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The Essential Oil of *Balsamorhiza sagittata* from Southwestern Idaho: Chemical Composition and Enantiomeric Distribution

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Abstract

Background/Objective: Arrowleaf balsamroot (*Balsamorhiza sagittata* (Pursh) Nutt., Asteraceae) is a conspicuous forb growing in open hillsides and prairies at mid to upper elevations in the Intermountain West and Rocky Mountain regions. The plant is important forage for deer and elk and several Native American tribes used arrowleaf balsamroot as food and medicine. The volatile phytochemicals of *B. sagittata* have not been previously examined. **Methods/Results:** Nine individual samples of *B. sagittata* were collected, the essential oils obtained by hydrodistillation using a Likens-Nickerson apparatus in yields of 0.069-0.956%, and the essential oils analyzed by gas chromatographic techniques (GC-MS, GC-FID, and chiral GC-MS). The major components in the leaf essential oil of *B. sagittata* were (−)-germacrene D (10.8-34.5%), (+)-β-phellandrene (6.4-19.4%), (−)-(*E*)-β-caryophyllene (1.4-15.0%), and (*E*)-β-ocimene (3.1-8.4%). **Conclusion:** There was modest variation in composition or yield with respect to geographical location or elevation in the samples from southwestern Idaho, but comparison with essential oils from locations outside of Idaho should more completely define the volatile phytochemistry of this plant.

Keywords

arrowleaf balsamroot, Great Basin, volatile phytochemistry, chiral GC-MS, multivariate analysis

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Introduction

Balsamorhiza sagittata (Pursh) Nutt. (Family Asteraceae, Heliantheae tribe, Engelmanniinae subtribe) (Figure 1) occurs throughout western North America from Alberta and British Columbia, east to the Dakotas, and south to Southern California, Nevada, Colorado, and Arizona (Figure 2). The preferred habitats for *B. sagittata* are open hillsides and prairies at mid to upper elevations in the Intermountain West and Rocky Mountain regions and is most common in upland rangeland shrubland types dominated by antelope bitterbrush (*Purshia tridentata*), basin big sagebrush (*Artemisia tridentata* subsp. *tridentata*), Wyoming big sagebrush (*A. t.* subsp. *wyomingensis*), and mountain big sagebrush (*A. t.* subsp. *vaseyana*).[1,2]

B. sagittata was important in the ethnobotanical traditions of Native American cultures.[3] For example, the Cheyenne used a decoction of the plant to treat colds and stomach pains; the Flathead used a poultice of the leaves on burns; the Shuswap used an infusion of the leaves as a wash for poison ivy and running sores.

Leaves of *B. sagittata* were found to contain a single major flavonoid, 6-hydroxy-kaempferol 7-methyl ether.[4] From the aerial parts of *B. sagittata*, several sesquiterpene lactones, guaianolides, heliangolides, germacranolides, eudesmane acids, and geranylnerol derivatives were isolated and characterized.[5,6] As part of our interest in characterizing the volatile phytochemistry of Great Basin aromatic plants, the focus of this study was to obtain and analyze the leaf essential oil of *B. sagittata*; as far as we are aware, there have been no reports on the essential oil of *B. sagittata* or any other *Balsamorhiza* species.

Results and Discussion

Essential Oil Composition

The leaves of *B. sagittata* were hydrodistilled to give pale blue essential oils in yields ranging from 0.069% to 0.917% (w/w). Gas chromatographic analysis of the nine essential oil samples led to identification of a total of 184 compounds (Supplemental Table 1). The major components are summarized in Table 1.

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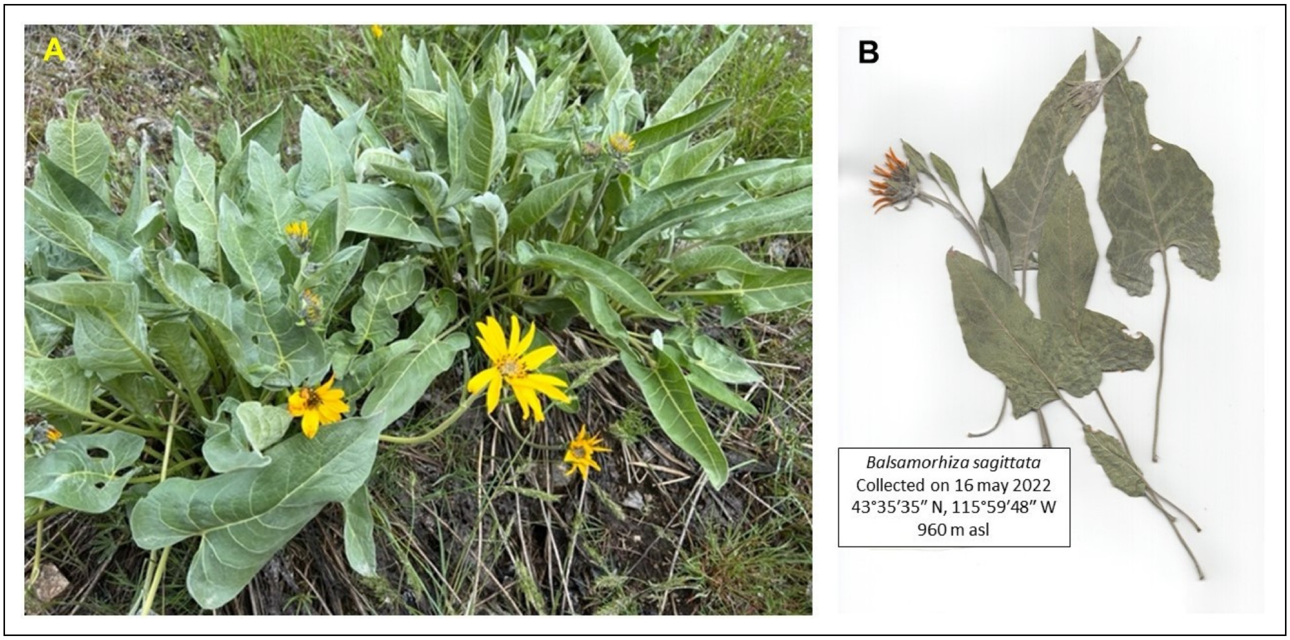

Figure 1. *Balsamorhiza sagittata*. (A) Photograph of plant (by K. Swor). (B) Scan of pressed plant (by W.N. Setzer).

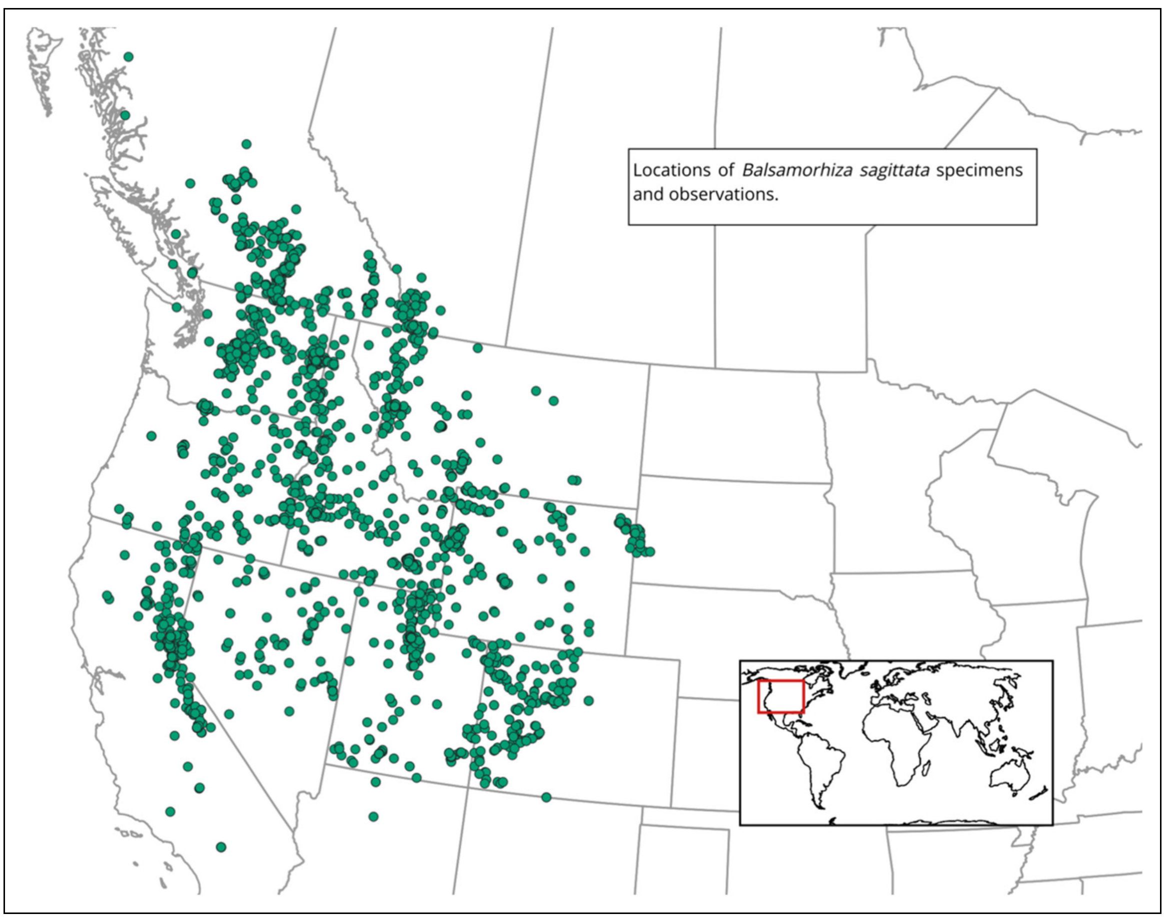

Figure 2. Range of *Balsamorhiza sagittata* in North America (by Looncreek—Own work, CC BY-SA 4.0, https://commons.wikimedia.org/w/index.php?curid=98731965).[24]

Table 1. Major Components (%) of the Leaf Essential Oils of *Balsamorhiza sagittata*.

RI_{calc}	RI_{db}	Compound	Sample number 1	2	3	4	5	6	7	8	9
782	782	Prenol	0.7	tr	1.2	2.7	1.2	1.0	0.3	0.5	0.2
973	971	Sabinene	3.4	0.5	3.2	7.4	2.0	1.2	2.9	4.1	1.4
988	989	Myrcene	1.2	1.1	2.2	2.6	1.9	1.1	2.0	2.3	2.2
991	986	6-Methyl-5-hepten-2-one	0.8	0.6	1.3	1.3	1.6	1.2	1.0	0.2	1.6
1005	1004	*p*-Mentha-1(7),8-diene	0.8	0.8	3.1	1.6	1.1	0.7	1.3	1.0	1.0
1007	1007	α-Phellandrene	0.1	1.3	-	4.7	6.6	3.2	tr	7.3	12.1
1024	1025	*p*-Cymene	0.2	1.4	5.5	0.5	1.1	0.7	0.2	1.8	1.0
1029	1030	Limonene	4.4	5.0	13.7	2.7	6.1	1.3	2.5	5.7	5.4
1030	1031	β-Phellandrene	8.8	10.0	4.9	19.4	11.8	6.4	13.7	9.0	12.7
1035	1034	(*Z*)-β-Ocimene	3.9	3.8	0.3	1.2	2.4	2.8	6.8	7.7	9.9
1045	1045	(*E*)-β-Ocimene	4.3	4.9	0.1	3.7	7.4	3.1	8.0	8.4	7.7
1173	1173	Borneol	0.7	2.0	1.9	0.5	0.7	4.0	0.1	1.8	0.1
1188	1187	Cryptone	0.1	0.5	5.4	0.1	0.1	0.1	0.5	0.2	tr
1420	1417	(*E*)-β-Caryophyllene	15.0	2.5	1.0	1.4	4.9	2.4	14.5	2.5	13.1
1455	1454	α-Humulene	2.0	1.7	1.0	0.6	1.2	1.1	1.9	0.9	1.4
1481	1480	Germacrene D	27.3	34.5	-	10.8	24.5	24.3	15.2	20.1	11.6
1495	1497	Bicyclogermacrene	5.6	3.3	-	0.3	2.9	0.9	1.8	2.8	-
1575	1576	Spathulenol	1.3	3.3	6.0	-	0.7	0.3	1.7	0.9	0.2
1655	1656	β-Eudesmol	-	-	-	12.7	0.4	4.1	1.9	1.6	-
1656	1655	α-Eudesmol	0.9	0.5	-	8.3	1.7	4.1	2.3	2.0	0.5

Abbreviations: RI_{calc}, retention index determined with respect to a homologous series of *n*-alkanes on a ZB-5 ms column using the linear equation of van den Dool and Kratz.[25] RI_{db}, reference retention index obtained from the databases.[20–23]

In order to compare the chemical compositions of *B. sagittata* leaf essential oils, an agglomerative hierarchical cluster (AHC) analysis was carried out (Figure 3). The AHC analysis revealed eight of the nine samples show at least 48% similarity in composition. However, one sample (#3) was very different. Samples #1, #2, and #4 through #9 were rich in germacrene D (10.8-34.5%), β-phellandrene (6.4-19.4%), (*E*)-β-caryophyllene (1.4-15.0%), and (*E*)-β-ocimene (3.1-8.4%). In contrast, limonene (13.7%), spathulenol (6.0%), *p*-cymene (5.5%), cryptone (5.4%), and β-phellandrene (4.9%) were the major components in sample #3, while germacrene D was not observed. A principal component analysis (PCA) was also carried out to verify the results of the AHC analysis (Figure 4). The PCA corroborates the AHC analysis; samples #1, #2, and #4 through #9 all show positive correlations with germacrene D, β-phellandrene, (*E*)-β-caryophyllene, and (*E*)-β-ocimene. Sample #3, on the other hand, positively correlates with limonene, spathulenol, *p*-cymene, and cryptone. It is not at all obvious why the chemical composition of sample #3 is so different from the other samples. It was collected on the same day from the same location as sample #2. Sample #1 was collected in May 2022 from one location and samples #2 and #3 were collected in May 2022 from a different location. On the other hand, samples #4 through #9 were collected in May 2023, from two different locations. The chemical compositions of #1, #2, and #4 through #9 are similar regardless of their different collection years, geographical locations, and elevations; sample #3 is just an outlier. The dendrogram (Figure 3) indicates 95% similarity between essential oil samples #2 and #6. Likewise, there is 94% similarity between essential oils #5 and #8. These four essential oil samples were all rich in germacrene D, but the #2/#6 subcluster had overall lower concentrations of monoterpene hydrocarbons than the #5/#8 subcluster.

There have been no previous reports on essential oils of *Balsamorhiza* species for comparison. However, several members of the Heliantheae tribe, Engelmanniinae subtribe of the Asteraceae have been published, and a comparison with the major components of *B. sagittata* is presented in Table 2.

Both germacrene D and (*E*)-β-caryophyllene seem to be major components in the Engelmanniinae subtribe of the Asteraceae, but those two sesquiterpenes are common components of many essential oils and may not, therefore, define this subtribe of the Asteraceae.

Terpenoid Enantiomeric Distributions

As a further characterization of the essential oil of *B. sagittata*, chiral GC/MS was carried out (see Table 3). Both α-pinene and β-pinene were virtually racemic in *B. sagittata* essential oils. The (−)-enantiomers were the dominant stereoisomers, when observed, for camphene, limonene, borneol, (*E*)-β-caryophyllene, and germacrene D, while (+)-sabinene, (+)-α-phellandrene, (+)-β-phellandrene, (+)-δ-cadinene, and (+)-(*E*)-nerolidol were dominant. There seems to be little consistency in enantiomeric distributions of monoterpenoids in the Asteraceae. For example, several *Gnoxys* species have been examined. *Gnoxys miniphylla* Cuatrec. showed 98.2% ee

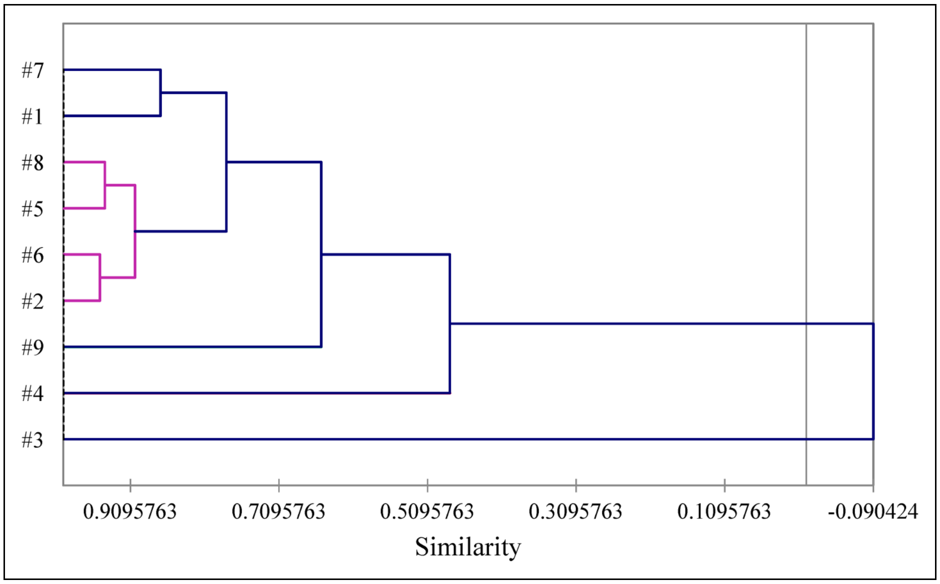

Figure 3. Dendrogram obtained by cluster analysis of the major components of *Balsamorhiza sagittata* leaf essential oils based on Pearson similarity correlation and using the unweighted pair-group method with arithmetic average (UPGMA).

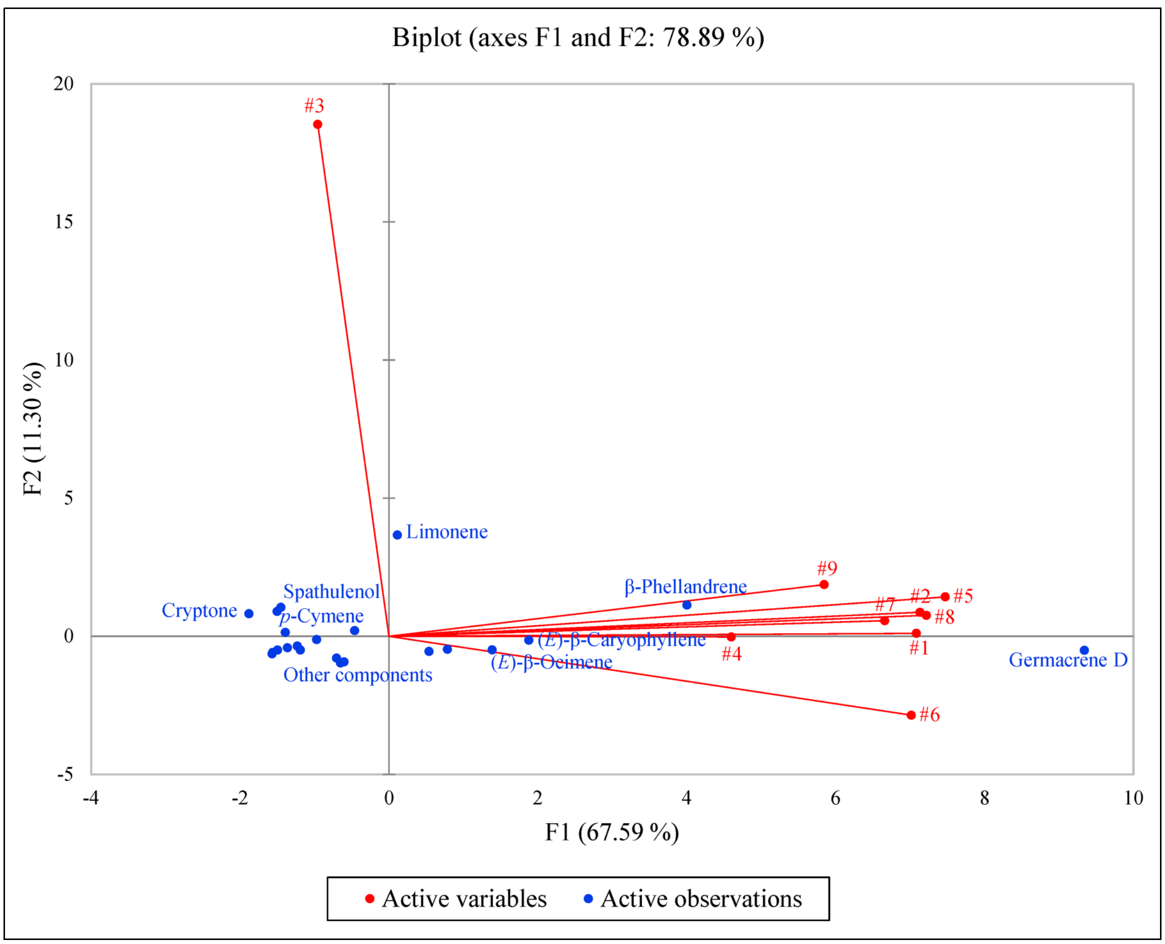

Figure 4. Principal component analysis of the leaf essential oils of *Balsamorhiza sagittata* in this study.

Table 2. Comparison of Percentages of Key Essential Oil Components in Members of the Heliantheae Tribe, Engelmanniinae subtribe of the Asteraceae.

Compound	*Borrichia frutescens*[7]	*Silphium perfoliatum*[8]	*Silphium integrifolium*[9]	*Silphium integrifolium*[10]	*Silphium trifoliatum*[9]
p-Cymene	0.0-0.3	traces	trace-0.2	trace	nd
Limonene	4.7-10.7	traces	1.4-2.4	1.8	traces
β-Phellandrene	9.0-21.0	nd	nd	0.3	nd
Cryptone	nd	nd	nd	nd	nd
(*E*)-β-Caryophyllene	0.2-1.3	2.8-4.0	2.8-4.8	2.5	6.7-14.8
Germacrene D	0.7-18.0	6.4-24.3	18.7-28.4	3.0	8.3-16.1
Spathulenol	nd	3.9-4.6	2.1-3.2	nd	2.7-4.9

Abbreviation: nd, not detected.

Table 3. Enantiomeric Distribution of Chiral Terpenoid Components (Percentage of Each Enantiomer) in *Balsamorhiza sagittata* Essential Oils.

			Sample number								
Compound	RI_{db}	RI_{calc}	1	2	3	4	5	6	7	8	9
(−)-α-Pinene	976	977	55.4	—	52.4	44.5	14.7	26.2	70.1	68.7	59.7
(+)-α-Pinene	982	983	44.6	—	47.6	55.5	85.3	73.8	29.9	31.3	40.3
(−)-Camphene	998	999	100.0	—	100.0	100.0	100.0	100.0	100.0	100.0	—
(+)-Camphene	1005	—	0.0	—	0.0	0.0	0.0	0.0	0.0	0.0	—
(+)-Sabinene	1021	1022	84.1	88.3	74.6	90.2	85.5	85.4	85.7	83.9	75.9
(−)-Sabinene	1030	1031	15.9	11.7	25.4	9.8	14.5	14.6	14.3	16.1	24.1
(+)-β-Pinene	1027	1027	49.2	—	34.0	58.5	63.2	37.6	59.4	34.9	—
(−)-β-Pinene	1031	1031	50.8	—	66.0	41.5	36.8	62.4	40.6	65.1	—
(−)-α-Phellandrene	1050	—	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
(+)-α-Phellandrene	1053	1053	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
(−)-Limonene	1073	1074	91.4	92.3	92.2	75.7	92.7	80.8	77.6	93.6	92.5
(+)-Limonene	1081	1082	8.6	7.7	7.8	24.3	7.3	19.2	22.4	6.4	7.5
(−)-β-Phellandrene	1083	1083	1.1	0.0	0.0	0.2	0.0	0.0	0.0	0.0	0.0
(+)-β-Phellandrene	1089	1089	98.9	100.0	100.0	99.8	100.0	100.0	100.0	100.0	100.0
(+)-Terpinen-4-ol	1297	1292	71.0	65.2	—	70.0	70.1	69.5	69.9	70.6	—
(−)-Terpinen-4-ol	1300	1295	29.0	34.8	—	30.0	29.9	30.5	30.1	29.4	—
(−)-Borneol	1335	1336	100.0	100.0	100.0	100.0	100.0	100.0	—	100.0	—
(+)-Borneol	1340	—	0.0	0.0	0.0	0.0	0.0	0.0	—	0.0	—
(−)-α-Copaene	1375	1375	100.0	100.0	100.0	—	100.0	100.0	100.0	100.0	—
(−)-(*E*)-β-Caryophyllene	1461	1462	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
(+)-Germacrene D	1519	—	0.0	0.0	—	0.0	0.0	0.0	0.0	0.0	0.0
(−)-Germacrene D	1522	1523	100.0	100.0	—	100.0	100.0	100.0	100.0	100.0	100.0
(−)-δ-Cadinene	1563	—	0.0	0.0	—	0.0	0.0	0.0	0.0	0.0	0.0
(+)-δ-Cadinene	1576	1577	100.0	100.0	—	100.0	100.0	100.0	100.0	100.0	100.0
(+)-(*E*)-Nerolidol	1677	1677	—	100.0	100.0	—	—	100.0	100.0	—	100.0
(−)-(*E*)-Nerolidol	1680	—	—	0.0	0.0	—	—	0.0	0.0	—	0.0

Abbreviations: RI_{db}, retention index from our in-house database; RI_{calc}, calculated retention index based on a homologous series of *n*-alkanes on a Restek B-Dex 325 capillary column; —, compound not detected.

(−)-α-pinene, 100% ee (−)-α-phellandrene and (−)-β-phellandrene, but nearly racemic β-pinene and sabinene.[11] Likewise, *Gnoxys laurifolia* (Kunth.) Cass. had 100% (−)-α-phellandrene and (−)-β-phellandrene, nearly racemic sabinene, but 100% (−)-β-pinene.[12] *Gnoxys rugulosa* Muschl., on the other hand, showed 100% ee (+)-β-pinene and (+)-β-phellandrene and nearly racemic terpinen-4-ol and α-terpineol.[13] In contrast, *Gnoxys buxifolia* (Kunth.) Cass. had 100% ee for (−)-α-pinene, (−)-β-pinene, (+)-β-phellandrene, and (−)-terpinen-4-ol.[14] In a comparison with enantiomeric distributions in sagebrush (*A. tridentata* Nutt.),[15] there are some similarities with *B. sagittata*. Both α- and β-pinene were variable in distribution, (−)-limonene was dominant is sagebrush as was (−)-borneol. Apparently only (−)-(*E*)-β caryophyllene is seen in higher plants, but (+)-(*E*)-β caryophyllene has been found in some liverworts.[16] Camphene did show variation in enantiomeric distribution in sagebrush, depending on the subspecies.

Conclusions

This work is the first presentation of the leaf essential oil composition of *B. sagittata*. Nine samples were collected from various locations in southwestern Idaho. Ignoring the composition of the outlier, sample #3, the leaf essential oil of *B. sagittata* can

Table 4. *Balsamorhiza sagittata* Collection and Hydrodistillation Details.

Sample number	Date	Location	Mass leaves (g)	Yield essential oil (mg)
1	13 May 2022	43°30′57″ N, 115°54′33″ W 1301 m asl	38.45	367.6
2	16 May 2022	43°35′35″ N, 115°59′48″ W 960 m asl	104.46	72.0
3	16 May 2022	43°35′35″ N, 115°59′48″ W 960 m asl	63.62	68.0
4	25 May 2023	43°30′25″ N, 115°55′35″ W 1460 m asl	99.67	796.1
5	25 May 2023	43°30′25″ N, 115°55′35″ W 1460 m asl	71.66	301.4
6	25 May 2023	43°30′25″ N, 115°55′35″ W 1460 m asl	59.01	266.0
7	25 May 2023	43°32′33″ N, 115°48′14″ W 1143 m asl	83.52	478.3
8	25 May 2023	43°32′33″ N, 115°48′14″ W 1143 m asl	73.51	290.2
9	25 May 2023	43°32′33″ N, 115°48′14″ W 1143 m asl	84.23	772.4

be summarized as containing (−)-germacrene D (10.8-34.5%), (+)-β-phellandrene (6.4-19.4%), (−)-(*E*)-β-caryophyllene (1.4-15.0%), and (*E*)-β-ocimene (3.1-8.4%) as major components. Furthermore, there was modest variation in composition or with respect to geographical location or elevation. A limitation of this study is the relatively narrow geographical range of the collections and likely does not adequately describe the essential oil composition of the species. It would be interesting to compare *B. sagittata* essential oils from geographical locations outside of Idaho to more definitively describe the volatile phytochemistry of this plant species. Likewise, comparison with essential oils of other *Balsamorhiza* species would be appealing.

Materials and Methods

Plant Material

Leaves of nine individuals of *B. sagittata* were collected during the flowering stage from the hills near Boise, Idaho. Plants were identified in the field by K. Swor by consulting the botanical descriptions,[17,18] and by comparison with samples from the New York Botanical Garden Virtual Herbarium (https://sweetgum.nybg.org/science/vh/, accessed on 28 May 2022). A voucher specimen (KS-Bs-5316) has been deposited in the University of Alabama in Huntsville herbarium. The aerial parts were fresh frozen and stored at −20°C until distilled.

Essential Oil Isolation

Each sample of plant material was hydrodistilled using a Likens-Nickerson apparatus for 3 h to give the essential oils (Table 4). Yields were calculated as 100% × essential oil mass/leaf mass.

GC-FID, GC/MS, and Chiral GC/MS Analyses

The essential oils of *B. sagittata* were analyzed by gas chromatography coupled with flame ionization detection (GC-FID), gas chromatography-mass spectrometry (GC/MS), and chiral GC/MS as previously described.[19] The essential oil components were identified by comparison of the mass spectral (MS) fragmentation patterns (>80% similarity match) and by comparison of retention index (RI) values (within 5 RI units) available in the Adams,[20] FFNSC 3,[21] NIST20,[22] and our own in-house database.[23] The identification of enantiomers was determined by comparison of retention indices with authentic samples obtained from Sigma-Aldrich (Milwaukee, WI, USA).

Multivariate Analysis

The agglomerative hierarchical cluster (AHC) analysis was carried out to assess the similarity of the essential oil samples using the distribution of the main essential oil constituents (Table 1). The nine *B. sagittata* samples were treated as operational taxonomic units (OTUs). Pearson correlation was selected as a measure of similarity, and the unweighted pair group method with arithmetic average (UPGMA) was used for cluster definition. PCA was carried out to verify the previous AHC analysis using the 20 main essential oil components (Table 1). The AHC and PCA analyses were performed using XLSTAT v. 018.1.1.62926 (Addinsoft, Paris, France).

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Data Availability Statement

The data presented in this study are available upon reasonable request from the corresponding author.

Declaration of Conflicting Interests

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Ethical Approval

Ethical approval is not applicable for this article.

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Statement of Human and Animal Rights

This article does not contain any studies with human or animal subjects.

Statement of Informed Consent

There are no human subjects in this article and informed consent is not applicable.

Supplemental Material

Supplemental material for this article is available online.

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