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Phytochemical Constituents of the Leaves of *Hosta longipes*†

Chung Sub Kim, Ki Hyun Kim, and Kang Ro Lee*

Natural Products Laboratory, School of Pharmacy, Sungkyunkwan University, Suwon 440-746, Korea

Abstract – Phytochemical investigation of the 80% MeOH extract from the leaves of *Hosta longipes* resulted in the isolation of sixteen compounds (**1** - **16**). The structures of the compounds were elucidated by spectroscopic methods to be methyl 10,10-dimethoxydecanoate (**1**), methyl 10-hydroxy-8*E*,12*Z*-octadecadienoate (**2**), methyl coriolate (**3**), *trans*-phytol (**4**), phytene-1,2-diol (**5**), phyton (**6**), (3*S*,5*R*,6*S*,7*E*,9*R*)-7-megastigmene-3,6,9-triol (**7**), (3*S*,5*R*,6*S*,9*R*)-3,6,9-trihydroxymegastigman-7-ene (**8**), shikimic acid (**9**), *p*-coumaramide (**10**), *trans*-*N*-*p*-coumaroyltyramine (**11**), *cis*-*N*-coumaroyltyramine (**12**), tryptophan (**13**), thymidine (**14**), adenosine (**15**), and deoxyadenosine (**16**). Compound **1** was synthesized, but not yet isolated from natural source, and compounds **2**-**16** were isolated for the first time from this plant source.
Keywords – *Hosta longipes*, Liliaceae, fatty acid, phenolic compound

Introduction

Hosta longipes (Fr. et Sav.) Matsumura (Liliaceae), widely distributed throughout Korea, China, and Japan, is an edible vegetable in Korea. It has long been used as a traditional Korean medicine for treating cough, sputum, laryngopharyngitis, burns, swelling, snake bites and inflammation.[1,2] Previous phytochemical investigations of this plant led to the isolation of steroidal saponins.[3,4] In the course of our continuing search for biologically active components from Korean medicinal plants, we investigated the constituents of the leaves of *H. longipes* and reported the isolation of steroidal saponins and flavonoids and their anti-inflammatory effects.[5,6] In our continuing study on this source, we further isolated sixteen compounds (**1** - **16**). Their structures were elucidated by physicochemical and spectroscopic methods. Compound **1** was isolated for the first time from nature and compounds **2** - **16** were isolated for the first time from this plant source.

Experimental

General experimental procedures – Optical rotations were measured on a Jasco P-1020 polarimeter in MeOH. IR spectra were recorded on a Bruker IFS-66/S FT-IR spectrometer. HRFABMS were obtained on a JEOL JMS700 mass spectrometer. NMR spectra were recorded on a Varian UNITY INOVA 500 NMR spectrometer operating at 500 MHz (^{1}H) and 125 MHz (^{13}C) with chemical shifts given in ppm (δ). Preparative HPLC was conducted using a Gilson 306 pump with Shodex refractive index detector and Apollo Silica 5 μ column (250 × 22 mm i.d.). Silica gel 60 (Merck, 70 - 230 mesh and 230 - 400 mesh) was used for column chromatography. The packing material for molecular sieve column chromatography was Sephadex LH-20 (Pharmacia Co.). TLC was performed using Merck precoated silica gel F_{254} plates. Spots were detected on TLC under UV light or by heating after spraying with 10% H_2SO_4 in C_2H_5OH (v/v).

Plant materials – Leaves of *H. longipes* were collected in Taebaek City, Korea, in June 2010. The plant was identified by one of the authors (K. R. Lee). A voucher specimen (SKKU-NPL 1103) of the plant has been deposited at the herbarium of the School of Pharmacy, Sungkyunkwan University, Suwon, Korea.

Extraction and isolation – Leaves of *H. longipes* (2.5 kg) were extracted with 80% MeOH at room temperature and filtered. The filtrate was evaporated under reduced pressure to give a MeOH extract (190 g), which was suspended in water (800 mL) and solvent-partitioned to give *n*-hexane (3 g), $CHCl_3$ (14 g), EtOAc (3 g), and *n*-BuOH (24 g) layers. The *n*-hexane (3 g) layer was separated over a silica gel column (*n*-hexane: EtOAc = 7 : 1 – 1 : 1) to yield nine fractions (H1 – H9). Fraction H2 (370 mg) was chromatographed on an RP-C_{18} silica

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*Author for correspondence
Kang Ro Lee, Natural Products Laboratory, School of Pharmacy, Sungkyunkwan University, Suwon 440-746, Korea
Tel: +82-31-290-7710; E-mail: krlee@skku.edu

gel column (90% MeOH) to give three subfractions (H21 – H23). Subfraction H21 (100 mg) was purified over a silica gel semi-prep. HPLC (hexane : $CHCl_3$: EtOAc = 9 : 2 : 1) to afford compounds **2** (10 mg, *Rt* = 13.1 min) and **3** (3 mg, *Rt* = 16.0 min). Subfraction H22 (20 mg) was purified by an RP-C_{18} semi-prep. HPLC (95% MeCN) to afford compound **4** (8 mg, *Rt* = 18.7 min). Fraction H7 (150 mg) was purified with an RP-C_{18} silica Lobar A®-column (80% MeOH) and silica gel semi-prep. HPLC (hexane : EtOAc = 3 : 1) to afford compound **5** (6 mg, *Rt* = 11.0 min). The $CHCl_3$ (14 g) layer was separated over a silica gel column (*n*-hexane : EtOAc = 7 : 1 – 1 : 1) to yield seven fractions (C1 – C7). Fraction C1 (1.0 g) was separated over an RP-C_{18} silica gel column (90% MeOH) and purified by a silica gel semi-prep. HPLC (hexane : EtOAc = 2 : 1) to afford compound **1** (11 mg, *Rt* = 13.4 min). Fraction C2 (200 mg) was purified with an RP-C_{18} silica Lobar A®-column (90% MeOH) and silica gel semi-prep. HPLC (hexane : EtOAc = 7 : 1) to afford compound **6** (4 mg, *Rt* = 17.0 min). The EtOAc (3 g) layer was chromatographed over a Sephadex LH-20 column (90% MeOH) to yield nine fractions (E1 – E9). Fraction E2 (800 mg) separated over a silica gel column ($CHCl_3$: MeOH = 12 : 1) and further purified with RP-C_{18} semi-prep. HPLC (50% MeOH) to afford compounds **7** (2 mg, *Rt* = 11.8 min) and **8** (3 mg, *Rt* = 12.9 min). Fraction E3 (700 mg) was separated over an RP-C_{18} silica gel column (90% MeOH) to give six subfractions (E31 – E36). Subfraction E31 (200 mg) was purified with an RP-C_{18} silica Lobar A®-column (80% MeOH) and silica gel semi-prep. HPLC ($CHCl_3$: MeOH = 2 : 1) to afford compounds **9** (7 mg, *Rt* = 16.7 min), **14** (6 mg, *Rt* = 18.7 min), **15** (3 mg, *Rt* = 20.3 min), and **16** (3 mg *Rt* = 25.3 min). Subfraction E33 (80 mg) was purified by an RP-C_{18} semi-prep. HPLC (30% MeCN) to afford compounds **11** (3 mg, *Rt* = 11.8 min) and **12** (3 mg, *Rt* = 10.4 min). Compound **10** (3 mg) was obtained by purification of subfraction E4 (210 mg) using a silica gel semi-prep. HPLC ($CHCl_3$: MeOH : H_2O = 9 : 2 : 0.2). Fraction E9 (100 mg) was purified with a silica gel semi-prep. HPLC ($CHCl_3$: MeOH : H_2O = 2 : 1 : 0.2) to yield compound **13** (7 mg, *Rt* = 15.0 min).

Methyl 10,10-dimethoxydecanoate (1) – Colorless gum. IR (KBr) ν_{max} cm^{-1}: 2951 (C-H), 1723 (C = O), 1284, 1032; ^1H and ^{13}C NMR: see Table 1; HRFABMS *m/z* 269.1733 $[M + Na]^+$; (calcd for $C_{13}H_{26}O_4$ Na, 269.1729).

Methyl 10-hydroxy-8*E*,12*Z*-octadecadienoate (2) – Colorless gum. $[\alpha]_D^{25}$: –3.0 (*c* 0.25, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz): δ 5.67 (1H, dt, *J* = 15.5, 6.5 Hz, H-12), 5.54 (1H, m, H-9), 5.48 (1H, m, H-13), 5.37 (1H, m, H-8), 4.08 (1H, m, H-10), 3.67 (3H, s, OCH_3), 2.30 (2H, t, *J* = 7.5 Hz, H-2), 2.25 (2H, m, H-11), 2.04 (4H, m, H-7 and 14), 1.62 (2H, m, H-17), 1.31 - 1.40 (12H, m, H-3 to H-6, H-15, H-16), 0.90 (3H, t, *J* = 7.0 Hz, H-18); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 174.5 (C-1), 133.4 (C-8), 132.5 (C-9), 132.4 (C-13), 125.0 (C-12), 72.7 (C-10), 51.6 (OCH_3), 35.7 (C-11), 34.3 (C-7), 32.1 (C-2), 31.5 (C-16), 29.8 (C-4), 29.3 (C-5, C-6, C-15), 27.6 (C-14), 25.2 (C-3), 22.4 (C-17), 14.1 (C-18). FABMS *m/z*: 311.3 $[M + H]^+$.

Table 1. ^1H (500 MHz) and ^{13}C NMR (125 MHz) data of **1** in $CDCl_3$

Position	**1**	
	δ_H (*J* in Hz)	δ_C[a]
1		174.5
2	2.29, t (7.5)	34.3
3	1.59, m	24.7
4	1.30 – 1.34, m	29.4
5	1.30 – 1.34, m	29.1
6	1.30 – 1.34, m	29.2
7	1.30 – 1.34, m	29.5
8	1.30 – 1.34, m	25.1
9	1.61, m	32.7
10	4.34, t (5.5)	104.8
1-OCH_3	3.66, s	51.6
10-OCH_3	3.30, s	52.8

[a]The assignments were based on HMQC and HMBC experiments.

Methyl coriolate (3) – Colorless gum. $[\alpha]_D^{25}$: +10.2 (*c* 0.30, $CHCl_3$); ^1H NMR ($CDCl_3$, 500 MHz): δ 6.49 (1H, dd, *J* = 15.0, 10.5 Hz, H-11), 5.97 (1H, t, *J* = 10.5 Hz, H-10), 5.66 (1H, dd, *J* = 15.0, 7.0 Hz, H-12), 5.45 (1H, dt, *J* = 10.5, 8.0 Hz, H-9), 4.16 (1H, q, *J* = 7.0, H-13), 3.67 (3H, s, H-OCH_3) 2.30 (2H, t, *J* = 7.5 Hz, H-2), 2.18 (2H, m, H-8), 1.26 - 1.62 (18H, m, H-3 to H-7, H-14 to H-17), 0.89 (3H, t, *J* = 7.5 Hz, H-18). FABMS *m/z*: 311.3 $[M + H]^+$.

***trans*-Phytol (4)** – Coloress oil. ^1H NMR (500 MHz, CD_3OD): δ 5.35 (1H, t, *J* = 7.0 Hz, H-2), 4.07 (2H, d, *J* = 6.5 Hz, H-1), 2.00 (2H, t, *J* = 6.5 Hz, H-4), 1.65 (3H, s, H-20), 1.52 - 1.05 (19H, m), 0.87 (9H, d, *J* = 6.5 Hz, H-16, 18, 19), 0.88 (3H, d, *J* = 6.5 Hz, H-17); ^{13}C NMR (125 MHz, CD_3OD): δ 138.4 (C-3), 123.6 (C-2), 58.2 (C-1), 39.8 (C-4), 39.4 (C-14), 37.4 (C-8), 37.3 (C-10), 37.2 (C-12), 36.6 (C-6), 32.8 (C-7), 32.7 (C-11), 27.9 (C-15), 25.1 (C-5), 24.7 (C-13), 24.3 (C-9), 22.0 (C-17), 21.9 (C-16), 19.1 (C-19), 19.0 (C-18), 15.0 (C-20). FABMS *m/z*: 319.3 $[M + Na]^+$

Phytene-1,2-diol (5) – Coloress gum. ^1H NMR (500

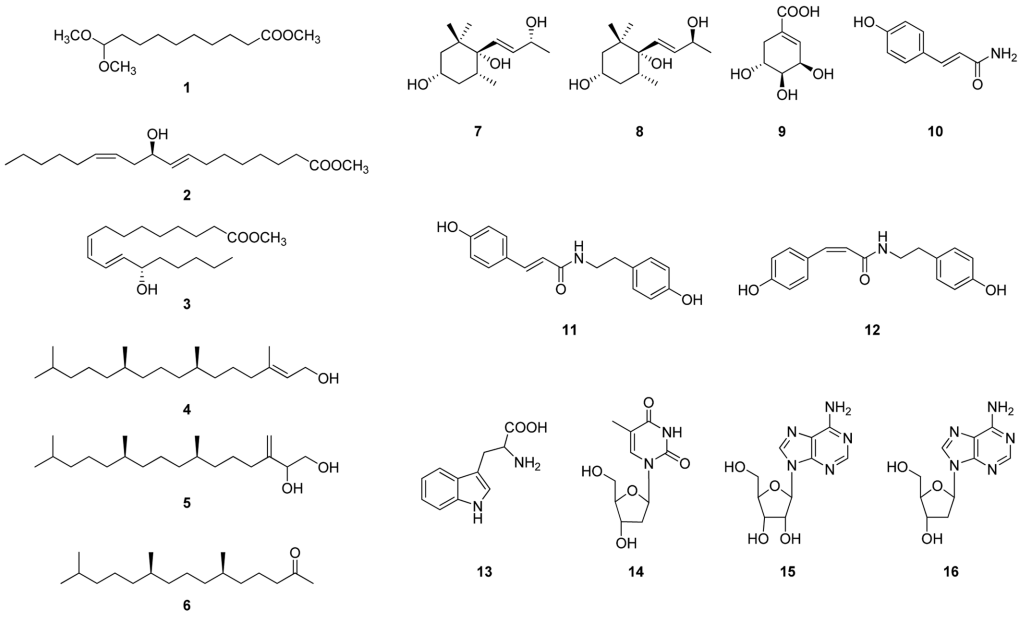

Fig. 1. The structures of **1** - **16** isolated from *H. longipes*.

MHz, CD_3OD): δ 5.07 (1H, s, H-20a), 4.89 (1H, s, H-20b), 4.07 (1H, dd, J = 7.0, 3.5 Hz, H-2), 3.58 (1H, dd, J = 6.5, 4.0 Hz, H-1a), 3.44 (1H, dd, J = 6.5, 2.5 Hz, H-1b), 2.09 - 1.97 (2H, m, H-4), 1.57 - 1.06 (19H, m), 0.88 (9H, d, J = 7.0 Hz, H-16, 18, 20), 0.86 (3H, d, J = 7.0 Hz, H-19); ^{13}C NMR (125 MHz, CD_3OD): δ 149.6 (C-3), 109.6 (C-17), 75.4 (C-2), 65.3 (C-1), 39.3 (C-14), 37.3 (C-8), 37.2 (C-10, 12), 36.8 (C-6), 32.7 (C-4), 32.5 (C-7, 11), 27.9 (C-15), 25.5 (C-5), 24.6 (C-13), 24.3 (C-9), 21.9 (C-20), 21.8 (C-16), 19.0 (C-19), 18.9 (C-18). EIMS *m/z*: 312.3 $[M]^+$.

Phyton (6) – Colorless gum. $[\alpha]_D^{25}$: +2.1 (*c* 0.15, $CHCl_3$); ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.40 (2H, t, J = 7.5 Hz, H-3), 2.12 (3H, s, H-1), 1.03 - 1.58 (17H, m, H-4 to H-14), 0.85 (12H, d, J = 7.0 Hz, H-15 to H-18); ^{13}C NMR (125 MHz, $CDCl_3$): 209.5 (C-2), 44.4 (C-3), 39.6 (C-13), 37.6, 37.5, 37.5 and 36.7 (C-5, C-7, C-9, C-11) 33.0 and 32.9 (C-6, C-10), 30.0 (C-1), 28.2 (C-4), 25.0 (C-14), 24.6 (C-12), 22.9 (C-8), 22.8 and 21.7 (C-15, C-18), 20.0 and 19.8 (C-16, C-17). FABMS *m/z*: 269.3 $[M + H]^+$.

(3*S*,5*R*,6*S*,7*E*,9*R*)-7-Megastigmene-3,6,9-triol (7) – Amorphous powder. $[\alpha]_D^{25}$: −11.9 (*c* 0.15, CH_3OH); ^{1}H NMR (500 MHz, CD_3OD): δ 5.72 (1H, dd, J = 16.0, 6.0 Hz, H-8), 5.55 (1H, dd, J = 16.0, 1.0 Hz, H-7), 4.29 (1H, m, H-9), 3.80 (1H, m, H-3), 1.93 (1H, m, H-5), 1.67 (1H, m, H-4a), 1.66 (1H, m, H-2a), 1.40 (1H, m, H-2b), 1.39 (1H, m, H-4b), 1.24 (3H, d, J = 6.0 Hz, H-10), 0.98 (3H, s, H-11),0.89 (3H, s, H-12), 0.81 (3H, d, J = 6.0 Hz, H-13); ^{13}C NMR (125 MHz, CD_3OD): δ 135.7 (C-8), 134.0 (C-7), 78.3 (C-6), 69.4 (C-9), 67.6 (C-3), 46.1 (C-2), 40.6 (C-1), 40.1 (C-4), 35.6 (C-5), 25.9 (C-11), 25.3 (C-12), 24.3 (C-10), 16.6 (C-13). FABMS *m/z*: 229.2 $[M + H]^+$.

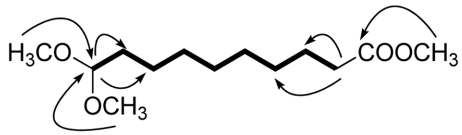

Fig. 2. Key HMBC (→) and ^{1}H-^{1}H COSY (▬) correlations of **1**.

(3*S*,5*R*,6*S*,9*R*)-3,6,9-Trihydroxymegastigman-7-ene (8) – Amorphous powder. $[\alpha]_D^{25}$: −15.9 (*c* 0.20, CH_3OH). ^{1}H-NMR (500 MHz, CD_3OD): 5.73 (1H, dd, J = 16.0, 6.0 Hz, H-8), 5.55 (1H, dd, J = 16.0, 1.0 Hz, H-7), 4.29 (1H, m, H-9), 3.80 (1H, m, H-3), 1.94 (1H, m, H-5), 1.68 (1H, m, H-4a), 1.66 (1H, t, J = 12.0 Hz, H-2a), 1.40 (1H, ddd, J = 12.0, 4.0, 2.0 Hz, H-2b), 1.39 (1H, q, J = 12.0 Hz, H-4b), 1.24 (3H, d, J = 6.0 Hz, H-10), 0.96 (3H, s, H-11), 0.86 (3H, s, H-12), 0.84 (3H, d, J = 7.0 Hz, H-13); ^{13}C-NMR (125 MHz, CD_3OD): δ 135.6 (C-8), 133.9 (C-7), 78.1 (C-6), 69.3 (C-9), 67.5 (C-3), 40.5 (C-1), 40.0 (C-4), 35.5 (C-5), 25.9 (C-12), 25.2 (C-11), 24.2 (C-10), 16.5 (C-13). FABMS *m/z*: 229.2 $[M + H]^+$.

Shikimic acid (9) – Amorphous powder. $[\alpha]_D^{25}$: −12.1 (*c* 0.20, CH_3OH). ^{1}H-NMR (500 MHz, CD_3OD): δ 6.48 (1H, m, H-6), 4.28 (1H, t, J = 4.0 Hz, H-5), 3.90 (1H, m, H-4), 3.53 (1H, m, H-3), 2.81 (1H, dd, J = 18.0, 5.5 Hz, H-2a), 2.16 (1H, dd, J = 18.0, 4.0 Hz, H-2b); ^{13}C-NMR (125 MHz, CD_3OD): δ 174.2 (C-7), 136.7 (C-1), 130.4

(C-6), 73.2 (C-5), 67.4 (C-4), 66.8 (C-3), 33.4 (C-2). FABMS *m/z*: 175.1 $[M + H]^+$.

***p*-Coumaramide (10)** – Amorphous powder. ^{1}H-NMR (500 MHz, CD_3OD): δ 7.35 (2H, d, *J* = 8.0 Hz, H-2, H-6), 7.32 (1H, d, *J* = 16.0 Hz, H-7), 6.75 (2H, d, *J* = 8.0 Hz, H-3, H-5), 6.33 (1H, d, *J* = 16.0 Hz, H-8). FABMS *m/z*: 164.1 $[M + H]^+$.

***trans*-*N*-*p*-Coumaroyltyramine (11)** – Amorphous powder. ^{1}H-NMR (500 MHz, CD_3OD): δ 7.33 (1H, d, *J* = 15.5 Hz, H-7), 7.02 (1H, d, *J* = 1.5 Hz, H-2), 7.00 (2H, d, *J* = 8.5 Hz, H-2', H-6'), 6.95 (1H, dd, *J* = 8.5, 1.5 Hz, H-6), 6.69 (1H, d, *J* = 8.5 Hz, H-5), 6.62 (2H, d, *J* = 8.5 Hz, H-3', H-5'), 6.30 (1H, d, *J* = 15.5 Hz, H-8), 3.38 (2H, t, *J* = 7.5 Hz, H-8), 2.66 (2H, t, *J* = 7.5 Hz, H-7); ^{13}C-NMR (125 MHz, CD_3OD): δ 167.2 (C-9), 155.6 (C-4'), 147.5 (C-4), 145.5 (C-3), 141.0 (C-7), 131.0 (C-1), 130.7 (C-2', C-6'), 127.1 (C-1), 121.0 (C-6), 117.3 (C-8), 116.3 (C-5), 116.0 (C-3', C-5'), 114.1 (C-2), 42.0 (C-8'), 34.0 (C-7'). FABMS *m/z*: 284.2 $[M + H]^+$.

***cis*-*N*-Coumaroyltyramine (12)** – Amorphous powder. ^{1}H-NMR (500 MHz, CD_3OD): δ 7.26 (2H, d, *J* = 8.5 Hz, H-2', H-6'), 6.91 (2H, d, *J* = 8.5 Hz, H-2, H-6), 6.65 (2H, d, *J* = 8.5 Hz, H-3', H-5'), 6.62 (2H, d, *J* = 8.5 Hz, H-3, H-5), 6.51 (1H, d, *J* = 12.5 Hz, H-8), 5.69 (1H, d, *J* = 12.5 Hz, H-7), 3.29 (2H, t, *J* = 7.5 Hz, H-8'), 2.59 (2H, t, *J* = 7.5 Hz, H-7'); ^{13}C-NMR (125 MHz, CD_3OD): δ 170.4 (C-9), 159.4 (C-4), 156.9 (C-4'), 138.1 (C-7), 132.3 (C-2', C-6'), 131.2 (C-1'), 130.7 (C-2, C-6), 127.9 (C-1), 121.4 (C-8), 116.2 (C-3, C-5), 116.0 (C-3', C-5'), 42.3 (C-8'), 35.5 (C-7'). FABMS *m/z*: 284.2 $[M + H]^+$.

Tryptophan (13) – Amorphous powder. ^{1}H-NMR (500 MHz, DMSO-d_6): δ 10.95 (1H, s, H-NH), 7.55 (1H, d, *J* = 7.5 Hz, H-4), 7.34 (1H, d, *J* = 7.5 Hz, H-7), 7.22 (1H, s, H-2), 7.05 (1H, t, *J* = 7.5 Hz, H-5), 6.96 (1H, t, *J* = 7.5 Hz, H-6), 3.53 (1H, m, H-12), 3.33 (1H, m, H-10a), 3.03 (1H, m, H-10b); ^{13}C-NMR (125 MHz, DMSO-d_6): δ 171.0 (C-12), 137.1 (C-8), 127.9 (C-9), 124.8 (C-2), 121.6 (C-4), 119.1 (C-5), 118.9 (C-6), 112.0 (C-7), 110.3 (C-3), 55.5 (C-11), 27.9 (C-10).

Thymidine (14) – Amorphous powder. ^{1}H-NMR (500 MHz, CD_3OD): δ 7.81 (1H, s, H-6), 6.28 (1H, t, *J* = 7.0 Hz, H-1'), 4.40 (1H, m, H-3'), 3.90 (1H, dd, *J* = 6.5, 3.5 Hz, H-4'), 3.79 (1H, dd, *J* = 12.0, 3.5 Hz, H-5'a), 3.72 (1H, dd, *J* = 12.0, 3.5 Hz, H-5'b), 2.20 (2H, m, H-2'), 1.88 (3H, s, H-7).

Adenosine (15) – Amorphous powder. ^{1}H-NMR (500 MHz, CD_3OD): δ 8.30 (1H, s, H-2), 8.18 (1H, s, H-8), 5.96 (1H, d, *J* = 6.0 Hz, H-1'), 4.74 (1H, t, *J* = 6.0 Hz, H-2'), 4.33 (1H, dd, *J* = 6.0, 2.0 Hz, H-3'), 4.17 (1H, m, H-4'), 3.88 (1H, dd, *J* = 12.5, 2.5 Hz, H-5'a), 3.74 (1H, dd, *J* = 12.5, 3.0 Hz, H-5'b)

Deoxyadenosine (16) – Amorphous powder. ^{1}H-NMR (500 MHz, CD_3OD): δ 8.32 (1H, s, H-2), 8.18 (1H, s, H-8), 6.43 (1H, d, *J* = 7.0 Hz, H-1'), 4.58 (1H, m, H-3'), 4.07 (1H, m, H-4'), 3.84 (1H, dd, *J* = 12.5, 2.5 Hz, H-5'a), 3.74 (1H, dd, *J* = 12.5, 3.0 Hz, H-5'b), 2.80 (1H, m, H-2'a), 2.41 (1H, m, H-2'b)

Result and Discussion

Column chromatographic separation of the 80% MeOH extract from the leaves of *H. longipes* led to the isolation of known compounds **2** - **16**, which were identified as methyl 10-hydroxy-8*E*,12*Z*-octadecadienoate (**2**),[7] methyl coriolate (**3**),[8] *trans*-phytol (**4**),[9] phytene-1,2-diol (**5**),[10] phyton (**6**),[11] (3*S*,5*R*,6*S*,7*E*,9*R*)-7-megastigmene-3,6,9-triol (**7**),[12] (3*S*,5*R*,6*S*,9*R*)-3,6,9-trihydroxymegastigman-7-ene (**8**),[13] shikimic acid (**9**),[14] *p*-coumaramide (**10**),[15] *trans*-*N*-*p*-coumaroyltyramine (**11**),[16] *cis*-*N*-coumaroyltyramine (**12**),[16] tryptophan (**13**),[17] thymidine (**14**),[17] adenosine (**15**),[17] and deoxyadenosine (**16**)[17] by comparing the spectroscopic data. All compounds were isolated for the first time from this plant source. The following describes the structure elucidation of compound **1**, which was synthesized[18] but was not yet isolated from natural source. 269.1733 $[M + Na]^+$; (calcd for $C_{13}H_{26}O_4$ Na, 269.1729).

Compound **1** was obtained as a colorless oil and had a molecular formula of $C_{13}H_{26}O_4$, as determined from the ion peak $[M + Na]^+$ at *m/z* 269.1733 $[M + Na]^+$; (calcd for $C_{13}H_{26}O_4$ Na, 269.1729) in positive ion HRFABMS. The IR spectrum indicated that **1** possessed C-H bond (2951 cm^{-1}) and carbonyl (1723 cm^{-1}) groups. The ^{1}H NMR spectrum showed an oxygenated methine [δ_H 4.34 (1H, t, *J* = 5.5 Hz, H-10)], three methoxy groups [δ_H 3.66 (3H, s, 1-OCH_3), 3.30 (6H, s, 10-OCH_3)], a methylene adjacent to carbonyl group [δ_H 2.29 (2H, t, *J* = 7.5 Hz, H-2)], and seven methylenes [δ_H 1.61 (2H, m, H-9), 1.59 (2H, m, H-3), 1.30-1.34 (10H, m, H-4 to H-8)]. The ^{13}C NMR spectrum contained 13 signals, including a carboxylic carbon [δ_C 174.5 (C-1)], an acetal carbon [δ_C 104.8 (C-10)], three methoxy carbons [δ_C 52.8 (×2) (10-OCH_3), 51.6 (1-OCH_3)], and eight methylene carbons [δ_C 34.3 (C-2), 32.7 (C-9), 29.5 (C-7), 29.4 (C-4), 29.2 (C-6), 29.1 (C-5), 25.1 (C-8), and 24.7 (C-3)]. This spectroscopic data were very similar to those of methyl 8,8-dimethoxyoctanoate[19] except that the presence of additional two methylene groups [δ_H 1.30-1.34; δ_C 29.2, 29.5]. The HMBC cross-peaks of 1-OCH_3/C-1 and 10-OCH_3/C-10 confirmed the location of three methoxy groups. Analyses of ^{1}H-^{1}H COSY, HMQC and HMBC spectra corroborated

the gross structure of **1**, which was elucidated as methyl 10,10-dimethoxydecanoate. Compound **1** was previously reported as a synthetic[18] without NMR assignment. We isolated compound **1** from natural source and performed full NMR assignment of **1** for the first time. But we suggest that compound **1** could be an artifact because MeOH was used as solvent during purification.

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