

## THE PHARMA INNOVATION

# Synthesis and Characterization of New Cycles of Selenazane and Thiazane

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The aim of this paper ,cyclic compounds from selenium and sulphur were prepared via condensation reaction of 4-methyl benzaldehyde with 4-toluidine or p-phenylene diamine to produce anil compound ,which reacts with seleno or thio-organic compounds to yield selenazane and thiazine [1-6].The synthesized compounds [1-6] have been investigated using several chemical techniques ,such as (H.NMR-spectra , FT.IR –spectra , (C.H.N) –analysis and melting points).

*Keyword:* Condensation of Anil Compounds, Seleno Cycles, Sulphur Cycles.

**INTRODUCTION:** Organosulphur and selenium chemistry has played a prominent role in our laboratory, both in the development of new synthetic methodology and in the design of biologically interesting compounds containing these elements<sup>(1-5)</sup>.They are class of compounds well known for a long time, and still continue the object of considerable interest , mainly due<sup>(6-14)</sup> to their applications in different fields such as:



In this paper set out to synthesize a new group of seleno and sulphur –hetero cycles namely selenazane and thiazane compounds [1-6] through a cyclo addition between the anile group and selenium compounds or sulphur compounds in the following ,scheme<sup>(1)</sup>.

Experimental: All chemical used where supplied from Merck & BDH-chemical company. And all measurement where carried out by :

- Melting points :Electro thermal 9300 , melting point Engineering LTD ,U.K .
- FT.IR-spectra: Fourier transform infrared shimadzu (8300) , (FT.IR) , KBr-disc was performed by CO.S.Q. Iraq .
- H.NMR-Spectra &( C.H.N)-Analysis : in Jordan .

### Synthesis of bis (4-methyl phenyl) imine[1]:

Condensation reaction<sup>(23,24)</sup> by refluxing ethanolic mixture of equimolar amounts (0.01 mole,1.2g) of 4-methyl benzaldehyde and (0.01 mole ,1.07g) of 4-toluidine were reacts for (2 hrs) , the precipitate was filtered and recrystallized to give %85 of compound<sup>[1]</sup> .

**2-(4-methyl phenyl)-3-(4-methyl phenyl)-1,3-selenazane-4-one[2]:**

Refluxing mixture of bis (4-methyl phenyl) imine[1] (0.01 mole ,2.09g) with (0.01 mole,1.9g of sodium selenide ethyl chloride) , was refluxed for (4 hrs) , the precipitate was filtered and recrystallized to pduce 87% of compound [2] .

**2-(4-methyl phenyl)-3-(4-methyl phenyl)-1,3-thiazane-4-one [3] :**

Refluxing mixture of bis(4-methyl phenyl) imine[1] (0.01 mole ,2.09g) with (0.01mole ,1.24g of 3-mercapto propoyl chloride) , was refluxed for (4 hrs) , the precipitate was filtered and recrystallized to pduce 84% of compound [3].

**Synthesis of bis (4-methyl phenyl) -4-phenylene di imine [4] :**

(0.01mole ,1.08g)of P-phenylene diamine was condensed with (0.02 mole,2.4g)of P-methyl benzaldehyde in presence of absolute ethanol(100 ml),the precipitate was filtered and recrystallized to give 85% of compound [4] .

**1,4-bis(2-(4-methyl phenyl)-1,3-selenazane-4-one benzene [5] :**

Refluxing mixture of bis (4-toluidine) -4-phenylene diamine [4] (0.01 mole,3.1g) with (0.02 mole,3.8g of sodium selenide propoyl chloride) were refluxed for (5 hrs) ,after cooling , the precipitate was filtered and recrystallized to yield 86% of compounds [5] .

**1,4-bis(2-(4-methyl phenyl)-1,3-thiazane-4-one)-benzene [6]:**

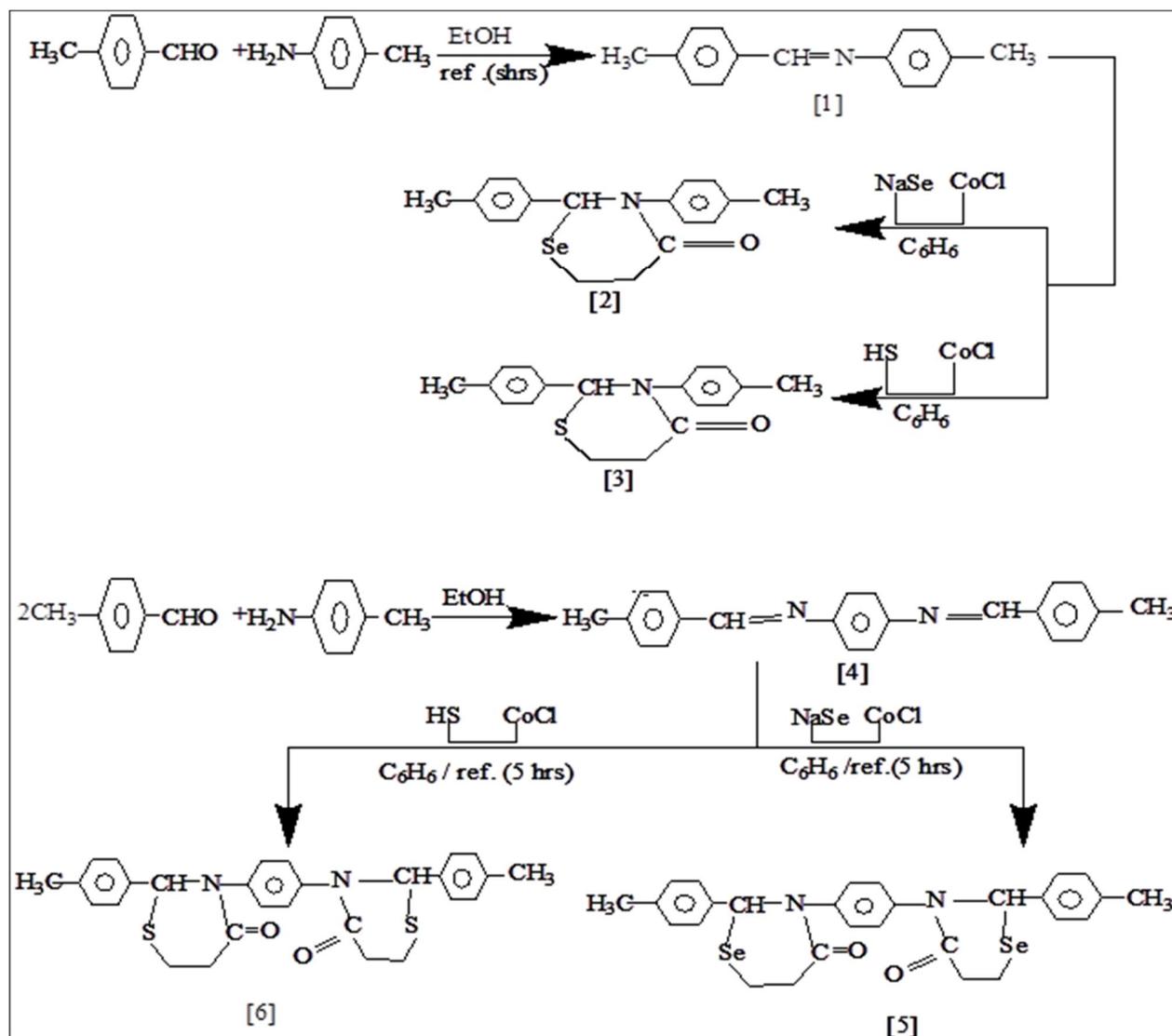
Refluxing mixture of bis(4-methyl phenyl)-4-phenylene di imine [4] (0.01 mole,3.1g) with (0.02 mole ,2.4 g of 3-mercapto propoyl chloride) were refluxed for (5 hrs) ,after cooling , the precipitate was filtered and recrystallized to yield 84% of compounds [6] .

**Table (1) : FT.IR data(cm<sup>-1</sup>) of compounds [1-6]**

| Comp. No. | (CH=N) of anile group | carbonyl of amide | (CH-Se) Endocycle | (CH-S) Endocycle |
|-----------|-----------------------|-------------------|-------------------|------------------|
| [1]       | 1620 S                | -----             | -----             | -----            |
| [2]       | -----                 | 1710 S            | 1655 S            | -----            |
| [3]       | -----                 | 1708 S            | -----             | 1660 S           |
| [4]       | 1618 b                | -----             | -----             | -----            |
| [5]       | -----                 | 1696 S            | 1686 S            | -----            |
| [6]       | -----                 | 1705 S            | -----             | 1645 S           |

**Table (2): Melting points , MF , & Elemental analysis of compound [1-6]**

| Comp. No. | M.F   | m.p C° | Calc./ Found. C% | H %            | N %            |
|-----------|---|--------|------------------|----------------|----------------|
| [1]       | C <sub>15</sub> H <sub>15</sub> N   | 105    | 86.124<br>86.006 | 7.177<br>7.024 | 6.698<br>6.437 |
| [2]       | C <sub>18</sub> H <sub>19</sub> NOSe  | 158    | 62.797<br>62.544 | 5.523<br>5.308 | 4.070<br>3.910 |
| [3]       | C <sub>18</sub> H <sub>19</sub> NOS   | 164    | 72.727<br>72.677 | 6.397<br>6.206 | 4.713<br>4.587 |
| [4]       | C <sub>22</sub> H <sub>20</sub> N <sub>2</sub>                                | 122    | 84.615<br>84.465 | 6.410<br>6.278 | 8.974<br>8.815 |
| [5]       | C <sub>28</sub> H <sub>28</sub> N <sub>2</sub> O <sub>2</sub> Se <sub>2</sub> | 188    | 57.739<br>57.507 | 4.811<br>4.645 | 4.811<br>4.734 |
| [6]       | C <sub>28</sub> H <sub>28</sub> N <sub>2</sub> O <sub>2</sub> S <sub>2</sub>  | 176    | 68.852<br>68.764 | 5.737<br>5.617 | 5.737<br>5.568 |



Synthesis scheme (1)

## RESULTS AND DISCUSSION :

These compounds [1-6] are saturated six-membered cycles which are important building blocks in various biologically active compounds, compose the core structures of some pharmaceutical drugs and in other fields<sup>(15-22)</sup>. for this reason, several synthetic methods for selenium and sulphur cyclic<sup>(19)</sup> compounds have

been developed which will also open new ways in organic synthesis chemistry. All the synthesized compounds<sup>[1-6]</sup> have been characterized by their melting points and spectroscopic methods (FT.IR-spectra, (C.H.N)-analysis, and H.NMR-spectra):

In FT.IR spectra, the reaction is followed by appearance (CH=N) absorption band of anil group<sup>(23,24)</sup> at  $1620\text{cm}^{-1}$  in compound [1], while

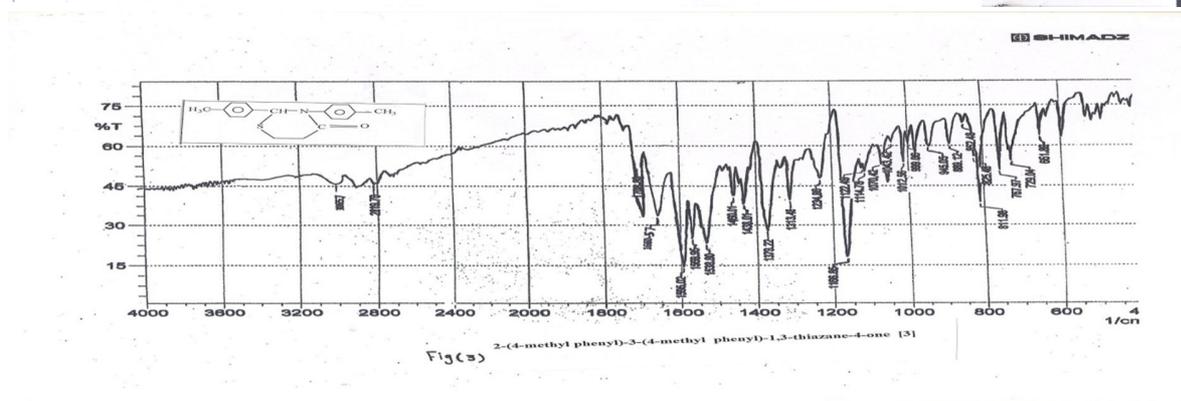
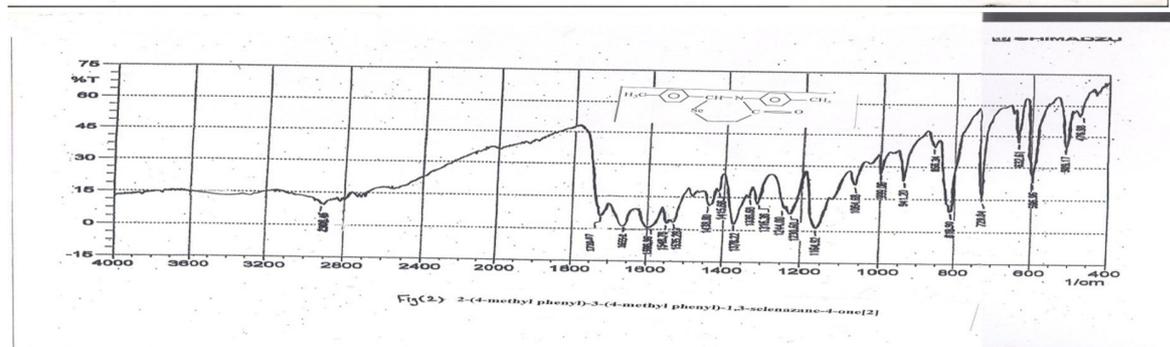
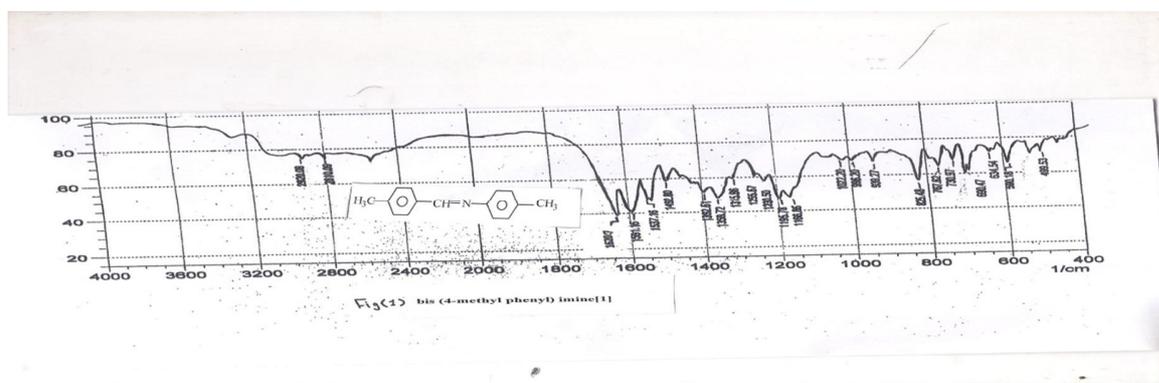
this band disappears and other bands are appear at (1710,1655) $\text{cm}^{-1}$  due to carbonyl group of amide (  $\text{—}\overset{\text{O}}{\parallel}\text{C—NH}$  ) and (CH-Se) respectively in compound [2] and at (1708,1660) $\text{cm}^{-1}$  due to carbonyl group of amide (  $\text{—}\overset{\text{O}}{\parallel}\text{C—NH}$  ) and (CH-S)<sup>(19)</sup> respectively in compound [3] .

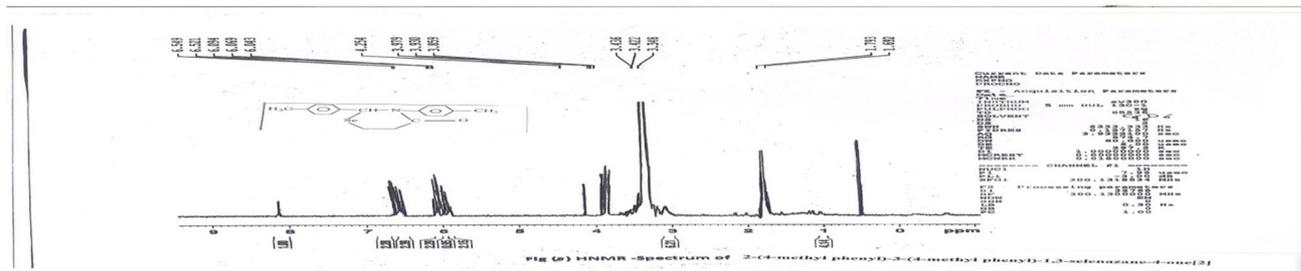
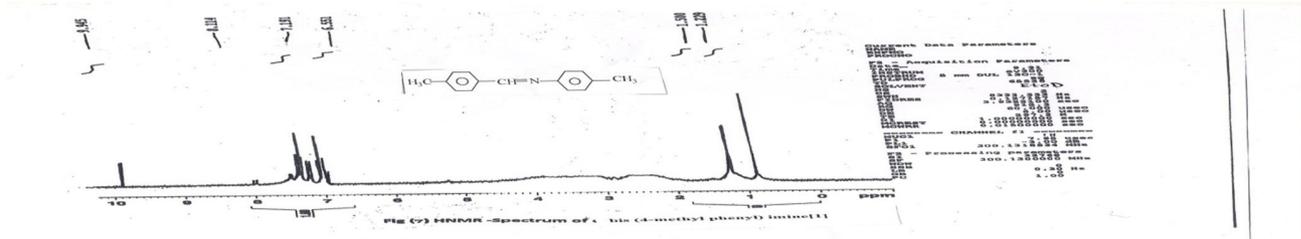
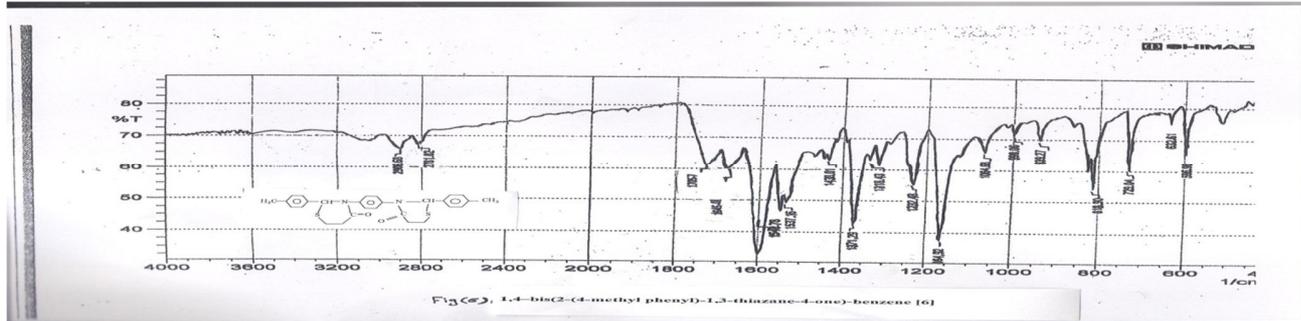
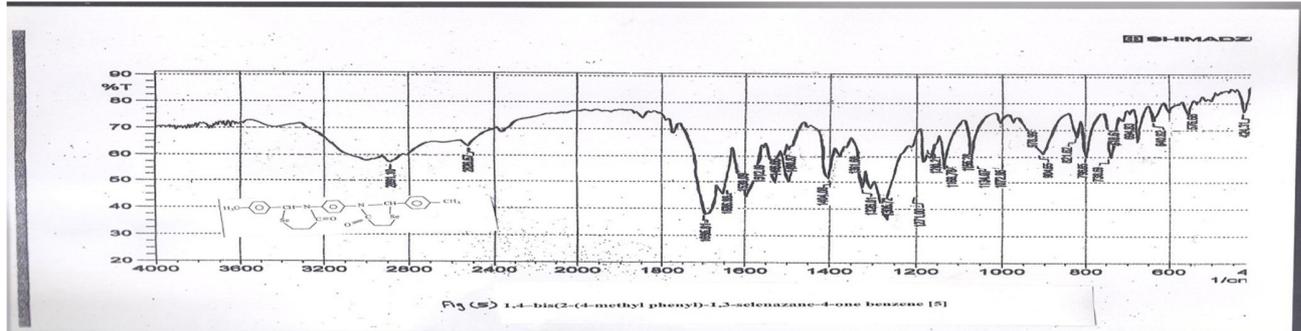
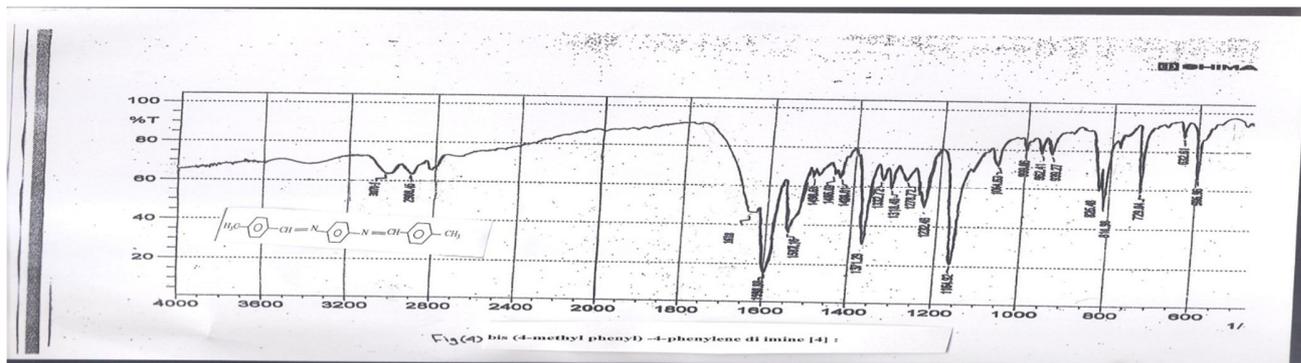
FT.IR –showed appearance of broad band at (1618)  $\text{cm}^{-1}$  due to anil group (CH=N)<sup>(17,18)</sup> of compound [4] ,while this band disappears and other bands are appear at (1696,1686) $\text{cm}^{-1}$  due to

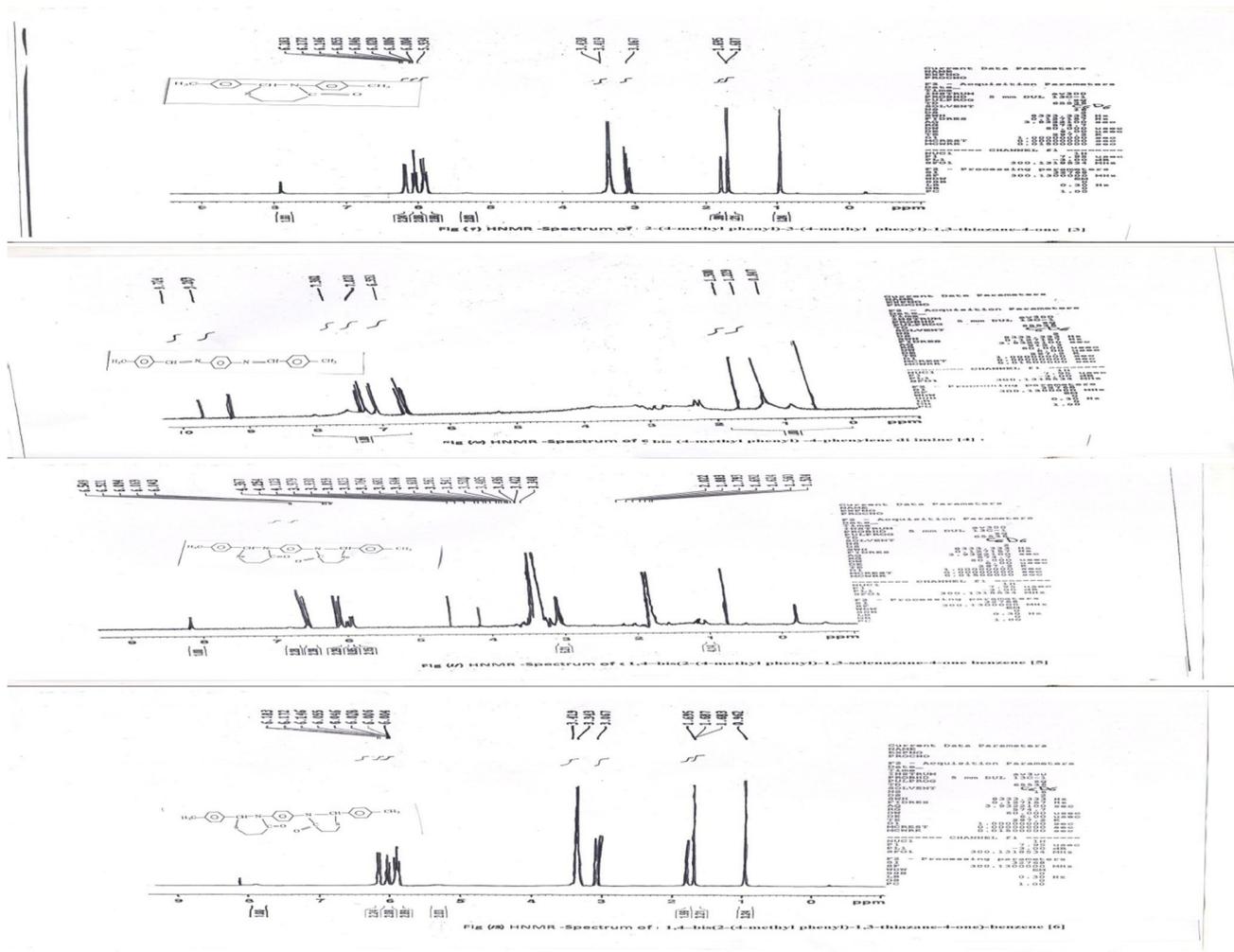
carbonyl group of amide (  $\text{—}\overset{\text{O}}{\parallel}\text{C—NH}$  ) and(CH-Se) respectively in compound [5] ,and at (1705,1645)  $\text{cm}^{-1}$  due to carbonyl group of amide (  $\text{—}\overset{\text{O}}{\parallel}\text{C—NH}$  ) and (CH-S) endo cyclic<sup>(19)</sup> , respectively in compound [6] .

Appearance of these bands strong evidence to formation of compounds

[1-6] , other data of functional groups shown in the following , table(1) and figures (1-6)







H.NMR-spectrum of compounds [1-6] showed :

- Singlet signal at  $\delta$ 9.94 for one proton of anil groups (-CH=N) , singlet signal at  $\delta$  1.5 of proton of methyl group (- CH<sub>3</sub>) and doublet of doublet signals<sup>(18)</sup> at  $\delta$  6.55 for protons of phenyl group in compound [1] .
- Singlet signal at  $\delta$  4.25 for one proton of (Se-CH-N) endo cyclic ,signal at  $\delta$  3.97 for protons of (Se-CH<sub>2</sub>-CH<sub>2</sub>) end o cyclic ,singlet signal at  $\delta$  1.69 for protons of methyl group (- CH<sub>3</sub>) and doublet of doublet signals at  $\delta$  6.54 in compound [2].
- Singlet signal at  $\delta$  3.43 for one proton of (S-CH-N) endo cyclic , signal at  $\delta$  3.06 for protons of (S-CH<sub>2</sub>CH<sub>2</sub>) endo cyclic<sup>(19)</sup> in compound [3].
- Singlet of two signals at  $\delta$  9.72 ,  $\delta$  9.26 for proton of two anil groups<sup>(23,24)</sup> (CH=N) in compound [4].
- Singlet of two signals at  $\delta$  4.36,  $\delta$  4.12 for proton of two groups of (Se-CH-N) endo cyclic and signals at  $\delta$  3.85 ,  $\delta$  3.76 for protons of two groups of (Se-CH<sub>2</sub>CH<sub>2</sub>) endo cyclic in compound [5] .
- Singlet of two signals at  $\delta$  3.41 , 3.34 for proton of two groups of (S-CH-N) endo

cyclic and signals at  $\delta$  3.06 for protons of two groups of (S-CH<sub>2</sub>CH<sub>2</sub>) endo cyclic<sup>(19)</sup> in compound [6].

And other peaks showed in the following, figures (7-12).

(C.H.N)-analysis: It was found from compared the calculated data with found data of these compounds. The results were compactable; the data of analysis, M.F, and melting points are listed in table (2)

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