Chemical Constituents from the Pericarp of *Trewia nudiflora*

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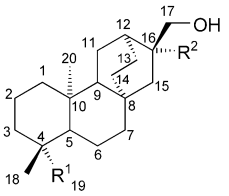
Three new *ent*-atisane diterpenes, *i.e.*, 17-hydroxy-*ent*-atisan-19-oic acid (**1**), 17-hydroxy-*ent*-atisan-19-oic acid methyl ester (**2**), and 16α,17-dihydroxy-*ent*-atisan-19-al (**3**), together with five known phenolic compounds, *i.e.*, gallic acid, ethyl gallate, protocatechuic acid, 3,4,4′-tri-*O*-methylellagic acid, and α-tocopherol, and two other known compounds, *i.e.*, *trans*-cinnamic acid and taraxerone, were isolated from the pericarp of *Trewia nudiflora* collected in Xishuangbanna, Yunnan Province, China. Their structures were elucidated by spectroscopic analysis including 1D- and 2D-NMR. Trace amounts of maytansinoids were isolated by antifungal-activity-guided fractionation and determined by LC-ESI-MS analysis; they were prominent antifungal constituents in the pericarp of *Trewia nudiflora*.

1. Introduction. – *Trewia nudiflora* L. (Euphorbiaceae) is a tropical plant mainly distributed in India, Malaysia, and the south of China [1]. The seed of *T. nudiflora* contains highly unusual glyceride oil [2], several novel pyridinone alkaloids [3], and an inhibitor of protein synthesis [4]. The seed is also a rich source of maytansinoids [5–7]. In a search for antifungal agents from plant sources, we found that EtOH extracts of the pericarp of *T. nudiflora* showed significant antifungal activity against *Penicillium avellaneum* UC-4376, while the chemical constituents of the pericarp have not been investigated so far. Bioactivity-guided isolation as well as traditional phytochemical isolation were used in our studies on the constituents of the pericarp. Herein, we report the isolation and structural elucidation of compounds from the pericarp of *T. nudiflora*.

2. Results and Discussion. – The pericarp of *T. nudiflora* was extracted with 95% EtOH. The petroleum ether soluble part of the EtOH extract was successively chromatographed over silica gel and *Sephadex LH-20* to afford compounds **1**, **2**, taraxerone (=(13α)-13-methyl-27-norolean-14-en-3-one), and α-tocopherol (=vitamin E=(2*R*)-3,4-dihydro-2,5,7,8-tetramethyl-2-[(4*R*,8*R*)-4,8,12-trimethyltridecyl]-2*H*-1-benzopyran-6-ol). The BuOH-soluble part of the EtOH extract yielded gallic acid (=3,4,5-trihydroxybenzoic acid), ethyl gallate, and protocatechuic acid (=3,4-dihydroxybenzoic acid). The $CHCl_3$-soluble part of the EtOH extract showed antifungal activity against *Penicillium avellaneum* UC-4376. Bioassay-guided fractionation of this part led to the isolation of trace amount of maytansinoids, which were identified by LC-ESI-MS. The nonactive fraction of the $CHCl_3$-soluble part was subjected to repeated chromatography to afford compound **3**, *trans*-cinnamic acid (=(2*E*)-3-phenylprop-2-enoic acid), and 3,4,4′-tri-*O*-methylellagic acid (=8-hydroxy-2,3,7-trimethoxy[1]-benzopyrano[5,4,3-*cde*][1]benzopyran-5,10-dione). Compounds **1**–**3** were very similar to each other according to their ^{1}H- and ^{13}C-NMR spectra (*Table 1*).



1 R^1 = COOH, R^2 = H
2 R^1 = COOMe, R^2 = H
3 R^1 = CHO, R^2 = OH

Table 1. *^{1}H- and ^{13}C-NMR Data* ($CDCl_3$) *for Compounds* **1**–**3**. δ in ppm, J in Hz.

	1		**2**		**3**	
	δ(H)	δ(C)	δ(H)	δ(C)	δ(H)	δ(C)
H_α–C(1)	1.86 (*m*, 1 H)	40.7 (*t*)	1.83 (*m*, 1 H)	40.8 (*t*)	1.80 (*m*, 1 H)	39.8 (*t*)
H_β–C(1)	0.80 (*m*, 1 H)		0.77 (*m*, 1 H)		0.80 (*m*, 1 H)	
H_α–C(2)	1.41 (*m*, 1 H)	19.1 (*t*)	1.40 (*m*, 1 H)	19.2 (*t*)	1.56 (*m*, 2 H)	18.5 (*t*)
H_β–C(2)	1.89 (*m*, 1 H)		1.84 (*m*, 1 H)			
H_α–C(3)	2.16 (*m*, 1 H)	37.8 (*t*)	2.15 (*m*, 1 H)	38.1 (*t*)	2.15 (*m*, 1 H)	34.4 (*t*)
H_β–C(3)	0.99 (*m*, 1 H)		0.96 (*m*, 1 H)		0.94 (*m*, 1 H)	
C(4)		43.7 (*s*)		43.8 (*s*)		48.5 (*s*)
H_β–C(5)	1.05 (*m*, 1 H)	57.0 (*d*)	1.01 (*m*, 1 H)	57.1 (*d*)	1.14 (*m*, 1 H)	56.7 (*d*)
H_α–C(6)	1.75 (*m*, 1 H)	22.4 (t)	1.71 (*m*, 1 H)	22.5 (*t*)	1.56 (*m*, 1 H)	18.4 (*t*)
H_β–C(6)	1.82 (*m*, 1 H)		1.85 (*m*, 1 H)		1.01 (*m*, 1 H)	
H–C(7) or CH_2(7)	1.44 (*m*, 2 H)	41.6 (*t*)	1.43 (*m*, 2 H)	41.7 (*t*)	1.71 (*m*, 1 H)	42.0 (t)
H′–C(7)					1.52 (*m*, 1 H)	
C(8)		44.8 (*s*)		44.8 (*s*)		44.6 (*s*)
H–C(9)	1.03 (*m*, 1 H)	55.3 (*d*)	0.98 (*m*, 1 H)	55.3 (*d*)	1.01 (*m*, 1 H)	55.4 (*d*)
C(10)		39.6 (*s*)		39.4 (*s*)		40.7 (*s*)
H–C(11) or CH_2(11)	1.50 (*m*, 1 H)	18.9 (*t*)	1.57 (*m*, 2 H)	18.9 (*t*)	1.56 (*m*, 1 H)	20.2 (*t*)
H′–C(11)	1.60 (*m*, 1 H)				1.92 (*m*, 1 H)	
H–C(12)	2.06 (*m*, 1 H)	38.2 (*d*)	2.04 (*m*, 1 H)	38.2 (*d*)	2.02 (*m*, 1 H)	45.5 (*d*)
H–C(13)	1.84 (*m*, 1 H)	37.2 (*t*)	1.80 (*m*, 1 H)	37.2 (*t*)	1.90 (*m*, 1 H)	37.5 (*t*)
H′–C(13)	0.97 (*m*, 1 H)		0.97 (*m*, 1 H)		1.66 (*m*, 1 H)	
H–C(14) or CH_2(14)	1.58 (*m*, 1 H)	31.4 (*t*)	1.55 (*m*, 1 H)	31.4 (*t*)	1.54 (*m*, 2 H)	26.1 (*t*)
H′–C(14)	1.42 (*m*, 1 H)		1.41 (*m*, 1 H)			
H–C(15)	1.55 (*m*, 1 H)	45.0 (*t*)	1.53 (*m*, 1 H)	45.0 (*t*)	1.59 (*m*, 1 H)	53.2 (*t*)
H′–C(15)	0.88 (*m*, 1 H)		0.89 (*dd*, J = 5.0, 13.0)		1.48 (*m*, 1 H)	
H–C(16)	1.95 (*m*, 1 H)	43.3 (*d*)	1.93 (*m*, 1 H)	43.4 (*d*)		81.8 (*s*)
H–C(17) or CH_2(17)	3.44 (*m*, 2 H)	67.5 (*t*)	3.41 (*m*, 2 H)	67.6 (*t*)	3.78 (*m*, 1 H)	66.4 (*t*)
H′–(17)					3.66 (*m*, 1 H)	
Me(18)	1.23 (*s*, 3 H)	29.0 (*q*)	1.14 (*s*, 3 H)	28.8 (*q*)	0.97 (*s*, 3 H)	24.3 (*q*)
C(19)OO or CH(19)O		183.7 (*s*)		178.0 (*s*)	9.72 (*s*, 1 H)	205.7 (*s*)
Me(20)	0.93 (*s*, 3 H)	15.6 (*q*)	0.79 (*s*, 3 H)	15.4 (*q*)	0.86 (*s*, 3 H)	
MeO			3.62 (*s*)	51.1 (*q*)		

Compound **1** (colorless crystals) was determined to have the molecular formula $C_{20}H_{32}O_3$ based on the HR-FAB-MS (*m*/*z* 321.2427 ($[M+H]^+$; calc. 321.2430)). The spectroscopic data and their comparison with those of known compounds established its structure as being 17-hydroxy-*ent*-atisan-19-oic acid (**1**).

The IR spectra of **1** revealed the presence of an OH (3431, 2929 cm^{-1}) and a COOH function (1698 cm^{-1}). The COOH signal was also present in the ^{13}C-NMR spectrum (δ(C) 183.7 (*s*)). The remaining four degrees of unsaturation indicated that **1** was a tetracyclic diterpene. The ^{13}C-NMR and DEPT spectra (*Table 1*) showed 20

resonances for four quaternary C-atoms and four CH, ten CH_2, and two Me groups. In the ^{1}H-NMR spectra, two angular Me groups appeared at δ(H) 1.23 and 0.93 and a CH_2OH signal at δ(H) 3.44. HMQC, HMBC, and ^{1}H,^{1}H-COSY experiments yielded sufficient data to define the molecular connectivity. In the HMBC experiment (*Table 2*), the ^{1}H,^{13}C-NMR long-range correlations between the protons of the Me group at δ(H) 1.23 (δ(C) 29.0) and the C-atoms at δ(C) 37.8 (*t*), 43.7 (*s*), 57.0 (*d*), 183.7 (*s*), and 19.1 (*t*) (weak), and between the protons of the Me group at δ(H) 0.93 (δ(C) 15.6) and the C-atoms at δ(C) 39.6 (*s*), 40.7 (*t*), 55.3 (*d*), and 57.0 (*d*) afforded the fragment **1a** (*Figure*). The ^{13}C-NMR chemical shifts were similar to those of known *ent*-atisane diterpenes in the A ring [8]. Analysis of the ^{1}H-,^{1}H-COSY plots revealed that the protons of CH_2OH at δ 3.44 (CH_2 (17)) were correlated to the protons at δ 1.95 (H–C(16)), which together with the HMBC data led to the fragment **1b** (*Figure*). The *ABM*$_2$- and *ABN*-spin systems revealed by the ^{1}H-,^{1}H-COSY experiment were attributed to CH_2(6) at δ 1.82 (*m*) and 1.75 (*m*) correlating with CH_2(7) at δ 1.44 and H–C(5) at δ 1.05, thus determining the structure of fragment **1c**. The quaternary C-atom at δ 39.6 (C(10)) showed long-range correlation to the protons at δ 1.03 (H–C(9)) and 0.93 (Me(20)) in the fragment **1a**, and to the proton at δ 1.60 (H′–C(11)), determining the structure of fragment **1d** (*Figure*). Fragments **1b** and **1d** can be connected based on the correlation between the proton at δ 1.50 (H–C(11)) and the C-atom at δ(C) 38.2 (*d,* C(12)). Based on the correlations between **1c** and **1d**, all of the four substructures were connected to give the full molecular connectivity. The relative configuration was deduced from the ROESY experiment and based on the fact that all previously reported atisane-type diterpenes from the family Euphorbiaceae are *ent*-atisane diterpenes [9]. An NOE was observed for Me(18) (δ 1.23) and H_β–C(5) (δ 1.05), which, therefore, determined the β-position of Me(18) and the α-position of C(19)OOH. The β-position of CH_2(17)OH was evidenced by the NOE for CH_2(17) (δ 3.44) and Me(20) (δ 0.93) and H–C(12) (δ 2.06).

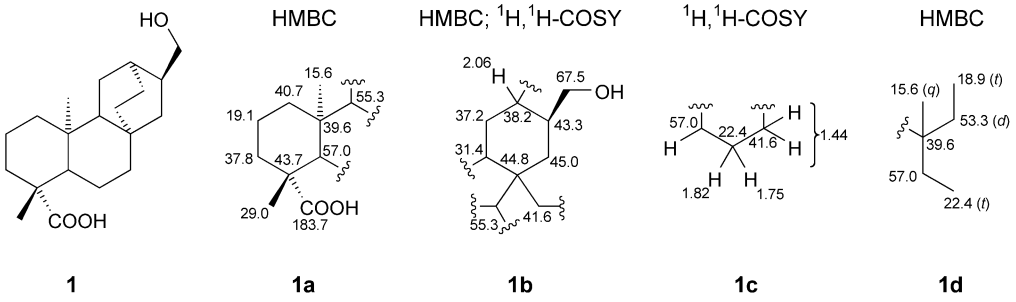


Figure. *^{1}H- and ^{13}C-NMR Chemical shifts of substructures of compound* **1** *as determined by HMBC and COSY experiments*

A less polar fraction of the extract yielded compound **2**, which was assigned the formula $C_{21}H_{34}O_3$ by HR-FAB-MS (*m/z* 335.2566 ($[M+H]^+$; calc. 335.2586)). A comparative study of the ^{1}H- and ^{13}C-NMR values of **2** and **1** indicated that the two compounds are very similar. The principal difference in the NMR data was that **2** exhibited methyl ester signals (δ(H) 3.61, δ(C) 51.1) that were missing in the spectra of **1**. This was confirmed by the IR spectra (**1**: COOH at 1698 cm^{-1}; **2**: COOMe at 1724 cm^{-1}). Thus, **2** was identified as 17-hydroxy-*ent*-atisan-19-oic acid methyl ester.

A more polar fraction of the extract yielded the minor component **3**. The EI-MS of compound **3** showed a weak (1%) peak for the molecular ion (*m/z* 320), which was assigned the formula $C_{20}H_{32}O_3$ from an accurate mass measurement of the base peak (*m/z* 289) corresponding to $[M-CH_2OH]^+$. The spectroscopic data established the structure of **3** to be 16*β*,17-dihydro-*ent*-atisan-19-al. The *ent*-configuration of **1**–**3** was assumed from the co-occurrence and close similarity of their structures.

Table 2. *HMBC Data of Compounds* **1**–**3**

	1	**2**	**3**
H_α–C(1)	C(5)	C(4), C(5)	C(9)
H_β–C(1)	C(2), C(9), C(20)	C(5), C(9), C(20)	C(2)
H_α–C(2)	C(3), C(10)	–	C(1)
H_β–C(2)	C(10), C(20), (*w*)	C(4)	–
H_α–C(3)	C(4), C(5)	–	–
H_β–C(3)	C(4), C(5), C(19)	C(2)	C(4), C(5)
H–C(4)	–	–	–
H_β–C(5)	C(6), C(20)	C(6), C(9), C(18), C(20)	C(4), C(7), C(19)
H_α–C(6)	C(7)	C(5), C(7)	–
H_β–C(6)	C(5), C(10), C(15) (*w*)	C(5)	C(4), C(10)
H–C(7)	C(5), C(6), C(15)	C(5)	–
H′–C(7)	–	–	–
H–C(8)	–	–	–
H–C(9)	C(1), C(4) (*w*), C(10), C(20)	C(1), C(14), C(20)	C(1), C(5), C(20)
H–C(10)	–	–	–
H–C(11)	C(12)	C(8), C(12)	C(9), C(17) (*w*)
H′–C(11)	C(9), C(10)	–	C(15) (*w*), C(16)
H–C(12)	C(15), C(17)	–	C(15)
H–C(13)	–	–	–
H′–C(13)	C(8), C(12), C(13)	–	–
H–C(14)	–	C(9)	–
H′–C(14)	C(15)	C(13)	–
H–C(15)	C(8)	C(8), C(14), C(17)	C(8), C(9), C(17)
H′–C(15)	C(9), C(16), C(17)	C(7), C(8), C(9), C(17)	C(7), C(9), C(16)
H–C(16)	C(13), C(17)	C(13, C(17)	–
H–C(17)	C(12), C(15)	C(12), C(15), C(16)	C(15)
H′–C(17)	–	–	–
Me(18)	C(3), C(4), C(5), C(19)	C83), C(4), C(5), C(19)	C(3), C(4), C(5)
H–C(19)	–	–	C(4)
Me(20)	C(1), C(5), C(9), C(10)	C(1), C(5), C(9), C(10)	C(1), C(9)
MeO	–	C(19)	–

The IR spectra of **3** revealed the presence of OH (3425 cm^{-1}) and CHO functions (2928, 2868, 1717 cm^{-1}). In the ^{1}H-NMR spectrum, the signals of an aldehyde function (δ(H) 9.72), two angular Me groups (δ(H) 0.86 and 0.97), and a CH_2OH group (δ(H) 3.66, 3.78) were present. The ^{13}C-NMR spectra showed that the O-atoms arose from an aldehyde (δ(C) 205.7, δ(H) 9.72), primary alcohol (δ(C) 66.4, δ(H) 3.78, 3.66), and tertiary alcohol (δ(C) 81.8). These data, together with the similarity of the spectra of **3** with those of **1** and **2**, were consistent with the presence of an atisane skeleton. The principal differences among them were that the CH(16) group (δ(C) 43.3) of **1** was replaced by a quaternary C-atom (δ(C) 81.8) in **3** suggesting its substitution by an OH group and that C(19) of **3** appeared as a CHO (δ(C) 205.7) instead of a COOH (δ(C) 183.7) function.

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Experimental Part

1. General. TLC: precoated plates (*Si gel G*) from *Qingdao Marine Chemical Factory*, Qingdao, P. R. China. Column chromatography (CC): silica gel (200–300 mesh) from *Quingdao Marine Chemical Factory*; reversed-

phase C_{18} silica gel from *Merck*, *Sephadex-LH-20* from *Amersham Biosciences*. M.p.: *XRC*-1 micromelting apparatus; uncorrected. Optical rotations: *Jasco DIP-370* digital polarimeter; $CHCl_3$ soln. IR Spectra: *Bio-Rad FTS-135* IR spectrometer; KBr pellets; in cm^{-1}. NMR-Spectra: *Inova 400* and *Bruker AM-400* or *DRX-500* spectrometers; $SiMe_4$ as internal standards; δ in ppm, J in Hz. MS: *VG Auto-Spec-3000* spectrometer in m/z (rel. %). LC-MS: *Waters HPLC-2695* and *Thermo-Finnigan LCQ-Advantage* spectrometer.

2. *Plant Material.* The pericarp of *Trewia nudiflora* was collected in Xishuangbanna, Yunnan Province, P. R. China. A voucher specimen (No. 20159, *K. M. Feng*) is deposited in the Herbarium of the Kunming Institute of Botany, Chinese Academy of Science.

3. *Extraction and Isolation.* The air-dried pericarp of *Trewia nudiflora* (2 kg) was ground and extracted with boiling 95% EtOH (3×). After evaporation, the residues were suspended in H_2O and then extracted successively with petroleum ether, $CHCl_3$, and BuOH. The petroleum ether extract (16 g) was subjected to CC (silica gel (300 g); petroleum ether/$CHCl_3$ 1:1 → $CHCl_3$, then $CHCl_3$/Me_2CO 20:1 → Me_2CO). The fraction (239 mg) eluted with petroleum ether/$CHCl_3$ 1:1 was purified by repeated CC (silica gel; petroleum ether/AcOEt 100:1 → 50:1, petroleum ether/$CHCl_3$ 4:1, resp.) to yield *α-tocopherol* (7 mg). *Taraxerone* was isolated from the fraction (65 mg) eluted with petroleum ether/$CHCl_3$ 1:2 after repeated CC (silica gel; petroleum ether/AcOEt 100:1, petroleum ether/Et_2O 100:3, resp.), isolation, and purification by CC (*Sephadex LH-20*, acetone). The fraction (324 mg) eluted with $CHCl_3$/Me_2CO 20:1 was subjected to CC (silica gel; $CHCl_3$/AcOEt 50:1) and then purified by CC (*Sephadex LH-20*): **2** (9 mg). The fraction eluted with $CHCl_3$/Me_2CO 10:1 afforded **1** (7 mg), after fractionation by repeated CC (silica gel; petroleum ether/Me_2CO 5:1, $CHCl_3$/Me_2CO 100:3 → 10:1, resp.) and further purification by CC (*Sephadex LH-20*, acetone). The $CHCl_3$ extract (9 g) was subjected to MPLC (*RP18* (130 g, 40–63 μ); MeOH/H_2O 1:1 and 7:3, then MeOH): *Fractions A.1–A.3*. *Fr. A.2* (active fraction) was further separated: *Fr. A.2.1–A.2.7*. *Fr. A.2.3* (active fraction) was separated by CC (*Sephadex LH-20*, MeOH): *Fr. A.2.3.1–A.2.3.4*. *Fr. A.2.3.2* was subjected to CC (silica gel *HF254*, $CHCl_3$/Me_2CO 20:1): **3** (3 mg) and trace amounts of *maytansinoids*. The *3,4,4′-tri*-O-*methylellagic acid* was obtained from *Fr. A.2*, and trans-*cinnamic acid* was obtained from *Fr. A.1*. The BuOH extract (20 g) was fractionated by CC (silica gel (200 g; 200–300 mesh); $CHCl_3$/MeOH 100:3, 20:1, 10:1, 5:1, 2:1 → 100% MeOH): *Fr. B.1–B.11*. The intermediate fractions were further purified. *Fr. B.4* was subjected to CC (silica gel, $CHCl_3$/AcOEt 4:1, 3:1): *Fr. B.4.1–B.4.5*, of which *Fr. B.4.4* afforded *ethyl gallate* (30 mg) and *protocatechuic acid* (20 mg) after further purification by CC (*Sephadex LH-20*). *Gallic acid* (60 mg) was obtained from *Fr. B.6* after purification by CC (*Sephadex LH-20*) and recrystallization from petroleum ether/AcOEt.

Antifungal-Activity Assay. Antifungal activity against *Penicillium avellaneum* UC-4376 of each fraction of the $CHCl_3$-soluble part was determined by the disk-diffusion assay on agar plates as described [10].

17-Hydroxy-ent-*atisan-19-oic Acid* (=*(4α,5β,8α,9β,10α,12α,16β)-17-hydroxyatisan-18-oic Acid*; **1**). Colorless needles. M.p. 166–168°. $[\alpha]_D^{25} = -44.4$ ($c = 0.45$, $CHCl_3$). IR (KBr): 3431, 2929, 2853, 1698, 1634, 1468, 1450, 1257, 1164, 1015. ^{1}H- and ^{13}C-NMR: see *Table 1*. EI-MS: 320 (22, M^+), 302 (90), 274 (100).

17-Hydroxy-ent-*atisan-19-oic Acid Methyl Ester* (**2**). Colorless needles. M.p. 97–98°. $[\alpha]_D^{25} = -87.5$ (c = 0.4, $CHCl_3$). IR (KBr): 3432, 2930, 2854, 1724, 1631, 1467, 1449, 1234, 1194, 1157. ^{1}H- and ^{13}C-NMR: see *Table 1*. EI-MS: 334(21, M^+), 302 (30), 275 (100).

16α,17-Dihydroxy-ent-*atisan-19-al* (=*4α,5β,8α,9β,10α,12α,16α)-16,17-Dihydroxyatisan-18-al*; **3**). Colorless needles. M.p. 154–156°. $[\alpha]_D^{25} = -46.7$ ($c = 0.15$, $CHCl_3$). IR (KBr): 3425, 2928, 2868, 1717, 1630, 1466, 1067, 1041, 1023. ^{1}H- and ^{13}C-NMR: see *Table 1*. EI-MS: 320 (1, M^+), 302 (2), 290 (15), 289 (100), 271 (30).

Gallic Acid. Colorless needles. ^{1}H-NMR (400 MHz, C_5D_5N): 8.08 (*s*, H–C(2), H–(6)). ^{13}C-NMR (400 MHz, C_5D_5N): 123.0 (C(1)); 110.7 (C(2); C(6)); 147.8 (C(3); C(5)); 140.7 (C(4)); 169.9 (COOH). EI-MS: 170 (100, M^+), 153 (80), 126 (92).

Ethyl Gallate. Colorless needles. ^{1}H-NMR (400 MHz, C_5D_5N): 7.78 (*s*, H–C(2), H–C(6)); 4.26 (*m*, MeCH_2); 1.16 (*m*, *Me*CH_2). ^{13}C-NMR (400 MHz, C_5D_5N): 121.8 (C(1)); 110.6 (C(2); C(6)); 148.0 (C(3)); 141.4 (C(4)); 148.0 (C(5)); 167.7 (COO); 61.1 (MeCH_2); 15.0 (*Me*CH_2). FAB-MS: 197 ($[M-1]^+$).

Protocatechuic Acid. Colorless needles. ^{1}H-NMR (400 MHz, C_5D_5N): 8.38 (*d*, $J = 2.0$, H–C(2)); 8.10 (*dd*, $J = 8.4$, 2.0, H–C(5); 7.35 (*d*, $J = 8.4$ H–C(6)). ^{13}C-NMR (400 MHz, C_5D_5N): 123.3 (C(1)); 147.1 (C(2)); 118.2 (C(3)); 152.2 (C(4)); 123.3 (C(5)); 116.2 (C(6)); 169.5 (COOH)). EI-MS: 154 (100, M^+), 137 (96), 109 (33).

trans-*Cinnamic Acid*. Pale yellow crystals. Identified by ^{1}H- and ^{13}C-NMR and comparison with data in [11]. ^{1}H-NMR (400 MHz, C_5D_5N): 8.07 (*d*, $J = 16.0$, 1 olef. H); 6.95 (*d*, $J = 16.0$, 1 olef. H); 7.32–7.63 (*m*, Ph). ^{13}C-NMR (400 MHz, C_5D_5N): 144.3 (olef. C); 121.0 (olef. C); 169.4 (COOH). EI-MS: 148 (95, M^+), 147 (93), 131 (76), 103 (95), 91 (79), 77 (100).

*3,3′,4-Tri-*O*-methylellagic Acid*. Pale yellow crystal. Identified by ^{1}H- and ^{13}C-NMR and comparison with data in [12]. EI-MS: 344 (100, M^+), 329 (20), 286 (22).

Taraxerone: Colorless needles. Identified by ^{1}H- and ^{13}C-NMR and comparison with data in [13]. Mp. 240–243°. EI-MS: 424 (M^+).

α-Tocopherol: Pale yellow oil. Identified by ^{1}H- and ^{13}C-NMR and comparison with data in [14]. EI-MS: 430 (M^+).

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