



## Friedolanostanes and xanthenes from the twigs of *Garcinia hombroniana*

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### ABSTRACT

The twigs of *Garcinia hombroniana* yielded six compounds: two 17,14-friedolanostanes (garcihombronanes K–L, **1–2**) and four xanthenes (garcihombronones A–D, **3–6**) together with 14 known compounds including four friedolanostanes, one lanostane, six xanthenes, two benzoic acid derivatives and one biflavonoid. Their structures were elucidated by analysis of spectroscopic data and comparison of the NMR data with those reported previously. Their antibacterial activity against methicillin-resistant *Staphylococcus aureus* and *S. aureus* was evaluated.

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## 1. Introduction

*Garcinia hombroniana*, belonging to the Guttiferae family, is widely distributed in the southern part of Thailand. Its local name is “Waa”. Plants in the genus *Garcinia* are a rich source of prenylated xanthenes (Rukachaisirikul et al., 2000a, 2005a; Zhang et al., 2010; Zhou et al., 2011). So far, lanostanes and friedolanostanes were isolated from three *Garcinia* species, *Garcinia benthami* (Nguyen et al., 2011), *G. hombroniana* (Rukachaisirikul et al., 2000b, 2005b) and *Garcinia speciosa* (Vieira et al., 2004a,b, 2005). Some of these compounds exhibited interesting biological activities, such as antibacterial (Rukachaisirikul et al., 2003, 2005a; Sukpondma et al., 2005b), apoptotic (Vieira et al., 2004b) and antioxidant (Lannang et al., 2005) properties. In a previous investigation on *Garcinia* plants, this resulted in the isolation of antibacterial prenylated xanthenes against methicillin-resistant *Staphylococcus aureus* (Rukachaisirikul et al., 2003, 2005a). In continuation of the studies on the antibacterial substances from *Garcinia* plants, it was established that a crude methanol extract from the twigs of *G. hombroniana* showed antibacterial activity. This paper deals with the isolation and structural elucidation of six new compounds, two friedolanostanes and four xanthenes, together with 14 known compounds and their antibacterial activity against methicillin-resistant *S. aureus* and *S. aureus*.

## 2. Results and discussion

The twigs of *G. hombroniana* were extracted with MeOH and the resulting MeOH extract was then subjected to various chromatographic purification procedures to obtain six new compounds: two 17,14-friedolanostanes (garcihombronanes K–L, **1–2**), four xanthenes (garcihombronones A–D, **3–6**) along with 14 known compounds: (2Z,24E)-3β,9α-dihydroxy-17,14-friedolanosta-14,22,24-trien-26-oic acid (**7**) (Nguyen et al., 2011), garcihombronane F (Rukachaisirikul et al., 2005b), garcihombronane B (**8**) (Rukachaisirikul et al., 2000b), garcihombronanes C and D (Rukachaisirikul et al., 2000b), norathyriol (Rukachaisirikul et al., 2006), gentisein (Zhong et al., 2008), 1,3,6,7-tetrahydroxy-8-prenylxanthone (Ishiguro et al., 1995), cheffouxanthone (**9**) (Lannang et al., 2006), bangangxanthone A (**10**) (Lannang et al., 2005), toxyloxanthone B (Iinuma et al., 1996), vokensiflavone (Sukpondma et al., 2005a), 4-hydroxybenzoic acid (Cho et al., 1998) and protocatechuic acid methyl ester (Chin et al., 2008). Their structures were determined by analysis of 1D and 2D NMR spectroscopic data and comparison of the NMR data with those reported in the literature. Compound **1** was isolated as a monoacetate derivative due to the difficulty in its isolation (Fig. 1).

The 3-acetoxy derivative of garcihombronane K (**1**) was obtained as a colorless gum. The monoacetate (**1a**) gave a molecular formula C<sub>32</sub>H<sub>50</sub>O<sub>5</sub> (*m/z* 514.3651) by HREIMS. Its IR spectrum indicated the presence of a hydroxyl group (3434 cm<sup>-1</sup>), a carbonyl group of an α,β-unsaturated carboxylic acid (1669 cm<sup>-1</sup>) and a saturated ester carbonyl group (1714 cm<sup>-1</sup>). The UV spectrum displayed an absorption band at λ<sub>max</sub> 264 nm, indicating that **1a**

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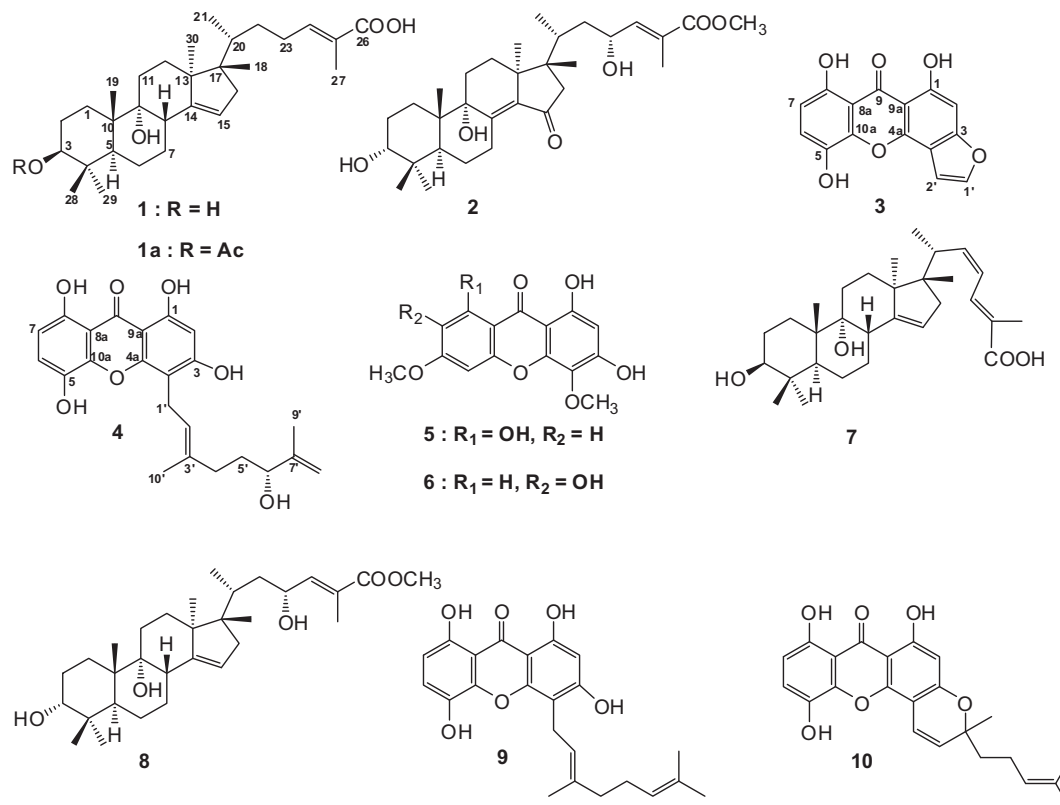


Fig. 1. Structures of compounds 1–10.

possessed an  $\alpha,\beta$ -unsaturated carboxylic acid chromophore (Rukachaisirikul et al., 2005b). The  $^1\text{H}$  NMR spectrum was similar to that of **7** except for the absence of *trans*-olefinic protons of the side-chain ( $\delta_{\text{H}}$  5.98 and 6.22). In addition, two sets of nonequivalent methylene protons ( $\delta_{\text{H}}$  2.18/1.10 and 2.04/1.71) were observed. These results indicated that **1a** contained a  $-\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_2\text{CH}=\text{C}(\text{CH}_3)\text{COOH}$  side-chain which was confirmed by analysis of  $^1\text{H}-^1\text{H}$  COSY and HMBC data (Fig. 2). In the  $^1\text{H}-^1\text{H}$  COSY spectrum, the methine H-20 ( $\delta_{\text{H}}$  1.92) showed correlations with H<sub>3</sub>-21 ( $\delta_{\text{H}}$  0.85) and H<sub>2</sub>-22 ( $\delta_{\text{H}}$  2.18 and 1.10) which was further coupled with H<sub>2</sub>-23 ( $\delta_{\text{H}}$  2.04 and 1.71). In addition, H<sub>2</sub>-23 correlated with an olefinic H-24 ( $\delta_{\text{H}}$  6.90). The olefinic H-24 showed HMBC correlations with C-27 ( $\delta_{\text{C}}$  12.0) and a carbonyl carbon of a carboxylic group ( $\delta_{\text{C}}$  171.2). In the NOESY spectrum, H<sub>3</sub>-27 ( $\delta_{\text{H}}$  1.85) gave a cross-peak with H<sub>2</sub>-23, indicating an *E*-configuration of the C-24/C-25 double bond in the side-chain. According to the NOESY data (Fig. 2), the relative configurations of the tetracyclic ring in **1a** were identical to those of **7**. Thus, **1a** was assigned as the 3 $\beta$ -acetoxy derivative of (24*E*)-3 $\beta$ -hydroxy-9 $\alpha$ -hydroxy-17,14-friedolanosta-14,24-dien-26-oic acid (**1**).

Garcihombrone L (**2**), a colorless gum, had the molecular formula  $\text{C}_{31}\text{H}_{48}\text{O}_6$  (*m/z* 516.3442) by HREIMS. Analysis of the IR

spectrum indicated the presence of a hydroxyl group ( $3443\text{ cm}^{-1}$ ), a carbonyl group of an  $\alpha,\beta$ -unsaturated ester ( $1698\text{ cm}^{-1}$ ) and a carbonyl group of a ketone ( $1742\text{ cm}^{-1}$ ). The  $^{13}\text{C}$  NMR spectrum showed two carbonyl carbons at  $\delta_{\text{C}}$  168.3 and 207.8, supporting the presence of  $\alpha,\beta$ -unsaturated ester and  $\alpha,\beta$ -unsaturated ketone functionalities (Vieira et al., 2004a). The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopic data were similar to those of **8**. A significant difference in the  $^1\text{H}$  NMR spectrum was the lack of a signal for an olefinic proton in the tetracyclic system in **2**. Moreover, the  $^{13}\text{C}$  NMR spectrum displayed carbon resonances of a tetrasubstituted  $\alpha,\beta$ -unsaturated ketone at  $\delta_{\text{C}}$  207.8, 151.6 and 140.2. The location of this moiety was established by analysis of HMBC data (Fig. 3). The methylene protons (H<sub>2</sub>-16,  $\delta_{\text{H}}$  2.39 and 2.09) showed HMBC correlations with C-14 ( $\delta_{\text{C}}$  140.2) and C-15 ( $\delta_{\text{C}}$  207.8), while the methylene protons (H<sub>2</sub>-7,  $\delta_{\text{H}}$  4.09 and 2.20) gave cross-peaks with C-8 ( $\delta_{\text{C}}$  151.6) and C-14. Consequently, the location of the trisubstituted double bond and carbonyl group was assigned at C-8/C-14 and C-15, respectively. In addition, one of H<sub>2</sub>-7 appeared at  $\delta_{\text{H}}$  4.09, supporting this assignment (Vieira et al., 2004a). The relative configurations of **2** were identical to those of **8** on the basis of identical NOEDIFF data (Fig. 3). The absolute configuration at C-23 was proposed to be *R* on the basis of the coupling constants involving

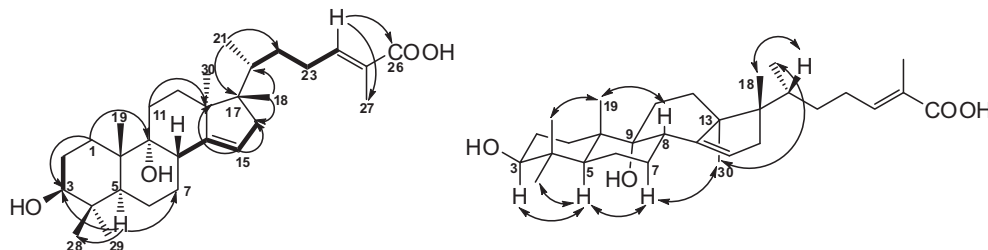


Fig. 2. Selected  $^1\text{H}-^1\text{H}$  COSY (bold), HMBC (H  $\rightarrow$  C) and NOESY (H  $\leftrightarrow$  H) correlations of garcihombrone K (**1**).

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