Thi-Qar Medical Journal (TQMJ): Vol(4) No(1):2010(89-103)

Synthesis Characterization of new compounds derivatives from amino acids

(Alanin, Argenine and Aspartic acid)

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ABSTRACT

This work involves preparation many compounds from cyclization of amino acid (Alanin, Argenine and Aspartic acid) by acetic an hydride to give (1-3) compounds, the last react with hydrazine to give its derivatives 1-amino -1- H –imidazol 5(4H) one. Schiff's bases was prepared from react (4-6) compound with various aldehydes and ketones ethyl bromo acetate react with (4-6) will be prepare (15-17) which react with hydrazine to give hydrazide derivatives (18-20) which condensation with aldehyde to keton to form Schiff's bases (21-25).on the other side compounds (15-17) react with thiosemicarbazide to obtain (26-28) which convert to (24-30) by deal with H_2SO_4 and to (31-33) by react with Sodium hydroxide.

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INTRODUCTION

Imidazol is an organic compound with the formula $C_3H_4N_2$. This aromatic hetro cyclic Is classified as an alkaloid. Imidazol refers to the parent compound whereas imidazoles are class of hetro cycles with similar ring structure but varying substituent's. this ring system is present in important biological building blocks such as histamine. Imidazol can serve as base and as weak acid. Many drugs contain an imidazol ring, such as antifungal drugs and nitroimidazol (1-5).Imidazol is incorporated into many important biological molecules. The most pervasive is amino acid histidine, which has an imidazol side chain. Histidine is present in many proteins ang enzymes and plays a vital part in the structures and binding functions of hemoglobin. Histidine can be decarboxy lated to histamine, which is also a common biological compound. It is component of the toxin thet causes urticaria, which is another name for allergic hives (6-10). One of the application of imidazol is in the purification of his tagged proteins in immobilized metal affinity chromatography (IMAC). Imidazol is used to elute tagget proteins bound to Ni ions.

EXPERIMENTAL SECTION

Synthesis of compounds (1-3)

Add 25ml of Ac_2O for (10 gm) from amino acid (Alanin, Argenine and Aspartic acid) and refluxed for 7 hours then evaporated it. A compounds 1,2 and 3 will be form. Table (1) show the physical properties of compounds.

Synthisis of compounds (4-6) (18-20)

(0.1) mole from compounds (A_1,A_2,A_3,A_{15},A_{16}, and A_{17}) add to (0.1)mole from hydrazine in absolute ethanol and refluxed the mixture for additional 5 hours. Then having evaporate to obtain compounds (4,5,8,18,19,20) as shown in table(2).

Synthisis of compounds (7-14) and (21-25)

A solution of (0.01) mole from compounds (4-1) and (18-20) in 25 ml of ethanol was added to equivalent amount of aldehyde or keton and refluxed the mixture for 3 hours. Then cooled to 0c and precipitate filtered of and recrystallized from wthanol 50%.Table(3)show some of physical properties of compounds which prepared.

Synthesis of compounds (15-17):

(0.1mole) of compounds(4,5,6) was dissolved in 50 ml of absolute ethanol, ethyl bromo acetate (0.1mole)was added and refluxed for 5hours.Having evaporated at room temperature and table (4) show the physical properties of this compounds.

Synthesis of =compounds (26-28)

(0.01mole) from compounds=(15,16,17) in 25 ml of ethanol added to equivaient amount of thiosemicarbazide and refluxed the mixture for 6 hours, a solid obtained recrystalized from ethanol.

Synthesis of (29-30)

(0.01mole) from compounds(A27,A28) dissolve in 25ml of H2SO4 to residu with vigorous stirring for 24hours.Cooled with ice bath and neutralized with sodium carbonate,the precipitate filtered off and wash with water .The solid obtained recrystalized from ethanol.

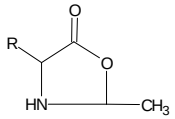
Synthesis of (31-33)

(0.01mole) of compounds (27,28) add to (0.01 mole) of NaOH in 25 ml of ethanol absolute and refluxed the mixture for 5 hours. Cooled the mixture then the precipitate filtered off and recrystallized from ethanol 50%.

RESULT AND DISCUSSION:

The synthesis of new target compound (1-33) began with cyclization of amino acid (Alanin, Argenine and Aspartic acid) which obtain in good yield by acetic an hydride. The formation of compounds (1-3) were indicated by the presence in their I.R spectra and C-O starching bands between (1770-1790) cm^{-1} and (1125-1230) cm^{-1} respectively,and stretching at (3345-3350) cm^{-1} disappearance of the stretching band 3600 for COOH group U.V. spectrum gave distinguished absorption at (309-339)nm and (201-245)nm which due to ($n \rightarrow \pi^*$) & ($\pi \rightarrow \pi^*$) transition respectively Compounds (4-6) characterized by disappear of c-o at 1125 cm^{-1} and show clear bands at 3500 cm due to NH_2 as show in table (2). The Schiff's based (18-20) (7-14) appearance of I.R new absorption bands in general region (1625-1690) cm^{-1} was attentively belonged to azom ethane (C=N) moiety. Moreover, the bands attributed to D (NH2) were disappeared. U.V spectry gave distinguished maximum at (309-375) and (206-215) nm which indicated the existence of ($n\rightarrow \pi^*$) and ($\pi\rightarrow \pi^*$) respectively (table 3) (table 6). Compounds (15-17) product from reaction of compounds (4-6) with ethyl bromoacetate, I.R spectrum of these compounds give bands at (1735-1745) cm and (1128-1200) cm which due to c for ester and C=O respectively. U.V spectra showed (337-371) nm and (206-243) nm due to ($n\rightarrow \pi^*$) and ($\pi\rightarrow \pi^*$) (table4). These compounds (18-20) obtain by reaction of compound (15-17) with hydrazine, in I.R spectrum observed new band at (3500-3550) cm^{-1} which due to (NH_2) and disappear band of c-o for ester at 1125 cm^{-1} . also U.V spectrum show absorption at (306-334) and (220-229) due ($n\rightarrow \pi^*$) and ($\pi\rightarrow \pi^*$) respectively table (5). A number of Thiosemicorbazide's derivative (26-28) were prepared and characterized by I.R spectrum which show new bands at (1025-1078) cm^{-1} due to C=S . and at (3450-3550) cm^{-1} due to NH_2. U.V spectry give absorption at (201-222) and (305-371) nm which attributed to ($\pi\rightarrow \pi^*$) ($n\rightarrow \pi^*$) respectively table(7). Thiosemicorbazide's

derivatives closed with H2SO4 to obtain compound (92-30) contain thiadiazal ring show many band. In I.R spectra shah as band at (1517)-1440) which due to D (C=N) of thiadiazal ring and bands at 3500 cm which refers to form thiadiazal ring. U.V spectra give (311-321) and (205-243) due to (n→ π*) and (π→ π*) (table 8). Finally , compounds (26-28) closed with NaOH to give compound (31-33) compound thiazol ring which indicated by I.R when the last show bands at (1520-1400) cm^{-1} due to C=N in ring and bands at (2565-2590) cm^{-1} due to -SH. U.V. spectra show absorption at (307-339) and (273-289) nm due to (n→ π*) and (π→ π*) (table 9).



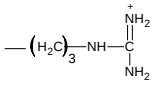
Comp.No.	-R	M.P	Yield%	Recrystalization
1	$-CH_3$	oily	95	EtOH 50%
2		oily	80	EtOH 50%
3	$-CH_2$ COOH	oily	47	EtOH 50%

Table (1) physical properties of compounds (1-3)

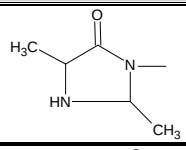
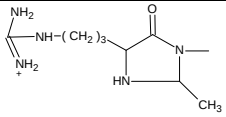
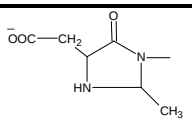
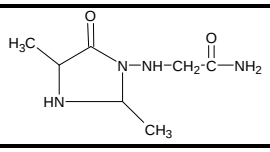
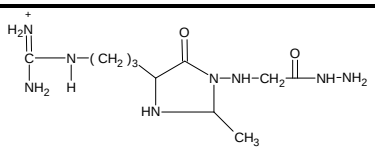
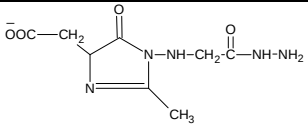
Comp. No	-R	M.P C°	P%	R.S
4		160-162	70	EtOH 50%
5		193-195	65	EtOH 50%
6		157-159	72	EtOH 50%
18		231-233	60	EtOH 50%
19		262-264	67	EtOH 50%
20		268-270	61	EtOH 50%

Table (2) physical properties of compounds (4-6) , (18-20) which have the chemical structure:R-NH_2

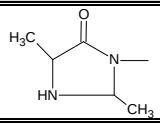
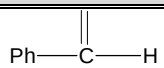
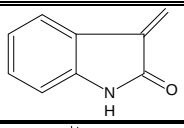
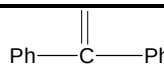
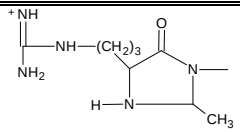
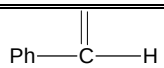
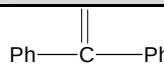
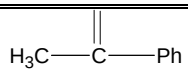
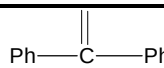
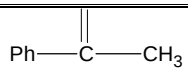
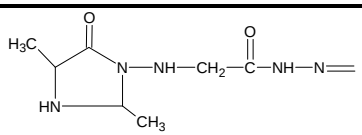
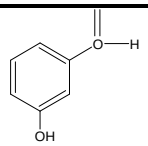
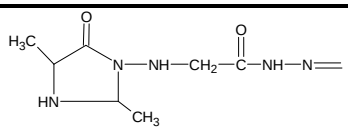
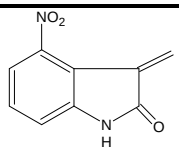
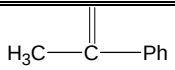
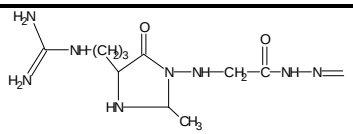
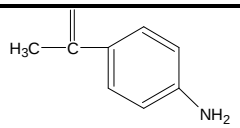
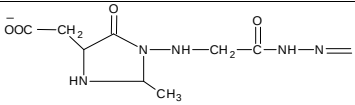
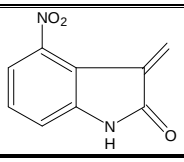
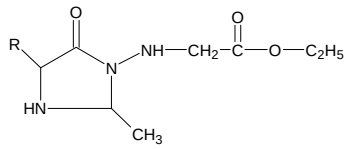
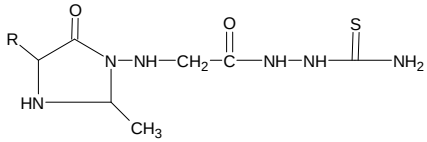
Comp.No.	-R	R`	M.P	P%	R.S
7			180-182	45	EtOH 50%
8	=		155-158	77	EtOH 50%
9	=		143-145	70	EtOH 50%
10			160-163	50	EtOH 50%
Comp.No.	**-R**	**R`**	**M.P**	**P%**	**R.S**
11	=		215-219	65	EtOH 50%
12	=		237-239	68	EtOH 50%
13	-CH_2 COOH		265-217	78	EtOH 50%
14	-CH_2 COOH		197-199	79	EtOH 50%
21			201-203	65	EtOH 50%
22			225-228	77	EtOH 50%
23	=		195-197	58	EtOH 50%
24			199-201	45	EtOH 50%
25			253-255	92	EtOH 50%

Table (3) physical properties of compounds (7-14) , (21-25) which have the chemical structure: R-N = R`



Comp.No.	-R	M.P	P%	R.S
15	$-CH_3$	125-127	74	EtOH 50%
16	$-(H_2C)_3-NH-C(=NH^+)-NH_2$	300(dec.)	77	EtOH 50%
17	$-CH_2$ COOH	290-292	65	EtOH 50%

Table (4) physical properties of compounds (15-17) which have the chemical structure:

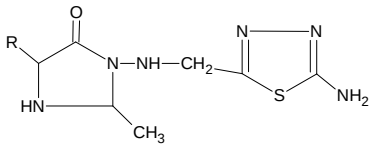


comp NO	-R	M.P	P%	R.S
26	$-CH_3$	233-238	69	EtOH 50%
27	$-(H_2C)_3-NH-C(=NH_2^+)-NH_2$	310-212	77	EtOH 50%
28	$-CH_2$ COOH	300 (dec.)	95	EtOH 50%

Table (5) physical properties of compounds (26-28) which have the chemical structure:

Synthesis Characterization of new compounds derivatives from amino acids

(Alanin, Argenine and Aspartic acid)



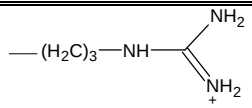
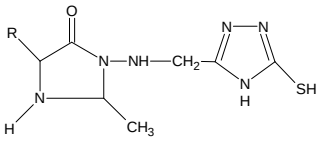
Comp. No	-R	M.P	P%	R.S
29	$-CH_2\ COOH$	310 (dec.)	81	EtOH 50%
30		205-207	95	EtOH 50%

Table (6) physical properties of compounds (29-30) which have the chemical structure:



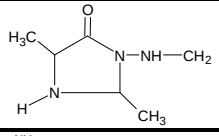
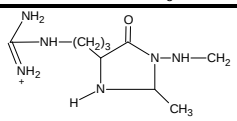
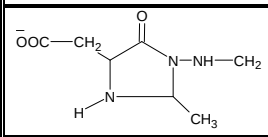
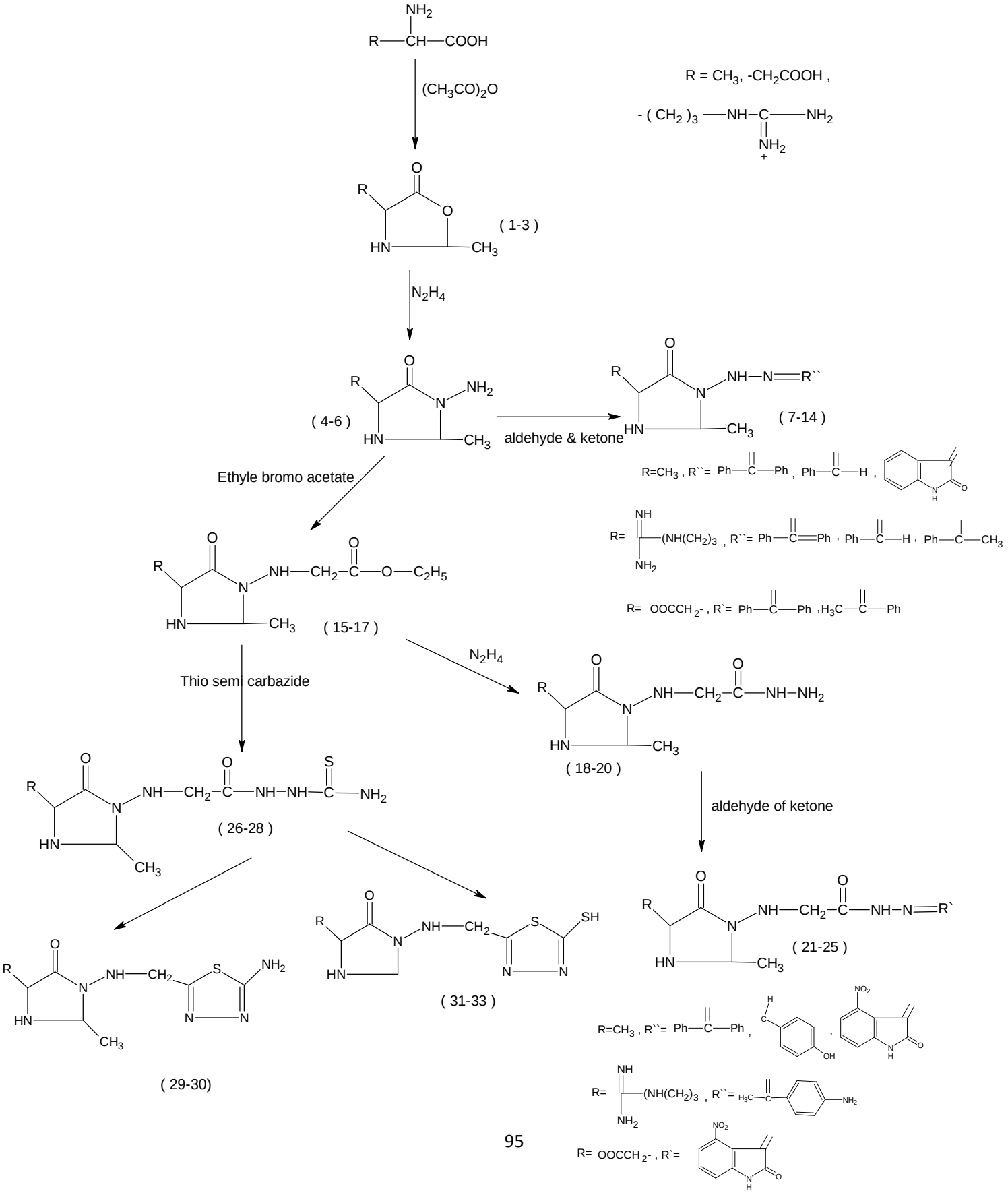
Comp.No.	-R	M.P	P%	R.S
31		273-275	65	EtOH 50%
32		289-291	50	EtOH 50%
33		221-223	78	EtOH 50%

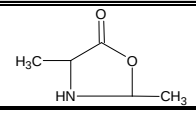
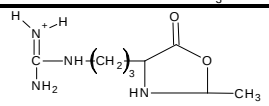
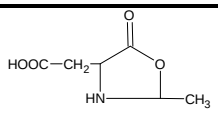
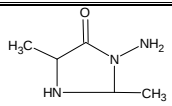
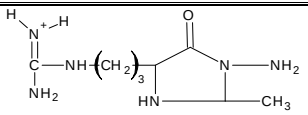
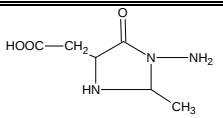
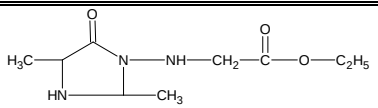
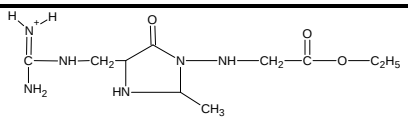
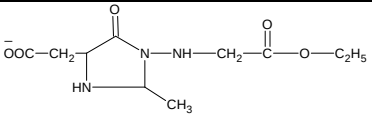
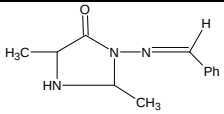
Table (7) physical properties of compounds (31-33) which have the chemical structure:

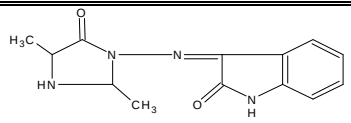
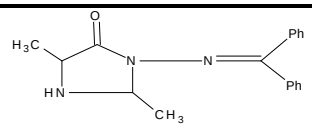
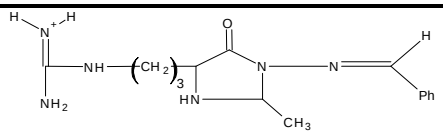
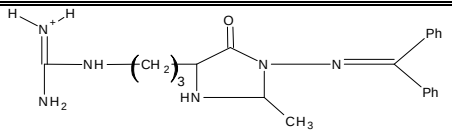
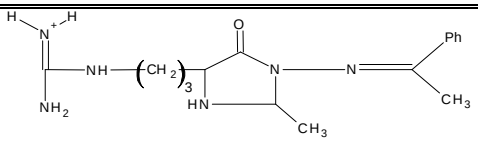
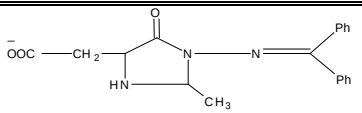
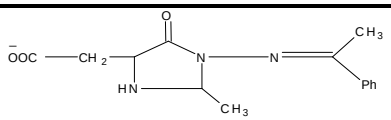
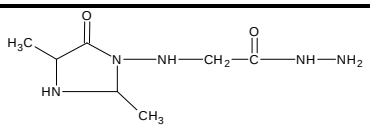
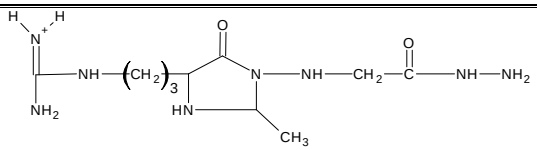
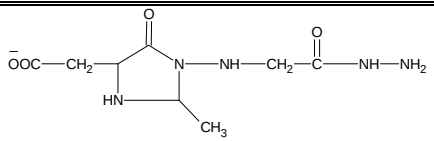

R = CH3, -CH2COOH ,
(1-3)
(4-6)
aldehyde & ketone
(7-14)
Ethyle bromo acetate
(15-17)
Thio semi carbazide
(18-20)
aldehyde of ketone
(21-25)
(26-28)
(29-30)
(31-33)

Synthesis Characterization of new compounds derivatives from amino acids

(Alanin, Argenine and Aspartic acid)

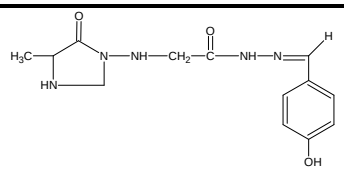
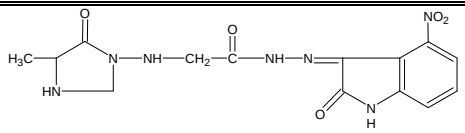
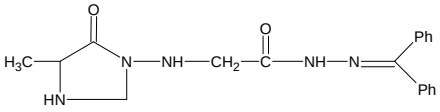
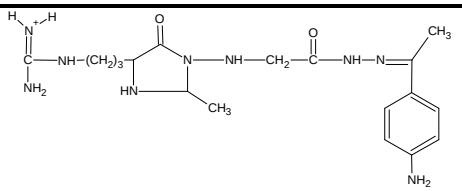
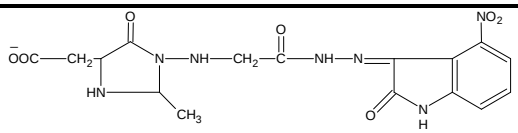
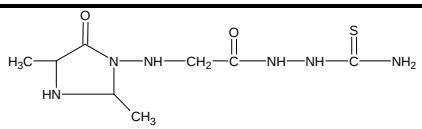
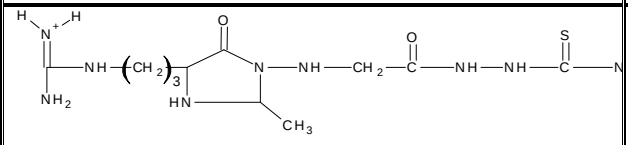
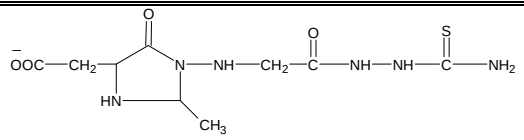
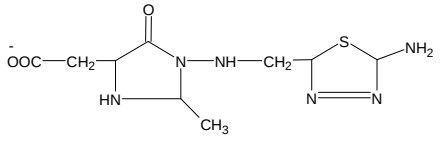
Scientific name and chemical structure of preparation compounds.

Compound structure	Compound name	Comp.No
	2,4-dimethyl-1,3-oxazolidin-5-one	1
	amino{[3-(2-methyl-5-oxo-1,3-oxazolidin-4-yl)propyl]amino}methaniminium	2
	(2-methyl-5-oxo-1,3-oxazolidin-4-yl)acetate	3
	3-amino-2,5-dimethylimidazolidin-4-one	4
	amino{[3-(1-amino-2-methyl-5-oxoimidazolidin-4-yl)propyl]amino}methaniminium	5
	(1-amino-2-methyl-5-oxoimidazolidin-4-yl)acetic acid (1-amino-2-methyl-5-oxoimidazolidin-4-yl) acetate	6
	ethyl [(2,4-dimethyl-5-oxoimidazolidin-1-yl)amino]acetate	15
	amino[({1-[(2-ethoxy-2-oxoethyl)amino]-2-methyl-5-oxoimidazolidin-4-yl}methyl)amino]methaniminium	16
	{ {1-[(2-ethoxy-2-oxoethyl)amino]-2-methyl-5-oxoimidazolidin-4-yl}acetic acid	17
	3-(benzylideneamino)-2,5-dimethylimidazolidin-4-one	7

	3-[(2,4-dimethyl-5-oxoimidazolidin-1-yl)imino]-1,3-dihydro-2*H*-indol-2-one	8
	3-[(diphenylmethylene)amino]-2,5-dimethylimidazolidin-4-one	9
	amino({3-[1-(benzylideneamino)-2-methyl-5-oxoimidazolidin-4-yl]propyl}amino)methaniminium	10
	amino[({1-[(diphenylmethylene)amino]-2-methyl-5-oxoimidazolidin-4-yl}propyl)amino]methaniminium	11
	amino[(3-{2-methyl-5-oxo-1-[(1-phenylethylidene)amino]imidazolidin-4-yl}propyl)amino]methaniminium	12
	{1-[(diphenylmethylene)amino]-2-methyl-5-oxoimidazolidin-4-yl} acetate	13
	{ {2-methyl-5-oxo-1-[(1-phenyl ethylidene) amino]imidazolidin-4-yl} acetate	14
	{2-methyl-5-oxo-1-[(1-phenylethylidene)amino]imidazolidin-4-yl}acetic acid	18
	amino[(3-{1-[(2-hydrazino-2-oxoethyl)amino]-2-methyl-5-oxoimidazolidin-4-yl}propyl)amino]methaniminium	19
	{1-[(2-hydrazino-2-oxoethyl)amino]-2-methyl-5-oxoimidazolidin-4-yl}acetate	20

Synthesis Characterization of new compounds derivatives from amino acids

(Alanin, Argenine and Aspartic acid)

	*N*'-(4-hydroxybenzylidene)-2-[(4-methyl-5-oxoimidazolidin-1-yl)amino]acetohydrazide	21
	2-[(4-methyl-5-oxoimidazolidin-1-yl)amino]-*N*'-(4-nitro-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)acetohydrazide	22
	*N*'-(diphenylmethylene)-2-[(4-methyl-5-oxoimidazolidin-1-yl)amino]acetohydrazide	23
	amino({3-[1-({2-[2-(1-4`-amino phenyl oxoethyl}amino)-2-methyl-5-oxoimidazolidin-4-yl]propyl}amino)methaniminium	24
	[2-methyl-1-({2-[2-(4-nitro-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)hydrazino]-2-oxoethyl}amino)-5-oxoimidazolidin-4-yl] acetate	25
	2-{[(2,4-dimethyl-5-oxoimidazolidin-1-yl)amino]acetyl}hydrazinecarbothioamide	26
	amino({3-[1-({2-[2-(aminocarbonothioyl)hydrazino]-2-oxoethyl}amino)-2-methyl-5-oxoimidazolidin-4-yl]propyl}amino)methaniminium	27
	[1-({2-[2-(aminocarbonothioyl)hydrazino]-2-oxoethyl}amino)-2-methyl-5-oxoimidazolidin-4-yl]acetate	28
	(1-{[(5-amino-2,5-dihydro-1,3,4-thiadiazol-2-yl)methyl]amino}-2-methyl-5-oxoimidazolidin-4-yl)acetic acid acetate	29

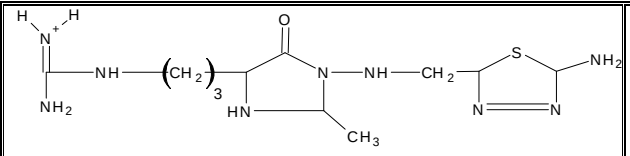
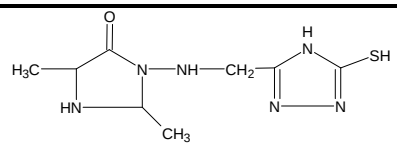
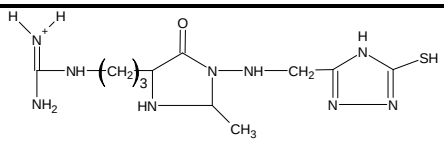
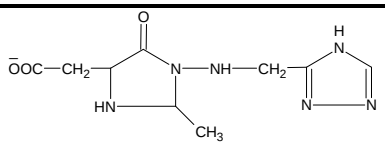
	amino{[3-(1-{[(5-amino-2,5-dihydro-1,3,4-thiadiazol-2-yl)methyl]amino}-2-methyl-5-oxoimidazolidin-4-yl)propyl]amino}methaniminium	30
	3-{[(5-mercapto-4*H*-1,2,4-triazol-3-yl)methyl]amino}-2,5-dimethylimidazolidin-4-one	31
	amino{[3-(1-{[(5-mercapto-4*H*-1,2,4-triazol-3-yl)methyl]amino}-2-methyl-5-oxoimidazolidin-4-yl)propyl] amino}methaniminium	32
	{2-methyl-5-oxo-1-[(4*H*-1,2,4-triazol-3-ylmethyl)amino]imidazolidin-4-yl}acetate	33

Table (1)Caracteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No.	U.V. λ_{max} nm	ν (CH) cm^{-1}	ν (c) cm^{-1}	ν (C-O) cm^{-1}	ν (NH)cm^{-1} For ring	Others cm^{-1}
1	309,245	2980,2890	1770	1125	3345	CH_3=1460 as,1360 s
2	334,255	2985,2892	1780	1130	3340	ν (NH_2) 3500, δ NH_2 1515
3	339,201	2986,2806	1790	1230	3350	Carboxylate Anion=1650 as, 1400 s

Table (2) Characteristic bands of I.R spectra (4-6)

Comp No	U.V. λ_{max} nm	ν CH cm^{-1}	ν C=O cm^{-1}	νNH_2 cm^{-1}	Others cm^{-1}
4	399,241	2922,2981,2890	1730	3480	ν (-NH)= 3430, δ (-NH)=1600, CH_3 = 1460as
5	301,205	2980,2890,2920	1735	3500	δ NH =1515,ν NH =3300
6	361,211	2920,2986,2895	1745	3540	Carboxylate Anion =1650 as,1400 s

Table (3): Characteristic bands of I.R and UV. spectra (7-11)

Comp No	U.V. λ_{max} nm	ν(CH)alph.	ν(C=N) cm^{-1}	ν(C=O) cm^{-1}	Others cm^{-1}
7	375,206	2978,2892,2927	1635	1730	ν (c=c)arm =1540,ν (NH) of ring 3345
8	309,215	2988,2890,2928	1625	1732	ν (C) of esatin 1745,ν (C=C) arm =1585,ν (C-H) arm =3100
9	320,207	2920,2986,2890	1665	1737	ν (C=C) ar =1565,ν (C-H) ar= 3095
10	327,205	2980,2920,2890	1645	1734	ν (-NH2)=3500,ν (C=C) ar=1542
11	333,219	2981,2920,2892	1640	1791	ν (NH)=3350,ν (C=C) ar=1555
12	325,230	2978,2925,2885	1650	1715	CH3 = 1462 as, =1365 s
13	356,222	2930,2986,2895	1680	1738	CH3 =1460 as,1355 s
14	347,210	-	-	1750	Carboxylate Anion=1650 as, 1400 s

Table (4): Characteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No.	U.V. λ_{max} nm	ν CH	ν C=O of ester	others
15	371,243	2978,2922,2890	1735 interference	ν (-NH) =3345
16	356,206	2985,2926,2895	1735 interference	CH_2= 1455 as, 1360 s
17	337,219	2986,2925,2890	1745 interference	ν (-NH) =3350 Carboxylate Anion=1650 as,1400 s

Table (5) : Characteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No.	U.V. λ_{max} nm	ν CH cm^{-1}	ν C=O of amide	D(NH_2) cm^{-1}	Others cm^{-1}
18	319,220	2981,2922,2890	1680	3500	ν c of lactum 1720,ν (NH) =3400,ν (NH) of ring 3350
19	334,221	2980,2920,2885	1688	3510	ν c of Lactum 1735,ν (-NH) 3405
20	306,229	2925,2986,2895	1685	3550	ν (C=O) of Lactum 1748,ν (-NH) 3430

Table (6) Characteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No.	U.V. λ_{max} nm	ν(C-H) cm^{-1}	ν (C=O) amid	ν(C=N)cm^{-1}	Others cm^{-1}
A_{21}	345,207	2920,2978,2890	1680	1635	ν (C=C) =1540,ν (NH) of ring =3345
A_{22}	309,214	2922,2980,2891	1678	1630	ν (-NO_2)=1520,ν(C)for esatin 1745
A_{23}	391,248	2920,2980,2888	1680	1631	(C=C) ar =1565,ν(CH) ar=3100
A_{24}	301,211	2981,2920,2885	1688	1630	ν (NH_2)=3500,ν (C=C) ar=1555
A_{25}	339,211	2922,2985,2895	1685	1638	Carboxylate Anion=1650 as,1400s,(C=O) of Lactum 1750

Table (7): Characteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No	U.V. λ_{max} nm	ν CH alph	ν (C=S)cm^{-1}	Others cm^{-1}
26	345,215	2919,2978,2890	1025	ν c of Lactam 1730,ν (NH) =3345
27	305,201	2980,2920,2812	1075	ν (NH_2) =3500
28	371,222	2922,2985,2896	1078	

Table (8): Characteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No	U.V λ_{max} nm	ν CH alph	ν C=N of	Others cm^{-1}
29	311,243	2989,2917,2820	1517	ν (-NH_2) 3500,ν (C) 1735 of Lactam
30	321,205	2987,2919,2825	1440	ν (-NH_2) 3500

Table (9): Characteristic bands of I.R spectra (cm^{-1},KBr disc)

Comp No	U.V.	ν(NH)cm^{-1}	ν SH cm^{-1}	others
31	307,273	3338	2572	ν (CH_2) 2926,ν (C-N) 1025,ν (C=N) for thiazol 1520
32	332,289	3345	2565	ν (CH)=2960, 2871,ν (c) of Lactam 1730,C=N 1570
33	329,288	3350	2590	Carboxylate Anion =1690 as, 1400 s

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Thi-Qar Medical Journal (TQMJ): Vol(4) No(1):2010(89-103)

تحضير وتشخيص مركبات جديدة مشتقة من الاحماض الامينية (الانين،ارجنين و اسبارتك اسيد)

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الخلاصــة:-

يتضمن هذا البحث مركبات مشتقة من حولقة الاحماض الامينية (الانين، ارجنين وسبارتك) بواسطة استيك انهيدريد ليعطي المركبات (١-٣) ثم تفاعل الاخيرة مع الهيدرازين لتعطي مشتقات - 1- amino 1- H –imidazol 5(4H) one .(٤-٦) اما قواعد شيف فقد حضرت من تفاعل المركبات (٤-٦) مع الالدهايدات والكيتونات. ومن مفاعلة (٤-٦) مع المركب اثيل بروموا سيتيت تم تحضير المركبات (١٥-١٧) والتي عوملت مع الهيدرازين لتعطي مشتقات الهيدرازايد (١٨-٢٠) والاخيرة تتفاعل مع الالديهايدات والكيتونات لتعطي قواعد شيف (٢١-٢٥) وبتفاعل (١٥-١٧) مع الثايوسيكارباز ايد تم تحضير (٢٦-٢٨) والتي حولت الى المركبات (٣٠-٢٩) بتفاعلها مع حامض الكبريتيك والى (٣١-٣٣) بوجود هيدروكسيد الصوديوم.

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