

Isolation and Identification of Methyl cinnamate from Syrian *Ocimum Basilicum*

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Abstract

A phytochemical study of *Ocimum Basilicum*, a plant collected from AL-Hasakah area in Syria, led to isolate and identified the methyl cinnamate **1** from the chloroform extract. The purification, the isolation and the structural identification of this compound was achieved by means of the chromatographic (CC and TLC) analysis and the spectroscopic: nuclear magnetic resonance with different applications (¹H-NMR, ¹³CNMR, COSY, HMQC, HMBC) and FT-IR spectroscopy.

Keywords: *Ocimum basilicum*, Methyl cinnamate, ¹H-NMR, ¹³CNMR,

1. Introduction:

Lamiaceae is a big family with 252 genus and 6700 spcies. it is known as wealth species with medicinal properties, many of these species are common in Mediterranean region. The genus *ocimum* consists of more than 150 species with a wide diversity in growth characteristics , leaf, size, and flower color [1].

O. basilicum, is an annual plant of the *Ocimum* genus commonly known as holy basil, clove basil (wild basil/East India basil) and sweet basil, respectively, are frequently cultivated in several countries of East Asia, Europe, America and Australia for the production of essential oils. Traditionally, these *Ocimum* species have been extensively utilized in food and perfumery industries. Fresh leaves of basil plant are used as an ingredient in various dishes and food preparation, especially in the Mediterranean cuisine [2-4].

The aerial parts of the plants are considered as antispasmodic, stomachic and carminative in native medicine. Recently the potential uses of *O. sanctum*, *O. gratissimum* and *O. basilicum* essential oils particularly as ntioxidate and antimicrobial agents have also been explored [5-8]. This study describes the isolation and structural determination of the methyl cinnamate from *Ocimum Basilicum* cultivated in Syria.



Figure 1.: photo of *Ocimum basilicum*

2. Materials and Methods:

2.1 General:

- 1- Melting points were measured on an Electrothermal melting point apparatus.
- 2- Rotational evaporator (Heidolph) from Germany.
- 3- ¹H-NMR, ¹³C-NMR, and IR spectra were recorded on Bruker Ultra Shield 400MHz and Jasco FT-IR 410 respectively.

2.2 potanic material and Extraction method of the compound 1:

The green parts of *Ocimum basilicum* were collected from AL-Haskah in Syria in 2010, and air-dried (500 g) were extracted with MeOH in soxhlet. The extracts were combined and concentrated under low pressure to give 45 g of extract I. The residue from extract I. was extracted once with CHCl₃ and the extract concentrated under vacuum

to give 11 g of extract II . 2 g from extract II were loaded on chromatographic column (2 cm diameter, 120 cm. long) over silica gel (230 – 400 mesh, ASTM).

The column was eluted successively with: n-hexane (300 ml.), n-hexane/ chloroform (30 : 70, 500 ml.) and chloroform (500 ml.).

Methyl cinnamate : was obtained from the latter fraction II, purified on preparative TLC by using mixture of CHCl_3 /Hexane: 70 : 30 ,the R f = 0.48), it is a yellow crystalline solid, its melting point 35-38°C.

3. Results and discussion:

3.1 structure identification of Methyl cinnamate:

The structural determination of the of Methyl cinnamate based on the usual spectral methods. The IR spectrum shows a broad band at 2925 cm^{-1} (C-H stretching), (C=O esters) at 1755 cm^{-1} and three medium bands at 1629 cm^{-1} (C=C stretching), 1375 cm^{-1} (CH bending) and (C-O-C vibration bond) at 1222 cm^{-1} .

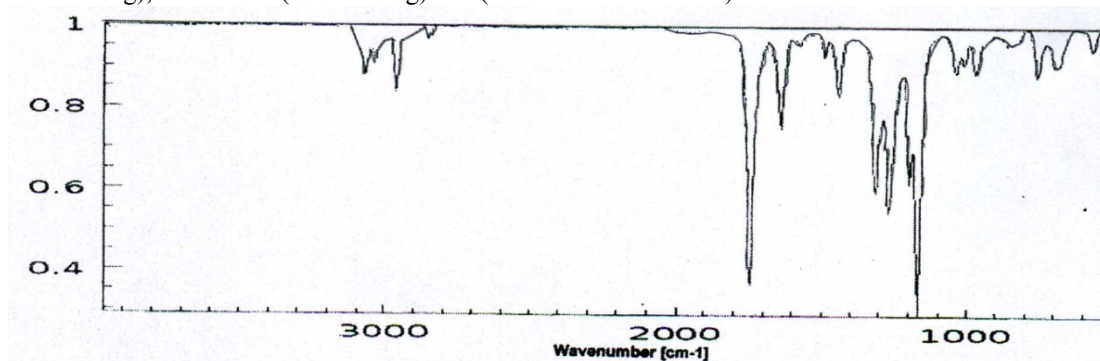


Figure 2: IR of Methyl cinnamate Ethyl benzoate in KBr

The ^{13}C -NMR exhibits 8 absorption signals indicating the presence of 10 carbon atoms in the molecule, these absorption indicate the presence of Aromatic carbones in the compound **1**(Table 1, Figure 3).

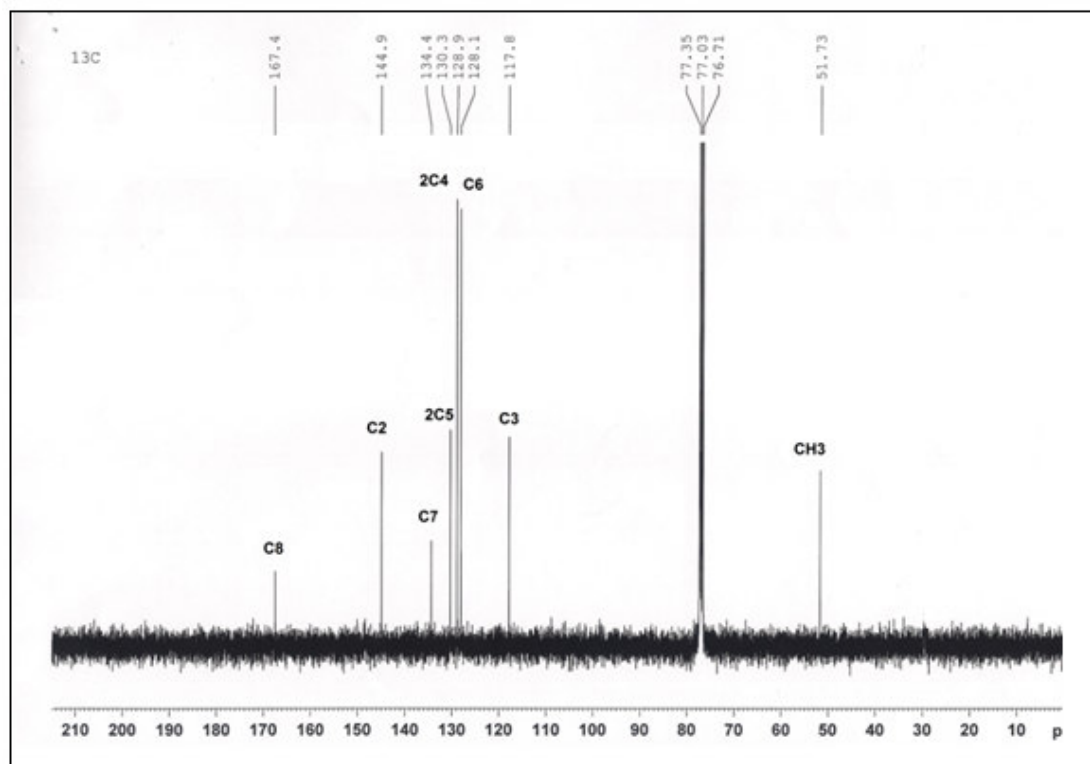


Figure 3: ^{13}C -NMR Spectrum of Methyl cinnamate (100 MHz, CDCl_3 , $\delta_{\text{TMS}} = 0\text{ ppm}$)

The analysis of the DEPT -135 spectrum, indicate the presence of one primary carbon, 3 secondary carbons, 2 tertiary carbons and 2 quaternary carbons (Figure 4).

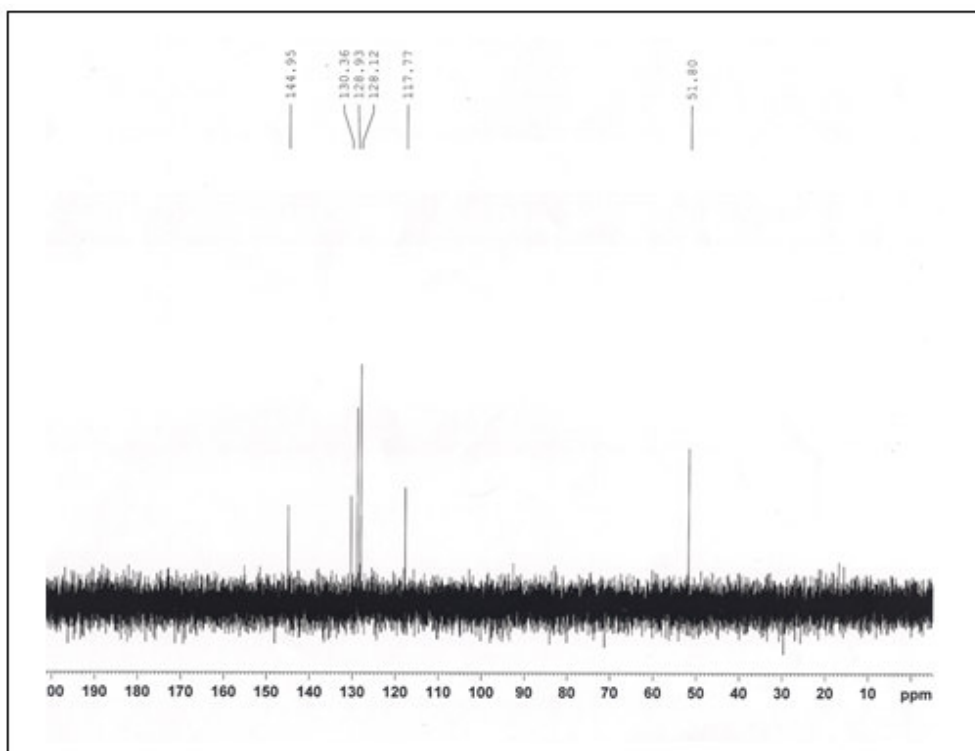


Figure 4: DEPT- 135 Specter of Methyl cinnamate (100 MHz, CDCl₃, δTMS = 0 ppm)

The ¹H-NMR spectrums shows 5 absorptions indicating the presence of 10 hydrogen atoms in the molecule **1**: one isolated absorption at 3.75 (3H, s), two olefin protons at 6.47 (1H, d, J= 16Hz, H-3) and at 7.73 (1H, d, J= 16Hz, H-2), and five aromatic protons: 7.74 (2H, d, J= 6Hz, H-4, H-4'), 7.55 (2H, d, J= 6 Hz, H-5, H-5'), 7.42(1H, t, J= 6Hz, H-6), see (Table 1, Figure 5).

Table 1: chemical shift (¹H-NMR and ¹³C-NMR) of compound **1**

N	δ _H (ppm)	δ _C (ppm)
1	3.75 (3H, s)	51.73
2	7.73 (1H, d, J= 16 Hz, H-2)	144.91
3	6.47 (1H, d, J= 16 Hz, H-3)	117.82
4, 4'	7.55: (2H, d, J= 16 Hz, H-4, H-4')	128.91
5, 5'	7.55 (1H, d, J= 6 Hz, H-5, H-5'),	130.32
6	7.42 (1H, d, J= 6 Hz, H-6),	128.10
7	-----	134.41
8	-----	167.47

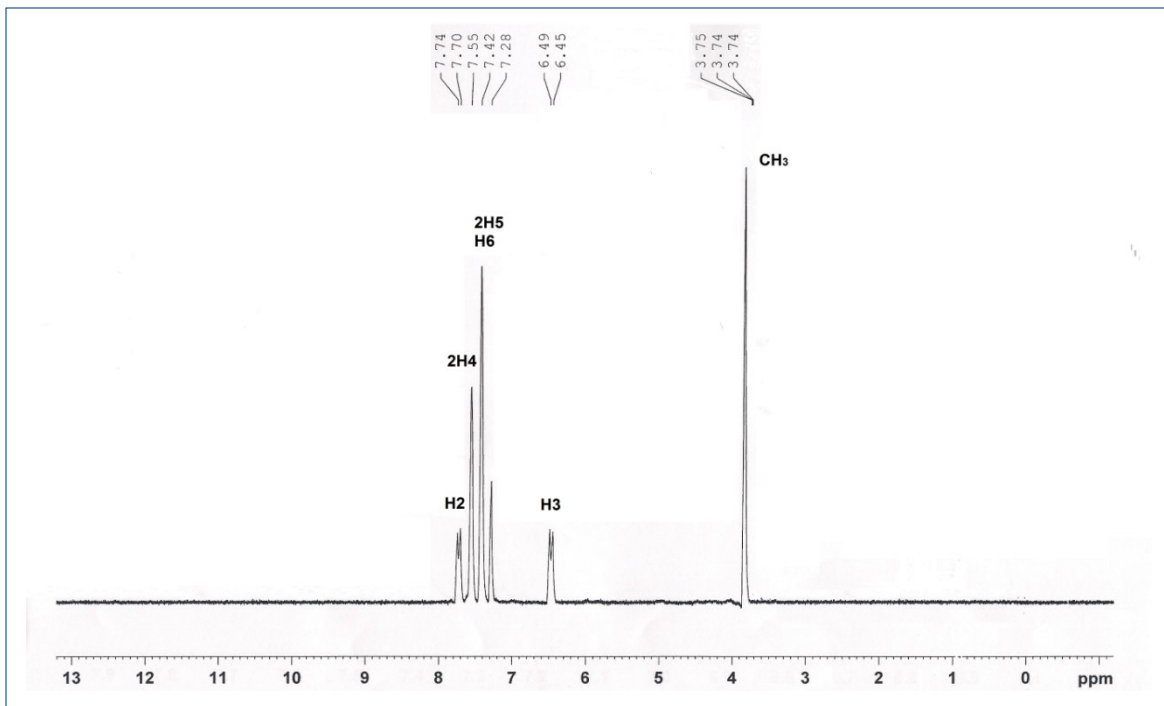


Figure 5: $^1\text{H-NMR}$ Spectrum of Methyl cinnamate (400 MHz, CDCl_3 , $\delta_{\text{TMS}} = 0$ ppm)

we can indicate from the COSY spectrum (Figure 6), the presence of one (spin – spin) coupling system between the proton at ($\delta_{\text{H}} = 7.73$ ppm, H-3) and the proton at ($\delta_{\text{H}} = 6.47$ ppm, H-2), we can confirm that the two protons are olefin protons in trans position, from the value of the coupling constant (16Hz) and the chemical shifts in the $^1\text{H-NMR}$ Spectrum [9].

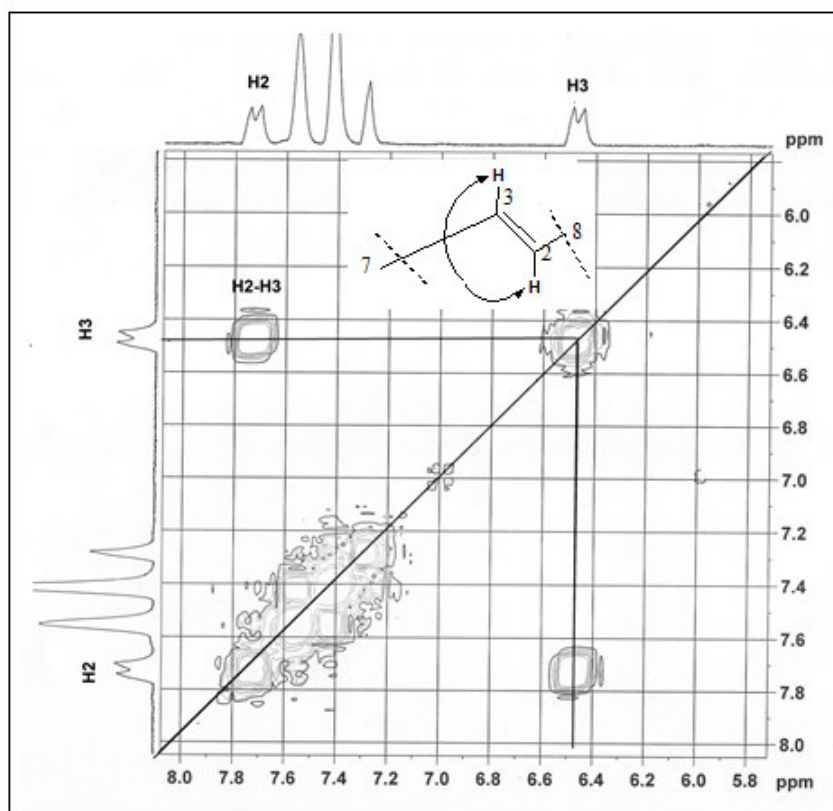


Figure 6: COSY Spectrum of Methyl cinnamate (400 MHz, CDCl_3 , $\delta_{\text{TMS}} = 0$ ppm)

we determinate from the HMQC spectrum, the heteroatom correlations between hydrogen systems and the carbon atoms carrying these protons (Figure 7,8) (9).

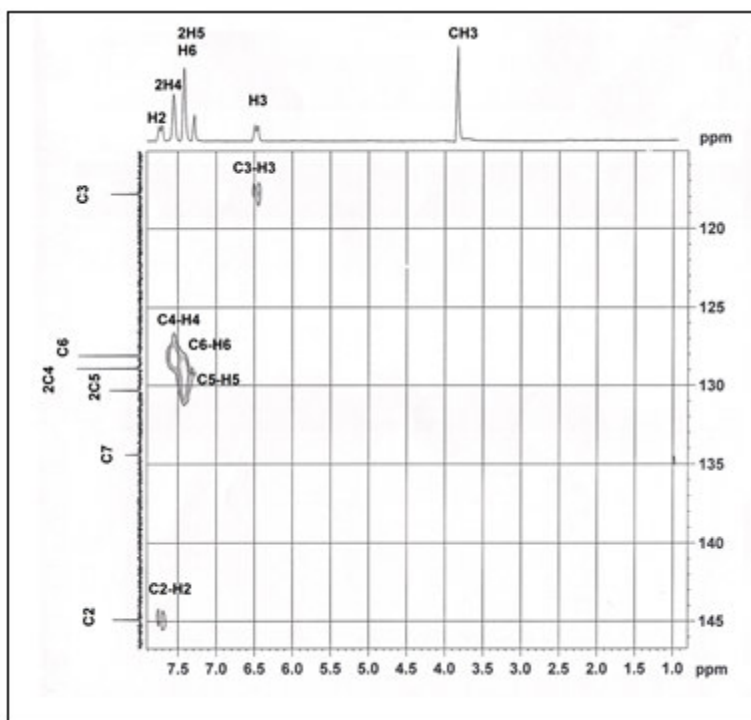


Figure 7: HMBC Spectrum of Methyl cinnamate (400 MHz, CDCl₃, $\delta_{TMS} = 0$ ppm)

To determine the hetero-atoms correlation (J^2 , J^3 , J^4 , J^5) for obtaining the detail of the skeleton of compound **1**, we analysed the HMBC spectrum (figure 8 and 9), so the correlation between the H₂ and C₄, 4' and C₈, and the H₃ correlate with C₇, so we can attach the olefin group with ester group from one side and with aromatic cycle from other side, in addition the correlation between the H₇ and the C₃ confirm this suggestion.

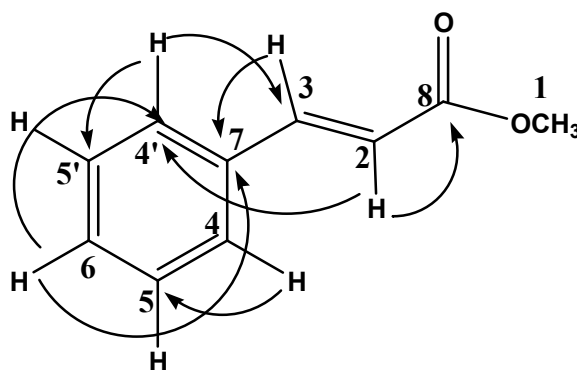


Figure 8: HMBC correlations of compound **1**

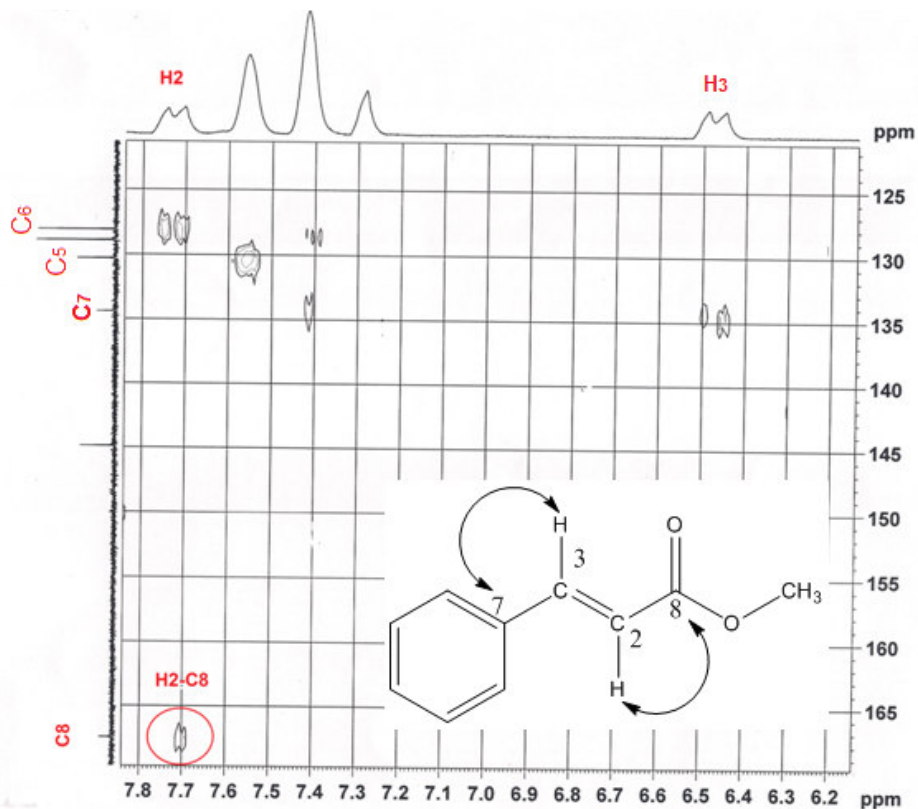
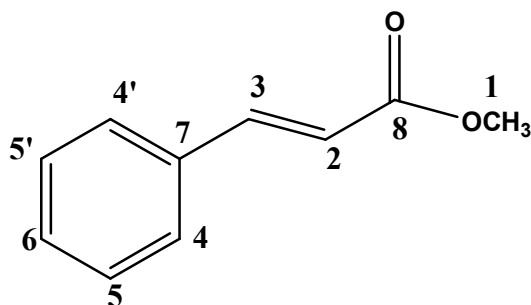


Figure 9: HMBC correlations of compound 1

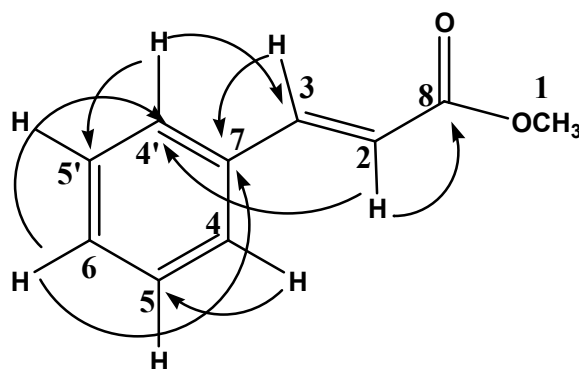
So we can suggest the structure of compound 1 as: (E)-methyl-3-phenyl acrylate or: (E)-methyl methyl cinnamate



(E)-methyl-3-phenyl acrylate
 Or (E)-methyl cinnamate

Table (2):, DEPT, COSY and HMBC data of compound 1

C	DEPT (135-90)	COSY δ_H (ppm)	HMBC
1	CH ₃		-----
2	CH	7.73	C-4', C-4, C-8
3	CH	6.47	C-7
4, 4'	CH	-----	C-5, C-5'
5, 5'	CH	-----	
6	CH	-----	C-4', C-7
7	C	-----	-----
8	C	-----	-----



4. References:

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