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Short Communication

1-Acetyl D-erythritol from the leaves of Cestrum nocturnum

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A R T I C L E I N F O

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Chemical Investigation of leaves of Cestrum nocturnum (Solanaceae) led to the isolation of a compound which was characterized as 1-acetylbutane 1,2(S), 3(S)-4-tetraol or 1-acetyl D-erythritol (I) based on spectroscopic studies.

1. Introduction

Cestrum nocturnum belonging to the family of Solanaceae commonly cultivated in India for fragrant flowers was investigated for its chemical constituents. Earlier studies on this plant have led to the isolation of alkaloids[1], Saponius[2], glycosides.[1] The saponins and sapogenin-containing fractions of the plants were reported to show interesting biological activity.

2. Experimental (Material and Method)

The leaves of Cestrum nocturnum Linn was collected from the campus of BHU Varanasi India. A specimen of the plant material has been preserved in the department of Medicinal chemistry I.M.S BHU Varanasi.

MP were taken on a Yazawa hot stage microscope and are uncorrected. The UV Spectra were obtained on a Shimadzu UV 200 spectrometer using spectral methanol. I.R spectra were recorded on a JASCO. J.R.130 spectrometer in $CHCL_3$. ^1H and ^{13}C-nmr were recorded on JEOL 270 Hz or Brucker 400 or JEOL GSX 500 spectrometer using $CDCL_3$ solution with TMS as an internal reference. Optical rotations were recorded on Yonoco-OR-50 automatic polarimeter (Cell length 1 cm) GC-MS and direct inlet mass spectrum(7uev) was obtained with a Shimadzu-GC-MS DF 9020 instrument using OV-1 capillary column. Silica for column chromatography refers to E-MercK (India) 60-120 mesh and TLC was carried on silica gel G (Merck) In different solvents.[1–4]

The dried leaves of Cestrum nocturnum (0.6 Kg) were powdered and soxhletted with ethyl acetate. The ethyl acetate extract was concentrated under reduced pressure and chromatographed over a column of silica gel and eluted with solvents of increasing polarity. The benzene ethyl acetate (1:4) eluate was a mixture of two components.

The preparative HPLC of the mixture was carried out on the Shimadzu-HPLC apparatus using a reverse-phase silica column (RP-18). The flow rate was 8ml/min and eluent used MeOH-H_2O (9:1),compound I was eluted after 8 minutes. The eluent from the period 7.5 minutes to 8.5 minutes was collected and the solvent was removed under reduced pressure which yielded solid residue 0.05 gm of I, MP 75 degree Celsius $[\alpha]_D + 8.52$(MeOH) IR(γ_{max}): 3400,1735,1370 1240,1075 and 1040 cm^{-1}

^1H-nmr (270 MHz) $CDCL_3$ and two drops of CD_3OD : δ 2.11 (3H, s, -OAc), 3.37(4H, br.m), δ 4.24 (2H, ddd, J=11.7, 6.3 and 3.4 Hz, 1-H_z) ^{13}C-nmr : 20.7(q-$COCH_3$), 63.2(t-CH_2OCOCH_3), 65.9 (t- CH_2OH),71.5, 70.4 (q each,

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-CHOH), 172.0 (s-COCH$_3$)

3. Acetylation of 1 to 1a

A mixture of 1(0.019), AC$_2$O (0.2 ml), and pyridine (01 ml) was kept overnight under anhydrous conditions. Usual workup yielded a crystalline solid of CNL-C-1 triacetate (1a) m.p 89 degree Celsius, $[\alpha]_D$-30 (EtOH).

IR(γ_{max}): 1742, 1440, 1375, 1230, 1208, 1050, 1022, 975 and 950 cm^{-1}.

^{1}H nmr (270MHz, CDCl$_3$) : δ 2.07, 2.09(6H, s-each, 4x-OAc) : 4.19, 4.33 (2H, dd each ; J=12.2, 6.8Hz 1 & 4- H) , 5.26(2H,t, J= 2.5 Hz, 2 & 3-H)

C^{13}-NMR (67.5MH$_z$); 20.8,20.6 (q each,-OCOCH$_3$) 61.7(d,- CHOAc), 69.1(t -CH$_2$OAc), 170.5,169.8 (S each, -COCH$_3$)

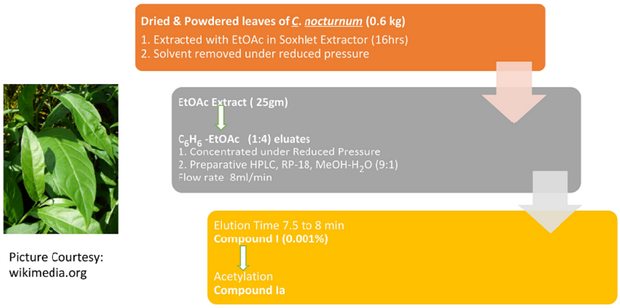
4. Results and Discussion

The ethyl acetate extract of Cestrum nocturnum leaves was eluted with solvents of increasing polarity the C$_6$H$_6$: EtoAc (1:4) eluates concentrated under reduced pressure and purified by preparative HPLC RP-18 MeOH-H$_2$O (9:1) flowrate 8ml/min elution time 7.5 to 8.5 min yielded component(I).

Compound(I) was obtained as colorless solid mp 75 degrees Celsius $[\alpha]_D$ + 3.8(EtoH) M.F C$_6$H$_{12}$O$_5$. The presence of a smaller number of carbon and high oxygen content in the molecule was considered to be due to polyhydroxy compound. The presence of strong i.r absorption at 3400 cm^{-1} and 1735 cm^{-1}prove the presence of hydroxyl and ester carbonyl group. It shows the signals for methylene hydrogens at 84.24 as doublet of double doublet J= 11.7, 6.3 and 3.4 Hz. The low shift of methylene hydrogens is probably due to the linkage of ester group to methylene carbon. ^{1}H nmr also shows signals indicate that component I possess a-CH$_2$OCOCH$_3$ group.

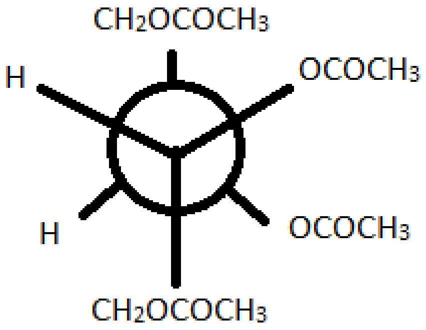
The number and nature of carbon present in the molecule was determined by ^{13}C-nmr Spectrum. ^{13}C-nmr spectrum of I showed the presence of one acetate group (δ_c 65.9 and 63.2 each) and two methine carbon (δc 71.5 and 70.4mof each) The chemical shifts of the carbon for methylene and methine clearly suggested them to be bound with oxygen functionalities and up field carbon signals at δc 63.2 was considered to be for methylene bound to acetate group. It was assumed from ^{1}H and^{13}Cnmr spectral studies that compound I is probably 1-acetoxy n-buta 2,3,4 triol.

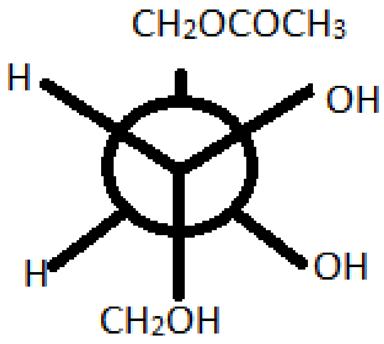
Acetylation of I yielded triacetate Ia M.F C$_{12}$H$_{18}$O$_8$ $[\alpha]$D – 20(EtoH). IR spectrum of 1a Showed absence of hydroxyl absorption and strong ester. Carbonyl absorption (γ_{max} 1742 cm^{-1}) ^{1}H nmr showed two singlets for acetate group at δ 2.07 and 2.09, two dd at δ 4.19 (J= 12.2 and 6.8 Hz) and 4.33(J+ 12.2 and 2.5 Hz) and five line signals at δ 5.26. These signals were found to be in a integral ratio of 3:3:1:1:1 accounting for 9H. A comparison of hydrogen integral ratio of H-nmr spectrum to the M.F of Ia it is concluded that hydrogen were in the ratio 6:6:2:2:2. The downfield shift of the signal at 85.26 suggested it to be originated from CH$_2$OAc group which was further linked to a methylene, the H of which appeared as a double doublet at δ 4.19 and 4.33. Thus, it is concluded that triacetate of I(Ia) is nothing but tetraacetate of butane 1,2,3,4- tetrol.



Picture Courtesy: wikimedia.org

Fig. 1:





The stereochemistry of I was settled by comparison of m.p of Ia with those of prepared tetraacetate od D-erythritol which were found to be identical A comparison of the specific rotation of I with reported values of all the three isomeric forms of butane 1,2,3,4 tetrol was very informative. Thus compound I may be written as 1-acetyl butane 1,2(S), 3(S), 4-tetraol or 1-acetyl D-erythritol.

This is the first report of 1-acetyl D-erythritol as a natural as well as synthetic source.

5. Source of Funding

This research was not financially supported by any funding agencies.

6. Conflict of Interest

None.

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