Triterpenoid Saponins from *Rubus ellipticus* var. *obcordatus*

Wei Li,*,† Hongwei Fu,†,‡ Hong Bai,§ Tatsunori Sasaki,† Hiroyoshi Kato,† and Kazuo Koike†

Faculty of Pharmaceutical Sciences, Toho University, Miyama 2-2-1, Funabashi, Chiba 274-8510, Japan, Zhejiang Provincial Key Laboratory for Chinese Medicine Screening, Exploitation & Medicinal Effectiveness Appraise for Cardio-cerebral Vascular & Nervous System, The Key Laboratory of Biomedical Engineering Ministry of Education, China, Department of Biomedical Engineering, Zhejiang University, Hangzhou 310027, People's Republic of China, and Institute of Materia Medica, Shandong Academy of Medical Sciences, Jinan 250062, People's Republic of China

Received April 30, 2009

Ten new triterpenoid saponins (**1**−**10**), named rubusides A−J, and 21 known saponins (**11**−**31**) were isolated from the roots of *Rubus ellipticus* var. *obcordatus*. The structures of **1**−**10** were established on the basis of spectroscopic analyses, mainly NMR and MS, and chemical degradations. The compounds demonstrated inhibitory activities against α-glucosidase with IC_{50} values in the range 0.65−3.09 mM.

Rubus ellipticus Smith var. *obcordatus* Focke (Rosaceae) is widely distributed in Sichuan, Yunnan, Shanxi, and Guizhou Provinces in China. The roots have been used in folk medicine for the treatment of bacterial dysentery, tonsillitis, and icteric hepatitis.[1] Only a few compounds have been reported from *R. ellipticus*.[2,3] The potential medicinal importance and our interest in the chemistry of saponins prompted us to investigate the triterpenoid saponins of this plant, which resulted in the isolation of nine new ursane-type triterpenoids (**1**−**8** and **10**), one new lupane-type triterpenoid (**9**), and 21 known saponins. The saponins were classified into seven types: 12,18-dien ursanes (**1**−**3** and **11**), 12,19-dien ursanes (**4**, **5**, and **12**), 12,19(29)-dien ursanes (**6**, **13**, and **14**), 11,13(18)-dien ursanes (**7** and **8**), lupane (**9**), 19α-hydroxy oleanes (**15**−**17**), and 19α-hydroxy ursanes (**10** and **18**−**31**). This paper deals with the isolation and structure elucidation of the new compounds and their inhibitory activities against α-glucosidase.

Results and Discussion

A methanolic extract of the roots of *R. ellipticus* var. *obcordatus* was partitioned between *n*-BuOH and H_2O. The *n*-BuOH-soluble fraction was subjected to a Diaion HP-20 column, followed by washing with MeOH and H_2O mixtures in different ratios. Separations of the 70% MeOH- and MeOH-eluted fractions using a combination of silica gel column chromatography (CC), ODS CC, and preparative HPLC afforded 31 triterpenoid saponins: 10 new (**1**−**10**) and 21 known (**11**−**31**) compounds.

Compounds **1**−**10**, which we named rubusides A−J, possessed the same glycosyl moiety but different aglycones. On acid hydrolysis, they afforded D-glucose as the component sugar, which was identified by GLC analysis of their trimethylsilyl D-cysteine derivatives.[4] The chemical shifts of the anomeric protons (δ_H 6.30−6.42, d, J = 8.0−8.4 Hz) and carbons (δ_C 95.5−96.5) revealed that the β-glucopyranosyl moieties were attached to the C-28 carboxyl groups of **1**−**10**. The molecular formulas of compounds **1**−**10** were determined primarily by positive-ion HRFABMS.

Rubuside A (**1**) was obtained as an amorphous solid with the molecular formula $C_{36}H_{56}O_9$. The ^{13}C NMR spectrum showed 36 carbon signals including seven methyl (δ_C 17.7, 17.8, 18.6, 18.7, 19.6, 22.2, and 29.4), four olefinic (δ_C 126.6, 133.8, 136.0, and 138.8), of which δ_C 126.6 and 138.8 were typical of a double bond at C-12(13) of an ursane-type triterpene,[5] and a carboxyl signal (δ_C 174.8) (Table 1). The ^1H NMR spectrum of **1** displayed signals corresponding to six tertiary methyl (δ_H 1.05, 1.08, 1.10, 1.18, 1.27, and 1.80) and one secondary methyl (δ_H 1.04) group and one olefinic proton (δ_H 5.70) (Table 2). Signals assignable to H-2 and H-3 of the aglycone were also observed [(δ_H 4.12 (ddd, J = 12.6, 9.4, 4.6 Hz) and 3.41 (d, J = 9.4 Hz)], suggesting that the protons were 2β and 3α, respectively. The NMR data of **1** were similar to those of 2α,3β,23-trihydroxyurs-12,18-dien-28-oic acid 28-*O*-β-D-glucopyranoside (**11**) isolated from the same extract, except for the presence of an α-methyl [δ_H 1.27; δ_C 29.4] at C-4 in **1** instead of a hydroxymethyl as in **11**, which was confirmed by the HMBC correlation between δ_H 1.08 (H-24) and δ_C 29.4 (C-23). The above results indicated that **1** was a 28-*O*-β-D-glucopyranoside of 2α,3β-dihydroxyurs-12,18-dien-28-oic acid (goreishic acid I) (**1a**).[6] Thus, rubuside A (**1**) was 2α,3β-dihydroxyurs-12,18-dien-28-oic acid 28-*O*-β-D-glucopyranoside.

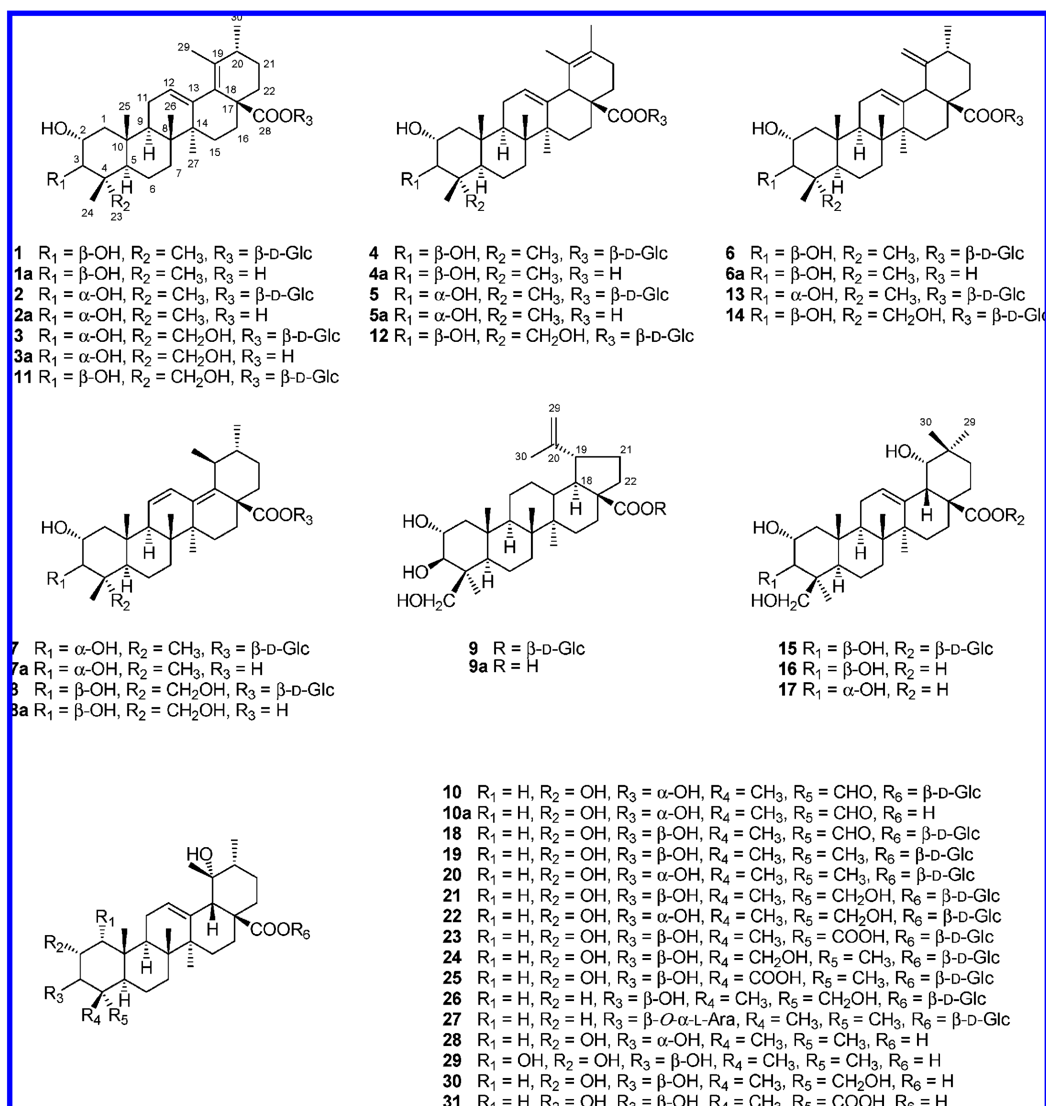
Rubuside B (**2**) and rubuside C (**3**) were assigned the elemental compositions $C_{36}H_{56}O_9$ and $C_{36}H_{56}O_{10}$, respectively, using HRFABMS. The ^1H and ^{13}C NMR data of **2** (Tables 1 and 2) were almost superimposable with those of **1**, except for the A ring. The splitting pattern of H-3 (d, J = 1.8 Hz) suggested that **2** was the C-3 epimer of **1**. Thus, the C-3 hydroxy group of **2** was α-oriented, and the aglycone (**2a**) was 2α,3α-dihydroxyurs-12,18-dien-28-oic acid.[7] Thus, **2** was concluded to be 2α,3α-dihydroxyurs-12,18-dien-28-oic acid 28-*O*-β-D-glucopyranoside. Comparing the NMR spectra of **3** with those of **2** showed that **3** had one more OH group. After several NMR experiments, including DQF-COSY, HMQC, HMBC, and NOESY, it was apparent that the signal due to the α-methyl group at C-4 in **2** was replaced by a signal due to a hydroxymethyl group [δ_H 3.75 and 3.91 (each, d, J = 10.8 Hz), and δ_C 71.3] in **3**. Enzymatic hydrolysis of **3** afforded the aglycone (**3a**), which was confirmed to be 2α,3α,23-trihydroxyurs-12,18-dien-28-oic acid. Thus, **3** was determined to be 2α,3α,23-trihydroxyurs-12,18-dien-28-oic acid 28-*O*-β-D-glucopyranoside.

The molecular formulas of rubuside D (**4**) and rubuside E (**5**) were both established as $C_{36}H_{56}O_9$. The ^1H and ^{13}C NMR data (Tables 1 and 2) of **4** were similar to those of **1**, but they differed by the disappearance of the secondary (C-20) methyl group and the appearance of a tertiary methyl group signal (δ_H 1.59; δ_C 20.4). The second double bond was determined to be at $\Delta_{19,20}$ by long-range correlations between the methyl groups (δ_H 1.59, 1.63) and the olefinic carbons (δ_C 123.7, 128.7) in the HMBC spectrum. Thus, **4** was a 28-*O*-β-D-glucopyranoside of 2α,3β-dihydroxyurs-12,19-dien-28-oic acid (**4a**),[8] and its structure was determined to be 2α,3β-dihydroxyurs-12,19-dien-28-oic acid 28-*O*-β-D-glucopyranoside. Comparison of the NMR data of **5** and **4** indicated that **5** was the

* To whom correspondence should be addressed. Tel: +81-47-4721161. Fax: +81-47-4721404. E-mail: liwei@phar.toho-u.ac.jp.
† Toho University.
‡ Zhejiang University.
§ Shandong Academy of Medical Sciences.

10.1021/np900237a CCC: $40.75 © 2009 American Chemical Society and American Society of Pharmacognosy
Published on Web 10/01/2009

Chart 1



C-3 epimer of **4**. The proton signal assignable to H-3 in **5** was observed at δ_H 3.76 (d, J = 2.5 Hz), indicating that H-3 was β-*o*riented, and the aglycone was 2α,3α-dihydroxyurs-12,19-dien-28-oic acid (**5a**).[8] Thus, compound **5** was concluded to be 2α,3α-dihydroxyurs-12,19-dien-28-oic acid 28-*O*-β-D-glucopyranoside.

Rubuside F (**6**) had the same molecular formula as **4** ($C_{36}H_{56}O_9$). The ^{1}H and ^{13}C NMR spectra of **6** were also similar to those of **4**, except for the presence of signals due to an *exo*-olefin (δ_H 5.05, 5.12; δ_C 110.3), a secondary methyl (δ_H 1.07; δ_C 19.3), and a methine (δ_H 1.87; δ_C 37.4) in **6** instead of the signals of two vinyl methyl groups (δ_H 1.63; δ_C 17.3 and δ_H 1.59; δ_C 20.4) and a tetrasubstituted olefin (δ_C 123.7, 128.7) in **4**. Thus, **6** was a 28-*O*-β-D-glucopyranoside of an ursane-type triterpene having a double bond at C-19(29). Namely, the aglycone of **6** was 2α,3β-dihydroxyurs-12,19(29)-dien-28-oic acid (**6a**). The NMR data of **6** were similar to those of quadranoside VIII (2α,3β,23-trihydroxyurs-12,19(29)-dien-28-oic acid 28-*O*-β-D-glucopyranoside) (**14**) isolated from the same extract, except for the absence of the OH group at C-23. On the basis of the above results, compound **6** was concluded to be 2α,3β-dihydroxyurs-12,19(29)-dien-28-oic acid 28-*O*-β-D-glucopyranoside.

The molecular formula of rubuside G (**7**) was $C_{36}H_{56}O_9$ (HR-FABMS). The ^{13}C NMR data (Table 1) in combination with analysis of the DEPT and HMQC spectra revealed 36 signals due to eight quaternary, 13 methine, eight methylene, and seven methyl carbons, of which 30 were assigned to the aglycone part including a carbonyl carbon at δ_C 176.7. The resonances of two double-bond protons [δ_H 6.51 (dd, J = 10.5, 2.9 Hz) and 5.87 (dd, J = 10.5, 0.7 Hz)] and four carbons [δ_C 126.2, 127.4 and 135.7, 138.3] revealed the presence of a diene moiety. The signals observed at δ 4.41 (ddd, J = 11.7, 3.9, 2.0 Hz) and 3.81 (d, J = 2.0 Hz) placed these at 2β and 3β. The NMR data of **7** were similar to those of **2** except for the positions of two double bonds. The double bonds were placed at $\Delta_{11,12}$ and $\Delta_{13,18}$ as determined by the HMBC correlations between δ_H 2.27 (H-9) and δ_C 127.4 (C-12), between δ_H 6.51 (H-11), 0.90 (H-27) and δ_C 135.7 (C-13), and between δ_H 5.87 (H-12), 0.99 (H-29) and δ_C 138.3 (C-18). Enzymatic hydrolysis of **7** afforded the aglycone (**7a**), which was determined to be 2α,3α-dihydroxyurs-11,13(18)-dien-28-oic acid. Thus, **7** was concluded to be 2α,3α-dihydroxyurs-11,13(18)-dien-28-oic acid 28-*O*-β-D-glucopyranoside. To our knowledge, this is the first example of an ursane-type triterpenoid with double bonds at C-11(12) and C-13(18).

Rubuside H (**8**) had the molecular formula $C_{36}H_{56}O_{10}$ (HR-FABMS), one more oxygen atom than **7**. The presence of a hydroxymethyl group at C-4 was determined by the HMBC correlation between δ_H 1.07 (H_3-24) and δ_C 66.2 (C-23). Furthermore, the β-OH at C-3 was assigned from the splitting pattern of H-3 (d, J = 9.2 Hz), and therefore the aglycone (**8a**) was 2α,3β,23-trihydroxyurs-11,13(18)-dien-28-oic acid. Thus, **8** was concluded to be 2α,3β,23-trihydroxyurs-11,13(18)-dien-28-oic acid 28-*O*-β-D-glucopyranoside.

Table 1. ^{13}C NMR Data (δ) of **1**−**10** (125 MHz in pyridine-d_5)[a]

position	1	2	3	4	5	6	7	8	9	10
1	48.5	43.4	43.4	48.4	43.4	47.9	42.7	47.4	48.2	42.3
2	68.7	66.0	66.3	68.7	66.1	68.5	66.1	69.0	68.9	65.7
3	83.8	79.2	79.1	83.8	79.3	83.7	79.5	78.2	85.7	76.4
4	39.9	38.7	42.0	39.9	38.8	39.7	38.9	43.7	44.0	53.3
5	56.1	48.7	43.7	56.1	48.9	56.0	48.9	47.8	56.6	43.7
6	18.8	18.3	18.2	18.8	18.4	18.8	18.3	18.5	19.2	20.7
7	35.1	34.9	34.8	34.2	34.2	33.3	32.7	32.4	35.0	33.1
8	39.6	39.6	39.7	39.9	40.0	39.8	41.2	41.0	41.3	40.9
9	48.5	48.1	48.4	48.4	48.2	48.1	54.8	55.0	51.1	47.6
10	38.4	38.4	38.3	38.5	38.6	38.5	38.6	38.1	38.6	38.3
11	23.7	23.4	23.6	23.9	23.8	23.9	126.2	126.1	21.5	24.1
12	126.6	126.5	126.6	127.7	127.7	128.4	127.4	127.4	26.0	128.2
13	138.8	138.7	138.9	137.9	137.9	137.5	135.7	135.7	38.4	139.4
14	45.1	44.9	45.1	43.8	43.8	42.8	42.6	42.6	42.8	42.3
15	29.1	28.9	29.1	28.6	28.6	29.0	25.7	25.7	30.1	29.2
16	35.4	35.2	35.3	23.7	23.7	25.7	32.9	32.9	32.3	26.1
17	50.0	49.8	50.0	47.4	47.4	49.7	47.3	47.2	57.0	48.7
18	133.8	133.7	133.9	50.4	50.4	52.1	138.3	138.3	49.9	54.5
19	136.0	135.4	136.0	128.7	128.8	153.3	34.8	34.8	47.5	72.7
20	34.7	34.5	34.7	123.7	123.8	37.4	27.9	27.9	150.2	42.2
21	26.8	26.7	26.8	28.5	28.5	30.6	25.1	25.1	30.9	26.7
22	31.1	30.9	30.0	32.9	32.9	37.0	32.1	32.1	36.9	37.7
23	29.4	29.4	71.3	29.4	29.5	29.2	29.4	66.2	24.1	208.5
24	17.8	22.2	17.8	17.8	22.4	17.6	21.7	13.8	65.6	14.5
25	17.7	17.3	17.8	17.5	17.2	17.0	19.5	20.1	18.0	17.0
26	18.6	18.5	18.7	18.2	18.2	17.3	17.2	17.2	16.3	17.2
27	22.2	22.0	22.1	22.2	22.1	26.1	21.3	21.3	14.9	24.6
28	174.8	174.7	174.8	176.3	176.3	175.9	176.7	176.6	175.0	177.0
29	19.6	19.4	19.6	17.3	17.3	110.3	16.5	16.5	110.1	27.0
30	18.7	18.6	18.7	20.4	20.4	19.3	19.5	19.5	19.4	16.7
Glc-1	95.9	95.7	95.8	95.9	95.9	95.8	96.5	96.5	95.5	95.9
Glc-2	74.2	74.0	74.2	74.2	74.2	74.0	74.2	74.2	74.4	74.1
Glc-3	78.9	78.8	79.0	78.9	78.9	78.8	78.9	78.9	78.9	79.0
Glc-4	71.3	71.2	71.3	71.2	71.2	71.1	71.4	71.4	71.2	71.3
Glc-5	79.0	79.0	79.1	79.3	79.3	79.2	79.3	79.3	79.5	79.3
Glc-6	62.4	62.3	62.4	62.3	62.3	62.2	62.5	62.5	62.3	62.4

[a] Assignments were based on DEPT, CHSHF, HMQC, HMQC-TOCSY, and HMBC experiments.

The molecular formula of rubuside I (**9**) was $C_{36}H_{58}O_{10}$. The ^{1}H and ^{13}C NMR data of the aglycone of **9** were similar to those of 2α,3β,23-trihydroxylup-20(29)-en-28-oic acid from *Hovenia trichocarea*,[9] but the proton signal at δ_H 1.56 assigned to H-23 of **9** was shifted by +0.52 ppm compared to that of 2α,3β,23-trihydroxylup-20(29)-en-28-oic acid, suggesting that the aglycone of **9** was the C-4 epimer. The hydroxymethyl at C-4 was β-oriented, as deduced from the NOESY correlation between the C-25 methyl and C-24 hydroxymethyl groups. Thus, the aglycone was determined as 2α,3β,24-trihydroxylup-20(29)-en-28-oic acid (**9a**), and the structure of **9** was concluded to be 2α,3β,24-trihydroxylup-20(29)-en-28-oic acid 28-*O*-β-D-glucopyranoside.

Rubuside J (**10**) had the molecular formula $C_{36}H_{56}O_{11}$. A pair of NMR signals at δ_C 128.2 (C-12) and 139.4 (C-13) were characteristic of the double bond of an ursane-12-ene system and readily distinguished **10** from an isomeric olean-12-ene, in which the difference between the chemical shifts of the corresponding pair of signals could be expected to be greater.[5] The ^{1}H and ^{13}C NMR data of **10** were similar to those of pinfaensin (2α,3β,19α-trihydroxyurs-12-en-23-formyl-28-oic acid 28-*O*-β-D-glucopyranoside) (**18**) isolated from the same extract, except for H-3, which was observed at δ_H 4.17 (d, J = 1.6 Hz), suggesting that it was β-oriented. Enzymatic hydrolysis of **10** afforded the aglycone (**10a**), which was determined to be 2α,3α,19α-trihydroxyurs-12-en-23-formyl-28-oic acid. Thus, **10** was concluded to be 2α,3α,19α-trihydroxyurs-12-en-23-formyl-28-oic acid 28-*O*-β-D-glucopyranoside.

The known compounds were identified as 2α,3β,23-trihydroxyurs-12,18-dien-28-oic acid 28-*O*-β-D-glucopyranoside (**11**),[10] 2α,3β,23-trihydroxyurs-12,19-dien-28-oic acid 28-*O*-β-D-glucopyranoside (**12**),[10] alpinoside (**13**),[11] quadranoside VIII (**14**),[12] sericoside (**15**),[13] sericic acid (**16**),[13] buergericic acid (**17**),[14] pinfaensin (**18**),[10] rosamutin (**19**),[11] kaji-ichigoside F1 (**20**),[15] niga-ichigoside F1 (**21**),[15] niga-ichigoside F2 (**22**),[15] sauvissimoside R1 (**23**),[16] 4-epi-niga-ichigoside F1 (**24**),[17] trachelosperoside A1 (**25**),[18] pedunculoside (**26**),[19] ziyu-glycoside (**27**),[20] euscaphic acid (**28**),[15] 1α,2α,3β,19α-tetrahydroxyurs-12-en-28-oic acid (**29**),[21] 19α-hydroxyasiatic acid (**30**),[22] and 2α,3β,19α-trihydroxyurs-12-en-23,28-dioic acid (**31**)[16] by detailed NMR analysis and comparison with data in the literature.

The activity of compounds **1**−**31** against α-glucosidase was examined since some triterpenoids are known α-glucosidase inhibitors.[23,24] When the percentage inhibition was higher than 30% at a concentration of 1 mg/mL, the IC_{50} values were evaluated except for compounds **3**, **10**, **25**, and **26**. The results are listed in Table 3, with acarbose used as a positive control. Compounds **1**, **2**, **6**, **11**−**14**, **16**, **17**, and **28**−**31** exhibited α-glucosidase inhibitory activities with IC_{50} values of 0.65−3.09 mM. Euscaphic acid (**28**) showed the most potent activity (IC_{50} 0.65 mM), which was comparable with the positive control. On the basis of these results, *R. ellipticus* saponins could be potential hypoglycemic agents for diabetes chemotherapy.

Experimental Section

General Experimental Procedures. Optical rotations were measured with a JASCO DIP-370 digital polarimeter in a 0.5 dm length cell. IR spectra were measured with a JASCO FT/IR-300E (by a KBr disk method) spectrometer. ESIMS and HRFABMS were taken on JEOL D-300 and JEOL JMS-700 MStation instruments, respectively. ^{1}H and ^{13}C NMR spectra were measured with a JEOL ECP-500 or JEOL AL-400 spectrometer in δ (ppm) referenced to TMS. Preparative HPLC was performed on a JASCO model PU-2080 HPLC system, equipped with a Shodex RI-101 differential refractometer detector and YMC-Pack RP-C_{18} column (150 × 20 mm i.d.). Diaion HP-20 (Mitsubishi Chemical Corporation, Tokyo, Japan),

Table 2. ^{1}H NMR Data of **1**−**10** (500 MHz in pyridine-d_5)

position	**1**	**2**	**3**	**4**	**5**
1	1.31 (t, 12.6)	1.77 (t, 12.1)	1.22 (t, 12.8)	1.31 (t, 12.4)	1.29 (t, 12.4)
	2.34 (dd, 12.6, 4.6)	2.00 (dd, 12.1, 4.3)	2.03 (dd, 12.1, 4.1)	2.30 (dd, 12.4, 4.2)	1.96 (dd, 11.5, 4.2)
2	4.12 (ddd, 12.6, 9.4, 4.6)	4.32[a]	4.29[a]	4.11 (ddd, 12.4, 9.4, 4.2)	4.31 (ddd, 11.5, 4.2, 2.5)
3	3.41 (d, 9.4)	3.76 (d, 1.8)	4.14 (d, 2.5)	3.40 (d, 9.4)	3.76 (d, 2.5)
5	1.02[a]	1.65 (br d, 12.0)	2.05[a]	1.01 (br d, 11.4)	1.60 (br d, 11.6)
6	1.38 (qd, 12.5, 3.0)	1.33 (qd, 12.0, 2.0)	1.37 (td, 12.4, 2.3)	1.36 (qd, 11.0, 3.2)	1.32 (qd, 12.6, 2.7)
	1.56 (br dd, 12.5, 3.4)	1.49 (br d, 12.0)	1.58 (br d, 12.4)	1.53 (dt, 11.0, 3.5)	1.45 (br d, 12.6)
7	1.63 (td, 12.5, 3.4)	1.64[a]	1.77[a]	1.57 (br dd, 11.0, 3.5)	1.58[a]
	1.48 (dt 12.5, 3.0)	1.48[a]	1.48 (br dd, 12.4, 3.4)	1.40 (m)	1.38 (dt, 12.6, 2.7)
9	1.63 (br d, 13.8)	1.73 (br d, 8.5)	1.80 (br d, 8.0)	1.65 (br d, 8.5)	1.72 (br d, 11.9)
11	2.07 (2H, m)	2.07 (2H, br dd, 8.5, 2.5)	2.09 (2H, m)	2.01 (2H, br td, 8.5, 3.6)	2.01(2H, m)
12	5.70 (t, 3.7)	5.69 (t, 3.5)	5.63 (t, 3.9)	5.64 (t, 3.2)	5.63 (t, 3.4)
15	2.45 (td, 13.6, 3.5)	2.43 (td, 13.5, 3.7)	2.43 (td, 13.5, 2.5)	2.39 (td, 13.0, 5.5)	2.37 (td, 13.5, 4.6)
	1.24 (dt, 13.6, 3.4)	1.22 (dt, 13.5, 3.0)	1.19[a]	1.22 (dt, 13.0, 2.3)	1.19 (dt, 13.5, 3.0)
16	1.53 (td, 13.6, 3.4)	1.49 (td, 13.5, 3.0)	1.48 (td, 13.5, 3.4)	2.05 (2H, m)	2.02 (2H, m)
	2.58 (dt, 13.6, 3.5)	2.56 (dt, 13.5, 3.4)	2.55 (dt, 13.5, 3.0)		
18				3.35 (br s)	3.51 (br s)
20	2.13 (m)	2.10 (m)	2.09 (m)		
21	2.09 (m), 1.27[a]	2.08[a], 1.25 (m)	2.07 (m), 1.26 (br d, 13.5)	2.22 (m), 1.75 (m)	2.21 (m), 1.73 (m)
22	1.71 (td, 13.7, 2.5)	1.69 (br t, 13.7)	1.68 (td, 13.5, 3.0)	1.79 (m)	1.76 (m)
	2.19 (dt, 13.7, 3.0)	2.18 (dt, 13.7, 3.4)	2.18 (dt, 13.5, 3.2)	1.95 (m)	1.94 (m)
23	1.27 (s)	1.27 (s)	3.75 (d, 10.8), 3.91 (d, 10.8)	1.26 (s)	1.25 (s)
24	1.08 (s)	0.91 (s)	0.87 (s)	1.08 (s)	0.90 (s)
25	1.05 (s)	1.02 (s)	1.07 (s)	1.03 (s)	1.00 (s)
26	1.18 (s)	1.18 (s)	1.20 (s)	1.14 (s)	1.14 (s)
27	1.10 (s)	1.00 (s)	1.02 (s)	1.08 (s)	0.96 (s)
29	1.80 (s)	1.77 (s)	1.78 (s)	1.63 (s)	1.59 (s)
30	1.04 (d, 6.7)	1.02 (d, 5.3)	1.01 (d, 7.3)	1.59 (s)	1.58 (s)
Glc-1	6.32 (d, 8.0)	6.32 (d, 8.4)	6.32 (d, 8.2)	6.34 (d, 8.0)	6.33 (d, 8.2)
Glc-2	4.16 (t, 8.6)	4.15 (t, 8.4)	4.16 (t, 8.2)	4.21 (t, 8.3)	4.20 (t, 9.0)
Glc-3	4.26 (t, 8.6)	4.26 (t, 9.0)	4.26 (t, 8.9)	4.29 (t, 8.9)	4.29 (t, 9.0)
Glc-4	4.32 (t, 9.4)	4.32 (t, 9.0)	4.33 (t, 9.2)	4.38 (t, 9.4)	4.37 (t, 9.4)
Glc-5	3.99 (ddd, 9.4, 4.2, 2.8)	3.98 (ddd, 9.0, 4.0, 2.8)	3.99 (ddd, 9.2, 4.4, 2.8)	4.02 (ddd, 9.4, 4.1, 2.8)	4.01 (ddd, 9.4, 3.9, 2.5)
Glc-6	4.36 (dd, 11.7, 4.2)	4.36 (dd, 11.7, 4.0)	4.37 (dd, 11.9, 4.4)	4.39 (dd, 11.9, 4.1)	4.38 (dd, 11.7, 3.9)
	4.40 (dd, 11.7, 2.8)	4.40 (dd, 11.7, 2.8)	4.40 (dd, 11.9, 2.8)	4.44 (dd, 11.9, 2.7)	4.43 (dd, 11.7, 2.5)

position	**6**	**7**	**8**	**9**	**10**
1	1.85 (t, 11.7)	1.45 (t, 12.4)	1.82 (t, 12.1)	1.22 (t, 12.8)	1.88 (t, 12.1)
	2.25 (dd, 12.4, 4.4)	2.28 (dd, 11.7, 3.9)	2.63 (dd, 12.4, 3.9)	2.31 (dd, 12.8, 4.3)	1.96 (br d, 12.1)
2	4.11 (ddd, 12.4, 9.4, 4.4)	4.41 (ddd, 11.7, 3.9, 2.0)	4.35[a]	4.28[a]	4.28[a]
3	3.42 (d, 9.4)	3.81 (d, 2.0)	4.25 (d, 9.2)	3.56 (d, 9.4)	4.17 (d, 1.6)
5	1.06[a]	1.75 (br d, 10.1)	1.88 (br d, 11.7)	1.09 (br d, 10.5)	2.48 (br d, 11.9)
6	1.40 (m)	1.40 (qd, 12.6, 5.3)	1.51 (qd, 12.1, 3.0)	1.42 (m)	1.24[a]
	1.54 (m)	1.53 (dq, 12.6, 2.5)	1.78 (br d, 12.1)	1.65 (dt, 13.5, 3.0)	1.46 (m)
7	1.55 (m)	1.38 (td, 12.6, 5.3)	1.41 (td, 12.1, 3.0)	1.41 (m)	1.83 (td, 12.6, 4.1)
	1.44 (m)	1.32 (m)	1.30 (dt, 12.1, 2.5)	1.35 (m)	1.43 (t, 12.6)
9	1.88[a]	2.27 (br s)	2.25 (br s)	1.45 (m)	2.16 (br d, 10.8)
11	2.06 (2H, m)	6.51 (dd, 10.5, 2.9)	6.51 (dd, 10.5, 2.8)	1.33 (m), 1.45 (m)	2.15 (2H, br td, 10.8, 3.9)
12	5.53 (t, 3.3)	5.87 (dd, 10.5, 0.7)	5.86 (br d, 10.5)	1.12[a], 1.85 (br d, 12.4)	5.55 (t, 3.5)
13				2.69 (br dd, 12.4, 3.7)	
15	2.43 (td, 14.0, 3.0)	0.98[a]	0.94 (td, 13.5, 3.4)	2.05 (td, 13.5, 3.2)	2.46 (td, 13.1, 5.2)
	1.17 (dt, 14.2, 3.9)	2.09 (br d, 11.6)	2.06 (m)	1.20 (m)	1.21[a]
16	1.82 (td, 14.0, 3.9)	1.66 (m)	1.65 (td, 13.5, 2.5)	1.51 (td, 13.5, 3.0)	3.08 (td, 13.5, 4.4)
	1.93 (br d, 14.0)	2.15 (dt, 11.6, 3.2)	2.13 (dt, 13.5, 3.0)	2.67 (br dd, 13.5, 3.2)	2.01 (t, 13.5)
18	3.80 (s)			1.75 (t, 11.0)	2.93 (br s)
19		2.84 (m)	2.84 (dq, 14.2, 7.1)	3.40 (td, 11.0, 4.6)	
20	1.87 (m)	2.33 (m)	2.33 (m)		1.37 (m)
21	1.27[a], 1.45[a]	2.05 (m), 1.24[a]	2.04 (m), 1.21[a]	2.13 (m), 1.41 (m)	1.22[a], 1.40 (m)
22	1.84 (td, 13.6, 3.5)	1.48 (td, 9.4, 4.4)	1.43 (td, 13.3, 4.2)	1.51 (m)	1.84 (td, 12.8, 3.2)
	2.01 (dt, 13.6, 2.7)	2.46 (dd, 13.3, 9.4)	2.45 (br dd, 13.3, 9.4)	2.19 (m)	2.06 (dt, 12.8, 2.5)
23	1.27 (s)	1.26 (s)	3.73 (d, 10.3), 4.23 (d, 10.3)	1.56 (s)	10.02 (s)
24	1.09 (s)	0.92 (s)	1.07 (s)	3.71 (d, 11.0), 4.42 (d, 11.0)	1.15 (s)
25	1.04 (s)	1.03 (s)	1.12 (s)	0.91 (s)	1.07 (s)
26	1.16 (s)	1.20 (s)	1.21 (s)	1.11 (s)	1.22 (s)
27	1.24 (s)	0.90 (s)	0.91 (s)	1.07 (s)	1.63 (s)
29	5.05 (br s), 5.12 (br s)	0.99 (d, 7.1)	0.99 (d, 7.1)	4.74 (s), 4.88 (s)	1.38 (s)
30	1.07 (d, 6.4)	0.80 (d, 6.7)	0.80 (d, 6.6)	1.74 (s)	1.07 (d, 4.2)
Glc-1	6.32 (d, 8.2)	6.32 (d, 8.3)	6.30 (d, 8.3)	6.42 (d, 8.0)	6.30 (d, 8.2)
Glc-2	4.22 (t, 9.0)	4.17 (t, 8.9)	4.16 (t, 8.3)	4.19 (t, 8.5)	4.23 (t, 8.5)
Glc-3	4.30 (t, 9.0)	4.25 (t, 8.9)	4.25 (t, 9.2)	4.29 (t, 8.5)	4.31 (t, 8.9)
Glc-4	4.36 (t, 9.2)	4.31 (t, 9.2)	4.30 (t, 9.2)	4.37 (t, 9.4)	4.37 (t, 8.9)
Glc-5	4.05 (ddd, 9.2, 4.4, 2.4)	4.01 (ddd, 9.2, 4.4, 2.8)	4.02 (ddd, 9.2, 4.4, 2.8)	4.05 (ddd, 9.4, 4.1, 2.5)	4.06 (ddd, 9.2, 4.1, 2.5)
Glc-6	4.40 (dd, 11.9, 4.4)	4.36 (dd, 11.9, 4.4)	4.35 (dd, 11.3, 4.4)	4.40 (dd, 11.9, 4.1)	4.41 (dd, 11.9, 4.3)
	4.48 (dd, 11.9, 2.4)	4.45 (dd, 11.9, 2.8)	4.44 (br d, 11.3)	4.46 (dd, 11.9, 2.5)	4.49 (dd, 11.9, 2.0)

[a] Overlapped signals.

silica gel (silica gel 60N, Kanto Chemical Co., Inc., Tokyo, Japan), and ODS (100−200 mesh, Chromatorex DM1020T ODS, Fuji Silysia Chemical Co., Ltd., Aichi, Japan) were used for column chromatography (CC). TLC was conducted using Kieselgel 60 F_{254} plates (E. Merck). GLC was carried out on a PerkinElmer Clarus 500 GC-MS instrument.

Table 3. In Vitro α-Glucosidase Inhibition Assay

compound	IC_{50} (mM)	compound	IC_{50} (mM)
1	2.54 ± 0.05	**16**	1.37 ± 0.10
2	2.84 ± 0.08	**17**	1.80 ± 0.24
6	1.31 ± 0.14	**28**	0.65 ± 0.09
11	1.42 ± 0.09	**29**	1.49 ± 0.09
12	3.09 ± 0.16	**30**	1.37 ± 0.08
13	1.04 ± 0.13	**31**	1.68 ± 0.09
14	1.85 ± 0.32	acarbose	0.82 ± 0.11

Plant Material. Plant material used in this research was collected from Yunnan Province, People's Republic of China, in September 2002, and identified by one of the authors (W.L.). A specimen of the plant (Toho2202) is kept in the herbarium of the Faculty of Pharmaceutical Sciences, Toho University.

Extraction and Isolation. Roots of *R. ellipticus* (3.0 kg) were extracted with MeOH. Evaporation of the solvent under reduced pressure yielded 223 g of extract. The extract was then partitioned between *n*-BuOH and H_2O. The *n*-BuOH layer (143 g) was subjected to a Diaion HP-20 column eluted with 30%, 70%, and 100% MeOH. The 70% MeOH (53.5 g) and 100% MeOH fractions (35.7 g) were combined and then subjected to silica gel CC using a gradient of $CHCl_3$−MeOH−H_2O to give nine fractions (A−I). Fraction E (1.4 g) was purified by repeated RP-HPLC to give **2** (35 mg), **5** (17 mg), **7** (5 mg), **16** (2 mg), **17** (15 mg), **28** (4 mg), **29** (3 mg), and **30** (2 mg). Fraction F (2.3 g) was separated by repeated RP-HPLC to give **1** (2 mg), **3** (8 mg), **4** (5 mg), **6** (4 mg), **9** (2 mg), **10** (5 mg), **13** (33 mg), **18** (18 mg), and **20** (145 mg). Fraction G (1.6 g) was fractionated by repeated RP-HPLC to give **8** (16 mg), **11** (59 mg), **12** (12 mg), **14** (70 mg), **15** (40 mg), **19** (43 mg), **22** (78 mg), **24** (14 mg), **25** (7 mg), **26** (5 mg), **27** (25 mg), and **31** (6 mg). Fraction H (2.9 g) and fraction I (1.7 g) were purified by repeated RP-HPLC to give **21** (1.03 g) and **23** (51 mg), respectively.

Rubuside A (1): amorphous solid, $[\alpha]^{22}_D$ +102.0 (*c* 0.9, MeOH); IR (KBr) ν_{max} 3419, 2933, 2864, 1728, 1456, 1377, 1072 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 655 $[M + Na]^+$; positive-ion HRFABMS *m/z* 655.3822 (calcd for $C_{36}H_{56}O_9Na$, 655.3822).

Rubuside B (2): amorphous solid $[\alpha]^{22}_D$ +149.8 (*c* 1.0, MeOH); IR (KBr) ν_{max} 3435, 2937, 2874, 1728, 1455, 1379, 1072 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 655 $[M + Na]^+$; positive-ion HRFABMS *m/z* 655.3806 (calcd for $C_{36}H_{56}O_9Na$, 655.3822).

Rubuside C (3): amorphous solid, $[\alpha]^{22}_D$ +88.4 (*c* 0.7, MeOH); IR (KBr) ν_{max} 3859, 2931, 2865, 1723, 1375, 1033 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 671 $[M + Na]^+$; positive-ion HRFABMS *m/z* 671.3782 (calcd for $C_{36}H_{56}O_{10}Na$, 671.3771).

Rubuside D (4): amorphous solid, $[\alpha]^{22}_D$ +5.2 (*c* 0.5, MeOH); IR (KBr) ν_{max} 3396, 2929, 2864, 1736, 1381, 1072 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 655 $[M + Na]^+$; positive-ion HRFABMS *m/z* 655.3832 (calcd for $C_{36}H_{56}O_9Na$, 655.3822).

Rubuside E (5): amorphous solid, $[\alpha]^{22}_D$ +1.4 (*c* 1.0, MeOH); IR (KBr) ν_{max} 3412, 2935, 2874, 1728, 1193, 1072 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 655 $[M + Na]^+$; positive-ion HRFABMS *m/z* 655.3807 (calcd for $C_{36}H_{56}O_9Na$, 655.3822).

Rubuside F (6): amorphous solid, $[\alpha]^{22}_D$ +12.0 (c 0.6, MeOH); IR (KBr) ν_{max} 3408, 2934, 2864, 1730, 1455, 1071 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 655 $[M + Na]^+$; positive-ion HRFABMS *m/z* 655.3813 (calcd for $C_{36}H_{56}O_9Na$, 655.3822).

Rubuside G (7): amorphous solid, $[\alpha]^{22}_D$ −9.6 (*c* 0.2, MeOH); IR (KBr) ν_{max} 3860, 3427, 2931, 1723, 1382, 1072 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 655 $[M + Na]^+$; positive-ion HRFABMS *m/z* 655.3815 (calcd for $C_{36}H_{56}O_9Na$, 655.3822).

Rubuside H (8): amorphous solid, $[\alpha]^{22}_D$ −58.4 (*c* 1.0, MeOH); IR (KBr) ν_{max} 3403, 2936, 2874, 1728, 1379, 1068 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 671 $[M + Na]^+$; positive-ion HRFABMS *m/z* 671.3762 (calcd for $C_{36}H_{56}O_{10}Na$, 671.3771).

Rubuside I (9): amorphous solid, $[\alpha]^{22}_D$ −0.4 (*c* 0.3, MeOH); IR (KBr) ν_{max} 3860, 2939, 2856, 1739, 1381, 1072 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion ESIMS *m/z* 673 $[M + Na]^+$; positive-ion HRFABMS *m/z* 673.3926 (calcd for $C_{36}H_{58}O_{10}Na$, 673.3927).

Rubuside J (10): amorphous solid, $[\alpha]^{22}_D$ −3.0 (*c* 0.4, MeOH); IR (KBr) ν_{max} 3396, 2930, 1718, 1384, 1074 cm^{-1}; ^1H NMR (pyridine-d_5, 500 MHz) and ^{13}C NMR (pyridine-d_5, 125 MHz), see Tables 1 and 2; positive-ion FABMS *m/z* 687 $[M + Na]^+$; positive-ion HRFABMS *m/z* 687.8130 (calcd for $C_{36}H_{56}O_{11}Na$, 687.8147).

Acid Hydrolysis and Determination of the Absolute Configuration of the Monosaccharides. A solution of **1** (1 mg) in 1 M HCl (dioxane−H_2O, 1:1, 2 mL) was heated at 100 °C for 2 h under an Ar atmosphere. After dioxane was removed, the solution was extracted with EtOAc (2 mL × 3) to remove the aglycone. The aqueous layer was neutralized by passing through an ion-exchange resin (Amberlite MB-3, Organo, Tokyo, Japan) column and concentrated under reduced pressure to dryness to give the sugar fraction. The residue was dissolved in pyridine (0.1 mL) to which 0.08 M D-cysteine methyl ester hydrochloride in pyridine (0.15 mL) was added. The mixture was kept at 60 °C for 1.5 h. After the reaction mixture was dried in vacuo, the residue was trimethylsilylated with 1-trimethylsilylimidazole (0.1 mL) for 2 h. The mixture was partitioned between hexane and H_2O (0.3 mL each), and the hexane extract was analyzed by GC-MS under the following conditions: capillary column, EQUITY-1 (30 m × 0.25 mm × 0.25 μm, Supelco); column temperature, 230 °C; injection temperature, 250 °C; carrier gas, N_2. In the acid hydrolysate of **1**, D-glucose was confirmed by comparison of the retention time of their derivatives with those of D-glucose and L-glucose derivatives prepared in a similar way, which showed retention times of 20.00 and 19.43 min, respectively. Sugars from compounds **2**−**10** were also identified by the same method.

Enzymatic Hydrolysis. A solution of **3** (2.1 mg) in 0.1 M acetate buffer (pH 4.0, 1.0 mL) was treated with naringinase (Sigma Chemical Co., 2 units), and then the reaction mixture was stirred at 40 °C for 120 h. The reaction mixture was passed through a Sep-Pak ODS cartridge (Waters) and washed with H_2O and CH_3CN to give the aglycone **3a** (1.5 mg). Through a similar procedure, enzymatic hydrolysis of **7** (1.0 mg) and **10** (1.9 mg) was carried out to afford the aglycones **7a** (0.7 mg) and **10a** (1.3 mg).

2α,3α,23-Trihydroxyurs-12,18-dien-28-oic Acid (3a): amorphous solid, $[\alpha]^{24}_D$ +47.2 (*c* 0.1, $CHCl_3$); ^1H NMR (pyridine-d_5, 500 MHz) δ 5.71 (1H, dd, *J* = 5.3, 2.1 Hz, H-12), 4.30 (1H, dt, *J* = 12.1, 4.1 Hz, H-2), 4.14 (1H, d, *J* = 2.6 Hz, H-3), 3.92 (1H, d, *J* = 10.6 Hz, H-23a), 3.77 (1H, d, *J* = 10.6 Hz, H-23b), 2.55 (1H, m, H-16a), 2.53 (1H, m, H-15a), 2.28 (1H, m, H-22a), 2.03−2.14 (overlapped signals, H-21a, H-20, H-11, H-5, and H-1a), 1.82 (3H, s, H_3-29), 1.26−1.80 (overlapped signals, H-9, H-7a, H-22b, H-6a, H-16b, H-7b, H-6b, H-21b, H-1b, H-15b), 1.26 (3H, br s, H_3-26), 1.06 (3H, s, H_3-25), 1.04 (3H, s, H_3-27), 0.91 (3H, d, *J* = 7.5 Hz, H_3-30), 0.88 (3H, s, H_3-24); ^{13}C NMR (pyridine-d_5, 125 MHz) δ 178.3 (C-28), 138.8 (C-13), 136.0 (C-19), 133.0 (C-18), 125.7 (C-12), 78.7 (C-3), 71.7 (C-23), 66.1 (C-2), 49.6 (C-17), 48.2 (C-9), 45.0 (C-14), 43.6 (C-5), 43.2 (C-1), 41.8 (C-4), 39.4 (C-8), 38.2 (C-10), 35.5 (C-16), 34.9 (C-7), 34.7 (C-20), 29.9 (C-22), 29.8 (C-15), 26.9 (C-21), 23.2 (C-11), 22.0 (C-27), 19.5 (C-29), 18.7 (C-30), 18.1 (C-6), 18.1 (C-26), 17.8 (C-25), 17.6 (C-24).

2α,3α-Dihydroxyurs-11,13(18)-dien-28-oic Acid (7a): amorphous solid, $[\alpha]^{22}_D$ −8.8 (*c* 0.03, $CHCl_3$); ^1H NMR (pyridine-d_5, 500 MHz) δ 6.55 (1H, dd, *J* = 10.5, 2.9 Hz, H-11), 5.86 (1H, dd, *J* = 10.5, 1.8 Hz, H-12), 4.36 (1H, m, H-2), 3.77 (1H, d, *J* = 2.5 Hz, H-3), 2.86 (1H, m, H-19), 2.48 (1H, dd, *J* = 13.9, 8.7 Hz, H-22a), 2.31 (1H, m, H-20), 2.26 (1H, br s, H-9), 2.25 (1H, dd, *J* = 12.0, 4.4 Hz, H-1a), 2.15 (1H, br d, *J* = 10.4 Hz, H-15a), 2.10 (1H, dt, *J* = 10.4, 3.1 Hz, H-16a), 2.02 (1H, m, H-21a), 1.83 (1H, m, H-1b), 1.74 (1H, br d, *J* = 10.2 Hz, H-5), 1.63 (1H, m, H-16b), 1.54 (1H, m, H-6a), 1.48 (1H, dd, *J* = 9.5, 3.8 Hz, H-21b), 1.44 (1H, m, H-6b), 1.40 (1H, m, H-22b), 1.37 (1H, m, H-7a), 1.31 (1H, m, H-7b), 1.24 (3H, s, H_3-23), 1.17 (3H, s, H_3-26), 1.06 (3H, s, H_3-25), 1.02 (1H, m, H-15b), 0.98 (3H, d, *J* = 7.1 Hz, H_3-29), 0.91 (3H, s, H_3-24), 0.88 (3H, s, H_3-27), 0.77 (3H, s, H_3-30).

2α,3α,19α-Trihydroxyurs-12-en-23-formyl-28-oic Acid (10a): amorphous solid, $[\alpha]^{22}_D$ +3.4 (*c* 0.1, $CHCl_3$); ^1H NMR (pyridine-d_5, 500 MHz), δ 10.03 (1H, s, H-23), 5.58 (1H, t, *J* = 3.5 Hz, H-12), 4.26 (1H, ddd, *J* = 9.4, 4.5, 2.2 Hz, H-2), 4.17 (1H, d, *J* = 2.2 Hz, H-3), 3.10 (1H, td, *J* = 13.3, 4.6 Hz, H-16a), 3.04 (1H, br s, H-18), 2.49

(1H, br d, $J = 11.0$ Hz, H-5), 2.31 (1H, td, $J = 13.3$, 5.3 Hz, H-15a), 2.17 (overlapped signal, H_2-11), 2.16 (1H, td, $J = 14.7$, 5.5 Hz, H-22a), 2.15 (overlapped signal, H-9), 2.06 (1H, t, $J = 13.3$ Hz, H-16b), 2.04 (overlapped signal, H-22b), 1.93 (2H, m, H_2-1), 1.92 (3H, s, H-26), 1.81 (1H, br t, $J = 12.2$ Hz, H-7a), 1.71 (1H, m, H-21a), 1.66 (3H, s, H_3-27), 1.46, (overlapped signal, H-6a), 1.44 (overlapped signal, H-20), 1.41 (overlapped signal, H-7b), 1.41 (3H, s, H_3-29), 1.30 (1H, H-6b), 1.26 (1H, overlapped signal, H-15b), 1.25 (1H, overlapped signal, H-21b), 1.12 (3H, s, H_3-24), 1.11 (3H, d, $J = 5.5$ Hz, H_3-30), 1.01 (3H, s, H_3-25); ^{13}C NMR (pyridine-d_5, 125 MHz) δ 208.7 (C-23), 180.0 (C-28), 139.9 (C-13), 127.7 (C-12), 76.3 (C-3), 72.6 (C-19), 65.6 (C-2), 54.5 (C-18), 53.2 (C-4), 48.2 (C-17), 47.4 (C-9), 43.5 (C-5), 42.2 (C-14), 42.2 (C-20), 42.0 (C-1), 40.5 (C-8), 38.4 (C-22), 38.1 (C-10), 33.0 (C-7), 29.1 (C-15), 27.0 (C-29), 26.8 (C-21), 26.2 (C-16), 24.5 (C-27), 23.9 (C-11), 20.5 (C-6), 19.3 (C-26), 17.0 (C-30), 16.7 (C-25), 14.4 (C-24).

α-Glucosidase Inhibition Assay.[25] α-Glucosidase from Baker's yeast was purchased from the Sigma Chemical Co. (St. Louis, MO). α-Glucosidase (25 μL, 0.2 U/mL), 25 μL of various concentrations of samples, and 175 μL of 67 mM phosphate buffer (pH 6.8) were mixed at room temperature for 10 min. Reactions were initiated by the addition of 25 μL of 23.2 mM *p*-nitrophenyl-α-D-glucopyranoside. The reaction mixture was incubated for 15 min at 37 °C in a final volume of 250 μL; then 50 μL of 1 M Na_2CO_3 was added to the incubation solution to stop the reaction. The activities of glucosidase were detected in a 96-well plate, and the absorbance was determined at 405 nm (for *p*-nitrophenol). The negative control was prepared by adding phosphate buffer instead of the sample in the same way as the test. Acarbose was utilized as the positive control. The blank was prepared by adding phosphate buffer instead of the α-glucosidase using the same method. Inhibition rate (%) = $[(OD_{\text{negative control}} - OD_{\text{blank}}) - (OD_{\text{test}} - OD_{\text{test blank}})]/(OD_{\text{negative blank}} - OD_{\text{blank}}) \times 100\%$. IC_{50} values of the samples were calculated using the IC_{50} software.

Supporting Information Available: This material is available free of charge via the Internet at http://pubs.acs.org.

References and Notes

(1) Zhao, S. X.; Huang, T. K.; Ding, Z. Z.; Gao, X. L.; Zhang, Z. D. *Zhong Yao Ci Hai*; China Medicine Scientific Technological Publisher: Beijing, 1997; Vol. 3, p 143.
(2) Dutta, S.; Ghatak, K. L.; Ganguly, S. N. *Nat. Prod. Sci.* **1997**, *3*, 108–110.
(3) Pal, R.; Rastogi, G. C.; Khanna, N. M. *Indian J. Chem., Sect. B: Org. Chem. Incl. Med. Chem.* **1991**, *30*, 292–293.
(4) Hara, S.; Okabe, H.; Mihashi, K. *Chem. Pharm. Bull.* **1987**, *35*, 501–506.
(5) Mahato, S. B.; Kundu, A. P. *Phytochemistry* **1994**, *37*, 1517–1575.
(6) Numata, A.; Takahashi, C.; Miyamoto, T.; Yoneda, M.; Yang, P. M. *Chem. Pharm. Bull.* **1990**, *38*, 942–944.
(7) Ruecker, G.; Mayer, R.; Shin-Kim, J. S. *Planta Med.* **1991**, *57*, 468–470.
(8) Yang, S. C.; Fang, J. M.; Cheng, Y. S. *J. Chin. Chem. Soc.* **1995**, *42*, 573–577.
(9) Yoshikawa, K.; Kimura, Y. K. E.; Arihara, S. *Phytochemistry* **1998**, *49*, 2057–2060.
(10) Durham, D. G.; Liu, I. X.; Richards, R. M. E. *Phytochemistry* **1996**, *42*, 505–508.
(11) Jia, Z. J.; Liu, X. Q.; Liu, Z. M. *Phytochemistry* **1993**, *32*, 155–159.
(12) Adnyana, I. K.; Tezuka, Y.; Awale, S.; Banskota, A. H.; Tran, K. Q.; Kadota, S. *Chem. Pharm. Bull.* **2000**, *48*, 1114–1120.
(13) Bombardelli, E.; Bonati, A.; Gabetta, B.; Mustichi, G. *Phytochemistry* **1974**, *13*, 2559–2562.
(14) Deng, Y.; Peng, S. L.; Zhang, X. R.; Ding, L. S.; Wang, M. K. *Acta Bot. Sin.* **2001**, *43*, 644–646.
(15) Seto, T.; Tanaka, T.; Tanaka, O.; Naruhashi, N. *Phytochemistry* **1984**, *23*, 2829–2834.
(16) Gao, F.; Chen, F. H.; Tanaka, T.; Kasai, R.; Seto, T.; Tanaka, O. *Chem. Pharm. Bull.* **1985**, *33*, 37–40.
(17) Zhou, X. H.; Kasai, R.; Ohtani, K.; Tanaka, O.; Nie, R. L.; Yang, C. R.; Zhou, J.; Yamasaki, K. *Phytochemistry* **1992**, *31*, 3642–3644.
(18) Abe, F.; Yamauchi, T. *Chem. Pharm. Bull.* **1987**, *35*, 1748–1754.
(19) Amimoto, K.; Yoshikawa, K.; Arihara, S. *Chem. Pharm. Bull.* **1992**, *40*, 3138–3141.
(20) Yaguchi, E.; Miyase, T.; Ueno, A. *Phytochemistry* **1995**, *39*, 185–189.
(21) Li, B. Z.; Wang, B. G.; Jia, Z. J. *Phytochemistry* **1998**, *49*, 2477–2481.
(22) Higuchi, R.; Kawasaki, T.; Biswas, M.; Pandey, V. B.; Dasgupta, B. *Phytochemistry* **1982**, *21*, 907–910.
(23) Tchinda, A. T.; Khan, S. N.; Fuendjiep, V.; Ngandeu, F.; Ngane, A. N.; Choudhary, M. I. *Chem. Pharm. Bull.* **2007**, *55*, 1402–1403.
(24) Mbaze, L. M.; Poumale, H. M. P.; Wansi, J. D.; Lado, J. A.; Khan, S. N.; Iqbal, M. C.; Ngadjui, B. T.; Laatsch, H. *Phytochemistry* **2007**, *68*, 591–595.
(25) Hu, L. H.; Zou, H. B.; Gong, J. X.; Li, H. B.; Yang, L. X.; Cheng, W.; Zhou, C. X.; Bai, H.; Gueritte, F.; Zhao, Y. *J. Nat. Prod.* **2005**, *68*, 342–348.

NP900237A