

## ECDYSTEROIDS AND IRIDOIDAL GLYCOSIDES FROM *Vitex agnus-castus*

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The isolation from plants of the *Vitex* genus of ecdysteroids and iridoids has been reported [1-6]. We studied leaves (2 kg) of *Vitex agnus-castus*. The air-dried ground leaves were extracted with ethanol.

The alcohol extracts were condensed, diluted with water (0.5 L), and treated successively with  $\text{CHCl}_3$  and ethylacetate ( $6 \times 500$  mL) and then butanol ( $4 \times 300$  mL). The ethylacetate fraction after removal of solvent was chromatographed over a column of  $\text{Al}_2\text{O}_3$  by  $\text{CHCl}_3:\text{CH}_3\text{OH}$  (9:1) to afford compounds **1** and **2**.

**Compound 1**,  $\text{C}_{29}\text{H}_{46}\text{O}_8$ , mp 196-198°C (acetone),  $[\alpha]_{\text{D}}^{22} +59.8^\circ$  (methanol). The IR spectrum of **1** contains absorption bands ( $\text{cm}^{-1}$ ) at 3430 (OH), 1670 (7-en-6-ketone), and 1730 and 1275 (ester).

The PMR spectrum of **1** exhibits a signal for the acetyl methyl at 1.79 ppm.

Comparison of the PMR, IR, and mass spectra with those in the literature and the  $R_f$  value on TLC with an authentic sample identified **1** as viticosterone E [1, 2].

**Compound 2**,  $\text{C}_{27}\text{H}_{44}\text{O}_6$ , mp 241-242°C (acetone),  $[\alpha]_{\text{D}}^{22} +63.2^\circ$  (methanol).

The IR spectrum of **2** contains absorption bands ( $\text{cm}^{-1}$ ) characteristic of ecdysteroids at 3470 (OH) and 1665 (7-en-6-ketone). The UV spectrum of **2** has a maximum at 245 nm ( $\log \epsilon$  4.15).

The mass spectrum of **2** exhibits peaks for ions with  $m/z$  462 [M -  $\text{H}_2\text{O}$ ], 444, 426, 408, 363, 345, 327, 309, 300, 99, 81, and 69, the mass numbers of which coincide with fragments of ecdysterone [7].

The physicochemical constants and spectral properties of **2** indicate that it is identical to ecdysterone [8-10].

Further elution of the column with  $\text{CHCl}_3:\text{CH}_3\text{OH}:\text{H}_2\text{O}$  (4:1:0.1) produced compounds **3** and **4**.

**Compound 3**,  $\text{C}_{17}\text{H}_{26}\text{O}_{11}$ , mp 154-156°C,  $[\alpha]_{\text{D}}^{20} -131^\circ$  (methanol). The IR spectrum of **3** showed absorption bands ( $\text{cm}^{-1}$ ) at 3400-3500 (OH), 1650 (C-3—C-4 double bond), and 1710 (ester).

The physicochemical constants and spectral (PMR and  $^{13}\text{C}$  NMR) properties of **3** indicate that it is identical to 8-O-acetylharpagide [5].

**Compound 4**, amorphous,  $[\alpha]_{\text{D}}^{20} -154^\circ$ . The IR spectrum contains absorption bands ( $\text{cm}^{-1}$ ) at 3300-3500 (OH) and 1655.

Comparison of the constants and spectral (PMR and  $^{13}\text{C}$  NMR) properties with the literature [9, 10] identified it as harpagide [6].

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