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RESEARCH ARTICLE

ISOLATIONS AND CHARACTERIZATION OF NEW COMPOUND FROM ROOTS OF CHLAROPHYTUM ARRUNDENACEUM

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Abstract

Two Novels compounds have been isolated and identified from the roots of Chlarophytum arrundenaceum. The compound have been characterized as 2,3- diaethyl -4-mehtyl-benzoic acid and 22,23 dihydro-7-stigmastene-3-β – D- glucopyanoacetate by speetral and chemical analysis.

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Introduction

Chlarophytum arrundenaceum belongs to family Liliaceae, commonly known as "Safed Musli", it is a herbaceous scrub. The tuberous roots are used as demulcent and galactogogue it is useful in impotency, general debility, diarrhoea and dysentery. It also exhibit antifungal activity. (Chopra et al., 1956, Iyngar et al., 1975, Dey et al., 1936). Its roots are used as a toxic after the birth of child and also during pregnancy (Ambasta et. al., 1986). It uses as a cultural practice of tissue cultured safed musli.

It is very rich source of proteins, steroids, saponins, carbohydrates and alkaloids. The key of root tubers has been provided. (Barathi et al., 2003). Roots extract shows adaptogenic activity, saponins and sugars present in methanol extract of Chlarophytum arrundenaceum. The sugers identified were arabinose, xylose and glucose by comparison with authentic materials Sapogenins provides sapogenin A-D (Gupta et al., 1979, Tondon et al., 1999). Its ethanol extract yielded four compounds. These are α—terpenyl acetate, β-terpenyl acetate, 4 methyl valeranol, 24- hydroxyl methyl cholest = 6, 22-en-3-ol. Here in we describe the isolation and structure elucidation of two new compounds from ethanol extract.

Result and Discussion:-

The shade dried powdered roots were extracted in shoxlet extraction serially with n- hexane, benzene and ethanol. The ethanol extract was subjected to column chromatography on silica gel. The column afforded is compound in pure form and other compounds failed to separate by repeated column chromatography. Hence it was acetylated. The acetylated fraction was then subjected to silica gel column chromatography to yield one compound in pure form. Their structures were established by spectroscopic and chemical means.

2, 3- diacetoxy-4-methyl benzoic acid (compound-1) –

The mass spectrum showed M^+ at 220, $C_{12}H_{12}O_4$ (50mg, chloroform: methanol01) its IR spectrum in KBR showed the presence of carboxylic acid, carbonyl group (3120cm^{-1}, 1735 cm^{-1}). The bands at 1610, 1590, 1480, 1430 cm-1 below 1000 indicated the presence of benzene ring in the molecule. The bands at 1300-1100 cm^{-1} due to c-c-c

stretching and bending of acetates. A band 1070-1020 cm-1 due to C-O stretching vibration of carboxylic group (Bellany et al., 1975). H^1 NMR spectrum (400 MHz, $CDCl_3$, TMS) showed a singlet at δ.2.25 for the methyl group attached an aromatic ring. The intense singlet for six protons at δ 3.1 was due to two $COCH_3$ groups attached to an aromatic ring. The two doublets at δ7.39 & 7.61 was assigned aromatic protons at C-5 & C-6. The singlet at δ 8.99 due to carboxylic proton (Silverstien et al., 1984, Kalsi et al., 1995). The abundant fragments at m/z 191,177, 134, 133, 119, 103, 77 were also in agreement with the proposed structure (Mclafferty et al., 1994). The ^{13}CNMR (BB, DEPT I, II) showed peaks at 128.8, 128.13, 128.40, 138.25, 144.49, 157.75 ppm corresponding to benzene ring. The carbonyl group resonated at 177.05 ppm. The methyl group resonated at 39.29, 40.1 and13.37 ppm while carboxylic group resonated at 184.0 ppm. Thus on the basis of the above evidences the compound 1 was characterized as 2,3-diacetoxy -4 methyl benzoic acid. It is a new compound being reported for the first time by us.

22, 23 –dihydro- 7- stigmastene -3- β-D- glucopyrano acetate –

The mass spectrum showed M^+ at 744, $C_{43}H_{68}O_{10}$ (45 mg, methanol) its IR spectrum & in KBr showed the presence of acetyl group at 1750 and 1220 cm-1 unsaturation was indicated by absorption at 1660 cm-1, 1380-70 cm-1 indicated the presence of an isopropyl group bands in the region 1140-1120 cm-1 were attributed to C-O-C linkage in the molecule, steroidal structure was suggested by absorption bands at 2960, 2940, 2880, 1440-1380 cm^{-1}.(Dyer et al., 1984, Hoh et al., 1978). HNMR spectrum (400 MHz, $CDCl_3$ TMS) of the acetate exhibited five- three protons singlet's due to five acetoxyl group of the sugar moiety which clear indicated the presence of only one sugar unit in the molecule. The presence of six methyl signals between δ 0.58-0.78 and a broad multiplet at δ 3.58 was due to carbinolic proton at position 3. The triplets at δ 5.14 corresponds to the double bond at C-7, C-8 position (Simon et al., 1979 and Jacques et al., 1975). The absence of OH, peak and presence of bunch of singlets at δ 1.94-2.2 suggested the presence of acetyl groups (Mallavarapu et al., 1988). Aromaric protons of sugar moiety resonated as a triplet at δ4.52. rest of the protons of sugar moiety resonated at δ 4.1, 4.18, 4.88, 4.92 and 5.12 indicated the presence of glycopyranosyl moiety attached to C-3 of sterol (Riccio et al., 1986). The molecular formula $C_{43}H_{68}O_{10}$ as established on the basis of FABMS. The molecular ion peaks was at m/z 744. The Aglycone part of compound-2 was subjected to mass spectral analysis. The fragmentation pattern so observed was typically that of sitosterol molecule. The diagnostically important fragments were obtained at m/z 40 $[M\text{-}Me]^+$, 396 $[M\text{-}H_2O]^+$, 381 $[396\text{-}M]^+$, 303 [M-111], 273 $[M\text{-sidechain}]^+$, 255 $[M\text{-sidechain+ }H_2O]^+$ and 213 [255 ring D fission] (Naquie et al., 1973 and Osman et al., 1975). These fragments suggested the aglycon of compound-2 to be a C-29 sterol with one double bond, one hydroxyl group and a C-10 saturated side chain in fragments at m/z 55 (ion a)$^+$, 69 (ion b)$^+$, 83 (ion c)$^+$ and 111 (ion d)$^+$ indicated that the hydroxyl group was located in ring which was placed at C-3 position on biogenetic grounds. Ethyl group assigned at C-24 on the basis of biogenetic analogy as well as close similarities. In the chemical shift values of the protons and carbons of the side chain with related compounds (Gupta et al., 1992)

The peaks in FAB- mass spectrum so obtained were as sodiated fragments (Grigsby et al., 1982). Molecular ion peak came at m/z 434 $[M^+ + Na]$, elimination of methyl group with one sodium atoms gives a fragment at m/z 395, while elimination of acetyl group gave a fragment at m/z 377. The sugar moiety of the compound confirming the prominent peak at m/z 572 based on the above spectral evidence the compound – 2 was characterized as 22, 23-dihydro-7- stregmastenc -3-β-D glucopyrano acetate.

Experimental section:-

General procedure: Melting point were uncorrected. ^{1}HMR spectra were recorded on 400 MHz Brucker WM spectrometer, ^{13}CNMR spectra on a varian XL 100 MHz, spectrometer, IR spectra in KBR on a Perkin Elmer -377 spectrometer. FAB mass and E IMS on a Jeol-JMSD 300 mass spectrometer. The column chromatography was carried out on silica gel. The roots of C. arrundenaceum collected from the nearby area Badwani and Barwaha region west nimar. It identified from school of studies in Botany, Vikram University Ujjain.

Extraction and Isolation of the compound:-

The roots (2 kg) shade dried, cleaned, powdered and extracted with n-hexane and ethanol in soxhlet extractor for 72-96 hrs. from the ethanol extract the excess of solvent was removed by rotary film evaportar and the concentrated extract was obtained as Brown solid Mass. The ethanol extract was separated by column chromatography using silica gel as an absorbent.

The column was eluted with solvents of increasing polarity starting with n-hexane. Fractions were checked by TLC and those of similar compositions were combined and solvent was removed. Compound 1 was obtained from the rechromatography of ethanol eluted of ethanol extract. While compounds- 2 obtained from the acetylelated fraction was then subjected to silica gel. Column chromatography to yield compound-2 in pure form.

2, 3- diacetoxy-4-methyl benzoic acid Compound 1:-

M^+ 220 $C_{12}H_{12}O_4$ (50mg $CHCl_3$: methanol), m.p. 80°C isolated from Benzene: Eto AC (9.5:05v/v) fractions of column is showed single spot on TLC. IR (KBR): 3120, 1735, 1610, 1590, 1480, 1430, 1300, 1100, 1070, 1020 and 750-720 cm^{-1}, 1 HNMR(400 MHz, CDCl3): 2.25 (S, 3H-CH3), 3.1 (s, 6H, COCH3), 7.39 (d, 1H, ArH), (7.61 (d, 1H, ArH) and 8.99 (S, 1H-COOH) EIMS m/z (rel. in.) M+ 220 (9.0), 191 (2.2) 178 (4.9), 177 (34.7), 176 (2.0), 171 (1.2), 161 (1.4), 166(3.1), 136(1.0), 135(7.5), 133(100), 132(1.3), 120(5.9), 119(43.1), 118(9.8), 117(24.9), 116(1.2), 106(2.4), 105(2.8), 104(17.4), 103(29.2), 102(6.0) and 77(89.8).

^{13}CNMR (100 MHz, CDCl3, PPM): δ 184, 177.05, 157.75, 144.49, 138.25, 128.40, 128.13, 128.8, 39.29, 39.70 and 13.37 PPM.

22,23 –dihydro-7-stigmastence -3-β-D-glucopyrano acetate:- (Compound -2)

M^+ 760, $C_{43}H_{68}O_{11}$ (45 mg, $CHCl_3$: methanol), m.p. 120°C isolated from Benzene: Eto AC(3:1 v/v) fraction of column. It showed single spot on TLC IR (KBR): 3420, 2960, 2940, 2880, 1750, 1660, 1440, 1380, 1370, 1220 and 1140-1120 cm-1. ^{1}HNMR (400 MHz, $CDCl_3$, TMS), 5.14 (+, 1H-CH=C), 5.12 (dd, 1H, HC-O, J=4 Hz, C-3), 4.8 (+, J=3.5 Hz), 4.18 (d, J=3Hz), 4.1 (d,J=3Hz), 3.58 (m, 1H,-CH, C-3), 1.9-2.3 (S, -OCO-CH3), 0.76 (S, 3H, me), 0.74 (S, 3H, me), 0.71 (S, 3H, me), 0.70 (S, 3H, Me), 0.62 (S, 3H, Me) and 0.58 (S, 3H, Me). EIMS M/Z (rel. int., %) : M+ 574 (66.1), 462(6.8), 414(11.36), 413(27.2), 398(50.2), 396(48.1), 381(3.4), 351(2.2), 313(4.4), 311(4.4), 303(1.1), 273(7.9), 271(9.0), 255(45.4), 229(20.4), 218(1.1). 213(11.3), 203(4.5), 169(1.1), 157(6.8), 111(0.22), 94 (29.5), 83(100), 69(47.7) and 55 (25.0) etc.

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