Chemical Constituents from the Leaves and Stems of *Schisandra lancifolia*

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A new nortriterpenoid, 20-hydroxymicrandilactone D (1) and a novel lignan glycoside, lancilignanside A (2) were isolated from leaves and stems of *Schisandra lancifolia*, together with three known nortriterpenoids (3—5) and nine known phenolics (6—14). The structures of new compounds 1 and 2 were determined by detailed analysis of their 1D and 2D NMR spectra, and chemical evidences. In addition, compounds 1—2, 6—7, and 9—11 showed anti-human immunodeficiency virus (HIV)-1 activities with 50% effective concentration (EC_{50}) in the range of 3.0—99.0 μg/ml. Compound 12 was not bioactive in this assay with EC_{50} more than 200 μg/ml.

Key words nortriterpenoid; lignan; *Schisandra lancifolia*; anti-human immunodeficiency virus-1 activity

Plants of the genus *Schisandra* (Schisandraceae) is economically and medicinally valuable and is used widely in traditional Chinese medicines. They were proved to be rich sources of bioactive lignans, which possessed various beneficial pharmacological effects such as antihepatitis, antitumor and anti-human immunodeficiency virus (HIV-1) activity.[1—5] During the past 10 years, great efforts of our group have been devoted to the phytochemical investigations on more than 10 medicinal plants of the genus *Schisandra*. A series of structurally attractive nortriterpenoids have been isolated so far, which can be mainly divided into schiartane, 18-norschiartane, 18(13→14)-*abeo*-schiartane, schisanartane, pre-schisanartane and wuweiziartane types.[6] Some of them exhibited modest or strong anti-HIV activities [7—9] and cytotoxicity.[10,11]

Schisandra lancifolia (Rehd. *et* Wils) A. C. Smith, is a climbing plant mainly distributed in Mainland of China. Our previous research on this plant collected from Yunnan region of China led to the discovery of some new highly oxygenated nortriterpenoids.[8,9,12—16] Reinvestigation of the leaves and stems of this plant led to the isolation of a new nortriterpenoid, 20-hydroxymicrandilactone D (**1**), and a novel lignan glycoside, lancilignanside A (**2**), together with three known triterpenoids, micrandilactone A (**3**),[17] micrandilactone D (**4**),[18,19] and lancifodilactone L (**5**),[12] and nine known phenolics including 4,4-di(4-hydroxy-3-methoxyphenyl)-2,3-dimethylbutanol (**6**),[20] (+)-1-hydroxypinoresinol (**7**),[21] prinsepiol (**8**),[22] evofolin (**9**),[23] balaphonin (**10**),[24] phyllocoumarin (**11**),[25] (−)-epicatechin (**12**),[26] quercetin (**13**),[27] and 3,4′,5,7-tetrahydroxyflavone (**14**) (Fig. 1).[28] The structures of **1** and **2** were determined by extensive NMR spectroscopic experiments, including ^1H–^1H correlation spectroscopy (COSY), heteronuclear single quantum coherence (HSQC), heteronuclear multiple bond correlation (HMBC), and rotating frame Overhauser enhancement spectroscopy (ROESY) techniques, together with chemical evidences. In addition, some isolates were evaluated for anti-HIV activity. The present paper reports the isolation, structural elucidation, and biological evaluation of the new compounds.

Results and Discussion

Compound **1** gave a quasi-molecular ion at *m/z* 599 $[M+Na]^+$ in its positive electrospray ionization (ESI)-MS spectrum and possessed a molecular formula of $C_{29}H_{36}O_{12}$, which was confirmed by HR-ESI-MS (Found $[M+Na]^+$ 599.2213, Calcd 599.2207). Its ^1H-NMR spectrum showed five tertiary methyl signals. Its ^{13}C- and distortionless en-

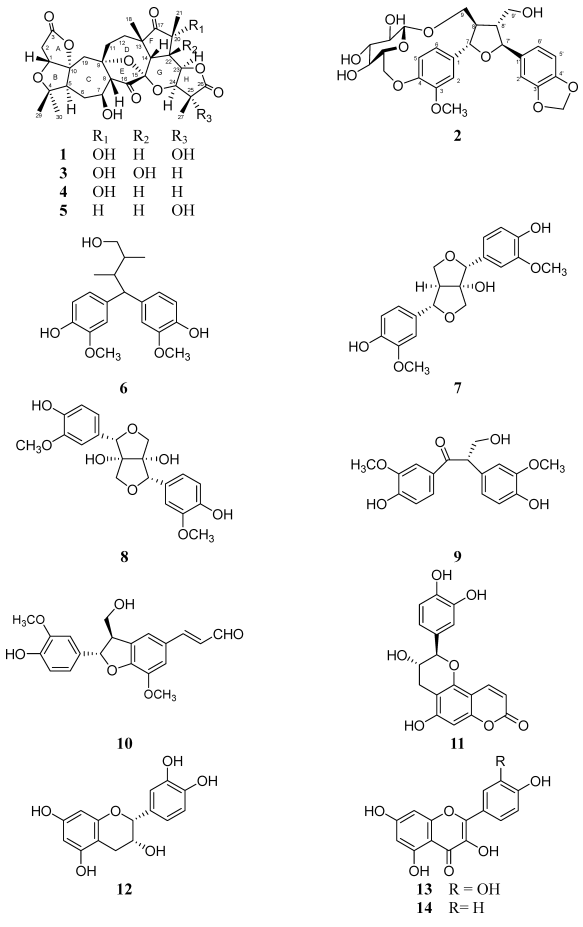

Fig. 1. Structures of Compounds **1**—**14**

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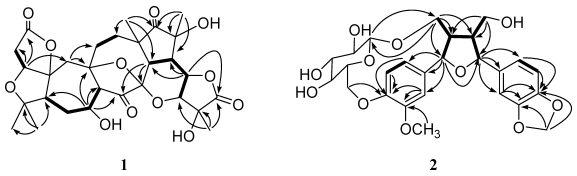
Table 1. ^{1}H- and ^{13}C-NMR Data for Compounds **1** and **2**[a)]

Position	**1**		Position	**2**	
	δ_C	δ_H		δ_C	δ_H
1	81.5 d	4.20 (d, 6.1)	1	138.4 s	
2α	35.0 t	2.69 (d, 15.4)	2	107.4 d	6.99 (d, 1.3)
2β		2.85 (dd, 6.1, 15.4)	3	148.4 s	
3	174.9 s		4	148.2 s	
4	84.0 s		5	115.4 d	6.76 (d, 8.0)
5	58.2 d	2.51 (dd, 3.5, 10.5)	6	120.4 d	6.92 (d, 1.3, 8.0)
6α	36.6 t	2.19 (m)	7	83.3 d	5.01 (d, 8.2)
6β		2.05 (m)	8	52.6 d	2.40 (m)
7	67.9 d	4.60 (m)	9	69.4 t	3.57 (overlapped)
8	60.3 d	3.02 (d, 7.7)			3.98 (dd, 4.4, 9.6)
9	82.4 s		1′	135.3 s	
10	95.7 s		2′	110.8 d	7.05 (d, 1.7)
11α	41.4 t	2.10 (m)	3′	147.6 s	
11β		1.75 (m)	4′	146.8 s	
12α	30.8 t	1.62 (m)	5′	108.5 d	6.80 (d, 8.1)
12β		1.90 (m)	6′	120.0 d	6.88 (dd, 1.7, 8.1)
13	49.3 s		7′	83.5 d	4.94 (d, 8.6)
14	44.8 d	2.93 (d, 8.3)	8′	55.2 d	2.28 (m)
15	98.5 s		9′	61.4 t	3.58 (overlapped)
16	208.2 s				3.70 (m)
17	220.2 s		G-1	104.6 d	4.27 (d, 7.1)
18	26.7 q	1.03 (s)	G-2	74.2 d	3.16 (overlapped)
19α	42.6 t	2.19 (AB d, 12.9)	G-3	77.3 d	3.35 (t, 7.6)
19β		2.51 (AB d, 12.9)	G-4	70.7 d	3.46 (overlapped)
20	74.9 s		G-5	77.3 d	3.45 (overlapped)
21	24.5 q	1.55 (s)	G-6	66.2 t	3.18 (overlapped)
22	41.9 d	3.27 (d, 8.3)			3.79 (dd, 5.7, 10.8)
23	73.5 d	5.61 (br s)	OMe	56.2 q	3.83 (s)
24	76.5 d	5.20 (br s)	OCH_2O	101.8 t	5.97 (s)
25	76.5 s				
26	177.2 s				
27	17.5 q	1.70 (s)			
29	21.0 q	1.10 (s)			
30	29.8 q	1.25 (s)			

a) Data were recorded in C_5D_5N for **1** and in acetone-d_6 for **2** at 125 MHz (^{13}C-NMR) and 500 MHz (^{1}H-NMR); chemical shifts (δ) are expressed in ppm.

hancement by polarization transfer (DEPT) NMR data showed the signals for 29 carbons, including two ester groups, two ketone groups, seven quaternary carbons (six oxygenated ones), eight methines (four oxygenated ones), four methylenes, and five methyls (Table 1). This hint that **1** was a highly oxygenated nortriterpene and contained eight rings. Analysis of the ^{1}H- and ^{13}C-NMR data of **1** revealed that they are similar to those reported for micrandilactone D (**4**),[18,19] which was also isolated in this plant. Comparison of 1D NMR data, together with detailed HMBC and ^{1}H–^{1}H COSY analyses indicated that the difference was due to a methine (C-25) in **4** was replaced by an oxygenated quaternary carbon (C-25, δ_C 76.5) in **1** (Fig. 2). This was confirmed by HMBC correlations from Me-27 (δ_H 1.70, s) to C-25 (δ_C 76.5), C-24 (δ_C 76.5), and C-26 (δ_C 177.2).

The relative stereochemistry of **1** was established by analysis of its ROESY NMR data and chemical shift comparison with those of micrandilactone D.[18,19] Biogenetically, H-C(5) was α- and Me(18) was β-oriented.[18,19] The ROESY correlation of H-5 with H-7 indicated α-orientation of H-7 (Fig. 3). Accordingly, OH-7 was assigned as β-oriented. ROESY correlations of Me-18 with H-14, H-22, and Me-21 indicated that H-14, H-22, and Me-21 were on the same face and all were β-oriented. In addition, ROESY correlations of Me-27 with H-14 and H-22 indicated Me-27 was β-oriented and

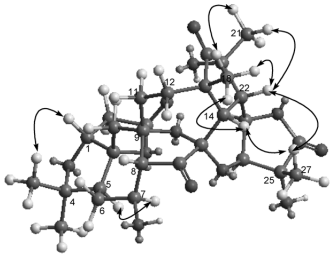


1 **2**

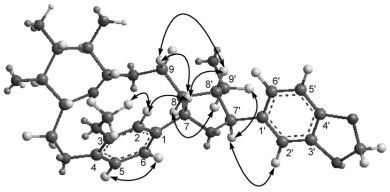
Fig. 2. Selected HMBC (→) and ^{1}H–^{1}H COSY (—) Correlations of **1** and **2**

therefore, OH-25 was α-oriented. Thus, compound **1** was determined to be a 20-hydroxy derivative of micrandilactone D, and gave the name as 20-hydroxymicrandilactone D.

Compound **2**, $[\alpha]_D^{25.5}$ +12.5° (c=0.115, CH_3OH), was isolated as an amorphous powder. The molecular formula was determined to be $C_{26}H_{31}O_{11}$ by HR-ESI-MS (Found $[M+H]^+$ 519.1782, Calcd 519.1788). The ^{1}H-NMR spectrum showed obvious signals due to two methines [δ_H 2.40 (1H, m, H-8), 2.28 (1H, m, H-8′)], one methoxyl group (δ_H 3.83, 3H, s), one methylenedioxy group (δ_H 5.97, 2H, s), an anomeric proton (δ_H 4.27, 1H, d, J=7.1 Hz), and two sets of 1,3,4-trisubstituted benzene protons [δ_H 6.99 (1H, d, J=1.3 Hz, H-2), 6.76 (1H, d, J=8.0 Hz, H-5) , 6.92 (1H, dd, J=1.3, 8.0 Hz, H-6), 7.05 (1H, d, J=1.7 Hz, H-2′), 6.80 (1H, d, J=8.1 Hz, H-5′), 6.88 (1H, dd, J=1.7, 8.1 Hz, H-6′)] (Table 1). The ^{13}C- and DEPT NMR spectra showed signals due to



1



2

Fig. 3. Key ROESY Correlations of **1** and **2**

twelve aromatic carbons, two methines (C-8 and C-8′), two oxymethines (C-7 and C-7′), two oxymethylenes (C-9 and C-9′), one methoxyl group, one methylenedioxy group, and six carbon signals due to a hexose (Table 1). From these functionalities, the aglycone suggested a 2,5-diaryltetrahydrofuranoid-type lignan.[29,30] In addition, HMBC correlations from the protons of one methoxyl group (δ_H 3.83, 3H, s) to C-3 (δ_C 148.4) showed that the methoxyl group was located at C-3, which was further confirmed by the ROESY correlation of the methoxyl group protons with H-2. HMBC correlations of protons of one methylenedioxy group with C-3′ and C-4′ determined the methylenedioxy group was located between C-3′ and C-4′. The hexose was deduced to be glucose by the comparison of chemical shifts of sugar unit with those of 7*S*,7′*S*,8*R*,8′*R*-icariol A_2-9-*O*-β-D-glucopyranoside,[29] and was further confirmed by acid hydrolysis of **2**, which afforded D-glucose. The glucose was unambiguously assigned to C-9 (δ_C 69.4) due to the obvious ^1H–^{13}C long-range correlations between the anomeric proton of glucose and C-9 and between H-9 and the anomeric carbon of glucose (Fig. 2). The anomeric proton was determined to be β-configuration on the basis of the large coupling constants of the anomeric protons (δ_H 4.27, d, J=7.1 Hz). Furthermore, HMBC correlation of G-6 protons of the glucose with C-4 showed the glucose was further connected with C-4 through G-6, which was also supported by the downfield shift of G-6 to δ_C 66.2. Therefore, the planar structure was established as shown.

The relative stereochemistry of **2** was established by analysis of its ROESY NMR data and comparisons of coupling constants of H-7 and H-7′ and chemical shifts with those of 7*S*,7′*S*,8*R*,8′*R*-icariol A_2-9-*O*-β-D-glucopyranoside.[29] The relative configurations between C-7 and C-8, and between C-7′ and C-8′ were both established as *trans*-configurations by ROESY correlations of H-7 with H-9, and H-7′ with H-9′, respectively, which also supported by the similarity of coupling constants of H-7 (J=8.2 Hz) and H-7′ (J=8.6 Hz) with those of 7*S*,7′*S*,8*R*,8′*R*-icariol A_2-9-*O*-β-D-glucopyranoside (H-7, J=8.3 Hz; H-7′, J=8.5 Hz).[29] The relative configuration between C-8 and C-8′ was established as *trans*-configuration by ROESY correlations of H-8 with H-9′, H-8′ with H-9, and no ROESY correlation observed between H-8 and H-8′. Thus, the structure of **2** was established, and given the trivial name, lancilignanside A.

Table 2. Summary of Cytotoxicities and Anti-HIV-1 Activities

Compounds	Cytotoxicity CC_{50} (μg/ml)[a]	Anti-HIV-1_{IIIB} activitiy EC_{50} (μg/ml)	Selectivity index CC_{50}/EC_{50}
1	>200	99.0	>2.0
2	>200	76.8	>2.6
6	>200	10.5	9.5
7	>200	79.4	>2.5
9	109.7	37.2	3.0
10	12.6	3.0	4.3
11	>200	43.7	4.6
12	>200	>200	

a) Minimal cytotoxicity against C8166 cells when CC_{50} >200 (μg/ml).

The anti-HIV-1 activities of compounds **1**—**2**, **6**—**7**, and **9**—**12** were evaluated in preventing the cytopathic effects of HIV-1 in C8166, and cytotoxicity was measured in parallel with the determination of antiviral activity using azidothymidine (AZT) as a positive control (50% effective concentration (EC_{50})=0.0033 μg/ml and CC_{50}>200 μg/ml).[31] Compounds **1**—**2**, **6**—**7**, and **9**—**12** exerted minimal cytotoxicity against C8166 cells (CC_{50}>200 μg/ml) and compounds **1**—**2**, **6**—**7**, and **9**—**11** showed anti-HIV-1 activities with EC_{50} values in the range of 3.0—99.0 μg/ml. Compound **12** was not bioactive in this assay with EC_{50} value more than 200 μg/ml (Table 2).

Experimental

General Experimental Procedures Optical rotations were measured with a Horiba SEPA-300 polarimeter. UV spectra were obtained using a Shimadzu UV-2401A spectrophotometer. A Tenor 27 spectrophotometer was used for scanning IR spectroscopy with KBr pellets. 1D and 2D NMR spectra were recorded on DRX-500 spectrometers with TMS as internal standard. Unless otherwise specified, chemical shifts (δ) were expressed in ppm with reference to the solvent signals. HR-ESI-MS was performed on an API QSTAR time-of-flight spectrometer and a VG Autospec-3000 spectrometer, respectively. Preparative HPLC was performed on a Shimadzu LC-8A preparative liquid chromatograph with a ZORBAX PrepHT GF (21.2 mm×25 cm, 7 μm) column. Column chromatography was performed with Si gel (200—300 mesh, Qing-dao Marine Chemical, Inc., Qingdao, China), Lichroprep RP-18 gel (40—63 μm, Merck, Darmstadt, Germany). The fractions were monitored by TLC, and spots were visualized by heating Si gel plates sprayed with 5% H_2SO_4 in EtOH.

Plant Material The leaves and stems of *S. lancifolia* were collected in Dali Prefecture, Yunnan Province, People's Republic of China, in August 2006. The specimen was identified by Prof. Xi-Wen Li and a voucher specimen (No. KIB 2006-08-14) has been deposited at the State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences.

Extraction and Isolation The leaves and stems of *S. lancifolia* (2.5 kg) was ground and exhaustively extracted with 70% aqueous Me_2CO at room temperature. The solvent was evaporated *in vacuo*, and the crude extract (170 g) was dissolved in H_2O and partitioned with EtOAc. The EtOAc portion (78 g) was chromatographed on a silica gel column eluting with $CHCl_3$–Me_2CO (1 : 0, 9 : 1, 8 : 2, 2 : 1, 1 : 1, 0 : 1) to afford fractions A—F. Fraction B (13.4 g) was repeatedly chromatographed on silica gel (200—300 mesh) and Sephadex LH-20, and finally by semi-preparative HPLC (MeOH : H_2O, 45 : 55) to yield compounds **2** (7 mg), **6** (8 mg), and **11** (9 mg). Fraction C (15.6 g) was chromatographed on a silica gel column eluting with $CHCl_3$–MeOH (10 : 1, 5 : 1, 2 : 1, 1 : 1) to afford five fractions. Fraction C-1 (3.1 g) was purified by recrystallization and repeated chromatography over silica gel, RP-18 and Sephadex LH-20 (MeOH), and fol-

lowed by semi-preparative and preparative HPLC (MeOH : H_2O, 35 : 65 and MeOH : CH_3CN : H_2O, 15 : 33 : 52) to yield compounds **1** (5 mg), **3** (6 mg), **5** (9 mg), **10** (18 mg), and **14** (44 mg). Similarly, Fraction C-2 (2.6 g) and C-3 (1.4 g) were respectively purified using the chromatography methods abovementioned, to yield compounds **4** (10 mg), **7** (26 mg), **8** (2 mg), **9** (9 mg), **12** (20 mg), and **13** (16 mg).

Compound **1**: White powder. $[\alpha]_D^{25.4}$ +51.1° (c=0.101, CH_3OH). UV (CH_3OH): λ_{max} (log ε) 202 (3.35) nm. IR (KBr): ν_{max} 3455, 2925, 2846, 1768, 1632, 1378, 1125, 1019, 578 cm^{-1}. Positive ESI-MS *m/z*: 599 $[M+Na]^+$. HR-ESI-MS *m/z*: 599.2213 $[M+Na]^+$ (Calcd 599.2207 for $C_{29}H_{36}O_{12}Na$). NMR data: see Table 1.

Compound **2**: Amorphous powder. $[\alpha]_D^{25.5}$ +12.5° (c=0.115, CH_3OH). UV (CH_3OH): λ_{max} (log ε) 210 (3.87), 240 (3.65), 272 (3.1) nm. IR (KBr): ν_{max} 3455, 2925, 2877, 1771, 1726, 1622, 1460, 1167, 1075, 1021, 879 cm^{-1}. Positive ESI-MS *m/z*: 519 $[M+H]^+$. HR-ESI-MS *m/z*: 519.1782 $[M+H]^+$ (Calcd 519.1788 for $C_{26}H_{31}O_{11}$). NMR data: see Table 1.

Acid Hydrolysis of Compound 2 A solution of **2** (3.5 mg) in 2 M HCl (4 ml) was heated in a water bath at 80 °C for 6 h. After cooling, the reaction mixture was neutralized with $NaHCO_3$ and extracted with $CHCl_3$. We were not able to get the aglycone of **2** unfortunately, because the TLC inspection of the $CHCl_3$ part indicated that at least four products were yielded, which were not subjected to further isolation and identification due to limited amount. However, through co-TLC with authentic sample using $CHCl_3$–MeOH–H_2O–HOAC (7 : 3 : 0.8 : 1) as developing system, glucose was detected in the water layer (Rf=0.49), which was further concentrated to dryness and subjected to silica gel chromatography eluting with $CHCl_3$–MeOH (1 : 1) to give purified D-glucose (0.9 mg): $[\alpha]_D^{18.8}$ +24.7 (c=0.12, H_2O).

Anti-HIV-1 Assay The anti-HIV activities and cytotoxicities were assessed by microtiter syncytium formation infectivity assay, using the method previously described, with AZT as a positive control.[31]

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