

## 牛尾草中一新的对映 - 贝壳杉烷型二萜

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**摘要:** 从牛尾草 [*Isodon ternifolius* (D. Don) Kudo] 的地上部分分离得到一个新的对映 - 贝壳杉烷型二萜, 命名为牛尾草素 H (1), 通过波谱方法鉴定了它的结构。此外, 还分离得到 5 个已知的对映 - 贝壳杉烷型二萜化合物: 香茶菜醛 (2), 长管香茶菜素 A, E 和 G (3-5), 开展香茶菜素 E (6), 以及木樨草素 (7), 芹菜素 (8),  $\alpha$ - 香树脂醇 (9), 乌索酸 (10) 和 2 $\alpha$ - 羟基乌索酸 (11)。

**关键词:** 牛尾草; 唇形科; 对映 - 贝壳杉烷型二萜; 牛尾草素 H

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## A New *ent*-Kauranoid from *Isodon ternifolius*

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**Abstract:** A new *ent*-kauranoid named rabdoternin H (1) was isolated from the aerial part of *Isodon ternifolius* and its structure was determined by the spectroscopic methods. Five known *ent*-kaurane diterpenoids, isodonal (2), longikaurin A, E, G (3-5) and effusanin E (6), together with luteolin (7), apigenin (8),  $\alpha$ -amyrin (9), ursolic acid (10) and 2 $\alpha$ -hydroxy-ursolic acid (11) were also reported in this paper.

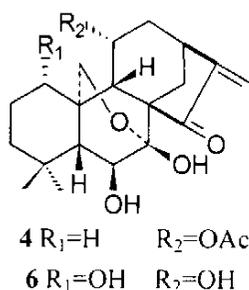
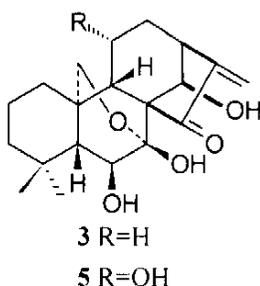
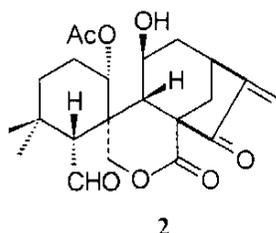
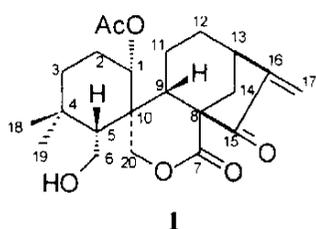
**Key words:** *Isodon ternifolius*; Labiatae; *ent*-Kauranoid; Rabdoternin H

*Isodon ternifolius* (D. Don) Kudo, a perennial herb or shrub mainly distributed in Yunnan, Guizhou, Guangdong and Guangxi Province, has been used to treat dysenteric enteritis, pharyngitis, tonsillitis etc (Wu *et al*, 1977). A series of *ent*-kaurane diterpenoids from this plant have been reported previously (Sun *et al*, 1982; Takeda *et al*, 1990; Takeda *et al*, 1994). Our re-investigation on this plant led to the isolation of a new *ent*-kaurane diterpenoid, rabdoternin H (1) and ten known compounds, isodonal (2) (Sun *et al*, 1982), longikaurin A (3) (Takeda *et al*, 1988a), longikaurin E (4) (Sun *et al*, 1982), longikaurin G (5) (Takeda *et al*, 1988b), effusanin E (6) (Wang *et al*,

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1989), luteolin (**7**) (Markham *et al*, 1978), apigenin (**8**) (Markham *et al*, 1978),  $\alpha$ -amyrin (**9**) (Mahato *et al*, 1994), ursolic acid (**10**) and 2 $\alpha$ -hydroxy-ursolic acid (**11**).

Rabdotemin H (**1**), colorless needles, showed an EIMS molecular ion peak at  $m/z$  390 in accordance with the formula  $C_{22}H_{30}O_6$ , which was confirmed by analysis of its  $^{13}C$  NMR (DEPT) spectra. It possessed an *exo*-methylene group conjugated with a carbonyl group on a five-membered ring from the following spectral data: UV  $\lambda_{max}^{MeOH}$  nm: 232.0; IR  $\nu_{max}^{KBr}$   $cm^{-1}$ : 1712 and 1648;  $^1H$  NMR:  $\delta$  5.95 and 5.33 (each 1H, brs);  $^{13}C$  NMR:  $\delta$  118.3 ( $CH_2$ ), 151.4 (C) and 202.5 (C). In addition to the above-mentioned signals, the  $^{13}C$  NMR spectrum also showed the presence of an acetoxy group, two methyl, seven methylenes (including two oxygenated ones), four methines (including one oxygen-bearing one), three quaternary carbons and a lactone carbonyl group. With consideration of the types of diterpenoids in the *Isodon* genus, these facts indicated that **1** was an *ent*-kauranoid.

There were no correlations between H-5, H-6 and C-7; H-1 and C-7 in HMBC spectrum, which indicated the basic skeleton of **1** was 6, 7-*seco*-spiro-lacton-*ent*-kauranoid. The NOE effects (H-20a with Me-19, H-5 $\beta$  with H-9 $\beta$ ) also confirmed the presumption. On the basis of  $^1H$ - $^1H$  COSY spectrum, a hydroxyl was assigned to C-6. The acetoxy was assigned to C-1, because the methine at  $\delta$  77.1 (C-1) and the correlation between H-1 and the ester carbonyl at  $\delta$  170.2 in HMBC spectrum were observed. The acetoxy group was judged to be  $\alpha$ -orientated due to the observation of NOE effects between H-1 and H-5 $\beta$ , H-11. In conclusion, rabdotemin H (**1**) was elucidated as 1 $\alpha$ -acetoxy-6-hydroxy-6, 7-*seco-ent*-kaur-16-en-15-one-7, 20-olide.

Compounds **2-9** were identified as isodonal (**2**), longikaurin A, E, G (**3-5**), effusanin E (**6**), luteolin (**7**), apigenin (**8**),  $\alpha$ -amyrin (**9**), ursolic acid (**10**) and 2 $\alpha$ -hydroxy-ursolic acid (**11**), respectively, by comparing their physical and spectral data with those reported in the literature.

## Experimental

**General** Melting point was measured on an XRC - 1 micro melting point apparatus and uncorrected. Optical rotation was taken on a SEPA - 300 polarimeter. IR spectral data was measured on a Bio-Rad FTS - 135 spectrometer with KBr pellets. UV spectra was obtained on a UV 210A spectrometer. MS spectra were recorded on a VG Auto Spec-3000 spectrometer. NMR spectra were run on a Bruker AM - 400 and a DRX - 500 instrument with TMS as internal standard.

**Extraction and Isolation** Plant material was collected in Malipo County of Yunnan Province in October, 1994, and identified as *Isodon ternifolius* (D. Don) Kudo by Prof. Zhong-Wen Lin. A voucher specimen was deposited in the Laboratory of Phytochemistry, Kunming Institute of Botany, Chinese Academy of Sciences.

The air-dried and powdered plants (8.0 kg) were extracted with 70% acetone at room temperature for 3 days each time. The extract was concentrated and filtered, and the filtrate was partitioned with petroleum-ether and EtOAc successively. The EtOAc extract (109 g) was subjected to column chromatography on a Si gel column and eluted with  $\text{CHCl}_3$  containing increasing amounts of  $\text{Me}_2\text{CO}$  system to give six fractions (I-VI). Fractions I-V were further purified by repeated column chromatography on Si gel and recrystallization to yield compounds **1** (23 mg), **2** (21 mg), **3** (37 mg), **4** (35 mg), **5** (43 mg), **6** (1.2 g), **7** (21 mg), **8** (11 mg), **9** (23 mg), **10** (5 g) and **11** (137 mg).

Table 1  $^1\text{H}$ ,  $^{13}\text{C}$  NMR,  $^1\text{H} - ^1\text{H}$  COSY and HMBC data of **1** in  $\text{C}_5\text{D}_5\text{N}$

C	$^{13}\text{C}$ NMR (125 MHz) $\delta$ (mult)	H	$^1\text{H}$ NMR (500 Hz) $\delta$ (mult, J in Hz)	COSY	HMBC (H to C)
1	77.1 (d)	$1\beta$	5.01 (m)	2	20, OAc
2	24.4 (t)	$2\alpha, \beta$	1.88 (m)	1, 3	1, 3
3	40.0 (t)	$3\alpha, \beta$	1.38 (m)	2	1, 2, 4
4	33.9 (s)	$5\beta$	1.72 (br s)	6a, 6b	4, 6
5	53.6 (d)	6a	3.83 (overlap)	5, 6b	4, 5, 10
6	58.9 (t)	6b	3.80 (overlap)	5, 6a	4, 5, 10
7	170.9 (s)	$9\beta$	3.21 (d, 13.1)	$11\alpha$	1, 5, 7, 8, 9, 10, 11, 12, 14, 15
8	58.7 (s)	$11\alpha$	1.40 (m)	9, $11\beta$	8, 9, 12
9	42.3 (d)	$11\beta$	1.85 (m)	$11\alpha$	8, 9, 10, 12, 13
10	44.4 (s)	$12\alpha$	1.99 (m)	$12\beta$ , $13\alpha$	9, 11, 13, 14, 16
11	17.9 (t)	$12\beta$	1.34 (m)	$12\alpha$	14, 16
12	30.2 (t)	$13\alpha$	2.91 (m)	$12\alpha$ , $14\beta$	8, 11, 15, 16, 17
13	35.3 (d)	$14\alpha$	2.15 (overlap)	$14\beta$	7, 8, 9, 12, 13, 15
14	29.3 (t)	$14\beta$	2.58 (dd, 4.4, 12.3)	$13\alpha$ , $14\alpha$	8, 9, 12, 13, 15, 16, 17
15	202.5 (s)	17a	5.95 (br s)	17b	13, 15, 16
16	151.4 (s)	17b	5.33 (br s)	17a	13, 15
17	118.3 (t)	Me - 18	0.99 (s)		3, 4, 5, 19
18	33.6 (q)	Me - 19	0.78 (s)		3, 4, 5, 18
19	23.6 (q)	20a	5.12 (ABd, 12.2)	20b	1, 7, 9
20	68.9 (t)	20b	4.84 (ABd, 12.2)	20a	1, 9
OAc	170.2 (s), 21.5 (q)	OAc	2.17 (s)		1

Rabdotermin H (**1**),  $\text{C}_{22}\text{H}_{30}\text{O}_6$ ; colorless needles (MeOH); mp 246 - 248 °C;  $[\alpha]_{\text{D}}^{25} + 36.3^\circ$  (c 0.903, MeOH); UV  $\lambda_{\text{max}}^{\text{MeOH}}$  nm (log  $\epsilon$ ): 232.0 (3.87); IR  $\nu_{\text{max}}^{\text{KBr}}$   $\text{cm}^{-1}$ : 3415, 2948, 1740, 1712, 1648, 1447, 1407, 1366, 1293, 1267, 1233, 1188, 1046; EI-MS (70eV)  $m/z$  (%): 390 [M]<sup>+</sup> (78), 362 (18), 348 (15), 330 [M-AcOH]<sup>+</sup> (23), 312 (20), 284 (21),

257 (30), 239 (26), 227 (31), 192 (16), 178 (31), 133 (48), 119 (40), 105 (69), 91 (100), 81 (65);  $^1\text{H}$  and  $^{13}\text{C}$  NMR data see Table 1.

Isodonol (**2**),  $\text{C}_{22}\text{H}_{28}\text{O}_7$ ; colorless needles (MeOH); EI-MS (70eV)  $m/z$  (%): 404 [M]<sup>+</sup> (25), 386 [M-H<sub>2</sub>O]<sup>+</sup> (9), 344 [M-AcOH]<sup>+</sup> (100), 326 [M-AcOH-H<sub>2</sub>O]<sup>+</sup> (12), 316 (28), 298 (23), 270 (20), 245 (87), 227 (57) 217 (52), 149 (67), 81 (70);  $^1\text{H}$  NMR (500 MHz,  $\text{C}_5\text{D}_5\text{N}$ )  $\delta$ : 10.01 (1H, d,  $J = 1.8$  Hz, CHO), 6.03 and 5.40 (each 1H, s, H<sub>2</sub>-17), 5.51 (1H, m, H-1 $\beta$ ), 5.44 and 5.22 (each 1H, ABd,  $J = 12.4$  Hz, H<sub>2</sub>-20), 4.41 (1H, m, H-11 $\alpha$ ), 2.93 (1H, d,  $J = 4.5$  Hz, H-5 $\beta$ ), 2.15 (3H, s, OAc), 0.98 and 0.95 (each 3H, s, 2  $\times$  Me);  $^{13}\text{C}$  NMR (125 MHz,  $\text{C}_5\text{D}_5\text{N}$ )  $\delta$ : 204.9 (d, C-6), 200.8 (s, C-15), 170.3 (s, C-7), 150.6 (s, C-16), 119.3 (t, C-17), 76.0 (d, C-1), 67.1 (t, C-20), 65.2 (d, C-11), 61.2 (d, C-5), 58.5 (s, C-8), 46.8 (d, C-9), 44.6 (s, C-10), 41.4 (t, C-12), 40.2 (t, C-3), 34.6 (d, C-13), 34.5 (s, C-4), 33.2 (q, C-18), 29.9 (t, C-14), 24.5 (q, C-19), 24.4 (t, C-2), OAc: 170.3, s, 21.4, q.

Longikaurin A (**3**),  $\text{C}_{20}\text{H}_{28}\text{O}_5$ ; colorless needles (MeOH); EI-MS (70eV)  $m/z$  (%): 348 [M]<sup>+</sup> (83), 330 [M-H<sub>2</sub>O]<sup>+</sup> (35), 319 (16), 302 (45), 284 (22), 269 (20), 217 (39), 177 (36), 167 (60), 151 (83), 133 (43), 109 (58), 85 (68);  $^1\text{H}$  NMR (500 MHz,  $\text{C}_5\text{D}_5\text{N}$ )  $\delta$ : 6.86 (1H, d,  $J = 10.0$  Hz, OH-6 $\beta$ ), 6.26 and 5.50 (each 1H, s, H<sub>2</sub>-17), 5.11 (1H, s, H-14 $\alpha$ ), 4.16 (1H, dd,  $J = 10.0, 6.3$  Hz, H-6 $\alpha$ ), 4.13 and 3.93 (each 1H, ABd,  $J = 10.0$  Hz, H<sub>2</sub>-20), 3.15 (1H, d,  $J = 9.5$  Hz, H-13 $\alpha$ ), 1.23 and 1.04 (each 3H, s, 2  $\times$  Me);  $^{13}\text{C}$  NMR (125 MHz,  $\text{C}_5\text{D}_5\text{N}$ )  $\delta$ : 208.8 (s, C-15), 153.0 (s, C-16), 119.6 (t, C-17), 98.4 (s, C-7), 74.2 (d, C-6), 73.6 (d, C-14), 66.3 (t, C-20), 62.7 (s, C-8), 60.8 (d, C-9), 52.5 (d, C-5), 43.9 (d, C-13), 41.5 (t, C-3), 36.5 (s, C-10), 34.0 (s, C-4), 33.7 (q, C-18), 30.7 (t, C-1), 30.2 (t, C-12), 22.4 (q, C-19), 19.0 (t, C-11), 16.7 (t, C-2).

Longikaurin E (**4**),  $\text{C}_{22}\text{H}_{30}\text{O}_6$ ; colorless needles (MeOH); EI-MS (70eV)  $m/z$  (%): 390 [M]<sup>+</sup> (100), 372 [M-H<sub>2</sub>O]<sup>+</sup> (4), 330 [M-AcOH]<sup>+</sup> (35), 312 [M-AcOH-H<sub>2</sub>O]<sup>+</sup> (30), 284 (27), 269 (18), 255 (13), 227 (10), 213 (14), 200 (16), 179 (16), 151 (35), 120 (24);  $^1\text{H}$  NMR (500 MHz,  $\text{C}_5\text{D}_5\text{N}$ )  $\delta$ : 6.61 (1H, d,  $J = 11.0$  Hz, OH-6 $\beta$ ), 5.99 and 5.30 (each 1H, s, H<sub>2</sub>-17), 5.43 (1H, t,  $J = 4.5$  Hz, H-11 $\beta$ ), 4.40 and 4.22 (each 1H, ABd,  $J = 9.2$  Hz, H<sub>2</sub>-20), 4.30 (1H, dd,  $J = 11.0, 7.5$  Hz, H-6 $\alpha$ ), 2.07 (3H, s, OAc), 1.28 and 1.06 (each 3H, s, 2  $\times$  Me);  $^{13}\text{C}$  NMR (125 MHz,  $\text{C}_5\text{D}_5\text{N}$ )  $\delta$ : 209.7 (s, C-15), 153.1 (s, C-16), 117.2 (t, C-17), 96.2 (s, C-7), 75.0 (d, C-6), 68.8 (d, C-11), 68.7 (t, C-20), 60.1 (d, C-9), 59.2 (s, C-8), 53.4 (d, C-5), 41.7 (t, C-3), 38.0 (t, C-12), 37.2 (s, C-10), 34.5 (t, C-1), 34.2 (q, C-18), 33.9 (s, C-4), 31.2 (d, C-13), 27.7 (t, C-14), 22.8 (q, C-19), 18.8 (t, C-2), OAc: 169.8, s, 21.6, q.

Longikaurin G (**5**),  $\text{C}_{20}\text{H}_{28}\text{O}_6$ ; colorless needles (MeOH); EI-MS (70eV)  $m/z$  (%): 364

[ M ]<sup>+</sup> ( 68 ), 346 [ M-H<sub>2</sub>O ]<sup>+</sup> ( 51 ), 328 ( 12 ), 315 ( 31 ), 300 ( 17 ), 269 ( 10 ), 215 ( 17 ), 175 ( 24 ), 167 ( 42 ), 151 ( 69 ), 136 ( 36 ), 123 ( 43 ), 109 ( 57 ), 85 ( 100 ), 69 ( 74 ); <sup>1</sup>H NMR ( 500 MHz, C<sub>5</sub>D<sub>5</sub>N ) δ : 6.79 ( 1H, d, J = 11.0 Hz, OH - 6β ), 6.39 ( 1H, s, H - 14α ), 6.27 and 5.50 ( each 1H, s, H<sub>2</sub> - 17 ), 5.16 and 4.29 ( 1H, ABd, J = 8.6 Hz, H<sub>2</sub> - 20 ), 4.40 ( 1H, m, H - 11β ), 4.31 ( 1H, dd, J = 7.5, 11.0 Hz, H - 6α ), 1.40 ( 1H, d, J = 7.5 Hz, H - 5β ), 1.32 and 1.11 ( each 3H, s, 2 × Me ); <sup>13</sup>C NMR ( 125 MHz, C<sub>5</sub>D<sub>5</sub>N ) δ : 209.4 ( s, C - 15 ), 153.5 ( s, C - 16 ), 118.7 ( t, C - 17 ), 98.9 ( s, C - 7 ), 75.1 ( d, C - 14 ), 72.5 ( d, C - 6 ), 69.2 ( t, C - 20 ), 65.2 ( d, C - 11 ), 63.0 ( s, C - 8 ), 60.0 ( d, C - 9 ), 56.4 ( d, C - 5 ), 43.5 ( d, C - 13 ), 42.7 ( t, C - 12 ), 41.5 ( t, C - 3 ), 37.6 ( s, C - 10 ), 34.6 ( q, C - 18 ), 34.0 ( s, C - 4 ), 31.2 ( t, C - 1 ), 23.0 ( q, C - 19 ), 19.0 ( t, C - 2 ).

Effusanin E ( **6** ), C<sub>20</sub>H<sub>28</sub>O<sub>6</sub>; colorless needles ( MeOH ); EI-MS ( 70eV ) *m/z* ( % ): 364 [ M ]<sup>+</sup> ( 76 ), 346 [ M-H<sub>2</sub>O ]<sup>+</sup> ( 8 ), 300 ( 8 ), 285 ( 10 ), 267 ( 9 ), 259 ( 10 ), 229 ( 12 ), 192 ( 16 ), 179 ( 18 ), 161 ( 31 ), 149 ( 24 ), 135 ( 30 ), 121 ( 42 ), 107 ( 45 ), 95 ( 50 ), 85 ( 75 ); <sup>1</sup>H NMR ( 500 MHz, C<sub>5</sub>D<sub>5</sub>N ) δ : 6.88 ( 1H, d, J = 11.0 Hz, OH - 6β ), 5.95 and 5.28 ( each 1H, s, H<sub>2</sub> - 17 ), 5.18 and 4.37 ( each 1H, ABd, J = 9.4 Hz, H<sub>2</sub> - 20 ), 4.57 ( 1H, br s, H - 11β ), 4.33 ( 1H, dd, J = 11.0, 7.0 Hz, H - 6α ), 3.87 ( 1H, dd, J = 10.0, 6.2 Hz, H - 1β ), 3.68 ( 1H, d, J = 11.6 Hz, H - 14α ), 1.31 and 1.12 ( each 3H, s, 2 × Me ); <sup>13</sup>C NMR ( 125 MHz, C<sub>5</sub>D<sub>5</sub>N ) δ : 211.4 ( s, C - 15 ), 154.5 ( s, C - 16 ), 115.3 ( t, C - 17 ), 96.3 ( s, C - 7 ), 75.4 ( d, C - 1 ), 73.5 ( d, C - 6 ), 67.0 ( d, C - 11 ), 65.7 ( t, C - 20 ), 60.9 ( d, C - 5 ), 60.0 ( s, C - 8 ), 55.1 ( d, C - 9 ), 43.1 ( s, C - 10 ), 39.4 ( t, C - 3 and 12 ), 34.9 ( q, C - 18 ), 34.0 ( s, C - 4 ), 33.8 ( d, C - 13 ), 29.4 ( t, C - 2 ), 27.1 ( t, C - 14 ), 22.5 ( q, C - 19 ).

Luteolin ( **7** ), C<sub>15</sub>H<sub>10</sub>O<sub>6</sub>; yellow powder; EI-MS ( 70eV ) *m/z* ( % ): 286 [ M ]<sup>+</sup> ( 100 ), 258 ( 20 ), 229 ( 10 ), 153 [ A<sub>1</sub> + 1 ]<sup>+</sup> ( 31 ), 134 [ B<sub>1</sub> ]<sup>+</sup> ( 16 ), 69 ( 15 ). Its <sup>1</sup>H and <sup>13</sup>C NMR data are consistent with those of luteolin reported in the literature ( Markham *et al*, 1978 ).

Apigenin ( **8** ), C<sub>15</sub>H<sub>10</sub>O<sub>5</sub>; yellow powder; EI-MS ( 70eV ) *m/z* ( % ): 270 [ M ]<sup>+</sup> ( 100 ), 242 ( 31 ), 213 ( 7 ), 153 [ A<sub>1</sub> + 1 ]<sup>+</sup> ( 31 ), 121 [ B<sub>1</sub> ]<sup>+</sup> ( 35 ), 96 ( 11 ), 69 ( 26 ). Its <sup>1</sup>H and <sup>13</sup>C NMR data are consistent with those of apigenin reported in the literature ( Markham *et al*, 1978 ).

α-Amyrin ( **9** ), C<sub>30</sub>H<sub>50</sub>O; white powder; EI-MS ( 70eV ) *m/z* ( % ): 426 [ M ]<sup>+</sup> ( 18 ), 411 ( 4 ), 218 ( 100 ), 203 ( 27 ), 189 ( 16 ), 161 ( 9 ), 149 ( 20 ), 135 ( 21 ), 81 ( 24 ), 69 ( 33 ), 55 ( 37 ). Its <sup>1</sup>H and <sup>13</sup>C NMR data are consistent with those of α-amyrin reported in the literature ( Mahato *et al*, 1994 ).

Ursolic acid ( **10** ), C<sub>30</sub>H<sub>48</sub>O<sub>3</sub>; white powder; EI-MS ( 70eV ) *m/z* data and R<sub>f</sub> value on TLC are consistent with those of authentic sample.

2α-Hydroxy-ursolic acid ( **11** ), C<sub>30</sub>H<sub>48</sub>O<sub>4</sub>; white powder; EI-MS ( 70eV ) *m/z* data and R<sub>f</sub> value on TLC are consistent with those of authentic sample.

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