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Two new xanthons from *Garcinia rigida* leaves

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Two new xanthons, musaxanthone (**1**) and asmaxanthone (**2**) were isolated from the leaves of *Garcinia rigida*. The structures were determined by means of spectroscopic analysis.

Keywords: *Garcinia rigida*; Guttiferae; Musaxanthone; Asmaxanthone

1. Introduction

Plants of genus *Garcinia* (Guttiferae) grow mainly in Southeast Asia. We here report on the isolation and structural elucidation of two new xanthons, Musaxanthone (**1**) and Asmaxanthone (**2**) from the hexane extract.

2. Results and discussion

The hexane extract of *Garcinia rigida* leaves was fractionated on silica gel column, affording 8 fractions. Fraction 5 gave **1** (40 mg) and fraction 7 gave **2** (132 mg).

Musaxanthone (**1**), isolated as yellow light needles, had a molecular formula $C_{20}H_{16}O_6$ (352.1020 Calcd 352.1025). In the 1H -NMR spectrum, one aromatic proton singlet [δ 6.44 (1H, s, H-2)], two *cis*-olefinic protons in doublets [δ 6.85 (1H, d, $J=8.5$ Hz, H-7) and 7.86 (1H, d, $J=8.5$ Hz, H-8)], implying the presence of a dimethylchromene ring were fused in a shoulder fashion to the xanthon nucleus. The orientation of the dimethylchromene ring was precisely determined by 2D NMR techniques (HMQC and HMBC). The spectrum further showed the presence of two *cis*-olefinic protons in doublets [δ 6.61 (1H, d, $J=10.0$ Hz, H-1') and 5.58 (1H, d, $J=10.0$ Hz, H-2')]. All carbons bearing protons were assigned from 1H - ^{13}C COSY NMR spectrum are

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shown in table 1. In the ROESY, cross peak was observed between the H at 1-OMe and H-2. This structural assignment was confirmed by its HMBC and ROESY (figure 1) spectra.

Asmaxanthone (**2**) isolated as yellow needles, had a molecular formula $C_{21}H_{20}O_7$ (384.1281 Calcd 384.1287) in the HREIMS spectrum. A comparison of the 1H - and ^{13}C -NMR data of **2** with those of **1** revealed that the only difference was the substituent at C₁, C₅, C₆, and C₈, the methoxyl group in **1** was replaced by the hydroxyl group in **2**, the methylenedioxy in **1** was replaced by the methoxyl groups in **2** and the proton in **1** was replaced by methoxy in **2**. In the HMBC spectrum and the proton in **1** replaced by methoxy in **2**, cross peaks between proton C-1' and C-2 (δ 94.9), C-4 (δ 105.2), C-4a (δ 158.9), C-5' (δ 28.2) were observed. On the other hand, in the HMBC spectrum cross peaks between proton C-7 and C-5 (δ 130.1), C-6 (δ 158.4), C-8 (δ 159.4) and C-8a (δ 159.4) were observed. In the ROESY cross peaks were observed between the H at 6-OMe and H-7; and between the H-7 and H at 8-OMe (figure 1). All carbons bearing protons were assigned from 1H - ^{13}C COSY NMR spectrum, shown in table 1.

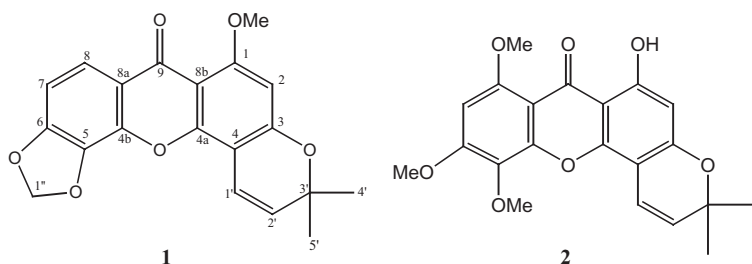


Table 1. ^{13}C -NMR assignment for musaxanthone (**1**) and asmaxanthone (**2**) in $CDCl_3$.

C	1		2	
	1H	^{13}C	1H	^{13}C
1	—	159.4	—	159.9
2	6.44 (1H, s)	91.4	6.30 (1H, s)	91.6
3	—	158.1	—	151.4
4	—	107.2	—	105.2
4a	—	155.4	—	158.0
4b	—	152.3	—	155.9
5	—	133.6	—	130.1
6	—	155.4	—	157.7
7	6.85 (1H, d, $J=8.5$ Hz)	107.2	6.68 (1H, s)	94.5
8	7.86 (1H, d, $J=8.5$ Hz)	121.4	—	157.3
8a	—	119.3	—	104.7
9	—	174.0	—	181.1
8b	—	112.8	—	103.3
1'	6.61 (1H, d, $J=10.0$ Hz)	115.8	6.66 (1H, d, $J=10.0$ Hz)	115.5
2'	5.58 (1H, d, $J=10.0$ Hz)	127.7	5.71 (1H, d, $J=10.0$ Hz)	127.1
3'	—	79.1	—	77.9
4'	1.55 (3H, s)	27.8	1.43 (3H, s)	28.2
5'	1.55 (3H, s)	27.8	1.43 (3H, s)	28.2
1''	6.16 (2H, s)	102.9	—	—
1-OMe	3.89 (3H, s)	55.9	—	—
5-OMe	—	—	3.84 (3H, s)	61.5
6-OMe	—	—	3.95 (3H, s)	56.4
8-OMe	—	—	4.03 (3H, s)	56.2

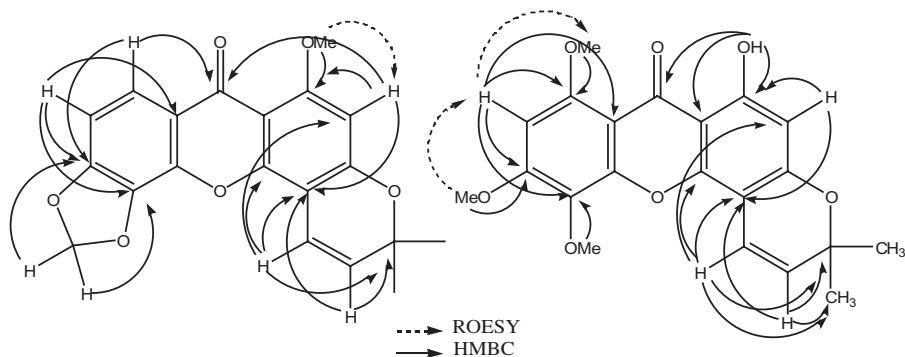


Figure 1. Selected HMBC and ROESY correlations.

3. Experimental

3.1. General experimental procedures

FTIR spectra were measured on Bio-Rad Merlin Spectrophotometer. MS were performed on an Autospec 3000 spectrometer at 70 eV. The NMR spectra were recorded on Bruker AM-400 and DRX-500 spectrometers.

3.2. Plant material

The leaves of *G. rigida* (Guttiferae) were collected in Bogor, Indonesia, in October 2002. A voucher specimen has been deposited in the Pharmacy Department of the University of Indonesia.

3.3. Extraction and isolation

The air-dried leaves (900 g) were extracted with hexane for a week. The extract hexane was concentrated to give a residue (10.0 g) that was subjected to column chromatography on Silica gel with petroleum ether–ethyl acetate system, affording 8 fractions. Fraction 5 gave **1** (40 mg) and fraction 7 gave **2** (132 mg).

Musaxanthone (1): Yellow light needles, m.p. 141.0–143.0°C, IR ν_{\max} 3068, 2959, 2927, 2870, 1656, 1590, 1444, 1295 and 1122 cm $^{-1}$. EIMS m/z 352 [M] $^{+}$, 337, 322, 294, 168. HREIMS [M] $^{+}$ m/z 352.1020 (Calcd 352.1025) for C $_{20}$ H $_{16}$ O $_6$. 1 H- and 13 C-NMR assignments are shown in table 1.

Asmaxanthone (2): Yellow needles, m.p. 173.0–175.0°C, IR ν_{\max} 2968, 2843, 2676, 1610, 1657, 1568, 1292 and 1156 cm $^{-1}$. EIMS m/z 384 [M] $^{+}$, 369, 339, 184, 177, 163. HREIMS [M] $^{+}$ m/z 384.1281 (Calcd 384.1287) for C $_{21}$ H $_{20}$ O $_7$. 1 H- and 13 C-NMR assignments are presented in table 1.

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