

Synthesis and Characterization of New Schiff's Bases Derived from D-Erythroascorbic Acid and Pyrimidines

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Abstract

The new C-5 schiff bases derived from D-erythroascorbic acid containing pyrimidine unit were synthesized by condensation of D-erythroascorbic acid with aromatic amine (containing pyrimidine unit) in dry benzene using glacial acetic acid as a catalyst.

D-erythroascorbic acid was synthesized by four steps (Schem 1), while the aromatic amine which is containing oxypyrimidine or thiopyrimidine synthesized by the reaction of chalcone urea or thiourea in acid or basic medium, respectively .

The structure of synthesized compounds have been characterized by their melting points , FTIR , UV-Vis and ¹HNMR spectroscopy . All the synthesized compounds have been screened for their antibacterial activities. They exhibited good antibacterial activity against Escherichia coli (G-) and Staphylococcus aureus (G+) , while the compounds [V]_b , [VI]_b and [VII]_b did not show any biological activity against this type of bacteria.

Key word : Schiff bases , L-Ascorbic acid , Pyrimidines

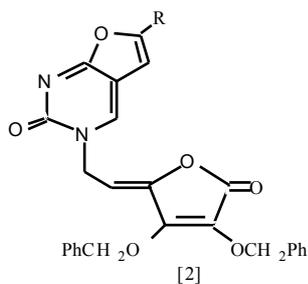
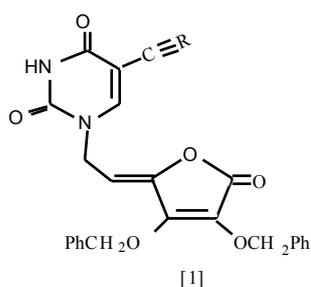
Introduction

L-Ascorbic acid is one of the most important biomolecules . It acts as an antioxidant and radical scavenger widely distributed in aerobic organisms [1]. L-Ascorbic acid derivatives have been found to possess antitumor and antiviral activities [2-4].

Pyrimidines have a great interest due to the wide variety of interesting biological activities observed for these compounds such as antiviral [5], antitumor [6], anticancer, anti-inflammatory [7] and antimicrobial [8] activities.

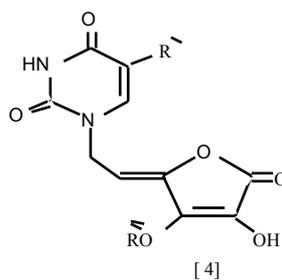
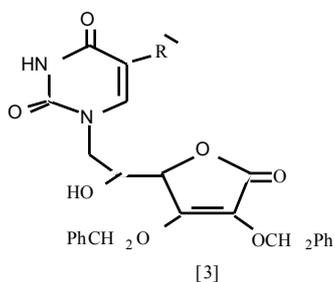
Pyrimidine nucleosides containing C-5 alkynyl groups have been shown to possess significant antiviral and anticancer properties [9].

Herdewijn [9] synthesized many C-5 substituted pyrimidine derivatives (1) and (2) of L-ascorbic acid. These compounds exhibited antiviral and cytostatic evaluations.



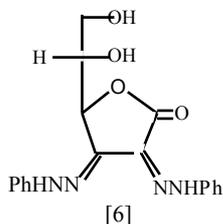
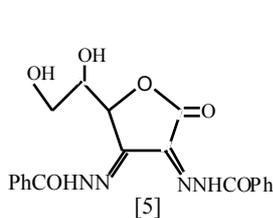
R = Alkyl or Aryl groups

Recently, Malic and et al [10] synthesized anti tumor pyrimidine derivatives of L-ascorbic acid (3) and (4).

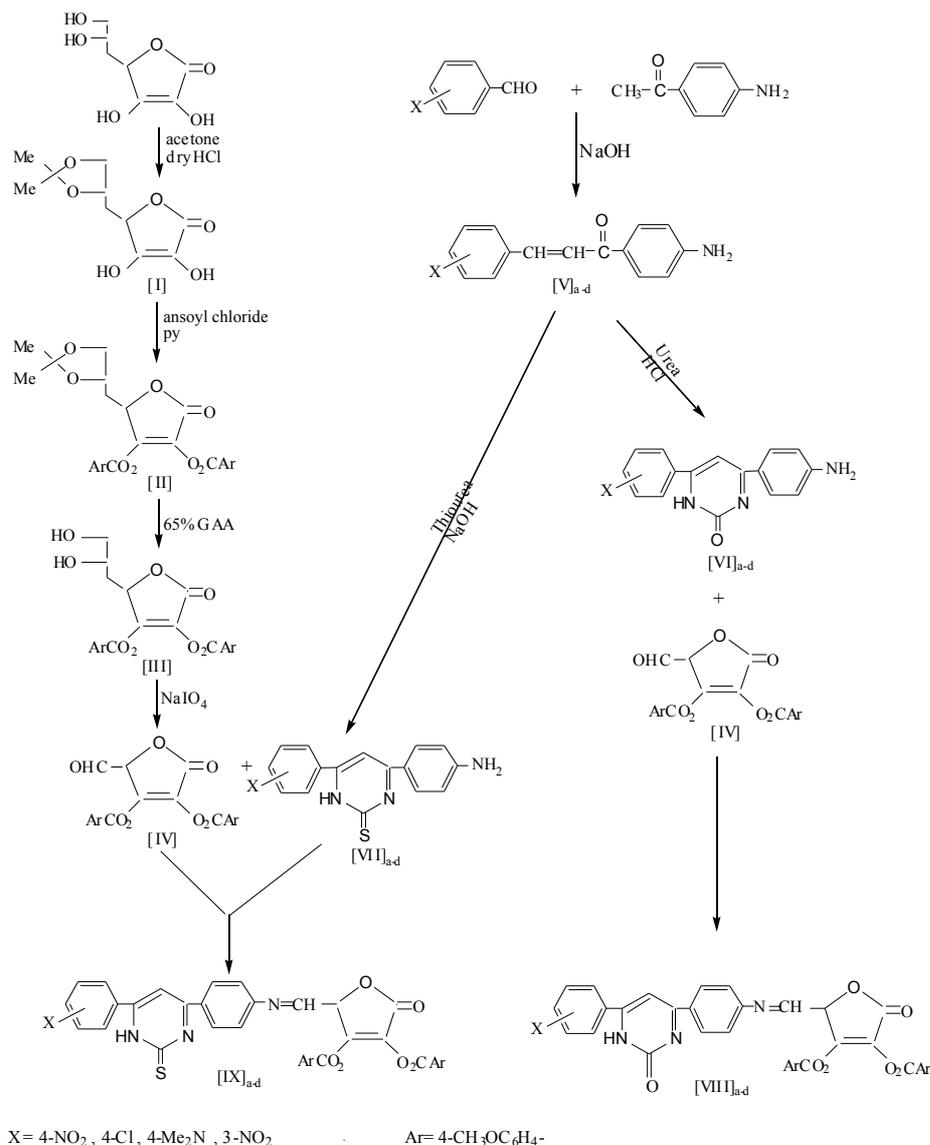


R' = H, F, CF₃ and R = H or CH₃

Ali et al [11] synthesized carbohydrate derivatives containing imine group as antibacterial. More recently, El-Sayed et al [12] and EL-Sekily [13] synthesized L-ascorbic acid derivatives containing imine group at 2- and 3- position, compounds (5) and (6), respectively.



Here in this work (Scheme 1), we reported the synthesis, characterization and antibacterial activity of novel imines of L-ascorbic acid containing pyrimidine unit.



Scheme 1

Experimental

Materials : All chemicals were supplied from Merck, GCC and Aldrich Chemicals Co. and used as received.

Techniques : FTIR spectra were recorded using potassium bromide discs on a 8400s Shimadzu spectrophotometer and FTIR spectrophotometer, Shimadzu (Ir prestige-21). ¹HNMR spectra were carried out by: Bruker, model: ultra shield 300 MHz, origin: Switzerland and are reported in ppm(S), DMSO was used as a solvent with TMS as an

internal standard . Measurements were made at chemistry department, Al-albyat university , Uncorrected melting points were determined by using Hot-Stage, Gallen Kamp melting point apparatus. UV spectra of solutions were performed on CECL 7200 Ingrand Spectrophotometer using CHCl_3 as a solvent .

Synthesis

preparation of 5,6-O-isopropylidene-L-ascorbic acid[I]:

This compound was prepared from the reaction of L-ascorbic acid with Acetone in a acidic media , following Salomon methode [14].

Synthesis of 2,3-O-dianisoyl-5,6-O-isopropylidene-L-ascorbic acid [II]:

To a cold solution of [I](10gm , 0.046mol) in pyridine(50 mL) , Anisoyl chloride was added (17.5mL , 0.129mol) with stirring for 2 hrs, then kept in dark place at room temperature for 24 hrs.The mixture was poured into ice-water the oil layer was extracted with (150ml) chloroform,washed with water and dried over anhydrous magnesium sulfate [15]. Filtered and the solvent evaporated, purified from chloroform:petroleum ether(1:5) to give[II] (15gm,76.5%) as a pale yellow solid ,m.p(102-104 $^{\circ}\text{C}$) Rf(0.80) (benzene:methanol) (5:5).

Synthesis of 2,3-O-dianisoyl-L-ascorbic acid[III]:

Compound[II] (10gm, 0.0236 mol) was dissolved in a mixture of (65%) acetic acid (30ml), absolute methanol(10mL) and stirred for 48 hrs at room temperature. To the resulting solution a benzene(40ml) was added and evaporated to yield[III] [16], yield (7gm, 78%) as a white crystals, m.p(130-132 $^{\circ}\text{C}$), Rf (0.42) (benzene:methanol) (4:6).

Synthesis of pentulosono-Lacton-2,3-ene - dianisoate [IV]:

To a stirred solution of sodium periodate (5.6gm) in distilled water (60mL) at (0 $^{\circ}\text{C}$), a solution of [III] (10gm, 0.026mol) in absolute ethanol (60mL) was added dropwise . After stirring 15 min, ethylene glycol (0.5mL) was added and stirring for one hour. The mixture was extracted with ethyl acetate (3x50ml)[17]. The extracts dried over anhydrous MgSO_4 , filtered and the solvent evaporated , the residue recrystallized from benzene to yield [IV] (4 gm, 45%) as a white crystals , m.p (156-158 $^{\circ}\text{C}$), Rf(0.7) (benzene: methanol) (6:4).

Synthesis of chalcone: 4-[3-(4'-substituted phenyl)-2-propene-1-one]-aniline [V]_{a-d}:

Equimolar quantities of 4-amino acetophenone (0.01 mol),(1.35 g) and 4- or 3-substituted benzaldehyde (0.01 mol) were dissolved in minimum amount of alcohol. Sodium

hydroxide solution (0.02 mol) was added slowly and the mixture became cold. Then the mixture was poured slowly into 400 mL of ice water with constant stirring and kept in refrigerator for 24 hrs [18]. The precipitate obtained was filtered , washed and recrystallized from chloroform .

Synthesis of 4[6-(4`- substituted phenyl)-2-oxo-1,2, -di -hydropyrimidine-4-yl] aniline [VI]_{a-d} :

A mixture of chalcone[V]_{a-d} (0.001 mol) and urea (0.06 gm, 0.001mol) in ethanol (20mL) and conc.hydrochloric acid (5mL) was refluxed for 6 hrs .The reaction mixture was then concentrated to half of its volume .Cooled and

neutralized with ammonium hydroxide. The precipitated solid was filtered off , washed with water [19] , dried and recrystallized from ethanol .

Synthesis of 4-[6-(4`-substituted phenyl)-2-thioxo-1,2-dihydropyrimidine-4-yl] aniline [VII]_{a-d} :

A mixture of chalcone [VI]_{a-d} (0.001mol), thiourea (0.076gm , 0.001mol) and sodium hydroxide (0.1 g) in (25 mL) of 80% (v\ v) ethanol was refluxed for 6hrs. The reaction mixture was concentrated , cooled and the solid was filtered off , washed with water[19] , dried and then crystallized from ethanol.

Physical data of compound[V]_{a-d} , [VI]_{a-d} and [VII]_{a-d} are given in Table 1.

Synthesis of Schiff bases [VIII]_{a-d} and [IX]_{a-d}

A mixture of new amino compounds [VI]_{a-d} (0.01 mol) , aldehyde [IV] (0.012 mol) , dry benzene (15 mL) and 2 drops of glacial acetic acid was refluxed for 6hrs . The solvent was evaporated under vacuum and the residue crystallized from chloroform. The physical data of all Schiff bases are listed in Table 2.

Results and Discussion

5,6-O-isopropylidene-L-ascorbic acid[I] was prepared by the reaction of L-ascorbic acid with acetone in dry HCl (14) . The FTIR spectrum showed a broad stretching band at (3240-3074) cm^{-1} for(O-H) vinylic, stretching bands at (2993-2908) cm^{-1} for (C-H) aliphatic, acetal linkage stretching band at(1755) cm^{-1} due to (C=O) of Lactone ring , stretching band at(1685) cm^{-1} for (C=C) and stretching bands at (1141-900) cm^{-1} for C-O stretching .

Compound [I] reacts with excess of anisoyl chloride in dry pyridine to give the corresponding ester [II]. The FTIR spectrum exhibited appearance of stretching band

(C=O) of the ester, and disappearance of the stretching bands for (O-H) of compound [I], stretching bands at (2961-2935) cm^{-1} for (C-H) aliphatic group, finally stretching band at (1604) cm^{-1} could be attributed to (C=C) aromatic. The hydrolysis of compound [II] in acid media result hydrolyzed of isopropylidene ring to yield 2,3-O-dianisoyl-L-ascorbic acid [III] which characterized by melting point and FTIR. The FTIR spectrum showed a band at (3445) cm^{-1} for (O-H), a stretching at (3074) cm^{-1} for (C-H) aromatic.

Glycols [III] oxidized by periodate, which cleaves the C5- C6 bond (bearing OH groups) and formation the aldehyde compound D-erythroascorbic acid [IV]. This compound is characterized by melting point, FTIR, UV-VIS, Mass and ^1H NMR spectroscopy. The FTIR spectrum showed two bands at (2839-2677) cm^{-1} for (C-H) aldehyde stretching, a stretching band at (1715) cm^{-1} for (C=O) of aldehydic group, UV-Vis showed λ_{max} at 300 nm. Mass spectrum showed $M + 1 = 413$. ^1H NMR spectrum (δ , DMSO) showed the following signals: a singlet signal at δ (12.5) ppm that could be attributed to the aldehydic proton. Two doublets of doublets in the region δ (7.00 – 7.97) ppm due to eight aromatic protons, a singlet at δ (3.86) ppm for proton of lactone ring at C4. A sharp singlet at δ (3.82) ppm for the (OCH₃) group.

Chalcones [V]_{a-d} are synthesized by Claisen-Schmidt condensation of 4-aminoacetophenone and 4- or 3- substituted benzaldehyde by base catalyzed followed by dehydration to yield the desired chalcones. The structural assignments of the chalcones are based on melting points and their spectral data of FTIR, UV-Vis and ^1H NMR spectroscopy. The FTIR spectra indicated the appearance of two bands in the region (3483-3273) cm^{-1} which could be attributed to a symmetric and asymmetric stretching vibration of NH₂ group, a weak band at (3119-3105) cm^{-1} due to stretching vibration of (CH=CH) group, two peaks at (1650) cm^{-1} and (1635) cm^{-1} are due to (C=O) and (C=C) stretching vibration, respectively. The FTIR spectral data and UV-Vis data for the chalcones are listed in Table (3). The ^1H NMR of chalcone [V]_a (δ , DMSO) fig (1), shows the following features: two pairs of doublets of doublets in the region δ (7.6-8.2) ppm which can be attributed to eight protons of two p-substituted benzene rings showing different substitution at positions 1,4. A doublet band at δ (6.6) ppm is due to two protons of (COCH=CH) [19] moiety and a doublet band at δ (8.3) ppm for proton of (=CHAr). The two protons of amine group appear as a singlet band at δ (6.24) ppm.

The oxopyrimidine was synthesized from reaction of chalcone [V]_{a-d} with urea in acidic medium. The structure of the oxopyrimidine [VI]_{a-d} characteristic by FTIR spectra which are shown the disappearance of two absorption bands and new absorption bands for NH, C=O (amide) and C=N (endocyclic at (3433) cm^{-1} , (1639) cm^{-1} and (1610) cm^{-1} , respectively. FTIR characteristic bands and UV-Vis data of the synthesized compounds [VI]_{a-}

_d listed in Table (4) . ¹HNMR spectrum of compound [VI]_b fig (2) , shows the following signals: eight aromatic protons appeared as two pairs of doublet at δ (6.7-7.5) ppm and δ (7.9-8.0) ppm , a singlet signal at δ (7.6) ppm could be attributed to the one proton of 1H (oxo-pyrimidine) and a singlet at δ (7.5) ppm due to the proton of CH(oxo-pyrimidine) , as singlet broad signal two protons of NH₂ group appeared as δ (5.0) ppm [19].

Thiopyrimidines[VII]_{a-d} were synthesized from the reaction of chalcones[V]_{a-d} with thiourea in basic medium .The structure of the compounds [VII]_{a-d} is characterized by FTIR, UV-VIS and ¹HNMR spectroscopy . The characteristic FTIR adsorption band of thiopyrimidines showed the disappearance of two absorption bands of the (CH=CH) and (C=O) groups in the chalcones and appearance of new absorption bands for(NH, C=N and C=S) groups around (3341)cm⁻¹, (1620)cm⁻¹ and (1305)cm⁻¹, respectively[19]. The FTIR spectral data and the UV-Vis data of these compounds are shown in Table 4 . ¹HNMR spectrum of thiopyrimidine [VII]_{a-d} exhibited eight aromatic protons appeared as many pairs of doublet at δ (6.5-7.9)ppm,a singlet signal at δ (6.05)ppm could be attributed to the NH(Thiopyrimidine) and a singlet at δ (6.55)ppm for proton of -CH(Thiopyrimidine) , A sharp singlet at δ (6.18)ppm due to two protons of NH₂ group.

The novel Schiff bases[VIII]_{a-d} and [IX]_{a-d} were synthesized by refluxing equemolare of D-erythroascorbic acid [VI] with amino compounds of pyrimidine[VI]_{a-d} or [VII]_{a-d} in dry benzene with some drops of glacial acetic the sixteen aromatic protons , and a singlet signal at δ (3.86)ppm that could be attributed to the proton at C4 of Lactone ring.

Biological Activity

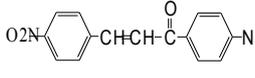
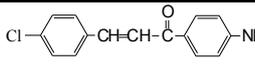
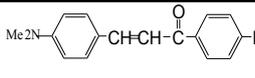
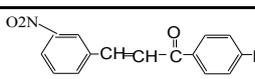
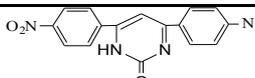
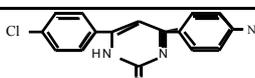
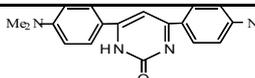
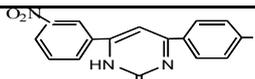
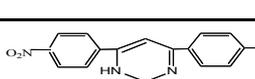
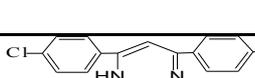
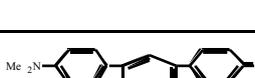
The antibacterial activity of the synthesized compounds was performed according to the agar diffusion method [20]. The prepared compounds were tested against E.coli and Staph. aureus .Each compounds was dissolved in DMSO to give concentration 1ppm. The plates were then incubated at 37 °C and examined after 24 hrs. The zones of inhibition formed were measured in millimeter and are represented by (-), (+), (+ +) and (+ + +) depending upon the diameter and clarity as in Table (6). All the compounds exhibit the highest or low biological activity while the compounds[V]_b , [VI]_b , and [VII]_b showed no activity against both the organisms, and compound [VI]_d did not show activity against only (G+) . The compounds showed good inhibition against of the two types of the bacteria, this could be related to the presence of the D-erythroascorbic acid , oxopyrimidine , thiopyrimidine and imine linkage.

References

1. Du ,c.; Liu , j.; Su, w.; Ren, y. and Wei, d. (2003) "The protective effect of ascorbic acid derivative on PC12 cells,Involvement of Its ROS scavenging ability" , Life Sci. , 74:771-780.
2. Tanuma, S.; Shiokawa, D.; Tanimoto,Y.; Ikekita, M. and Takeda , M.,(1993). "Benzylidene ascorbate induces apoptosis in L929 Tumor cell, Biochem, Biophys, Rrs, Commun., 194 :29-35.
3. Velri, R.; Fodor, G.; Liu, C. and Woolverton , C. (1986) "A new class of synthetic biological response modifiers,the methylfuryl butyrolactones" , J. Biol, Res. Mod. , 5: 444-461.
4. Woolverton,C.; Veltri, R.and Snyder , I. (1986) "Stimulation of human pmn in vitro by asuccinimide molecular complex of methylfuryl butyrolactones", J.Biol,Res.Mod., 5 :527-538
5. Nasr, M. and Gineinah, M. , (2002) "Pyrido[2,3-d]pyrimidin pyrimido[5',4':5,6]-pyrido[2,3-d] pyrimidines as new antiviral agents: Synthesis and biological activity" Arch.pharm., 335 :289-295.
6. Baraldi, P.; Pavani, M.; Nunez, M.; Brigidi, P. ; Vitali, B.; Gambari, R. and Romagnoli R. (2002)" Antimicrobial and antitumor activity of N-heteroimine-1,2,3-diathiazoles and their transformation in triazolo-, imidazo- and pyrazolopyrimidines" .Bio.org.Med.Chem. 10: 449-456.
7. Sondhi, S.; Johar, M.; Rajvanshi,S.; Dastidar, G. ; Shukla, R.; Raghbir , R. and Lown, J. (2001)" Anticancer, anti-inflammatory and analgesic activity evaluation of heterocyclic compounds synthesized by the reaction of 4-isothiocyanato-4-methylpentan-2-one with substituted o-phenylenediamines, o-diaminopyridine and (unsubstituted o-diaminopyrimidines". Austr. J. Chem. , 54: 69-74.
8. Chowdhury, A.; Matin ,M.; and Anwar, M. (1997) "Synthesis and antimicrobial activities of fused pyrimidines: Benzothieno[2,3- d]imidazol[1,2-c] pyrimidine", Chittagong Chittagong Univ. stud. Part(II) , 21 :79-83.
9. Herdewijn,P. (1994) "5-substituted-2-deoxy uridines as anti-HSV-1 Agents: synthesis and structure-activity relationship, Antiviral Chem,Chemother". 5: 131-146.
10. Malic, S.; Sverdnzic, D.; Gazivoda , T. and Marunovic, A.(2000) "Synthesis and antitumor activities of novel pyrimidine derivatives of 2,3-O,O-Dibenzyl-6-deoxy-L-Ascorbic acid",J. Med. Chem. 43: 4806-4811.

11. Ali, U .; AL-Rawi , A. and AL-Rawi , M. (1998) "Synthesis of some new Schiff bases derived from glucosamine of possible anti microbial activity" J.Drrasat,Natural and Engineering sciences,25(1):95.
12. El- Sayed , H. ; Atta , K. ; Aboul-Ela , S. and Beldi , R. (2007)" Microwave Assistant synthesis of Mono- and Bis- Phenylhydrazones of L-Threo-and-D-Erythro-2,3-Hexodiulosono-1,4-Lactones for The Synthesis of an Array of Heterocyclic Compounds", Jordan J. of Chemistry ,2(1): 117-124.
13. EL-Sekily , A. (2008) "Hydrogenation products from dehydro-D-Erythro- and L-Threo-Ascorbic acids mono- and bishydrazones".,The Arabian Jou.for science and engineering,33(1A), 7-13.
14. Salomon, L.(1963) Experientia ,19(12) :619.
15. Jwad, R. (2006) "Synthesis of new oxazepine derivatives starting from L-ascorbic acid", Thesis , Education College Ibn Al-Haitham, University of Baghdad.
16. Loudon, G. (2002) "Organic Chemistry",4th Ed.,Oxford University press,Inc.,New York, ,855-856,869 and 873.
17. Al-Ogiady, R. (2010)"Synthesis of new Malonate and Barbiturate derivatives of D-Erythroascorbic acid and their Metal complexes",Ph.D.Thesis, Education College Ibn Al-Haitham ,University of Baghdad.
18. Kalirajan , R.; Sivakumar, S.; Gowramma , S. ; Jubic, B. and Saresh , B. (2009) International J. Chem.Tech Res.,1(1): 27-34.
19. Fathalla , O . ; Awad, S. and Mohamed, M. (2005) "Synthesis of new 2-Thiouracil-5-Sulphonamide derivatives with Antybacterial and Anti Fungl activity",Arch pharm Res.,28(11):1205-1212.
20. Allawy, H. (2000) "Synthesis of some new bis-1,4-substituted butane derivatives containing 1,3,4-Oxadiazole or 1,2,4-Trizole unit", M.Sc Thesis, Education College Ibn Al-Haitham,Baghdad University.

Table(1):The physical properties compounds[V]_{a-d},[VI]_{a-d} and [VII]_{a-d}.

Comp. No.	Nomenclature	Structural Formula	Molecular Formula	M.P C ⁰	Yield %	Color
[V] _a	4[3-(4'-nitrophenyl)-2-propene-1-one] aniline		C ₁₅ H ₁₂ N ₂ O ₃	210	90	Orange
[V] _b	4[3-(4'-chlorophenyl)-2-propene-1-one] aniline		C ₁₅ H ₁₂ NOCl	164	75	Yellow
[V] _c	4[3-(4'-N,N-dimethylphenyl)-2-propene-1-one] aniline		C ₁₇ H ₁₈ N ₂ O	140	60	Red
[V] _d	4[3-(3'-nitrophenyl)-2-propene-1-one] aniline		C ₁₅ H ₁₂ N ₂ O ₃	204	80	Dark Orange
[VI] _a	4[6-(4'-nitrophenyl)-2-oxo-1,2-dihydro-pyrimidine-4-yl]aniline		C ₁₆ H ₁₂ N ₄ O ₃	214-218	70	Orange
[VI] _b	4[6-(4'-chlorophenyl)-2-oxo-1,2-dihydro-pyrimidine-4-yl]aniline		C ₁₆ H ₁₂ N ₃ OCl	208	55	Yellow
[VI] _c	4[6-(4'-N,N-dimethyl phenyl)-2-oxo-1, 2-Dihydro pyrimidine -4-yl] aniline		C ₁₈ H ₁₈ N ₄ O	212	50	Red
[VI] _d	4[6-(3'-nitrophenyl)-2-oxo-1, 2-Dihydro pyrimidine -4-yl] aniline		C ₁₆ H ₁₂ N ₄ O ₃	197	70	Orange
[VII] _a	4[6-(4'-nitrophenyl)-2-thioxo-1, 2-dihydro-pyrimidine-4-yl]aniline		C ₁₆ H ₁₂ N ₄ O ₂ S	276	60	Pale brown
[VII] _b	4[6-(4'-chlorophenyl)-2-thioxo-1, 2-Dihydro pyrimidine -4-yl] aniline		C ₁₆ H ₁₂ N ₃ SCl	200	65	Yellow
[VII] _c	4[6-(4'-N,N-dimethyl phenyl)-2-thioxo-1, 2-Dihydro pyrimidine -4-yl] aniline		C ₁₈ H ₁₈ N ₄ S	162-164	50	Yellow
[VII] _d	4[6-(3'-nitrophenyl)-2-thioxo-1, 2-Dihydro pyrimidine -4-yl] aniline		C ₁₆ H ₁₂ N ₄ O ₂ S	150	50	Brown

Table(2):The physical properties compounds [VIII]_{a-d} and [IX]_{a-d}.

Comp. No.	Structural formula	R _f	Molecular formula	M.P °C	Yield %	Color
[VIII] _a		0.31	C ₃₇ H ₂₆ O ₁₁ N ₄	185-187	50	Yellow
[VIII] _b		0.35	C ₃₇ H ₂₆ O ₉ N ₃ Cl	190	55	Brown
[VIII] _c		0.16	C ₃₉ H ₃₂ O ₉ N ₄	>300	50	Brown
[VIII] _d		0.36	C ₃₇ H ₂₆ O ₁₁ N ₄	124-126	60	Orange
[IX] _a		0.37	C ₃₇ H ₂₆ O ₁₀ N ₄ S	>300	55	Brown
[IX] _b		0.47	C ₃₇ H ₂₆ O ₈ N ₃ ClS	>300	50	Brown
[IX] _c		0.28	C ₃₉ H ₃₂ O ₈ N ₄ S	170	60	Red
[IX] _d		0.21	C ₃₇ H ₂₆ O ₁₀ N ₄ S	144	65	Yellow

Table(3) :Charcteristic FTIR absorption band and UV data (λ_{max}) of compound [V]

Comp. No.	UV data	Characteristic bands FTIR spectra (cm ⁻¹)			
	λ_{max} (nm) in CHCl ₃	ν NH ₂ asy. , sy.	ν C=O	ν CH=CH	others
[V] _a	306	3483 , 3387	1650	1635	4-NO ₂ :1504,1342
[V] _b	341.5	3385-3273	1670	1632	4-Cl:1089
[V] _c	342.5	3480-3236	1662	1630	4-N(Me) ₂ :1165
[V] _d	284.5	3426-3333	1651	1632	3-NO ₂ :1346

Table(4):Characteristic FTIR absorption bands and UV data (λ_{max}) of compounds [VI]a-d and [VII]a-d

Comp. No.	UV data	Characteristic bands FTIR spectra (cm ⁻¹)						
	λ_{max} (nm) in CHCl ₃	ν NH ₂ asy. , sy. and ν NH	ν C-H aromatic	ν C=O amide	ν C=N endocyclic	ν C=C aromatic	ν C=S	others
[VI] _a	307	3484,3387,3256	3100	1640	1610	1589		4-NO ₂ :1508,1343
[VI] _b	318.5	3460,3342,3217	3053	1645	1630	1605		4-Cl:1080
[VI] _c	339	3425,3390,3213	3036	1643	1610	1567		4-N(Me) ₂ :1168
[VI] _d	279	3472,3418,3341	3094	1645	1636	1609		3-NO ₂ :1346
[VII] _a	354.5	3460, 3333,3221	3067		1628	1597	1312	4-NO ₂ :1512,1338
[VII] _b	319.5	3456,3341,3221	3050		1620	1597	1288	4-Cl:1087
[VII] _c	408.5	3476,3433,3329	3053		1620	1597	1304	4-N(Me) ₂ :1168
[VII] _d	266	3410,3383,3360	3086		1628	1589	1308	3-NO ₂ :1346

Table(5):Characteristic FTIR absorption bands and UV data (λ_{\max}) of compounds [VII]_{a-d} and [IX]_{a-d}

Comp. No.	UVdata	Characteristic bands FTIR spectra (cm-1)										
	λ_{\max} (nm) in CHCl ₃	vNH	v C-H arom.	vC-H aliph.	VC=O Lacton	vC=O ester	vC=O amid	vC=N exocyc c	vC=N endocyc c.	vC=C aro m.	vC=S	vC-O
[VIII] _a	258	3390	3052	2982-2847	1760	1738	1653	1637	1615	1602		1263
[VIII] _b	234	3417	3059	2962-2839	1767	1735	1659	1630	1605	1590		1261
[VIII] _c	258.5	3402	3060	2978-2839	1766	1715	1650	1640	1605	1585		1257
[VIII] _d	268.9	3337	3059	2984-2878	1770	1724	1642	1632	1605	1580		1237
[IX] _a	252.6	3348	3078	2962-2843	1766	1720	1645	1630	1610	1601	1310	1257
[IX] _b	261	3406	3050	2981-2843	1766	1730	1650	1627	1605	1585	1300	1261
[IX] _c	258.5	3383	3078	2908-2949	1766	1715	1642	1630	1600	1580	1305	1258
[IX] _d	252.4	3368	3059	2982-2843	1769	1720	1645	1627	1605	1580	1304	1263

Table(6): Antibacterial activity of the prepared compounds

Comp. No.	E.Coli (G-)	Staph.aurus (G+)	Comp. No.	E.Coli (G-)	Staph.aureus (G+)
[V] _a	++	+++	[VIII] _a	++	+++
[V] _b	-	-	[VIII] _b	+++	+++
[V] _c	++	++	[VIII] _c	+++	+++
[V] _d	+++	+++	[VIII] _d	+++	+++
[VI] _a	++	++	[IX] _a	+	++
[VI] _b	-	-	[IX] _b	+++	+++
[VI] _c	+++	+++	[IX] _c	+++	+++
[VI] _d	+	-	[IX] _d	+++	+++
[VII] _a	+++	++	[IV]	++	++
[VII] _b	-	-			
[VII] _c	+	++			
[VII] _d	+++	+++			

Key to symbols: Highly active = +++(mor than)15 mm.

Moderately active = ++(11-15) mm. and Slightly active = + (5-10) .

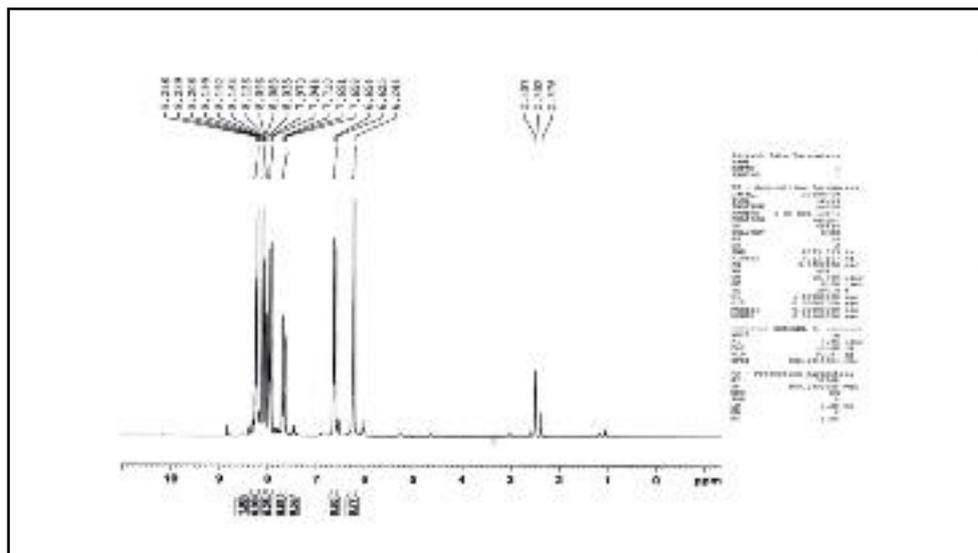


Fig. (1): ¹HNMR- spectrum of compound[V]_a

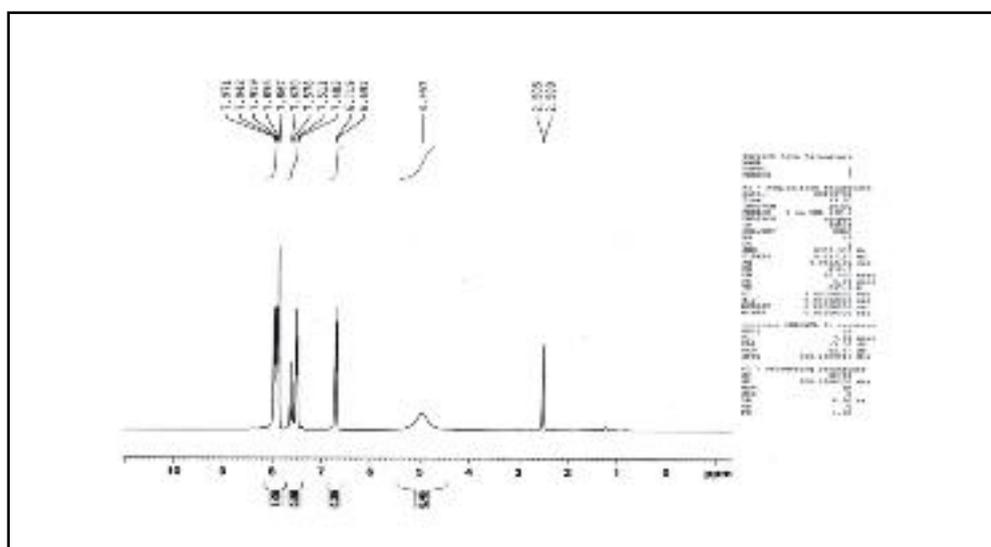


Fig . (2): ¹HNMR-spectrum of compound[VI]_b

تحضير و تشخيص مشتقات جديدة من قواعد شف لحامض D-ارثرو اسكوربيك و البيرميدين

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الخلاصة

حضرت المركبات الجديدة لقواعد شف المشتقة من حامض D-ارثرو اسكوربيك مع مركبات البيريميدين التي حضرت من تكاثف حامض D-ارثرواسكوربيك مع الامينات الاروماتية (المحتوية على وحدة البيريميدين) في البنزين الجاف وباستعمال قطرات من حامض الخليك الثلجي محفزا. حضر الحامض D - ارثرواسكوربيك بابع خطوات متتالية كما في المخطط رقم (1) بينما حضر الامين الاروماتي من تفاعل الجالكونات مع اليوريا او الثايوريا في وسط حامضي او قاعدي وعلى التوالي .

شخصت جميع المركبات المحضرة بقياس درجات انصهارها وبوساطة طيف FTIR , UV-Vis وطيف ¹HNMR. درست الفعالية البيولوجية للمركبات المحضرة ضد نوعين من البكتريا واطهرت النتائج فعالية بيولوجية جيدة ضد البكتريا بنوعيه Staphylococcus (G+), Echerichia coli (G-). بينما لم تظهر المركبات [V]_b , [VI]_b , [VII]_b اي فعالية بيولوجية ضد هذا النوع من البكتريا.

الكلمات المفتاحية: قواعد شف , حامض الاسكوربيك , بيرميدين .