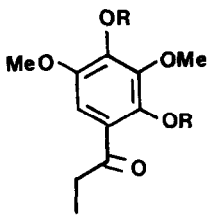
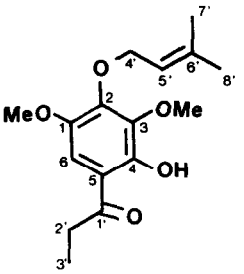
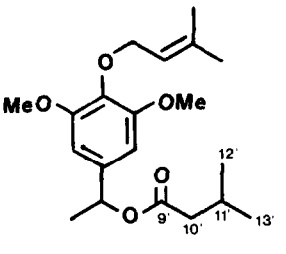


1 R=H
1a R=Ac



2



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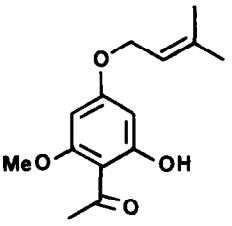

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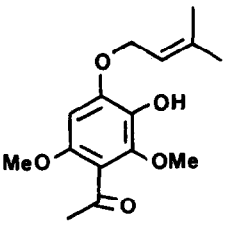

5

genated aromatic ring. The IR spectrum showed intense absorption at 1730 cm^{-1} (C=O, ester) and the ^1H NMR spectrum (Table 1) was in agreement with a 1,2,3,4-tetrasubstituted aromatic ring with *O*-isopentenyl, $MeCHOOCCHMe_2$ and two methoxyl groups as substituents. The chemical shifts for the two aromatic protons (δ7.19) in the ^1H NMR and the aromatic carbon atoms in the ^{13}C NMR (Table 2), showed that two methoxyl groups were at C-1 and C-3, and the *O*-isopentenyl group, at C-2. All these data allowed us to identify **3** as 5-(1′-isovalerianoyloxy)ethyl-2-*O*-isopentenyl-1,3-di-*O*-methylpyrogallol.

Table 1. ^1H NMR spectral data for **1**, **1a**, **2** and **3** [200 MHz, $CDCl_3$, *J* (Hz) in parentheses]

H	1	1a	2	3
H-4	—	—	—	7.19 *s*
H-6	6.91 *s*	7.11 *s*	6.96 *s*	7.19 *s*
H-1′	—	—	—	5.96 *q* (6.9)
H-2′	2.95 *q* (7.2)	2.89 *q* (7.2)	2.90 *q* (7.3)	1.52 *d* (6.9)
H-3′	1.23 *t* (7.2)	1.23 *t* (7.2)	1.22 *t* (7.3)	—
H-4′	—	—	4.58 *d* (7.2)	4.58 *d* (7.4)
H-5′	—	—	5.49 *t* (7.2)	5.52 *t* (7.4)
H-7′	—	—	1.72 *s*	1.68 *s*
H-8′	—	—	1.74 *s*	1.75 *s*
H-10′	—	—	—	2.27 *d* (6.3)
H-11′	—	—	—	2.17 *m*
H-12′	—	—	—	0.99 *d* (6.5)
H-13′	—	—	—	0.99 *d* (6.5)
OMe	3.85 *s*	3.82 *s*	3.77 *s*	3.90 *s*
OMe	3.95 *s*	3.86 *s*	3.88 *s*	3.90 *s*
2-OAc	—	2.36 *s*	—	—

EXPERIMENTAL

Mps are uncorr. UV spectra were recorded in MeOH and optical rotations in $CHCl_3$. ^{13}C and ^1H NMR were recorded in $CDCl_3$ on a 200 MHz spectrometer. Chemical shifts are reported

Table 2. ^{13}C NMR data of compounds **1**, **1a**, **2** and **3** (50.3 MHz, $CDCl_3$)

Carbon	1	1a	2	3
1	153.00	150.11	153.18	153.84
2	140.10	137.04	142.78	129.73
3	135.19	146.02	135.20	153.84
4	146.21	137.02	145.83	106.37
5	111.33	129.35	114.20	142.44
6	106.85	106.73	107.21	106.37
1′	205.21	199.43	205.34	70.77
2′	31.42	35.28	31.66	25.81
3′	8.46	8.24	8.35	—
4′	—	—	70.14	69.71
5′	—	—	120.32	120.50
6′	—	—	138.80	138.60
7′	—	—	25.78	25.81
8′	—	—	18.05	17.32
9′	—	—	—	172.52
10′	—	—	—	43.22
11′	—	—	—	25.47
12′	—	—	—	22.42
13′	—	—	—	22.42
OMe	56.94	56.50	57.02	56.46
OMe	60.80	61.38	60.90	56.46
OAc	—	167.81	—	—
	—	20.14	—	—
OAc	—	168.92	—	—
	—	20.78	—	—

in δ-values downfield from int. TMS and the coupling constants are quoted in Hz.

Collection and extraction. L. pallida subsp. *flaveola*, was collected in the Sierra de Béjar (Candelario, Salamanca, W. Spain) at the end of May, 1982. The plant material was identified by Prof. M. Ladero, from Botany Department of the Faculty of Pharmazie (Salamanca) where a specimen is held (Herbarium No 8556).

Aerial parts of the plant (3.5 kg), air dried and finely ground, were extracted with hot $CHCl_3$–MeOH (4:1). The extract was concd in *vacuo* and the residual syrup (204 *g*) extracted with EtOH–H_2O (2:3). The alcoholic soln was extracted with $CHCl_3$. The $CHCl_3$ extract was washed with aq. $NaHCO_3$, dried and the solvent removed to give 10.8 *g* of residue. This was chromatographed on silica gel (hexane, hexane–EtOAc) giving **1** (41 mg), **2** (36 mg), **3** (73 mg), **4** (137 mg), **5** (30 mg), (+)-sesamin (92 mg) and cumambrin A (180 mg).

4-Hydroxy-5-propionyl-1,3-di-O-methylpyrogallol, **1**. Colourless solid, mp 122–123°; UV λ_{max} nm (ε): 217 (5200), 284 (3480); $\lambda_{max}^{AlCl_3-HCl}$: 213 (6989), 312 (5000); IR ν_{max} cm^{-1}: 3500, 1640, 1600, 1500, 1210, 1180, 1110, 1075, 1025, 850; Acetylation of **1** gave **1a**, viscous oil; IR ν_{max} cm^{-1}: 1770, 1695, 1605, 1500, 1240, 1210, 1180, 1150, 1075, 930, 880, 815; EIMS *m/z* (%): 310 $[M]^+$ (4.2), 268 (33), 226 (100), 197 (93), 182 (3), 167 (2), 139 (1).

4-Hydroxy-5-propionyl-1,3-di-O-methyl-2-O-isopentenylpyrogallol, **2**. UV λ_{max} nm (ε): 242 (5800), 275 (8930) nm; $\lambda_{max}^{AlCl_3-HCl}$: 238 (6074), 300 (7618); IR ν_{max} cm^{-1}: 3500, 1645, 1605, 1500, 1240, 1170, 1120, 1080, 950, 840.

5-(1'-Isovalerianoyloxy)ethyl-2-O-isopentenyl-1,3-di-O-methylpyrogallol, **3**.

λ Hg	578	546	436	365
α	−11.6	−10.46	−8.60	−6.74

(*c* 0.43).

UV λ_{max} nm (ε): 215 (5000), 284 (8590); IR ν_{max} cm^{-1}: 1730, 1630, 1590, 1500, 1240, 1180, 1130, 1050, 970, 860, 830.

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