

Chemical investigation of epoxy-hydroxy-oxyacetyl bicyclo-ketone from *Tinospora* species

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ABSTRACT

The compound 4, 5-epoxy-7-hydroxy-6-oxyacetyl-2, 2, 6-trimethyl-bicyclo [4, 4, 0]-oct-7-ene-1-one has been isolated from *Tinospora cordifolia* and characterized by CI MS, I.R., U.V., ^1H NMR and ^{13}C NMR spectroscopic techniques.

Key words: *Tinospora cordifolia*, chemical analysis.

INTRODUCTION

Tinospora cordifolia, known as *Amrita* or *Guduchi*, is a large, glabrous, deciduous climbing shrub belonging to the family Menispermaceae¹. It is widely used in veterinary folk and ayurvedic system of medicine for its general tonic, anti-pyretic, anti-spasmodic, anti-inflammatory, anti-arthritis, anti-allergic, anti-diabetic, anti-leishmanial, analgesic and diuretic properties^{2, 3}. Literature survey revealed the presence of various classes of chemical constituents in *Tinospora* species viz., flavonoids⁴, steroids⁵, terpenoids⁶, alkaloids, lignans, saponins and furano diterpenes etc. In the present paper, we report the isolation and structural elucidation of sesquiterpene 4, 5-epoxy-7-hydroxy-6-oxyacetyl-2, 2, 6-trimethyl-bicyclo [4, 4, 0]-oct-7-ene-1-one from *T. cordifolia*.

MATERIAL AND METHODS

Plant Material

Tinospora cordifolia was collected from Meerut and authenticated by the Department of Botany of the Institute.

Extraction and Isolation. Air dried and powdered stems of the plant were Soxhlet extracted

with 95% ethanol. The extract was freed from solvent under reduced pressure to a thick brown mass. It was then partitioned between water and *n*-hexane to yield hexane soluble fraction. The defatted residue was further partitioned between water and *n*-butanol. The butanol soluble fraction was extracted by refluxing with CHCl_3 and $\text{CHCl}_3\text{-CH}_3\text{OH}$ (20%). The extract was purified by column chromatography and rechromatographed by MPLC using silica gel.

Instruments

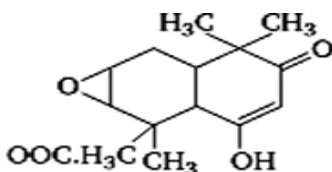
I.R. spectrum of the isolated compound was recorded on Perkin-Elmer 270 spectrophotometer in KBr pellets, CI-MS on Jeol SX-102 model mass spectrometer and UV on Perkin-Elmer Lambda 15-spectro- photometer. ^1H and ^{13}C NMR spectra were run on Bruker or Superior spectrophotometers using trimethyl silane as standard.

RESULTS AND DISCUSSION

The pure fraction collected from $\text{CHCl}_3\text{-CH}_3\text{OH}$ (19 : 1) gave colourless crystalline solid having melting point 183–185°C. The CI mass spectrum showed molecular ion peak at m/z 280 $[\text{M}]^+$ corresponding to the molecular formula $\text{C}_{15}\text{H}_{20}\text{O}_5$. Other peaks were observed at m/z 283

$[M+3H]^+$, 281 $[M+H]^+$, 264 $[M-16]$, 237 $[M-COCH_3]$, 221 $[OCOCH_3]^+$.

Infrared spectrum of the compound exhibited absorption bands at 1728 cm^{-1} (for $-OCOCH_3$), $3510-3450\text{ cm}^{-1}$ ($-OH$ group), 1650 cm^{-1} (α, β -unsaturated $>C=O$ group), 3015 cm^{-1} ($-CH$ aromatic), 2952 cm^{-1} ($-CH$ unsaturated), 1220 cm^{-1} (symmetrical stretching, ν_{sym}) and $950-840\text{ cm}^{-1}$ (ν_{asym}) for epoxide in the ring⁸. Ultraviolet spectrum showed major bands at λ_{max} 240 and 257 nm



indicating the presence of α, β -unsaturated carbonyl group with hydroxyl group at β -position⁹. The bathochromic shift of 30 nm with $AlCl_3$ in band I revealed the presence of $-OH$ group at C-7 position in the isolated compound. H^1 NMR (200 MHz, $CDCl_3$) spectrum of the compound exhibited three sharp singlets at δ 1.05, 1.16 and 1.25 integrating for three protons suggested the presence of three methyl groups in the molecule¹⁰. The signals in C^{13} NMR at δ 24.0, 24.5 and 28.6 assigned to three $-CH_3$ groups confirmed their presence in the

compound. The existence of carbonyl group as shown by infrared spectrum was supported by the sharp singlet at δ 2.06. Further, signals at δ 20.56 and 170.5 in H^1 and C^{13} NMR spectra confirmed the presence of acetate group at position C-6 in the molecule. The acetate group was again confirmed by the loss of an ion at m/z 237 and 221 from the molecular ion peak. One $-CH_3$ signals which occurred downfield at δ 1.28 is due to anisotropic effect of acetate group. It suggested that methyl group is attached to the same carbon to which acetate group is attached. A broad doublet at δ 2.77 and a broad singlet at δ 2.97 attributed to the protons of epoxide ring were supported by the appearance of signals at δ 56.8 and 54.7 for C-4 and C-5 carbon respectively in C^{13} NMR¹⁰.

The presence of tertiary hydroxyl group at C-7 as evidenced by I.R. spectrum was confirmed by the broad singlet at δ 3.90 in C^{13} NMR. The signals at δ 205.2, 126.4 and 122.5 revealed the presence of α, β -unsaturated ketone with hydroxyl group at β -position¹¹. All these spectral data (I.R., U.V., H^1 and C^{13} NMR) confirm the presence of three tertiary methyles, one acetate group, one epoxide ring, one tertiary hydroxyl group and α, β -unsaturated ketone group in the compound. Since this oxygenated derivative exhibited C-15 skeleton¹², so it may be assigned as following sesquiterpene.

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