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Short communication

Stilbene glucosides from the bulbs of *Iris tingitana*

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ABSTRACT

Two new dimeric stilbene glucosides, tingitanol A (**1**) and tingitanol B (**2**) together with *trans*-resveratrol 3-*O*-glucopyranoside (**3**) in addition to three known isoflavones, 5-*O*-methylgenistein (**4**), 5-*O*-methylgenistein 7-*O*-β-D-glucopyranoside (**5**) and betavulgarin (**6**) have been isolated for the first time from the fresh bulbs of *Iris tingitana* Boiss. & Reut. Their structures were established on the basis of the spectral data and direct comparison with values from previously identified analogues. Additionally, the isolated compounds (**1**–**6**) were evaluated for the free radical scavenging activity.

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1. Introduction

Iridaceae is a family of more than 60 genera and 800 species and most abundant in both tropical and temperate regions. The genus *Iris* belongs to this family and comprises about 300 species (Evans, 2002). *Iris* species have an immense medicinal importance and are used in the treatment of biliousness with liver dysfunction (Evans, 2002), cancer, inflammations in addition to bacterial and viral infections (Atta-ur-Rahmann et al., 2003). Also, they are used as antispasmodic (Atta-ur-Rahmann et al., 2003), emetic, laxative (Seki et al., 1994), antidote and haemostatic agents (Seki et al., 1995). Furthermore, they exhibited significant anthelmintic activity against gastrointestinal nematodes of sheep (Tariq et al., 2008) and molluscicidal activity (Singab et al., 2006). The compounds isolated from these species were reported to have anti-tumor (Atta-ur-Rahmann et al., 2003; Farag et al., 2009), piscicidal, antioxidant, antituberculousis, anti-inflammatory (Atta-ur-Rahmann et al., 2003), antiplasmodial and antifungal (Benoit-Vical et al., 2003) properties.

Phytochemical investigations of various *Iris* species showed the presence of flavones, isoflavones (Ali et al., 1983; Farag et al., 1999; Choudhary et al., 2001a), flavanones (Kohma et al., 1997), xanthones (Fujita and Inoue, 1982; Al-Khalil et al., 1995), iridals (Miyake et al., 1997; Taillet et al., 1999; Marner et al., 2002), stilbenes (Keckeis et al., 2000; Wang et al., 2003), peltogynoids (Choudhary et al., 2001b), quinones (Marner and Horper, 1992; Seki et al., 1995) and triterpenes (Seki et al., 1994; Ito et al., 1995). The previous phytochemical study of *Iris tingitana* Boiss. & Reut. fresh bulbs led to the isolation of isoflavonoids and acetovanillone (El-Emary et al., 1980). In this paper, two new dimeric stilbene glucosides, tingitanol A (**1**) and tingitanol B (**2**) together with four known compounds, *trans*-resveratrol 3-*O*-glucopyranoside (**3**), 5-*O*-methylgenistein (**4**), 5-*O*-methylgenistein 7-*O*-β-D-glucopyranoside (**5**) and betavulgarin (**6**) were isolated from the fresh bulbs of *I. tingitana* Boiss. & Reut. Herein, we report the isolation and structural elucidation of the new compounds. Additionally, we have determined the free radical scavenging properties of these compounds.

2. Results and discussion

The methanolic extract from *I. tingitana* Boiss. & Reut. fresh bulbs was partitioned with *n*-hexane, $CHCl_3$, EtOAc, and *n*-BuOH, successively. The *n*-BuOH, EtOAc, and $CHCl_3$ soluble fractions were separated by a combination of chromatographic procedures to yield two new dimeric stilbene glucosides, tingitanol A (**1**) and tingitanol B (**2**) along with four known compounds. The known compounds were identified as *trans*-resveratrol 3-*O*-glucopyranoside (**3**) (Orsini et al., 1997), 5-*O*-methylgenistein (**4**) (Tahara et al., 1986), 5-*O*-methylgenistein 7-*O*-β-D-glucopyranoside (**5**) (De Rodriguez et al., 1990), and betavulgarin (**6**) (Jong and Hwang, 1995), by the comparison of their spectral data with those reported in the literature.

Compound **1** was obtained as brown powder. It was found to have the molecular formula $C_{40}H_{42}O_{16}$ determined by HRFABMS. Its ^{1}H NMR spectrum (Table 1) exhibited signals due to two 1,4-

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Table 1
^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) data for **1** and **2** in DMSO-d_6.

Position	**1**		**2**	
	δ_H	δ_C	δ_H	δ_C
1a		131.5 s		132.0 s
2a	7.15 d (8.7)	127.0 d	7.17 d (8.6)	127.2 d
3a	6.76 d (8.7)	115.3 d	6.75 d (8.6)	115.5 d
4a		157.4 s		157.5 s
5a	6.76 d (8.7)	115.3 d	6.75 d (8.6)	115.5 d
6a	7.15 d (8.7)	127.0 d	7.17 d (8.6)	127.2 d
7a	5.42 d (4.8)	92.4 d	5.50 d (4.9)	93.1 d
8a	4.58 d (4.8)	54.6 d	4.75 d (4.9)	55.7 d
9a		145.7 s		145.9 s
10a	6.30 br s	107.8 d	6.19 br s	107.8 d
11a		158.9 s		158.8 s
12a	6.35 br s	101.7 d	6.31 br s	101.7 d
13a		158.7 s		158.8 s
14a	6.28 br s	106.8 d	6.28 br s	106.9 d
1b		131.5 s		131.8 s
2b	7.16 d (8.7)	127.9 d	8.93 d (2.0)	112.1 d
3b	6.70 d (8.7)	115.5 d		156.2 s
4b		157.5 s	6.99 dd (8.6, 2.0)	115.5 d
5b	6.70 d (8.7)	115.5 d	7.64 d (8.6)	129.9 d
6b	7.16 d (8.7)	127.9 d		124.7 s
7b	6.98 d (16.4)	129.9 d	7.50 d (8.6)	128.8 d
8b	6.60 d (16.4)	121.3 d	7.02 d (8.6)	119.2 d
9b		134.9 s		130.9 s
10b		120.8 s		114.3 s
11b		160.5 s		158.3 s
12b	6.53 d (2.0)	96.6 d	7.11 s	95.8 d
13b		158.9 s		158.1 s
14b	6.94 d (2.0)	104.3 d		115.6 s
1′	4.72 d (7.7)	100.5 d	4.71 d (8.8)	100.1 d
2′	3.16 m	73.2 d	3.77 m	73.1 d
3′	3.32 m	76.5 d	3.21 m	76.6 d
4′	3.16 m	69.5 d	3.15 m	69.6 d
5′	3.32 m	76.9 d	3.51 m	77.1 d
6′	3.77 m	60.5 t	3.77 m	60.7 t
	3.48 m		3.64 m	
1″	4.88 d (7.7)	100.8 d	5.27 d (8.1)	100.9 d
2″	3.29 m	73.3 d	3.15 m	73.2 d
3″	3.32 m	76.8 d	3.21 m	76.9 d
4″	3.20 m	70.0 d	3.31 m	69.9 d
5″	3.40 m	77.2 d	3.51 m	77.3 d
6″	3.64 m	60.9 t	3.77 m	60.9 t
	3.49 m		3.64 m	

disubstituted benzene rings [δ_H 6.70 and 7.16 (each 2H, d, J = 8.7 Hz), 6.76 and 7.15 (each 2H, d, J = 8.7 Hz)]; one 1,3,5-trisubstituted benzene ring [δ_H 6.28, 6.30 and 6.35 (each 1H, br s)]; one 1,2,3,5-tetrasubstituted benzene ring [δ_H 6.53 and 6.94 (each 1H, d, J = 2.0 Hz)]; two *trans*-olefinic protons [δ_H 6.60 and 6.98 (each 1H, d, J = 16.4 Hz)]. The aliphatic hydrogen signals at δ 4.58 and 5.42 (each 1H, d, J = 4.8 Hz) suggested the presence of one dihydrobenzofuran moiety bearing 4-oxyphenyl and 3,5-dioxyphenyl groups characteristic of oligostilbenes derived from resveratrol molecule (Yan et al., 2001). The coupling constant $J_{7a,\ 8a}$ = 4.8 Hz suggested *trans* configuration of H-7a and H-8a (Ito and Niwa, 1996; Wang et al., 2003). These data are in good agreement with the published data for (+)-ε-viniferin (Ito et al., 1999; Yan et al., 2001). In addition, the ^{1}H NMR spectrum showed two anomeric proton signals at δ 4.71 (1H, d, J = 8.8 Hz) and 5.27 (1H, d, J = 8.1 Hz) indicated the presence of two β-sugar units (Agrawal, 1992). The ^{13}C NMR and DEPT ^{13}C spectra (Table 1) revealed the presence of 12 carbon signals for two β-glucopyranosyl units (Agrawal, 1992) along with 28 carbon signals including 17 methine and 11 quaternary carbons due to the stilbenoid moieties. The HMBC spectrum showed long-range correlations at H-1′/C-13b; H-1″/C-11a; H-5a/C-1a and C-4a; H-6a/C-4a and C-7a; H-7a/C-6a, C-9a, C-10b and C-11b; H-8a/C-1a, C-7a, C-9a, C-10b and C-11b; H-10a/C-8a, C-11a, C-12a, C-14a; H-12a/C-11a and C-14a; H-14a/C-10a; H-5b/C-1b and C-4b; H-6b/C-7b and C-4b; H-7b/C-6b and C-9b; H-8b/C-10b and C-14b; H-12b/C-13b and C-14b; H-14b/C-12b and C-13b. From this evidence, the structure of (+)-tingitanol A was characterized as **1** (Fig. 1).

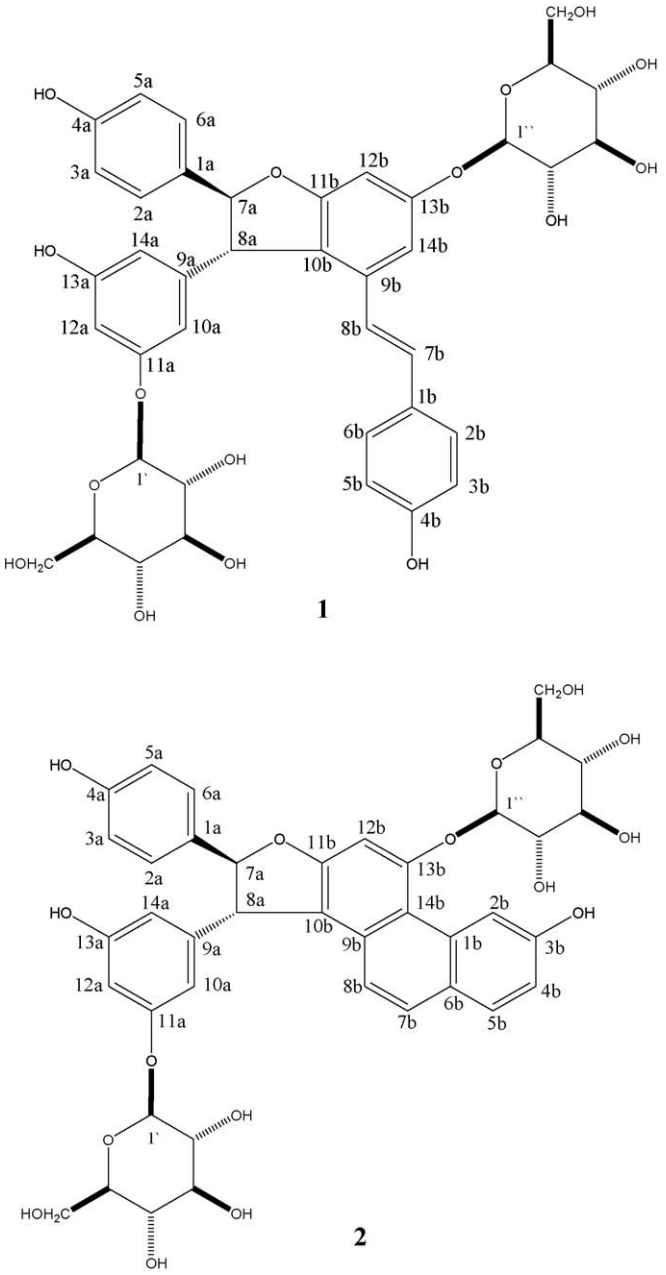

Fig. 1. Structures of compounds **1** and **2**.

Compound **2** was obtained as yellowish brown powder. Its molecular formula was determined to be $C_{40}H_{40}O_{16}$ by HRFABMS. The ^{1}H NMR spectral data (Table 1) are very close to those of **1** except for the replacement of one 1,4-disubstituted benzene ring, the 1,2,3,5-tetrasubstituted benzene ring and the *trans*-olefinic protons with one 1,2,4,6-tetrasubstituted phenanthrene nucleus [δ_H 6.99 (1H, dd, J = 8.6, 2.0 Hz), 7.02 (1H, d, J = 8.6 Hz), 7.11 (1H, s), 7.50 (1H, d, J = 8.6 Hz), 7.64 (1H, d, J = 8.6 Hz) and 8.93 (1H, d, J = 2.0 Hz)] (Baderschneider and Winterhalter, 2000; Wang et al., 2003). The ^{13}C and DEPT ^{13}C NMR spectra (Table 1) displayed 12 signals for two β-glucopyranosyl units (Agrawal, 1992) in addition to 28 carbon signals including 15 methine and 13 quaternary carbons for the stilbenoid moieties. Complete assignment was

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