

# 玉清安液中冰片的含量测定研究

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**摘要** 用气相色谱法测定玉清安液中冰片含量。以乙酸乙酯为提取液,正十四烷为内标,分别用10%OV-17和10%PEG-20M2种填充柱,可以产生不同峰形的分析结果。

**关键词** 玉清安液 冰片 OV-17柱 PEG-20M柱

玉清安液是妇科清热消炎止痒的内阴用药,是由赤芍、黄柏、冰片等多味中药精制而成的粘稠液体制剂,粘稠度 $80\text{mP}\cdot\text{s}$ 左右。在研制过程中,同时对该品种的冰片质量标准研究,采用气相色谱法测定粘性液体冰片含量,对OV-17色谱柱与PEG-20M色谱柱进行了比较。

## 1 仪器和材料

**仪器:** HP-5890A气相色谱仪、HP-3390A积分仪、HP 1 $\mu\text{l}$ 进样器及METTLER AE 240电子天平。

**玉清安液:** 由无锡新兴制药有限公司提供;冰片对照品:购自中国药品生物制品检定所,批号743-8902;内标物正十四烷:色谱纯;溶剂乙酸乙酯:分析纯。

## 2 色谱条件

**2.1 色谱柱:** HP-10% OV-17 Chromosorb WHP  $2\text{m}\times 2\text{mm}$  (ID) 不锈钢柱(简称OV-17柱),柱温 $170^{\circ}\text{C}$ ,汽化室温度 $230^{\circ}\text{C}$ ,检测室温度 $210^{\circ}\text{C}$ ,FID,氮气压力 $250\text{kPa}$ ,进样量 $0.5\mu\text{l}$ ,积分仪纸速 $0.3\text{cm}/\text{min}$ ,选择适当的峰形处理参数与计算参数。

**2.2 色谱柱:** HP-10% PEG-20M Chromosorb WHP  $2\text{m}\times 2\text{mm}$  (ID) 不锈钢柱(简称PEG-20M柱),柱温 $155^{\circ}\text{C}$ ,汽化室温度 $210^{\circ}\text{C}$ ,检测室温度 $200^{\circ}\text{C}$ ,其它条件同OV-17柱。

## 3 溶液制备

**3.1 内标溶液:** 取正十四烷适量,加乙酸乙酯溶解并稀释成0.2%(V/V)的溶液。

**3.2 标准品溶液:** 取冰片约20mg,精密称定,加乙酸乙酯溶解并稀释至每1ml含2.0mg的溶液。

**3.3 对照溶液:** 取内标溶液和标准品溶液各1.0ml,混匀。

**3.4 供试品溶液:** 取样品原液50ml,减量法精密称定,准确加入2.5ml乙酸乙酯,在分液漏斗中振摇10min,静置30min,待分层清晰后,取上层液1.0ml及内标溶液1.0ml,混匀。

## 4 线性试验

精密称定冰片对照品5份,各用内标溶液准确稀释至1ml,分别用OV-17柱及PEG-20M柱色谱分析,以冰片浓度(mg/ml)为X,以冰片与内标物的峰面积比为Y,回归方程OV-17柱  $Y = 0.6323X - 0.1218$ ,  $r = 0.9989$ ; PEG-20M柱  $Y = 0.6354X - 0.08821$ ,  $r = 0.9999$ 。

## 5 系统适用性试验

取80%、100%、120% 3组冰片标准品溶液,按对照溶液制备法制备,各进样3次。

OV-17柱,正十四烷保留时间3.40min;冰片保留时间2.64min,其理论板数为1110;正十四烷与冰片分离度2.2;校正因子0.8502, CV = 1.1%。色谱图见图。

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PEG-20M柱, 正十四烷保留时间2.05 min, 异龙脑保留时间5.92min, 龙脑保留时间6.6min; 理论板数以龙脑峰计为2170; 正十四烷与异龙脑的分离度大于3, 异龙脑与龙脑的分离度为1.3; 校正因子(异龙脑和龙脑峰面积之和计算)为0.8617, CV = 0.4%。色谱图见图。

6 回收率试验

准备3份不含冰片的玉清安液50g, 各准确加入50mg冰片对照品, 再加入25 ml乙酸乙酯, 振摇, 混匀, 按供试品溶液制备, OV-17色谱柱分析, 内标法计算含量, 结果平均回收率为95.4%, CV = 0.83%。

7 样品测定

按供试品溶液方法制备, 采用OV-17柱(批号930810, 第2份样品采用PEG-20M柱分析)各进样3次, 内标法计算含量。结果见表。

8 干扰试验

由厂方提供的无冰片玉清安液及单味药液, 按供试品溶液方法制备, 用OV-17色谱柱, 无干扰峰出现。

9 讨论

冰片作为妇科外用制剂组分之一, 应控制其含量, 既确保起到协同作用, 又避免不良刺激。OV-17柱在冰片含量分析中, 能以单峰形式出现, 即异龙脑与龙脑在同一范围内出峰, 分析时间短, 便于计算; 当改变柱温或载气流速, 异龙脑与龙脑能分开, 但不理想。PEG-20M柱使异龙脑与龙脑较为理想地分开〔陆善松. 现代应用药学, 1987, 4(4): 13〕, 这对考察冰片质量有参考价值〔王强, 等. 中草药, 1994, 25(5): 241〕。

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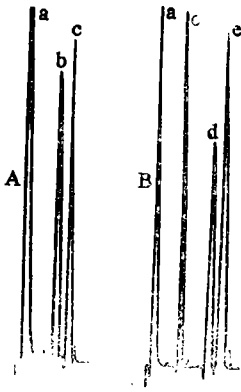


图 冰片色谱图  
A-OV-17柱 a-乙酸乙酯 b-冰片 c-正十四烷  
B-PEG-20M柱 d-异龙脑 e-龙脑

表 样品中冰片含量测定结果

批号	冰片含量 (g/g)	CV (%)
930810	0.097 0.103	0.6 0.6
940201	0.120	0.8
940202	0.109	1.9

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# ABSTRACTS OF ORIGINAL ARTICLES

## Studies on the Chemical Constituents of *Sargentodoxa yunnanensis*

(*Sargentodoxa cuneata*)

Miao Kangli, Zhang Jianzhong, Wang Feiyin, et al

Six compounds were isolated from the EtOH extract of stems of *Sargentodoxa cuneata*. They were identified as  $\beta$ -sitosterol (I),  $\beta$ -daucosterol (II), madasiatic acid (III), acanthoside D (IV), sargencuneside (V), and sucrose (VI) on the basis of chemical and spectral analyses. Among them, V is a new compound.

(Original article on page 171)

## A Study on Polysaccharide of the Hongshier (*Umbilicaria hypococeinea*)

Ding Dongning, Yan Baoqi, et al

UMH polysaccharide was isolated and purified with alcohol precipitation from hot water extract of *Umbilicaria hypococeinea* Lian. By Sephadex G-150 column chromatography, UMH was shown to be a single homogeneous substance, sugar content 90.4%. By gas chromatography analysis, UMH was composed of glucose, mannose and glucuronic acid, their molecule ratio was about 45:1:9. Its mean molecular weight was estimated to be  $40 \times 10^4$ . IR analysis, periodate oxidation and Smith degradation showed that the main chain of UMH is composed, of  $\alpha(1 \rightarrow 6)$  and  $(1 \rightarrow 4)$  linkage, and was an acidic hetro-saccharide.

(Original article on page 175)

## Studies on the Chemical Constituents Bigflower Rhodiola (*Rhodiola crenulata*)

Peng Jiangnan, Ma Chengyu, Ge Yongchao, et al

Five compounds were isolated from the rhizome and roots of *Rhodiola crenulata* S.H.Fu. They were identified as rhodionin (I), rhodiosin (II), tyrosol (III), salidroside (IV) and gallic acid (V), respectively, by UV, MS,  $^1\text{H}$  and  $^{13}\text{C}$ NMR spectroscopic and chemical reactions.

(Original article on page 177)

## Determination of Glycyrrhizic Acid and Chlorogenic Acid by HPLC

Rong Zhifen, Zhang Weiqing, Hu Wenjie, et al

A method for the determination of glycyrrhizic acid and chlorogenic acid in Chinese medicinal preparation by Rp-HPLC-UV was described. Mobile phase: methanol (0.5% HAc) :  $\text{H}_2\text{O}$  (0.5% HAc);  $\text{C}_{18}$  column and external standard was used.

(Original article on page 181)

## Quantitative Determination of Borneol in Yuqingan Liquid

Wei Xiaoshu

Borneol in Yuqingan liquid was determined quantitatively by GC. The sample was first extracted by ethyl acetate and component determined by two types of column packed with 10% OV-17 and 10% PEG-20M/chromosorb WHP respectively. N-tetradecane was used as an

## Determination of Tanshenoside I in Taidangshen (*Codonopsis pilosula*) and Cultivated Ludangshen (*C. pilosula*) of Different Years Old

Dai Jing, Feng Li, Han Guiru et al

Amounts of tanshenoside I in cultivated Ludangshen of different years of cultivation history was determined by TLC densitometry in comparison with that in wildly grown Ludangshen and cultivated Taidangshen. Results showed that tanshenoside I in cultivated Ludangshen decreases with increased years of cultivation history while that in cultivated Taidangshen is slightly lower than that of Ludangshen of the same years of cultivation. Tanshenoside I in wild Taidangshen is also lower than that in cultivated Taidangshen.

( Original article on page 185 )

## Study on Biological Effects of Songzike Acidic Polysaccharide

Hou Fangyu, Yu Qinghua, et al

The biological effects of Songzike acidic polysaccharide (ST90) were studied in vivo and vitro. It was found that ST90 had distinct antineoplastic, antibacterial and antiviral effects. It markedly inhibits proliferation of S180 solid tumor in mice and reduces the mortality of mice infected by *Salmonella typhimurium*, and shows a protective effect on FL cells infected with  $adv_1$ ,  $adv_2$ , vsv and  $CB_4$ .

( Original article on page 193 )

## Protective Effect of Tea Polyphenol on Rat Myocardial Injury Induced by Isoproterenol

Tang Shengxing, Ye Ting, Zhao Zhengdong

Retreatment with tea polyphenol (TP) at a dose of 10mg/kg ip to rats five days before isoproterenol (ISO) challenge (1mg/kg sc, for two days), resulted in decreases of malondialdehyde concentration, creatine phosphokinase, lactic dehydrogenase (LDH) and  $LDH_1$  activities, increased  $LDH_2/LDH_1$  ratio and inhibited the extent of myocardial injury, similar to the action of propranol. At the same time TP decreased rat plasma renin activity. The results suggested that the mechanism by which TP protects heart from ISO-induced myocardial injury is due to its antioxygen free radical and inhibition of renin activities.

( Original article on page 197 )

## Effects of Emodin on the Cytoplasmic free $Ca^{2+}$ in Peritoneal Macrophages From Mice

Cui Rongfen, Lin Xiuzhen