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Phytochemistry 63 (2003) 467–470

PHYTOCHEMISTRY

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Xanthones from the bark of *Garcinia merguensis*

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Received 14 June 2002; received in revised form 28 August 2002

Abstract

The bark of *Garcinia merguensis* yielded 10 xanthones, merguenone, 1,5-dihydroxy-6′-methyl-6′-(4-methyl-3-pentenyl)-pyrano(2′,3′:3,2)-xanthone, subelliptenone H, 8-deoxygartanin, rheediaxanthone A, morusignin G, 6-deoxyjacareubin, 1,3,5-trihydroxy-4,8-di(3-methylbut-2-enyl)-xanthone, rheediachromenoxanthone and 6-deoxyisojacareubin. The structure of merguenone was determined using spectroscopic techniques, mainly 1D and 2D NMR spectroscopy.

Keywords: *Garcinia merguensis*; Guttiferae; Merguenone; Xanthones

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1. Introduction

Garcinia merguensis Wight is a medium size tree found in Indo-China, Thailand, Myanmar and Indonesia. The fruits are edible and the bark is used as a yellow dye while the leaves are used in folk medicine for the treatment of oedemas (Vo, 1997). Although there have been phytochemical studies on more than 50 *Garcinia* species, no investigation on *G. merguensis* has been reported. We have now chemically examined the bark of this species collected in Vietnam.

2. Results and discussion

Extensive column chromatography of a petroleum ether extract of the bark of *G. merguensis* led to the isolation of a new xanthone, merguenone (**1**), and nine known xanthones, 1,5-dihydroxy-6′-methyl-6′-(4-methyl-3-pentenyl)-pyrano(2′,3′:3,2)-xanthone (Cruz et al., 2001), subelliptenone H (Iinuma et al., 1995), 8-deoxygartanin (**2**) (Govindachari et al., 1971), rheediaxanthone A (**3**) (Delle Monache et al., 1981), morusignin G (Hano et al., 1991), 6-deoxyjacareubin (Gottlieb et al., 1968), 1,3,5-trihydroxy-4,8-di(3-methylbut-2-enyl)-xanthone (**4**) (Waterman and Hussain, 1982), rheediachromenoxanthone (Delle Monache et al., 1983) and 6-deoxyisojacareubin (Owen and Scheinmann, 1974).

Merguenone (**1**), $C_{23}H_{22}O_5$, was obtained as yellow needles, mp 194–195 °C. The compound gave a dark green colour with methanolic ferric chloride, indicating that it was phenolic. The UV spectrum revealed maxima at 252, 266, 338 and 378 nm and the IR exhibited absorption bands at ν_{max} 3443 (O–H), 1650 (chelated C=O), 1580 (aromatic ring) cm^{-1}. The ^1H and ^{13}C NMR spectra of **1** contained resonances for one chelated hydroxyl group [δ_H 13.34 (1H, *s*, exchangeable with D_2O, 1-OH)] and the corresponding chelated carbonyl carbon [δ_C 183.3 (*s*, C-9)], one free hydroxyl group [δ_H 5.56 (1H, *br s*, exchangeable with D_2O, 5-OH)], two *ortho* coupling protons [δ_H 7.21 (1H, *d*, $J = 8.3$ Hz, H-6) and 7.04 (1H, *d*, $J = 8.3$ Hz, H-7); δ_C 125.7 (*d*, C-7) and 120.1 (*d*, C-6)], one isolated aromatic proton [δ_H 6.25 (1H, *s*, H-2); δ_C 100.1 (*d*, C-2)], one trisubstituted double bond [δ_H 5.35 (1H, *t sept*, $J = 7.0$ and 1.2 Hz, H-17); δ_C 133.1 (*s*, C-18) and 123.3. (*d*, C-17)], one benzylic methylene group [δ_H 3.97 (2H, *d*, $J = 7.0$ Hz, H_2-16); δ_C 33.4 (t, C-16)], two vinyl methyls [δ_H 1.74 (6H, *br s*, H_3-19 and H_3-20); δ_C 26.3 (*q*, C-19) and 18.4

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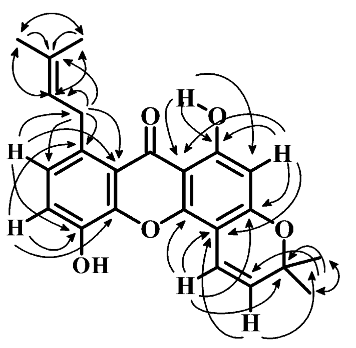
0031-9422/03/$ - see front matter © 2003 Published by Elsevier Science Ltd.
PII: S0031-9422(02)00433-8

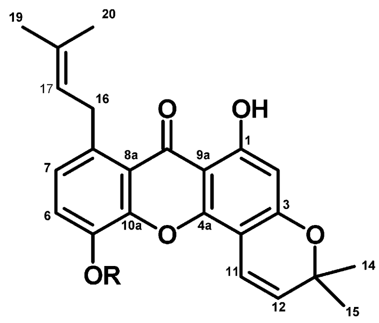
(*q*, C-20)], a *cis* olefinic group [δ_H 6.72 (1H, *d*, J=9.8 Hz, H-11) and 5.62 (1H, *d*, J=9.8 Hz, H-12); δ_C 127.9 (*d*, C-12) and 115.0 (*d*, C-11)] and two tertiary methyls attached to an oxygenated carbon [δ_H 1.48 (6H, *s*, H_3-14 and H_3-15), δ_C 78.5 (*s*, C-13) and 28.6 (*q* × 2, C-14 and C-15)] as well as nine substituted aromatic carbons [δ_C 164.0 (*s*, C-1), 160.9 (*s*, C-3), 150.6 (*s*, C-4a), 145.6 (*s*, C-10a), 142.9 (*s*, C-5), 136.2 (*s*, C-8), 118.9 (*s*, C-8a), 104.6 (*s*, C-9a) and 100.8 (*s*, C-4)], five of which were oxygenated. These data suggested a dihydroxyxanthone with a 3-methylbut-2-enyl group and a 2,2-dimethylpyran ring. Methylation of **1** with CH_3I and K_2CO_3 in acetone furnished a monomethyl ether (**5**) [δ_H 3.95 (3H, *s*, 5-OMe)] which still retained the chelated hydroxyl group [δ_H 13.29 (1H, *s*, 1-OH)], confirming the presence of one chelated and one free hydroxyl group in the parent compound.

HMQC and HMBC spectroscopy (see Fig. 1) as well as COSY and NOE experiments were used to determine the structure and the ^1H and ^{13}C NMR spectroscopic assignments for **1**. The chelated hydroxyl proton correlated to one oxygenated carbon [δ_C 164.0], a protonated aromatic carbon [δ_C 100.1] and a substituted aromatic carbon [δ_C 104.6] which had to be C-1, C-2 and C-9a, respectively. The aromatic proton [H-2] correlated to C-1, C-9a, an oxygenated aromatic carbon [δ_C 160.9] and a substituted aromatic carbon [δ_C 100.8] while the more deshielded *cis* olefinic proton [H-11] gave cross-peaks to two oxygenated aromatic carbons [δ_C 160.9 and 150.6], revealing that C-3 was oxygenated and the 2,2-dimethylpyran ring was angular to the xanthone A ring. Further evidence came from NOE experiment in which irradiation of H-2 gave rise to no enhancement of H-11.

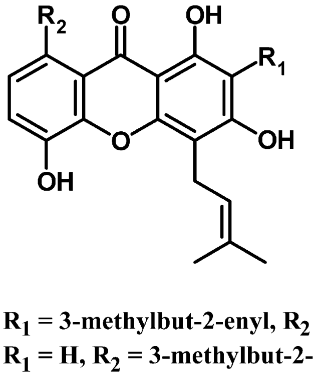
The xanthone B ring therefore carried the 3-methylbut-2-enyl group and the free hydroxyl group. The presence of the 3-methylbut-2-enyl group was confirmed by the COSY spectrum which showed correlations between H-16 and H-17 as well as H-17 and H_3-19 and H_3-20. The chemical shift [δ_H 3.99] of the benzylic methylene protons [H_2-16] revealed that the group was attached to the *peri* position [C-8]. These protons correlated to an unsubstituted aromatic carbon [δ_C 125.7] and to two substituted aromatic carbons [δ_C 136.2 and 118.9]. The protonated carbon had to be C-7. The *ortho* coupling partner of H-7 was H-6 and the hydroxyl group was therefore located at C-5. H-6 gave crosspeaks to the substituted carbon at δ_C 136.2 and to two oxygenated carbons [δ_C 142.9 and 145.6]. The former carbon had to be C-8 and the signal due to C-8a was then identified [δ_C 118.9]. H-7 correlated with one of the two oxygenated carbons [δ_C 142.9] which had to be C-5 and the remaining carbon was therefore assigned to C-10a. Structure **1**



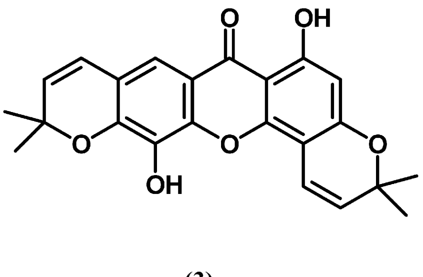
HMBC correlations for (1)



(1) R = H
(5) R = Me



(2) R$_1$ = 3-methylbut-2-enyl, R$_2$ = H
(4) R$_1$ = H, R$_2$ = 3-methylbut-2-enyl



(3)

Fig. 1. HMBC correlations for (**1**).

was then unambiguously established for merguenone {6,11-dihydroxy-3,3-dimethyl-8-(3-methyl-2-butenyl)-3H,7H-pyrano[2,3-c]xanthen-7-one}, which is a new natural product. The observed NOE enhancements (see Section 3) were in agreement with the structure.

The ^{13}C NMR spectra of 8-deoxygartanin (**2**) and 1,3,5-trihydroxy-4,8-di(3-methylbut-2-enyl)-xanthone (**4**) are reported here for the first time. The ^{1}H and ^{13}C NMR assignments for rheediaxanthone A (**3**) are reported to correct previous literature assignments.

3. Experimental

3.1. General conditions

Mps: uncorr. UV: EtOH. IR: KBr. NMR: 400 MHz (^{1}H) and 100 MHz (^{13}C) in $\mathrm{CDCl_3}$ (unless otherwise specified) relative to TMS at $\delta = 0.00$. ^{13}C Multiplicities were determined using the DEPT pulse sequence. CC: silica gel (Merck, 40 μm) or DIOL (Lichrosorb 40–63 μm). GPC: Sephadex LH-20 ($\mathrm{CHCl_3}$–MeOH, 1:1 as eluent). Petroleum ether refers to the fraction of bp 50–85 °C.

3.2. Plant material

The bark of *Garcinia merguensis* was collected in June, 2000 at Saigon Botanical and Zoological Garden. A voucher specimen (GM-HCMC) is retained in the Chemistry Department, College of Natural Sciences, National University of Ho Chi Minh City.

3.3. Extraction and isolation

The air-dried and ground plant material (1 kg) was subjected to exhaustive extraction using hot petroleum ether and concentrated in vacuo to afford a crude petroleum ether extract (65.0 g). The crude extract was fractionated over a column of silica gel with an EtOAc–petroleum ether gradient to give nine frs (1–9).

The main fr 8 (10.6 g) was subjected to flash CC (silica gel, EtOAc–petroleum ether gradient) to furnish 6 frs (8.1–8.6). Fr 8.1 (1.1 g) afforded *merguenone* (**1**) (16 mg) and 1,5-dihydroxy-6′-methyl-6′-(4-methyl-3-pentenyl)-pyrano(2′,3′:3,2)-xanthone (7 mg) after flash CC (silica gel, 20–60% $\mathrm{C_6H_6}$-petroleum ether followed by 5–20% $\mathrm{Me_2CO}$–petroleum ether) and GPC. The $\mathrm{Me_2CO}$ soluble portion (700 mg) of fr 8.2 (800 mg) was subjected to GPC and then purified by flash CC (silica gel, 5–30% $\mathrm{CHCl_3}$-petroleum ether followed by 5–20% $\mathrm{Me_2CO}$-petroleum ether) to give subelliptenone H (3.5 mg). Flash CC (silica gel, 40–90% $\mathrm{C_6H_6}$–petroleum ether) of fr 8.3 (2.3 g) gave 5 frs (8.3.1–8.3.5). Flash CC of 8.3.3 (40 mg) (silica gel, 5–20% $\mathrm{Me_2CO}$–petroleum ether) afforded 8-deoxygartanin (**3**) (8 mg). GPC of fr 8.3.5 (1.3 g) furnished two frs. Flash CC (silica gel, 1–15% $\mathrm{Me_2CO}$–$\mathrm{C_6H_6}$) of the first fr (70 mg) afforded rheediaxanthone A (**3**) (8 mg) and morusignin G (9 mg). Flash CC (silica gel, 8–30% EtOAc–petroleum ether) of the second fr (30 mg) afforded 6-deoxyjacareubin (7 mg). The $\mathrm{Me_2CO}$ soluble portion (1.2 g) of fr 8.5 (1.5 g) was subjected to repeated flash CC (silica gel, 60–80% $\mathrm{Me_2CO}$–petroleum ether and DIOL, 60–80% $\mathrm{CHCl_3}$–$\mathrm{C_6H_6}$) to give 1,3,5-trihydroxy-4,8-di(3-methylbut-2-enyl)-xanthone (**4**) (30 mg). Fr 8.6 (2.9 g) was fractionated using GPC followed by flash CC (silica gel, 5–30% EtOAc–petroleum ether and 3–5% $\mathrm{Me_2CO}$–$\mathrm{C_6H_6}$) to furnish rheediachromenoxanthone (8 mg) and 6-deoxyisojacareubin (7 mg).

3.3.1. Merguenone (**1**)

Yellow needles, mp 194–195 °C ($\mathrm{CHCl_3}$–petroleum ether); HREI-MS *m/z* 378.1465 ($\mathrm{C_{23}H_{22}O_5}$ requires *m/z* 378.1467); UV $\lambda_{\max}$ nm: 252, 266, 338, and 378; IR $\nu_{\max}$ cm^{-1}: 3443, 2919, 1650, 1580, 1492, 1280, 1144, 822; ^{1}H NMR: 13.34 (1H, *s*, 1-OH), 7.21 (1H, *d*, $J = 8.3$ Hz, H-6), 7.04 (1H, *d*, $J = 8.3$ Hz, H-7), 6.72 (1H, *d*, $J = 9.8$ Hz, H-11), 6.25 (1H, *s*, H-2), 5.62 (1H, *d*, $J = 9.8$ Hz, H-12), 5.56 (1H, *br s*, 5-OH), 5.35 (1H, *t sept*, $J = 7.0$ and 1.2 Hz, H-17), 3.97 (1H, *d*, $J = 7.0$ Hz, $\mathrm{H_2}$-16), 1.74 (6H, *s*, $\mathrm{H_3}$-19 and $\mathrm{H_3}$-20) and 1.48 (6H, *s*, $\mathrm{H_3}$-14 and $\mathrm{H_3}$-15); ^{13}C NMR: 183.3 (*s*, C-9), 164.0 (*s*, C-1), 160.9 (*s*, C-3), 150.6 (*s*, C-4a), 145.6 (*s*, C-10a), 142.9 (*s*, C-5), 136.2 (*s*, C-8), 133.1 (*s*, C-18), 127.9 (*d*, C-12), 125.7 (*d*, C-7), 123.3 (*d*, C-17), 120.1 (*d*, C-6), 118.9 (*s*, C-8a), 115.0 (*d*, C-11), 104.6 (*s*, C-9a), 100.8 (*s*, C-4), 100.1 (*d*, C-2), 78.5 (*s*, C-13), 33.4 (*t*, C-16), 28.6 (*q* × 2, C-14 and C-15), 26.3 (*q*, C-19) and 18.4 (*q*, C-20); HMBC: H-2 [C-1, C-3, C-4, C-9a], H-6 [C-5, C-8, C-10a], H-7 [C-5, C-8a, C-16], H-11 [C-3, C-4, C-4a, C-13], H-12 [C-4, C-14, C-15], $\mathrm{H_3}$-14 [C-12, C-13, C-15], $\mathrm{H_3}$-15 [C-12, C-13, C-14], $\mathrm{H_2}$-16 [C-7, C-8, C-8a, C-17, C-18], H-17 [C-19, C-20], $\mathrm{H_3}$-19 [C-17, C-18, C-20], $\mathrm{H_3}$-20 [C-17, C-18, C-19] and 1-OH [C-1, C-2, C-9a]; NOE: H-2 [no enhancement], H-6 [H-7], H-7 [H-6, $\mathrm{H_2}$-16, H-17], $\mathrm{H_2}$-16 [H-7, H-17].

3.3.2. Methylation of merguenone (**1**)

Treatment of **1** (10 mg) with $\mathrm{CH_3I}$ (1 ml) and $\mathrm{K_2CO_3}$ (20 mg) in $\mathrm{Me_2CO}$ at 40 °C for 3 h followed by purification using flash CC (silica gel, 1–4% $\mathrm{Me_2CO}$–petroleum ether) furnished *5-O-methylmerguenone* (**5**) (9.5 mg) as dark yellow needles from $\mathrm{CH_2Cl_2}$–petroleum ether, mp 120–121 °C; UV $\lambda_{\max}$ nm: 252, 266, 312, 336 and 378; ^{1}H NMR: 13.29 (1H, *s*, 1-OH), 7.08 (1H, *d*, $J = 8.4$ Hz, H-6), 7.00 (1H, *d*, $J = 8.4$ Hz, H-7), 6.85 (1H, *d*, $J = 10.0$ Hz, H-11), 6.22 (1H, *d*, H-2), 5.58 (1H, *d*, $J = 10.0$ Hz, H-12), 5.37 (1H, *t sept*, $J = 6.9$ and 1.2 Hz, H-17), 3.96 (1H, *d*, $J = 6.9$ Hz, $\mathrm{H_2}$-16), 3.95 (3H, *s*, 5-OMe), 1.74 (6H, *s*, $\mathrm{H_3}$-19 and $\mathrm{H_3}$-20) and 1.48 (6H, *s*, $\mathrm{H_3}$-14 and $\mathrm{H_3}$-15).

3.3.3. 8-Deoxygartanin (**2**)

^{1}H NMR (Me$_2$CO-d_6): 13.33 (1H, *s*, 1-OH), 7.70 (1H, *dd*, J = 7.9 and 1.6 Hz, H-8), 7.38 (1H, *dd*, J = 7.9 and 1.6 Hz, H-6), 7.26 (1H, t, J = 7.9 Hz, H-7), 5.31 and 5.25 (each 1H, *t sept*, J = 7.0 and 1.3 Hz, H-12 and H-17), 3.69 (2H, *s*, J = 7.0 Hz, H$_2$-16), 3.46 (2H, *s*, J = 7.0 Hz, H$_2$-11), 1.87 and 1.80 (each 3H, *d*, J = 1.3 Hz, H$_3$-15 and H$_3$-20), 1.67 and 1.66 (each 3H, J = 1.3 Hz, H$_3$-14 and H$_3$-19); ^{13}C NMR (Me$_2$CO-d_6): 182.5 (*s*, C-9), 162.0 (*s*, C-3), 159.8 (*s*, C-1), 153.9 (*s*, C-4a), 147.6 (*s*, C-5), 146.8 (*s*, C-10a), 133.0 and 132.9 (each *s*, C-13 and C-18), 124.9 (*d*, C-7), 123.6 and 123.4 (each *s*, C-12 and C-17), 122.5 (*s*, C-8a), 121.6 (*d*, C-6), 116.6 (*d*, C-8), 111.6 (*s*, C-2), 107.8 (*s*, C-4), 104.2 (*s*, C-9a), 26.34 and 26.26 (each *q*, C-14 and C-19), 22.8 and 22.5 (each *t*, C-11 and C-16), 18.5 and 18.4 (each *q*, C-14, C-15, C-19 and C-20); HMBC: H-6 [C-8, C-10a], H-7 [C-5, C-8a], H-8 [C-6, C-9, C-10a], H$_2$-11 [C-1, C-2, C-3, C-12, C-13], H$_3$-14 [C-12, C-13, C-15], H$_3$-15 [C-12, C-13, C-14], H$_2$-16 [C-3, C-4, C-4a, C-17, C-18], H$_3$-19 [C-17, C-18, C-20], H$_3$-20 [C-17, C-18, C-19] and 1-OH [C-1, C-2, C-9a].

3.3.4. Rheediaxanthone A (**3**)

^{1}H NMR (Me$_2$CO-d_6): 13.23 (1H, *s*, 1-OH), 7.44 (*s*, H-8), 6.93 (1H, *dd*, J = 10.0 and 0.5 Hz, H-11), 6.60 (1H, *d*, J = 10.0 Hz, H-16), 6.16 (1H, *d*, J = 0.5 Hz, H-2), 5.92 (1H, *d*, J = 10.0 Hz, H-17), 5.77 (1H, *d*, J = 10.0 Hz, H-12), 1.51 (6H, *s*, H$_3$-19 and H$_3$-20) and 1.49 (6H, *s*, H$_3$-14 and H$_3$-15); ^{13}C NMR (Me$_2$CO-d_6): 181.6 (*s*, C-9), 164.5 (*s*, C-1), 161.6 (*s*, C-3), 152.8 (*s*, C-4a), 147.1 and 147.0 (each *s*, C-6 and C-10a), 134.9 (*s*, C-5), 133.0 (*d*, C-17), 128.6 (*d*, C-12), 122.4 (*d*, C-16), 119.9 (*s*, C-8a), 116.0 (*d*, C-11), 115.7 (*s*, C-7), 113.7 (*d*, C-8), 104.1 (*s*, C-9a), 102.5 (*s*, C-4), 99.9 (*d*, C-2), 79.43 and 79.35 (each *s*, C-13 and C-18), 28.8 and 28.7 (each *q*, C-14 and C-15); HMBC: H-2 [C-1, C-3, C-9a], H-8 [C-6, C-8a, C-9, C-10a], H-11 [C-3, C-4a, C-13], H-12 [C-4, C-13], H$_3$-14 [C-12, C-13, C-15], H$_3$-15 [C-12, C-13, C-14], H-16 [C-6, C-7, C-8, C-18], H-17 [C-7, C-18], H$_3$-19 [C-17, C-18, C-20], H$_3$-20 [C-17, C-18, C-19] and 1-OH [C-1, C-2, C-9a].

3.3.5. 1,3,5-Trihydroxy-4,8-di(3-methylbut-2-enyl) xanthone (**4**)

^{1}H NMR (Me$_2$CO-d_6): 13.27 (1H, *s*, 1-OH), 7.27 (1H, *s*, J = 7.2 Hz, H-6), 7.04 (1H, *s*, J = 7.2 Hz, H-7), 6.34 (*s*, H-2), 5.39 (2H, *m*, H-12 and H-17), 4.00 (2H, *d*, J = 7.2 Hz, H$_2$-16), 3.58 (2H, *d*, J = 7.3 Hz, H$_2$-11), 1.84 (3H, *br s*, H$_3$-15), 1.74 (3H, *br s*, H$_3$-20), 1.71 (3H, *d*, J = 1.0 Hz, H$_3$-19), 1.64 (3H, *d*, J = 1.0 Hz, H$_3$-14); ^{13}C NMR (Me$_2$CO-d_6): 184.7 (*s*, C-9), 164.0 (*s*, C-3), 162.9 (*s*, C-1), 155.3 (*s*, C-4a), 148.0 (*s*, C-10a), 145.7 (*s*, C-5), 135.3 (*s*, C-8), 132.6 and 132.1 (each *s*, C-13 and C-18), 126.2 (*d*, C-7), 125.0 (*d*, C-17), 124.0 (*d*, C-12), 121.2 (*d*, C-6), 119.8 (*s*, C-8a), 107.4 (*s*, C-4), 104.8 (*s*, C-9a), 98.9 (*d*, C-2), 34.0 (*t*, C-16), 26.3 (q × 2, C-14 and C-19), 22.5 (*t*, C-11), 18.41 and 18.38 (each *q*, C-15 and C-20); HMBC: H-2 [C-1, C-3, C-4, C-9a], H-6 [C-5, C-8, C-10a], H-7 [C-5, C-8a, C-16], H$_2$-11 [C-3, C-4, C-4a, C-12, C-13], H-12 [C-14, C-15], H$_3$-14 [C-12, C-13, C-15], H$_3$-15 [C-12, C-13, C-14], H$_2$-16 [C-7, C-8, C-8a, C-17, C-18], H-17 [C-19, C-20], H$_3$-19 [C-17, C-18, C-20], H$_3$-20 [C-17, C-18, C-19] and 1-OH [C-1, C-2, C-9a].

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