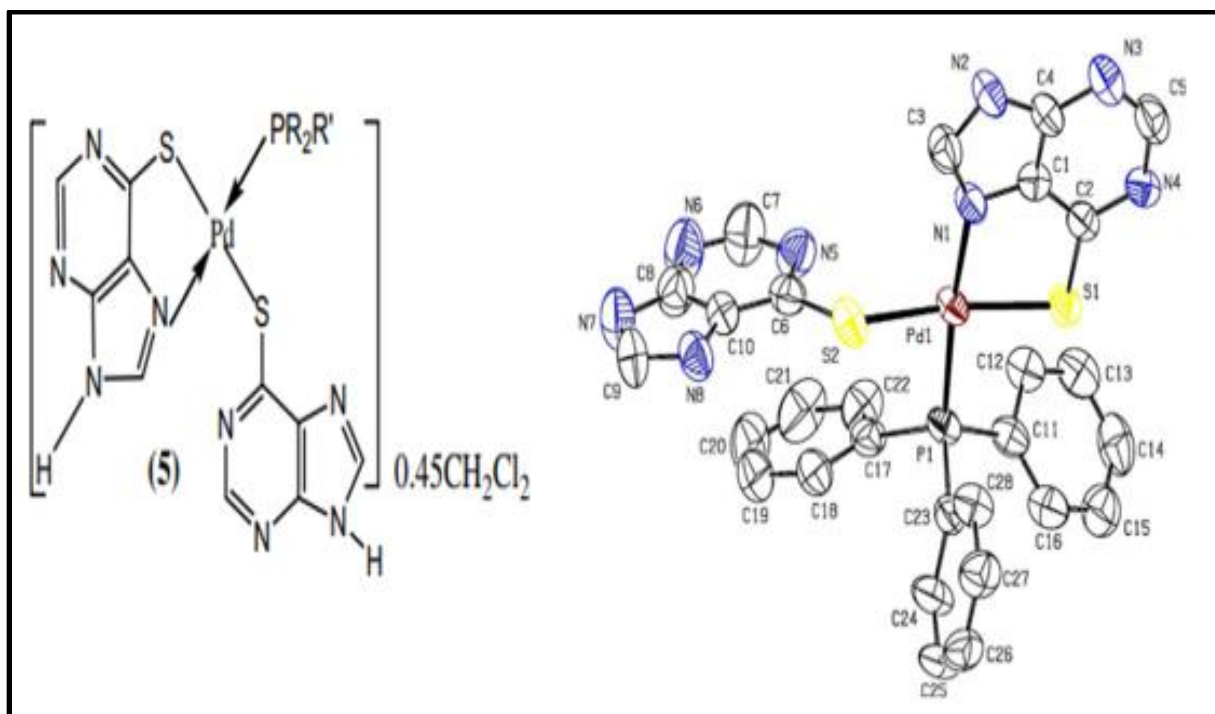


Fig. (2.3): ORTEP diagram of $[Pd(L)_nCl(R'R_2P)]$ with atomic numbering scheme, $R = \text{phenyl}$, $R' = \text{phenyl, o-tolyl}$

In 2012, Miranda S. et al. synthesized of the $[MCl_2(P-P)]$ ($M = Pd$ and Pt ; $P-P = dppe$ and $dppm$) reacts with $NaC_4H_3SN_2$, in a 1:2 molar ratio at room temperature, to give $[M(S-C_4H_3SN_2)_2(dppe)]$ [$M = Pd$ (1); $M = Pt$, (2)], $[Pd(S-C_4H_3SN_2)_2(dppm)_2]$ (3) and $trans-[Pt(S-C_4H_3SN_2)_2(dppm)]$ (5), as well as other by-products such as $[Pd_2(S,N-C_4H_3SN_2)_4]$ (4) and $trans-[Pt(S-C_4H_3SN_2)_2(PPh_2Me)_2]$ (6). mechanism for the formation of 6, which requires cleavage of P–C bonds of the $dppm$ ligands, is proposed (Miranda et al., 2012).

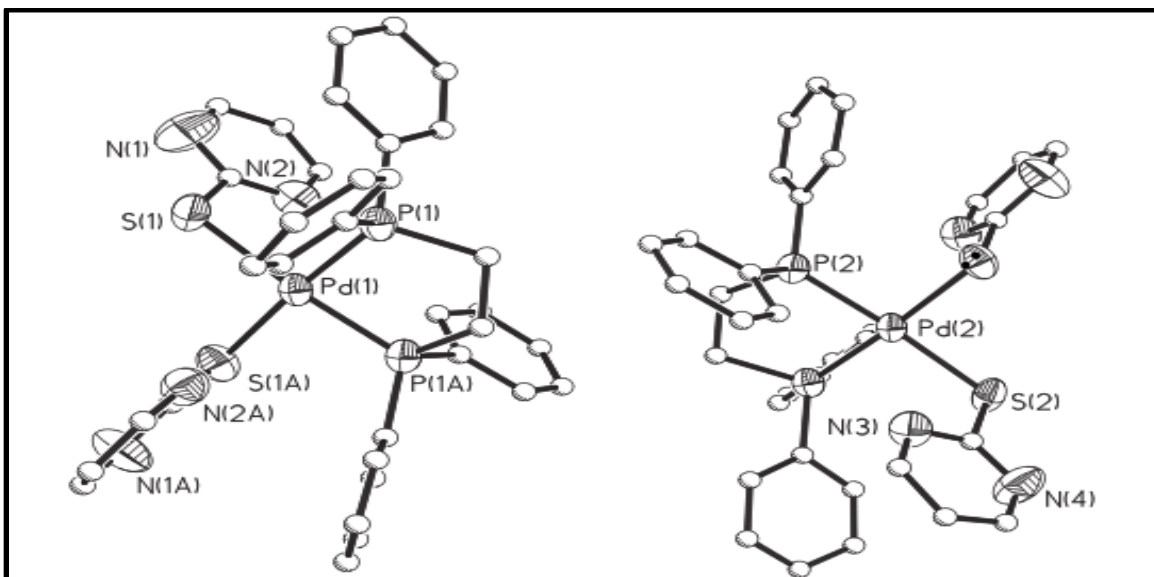


Fig. (2.5): Structure of the two independent molecules in complex 1.

Thermal ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

In 2020, Al-Jibori S. A. et al synthesized Benzotriazole (Hbta) complexes with Hg(II) and Pd(II) of the types: $[\text{Hg}(\text{bta})_2]$ (1), $[\text{Hg}(\text{bta})_2(\text{diphos})]$; diphos= dppe (2), dppp (3) or dppb (4), $[\text{Hg}(\text{bta})_2(\text{PPh}_3)_2]$ (5), $[\text{Pd}(\text{Hbta})_2\text{Cl}_2]$ (6), $[\text{Pd}(\text{bta})\text{Cl}]_2$ (7) and $[\text{PdCl}(\text{bta})(\text{PPh}_3)]_2$. DMSO (8) were prepared and characterized by elemental analyses, conductivity measurements, infrared, ^1H - and ^{31}P - $\{^1\text{H}\}$ NMR spectra. $[\text{Pd}(\text{PPh}_3)(\mu\text{-bta})\text{Cl}]_2$. DMSO (8) was structurally characterized by single-crystal X-ray diffraction. Structural information exposed that benzotriazole acts as a bidentate bridging ligand bonded through the deprotonated nitrogen and the neutral nitrogen atoms (Al-Jibori et al., 2020).

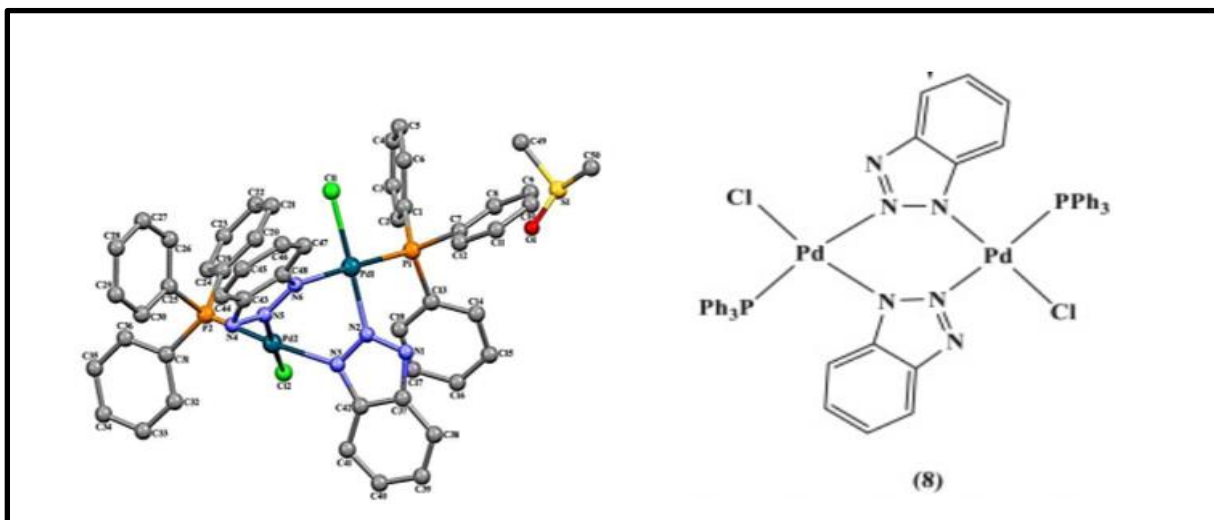


Fig. (2.6): Molecular structure of $[\text{Pd}(\text{PPh}_3)(\mu\text{-bta})\text{Cl}]_2$.DMSO (8)

In 2020, Mohamad H. A. and coworkers prepared the complex with chemical formula: $[\text{Hg}(\text{L})_2(\text{PPh}_3)_2]$. The complex was synthesized by reaction of 5-phenyl-1,3,4-oxadiazole-2-thiol with mercury(II) acetate in ethanol led to the isolation of the complex $[\text{Hg}(\text{L})_2]$. Further reaction of $[\text{Hg}(\text{L})_2]$ with two molar excess of triphenylphosphine led to the isolation of $[\text{Hg}(\text{L})_2(\text{PPh}_3)_2]$. The complex was characterized by infrared, ultraviolet– visible and nuclear magnetic resonance spectroscopy techniques, as well as by X-ray diffraction analysis (Mohamad et al., 2020).

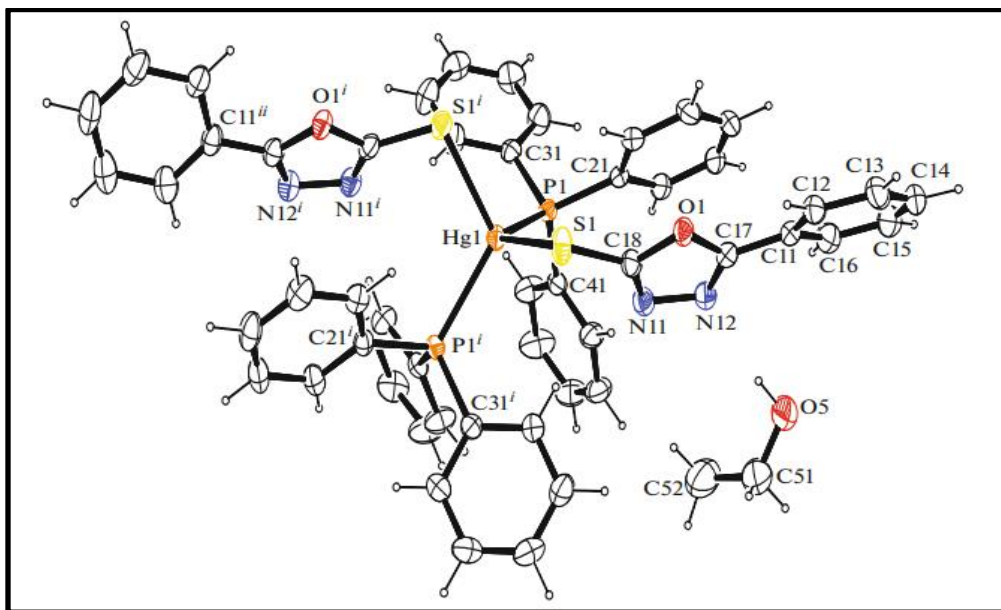


Fig. (2.7): Molecular structure of $[\text{Hg}(\text{L})_2(\text{PPh}_3)_2] \cdot 2\text{EtOH}$.

In 2021, Mohamad H. A. et al synthesized the Two new complexes of copper(I) and copper(II) with phenanthridine (Phend) of the following composition: $[\text{Cu}(\kappa^1\text{-Phend})_2\text{Cl}_2]$ (1) and $[\text{Cu}_2(\kappa^1\text{-Phend})_2(\kappa^1\text{-PPh}_3)_2(\mu\text{-Cl})_2]$ (2) have been prepared in the presence of triphenylphosphine (PPh_3) as a co-ligand. The structures of these complexes have been investigated by elemental analysis, molar conductivity, FT - IR, UV -Vis, and single -crystal X - ray diffraction. X - ray diffraction analysis of typical complex 1 reveals the distorted square planar geometry around copper(II) whereas the binuclear complex 2 was irregular tetra- hedral geometry around Cu(I) center containing bridge Cl- ion (Mohamad et al., 2021).

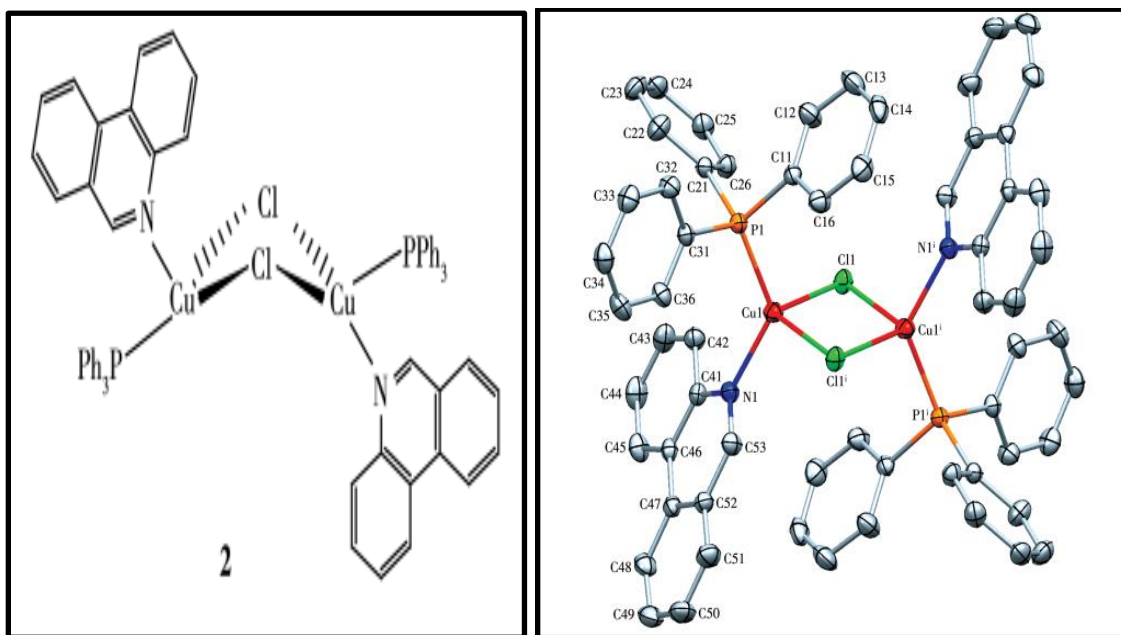


Fig. (2.8): Molecular structure of complex $[\text{Cu}_2(\kappa^1\text{-Phend})_2(\kappa^1\text{-PPh}_3)_2(\mu\text{-Cl})_2]$

In 2022, Al-Mouqdady O. D. H. and coworkers synthesized complexes, $[\text{Pd}(\text{mtzS})_2(\text{Ph}_2\text{P}(\text{CH}_2)_n\text{PPh}_2)]$ (3–5), $[\text{Pd}(\text{mtzS})_2(\text{dppf})]$ (6), $[\text{Pd}(\text{mtzS})_2(\text{Ph}_3\text{P})_2]$ (7) or $[\text{Pd}(\text{mtzS})_2(\text{Ph}_3\text{PS})_2]$ (8). An X-ray structure of $[\text{Pd}(\text{mtzS})_2(\text{dppf})]$ (6) shows that the mtzS ligand is now coordinated in a monodentate fashion via the sulfur atom. The cytotoxicity of the prepared complexes were evaluated against colon mammalian cancer (LoVo) and breast cancer (MCF-7) cell lines by MTT assay. (Al-Mouqdady et al., 2022).

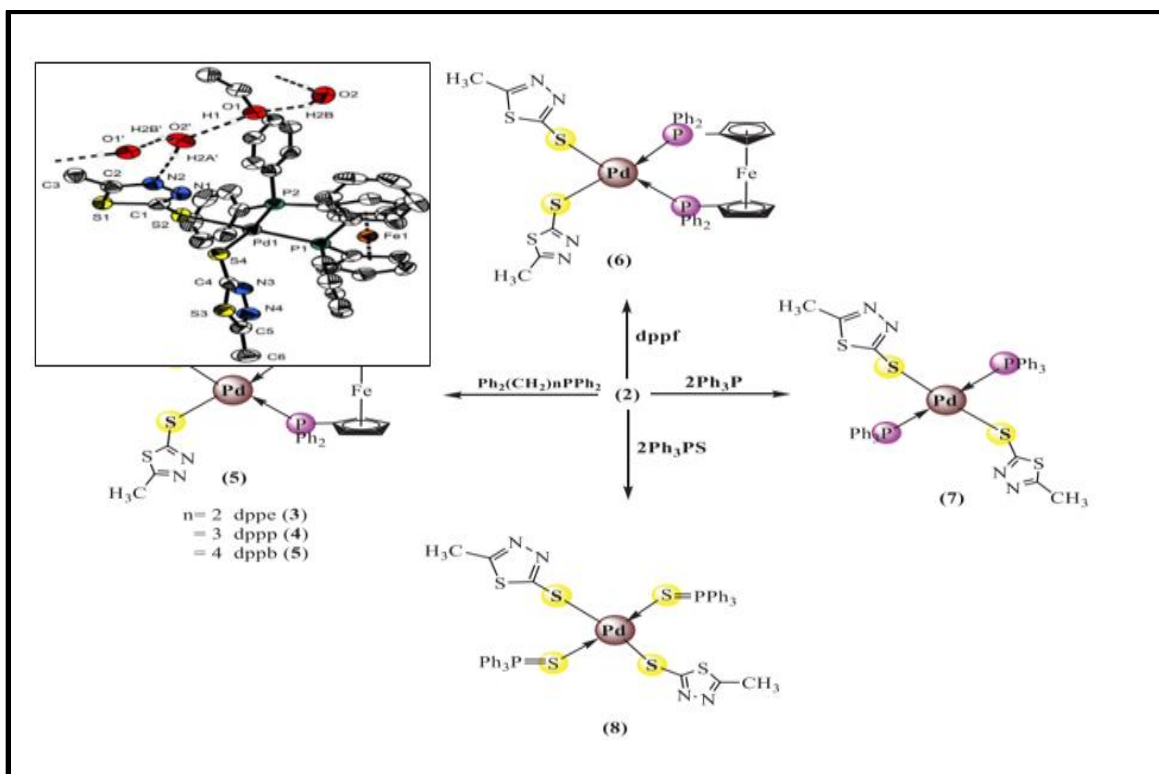


Fig. (2.9): Preparation of complexes (3–8) and Molecular structure of $[\text{Pd}(\text{mtzS})_2(\text{dppf})]\cdot\text{H}_2\text{O}\cdot\text{CH}_3\text{CH}_2\text{OH}$ with the hydrogen bridge network. Thermal ellipsoids are drawn at the 50% probability level. Hydrogen atoms

In 2022, Mohamad H. A. et al. synthesized A novel distorted square-planar palladium(II) complex of the type $[\text{Pd}(\text{Hdz})(\text{PPh}_3)\text{Cl}]$, where (Hdz = dithizone mono deprotonate and PPh_3 = triphenylphosphine), was synthesized in dichloromethane reactions between PdCl_2 and a mixture of Hdz and PPh_3 . The new Pd(II) complex has been identified by FT-IR, electronic spectra, molar conductivity, and single-crystal X-ray diffraction. An X-ray diffraction study revealed the structure of this complex, indicating distorted square planar coordination geometry around the Pd(II) ion by N, S, P, and Cl donor atoms (Mohamad et al., 2022).

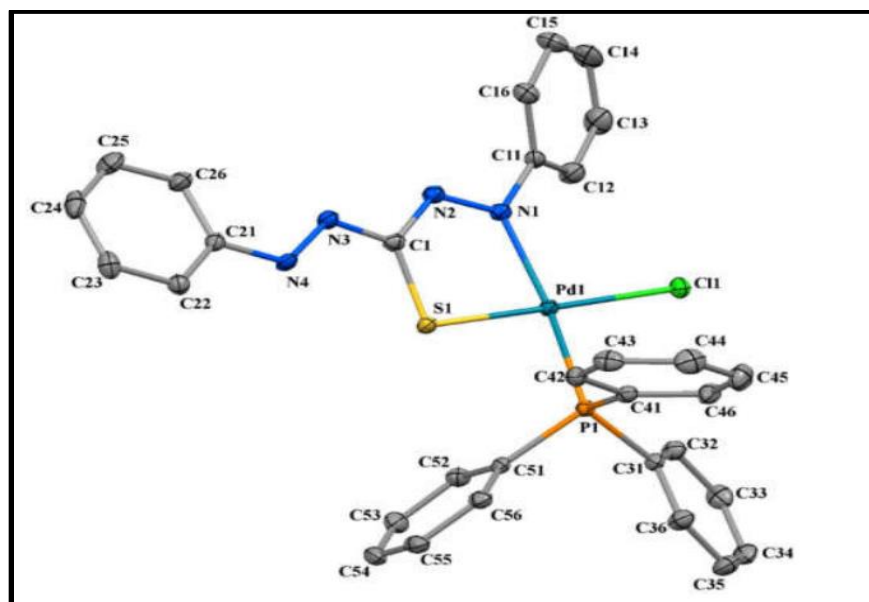


Fig. (2.10): The synthesis method of the target complex and Crystal structure of $[\text{Pd}(\text{HDZ})(\text{PPh}_3)\text{Cl}]$ shown at 30% ellipsoid probability, H atoms are omitted for clarity.

In 2023, Mohamed D. S. et al. synthesized complexes of $[\text{PtCl}_2(\text{L-L})]$ ($\text{L-L} = \text{dppe}$, dppp , dppb , dppf , Phen and Bipy) or $[\text{PtCl}_2(\text{PPh}_3)_2]$ with 1-benzyl-3-phenylthiourea (H_2BPT) in a basic medium ($\text{CHCl}_3/\text{EtOH}$) created new coordinated square planar $\text{Pt}(\text{II})$ complexes with $[\text{Pt}(\text{BPT})(\text{L-L})]$ (1–4). These complexes were fully characterized by analytical and spectroscopic techniques (i.e., IR, UV. Vis., ^1H , and ^{31}P NMR). The results indicated that the thiourea derivative ligand act as a dianion ligand bonded through both S and N atoms in a chelating mode or as a mono-anion ligand coordinated through a sulfur atom with $\text{Pt}(\text{II})$ ion. Cytotoxicity activity was performed by the MTT assay to determine anti-cancer activities against MCF-7 breast cancer cells (Mohamed et al., 2023).

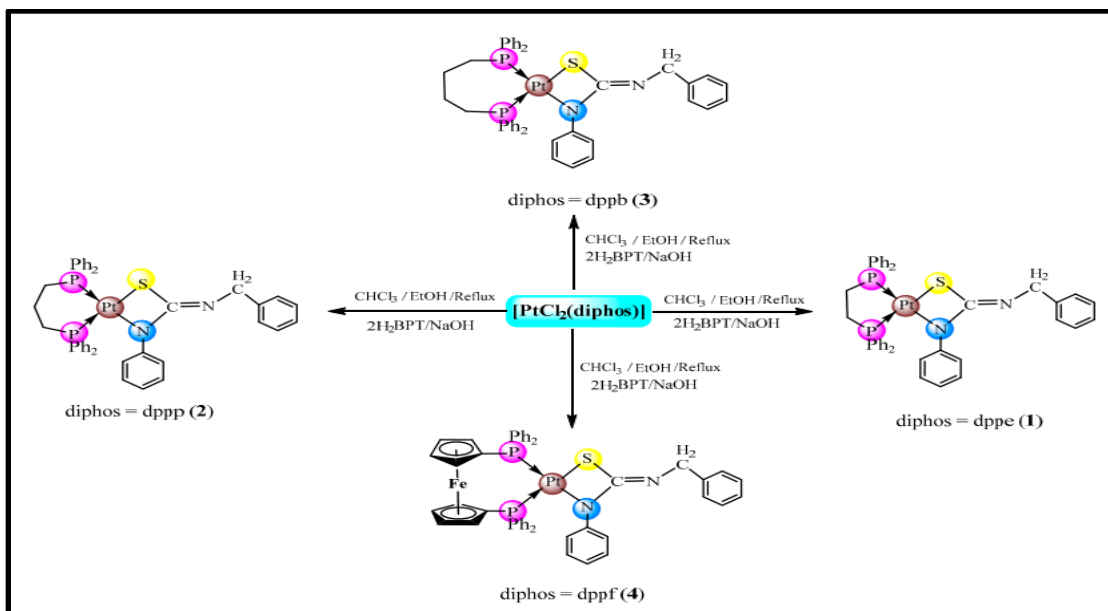


Fig.(2.11): Preparation of the complexes (1-4).

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