

Comparison of Yellow Starthistle Volatiles from Different Plant Parts

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Volatile components of yellow starthistle (*Centaurea solstitialis* L.) flowerhead buds, flowers, and leaves and stems were identified by gas chromatography-mass spectrometry. Sesquiterpenes (predominantly germacrene D) constituted the major part of the volatiles. Four 1-alkenes and a variety of C₁₃ polyacetylenes were found in flowers and flowerhead buds but not in leaves and stems. Of 62 compounds identified, 52 were not previously reported in yellow starthistle.

Yellow starthistle (*Centaurea solstitialis* L.) is a naturalized weed of rangelands and croplands in the western United States (Maddox et al., 1985). In California alone, the infestation area of yellow starthistle exceeds 3 million ha. In 1985, the area affected was about 6.5 times as great as in 1958 (Maddox and Mayfield, 1985). It is a weed in hay and grain crops, orchards, vineyards, and pastures and is the cause of a potentially fatal neurological disease in horses (Cordy, 1978).

Efforts to control yellow starthistle involve biological control through the introduction of natural enemies of the plant from its areas of origin in Europe and the Middle East. The flowerhead weevil *Bangasternus orientalis* Capiomont was introduced from Greece in 1985 (Maddox et al., 1986) and is presently established in California, Idaho, Oregon, and Washington. *B. orientalis* females oviposit on the stem or the small leaves arising from the stem below the nonflowering flowerhead buds. After hatching, young larvae make their way to the base of the flowerhead bud, enter, and feed on receptable tissue. The fruit fly *Chaetorellia australis* Hering is also now being released. Females oviposit on the inner surface of the involucre bracts on the host flowerhead shortly before anthesis. Hatched larvae burrow into the host flowerhead where they feed on ovules and developing seeds (Turner et al., 1988). *B. orientalis* is strongly host-plant specific to yellow starthistle (Maddox and Sobhian, 1987). *C. australis* shows host specificity for yellow starthistle and *Centaurea cyanus* L. (Turner et al., 1988). These host-finding and specificity behaviors may be mediated by chemical cues from the plants and so it is of interest to determine the composition of volatiles emanating from them.

There are two previous reports of yellow starthistle volatile compounds. Two polyacetylenic compounds, (*E*)-1,11-tridecadiene-3,5,7,9-tetrayne and 12,13-epoxy-10-tridecene-2,4,6,8-tetrayne, were found in roots, and 6,8,10,12-pentadecatetraenal was found in leaves (Bohlmann et al., 1966). Compounds from samples of buds and of leaves and stems were isolated by Tenax trapping and analyzed by GC-MS (Buttery et al., 1986). Sixteen components were identified; the major one was germacrene D. We have isolated the volatile components by a different procedure and have identified many other compounds.

EXPERIMENTAL SECTION

Materials. Yellow starthistle was collected in July 1987 from a ranch four miles east of Napa, CA. Three samples were pre-

pared: flowerhead buds, flowers, leaves and stems. Flowerhead buds in growth stages 1-4 (Maddox, 1981) were snipped from stems and pooled. To obtain the flower samples, a razor cut was made across the base of each flowerhead and the involucre was separated and discarded.

Isolation of Volatiles. Flowerhead buds (608 g) were blended with doubly distilled acetone in a Waring Blendor. The mixture was filtered, and the filter cake was rinsed with acetone before being reblended with fresh solvent. Acetone was removed from the extract by distillation under reduced pressure, and the distilled acetone plus some volatile plant components was collected in a flask chilled by solid carbon dioxide. Distilled water was added to the extract residue, and vacuum steam distillation was conducted for 4 h. Distillation of the previously collected acetone through a packed column at atmospheric pressure removed most of the acetone and left a watery solution of yellow starthistle volatiles. This was added to the steam distillate. The mixture was saturated with salt and extracted three times with freshly distilled ether. The ether solution was dried with anhydrous sodium sulfate and concentrated to 2 mL by distillation of ether. In similar fashion, concentrates of volatiles were obtained from 77 g of flowers and 103 g of leaves and stems.

Gas Chromatography. Chromatographic separations were carried out with Hewlett-Packard 5830 gas chromatographs fitted with flame ionization detectors. DB-1 and DB-Wax 60 m × 0.32 mm fused silica columns (J&W Scientific) were employed. Operating conditions for the DB-1 column were as follows: head pressure 24 psi; temperature program 50-230 °C at 4 °C/min and then 230 °C for 10 min. Operating conditions for the DB-Wax column were the same except head pressure was 21.5 psi. A measured amount of undecane was added to an aliquot of a volatiles concentrate in order to calculate yields. Based on initial weight of plant part sample, about 6 µg/g was added. Correction for detector response was not attempted; yields were calculated by comparison of undecane peak area in chromatograms with peak areas of yellow starthistle compounds.

Component Identification. Identifications were based on mass spectral data obtained with a Finnigan MAT 4500 gas chromatograph-mass spectrometer-data system and were verified by Kovats index comparisons on the DB-1 or DB-Wax column.

RESULTS AND DISCUSSION

Table I lists the volatile compounds identified in yellow starthistle buds, flowers, and leaves and stems, the quantities found, and the retention indices of the compounds on the DB-1 and DB-Wax columns. Each compound listed was identified by its mass spectrum obtained during a GC-MS run. Some of the entries in Table I are for compounds of unknown identity. An unknown was listed as a yellow starthistle component if the same mass

Table I. Volatile Components of Yellow Starthistle

compound	concentration, $\mu\text{g/g}$			Kovats index ^a	
	buds	flowers	leaves/stems	DB-1	DB-Wax
2-pentanone	0.04			600	882
hexanal	0.19		0.48	772	1078
(E)-2-hexenal ^b	0.06	0.05	0.27	823	1215
3-methylpentanol	0.13			825	1327
2-methylbutanoic acid	0.12			829	
(Z)-3-hexenol ^b	0.01	0.09	0.20	834	1378
ethylbenzene		0.05		844	1134
hexanol ^b	0.05		0.05	848	1352
benzaldehyde	0.07	0.05	0.06	926	1516
(E)-2-heptenal	0.04		0.03	927	1320
oct-1-en-3-ol	0.12			961	1449
unknown	0.02			(963)	(1847)
6-methylhept-5-en-2-one	0.07	0.19		966	1332
octanal	0.09	0.06	0.17	979	1284
myrcene ^b		0.21	0.02	981	1160
phenylacetaldehyde	0.59	0.32	0.05	1006	1636
limonene ^b	0.05			1020	1198
unknown	0.05			(1066)	(1642)
2-phenylethanol	0.04		0.09	1081	1902
nonanal	0.05	0.26	0.06	1082	1388
(E)-2-nonenal	0.04			1133	1532
(Z)-3-hexenyl butanoate			0.03	1167	1456
decanal	0.04	0.38	0.02	1184	1495
(E,E)-2,4-nonadienal	0.05			1184	1697
benzothiazole			0.03	1186	1951
unknown	0.06			(1229)	(1536)
unknown	0.05			(1267)	(1755)
1-tridecene	0.33	0.11		1289	1341
unknown	0.12	0.89		(1303)	(1894)
α -cubebene	0.01			1347	1455
β -damascenone	0.03	0.02	0.06	1360	1819
unknown (sesquiterpene)	0.44	1.57		(1364)	(1483)
cyclosativene	0.59	2.11		1367	1478
α -copaene ^b	1.11	2.50	0.35	1373	1489
β -bourbonene	0.05		0.02	1381	1518
β -cubebene	0.10	0.15	0.06	1384	1535
1-tetradecene	0.23	0.06		(1388)	(1443)
unknown	0.50	3.08		(1392)	(1524)
unknown	0.12	0.35	0.07	(1408)	(2001)
caryophyllene ^b	1.07	1.39	0.59	1415	1594
β -copaene	0.04		0.03	1424	1588
(E)- α -bergamotene	0.07	0.20		1431	1580
(E)- β -farnesene ^b	0.13	0.22	0.67	1447	1660
humulene	0.18	0.30	0.10	1448	1665
β -santalene	0.03	0.14	0.03	1453	1649
α -muurolene	0.17	0.14		1469	1685
germacrene D ^b	11.57	5.59	12.72	1473	1704
unknown (sesquiterpene)	0.62	4.40		(1478)	(1696)
β -selinene	0.34	0.67		1480	1717
1-pentadecene ^b	4.90	1.59	0.20	1489	1545
bicyclogermacrene ^b	0.75	0.32	1.52	1489	1733
α -muurolene	0.36	1.87		1492	1720
γ -cadinene	0.08			1504	1755
calamenene	0.04			1507	1827
unknown	0.12	1.56		(1512)	(2108)
δ -cadinene	0.48	0.29	0.03	1513	1752
calacorene (tentative)	0.03	0.03		(1527)	(1926)
(E,Z,E)-1,3,5,11-tridecatetraene-7,9-diyne	0.22	0.23		1597	2310
unknown	0.19	0.07	0.04	1606	2062
(E,E,E)-1,3,5,11-tridecatetraene-7,9-diyne	0.04	0.07		1648	2405
(Z,E,E)-1,3,5,11-tridecatetraene-7,9-diyne	0.01			1650	2310
(Z,Z)-1,8,11-heptadecatriene	0.77	0.29		1655	1805
(Z,E)-1,3,11-tridecatriene-5,7,9-triyne	0.08	0.08		1656	2392
(Z,Z,Z)-1,8,11,14-heptadecatetraene	2.15	0.95	0.02	1657	1867
unknown	1.17	1.66	0.05	(1665)	(2363)
(Z)-1,11-tridecadiene-3,5,7,9-tetrayne	0.02	0.04		1673	2391
(E,E)-1,3,11-tridecatriene-5,7,9-triyne	0.10	0.57		1685	2462
1-heptadecene	0.09	0.04		(1690)	(1746)
unknown	0.06	0.08		(1698)	(1945)
(E)-1,11-tridecadiene-3,5,7,9-tetrayne ^b	0.02	0.12		1710	2484
mint sulfide (tentative)	0.04	0.07	0.03	(1722)	
(Z)-1,3-tridecadiene-5,7,9,11-tetrayne (tentative)	0.03	0.95		(1727)	(2300)
(E,E)-1,3,5-tridecatriene-7,9,11-triyne (tentative)	0.05	0.83		(1732)	
(E)-1,3-tridecadiene-5,7,9,11-tetrayne	0.09	1.56		1758	2277
1-tridecene-3,5,7,9,11-pentayne (tentative)		0.33		(1778)	

^a Experimental Kovats indices were within a few units of the listed reference indices. Indices in parentheses are experimental values observed for compounds that were not available for determination of reference index values. ^b Compound previously identified in yellow starthistle.

spectrum was found in both the DB-1 and DB-Wax GC-MS runs. Partial or tentative identifications were made

when the mass spectrum of the compound was strongly distinctive, but no reference samples were available. The

Table II. Mass Spectral Data from Major Unknowns and Polyacetylenes (Obtained during GC-MS Run, 70 eV)

compound	M ⁺	mass spectral data, <i>m/z</i> (relative intensity)
unknown (KI 1303, DB-1)		144 (1), 103 (31), 101 (14), 85 (100), 84 (25), 57 (49), 56 (16), 55 (18), 43 (20), 41 (28)
unknown (KI 1364, DB-1)	204	204 (23), 189 (13), 161 (52), 149 (18), 133 (28), 119 (30), 106 (44), 105 (100), 94 (41), 93 (61)
unknown (KI 1392, DB-1)	204	204 (2), 138 (10), 137 (100), 136 (15), 121 (26), 105 (9), 95 (21), 91 (11), 79 (7), 77 (7)
unknown (KI 1478, DB-1)	204	204 (4), 123 (100), 121 (31), 107 (19), 95 (21), 94 (53), 93 (34), 91 (19), 81 (18), 79 (22)
unknown (KI 1512, DB-1)		171 (9), 103 (73), 95 (30), 85 (100), 84 (21), 69 (53), 68 (34), 57 (68), 55 (23), 41 (48)
(<i>E,Z,E</i>)-1,3,5,11-tridecatetraene-7,9-diyne	168	168 (36), 167 (21), 166 (14), 165 (43), 153 (35), 152 (100), 141 (13), 139 (19), 128 (14), 115 (33)
(<i>E,E,E</i>)-1,3,5,11-tridecatetraene-7,9-diyne	168	168 (58), 167 (15), 166 (15), 165 (44), 153 (28), 152 (100), 141 (14), 139 (19), 128 (14), 115 (35)
(<i>Z,E,E</i>)-1,3,5,11-tridecatetraene-7,9-diyne	168	168 (51), 167 (16), 166 (10), 165 (34), 153 (30), 152 (100), 141 (16), 139 (16), 128 (16), 115 (38)
(<i>Z,E</i>)-1,3,11-tridecatriene-5,7,9-triyne	166	166 (64), 165 (100), 164 (26), 163 (13), 150 (9), 139 (26), 138 (14), 126 (21), 115 (10), 110 (9)
unknown (KI 1665, DB-1)	236	236 (2), 220 (1), 202 (8), 187 (4), 177 (16), 159 (37), 109 (100), 91 (75), 81 (71), 79 (66)
(<i>Z</i>)-1,11-tridecadiene-3,5,7,9-tetrayne	164	165 (18), 164 (100), 163 (69), 149 (2), 139 (3), 138 (38), 137 (11), 110 (30), 98 (17), 86 (11)
(<i>E,E</i>)-1,3,11-tridecatriene-5,7,9-triyne	166	166 (60), 165 (100), 164 (29), 163 (17), 150 (5), 139 (29), 138 (8), 126 (11), 115 (11), 110 (7)
(<i>E</i>)-1,11-tridecadiene-3,5,7,9-tetrayne	164	165 (13), 164 (100), 163 (71), 146 (4), 139 (4), 138 (34), 137 (14), 122 (13), 110 (26), 98 (18)
(<i>Z</i>)-1,3-tridecadiene-5,7,9,11-tetrayne	164	165 (12), 164 (90), 163 (100), 139 (9), 138 (78), 137 (24), 122 (13), 110 (18), 98 (18), 86 (20)
(<i>E,E</i>)-1,3,5-tridecatriene-7,9,11-triyne	166	166 (62), 165 (100), 164 (31), 163 (21), 150 (2), 139 (33), 138 (3), 126 (3), 115 (13), 110 (5)
(<i>E</i>)-1,3-tridecadiene-5,7,9,11-tetrayne	164	165 (14), 164 (98), 163 (100), 139 (9), 138 (79), 137 (25), 122 (13), 110 (20), 98 (19), 86 (21)
1-tridecene-3,5,7,9,11-pentayne	162	163 (14), 162 (100), 161 (20), 157 (4), 136 (38), 135 (16), 134 (18), 110 (42), 98 (2), 86 (13)

62 compounds identified included 22 sesquiterpenes, 11 C₁₃ polyacetylenes, 10 aldehydes, 7 acyclic and 1 cyclic olefinic hydrocarbons, 5 alcohols, 2 ketones, and 1 acid and 1 ester. Mass spectral data on major unknown components and on polyacetylenes are given in Table II.

The sesquiterpenes are present in greatest quantity. Their approximate concentrations are 18.3 μg/g in buds, 22.0 μg/g in flowers, and 16.2 μg/g in leaves and stems. These amounts represent 58% of total volatiles in buds, 56% in flowers, and more than 89% in leaves and stems. As found by Buttery et al. (1986), germacrene D is the major sesquiterpene. These workers tentatively identified bicyclogermacrene. We isolated some of the putative bicyclogermacrene by chromatography on silica gel. Its NMR spectrum matched the published spectrum (Nishimura et al., 1973) so the identification is confirmed. It has also been reported in *Centaurea canariensis* (Bohlmann and Gupta, 1981). Mint sulfide, (1*R*)-*cis*-2,6-epithio-*cis*-8-isopropyl-1-methyl-5-methylene-*cis*-bicyclo-[5.3.0]decane, has been reported in other Compositae species and is presumably derived from germacrene D (Takahashi et al., 1981). We recently identified mint sulfide in pineapple guava (Binder et al., 1989) and determined its DB-1 retention index.

The C₁₃ polyacetylenes were found in buds and in greater concentration in flowers. The same compounds were previously identified in *Centaurea scabiosa* roots (Anderson et al., 1977), in safflower seedlings (Binder et al., 1978), and in immature safflower seeds (Ichihara and Noda, 1975). Their biosynthesis proceeds from oleic acid (Ichihara and Noda, 1977). Some polyacetylenes have shown strong biological activity, for example as nematocides (Kogiso et al., 1976), so their presence in developing seed may be part of a plant defense mechanism.

1-Pentadecene and 1,8,11,14-heptadecatetraene (aplotaxene) are major components of bud and flower vola-

tiles. These are derived from palmitic and linolenic acids (Ney and Boland, 1987). Correspondingly, 1-tridecene and 1-heptadecene would probably derive from myristic and stearic acids, and dihydroaplotaxene would come from linoleic acid.

Initial tests of attractancy of germacrene D for *B. orientalis* were inconclusive, but some increase in copulative behavior may have occurred (Light, 1989). Further bioassays of germacrene D and other yellow starthistle volatiles will be conducted during the 1989 season.

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Registry No. 2-Pentanone, 107-87-9; hexanal, 66-25-1; (*E*)-2-hexenal, 6728-26-3; 3-methylpentanol, 589-35-5; 2-methylbutanoic acid, 116-53-0; (*Z*)-3-hexenol, 928-96-1; ethylbenzene, 100-41-4; hexanol, 25917-35-5; benzaldehyde, 100-52-7; (*E*)-2-heptenal, 18829-55-5; oct-1-en-3-ol, 3391-86-4; 6-methylhept-5-en-2-one, 110-93-0; octanal, 124-13-0; myrcene, 123-35-3; phenylacetaldehyde, 122-78-1; limonene, 138-86-3; 2-phenylethanol, 60-12-8; nonanal, 124-19-6; (*E*)-2-nonenal, 18829-56-6; (*Z*)-3-hexenyl butanoate, 16491-36-4; decanal, 112-31-2; (*E,E*)-2,4-nonadienal, 5910-87-2; benzothiazole, 95-16-9; 1-tridecene, 2437-56-1; α -cubebene, 17699-14-8; β -damascenone, 23726-93-4; cyclosatirene, 22469-52-9; α -copaene, 3856-25-5; β -bourbonene, 5208-59-3; β -cubebene, 13744-15-5; 1-tetradecene, 1120-36-1; caryophyllene, 87-44-5; β -copaene, 18252-44-3; (*E*)- α -bergamotene, 13474-59-4; (*E*)- β -farnesene, 18794-84-8; humulene, 6753-98-6; β -santalene, 511-59-1; α -muurolene, 10208-80-7; germacrene D, 23986-74-5; β -selinene, 17066-67-0; 1-pentadecene, 13360-61-7; bicyclogermacrene, 24703-35-3; α -muurolene, 10208-80-7; γ -cadinene, 39029-41-9; calamenene, 483-77-2; δ -cadinene, 483-76-1; calacorene, 38599-17-6; (*E,Z,E*)-1,3,5,11-tridecatetraene-7,9-diyne, 63366-81-4; (*E,E,E*)-1,3,5,11-tridecatetraene-7,9-diyne, 17091-00-8; (*Z,E,E*)-1,3,5,11-tridecatetraene-7,9-diyne, 124604-43-9; (*Z,Z*)-1,8,11-heptadecatriene, 56134-03-3; (*Z,E*)-1,3,11-tridecatriene-5,7,9-triyne, 124604-44-0; (*Z,Z,Z*)-1,8,11,14-heptadecateetraene, 10482-53-8; (*Z*)-1,11-tridecadiene-3,5,7,9-tetrayne, 59950-58-2; (*E,E*)-1,3,11-tridecatriene-5,7,9-triyne, 50739-51-0; 1-heptadecene, 6765-39-5; (*E*)-1,11-tridecadiene-3,5,7,9-tetrayne, 26130-86-9; mint sulfide, 72445-42-2; (*Z*)-1,3-tridecadiene-5,7,9,11-tetrayne, 124604-45-1; (*E,E*)-1,3,5-tridecatriene-7,9,11-triyne, 6581-77-7; (*E*)-1,3-tridecadiene-5,7,9,11-tetrayne, 3760-28-9; 1-tridecene-3,5,7,9,11-pentayne, 2060-59-5.

Stereoisomeric Flavor Compounds. 33. Multidimensional Gas Chromatography Direct Enantiomer Separation of γ -Lactones from Fruits, Foods, and Beverages

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Chiral γ -lactones from the raw flavor extract of strawberries and of some commercially available fruit-containing foods and beverages were directly stereoanalyzed by multidimensional gas chromatography (MDGC), employing heart-cutting techniques from DB 1701 as the preseparation column onto heptakis(3-*O*-acetyl-2,6-di-*O*-pentyl)- β -cyclodextrin as the chiral stationary phase.

The influence of optical isomerism to odor quality is well appreciated (Russell and Hills, 1971; Friedman and Miller, 1971; Ohloff, 1986; Mosandl, 1982), and in our opinion, research on the structure-function relationships of flavor substances will become increasingly important.

Chirality evaluation is a convenient method to differentiate between flavor compounds of natural origin and synthetic racemates, if comprehensive data about optical purity and fruit-specific distribution are available (Mosandl et al., 1988; Gessner et al., 1988).

Recently we reported on the first direct chiroselective analysis of chiral γ -lactones from foods and other commercially available fruit-containing preparations by off-line coupling of HPLC with HRGC on modified chiral α -cyclodextrin as a suitable chiral stationary phase (Mosandl and Kustermann, 1989b). This paper reports on multidimensional gas chromatography (MDGC) (Schomburg et al., 1984) as a very sensitive method of high selectivity to differentiate γ -lactone mirror images from complex flavor extracts, using heart-cutting techniques from DB-1701 as the nonchiral preseparation col-