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Research Article

Synthesis and Studies of Potential Antifungal and Antibacterial Agents New Aryl Thiazolyl Mercury (II) Derivatives Compounds

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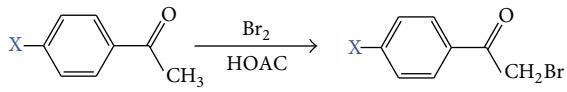
Combination of mercaptothiazoles and mercury phenyl chloride synthesized some new compounds of thiazoles. Firstly some mercaptothiazoles with different sorts have been synthesized, and then synthesized compounds were reacted with different mercury phenyl chloride structures. At last, each of these synthesized compounds was purified. Consequently, these structures were recrystallized using oil ether. Forming product through chromatogram (TLC) and combination of R_f with other compound's R_f of were identified, and their purity percent was recognized. The ^{1}H-NMR and other methods like FT-IR and mass spectroscopy have determined all of compounds. Obtained results of synthesized compounds showed that reactions were carried out with suitable speed and high yield.

1. Introduction

Having had various biological and pharmaceutical activities, chemistry of heterocyclic compounds such as thiazoles has been specifically noted and spread widely. These compounds have shown different pharmaceutical and also antibacterial and antiviral activities; therefore they are of great remedial importance [1–12]. In November 18th, 1887, molecular structure of thiazole was found and confirmed by Hantzschet al.; they proved carbon existing between sulfur and nitrogen to be of high activity. The property led them to suggest prefix "meso" for this situation. In 1889, the first thiazole derivative was obtained by Popp et al. They provided 2-amino thiazole structure by diazotation. They are present in vitamin B1 [13] too. Thiazoles are such compounds applicable as antihyperglycemic [5]. This property has caused these compounds to have abundant use in medicine and pharmaceutical industries as being used in antituberculosis medicine for instance [7]. They are also in insecticides and antivermin compounds related to agriculture. Due to having two different nitrogenous and sulfurous groups, the compounds tend to attend such different chemical reactions as alkylation, oxidation, and cycloaddition [8–15]. High reactivity of the compounds has caused in many derivatives from them, which there are atoms of nitrogen, sulfur, and carbon being between them [16]. As a ligand in complexometric reaction is another use they have [17]. New compounds synthesis by some derivatives of phenacyl bromide, mercaptothiazole, and mercury (II) chloride is what being tried to be studied in present research. Of course it should be pointed out that these compound's applicable and pharmaceutical properties can be studied in detail in future.

2. Experimental Details

2.1. General Method. All chemical materials were purchased from Merck, a Germany firm. For this study, all instruments



X = CH_3 and Br

Figure 1: Synthetic route for the phenacyl bromide derivatives structure.

that are being used are as follows: microwave oven model LG-SOLARDOMLF-5901WCR, Japanese weighing with a sensitivity of 0.100 g model ANDGF-300, hitter and stirrer model Heidolph Rn 3004 Safty, Merck's thin-layer chromatography sheets Art number: 1:0554, electrothermal apparatus of melting point measurement, Memmert (oven) materials and glossy instruments of desiccators (oven), vacuum pomp Emerson model: C55-JJXH4205, Rmp: 1425/1725, Heidolph model: Labrota rotary solution. In ^{1}H-NMR spectra studying done by Bruker Avance 300 spectrometer with the processing software XWINNMR version 3.1. measured chemical shifts of TMS. FT-IR by a Perkin Elmer spectrum of 1420 spectrometer in the frequency range of 4000–400 cm^{-1} using KBr discs are was reported on 1 scale. The IR spectra were recorded at room temperature with the spectral resolution of 1 cm^{-1}. Used glossy instruments are as follows: round-bottom emery-top flasks with one or two ports species. Simple, bubble, and spiral radiator, Erlenmeyer flask, beakers, three and two ports, links, addition funnel, Buchner funnel, capillary tube, and so forth.

2.2. Synthesis Methods. Thiazoles and mercury phenyl chloride family separately were synthesized, and, consequently, many new kinds of aryl mercury (II) thiazolyl were obtained from these compounds.

2.2.1. Preparation of Ammonium Dithiocarbamate. 100.0 mL of ethanol 95% was poured in 250.0 mL of Erlenmeyer. It was kept in the ice bath, and then it was exposed to ammonia gas until weight increasing approximately 39.0 g was obtained. 60.0 mL (1.0 mol) of carbon disulfide and 200.0 mL of diethyl ether were added to the solution, and it was kept for 2-3 hours in the ice container and then for 1 day in the refrigerator. After this time, yellow crystals of ammonium dithiocarbamate were formed. Then content of container was strained and washed by ether (50.0 mL). 80.0 g ammonium dithiocarbamate was produced.

2.2.2. Synthesis of Phenacyl Bromide Derivatives Structure. For synthesis of 4-bromophenacyl bromide structure (1) in a Erlenmeyer flask of 250 mL (50.0 g), *p*-bromoacetophenone was solved in 100.0 mL of pure acetic acid in an Erlenmeyer flask of 250 mL, 5.1 mL molecular bromide was added slowly to obtained solution. Reaction plate temperature was 20 centigrades. Need le-like crystals are seen after 30 minutes, then obtained sediment in ethanol 95% was recrystallized. Then it was rinsed and dried. Result of this reaction was production of 48.0 g compound with a melting point of (108-109°C). The residue was purified by thin-layer chromatography on silica gel (1 : 2 cyclohexane-acetone) to give (48.0 g, 95%); a white solid; Mp: 108-109; IR (KBr): 3065, 2950, 2064, 1693, 1180, 644; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 4.44 (s, 2H, CH_2Br), 7.66–7.68 (d, 2H, 2-H, 6-H), 7.87–7.90 (J = 9 Hz, d, 2H, 3-H, 5-H).

Synthesis method of 4-bromophenacyl bromide (2) is similar to synthesis method of compound (1); however *p*-methylacetophenone is used instead of compound *p*-bromoacetophenone: to give (30.0 g, 85%); a white solid; Mp: 80–82; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.40 (s, 3H, CH_3), 4.53 (s, 2H, CH_2Br), 7.53-7.54 (d, 2H, 3-H, 5-H), 8.174–8.177 (J = 9 Hz, d, 2H, 2-H, 6-H).

2.2.3. Synthesis of Mercaptothiazole Derivatives Structure. For synthesis of 4-(4′-bromophenyl)-2-mercaptothiazole (3) in a 500 mL flask 5.1 g of 4-bromophenacyl bromide was solved in ethanol. Then 36.9 g ammonium dithiocarbamate was added and refluxed for 3 hours. Distiller is used for reaction in constant pressure and control of free gases. Existing solvent by Rotary led to produced yellowish solid being refluxed by 125.0 mL of benzenes for 15 minutes. Then it was cooled for producing crystalline sediments; then it was rinsed and dried, the outcome of reaction was 18 g product with melting point of (218°C). The residue was purified by thin-layer chromatography on silica gel (1 : 2 cyclohexane-acetone) to give (18.0 g, 99%); a pale yellow; Mp: 216–218; IR (KBr): 3128, 3050, 1628, 1401, 1259, 1180, 746; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 3.46 (s, 1H, SH Aliphatic), 7.37 (s, 1H, H-5), 7.61–7.63 (d, 2H, 2′-H, 6′-H), 7.67–7.69 (J = 9 Hz, d, 2H, 3-H′, 5-H′).

Synthesis method of compound 4-(4′-methylphenyl)-2-mercaptothiazole (4) is similar to synthesis method of compound (3); however, 4-methyl phenacyl bromide is used instead of 4-bromophenacyl bromide: to give (12.0 g, 80.3%); a yellow solid; Mp: 191–193; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.40 (s, 3H, CH_3), 3.16 (s, 1H, SH Aliphatic), 6.92 (s, 1H, H-5), 7.48-7.49 (d, 2H, 2′-H, 6′-H), 7.28–7.30 (J = 9 Hz, d, 2H, 3-H′, 5-H′).

2.2.4. Synthesis of Mercury (II) Chloride Derivatives Structure. For synthesis of 4-methoxyphenyl mercury (II) chloride (5) in an Erlenmeyer flask 27.4 g *p*-anisidine was solved in 100.0 mL concentrated HCl; until it reached cooling 5°C. Then 81.1 g of $NaNO_2$ solved in 313.0 mL H_2O was consequently added to mixture of reaction. 56.7 g of mercury chloride solved 17.7 mL and concentrated was added to HCl solution; resulted white sediment was filtered in vacuum distillation, rinsed by with cold water, and then dried. The 24.5 g NaCl was added to 2.0 g copper powder particles suspended in 25.0 mL H_2O at 5°C. Then mixture reaction was blended for 4 hours and was relaxed for 24 hours. Obtained sediment after filtering was rinsed with ethanol and extracted with acetone. However, resulting solid was recrystallized by Benzene. 2.1 g of yellowish crystal was obtained with a melting point of (243–245°C). The residue was purified by

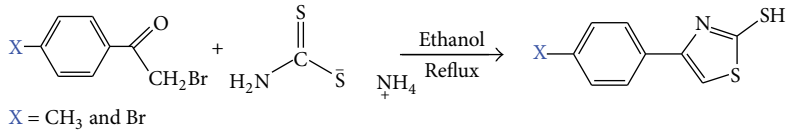


Figure 2: Synthetic route for the mercaptothiazole derivatives structure.

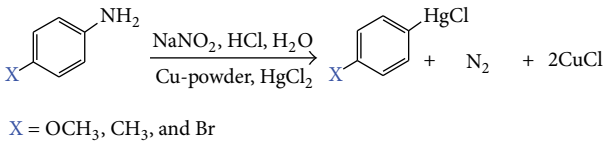


Figure 3: Synthetic route for the mercury (II) chloride derivatives structure.

thin layer chromatography on silica gel (1 : 2 cyclohexane-acetone) to give (2.1 g, 98%); A yellow crystal; Mp: 243–245; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 3.93 (s, 3H, OCH_3), 7.00 (J = 9 Hz, d, 2H, 2H, 2-H, 6-H), 7.90 (J = 9 Hz, d, 2H, 3-H, 5-H); MS m/z 343 (M^+, 89.24), m/z 107 (M^+, 100), m/z 77 (M^+, 52.82).

Synthesis method of 4-Bromophenyl Mercury (II) chloride (6) is similar to synthesis method of compound (5), however p-bromoaniline is used instead of p-anisidine: to give (1.7 g, 89%); A yellow solid; Mp: 249-250; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.69-7.70 (J = 9 Hz, d, 2H, 2-H, 6-H), 7.83-7.84 (J = 9 Hz, d, 2H, 3-H, 5-H).

Synthesis method of 4-methylphenyl mercury (II) chloride (7) is similar to synthesis method of compound (5); however, p-methyl aniline is used instead of p-anisidine: to give (1.9 g, 93%); A yellow crystal; Mp: 238-239; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.35 (s, 3H, CH_3), 7.12-7.13 (J = 9 Hz, d, 2H, 2-H, 6-H), 7.72–7.74 (J = 9 Hz, d, 2H, 3-H, 5-H).

2.2.5. Synthesis of Phenylmercury (II) Thiothiazole Derivatives Structure. For synthesis of 2-(4$''$-methoxy phenyl mercury (II) thio)-4-(4$'$-bromophenyl) thiazole (8) in an Erlenmeyer flask of 250 mL, 0.8 g p-methoxy phenyl mercury chloride was added to a solution of 0.5 g, 2-mercapto-4-(p-bromophenyl) thiazole in 25.0 mL of tetrahydrofuran in at (25–30°C). After blending of mixture for 3 hours and extracting solvent, solution was filtered by vacuum distillation and then closed for rotary to be done. Solid resulting from mixture of tetrahydrofuran oil was recrystallized. Through chromatogram (TLC), reaction was recognized by new sport. 1.0 g of white solid with melting point (235°C) resulted and output of 85%. The residue was purified by thin-layer chromatography on silica gel (1 : 2 ethyl acetate-CCl_4) to give (1.0 g, 85%); a white crystal; Mp: 233–235; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 3.97 (s, 3H, OCH_3), 7.54 (J = 9 Hz, d, 2H, 3$''$-H, 5$''$-H), 7.55 (s, 1H, 5-H), 7.57 (J = 9 Hz, d, 2H, 3$'$-H, 5$'$-H), 7.70 (J = 9 Hz, d, 2H, 2$'$-H, 6$'$-H), 7.90 (J = 9 Hz, d, 2H, 2$''$-H, 6$''$-H); MS m/z 578 (M^+, 83.36), m/z 471 (M^+, 100), m/z 239 (M^+, 52.82), m/z 156 (M^+, 43.52), m/z 107 (M^+, 63.03), m/z 77 (M^+, 51.52).

Synthesis method of 2-(4$''$-methoxyphenyl mercury (II) thio)-4-(4$'$-methylphenyl) thiazole (9) is similar to synthesis method of compound (8); however, 2-mercapto-4-(p-methyl phenyl) thiazole is used instead of 2-mercapto-4-(p-Bromophenyl) thiazole: to give (0.9 g, 82%); a white crystal; Mp: 108–200; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.12 (s, 3H, CH_3), 3.63 (s, 3H, OCH_3), 6.95 (J = 9 Hz, d, 2H, 3$''$-H, 5$''$-H), 7.30 (J = 9 Hz, d, 2H, 3$'$-H, 5$'$-H), 7.45 (J = 9 Hz, d, 2H, 2$'$-H, 6$'$-H), 7.49 (s, 1H, 5-H), 7.75 (J = 9 Hz, d, 2H, 2$''$-H, 6$''$-H).

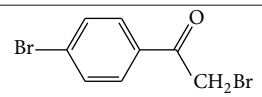
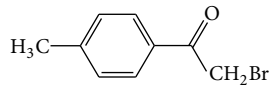
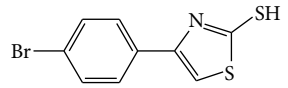
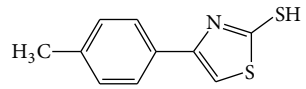
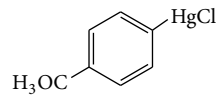
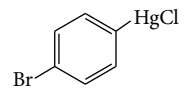
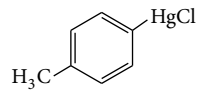
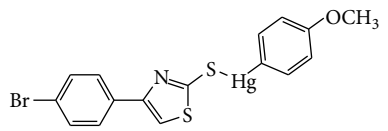
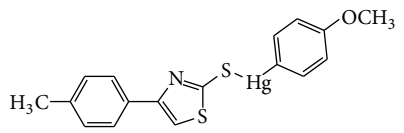
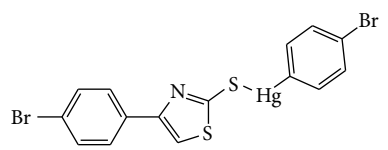
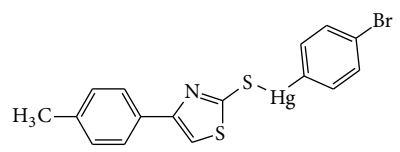
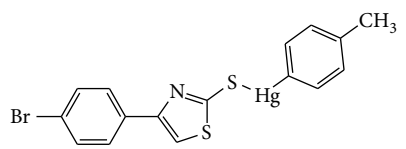
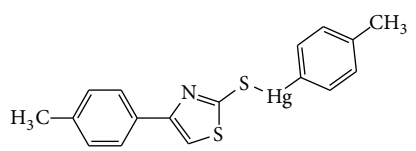
Synthesis method of 2-(4$''$-bromophenyl mercury (II) thio)-4-(4$'$-bromophenyl) thiazole (10) is similar to synthesis method of compound (8); however, 2-mercapto-4-(p-bromo phenyl) thiazole is used instead of 2-mercapto-4-(p-bromophenyl) thiazole: to give (1.15 g, 90%); a white crystal; Mp: 289–230; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.67-7.68 (J = 9 Hz, d, 2H, 3$''$-H, 5$''$-H), 7.69 (s, 1H, 5-H), 7.71-7.72 (J = 9 Hz, d, 2H, 3$'$-H, 5$'$-H), 7.80-7.81 (J = 9 Hz, d, 2H, 2$'$-H, 6$'$-H), 7.91-7.92 (J = 9 Hz, d, 2H, 2$''$-H, 6$''$-H).

Synthesis method of 2-(4$''$-bromophenyl mercury (II) thio)-4-(4$'$-methylphenyl) thiazole structure (11) is similar to synthesis method of compound (8); however, 2- mercapto-4-(p-methyl phenyl) thiazole is used instead of 2-mercapto-4-(p-Bromophenyl) thiazole: to give (1.05 g, 89%); a yellow crystal; Mp: 196-197; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.25 (s, 3H, CH_3), 7.15 (J = 9 Hz, d, 2H, 3$''$-H, 5$''$-H), 7.32 (J = 9 Hz, d, 2H, 3$'$-H, 5$'$-H), 7.49 (s, 1H, 5-H), 7.76 (J = 9 Hz, d, 2H, 2$'$-H, 6$'$-H), 7.85 (J = 9 Hz, d, 2H, 2$''$-H, 6$''$-H).

Synthesis method of 2-(4$''$-methylphenyl mercury (II) thio)-4-(4$'$-bromophenyl) thiazole structure (12) is similar to synthesis method of compound (8); however, 2-mercapto-4-(p-bromophenyl) thiazole is used instead of 2-mercapto-4-(p-bromophenyl) thiazole: to give (1.1 g, 93%); a white crystal; Mp: 243–245; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.35 (s, 3H, CH_3), 7.48 (s, 1H, 5-H), 7.50 (J = 9 Hz, d, 2H, 3$''$-H, 5$''$-H), 7.52 (J = 9 Hz, d, 2H, 2$'$-H, 6$'$-H), 7.75 (J = 9 Hz, d, 2H, 3$'$-H, 5$'$-H), 7.87 (J = 9 Hz, d, 2H, 2$''$-H, 6$''$-H).

Synthesis method of 2-(4$''$-methyl phenyl mercury (II) thio)-4-(4$'$-methyl phenyl) thiazole structure (13) is similar

Table 1: Yields and reaction conditions of the synthesized compounds.

Entry	Product(s)	Color	Time (min)	Yield (%)	M.p (°C)
1		White	30	95%	108-109
2		White	30	85%	80–82
3		Yellow	180	99%	216–218
4		Yellow	180	80.3%	191–193
5		Yellow	300	98%	243–245
6		Yellow	240	89%	249-250
7		Yellow	360	93%	238-239
8		White	180	81%	233–235
9		White	210	82%	108–200
10		White	240	90%	289–230
11		Yellow	240	89%	196-197
12		White	180	93%	243–245
13		Yellow	300	89%	185-186

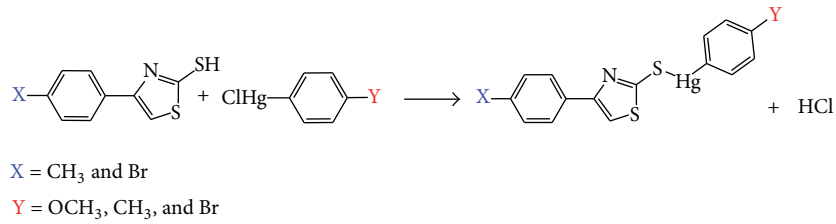


Figure 4: Synthetic route for the aryl thiazolyl mercury (II) derivatives structure.

to synthesis method of compound (8); however, 2-mercapto-4-(4′-methyl phenyl) thiazole is used instead of 2-mercapto-4-(*p*-bromophenyl) thiazole: to give (0.9 g, 89%); A pale yellow crystal; Mp: 185-186; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 2.15 (s, 3H, CH_3), 2.35 (s, 3H, CH_3), 7.10 (J = 9 Hz, d, 2H, 3″-H, 5″-H), 7.39 (J = 9 Hz, d, 2H, 3′-H, 5′-H), 7.45 (s, 1H, 5-H), 7.58 (J = 9 Hz, d, 2H, 2′-H, 6′-H), 7.67 (J = 9 Hz, d, 2H, 2″-H, 6″-H).

3. Result and Discussion

For 6 new kinds of synthetic compounds of aryl thiazolyl mercury (II) firstly, some compounds of Mercaptothiazoles were separately synthesized. After comparing R_f with other R_f, other compound identified and using ^{1}H-NMR spectrometer, IR spectrometer and finally mass spectrometer completed analyzing. For 6 synthetic compounds of thiazolyl mercury (II), the produce is shown in Figure 1.

Thiazoles family had shown that substitution of hydrogen atom by methyl group was caused to the decrease of yield and sigma electron density in carbon atom of the cycle. But there is small increasing in electron density when hydrogen atom is substituted by sulfur atom. But quality of substitution mechanisms is different in thiazoles group. For example, substitution of methyl group in thiazoles structure is done by heteroatomic and hyperconjugation models. It is done by changing of pay electron.

Finally, after analyzing the process and identifying considered compounds, 13 new compounds were recognized in which 6 of them were of thiazolyl mercury (II). Total process and obtained results are shown in Table 1 and Figures 2, 3, and 4.

The reaction of 4-methoxy-phenyl mercury (II) chloride with 2-mercaptothiazoles leads to formation of 2-(4″-methoxy-phenyl mercury-thio-thiazoles). *p*-Anisidine is used as an initiator, then 4-methoxy-phenyl mercury (II) chloride is prepared with news Meyanov's method. This was due to Diazo compound of *p*-anisidine and then reaction with HgCl and then reaction with copper powder; for the first reaction of 2-mercapto-4-(4′-methyl-phenyl) thiazole with 4-methoxy-phenyl mercury (II) chloride (with molar ratio of 1 : 1) in tetrahydrofuran (THF), then yellow wish crystal compound. In the NMR proton spectrum, have recognized the existence of all of signals of thiazoles part with substituent phenyl ring signals in the forth position. All of mentioned synthesis have followed from this method. We can explain this reaction in this way: because mercury is a soft acid thus can conjugate easily with donor atoms in polarizable soft bases. Mercury ion has great tendency for bounding with sulfur and the ligands containing sulfur atom. For above reasons, because of tendency bounding between mercury and sulfur in this research, we can bound two compounds of 4-(4′-bromophenyl)-2-mercaptothiazole with *p*-methoxy mercury chloride molecular and *p*-bromomercury chloride. Then consequently with exiting HCl we can obtain this bounding. For proving this theory such methods like TLC, ^{1}H-NMR, GC MASS, and IR are used in which all of them have recognized above claims. In this research, abundant usages of mercury in the dentistry and pharmacy and as a premier material of amalgam in dentistry that has repair usages. SCN bounds have biological and treatment properties, we have tried to investigate new ways and methods for synthesizing different thiazole derivatives. In this research, the physiological role of thiazolyl compounds has been examined and we have tried to synthesize some of its derivatives. The mechanism preparation 4-(4/-bromo-phenyl)-2-mercapto-thiazole with using *p*-bromoacetophenone and ammonium compounds and combination of ammonium dithiocarbamate in refluxed with ethanol and then benzene, 4-(4′-bromophenyl-2-mercaptothiazole) was obtained. For making aromatic mercerized compound aromatic aniline (type I) was used.

4. Conclusions

Methods of aromatic system were used to increase speed of mechanism of separating and purifying of products does not need complex process. Production of these structures of thiazolyl mercury (II) through mention mechanism and producing HCl. Practically, these compounds have higher functions than the same synthetic compounds.

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