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Isolation, purification and characterisation of 2, 7, (14), 10 bisabolatriene- 1,9,12 triol, a bisabolene type sesquiterpene isolated from *Curcuma longa* L.

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ABSTRACT

Several bisabolene types sesquiterpenes have been isolated, identified and reported from the genus *Curcuma*. This particular compound 2, 7, (14), 10 bisabolatriene- 1,9,12 triol was reported by Huneck et al., 1986; Uehara et al., 1990 [19-20] from *Curcuma xanthorrhiza*. So far as literature survey reveals our work is the first report of presence of 2, 7, (14), 10 bisabolatriene- 1,9,12 triol in *Curcuma longa* L. The compound was isolated, purified and chemically characterized by various standard spectroscopic methods. The acetylated derivative of the compound was also studied and analyzed spectroscopically.

KEYWORDS

Bisabolenes;
2,7, (14);
10 bisabolatriene- 1, 9, 12 triol;
Acetylated derivative;
Curcuma longa L.

INTRODUCTION

Curcuma longa L. is a perennial herb widely cultivated through out Southeast Asia, especially in India and China. Its rhizome have been used for centuries as a traditional herbal medicine in China, India and Southeast Asia for the treatment of cold, diabetes, rheumatism, liver ailments, parasitic infections, skin diseases, inflammation conditions and billary disorders[1,2]. Chemical investigation revealed that the rhizomes of *Curcuma longa* L. contained three main groups of compounds like curcuminoids, sesquiterpenoids and monoterpenoids etc[3,6-8] which are responsible for medicinal properties of this plant[9-11]. Sesquiterpenes and monoterpenes possess a variety of commendable biological activities such as antitumor, antioxidant, antinociceptive, antifungal and antibacterial activities[12-16].

EXPERIMENTALS

Plant collection

Whole plants of *C. longa* were collected in the month of July, 2010 from experimental garden of Department of Botany, University of Kalyani, and was identified in the Department of Botany, University of Kalyani, Nadia.

Isolation, purification and characterisation of the secondary metabolite

2.5 kg shade dried rhizomes of turmeric plant was

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powdered of approximately and extracted three times with 95% EtOH (each 500 ml, 48 h) at room temperature to give an extract of 375 gms. The extract was evaporated under reduced pressure and a solid residual mass was obtained. The above obtained residual sample was purified by repeated preparative thin layer chromatography using different solvent systems 1. Methanol (5%): benzene (95%) and solvent system 2. Chloroform (60%): benzene (30%): acetic acid (10%). Four homogeneous spots were collected in solvent system 2, having Rf values of 0.8, 0.7, 0.65 and 0.6 respectively. The sample with Rf value 0.65 was positive in Liberman's Burchard test[17] and gave purple colour indicating terpenoid nature of the compound and had melting point of 82^{0}C. Subsequent analysis of the sample was performed by various spectroscopic techniques like UV spectroscopy, FT-IR spectroscopy and High Resolution Mass spectroscopy.

Acetylation of the sample

20 mg of the isolated terpene was dissolved in 2 ml of spectral grade chloroform and the compound got solubilised in 3 ml of pyridine. When the pyridine got fully dissolved in the reaction mixture, then 3.6 ml of acetic anhydride was added to this reaction mixture and the reaction mixture was kept at 0^{0}C for 12 hours in dark. The acetylated product was spotted along with the non acetylated sample on preparative TLC using solvent system, chloroform: benzene: acetic acid = 60:30:10. Difference in the Rf value (0.5) of the acetylated sample indicated a change in it.

RESULTS AND DISCUSSION

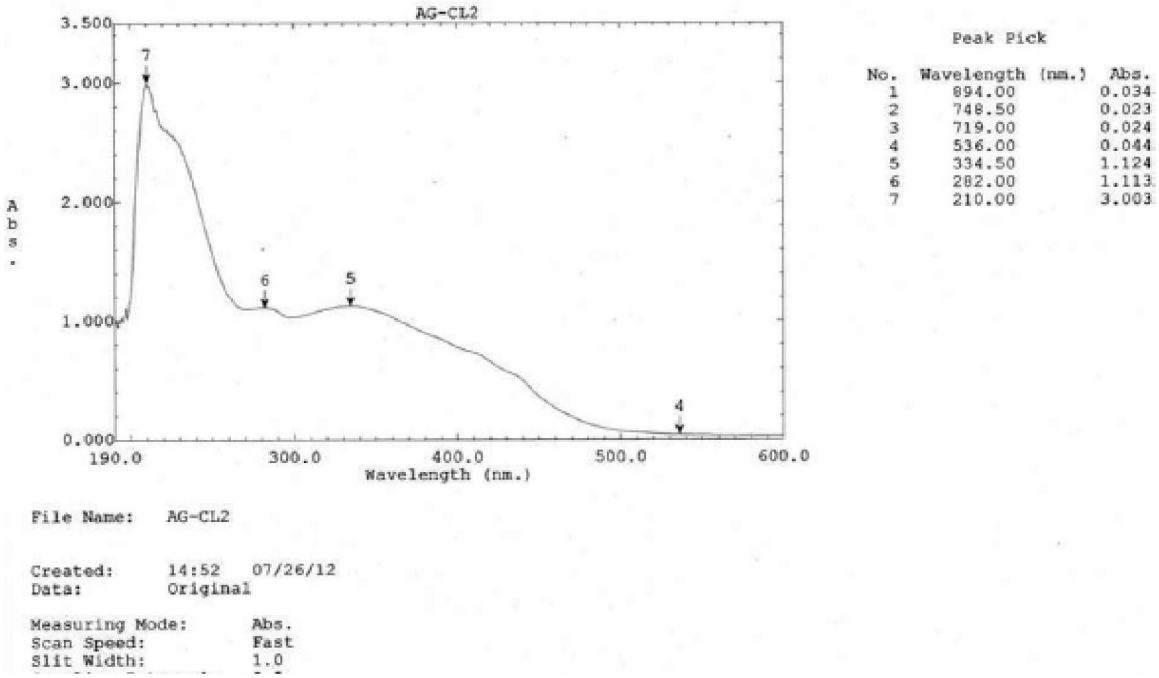
Chemical characterization of the isolated sample

The compound was yellowish brown in colour and was soluble in spectral grade methanol (Brand-Spectrochem). The melting point of the sample was 82^{0}C and it turned purple in Liberman's Burchard test[17].

UV spectroscopy of the isolated sample

The methanolic spectrum of the sample showed λmax at 210 nm, Absorbance =3.0030 (Spectrum 1).

Spectrum 1 = UV spectrum of 2, 7, (14), 10 Bisabolatriene- 1,9,12 triol



IR (FT-IR) spectroscopy of the isolated sample

The IR spectrum[18] of the sample showed n- (cm-1): 3272.33, 2923.14, 2851.62, 1716.12, 1602.34, 1574.96, 1513.88, 1446.12, 1378.49, 1262.37, 1168.89, 1140.32, and 1104.19 (Spectrum 2). IR spectra were recorded on a Perkin-Elmer Spectrum-1 instrument using KBr disks, chloroform solution or as neat.

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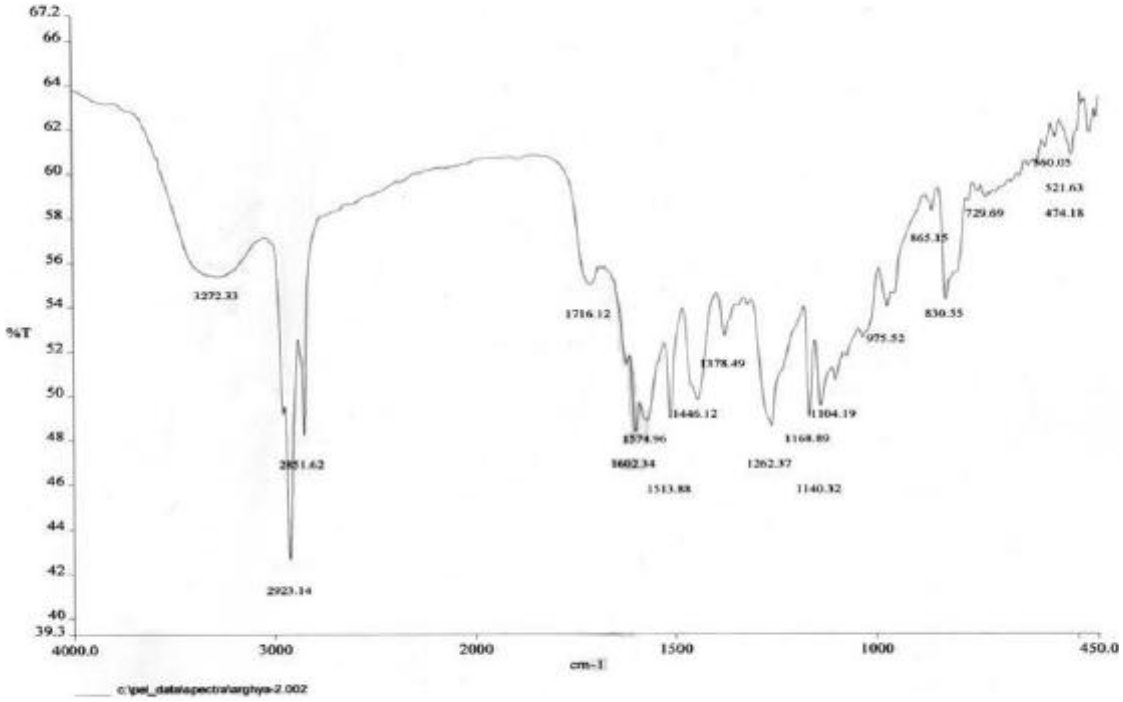
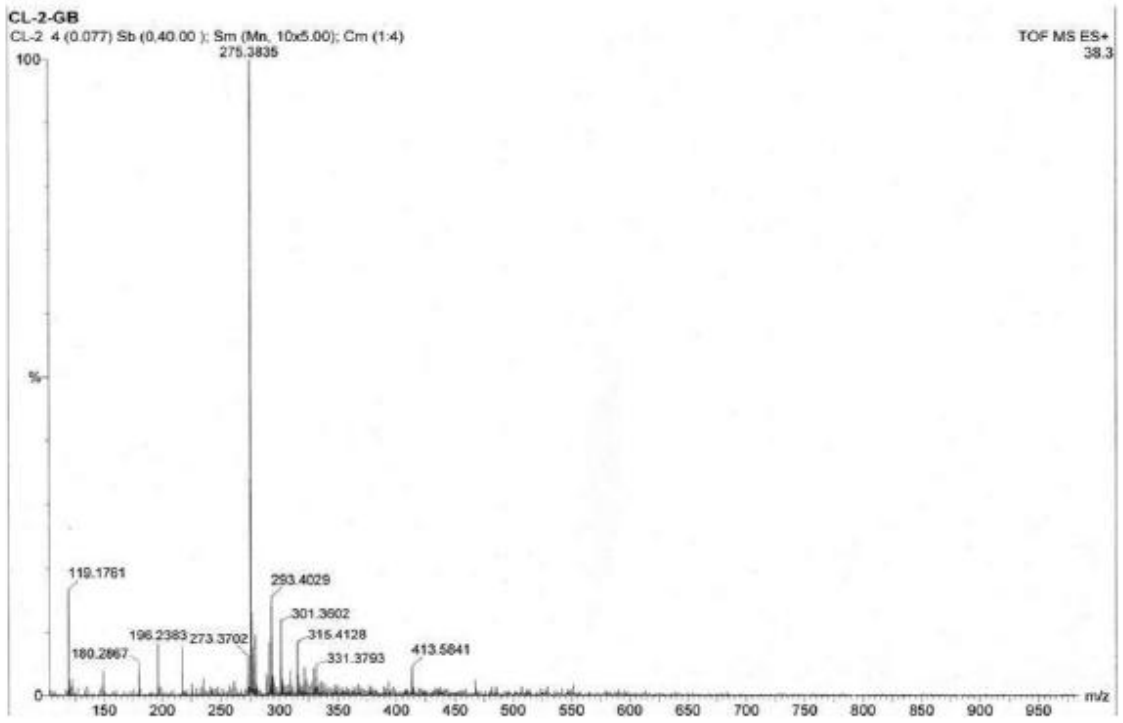
High resolution mass spectroscopy of the isolated sample

High Resolution Mass spectroscopy (HRMS) spectrum was performed in a JEOL-JNM mass spectrometer. The mass of the sample was noted as to be (m/z ratio) 252.3835 (Spectrum 3).

Spectrum 2 = FT-IR spectrum of 2, 7, (14), 10 Bisabolatriene- 1,9,12 triol



Spectrum 3 = High Resolution Mass spectrum of 2, 7, (14), 10 Bisabolatriene- 1,9,12 triol (The m/z ratio of the compound is 252.3835 + 23 for m/z ratio of sodium ion)



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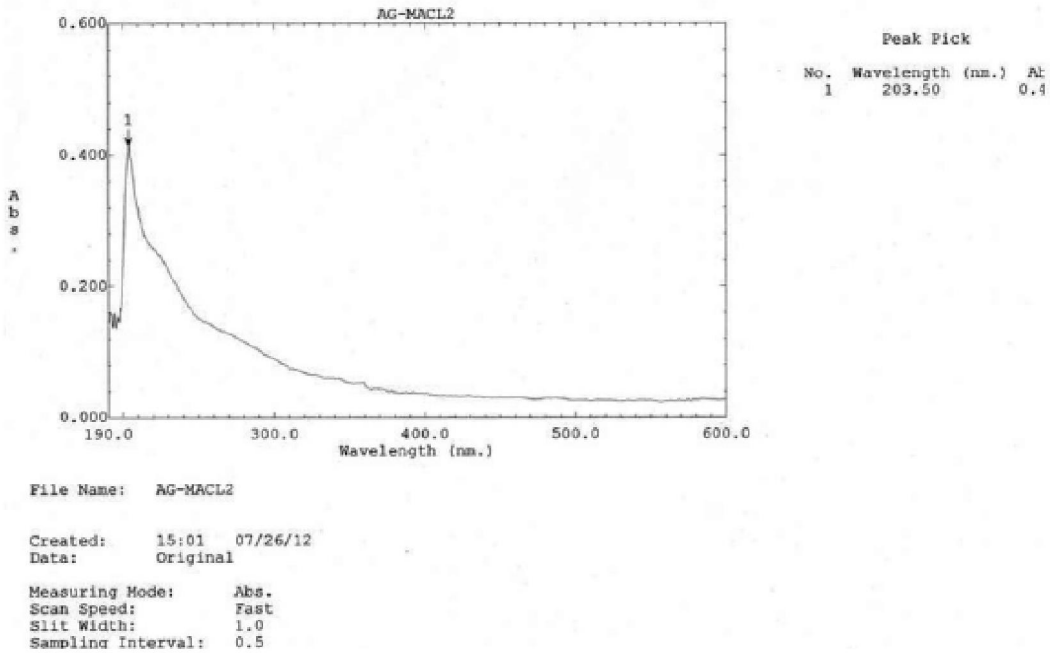
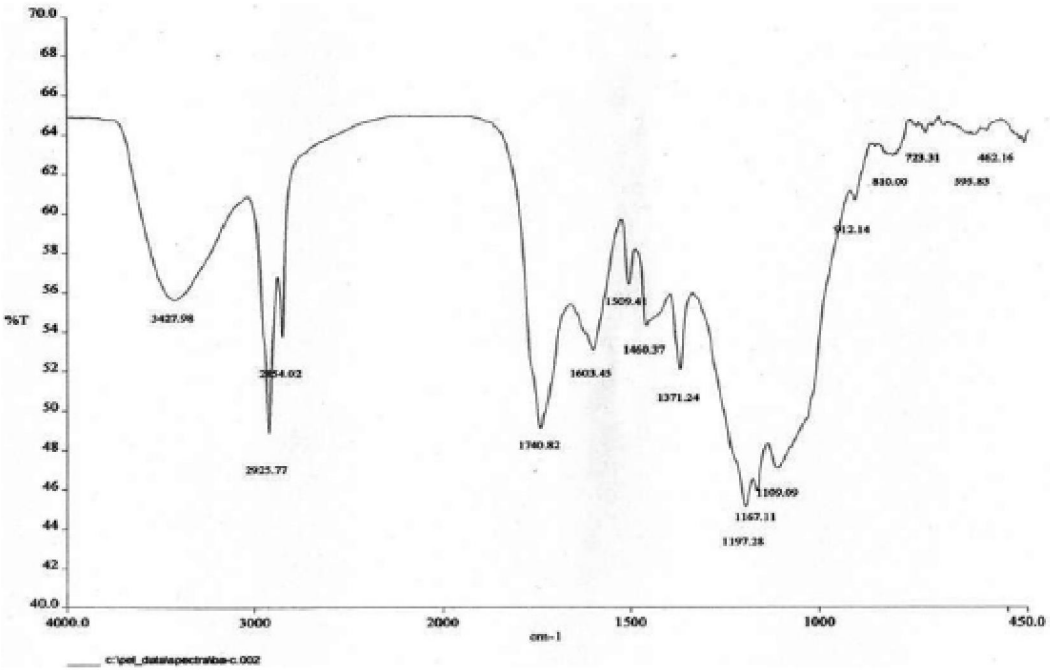
Spectrum 4 = UV spectrum of the acetylated product of 2, 7, (14), 10 Bisabolatriene-1,9,12 triol



Spectrum 5 = FT-IR spectrum of the acetylated product of 2, 7, (14),10 Bisabolatriene- 1,9,12 triol



Chemical characterization of the acetylated derivative

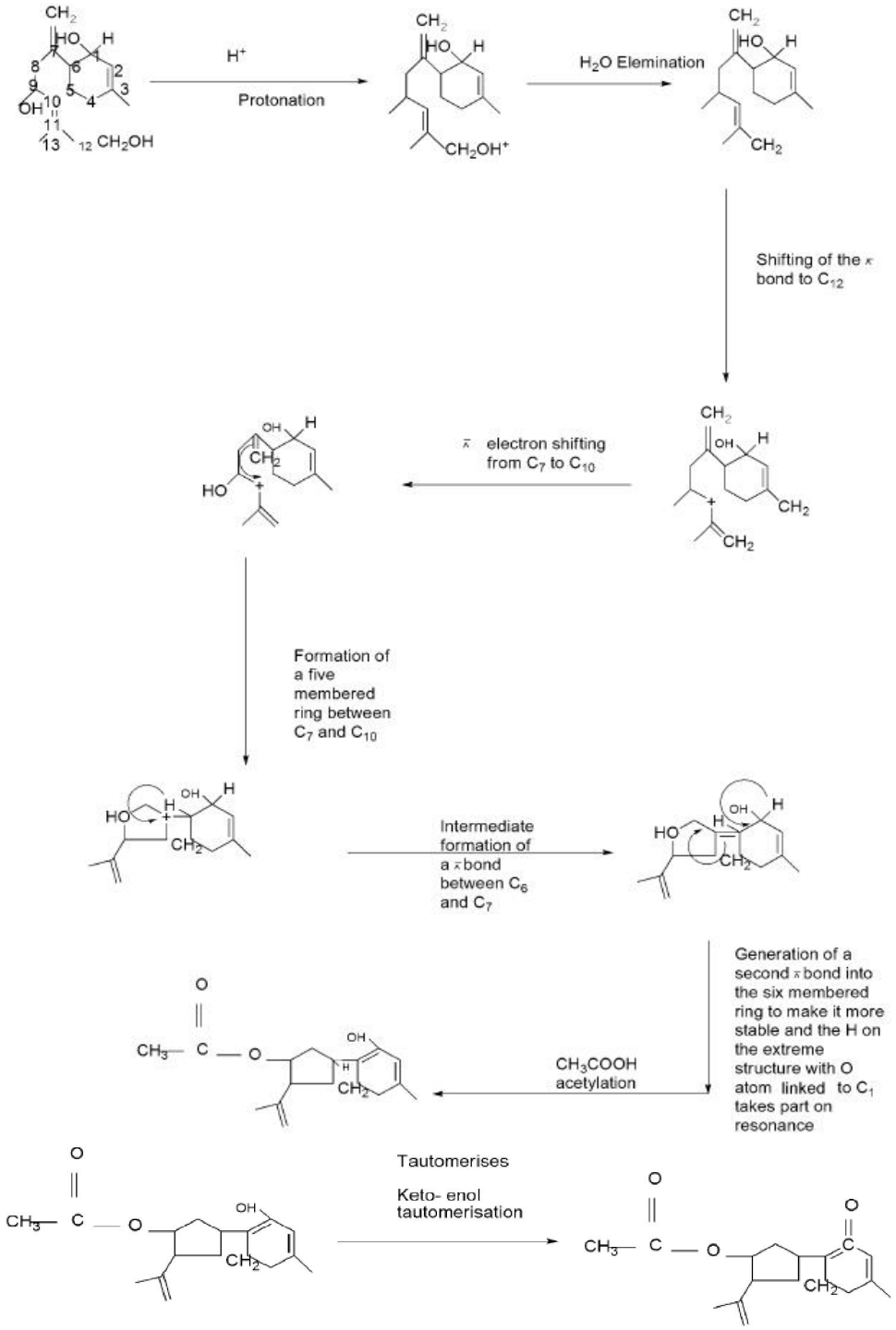
The acetylated derivative was dark brown in colour and was soluble in spectral grade methanol. (Brand-Spectrochem). The melting point of the sample was 95^0C and it turned purple in Liberman's Burchard test[17]. The Rf value of the acetylated derivative was noted as 0.5 using solvent system, chloroform: benzene: acetic acid = 60:30:10.

UV spectroscopy of the acetylated derivative

The methanolic spectrum of the acetylated product showed λmax at 203.50 nm, Absorbance =0.4139 (Spectrum 4).

IR (FT-IR) spectroscopy of the acetylated product

The IR spectrum[18] of the acetylated sample showed n- (cm-1): 3427.98, 2925.77, 2854.02, 1740.82, 1603.45, 1509.41, 1460.37, 1371.24, 1197.28, 1167.11, and 1109.09 (Spectrum 5). The presence of absorbtion peak at 1740.82 nm designated presense of a saturated cyclic ketone group with five membered rings in the compound. IR spectrum was recorded on a Perkin-Elmer Spectrum-1instrument.



Scheme 1 : Pathway of acetylation of the parent compound.

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CONCLUSION

Finally the compound was identified as 2, 7, (14), 10 Bisabolatriene- 1,9,12 triol and its presence is reported first in *Curcuma longa* L. This is the same compound reported by Huneck et al., 1986; Uehara et al., 1990[19,20] from *Curcuma xanthorrhiza*.

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