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Research Article

Essential oil profile of *Valeriana acutiloba* Rydb. (Caprifoliaceae) from Utah (USA)

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Abstract

Valeriana acutiloba Rydb. is an essential oil-bearing plant that is native to much of the western region of the United States, including the state of Utah. Roots of the flowering plant were collected, and steam distilled. The resulting essential oils (n = 3) were analyzed by GC/MS and GC/FID. Prominent compounds of *V. acutiloba* essential oil include α-fenchene (avg. 5.0%), bornyl acetate (avg. 6.0%), β-gurjunene (avg. 6.8%), kessane (avg. 5.5%), and calarene epoxide (avg. 5.6%). The essential oil contains several prominent unknown compounds that were unidentifiable with commercially available mass spectral libraries, including an unidentifiable compound with an average relative area of 19.6% (prominent mass spectral ions: 43.0, 109.1, 125.1, 238.2). The essential oil yield was 0.16% (*w*/*w*). This study is the first, to the best knowledge of the authors, to characterize the essential oil profile of this species and establishes a foundation for future chemotaxonomic and other investigations.

1. Introduction

Valeriana acutiloba Rydb. is a perennial flowering plant that is native to the Intermountain Region, including the state of Utah [1-4]. *Valeriana acutiloba* had long been part of the Valerianaceae family [2,4] but is now part of the Caprifoliaceae family [5-7].

Valeriana spp. throughout the world have long been referenced in folklore and used in medicine, cosmetics, and foods [8,9]. Of these species, *V. officinalis* is the most studied, primarily due to its purported benefits of relaxation and sleep induction [8,9]. In North America, *Valeriana spp.* have been used by Native peoples [10-12] as well. However, there is little mention of *V. acutiloba*, apart from phylogenetic and taxonomic literature. Cronquist and associates [2] account for 3 varieties of *V. acutiloba* that are native to the Intermountain Region, which are poorly defined taxonomically. Population dispersity and high elevation growth may account for the sparse usage, literature, and research on *V. acutiloba*.

In this study, *V. acutiloba* plant material was collected from the Oquirrh mountain range (Tooele, Utah, USA), steam distilled, and the resulting essential oils analyzed by GC/MS and GC/FID. The plant yielded a complex, unique essential oil. This study is the first, to the best knowledge of the authors, to characterize the essential oil profile of this species and establishes a foundation for future chemotaxonomic and other investigations.

2. Materials and methods

Valeriana acutiloba plant material was collected on June

25, 2021, from a native population located on public lands (Bureau of Land Management) from the Oquirrh mountain range in Tooele, Utah, USA (40°28′3″ N 112°10′23″ W; 2832 m elevation). Roots from the flowering plant were cleaned and divided into three groups to determine weight, yield, and composition of the extracted essential oil (Fig. 1). For simplicity and consistency, each sample is referred to by a letter, A-C. Representative voucher samples are held in two herbaria: Utah Valley University Herbarium (*V. acutiloba* Rydb., Wilson 2022-01 (UVSC)) and Young Living Aromatic Herbarium (*V. acutiloba* Rydb., Wilson 2022-02 (YLAH)) (Fig. 2).

Figure 1. Photo of distilled and dried *Valeriana acutiloba* root

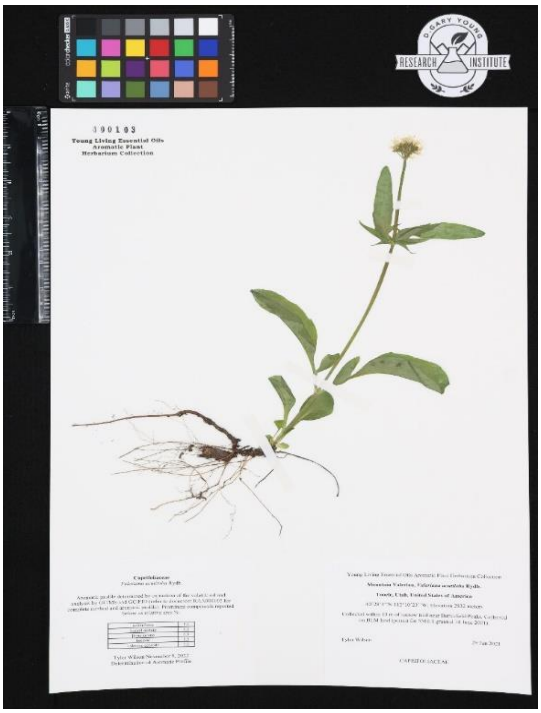

Figure 2. Photo of *Valeriana acutiloba* voucher sample. Submitted to Young Living Aromatic Herbarium (YLAH).

Plant material was prepared for laboratory-scale distillation as follows: roots were cleaned, separated into three groups, bagged, and stored at -20 ± 2 °C until steam distilled. Steam distillation was performed in triplicate, resulting in 3 distillations over the course of this project.

Laboratory-scale distillation was as follows: 1.5 L of water added to 2 L steam generator that fed into a 2 L distillation chamber, plant material accurately weighed and added to the distillation chamber, distillation for 1.5 h from pass-over by indirect steam, essential oil separated by a cooled condenser and Florentine flask. Essential oil samples were each filtered and stored at room temperature in a sealed amber glass bottle until analysis.

The percent yield was calculated as the ratio of the mass of processed plant material immediately before distillation to the mass of essential oil produced, multiplied by 100.

Essential oil samples were analyzed, and volatile compounds identified, by GC/MS using an Agilent 7890B GC/5977B MSD (Agilent Technologies, Santa Clara, CA, USA) and Agilent J&W DB-5, 0.25 mm x 60 m, 0.25 µm film thickness, fused silica capillary column. Operating conditions: 0.1 µL of sample (20% soln. for essential oils in ethanol), 100:1 split ratio, initial oven temp. of 40 °C with an initial hold time of 5 min., oven ramp rate of 4.5 °C per min. to 310 °C with a hold time of 5 min. The electron ionization energy was 70 eV, scan range 35-650 amu, scan rate 2.4 scans per sec., source temp. 230 °C, and quadrupole temp. 150 °C. Volatile compounds were identified using the Adams volatile oil library [13] and 2020 NIST Mass Spectral Library using Chemstation library search in conjunction with retention indices. Volatile compounds were quantified and are reported as a relative area percent by GC/FID using an Agilent 7890B GC and Agilent J&W DB-5, 0.25 mm x 60 m, 0.25 µm film thickness, fused silica capillary column. Operating conditions: 0.1 µL of sample (20% soln. for essential oils in ethanol, 1% for reference compounds in ethanol, 0.1% soln. for C7-C30 alkanes in hexane), 25:1 split ratio, initial oven temp. of 40 °C with an initial hold time of 2 min., oven ramp rate of 3.0 °C per min. to 250 °C with a hold time of 3 min. Essential oil

samples were analyzed in triplicate by GC/FID to ensure repeatability (standard deviation ≤ 1 for all compounds). Compounds were identified using retention indices coupled with retention time data of reference compounds (MilliporeSigma, Sigma-Aldrich, St. Louis, MS, USA).

3. Results and discussion

The complete aromatic profile of *V. acutiloba* is detailed in Table 1. Each reported value is an average for each essential oil sample (n = 3) analyzed in triplicate to ensure reproducibility (SD < 0.5 for all compounds). Yields are detailed in Table 2.

Table 1. Aromatic profile of *Valeriana acutiloba* essential oil from roots (n = 3).

KI	Compound Name	A	B	C
921	Tricyclene	0.1	0.1	0.1
924	α-Thujene	0.1	0.1	0.1
932	α-Pinene	1.3	2.6	2.5
945	α-Fenchene	3.2	6.2	5.7
946	Camphene	1.5	2.9	2.9
969	Sabinene	0.3	0.5	0.5
974	β-Pinene	0.5	1.0	1.0
1022	o-Cymene	0.8	1.1	1.2
1227	Isothymol methyl ether	0.6	0.8	0.7
1232	Thymol methyl ether	0.5	0.5	0.5
1241	Carvacrol methyl ether	0.1	0.1	0.1
1284	Bornyl acetate	6.2	5.6	6.1
1395	Benzyl isovalerate	0.1	0.1	0.1
1409	α-Gurjunene	0.2	0.2	0.1
1424	Thymohydroquinone dimethyl-ether	0.3	0.3	0.4
1429	1,1,7,7a-tetramethyl-1a, 2,6,7,7a,7b-hexahydro-1H-cyclopropa [a] naphthalene	1.9	1.3	0.5
1431	β-gurjunene	8.1	6.0	6.3
1496	Viridiflorene	0.2	0.1	0.1
1500	α-Muurolene	0.1	0.1	0.1
1503	Dihydro-β-agarofuran	1.7	1.3	1.6
1513	γ-Cadinene	0.2	0.1	0.1
1514	Cubebol	0.2	0.1	0.1
1522	δ-Cadinene	0.2	0.2	0.2
1529	Kessane	5.9	5.4	5.1
1581	Unknown	0.2	1.1	2.6
1606	Unknown	0.8	1.0	1.0
1613	Unknown	1.5	1.1	1.2
1617	Calarene epoxide	4.1	7.3	5.4
1661	Unknown	1.4	1.4	1.0
1663	Unknown	1.1	0.7	0.7
1686	Unknown	10.4	9.4	6.2
1691	Unknown	0.8	1.0	0.9
1735	Unknown	0.9	1.4	0.8
1839	Unknown	1.7	1.5	1.5
1853	Unknown	2.7	2.7	1.9
1883	Unknown	21.7	18.1	18.9
1897	Unknown	t	1.0	6.7
Sum of identified compounds		38.4	44.0	41.5

Note: Compounds not detected in a sample are denoted as not detected (nd) and values less than 0.1% as traces (t). Unidentified compounds that are less than 1.0% but not present in all samples are not included. KI is the Kovat's Index using a linear calculation on DB-5 column [13]. Relative area percent in determined by GC/FID. Essential oils were analyzed in triplicate to ensure reproducibility (SD < 0.5 for all compounds). Each essential oil sample is referred to by a letter, A-C. **KI** indicated in bold font was calculated using alkane standards.

Essential oil obtained from steam distillation of *V. acutiloba* roots are largely composed of α-fenchene (avg. 5.0%), bornyl acetate (avg. 6.0%), β-gurjunene (avg. 6.8%), kessane (avg. 5.5%), and calarene epoxide (avg. 5.6%). These prominent compounds are characteristic of essential oils from other *Valeriana spp.* In *V. officinalis* essential oil, α-fenchene, bornyl acetate, and kessane have been found to be prominent volatile compounds [14,15]. In *V. jatamansi* essential oil, β-gurjunene is a prominent volatile compound [16]. While two volatile compounds, isovaleric acid and valerenic acid, are commonly found in essential oils of *Valeriana spp.*, neither compound was detected in samples from this study [14,15].

In this study, we were only able to identify 41.3 % (avg.) of each essential oil sample, leaving many compounds unidentified. The most intriguing of these is a compound that comprises, on average, 19.6% of the essential oil profile. The mass spectra of this compound in shown in Fig. 3. The incomplete identification of volatile compounds is in stark contrast to typical analyses from the current authors, where typically > 98% of the volatile profile is identified [17,18]. To identify these unknown compounds, future investigations could include separation based on compound class with silica gel and solvents or isolation of the unknown compounds

using preparative chromatography followed by analytical techniques suitable for structure elucidation such as nuclear magnetic resonance spectroscopy (NMR), crystallography, and exact mass, mass spectrometry (ex.: GC/TOF/MS). Future investigations can also explore functionality of volatile compounds isolated from this species, as well as their potential application for human use.

Table 2. Yield data, including mass of plant material distilled (g), essential oil yield (g), and calculated yield (w/w) from *Valeriana acutiloba* samples (n = 3).

Sample	Plant Mass Distilled (g)	Essential Oil Yield (g)	Essential Oils Yield (w/w)
A	120.85	0.10	0.08
B	142.52	0.28	0.20
C	137.03	0.29	0.21
Avg.:	133.47	0.22	0.16
Avg.: %RSD (n = 3)			43.05

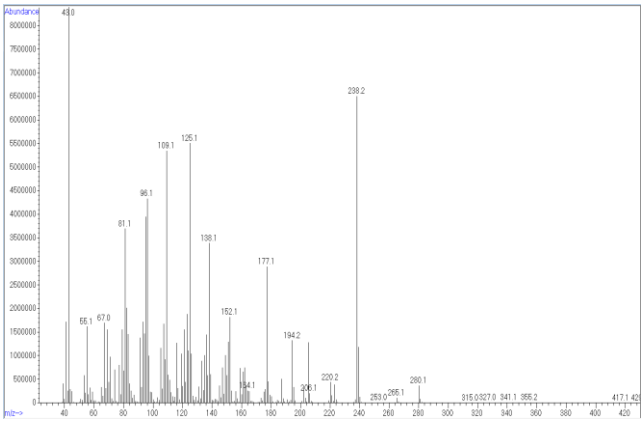

Figure 3. The mass spectra of a prominent unknown in *Valeriana acutiloba*, making up on average 19.6% of the essential oils analyzed. Prominent mass spectral ions include 43.0, 109.1, 125.1, 238.2 and the calculated KI is 1883.

4. Conclusions

The essential oil of *Valeriana acutiloba* is complex and comprised of many unidentifiable sesquiterpenes. Using commercially available mass spectral libraries (NIST 2020) [13]. the authors were only able to perform limited identifications with confidence. With so many apparent unique constituents, this essential oil warrants much further in-depth analysis with the aim of identifying these unknowns and adding them to mass spectral libraries. A complete identification of volatile compounds may also aid in future chemotaxonomic research of this species. Future investigations can also explore functionality of volatile compounds isolated from this species, as well as their potential application for human use.

Authors' contributions

Conceptualization, T.M.W.; Methodology, T.M.W.; Software, A.P. and T.M.W.; Validation, R.E.C.; Formal Analysis, A.P. and T.M.W.; Investigation, C.P., E.A.Z, and T.M.W.; Resources, C.P., E.A.Z, and T.M.W.; Data Curation, A.P.; Writing – Original Draft Preparation, A.P. and T.M.W.; Writing – Review & Editing, A.P., E.A.Z, R.E.C., and T.M.W.; Funding Acquisition, R.E.C.

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Conflicts of interest

The authors declare no conflict of interest. The funding entity had no role in the design of the study, in the collection, analysis, or interpretation of data, in the writing of the manuscript, or in the decision to publish the results.

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