Registry No. CIPC, 101-21-3; isopropyl *N*-(3-chloro-4-methoxyphenyl)carbamate, 94483-57-5.

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Volatile Components of Rooibos Tea (*Aspalathus linearis*)

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Rooibos tea (*Aspalathus linearis*) components have been identified by using capillary gas chromatography and gas chromatography/mass spectrometry. Samples were vacuum steam distilled/solvent extracted to yield a volatile oil for analysis. Among the 99 components positively or tentatively identified in the vacuum steam volatile oil are 26 ketones, 19 aldehydes, 16 alcohols, 12 esters, 9 hydrocarbons, 7 phenols, 4 acids, 3 ethers, and 3 miscellaneous components. The major components of the extract were shown to be guaiacol, 6-methyl-3,5-heptadien-2-one, damascenone, geranylacetone, β-phenylethyl alcohol, and 6-methyl-5-hepten-2-one. Headspace analysis of dry leaves yielded 218 positive or tentative identifications: 47 alcohols, 41 ketones, 39 aldehydes, 27 hydrocarbons, 24 esters, 13 ethers, 7 phenols, 6 acids, and 14 miscellaneous components.

Aspalathus linearis is a shrub native to the mountains of western South Africa. The stems are slender, and the leaves are linear and needlelike, 2–6 cm long. The leafy stems, when finely cut, fermented, and dried, are used as a substitute for tea from *Camellia sinensis* (Morton, 1983). Preliminary observations by one of the authors (J.F.M.) suggested that the plant might possess some repellent or antifeedant activity against cockroaches, so a study of Rooibos tea volatiles was initiated. Previous work had identified glycosylflavonoids and other high molecular weight components (Koeppen, 1962a,b, 1963, 1964; Koeppen and Roux, 1965), but no reference to more volatile components appears in the literature.

EXPERIMENTAL SECTION

Volatile Component Concentrate Preparation. Cured Rooibos tea was obtained from a South African producer. The material (450 g) was placed in a 5-L round-bottomed flask, and distilled water (2.5 L) was added. A modified Likens-Nickerson steam distillation/continuous extracton head was attached. Purified heptane (Burdick and Jackson; 110 mL) was used as the extracting solvent. The isolation was carried out under reduced pressure (40 mmHg) for 3-h intervals. The extraction head condenser was cooled with water/ethylene glycol at 0 °C, and a Dewar condenser filled with isopropyl alcohol/solid carbon dioxide was placed at the outlet of the system. After 3 h the heptane solution was replaced with fresh heptane. The process was then continued for a second 3-h period. This operation was repeated twice, for a total extraction period of 9 h. Each heptane extract was dried, filtered, and concentrated by careful vacuum distillation to remove the solvent. Capillary gas chromatography (GC) indicated all three extracts to be quite similar, so they were combined (4.3 mg, 9.6-ppm yield from the sample material; corrected for residual solvent).

Headspace Analysis. In a typical sequence, Rooibos tea (1 g) was placed in a sample tube (15 cm^3; 1.8 cm i.d. × 13.0 cm long) upstream from a Tenax-GC packed trap (0.635 cm o.d. × 7.6 cm long) and purified helium (25 cm^3/min) was passed through the sample, sweeping volatiles into the Tenax trap. A sampling of 1.5 h (at room temperature) was employed (2.25 L total). The trap was

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Table I. Volatile Components of Rooibos Tea

peak no.	component	headspace[d] Kovâts index[e] DB-1	headspace[d] relative[g] amount	extract[d] Kovâts index[e] DB-1	extract[d] relative[g] amount
1	(acetaldehyde)[a]	<500	0.03		
2	(methanol)[a]	<500	22.80		
3	(propanal)[a]	<500	0.30		
4	ethanol	<500	5.00[h]		
5	acetone	<500	4.68[h]		
6	2-propanol	<500	2.90[h]		
7	ethyl ether	504	0.21		
8	dichloromethane	511	tr[h]		
9	methyl acetate	513	2.99		
10	carbon disulfide	517	tr[h]		
11	nitromethane	527	0.06		
12	2-methylpropanol	530	0.19		
13	allyl alcohol	537	0.13		
14	1-propanol	548	8.14		
15	2,3-butanedione	561	5.42		
16	butanal	564	1.27		
17	2-butanone	570	0.61[h]		
18	(2-methylfuran)[b]	589	tr		
19	2-butanol	596	0.08		
20	*n*-hexane	600	tr		
21	chloroform	601	0.55[h]		
22	ethyl acetate	605	0.26[h]		
23	tetrahydrofuran	615	0.02[h]		
24	methyl propionate	618	0.11		
25	2-methyl-1-propanol	618	0.05		
26	2-butenal	622	0.50		
27	2-methyl-2-butanol	626	0.06		
28	1,1,1-trichloroethane	628	0.07		
29	3-buten-1-ol	629	0.08		
30	3-methylbutanal	632	0.30		
31	3-methyl-2-butanone	638	0.03[h]		
32	2-methylbutanal	639	0.12		
33	benzene	640	0.11		
34	tetrachloromethane	644	0.04		
35	(1-chloro-2-propanone)[b]	645	tr		
36	1-butanol	653	1.50[h]		
37	(4-penten-2-ol)[b]	657	0.12		
38	2-pentanone	662	0.03		
39	(2-methoxyethyl acetate)[b]	664	0.20		
40	3,4-dihydro-2*H*-pyran	668	0.20		
41	pentanal	671	1.00		
42	2,3-pentanedione	673	1.00		
43	3-methyl-3-buten-2-ol	673	1.00		
44	1-penten-3-ol	673	1.00		
45	(1-chloro-1-propanol)[b]	675	0.20		
46	(trichloroethylene)[b]	678	0.02		
48	3-pentanol	686	0.02		
49	2-pentanol	687	0.02		
50	2,5-dimethylfuran	695	tr		
51	*n*-heptane	700	0.10		
52	1-propyl acetate	703	0.12		
53	(2-ethylfuran)[b]	705	tr		
54	methyl *n*-butyrate	710	0.02		
55	3-penten-2-one	713	0.30		
56	3-methyl-2-butenal	717	0.09		
57	dimethyl disulfide	719	tr		
59	*trans*-2-pentenal	725	0.45		
60	3-methyl-1-butanol	727	0.33		
62	2-methyl-1-butanol	730	0.30		
63	dimethyl oxalate	742	0.02		
64	(4-penten-1-ol)[b]	745	0.16		
65	toluene	748	0.42		
67	cyclopentanone	752	tr		
68	1-pentanol	758	1.10		
69	methyl 3-methylbutyrate	761	0.20		
70	2-pentenol	764	1.00		
71	methyl 2-methylbutyrate	764	0.20		
72	2-hexanone	766	0.08		
73	hexanal	774	1.00		
74	*cis*-3-hexenal	775	0.90		
75	2-propylfuran	778	0.09		
76	3-hexanol	779	0.03		
77	2-hexanol	788	0.03		
78	(tetrachloroethylene)[b]	791	0.01		

Table I (Continued)

peak no.	component	headspace[d] Kovâts index[e] DB-1	headspace[d] relative[g] amount	extract[d] Kovâts index[e] DB-1	extract[d] relative[g] amount
79	1-butyl acetate	798	0.01		
80	furfural	799	0.38		
81	*n*-octane	800	tr		
82	methyl *n*-valerate	807	0.01		
83	(3,5-hexadien-2-one)[b]	811	0.01		
84	*trans*-2-hexenal	824	1.21		
85	acetic acid	621~827	0.20		
86	(4-methyl-1-penten-3-one)[b]	830	0.04		
87	methyl furoate	832	0.01		
88	3-hydroxy-2-butanone	670~837	2.00		
89	2-ethoxyethanol	840	0.10		
90	*cis*-3-hexenol	842	0.04		
91	ethylbenzene	842	0.10		
92	propionic acid	712~846[f]	0.28		
93	4-heptanone			850	0.21
94	*p*-xylene	851	0.21		
95	*m*-xylene	852	0.09		
96	*trans*-2-hexenol	854	0.10		
97	cyclohexanone	854	tr		
98	1-hexanol	856	0.10	856	0.07
99	3-heptanone	860	0.08	862[i]	0.47
100	1-hepten-3-ol	860	0.08	861	0.20
101	2,4-pentanediol	860~863	0.08		
102	2-heptanone	864	0.08	864[i]	0.64
103	2,5-hexanedione	864	0.08	864[i]	0.15
104	(4-heptenal)[b]	870	0.01	870	0.20
105	4-heptanol	872	tr	871[i]	0.39
106	*o*-xylene	877	0.08		
107	heptanal	877	0.09	875	0.17
108	3-heptanol	877	tr	877[i]	0.67
109	2-acetylfuran	878	0.09		
110	2-heptanol	881	tr	881[i]	0.96
111	2-methyl-1-hexanol	886	tr		
112	*n*-butyric acid	795~891[f]	0.06		
113	(2-methylbutyric acid)[b]	~896	tr		
114	(2-butoxyethanol)[b]	901	0.01		
115	(methyl hexanoate)[b]	906	0.13	907	0.06
116	dihydro-2-(3*H*)-furanone	914	0.04		
117	*n*-pentanoic acid	861~917[f]	0.04		
118	4-methylmethoxybenzene	917	0.01		
119	benzaldehyde	925	0.64	925[i]	1.59
120	α-pinene	926	tr		
121	5-methylfurfural	926	0.01		
122	2-heptenal	928	0.04	926	0.15
123	(3-methyldihydro-2(3*H*)-furanone)[b]	941	0.01	941	0.05
124	2,5-hexanediol	942~968[f]	0.02		
125	δ-valerolactone	943	0.10	943	0.04
126	(5,5-dimethyl-2(5*H*)-furanone)[b]	946	0.04	946	0.05
127	phenol	954	0.03	948	0.07
128	1-heptanol	955	0.05	953	0.05
129	cumene	961	0.08		
130	(2-methyl-2-hepten-4-one)[b]			961[i]	0.59
131	6-methyl-5-hepten-2-one	961	0.71	963[i]	3.97
132	1-octen-3-ol	964	0.63	964[i]	0.64
133	2,4-heptadienal (isomer a)	966	0.16	967[i]	2.46
134	2-octanone	969	0.05	968[i]	0.08
135	2-pentylfuran	978	0.10	977	0.10
136	octanal	979	0.10		
137	2,4-heptadienal (isomer b)	981	0.36	979[i]	2.77
138	2-octanol	983	0.15	982	0.41
139	myrcene	983	tr	983	tr
140	(1,2-dichlorobenzene)[b]	986	0.07	984	tr
141	benzyl alcohol	987	0.03	987	tr
142	*n*-hexanoic acid	997	tr		
143	1-hexyl acetate	997	0.02	997	0.04
144	*n*-decane	1000	0.06		
145	α-terpinene	1002	0.01	1001	tr
146	phenylacetaldehyde	1005	tr	1004[i]	1.17
147	methyl heptanoate	1006	tr		
148	(5-methyl-4-hepten-3-one)[b]	1007	0.10		
149	salicylaldehyde	1008	tr	1005	0.10
150	2,2,6-trimethylcyclohexanone	1012	0.30	1007[i]	0.37
151	(3-octen-2-one)[b]	1016	0.03	1011	0.46
152	2-ethylhexanol	1016	0.28		

Table I (Continued)

peak no.	component	headspace[d] Kovâts index[e] DB-1	headspace[d] relative[g] amount	extract[d] Kovâts index[e] DB-1	extract[d] relative[g] amount
153	limonene	1019	0.08	1016	tr
154	(5-ethyldihydro-2(3*H*)-furanone)[b]	1023	0.12		
155	*o*-cresol	1024	0.02		
156	acetophenone	1031	tr	1026[i]	0.40
157	2-octenal	1031	0.04	1027	0.49
158	*p*-methylbenzaldehyde	1035	tr	1032	tr
159	(2,6,6-trimethylcyclohexenone)[b]	1036	0.04	1032[i]	0.71
160	3,5-octadien-2-one (isomer a)	1040	0.21	1040[i]	2.42
161	*p*-cresol	1048	0.05	1046	tr
162	γ-terpinene	1048	tr	1046	tr
163	2-hepten-1-ol	1051	0.03	1052	0.50
164	linalool oxide (*trans*-THF)	1055	tr		
165	1-octanol	1057	0.06	1056[i]	0.23
166	guaiacol	1059	0.60	1057[i]	24.00
167	3,5-octadien-2-one (isomer b)	1065	0.05	1063[i]	1.15
168	methyl benzoate	1065	tr	1065	0.06
169	2-nonanone	1009	0.09	1069	0.19
170	6-methyl-3,5-heptadien-2-one (isomer a)[j]			1073[i]	5.18
171	linalool oxide (*cis*-THF)	1073	0.03	1073[i]	0.20
172	6-methyl-3,5-heptadien-2-one (isomer a)[J]	1076	0.22		
173	2,4-octadienal	1078	0.03	1078	0.30
174	nonanal	1083	0.24	1081	0.28
175	β-phenylethyl alcohol	1083	tr	1081[i]	4.07
176	linalool	1083	tr	1083[i]	0.54
177	6-methyl-3,5-heptadien-2-one (isomer b)	1086	0.05	1083	0.19
178	3,5,5-trimethyl-2-cyclohexenone	1089	0.07	1088[i]	0.05
179	(phenylethyl mercaptoacetate)[b]	1093	tr		
180	2-nonenal	1094	0.04		
181	methyl octanoate	1107	tr	1106	0.13
182	4-decanone	1111	0.08		
183	camphor	1113	tr		
184	2,6-nonadienal	1124	0.08	1122	0.42
185	2-nonenal	1133	0.11	1132	0.52
186	(1,2,3,4-tetrahydronaphthalene)[b]	1137	0.04		
187	isoborneol	1138	tr		
188	*p*-ethylphenol	1142	0.02	1141	0.16
189	(2,4-dimethylbenzaldehyde)[b]	1143	0.01	1140	0.13
190	methyl phenylacetate	1144	0.01	1143	0.79
191	borneol	1147	tr	1146	0.05
192	*p*-methylacetophenone	1150	0.03	1152[i]	0.50
193	1-nonanol	1155	tr		
194	naphthalene	1155	0.29	1153[i]	0.43
195	(5-methyl-1(3*H*)-isobenzofuran)[b]	1159	0.05		
196	β-terpineol	1160	0.02	1157	0.43
197	methyl salicylate	1165	0.04	1166[i]	0.42
198	α-terpineol	1172	0.16	1170[i]	0.42
199	safranal	1173	0.08	1171[i]	0.40
200	decanal	1183	0.14		
201	benzothiazole	1183	0.01	1183[i]	0.34
202	(*tert*-butylphenol)[b]	1187	0.02		
203	(2,3-dihydrobenzofuran)[b]			1191	0.32
204	[*p*-(1-methylethyl)benzaldehyde][b]	1191	0.01		
205	β-cyclocitral	1193	0.17	1194[i]	1.02
206	*n*-dodecane	1200	0.13		
207	methyl nonanoate	1206	0.05		
208	1-methyl-1,2,3,4-tetrahydronaphthalene	1212	0.04	1205[i]	0.36
209	β-phenylethyl acetate	1223	0.05	1224[i]	1.03
210	8-methyl-1,2,3,4-tetrahydronaphthalene	1243	0.06		
211	γ-octalactone	1243	tr	1230	0.42
212	neryl acetate	1245	tr	1237	0.20
213	(2,4-dihydroxyacetophenone)[b]			1244	0.21
214	2-methylnaphthalene	1267	0.21	1264[i]	0.68
215	2-undecanone	1272	tr		
216	1-methylnaphthalene	1272	0.23	1278	0.40
217	thymol			1278[i]	0.22
218	undecanal	1285	tr	1294	0.62
219	*n*-tridecane	1300	0.45		
220	γ-nonalactone			1315	0.32
221	eugenol	1325	tr	1325	1.36
222	damascenone	1360	tr	1360[i]	5.05
223	dodecanal	1385	tr	1394	0.10
224	*n*-tetradecane	1400	1.30		
225	geranylacetone	1429	tr	1429[i]	4.23
226	5,6-epoxy-β-ionone	1459	tr	1459[i]	1.61

Table I (Continued)

peak no.	component	headspace[d] Kovâts index[e] DB-1	headspace[d] relative[g] amount	extract[d] Kovâts index[e] DB-1	extract[d] relative[g] amount
227	β-ionone	1465	tr	1463[i]	1.70
228	(dihydroactinidiolide)[b]			1483[i]	2.61
229	acetyleugenol			1485[i]	0.92
230	*n*-pentadecane	1500	0.36	1500	0.19
231	(dimethylquinoline)[b]			1525	0.25
232	diethyl phthalate			1551	0.37
233	(6,10-dimethyl-3,5,9-undecatrien-2-one)[b]			1556	0.25
234	*n*-hexadecane	1600	tr	1600	0.15
235	(*n*-nonanoic acid)[c]			(2140)[c]	0.40
236	(*n*-decanoic acid)[c]			(2243)[c]	0.20
237	(*n*-undecanoic acid)[c]			(2346)[c]	0.20
238	(*n*-dodecanoic acid)[c]			(2446)*c*	0.10

[a-c] Tentative identifications in parentheses: (*a*) tentatively identified by DB-1 GC/FID retention time alone; (*b*) tentatively identified by DB-1 GC/MS spectral data alone; (*c*) tentatively identified by Carbowax 20M GC/MS spectral data alone. [d] See the text for GC operating conditions. [e] Experiment Kovâts index values from DB-1 GC/FID data, except for last four components (Carbowax 20M); reference index values determined with authentic components agree within ±2 units. [f] Retention index variable in DB-1 GC/FID and GC/MS runs. [g] Peak area percentages from GC/FID data (response factors = 1); "tr" represents less than 0.01%. [h] Appeared in headspace blank GC/FID run also. [i] Presence further verified by mass spectral and Kovâts index data from Carbowax 20M GC/MS run. [j] Elution order of 6-methyl-3,5-heptadien-2-one (isomer a) and *cis*-tetrahydrofuranyllinalool oxide differ in the headspace and the liquid injection GC runs.

reversed, attached to the headspace GC valving apparatus, and heated to 210 °C over a 30-min period to desorb the trapped material. The volatile components were collected in a small stainless steel spiral trap cooled with liquid nitrogen. The spiral trap was then heated rapidly to 225 °C, causing the sample to be swept into the gas chromatographic capillary column. Additional headspace samples were collected at sampling rates of 20, 50, and 100 cm^3/min, with total vapor sample volumes ranging from 0.1 to over 30 L. Details of the equipment and procedures have been described previously (Noble et al., 1980; Flath and Ohinata, 1982).

Chromatographic Separations. Hewlett-Packard 5830A and 5840A gas chromatographs with flame ionization detectors (FID) were employed. Two fused silica columns were used for liquid extract sample comparisons and quantitation: a 60 m × 0.32 mm i.d. DB-1 column (J & W Scientific; bonded methyl silicone phase) and a 50 m × 0.31 mm i.d. Carbowax 20M column (Hewlett-Packard; polyethylene glycol phase). Temperature programs for gas chromatographic/mass spectrometric analysis of the volatiles extract were as follows: DB-1, 50 to 250 °C at 3 °C/min; Carbowax 20M, 50 to 210 °C at 3 °C/min. Headspace/FID samples were separated on the DB-1 column only by using a 3 °C/min program rate from 0 to 250 °C. Relative retention time data for the component peaks of each Rooibos sample were obtained by coinjection of a normal hydrocarbon series (C_5–C_{26}).

A Finnigan MAT 4500 series quadrupole gas chromatograph/mass spectrometer/data system was used for acquisition of mass spectral data. The instrument was operated in the electron impact mode at an ionization voltage of 70 eV. The ion source temperature was 180 °C. The effluent end of the fused silica column was inserted directly into the ion source block. A scan speed of 1 s was used over a mass range of 33–350 amu. A split injector was used for liquid extract introduction, and a headspace valving system was employed for Tenax-trapped sample desorption and transfer. The liquid extract was run on both the DB-1 and the Carbowax 20M columns; only the DB-1 column was used for headspace samples.

RESULTS AND DISCUSSION

Table I lists the Rooibos tea components identified in this study. The Kovâts indices listed in the table were determined on the DB-1 column, and MS identifications were verified by comparison with KI values of authentic

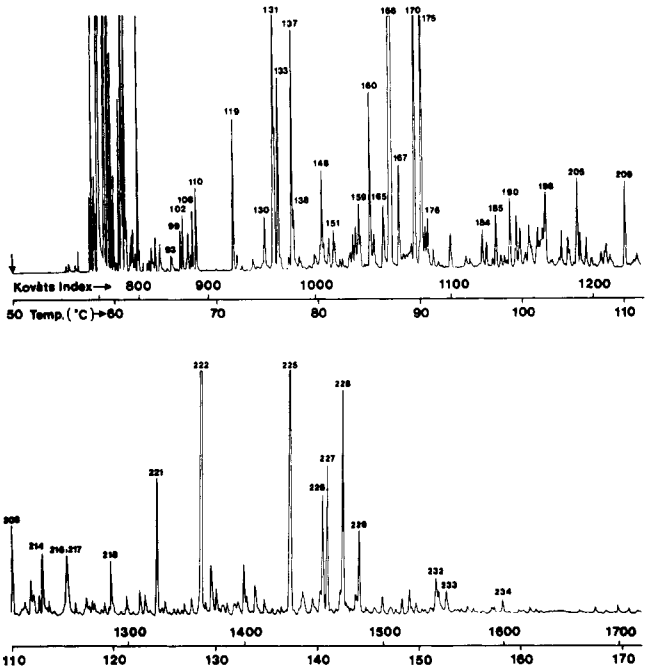

Figure 1. Typical capillary GC/FID analysis of the vacuum steam distillate oil from Rooibos tea. Programmed at 3 °C/min from 50 to 250 °C on a 60 m × 0.32 mm i.d. DB-1 column.

samples. The major components of the Rooibos tea vacuum steam distillate oil include guaiacol (24.0%), 6-methyl-3,5-heptadien-2-one isomer (5.2%), damascenone (5.0%), geranylacetone (4.2%), β-phenylethyl alcohol (4.1%), 6-methyl-5-hepten-2-one (4.0%), 2,4-heptadienal (two isomers, 2.5% and 2.8%), 3,5-octadien-2-one (two isomers, 2.4% and 1.2%), dihydroactinidiolide (tentatve identification by GC/MS only, 2.6%), β-ionone (1.7%), 5,6-epoxy-β-ionone (1.6%), and benzaldehyde (1.5%). Nonanoic, decanoic, undecanoic, and dodecanoic acids were tentatively identified in Carbowax 20M GC/MS runs. Diethyl phthalate is thought to be an artifact. Figure 1 shows a typical capillary GC/FID analysis of the vacuum steam distillate oil from Rooibos tea, using the DB-1 fused silica column. Chromatographic peaks before peak 93 (4-heptanone) are from the extracting solvent.

The major components found in the Rooibos tea headspace analysis include methanol, ethanol, acetone, 2-propanol, methyl acetate, 1-propanol, 2,3-butanedione,

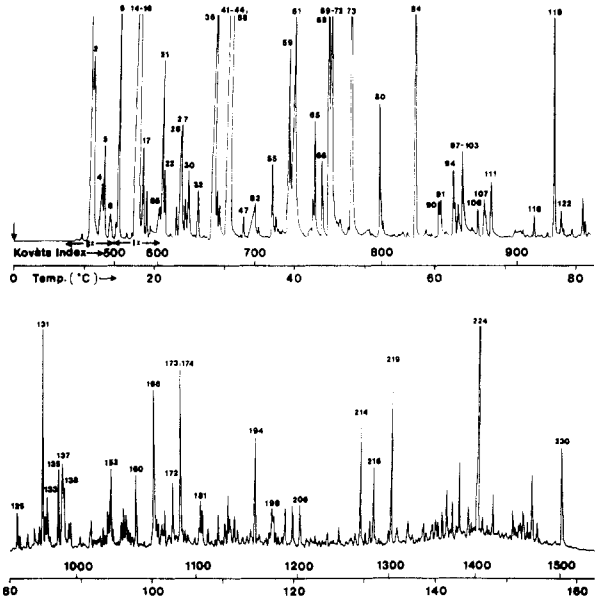

Figure 2. Typical capillary GC/FID headspace analysis of Rooibos tea leaves. Programmed at 3 °C/min from 0 to 250 °C on a 60 m × 0.32 mm i.d. DB-1 column.

3-hydroxy-2-butanone, acetic acid, and a number of aromatic and aliphatic hydrocarbons. Because headspace Tenax trapping is relatively inefficient with low molecular weight compounds such as methanol, ethanol, and acetone, the semiquantitative data for such compounds in Table I must be considered as minimum values only (Brown and Purnell, 1979). Retention behaviors of the more polar components, such as free carboxylic acids and diols were quite variable on the nonpolar DB-1 phase employed. Components displaying this variability are marked in Table I ("*f*"). The numerous hydrocarbons detected in the headspace analysis are primarily alkylbenzenes, napthalenes, indenes, and aliphatic hydrocarbons. The origin of these components is uncertain; some are very likely sample contaminants adsorbed on the tea surface. The various chlorinated constitutents listed, as well as *tert*-butylphenol and carbon disulfide, are also thought to be artifacts. Figure 2 shows a typical capillary/FID GC analysis of Rooibos tea headspace volatiles. The elution order of 6-methyl-3,5-heptadien-2-one (isomer a) and linalool oxide (*cis*-tetrahydrofuranyl) differs on the headspace and extract GC runs. Presumably this is due to the different GC operating conditions employed.

A comparison of reported green and black tea volatiles (Yamanishi, 1981) with Table I shows some similarities but several major differences. Guaiacol is present in high concentration in the Rooibos tea steam distillate extract but not in *C. sinensis* teas. In contrast, linalool and geraniol are present in high concentrations in green tea or black tea, but in the Rooibos tea volatiles extract linalool is present only in moderate concentration, and geraniol was not found.

An evaluation of repellent activity against German cockroaches (*Blattela germanica*) detected no significant activity for either cured Rooibos tea or the vacuum steam distillate in a standard screening procedure (Patterson, 1983).

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Registry No. **1**, 75-07-0; **2**, 89-78-1; **3**, 123-38-6; **4**, 64-17-5; **5**, 67-64-1; **6**, 67-63-0; **7**, 60-29-7; **8**, 75-09-2; **9**, 79-20-9; **10**, 75-15-0; **11**, 75-52-5; **12**, 78-83-1; **13**, 107-18-6; **14**, 71-23-8; **15**, 431-03-8; **16**, 123-72-8; **17**, 78-93-3; **18**, 534-22-5; **19**, 78-92-2; **20**, 110-54-3; **21**, 67-66-3; **22**, 141-78-6; **23**, 109-99-9; **24**, 554-12-1; **25**, 78-83-1; **26**, 4170-30-3; **27**, 75-85-4; **28**, 71-55-6; **29**, 627-27-0; **30**, 590-86-3; **31**, 563-80-4; **32**, 96-17-3; **33**, 71-43-2; **34**, 56-23-5; **35**, 78-95-5; **36**, 71-36-3; **37**, 625-31-0; **38**, 107-87-9; **39**, 110-49-6; **40**, 110-87-2; **41**, 110-62-3; **42**, 600-14-6; **43**, 10473-14-0; **44**, 616-25-1; **45**, 94484-16-9; **46**, 79-01-6; **48**, 574-02-7; **49**, 6032-29-7; **50**, 625-86-5; **51**, 142-82-5; **52**, 109-60-4; **53**, 3208-16-0; **54**, 623-42-7; **55**, 625-33-2; **56**, 107-86-8; **57**, 624-92-0; **59**, 1576-87-0; **60**, 123-51-3; **62**, 137-32-6; **63**, 553-90-2; **64**, 821-09-0; **65**, 108-88-3; **67**, 120-92-3; **68**, 71-41-0; **69**, 556-24-1; **70**, 60544-74-3; **71**, 868-57-5; **72**, 591-78-6; **73**, 66-25-1; **74**, 6789-80-6; **75**, 4229-91-8; **76**, 623-37-0; **77**, 626-93-7; **78**, 127-18-4; **79**, 123-86-4; **80**, 98-01-1; **81**, 111-65-9; **82**, 624-24-8; **83**, 2957-06-4; **84**, 6728-26-3; **85**, 64-19-7; **86**, 1606-47-9; **87**, 611-13-2; **88**, 513-86-0; **89**, 110-80-5; **90**, 928-96-1; **91**, 100-41-4; **92**, 79-09-4; **93**, 123-19-3; **94**, 106-42-3; **95**, 108-38-3; **96**, 928-95-0; **97**, 108-94-1; **98**, 111-27-3; **99**, 106-35-4; **100**, 4938-52-7; **101**, 625-69-4; **102**, 110-43-0; **103**, 110-13-4; **104**, 62238-34-0; **105**, 589-55-9; **106**, 95-47-6; **107**, 111-71-7; **108**, 589-82-2; **109**, 1192-62-7; **110**, 543-49-7; **111**, 624-22-6; **112**, 107-92-6; **113**, 116-53-0; **114**, 111-76-2; **115**, 106-70-7; **116**, 96-48-0; **117**, 109-52-4; **118**, 104-93-8; **119**, 100-52-7; **120**, 80-56-8; **121**, 620-02-0; **122**, 2463-63-0; **123**, 1679-47-6; **124**, 2935-44-6; **125**, 542-28-9; **126**, 20019-64-1; **127**, 108-95-2; **128**, 111-70-6; **129**, 98-82-8; **130**, 22319-24-0; **131**, 110-93-0; **132**, 3391-86-4; **133**, 5910-85-0; **134**, 111-13-7; **135**, 3777-69-3; **136**, 124-13-0; **138**, 123-96-6; **139**, 123-35-3; **140**, 95-50-1; **141**, 100-51-6; **142**, 142-62-1; **143**, 142-92-7; **144**, 124-18-5; **145**, 99-86-5; **146**, 122-78-1; **147**, 106-73-0; **148**, 1447-26-3; **149**, 90-02-8; **150**, 2408-37-9; **151**, 1669-44-9; **152**, 104-76-7; **153**, 138-86-3; **154**, 695-06-7; **155**, 95-48-7; **156**, 98-86-2; **157**, 2363-89-5; **158**, 104-87-0; **159**, 69247-91-2; **160**, 38284-27-4; **161**, 106-44-5; **162**, 99-85-4; **163**, 22104-77-4; **164**, 34995-77-2; **165**, 111-87-5; **166**, 90-05-1; **168**, 93-58-3; **169**, 821-55-6; **171**, 5989-33-3; (*E*)-**172**, 16647-04-4; **173**, 5577-44-6; **174**, 124-19-6; **175**, 60-12-8; **176**, 78-70-6; (*Z*)-**177**, 29178-96-9; **178**, 78-59-1; **179**, 94484-17-0; **180**, 2463-53-8; **181**, 111-11-5; **182**, 624-16-8; **183**, 76-22-2; **184**, 26370-28-5; **185**, 2463-53-8; **186**, 119-64-2; **187**, 124-76-5; **188**, 123-07-9; **189**, 15764-16-6; **190**, 101-41-7; **191**, 507-70-0; **192**, 122-00-9; **193**, 143-08-8; **194**, 91-20-3; **195**, 54120-64-8; **196**, 138-87-4; **197**, 119-36-8; **198**, 98-55-5; **199**, 116-26-7; **200**, 112-31-2; **201**, 95-16-9; **202**, 27178-34-3; **203**, 496-16-2; **204**, 122-03-2; **205**, 432-25-7; **206**, 112-40-3; **207**, 1731-84-6; **208**, 1559-81-5; **209**, 103-45-7; **210**, 2809-64-5; **211**, 104-50-7; **212**, 141-12-8; **213**, 89-84-9; **214**, 91-57-6; **215**, 112-12-9; **216**, 90-12-0; **217**, 89-83-8; **218**, 112-44-7; **219**, 629-50-5; **220**, 104-61-0; **221**, 97-53-0; **222**, 23726-93-4; **223**, 112-54-9; **224**, 629-59-4; **225**, 3796-70-1; **226**, 23267-57-4; **227**, 79-77-6; **228**, 17092-92-1; **229**, 93-28-7; **230**, 629-62-9; **231**, 28351-04-4; **232**, 84-66-2; **233**, 141-10-6; **234**, 544-76-3; **235**, 112-05-0; **236**, 334-48-5; **237**, 112-37-8; **238**, 143-07-7.

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