Three New Sucrose Fatty Acid Esters from *Equisetum hiemale* L.

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Three new monosubstituted sucrose fatty acid esters, **1**–**3**, were isolated from *Equisetum hiemale* L., together with nine known compounds, **4**–**12**. Their structures were elucidated by spectroscopic analyses. Compounds **5**, **6**, and **10**–**12** were isolated from the title plant for the first time. All these compounds were evaluated for their cytotoxic activity. However, none of them was cytotoxic.

Introduction. – The family Equisetaceae, comprised of two genera (*Equisetum* and *Hippochaete*) and about 25 species, is widely distributed in temperate regions [1]. Members of this family are known to contain several types of compounds such as flavonoids, steroids, megastigmanes, and fatty acids [2–7]. *Equisetum hiemale* L. (Equisetaceae) was used as a Traditional Chinese Medicine called as '*Muzei*' in Chinese for the treatment of eye diseases [8]. Previous chemical investigations on this plant have led to the isolation of a series of flavonoids and phenolic compounds [9]. In the course of our search for structurally unique and potentially bioactive natural products, three new monosubstituted sucrose fatty acid esters, 6-*O*-[(9*Z*,12*Z*,15*Z*)-octadeca-9,12,15-trienoyl]-β-D-fructofuranosyl α-D-glucopyranoside (**1**), 6-*O*-[(7*Z*,10*Z*,13*Z*)-hexadeca-7,10,13-trienoyl]-β-D-fructofuranosyl α-D-glucopyranoside (**2**), and 6-*O*-[(7*Z*,10*Z*)-hexadeca-7,10-dienoyl]-β-D-fructofuranosyl α-D-glucopyranoside (**3**), together with nine known compounds, β-sitosterol (**4**) [10], α-tocopherolquinone (**5**) [11], 3,3′-[propane-2,2-diylbis(benzene-4,1-diyloxy)]bis(propane-1,2-diol) (**6**) [12], *trans*-feruloyl-4-β-glucoside (**7**) [13], *trans*-ferulic acid (**8**) [14], vanillic acid (**9**) [14], (6*R*,7a*S*)-5,6,7,7a-tetrahydro-6-hydroxy-4,4,7a-trimethylbenzofuran-2(4*H*)-one (**10**) [15], 3β-hydroxy-5α,6α-epoxy-7-megastigmen-9-one (=(3*E*)-4-[(1*S*,4*R*,6*R*)-4-hydroxy-2,2,6-trimethyl-7-oxabicyclo[4.1.0]hept-1-yl]but-3-en-2-one; **11**) [16], 4-hydroxy-2,3-dimethylnon-2-en-4-olide (=5-hydroxy-3,4-dimethyl-5-pentylfuran-2(5*H*)-one; **12**) [17], were isolated from the aerial parts of *E. hiemale* L. Herein, we describe the isolation and structure elucidation of the isolates.

Results and Discussion. – The AcOEt extract of the aerial parts of *E. hiemale* L. was subjected to various column chromatographic separation columns, as well as preparative HPLC, to afford three new compounds, **1**–**3**.

Compound **1** was isolated as a colorless oil. The molecular formula was established as $C_{30}H_{50}O_{12}$ by HR-ESI-MS (*m*/*z* 625.3186 ([*M* + Na]$^+$); calc. 625.3200), correspond-

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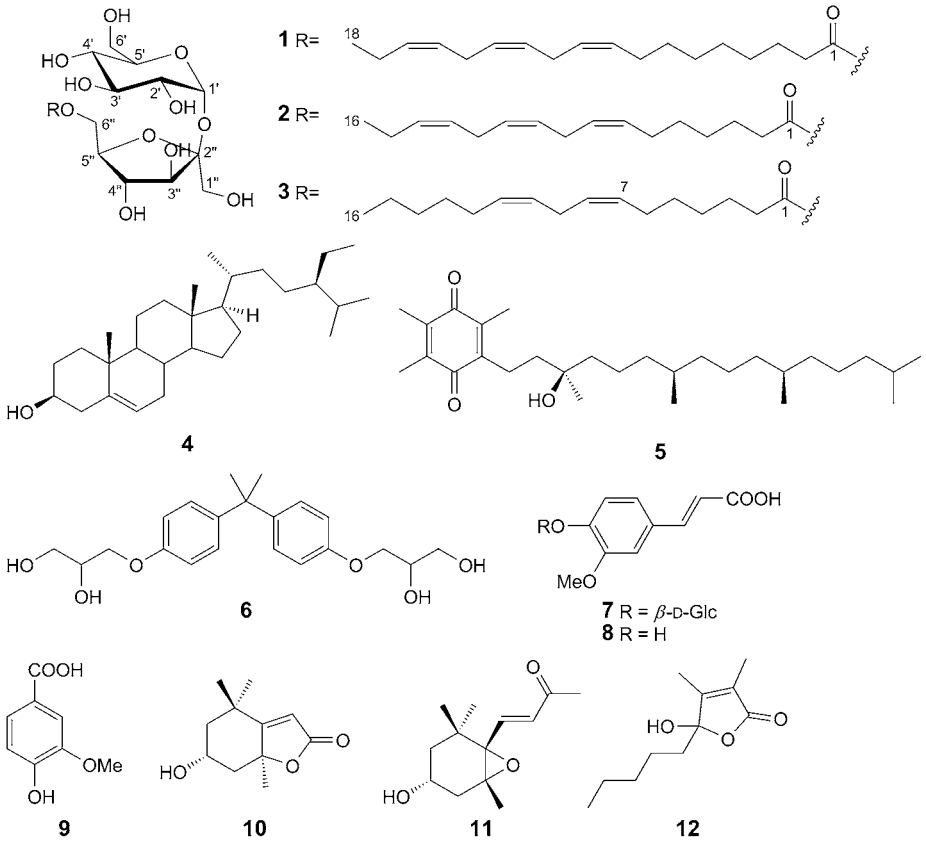

ing to six degrees of unsaturation. The IR absorption bands at 3428 and 1726 cm^{-1} implied the presence of ester and OH groups. The ^{1}H-NMR data (*Table 1*) revealed the occurrence of six olefinic H-atoms (δ(H) 5.31–5.39), four CH_2 groups (δ(H) 1.25–1.33), and one terminal Me group (δ(H) 0.98, *t*, $J = 7.5$, Me(18)). Correspondingly, the ^{13}C-NMR spectrum (*Table 2*) exhibited the signals of six olefinic C-atoms (δ(C) 132.7, 131.1, 129.2, 129.1, 128.9, 128.3), ten CH_2 groups (δ(C) 21.5–34.9), a Me group (δ(C) 14.7 (*q*, C(18)), and a CO group (δ(C) 175.5 (*s*, C(1))). These data suggested that **1** possessed a linolenic acid residue, which was confirmed by comparison of the spectroscopic data with those reported in the literature [18]. The other twelve C-atom resonances were displayed in the region of δ(C) 62.5–105.5, suggesting the existence of a disaccharide moiety, which was determined as sucrose and confirmed by alkaline hydrolysis of compound **1**. Alkaline hydrolysis of **1** with 0.5% NaOH yielded linolenic acid and sucrose (see *Exper. Part*). The fatty acid residue is attached to HO–C(6″) of sucrose, as deduced by HMBCs of the signals of CH_2(6″) (δ(H) 4.39 and 4.33) with the one of C(1) (δ(C) 175.5 (*s*)) of **1**. Based on these data, compound **1** was characterized as 6-*O*-[(9*Z*,12*Z*,15*Z*)-octadeca-9,12,15-trienoyl]-β-D-fructofuranosyl α-D-glucopyranoside.

Table 1. *^{1}H-NMR Data of Compounds* **1**–**3**. Recorded in CD_3OD; δ in ppm, J in Hz.

H-Atom	**1**	**2**	**3**
CH_2(2)	2.35 (t, J = 7.4)	2.35 (t, J = 7.5)	2.35 (t, J = 7.4)
CH_2(3)	1.60–1.61 (m)	1.61–1.65 (m)	1.61–1.65 (m)
CH_2(4)	1.25–1.33 (m)	1.29–1.38 (m)	1.30–1.40 (m)
CH_2(5)	1.25–1.33 (m)	1.29–1.38 (m)	1.30–1.40 (m)
CH_2(6)	1.25–1.33 (m)	2.07–2.11 (m)	2.06–2.09 (m)
CH_2(7) or H–C(7)	1.25–1.33 (m)	5.31–5.40 (m)	5.31–5.38 (m)
CH_2(8) or H–C(8)	2.05–2.10 (m)	5.31–5.40 (m)	5.31–5.38 (m)
H–C(9) or CH_2(9)	5.31–5.39 (m)	2.80–2.83 (m)	2.78 (dd, J = 6.2, 6.2)
H–C(10)	5.31–5.39 (m)	5.31–5.40 (m)	5.31–5.38 (m)
CH_2(11) or H–C(11)	2.80–2.83 (m)	5.31–5.40 (m)	5.31–5.38 (m)
H–C(12) or CH_2(12)	5.31–5.39 (m)	2.80–2.83 (m)	2.06–2.09 (m)
H–C(13) or CH_2(13)	5.31–5.39 (m)	5.31–5.40 (m)	1.30–1.40 (m)
CH_2(14) or H–C(14)	2.05–2.10 (m)	5.31–5.40 (m)	1.30–1.40 (m)
H–C(15) or CH_2(15)	5.31–5.39 (m)	2.07–2.11 (m)	1.30–1.40 (m)
H–C(16) or Me(16)	5.31–5.39 (m)	0.98 (t, J = 7.5)	0.91 (t, J = 6.6)
CH_2(17)	2.05–2.10 (m)	–	–
Me(18)	0.98 (t, J = 7.5)	–	–
H–C(1′)	5.34 (overlap)	5.34 (overlap)	5.34 (overlap)
H–C(2′)	3.42 (dd, J = 9.7, 3.6)	3.41 (dd, J = 9.8, 3.8)	3.41 (dd, J = 9.8, 3.8)
H–C(3′)	3.72 (overlap)	3.72 (overlap)	3.71 (overlap)
H–C(4′)	3.34 (dd, J = 9.5, 9.5)	3.32 (dd, J = 9.5, 9.5)	3.33 (dd, J = 9.5, 9.5)
H–C(5′)	3.83 (overlap)	3.83 (overlap)	3.83 (overlap)
CH_2(6′)	3.72 (overlap), 3.83 (overlap)	3.72 (overlap), 3.83 (overlap)	3.71 (overlap), 3.83 (overlap)
CH_2(1″)	3.62 (br. s)	3.62 (br. s)	3.62 (br. s)
H–C(3″)	4.10 (d, J = 8.2)	4.09 (d, J = 8.2)	4.09 (d, J = 8.2)
H–C(4″)	4.01 (t-like, J = 8.1)	4.00 (t-like, J = 8.1)	4.01 (t-like, J = 8.1)
H–C(5″)	3.92 (td, J = 7.8, 3.0)	3.92 (td, J = 7.8, 3.3)	3.92 (td, J = 7.8, 3.1)
CH_2(6″)	4.39 (dd, J = 11.5, 7.9), 4.33 (dd, J = 11.6, 3.0)	4.39 (dd, J = 11.7, 7.8), 4.32 (dd, J = 11.7, 3.3)	4.39 (dd, J = 11.7, 7.8), 4.32 (dd, J = 11.6, 3.1)

Compound **2**, a colorless oil, showed a molecular-ion peak at m/z 597 ($[M+Na]^+$) in the ESI-MS, corresponding to the molecular formula $C_{28}H_{46}O_{12}$. The ^{1}H- and ^{13}C-NMR spectra (*Tables 1* and *2*, resp.) revealed that compound **2** was quite similar to compound **1**. The most striking difference was the absence of two non-O-bearing CH_2 groups. Detailed analysis of the ^{1}H,^{1}H-COSY and HMBC spectra revealed the occurrence of a partial structure, –CH_2–(CH=CH–CH_2)$_3$–Me with the following chemical shifts: δ(C) 28.0 (t), 128.1 (d), 128.9 (d), 26.3 (t), 129.1 (d), 129.2 (d), 26.5 (t), 130.8 (d), 132.7 (d), 21.4 (t), and 14.6 (q). The two CH_2 groups absent in **2** were part of the CH_2 chain between the olefinic and ester groups in compound **1**, which suggested that compound **2** possessed a hexadeca-7,10,13-trienoic acid residue. In the ^{13}C-NMR spectrum, the three allylic CH_2 C-atoms (C(6), C(9), and C(12)) were shifted upfield to δ(C) 28.0, 26.3, and 26.5, which indicated that the geometry of C=C bonds is most probably (Z) [19–21]. The HMBCs of the signals of CH_2(6″) (δ(H) 4.39 and 4.32) with the one of C(1) (δ(C) 175.4 (s)) indicated that the hexadeca-7,10,13-trienoic acid residue was attached to HO–C(6″) of sucrose. Accordingly, the structure of compound

Table 2. *^{13}C-NMR Data of Compounds* **1** (100 MHz), **2** (100 MHz), *and* **3** (150 MHz). Recorded in CD_3OD; δ in ppm.

C-Atom	**1**	**2**	**3**	C-Atom	**1**	**2**	**3**
C(1)	175.5 (*s*)	175.4 (*s*)	175.6 (*s*)	C(16)	132.7 (*d*)	14.6 (*q*)	14.6 (*q*)
C(2)	34.9 (*t*)	34.8 (*t*)	35.0 (*t*)	C(17)	21.5 (*t*)		
C(3)	26.0 (*t*)	25.8 (*t*)	26.0 (*t*)	C(18)	14.7 (*q*)		
C(4)	30.2 (*t*)[a])	29.8 (*t*)	30.0 (*t*)	C(1′)	93.5 (*d*)	93.4 (*d*)	93.6 (*d*)
C(5)	30.2 (*t*)[a])	30.4 (*t*)	30.6 (*t*)[b])	C(2′)	73.3 (*d*)	73.2 (*d*)	73.3 (*d*)
C(6)	30.4 (*t*)[a])	28.0 (*t*)	28.1 (*t*)[c])	C(2′)	74.7 (*d*)	74.6 (*d*)	74.7 (*d*)
C(7)	30.7 (*t*)	128.1 (*d*)[d])	129.2 (*d*)[e])	C(4′)	71.5 (*d*)	71.4 (*d*)	71.6 (*d*)
C(8)	28.2 (*t*)	128.9 (*d*)[d])	129.4 (*d*)[e])	C(5′)	74.2 (*d*)	74.2 (*d*)	74.4 (*d*)
C(9)	128.3 (*d*)[f])	26.3 (*t*)[g])	26.7 (*t*)	C(6′)	62.5 (*t*)	62.4 (*t*)	62.6 (*t*)
C(10)	128.9 (*d*)[f])	129.1 (*d*)[d])	130.8 (*d*)[e])	C(1″)	63.8 (*t*)	63.7 (*t*)	63.8 (*t*)
C(11)	26.4 (*t*)[h])	129.2 (*d*)[d])	131.1 (*d*)[e])	C(2″)	105.5 (*s*)	105.4 (*s*)	105.6 (*s*)
C(12)	129.1 (*d*)[f])	26.5 (*t*)[g])	28.3 (*t*)[c])	C(3″)	78.9 (*d*)	78.8 (*d*)	78.9 (*d*)
C(13)	129.2 (*d*)[f])	130.8 (*d*)[d])	30.7 (*t*)[b])	C(4″)	76.9 (*d*)	76.8 (*d*)	77.0 (*d*)
C(14)	26.6 (*t*)[h])	132.7 (*d*)	32.8 (*t*)	C(5″)	80.6 (*d*)	80.6 (*d*)	80.8 (*d*)
C(15)	131.1 (*d*)[f])	21.4 (*t*)	23.8 (*t*)	C(6″)	67.0 (*t*)	66.9 (*t*)	67.1 (*t*)

[a])–[h]) Assignments may be interchanged.

2 was determined as 6-*O*-[(7*Z*,10*Z*,13*Z*)-hexadeca-7,10,13-trienoyl]-β-D-fructofuranosyl α-D-glucopyranoside.

Compound **3** was obtained as a colorless oil. The molecular formula was established as $C_{28}H_{48}O_{12}$ by the HR-ESI-MS (*m/z* 599.3036 ($[M + Na]^+$); calc. 599.3043), indicating five degrees of unsaturation. The ^{13}C-NMR spectrum (*Table 2*) indicated the presence of two C=C bonds (δ(C) 131.1 (*d*), 130.8 (*d*), 129.4 (*d*), 129.2 (*d*)), ten non-O-bearing CH_2 groups, as well as one Me group (δ(C) 14.6 (*q*, C(16)). Comparison of the spectroscopic data with those of **2** revealed an overall similarity, except for the absence of one C=C bond. The upfield shift of the signal of CH_2(15) (δ(H) 1.30–1.40 (overlap)) suggested that the C=C bond between C(13) and C(14) of compound **2** was saturated in compound **3**, which was confirmed by HMBC and ^{1}H,^{1}H-COSY correlations (*Fig.*). On the basis of the fact that signals of the allylic C-atoms were shifted upfield (δ(C) 28.1 and δ(C) 28.3), the geometry of C=C bonds in compound **3** was determined as (*Z*) [19–21]. The fatty acid residue was attached to HO–C(6″) of sucrose, as deduced by HMBCs of the signals of CH_2(6″) (δ(H) 4.39 and 4.32) with the

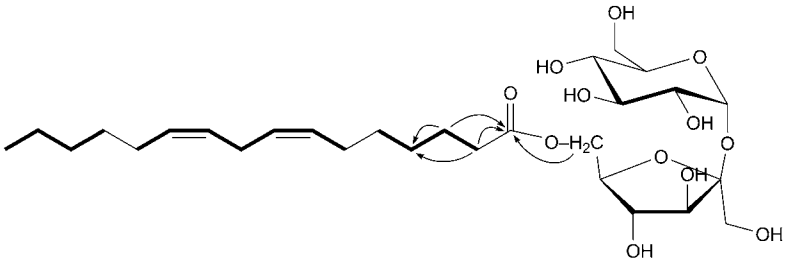

Figure. *Key ^{1}H,^{1}H-COSY correlations* (—) *and HMBCs* (H → C) *of* **3**

one of C(1) (δ(C) 175.6 (*s*)). Consequently, the chemical structure of compound **3** was determined to be 6-*O*-[(7*Z*,10*Z*)-hexadeca-7,10-dienoyl]-β-D-fructofuranosyl α-D-glucopyranoside.

The structures of the known compounds were identified by comparison of their spectroscopic data with those reported in the literature.

Compounds **1**–**12** were tested for cytotoxicity against HL-60, A-549, SMMC-7721, MCF-7, and SW480 cell lines *in vitro*. However, all of them were inactive.

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Experimental Part

General. Solvents were distilled before use. Column chromatography (CC): silica gel (SiO_2; 200–300 mesh, 10–40 μm; *Qingdao Marine Chemical Inc.*, Qingdao, P. R. China), *MCI* gel (75–150 μm; *Mitsubishi Chemical Corporation*, Japan), and *Sephadex LH–20* (*Amersham Pharmacia Biotech*, Sweden). Fractions were monitored by TLC, and spots were visualized by heating SiO_2 plates sprayed with 10% H_2SO_4 in EtOH. Semi-prep. HPLC: *Zorbax SB-C-18* column (i.d. 9.4 × 250 mm; *Agilent Co., Ltd.*). Prep. HPLC: *Shimadzu PRC-ODS (K)* column, *Shimadzu LC-8A* prep. liquid chromotography. Optical rotations: *JASCO P-1020*. UV Spectra: *Shimadzu UV-2401PC*. IR Spectra: *Tensor 27*; KBr pellets. 1D- and 2D-NMR spectra: *Bruker AM-400* and *Advance III 600* spectrometers with TMS as internal standard; unless specified, chemical shifts (δ) in ppm with reference to the solvent signals, *J* in Hz. MS: *API QSTAR Pulsar-1* mass spectrometer.

Plant Material. The aerial parts of *E. hiemale* were collected from the area of Changbaishan, Jilin Province, P. R. China, in July 2010. The sample was identified by *X. C.*, and a voucher specimen (KIB 100701) has been deposited with the State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences.

Extraction and Isolation. The air-dried and powdered aerial parts of the plant (5 kg) were extracted with EtOH 95% (3 × 15 l, 24 h each) at r.t. and filtered. The filtrate was evaporated to give a residue (420 g), which was suspended in H_2O and then extracted with AcOEt. The AcOEt extract (160 g) was decolorized over *MCI* gel (eluted with 90% MeOH) and then was subjected to CC (SiO_2; petroleum ether (PE)/Me_2CO, gradient system) to afford *Frs. 1–5*. Repeated crystallization from a mixture of Me_2CO/MeOH from *Fr. 1* gave compound **4** (5 g). *Fr. 2* was subjected to CC (SiO_2; PE/Me_2CO 99 : 1, and *Sephadex LH-20*; $CHCl_3$/MeOH, 1 : 1) to yield compound **5** (46 mg). *Fr. 3* was subjected to CC (SiO_2; PE/Me_2CO, increasing polarity) to provide *Frs. 3.1–3.8*. *Fr. 3.2* was purified by using semi-prep. HPLC (20% MeOH in H_2O) to yield compounds **10** (88 mg) and **11** (9 mg). After the purification of *Fr. 3.7* with *Sephadex LH-20* ($CHCl_3$/MeOH 1 : 1) and prep. HPLC (65% MeOH in H_2O) compound **12** (60 mg) was isolated. *Fr. 3.8* was subjected to prep. HPLC (35% MeOH in H_2O), compounds **8** (16 mg) and **9** (23 mg) were obtained. *Fr. 5* was subjected to CC (SiO_2; $CHCl_3$/MeOH 95 : 5 → 1 : 1) to provide *Frs. 5.1–5.10*. Repeated chromatography of *Fr. 5.3* with a gradient system of $CHCl_3$/MeOH 95 : 5 → 7 : 3 yielded compound **6** (25 mg). From *Fr. 5.7*, compound **7** (30 mg) was obtained after prep. HPLC (60% MeOH in H_2O). *Fr. 5.8* was first subjected to CC (SiO_2; $CHCl_3$/MeOH 93 : 7), then to prep. HPLC (78% MeOH in H_2O) to yield compounds **1** (60 mg), **2** (6 mg), and **3** (3 mg).

6-O-[(9Z,12Z,15Z)-Octadeca-9,12,15-trienoyl]-β-D-fructofuranosyl α-D-Glucopyranoside (**1**). Colorless oil. $[\alpha]_D^{25.8} = +35.9$ ($c = 0.50$, MeOH). UV (MeOH): 268 (3.35), 232 (3.60), 202 (3.69). IR (KBr): 3428, 2931, 1726, 1063. ^{1}H- and ^{13}C-NMR: see *Tables 1* and *2*, resp. ESI-MS (pos.): 625 ($[M + Na]^+$). HR-ESI-MS: 625.3186 ($[M + Na]^+$, $C_{30}H_{50}NaO_{12}^+$; calc. 625.3200).

6-O-[(7Z,10Z,13Z)-Hexadeca-7,10,13-trienoyl]-β-D-fructofuranosyl α-D-Glucopyranoside (**2**). Colorless oil. $[\alpha]_D^{25.7} = +30.7$ ($c = 0.30$, MeOH). UV (MeOH): 268 (3.33), 231 (3.42), 203 (3.96). IR (KBr):

3425, 2930, 1726, 1065. ^{1}H- and ^{13}C-NMR: see *Tables 1* and *2*, resp. ESI-MS (pos.): 597 ($[M+Na]^+$). HR-ESI-MS (pos.): 597.2872 ($[M+Na]^+$, $C_{28}H_{46}NaO_{12}^+$; calc. 597.2887).

*6-O-[(7Z,10Z)-Hexadeca-7,10-dienoyl]-β-*D*-fructofuranosyl α-*D*-Glucopyranoside* (**3**). Colorless oil. $[\alpha]_D^{25.8} = +44.6$ ($c = 0.20$, MeOH). UV (MeOH): 229 (3.43), 202 (3.97). IR (KBr): 3422, 2928, 1736, 1064. ^{1}H- and ^{13}C-NMR: see *Tables 1* and *2*, resp. ESI-MS (pos.): 599 ($[M+Na]^+$). HR-ESI-MS (pos.): 599.3036 ($[M+Na]^+$, $C_{28}H_{48}NaO_{12}^+$; calc. 599.3043).

Alkaline Hydrolysis of **1**. Compound **1** was treated with 0.5% NaOH (0.5 ml) in MeOH (3 ml) at r.t. for 18 h. The mixture was neutralized with 1N HCl and extracted with $CHCl_3$. The org. layer and the H_2O layer were concentrated under reduced pressure. From the org. layer of compound **1**, linolenic acid was identified with authentic samples and by TLC. Sucrose was obtained from the H_2O layer, and identified as sucrose by comparison with authentic samples and by TLC behavior, solvent: $CHCl_3$/MeOH/H_2O 45 : 30 : 1.

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