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Spirostanol saponins from *Ypsilandra parviflora* induce platelet aggregation

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ABSTRACT

Phytochemical investigation on the whole plants of *Ypsilandra parviflora* led to the isolation of seven new spirostanol saponins, named ypsiparosides A–G, together with 14 known saponins. Their structures were unambiguously established based on extensive spectroscopic evidence and chemical methods. The induced rabbit platelet aggregation activities of the isolates were tested. Compounds **4**, **15**, and **17** showed maximal platelet aggregation rates ranging from 43 to 55% at a concentration of 300 μg/mL. Further experiments exhibited that compounds **4**, **15**, and **17** possessed EC_{50} values of 642.9, 95.3, and 300.8 μg/mL, respectively.

1. Introduction

Plants of genus *Ypsilandra* (Liliaceae) containing five species are mainly distributed in Myanmar and southwest China, of which four are present in China [1]. They have been used in traditional Chinese medicine (TCM) for the treatments of scrofula, urination, edema, uterine bleeding, and traumatic hemorrhage [2]. Previous studies on this genus have led to the isolation of a series of steroidal saponins responsible for diverse bioactivities, such as cytotoxic, antifungal, hemostatic, and anti-HIV effects [3–8].

Y. parviflora, an erect herb, widely grows in mountain slopes and streams at the altitude between 1000 and 1400 m in Guizhou, Hunan, Guangxi, and Guangdong provinces of China [1]. However, the phytochemicals and the biological activities of *Y. parviflora* have not been reported so far. Our bioassay showed that the 70% EtOH extract of *Y. parviflora* showed 67.5% maximal rabbit platelet aggregation rate at a concentration of 1.5 mg/mL. In order to clarify it's bioactive constituents, seven new spirostanol saponins, ypsiparosides A–G (**1**–**7**) (Fig. 1), and 14 known analogues (**8**–**21**) were isolated from the 70% EtOH extract of the whole plants of title species. The known saponins were identified as ypsilandroside C (**8**) [4], ypsilandroside D (**9**) [4], pennogenin-3-*O*-*α*-L-rhamnopyranosyl-(1→4)-*α*-L-rhamnopyranosyl-(1→4)-*β*-D-glucopyranoside (**10**) [9], paris saponin VII (**11**) [10], polyphylloside III [11] [10], taccasuboside B (**13**) [12], isoypsilandroside B (**14**) [3], paris saponin II (**15**) [13], ypsilandroside G (**16**) [4], ypsilandroside M (**17**) [14], ypsilandroside K (**18**) [5], parispseudoside A (**19**) [15], proto-Pb (**20**) [16], and pseudoproto Pb (**21**) [17] by comparison of their spectroscopic data with those reported in the literature. The current paper reports the isolation, structural elucidation, and the induced platelet aggregation activities of these isolates.

2. Experimental

2.1. General experimental procedures

Optical rotations were measured on a JASCO P-1020 digital polarimeter. UV spectra were measured using a Shimadzu UV-2401 PC spectrophotometer. IR spectra were obtained on Bruker Tensor-27 infrared spectrophotometer with KBr pellets. ESI-MS spectra were recorded on a Bruker HTC/Esquire spectrometer, HRESIMS spectra were recorded on an API Qstar Pulsar instrument. NMR experiments were performed on Bruker AM-400, DRX-500, and Avance III 600 instruments with TMS as the an internal standard. Chemical shifts (δ) are expressed in ppm with reference to the solvent signals. Column

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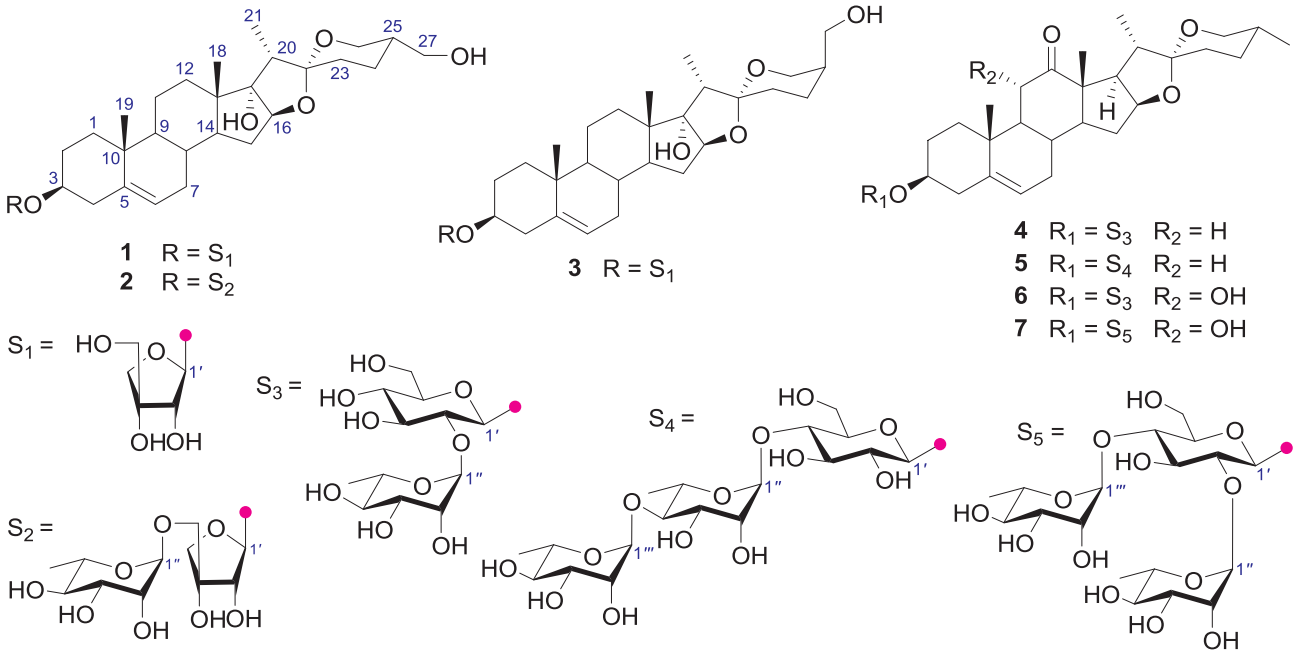

Fig. 1. Chemical structures of compounds **1–7**.

chromatography (CC) was performed on silica gel (100–200, 200–300, and 300–400 mesh, Qingdao Marine Chemical Co, China), RP-18 (40–63 μm, Merck), and Sephadex LH-20 (GE Healthcare, Sweden). Semi-preparative HPLC was run on Agilent 1100 liquid chromatograph with diode array detector (DAD), Zorbax-SB-C18 column (5 μm; 25 cm × 9.4 mm i.d.). TLC was performed on $HSGF_{254}$ (0.2 mm, Qingdao Marine Chemical Co, China) or RP-18 F_{254} (0.25 mm, Merck). Fractions were monitored by TLC and spots were visualized by heating silica gel plates sprayed with 10% H_2SO_4 in EtOH.

2.2. Plant material

The whole plants of *Y. parviflora* were collected in August 2012, from Leishan County, Guizhou Province, China, and identified by Dr. Rong Li of Kunming Institute of Botany, CAS. A voucher specimen (No. HY0020) was deposited at State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, CAS.

2.3. Extraction and isolation

Dried and powdered plant materials of *Y. parviflora* (4.5 kg) were extracted three times with 70% EtOH (20 L × 3) under reflux for a total of 6 h (3 × 2 h) and the combined extract was concentrated under reduced pressure. The crude extract was partitioned between *n*-BuOH and water to afford *n*-BuOH soluble portion (666 g). Then, the concentrated *n*-BuOH-soluble portion was subjected to CC (silica gel, 200-300 mesh; gradient $CHCl_3$-MeOH 20:1→0:1, v/v) to afford six fractions: A–F (60 g, 40.5 g, 40 g, 82 g, 210 g, and 105 g, respectively). Fr. B was passed through an MCI gel column and eluted with EtOH-H_2O gradient (40:60→90:10, v/v) followed by silica gel CC ($CHCl_3$-MeOH, 25:1→1:1, v/v) to give **13** (8 mg). Fr. D was purified by RP-18 gel (MeOH-H_2O, 40:60→90:10, v/v), repeated silica gel CC ($CHCl_3$-MeOH, 15:1→1:1, v/v), and finally purified by semi-preparative HPLC (MeCN-H_2O, 40:60 v/v; flow rate: 3.0 mL/min) to give **1** (11 mg), **2** (20 mg), **3** (12 mg), **4** (20 mg), **6** (15 mg), **7** (10 mg), and **10** (10 mg). Fr. E was fractionated by a silica gel column ($CHCl_3$–MeOH, 10:1→1:1, v/v) to give two fractions: Fr. 5-1 (80 g) and Fr. 5-2 (70 g). Fr. 5-1 was separated by an RP-18 column (MeOH–H_2O, 45:55→100:0, v/v) and further purified by semi-preparative HPLC (MeCN-H_2O, 40:60 v/v; flow rate: 3.0 mL/min) to afford **5** (8 mg), **6** (30 mg), and **9** (181 mg). Fr. 5-2 was subjected to repeated silica gel CC ($CHCl_3$-MeOH, 10:1→1:1, v/v) and separated by semi-preparative HPLC (MeCN-H_2O, 40:60 v/v; flow rate: 3.0 mL/min) and afforded **11** (15 mg), **15** (20 mg), **16** (23 mg), and **17** (13 mg). Fr. F was subjected to MCI gel column and eluted with aqueous EtOH (70% v/v), repeated silica gel CC ($CHCl_3$-MeOH, 8:1→1:1, v/v), and finally purified by semi-preparative HPLC (MeCN-H_2O, 30:70 v/v; flow rate: 3.0 mL/min) to yield **8** (33 mg), **12** (18 mg), **14** (20 mg), **18** (29 mg), **19** (50 mg), **20** (40 mg), and **21** (92 mg).

2.3.1. Ypsiparoside A (1)

White amorphous powder; $[\alpha]_D^{21}$ −163.7 (*c* 0.6, MeOH); IR (KBr) ν_{max} 3424, 2932, 1631, 1453, 1089, 1042, 910 cm^{-1}; ^1H NMR data see Table 1; ^{13}C NMR data see Table 2; positive ESIMS: *m/z* 601 $[M+Na]^+$; HRESIMS: *m/z* 601.3352 $[M+Na]^+$ (calcd for $C_{32}H_{50}O_9Na$, 601.3353).

2.3.2. Ypsiparoside B (2)

White amorphous powder; $[\alpha]_D^{21}$ −122.7 (*c* 0.6, MeOH); IR (KBr) ν_{max} 3426, 2932, 1640, 1453, 1383, 1135, 1052, 979 cm^{-1}; ^1H NMR data see Table 1; ^{13}C NMR data see Table 2; positive ESIMS: *m/z* 747 $[M+Na]^+$; HRESIMS: *m/z* 747.3923 $[M+Na]^+$ (calcd for $C_{38}H_{60}O_{13}Na$, 747.3932).

2.3.3. Ypsiparoside C (3)

White amorphous powder; $[\alpha]_D^{21}$ −141.2 (*c* 0.7, MeOH); IR (KBr) ν_{max} 3425, 2934, 2902, 1631, 1455, 1379, 1221, 1062, 919 cm^{-1}; ^1H NMR data see Table 1; ^{13}C NMR data see Table 2; positive ESIMS: *m/z* 601 $[M+Na]^+$; HRESIMS: *m/z* 601.3345 $[M+Na]^+$ (calcd for $C_{32}H_{50}O_9Na$, 601.3353).

2.3.4. Ypsiparoside D (4)

White amorphous powder; $[\alpha]_D^{21}$ −80.4 (*c* 0.7, MeOH); IR (KBr) ν_{max} 3428, 2930, 2909, 1707, 1637, 1454, 1381, 1048, 981, 919, 899, 868 cm^{-1} (intensity: 899 > 919 cm^{-1}); ^1H NMR data see Table 1; ^{13}C NMR data see Table 2; positive ESIMS: *m/z* 759 $[M+Na]^+$; HRESIMS: *m/z* 759.3929 $[M+Na]^+$ (calcd for $C_{39}H_{60}O_{13}Na$, 759.3932).

2.3.5. Ypsiparoside E (5)

White amorphous powder; $[\alpha]_D^{21}$ −62.5 (*c* 0.7, MeOH); IR (KBr) ν_{max} 3426, 2927, 2853, 1707, 1634, 1455, 1384, 1043, 981, 921, 900, 866 cm^{-1} (intensity: 900 > 921 cm^{-1}); ^1H NMR data see Table 1; ^{13}C

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