Zagazig J.Agric. Res., Vol .30 No.(2) 2003 *519-537*

ISOLATION AND IDENTIFICATION OF SOME BIOCHEMICAL CONSTITUENTS FROM *JUNIPERUS PHOENICEA*

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Received 27 / 2 / 2003 *Accepted 1 / 3 / 2003*

ABSTRACT: *Juniperus phoenicea L* The wild plant from North Sinai have used as antispasmotic, antidiabtic, and for treatment of hypertension and chronic cough and hemorrhoids. Four flavonoid identified as Jaceidine(3,6,3′-trimethoxy-5,7,4′trihydroxy flavone); quercetin (3,5,7,3′,4′pentahydroxy flavone);quercetrin (quercetin-3-o-α-L rhamnoside)and Kaempeferol -3-o-α-L rhamnoside (5,7,3′trihydroxy flavone-3-o-∝-L rhamnoside) were isolated from *J.phoenicea* ethanol 70% along side with two new lignans,5-hydroxy bursehernin(3,4-dimethoxy-5-hydroxy-3′,4′methelendioxy-dibenzylbutryo lactone) and 7′-hydroxy-3,4,3′,4′tetramethoxy dibenzylbutryolactone from *Juniperus phoenicea*. acetone extracts .The structures of isoltats were achiaved by UV,IR,H^1-NMR, MS spectra.

INTRODUCTION

Juniperus phoenicea (L) Fam. *Cupressaceae* is a wild plant very rarely seen in North Sinai at the mountains of Halal, El-Maghara and Yelleq, mostly on high altitudes. Tackholm (1974). *J.phoenicea L* leaves and berries were used for the treatment of chronic cough and hemorrhoids and as an antispasmodic (Al-Antaki 1923 and Issa-Bey 1930). Recently, it is being used by the natives as an antidiabtic Amer *et al* (1994), Sanchez *et al* (1994) and Gabr (1998). Namboodripad *et al* (1968) stated that *Juniperus species* have been used for treatment of hypyrtension. Cairnes *et al* (1980) isolated desoxy-podophyllotoxin and beta peltantin-A methyl ethar which were identified as the major cytotoxic components in twigs and leaves. Beta peltantin A-methyl ether showed a LD_{50} of 0.026

mg/ml in the KB cancer cell culture system.

El- Saadany *et al* (1994) proved the role of *J.phoenicea* berries and leaves as a remedy of obesity. Ono *et al* (1990) assessed the biological activity of quercetin, myriceyin, boicalein and quercetagnin in a cell based assay (Human immuno-deficiency virus (HIV-I) demonstrated that all compounds were cytotxic at concentrations which did not significantly inhibit (HIV-1) reverse transcriptase. Pakanaev *et al* (1980) studied the role of flavonoids; kaempferol, querctin and querctrin as an anti-inflammators and illustrated the biological activity of these substances depended on the number of hydroxy groups in the side phenyl redical of the flavonoids and on the nature of the sugar component. Tsukamato *et al* (1984a) showed that lignan which has been used since old times as an antipyretic, an antirhematic, a tonic and a remedy for tubercles like disease of the lymph glands generally localized in the neck. Pfeifer *et al* (1992) studied the anti-HIV activity of lignans isolated from *Ipomoea cairica*. (-). Arctmgenin and (-)-trachelogenin as tested towards (HIV-1) in vitro the tested lignans showed antiviral activity causing the inhibition of HIV proviral DNA integration cellular DNA.

In the present study the aim of the work is to isolate flavonoids and lignans from mixture of leaves and berries of the plant and identified by UV, IR, MS and H^1 – NMR Spectral data.

MATERIALS AND METHODS

Leaves and fruits samples of *Juniperus phoenicea* were obtained from North Sinai and classified by the plant Taxonomy Department, Faculty of Science, Cairo University. H1- NMR (200MHz) in CDCL3; chemical shifts are given in δ ppm.MS were meadured using a HP model / MS – 5000 spectrometer, chemical ionization method (CL). IR obtained in CHCl3 in KBr discs, U.V spectra were determined in MeOH for all compound and also, in MeONa, AlCl3 AlCl3 / HCl, NaOAC and NaOAC / boric acid for flavonoid only.

Flavonoids'isolations:

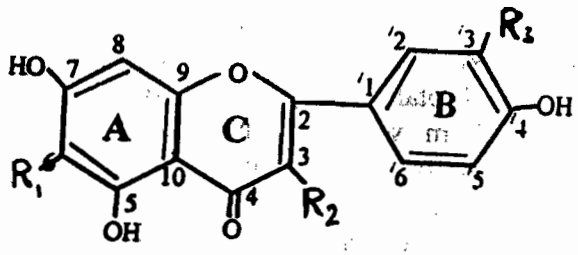
The finely powdered materials 1.166 kg. was soaked in ethanol 70% for one week. The alcoholic extract was filtrated and concentrated under vacuum at 60 °C to 500 ml and resinous and fatty materials were removed by decantation from refrigerated

extract. The ethanol extract was evaporated to dryness. A total of dry ethanol extract 35 gm was dissolved in small amount of ethanol and mixed with 20 gm of silica gel before drying on water bath. Then separated on column (120 x 5) Cm packed with 500gm of silica gel used gradients of methanol and chloroform. One and two dimensional paper chromatography were used for R_f values and spots appearance on UV light with and without ammonia the isolated flavonoids were purified by paper chormatography Whatman 3MM using n-Butanol: Acetic acid: Water (4:1:5)

Acid hydrolysis of glycosides was obtaind according to the method of Mabry *et al.* (1970) by treated five milligrams of the pure glycoside was treated with 3 ml. of hydrochloric acid 2N and few drops of methanol then mixture was heated in a test tube on boiling water bath for two hours, After cooling, the aglycone was extracted by shaking with ethylacetate and the extract was dried over anhydrous sodium sulfate and finally the solvent was evaporated under vacuum at 60-70 °C.The dry residue was subjected to one-dimensional paper chromatography in comparison with authentic samples of aglycones.Sugars were detected after neutralizing the mother liquor with $NaHCO_3$ and the solvent was evaporated under vacuum. The residue was dissolved in 10% isopropyl alcohol and spotted on paper chromatography (Whatman No.1) with authentic sugars using (benzene:n-butanol:pyridine:water) (1:5:3:3) system for the run.

Lignans'isolatiol:

Two kilograms of the fine powdered leaves and berries of *Juniperus phoenicea* were soaked in acetone for one week. The biomass was filtered from the extract and the solvent was evaporated under vacuum to yield about 30 gm of green-brown residue. The residue was diluted with 500 ml distilled water and re-extracted with ethylacatate three times using 500 ml each. The combined ethylacetate extracts were filtrated and concentrated under vacuum to give 20 gm of green resinous residue (Fang *et al.*, 1992). The residue was chromatographed on column (60 x 5 cm.) packed with 200 gm. of silica gel (320-400 mech, Merk No. 9583 on the column and developed with hexane, ethylacetate, acetone mixturs TLC was performed on silica gel $60GF_{254}$, All fraction from the column were tested by spotting on



1) $R_1, R_2, R_3 = -OCH_3$
   1a) R_1=H, R_2, R_3=OH
   2) R_1=H, R_2= α-L-rhamnose, R_3=OH
   2a) R_1 =H, R_2 ,R_3=OH
   3) R_1 =H , R_2= α-L-rhamnose, R_3=H
   3a) R_1 =H , R_2 =OH, R_3 =H

silica gel plates and developed with Ethyl acetate: Hexane (3:1), Chloroform: Methanol (9:1) and Acetone : Hexane (1:3). Preparative TLC (20 ×20 cm.) prepared according to the method of Kirchener (1967) were used for purification of isolated lignans

RESULTS AND DISCUSSION

Jaceidine (1): 3, 6, 3′ trimethyl – 5, 7 ,4′ trihydroxy flavone, appearese as brown spot on paper under UV light and greenish – brown one with NH3 . this compound showed Rf 0.68, 0.19 and 0.56 as R_f valuese.

The λ max of methanol spectrum (Table1) gave 351nm for band I and 269nm for band II. This may reveal the presence of flavonol skeleton with substitution at 3-position (methylation or glycosylation) since the range 328-357 in band I was reported by Mabry et al (1970) A bathochromic shift was noted in band II 269nm with methanol observing the presence of 5-hydroxyl group in A ring while band I show a notable hypsochromic shift confirming that the hydroxyl groups at 3.4′ or 5 positions in flavonol nuclear is methylated or glycosylated. The presence of one peak with methanol 269nm in band II confirmed that the compound contains 4′ –free hydroxyl group as previously concluded by Aytta (1983).The addition of sodium methoxide in methanol produced a larg bathochromic shift at band I (59nm) without a decrease in intensity the absence of free

hydroxyl group at 3 position Mabry et al (1970). The presence of 320-330nm peak with sodium methoxide (λ max 330nm) indicate to that the position-7 is free from glycosyl structure and contains a free hydrexyl group similar to what was previously observed for aglycones (Harborne.1970). The Aluminum chloride $AlCl_3$ is chelated by the functional groups such as the 5-hydroxy-4 keto and ortho-dihydroxyl systems, giving rise to bathochromic shift of one or both bands. The $AlCl_3$ UV spectrum of compound gave 36nm bathochromic shift for band I, while in presence of a small amount of aqueous hydrochloric acid (HCl) the spectrum showed a 17 nm hypsochromic shift in the same band. That revealed the absence of O-dihydrexyl groups in B-ring and the presence of free 5-hydroxyl group and oxygenated 6-position as evidenced by Mabry et al (1970); Mears and Mabry(1975)The sodium acetate (NaOAc) spectrum showed a bathochrochromic shift for band I and II 43,5nm cofirming the presence of both free 4',7 hydroxy groups. Also, the spectrum of sodium acetate/boric acid mixture gave no bathochromic shift in band I confirming the absence of O-dihydroxyl groups in B-ring in accordance with Harborne ,(1975) .The mass spectrum (Table2) gave a molecular ion at m/z=389,362 and 361 of parent mass at 360 consistent with hexaoxygenated flavonoid with three methoxyl-groups. Also,a comparable peak at 345 indicates the expected loss of-CH3 group from the 6-position and the peak at 331 and 285 refers to the expected loss of 2(-CH3) groups from 3', 3positions.(Mabry and Markham ,1975).

Quercetagetine (1a): 3, 5, 6, 7, 3', 4' hexahydroxy-flavone resulted from the demethylation of compound 1 ; it appearese as dull black spot under UV light and became dark brown with NH_3 showing 0.31, 0.42 and 0.12 as R_f values similar with these of Quercetagetine authentic sample when compared on paper chromatography.

Quercetrin (2): 5, 7, 3' ,4' tetrahydroxy falvone 3-o-α-L rhamnoside, isolated with 50% methanol in chloroform and appearse as purple spot under UV and yellowish – green with NH_3 recording 0.72, 0.60 and 0.58 as R_f values It can be suggested that, 2 has either flavone or flavonol skeleton because of the presence of 350nm. absorption peaks for band I in methanol spectrum (Table1) but ,the presence of hypsochromic

Table (1): U.V. spectral data of isolated flavonoid.

Comp	band	(nm)					
		MeOH	MeONa	$AlCl_3$	$AlCl_3$/HCl	NaOAc	NaOAc/Boric
1	I	351	410hr	301sh,387	370,406sh	394	355
	II	269	280,330sh	267,281sh	264,280sh	274,323	257,270
2a	I	300sh,371	326,417deg	456hr	360,424	392deg	388
	II	255,268sh	249sh	271,303sh	266	273,326sh	261,302sh
2	I	301sh,350	399hr	332sh,429	354,398	374	367.5
	II	256,265sh	270,325sh	274,303sh	271	272,303sh	260
3	I	410hr	349hr	348,394sh	344,393	373	345
	II	265,282sh	272,322sh	273,304sh	274,301sh	265,284sh	265,284sh

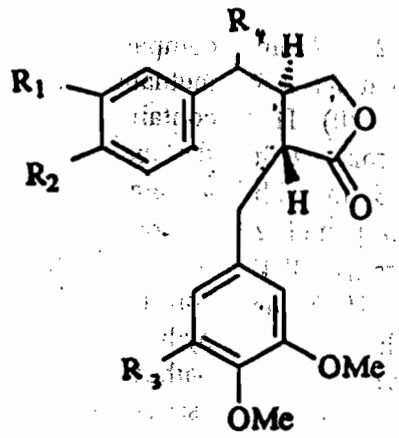
sh = shoulder

hr = hyperchromic shift

deg = degeneration in few minutes

shift in band I of 2 21nm confirmed the substition at 3,4′or 5 positions Mabry *et al* (1970). The presence of one peak with methanol in band II at 256nm with shoulder at 265nm compound 2 suggests that 2 contains 3′,4′ dihydoxy flavones Harborne (1967). In sodium methoxide UV spectrum, the occurrence of bathochromic shift in band I (49nm) of compound 2 may indicate the presence of free 4′-hydroxy group. This bathochromic shift occurred without a decrease in the intensity or degeneration of the absorption peaks, confirming that the compound 2 contains substituted 3-hydroxy flavone as being reported by (Mabry et al. 1970; Dechene, 1951 and Jurd and Horowitz, 1957).The Aluminium chlorid ($AlCl_3$) and Aluminium chlorid with hydrochloric acid ($AlCl_3$/HCl) spectrums confirming the presence of ortho-3′,4′ dihydroxy in B-ring with both compounds 2 because of the occurrence of bathochromic shift in band I with $AlCl_3$ compared with methanol spectrum (79nm for 2) and the hypsochromic shift in the same band with $AlCl_3$ / HCl spectrum compared with $AlCl_3$ spctrum (31nm for 2) as explained by Mabry *et al* (1970).On the other hand the bathochromic shift recorded band Ia with $AlCl_3$/HCl compared with band I with methanol 48nm reveals that 2 contains a 5-hydroxy, 3-substituted flavonol structure and.Sodium acetate spectrum of 2 revealed a bathochromic shift (15.5nm) in band II compared with methanol confirming the presence of free-7-hydroxyl group, while the bathochromic shift (25nm) associated with band I confirm the presence of free/4-hydroxyl group. The addition of boric to sodium acetate gave a bathochromic shift in both compounds 3 and 2 (17 and 7.5nm) compared with Na OAc spectrum in band I, confirming the presence of 3′,4′ -ortho dihydroxyl group in B ring according to Harborne,(1975). Mass spectrum data (Table2) of 2 showed a molecular ions at m/z 477 and 449 of parent mass at 448 for 2 consistent with pentaoxygenated mono-glycosylated falvonoid. Comparable peaks were detected at 302,303 and 331 may refer to loss of –Rhammosyl moiety from 3-glycosyl flavonoid ions (Mabry and Markham,1975)

Quercetin: (2a) 3, 5, 7, 3′ ,4′ benta hydroxy flavone isolated with 10% methanol in chloroform also, resulted from the complet acid hydrolysis of 2. It gave a yellow colore under UV and yellowish-green with NH_3 ,



4) $R_1, R_2 = OCH_3, R_3 = H$
$R_4 = OH$

5) $R_1 + R_2 = O\text{-}CH_2\text{-}O, R_3 = OH$
$R_4 = H$

recording 0.64, 0.03, 0.29 as R_f values. It can be suggested that 2a has either flavone or flavonol skeleton because of the presence of 371nm. Absorption peaks for band I in methanol spectrum (Table1), it has no hypsochromic shift in this band compared to the standard value of flavonol or flavone structure (258-380) as defined by Mabry *et al* (1970). So, it can be concluded that, there is no methylation or glycosylation at 3,4′ or 5 positions as previously defined by Mabry *et al* (1970).

Also 2a contains 3′,4′ dihydroxyl structure because of the presence of one peak at 255 in band II with shoulder at 265nm according to Harborne, (1967). While, the absorption peak in band I at 417nm of 3 was degenerated in few minutes, suggests that 3 has a 3, 4′ free dihydroxyl group as being reported by (Mabry et al.1970;Dechene, 1951 and Jurd and Horowitz,1957).The same spectrum of 3 displayed an absorption peak at 326nm (band Ib). confirming the presence of free-7-hydroxyl group according to Harborne (1970) Aluminium chlorid ($AlCl_3$) and Aluminium chlorid with hydrochloric acid ($AlCl_3$/HCl) spectrums confirming the presence of ortho-3′, 4′ dihydroxy in B-ring with compound 2a because of the occurrence of bathochromic shift in band I with $AlCl_3$ compared with methanol spectrum (85nm) and the hypsochromic shift in the same band with $AlCl_3$ / HCl spectrum compared with $AlCl_3$ spctrum (32nm) as explained by Mabry et al (1970). The absence of bathochromic shift recorded band

Table (2): Mass spectral data of isolated flavonoids

Fragments	Compound							
	1	%	2a	%	2	%	3	%
$M^+ + 29$	389	(7)	331	(12)	477	(5)	---	---
$M^+ + 2$	362	(10)	304	(8)	---	---	---	---
$M^+ + 1$	361	(42)	303	(100)	449	(22)	433	(5)
$M^+ +$	360	(23)	302	(4)	448	(1)	434	(1)
$M^+ - 1\text{-}CH_3$	345	(17)	---	---	---	---	---	---
$M^+ + 2\text{-}CH_3$	331	(19)	---	---	---	---	---	---
$M^+ + 3\text{-}CH_3$	285	(30)	---	---	---	---	---	---
$M^+ + 1$-Rhamnose	---	---	---	---	303	(4)	286	(50)
M^+ - Rhamnose	---	---	---	---	302	(100)	287	(100)
M^+ - 29-Rgamnose	---	---	---	---	311	(12)	351	(12)
Unknown	259	(36)	207	(4)	147	(16)	129	(89)
Unknown	85	(100)	147	(30)	125	(87)	85	(69)

% = Abundance.

--- absent

Ia with $AlCl_3$/HCl compared with band I in 2a spectrum confirmed the presence of 3′, 5 free hydroxyl group Mabry *et al*, (1970). In sodium acetate spectrum, 2a gave the same bathochromic shift in band I confirming the presence of free7-hydroxyl group but, the observed degeneration of band I absorption peak after several minutes confirms the presence of alkali-sensitive grouping so, the compound 3 may contain 5,7,8 or 3,3′,4′ free hydroxyl groups according to Jurd and Horowitz, (1957). Mass molecular ions (Table2) at m/z 331,304 and 303 of parent mass at 302 are consistent with pentaoxygenated flavonoid without methylation or glycosylation patterns as evidenced by the absence of comparable peaks of this fragments (Mabry and Markham,1975).

Kaempeferol-3-O- α - L rhamnosid (3): isolated with 50-60% methanol in chloroform appearse as purple spot under UV and yellowish-green with NH_3 recording 0.078, 0.49 and 0.78 as R_f values. The UV spectral data (Table1) of 4 displayed λ max of methanol 410nm for band I and 265nm for band II with a shoulder at 282nm, confirming the presence of flavone or flavonol skeleton. Mabry *et al* (1970).A bathochromic shift was noted in band II 285nm relative to the standard (258)nm. Revealing the presence of free 5-hydroxyl group in A-ring as explained by Mabry *et al* (1970).In sodium methoxide spectrum a bathochromic shift in band I without a decrease in intensity was noticed indicating the presence of free ′4 hydroxyl group and substituted 3-hydroxyl group according to Mabry *et al* (1970).There was bathochromic shift in band II 8nm of alumminium chloroide spectrum compared to methanol spectrum while no shifts was observed after the addition of HCl in the same band. This confirmed the presence of free 5-hydroxyl group in A-ring while the presence of ′4-mono hydroxyl group in B- ring was confirmed by the absence of shift in band I with both spectrums.In sodium acetate spectrum bathochromic shifts were recorded in both bands (Band I and II) . There was no degeneration of the spectrum, confirming the presence of ′4, 7 dihydroxyl groups and a substituted 3- hydroxyl. Also, sodium acetate mixed with boric acid gave no bathochromic shifts in band I confirming the absence of O-dihydroxyl group in B-ring (Harborne 1975). Mass spectrum (Table2) of 3 showed a molcular

ion at m/z 434 of parent mass at 433 consistent with tetraoxygenated mono - glycosylated flavone. Comparable peaks were detected at 286 and 287 may refer to the loss of Rhamnosyl moiety from 3- glycosyl flavonoid ions (Mabry and Markham , 1975)

Kaempeferol (3a): 3, 5, 7, 4' tetra hydroxy flavone. Resulted from the complet acid hydrolysis of 3 . It gave yellow colore under UV and yellowish-green with NH_3 recording 0.83, 0.40 and 0.58 as R_f values the UV spectral data (Table1) of the hydrolysis product 3a recorded a similar results for the corresponding glycosides with results for the corresponding glycosides with methanol, $AlCl_3$ and $AlCl_3$ / HCl. The sodium methoxide spectrum showed a degenera -tion in band I that gave a good indication for the liberation by hydrolysis from 3- position and the presence of free 3- hydroxyl group .Also, the presence of free 7- hydroxyl group in A-ring was detected by the presence of 320- 330nm peak in band II, degeneration of UV absorption peak of sodium acetate in band I confirmed the presence of free 3- hydroxyl group and hydroxyl group that indicated the liberation of the sugar moiety from 3- position.The lack of shifts in sodium acetate with boric acid for band I illustrated the absence of ortho - dihydroxy group at B- ring.

*lignan(4): 7-hydroxy 3, 4, 3', 4' tetramethoxyl dibenzylbutry-olactone*isolated from acetone extract with (60:30:10) hexane: ethyl actate: acetone. The UV absorptions λ max with methanol at 207nm for band I and a shoulder at 218nm while band II recorded at 287nm. This absorpations of UV suggesting the presence of dibenzyl butyrolacotone lignan or trisubstituted furanoid lignan skeleton (Banerji et al, 1984 and Chen et al, 1997) Data from IR spectrum showed that 4 compound characterized by strong and broad band at 3431cm-1 for a free hydroxyl group (unphenolic) at 1547-1512cm-1 confirming the presence of aromatic rings or unsaturated bonds. Also, the IR absorptions at 1285-1381cm-1 revealed the (C-O) stretches peak may indicating the methoxyl groups.A strong IR band at 1730cm-1 was detected confirming the presence of a lactone carbonyl skeletal. This suggested the dibenzyl butyrolactone lignan structure for 4 (Chen. et al, 1997 and Marlinez et al, 1998).The H-NMR spectral data (Table3) showed doublets of 7-protons at

Table (3): H^1-NMR data of isolated lignans 4 and 5

Protons (s) at C-atom No	Chemical shift)*∝in ppm)	
	4	5
'7α	2.35(d)	2.27(d)
β	2.91(d)	2.89(d)
7α	2.69(d)	2.30(d)
β	--------	2.96(d)
'8	4.83(m)	4.85(m)
8	5.37(m)	5.78(m)
'9α	4.31(t)	4.37(t)
β	4.12(t)	4.07(t)
-OH	7.26(s)	7.25(s)
-OCH3	unclear	3.64(s)
O-CH2-O	---------	6.61(s)
'2	6.98(d)	6.94(d)
2	7.16(d)	6.81(d)
'5	7.52(q)	7.58(d)
5	7.74(q)	------
'6	7.78(q)	7.72(q)
6	7.63(q)	7.50(q)

2.27, 2.89ppm confirming presence of (2H). and one doublet signals at 2.69ppm confirming one (C-7) and a substitution at 7 position. The C-9 proton appears as triplets at 4.31 and 4.12ppm for two protons (2H) while C-8 and C-8 protons appears as multiplets signals at 5.37 and 4.83ppm. these data confirming the lactone skeletal for 4.In addition of aromatic proton 2',2 appears as doublets signals at 6.89 and 7.16ppm but the other aromatic proton 5',6',5,6 appears as quarted signals 7.52-7.78ppm. Indicating the 3,4 and 3',4' substitution of the dibenzyl butyrolactone.A singelet broad peak at 7.26ppm. revealed the presence of free hydroxyl group. The unclear peak at 3.6ppm may reveals the presence of methoxyl groups at the substitution positions of 3,3',4,4' (Ayres and loike, 1990).Mass spectral data showed molcular ions of 4 at m/z 403 of parent mass 402. The appearance of peaks m/z 151 and 165 indicating the presence of 3,4 methoxy phenyl substituents. The greater abundance of the fragment 151 than that m/z 165 was taken as evidence that the dimethoxy substituted aromatic group was a benzyl rather than a phenyl, substituent. (Mac Rae and Towers 1985).The fragments m/z 251 and 237 showed the fragmentation of the benzyl butyrolactone while fragments at m/z 85 and 57 showed fragmentation of lactone skeletal, that indicating the dibenzyl butyrolactone sturture for L1 (Chen et al 1997). The position of hydroxy group at C-7 was supported by fragmentation (a) and (b) in (Fig 1).

5 hydroxy-bursehernin (5): 3, 4-dimethoxy-5 hydroxy-3',4' methelendioxy – dibenylbutyro - lacton. The UV Spectral data showed two bands as λ max in methanol at 215.5nm for band I and 278nm for band II, confirming the furanoid or dibenzylbutryolactone lignan structure for compound 5.As shown, the compound 5 was generally characterized by broad IR band at 1271 cm^{-1} 2854-2988 cm^{-1} for (C-O) stretches and (C-H) aliphatic bonds together with the aromatic skeletal band (C = C) at 1461-1546 Cm^{-1}. The IR spectrum revealed the presence of a lactone carbonyl absorption at 1726.7 cm^{-1} as recommeded by Chen *et al* 1997. That indicated the dibenzylbutyrolactone skelaton for 5. The presence of phenolic hydroxyl group (-OH) in the molecule was indicated by IR absorption at 3380 cm^{-1}. Gamal *et al* (1997). The IR spectrum also revealed the presence of a

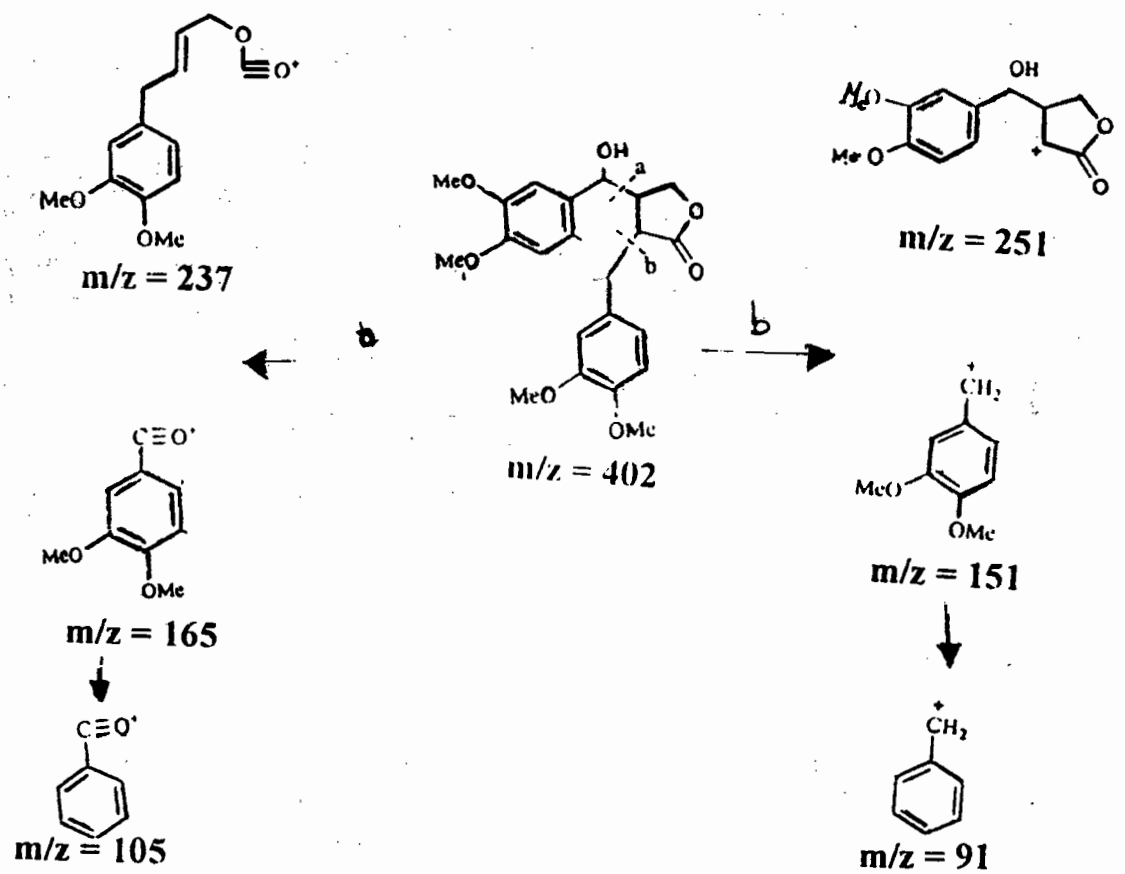


Fig. (1) : Fragmentation of 4

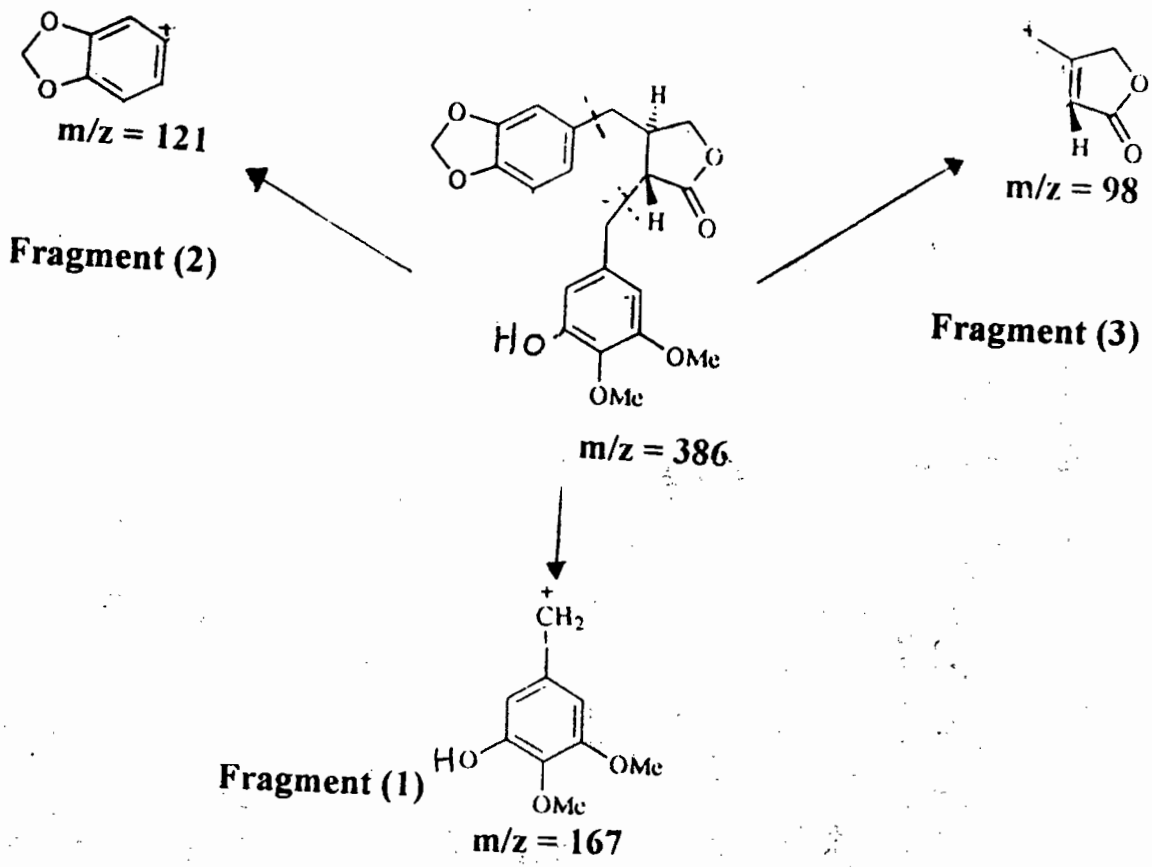


Fig. (2) : Fragmentation of 5

methylenedioxy group at 1030 and 910 cm^{-1} (Chen *et al* 1997). the H^1 NMR spectrum of 5 revealed a dibenzybutgrolactone skeleton. The appearance of doublets at 2.27 and 2.96 ppm of C-7 (4-protons) and the triplets at 4.37,4.07ppm of C-9 proton (2H) together with the multiblets at 4.83 and 5.703ppm of C-8 and 8 (protons) (2H) confirming the butyrolactone structure. The aromatic protons of C-2 and C-2′ appears as doublet signals at 6.942 and 6.861ppm with the quartets of 5,6, at 7.50, and 7.69ppm while the aromatic proton of 6 appeared at 7.205ppm. The single broad peak at 7.25ppm confirming the presence of free hydroxyl group. While the same single peak appears at 3.647ppm supporting the presence of methoxyl protons. Also, the single peak at 6.615ppm revealed the presence of two protons of methylenedioxy group.the fragmentation's of the parent mass (Fig2) at m/z 386 of 5 showed the presence of fragments m/z = 121 and 167 confirming the 3,4 methoxy-5 hydroxl phenyl substituents and 3′,4′ methelendioxy substituents of 5. The higher abundance of fragment m/z 121 confirms that the 3,4 methylenedioxy substituted aromatic group was a benzyl rather than phenyl substituent and the-OH group was substituted with (C-6) position. (Mac Rae and Towers 1985).

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فصل بعض المكونات الحيوية من نبات العرعر والتعرف عليها

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ينمو نبات العرعر بريا في منطقة شمال سيناء حيث استخدم قديما كمضاد للتقلصات مخفض للسكر في الدم، وأيضا لعلاج الحساسية والسعال المزمن وقد تم فصل أربعة مركبات فلافونية من المستخلص الكحولي ٧٠%وهى:جاسيدين(٣،٦، ٣/ ثلاثي ميثوكسى-٥،٧،٤/ ثلاثى هيدروكسى فلافون)،كيرستين(٣،٧،٥،٣/،٤/خماسي هيدروكسى فلافون):كيرسترين (كيرستين-٣ -L-α-O رامنوزيد) وكمبفيرول- ٣ -L-α- O رامنوزيد (٣/ثلاثى هيدروكسى -٣ -L-α-O رامنوزيد) بالإضافة إلى مركبين من مركبات الليجنان من مستخلص الأسيتون وهى ٥-هيدروكسى بيورسيهيرنين(٣،٤ ثنائي ميثوكسى - ٥هيدروكسى-٤/٣،/ميثلين داى ميثوكسى ثنائى بنزيل بيترولاكتون و ٧-هيدروكسي - ٣،٣،٤/ ،٤/ رباعي ميثوكسي ثنائي بنزيل بيترولاكتون وقد تم التعرف على تركيب المركبات المفصولة باستخدام تحليل طيف الأشعة فوق البنفسجية، طيف الأشعة تحت الحمراء ، طيف الكتلة والرنين المغناطيسي للبروتونات.