

气相色谱-质谱联用分析青风藤挥发油中化学成分*

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摘 要:目的:方法:用药典记载的提取方法提取药材青风藤的挥发油,经过气相色谱-质谱联用分析,共分离出 200 多个峰,通过与软件中的质谱标准谱图库比较鉴定出其中 101 种化合物。结果:青风藤中主要物质为 n-十六烷酸和(Z,Z)-9,12-十八烷酸。意义:该研究为青风藤的物质基础研究和进一步的开发应用提供实验依据。

关键词:青风藤 挥发油 气相色谱-质谱联用

挥发油是中草药的一大类成分,是一种能随水蒸气蒸馏的以萜烯、倍半萜烯及其含氧衍生物为主要成分的油状液体,广泛存在于中药、香料中。气相色谱法(GC)可分离挥发油中的成分,结合质谱检测可以对挥发油中的成分作出定性和定量分析。

青风藤(Caulis Sinomenii)为较常用中药,别名清风藤(通称),风龙(广东),《本草纲目》又称之为青藤,寻风藤,商品为防己科植物青藤与毛青藤的藤茎。用于祛风湿,通经络,利小便。用于风湿痹痛,关节肿胀,麻痹搔痒。很多中药的挥发油可以入药,目前为止,国内对青风藤的研究大部分集中在用液相色谱分析其提取物,而对于青风藤挥发油的化学成分研究则未见有人报道。旨在研究青风藤的物质基础和开发青风藤的新用途,本实验用药典中记载的挥发油提取方法提取了青风藤的挥发油,采用气相色谱-质谱联用分析了其组成,发现其以脂肪酸为主

要成分。

一、实验部分

1. 试剂与仪器

(1)药材。

青风藤(产地安徽,购于大连市美罗药房),

(2)仪器。

Agilent-6890GC/5973MS 气质联用系统(美国安捷伦公司),

(3)试剂。

正己烷(色谱纯,天津市科密欧化学试剂开发中心);正己烷(分析纯,天津市科密欧化学试剂开发中心);异丙醇(色谱纯,山东禹王实业有限公司禹城化工厂);水为超纯水。

2. 样品处理方法

称取 31.64g 青风藤生药粉末,放入挥发油提取器,加入 300mL 超纯水,回流提取 5h,馏出液用正己

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烷:水以 2:1 的体积比萃取 3 次,分出正己烷层,并向其中加入无水硫酸钠干燥。然后将干燥好的正己烷层旋转蒸发,最后用氮气吹扫定容至 0.1mL。

3. GC/MS 分析

(1) 色谱条件。

色谱柱选用 Varian 公司的 60m × 0.25mm × 0.25μm DB-5 石英毛细管柱;载气选用氮气;进样口温度选择为 260℃;进样量为 1μL;分流比经过比较选择用 50:1;柱前压为 15psi;程序升温条件为初始柱温选在 50℃,保持 3min,然后以 1.5℃/min 的速率升至 160℃,再以 3℃/分钟的速率升至 280℃,保持 40min。

(2) 质谱条件。

电离方式采用电子离子化(EI)方式;电子能量为 70eV;离子源温度是 230℃;四级杆温度为 150℃;传输线温度是 280℃;溶剂延迟测定后选为 7min;扫描范围是 30–500maU。

(3) 分离与鉴定。

青风藤挥发油提取物经气相色谱分离,质谱仪记录质谱图,得到 GC-MS 谱图,各色谱峰相应的质谱图经计算机谱库检索及人工解析确定其化学结构。

二、结果与讨论

本实验选择最佳程序升温对青风藤挥发油样品进行了测定,获得了满意的分析结果。

由于青风藤挥发油中各化合物含量悬殊很大,故无法在一次实验中同时测定,所以选取不同浓度的样品进行了两次分析。图 1 和图 2 即分别为样品稀释前和稀释 200 倍后的气相色谱图。

经过气相色谱-质谱联用分析得到了青风藤挥发油的总离子流色谱图图 3。对图 3 上的质谱信息进行分析,根据其在工作站软

件中的质谱标准谱图库的匹配度,结合人工解析,鉴定了其中的 101 个化合物,结果如表 1。

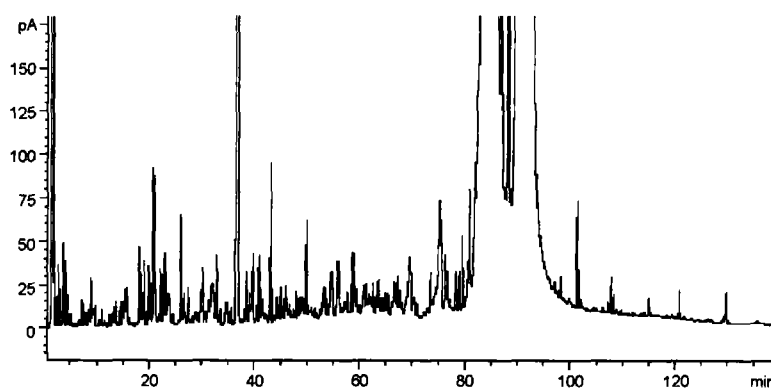


图 1 青风藤挥发油气相色谱图

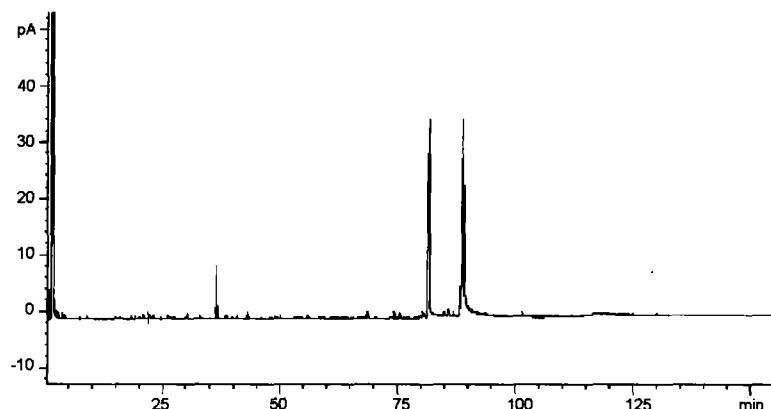


图 2 稀释 200 倍的青风藤挥发油气相色谱图

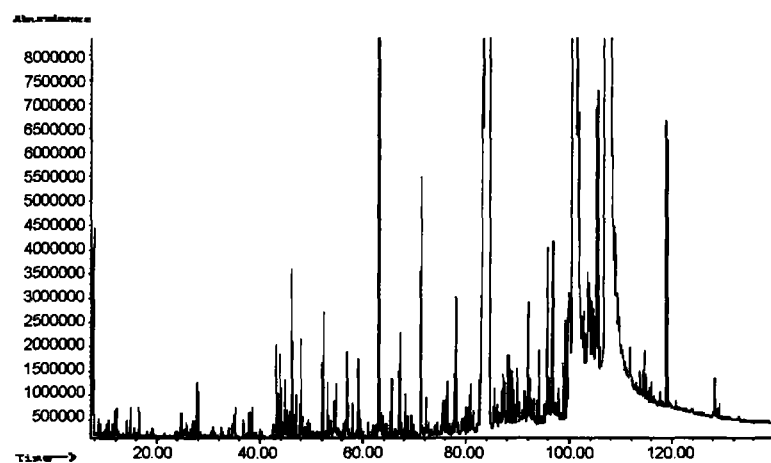


图 3 青风藤挥发油总离子流色谱图

表 1 青风藤挥发油的化学成分分析结果

序号	保留时间(min)	化学成分	分子式	相对分子质量	相似度(%)
1	8.69	1,3-dimethyl-Cyclopentane	C ₇ H ₁₄	98.11	94
2	8.78	methyl-Cyclohexane	C ₇ H ₁₄	98.11	91
3	10.20	2-methyl-Heptane	C ₈ H ₁₈	114.14	97
4	11.94	Octane	C ₈ H ₁₈	114.14	94
5	14.13	Furfural	C ₅ H ₄ O ₂	96.02	94
6	14.32	ethyl-Cyclohexane	C ₈ H ₁₆	112.13	90
7	15.57	(E)-2-Hexenal	C ₆ H ₁₀ O	98.07	96
8	18.13	2-Heptanone	C ₇ H ₁₄ O	114.10	90
9	21.40	1,1,2,3-tetramethyl-Cyclohexane	C ₁₀ H ₂₀	140.16	90
10	24.48	5-methyl-2-Furancarboxaldehyde	C ₆ H ₆ O ₂	110.04	97
11	24.73	Benzaldehyde	C ₇ H ₆ O	106.04	96
12	27.85	Decane	C ₁₀ H ₂₂	142.17	97
13	30.52	1-methyl-3-(1-methylethyl)-Benzene	C ₁₀ H ₁₄	134.11	90
14	30.98	D-Limonene	C ₁₀ H ₁₆	136.13	92
15	32.65	Benzeneacetaldehyde	C ₈ H ₈ O	120.06	94
16	33.93	(E)-2-Octenal	C ₈ H ₁₄ O	126.10	96
17	34.82	Acetophenone	C ₈ H ₈ O	120.06	92
18	35.18	alpha.-Methyl-.alpha.-[4-methyl-3-pentenyl]oxiranemethanol	C ₁₀ H ₁₈ O ₂	170.13	94
19	37.94	3,7-dimethyl-1,6-Octadien-3-ol	C ₁₀ H ₁₈ O	154.14	90
20	42.58	1,2-dimethoxy-Benzene	C ₈ H ₁₀ O ₂	138.07	93
21	43.22	(1R)-1,7,7-trimethyl-Bicyclo[2.2.1]heptan-2-one	C ₁₀ H ₁₆ O	152.12	98
22	43.95	trans-5-methyl-2-(1-methylethyl)-Cyclohexanone	C ₁₀ H ₁₈ O	154.14	98
23	44.18	(E)-2-Nonenal	C ₉ H ₁₆ O	140.12	95
24	44.90	5-methyl-2-(1-methylethyl)-Cyclohexanone	C ₁₀ H ₁₈ O	154.14	98
25	45.02	Octanoic Acid	C ₈ H ₁₆ O ₂	144.12	90
26	45.32	(1.alpha.,2.alpha.,5.alpha.)-5-methyl-2-(1-methylethyl)-Cyclohexanol	C ₁₀ H ₂₀ O	156.15	95
27	45.70	(1S-endo)-1,7,7-trimethyl-Bicyclo[2.2.1]heptan-2-ol	C ₁₀ H ₁₈ O	154.14	90
28	46.12	[1R-(1.alpha.,2.beta.,5.alpha.)]-5-methyl-2-(1-methylethyl)-Cyclohexanol	C ₁₀ H ₂₀ O	156.15	96
29	46.44	4-methyl-1-(1-methylethyl)-3-Cyclohexen-1-ol	C ₁₀ H ₁₈ O	154.14	90
30	47.05	Naphthalene	C ₁₀ H ₈	128.06	93
31	47.92	Dodecane	C ₁₂ H ₂₆	170.20	95
32	48.14	1-methoxy-4-(1-propenyl)-Benzene	C ₁₀ H ₁₂ O	148.09	98
33	48.74	Decanal	C ¹⁰ H ²⁰ O	156.15	97
34	48.94	3-Methylene-1,5,5-trimethylcyclohexene	C ₁₀ H ₁₆	136.13	92
35	49.43	1,2,3,4-tetrahydro-1,1,6-trimethyl-Naphthalene	C ₁₃ H ₁₈	174.14	90
36	51.21	1,2-Benzisothiazole	C ₇ H ₅ NS	135.01	91
37	51.80	2,3-Dimethoxytoluene	C ₉ H ₁₂ O ₂	152.08	91
38	52.23	5-methyl-2-(1-methylethylidene)-Cyclohexanone	C ₁₀ H ₁₆ O	152.12	98
39	52.80	(S)-2-methyl-5-(1-methylethenyl)-2-Cyclohexen-1-one	C ₁₀ H ₁₄ O	150.10	95
40	53.67	1,2,3,4-tetrahydro-1,1,6-trimethyl-Naphthalene	C ₁₃ H ₁₈	174.14	92
41	53.81	2-isopropyl-5-methyl-3-Cyclohexen-1-one	C ₁₀ H ₁₆ O	152.12	93
42	54.78	3,5-Dimethoxytoluene	C ₉ H ₁₂ O ₂	152.08	95
43	55.73	3-phenyl-2-Propenal	C ₉ H ₈ O	132.06	95
44	56.10	4-Hydroxy-3-methylacetophenone	C ₉ H ₁₀ O ₂	150.07	90

续表 1

序号	保留时间(min)	化学成分	分子式	相对分子质量	相似度(%)
45	56.58	(1S-endo)-1,7,7-trimethyl-Bicyclo[2.2.1]heptan-2-ol acetate	C ₁₂ H ₂₀ O ₂	196.15	94
46	56.86	1-methoxy-4-(1-propenyl)-Benzene	C ₁₀ H ₁₂ O	148.09	98
47	57.05	2-methyl-5-(1-methylethyl)-Phenol	C ₁₀ H ₁₄ O	150.10	93
48	57.25	5-(2-propenyl)-1,3-Benzodioxole	C ₁₀ H ₁₀ O ₂	162.07	90
49	57.97	1-methyl-Naphthalene	C ₁₁ H ₁₀	142.08	94
50	59.12	2-Methoxy-4-vinylphenol	C ₉ H ₁₀ O ₂	150.07	91
51	59.45	2-methyl-Naphthalene	C ₁₁ H ₁₀	142.08	90
52	59.60	2,6,10,10-Tetramethyl-1-oxa-spiro[4.5]dec-6-ene	C ₁₃ H ₂₂ O	194.17	90
53	59.87	1,2,3,4-tetramethyl-4-(1-methylethenyl)-Benzene	C ₁₃ H ₁₈	174.14	93
54	61.05	2-hydroxy-4-methoxy-Benzaldehyde	C ₈ H ₈ O ₃	152.05	90
55	62.37	(+)-4-Carene	C ₁₀ H ₁₆	136.13	90
56	63.08	3-Allyl-6-methoxyphenol	C ₁₀ H ₁₂ O ₂	164.08	98
57	64.47	1,2,4-Trimethoxybenzene	C ₉ H ₁₂ O ₃	168.08	96
58	65.51	(E)-1-(2,6,6-trimethyl-1,3-cyclohexadien-1-yl)-2-Buten-1-one	C ₁₃ H ₁₈ O	190.14	96
59	66.24	[1S-(1.alpha.,4.alpha.,7.alpha.)]-1,2,3,4,5,6,7,8-octahydro-1,4,9,9-tetramethyl-4,7-Methanoazulene	C ₁₅ H ₂₄	204.19	90
60	66.99	Tetradecane	C ₁₄ H ₃₀	198.24	98
61	67.30	1,2-dimethoxy-4-(2-propenyl)-Benzene	C ₁₁ H ₁₄ O ₂	178.10	97
62	68.63	[1S-(1.alpha.,3a.beta.,4.alpha.,8a.beta.)]-decahydro-4,8,8-trimethyl-9-methylene-1,4-Methanoazulene	C ₁₅ H ₂₄	204.19	99
63	69.38	1,4-dimethyl-Naphthalene	C ₁₂ H ₁₂	156.09	94
64	69.81	2,3-dimethyl-Naphthalene	C ₁₂ H ₁₂	156.09	91
65	71.28	1-(2-hydroxy-4-methoxyphenyl)-Ethanone	C ₉ H ₁₀ O ₃	166.06	91
66	72.81	2,6-bis(1,1-dimethylethyl)-2,5-Cyclohexadiene-1,4-Dione	C ₁₄ H ₂₀ O ₂	220.15	93
67	74.13	(1.alpha.,4a.alpha.,8a.alpha.)-1,2,3,4,4a,5,6,8a-octahydro-7-methyl-4-methylene-1-(1-methylethyl)-Naphthalene	C ₁₅ H ₂₄	204.19	91
68	74.46	1-(1,5-dimethyl-4-hexenyl)-4-methyl-Benzene	C ₁₅ H ₂₂	202.17	95
69	75.54	[1aR-(1a.alpha.,7.alpha.,7a.beta.,7b.alpha.)]-1a,2,3,5,6,7,7a,7b-octahydro-1,1,4,7-tetramethyl-1H-Cycloprop[e]azulene	C ₁₅ H ₂₄	204.19	90
70	75.79	Pentadecane	C ₁₅ H ₃₂	212.25	90
71	76.17	(1.alpha.,4a.alpha.,8a.alpha.)-1,2,4a,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-Naphthalene	C ₁₅ H ₂₄	204.19	96
72	76.33	Butylated Hydroxytoluene	C ₁₅ H ₂₆ O	220.18	94
73	76.78	(S)-1-methyl-4-(5-methyl-1-methylene-4-hexenyl)-Cyclohexene	C ₁₅ H ₂₄	204.19	93
74	77.46	(1.alpha.,4a.beta.,8a.alpha.)-1,2,3,4,4a,5,6,8a-octahydro-7-methyl-4-methylene-1-(1-methylethyl)-Naphthalene	C ₁₅ H ₂₄	204.19	94
75	77.79	[1S-(1.alpha.,4a.beta.,8a.alpha.)]-1,2,4a,5,8,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-Naphthalene	C ₁₅ H ₂₄	204.19	93
76	78.02	4-methoxy-6-(2-propenyl)-1,3-Benzodioxole	C ₁₁ H ₁₂ O ₃	192.08	98
77	79.49	(4aR-trans)-decahydro-4a-methyl-1-methylene-7-(1-methylethylidene)-Naphthalene	C ₁₅ H ₂₄	204.19	91
78	79.80	(Z)-1,2,4-trimethoxy-5-(1-propenyl)-Benzene	C ₁₂ H ₁₆ O ₃	208.11	89
79	80.72	[S-(Z)]-3,7,11-trimethyl-1,6,10-Dodecatrien-3-ol	C ₁₅ H ₂₆ O	222.20	90
80	81.04	1,2,3-trimethoxy-5-(2-propenyl)-Benzene	C ₁₂ H ₁₆ O ₃	208.11	94

续表 1

序号	保留时间(min)	化学成分	分子式	相对分子质量	相似度(%)
81	88.54	Patchouli alcohol	C ₁₅ H ₂₆ O	222.20	91
82	88.82	2',3',4'-Trimethoxyacetophenone	C ₁₁ H ₁₄ O ₄	210.09	90
83	89.04	Heptadecane	C ₁₇ H ₃₆	240.28	93
84	89.15	2,6,10,14-tetramethyl-Pentadecane	C ₁₉ H ₄₀	268.31	97
85	89.18	2,6,10-trimethyl-Hexadecane	C ₁₉ H ₄₀	268.31	91
86	92.12	Tetradecanoic acid	C ₁₄ H ₂₈ O ₂	228.21	93
87	93.91	Hexadecane	C ₁₆ H ₃₄	226.27	93
88	94.22	Anthracene	C ₁₄ H ₁₀	178.08	93
89	95.68	2-hydroxy-Cyclopentadecanone	C ₁₅ H ₂₈ O ₂	240.21	90
90	95.80	6,10,14-trimethyl-2-Pentadecanone	C ₁₈ H ₃₆ O	268.28	99
91	95.97	Oxacyclohexadecan-2-one	C ₁₅ H ₂₈ O ₂	240.21	91
92	96.62	Pentadecanoic acid	C ₁₅ H ₃₀ O ₂	242.22	97
93	99.20	Hexadecanoic acid, methyl ester	C ₁₇ H ₃₄ O ₂	270.26	95
94	99.24	9-Hexadecenoic acid	C ₁₆ H ₃₀ O ₂	254.22	99
95	99.99	Z-11-Hexadecenoic acid	C ₁₆ H ₃₀ O ₂	254.22	95
96	100.66	Dibutyl phthalate	C ₁₆ H ₂₂ O ₄	278.15	90
97	101.38	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	256.24	98
98	105.43	6,9-Octadecadienoic acid, methyl ester	C ₁₉ H ₃₄ O ₂	294.26	95
99	107.55	(Z,Z)-9,12-Octadecadienoic acid	C ₁₈ H ₃₂ O ₂	280.24	99
100	107.79	(Z,Z,Z)-9,12,15-Octadecatrienoic acid	C ₁₈ H ₃₀ O ₂	278.23	99
101	107.97	Oxacycloheptadec-8-en-2-one	C ₁₆ H ₂₈ O	252.21	90
81	88.54	Patchouli alcohol	C ₁₅ H ₂₆ O	222.20	91
82	88.82	2',3',4'-Trimethoxyacetophenone	C ₁₁ H ₁₄ O ₄	210.09	90
83	89.04	Heptadecane	C ₁₇ H ₃₆	240.28	93
84	89.15	2,6,10,14-tetramethyl-Pentadecane	C ₁₉ H ₄₀	268.31	97
85	89.18	2,6,10-trimethyl-Hexadecane	C ₁₉ H ₄₀	268.31	91
86	92.12	Tetradecanoic acid	C ₁₄ H ₂₈ O ₂	228.21	93
87	93.91	Hexadecane	C ₁₆ H ₃₄	226.27	93
88	94.22	Anthracene	C ₁₄ H ₁₀	178.08	93
89	95.68	2-hydroxy-Cyclopentadecanone	C ₁₅ H ₂₈ O ₂	240.21	90
90	95.80	6,10,14-trimethyl-2-Pentadecanone	C ₁₈ H ₃₆ O	268.28	99
91	95.97	Oxacyclohexadecan-2-one	C ₁₅ H ₂₈ O ₂	240.21	91
92	96.62	Pentadecanoic acid	C ₁₅ H ₃₀ O ₂	242.22	97
93	99.20	Hexadecanoic acid, methyl ester	C ₁₇ H ₃₄ O ₂	270.26	95
94	99.24	9-Hexadecenoic acid	C ₁₆ H ₃₀ O ₂	254.22	99
95	99.99	Z-11-Hexadecenoic acid	C ₁₆ H ₃₀ O ₂	254.22	95
96	100.66	Dibutyl phthalate	C ₁₆ H ₂₂ O ₄	278.15	90
97	101.38	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	256.24	98
98	105.43	6,9-Octadecadienoic acid, methyl ester	C ₁₉ H ₃₄ O ₂	294.26	95
99	107.55	(Z,Z)-9,12-Octadecadienoic acid	C ₁₈ H ₃₂ O ₂	280.24	99
100	107.79	(Z,Z,Z)-9,12,15-Octadecatrienoic acid	C ₁₈ H ₃₀ O ₂	278.23	99
101	107.97	Oxacycloheptadec-8-en-2-one	C ₁₆ H ₂₈ O	252.21	90

以图中两个含量最高的峰的判定为例,将 101.38min 时的实际质谱图与软件自带谱库中的谱图比较(图 4)可以看出,上面的实际质谱图与下面的 *n*-十六烷酸的标准谱图无论在碎片信息还是在碎片丰度的比例上都十分吻合,而且其在 *m/z* 为 60 的位置这一直链脂肪酸的特征碎片离子有较强的碎片因此判定该化合物为 *n*-十六烷酸。

同理,比较 107.55min 的实际质谱图与软件自带谱库中的谱图比较(图 5)可以判定出,此化合物为(*Z,Z*)-9,12-十八烷酸。

结果表明,青风藤挥发油中含有多种化合物,其主要化学成分为脂肪酸。根据质谱库中的数据和实际谱图的比较,推断含量最高的两个色谱峰分别是 101.38min 时的 *n*-十六烷酸和 107.55min 时的 9,12-十八烷酸。目前国内尚没有对青风藤挥发油化学成分的研究,此研究方法为青风藤的物质基础研究和进一步的开发应用提供了实验依据。

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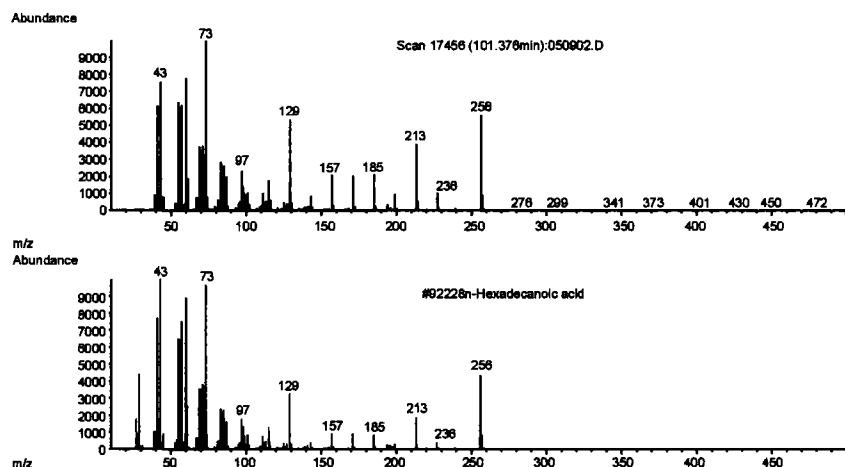


图 4 101.38 分钟时的实际质谱图与谱库中的标准谱图

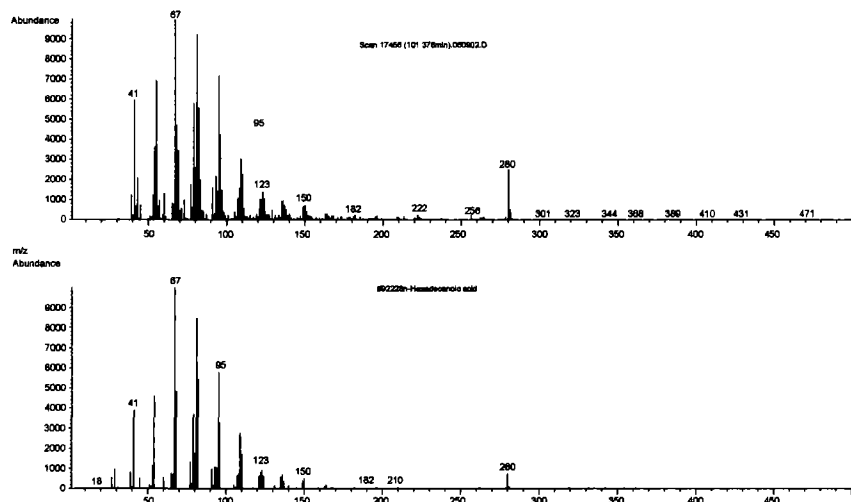


图 5 107.55 分钟时的质谱图与谱库中的标准谱图

Study on quantitative classification and regionalization of *Panax notoginseng* based on TCMGIS- I *

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Appropriate producing area of *Panax notoginseng* in China was evaluated by TCMGIS- I system (The geographic information system of suitable producing area evaluation of Traditional Chinese Medicine), basing on the ecological factors of Wenshan country in Yunnan province. The results showed the suitable producing areas of *Panax notoginseng* in China distribute widely. But the distribution is centralized in the southeastern of Yunnan province, northwestern of Guangxi province, or the southeastern of Guizhou. The complex planting technologies such as transplanting, construction shed for shade in the planting procedure account for only several areas in Yunnan and Guangxi province being the main producing areas of *Panax notoginseng*. Thus, social factors play an important role in the development of famous-region of *Panax notoginseng*. The result is an important reference to the development of *Panax notoginseng* cultivation.

Keywords: TCMGIS- I ; Suitable producing area; *Panax notoginseng*

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Analysis of Volatile Oils of *Caulis Sinomenii* by Gas Chromatography-Mass Spectrometry

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Abstract: The volatile oil of *Caulis Sinomenii* was extracted using the method mentioned in Chinese Pharmacopoeia. Adapting the method gas chromatography-mass spectrometry, more than 200 peaks were separated and 101 peaks were identified by comparing the standard mass spectrometry in the software and the experimental mass spectrometry. And the two main compents were identified as n-Hexadecanoic acid and (Z,Z)-9,12-Octadecadienoic acid respectively. This experiment provides the experimental gist of the exploration and study for *Caulis Sinomenii*.

Keywords: *Caulis Sinomenii*; volatile oil; gas chromatography-mass spectrometry

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