

# “副鼻窦冲剂”中川芎嗪的HPLC法测定

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**摘要** 副鼻窦冲剂其主药为川芎。本文应用HPLC法测定了该制剂中川芎嗪的含量,利用YWG-C<sub>18</sub>柱,检测波长为292nm,流动相为甲醇-水(52:48),以无水乙醇溶解样品,过滤后进样。操作简便、快速、准确、重现性好,实验结果:回收率为99.2%,RSD为1.1%(n=4)最低检测量为0.5μg/ml。

**关键词** 反相高效液相色谱法 “副鼻窦冲剂” 川芎嗪

“副鼻窦冲剂”是本院研制的一种新中药制剂,具有清热解毒、消炎之功效。临床用于急、慢性副鼻窦炎,鼻窦炎的治疗收到良好效果。该药主要由川芎、细辛、连翘、蒲公英等多味中药组成,川芎是其主药。作者应用HPLC法对制剂中川芎嗪的含量进行了测定<sup>[1,2]</sup>,结果表明:该法操作简便、快速、准确、重现性好。

## 1 仪器与试剂

**仪器:**日本岛津LC-4A高效液相色谱仪,紫外检测器SPD-2AS,数据处理仪C-R2AX。

**试剂:**川芎嗪,北京制药工业研究所。“副鼻窦冲剂”由本院中药制剂室提供。甲醇为分析纯。

## 2 色谱条件

ODS柱,10μm(填料),色谱柱(4mm×250mm),柱温25℃,流动相为甲醇-水(52:48),流速为1ml/min,检测波长为292nm,灵敏度0.08AUFS,纸速3mm/min。

## 3 标准曲线的制作

精密称取川芎嗪对照品约2.5mg置25ml量瓶中,加甲醇至刻度,摇匀,得原液(0.1mg/ml),再分别吸取原液0.02、0.04、0.08、0.16ml置10ml量瓶中,加甲醇至足量,得不同浓度之标准液。在上述条件下分别进样(15μl)分析,所得峰高与浓度作图,得一线性较好的标准曲线,回归方程为 $Y = -0.4247 + 22.0X$ ,  $r = 0.9993$ 。

## 4 样品含量测定

精密称取不同批号之样品各5g,分别加无水乙醇(12,10ml)溶解,并过滤至25ml量瓶中,加无水乙醇至刻度,摇匀。分析前以微孔滤膜过滤器(0.45μm)过滤后进样分析,测得峰高并计算其含量,结果见表。

## 5 回收率及精密度试验

**回收率:**按处方量制备不含川芎的空白冲剂,并各称取5g,分别以3种已知浓度的川芎嗪无水乙醇溶液溶解、过滤至25ml量瓶中,后操作同样品测定。结果:平均回收率为99.2%,RSD为1.1%(n=4)。

**精密度实验:**取2种不同含量(0.02,0.06mg/ml)的对照品溶液和一批号(930226)的样品液在上述条件下同日及一周内各测定6次,计算得日内和日间RSD分别为1.09、0.86

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表2 1989年内桂剥皮后不同处理再生情况比较

处 理	不 同 高 度 再 生 ( % )					与CK比较再 生皮数量 ( % )	注
	10cm	50cm	100cm	合 计	平 均		
剥皮1/2涂NAA50	78.57	51.28	45.62	175.47	58.49	101.25	为12月观察 平均数。 “包”表示 剥皮后包塑 料薄膜
剥皮1/2涂NAA100	70.80	62.44	71.25	204.49	68.16	117.99	
剥皮1/2涂6-BA50	36.66	36.38	42.68	115.71	38.57	66.77	
剥皮1/2涂6-BA100	53.89	44.78	46.75	145.42	48.47	83.90	
剥皮1/2涂京第2B	41.64	54.90	55.60	152.14	50.71	87.78	
膜剂30倍液							
剥皮1/2涂京第2B	58.24	38.79	47.46	149.49	49.83	86.26	
膜剂60倍液							
剥皮1/2不涂药剂 (CK)	57.12	52.20	64.00	173.32	57.77	100.00	
剥皮1/2涂NAA50,包	95.77	100.00	82.86	278.62	92.87	134.07	
剥皮1/2涂NAA100,包	68.84	66.82	50.36	186.02	62.01	89.52	
剥皮1/2涂6-BA50,包	60.63	53.05	50.67	164.35	54.79	79.10	
剥皮1/2涂6-BA100,包	88.60	46.17	54.85	189.63	63.21	91.25	
剥皮1/2涂京第2B膜剂30 倍液,包	68.18	71.06	75.84	215.07	71.69	103.49	
剥皮1/2涂京第2B膜剂60 倍液,包	79.86	43.49	100.00	222.34	74.11	106.99	
剥皮1/2不涂药剂, 包(CK)	68.06	65.47	74.29	207.81	69.27	100.00	

2.2.2 剥皮后再生皮大多是从两面切口形成层细胞分裂,向中间延伸的 新生组织,1~2年的再生皮的厚度可达0.1~0.2cm,但肉桂特有的香气和甜辣味淡薄。同时发现树干下部剥皮面新皮再生较好,向上延伸,再生皮逐渐减少,再生皮的多少与高度成反比例。

2.2.3 1988年试验结果以剥皮1/2、涂6-BA100mg/L较好,再生皮为剥皮面的79.10%,1989年则以剥皮1/2+涂药+包薄膜的好,其中以涂NAA50mg/L+包薄膜处理,再生皮高达92.87%,保持剥皮面的适当湿度,是提高肉桂皮再生量的关键之一。

### 3 讨论

上述结果证明肉桂皮采剥后,有较好的再生力,通过涂生长素和包扎塑料薄膜可以提高树皮的再生率,但对肉桂皮再生的生理机制和再生皮中有效成分的积累过程,以及肉桂剥皮后再生在生产经营上是否有效益,尚待进一步研究。

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及1.96%。最低检测量为0.5μg/ml。

### 6 小结

6.1 本文对文献<sup>[1, 2]</sup>的方法进行了改进,应用甲醇-水(52:48)为流动相,样品以无水乙醇溶解、过滤后进样。方法简便、快速、准确、重现性好。

6.2 空白冲剂分析结果表明,制剂中其他成分不干扰川芎嗪的含量测定。

6.3 该制剂为本院制剂,临床使用数年,治疗急、慢性付鼻窦炎效果较好。

### 参 考 文 献

1 赵志春,等.中国中药杂志,1991,16(12):

2 蔡伟.药学报,1989,24(12):886

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# ABSTRACTS OF ORIGINAL ARTICLES

## Studies on the Chemical Constituents of the Fresh Inflorescences of Desertliving Cistanche (*Cistanche deserticola*)

Tu Pengfei, He Yanping and Lou Zhicen

Four known compounds are isolated from the butanol fraction of the fresh inflorescences of *Cistanche deserticola* Ma (*Orobanchaceae*) for the first time. They are identified as 6-deoxycatalpol (I), liriiodendrin (II), 3-epiloganic acid (III) and galactitol (IV). These constituents are similar to the main constituents of the dried fleshy stem. It is suggested that the inflorescence may be exploited as a substitute for herb *C. deserticola* Ma.

(Original article on page 451)

## Studies on the Alkaloids of shezushishan (*Huperzia serrata*)

Yuan Shanqin, Feng Rui and Gu Guoming

Three alkaloids were isolated from *Huperzia serrata* (Thunb.) Trev. Their structure were identified as 8-deoxyserratinine (I), lycodine (II) and phlegmariurine B (III) by means of spectral analysis (UV, IR, NMR, MS). Compound II and III were isolated for the first time from this species.

(Original article on page 453)

## Studies on the Chemical Constituents of Common Leafy Flower (*Phyllanthus urinaria*)

Wan Zhenxian, Zhou Guoping and Yi Yanghua

Six compounds were isolated from the whole herb of *Phyllanthus urinaria* L. They were identified as ellagic acid (I), 3, 3', 4-tri-O-methylellagic acid (II), succinic acid (III), ferulic (IV),  $\beta$ -sitosterol-glucoside (V) and gallic acid (VI) by means of spectral analysis and chemical reaction. Compound II, III, IV, and V were isolated for the first time from this plant.

(Original article on page 455)

## Studies on the Quality Standard of Shenrongsanshen Medicinal Wine

Yang Guohong

Quality standard of Shenrongsanshen medicinal wine was discussed. A TLC method was used in the identification of the chemical constituents of some of the medicinal herbs used in the wine, such as, fruit of *Lycium chinense* Mill. fruit of *Psoralea corylifolia* L., rhizome of *Vladimiria souliei* (Franch.) Ling. A HPLC method was used in the determination of icariine. The average recovery was 99.87%, RSD=0.04% (n=5).

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## High Performance Liquid Chromatographic Assay of Tetramethylpyrazine in "Fubidou Granule"

Jiang Zhirong

Fubider Granule is a new Chinese herbal preparation. It consists of Rizoma Chuansiong, Fructus Forsythiae and Herba Asari. It is used for the treatment of acute and subacute paranasal sinusitis. This work reports a new HPLC method for the determination of tetramethylpyrazine in the chinese herbal preparation. A reverse phase column  $C_{18}$  was used for the separation with methanol-water (52:48) at 1.0ml/min as eluent and measured by UV detector at 292nm. The extraction recovery was 99.2%, RSD was 1.1% ( $n=4$ ) and the sensitivity limit of quantitative analysis was 0.5 $\mu$ g/ml. The assay is simple, rapid and sensitive with good reproduction.

( Original article on page 459 )

## Experimental Studies on the Effect of "Liver-Softening Anti-Fibrotic Decoction" on Active and Inactive Fibrotic Rats

Jing Shugen, Wang Lingtai, Ren Jiawei, et al

"Liver-Softening Anti-Fibrotic Decoction", a preparation consisted mainly of medicinal herbs with tonifying "Qi", flourishing "Yin", activating blood and eliminating stasis, was given by gavage to Dimethyl nitrosamine (DMN) induced fibrotic rat to assess its effect on active and inactive liver fibrosis. It was found that this preparation, besides its promoting growth and protein metabolism activities, can also lower serum alkaline phosphatase, decrease serum hyaluronate and liver hydroxy proline, with a better curative effect on experimental liver fibrosis in rats than its prophylactic effect.

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## Effect of Lingzhi (*Ganoderma lucidum*) on Water-Immersion Stress Ulcer in Mice and Its Antagonism to Acetylcholine in Vitro

Cheng Zhanghua, Masao Mori, et al

Mice were pretreated po with 0.4g/kg, 1.0g/kg, 2.0g/kg Lingzhi aqueous extract once daily for 3 days respectively. 1h after the last dose, they were kept under restraint plus water-immersion stress for 22h. Lingzhi 1.0g/kg, 2.0g/kg, and atropine 0.04g/kg markedly decreased ulcer formation and hemorrhage incidences compared with control ( $P<0.01$ ). Lingzhi in different dosages could produced by acetylcholine. Its  $IC_{50}$  was found to be  $8.5 \times 10^{-4}$ g/ml. Ulcer formation under stress condition is due to stimulation of the excessive central hypothalamus and parasympathetic nervous system; especially vagal overactivity plays an important role. The results suggest that Lingzhi possesses a blocking effects on peripheral para-sympathetic nervous system.

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## Effect of Ginseng and Angelica Sinensis Decoction (GASD) on Learning and Memory of Dementia Rat with Hippocampal Lesions Induced by Quinolinic Acid

Song Qianliu, Zong Ruiyi and Xie Xianglin