



# Structural and optical studies of 7-methoxy-8-(3-methylbut-2-enyl)-2-chromenone (osthol), a plant based coumarin



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## ABSTRACT

Osthol, [7-Methoxy-8-(3-methylbut-2-enyl)-2-chromenone], a simple Coumarin (Mol. Formula =  $C_{15}H_{16}O_3$ ), was isolated from petroleum ether extract of the root parts of the plant *Prangos pabularia*. The compound was characterized by MS,  $^1H$  NMR,  $^{13}C$  NMR, IR, X-ray diffraction (XRD), scanning electron microscopy (SEM), UV–vis spectroscopy and photoluminescence (PL) measurements. Structural analysis confirmed the monoclinic structure for the system. The exploration of SEM predicted the compound to be comprising of micro crystals with irregular morphology. From UV–vis spectroscopy, high transmission was displayed by these crystals in the entire visible range. The optical band gap ( $E_g$ ) was found to be around 3.39 eV and the compound exhibited indirect allowed transitions. PL data showed good emission at certain wavelengths in visible region. The present properties of the compound can be explored for optoelectronics.

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## 1. Introduction

Rapid development of organic electronics in present day world is a result of significant physical and chemical properties displayed by organic semiconductors [1–9], in addition to their mechanical and thermal properties, ensuring their application in the field of flexible electronics, chemical stability and solubility being the best feature permitting the incorporation of functionalities through atomic or molecular design. Possible application of polycyclic aromatic hydrocarbon skeleton bearing organic compounds like, naphthalene, anthracene, perylene, fluorene, carbazole, pyrene, etc., in Organic-Light-Emitting Diodes (OLEDs) has already been reported [10–13] and have been mostly used as efficient electron/hole-transporting materials or host emitting materials in OLED. The synthesis and photo-physical properties of pyrene based, multiple conjugated, fluorescent light-emitting materials are also reported [10].

Highly pure single crystals of organic materials can provide the a very well information pertaining to the structure–property relationship in organic semiconductors thereby leading to further development of organic electronics technology. The performance of organic materials in electronic devices is primarily based on their

purity and ordered molecular packing thereby facilitating the study of structure–property relationship.

The nonlinearity of the organic material is increased due to the delocalized  $\pi$ -electron system between donor and acceptor groups, which enhances their asymmetric polarizability [8–10]. In continuation to our previous studies on coumarins [14–18], we report hereby the structural and optical properties of osthol, one of the major compounds from the root parts of the plant *Prangos pabularia*.

## 2. Experimental

The compound osthol having molecular formula  $C_{15}H_{16}O_3$  and molecular mass 244 was isolated from petroleum ether extract of the roots of the plant *P. pabularia* by column chromatography using silica gel as adsorbent. The structure of this compound was confirmed by comparison of its spectral data [ $^1H$  NMR,  $^{13}C$  NMR, Ultra-Violet (UV), Infrared (IR) and Mass spectroscopy (MS)] with the data values reported in literature [19,20].

Melting points (mp) were determined in centigrade scale in one end open capillary on Buchii 570 melting point apparatus. The  $^1H$  and  $^{13}C$  NMR spectra were recorded by Bruker 500 (125 MHz) instrument using TMS as internal standard and  $CDCl_3$  as solvent. The UV spectra were recorded on a Double beam UV–vis spectrophotometer (PG Instrument T80). The Column chromatography was carried out with Merk silica gel (60–120 mesh and 100–200 mesh). Aluminium sheets, precoated with silica gel 60

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$F_{254}$  ( $20 \times 20$  cm, 0.2 mm thick; E-Merck) were used for TLC to check the purity of the compounds and were visualized under UV light (254 and 366 nm) followed by ceric sulfate as spraying reagent. The crystalline structure of this compound was analyzed by a PW 3050 X-ray diffractometer with  $\text{Cu K}\alpha$  radiation with wavelength ( $\lambda$ ) = 1.54060 Å and a graphite monochromator. IR spectrum was recorded with Perkin Elmer RX-1 FT-IR Spectrophotometer. SEM images were carried out using Hitachi SEM Model S-3600N. Photoluminescence (PL) experiments were carried out by using Perkin-Elmer LS-55 spectrometer at room temperature with He–Cd laser as excitation source. PL spectra for the samples were detected with a grating mono-chromator and a standard CCD detection system.

### 3. Results and discussions

#### 3.1. Structural analysis

Fig. 1 shows the molecular structure of osthol (as established by IR, MS and NMR data). The melting point of the compound was observed at around 82–84 °C

In its  $^1\text{H}$  NMR spectrum (Fig. 2), osthol displayed down field resonance signals due to olefinic protons at  $\delta$  6.18 (1H, *d*,  $J=9.1$  Hz, H-3), 8.14 (1H, *d*,  $J=9.1$  Hz, H-4) and a side chain olefinic proton at  $\delta$  5.26 (1H, *t*, H-2'). The down field resonance signals at  $\delta$  7.38 and 7.10 were displayed due to aromatic protons (1H, *d*,  $J=8.5$  Hz, H-5) and (1H, *d*,  $J=8.5$  Hz, H-6), respectively, besides a signal at  $\delta$  3.93, due to three singlet methoxy protons at C-7. The signals at  $\delta$  1.84 (3H, *s*), 1.81 (3H, *s*) were assigned to C-3' gemdimethyl protons.

In the  $^{13}\text{C}$  NMR spectrum (Fig. 3), 15 carbon signals were observed. Nine carbons were assigned for a coumarin nucleus at  $\delta_{\text{C}}$  160.7 (–OC=O, C-2), 112.6 (=CH, C-3), 143.4 (–CH, C-4), 117.5 (C, C-4a), 126.2 (C, C-5), 112.4 (C, C-6), 159.9 (–C–O, C-7), 107.2 (C, C-8), 152.3 (C, C-8a). The rest of the other carbon signals suggested the presence of a prenyl group at  $\delta_{\text{C}}$  21.8 (–CH<sub>2</sub>–, C-1'), 121.0

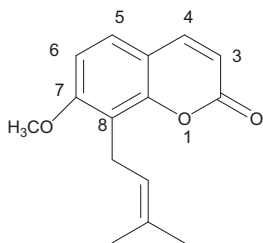


Fig. 1. Structure of osthol.

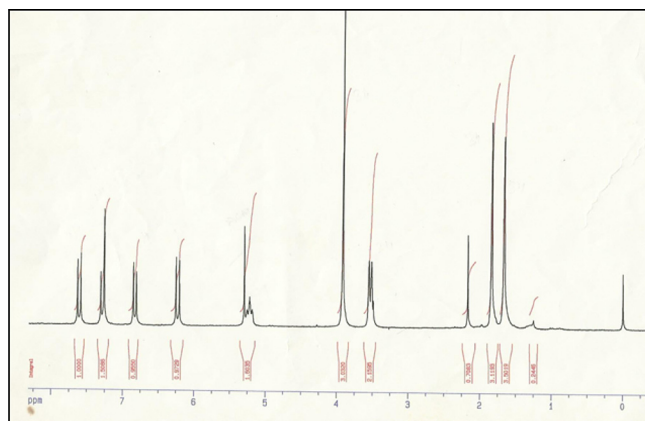


Fig. 2.  $^1\text{H}$  NMR of osthol.

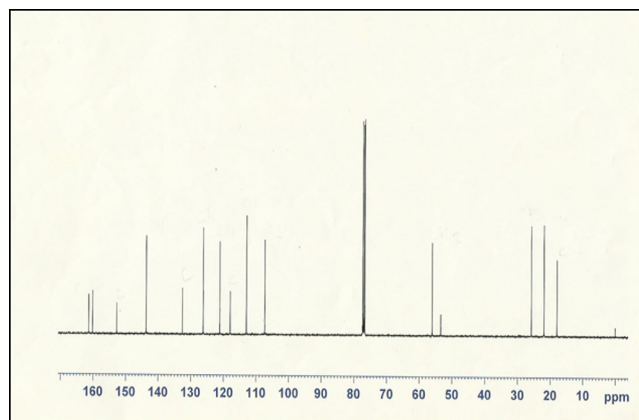


Fig. 3.  $^{13}\text{C}$  NMR of osthol.

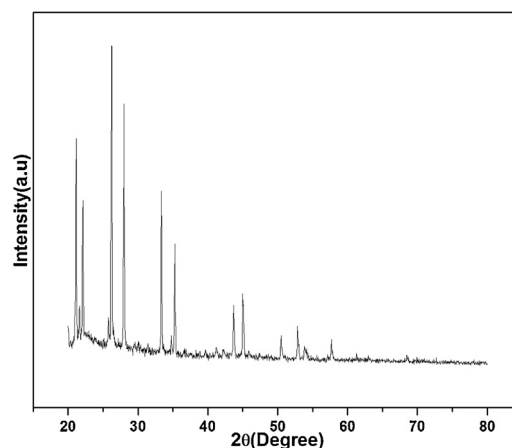


Fig. 4. XRD pattern of osthol.

(–CH=, C-2'), 132.3 (=C, C-3'), 25.6 (CH<sub>3</sub>, C-3'), 17.9 (CH<sub>3</sub>, C-3') and 56.1 (7-methoxy).

The powder X-ray diffraction (XRD) of osthol compound is shown in Fig. 4. The crystalline character of the system is quite clear from the observed XRD pattern. The sharp peaks of the sample occur at specific Bragg's angles.

The obtained diffraction pattern of the sample was indexed using Powder-X software. Calculation of cell parameters reveals that osthol crystallizes in a monoclinic structure with four molecules per unit cell having space group  $P_{111}$ . The lattice parameters for the system are:  $a=16.84198$  Å,  $b=6.15767$  Å and  $c=25.12480$  Å,  $\alpha=90$ ,  $\beta=90$ ,  $\gamma=120^\circ$ . The crystallographic data are in accordance with similar systems reported in literature [21–23].

SEM image of osthol is shown in Fig. 5. The micro crystals have grown with irregular morphology with a sluggish or blurred quality. The possible reason behind this observation may be the nucleation centers and the ordering of carbon molecules [24], due to high evaporation rate at low temperature, leading to irregular morphology with dislocation/defect centre [25].

FT-IR spectrum of osthol is shown in Fig. 6. Various characteristic bands with varying intensities were observed. The compound displayed a band with strong intensity at around  $1722\text{ cm}^{-1}$ , characteristic of  $\alpha,\beta$ -unsaturated- $\delta$ -lactone. The bands at around  $1600$  and  $1492\text{ cm}^{-1}$  are due to the presence of aromatic moiety and bands at  $1383$ ,  $1386\text{ cm}^{-1}$  are because of gemdimethyl. The band at  $2973\text{ cm}^{-1}$  was assigned to an asymmetric C–H stretching of methyl groups and another at  $2843\text{ cm}^{-1}$  was assigned to its symmetric one. The compound also showed bands at  $1465\text{ cm}^{-1}$  and  $1380\text{ cm}^{-1}$  due to asymmetric and symmetric bending vibration of

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