



[Science Archives \(ISSN:2582-6697\)](http://www.sciencearchives.org)

Journal homepage: www.sciencearchives.org



<https://doi.org/10.47587/SA.2023.4308>

Research Article



Gas chromatography- mass spectrometry analysis of natural products in *Pimpinella olivieri* from North Iraq

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Received: Aug 18, 2023/ Revised: Sept 8 24, 2023/Accepted: Sep 10, 2023

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Abstract

Wild plants possess many bioactive ingredients derived from by-metabolic process, which have a wide broad uses as medication and pharmaceutical importance. Therefore, the aim of the present study on a species of genus *Pimpinella* that belong to the family Apiaceae (Umbelliferae) developing northern Iraq *Pimpinella olivieri*, is the qualitative analysis of the methanolic extract of the plant to find out the active compounds in it by gas chromatographic technique equipped with mass spectroscopy. The results of the extract showed the presence of phenols, alkaloids, steroids, flavonoids, glycosides, fatty acids, terpenoids and carbohydrates. The results of GC-Mass analysis revealed presence of 20 biological compounds in the methanolic extract of the plant, such as sesquiterpenoid, lipophilic compounds. Bisbollene, Cis-13-octadecenoic acid, methyl ester (highest percentage of 72.05 and 5.14) % at 15.1 and 24 minutes respectively of the detention time and the proportions of the rest of the terpenes in the plant extract were varied. The Benzene methanol was phenolic compound, 3,5-dimethyl, in ratio 12.96 at 13.3 minutes of detention time. While the two compounds (1,3,5,7-tetraethyl-1-methox cycl tetra siloxane and Cyclohexane, 1R-acetamido-2cis,4trans-bis(acetoxy)-3trans-azido) gave the lowest percentage of 0.13%.

Keywords: Methanolic Extract, *Pimpinella olivieri* Leaves, GC-Mass Analysis.

Introduction

Apiaceae (Umbelliferae) is one of the most important flowering plant families in Iraq, with 60 wild genera and 143 species (Al-Rawi 1987). Ghazanfar and McDaniel (2016) was revealed this family is the fifth-largest plant family in Iraq with approximately 67 genera and 155 species. The genus *Pimpinella* L., with about 170–180 species in the world, is one of the largest genera of the family Apiaceae, these species has a huge economic, medical and pharmaceutical importance, some of them act as anti (parasitic, fungal and microbial) (Akhtar, 2008).

Some species also act as an appetizer, co- digestriator and a diuretic. It is also used to treat some cases of depression and anti-epilepsy and muscular relaxant. Tetik (2013) Studies have revealed that extracted seeds oil act as antibacterial against

both Gram-negative (*Bacillus subtilis* and *Staphylococcus aureus*) and Gram-positive bacteria (*E.coli*, *Pseudomons aeruginosa* and *Klebsiella*) (Askari et al., 2013).

Wild plants are medicinal plants that have taken a wide broad spectrum in traditional medicine (Okoye, 2014), the bioactive components resulting from the specialized metabolism of the plant play a major role as antioxidants, antibacterial, antibacterial and antiviral mechanisms are proposed for the reduction of inflammation (Mary, 2016; Devi et al., 2018) the effective chemical groups of medical and pharmaceutical importance (phenols, flavonoids, saponins, steroids, coumarins, terpenes, alkaloids, glycosides, tannins and others (Thomas and Momin, 2020).

In general, phenols have an important role in the growth and reproduction of plants as well as their importance in anti-

environmental stresses such as low- temperatures, ultraviolet, nutrient deficiencies and low humidity, as they make the cell walls impermeable to water and gases and are responsible for giving the plant hardness, natural resistance to bacterial, fungal and insect infections (Stafford, 1991; Lattanzio; et al. 2009).

Flavonoids also have several therapeutic heart disease and some types of cancer tumors and pharmacological activities (antioxidants and anti-inflammatory) (Agati et al., 2012). Sofiana, (2020) showed the plants of Apiaceae family produce about 12,000 types of alkaloids that have vital effectiveness as an antibacterial and antioxidant. Glycosides are bioactive components act as anti-viruses and many pathogenic lesions (Brito-Arias, 2022).

In Iraq, the first study that detect the phenolic and alkaloid content of *Pimpinella olivieri* was by Ameen (2021). In the current study, the extract of bioactive components of *Pimpinella olivieri* from aqueous alcoholic extract by using (GC-Mass) technique.

Materials and methods

1- Collection of plant material

Field trips to Azmar Mount in Sulaymaniyah Governorate - northern Iraq, leaves of the *Pimpinella olivieri* were collected, washed, dried and then grinding to powder leaves by an homogenizer.

2- Preparation of aqueous alcoholic extract

The extract was prepared by taking 50 gm of dried plant leaf powder and mixed with 500 ml ethyl alcohol 70% in a glass flask of 1000 ml and closed by cotton and aluminum foil and placed in a shacking incubator 24°C/24 hours. Alcoholic mixture was filtered by a medical gauze to get separate of unwanted large particles, next step centrifuged filtered solution 3000 rcf / 10 minutes, the extract was filtered by filter papers grade one/1.0 mm Whatman for separate solution extract from precipitation, the extract solution ready for Gas-Chromatography-Mass Spectrometry (Hernández-Pérez et al., 1994).

The analysis was performed by using the GC-MS A7820 device modle/ Agelint company. The procedure of the analysis was

- Colum: Hp-5ms ultra Ineit (30m length x
- 250 nm diameter x 0.25 um in side diameter
- Injection volume: 1 cup
- Pressure: 11.933 psi
- GC inlet line temperature: 250°C
- Aux heaters temperature: 310°C
- Carrier Gas: He 99.99%
- Injection: Temperature: 250°C
- Injection type: Split less
- Scan range: m/z 50-500
- Oven Program:
- Ramp1: 50°C Hold 1 min., Ramp1: 50°C to 150°C, 5°C/ min
- Ramp2: 150°C to 280°C, 8°C /min
- Time amounted to about 37 min

Table 1. compounds identified from methanol extract of leaves of *Pimpinella olivieri* and Phytochemicals, Retention time and peak Area%

S.No.	Compound	Phytochemicals	Retention time	Area(%)
1.	2-methyl-3,5-dinitrobenzyl alcohol, tetra butyl dimethylsily ether	Alkaloids	59	0.16
2.	Tri (tert-butyl)dimethyl siloxy) arsane	Polymer	6.1	0.33
3.	1,3,5,7-tetraethyl-1-methox cycl tetra siloxane	Polymer	6.3	0.13
4.	Cyclo tetra iloxane, octamethyl	Polymer	9.3	0.39
5.	Phyenyln-butane-2,3- dienylether	Phenol	11.3	0.18
6.	Copaene	essential oil	12.5	0.43
7.	Benzene methanol, 3,5-dimethyl	Phenol	13.3	12.96
8.	Cis-(-)-2,4a,5,6,9a-Herxahydro-3,5,5,9-tetramethyl(1H) benzocycloheptene	Alkane	14.6	1.20
9.	Beta. Bisbollene	Sesquiterpene	15.1	72.05
10.	Diazo progesterone	Alkaloids	15.4	---
11.	Cyclophyllene Oxide	Phenol	16.6	0.45
12.	2-hydroxy-4-methoxy benezaldehyde, trimethyl silyl ether	Flavonoid	18.1	0.65
13.	Hexadecanoic acid, methylester	Fatty Acid Ester	21.6	1.30
14.	Phenol, 2- (2- aminoethyl)	Phenol	23.0	0.27
15.	Cyclohexane, 1R-acetamido-2cis,4trans-bis(acetoxy)-3trans-azido	Alkaloids	23.6	0.13
16.	Cis-13-octadecenoic acid, methyl ester	Sesquiterpenoid	24.0	5.14
17.	Methyl stearate	fatty acids	24.3	1.64
18.	Estriol, tris(trifluoroacetate)	Hormone	26.7	0.64
19.	Nonahexacontanoic acid	Alkanoic acid	28.6	0.78
20.	Hexadecanoic acid, 2-(octadecyloxy) ethylester	Esters	30.7	1.00

Results and discussion

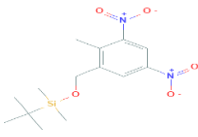
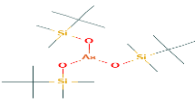

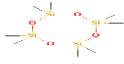

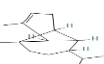
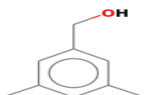
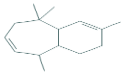

The results of aqueous alcoholic extract of *Pimpinella olivieri* leaves obtained from gas chromatography technique with mass spectroscopy, revealed 20 bioactive compounds were shown in Tables (1, 2). These compounds are (Beta. Bisbollene, Benzene methanol, 3,5-dimethyl, Cis-13-octadecenoic acid, methyl ester, Methyl stearate, Hexadecanoic acid, ethylester, Cis-(-)-2,4a,5,6,9a-Herxahydro-3,5,5,9-tetramethyl(1H) benzocycloheptene and Hexadecanoic acid, 2-(octadecycloxy) ethylester).

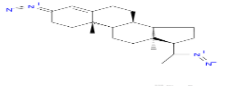
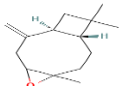
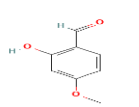


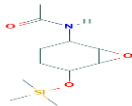
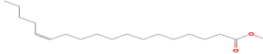

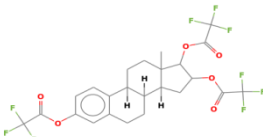

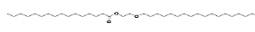
The results as shown in the Table below revealed maximum ratios (1.00, 1.20, 1.30, 1.64, 5.14, 72.05, 12.96) respectively, while the compounds (1,3,5,7-tetraethyl-1-methox cycl tetra

siloxane and Cyclohexane, 1R-acetamido-2cis,4trans-bis(acetoxy)-3trans-azido) minimum ratios about 0.13%.

As shown in Table (1) there are many volatile oils (Sesquiterpenoid) that appeared in maximum proportions 72.05%, 5.14% in both compounds (Bisbollene, Cis-13-octadecenoic acid, methyl ester) respectively at the time of detention 15.1 and 24.0 respectively. The rest of the secondary metabolits also had a huge percentage of the total compounds appeared in chemical analysis. Groups of phenols, alkalis flavonoids and fatty acids, which showed the high maximum proportions of (Benzene methanol, 3,5-dimethyl) 12.96% at the 13.3 minute of the detention time and 1.64% at the 24.3rd minute of Time of detention of fatty acid Methyl stearate, also showed the detection of the presence of many polymers, esters and hormones.

Table 2. Chemical formulas and compositions of the separated compounds

S.No.	Compound	Structure	Molecular Formula
1.	2-methyl-3,5-dinitrobenzyl alcohol, tetra butyl dimethylsily ether		C ₁₄ H ₂₂ N ₂ O ₅ Si
2.	Tri (tert-butyldimethyl siloxy) arsane		C ₁₈ H ₄₅ AsO ₃ Si ₃
3.	1,3,5,7-tetraethyl-1-methox cycl tetra siloxan		C ₁₂ H ₂₉ O ₅ Si ₄
4.	Cyclo tetra siloxan, octamethyl		C ₈ H ₂₄ O ₄ Si ₄
5.	Phyenyl-buta-2,3- dienylether		C ₁₀ H ₁₀ O
6.	Copaene		C ₁₅ H ₂₄
7.	Benzene methanol, 3,5-dimethyl		C ₉ H ₁₂ O
8.	Cis-(-)-2,4a,5,6,9a-Herxahydro-3,5,5,9-tetramethyl(1H) benzocycloheptene		C ₁₅ H ₂₄
9.	Beta. Bisbollene		C ₁₅ H ₂₄

10.	Diazoprogesterone		C ₂₁ H ₃₀ N ₄
11.	Cyclophyllene Oxide		C ₁₄ H ₂₂ O
12.	2-hydroxy-4-methoxy benzaldehyde, trimethyl silyl ether		C ₈ H ₈
13.	Hexadecanoic acid, methylester		C ₁₈ H ₃₆ O ₂
14.	Phenol, 2- (2- aminoethyl)		C ₈ H ₁₁ NO
15.	Cyclohexane, 1R-acetamido-2cis,4trans-bis(acetoxy)-3trans-azido		C ₁₁ H ₂₁ NO ₃ Si
16.	Cis-13-octadecenoic acid, methyl ester		C ₁₉ H ₃₆ O ₂
17.	Methyl stearate		C ₁₉ H ₃₈ O ₂
18.	Estriol, tris(trifluoroacetate)		C ₂₄ H ₂₁ F ₉ O ₆
19.	Nonahexacontanoic acid		C ₆₉ H ₁₃₈ O ₂
20.	Hexadecanoic acid, 2-(octadecyloxy) ethylester		C ₃₆ H ₇₂ O ₃

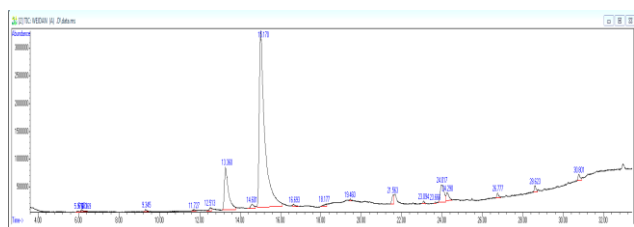


Fig. 1 GC–Mass total ion chromatograms of compounds identified from methanol extract of leaves of *Pimpinella olivieri*

The results demonstrate in Fig. 2, 3, 4 and 5 indicate the G-mass spectra of the essential compounds, which were present in maximum proportions in the aqueous alcoholic extract of plant leaves; Fig. 2 indicates the mass spectrum of the compounds: Benzene methanol and 3,5-dimethyl where the molecular weight of the compound is 136 which represents the base peak in the spectrum, while Fig. 3 shows the mass spectrum of the compound: Beta. Bisbollene where the molecular weight of the compound is 204 while the base peak is 69.

Fig. 4 showed the mass spectrum of Hexadecanoic acid, methyl ester with molecular weight of 270 and base peak 74.

Fig. 5 refers to the mass spectrum of the compound: Cis-(-)-2,4a,5,6,9a-Herxahydro-3,5,5,9-tetramethyl (1H) benzocyclo heptene with molecular weight of compound 204 and the base peak of 93.

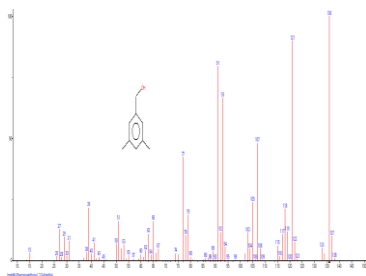


Fig. 2 GC-Mass Benzenemethanol, 3,5-dimethyl-

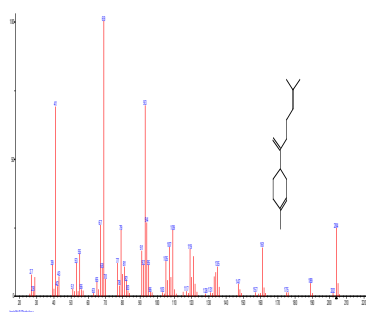


Fig. 3 GC-Mass β-Bisabole

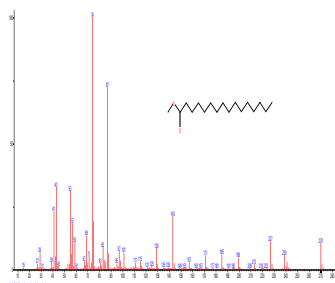


Fig. 4 GC-Mass Benzenemethanol, 3,5-dimethyl-

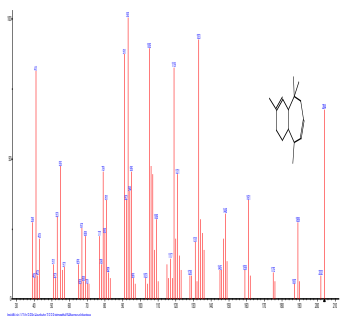


Fig. 5 GC-Mass cis-(-)-2,4a,5,6,9a-Hexahydro-3,5, 5,9-tetramethyl(1H) benzocycloheptene

The results of the present study were demonstrated the medicinal and pharmaceutical importance of the plant extract. This importance represented through bioactive secondary molecules of plants metabolisms, especially the essential and volatile oils that are characterized in many species of this family, which are very effective against many microorganisms such as bacteria and fungi (Hajji, et al., 2010; Sahebkar and Iranshahi, 2010). The results also showed vegetable oils, especially sesquiterpenoid, that have a maximum value in the extracted bioactive compounds, Paksoy et al. (2016) confirmed that oils extracted from the Apiaceae family, which acts a major roles as biological effectiveness, like an antimicrobial agents. Mehrabanjoubani et al., (2021) confirmed that *Pimpinella anisum* L. extracted oils act as antioxidants.

Alfekaiki, (2018) revealed that fatty acids extracted from *Pimpinella anisum* L. have a positive effective as antioxidants. Esterial fatty acids in plants of the Apiaceae family have a major role in antibacterial growth factor (Lu et al., 2020).

Adesalu et al., (2022) showed that the compound Nonahexacontanoic acid act as an antioxidant and antiviral factors, in addition to its importance in food preservation and with its importance in many petrochemical industries such as bio pesticides (pheromones), paraffin wax production, and others.

Compounds Hexadecanoic acid, 2-(octadecycloxy) ethylester is an ester compound that have high effectiveness as antifungal factors (Hamid et al., 2022).

Conclusion

In conclusion, the qualitative analysis of the methanolic extract from *Pimpinella olivieri*, a plant species native to northern Iraq and belonging to the Apiaceae (Umbelliferae) family, using gas chromatographic technique equipped with mass spectroscopy, has revealed a diverse array of bioactive compounds. The extract exhibited the presence of various important chemical classes, including phenols, alkaloids, steroids, flavonoids, glycosides, fatty acids, terpenoids, and carbohydrates. Moreover, the GC-Mass analysis identified a total of 20 biological compounds in the extract, with sesquiterpenoids and lipophilic compounds being prominent constituents. Notably, Bisbollene and Cis-13-octadecenoic acid methyl ester were the major compounds, constituting the highest percentages in the extract.

Furthermore, the phenolic compound 3,5-dimethylbenzene methanol was detected in significant proportions. Conversely, two compounds, namely 1,3,5,7-tetraethyl-1-methoxycyclotetrasiloxane and Cyclohexane 1R-acetamido-2cis,4trans-bis(acetoxy)-3trans-azido, were found in minimal amounts. These findings underscore the pharmacological and medicinal potential of *Pimpinella olivieri* and highlight the diverse range of bioactive compounds that may contribute to its therapeutic properties. Further research is warranted to

elucidate the specific pharmacological activities and potential applications of these compounds in medicine and pharmaceuticals.

Conflict of Interest

The author hereby declares no conflict of interest.

Consent for publication

The author declares that the work has consent for publication.

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How to cite this article

Aziz, W. S., Al Tikriti, A. H. A. and Muhammed, Z. B. (2023). Gas chromatography- Mass spectrometry analysis of natural products in *Pimpinella olivieri* from North Iraq. *Science Archives*, Vol. 4(3), 226-231. <https://doi.org/10.47587/SA.2023.4308>

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