

Synthesis and characterization of some new heterocyclic compounds with two Heteroatom's (Nitrogen)in their cyclic

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Abstract

The Pyrimidines derivatives play an essential role in several biological processes and have considerable important compounds for pharmaceutical medicinal and industrial material applications. The work describes the synthesis and characterization of some new pyrimidines derivatives: 2- mercapto 3,4 di hydro -4-oxo -[6- (4- alkoxy phenyl)- 4H] pyrimidine -5- carbonitriles and 2- mercapto 3,4 di hydro -4-oxo -[6-(4- substituted benzene - 4H)] pyrimidines -5- carbonitriles(3a- d). These Compounds (3a-d) were prepared from the reaction between ethylcyano acetate (0.01mole)and numbers of substituted benzaldehydes compounds (0.01mole) as starting materials. The compounds (4a-d) were obtained from the reaction of (3a-d) with aryl halide or alkyl halide under reflux for 5hrs, while the compounds (5a-d) were formed from the reaction of (4a-d) with phosphorous oxy chlorid. The compounds (6a-d) were synthesized from the reaction of (5a-d) with thiourea under reflux for 6hrs and the compounds (8a-d) were obtained from the reaction between (6a-d)with chloro acetic acid (0.01mole) under refluxed for 3hrs. All the synthesized compounds of pyrimidines derivatives were identified by the physical properties by it's melting points and colors, and the yields were characterized by the elemental (CHN)analysis, IR, UV, and visible spectra data

Key words: Ethyl cyano acetate, substituted benzaldehydes, aryl and alkyl halides, phosphorous oxy chloride, thiourea, chloro acetic acid, sodium ethoxide pyrimidines derivatives, elemental analyzer (CHN), IR, UV spectrum.

Introduction

A heterocyclic compounds are these which possess acyclic structure with two different kinds of atoms in the ring such as Nitrogen, Oxygen and sulfur are the most common heteroatom's. Most of the sugar, vitamins, alkaloids, which are Nitrogenous bases occurring in many antibiotics, including penicillin, are heterocyclic compounds(18). Pyrimidines derivatives have been found to possess interesting antibacterial(1), antipsychotic (22), anticancer (20), anti schizophrenia(5), and antihypertensive(12) activity anti viral(15), anti titumer(2),anti inflammatory (19), anti microbial (6)and anti fungal(11) anti histaminic (21), and analgesic, malaria, Alzheimers disease, Parkinson's disease (3) and anti oxidant properties (14). Pyrimidine nucleus occurs in a wide range of compounds having biological activity(10,7) and therefore, it was decided to synthesis some new pyrimidines derivatives, The pyrimidines ring is fused to various heterocyclic such as: (purine s in nucleic acids, pyrrolo pyrimidines, pyrido pyrimidines, pteridines, quinazolines, tri azolo Pyrimidines, pyrazolopyrimidines and furopyrimidines) are agro chemicals, and veterinary products (4, 16,17); fused pyrimidine continues to attract considerable attention because of their great practical usefulness primarily, due to very wide spectrum of biological activity(13). Thienopyrimidines occupy a special position among these compounds,

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along with some other pyrimidines systems containing an annulated five - membered heterocyclic ring. Thienopyrimidines are structural analogs of biogenic purines and can be considerable as potential nucleic acid antimetabolites(8). Aromatic and heteroaromatic compounds bearing an O-aminoester group are useful substrates for the preparation of various condensed pyrimidine heterocyclic system(9).

A series of Pyrimidines derivatives were synthesized by reacting ethyl cyano acetate with substituted aldehydes (1a-d) to form (3a-d) derivatives in (scheme -1-). The chemical names of the derivatives benzaldehydes : a= para- methoxy benzaldehyde , b= para- methyl benzaldehyde c= para- hydroxy benzaldehyde d= para- ethyl benzaldehyde. These Compounds (3a-d) were prepared from the reaction between ethylcyano acetate (0.01mole) and numbers of substituted benzaldehydes compounds (0.01mole) as starting materials. The compounds (4a-d) were obtained from the reaction of (3a-d) with aryl halide or alkyl halide under reflux for 5hrs ,while the compounds (5a-d) were formed from the reaction of (4a-d) with phosphorous oxy chlorid . The compounds (6a-d) were synthesized from the reaction of (5a-d) with thiourea under reflux for 6hrs and the compounds (8a-d) were obtained from the reaction between (6a-d)with chloro acetic acid (0.01mole) under refluxed for 3hrs. All the synthesized compounds of pyrimidines derivatives were identified by the physical properties by it's melting points and colors and the yields were characterized by the elemental (CHN) analysis, IR, U, and visible spectra data.

Aim of the Study

Synthesis and characterization of some new Pyrimidines derivatives by using ethyl cyano acetate and substituted Aldehyde as starting materials.

Materials and Methods

All melting points were recorded using electro thermal 9100 melting point apparatus and fourier transform infrared spectra were recorded using the KBr disc technique on A JASCO 440 FTIR spectra photo meter. The elementals (CHN) analysis were performed using an Exeter CE -440 Elemental Analyzer, and the UV spectrum was recorded on ashimadzu mini -1240 spectrophotometer.

The general procedures for preparation:

Method A: Preparation of Compounds (3a-d)

A mixture of substituted aldehydes (0.01mole) **ethyl cyano acetate** (0.01mole) Thiourea (0.01mole) and potassium carbonate (0.01mole) in ethanol was heated under reflux for 6hrs .The solid precipitated during the reaction was collected by stirred water and acidified with acetic acid .The deposited precipitate was collected, washed with water and crystallized from the proper solvent to give the products.

Method B: Preparation of Compounds (4a-d).

A mixture of (3a-d) (0.01mole) alkyl halide or aryl halide and potassium carbonate 0.01mole) in ethanol 40ml was heated under reflux for 5hrs ,allowed to cool and diluted with water. The solid product was filtered off and recrystallized from proper solvent .

Method C: Preparation of Compounds (5a-d)

A solution of (4a-d) 0.01mole in dioxane 40ml was heated with phosphorous oxy chloride 20ml and heated under reflux for 4hrs .The reaction mixture was cooled and poured into ice water. The solid form was collected, dried and recrystallized from the proper solvent.

Method D: Preparation of Compounds (6a-d)

A mixture of (5a-d) 0.01mole and thiourea 0.01mole in ethanol was heated under reflux for 6hrs. The reaction mixture was left to cool, then the solid form was filtered and recrystallized from the proper solvent.

Method E: Preparation of Compounds (8a-d)

A mixture of compounds (6a-d) 0.01mole, chloro acetic acid 0.01mole and sodium ethoxide (0.012mole) in ethanol 30ml was refluxed for 3hrs . The reaction mixture allowed to cool ,then filtered off and recrystallized from ethanol or chloroform.

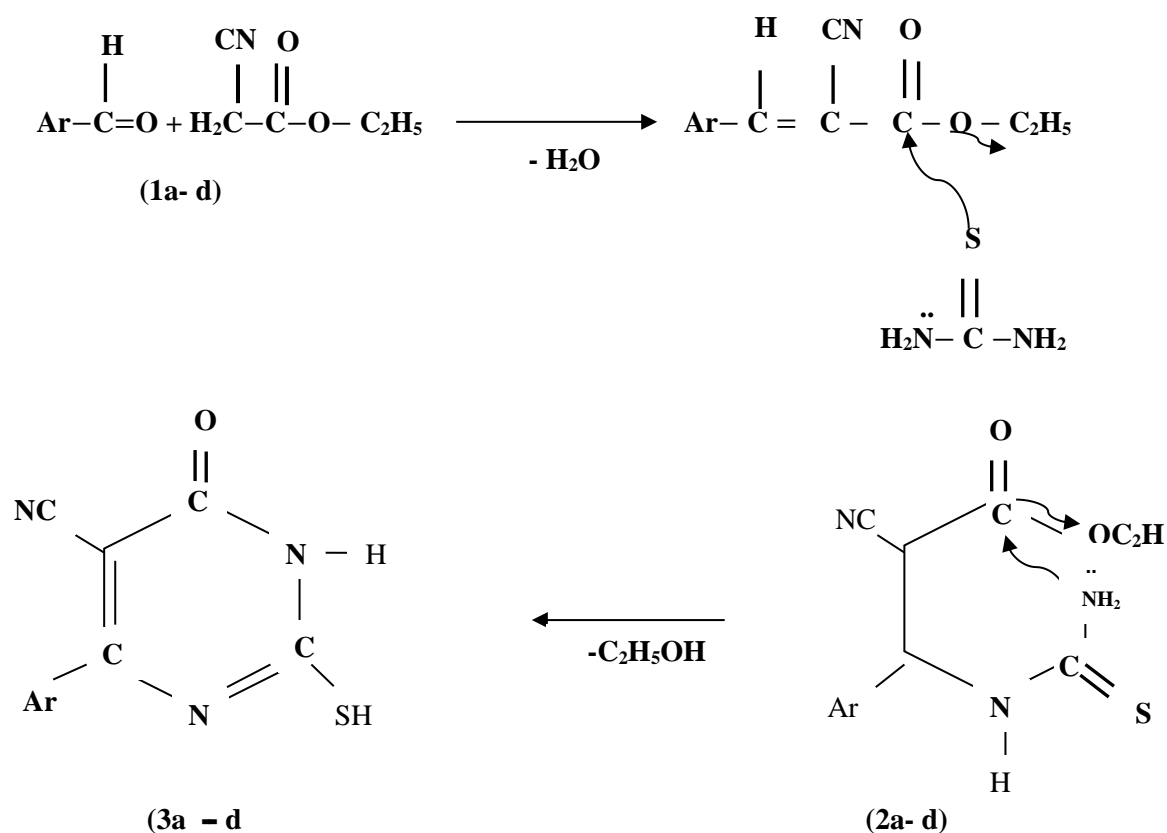
Results and Discussions

The pyrimidines derivatives were synthesized by the reaction between substituted benzaldehydes(1a-d)with ethyl cyano acetate and potassium carbonate in the present of ethanol as a solvent to give the intermediate compounds (2a-d)and the cyclo condensation of compounds (2a-d) with thiourea in ethanol yield of the products compounds (3a-d). The pyrimidines derivatives (3a-d) were identified by it's melting points(234-235°C) ,(242-243 °C),168.2 °C ,304decompose corresponding, while the yields of these compoundsare :73% ,65.7% ,56.5%,61.68%.

Table (1): The physical properties for the synthesized compounds (3a-d)

Compounds NO	Aryl	M. Formula M. weight g/mole	Yield % Color	M.P C°	C/F				
					C%	H%	N%	O%	S%
3a	C ₆ H ₅ -P-OCH ₃	C ₁₂ H ₉ N ₃ SO ₂ 254	73 white	234-235	55.59	3.47	16.21	12.15	12.35
					55.50	3.42	16.30	12.20	12.25
3b	-CH ₃ C ₆ H ₅ - p	C ₁₂ H ₉ N ₃ SO 243	65.7 white	242-243	59.25	3.70	17.28	6.58	13.16
					59.22	3.68	17.26	6.50	13.12
3c	C ₆ H ₅ – OH-P	C ₁₁ H ₇ N ₃ SO ₂ 245	56 white	168.2	53.87	2.85	17.10	13.06	13.06
					53.82	2.83	17.11	13.04	13.04
3d	C ₆ H ₅ CH ₂ CH ₃ -P	C ₁₃ H ₁₁ N ₃ SO 257	61.86 white	304	60.70	4.28	16.34	6.22	12.45
				Decompose	60.67	4.20	16.30	6.20	12.40

Scheme (1)



Where 2a and 3a Ar = P-OCH₃-C₆H₅

2b and 3b Ar = P-CH₃-C₆H₅

2c and 3c Ar = P-OH-C₆H₅

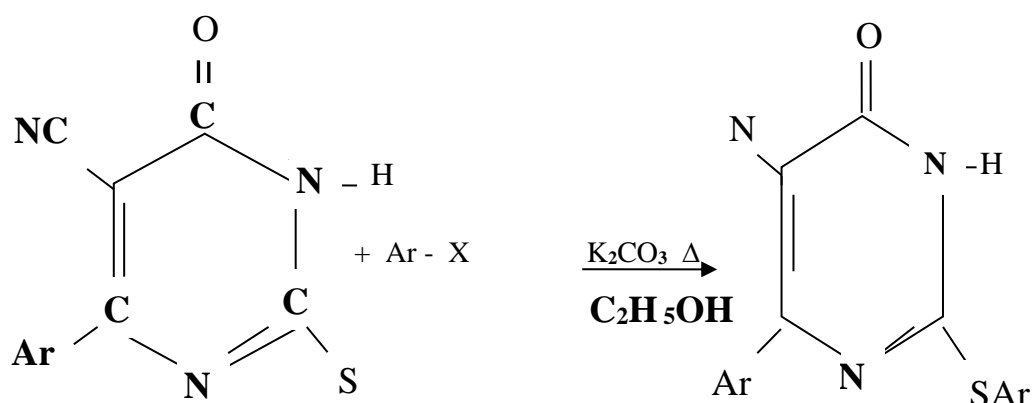
2d and 3d Ar = P-CH₃-CH₂-C₆H₅

The pyrimidines derivatives 4a, 4b, 5a, 6a, 8a were identified by melting points 4a= (156-157 °c) 4b= (281-282 °c) 5a=(180-181 °c) , 6a= 245 °c, 8a=304decompose corresponding, while the yields of these compounds are :70.83% 52.66% , 80% , 60% , 61.86. The synthetic pathway of the compounds (4a, 4b) were shown in Scheme (2) and (5a) in Scheme (3) and the compound (6a) in Scheme (4) ,while the synthetic of compound(8a) is shown in Scheme(5)

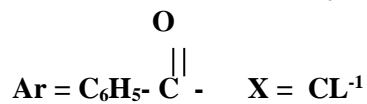
Table (2): The physical properties for the synthesized compounds 4a, 4b, 5a, 6a, 8a

Compounds NO	Aryl	M. Formula M. weigh g/mole	% yield color	M.P C°	C/F					
					C%	H%	N%	O%	S%	CL%
4a	C ₆ H ₅ OCH ₃	C ₁₉ H ₁₃ N ₃ S ₃ O ₃ 363	70.83 white	156-157	62.80	3.58	11.57	13.22	8.81	-
					62.78	3.57	11.50	13.20	12.25	
4b	C ₆ H ₅ CH ₃	C ₁₄ H ₁₃ N ₃ SO	52.66 white	281-282	61.99	4.79	15.49	5.90	11.80	-
					61.90	4.70	15.40	5.85	11.79	
5a	C ₆ H ₅ OCH ₃	C ₁₉ H ₁₂ N ₃ S ₂ O ₂ CL 381.5	80 white	180- 181	59.76	3.14	11.00	8.38	8.38	9.30
					59.70	3.10	10.90	8.33	8.30	
6a	C ₆ H ₅ OCH ₃	C ₁₉ H ₁₃ N ₃ S ₂ O ₂ 379	60 yellow	245	60.15	3.43	3.69	6.22	16.86	-
					60.10	3.40	3.60	6.20	16.80	
8a	C ₆ H ₅ CH ₂ CH ₃	C ₁₃ H ₁₁ N ₃ SO 257	61.86 white	304 decompose	60.70	4.28	16.34	6.22	12.45	-
					60.67	4.20	16.30	6.20	12.40	

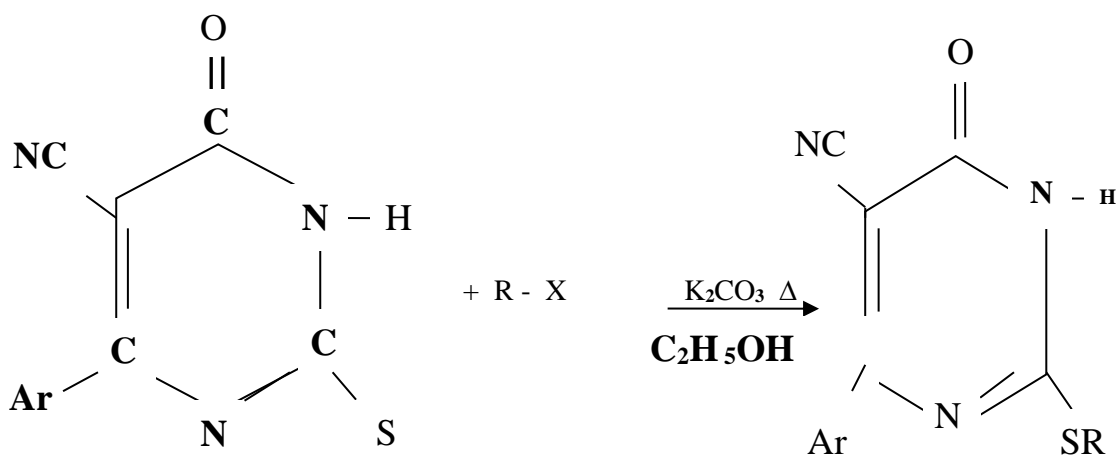
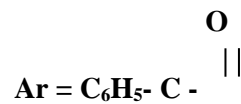
Scheme (2)



(3a)
Where 3a Ar = P- OCH₃ -C₆H₅



(4a)
Where 4a Ar = P- OCH₃ -C₆H₅



(3b-d)
where 3b Ar = p - CH₃ - C₆H₅ 4b

3C Ar = P- OH - C₆H₅

R = -CH₂-CH₃ X= I⁻

3d Ar = P- CH₃- CH₂ - C₆H₅

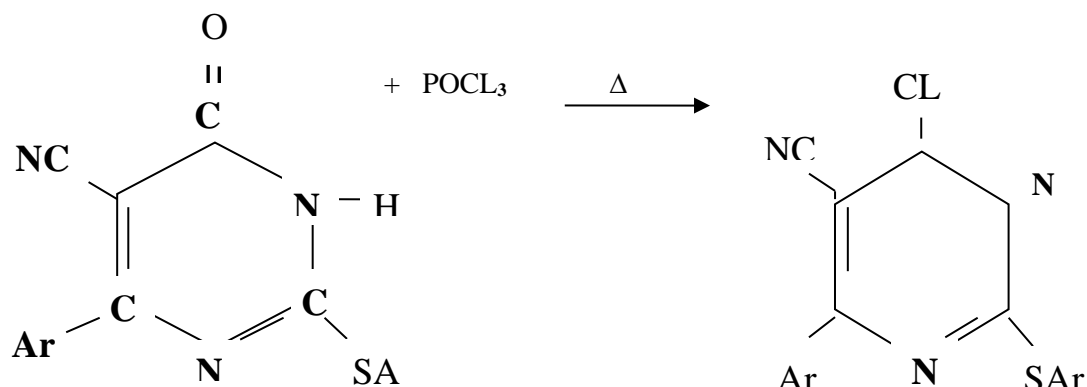
X= I⁻

(4b-d)
R = -CH₂-CH₃ R = -CH₂-CH₃ X= I⁻

4C R = -CH₂-CH₃

4d R= - CH₃R= - CH₃

Scheme (3)

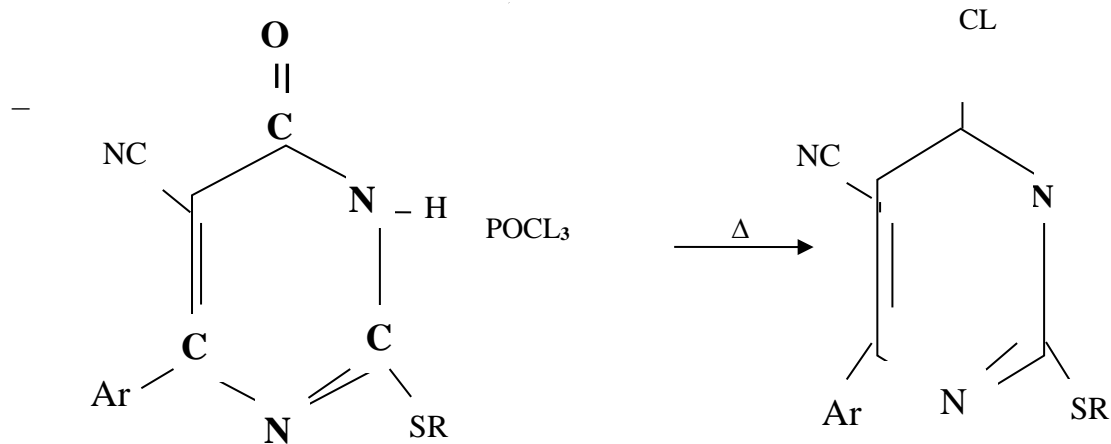
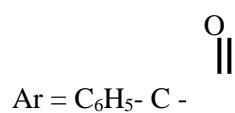
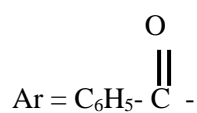


(4a)

Where 4a Ar = P- OCH₃ -C₆H₅

(5a)

Where 5a Ar = P- OCH₃ -C₆H₅



(4b -d)

(5b-d)

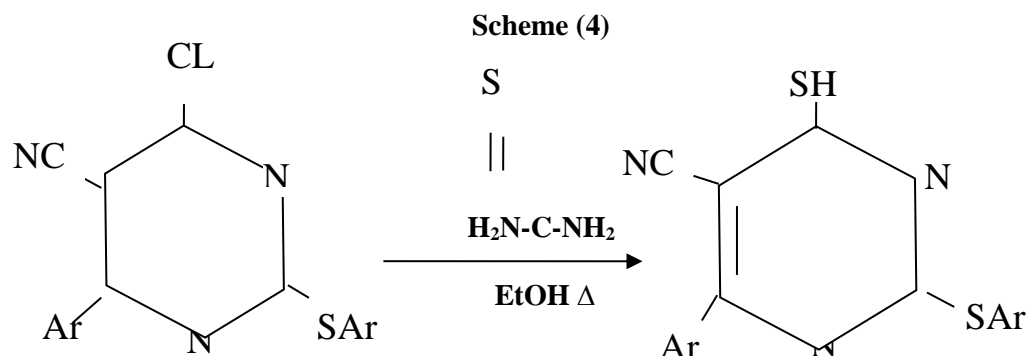
where 4b and 5b Ar = p - CH₃ - C₆H₅ R = - CH₂ -CH₃

4C and 5C Ar = P- OH - C₆H₅

R = - CH₂ -CH₃

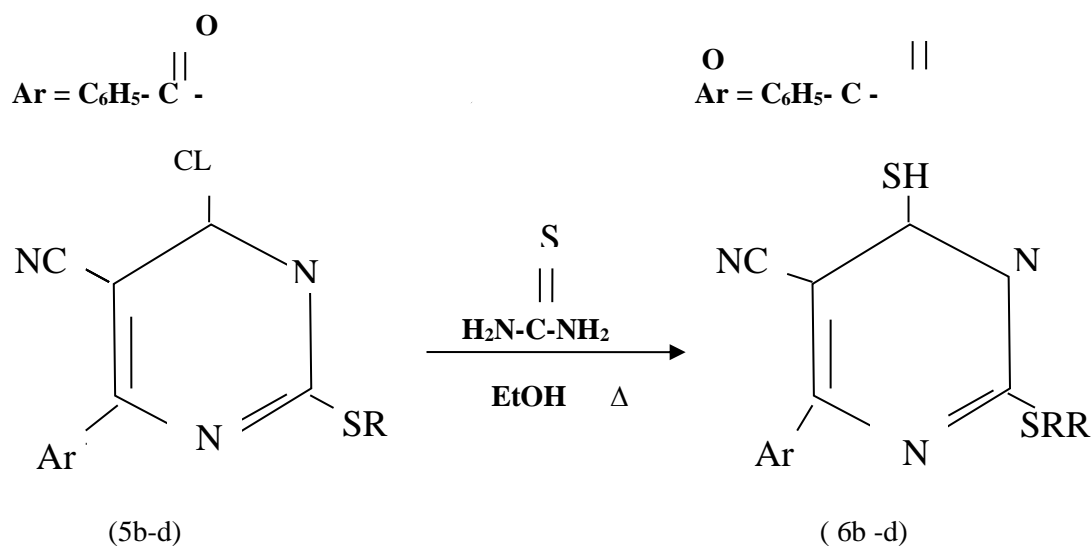
4d and 5d Ar = P- CH₃- CH₂ - C₆H₅

R= - CH₃



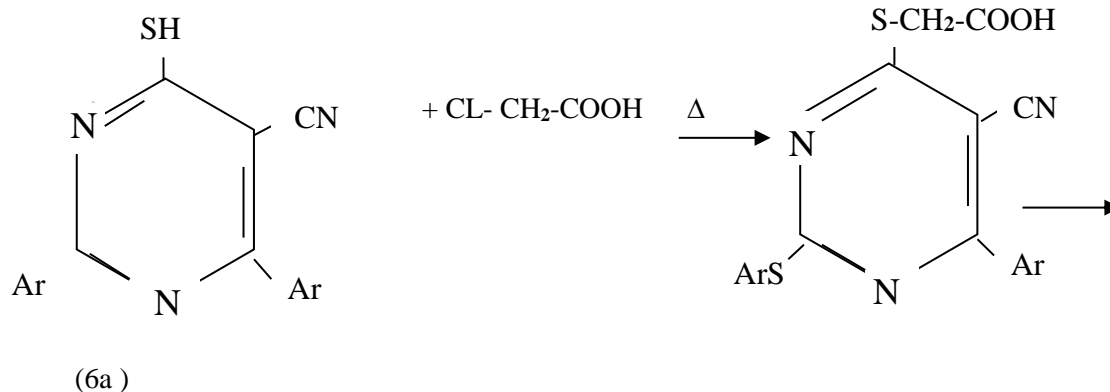
(5a)
Where 5a Ar = P- OCH₃ -C₆H₅

(6a)
Where 6a Ar = P- OCH₃ -C₆H₅

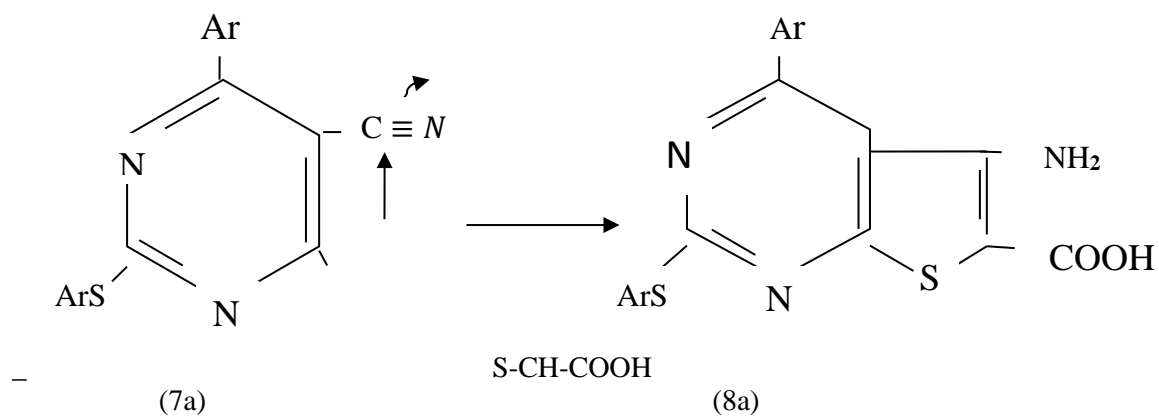
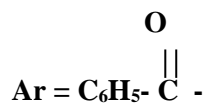


where 4b and 5b Ar = p - CH₃ - C₆H₅
 R = - CH₂ -CH₃
 4C and 5C Ar = P- OH - C₆H₅
 R = - CH₂ -CH₃
 4d and 5d Ar = P- CH₃- CH₂ - C₆H₅
 R = - CH₃

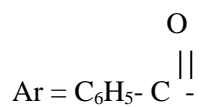
Scheme (5)

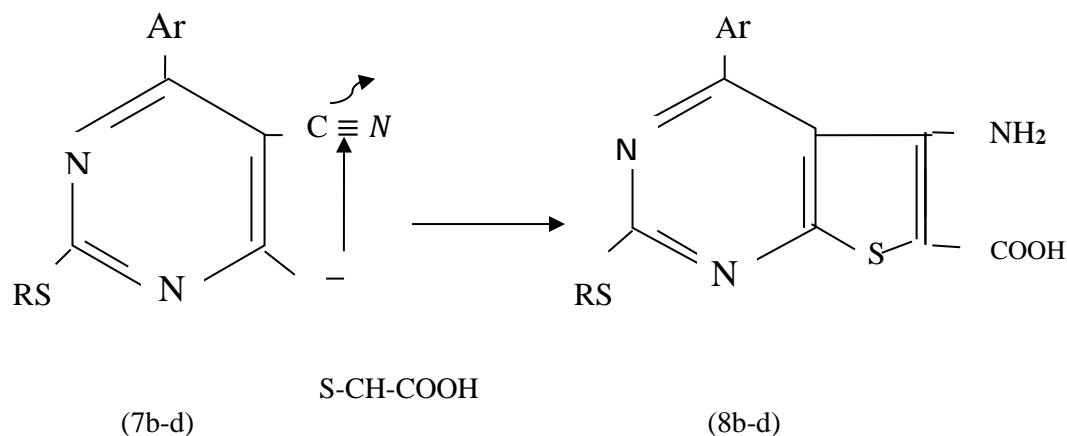
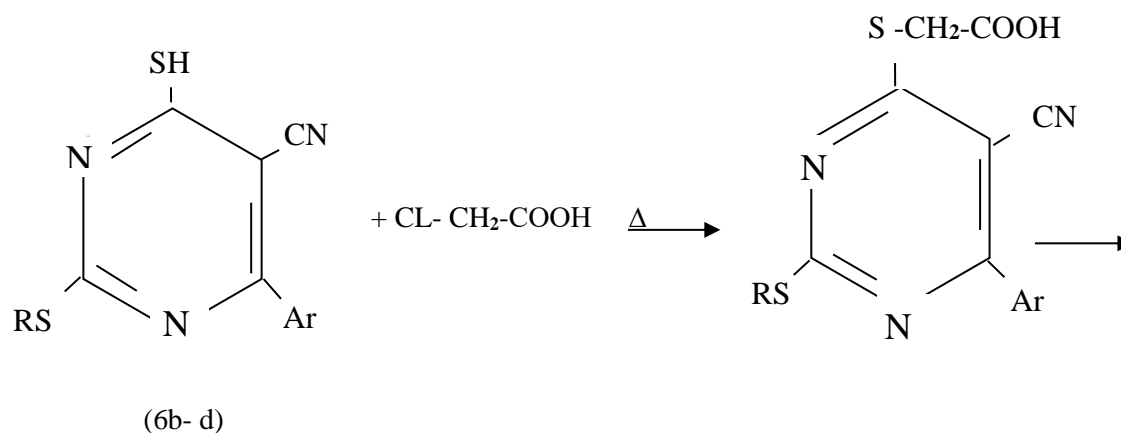


Where 6a Ar = P- OCH₃ -C₆H₅



Where 6a Ar = P- OCH₃ -C₆H₅





where 7b and 8b Ar = p - CH₃ - C₆H₅
 R = - CH₂ -CH₃
 7C and 8C Ar = P- OH - C₆H₅
 R = - CH₂ -CH₃
 7d and 8d Ar = P- CH₃- CH₂ - C₆H₅
 R= - CH₃

FT Infra Red Analysis of Compounds (3a - d):

The selected FTIR data are listed in Table (3) and the spectra of 3a, 3b, 3c, 3d are shown in Figures (1 - 4). The IR spectrum of compounds (3a, 3b, 3c, 3d) showed characteristic absorption bands at (3446 -3177) cm⁻¹ for (-NH) stretching bands and the appearance of broad band of (3c) compound at (3545 - 3445) cm⁻¹ was assigned to the stretching of ν - OH band. The main absorptions bands are observed at (1620- 1519)cm⁻¹ for (C = C), but it showed that (C= N) stretching vibration bands are (1666- 1588) cm⁻¹ and the appearance a strong bands at (1705- 1660) cm⁻¹ for (C= O) pyrimidinon; the bands with medium intensity observed (C ≡N) at (2236-2229) cm⁻¹ the appearance band observed the substituted benzen ring at (3063- 3023) cm⁻¹ scribed to the stretching of aromatic (C_{Ar}yl - H) and the bands observed at (2987- 2818) cm⁻¹ for the (C - H) and strong band for (C= S) at (1272 - 1228) cm⁻¹, while in compounds (3a, 3c) the stretching bands for (C- O) were at (1146- 1100) cm⁻¹ for methoxy and hydroxy groups respectively.

Table (3): The FTIR absorptions of the prepared compounds (3a-d)

Compounds No	- NH - OH	C _{Aryl} - H	C _{Ali} - H	C = C C = N	C ≡ N	C = O C - O	C = S
3a	3440 -	3063	2840	1573 1666	2236	1705 1146	1272
3b	3446 -	3059	2949-2818	1620 1640	2223	1680 -	1228
3c	3420-3290 3545-3445	3023	2987-2903	1519 1588	2231	1660 1100	1230
3d	3446-3177 -	3049	2968-2873	1567 1607	2229	1679 -	1240

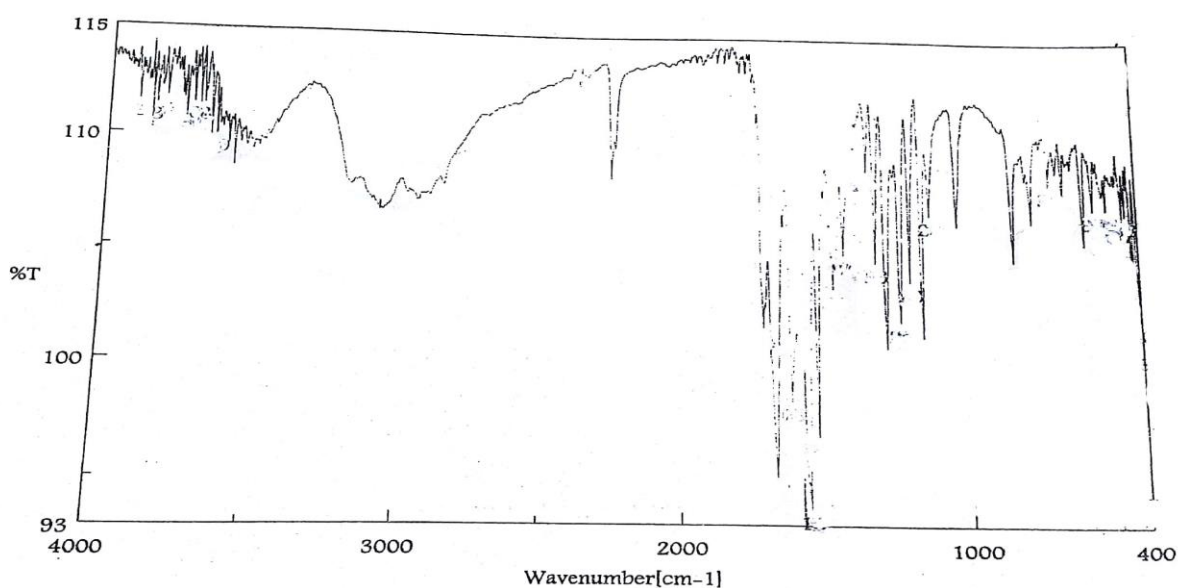


Fig (1): IR spectrum of compound (3a)

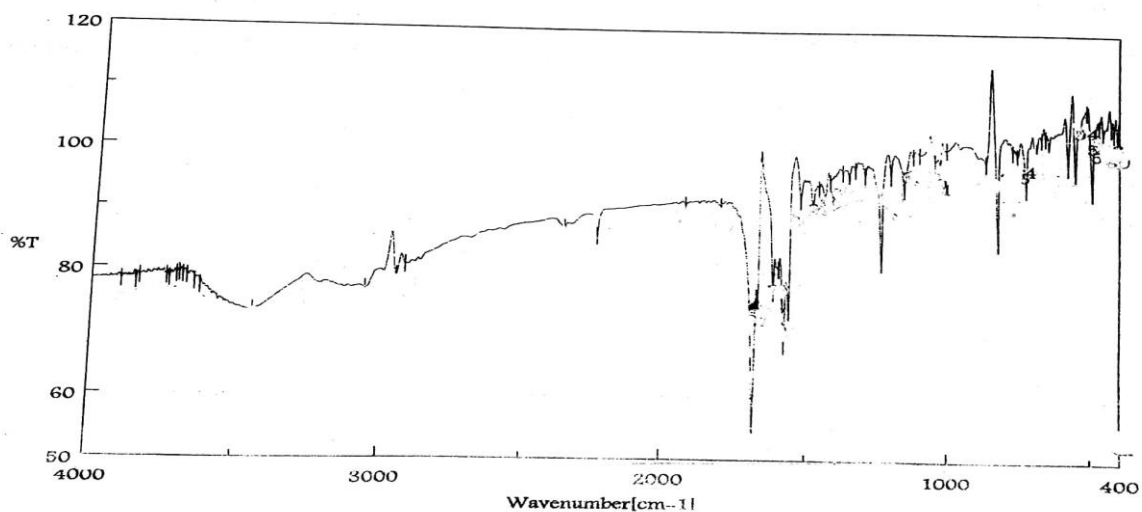


Fig (2): IR spectrum of compound (3b)

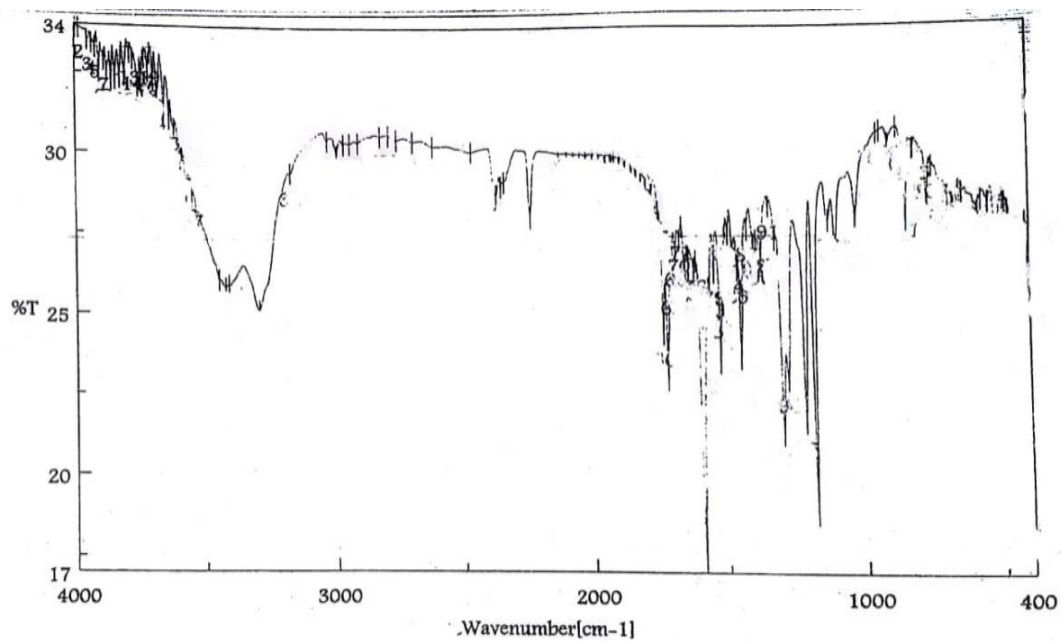


Fig (3): IR spectrum of compound (3C)

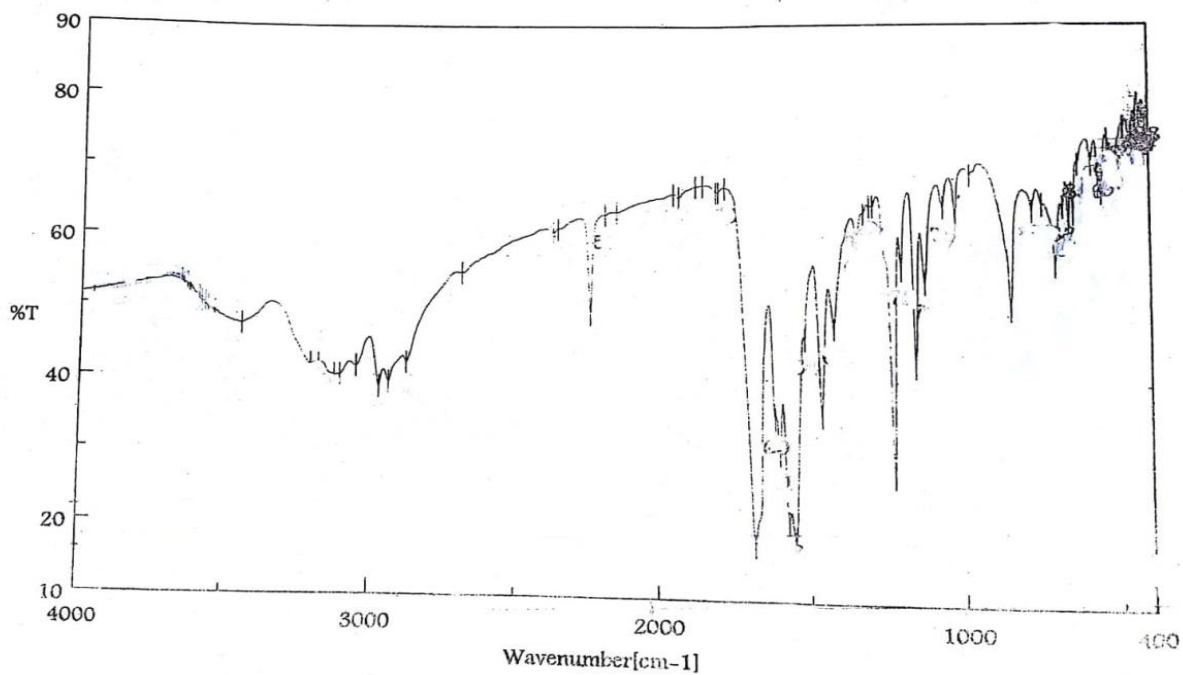


Fig (4): IR spectrum of compound (3d)

The Ultra Violet Spectrums of compounds (3a-d):

The selected VU data are listed in Table (4) and the spectra of 3a, 3b , 3c, 3d are shown in Figures (5 - 8). The appearance of three big bands in the compounds (3a, 3d) and absorptions of these compounds at (331- 222) nm indicated the presence of achromophoric group in (C = S) or (C = C) or (C= N) from ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) and the last band in the visible absorption . while in the compounds (3b,3c)appearance absorption was two big bands at(340-203) nm

Table (4): The UV absorptions of the prepared compounds (3a- d)

Compounds No	λ nm	ϵ	A
3a	222	73970	0.7397
	286	19300	0.1930
	330	11030	0.1103
3b	203	70470	0.7047
	340	29940	0.2094
3c	221	56330	0.5633
	338	15900	0.1590
3d	222	73580	0.7358
	268	19570	0.1967
	331	12210	0.1221

$A = \epsilon \cdot C \cdot L$

A = absorbance

ϵ = Molarity absorbance

C = the concentrate Molarities

L = length of cell

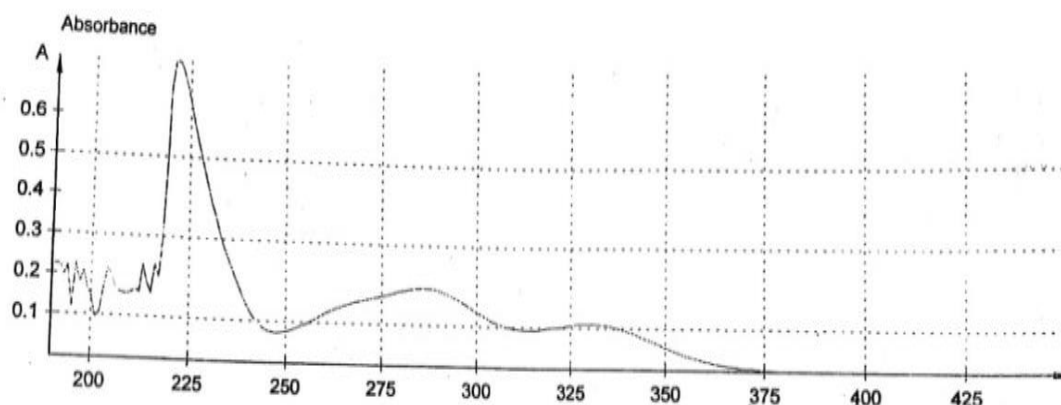
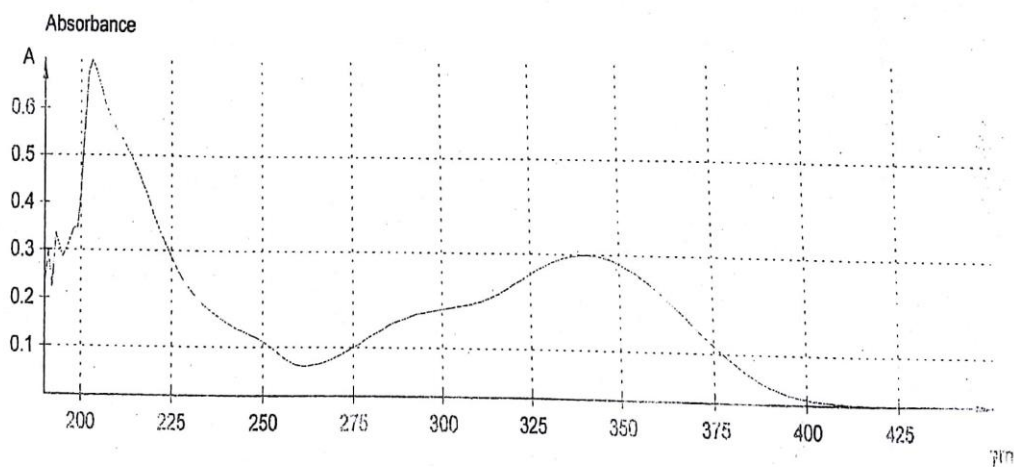


Fig (5): UV Spectrum of compound (3a)



Fig(6): UV Spectrum of compound (3b)

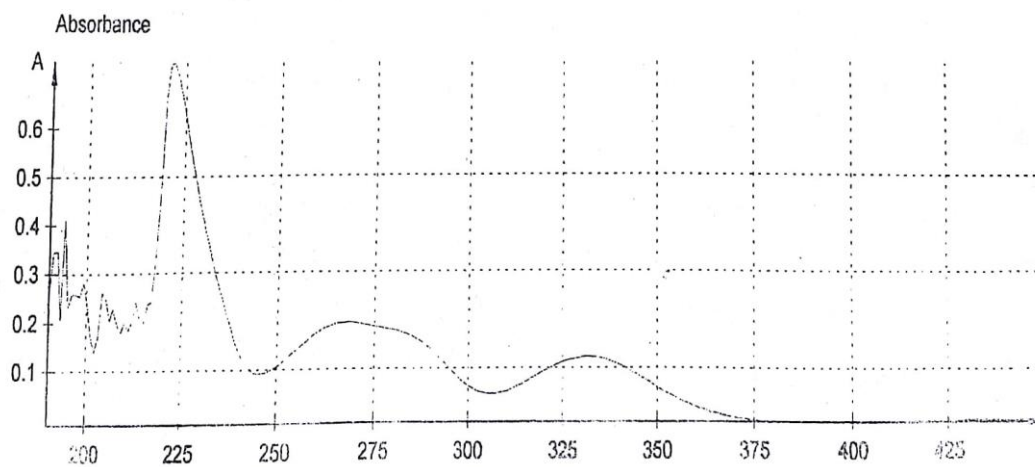


Fig (7): UV Spectrum of compound (3c)

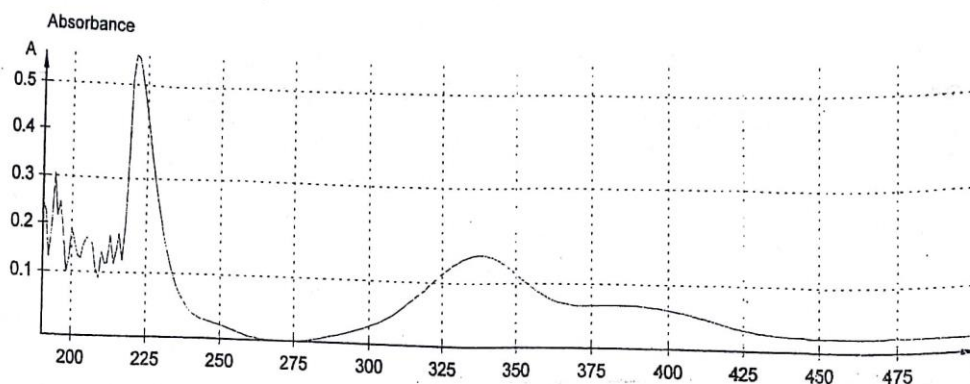


Fig (8): UV Spectrum of compound (3d)

The FT Infra Red Analysis of compounds (4a , 5a, 8a) :

The selected FTIR data of compounds 4a, 5a, 8a are listed in Table (5) and the spectra compound (4a) is shown in Figure (9). The IR spectrum of compound (4a) showed a characteristic absorption band at 3422 cm^{-1} for (-NH) and 1613 cm^{-1} for (C= N) 1676 cm^{-1} for (C = O) , 2218 cm^{-1} for (C \equiv N) and 1518 cm^{-1} for (C= C),1260 cm^{-1} for (C – O) and651 cm^{-1} or(C – S). The IR spectrum of compound (5a) showed characteristic absorption band at 1607 cm^{-1} for (C= N) , and 1532 cm^{-1} for (C= C) , 2224 cm^{-1} for (C \equiv N) , and 1276 cm^{-1} for (C – O) , and 840 cm^{-1} for (C – S), while 1148 cm^{-1} for (C_{Aryl}- cl). The spectra compound (5a) is shown in figure(10). The IR spectrum of compound (8a) showed a characteristic absorption band at 1646 cm^{-1} for (C = N) and 1577 cm^{-1} for (C = C), while the appearance of the observed big wide band for (-COOH) at 3423 cm^{-1} , and 1160 cm^{-1} for (C– O) , and 890 cm^{-1} for (C– S) the spectra compound of (8a) is shown in Figure(11).

Table (5) : The FTIR absorptions of the prepare compounds 4a , 5a, 8a .

Compo uns NO	- NH -OH -COOH	C _{Aryl} -H	C-H	C =C C = N	C \equiv N	C = O C – O	C – S	C _{Aryl} - cl
4a	3422 –	3015	2930- 2819	1518 1613	2218	1676 1260	661	–
5a	–	3071- 3006	2979-2843	1532 1607	2224	– 1276	840	1148
8a	- - 3423	–	2902	1577 1646	-	– 1160	890	–

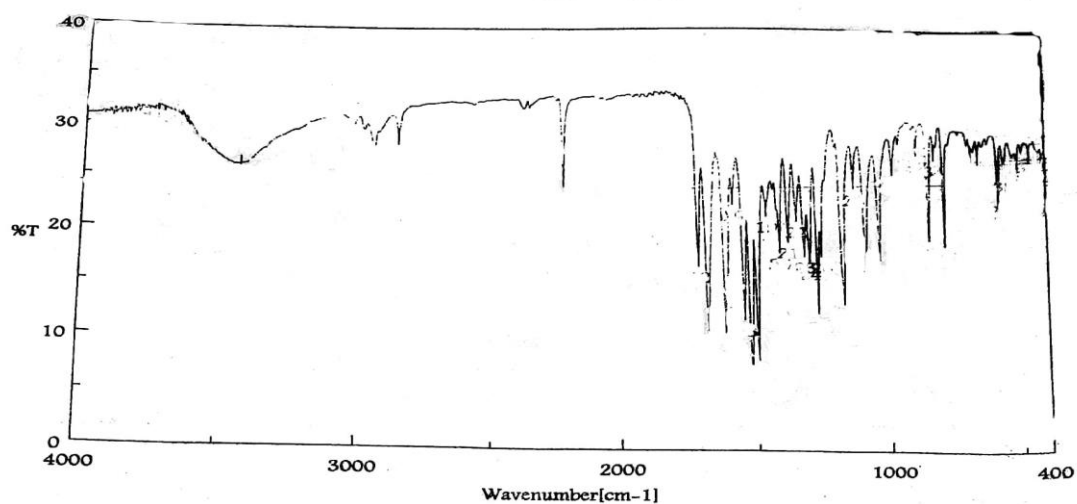


Fig (9): IR Spectrum of compound (4a)

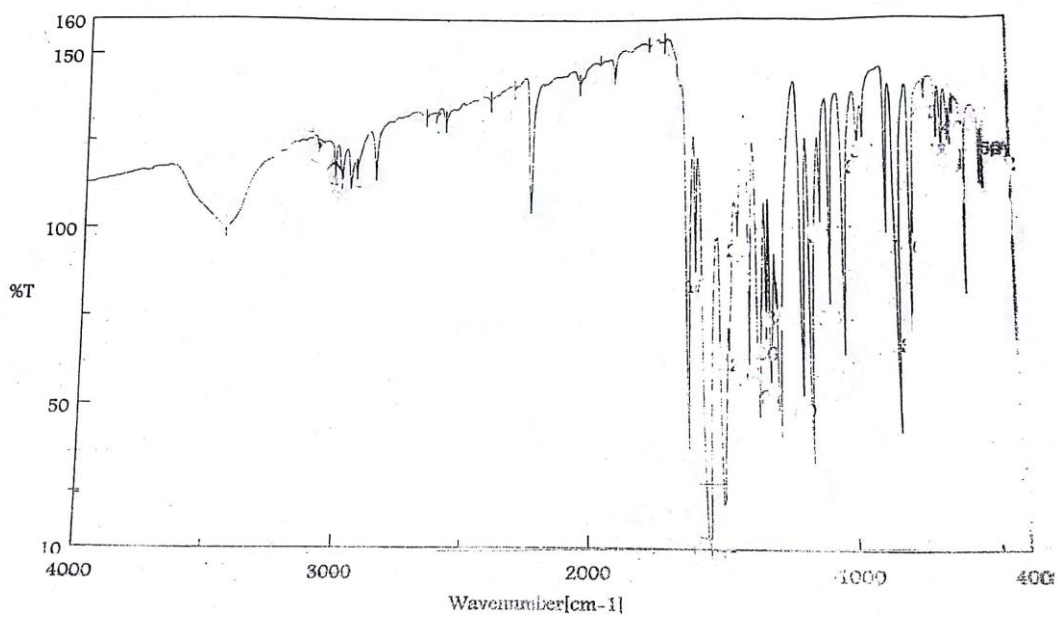


Fig (10): IR Spectrum of compound (5a)

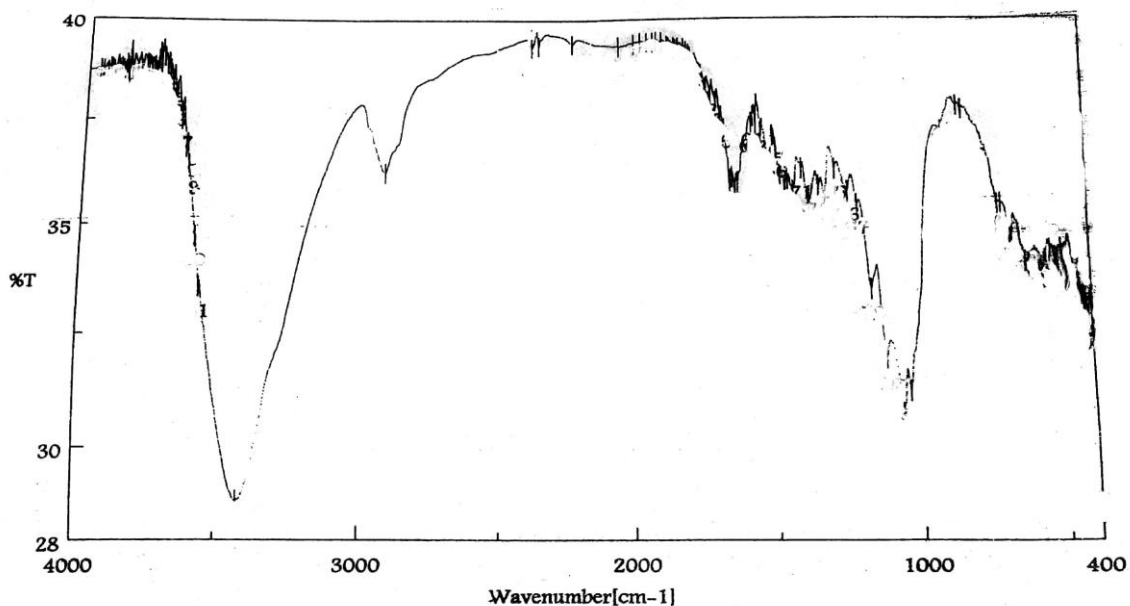


Fig (11): IR Spectrum of compound (8a)

The Ultra Violet spectrums of compounds(4a ,5a,8a)

The selected UV data are listed in Table (6) and the spectra of compound (4a) is shown in Figure (12) . The appearance observed only one big band absorption at 202 nm to indicate the presence of achromophoric group of double bonds in (C = C) or (C = N). The band absorption indicated the transfer from ($\pi \rightarrow \pi^*$) , while the VU absorption of compound (5a) showed two bands absorption at 202nm and215 nm. The spectra of this compound is shown in Figure (13) . The VU absorption of compound (8a) showed only one absorption band at 202nm and indicated the presence of achromophoric group for (– COOH) . The spectra of compound (8a) is shown in Figure (14)

Tabel (6): UV absorptions of the prepare compounds 4a , 5a , 8a

Compounds No	λ nm	ϵ	A
4a	202	20270	0.2027
5a	202	19660	0.1966
	215	10860	0.1086
8a	202	24980	0.2498

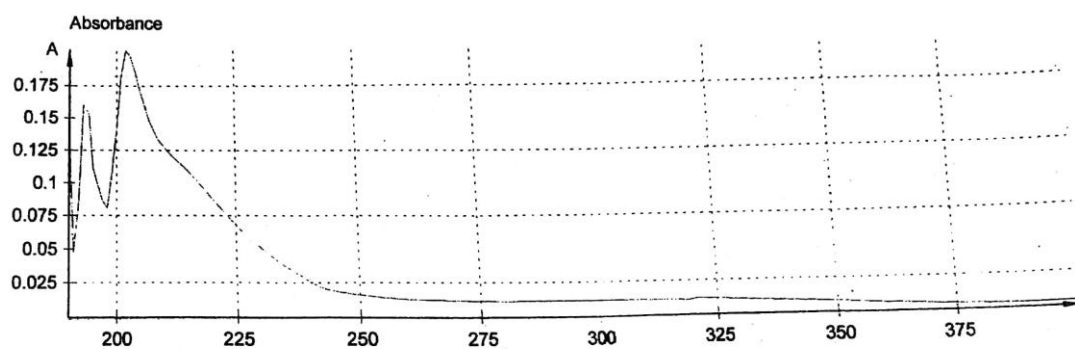


Fig (12): UV Spectrum of compound (4a)

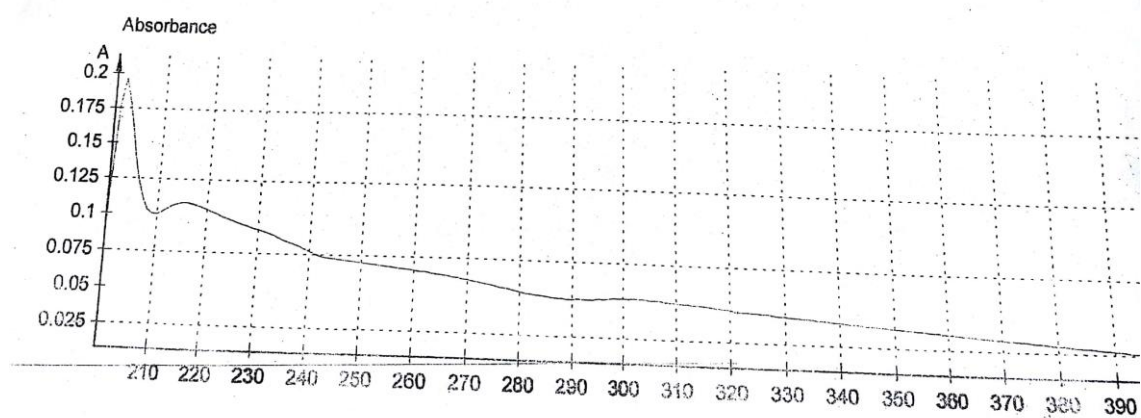


Fig (13): UV Spectrum of compound (5a)

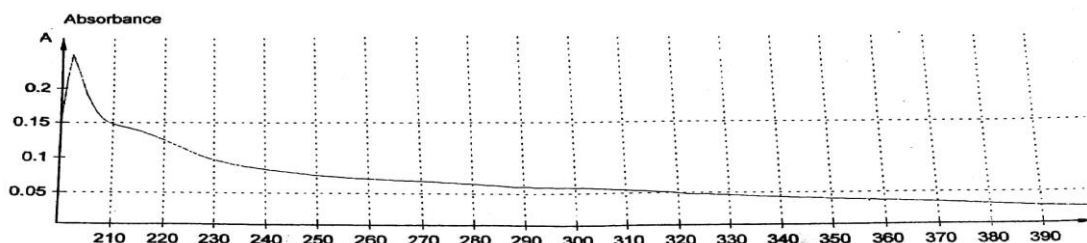


Fig (14): UV Spectrum of compound (8a)

Conclusion

- 1- A heterocyclic compounds are these which possess acyclic structure with two different kinds of atoms in the ring such as: Nitrogen, Oxygen and sulfur which are the most common heteroatom's. Most of the sugar, vitamins, alkaloids, which are Nitrogenous bases occurring in many antibiotics, including penicillin, are heterocyclic compounds. Pyrimidines derivatives have found to possess interesting antibacterial, antipsychotic, anticancer, antischizophrenia, and antihypertensive activity anti viral, anti tumor, anti-inflammatory, antimicrobial and antifungal, antihistaminic, and analgesic, malaria, Alzheimers disease, Parkinson's disease and antioxidant properties.
- 2- All the synthesized compounds of pyrimidines derivatives were identified by the physical properties by its melting points and colors and the yield were characterized by the elemental (CHN) analysis, IR, UV and visible spectra data.
- 3- The IR spectrum of the prepared compounds were characterized by higher intensities.
- 4- The absorption bands show higher frequencies for the prepared compounds, while in compound (8a) the appearance of a broad band was assigned to the stretching of ν -COOH band, and especially. The ν -(C=C), ν (C=N) band; this fact together with the chromophoric shift in (C=S) or (C=C) or (C=N) from ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) the UV Spectra.
- 5- Entrance of substituent's into the prepared compound (8a) leads to decreased intensity was suggested due to intermolecular hydrogen bonding, with disappearance of (C \equiv N) stretching which present in the parent compound.

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تخليق وتشخيص بعض المركبات الحلقية الجديدة غير المتجانسة المحتوية على ذرتي

نتروجين في تركيبها الحلقية

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الملخص

تقوم مشتقات البيريبيدين بوظيفة أساسية ومهمة في مختلف العمليات البيولوجية كما أن لها حضور في تركيب العديد من الأدوية والمستحضرات الطبية إذ أنها تستخدم صناعياً في إنتاج العديد من العقاقير الطبية كالمضادات الحيوية ومسكن الألم ومضادات البكتيريا ومضادات الالتهابات ومضادات الأكسدة والفطريات. وفي هذا العمل تم تخليق وتشخيص بعض المشتقات الجديدة للبيريبيدين وهي:

2-مركبتو3,4 ثنائي الهيدرو - 4 - أوكسو [6 - (4 - الكوكسي فينيل) - 4 H] بيريميدين - 5 - كاربونيتريل وكذلك 2-مركبتو3,4 ثنائي الهيدرو - 4 - أوكسو [6 - (4 - مشتقات (مستبدلات) البنزين - 4 H] بيريميدين - 5 - كاربونيتريل (3a - d). أما المشتقات الأخرى (4a-d) فقد تم الحصول عليها من التفاعل بين المركبات الناتجة (3a-d) التي استخدمت كمواد أولية مع أريل أو الكيل الهاليد وذلك بتسخينها باستخدام المكثف العاكس لمدة 5 ساعات تم الحصول على المركبات (5a- d) من تفاعل الفوسفواوكسي كلورايد مع مشتقات البيريبيدين (4a-d) وذلك بتسخينها باستخدام المكثف العاكس لمدة 4 ساعات. كذلك تم تحضير المركبات (6a-d) من تفاعل الثيوريا مع مشتقات البيريبيدين كمادة أولية (5a-d) في وجود الأيثانول وذلك بتسخينها باستخدام المكثف العاكس لمدة 6 ساعات، ناتج التفاعل من الخطوة السابقة (6a-d) تم معاملته مع كلوريد حمض الخليك وايتوكسي الصوديوم وبوجود الأيثانول كمذيب وباستخدام المكثف العاكس لمدة 3 ساعات تم الحصول على المركبات الحلقية لمشتقات البيريبيدين (8a- d).

وقد خضعت المركبات التي تم تخليقها لدراسة خواصها الفيزيائية مثل تعيين درجات الانصهار وتحديد ألوانها وكذلك المردود المئوي% (الحصيلة) كما تم تشخيصها من خلال تحليل العناصر ودراسة أطياف الأشعة فوق البنفسجية والأشعة تحت الحمراء.

الكلمات المفتاحية: أيثيل سيانو أسيتات، مشتقات (مستبدلات) البنزالدهيد، أريل والكيل الهاليد، الفوسفواوكسيكلورايد، الثيوريا، كلوريد حمض الخليك، أوكسي الصوديوم، مشتقات البيريبيدين، أطياف الأشعة فوق البنفسجية UV، الأشعة فوق الحمراء IR.