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Pentasaccharide Glycosides from the Tubers of Sweet Potato (*Ipomoea batatas*)

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Sweet potato (*Ipomoea batatas*) has been used as food and herb in many countries. In this research on the active constituents of sweet potato, nine compounds were isolated and identified, including seven new resin glycosides, batatosides A−G (**1**–**7**), along with two known compounds, batatinoside I (**8**) and simonin IV (**9**). The structures of **1**–**9** have been established by a combination of spectroscopic and chemical methods. The major characteristics of the new compounds are the presence of three different substituents. The absolute configuration of aglycones was established as *S* by Mosher's method. Batatoside E (**5**) showed weak cytotoxic activity against Hep-2 cells.

KEYWORDS: ***Ipomoea batatas*; resin glycoside; cinnamic acid; batatoside**

INTRODUCTION

Ipomoea batatas (L.) Lam. (Convolvulaceae) with the common name of sweet potato is also known as "shanyu", "hongshu", "digua", and "hongshao" in mainland of China. The colors of the peel and flowers of sweet potato are different depending on the location of growth. The aerial part of the sweet potato is used as a vegetable and the underground part is used as food or a raw material in industry (*1*). Scammony root (*Convolvulus scammonia*) of Mexican jalaps, which was introduced to Europe by Spanish colonists, was utilized as a purgative medicine (*2, 3*). The antibacterial activities of the 22 oligosaccharides from *Ipomoea tricolor* and *Ipomoea orizabensis* were evaluated against a panel of *Staphylococcus aureus* strains possessing or lacking specific efflux pumps (*4*). One of the six pentasaccharides from *Ipomoea murucoides* exhibited marginal cytotoxicity against Hep-2 cells (*5*). In Chinese traditional medicine, the tubers of *I. batatas* have been used as a medicinal herb to promote the production of body fluids, hemostasis, and apocenosis (*6*). Recently, two new ester-type dimer resin glycosides were described from *I. batatas* tubers collected in Mexico (*7*). Considering the usage as food and vegetable as well as pharmacological activities of *I. batatas*, we have been encouraged to investigate the constituents further. This study was designed to isolate and structurally elucidate new resin glycosides batatosides A (**1**)−G (**7**) as well as known resin glycosides batatinoside I (**8**) and simonin IV (**9**) and to investigate the cytotoxic activity of these compounds against Hep-2 cells.

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MATERIALS AND METHODS

General Procedures. Optical rotations were measured with a JASCO P-1020 polarimeter. UV and IR spectra were recorded with Shimadzu UV-2501PC and Nicolet Impact 410 spectrometers, respectively. ^{1}H and ^{13}C NMR, HSQC, HMBC, and TOCSY spectra were taken with Bruker ACF-600 spectrometers (600 and 150 MHz, respectively) in pyridine-d_5; chemical shifts are reported in parts per million as δ relative to Me_4Si (internal standard). Mass spectra were obtained on an MS Agilent 1100 series LC/MSD ion trap mass spectrometer (ESIMS), and HR-ESIMS were recorded with an Agilent TOF MSD 1946D spectrometer. TLC was performed on precoated silica gel 60 F_{254} (Qingdao Marine Chemical Co. Ltd.) and detected by spraying with 10% H_2SO_4/EtOH. Column chromatography was carried out with silica gel H (Qingdao Marine Chemical Co. Ltd.), Sephadex LH-20 (20–100 μ, Pharmacia), and ODS-C_{18} (100–200 μ, Waters). Preparative HPLC was carried out using an Agilent 1100 series instrument with a 200 mm × 20 mm i.d. Shimpak RP-C_{18} column (Shimadzu) and UV detector at 280 nm.

Plant Material. The tubers of *I. batatas* were collected in Yanlin County, Hunan Province, People's Republic of China, in September 2004 and identified by Prof. Min-jian Qin, Department of Medicinal Plants, China Pharmaceutical University. A voucher specimen (No. 040912) was deposited in the Department of Natural Medicinal Chemistry, China Pharmaceutical University.

Extraction and Isolation. Tubers (18 kg) of *I. batatas* were crushed and dried in the shade for 1 week. The pieces were extracted with 95% EtOH (3 × 20 L × 2 h) at 80 °C, and the extraction solution was concentrated under a vacuum and allowed to stand overnight. The solution was further concentrated to produce a residue, which was partitioned between $CHCl_3$ (5 × 0.5 L) and water (0.5 L) to give 45 and 18 g of extracts from these two layers, respectively. The $CHCl_3$ extract was subjected to chromatography on a 60 cm × 5 cm 200–300 mesh silica gel (300 g), eluted with $CHCl_3$/MeOH (100:3→100:50). Fractions of 245−272 (1.8 g) eluted with $CHCl_3$/MeOH (100:10) were further submitted to chromatography on a 30 cm × 1.5 cm (60 μm) RP-C_{18} and eluted with MeOH/H_2O (90:10→100:0) to afford fraction 1 (0.3 g) with 90:10 MeOH/H_2O, fraction 2 (0.9 g) with 95:5 MeOH/

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H_2O, and fraction 3 (0.6 g) with MeOH, respectively. Using preparative HPLC (UV detector at 280 nm) over a C_{18} column, two peaks were collected from fraction 1 eluted with 91% MeOH/H_2O, to afford batatoside A (**1**, 4.5 mg, t_R 8.25 min) and batatoside B (**2**, 3.8 mg, t_R 9.54 min). Fraction 2 afforded batatoside C (**3**, 18.4 mg, t_R 11.10 min), batatoside F (**6**, 15.6 mg, t_R 5.91 min), batatoside G (**7**, 8.7 mg, t_R 5.36 min), batatinoside I (**8**, 26.0 mg, t_R 7.91 min), and simonin IV (**9**, 46.2 mg, t_R 19.60 min), when eluted with 95% MeOH/H_2O. Fraction 3 gave batatoside D (**4**, 208.4 mg, t_R 24.50) and batatoside E (**5**, 361.3 mg, t_R 22.00min), when eluted with 98% MeOH/H_2O.

Batatoside A (1): amorphous white powder; mp 122–124 °C; $[\alpha]_D^{25}$ −11.6 (*c* 0.55, MeOH); IR ν_{max} (KBr) 3443, 2934, 2859, 1722, 1637, 1137, 1097 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.17) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **2**; ESIMS *m/z* 1267 $[M - H]^-$; HRESIMS *m/z* 1267.6425 $[M - H]^-$ (calcd for $C_{64}H_{99}O_{25}$, 1267.6475).

Batatoside B (2): amorphous white powder; mp 119–121 °C; $[\alpha]_D^{25}$ −12.1 (*c* 0.50, MeOH); IR ν_{max} (KBr) 3444, 2929, 2856, 1722, 1637, 1136 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.37) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **2**; ESIMS *m/z* 1253 $[M - H]^-$; HRESIMS *m/z* 1253.5944 $[M - H]^-$ (calcd for $C_{63}H_{97}O_{25}$, 1253.6324).

Batatoside C (3): amorphous white powder; mp 109–111 °C; $[\alpha]_D^{25}$ −22.0 (*c* 0.55, MeOH); IR ν_{max} (KBr) 3444, 2931, 2857, 1737, 1636, 1137, 1064 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.27) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **2**; ESIMS *m/z* 1415 $[M + Cl]^-$; HRESIMS *m/z* 1415.7580 $[M + Cl]^-$ (calcd for $C_{72}H_{116}ClO_{25}$, 1415.7494).

Batatoside D (4): amorphous white powder; mp 105–107 °C; $[\alpha]_D^{25}$ −10.2 (*c* 0.15, MeOH); IR ν_{max} (KBr) 3444, 2931, 2857, 1737, 1636, 1137, 1064 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.19) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **3**; ESIMS *m/z* 1415 $[M + Cl]^-$; HRESIMS *m/z* 1415.7487 $[M + Cl]^-$ (calcd for $C_{72}H_{116}ClO_{25}$, 1415.7494).

Batatoside E (5): amorphous white powder; mp 115–117 °C; $[\alpha]_D^{25}$ −20.0 (*c* 0.54, MeOH); IR ν_{max} (KBr) 3445, 2929, 2857, 1725, 1636, 1136, 1070 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.04) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **3**; ESIMS *m/z* 1415 $[M + Cl]^-$; HRESIMS *m/z* 1415.7584 $[M + Cl]^-$ (calcd for $C_{72}H_{116}ClO_{25}$, 1415.7494).

Batatoside F (6): amorphous white powder; mp 110–112 °C; $[\alpha]_D^{25}$ −23.1 (*c* 0.55, MeOH); IR ν_{max} (KBr) 3442, 2928, 2856, 1728, 1635, 1135, 1070 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (3.90) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **3**; ESIMS *m/z* 1415 $[M + Cl]^-$; HRESIMS *m/z* 1415.7305 $[M + Cl]^-$ (calcd for $C_{72}H_{116}ClO_{25}$, 1415.7494).

Batatoside G (7): amorphous white powder; mp 106–108 °C; $[\alpha]_D^{25}$ −27.3 (*c* 0.17, MeOH); IR ν_{max} (KBr) 3443, 2930, 2857, 1726, 1637, 1137, 1071 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.10) nm; ^1H NMR (C_5D_5N, 600 MHz) and ^{13}C NMR (C_5D_5N, 150 MHz) data, see **Tables 1** and **3**; ESIMS *m/z* 1373 $[M + Cl]^-$; HRESIMS *m/z* 1373.6898 $[M + Cl]^-$ (calcd for $C_{69}H_{110}ClO_{25}$, 1373.7025).

Batatinoside I (8): amorphous white powder; mp 126–128 °C; $[\alpha]_D^{25}$ −9.7 (*c* 0.90, MeOH); IR ν_{max} (KBr) 3444, 2931, 1737, 1636, 1137, 1064 cm^{-1}; UV (MeOH) λ_{max} (log ϵ) 280 (4.13), 217 (3.98), 205 (3.97) nm; ESIMS *m/z* 1415 $[M + Cl]^-$, HRESIMS *m/z* 1415.7487 $[M + Cl]^-$ (calcd for $C_{72}H_{116}ClO_{25}$, 1415.7494); identified by comparison of NMR data (^{1}HNMR, ^{13}CNMR, HSQC, HMBC, TOCSY) with published values (*7*).

Simonin IV (9): amorphous white powder; mp 122–124 °C; $[\alpha]_D^{25}$ −48.0 (*c* 0.50, MeOH); IR ν_{max} (KBr) 3451, 2976, 2935, 1737, 1638, 1137, 1060; ESIMS *m/z* 1249 $[M - H]^-$; identified by comparison of NMR data (^{1}HNMR, ^{13}CNMR) with published values (*8*).

Alkaline Hydrolysis of 1–7. Compounds **1–7** (3 mg each) in 5% KOH (3 mL) were refluxed at 90 °C for 2 h, separately. The reaction mixtures were acidified to pH 4 and extracted with ether (30 mL) and *n*-BuOH (30 mL). The ether layer was washed with H_2O, dried over anhydrous Na_2SO_4, methylated with MeOH, and catalyzed with 0.5 N H_2SO_4. The methyl esters were analyzed by GC-MS on a model 3800 GC interfaced with a model 2200 MS (Varian) at 70 eV under the following conditions: 30 m × 0.25 mm i.d., 0.25 m, VF-5 ms capillary column (Varian); column temperature, 160–240 °C temperature programmed at 10 °C/min; carrier gas, N_2 (30 mL/min). Peaks in the chromatograms were detected from the hydrolysis mixtures and identified by comparison with authentic samples as *n*-dodecanoyl acid methyl ester (t_R 14.020 min) *m/z* $[M + H]^+$ 215 (23), 171 (31), 143 (55), 87 (85), 74 (100), 55 (86), 43 (96), 41 (56), from **3**−**7**; *trans*-cinnamic acid methyl ester (t_R 12.498 min) *m/z* $[M]^+$ *m/z* 162 (45), 131 (100), 103 (76), 77 (35), from **1**−**7**; (*S*)-2-methylbutyric acid methyl ester (t_R 3.593 min) *m/z* $[M + H]^+$ 117 (5), 101 (23), 88 (87), 57 (100), 41 (57), from **1**−**6**; butyric acid methyl ester (t_R 2.349 min) *m/z* $[M - H]^-$ 101 (3), 74 (39), 71 (63), 43 (100), 41 (53), from **2**; isobutyric acid methyl ester (t_R 2.368 min) *m/z* $[M - H]^-$ 101 (32), 87 (48), 71 (50), 43 (100), from **1**; and acetic acid methyl ester (t_R 2.100 min) *m/z* $[M - H]^-$ 73 (57), 59 (100), 45 (75), 41 (58), from **7**. The ether extract

Table 1. ^{13}C NMR Data of Compounds **1–7** (C_5D_5N, 150 MHz)[a]

carbon[b]	1	2	3	4	5	6	7
Fuc-1	101.7	101.4	101.7	104.3	104	104.3	104.3
2	73.6	73.6	73.6	80.7	80.3	80.3	80.3
3	76.6	76.6	76.6	73.5	73.4	73.3	73.2
4	73.7	73.6	73.7	73.1	73.0	73.0	73.0
5	71.3	71.3	71.2	70.6	70.8	70.8	70.8
6	17.2	17.2	17.2	17.4	17.4	17.4	17.4
Rha-1	100.3	100.2	100.2	98.7	98.8	98.8	98.8
2	69.9	69.9	69.8	73.5	73.9	73.8	73.9
3	77.9	78.0	77.8	69.8	69.9	69.7	69.7
4	77.6	77.3	77.7	80.3	80.3	80.8	80.7
5	68.0	68.2	68.0	70.8	68.2	70.7	70.7
6	18.8	18.8	18.8	19.4	19.5	19.4	18.9
Rha′-1	99.0	99.1	99.0	99.4	99.1	99.3	99.4
2	72.9	72.8	72.9	72.9	73.1	73.1	73.1
3	79.6	79.6	79.8	79.4	79.0	78.5	80.2
4	79.1	80.7	79.1	80.1	80.1	79.4	79.7
5	68.5	68.5	68.5	68.3	68.6	68.6	68.2
6	18.7	19.2	18.7	18.9	18.9	18.9	19.2
Rha″-1	100.4	103.8	100.3	103.6	100.3	100.2	103.6
2	71.2	70.1	71.2	70.1	74.1	71.2	70.3
3	73.1	73.3	73.0	73.9	68.2	73.6	73.4
4	71.3	71.6	71.3	71.7	75.2	73.1	71.6
5	70.9	68.2	70.8	68.5	68.5	70.8	68.4
6	18.9	17.7	19.1	17.8	18.0	18.6	18.6
Rha‴-1	104.5	104.5	104.5	104.3	104.8	104.5	104.5
2	72.3	72.7	72.3	72.6	72.2	72.3	72.7
3	72.6	72.6	72.4	72.5	72.6	72.6	72.6
4	73.6	73.6	73.6	73.2	73.5	73.6	73.2
5	70.8	70.7	70.8	68.6	68.6	68.4	68.7
6	19.1	18.9	19.2	18.5	18.6	18.3	17.7
Ag-1	174.8	174.8	173.3	173.1	173.1	173.1	173.1
2	33.8	33.8	33.8	34.3	33.2	33.2	34.4
11	79.6	79.6	79.6	82.4	82.4	82.4	82.4
16	14.5	14.5	14.3	14.3	14.3	14.3	14.3
Cna-1	166.5	166.2	166.6	166.4	166.8	166.6	166.4
2	118.3	118.4	118.3	118.4	118.5	118.3	118.3
3	146.1	145.4	146.0	145.5	145.6	146.1	145.6
Iba-1	176.5	176.3					
2	34.4	34.5					
3	19.2	18.8					
3′	18.3	19.2					
Mba-1	175.4		175.4	175.4	175.4	175.4	
2	41.5		41.5	41.5	41.3	41.5	
CH_3-2	17.0		16.9	16.8	16.8	16.8	
4	11.9		11.8	11.8	11.8	11.8	
Dodeca-1			174.8	173.2	173.5	173.6	172.9
2			34.5	34.6	34.6	34.4	34.4
12			14.5	14.3	14.3	14.3	14.3
Aa-1							176.4
2							19.5
Ba-1		176.0					
2		34.3					
4		19.0					

[a] Chemical shifts (δ) are in ppm relative to TMS. All assignments are based on HMQC and HMBC experiments. [b] Abbreviations: Fuc, fucose; Rha, rhamnose; Ag, 11-hydroxyhexadecanoyl; Dodeca, *n*-dodecanoyl; Mba, (*S*)-2-methylbutanoyl; Iba, isobutanoyl; Cna, *trans*-cinnamoyl; Ba, *n*-butanoyl; Aa, acetonoyl.

Table 2. ^{1}H NMR Data of Compounds **1**−**3** (C_5D_5N, 600 MHz)[a]

proton[b]	1	2	3
Fuc-1	4.80, d (7.9)	4.80, d (7.9)	4.79, d (7.8)
2	4.50, dd (7.9, 9.5)	4.51, dd (7.9, 9.5)	4.51, dd (7.8, 9.4)
3	4.16, dd (9.5, 3.4)	4.16, dd (9.5, 3.4)	4.17 *
4	3.90, d (3.4)	3.90, d (3.4)	3.90 *
5	3.79, q (6.4)	3.79, q (6.5)	3.78, q (6.2)
6	1.49, d (6.4)	1.49, d (6.4)	1.49, d (6.2)
Rha-1	6.31, d (1.5)	6.31, d (1.5)	6.32, br s
2	5.29, br s	5.29, brs	5.29, br s
3	5.60, dd (3.3, 10.0)	5.62, dd (3.0, 10.0)	5.62 *
4	4.61, dd (10.0, 10.0)	4.66, dd (10.0, 10.0)	4.62,dd (10.0, 10.0)
5	5.00, dd (10.0, 6.3)	5.01, dd (10.0, 6.3)	5.00, dd (10.0, 6.0)
6	1.56, d (6.3)	1.57, d (6.3)	1.54, d (6.0)
Rha′-1	5.58, br s	5.60, br s	5.59, br s
2	5.79, br s	5.79, br s	5.80, br s
3	4.57, dd (3.0, 9.5)	4.59, dd (3.0, 9.5)	4.64, dd (3.0, 9.6)
4	4.24, dd (9.5, 9.5)	4.24, dd (9.5, 9.5)	4.24 *
5	4.34, dd (9.5, 6.3)	4.36, dd (9.5, 6.2)	4.34 *
6	1.54, d (6.3)	1.58, d (6.2)	1.55, d (6.0)
Rha″-1	5.83, br s	5.90, brs	5.86, br s
2	6.04, br s	4.87, brs	6.06, br s
3	5.81, dd (3.0, 10.0)	5.82, dd (3.0, 10.0)	5.85 *
4	4.26, dd 10.0, 10.0)	6.01, dd (10.0, 10.0)	4.23, dd (10.0, 10.0)
5	4.41, dd (10.0, 6.3)	4.45, dd (10.0, 6.3)	4.43, dd (10.0, 6.0)
6	1.69, d (6.3)	1.39, d (6.3)	1.68, d (6.0)
Rha‴-1	5.58, br s	5.63, br s	5.58, br s
2	4.72, br s	4.74, dd (1.8, 3.4)	4.73, br s
3	4.30 *	4.40, dd (3.4, 9.5)	4.30 *
4	4.18, dd (9.5, 9.5)	4.18, dd (9.5, 9.5)	4.17 *
5	4.25, dd (9.5, 6.1)	4.25, dd (9.5, 6.1)	4.25 *
6	1.69, d (6.1)	1.69, d (6.1)	1.70, d (6.2)
Ag-2	2.23, ddd (4.3, 7.1, 15.5)	2.25, ddd (4.2, 7.0, 15.0)	2.41, m
	2.88, ddd (4.3, 7.1, 15.5)	2.86, ddd (4.2, 7.0, 15.0)	2.25, m
11	3.87, m	3.86, m	3.85, m
16	0.88, t (6.8)	0.87, t (7.2)	0.84, t (7.2)
Cna-2	6.82, d (15.9)	6.50, d (15.9)	6.82, d (15.9)
3	7.90, d (15.9)	7.80, d (15.9)	7.91, d (15.9)
Iba-2	2.54, m	2.60, m	
3	1.13, d (7.0)	1.17, d (7.2)	
3′	1.14, d (7.0)	1.17, d (7.2)	
Mba-2	2.38, t q (7.0, 7.0)		2.34, t q (7.0, 7.0)
2-CH3	1.07, d (7.0)		1.12, d (6.5)
4	0.99, t (7.4)		0.87, t (7.4)
Dodeca-2			2.88, t (7.0)
12			1.01, t (7.0)
Ba-2		1.10, t (7.0)	
4		0.96, t (6.9)	

[a] Chemical shifts (δ) are in ppm relative to TMS. The spin coupling (J) is given in parentheses (Hz). Chemical shifts marked with an asterisk (*) indicate overlapped signals. Spin-coupled patterns are designated as follows: s, singlet; br s, broad singlet; d, doublet; t, triplet; m, multiplet; q, quartet. All assignments are based on ^{1}H−^{1}H TOCSY experiments. [b] Abbreviations: Fuc, fucose; Rha, rhamnose; Ag, 11-hydroxyhexadecanoyl; Iba, isobutanoyl; Mba, (*S*)-2-methylbutanoyl; Cna, *trans*-cinnamoyl; Dodeca, *n*-dodecanoyl; Ba, *n*-butanoyl.

(1.1 mg) of the alkaline hydrolysis of **5** was purified by RP-C_{18} chromatography, eluted with MeOH/H_2O (25:75), to give 2-methylbutyric acid (0.3 mg). This was proved to be in the *S* configuration by comparing the optical rotation ($[\alpha]_D^{25}$ + 19.0) with that of authentic (*S*)-2-methylbutyric acid.

Acid Hydrolysis. The ether-insoluble layer from the alkaline hydrolysis of **5** was extracted with *n*-BuOH (30.0 mL) to afford **10** (*9, 10*), which was methylated with diazomethane. The methylation product was hydrolyzed by 1 N H_2SO_4 and then extracted with ether (30.0 mL) to yield **11** (11-hydroxyhexadecanoic acid methyl ester, 12%). A solution of (*R*)-methoxyphenylacetic acid (12.0 mg, MPA) and 4-dimethylaminopyridine (10.0 mg, DMAP) in CH_2Cl_2 (1.0 mL) was added to CH_2Cl_2 (1.5 mL) containing **11** (2.0 mg), followed by *N*,*N*-dicyclohexylcarbodiimide (10.0 mg, DCC). The mixture was stirred for 17.0 h at 25.0 °C. EtOAc (30.0 mL) was added to quench the reaction and filtered. The filtrate was concentrated and purified by silica gel chromatography eluted with cyclohexane/ethyl acetate (95:5) to give **12** [2.6 mg, 94%; 11-(*R*-MPA)-hexadecanoic acid methyl ester]. Treatment of **11** with (*S*)-MPA by the same procedure yielded **13** [2.3 mg, 85%; 11-(*S*-MPA)-hexadecanoic acid methyl ester]. The chemical shift differences of **12** and **13** ($\Delta\delta_{H10}^{RS} = +0.06$, $\Delta\delta_{H12}^{RS} = -0.13$, $\Delta\delta_{H16}^{RS} = -0.07$ ppm) (*10–14*) made it possible to conclude the chiral C-11 of **11** is in the *S* configuration, the same as in the literature (*15*). The *n*-BuOH layer of acid hydrolysis was neutralized by passage through an ion-exchange resin (Amberlite MB-3) column and concentrated to yield a saccharide residue, which was treated with water (0.05 mL) and pyridine (0.03 mL) at 60 °C for 1 h with stirring. After the solvent was evaporated and the reaction mixture was dried, pyridine (0.5 mL), hexamethyldisilazane (0.8 mL), and trimethylsilyl chloride (0.4 mL) were added to the residue. The reaction mixture was heated at 60 °C for 30 min. Under the same conditions as above, the supernatant was applied to GC-MS to afford D-fucose [t_R 4.57 min, $[\alpha]_D^{25}$ +66.4 (*c* 0.8, H_2O)] and L-rhamnose [t_R 5.09 min, $[\alpha]_D^{25}$ −9.7 (*c* 1.0, H_2O)], by comparison with authentic samples.

11-(*R*-MPA)-hydroxyhexadecanoic acid methyl ester (12): colorless oil ($CHCl_3$), $[\alpha]_D^{25}$ −2.0 (*c* 0.10, $CHCl_3$); IR ν_{max} (KBr) 3442, 2927, 2855, 1743, 1261, 802 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 7.44 (m, 2, $C_6\underline{H}_2$), 7.34 (m, 3, $C_6\underline{H}_3$), 4.90 (m, 1, OC$\underline{H}$-11), 4.73 (s, 1, OC$\underline{H}$), 3.67 (s, 3, OC$\underline{H}_3$), 3.41 (s, 3, OC$\underline{H}_3$), 2.30 (t, 2, J = 7.4 Hz, OCOC$\underline{H}_2$-2), 1.67 (m, 2, C$\underline{H}_2$-10), 1.41 (m, 2, C$\underline{H}_2$-12), 0.77 (t, 3, J = 7.1 Hz, C$\underline{H}_3$-16); ESIMS *m*/*z* 457 $[M + Na]^+$; 435 $[M + H]^+$.

11-(*S*-MPA)-hdroxyhexadecanoic acid methyl ester (13): colorless oil ($CHCl_3$), $[\alpha]_D^{25}$ +1.4 (*c* 0.20, $CHCl_3$); IR ν_{max} (KBr) 3453, 2961, 2926, 2852, 1742, 1261 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ_H 7.44 (m, 2, $C_6\underline{H}_2$), 7.34 (m, 3, $C_6\underline{H}_3$), 4.90 (m, 1, OC$\underline{H}$-11), 4.73 (s, 1, OC$\underline{H}$), 3.67 (s, 3, OC$\underline{H}_3$), 3.41 (s, 3, OC$\underline{H}_3$), 2.30 (t, 2, J = 7.6 Hz, OCOC$\underline{H}_2$-2), 1.61 (m, 2, C$\underline{H}_2$-10), 1.54 (m, 2, C$\underline{H}_2$-12), 0.84 (t, 3, J = 7.1 Hz, C$\underline{H}_3$-16); ESIMS *m*/*z* 457 $[M + Na]^+$.

Bioassay. The assay of cytotoxic activity against laryngeal carcinoma (Hep-2) cells of compounds **1**–**9** was performed according to the published method (*4*).

RESULTS AND DISCUSSION

Structure Elucidation of Seven Resin Glycosides Compounds. The 95% EtOH extract of the dried tubers of *I. batatas* was partitioned between $CHCl_3$ and water to provide a jalapin-like fraction. To examine the presence of resin glycosides, the fraction was subjected to alkaline and acid hydrolysis, successively. GC-MS analysis of the ether-soluble fraction of the alkaline hydrolysis products demonstrated the presence of six peaks identified as acetic acid, *n*-butyric acid, isobutyric acid, (*S*)-2-methylbutyric acid, *n*-dedocanoic acid, and *trans*-cinnamic acid by comparison of their mass spectra, retention times, and optical rotations with those of authentic samples (*7*). The alkaline hydrolysis of the ether-insoluble fraction gave simonic acid B (**10**) (*8*), which has also been obtained previously from *I. batatas* (cv. Simon). The lactone linkages of compounds **1**−**3** were all at C-3 of rhamnose (Rha); the difference was the substituents in Rha′ and Rha″, whereas by comparison with **1**–**3**, the lactone linkages of compounds **4**–**7** were at C-2 of the Rha unit and the difference was also the substituents in Rha′ and Rha″, which were the structural characteristics of these compounds.

Batatoside A (**1**), an amorphous white powder, gave a quasi-molecular ion at *m*/*z* 1267.6425 $[M - H]^-$ ($C_{64}H_{99}O_{25}$) in the negative-ion HRESIMS. Alkaline hydrolysis with 5% KOH afforded an organic acid mixture. The organic acids were identified as isobutyric acid (Iba), (*S*)-2-methylbutyric acid (Mba), and *trans*-cinnamic acid (Cna), respectively, by GC-MS and optical rotation, when compared with authentic samples. The ^{1}H NMR spectrum of **1** showed a pair of *trans*-coupled olefinic protons at δ_H 6.82 (d, J = 15.9 Hz, H-2 of Cna) and 7.90 (d, J = 15.9 Hz, H-3 of Cna), and 7.27–7.45 (m, C_6H_5)

Table 3. ^{1}H NMR Data of Compounds **4**−**7** (C_5D_5N, 600 MHz)[a]

proton[b]	4	5	6	7
Fuc-1	4.75, d (7.5)	4.75, d (7.5)	4.75, d (7.4)	4.76, d (7.4)
2	4.16, dd (7.5, 9.4)	4.16, dd (7.5, 9.4)	4.18, dd (7.4, 9.4)	4.18, dd (7.4, 9.4)
3	4.08, dd (9.4, 3.5)	4.08, dd (9.4, 3.5)	4.11, dd (9.4, 3.5)	4.11, dd (9.4, 3.0)
4	3.98, d (3.5)	3.98, d (3.5)	3.98, d (3.5)	3.99, d (3.0)
5	3.77, q (6.4)	3.77, q (6.4)	3.77, q (6.4)	3.78, q (6.9)
6	1.50, d (6.4)	1.50, d (6.4)	1.50, d (6.4)	1.50, d (6.4)
Rha-1	5.48, br s	5.47, br s	5.48, br s	5.50, d (2.0)
2	5.98, br s	5.91, br s	5.97, br s	5.97, dd (2.0, 3.2)
3	5.02, dd (3.3, 9.5)	5.01, dd (3.2, 9.5)	5.05, dd (3.2, 9.5)	5.03, dd (3.2, 9.4)
4	4.20, dd (9.5, 9.5)	4.21, dd (9.5, 9.5)	4.22, dd (9.5, 9.5)	4.23, dd (9.4, 9.4)
5	4.27, dd (9.5, 6.2)	4.44, dd (9.5, 6.1)	4.27, dd (9.5, 6.0)	4.27, dd (9.4, 6.3)
6	1.61, d (6.2)	1.62, d (6.1)	1.60, d (6.0)	1.64, d (6.3)
Rha′-1	6.05, br s	6.11, br s	6.03, br s	6.11, d (1.8)
2	5.96, br s	5.99, br s	6.01, br s	6.04, br s
3	4.68, dd (3.0, 10.0)	4.65, dd (3.0, 9.1)	4.61, dd (3.0, 9.0)	4.67, dd (3.0, 8.9)
4	4.29, dd (10.0, 10.0)	4.32, dd (9.1, 9.3)	4.34, dd (9.0, 9.0)	4.37, dd (8.9, 8.9)
5	4.37, dd (10.0, 6.2)	4.37, dd (9.3, 5.9)	4.44, dd (9.0, 5.7)	4.30, dd (8.9, 6.3)
6	1.65, d (6.2)	1.67, d (5.9)	1.55, d (5.7)	1.66, d (6.3)
Rha″-1	5.96, br s	5.94, br s	5.89, br s	5.99, br s
2	4.97, br s	5.98, br s	6.15, br s	4.98, br s
3	5.91, dd (3.3, 10.0)	4.68, dd (3.0, 9.3)	5.92, dd (3.4, 9.3)	5.93, dd (3.0, 10.0)
4	6.09, dd (10.0, 10.0)	5.78, dd (9.3, 9.3)	4.21, dd (9.3, 9.3)	6.06, dd (10.0, 10.0)
5	4.45, dd (10.0, 6.2)	4.37, dd (9.3, 6.2)	4.47, dd (9.3, 6.0)	4.48, dd (10.0, 6.1)
6	1.45, d (6.2)	1.65, d (6.2)	1.58, d (6.0)	1.60, d (6.1)
Rha‴-1	5.67, br s	5.67, br s	5.57, br s	5.65, d (1.0)
2	4.78, br s	4.78, br s	4.71, br s	4.79, br s
3	4.41, dd (3.0, 9.3)	4.39, dd (3.3, 9.0)	4.30 *	4.48 *
4	4.29, dd (9.3,9.3)	4.19, dd (9.0, 9.0)	4.17 *	4.21 *
5	4.50, dd (9.3,6.0)	4.26, dd (9.0, 6.0)	4.25 *	4.48 *
6	1.54, d (6.0)	1.54, d (6.0)	1.70, d (6.2)	1.51, d (6.4)
Ag-2	2.37, ddd (4.2,7.0,15.0)	1.91, m	2.23 *	2.27, ddd (4.3,7.0,15.0)
	2.22, ddd (4.2,7.0,15.0)	2.23, m	2.37 *	2.43, ddd (4.3,7.0,15.0)
11	3.87, m	3.85, m	3.86, m	3.88, m
16	0.84, t (7.5)	0.87, t (7.0)	0.83, t (7.3)	0.85, t (7.2)
Cna-2	6.56, d (15.9)	6.54, d (15.9)	6.81, d (16.0)	6.55, d (15.9)
3	7.82, d (15.9)	7.80, d (15.9)	7.90, d (16.0)	7.82, d (15.9)
Mba-2	2.36, m	2.34, m	2.37 *	
2-CH_3	1.06, d (7.0)	1.09, d (7.1)	1.07, d (7.0)	
4	0.86, t (7.0)	0.85, t (7.3)	0.84, t (7.4)	
Dodeca-2	2.47, t (7.3)	2.47, m	2.43 *	2.64, t (7.0)
12	0.87, t (6.8)	0.87, t (6.8)	0.85, t (7.2)	0.88, t (7.2)
Aa-2				1.12

[a] Chemical shifts (δ) are in ppm relative to TMS. The spin coupling (J) is given in parentheses (Hz). Chemical shifts marked with an asterisk (*) indicate overlapped signals. Spin-coupled patterns are designated as follows: s, singlet; br s, broad singlet; d, doublet; t, triplet; m, multiplet; q, quartet. All assignments are based on ^{1}H−^{1}H TOCSY experiments. [b] Abbreviations: Fuc, fucose; Rha, rhamnose; Ag, 11-hydroxyhexadecanoyl; Aa, acetonoyl; Mba, (*S*)-2-methylbutanoyl; Cna, *trans*-cinnamoyl; Dodeca, *n*-dodecanoyl.

due to five phenyl protons, indicating the presence of a 3-phenylprop-2-enoyl (Cna) moiety. The methyl protons at δ_H 1.13 (d, J = 7.0 Hz, CH_3-3 of Iba) and 1.14 (d, J = 7.0 Hz, CH_3′-3 of Iba) showed HMBC correlations with the carbon signals at δ_C 176.5 (C-1 of Iba) and 34.4 (C-2-Iba), suggesting the presence of an isobutyryl moiety. The protons at δ_H 0.99 (t, J = 7.4 Hz, H-4 of Mba), 1.07 (d, J = 7.0 Hz, CH_3′-2 of Mba), and 2.38 (tq, J = 7.0 and 7.0 Hz, H-2 of Mba) displayed in one spin system from the TOCSY spectrum, consistent with the occurrence of an (*S*)-2-methylbutyryl moiety.

Diagnostic signals of the aglycone of **1** were the methyl triplet at δ_H 0.88 (t, J = 6.8 Hz, H-16 of aglycone), the methylenes at δ_H 2.23 (ddd, J = 4.3, 7.1 and 15.5 Hz, H-2a of aglycone) and 2.88 (ddd, J = 4.3, 7.1, and 15.5 Hz, H-2b of aglycone), and the oxygenated methane at δ_H3.87 (m, H-11 of aglycone). Three substituents were identified in the NMR spectra to be the same as those from the analysis results of GC-MS. The lactone position at C-3 of rhamnose was proved by cross-peak in the HMBC spectrum. The acylation pattern of **1** was anchored by HMBC correlations from the proton of sugar junction resonances to the carbonyl of three substituents. The H-2 of rhamnose′ (δ_H 5.79) showed HMBC cross-peaks with resonances at δ_C 175.4 ([C-1-(*S*)-2-methylbutanoyl]), and an HMBC correlation was observed from the H-2 of rhamnose″ (δ_H 6.04) and H-3 of rhamnose″ (δ_H 5.81) to δ_C 166.5 (C-1 of *trans*-cinnamoyl) and δ_C 176.5 (C-1 of isobutanoyl), respectively (**Figure 1**). The assignments of the carbon and proton signals were based on TOCSY, HMQC, and HMBC spectra, which are listed in **Tables 1** (^{13}C NMR) and **2** (^{1}H NMR). The structure of **1** was finally established as (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[2-*O*-*trans*-cinnamoyl-3-*O*-isobutanoyl-α-L-rhamnopyranosyl-(1→4)]-*O*-[2-*O*-(*S*)-2-methylbutyryl]-α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,3″-ester.

Batatoside B (**2**) was obtained as an amorphous white powder. Compound **2** showed a quasi-molecular ion at *m*/*z* 1253.5944 $[M - H]^-$ ($C_{63}H_{97}O_{25}$) in the negative-ion HRESIMS. The difference of 14 mass units observed in the mass spectra of compounds **1** and **2** indicated there is a CH_2 different between them. GC-MS analysis of the alkaline hydrolysis mixture of **2** showed the presence of cinnamic acid, isobutyric acid, and *n*-butyric acid, and the aqueous layer afforded simonic acid B. The linkage sites of the three substituent groups and the lactone site were determined by the correlations in HMBC spectrum.

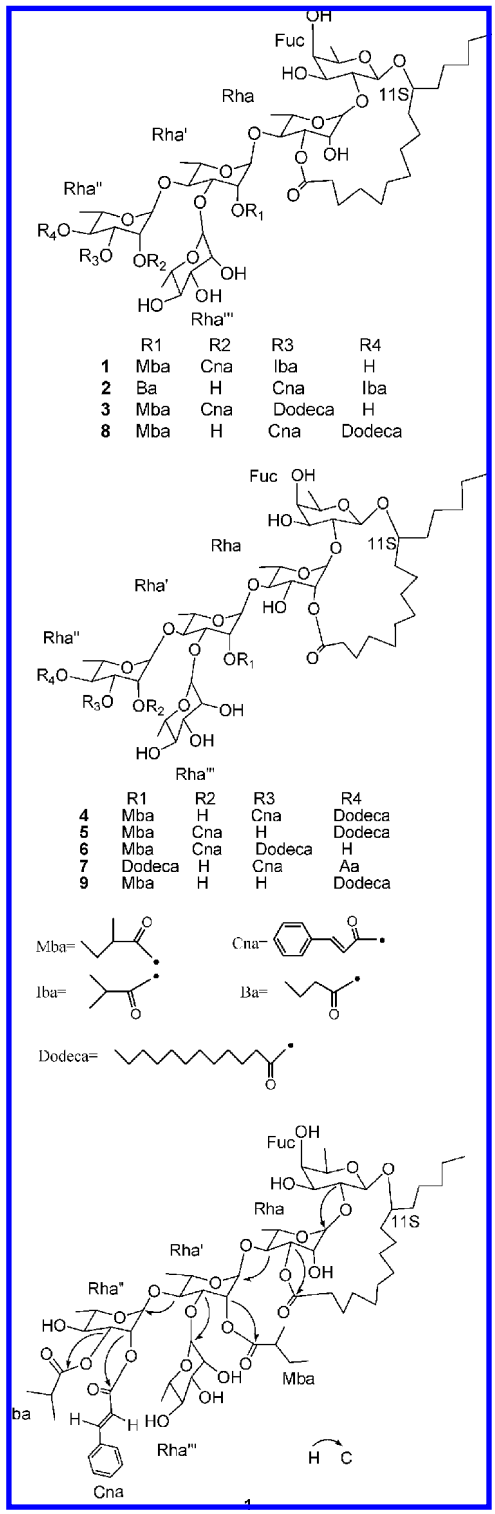

Figure 1. Key HMBC correlations from H to C for compound **1**.

The H-3 signal of rhamnose displayed a HMBC correlation to a carbonyl at δ_C 174.8 (C-1 of aglycon), whereas the H-2 resonance of rhamnose′ showed a HMBC correlation to the carbonyl at δ_C 176.0 [C-1 of (*S*)-2-methylbutanoyl]. Two cross-peaks have also been observed between protons of H-3 and H-4 of rhamnose″ (δ_H 5.82 and 6.01) and the carbon atoms of δ_C 166.2 (C-1 of *trans*-cinnamoyl) and 176.3 (C-1 of isobutanoyl), respectively. The structure of **2** was identified as (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[3-*O*-*trans*-cinnamoyl-4-*O*-isobutanoyl-α-L-rhamnopyranosyl-(1→4)]-*O*-(2-*O*-butanoyl)-

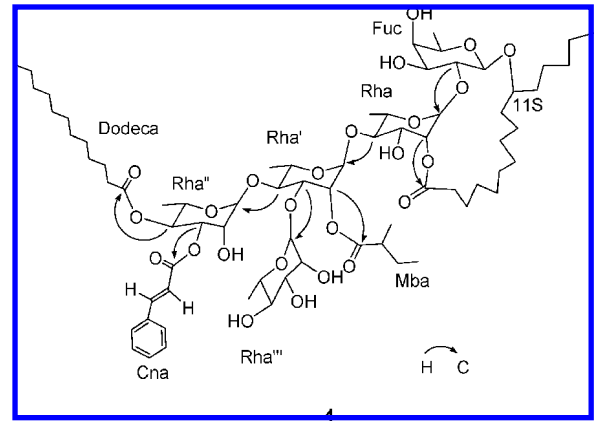

Figure 2. Key HMBC correlations from H to C for compound **4**.

α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,3″-ester.

Batatoside C (**3**) was obtained as an amorphous white powder and displayed a quasi-molecular ion peak $[M + Cl]^-$ at *m*/*z* 1415.7580 in the negative HRESIMS, consistent with a molecular formula of $C_{72}H_{116}O_{25}$. Some of the ^{13}C and ^{1}H NMR signals of **3** were similar to those of **1** (**Tables 1** and **2**). For this reason, the acylation sites of **3** were assumed to be the same as those of **1**, which were supported by the following HMBC cross-peaks: δ_H 5.62 (H-3 of rhamnose) with δ_C 173.3 (C-1 of aglycon); δ_H 5.80 (H-2 of rhamnose″) with δ_C 175.4 ([C-1 of (*S*)-2-methylbutanyol]); and δ_H 6.06 (H-2 of rhamnose″) and 5.85 (H-3 of rhamnose″) with δ_C 166.6 (C-1 of *trans*-cinnamoyl) and 174.8 (C-1 of *n*-dedocanoyl), respectively. The assignments of the ^{13}C and ^{1}H NMR signals were determined according to the corrolations in TOCSY, HMQC, and HMBC spectra (**Tables 1** and **2**). On the basis of the above results, the structure of **3** was confirmed as (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[2-*O*-*trans*-cinnamoyl-3-*O*-*n*-dedocanoyl-α-L-rhamnopyranosyl-(1→4)]-*O*-[2-*O*-(*S*)-2-methylbutyryl]-α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,3″-ester.

Batatoside D (**4**), batatoside E (**5**), and batatoside F (**6**) were also obtained as amorphous white powders. The molecular formulas of **4**–**6** were all $C_{72}H_{116}ClO_{25}$, according to the quasi-molecular ion peaks $[M + Cl]^-$ at *m*/*z* 1415.7487, 1415.7584, and 1415.7305 in their respective negative HRESIMS. Alkaline hydrolysis of compounds **4**−**6** afforded the same organic acids as those of **3**. The lactone linkages to the positions of C-2 in the rhamnoses, which showed a major difference between **4**−**6** and **3**, have been proved by the correlations in their HMBC spectrum. Accordingly, Ccompounds **4**−**6** and **3** could be thought to be regioisomers.

The substituents as well as their positions and lactone linkage of **4**−**6** were determined by HMBC spectrum. A HMBC correlation of **4** (**Figure 2**) was observed from δ_H 5.98 (H-2 of rhamnose) to δ_C 173.1 (C-1 of the aglycon). The H-2 signal of rhamnose′ (δ_H 5.96) showed a cross-peak with δ_C 175.4 ([C-1 of (*S*)-2-methylbutanoyl]). H-3 of rhamnose″ (δ_H 5.91) and H-4 of rhamnose″ (δ_H 6.09) displayed HMBC correlations with δ_C 166.4 (C-1 of *trans*-cinnamoyl) and δ_C 173.2 (C-1 of *n*-dedocanoyl). The unambiguous assignments of signals were achieved from the TOCSY, HMQC, and HMBC spectra (**Tables 1** and **3**). The structure of **4** was established as (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[3-*O*-*trans*-cinnamoyl-4-*O*-*n*-dedocanoyl-α-L-rhamnopyranosyl-(1→4)]-*O*-[2-*O*-(*S*)-2-methylbutyryl]-α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,2″-ester.

The linkage sites of **5** were also determined by HMBC correlations, in which H-2 of rhamnose (δ_H 5.91) showed a correlation to the carbon atom in the carbonyl group at δ_C 173.1 (C-1 of aglycon), and a correlation was observed from H-2 of rhamnose′ (δ_H 5.99) to δ_C 175.4 (C-1 of (*S*)-2-methylbutanoyl). The protons of H-2 of rhamnose″ (δ_H 5.98) and H-4 of rhamnose″ (δ_H 5.78) displayed cross-peaks with δ_C 166.8 (C-1 of *trans*-cinnamoyl) and δ_C 173.5 (C-1 of dodecanoyl), respectively. The structure of **5** was concluded as (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[2-*O*-*trans*-cinnamoyl-4-*O*-*n*-dedocanoyl-α-L-rhamnopyranosyl-(1→4)]-*O*-[2-*O*-(*S*)-2-methylbutyryl]-α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,2″-ester.

The linkage positions of three constituents groups and lactone site of **6** were also determined from the HMBC spectrum, in which proton H-2 of rhamnose (δ_H 5.97) showed a cross-peak with δ_C 173.1 (C-1 of aglycon), and H-2 of rhamnose′ displayed a correlation to a carbonyl at δ_C 175.4 ([C-1 of (*S*)-2-methylbutanoyl]). In addition, the protons at δ_H 6.15 (H-2 of rhamnose″) and 5.92 (H-3 of rhamnose″) correlated with δ_C 166.6 (C-1 of *trans*-cinnamoyl) and 173.6 (C-1 of dodecanoyl) in the HMBC spectrum, respectively. The structure of **6** was elucidated as (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[2-*O*-*trans*-cinnamoyl-3-*O*-dodecanoyl-α-L-rhamnopyranosyl-(1→4)]-[2-*O*-(*S*)-2-methylbutyryl]-α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,2″-ester.

Batatoside G (**7**) was obtained as an amorphous white powder. The molecular formula of **7** was $C_{69}H_{110}O_{25}$ according to quasi-molecular ion peaks $[M + Cl]^-$ at *m*/*z* 1373.6898 in the negative HRESIMS. The alkaline hydrolysis of **7** afforded *n*-dedocanoic acid, acetic acid, and *trans*-cinnamic acid, by comparison with authentic samples proved by GC-MS. The aqueous layer afforded simonic acid B. The linkage sites of three constituents groups and lactone site were determined by the following HMBC correlations. An HMBC correlation was observed from the H-2 of rhamnose (δ_H 5.03) to δ_C 173.1 (C-1 of aglycon). H-2 of rhamnose′ (δ_H 6.04) displayed a HMBC correlation to δ_C 172.9 (C-1 of dodecanoyl). H-3 (δ_H 5.93) and H-4 (δ_H 6.06) of rhamnose″ showed HMBC cross-peaks with δ_C 166.4 (C-1 of *trans*-cinnamoyl) and 176.4 (C-1 of acetyl), respectively. Accordingly, the structure of **7** was concluded to be (*S*)-jalapinolic acid 11-*O*-α-L-rhamnopyranosyl-(1→3)-*O*-[3-*O*-*trans*-cinnamoyl-4-*O*-acetanoyl-α-L-rhamnopyranosyl-(1→4)]-(2-*O*-*n*-dedocanoyl]-α-L-rhamnopyranosyl-(1→4)-*O*-α-L-rhamnopyranosyl-(1→2)-*O*-β-D-fucopyranoside, intramolecular 1,2″-ester.

Cytotoxic Activity Assay. The assay of cytotoxic activity against laryngeal carcinoma (Hep-2) cells of compounds **1**–**9** was performed according to the published method (*4*). The results indicated that compound **5** showed weak cytotoxic activities against Hep-2 cells; its ED_{50} value was 6 μg/mL. The ED_{50} values of other compounds were >40 μg/mL, which could be thought to be inactive, whereas that of the positive drug control, vinblastine, was 0.002 μg/mL.

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Supporting Information Available: Spectra (HRESIMS, ^1H, ^{13}C NMR, TOCSY, HMQC, HMBC) of batanosides A−G (**1**–**7**). This material is available free of charge via the Internet at http://pubs.acs.org.

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