

1,1,4,7-Tetramethyldecahydro- 1H-cyclopropa[e]azulen- 7-ol from the Stembark *Chisocheton* *pentandrus*

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1,1,4,7-Tetramethyldecahydro-1H-cyclopropa[e]azulen-7-ol from the Stembark *Chisocheton pentandrus*

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Abstract: A new aromadendrane-type sesquiterpenoid, namely dehydrospatulanol (**1**), has been isolated from the stembark of *Chisocheton pentandrus*. The chemical structure of **1** was characterized on the basis of spectroscopic evidences including mainly one dimension and two dimension Nuclear Magnetic Resonance, and Mass Spectroscopy as well as through a comparison with those related compounds previously reported.

Keywords: aromadendrane *Chisocheton pentandrus*; Meliaceae; sesquiterpenoid

1. Introduction

Chisocheton plants have been known to be a rich source of secondary metabolites including various sterols, limonoids, terpenoids, and alkaloids with biological properties such as antifungal, antibacterial, antiviral, anti-inflammatory, cytotoxic, and antiplasmodial agents [1–4]. In our previous research for novel cytotoxic constituents from Indonesia *Chisocheton*, we isolated and described limonoids, dysobinol from the seed *C. macrophyllus* [5], pentandricine from stem bark *C. pentandrus* [6], four new apo-euphane-type triterpenoid from the bark of *C. patens* [1] and a triterpenoid from *C. cumingianus* and *C. celebicus* [7,8]. In the further search for anticancer candidate compounds from *C. pentandrus*, we found a new aromadendrane-type sesquiterpenoid, namely dehydrospatulanol (**1**) from the stembark of *C. pentandrus*. In this communication, the isolation and structural determination of the new aromadendrane-type sesquiterpenoid are described.

2. Results

11 Extraction and Isolation

The dried stem bark of *C. pentandrus* (3.8 kg) was extracted with MeOH at room temperature to give a crude MeOH extract (560 g) after solvent was removed. The crude MeOH extract (560 g) was partitioned between n-hexane and ether to give the n-hexane fraction (96.6 g) after evaporation of the solvent. The n-hexane soluble fraction was separated by column chromatography (CC) using gradient n-hexane/EtOAc to give eight fractions (A–H). Fraction A (3.3 g) was separated by medium pressure

pressure liquid chromatography (MPLC) on silica using isocratic of MeOH:H₂O (8:2) to give 12 liquid chromatography (MPLC) on silica using isocratic of MeOH:H₂O (8:2) to give 12 subfractions (A1–12). Subfraction A9 (1.5 g) was subjected to column chromatography (CC) using (A1–12). Subfraction A9 (1.5 g) was subjected to column chromatography (CC) using CH₂Cl₂ to give CH₂Cl₂ to give three subfractions (A9.1–9.3). Compound 1 (335 mg) (Figure 1) was obtained by further three subfractions (A9.1–9.3). Compound 1 (335 mg) was obtained by further purification of subfraction A9.3 (0.6 g) on silica gel eluted with n-hexane as a mobile phase.

Dehydroshatulanol (1), colorless oil, $[\alpha]_D^{25} +7.2$ (c, 0.17, CH₃OH), ¹H NMR (CDCl₃, 500 MHz), Dehydroshatulanol (1), colorless oil, $[\alpha]_D^{25} +7.2$ (c, 0.17, CH₃OH), ¹H NMR (CDCl₃, 500 MHz), ¹³C NMR (CDCl₃, 125 MHz), C (ppm), see Table 1. HR-TOFMS *m/z* 223.2064 [M + H]⁺ see Table 1. ¹³C NMR (CDCl₃, 125 MHz), C (ppm), see Table 1. HR-TOFMS *m/z* 223.2064 [M + H]⁺ (calcd. for C₁₅H₂₆O, *m/z* 222.2084).

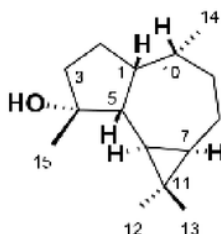


Figure 1. Chemical structure of compound 1.

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Table 1. ¹³C NMR (CDCl₃) and ¹H NMR (CDCl₃) data for compound 1 (2019) in CDCl₃ (500 MHz).

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C	δ _c (ppm)	δ _H (ppm)	Integration	Multiplicity	J (Hz)
1	39.7	1.72	1.72	(1H, m)	
1	4.7	1.72	1.72	(1H, m)	
1,15	29.1	1.15	1.15	(1H, m)	
2	29.1	1.68	1.68	(1H, m)	
3	37.8	1.59	1.45	(1H, m)	
4	76.6	1.69	1.69	(1H, m)	
5	58.2	0.10	0.10	(1H, t, 9.3)	
6	22.3	0.51	0.51	(1H, ddd, 6.0, 9.6)	
6	28.6	0.51	0.51	(1H, m)	
7	28.6	1.49	1.49	(1H, m)	
8	18.8	1.54	1.54	(1H, m)	
9	25.8	1.85	1.85	(1H, m)	
10	25.8	1.85	1.85	(1H, m)	
10	18.438.5	-1.85	-1.85	(1H, m)	
12	16.3	0.88	0.88	(3H, s)	
11	16.3	0.88	0.88	(3H, s)	
12	16.3	0.84	0.84	(3H, d, 6.8)	
13	28.7	0.92	0.92	(3H, s)	
14	16.1	0.84	0.84	(3H, d, 6.8)	
15	32.1	1.04	1.04	(3H, s)	

3. Discussion

Compound 1 was obtained as a colorless oil with $[\alpha]_D^{25} +7.2$ (c, 0.17, CH₃OH) and the High Resolution ⁴¹ne of Flight-Mass Spectroscopy (HRTOF-MS) spectra showed a pseudomolecular ion peak at 223.2064 [M + H]⁺, corresponding to the molecular formula C₁₅H₂₆O (calculated *m/z* 222.2084). Compound 1 was obtained as a colorless oil with $[\alpha]_D^{25} +7.2$ (c, 0.17, CH₃OH) and the High ¹H NMR spectrum of 1 showed four methyls at δ 0.82 (3H, d, J = 6.82 Hz, Me-14), 0.88 (3H, s, Me-Resoluto), ⁴⁰ne of Flight-Mass Spectroscopy (HRTOF-MS) spectra showed a pseudomolecular ion peak at 223.2064 [M + H]⁺ corresponding to the molecular formula C₁₅H₂₆O (calculated *m/z* 222.2084). ³⁹ (1H, m), 1.29 and 1.52 (2H, m, H-8), 1.49 and 1.54 (2H, m, H-9), 1.45 and 1.59 (2H, m, H-3), five The ¹H NMR spectrum of 1 showed four methyls at δ 0.82 (3H, d, J = 6.82 Hz, Me-14), 0.88 (3H, s, methine protons at δ 0.01 (1H, t, J = 9.3 Hz, H-6), 0.51 (1H, ddd, J = 6.05 and 9.6 Hz, H-7), 0.69 (4H, m, Me-12), 0.92 (3H, s, Me¹⁴-13), and 1.04 (3H, s, Me-15), each 3H, four methylene proton at δ 1.15 and 1.68 (2H, m, H-1), and 1.85 (1H, m, overlap, H-10). The ¹³C NMR (Table 1) and Distortionless (2H, m, H-2), 1.29 and 1.52 (2H, m, H-8), 1.49 and 1.54 (2H, m, H-9), 1.45 and 1.59 (2H, m, H-3), five Enhancement by Polarization Transfer (DEPT) spectra revealed 15 carbon resonances due to two sp³ quaternary carbons at δ_c 18.4 (C-11) and 76.6 (C-4) and five sp³ methines at δ_c 22.3 (C-6), 28.6 (C-7),

19 were recorded on a Bruker Topspin spectrometer at 600 MHz for ¹H and 150 MHz for ¹³C using Tetramethylsilane (TMS) as an internal standard (Bruker, Billerica, MA, USA). Medium performance liquid chromatography was undertaken using a Buchi Pump Controller C-610, Buchi Pump Modules C-605 with FLH-R10030B SiliCycle column-ISO04 (SiliasepTM, Buchi, Switzerland). Silica gel 60 was used for column chromatography (Merck, Darmstadt, Germany). Thin layer chromatography plates were precoated with silica gel GF254 (Merck, Darmstadt, Germany, 0.25 mm) and detection was achieved by spraying with 10% H₂SO₄ in EtOH, followed by heating and irradiation under UV-Vis light at wavelengths of 254 and 364 nm.

25 4.2. Plant Material

The stem bark of *C. pentandrus* was collected in Halimun Salak Mountain National Park, Sukabumi, West Java Province, Indonesia. The plant was identified by the staff of the Bogoriense Herbarium, Bogor, Indonesia. A voucher specimen (MSF-G01) was deposited at the herbarium.

5. Conclusions

A new aromadendrane-type sesquiterpenoid, namely, dehydrospathulenol (**1**), was isolated from the stem bark of *Chisocheton pentandrus*. This examination confirms that *Chisocheton pentandrus* is capable of producing sesquiterpenoid-type compounds.

1 **Supplementary Materials:** The following are available online, Figure S1: ¹H-NMR Spectrum of **1** (500 MHz in CDCl₃), Figure S2: ¹³C-NMR Spectrum (125 MHz in CDCl₃), Figure S3: DEPT-135 Spectrum of **1** (in CDCl₃),

Figure S4: HSQC Spectrum of **1**, Figure S5: HMBC Spectrum of **1**, Figure S6: H-H-COSY Spectrum of **1**, Figure S7: NOE-1D Spectrum of **1** (500 MHz in CDCl₃), Figure S8: HRESI-TOF-MS Spectrum of **1**, Figure S9: TLC profile of **1**.

Author Contributions: Conceptualization, K.A.; Data curation, D.G.K.; Formal analysis, M.H.H.; Investigation, M.S.F.; Methodology, N.; Supervision, U.S.

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

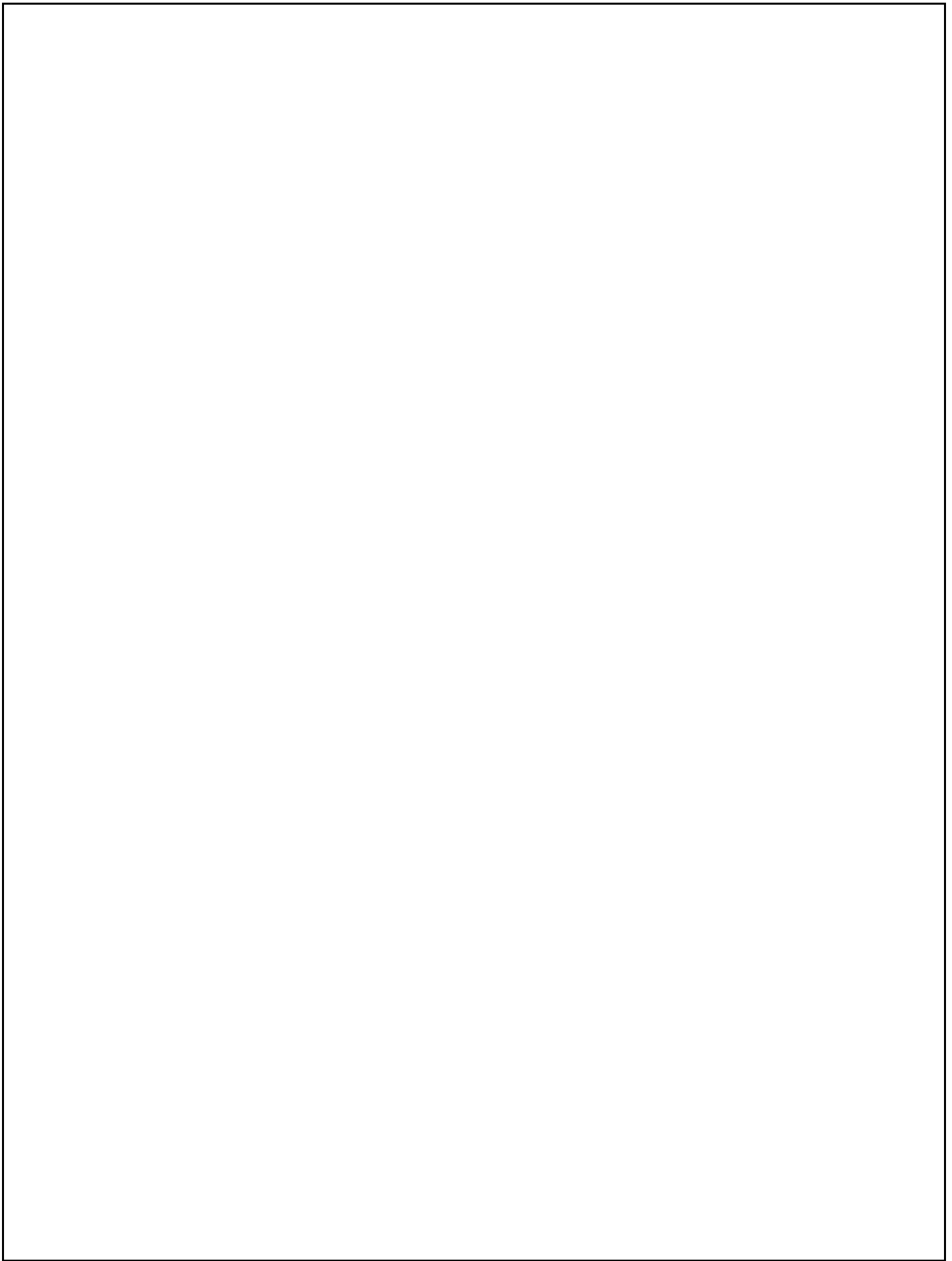
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