

Qualitative Analysis of Oral Rinse “Splat Medical Herbs” Containing Aqueous Alcoholic Extracts Of Chamomile Flowers, Sage Leaves and Essential Oil of *Pelargonium Graveolens* by GC-FID

A. A. Filippova^{1*}, T. M. Szhenov¹, O. V. Nesterova¹, N.V. Golovina¹, N.Yu. Garnova¹, D.O. Bokov^{1,2}

¹Sechenov First Moscow State Medical University, Trubetskaya Street, 8/2, Moscow, Russian Federation, 119991

²Federal Research Center for Nutrition, Biotechnology and Food Safety, Ustyinsky Driveway, 2/14, Moscow, Russian Federation, 109240

Abstract:

The article describes the standardization approaches for toothpastes and oral rinses containing herbal treatment-and-prophylactic additives in the form of various extracts, essential oils, individual biologically active substances. The method of identification representative components of chamomile flowers and sage leaves aqueous alcoholic extracts, geranium essential oil in oral rinse composition was developed. The inclusion of a new indicator “representative components of treatment-and-prophylactic additives” in the existing interstate and state standards for oral rinses containing herbal additives is proposed.

Keywords: oral rinse, essential oil, *Pelargonium graveolens*, geraniol, gas chromatography, flame ionization detection, representative components, oral hygiene products, medicinal plant raw materials.

INTRODUCTION

The growth in the range of toothpastes and oral rinses containing herbal additives in the form of various extracts, essential oils (EOs), and individual biologically active substances (BAS) has been noted in the last ten years on the Russian market of oral hygiene products (OHPs). However, EOs are widely used as treatment-and-prophylactic additives as part of OHPs in the prevention of caries, inflammatory periodontal diseases (IPD) and oral mucosa (OM) [1]. This is due to their ability to exert a bacteriostatic and bactericidal effect on oral biofilms; its efficacy is comparable with chlorhexidine and triclosan, but the safety profile of these drugs is much better [2-5].

Technical regulations (TP TS-009-2011) “On safety of perfumery and cosmetic products”, which reflect the mandatory requirements for the composition, physical and chemical indicators, etc. are currently valid on the territory of the Russian Federation and the Member States of the Customs Union. However, information about the individual BAS of essential oil medicinal plant materials (MPM) of natural origin, which are part of the OHPs in the form of EO and other treatment-and-prophylactic additives is not available in this document. Consequently, quality control methods for them are also absent [6]. In this regard, the introduction of new physico-chemical parameters in interstate and state standards for oral rinses, regulating the quality and content of individual specific BAS of EO and treatment-and-prophylactic additives based on EO MPM, as well as the developed quality control methods for them is an urgent and well-timed task of standardizing perfumery cosmetic products. This will improve the safety of prophylactic OHPs.

The purpose of this study was to develop a method for identifying representative components of treatment-and-prophylactic additives of EO MPM (chamomile, sage aqueous alcoholic extracts and geranium EO) as part of oral rinse.

METHODS

The object of study is the oral rinse Professional series “SPLAT Medical Herbs” (GOST R 51577-2000, TR TS 009/2011). Five samples of one series (“011119” means the batch No./month/year, respectively) have been analyzed.

Oral rinse “Medical Herbs” is a solution of emerald color for local administration. According to the recipe, it contains such excipients as purified water, glycerin, glutamate diacetate tetrasodium, alcohol, benzyl alcohol, polyglyceryl-4 laurate/sebacate, polyglyceryl-6 caprilate/caprinate, coco-sodium

sulfate, sodium benzoate, flavoring, potassium sorbate, carboxymethylcellulose, citric acid, papain, maltitol, calcium lactate, sodium hydroxide, potassium thiocyanate, lactoferrin, lactoperoxidase, glucooxidase, glucose pentaacetate, CI 75810 (chlorophyllin copper complex - natural green coloring substance), without fluoride and following treatment-and-prophylactic additives:

- aqueous alcoholic extracts of chamomile flowers (*Chamomillae recutitae flores*) and sage leaves (*Salviae officinalis folia*), stevia extract (*Steviae rebaudiana folia*), licorice roots extract (*Glycyrrhizae glabrae radices*), hawthorn flowers extract (*Crataegi monogynae flores*), Sea buckthorn fruit extract (*Hippophaes rhamnoides fructus*).
- Geranium herb essential oil (*Pelargonii graveolentis herba*), camphor tree bark essential oil (*Cinnamomi camphorae cortex*).

Equipment and reagents

The “Crystallux-4000M” gas chromatograph (Meta-Chrom, Russia) was used in the analysis; the applied conditions are shown in Table 1.

Table 1. Chromatographic conditions for GC-analysis.

Chromatographic column	HP-5ms capillary quartz column (Agilent Technologies, USA), 30 m × 0.25 mm, 0.25 μm with bonded phase: 5% phenyl-95% -methylpolysiloxane
Mobile phase (MP)	nitrogen
Carrier gas flow rate	30 cm ³ /min - 30 cm ³ /min - 60 cm ³ /min
Vaporizer temperature	200 °C
Column temperature gradient	from 100 °C to 150 °C, heating 5 °C/min
Pressure on capillary column	1 atm
Detector	FID (flame ionization detector)
Detector temperature	250 °C
Hydrogen flow rate	35 cm ³ /min
Air flow rate	350 cm ³ /min
Injection sample volume	1 μl

During the study, we used the following reagents and standard samples: ethyl alcohol 95% (analytical grade), as well as

standard samples: β -farnesene (Sigma-Aldrich cat. No. 73492), α -pinene (Sigma-Aldrich cat. No. 80605), β -pinene (Sigma-Aldrich Cat. No. 80607), limonene (Sigma-Aldrich Cat. No. 62118), camphor (Sigma-Aldrich Cat. No. 148075), bornyl acetate (Sigma-Aldrich Cat. No. B-6759), cymene (Sigma-Aldrich Cat. No. C121452), terpinene (Sigma-Aldrich Cat. No. 86476), borneol (Sigma-Aldrich Cat. No. B-6759), citronellol (Sigma-Aldrich Cat. No. W230901), geraniol (Sigma-Aldrich Cat. No. 48798).

RESULTS AND DISCUSSION

The preparation method of the oral rinse test solution: 5 ml of 95% ethanol was placed in a 25 ml volumetric flask with friction-fitted lid, 5 ml of oral rinse "SPLAT Medical Herbs" was added. The resulting solution was filtered through a paper filter into a 25 ml volumetric flask and the volume of the filtrate was adjusted to the mark with the same solvent. Then, an aliquot (1 μ l) - sample volume - of the obtained extract was chromatographed. To obtain statistically reliable data fivefold test was performed.

A general view of the GC-FID-chromatogram is shown in Figure 1, the interpretation of the chromatographic peaks is presented in Table 2.

Figure 1 and Table 2 show:

1. The most important and main representative components of the three treatment-and-prophylactic additives in the composition of the oral rinse "SPLAT Medical Herbs":

- farnesene for a aqueous alcoholic extract of chamomile flowers [7-9];
- α -pinene, camphor, borneol, and bornyl acetate for the sage leaves aqueous alcoholic extract [7, 10];
- citronellol, geraniol for geranium essential oil [11, 12].

2. The most intense peaks (peaks with the largest area) correspond to 4 main substances: cymene (52%), borneol (22%), camphor (5%), terpinen (4%), limonene (3%), citronellol (2%).

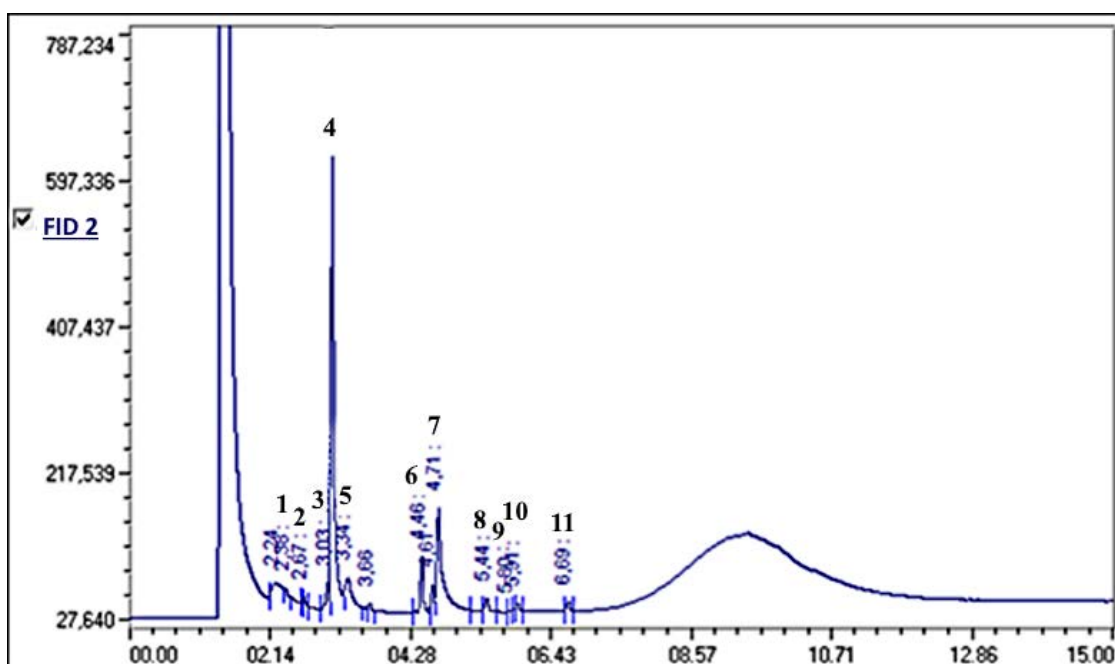


Figure 1. GC-FID-chromatogram of oral rinse "SPLAT Medical Herbs" extraction. Designations: 1 – α -pinene, 2 – β -pinene, 3 – limonene, 4 – cymene, 5 – terpinene, 6 – camphor, 7 – borneol, 8 – citronellol, 9 – farnesene, 10 – geraniol, 11 – bornyl acetate.

Table 2. Chemical composition of oral rinse "SPLAT Medical Herbs" extraction.

No	Time, min	Component	Height, mv	Area, mv * min	Height, %	Area, %	Width, sec
-	2.24	-	19.286	4.2853	1.9846	7.5624	6.76
1.	2.38	α -pinene	4.838	0.1925	0.4979	0.3397	2.32
2.	2.67	β -pinene	10.185	0.3357	1.0481	0.5924	2.12
3.	3.03	limonen	34.482	1.3946	3.5484	2.4610	7.16
4.	3.09	cymene	587.695	29.7151	60.4765	52.4391	2.52
5.	3.34	terpinene	27.692	2.1679	2.8496	3.8257	4.48
-	3.66	-	7.222	0.2363	0.7432	0.4170	2.96
6.	4.46	camphor	72.176	2.7842	7.4273	4.9134	9.16
-	4.62	-	34.467	1.4910	3.5468	2.6313	2.80
7.	4.71	borneol	135.636	12.3912	13.9575	21.8672	3.48
8.	5.44	citronellol	17.508	0.8133	1.8016	1.4353	4.40
9.	5.80	farnesene	1.751	0.0559	0.1802	0.0986	2.92
10.	5.91	geraniol	7.456	0.2877	0.7673	0.5077	1.92
11.	6.69	bornyl acetate	11.380	0.5152	1.1710	0.9091	4.44
Total			971.774	56.6659	100.0000	100.0000	6.76

CONCLUSION

The quality of oral rinse “SPLAT Medical Herbs” was confirmed by identifying representative components/specific substances-markers of treatment-and-prophylactic additives. The determination of borneol, bornyl acetate, α -pinene, and camphor gives circumstantial evidence of the presence of an essential oil fraction in the sample of an aqueous-alcoholic extract of sage leaves. The determination of farnesen is proof of presence of chamomile flowers aqueous-alcoholic extract; determination of citronellol and geraniol proves presence of geranium EO.

The method for identification representative components of treatment-and-prophylactic additives of essential oil MPM (aqueous alcoholic extracts of chamomile, sage and geranium EO) in oral rinses was developed. It will help to standardize this OHP type based on similar additives according to the new indicator “Representative components of treatment-and-prophylactic additives”. Recommendations for the implementation of such quality indicator in interstate and state standards for prophylactic oral rinses containing herbal additives are proposed.

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