

## Synthesis and Characterization of New Heterocyclic Compounds from 2, 5- dimercapto -1, 3, 4-Thiadiazole and Their Resins

*Shetha F.Al-Zubiady\**

*Israa Abdul Satar Ismael\*\**

*Ban Dh. Ismael\**

\*Department of Chemistry, College of Science for Women, University of Baghdad, Baghdad, Iraq

\*\*Department of Materials Research-Ministry of Science and Technology, Baghdad, Iraq

Email: [israaldoori@yahoo.com](mailto:israaldoori@yahoo.com)

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### Abstract:

In this research, a new 1, 3, 4-Thiadiazole derivatives have been synthesized by many heterocyclic reactions. Starting from (2, 5 – dimercapto -1, 3, 4-Thiadiazole) a variety of derivatives have been synthesis. Compound (1) was synthesized by the reaction of hydrazine hydrate with carbon disulphide in absolute ethanol. The compound (1) was reacted with 1, 2-dibromoethane in presence of alkali ethanol to give the compound (2). The compound (3) was formed from the reaction of compound (2) with hydrazine hydrate. Schiff base (4) was obtained by reacting of compound (3) with the compound (p-hydroxybenzaldehyde) in absolute ethanol. A variety of phenolic Schiff base (Methylolic, Etheric, and Epoxy) derivatives have been synthesized. Methylolic derivative was synthesized by the reaction of Schiff base (4) with formaldehyde in tetrahydrofuran (THF). Etheric derivative was formed from the reaction of a methylolic Schiff base (5) with saturated alcohol (Methanol). Epoxy derivative was synthesized by the reaction of epichlorohydrine with etheric derivative (6). The last step of this research was the preparation of a composite material from mixing the Epoxy resin derivative(7), TiO<sub>2</sub> and morpholine via ring opening. All these derivatives were verified by using (FT-IR, UV) spectra photometer and <sup>1</sup>H-NMR spectra. Also, these derivatives were characterized using elemental analysis (C.H.N.S).

**Key words:** Synthesis, new heterocyclic compounds, 2, 5 – dimercapto -1, 3, 4-Thiadiazole, their resins

### Introduction:

1, 3, 4-Thiadiazole is one of the most important biochemical materials and it is one of the most popular isomers

in the pharmaceutical and the industrial fields. The studies have shown that heterogeneous quintet rings (thiadiazole) have different activities. The first one

who described 1, 3, 4-Thiadiazole was Fisher in 1882, but the real nature of the ring system described for the first time in 1890 by Freund and Kuh [1]. They found when a substitution occur in locations (2,5) of the Thiadiazole compound with two groups of thiol (SH) this will give a more power to the compound with a wide range of uses. 2,5- disubstituted-1, 3, 4-thiadiazole derivative has biological activities including anticancer [2] antifungal [3] anti-tubercular [4] and anti-inflammatory [5]. Schiff bases are used in a wide range of applications, in biological field [6], industrial applications such as anticorrosion material for steel and alloys, photo stabilizers for poly ethylene, and the methylolic derivatives of phenolic. Schiff bases are used in the production of industrial polymers, such as PVC [7]. Epoxy derivatives are poly ethers contain an epoxy groups at the end of the chain, known as oxiranes [8]. These resins undergo polymerization reactions through their ring opening and their participation in a process known industrially as "curing", using hardeners such as the amine compounds. Epoxy resins are used in adhesives, coatings and composite materials [9,10]. The main problem of the epoxy resin is with the passage of time suffered from break covalent bonds for epoxy resins, by sun light, therefore the addition of additives inorganic like TiO<sub>2</sub> and Zinc oxide to the epoxy derivative will absorb ultraviolet light in the range (300-400) nm strongly [11], leading to non-absorption of these rays by the epoxy molecules and active functional groups [12].

## Materials and Methods

Melting points were determined on electro thermal capillary apparatus and were uncorrected; FT-IR spectra were recorded using solid KBr disc by testing Shimadzu (FT-IR 8300). Ultra-violet spectra were recorded using

Shimadzu (UV-Visible) within range (200 -900) nm, the elements analysis of compounds (C.H.N.S) by using a Perkin-Elmer, RE 2400, measurement was made at College of Education for Pure Science (Ibn Al- Haitham), Baghdad University. <sup>1</sup>H-NMR spectra were carried out in Al-al Bayt University (Jordan) operating at 300 MHz in DMSO-d<sub>6</sub> as solvent with the TMS as internal standard. All the chemical materials and solvents were purchased from (Isotecinc, BDH and Merck) companies.

### Preparation of Compound (1) [13]: (2, 5-dimercapto-1, 3, 4-thiadiazole)

A mixture of (0.1 mol, 4.8 ml) hydr-azine hydrate (80%) with carbon disulfide (0.2 mol, 12ml) in absolute ethanol (20ml) was prepared. The mixture was refluxed with stirring for (4hrs) until the H<sub>2</sub>S emission stoppage. Then, the reaction mixture was cooled, filtered and recrystallized in distilled water.

### Preparation of Compound (2) [14]: (2,2-dimercapto-bis-(1,3,4-Thiadiazole)-5-disulfide Alkyl)

In absolute ethanol (0.7g) from sodium hydroxide was dissolved with stirring for (1hr). (0.011mol, 1.7g) of the compound (1) was dissolves in absolute ethanol (7.26ml). Then the first prepared solution was added to the second solution, the mixture was stirred for 10 minutes. Then (0.005 mol, 0.5ml) from 1, 2-dibromoethane was added slowly to this mixture and refluxed for (6hrs) and then the mixture was cooled at room temperature, filtered, and recrystallized in absolute ethanol.

### Preparation of Compound (3)[15]: (2, 2-dihydrazino-bis-1,3,4- thiadiazole-5-disulfide- ethane)

The compound (2) (0.01 mol, 3.2 g) was dissolved in absolute ethanol (24.5 ml) then (0.02 mol, 0.96ml) of hydrazine hydrate (80%) were added. The reaction mixture was refluxed for (6hrs), the emission of hydrogen sulfide

(H<sub>2</sub>S) was tested by lead paper, and then the mixture was cooled to room temperature, filtered, recrystallized from absolute ethanol.

#### Preparation of Compound (4) [16]:(Schiff Base)

The compound (2, 2-dihydrazino-bis-1, 3, 4-thiadiazole -5-disulfide ethane) (1mol) with (2mol) of the compound (p-hydroxybenzaldehyde) were mixed in absolute ethanol (40ml), refluxed for (8hrs) then cooled, filtered and recrystallized in absolute ethanol.

#### Preparation of Compound (5) (Methyloic Derivative) [17]:

Schiff Base (4) (5.3g, 0.01mol) and (1.2g, 0.04mol) of formaldehyde were mixed in THF (75ml), the reactants were mixed gently with 10% of alcoholic sodium hydroxide solution to keep the acidity of the solution at pH (9-10). The reaction mixture was refluxed at (50-60) °C for (3hrs) the mixture then neutralized with 10% of alcoholic phosphoric acid. The mixture was filtered and recrystallized from (THF).

#### Preparation of Saturated Etheric Derivative (6)[18]:

The saturated alcohol (Methanol) (0.8mol) with (1ml) of sulphuric acid were mixed at (5) °C, then (0.2 mol) of a Methyloic Schiff Base (5) was added gradually with stirring at room temperature for (1hr) with the rise of the temperature to the boiling point of the alcohol used. The reaction mixture was refluxed for (24hrs) then cooled, the mixture was neutralized with sodium hydroxide. The derivative was extracted using chloroform, recrystallized from chloroform, and then dried.

#### Preparation of Epoxy Derivative (7) [19]:

The epichlorohydrine (24.5 ml, 0.31 mol) was mixed with (7.2 ml, 0.12mol) of butanol and (0.01mole) of an etheric derivative, for 15 mints at (50) °C, then gently added (4g, 0.1mol) of sodium hydroxide in two stages with a maintained temperature below (65) °C

.The mixing continues for (2hrs). After the first addition the aqueous layer was separated from the organic layer, then a second batch of sodium hydroxide added and mixed for (1hr) the aqueous layer were separated while the organic layer dried and recrystallized from (THF).

#### Preparation of Resin (8) with Opening of the Ring [20]:

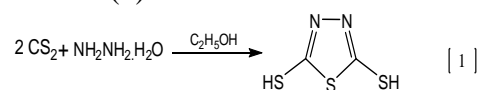
The epoxy derivative (0.01mol) was mixed with (20ml) of methanol, then (1.74ml, 0.02 mole) of morpholine was added, refluxed for (72hrs) then cooled, filtered and recrystallized from methanol

#### Preparation of Composite Material (9) from Epoxy Resin Derivative with Opening of the Ring [21]:

The Epoxy resin (0.01mole) was mixed with Methanol (20 ml), a weight ratios of 40%, 30%, 10% and 5% (of the resin weight used) of titanium dioxide (TiO<sub>2</sub>) pigment will be added, with a continuous stirring, then (1.74ml, 0.02 mole) of morpholine was added, refluxed for (72hrs) the product was painted on a glass slide of quartz and left to dry for (7-10) days.

## Results and Discussion

Compound (1) was prepared as shown in scheme (1).Some physical properties for this compound were listed in Table (1).



#### Scheme (1): Chemical equation for the preparation of derivative (1)

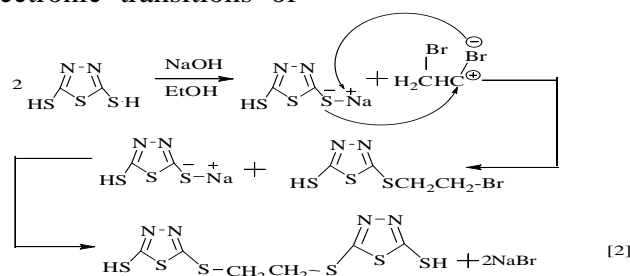
Compound (1) was characterized by its melting point and by (FT-IR, UV/Vis) spectrum. Melting point was recorded (159-161) °C. The FT-IR spectrum of compound (1), showed a medium intensity band at 1620 cm<sup>-1</sup> was due to ν (C=N). 1273 cm<sup>-1</sup> due to ν (C=S) and showed the stretching vibration (2580)

$\text{cm}^{-1}$  due to  $\nu$  (S-H thiol) and showed the absorption band at  $(3251) \text{ cm}^{-1}$ , due to  $\nu$  (NH, thion form). This means that compound (1) can exist in the thiol and thion form.

The (UV/Vis) spectrum of compound (1), Figure (6), showed the absorption band at  $331 \text{ nm}$  for  $(n-\pi^*)$  resulting from a nonbonding electronic transitions of

atoms heterogeneous (S, N) to thiadiazole ring and at  $236 \text{ nm}$  for  $(\pi-\pi^*)$  belonging to the (C=N, azomethine group).

Compound (2) was prepared as shown in scheme (2). Some physical properties for this compound were listed in the Table (1).

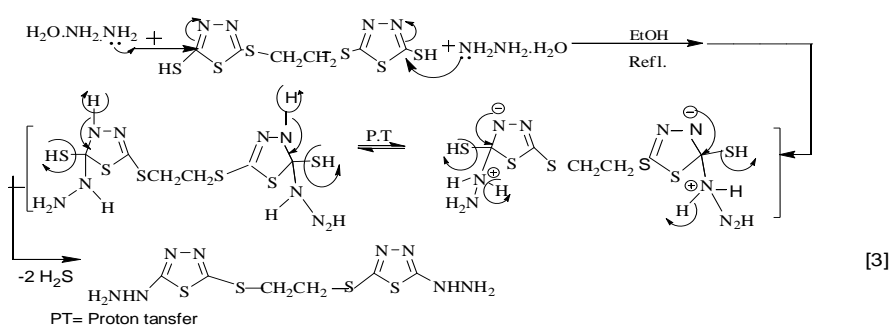


**Scheme (2): Mechanism steps for the preparation of derivative (2)**

Compound (2) was characterized by FT-IR spectroscopy; FT-IR spectrum of compound (2), Figure (1), shows the absorption band at  $(3205-3275) \text{ cm}^{-1}$ , due to  $\nu$  (NH, thion form) and  $\nu$  (S-H thiol) showed weak absorption band at  $(2569) \text{ cm}^{-1}$ . Bands at  $(2962-2924) \text{ cm}^{-1}$  were due to  $\nu$  ( $\text{CH}_2$ ) groups that mean compound (2) can exist in the thiol and

thion form. Band absorption at  $(1639) \text{ cm}^{-1}$  was due to  $\nu$  (C=N, azomethine group). Absorption band at  $(1141) \text{ cm}^{-1}$  was due to  $\nu$  (C=S).

Compound (3) was prepared as shown in scheme (3). Some physical properties for this compound were listed in the Table (1).

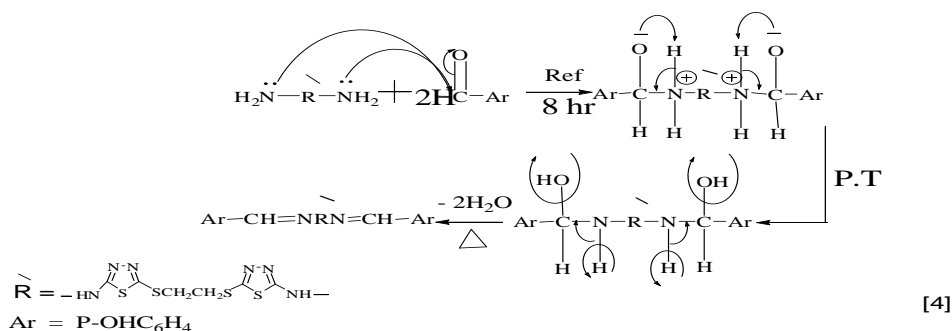


**Scheme (3): Mechanism steps for the preparation of derivative (3)**

Compound (3) was characterized by FT-IR spectroscopy; FT-IR spectrum shows the absorption band at  $(3209) \text{ cm}^{-1}$ , due to  $\nu$  (NH) and  $\nu$  ( $\text{NH}_2$ ) bands showed at  $(3227-3305) \text{ cm}^{-1}$ , bands at  $(2962) \text{ cm}^{-1}$  and  $(2824) \text{ cm}^{-1}$ , due to aliphatic  $\nu$  ( $\text{CH}_2$ ) group. Absorption band at  $(1643) \text{ cm}^{-1}$  was due to  $\nu$  (C=N) stretching vibration.  $^1\text{H-NMR}$  spectrum of compound (3), Figure (9), showed

characteristic signals at  $\delta = (0.9 - 1.55) \text{ ppm}$  due to (d,4H, (2 $\text{CH}_2$ )), singlet signal at  $\delta = (3.92-5.191) \text{ ppm}$  belongs to (s,4H,(2  $\text{NH}_2$ )), (8.6-9.4) ppm due to (s,2H,(2NH)) and at 2.50 ppm was due to the solvent DMSO- $d_6$ .

Compound (4) was prepared as shown in scheme (4). Some physical properties for this compound were listed in the Table (1).



P.T = Proton transfer .

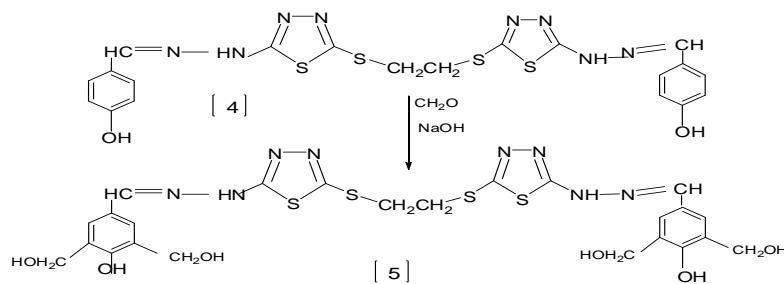
#### Scheme (4): Mechanism steps for the preparation of derivative (4)

Compound (4) was characterized by (FT-IR, UV) spectrum; The FT-IR spectrum of compound (4), Figure (2), showed the bands at  $(3286-3387) \text{ cm}^{-1}$ , due to  $\nu$  (NH) and  $\nu$  (OH) band showed at  $(3498-3444) \text{ cm}^{-1}$ , bands at  $(2969-2808) \text{ cm}^{-1}$ , due to aliphatic  $\nu$  ( $\text{CH}_2$ ) group. Absorption bands at  $(1670-1600) \text{ cm}^{-1}$  was due to  $\nu$  ( $\text{C}=\text{N}$ ) stretching and  $(1273-1249) \text{ cm}^{-1}$  absorption bands for phenolic group  $\nu$  ( $\text{C}-\text{O}$ ).

The (UV/Vis) spectrum of compound (4) Figure (7) showed the absorption band at  $(315\text{nm})$  for electronic transitions ( $n-\pi^*$ ) and at  $(226 \text{ nm})$  for transitions ( $\pi-\pi^*$ ).

$^1\text{H-NMR}$  spectrum of compound (4), Figure (10), showed characteristic signals at  $\delta = (0.9 - 1.6)$  ppm due to  $(d, 4\text{H}, (2\text{CH}_2))$ , singlet signal at  $\delta = (9.4 - 9.6)$  ppm due to  $(s, 2\text{H}, (2\text{NH}))$ , at  $\delta = (10.1 - 11.3)$  ppm for  $(s, 2\text{H}, (2\text{OH}))$ , at  $\delta = (6.78 - 8.190)$  ppm belongs to  $(m, 10\text{H}, 2(-\text{CH}-\text{C}_6\text{H}_4\text{OH}))$  and at  $2.50$  ppm was due to the solvent  $\text{DMSO}-d_6$ . Elemental analysis for compound (4)  $\text{C}_{20}\text{H}_{18}\text{N}_8\text{S}_4\text{O}_2$ : [C, 45.28(45.589); H, 3.341(4.251); N, 21.13(21.634); S, 24.15 (24.634)]

Compound (5) was prepared as shown in scheme (5). Some physical properties for this compound was listed in Table (1).

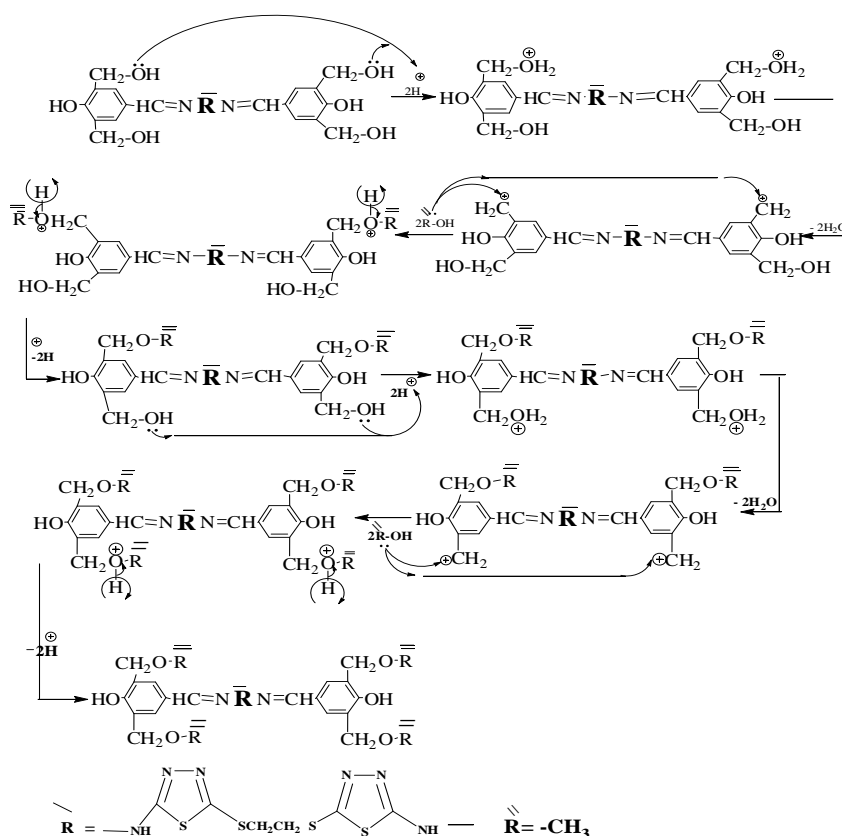


#### Scheme (5): Chemical equation for the preparation of derivative (5)

Compound (5) was characterized by (FT-IR) spectrum; The FT-IR spectrum of compound (5), showed the bands at  $(3271-3236) \text{ cm}^{-1}$ , due to  $\nu$  (NH) and  $\nu$  (OH) band showed at  $(3363) \text{ cm}^{-1}$ , bands at  $(2943-2931) \text{ cm}^{-1}$ , due to  $\nu$  ( $\text{CH}_2$ ) group. absorption band at  $(1604) \text{ cm}^{-1}$  was due to  $\nu$  ( $\text{C}=\text{N}$ ). Also other absorption band appeared at  $(1512) \text{ cm}^{-1}$ , was due to aromatic group  $\nu$  ( $\text{C}=\text{C}$ ).

Elemental analysis for compound (5)  $\text{C}_{24}\text{H}_{26}\text{N}_8\text{S}_4\text{O}_6$ : [C, 44.3 (44.5); H, 4.0(4.951); N, 17.2(17.8); S, 19.2 (19.5)].

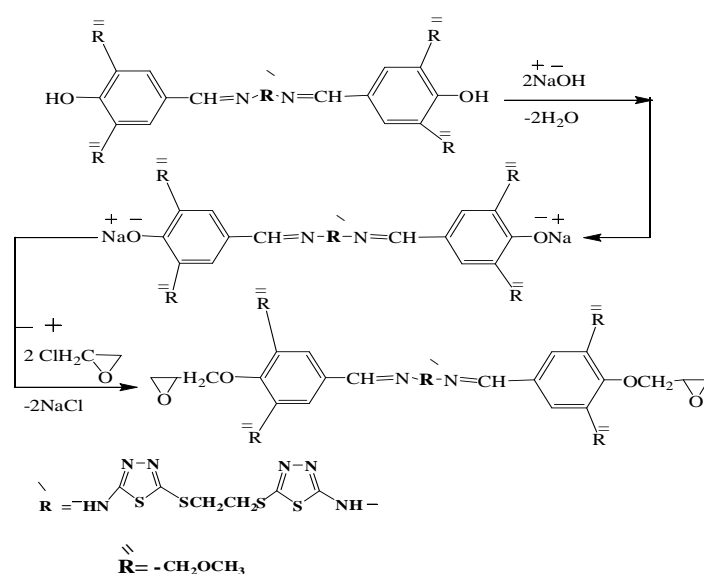
Compound (6) was prepared as shown in scheme (6). Some physical properties for this compound were listed in table (1). The suggested mechanism for this reaction is shown in the scheme (6)



**Scheme (6): Mechanism steps for the preparation of derivative (6)**

Compound (6) was characterized by (FT-IR) spectrum; The FT-IR spectrum of compound (6), the appearance of new absorption bands at (1165 -1122)  $\text{cm}^{-1}$  was due to  $\nu$  (C-O-C).

Compound (7) was prepared as shown in scheme (7). Some physical properties for this compound were listed in table (1).

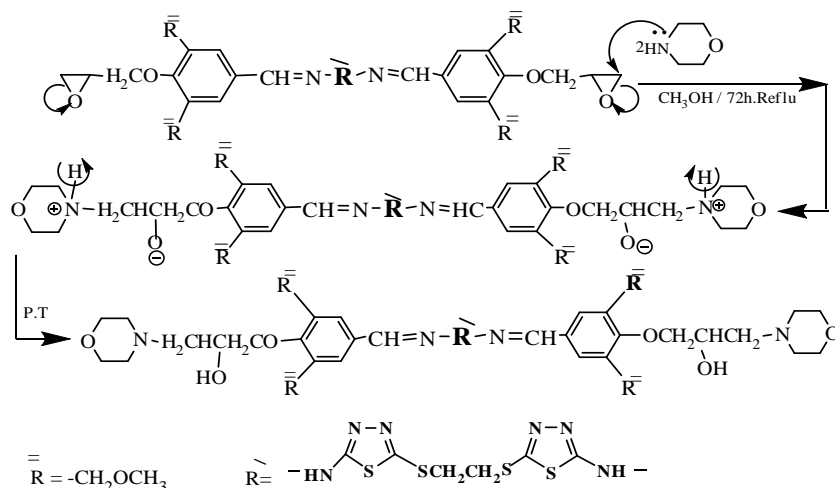


**Scheme (7): Mechanism steps for the preparation of derivative (7)**

Compound (7) was characterized by (FT-IR) spectrum; The FT-IR spectrum of compound (7), Figure (3), showed the absorption bands at  $(864-914)\text{cm}^{-1}$ , due to epoxy group, known as oxarine which attack on the phenolic  $\nu(\text{OH})$  group, absorption bands at  $(1114-1172)\text{cm}^{-1}$ ,

due to  $\nu(\text{C-O-C})$  and strong bands  $(2947-2870)\text{cm}^{-1}$  for  $\nu(\text{CH}_2)$ .

Compound (8) was prepared as shown in scheme (8). Some physical properties for this compound were listed in Table (1).

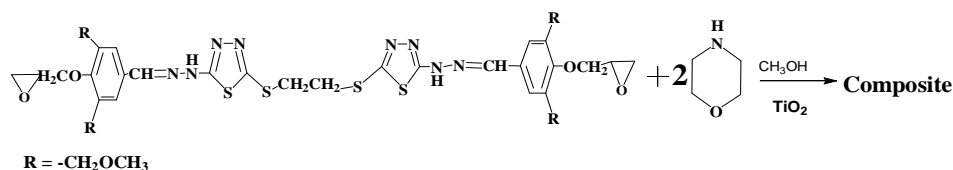


#### Scheme (8): Mechanism steps for the preparation of derivative (8)

Compound (8) was characterized by (FT-IR) spectrum; The FT-IR spectrum of compound (8), Figure(4), showed the absorption bands at  $(3398-3400)\text{cm}^{-1}$ , due to  $\nu(\text{OH})$  and  $\nu(1249)\text{cm}^{-1}$  for  $\nu(\text{C-O})$ . Absorption bands at  $(1134-1249)$

$\text{cm}^{-1}$ , due to  $\nu(\text{C-O-C})$  and strong bands  $(2943-2912)\text{cm}^{-1}$  for  $\nu(\text{CH}_2)$ .

Compound (9) was prepared as shown in scheme (9). Table (3) shows the physical properties of epoxy resin derivatives with  $(\text{TiO}_2)$  pigment with ring opening.



#### Scheme (9): Chemical equation for the preparation of derivative (9)

Compound (9) was characterized by (FT-IR) spectrum; The FT-IR spectrum of compound (9) Figure (5) showed the absorption band at  $(3390)\text{cm}^{-1}$  was due to  $\nu(\text{OH})$ ,  $(1114-11168)\text{cm}^{-1}$  was due to  $\nu(\text{C-O-C})$ , and strong bands  $(2924-2858)\text{cm}^{-1}$  for  $\nu(\text{CH}_2)$  showed characteristic FT-IR band at  $(1604)\text{cm}^{-1}$ , due to  $\nu(\text{C=N})$ .

By comparison between (FT-IR) spectrum of compound (9), Figure (5) with (FT-IR) spectrum of compound (8), Figure (4), showed the absorption similar bands which means that  $\text{TiO}_2$ , wasn't changed from the structural formula of the compound (8), as shown in table (2)

The (UV/Vis) spectrum of compound (9, 8), Figure (8), showed the absorption bands at [ (323nm, 322 nm, 321nm and 321nm) ] for electronic transitions ( $\pi - \pi^*$ ), also due to ratio [40% , 30% , 10% and 5% of TiO<sub>2</sub> and the highest value for the absorption was at (2.161),(1.878) due to ratio 40%,30% of TiO<sub>2</sub>. And appeared medium value for the absorption at (1.562),(1.358), due to ratio 10%, 5% of TiO<sub>2</sub>.while the (UV/Vis) spectrum of compound (8)Figure (8), showed the absorption band at (322nm) for electronic transitions ( $\pi - \pi^*$ ), and has been weakly


in value of the absorption, which recorded (1.023), due to ratio 0% of TiO<sub>2</sub>.

Through the (UV/Vis) spectrum of compound (9, 8), Figure (8) showed the absorbance different which means that TiO<sub>2</sub> improve of properties the epoxy resin through protecting it from UV ray, the addition of additives (inorganic like TiO<sub>2</sub>) to the epoxy resin will absorb ultraviolet light in the range (300-400) nm strongly [11], leading to non-absorption of these rays by the epoxy molecules and active functional groups, as shown in table (5).

**Table (1) Shows the physical properties of compounds (1-8) with formula.**

No.	Molecular Formula	M.P °C	Colour	Yield %	Purify-Solvent	No	Molecular Formula	M.P°C	Colour	Yield %	Purify-Solvent
1	C <sub>2</sub> H <sub>2</sub> N <sub>2</sub> S <sub>3</sub>	159-161	Light yellow	55	Distilled water	6	C <sub>28</sub> H <sub>34</sub> N <sub>8</sub> S <sub>4</sub> O <sub>6</sub>	Oily	Brown	55	THF
2	C <sub>6</sub> H <sub>6</sub> N <sub>4</sub> S <sub>6</sub>	200-202	White	50	Ethanol	7	C <sub>34</sub> H <sub>42</sub> N <sub>8</sub> S <sub>4</sub> O <sub>8</sub>	Oily	Brown-yellow	44	THF
3	C <sub>6</sub> H <sub>10</sub> N <sub>8</sub> S <sub>4</sub>	230-232	Grey	79	Ethanol	8	C <sub>42</sub> H <sub>44</sub> N <sub>8</sub> S <sub>4</sub> O <sub>10</sub>	Oily	Brown	60	Methanol
4	C <sub>20</sub> H <sub>18</sub> N <sub>8</sub> S <sub>4</sub> O <sub>2</sub>	155-157	Yellow	77	Ethanol						
5	C <sub>24</sub> H <sub>26</sub> N <sub>8</sub> S <sub>4</sub> O <sub>6</sub>	Oily	Dark red	52	THF						

**Table (2): (FT-IR) spectrum data of compounds (1-9)**

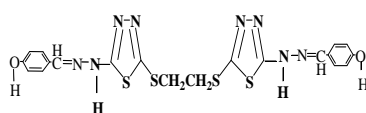
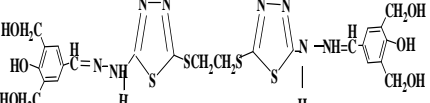
No.	$\nu$ OH	$\nu$ CH <sub>2</sub>	$\nu$ C=N	$\nu$ C-O-C	Others
1	-	-	1620	-	$\nu$ SH (2580) , $\nu$ C=S(1137) $\nu$ NH(3251) , $\nu$ C-S-C(698)
2	-	2962- 2924	1639	-	$\nu$ C=S(1141-1014), $\nu$ C-S-C(597)
3	-	2962-2927	1643	-	$\nu$ NH NH <sub>2</sub> (3209-3448), $\nu$ C-N (1358) , $\nu$ N-N (1431)
4	3498 3444	2989-2808	1600	-	$\nu$ C=C (1550) , $\nu$ C-O(1247) $\nu$ C-H aromatic (3100) , $\nu$ NH (3387-3286)
5	3363	2943-2931	1604	-	$\nu$ C=C (1512) , $\nu$ C-O(1253)
6	3460	2939-2930	1604-1543	1165-1122	$\nu$ C-H aromatic (3020) $\nu$ N-N (1442), $\nu$ C-O (1234)
7	3437 3417	2947-2870	1604-1658	1114-1172	$\nu$ C-H out of plane (659-756) ,  (864-914)
8	3398 3400	2943-2912	1604	1134-1249	$\nu$ C-H out of plane (732), $\nu$ C-N(1354) $\nu$ C-O (1249)
9	3390	2927-2858	1604	1138-1114	$\nu$ C=C (1508) , $\nu$ C-O(1253) , $\nu$ N-N (1454)

**Table (3) Shows the physical properties of compound (9) with formula.**

Comp. No.	Molecular Formula	TiO <sub>2</sub> %	Colour	The mixing ratio	A gloss degree at an angle 60 <sup>0</sup>	Plating thickness
9	C <sub>42</sub> H <sub>44</sub> N <sub>8</sub> S <sub>4</sub> O <sub>10</sub>	(40%) (30%) (10%) (5%)	Yellowish white	4:1	1.2 <sup>0</sup> 1.3 <sup>0</sup> 1.7 <sup>0</sup> 1.9 <sup>0</sup>	100-500 Micron

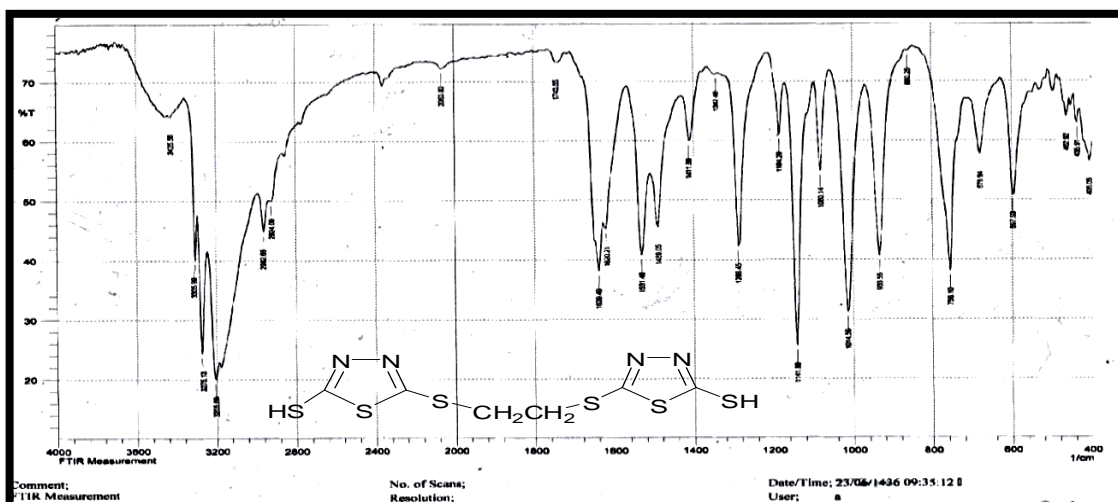


**Table (4): Shows the elemental analysis (C.H.N.S) of the derivatives (4, 5)**

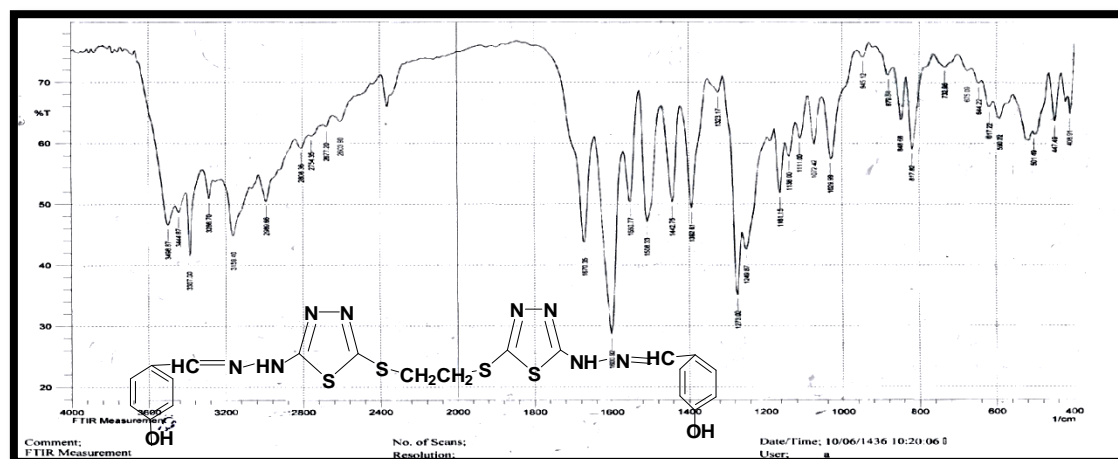
Comp. No	Structure Formula	(C.H.N.S) Analysis % calculated (Found)			
		% C	% H	% N	% S
4		45.28 (45.589)	3.341 (4.251)	21.13 (21.634)	24.15 (24.634)
5		44.3(44.51)	4.0(4.951)	17.2(17.8)	19.2(19.5)

**Table (5): (UV/ Vis) spectrum data of the compounds (9,8)**

Comp. No.	Molecular Formula	TiO2 %	(UV/ Vis) spectrum			
			λ max nm	Abs	λ max nm	Transmittance (T %)
8	C <sub>42</sub> H <sub>44</sub> N <sub>8</sub> S <sub>4</sub> O <sub>10</sub>	B1 (0%)	322	1.023	322	2.250
9	C <sub>42</sub> H <sub>44</sub> N <sub>8</sub> S <sub>4</sub> O <sub>10</sub>	B2 (5%)	322	1.358	323	1.558
		B3 (10%)	323	1.562	323	1.0
		B4 (30%)	323	1.878	323	0.6
		B5 (40%)	323	2.161	323	0.42



**Fig.(1): FT-IR spectrum of compound (2)**



**Fig. (2): FT-IR spectrum of compound (4)**

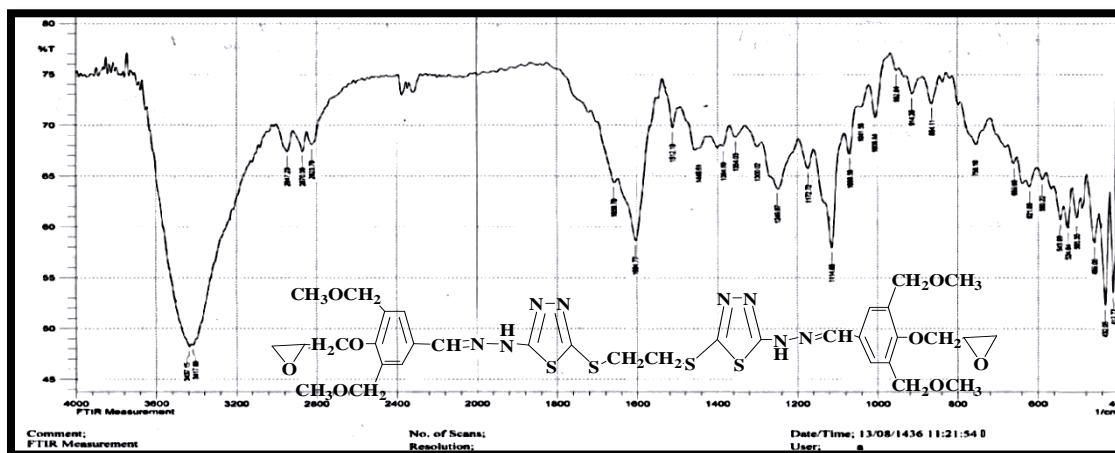


Fig. (3): FT-IR spectrum of compound (7)

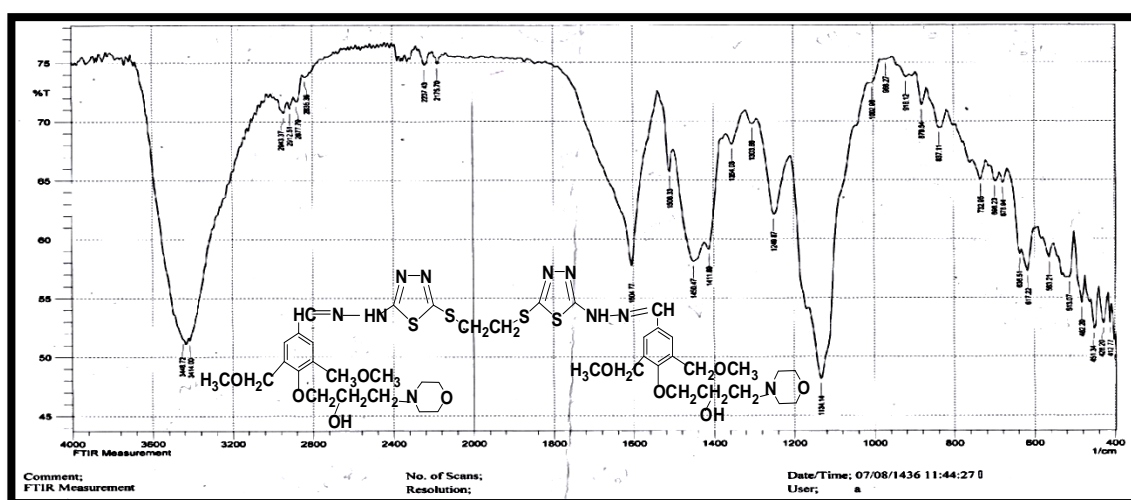


Fig. (4): FT-IR spectrum of compound (8)

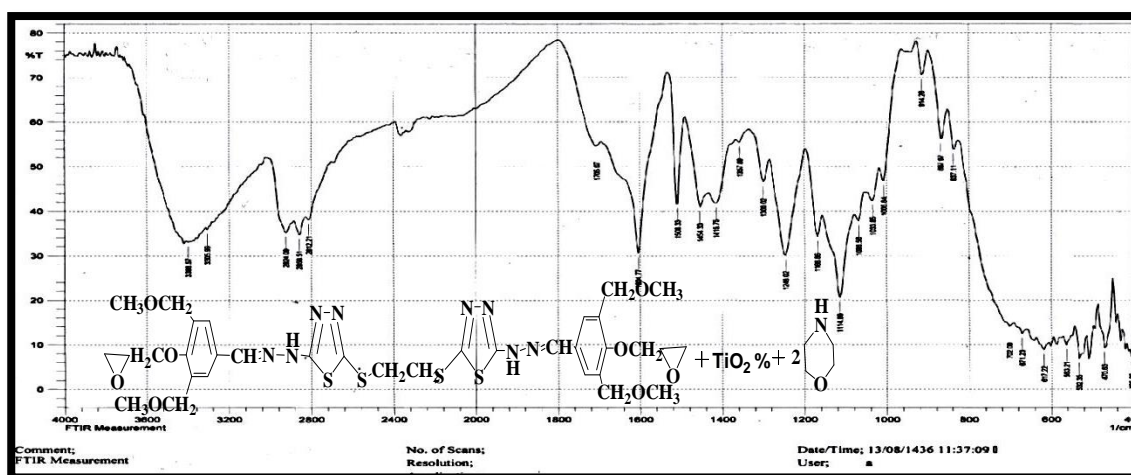


Fig. (5): FT-IR spectrum of compound (9)

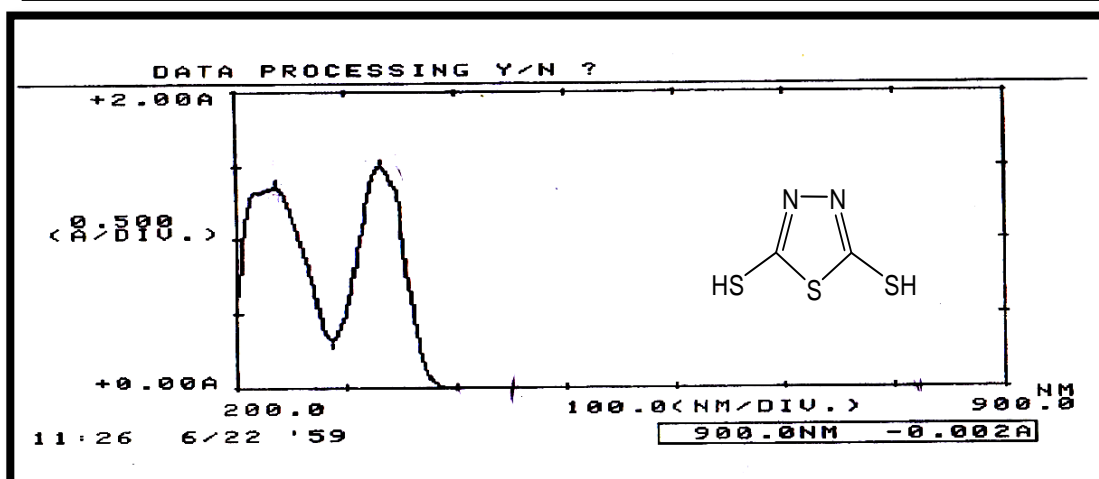


Fig. (6): UV spectrum of compound (1)

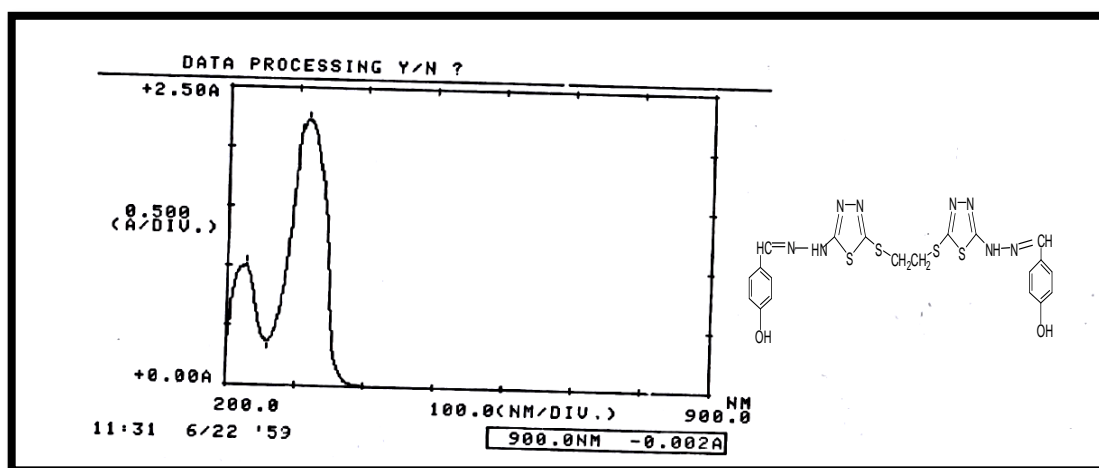


Fig. (7): UV spectrum of compound (4)

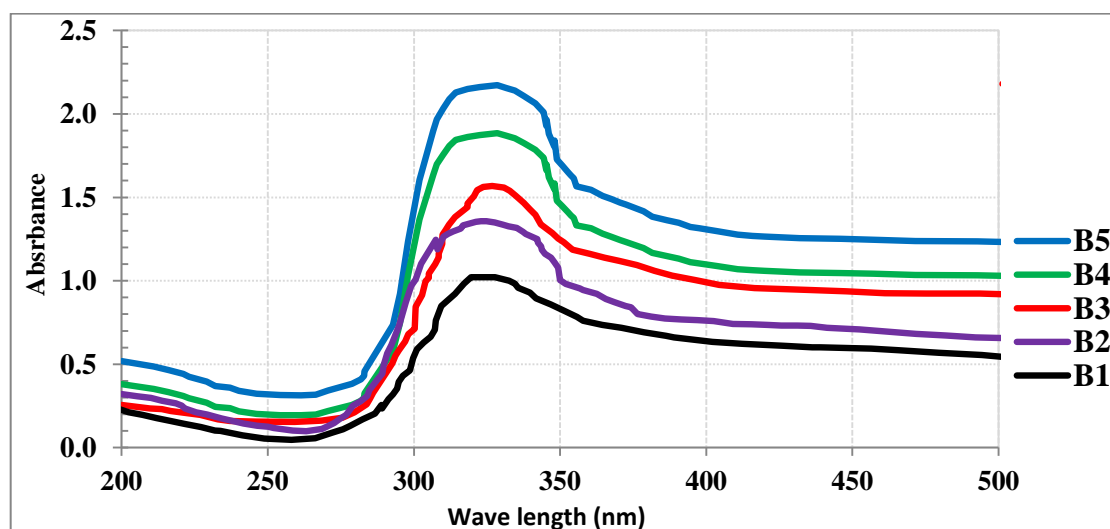
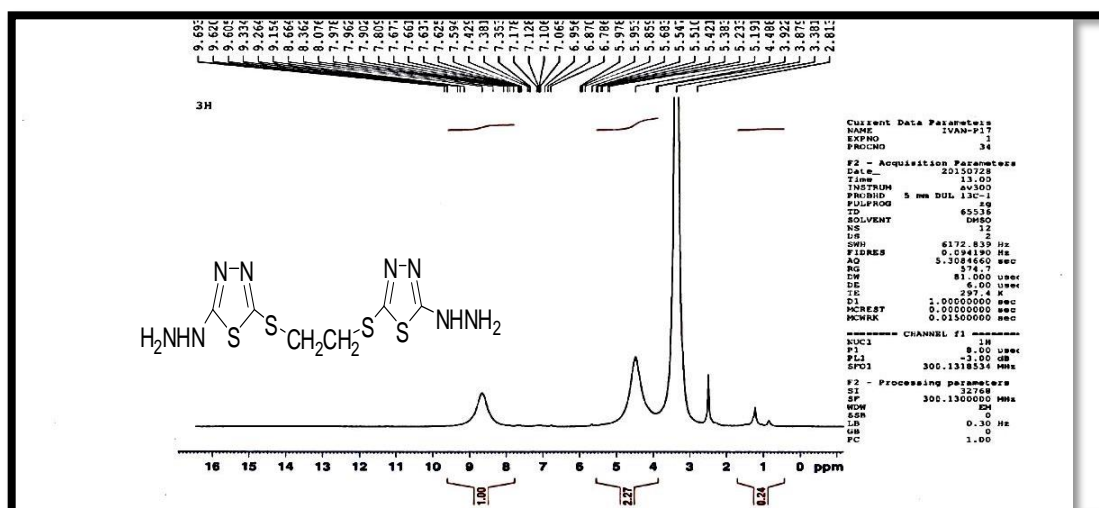
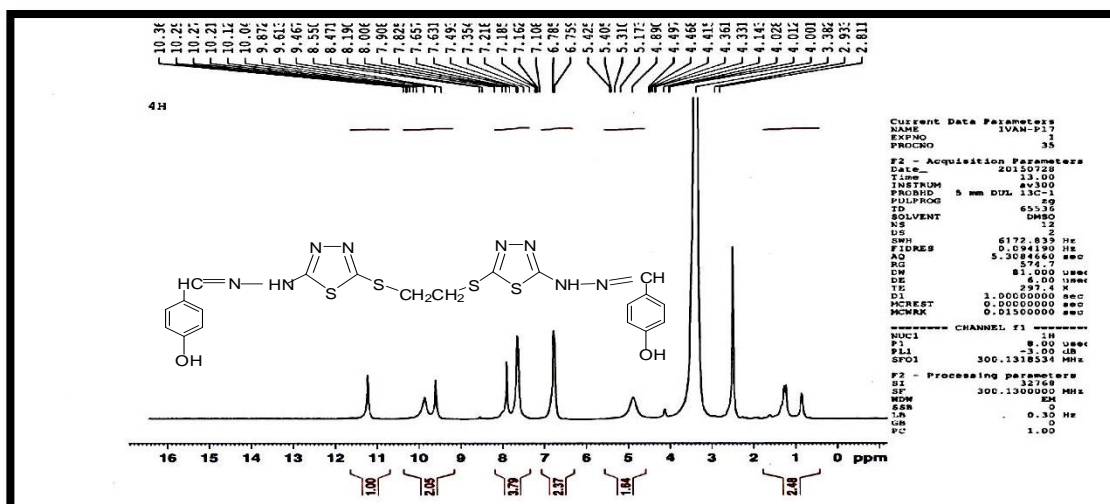


Fig. (8): shows the variation of Absorbance of the compound (9, 8)

Fig. (9): <sup>1</sup>H-NMR spectrum of compound (3)Fig. (10): <sup>1</sup>H-NMR spectrum of compound (4)

### Conclusion:

During the preparation of the new derivatives from the basic component, (2, 5-dimercapto-1, 3, 4- thiadiazole), the epoxy and methylol derivatives have been prepared, and these have great importance because they are used, widely, as industrial applications especially paints and glue industry.

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## تحضير وتشخيص مركبات حلقية غير متجانسة جديدة من 5،2- ثنائي مركبتو - 1، 3، 4 - ثيادايازول وراتنجاتها

اسراء عبد الستار اسماعيل\*\*

شذى فاضل الزبيدي\*\*

بان ذنون اسماعيل\*\*

\*قسم الكيمياء، كلية العلوم للبنات، جامعه بغداد، بغداد، العراق  
\*\* دائره بحوث المواد، وزاره العلوم والتكنولوجيا، بغداد، العراق

### الخلاصه :

في هذا البحث تم تحضير مشتقات جديده بواسطه العديد من التفاعلات الحلقية الغير متجانسه وبدايه من مركب الاساس (5,2- ثنائي مركبتو- 4,3,1- ثيادايازول .حضرت العديد من المشتقات . فالمركب (1) قد حضر بواسطه تفاعل الهيدرازين المائي (80%) مع ثاني كبريتيد الكربون ويتم مفاعله مع ثنائي بروموايثان بوجود القاعده الكحوليه ليعطي المركب (2). اما المركب (3) تكون من تفاعل المركب (2) مع الهيدرازين المائي . قاعده شف (4) حضرت بواسطه تفاعل المركب (3) مع بارا هايدروكسي بنزالديهايد بوجود الايثانول. تم تحضير العديد من المشتقات (الفينولية، الايتريه، الايبوكسيه ) من قاعده شف الفينولية، وكانت اخر خطوه في هذا البحث هي تحضير ماده المركبه من مزج مشتق راتنج الايبوكسي (7) والصبغه اللاعضويه (TiO<sub>2</sub>) مع المورفلين وبوجود الميثانول .شخصت هذه المشتقات بواسطه استعمال الطرائق الطيفيه وهي FT-IR ( <sup>1</sup>H-NMR،UV/Vis)، وتم قياس التحليل الكمي الدقيق للعناصر ( C.H.N .S )

الكلمات المفتاحيه : تحضير ، مركبات حلقية جديده ، 5,2- ثنائي مركبتو- 4,3,1- ثيادايازول ، راتنجاتها