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A New 2,8-dihydroxy-1,6-dimethoxy Xenthones from *Cythula tomesntosa*

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ABSTRACT

From alcoholic extract of whole plant *Cyathula tomentosa* a new 2,8-dihydroxy-1,6-dimethoxyxanthone has been isolated and characterized with help of FAB-mass, ^{1}H, ^{13}C NMR and 2D studies & antimicrobial activities of its extract.

Key words: *Cyathula tomentosa,* amaranthaceae, 2,8-dihydroxy-1,6-dimethoxyxanthone.

INTRODUCTION

Cyathula tomentosa (Kurru) belongs to family Amaranthaceae, is a perennial under shrub occurs throughout Garhwal Himalayas up to 600-2000 meter altitude. *Cyathula tomentosa* have been used in snake bite and has emetic properties[1]. From *Cyathula capitate* and *Cyathula officinales* isolated ecdyson content is 0.046% and 0.057% respectively[2] and *Cyathula prostrata* show antifungal free redical scavenging activities 2,2-diphenyl-1-picryl hydrazyl [DPPH] radical[3]. The chemical examinants at the basis of Malikov & Yuldavshev, it has been reviewed and found no chemical analysis from literature survey on *Cythula tomentosa*. From the ethanolic extract of *Cyathula tomentosa* a new flavanone compound is isolated. The structure of compound has been through mass, ^{1}H, ^{13}C. NMR and 2 D-NMR spectra.

EXPERIMENTAL

General

^{1}H-NMR at (400 MHz),^{13}C-NMR at (75 MHz) TMS as internal standard, using DMSO as solvent, Columan Chromatography was carried out on silica-gel 60-120 mesh (Merck). TLC was performed on percoated silica-gel. The eluting solvent was $CHCl_3$-MeOH spots were visualized by 7% H_2SO_4 followed by heating.

Plant material

The whole plant of *Cyathala tomentosa* were collected from Bacchear District. Chamoli Garhwal Uttrakhand in the month of October and identified by Department Botany, P.G. College Gopeshwar where Vaucher specimen was deposited.

Extraction and isolation

The air dried whole plant (3kg) was exhaustively extracted with 90% aqueous EtOH for 72 hours. The ethanolic extract was concentrated to dryness. The dry ethanolic extract was chromatographed over silica-gel using Methanol Chloroform (30:70) as eluting solvent which afforded the compound.

RESULTS

It was crystallized from methanol as yellow amorphous solid m.p. 236^{0}C compound was obtained as yellow amorphous solid from methanol was found to have molecular weight 288 as presumed by the presence of molecular ion peak at m/z 288 in the EI-positive mass spectrum. MS-EI$^+$; m/z 288, 270, 245, 214, 199, 184, 123, 69 etc. Elemental analysis of compound found values, C=62.47%,H=2.6%, required values for C15H12O16; C=62.50%, H=4.25%; molecular weight 288 showing ten double bond equivalents (DBE) in the molecule its IR (ν_{max} KBr): cm^{-1} at 3372, 2843, 16655, 1612, 1576, 1508, 1475 etc. and showed absorption at 3372, [for hydroxyl group (s)] 2843 (alkane) and 1655 and 1612 cm^{-1} (for α-β unsaturated carbonyl group). The UV spectrum showed absorption maxima at 207, 230, 254, 267, 279 and 300 nm, the characteristic of xanthone[5].

The ^{13}C-NMR spectrum of compound disclosed presence of fifteen carbon atoms out of which four were methine, two methyl and nine quaternary carbon atoms. The methine and methylene carbon atoms were promptly assigned by heteronuclear multiple quantum correlation (HMQC) experiment which allowed one bond coupling between a given proton and carbon atom.

Table 1.

C/H	δC	Multiplicity	δH (J in Hz)	HMBC Correlation (H→C)
1	146.4	C	-	-
2	150.4	C	-	-
3	125.2	CH	7.59, d (9.03)	C-1, C-2, C-4a
4	113.9	CH	7.23, d (9.03)	C-2, C-4a, C-9a
4a	148.5	C	-	-
4b	157.6	C	-	-
5	92.2	CH	6.47, brs	C-4b, C-6, C-7, C-8a
6	166.7	C	-	-
7	97.4	CH	6.55, brs	C-5, C-6, C-8, C-8a
8	164.2	C	-	-
8a	104.5	C	-	-
9	181.5	C	-	-
9a	115.9	C	-	-
1-OMe	61.6	CH_3	4.05,s	C-1
6-OMe	53.9	CH_3	3.74, s	C-6
2-OH	-	-	11.8, s	C-2
8-OH	-	-	13.9, s	C-7, C-8, C-8a

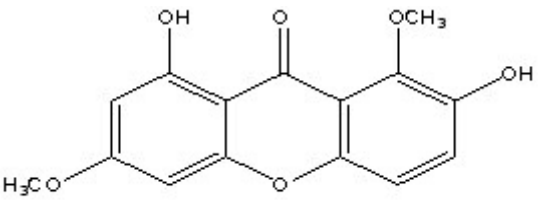
The ^{1}H-NMR spectrum of compound disclosed the presence of two AMX- type doublets (J=1.5 Hz) at 6.47 and 6.55 and two AB-type doublets (J=9.0 Hz) at 7.59 and 7.23 implying the presence of two tetra-substituted benzene nuclei in the molecule. The broad singlet at 11.8 and 13.9 were attributed to two phenolic groups. Two singlets (each for 3H) at 4.05 and 3.74 were correlated with the methoxy proton attached at benzene ring.

In the ^{13}C –NMR spectrum of compound a downfield signal at 181.5 was attributed to alpha,beta- unsaturated carbonyl carbon, which is

compatible with the carbonyl group of xanthone [6]. The carbon resonance at 146.4, 150.4, 166.7, 164.2 showed the presence of four oxygenated carbon atoms and are compatible with the 1,2,6,8-oxygenated carbon atoms of xanthone [6]. Two carbon signals at 61.1 and 53.9 in the ^{13}C-NMR spectrum were assigned for methoxy carbon atoms.

The position of methoxy groups was authenticated to be C-1 and C-6 by comparison of ^{13}C –NMR data with related compound [6]. The downfield chemical shift of both carbon and hydrogen of a methoxy group at δ 61.6 and 4.05 respectively as compare to the other methoxy group at 53.9 and 3.74, suggested that the former be chelated with the carbonyl group. This indicate that the methoxy group should be either at C-1 or C-8 position. The HMQC spectra of compound showed $^{2}J_{CH}$ interaction of ^{1}H-NMR signal at 4.05 with C-1 carbon atom at 146.4 and the methoxy signal at 3.74 showed $^{2}J_{CH}$ correlation with C-6 at 166.7. These long-range correlations suggested the allocation of methoxy group at C-1 and C-6 position of molecule, The phenolic proton at 13.9 showed long range correlation with C-8, C-7 and C-8a in the HMQC spectrum confirmed the allocation of OH function at C-8 carbon atom. The other long-range correlations identified by the HMBC spectrum are showed in Table.

On the basis of the above discussed spectrum data compound was characterized as 2,8-dihydroxy-1,6-dimethoxyxanthone which was further confirmed by the comparisons of its 1H, 13C-NMR data with reported data[6].



2,8-dihydroxy-1,6-dimethoxy-9*H*-xanthen-9-one

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