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Research Article

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1,8-dihydroxy-3,5-dimethoxy xanthone from *Cythula tomesntosa*

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ABSTRACT

From alcoholic extract of whole plant Cyathula tomentosa a new 1,8-dihydroxy-3,5-dimethoxy xanthone have been isolated and characterized with help of FAB-mass, ^{1}H, ^{13}C NMR and 2D studies.

Keywords: *Cyathula tomentosa*, amaranthaceae, 1,8-dihydroxy-3,5-dimethoxy xanthone.

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 [4] has been revieweds We 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 SECTION

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.

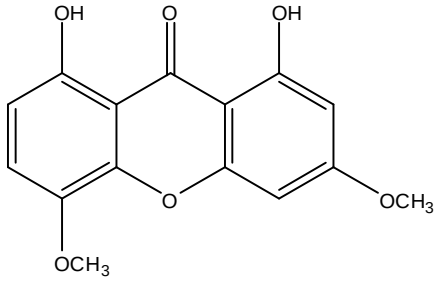
RESULTS

It was crystallized from MeOH as yellow crystalline solid M.P.202^{0}C compound was found to have molecular weight 288 as deduced by the presence of molecular ion peak $[M]^{+}$, 273, 241, 230, 178, 151, 123, 69 etc. Its elemental analysis found values, C=62.61%, H=4.26%, required values for C15H12O6; C=62.5%, H=4.25%,

molecular weight 288. The IR (vmax KBr): cm^{-1} 3856-3630, 1662, 1637, 1578, 1560, 1283, 1163, 1105 etc. showed characteristic absorption of chelated OH group(s) at 3856-3630 and of α-β-carbonyl group at 1663 and 1637 cm^{-1} showing presence of OH group(s) and carbonyl group in the molecule. The presence of OH and carbonyl functions was confirmed by its UV spectrum. Like compound displayed four aromatic two aliphatic and two phenolic proton signals in ^{1}H-NMR spectrum two aliphatic and thirteen aromatic carbon atoms in the ^{13}C-NMR spectrum. The multiplicity of carbon signals were determined by the DEPT spectrum, which confirmed the presence of four methine, two methyl and nine quaternary carbon atoms. The assignment of methine and methyl carbon atoms and proton attached with them were readily made by the HMQC experiment.

The ^{1}H-NMR and ^{13}C-NMR spectra of compound were similar to those of compound. The aromatic substitution pattern was determined by the presence of two meta-coupled doublets (J=2.2 Hz) and two AB-type doublets (J=8.8 Hz). The doublet at δ 6.35 and 6.54 were assigned for H-2 and H-4 respectively while two AB-type doublets at δ 7.22 and 6.71 were allocated for H-6 and H-7. Two singlets (each for 3H) at δ 3.89 and 3.96 showed presence of two methoxy groups in the molecules which was further authenticated by ^{13}C- chemical shift at δ 56.0 and 57.4. The presence of two broad signlets at δ 11.98 and 11.39 was corroborated with the presence of two phenolic groups in the molecule.

The downfield ^{13}C-chemical shifts at δ 162.8 (C-1), 167.4 (C-3), 139.9 and 154.1 (C-8) are in accordance with the 1,3,5,8-tetrasubstituted xanthone [6]. The presence of carbonyl carbon was confirmed by the ^{13}C-chemical shift at δ 184.6.The various fragments to be connected in the molecules were determined by HMBC spectrum. The methoxy protons at δ 3.89 and 3.96 showed $^{2}J_{CH}$ interaction with C-3 (δ 167.4). and C-5 (δ 139.9) carbon atoms, which confirmed their allocation at C-3 and C-5 respectively. The phenolic proton at δ 11.98 showed $^{2}J_{CH}$ interaction with C-1 and $^{3}J_{CH}$ interaction with C-2 and C-9a and $^{4}J_{CH}$ interaction with C-3 while the phenolic proton at δ 11.39 showed $^{2}J_{CH}$ interaction with C-8 and $^{3}J_{CH}$ interaction with C-7. These long-range correlation of the phenolic protons allocated the C-1 and C-8 position of phenolic groups. Other long range correlations established by HMBC experiments are showed in table position of phenolic groups. Other long-range correlations established by HMBC experiments showed in table.



1,8-dihydroxy-3,5-dimethoxy-9*H*-xanthen-9-one

On the basis of above discussed spectral data of compound was characterized as methylbellifolin; 1,8-dihydroxy-3,5-dimethoxyxanthone which was further confirmed by comparison of its ^{1}H and ^{13}C-NMR data with the reported data[5].

Table 1

C/H	δC	Multiplicity (DEPT)	δH (J in Hz)	HMQC Correlation (H→C)
1	162.8	C	--	--
2	97.9	CH	6.35, d (2.2)	C-1, C-3, C-4, C-9a
3	167.4	C	--	--
4	93.1	CH	6.54, d (2.2)	C-2, C-3, C-4a, C-9a
4a	157.8	C	--	--
4b	145.4	C	--	--
5	139.9	C	--	--
6	120.4	CH	7.22, d (8.8)	C-4a, C-5, C-8
7	109.3	CH	6.71, d (8.8)	C-5, C-8, C-8a
8	154.1	C	--	--
8a	108.1	C	--	--
9	184.6	C	--	--
9a	102.8	C	--	--
3-OMe	56.0	CH_3	3.89, s	C-3
5-OMe	57.4	CH_3	3.96, s	C-5
1-OH	--	---	11.98, s	C-1, C-2, C-3, C-9a
8-OH	--	--	11.39, s	C-7, C-8

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