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Synthesis of New Phosphine-Containing Hg Complexes Derived from 2-Thiouracil and Evaluation of Their Biological Activity

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Abstract: *The study involved the formation of several new mercury complexes by reacting 2-thiouracil with mercury chloride and sodium hydroxide to yield the mercury complex L1. This reaction was considered the basis for preparing other complexes, as it reacts with two moles of triphenylphosphine (PPh3) to form complex L2, and through the reaction of complex L1 with bis(diphenylphosphanyl)methane (dppm) to create complex L3, and with 1,2-bis(diphenylphosphanyl)ethane (dppe) to form complex L4, and with 1,3-bis(diphenylphosphanyl)propane (dppp) to form complex L5. The validity of the prepared complexes was confirmed through spectral measurements, including the infrared spectrum and the proton and phosphorus nuclear magnetic resonance spectra. The presence of new bands in the infrared spectrum, such as the (Hg-N) and (Hg-O) bands, provided evidence of a bond. Mercury with thiouracil as between the bands belonging to (P-Ph) and (C-P), proof of the association of phosphine with the metal, and between the 1H-NMR spectrum, there are signals belonging to the benzene ring in phosphine, and this was confirmed by their complements, which were identical to the protons of the benzene rings. Between the 31P-NMR spectrum, there is a single signal indicating that the association is double in phosphine with the presence of one isomer of the complexes. The effectiveness of the prepared complexes was tested on two types of bacteria, positive and negative, using the antibiotic amoxicillin as a control sample. The confounders showed a direct relationship with the concentration, and the L2 complex showed the highest effectiveness against the two types of bacteria studied.*

Keywords: *Coordination Chemistry, 2-Thioracil, Complex.*

Introduction

Without a question, the most active field of study in inorganic chemistry is coordination chemistry. . Hundreds of coordination complexes have been developed and investigated in recent decades. Since the significance of the coordinating phenomenon in biological processes was recognized, a number of metal-containing macromolecules have been created and investigated in order to comprehend the function of these ligands in biological systems. They also encourage the creation of novel metal-based chemotherapeutic medications[1]. Because metal chelates are more antibacterial than the chelating agents themselves in several instances, bioinorganic chemistry has emerged as a significant area of

inorganic chemistry. Pyrimidine derivatives are substantial due to their applications in biology, medicine, and agriculture [2]. Pyrimidine metal complexes have been the subject of considerable research recently due to their diverse range of biological activities, which include antimalarial, antibacterial, anticancer, and antiviral properties, among others [3,4]. Numerous studies have been conducted on the usage of uracil, thiouracil, and pyrimidine thiones in the biological process of supplying bonding sites for metal ions [5]. Pyrimidine 2-thione (pymt) and its derivatives are essential antitumor and antithyroid drugs [6], and they are found in soluble ribonucleic acid [7]. The molecular structures of 4-thiouracil, 2-thiocytosine, and pyrimidine thiones (pymt) are closely related to those of 4-dimethyl Pyrimidine-2-thione (dimpymt), which, like thiobases, exhibit anticancer properties and limit RNA creation under certain conditions [8]. Because of its significance to the necessary, therapeutic, or harmful bioactivity of metal, it is exciting to see how metal ions interact with nucleobases. In metalloproteins, nucleobase molecules can coordinate as foreign ligands and serve as cofactors in the enzymatic systems [9].

Methodology

Chemicals and Instruments

Sigma-Aldrich, BDH, Fluka, and Merck supplied high-purity materials and solvents. Melting points were measured using a Cole-Paramer MP-200D-120 Stuart digital melting point spectrometer. A SHIMADZU FT-IR-8400S was used for FT-IR spectra. A Varian-INOVAUSA at 500 MHz was used for ^{1}H-NMR and ^{31}P-NMR spectroscopy (400 and 162 MHz) using DMSO-D_6 as the solvent and TMS as the internal standard at the University of Basrah.

Preparation of Ligand (L1)[10]:

In 10 millilitres of 100% ethanol, a solution of 2-thiouracil (1 mol 1 g) was mixed with a solution of NaOH (1 mol 0.312 g). At room temperature, the mixture was agitated for an hour. Next, 10 millilitres of 100% ethanol were added to a solution of HgCl (0.5 mol, 1.069 g). At room temperature, the mixture was agitated for three hours, during which a pale brown precipitate formed. From a combination of EtOH and DMF solvents, the resultant material was filtered, cleaned with ethanol, vacuum-dried, and recrystallised.

$[Hg(L)_2]$

Brown yield: 74%; mp = 187–189 °C; IR (KBr) v(cm^{-1})= 3288(NH), 3138(=CH), 3064(Ar-CH),1660 (C=O), 1599 (C=C), 1577,1508 (Ar-C=C), 1084 (C=S), 489 (Hg-O), 424(Hg-N) cm^{-1}; ^{1}H-NMR (DMSO-d^6, 400 MHz): δ(ppm) = 7.45-7.43 (2H, d, =CH), 6.34, 6.32 (2H, d, HC=), 7.16 (2H, s, NH)

Preparation of Complex (L2)[11]:

In 10 milliliters of 100% ethanol, a solution of $2PPh_3$ (2 mol, 0.2 g) was mixed with a solution of [L1] (1 mol, 0.115 g). A pale brown precipitate appeared when the liquid was agitated for three hours at room temperature. The precipitate was filtered and vacuum-dried in an oven (0.700 g, 76%) .

$[Hg(L)_2(pph3)_2]$

brown *yield: 76%; mp = 231–233 °C; IR (KBr) ν(cm⁻¹)= 3279(NH), 3171(=CH), 3051(Ar-CH),1662 (C=O), 1639 (C=C), 1500 (Ar-C=C), 1437 (P-Ph),1097 (C=S), 1066 (C-P),499 (Hg-O), 447(Hg-N) cm⁻¹; ¹H-NMR (DMSO-d⁶, 400 MHz): δ(ppm) = 7.53,7.52 (2H, d, =CH), 7.51-7.36 (30H, m, ph), 5.79, 5.77 (2H, d, HC=), 7.11 (2H, s, NH); ³¹P-NMR (DMSO-d⁶, 162 MHz): δ(ppm) = 25.68 (P-ph3)*

Preparation of Complex (L3)[12]:

In 20 milliliters of 100% ethanol, a solution of dppm (1 mol, 0.1 g) was mixed with a solution of [L1] (1 mol, 0.158 g). After three hours of stirring at room temperature, a white precipitate developed. After filtering, the precipitate was vacuum-dried in an oven (0.700 g, 73%).

[Hg(L)$_2$(dppm)]

White yield: 73%; mp = 256–258 °C; IR (KBr) ν(cm⁻¹)= 3292(NH), 3169(=CH), 3049(Ar-CH), 2904, 2870 ($CH_{Aliphatic}$), 1664 (C=O), 1612 (C=C), 1498 (Ar-C=C), 1435 (P-Ph),1095 (C=S), 1060 (C-P),482 (Hg-O), 449(Hg-N) cm⁻¹; ¹H-NMR (DMSO-d⁶, 400 MHz): δ(ppm) = 7.38,7. 37(2H, d, =CH), 7.52-7. 29(20H, m, ph), 5.79, 5.7 8(2H, d, HC=), 7. 26(2H, s, NH), 1.83 (2H, s, CH_2); ³¹P-NMR (DMSO-d⁶, 162 MHz): δ(ppm) = 36. 01(P)

Preparation of Complex (L4)[12]:

In 20 milliliters of 100% ethanol, a solution of dppe (1 mol, 0.1 g) was mixed with a solution of [L1] (1 mol, 0.158 g). After three hours of stirring at room temperature, a white precipitate developed. After filtering, the precipitate was vacuum-dried in an oven (0.700 g, 73%).

L4=[Hg(L)$_2$(dppe)]

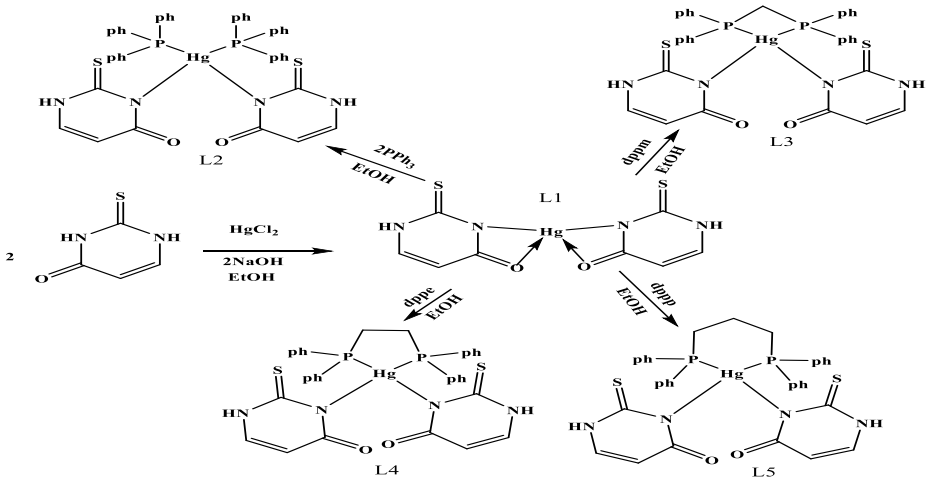
White yield: 73%; mp = 268–270 °C; IR (KBr) ν(cm⁻¹)= 3284(NH), 3173(=CH), 3066(Ar-CH), 2924, 2860 ($CH_{Aliphatic}$), 1660(C=O), 1610(C=C), 1498 (Ar-C=C), 1433 (P-Ph),1099 (C=S), 1066 (C-P),476 (Hg-O), 445(Hg-N) cm⁻¹; ¹H-NMR (DMSO-d⁶, 400 MHz): δ(ppm) = 7.43-7.35 (20H, m, ph), 7.33,7.32 (2H, d, =CH), 5.60, 5.59 (2H, d, HC=), 7.01 (2H, s, NH), 2.23-2.21 (4H, t, CH_2-CH_2); ³¹P-NMR (DMSO-d⁶, 162 MHz): δ(ppm) = 30.58 (P)

Preparation of Complex (L6)[12]:

In 10 milliliters of 100% ethanol, a solution of dppp (1 mol, 0.1 g)was mixed with a solution of [L1] (1 mol, 0.158 g). After three hours of stirring at room temperature, a white precipitate developed. After filtering, the precipitate was vacuum-dried in an oven (0.700 g, 73%).

L5=[Hg(L)$_2$(dppp)]

White yield: 73%; mp = 283–285 °C; IR (KBr) ν(cm⁻¹)= 3292 (NH), 3173(=CH), 3053 (Ar-CH), 2912, 2870 ($CH_{Aliphatic}$), 1658 (C=O), 1610(C=C), 1498 (Ar-C=C), 1433 (P-Ph),1101 (C=S), 1064 (C-P),480 (Hg-O), 449 (Hg-N) cm⁻¹; ¹H-NMR (DMSO-d⁶, 400 MHz): δ(ppm) = 7.65-7.34 (20H, m, ph), 7.22,7.20 (2H, d, =CH), 5.66, 5.63 (2H, d, HC=), 7.08 (2H, s, NH), 2.13-2.09 (4H, t, CH_2-P), 1.81-1.68 (2H, q, CH_2); ³¹P-NMR (DMSO-d⁶, 162 MHz): δ(ppm) = 32.88 (P)



Scheme 1: Preparation of complexes (L1-L5)

Biological activity evaluation

Two kinds of bacteria—Staphylococcus aureus and Escherichia coli—were gathered from the labs of Tikrit Central University. Dimethyl sulfoxide (DMSO) solvent was used to create chemical solutions of the produced complexes at three different concentrations of (0.01, 0.001, 0.001) mg/ml for each of these solid derivatives [13,14]. Following its insertion into the tubes holding the diluted bacterial growth, a sterile cotton swab was used to inoculate the Mueller-Hinton agar (MHA) medium. The surplus inoculum was then removed by pressing the swab against the tube's inner walls. The inoculum was then uniformly distributed by wiping the culture media in three different directions. To absorb the culture and dry the medium, the plates were left for ten to fifteen minutes [15,16].

Result and Discussion

Characterization of complexes (L1-L5)

The prepared complexes were identified through spectroscopic measurements, including FT-IR. The complex $[Hg(L)_2]$ showed a new band at (489)cm^{-1} attributed to (Hg-O)[17], and a second band at (424)cm^{-1} attributed to (Hg-N), with a decrease in the value of (C=O) to appear at (1660) cm^{-1}. The rest of the groups maintained the same ranges[18]. The (NH) band appeared at (3288) cm^{-1}, a band at position (3064) cm-1 attributed to (Ar-CH), and a band at position (1084) cm^{-1} attributed to (C=S). When studying the ^{1}H-NMR spectrum of this complex, the (NH) signal, intermediate between (C=O) and (C=S), disappeared, and the rest of the protons maintained their ranges. A binary signal appeared at (7.45, 7.43) ppm attributed to (=CH). Adjacent to the carbonyl, another double signal at (6.34, 6.32) ppm is attributed to (=CH) adjacent to the (NH) group, whose proton (NH) appears at position (7.16) ppm. When identifying the complexes (L2-L5), the FT-IR spectrum was studied, which showed the presence of a new band in the range (1437-1433) cm^{-1}, usually for (P-Ph), and another band in the range (1066-1060)cm^{-1} several for (C-P), indicating the association of phosphines with L1 [19,20], and the appearance of a double band in the complexes (L3-L5) in the range (2924-2904 & 2870-2860) cm^{-1}, usually for the aliphatic (CH), and the other

groups were at the same ranges, as a band appeared at (499-476) cm^{-1}, usually for (Hg-O), and a band in the range (449-445) cm^{-1}, usually for (Hg-N), and the (C=O) group appeared in the range (1664-1658) cm^{-1}, while the (C=S) group band appeared in the range (1101 -1095) cm^{-1}.

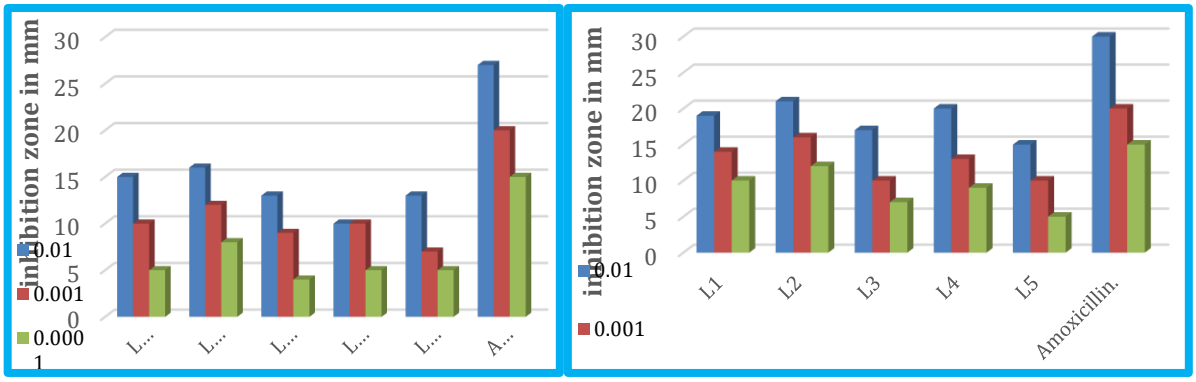
When studying the ^{1}H-NMR spectrum of complexes (L2-L5), multiple signals appeared, attributed to the protons of the benzene rings bound to phosphine in the range (7.65-7.29) ppm. In complex L3, a single signal was found at (1.83) ppm, typically for (CH_2) phosphine. In complex L4, a triplet signal, typically for (CH_2-CH_2) in the range (2.23-2.21) ppm, was found. Complex L5 showed a triplet signal, typically for (CH_2-P) bound to phosphine, at (2.13-2.09) ppm, and a pentagonal signal, attributed to the intermediate (CH_2) in the range (1.81-1.68) ppm [21]. When studying the ^{31}P-NMR spectrum of the complexes (L2-L5), there is a signal indicating that the complexes have one isomer in the range (36.01-25.68) ppm [22].

Biological activity test results

The study of the biological activity of the prepared complexes showed a direct relationship between the concentration and inhibition of these complexes. The higher the concentration, the greater the inhibition diameter. Complex L2 also showed the highest effectiveness at high concentrations against both species used in the study [23,24]. The inhibition diameter against Escherichia coli reached about 16 mm, while against Staphylococcus aureus it was 21 mm at a concentration of 0.01 mg/ml. In general, the complexes showed higher effectiveness against positive bacteria compared to negative bacteria. This is due to the ease of binding of the complexes to the cell walls of these bacteria compared to the antibiotic amoxicillin, which maintained its superiority against the studied bacteria [25-28].As in Table 1 and Scheme 2

Table (1): Antibacterial activity of the synthesized compounds (inhibition zone in mm).

Comp. No.	*E.coli*.mg/ml			*Staph. aureus* mg/ml		
	0.01	**0.001**	**0.0001**	**0.01**	**0.001**	**0.0001**
L_1	15	10	5	19	14	10
L_2	16	12	8	21	16	12
L_3	13	9	4	17	10	7
L_4	10	10	5	20	13	9
L_5	13	7	5	15	10	5
Amoxicillin.	27	20	15	30	20	15



Scheme (2): Inhibitory activity of (L_1-L_5) for *E.coli* & *Staph. aureus*

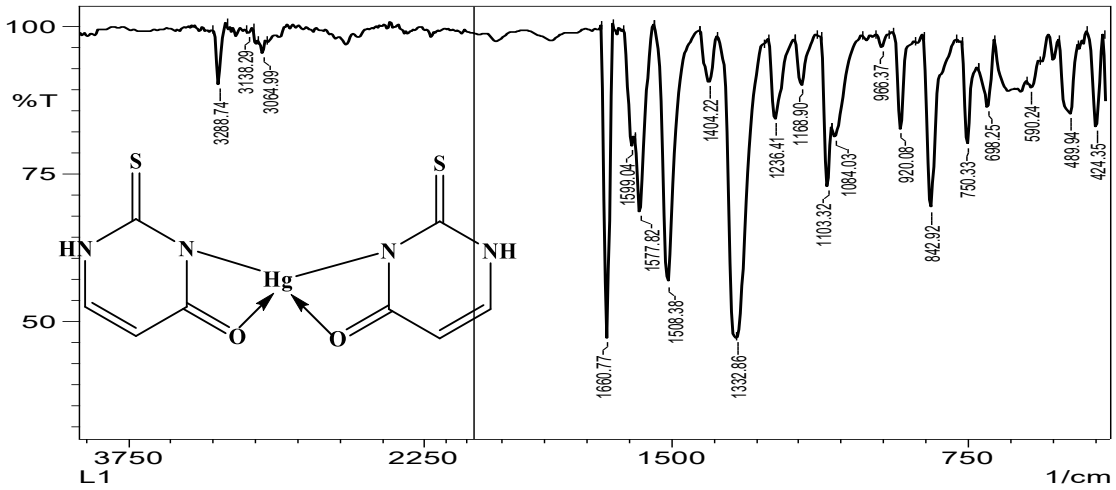


Figure (1): FT-IR spectra (L1)

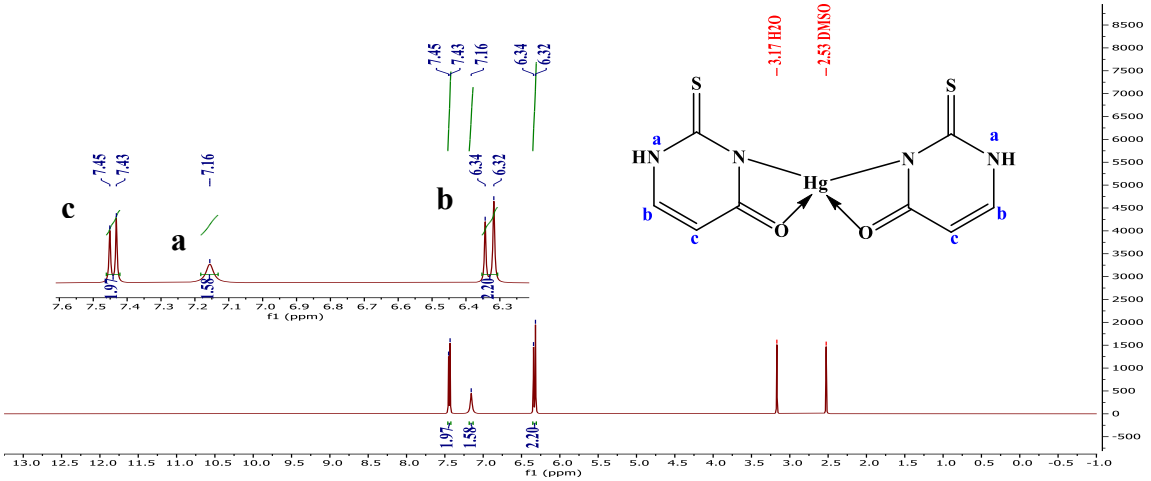


Figure (2): ^{1}H-NMR spectra (L1).

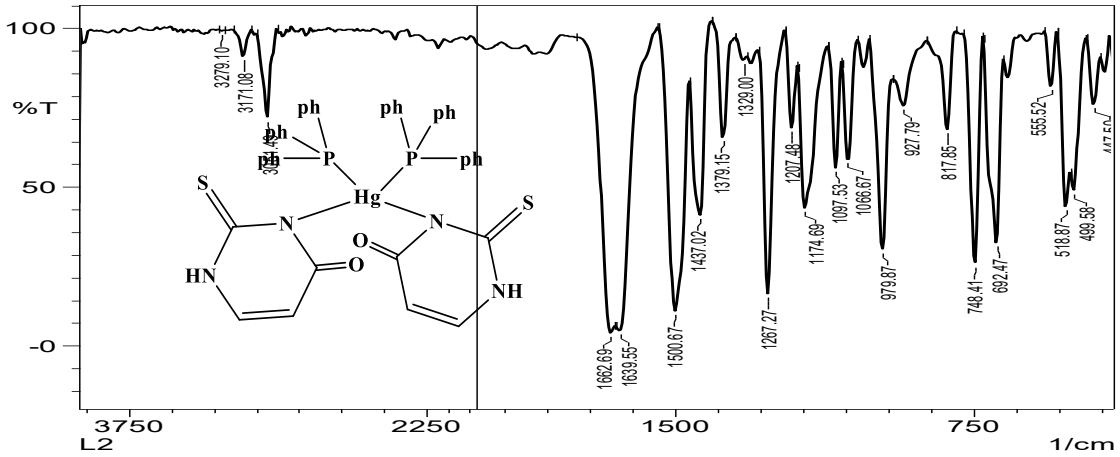


Figure (3): FT-IR spectra (L2).

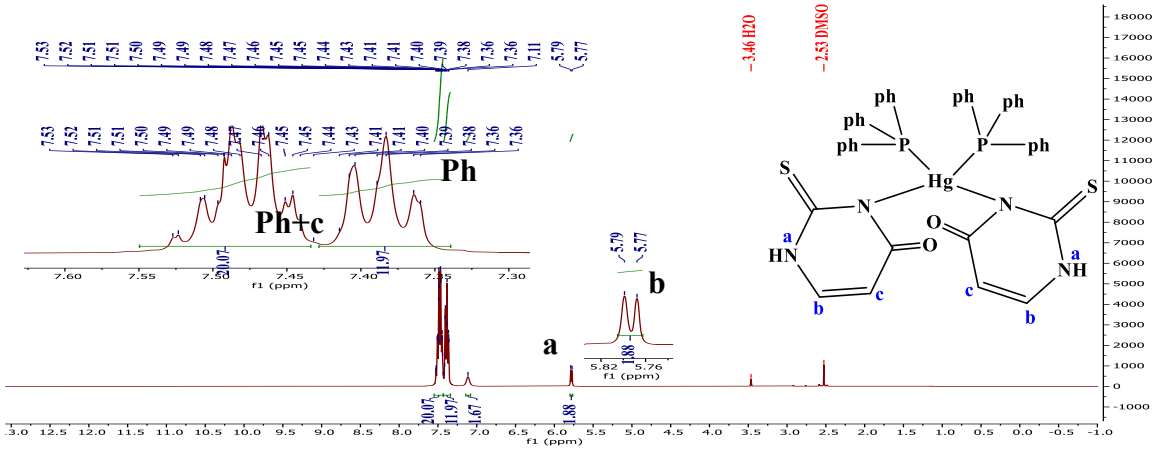


Figure (4): ^{1}H-NMR spectra (L2).

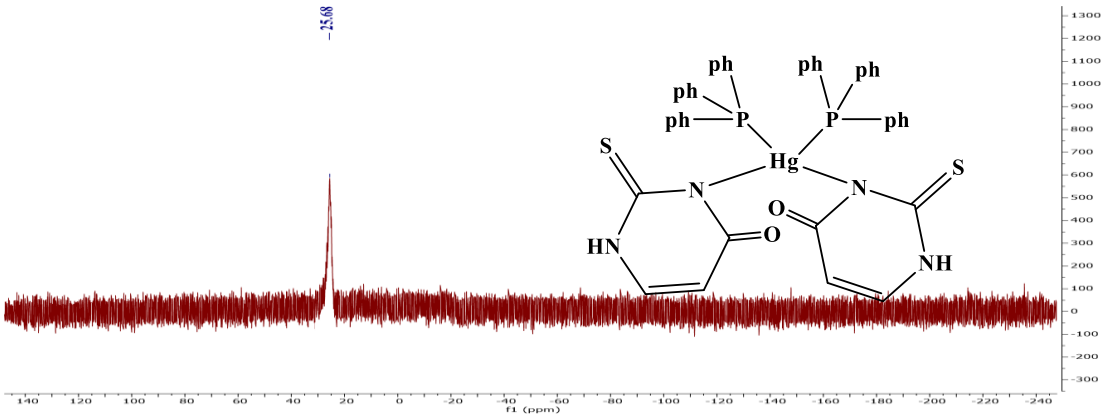


Figure (5): ^{31}P-NMR spectra (L2).

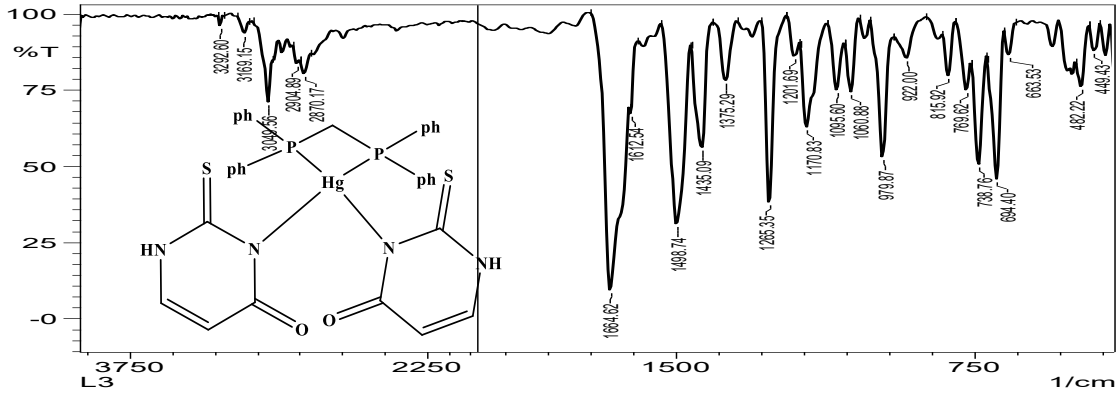


Figure (6): FT-IR spectra (L3).

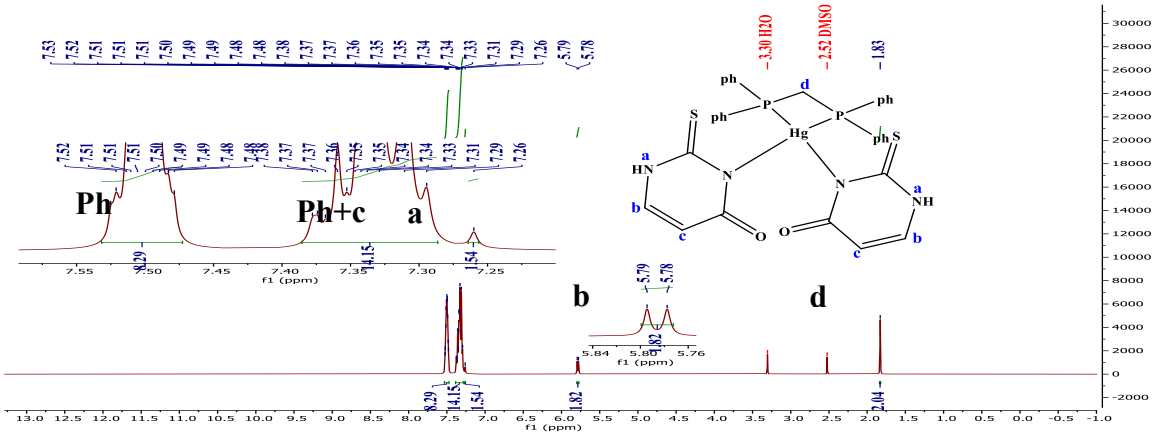


Figure (7): 1H-NMR spectra (L3).

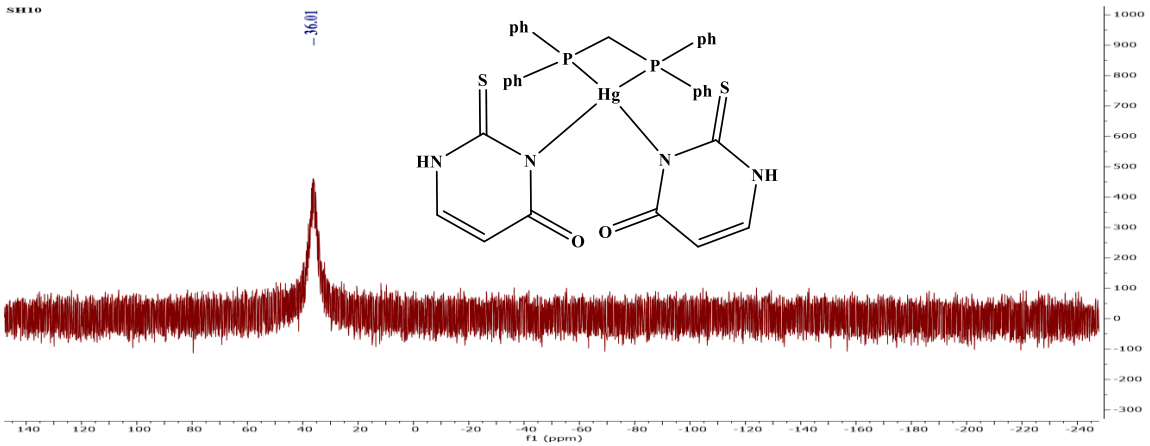


Figure (8): ^{31}P-NMR spectra (L3).

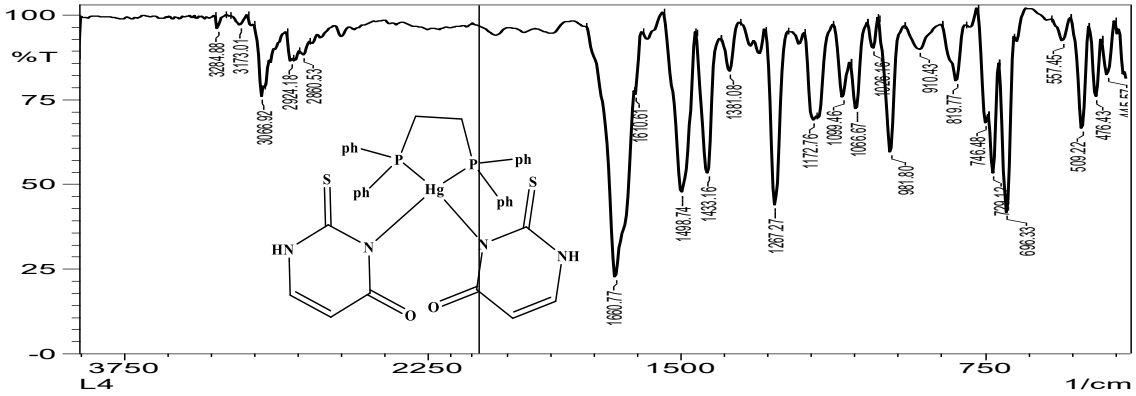


Figure (9): FT-IR spectra (L4).

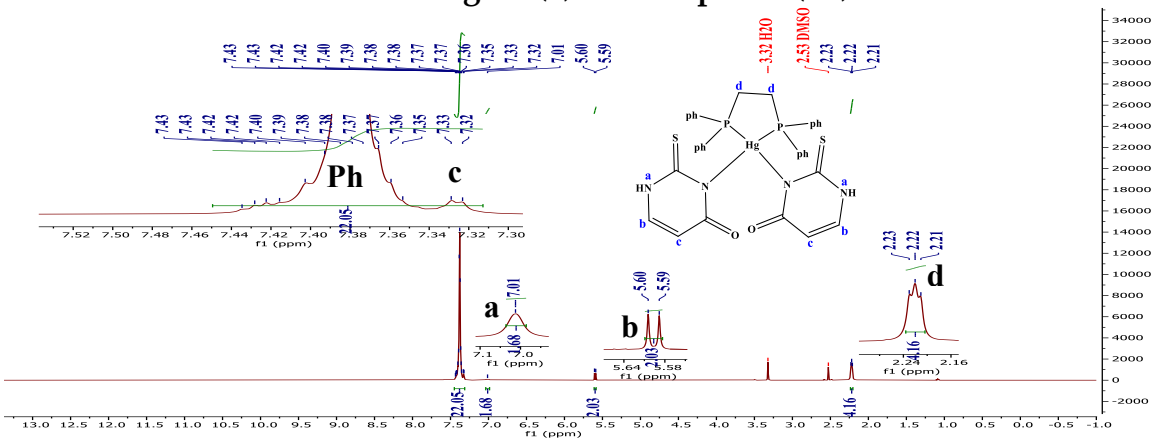


Figure (10): ^{1}H-NMR spectra (L4).

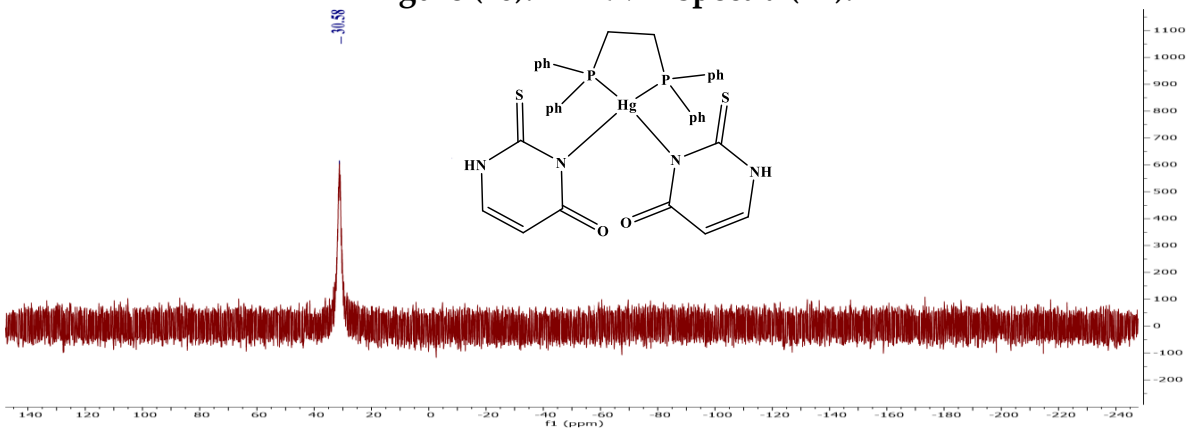


Figure (11): ^{31}P-NMR spectra (L4).

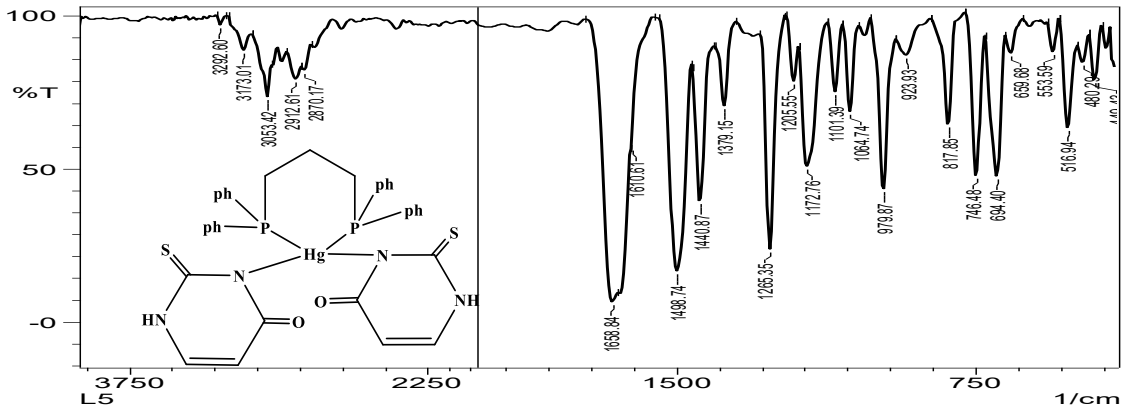


Figure (12): FT-IR spectra (L5).

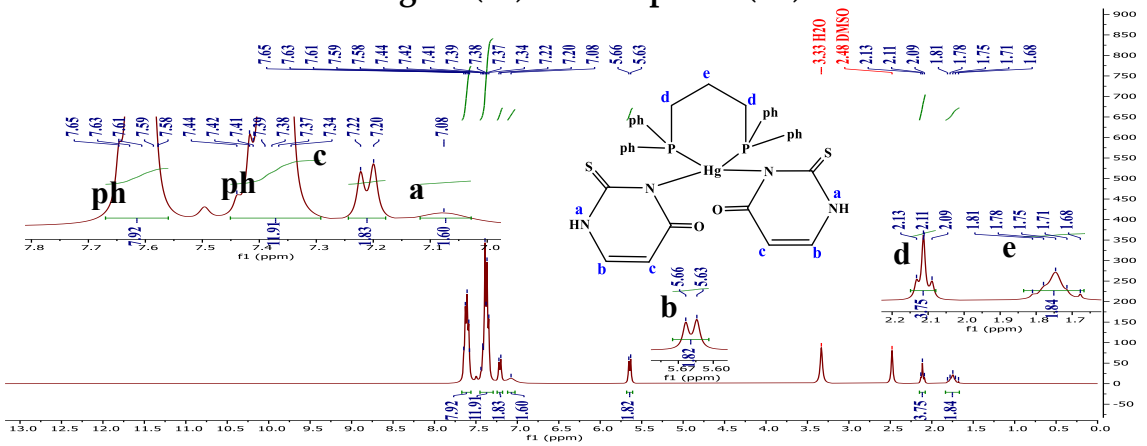


Figure (13): 1H-NMR spectra (L5).

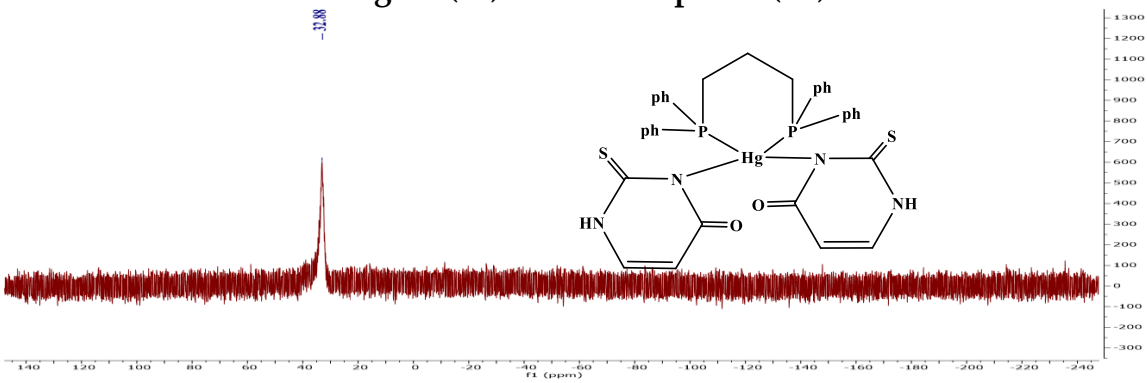


Figure (14): ^{31}P-NMR spectra (L5).

Conclusion

2-Thiouracil glycanide was used in the preparation of several phosphine complexes. The accuracy and validity of the results were confirmed by spectroscopic measurements, as bands were observed belonging to (Hg-N) and (Hg-O), indicating the association of mercury with the ligand, and the appearance of bands such as (P-Ph) and (C-P), indicating the association of phosphine with mercury. The 31P NMR spectrum showed a single signal, indicating that the association is diastereomeric, with the presence of only one isomer in the complexes containing phosphine. The complexes showed good effectiveness against the positive and negative bacteria under study. Complex L2 showed the highest effectiveness against both strains of bacteria under study.

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