



SYNTHESIS AND CHARACTERIZATION OF NEW THIAZOLIDINONES AND 2-OXOPYRROLIDINES DERIVED FROM SCHIFF BASES

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Keywords: thiazolidinones; five-membered γ -lactams; imines.

New thiazolidinones and γ -lactams were prepared from mixtures of Schiff base (imine) and thioglycolic acid or phenylsuccinic anhydride, respectively, in moderate yields (52-71 %). The structures of these new thiazolidinones and γ -lactams were established on the basis of the IR, ¹H-NMR, ¹³C-NMR, ¹³C-NMR DEPT and mass spectral data.

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Introduction

Thiazolidinones (Figure 1a) are classified as doubly unsaturated five-membered heterocyclic compounds contain one nitrogen, one sulfur and three carbon atoms including a carbonyl group. Thiazolidinones and their derivatives show a large variety of biological activities such as antibiotic, diuretic, tuberculostatic, organoleptic, antileukemic and antiparasitic.^{1,2} As far as literature is concerned, only a few information is available about thiazolidinones and their bioactivity. The chemistry of thiazolidin-4-one ring system is considerable interest because it is the core structure in various pharmaceuticals.

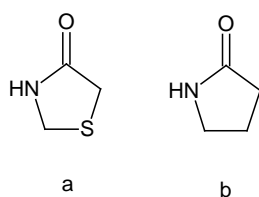


Figure 1. Structure of thiazolidinones and 2-oxopyrrolidines (γ -lactams).

Five-membered ring lactams, which are known as γ -lactams or 2-oxopyrrolidines (Figure 1b), are essential structural motifs in biologically active natural products and used in medicines and approved drugs.³ γ -Lactams have attracted considerable attention in recent years because they are valuable building blocks in the structure of several biologically active molecules.⁴ Substituted γ -lactams, in particular, have potential application in drug synthesis, but the development of the stereoselective synthesis of chiral γ -lactams remains a challenge.^{5,6} Various γ -lactams are components of natural products,⁷ and some biologically important lactams⁸ are obtained from the reaction of imines with phenylsuccinic anhydride.

Experimental part

All solvents were distilled/dried prior to use, whenever this seemed necessary, by standard methods. All solvent extracts were dried over anhydrous sodium sulfate unless otherwise specified.

FT-IR spectra were recorded using a Shimadzu FT-IR spectrophotometer as KBr. The absorption bands of interest are reported and expressed in cm^{-1} .

¹H-NMR spectra were recorded using a Bruker Varian NMR spectrometer (500 MHz). The chemical shift values are expressed in δ (ppm), using tetramethylsilane (TMS) as internal standard and DMSO- d_6 as a solvent. ¹³C-NMR spectra and ¹³C-NMR DEPT spectra were recorded using a Bruker Varian spectrometer (75 MHz). The chemical shift values are expressed in δ (ppm), using tetramethylsilane (TMS) as internal standard and CDCl_3 as a solvent.

Mass spectra were recorded using a 70 eV HPLC-LCQ Fleet/Thermo Scientific instrument with 5973 type mass selective detector.

General procedure for preparation of imines

In general, the imines (**2a-2d**) were prepared by reaction the corresponding amines with an aldehyde or a ketone in 40 mL of methanol and 4-6 drops of glacial acetic acid with refluxing the reaction mixtures for 1-5 h under stirring. The progress of the reaction is followed by TLC. After completion the reaction, the solvent was evaporated then the residue was recrystallized from a suitable solvent. The physical data of the prepared imines (**2a-2f**) are gathered in Table 1.

3-Bromo-2-(pyridin-2-yliminomethyl)phenol (**2a**)

This compound was prepared by reacting of 2-aminopyridine (0.01 mol, 1 g) with 5-bromo-2-hydroxybenzaldehyde (0.01 mol, 2.4 g). $R_f=1.2$. Yield = 79.6 %, m.p. = 138-139 °C. IR (KBr disk): 1610 cm^{-1} (C=N).

3-Bromo-2-(pyridin-3-yliminomethyl)phenol (2b)

This compound was prepared by reacting of 3-aminopyridine (0.01 mol, 1 g) with 5-bromo-2-hydroxybenzaldehyde (0.01 mol, 2.4 g). $R_f=0.4$, yield was 81.6 %, m.p. = 125-126 °C. IR (KBr disk): 1615 cm^{-1} (C=N).

4-(5-Aminonaphthalenylimino)pentan-2-one (2c)

This compound was prepared by reacting of 1,5-diamino naphthalene (0.006 mol, 1 g) with acetylacetone (0.006 mol, 0.63 g, 0.65 ml). $R_f=0.5$, Yield = 52.6 %, m.p = 200 °C. IR (KBr disk): 1612 cm^{-1} (C=N).

4-(4-Aminophenylimino)pentan-2-one (2d)

This compound is prepared by reacting of p-phenylenediamine (0.0099 mol, 1 g) with acetylacetone (0.0099 mol, 0.93 g, 0.95 ml). $R_f=0.5$, yield = 90.9 %, m.p = 94-95 °C. IR (KBr disk): 1601 cm^{-1} (C=N).

4-(Pyridin-3-ylimino)pentan-2-one (2e)

This compound was prepared by reacting of 3-aminopyridine (0.01 mol, 1 g) with acetylacetone (0.01 mol, 1.06 g, 1.09 ml). $R_f=2$, yield = 93.5 %, b.p. = 129-132 °C. IR (KBr disk): 1612 cm^{-1} (C=N).

3-(4-Chlorobenzylideneimino)pyridine (2f)

This compound was prepared by reacting of 3-aminopyridine (0.006 mol, 1 g) with p-chlorobenzaldehyde (0.006 mol, 2.54 g). $R_f=2$, yield = 97 %, m.p = 73-74 °C. IR (KBr disk): 1623 cm^{-1} (C=N).

General procedure for preparation of thiazolidinones (3)

A mixture of compound **2a-2d** and thioglycolic acid in chloroform (15) ml was allowed to react in a Teflon beaker in a microwave oven at 100 W for 6-12 minutes. Progress of the reaction is checked by TLC using hexane-ethyl acetate as eluent. After the completion of reaction, chloroform was removed by distillation to give a solid residue. The solids were washed successively with 1 N HCl (20 mL), water (2×20 mL), 5% NaHCO_3 (20 mL) and brine (20 mL). The organic layer was dried with Na_2SO_4 . The solvent was removed by evaporation. The following thiazolidinones were prepared:

2-(2-Bromo-6-hydroxyphenyl)-3-(pyridin-2-yl)-thiazolidin-4-one (3a)

This compound was prepared by reacting **2a** (0.003 mol, 1 g) and (0.003 mol, 0.33 g, 0.25 mL) of thioglycolic acid. $R_f=0.7$, yield = 63 %, m. p. = 88-89 °C. IR (KBr disk): 1673 cm^{-1} (–N–C=O), 1727 cm^{-1} (HO–C=O). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6 , δ , ppm) 3.81(d, 1H), 4.19(d, 1H), 5.68(s, 1H), 6.15-7.51 (m, 3H), 6.09-8.12 (m, 4H) and 10.21 (s, 1H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , δ , ppm) 38.65, 48.02, 122.03-151.02 and 177.86

2-(2-Bromo-6-hydroxyphenyl)-3-(pyridin-3-yl)thiazolidin-4-one (3b)

This compound was prepared by reacting **2b** (0.003 mol, 1 g) and (0.003 mol, 0.33 g, 0.25 mL) of thioglycolic acid. $R_f=1.2$, yield = 71 %, m. p. = 98-99 °C. IR (KBr disk): 1672 cm^{-1} (–N–C=O). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6 , δ , ppm) 3.91(d, 1H), 4.31(d, 1H), 5.61(s, 1H), 6.22-7.51 (m, 3H), 6.01-8.12 (m, 4H) and 10.26 (s, 1H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , δ , ppm) 42.96, 52.12, 128.13-133.30, 135.95-151.34 and 180.52.

3-(5-Aminonaphthalen-1-yl)-2-methyl-2-(2-oxopropyl)-thiazolidin-4-one (3c)

This compound was prepared by reacting **2c** (0.001 mol, 0.41 g) and (0.001 mol, 0.157 g, 0.12 mL) of thioglycolic acid. $R_f=0.6$, Yield = 69 %, m. p. = 139-140 °C. IR (KBr disk): 1676 cm^{-1} (–N–C=O). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6 , δ , ppm) 3.88 (d, 1H), 4.07 (d, 1H), 1.90 (s, 3H), 2.79 (s, 3H), 4.29 (s, 2H), 4.60 (s, 2H) and 7.30-8.02 (m, 6H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , δ , ppm) 40.80, 61.85, 18.28, 26.78, 36.95, 124.44-156.76, 179.17 and 177.86.

3-(4-Aminophenyl)-2-methyl-2-(2-oxopropyl)-thiazolidin-4-one (3d)

This compound was prepared by reacting **2d** (0.005 mol, 1 g) and (0.005 mol, 0.48 g, 0.36 mL) of thioglycolic acid. $R_f=1.2$, Yield = 57 %, m. p. = 86-87 °C. IR (KBr disk): 1681 cm^{-1} (–N–C=O). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6 , δ , ppm) 3.98 (d, 1H), 4.25 (d, 1H), 1.89 (s, 3H), 2.85 (s, 3H), 4.65 (s, 2H), 4.75 (s, 2H) and 7.55-7.73 (m, 4H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , δ , ppm) 18.38, 26.75, 35.92, 40.82, 61.55, 124.66-152.78, 178.15 and 201.15.

General procedure of γ -lactams (4)

In general the γ -lactam were prepared by reaction the mixture of imines **2a**, **2b**, **2e** and **2f** with phenylsuccinic anhydride in 20 mL of chloroform, then the mixture was refluxed for 1-12 h with stirring. The progress of the reaction was followed by TLC. After completion the reaction, the solvent was evaporated and the residue was recrystallized from ethanol. The following γ -lactams were prepared:

2-(2-Bromo-6-hydroxyphenyl)-5-oxo-3-phenyl-1-(pyridin-2-yl)pyrrolidine-3-carboxylic acid (4a)

This compound was prepared by reacting **2a** (0.003 mol, 1 g) and (0.003 mol, 0.64 g) of phenylsuccinic anhydride. $R_f=0.7$, yield = 55 %, m. p. = 121-122 °C. IR (KBr disk): 1638 cm^{-1} (–N–C=O), 1727 cm^{-1} (HO–C=O). $^1\text{H-NMR}$ (500 MHz, DMSO-d_6 , δ , ppm) 3.28 (d, 1H), 3.52 (d, 1H), 4.12 (s, 1H), 6.15-6.70 (m, 5H), 7.63-8.35 (m, 7H), 10.31 (s, 1H) and 11.42 (s, 1H). $^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , δ , ppm) 42.88, 53.02, 59.79, 121.23-158.52, 125.95-135.95, 127.89-138.11, 172.13 and 185.88.

2-(2-Bromo-6-hydroxyphenyl)-5-oxo-3-phenyl-1-(pyridin-3-yl)pyrrolidine-3-carboxylic acid (4b)

This compound was prepared by reacting **2b** (0.003 mol, 1 g) and (0.003 mol, 0.64 g) of phenylsuccinic anhydride. $R_f=0.6$, yield = 61 %, m. p. = 113-114 °C. IR (KBr disk): 1655 cm^{-1} ($-\text{N}-\text{C}=\text{O}$), 1719 cm^{-1} ($\text{HO}-\text{C}=\text{O}$). $^1\text{H-NMR}$ (500 MHz, $\text{DMSO}-d_6$, δ , ppm) 3.51 (d, 1H), 3.81 (d, 1H), 4.55 (s, 1H), 6.08-6.54 (m, 5H), 7.18-8.02 (m, 7H), 9.98 (s, 1H) and 11.40 (s, 1H). ^{13}C NMR (75 MHz, CDCl_3 , δ , ppm) 41.56, 51.07, 57.96, 120.53-152.74, 124.45-133.90, 171.25 and 178.25.

2-(2-Bromo-6-hydroxy-phenyl)-5-oxo-3-phenyl-1-(pyridin-3-yl)pyrrolidine-3-carboxylic acid (4e)

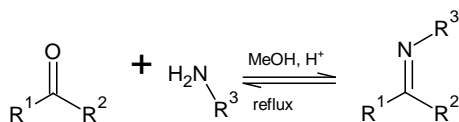
This compound was prepared by reacting **2e** (0.005 mol, 1 g) and (0.005 mol, 1 g) of phenylsuccinic anhydride. $R_f=0.5$, yield = 68.4 %, m. p. = 168-169 °C. IR (KBr disk): 1602 cm^{-1} ($-\text{N}-\text{C}=\text{O}$), 1697 cm^{-1} ($\text{HO}-\text{C}=\text{O}$). ^1H NMR (500 MHz, $\text{DMSO}-d_6$, δ , ppm) 3.25 (d, 1H), 3.56 (d, 1H), 1.91 (s, 3H), 2.28 (s, 3H), 3.98 (s, 2H), 6.21-8.08 (m, 9H), and 11.07 (s, 1H). ^{13}C NMR (75 MHz, CDCl_3 , δ , ppm) 16.02, 27.23, 37.84, 41.12, 52.11, 59.23, 121.66-157.17, 125.33-139.17, 168.94, 180.73 and 206.85.

2-(4-Chlorophenyl)-5-oxo-3-phenyl-1-(pyridin-3-yl)pyrrolidine-3-carboxylic acid (4f)

This compound was prepared by reacting **2f** (0.0046 mol, 1 g) and (0.0046 mol, 0.8 g) of phenylsuccinic anhydride. $R_f=0.1$, yield = 55 %, m. p. = 159-160 °C. IR (KBr disk): 1602 cm^{-1} ($-\text{N}-\text{C}=\text{O}$), 1695 cm^{-1} ($\text{HO}-\text{C}=\text{O}$). ^1H NMR (500 MHz, $\text{DMSO}-d_6$, δ , ppm) 3.32 (d, 1H), 3.56 (d, 1H), 4.53 (s, 1H), 6.21-6.64 (m, 5H), 7.30-8.20 (m, 7H), and 11.40 (s, 1H). ^{13}C NMR (75 MHz, CDCl_3 , δ , ppm) 40.13, 51.28, 58.54, 121.67-151.66, 124.56-138.54, 169.18 and 183.05.

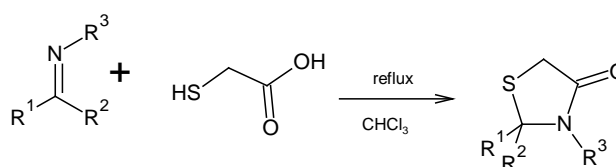
Results and discussion

The Schiff bases are formed by the condensation of primary amines and an aldehyde or ketone.



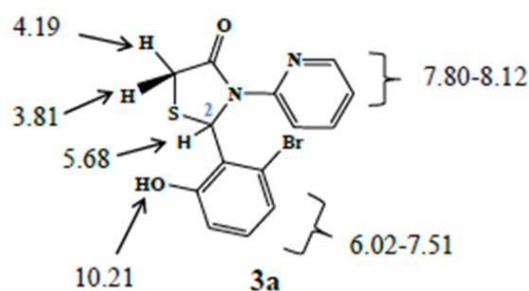
A simple synthetic way to prepare the biologically active thiazolidinones^{9,10} is based on the reaction of imines with thioglycolic acid:

The IR spectra of imines **2a-2f** made is characterized by four principal band groups correspond to the stretching vibrations of the aromatic C-H bonds, aliphatic C-H bonds, azomethine bonds (C=N), and aromatic C=C bonds of the and substituted aromatic ring, which occur within the ranges of 3224-3047, 3007-2777, 1638-1610, and 1586-1475 cm^{-1} , respectively.

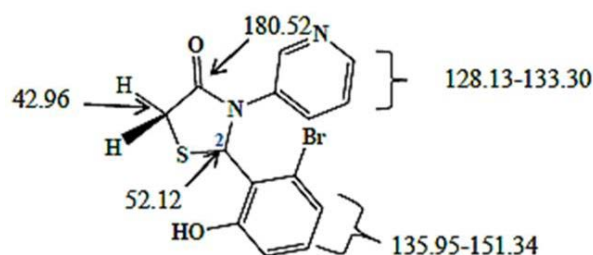


Six band groups can characterize the IR spectra of thiazolidinones **3a-3d** correspond to the stretching vibration of the aromatic C-H, aliphatic C-H, carbonyl amide group ($\text{N}-\text{C}=\text{O}$), aromatic C=C bonds, C-N bonds, and bending vibration of C-S bond, which occurs within the ranges of 3369-3050, 2946-2800, 1720-1672, 1631-1546, 1282-1271 and 778-626 cm^{-1} , respectively.

The $^1\text{H-NMR}$ spectrum of 2-(2-bromo-6-hydroxyphenyl)-3-(pyridin-2-yl)thiazolidin-4-one (**3a**) (see Electronic Supporting Information) is evaluated as an example. The signal at chemical shift δ 2.50-2.51 ppm belongs to the $\text{DMSO}-d_6$ solvent. There are two doublet signals at chemical shift δ 3.81 ppm ($J=3$ Hz) and δ 4.19 ppm ($J=3$ Hz) for methylene protons of thiazolidin-4-one ring (Fig.1). The chiral carbon atom (No.2) gives a singlet signal at δ 5.68 ppm (racemic mixture (R, S) configuration) and there is a multiple signal system for three protons of the phenol ring at δ 6.02-7.51. A multiplet signal for four protons of pyridine ring at δ 7.80-8.12 ppm also appears.

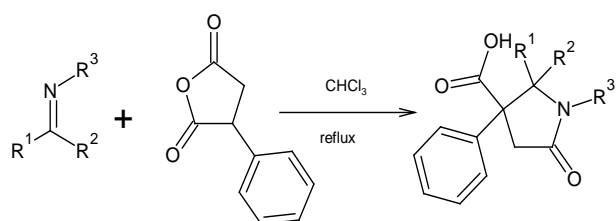


The ^{13}C NMR spectrum of 2-(2-bromo-6-hydroxyphenyl)-3-(pyridin-3-yl)thiazolidin-4-one (**3a**) as example (see Electronic Supplementary Information) shows a signal at δ 76.34-76.96 ppm for the CDCl_3 solvent. There are two singlets at δ 42.96 ppm and 52.12 for the methylene group carbon and No.2 carbon of the thiazolidin-4-one ring. A multiplet signal between δ 128.13-151.34 ppm for aromatic carbon atoms (pyridine + phenol) ring and a singlet signal at δ 180.52 ppm for the carbon of the amide carbonyl group are also identified.



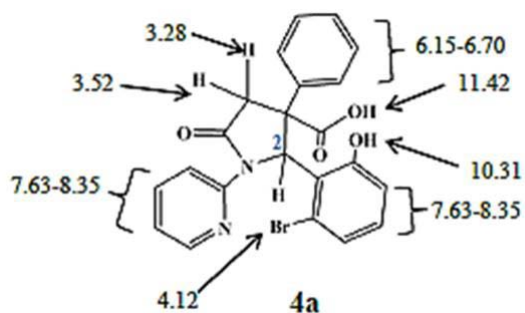
The ^{13}C -NMR DEPT spectrum of **3a** shows a signal at δ 48 (-) ppm for (CH_2), and a signal at δ 38(+) ppm for CH carbon of thiazolidin-4-one ring. There are signals belong to the aromatic region in the range (125-135) (+) ppm and there are signals at δ 122-148 (+) and (177) (+) ppm for the pyridine ring and the amide carbonyl.

γ -Lactams also represent important substructures for the synthesis of biologically relevant compounds in drug discovery¹¹ and natural products.^{12,13} The prevalence of these structures^{14,15} resulted in the development of several efficient methods¹⁶ and preparation of diverse libraries of small molecules for biological evaluation.^{17,18} Based on these earlier studies, a practical way, the cyclization of imines with phenylsuccinic anhydride in chloroform was followed:



Bands characterize the IR spectra of γ -lactams **4a**, **4b**, **4e** and **4f** belong to the stretching vibrations of the carboxylic OH, aromatic C-H, aliphatic C-H, carboxylic carbonyl group, carbonyl amide group, aromatic C=C and substituted aromatic ring in the ranges of 3134-3026, 3064-2742, 1727-1695, 1651-1586, 1602-1536 and 939-809 cm^{-1} , respectively.

The ^1H -NMR spectrum of 2-(2-bromo-6-hydroxyphenyl)-5-oxo-3-phenyl-1-(pyridin-2-yl)pyrrolidine-3-carboxylic acid (**4a**) (see Electronic Supplementary Information) showed signal at δ 2.57-2.58 ppm belongs to DMSO- d_6 solvent. Two doublet signals are appeared at δ 3.28 ppm and 3.52 ppm, ($J=4$ Hz) for methylene protons of pyrrolidine ring, a singlet at δ 4.12 ppm for the proton of the chiral carbon (No 2) atom (racemic), and two multiplet signals for the five protons of benzene ring at δ 6.15-6.70 and δ 7.63-8.35 for seven aromatic protons of phenol and pyridine rings, respectively.



There are two singlet signals at δ 10.31 ppm and 11.42 ppm of phenol and carboxyl hydroxy groups, respectively.

The mass spectrum of **4a** showed the molecular ion peak in 453,455 m/z and important fragmentation peaks at $m/z=452$, 454 $m/z=424$, 426, m/z , 399, 401, $m/z=382$, 384, $m/z=354$, 356 m/z (these fragments contains two bromine isotopes), and fragments without bromine isotopes at m/z , 223, 181 103 78, 77 and 65.

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Received: 11.12.2018.

Accepted: 01.01.2019.