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Synthesis of and characterization of some Heterocyclic Compounds derived from Thiophenol

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Abstract

This research work involved preparation of heterogeneous pent lateral cyclic compounds (thiazolidine -4- one, benzothiazole, triazole, 4-oxothiazolidin) using thiophenol as raw materials: Thiophenol was reacted with mono chloroacetic acid in the presence of potassium hydroxide to prepare (sh_1) followed by ortho amino aniline results the (sh_2). The reaction of thiophenol with ethylchloroacetate afforded (sh_3) and the reaction of (sh_3) with thiosemicarbazide and 4% NaOH leads to ring closure giving 1,2,4- triazole (sh_5). A treatment of thiophenol with hydrazine hydrate to obtain the intermediate (sh_6) with aromatic aldehyde synthesized azomethines (sh_7- sh_9) then treated with mercaptoacetic acid to obtained (sh_{10}-sh_{12}). A treatment of thiophenol with chloroacetyl chloride produced (sh_{13}) compound then treated with hydrazine hydrate to obtain (sh_{14}) compound followed by bromobenzaldehyde synthesized azomethine (sh_{15}) compound then treated with mercaptoacetic acid to obtained (sh_{16}) compound. Characterization results for the prepared compounds using IR spectroscopy, NMR and melting points confirmed their chemical structures.

Keywords: Thiophenol, Thiophenol derivatives, triazole, 4-oxothiazolidin, benzothiazol

1. Introduction

Thiols (R-SH) are a category of compounds with a lot of (– SH) moieties [1-2]. This moiety is extremely reactive, and it is often found conjugated to both organic and inorganic molecules [3]. The total thiol status in the body, especially the thiol (-SH) groups found in protein, are considered to be major plasma antioxidants in vivo, and major of them are found over albumin and they are the major reducing groups present in our body fluids [4]. In free radical polymerization, thiol compounds are also used as efficient, almost ideal chain transfer agents. The high reactivity of thiol radicals and the weakening of the S-H bond are responsible for this. The addition of thiols reduces the molecular weight of the polymer, while not affect the rate of polymerization. As a result, thiols are commonly used as additives to lower the molecular weights of a broad variety of polymers [5-7]. They are highly effective and valuable compounds in a variety of fields, including medicine, agriculture, industry, pharmaceutical, and heterocyclic chemistry [8]. They can also be served as building blocks for a variety of organ sulphur compounds [9]. The S-alkylation reactions are active for the synthesis of these compounds in both industry biology, especially in the amino acid methionine and the cofactor biotin [10-11]. Thiols can be reacted with some heavy metal ions such as Hg(II), Au(I), Ag(I), Cd(II), Zn(II), Cu(II), or As(III) across of Lewis acid-base interactions. This beaver leads to use as a selective sorbent for
the removal of several heavy metal ions in the water and wastewater treatment processes [12-14]. The aromatic thiols (thiophenols) are far more harmful than aliphatic thiols for example, in fish; thiophenols have a median lethal concentration (LC50) of 0.01–0.4 mm and in a mouse, a median lethal dose (LD50) of (46.2) $mg.kg^{-1}$ [15]. Several recent investigations have looked at the oxidation of thiols to disulfides or sulphonic acid in the absence and presence of catalysts [16-17]. The aim of the research is; synthesized new 2-((phenylthio) methyl)-1H-benzo[d]imidazole as shown at **Scheme I**; prepared new 5-(phenylthio) methyl-4H-1,2,4-triazole-3-thiol as shown at **Scheme I**; made cyclization of derivatives of benzylidene -2-phenyl hydrazine from its reaction with mercaptoacetic acid as shown at

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Scheme I; synthesized new N- (2-(4- bromophenyl)-4-oxothiazolidin -3-yl)-2-(phenylthio) acetamide as shown at **Scheme I** and characterized these derivatives using different available spectroscopic technique.

2. Experimental:

A. Materials

Chemical materials: The chemicals used in this work were obtained from BDH and Fluka and used directly without recrystallization.

B. Methods:

Synthesis of Thiophenol and some its derivatives (sh_1-sh_{16}) as presented in **Table 1** [18-20]

The compound of 2-phenylthioacetic acid (Sh_1) preparation

0.02 (mole) of KOH was added to [0.02 (mole),3.203(g)] Thiophenol in 25 (mL) of ethanol absolute and then [0.02(mole), 0.118(g)] of mono chloroacetic acid was added to the mixt, mixture was refluxed for 8 hrs., the precipitate been washed and recrystallized with ethanol.

The compound 2-((phenylthio)methyl)-1H-benzo[d]imidazole (Sh_2) preparation

[0.01(mole), 1.68(g)] of compound (Sh_2) been leaved for 10 hrs. in 4 (N)HCl 20 (mL) with [0.01(mole), 1.08 (g)] O-phenylene diamine , the ammonia was neutralized the mixture , the precipitate Benz imidazole was filtered and recrystallized from ethanol.

The compound ethyl 2-(phenylthio) acetate (Sh_3) preparation

Ethyl chloro acetate [0.02 (mole), 2.46 (g)] was added drop wise to a hot solution of compound thiophenol [0.02 (mole),2.20 (g)] and potassium carbonate in acetone as a solvent. The mixture was refluxed for 4 hrs, and allowed to stand evaporating the solvent under reduced pressure. water was added and the crude product was extracted with ethyl acetate and dried over anhydrous magnesium sulphate evaporating of the organic layer gave solid products, recrystallized from ethanol.

The compound of 2-(2-(phenylthio) acetyl) hydrazine -1- carbothioamide (Sh_4) preparation

To stirring solution of compound (sh_3) [0.01(mole),1.96(g)] in 20(mL) ethanol absolute was added thiosemicarbazide [0.01(mole), 0.91 (g)] the mixture was refluxed for 4 hrs. The cold was filtered and recrystallized from ethanol and water.

The compound of 5-(phenylthio) methyl-4H-1,2,4-triazole-3-thiol (Sh_5) preparation

Exact [0.01(mole),2.23(g)] of compound (sh_4) in 10 (mL) of (4%of NaOH] and heated under reflux for 3hrs then acidified with 10% HCl solution the solvent was removed and the formed solid was recrystallized from ethanol.

The compound of phenyl hydrazine (sh_6) preparation

A mixture [0.01(mole), 1.101 (g)] of thiophenol was added to 10(mL) hydrazine hydrate mixture was refluxed for 4 hrs., and the 15 (mL) of ethanol absolute was added to the mixture was refluxed for 3 hrs. After separate, the mixture filtered the precipitate and has been washed with cold water and recrystallized from ethanol.

The compounds of 1-(derivatives of benzylidene)-2-phenyl hydrazine (sh_7- sh_9) preparation

A suspension of p- bromobenzaldehyde [0.01 mole] in 15mL ethanol and 0.01(mole) compound (6) were mixed with two drops of glacial acetic acid and heated under reflux for 6 hrs. The product was collected after cooling and recrystallized using ethanol, in the same way for preparation compounds (8,9).

The compounds of 2-(derivatives)-3- (phenyl amino) thiazolidin-4-one (sh_{10}- sh_{12}) preparation

0.02(mole) mixture of Schiff bases (sh_7- sh_9) and [0.04 (mole), 0.26 (mL)] mercaptoaceticacid in 30 (mL) in 30 mL from dry benzene was mixed, the reaction mixture was refluxed for 12 hrs. The precipitate was filtered off and recrystallized with using ethanol.

The compound 2-phenylthion acetyl chloride(sh_{13}) preparation

Freshly distilled chloroacetyl chloride (2.5) mL dissolved in dry benzene 100 (mL) was gradually added to mixture of [0.033(mole), 5.61(g)] Thiophenol in dry benzene 30 (mL), the reaction mixture was refluxed on a water bath for (2hrs.). Benzene was distilled off and the residue washed with 5% sodium bicarbonate solution and finally washed with distilled water. The precipitate was filtered off and recrystallized with using ethanol.

The compound 2- phenylthio acetohydrazide(sh_{14}) preparation

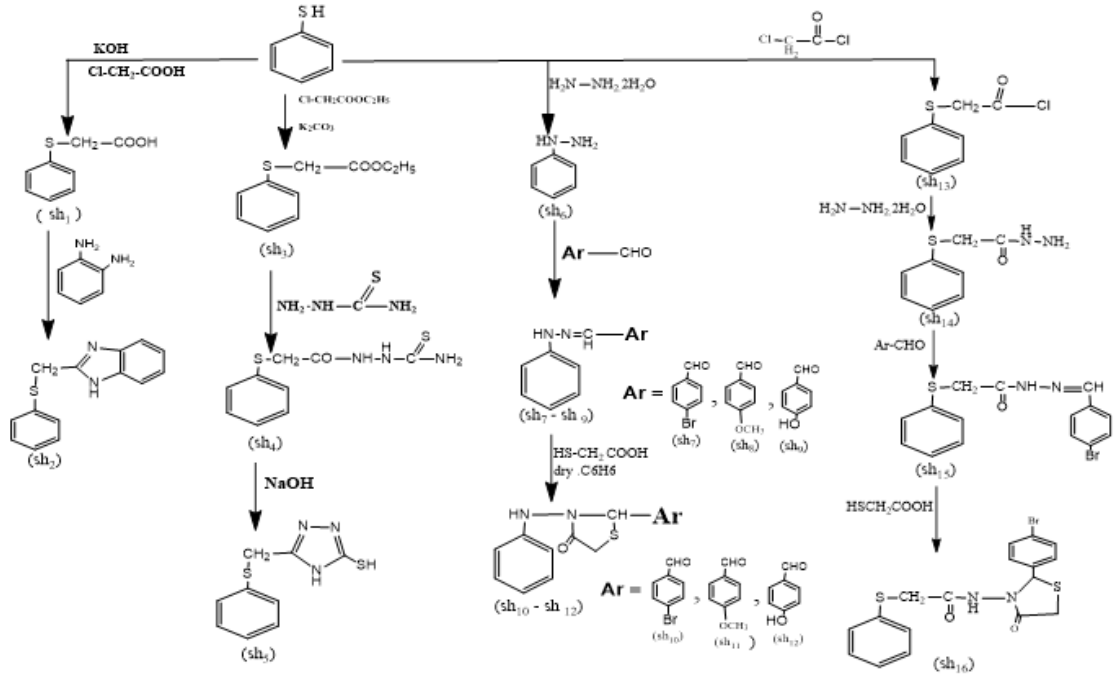
A mixture of [0.01 (mole), 1.82 (g)] from compound (sh_{13}) was added to 10 (mL) hydrazine–hydrate mixture was refluxed in 15 (mL) of ethanol, were heated under reflux for 7 hrs., and then the reaction mixture was filtered, recrystallization of the product by ethanol and dried to give the final product.

The compound N`-(4-bromobenzylidene)-2-(phenylthio)acetohydrazide (sh_{15})preparation

A suspension of [0.01(mole), 1.82(g)] (sh_{14}) compound and [0.01(mole), 1.85(g)] of bromobenzaldehyde in 10(mL) ethanol. The reaction mixture was refluxed for 7hrs. and then the reaction mixture was filtered, recrystallization of the product by ethanol and dried to give the final product.

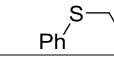
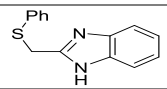
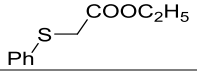
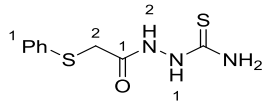
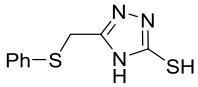
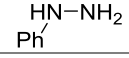
The compound N- (2-(4- bromophenyl)-4-oxothiazolidin -3-yl)-2-(phenylthio) acetamide (sh_{16}) preparation

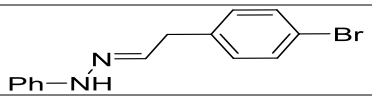
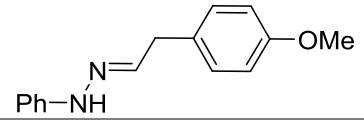
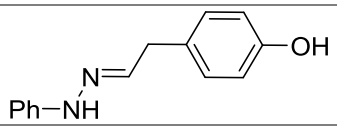
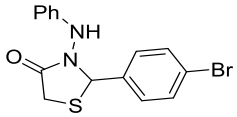
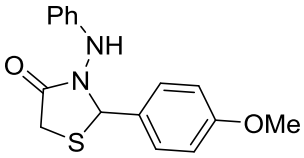
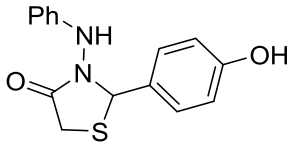
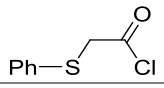
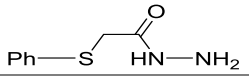
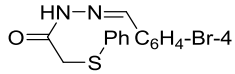
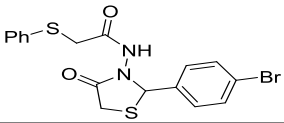
[0.002 (mole), 0.6 (g)] mixture of Schiff base (sh_{15}) and [0.004 (mole), 0.36 (mL)] mercaptoaceticacid in 20 (mL) from dry benzene were mixed, refluxed and stirring on a water bath at 75^{0}C for 10 hrs. dried and recrystallized from ethanol.



Scheme I. Schematic diagram of prepared compounds in the present work

Table 1: Physical properties of the prepared compounds (Sh_1-Sh_{16})

NO	Structure and nomenclature	yield	color	M.P	FT-IR
Sh_1		%77	white	276-278	**3445(υ_{O-H}),3191(υ C-H, benzene ring), 2923 (υ_{as} C-H ,CH2)1727($\upsilon_{C=O}$) , 1331(υ_{C-O}).**
	2-(phenylthio)acetic acid				
Sh_2		%92	brown	182-185	**3362and 3163(υ_{N-H}), 3058(υ C-H, benzene ring),2922(υ_{as} C-H ,CH2) ,1662 (υ C=N),1618(δ_{N-H}) .**
	2-((phenylthio)methyl)-1H-benzo[d]imidazole				
Sh_3		%65	Pale yellow	287-291	**3080 (υ C-H, benzene ring),2924 (υ_{as} C-H ,CH2),1730 ($\upsilon_{C=O}$) , 1616 (υ C=C),1095 (υ_{C-O}).**
	ethyl 2-(phenylthio)acetate				
Sh_4		%95	Pale brown	196(dec.)	**3337 and 3166(υ_{NH2}), 3060 (υ C-H, benzene ring), 2926 (υ_{as} C-H ,CH2), 1663($\upsilon_{C=O}$) ,1509($\upsilon_{C=C}$) ,1165($\upsilon_{C=S}$) .**
	2-(2-(phenylthio)acetyl)hydrazine-1-carbothioamide				
Sh_5		%63	Brown	211-215	**3060(υ C-H, benzene ring), 2924(υ_{as} C-H ,CH2), 2581(υ S -H) , 1650 (υ C=N), 696(υ_{C-S}) .**
	5-((phenylthio)methyl)-4H-1,2,4-triazole-3-thiol				
Sh_6		%52	white	206 (dec.)	**3058 (υ C-H, benzene ring), 3330and3235 (υ N -H), 1330(υ_{C-N}) .**
	phenylhydrazine				

Sh7		%55	yellow	269-271	**3061(υ C-H, benzene ring),1650(υ C=N), 1066 (υC-Br) .**
	1-(4-bromobenzylidene)-2-phenylhydrazine				
Sh8		%57	Pale yellow	211-214	**3055(υ C-H, benzene ring),1636 (υ C=N) , 1269(υasC-O-C).**
	1-(4-methoxybenzylidene)-2-phenylhydrazine				
Sh9		%61	yellow	202-204	**3453(υ O-H) , 3036(υ C-H, benzene ring),1650 (υ C=N) ,1305(dO-H),1128(υc-o).**
	4-((2-phenylhydrazineylidene)methyl)phenol				
Sh10		%66	Pale brown	203-207	**3271(υ N -H),1676(υ C=O), 1251(υC-N-H),693(υC-S),1062(υC-Br) .**
	2-(4-bromophenyl)-3-(phenylamino)thiazolidin-4-one				
Sh11		%72	brown	225-229	**3269(υ N -H), 1670(υ C=O), 1249(υC-N-H) , 690(υC-S), 1266(υasC-O-C).**
	2-(4-methoxyphenyl)-3-(phenylamino)thiazolidin-4-one				
Sh12		%63	white	217-220	**3449(υ O-H) ,3191(υ N -H),3060(υ C-H, benzene ring),1680(υC=O) ,1615(dN-H).**
	2-(4-hydroxyphenyl)-3-(phenylamino)thiazolidin-4-one				
Sh13		%55	brown	232-235	**2927(υas C-H ,CH2),2851(υ C-H ,CH2),1715(υC=O) ,3049(υ C-H, benzene ring),1093(υc-s-c).**
	2-(phenylthio)acetyl chloride				
Sh14		%34	Pale brown	265(dec.)	**3450,3277(υNH2), 3174(υ N -H), 2925,2851(υ C-H ,CH2) ,1085(υc-s-c),1614(υC=O).**
	2-(phenylthio)acetohydrazide				
Sh15		%67	yellow	198-201	**1650(υC=O), 3188(υ N -H),2920and 2842(υ C-H ,CH2) , 1667(υC=N) ,1069(υC-Br) .**
	N'-(4-bromobenzylidene)-2-(phenylthio)acetohydrazide				
Sh16		%82	brown	275-278	**2923 and 2839(υ C-H ,CH2) , 3195 (υ N -H),1648(υC=O), 1083(υc-s-c),1071(υC-Br) .**
	N-(2-(4-bromophenyl)-4-oxothiazolidin-3-yl)-2-(phenylthio)acetamide				

3. Results and Discussion

Compound (sh_2) was confirmed spectroscopically by FT-IR which[21] showed (3362-3163) cm^{-1} attributed to asymmetric and symmetric stretching vibration of (-NH) group, the peak (2922) cm^{-1} maybe beyond to the (- CH_2)group and strong band at 1662 cm^{-1} referred to the stretching vibration of imine group (C=N) group ,the stretching band at (1618) cm^{-1} was due to the scissoring of (N-H) group. As indicated in **Figure 1**.

H^1-NMR spectrum of compound (sh_2) in (DMSO as solvent) was pictured in **Figure 2**, A singlet signal at 3.9ppm could be attributed to protons of methylene group (-CH_2-). Aromatic protons appear at (7.6 -7.8) ppm. A singlet signal appears at 12.1 ppm referred to N-H group.

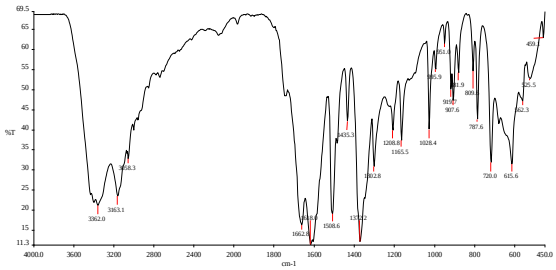


Figure 1. FT-IR spectrum of compound (sh_2)

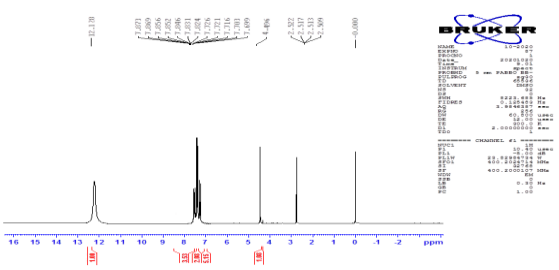


Figure 2. H^1-NMR spectrum of compound(sh_2)

Figure 3 shows the FT- IR spectrum of compound (sh_4). showed [22] strong absorption at (3337- 3166) cm^{-1} which represent to the asymmetric and symmetric stretching vibration of (- NH_2),while the band at 3060 cm^{-1} related to the aromatic ring , while the band at 2926 cm^{-1} attributed to the stretching vibration (-CH_2), showed appearance of the band at 1633 cm^{-1} was due to the stretching vibration of the (C=O) group ,beside this the appearance of (C=S) stretching band at 1165 cm^{-1}.

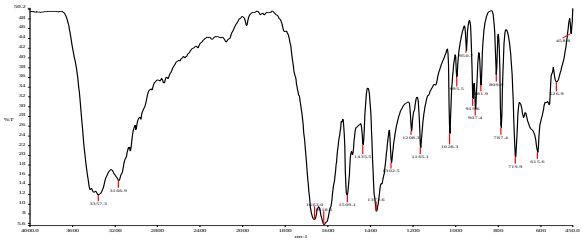


Figure 3. FT-IR spectrum of (sh_4) compound

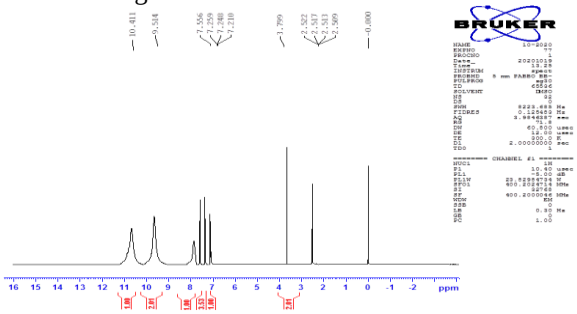


Figure 4. H^1-NMR spectrum of (sh_4) compound

From **Figure 4** which indicated H^1-NMR for compounds (sh_4) showed A signal at 3.7 ppm (2H) due to the protons of (CH_2), another multiple signals at (7.21-7.25) ppm referred to aromatic protons, the signal at 7.55ppm referred to NH-CO, the signal at 9.5 ppm (2H) was due to the protons of (-NH_2) group, the signal at 10.4 ppm (1H) due to the proton of (NH-C=S) group.

Figure 5 indicates FT- IR for compound (sh_5) [23]and found a band at 3060 due to stretching of aromatic ring showed appearance peak (2924) cm^{-1} maybe beyond to the (-CH_2-), The spectra also showed appearance band at (2581) cm^{-1} assigned to (-SH) group, and appearing (1650) cm^{-1} which assigned to υ (C=N) group, the stretching band at 696 cm^{-1} belong to the (C-N) group.

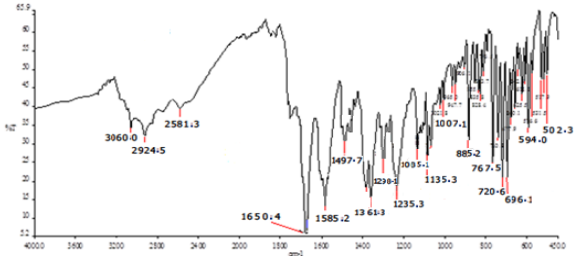


Figure 6. H^1-NMR spectrum of (sh_5) compound.

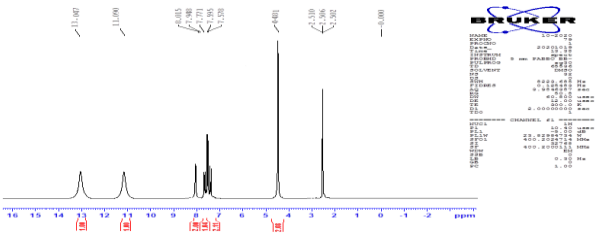


Figure 5. FT-IR spectrum (sh_5) compound

Figure 6 shows the H^1-NMR spectrum (400 MHZ, in DMSO) of compounds (sh_5), indicated which showed a signal at 4.4ppm referred to proton of CH_2 group, aromatic protons appear at (7.5- 8.0) ppm as amultiplate peaks, a signal at 11.0 ppm was due to the absorption of proton for (NH) group of triazole ring, A signal at 13.0 ppm referred to (-SH) group.

Figure 7 explains H^1-NMR for compounds (sh_7), aromatic protons appeared at the range (7.06- 7.64) ppm, signal at 8.06 ppm was due to the proton of the (N=CH) group, A signal at 10.5ppm which belong to (-NH) group.

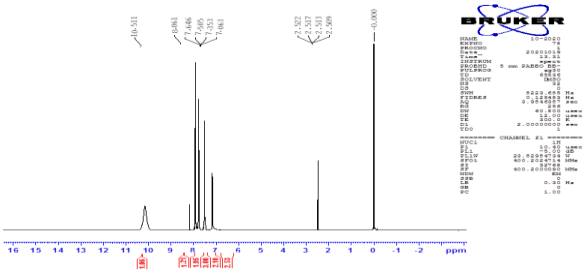


Figure 7. H^1-NMR spectrum of (sh_7) compound

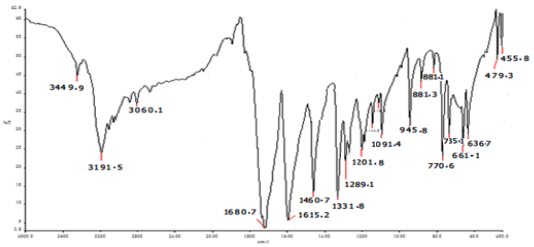


Figure 8. FT-IR spectrum (sh_{12}) compound.

Figure 8 explains theFT-IR spectrum of compound (sh_{12})[24-25], the stretching vibration band at 3449 cm^{-1} was due to the (OH) group and appearing 3191 cm^{-1} due to the stretching vibration of (N-H) group ,aromatic protons of (C-H) at 3060 cm^{-1} besides this appearance of scissoring of the (N-H) band at 1615 cm^{-1}.

Figure 9 indicates to the FT-IR spectrum of compound(sh_{14}), which appearance of the band at 3450) cm^{-1} and (3277) cm^{-1}attributed to asymmetric and symmetric stretching vibrations of amino groups (NH_2) , and appearing of absorption band at (3174) $cm^{-1}$was due to the stretching vibration of the (-NH-) group , the bands 2926 cm^{-1} and 2853 cm^{-1} belong to the asymmetric and symmetric stretching vibrations of ($-CH_2$)in the case of compound(sh_{14}) three further bands appear at 1683 cm^{-1}, 1614 cm^{-1} and 1085 cm^{-1}which were belong to the Ʊ(C=O), δ (N-H) and Ʊ (C-S-C) groups [26-28].

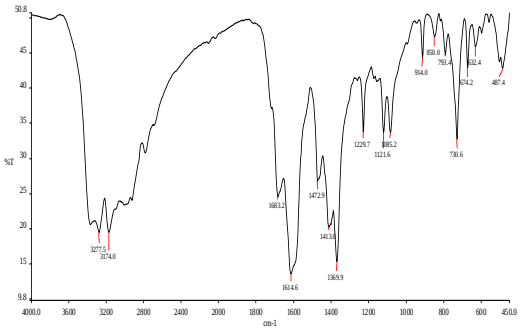


Figure 9. FT-IR spectrum (sh_{14}) compound.

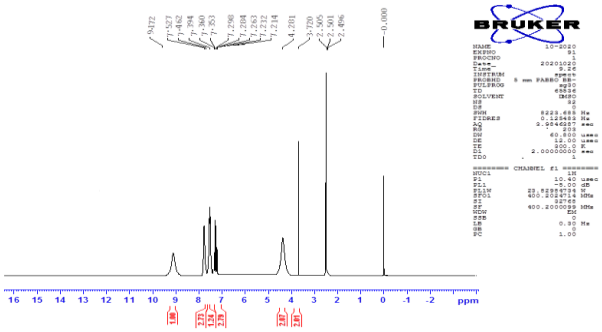


Figure 10. H^1-NMR spectrum of (sh_{14}) compound

Based on **Figure 10**, the H^1-NMR spectrum of compound (sh_{14}) [29] referred to the protons of ($-CH_2$), A signal at 4.2ppm that due to two protons of NH_2 group, Aromatic protons appear at (7.21-7.52) ppm peaks as a multiplate, a singlet signal appear at (9.17) ppm was due to one proton of (-NH-) group.
H^1-NMR spectrum of compound (sh_{16}) [30]in (DMSO as solvent) was pictured in **Figure 11**, A singlet signal appear at 3.7ppm could be attributed to protons of methylene group ($-CH_2$-). The signal at 3.8 ppm (2H) due to the protons of ($-CH_2$-) (4-oxothiazolidin) ring. The signal at 5.9 ppm which belong to (-CH-) of (4- oxothiazolidin) ring, another multiple signals at (7.1-7.4) ppm belongs to aromatic protons. Also, the spectrum showed a signal at 11.7 ppm could be attributed to protons of (-NH-) group.

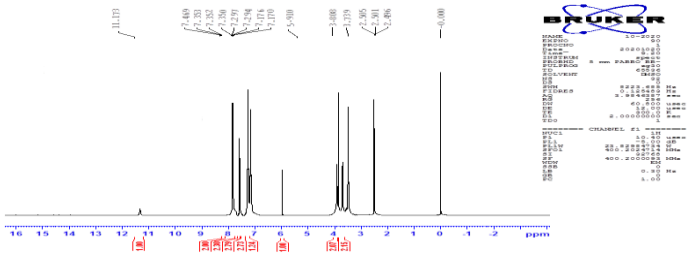


Figure 11. H^1-NMR spectrum of (sh_{16}) compound

4. Conclusion

New heterocyclic derivatives were successfully synthesized by treating Thiophenol with different organic reagents. The elemental and spectroscopy analysis of FTIR, H^1-NMR were in good agreement with proposed structure.

5. Conflicts of interest

"There are no conflicts to declare".

6. Formatting of funding sources

Self.

7. Acknowledgments

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