

## Synthesis of New Pyrazoline- Phenoxathiin Derivatives

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### Abstract:

Phenoxathiin was prepared by the reaction of diphenyl ether with sulfur in the presence of anhydrous aluminum chloride. This work comprised the synthesis of new phenoxathiin derivatives containing heterocyclic moieties. These heterocyclic compounds were synthesized in three groups. The first group was made up of 2-(oxoalken-1-yl) phenoxathiin derivatives (**3a-3j**) obtained from the reaction of 2-acetylphenoxathiin with different aromatic aldehyde in the presence of sodium hydroxide. The other two groups involved compounds produced from the reaction of (**3a-3j**) with hydrazine hydrate in acetic acid to get 2-(1-acetylpyrazolin-3-yl) phenoxathiin derivatives (**4a-4j**), and phenyl hydrazine in the presence of piperidine to afford 2-(1-phenylpyrazolin-3-yl) phenoxathiin derivatives (**5a-5j**). All these compounds of two groups above were substituted in position (5) in pyrazoline ring with different aryl groups according to aromatic aldehyde used in the preparation of the first group series compounds.

**Keywords:** Phenoxathiin, Oxoalken derivatives, Pyrazoline.

### Introduction:

Phenoxathiin is given as the preferred name by Patterson and Capell [1-3]. Most widely method of preparation of phenoxathiin has been used alkyl phenoxathiin oxides by Ferrario [4-10] and dioxides of cycloalkylphenoxathiins and their halogen derivatives have been recommended as modifiers in plastic materials, intermediates antioxidants and as rubber and gum inhibitors [11-13]. Several of crystalline phenoxathiin cation radicals have allowed us to be studying the chemistry of the cation radical in homogenous solution. Several of phenoxathiin compounds are reddish

brown dyes on cotton [14]. Alkylated phenoxathiin has excellent oxidative stability and excellent anti-wear properties, so they are beneficial as lubricant additives, lubricant base stocks, or intermediates to lubricant base stock to improve viscosity and wear properties [15]. In biological field, they are used in many drugs as bacteriostatic, fungicides, anthelmintic, insecticides and antiviral agents such as distemper virus, influenza virus, hepatitis virus, neurotropic virus and especially influenza and herpetic viruses [16]. The phenoxathiin and its derivatives are used subunits to prepared different compounds exemplified dihydroazulenes (DHAS) [17].

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Polyimides were readily prepared by the polycondensations of phenoxathiindiamines with aromatic diacyl chlorides and aromatic diamines with new phenoxathiindiacyl chlorides[18]. The acetyl derivatives are obtained by the action of acetyl chloride on the amino acid. Phenoxathiin derivatives have recently gained attention owing to their fluorescent properties [19,20]. Organometallic derivatives of phenoxathiin have been prepared with different elements such as lithium and silicon [21-31].

### Materials and Methods:

FT-IR spectra were recorded on [SHIMADZU] FT-IR 8400s spectrophotometer; Solid samples were run in KBr disc, Liquid were run as smears. UV spectra were recorded on UV-Visible Spectrophotometer [SHIMADZU] UV-160A. <sup>1</sup>H-NMR spectra were recorded on ultra shield 300 MHz with tetramethylsilane as internal standard. Melting points were determined in a [GallenKamp] melting point apparatus with sample contained in open capillary glass tube in an electrically heated metal block apparatus. Thin Layer Chromatography[TLC] was performed on pre-coated plastic sheet with 0.25 mm layer of silica-gel F254. Spots were detected with iodine vapour.

### General procedure for synthesis of phenoxathiin and its

#### derivatives phenoxathiin(1)

A mixture of 188.6 g. (1.1 mol) of phenyl ether, 25.6 g. of sulfur (flowers) and 51.0g. (0.38 mol) of anhydrous aluminum chloride,

maintained on steam bath for 4 hrs. The reaction mixture was poured slowly, with stirring, into ice bath to which (25 ml.) of concentrated hydrochloric acid was added. After the two layers were separated the water layer was discarded and the (phenyl ether-phenoxathiin) layer dried overnight with calcium chloride, this mixture was distilled at (5 mm.) pressure from a 500-ml special Claisen flask. After removal of the phenyl ether the fraction boiling at (140-160°C / 5mm.), phenoxathiin was collected at (150-152)°C. The product was crystallized from methyl alcohol, m.p.(56-57)°C, yield 80%. 2-

#### acetylphenoxathiin(2)

A mixture of (22.9 g, 0.114 mol) phenoxathiin, (9.7 g, 0.155 mol, 8.8 ml) acetyl chloride and carbon disulphide (120 ml) was stirred while anhydrous aluminum chloride (15.5 g, 0.116 mol) was added in small portions. The red mixture was stirred for (2hrs.) at room temperature and refluxed on the water bath for a further (24 hours), the mixture was cooled, poured on to a mixture of ice and hydrochloric acid, product was crystallized once from ethanol and twice from petroleum ether b.p.(80-100)°C, m.p. 112°C, yield 52.5%. IR: 1665 cm<sup>-1</sup> (C=O) str. **Part One**

#### 2-(oxoalken-1-yl) phenoxathiin derivatives (3a-3j)

A mixture of (3g, 0.013 mol) 2-acetylphenoxathiin and (1.56 g, 0.0147 mol) of appropriate benzaldehyde in (80 ml) of ethanol and (1.5ml) of (1% NaOH) solution was refluxed for (2 hrs). The reaction mixture was poured in cold water, the

precipitate filtered off and recrystallized from (ethanol-water) (3:1) to give (3a-3j). FT-IR spectra of these compounds showed (C=O) str. band at (1670-1685)  $\text{cm}^{-1}$  and (1608-1600)  $\text{cm}^{-1}$  aliphatic (C=C) str. Table (1) represent the physical data of compounds (3a-3j). Characteristic bands of FT-IR spectra of compounds (3a-3j) are listed in Table (2).

## Part Two

### 2-(1-acetylpyrazolin-3-yl)

#### phenoxathiin derivatives(4a-4j)

To a solution of 2-(3-phenyl-1-oxypropen-1-yl) phenoxathiin(3a) (0.313g, 0.001 mol) in acetic acid (96%, 1 ml) hydrazine hydrate (0.4 ml, 0.008 mol) was added and the mixture was refluxed for 5 hrs., the product separated and out on cooling was crystallized from (ethanol-water) (3:1) to give (4a), the following compounds were prepared in this manner. FT-IR of these compounds showed absorption bands at (1460-1585)  $\text{cm}^{-1}$  aromatic (C=C) str., (1597-1612)  $\text{cm}^{-1}$  (C=N) str. and (1227-1258)  $\text{cm}^{-1}$  (C-N) str. Table (3) represent the physical data of compounds(4a-4j). Characteristic bands of FT-IR spectra of compounds (4a-4j) are listed in Table (4).

## Part Three

### 2-(1-phenylpyrazolin-3-yl)

#### phenoxathiin derivatives(5a-5j)

To a solution of 2-(3-phenyl-1-oxypropen-1-yl) phenoxathiin(3a) (1.65 g, 0.005 mol), phenyl hydrazine (0.830 g, 0.007 mol) in ethanol (80 ml) and few drops of piperidine were refluxed for 3 hrs. On concentration and cooling, gummy deposit separated out, this was crystallized from

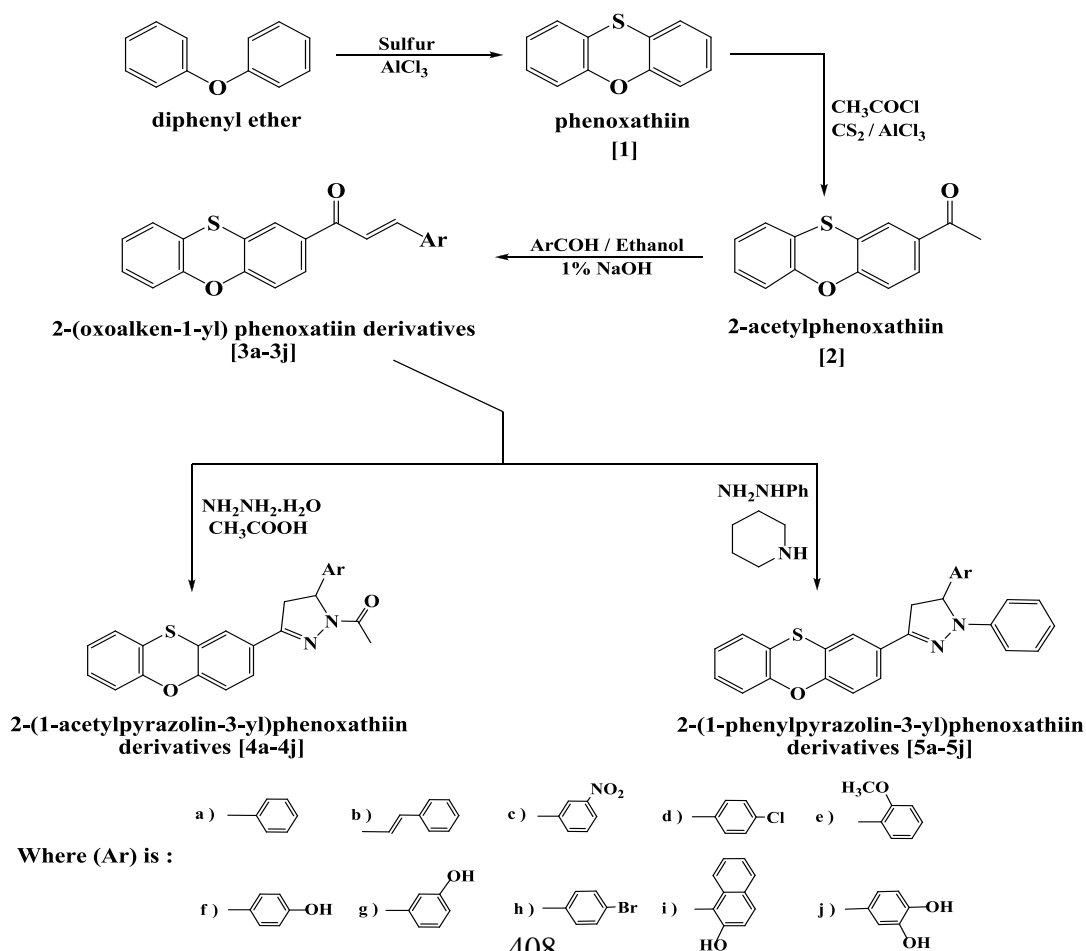
(ethanol-water) (3:1) to give (5a), the following compounds were prepared in this manner. FT-IR spectra of this compound and the following compounds showed absorption bands at (1460-1600)  $\text{cm}^{-1}$  aromatic (C=C) str., (1681-1682)  $\text{cm}^{-1}$  (C=N) str. and (1249-1355)  $\text{cm}^{-1}$  (C-N) str. Table (5) represent the physical data of compounds(5a-5j). Characteristic bands of FT-IR spectra of compounds (5a-5j) are listed in Table (6).

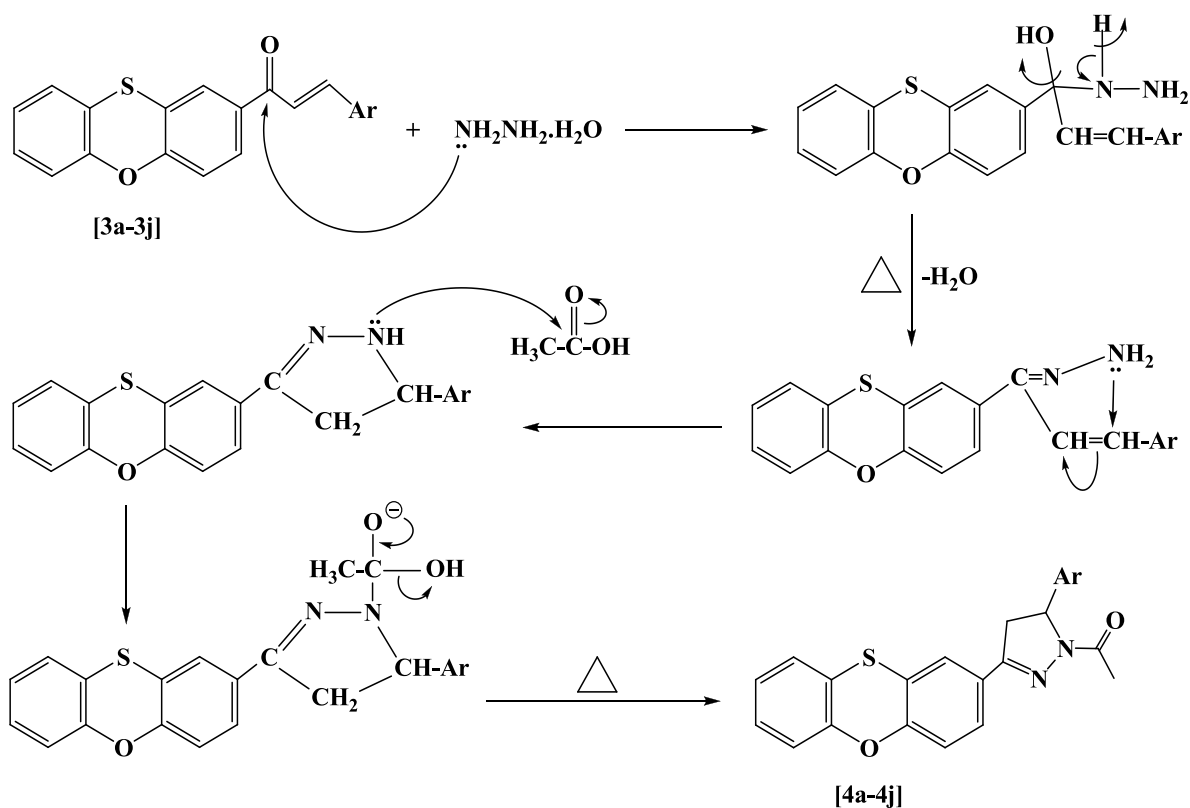
## Results and Discussion:

Phenoxathiin reacted with acetyl chloride in dry carbon disulfide in presence of anhydrous aluminum chloride to get 2-acetyl phenoxathiin through Friedel Crafts acylation method. FT-IR spectrum of phenoxathiin showed strong bands at 3063  $\text{cm}^{-1}$  aromatic (C-H) str., 1585  $\text{cm}^{-1}$  and 1450  $\text{cm}^{-1}$  assigned to the aromatic stretching system (C=C) str., 1219  $\text{cm}^{-1}$  and 1026  $\text{cm}^{-1}$  assigned to asym. and sym. (C-O-C) str. The  $^1\text{H-NMR}$  spectrum showed signals between  $\delta$ (6.8-7.3) ppm assigned to aromatic protons. FT-IR spectrum of compound (2) showed weak bands at 3078  $\text{cm}^{-1}$  for aromatic (C-H) str., 2962  $\text{cm}^{-1}$ , 2931  $\text{cm}^{-1}$  and 2877  $\text{cm}^{-1}$  aliphatic (C-H) str. of (CH<sub>3</sub>) acetyl group, strong bands at 1674  $\text{cm}^{-1}$  (C=O) str., two bands at 1558  $\text{cm}^{-1}$  and 1465  $\text{cm}^{-1}$  aromatic system (C=C) str. and 756  $\text{cm}^{-1}$  (C-H) aromatic ring. The  $^1\text{H-NMR}$  spectrum showed a signal at  $\delta$  2.6 ppm assigned to the three protons of the acetyl group and signals between  $\delta$  (7.0-7.3) ppm assigned to aromatic protons. Through nucleophilic addition reaction as the typical reaction of aldehydes and ketones, compound (2) undergoes the

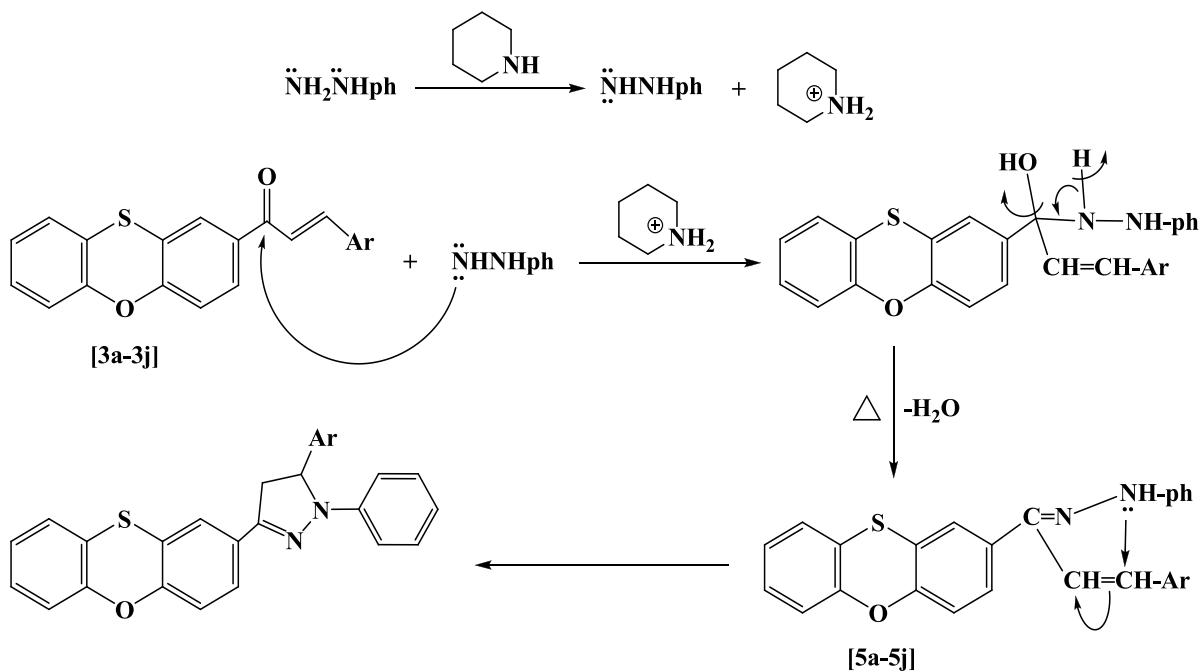
characteristic condensation reaction with different kinds of aromatic aldehydes in ethanol instead of 1% NaOH solution as a catalyst to afford aliphatic (C=C)str. The  $^1\text{H-NMR}$  spectrum showed a signal at  $\delta$  2.6 ppm assigned to aliphatic three protons of methoxy group, signals between  $\delta$ (7.0-7.3) ppm assigned to both olefinic H1 and H2 respectively and signals at  $\delta$  7.5 ppm and  $\delta$  7.9 ppm assigned to aromatic protons. The additive property of the exocyclic (C=C) in (3) conjugated with the carbonyl group promoted us to investigate their behavior towards the action of hydrazine hydrate, phenyl hydrazine react with (3) in presence of glacial acetic acid giving mono acetyl pyrazine(4a-4j). The structure of [4] has been established from IR spectra and UV. FT-IR spectrum showed absorption band at  $3040\text{ cm}^{-1}$  aromatic (C-H)str,  $2970\text{ cm}^{-1}$ ,  $2915$

(3a-3j). The IR spectra of compounds (3a-3j) showed absorption bands at  $(1670-1648)\text{ cm}^{-1}$  (C=O)str.,  $(1670-1685)\text{ cm}^{-1}$  and  $(1600-1608)\text{ cm}^{-1}$   $\text{cm}^{-1}$  and  $2800\text{ cm}^{-1}$  aliphatic (C-H)str. Strong bands at  $1665\text{ cm}^{-1}$  (C=O)str and  $1600\text{ cm}^{-1}$  (C=N)str,  $1550\text{ cm}^{-1}$  aromatic (C=C)str. Phenyl hydrazine reacted with (3) in ethanol in presence of piperidine giving 2-phenyl pyrazoline(5a-5j). The structure of these compounds was established from IR and UV. FT-IR spectrum showed reactivity medium weak bands at  $3055\text{ cm}^{-1}$  aromatic (C-H)str,  $2975\text{ cm}^{-1}$  and  $2865\text{ cm}^{-1}$  aliphatic (C-H)str. Strong bands at  $1620\text{ cm}^{-1}$  (C=N)str,  $1600\text{ cm}^{-1}$  (C=C)str. The  $^1\text{H-NMR}$  spectra for [5a] showed a signal at  $\delta$  1.2 ppm assigned to aliphatic protons (two H4 and H5) of pyrazoline ring respectively and a signal at  $\delta$  (6.6-7.5)ppm assigned to aromatic protons.



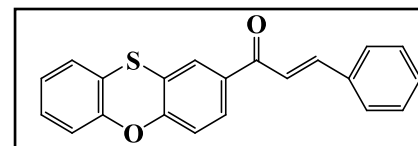


Reaction mechanism for the formation of compounds [4a-4j]



Reaction mechanism for the formation of compounds [5a-5j]

Table (1) represent the physical data of compounds(3a-3j)



| Comp. No. | Scientific name  | m.p. °C | Yield % | Color of crystal | Chemistry structure |
|-----------|--|---------|---------|------------------|---------------------|
| 3a        | 2-(3-phenyl-1-oxoprop-1-en-1-yl)phenoxathiin                 | 100-102 | 73.0    | Yellowish        |                     |
| 3b        | 2-(5-phenyl-1-oxypentadien-1-yl)phenoxathiin                 | 102-104 | 53.0    | Light-yellow     |                     |
| 3c        | 2-[3-(3-nitrophenyl)-1-oxoprop-1-en-1-yl]phenoxathiin        | 92-94   | 65.0    | Yellow           |                     |
| 3d        | 2-[3-(4-chlorophenyl)-1-oxoprop-1-en-1-yl]phenoxathiin       | 94-96   | 45.2    | Deep-yellow      |                     |
| 3e        | 2-[3-(2-methoxyphenyl)-1-oxoprop-1-en-1-yl]phenoxathiin      | 96-98   | 60.0    | Deep-yellow      |                     |
| 3f        | 2-[3-(4-hydroxyphenyl)-1-oxoprop-1-en-1-yl]phenoxathiin      | 103-105 | 67.0    | Reddish          |                     |
| 3g        | 2-[3-(3-hydroxyphenyl)-1-oxoprop-1-en-1-yl]phenoxathiin      | 102-104 | 67.2    | Yellow-reddish   |                     |
| 3h        | 2-[3-(4-bromophenyl)-1-oxoprop-1-en-1-yl]phenoxathiin        | 106-108 | 55.9    | Yellow           |                     |
| 3i        | 2-[3-(2-hydroxy-1-naphthyl)-1-oxoprop-1-en-1-yl]phenoxathiin | 92-94   | 53.9    | Black            |                     |
| 3j        | 2-[3-(3,4-dihydroxyphenyl)-1-oxoprop-1-en-1-yl]phenoxathiin  | 93-95   | 66.0    | Brown            |                     |

Table (2) Infrared absorption data for compounds (3a-3j)

| Comp. No. | Chemistry structure | FTIR spectral data $\text{cm}^{-1}$ |                               |                               |                          |                                    |
|-----------|---------------------|-------------------------------------|-------------------------------|-------------------------------|--------------------------|------------------------------------|
|           |                     | $\nu(\text{C}=\text{O})$            | $\nu(\text{C-H})$<br>aromatic | $\nu(\text{C-H})$<br>olefinic | $\nu(\text{C}=\text{C})$ | other bands                        |
| 3a        |                     | 1680                                | 3076                          | 3018                          | 1608                     | -                                  |
| 3b        |                     | 1681                                | 3090                          | 3010                          | 1600                     | (C-H)<br>olefinic<br>3010          |
| 3c        |                     | 1681                                | 3070                          | 2977                          | 1600                     | (NO <sub>2</sub> )<br>1535<br>1350 |
| 3d        |                     | 1674                                | 3078                          | 3009                          | 1600                     | (C-Cl)<br>1095                     |
| 3e        |                     | 1674                                | 3078                          | 3008                          | 1600                     | (C-O-C)<br>1249<br>1026            |
| 3f        |                     | 1674                                | 3078                          | 3009                          | 1600                     | (O-H)<br>3433                      |
| 3g        |                     | 1674                                | 3075                          | 3030                          | 1600                     | (O-H)<br>3440                      |
| 3h        |                     | 1674                                | 3078                          | 3009                          | 1600                     | (C-Br)<br>632                      |
| 3i        |                     | 1674                                | 3078                          | 3008                          | 1600                     | (O-H)<br>3409                      |
| 3j        |                     | 1674                                | 3078                          | 3009                          | 1600                     | (O-H)<br>3471                      |

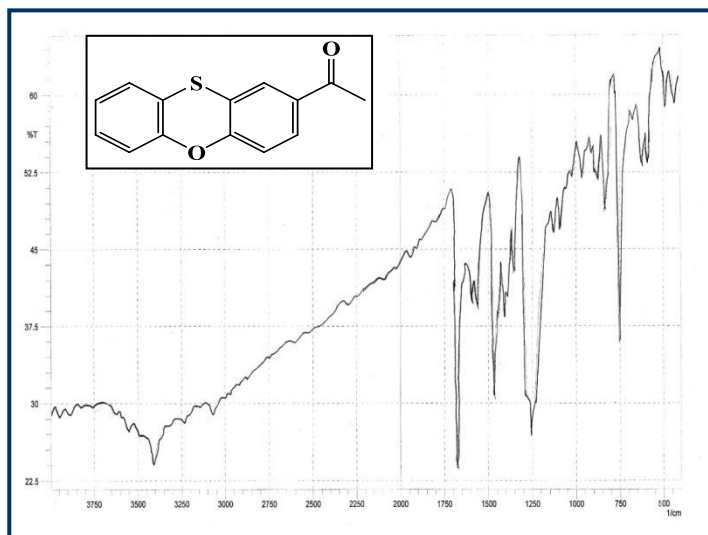


Fig.(1): FT-IR spectrum for compound(2)

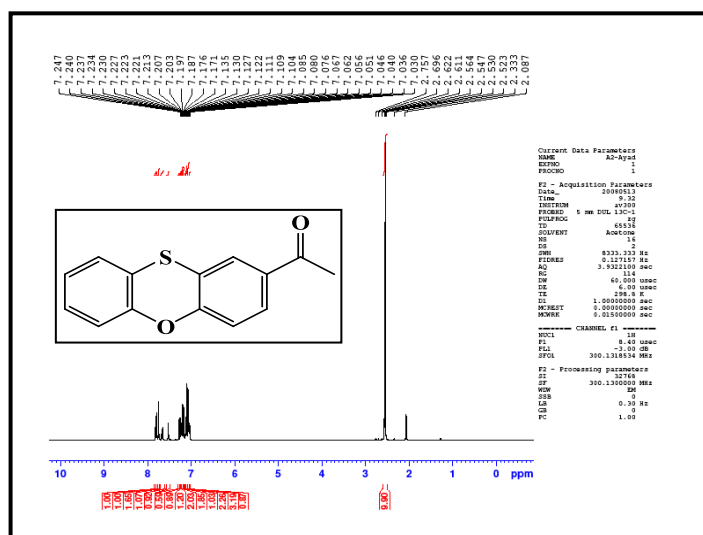


Fig.(2): <sup>1</sup>H-NMR spectrum for compound (2)

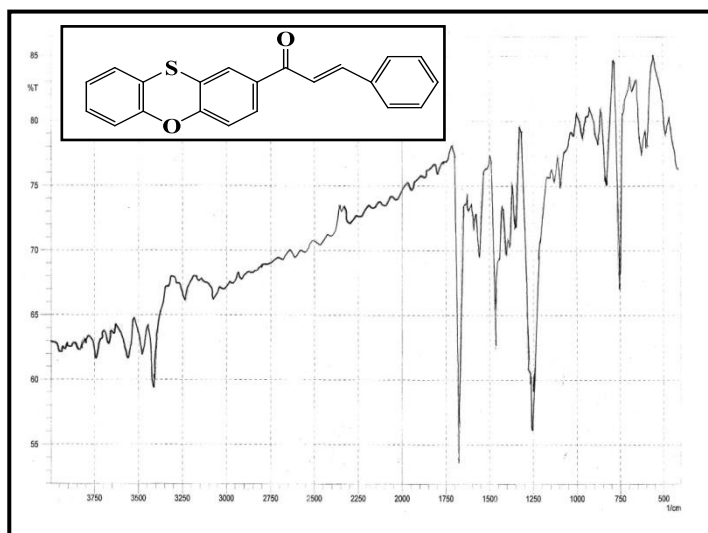


Fig.(3): FT-IR spectrum for compound(3a)

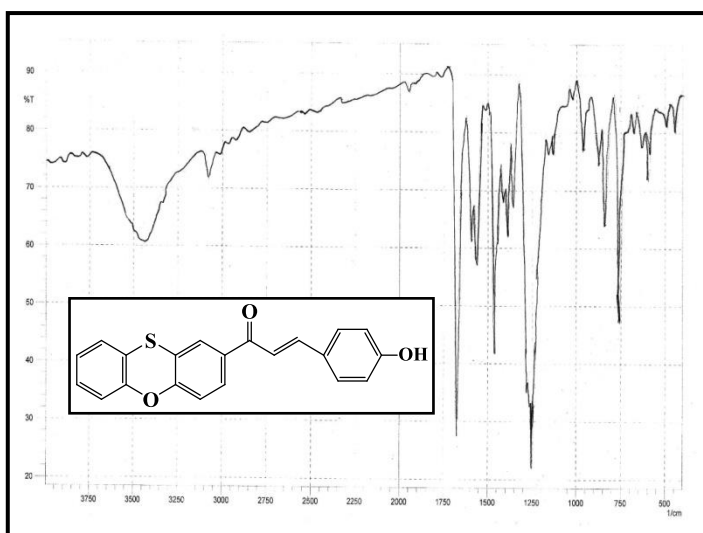


Fig.(4): FT-IR spectrum for compound(3f)

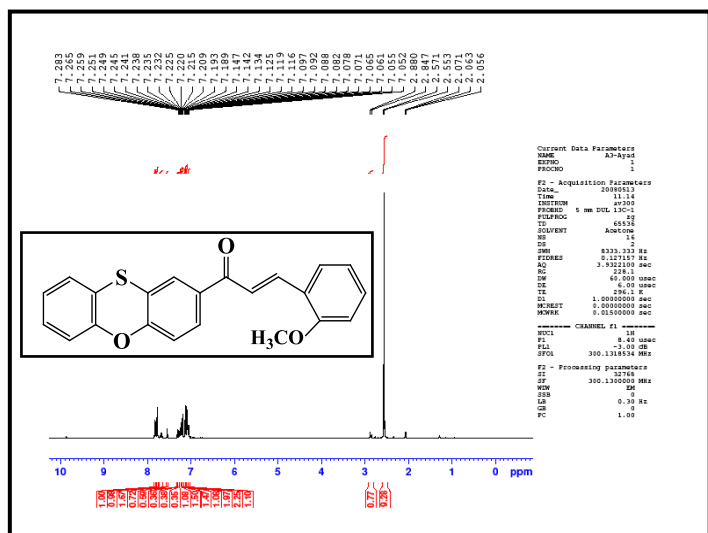


Fig.(5): <sup>1</sup>H-NMR spectrum for compound (3e)

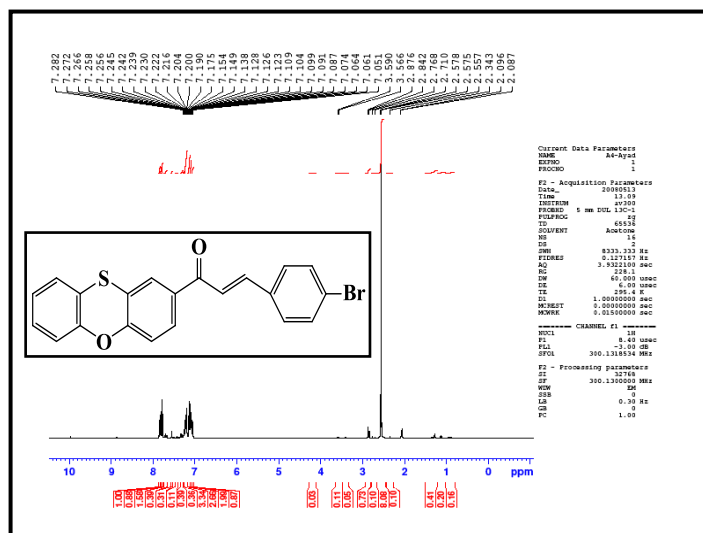
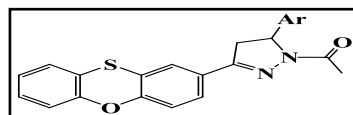


Fig.(6): <sup>1</sup>H-NMR spectrum for compound (3h)



Table (3) represent the physical data of compounds(4a-4j)

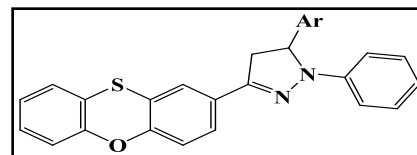


| Comp. No. | Scientific name   | m.p. °C | Yield % | Color of crystal | Chemistry structure |
|-----------|---|---------|---------|------------------|---------------------|
| 4a        | 2-(1-acetyl-5-phenylpyrazolin-3-yl) phenoxathiin                  | 155-157 | 46.1    | Yellow-brown     |                     |
| 4b        | 2-(1-acetyl-5-styrenyl pyrazolin-3-yl) phenoxathiin               | 182-184 | 47.8    | Yellow-brown     |                     |
| 4c        | 2-[1-acetyl-5-(3-nitrophenyl) pyrazolin-3-yl] phenoxathiin        | 110-112 | 64.1    | Yellow-brown     |                     |
| 4d        | 2-[1-acetyl-5-(4-chlorophenyl) pyrazolin-3-yl] phenoxathiin       | 115-117 | 86.0    | Brown            |                     |
| 4e        | 2-[1-acetyl-5-(2-methoxyphenyl) pyrazolin-3-yl] phenoxathiin      | 118-120 | 87.8    | Dark-yellow      |                     |
| 4f        | 2-[1-acetyl-5-(4-hydroxyphenyl) pyrazolin-3-yl] phenoxathiin      | 116-118 | 88.0    | Yellow-reddish   |                     |
| 4g        | 2-[1-acetyl-5-(3-hydroxyphenyl) pyrazolin-3-yl] phenoxathiin      | 117-119 | 79.8    | Yellow           |                     |
| 4h        | 2-[1-acetyl-5-(4-bromophenyl) pyrazolin-3-yl] phenoxathiin        | 121-123 | 80.2    | Pale yellow      |                     |
| 4i        | 2-[1-acetyl-5-(2-hydroxynaphthyl) pyrazolin-3-yl] phenoxathiin    | 122-124 | 58.4    | Black            |                     |
| 4j        | 2-[1-acetyl-5-(3,4-dihydroxy phenyl) pyrazolin-3-yl] phenoxathiin | 132-134 | 81.2    | Deep yellow      |                     |

Table (4) Infrared absorption data for compounds (4a-4j)

| Comp. No. | Chemistry structure | FTIR spectral data $\text{cm}^{-1}$ |                                      |                                       |                                      |                          |                          |                                    |
|-----------|---------------------|-------------------------------------|--------------------------------------|---------------------------------------|--------------------------------------|--------------------------|--------------------------|------------------------------------|
|           |                     | $\nu(\text{C}=\text{O})$            | $\nu(\text{C}-\text{H})$<br>aromatic | $\nu(\text{C}-\text{H})$<br>aliphatic | $\nu(\text{C}=\text{C})$<br>aromatic | $\nu(\text{C}=\text{N})$ | $\nu(\text{C}-\text{N})$ | other bands                        |
| 4a        |                     | 1681                                | 3078                                 | 2923<br>2854                          | 1500<br>1465                         | 1604                     | 1258                     | -                                  |
| 4b        |                     | 1674                                | 3062                                 | 2923<br>2862                          | 1551<br>1466                         | 1604                     | 1227                     | (C-H)<br>olefinic<br>3030          |
| 4c        |                     | 1682                                | 3086                                 | 2923<br>2854                          | 1585<br>1550                         | 1605                     | 1257                     | (NO <sub>2</sub> )<br>1500<br>1350 |
| 4d        |                     | 1682                                | 3078                                 | 2932<br>2870                          | 1558<br>1465                         | 1605                     | 1257                     | (C-Cl)<br>705                      |
| 4e        |                     | 1682                                | 3078                                 | 2924<br>2870                          | 1566<br>1466                         | 1612                     | -                        | (C-O-C)<br>1257<br>1018            |
| 4f        |                     | 1674                                | 3070                                 | 2924<br>2854                          | 1574<br>1460                         | 1597                     | 1257                     | (O-H)<br>3417                      |
| 4g        |                     | 1684                                | 3078                                 | 2932<br>2862                          | 1575<br>1466                         | 1605                     | 1257                     | (O-H)<br>3418                      |
| 4h        |                     | 1682                                | 3070                                 | 2935<br>2855                          | 1566<br>1466                         | 1597                     | 1257                     | (C-Br)<br>625                      |
| 4i        |                     | 1674                                | 3047                                 | 2932<br>2854                          | 1570<br>1465                         | 1597                     | 1258                     | (O-H)<br>3479                      |
| 4j        |                     | 1682                                | 3078                                 | 2932<br>2870                          | 1575<br>1465                         | 1605                     | 1258                     | (O-H)<br>3472                      |

Table (5) represent the physical data of compounds(5a-5j)



| Comp. No. | Scientific name   | m.p. °C | Yield % | Color of crystal | Chemistry structure |
|-----------|---|---------|---------|------------------|---------------------|
| 5a        | 2-(1,5-diphenylpyrazolin-3-yl) phenoxathiin                       | 86-88   | 38.9    | Red              |                     |
| 5b        | 2-[1-phenyl-5-styrenyl pyrazolin-3-yl] phenoxathiin               | 90-92   | 73.1    | Red              |                     |
| 5c        | 2-[1-phenyl-5-(3-nitrophenyl) pyrazolin-3-yl] phenoxathiin        | 94-96   | 71.0    | Reddish          |                     |
| 5d        | 2-[1-phenyl-5-(4-chlorophenyl) pyrazolin-3-yl] phenoxathiin       | 93-95   | 82.2    | Reddish          |                     |
| 5e        | 2-[1-phenyl-5-(2-methoxy phenyl) pyrazolin-3-yl]phenoxathiin      | 112-114 | 70.7    | Red              |                     |
| 5f        | 2-[1-phenyl-5-(4-hydroxyphenyl) pyrazolin-3-yl]phenoxathiin       | 126-128 | 74.5    | Red              |                     |
| 5g        | 2-[1-phenyl-5-(3-hydroxyphenyl) pyrazolin-3-yl]phenoxathiin       | 114-116 | 77.3    | Brown            |                     |
| 5h        | 2-[1-phenyl-5-(4-bromophenyl) pyrazolin-3-yl]phenoxathiin         | 120-122 | 78.0    | Brown            |                     |
| 5i        | 2-[1-phenyl-5-(2-hydroxy naphthyl) pyrazolin-3-yl] phenoxathiin   | 150-152 | 65.0    | Black            |                     |
| 5j        | 2-[1-phenyl-5-(3,4 dihydroxy phenyl) pyrazolin-3-yl] phenoxathiin | 167-169 | 57.3    | Brown            |                     |

Table (6) Infrared absorption data for compounds (5a-5j)

| Comp. No. | Chemistry structure | FTIR spectral data $\text{cm}^{-1}$ |                                |                               |                   |                   |                                    |
|-----------|---------------------|-------------------------------------|--------------------------------|-------------------------------|-------------------|-------------------|------------------------------------|
|           |                     | $\nu(\text{C-H})$<br>aromatic       | $\nu(\text{C-H})$<br>aliphatic | $\nu(\text{C=C})$<br>aromatic | $\nu(\text{C=N})$ | $\nu(\text{C-N})$ | other bands                        |
| 5a        |                     | 3065                                | 2930<br>2850                   | 1566<br>1470                  | 1681              | 1249              | -                                  |
| 5b        |                     | 3062                                | 2920<br>2860                   | 1600<br>1566<br>1465          | 1681              | 1257              | (C-H)<br>olefinic<br>3020          |
| 5c        |                     | 3050                                | 2960<br>2910                   | 1597                          | 1682              | 1250              | (NO <sub>2</sub> )<br>1560<br>1358 |
| 5d        |                     | 3062                                | 3008<br>2923<br>2860           | 1597<br>1465                  | 1681              | 1257              | (C-Cl)<br>700                      |
| 5e        |                     | 3063                                | 3008<br>2910                   | 1589<br>1466                  | 1681              | 1350              | (C-O-C)<br>1257<br>1041            |
| 5f        |                     | 3063                                | 2962<br>2923<br>2854           | 1597<br>1465                  | 1682              | 1357              | (O-H)<br>3340                      |
| 5g        |                     | 3062                                | 3000<br>2940<br>2850           | 1597<br>1460                  | 1681              | 1355              | (O-H)<br>3330                      |
| 5h        |                     | 3060                                | 2910<br>2840                   | 1558<br>1460                  | 1681              | 1350              | (C-Br)<br>610                      |
| 5i        |                     | 3070                                | 3020<br>2915                   | 1558<br>1466                  | 1681              | 1350              | (O-H)<br>3479                      |
| 5j        |                     | 3063                                | 2923<br>2862                   | 1597<br>1460                  | 1682              | 1350              | (O-H)<br>3448                      |

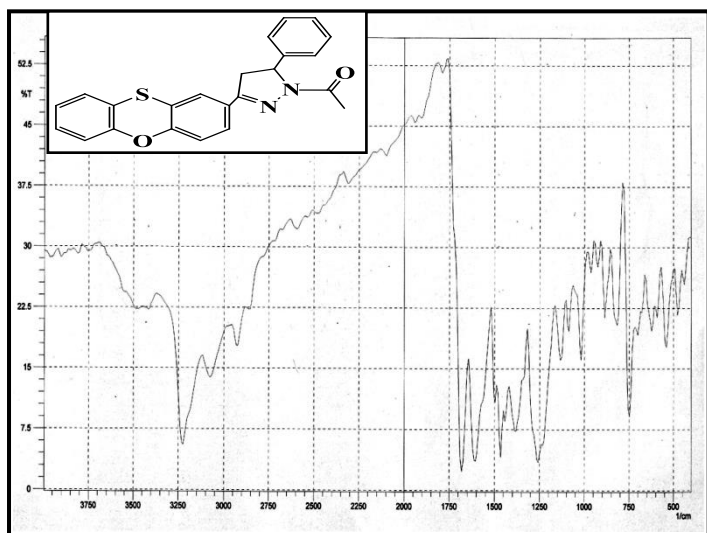


Fig.(7): FT-IR spectrum for compound(4a)

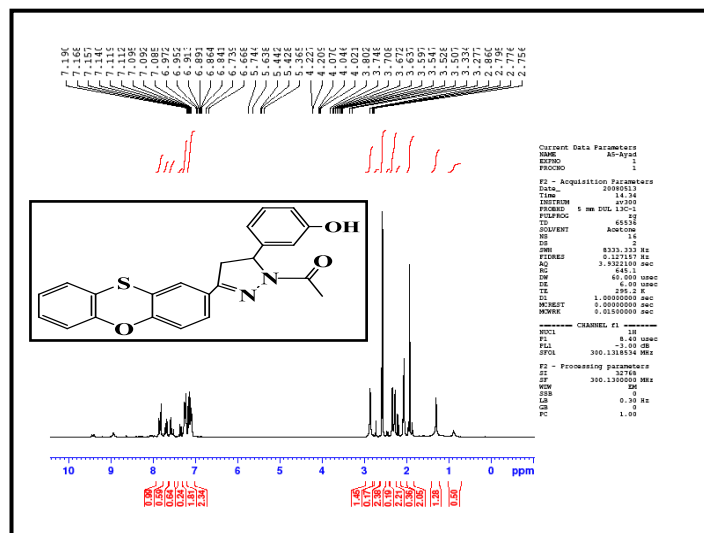


Fig.(8): <sup>1</sup>H-NMR spectrum for compound (4g)

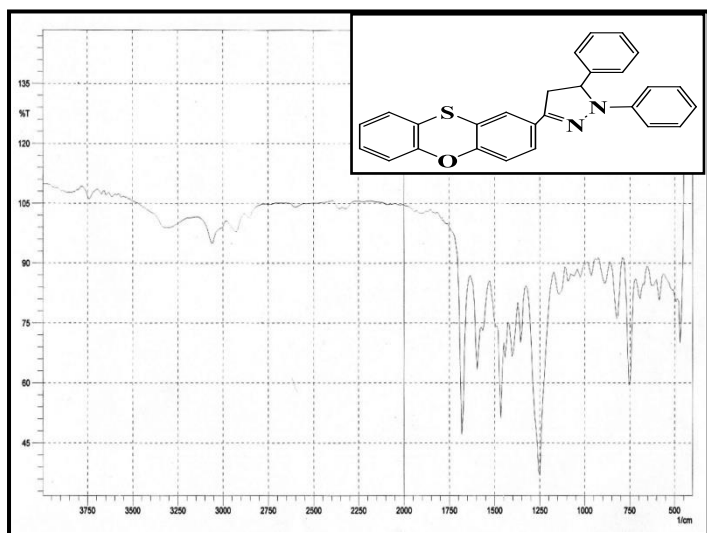


Fig.(9): FT-IR spectrum for compound(5a)

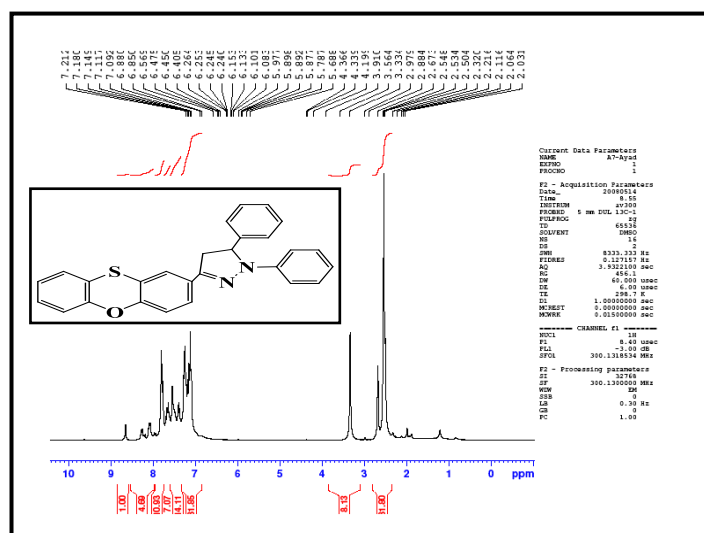


Fig.(10): <sup>1</sup>H-NMR spectrum for compound (5a)

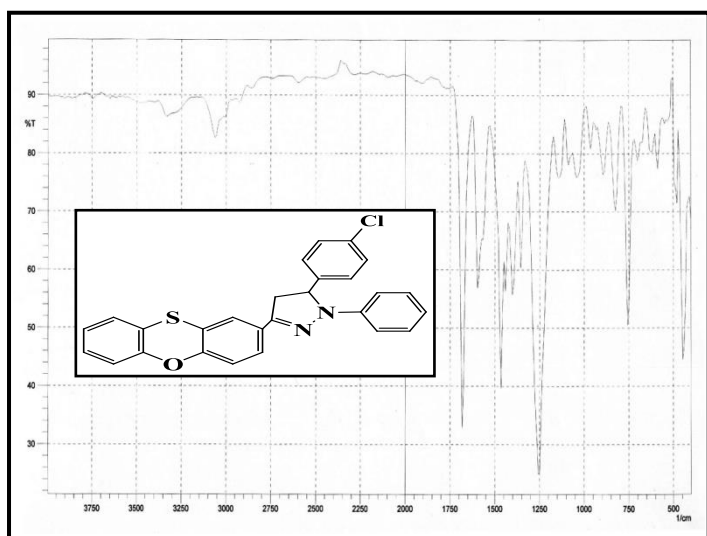


Fig.(11): FT-IR spectrum for compound(5d)

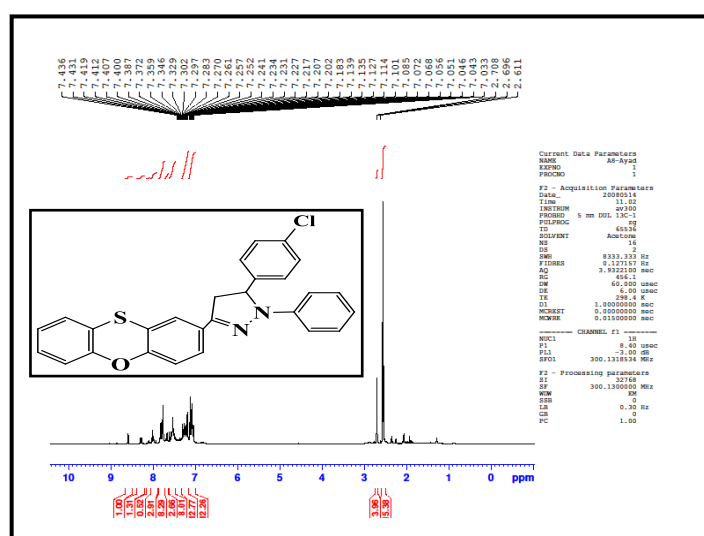


Fig.(12): <sup>1</sup>H-NMR spectrum for compound (5d)

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### تحضير مشتقات جديدة للبايرازولينفينوكسيثين سعاد مصطفى الاعرجي\* ، أياد احمد

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#### الخلاصة:

تم تحضير الفينوكسيثين من تفاعل ثنائي فنيلى ايتير بوجود كلوريد الالمنيوم الجاف. تضمن البحث تحضير مشتقات جديدة من الفينوكسيثين التي تحتوي على حلقات غير متجانسة. وقد صنفت جميع هذه المركبات المحضرة الى ثلاث مجاميع تحتوي كلاها منها على عشرة مركبات. المجموعة الاولى هي مشتقات ل-2-(أوكسو الكين-1-يل) الفينوكسيثين (3a-3j) والمحضرة من تفاعل 2-أسيتيل فينوكسيثين مع مختلف المركبات العطرية الالهيدية وبوجود هيدروكسيد الصوديوم. اما مركبات المجموعتين الثانية والثالثة فقد تم تحضيرها عن طريق مفاعلة مركبات المجموعة الاولى (3a-3j) مع كل من الهيدرازين بوجود حامض الخليك للحصول على مشتقات ل-2-(1-أسيتيل بايرازولين-3-يل) للفينوكسيثين (4a-4j)، ومعالفنيلى هيدرازين بوجود البايبيريدين لتعطي مشتقات ل-2-(1-فنيلى بايرازولين-3-يل) فينوكسيثين (5a-5j). جميع مركبات المجموعتين أعلاه معوضة فيالموقع

(5) في حلقة البايرازولين بمجاميع أريل وحسب المركبات العطرية الالدهايدية المستخدمة في تحضير مركبات المجموعة الأولى.