Secoiridoids from *Jasminum odoratissimum*

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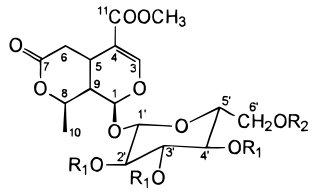
Received June 2, 1997⊗

Five new secoiridoids, *trans*- and *cis*-6′-*O*-*p*-coumaroyl-8-epikingiside (**1a**) and (**1b**), *trans*- and *cis*-10-(*p*-coumaroyloxy)oleoside dimethyl ester (**2a**) and (**2b**), and 6′-*O*-acetyl-10-acetoxyoleoside (**2e**), were isolated from *J. odoratissimum* and their structures determined by spectroscopic means. Compounds **1a** and **1b** and **2a** and **2b** were characterized as their peracetylated derivatives (**1c** and **1d** and **2c** and **2d**, respectively).

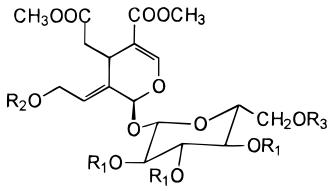
In a previous communication,[1] we reported on the isolation and structure of a new secoiridoid, 10-acetoxyoleoside dimethyl ester, from *Jasminum odoratissimum* L. (Oleaceae), a species endemic to the Canary Islands,[2] where it grows in dry thermophilic zones. In this paper, we describe a further study of this species, which has afforded five new secoiridoids, *trans*- and *cis*-6′-*O*-*p*-coumaroyl-8-epikingiside (**1a**) and (**1b**), *trans*- and *cis*-10-(*p*-coumaroyloxy)oleoside dimethyl ester (**2a**) and (**2b**), and 6′-*O*-acetyl-10-acetoxyoleoside (**2e**). These compounds are closely related to ibotalactone isolated previously from *Ligustrum obtusifolium*[3] and jaslanceosides B[4] and D[5] from *Jasminum lanceolarium*.

Secoiridoids **1a** and **1b** were obtained as a mixture of geometrical isomers. The ^{1}H NMR spectrum (Table 1) of the mixture showed, in addition to typical secoiridoid signals, resonances for aromatic and *trans*- and *cis*-olefinic protons that were assignable to a *trans*- and *cis*-*p*-coumaroyl group, respectively, as well as signals attributable to a sugar moiety. Signals at δ 21.5 and 21.7 (C-10 methyl group) in the ^{13}C NMR spectrum (Table 2) were characteristic for an 8-epikingiside-type secoiridoid.[6] The signals at δ 76.0 and 63.9 were shifted −1.9 and +1.0, respectively, with regard to the normal values for C-5′ and C-6′ of a glucose molecule, which is in agreement with the coumaroyl group being linked to C-6′ of a glucose moiety.[7] In order for the mixture of **1a** and **1b** to be separated, it was acetylated and the isomers **1c** and **1d** were obtained as tetraacetates.

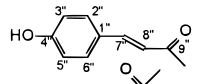
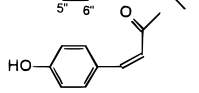
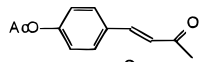
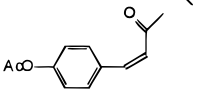
The ^{1}H NMR spectrum (Table 3) of compound **1c** showed signals that confirmed this substance to be a secoiridoid-type compound. Thus, signals at δ 7.42 and 5.25, which appeared as doublets with coupling constants 1.2 and 5.9 Hz, respectively, were considered diagnostic, with the former being assigned as the H-3 proton and the latter as the acetal H-1 proton in the proposed 8-epikingiside structure **1**.[6] Doublets at δ 7.54 and 7.11, with a coupling constant of 8.5 Hz, each integrating two protons, were attributable to an AA′BB′ system in a *p*-substituted aromatic group. In addition, the olefinic resonances at δ 7.66 and 6.38 (d, J = 16 Hz) gave evidence for the presence of a *p*-coumaroyl group, commonly found in this type of compound.[8] ^{1}H NMR assignments shown in Table 3 for **1c** were established after a COSY experiment was performed. The correlation of a series of signals between δ 3.79 and 5.23 in this experiment showed the presence of an esterified glucose moiety in **1c**, which was also confirmed by an ion at 477.1399 in HRFABMS of an acetylated glucose moiety with a *p*-acetoxycoumaroyl group.[9] The ^{13}C NMR spectral assignments (Table 4) were made after running HMQC and HMBC experiments, with the resonances at 93.8 and 96.3 ppm correlated to the acetal carbons C-1 and C-1′. The HMBC experiment also showed a correlation between one of the protons of the C-6′ methylene with the C-9″ carbonyl carbon, consistent with the *p*-coumaroyl group being bonded to the C-6′ carbon of the glucose moiety. All the observed correlations in the HMQC and HMBC



1



2

	R_1	R_2	R_3
1a, 2a	H		H
1b, 2b	H		H
1c, 2c	Ac		Ac
1d, 2d	Ac		Ac
2e	H	Ac	Ac
2f	Ac	Ac	Ac

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⊗ Abstract published in *Advance ACS Abstracts,* November 15, 1997.

S0163-3864(97)00272-3 CCC: $14.00

Table 1. ^{1}H NMR Data in CD_3OD of Compounds **1a**,**b** and **2a**,**b**

H	1a	1b	2a	2b
1	5.26 d (7.4)	5.21 d (7.4)	5.98 s	5.97 s
3	7.49 s	7.49 s	7.54 s	7.54 s
5	2.98 m	2.98 m	4.08 m	4.08 m
6_A	2.26 dd (16.4, 11.3)	2.26 dd (16.4, 11.3)	2.52−2.57	2.52−2.57
6_B	2.75 m	2.75 m	2.75−2.85	2.75−2.85
8	4.24−4.38 m	4.24−4.38 m	6.18 t (6.1)	6.14 t (7.8)
9	1.95−2.05 m	1.95−2.05 m		
10			4.90 m	4.90 m
CH_3	1.37 d (6.3)	1.37 d (6.3)		
1′	4.66 d (7.8)	4.62 d (7.8)	4.73 overlapped with MeOH	4.73 overlapped with MeOH
2′ − 6′	3.30−4.50	3.30−4.50	3.30−4.50	3.30−4.50
2″ + 6″	7.41 d (8.6)	7.56 d (8.6)	7.45 d (8.7)	7.58 d (8.7)
3″ + 5″	6.74 d (8.6)	6.70 d (8.6)	6.80 d (8.7)	6.76 d (8.7)
7″	7.55 d (16.0)	6.78 d (12.8)	7.61 d (16.0)	6.86 d (12.8)
8″	6.28 d (16.0)	5.72 d (12.8)	6.31 d (16.0)	6.30 d (16)
CH_3	3.72 s	3.71 s		
CH_3	3.63 s	3.67 s	3.66 s	3.63 s

Table 2. ^{13}C NMR Data in CD_3OD of Compounds **1a,b** and **2a,b**

C	1a	1b	2a	2b
1	96.8 d	96.8 d	94.2 s	94.2 s
3	154.4 d	154.4 d	153.9 d	153.9 d
4	109.6 s	109.8 s	110.0 s	110.0 s
5	28.1 d	28.1 d	33.0 d	33.0 d
6	34.6 t	34.6 t	41.1 t	41.1 t
7	174.7 s	174.5 s	173.7 s	173.7 s
8	75.9 d	76.1 d	124.0 d	124.0 d
9	41.9 d	41.9 d	135.0 s	135.0 s
10	21.7 q	21.7 q	62.0 t	62.0 t
11	168.9 s	168.9 s	167.6 s	167.6 s
1′	101.0 d	101.0 d	101.0 d	101.0 d
2′	74.9 d	74.9 d	74.9 d	74.9 d
3′	78.5 d	78.5 d	78.6 d	78.6 d
4′	71.7 d	71.7 d	71.6 d	71.6 d
5′	77.9 d	77.9 d	78.0 d	78.0 d
6′	62.9 t	62.9 t	62.8 t	62.8 t
1″	126.8 s	127.4 s	127.2 s	127.7 s
2″ + 6″	131.3 d	133.8 d	131.3 d	133.8 d
3″ + 5″	115.9 d	116.8 d	117.0 d	116.1 d
4″	161.3 s	161.3 s	161.4 s	160.2 s
7″	145.3 d	147.2 d	145.2 d	146.7 d
8″	114.6 d	116.1 d	116.6 d	118.8 d
9″	168.0 s	167.8 s	168.4 s	168.9 s

experiments supported the proposed structure **1c** for this compound.

The ^{1}H NMR data (Table 3) for compound **1d** were almost the same as those for **1c**, with the main difference being the olefinic proton chemical shifts, δ 7.98 and 5.97 and coupling constants (12.6 Hz), which were indicative of a *cis*-substituted double bond. The ^{13}C NMR values (Table 4) and additional spectroscopic and mass spectrometric data were in agreement with structure **1d**.

Chemical shifts at δ 7.54 and 7.10, in the ^{1}H NMR spectrum for **2c**, appeared as doublets with coupling constants of 8.8 Hz and integrated for two protons each. These signals, along with two doublets at δ 7.69 and 6.40 (16.0 Hz), with each signal integrating for one proton, also indicated the presence of a *p*-coumaroyl moiety. A COSY experiment showed that a series of signals between δ 5.27 and 5.05 were correlated to one another, which along with a multiplet at δ 3.80−3.75 and two double doublets (δ 4.31 and 4.14) were representative of a glucose moiety. Diamagnetic shifts for H-5 (0.96 ppm) and H-8 (1.71 ppm) with respect to **1c** were observed, with the latter signals being less complex than in compounds **1c** and **1d**, being a triplet in this case. Another important difference was that there were no chemical shifts assignable to H-9 or CH_3-10. The HMBC experiment showed correlations between C-8 (two bonds), C-9 (three bonds), and signals at δ 4.94 and 4.88, in accordance with C-10 being a methylene group as in structure **2c**. The observed correlation between the C-10 methylene protons and the carbonyl carbon C-9′ established that the *p*-coumaroyl moiety was bonded to C-10 rather than to C-6 of the glucose moiety.

The spectroscopic data for compounds **2c** and **2d** were almost the same, except for those referring to the *p*-coumaroyl double bond. The coupling constant for this system (12.8 Hz) clearly indicated the presence of a *cis*-*p*-coumaroyl substituent in **2d**.

The ^{1}H NMR spectrum of compound **2e** (Table 3) showed singlets at δ 7.48 (H-3) and 5.75 (H-1), again characteristic of a secoiridoid. A doublet at δ 4.83 (CH_3-10) and a series of signals between δ 4.51 and 3.51 indicated the presence of a secoiridoid glycoside. A COSY experiment enabled assignments to be established for the protons of this sugar moiety and showed a correlation between a vinylic proton chemical shift at δ 6.10 and the signal attributable to the C-10 methylene protons (δ 4.75 and the signal superimposed on the anomeric proton). It was evident that a double bond was present between C-8 and C-9, which was supported by the respective values for these carbons (124.0 and 131.6 ppm) in the ^{13}C NMR spectrum. The presence of signals at δ 3.74 and 3.62, integrating for three protons in each case, were assignable to two methyl ester groups, while the signals at δ 2.07 and 2.13 corresponded to two acetyl groups. The HMBC experiment led to the observation of a correlation between the methoxy signal at δ 3.74 and the carbonyl carbon at δ 166.4 ppm. A cross-peak of the latter with H-3 indicated that the C-11 carbonyl carbon appeared at 166.4 ppm. In addition, chemical shifts at δ 3.62 and 171.1 ppm were correlated with the latter also being associated with the methylene group at C-6. This observation strongly indicated that the C-7 carbonyl resonated at δ 171.1. The HMBC experiment also showed cross-peaks for the acetyl group at δ 2.07 with the carbonyl carbon at 171.1 ppm, and the latter value with the methylene group at C-10. Therefore, both the C-7 carbonyl carbon and the C-10 acetyl group resonated at about δ 171. The acetyl group at δ 2.13, corresponding to the carbonyl signal at 171.8 ppm was assigned to the position C-6′, due to the fact that the protons of this last group were shifted downfield with respect to the remainder of the

Table 3. ^{1}H NMR Data in $CDCl_3$ of Compounds **1c**,**d** and **2c**−**e**

H	1c	1d	2c	2d	2e
1	5.25 d (5.9)	5.24 d (4.7)	5.75 s	5.72 s	5.75 s
3	7.42 d (1.2)	7.43 d (1.2)	7.48 s	7.46 s	7.48 s
5	3.08 m	3.10 m	4.04 dd (9.3, 4.1)	4.00 dd (9.3, 4.1)	4.02 dd (9.5, 4.0)
6_A	2.32 dd (17.0, 9.5)	2.35 dd (17.0, 8.9)	2.46 dd (15.2, 9.3)	2.43 dd (15.2, 9.3)	2.43 dd (15.3, 9.5)
6_B	2.98 dd (17.0, 8.3)	3.01 dd (17.0, 6.6)	2.83 dd (15.2, 4.1)	2.80 dd (15.2, 4.1)	2.83 dd (15.3, 4.0)
8	4.32−4.28 m	4.35−4.21 m	6.11 t (6.2)	6.05 t (6.2)	6.10 t (6.2)
9	2.06−2.01 m	2.07−2.01 m			
10_A			4.88 dd (13.8, 6.2)	4.79 dd (13.8, 6.2)	4.75 dd (13.2, 6.2)
10_B			4.94 dd (13.8, 6.2)	4.88 dd (13.8, 6.2)	4.88−4.82 m
CH_3	1.44 d (6.2)	1.42 d (6.4)			
1′	4.88 d (6.4)	4.86 d (8.0)	5.05 d (7.8)	5.04 d (7.8)	4.83 d (7.8)
2′	5.01 dd (6.4, 7.7)	4.99 dd (8.0, 9.6)	5.13 t (8.8)	5.13 m	3.55−3.42 m
3′	5.23 t (7.7)	5.23 t (9.6)	5.27 t (9.3)	5.27 t (9.3)	3.64 (overlapped with H-1′)
4′	5.13 t (7.7)	5.06 t (9.6)	5.13 t (8.8)	5.13 m	3.55−3.42 m
5′	3.79 m	3.77−3.73 m	3.80−3.75 m	3.79−3.76 m	3.55−3.42 m
$6'_A$	4.32−4.28 m	4.35−4.21 m	4.14 dd (12.4, 2.3)	4.14 dd (12.4, 2.3)	4.27 dd (12.3, 2.0)
$6'_B$	4.39 dd (12.4, 2.6)	4.35−4.21 m	4.31 dd (12.4, 4.7)	4.31 dd (12.4, 4.7)	4.51 dd (12.3, 4.3)
2″ + 6″	7.54 d (8.5)	7.66 d (8.7)	7.54 d (8.8)	7.67 d (8.8)	
3″ + 5″	7.11 d (8.5)	7.09 d (8.7)	7.10 d (8.8)	7.09 d (8.8)	
7″	7.66 d (16.0)	6.98 d (12.6)	7.69 d (16.0)	6.91 d (12.8)	
8″	6.38 d (16.0)	5.97 d (12.6)	6.40 d (16.0)	5.96 d (12.8)	
CH_3CO-7			3.65 s	3.63 s	3.62 s
CH_3CO-11	3.70 s	3.73 s	3.74 s	3.73 s	3.74 s
CH_3-OAc	1.95, 1.99, 2.03, 2.29, each s	1.96, 2.01, 2.02, 2.30, each s	2.01, 2.02, 2.03, 2.09, 2.31, each s	2.01, 2.02, 2.03, 2.08, 2.31, each s	2.07, 2.13, each s

Table 4. ^{13}C NMR Data in $CDCl_3$ of Compounds **1c**,**d** and **2c**−**e**

C	1c	1d	2c	2d	2e
1	93.8 d	93.8 d	92.8 d	92.8 d	93.3 d
3	151.4 d	151.4 d	152.9 d	152.9 d	153.0 d
4	109.9 s	110.1 s	108.3 s	108.3 s	108.2 s
5	25.3 d	25.2 d	30.9 d	30.9 d	30.9 d
6	33.3 t	33.3 t	39.9 t	40.0 t	39.9 t
7	170.6 s	170.6 s	171.1 s	171.0 s	171.1 s
8	73.2 d	73.2 d	124.2 d	124.0 d	124.0 d
9	40.7 d	40.8 d	131.3 s	131.4 s	131.6 s
10	20.4 q	20.4 q	60.7 t	60.5 t	60.6 t
11	166.1 s	166.1 s	166.3 s	166.3 s	166.4 s
1′	96.3 d	96.4 d	97.0 d	97.0 d	99.5 d
2′	70.5 d	70.5 d	72.2 d	70.7 d	73.1 d
3′	72.3 d	72.2 d	72.4 d	72.5 d	74.5 d
4′	68.3 d	68.4 d	68.1 d	68.2 d	75.8 d
5′	72.3 d	72.2 d	72.4 d	72.2 d	69.5 d
6′	61.5 t	61.7 t	61.6 t	61.7 t	62.9 t
1″	131.7 s	132.1 s	132.1 s	132.2 s	
2″ + 6″	129.3 d	131.3 d	129.3 d	131.4 d	
3″ + 5″	122.1 d	121.2 d	122.1 d	121.2 d	
4″	152.3 s	151.4 s	152.1 s	151.2 s	
7″	144.8 d	144.1 d	144.0 d	142.8 d	
8″	117.0 d	118.4 d	117.9 d	119.1 d	
9″	166.1 s	165.1 s	166.4 s	165.5 s	
$OCOCH_3$	21.1 s	20.3 s	20.4 s	20.5 s	20.8 q
	20.6 s	20.4 s	20.5 s	20.5 s	
	20.5 s	20.5 s	20.6 s	20.6 s	
	20.3 s	21.1 s	21.1 s	21.1 s	
$OCOCH_3$	170.0 s	170.1 s	170.6 s	169.1 s	171.8 s
	169.2 s	169.3 s	170.1 s	169.2 s	166.4 s
	168.9 s	169.1 s	169.3 s	169.3 s	
		169.0 s	169.2 s	170.1 s	
			169.1 s	170.5 s	

protons of the sugar system. Acetylation of **2e** gave the 10,2′,3′,4′,6′-pentaacetoxyoleoside dimethyl ester **2f**, described previously by Inouye *et al.*[10]

Experimental Section

General Experimental Procedures. IR spectra were taken on a Perkin-Elmer PE1605 FT-IR spectrophotometer. UV spectra were measured on a JASCO V-560 instrument. Optical rotations were determined with a Perkin-Elmer Model 241 polarimeter. ^{1}H and ^{13}C NMR spectra were measured on a Bruker AMX-400 spectrometer using TMS as internal standard. MS were obtained using a Micromass Autospec spectrometer. HPLC separations were run on a SMI Concept apparatus using a Kromasil 100 C_{18} column (5 μm, 25 × 1.0 cm) and methanol−water mixtures as eluants.

Plant Material. Fresh aerial parts of *J. odoratissimum* (1.1 kg) were collected in September 1994 in San Andrés (Tenerife, Canary Islands). A voucher specimen is deposited at the Herbarium of the Centro de Productos Naturales "Antonio González" (no. HPJ-A0566).

Extraction and Isolation. The aerial parts of *J. odoratissimum* (600 g) were extracted with refluxing EtOH and were then concentrated under a vacuum. The residue was partitioned between EtOAc and H_2O, and the vacuum-concentrated EtOAc extract (390 g) was chromatographed over a silica gel column, eluted with hexanes−EtOAc and EtOAc−MeOH mixtures of increasing polarity. The vacuum-concentrated 100% MeOH fraction weighed 82.5 g and was chromatographed over a further silica gel column using hexanes−EtOH mixtures. The fractions so obtained were purified by reversed-phase HPLC using MeOH−H_2O for elution, yielding 47.5 mg of a mixture of **1a** and **1b**, 30 mg of a mixture of **2a** and **2b**, and 14.4 mg of **2e**.

6′-*O*-*trans*-*p*-Coumaroyl-8-epikingiside (1a) and 6′-*O*-*cis*-*p*-coumaroyl-8-epikingiside (1b): oil; $[\alpha]^{25}_D$ −43.8° (*c* 1.2, MeOH); UV (MeOH) λ_{max} (log ϵ) 230 (3.82), 313 (3.75); ^{1}H NMR data, see Table 1; ^{13}C NMR data, see Table 2; FABMS *m*/*z* $[M + Na]^+$ 573 (29), $[M + H]^+$ 551 (9), 453 (13), 425 (16), 281 (15), 147 (100)); HR-FABMS *m*/*z* $[M + Na]^+$ 573.1579 (calcd for $C_{26}H_{30}NaO_{13}$, 573.1584).

10-*trans*-(*p*-Coumaroyloxy)oleoside dimethyl ester (2a) and 10-*cis*-(*p*-Coumaroyloxy)oleoside dimethyl ester (2b): oil; $[\alpha]^{25}_D$ −165.8° (*c* 1, MeOH); UV (MeOH) λ_{max} (log ϵ) 231 (4.03), 300 (3.73), 312 (3.75); ^{1}H NMR data, see Table 1; ^{13}C NMR data, see Table 2; FABMS *m*/*z* $[M + Na]^+$ 603 (24), $[M + H]^+$ 581 (15), 503 (7), 429 (38), 355 (48), 149 (100)); HRFABMS *m*/*z* $[M + Na]^+$ 603.1682 (calcd for $C_{27}H_{32}NaO_{14}$, 603.1689).

Formation of Acetyl Derivatives (1c,d, and 2c,d).

Each compound (10 mg) was acetylated with acetic anhydride and pyridine (1:1) at room temperature overnight.

6′-*O*-*trans*-*p*-Coumaroyl-2′,3′,4′,4″-*O*-tetraacetyl-8-epikingiside (1c): oil; $[\alpha]^{25}_D$ −17° (*c* 2, $CHCl_3$); UV (EtOH) λ_{max} (log ϵ) 219 (4.33), 223 (4.34), 283 (4.32) nm; IR ν_{max} 2942 (CH), 1754 and 1713 (C═O), 1637, 1601, 1501, 1437, 1372, 1161, 1095, 1072, 1037, 1008, 979 cm^{-1}; ^1H NMR data, see Table 3; ^{13}C NMR data, see Table 4; FABMS *m/z* $[M + Na]^+$ 741 (91), $[M + H]^+$ 719 (25), 477 (16), 189 (100), 176 (48), 154 (57), 147 (52), 139 (34), 137 (38); HRFABMS *m/z* $[M + Na]^+$ 741.2015 (calcd for $C_{34}H_{38}NaO_{17}$, 741.2007), [M − esterified glucose]$^+$ 477.1399 (calcd for $C_{23}H_{25}O_{11}$, 477.1396).

6′-*O*-*cis*-*p*-Coumaroyl-2′,3′,4′,4″-*O*-tetraacetyl-8-epikingiside (1d): oil; $[\alpha]^{25}_D$ −20° (*c* 0.7, $CHCl_3$); UV (EtOH) λ_{max} (log ϵ) 219 (4.33), 223 (4.32), 281 (4.29) nm; IR ν_{max} 2953 (CH), 1757 and 1713 (C═O), 1636, 1602, 1505, 1438, 1370, 1246, 1096, 1074, 1038, 980, 915, 856 cm^{-1}; ^1H NMR data, see Table 3; ^{13}C NMR data, see Table 4; FABMS *m/z* $[M + Na]^+$ 741 (100), $[M + H]^+$ 719 (27), 477 (13), 189 (31), 176 (22), 154 (30), 147 (23), 139 (16), 137 (19); HRFABMS *m/z* $[M + Na]^+$ 741.2009 (calcd for $C_{34}H_{38}NaO_{17}$, 741.2007).

10-*trans*-(*p*-Coumaroyloxy)-2′,3′,4′, 6′,4″-*O*-pentaacetyloleoside dimethyl ester (2c): syrup; $[\alpha]^{25}_D$ −107° (*c* 0.48, $CHCl_3$); UV (EtOH) λ_{max} (log ϵ) 219 (4.22), 223 (4.20), 283 (4.37) nm; IR ν_{max} 2954 (CH), 1755 and 1707 (C═O), 1637, 1601, 1508, 1437, 1367, 1161, 1102, 1067, 1043, 985, 908, 832 cm^{-1}; ^1H NMR data, see Table 3; ^{13}C NMR data, see Table 4; FABMS *m/z* $[M + Na]^+$ 813 (100), 443 (27), 331(53), 189 (24), 176 (25), 169 (76), 154 (35); HRFABMS *m/z* $[M + Na]^+$ 813.2209 (calcd for $C_{37}H_{42}NaO_{19}$, 813.2218).

10-*cis*-(*p*-Coumaroyloxy)-2′,3′,4′,6′,4″-*O*-pentaacetyloleoside dimethyl ester (2d): syrup; $[\alpha]^{25}_D$ −144° (*c* 1, $CHCl_3$); UV (EtOH) λ_{max} (log ϵ) 220 (4.37), 224 (4.37), 240 (sh, 4.33) 278 (4.26) nm; IR ν_{max} 2919 (CH), 1754 and 1707 (C═O), 1631, 1601, 1502, 1437, 1367, 1161, 1096, 1072, 1043, 908 cm^{-1}; ^1H NMR data, see Table 3; ^{13}C NMR data, see Table 4; FABMS *m/z* $[M + Na]^+$ 813 (100), 443 (37), 331(69), 189 (45), 176 (65), 169 (93), 154 (74); HRFABMS *m/z* $[M + Na]^+$ 813.2211 (calcd for $C_{37}H_{42}NaO_{19}$, 813.2218).

6′-*O*-Acetyl-10-acetoxyoleoside dimethyl ester (2e): oil; $[\alpha]^{25}_D$ −63° (*c* 0.55, $CHCl_3$); UV (EtOH) λ_{max} (log ϵ) 235 (4.11), 314 (3.03) nm; IR ν_{max} 2919 (CH), 1731 (C═O), 1637, 1601, 1437, 1372, 1296, 1237, 1161, 1079, 1038, 949, 908 cm^{-1}; ^1H NMR data, see Table 3; ^{13}C NMR data, see Table 4; FABMS *m/z* $[M + Na]^+$ 541 (80), $[M + H]^+$ 519 (6), 329 (47), 307 (37), 297 (28), 176 (94), 154 (100), 136 (52); HRFABMS *m/z* $[M + Na]^+$ 541.1537 (calcd for $C_{22}H_{30}NaO_{14}$, 541.1533).

Formation of acetate (2f). Compound **2e** (8 mg) was acetylated with acetic anhydride and pyridine at room temperature overnight to give **2f**.

Acknowledgment. This work was supported by the Gobierno de Canarias, Consejería de Educación, Grant No. 92/136.

References and Notes

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NP970272O