

## 绿槽枝衣的化学成分

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**摘要:** 从地衣绿槽枝衣 (*Sulcaria virens*) 中分离得到一个新的亚油酸异丙叉衍生物, 通过波谱学方法包括 2D-NMR 确定其化学结构为: 9, 10-O-异丙叉基-(12 Z)-十八碳烯酸 (1)。同时还得到其它 12 个已知化合物: (9 Z, 12 Z)-十八碳二烯酸 (2), 扁枝衣二酸 (3), (R)-松萝酸 (4), 枕酸甲酯 (5), 黑茶渍素 (6), virensic acid (7), abieslactone (8), 3-羟基羊毛甾-7, 24-二烯-26, 23 R-内酯 (9), 蒲公英赛醇 (10), 蒲公英赛酮 (11), (22 E, 24 R)-5, 8-过氧麦角甾-6, 22-二烯-3-醇 (12) 和 2, 2-四氢角鲨烯 (13)。

**关键词:** 地衣; 绿槽枝衣; 化学成分; 亚油酸衍生物

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## Chemical Investigation of *Sulcaria virens* (Alectoriaceae)

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**Abstract:** A new linoleic acid derivative, was isolated from the lichen *Sulcaria virens* together with 12 known compounds. The structure of the new compound was determined as 9, 10-O-isopropylidene -(12 Z)-en-octadecaic acid (1) by spectroscopic analysis. Other 12 known compounds were (9 Z, 12 Z)-octadecadienoic acid (2), evernic acid (3), (R)-usnic acid (4), vulpinic acid (5), atranorin (6), virensic acid (7), abieslactone (8), 3-hydroxylanosta-7, 24-dien-26, 23 R-olide (9), taraxerol (10), taraxerone (11), 5, 8-epidioxy-(22 E, 24 R)-ergosta-6, 22-dien-3-ol (12), 2, 2-tetrahydrosqualene (13).

**Key words:** Lichen; *Sulcaria virens*; Component; Linoleic acid derivative

*Sulcaria virens* (Tayl.) Bystr. ex Brodo & Hawksw. belongs to the family Alectoriaceae of lichenized Ascomyceta (Nash, 1996), is mainly distributed to the Himalaya. It is a kind of fruticose lichen and its thallus is bright yellow, corticolous, pendulous (Awasthi, 1985). Previous investigations of lichen in the genus *Sulcaria* have reported the isolation of virensic acid, convirensic acid, vulpinic acid (Culberson *et al.* 1981), and

two depsidons (Elix *et al.* 1999; Elix *et al.* 2000). We have carried out a detailed chemical investigation on this lichen and isolated 13 compounds from its acetone extracts including a new linoleic acid derivative, 9, 10-O-isopropylidene -(12 Z)-en-octadecaic acid (1). This paper reports the isolation and structural elucidation of the new compound. In addition, the <sup>1</sup>H- and <sup>13</sup>C-NMR data of 13 were assigned for the first time.

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## Results and Discussion

The 13 compounds from *S. virens* are mainly classed to depsides, depsidones, dibenzofuran derivatives and triterpenes, which are abundant in lichen (Fig. 1). Compounds **2**, **5** and **12** are also often found in fungi.

Compound **1** was obtained as colorless oil with a molecular formula  $C_{21}H_{38}O_4$ . Its  $^1H$ -NMR (Table 1) showed one methyl at  $\delta_H$  0.89 (3H, t,  $J = 7.0$  Hz, H - 18), three methylenes at  $\delta_H$  1.63 (2H, m, H - 3),  $\delta_H$  2.03 (2H, m, H - 14),  $\delta_H$  2.35 (2H, t,  $J = 7.2$  Hz, H - 2), a double bond at  $\delta_H$  5.43 (1H, dt,  $J = 10.6, 7.7$  Hz, H - 12), 5.52 (1H, dt,  $J = 10.6, 7.2$  Hz, H - 13) and two methyls at  $\delta_H$  1.38 (6H, s),

which is characteristic of the unsaturated fatty acid derivatives. A quaternary carbon at  $\delta_C$  107.9 (s, C - 19) and the methyls protons at  $\delta_H$  1.38 (6H, s) showed important correlations in HMBC spectrum (Fig. 2), characteristic of the isopropylidene moiety. The  $^{13}C$ -NMR (Table 1) of compound **1** showed total 21 carbons signals, three of which located at the isopropylidene moiety, and eighteen of which located at the long chain unsaturated fatty acid skeleton. In HMBC spectrum of **1**, appearance of key correlations between  $\delta_H$  2.03 (2H, m, H - 14) and  $\delta_C$  31.5 (t, C - 16),  $\delta_H$  0.89 (3H, t,  $J = 7.0$  Hz, H - 18) and  $\delta_C$  31.5 (t, C - 16), but no correlations between olefinic proton  $\delta_H$  5.52 (1H, dt,  $J = 10.6, 7.2$  Hz, H - 13) and  $\delta_C$  31.5

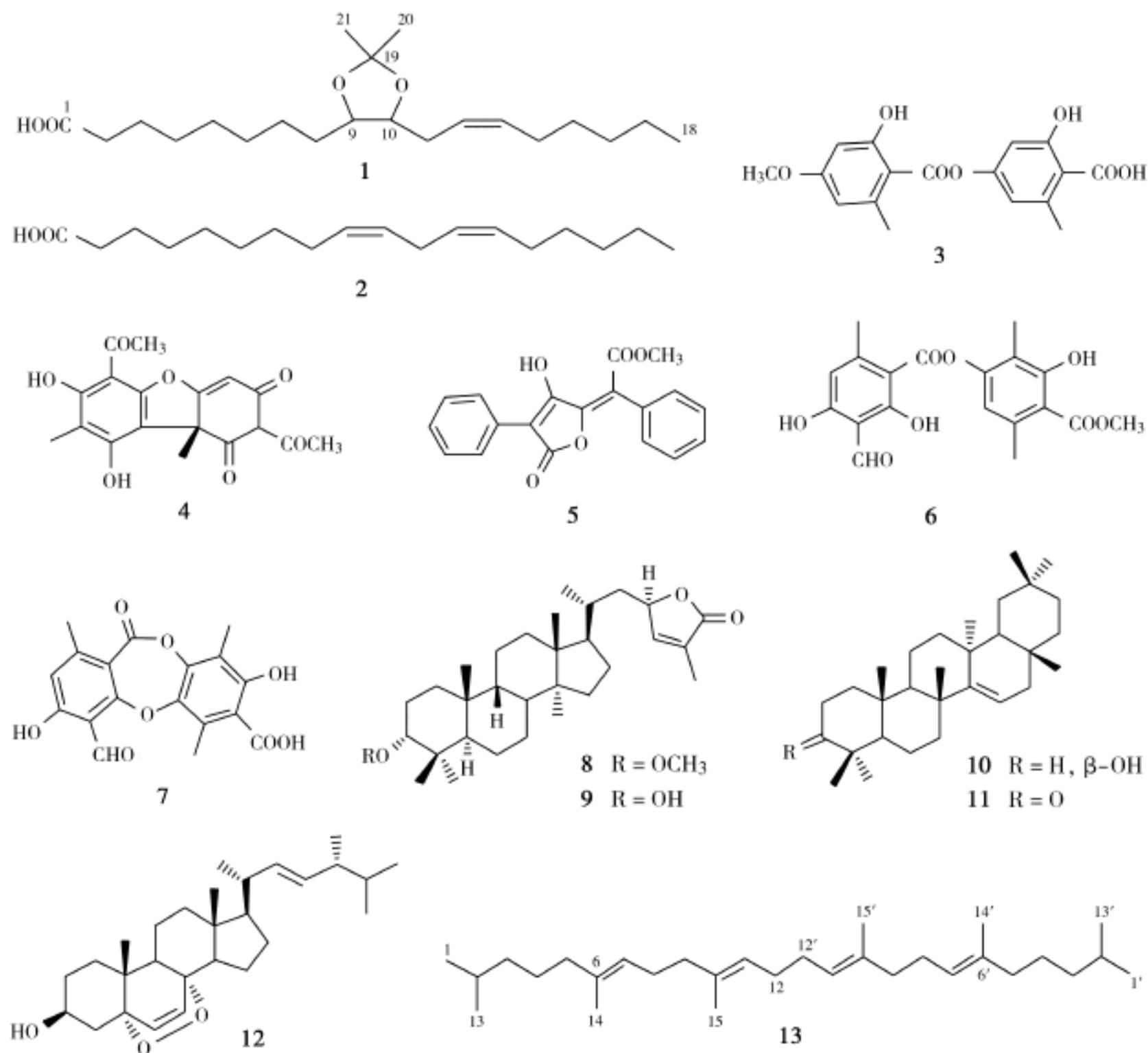


Fig. 1 Structures of compounds 1 - 13



Fig . 2 The key HMBC correlations of compound 1

Table 1  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR data for **1** in  $\text{CDCl}_3$ 

NO	H	C
1		179.1 (s)
2	2.35 (t, 7.2)	33.9 (t)
3	1.63 (m)	24.6 (t)
4	1.25 - 1.36 <sup>a</sup>	29.0 (t) <sup>b</sup>
5	1.25 - 1.36 <sup>a</sup>	29.1 (t) <sup>b</sup>
6	1.25 - 1.36 <sup>a</sup>	29.2 (t) <sup>b</sup>
7	1.25 - 1.36 <sup>a</sup>	26.1 (t)
8	1.51 (m)	33.0 (t)
9	3.65 (m)	80.6 (d)
10	3.65 (m)	80.4 (d)
11	2.33 (t, 7.7)	30.6 (t)
12	5.43 (dt, 10.6, 7.7)	124.2 (d)
13	5.52 (dt, 10.6, 7.2)	132.5 (d)
14	2.03 (m)	27.4 (t)
15	1.25 - 1.36 <sup>a</sup>	29.5 (t) <sup>b</sup>
16	1.25 - 1.36 <sup>a</sup>	31.5 (t)
17	1.25 - 1.36 <sup>a</sup>	22.6 (t)
18	0.89 (t, 7.0)	14.0 (q)
19		107.9 (s)
20	1.38 (s)	27.2 (q) <sup>c</sup>
21	1.38 (s)	27.3 (q) <sup>c</sup>

<sup>a</sup> Overlapped signals . <sup>b, c</sup> Assignments maybe interchanged .

(t, C - 16),  $^1\text{H}$  2.03 (2H, m, H - 14) and  $^{13}\text{C}$  22.6 (t, C - 17) suggested the double bond located at C - 12 and C - 13 . The correlations between  $^1\text{H}$  2.33 (2H, t,  $J = 7.7$  Hz, H - 11) and  $^{13}\text{C}$  80.6 (d, C - 9), 80.4 (d, C - 10), 124.2 (d, C - 12), 132.5 (d, C - 13),  $^1\text{H}$  3.65 (1H, m, H - 10) and olefinic carbon at  $^{13}\text{C}$  124.2 (d, C - 12) indicated the isopropylidene located at C - 9 and C - 10 . The *Z* configuration for the double bond was suggested by its coupling constant ( $J = 10.6$  Hz) . Thus, the structure of **1** was elucidated as 9, 10-O-isopropylidene -(12 *Z*)-en -octadecaic acid . Compound **1** seems to be a work-up product of compound **2**, but when we carried out a reaction of compound **2** and acetone, compound **1** was failed to be detected . In our research, compound **2** was often found in higher fungi, while isopropylidene derivatives of fatty acids were not isolated as before .

## Experimental

**General** CC: Column chromatography . TLC: Visualization by heating silica-gel plates sprayed with 10%  $\text{H}_2\text{SO}_4$  in EtOH . Silica gel (200 - 300 mesh, Qingdao Marine Chemical Inc, P . R . China) and Sephadex LH-20 (Amersham Biosciences, Sweden) were used for column chromatography . Pre-coated silica gel GF-254 plates (Qingdao Marine Chemical Inc, P . R . China) . Optical rotation: Horiba SEPA-300 spectropolarimeter .  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR spectra: Bruker AV-400 and DRX-500 spectrometers, in ppm,  $J$  in Hz . MS: VG Autospec-3000 spectrometer,  $m/z$  (rel . int .) .

**Lichen Material** The fresh lichen thallus were collected from Laojun Mountains at an altitude of 3 700 m of Lijiang County, Yunnan Province, China, on bark of *Rhododendron* sp . in June 2005 and identified by Wang Li-Song . A voucher specimen (Wang 05 - 24631) was deposited at the Herbarium of Kunming Institute of Botany, Chinese Academy of Sciences .

**Extraction and Isolation** The dried lichen thallus (180 g) was extracted twice with acetone, then extracted once with methanol at room temperature . The combined extracts were concentrated to dryness under reduced pressure to give a residue (28 g) which was applied on a silica gel column with a gradient elution of petroleum ether and acetone (100 0, 98 2, 95 5, 90 10, 80 20, 50 50, 0 100), subsequently seven fractions (A-G) obtained . Fraction A was further subjected to column chromatography over silica gel (pure petroleum ether) and Sephadex LH-20 ( $\text{CHCl}_3/\text{MeOH}$ , 1/1) to afford compound **13** (9.2 mg) . Fraction B was rechromatographed on Sephadex LH-20 ( $\text{CHCl}_3/\text{MeOH}$ , 1/1) to give two subfractions, which were recrystallized to afford pure compounds **8** (6.2 mg) and **11** (3.1 mg) . Recrystallization of fraction C and fraction F gave a large amount of compounds **5** (7 500 mg) and **7** (5 400 mg), respectively . The rest of fraction C was further purified with Sephadex LH-20 ( $\text{CHCl}_3/\text{MeOH}$ , 1/1) to afford compounds **9** (1.0 mg) and **10** (0.8 mg) . From the rest of fraction F, compounds **2** (17.2 mg) and **3** (7.6 mg) were obtained with repeated chromatography ( $\text{CHCl}_3/\text{MeOH}$ , 125/1 - 60/1) on silica gel column . The crystals from fraction D were rechromatographed on Sephadex LH-20 ( $\text{CHCl}_3/\text{MeOH}$ , 1/1) to yield compounds **4** (35.5 mg) and **6**

(0.6 mg). The fraction E was purified by repeated silica gel column (petroleum ether/acetone, 200/1 - 100/1) to give compounds **1** (6.4 mg) and **12** (9.1 mg).

**9, 10-O-isopropylidene - (12 Z) - en - octadecanoic acid (1)**,  $C_{21}H_{38}O_4$ , colorless oil; EI-MS  $m/z$  (%): 354 [M]<sup>+</sup> (25), 339 (65), 279 (35), 243 (100), 225 (83), 207 (13), 185 (98), 167 (50), 149 (30), 139 (37), 121 (52), 95 (27); <sup>1</sup>H- and <sup>13</sup>C-NMR, Table 1.

**Evernic acid (3)**,  $C_{17}H_{16}O_7$ ; white powder; FAB-MS (neg.)  $m/z$ : 331 [M-H]<sup>-</sup>; <sup>13</sup>C-NMR data (CD<sub>3</sub>COCD<sub>3</sub>):  $\delta$  173.7 (s), 170.3 (s), 166.7 (s), 165.8 (s), 165.5 (s), 144.7 (s), 144.3 (s), 117.3 (d), 112.1 (d), 111.3 (s), 109.5 (d), 105.6 (s), 99.7 (d), 55.9 (q), 24.3 (q), 23.9 (q); <sup>1</sup>H- and <sup>13</sup>C-NMR data: same as the data reported in Narui *et al.* (1998).

**(R)-Usnic acid (4)**,  $C_{18}H_{16}O_7$ , yellow needles (CHCl<sub>3</sub>/MeOH);  $[\alpha]_D^{16} = +449^\circ$  ( $c = 0.185$ , CHCl<sub>3</sub>); EI-MS  $m/z$  (%): 344 [M]<sup>+</sup> (45), 260 (55), 233 (100), 217 (25), 189 (3), 161 (4); The data of optical rotation are the same as reported in Huneck *et al.* (1976); MS data are similar to the data reported in Huneck *et al.* (1968).

**Vulpinic acid (5)**,  $C_{19}H_{14}O_5$ , yellow plates (petroleum ether/acetone); EI-MS  $m/z$  (%): 322 [M]<sup>+</sup> (20), 290 (80), 261 (10), 234 (16), 178 (15), 145 (100), 117 (24), 89 (34), 77 (6), 63 (5); <sup>13</sup>C-NMR data (CD<sub>3</sub>COCD<sub>3</sub>):  $\delta$  172.7 (s), 166.4 (s), 161.6 (s), 155.0 (s), 133.4 (s), 130.9 (d, 2 × C), 130.1 (s), 129.16 (d, 2 × C), 129.13 (d), 128.9 (d), 128.7 (d, 2 × C), 128.3 (d, 2 × C), 117.0 (s), 104.7 (s), 55.0 (q); <sup>1</sup>H-NMR data: same as the data reported in Duncan *et al.* (2003); <sup>13</sup>C-NMR data: same as the data reported in K nig *et al.* (1999).

**Atranorin (6)**,  $C_{19}H_{18}O_8$ , colorless needles; EI-MS  $m/z$  (%): 374 [M]<sup>+</sup> (1), 196 (52), 179 (13), 164 (100), 150 (7), 136 (94), 121 (4), 107 (11), 79 (9), 67 (4), 53 (3); MS data are the same as reported in Huneck *et al.* (1968).

**Virensic acid (7)**,  $C_{18}H_{14}O_8$ , white powder; EI-MS  $m/z$  (%): 358 [M]<sup>+</sup> (50), 340 (30), 314 (100), 299 (15), 285 (20), 272 (36), 258 (70), 243 (40), 230 (50), 190 (8), 179 (44); <sup>13</sup>C-NMR data (CD<sub>3</sub>COCD<sub>3</sub>):  $\delta$  194.3 (s), 174.0 (s), 166.1 (s), 165.6 (s), 161.6 (s), 160.1 (s), 154.4 (s), 147.4 (s), 143.1 (s), 130.9 (s), 118.1 (d), 116.9 (s), 113.6 (s), 111.8 (s), 110.9 (s), 22.0 (q), 15.5 (q), 9.2 (q); The data of <sup>1</sup>H- and <sup>13</sup>C-NMR are similar to those data reported in Elix *et al.* (2000).

**Abieslactone (8)**,  $C_{31}H_{48}O_3$ , colorless needles; EI-MS  $m/z$  (%): 468 [M]<sup>+</sup> (5), 453 (14), 421 (95), 393 (3),

351 (3), 325 (4), 314 (30), 299 (22), 227 (30), 213 (32), 201 (22), 187 (78), 175 (77), 159 (62), 147 (71), 135 (100); <sup>13</sup>C-NMR data (CDCl<sub>3</sub>):  $\delta$  30.0 (t), 23.0 (t), 85.9 (d), 37.6 (s), 42.8 (d), 22.8 (t), 121.6 (d), 149.8 (s), 48.5 (d), 35.7 (s), 20.3 (t), 35.3 (t), 43.7 (s), 52.8 (s), 33.2 (t), 28.6 (t), 53.9 (d), 23.8 (q), 24.5 (q), 33.4 (d), 18.3 (q), 40.4 (t), 79.0 (d), 149.8 (d), 129.4 (s), 174.5 (s), 10.7 (q), 23.8 (q), 28.7 (q), 30.9 (q), 56.8 (q); <sup>1</sup>H- and <sup>13</sup>C-NMR data: same as the data reported in Tkachev *et al.* (1992).

**3-Hydroxy-9-lanosta-7, 24-dien-26, 23 R-olide (9)**,  $C_{30}H_{46}O_3$ , colorless needles; EI-MS  $m/z$  (%): 454 [M]<sup>+</sup> (6), 439 (13), 421 (100), 403 (3), 314 (20), 299 (21), 281 (14), 272 (12), 227 (20), 213 (23), 201 (15); MS and <sup>1</sup>H-NMR data: same as the data reported in Tanaka *et al.* (1991).

**Taraxerol (10)**,  $C_{30}H_{50}O$ , colorless needles; EI-MS  $m/z$  (%): 426 [M]<sup>+</sup> (7), 411 (7), 393 (2), 302 (36), 287 (40), 269 (23), 257 (14), 245 (8), 231 (11), 218 (28), 204 (100), 189 (38), 135 (52); MS data are similar to the data reported in Budzikiewicz *et al.* (1963). <sup>1</sup>H-NMR data: same as the data reported in Corbett *et al.* (1972).

**Taraxerone (11)**,  $C_{30}H_{48}O$ , colorless needles; EI-MS  $m/z$  (%): 424 [M]<sup>+</sup> (13), 409 (10), 300 (70), 285 (59), 272 (15), 257 (15), 243 (14), 231 (10), 217 (21), 204 (100), 189 (40), 133 (73); <sup>13</sup>C-NMR data (CDCl<sub>3</sub>):  $\delta$  38.4 (t), 34.2 (t), 217.5 (s), 47.5 (s), 55.8 (d), 20.0 (t), 35.1 (t), 38.9 (s), 48.7 (d), 37.6 (s), 17.5 (t), 35.8 (t), 37.7 (s), 157.6 (s), 117.2 (d), 36.7 (t), 37.7 (s), 48.8 (d), 40.7 (t), 28.8 (s), 33.6 (t), 33.1 (t), 26.1 (q), 21.5 (q), 14.8 (q), 29.9 (q), 25.6 (q), 29.8 (q), 33.4 (q), 21.4 (q); <sup>13</sup>C-NMR data: same as the data reported in Sakurai *et al.* (1987).

**5, 8-Epidioxy-(22 E, 24 R)-ergosta-6, 22-dien-3-ol (12)**,  $C_{28}H_{44}O_3$ , white needles; EI-MS  $m/z$  (%): 428 [M]<sup>+</sup> (1), 410 (3), 396 (8), 376 (5), 363 (2), 337 (2), 301 (3), 285 (5), 267 (10), 251 (20), 161 (32), 145 (15), 81 (39), 69 (100), 55 (52); <sup>1</sup>H- and <sup>13</sup>C-NMR data: same as the data reported in R secke *et al.* (2000).

**2, 2-Tetrahydro-squalene (13)**,  $C_{30}H_{54}$ , colorless oil; EI-MS  $m/z$  (%): 414 [M]<sup>+</sup> (8), 399 (1), 3290 (4), 301 (15), 275 (6), 259 (3), 233 (5), 193 (15), 83 (100); <sup>1</sup>H-NMR data (CDCl<sub>3</sub>):  $\delta$  0.87 (12H, d,  $J = 6.8$  Hz, H-1, 1, 13, 13), 1.51 (2H, m, H-2, 2), 1.14 (4H, q,  $J = 7.0$  Hz, H-3, 3), 1.37 (4H, m, H-4, 4), 1.93 (4H, t,  $J = 7.6$  Hz, H-5, 5), 5.10 (2H, t,  $J = 7.0$  Hz, H-7, 7), 2.09 (4H, q,  $J = 7.2$  Hz, H-8, 8), 2.00 (4H, m, H-9, 9), 5.15 (2H, br. s, H-11, 11), 1.60 (6H, s, H-14,

14 or 15, 15), 1.58 (6H, s, H-15, 15 or 14, 14); <sup>13</sup>C-NMR data (CDCl<sub>3</sub>): δ 22.7 (q, C-1, 1, 13, 13), 27.9 (d, C-2, 2), 38.6 (t, C-3, 3), 25.7 (t, C-4, 4), 39.9 (t, C-5, 5 or C-9, 9), 135.0 (s, C-6, 6 or C-10, 10), 124.0 (d, C-7, 7), 26.6 (t, C-8, 8), 39.8 (t, C-9, 9 or C-5, 5), 135.3 (s, C-10, 10 or C-6, 6), 124.3 (d, C-11, 11), 28.3 (t, C-12, 12), 16.0 (q, C-14, 14 or C-15, 15), 15.9 (q, C-15, 15 or C-14, 14).

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## 《云南植物研究》 征订启事

《云南植物研究》是国家科委(79)国科发条字341号文批准创办的植物学专业学报,是中国科学院主管的全国性自然科学期刊。现为我国植物科学研究发表论文的主要学术性刊物之一,并被评为“中国自然科学核心期刊”,“中国生物学类科技核心期刊”。本刊荣获中科院优秀期刊二等奖(1996)及一等奖(2000)、第二届全国优秀期刊三等奖(1997)及云南省优秀科技期刊一等奖(1997)等,并作为中国科学院首批向美国SCI推荐的刊物之一。并入选国家“双效期刊”。本刊所发表的论文在国内生物、农林、医药、轻工等二次文献刊物都有摘报;国外CA(美国化学文摘)、BA(美国生物学文摘)等从1980年起就连续摘报,还有生物科学的当代进展(CABS)、科学引文索引(SCI)的CI部分以及俄罗斯文摘杂志(P)和国际农业科技情报系统(Agris)等摘报。乌利希国际期刊指南(UIPD)从80年代就刊载本刊出版事宜。现本刊已同30多个国家和地区有发行和交换关系;在国内外同行中有一定的影响。本刊现已加中国学术期刊光盘版、中国学术期刊网及万方数据库资源系统。

本刊主要报道植物学各分支学科具有创造性或较高学术水平的研究论文和简报;植物学领域的新发现及重大应用价值的新成果;有关植物学资源开发利用和保护的创新性研究成果;植物学研究的新技术、新方法;反映本学科重要领域的国内外植物科学研究的最新进展的评述,中英文稿件均受欢迎。本刊设有植物系统学与生物地理学、植物化学与化学生物学、生物多样性保护与民族植物学、植物生态学与资源管理、植物生理与分子生物学5个专栏。

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