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Constituents of the Stems and Fruits of *Opuntia ficus-indica* var. *saboten*

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From the stems and fruits of *Opuntia ficus-indica* var. *saboten*, eight flavonoids, kaempferol (**1**), quercetin (**2**), kaempferol 3-methyl ether (**3**), quercetin 3-methyl ether (**4**), narcissin (**5**), (+)-dihydrokaempferol (aromadendrin, **6**), (+)-dihydroquercetin (taxifolin, **7**), eriodictyol (**8**), and two terpenoids, (6*S*,9*S*)-3-oxo-α-ionol-β-D-glucopyranoside (**9**) and corchoionoside C (**10**) were isolated and identified by means of chemical and spectroscopic. Among these isolates, compounds **3~5** and **8~10** were reported for the first time from the stems and fruits of *O. ficus-indica* var. *saboten*.

Key words: *Opuntia ficus-indica* var. *saboten*, Quercetin 3-methyl ether, Dihydroquercetin, (6*S*,9*S*)-3-Oxo-α-ionol-β-D-glucopyranoside, Corchoionoside C, Narcissin

INTRODUCTION

Opuntia ficus-indica var. *saboten* Makino belongs to the family Cactaceae, and is found on Cheju Island, South Korea. It is known for rapid growth, good adaptation to poor soils and low requirement for water (Mohamed *et al.*, 1995). Its fruits and stems have been used as Korean folk medicine to treat diabetes, hypertention, asthma, burns, edema and indigestion (Ahn, 1998; Lopez, 1995). Currently, it is cultivated on Cheju Island for use in the manufacture of health foods such as a tea, jam and juice. The isolation of two alkaloids (indicaxanthin and neobetanin) (Impellizzeri *et al.*, 1972; Strack *et al.*, 1987) and five flavonoids (isorhamnetin, quercetin, kaempferol, dihydrokaempferol, and dihydroquercetin) (Jeong *et al.*, 1999) from this species has been previously reported.

By means of chromatographic separation, we have isolated eight flavonoids: kaempferol (**1**), quercetin (**2**), kaempferol 3-methyl ether (**3**), quercetin 3-methyl ether (**4**), narcissin (**5**), (+)-dihydrokaempferol (aromadendrin) (**6**), (+)-dihydroquercetin (taxifolin, **7**), eriodictyol (**8**); and two terpenoids: (6*S*,9*S*)-3-oxo-α-ionol-β-D-glucopyranoside (**9**) and corchoionoside C (**10**). Among these isolates, compounds **3-5** and **8-10** were reported for the first time from the stems and fruits of *O. ficus-indica* var. *saboten*. In this paper, we describe the isolation of the compounds and their subsequent structural determination by spectroscopic analysis.

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MATERIALS AND METHODS

General experimental procedures

Optical rotations were determined on an Autopol III Automatic polarimeter (Rudolph Research Flanders, NJ). Circular dichroism (CD) spectra were measured on a JASCO J-715 spectropolarimeter. IR spectra were recorded on a Midac High Resolution FT-IR spectrometer in KBr disks. FABMS (positive ion mode) and CIMS were obtained on a JEOL JMS-700. NMR spectra were recorded on a Bruker 300 spectrometer. ^{1}H-^{1}H COSY, HMQC and HMBC NMR spectra were obtained with the usual pulse sequences. TLC and column chromatography were carried out on precoated silica gel F_{254} plates (Merck, art. 5715), RP-18 F_{254S} plates (Merck, art. 15423), silica gel 60 (230-400 mesh, Merck), Sephadex LH-20 (Bead size 25-100μ,

Sigma) and LiChroprep RP-18 (40-63 μm, Merck).

Plant material

The stems and fruits of *Opuntia ficus-indica* var. *saboten* were purchased from a local, Korean herbal drug market in January 2001. Voucher specimens (901-15) have been deposited in the laboratory of Korea Institute of Science & Technology (KIST).

Extraction and isolation

Fresh stems (32.5 kg) were cut into small pieces and extracted three times with MeOH at room temperature. The methanol extract (819 g) was suspended in water and then partitioned in turn with CH_2Cl_2, EtOAc, and *n*-butanol. The EtOAc extract (4.99 g) was first subjected to Sephadex LH-20 gel filtration column chromatography eluting with MeOH to provide compounds **1** (2.6 mg) and **3** (3.7 mg). Fractions were combined based on their TLC pattern to yield the fractions designated as F1-F13. Fraction F2 was further purified by RP-18 column chromatography eluting with water and increasing proportions of MeOH (40%→60%) to yield **9** (132.1 mg) and **10** (6.1 mg). Fraction F9 was rechromatographed over RP-18 and eluted with 40% MeOH, followed by increasing proportions of MeOH (40%→60%) in H_2O to yield **2** (6.4 mg), **4** (72.2 mg), **6** (26.9 mg), **7** (77.5 mg), and **8** (4.0 mg).

After removing the seeds, fresh fruits (7.8 kg) were cut into small pieces and extracted three times with MeOH at room temperature. The methanol extract (498 g) was suspended in water and then partitioned in turn with CH_2Cl_2, EtOAc, and *n*-butanol. The EtOAc extract (3.5 g) was divided by column chromatography on Sephadex LH-20 with MeOH, yielding a yellow powder of compounds **1** (2.4 mg) and **2** (3.6 mg). The fractions were combined based on their TLC pattern to yield the fractions designated as E1-E12. Fraction E5 (173.5 mg) was purified by column chromatography over silica gel with CH_2Cl_2:MeOH:H_2O (4:1:0.1→1:1:0.1) to give compound **5** (19.8 mg). Fraction E7 (22.6 mg) was purified by preparative TLC on RP-18 (0.25 mm, 20×20 cm, Merck) developing with 60% MeOH to yield compound **6** (6.1 mg). Subfraction E8 (13.3 mg) was chromatographed on silica gel eluting with CH_2Cl_2:MeOH (20:1→5:1) to give compound **4** (2.3 mg) and **7** (2.2 mg).

Kaempferol 3-methyl ether (3)

Yellow powder; CI-MS *m/z* 301 $[M+H]^+$; IR (KBr): 3384, 1652, 1608, 1504, 1222, 1176 cm^{-1}; ^1H and ^{13}C-NMR data (see Table I and II).

Quercetin 3-methyl ether (4)

Yellow powder; FAB-MS *m/z* 317 $[M+H]^+$; IR (KBr): 3412, 1652, 1606, 1506, 1300, 1211, 1170, 1114 cm^{-1}; ^1H and ^{13}C-NMR data (see Table I and II).

Narcissin (5)

Yellow powder; FAB-MS *m/z* 647 $[M+Na]^+$; IR (KBr): 3400, 2927, 1654, 1604, 1508, 1356, 1206, 1062 cm^{-1}; ^1H and ^{13}C-NMR data (see Table I and II).

(+)-Dihydrokaempferol (aromadendrin) (6)

Yellow powder; $[\alpha]_D^{23}$ +22.8° (*c* 1.32, MeOH); FAB-MS *m/z* 289 $[M+H]^+$; IR (KBr): 3426, 1640, 1468, 1372, 1166, 1086 cm^{-1}; ^1H and ^{13}C-NMR data (see Table I and II).

(+)-Dihydroquercetin (taxifolin) (7)

Yellow powder; $[\alpha]_D^{23}$ +22.0° (*c* 1.68, MeOH); FAB-MS *m/z* 305 $[M+H]^+$; IR (KBr): 3366, 2370, 1640, 1468, 1360,

Table I. ^1H-NMR data for compounds **3-8** (300 MHz, CD_3OD)

NO.	3	4	5	6	7	8
2				4.99 (d, 11.6)	4.80 (d, 11.5)	5.18 (dd, 12.7, 3.1)
3				4.55 (d, 11.6)	4.40 (d, 11.5)	2.59 (dd, 17.1, 3.1), 2.97 (dd, 17.1, 12.7)
6	6.10 (d, 1.9)	6.09 (d, 1.8)	6.22 (d, 2.1)	5.89 (d, 2.1)	5.77 (d, 1.7)	5.77 (d, 2.1)
8	6.30 (d, 1.9)	6.29 (d, 1.8)	6.42 (d, 2.1)	5.93 (d, 2.1)	5.81 (d, 1.7)	5.79 (d, 2.1)
2′	7.88 (d, 8.9)	7.52 (d, 2.0)	7.95 (d, 2.0)	7.37 (d, 8.6)	6.86 (d, 1.4)	6.81 (br s)
3′	6.82 (d, 8.8)			6.84 (d, 8.6)		
5′	6.82 (d, 8.8)	6.79 (d, 8.5)	6.92 (d, 8.5)	6.84 (d, 8.6)	6.69 (d, 8.1)	6.68 (br s)
6′	7.88 (d, 8.9)	7.43 (dd, 8.5, 2.0)	7.64 (dd, 8.5, 2.1)	7.37 (d, 8.6)	6.74 (dd, 8.1, 1.5)	6.69 (br s)
OMe	3.68 (s)	3.68 (s)	3.96 (s)			
Glc-1			5.24 (d, 7.5)			
2-6_a			3.26-3.47 (m)			
6_b			3.84 (br d, 10.0)			
Rha-1			4.54 (d, 1.3)			
2-5			3.26-3.47 (m)			
6			1.11 (d, 6.2)			

Table II. ^{13}C-NMR data of compounds **3-8** (75 MHz, CD_3OD)

NO.	3	4	5	6	7	8
2	158.5	158.4	159.3	84.0	85.5	80.9
3	139.9	139.9	135.9	72.7	74.1	44.5
4	180.4	180.4	179.7	197.6	198.8	198.1
5	163.5	163.5	163.4	164.4	165.7	165.9
6	100.2	100.1	100.4	96.5	97.7	97.4
7	166.5	166.3	166.4	167.8	169.2	168.8
8	95.2	95.1	95.4	95.4	96.7	96.6
9	158.9	158.8	158.8	163.5	164.9	165.2
10	106.3	106.2	106.1	101.0	102.2	103.7
1′	123.0	123.3	123.4	124.3	130.3	132.2
2′	131.8	116.8	115.0	129.5	116.3	115.1
3′	117.0	146.8	148.7	115.3	146.7	146.9
4′	162.1	150.3	151.2	158.4	147.5	147.3
5′	117.0	116.9	116.5	115.3	116.5	116.6
6′	131.8	122.7	124.4	129.5	121.3	119.6
OMe	60.9	60.9	57.2			
Glc-1			104.9			
2			76.3			
3			77.7			
4			72.0			
5			78.6			
6			68.9			
Rham-1			102.9			
2			72.5			
3			72.7			
4			74.2			
5			70.2			
6			18.3			

1164, 1086 cm^{-1}; ^{1}H and ^{13}C-NMR data (see Table I and II).

(2*S*)-Eriodictyol (8)

Yellow powder; FAB-MS *m/z* 289 $[M+H]^+$, IR (KBr): 3386, 2937, 1640, 1460, 1362, 1272, 1166, 1084 cm^{-1}; ^{1}H and ^{13}C-NMR data (see Table I and II); CD [MeOH, nm ($\Delta\varepsilon$)]: 292 (-4.2), 251 (+2.67).

(6*S*,9*S*)-3-Oxo-α-ionol-β-D-glucopyranoside (9)

White powder; $[\alpha]_D^{23}$ -235.6° (*c* 6.43, MeOH); FAB-MS *m/z* 371 $[M+H]^+$; IR (KBr): 3418, 2966, 1656, 1436, 1374, 1258, 1158, 1038 cm^{-1}; ^{1}H and ^{13}C-NMR data (see Table III); CD [MeOH, nm ($\Delta\varepsilon$)]: 243 (-63), 325 (+2).

Corchoionoside C (10)

White powder; $[\alpha]_D^{23}$ +54.5° (*c* 0.2, MeOH); FAB-MS *m/z* 409 $[M+Na]^+$; IR (KBr): 3412, 2928, 2366, 1654, 1374, 1087.9, 1038 cm^{-1}; ^{1}H and ^{13}C-NMR data (see Table III); CD [MeOH, nm ($\Delta\varepsilon$)]:243 (+59), 324 (-3).

Table III. ^{1}H and ^{13}C-NMR data for compounds **9** and **10** (CD_3OD)

NO.	9		10	
	^{1}H	^{13}C	^{1}H	^{13}C
1		37.5		42.8
2	1.96 (d, 16.7) 2.34 (d, 16.7)	48.7	2.06 (d, 16.9) 2.51 (d, 16.9)	51.1
3		202.3		201.6
4	5.78 (s)	126.6	5.77 (s)	127.5
5		165.9		167.5
6	2.60 (d, 9.1)	57.3		80.4
7	5.64 (dd, 15.3, 9.2)	131.8	5.87 (d, 15.6)	134.1
8	5.47 (dd, 15.3, 7.7)	137.5	5.62 (dd, 15.6, 7.2)	134.2
9	4.38 (m)	75.2	4.43 (quin)	75.0
10	1.18 (d, 6.4)	22.6	1.18 (d, 6.4)	22.6
11	0.94 (s)	28.1	0.93 (s)	23.9
12	0.92 (s)	28.5	0.91 (s)	25.1
13	1.84 (d, 0.9)	24.1	1.84 (d, 1.3)	20.0
Glc-1	4.23 (d, 7.5)	101.4	4.17 (d, 7.5)	101.6
2	3.09-3.16 (m)	75.4	3.06-3.15 (m)	75.4
3		78.6		78.6
4		72.2		72.1
5		78.7		78.8
6	3.53 (dd, 11.8, 5.8) 3.77 (dd, 11.8, 1.8)	63.3	3.52 (dd, 11.9, 6.1) 3.74 (dd, 11.9, 2.2)	63.2

RESULTS AND DISCUSSTION

The MeOH extract of the stems and fruits of *Opuntia ficus-indica* var. *saboten* was suspended in water and then consecutively partitioned with CH_2Cl_2, EtOAc, and *n*-butanol. The soluble part of the EtOAc extract was purified by column chromatography using Sephadex LH-20, as well as a combination of chromatography over silica gel and RP-18, to yield the eight known flavonoids (**1-8**) and two known terpenoids (**9** and **10**).

By analysis of the NMR data that was generated in this study, and by the comparison of the physical and spectral data with those of literature, compounds **1**, **2**, **6** and **7** were identified as kaempferol, quercetin (Jeong *et al.*, 1999), (+)-dihydrokaempferol (aromadendrin) (Markham *et al.*, 1984) and (+)-dihydroquercetin (taxifolin) (Nonaka *et al.*, 1987), respectively.

Compound **3** was isolated as a yellow powder and its molecular formula was established as $C_{16}H_{12}O_6$ by FAB-MS (*m/z* 301 $[M+H]^+$). IR absorptions suggested the presence of a hydroxy group (3384 cm^{-1}), an α,β-unsaturated carbonyl group (1652 cm^{-1}), and an aromatic ring (1608 and 1504 cm^{-1}). The ^{1}H-NMR spectrum suggested that compound **3** has a kaempferol moiety. The signals at δ 7.88 (d, *J* = 8.8 Hz) and 6.82 (d, *J* = 8.8 Hz) are characteristic for the B ring as a *para*-substituent with two *meta*-coupled doublets at δ 6.30 (d, *J* = 1.9 Hz) and 6.10

(d, J = 1.9 Hz) for the A ring. In addition, the signal at δ 3.68 (s, 3H) was assigned to one methoxy signal. In the ^{13}C-NMR data (Table II) of **3**, the signals at δ 180.4 (C-4) and 60.9 (OMe) supported evidence of the presence of carbonyl and methoxy groups. The methoxy group was located at C-3 of the C ring, since the methoxy signal at δ 3.68 correlated with δ 139.9 (C-3) in the HMBC spectrum. Thus, compound **3** was elucidated as kaempferol 3-methyl ether (Stevens *et al.*, 1999).

Compound **4** was obtained as a yellow powder and its molecular formula was established as $C_{16}H_{12}O_7$ by FAB-MS (*m/z* 317 $[M+H]^+$). IR absorptions suggested the presence of a hydroxy group (3412 cm^{-1}), an α,β-unsaturated carbonyl group (1652 cm^{-1}) and an aromatic ring (1606 and 1506 cm^{-1}). The ^{1}H-NMR spectrum suggested that compound **4** has a quercetin moiety. Signals at δ 6.79 (d, J = 8.5 Hz), 7.43 (dd, J = 2.0 and 8.5 Hz), and 7.52 (d, J = 2.0 Hz) were characteristic for a 3,4-disubstituted B ring. In ^{13}C-NMR spectrum, the signals at δ 146.8 (C-3') and 150.3 (C-4') were assignable to two aromatic carbons of B ring, the signals at δ 60.9 (OMe), 139.9 (C-3) and 180.4 (C-4) also supported the above assignment. The methoxy group was located at C-3 of the C ring, since the methoxy signal at δ 3.68 correlated with δ 139.9 (C-3) in the HMBC spectrum. Thus, compound **4** was elucidated as quercetin 3-methyl ether (Roitman *et al.*, 1985).

Compound **5** was obtained as a yellow powder and its molecular formula was established as $C_{28}H_{32}O_{16}$ by CI-MS (*m/z* 625 $[M+Na]^+$). IR absorptions suggested the presence of a hydroxy group (3400 cm^{-1}), an α,β-unsaturated carbonyl group (1654 cm^{-1}) and an aromatic ring (1604 and 1508 cm^{-1}). The ^{1}H-NMR spectrum of **5** had similar peak patterns to those of compound **4**, except that it contained two sugar moieties. Signals at δ 6.92 (d, J = 8.5 Hz), 7.64 (dd, J = 2.1 and 8.5 Hz) and 7.95 (d, J = 2.0 Hz) are assignable to three aromatic protons of the ABX system of the B ring. In addition, the signals of the anomeric proton appeared at δ 4.54 (d, J = 1.3 Hz) and 5.24 (d, J = 7.5 Hz) with characteristic coupling constants of a α- and β-configuration, respectively. In the ^{13}C-NMR spectrum of **5**, the signals at δ 104.9 and δ 102.9 corresponded to the glucosyl and rhamnosyl anomeric carbon, respectively (Agrawal, 1989). In particular, by the chemical shift of the ten carbons of **5** at δ 76.3 (C-2"), 77.7 (C-3"), 72.0 (C-4"), 78.6 (C-5") and 68.9 (C-6"), and δ 72.5 (C-2'"), 72.7 (C-3'"), 74.2 (C-4'"), 70.2 (C-5'") and 18.3 (C-6'") in the ^{13}C-NMR spectrum, the sugars were confirmed to be glucose and rhamnose, respectively. The downfield shift of the signal at δ 68.9 assignable to C-6", in comparison with glucose indicated C-6" to be the glucosylation site. The signal at δ 4.54 (H-1'") showed a correlation with the signal at δ 68.9 (C-6") of the glucose moiety in the HMBC spectrum. Therefore, these data showed that rhamnose was attached at C-6" of glucose. Also the signal at δ 5.24 (H-1") of the anomeric proton of glucose was correlated with the signal at δ 135.9 (C-3) of aglycon, indicating that the C-3 position of aglycon was glucosylated. Moreover, the methoxy signal at δ 3.96 showed a correlation with the C-3' signal at δ 148.7 of the B ring, indicating that the methoxy group was connected at the C-3' position. On the basis of these data, compound **5** can be assigned as narcissicin (Nakano *et al.*, 1989), as shown in Fig. 1.

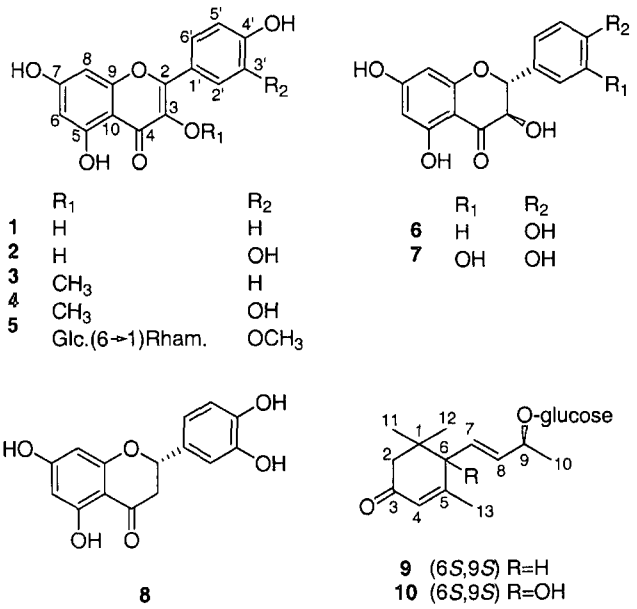

Fig. 1. Structures of Compounds **1**-**10** from *Opuntia ficus-indica* var. *saboten*.

Compound **8** was also obtained as a yellow powder and its molecular formula was established as $C_{15}H_{12}O_6$ by FAB-MS (*m/z* 289 $[M+H]^+$). The ^{1}H-NMR spectrum, signals at δ 6.81 (br s, H-2'), 6.68 (br s, H-5') and 6.69 (br s, H-6') are assigned to three aromatic protons for the B ring, with two *meta*-coupled doublets at δ 5.77 and 5.79 for the A ring. ^{1}H-^{1}H COSY spectra of **8**, the H-2 methine proton at δ 5.18 (dd, J = 12.7 and 3.1 Hz) showed a correlation with the methylene proton (H-3) signals at δ 2.59 (dd, J = 17.1 and 3.1 Hz) and 2.97 (dd, J = 17.1 and 12.7 Hz), indicating that compound **8** has a flavanone skeleton. In the ^{13}C-NMR data (Table II) of **8**, the signals at δ 80.9 (C-2), 44.5 (C-3), and 198.1 (C-4) of the flavanone moiety supported the above assignment. The absolute stereochemistry of **8** was determined on the basis of circular dichroism (CD) spectroscopic analysis. The CD spectrum of **8** showed negative Cotton effects [292 (Δε -4.2), 251 (+2.67) nm], which indicated the absolute configuration of the 2-position be *S* orientation (Gaffield, 1970; Yoshikawa *et al.*, 1998; Matsuda *et al.*, 2002). On the basis of the evidence obtained, compound **8** was assigned to (2*S*)-eriodictyol (Agrawal, 1989).

Compound **9** was obtained as a white powder and its molecular formula was established as $C_{19}H_{30}O_7$ by FAB-MS (*m/z* 371 $[M+H]^+$). IR absorptions at 3418 (OH) and 1656 (C=O) cm^{-1} suggested the presence of a hydroxy group and an α,β-unsaturated carbonyl group. The ^{1}H-NMR spectrum of compound **9** showed the four methyl groups; the two singlets at δ 0.92 and 0.94, and the two doublets at δ 1.84 (*J* = 0.9 Hz) and 1.18 (*J* = 6.4 Hz). In addition, the signals at δ 5.64 (dd, *J* = 15.3 and 9.2 Hz) and 5.47 (dd, *J* = 15.3 and 7.7 Hz) corresponded to the *trans* olefinic protons and the broad singlet at δ 5.78 (H-4), assignable to the vinyl proton indicated the presence of a substituted double bond. The signal of the anomeric proton appeared at δ 4.23 (d, *J* = 7.5 Hz) with coupling constants characteristic of a β-configuration. The chemical shifts of seven carbons of **9** at δ 37.5 (s), 48.7 (t), 126.6 (d), 165.9 (s), 57.3 (d), 75.2 (d) and 202.3 (s) in the ^{13}C-NMR and DEPT spectra implied that this system contained one methylene carbon, three tertiary carbons, two quaternary and one carbonyl group. In particular, by the chemical shift of the carbons at δ 101.4 (C-1'), 75.4 (C-2'), 78.6 (C-3'), 72.2 (C-4'), 78.7 (C-5') and 63.3 (C-6') in the ^{13}C-NMR spectrum (Table III), the sugar was identified as glucose (Roitman *et al.*, 1985). In the COSY spectrum, the signal at δ 5.64 (H-7) showed correlations with the signals at δ 5.47 (H-8) and 2.60 (H-6), and the signal at δ 5.47 (H-8) correlated with the signals at δ 5.64 (H-7) and 4.38 (H-9), respectively. The H-4 methine proton (δ 5.78) appearing as a broad singlet showed a correlation only with the proton signal (H-13) at δ 1.84. In the HMBC spectrum of **9**, the signals at δ 0.92 (H-12) and 0.94 (H-11) showed a correlation with the carbon signal at δ 37.5 (C-1), and the signals at δ 1.96 and 2.34 (H-2) were correlated with the carbonyl signal at δ 202.3 (C-3) and 37.5 (C-1). Therefore, the positions of these two methyl groups were assigned to C-1 and the carbonyl group was proposed as residing at C-3. The signal at δ 4.23 (H-1') of the anomeric proton was correlated with the signal at δ 75.2 (C-9), indicating that the glucose was connected through C-9. The methine proton signal at δ 5.64 (H-7) showed a correlation with the signals at δ 165.9 (C-5), 57.3 (C-6) and 75.2 (C-9), and the other methine proton signal at δ 5.47 (C-8) was correlated with the signals at δ 22.6 (C-10) and 57.3 (C-6) indicating the location of these carbons as shown in Fig. 1.

Stereochemistry at C-6 was established on the basis of circular dichroism (CD) with comparison to literature data (Pabst *et al.*, 1992). It was reported that (6*R*,9*S*)-3-oxo-α-ionol-β-D-glucopyranoside exhibited a positive maximum at 243 nm. In contrast, the CD extreme values of compound **9** [243 (Δε -63), 325 (+2) nm] were opposite to those of the (6*R*,9*S*)-3-oxo-α-ionol-β-D-glucopyranoside. Therefore, the stereochemistry at C-6 in compound **9** was assigned the *S*-configuration. In the literature data, the ^{13}C-NMR chemical shifts of the β-D-glucopyranoside of (9*R*)- and (9*S*) 3-oxo-α-ionol to be δ 77.0 and 74.7, respectively (Pabst *et al.*, 1992). The absolute configuration at the C-9 of **9** was assigned to the *S*-configuration, since the ^{13}C-NMR chemical shifts of the 8-, 9- and 10-positions have essentially the same chemical shifts as those of (6*R*,9*S*)-3-oxo-α-ionol-β-D-glucopyranoside. Therefore, on the basis of above and literature data (Cui *et al.*, 1993), the compound **9** could be characterized as (6*S*,9*S*)-3-oxo-α-ionol-β-D-glucopyranoside.

Compound **10** was obtained as a white powder and its molecular formula was established as $C_{19}H_{30}O_8$ by FAB-MS (*m/z* 409 $[M+Na]^+$). IR absorptions suggested the presence of a hydroxy group, and an α,β-unsaturated and carbonyl group. The ^{1}H and ^{13}C-NMR spectra of **10** had similar peak patterns to those of compound **9**, except that it contains an hydroxy group located on C-6. The ^{1}H-NMR spectrum of compound **10** showed the signal at δ 5.87 (1H, d, *J* = 15.6, H-7) and no proton signal corresponding to the H-6 position of compound **9**, confirming the presence of a substituent at C-6 in compound **10**. Finally, compound **10** was described as corchoionoside C by the comparison of literature data (Çaliş *et al.*, 2002).

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