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Synthesis and Identification of New Amide and ThioUrease Compounds (II)

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ABSTRACT

The reaction of some new Schiff bases (2E,3Z)-3-hydrazonobutan-2-one oxime and 3-((Z)- (E))-3-(hydroxyimino)butan-2-ylidene)hydrazono)butan-2-one oxime with benzoyl chloride and acetyl chloride were carried out. Subsequent reactions of these products. N-[Chloro-(2-hydroxy-phenyl)-N-(4,6-diamino-[1,3,5]triazin-2-yl)-acetamide and N-[Chloro-(2-hydroxy-phenyl)-N-(4,6-diamino-[1,3,5]triazin-2-yl)-benzamide with thiourea afforded thioureas compounds.The synthesized compounds were confirmed by their , UV,FT- IR spectra and C.H.N. analysis.

Introduction:-

It is known that Schiff bases react smoothly with acid chlorides and anhydrides to give the corresponding addition products [1-5]

Reaction of Schiff base with acid chloride gave imide compounds and reaction of imide compounds with thiourea afforded thioureas compounds [6]

The synthesis of some new Phenobarbital compounds from the reaction of some Phenobarbital system containing Schiff bases moiety with benzoyl and 3,5-dinitro benzoyl chlorides .Subsequent reactions of these products with thiourea afforded thioureas compounds [7, 9] .

The reaction of a new Schiff bases (2-[(2-amino-ethylimino)-methyl]-R, 2-({2-[(R-benzylidene)-amino]- ethylimino}-methyl)-R with acetyl chloride or benzoyl chloride were carried out. Subsequent reactions of these products N-(2-amino-ethyl)-N-[Chloro-(R)-methyl] –benzamide or N-(2-{R-[chloro-(R) –methyl]-amino}-ethyl)-N-[chloro-(R)-methyl]-benzamide with thiourea afforded thioureas compounds. [10].

The new dioxime ligand, (2E,3E)-3-[(6-{[(1E,2E)-2-(hydroxyimino)-1-methylpropylidene]amino}-pyridin-2-yl)imino] butan-2-one oxime, (H2pymdo) has been synthesized in H2O by reacting 2,3-butenedione monoxime with 2,6-diaminopyridine[11] .The ligand incorporating a dioxime moiety, (2E,3E)-3-[(6-{[(1E,2E)-2-(hydroxyimino)-1- methyl propylidene] amino}-phenyl) imino]butan-2-one oxime,(H2phdo) has been prepared by reacting 2,3-butanedione-mono -{O-[4-(1-methy-2-oxo-propylidene amino oxy)-2,3-bis-(1-methyl-2-oxo-propylideneaminooxy-methyl)-but-2-enyl]-oxime with 1,2-phenylene diamine. [12] .

Experimental:-

Melting points were recorded on Gallenkamp melting points apparatus and were uncorrected. Elemental analysis was carried out in Tekreet University on PerkinElmer 2400 CHN Elemental analyzer. FT-IR spectra were recorded on FT-IR spectrophotometer -8400s Shimadza (KBr)at the chemistry department of Education for women college AL-Anbar University and UV-Visible spectra were recorded (in ethanol) On Schimadza Reco- 160x Spectrophotometer.

Preparation of Schiff bases:-

(2E,3Z)-3-hydrazonobutan-2-one oxime and 3-((Z)-((E)-3- (hydroxyimino) butan-2-ylidene) hydrazono) butan-2-one oxime were prepared by condensation of 2-(amino methyl)-2-(hydroxymethy l0propane-1,3-diol (Oxime) with some aldehyde and Keton compounds .

To a solution of (0.05mol) of 2-(amino methyl)-2-(hydroxymethyl) propane-1,3-diol (Oxime) in 25ml of (absolute) Ethanol was added (0.05mol) of substituted benzaldehyde,4-aminoantipyrine and glutaraldehyde and refluxed 3hr. Whereby (a yellow–Orange)

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crystalline solid separated out. The solid was filtered and recrystallized from ethanol (absolute) . [8]

Preparation of (E)-N-(2-chloro-3-(hydroxyimino) butan-2-yl) acetohydrazide:-

To an appropriate Schiff base (2E,3Z)-3-hydrazonobutan-2-one oxime (0.015 mole), Acetyl chloride (0.015 mole) in (absolute) ethanol (25 ml) was added. The mixture was refluxed for (6 hr), cooled, filtered and recrystallized from absolute ethanol (Tables).

Preparation of (E)-2-(1-acetylhydrazinyl)-3-(hydroxyimino)butan-2-ylcarbamimidothioate:-

To an appropriate (E)-N-(2-chloro-3-(hydroxyimino) butan-2-yl) acetohydrazide (0.001mole) thiourea (0.001 mole) and Na2CO3 (0.002 mole) in absolute ethanol (30ml) were added. The mixture was refluxed for (3 hrs), cooled and filtered. The filtrate was poured into crushed ice, the separated solid was collected and recrystalized from 1,4-dioxan solvent (Tables).

Results and Discussion :-

Schiff bases (1-4) were prepared by condensation of 2-(amino methyl)-2-(hydroxymethyl)propane-1,3-diol (Oxime)with (hydrazine and ethylene diamine). The reaction was followed by the appearance of absorption bands(1630-1640 cm-1) for (υ C=N) at in their FT-IR spectra. In this work the reaction of Schiff-bases compounds with acetyl or benzoyl chlorides and subsequent reactions of above reaction products (5-16) with thiourea were carried out as shown in scheme (1).

However, treatment of Schiff bases with acid halides results in the formation of compounds (5-16) in which two groups (Cl,RC=O) were introduced in the same step of the reaction. This reaction was followed by disappearance of absorption bands at (1230-1255cm-1) and (720-770cm-1) which were attributed to (C-N) and (C-Cl) moieties.

The reaction was involved the attack of the azomethine nitrogen by the carbonyl group of the aroyl chlorides, displacing the chloride as chloride anion and forming the iminium cation.

However, iminium cation was unstable, so the Cl-attacked- +N=C moiety and afforded more stable covalently bonded compounds (5-16) Scheme (2).

Moreover, the reactions of acid halides addition products (5-16) with thiourea were afforded thioureas products (17- 26). So, heating compounds (5-16) under reflux with thiourea in the presence of Na2CO3 for (3 hrs) led to the nucleophilic substitution of Cl by and compounds (17-26) were formed through the following mechanism (Scheme 3).

These compounds (17-26) were characterized by their FT-IR spectra. New doublet absorption bands in the region (3440-3245cm-1) were attributed to (NH2) and (NH) functional moieties. Other characteristic bands in the region (650-710cm-1) correlated to (υ C-S) moiety. Moreover, (υ C-Cl) around (725-750cm-1) disappeared.

They reveated strong absorption ranging from (222-2340nm) and from (240-295nm). These absorptions were due (π-π*) transition or η- electrons of nitrogen atom which was in conjugation with neighboring groups.

The Uv-Visible spectra showed the following maxima (220-240nm) and (300-385nm), due to the presence of aromatic rings and avariety of substituted groups.

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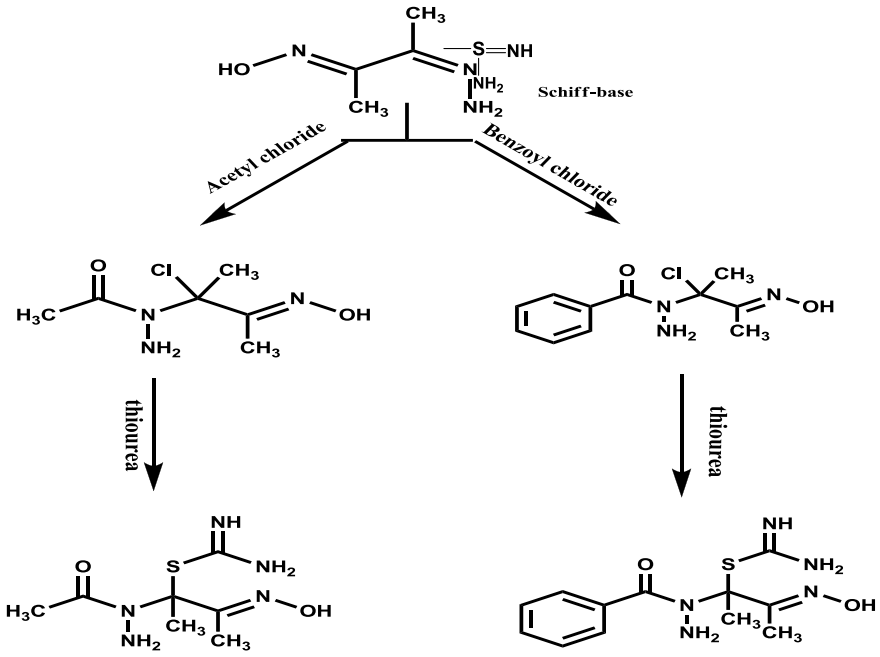
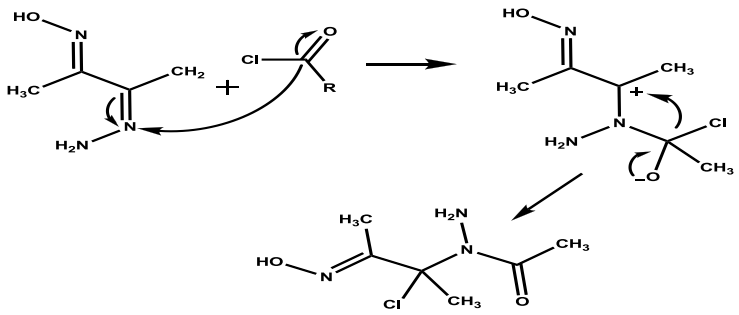
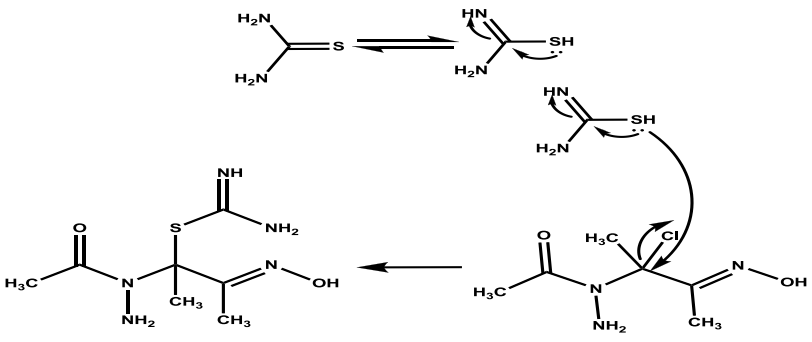


(Scheme 1)



(Scheme 2)



(Scheme 3)

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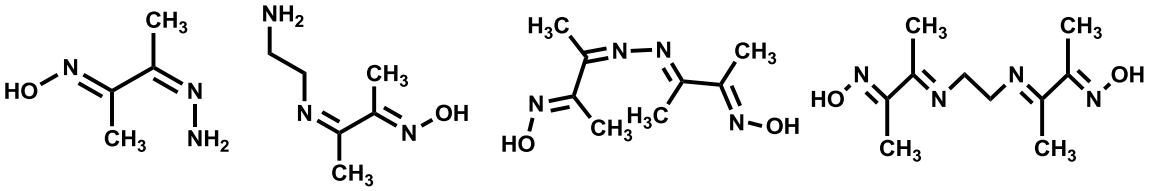
Table(1):Some physical properties and C.H.N. analysis of Schiff base (1 - 4).



(1) **(2)** **(3)** **(4)**

Comp.	M.F.	Yield%	m.p/c°	Co lour	Cal.			Found		
					C	H	N	C	H	N
1	$C_4H_9N_3O$	80		Yellow	41.73	7.88	36.50	41.35	7.52	36.31
2	$C_6H_{13}N_3O$	75		Yellow	50.33	9.15	29.35	50.02	9.10	29.12
3	$C_8H_{14}N_4O_2$	85		Red	48.47	7.12	28.26	48.34	7.00	28.03
4	$C_{10}H_{18}N_4O_2$	70		Deep-red	53.08	8.02	24.76	52.96	7.89	24.60

Table(2):IR spectral data of compounds(1-4).

Characteristic bands of FT-IR spectrum						
Comp.	υ (OH) (cm^{-1})	υ (NH_2) (cm^{-1})	υ (C=N)imine (cm^{-1})	υ (C=N–OH) (cm^{-1})	υ (C-N) (cm^{-1})	υ (N-O) (cm^{-1})
1	3470	3270,3320	1645	1585	1420	1365
2	3460	3315,3265	1650	1590	1410	1370
3	3465	3320,3210	1640	1595	1420	1355
4	3470	3325,3200	1650	1590	1415	1360

Table (3): UV spectral data of compounds(1-4).

Comp. No.	UV-Visible absorption maxima λ/nm
1	310,275,255
2	320,280,260
3	305,285,265
4	300,275,260

Table(4):Some physical properties and C.H.N. analysis of compound (5 - 17).

Comp.	M.F.	Yield%	m.p/c°	Co lour	Cal.			Found		
					C	H	N	C	H	N
5	$C_6H_{12}ClN_3O_2$	56	223-221	orange	37.22	6.25	21.70	37.02	6.01	21.48
6	$C_8H_{16}ClN_3O_2$	67	198-196	yellow	43.34	7.27	18.95	43.18	7.05	18.65
7	$C_{10}H_{17}ClN_4O_3$	77	212-210	Brown	43.40	6.19	20.25	43.22	5.98	20.00
8	$C_{12}H_{20}Cl_2N_4O_4$	75	233-231	orange	40.57	5.68	15.77	40.52	5.41	15.48
9	$C_{12}H_{21}ClN_4O_3$	66	165-163	yellow	47.29	6.65	18.38	47.21	6.83	18.11
10	$C_{14}H_{24}Cl_2N_4O_4$	59	177-175	orange	43.87	6.31	14.62	43.65	6.08	14.48
11	$C_{11}H_{14}ClN_3O_2$	62	137-135	Brown	51.67	5.52	16.43	51.55	5.37	16.33
12	$C_{13}H_{18}ClN_3O_2$	73	227-225	yellow	55.03	6.39	14.81	54.87	6.19	14.66
13	$C_{15}H_{19}ClN_4O_3$	80	190-188	orange	53.18	5.65	16.54	53.16	5.49	16.45
14	$C_{22}H_{24}Cl_2N_4O_4$	67	161-159	orange	55.12	5.05	11.69	55.02	4.88	11.50.
15	$C_{17}H_{23}ClN_4O_3$	71	200-198	yellow	55.66	6.32	15.27	55.42	6.13	15.06
16	$C_{24}H_{28}Cl_2N_4O_4$	70	240-238	yellow	56.81	5.56	11.04	56.65	5.39	10.88

Table(5):IR spectral data of compounds(5-17).

Characteristic bands of FT-IR spectrum							
Comp.	υ (OH) (cm^{-1})	υ (NH_2) (cm^{-1})	υ (C=O) (cm^{-1})	υ (C=N)imine (cm^{-1})	υ (C=N–OH) (cm^{-1})	υ (C-O) (cm^{-1})	υ (C-Cl) (cm^{-1})
5	3520	3440,3250	1680	-	1605	1250	725
6	3510	3450,3220	1670	-	1590	1260	750
7	3500	-	1680	1640	1580	1240	740
8	3525	-	1675	-	1585	1270	735
9	3530	-	1660	1635	1575	1280	725
10	3520	-	1670	-	1595	1250	730
11	3520	3445,3240	1680	-	1590	1260	750
12	3535	3450,3230	1665	-	1600	1290	740
13	3510	-	1670	1640	1605	1270	730
14	3505	-	1680	-	1585	1280	740
15	3500	-	1685	1645	1580	1285	745
16	3520	-	1670	-	1590	1290	740

Table(6):Some physical properties and C.H.N. analysis of compound (17 - 26).

Comp.	M.F.	Yield %	m.p/c°	Co lour	Cal.			Found		
					C	H	N	C	H	N
17	$C_7H_{15}N_5O_2S$	77	144-142	Brown	36.04	6.48	30.02	35.87	6.23	29.93
18	$C_5H_{13}N_5OS$	68	163-161	yellow	31.40	6.85	36.62	31.25	6.72	36.44
19	$C_{11}H_{20}N_6O_3S$	60	210-208	orange	41.76	6.37	26.56	41.56	6.20	26.47
20	$C_7H_{15}N_5O_2S$	58	230-228	Brown	36.04	6.48	30.02	35.88	6.25	29.86
21	$C_9H_{19}N_5O_2S$	70	186-184	Light brown	41.36	7.33	26.80	41.30	7.29	26.76
22	$C_{13}H_{24}N_6O_3S$	72	175-173	yellow	45.33	7.02	24.40	45.31	6.95	24.20
23	$C_{12}H_{17}N_5O_2S$	80	246-244	orange	48.80	5.80	23.71	48.24	5.66	23.65
24	$C_{14}H_{21}N_5O_2S$	73	250-248	yellow	51.99	6.54	21.65	51.79	6.38	21.40
25	$C_{16}H_{22}N_6O_3S$	66	220-218	orange	50.87	5.86	22.21	50.65	5.69	22.04
26	$C_{18}H_{26}N_6O_3S$	68	178-185	yellow	53.18	6.45	20.67	53.00	6.32	20.63

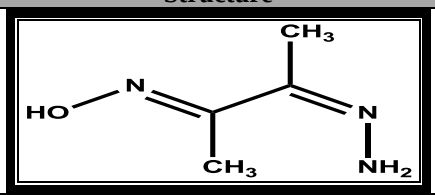
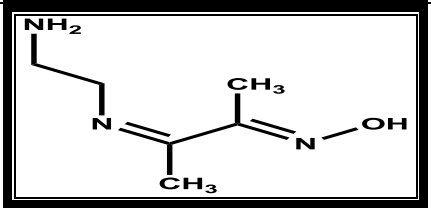
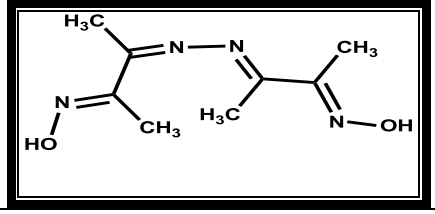
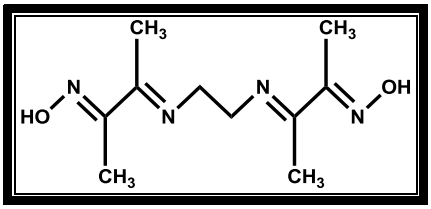
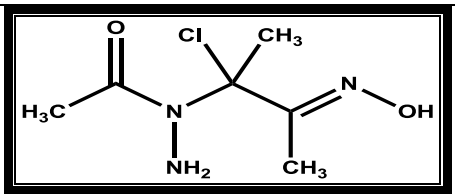
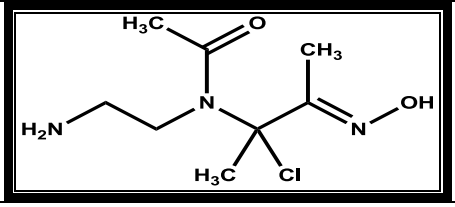
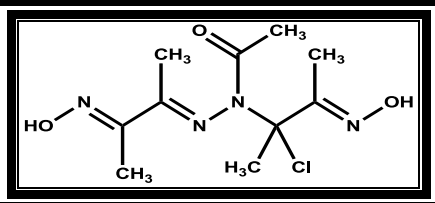
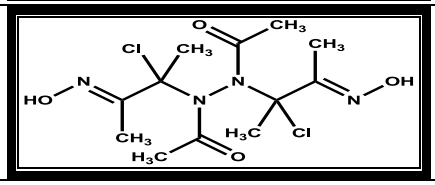
Table(7):IR spectral data of compounds(17-26).

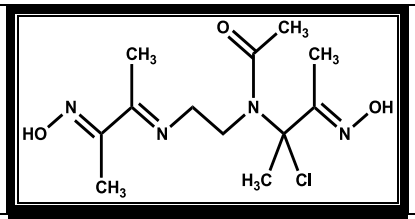
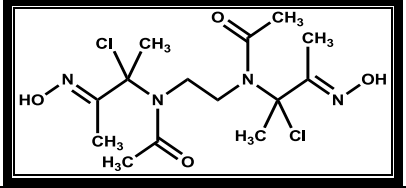
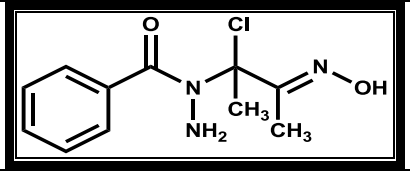
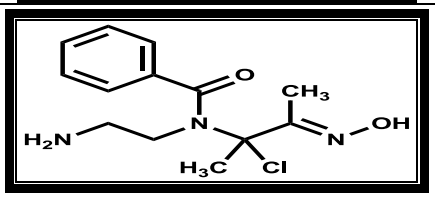
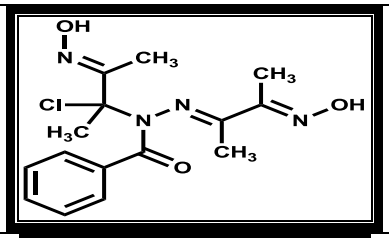
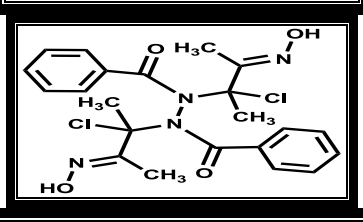
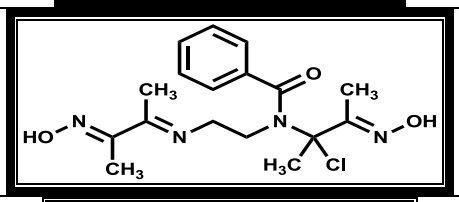
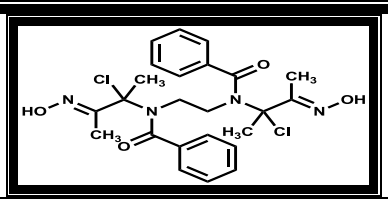
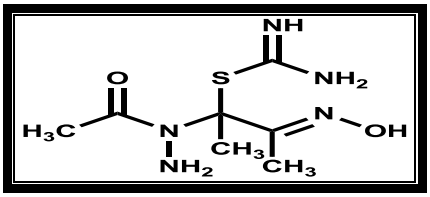
Characteristic bands of FT-IR spectrum							
Comp.	υ (NH_2) (cm^{-1})	υ (N-H) (cm^{-1})	υ (C-H) (cm^{-1})	υ (C=O) (cm^{-1})	C)=N υ((cm^{-1})	υ (C-S) (cm^{-1})	υ (C-N) (cm^{-1})
17	3410	3250	2890	1665	-	1250	1230
18	3440	3245	2880	1675	-	1265	1220
19	3410	3240	2895	1670	1635	1270	1225
20	3400	3230	2875	1680	-	1250	1230
21	3440	3200	2880	1680	-	1260	1240
22	3425	3220	2870	1680	1630	1280	1230
23	3430	3240	2890	1660	-	1280	1210
24	3425	4210	2890	1670	-	1290	1230
25	3400	4200	2870	1680	-	1275	1240
26	3430	3240	2880	1675	-	1285	1245

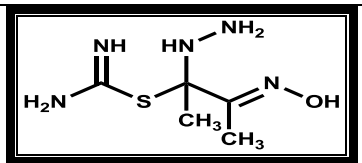
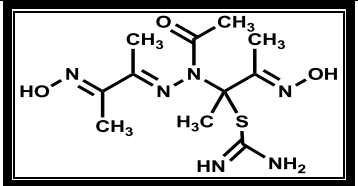
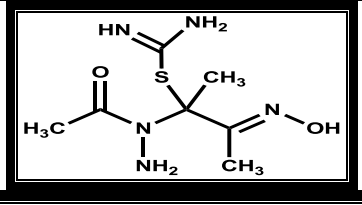
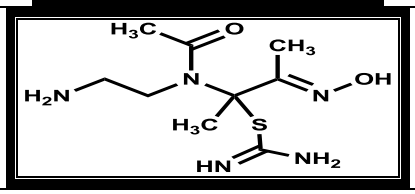
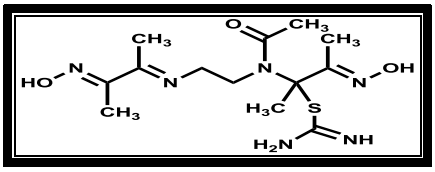
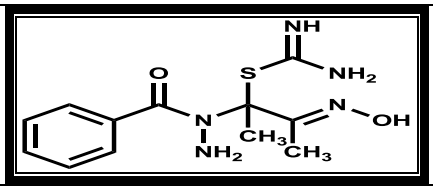
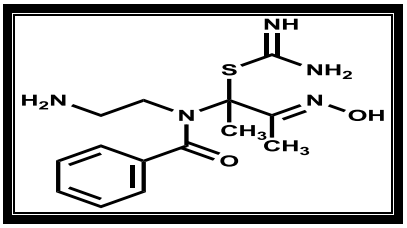
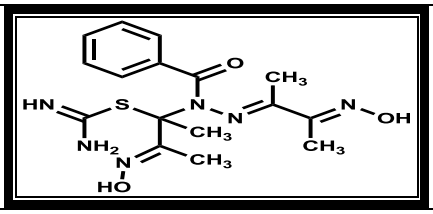
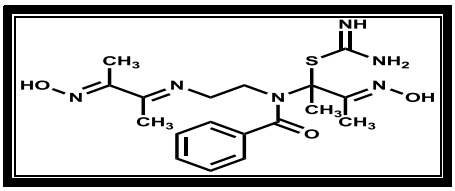
Table(8):UV Spectral data of Compounds (5-26)

Comp.No.	UV-Visible absorption maxima λ/nm
5	240,238, 230,226,223
6	239,236,230,226 222
7	238,235,231,229,225
8	240,237,234,226,222
9	240,239,236,232,226
10	238,235,230,229,225
11	340,320,255,236,232,225
12	356,340,278,244,237,230,226,222
13	354,320,275,240,238,234,228,224
14	350,332,295,245,238,230,225,220
15	366,343,300.259,240,236,233,226
16	370,337,299,262,240,237,230,225
17	240,238,235,229,226,224
18	238,236,230,228,227,223
19	239,237,234,229,226,220
20	240,237,236,230,227,225
21	238,235,230,229,226,224

22	238,235,233,228,226,220
23	365,330,300,287,240,236,233,226
24	370,346,332,305,277,250,240,236,230,226
25	374,356,306,295,266,239,237,230,227,223
26	380,370,337,300,287,254,240,237,230,228,225

No.	Name of compounds	Structure
1	(2*E*,3*Z*)-3-hydrazonobutan-2-one oxime	
2	(2*E*,3*Z*)-3-(2-aminoethylimino)butan-2-one oxime	
3	(2*E*,2'*E*,3*Z*,3'*E*)-3-((*E*)-((*E*)-3-(hydroxyimino) butan-2-ylidene)hydrazono)butan-2-one oxime	
4	(2*E*,2'*E*,3*Z*,3'*E*)-3-(2-((*E*)-((*E*)-3-(hydroxyimino)butan-2-ylidene)amino)ethylimino)butan-2-one oxime	
5	(*E*)-*N*-(2-chloro-3-(hydroxyimino)butan-2-yl)acetohydrazide	
6	(*E*)-*N*-(2-aminoethyl)-*N*-(2-chloro-3-(hydroxyimino)butan-2-yl)acetamide	
7	(*E*)-*N*-((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)-*N*'-((*E*)-3-(hydroxyimino)butan-2-ylidene)acetohydrazide	
8	*N*'-acetyl-*N*,*N*'-bis((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)acetohydrazide	

9	*N*-((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)-*N*-(2-((*E*)-((*E*)-3-(hydroxyimino)butan-2-ylidene)amino)ethyl)acetamide	
10	*N*,*N*'-(ethane-1,2-diyl)bis(*N*-((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)acetamide)	
11	(*E*)-*N*-(2-chloro-3-(hydroxyimino)butan-2-yl)benzohydrazide	
12	(*E*)-*N*-(2-aminoethyl)-*N*-(2-chloro-3-(hydroxyimino)butan-2-yl)benzamide	
13	(*E*)-*N*-((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)-*N*'-((*E*)-3-(hydroxyimino)butan-2-ylidene)benzohydrazide	
14	*N*'-benzoyl-*N*,*N*'-bis((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)benzohydrazide	
15	*N*-((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)-*N*-(2-((*E*)-((*E*)-3-(hydroxyimino)butan-2-ylidene)amino)ethyl)benzamide	
16	*N*,*N*'-(ethane-1,2-diyl)bis(*N*-((*E*)-2-chloro-3-(hydroxyimino)butan-2-yl)benzamide)	
17	(*E*)-2-(1-acetylhydrazinyl)-3-(hydroxyimino)butan-2-yl carbamimidothioate	

18	(*E*)-2-hydrazinyl-3-(hydroxyimino) butan-2-yl carbamimidothioate	
19	(*E*)-2-((*E*)-1-acetyl-2-((*E*)-3-(hydroxyimino) butan-2-ylidene)hydrazinyl)-3-(hydroxyimino)butan-2-yl carbamimidothioate	
20	(*E*)-2-(1-acetylhydrazinyl)-3-(hydroxyimino)butan-2-yl carbamimidothioate	
21	(*E*)-2-(*N*-(2-aminoethyl)acetamido)-3-(hydroxyimino)butan-2-yl carbamimidothioate	
22	(*E*)-3-(hydroxyimino)-2-(*N*-(2-((*E*)-((*E*)-3-(hydroxyimino)butan-2-ylidene)amino)ethyl)acetamido)butan-2-yl carbamimidothioate	
23	(*E*)-2-(1-benzoylhydrazinyl)-3-(hydroxyimino) butan-2-yl carbamimidothioate	
24	(*E*)-2-(*N*-(2-aminoethyl)benzamido)-3-(hydroxyimino)butan-2-yl carbamimidothioate	
25	(*E*)-2-((*E*)-1-benzoyl-2-((*E*)-3-(hydroxyimino) butan-2-ylidene)hydrazinyl)-3-(hydroxyimino) butan-2-yl carbamimidothioate	
26	(*E*)-3-(hydroxyimino)-2-(*N*-(2-((*E*)-((*E*)-3-(hydroxyimino)butan-2-ylidene)amino)ethyl)benzamido)butan-2-yl carbamimidothioate	

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اون -2- هيدرازونوبيوتان -3- (3E,2E)تحضير وتشخيص آ ميدات وثايوبوريزجديدة من تفاعل اون -2- يلدين) هيدرازونو) بيوتان-2- (هيدروكسي إمينو) بيوتان-3- ((E))-((Z)) 3-أوكسيم و اوكسيم مع كلوريد الاستيل والثايوبوريا

وليد فرج حمادي عبير عبيد وهب

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الخلاصة

-2- (هيدروكسي إمينو) بيوتان-3- ((E))-((Z)) 3- اون أوكسيم و -2- هيدرازونوبيوتان -3- (3E,2E)تعطي تفاعلات قواعد شيف جديدة 1- (-3- كلورو -3- (E) اون اوكسيم مع كل من كلوريد الاسيتايل وكلوريد البنزويل إيمايدات جديدة ، مشتقات -2-يلدين) هيدرازونو) بيوتان (-3- كلورو -2-) E((N– ثنائي يل) بس (2,1 (إيثان – N/,N– اون اوكسيم ومشتقات -2- يل) هيدرازينيل) بيوتان-2- ايـن-1-بروبان تم التثبت من صحة التراكيب المحضرة من خلال طيف . اسيتاميد) عند تفاعلها مع الثايو يوريا تعطي مركبات الثايوبوريز الجديدة -2-هيدروكسي ايمينو) بيوتان C.H.N.) والتحليل العنصري الدقيق FT-IRالأشعة فوق البنفسجية – المرئية وطيف الأشعة تحت الحمراء .)