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GC-MS analysis of liposoluble components from SPIRAEA HYPERICIFOLIA L.

Abstract. *Spiraea hypericifolia L. is a medicinal plant of Kazakhstan of which phytochemical analysis has not been explored. The present study found out that chemical constituents of dichloromethane extract from aerial part of the medicinal plant Spiraea hypericifolia L. have been identified using Gas Chromatography/Mass Spectrometry (GC/MS) analysis. GC-MS analysis of dichloromethane extracts of Spiraea hypericifolia L. revealed the existence of fifty-seven compounds, in which the main components are 1-tetracosene (18.41%), 9-tricosene, (Z)- (10.55%), n-hexadecanoic acid (5.72%), lanostan-3-one, 11.beta.,18-epoxy- (4.10%), stigmasta-5,22-dien-3-ol, acetate, (3.beta.)- (3.57%), tetracosanoic acid, methyl ester (3.28%). From the results, it could be concluded that Spiraea hypericifolia L. contains bioactive compounds with diversive biological activities. Therefore, it is recommended as a plant of phytopharmaceutical importance.*

Keywords: *S. hypericifolia L., dichloromethane extract, liposoluble components, GC-MS.*

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Introduction. In recent years, worldwide interest in medicines based on natural raw materials has remained high. Medicinal plants contain biologically active substances (BAS), which have a wide range of therapeutic effects, are low-toxic and are characterized by a stable pharmacological effect.

New sources of herbal remedies may be representatives of the genus *Spiraea - Spiraea* L. 22 species of the genus *Spiraea* grow on the territory of the CIS (Commonwealth of Independent States), and 13 species are found in Eastern Siberia [1]. There are 10 species distributed in Kazakhstan, and 3 species are found in Central Kazakhstan [2-4]. The genus *Spiraea* L. (meadowsweet, *spiraea*) belongs to the family *Rosaceae Juss.* the subfamily Spiroideae Agardh., which consists of 80-100 species [5].

The chemical composition of the genus *Spiraea* began to be studied in the XIX century. In 1838, the Italian chemist R. Piria isolated salicylic aldehyde from the flower buds of *spirea* [6,7].

Spiraea hypericifolia L. is a species of dicotyledonous flowering plant in the genus *Spiraea* of the rose family (*Rosaceae*). Shrub with small obovate leaves with umbels of small white five-membered flowers [8-11].

In the years 1974-1976 T.K. Chumbalov with co-authors in *S. hypericifolia* studied the composition of flavonoids derived from flavan. From this species, (+)- catechin, (-)- epicatechin and catechin glycosides were isolated and identified: 7-0-α-rhamnopyranoside-(+)-catechin, 7-0-β-xylopyranoside-(+)-catechin, 7-0-a-β-arabinoside-(+)-catechin. Apigenin, luteolin and their 5-0-glycosides are isolated from *S. hypericifolia* flavone derivatives: apigenin-5-0-β -glucopyranoside, luteolin-5-O- β-D-glucopyranoside [12-15].

In Tibetan medicine, the roots, bark, and leaves of *S. hypericifolia* is used for gastrointestinal diseases, rheumatism, helminthiasis, gynecological diseases, in traditional Kazakh medicine-for the treatment of dermatoses [16-17].

We have previously reported the chemical investigation results on total bioactive components from aerial part of *S. hypericifolia* L. And same time, twenty amino acids and eight fatty acids were analyzed from this plant [18].

In our continuously study of the plant, fivety seven liposoluble constituents in dichloromethane extract

from medicinal plant, *S. hypericifolia* L. have been identified by GC-MS methods which grown in Almaty region of Kazakhstan for the first time.

Materials and methods. *Plant material.* The aerial part of the plant material *S. hypericifolia* L. was collected in the Almaty region of Kazakhstan in autumn (October) 2018. The aerial part of *S. hypericifolia* L. dried in air was cut into small pieces and stored at room temperature.

Extraction and isolation. Naturally dried aerial parts of *S. hypericifolia* L. (100 g) were ground, then extracted with 90% ethyl alcohol (1:8) three times (seven days each time) at room temperature. After evaporation of the solvent at low pressure, the residue was dissolved in water, subsequently the resulting solution was sequentially separated with hexane, dichloromethane, ethyl acetate and n-butanol to obtain the corresponding extracts. The resulting hexane extract was analyzed by GC-MS.

Experimental part. The liposoluble components in the hexane extract of the medicinal plant were analyzed using the GC-MS method. The work was carried out on a gas chromatograph with mass selective detector Agilent 7890A -5975C. Used capillary column HP-5MS length 30 m, internal diameter 0.25 mm, film thickness of stationary phase 0.25 μm. Chromatography conditions: carrier gas-helium; flow rate 1 ml / min; column temperature: initial temperature of 50°C (10 min), temperature rise from 10°C / min from 50°C to 300°C, final temperature of 300°C (40 min), scanning range of 30-1000 AU, electronic shock mode at 70eV. The temperature of the ion source is 230°C. 1 μl of the sample was injected Into the chromatograph evaporator. Samples were introduced by splitting with a 5: 1 split ratio.

Identification of the compounds: GC-MS is a valuable aid for identifying unknown peak as well as for confirming the identification of identified phytoconstituents. In some cases when no identical spectra were found, the structural type of the corresponding component was suggested only on the basis of its mass spectral fragmentation and retention data. Identification of components was based on directs comparison of the retention times and mass spectral data with those for standard compounds and computer matching with the library (Wiley library, NIST data bank, database NIST 98) as well as by comparison of the retention time [19].

Results and discussion. In the GC-MS analysis, 57 chemical components were identified in the dichloromethane extract of the aerial part of *S. hypericifolia* L. The compounds present in the dichloromethane extract of *S. hypericifolia* L. revealed by GC-MS analysis are shown in Fig.1. The identification of phytochemical compounds is based on the peak area.

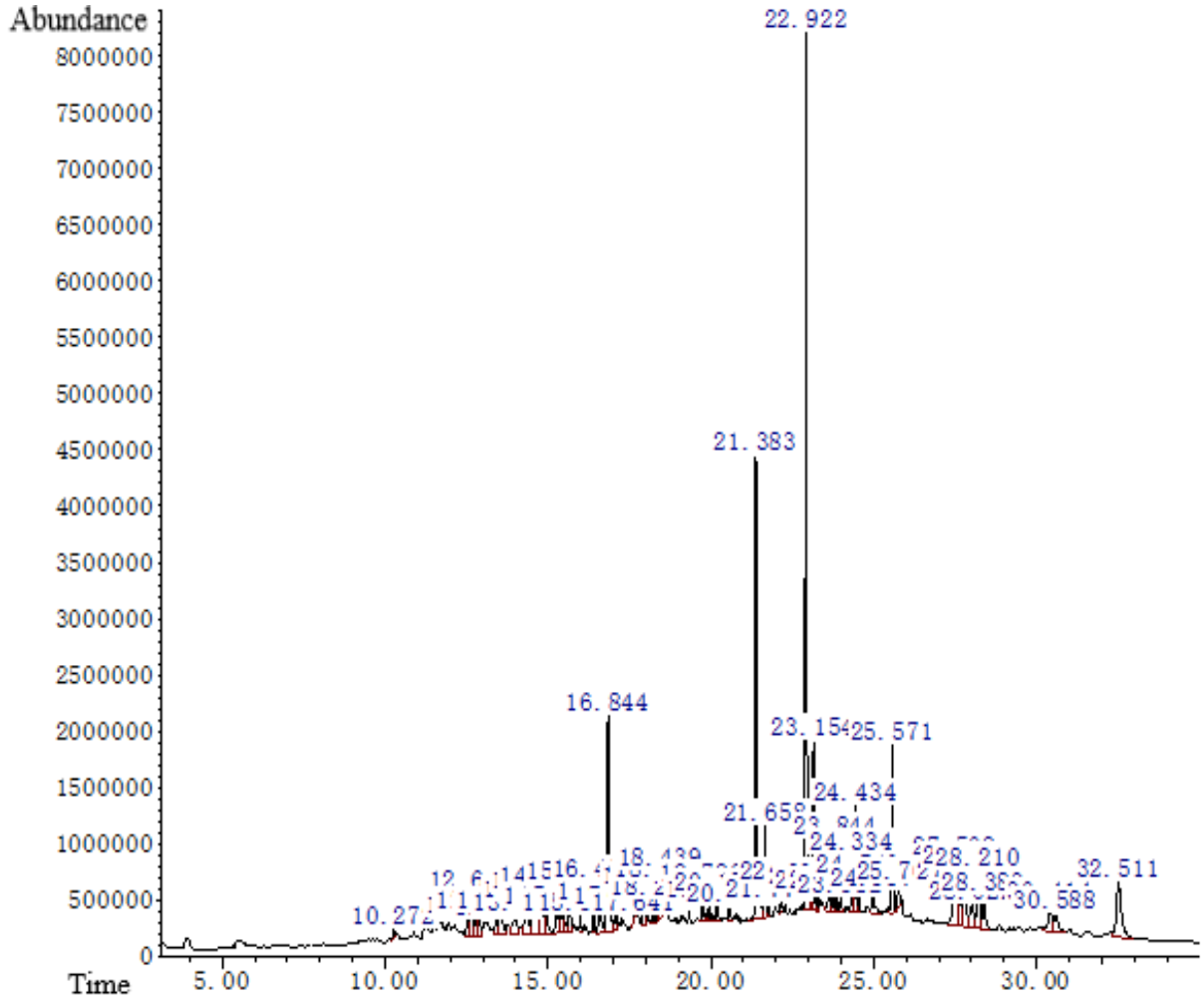

Figure 1 – GC-MS chromatogram of dichloromethane extract of the aerial part of *S. hypericifolia* L.

The active principles with their retention time (RT), molecular formula, molecular weight (MW) and concentration (%) in the dichloromethane extract of the aerial part of *S. hypericifolia* L. are presented in Table -1. The prevailing compounds in dichloromethane extract were 1-tetracosene (18.41%), 9-tricosene, (Z) - (10.55%), n-hexadecanoic acid (5.72), lanostan-3-one, 11.beta.,18-epoxy- (4.10%), stigmasta-5,22-dien-3-ol, acetate, (3.beta.)- (3.57%), tetracosanoic acid, methyl ether (3.28%), beta.- sitosterol (2.98%). 1-tetracosene exhibits cytotoxicity against AGS, MDA-MB-231, HT29, and NIH 3T3 cells [20-22]. Among the identified phytochemicals, n-hexadecanoic acid have the property of anti-inflammatory [23], antioxidant, hypocholesterolemic nematicide, pesticide, lubricant activities, antipsychotic, hemolytic, 5-alpha is a reductase inhibitors [24-26] and antibacterial [27].

The available literature supports that the identified compounds of *S. hypericifolia* L. has the biological activities like antioxidant, antibacterial, anti-inflammatory and anticancer activities. In addition to this, the results of the GC-MS profile can be used as phytochemical tool for the identification of the bioactive components.

Table 1

Percentage composition of the liposoluble components from the aerial part of *S. hypericifolia* L.

Peak	**R. time**	**Area %**	**Name**	**MF**	**MW**
1	**2**	**3**	**4**	**5**	**6**
1	10.274	0.19	6-Ethoxy-6-methyl-2-cyclohexenone	$C_9H_{14}O_2$	154.099
2	12.551	0.71	2(1H)-Naphthalenone, octahydro-1,1,4a-trimethyl-, trans-	$C_{13}H_{22}O$	194.167
3	12.644	1.44	Dodecanoic acid	$C_{12}H_{24}O_2$	200.178
4	12.789	0.68	Vanilic acid hydrazide	$C_8H_{10}N_2O_3$	182.069
5	12.865	0.85	5-(Trifluoromethyl)-2H-pyrazol-3-amine	$C_4H_4F_3N_3$	151.036
6	13.4	0.57	Benzene, 1-(1,1-dimethylethoxy)-4-methyl-	$C_{11}H_{16}O$	164.12
7	13.511	1.21	Ethanone, 1-(3,4-dihydroxyphenyl)-2-(3-nitro-[1,2,4]triazol-1-yl)-	$C_{10}H_8N_4O_5$	264.049
8	13.986	1.19	Cyclohexaneacetonitrile, 2-oxo-	$C_8H_{11}NO$	137.084
9	14.309	1.71	6-Amino-5,7-dimethyl-1,3-diazaadamantane	$C_{10}H_{19}N_3$	181.158
10	14.59	2.79	4-(1-Hydroxyallyl)-2-methoxyphenol	$C_{10}H_{12}O_3$	180.079
11	14.802	1.59	Undecanoic acid	$C_{11}H_{22}O_2$	186.162
12	14.929	1.04	3,5,7-Triamino-1-azaadamantane	$C_9H_{18}N_4$	182.153
13	15.286	0.76	9-Borabicyclo[3.3.1]nonane, 9-methyl-	$C_9H_{17}B$	136.142
14	15.363	0.89	Isosafrole Glycol	$C_{10}H_{12}O_4$	196.074
15	15.49	0.60	2,6-Dimethyl-3-aminobenzoquinone	$C_8H_9NO_2$	151.063
16	15.643	1.51	1,6-Dimethyl-9-(1-methylethylidene)-5,12-dioxatricyclo[9.1.0.0(4,6)]dodecan-8-one	$C_{15}H_{22}O_3$	250.157
1	**2**	**3**	**4**	**5**	**6**
17	15.991	0.32	Z-8-Hexadecene	$C_{16}H_{32}$	224.25
18	16.425	0.90	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	270.256
19	16.595	1.25	Hexadecenoic acid, Z-11-	$C_{16}H_{30}O_2$	254.225
20	16.841	5.72	n-Hexadecanoic acid	$C_{16}H_{32}O_2$	256.24
21	17.087	0.60	D-Xylopyranose, 5-C-(acetyloxy)-2,3,4-tri-O-methyl-, acetate	$C_{12}H_{20}O_8$	292.116
22	17.639	0.04	Tetrahydroedulan	$C_{12}H_{24}O$	196.183
23	17.962	1.07	Methyl 10-trans,12-cis-octadecadienoate	$C_{19}H_{34}O_2$	294.256
24	18.226	0.50	Hexadecane, 2,6,10,14-tetramethyl-	$C_{20}H_{42}$	282.329
25	18.379	0.85	9,12-Octadecadienoic acid (Z,Z)-	$C_{18}H_{32}O_2$	280.24
26	18.438	1.96	cis-Vaccenic acid	$C_{18}H_{34}O_2$	282.256

27	19.729	0.88	Cyclotetracosane	$C_{24}H_{48}$	336.376
28	19.95	0.39	Dodecane, 1,1'-oxybis-	$C_{24}H_{50}O$	354.386
29	20.18	0.59	Hexanoic acid, 2-tetradecyl ester	$C_{20}H_{40}O_2$	312.303
30	20.57	0.43	1-Acetoxynonadecane	$C_{21}H_{42}O_2$	326.318
31	21.386	10.55	9-Tricosene, (Z)-	$C_{23}H_{46}$	322.36
32	21.658	1.90	Methyl 20-methyl-heneicosanoate	$C_{23}H_{46}O_2$	354.35
33	21.768	0.27	1-Docosene	$C_{22}H_{44}$	308.344
34	22.167	0.54	Nonadecyl pentafluoropropionate	$C_{22}H_{39}F_5O_2$	430.287
35	22.924	18.41	1-Tetracosene	$C_{24}H_{48}$	336.376
36	23.042	0.57	Ethanol, 2-(tetradecyloxy)-	$C_{16}H_{34}O_2$	258.256
37	23.153	3.28	Tetracosanoic acid, methyl ester	$C_{25}H_{50}O_2$	382.381
38	23.263	0.40	Oleic Acid	$C_{18}H_{34}O_2$	282.256
39	23.62	0.69	Ethyl 9-hexadecenoate	$C_{18}H_{34}O_2$	282.256
40	23.731	0.44	2-Piperidinone, N-[4-bromo-n-butyl]	$C_9H_{16}BrNO$	233.042
41	23.841	1.39	Supraene	$C_{30}H_{50}$	410.391
42	23.985	0.32	12-Methyl-E,E-2,13-octadecadien-1-ol	$C_{19}H_{36}O$	280.277
43	24.334	1.33	1-Hexacosene	$C_{26}H_{52}$	364.407
44	24.436	2.16	Heptacosane, 1-chloro-	$C_{27}H_{55}Cl$	414.399
45	24.538	0.67	Hexacosanoic acid, methyl ester	$C_{27}H_{54}O_2$	410.412
1	**2**	**3**	**4**	**5**	**6**
46	24.954	0.95	10-Methyl-9-oxabicyclo[6.4.0]dodecan-1(8)-ene	$C_{12}H_{20}O$	180.151
47	25.574	3.57	Stigmasta-5,22-dien-3-ol, acetate, (3.beta.)-	$C_{31}H_{50}O_2$	454.381
48	25.761	0.63	Cyclopentane, (4-octyldodecyl)-	$C_{25}H_{50}$	350.391
49	27.503	2.98	.beta.-Sitosterol	$C_{29}H_{50}O$	414.386
50	27.621	1.63	Stigmastanol	$C_{29}H_{52}O$	416.402
51	27.783	2.52	Chola-5,22-dien-3-ol, (3.beta.,22Z)-	$C_{24}H_{38}O$	342.292
52	28.021	1.35	4,7,7-Trimethylbicyclo[2.2.1]heptan-2-one O-allyloxime	$C_{13}H_{21}NO$	207.162
53	28.208	2.31	Lupeol	$C_{30}H_{50}O$	426.386
54	28.386	1.38	Stigmasta-3,5-dien-7-one	$C_{29}H_{46}O$	410.355
55	30.416	1.39	Lanosterol	$C_{30}H_{50}O$	426.386
56	30.586	1.05	Succinic acid, 2,3,4,5-tetrafluorobenzyl tridecyl ester	$C_{24}H_{34}F_4O_4$	462.239
57	32.515	4.10	Lanostan-3-one, 11.beta.,18-epoxy-	$C_{31}H_{55}O$	442.381

Conclusion. In the present study 57 compounds from the dichloromethane extract of the aerial part of *S. hypericifolia* L. were identified by Gas Chromatography-Mass Spectrometry (GC-MS) analysis. Thus, GC-MS analysis is the first step towards understanding the nature of active principles in this medicinal plant and this type of study will be helpful for detailed study in future. Of the dichloromethane extract, the dominant compounds are 1-tetracosene (18.41%), 9-tricosene, (Z) - (10.55%), n-hexadecanoic acid (5.72), lanostan-3-one, 11.beta.,18-epoxy- (4.10%), stigmasta-5,22-dien-3-ol, acetate, (3.beta.)- (3.57%), tetracosanoic acid, methyl ester (3.28%). Further investigations in the pharmacological importance of *S. hypericifolia* L. and their diversity and

detailed biochemistry may add new knowledge to the information in the traditional medicinal system.

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SPIRAEA HYPERICIFOLIA L.-нен майда еритін компоненттерді ГХ-МС анықтау

Аңдатпа. *Spiraea hypericifolia* L. – фитохимиялық талдауы әлі зерттелмеген Қазақстанның дәрілік өсімдігі. Осы зерттеуде *Spiraea hypericifolia* L. дәрілік өсімдігінің жер үсті бөлігінің дихлорметан сығындысының химиялық компоненттері газды хроматография/масс-спектрометрия (ГХ/МС) әдісімен анықталғаны көрсетілді. ГХ-МС әдісі көмегімен *Spiraea hypericifolia* L дихлорметанды сығындысынан елу жеті қосылыстардың бар екендігін анықталды, негізгі компоненттері 1-тетракозен (18,41%), 9-трикозен, (Z)- (10,55%), н - гексадекан қышқылы (5,72%), ланостан-3-он, 11. бета., 18--эпоксидті шайыр (4,10%), стигмаста-5,22-диен - 3-ол, ацетаты, (3 бета) - (3,57%), тетракозан қышқылының метил эфирі (3,28%). Нәтижелер бойынша *Spiraea hypericifolia* L. құрамында әртүрлі биологиялық белсенділікке ие биологиялық белсенді қосылыстар бар деп қорытынды жасауға болады. Сондықтан фитофармацевтикалық маңызы бар өсімдік ретінде ұсынылады.

Түйін сөздер: *Spiraea hypericifolia* L., дихлорметан сығындысы, майда еритін компоненттер, ГХ-МС.

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ГХ-МС определение жирорастворимых компонентов из *SPIRAEA HYPERICIFOLIA* L.

Аннотация. *Spiraea hypericifolia* L. – лекарственное растение Казахстана, фитохимический анализ которого еще не изучен. В настоящем исследовании показано, что химические составляющие дихлорметанового экстракта из надземной части лекарственного растения *Spiraea hypericifolia* L. были идентифицированы с помощью анализа газовой хроматографии / масс-спектрометрии (ГХ / МС). ГХ-МС анализ дихлорметановых экстрактов *Spiraea hypericifolia* L. выявил существование пятидесяти семи соединений, в которых основными компонентами являются 1-тетракозен (18,41%), 9-трикозен, (Z)- (10,55%), n-гексадекановая кислота (5,72%), ланостан-3-он, 11.бета.,18-эпоксидная смола- (4,10%), стигмаста-5,22-диен-3-ол, ацетат, (3 бета) - (3,57%), метиловый эфир тетракозановой кислоты (3,28%). По результатам можно сделать вывод, что *Spiraea hypericifolia* L. содержит биологически активные соединения с разнообразной биологической активностью. Поэтому его рекомендуют как растение, имеющее фитофармацевтическое значение.

Ключевые слова: S. hypericifolia L., экстракт дихлорметановый, жирорастворимые компоненты, ГХ-МС.

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