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A new iridoid glycoside from the root of *Psychotria rubra*



Hai-Xiao Lu^{a,*}, Li-Ye Liu^a, Dian-Peng Li^b, Jia-Zhou Li^a, Lan-Cheng Xu^a

^a School of Life Science and Technology, Yulin Normal University, Yulin 537000, PR China

^b Guangxi Key Laboratory of Functional Phytochemicals Research and Utilization, Guangxi Institute of Botany, Guangxi Zhuang Autonomous Region and Chinese Academy of Sciences, Guilin 541006, PR China

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ABSTRACT

Phytochemical investigation on the root of *Psychotria rubra* led to the isolation of two iridoid glycosides (**1**, **4**), two flavones (**2**–**3**). To our knowledge, all compounds were isolated from this plant for the first time. Compound **1** (psyrubrin A) was a new compound, and compounds **2**–**4** were reported for the first time from the genus *Psychotria*. The chemotaxonomic significances of these compounds were summarized.

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1. Subject and source

The genus *Psychotria* (Rubiaceae) comprises about 1600 species worldwide (Davis et al., 2009) and is mainly distributed in tropical and subtropical regions, and also the largest genus in family Rubiaceae (Taylor, 1996). There are ca. 17 species and 1 varieties growing in southwest and east China (Zhang and Wu, 1999). The root of *Psychotria rubra* was collected in Yulin City, Guangxi autonomous region, China and identified by Professor Liu ShouYang, Guangxi University of Chinese Medicine. The voucher specimen (No. 201307001) was deposited in the Laboratory of Phytochemistry, Department of Pharmaceutical Engineering, School of Life Science and Technology, Yulin Normal University.

2. Previous work

Psychotria species have been extensively investigated and are well-known to be rich sources of the secondary metabolites type, including iridoids, tryptamine-iridoid alkaloids, and alkaloids of polyindoline type. The tryptamine-iridoid alkaloids were considered to be main phytochemical taxonomic characterized of *Psychotria* by Lopes et al. (2004). Previous phytochemical investigation on the ground stem of *P. rubra* has reported the presence of four constituents, i.e., helenalin, psychorubrin, psychorubin acetate and ¹H-naphtho[2,3-c]pyran-5,10-dione (Hayashi et al., 1987).

3. Present work

The air-dried root of *P. rubra* (5 kg) was macerated with 95% EtOH three times (6 L for each extraction) at room temperature. The filtrate was evaporated *in vacuo* to produce a residue (1818.2 g), which was suspended in H₂O, and then

* Corresponding author. Tel.: +86 15907758504.

E-mail address: luhaixiao76@163.com (H.-X. Lu).

partitioned successively with petroleum ether (PE), ethyl acetate (EtOAc), n-butanol (n-BuOH) to provide PE-soluble, EtOAc-soluble, n-BuOH-soluble and H₂O-soluble portions, respectively.

The H₂O-soluble portion (173.1 g) was fractionated by a D101 macroporous resin column chromatography eluted successively with methanol (MeOH) and H₂O, to give two fractions (frs. W1-2). Fr. W2 (23.5 g) was separated by silica gel column chromatography (CC) eluted with a gradient of CHCl₃–MeOH (30:1 → 1:1) to give sixteen fractions (frs. 1–16). Fr. 3 (0.194 g) was repeatedly chromatographed by sephadex LH-20 eluted by CHCl₃–MeOH (1:1) to afford **3** (18.0 mg). Fr. 7 (1.57 g) was repeatedly chromatographed by CC over silica gel eluted by CHCl₃–MeOH (4:1) to afford **1** (6.6 mg) and **2** (102.5 mg). Fr. 10 (0.328 g) was repeatedly chromatographed by sephadex LH-20 eluted by CHCl₃–MeOH (1:1) to afford **4** (22.0 mg).

By comparison of their ¹H, ¹³C NMR and ESI-MS spectral data with those reported in literatures, the known compounds **2–4** (Fig. 1) were identified as 6-hydroxy-luteolin-7-O- rutinoside (**2**) (Zhou et al., 2008), luteolin-7-O-rutinoside (**3**) (Zhou et al., 2008), 6 α -hydroxygeniposide (**4**) (Andreas et al., 2011).

The new compound **1** was isolated as a white amorphous powder. The molecular formula C₁₉H₂₈O₁₂ was determined from the negative-mode HR-ESI-MS (*m/z* 431.4113 ([M–H][–], calcd for 431.4111)), indicating six degrees of unsaturation. The UV spectrum displays absorptions at $\lambda_{\max}^{\text{MeOH}}$ nm: 221(2.22). The IR spectrum suggested C=C group (1635.19 cm^{–1}), carboxylic ester group (1735.00, 1158.98, 1074.00 cm^{–1}), and =C–O–C group (1247.8, 1043.91 cm^{–1}) in **1**. On the basis of the characteristic signals of two CH groups (δ (C) 45.5, 41.6), five O-bearing CH groups (δ (C) 77.5, 76.9, 73.9, 73.8, 70.4), two hemiketal quaternary C-atom (δ (C) 99.6, 99.4), a carbonyl C-atom (δ (C) 170.6), four alkenyl C-atom (δ (C) 143.1, 132.3, 143.1, 128.2), three O-bearing CH₂ groups (δ (C) 62.5, 61.7, 61.5), two Me groups (δ (C) 21.1, 15.6). We assume that **1** should be an iridoid glycoside, similar to asperulosidic acid (Andreas et al., 2011). Comparison with the ¹H and ¹³C NMR data of asperulosidic acid indicated that **1** (Table 1) was absent a carbonyl group and an additional ethoxy group substituent at C-4. The above conclusion was further confirmed by the degrees of unsaturation, the HMBC and the NOESY correlations (Fig. 2) of **1**. Therefore, **1** is identified

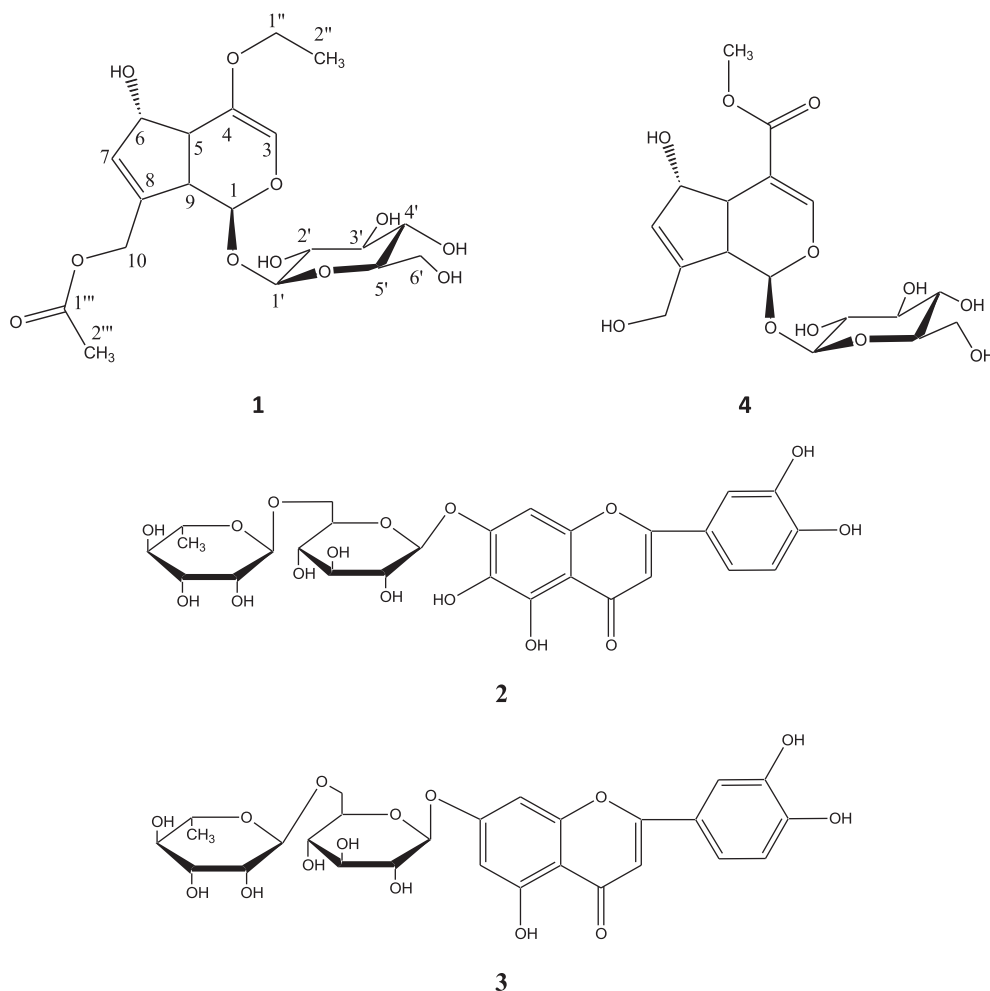


Fig. 1. Chemical structures of compounds 1–4.

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