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Isolation and structure elucidation of a new alkaloid from the Indonesian Blue-Green Alga *Arthrospira platensis*

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Abstract

A new alkaloid (**1**) and two known compounds (**2** and **3**) were isolated from the blue-green alga *Arthrospira platensis*. Their structures were determined on the basis of spectroscopic data. According to the obtained data and values in the literature, the compounds were concluded to be eckol.

Keywords: Isolation and structure elucidation; Alkaloid; *Athrospira platensis;* eckol.

1. Introduction

Arthrospira platensis is a very common blue-green alga that inhabits the South Pacific coast around East Indonesia. Nakamura et al. reported the isolation of eckol and dieckol from this raw material and described the antioxidant activity of these compounds [1]. In another paper, Iwahori et al. also described the inhibitory effect of *A. platensis* on aldose reductase [2], which reduces glucose conversion to sorbitol in cells. Accumulation of sorbitol in cells leads to the development of various chronic complications of diabetes, such as cataracts, neuropathy, and retinopathy. Meanwhile, carrier proteins in blood vessels, structural proteins, and enzymes in the body are modified by glucose in a process called glycation. It is known that accumulation of these glycated proteins (AGE) causes diabetic complications. An inhibitor of glycation has been shown to retard the development of diabetic complications in experimental diabetes [3]. In the present paper, we report the isolation of one new and two known phloroglucinol derivatives from *A. platensis*.

2. Experimental

2.1. Plant Material

A. platensis was purchased from CV. Bahari Perkasa (Jakarta, Indonesia). A voucher specimen was deposited at the Organic Chemistry Laboratory, School of Chemistry, University of Wollongong, Australia.

2.2. General Procedures

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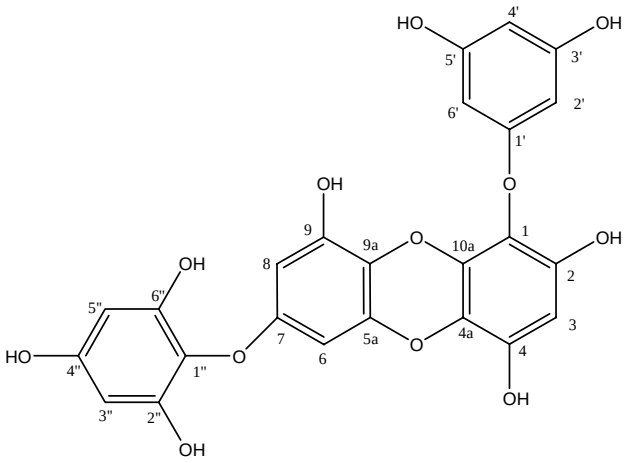
A. platensis (10 kg) was extracted with MeOH (18 L) three times for 3 h under reflux, and the solvent was evaporated in vacuo to give MeOH extract (329 g). This extract was subjected to Diaion HP-20 (to yield the corresponding fractions. The MeOH fraction (123.9 g) was subjected to Diaion HP-20 to give fractions 1-10. Fraction 4 (6.2 g) was chromatographed over TOYOPEARL HW-40F [MeOH] to afford fractions 11-14. Fraction 13 (927.8 mg) was subjected to CPC (*n*-butanol-*n*-propanol-H_2O, 4:1:5) to give fractions 15-23. Fraction 21 (696 mg) and fraction 22 (62.4 mg) were subjected to reversed-phase HPLC (C-8, 18, 22) eluting with a MeOH-H_2O mixture. **3** (8.6 mg) was isolated from fraction 21, and **1** (4.5 mg) and **2** (3.2 mg) were isolated from fraction 22.



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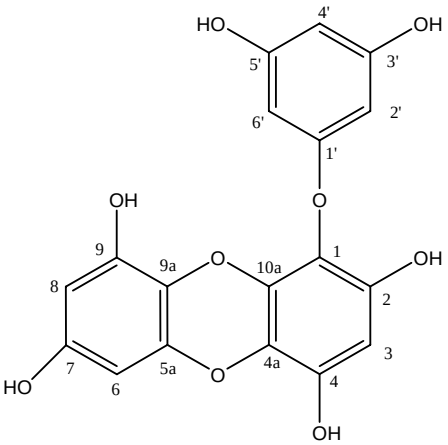

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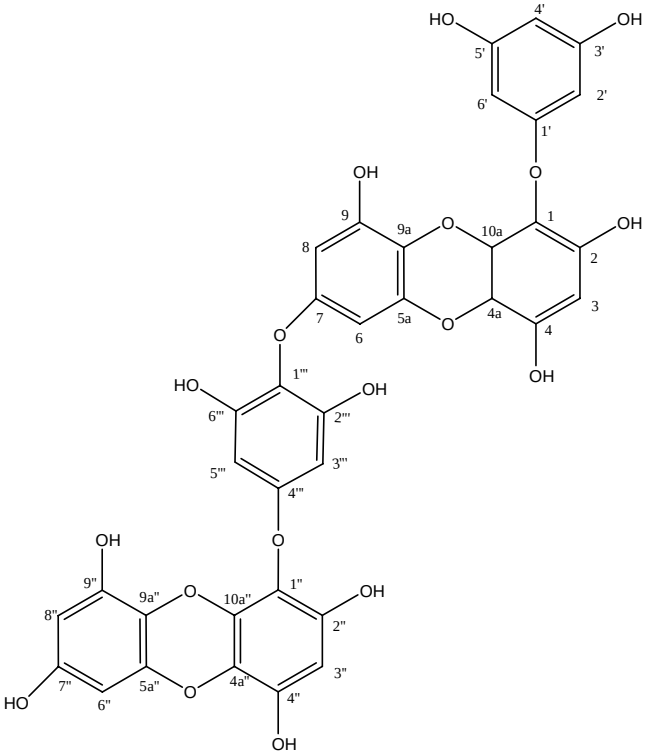

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3. Results and discussion

Compound **1** had the molecular formula $C_{24}H_{16}O_{12}$ as determined from its HRFABMS, ^{13}C NMR, and ^{13}C DEPT spectral data. The ^{1}H NMR spectrum of **1** showed an AB_2 system at δ 5.71 (2H, *J*) 2.1 Hz), 5.79 (1H, *J*) 2.1 Hz), an AB system at δ 5.78 (1H, *J*) 2.8 Hz), 6.00 (1H, *J*) 2.8 Hz), and two singlets at δ 6.13 (1H) and 5.85 (2H) as well as eight phenolic OH protons at δ 9.00 (1H), 9.13 (2H), 9.14 (2H), 9.20 (1H), 9.40 (1H), and 9.61 (1H). The ^{13}C NMR spectrum indicated the presence of eight nonsubstituted and 16 O-bearing aromatic carbons. The ^{13}C NMR spectrum is very similar to that of eckol (**2**), except for four extra signals, indicating that **1** was composed of four phloroglucinol units. The molecular weight of **1** was 124 more than eckol (496 vs 372). Detailed assignment of the protons and carbons was accomplished by means of the HMQC, HMBC, and NOE experiments (Fig. 1).

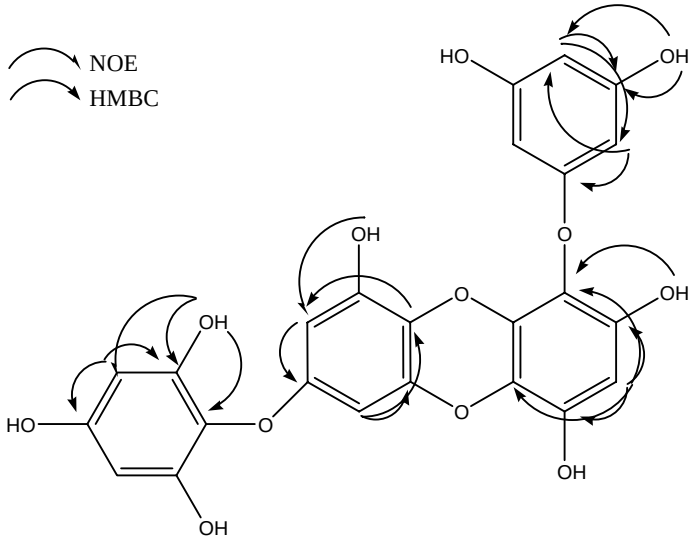

Fig. 1. HMBC and NOE of compound **1**.

The position of the additional phloroglucinol moiety was determined to be C-7 from the fact that the ^{13}C NMR signals for the basic skeleton in **1** were almost identical with those of **2** except for C-7 and C-9a. These signals were observed at low field compared to those of **2**. A similar phenomenon was reported by Suzuki et al. [4] on elucidation of another phloroglucinol derivative, 2-phloroeckol. Consequently, it was concluded that **1** was 1-(3',5'-dihydroxyphenoxy)-7-(2',4'',6''-trihydroxyphenoxy)-2,4,9-trihydroxydibenzo-1,4-ioxin (**1**).

Compound **2** had the molecular formula $C_{18}H_{12}O_9$ as determined from EIMS, ^{13}C NMR, and ^{13}C DEPT spectral data. In the ^{1}H NMR spectrum of **2**, an AB_2 system at δ 5.71 (2H, *J*) 2.0 Hz), 5.79 (1H, *J*) 2.0 Hz), an AB system at δ 5.78 (1H, *J*) 2.6 Hz), 5.95 (1H, *J*) 2.6 Hz), and a singlet at δ 6.13 (1H) were observed in addition to six phenolic OH protons at δ 9.12 (2H), 9.15 (2H), 9.42 (1H), and 9.52 (1H). The ^{13}C NMR spectrum indicated the presence of six nonsubstituted and 12 O-bearing aromatic carbons. The ^{1}H and ^{13}C NMR signals were assigned with the aid of HMQC, HMBC, and NOE experiments. According to the above data and values in the literature [5, 6]. **2** was concluded to be eckol.

Compound **3** had the molecular formula $C_{36}H_{22}O_{18}$ as determined from FABMS, ^{13}C NMR, and ^{13}C DEPT spectral data. The ^{1}H NMR spectrum of **3** showed an AB_2 system at δ 5.78 (2H, *J*) 2.0 Hz), 5.86 (1H, *J*) 2.0 Hz) and two AB systems at δ 5.88 (1H, *J*) 2.7 Hz), 6.08 (1H, *J*) 2.7 Hz) and δ 5.87 (1H, *J*) 2.7 Hz), 6.05 (1H, *J*) 2.7 Hz). Furthermore, three singlet signals in the ^{1}H NMR at δ 6.01 (2H), 6.02 (1H), and 6.22 (1H) and nine phenolic OH proton signals at δ 9.18 (2H), 9.24 (1H), 9.26 (1H), 9.31 (1H), 9.38 (2H), 9.48 (1H), 9.53 (1H), 9.63 (1H), and 9.73 (1H) were observed. The ^{13}C NMR signals indicated the presence of nine nonsubstituted and 23 O-

bearing aromatic carbons. The above data for **3** were consistent with the literature values for dieckol [1, 5, 6].

Table 1
^{1}H NMR data for compound **1** in DMSO-d_6.

3	6.13 (1H, s)
6	5.78 (1H, d, 2.8
8	6.00 (1H, d, 2.8)
2',6'	5.71 (2H, d, 2.1)
4'	5.79 (1H, t, 2.1)
3''	5.85 (1H, s)
5''	5.85 (1H, s)
6''	
8''	
2''',6'''	
2-OH	9.20 (1H, s)
4-OH	
7-OH	9.40 (1H, s)
9-OH	9.61 (1H, s)
3',5'-OH	9.14 (2H, s)
2''-OH	9.13 (1H, s)
4''-OH	9.00 (1H, s)
6''-OH	9.13 (1H, s)
7''-OH	
9''-OH	
3''',5'''-OH	

Table 2
^{13}C data for compounds **1**-**3** in DMSO-d_6.

	1[a]	**2**[b]	**3**[c]
1	122.2 s	122.9 s	122.1 s
2	145.9 s	145.6 s	145.7 s
3	98.3 d	97.9 d	98.1 d
4	141.9 s	141.5 s	141.6 s
4a	123.2 s	121.9 s	123.1 s
5a	142.4 s	142.1 s	142.3 s
6	93.5 d	92.3 d	93.2 d
7	154.5 s	154.3 s	154.4 s
8	98.2 d	97.4 d	97.8 d
9	146.1 s	145.7 s	146.0 s
9a	124.0 s	123.4 s	123.7 s
10a	137.1 s	136.5 s	137.0 s
1'	160.3 s	160.1 s	160.2 s
2',6'	93.7 s	92.4 s	93.5 s
3',5'	158.8 s	158.2 s	158.6 s
4'	96.2 d	95.6 d	96.1 d
1''	122.6 s		122.1 s
2''	151.2 s		145.7 s
3''	94.9 d		98.1 d
4''	154.8 s		141.8 s
4a''			123.1 s
5''	94.9		
5a''			142.4 s
6''	151.2 s		93.7 d
7''			152.8 s
8''			98.4 d
9''			145.4 s
9a''			122.4 s
10a''			136.1 s
1'''			156.7 s
2'''			94.2 s
3'''			150.3 s
4'''			124.1 s

4. Conclusion

In conclusion, we have reported a new alkaloid and two known compounds isolated from the blue-green alga *Arthrospira platensis*.

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