ejpmr, 2016,3(1), 402-404

SJIF Impact Factor 2.026

EUROPEAN JOURNAL OF PHARMACEUTICAL AND MEDICAL RESEARCH

www.ejpmr.com

Research Article

ISSN 3294-3211

EJPMR

A NEW TRITERPENOID ISOLATED FROM *LANTANA CAMARA*

***Kavithapriya.Kummara[1], Anuradha Vejendla[2], Y. Narasimharao[3]**

[*1]Department of Chemistry, ANU, Guntur, A.P. INDIA.
[2]Department of Chemistry, VNITSW, Guntur, A.P. INDIA.
[3]Department of Bs and H, Vignans LARA Vadlamudi, A.P.INDIA.

***Correspondence for Author: Kavithapriya.Kummara**
Department of Chemistry, ANU, Guntur, A.P. INDIA.

Article Received on 20/11/2015 **Article Revised on 12/12/2015** **Article Accepted on 01/01/2016**

ABSTRACT

From the whole plant of Lantana camara, a new triterpenoid was isolated. Its structure was elucidated as 3*β*,17-dihydroxy –olean – 12- ene on the basis of spectroscopic data. This is the first report from Lantana camara and from the nature.

KEYWORDS: *Lantana camara Verbenceae*, triterperiod, 3β,17-dihydroxy-olean-12-enc[1]

INTRODUCTION

In the course of investigations on chemical constituents of *Lantana camara*, several triterpenoids- β–sitosterol[1], Oleanolicacid[2], Lantanilicacid[3], and flavonoids- Luteolin[4], Pectolinarigenin[5] were reported so far. In our investigations, besides the above reported triterpenoids and flavonoids, we report the isolation and characterization of a new triterpenoid, 3β,17-dihydroxy-olean-12-ene[1] from the whole plant of *Lantana camara*.

1. EXPERMENTAL WORK

1.1 MATERIAL AND METHODS: The plant material of *Lantana camara* of about 7kgs was collected from the collectorate compounds in Guntur along with roots during October 2006 and was air-dried under shade.

1.1.1 ***EXTRACTION AND ISOLATION***: The air dried whole plant of *Lantana camara* was powdered and extracted repeatedly with hexane and methanol and finally with ethanol. The alcoholic extracts were combined and impregnated on minimum amount of silica gel and washed successively with hexane, chloroform and ethanol. The chloroform soluble fraction was partitioned into ethyl acetate soluble and insoluble parts. The ethyl acetate soluble part was treated with 4% NaOH to separate acidic and neutral fractions. The neutral fraction on repeated column chromatography using petroleum ether, chloroform and methanol followed by preparative chromatography, in chloroform and methanol [1:1] yielded the new triterpenoid 3β,17- dihydroxy-olean-12-ene on crystallization with ethyl acetate . It was recrystallised as colourless needles (mp –197-199^{o}C), analysed for $C_{29}H_{46}O_3$ [[M+] m/z= 322, (C=3.44%, H=2.173%, O=33.3%)

UV λ_{max}^{EtOH} = 250nm.

IR ν_{max}^{KBr} = 3150cm^{-1}, 3700cm^{-1}, 2860cm^{-1}, 2950cm^{-1}, 1690cm^{-1}

^{1}HNMR (δppm) 1.11(12H, s, 4xCH_3), 1.16(6H, s, 2xCH_3), 1.25((2H, s, CH_2-15), 1.28(3H, s,H-1xCH_3) 1.36(6H, s, CH_2-6, CH_2-19,CH_2-21)1.38 (2H, s,CH_2-1), 1.39 (1H,s,H10), 1.40(4H, s, H-5,H-16), 1.55 (2H,s, H-22) 1.59 (2H,s,H-2) 2.0(brs ,2H, 2xOH),2.19(1H,t,J=4.6Hz,H-18) 2.26(1H,s,H-8) 3.15(dd,1H, H-3,J=10.1Hz,5.0Hz),5.77(1H,S,H-12)

^{13}CNMR (δppm):- 27.5(C-1), 25.3(C-2), 76.2(C-3), 34.6(C-4), 18.1(C-5), 29.7(C-6), 29.6(C-7), 63.0(C-8), 23.8(C-9), 48.7(C-10), 200.7(C-11), 119.8(C-12), 162.6(C-13), 44.6(C-14), 27.5(C-15), 37.3(C-16), 72.7(C-17), 40.7(C-18), 34.0(C-19), 25.1(C-20), 31.9(C-21), 31.3(C-22), 27.3(C-23), 22.3(C-24), 19.1(C-25), 19.1(C-26), 20.7(C-27), 18.3(C-28), 17.8(C-29).

Acetylation of 3-β-17 dihydroxy-olean-12-ene **:-** 4mg of 3-β-17 dihydroxy-olean-12-ene of in pyridine(1ml) and acetic anhydride (2ml) was heated on a water bath at 100^{o}C for 3hrs, excess reagent and pyridine were removed under vacuum. The residue was dissolved in chloroform and washed with water. The chloroform extract was dried over anhydrous Na_2SO_4 and concentrated under vacuum. The residue was recrystallised from MeOH yielding colourless needles of 3β,17- dihydroxy-olean-12-ene acetate. It was obtained as colourless needles.

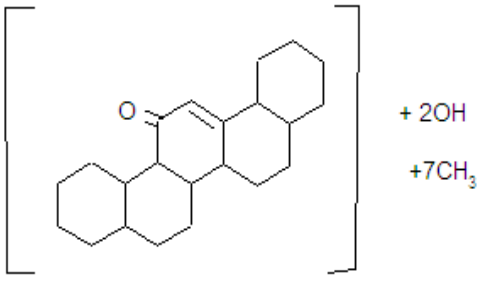
Yield =2mg, m.p = 215°C, m/z = 484, (Analyzed for $C_{31}H_{48}O_4$, C=3.225%, H=2.04%, O=25%)

^{1}HNMR: 1.36(1H, s, H-1), 1.68(1H, s, H-2), 4.15(1H, t, H-3), 1.40(1H, s, H-5), 1.36(1H, s, H-6), 2.26(1H, s, H-8), 5.77(1H, s, H-12), 2.19(1H, t, H-18), 2.01(3H, s, acetyl).
^{13}CNMR: 27.7(C-1), 22.0(C-2), 83.0(C-3), 34.6(C-4), 17.8(C-5), 29.7(C-6), 29.6(C-8), 63.0(C9), 23.8(C-10), 200.7(C-11), 119.8(C-12), 162.6(C-13), 44.6(C-14), 27.5(C-15), 37.3(C-16), 72.7(C-17), 40.7(C-18), 34.0(C-19), 25.1(C-20), 31.9(C-21), 31.3(C-22), 27.3(C-23), 27.3(C-24), 171.0(-C=O), 19.3(C-25), 19.3(C-26), 20.7(C-27), 18.3(C-28), 17.8(C-29).

2. RESULTS AND DISCUSSION

3β,17-dihydroxy-olean-12-ene was recrystallised as colourless needles (mp –197-199°C), analysed for $C_{29}H_{46}O_3$[[M+] m/z = 322].C=3.44%, H=2.173%, O=33.3%). It showed IR bands at IR ν_{max}^{KBr} at 3150 (-OH), 2860, 2960 cm^{-1}, 1690cm^{-1} (ά,β- unsaturated ketone) indicating the presence of alcoholic –OH and ά,β- unsaturated carbonyl systems. It formed a monoacetate (mp –185-187 °C) analysed for [[M$^+$] m/z =484] $C_{31}H_{48}O_4$.IR showed ν_{max}^{KBr} 1730 cm^{-1} ^{1}HNMR at δ2.01 (3H,s, -$COCH_3$) ppm supporting the presence of one hydroxyl group. This accounts for two oxygens out of three in the molecular formula of 3β, 17- dihydroxy-olean-12-ene . The third oxygen may be in the form of a tertiary hydroxyl or a epoxy group, this supports the presence of three oxygen atoms in the molecular formula. The ^{1}HNMR spectrum showed 7-methyl singlet signals at (δ1.11,4XCH_3, δ1.16, 2XCH_3 &δ1.28,1XCH_3) accounting for seven carbons in the molecular formula. It displayed resonance for an olefinic proton at δ5.77 (s,1H) and ν_{max}^{KBr} at 1690 cm^{-1} indicated the presence of a trisubstituted ά,β-unsaturated ketone. This was further supported by ^{13}C-NMR at δ 119.8 and δ162.6 for C-12 and C-13 respectively.

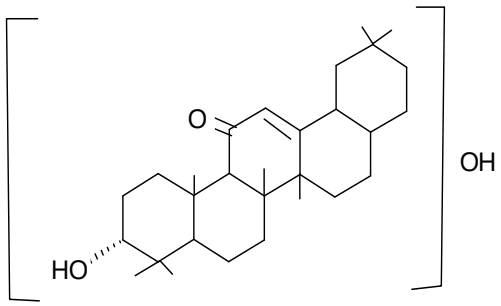
The PMR and CMR data of 3β,17-dihydroxy-olean-12-ene indicates that it belongs to β-amyrin oleanane type of terpenoids.[6] This further supported the presence of (OH) attached to a tertiary carbon. Thus partial structure of 3β,17- dihydroxy-olean-12-ene can be given as below



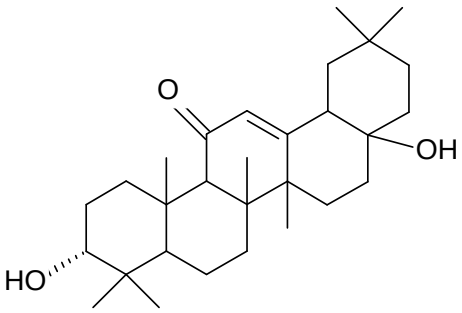
The down field region of the PMR spectrum of 3-β-17 dihydroxy-olean-12-ene showed the presence of two signals the triplet at δ3.15 (1H) ppm and 5.77 (s,1H) ppm. The triplet at δ3.15(1H) ppm shifted to downfield and appeared at δ4.30(dd,1H,J=10.2,5.1Hz) ppm in the monoacetate.[7] Thus, this signal is assigned to methine proton attached to the carbon containing hydroxyl group. Absence of any other downfield signal in acetate supported the presence of hydroxyl attached to a tertiary carbon.

Many β-amyrin triterpenoids were reported from the same plant based on the substituents pattern and PMR data the methyls were assigned to 4a, 4b, 8,10,14, 20a and 20b.[8]

Thus, the partial structure is-



The characteristic triplet signal at δ2.19(1H) indicated the presence H- 18 leaving C-17 the only position for –OH.[9]



Thus, the structure of 3β,17- dihydroxy-olean-12-ene is given as above. This is the first report of such compound form *Lantana camara* and triterpenes.

3. CONCLUSION: Many triterpenoids were reported from *Lantana camara* as well as from other plants. Triterpenoids show excellent medicinal properties. This is the first report of the triterpenoid 3β,17- dihydroxy-olean-12-ene from nature and from *Lantana camara.*

ACKNOWLEDGEMENT
Firstly I am thankful to GOD . I extend my sincere thanks to my research guide, Chairman and Principal

of VNITSW, Guntur for providing facilities to carry out my research activity.

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