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Stilbene dimer xylosides and flavanols from the roots of *Lysidice rhodostegia* and their antioxidant activities

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ABSTRACT

Eight new stilbene dimer xylosides (**1**–**8**) and one new flavanol (**9**), along with seven known ones (**10**–**16**) were isolated from the roots of *Lysidice rhodostegia*. Their structures were elucidated by extensive analysis of spectroscopic data (IR, UV, HR-ESI-MS, 1D and 2D NMR), ECD calculations and acid hydrolysis. Compounds **1**–**16** were evaluated for their antioxidant activities using DPPH radical-scavenging assay. Especially, compounds **9** and **10** exhibited stronger antioxidant effects than the positive control (vitamin E), with IC_{50} values of 9.57 ± 1.30 and 13.60 ± 1.47 μM, respectively.

1. Introduction

Lysidice rhodostegia Hance (Fabaceae) is known as "Yihua" in China [1], and mainly distributes in south and southwest of China, including Guangdong, Guangxi and Yunnan Provinces [2]. The roots of L. *rhodostegia* are used as a traditional medicine for the treatment of hemorrhage (topically), rheumatic arthralgia (orally and topically), fracture (topically), *etc.* [1–3]. Modern pharmacological investigations demonstrated that the plants of the genus *Lysidice* possessed antioxidant [4–6], vasodilatatory [7], and antiarrhytmic activities [8]. Previous phytochemical studies on this plant showed that derivatives of phloroglucinols, stilbenes and flavonoids are the main bioactive components [9–11].

Previously, acylphloroglucinol glucosides were isolated from L. *rhodostegia* by our group, and the antioxidant activity of the compounds was investigated [4]. Further investigation of L. *rhodostegia* led to the isolation of eight new stilbene dimer xylosides (**1**–**8**) and one new flavanol (**9**) (Fig. 1), along with seven known ones, (2*R*,3*S*,4*R*)-2,3-trans-3,4-*trans*-4-(2,4,6-trihydroxyphenyl)-3′,4′,5,7-tetrahydroxyflavan-3-ol (**10**) [12], *trans*-resveratrol (**11**) [13], piceid (**12**) [14], 3-hydroxy-5-[(1*Z*)-2-(4-hydroxyphenyl)ethenyl] phenyl (**13**) [15], (*E*)-resveratrol-3-*O*-rutinoside (**14**) [16], lysidiside N (**15**) [16], lysidiside E (**16**) [17]. The isolation and structural elucidation of the new compounds are reported in this paper. Moreover, compounds **1**–**16** were evaluated for their antioxidant activities using the DPPH radical-scavenging assay. All tested isolates showed various levels of antioxidant activities, with IC_{50} values ranging from 9.57 ± 1.30 to 56.92 ± 1.46 μM. Especially, compounds **9** and **10** exhibited stronger antioxidant effects than the positive control (vitamin E), with IC_{50} values of 9.57 ± 1.30 and 13.60 ± 1.47 μM, respectively.

2. Experimental

2.1. General experimental procedures

Optical rotations were measured on a JASCO P-1020 polarimeter. ECD data were measured by a JASCO J-810 spectrometer. IR spectra were scanned using a JASCO FT/IR-480 plus FT-IR spectrometer with KBr pellets. UV spectra were obtained by a JASCO V-550 UV/VIS spectrophotometer. Thin-layer chromatography (TLC) was used on silica gel GF254 plates (Yantai Chemical Industry Research Institute, Yantai, China). Column chromatography (CC) was performed using silica gel

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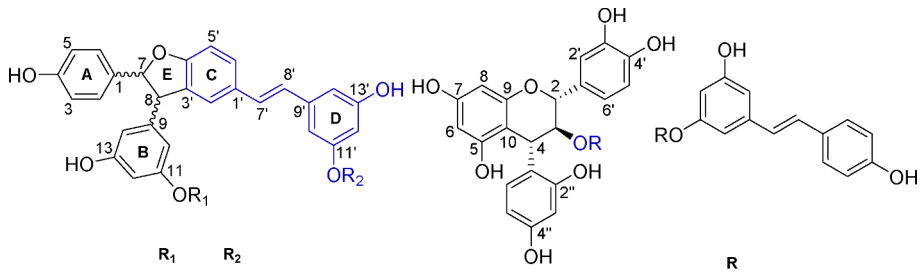

Fig. 1. Chemical structures of **1**–**16**.

(100–200 and 200–300 mesh, Qingdao Marine Chemical Plant, Qingdao, P. R. China), ODS (50 μm, YMC, Kyoto, Japan) and Sephadex LH-20 (Pharmacia Biotech, Uppsala, Sweden). HR-ESI-MS analyses were performed using an Agilent 6210 ESI/TOF mass spectrometer. Analytical HPLC was carried out on a Waters system (600E Multisolvent Delivery System, 2487 Dual λ Absorbance Detector) with a Cosmosil C18 analytical column (5 μm, 4.6 × 250 mm). Preparative HPLC was applied on an Agilent 1260 Chromatograph equipped with a G1311C pump and a G1315D photodiode array detector (Agilent Technologies, CA,USA) with a Cosmosil C18 preparative column (5 μm, 20 × 250 mm). NMR spectra were recorded on a Bruker AV-500 spectrometer with TMS as internal standard. The chemical shifts (δ) are expressed in ppm and coupling constants (J) in Hz. All the reagents were purchased from Tianjin Damao Chemical Company (Damao,Tianjin, China).

2.2. Plant materials

The roots of L. *rhodostegia* were collected from Jiangmen (E113.06^0, N22.61^0), Guangdong Province of China, in March of 2018, which were authenticated by Zhenqiu Mai, the senior engineer of Guangdong Province. A voucher specimen (20180322) was stored in the Institute of Traditional Chinese Medicine & Natural Products, Jinan University, Guangzhou, China.

2.3. Extraction and isolation

The dried and powdered roots (14.0 kg) of L. *rhodostegia* were extracted with 95% EtOH at room temperature. After the concentration of the combined ethanol extract, the residue (1.5 kg) was suspended in H_2O and then partitioned with PE, EtOAc and *n*-BuOH, respectively. The EtOAc extract (400 g) was subjected to a silica gel CC eluted with $CHCl_3$/CH_3OH (1:0 to 0:1, *v/v*) solvent system to afford five fractions (Fr. A-E). Fr. B (27.9 g) was separated by ODS CC, using the CH_3OH/H_2O (4:6 to 1:0, *v/v*) system to get five subfractions (Fr. B1-B5). Then, Fr. B2 (3.8 g) was separated by Sephadex LH-20 with CH_3OH to afford compounds **1** (4.8 mg), **2** (6.8 mg), **11** (10.5 mg) and **12** (12.3 mg). Compounds **3** (5.5 mg, t_R = 12.5 min), **4** (10.5 mg, t_R = 21.3 min), **13** (8.3 mg, t_R = 27.5 min) and **14** (11.2 mg, t_R = 30.1 min) were afforded from Fr. B3 (4.3 g) by the preparative HPLC using CH_3OH/H_2O (70:30, *v/v*, 7 mL/min). Fr. B4 (7.2 g) was further purified *via* preparative HPLC (CH_3OH/H_2O, 75:25, *v/v*, 7 mL/min) to obtain **5** (7.8 mg, t_R = 17.5 min), **6** (14.6 mg, t_R = 25.1 min), **15** (13.2 mg, t_R = 28.3 min) and **16** (14.1 mg, t_R = 32.1 min). Fr. B5 (5.1 g) was separated using Sephadex LH-20 with CH_3OH and then purified by preparative HPLC (CH_3OH/H_2O, 70:30, *v/v*, 7 mL/min) to obtained **7** (7.2 mg, t_R = 28.3 min), **8** (8.5 mg, t_R = 34.3 min), **9** (9.5 mg, t_R = 37.8 min) and **10** (11.4 mg, t_R = 40.2 min).

2.3.1. Lysidostegin A (1)

Brown oil; $[\alpha]_D^{25}$ +29.3 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 198, 285 nm; IR (KBr) ν_{max}: 3403, 2921, 2849, 1610, 1515, 1489, 1285, 1158, 984, 699 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 1; HR-ESI-MS *m/z* 609.1723 [M + Na]$^+$ (calcd for $C_{33}H_{30}NaO_{10}$, 609.1731).

2.3.2. Lysidostegin B (2)

Brown oil; $[\alpha]_D^{25}$ +14.5 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 197, 284 nm; IR (KBr) ν_{max}: 3415, 2928, 2842, 1604, 1515, 1489, 1292, 1147, 988. 715 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 1; HR-ESI-MS *m/z* 609.1720 [M + Na]$^+$ (calcd for $C_{33}H_{30}NaO_{10}$, 609.1731).

2.3.3. Lysidostegin C (3)

Brown oil; $[\alpha]_D^{25}$ +32.7 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 198, 206 nm; IR (KBr) ν_{max}: 3393, 1675, 1601, 1522, 1486, 1240, 1168, 832 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 2; HR-ESI-MS *m/z* 609.1733 [M + Na]$^+$ (calcd for $C_{33}H_{30}NaO_{10}$, 609.1731).

2.3.4. Lysidostegin D (4)

Brown oil; $[\alpha]_D^{25}$ +18.6 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 198, 206 nm; IR (KBr) ν_{max}: 3390, 1680, 1595, 1528, 1483, 1240, 1045, 847 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 2; HR-ESI-MS *m/z* 609.1722 [M + Na]$^+$ (calcd for $C_{33}H_{30}NaO_{10}$, 609.1731).

2.3.5. Lysidostegin E (5)

Brown oil; $[\alpha]_D^{25}$ +35.2 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 197, 310 nm; IR (KBr) ν_{max}: 3361, 2920, 1678, 1601, 1450, 1207, 1042, 835 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 3; HR-ESI-MS *m/z* 741.2153 [M + Na]$^+$ (calcd for $C_{38}H_{38}NaO_{14}$, 741.2154).

2.3.6. Lysidostegin F (6)

Brown oil; $[\alpha]_D^{25}$ +16.4 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 199, 310 nm; IR (KBr) ν_{max}: 3375, 2927, 1688, 1605, 1487, 1235, 1179, 1043, 836 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 3; HR-ESI-MS *m/z* 741.2141 [M + Na]$^+$ (calcd for $C_{38}H_{38}NaO_{14}$, 741.2154).

2.3.7. Lysidostegin G (7)

Brown oil; $[\alpha]_D^{25}$ +31.9 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 197, 308 nm; IR (KBr) ν_{max}: 3334, 1583, 1512, 1447, 1206, 1172, 1015, 962, 837, 678 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 4; HR-ESI-MS *m/z* 771.2272 [M + Na]$^+$ (calcd for $C_{39}H_{40}O_{15}Na$, 771.2259).

2.3.8. Lysidostegin H (8)

Brown oil; $[\alpha]_D^{25}$ +22.9 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 198, 308 nm; IR (KBr) ν_{max}: 3331, 2929, 1598, 1514, 1444, 1314, 1173, 1074, 959, 835, 675 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 4; HR-ESI-MS *m/z* 771.2256 [M + Na]$^+$ (calcd for $C_{39}H_{40}O_{15}Na$, 771.2259).

Table 1
NMR data of **1** and **2** (in CD_3OD, 600 MHz for ^1H, δ in ppm, J in Hz).

Position	**1**		**2**		^1H–^1H COSY	HMBC
	δ_H	δ_C	δ_H	δ_C		
1	–	132.4, C	–	132.7, C	–	–
2	7.19 (d, 8.6)	128.7, CH	7.19 (d, 8.5)	128.7, CH	H-3	C-4, 6
3	6.81 (d, 8.6)	116.4, CH	6.82 (d, 8.5)	116.4, CH	H-2	C-1, 5
4	–	158.8, C	–	158.8, C	–	–
5	6.81 (d, 8.6)	116.4, CH	6.82 (d, 8.5)	116.4, CH	H-6	C-1, 3
6	7.19 (d, 8.6)	128.7, CH	7.19 (d, 8.5)	128.7, CH	H-5	C-2, 4
7	5.39 (d, 9.0)	94.9, CH	5.37 (d, 9.0)	94.8, CH	H-8	C-2, 6, 9
8	4.50 (d, 9.0)	58.7, CH	4.47 (d, 9.0)	58.7, CH	H-7	C-1, 10, 14
9	–	145.4, C	–	145.3, C	–	–
10	6.33 (t, 1.8)	110.3, CH	6.33 (t, 1.8)	110.3, CH	–	C-12, 14
11	–	160.0, C	–	160.0, C	–	–
12	6.47 (t, 1.8)	103.7, CH	6.46 (t, 1.8)	104.0, CH	–	C-10, 14
13	–	160.2, C	–	160.4, C	–	–
14	6.38 (t, 1.8)	108.9, CH	6.36 (t, 1.8)	109.1, CH	–	C-10, 12
1′	–	141.1, C	–	141.1, C	–	–
2′	7.21 (br s)	124.1, CH	7.19 (br s)	124.1, CH	–	C-8, 4′, 6′, 7′
3′	–	132.1, C	–	132.1, C	–	–
4′	–	161.0, C	–	161.0, C	–	–
5′	6.89 (d, 8.2)	110.4, CH	6.82 (d, 8.3)	110.4, CH	H-6′	C-1′, 3′
6′	7.41 (d, 8.2)	128.8, CH	7.39 (d, 8.3)	128.9, CH	H-5′	C-2′, 4′
7′	7.01 (d, 16.0)	129.3, CH	7.00 (d, 16.0)	129.3, CH	H-8′	C-2′, 6′, 9′
8′	6.79 (d, 16.0)	127.6, CH	6.78 (d, 16.0)	127.6, CH	H-7′	C-1′, 10′, 14′
9′	–	132.4, C	–	132.4, C	–	–
10′	6.45 (d, 2.0)	105.8, CH	6.43 (d, 2.0)	105.8, CH	–	C-8′, 12′, 14′
11′	–	159.7, C	–	159.6, C	–	–
12′	6.17 (d, 2.0)	102.7, CH	6.14 (d, 2.0)	103.0, CH	–	C-10′, 14′
13′	–	159.7, C	–	159.6, C	–	–
14′	6.45 (d, 2.0)	105.8, CH	6.43 (d, 2.0)	105.8, CH	–	C-8′, 10′, 12′
1″	4.82 (d, 7.5)	102.4, CH	4.74 (d, 7.5)	102.8, CH	H-2″	C-11, 3″
2″	3.39–3.43 (m)	74.6, CH	3.35–3.39 (m)	74.6, CH	H-1″, 3″	C-3″
3″	3.29–3.31 (m)	77.7, CH	3.31–3.34 (m)	77.7, CH	H-2″, 4″	C-1″, 5″
4″	3.54–3.58 (m)	71.0, CH	3.50–3.55 (m)	71.0, CH	H-3″, 5″	C-2″
5″	3.85–3.80 (m) 3.26–3.30 (m)	66.9, CH_2	3.85–3.89 (m) 3.21–3.28 (m)	66.9, CH_2	H-4″	C-3″

Table 2
NMR data of **3** and **4** (in DMSO-d_6, 600 MHz for ^1H, δ in ppm, J in Hz).

Position	**3**		**4**		^1H–^1H COSY	HMBC
	δ_H	δ_C	δ_H	δ_C		
1	–	130.3, C	–	130.3, C	–	–
2	7.18 (d, 8.6)	127.9, CH	7.19 (d, 8.6)	127.9, CH	H-3	C-4, 6
3	6.75 (d, 8.6)	115.3, CH	6.75 (d, 8.6)	115.3, CH	H-2	C-1, 5
4	–	157.6, C	–	157.6, C	–	–
5	6.75 (d, 8.6)	115.3, CH	6.75 (d, 8.6)	115.3, CH	H-6	C-1, 3
6	7.18 (d, 8.6)	127.9, CH	7.19 (d, 8.6)	127.9, CH	H-5	C-2, 4
7	5.38 (d, 9.0)	92.7, CH	5.37 (d, 9.0)	92.7, CH	H-8	C-2, 6, 9
8	4.44 (d, 9.0)	55.7, CH	4.47 (d, 9.0)	55.7, CH	H-7	C-1, 10, 14
9	–	143.7, C	–	143.7, C	–	–
10	6.03 (t, 1.8)	106.0, CH	6.03 (t, 1.8)	106.0, CH	–	C-12, 14
11	–	158.7, C	–	158.7, C	–	–
12	6.29 (t, 1.8)	102.9, CH	6.30 (t, 1.8)	102.9, CH	–	C-10, 14
13	–	158.7, C	–	158.7, C	–	–
14	6.03 (t, 1.8)	106.0, CH	6.03 (t, 1.8)	106.0, CH	–	C-10, 12
1′	–	139.3, C	–	139.3, C	–	–
2′	7.24 (br s)	123.0, CH	7.19 (br s)	123.0, CH	–	C-8, 4′, 6′, 7′
3′	–	131.3, C	–	131.3, C	–	–
4′	–	159.0, C	–	159.0, C	–	–
5′	6.89 (d, 8.3)	109.4, CH	6.82 (d, 8.4)	109.4, CH	H-6′	C-1′, 3′
6′	7.41 (d, 8.3)	127.8, CH	7.44 (d, 8.4)	127.8, CH	H-5′	C-2′, 4′
7′	7.09 (d, 16.0)	128.1, CH	7.09 (d, 16.0)	128.1, CH	H-8′	C-2′, 6′, 9′
8′	6.88 (d, 16.0)	125.0, CH	6.89 (d, 16.0)	125.9, CH	H-7′	C-1′, 10′, 14′
9′	–	130.3, C	–	130.3, C	–	–
10′	6.63 (t, 2.0)	105.0, CH	6.43 (t, 2.0)	105.0, CH	–	C-8′, 12′, 14′
11′	–	158.6, C	–	158.6, C	–	–
12′	6.09 (t, 2.0)	101.4, CH	6.10 (t, 2.0)	101.4, CH	–	C-10′, 14′
13′	–	158.5, C	–	158.5, C	–	–
14′	6.57 (t, 2.0)	107.1, CH	6. 58 (t, 2.0)	107.1, CH	–	C-8′, 10′, 12′
1″	4.82 (d, 7.2)	100.8, CH	4.81 (d, 7.2)	100.8, CH	H-2″	C-11′, 3″
2″	3.15–3.19 (m)	73.4, CH	3.14–3.18 (m)	73.1, CH	H-1″, 3″	C-3″
3″	3.20–3.24 (m)	76.5, CH	3.19–3.23 (m)	76.5, CH	H-2″, 4″	C-1″, 5″
4″	3.31–3.36 (m)	69.3, CH	3.35–3.39 (m)	69.4, CH	H-3″, 5″	C-2″
5″	3.69–3.72 (m) 3.24–3.27 (m)	65.7, CH_2	3.69–3.73 (m) 3.25–3.29 (m)	65.7, CH_2	H-4″	C-3″

Table 3
NMR data of **5** and **6** (in DMSO-d_6, 600 MHz for ^{1}H, δ in ppm, J in Hz).

Position	5		6		^{1}H–^{1}H COSY	HMBC
	δ_H	δ_C	δ_H	δ_C		
1	–	130.1, C	–	130.1, C	–	–
2	7.19 (d, 8.6)	127.9, CH	7.18 (d, 8.6)	127.8, CH	H-3	C-4, 6
3	6.76 (d, 8.6)	115.3, CH	6.76 (d, 8.6)	115.3, CH	H-2	C-1, 5
4	–	157.7, C	–	157.7, C	–	–
5	6.76 (d, 8.6)	115.3, CH	6.76 (d, 8.6)	115.3, CH	H-6	C-1, 3
6	7.19 (d, 8.6)	127.9, CH	7.18 (d, 8.6)	127.8, CH	H-5	C-2, 4
7	5.40 (d, 9.0)	92.5, CH	5.44 (d, 9.0)	92.4, CH	H-8	C-2, 6, 9
8	4.50 (d, 9.0)	55.5, CH	4.52 (d, 9.0)	55.5, CH	H-7	C-1, 10, 14
9	–	143.7, C	–	143.7, C	–	–
10	6.24 (t, 1.6)	107.4, CH	6.24 (t, 1.6)	107.1, CH	–	C-12, 14
11	–	158.6, C	–	158.5, C	–	–
12	6.36 (t, 1.6)	102.1, CH	6.30 (t, 1.6)	102.0, CH	–	C-10, 14
13	–	158.7, C	–	158.6, C	–	–
14	6.25 (t, 1.6)	108.5, CH	6.25 (t, 1.6)	108.5, CH	–	C-10, 12
1′	–	139.2, C	–	139.2, C	–	–
2′	7.26 (br s)	123.0, CH	7.24 (br s)	123.0, CH	–	C-8, 4′, 6′, 7′
3′	–	131.0, C	–	131.1, C	–	–
4′	–	159.0, C	–	158.9, C	–	–
5′	6.91 (d, 8.6)	109.4, CH	6.90 (d, 8.6)	109.4, CH	H-6′	C-1′, 3′
6′	7.46 (d, 8.6)	127.8, CH	7.44 (d, 8.4)	127.8, CH	H-5′	C-2′, 4′
7′	7.08 (d, 16.0)	128.4, CH	7.09 (d, 16.0)	128.4, CH	H-8′	C-2′, 6′, 9′
8′	6.91 (d, 16.0)	125.9, CH	6.90 (d, 16.0)	125.9, CH	H-7′	C-1′, 10′, 14′
9′	–	130.3, C	–	131.1, C	–	–
10′	6.59 (t, 2.0)	107.1, CH	6.63 (t, 2.0)	107.0, CH	–	C-8′, 12′, 14′
11′	–	158.4, C	–	158.4, C	–	–
12′	6.30 (t, 2.0)	102.9, CH	6.31 (t, 2.0)	103.0, CH	–	C-10′, 14′
13′	–	158.5, C	–	158.5, C	–	–
14′	6.64 (t, 2.0)	104.9, CH	6. 58 (t, 2.0)	105.0, CH	–	C-8′, 10′, 12′
1″	4.75 (d, 7.0)	101.1, CH	4.81 (d, 7.2)	100.9, CH	H-2″	C-11, 3″
2″	3.15–3.20 (m)	73.1, CH	3.14–3.18 (m)	73.1, CH	H-1″, 3″	C-3″
3″	3.20–3,23 (m)	76.5, CH	3.19–3.23 (m)	76.5, CH	H-2″, 4″	C-1″, 5″
4″	3.33–3.37 (m)	69.3, CH	3.35–3.39 (m)	69.4, CH	H-3″, 5″	C-2″
5″	3.70–3.74 (m) 3.24–3.38 (m)	65.7, CH_2	3.69–3.73 (m) 3.25–3.29 (m)	65.7, CH_2	H-4″	C-3″
1‴	4.81 (d, 7.0)	100.8, CH	4.79 (d, 7.0)	100.6, CH	H-2‴	C-11′, 3‴
2‴	3.15–3.20 (m)	73.0, CH	3.14–3.18 (m)	73.0, CH	H-1‴, 3‴	C-3‴
3‴	3.20–3.22 (m)	76.3, CH	3.19–3.23 (m)	76.4, CH	H-2‴, 4‴	C-1‴, 5‴
4‴	3.33–3.37 (m)	69.4, CH	3.35–3.39 (m)	69.3, CH	H-3‴, 5‴	C-2‴
5‴	3.70–3.73 (m) 3.24–3.28 (m)	65.7, CH_2	3.69–3.73 (m) 3.25–3.29 (m)	65.6, CH_2	H-4‴	C-3‴

2.3.9. Lysidostegin I (9)

Yellow powder; $[\alpha]_D^{25}$ +15.5 (*c* 1.0, MeOH); UV (MeOH) λ_{max}: 200, 279 nm; IR (KBr) ν_{max}: 3308, 1625, 1506, 1385, 1290, 1110, 1018, 829 cm^{-1}; ^{1}H and ^{13}C NMR data, see Table 5; HR-ESI-MS *m/z* 413.1213 $[M + H]^+$ (calcd for $C_{22}H_{21}O_8$, 413.1231).

2.4. Determination of the absolute configurations of sugars

The methods of acid hydrolysis and HPLC analysis were used to confirm the absolute configurations of sugars in compounds **1**–**8** [18]. The compound (3 mg) were placed into 10 mL of HCl (2 M), and the mixture was heated under reflux in a 90 °C water bath for 6 h. Then, the solution was extracted with CH_2Cl_2 three times. After combination of the CH_2Cl_2 extracts, the solvent was evaporated, and the residue was reacted with anhydrous pyridine, L-cysteine methyl ester hydrochloride and 2-methylphenyl isothiocyanate at 60 °C for 1 h. Finally, the reaction mixtures were subjected to HPLC analysis using CH_3CN-H_2O (20:80, 1 mL/min). As shown in Fig. S2, S13, S24, S35, S46 and S57 (Supporting Information), all of the reaction mixtures of compounds **1**–**6** displayed peaks at the same retention times as the D-xylose derivative standard (t_R = 19.4 min, Fig. S1, Supporting Information), determining that the xylose moieties in compounds **1**–**6** are D-configured. The retention times of the sugar derivatives in compound **7**/**8** were 16.5 and 19.4 min (Figs. S68 and S79, Supporting Information), demonstrating that the sugar derivatives in compound **7**/**8** were D-glucose (t_R = 16.5 min, Fig. S1, Supporting Information) and D-xylose, respectively.

2.5. Antioxidant activity

Compounds **1**–**16** were tested for their antioxidant activities using the DPPH method [18], with the vitamin E as a positive control. The assay was conducted in a 96-well format using serial dilutions of 100 μL aliquots of tested compounds (ranging from 1000 to 31.25 μM) and vitamin E (1000–31.25 μM), respectively. Then, the absorbance was measured at 515 nm after 30 min in the dark. Three replicate wells were set in parallel for every group. Methanol was used as a negative control. The DPPH scavenging capacity (SC) was calculated according to the following formula: SC% = [(A0-A1/A0)] × 100%. A0 is the absorbance value of the control group; A1 is the absorbance value of the sample group.

3. Results and discussion

Compound **1** was isolated as brown oil with $[\alpha]_D^{25}$ +29.3 (*c* 1.0, MeOH). Its molecular formula was deduced as $C_{33}H_{30}O_{10}$ on the basis of its ^{13}C NMR and HR-ESI-MS (*m/z* 609.1723 $[M + Na]^+$, calcd for $C_{33}H_{30}NaO_{10}$, 609.1731) data. The IR spectrum suggested the presence of a hydroxy group (3403 cm^{-1}) and an aromatic ring (1601 cm^{-1}). The ^{1}H NMR spectrum (Table 1) showed characteristic signals for an aromatic ABX spin system [δ_H 7.41 (1H, d, J = 8.2 Hz), 7.21 (1H, br s), 6.89 (1H, d, J = 8.2 Hz)], an aromatic AA′XX′ spin system [δ_H 7.19 (2H, d, J = 8.6 Hz), 6.81 (2H, d, J = 8.6 Hz)], an aromatic ABC spin system [δ_H 6.47

Table 4
NMR data of **7** and **8** (in DMSO-d_6, 600 MHz for ^{1}H, δ in ppm, J in Hz).

Position	**7**		**8**		^{1}H–^{1}H COSY	HMBC
	δ_H	δ_C	δ_H	δ_C		
1	–	130.1, C	–	130.1, C	–	–
2	7.19 (d, 8.6)	127.9, CH	7.19 (d, 8.6)	127.7, CH	H-3	C-4, 6
3	6.76 (d, 8.6)	115.3, CH	6.77 (d, 8.6)	115.3, CH	H-2	C-1, 5
4	–	157.7, C	–	157.7, C	–	–
5	6.77 (d, 8.6)	115.3, CH	6.77 (d, 8.6)	115.3, CH	H-6	C-1, 3
6	7.19 (d, 8.6)	127.9, CH	7.19 (d, 8.6)	127.7, CH	H-5	C-2, 4
7	5.46 (d, 9.0)	92.3, CH	5.46 (d, 9.0)	92.4, CH	H-8	C-2, 6, 9
8	4.52 (d, 9.0)	55.6, CH	4.52 (d, 9.0)	55.6, CH	H-7	C-1, 10, 14
9	–	143.6, C	–	143.8, C	–	–
10	6.28 (t, 1.6)	107.1, CH	6.28 (t, 1.6)	107.1, CH	–	C-12, 14
11	–	158.7, C	–	158.6, C	–	–
12	6.38 (t, 1.6)	102.0, CH	6.38 (t, 1.6)	101.9, CH	–	C-10, 14
13	–	158.8, C	–	158.8, C	–	–
14	6.23 (t, 1.6)	108.3, CH	6.23 (t, 1.6)	108.4, CH	–	C-10, 12
1′	–	139.2, C	–	139.2, C	–	–
2′	7.25 (br s)	123.0, CH	7.25 (br s)	123.0, CH	–	C-8, 4′, 6′, 7′
3′	–	131.2, C	–	131.1, C	–	–
4′	–	158.9, C	–	158.9, C	–	–
5′	6.91 (d, 8.6)	109.4, CH	6.91 (d, 8.6)	109.3, CH	H-6′	C-1′, 3′
6′	7.45 (d, 8.6)	127.8, CH	7.45 (d, 8.6)	127.7, CH	H-5′	C-2′, 4′
7′	7.08 (d, 16.0)	128.4, CH	7.08 (d, 16.0)	128.4, CH	H-8′	C-2′, 6′, 9′
8′	6.91 (d, 16.0)	125.9, CH	6.91 (d, 16.0)	125.9, CH	H-7′	C-1′, 10′, 14′
9′	–	130.3, C	–	130.3, C	–	–
10′	6.58 (t, 2.0)	107.1, CH	6.58 (t, 2.0)	107.1, CH	–	C-8′, 12′, 14′
11′	–	158.4, C	–	158.4, C	–	–
12′	6.30 (t, 2.0)	102.9, CH	6.30 (t, 2.0)	103.0, CH	–	C-10′, 14′
13′	–	158.5, C	–		–	–
14′	6.64 (t, 2.0)	104.9, CH	6.64 (t, 2.0)	158.5, C	–	C-8′, 10′, 12′
1″	4.74 (d, 7.4)	100.1, CH	4.78 (d, 7.4)	105.0, CH	H-2″	C-11, 3″
2″	3.15–3.18 (m)	73.2, CH	3.15–3.18 (m)	99.9, CH	H-1″, 3″	C-3″
3″	3.19–3.21 (m)	77.0, CH	3.19–3.21 (m)	73.1, CH	H-2″, 4″	C-1″, 5″
4″	3.12–3.15 (m)	69.6, CH	3.12–3.15 (m)	76.8, CH	H-3″, 5″	C-2″
5″	3.21–3.24 (m)	76.6, CH	3.21–3.24 (m)	69.4, CH	H-4″, 6″	C-3″
6″	3.45–3.48 (m)	60.6, CH_2	3.45–3.48 (m)	76.6, CH	H-5″	C-4″
1‴	4.81 (d, 7.0)	100.8, CH	4.81 (d, 7.0)	60.4, CH_2	H-2‴	C-11′, 3‴
2‴	3.15–3.20 (m)	73.0, CH	3.15–3.20 (m)	100.9, CH	H-1‴, 3‴	C-3‴
3‴	3.20–3.23 (m)	76.5, CH	3.20–3.23 (m)	73.1, CH	H-2‴, 4‴	C-1‴, 5‴
4‴	3.31–3.38 (m)	69.4, CH	3.31–3.38 (m)	76.5, CH	H-3‴, 5‴	C-2‴
5‴	3.70–3.74 (m) 3.24–3.28 (m)	65.7, CH_2	3.70–3.74 (m) 3.24–3.28 (m)	69.4, CH 65.7, CH_2	H-4‴	C-3‴

Table 5
NMR data of **9** (600 MHz for ^{1}H in DMSO-d_6, ^{13}C in CD_3OD, δ in ppm, J in Hz).

Position	δ_H	δ_C	^{1}H–^{1}H COSY	HMBC
2	4.49 (d, 9.5)	83.4, CH	H-3	C-4
3	4.22 (t, 9.5)	81.0, CH	H-2, H-4	C-10, 1′
4	4.41 (d, 9.5)	40.5, CH	H-3	C-2, 5, 9, 2′, 6′
5	–	158.4, C	–	–
6	5.70 (d, 1.6)	96.4, CH	–	C-8, 10
7	–	159.2, C	–	–
8	5.87 (d, 1.6)	95.3, CH	–	C-6, 10
9	–	157.8, C	–	–
10	–	107.8, C	–	–
1′	–	132.6, C	–	–
2′	6.85 (d, 1.2)	116.0, CH	–	C-4′, 6′
3′	–	146.0, C	–	–
4′	–	146.3, C	–	–
5′	6.72 (d, 8.4)	160.0, CH	H-6′	C-1′, 3′
6′	6.69 (dd, 8.4, 1.2)	120.8, CH	H-5′	C-2′, 4′
1″	–	119.3, C	–	–
2″	4.18 (d, 6.5)	156.7, CH	–	–
3″	6.10 (d, 2.4)	103.2, CH	–	C-1″, 5″
4″	–	156.2, C	–	–
5″	6.15 (dd, 8.2, 2.4)	109.4, CH	H-6‴	C-1″, 3″
6″	6.38 (d, 8.2)	129.7, CH	H-5‴	C-2″, 4″
3-OCH_3	2.68 (s)	59.6, CH_3	–	C-3, 4

(1H, t, J = 1.8 Hz), 6.38 (1H, t, J = 1.8 Hz), 6.33 (1H, t, J = 1.8 Hz)], an aromatic AB_2 spin system [δ_H 6.45 (2H, d, J = 2.0 Hz), 6.17 (1H, d, J = 2.0 Hz)], a *trans* olefinic proton system [δ_H 7.01 (1H, d, J = 16.0 Hz), 6.79 (1H, d, J = 16.0 Hz)], two methines [δ_H 5.39 (1H, d, J = 9.0 Hz), 4.50 (1H, d, J = 9.0 Hz)], and an anomeric proton [δ_H 4.82 (1H, d, J = 7.5 Hz)]. The analysis of its ^{13}C NMR data (Table 1) revealed the presence of 33 carbon resonances, including 12 quaternary carbons (7 oxygenated), 20 methines (4 oxygenated), and 1 methylene. The NMR data of compound **1** resembled those of resveratrol (*E*)-dehydrodimer-11-*O*-*β*-D-glucopyranoside [19], except for the absence of a glucose unit and the presence of a xylose unit (δ_C 102.4, 77.7, 74.6, 71.0, 66.9) in **1**. The differences indicated the glucose unit in resveratrol (*E*)-dehydrodimer-11-*O*-*β*-D-glucopyranoside was replaced by a xylose unit in **1**. Moreover, the type of sugar residue was also confirmed by the acid hydrolysis of **1** (Figs. S1 and S2, Supporting Information). The HMBC cross-peak from H-1″ (δ_H 4.82) to C-11 (δ_C 160.0) indicated that the D-xylose moiety was connected to C-11 (Fig. 2). The $^3J_{H1\text{-}H2}$ coupling constant demonstrated *β*-configuration for the xylosyl bond ($^3J_{H1''\text{-}H2''}$ = 7.5 Hz). Moreover, the NOE correlations between H-7 (δ_H 5.39) and H-10 (δ_H 6.33)/H-14 (δ_H 6.38) and between H-8 (δ_H 4.50) and H-2 (δ_H 7.19)/H-6 (δ_H 7.19) indicated that H-7 and H-8 were on the opposite side (Fig. 3). The absolute configuration of **1** was confirmed by quantum chemical ECD calculations. The ECD spectra for (7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*)-**1** and its enantiomer were performed using the TDDFT-ECD method, and were calculated at the B3LYP/6–31 + G(d) level in the gas phase. The experimental ECD spectrum of **1** exhibited a negative cotton effect at 213 nm ($\Delta\varepsilon$ − 13.2) and a positive cotton effect at 235 nm ($\Delta\varepsilon$ + 4.7), which were similar to those of the calculated ECD

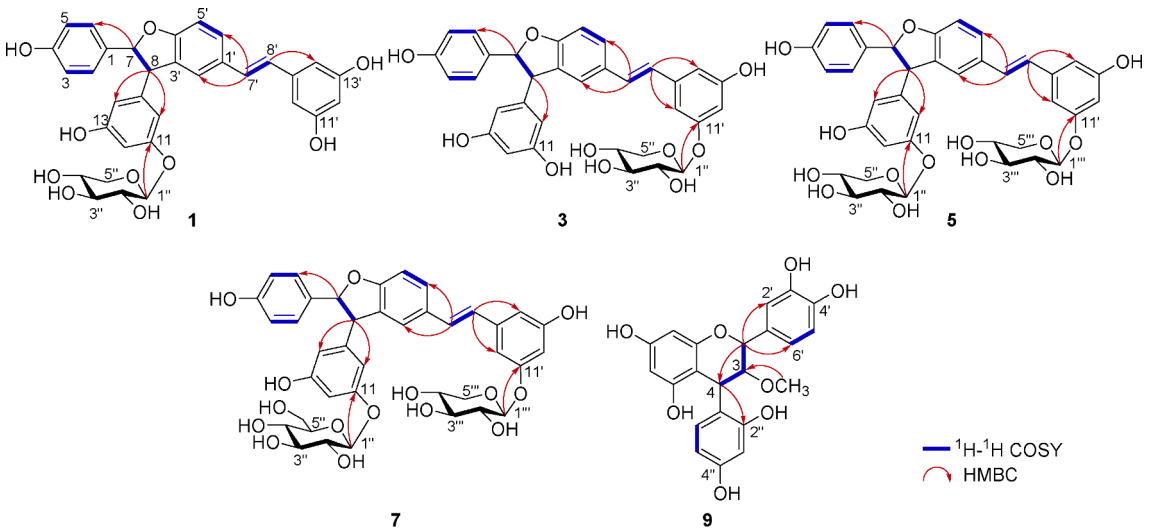

Fig. 2. Key ^{1}H—^{1}H COSY and HMBC correlations of **1**, **3**, **5**, **7** and **9**.

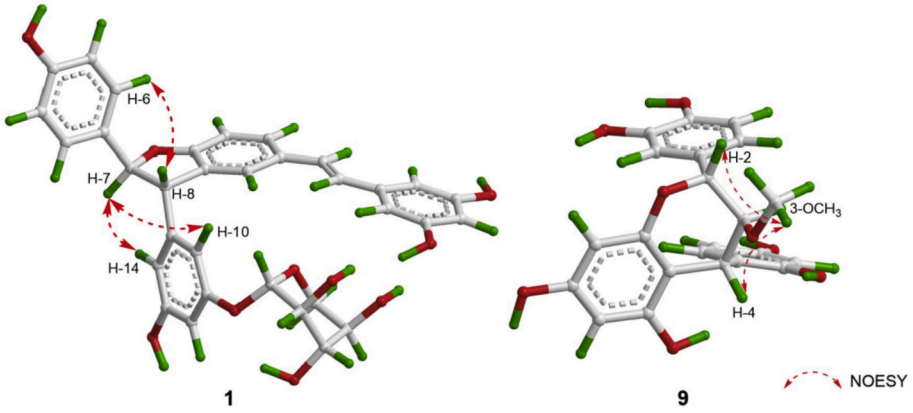

Fig. 3. Key NOESY correlations of **1** and **9**.

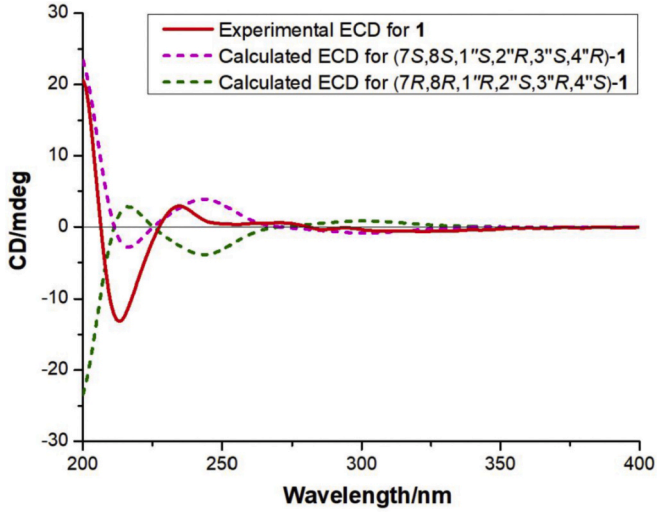

Fig. 4. Experimental and calculated ECD spectra of compound **1**.

spectrum for (7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*)-**1** (Fig. 4). Hence, the absolute configuration of **1** was elucidated as 7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*. Based on the above analysis, the structure was elucidated as shown and named lysidostegin A.

Compound **2** was isolated as brown oil with $[\alpha]_D^{25}$ +14.5 (*c* 1.0, $CHCl_3$). The HR-ESI-MS of compound **2** showed an $[M + Na]^+$ ion peak at *m/z* 609.1720 (calcd for $C_{33}H_{30}NaO_{10}$, 609.1731), consistent with the molecular formula of $C_{33}H_{30}O_{10}$. The NMR spectroscopic data (Table 1) of **2** was almost identical to those of **1**, which indicated that **2** possessed the same planar structure as that of **1**. The *β*-configuration of the xylosyl bond in **2** was determined by the anomeric proton signal at δ_H 4.74 (1H, d, $J = 7.5$ Hz). Analysis of the NOESY spectrum, H-7 and H-8 were also located on the opposite side, indicating that the relative configuration of **2** was same as that of **1**. Interestingly, compounds **1** and **2** had the same NMR data, but showed different retention times in HPLC analysis. And the rotation data of **1** (+29.3) and **2** (+14.5) were all positive, indicating they were not a pair of enantiomers. These informations implied that compounds **1** and **2** could be a pair of diastereoisomers with the oppositely absolute configurations of C-7 and C-8. Furthermore, the absolute configuration of **2** was established by quantum-chemical ECD calculation. The predicted ECD spectra of **2**, (7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*)-**2**, and its enantiomer, were compared with the experimental spectra, indicating the absolute configuration of **2** was 7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R* (Fig. S100, Supporting Information). Accordingly, compound **2** was elucidated and named lysidostegin B.

Compound **3** had the molecular formula $C_{33}H_{30}O_{10}$ assigned by HR-ESI-MS (*m/z* 609.1733 $[M + Na]^+$, calcd for $C_{33}H_{30}NaO_{10}$, 609.1731). According to its 1D and 2D NMR spectra (Table 2), compound **3** has the same stilbene dimer skeleton as that of **1**, with the ^{1}H—^{1}H COSY correlations between H-2 (δ_H 7.18) and H-3 (δ_H 6.75), between H-5 (δ_H

6.75) and H-6 (δ_H 7.18), between H-7 (δ_H 5.38) and H-8 (δ_H 4.44), between H-5′ (δ_H 6.89) and H-6′ (δ_H 7.43), between H-7′ (δ_H 7.09) and H-8′ (δ_H 6.88) (Fig. 2). In addition, the HMBC cross-peaks from H-8 to C-10 (δ_C 106.0)/C-14 (δ_C 106.0), and from H-8′ to C-10′ (δ_C 105.0)/C-14′ (δ_C 107.1) indicated that B and D rings were aromatic AB_2 and ABC spin systems, respectively, which were different from those of **1**. These differences indicated that the D-xylose unit was connected to C-11′ in **3**, which was supported by the HMBC cross-peaks from H-1″ (δ_H 4.82) to C-11′ (δ_C 158.6) (Fig. 2). The xylosyl bond was β-configuration in **3**, which was determined by the anomeric proton signal at δ_H 4.82 (1H, d, $J = 7.2$ Hz). The *trans*-configuration of C-7/C-8 was consistent with the coupling constant (J_{7-8} = 9.0 Hz) observed in ^{1}H NMR spectrum. Thus, the relative stereochemistry of C-7 and C-8 in **3** was 7*S**, 8*S** or 7*R**,8*R**. The sign of the calculated ECD spectra for (7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*)-**3** correlated well with the overall shape of the experimental ECD spectra for **3** (Fig. S101, Supporting Information). Thus, the absolute configuration of **3** was established as 7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*. And it was named as lysidostegin C.

The HR-ESI-MS of compound **4** showed an $[M + Na]^+$ ion peak at *m/z* 609.1722 (calcd for $C_{33}H_{30}NaO_{10}$, 609.1731), consistent with the molecular formula of $C_{33}H_{30}O_{10}$. The 1D and 2D NMR spectroscopic data (Table 2) of **4** was nearly the same as those of **3**, however, with different rotation data (**3**: $[\alpha]_D^{25}$ +32.7 and **4**: $[\alpha]_D^{25}$ +18.6). Similarly to compounds **1** and **2**, compounds **3** and **4** might also be a pair of diastereoisomers with the opposite absolute configuration of C-7 and C-8. Furthermore, the calculated ECD spectrum for (7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*)-**4** was in good agreement with the experimental data for **4** (Fig. S102, Supporting Information). Thus, the absolute configurations of **4** was established as 7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*. Therefore, the structure of **4** was identified and named lysidostegin D.

Compound **5** showed an $[M + Na]^+$ ion peak at *m/z* 741.2153 (calcd for $C_{38}H_{38}NaO_{14}$, 741.2154) in the HR-ESI-MS spectrum, consistent with the molecular formula of $C_{38}H_{38}O_{14}$. The ^{1}H and ^{13}C NMR data (Table 3) of **5** were similar to those of **1**. The most notable differences between them were the presence of an additional xylose unit in **5**, and the aromatic AB_2 spin system (D ring) of **1** was replaced by an aromatic ABC spin system (D ring) of **5**. These suggested that the additional xylose unit was connected to C-11′ in **5**, which was verified by the HMBC cross-peak from H-1‴(δ_H 4.81) to C-11′ (δ_C 158.4) (Fig. 2). Moreover, the type of sugar residue was confirmed by the acid hydrolysis of **5**, wherein only the D-xylose was detected. The β-anomeric configurations of the two xylosyl units were determined by the anomeric proton signals at δ_H 4.81 (1H, d, J = 7.0 Hz) and 4.75 (1H, d, J = 7.0 Hz) in **5**. The *trans*-configuration of C-7/C-8 was consistent with the coupling constant (J_{7-8} = 9.0 Hz) observed in ^{1}H NMR spectrum. Furthermore, the calculated ECD spectrum for (7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*,1‴*S*,2‴*R*,3‴*S*,4‴*R*)-**5** matched well with that of measured for **5** (Fig. S103, Supporting Information). Thus, the absolute configuration of **5** was established as 7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*,1‴*S*,2‴*R*,3‴*S*,4‴*R*. Thus, the structure of **5** is determined and named as lysidostegin E.

The molecular formula of **6** was determined to be $C_{38}H_{38}O_{14}$ by HR-ESI-MS (*m/z* 741.2141 $[M + Na]^+$, calcd for $C_{38}H_{38}NaO_{14}$, 741.2154). Its NMR data (Table 3) were similar to those of **5**, indicating that **6** possessed the same skeleton of stilbene dixyloside. The β-D-xylose moieties were determined by the acid hydrolysis and the anomeric proton signals at δ_H 4.81 (1H, d, $J = 7.0$ Hz) and 4.79 (1H, d, $J = 7.0$ Hz). However, the rotation data of **6** (+16.4) and **5** (+35.2) were different, which indicated that compounds **6** and **5** might be a pair of diastereoisomers with the oppositely absolute configurations of C-7 and C-8. Furthermore, the calculated ECD spectrum for (7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*,1‴*S*,2‴*R*,3‴*S*,4‴*R*)-**6** was in good agreement with the experimental data for **6** (Fig. S104, Supporting Information). Thus, the absolute configuration of **6** was established as 7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*,1‴*S*,2‴*R*,3‴*S*,4‴*R*. Therefore, the structure of **6** was determined and named as lysidostegin F.

Compound **7** was assigned the molecular formula $C_{39}H_{40}O_{15}$ by HR-ESI-MS (*m/z* 771.2272 $[M + Na]^+$, calcd for $C_{39}H_{40}O_{15}Na$, 771.2259). According to 1D and 2D NMR spectra (Table 4), compound **7** has the same stilbene dimer skeleton as resveratrol (*E*)-dehydrodimer-11-*O*-β-D-glucopyranoside [19], except for the presence of an additional xylose unit (δ_C 100.8, 76.5, 73.0, 69.4, 65.7) in **7**, and the aromatic AB_2 spin system (D ring) of resveratrol (*E*)-dehydrodimer-11-*O*-β-D-glucopyranoside was replaced by an aromatic ABC spin system (D ring) of **7**. This indicated an additional xylose unit connecting to C-11′ in **7**, which was verified by the HMBC cross-peak from H-1‴(δ_H 4.81) to C-11′ (δ_C 158.4) (Fig. 2). The β-anomeric configurations of xylosyl and glucosyl units were determined by the anomeric proton signals at δ_H 4.81 (1H, d, J = 7.0 Hz) and 4.74 (1H, d, $J = 7.4$ Hz) in **7**. The *trans*-configuration of C-7/C-8 was consistent with the observed coupling constant (J_{7-8} = 9.0 Hz). Furthermore, the calculated ECD spectrum for (7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*,5″*S*,1‴*S*,2‴*R*,3‴*S*,4‴*R*)-**7** matched well with that measured for **7** (Fig. S105, Supporting Information). Thus, the absolute configuration of **7** was established as 7*S*,8*S*,1″*S*,2″*R*,3″*S*,4″*R*,5″*S*,1‴*S*,2‴*R*,3‴*S*,4‴*R*. Therefore, the structure of compound **7** was identified as shown, and it was named lysidostegin G.

The molecular formula of **8** was established to be $C_{39}H_{40}O_{15}$ by its HR-ESI-MS *m/z* 771.2256 $[M + Na]^+$ (calcd for $C_{39}H_{40}O_{15}Na$, 771.2259). Its NMR data (Table 4) were similar to those of **7**, indicating that **8** possessed the same skeleton of stilbene dimer. The β-D-xylose and β-D-glucose moieties were determined by the acid hydrolysis and the anomeric proton signals at δ_H 4.81 (1H, d, $J = 7.0$ Hz) and 4.78 (1H, d, J = 7.4 Hz). However, the rotation data of **8** (+22.9) and **7** (+31.9) were different, which indicated that compounds **8** and **7** might be a pair of diastereoisomers with the oppositely absolute configurations of C-7 and C-8. Furthermore, the calculated ECD spectrum for (7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*,5″*S*,1‴*S*,2‴*R*,3‴*S*,4‴*R*)-**8** was in good agreement with the experimental data for **8** (Fig. S106, Supporting Information). Thus, the absolute configuration of **8** was established as 7*R*,8*R*,1″*S*,2″*R*,3″*S*,4″*R*,5″*S*,1‴*S*,2‴*R*,3‴*S*,4‴*R*. And it was named lysidostegin H.

Compound **9** was isolated as yellow powder with $[\alpha]_D^{25}$ +15.5 (*c* 1.0, MeOH). Its molecular formula was deduced as $C_{22}H_{20}O_8$ on the basis of its ^{13}C NMR and HR-ESI-MS (*m/z* 413.1213 $[M + H]^+$, calcd for $C_{22}H_{21}O_8$, 413.1231) data. The IR spectrum suggested the presence of a hydroxy group (3308 cm^{-1}) and an aromatic ring (1625 and 1506 cm^{-1}). The ^{1}H NMR spectrum (Table 5) showed characteristic signals for an aromatic AX spin system [δ_H 5.87 (1H, d, $J = 1.6$ Hz), 5.70 (1H, d, J = 1.6 Hz)], an aromatic ABX spin system [δ_H 6.85 (1H, d, $J = 1.2$ Hz), 6.72 (1H, d, $J = 8.4$ Hz), 6.69 (1H, dd, $J = 8.4$, 1.2 Hz)], an aromatic ABC spin system [δ_H 6.38 (1H, d, $J = 8.2$ Hz), 6.15 (1H, dd, $J = 8.2$, 2.4 Hz), 6.10 (1H, d, J = 2.4 Hz)], three methines [δ_H 4.49 (1H, d, J = 9.5 Hz), 4.41 (1H, d, J = 9.5 Hz), 4.22 (1H, t, J = 9.5 Hz)], and a methoxy group [δ_H 2.68 (3H, s)]. The analysis of its ^{13}C NMR data (Table 5) revealed the presence of 22 carbon resonances, including 10 quaternary carbons (7 oxygenated), 11 methines (2 oxygenated), and 1 methoxy group. The NMR data of compound **9** resembled those of the known compound **10** [12], except for the presence of an additional methoxy group (δ_C 59.6) in **9** and the chemical shift of C-3 shifted from δ_C 68.0 in the known one to δ_C 81.0 in **9**. The differences indicated the hydroxy group at C-3 in **9** was replaced by a methoxy group. This was confirmed by the ^{1}H—^{1}H COSY cross-peaks between H-3 (δ_H 4.22) and H-2 (δ_H 4.49)/H-4 (δ_H 4.41), together with the HMBC cross-peaks from 3-OCH_3 (δ_H 2.68) to C-3 (δ_C 81.0)/C-4 (δ_C 40.5) (Fig. 2). The relative stereochemistry of **9** was similar to the known compound **10**, which was determined by the NOESY correlations between 3-OCH_3 and H-2/H-4 (Fig. 3). The absolute configuration of **9** was confirmed by quantum-chemical ECD calculations. The experimental ECD spectrum of **9** was similar to that of the calculated ECD spectrum for (2*R*,3*S*,4*R*)-**9** (Fig. S107, Supporting Information). Hence, the absolute configuration of **9** was elucidated as 2*R*,3*S*,4*R*. Based on the above analysis, the structure was elucidated as shown and named lysidostegin I.

Table 6
Antioxidant activities of compounds **1**–**16.**

Compounds	IC_{50} (μM)[a]	Compounds	IC_{50} (μM)[a]
1	26.59 ± 1.78	**10**	13.60 ± 1.47
2	25.12 ± 2.33	**11**	35.51 ± 1.92
3	30.71 ± 1.83	**12**	31.60 ± 2.45
4	28.05 ± 1.69	**13**	49.37 ± 1.93
5	32.54 ± 1.68	**14**	40.38 ± 2.57
6	34.17 ± 2.29	**15**	45.76 ± 1.12
7	22.71 ± 1.19	**16**	56.92 ± 1.46
8	24.31 ± 1.22	Vitamin E[b]	17.39 ± 1.72
9	9.57 ± 1.30		

[a] Data were expressed as mean ± SD.
[b] Positive control.

In addition, seven known compounds, (2*R*,3*S*,4*R*)-2,3-trans-3,4-*trans*-4-(2,4,6-trihydroxyphenyl)-3′,4′,5,7-tetrahydroxyflavan-3-ol (**10**) [12], *trans*-resveratrol (**11**) [13], piceid (**12**) [14], 3-hydroxy-5-[(1*Z*)-2-(4-hydroxyphenyl)ethenyl] phenyl (**13**) [15], (*E*)-resveratrol-3-*O*-rutinoside (**14**) [16], lysidiside N (**15**) [16], and lysidiside E (**16**) [17], were identified from L. *rhodostegia*, by comparing their spectroscopic and physical data with those of related literatures.

The isolates **1**–**16** were evaluated for their antioxidant activities using the DPPH radical-scavenging assay, with vitamin E as a positive control. As shown in Table 6, all compounds showed antioxidant activities with IC_{50} values ranging from 9.57 ± 1.30 to 56.92 ± 1.46 μM. Among them, flavanols **9** and **10** exhibited stronger antioxidant effects than the positive control and the other compounds, with IC_{50} values of 9.57 ± 1.30 and 13.60 ± 1.47 μM, respectively. These results revealed that the flavanols may be the important active antioxidant ingredients in the roots of L. *rhodostegia*.

In conclusion, nine new compounds and seven known ones were isolated from the roots of *Lysidice rhodostegia*. Their structures were characterized by extensive analysis of the spectroscopic data, ECD calculations and acid hydrolysis. The antioxidant activities of these compounds were evaluated on DPPH radical-scavenging assay. And all tested isolates show various levels of antioxidant activities.

Conflicts of interest

The authors declare no competing financial interest.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fitote.2021.104997.

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