印度红树植物 *Xylocarpus moluccensis* 种子中柠檬苦素成分研究

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摘　要：目的　对印度木果楝属红树植物 *Xylocarpus moluccensis* 种子中的柠檬苦素成分进行研究。**方法**　利用正、反相硅胶柱以及高效液相等色谱手段进行分离制备，并利用核磁共振波谱和离子阱电喷雾质谱鉴定化合物结构。**结果**　从 *X. moluccensis* 种子中共分离得到 18 个柠檬苦素类化合物，包括 14 个 mexicanolides、2 个 gedunins、1 个 andirobine 和 1 个原柠檬苦素；分别鉴定为 fissinolide（**1**）、3-propanoylproceranolide（**2**）、3β-hydroxyangustidienolide（**3**）、3β-acetoxy-6-deoxy-swietenine（**4**）、febrifugin（**5**）、granatumin I（**6**）、3-de (2-methylbutanoyl)-3-propanoylcipadesin（**7**）、granatumin H（**8**）、tigloylseneganolide A（**9**）、ruageanin A（**10**）、swietemahonolide（**11**）、khayasin T（**12**）、andhraxylocarpin D（**13**）、granatumin D（**14**）、7-oxo-7-deacetoxygenunin（**15**）、gedunin（**16**）、methyl angolensate（**17**）、20,21,22,23-tetrahydro-23-oxoazadirone（**18**）。**结论**　化合物 **2** 为新化合物，命名为 3-propanoylproceranolide；化合物 **3** 为新天然产物。

关键词：红树植物；木果楝属；*Xylocarpus moluccensis* (Lamk) M. Roem.；柠檬苦素；3-propanoylproceranolide

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Limonoids from Indian mangrove, seeds of *Xylocarpus moluccensis*

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Abstract: Objective　To investigate limonoids in the seeds of an Indian mangrove, *Xylocarpus moluccensis*. **Methods**　Compounds were isolated and purified by silica gel, ODS gel column chromatographies, and HPLC. The structures of the obtained limonoids were identified by NMR spectra and ESI-MS. **Results**　Eighteen limonoids, such as fissinolide (**1**), 3-propanoylproceranolide (**2**), 3β-hydroxyangustidienolide (**3**), 3β-acetoxy-6-deoxy-swietenine (**4**), febrifugin (**5**), granatumin I (**6**), 3-de(2-methylbutanoyl)-3-propanoylcipadesin (**7**), granatumin H (**8**), tigloylseneganolide A (**9**), ruageanin A (**10**), swietemahonolide (**11**), khayasin T (**12**), andhraxylocarpin D (**13**), granatumin D (**14**), 7-oxo-7-deacetoxygenunin (**15**), gedunin (**16**), methyl angolensate (**17**), and 20,21,22,23-tetrahydro-23-oxoazadirone (**18**), were isolated and identified. **Conclusion**　3-propanoylproceranolide (**2**) is a new compound, and compound **3** is a new natural product.

Key words: mangrove; *Xylocarpus* Koenig; Xy*locarpus moluccensis* (Lamk) M. Roem.; limonoids; 3-propanoylproceranolide

红树植物是一类生长在热带、亚热带海洋潮间带的耐盐植物群落。全球现有红树植物 84 种，可分为真红树和半红树两大类。其中真红树植物 70 种，半红树植物 14 种[1]。全球属于楝科的红树植物仅有真红树植物木果楝一属。据文献报道，木果楝属红树植物的主要次生代谢产物为柠檬苦素。柠檬苦素是一类高度氧化的四降三萜类化合物，主要存在于楝科、芸香科、苦木科和叶柄花科等植物中。该类化合物是由具有 4,4,8-三甲基-17-呋喃甾体骨架的前体经过一系列的氧化重排衍生而来的。柠檬苦素主要有拒食、杀虫、抗菌、抗疟和抗癌等多种生物活性[2-4]。迄今，从木果楝属红树植物中共分离鉴定了 213 个柠檬苦素类化合物，包括 106 个 mexicanolides、88 个 phragmalins、4 个 andirobins、4 个 gendunins、1 个 obacunol[5-6]。本实验对印度木果楝属红树植物 *Xylocarpus moluccensis* (Lamk) M.

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Roem. 种子中的柠檬苦素成分进行了研究，共分离鉴定了 18 个柠檬苦素化合物，包括 14 个 mexicanolides、2 个 gedunins、1 个 andirobine 和 1 个原柠檬苦素，分别为 fissinolide（**1**）、3-propanoyl-proceranolide（**2**）、3β-hydroxyl- angustidienolide（**3**）、3β-acetoxy-6-deoxy-swietenine（**4**）、febrifugin（**5**）、granatumin I（**6**）、3-de (2-methylbutanoyl)-3-propanoylcipadesin（**7**）、granatumin H（**8**）、tigloylseneganolide A（**9**）、ruageanin A（**10**）、swietemahonolide（**11**）、khayasin T（**12**）、andhraxylocarpin D（**13**）、granatumin D（**14**）、7-oxo-7-deacetoxygenunin（**15**）、gedunin（**16**）、methyl angolensate（**17**）、20,21,22,23-tetrahydro-23-oxoazadirone（**18**）。其中化合物 **2** 为新化合物，化合物 **3** 为新天然产物。

1 仪器与材料

Waters 2535Q 半制备高效液相色谱仪（美国 Waters 公司），Waters 2489 双通道紫外可见光检测器（美国 Waters 公司），Bruker AVANCE III 400 型核磁共振波谱仪（瑞士 Bruker 公司），AmaZon SL 离子阱电喷雾质谱仪（德国 Bruker Daltonics 公司）。正相硅胶（100～200 目）为青岛海洋化工厂产品，反相硅胶（C_{18}）为日本 YMC 公司产品，乙腈、甲醇为色谱纯（德国 Merck 公司），其他试剂均为分析纯。

Xylocarpus moluccensis (Lamk) M. Roem. 种子于 2007 年 9 月采集于印度安德拉邦哥达瓦里河红树林湿地。由 Tirumani Satyanandamurty（Government Degree College at Amadala valasa，Srikakulam District，Andhra Pradesh，India）鉴定。样品标本（No. Indian XM-03）保存于暨南大学药学院海洋药物研究中心。

2 提取与分离

干燥的 *X. moluccensis* 种子 8.7 kg，粉碎后用 95%的乙醇室温下浸提 5 次，每次浸泡 48 h。提取液合并减压浓缩得到粗浸膏 750 g。粗浸膏加水混悬，继而用醋酸乙酯萃取 5 次，减压浓缩得醋酸乙酯萃取部分 198 g。醋酸乙酯萃取物（198 g）经正相硅胶（100～200 目）柱色谱，氯仿-甲醇（100∶0→5∶1）梯度洗脱，共得到 127 个流分。通过薄层色谱和高效液相色谱等手段分析后合并 47～62 流分（66.2 g），经反相硅胶柱色谱，乙腈-水（50∶50→100∶0）梯度洗脱，得到 132 个流分。其中流分 21 经 HPLC 分离制备得到化合物 **1**（255.5 mg）、**3**（1.5 mg）、**4**（73.5 mg）、**5**（20.1 mg）、**15**（13.7 mg）、**17**（38.0 mg）。流分 28 经 HPLC 分离制备得到化合物 **2**（0.6 mg）、**6**（1.5 mg）、**7**（2.2 mg）、**8**（7.4 mg）、**9**（1.8 mg）、**10**（3.4 mg）、**11**（4.1 mg）、**12**（1.8 mg）、**13**（3.8 mg）、**14**（1.1 mg）、**16**（12.8 mg）、**18**（3.0 mg）。

3 结构鉴定

化合物 **1**：白色无定形粉末。ESI-MS *m*/*z*: 535.04 $[M+Na]^+$，分子式为 $C_{29}H_{36}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) δ: 3.17 (1H, m, H-2), 5.01 (1H, d, J = 10.0 Hz, H-3), 3.23 (1H, dd, J = 8.4, 4.0 Hz, H-5), 2.39 (1H, overlap, H-6a), 2.38 (1H, overlap, H-6b), 2.07 (1H, m, H-9), 1.79 (1H, overlap, H-11α), 1.80 (1H, overlap, H-11β), 1.13 (1H, m, H-12α), 1.81 (1H, overlap, H-12β), 3.47 (1H, dt, J = 20.8, 2.8 Hz, H-15α), 3.79 (1H, d, J = 20.8 Hz, H-15β), 5.71 (1H, s, H-17), 1.09 (3H, s, H-18), 1.17 (3H, s, H-19), 7.58 (1H, brs, H-21), 6.50 (1H, brs, H-22), 7.43 (1H, brs, H-23), 0.82 (3H, s, H-28), 0.74 (3H, s, H-29), 2.15 (1H, dd, J = 15.2, 5.6 Hz, H-30α), 2.84 (1H, dd, J = 15.2, 2.5 Hz, H-30β), 3.73 (3H, s, 7-OCH_3), 2.20 (3H, s, H-2′)；^{13}C-NMR (100 MHz, $CDCl_3$) δ: 218.0 (C-1), 52.3 (C-2), 78.4 (C-3), 38.3 (C-4), 48.0 (C-5), 33.4 (C-6), 174.3 (C-7), 131.8 (C-8), 40.9 (C-9), 53.0 (C-10), 18.8 (C-11), 29.2 (C-12), 38.1 (C-13), 127.8 (C-14), 33.5 (C-15), 170.4 (C-16), 80.7 (C-17), 21.3 (C-18), 16.8 (C-19), 120.6 (C-20), 141.8 (C-21), 110.0 (C-22), 142.9 (C-23), 23.2 (C-28), 18.0 (C-29), 33.4 (C-30), 52.1 (7-OCH_3), 169.9 (C-1′), 20.5 (C-2′)。以上数据与文献报道一致[6]，故鉴定化合物 **1** 为 fissinolide。

化合物 **2**：白色无定形粉末。$[\alpha]_D^{25}$ −90° (*c* 0.043, MeOH)。UV λ_{max}^{MeOH} (nm): 212。ESI-MS *m*/*z*: 549.08 $[M+Na]^+$；HR-ESI-MS *m*/*z*: 549.246 7 $[M+Na]^+$（$C_{30}H_{38}NaO_8^+$，计算值为 549.246 4），确定其分子式为 $C_{30}H_{38}O_8$，由此可见该化合物的不饱和度为 12。NMR 数据（表 1）表明其中 7 个不饱和度来源于 4 个羰基（包括 1 个酮羰基和 3 个酯羰基）和 3 个碳碳双键，因此，该化合物的化学结构中应有 5 个环系。由 DEPT 谱可知该化合物含有 6 个甲基（1 个甲氧基、4 个柠檬苦素母核上的甲基和 1 个取代基上的甲基），6 个亚甲基，8 个次甲基（其中，3 个为烯碳次甲基，且 2 个与 O 相连）和 10 个季碳（包括 1 个酮羰基、3 个酯羰基、3 个烯碳季碳）。化

合物 **2** 的 NMR 数据（表 1）与 fissinolide（**1**）类似，两者的区别仅在于 C-3 位的取代基不同。Fissinolide 的 C-3 位为乙酰氧基取代，而化合物 **2** 则被丙酰氧基 [δ_H 2.47 (2H, q, J = 7.6 Hz, H-2′), 1.22 (3H, t, J = 8.4 Hz, H-3′)；δ_C 173.9 (C-1′), 28.0 (C-2′), 9.4 (C-3′)] 取代。^{1}H-^{1}H COSY 谱上 H-2′与 H-3′的相关以及 HMBC 谱上 H-2′与 C-1′、H-3′与 C-1′相关证明了以上推断。H-3 与 C-1′的 HMBC 远程相关也进一步证实丙酰氧基连在 C-3 上（表 1、图 1）。H-3 与 H-29 之间明显的 NOE 相关信号表明 H-3 为 α 构型，而与 C-3 相连的丙酰氧基则为 β 构型。H-3/H-2、H-9/H-18、H-9/H-19 的 NOE 相关信号表明 H-2、H-9、H-18、H-19 为 α 构型，而 H-17 与 H-11β、H-12β 之间的 NOE 相关信号证实 H-17 为 β 构型，由以上数据可推断化合物 **2** 与化合物 **1** 的相对立体构型相同。化合物 **2** 的比旋光度与化合物 **1**（$[\alpha]_D^{25}$ −57°）[6] 同为负值，亦佐证了上述推断。因此，化合物 **2** 被鉴定为 1 个新化合物，命名为 3-propanoyl-proceranolide。

化合物 **3**：白色无定形粉末，易溶于氯仿、丙

表 1　化合物 2 的 ^{1}H/^{13}C-NMR (400/100 MHz, $CDCl_3$) 数据、^{1}H-^{1}H COSY 相关和 HMBC 相关

Table 1　^{1}H/^{13}C-NMR (400/100 MHz, $CDCl_3$) data, ^{1}H-^{1}H COSY and HMBC correlations for compound 2

碳位	δ_H	δ_C	^{1}H-^{1}H COSY	HMBC
1		218.1		
2	3.15 (1H, m)	48.1	3, 30	
3	5.01 (1H, d, J = 10.0 Hz)	78.2	2	C-4, 5, 1′
4		38.4		
5	3.24 (1H, dd, J = 8.8, 3.6 Hz)	40.4	6	
6	2.39 (1H, s)	33.3	5, 6b	
	2.38 (1H, d, J = 6.0 Hz)		5, 6a	
7		174.3		
8		131.8		
9	2.07 1H, (m)	52.1	11	
10		53.0		
11	1.79 (2H, m)	18.8	9, 12	
12	1.13 (1H, m)	29.1	11, 12β	
	1.78 (1H, m)		11, 12α	
13		38.1		
14		127.8		
15	3.46 (1H, dt, J = 20.8, 2.8 Hz), 3.78 (1H, overlapped)	33.5	15β, 15α	
16		170.3		
17	5.70 (1H, s)	80.7		C-18, 20, 21, 22
18	1.09 (3H, s)	17.4		C-12, 14, 17
19	1.18 (3H, s)	16.7		C-1, 5, 9, 10
20		120.6		
21	7.58 (1H, brs)	141.8	22, 23	C-17, 21, 23
22	6.50 (1H, brs)	110.0	21, 23	C-21, 23
23	7.43 (1H, brs)	142.9	21, 22	
28	0.83 (3H, s)	23.2		C-3, 4, 5, 29
29	0.74 (3H, s)	20.5		C-3, 4, 5, 28
30	2.15 (1H, overlapped)	33.4	2, 30β	C-1, 8
	2.84 (1H, dd, J = 15.2, 2.0 Hz)		2, 30α	
7-OCH_3	3.73 (3H, s)	52.1		
1′		173.9		
2′	2.47 (2H, q, J = 7.6 Hz)	28.0	3′	C-1′, 3′
3′	1.22 (3H, t, J = 7.6 Hz)	9.4	2′	C-1′, 2′

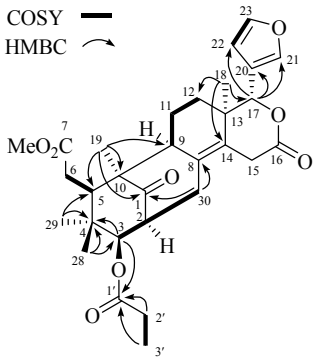

图 1 化合物 2 的主要 ^{1}H-^{1}H COSY 和 HMBC 相关

Fig. 1 Key ^{1}H-^{1}H COSY and HMBC correlations of compound 2

酮。ESI-MS *m*/*z*: 491.10 [M＋Na]$^+$，分子式为 $C_{27}H_{32}O_7$。^{1}H-NMR (400 MHz, $CDCl_3$) 数据与化合物 angustidienolide 类似，两者的区别仅在于 C-3 位的取代基不同，angustidienolide 的 C-3 位为乙酰氧基取代，而化合物 **3** 中 C-3 位由羟基取代。该化合物结构已见文献报道[7]，是由 angustidienolide C-3 位酯键水解得到的人工产物，水解后 C-3 位的质子信号由 δ 4.91 (1H, d, *J* = 9.0 Hz) 迁移到了 δ 4.12 (1H, d, *J* = 7.5 Hz)，本实验中 C-3 位的质子信号现于 δ 3.91 (1H, d, *J* = 6.8 Hz)。^{1}H-NMR (400 MHz, $CDCl_3$) δ: 3.04 (1H, t, *J* = 6.4 Hz, H-2), 3.91 (1H, d, *J* = 6.8 Hz, H-3), 2.84 (1H, t, *J* = 5.6 Hz, H-5), 2.27 (1H, overlap, H-6a), 2.27 (1H, overlap, H-6b), 2.26 (1H, m, H-11α), 2.56 (1H, m, H-11β), 1.49 (1H, m, H-12α), 1.63 (1H, m, H-12β), 5.90 (1H, s, H-15), 5.13 (1H, s, H-17), 1.11 (3H, s, H-18), 1.18 (3H, s, H-19), 7.52 (1H, brs, H-21), 6.47 (1H, brs, H-22), 7.46 (1H, brs, H-23), 1.00 (3H, s, H-28), 1.02 (3H, s, H-29), 2.51 (1H, m, H-30α), 3.25 (1H, d, *J* = 16.8 Hz, H-30β), 3.71 (3H, s, 7-OCH_3)。根据以上数据鉴定化合物 **3** 为 3β-hydroxyangustidienolide。

化合物 **4**：白色无定形粉末。ESI-MS *m*/*z*: 535.09 [M＋Na]$^+$，分子式为 $C_{29}H_{36}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) δ: 3.52 (1H, dd, *J* = 8.2, 8.0 Hz, H-2), 4.76 (1H, d, *J* = 9.2 Hz, H-3), 3.38 (1H, dd, *J* = 7.6, 4.4 Hz, H-5), 2.38 (1H, s, H-6a), 2.39 (1H, d, *J* = 3.4 Hz, H-6b), 2.25 (1H, m, H-9), 1.67 (1H, m, H-11α), 2.14 (1H, m, H-11β), 1.43 (1H, m, H-12α), 1.67 (1H, m, H-12β), 2.25 (1H, m, H-14), 2.95 (1H, dd, *J* = 18.4, 5.6 Hz, H-15α), 2.86 (1H, d, *J* = 18.4 Hz, H-15β), 5.73 (1H, s, H-17), 1.10 (3H, s, H-18), 1.16 (3H, s, H-19), 7.82 (1H, brs, H-21), 6.48 (1H, brs, H-22), 7.44 (1H, brs, H-23), 0.84 (3H, s, H-28), 0.81 (3H, s, H-29), 5.37 (1H, d, *J* = 7.2 Hz, H-30), 3.74 (3H, s, 7-OCH_3), 2.11 (3H, s, H-2′)；^{13}C-NMR (100 MHz, $CDCl_3$) δ: 217.0 (C-1), 48.4 (C-2), 77.5 (C-3), 38.1 (C-4), 41.6 (C-5), 33.6 (C-6), 174.1 (C-7), 138.4 (C-8), 56.4 (C-9), 49.9 (C-10), 20.5 (C-11), 34.4 (C-12), 36.8 (C-13), 45.2 (C-14), 45.2 (C-15), 169.6 (C-16), 77.4 (C-17), 22.1 (C-18), 15.9 (C-19), 120.7 (C-20), 141.9 (C-21), 109.8 (C-22), 142.9 (C-23), 22.5 (C-28), 20. 4 (C-29), 120.7 (C-30), 52.2 (7-OCH_3), 170.9 (C-1′), 20.4 (C-2′)。以上数据与文献报道一致[8]，故鉴定化合物 **4** 为 3β-acetoxy-6-deoxy-swietenine。

化合物 **5**：白色无定形粉末。ESI-MS *m*/*z*: 575.10 [M＋Na]$^+$，分子式为 $C_{32}H_{40}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) δ: 3.55 (1H, dd, *J* = 8.8, 7.6 Hz, H-2), 4.85 (1H, d, *J* = 9.2 Hz, H-3), 3.48 (1H, dd, *J* = 8.8, 3.2 Hz, H-5), 2.39 (2H, m, H-6), 2.21 (1H, m, H-9), 1.65 (1H, m, H-11α), 1.65 (1H, m, H-11β), 2.10 (1H, m, H-12α), 1.42 (1H, m, H-12β), 2.25 (1H, overlap, H-14), 2.87 (1H, dd, *J* = 18.8, 6.4 Hz, H-15α), 2.83 (1H, d, *J* = 18.8 Hz, H-15β), 5.65 (1H, s, H-17), 1.10 (3H, s, H-18), 1.17 (3H, s, H-19), 7.85 (1H, brs, H-21), 6.49 (1H, brs, H-22), 7.44 (1H, brs, H-23), 0.83 (3H, s, H-28), 0.86 (3H, s, H-29), 5.34 (1H, d, *J* = 7.2 Hz, H-30), 3.74 (3H, s, 7-OCH_3), 6.94 (1H, q, *J* = 7.2 Hz, H-3′), 1.75 (3H, d, *J* = 7.2 Hz, H-4′), 1.84 (3H, s, H-5′)；^{13}C-NMR (100 MHz, $CDCl_3$) δ: 217.3 (C-1), 48.8 (C-2), 77.1 (C-3), 38.6 (C-4), 41.3 (C-5), 32.9 (C-6), 174.1 (C-7), 138.5 (C-8), 56.8 (C-9), 49.8 (C-10), 20.7 (C-11), 34.5 (C-12), 37.0 (C-13), 45.2 (C-14), 30.0 (C-15), 168.9 (C-16), 76.6 (C-17), 21.7 (C-18), 15.8 (C-19), 120.8 (C-20), 141.9 (C-21), 109.7 (C-22), 143.0 (C-23), 22.7 (C-28), 20.2 (C-29), 123.1 (C-30), 52.2 (7-OCH_3), 167.1 (C-1′), 127.5 (C-2′), 139.6 (C-3′), 14.1 (C-4′), 11.8 (C-5′)。以上数据和文献报道一致[9]，故鉴定化合物 **5** 为 febrifugin。

化合物 **6**：白色无定形粉末。ESI-MS *m*/*z*: 561.09 [M＋Na]$^+$，分子式为 $C_{31}H_{38}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) δ: 3.56 (1H, dd, *J* = 8.8, 7.2 Hz, H-2), 4.85 (1H, d, *J* = 9.2 Hz, H-3), 3.50 (1H, dd, *J* = 8.8, 3.2 Hz, H-5), 2.40 (1H, brs, H-6a), 2.42 (1H, d, *J* = 9.6 Hz, H-6b), 2.24 (1H, m, H-9), 1.66 (1H, m, H-11α), 2.10

(1H, m, H-11β), 1.42 (1H, m, H-12α), 1.66 (1H, m, H-12β), 2.24 (1H, m, H-14), 2.90 (1H, dd, *J* = 19.2, 6.4 Hz, H-15α), 2.78 (1H, d, *J* = 17.6 Hz, H-15β), 5.66 (1H, s, H-17), 1.11 (3H, s, H-18), 1.18 (3H, s, H-19), 7.82 (1H, brs, H-21), 6.48 (1H, brs, H-22), 7.44 (1H, brs, H-23), 0.84 (3H, s, H-28), 0.87 (3H, s, H-29), 5.37 (1H, d, *J* = 6.8 Hz, H-30), 3.74 (3H, s, 7-OCH_3), 5.62 (1H, brs, H-3′), 6.20 (1H, s, H-3′), 1.97 (3H, s, H-4′)。以上数据与文献报道一致[10]，故鉴定化合物 **6** 为 granatumin I。

化合物 **7**：白色无定形粉末。ESI-MS *m*/*z*: 549.09 $[M+Na]^+$，分子式为 $C_{30}H_{38}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.53 (1H, dd, *J* = 8.4, 7.2 Hz, H-2), 4.78 (1H, d, *J* = 9.2 Hz, H-3), 3.41 (1H, dd, *J* = 7.6, 4.0 Hz, H-5), 2.43 (1H, d, *J* = 7.6 Hz, H-6a), 2.39 (1H, d, *J* = 5.2 Hz, H-6b), 2.25 (1H, m, H-9), 1.67 (1H, m, H-11α), 2.16 (1H, m, H-11β), 1.43 (1H, m, H-12α), 1.67 (1H, m, H-12β), 2.25 (1H, m, H-14), 2.92 (1H, dd, *J* = 18.4, 6.0 Hz, H-15α), 2.81 (1H, d, *J* = 17.6 Hz, H-15β), 5.72 (1H, s, H-17), 1.11 (3H, s, H-18), 1.17 (3H, s, H-19), 7.82 (1H, brs, H-21), 6.49 (1H, brs, H-22), 7.44 (1H, brs, H-23), 0.84 (3H, s, H-28), 0.81 (3H, s, H-29), 5.38 (1H, d, *J* = 6.8 Hz, H-30), 3.74 (3H, s, 7-OCH_3), 2.39 (2H, q, *J* = 15.2, 7.6 Hz, H-2′), 1.45 (3H, t, *J* = 7.5 Hz, H-3′)；^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 217.2 (C-1), 48.6 (C-2), 77.3 (C-3), 38.3 (C-4), 41.5 (C-5), 32.9 (C-6), 174.2 (C-7), 138.4 (C-8), 56.4 (C-9), 49.9 (C-10), 20.5 (C-11), 34.4 (C-12), 36.7 (C-13), 45.2 (C-14), 30.0 (C-15), 169.7 (C-16), 77.3 (C-17), 20.4 (C-18), 15.8 (C-19), 120.7 (C-20), 141.9 (C-21), 109.8 (C-22), 142.9 (C-23), 22.5 (C-28), 22.0 (C-29), 122.9 (C-30), 52.2 (7-OCH_3), 174.1 (C-1′), 27.1 (C-2′), 8.9 (C-3′)。以上数据与文献报道一致[11]，故鉴定化合物 **7** 为 3-de (2-methylbutanoyl)-3-propanoylcipadesin。

化合物 **8**：白色无定形粉末，易溶于氯仿、丙酮。ESI-MS *m*/*z*: 563.06 $[M+Na]^+$，分子式为 $C_{31}H_{40}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.52 (1H, dd, *J* = 8.8, 7.6 Hz, H-2), 4.78 (1H, d, *J* = 9.2 Hz, H-3), 3.44 (1H, dd, *J* = 8.0, 4.0 Hz, H-5), 2.38 (1H, s, H-6a), 2.39 (1H, d, *J* = 4.4 Hz, H-6b), 2.25 (1H, m, H-9), 1.68 (1H, m, H-11α), 2.11 (1H, m, H-11β), 1.42 (1H, m, H-12α), 1.68 (1H, m, H-12β), 2.91 (1H, dd, *J* = 18.4, 6.0 Hz, H-15α), 2.81 (1H, d, *J* = 18.4 Hz, H-15β), 5.71 (1H, s, H-17), 1.11 (3H, s, H-18), 1.17 (3H, s, H-19), 7.82 (1H, brs, H-21), 6.48 (1H, brs, H-22), 7.43 (1H, brs, H-23), 0.81 (3H, s, H-28), 0.84 (3H, s, H-29), 5.34 (1H, d, *J* = 7.2 Hz, H-30), 3.74 (3H, s, 7-OCH_3), 2.65 (1H, m, H-2′), 1.18 (3H, d, *J* = 7.2 Hz, H-3′)；^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 217.1 (C-1), 48.7 (C-2), 77.1 (C-3), 38.6 (C-4), 41.4 (C-5), 33.0 (C-6), 174.0 (C-7), 138.5 (C-8), 56.7 (C-9), 49.9 (C-10), 20.6 (C-11), 34.5 (C-12), 36.9 (C-13), 45.2 (C-14), 29.9 (C-15), 169.5 (C-16), 77.1 (C-17), 21.9 (C-18), 15.8 (C-19), 120.7 (C-20), 142.0 (C-21), 109.8 (C-22), 143.0 (C-23), 22.4 (C-28), 20.5 (C-29), 122.8 (C-30), 52.2 (7-OCH_3), 176.6 (C-1′), 33.8 (C-2′), 18.6 (C-3′), 19.1 (C-4′)。以上数据与文献报道一致[10]，故鉴定化合物 **8** 为 granatumin H。

化合物 **9**：白色无定形粉末，易溶于氯仿、丙酮。ESI-MS *m*/*z*: 573.07 $[M+Na]^+$，分子式为 $C_{32}H_{38}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.77 (1H, m, H-2), 4.95 (1H, d, *J* = 9.2 Hz, H-3), 3.37 (1H, dd, *J* = 9.6, 2.0 Hz, H-5), 2.42 (1H, dd, *J* = 16.8, 10.0 Hz, H-6a), 2.35 (1H, dd, *J* = 16.8, 2.0 Hz, H-6b), 2.29 (1H, d, *J* = 12.4 Hz, H-9), 1.78 (1H, m, H-11α), 1.51 (1H, m, H-11β), 1.31 (1H, m, H-12α), 1.78 (1H, m, H-12β), 6.18 (1H, s, H-15), 5.18 (1H, s, H-17), 1.06 (3H, s, H-18), 1.22 (3H, s, H-19), 7.53 (1H, brs, H-21), 6.50 (1H, brs, H-22), 7.46 (1H, brs, H-23), 0.85 (3H, s, H-28), 0.82 (3H, s, H-29), 6.28 (1H, dd, *J* = 6.0, 2.8 Hz, H-30), 3.72 (3H, s, 7-OCH_3), 7.04 (1H, m, H-3′), 1.93 (3H, d, *J* = 7.2 Hz, H-4′), 1.95 (3H, brs, H-5′)。以上数据与文献报道一致[12]，故鉴定化合物 **9** 为 tigloylseneganolide A。

化合物 **10**：白色无定形粉末。ESI-MS *m*/*z*: 579.11 $[M+Na]^+$，分子式为 $C_{31}H_{40}O_9$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.59 (1H, dd, *J* = 9.6, 2.4 Hz, H-2), 5.11 (1H, d, *J* = 9.6 Hz, H-3), 3.80 (1H, dd, *J* = 7.8, 3.6 Hz, H-5), 2.37 (2H, m, H-6), 1.96 (1H, m, H-9), 1.97 (1H, m, H-11α), 1.95 (1H, m, H-11β), 1.21 (1H, m, H-12α), 1.98 (1H, m, H-12β), 1.62 (1H, m, H-14), 2.81 (1H, dd, *J* = 16.0, 4.8 Hz, H-15α), 3.72 (1H, dd, *J* = 15.2, 14.8 Hz, H-15β), 5.20 (1H, s, H-17), 1.04 (3H, s, H-18), 1.10 (3H, s, H-19), 7.50 (1H, brs, H-21), 6.48 (1H, brs, H-22), 7.45 (1H, brs, H-23), 0.84

(3H, s, H-28), 0.82 (3H, s, H-29), 3.34 (1H, d, *J* = 2.4 Hz, H-30), 2.76 (1H, m, H-2′), 1.28 (3H, d, *J* = 6.0 Hz, H-3′), 1.30 (3H, d, *J* = 6.0 Hz, H-4′); ^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 214.3 (C-1), 48.9 (C-2), 77.2 (C-3), 39.4 (C-4), 42.6 (C-5), 33.2 (C-6), 174.2 (C-7), 60.7 (C-8), 55.9 (C-9), 48.3 (C-10), 19.4 (C-11), 33.4 (C-12), 36.4 (C-13), 45.9 (C-14), 34.0 (C-15), 172.0 (C-16), 78.8 (C-17), 26.4 (C-18), 15.9 (C-19), 120.1 (C-20), 141.0 (C-21), 110.3 (C-22), 143.1 (C-23), 21.0 (C-28), 22.5 (C-29), 63.4 (C-30), 52.4 (7-OCH_3), 176.1 (C-1′), 34.2 (C-2′), 19.5 (C-3′), 19.0 (C-4′)。以上数据与文献报道一致[13]，故鉴定化合物 **10** 为 ruageanin A。

化合物 **11**：白色无定形粉末，易溶于氯仿、丙酮。ESI-MS *m*/*z*: 591.07 $[M+Na]^+$，分子式为 $C_{32}H_{40}O_9$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.66 (1H, dd, *J* = 9.2, 2.4 Hz, H-2), 5.06 (1H, d, *J* = 9.2 Hz, H-3), 3.34 (1H, dd, *J* = 8.4, 3.2 Hz, H-5), 2.38 (1H, overlap, H-6a), 2.38 (1H, overlap, H-6b), 1.82 (1H, m, H-9), 1.82 (1H, m, H-11α), 1.82 (1H, m, H-11β), 1.22 (1H, m, H-12α), 1.82 (1H, m, H-12β), 1.58 (1H, dd, *J* = 14.0, 4.8 Hz, H-14), 2.78 (1H, dd, *J* = 16.0, 4.8 Hz, H-15α), 3.59 (1H, dd, *J* = 16.0, 14.4 Hz, H-15β), 5.18 (1H, s, H-17), 1.02 (3H, s, H-18), 1.10 (3H, s, H-19), 7.50 (1H, brs, H-21), 6.46 (1H, brs, H-22), 7.45 (1H, brs, H-23), 0.85 (3H, s, H-28), 0.86 (3H, s, H-29), 3.25 (1H, d, *J* = 2.4 Hz, H-30), 3.77 (3H, s, 7-OCH_3), 7.05 (1H, m, H-3′), 1.96 (3H, overlap, H-4′), 1.96 (3H, overlap, H-5′)。以上数据与文献报道一致[14]，故鉴定化合物 **11** 为 swietemahonolide。

化合物 **12**：白色无定形粉末。ESI-MS *m*/*z*: 575.08 $[M+Na]^+$，分子式为 $C_{32}H_{40}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.24 (1H, m, H-2), 4.86 (1H, d, *J* = 9.6 Hz, H-3), 3.38 (1H, dd, *J* = 10.0, 3.2 Hz, H-5), 2.41 (1H, brs, H-6a), 2.44 (1H, d, *J* = 12.4 Hz, H-6b), 2.08 (1H, m, H-9), 1.72 (1H, m, H-11α), 1.72 (1H, m, H-11β), 1.12 (1H, m, H-12α), 1.72 (1H, m, H-12β), 3.30 (1H, dt, *J* = 21.2, 3.2 Hz, H-15α), 3.67 (1H, d, *J* = 21.2 Hz, H-15β), 5.57 (1H, s, H-17), 1.05 (3H, s, H-18), 1.19 (3H, s, H-19), 7.59 (1H, brs, H-21), 6.50 (1H, brs, H-22), 7.44 (1H, brs, H-23), 0.84 (3H, s, H-28), 0.80 (3H, s, H-29), 2.09 (1H, m, H-30α), 2.70 (1H, dd, *J* = 15.2, 2.0 Hz, H-30β), 3.75 (3H, s, 7-OCH_3), 6.99 (1H, m, H-3′), 1.86 (3H, d, *J* = 6.8 Hz, H-4′), 1.92 (3H, s, H-5′)。以上数据与文献报道一致[15]，鉴定化合物 **12** 为 khayasin T。

化合物 **13**：白色无定形粉末，易溶于氯仿、丙酮。ESI-MS *m*/*z*: 573.10 $[M+Na]^+$，分子式为 $C_{32}H_{38}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 5.87 (1H, d, *J* = 6.3 Hz, H-2), 4.84 (1H, dd, *J* = 6.4, 2.0 Hz, H-3), 3.84 (1H, dd, *J* = 12.0, 5.0 Hz, H-5), 3.90 (1H, dd, *J* = 16.0, 2.2 Hz, H-6a), 2.55 (1H, dd, *J* = 16.0, 12.8 Hz, H-6b), 2.70 (1H, brd, *J* = 6.0 Hz, H-9), 1.66 (1H, m, H-11α), 1.92 (1H, m, H-11β), 1.06 (1H, dt, *J* = 13.6, 3.6 Hz, H-12α), 1.35 (1H, brt, *J* = 14.0 Hz, H-12β), 3.11 (1H, brs, H-15α), 3.11 (1H, brs, H-15β), 5.47 (1H, s, H-17), 1.13 (3H, s, H-18), 1.01 (3H, s, H-19), 7.58 (1H, brs, H-21), 6.50 (1H, brs, H-22), 7.43 (1H, brs, H-23), 1.17 (3H, s, H-28), 2.06 (1H, d, *J* = 17.6 Hz, H-29α), 2.45 (1H, d, *J* = 17.4 Hz, H-29β), 5.89 (1H, brs, H-30), 3.76 (3H, s, 7-OCH_3), 7.33 (1H, q, *J* = 6.8 Hz, H-3′), 1.75 (3H, q, *J* = 7.2 Hz, H-4′), 1.80 (3H, s, H-5′)。以上数据与文献报道一致[16]，故鉴定化合物 **13** 为 andhraxylocarpin D。

化合物 **14**：白色无定形粉末，易溶于氯仿、丙酮。ESI-MS *m*/*z*: 577.10 $[M+Na]^+$，分子式为 $C_{32}H_{42}O_8$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.16 (1H, m, H-2), 4.92 (1H, d, *J* = 8.8 Hz, H-3), 3.80 (1H, overlap, H-5), 2.36 (2H, overlap, H-6), 1.95 (1H, m, H-8), 1.74 (1H, m, H-9), 2.36 (1H, m, H-11α), 1.62 (1H, m, H-11β), 1.22 (1H, m, H-12α), 1.62 (1H, m, H-12β), 1.90 (1H, m, H-14), 2.95 (1H, dd, *J* = 18.8, 7.2 Hz, H-15α), 2.35 (1H, overlap, H-15β), 5.84 (1H, s, H-17), 1.00 (3H, s, H-18), 1.11 (3H, s, H-19), 7.75 (1H, brs, H-21), 6.49 (1H, brs, H-22), 7.46 (1H, brs, H-23), 0.87 (3H, s, H-28), 0.82 (3H, s, H-29), 1.73 (1H, m, H-30α), 2.88 (1H, dd, *J* = 14.0, 7.2 Hz, H-30β), 3.77 (3H, s, 7-OCH_3), 6.89 (1H, m, H-3′), 1.78 (3H, d, *J* = 7.2 Hz, H-4′), 1.85 (3H, s, H-5′)。以上数据与文献报道一致[17]，故鉴定化合物 **14** 为 granatumin D。

化合物 **15**：白色无定形粉末。ESI-MS *m*/*z*: 461.05 $[M+Na]^+$，分子式为 $C_{26}H_{30}O_6$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 7.10 (1H, d, *J* = 10.0 Hz, H-1), 5.93 (1H, d, *J* = 10.0 Hz, H-2), 2.94 (1H, t, *J* = 14.4 Hz, H-5), 2.24 (1H, brd, *J* = 12.2 Hz, H-6a), 2.18 (1H, dd,

J = 14.7, 3.0 Hz, H-6b), 2.41 (1H, brd, *J* = 13.6 Hz, H-9), 2.02 (1H, m, H-11α), 1.89 (1H, m, H-11β), 1.50 (1H, m, H-12α), 1.80 (1H, m, H-12β), 3.89 (1H, s, H-15), 5.49 (1H, s, H-17), 1.37 (3H, s, H-18), 1.24 (3H, s, H-19), 7.43 (1H, brs, H-21), 6.38 (1H, brs, H-22), 7.41 (1H, brs, H-23), 1.16 (3H, s, H-28), 1.15 (3H, s, H-29), 1.17 (3H, s, H-30)；^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 156.0 (C-1), 126.4 (C-2), 203.3 (C-3), 45.2 (C-4), 54.6 (C-5), 36.7 (C-6), 208.2 (C-7), 53.6 (C-8), 47.6 (C-9), 39.6 (C-10), 17.2 (C-11), 32.2 (C-12), 37.7 (C-13), 65.6 (C-14), 53.4 (C-15), 166.9 (C-16), 78.0 (C-17), 20.9 (C-18), 19.8 (C-19), 120.2 (C-20), 141.0 (C-21), 109.8 (C-22), 143.1 (C-23), 20.9 (C-28), 20.7 (C-29), 17.4 (C-30)。以上数据与文献报道一致[18]，故鉴定化合物 **15** 为 7-oxo-7-deacetoxygenunin。

化合物 **16**：白色无定形粉末。ESI-MS *m*/*z*: 505.09 $[M+Na]^+$，分子式为 $C_{28}H_{34}O_7$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 7.11 (1H, d, *J* = 10.0 Hz, H-1), 5.87 (1H, d, *J* = 10.0 Hz, H-2), 2.20 (1H, dd, *J* = 13.2, 2.4 Hz, H-5), 1.81 (2H, m, H-6), 4.57 (1H, s, H-7), 2.51 (1H, dd, *J* = 12.4, 6.0 Hz, H-9), 1.96 (1H, m, H-11α), 1.70 (1H, m, H-11β), 2.02 (2H, m, H-12), 3.55 (1H, s, H-15), 1.88 (2H, m, H-16), 5.64 (1H, s, H-17), 1.27 (3H, s, H-18), 1.17 (3H, s, H-19), 7.43 (1H, brs, H-21), 6.36 (1H, brs, H-22), 7.42 (1H, brs, H-23), 1.09 (3H, s, H-28), 1.08 (3H, s, H-29), 1.24 (3H, s, H-30), 2.13 (3H, s, H-2′)；^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 157.0 (C-1), 126.0 (C-2), 204.0 (C-3), 44.1 (C-4), 46.0 (C-5), 23.3 (C-6), 73.2 (C-7), 42.6 (C-8), 39.5 (C-9), 40.0 (C-10), 15.0 (C-11), 26.0 (C-12), 38.7 (C-13), 69.8 (C-14), 56.9 (C-15), 167.5 (C-16), 78.3 (C-17), 17.7 (C-18), 19.8 (C-19), 120.4 (C-20), 143.1 (C-21), 109.9 (C-22), 141.2 (C-23), 27.2 (C-28), 21.2 (C-29), 18.3 (C-30), 169.9 (C-1′), 21.1 (C-2′)。以上数据与文献报道一致[19]，故鉴定化合物 **16** 为 gedunin。

化合物 **17**：白色无定形粉末，易溶于氯仿、丙酮。ESI-MS *m*/*z*: 493.08 $[M+Na]^+$，分子式为 $C_{27}H_{34}O_7$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 3.54 (1H, dd, *J* = 6.4, 4.0 Hz, H-1), 2.51 (1H, dd, *J* = 14.4, 4.0 Hz, H-2α), 2.94 (1H, dd, *J* = 14.4, 6.0 Hz, H-2β), 2.90 (1H, overlap, H-5), 2.29 (1H, d, *J* = 16.4 Hz, H-6a), 2.64 (1H, dd, *J* = 16.4, 10.4 Hz, H-6b), 2.19 (1H, m, H-9), 1.59 (1H, m, H-11α), 2.20 (1H, m, H-11β), 1.18 (1H, m, H-12α), 1.92 (1H, m, H-12β), 2.57 (1H, d, *J* = 18.0 Hz, H-15α), 2.96 (1H, d, *J* = 18.0 Hz, H-15β), 5.68 (1H, s, H-17), 0.88 (3H, s, H-18), 0.96 (3H, s, H-19), 7.45 (1H, brs, H-21), 6.40 (1H, brs, H-22), 7.40 (1H, brs, H-23), 1.06 (3H, s, H-28), 1.21 (3H, s, H-29), 4.91 (1H, s, H-30α), 5.17 (1H, s, H-30β), 3.73 (3H, s, 7-OCH_3)；^{13}C-NMR (100 MHz, $CDCl_3$) *δ*: 77.2 (C-1), 39.4 (C-2), 212.8 (C-3), 48.0 (C-4), 42.9 (C-5), 32.7 (C-6), 173.9 (C-7), 145.8 (C-8), 49.9 (C-9), 33.8 (C-10), 23.7 (C-11), 29.3 (C-12), 41.4 (C-13), 80.2 (C-14), 33.8 (C-15), 170.1 (C-16), 79.6 (C-17), 13.8 (C-18), 21.6 (C-19), 120.8 (C-20), 140.8 (C-21), 109.9 (C-22), 142.8 (C-23), 25.8 (C-28), 21.5 (C-29), 111.6 (C-30) 52.1 (7-OCH_3)。以上数据与文献报道一致[20]，故鉴定化合物 **17** 为 methyl angolensa。

化合物 **18**：白色无定形粉末。ESI-MS *m*/*z*: 477.08 $[M+Na]^+$，分子式为 $C_{28}H_{38}O_5$。^{1}H-NMR (400 MHz, $CDCl_3$) *δ*: 7.15 (1H, d, *J* = 10.4 Hz, H-1), 5.87 (1H, d, *J* = 10.4 Hz, H-2), 2.23 (1H, m, H-5), 1.93 (1H, m, H-6a), 1.75 (1H, m, H-6b), 5.26 (1H, m, H-7), 2.23 (1H, m, H-9), 1.75 (1H, m, H-11α), 1.92 (1H, m, H-11β), 1.58 (1H, m, H-12α), 1.75 (1H, m, H-12β), 5.32 (1H, m, H-15), 2.23 (1H, m, H-16α), 2.06 (1H, m, H-16β), 1.75 (1H, m, H-17), 1.04 (3H, s, H-18), 1.20 (3H, s, H-19), 2.72 (1H, m, H-20), 4.49 (1H, t, *J* = 8.4 Hz, H-21α), 3.94 (1H, t, *J* = 8.8 Hz, H-21β), 2.53 (1H, dd, *J* = 17.2, 8.0 Hz, H-22α), 2.23 (1H, m, H-22β), 1.20 (3H, s, H-28), 1.10 (3H, s, H-29), 1.10 (3H, s, H-30)。以上数据与文献报道一致[21]，故鉴定化合物 **18** 为 20,21,22,23-tetrahydro-23-oxoazadirone。

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