

ESSENTIAL OIL OF *XYLOPIA SERICEA*. A COMPARATIVE ANALYSIS

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*Xylopia sericea* St. Hil. (Annonaceae) grows wild in Northeastern Brazil, where it is known as "Embiriba." An infusion made from its fruits has found some popular use as a treatment for upset stomach.

This is the first chemical report on the volatiles from this species. Preliminary examination of the essential oil extracted from the fruits showed by capillary gc a large number of components. Many of these were present only as traces or in amounts too small for a successful identification. Therefore, to improve the confidence of the results, we decided to examine the oil samples by gc/ms techniques independently in two places: Nuernberg (FRG) and in Fortaleza-Ceara (Brazil). The identifications consistent between both analyses represented 88.57% of the total oil composition (see Table 1), along with 9.83% identified only by molecular formulas ( $C_{10}H_{16}$ ,  $C_{10}H_{16}O$ ,  $C_{15}H_{24}$ ,  $C_{15}H_{24}O$ ) and 1.60% unidentified. Among the compounds identified by molecular formulas only were two sesquiterpene alcohols in relatively high concentrations (4.45 and 2.61%). These will be the object of further investigation.

## EXPERIMENTAL

**PLANT MATERIAL AND ESSENTIAL OIL.**—The plant materials from *X. sericea* were collected in September 1981, in Viçosa, Ceara, Brazil. Essential oil was steam distilled from the dried fruits of the plant using standard procedures (1); yield was 0.27%.

**GENERAL PROCEDURES.**—Chromatograms were recorded on a Varian 1400 gas chromatograph (FID) equipped with a fused silica SP-2100 capillary column (25 m, i.d. = 0.25 mm) temperature programmed from 50-250° at 4°/min, a Perkin-Elmer Sigma 1 (FID) with fused silica SE-54 capillary column (50 m, i.d. = 0.25 mm) 4 min isothermal at 60° and then temperature programmed 60-260° at 3°/min, a Perkin-Elmer 990 (FID) with fused silica carbowax ZOM capillary column (50 m, i.d. = 0.25 mm) 8 min isothermal at 70° and then temperature programmed 70-200° at 4°/min. Carrier gas and flow rate not specified in gc experiments means  $N_2$  as carrier gas and 1 ml/min as flow rate.

Mass spectra were recorded with a Hewlett-Packard 5995 gc/ms equipped with a fused silica SP-2100 capillary column (30 m, i.d. = 0.25 mm) using helium as carrier gas (1 ml/min) and temperature programmed from 50-250° at 4°/min, and a Finnigan 3200E gc/ms equipped with fused silica CP-SIL19CB (38 m, i.d. = 0.25 mm) and SE-30 (27 m, i.d. = 0.25 mm) capillary columns, helium (1 ml/min) as carrier gas, isothermal 5 min at 60° then temperature programmed 60-240° at 4°/min, and 3 min at 70°, then temperature programmed 70-220° at 3°/min, respectively.

**IDENTIFICATION PROCEDURES.**—The Kovats retention indices were determined on SP-2100, SE-54, and carbowax 20M using a temperature program producing a linear sequence of retention times for the *n*-alkanes C8 to C18 and compared with those of authentic compounds from the literature (2) and from previous work (3-5). Basic library search was based on library search programs using Kovats indices as preselection criteria (6).

Mass chromatograms were recorded using *m/z* values depending on the molecular formulas such as:  $C_{10}H_{16}$ : *m/z* 93, 121, and 136;  $C_{15}H_{24}$ : *m/z* 93, 161, and 204;  $C_{10}H_{14}O$ : *m/z* 132, 135, and 150;  $C_{10}H_{16}O$ : *m/z* 134, 137, and 152;  $C_{10}H_{18}O$ : *m/z* 136, 139, and 154;  $C_{15}H_{26}$ : *m/z* 204, 207, and 222.

Confirmation of the mass spectroscopic identifications was achieved by visual comparisons of the unknown spectra with spectra of authentic samples, with spectra from the literature (7,8), with those from previous work (3-5), and by the methods indicated in Table 1. Concentrations (%) presented in Table 1 are the means from the data obtained in Brazil (HP-5995A) and in Germany (Finnigan 3200E).

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TABLE 1. Chemical Constituents of the Essential Oil of *Xylopiia sericea*

Compound	Kovats <sup>a</sup> (SE-54)	Kovats <sup>b</sup> (SP-2100)	Yield <sup>c</sup> (%)	Identification <sup>d</sup>
Tricyclene	922	—	0.04	ri gc
$\alpha$ -Thujene	926	921	0.34	ri ms gc cc
$\alpha$ -Pinene	934	927	6.35	ri ms gc
C <sub>10</sub> H <sub>16</sub>	943	935	tr	ms
Camphene	948	940	0.69	ri ms gc
Fenchene	953	—	0.13	ri gc
C <sub>10</sub> H <sub>16</sub>	961	—	tr	ms
Sabinene	973	960	1.10	ri ms gc
$\beta$ -Pinene	978	965	6.85	ri ms gc
Myrcene	991	979	0.82	ri ms gc
$\alpha$ -Phellandrene	1005	992	0.32	ri ms gc cc
Car-3-ene	1011	—	0.03	ri gc cc
$\alpha$ -Terpinene	1017	1008	0.12	ri mc gc
<i>p</i> -Cymene	1029	1009	17.92	ri ms cc
Limonene	1029	1020	6.41	ri gc
1,8-Cineole	1034	1017	22.32	ri ms gc
Z- $\beta$ -Ocimene	1038	1030	0.05	ms
E- $\beta$ -Ocimene	1048	—	0.28	ri ms gc
$\gamma$ -Terpinene	1059	1045	0.17	ri ms gc
<i>p</i> -Cymenene	1059	1067	0.05	ri ms gc
C <sub>10</sub> H <sub>16</sub>	1068	—	0.05	ms
Unknown	1072	—	0.07	ms
Unknown	1077	—	0.04	ms
Terpinolene	1089	1067	0.38	ri gc cc
Fenchone	1090	1080	tr	ri cc
Linalol	1097	1081	0.12	ri ms
C <sub>10</sub> H <sub>14</sub> O	1100	—	0.44	ms
Thujone	1106	1100	0.10	ri cc
Isothujone	1116	1106	0.10	ms
Unknown	1122	—	0.11	ms
C <sub>10</sub> H <sub>18</sub> O	1127	—	0.13	ms
C <sub>10</sub> H <sub>18</sub> O	1132	—	0.05	ms
E-Pinocarveol	1140	1109	0.70	ri ms cc
Camphor	1145	1115	0.02	ms
Isoborneol	1159	1137	0.19	ms
C <sub>10</sub> H <sub>18</sub> O	1163	—	0.22	ms
Borneol	1169	1145	0.43	ri ms
C <sub>10</sub> H <sub>18</sub> O	1176	—	0.30	ms
Terpinen-4-ol	1179	1148	1.05	ri ms gc
Isopropyl anisole	—	1154	tr	ms
Myrtenal	1188	1161	1.04	ri ms
$\alpha$ -Terpineol	1194	1164	1.88	ri ms gc
Myrtenol	1200	1172	0.55	ri ms gc cc
<i>trans</i> -Carveol	1122	1197	0.02	ms ri
Verbenone	1212	1200	0.22	ri cc
Cuminaldehyde	—	1205	0.37	ms
Carvone	1248	1208	0.32	ms
<i>p</i> -Mentha-1,5,8-trien-4-ol	—	1239	0.33	ms
Geraniol	1256	1244	0.20	cc
Campholenaldehyde	1251	1239	0.12	ms
Unknown	1280	—	0.78	ms
Isobornyl acetate	1292	1263	1.26	ms ri
Thymol	1296	1273	0.01	ms ri
Carvacrol	1305	1290	0.44	ms ri
Terpinyl acetate	1352	1267	0.50	ms

TABLE 1. Continued

Compound	Kovats <sup>a</sup> (SE-54)	Kovats <sup>b</sup> (SP-2100)	Yield <sup>c</sup> (%)	Identification <sup>d</sup>
β-Cubebene . . . . .	—	—	0.07	ms
Neryl acetate . . . . .	1370	1350	0.06	ri ms
Ylangene . . . . .	1374	1352	0.05	ms cc
α-Cubebene . . . . .	1379	1369	0.92	ri ms gc cc
Unknown . . . . .	1385	—	0.11	
α-Copaene . . . . .	1379	1362	0.92	ri ms gc cc
Geranyl acetate . . . . .	1384	1372	0.06	ri ms
β-Caryophyllene . . . . .	1423	1404	0.93	ri ms cc
α-Bergamotene . . . . .	—	1415	0.01	ms
C <sub>15</sub> H <sub>24</sub> . . . . .	—	—	0.13	ms
α-Humulene . . . . .	1457	1431	0.28	ri gc cc
Unknown . . . . .	1464	1440	0.16	gc
γ-Murolene . . . . .	1477	1461	0.22	ri ms
C <sub>15</sub> H <sub>24</sub> . . . . .	1480	—	0.15	ms
Germacrene ? . . . . .	1496	1476	0.27	ms
n-Pentadecane . . . . .	1500	1500	tr	ms
α-Murolene . . . . .	1503	1484	0.41	ri ms
α-Elemene . . . . .	—	1490	0.16	ms
γ-Cadinene . . . . .	1520	1520	0.17	ms ri cc
Calamenene . . . . .	—	1505	tr	ri ms
Δ-Cadinene . . . . .	1526	1510	1.77	ri ms gc
Unknown . . . . .	1547	1525	0.28	gc
C <sub>15</sub> H <sub>24</sub> O . . . . .	1558	1535	0.35	ms
C <sub>15</sub> H <sub>24</sub> O . . . . .	—	1535	0.35	ms
C <sub>15</sub> H <sub>24</sub> O . . . . .	—	1554	0.12	ri ms cc
C <sub>15</sub> H <sub>24</sub> O . . . . .	—	1563	2.61	ms
C <sub>15</sub> H <sub>24</sub> O . . . . .	—	1473	4.45	ms
C <sub>15</sub> H <sub>24</sub> O . . . . .	—	1578	0.48	ms
n-Hexadecane . . . . .	1600	1600	0.23	ms

<sup>a</sup>Gc/ms run on a Finnigan 3200E (Nuernberg-RFG).

<sup>b</sup>Gc/ms run on a HP-5995A (Fortaleza-Ceará-Brazil).

<sup>c</sup>Yields were calculated as the means of the concentrations obtained on column SE-54 and on column SP-2100; tr % less than 0.01%.

<sup>d</sup>ri = retention index

gc = gas chromatography

ms = mass spectrum

cc = co-chromatography.

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