

Short Note

(S*)-2,7,8-Trihydrochroman-4-one

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Abstract: Reticumanone (**1**), a new chromanone, isolated from the leaves of *Cinnamomum reticulatum* Hay (Lauraceae), has been characterized as (S*)-2,7,8-trihydrochroman-4-one, by means of spectroscopic methods.

Keywords: *Cinnamomum reticulatum* Hay; Lauraceae; reticumanone; chromanone

1. Introduction

The *Cinnamomum* species (Lauraceae) have been used in folk medicine for sweating, antipyretic, and analgesic effect [1]. There is only one paper describing the constituents of *Cinnamomum reticulatum* Hay [1]. In the course of screening for biologically and chemically novel agents from Formosan Lauraceous plants [2–17], *C. reticulatum* Hay was chosen for further phytochemical investigation. In this paper, we report the isolation and structural elucidation of this new chromanone compound (**1**).

2. Results and Discussion

Reticumanone (**1**), obtained as a white amorphous powder (CHCl₃), was assigned the molecular formula C₉H₈O₅ by HR-EIMS at *m/z* [M]⁺ 196.0374 (calcd for C₉H₈O₅, 196.0372). UV λ_{max} at 273, 310 (sh), 322 nm, IR bands at 1660 cm⁻¹ and a signal appearing at δ 182.4 in the ¹³C-NMR spectrum indicate the chromanone skeleton of this compound [18]. The IR spectrum revealed the presence of a

hydroxyl group absorption at ν 3400 cm^{-1} . The $^1\text{H-NMR}$ spectrum of **1** showed one set of AB doublet signals at δ 6.81 (1H, d, $J = 8.8$), and 7.31 (1H, d, $J = 8.8$), one methine proton at δ 5.32 (1H, dd, $J = 9.0, 3.4$), and two methylene protons at δ 2.67 (1H, dd, $J = 16.8, 9.0$)/3.08 (1H, dd, $J = 16.8, 3.4$), indicating that **1** was probably a 2,7,8-trihydroxychroman-4-one. The $^{13}\text{C-NMR}$ spectrum indicated that compound **1** had a total of 9 carbons, with the skeleton consisting of a chroman-4-one. The carbons of the chroman-4-one were assigned, from $^{13}\text{C-NMR}$ and DEPT experiments, one methylene at δ 45.9 (C-3); three methines at δ 92.4 (C-2), 126.8 (C-6) and 131.4 (C-5); and five quaternary carbons at δ 111.8 (C-4a), 135.7 (C-8), 150.6 (C-8a), 152.1 (C-7) and 182.9 (C-4). The structure of **1** was also confirmed by 2D NMR experiments. A COSY correlation was observed between H-2 and H-3, and between H-5 and H-6 (Figure 1). The HETCOR experiment showed that the carbon signals at δ 92.4 for C-2, 45.9 for C-3, 131.4 for C-5 and 126.8 for C-6 were correlated to the proton signals at δ 5.32 for H-2, 2.67/3.08 for H-3, 7.31 for H-5 and 6.81 for H-6, respectively. The relative configuration of **1** was determined by 2D NOESY analysis. The observation of the NOESY correlation from H-3eq. to H-2 suggested that H-2 was in the β -configuration (Figure 2). Thus, the structure of **1** was determined to be (S^*)-2,7,8-trihydroxychroman-4-one and has been named reticumanone.

Figure 1. COSY and HMBC Correlation of reticumanone (**1**).

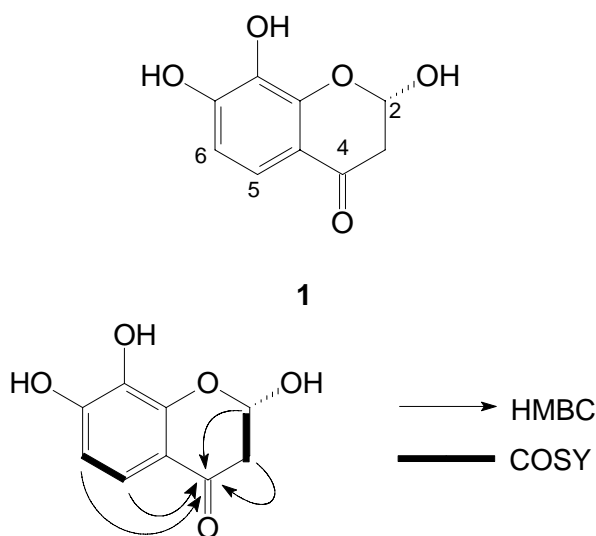
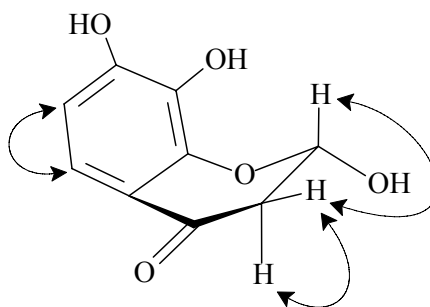


Figure 2. NOESY Correlations of reticumanone (**1**).



3. Experimental

3.1. General

UV spectra were obtained on a Jasco UV-240 spectrophotometer in CH₃CN, IR spectra were measured on a Hitachi 260-30 spectrophotometer. ¹H NMR (400 MHz), HETCOR, HMBC, COSY, NOESY, and DEPT spectra were obtained on a Varian (Unity Plus) NMR spectrometer. Low-resolution EIMS spectra were collected on a Jeol JMS-SX/SX 102A mass spectrometer or Quattro GC/MS spectrometer having a direct inlet system. High-resolution EIMS spectrum was measured on a Jeol JMS-HX 110 mass 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, visualized with 50% H₂SO₄.

3.2. Plant Material

The leaves of *C. reticulatum* Hay were collected from Pingtung County, Taiwan, May 2005. Plant material was identified by Professor Fu-Yuan Lu (Department of Forestry and Natural Resources College of Agriculture, National Chiayi University). A voucher specimen (Cinnamo. 6) was deposited in the School of Medical and Health Sciences, Fooyin University, Kaohsiung County, Taiwan.

3.3. Extraction and Isolation

The air-dried leaves of *C. reticulatum* Hay (3.4 kg) were extracted with *n*-hexane (30 L × 5) and CHCl₃ (30 L × 5) at room temperature and a *n*-hexane extract (43.5 g) and CHCl₃ extract (151.5 g) were obtained upon concentration under reduced pressure. The *n*-hexane extract (43.5 g) was chromatographed over silica gel (980 g, 70–230 mesh) using *n*-hexane/EtOAc/Acetone mixtures as eluents to produce five fractions. Part of fraction 4 (10.62 g) was subjected to silica gel chromatography by eluting with CHCl₃-MeOH (60:1), enriched with MeOH to furnish five further fractions (4-1–4-5). Fraction 4-2 (2.56 g) was further purified on a silica gel column using CHCl₃-MeOH mixtures to obtain reticumanone (4 mg).

Reticumanone ((*S**)-2,7,8-trihydroxychroman-4-one) (**1**): White amorphous powder (CHCl₃); [α]_D²⁵–12.6° (c 0.005, CHCl₃); UV/Vis (CH₃CN): λ_{max} (log ε): 273 (3.62), 310 (sh), 322 (3.00) nm; IR (neat) ν_{max}: 3400 (br, OH), 2920, 2850, 1660 (C=O), 1250 cm⁻¹; MS (EI, 70 eV): *m/z* (%): 196 [M]⁺ (45), 179 (57), 163 (64), 147 (100), 90 (32); HR-MSEI: *m/z* [M]⁺ calcd for C₉H₈O₅: 196.0372; found: 196.0374; ¹H NMR (400 MHz, CDCl₃) δ 2.67 (1H, dd, *J* = 16.8, 9.0 Hz, H-3ax.), 3.08 (1H, dd, *J* = 16.8, 3.4 Hz, H-3eq.), 5.32 (1H, dd, *J* = 9.0, 3.4 Hz, H-2), 6.81 (1H, d, *J* = 8.8 Hz, H-6), 7.31 (1H, d, *J* = 8.8 Hz, H-5); ¹³C NMR (100 MHz, CDCl₃): 45.9 (C-3, CH₂), 92.4 (C-2, CH), 111.8 (C-4a, C), 126.8 (C-6, CH), 131.4 (C-5, CH), 135.7 (C-8, C), 150.6 (C-8a, C), 152.1 (C-7, C), 182.9 (C-4, C).

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