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TWO PHENOLIC GLYCOSIDES FROM THE ROOT BARK OF THE CULTIVATED MULBERRY TREE (*MORUS LHOU*)[1]

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ABSTRACT.—Two new phenolic glycosides, mulberrosides A (**1**) and C (**2**), were isolated from the Me_2CO extract of the root bark of the cultivated mulberry tree (*Morus lhou*) and identified as oxyresveratrol-4,3′-di-*O*-β-D-glucopyranoside and moracin P-3′-β-D-xylopyranoside, respectively.

In previous papers, we reported the structure determination of a series of natural Diels-Alder-type adducts, isoprenylated flavonoids, and 2-arylbenzofuran derivatives isolated from the root bark of *Morus lhou* Koidz. (Japanese name, "Rosō") (1-6). Further extensive fractionation of Me_2CO extracts of the root bark has now led to the isolation of two new phenolic glycosides, named mulberrosides A (**1**) and C (**2**).

The dried root bark of the cultivated mulberry tree was extracted successively with *n*-hexane, C_6H_6, and Me_2CO. Mulberrosides A (**1**) and C (**2**) were isolated from the Me_2CO extract as described in the Experimental section.

Mulberroside A (**1**) is a colorless amorphous powder, $[\alpha]^{25}D$ −78°, $FeCl_3$ test (negative); it gave its decamethyl ether (**1a**), an oily substance, as the exhaustive methylation product by Hakomori's method (7). The field desorption mass spectrum (fdms) of **1** showed a molecular ion peak at *m/z* 568, and the ^{13}C-nmr spectrum indicated the presence of twenty-six carbons (Table 1). The molecular formula of **1a** was determined to be $C_{36}H_{52}O_{14}$ (M^+ 708.3381) by hrms. These results indicated the composition of mulberroside A to be $C_{26}H_{32}O_{14}$.

The ir spectrum of **1** disclosed absorption bands due to hydroxyl, conjugated double bond, and benzene ring moieties, and the uv spectrum exhibited maxima at 218, 235 (sh), 290, 300, 325, and 336 (infl), and was similar to those of oxyresveratrol (**3**) (8) and its derivatives obtained from mulberry root bark (3). These results suggested that **1** is an oxyresveratrol derivative. This suggestion was further supported by examination of the ^1H-nmr and the ^{13}C-nmr spectrum of **1**. The ^1H-nmr spectrum (CD_3OD) of **1** showed, along with signals due to *trans*-olefinic protons (δ 6.95 and 7.32, each 1H, d, J=16 Hz), the occurrence of two independent aromatic rings as follows: δ 6.60 (1H, dd, J=2 and 8 Hz), 6.61 (1H, d, J=2), 7.43 (1H, d, J=8); 6.46 (1H, t, J=2), 6.64 and 6.77 (each 1H, br s). The spectrum, measured by the water elimination Fourier transform (WEFT) method, exhibited two sugar anomeric proton doublets (J=8 Hz) at δ 4.88 and 4.90 (each 1H), and other sugar proton signals appeared in the range of δ 3.30-3.95 (δ 3.30-3.50, overlapping with the solvent). The ^{13}C-nmr spectrum indicated the presence of two aliphatic carbons (−CH=CH−), 12 aromatic carbons (CH×6, C×2, C-O×4), and 12 sugar carbons (Table 1). The hrms of **1a** showed significant fragment ions at *m/z* 272 (**4**, $C_{16}H_{16}O_4$), 218 (**5**, $C_{10}H_{18}O_5$) due to dimethyloxyresveratrol and tetramethylhexose, respectively.

The hydrolysis of **1** with β-glucosidase (β-D-glucoside glucohydrolase) as well as acid hydrolysis gave glucose and an aglycone, which was identified as oxyresveratrol (**3**). From the above results, mulberroside A was identified as an oxyresveratrol-di-*O*-glucoside.

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[1]Part 30 in the series: "Constituents of the Cultivated Mulberry Tree." [Part 7 on "Constituents of the Root Bark of *Morus lhou*." For Part 6, see Fukai *et al.* (6)]. For Part 29, see Y. Hano, H. Kohono, M. Itoh, and T. Nomura, *Chem. Pharm. Bull.*, **33**, 5294 (1985).

TABLE 1. ^{13}C-nmr Chemical Shifts of Mulberrosides A (**1**) and C (**2**), Compounds **3, 9, 10,** and **11**

C	**1**[a]	**3**[b]	C	**2**[a]	C[c]	**1**	C[c]	**2**	**9**[d]	**10**[e]	**11**[e]
1	119.8	117.8	2	153.2[f]	G-1 and 1′	101.6, 101.9	X-1	100.7	103.7 or 103.9	105.1	109.7
2	156.4[f]	156.8	3	101.2	G-2 and 2′	74.5, 74.6	X-2	72.7	73.7	74.0	81.0
3	103.6	102.2	3a	121.3	G-3 and 3′	77.65 (br)[f]	X-3	76.0	75.9	76.9	76.0
4	158.8[f]	158.6	4	120.3	G-4 and 4′	71.1 (br)	X-4	69.0	69.4 or 70.5	70.4	83.6
5	109.0	108.5	5	116.6	G-5 and 5′	77.71[f], 77.8[f]	X-5	65.3	65.5	66.3	62.2
6	124.4	124.4	6	150.4[f]	G-6 and 6′	62.2, 62.3					
α }	127.1	126.4	7	97.8							
β }	127.9	128.5	7a	153.1[f]							
1′	141.3	142.0	1′	130.9							
2′	106.9	105.7	2′	103.4							
3′	159.6[f]	159.0	3′	157.9[f]							
4′	104.6	103.4	4′	103.0							
5′	158.7[f]	159.0	5′	158.0[f]							
6′	107.8	105.7	6′	104.5							
			1″	31.1							
			2″	67.7							
			3″	76.9							
			4″	25.6							
			5″	20.2							
solvent	CD_3OD	CD_3OD		DMSO-d_6					DMSO-d_6	D_2O	D_2O

[a]At 100.4 MHz (digital resolution: 0.0073 ppm).
[b]Data from Hirakura *et al.* (3).
[c]The number of sugar carbons.
[d]Data from Markham and Chari (13).
[e]Data from Markham and Chari (10).
[f]Assignments may be interchanged in each column.

The location of the sugar moieties in **1** was confirmed by comparing the ^{13}C-nmr spectrum of **1** with that of **3**, and by the observation of the nOe between the anomeric protons and the aromatic protons of **1a**. In the ^{13}C-nmr spectrum of **1**, the chemical shift values of the carbon atoms of the stilbene skeleton were similar to those of the relevant carbon atoms of **3**, except for the signals of the carbon atoms at the C-1 and C-6′ positions, which are subjected to an additional effect (Table 1). The nOe measurement was carried out as follows, and the results are shown in Figure 1. When the anomeric proton signal at δ 4.85 (2H, d, J=7 Hz) was irradiated, the nOe was clearly observed between the anomeric protons and the aromatic protons at C-3, C-5, C-2′, and C-4′ positions. The nOe between the methoxyl signals and the aromatic protons at C-3, C-4′, and C-6′ positions was observed by the irradiation of the methoxyl signals both at δ 3.82 and 3.86. The configuration at the anomeric center of **1** was concluded to be β on the basis of the above-mentioned ^{1}H-nmr coupling constant value (J=8 Hz). The chemical shift values of the glucose carbon atoms indicated its β-pyranose form by comparison of the ^{13}C-nmr spectrum of **1** with that of methyl-β-D-glucopyranoside (9, 10). Thus, mulberroside A was confirmed to be oxyresveratrol-4,3′-di-*O*-β-D-glucopyranoside.

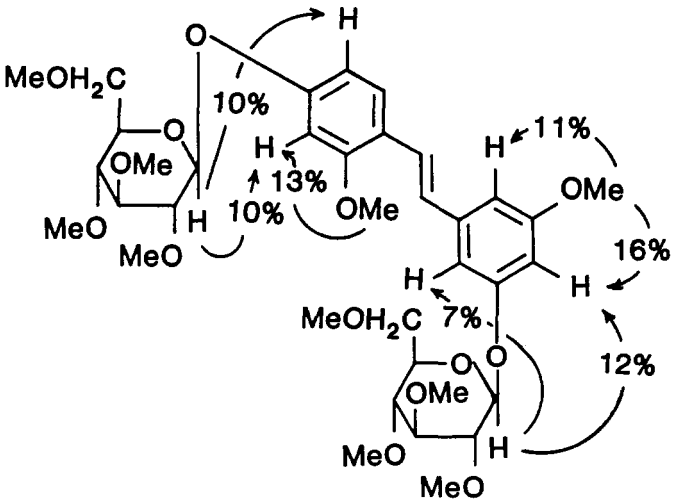
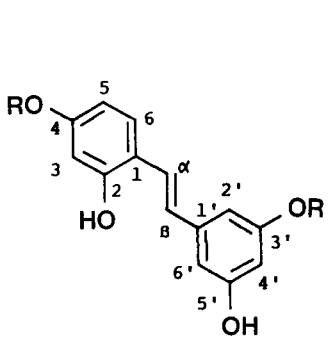


FIGURE 1. The nOe values for mulberroside A decamethyl ether (**1a**).

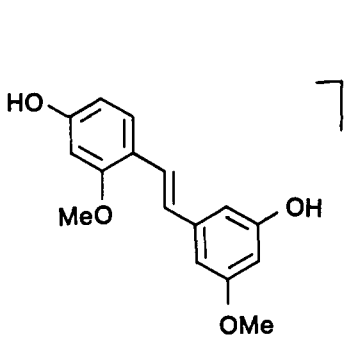
Mulberroside C (**2**), a colorless amorphous powder, $[\alpha]^{19}_D$ −23°, $FeCl_3$ test (negative), gave its pentamethyl ether (**2a**), an oily substance, as the exhaustive methylation product by Hakomori's method. The fdms of **2** showed a molecular ion peak at *m/z* 458 and the ^{13}C nmr indicated the presence of twenty-four carbons (Table 1). The molecular formula of **2a** was determined by hrms to be $C_{29}H_{36}O_9$ (M^+ 528.2358). These results revealed the molecular formula of **2** to be $C_{24}H_{26}O_9$.

The ir spectrum of **2** disclosed absorption bands due to hydroxyl, conjugated double bond, and benzene ring moieties. The uv spectrum exhibited maxima at 218, 257 (sh), 288 (sh), 298 (sh), 309 (infl.), 322, and 337 nm, and was similar to those of moracin M (6,3′,5′-trihydroxy-2-arylbenzofuran) and other 6,3′,5′-trioxygenated-2-arylbenzofuran derivatives obtained from the mulberry tree (4, 11). These results suggested that **2** is one of the 2-arylbenzofuran derivatives. In the ^{1}H-nmr spectrum of **2**, the signals of protons in a 5-substituted-6,3′,5′-trioxygenated-2-arylbenzofuran moiety were observed at δ 6.57 (1H, t, J=2 Hz), 6.89 (1H, br s), 7.03 and 7.04 (each 1H, dd, J=1.5 and 2 Hz), 7.08 (1H, br s), 7.27 (1H, s), as well as the signals of two methyl groups at δ 1.26 and 1.38 (each 3H, s), methylene protons at δ 2.84 (1H, dd, J=8 and

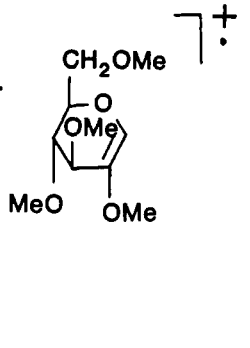
16 Hz), 3.11 (1H, dd, J=5.5 and 16 Hz), and methine proton at δ 3.83 (1H, d t, J=5.5 and 8 Hz). The spectrum also exhibited a sugar anomeric proton doublet (J=7 Hz) at δ 5.00, and other sugar signals appeared at δ 3.40-4.65 (8H). The ^{13}C-nmr spectrum indicated the presence of seven aliphatic carbons (CH_3-x2, -CH_2-x1, R_2CH-O-x1, R_3C-O-x1, -CH=CR-O-x1), 12 aromatic carbons (CHx5, Cx3, C-Ox4), and five sugar carbons (Table 1). The hrms of **2a** showed significant fragment ions at *m/z* 354 (**7**, $C_{21}H_{22}O_5$), 175 (**8**, $C_8H_{15}O_4$) due to dimethylmoracin P and trimethylpentose, respectively. The hydrolysis of **2** with β-D-xylosidase gave an aglycone which was identified as moracin P (**6**) (12). From the above results, mulberroside C was identified as a moracin P-*O*-pentoside.



1 R=β-D-glucopyranosyl
3 R=H

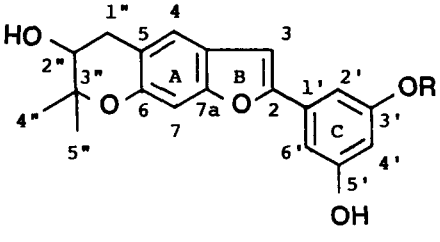


4 *m/z* 272

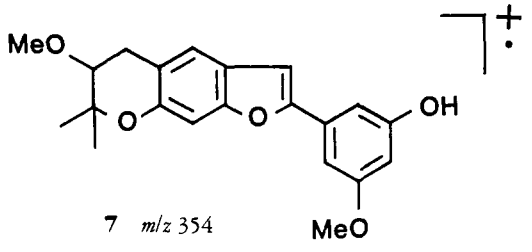


5 *m/z* 218

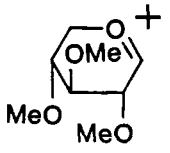
The sugar moiety of **2** was identified as xylose by comparative examination of the ^{13}C-nmr spectra of **2** and model compounds, such as adonivernith (**9**) (13), methyl-β-D-xylopyranoside (**10**) (10), and methyl-β-D-xylofuranoside (**11**) (10) (Table 1). The β-anomeric center is evident from the above-mentioned ^{1}H-nmr coupling constant value (J=7 Hz) (14). The pyranose form was also supported by comparing the chemical shift values of the sugar carbons of **2** with those of model compounds (**10,11**) (10) (Table 1). The location of the sugar moiety in **2** was confirmed by comparing the ^{1}H-nmr spectra of **2** and **6** with each other. In the spectrum of **2**, chemical shift values of the protons at the C-2′ and C-6′ positions appeared nonequivalent (δ 7.03 and 7.04,



2 R=β-D-xylopyranosyl
6 R=H



7 *m/z* 354



8 *m/z* 175

each 1H), while those of the relevant protons of **6** appeared equivalent (δ 6.86, 2H, *d*, *J*=2 Hz). Moreover, in the ^{13}C-nmr spectrum of **2**, the chemical shift values of C-2′ and C-6′ carbon atoms also appeared nonequivalent (Table 1). These results suggest that one of the hydroxyl groups in the C ring formed a glycoside linkage. Accordingly, mulberroside C can be characterized as moracin P-3′-*O*-β-D-xylopyranoside. To the author's knowledge, **2** is the first example of a 2-arylbenzofuran glycoside found in nature.

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURES.—Uv spectra were measured with a Hitachi 340 model spectrophotometer, and the ir spectra with a Hitachi 260-30 spectrophotometer. ^{1}H-nmr and ^{13}C-nmr spectra were recorded with a JEOL GX-400 spectrometer. TMS was used as an internal standard, and chemical shifts are reported on the δ (ppm) scale. Optical rotations were measured with JASCO model DIP-4 digital polarimeter. Eims and fdms were obtained with a JEOL model JMS 01SG-2 instrument and hrms with a Hitachi model RMU-7M spectrometer. Gc analyses were carried out with a Hitachi model 063 gas chromatograph with a F.I.D. detector. Wakogel C-200 (Si gel, Wako Pure Chemical Industries, Osaka, Japan) was used for column chromatography. Analytical tlc was performed on Wakogel B-5FM (Si gel, Wako Pure Chem. Ind.), and the spots were visualized under uv light. Preparative tlc was run on Wakogel B-5F (Si gel, Wako Pure Chem. Ind.).

PLANT MATERIAL.—Root bark of the cultivated mulberry tree, whose Japanese name is "Rosō," a variety of *M. lhou* (15), was collected in the neighborhood of Takasaki, Gunma prefecture, Japan, in December, 1981 (1-6). A sample has been deposited in TOHO herbarium of Toho University.

ISOLATION OF MULBERROSIDES A (**1**) AND C (**2**).—The dried root bark of the cultivated mulberry tree (4 kg) was extracted successively with *n*-hexane, C_6H_6, and Me_2CO. Evaporation of the Me_2CO solution to dryness yielded 220 g of residue (3). The Me_2CO extract (200 g) was chromatographed on Si gel (400 g) using C_6H_6-MeOH (1:0→85:15) and then Me_2CO as an eluent, each fraction being monitored by tlc. The fractions eluted with Me_2CO only were concentrated under reduced pressure. After the solution was allowed to stand at room temperature, the colorless precipitate was collected and washed with Me_2CO to give mulberroside A (**1**, 785 mg, 1.9×10^{-2}% yield from the dried root bark). The fractions eluted with C_6H_6 containing 5% MeOH were evaporated to give the residue (7 g), which was dissolved in Me_2CO. The Me_2CO solution was allowed to stand until pale yellowish precipitate separated. The mother liquor was evaporated to give the residue (6.3 g), which was rechromatographed on silica gel (300 g) using $CHCl_3$-MeOH as an eluent. From the fractions eluted with $CHCl_3$ containing 5% MeOH, mulberroside C (**2**, 15 mg, 3.8×10^{-4}% yield from the dried root bark) was obtained by using preparative tlc [solvent system: $CHCl_3$-MeOH (5:1); $CHCl_3$-Me_2CO (1:3)].

MULBERROSIDE A (**1**).—Mulberroside A (**1**) was obtained as an amorphous powder. Although only one spot was detected on tlc (several solvent systems), **1** could not be isolated in a crystalline form. $[\alpha]^{25}D$ −78° (c=0.88, MeOH). $FeCl_3$ test: negative. Fdms *m/z* 568 $(M)^+$, 406. Uv λ max (EtOH) (log ε) 218 (4.59), 235 (sh 4.42), 290 (4.43), 300 (4.43), 325 (4.56), 336 nm (infl 4.54). Ir ν max (KBr) 3350, 1610 cm^{-1}. ^{1}H nmr (400 MHz, CD_3OD) δ 3.00-3.95 (sugar protons), 6.46 (1H, t, *J*=2 Hz, H-4′), 6.60 (1H, dd, *J*=2 and 8 Hz, H-5), 6.61 (1H, d, *J*=2 Hz, H-3), 6.64 and 6.77 (each 1H, br s, H-2′ or -6′), 6.95 and 7.32 (each 1H, d, *J*=16 Hz, H-α or -β), 7.43 (1H, d, *J*=8 Hz, H-6); (WEFT method), 3.30-3.50 (sugar protons, overlapping with the solvent), 3.67-3.95 (sugar protons), 4.88 and 4.90 (each 1H, d, *J*=8, anomeric protons).

MULBERROSIDE A DECAMETHYL ETHER (**1a**).—Compound **1** (46 mg) was permethylated by Hakomori's method. The product was purified by preparative tlc [*n*-hexane-Me_2CO (2:1)] to give an oily substance (**1a**, 33 mg). Hrms *m/z* $(M)^+$ 708.3381 (calcd for $C_{36}H_{52}O_{14}$ 708.3353), 272.1019 (calcd for $C_{16}H_{16}O_4$ 272.1047), 218.1170 (calcd for $C_{10}H_{18}O_5$ 218.1153). Eims (75 eV) *m/z* (rel. int.) 708 (M^+, 20), 490 (57), 456 (100), 272 (35), 218 (35), 187 (81), 127 (26), 111 (60), 101 (76). Ir ν max ($CHCl_3$) 1600, 1100 cm^{-1}. ^{1}H nmr (400 MHz, $CDCl_3$) δ 3.20-3.70 (10 H, m, overlapping with the signals of methoxyl group, sugar protons), 3.385, 3.39, 3.670, 3.672 (each 3H, s, OCH_3), 3.56 and 3.66 (each 6H, s, OCH_3), 3.82 (3H, s, OCH_3-5′), 3.86 (3H, s, OCH_3-2), 4.85 (2H, d, *J*=7 Hz, anomeric protons), 6.51 (1H, t, *J*=2 Hz, H-4′), 6.63 (1H, dd, *J*=2 and 8 Hz, H-5), 6.64 (1H, H-3, combined with a part of the H-5 signal), 6.74 [1H, br s (dd like), H-6′], 6.80 [1H, br s (dd like), H-2′], 6.93 and 7.34 (each 1H, d, *J*=16 Hz, H-α or -β), 7.46 (1H, d, *J*=8 Hz, H-6).

ACID HYDROLYSIS OF MULBERROSIDE A (**1**).—A mixture of **1** (63 mg) and 1% H_2SO_4 (5 ml) was re-

fluxed for 30 min. The reaction mixture was extracted with Et_2O. The Et_2O layer was treated as usual to give oxyresveratrol (**3**, 3 mg), which was identical with the authentic sample on the basis of the ^{1}H-nmr spectroscopy and tlc [C_6H_6-Me_2CO (2:3)]. The aqueous layer was neutralized with $BaCO_3$ and evaporated to dryness under reduced pressure. The residue was reacted with TMSi reagent [pyridine-hexamethyldisilazane-trimethylchlorosilane (10:2:1, v/v), 0.1 ml]. Analysis of the TMSi derivative by means of gc showed it to be identical with TMSi-glucose (R_t, 2.00 and 2.66 min; column, 2 m×3 mm packed with 1.5% SE-30 on Chromosorb W, column temperature 170°; N_2 at 20 ml/min).

ENZYMATIC HYDROLYSIS OF MULBERROSIDE A (**1**).—A solution of **1** (29 mg) and β-glucosidase (30 mg, Tokyo Kasei) in 0.02 M acetate buffer (3 ml, pH 4.8) was incubated at 37° for 50 h. The reaction mixture was then extracted with Et_2O. The Et_2O layer was treated as usual, and the residue was purified by preparative tlc [C_6H_6-Me_2CO (2:3)] to give oxyresveratrol (**3**, 9 mg), identified with an authentic sample as described above. The aqueous layer was deionized with Dowex 50 W (H^+) and 11 (OH^-) and evaporated to dryness under reduced pressure. This residue was treated as above and was shown by gc to contain glucose.

MULBERROSIDE C (**2**).—Mulberroside C (**2**) was obtained as an amorphous powder. Although only one spot was detected on tlc [CH_2Cl_2-MeOH (6:1), $CHCl_3$-Me_2CO (1:3), $CHCl_3$-MeOH (5:1)], **2** could not be isolated in a crystalline form. $[\alpha]^{19}D$ −23° (c=0.12, MeOH) $FeCl_3$ test: negative. Fdms *m/z* 458 $(M)^+$, 326. Eims (75 eV) *m/z* (rel. int.) 326 (100), 308 (9), 255 (89). Uv λ max (EtOH) (log ε) 218 (4.45), 257 (sh 3.80), 288 (sh 4.05), 298 (sh 4.07), 309 (infl. 4.22), 322 (4.41), 337 nm (4.36). Ir ν max (KBr) 3400 (br), 1620, 1600 cm^{-1}. ^{1}H nmr [400 MHz, $(CD_3)_2CO$] δ 1.26 and 1.38 (each 3H, s, H-4″ or -5″), 2.84 (1H, dd, *J*=8 and 16 Hz, H-1″), 3.11 (1H, dd, *J*=5.5 and 16 Hz, H-1″), 3.83 [1H, d t, *J*=5.5 and 8 Hz, H-2″; 3.83 (1H, dd, *J*=5.5 and 8 Hz, addition of D_2O)], 3.40-4.65 (9H, m, sugar protons and OH-2″), 5.00 (1H, d, *J*=7 Hz, anomeric proton), 6.57 (1H, t, *J*=2 Hz, H-4′), 6.89 (1H, br s, H-7), 7.03 and 7.04 (each 1H, dd, *J*=1.5 and 2 Hz, H-2′ or -6′), 7.08 (1H, br s, H-3), 7.27 (1H, s, H-4).

MULBERROSIDE C PENTAMETHYL ETHER (**2a**).—Compound **2** (6 mg) was permethylated by Hakomori's method and treated as usual. The reaction mixture was extracted with $CHCl_3$, and the $CHCl_3$ extract was purified by preparative tlc [*n*-hexane-Me_2CO (2:1)] to give an oily substance (**2a**, 10 mg). Hrms *m/z* $(M)^+$ 528.2358 (calcd for $C_{29}H_{36}O_9$ 528.2357), 354.1426 (calcd for $C_{21}H_{22}O_5$ 354.1465), 175.0969 (calcd for $C_8H_{15}O_4$ 175.0969). Eims (75 eV) *m/z* (rel. int.) 528 (100), 354 (98), 175 (17), 143 (46), 115 (31), 101 (67). Ir ν max ($CHCl_3$) 1610 (sh), 1600, 1460, 1090 cm^{-1}. ^{1}H nmr (400 MHz, $CDCl_3$) δ 1.30 and 1.41 (each 3H, br s, H-4″ or -5″), 2.86 (1H, br dd, *J*=8 and 16 Hz, H-1″), 3.16 (1H, br dd, *J*=5 and 16 Hz, H-1″), 3.23-3.43 (5H, m, sugar protons), 3.47, 3.50, 3.65, 3.67, and 3.87 (each 3H, s, OCH_3), 4.08 (1H, dd, *J*=5 and 8 Hz, H-2″), 4.99 (1H, d, *J*=7 Hz, anomeric proton), 6.56 (1H, t, *J*=2 Hz, H-4′), 6.86 (1H, d, *J*=1 Hz, H-3), 6.96 (1H, s, H-4), 7.05 and 7.08 (each 1H, dd, *J*=1 and 2 Hz, H-2′ or -6′), 7.22 (1H, br s, H-7).

ENZYMATIC HYDROLYSIS OF MULBERROSIDE C (**2**).—A solution of **2** (3 mg) and β-D-xylosidase (Sigma Chemical Company, from *Aspergillus niger*, 0.21 mg in 0.42 ml solution) in 0.05 M acetate buffer (3 ml, pH 5.0) was incubated at 25° for 50 h. The reaction mixture was then extracted with Et_2O. The Et_2O layer was treated as usual, and the residue was purified by preparative tlc [CH_2Cl_2-MeOH (6:1)] to give moracin P (**6**, 1 mg), identified with an authentic sample on the basis of ^{1}H-nmr spectroscopy.[2] Compound **6** thus obtained showed the following spectra. Eims (75 eV) *m/z* (rel. int.) 326 (M^+, 46), 255 (78), 142 (18). ^{1}H nmr (400 MHz, $(CD_3)_2CO$) δ 1.26 and 1.38 (each 3H, s, H-4″ or -5″), 2.80-2.90 [m, overlapping with H_2O, H-1″; 2.84 (1H, dd, *J*=8 and 16 Hz, addition of D_2O)], 3.11 (1H, dd, *J*=5.5 and 16 Hz, H-1″), 3.82 (1H, d t, *J*=5.5 and 8 Hz, H-2″), 4.26 (1H, dd, $J_{OH,2''}$=5.5 Hz, $J_{OH,1''}$=2 Hz, OH-2″), 6.36 (1H, t, *J*=2 Hz, H-4′), 6.86 (2H, d, *J*=2 Hz, H-2′ and -6′), 6.87 (1H, br s, H-7), 7.00 (1H, d, *J*=1 Hz, H-3), 7.26 (1H, s, H-4), 8.41 and 8.42 (each 1H, s, OH).

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[2]Compound **6** is reported in Shirata *et al.* (12), but no data are provided to support the proposed structure. The data for the identification were sent from Dr. Takasugi.

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