# " synthesis and characterization of some 1,3-oxazepines derived from Schiff-bases of hydrazide derivative from 4-nitroisonenzofuran-1,3-dione"

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**ABSTRACT**:

New 1,3-oxazepine derivatives (V-XIX) were synthesized from the reaction of Schiff-bases (I) derived from certain hydrazide of 3-nitrophthalic anhydride and three types of anhydrides (phthalic anhydride (II), naphthalic anhydride (III) and 3-nitrophthalic an hydride(IV) ) (1-nitroisobenzofuran-1,3-dione) respectively . these 1,3-oxazepines were characterized by their m . p , FTTR, C.H. N analysis and $^{1}$HMNR spectra of some of them . Scheme(1) show the reaction path used to prepare the proposed 1,3-oxazepine .

Key words : 4-nitroisobenzofura-1,3-dione , hydrazide , 1,3-oxazepine.

## Introduction:

1,3-oxazepines are a seven member ring compound having two hetoro atoms at 1- and 3- positions for nitrogen and oxygen atoms respectively . many 1,3-oxazepine derivatives were synthesized by many workers [1-7] and find their biological activities . some of 1,3-oxazepines derived from hydrazide derivative of pyromellitic dianhydride were also prepared and their IR , $^{1}$HNMR and UV spectra were studied too [8].

Many methods of preparation of 1,3-oxazepines were studied using different solvents such as benzene , dioxane and ethanol [9,10] .

Thus , the synthesis of 1,3-oxazepine derivatives [11] and the discovery of many many other 1,3-oxazepine derivatives helped to extend these reactions [12] . the aim of the present research is to synthesis of new 1,3-oxazepines derived from Schiff-bases of hydrazide derivatives from 4-nitroisobenzofuran-1,3-dian , and characterized some of their physical properties .

## Experimental:-

Materials:- all the chemicals were supplied from Mark , Aldrich and Fluka chemical Co. , were used as received without any further purification or recrystallization. Benzene was dried by sodium foils .

**Techniques:-**

FTIR spectra were recorded by using KBR discs , and on 8400s shimadza Spector photo meter , while HNMR spectra were done by company :ULTRA shield 300MHZ , Bruker , Switerland at AI-Bayt university , Jordan , using DMSO or CDCL3 as a certain solvents as desired , with TMS as an internal standard , all the examined samples were reported in ppm . ULTRA violate- visible spectra were performed using CECL 7200 spectra photo meter in methanol as a solvent . C.H.N micro an a lysis were done by: Euro vector , model EA 3000 A , Italy . uncorrected m .p were measured using Hot-stage Gallen Kamp melting point apparatus .

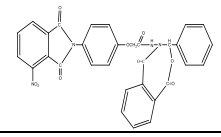
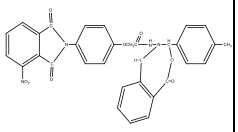
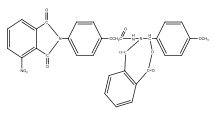
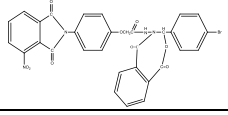
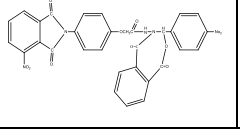
**General Procedure :-**

Synthesis of all the some Schiff-bases of hydrazide derived from 4-nitroisobenzofuran-1,3-dion [I] was done as in previous work [13] , these Schiff-bases were worked to produce[1,3] oxazepine derivatives as follows :-

Synthesis of N-(3,4-arly )-1,5-dioxobenzo[e] [1,3]oxazepine -4 (1H,3H,5H)-yl) -2-(4-(4-nitro-1,3-dioxoisoindolin-2-yl) phenoxy ) acetamide .[V-IX]. [7,8,12]: to a solution of (0.01 mole , 148 gm) of phthalic anhydrivde in 15 ml of benzene placed in 100ml round . bottom flask and heated to 70 C in a water bath was added a solution of (0.01 mole) of Schiff-bases[I] in about 10ml. of benzene . the mixture was further heated and refluxed with continuous stirring at 70-80$^0$ C for 6-8hrs,Then The Mixture was left to cool in ice-bath where by crystallization was taken place and the products separated out . Oxazepine derivatives that obtained were first washed with dry benzene (2-5 ml) and recrystallized from dry benzene .

All physical data , element analysis (C.H.N) and reflexing time for the synthesized compound [V-IX] are listed in (table-1) .

Table -1- The physical data , element analysis of compounds [V-IX]

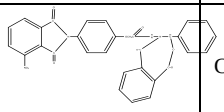
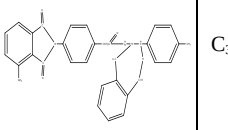
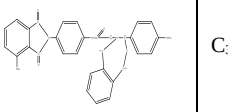
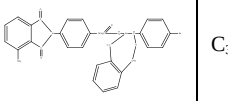
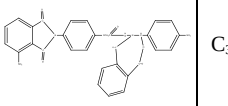
| Comp. No. | Nomenclature | Structural formula | Molecular formula | Element analysis | | | Refluxing Time hr | m. p °c | Yield % | Color |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | C% | H% | N% | | | | |
| V | N-(1,5-dioxo-3-phenyl benzene[e][1,3]oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoiso indolin-2-yl) phenoxy) acetamide. |  | $C_{31}H_{20}N_4O_9$ | Cal. 62.83 | 3.37 | 9.45 | 6 | 199-197 | 80 | Yellow |
| | | | | Found 62.92 | 3.41 | 9.33 | | | | |
| VI | N-(1,5-dioxo-3-(4-tolyl)benzene[e][1,3] oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoiso indolin-2-yl) phenoxy) acetamide. |  | $C_{32}H_{22}N_4O_9$ | Cal. 63.36 | 3.63 | 9.24 | 6 | 184-182 | 85 | Deep Yellow |
| | | | | Found 63.55 | 6.29 | 9.40 | | | | |
| VII | N-(1,5-dioxo-3-(4-methoxy phenyl) benzene[e][1,3]oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoiso indolin-2-yl) phenoxy) acetamide. |  | $C_{32}H_{22}N_4O_{10}$ | Cal. 61.73 | 3.53 | 9.00 | 7 | 169-167 | 85 | Orange |
| | | | | Found 62.01 | 3.66 | 9.37 | | | | |
| VIII | N-(1,5-dioxo-3-(4-bromo phenyl)-benzene[e][1,3]oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoiso indolin-2-yl) phenoxy) acetamide. |  | $C_{31}H_{19}BrN_4O_9$ | Cal. 55.43 | 2.83 | 8.34 | 8 | 192-190 | 80 | Deep Yellow |
| | | | | Found 55.81 | 2.67 | 8.11 | | | | |
| IX | N-(1,5-dioxo-3-(4-nitro phenyl) benzene[e][1,3]oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoiso indolin-2-yl) phenoxy) acetamide. |  | $C_{31}H_{19}N_5O_{11}$ | Cal. 58.39 | 2.98 | 10.98 | 70 | 203-201 | | Yellow |
| | | | | Found 58.61 | 3.11 | 10.67 | | | | |

-Synthesis of N-(3,4-aryl)-1,5-dinaphtho [2,3-e][1,3]oxazepine -4(1H,3H,5H)-yl) -2-(4-(4-nitro-1,3-dioxoiso indolin-2-yl) phenoxy) acet amide[X –VX ].[7,12].

To a solution of(0.02 mole 3.96 gm) of naphthalic anhydride in 20ml of toluene placed in 100ml round-bottom flask and heated to 70-75$^0$ C in a water bath was added a solution of (0.02mole) of Schiff-bases [I] in about 15ml of toluene . the mixture was refluxed with continuous stirring for 5 hrs. using a condenser fitted with ($CaCl_2$) tube by means of hot-plate then the solvent was evaporated by Vacuum , the solid derivative was collected and washed with EtOH and benzene and recrystallized from toluene .

All physical ,elemental analysis (C.H.N) for the new compounds [X-VX] are listed in ( table 2)

Table -2- The physical data , element analysis of compounds [X- VX]

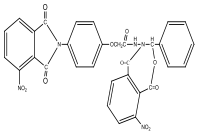
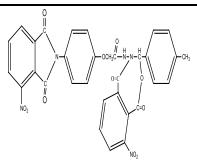
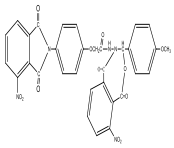
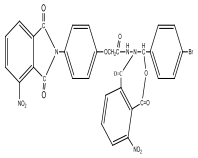
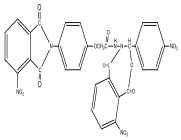
| Comp. No. | Nomenclature | Structural formula | Molecular formula | Element analysis | | | m. p °c | Yield % | Color |
|---|---|---|---|---|---|---|---|---|---|
| | | | | C% | H% | N% | | | |
| X | N-(1,5-diox-3-phenyl naphtha[2,3-e][1,3][oxazepine-4(1H,3H,5H)-2-4-(4-nitro-1,3-dioxoisoindlin-2-yl)phenoxy)acetamide |  | $C_{35}H_{22}N_4O_9$ | Cal. 65.42 | 3.42 | 8.72 | 208-210 | 84 | Yellow |
| | | | | Found 65.61 | 3.65 | 8.48 | | | |
| XI | N-(1,5-diox-3-(phenyl)naphtha[2,3-e][1,3][oxazepine-4(1H,3H,5H)-yl)-2-4-(4-nitro-1,3-dioxoisoindlin-2-yl)phenoxy)acetamide |  | $C_{36}H_{24}N_4O_9$ | Cal. 56.85 | 3.65 | 8.53 | 244-246 | 80 | Yellow |
| | | | | Found 56.93 | 3.77 | 8.33 | | | |
| XII | N-(3-(4-methoxyphenyl)-1,5-dioxnaphtho [2,3-e][1,3][oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoisoindlin-2-yl)phenoxy)acetamide |  | $C_{36}H_{24}N_4O_{10}$ | Cal. 64.28 | 3.57 | 8.33 | 255-257 | 83 | Orange |
| | | | | Found 64.11 | 3.47 | 8.06 | | | |
| XIII | N-(3-(4-Bromophenyl)-1,5-dioxnaphtho [2,3-e][1,3][oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoisoindlin-2-yl)phenoxy)acetamide |  | $C_{35}H_{21}BrN_4O_9$ | Cal. 58.25 | 2.91 | 7.45 | 181-183 | 75 | Deep Yellow |
| | | | | Found 58.38 | 3.03 | 7.18 | | | |
| XIV | N-(3-(4-nitrophenyl)-1,5-dioxnaphtho [2,3-e][1,3][oxazepine-4(1H,3H,5H)-yl)-2-(4-(4-nitro-1,3-dioxoisoindlin-2-yl)phenoxy)acetamide |  | $C_{35}H_{21}N_5O_{11}$ | Cal. 61.13 | 3.05 | 10.18 | 178-180 | 70 | Pale Yellow |
| | | | | Found 60.98 | 3.11 | 9.92 | | | |

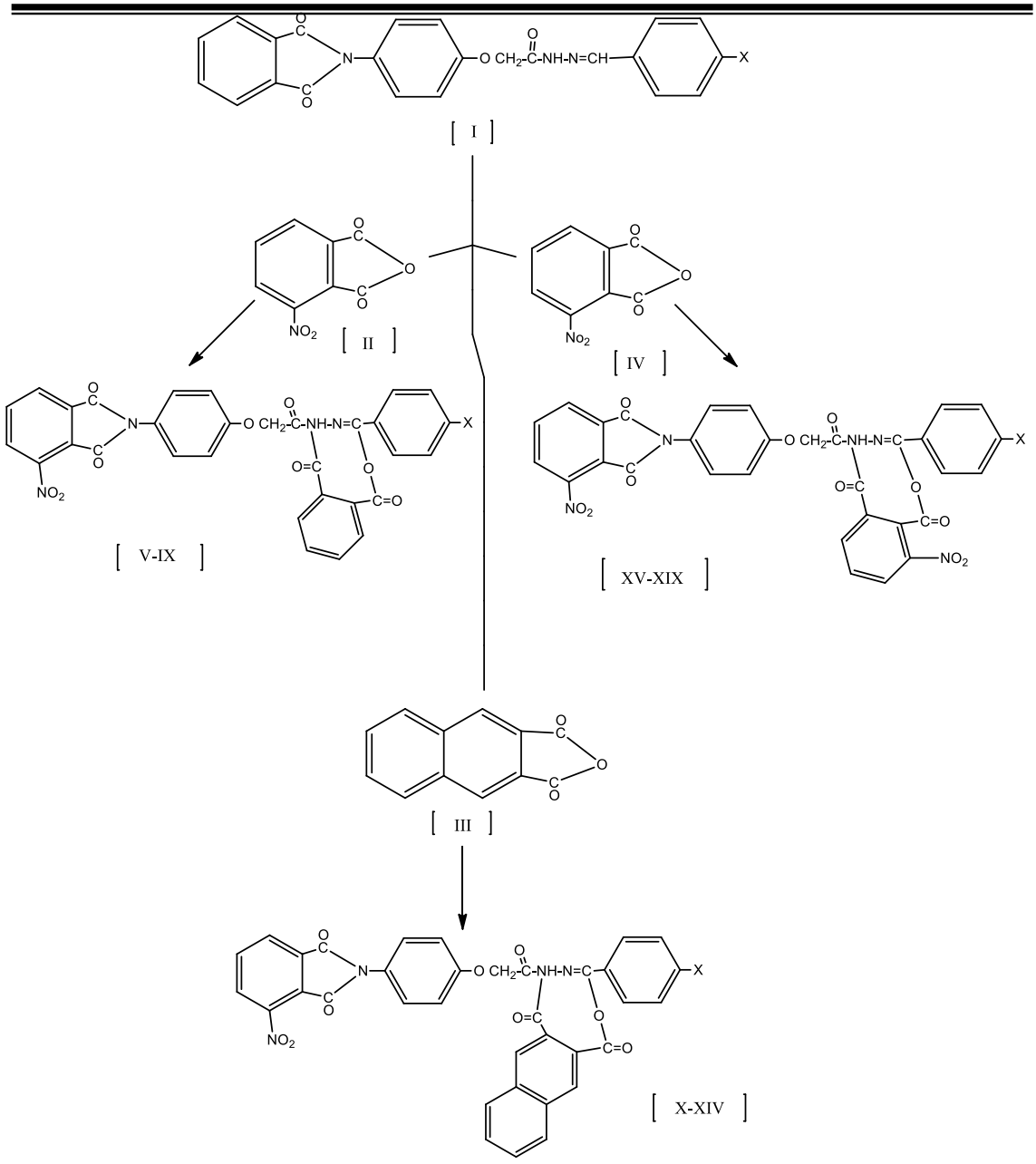
-Synthesis of N-(3-(4-aryl)- 9-nitro-1,5- dioxin benzene [e][1,3] oxazepine -4(1H,3H,5H)-yl)-2-(4-nitro-1,3-dioxoiso in dolin-2-yl ) phenoxy )a catamite . [XV-XIX][8,12].

As previous procedures- to a solution of (0.01mol 1.93gm) of pure 4-nitroisobenzenofuran-1,3-dione (3-nitrophthalic anhydride) in 25ml benzene placed in 250ml round-bottom flask and heated to $85^0$ C by means of water both was added a solution of (0.01mol) of Schiff-bases [I]dissolved in 25ml benzene . the reacting mixture was refluxed for 8hrs using a condenser filled with $CaCl_2$ tube . The product was allowed to cool at room temperature , filtered and washed with dry benzene and recrystallized from ethanol- benzene [1:1] mixture .

All physical properties , elemental analysis (C.H.N) for novel compounds [XV-XIX] are listed in (table 3).

Table -3- The physical data , element analysis of compounds [XV-XIX]

| Comp. No. | Nomenclature | Structural formula | Molecular formula | Element analysis | | | m.p$^0$C | Yield % | Color |
|---|---|---|---|---|---|---|---|---|---|
| | | | | C% | H% | N% | | | |
| XV | N-(9-Nitro-1,5-dioxo-3-phenyl benzo[e] [1,3]oxazepine-4 (1H,3H,5H)-yl)-2-(4-(4-nitro dioxoiso in dolin-2-yl)phenoxy) acetamide |  | $C_{31}H_{19}N_5O_{11}$ | Cal. 58.39 | 2.98 | 10.98 | 222-224 | 82 | yellow |
| | | | | Found 58.21 | 3.11 | 11.08 | | | |
| XVI | N-(3-tolyl)-(9-Nitro-1,5-dioxo-3-phenyl benzo [e] [1,3] oxazepine-4 (1H, 3H, 5H) -yl) -2-(4-(4-nitro dioxoiso in dolin-2-yl)phenoxy) acetamide |  | $C_{32}H_{21}N_5O_{11}$ | Cal. 58.98 | 3.22 | 10.75 | 246-248 | 81 | Yellow |
| | | | | Found 58.69 | 3.33 | 10.21 | | | |
| XVII | N-(3-(methoxy phenyl)- (9-Nitro-1,5-dioxo-3-phenyl benzo[e] [1,3] oxazepine-4 (1H, 3H, 5H)- yl) -2-(4-(4-nitro dioxoiso in dolin-2-yl) phenoxy) acetamide |  | $C_{32}H_{21}N_5O_{12}$ | Cal. 57.57 | 3.14 | 10.49 | 200-202 | 67 | Deep Yellow |
| | | | | Found 56.99 | 3.61 | 10.30 | | | |
| XVIII | N-(3-(4-bromo phenyl)- (9-Nitro-1,5-dioxo-3-phenyl benzo [e] [1,3] oxazepine-4 (1H, 3H, 5H) -yl) -2- (4-(4-nitro dioxoiso in dolin-2-yl) phenoxy) acetamide |  | $C_{31}H_{18}BrN_5O_{11}$ | Cal. 51.95 | 2.09 | 9.77 | 188-190 | 65 | Orange |
| | | | | Found 52.21 | 1.81 | 7.13 | | | |
| XIX | 2-(4-(4-nitro-1,3-dioxoisoindolin-2-yl)phenoxy-N- (9-Nitro-3- (4-nitro phenyl) 1,5- benzo [e] [1,3] oxazepine-4(1H,3H,5H)-yl) acetamide |  | $C_{31}H_{18}N_6O_{13}$ | Cal. 54.54 | 2.63 | 12.31 | 190-192 | 60 | Yellow |
| | | | | Found 54.40 | 2.22 | 12.08 | | | |




Where: X= $CH_3$ , $OCH_3$ , Br , $NO_2$

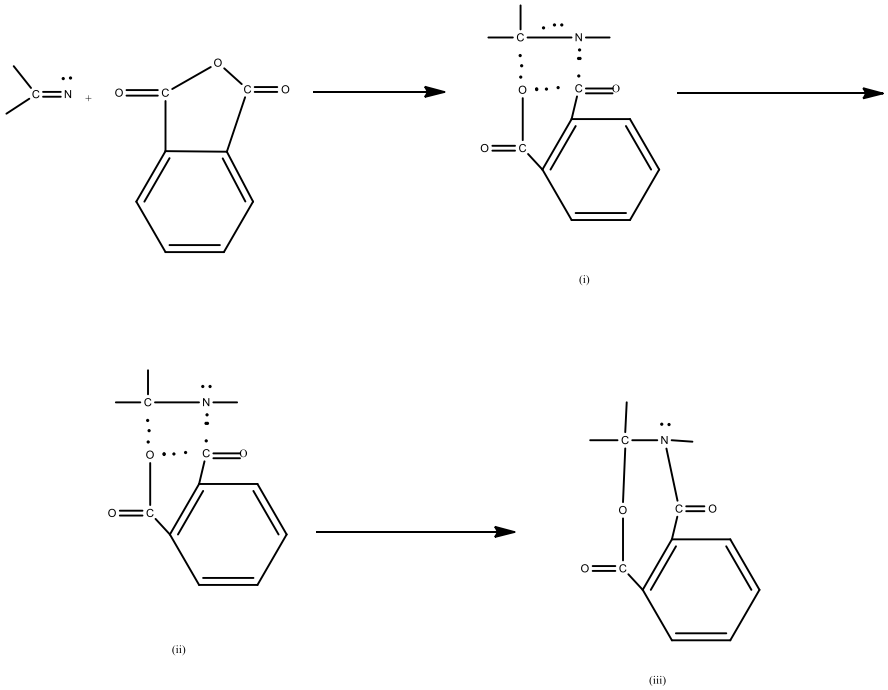
Scheme (I)

## Result and Discussion :-

Because the aim of this research is to synthesis of novel 1,3-oxazepine, the author started to prepare the Schiff-bases [I] of hydrazide derivative from 4-nitro isobenzenefurane-1,3-dione[13]. The novel 1,3-oxazepine derivatives were obtained by addition reaction of equi molar of Schiff-bases [I] with certain anhydrides (phthalic anhydride , naphthalic anhydride and 3-nitro phthalic an hydride ) in deferent solvents (as shown in the experimental part ).

The novel oxazepines [V-XIX] were obtained by refluxing compounds [I] with the above anhydrides .

The following mechanism was suggested to their reaction [14] to prepare the desired compounds



In this mechanism one of 6-carbomyl group added to T-bound of the Schiff-bases (N=C) which will give four-member cyclic ring (i) which will give five-member cyclic ring (ii) in the same transition state of the anhydride .

The later transition stat will destroy and opens to give seven-member cyclic ring of 1,3-oxazepine.

The new synthesized compounds were identified by their melting point FTIR $^{1}$HMNR spectroscopy and element analysis (C.H.N) .

In FTIR absorption spectra the author recorded the disappearance of certain bands due to imine group (C=N) which present on Schiff-bases moiety , and also the disappearance of anhydride moiety due to (O=C-O-C=O) group and the cyclic anhydride , and the appearance of two bands at ~1770 $cm^{-1}$ and ~1710 $cm^{-1}$ for two carbonyl group in lactone ring and oxazepine ring, also bands near (1280 and 1103 $cm^{-1}$ ) due to asymmetric and symmetric (C-O-C) band [15-17].

The FTIR data of all the oxazepines are listed in table[4].

[ Table 4 ] characteristic FTIR Absorption bands of compounds [ V –XIX ] Characteristic absorptions bands $cm^{-1}$

| Comp. No. | Vc-H st. Benzylic | Vc-H st. aromatic | Vc-H st. aliphatic | Vc =0 lactone | Vc=0 lactum | Vc=0 aromatic | Vn-H | Additional peaks |
|---|---|---|---|---|---|---|---|---|
| V | 3310 | 3090 | 2940-2851 | 1765,1640 | 1730 | 1600 | 3405 | V $No_2$ 1530,1355 |
| VI | 3300 | 3095 | 2950-2851 | 1770,1680 | 1735 | 1603 | 3510 | V $No_2$ 1530,1350 |
| VII | 3305 | 3070 | 2927-2840 | 1775,1685 | 1735 | 1605 | 3390 | V $No_2$ 1535,1355 |
| VIII | 3315 | 3072 | 2938-2850 | 1778,1650 | 1743 | 1600 | 3400 | V $No_2$1530,1353 Vc-Br 735 |
| IX | 3310 | 3083 | 2940-2850 | 1766,1653 | 1738 | 1606 | 3440 | V $No_2$ 1535,1355 |
| X | 3320 | 3025 | 2920-2855 | 1770,1675 | 1740 | 1625 | 3446 | V $No_2$ 1530,1355 |
| XI | 3310 | 3070 | 2928-2850 | 1770,1685 | 1740 | 1595 | 3405 | V $No_2$ 1535,1350 |
| XII | 3300 | 3065 | 2940-2830 | 1775,1680 | 1739 | 1600 | 3385 | V $No_2$ 1530,1350 |
| XIII | 3310 | 3070 | 2940-2850 | 1780,1685 | 1735 | 1605 | 3425 | V $No_2$ 1535,1355 Vc-Br 740 |
| XIV | 3305 | 3090 | 2928-2850 | 1770,1650 | 1745 | 1608 | 3330 | V $No_2$ 1535,1350 |
| XV | 3308 | 393 | 2940-2840 | 1770,1680 | 1715 | 1600 | 3450 | V $No_2$ 1530,1350 |
| XVI | 3310 | 3085 | 2930-2850 | 1760,1665 | 1720 | 1680 | 3435 | V $No_2$ 1525,1355 |
| XVII | 3315 | 3025 | 2910-2843 | 1770,1660 | 1718 | 1605 | 3405 | V $No_2$ 1530,1355 |
| XVIII | 3300 | 3030 | 2950-2840 | 1775,1668 | 1730 | 1630 | 3470 | V $No_2$ 1530,1355 Vc-Br 735 |
| XIX | 3300 | 3035 | 2953-2845 | 1775,1665 | 1735 | 1630 | 3460 | V $No_2$ 1530, 1355 |

1- The $^1$HNMR spectrum (in DMSO) DF compound [V] showed the following signals:-

***a-*** Sixteen aromatic protons appear at the ring (ȝ7.08 ~ 7.85)ppm

***b-*** A singlet signal at ȝ6.85 ppm due to N-CH proton moiety

***c-*** A sharp signal due N- H proton appeared at ȝ10.20 ppm

***d-*** Two protons of $CH_2$ group of (-$OCH_2$C=O) moiety at ȝ4.30ppm

2- (in DMSO) of compound [IV] showed the following signals:

***a-*** Fifteen aromatic protons appears at the range (ȝ7.45 – 8.15ppm)

***b-*** A singlet signal at ȝ7.05 ppm which could attribute to N-CH proton in (-N-CH Ar) moiety

***c-*** A sharp signal at ȝ10.22 ppm due to N-H proton

***d-*** A singlet two protons of $CH_2$ group at ȝ4.30 ppm due to (-O-$CH_2$-C=O) moiety

***e-*** Three protons of (-$CH_3$) group at ȝ2.35 ppm [18]

3- The $^1$HNMR spectrum (in DMSO) of compound [VII] showed the following signals:

***a-*** The fifteen aromatic protons appears at about ) ȝ7.65 – 8.25 ppm)

***b-*** A signal due to N-CH proton in (-N-CH-Ar) moiety at ȝ 7.00 ppm

***c-*** Two protons appear at ȝ4.32 ppm due to $CH_2$ in (-O-$CH_2$-C=O) group

***d-*** Sharp signals at ȝ 3.82 ppm due to the three protons of (-$OCH_3$) group (methoxy group)[16]

***e-*** A sharp signal at ȝ10.20 ppm due to -NH group

4- The $^1$HNMR spectrum (in DMSO) of compound [VIII] showed the following ƺ's in its HNMR spectra
   - ***a-*** Fifteen aromatic protons at about ƺ(7.20 – 8.10)
   - ***b-*** A signal at ƺ(7.05 ppm which could attribute to N-CH moiety
   - ***c-*** A signal at ƺ4.35 ppm due to $CH_3$ group in (-O-$CH_2$ C=O) moiety
   - ***d-*** A sharp signal due to NH proton at ƺ10.25 ppm

5- The $^1$HNMR spectrum (in DMSO) of compound [IV] showed the following signals:
   - ***a-*** Fifteen aromatic protons appears at the range ƺ(7.55 – 8.5 ppm)
   - ***b-*** A singlet signal at ƺ 7.05 ppm due to N=CH proton moiety
   - ***c-*** Sharp signal at ƺ10.25 ppm due to NH proton
   - ***d-*** Two protons of $CH_2$ in (-O$CH_2$- C=O) moiety appeared at ƺ 4.31 ppm

6- The $^1$HNMR spectrum (in DMSO) of compound [X] gave the following signals:
   - ***a-*** Eighteen aromatic protons appear at the range (ƺ7.02 – 7.88) ppm
   - ***b-*** A singlet signal at ƺ6.88 ppm correspond to N-CH proton group
   - ***c-*** Sharp signal due to N-H proton appeared at ƺ 10.18 ppm
   - ***d-*** Two protons of $CH_2$ group in (-O$CH_2$ C=O) moiety at ƺ 4.33 ppm

7- The $^1$HNMR spectrum (in DMSO) of compound [XI] showed the following signals:
   - ***a-*** Seventeen aromatic protons appear at about ƺ( 7.37 – 7.81)ppm
   - ***b-*** A singlet signal at ƺ7.03 ppm which could be attributed to N-CH proton
   - ***c-*** A sharp signal appeared at ƺ10.22 ppm due to N-H proton
   - ***d-*** A singlet two protons of $CH_2$ moiety at ƺ4.30 ppm due to (-O-$CH_2$-C=O) group
   - ***e-*** Three protons of the methyl group appeared at ƺ 2.35 ppm [18]

8- The $^1$HNMR spectrum (in DMSO) of compound [XII] showed the following signals:
   - ***a-*** Seventeen aromatic protons appear at the range (ƺ7.35 – 791 )ppm
   - ***b-*** A singlet signal at ƺ 6.89 ppm due to N-CH proton's group.
   - ***c-*** A sharp single due to N-H proton appeared at ƺ 10.21 ppm.
   - ***d-*** Two protons of $CH_2$ group appeared at 4.33 ppm due to (-O-$CH_2$C=O)moiety.
   - ***e-*** Sharp singlet signal of the methoxy group appeared at ƺ 3.8 ppm[16].

9- The $^1$HNMR spectrum (in DMSO) of compound [XIII] showed the following signal:-
   - ***a-*** The seventeen aromatic proton at the range (ƺ 7.20-8.0) ppm.
   - ***b-*** A signal at ƺ7.04 ppm which could be attribute to N-CH group.
   - ***c-*** A signal at 84.34 ppm due to $CH_2$ group in (-O-$CH_2$C=O)moiety.

10-The $^1$HNMR spectrum(in DMSO) (in DMSO) of compound [ XIV] showed the following signals:

- ***a-*** Seventeen aromatic protons appeared at the range ƺ(7.56 – 8.4)ppm
- ***b-*** A singlet signal at ƺ7.06 ppm due to N-CH proton
- ***c-*** A sharp signal at ƺ 10.25 ppm due to NH proton
- ***d-*** Two protons of $CH_2$ in (-O $CH_2$ C=O) moiety appeared at ƺ4.32 ppm

***11-*** The $^1$HNMR spectrum (in DMSO) of compound [XV] showed the following signals :

- ***a-*** Fifteen aromatic protons appeared at the range (ƺ7.50 – 8.30)ppm
- ***b-*** A singlet signal at ƺ6.88 ppm due to N-CH proton
- ***c-*** A sharp singlet signal due to N-H proton at ƺ10.21 ppm
- ***d-*** Two protons of $CH_2$ group in (-$OCH_2$ C=O) moiety appeared at ƺ4.33 ppm

***12-*** The $^1$HNMR spectrum (in DMSO) of compound [XVI] showed the following signals:

- ***a-*** Following aromatic protons appeared at ƺ(7.55 – 8.35 )ppm
- ***b-*** A singlet signals at ƺ7.03 ppm which could be attribute to N-CH proton in (N-CH-Ar) moiety
- ***c-*** A sharp signal at ƺ10.30 ppm due to N-H proton
- ***d-*** Two protons (singlet ) at ƺ4.33 ppm due to $CH_2$ in (_$OCH_2$ C=O) moiety
- ***e-*** Three protons of (-$CH_3$) group at ƺ2.35 pp [18]

***13-*** The $^1$HNMR spectrum (in DMSO) of compound [XVII] showed the following signals:

- ***a-*** Fourteen aromatic protons appeared at ƺ(7.33 – 7.95)ppm
- ***b-*** A singlet signal at ƺ6.95ppm due to N-CH proton
- ***c-*** A singlet signal due to N-H proton appeared at ƺ10.15 pm
- ***d-*** The two protons of $CH_2$ group in (-O-$CH_2$ CO) moiety appeared at ƺ 4.32 ppm
- ***e-*** The three protons of –$CH_3$ group at ƺ3.80ppm [16]

***14-*** The $^1$HNMR spectrum (in DMSO) of compound [XVIII] showed the following result :

- ***a-*** The fourteen aromatic protons appeared at ƺ(7.50 – 8.30)ppm
- ***b-*** A signal at ƺ7.0 ppm due to N-CH moiety
- ***c-*** A signal at ƺ 4.30ppm due to $CH_2$ group in (-$OCH_2$ C=O) moiety
- ***d-*** Sharp signal at ƺ10.2ppm due to NH proton

***15-*** The $^1$HNMR spectrum (in DMSO) of compound [XIX] showed the following signals:

- ***a-*** The fourteen aromatic protons appeared at ƺ(7.25 – 7.95)ppm
- ***b-*** A singlet signal at ƺ7.10ppm due to N-CH proton
- ***c-*** Sharp signal at ƺ10.20ppm due to NH proton

***d-*** Two protons of $CH_2$ in (-$OCH_2$ C=O) moiety appeared at 4.30ppm

From all these result m.p , FTIR , C.H.N analysis and $^1$HNMR which all fit with the propose structure of 1,3 – Oxazepines derived from Schiff – bases of hydrazide derivative from 4- nitroiso benzo furane – 1,3 – dione.

The partial hydrolysis of thede 1,3 – oxazepines derivatives with NaOH solution and HCl will be transferred to further researches.

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# تحضير وتشخيص 1,3 اوكسازبينات المشتقة من قواعد شف من الهيدرازيد المنتمية الى 4- نايترو ايزونانز فيوران (1,3) دايون


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**الخلاصة :-**

تم تحضير مشتقات جديدة ل ( 1,3- اوكسازبين) (V-XIX) من تفاعل قواعد شيف (I) المشتقة من بعض الهيدرازايدات ( 3-نتروانهدريد الخليك) مع ثلاثة انواع من الانهيدرايدات هي (فثاليك انهدريد (II), نفثاليك انهدريد (III) و 3 -نتروفثاليك (VI) على التوالي .

شخصت المركبات المحضرة الجديدة من خلال درجات الانصهار , اطياف الاشعه تحت الحمراء , تحاليل العناصر ( الكاربون , الهيدروجين , النتروجين ) واطياف الرنين النووي المغناطيسي . والتي اظهرت تطابق النتائج مع التراكيب الكيميائية للمركبات المحضرة .

المخطط (1) يمثل طريق سير التفاعل للمركبات المحضرة .



الكلمات المفتاحية : 4- نايتر ايزونانز – 1,3 دايون , هيدرازايد , 1,3 – اوكسازبين .