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# Synthesis and Characterization of Some New Oxazolidinone and Thiazolidinone Derivatives Via Reaction of Schiff Bases Derived from some Amino Acids

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**Keywords:**
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Imines.

**ABSTRACT**

New derivatives of 2,3- substituted – oxazolidinone and thiazolidinone were synthesized by cycloaddition reaction of glycolic acid and thioglycolic to various Schiff bases derived from some commercially available amino acids in anhydrous 1,4-dioxane under dry and reflux conditions. Schiff's bases were synthesized by refluxing various amino acids with aromatic aldehydes and ketones with few drops of glacial acetic acid as a catalyst in absolute methanol. The products were isolated, purified and characterized by their melting point, FT-IR and $^{1}$HNMR spectra and C.H.N analysis.

## 1- Introduction

Oxazolidinones and Thiazolidinone are a class of five-membered ring heterocyclic compounds, containing two heteroatoms, oxygen in oxazolidinones or Sulfur in thiazolidinone and nitrogen in position 1and 2 in the cyclopentane ring respectively and carbonyl group located at 2,4 or 5 positions with respect the oxygen atoms (position 1) and other substituents, (figure1).[1-3]

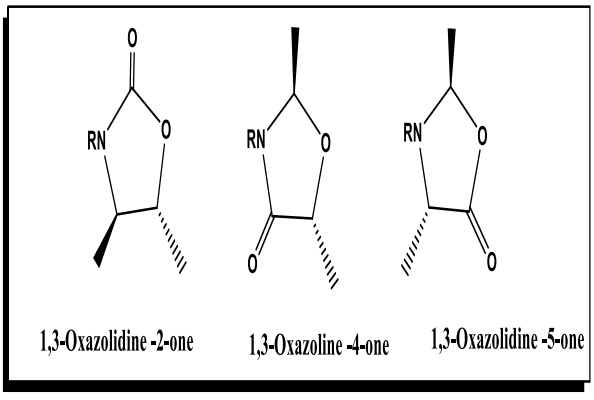


**Figure 1: Chemical structures of oxazolidinones**

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Oxazolidinones are potent bioactive compounds, and versatile chiral auxiliaries and key intermediates for organic and bioorganic, dyes, agrochemicals and natural products synthesis they have been used as :antibacterial[4], antimicrobial[5], anticonvulsant[6], antitumor[7], antiviral[8], anti-inflammatory[9], psychotropic[10], cardiotonic[11], antifungal [12], antihyperglycemic, analagesic[13], antitbercular, anticoagulant, antidepressant, phospholipase inhibitor, anticonvulsant ,agriculture fungicide, CNS depressants,antithyoid, antiblastic and urinary tract infection agents . [14]

Thiazolidinone derivatives were informed to display antitumor [15], antituberculor[16], anti-HIV[17], analgesic[18], anti-inflammatory([19], ulcerogenic [20], and antibacterial [21] and antifungal [22] activities. Therefore it was envisaged that compounds containing thiazolidinone moieties would result in compounds of interesting biological properties. . The best method of preparation of thiazaolidinone by the reaction of thioglycolic acid with imines (Schiff bases) [20]

synthesis of oxazolidinones essentially based on the reactions of commercially available materials such amino acid, amino alcohols, Ethyl carbonate, triphosgene, Urea . azidochloro carbonates, chiral aziridines N-aryl carbamates imines, chloroacetic acid and glycolic acid .These reagents have been used to synthesize various oxazoline -2-one and oxazolidine -5-one derivatives in efficient yield . [23-26]

## 2. Experimental

### 2.1. Instrumentation

Melting points were determined in open capillary tubes and are uncorrected. The FT-IR spectra were recorded, between (4000- 600 $cm^{-1}$) range on an Infrared spectrophotometer Model Tensor 27 Bruker Co., Germany. The $^{1}$HNMR spectra were recorded on a Bruker Ultershield *300MHz* NMR spectrometer, Co., Germany, in DMSO- $d_6$ as a solvent, the chemical shifts are reported as δ values in part per million (ppm) relative to TMS δ=0, as internal standard. The C.H.N. elemental analyses were performed by Euro EA Elemental Analyser.

### 2.2General procedure for synthesis of Schiff's Bases 3(a-h).

A solution of equimolar mixture of (18.8 mmole) of aromatic aldehydes (1) and (13.4 mmole) the specific amino acid (2) in presence of a few drops of glacial acetic acid as a catalyst in absolute methanol (40 ml) was refluxed for (2-3) hrs. with continuous stirring .The reaction mixture was allowed to cool down in ice bath, whereby a crystalline solid product was separating during cooling. The solid product was filtered off, washed with distilled water, dried and recrystallized from the same solvent. The structure, IR characteristic absorption, yield %, melting point, colors, and the reaction time are given in table (1).

### 3.3 *General procedure for synthesis of* oxazolidinones *4(a-c).*

In a well dried 100-ml round- bottom flask equipped with condenser and anhydrous calcium chloride guard tube, solution of equimolar amount (13.3 mmol) of compounds 3(a-c) and (9.5 mmole) glycolic acid in anhydrous 1,4-dioxan (50ml) was refluxed for (4-7 hrs). The reaction mixture was allowed to cool down in ice bath, whereupon a crystalline solid product was separating out during cooling. The product was filtered off, washed with distilled water, dried and recrystallized from ethanol . The chemical formula, molecular weights, C.H.N %, yield%, melting points, colors, are given in table (2).

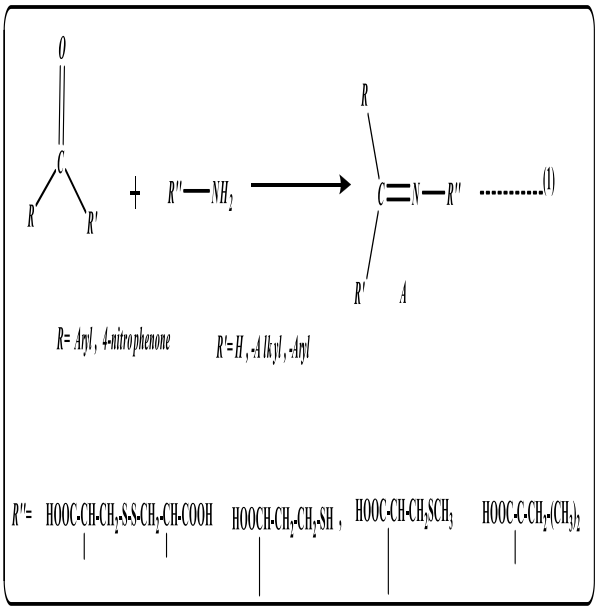
### 3.3 *General procedure for synthesis of* Thiazolidinone *4(d-i).*

In a well dried 100-ml round- bottom flask equipped with condenser and anhydrous calcium chloride guard tube, solution of equimolar amount (13.3 mmol) of compounds 3(c-) and (10.8 mmol) thioglycolic acid in anhydrous 1,4-dioxan (50ml) was refluxed for (4-7 hrs). The reaction mixture was allowed to cool down in ice bath, whereupon a crystalline solid product was separating out during cooling. The product was filtered off, washed with distilled water, dried and recrystallized from ethanol. The FT-IR spectra and chemical formula, molecular weights, C.H.N %, yield%, melting points, colors, are given in table (1), and table(3).

## 3. Result and Discussion

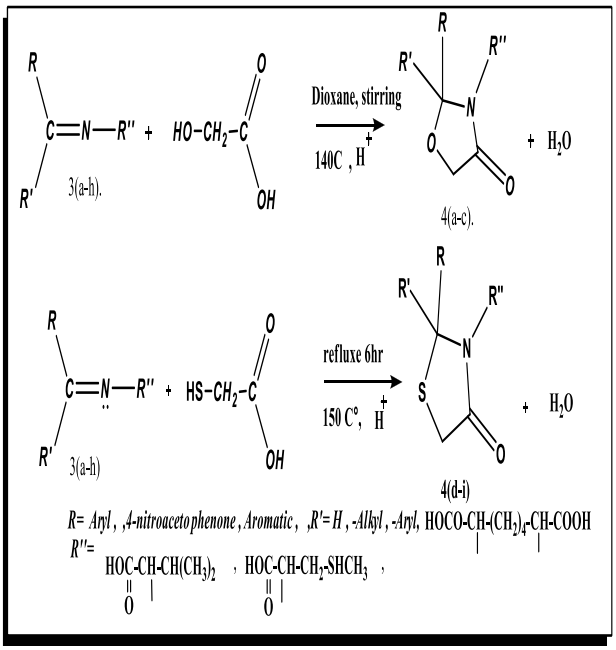
In this work, the reaction of Glycolic acid and thioglycolic acid as electrophilic reagent with imines (schiff's bases) as mild Nucleophilic reagents in reported. Schiff's Bases were synthesized from substituted, 4-nitro benzaldehyde, benzaldehyde and valine, cysteine, Methionine and cystine by acid catalyzed thermal condensation reaction according to a well- known procedure.

The equation of imine formation is thoroughly elucidated in the literatures.[27-28]

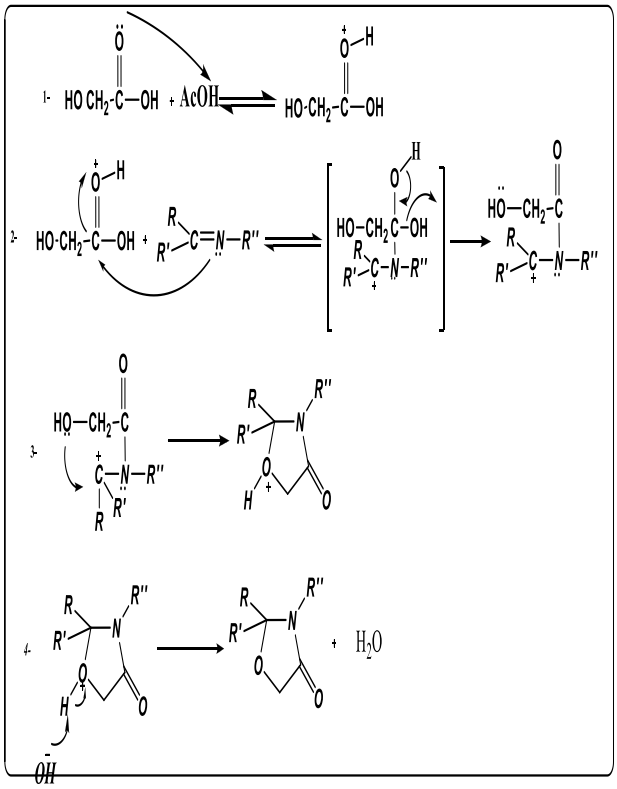



Characterization of the synthesized Schiff's bases table (1). The FT-IR spectra showed the disappearance of the characteristic stretching absorption band of (-$NH_2$) group at (3300-3500) $cm^{-1}$ of the amino group and that of the (C=O) group at (1710-1740) $cm^{-1}$ of the carbonyl compounds, and the appearance of the stretching frequencies of azomethine (C=N) group (1615-1690) in addition, to that of the substituted groups in each individual structure.

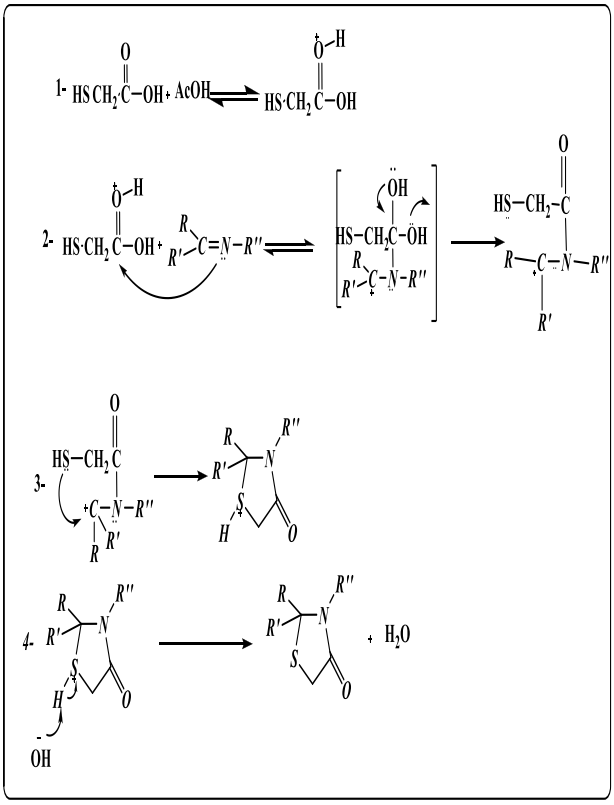
Schiff's bases were reacted with glycolic acid and thioglycolic acid in an anhydrous 1,4- Dioxane under in dry atmosphere and reflux conditions.




Of particular importance is to understand how this transformation occurs, therefor a plausible mechanism can be suggested as follows:



***Scheme-2*): Mechanism of ( oxazolidin-4-one (formation)**



***(Scheme-3): (1,3-Thiazolidine-4-one formation) Mechanism of***

The first step includes addition of a proton to the carbonyl group and in the second step the nucleophilic azomethine group attacks the electrophilic carbon atom of the carbonyl group to give a dipolar reactive intermediate which collapses to give protonated 5-membered ring in third step . The expulsion of the water molecule occurred by the fourth step, to give the target molecule . The products were characterized by their melting point, C.H.N analysis and FT-IR and $^1$HNMR spectra, tables, (2) and (3).

The structures of the synthesized oxazolidin-4-one and *1*,3-Thiazolidine-4-one were confirmed by their melting points and both FT-IR and $^1$HNMR spectra and the C.H.N.% of the products, table 2 and table 3. The FT-IR spectra showed of the characteristic absorption frequencies (bands) of both (C=O) group at (1690-1720) $cm^{-1}$ of glycolic acid and thioglycolic acid that showed the disappearance the characteristic absorption frequencies of azomethine group (C=N) at (1609-1657) $cm^{-1}$. and the appearance of the characteristic absorption frequencies of both (C=O) at (1690-1715) $cm^{-1}$ and (O-H) at (2900-3200) $cm^{-1}$ in addition to the appearance of stretching absorption of the other groups in the structure of each individual compounds. The resulting signals at the chemical shifts in the$^1$HNMR spectra of each individual molecular structure of the product are in fair consistency with the expected signals of each proton in the different environment. Additional evidence was obtained from C.H.N. %, since the founded percentage of these elements is in high agreement with the calculated figures.

Table (1): The structural formula, IR characteristic absorption, yield, melting point, colors, and the reaction time of Schiff's Bases 3(a-h).

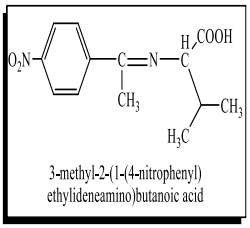
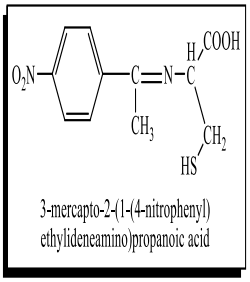
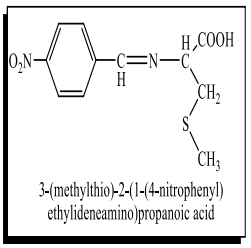
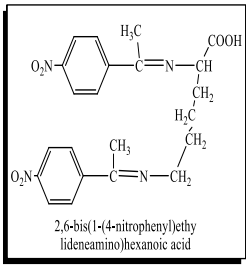
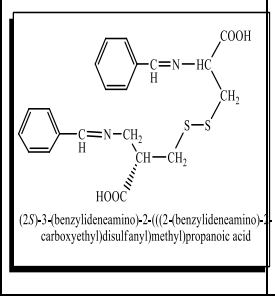
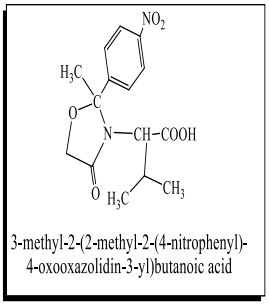
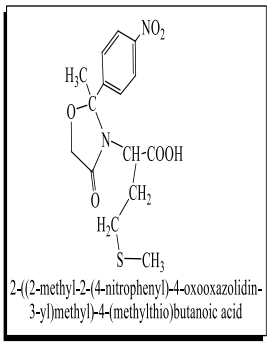
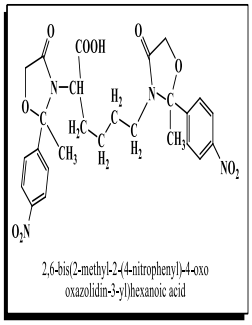
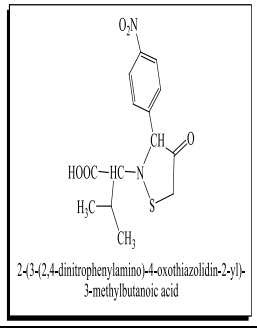
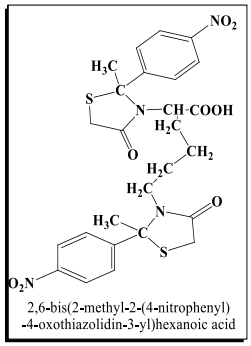
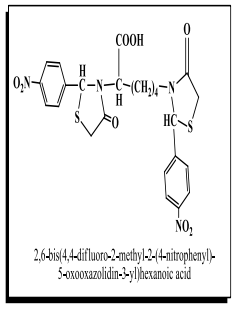
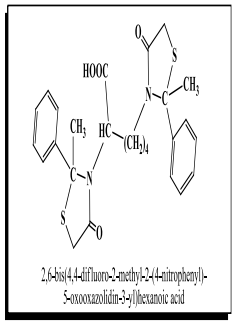
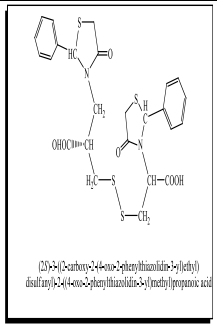
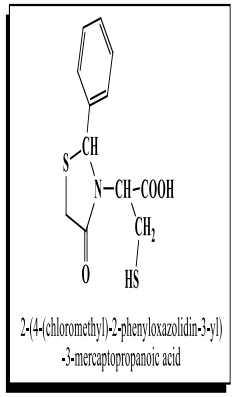
| Comp *Cod* | *Structure* Molecular | *I.R Characteristic Absorption Frequencies, $Cm^{-1}$* | *m.p C°* | *Yield %* | *Reaction Time (hr.)* | *Colour* |
|---|---|---|---|---|---|---|
| 3a |  3-methyl-2-(1-(4-nitrophenyl) ethylideneamino)butanoic acid | *2596 vs O-H broad, 3032 vs C-H Aromatic, 1659 vs Imine C=N, 1597. vs C=C Aromatic, 1528 NO2, 869H out of-plane* | *122* | *72* | *2* | *white* |
| 3b |  3-mercapto-2-(1-(4-nitrophenyl) ethylideneamino)propanoic acid | *2576 $v_s$ O-H broad, 3047$v_s$ C-H Aromatic, 2998$v_s$ C-H Aliphatic, 1672$v_s$ Imine C=N, 1579$v_s$ C=C,1573 NO2, Aromatic,674$v_s$ C-S, 889 H out of-plane* | *139-140* | *62* | *3* | *white* |
| 3c |  3-(methylthio)-2-(1-(4-nitrophenyl) ethylideneamino)propanoic acid | *2594 $v_s$ O-H broad ,3109$v_s$ C-H Aromatic, 1691 Imine $v_s$ C=N, 1521 C=C Aromatic, 1538 NO2, 1242 $v_s$ C-N, 854.-H out of-plane.* | *120-123* | *62* | *3* | *Red* |
| 3d |  2,6-bis(1-(4-nitrophenyl)ethy lideneamino)hexanoic acid | *2578 $v_s$ O-H broad , 3011 $v_s$ C-H Aromatic, 1690 $v_s$ Imine C=N, 1606$v_s$ Imine C=N 1520 $v_s$ C=C Aromatic, 1543 NO2, 1340 C-H bending, 854 out of-plane* | *185-188* | *75* | *2* | *Yellow* |
| 3h |  (2*S*)-3-(benzylideneamino)-2-(((2-(benzylideneamino)-2-carboxyethyl)disulfanyl)methyl)propanoic acid | *2849$v_s$ O-H broad, 3044 $v_s$ C-H Aromatic,1702$v_s$ C=O 1605$v_s$ Imine C=N, 1530 $v_s$ C=C Aromatic, 676$v_s$ C-S, 1324 C-H bending 849 H out of-plane* | *108-110* | *66* | *3* | *White* |

Table (2): The chemical formula, molecular weights, C.H.N %, yield, melting points, colors, and the reaction time, oxazolidin-4-one and Thiazolidine-4-one

| Code | 4a | 4b | 4c | 4d | 4e | 4f | 4g | 4h | 4i |
|---|---|---|---|---|---|---|---|---|---|
| Chemical Formula | $C_{15}H_{18}N_2O_6$ | $C_{15}H_{18}N_2O_6S$ | $C_{26}H_{28}N_4O_{10}$ | $C_{14}H_{16}N_2O_5S$ | $C_{26}H_{28}N_4O_8S_2$ | $C_{24}H_{24}N_4O_8S_2$ | $C_{26}H_{30}N_2O_4S_2$ | $C_{25}H_{26}N_2O_6S_4$ | $C_{12}H_{13}NO_3S_2$ |
| M.wt g\mole | 322.12 | 354.09 | 556.18 | 324.09 | 588.13 | 560.60 | 498.16 | 578.74 | 283.03 |
| C.H.N Cal.(Found) C % | 55.90 (56.043) | 50.84 (51.2 83) | 56.11 (55.723) | 51.84 (52.274) | 53.05 (52.728) | 51.42 (51.0 30) | 62.62 (62.479) | 51.88 (52.370) | 50.86 (51.294) |
| H% | 5.63 (6.13) | 5.12 (5.058) | 5.07 (4.526) | 4.97 (5.063) | 4.79 (5.436) | 4.32 (4.047) | 6.06 (5.839) | 4.53 (4.973) | 4.62 (4.493) |
| N% | 8.69 (9.292) | 7.90 (7.832) | 10.07 (9.622) | 8.64 (8.993) | 9.52 (10.033) | 9.99 (9.784) | 5.62 (5.826) | 4.84 (4.568) | 4.94 (5.241) |
| Yield | 65% | 88% | 65% | 77% | 82% | 91% | 90% | 80% | 80% |
| M.P | 178-180 | 123-125 | 134-137 | 180-183 | 100-102 | 160-162 | 105-107 | 90-93 | 203-206 |
| Color | white | white | Yellow | white | yellow | Red | white | white | yellow |
| Reaction | 6 hr. | 7 hr. | 4 hr. | 6hr. | 7 hr. | 6hr. | 7hr. | 7 hr. | 6hr. |

Table 3: Molecular structure, IR Characteristic Absorption, Chemical Shift δ ppm of oxazolidin-4-one and Thiazolidine-4-one 4(a-j).

| Comp. No. | Molecular structure | *I.R Characteristic Absorption Frequencies, $Cm^{-1}$* | *Chemical Shift ppm* |
|---|---|---|---|
| 4a |  3-methyl-2-(2-methyl-2-(4-nitrophenyl)-4-oxooxazolidin-3-yl)butanoic acid | *2587 –OH broad, 3095 $\nu_s$ C-H Aromatic , 1691 $\nu_s$ C=O amide, 1520 $\nu_s$ C=C Aromatic, 1242 $\nu_s$ C-N, 1521 $NO_2$, 855 H out of-plane.* | *1.85( s,9H –$CH_3$), 2.25 (s, 2H –$CH_2$ ) ,4.57 (t, 2H –CH) , 7.04-8.95 (d, 4H Aromatic Protons) 11.09 (s,1H –OH)* |
| 4b |  2-((2-methyl-2-(4-nitrophenyl)-4-oxooxazolidin-3-yl)methyl)-4-(methylthio)butanoic acid | *3274 $\nu_s$- OH broad , 3089 $\nu_s$ C-H Aromatic, 1691 $\nu_s$ C=O amide, 1521 $\nu_s$ C=C Aromatic, 1537 $NO_2$ 1242 $\nu_s$ C-N,865 -H out of-plane* | *1.58(s, 3H –$CH_3$) 2.25(s,3H-$CH_3$)2.57(d, 4H-$CH_2$) 4.09(s,2H-$CH_2$) 4.63(s, 1H –CH) 7.03-8.64 (m, 4H Aromatic Protons) 11.67 (s, 1H –OH)* |
| 4c |  2,6-bis(2-methyl-2-(4-nitrophenyl)-4-oxo oxazolidin-3-yl)hexanoic acid | *2848 $\nu_s$- OH broad, 3049 $\nu_s$ C-H Aromatic, 1660 $\nu_s$ C=O amide, 1553 $\nu_s$ C=C Aromatic, 1242 $\nu_s$ C-N, 1567 $NO_2$, 875 -H out of-plane* | *1.08(d, 6H- $CH_3$) 1.25(s, 2H –$CH_2$ ) 1.75 (s,2H-$CH_2$ ) 1.95(s, 2H –$CH_2$ ) 2.34(s, 2H- $CH_2$) 3.57(d, 4H-$CH_2$) 4.56(s,1H –CH) 7.25 -8.64 (m, 8H Aromatic Protons ) 11.25 (s, 1H –OH)* |
| 4d |  2-(3-(2,4-dinitrophenylamino)-4-oxothiazolidin-2-yl)-3-methylbutanoic acid | *2506 $\nu_s$ -OH broad, 3096 $\nu_s$ C-H Aromatic, 1675 $\nu_s$ C=O amide, 1537 $\nu_s$ C=C Aromatic, 1223 $\nu_s$ C-N, 1594 NO2, 898 H out of-plane* | *1.25(s, 6 H –$CH_3$) 2.43(s,1H-CH) 2.56(s,1H-CH) 3.57(d,2H-$CH_2$) 5.73(s,1H-CH) 7.05 -8.94(m, 4H Aromatic Protons ) 11.55 (s, 1H –OH)* |

| | | | |
|---|---|---|---|
| **4E** |  2,6-bis(2-methyl-2-(4-nitrophenyl) -4-oxothiazolidin-3-yl)hexanoic acid | ***2546$\nu_s$ -OH broad, 3046 $\nu_s$ C-H Aromatic, 1695 $\nu_s$ C=O amide, 1537 $\nu_s$ C=C Aromatic, 1283$\nu_s$ C-N, 1576 NO2, 878 H out of-plane*** | ***1. 14 (d, 6H- $CH_3$) 1.35 (s, 2H –$CH_2$) 1.92 (s,2H-$CH_2$) 2.03(s, 2H –$CH_2$) 2.58 (s, 2H-$CH_2$) 3.69 (d, 4H-$CH_2$) 4.37(s,1H –CH) 7.05 -8.96 (m, 8H Aromatic Protons) 11. 79 (s, 1H -OH)*** |
| **4F** |  2,6-bis(4,4-difluoro-2-methyl-2-(4-nitrophenyl)-5-oxooxazolidin-3-yl)hexanoic acid | ***2563 $\nu_s$ O-H broad, 3037 $\nu_s$ C-H Aromatic, 1690 $\nu_s$ C=O amide, 1275 C-N, 1483 C=C Aromatic 1331 C-H bending, 1537 NO2, 882 H out of-plane*** | ------- |
| **4g** |  2,6-bis(4,4-difluoro-3-methyl-2-(4-nitrophenyl)-5-oxooxazolidin-3-yl)hexanoic acid | ***2743 –OH broad, 30906 $\nu_s$ C-H Aromatic, 1670 $\nu_s$ C=O, 1571 $\nu_s$ C=C Aromatic, 1282 $\nu_s$ C-N, 894 H out of-plane*** | ---------- |
| **4h** |  (2S)-3-[(2-carboxy-2-(4-oxo-2-phenylthiazolidin-3-yl)ethyl) disulfanyl]-2-(4-oxo-2-phenylthiazolidin-3-yl)methyl]propanoic acid | ***25389 $\nu_s$ -OH broad, 3029 $\nu_s$ C-H Aromatic, 1691 $\nu_s$ C=O amide, 1524 C=C Aromatic 1384.08 C-H bending 857 H out of-plane*** | -------- |
| **4i** |  2-(4-(chloromethyl)-2-phenyloxazolidin-3-yl) -3-mercaptopropanoic acid | ***2685 –OH broad, 3023 $\nu_s$ C-H Aromatic, 1679 $\nu_s$ C=O , 1579 $\nu_s$ C=C Aromatic, 1296 $\nu_s$ C-N,1337 C-H bending, 845 -H out of-plane*** | ---------- |

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# تحضير وتشخيص بعض مشتقات أوكسازوليدين –4–اون و ثيازوليدين–4–اون جديدة من تفاعل قواعد شيف المشتقة من بعض الأحماض الأمينية

**هاجر حسام عباس عبيد حسن عبد**

**الخلاصة:**

حضرت مشتقات جديده من اوكسازوليدين –4–اون وثايازوليدين *4–اون* المعوضة في الموقع 2–,3– من تفاعل الغلق الحلقي بواسطة بالحامض الكلايكول والمركبتو و لمركبات شيف المختلفة بتصعيدها في ظروف جافه في مذيب دايوكسان الجاف. وقد حضرت الايمينات بالتكثيف الحراري المحفز بالحامض لمجموعة الامينية الاحماض الامنية مع مجموعة الكاربونيل في الالديهايدات والكيتونات الاروماتية في الكحول المثيلي المطلق. وقد شخصت النواتج من خلال درجات الانصهار ونسبه C.H.N المئوية والاطياف FTIR و$^{1}$HNMR لهذه المركبات.