

Gas chromatography with mass-spectrometric detection of the components of the essential oils from *Achillea carpatica* Blocki ex Dubovik and *Echinacea pallida* (Nutt.) Nutt.

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S u m m a r y

The essential oils were obtained from *Achillea carpatica* Blocki ex Dubovik grass and *Echinacea pallida*'s (Nutt.) Nutt. roots by method of hydrodistillation. The quantitative content of the essential oil in *A. carpatica* grass was about 0.2%; in *E. pallida* (Nutt.) Nutt. roots – 0.7%. The chemical composition was determined by GC/MS analysis, performed on Hewlett-Packard gas chromatograph HP-6890 with mass-selective detector HP-5972. Identification of individual compounds was made by comparison of their mass spectra with their internal reference mass spectra electronic libraries Nist 02 and Wiley 138k. 41 compounds of *Achillea carpatica* Blocki ex Dubovik grass essential oil and 17 substances of *Echinacea pallida*'s (Nutt.) Nutt. roots have been identified.

Key words: *Achillea carpatica*, *Echinacea pallida*, essential oil, gas chromatography with mass-spectrometric detection (GC/MS)

The *Achillea* L. genus includes perennial flowering plants of the *Asteraceae* (*Compositae*) family. It numbers more than 150 species in the world flora, about 20 of which grow in Ukraine. Unofficial species are of special scientific interest though they are widely used in traditional medicine to treat bleeding of various ethiology including uterine, hemorrhoidal, and nasal bleeding. The galenic preparations of the *A. L.* grass have a spasmolytic effect on smooth muscles of the intestines and uterine and bile canals. They are also known for their sedative, cytostatic, and antiallergic effects [1, 2].

The *Echinacea* genus includes flowering plants of the Asteraceae (Compositae) family. It numbers 9 species growing wild in North America. The representatives of the *E. Moench* family are cultivated in many countries as decorative, melliferous, feed crop plants and as a source of volatile oil. Nowadays there are only three species, which are cultivated in Ukraine and used for production of medicines: *E. purpurea* (L.) Moench, *E. pallida* (Nutt.) Nutt., *E. angustifolia* (DC) [3].

Unique complex combination of *Echinacea* biological active substance determines its pharmacological effect. The extracts of *Echinacea* stimulate phagocytosis, make more active organism antiviral resistance, have antimicrobial effect, increase production of cytotoxins and antibody kinesis [4-7].

MATERIALS AND METHODS

The essential oils were obtained from *A. carpatica* Błocki ex Dubovik grass and *E. pallida*'s (Nutt.) Nutt. (Krasunia preriy sort) roots by the method of hydrodistillation. The quantitative content of the essential oil in *A. carpatica* Błocki ex Dubovik grass was about 0,2%; in *E. pallida* (Nutt.) Nutt. (Krasunia preriy sort) roots – 0,7%.

The chemical composition was determined by GC/MS analysis, performed on Hewlett-Packard gas chromatograph HP-6890 with mass-selective detector HP-5972. At first, the essential oil was dissolved in methylene chloride. The separation of compounds was carried out, using a silica capillary HP-INNOWAX column (60 m x 0.25 mm i.d.; the film thickness was 0.25 μm (polyethylene glycol). Helium was used as carrier gas at a flow rate of 1 ml/min; initial temperature was 60°C for 5 min. then heated at the rate of 5°C/min., finally isothermally at 280°C for 10 min.

The samples volume was 1 μl . The temperature of evaporator was 280°C, of the detector – 280°C, ionization voltage – 70 eV. Mass range was from 40 to 450 *m/z*. Identification of individual compounds was made by comparison of their mass spectra with their internal reference mass spectra in electronic libraries NIST 02 and Wiley 138k. To determine their quantity, percentages of relative area were calculated without using the correction factors [8, 9]. Results are presented in tables 1–3.

Table 1.

Components of the essential oils from *A. carpatica* Błocki ex Dubovik (the content higher than 1%)

No	compound	T	grass of <i>A. carpatica</i> Błocki ex Dubovik
1	α -pinene	3.27	4.23
2	β -pinene	4.56	16.92
3	sabinene	4.78	4.69
4	1,8-cineole	6.55	9.62
5	γ -terpinene	7.49	1.91
6	camphor	14.92	1.21

7	pinocarvone	16.47	1.45
8	β -caryophyllene	17.32	7.20
9	terpinene-4-ol	17.56	3.22
10	4-thujenyl acetate	18.30	1.44
11	α -humulene	19.30	1.05
12	α -terpineol	20.26	3.36
13	germacrene D	20.43	3.09
14	caryophyllene oxide	27.10	1.97
15	chamazulene	32.50	22.93

Table 2.

Components of the essential oils from *A. carpatica* Blocki ex Dubovik (the content lower than 1%)

No.	compound	T	grass of <i>A. carpatica</i> Blocki ex Dubovik
1	α -tujene	3.32	0.47
2	camphene	3.86	0.23
3	α -phellandrene	5.61	0.63
4	α -terpinene	5.93	0.93
5	limonene	6.35	0.80
6	trans-ocymene	7.25	0.25
7	cys-ocymene	7.67	0.21
8	cymene	8.13	0.63
9	terpinolene	8.43	0.45
10	copaene	14.27	0.14
11	β -bourbonene	15.03	0.25
12	linalool	16.10	0.14
13	myrtenal	18.19	0.16
14	pinocarveol	19.02	0.89
15	δ -terpineol	19.55	0.18
16	carveol	19.70	0.12
17	α -amorphene	19.89	0.16
18	zingiberene	20.87	0.45
19	bicyclogermacrene	21.09	0.25
20	δ -germacrene	21.81	0.55
21	myrtenol	22.83	0.29
22	nerolidol	28.23	0.27
23	viridiflorol	28.79	0.61
24	spatulanol	29.35	0.51
25	eugenol	29.97	0.32
26	α -cadinol	31.16	0.73

Table 3.

Components of the essential oils from *E. pallida* (Nutt.) Nutt. (Krasunia Preriy sort) roots

No.	compound	T	roots of <i>E. pallida</i> (Nutt.) Nutt. (%)
1	1-pentadecene	16.03	0.56
2	1,8-pentadecadiene	16.68	2.64
3	1,8,10-pentadecatriene	18.17	0.54
4	4-ethyl-2-methylhexa-2,3-diene	18.26	0.62
5	tridecanon-2 + 1,8,11-heptadecatriene	23.31	0.54
6	1,8,11- heptadecatriene (isomer)	23.72	0.61
7	methyl dodecadienoate	25.20	0.18
8	cyclododecylethanone	26.52	0.21
9	pentadecan-2-one	27.93	2.66
10	cyclopentadecanone	28.42	41.28
11	cyclopentadecan (isomer)	28.51	0.86
12	8-cyclopentadecen-2-one (isomer)	29.12	2.03
13	8-cyclopentadecen-2-one	29.23	18.51
14	8,10-cyclopentadecadien-2-one	29.94	1.56
15	6-hexadecene-4-in	31.40	1.19
16	ethyl linoleate	32.14	0.67
17	methylic ester of 7,10-pentadecadiynic acid (?)	32.37	9.46
18	methylic ester of 7,10-pentadecadiynic acid (?)	33.76	8.28

RESULTS AND DISCUSSION

It was determined that chamazulene, 1,8-cineole, β -pinene prevail in the essential oil obtained from *A. carpatica* Błocki ex Dubovik grass.

Cyclopentadecanone and 8-cyclopentadecen-2-one prevail in the essential oil obtained from *E. pallida*'s (Nutt.) Nutt. (Krasunia preriy sort) roots (tab. 3).

CONCLUSIONS

The essential oils from *A. carpatica* Błocki ex Dubovik grass and *E. pallida*'s (Nutt.) Nutt. (Krasunia preriy sort) roots were obtained using the method of hydrodistillation. The quantitative content of the essential oil in *A. carpatica* Błocki ex Dubovik grass was about 0.2%; in *E. pallida* (Nutt.) Nutt. (Krasunia preriy sort) roots – 0.7%. It was also detected that the essential oil, obtained from *A. carpatica* Błocki ex Dubovik grass consists of 41 compounds and the essential oil and from *E. pallida*'s

(Nutt.) Nutt. (*Krasunia preriy* sort) roots – of 18 substances. Cyclopentadecanone and 8-cyclopentadecen-2-one prevail in the essential oil obtained from *E. pallida*'s (Nutt.) Nutt. (*Krasunia preriy* sort) roots; chamazulene, 1,8-cineole, β -pinene – in the essential oil obtained from *A. carpatica* Błocki ex Dubovik grass

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OZNACZANIE SKŁADNIKÓW OLEJKÓW ETERYCZNYCH *ACHILLEA CARPATICA* BŁOCKI EX DUBOVIK AND *ECHINACEA PALLIDA* (NUTT.) NUTT. ZA POMOCĄ SPEKTROMETRII GAZOWEJ SPRĘŻONEJ Z CHROMATOGRAFIĄ GAZOWĄ

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Streszczenie

Metodą hydrodestylacji izolowano olejki eteryczne z ziela *Achillea carpatica* Błocki ex Dubovik i korzeni *Echinacea pallida*'s (Nutt.) Nutt. Ilościowa zawartość olejku eterycznego w ziele *Achillea carpatica* Błocki ex Dubovik wynosiła około 0,2%, w korzeniach *Echinacea pallida*'s (Nutt.) Nutt. – 0,7%. Skład chemiczny oznaczono za pomocą analizy GC/MS przeprowadzonej na chromatografii gazowej Hewlett-Packard HP-6890 z detektorem masy HP-5972. Identyfikację poszczególnych składników przeprowadzono, porównując ich widma mas z ich wartościami referencyjnymi Nist 02 i Wiley 138k. W olejkach eterycznych z ziela *Achillea carpatica* Błocki ex Dubovik zidentyfikowano 41 substancji, a w korzeniach *Echinacea pallida*'s (Nutt.) Nutt. 17 substancji.

Słowa kluczowe: *Achillea carpatica*, *Echinacea pallida*, olejek eteryczny, chromatografia gazowa sprzężona ze spektrometrią gazową (GC/MS)