The Essential Oil Composition of *Psorothamnus scoparius* (A. Gray) Rydb.

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Abstract

Psorothamnus scoparius (broom dalea) was collected from the Jornada Experimental Range in south central New Mexico. Current year's growth was collected from 10 plants, all found within an approximate 50 m radius of the GPS coordinates N32°41.286' and W106°46.922' during June, 2000. Composite samples were steam distilled, and the essential oil was analyzed using GC with FID and GC/MS. Mass spectra and retention indices were used to identify 64 compounds. Retention indices and EI mass spectra are also provided for 15 unknowns. γ-Terpinene (22.3%), p-cymene (14.0%) and α-pinene (9.0%) were the major constituents of the oil.

Key Word Index

Psorothamnus scoparius, Fabacae, broom dalea, essential oil composition, p-cymene, γ-terpinene

Introduction

Psorothamnus scoparius (A. Gray) Rydb.,(syn. *Dalea scoparia* A. Gray; *Parosela scoparia* (A. Gray) A. Heller) commonly known as broom dalea, is a woody dicot shrub of the family Fabaceae found in sandy soils of the Chihuahuan Desert. Natural products chemistry of *P.* species has received little attention. Raffauf (1) included *P. fremontii* (referred to therein as *Dalea fremontii*) in his survey of plant alkaloids, as one of less than 22% of legumes analyzed which tested positive for alkaloids. Raffauf's assays were qualitative, based on colorimetric changes following spotting with Dragendorff's reagent. Two antimutagenic isoflavones isolated from *P. fremontii* (2), a protein kinase C inhibitor from *P. junceus* (3), and coumarin, dalrubone, and their methoxy- derivatives (4) are the only specific compounds we found in the literature pertaining to *Psorothamnus*. Although the plant releases a pleasant aroma, making it popular among southwestern gardeners, we found no descriptions of the volatile oil composition of any plant within the *Psorothamnus* genus. No obvious physical barriers, such as thorns, are present in *P. scoparius*, yet it is unpalatable to livestock. Previous studies with other plant species have demonstrated that leaf surface volatiles may deter herbivory (5-7). In this report, we examine the oil present in *P. scoparius*.

Experimental

Plant material was collected from the Jornada Experimental Range in southern New Mexico, at an altitude of 1,344 m above sea level. Ten plants were randomly selected from within an approximate 50 m radius of the GPS coordinates N32°41.286' and W106°46.922'. Coordinates were determined using a Garmin GPS12 personal navigator. Samples consisted of 10 15-cm leaders of current year's growth from each of 10 plants. These samples were placed on dry ice immediately after clipping, and were stored at 20°C until steam distillations and dry matter analyses were performed. A voucher specimen, identified as *P. scoparius* (Gray) Rydb, was placed in the Jornada Experimental Range Herbarium located in Las Cruces, NM.

Collected tissue was ground in liquid nitrogen and 20 g were steam distilled for 6 h as described by Tellez et. al. (8), using a 500 mL flask and 250 mL water. The resulting oil was dissolved in 100% ethanol. Both batches were analyzed by GC/MS using a Varian model 3400 GC with a DB-5 column (30 m x 0.25 mm fused silica capillary column, film thickness 0.25 μm) coupled to a Finnigan ion trap mass spectrometer (EI, 70 eV). Helium at 1 ml/min was used as a carrier gas, and injector and transfer line temperatures were set at 220°C and 260°C, respectively. The initial column temperature was 60°C,

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Received: August 2001
Revised: October 2001
Accepted: October 2001

1041-2905/03/0002-0108$6.00/0—

Table I. Compounds identified in the oil of *Psorothamnus scoparius*

RI	Component Identification	Percent of Total Peak Area (FID)	Percent of Total Peak Area (TIC)
927	tricyclene	2.0	2.8
940	α-pinene	9.0	11.9
954	camphene	3.7	4.5
981	β-pinene	0.3	0.3
987	6-methyl-5-hepten-2-one	t	0.1
992	myrcene	2.7	2.5
1006	α-phellandrene	0.1	0.5
1019	α-terpinene	0.1	1.1
1027	p-cymene	14.0	9.3
1032	limonene	2.6	2.3
1041	(Z)-β-ocimene	0.9	0.9
1045	phenylacetaldehyde	t	t
1052	(E)-β-ocimene	0.7	0.7
1062	γ-terpinene	22.3	23.4
1089	terpinolene	0.2	0.5
1114	α-fenchol	t	0.1
1121	*cis*-p-menth-2-en-1-ol	0.1	t
1127	α-campholenal	t	0.5
1141	*trans*-pinocarveol	t	0.1
1150	camphene hydrate	t	0.2
1167	borneol	0.1	0.2
1171	ethyl benzoate	t	t
1178	terpinen-4-ol	t	1.5
1183	4-methyl-acetophenone	t	t
1184	p-cymen-8-ol	t	0.1
1190	α-terpineol	0.2	0.3
1238	ascaridole	0.1	0.2
1271	geranial	t	0.0
1287	p-cymen-7-ol	t	0.1
1300	carvacrol	t	t
1373	α-ylangene	1.4	1.4
1377	α-copaene	0.5	0.5
1384	(E)-β-damascenone	0.1	0.1
1384	β-bourbonene	t	0.1
1396	(Z)-jasmone	t	0.1
1403	italicene	t	t
1410	α-gurjunene	0.2	0.2
1412	α-cedrene	t	t
1420	β-caryophyllene	0.2	1.4
1437	*trans*-α-bergamotene	2.1	0.8
1440	aromadendrene	2.1	1.3
1455	α-humulene	1.0	1.1
1462	allo-aromadendrene	0.9	0.7
1473	γ-gurjunene	0.1	0.1
1476	γ-muurolene	1.7	1.8
1486	β-selinene	0.7	0.7
1491	*cis*-β-guaiene	0.6	0.4
1498	epi-zonarene	t	t
1512	β-curcumene	1.4	t
1514	γ-cadinene	1.4	1.6
1524	δ-cadinene	2.8	3.5
1533	cadina-1,4-diene[1]	t	0.2
1538	α-cadinene	0.3	0.4
1543	α-calacorene	1.1	1.1
1564	β-calacorene	0.1	0.1
1578	spathulenol	0.1	0.1
1584	globulol	0.5	0.6
1608	humulene epoxide II	0.1	0.1
1628	1-epi-cubenol	0.7	0.3
1639	epi-α-cadinol	0.3	0.7
1646	α-muurolol	0.1	0.2
1650	β-eudesmol	0.1	0.2
1672	β-bisabolol	t	t
1674	cadalene	0.1	0.4

[1][1] 2,3-dioxabicyclo(2.2.2)oct-5-ene, 1-methyl-4-(1-methylethyl)-, (1-α, 4-β); t = trace (<0.1%)

Table II. Mass Spectra and Retention Indices of unknowns from the essential oil of *Psorothamnus scoparius*

RI	Percent of Total Peak Area (FID)	Percent of Total Peak Area (TIC)	MS of Unknowns at 70 eV
1120	0.1	0.1	134(39), 119(31), 105(23), 91(100), 79(36), 65(22), 41(96)
1258	1.1	0.1	152(3), 139(2), 125(20), 107(7), 95(18), 79(19), 69(42), 55(30), 41(100)
1390	0.2	0.1	204(14), 189(20), 161(42), 147(18), 133(37), 119(49), 105(65), 91(81), 79(49), 67(24), 55(15), 43(25), 41(100)
1425	0.2	0.2	204(1), 189(86), 161(45), 148(16), 133(59), 119(26), 105(44), 91(85), 79(84), 67(100), 55(34), 41(96)
1450	0.1	0.1	204(17), 189(22), 175(8), 161(31), 147(28), 133(33), 119(48), 105(100), 91(72), 79(55), 67(24), 55(28), 41(91)
1458	0.1	0.2	204(21), 189(24), 161(45), 147(20), 133(51), 119(25), 105(77), 91(71), 79(60), 67(43), 53(24), 41(100)
1481	3.1	3.3	222(1), 204(10), 161(40), 133(15), 119(32), 105(100), 94(34), 93(48), 92(17), 91(50), 79(45), 67(13), 55(13), 41(73)
1494	1.5	1.7	204(2), 189(21), 161(62), 149(13), 133(32), 119(55), 105(82), 91(77), 79(73), 67(28), 55(25), 41(100)
1507	1.3	2.1	204(14), 161(45), 119(41), 105(100), 93(31), 91(42), 79(33), 57(20), 43(36), 41(87)
1568	0.1	0.2	204(5), 189(4), 175(3), 161(11), 147(8), 133(8), 122(11), 111(15), 93(16), 79(22), 67(20), 55(22), 44(100), 41(64)
1592	0.4	0.6	222(2), 204(17), 189(8), 161(47), 149(8), 133(13), 119(46), 105(57), 93(46), 79(60), 67(27), 55(24), 43(100), 41(91)
1611	0.2	0.2	204(10), 179(70), 161(37), 119(25), 105(34), 95(28), 79(39), 67(26), 55(32), 43(79), 41(100)
1622	0.2	0.8	204(10), 200(10), 185(20), 161(25), 149(65), 143(11), 135(31), 121(25), 107(56), 91(67), 79(73), 67(44), 59(99), 41(100)
1631	0.6	1.6	204(4), 189(10), 179(35), 161(78), 133(16), 119(65), 105(71), 91(49), 81(41), 67(20), 55(30), 41(100)
1650	0.5	0.6	204(21), 189(11), 161(46), 149(48), 133(18), 119(34), 105(61), 91(50), 79(63), 67(25), 55(25), 43(90), 41(100)

and a linear temperature increase of 3°C/min was programmed into each run. A series of 40-, 400-, and 600-ng injections was used to validate retention times for both low- and high-concentration components. Compounds were identified by comparing mass spectra and retention indices (9) with literature data (10, 11) and with our own MS library, which was developed using authentic standards.

To validate the peak area percentages revealed on the total ion chromatogram, the oil was also analyzed using a Shimadzu GC8APF equipped with a flame ionization detector and fitted for use with capillary columns. A split/splitless injector was used, and the column and temperature gradient were identical to those described above for the GC/MS. The injector temperature was 250°C. Dry matter percent was determined by drying triplicate, 2 g samples of ground tissue at 100°C for 24 h.

Results and Discussion

Dry matter accounted for 55% of the tissue fresh weight. The volatile fraction accounted for 0.03% of the dry matter. Table I shows the identity, retention indices, and percent composition of the oil components identified by GC/MS in 400-ng injections of *P. scoparius* oil. Larger injections were required to detect the same peaks by FID. Peak area percentages varied slightly between the two types of chromatogram. Both values are shown. Identified peaks comprising less than 0.1% of the total peak area are marked "t" (trace). Sixty-five compounds were positively identified, accounting for 80% of the composition of the oil. Unknowns accounted for the remainder of the total detected peak area. Unknowns comprising at least 0.1% of the oil detected by FID are listed in Table II, along with their EI mass spectra. Since soft ionization was not available to verify the molecular ions, no molecular ions have been designated. Mass spectra of the unknowns bore no resemblance to the spectra reported for the previously isolated compounds (2-4), nor would any of these compounds be expected to partition with the oil fraction.

γ-Terpinene was by far the most abundant compound in the volatile fraction, constituting over 22% of the oil. γ-Terpinene is a biosynthetic precursor to cavacrol (12), which was also present, albeit at trace levels. Cavacrol is antimicrobial (12), as is thymol, which is synthesized from p-cymene. The large stores of γ-terpinene and p-cymene seen in *P. scoparius* could perhaps serve as a biochemical reserve, enabling the plant to rapidly respond response to microbial attack or other stress. The other major volatile, α-pinene, has been demonstrated to deter feeding by sheep when applied to alfalfa pellets (13).

In summary, γ-terpinene dominated the oil profile of *P. scoparius*. α-Pinene and p-cymene were also present in significant proportions. Roughly 10% of the total peak area within the FID chromatograms of the oil profile was accounted for by 15 compounds whose mass spectra did not correlate well with spectra contained in Adams (11) or NIST (10) libraries, and may belong to novel compounds. The remainder of the profile was composed of very small peaks for which clear mass spectra could not be obtained due to the low signal to noise ratio.

Acknowledgements

The authors extend thanks and appreciation to Yuan Feng Wang for assistance with GC/MS analysis.

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