

# DETERMINATION OF BIOACTIVE COMPOUNDS IN LEAVES OF UROSPERMUM PICROIDES (L.) DESF

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#### Abstract

A total of thirty- three bioactive chemical compounds in the methanolic extract of Urospermum picroides (L.) Desf. leaves were identified, the identification was done using GC-MS technique, and based on Retention time, peak area, molecular Weight and exact mass. The compounds encompass. 1.6-Anhydro-2.4-dideoxy-β-D-arabo-hexopyranose, 2-(Benzylsulfanylfluoro-methylene) – malonic acid dir, Pyrido [2.3-d] pyrimidine -2.4.7(1H.3H.8H)-trione. 8-b, 3.6-Octadecadiynoic acid. methyl ester, 3-Isopropyl-2-phenyl-pent-4-en-2-ol, 2-Dodecenal. (E)-, 2-Dodecenoic acid, 5.7-Dodecadiyn-1.12-diol, d-Lyxo-d-mannononoic-1.4-lactone, Cyclopropaneacetic acid. 2-hexyl-, 10.12-Octade cadiynoic acid, 2H-Indeno [1.2-b] furan-2-one. 3.3.a.4.5.6.7.8.8b-oct, 3-Hydroxy-7.8-dihydro-β-ionol, 3-(N.N-Dimethyllaurylammonio) propanesulfonate, 3(N.N-Dimethylmyristylammonio) propanesulfonate, (7R.8R)-Ethyl8-hydroxy-trans-bicyclo [4.3.0]-3-none, Tetra decanoic acid, 9.10-Dimethyltricyclo [4.2.1.1(2.5)] decane - 9.10-diol, 2.6.8-Trimethylbicyc lo [4.2.0] oct-2-ene-1.8-diol, Butyl 9-hexadecenoate, 3.6.9.12-Tetraoxatetradecan-1-ol.14 [4-(1.1.3.3-tet, Cinnamic acid. 4-hydroxy-3-methoxy-. {5-hydroxy-2-, trans-13-Octadecenoic acid, Octadecanoic acid, 7-Methyl-Z-tetradecen-1-ol acetate, [1.1'-Bicyclopro pyl] -2-octanoic acid. 2'-hexyl-.meth, 14.15.16-Trinor-8 xi.-labdan-66-ol. 8. 13-epoxy-, 2-(1-Butyl-2-nitroallyl) cyclohexanone, Benzeneethanamine, 2.5-difluoro-6.3.4-trihydroxy-,6-Octa decenoic acid. (Z)-, Hexadecanoicacid. 2-hyd roxy-1-(hydroxymethyl)eth, Diisooctyl phthalate, β-Tocopherol. Which fatty acids, their derivatives and alcohols are the compounds most frequently isolated in the leaves of the species, in addition to alkaloids, sesquiterpene lactone and other known compounds these compounds may be important as antioxidant, antimicrobial, antiinflammentory.

Key words : Urospermum, Hyphochaeridinae, Asteraceae, Sesquiterpene lactone.

### Introduction

The genus Urospermum picroides (L.) Desf. Belongs to the family Asteraceae; tribe : Lactuceae ; sub tribe : Hyphochaeridinae (Enk. et al., 2012). It includes two wild species in the Mediterranean region (Thomb, 1977; Thompson, 2007), only one species for the genus was found in Iraq (Ridda & Daood, 1982). The genus has many chemical classes of secondary compounds include phenoics (Giner et al., 1992), (Enk. et al., 2012), indole-alkaloids (Ji et al., 2008) Glycosides (Amer et al., 1984), (Balboul et al., 1997), Saponin, tannin and flavonoids (Enk. et al., 2012), (El-Amier et al., 2016) and sesquiterpene lactone, (Balboul et al., 1997), (Killan et al., 2009). From the literature, no chemical studies on the genus Urospermum in Iraq. Thus, we have studied the occurrence of different chemical compounds in the leaves of the genus to obtain a better chemotaxanomic

description for it.

## Materials and Methods

#### Collection and preparation of plant materials

Urospermum picroides (L.) Desf. Leaves were collected through field trips from different localities in the central and south of Iraq. After cleaning and removal foreign materials the leaves were washed with tap water then with distilled water and dried under shed for ten days at room temperature. Dried leaves stored in airtight container at room temperature until further use. Eighteen gram of each methanolic extract of plants powdered were soaked in 40 ml methanol for ten hours in a rotator shaker (Huda *et al.*, 2015a). Whatman No.1 filter paper was used to separate the extract of plant and again filtered through sodium sulphate in order to remove the moisture.

Gas chromatography – mass spectrum (GC/MS)

#### analysis.

The GC-MS analysis of the plants extract was made in a (QP 2010 Plus SHIMADZU) instrument under computer control at 70 eV. About 1µL of them ethanol extract was injected into the GC-MS using a micro syringe and the scanning was done for 45minutes. As the compounds were separated, they eluted from the column and entered a detector which was capable of creating an electronic signal whenever a compound was detected. The greater the concentration in the sample, bigger was the signal. obtained which was then processed by a computer. The time from when the injection was made (Initial time) to when elution occurred is referred to as the; Retention Time (RT) (Mohammed and Imad, 2013; Muhanned *et al.*, 2015; Imad *et al.*, 2014a). While the instrument was run, the computer generated a graph from the signal called a Chromatogram. Each peak in the chromatogram represents the signal created when a compound eluted from the Gas chromatography column into the detector. The X-axis showed the RT and the Yaxis measured the intensity of the signal to quantify the component in the sample injected. As individual compounds eluted from the Gas chromatographic column, they entered the electron ionization (mass spectroscopy) detector, where they were bombarded with a stream of electrons causing them to break apart into fragments. The fragments obtained were actually/ charged ions with a certain mass. The M/Z (Mass/Charge) ratio obtained was calibrated from the graph obtained, which was called as the Mass spectrum graph which is the fingerprint of a

Table 1: Major Phytochemical compound identified in methanolic extract of Urospermum picroides

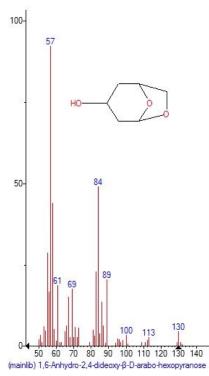
S. No.	Phytochemical compound	RT (min)	Exact mass	Nature of compound
1	1.6-Anhydro-2.4-dideoxy-β-D-arabo-hexopyranose	3.630	130.062994	Glycoside
2	2-(Benzylsulfanyl-fluoro-methylene) – malonic acid dir	3.825	284.051859	Alcohol
3	Pyrido[2.3-d]pyrimidine-2.4.7(1H.3H.8H)-trione. 8-b	3.836	313.106256	Alkaloid
4	3.6-Octadecadiynoic acid. methyl ester	4.174	290.22458	Fatty acid ester
5	3-Isopropyl-2-phenyl-pent-4-en-2-ol	4.638	204.151415	Alcohol
6	2-Dodecenal. (E)-	5.633	182.167066	Fatty acid ester
7	2-Dodecenoic acid	6.697	198.16198	Fatty acid
8	5.7-Dodecadiyn -1.12-diol	7.630	194.13068	Fatty acid
9	d-Lyxo-d-manno-nonoic-1.4- lactone	7.630	268.079432	Carbonyl compound
10	Cyclopropaneacetic acid. 2-hexyl-	7.659	184.14633	Propanoic acid
11	10.12-Octadecadiynoic acid	8.317	276.208931	Fatty acid
12	2H-Indeno[1.2-b]furan-2-one. 3.3a.4.5.6.7.8.8b-oct	8.488	206.13068	Hydrocarbon
13	3-Hydroxy-7.8-dihydro-β-ionol	10.503	208.14633	Alcohol
14	3-(N.N-Dimethyllaurylammonio) propanesulfonate	9.719	335.249414	Alkaloid
15	3(N.N-Dimethylmyristylammonio) propanesulfonate	12.025	363.280716	Alkaloid
16	(7R.8R)-Ethyl 8-hydroxy-trans-bicyclo[4.3.0]-3-none	12.242	210.125594	Sesquiterpene
17	Tetradecanoic acid	12.648	228.20893	Fatty acid
18	9.10-Dimethyltricyclo [4.2.1.1(2.5)]decane –9.10-diol	12.837	196.14633	Alcohol
19	2.6.8-Trimethylbicyclo [4.2.0]oct-2-ene-1.8-diol	13.060	182.13068	Alcohol
20	Butyl 9-hexadecenoate	14.096	310.28718	Butanoic acid
21	3.6.9.12-Tetraoxatetradecan-1-ol.14[4-(1.1.3.3-tet	14.096	426.29814	Alcohol
22	Cinnamic acid. 4-hydroxy-3-methoxy {5-hydroxy-2-	14.079	652.23672	Aromatic compound
23	trans -13-Octadecenoic acid	15.830	282.25588	Fatty acid
24	Octadecanoic acid	16.619	284.27153	Stearic acid
25	7-Methyl-Z-tetradecen-1-ol acetate	16.808	268.24023	Hydrocarbon
26	[1.1'-Bicyclopropyl]-2-octanoic acid. 2'-hexylmeth	17.034	322.28718	Hydrocarbon
27	14.15.16-Trinor-8 xilabdan-6β-ol. 8. 13-epoxy-	17.775	266.22458	Alcohol
28	2-(1-Butyl-2-nitroallyl) cyclohexanone	17.588	239.1521435	Alcohol
29	Benzeneethanamine. 2.5-difluoro-β.3.4-trihydroxy-	18.319	219.0707	Alcohol
30	6-Octadecenoic acid. (Z)-	19.246	282.25588	Fatty acid
31	Hexadecanoicacid. 2-hydroxy-1-(hydroxymethyl)eth	19.446	330.27701	Fatty acid
32	Diisooctyl phthalate	19.749	390.27701	Sesquiterpene
33	β-Tocopherol	23.938	416.36543	Sterol

molecule. Before analyzing the extract using Gas Chromatography and Mass Spectroscopy, the temperature of the oven the flow rate of the gas used and the electron gun were programmed initially (Imad et al., 2014b). The temperature of the oven was maintained at. Helium gas was used as a carrier as well as 10°C an eluent. The flow rate of helium was set to 1ml per minute. The electron gun of mass detector liberated electrons having energy of about 70eV. The column employed here for the separation of components was Elite 1 (100% dimethyl poly siloxane). The identity of the components in the extracts was assigned by the comparison of their retention indices and mass spectra fragmentation patterns with those stored on the computer library and also with published literatures. Compounds were identified by comparing their spectra to those of the Wiley and NIST/ EPA/NIH mass spectral libraries (Imad et al., 2015c).

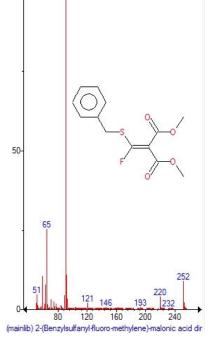
## **Results & Discussion**

The phytochemical analysis of *Urospermum picroides* (L.) Desf. Leaves by using GC-MS mechanism indicated that the plant is rich different secondary compounds (El-Amier *et al.*, 2016). Compounds 1-33 (Table 1) were identified as fatty acids, include, 3.6-Octadecadiynoic acid. methyl ester, 2-Dodecenal. (E)-, 2-Dodecenoic acid, 5.7-Dodecadiyn-1.12-diol, 10.12-

Octade cadiynoic acid, Tetra decanoic acid, trans -13-Octadecenoic acid, Octadecanoic acid, 6-Octa decenoic acid. (Z)-, Hexadecanoicacid. 2-hyd roxy-1-(hydroxymethyl) eth. Alcohols include 2-(Benzylsulfanylfluoro-methylene) - malonic acid dir, 3-Isopropyl-2phenyl-pent-4-en-2-ol, 3-Hydroxy-7.8-dihydro-β-ionol, 9.10-Dimethyltricyclo [4.2. 1.1(2.5)] decane – 9.10-diol, 2.6.8-Trimethylbicyc lo [4.2.0] oct-2-ene-1.8-diol, 3.6.9.12-Tetraoxatetradecan-1-ol.14 [4-(1.1.3.3-tet, 14.15.16-Trinor-8 xi.-labdan-6β-ol. 8. 13-epoxy-, 2-(1-Butyl-2-nitroallyl) cyclohexanone, Benzeneethanamine, 2.5-difluoro- $\beta$ .3.4-trihydroxy- these compounds are the most common followed by alkaloids which include Pyrido [2.3-d]pyrimidine- 2.4.7 (1H.3H.8H) -trione. 8-b, 3(N.N-Dimethylmyristylammonio) propanesulfonate, Hydrocarbon include. 2H-Indeno[1.2-b] furan-2-one. 3.3.a.4.5.6.7.8.8b-oct,7-Methyl-Z-tetradecen-1-ol acetate, [1.1'-Bicyclopro pyl] -2-octanoic acid. 2'-hexyl-.meth,Organic acids include Cyclopropaneacetic acid. 2hexyl-, Butyl 9-hexadecenoate, sesquterpene lactones include.(7R.8R)-Ethyl8-hydroxy-trans-bicyclo [4.3.0]-3none, Diisooctyl phthalate, with other compounds. 1.6-Anhydro-2.4-dideoxy-β-D-arabo-hexopyranose,as glycoside, Cinnamic acid. 4-hydroxy-3-methoxy-.{5hydroxy-2- as aromatic compounds and β-Tocopherol as



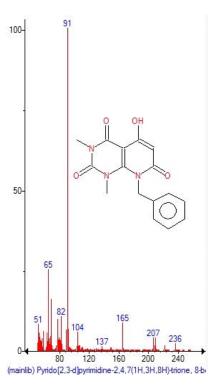
Phytochemical compound:1.6-Anhydro-2.4- dideoxy - β-D-arabohexopyranose RT (min): 3.630 Molecular Weight: 130.062994



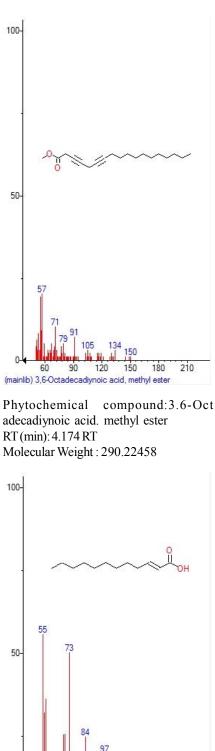
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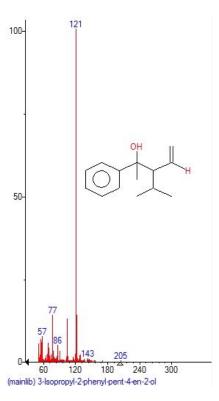
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Phytochemical compound:2-(Benzylsulfanyl -fluoro-methylene) – malonic acid dir RT (min): 3.825 Molecular Weight: 284.051859

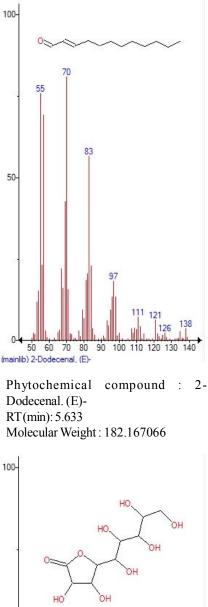


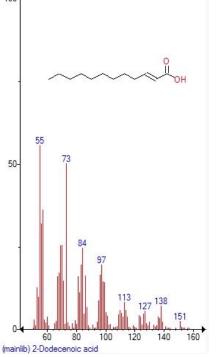
Phytochemical compound:Pyrido[2.3-D-]pyrimidine-2.4.7(1H.3H.8H)-trione. 8-b RT(min): 3.836 Molecular Weight: 313.106256



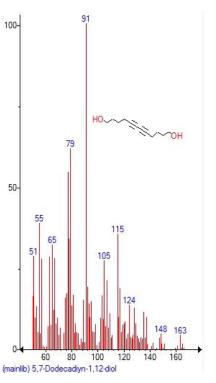


Phytochemical compound:3-Isopropy l-2-phenyl-pent-4-en-2-ol RT(min): 4.638 Molecular Weight: 204.151415

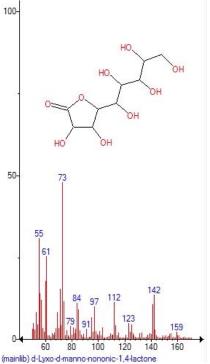




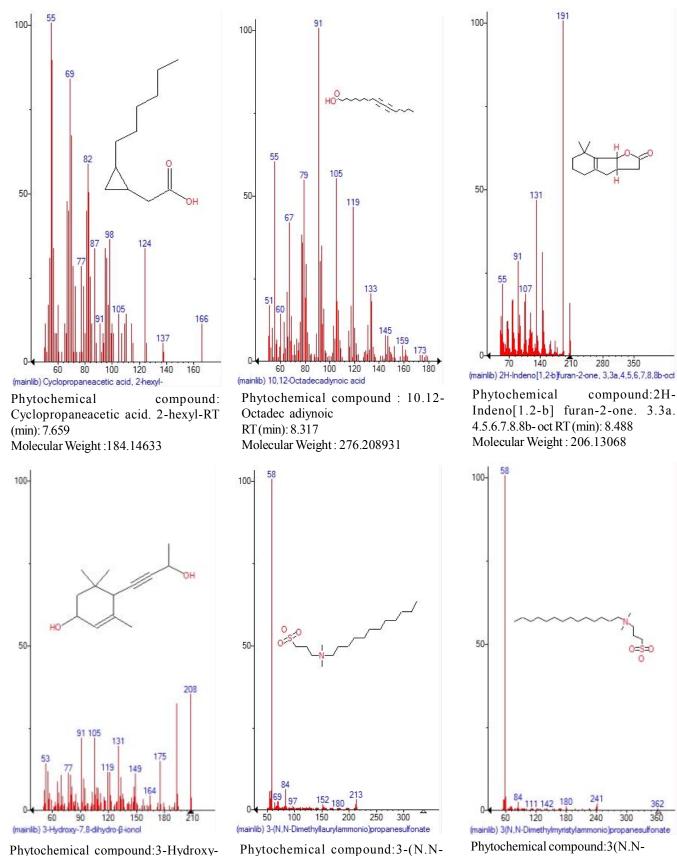
Phytochemical compound:2-Dodecenoic acid RT(min): 6.697 Molecular Weight: 290.22458



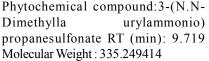
Phytochemical compound:5.7-Dodecadiyn -1.12-diol RT(min): 7.630 Molecular Weight: 194.13068



Phytochemical compound:d-Lyxo-dmanno-nonoic-1.4- lactone RT(min): 7.630 Molecular Weight :268.079432



Phytochemical compound:3-Hydroxy-7.8- dihydro -β-ionol RT (min): 10.503 RT Molecular Weight: 208.14633



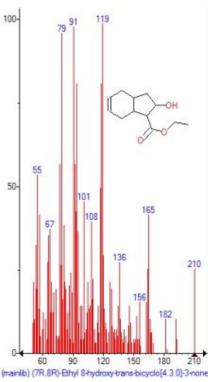
Phytochemical compound:3(N.N-Dimethylmyristylammonio) propanesulfonate RT (min): 12.025 Molecular Weight: 363.280716

362

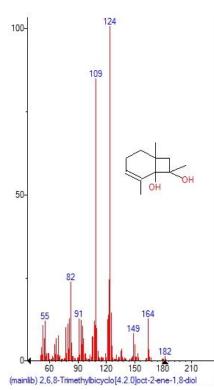
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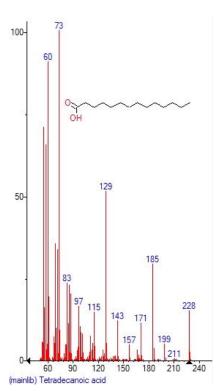
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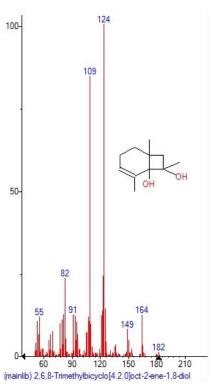
Phytochemical compound:(7R.8R)-Ethyl 8-hydroxy-trans-bicyclo[4.3.0]-3- None RT(min): 12.242 Molecular Weight : 210.125594



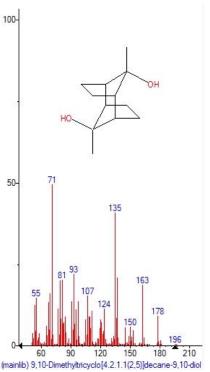
Phytochemical compound:2.6.8-Trimethy lbicyclo [4.2.0]oct-2-ene-1.8-diol RT(min): 13.060 Molecular Weight : 182.13068



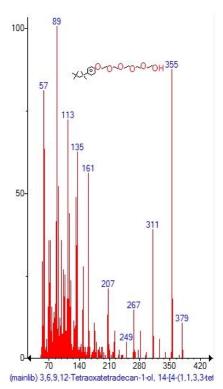
Phytochemical compound: Tetradecanoic acid RT (min): 12.648 Molecular Weight : 228.20893



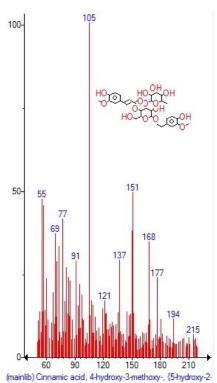
Phytochemical compound:Butyl 9-hexa decenoate RT (min): 14.096 Molecular Weight :310.28718



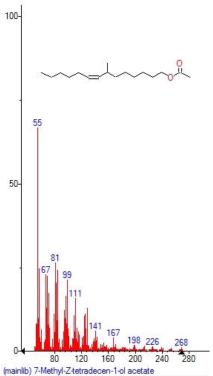
Phytochemical compound:9.10-Dimethyltricyclo [4.2.1.1(2.5)]decane – 9.10-diol RT(min): 12.837 Molecular Weight : 196.14633



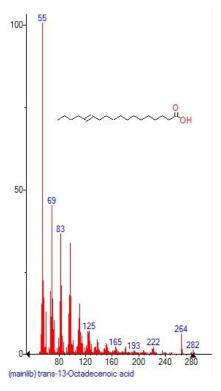
Phytochemical compound : 3.6.9.12-Tetraoxa tetradecan-1-ol.14[4-(1.1.3.3-tet RT(min): 14.096 Molecular Weight :426.29814



Phytochemical compound:Cinnamic acid.4-hydroxy-3-methoxy-. {5-hydroxy-2- RT (min): 14.079 Molecular Weight : 652.23672



Phytochemical compound:7-Methyl-Ztetr adecen-1-ol acetate RT (min): 16.808 Molecular Weight : 268.24023



Phytochemical compound:trans -13-Octadecenoic acid RT(min): 15.830 Molecular Weight : 282.25588

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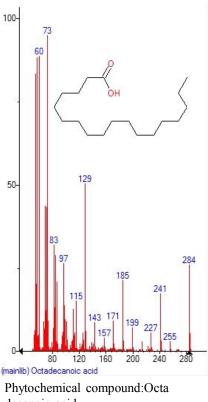
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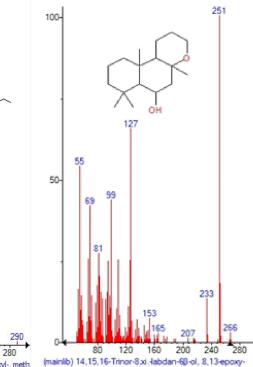
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decanoic acid RT (min): 16.619 Molecular Weight : 284.27153

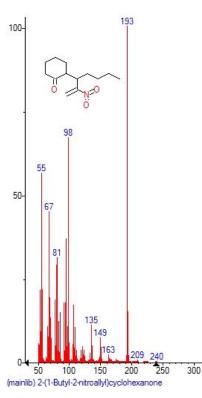


(mainlib) [1,1'-Bicyclopropy]-2-octanoic acid. 2'-hexyl-, meth Phytochemical compound: [1.1'-Bicyclopropyl]-2- octanoic acid. 2'hexyl-, meth RT (min): 17.034 Molecular Weight: 322.28718

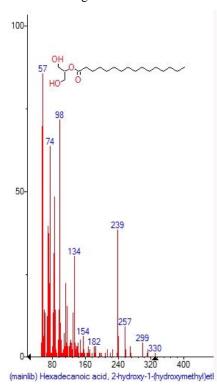
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Phytochemical compound : 14.15.16-Trinor-8 xi. -labdan-6β-ol. 8. 13-epoxy-RT (min): 17.775 Molecular Weight : 266.22458

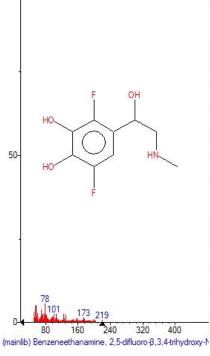
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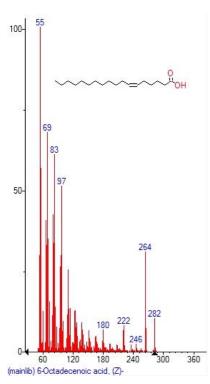
Phytochemical compound : 2-(1-Butyl-2nitroallyl) cyclohexanone RT (min): 17.588 Molecular Weight : 239.1521435



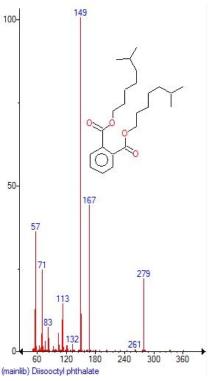
Phytochemical compound:Hexadecanoic acid. 2-hydroxy-1-(hydroxymethyl)eth RT(min): 19.446 Molecular Weight : 330.27701



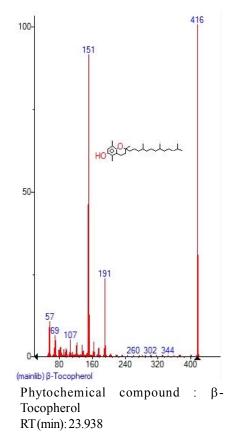
Phytochemicalcompound:Benzeneethan amine. 2.5-difluoro-β.3.4-trihydroxy- RT (min): 18.319Molecular Weight : 219.0707



Phytochemical compound:6-Octa decenoic acid. (Z)-RT(min): 19.246 Molecular Weight : 282.25588



Phytochemical compound:Diisooctyl phthalate RT (min): 19.749 Molecular Weight : 390.27701



Molecular Weight: 416.36543

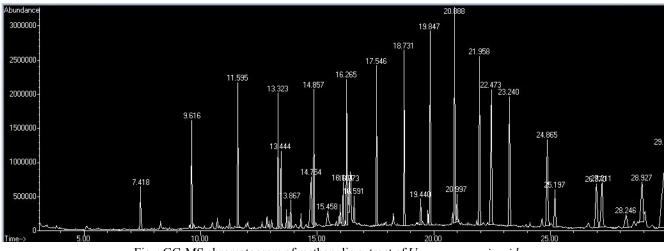


Fig. : GC-MS chromatogram of methanolic extract of Urospermum picroides.

sterol. some of these compounds have biological action as antimicrobial (El-Nabawy *et al.*, 2015) antiinflammatory (Strzeleeka *et al.*, 2005), (El-Amier *et al.*, 2016), antioxidant (El-Amier *et al.*, 2016), also some of these compound consider as chemotaxonomic marker in tribe Lactuceae as flavonoids & sesquiterpene (Hegnauer, 1977; Enk *et al.*, 2012).

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