A New Highly Oxygenated Pseudoguaianolide from a Collection of the Flowers of *Parthenium hysterophorus*\(^1\)

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A new highly oxygenated pseudoguaianolide, 8-\(\beta\)-acetoxyhysterone C, along with the known compounds, parthenin, coronopilin and hysterone C, has been isolated from a collection of the flowers of *Parthenium hysterophorus*. The structure of the new compound was derived from the extensive studies of its spectral (mainly 1D and 2D NMR) data.

**Key words** Parthenium hysterophorus; pseudoguaianolide; 8-\(\beta\)-acetoxyhysterone C

*Parthenium hysterophorus* Linn (Compositae), an obnoxious weed, grows wild in different regions of India. The plant is known to create contact dermatitis and allergic rhiinitis in animals\(^3\)\(^-\)\(^6\) and to possess significant allelopathic properties.\(^3,4\)

In continuation of our recent work\(^7,8\) on the constituents of different parts of *Parthenium hysterophorus* we report here the isolation of a new pseudoguaianolide, 8-\(\beta\)-acetoxyhysterone C (2) along with the known compounds, parthenin (1),\(^3\) coronopilin (3)\(^9\) and hysterone C (4)\(^9\) from a collection of the flowers of the plant (Fig. 1). The new compound (2) was isolated as a viscous mass. Its molecular formula was as derived from its 1H- and 13C-NMR values which clearly suggested the presence of 17 carbons in the molecule. The IR spectrum indicated\(^7\) the compound to be a pseudoguaianolide related to parthenin (1). In ring A, a C-2, C-3 double bond was present in 2 but the carbonyl group at C-4 was replaced by a hydroxyl (\(\delta\) 6.22, 1H, dd, J=6.0, 1.5 Hz, H-2; 5.94, 1H, dd, J=6.0, 2.4 Hz, H-3; 5.08, 1H, dd, J=2.4, 1.5 Hz, H-4). The DQF-COSY spectrum clearly showed a correlation between H-2 and H-3, H-3 and H-4 and H-2 and H-4. However, the double bond in ring C (that is, C-12, C-13 double bond) was saturated (\(\delta\) 2.34, 1H, m, H-12; 1.24, 3H, d, J=7.0 Hz, Me-13). The DQF-COSY spectrum showed a correlation between Me-13 and H-12, H-12 and H-7 (\(\delta\) 2.48, 1H, m) and H-7 and H-6 (\(\delta\) 5.21, 1H, d, J=8.6 Hz). The two methyl groups (Me-14, Me-15) appeared as singlets while Me-14 of the former is closely related to the \(\beta\)-configuration of the hydroxyl group at C-4. Me-14 and Me-15 were correlated but they were not related to Me-13 which was related to H-6, H-7 and H-8. These correlations suggested the \(\beta\)-orientation of Me-14 and Me-15 while \(\alpha\)-orientation of Me-13 and H-8. The structure of the new pseudoguaianolide was thus clearly settled as 8-\(\beta\)-acetoxyhysterone C (2).

The known compounds, parthenin (1),\(^3\) coronopilin (3)\(^9\) and hysterone C (4)\(^7\) isolated from the same plant part, were characterized by comparison of their physical (TLC, mp, and [\(\alpha\)\(_D\)]) and spectral (IR, 1H-NMR, MS) properties with those of authentic samples available in our laboratory. All the constituents were found (by TLC) to be present in the original

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extract of the plant materials.

**Experimental**

Melting points were measured in a Buchi-510 instrument and are uncorrected. The spectra were recorded with the following instruments: IR: Perkin Elmer spectrophotometer, $^1$H- and $^{13}$C-NMR: Varian Gemmini 200 MHz spectrometer and LSI-MS: Finnigan-MAT 1020 instrument. Optical rotations were determined with a Jasco DIP 360 digital polarimeter. Column chromatography was performed with silica gel (BDH, 100—200 mesh) and TLC with silica gel GF$_{254}$.

**Plant Materials** The flowers of Parthenium hysterophorus were collected from West Bengal in the month of July, 2003 and were botanically identified. A voucher specimen (IICT-5210) was preserved in the herbarium of Indian Institute of Chemical Technology.

**Extraction and Isolation** The air-dried and powdered plant materials (1 kg) was extracted with CHCl$_3$–MeOH (1 : 1, 3 l) at room temperature for 120 h. The extract was concentrated under reduced pressure to afford a gummy mass (18 g). The residue was subjected to column chromatography over silica gel using solvents of increasing polarity from $n$-hexane through EtOAc. The following compounds were obtained according to the increasing order of polarity: coronopilin (18 mg), parthenin (7.4 g), 8-$\beta$-acetoxyhysterone C (16 mg) and hysterone C (24 mg) (Fig. 1).

**8-$\beta$-Acetoxyhysterone C**

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<th>Chemical</th>
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| $\delta$ (CDCl$_3$) | 180.2 (C-11), 171.0 (–O–CO–Me), 138.2 (C-2), 132.5 (C-3), 87.3 (C-1), 85.1 (C-4), 82.2 (C-8), 81.9 (C-6), 77.4 (C-10), 57.4 (C-5), 46.7 (C-12), 42.5 (C-7), 36.2 (C-9), 25.0 (Me-14), 21.3 (–O–CO–Me), 15.3 (Me-15), 12.8 (Me-13); LSI-MS $m/z$: 341 (M$^+$/1); Anal. Calcd for C$_{17}$H$_{24}$O$_7$: C, 60.0; H, 7.06%. Found: C, 59.86; H, 7.12%.

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**References**