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# A New Homosesquiterpenoid of *Cinnamomum Macrostemon*

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## ABSTRACT

*A new homosesquiterpenoid, cinnamastemol (**1**) was isolated from the roots of Cinnamomum macrostemon (Lauraceae). The structure of the new homosesquiterpenoid was elucidated by chemical and physical evidence.*

## Keywords

*Cinnamomum macrostemon*, *Lauraceae*, Homosesquiterpenoid.

## Introduction

*Cinnamomum macrostemon* Hayata is a medium-sized evergreen tree, and it is endemic in Taiwan, distributed at medium altitudes throughout the island. In the course of screening for biologically and chemically novel agents from Formosan Lauraceous plants, *C. macrostemon* Hayata was chosen for further phytochemical investigation. Previously, we isolated 12 compounds, including three coumarins, two benzenoids, two steroids, two lignan and three dibenzocycloheptenes from the roots of this plant. In the course of screening for biologically and chemically novel agents from Formosan plants in the family Lauraceae [1-79], *C. macrostemon* was chosen for further phytochemical investigation. In this paper, we report the isolation and structural elucidation of this new homosesquiterpenoid.

Cinnamastemol (**1**) was obtained as a colorless oil. Its molecular formula was established as $C_{16}H_{28}O$ by HRESIMS (*m/z* 259.2035 $[M + Na]^+$; calc. 259.2038). The IR spectrum revealed the presence of hydroxyl group absorption at 3300 $cm^{-1}$. The $^1H$ NMR spectrum of **1** showed four methyl groups at δ 0.77 (3H, d, J = 7.2), 0.92 (3H, d, J = 7.2), 1.11 (3H, s) and 1.67 (3H, br s), five methine protons at δ 1.03, 1.23, 1.75, 2.16 and 5.50, five methylene protons at δ 1.14/1.64, 1.28/1.99, 1.28/2.02, 1.29 and 1.44/1.81, indicating that **1** was probably a sesquiterpene possessing a hydroxyl group in the structure. The $^{13}C$ NMR spectrum and a DEPT experiments indicated that compound **1** had a total of 16 carbons, with the skeleton consisting of 16 carbons, consistent with a homosesquiterpenoid. The carbons of the homosesquiterpenoid were assigned, from $^{13}C$ NMR and DEPT experiments, as four methyls at δ 15.1, 20.8, 21.5 and 23.8; five methylenes at δ 21.9, 22.6, 29.7, 30.9 and 42.2; five methines at δ 26.0, 39.8, 46.7, 50.0 and 122.3 and two quaternary carbons at δ 72.4 and 135.0. The structure of **1** was also confirmed by 2D NMR experiments. Examination of the $^1H$-$^1H$ COSY and $^1H$-$^{13}C$ COSY spectra provided one continuous fragment as shown

by bold lines in Figure 1. The HETCOR experiment showed that the carbon signals at δ 22.6 for C-1, 30.9 for C-2, 122.3 for C-4, 39.8 for C-4a, 46.7 for C-5, 21.9 for C-6, 29.7 for C-7, 42.2 for C-8, 50.0 for C-9a and 26.0 for C-11 were correlated to the proton signals at δ 1.28/2.02 for H-1, 1.28/1.99 for H-2, 5.50 for H-4, 1.75 for H-4a, 1.03 for H-5, 1.14/1.64 for H-6, 1.29 for H-7, 1.44/1.81 for H-8, 1.23 for H-9a and 2.16 for H-11, respectively. The relative stereochemistry of **1** was determined through 2D NOESY analysis (Figure 2). Thus, the structure of this compound was determined to be a new homosesquiterpenoid, which was further confirmed by HMBC experiments (Table 1). The structure of **1** was determined to be a new homosesquiterpenoid and has been named cinnamastemol (**1**).

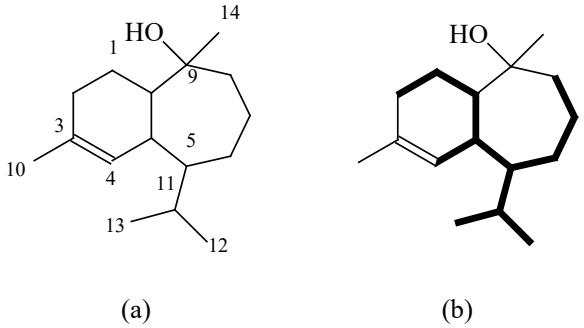


**Figure 1:** Chemical structure (a) and COSY (b) correlations of 1.

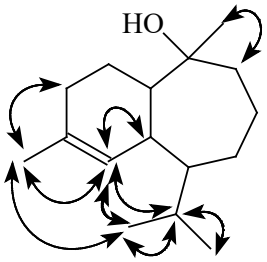


**Figure 2**: The NOESY correlations of 1.

**Table 1:** NMR data of **1** in $CDCl_3$ (δ in ppm, J in Hz, 400 MHz for $^1H$ NMR, and 100 MHz for $^{13}C$ NMR).

| Position | $\delta_C$ | $\delta_H$ | *mult., J (Hz)* | *HMBC ($^1H \rightarrow {}^{13}C$)* |
|---|---|---|---|---|
| 1 | 22.6 | 2.02<br>1.28 | m<br>m | C-2, C-9a<br>C-2, C-9a |
| 2 | 30.9 | 1.99<br>1.28 | m<br>m | C-1, C-3<br>C-1, C-3 |
| 3 | 135.0 | – | – | – |
| 4 | 122.3 | 5.50 | br s | C-3, C-4a |
| 4a | 39.8 | 1.75 | m | C-4, C-5, C-9a |
| 5 | 46.7 | 1.03 | m | C-4a, C-6, C-11 |
| 6 | 21.9 | 1.64<br>1.14 | m<br>m | C-5, C-7<br>C-5, C-7 |
| 7 | 29.7 | 1.29 | m | C-6, C-8 |
| 8 | 42.2 | 1.81<br>1.44 | dt, 12.0, 3.2<br>m | C-7, C-9, C-9a, C-14<br>C-7, C-9, C-9a, C-14 |
| 9 | 72.4 | – | – | – |
| 9a | 50.0 | 1.23 | m | C-1, C-4a, C-9 |
| 10 | 23.8 | 1.67 | br s | C-2, C-3, C-4 |
| 11 | 26.0 | 2.16 | m | C-5, C-12, C-13 |
| 12 | 21.5 | 0.92 | d, 7.2 | C-5, C-11, C-13 |
| 13 | 15.1 | 0.77 | d, 7.2 | C-5, C-11, C-12 |
| 14 | 20.8 | 1.11 | s | C-8, C-9, C-9a |

## Experimental

### General

UV spectra were obtained in MeCN, IR spectra were measured on a Hitachi 260-30 spectrophotometer. $^1H$ NMR (500 MHz, $CDCl_3$) and NOESY spectra were obtained on a Varian (Unity Plus) NMR spectrometer. Low-resolution ESI-MS spectra were obtained on an API 3000 (Applied Biosystems) and high-resolution ESI-MS spectra on a Bruker Daltonics APEX II 30e spectrometer. Silica gel 60 (Merck, 70~230 mesh, 230~400 mesh) was used for column chromatography. Precoated Silica gel plates (Merck, Kieselgel 60 F-254), 0.20 mm and 0.50 mm, were used for analytical TLC and preparative TLC, respectively, and visualized with 50% $H_2SO_4$.

### Plant Material

The roots of *Cinnamomum macrostemon* Hayata were collected from Pinglin Hsiang, Taipei County, Taiwan, and November 2009. Dr. Fu-Yuan Lu (Department of Forestry and Natural Resources College of Agriculture, National Chiayi University) identified plant material. A voucher specimen (Cinnamo. 9) was deposited in the School of Medical and Health Sciences, Fooyin University, Kaohsiung, Taiwan.

### Extraction and Isolation

The air-dried roots of *C. macrostemon* (2.3kg) were extracted with MeOH (10 L×5) at room temperature and a MeOH extract (90.3g) was obtained upon concentration under reduced pressure. The residue was placed on a silica gel column and eluted with $CHCl_3$ gradually enriched with MeOH to afford 4 fractions. Part of fraction 4 (15.2g) was subjected to silica gel CC, eluting with $CH_2Cl_2$–MeOH (10:1) and enriched gradually with MeOH, to obtain 5 fractions (4-1-4-5). Fraction 4-1 (2.8g) was further separated by silica gel CC using the same solvent system and purified by preparative TLC ($CH_2Cl_2$–MeOH, 90:1) to cinnamastemol (**1**) (4mg).

**Cinnamastemol (1):** Colorless oil. UV $\lambda_{max}$ (MeCN, log ε) 193 (3.11), 208 (2.56) nm. IR (neat) $\nu_{max}$ 3300 (br, OH), 1550, 1015 $cm^{-1}$; ESI-MS *m/z* 259 $[M+Na]^+$; HR-ESI-MS *m/z* 259.2035 $[M+Na]^+$ (calcd for $C_{16}H_{28}ONa$, 259.2038); $^1H$ NMR: see Table 1; $^{13}C$ NMR: see Table 1.


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