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Bio–chemical study of synthesized various compounds of anil– arabinose compound

■ NAGHAM MAHMOOD AL–JAMALI

Author for Correspondence -
NAGHAM MAHMOOD AL-JAMALI
Department of Chemistry, College of Education for Women, Kufa University,Kufa, IRAQ
Email : dr.nagham_mj@yahoo.com

ABSTRACT - In this paper, series of various organic compounds [1-11] were synthesized from anil –arabinose compound, which contain two imine –groups can be react as starting material with other compounds (sodium azide, chloro acetyl chloride, azo compound, thiol, secondary amine, maleic anhydride, primary amine) to produce cyclic and open cyclic compounds from (azitidine, form azane, diazepine, thiazine, diazane, sulfide). A detailed discussion of the structural elucidation of newly synthesized compounds [1-11] was confirmed by (melting points, elemental analysis C.H.N, FT.IR, H.NMR)–spectra, and antimicrobial study on the Gram –positive and Gram –negative bacteria.

Key words - Azetidine, Formazan, Diazepine, Sugar-imine

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Carbohydrate are amajor class of naturally occurring organic compounds,which involves only Two functional groups: ketone or aldehyde carbonyls and alcohol hydroxyl groups.During the Past few years carbohydrates have received increasing attention as stereo differentiating auxiliaries in stereo selective synthesis[1,2].

The presence of acarbohydratemoity side chain in any synthesized compound may overcome the Frequently observed water insolubility problem.

On the other hand,the incorporation of imine- mono saccahrides compound with other Compounds such as sodium azide or chloro acetyl chloride…etc., to produce fused rings and open rings compounds which was known to possess various pharmacological activities like antibacterial, analegesic, antiinflammatory, anticonvulsant, antimicrobial activities[3,4].

The hetero cyclic compounds bearing sugars in their structure have many applications in Biological science,and most of imine compounds bearing mono or bi cycles have chemical[5]and biological importance[6-10].

EXPERIMENTAL METHODOLOGY

All chemicals used (purity 99.98 %), FT.IR -spectra :were recorded on shimadzu 8300, KBr -disc, H.NMR-spectra were recorded on varian 300 MHz spectrometer using TMs as an internal standard and elemental analysis (C.H.N)–elemental (analyses system GmbH) –Germany Vario EL.III, in environmental science in Jordan. the melting points were determined in open capillary tubes by electro thermal 9300 LTD, U.K., microbial study in lab of Bio-Department in Education College.

Synthesis of compound [1]:

A mixture of (0.1 mole, 6.85 g) of hydrazine with (0.2 mole, 30 gm) of arabinose sugar reacted under refluxing for (4 hrs) in presence of glacial acetic acid (drops) and absolute ethanol as solvent with stirrer by used mechanical stirrer the precipitate filtered and dried, recrystallized from absolute ethanol to give 84 per cent from imine –arabinose named compound [1].

Synthesis of compounds [2-6] :

A mixture of compound [1] (0.01 mole, 2.96 g) with (0.02

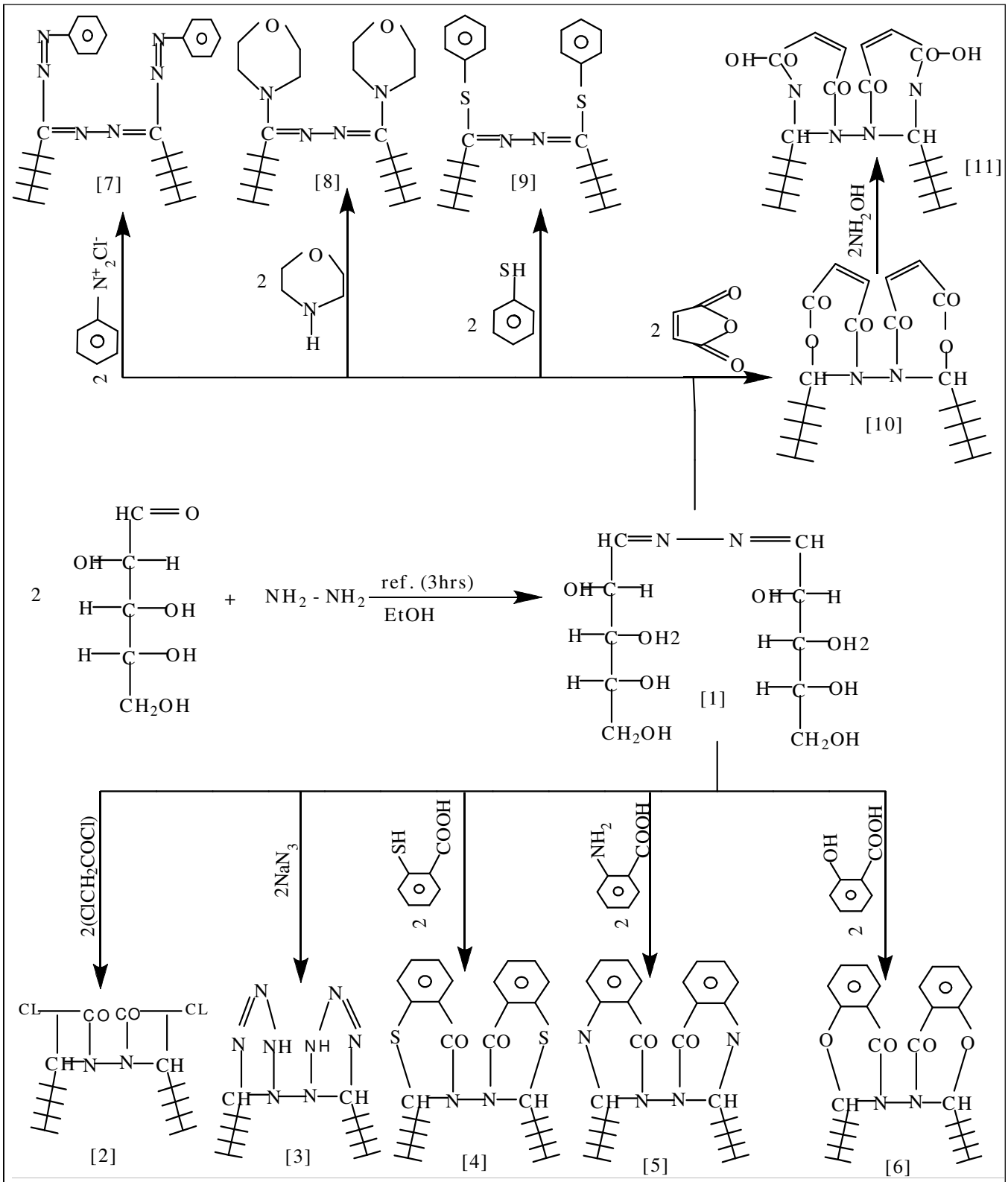

Fig. A : Synthesis of compounds

mole) from one of {(2.26 g of chloro acetyl chloride), (1.3 g of sodium azide), (2.4 g of thiol benzoic acid), (2 g of o-amine benzoic acid), (2 g of salicylic acid)}, respectively reacted in present of dioxan and stirrer for (5 hrs) then the precipitate filtered and dried, recrystallized to produce {compound[2] 88 %, compound[3] 85 %, compound[4] 88 %, compound[5] 84 %, compound[6] 83%}, respectively.

Synthesis of compounds [7-9] :

A mixture of compound [1] (0.01 mole, 2.96 g) in pyridine with one of (0.02 mole) of {(2.8 g of benzene diazonium),(1.7 g of morpholine), (2.2 g of benzene thiol)} in ice bath at (0-5)c for (6 hrs), the precipitate was filtered and washed till it was free from excess pyridine and recrystallized from ethanol to yield (86,87,89) per cent, respectively of formazane compound and other from compounds [7-9]

Synthesis of compounds [10,11] :

A mixture of compound [1] (0.01 mole, 2.9 gm) with (0.02 mole, 109 g) of maleic anhydride) were refluxed for (7 hrs) in presence of benzene, the precipitate filtered and dried which (0.01 mole, 4.9 g) refluxed with (0.02 mole, 1.3 g) of amine hydroxyl in presence of benzene for (6 hrs) the precipitate filtered and dried crystallized from benzene to yield 82 per cent from compound [11].

EXPERIMENTAL FINDINGS AND ANALYSIS

Pentose sugar–anil compound [1] is used as starting material in synthesis of cyclic compounds [2-6, 10-11] and open ring [7-9], in this work, arabinose sugar reacted with hydrazine compound to produce anil compound [1], which reacts with other compounds to yield (azetidine, tetrazole, oxazane, thiazine, oxazepine, diazepine, sulfide, formazane, diazane) named compounds [1-11].

Formazane is one of synthesized compound in this work named compound [7] which contains azo group with imine group at same molecule.

All synthesized compounds [1-11] have been characterized by their melting points and spectroscopic

Table 1 : (FT.IR) –data (cm^{-1}) of compounds [1-11]

Comp. No.	$I.R_{(KBr)}$ (only important groups)
1.	(CH=N) imine group: 1618 ; (OH) hydroxyl groups of arabinose sugar : 3317
2.	(CO–N) carbonyl of amide : 1688 ; (C–Cl) 728 , (OH) hydroxyl groups of arabinose sugar : 3312 .
3.	(NH): 3310 ; (N=N) endocycle : 1430 ; (C–N) endocycle : 1240 ; (OH) hydroxyl groups of arabinose sugar : 3390 .
4.	(CH–S): 1410 ; (CO–N) carbonyl of amide : 1695 ; (C–S) : 670 ; (OH) hydroxyl groups of arabinose sugar : 3395 .
5.	(NH) : 3305 ; (CO –N) carbonyl of amide : 1690 ; (OH) hydroxyl of sugar : 3396.
6.	(C–O–C): 1155 ;(CO–N): 1686 ; (OH) hydroxyl groups of arabinose sugar: 3428.
7.	(C=N) : 1610 ; (-N=N) azo : 1437 ; (OH) of sugar : 3330 .
8.	(C=N) : 1615 ; (OH) hydroxyl of sugar : 3395 .
9.	(C=N) : 1618 ; (C –S) : 670 ; (OH) hydroxyl of sugar : 3385 .
10.	(CO–O) of oxazepine : 1730 ; (CO–N) : 1696 ; (OH) of sugar : 3410 .
11	(CO–N) : 1696 , (OH) of sugar : 3317 .

Table 2 : H.NMR –data (δ_{PPm}) of compounds [1-11]

Comp. No.	H.NMR (only important peaks)
1.	8.86 (CH=N) proton of imine group ; (4.40 , 4.43 , 4.45 , 4.48) protons of (CH–OH) hydroxyl of arabinose sugar .
2.	3.4 (CH –N) ; 2.98 (CH –Cl) of azitidine ; (4.40 , 4.43 , 4.45 , 4.48) hydroxyl of arabinose sugar .
3.	3.9 (-N–NH–N) ; 3.4 (N–CH–N) ; (4.77 , 4.89 , 4.97 , 5.12) hydroxyl of arabinose sugar .
4.	4.48 (S–CH–N) ; (4.81 , 4.93 , 5.04 , 5.16) of (CH–OH) hydroxyl of arabinose sugar ; (6.72 – 7.30) protons of phenyl rings .
5.	3.6 (NH–CH–N) ; (4.76 , 4.84 , 4.98 , 5.12) of hydroxyl of arabinose ; (6.64–7.20) protons of phenyl rings .
6.	4.05 (O–CH–N) ; (4.40 , 4.43 , 4.45 , 4.46) protons of hydroxyl of arabinose ; (7.18 – 7.36) protons of phenyl rings .
7.	(4.79 , 4.88 , 5.00 , 5.13) protons of hydroxyl of arabinose ; (6.95 , 7.35) protons of phenyl rings .
8.	(3.81 , 4.10) protons of ($O-CH_2-CH_2-N$) ; (4.74 , 4.86 , 4.99 , 5.14) hydroxyl of arabinose sugar .
9.	(6.92 , 7.15) protons of phenyl rings , (4.65 , 4.79 , 4.88 , 4.97) protons of hydroxyl of arabinose
10.	9.23 (O–CH–N) proton of oxazepinering ; (2.33 , 2.51) proton of (CH=CH) of oxazepine ring ; (4.76 , 4.85 , 4.98 , 5.12) protons of hydroxyl of arabinose sugar.
11.	3.41 (N–CH–N) ; 4.18 (N–OH) ; (2.49 , 3.34) proton of (CH=CH) of oxazepine ring ; (4.53 , 4.55 , 4.67 , 4.81) protons of hydroxyl of arabinose sugar.

Table 3 : Physical properties and (C.H.N)–analysis of compounds [1-11]

Comp. No.	M.F	m.p (C?)$_{(+2)}$	Name of compound	Calc. / Found. C%	H%	N%
1.	$C_{10}H_{20}N_2O_8$	152	Bis (1–arabinose imine)	40.540	6.756	9.459
				40.431	6.613	9.324
2.	$C_{14}H_{22}N_2O_{14}Cl_2$	178	Bis(4–arabinose-3–chloro–azitidine-2–one)	37.416	4.899	6.236
				37.271	4.646	6.098
3.	$C_{10}H_{22}N_8O_8$	190	Bis (5–arabinose–tetrazole)	31.413	5.759	29.319
				31.286	5.516	29.20
4.	$C_{24}H_{28}N_2O_{10}S_2$	212	Bis(2–arabinose–5,6–benzo-4–one– 1,3 thiazane)	50.704	4.929	4.929
				50.551	4.801	4.783
5.	$C_{24}H_{30}N_4O_{10}$	186	Bis (2–arabinose–5,6–benzo-4–one–1,3 diazane)	53.932	5.617	10.486
				53.684	5.548	10.319
6.	$C_{24}H_{28}N_2O_{12}$	197	Bis(2–arabinose–5,6–benzo-4–one– 1,3 oxazane)	53.731	5.223	5.223
				53.573	5.084	5.104
7.	$C_{22}H_{28}N_6O_8$	182	Bis(1–arabinose-1–phenyl azo–imine)	52.380	5.555	16.66
				52.209	5.348	16.52
8.	$C_{18}H_{34}N_4O_{10}$	196	Bis(1–arabinose-1–morpholine- imine)	46.351	7.296	12.017
				46.208	7.148	12.019
9.	$C_{22}H_{28}N_2O_8S_2$	200	Bis(1–arabinose-1–phenyl Sulfide-imine)	51.562	5.468	5.468
				51.387	5.279	5.318
10.	$C_{18}H_{24}N_2O_{14}$	229	Bis(2–arabinose-4,7–dione–1,3–oxazepine)	43.902	4.878	5.691
				43.781	4.693	5.503
11.	$C_{18}H_{26}N_4O_{14}$	216	Bis(2–arabinose-1–hydroxy-4,7–di one–1,3–diazepine)	41.379	4.980	10.727
				41.198	4.814	10.603

methods (FT.IR, H.NMR, C.H.N) –analysis and biological study.

Their FT.IR –spectrum, showed an absorption band at (1618)cm^{-1} due to (CH=N) imine group[13,14] in compound [1], which disappeared and other bands appeared such as ((1688 of CO-N amide)[5,13], (728 of C-Cl of azetidine cycle)) in compound [2], bands at ((3310 of NH), (1430 of N=N end o cycle of tetrazole)) in compound [3], bands at ((1410 of CH-S)[5], (1695 of CO-N)) in compound [4], bands at ((3305 of NH)[3], (1690 of CO-N)) in compound [5], bands at ((1610-1618 of (C=N) imine[15] group))in compounds [7-9] and (1437 of N=N azo group) in compound [7] of formazane compound, bands at (1730 of CO-O of oxazepine)[11-14], (1696 of CO-N amide of diazepine) in compounds [10,11], respectively and other data of functional groups shown in Table 1 and Fig. 1-4.

Their H.NMR –spectrum showed signal at σ (8.86)due to (CH=N)proton of imine group[13-16] in compound [1], which disappeared and other signals appeared at ((3.4 of CH-N), (2.98 of CH-Cl)) of azetidine in compound [2], signals at á ((3.4 - 4.05)) due to ((N-NH-N),(N-CH-N),(S-CH-N)[5], (O-CH-N), (O-CH_2CH_2-N) in compounds [3-11], respectively, all compounds appeared signals at σ (4.40 – 5.16)due to hydroxyl groups of arabinose sugar, and other signals[13-17] shown in Table 2 and Fig. 5-8.

Their (C.H.N)- analysis and melting points, it was found from compared the calculated data with experimentally data of these compounds, the results compactable. the data of analysis, M.F and melting points are listed in Table 3.

Table 4 : Antibacterial activity of the compounds[1-11] {diameter of zone (mm)}

Compounds[1-11]	Diameter of zone(mm)	
	G+: Staphylococcus. aureus	*G-: E-Coli*
1. Compounds	11	7
2. Compounds	27	22
3. Compounds	28	24
4. Compounds	30	27
5. Compounds	19	14
6. Compounds	20	16
7. Compounds	23	20
8. Compounds	13	17
9. Compounds	17	10
10. Compounds	16	31
11. Compounds	34	
Ampicilline**		

*Minimum Inhibitory concentration (MIC)of compounds[1] (7mg/ml).
**Ampiciline (0.1mg/ml)

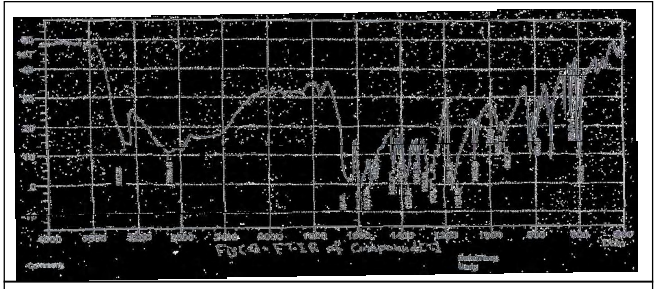

Fig. 1 : FT.I.R. of compound (1)

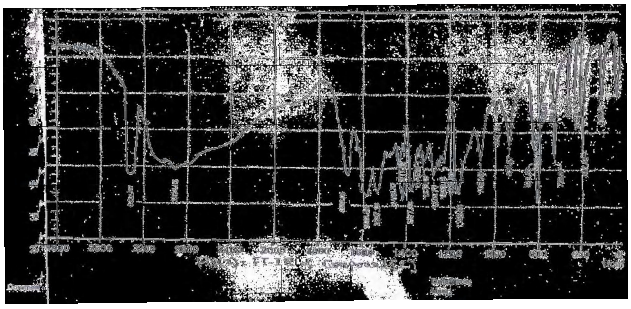

Fig. 2 : FT.I.R. of compound (2)

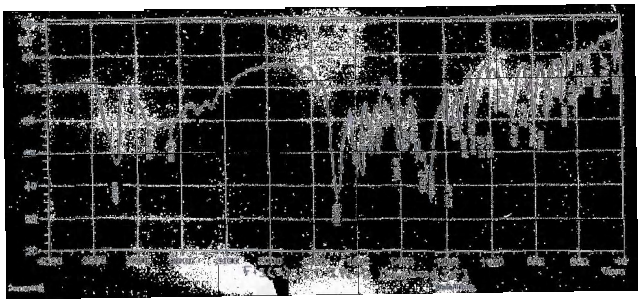

Fig. 3 : FT.I.R. of compound (6)

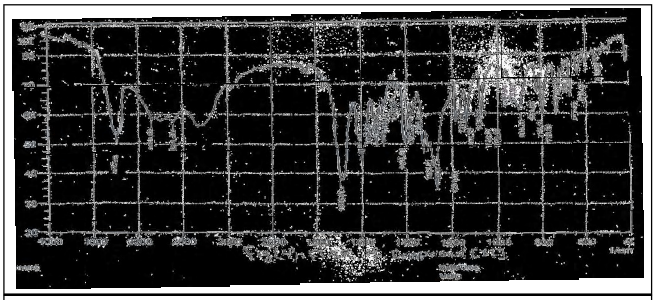

Fig. 4 : FT.I.R. of compound (11)

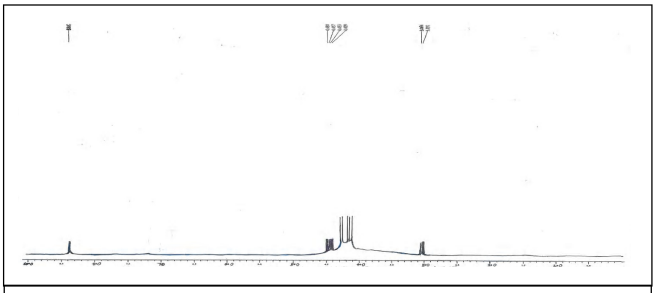

Fig. 5 : HNMR of compound (1)

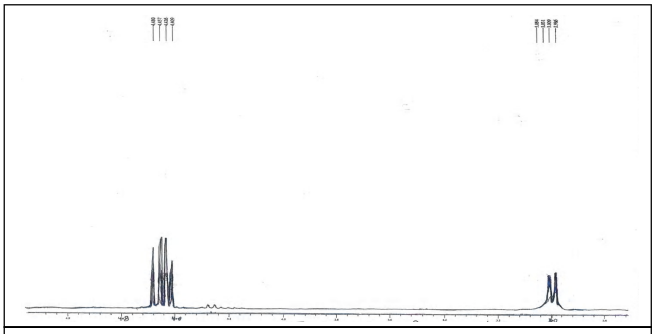

Fig. 6 : HNMR of compound (2)

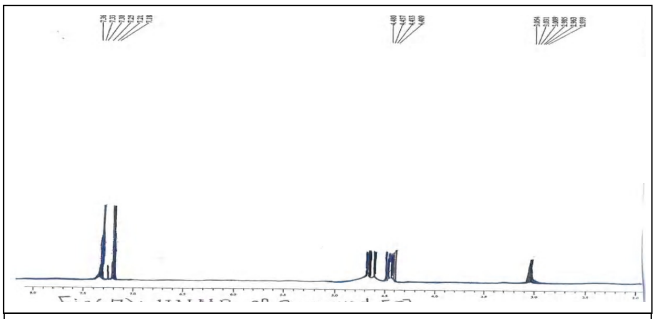

Fig. 7 : HNMR of compound (6)

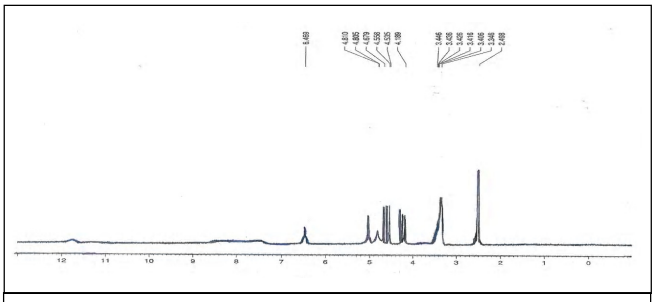

Fig. 8 : HNMR of compound (11)

Assay of antimicrobial activity [18]:

Antimicrobial activity was tested by the filter paper disc diffusion method against gram positivebacteria *(Staphylococcus. aureus)* and gram negative bacteria (*E-Coli*), 0.1 ml of the bacterial suspensions was seeded on agar. To determine minimum inhibitory concentration(MIC) for each compounds[1-11] were ranged between (1-15)mg/ml by dissolved in (DMSO) and preparation 0.1mg/ml standard

antibiotic ampiciline as positive standard and reference.

The positive results or sensitivity were established by the presence of clear zone of inhibition around active compounds which were measured with a meter rule and diameters were recorded based on (mm), the assays were performed with two replicates.

Generally, The results showed that the compounds[1-11] have great inhibitory effect against tested bacteria as compared with Synthetic antibiotic Ampiciline.

Table 4 showed the zone of inhibition of the compounds[1-11] in this study ranged (from 30 to 7) mm. From results, we noted that the compounds[2-4] have higher antibacterial activity against *S.aureus and E-Coli*is due to the presence of sulfur and nitrogenatomes(O, N, S)withlactame group in some structures. Consequently,these compounds become more effective in precipitating proteins on bacteria cell walls.These atoms form hydrogen bonds with cell wall protein and hence, destroying the cell membranes,these compounds had abroad antibacterial activity.

A knowledegement :

alsI would like to express my thanks to Mr. Uodai in Jordan for providing (C.H.N) element analytical, and H.NMR –spectra and melting points And express my thanks to(United Arabic Company) and (Zaidan Company of Chemical) for supplied some material.

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