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Note

Chemical examination of *Artabotrys odoratissimus* (leaves)

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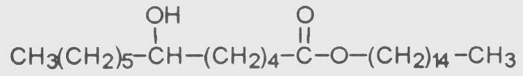
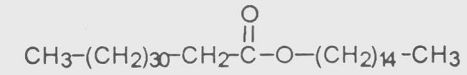
Chemical Investigation of *Artabotrys odoratissimus* leaves yield three novel compounds characterised as pentadecyl-7-hydroxy dodecanoate **1,** pentadecyl-tritriacontanoate **2** and 4,5-epoxy-26-ol-dopentacontane **3** , using IR ,PMR, CMR and Mass spectral data.

Extensive exploitation of *Artabotrys odoratissimus* in traditional medical treatment in Malwa regions of Madhya Pradesh (India) attracted our attention towards the intensive investigation of this plant for medicinal compounds. A decoction of the leaves is given in cholera in some of the islands of Malay Archipelago[1]. The antifertility activity of the plant has been confirmed in rats. Ethanol and benzene extracts of leaves of this plant has shown irregular oestrus cycle in albino rats [2] . Literature survey reveals that an alcohol, 24-methylene lanosta-7,9 (11) dien-3β-ol has been isolated from the pet. ether extract of the stem bark [3] and β-sitosterol from the methanol extract of the fruit pulp [4] .The essential oil obtained from the flowers is used in perfumery industries[5]. Herein we report the isolation and characterisation of three novel aliphatic oxygenated compounds from the hexane extract of leaves. The shade dried leaves were extracted from hexane and separated by column chromatography using alumina grade III to yield three novel compounds characterised as pentadecyl-7-hydroxy dodecanoate **1,** pentadecyl tritriacontanoate **2** and 4,5-epoxy-26-ol-dopentacontane **3.** The compounds were characterised by IR, PMR, CMR and mass spectral data.

Extraction and Isolation: Leaves of *A. odoratissimus* were collected from the near by area of Ujjain city, washed with clean water, air dried and ground to coarse powder.



1



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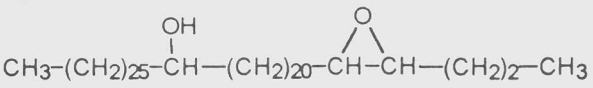

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Powdered leaves (8 kg) were exhaustively extracted in soxhlet by hexane. Removal of solvent by rotatory vacuum evaporator afforded (68 g) extract which was separated by column chromatography using alumina grade III as an adsorbent. The column was eluted by solvents of increasing polarity starting with n-hexane. The fractions were checked by TLC and those ones of similar compositions were combined and solvent was recovered from them. Compounds **1** and **2** were obtained from rechromatography of hexane eluate of hexane extract using hexane as solvent, while compound **3** was obtained from rechromatography of benzene eluate of hexane extract using hexane as solvent.

IR spectra were recorded in KBr on a Perkin Elmer-377 infracord spectrophotometer, PMR spectra (300 MHz) on Bruker AM-300 FT NMR spectrometer using TMS as internal standard, CMR spectra on Bruker AM-100 MHz FT NMR spectrometer and mass spectra on Jeol D 300 mass spectrometer. Silica gel G was used for TLC and spots were visualised by exposure to iodine.

Pentadecyl-7-hydroxy-dodecanoate **1** [$C_{27}H_{54}O_3$, M^+ 426], was recrystallised from methanol as a white crystalline solid , m.p. 72°C. Its IR spectrum KBr showed the presence of hydroxyl and ester groups (3400, 1740 cm^{-1}) and aliphatic nature of the molecule[6-7]. PMR spectrum (300 MHz, TMS, $CDCl_3$) showed peaks at δ 0.90 (t, 6H, 2-CH_3,

J=7Hz , due to two terminal methyl groups), 1.24 (br, s, 38H, 19-CH_2), 1.56 (s, H, –OH, D_2O exchangeable), 3.62 (t, H,-CH , *J*=7 Hz, due to methine proton attached to hydroxyl group),4.08 (t, 2H,–CH_2, *J*= 7Hz , due to methylene protons directly attached to ester group)[6], 2.3 (t, 2H, –CH_2, due to methylene protons to α-carbonyl group) and at 1.62 (m, 4H, –CH_2, due to methylene protons-β to ester group[8-11]).

The position of ester group was determined by its mass fragmentation pattern. In mass spectrum the separation of most of the peaks by 14 mass units and appearance of $C_nH_{2n}^+$, C_nH_{2n} and $C_nH_{2n}^{-1}$ ion series confirmed its long chain aliphatic nature[9,12]. Abundant fragments at m/z 270 and 156 formed by McLafferty rearrangement indicated the position of ester group at C-12 and formation of fragments at m/z 85, 341, 115 and 311 by α-cleavage indicated the position of hydroxyl group[12,13] at C-7, which confirmed the structure of the compound isolated as pentadecyl-7-hydroxy-dodeconoate. This is being reported for the first time by us.

Pentadecyl tritriacontanoate **2** ($C_{48}H_{96}O_2$, M^+ 704) was crystallised from benzene as white crystals, m. p. 78°C. Its IR spectrum showed it to be a long chain aliphatic ester. The position of ester group was determined from its mass fragmentation pattern.

Mass spectrum showed it to be a long chain aliphatic compound[9,12] . The α-cleavage gave fragment at m/z 494 indicating the position of carbonyl group at C-16. McLafferty rearrangement produced base beak at m/z 270 which confirmed the position of ester group[12,13]. The PMR spectrum (TMS, $CDCl_3$, 300 MHz) showed a triplet at δ 4.06 corresponding to methylene of ester group (CH_2OCO), another triplet at δ 2.3 for two protons was due to methylene linked to carbonyl group (CH_2CO). Six protons triplet at δ 0.89 was due to two terminal methyl groups. Rest of the methylene protons merged in to a single peak at δ 1.24. Thus the compound **2** was identified as pentadecyl tritriacontanoate [10,11].

4,5-Epoxy-26-ol-dopentacontane **3** ($C_{52}H_{104}O_2$, M^+ 760) was crystallised from methanol as white crystals, m.p. 69°C. Its IR spectrum showed it to be an aliphatic long chain compound (2920, 2250, 1460, 735-725 cm^{-1}), containing hydroxyl group (3400 cm^{-1}) and oxirane ring (3050-2990 cm^{-1}). the position of oxirane ring and hydroxyl groups was determined from its mass fragmentation pattern. The relatively abundant fragments at m/z 673, 717 and 395 formed by α-cleavage indicated the position of oxirane ring and hydroxyl group at C-4 and C-26 respectively.

The PMR spectrum (TMS, $CDCl_3$, 300 MHz) showed six proton triplet at δ 0.88 due to terminal methyl groups, the hydroxyl proton at δ 1.56 (D_2O exchangeable) and a triplet at δ 3.3 for one proton due to methine group attached to hydroxyl group. The proton of the methine group of oxirane ring appeared at δ 4.08 as a triplet. The methylene protons merged in to a single peak at δ 1.30. Thus the compound was identified as 4,5-epoxy-26-ol-dopentacontane.The above structure was further supported by CMR spectrum which was in consistent with the above structure. It displayed peaks at 64.36 and 34.41 ppm corresponding to methine attached to hydroxyl group and to oxirane ring respectively.The terminal methyl groups appeared at 14.06 ppm. The carbons α- and β- to the hydroxyl methine and epoxide methine resonated at 31.91, 29.34, 25.03, 29.15, and 28.67 ppm. The remaining methylenes merged in to a single peak at 29.68 ppm. Thus the compound **3** was characterised as 4,5 epoxy-26-ol-dopentacontane.

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