

**LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU PEER REVIEW
KARYA ILMIAH: Jurnal Ilmiah**

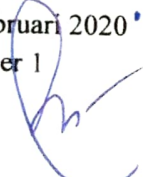
Judul karya ilmiah (artikel) : (22E, 24S)-24-Propylcholest-5en-3 α -acetate: A New Steroid from the Stembark of *Aglaia angustifoli* (Miq.) (Meliaceae)
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 Unit Kerja : FMIPA Universitas Negeri Gorontalo

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HASIL PENILAIAN SEJAWAT SEBIDANG ATAU PEER REVIEW

KARYA ILMIAH : Jurnal Ilmiah

Judul Artikel Ilmiah : (22E, 24S)-24-Propylcholest-5en-3 α -acetate: A New Steroid from the Stembark *Aglaia angustifoli* (Miq) (Meliaceae)
 Jumlah Penulis : 9 orang
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Prof. Dr. Ace Tatang Hidayat, M.Si
 NIP 196712291997021001
 Unit Kerja : Departemen Kimia FMIPA Unpad

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HASIL PENILAIAN SEJAWAT SEBIDANG ATAU PEER REVIEW
KARYA ILMIAH: Jurnal Ilmiah

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Nama : Prof. Dr. Feti Fatimah, MSi
 NIP : 196807291994 032002
 Unit Kerja : Kimia FMIPA Universitas Sam Ratulangi

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**LEMBAR
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Nama : Prof. Dr. Edi Suryanto, M.Si
 NIP : 196407241996031001
 Unit Kerja : Kimia FMIPA Universitas Sam Ratulangi

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(22E,24S)-24-Propylcholest-5en-
3 α -acetate: A New Steroid from
the Stembark *Aglaia angustifolia*
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(22E,24S)-24-Propylcholest-5en-3 α -acetate: A New Steroid from the Stembark *Aglaia angustifolia* (Miq.) (Meliaceae)

Ricson P. Hutagaol ^{1,2}, Desi Harneti ², Ace T. Hidayat ^{2,3}, Nurlelasari ², Rani Maharani ^{2,3}, Dewa Gede Katja ⁴, Unang Supratman ^{2,3,*}, Khalijah Awang ⁵ and Yoshihito Shiono ⁶

³ Department of Chemistry, Faculty of Mathematics and Natural Sciences, Nusa Bangsa University, Bogor 16166, West Java, Indonesia; ricson16001@mail.unpad.ac.id

² Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Padjadjaran, Jatinangor 45363, West Java, Indonesia; desi.harneti@unpad.ac.id (D.H.); hidayat_at@yahoo.com (A.T.H.); nurlelasari@unpad.ac.id (N.); r.maharani@unpad.ac.id (R.M.)

³ Central Laboratory, Universitas Padjadjaran, Jatinangor 45363, West Java, Indonesia

⁴ Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sam Ratulangi, Kampus Bahu, Manado 95115, North Sulawesi, Indonesia; dewakatja20@gmail.com

⁵ Department of Chemistry, Faculty of Science, University of Malaya, Kuala Lumpur 59100, Malaysia; khalijah@um.edu.my

⁶ Department of Food, Life, and Environmental Science, Faculty of Agriculture, Yamagata University, Tsuruoka, Yamagata 997-8555, Japan; yshiono@tds1.tr.yamagata-u.ac.jp

* Correspondence: unang.supratman@unpad.ac.id; Tel.: +62-22-779-4391

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Abstract: A new propylcholesterol-type steroid, namely (22E,24S)-24-propylcholest-5en-3 α -acetate ¹⁶ has been isolated from the stembark of *Aglaia angustifolia* (Miq.). The structure of **1** was determined on the basis of spectroscopic data including 1D- and 2D-NMR as well as high resolution mass spectroscopy analysis. Compound **1** showed weak activity against the MCF-7 breast cancer cell line.

Keywords: *Aglaia angustifolia*; Meliaceae; MCF-7; propylcholesterol

1. Introduction

The genus *Aglaia* is the largest genus in subtropical and tropical angiosperm plants belonging to the Meliaceae family, which consists of more than 130 species and is widely distributed in the Southern mainland of China, the Indo-Malaysian region, and the Pacific Islands [1,2]. Some parts of the stem of the *Aglaia* plant have been used in traditional medicine for the treatment of skin diseases, fever, and wounds [3,4]. During the course of our continuous investigation for biologically active compounds from Indonesian *Aglaia* plants, we already isolated several bioactive compounds from *A. smithii* [5], *A. eximia* [6–8], *A. argentea* [9,10], and *A. elliptica* [11,12]. In further investigation on *Aglaia* plants that grow in Indonesia, we found that *A. angustifolia* has not been studied phytochemically previously. In this paper, we report the isolation and structural elucidation of a new propylcholesterol-type steroid, (22E,24S)-24-propylcholest-5en-3 α -acetate (**1**) (Figure 1), along with its cytotoxic activity against MCF-7 breast cancer lines.

2. Results

Extraction and Isolation

The dried stem bark of *A. angustifolia* (1.97 kg) was extracted successively with *n*-hexane (3 × 4 L), ethyl acetate (3 × 4 L), and methanol (3 × 4 L) at rt to give a crude *n*-hexane extract (19.5 g), ethyl acetate (20.4 g), and methanol (35 g) after removal of the solvent. The *n*-hexane extract (19.0 g) was separated by vacuum liquid chromatography on Merck GF₂₅₄ silica gel using a 10% gradient of *n*-hexane-ethyl acetate-methanol solvent to give eight fractions (A–G). Fraction B (1.3 g) was subjected to column chromatography on silica gel using *n*-hexane:ethyl acetate (90:10) as a solvent to give ten subfractions (B1–B10). Subfraction B5 (44.2 mg) was further separated by column chromatography on silica gel using *n*-hexane:dichloromethane:ethyl acetate (90:9:1) to give **1** (5.6 mg).

(22*E*,24*S*)-24-propylcholest-5*en*-3*α*-acetate (**1**), white amorphous powder, $[\alpha]_{D_{29.4}}^{20} -0.56^{\circ}$ (*c*, 0.26, CHCl₃), IR (KBr) ν_{\max} : 2928, 2852, 1731, 1463, 1374, 1262, 1039 cm⁻¹; ¹H NMR (CDCl₃, 600 MHz) δ_{H} (ppm): 5.37 (1H, d, *J* = 6.0 Hz, H-6), 4.60 (1H, m, H-3), 2.34 (1H, m, H-4b), 2.13 (1H, m, H-4a), 2.03 (3H, s, Me-32), 2.01 (1H, m, H-12b), 1.97 (1H, m, H-7b), 1.87 (1H, m, H-2b), 1.86 (1H, m, H-1b), 1.84 (1H, m, H-16b), 1.67 (1H, m, H-25), 1.61 (1H, m, H-23b), 1.60 (1H, m, H-11b; H-15b), 1.59 (1H, m, H-2a), 1.57 (1H, m, H-28b), 1.54 (1H, m, H-7a), 1.46 (1H, m, H-11a), 1.45 (1H, m, H-8), 1.36 (1H, m, H-20), 1.32 (1H, m, H-22a; H-29b), 1.26 (1H, m, H-28a), 1.25 (1H, m, H-29a), 1.19 (1H, m, H-16a), 1.17 (1H, m, H-12a; H-23a), 1.13 (1H, m, H-1a), 1.12 (1H, m, H-17), 1.08 (1H, m, H-15a), 1.03 (3H, s, Me-19), 1.01 (1H, m, H-22b), 1.00 (1H, m, H-14), 0.97 (1H, m, H-9), 0.93 (1H, m, H-24), 0.91 (1H, d, *J* = 6.2 Hz, Me-27), 0.91 (3H, d, *J* = 6.0 Hz, Me-21), 0.84 (3H, t, *J* = 6.8 Hz, Me-30), 0.81 (3H, d, *J* = 6.2 Hz, Me-26), 0.68 (3H, s, Me-18); ¹³C NMR and DEPT-135 (CDCl₃, 150 MHz), δ_{C} (ppm): 170.9 (s, C-31), 139.8 (s, C-5), 122.8 (d, C-6), 74.2 (d, C-3), 56.9 (t, C-14), 56.3 (d, C-17), 50.2 (d, C-9), 46.0 (d, C-24), 42.5 (s, C-13), 39.9 (t, C-12), 38.3 (t, C-4), 37.2 (t, C-1), 36.8 (s, C-10), 36.3 (d, C-20), 34.1 (t, C-22), 32.1 (t, C-7), 32.0 (d, C-8), 29.9 (t, C-28), 29.3 (d, C-25), 28.4 (t, C-16), 28.0 (t, C-2), 26.2 (t, C-23), 24.4 (t, C-15), 23.3 (t, C-28), 21.2 (t, C-11), 21.6 (q, C-24), 20.8 (q, C-27), 19.5 (q, C-19), 19.2 (q, C-26), 19.0 (q, C-21), 12.2 (q, C-30), 12.0 (q, C-18); HR-TOFMS m/z 493.4088 [M + Na]⁺ (calcd. for C₃₂H₅₄O₂Na, m/z 493.4022).

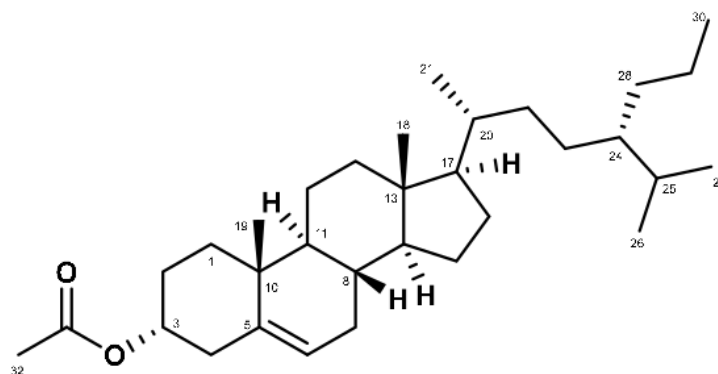


Figure 1. Chemical Structure of Compound 1.

3. Discussion

Compound **1** was isolated as a white amorphous powder with $[\alpha]_{D_{29.4}}^{20} -0.56^{\circ}$ (*c*, 0.26, CDCl₃). Its molecular composition was established to be C₃₂H₅₄O₂ from HRTOFMS m/z 493.4088 [M + Na]⁺, calculated for C₃₂H₅₄O₂Na (m/z 493.4022) and NMR spectral data. The Index of Hydrogen Deficiency (HD) was obtained from equation, HD = ΣC - ΣH/2 + 1, so the HD index of compound **1** was 50. The IR spectrum displayed bands that were ascribed to C–H stretching of aliphatic (2928 and 2852 cm⁻¹), C–H stretching of olefinic (3117 cm⁻¹), carbonyl ester (1731 cm⁻¹), and ether group (1262 and 1039 cm⁻¹).

The ^{13}C NMR spectrum showed 32 carbon resonances, which were classified by their chemical shifts, DEPT, and HMQC spectra as 7 methyl groups (2 tertiary, 3 secondary, 1 primary, and 1 acetyl), 12 methylene carbons, 9 methane carbons (1 oxygenated and 1 olefinic carbons), and 4 quaternary carbons (1 olefinic and 1 oxygenated carbons). These functionalities accounted for 2 out of the total 6 hydrogen deficiency indexes. The remaining four hydrogen deficiency indexes were consistent with the tetracyclic steroidal structure [13] with acetyl and olefinic as additional groups. The significant difference side chain structure of **1** with other sterol type steroids was the *n*-propyl side chain of **1** at C-24 instead of the ethyl group of other sterols like stigmasterol [10,11,13]. It was indicated by ^{13}C NMR spectrum of **1**, an additional chemical shift (29.9 ppm) at C-28 with δ_{H} (1.26 (1H, m, H-28a) and 1.57 (1H, m, H-28b)) and showed as CH_2 with DEPT spectra of **1**.

The ^1H NMR spectrum of **1** displayed the presence of two tertiary methyls at δ_{H} 0.68 (3H, s, Me-18) and 1.03 (3H, s, Me-19). Three secondary methyls of **1** were seen at δ_{H} 0.91 (3H, d, $J = 6.0$ Hz, Me-21), 0.81 (3H, d, $J = 6.2$ Hz, Me-26), and 0.91 (3H, d, $J = 6.2$ Hz, Me-27). One primary methyl of **1** was seen at δ_{H} 0.84 (3H, t, $J = 6.8$ Hz, Me-30); this ^1H NMR spectrum of primary methyl Me-30 corresponded to the C-30 methyl group of the *n*-propyl group side chain structure of compound **1** at C-24 [14]. The acetylenic, olefinic, and oxygenated methine signals were also observed in ^1H -NMR at δ_{H} 2.03 (3H, s, Me-32), 5.37 (1H, d, $J = 6.0$ Hz, H-6), and 4.60 (1H, m, H-3), respectively.

A comparison of the NMR data of **1** with 24 ϵ -*n*-propylcholesterol compounds, isolated from a cultured marine chrysophyte [14], and with (24*R*)-24-propylidenecholesterol isolated from *Aureocumbra lagunensis*, the Texas brown tide alga [15], and with a stereospecific synthesis 24-propylcholesterol isolated from the Texas brown tide [16], indicated that the structure of **1** is very similar to the synthetic compound (24*S*)-24-propylcholest-5-en-3 β -ol. The main difference was the absence of a hydroxyl group and the presence of an acetyl group, which suggested that **1** was an acetyl derivative of (24*E*,24*S*)-24-propylcholest-5-en-3 β -ol. The ^1H - ^{13}C COSY spectrum of **1** (Figure 2, see the Supplementary Materials), showed correlations in H₁-H₂-H₃-H₄, H₆-H₇-H₈-H₉-H₁₁-H₁₂, H₁₄-H₁₅-H₁₆-H₁₇-H₂₀-H₂₂-H₂₃-H₂₄-H₂₅-H₂₆, and H₂₄-H₂₈-H₂₉-H₃₀, supporting the presence of propylcholesterol structure with acetoxyl group in **1**. The HMBC correlations from the tertiary, secondary, and primary methyl protons to their neighboring carbons enabled the assignment of the two tertiary methyls at C-10 and C-13, secondary methyls at C-20 and C-25 (2 \times), as well as a primary methyl at C-29, respectively. Furthermore, the olefinic proton at δ_{H} 5.37 (1H, d, $J = 6.0$ Hz, H-6) was correlated to olefinic carbon at δ_{C} 139.8 at methylene carbon at δ_{C} 32.1, indicating an olefinic moiety was located at C-5 and C-6 ($\Delta^{5,6}$), similar to stigmaster-5en-3 α -acetate [10,11].

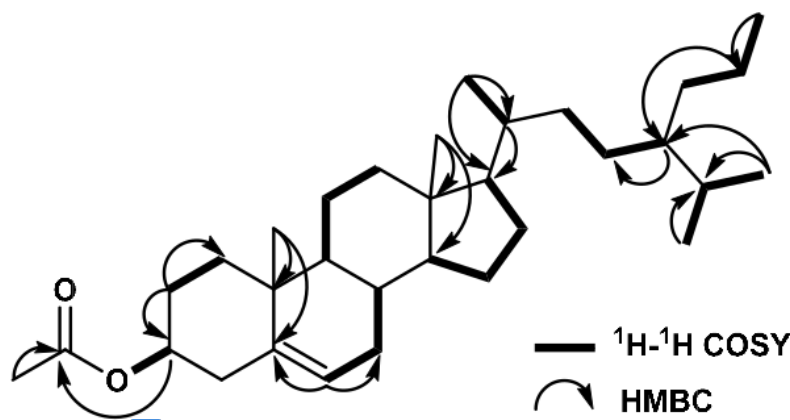


Figure 2. Selected ^1H - ^1H COSY and HMBC correlations for **1**.

Correlations from oxygenated methine proton at δ_{H} 4.60 (H-3) and methyl acetyl at δ_{H} 2.03 (Me-32) to carbonyl ester at δ_{C} 170.9 (C-31) were used to assign an acetyl group that was located on C-3.

The relative configuration of compound **1** was identified by a ^1H - ^1H NOESY experiment (Figure 3), which showed the NOESY correlations between Me-19 and H-8 as well as H-1 and H-3, which indicated that the acetyl group at C-3 was α -oriented. Similar to the observations from ^1H - ^1H NOESY, the cross peak between Me-18 and H-20, and H-20 and H-24 indicated that H-20 and H-24 were β -oriented. This result was supported also by comparing it to those of stigmast-5en-3 α -acetate [10,11], and the specific optical rotation of **1** ($[-\alpha]_D^{25} = -0.26^\circ$ (c , 0.26, CDCl_3) was the same negative sign to that of the previously reported (22*E*,24*S*)-24-propylcholest-5en-3 β -ol ($[\alpha]_D^{25} = -29.5^\circ$ (c 0.40, CH_2Cl_2) [16]. Therefore, compound **1** was elucidated to be a new derivative propylcholesterol-type steroid and was named (22*E*,24*S*)-24-propylcholest-5en-3 α -acetate.

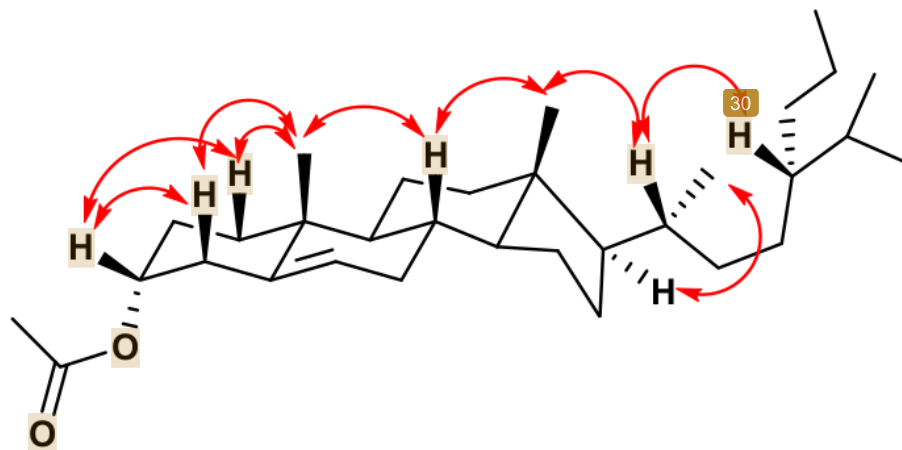


Figure 3. Selected NOESY correlations for **1**.

The compound (22*E*,24*S*)-24-propylcholest-5en-3 α -acetate (**1**), was evaluated for its cytotoxicity against the MCF-7 breast cancer cell line according to a method described previously [17], and cisplatin (IC_{50} 38.06 $\mu\text{g/mL}$) was used as a positive control [18,19]. Compound **1** showed weak activity against MCF-7 with an IC_{50} value of 60.4 μM . This result is in line with previously investigations where a steroid derivative showed weak activity against the breast cancer line [10,11].

4. Materials and Methods

4.1. General Experimental Procedures

The optical rotation was obtained with an Autopol IV automatic polarimeter. Mass spectra were measured with a Waters Xevo QTOFMS (Waters, Milford, MA, USA). NMR data were recorded on a Bruker Avance-600 spectrometer at 600 MHz for ^1H and 150 MHz for ^{13}C using TMS as an internal standard (Billerica, MA, USA). Chromatographic separations were carried out on silica gel (60, 70–230, and 200–400 mesh) (Merck, Darmstadt, Germany). TLC plates were precoated with silica gel GF₂₅₄ (Merck, Darmstadt, Germany, 0.25 mm), and detection was achieved by spraying with 10% H_2SO_4 in ethanol, followed by heating.

4.2. Plant Material

The stem bark of *A. angustifolia* (Miq.) was collected from Bogor Botanical Garden, Bogor, Indonesia in February 2017. The plant was identified by Mr. Ismail, the staff of the Bogoriense Herbarium, Indonesian Science Institute, Bogor, Indonesia. A voucher specimen (II.K.57a) was deposited at the herbarium.

4.3. Cytotoxic Bioassay

The cytotoxicity of the compounds against MCF-7 human breast cancer cells was measured using the PrestoBlue cell viability assay. The cells were maintained in a Roswell Park Memorial Institute (RPMI) medium supplemented with 10% (v/v) Fetal Bovine Serum (FBS) and 1 μ L/mL antibiotics. Cultures were incubated at 37 °C in a humidified atmosphere of 5% CO₂. Tumor cells were seeded in 96-well microliter plates at 1.7×10^4 cells per well. After 24 h, compounds were added to the wells. After 96 h, cell viability was determined by measuring the metabolic conversion of resazurin substrate into pink fluorescent resorufin product resulting from reduction in viable cells. The PrestoBlue assay results were read using a multimode reader at 570 nm. All compounds were tested at eight concentration (7.81; 15.63; 31.25; 62.50; 125.00; 250.00; 500.00; 1000.00 μ g/mL) in 100% DMSO with a final concentration of DMSO of 2.7% in each well. Each concentration of the compounds was tested in two parallels experiments. IC₅₀ values were calculated by the linear regression method using Microsoft Excel software.

5. Conclusions

A new propyl cholesterol type-steroid, namely (22E,24S)-24-propylcholest-5en-3 α -acetate (**1**), was isolated from the stem bark of *A. angustifolia* (Miq.) belonging to the Meliaceae family. This propyl cholesterol type-steroid was found in the *Aglaia* genus for the first time.

Supplementary Materials: The following are available online, Figure 1.1a,b. ¹H-NMR Spectrum of **1** (600 MHz in CDCl₃), Figure 1.2. ¹³C-NMR Spectrum of **1** (150 MHz in CDCl₃), Figure 1.3a,b. DEPT-135° Spectrum of **1** (in CDCl₃), Figure 1.4. HSQC Spectrum of **1**, Figure 1.5. HMBC Spectrum of **1**, Figure 1.6. ¹H-¹H COSY Spectrum of **1**, Figure 1.7. NOESY of Spectrum of **1** (600 MHz in CDCl₃), Figure 1.8. HR-TOF-MS Spectrum of **1**, Figure 1.9. TLC Profile of **1**.

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Conflicts of Interest: The authors declare no conflict of interest.

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