

ANALYSIS OF COLICS (FOENICULUM VULGARE) SYRUP PREPARATION CONTAINING ESSENTIAL OILS BY GAS CHROMATOGRAPHY

Suleymanov T.A., Hajibayli T.A.*

Azerbaijan Medical University, Department of Pharmaceutical Chemistry, Baku, Azerbaijan

Introduction. One of the important tasks facing pharmaceutical science is the search for sources of plant preparations, rich in various groups of biologically active substances the selection of biologically active substances from these sources and the creation of new medicines based on them. **The aim of the study** was conducting an analysis of the drug Colics (foeniculum vulgare), which contains essential oils by Gas Chromatography. **Material and methods.** Foeniculum vulgare, called fennel ordinary, is an erect, branching perennial plant, which is commonly grown in vegetable and herb gardens for its foliage and seeds, with anise-flavored, which are usually collected for culinary use. The analysis was carried out on an Agilent 7890B Gas Chromatograph and Chromatography-mass MS 5977A spectrometer. Column size 30 m*250 μm *0.25 μm brand HP-5 MS Ultra Inert. Split- the sample is divided into 20 parts, the first part is introduced into the apparatus. The initial temperature of 50°C is maintained for 2 minutes. Then, for each minute the temperature rises by 5°C and reaches 250°C and is held for 5 minutes. Hexane was used as a solvent, as a carrier gas was used (He) 1.0 ml/min. **Results.** As a result of the analysis, 32 components were obtained. These compounds are mainly terpenoids and various oxidation products of saturated hydrocarbons. Compounds – 9, 10, 21, 22, 23, 24, 25, 27 predominate in the analyzed syrup «Colix».

Keywords: gas chromatography, Colics, essential oils.

Introduction. In chemical analysis, chromatography is a laboratory method for separating a mixture into its components. The mixture dissolves in a liquid solvent, called the mobile phase, which carries it through a system on which a material, called the stationary phase, is attached.

Gas Chromatography is the process of separating compounds in a mixture by introducing a gaseous or liquid sample into a mobile phase, commonly referred to as a carrier gas, and passing the gas through the stationary phase. The mobile phase is usually an inert gas or non-reactive gas such as helium, argon, nitrogen or hydrogen [1].

Therefore, the GC method can analyse volatile or gaseous substances. Substances separated in the column are eluted from there by the carrier gas flow, are registered by the detector and fixed as peaks on the chromatogram. According to the areas of the peaks in the chromatogram, each component in the mixture is identified or quantified [2].

Gas chromatography consists of a system that regulates and measures the flow rate of the carrier gas, a system for introducing a sample of the test substance, a gas chromatography column, a temperature control and temperature control system in various parts of the instrument, as well as systems for processing, recording and detecting data [3].

The determination of the identity of medicinal substances by the GC method is carried out using standard samples (“witnesses”) or by relative shelf life. When checking the identity using standard samples, one of the components of the mixture of analyzed

medicinal substances and separate chromatography of the “witness substance” under the same conditions, the superimposition of the retention times of the peaks confirms that the substances are identical. A “witness substance” is added to the sample to determine identity by relative retention time, and then analyzed. Based on the formula, the relative retention value is calculated, which is a constant value for the drug under specific conditions. Quantitative determinations are also carried out under the specified conditions, when calculating the medicinal substance [4].

The aim of the work. Conducting an analysis of the drug Colics (foeniculum vulgare), which contains essential oils by Gas Chromatography.

Materials and methods. Foeniculum vulgare, called common fennel, is an erect, branching perennial plant commonly grown in vegetable and herb gardens for its anise-flavored foliage and seeds, which are commonly harvested for culinary use [5].

Foeniculum vulgare used in folk medicine to treat a wide range of diseases associated with digestive, endocrine, reproductive, breathing system. In addition, it is also used as a lactagon for breastfeeding mothers.

Analysis was performed on an Agilent 7890B gas chromatograph and a 5977A MS gas chromatography mass spectrometer. Column size 30 m*250 μm *0.25 μm brand HP-5 MS Ultra Inert. Split i.e. the sample is divided into 20 parts; the first part is brought into the apparatus. The initial temperature of 50°C is maintained for 2 minutes. Then for each min. the temperature rises by 5°C and reaches 250°C and is held for 5 minutes. Hexane was used

*e-mail: tmedzhidova@mail.ru

as a solvent, carrier gas rate (He) 1.0 ml/min [6].

Preparation. Using Colics (foeniculum vulgare) syrup, first evaporate the syrup for 1 hour until it evaporates the water, then add some menthol to the solution and mix well, add the solution to the flask and close it, put the flask (for example, 3rd position). After holding the temperature to 50°C, wait for the GC to reach the hold temperature, record the information, and start waiting 1 hour to get results [7].

Gas chromatography includes all those chromatographic processes in which a solid or liquid supported on a solid carrier is used as the stationary phase and a gas is used as the mobile phase.

In the gas chromatography method, the components of the mixture to be separated are dissolved in a suitable organic solvent and placed on top of a sta-

tionary phase column using an injection device.

They are then carried or transported through the stationary phase in gaseous or vaporous form.

Therefore, this method can only be used to separate substances that can evaporate at operating temperatures [8].

Results and discussion. 1 gram of a crystallized sample was taken and dissolved in methanol. It was filtered through a 0.2 µm membrane filter and injected into the device (Fig.).

Sample analysis was performed on a 6977A/7890B GC/MS and HP-5MS capillary column, 30 m×0.25 mm, film thickness 0.25 µm, temperature programmed as follows: 70°-250°C at 10°C/min and 5 min held. Carrier gas – He at a flow rate of 1.2 ml/min; the injector port was 250°C. Mass spectra were taken at 70 eV (tab.).

Table
List of defined components

№	Compound	Relative exit index	Peak area %
1	Heptane, 3,5-dimethyl-	2.863	0.098
2	Hexane, 2,4-dimethyl-	2.952	0.115
3	Cyclohexane, 1,3-dimethyl-, trans-	3.053	0.240
4	3-Pentanone, 2-methyl-	3.102	0.115
5	1-Octanol, 2,7-dimethyl-	3.160	0.204
6	1,2-Cyclopentanediol, trans-	3.210	0.219
7	Hydroperoxide, 1-methylhexyl	3.278	0.187
8	1-Octanol, 3,7-dimethyl-	3.306	0.136
9	Cyclohexane, 1,3-dimethyl-, trans-	3.408	7.116
10	Cyclopentane, 1-ethyl-3-methyl-, trans-	3.843	6.304
11	Cyclohexane, 1,2,4-trimethyl-	4.159	0.073
12	2S,3S)-(-)-3-Propyloxiranemethanol	5.672	0.114
13	Hydroperoxide, 1-ethylbutyl	6.221	0.754
14	Hydroperoxide, 1-methylpentyl	6.479	0.833
15	Oxirane, [(hexyloxy)methyl]-	6.813	0.311
16	psi.-Cumene	7.582	3.195
17	Decane	7.751	0.130
18	1-Hexanol, 2-ethyl-	8.625	0.496
19	N-α,N-ω-Di-cbz-L-arginine	8.811	0.528
20	Anthranilic acid, 1,5-dimethyl-1-vinyl-4-hexenyl ester	10.943	0.216
21	l-Menthone	12.762	9.206
22	.(+)-Isomenthone	13.125	4.243
23	Menthol, cis-1,3,cis-1,4-	13.391	2.544
24	α-Terpineol	14.011	4.278
25	Carvone	15.793	18.077
26	Spiro[cyclopropane-1,6'-[3]oxatricyclo[3.2.1.0(2,4)]octane]	15.918	0.118
27	Estragole	17.211	8.169
28	Hydrazinecarboxamide, 2-(2,6-cyclooctadien-1-ylidene)-	22.546	0.169
29	Propylparaben	27.533	27.500
30	Isopropyl-4-hydroxybenzoate	27.900	0.074
31	Phenol, 2,2'-methylenebis[6-(1,1-dimethylethyl)-4-methyl-	46.304	1.835
32	Heptasiloxane, 1,1,3,3,5,5,7,7,9,9,11,11,13,13-tetradecamethyl-	46.397	0.020

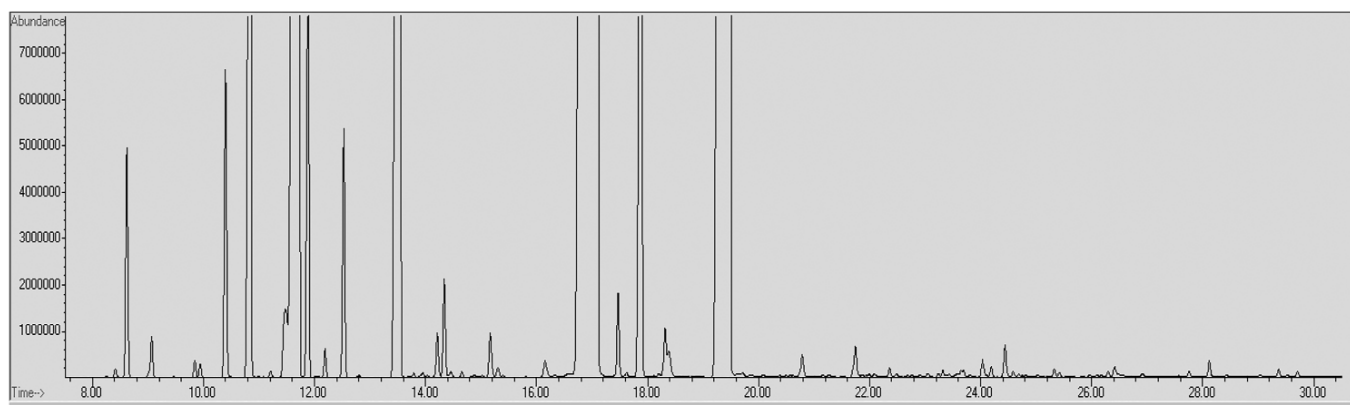


Fig. Gas chromatogram of the drug Colix.

Conclusion. As a result of the analysis, 32 components were obtained. These compounds are mainly terpenoids and various oxidation products of sat-

urated hydrocarbons. Compounds – 9, 10, 21, 22, 23, 24, 25, 27 predominate in the analyzed syrup «Colix».

REFERENCES – ƏDƏBİYYAT – LİTERATURYA

1. Joon-Bae Lee, Yong Ae Jeong, Dae Jun Ahn and Iel Soo Bang /SPME-GC/MS Analysis of Methanol in Biospecimen by Derivatization with Pyran Compound // *Molecules*.2019. (4) Dec. p. 20-25.
2. Moalesh D., Şpac AF, Dorneanu V. Validation of a gas chromatographic method for methanol determination // *Farmacia*. January (1) 2011. p.70-76.
3. Kapur B.M., Baber M. FASD: Folic acid and formic acid - an unholy alliance in the alcohol-abusing mother // *Biochem. Cell Biol.* 2018;96:189–197. doi: 10.1139/bcb-2017-0079.
4. Srinivasan S., KumariDubey k., Singhal R.S. Influence of food commodities on hangover based on alcohol dehydrogenase and aldehyde dehydrogenase activities // *Curr. Res. Nutr. Food Sci.* 2019;1:8–16. doi: 10.1016/j.crfs.2019.09.001.
5. Smith M.E. Interrelations in Ethanol and Methanol metabo-

lism // *J. Pharmacol. Exp. Ther.* 1961;134:233–237.

6. Kaya S., Mergen G., Dural E., Aliyev V., Yalçın S., Söylemezoglu T., Kayaalti Z. Simultaneous Headspace-GC–FID Analysis for Methanol and Ethanol in Blood, Saliva and Urine: Validation of Method and Comparison of Specimens // *LC GC Eur.* 2011;24:292–298.

7. Berrueta L.A., Gallo B., Vicente F. A review of solid phase extraction: Basic principles and new developments // *Chromatographia.* 1995; 40:474–483. doi: 10.1007/BF02269916.

8. Schummer C., Delhomme O., Appenzeller B., Wennig R., Millet M. Comparison of MTBSTFA and BSTFA in derivatization reactions of polar compounds prior to GC/MS analysis // *Talanta.* 2009; 77 : 1473–1482. doi: 10.1016/j.talanta.2008.09.043.

XÜLASƏ

TƏRKİBİNDƏ EFİR YAĞLARI OLAN KOLİKS PREPARATININ (FOENICULUM VULGARE) QAZ XROMATOQRAFİYASI ÜSULU İLƏ ANALİZİ

Süleymanov T.A., Hacıbəyli T.Ə.

Azərbaycan Tibb Universiteti, Əczaçılıq kimyası kafedrası, Bakı, Azərbaycan

Giriş. Əczaçılıq elminin qarşısında duran mühüm vəzifələrdən biri də bioloji aktiv maddələrin müxtəlif qrupları ilə zəngin olan bitki mənşəli preparatların mənbələrinin axtarışı, bu mənbələrdən bioloji aktiv maddələrin ayrılması və onların əsasında yeni dərman vasitələrinin yaradılmasıdır. **İşin məqsədi.** Tərkibində efir yağları olan Koliks (foeniculum vulgare) preparatının Qaz Xromatoqrafiyası üsulu ilə analizi. **Materiallar və metodlar.** Adı rəzyana adlanan *Foeniculum vulgare*, adətən kulinariya məqsədləri üçün yığılan rəzyana ətirli yarpaqları və toxumları üçün tərəvəz və ot bağlarında yetişdirilən dik, budaqlanan çoxillik bitkidir. Analiz Agilent 7890B qaz xromatoqrafında və MS 5977A xromato-kütlə spektrometrində aparılmışdır. Sütun ölçüsü 30 m*250 µm*0,25 µm marka HP-5 MS Ultra Inert istifadə olunmuşdur. Split yəni. nümunə 20 hissəyə bölünür, birinci hissə aparata gətirilir. 50°C-lik ilkin temperatur 2 dəqiqə saxlanılır. Sonra hər dəqiqə üçün. temperatur 5°C yüksəlir və 250°C-ə çatır və 5 dəqiqə saxlanılır. Həllədiçi kimi heksan, daşıyıcı qaz olaraq (He) 1,0 ml/dəq. istifadə olunur. **Nəticə.** Tədqiqat nəticəsində 32 komponent əldə edilmişdir. Bu birləşmələr əsasən terpenoidlər və doymuş karbohidrogenlərin müxtəlif oksidləşmə məh-

sullarıdır. Tədqiq edilən "Koliki" şərbətində üstünlük təşkil edən birləşmələr 9, 10, 21, 22, 23, 24, 25, 27-dir.
Açar sözlər: qaz xromatoqrafiyası, Koliks preparatı, efir yağları.

РЕЗЮМЕ

АНАЛИЗ ПРЕПАРАТА KOLIKS (FOENICULUM VULGARE), СОДЕРЖАЩЕГО ЭФИРНЫЕ МАСЛА МЕТОДОМ ГАЗОВОЙ ХРОМАТОГРАФИИ

Сулейманов Т.А., Гаджибейли Т.А.

Азербайджанский Медицинский Университет, кафедра фармацевтической химии, Баку, Азербайджан

Введение. Одной из важных задач, стоящих перед фармацевтической наукой, является поиск источников растительных препаратов, богатых различными группами биологически активных веществ, выделение биологически активных веществ из этих источников и создание новых лекарственных средств на их основе.

Цель работы. Проведение анализа препарата Koliks (foeniculum vulgare), содержащего эфирные масла методом Газовой Хроматографии. **Материалы и методы.** Foeniculum vulgare, называемый фенхелем обыкновенным, представляет собой прямостоячее, ветвящееся многолетнее растение, которое обычно выращивают в овощных и травяных садах из-за его листы и семян с анисовым вкусом, которые обычно собирают для кулинарного использования. Анализ проводили на газовом хроматографе Agilent 7890В и хромато-масс-спектрометре МС 5977А. Размер колонки 30 м*250 мкм*0,25 мкм марки HP-5 MS Ultra Inert. Сплит т.е. образец делится на 20 частей, первая часть вносится в аппарат. Начальную температуру 50°C поддерживают в течение 2 минут. Затем для каждой мин. температура повышается на 5°C и достигает 250°C и удерживается в течение 5 минут. Гексан использовался как растворитель, как носитель - газ использовался (He) 1,0 мл/мин. **Результаты.** В результате анализа было получено 32 компонента. Эти соединения представляют собой в основном терпеноиды и различные продукты окисления углеводов. В анализируемом сиропе «Колики» преобладают соединения - 9, 10, 21, 22, 23, 24, 25, 27.

Ключевые слова: газовая хроматография, препарат Koliks, эфирные масла.

Redaksiyaya daxil olub: 20.02.2023

Çapa tövsiyə olunub: 10.03.2023

Rəyçi: t.e.d. C.Y.Yusifova