Synthesis of Some New 2-thioxoimidazolidin-4-one Derivatives (part I)

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Abstract

This research includes the synthesis of new series of heterocyclic compounds. Reaction of 2-nitro benzylidene)thiosemicarbazide(1) with ethyl chloro acetate gave (2-nitro benzylidene amino)-2-thioxomidazolidine-4-one(2) ,treatment(2) with methyl iodide to give(3)which was reacted with hydrazine to give 2-hydrazinyl-1-[(2-nitrobenzylidene)amino]-1H-imidazol-5(4H)-one, andreation of compound(2) with aromatic aldehydes to give 5-arylidene -3-({2-nitro benzylidene}amino)2-thioxo-3,5-dihydro-4H-imidazole-4-one(5a,5b), which was reacted with ethyl aceto acetate to give 4-aryl-1-[2-nitrobenzylidene, amino -6-oxo-2-thioxo octa hydro-1H-benzo[d]imidazole-5-carboxylate and followed synthesis of β-lactamederivtives(9a,9b) by treatment derivatives(8a,8b)schiff bases with chloro acetyl chloride .some new compounds were characterized by of some protone NMR of them and IR spectroscopy.

Keywords: thiosemicarbazone, chalcone , beta lactone, imidazolidin-4-one .

Introduction

Thiosemicarbazones was initially used in the synthesis of antibacterial sulfathiazole . Destined for the synthesis of a certain thiosemicarbazone, Activity powerful tuberculosis brake of the precursor that has become a major drug tuberculosis ants [1] thiosemicarbazones derivatives are an important class of natural products belonging to the family flavonoids revealed [2] ,Which have been reported to possess awide range of biological activities,including anti-bacterial[3] ,an anti-fungal [4] , anti-inflammatory [5] , anti tumor[6] , and anti-mutation [7] . Additionally, it was found on some of thiosemicarbazones derivatives to prevent many important enzymes in cellular systems, such as xanthine oxidase [8] protein tyrosine kinase [9]One strategy that has been used for the synthesis of new acridine derivatives involves combining of loop acridine with a different moiety and work independently in a single hybrid compound covalently linked, using for example the nucleus of thiazolidine or imidazolidine [10].the are also β-lactame derivatives known to exhibit various biological activities like antibiotic,anti fungal and anti- inflammatory activities[11].

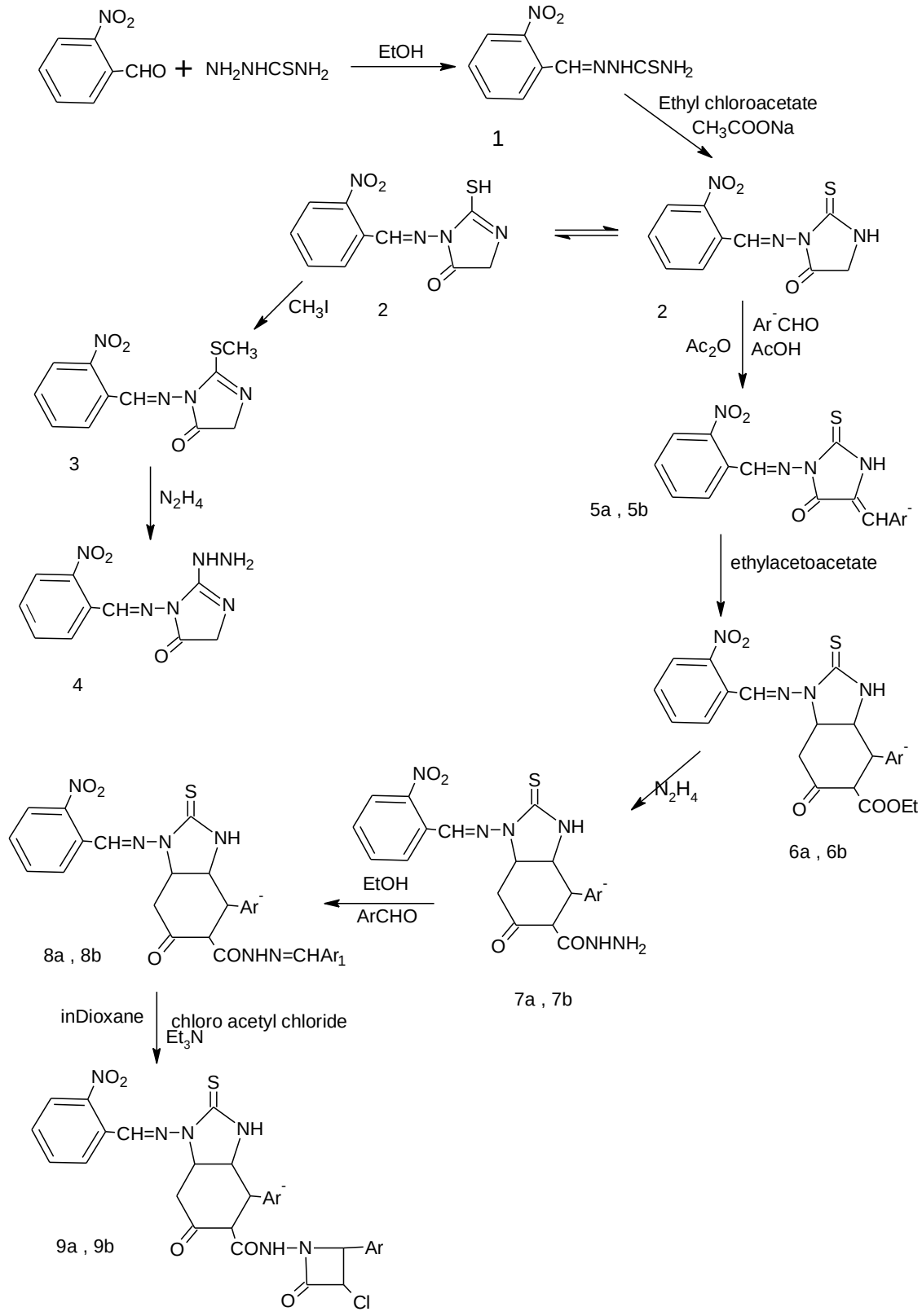
The Aim of work:

1-Synthesis of substrat Schiff base 2- nitro benzylidene)thiosemicarbazide.

2-Synthesis of chalconesderivtives -(arylidene)-3-[(2-nitrobenzylidene)amino]-2-thioxo -3,5-dihydro-4H-imidazol-4-one

3-Synthesis of hydrazidederivtives

4- Synthesis of beta lactamederivtives



Ar^- = 2 -NO_2 - C_6H_4 , p - Br - C_6H_4
Ar = p - Br - C_6H_4

Scheme 1

4.1. General

The IR Spectra were recorded by KBr discs using a Perkin-Elmer 1600 series FTIR spectrometer. 1HNMR Spectra were recorded on a Varian-Mercury 300 MH_Z Spectrometer. melting points were determined on ahot stage Gallen kamp melting point apparatus and are uncorrected..

Preparation of 2-(2-nitrobenzylidene)hydrazine carbothioamide (1)

To a mixture of 2-nitro benzaldehyde (0.01 mole, 1.51g) and thiosemicarbazide (0.01 mole) in ethanol (30mL) was refluxed for 3 hrs according to literature procedure[12]. After that, mixture was poured onto ice cold water and stirred for 5 min. The product thus obtained was recrystallized from (Ethanol). (Yield 62 %), (m.p, 162-164),FTIR. (ν,cm^{-1})3425,3246,(NH_2), 3159(NH),

1560 ,1539(C=C of aromatic.), 1602 (C=N), 1518,1338 (NO_2), ^{1}HNMR (DMSO-d6)(δ,ppm) 11.98 (NH), 8.62 (N=CH), 8.55- 7.81 (m, 4H, ArH), 3.67 (s, NH_2).

Synthesis of 3-[(2-nitrobenzylidene)amino]-2- thioxoimidazolidine--4-one (2)

A solution of compound (1) (0.01 mole, 2.42g) in ethanol (50 mL) with ethyl chloroacetate (0.01 mole) in presence of anhydrous sodium acetate(0.03 mole,0.246g) was refluxed for (20 hrs.) After that, the excess solvent was removed through distillation the resulting solid poured onto crash ice and stirred for 30 min and filtered , recrystalliztion from (Ethanol). (Yield 65 %), (m.p, C°195-197),),FTIR. (ν ,cm^{-1}) 3100 (NH) ,3088 (C-H aromatic), 2939 (CH aliph.), 1732 (C=O), 1604-1527 (C=Car.), 1633 (C=N), 1581,1350

(NO_2), ^{1}HNMR (DMSO-d6)(δ,ppm) 12.33 (s,2H,NH), 8.89 (N=CH), 7.92-8.31 (m, 4H, ArH), 4.23 (CH_2).

Synthesis of 2-(methylthio)-1-[(2-nitrobenzylidene)amino]-3,5-dihydro-4*H*-imidazol-4-one(3)

To a mixture of compound (2) (0.01 mole, 2.42g) and methyl iodide (0.01 mole,0.142g) in ethanol (30 mL) in presence of anhydrase potassium carbonate was refluxed for (4 hrs.) After completion of reaction it was poured onto 250 ml ice cold water kept for some time ,the solid formed was recrystallized from (Ethanol). (Yield 55 %), (m.p, C°185-187),),FTIR. (ν,cm^{-1}) 3101 (NH) ,3064 (CHar.), 2937, 2876 (CH aliph.), 1730 (C=O), 1604-1500 (C=Car.), 1631 (C=N), 1525,1350 (NO_2).

Synthesis of 2-hydrazinyl-1-[(2-nitrobenzylidene) amino]- 1H-imidazol-5(4H)-one (4

To a reaction mixture of compound (3) (0.01 mole, 2.72 g) with hydrazine hydrate (0.01 mole ,99%) in 100 mL round bottom flask in ethanol (30mL) was added , the reaction mixture was refluxed for (8 hrs.) After that, mixture was poured onto cold water and stirred for 5 min. The product was filtered and recrystallized from (Ethanol). (Yield 55 %), (m.p, C°,141-143),FTIR. (ν,cm^{-1}) 3458, 3417 (NH_2), 3210 (NH) ,3043 (CHar.), 2991,2876 (CH aliph.), 1720 (C=O), 1599-1521 (C=Car.), 1624 (C=N), 1552,1344 (NO_2).

Synthesisof(5)-5-(arylidene)-3-[(2-nitrobenzylidene)amino]-2-thioxo -3,5-dihydro-4*H*-imidazol-4-one (5a, 5b)

A mixture of compound (2) (0.01 mole, 2.42g) and aromatic aldehyde (0.01 mole) in acetic acid (5 mL) and acetic anhydride (20 mL) were reflaxed for (4 hrs.) After that, mixture was stirred for 30min. The precipitate was collected and purified from appropriate solvent.

(5)-5-(4-bromobenzylidene)-3-[(2-nitrobenzylidene)amino]-2-thioxo -3,5-dihydro-4H-imidazol-4-one(5a):

(Methanol) (Yield 50 %), (m.p, C°,220-222),FTIR. (ν ,cm^{-1}) 3277 (NH) 3134 (=CH), 3039 (CHar.), 2947,2862(CH aliph.), 1714 (C=O), 1593-1519 (C=Car.), 1632 (C=C alkene) , 1643 (C=N), 1550,1323 (NO_2), 687 (C-Br), ^{1}HNMR (DMSO-d6) (δ,ppm) 11.34 (NH), 8.4 (N=CH) 7.88-8.23 (m, 8H, ArH), 7.67 (s, C=CH).

(5)-5-(2-bromobenzylidene)-3-[(2-nitrobenzylidene)amino]-2-thioxo -3,5-dihydro-4H-imidazol-4-one(5b)

(Ethanol) (Yield 60 %), (m.p, C°,214-216),),FTIR. (ν,cm^{-1}) 3435 (NH), 3105 (NH) , 3037 (CHar.), 2933 ,2871(CH aliph.), 1724 (C=O), 1602-1485 (C=Car.), 1645 (C=C alkene), 1660 (C=N), 1533,1359 (NO_2), ^{1}HNMR (DMSO-d6)(δ,ppm) 11.03 (NH), 8.24 (N=CH), 7.86 -8.21- (m, 8H, ArH), 7.66 (s, C=CH).

Synthesis of ethyl 4-aryl-1-[(2-nitrobenzylidene)amino]-6-oxo-2-thioxo octa-hydro-1H-benzo[d]imidazole-5-carboxylate (6a , 6b)

A mixture of compound (5a , 5b) (0.01 mole) and ethyl acetoacetate (0.01 mole) in ethanol (20 mL) and sodium hydroxide (0.02 mole) was reflaxed for (6 hrs.) with constant stirring [13]the crude product was obtained after cooling the reaction mixture and removing excess of solvent under reduced pressure.the crude product was recrystallized from appropriate solvent.

Ethyl 4-(4-bromo phenyl)-1-[(2-nitrobenzylidene)amino]-6-oxo-2-thioxo octa-hydro-1H-benzo[d]imidazole-5-carboxylate(6a)

(DMF) (Yield 76 %), (m.p, C°,205-207), FTIR. (ν,cm^{-1}) 3275 (NH) , 3060 (CHar.), 2982, 2877 (CH aliph.), 1734 (C=O ester), 1716 (C=O ketone), 1602-1500 (C=Car.), 1649 (C=N), 1529,1327 (NO_2), 690(C-Br)

Ethyl 4-(2-nitro phenyl)-1-[(2-nitrobenzylidene)amino]-6-oxo-2-thioxo octa-hydro-1H-benzo[d]imidazole-5-carboxylate(6b)

(Ethanol) (Yield 71 %), (m.p, C°,223-225),), FTIR. (ν,cm^{-1}) 3427 (NH) , 3086 (C-H of aromatic.), 2980, 2899 (CH aliph.), 1722 (carbonyl ester), 1714 (C=O ketone), 1587-1514 (C=Car.), 1668 (C=N), 1550,1371 ($NO_{2)}$

Synthesis of 4-aryl-1-[(2-nitrobenzylidene) amino]-6-oxo-2-thioxo octa hydro -1*H*-benzo[d]imidazole-5-carbohydrazide (7a ,7b)

To a mixture of compound (6a, 6b) (0.01 mole) and hydrazine hydrate (0.01 mole) in ethanol (30mL) was refluxed for (5 hrs.) with constant stirring [14] ,the reaction mixture was allowed to cool at room temperature and stirred for 5 min. The crude product was separated by filtration, and purification by ethanol.

4-(4-bromo phenyl) -1-[(2-nitrobenzylidene) amino]-6-oxo-2-thioxo octa hydro -1H-benzo[d]imidazole-5-carbohydrazide (7a)

(Ethanol) (Yield 65 %), (m.p, C°,228-230),), FTIR. (ν,cm^{-1}) 3455, 3336(NH_2), 3154 (NH) , 3082 (CHar.), 2960, 2871 (C-H aliphatic.), 1691 (carbonyl amide), 1721 (C=O ketone), 1610-1510 (C=C of aromatic) 1656 (C=N), 1554,1377 (NO_2), 680 (C-Br).

4-(4-bromo phenyl) -1-[(2-nitrobenzylidene)amino]-6-oxo-2-thioxo octa hydro -1H-benzo[d]imidazole-5-carbohydrazide (7b)

(Ethanol) (Yield 71 %), (m.p, C°,231-233), FTIR. (ν,cm^{-1}) 3346, 3318 (NH_2), 3127 (NH) , 3068 (CHar.), 2978,2843 (C-H aliphatic.), 1686 (C=O amide), 1717 (carbonyl ketone), 1601-1499 (C=Car.), 1657 (C=N), 1543,1351 (NO_2).

Synthesis of 4-Aryl-*N*'-arylidene-1-[(2-nitrobenzylidene)amino]-6-oxo-2-thioxo octa hydro-1*H*-benzo[d]imidazole-5-carbohydrazid (8a , 8b)

A mixture of compound (7a, 7b) (0.01 mole) and 4-bromo benzaldehyde (0.01 mole,1.85g) in ethanol (30mL) resulted mixture was refluxed for (5 hrs.) After completion of reaction ,water was added,and the resulted precipitate was filtered off ,washed with water and recrystallized from ethanol.

4-(4-bromophenyl)-N'-4-bromo benzylidene -1-[(2-nitrobenzylidene)amino]-6-oxo-2-thioxo octa hydro-1H-benzo[d]imidazole-5-carbohydrazide(8a)

(Ethanol) (Yield 60 %), (m.p, C°,177-179), FTIR. (KBr,cm^{-1}) 3416(NH amide), 3273 (NH, imidazole) , 3054 (C-H aromatic.), 2985, 2867 (CH aliph.), 1637 (carbonyl amide), 1726 (C=O ketone), 1600-1503 (C=C of aromatic.), 1620 (C=N), 1541,1336 (NO_2), 657(C-Br)

4-(2-bromophenyl)-N'-4-bromo benzylidene -1-[(2-itrobenzylidene)amino]-6-oxo-2-thioxo octa hydro-1H-benzo[d]imidazole-5-carbohydrazide(8b)

(Ethanol) (Yield 61 %), (m.p, C°,165-167), FTIR. (KBr,cm^{-1}) 3308(NH amide), 3234 (NH, imidazole) , 3088 (CHar.), 2928, 2879 (C-H aliphatic.), 1656 (C=O amide), 1713 (carbonyl ketone), 1602-1500 (C=C of aromatic.), 1616 (C=N), 1550,1330 (NO_2).

Synthesis of *N*-(3-chloro-2-aryl-4-oxoazetidin-1-yl)-1-(2-nitrobenzylideneamino)-4-aryl-6-oxo-2-thioxooctahydro-1H-benzo[d]imidazole -5-carboxamide (9a , 9b)

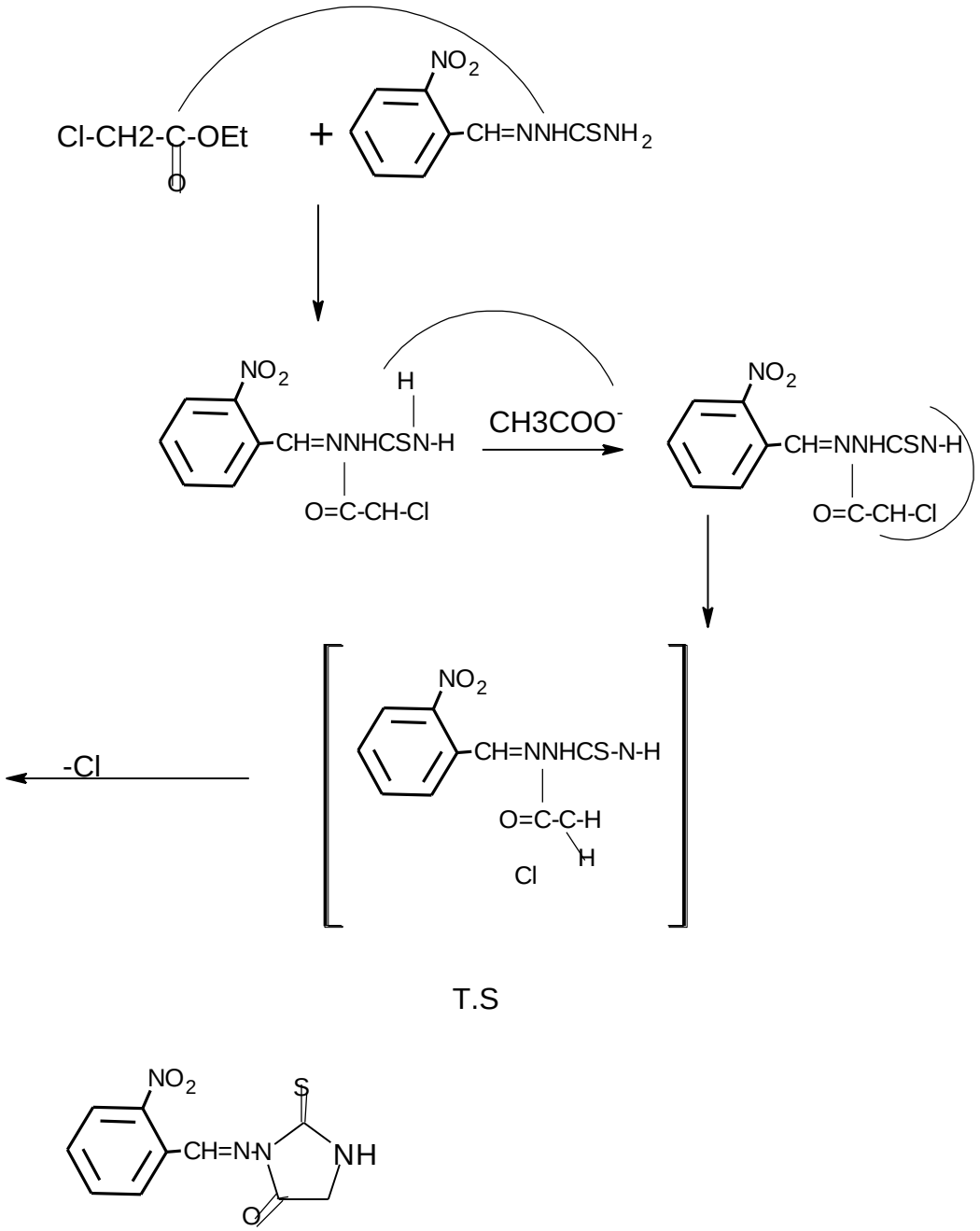
To a stirred solution of compound (8a, 8b) (0.01 mole), and triethyl amine (0.02 mole) in dioxane (30mL), chloro acetyl chloride(0.02 mole)was added dropwise at(0-5C). the reaction mixture was refluxed for (9 hrs.)[15] and the mixture was poured onto ice cold water and stirred for 5 min. The precipitate was obtained and purified from benzene.

N-(3-chloro-2-(4-bromo phenyl)-4-oxoazetidin-1-yl)-1-(2-nitrobenzylideneamino)-4-(4-bromo phenyl)6-oxo-2-thioxooctahydro-1H- benzo[d]imidazole -5-carboxamide (9a)

(Benzene) (Yield 55 %), (m.p, C°,247-249), FTIR. (ν,cm^{-1}) 3379(NH amide), 3226 (NH, imidazole) , 3051 (CHar.), 2983, 2861 (CH aliph.), 1734 (C=O beta lactame), 1683 (C=O amide), 1701 (carbonyl ketone), 1602-1500 (C=C ofaromatic.), 1651 (C=N), 1558,1363 (NO_2), 698 (C-Br), 790 (C-Cl).

Results and Discussion

Schemes (1) summarized the synthesis of different thiosemicarbazone derivatives **(1)** which was synthesized by treatment of thiosemicarbazide with 2-nitrobenzaldehyde[16]. The reaction is followed by the disappearance of the carbonyl group of aldehyde and the appearance of the new (C=N) band at (1602 cm^{-1}) and bands at (3425cm^{-1}) and at (3246 cm^{-1}) for stretching vibration of (NH_2 group) and the FTIR. Spectrum show other bands, 3159(NH) , 3028 (CHar.), 2978 (CH aliph.), 1580-1539 (C=Car.), 1518,1338 (NO_2)figure(1), the ^{1}HNMR spectrum shows signals at δ11.98 ppm(NH),δ8.62ppm (=CH),
δ7.81 -8.55 ppm (m, 4H, ArH), δ3.67ppm (s, NH_2 group)figure(2) ,imidiazolidine **(2)** have been prepared by the reaction of compound (1) with ethyl chloroacetate, the reaction proceeds by elimination of ethanol and HCl molecules cyclization reaction according the mechanisam(1) in scheme(2):

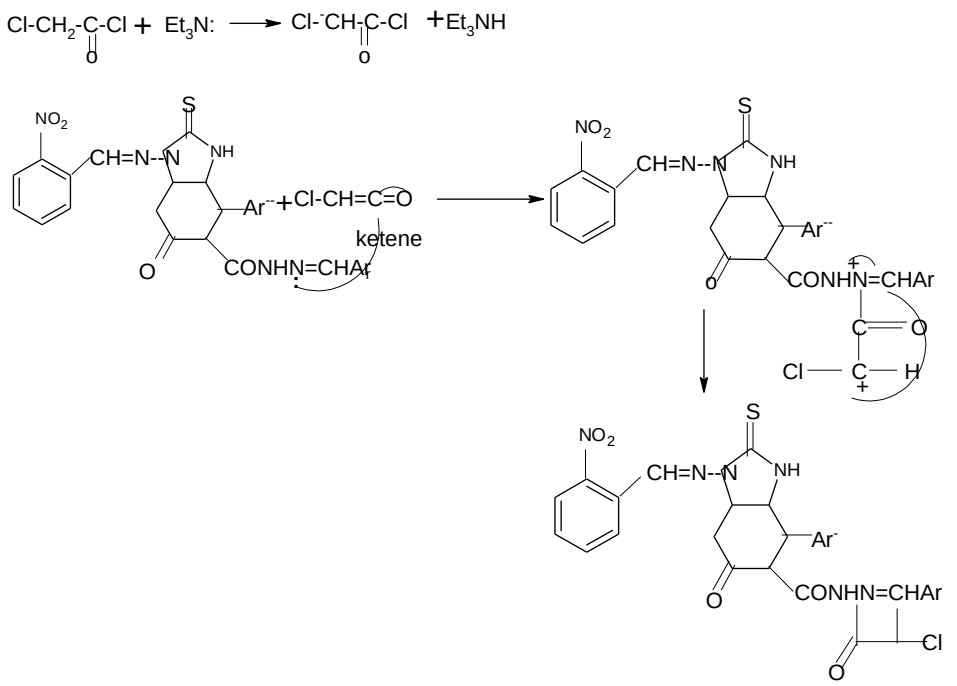


Scheme (2)

It follows a reaction by appearance of the new (C=O) band at 1732cm^{-1}, band at 3100 (NH) , and the FTIR. Spectrum shows other bands (cm^{-1}) at 3088 (CHar.), at 2939 (CH aliph.), at 1604-1527 (C=Car.), at 1633 (C=N) and at 1581,1350 (NO_2) the IR spectrum data of compound(2)fig(3), the ^{1}HNMR spectrum shows signals (δppm) at δ12.33ppm (NH), 8.89 (N=CH), δ7.92 -8.31ppm (m, 4H, ArH), 4.23ppm (CH_2) figure(4).The treatment of compound (2) with methyl iodide led to the formation of **(3).** Compound (3) has been identified by FTIR spectrum which shows appearance band at 3101for (NH) , band at 3064 (CHar.), band 1730 (C=O), bands at 1604-1500 due to (C=Car.),band at 1631 (C=N) and bands due to 1525,1350 (NO_2). Compound (4)have been obtained by reaction of the hydrazine hydrate with compound **(3)** . The. The FTIR spectrum of compound (4) shows the appearance of the two bands (asymmetric & symmetric at 3458, 3317 for (NH_2) group, while stretching

vibration of (NH) appearance at 3210 , band at 3043due to (CHar.),and the other bands appearance at 2991, (CH aliph.), 1720 (C=O), 1599-1521 (C=Car.), 1624 (C=N), 1552,1344 (NO_2). Imidazol-4-one derivatives (5a and 5b) are well known for their usefulress as starting material and biological activity, which were prepared by treatment of compounds (2) with aromatic aldehyde , take place by elimination of H_2O . The formation of chalcone was confirmed by appearance band of double bond at (1632 cm^{-1}) for (5a), and appearance of other bands as shown . 3277 (NH) 3134 (=CH), 3039 (CHar.), 2947 (CH aliph.), 1714 (C=O), 1593-1519 (C=Car.), 1632 (C=C alkene) , 1643 (C=N), 1550,1323 (NO_2), 687 (C-Br), the ^{1}HNMR spectrum shows signals (δ,ppm) atδ11.34ppm (NH), δ8.4ppm (N=CH), δ7.88 - 8.23ppm (m, 8H, ArH)δ 7.67ppm (s, C=CH). When the compounds (5a , 5b) were reaction with ethyl acetoacetate, derivatives (6a , 6b) were obtained in good result.The FTIR spectra exhibited a band due to (C=O) of ester at (1722 cm^{-1}) for compound (6b), and the spectrum shows other bands as shown , 3427 (NH) , 3086 (CHar.), 2980, 2899, 2831 (CH aliph.), 1714 (C=O ketone), 1587-1514 (C=Car.), 1668 (C=N), 1550,1371 (NO_2). Hydrazide derivative (7a, 7b) have been obtained by reaction of compounds (6a , 6b) with hydrazine hydrate , mechanism of this reaction[17] showed the lone pair of electron of amino group has attacked carbon of carbonyl group and then lose ethanol. The FTIR spectrum of compound (7a) shows band at (3455, 3336 cm^{-1}) for (NH_2) group , and the other bands appearance as shown , 3154 (NH) , 3082 (CHar.), 2960, 2871 (CH aliph.), 1691 (carbonyamide), 1721 (C=O ketone), 1610-1510 (C=C aromatic.), 1656 (C=N), 1554,1377 (NO_2),680 (C-Br). Refluxing compound (7a,7b) with p-bromo benzaldehyde offered good yields of compound (8a , 8b). The FTIR spectrum of compound (8b) shows disappearance bands of NH_2 and appearance band of(C=N) at 1616 cm^{-1} and the other bands at 3308(NH amide), 3234 (NH, imidazole) , 3088 (CHar.), 2928, 2879 (CH aliph.), 1656 (carbonyl amide), 1713 (C=O ketone), 1602-1500 (C=C aromatic.), 1550,1330 (NO_2) .Chloro acetyl chloride was of used as cyclizing agent for cyclization of Schiff's base derivatives (8a , 8b) to produce beta lactamaccording the mechanism(2) in scheme(3).The FTIR spectrum of compound (9a) shows appearance sharp band at 1734 for stretching vibration of (C=O) of beta lactam and other bands (9a) as shown, 3379(NH amide), 3226 (NH, imidazole) , 3051 (CHar.), 2983, 2861 (CH aliph.), 1683 (C=O amide), 1701 (carbonyl ketone), 1602-1500 (C=C aromatic.), 1651 (C=N), 1558,1363 (NO_2), 698 (C-Br), 790 (C-Cl)

Mech(2):-



Scheme (3)

No.of scheme	
Scheme no (1)	Synthesis of compouds(1-9b)
Scheme no(2)	Mechanism(1)
Scheme no(3)	Mechanism(2)

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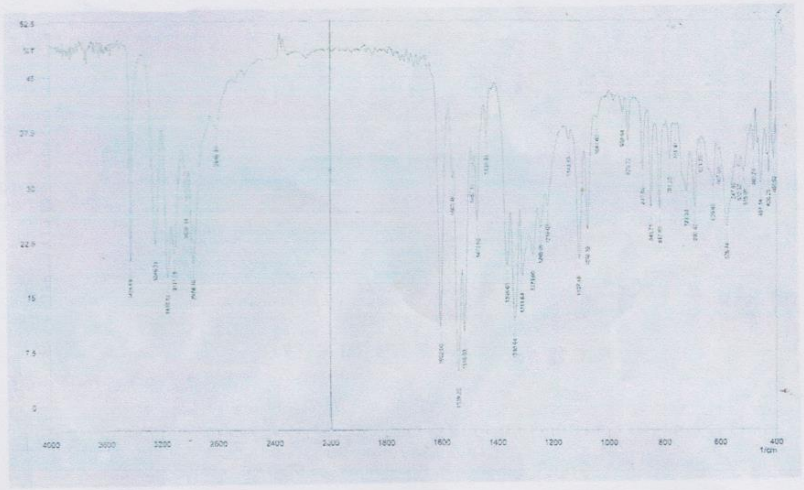

figure (1) FTIR for compound NO (1)

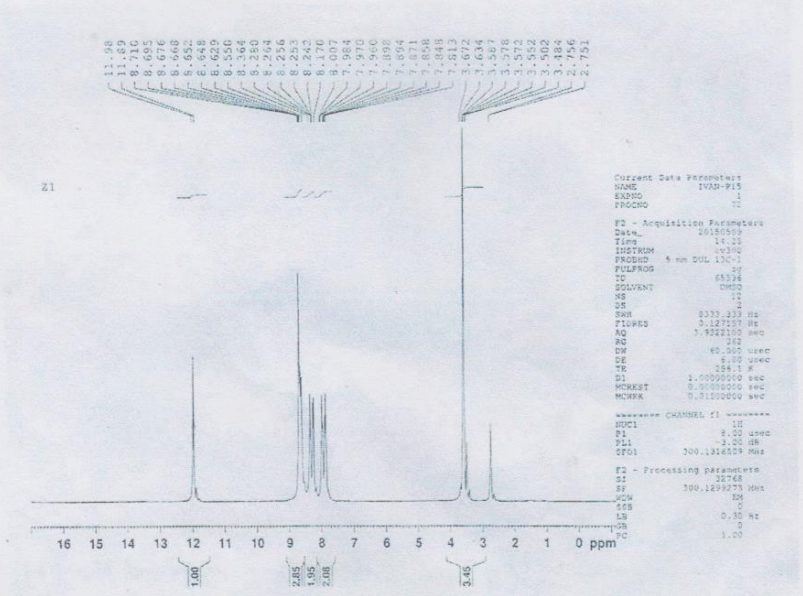

figure (2) ^{1}H-NMR for compound NO (1)

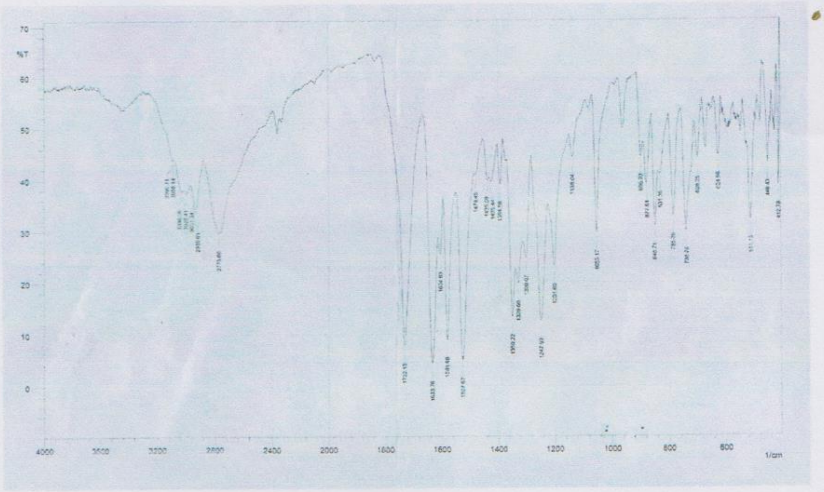

figure (3) FTIR for compound NO (2)

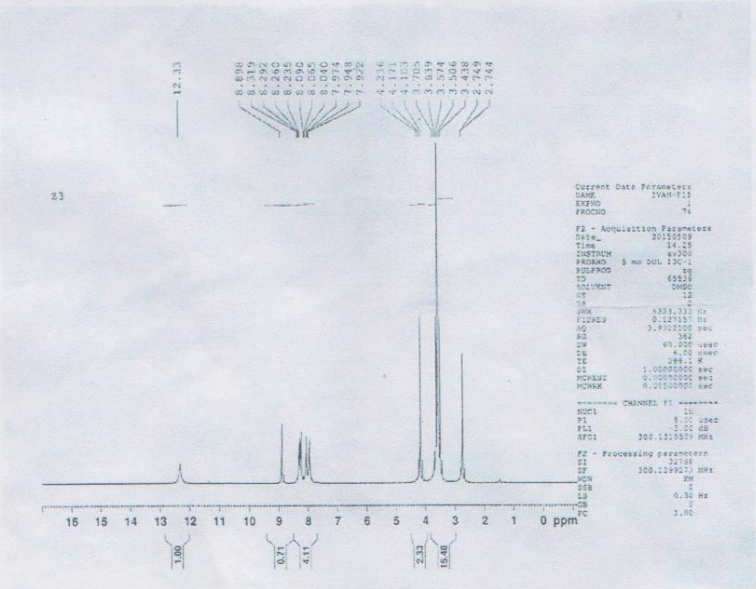

figure (4)^{1}H-NMR for compound NO (2)

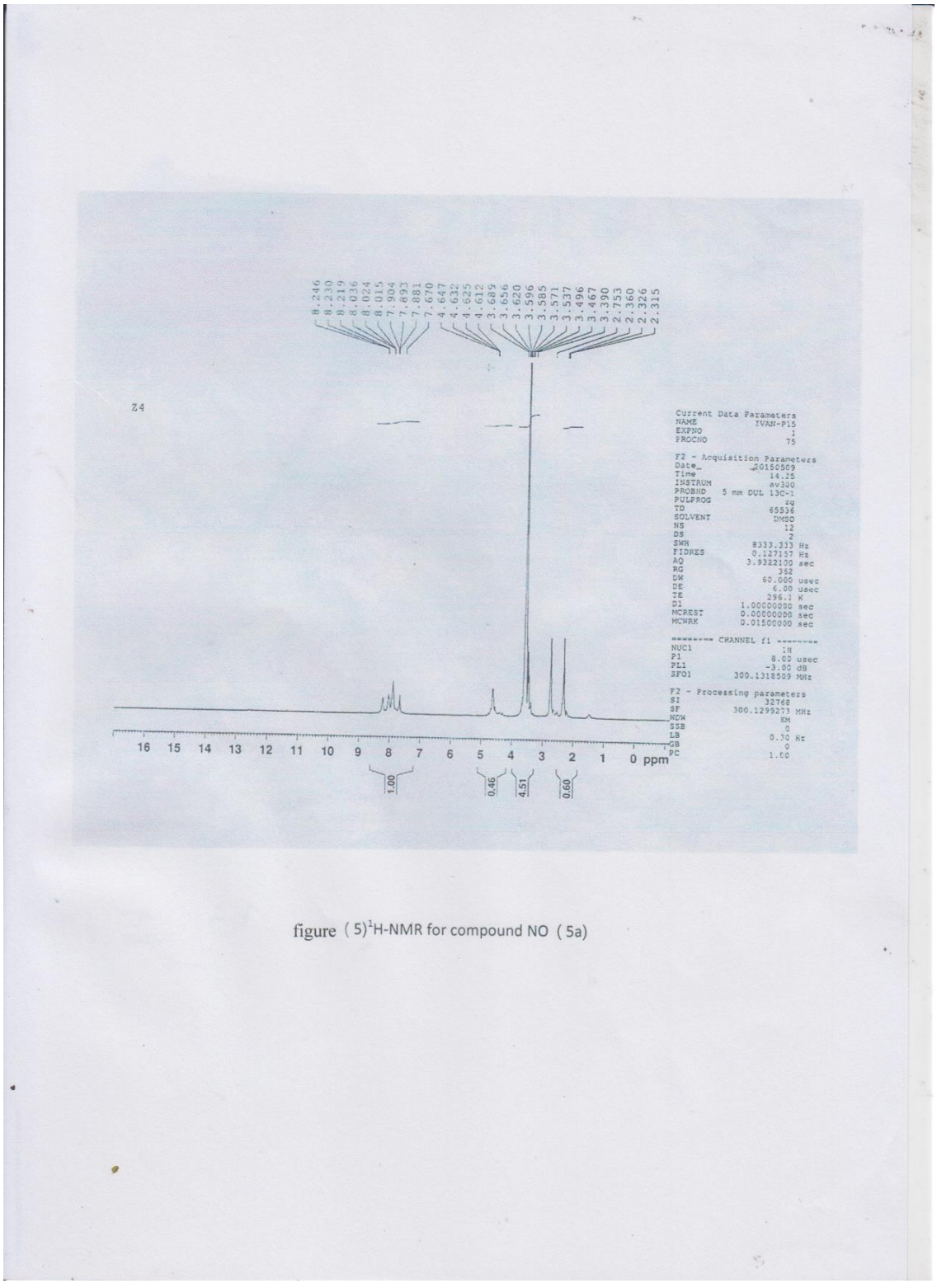

figure (5)^{1}H-NMR for compound NO (5a)

تحضير بعض المشتقات الجديدة من2- ثايوكسواميدازولدين – 4 – اون

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الخلاصة

تضمن هذا البحث تحضير سلسله جديدة من المركبات الحلقية غير المتجانسه ،يتفاعل2-نيتروبنزلدين ثايوسيمكاربزايد(1)مع اثيل كلورواستيت ليعطي (2-نيتروبنزلدين امينو) -2- ثايواكسواميدازولدين -4- اون) ،يعامل المركب (2) معالديهايدات اروماتية ليعطي 5- اريلدين3-(2-نيتروبنزلدين) امينو) 2-ثايو اكسو-5،3- داي هيدرو-H4- اميدازول -4-اونالذي يتفاعل مع الاثيل اسيتو اسيتيت ليعطي اريل امينو -6- اوكسو-2- ثايو اكسو اوكتا هيدرو - H1– بنزواميدازول-5-كاربوكسيليت ويتبعه تحضير مشتقات البيتالاكتام من معاملة المركبات (8a,b) قواعد شيف مع كلورو استيل كلورايد،ليعطي4-اريل -4- (2-نيتروبنزلدين) امينو -6-أكسو -2- اوكسو -2- ثايوكسواوكتاهيدرو-1 - بنزواميدازول - 5–كاربوكساميد ، وقد تم تشخيص المركبات المحضرة بواسطه تقنية طيف الرنين النووي المغناطيسي للبعض منها وطيف الأشعة تحت الحمراء .

الكلمات المفتاحية : الثايوسيماكاربزون ، جالكون ، بيتالاكتون ،اميدازولدين-4-اون