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Variation in the composition of the essential oil of *Valeriana officinalis* L. roots from Estonia

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Abstract. The volatile constituents from roots of *Valeriana officinalis* L. were investigated using GC and GC/MS methods. *Valerianae radix* samples were obtained from retail pharmacies or cultivated in Estonia. The roots of five *V. officinalis* samples yielded 0.28–1.16% essential oil in the cut drug, which usually corresponded (four samples) to the European Pharmacopeia standard (0.3%). The basic oil components among the identified 84 compounds were isovaleric acid (0–2.1%), α-pinene (0.4–3.6%), α-fenchene (0.6–5.8%), camphene (0.6–5.9%), bornyl acetate (8.8–33.7%), myrtenyl acetate (2.0–7.2%), alloaromadendrene (0.3–7.6%), myrtenyl isovalerate (1.1–2.5%), spathulenol (0.7–4.1%), sesquiterpene alcohol (0.8–6.6%), valerianol (0.3–16.7%), valeranone (0.5–9.4%), and valerenal (tr–14.7%). Valerian root oil from Estonia (four samples) was rich in bornyl acetate and valerenal. Bornyl acetate, valerianol, and valeranone dominated in one Estonian sample.

Key words: *Valeriana officinalis* L., essential oil, bornyl acetate, valerenal, valerianol, valeranone.

INTRODUCTION

Valerian (*Valeriana officinalis* L.) is a well-known and frequently used medicinal plant, which has a long proven history of efficacy. The plant is cultivated as a medicinal plant on a commercial scale in the northern parts of Europe and America. Valerian has been shown to encourage sleep, improve sleep quality, and reduce blood pressure [1]. The valerian root is sedative, mild anodyne, hypnotic, antispasmodic, carminative, and hypotensive. Traditionally, it has been used for hysterical states, excitability, insomnia, hypochondriasis, migraine, cramp, intestinal colic, rheumatic pains, etc. Modern interest in valerian preparations is

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focused on their use as a sedative and hypnotic [2, 3]. The *Valerianae radix* is often used as a milder alternative or a possible substitute for the stronger synthetic sedatives in the treatment of states of nervous excitation and anxiety-induced sleep disturbances [4]. The dried roots attract rats and cats, it can be used as a bait to lure them away from other areas. The essential oil from the leaves and roots is used as a flavouring in ice cream, baked goods, condiments, etc. [1].

The sedative activity of valerian root has been attributed to both the essential oil and iridoid valepotriate fractions [2]. The drug of the European Pharmacopoeia (EP) *Valerianae radix* contains not less than 5 mL/kg of essential oil for the whole drug and not less than 3 mL/kg (~0.3%) of essential oil for the cut drug, both calculated with reference to the dried drug [5]. The oil content of *V. officinalis* has been reported to vary from 0.1% to 2% [2, 6–11].

The composition of valerian root oil has been considerably studied [7–21]. The typical constituents of valerian roots are found to be valeric and isovaleric acid, monoterpenes (α-pinene, α-fenchene, camphene), monoterpenic esters (bornyl acetate, myrtenyl acetate, myrtenyl isovaleriate), oxygen containing sesquiterpenes, and valerian cyclopentanoid sesquiterpenes such as valerenal, valerenone, valerenol, valerenyl acetate, valerenic acid, and valerenyl isovalerate.

The valepotriates with rather unstable epoxide structures may be present in the root, but are generally absent from finished products. The essential oil and some compounds isolated from it (valerenic acid, valerenal, valeranone), injected intraperitoneally, show central depressive and/or muscle relaxant activity in mice [3].

The aim of this study was to determine the composition of the essential oil from *Valerianae radix* samples from Estonia. The variation in the content of the biologically active constituents was studied. The quality of Estonian valerian roots compared to the EP requirements was determined.

EXPERIMENTAL

Materials

Plant materials, *Valeriana officinalis* L. roots, were obtained from retail pharmacies or cultivated in Estonia in 2000 (sample 1 – cultivated in Järvamaa, local cultivar), in 2002 (sample 2 – cultivated in Tartumaa, seeds from the company CN Seeds, UK; and sample 3 – Vadi Gild OÜ, Põlvamaa), in 2003 (sample 4 – Energia farm, Viljandimaa), and in 2005 (sample 5 – Vadi Gild OÜ, Põlvamaa). Voucher specimens are deposited at the Institute of Pharmacy, University of Tartu, Estonia.

Isolation of essential oil

The essential oil was isolated from dried valerian roots by the distillation method described in the European Pharmacopoeia [5] using 40 g of crushed roots, a 2000 mL round-bottomed flask, and 500 mL of water as the distillation

liquid. Xylene (0.5 mL in a graduated tube) was added to take up the essential oil. The distillation time was 4 h at a rate of 3–4 mL/min.

Capillary gas chromatography

A Chrom 5 chromatograph with FID on two fused silica capillary columns with bonded stationary phases SPB-5 (30 m × 0.25 mm, Supelco) and SW-10 (30 m × 0.25 mm, Supelco) was used to carry out GC analysis. The film thickness of both stationary phases was 0.25 μm. Helium with a split ratio of 1 : 150 and flow rate of 30–35 cm/s was applied as the carrier gas. The temperature program was from 50 to 250 °C at 2 °C/min, the injector temperature was 200 °C. A Spectra-Physics SP 4100 computing integrator was used for data processing.

The oil components were identified by comparing their retention indices (RI) on two columns with the RI values of reference standards, our RI data bank, and literature data [6, 8, 10, 11, 20, 22, 23]. The percentage composition of the oils was calculated in peak areas using the normalization method without correction factors. The relative standard deviation of the percentages of the oil components of three repeated GC analyses of single oil samples did not exceed 5%.

Gas chromatography/mass spectrometry

GC/MS analysis was carried out using GCMS-QP2010 (Shimadzu, Japan) on a fused silica capillary column (30 m × 0.32 mm) with a bonded stationary phase: poly(5%-diphenyl-95%-dimethyl)siloxane) (ZB-5, Zebron). The film thickness of the stationary phase was 0.25 μm. The carrier gas was helium with the split ratio of 1 : 17, and the flow rate of 1.8 mL/min was applied. The temperature program was 2 min at 60 °C and then from 60 to 280 °C at 12 °C/min. The injector temperature was 280 °C.

RESULTS AND DISCUSSION

The identified compounds in the essential oils of five valerian samples from Estonia, and the range and mean % content and variation coefficients are presented in Table 1. High variation coefficients of the majority of compounds (>1) show that the content of these compounds strongly differs from sample to sample. Low variation coefficients (0.41–0.63) are seen for bornyl acetate, α-terpinyl acetate, (E)-β-caryophyllene, myrtenyl isovalerate, sesquiterpene alcohols, α-bisabolol, valerenal, etc. These compounds were identified in all the samples studied.

The essential oil yield for the five samples of valerian roots from Estonia varied between 0.28% and 1.16% (Table 2). In the EP [5] an essential oil content of *Valerianae radix* of at least 0.3% for cut drug is required. The oil content of *V. officinalis* has been reported to vary from 0.1% to 2% [6, 8–11]. The content of essential oil did not correspond to the EP standard in sample 4.

Table 1. Composition of the essential oil of *Valeriana officialis* L. roots from Estonia. Boldface designates the principal components

Compound	RI		Range, %	Mean, %; $n = 5$	Variation coefficient
	SPB-5	SW-10			
Isovaleric acid[MS]	840	1700	0–2.1	0.82	0.96
Tricyclene	920	1020	tr–0.1	0.09	0.24
α-Thujene[MS]	924	1026	0–0.1	0.06	0.70
α-Pinene[MS]	930	1024	0.4–**3.6**	**1.52**	0.98
α-Fenchene[MS]	944	1060	0.6–**5.8**	**2.70**	1.00
Camphene[MS]	945	1069	0.6–**5.9**	**2.90**	0.67
3-Methylvaleric acid	954	1810	0–0.2	0.08	1.37
Sabinene[MS]	970	1120	tr–0.2	0.11	0.75
β-Pinene[Ms]	972	1112	0.2–1.2	0.66	0.56
p-Cymene[MS]	1021	1273	tr–0.2	0.12	0.63
Limonene[MS]	1026	1203	0.2–2.3	1.08	0.95
β-Phellandrene[MS]	1028	1211	tr–0.7	0.23	1.16
γ-Terpinene[MS]	1054	1240	0–0.3	0.13	0.92
Isoamyl isovalerate[MS]	1102	1285	tr–0.1	0.05	0.70
Camphor	1140	1508	0–0.1	0.06	0.70
Borneol[MS]	1160	1702	0–0.6	0.28	0.96
Terpinen-4-ol[MS]	1176	1604	0.1–0.4	0.28	0.46
α-Terpineol[MS]	1190	1714	0–0.5	0.12	1.81
Myrtenol[MS]	1200	1794	0–0.5	0.18	1.07
(*E*)-Carveol	1232	1828	0.1–1.7	0.48	1.42
n-Hexyl isovalerate[MS]	1240	1450	0.1–0.3	0.22	0.38
Bornyl acetate[MS]	1285	1578	**8.8**–**33.5**	**20.18**	0.50
trans-Pinocarvyl acetate[MS]	1293	1600	0.1–0.5	0.34	0.45
Myrtenyl acetate[MS]	1324	1685	**2.0**–**7.2**	**3.64**	0.60
δ-Elemene[MS]	1337	1465	0–1.8	0.44	1.77
Terpinyl acetate[MS]	1349	1680	0.4–1.1	0.76	0.42
cis-Carveyl acetate	1366	1731	0–0.4	0.14	1.19
α-Copaene	1375	1484	0–0.4	0.12	1.33
β-Elemene[MS]	1394	1587	0–0.3	0.18	0.61
Paciforgiadiene isomer[MS] $C_{15}H_{24}$, MW = 204	1400	1528	0.1–0.8	0.56	0.48
2,6-Dimethoxy-*p*-pymene[MS]	1405	1596	0–0.2	0.04	2.23
Dihydroisolongifolene[MS]	1408		0–1.0	0.36	1.21
α-Gurjunene[MS]	1414	1520	0–1.5	0.68	1.00
(E)-β-Caryophyllene[MS]	1419	1587	1.2–**3.8**	1.92	0.57
β-Gurjunene[MS]	1429	1622	0–0.4	0.16	1.37
Epibicyclosesquiphellandrene[MS]	1434	1600	0.1–0.9	0.40	0.94
Aromadendrene[MS]	1438	1590	0–0.2	0.04	2.23
α-Guaiene[MS]	1444	1655	0–0.3	0.06	2.23
α-Humulene[MS]	1448	1622	0.3–2.2	0.88	0.89
Alloaromadendrene[MS]	1456	1632	0.3–**7.6**	**4.42**	0.68
Linalyl isovalerate[MS]	1473	1807	0.7–3.0	1.50	0.64
Germacrene D[MS]	1477	1694	0–0.7	0.30	1.11
ar-Curcumene[MS]	1480	1755	0.4–0.7	0.56	0.24
β-Ionone[MS]	1482	1926	0–3.7	0.84	1.91
Zingiberene[MS]	1490	1726	0.1–1.5	0.46	1.32

Table 1. *Continued*

Compound	RI		Range, %	Mean, %; $n = 5$	Variation coefficient
	SPB-5	SW-10			
α-Farnesene[MS]	1493	1738	0.3–2.3	1.30	0.61
Bornyl isovalerate	1500	1828	0.2–2.0	0.94	0.90
Bicyclogermacrene[MS]	1502	1714	0.1–1.4	0.59	0.86
Valencene	1507	1704	0–0.8	0.40	1.00
γ-Cadinene	1515	1748	0–1.1	0.50	0.95
δ-Cadinene	1521	1750	tr–0.6	0.30	2.35
Kessane[MS]	1542	1807	0–1.5	0.80	0.76
Valencene ketone[MS] $C_{15}H_{26}$, MW = 222	1547	2088	0.4–**3.0**	1.88	0.66
Myrtenyl isovalerate[MS]	1555	1855	1.1–**2.5**	1.52	0.41
Ledol[MS]	1559	2060	0.2–1.7	0.68	0.88
Selina-diene alcohol[MS]	1567	2100	0–0.3	0.12	1.08
Spathulenol[MS]	1574	2121	0.7–**4.1**	**2.48**	0.56
Caryophyllene oxide[MS]	1580	1989	0.2–0.7	0.46	0.45
Neryl isovalerate	1591	1875	0–0.4	0.12	1.49
Viridiflorol[MS]	1600	2078	0.1–0.6	0.34	0.61
Geranyl isovalerate	1605	1910	0–0.3	0.08	1.63
Epiglobulol[MS]	1614	2100	0.2–0.6	0.38	0.43
Sesquiterpene alcohol[MS] $C_{15}H_{24}O$, MW = 220	1625	2255	0.8–**6.6**	**4.46**	0.50
Sesquiterpene alcohol[MS] $C_{15}H_{24}O$, MW = 220	1629	2266	0–0.4	0.22	0.93
Sesquiterpene alcohol[MS] $C_{15}H_{24}O$, MW = 220	1635	2277	0.2–**3.4**	1.00	1.35
T-Muurolol[MS]	1642	2179	0.1–1.6	0.64	1.00
β-Eudesmol[MS]	1645	2272	0–1.1	0.36	1.25
Geranyl valerate*	1649		0–0.5	0.20	1.00
Valerianol	1652	2400	0.3–**16.7**	**3.86**	1.86
Kessyl alcohol*	1662		0–1.2	0.48	1.06
Valeranone[MS]	1671	2153	0.5–**9.4**	**4.22**	**0.88**
α-Bisabolol[MS]	1686	2209	0.2–0.7	0.50	0.55
(*Z*,*E*)-Farnesol*	1700	2350	0–0.2	0.06	1.48
Valerenal[MS]	1717	2207	0–**14.7**	**9.38**	0.63
Valerenol*	1729		0–0.8	0.26	1.23
Sesquiterpenoic acetate[MS] $C_{17}H_{28}O_2$, MW = 264	1772	2193	0–0.6	0.20	1.28
trans-Valerenyl acetate	1785	2224	0–0.8	0.16	2.24
Kessyl acetate[MS]	1806	2387	0.4–2.3	1.20	0.58
cis-Valerenyl acetate	1828	2226	0–1.6	0.94	0.64
Kessanyl acetate[MS]	1856	2450	0–2.0	0.64	1.38
Valerenic acid[MS]	1865	2830	0–0.9	0.28	1.46
Sesquiterpenoic acetate[MS] $C_{17}H_{26}O_2$, MW = 262	1900	2420	0–**4.1**	1.46	1.18
Palmitic acid[MS]	1954	2900	1–1.3	0.34	1.66
trans-Valerenyl isovalerate*	2052		0–1.1	0.50	0.95
Total			93.4–99.5	96.5	

tr – traces (<0.05%), * – tentatively identified, MS – identified by GC/MS.

Table 2. Principal components of the essential oil and content of essential oil in *Valeriana officinalis* L. roots from Estonia, %. Boldface designates the highest concentrations

Components	Sample No				
	1	2	3	4	5
Isovaleric acid	2.1	nd	0.8	0.8	0.4
α-Pinene	0.4	0.6	0.4	2.6	3.6
α-Fenchene	0.8	0.6	0.8	**5.5**	**5.8**
Camphene	3.1	1.9	0.6	3.0	**5.9**
Bornyl acetate	**33.7**	**27.1**	**8.8**	**13.4**	**18.1**
Myrtenyl acetate	**7.2**	2.0	2.6	3.9	2.4
Alloaromadendrene	0.3	**7.6**	**6.4**	5.4	2.4
Spathulenol	0.7	3.6	4.1	1.8	2.2
Myrtenyl isovalerate	1.1	1.1	2.5	1.8	1.1
Sesquiterpene alcohol	0.8	5.7	**6.6**	4.6	4.6
Valerianol	**16.7**	0.6	0.5	1.2	0.3
Valeranone	**9.4**	0.5	2.8	**6.7**	1.7
Valerenal	tr	**8.4**	**14.1**	**14.7**	**9.7**
Valerenic acid	nd	nd	0.5	nd	0.9
Oil yield, %	1.16	1.00	0.40	0.28[a]	0.53

nd – not determined, tr – traces (<0.05%).
[a] Below the minimum limit of EP (0.3% for the cut drug).

A total of 84 compounds, representing more than 93% of the total oil, were identified in the valerian samples studied. All the identified components have been reported previously in *V. officinalis* root oil [6, 8–11, 13–21]. It was possible to determine the compound group for nine peaks only by mass spectral data. Three unknown sesquiterpene alcohols with the same mass fragmentation pattern were detected (the $[M^+]$ at m/z 220 and characteristic peaks at m/z 43 (base peak)), 91, 105, 41, 93, 79, 119, and 107).

The basic oil components were isovaleric acid (0–2.1%), α-pinene (0.4–3.6%), α-fenchene (0.6–5.8%), camphene (0.6–5.9%), bornyl acetate (8.8–33.7%), myrtenyl acetate (2.0–7.2%), alloaromadendrene (0.3–7.6%), myrtenyl isovalerate (1.1–2.5%), spathulenol (0.7–4.1%), sesquiterpene alcohol (0.8–6.6%), valerianol (0.3–16.7%), valeranone (0.5–9.4%), valerenal (tr–14.7%), and valerenic acid (0–0.9%) (Table 2).

In four Estonian samples (2–5) bornyl acetate and valerenal dominated. These compounds were found to be the main compounds in valerian root oils in earlier studies [7–11, 16] too. Valeranone was the main compound in samples 1 and 4. The content of valerianol was highest in sample 1. A high content of valerianol was found in oil of *V. officinalis* roots from Serbia [16] and in some samples from The Netherlands [8]. Samples 4 and 5 from Estonia were comparatively rich in monoterpenic hydrocarbons α-fenchene and camphene (total 1.2–11.7%). Oil rich in monoterpenes (α-pinene, camphene, α-fenchene) was isolated from valerian roots in Switzerland [6]. The main sesquiterpene alloaromadendrene dominated in Estonian samples 2 and 3.

CONCLUSIONS

Several chemotypes of the essential oil of valerian roots have been distinguished earlier in the literature [8, 11], e.g. a valerianol type, and valeranone, cryptofauronol, and valerenal types. Valerian root oil from Estonia (four samples) was also rich in bornyl acetate and valerenal. Bornyl acetate, valerianol, and valeranone dominated in one Estonian sample. The *Valerianae radix* cultivated in Estonia usually (four samples) corresponds to the EP standards in the aspect of the essential oil contents.

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Eeterliku õli koostise varieeruvus Eestist pärineva *Valeriana officinalis* L. juurtes

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On uuritud hariliku palderjani (*Valeriana officinalis* L.) juurte lenduvaid ühendeid, kasutades GC- ja GC/MS-meetodit. Droogi *Valerianae radix* näidised on saadud jaemüügiapteekidest või kultiveeritud Eestis. Uuritud viies palderjani peenestatud droogis on eeterliku õli sisaldus 0,28–1,16%, neljas droogiproovis vastab see Euroopa farmakopöa eeterliku õli sisalduse standardnõudele (0,3%). 84 identifitseeritud komponendi hulgas on põhikomponentideks isopalderjanhape (0–2,1%), α-pineen (0,4–3,6%), α-fenheen (0,6–5,8%), kamfeen (0,6–5,9%), bornüülatsetaat (8,8–33,7%), mürtenüülatsetaat (2,0–7,2%), alloaromadendreen (0,3–7,6%), mürtenüülisovaleraat (1,1–2,5%), spatulenool (0,7–4,1%), seskviterpeenne alkohol (0,8–6,6%), valerianool (0,3–16,7%), valeranoon (0,5–9,4%) ja valerenaal (jälgedes – 14,7%). Eestist pärinevate palderjanijuurte eeterlik õli (neli droogiproovi) on rikas bornüülatsetaadi ja valerenaali sisalduselt. Ühes droogiproovis domineerivad bornüülatsetaat, valerianool ja valeranoon.