

# Xanthone and lignan glycosides from the aerial parts of *Polygonum bellardii* all growing in Egypt

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## ABSTRACT

A new long chain fatty alcohol acetate identified as 17-hydroxypentacosanyl acetate, (**1**) together with a new xanthone identified as 1,8-Dihydroxy-3,6-dimethoxy-xanthone-5-*O*-[ $\alpha$ -L-rhamnopyranosyl-(1'' $\rightarrow$ 2')] - $\beta$ -D-glucopyranoside (**3**), as well as two new lignans identified as (+)-Lyoniresinol-3a-*O*-[ $\alpha$ -L-rhamnopyranosyl-(1''' $\rightarrow$ 6'')] - $\beta$ -D-glucopyranoside (**4**) and (+)-Isolariciresinol-3a-*O*-[ $\alpha$ -L-rhamnopyranosyl-(1''' $\rightarrow$ 2'')- $\alpha$ -L-rhamnopyranosyl-(1'''' $\rightarrow$ 6'')] - $\beta$ -D-glucopyranoside (**5**), in addition to  $\beta$ -sitosterol-3-*O*-acetate (**2**) were isolated from the methanolic extract of the aerial parts of *Polygonum bellardii* growing in Egypt. Their structures were elucidated on the basis of different chemical and spectroscopic evidences. The total extract and its fractions, in addition to compounds (**3**, **4** and **5**) showed significant antioxidant potential by DPPH\* scavenging activity technique.

**Key words:** Antioxidant, lignans, *Polygonaceae*, *Polygonum bellardii*, xanthone

## INTRODUCTION

*Polygonum bellardii* (Polygonaceae) grows in sandy places around Alexandria, Mandara, Abukir, Damietta, Tel-El-Kebir, Assiut, Ekhmim, Dendera, Farafra and Kharge, known locally as Maksus-El-Gariyla, Qardab or Qordob.<sup>[1]</sup> Many species of the genus *Polygonum* had been reported to exhibit a variety of interesting biological activities as: Laxative, expectorant, diuretic, tonic, anti-aging, anti-inflammatory, antibacterial, anti-allergic, anthelmintic, astringent, haemostatic and demulcent.<sup>[2-5]</sup> Moreover, some other species were used in treatment of skin burns, gallstone, hepatitis, osteomyelitis, nephritis, cystitis, suppurative dermatitis, gonorrhoea, chronic gastritis, duodenal ulcers, gout, haemorrhoids and dental diseases.<sup>[6-9]</sup> A variety of chemical constituents such as flavonoids,<sup>[10,11]</sup> anthraquinones,<sup>[12]</sup> sesquiterpenoids,<sup>[13]</sup> lignans,<sup>[14]</sup> coumarins<sup>[15]</sup> and stilbenes<sup>[16]</sup> have been reported from the genus *Polygonum*.

Generally Polyphenolic compounds, including phenols, phenolic acids, flavonoids, tannins and lignans have been shown to be potent antioxidants because of their radical

scavenging activity and their ability to form complex with heavy metal ions.<sup>[17,18]</sup> The former chemical and biological information about the genus *Polygonum* motivated the investigation of *Polygonum bellardii* All. growing in Egypt for its chemical composition and antioxidant activity.

## MATERIAL AND METHODS

### General

The UV was measured on Ultrospec 1000, UV/visible Spectrometer, Pharmacia Biotech (England). FAB-MS was measured on JEOL JMS 600 Hz (Japan). IR was measured on Shimadzu Infra-red-470 spectrophotometer (Japan). NMR analysis (<sup>1</sup>H-NMR, <sup>13</sup>C-NMR, HMBC) were measured on JEOL TNM-LA 600 MHz FT- NMR spectrometer (Japan), Varian mercury 400 MHz Spectrometer (Oxford) using TMS as internal standard. Column chromatography was carried on silica gel (70-230, mesh, Merck), Sephadex LH-20 (20-100  $\mu$ m, Sigma-Aldrich chemicals) and RP-18 (30-50  $\mu$ m), TLC was carried on aluminium plates covered with a 0.25 mm silica (kieselgel 60 GF<sub>254</sub>, Merck, Germany). The plates were examined under UV light (at 365 and 254 nm) and visualized by spraying with 20% *v/v* H<sub>2</sub>SO<sub>4</sub> in EtOH, allowed to dry at room temperature followed by heating at 110-140°C for 1-2 min. The following solvent systems were used for TLC: I- *n*-Hexane-EtOAc (9:1 *v/v*) II- *n*-Hexane-EtOAc (8:2 *v/v*)

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- III- CHCl<sub>3</sub>-MeOH-H<sub>2</sub>O (8: 2:0.2 v/v)  
 IV- CHCl<sub>3</sub>-MeOH-H<sub>2</sub>O (7.5: 2.5:0.3 v/v)  
 V Toluene-(CH<sub>3</sub>)<sub>2</sub>C=O-HCHO (3:6:1 v/v)  
 VI- MeOH-H<sub>2</sub>O (6: 4 v/v)

### Plant material

The aerial parts of *Polygonum bellardii* All. were collected during flowering stage in April 2007 from Assiut governorate and kindly identified and authenticated by Prof. Dr. Moamen M. Zarea, Professor of Plant Taxonomy, Department of Botany, Faculty of Science, Assiut University, Assiut, Egypt. A voucher specimen was kept in the Department of Pharmacognosy, Faculty of Pharmacy, Al-Azhar University, Assiut, Egypt.

### Authentic sugar materials

D-glucose, L-rhamnose, L-arabinose and L-galactose [obtained from El-Nasr Pharmaceutical and Chemical Co., Egypt (ADWIC) and Sigma-Aldrich chemicals].

### Antioxidant materials

2,2-Diphenyl-1-picryl hydrazyl (DPPH), ascorbic acid and quercetin as an antioxidant standard (all were obtained from Sigma-Aldrich Chemicals Co.) and Digital Ultrasonic Cleaner, MTI Corporation, USA for mixing samples.

### Extraction and isolation

The air-dried powdered aerial parts (2 Kg) of *Polygonum bellardii* All. were extracted by maceration and percolation with methanol till complete exhaustion (1:4 ratio) [four times each 8 L; overnight]. The combined methanolic extracts were concentrated under reduced pressure till constant weight to give a syrupy residue (150 g). The extract was subjected to successive solvent fractionation on VLC with *n*-hexane, chloroform, ethyl acetate, *n*-butanol and methanol till complete exhaustion in each case to give *n*-hexane (20 g), chloroform (25 g), ethyl acetate (20 g), *n*-butanol (10 g) and methanol (50 g).

The chloroform-soluble fraction (10g) was chromatographed on silica gel column chromatography, eluted with *n*-hexane and *n*-hexane-ethyl acetate gradients. Fractions 100ml, each were collected. Similar fractions were pooled together, concentrated under reduced pressure to give 6 sub-fractions labeled PCH1-PCH6. A part of sub-fraction PCH3 (500mg), was rechromatographed on silica gel column chromatography and eluted with *n*-hexane, followed by *n*-hexane-ethyl acetate gradients. The fractions eluted with *n*-hexane- ethyl acetate (9:1) afforded compound **1** (35 mg), whereas fractions eluted with *n*-hexane-ethyl acetate (8:2) afforded compound **2** (30 mg).

The ethyl acetate-soluble fraction (10 g) was chromatographed on silica gel column, eluted with CHCl<sub>3</sub> followed by CHCl<sub>3</sub>-MeOH gradiently; eluates, 100 ml each were collected. Related fractions were grouped together and concentrated under reduced pressure to give 5 sub-fractions labeled PE1-PE5. Fraction PE4 (150mg) was chromatographed on Sephadex LH-20 using CHCl<sub>3</sub>-MeOH (1:1). Fractions 8-12 were pooled together, concentrated under reduced pressure and chromatographed on RP-18 column using MeOH-H<sub>2</sub>O (6:4), where fractions 5-8 afforded compound **3** (40 mg).

The *n*-butanol soluble fraction (5 g) was chromatographed on silica gel column chromatography eluted with CHCl<sub>3</sub> followed by CHCl<sub>3</sub>-MeOH gradients; fractions 100 ml, each were collected. Similar fractions were grouped and concentrated under reduced pressure to give 3 sub-fractions labeled PN1-PN3.

Sub-fraction PN2 (200 mg) was chromatographed on Sephadex LH-20 using CHCl<sub>3</sub>-MeOH (1:1). Fractions 10- 15 were collected, concentrated under reduced pressure and rechromatographed on RP-18 using MeOH-H<sub>2</sub>O (1:1), afforded compound **4** (50 mg). Furthermore fractions 19-25 rechromatographed on RP-18 using MeOH-H<sub>2</sub>O (1:1), afforded compound **5** (35 mg).

### Compound 1 [17-hydroxypentacosanyl acetate]:

Obtained as white amorphous powder from CHCl<sub>3</sub>, R<sub>f</sub> = 0.5 (system, I), [α]<sub>D</sub><sup>20</sup> +10.3 (MeOH, c.003). The IR spectrum (KBr) showed broad absorption band at 3410 cm<sup>-1</sup> (OH stretching), in addition to other bands at 2995 cm<sup>-1</sup> (CH stretching), 1722 cm<sup>-1</sup> (C = O stretching), 1450 cm<sup>-1</sup> (CH<sub>2</sub> bending) and 1217 cm<sup>-1</sup>, 1066 and 989 (C-O stretching). EI mass spectrum showed the molecular ion peak at *m/z* 426 [M]<sup>+</sup> (15%), other peaks at *m/z* 411 [M-CH<sub>3</sub>]<sup>+</sup> (3.8%), 408 [M-H<sub>2</sub>O]<sup>+</sup> (2.7%), 367 [M-CH<sub>3</sub>COO]<sup>+</sup> (10%), 313 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>]<sup>+</sup> (2.7%), 283 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>CHOH]<sup>+</sup> (3.3%), 269 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>7</sub>COO]<sup>+</sup> (1.4%), 157 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>16</sub>CHOH]<sup>+</sup> (2.6%), 143 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>16</sub>COO]<sup>+</sup> (6.3%), 113 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>16</sub>COOCHOH]<sup>+</sup> (58.2%), 59 [M-CH<sub>3</sub>(CH<sub>2</sub>)<sub>23</sub>CHOH]<sup>+</sup> (4%). Selected <sup>1</sup>H-NMR spectral data (CD<sub>3</sub>OD, 600 MHz): δ<sub>H</sub> 0.94 (3H, t, H<sub>3</sub>-25), 1.28-1.62 (30 H, m, CH<sub>2</sub> residues), 2.30 (2H, m, H<sub>2</sub>-2), 2.29 (3H, s, CH<sub>3</sub>-C=O), 3.98 (1H, m, H-17), and 4.06 (2H, t, H<sub>2</sub>-1). Selected <sup>13</sup>C-NMR spectral data (CD<sub>3</sub>OD, 150 MHz): δ<sub>C</sub> 13.94 (C-25), 23.66 (C-24), 26.66 (C-2), 29.89-30.29 (CH<sub>2</sub> residue), 31.77 (C-23), 35.03 (C-16 and C-18), 65.14 (C-1), and 69.00 (C-17) in addition to δ<sub>C</sub> 20.11, and 175.61(CH<sub>3</sub>CO).

**Compound 2 [β-sitosterol acetate]:** Obtained as white amorphous powder from CHCl<sub>3</sub>, R<sub>f</sub> = 0.6 (system, II).

Positive FAB-MS showed peak at  $m/z$  457  $[M+H]^+$  and 415  $[(M+1)\text{-acetyl}]^+$ .  $^1\text{H-NMR}$  spectral data ( $\text{CD}_3\text{OD}$ , 600 MHz):  $\delta_{\text{H}}$  0.72 (3H, s,  $\text{CH}_3$ -18), 0.85 (3H, d,  $J=6.8$  Hz,  $\text{CH}_3$ -26), 0.88 (3H, d,  $J=7.5$  Hz,  $\text{CH}_3$ -27), 0.90 (3H, d,  $J=7.5$  Hz,  $\text{CH}_3$ -29), 1.00 (3H, d,  $J=6.2$  Hz,  $\text{CH}_3$ -21), 1.05 (3 H, s,  $\text{CH}_3$ -19), 1.24-2.03 (m, other CH and  $\text{CH}_2$ ), 2.05 (3H, s,  $\text{CO-CH}_3$ ), 2.32 (1H, br.s, H-4), 3.45 (1 H, m, H-3), and 5.37 (1 H, br s, H-6).  $^{13}\text{C-NMR}$  ( $\text{CD}_3\text{OD}$ , 150 MHz):  $\delta_{\text{C}}$  37.29 (C-1), 30.62 (C-2), 77.79 (C-3), 39.67 (C-4), 142.86 (C-5), 122.76 (C-6), 32.92 (C-7), 32.92 (C-8), 49.42 (C-9), 37.29 (C-10), 21.63 (C-11), 39.67 (C-12), 42.13 (C-13), 58.34 (C-14), 25.92 (C-15), 30.26 (C-16), 57.56 (C-17), 12.23 (C-18), 19.32 (C-19), 37.29 (C-20), 19.32 (C-21), 34.98 (C-22), 25.92 (C-23), 47.43 (C-24), 30.26 (C-25), 20.07 (C-26), 21.63 (C-27), 24.02 (C-28), 14.36 (C-29), 20.07 ( $\text{CO-CH}_3$ ) and 175.20 ( $\text{CO-CH}_3$ ).

**Compound 3 [1,8-Dihydroxy-3,6-dimethoxyxanthone-5-O- $[\alpha\text{-L-rhamnopyranosyl-(1''\rightarrow 2')}]$ - $\beta\text{-D-glucopyranoside}$ ]:** Obtained as yellowish needles from MeOH (m.p. 177-180),  $R_f = 0.40$  (system, III),  $[\alpha]_{\text{D}}^{20} -9.8$  (MeOH, c.001). Positive FAB-MS showed peak at  $m/z$  613  $[M+1]^+$ , 467  $[(M+1)\text{-rhamnose}]^+$  and 305  $[(M+1)\text{-}(rhamnose + glucose)]^+$ .  $^1\text{H-NMR}$  spectral data ( $\text{DMSO-}d_6$ , 600 MHz) was listed in Table 1.  $^{13}\text{C-NMR}$  spectral data ( $\text{DMSO-}d_6$ , 150 MHz) was listed in Table 2.

**Compound 4 [Lyoniresinol-3a-O- $[\alpha\text{-L-rhamnopyranosyl-(1'''\rightarrow 6'')}]$ - $\beta\text{-D-glucopyranoside}$ ]:** Obtained as a white amorphous powder from MeOH,  $R_f = 0.40$  (system, IV),  $[\alpha]_{\text{D}}^{20} +9.6$  (MeOH, c.05). Positive FAB-MS showed peak at  $m/z$  729  $[M+1]^+$  and other peaks at 583  $[(M+1)\text{-rhamnose}]^+$  and 421  $[(M+1)\text{-}(glucose + rhamnose)]^+$ . The  $^1\text{H-NMR}$  spectral data ( $\text{DMSO-}d_6$ , 400 MHz) was listed in Table 3.  $^{13}\text{C-NMR}$  spectral data ( $\text{DMSO-}d_6$ , 100 MHz) was listed in Table 4.

**Compound 5 [(+)-Isolariciresinol-3a-O- $[\alpha\text{-L-rhamnopyranosyl-(1'''\rightarrow 2'')}]$ - $\alpha\text{-L-rhamnopyranosyl-(1'''\rightarrow 6'')}]$ - $\beta\text{-D-glucopyranoside}$ ]:** Obtained as white powder from MeOH,  $R_f = 0.35$  (system, IV),  $[\alpha]_{\text{D}}^{20} +11$  (MeOH, c. 02). Positive FAB-MS showed peak at  $m/z$  815  $[M+1]^+$  and other peaks at 669  $[(M+1)\text{-rhamnose}]^+$ , 521  $[(M+1)\text{-two rhamnose units}]^+$ , 361  $[(M+1)\text{-}(glucose + two rhamnose units)]^+$ . The  $^1\text{H-NMR}$  spectral data ( $\text{CD}_3\text{OD}$ , 600 MHz) was listed in Table 3 and  $^{13}\text{C-NMR}$  spectral data ( $\text{CD}_3\text{OD}$ , 150 MHz) was listed in Table 4.

#### DPPH radical-scavenging assay

Firstly, free radical scavenging activity of the total methanolic extract, its fractions in addition to compounds 3, 4 and 5 isolated from the aerial parts of *Polygonum bellardii* All. was carried out against stable DPPH $\cdot$  with a rapid TLC screening method using 0.2% DPPH $\cdot$  in methanol. Thirty

**Table 1:  $^1\text{H-NMR}$  spectral data of compound 3 ( $\text{DMSO-}d_6$ , 600 MHz)**

Chemical shift ( $\delta$ ) ppm	No. of protons, Multiplicity	Coupling constant (Hz)	Assignment
1.19	3H, d	6.0	H-6''
3.77	3H, s	-	3-OCH $_3$
3.91	3H, s	-	6-OCH $_3$
4.47-5.32	16H, m	-	Sugar protons
5.17	1H, br.s	-	H-1''
5.35	1H, d	7.4	H-1'
6.37	1H.,s	-	H-7
6.55	1H, br.s	-	H-2
6.67	1H, br.s	-	H-4
11.72	1H, s	-	1-OH
11.87	1H, s	-	8-OH

**Table 2:  $^{13}\text{C-NMR}$  spectral data of compound 3 ( $\text{DMSO-}d_6$ , 150 MHz)**

Carbon No.	Chemical shift	Carbon No.	Chemical shift
1	161.71	Glucose	
2	97.59	1'	99.01
3	164.10	2'	76.38
4	94.82	3'	77.03
5	128.40	4'	69.67
6	160.10	5'	77.10
7	95.60	6'	60.53
8	157.47		
9	182.86	Rhamnose	
4a	157.02	1''	100.53
4b	148.45	2''	70.41
8a	101.08	3''	70.48
8b	102.18	4''	71.85
3-OCH $_3$	56.66	5''	68.38
6-OCH $_3$	60.99	6''	18.07

minutes after spraying, the active compounds appear as yellow spots against purple background.<sup>[19]</sup>

In a second experiment, DPPH $\cdot$  scavenging activity was measured by spectrophotometric method.<sup>[20]</sup> Briefly, 1ml of a wide range of concentrations (25-200  $\mu\text{g/ml}$ ) of test sample (in methanol) was added to 1 ml of 200  $\mu\text{M}$  of methanolic solution of DPPH $\cdot$ . The mixture was mixed by Digital Ultrasonic Cleaner (sonicator) for 1 min and then left to stand at room temperature for 30 min in dark. When DPPH $\cdot$  reacts with an antioxidant compound, which can donate hydrogen, it is reduced and the changes in colour (from deep-violet to light-yellow) were measured at 517nm on a UV/visible light spectrophotometer. Absorption of blank sample containing the same amount of methanol and DPPH $\cdot$  solution was prepared and measured daily. The experiment was carried out in triplicate, using ascorbic acid and quercetin as a positive control standards.<sup>[21]</sup> The percentage reduction of the DPPH $\cdot$  was calculated by the following formula: %Inhibition (scavenging) =  $[(A_b - A_s)/A_b] \times 100$  Where:  $A_b$ -

**Table 3: <sup>1</sup>H-NMR spectral data of compound 4 and 5 (DMSO-*d*<sub>6</sub>, 400 and 600 MHz)**

Proton No	4			5		
	Chemical shift	Multiplicity	Coupling constant (Hz)	Chemical shift	Multiplicity	Coupling constant (Hz)
1	2.87	2H, m	-	3.21	2H, m	-
2	1.92	1H, m	-	1.84	1H, m	-
2a	3.41	2H, m	-	3.62	1H, dd	10.8, 4.5
-	-	-	-	3.65	1H, dd	10.8, 6.1
3	2.71	1H, m	-	2.72	1H, m	-
3a	4.06	1H, dd	11, 6	3.73	1H, dd	9.9, 3.1
-	4.15	1H, dd	11, 4.2	3.75	1H, dd	9.9, 4.6
4	4.26	1H, d	6	4.42	1H, d	5.8
5	-	-	-	6.56	1H, s	-
8	6.53	1H, s	-	6.53	1H, s	-
2'	6.29	1H, s	-	6.54	1H, d	2.1
5'	-	-	-	6.66	1H, d	7.9
6'	6.29	1H, s	-	6.49	1H, d	7.9, 2.1
5-OCH <sub>3</sub>	3.69	3H, s	-	-	-	-
7-OCH <sub>3</sub>	3.75	3H, s	-	3.70	3H, s	-
3'-OCH <sub>3</sub>	3.63	3H, s	-	3.67	3H, s	-
5'-OCH <sub>3</sub>	3.63	3H, s	-	-	-	-
1''	4.60	1H, d	7.6	4.18	1H, d	7.9
1'''	6.08	1H, s	-	6.07	1H, s	-
1''''	-	-	-	6.07	1H, s	-
6'''	1.11	1H, d	6.5	1.19	1H, d	6.5
6''''	-	-	-	1.09	1H, d	6.5

**Table 4: <sup>13</sup>C-NMR spectral data of compounds 4 and 5 (DMSO-*d*<sub>6</sub>, 100 and 150 MHz)**

Carbon No Aglycone	Chemical shift		Carbon No Sugar part	Chemical shift	
	4	5		4	5
1	35.91	33.56	Glucose		
2	41.10	40.07	1''	103.87	
2a	65.94	66.96	2''	73.41	102.24
3	48.70	48.31	3''	76.80	74.91
3a	70.75	70.09	4''	69.04	77.91
4	44.68	45.47	5''	77.29	68.01
5	146.50	117.14	6''	64.04	78.23
6	137.35	145.22	Rhamnose	100.86	65.36
7	147.06	147.23	1'''	69.74	101.92
8	106.70	113.54	2'''	70.86	71.64
9	128.59	128.90	3'''	70.99	72.51
10	124.90	134.00	4'''	69.55	73.91
1'	137.83	138.04	5'''	17.99	70.09
2'	105.65	116.08	6'''	-	17.90
3'	147.60	149.14	Rhamnose	-	101.22
4'	133.44	146.07	1''''	-	71.19
5'	147.60	117.13	2''''	-	72.29
6'	105.98	123.16	3''''	-	73.84
5-OCH <sub>3</sub>	58.90	-	4''''	-	70.09
7-OCH <sub>3</sub>	55.76	56.39	5''''	-	17.89
3'-OCH <sub>3</sub>	56.12	-	6''''	-	
5'-OCH <sub>3</sub>	56.12	56.39			

absorption of blank sample ( $t = 0$  min) and  $A_A$ -absorption of tested extract solution ( $t = 30$  min).

## RESULTS AND DISCUSSION

### Compound 1

The low resolution EI-MS showed the molecular ion

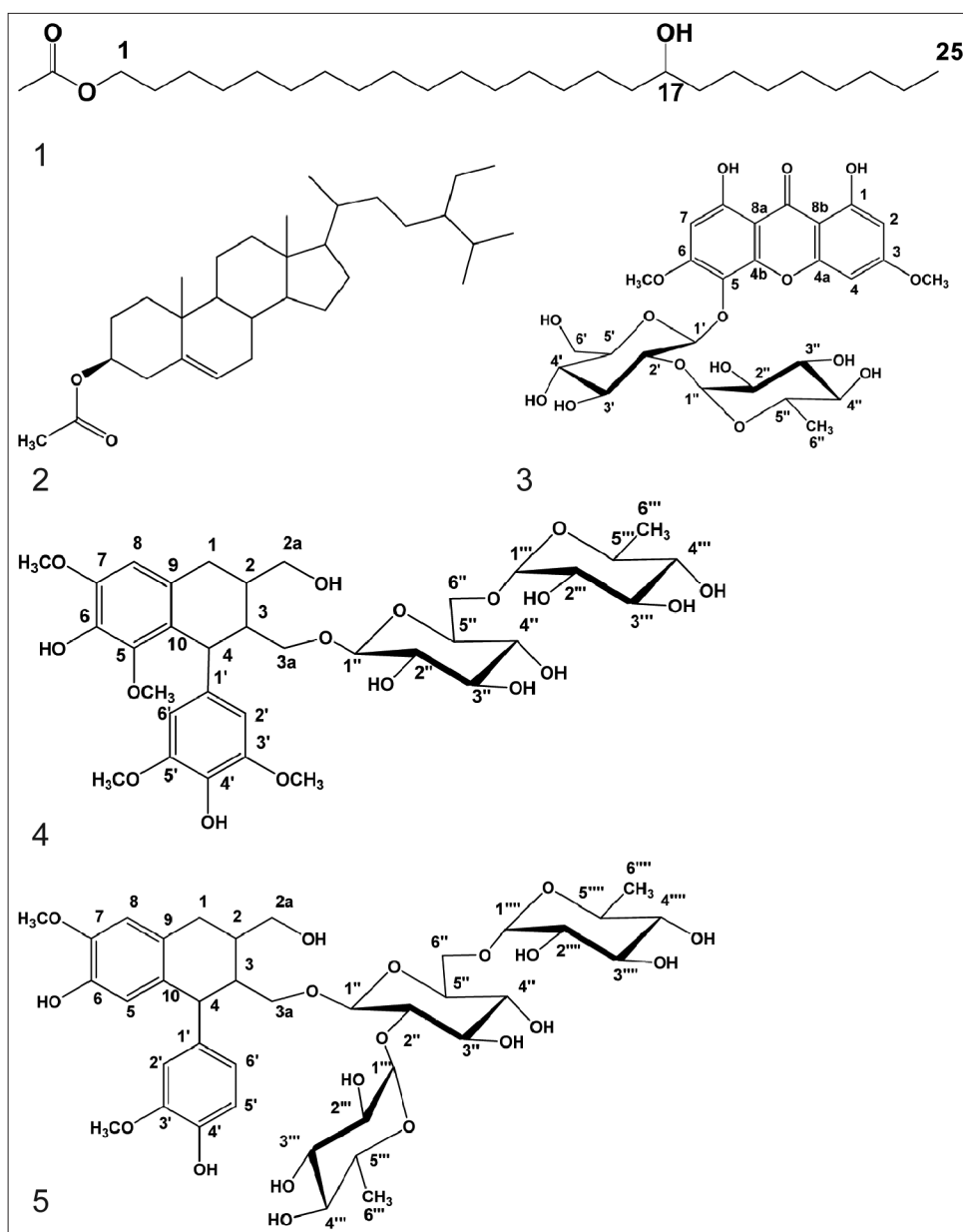
peak  $[M]^+$  at  $m/z$  426, together with IR, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectral data confirmed the molecular formula to be C<sub>27</sub>H<sub>54</sub>O<sub>3</sub>. Careful investigation of <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectral data of compound **1** demonstrated the presence of long chain aliphatic ester moiety. EI-MS showed a peak at 408  $[M-H_2O]^+$  (2.7%) indicated the presence of hydroxyl group, confirmed from IR band at 3410. Promising abundant peaks

at  $m/z$  143  $[M-CH_3(CH_2)_{16}COO]^+$  (63%) and 113  $[M-CH_3(CH_2)_{16}COOCHOH]^+$  (58.2%) indicated the presence of hydroxyl group at C-17. Additionally, it revealed the presence of an ester methyl moiety at  $m/z$  59 (4%). The  $^1H$ -NMR showed a triplet methyl at  $\delta_H$  0.94 (3H, t) attributed to terminal methyl at C-25 together with another methyl signal at  $\delta_H$  2.63 (3H, s) belonging to methyl protons of the acetyl group. It also showed a signal at  $\delta_H$  3.98 (1H, m) assigned to oxygenated methine group. Moreover, it revealed the presence of a triplet signal at  $\delta_H$  4.06 (2H, t, H-1) and a multiplet signal at  $\delta_H$  2.30 (2H, m, H-2). Moreover, a cluster of  $CH_2$  signals at  $\delta_H$  1.28-1.6 (30 H, m) indicative for long chain aliphatic moiety, which was obvious from  $^{13}C$ -NMR

signals at  $\delta_C$  29.89-30.29.<sup>[22,23]</sup> The  $^{13}C$ -NMR spectrum displayed two signals at  $\delta_C$  175.61 and 20.11, assigned to acetyl group. The oxygenated methine carbon appeared at  $\delta_C$  69.00, while the terminal methyl appeared at  $\delta_C$  13.94. Therefore, compound **1** was assigned as 17-hydroxypentacosanyl acetate,<sup>[22,23]</sup> a new natural product [Figure 1-3, Tables 1-4].

### Compound 2

Chemical investigation of compound **2** indicated its steroidal nature.<sup>[24]</sup>  $^1H$ -NMR spectrum of compound **2** showed six methyl signals at  $\delta_H$  0.72, 0.85, 0.88, 0.90, 1.00 and 1.05 each (3H, s) assigned to  $CH_3$ -18, 26, 27, 29, 21 and 19 respectively in addition to an extra downfield



**Figure 1:** Structures of the isolated compounds 1- 5

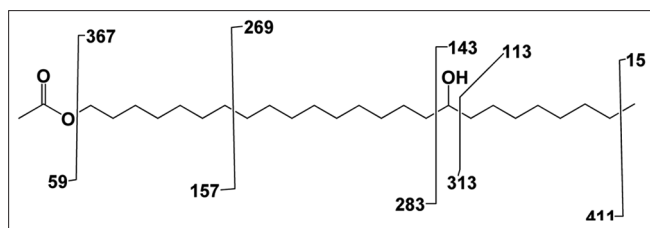


Figure 2: Significant fragments of compound 1

shifted oxygenated methyl signal at  $\delta_{\text{H}}$  2.05 (3H, s) allocated to acetyl group. It also showed signals at  $\delta_{\text{H}}$  2.32 (1H, br. s, H-4), 3.45 (1 H, m, H-3) and 5.37 (1H, br s, H-6). The  $^1\text{H-NMR}$  together with  $^{13}\text{C-NMR}$  data concluded the existence of acetylated  $\beta$ -sitosterol moiety, this was confirmed from  $^{13}\text{C-NMR}$  signals at  $\delta_{\text{C}}$  20.07 and 175.35 assigned to acetate moiety. The localization of acetyl group at C-3 was obvious from the downfield shift of C-3 at  $\delta_{\text{C}}$  77.79 upon comparison with non-substituted  $\beta$ -sitosterol.<sup>[25]</sup> From the previous data and upon comparison with literature,<sup>[26,27]</sup> compound 2 was identified as  $\beta$ -sitosterol-3-O-acetate, previously isolated from *Prunella vulgaris*<sup>[26]</sup> and firstly reported from the genus *Polygonum*.

### Compound 3

The  $^1\text{H}$  and  $^{13}\text{C-NMR}$  measurements of compound 3 indicated the presence of xanthone glycoside moiety.<sup>[28]</sup> Positive FAB-MS spectrum showed the quasi-molecular ion peak at  $m/z$  613  $[\text{M}+1]^+$ , together with  $^1\text{H}$ ,  $^{13}\text{C-NMR}$  spectral data confirmed the molecular formula to be  $\text{C}_{27}\text{H}_{32}\text{O}_{16}$ .  $^1\text{H-NMR}$  spectrum of compound 3 showed two downfield shifted singlet signals at  $\delta_{\text{H}}$  11.72, 11.87 each (1H, s) ascribed to chelated 1,8-OH groups respectively. The singlet signal at  $\delta_{\text{H}}$  6.37 and broad singlet signals at  $\delta_{\text{H}}$  6.55 and 6.67 were assigned to isolated aromatic protons H-7, H-2 and H-4 respectively,<sup>[29-32]</sup> two intense signals appeared at  $\delta_{\text{H}}$  3.91, 3.77 each (3H, s) indicating two aromatic methoxy groups. Moreover, the  $^1\text{H-NMR}$  showed signals at  $\delta_{\text{H}}$  5.35 (1H d,  $J = 7.4$  Hz) and 5.17 (1H, s) assigned to couple of anomeric sugar protons, indicative for the existence of bioside moiety. The previous  $^1\text{H-NMR}$  data together with diagnostic  $^{13}\text{C-NMR}$  signals listed in Table 2 were conclusive for the presence of pentasubstituted 1,8-dihydroxy bioside moiety. The site of sugar attachment at C-5 was obvious from  $^{13}\text{C-NMR}$  downfield shifted signals at  $\delta_{\text{C}}$  128.4 allocated to C-5 upon comparison with C-5 unsubstituted 1,8 dihydroxyxanthone series.<sup>[28-32]</sup>  $^{13}\text{C-NMR}$  displayed pair of diagnostic signals at  $\delta_{\text{C}}$  99.01, 100.53 attributed to two anomeric sugar carbons.<sup>[28-32]</sup> The aforementioned data associated with  $^{13}\text{C-NMR}$  prominent signals at  $\delta_{\text{C}}$  99.01, 76.38, 77.03, 69.67, 77.10 and 60.53 deduced the occurrence of C-2 substituted glucose moiety. The position of the intraglycosidic linkage was approved from  $^{13}\text{C-NMR}$  downfield shifted signal at

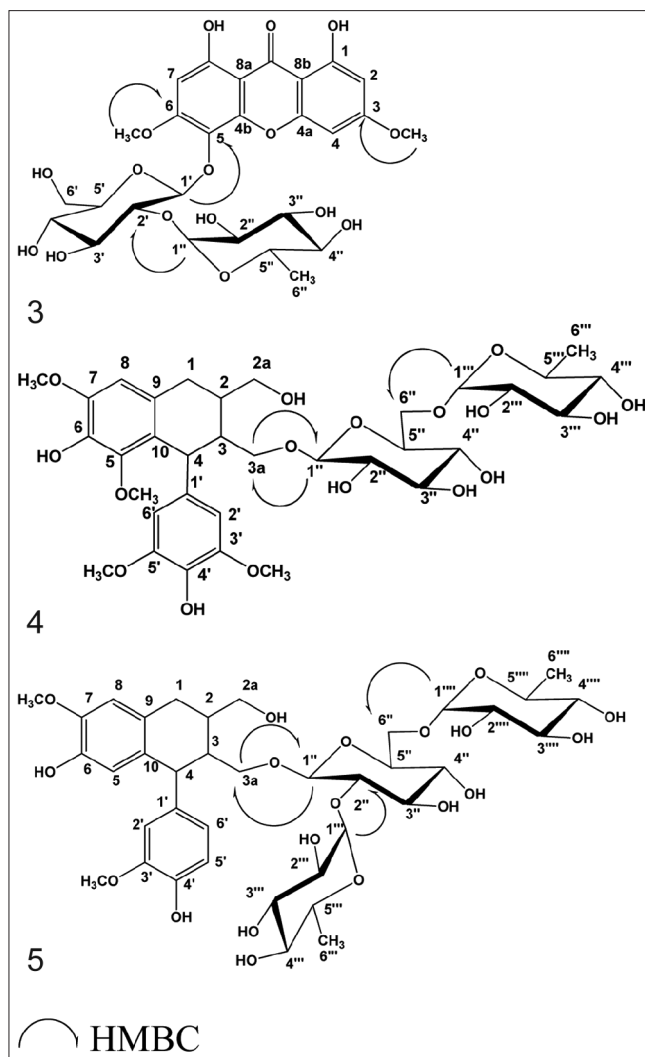


Figure 3: Significant HMBC correlations of compounds 3-5

$\delta_{\text{C}}$  76.38 belonging to C-2 of glucose.<sup>[28-32]</sup> Other  $^{13}\text{C-NMR}$  signals at  $\delta_{\text{C}}$  100.53, 70.41, 70.48, 71.85, 68.38 and 18.07 in addition to characteristic doublet methyl group at  $\delta_{\text{H}}$  1.19 (3H, d,  $J = 6$  Hz) in  $^1\text{H-NMR}$  were ascribed to rhamnosyl moiety.<sup>[28-32]</sup> The partial and complete acid hydrolysis of compound 3 in parallel with investigation of the hydrolysate by paper chromatography (system IV) revealed the presence of glucose and rhamnose sugars. On the other hand, HMBC measurements showed a significant correlation between the anomeric proton of glucose unit H-1' ( $\delta_{\text{H}}$  5.35) and C-5 ( $\delta_{\text{C}}$  128.40) of the aglycone part, supported our hypothesis about position of attachment of the sugar part to C-5 of the xanthone aglycone. It also showed an important correlation between anomeric proton of rhamnosyl moiety H-1'' ( $\delta_{\text{H}}$  5.17) and C-2' ( $\delta_{\text{C}}$  76.38) of glucose moiety, confirming the site of attachment of rhamnose to glucose at C-2'. Furthermore the HMBC experiment showed cross linkage between protons of two methoxy groups at C-3 ( $\delta_{\text{H}}$  3.77) and C-6 ( $\delta_{\text{H}}$  3.91) and carbons C-3 ( $\delta_{\text{C}}$  164.10) and C-6 ( $\delta_{\text{C}}$  160.10)

respectively, supporting the presence of 3,6-dimethoxylated xanthone moiety. From the previously mentioned chromatographic, spectral and results of acid hydrolysis data, compound **3** was identified as 1,8-Dihydroxy-3,6-dimethoxy-xanthone-5-*O*-[ $\alpha$ -L-rhamnopyranosyl-(1" $\rightarrow$ 2')] - $\beta$ -D-glucopyranosid, a new natural product.

#### Compound 4

Its positive FAB-MS showed the quasi-molecular ion peak at  $m/z$  729  $[M+1]^+$ , together with  $^1\text{H}$ -,  $^{13}\text{C}$ -NMR spectral data confirmed the molecular formula to be  $\text{C}_{34}\text{H}_{48}\text{O}_{17}$ . The detailed analysis of  $^1\text{H}$  and  $^{13}\text{C}$ -NMR spectra of compound **4** deduced the occurrence of glycosylated (+)-lyoniresinol lignan moiety;<sup>[33,37]</sup> However the  $^1\text{H}$ -NMR spectrum of compound **4** displayed signals at  $\delta_{\text{H}}$  4.15 (1H, dd,  $J=11, 6$  Hz), 4.06 (1H, dd,  $J=11, 4.2$  Hz) assigned for primary alcoholic methylene protons H-3a, in addition to signal at  $\delta_{\text{H}}$  2.87 (2H, m) assigned to methylene protons, H-1. The signals appeared at  $\delta_{\text{H}}$  3.69 (3H, s), 3.75 (3H, s) and 3.63 (6H, s) could be assigned to four methoxy groups attached to C-5, C-7 and C-3', C-5' respectively.<sup>[33-37]</sup> Moreover,  $^1\text{H}$ -NMR showed the oxygenated methylene protons (H-2a) as a multiplet signal at  $\delta_{\text{H}}$  3.41 (2H, m). The aliphatic methine protons H-2, H-3 and H-4 appeared at  $\delta_{\text{H}}$  1.92 (1H, br.s), 2.71 (1H, m) and 4.26 (1H, d,  $J=6$  Hz) respectively while the aromatic protons appeared at  $\delta_{\text{H}}$  6.53, (1H, s, H-8) and 6.29 (2H, s, H-2' and H-6'). The  $^{13}\text{C}$ - NMR spectrum of compound **4** [Table 4] illustrated the presence of 34 carbon atoms, of them 12 carbons belonging to sugar moiety and the rest 22 carbons indicated an aryl-tetralin-type lignin.<sup>[33-37]</sup> Concerning the sugar moiety,  $^1\text{H}$ -NMR spectrum showed the presence of two anomeric protons, one of them appeared at  $\delta_{\text{H}}$  4.60 (1H, d,  $J = 7.6$  Hz), the other proton noticed at  $\delta_{\text{H}}$  6.08 (1H, s). The  $^1\text{H}$ -NMR data in conjunction with  $^{13}\text{C}$ -NMR signals at  $\delta_{\text{C}}$  103.87, 100.86, indicating the existence of two anomeric carbons, in addition to the other signals at  $\delta_{\text{C}}$  73.41, 76.80, 69.04, 77.29 and 64.04 pointed out the incidence of C-6" substituted glucose. This was further supported from C-6" downfield shifted signal at  $\delta_{\text{C}}$  64.04 in comparison with non-substituted glucose moiety.<sup>[33-37]</sup> The appearance of a characteristic doublet at  $\delta_{\text{H}}$  1.11 (3H, d,  $J= 6.5$  Hz) in addition to other carbon signals at  $\delta_{\text{C}}$  69.74, 70.86, 70.99, 69.55 and 17.99, indicated the presence of rhamnose moiety attached to C-6" of glucose. The sugar linkage at C-3a was determined mainly by the comparison of the  $^1\text{H}$ ,  $^{13}\text{C}$ -NMR data of compound **4** with (+)-lyoniresinol, where two protons at C-3a ( $\delta_{\text{H}}$  4.06, 4.15) of compound **4** showed obvious downfield shift signal upon comparison with corresponding protons at C-3a ( $\delta_{\text{H}}$  3.48) of (+)-lyoniresinol (about 0.6 ppm) and C-3a shifted from  $\delta_{\text{C}}$  64.20 to 70.75 when compared with (+)-lyoniresinol.<sup>[33,38]</sup> The optical rotation of Compound **4** is +9.6 (MeOH, c.05) compared with

(+)-lyoniresinol with optical rotation +13.3 (MeOH, c.32),<sup>[38]</sup> indicated that, compound **4** is (+)-lyoniresinol derivative. Investigation of the HMBC spectrum revealed the presence of significant correlations between; methylene protons H-3a ( $\delta_{\text{H}}$  4.15, 4.06) of the aglycone and C-1" ( $\delta_{\text{C}}$  103.87) of glucose moiety, confirming the position of sugar linkage at C-3a. The HMBC spectrum showed diagnostic correlation between H-1" ( $\delta_{\text{H}}$  6.08) of rhamnose and C-6" ( $\delta_{\text{C}}$  64.04) of glucose, confirming the attachment of rhamnose to C-6" of glucose unit. From the above chemical, chromatographic and spectral data,<sup>[33-38]</sup> compound **4** was identified as (+)-Lyoniresinol-3a-*O*-[ $\alpha$ -L-rhamnopyranosyl-(1" $\rightarrow$ 6")] - $\beta$ -D-glucopyranoside,<sup>[33-38]</sup> a new natural product.

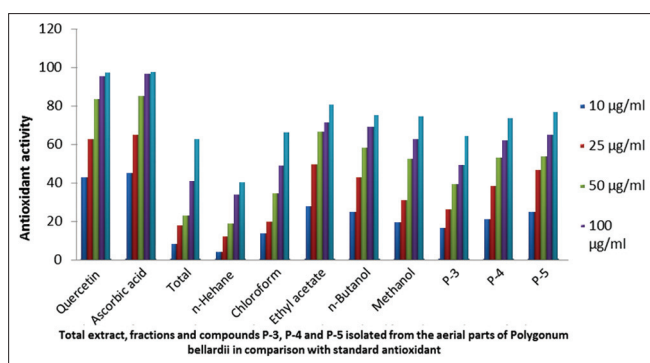
#### Compound 5

The positive FAB-MS spectrum showed the quasi-molecular ion peak at  $m/z$  815  $[M+1]^+$ , together with  $^1\text{H}$ -,  $^{13}\text{C}$ -NMR spectral data confirmed the molecular formula to be  $\text{C}_{38}\text{H}_{54}\text{O}_{19}$ . The  $^1\text{H}$ -NMR together with  $^{13}\text{C}$ - NMR spectral data illustrated the presence of 38-carbon atoms, among them, 18 carbons for two aromatic rings and six aliphatic carbon exhibiting the existence of an aryl-tetralin-type lignan, could be identified as (+)-isolariciresinol.<sup>[34-37]</sup> Remaining 18 carbons concluded the presence of substituted glucose moiety. The  $^1\text{H}$ -NMR of compound **5** displayed signals at  $\delta_{\text{H}}$  3.62 (1H, dd,  $J = 10.8, 4.5$ ) and 3.65 (1H, dd,  $J = 10.8, 6.1$ ), H-2a;  $\delta_{\text{H}}$  3.21 (2H, m), H-1;  $\delta_{\text{H}}$  3.70 and 3.67 each (3H, s), two methoxy groups at C-7 and C-3' respectively and  $\delta_{\text{H}}$  3.73 (1H, dd,  $J= 9.9, 3$  Hz), 3.75 (1H, dd,  $J= 9.9, 4.6$  Hz), H-3a. Moreover, it revealed the presence of the following signals: At  $\delta_{\text{H}}$  1.84 (1H, m, H-2), 2.72 (1H, br.s, H-3), 4.42 (1H, d,  $J= 5.8$  Hz, H-4), 6.56 (1H, s, H-5), 6.54 (1H, s, H-8), 6.49 (1H, d,  $J=7.9, 2.1$  Hz, H-6'), 6.66 (1H, d,  $J=7.9$  Hz, H-5') and 6.54 (1H, d,  $J=2.1$  Hz, H-2'). The anomeric protons appeared at  $\delta_{\text{H}}$  4.18 (1H, d,  $J= 7.9$  Hz) and 6.07 (2H, s). The  $^1\text{H}$ -NMR data in association with  $^{13}\text{C}$ -NMR signals at  $\delta_{\text{C}}$  102.24, 101.92 and 101.22, indicating the occurrence of three anomeric carbons, in addition to the other  $^{13}\text{C}$ -NMR signals at  $\delta_{\text{C}}$  74.91, 77.91, 68.01, 78.23 and 65.36 declared the presence of C-2" and C-6" substituted glucose moiety.<sup>[37-40]</sup> This was further supported from C-2", C-6" downfield shift signals at  $\delta_{\text{C}}$  74.91 and 65.36 accordingly upon comparison with non-substituted glucose moiety.<sup>[38]</sup> The two doublet signals at  $\delta_{\text{H}}$  1.09 and 1.19 each (3H, d,  $J= 6.5$  Hz), in addition to other  $^1\text{H}$  and  $^{13}\text{C}$ -NMR sugar signals [Tables 3 and 4] indicated the existence of two rhamnose moieties attached to C-2" and C-6" of glucose. The linkage of sugar was configured at C-3a mainly by the comparison of the  $^1\text{H}$  and  $^{13}\text{C}$ -NMR data of compound **5**, with those reported for (+)-isolariciresinol,

**Table 5: Antioxidant activity of total extract, fractions and isolated compounds (3-5) from the aerial parts of *Polygonum bellardii* All**

Fraction or Compound	Concentrations $\mu\text{g/ml}$					IC <sub>50</sub>
	10	25	50	100	200	
Ascorbic acid	47.1±2.03%	66.3±1.79%	86.9±3.12%	98.8±1.54%	99.6±3.10%	14.5±2.92
Quercetin	45.0±2.95%	65.0±2.88%	85.0±3.62%	97.3±0.91%	99.1±3.22%	15.1±2.25
Total extract	10.0±0.97%	19.8±1.24%	25.0±1.07%	42.9±2.14%	64.8±1.09%	135.4±2.05
<i>n</i> -Hexane F.	05.8±0.45%	13.8±1.21%	20.8±1.13%	35.9±3.14%	41.9±1.45%	253.4±1.91
Chloroform F.	15.8±3.20%	21.8±1.65%	36.8±1.88%	50.9±2.98%	67.9±0.78%	98.1±3.02
Ethyl acetate F.	29.9±3.12%	51.9±3.10%	68.2±3.51%	73.3±1.56%	82.5±3.21%	20.51±1.87
<i>n</i> -Butanol F.	26.8±2.66%	44.9±2.88%	60.1±2.43%	71.1±3.32%	77.3±1.88%	34.7±2.32
Compound 3*	18.2±1.56%	28.4±1.93%	41.1±0.99%	51.0±0.98%	66.1±0.87%	79.3±2.65
Compound 4**	22.8±1.99%	40.6±2.79%	54.8±1.76%	63.6±2.11%	75.3±3.08%	38.2±1.85
Compound 5***	26.4±2.11%	48.5±3.01%	55.6±3.13%	67.3±3.09%	78.6±2.31%	35.4±2.46

\*3: 1,8-Dihydroxy-3,6-dimethoxy-xanthone-5-O-[ $\alpha$ -L-rhamnopyranosyl-(1<sup>'''</sup>→2'')]- $\beta$ -D-glucopyranoside, \*\*4: (+)-Lyoniresinol-3a-O-[ $\alpha$ -L-rhamnopyranosyl-(1<sup>'''</sup>→6'')]- $\beta$ -D-glucopyranoside, \*\*\*5: (+)-Isolariciresinol-3a-O-[ $\alpha$ -L-rhamnopyranosyl-(1<sup>'''</sup>→2'')]- $\alpha$ -L-rhamnopyranosyl-(1<sup>'''</sup>→6'')]- $\beta$ -D-glucopyranoside



**Figure 4:** Antioxidant activity of the total extract, fractions and compounds 3, 4 and 5 isolated from the aerial parts of *Polygonum bellardii* All

however the methylene protons H-3a ( $\delta_{\text{H}}$  3.73, 3.75) was obviously downfield shifted.<sup>[39,40]</sup> The optical rotation of Compound 5 is +11 (MeOH, c 0.05) compared with (+)-isolariciresinol +34 (MeOH, c 0.10),<sup>[40]</sup> indicated that, compound 5 is (+)-isolariciresinol derivative. HMBC correlations showed significant cross linkage between the protons at C-3a ( $\delta_{\text{H}}$  3.73, 3.75) of the aglycone and anomeric carbon of glucose moiety C-1<sup>'''</sup> ( $\delta_{\text{C}}$  102.24), confirming the attachment of sugar part at C-3a. The inter-glycosidic linkages between glucose and two rhamnose units were diagnosed from HMBC correlations between the two anomeric protons of rhamnose units H-1<sup>'''</sup>, H-1<sup>''''</sup> ( $\delta_{\text{H}}$  6.07) and C-2<sup>''</sup> ( $\delta_{\text{C}}$  74.91) together with C-6<sup>''</sup> ( $\delta_{\text{C}}$  65.36) of glucose moiety which confirmed the site of attachment of rhamnose units to C-2<sup>''</sup> and 6<sup>''</sup> of glucose. From the above chemical, chromatographic and spectral data,<sup>[33-40]</sup> it could be concluded that compound 5 was identified as (+)-Isolariciresinol-3a-O-[ $\alpha$ -L-rhamnopyranosyl-(1<sup>'''</sup>→2'')]- $\alpha$ -L-rhamnopyranosyl-(1<sup>'''</sup>→6'')]- $\beta$ -D-glucopyranoside, a new natural product.

### Results of radical scavenging activity

The obtained results revealed that compounds 4, 5 in

addition to ethyl acetate, *n*-butanol and methanol extracts displayed the strongest radical scavenging activity, while the weakest radical scavenging activity was observed with *n*-hexane extract. The radical scavenging activities of the extracts were correlated to the presence of high concentrations of flavonoids and phenolic compounds in the extracts which were isolated from *Polygonum bellardii* in our previous work.<sup>[10]</sup> The pure compounds showed lower activity than that of crude extracts (ethyl acetate and *n*-butanol) may be due to synergism (synchronization) of high flavonoid content in the plant (18.52 mg/kg) and other phenolic constituents present in crude extracts of the plant [Table 5, Figure 4].

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