RESEARCH NOTE

Essential Oil of *Chrysothamnus pulchellus* (Gray) Greene ssp. *pulchellus*¹

Mario R. Tellez, Rick E. Estell,* Ed L. Fredrickson and Kris M. Havstad USDA-ARS, Jornada Experimental Range, Box 30003, MSC 3JER, Las Cruces, NM 88003

Abstract

The essential oil profile of *Chrysothamnus pulchellus* was examined. Samples were collected from the Jornada Experimental Range in south central New Mexico and GC/MS and retention indices were used to identify 48 oil components. Sesquicineole (22.7%), β -phellandrene (14.9%), (Z)- β -ocimene (9.4%), β -pinene (8.8%), (E)- β -ocimene (6.4%), β -caryophyllene (3.3%), and δ -cadinene (2.8%) were the major components of the steam distilled oil.

Key Word Index

 $\textit{Chrysothamnus pulchellus}, \ Asteraceae, \ essential \ oil \ composition, \ sesquicine ole, \\ \beta\text{-phellandrene}.$

Introduction

Chrysothamnus pulchellus (southwestern rabbitbrush) is a shrub (typical height of 0.3-0.5 M) found in deep sandy soils of the northern region of the Chihuahuan Desert, mesas and slopes in the Sagebrush Desert, the Desert Grassland, and the Great Plains Grassland (1). Geranyl derivatives of p-coumaric acid as well as several flavonoids and labdane have been isolated from and identified in *C. pulchellus* by Jakupovic et al. (2). Constituents of cyclohexane extracts of several other Chrysothamnus species have been published by Hegerhorst et al. (3). To our knowledge, the oil composition of *C. pulchellus* has not been previously reported.

Experimental

Samples of *Chrysothamnus pulchellus* subsp. *pulchellus* (as determined using herbarium specimens identified by L.C. Anderson, 1976, Jornada Experimental Station) were collected from 5 plants growing in deep sandy soils on the Jornada Experimental Range (JER). Fresh samples of terminal branch ends containing some of previous seasons' growth were collected randomly from several locations within each plant, composite samples were made, stored in labeled plastic bags and immediately frozen using dry ice. The plant material was stored at -20° C until steam distillations were performed. A voucher specimen of *C. pulchellus* (Gray) Greene subsp. *pulchellus* was placed in the JER herbarium located in Las Cruces, NM.

Received: January 1997

¹Mention of a trade name, proprietary product, or vendor does not constitute a warranty of the product by the USDA to imply its approval to the exclusion of other products or vendors that may also be suitable.

Address for correspondence

202 Tellez et al.

Table I. Constituents of Chrysothamnus pulchellus oil

Compound	Rì	RT	% RA
nonane	900	264	0.1
tricyclene	927	298	0.1
α-thujene	932	304	0.3
α-pinene	939	315	1.4
camphene	954	337	0.5
β-pinene	980	383	8.8
myrcene	991	405	2.6
α-phellandrene	1004	431	1.3
δ-3-carene	1011	443	0.1
α-terpinene	1017	455	1.9
p-cymene	1025	469	0.2
β-phellandrene	1031	480	14.0
(Z)-β-ocimene	1039	496	9.4
(E)-β-ocimene	1050	518	6.4
γ-terpinene	1061	542	2.2
terpinolene	1089	609	0.7
linalool	1099	635	1.3
terpinen-4-ol	1178	825	0.3
α-terpineol	1190	859	t.5
α-terphieor β-maaliene (or β-patchoulene)	1380	1350	0.2
β-hourbonene	1385	1363	t
socomene	1386	1367	0.1
solongifolene	1389	1376	0.1
solongilolene 1,7-di-epi-α-cedrene	1389	1403	U.2
•	1404	1416	0.1
ongifolene (or β-isocomene) α-cedrene	1411	1431	
α-cearene cis-α-bergamotene	1411	1441	t 0.1
· ·	1415	1450	3.3
β-caryophyllene			
trans-α-bergamotene	1437	1493	0.7
α-humulene	1454	1535	0.7
(E)-β-farnesene	1459	1548	t
allo-aromadendrene (or seychellene)	1461	1553	0.9
9-epi-(β)-caryophyllene (or β-acoradiene)	1467	1569	0.1
β-cadinene	1474	1587	0.2
γ-muurolene	1477	1594	0.5
y-curcumene	1481	1603	1.1
Ar-curcumene	1483	1610	t
β-selinene	1485	1616	1.2
α-zingiberene	1495	1642	1.0
epi-zonarene	1498	1649	0.3
x-muurolene	1499	1653	0.6
β-bisabolene	1509	1675	0.6
sesquicineole	1514	1686	22.7
δ-cadinene	1524	1709	2.8
cadina-1,4-diene	1532	1729	0.3
α-cadinene	1538	1743	0.6
selina-3,7(11)-diene	1542	1752	0.4
germacrene B	1557	1789	0.1

RI= retention index as determined on a DB-5 column using the homologous series of n-hydrocarbons; RT= retention time on a DB-5 column in seconds; RA= relative area (peak area relative to total peak area); t = trace (<0.05%)

Steam distillations were conducted in a Nikerson-Likens type apparatus (4,5). A 1000 mL flask, 250 mL of water, and 17.5 g of plant material were used. A large distilling flask was used to prevent the excessive foam that started to form after 2-3 h from entering the separation assembly of the Nickerson-Likens apparatus (presence of saponins in this plant is currently being investigated at this laboratory). The distillate was continuously extracted with pentane into a 10 mL pear shaped flask maintained at 58°-62°C. The total volume of pentane used was 12 mL and distillation time was 6 h. The 2 pentane fractions and an additional 8 mL of pentane used to rinse the apparatus, were combined and dried over anhydrous magnesium sulfate, filtered, and the solvent removed under reduced pressure using a rotary evaporator. A clear yellow oil was obtained in a yield of 16.4 mg (0.09% of fresh weight). The oil was redissolved in pentane (1 mg/mL) with octane and eicosane added to a final concentration of 100 µg/mL before injection.

Analyses were performed by gas chromatography coupled to mass spectrometry, using a Finnigan ion trap mass spectrometer (EI, 70 eV) in conjunction with a Varian model 3400 gas chromatograph equipped with a DB-5 column (30 m x 0.25 mm fused silica capillary column, film thickness 0.25 μ m) using helium as carrier gas (1 mL/min), 1 μ L injection size and a programmed (injector temperature: 220°C, transfer line temperature: 240°C, initial column temperature: 60°C, final column temperature 240°C, 3°C/min) temperature run (6). Identification of oil components was performed by a comparison of mass spectra with literature data (6,7), and by a comparison of their relative retention times with those of authentic compounds or by comparison of their retention indices with the literature (6). The relative amounts (RA) of individual components of the oil are expressed as percent peak area relative to total peak area.

Results and Discussion

Table I shows the identity, retention index, retention time and the percent composition of the oil of *C. pulchellus*. Forty-eight compounds were identified in the oil of southwestern rabbitbrush, accounting for over 90% of the composition of the oil, with no unidentified compound accounting for more than 1.2% of the total area. Among the identified compounds were 14 monoterpene hydrocarbons (50.8%), 27 sesquiterpene hydrocarbons (16.2%), 3 monoterpene alcohols (1.6%), and 1 oxygenated sesquiterpene (22.7%). The dominant compound, sesquicineole, is an unusual bicyclic bisabolene which has been used in the perfume and flavor industries (8,9). An extensive literature search revealed sesquicineole to be a known constituent of only four plant species studied: *Senecio subrubriflorus* O. Hoffm. (10), *Anthemis alpestris* (11), *Aydendron barbeyana* Mez. (12), and *Boronia megastigma* Nees. (13); and to have been the subject of two syntheses (8,14).

Hegerhorst et al. (3) examined the volatile constituents in ten species/subspecies of *Chrysothamnus* (but not *C. pulchellus*). These workers listed 60 compounds present in cyclohexane extracts of one or more species/subspecies, of which 42 were identified. Of the 34 compounds eluting before eicosane in their study that were present in one or more *Chrysothamnus* species/subspecies (3), we could determine presence or absence of 20 of these constituents in *C. pulchellus* (Hegerhorst et al. did not conclusively identify the remaining 14 compounds). Of these 20 compounds, 11 (α -pinene, β -pinene, myrcene, α -phellandrene, β -phellandrene, β -carene, β -patchoulene, β -caryophyllene, α -humulene, γ -muurolene and δ -cadinene) were present in *C. pulchellus*. When we compared the presence or absence of these 20 compounds in *C. pulchellus* and the species examined by Hegerhorst et al. (3), as many as 17 (*C. nauseosus* (Pall.) Britt. subsp. *bololeucus*) or as few as 5 (*C. viscidiflorus* (Hook) Nutt., *C. lintfolius* (H&C) Greene) compounds were in common with *C. pulchellus* in other *Chrysothamnus* species (i.e., commonality of profile in terms of both constituents and compounds not present).

In conclusion, *C. pulchellus* has a low oil content and a fairly typical profile of constituents. The exception is the dominance of sesquicineole in the profile, making the oil of *C. pulchellus* of possible interest to the perfume and flavor industries.

204 TELLEZ ET AL.

10-ene-3, 7-oxide. Tetrahedron, 44, 5109-5112 (1988).

Acknowledgment

The authors gratefully acknowledge the assistance of Dr. R. P. Gibbens in the identification and collection of plant material, and Dr. R. P. Adams for confirming the identification of sesquicineole.

References

1. L. Benson and R. A. Darrow, Trees and Shrubs of the Southwestern Deserts. 3rd Ed., The University of Arizona Press, Tucson, Arizona USA (1981). 2. J. Jakupovic, M. Grenz, M. Miski, F. Bohlmann and P. E. Boldt, Geranyl derivatives of p-coumaric acid from

- Chrysothamnus pulchellus, Phytochemistry, 29, 617-620 (1990). 3. D. F. Hegerhorst, D. J. Weber, E. D. McArthur and A. J. Khan, Chemical analysis and comparison of subspecies of Chrysothamnus nauseosus and other related species. Biochem. Syst. Ecol., 15, 201-208 (1987). 4. G. B. Nickerson and S. T. Likens, Gas chromatographic evidence for the occurrence of hop oil components in
- beer. J. Chromatog., 21, 1-5 (1966). 5. H. Maarse and R. E. Kepner, Changes in composition of volatile terpenes in Douglas fir needles during
- maturation. J. Agr. Food Chem., 6, 1095-1101 (1970). 6. R. P. Adams, Identification of Essential Oil Components by Gas Chromatography/Mass Spectroscopy, Allured
- Publ. Corp., Carol Stream, IL USA (1995). 7. Finnigan Standard Format NIST MS Library, National Institute of Science and Technology, Maryland (1990). 8. P. Weyerstahl and K. Krohn, Structure-odor correlation-IX, from 1,8-cineol - change of odor with structure.
- Tetrahedron, 46, 3503-3514 (1990). 9. Dictionary of Organic Compounds, 5th edition, Ninth Supplement. J. Buckingham, executive editor, Chapman & Hall, Cambridge, UK (1991).
- 10. F. Bohlmann and C. Zdero, Sandaracopimarene derivatives from Senecio subrubriflorus, Phytochemistry, 21, 1697-1700 (1982). 11. J. de Pascual-Teresa, E. Caballero, S. G. Pastor and M. C. Caballero, Studia Chemica, 11, 475 (1986).
- 12. P. Weyerstahl, H. Marschall-Weyerstahl and C. Christiansen, Constituents of ayou essential oil (Aydendron
- berbeyana Mez.). Flav. Fragr. J., 4, 93-98 (1989).
- 13. N. W. Davies and R. C. Menary, Volatile constituents of Boronia megastigma flowers, Perfum. Flavor., 8, 3-8 (1984). 14. J. de Pascual-Teresa, M. L. Hernandez, J. R. Moran, C. Caballero, J. Anaya and V. Alcazar, Synthesis of bisabol-