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Isolation and Characterization of Bis (2 – Methoxyethyl) Phthalate and Hexashydro-1 3 – Dimethyl – 4 – Phenyl – 1h – Azepine 4 – Carboxylic Acid from the Root of *Cissampelos* Owariensis (P. Beauv)

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ABSTRACT : The root of C*issampelos owariensis* yielded two new additional compounds. These compounds were identified on the basis of spectroscopic analysis , here reported for the first time from the plant as bis (2-methoxy ethyl) phthalate and hexa hydro-1,3-dimethyl -4-phenyl-IH-azepine-4-carboxylic acid

INTRODUCTION

The phytochemical investigation of the root and leaf extracts of *C. owariensis* yield the following alkaloids bisbenzylisoquinoline, isochrodrondendrin, berberine and cycleanine(Southen, 1989).

Recently, we described the isolation and identification of two sesquiter terpene, to be 2H-cyclopropa-naphthalene-2- one, 11a, 4,5,6, 7,7a ,7b octahydro-1,1, 7,7a tetramethyl, and 5(1H)-azulenone, 2,4,6,7,8 8a hexahydro 3,8-dimethyl(-4-(1-methyl ethylidene respectively (Efiom et al, 2009).

The isolation of this interesting group of new compounds, combined with our taxonomic interest in the Menspermaneae as a household name in an African traditional medicine, stimulated further an investigation of the root of *C. owariensis.* Two new compounds bis (2-methoxy ethyl) phthalate and he xahydro1,3-dimethyl(-4-phenyl-1H-azepine-4-carboxylic acid have now been isolated along with other compounds.

MATERIALS AND METHODS

Materials; The root of C. owariensis was collected in September 2006 from Gwagwa, Suleja, Nigeria and authenticated at the National Institute for Pharmaceutical Research and Development (NIPRD) Idu, Abuja, Nigeria. Voucher specimen has been deposited at the herbarium. IR (cm^{-1}) nujol as internal reference on 1310 spectrometer, MS; Kroto 80, EIMS using Hewlet Pack care ICMS, CC; Merk-Kiesel gel 60 ;(230-400mesh) TLC and PTLC: precoated Merk Kiesel gel 60F 254(0.25mm and 100mm respectively. TLC detector UV lamp (254to366Nm), Iodine vapour and Vanillin/H_2SO_4 1%w/V

Extraction: The dried pulverized root (1kg) was extracted with 95% ethanol ($2dm^3$), for six hours using soxhlet extractor. The extract was evaporated to dryness in rotatary evaporator to give 15g residue.

Fractionation of Ethanolic Extract *C.owariensis*: Ethanol extract (5g) was absorbed in silica gel 5g and loaded on column of silica gel. The column was then eluted using pump pressure and varying the eluent polarity ratios. The Elution with a mixture of ethyacetate in n-hexane 20% and the collection of 100ml portion. A total of 15 fractions were collected. The fractions were combined based on their R_f values. The R_f profile were found to be closely related 0.67 and 0.70 respectively.

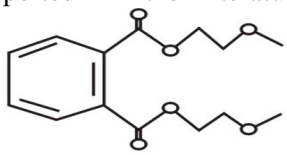
The combined fractions were given an identification number RB_1 and RB_2. Fractions RB_1 and RB_2 were further purified using PTLC on silica gel [ethyl acetate/hexane (4:1)] to give yellow gum (227mg) and a brown gum (200mg) respectively. They were subjected to spectral analysis as follows:

Characterization of Compound 1:IR: ν cm^{-1} 1729 1632 1434 1381 and 1070 GC scan at 363(RT=4.789) EIMS (relative intensities %m/z) 195 (M^+15%)149(M^+100%) 132 (M^+10%) 121(M^+4%) 104 (M^+3%) 93 (M^+3%) 83 (M^+2%) 70(M^+11%) 55(M^+12%) 41 (M^+12%) and 39(M^+2%)

Characterization of Compound 2:IR: ν (cm^{-1}) 3500 (-OH of COOH), 1725(C=O of COOH), 1640 (C=C), 1461 1283 and 1121. GC scan at 12 (RT=1.743), EIMS(relative intensity 5%) m/2 247 (M^+3%) 231 (M^+11%) 223(M^+13%) 202 (M^+10%) 184(M^+21%) 168(M^+39%) 141(M^+42%) 128(M^+29%) 115(M^+54%) 92 (M^+14%) 77(M^+100%) 64(M^+25%) 51(M^+35%) and (M^+13%)

RESULTS AND DISCUSSION

The roots of *Cissampelos owariensis* were extracted with 95% ethanol followed by CC over silica gel (see experimental). This procedure gave two compounds named bis (2-methoxy ethyl) phthalate (1) and hexahydro -1, 3-dimethyl -4-phenyl -IH-azepine -4-carboxylic acid (2). Identification of the known compounds were based on their spectroscopic evidence and comparison data reported in the literature.



Compound 1] The IR spectrum (CCl_4) showed carbonyl at 1729cm, typical of an aromatic ester ($ArCOO^-$) (James et al, 1982). This is also supported by the presence of strong bond in the C-O stretching at 1228cm.The unmistakable of a (C=C) group at 1632cm is characteristic of aromatic compound. The MS exhibited at m/z 149(100%) fragment mass ion, which is characteristic presence of phthalates (fig 1) corresponding to the simultaneous loss of a methoxy ethyl group to give phthalic acid anhydride moiety {William , 1993 and Sastry, 1995}. The m/z of phthalate as it represents loss of one side chain on the phthalate. The mass ion, M^+ which is not visible in the MS was expected at m/z 282 just as was reported in(fig1). The above spectral characteristics and comparison of the MS with the stored library mass spectral data and literature for phthalate were supportive of the fact that the compound is bis (2- methoxy ethyl) phthalate. Among the phthalates, di-n-octylphthalate has previously been isolated from marine brown alga, Sargasum wightii as the major compound responsible for its anti bacterial activity (Honghung et al, 2007 and Luigton, 1997).

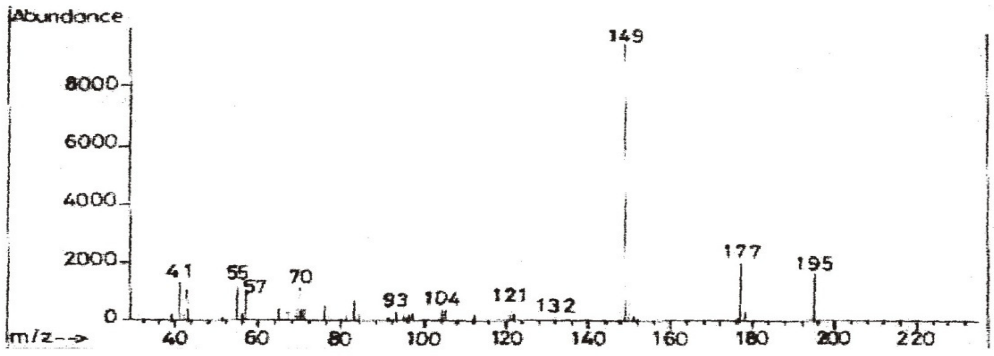


Figure 1 Mass spectrum of bis (2-methoxy ethyl) phthalate

Compound 2 with molecular formula $C_{15}H_{21}O_2N$ was shown by GCMS. It has two double bond equivalent which are easily identifiable from the IR spectrum , with carbonyl group absorbing at 1725cm and a C=C double bond at 1640cm. The bond at 1640cm showed the aromatic nature of the compound, which is partly supported by mass spectrum at m/z=77(base peak). The mass spectrum showed molecular ion M^+ at m/z 247. The fragment ion at (203-184) showed the loss of water molecule. The odd molecular ion at 247 also suggests a single nitrogen atom in the molecule of the compound. The high resolution mass spectrum of compound 2 gave the molecule mass as 247 corresponding to the $C_{15}H_{21}O_2N$ as shown in fig2

Conclusion: The results had also shown some possible chemical constituents of the root of *C.owariensis* to which the local usage of the medicinal plant may be attributed (Efiom et al, 2009 and Akinniyi, 2005)**.**The results also confirm the need to study the chemistry and biological activities of the root of *C owariensis* that have before now not been investigated. This study could also be chemotaxonomically significant.

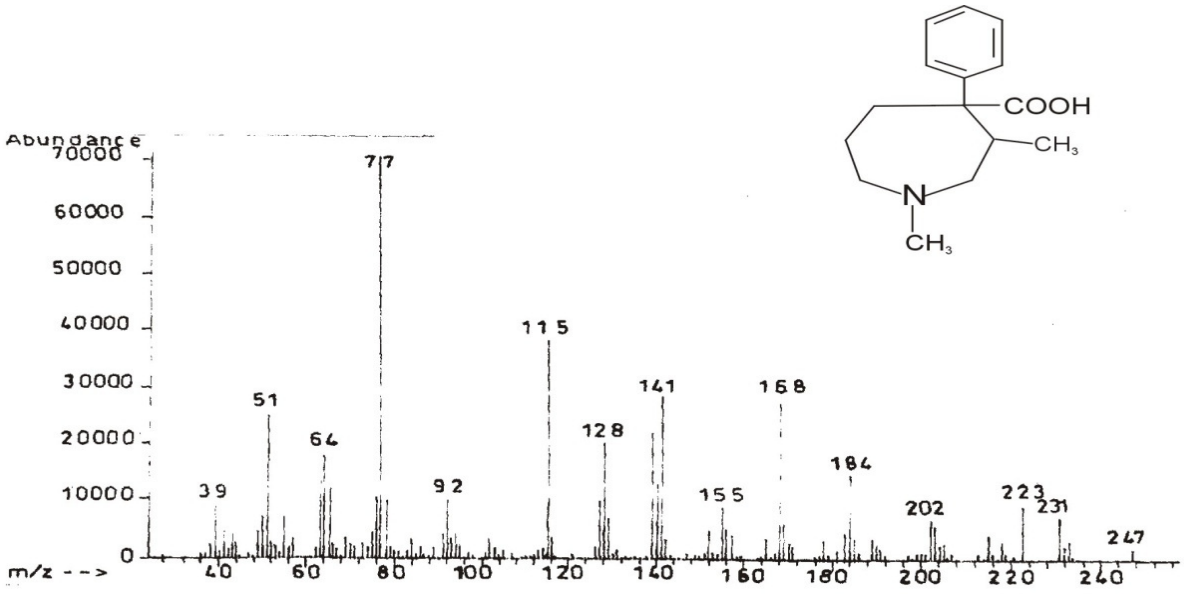


Figure 2: Mass spectrum of hexa hydro -1,3 - dimethyl -4- phenyl -IH- azepine -4- carboxylic acid

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