



ISSN NO. 2320-5407

Journal homepage: <http://www.journalijar.com>  
Journal DOI: [10.21474/IJAR01](https://doi.org/10.21474/IJAR01)

INTERNATIONAL JOURNAL  
OF ADVANCED RESEARCH

## RESEARCH ARTICLE

## ISOLATION AND CHARACTERIZATION SOME ORGANIC COMPOUNDS FROM BRECKLAND THYME.

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### Manuscript Info

### Abstract

#### Manuscript History:

Received: 18 March 2016  
Final Accepted: 22 April 2016  
Published Online: May 2016

#### Key words:

TLC, Thyme, Gas chromatography,  
Plant extract.

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This study includes extraction of volatile oil from thyme plant. Thin layer chromatography (TLC) was used to isolate some active organic compounds from this oil. Ethyl acetate: Toluene 5: 95% was used as mobile phase, TLC chromatogram appears more than five zones, and four of these zones were identified and characterized by different spectroscopic methods UV – VIS., I.R, HNMR Spectra besides other analytical methods called gas chromatography (G.C).

Results obtained indicate clearly that the following compounds, Carvacrol, p-Cymene, Camphor and thymol are present in the following percent 30%, 3%, 0.9%, 6.5% respectively are present in thyme oil besides other unknown compounds.

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### Introduction:-

Thyme has a long history of use in natural medicine in connection with chest and respiratory problems including coughs, bronchitis and chest congestion [1]. Only recently however, have researchers pinpointed some of the components in thyme that bring about its healing effects [2].

For thousands of years, herbs and species have been used to help preserve foods [3] protect them from microbial contamination, now research shows that both thyme and basil contain constituents that can both prevent contamination and decontamination previously contaminated foods [4].

Oil of thyme is derived from thyme, also known as thymus vulgaris [5]. The perennial herb, a member of the main family is used in aromatherapy, cooking, potpourri, mouth washes and elixirs, as well as, added to ointments [6]. Thyme has also a number of medical properties, which is due to the herb's essential oil [7].

The medical properties of thyme oils (which are extracted through steam distillation of fresh flowers and leaves) are due to their component Acne, Anticancer, Antispasmodic, Antirheumatic, Antiseptic, Bactericidal, tonic, cordial, carminative, insecticide stimulant, yeast killer and others [8].

The aim of present paper was focused on isolation and characterization active organic compounds in thyme.

### Experimental:-

Dry plant thyme was obtained from market, it is identified by Education College for pure science, university of Anbar, it was air dried and packed in plastic containers.

**Extraction of thyme oil:-**

In 1 liter a round bottomed flask, 200 cm<sup>2</sup> of distilled water was added to 40 gm of plant. The extraction process was carried out for 3.5 hours, and then the volatile oil was separated from the aqueous phase with ethers and then dried with Na<sub>2</sub>SO<sub>4</sub> anhydrous. Filtrate, evaporate in water bath at 40 °C the oils obtained were stored in dark bottle.

**Thin –layer chromatography:-**From different solvents, we choice the mixture of ethyl acetate and toluene in percent 5% and 95% respectively, as mobile phase while suitable plastic paper coated with silica gel was chosen as a stationary phase. The chromatogram shows five zones, each zone have been scratched, isolate and dissolve in ether, than filtration. Removal of the solvent gave the desired compounds. The identification process depends on UV-VIS. spectra, IR and H-NMR spectra and the value of R<sub>f</sub> when compared with standard. Compound under the same condition four zones were identified, while two zones are unknown. Gas chromatography also used to identify all components of oil.

**UV-VIS. spectra:-**UV-VIS. spectra were recorded using Shimadzo UV-VIS spectrophotometer 715 over the range 200-800 nm, by using DCM as solvent, to analyses the compounds that has been isolated in this study from the preparative TLC.

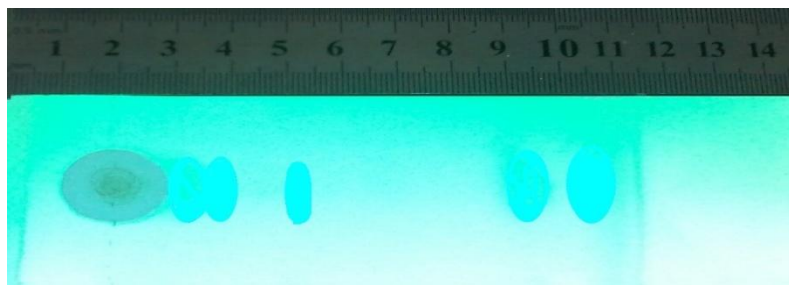
**IR spectra:-** IR spectra were recorded in University of Erbil College of science using Shimadzo spectrophotometer over the range 600-4000 cm<sup>-1</sup> to analyses the compounds that has been isolated.

**<sup>1</sup>H-NMR spectra:-** all <sup>1</sup>H-NMR spectrum were recorded in Irbaid University –Jordan, using CDCL<sub>3</sub> as solvent.

**Gas chromatography:-**Gas chromatography analysis was carried out in resources directorate Almaiah –Arbial by using a GC apparatus, using GC 717 from Shimadzo equipped with a flam ionization detector

**Results and discussion:-**

Fig(1) appear the TLC test of thyme oil, from this chromatogram, one could observe six zone ,four zones were characterized when compared with standard material, the results of these comparisons were explained in table(1).



**Fig 1:-** TLC chromatogram.

**Table 1:-** Retention factor (R<sub>f</sub>) of identification compound for volatile oil.

compound	Flow rate R <sub>f</sub>
p-Cymene	0.92
Thymol	0.87
Carvacrol	0.60
Unknown	0.31
Camphor	0.20

\*ethyl acetate: Toluene 5: 95 % as mobile phase

To identified each component of this chromatogram by spectroscopic methods. Each zone was crushed carefully isolated and dissolved in diethyl ether, which on filtration and removal of solvent gave the desired compounds.

IR spectrum of isolated compounds (1-4) were shown in figures(2-5) and table(2), from there table one could observed that compounds 1&2 exhibits a broad band appearing at 3382-3300 assigned to the stretching vibration of (OH) group. While compound (3) exhibits the band 1750 was related to C=O stretching.

Compound (4) exhibits the following bands, 3050 was due to aromatic C-H stretching band, 2965-2865 was stretching due to C-H methyl group, 1514 was due to C=C stretching of ring besides other characteristic bands.

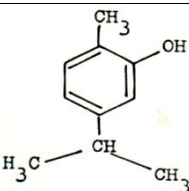
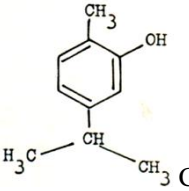
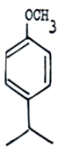
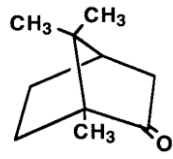
Figures (6-9) show the H-MNR spectra of compounds (1-4), while table (3) give the chemical shift of each proton in these compounds. Table(4) gives the maximum absorption bands ( $\lambda_{\max}$  in nm) of compounds (1-4). Figer (10) show the chromatogram of thyme oil which was measured by gas chromatogram (GC), the components of this oil were identified by comparing their relative retention times with those of authentic samples [9].The retention time of thymol is 3.91 minutes, Carvacrol is 46.08 minutes, camphor 22.25 minutes and P.cymene is 8.98 minutes.

**Table 2:-** some characteristic bands of compounds 1-4

compound	Name	Assignment $\text{cm}^{-1}$				
		OH	CH Str.	C=C Str.	OH bending	Isopropyl group
1	Thymol	3300 broad	2962-2870	1570,1480,1430	12,87-1345	1285
2	Carvacrol	3382	2960-8860	1585,1460,1458	1458-1241	1395
3	Camphor	-	2950	-	-	1390-1375
4	p-Cymene	CH aromatic str. 3050	2965-2865	1514		

C-O str. In phenol	CHaromatic bending	C
1246	800	
1251	802	
-	750	

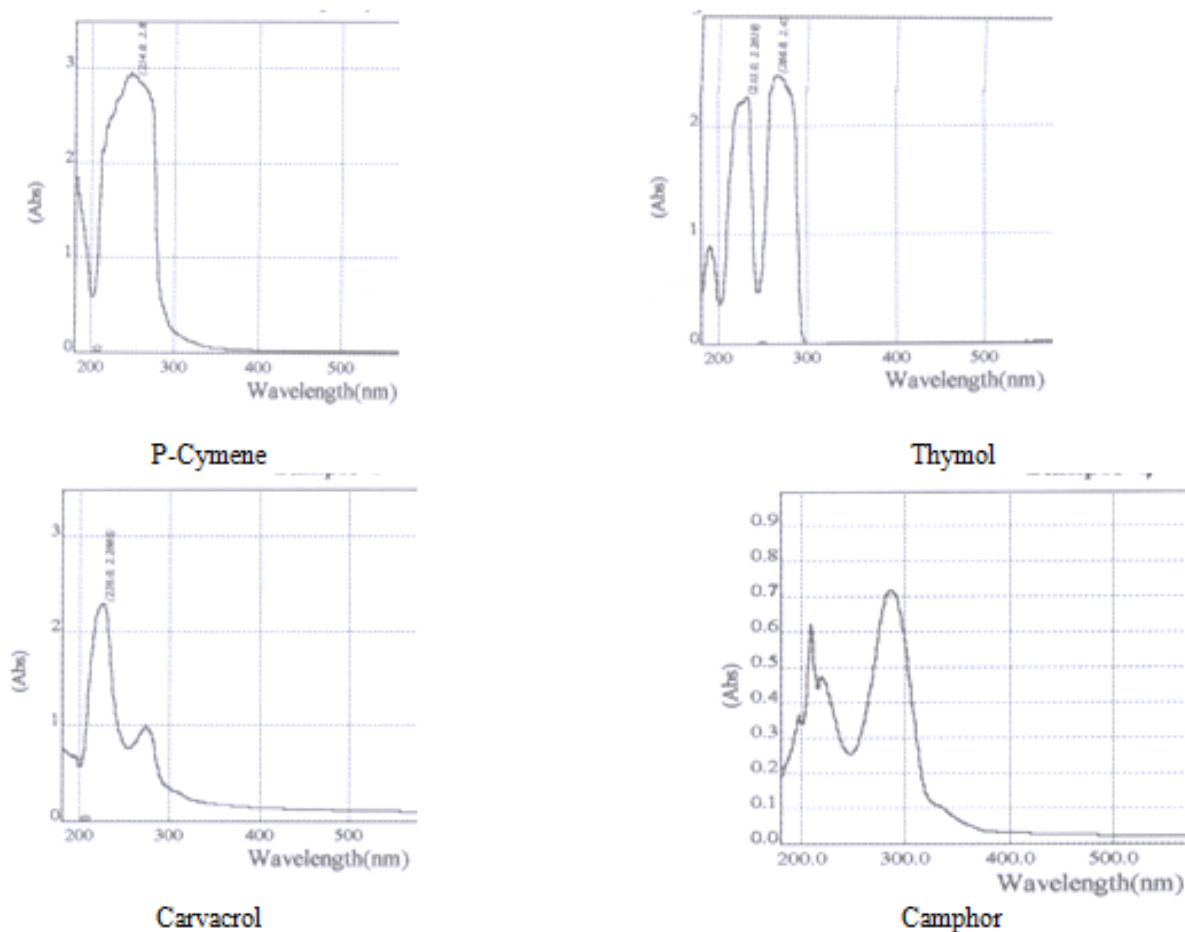
Table (3)  $^1\text{H-MNR}$  data of compounds

Compounds	Chemical shifts:PPM				
	A	B	C	D	Aromatic protons
 Thymol	1.2(d)	2.25(s)	3.17(m)	4.7(s)	d 6.80-710
 Carvacrol	1.3(d)	2.25(s)	2.9(m)	5.9(s)	7.0(m)
 p-Cymene	1.2(d)	2.3(s)	2.9(m)		7.2(m)
 Camphor	0.7(S)	0.8(S)	1.0(S)	Other (2.3)	Multiplet

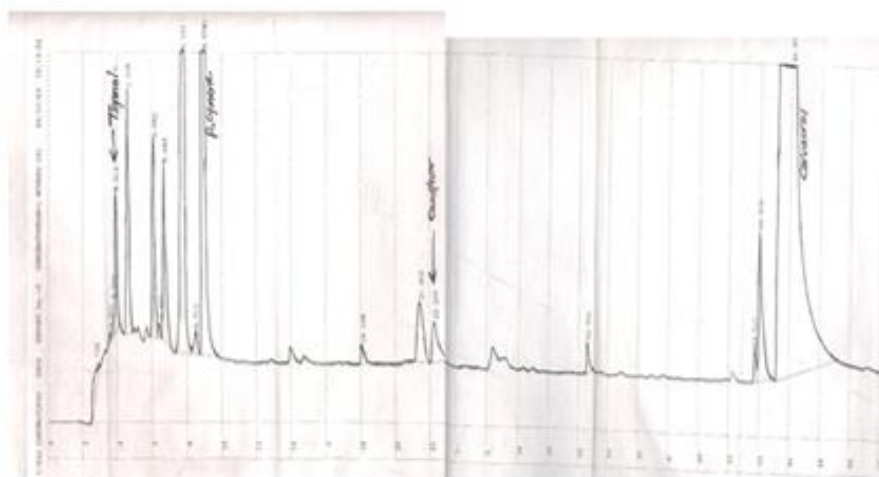
S: Single, d: doublet, m: multiplet

**Table 4:-** UV-VIS. Spectral data of compounds (1-4)

compound	Absorption maxima $\lambda_{max}$ (nm)
Thymol	256,290,374
Carvacrol	256,298,365
p-Cymene	257,265,274
Camphor	290



**Figure 2:-** The UV spectra of active organic compounds in volatile oil.



**Figure(3 )** Gas chromatogram of extracted volatile oil

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