白头婆花化学成分的分离与鉴定

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摘　要:本文为研究白头婆(*Eupatorium japonicum*)花的化学成分,利用多种色谱技术对白头婆花 70% 乙醇提取物进行分离得到 19 个化合物,经 NMR、MS 等波谱方法鉴定为香豆素(**1**)、2-hydroxy-2,6-dimethylbenzofuran-3(2H)-one(**2**)、1-(2-hydroxy-4-methylphenyl)propan-1,2-dione(**3**)、subamone(**4**)、(7R^*)-opposit-4(15)-ene-1β,7-diol(**5**)、2,5-二羟基苯甲酸(**6**))、咖啡酸(**7**)、9-羟基百里酚(**8**)、原儿茶酸(**9**)、反式邻羟基肉桂酸(**10**)、7-羟基香豆素(**11**)、槲皮素(**12**)、蒲公英甾醇(**13**)、9-acetoxythymol 3-*O*-tiglate(**14**)、蒲公英甾醇乙酸酯(**15**)、亚油酸(**16**)、9-angeloyloxythymol(**17**)、豆甾醇(**18**)、棕榈酸(**19**)。化合物 **2**~**9**、**11**~**13**、**16**、**19** 为首次从白头婆中分离得到,化合物 **4**~**6**、**16** 为首次从泽兰属中分离得到。

关键词:白头婆;化学成分;分离;鉴定
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Isolation and identification of chemical constituents in the flowers of *Eupatorium japonicum*

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Abstract: To study chemical constituents of the flowers of *Eupatorium japonicum*. Nineteen known compounds were isolated from the 70% ethanol extraction of the flowers of *E. japonicum* by various chromatographic methods. On the basis of NMR and MS spectroscopic analysis, their structures were identified as coumarin (**1**), 2-hydroxy-2,6-dimethylbenzofuran-3(2H)-one (**2**), 1-(2-hydroxy-4-methylphenyl)propan-1,2-dione (**3**), subamone (**4**), (7R^*)-opposit-4(15)-ene-1β,7-diol (**5**), gentisic acid (**6**), caffeic acid (**7**), 9-hydroxythymol (**8**), protocatechuic acid (**9**), trans-o-hydroxycinnamic acid (**10**), umbelliferone (**11**), quercetin (**12**), traxasterol (**13**), 9-acetoxythymol 3-*O*-tiglate (**14**), taraxasterol acetate (**15**), linoleic acid (**16**), 9-angeloyloxythymol (**17**), stigmasterol (**18**), palmitic acid (**19**). Compounds **2-9**, **11-13**, **16**, **19** were isolated from *E. japonicum* for the first time, and compounds **4-6**, **16** were isolated from the genus of *Eupatorium* for the first time.

Key words: *Eupatorium japonicum*; chemical constituents; isolation; identification

白头婆 *Eupatorium japonicum* 是菊科泽兰属植物,多年生草本,多生于山坡林下、河岸水旁[1]。我国东部及西南部各省份均有分布,日本、朝鲜广布。《中国民族药辞典》记载白头婆是我国 12 个少数民族使用的常用草药,具有治疗感冒发热、风湿咳嗽、跌打损伤、产后腰痛、蛇咬伤等多种功效。在湘西土家族地区,白头婆主要用于治疗跌打损伤,具有“强盗药”之称;土家药匠将白头婆的花泡酒喝,总结出具有抗疲劳、壮阳、治疗多种炎症的功效;在韩国,白头婆的花用于泡茶[2]。现代药理研究表明白头婆具有抗肿瘤、抗菌、抗炎等多种活性[3-5]。在本课题组研究基础之上,为继续挖掘白头婆的化学成分,本实验对白头婆花 70% 乙醇提取物进行系统的化学成分分离。

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1 仪器与材料

Bruker Avance Ⅲ 600 型核磁共振波谱仪(瑞士布鲁克);LTQ-Obitrap XL 质谱仪(赛默飞世尔);LC-20AT 型高效液相色谱(日本岛津);YMC-Pack ODS-A 色谱柱(250 mm × 10 mm,5 μm);R2002 旋转蒸发仪(上海 SENCO);R-210 旋转蒸发仪(瑞士 BUCHI);BZF-50 型真空干燥箱(上海博讯);WFH-203B 紫外暗箱(上海精科);BSZ-160F 电脑自动部分收集器(上海精科);柱色谱硅胶、薄层色谱硅胶 G_{254} 均购自青岛海洋化工有限公司;Sephadex LH-20 凝胶(Pharmacia);HW-40C 凝胶(Toyopearl);高效液相用甲醇为色谱纯(天津科密欧化工);其他试剂均为分析纯,水为怡宝水。

白头婆花于 2017 年 10 月采自湖南省龙山县石牌镇,经湘西自治州民族医药研究所田华咏教授鉴定为白头婆 *Eupatorium japonicum* 的花。植物标本(编号:JSU20181007)存于吉首大学湖南省土家医药研究中心。

2 提取与分离

白头婆干燥花(3.0 kg)用 30 L 的 70% 乙醇 80 ℃提取 5 次,每次 2 h,合并滤液,减压干燥,得浸膏 960.0 g。浸膏经 100 ~ 200 目硅胶拌样,依次用石油醚、乙酸乙酯、丙酮、甲醇洗脱,洗脱液浓缩至干。将乙酸乙酯部位浸膏拌样,经正相硅胶柱分离,石油醚-乙酸乙酯(7∶1→1∶2)梯度洗脱,得 5 个组分(Fr. 1 ~ 5)。Fr. 2(2.3 g)经硅胶柱层析反复分离(石油醚-乙酸乙酯系统和二氯甲烷-乙酸乙酯系统洗脱)、Sephadex LH-20 柱色谱分离得化合物 **1**(316.9 mg)、**2**(10.0 mg)、**3**(7.2 mg)。Fr. 3(2.4 g)经硅胶柱层析分离(石油醚-乙酸乙酯系统和正己烷-丙酮系统洗脱)、Sephadex LH-20 柱色谱分离、半制备型 HPLC 纯化得化合物 **4**(11.1 mg)、**5**(26.7 mg)、**8**(4.0 mg)。Fr. 4(22.6 g)经硅胶柱层析,用二氯甲烷-甲醇系统洗脱,经 MCI 柱色谱(50%、70%、90%、100% 乙醇洗脱)分离除去色素,经 HW-40C 凝胶柱色谱分离、制备薄层色谱(二氯甲烷∶甲醇 = 7∶1)分离得化合物 **6**(1.2 mg)、**7**(5.5 mg)、**9**(50.9 mg)、**11**(1.3 mg)、**10**(183.0 mg)。Fr. 5 经硅胶柱层析分离(二氯甲烷-甲醇 50∶1→20∶1)、HW-40C 凝胶柱色谱分离得化合物 **12**(14.1 mg)。将二氯甲烷部位浸膏硅胶拌样,经正相硅胶柱分离,石油醚-乙酸乙酯(20∶1→5∶1)洗脱得到 4 个组分(Fr. 1 ~ 4)。Fr. 2(11.2 g)经硅胶柱色谱分离得化合物 **13**(43.0 mg)、**14**(23.1 mg)、**15**(2.8 g)。Fr. 3 经硅胶柱反复分离、凝胶柱纯化得化合物 **16**(42.5 mg)、**17**(18.0 mg)、**18**(50.0 mg)。石油醚部位浸膏经硅胶柱分离,石油醚-二氯甲烷(50∶1→4∶1)洗脱得到 5 个组分(Fr. 1 ~ 5)。Fr. 4(1.7 g)通过硅胶柱分离得到化合物 **19**(8.2 mg)。

3 结构鉴定

化合物 1 白色粉末;ESI-MS:*m/z* 169 [M + Na]$^+$,$C_9H_6O_2$;^{1}H NMR(600 MHz,$CDCl_3$)δ:6.42(1H,d,*J* = 9.6 Hz,H-3),7.28(1H,td,*J* = 7.2,1.2 Hz,H-6),7.33(1H,dd,*J* = 7.8,1.2 Hz,H-8),7.49(1H,dd,*J* = 7.8,1.2 Hz,H-5),7.53(1H,td,*J* = 7.2,1.2 Hz,H-7),7.71(1H,d,*J* = 9.6 Hz,H-4);^{13}C NMR(150 MHz,$CDCl_3$)δ:116.8(C-3),117.0(C-8),118.9(C-10),124.6(C-6),128.0(C-5),132.0(C-7),143.6(C-4),154.1(C-9),160.9(C-2)。以上数据与文献[6]报道基本一致,故鉴定为香豆素。

化合物 2 黄色粉末;ESI-MS:*m/z* 201 [M + Na]$^+$,$C_{10}H_{10}O_3$;^{1}H NMR(600 MHz,$CDCl_3$)δ:1.64(3H,s,CH_3-2),2.43(3H,s,CH_3-6),6.85(1H,s,H-7),6.90(1H,d,*J* = 7.8 Hz,H-5),7.54(1H,d,*J* = 7.8 Hz,H-4);^{13}C NMR(150 MHz,$CDCl_3$)δ:22.2(CH_3-2),22.9(CH_3-6),103.8(C-2),113.6(C-7),116.1(C-9),124.1(C-5),125.1(C-4),151.7(C-6),170.7(C-8),198.4(C-3)。以上数据与文献[7]报道基本一致,故鉴定为 2-hydroxy-2,6-dimethylbenzofuran-3(2*H*)-one。

化合物 3 无色油状;ESI-MS:*m/z* 201 [M + Na]$^+$,$C_{10}H_{10}O_3$;^{1}H NMR(600 MHz,$CDCl_3$)δ:2.37(3H,s,Me-4),2.52(3H,s,H-10),6.74(1H,d,*J* = 8.4 Hz,H-5),6.84(1H,s,H-3),7.63(1H,d,*J* = 8.4 Hz,H-6);^{13}C NMR(150 MHz,$CDCl_3$)δ:22.4(Me-4),26.8(C-10),113.0(C-1),118.8(C-3),121.2(C-5),132.3(C-6),150.5(C-4),164.4(C-2),195.3(C-8),199.6(C-9)。以上数据与文献[7]报道基本一致,故鉴定为 1-(2-hydroxy-4-methylphenyl)propan-1,2-dione。

化合物 4 白色粉末;ESI-MS:*m/z* 191 [M + Na]$^+$,$C_{10}H_{16}O_2$;^{1}H NMR(600 MHz,CD_3OD)δ:0.86(3H,d,*J* = 6.6 Hz,CH_3-4),0.92(3H,d,*J* = 6.6 Hz,CH_3-6),1.70(3H,t,*J* = 1.8 Hz,CH_3-3),1.87(1H,m,H-6),2.12(1H,t,*J* = 16.2 Hz,H-5α),

2.16(1H,m,H-4),2.32(1H,dd,J =16.2,3.6 Hz,H-5β),4.25(1H,dt,J =9.6,1.8 Hz,H-7),6.68(1H,s,H-2);^{13}C NMR(150 MHz,CD_3OD)δ:15.4(CH_3-3),16.7(CH_3-4),20.9(CH_3-6),27.3(C-4),37.2(C-5),51.2(C-6),69.4(C-7),135.4(C-3),151.9(C-2),202.3(C-1)。以上数据与文献[8]报道基本一致,故鉴定为 subamone。

化合物 5　无色油状;ESI-MS:m/z 261 [M+Na]$^+$,$C_{15}H_{26}O_2$;^{1}H NMR(600 MHz,CD_3OD)δ:0.61(3H,s,H-14),0.86(3H,d,J =6.6 Hz,H-12),0.92(3H,d,J =6.6 Hz,H-13),1.31(1H,m,H-8α),1.35(1H,m,H-9α),1.50(1H,m,H-2β),1.72(1H,m,H-9β),1.74(1H,m,H-11),1.84(1H,d,J =10.8 Hz,H-5),1.87(1H,m,H-2α),1.90(1H,m,H-8β),2.18(1H,m,H-3α),2.24(1H,m,H-3β),2.31(1H,m,H-6),3.13(1H,dd,J =7.8,4.2 Hz,H-7),3.48(1H,dd,J =11.4,4.8 Hz,H-1),4.67(1H,s,H-15α),4.81(1H,s,H-15β);^{13}C NMR(150 MHz,CD_3OD)δ:12.9(C-14),16.4(C-12),21.3(C-13),27.5(C-8),32.4(C-11),32.7(C-2),36.0(C-3),38.6(C-9),40.9(C-6),50.7(C-10),56.2(C-5),79.8(C-1),82.8(C-7),107.4(C-15),149.4(C-4)。以上数据与文献[9]报道基本一致,故鉴定为(7R^*)-opposit-4(15)-ene-1β,7-diol。

化合物 6　白色粉末;ESI-MS:m/z 177 [M+Na]$^+$,$C_7H_6O_4$;^{1}H NMR(600 MHz,CD_3OD)δ:6.65(1H,d,J =8.4 Hz,H-3),7.28(1H,d,J =8.4 Hz,H-4),7.35(1H,s,H-6);^{13}C NMR(150 MHz,CD_3OD)δ:115.2(C-1),117.8(C-3),123.0(C-6),130.6(C-4),145.3(C-5),148.9(C-2),175.8(COOH)。以上数据与文献[10]报道基本一致,故鉴定为2,5-二羟基苯甲酸。

化合物 7　白色粉末;ESI-MS:m/z 203 [M+Na]$^+$,$C_9H_8O_4$;^{1}H NMR(600 MHz,CD_3OD)δ:6.16(1H,d,J =15.6 Hz,H-8),6.71(1H,d,J =8.4 Hz,H-5),6.86(1H,dd,J =8.4,1.8 Hz,H-6),6.97(1H,d,J =1.8 Hz,H-2),7.45(1H,d,J =15.6 Hz,H-7);^{13}C NMR(150 MHz,CD_3OD)δ:115.0(C-5),116.1(C-2),116.5(C-8),122.8(C-6),127.9(C-1),146.6(C-3),146.8(C-7),149.4(C-4),171.5(C-9)。以上数据与文献[11]报道基本一致,故鉴定为咖啡酸。

化合物 8　白色粉末;ESI-MS:m/z 189 [M+Na]$^+$,$C_{10}H_{14}O_2$;^{1}H NMR(600 MHz,CD_3OD)δ:1.19(3H,d,J =7.2 Hz,H-10),2.17(3H,s,H-7),3.18(1H,dd,J =13.2,6.6 Hz,H-8),3.43(1H,d,J =6.6 Hz,H-9β),3.66(1H,dd,J =10.2,5.4 Hz,H-9α),6.50(1H,s,H-2),6.54(1H,d,J =7.8 Hz,H-6),6.91(1H,d,J =7.8 Hz,H-5);^{13}C NMR(150 MHz,CD_3OD)δ:17.1(C-10),21.1(C-7),36.7(C-8),68.1(C-9),116.6(C-2),121.3(C-6),128.3(C-5),128.6(C-4),137.8(C-1),156.0(C-3)。以上数据与文献[12]报道基本一致,故鉴定为 9-羟基百里酚。

化合物 9　无色油状;ESI-MS:m/z 177 [M+Na]$^+$,$C_7H_6O_4$;^{1}H NMR(600 MHz,CD_3OD)δ:6.65(1H,d,J =8.4 Hz,H-5),7.28(1H,d,J =8.4 Hz,H-6),7.42(1H,d,J =1.8 Hz,H-2);^{13}C NMR(150 MHz,CD_3OD)δ:115.5(C-5),117.7(C-2),123.5(C-6),126.2(C-1),145.7(C-3),150.4(C-4),172.6(C-7)。以上数据与文献[11]报道基本一致,故鉴定为原儿茶酸。

化合物 10　白色粉末;ESI-MS:m/z 187 [M+Na]$^+$,$C_9H_8O_3$;^{1}H NMR(600 MHz,CD_3OD)δ:6.37(1H,d,J =16.2 Hz,H-8),6.64(1H,d,J =1.8 Hz,H-5),6.65(1H,d,J =1.8 Hz,H-3),7.00(1H,td,J =7.8,1.8 Hz,H-4),7.28(1H,dd,J =7.8,1.8 Hz,H-6),7.77(1H,d,J =16.2 Hz,H-7);^{13}C NMR(150 MHz,CD_3OD)δ:116.9(C-3),119.1(C-8),120.7(C-5),122.7(C-1),129.9(C-6),132.4(C-4),142.1(C-7),158.1(C-2),171.8(C-9)。以上数据与文献[13]报道基本一致,故鉴定为反式邻羟基肉桂酸。

化合物 11　黄色粉末;ESI-MS:m/z 185 [M+Na]$^+$,$C_9H_6O_3$;^{1}H NMR(600 MHz,CD_3OD)δ:6.13(1H,d,J =9.6 Hz,H-3),6.65(1H,d,J =2.4 Hz,H-8),6.74(1H,dd,J =8.4,2.4 Hz,H-6),7.40(1H,d,J =8.4 Hz,H-5),7.81(1H,d,J =9.6 Hz,H-4);^{13}C NMR(150 MHz,CD_3OD)δ:103.5(C-8),112.0(C-3),112.9(C-10),114.8(C-6),130.7(C-5),146.1(C-4),157.3(C-9),163.8(C-2),163.8(C-7)。以上数据[14]与文献报道基本一致,故鉴定为 7-羟基香豆素。

化合物 12　黄色粉末;ESI-MS:m/z 325 [M+Na]$^+$,$C_{15}H_{10}O_7$;^{1}H NMR(600 MHz,CD_3OD)δ:6.13(1H,s,H-8),6.34(1H,s,H-6),6.83(1H,d,J =

8.4 Hz,H-5′),7.58(1H,d,J =8.4 Hz,H-6′),7.69(1H,s,H-2′);^{13}C NMR(150 MHz,CD_3OD)δ:94.4(C-8),99.2(C-6),104.5(C-10),116.0(C-2′),116.2(C-5′),121.7(C-6′),124.1(C-1′),137.2(C-3),146.2(C-3′),148.0(C-2),148.7(C-4′),158.2(C-5),162.5(C-9),165.6(C-7),177.3(C-4)。以上数据与文献[15]报道基本一致,故鉴定为槲皮素。

化合物 13 白色粉末;ESI-MS:m/z 449 [M+Na]$^+$,$C_{30}H_{50}O$;^{1}H NMR(600 MHz,$CDCl_3$)δ:0.76(3H,s,CH_3-24),0.85(6H,s,CH_3-25,CH_3-28),0.93(3H,s,CH_3-27),0.97(3H,s,CH_3-23),1.01(3H,s,CH-26),1.01(3H,d,J =6.6 Hz,CH_3-29),3.20(1H,dd,J =11.4,4.8 Hz,H-3),4.60(1H,t,J =2.4 Hz,H-30β),4.61(1H,t,J =2.4 Hz,H-30α);^{13}C NMR(150 MHz,$CDCl_3$)δ:14.9(C-27),15.5(C-24),16.0(C-25),16.4(C-26),18.4(C-6),19.6(C-29),21.6(C-11),25.6(C-21),25.8(C-12),26.3(C-28),26.8(C-15),27.5(C-2),28.1(C-23),34.2(C-7),34.7(C-17),37.3(C-10),38.4(C-19),38.9(C-1),39.0(C-4,C-13),39.3(C-16),39.5(C-22),41.0(C-8),42.2(C-14),48.8(C-18),50.6(C-9),55.5(C-5),79.2(C-3),107.3(C-30),154.8(C-20)。以上数据与文献[16]报道基本一致,故鉴定为蒲公英甾醇。

化合物 14 无色油状;ESI-MS:m/z 313 [M+Na]$^+$,$C_{17}H_{22}O_4$;^{1}H NMR(600 MHz,$CDCl_3$)δ:1.24(3H,d,J =7.2 Hz,H-10),1.99(3H,s,$OCOCH_3$),2.07(3H,m,H-5′),2.08(3H,dd,J =7.2,1.2 Hz,H-4′),2.33(3H,s,H-7),3.23(1H,m,H-8),4.12(2H,d,J =7.2 Hz,H-9),6.28(1H,q,J =7.2 Hz,H-3′),6.89(1H,s,H-2),7.04(1H,d,J =7.8 Hz,H-6),7.19(1H,d,J =7.8 Hz,H-5);^{13}C NMR(150 MHz,$CDCl_3$)δ:16.1(C-4′),17.6(C-10),20.9(C-7),21.1(C-5′),21.1(CH_3CO-),32.0(C-8),68.7(C-9),123.3(C-2),127.1(C-6),127.1(C-5),127.2(C-2′),131.9(C-4),137.7(C-1),141.2(C-3′),148.6(C-3),166.4(C-1′),171.3(CH_3CO-)。以上数据与文献[17]报道基本一致,故鉴定为 9-acetoxythymol 3-*O*-tiglate。

化合物 15 白色粉末;ESI-MS:m/z 491 [M+Na]$^+$,$C_{32}H_{52}O_2$;^{1}H NMR(600 MHz,$CDCl_3$)δ:0.84,0.84,0.85,0.87,0.92,1.01,1.02(21H,7×-CH_3),2.04(3H,s,$COCH_3$),4.48(1H,dd,J =5.4,10.8Hz,H-3),4.60(2H,m,H-30);^{13}C NMR(150 MHz,$CDCl_3$)δ:14.9(C-27),16.0(C-25),16.5(C-26),16.6(C-24),18.3(C-6),19.6(C-28),21.5(C-11),21.6(COMe),23.8(C-2),25.6(C-29),25.7(C-21),26.3(C-12),26.8(C-15),28.1(C-23),34.1(C-7),34.7(C-17),37.2(C-10),37.9(C-4),38.4(C-16),38.6(C-1),39.0(C-22),39.3(C-13),39.5(C-19),41.0(C-8),42.2(C-14),48.7(C-18),50.5(C-9),55.6(C-5),81.1(C-3),107.6(C-30),154.8(C-20),171.2(C=O)。以上数据与文献[18]报道基本基本一致,故鉴定为蒲公英甾醇乙酸酯。

化合物 16 无色油状;ESI-MS:m/z 303 [M+Na]$^+$,$C_{18}H_{32}O_2$;^{1}H NMR(600 MHz,$CDCl_3$)δ:0.89(3H,t,J =6.8 Hz,H-18),1.32(14H,m,H-4,5,6,7,15,16,17),1.63(2H,m,H-3),2.05(4H,q,J =7.2 Hz,H-8,14),2.35(2H,t,J =7.2 Hz,H-2),2.77(2H,t,J =6.6 Hz,H-11),5.35(4H,m,H-9,10,12,13);^{13}C NMR(150 MHz,$CDCl_3$)δ:14.2(C-18),22.7(C-17),24.8(C-3),25.8(C-11),27.3(C-14),27.3(C-8),29.2(C-4),29.2(C-5),29.3(C-6),29.5(C-7),29.7(C-15),31.7(C-16),34.1(C-2),128.0(C-10),128.2(C-12),130.2(C-13),130.4(C-9),179.7(C-1)。以上数据与文献[19]报道基本一致,故鉴定为亚油酸。

化合物 17 无色油状;ESI-MS:m/z 271 [M+Na]$^+$,$C_{15}H_{20}O_3$;^{1}H NMR(600 MHz,$CDCl_3$)δ:1.37(3H,d,J =7.2 Hz,H-10),1.90(3H,s,H-5′),1.98(3H,d,J =7.2 Hz,H-4′),2.28(3H,s,H-7),3.38(1H,m,H-8),3.98(1H,dd,J =10.8,8.4 Hz,H-9β),4.38(1H,dd,J =10.8,4.8 Hz,H-9α),6.12(1H,m,H-3′),6.68(1H,s,H-2),6.72(1H,d,J =7.8 Hz,H-6),7.06(1H,d,J =7.8 Hz,H-5);^{13}C NMR(150 MHz,$CDCl_3$)δ:16.0(C-4′),16.6(C-10),20.7(C-5′),21.1(C-7),32.3(C-8),69.6(C-9),116.9(C-2),121.4(C-6),125.1(C-5),127.0(C-2′),127.7(C-4),138.0(C-1),139.3(C-3′),154.2(C-3),169.1(C-1′)。以上数据与文献[20]报道基本一致,故鉴定为 9-angeloyloxythymol。

化合物 18 白色粉末;ESI-MS:m/z 435 [M+Na]$^+$,$C_{29}H_{48}O$;^{1}H NMR(600 MHz,$CDCl_3$)δ:3.52(1H,m,3α-H),5.01(1H,dd,J =8.4,15.0 Hz,H-23),5.14(1H,dd,J =8.4,15.0 Hz,H-22),5.35(1H,s,H-6);^{13}C NMR(150 MHz,$CDCl_3$)δ:12.2(C-

18),12.42(C-29),19.1(C-26),19.6(C-19),21.2(C-21),21.3(C-7),21.4(C-11),24.5(C-15),25.6(C-28),29.1(C-16),31.8(C-1),32.0(C-2,7,8,25),36.6(C-10),37.4(C-12),39.8(C-4),40.7(C-20),42.4(C-13),50.3(C-9),51.4(C-24),56.1(C-14),57.0(C-17),72.0(C-3),121.9(C-6),129.4(C-23),138.5(C-22),140.9(C-5)。以上数据与文献[21]报道基本一致,故鉴定为豆甾醇。

化合物 19 白色粉末;ESI-MS:*m/z* 279 [M + Na]$^+$,$C_{16}H_{32}O_2$;^{1}H NMR(600 MHz,$CDCl_3$)δ:0.88(3H,t,J =6.6Hz,H-16),1.25(24H,s,12 × -CH_2),1.64(2H,m,H-3),2.35(2H,t,J =7.8Hz,H-2);^{13}C NMR(150 MHz,$CDCl_3$)δ:14.3,22.9,24.9,29.2,29.4,29.5,29.6,29.7,29.9,32.1,33.6。以上数据与文献[22]报道基本一致,故鉴定为棕榈酸。

4 结论

本实验通过多种色谱方法从白头婆花 70% 乙醇提取物中分离鉴定了 19 个化合物,其中化合物 2 ~9、11 ~13、16、19 为首次从白头婆中分离得到,化合物 4 ~6、16 为首次从泽兰属中分离得到。这些化合物主要为苯丙素类和萜类,其中苯丙素类化合物有主要有香豆素、1-(2-hydroxy-4-methylphenyl) propan-1,2-dione、咖啡酸、反式邻羟基肉桂酸、7-羟基香豆素、9-羟基百里酚;研究表明,香豆素具有广泛的生物活性,抑菌效果显著[23],咖啡酸具有抗病毒、抗菌、抗肿瘤等多种生物活性[24]。萜类化合物包括蒲公英甾醇、蒲公英甾醇乙酸酯、豆甾醇;豆甾醇生理活性强,具有抗炎,抗氧化,抗肿瘤等多种活性[25],其中化合物得量最多的是蒲公英甾醇乙酸酯(2.8 g),这与课题组前期对白头婆全草化学成分的研究结果相似[26],说明蒲公英甾醇乙酸酯可能是白头婆全草及花的主要化学成分,研究表明其具有抗炎[27]、抗疟疾[28]活性,但其是否可作为白头婆的药效物质,是否具有其他活性仍需进一步研究。以上分离鉴定的化合物大多具有多种生物活性,为白头婆的开发利用提供了参考依据。

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