

梔子及其口服液中梔子甙的含量测定

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摘要 用高效GF₂₅₄薄层板分离梔子甙,单波长线性扫描法测定梔子及其制剂中梔子甙的含量,方法简便,重现性好。

关键词 梔子 梔子甙 薄层扫描 含量测定

梔子是茜草科植物*Gardenia jasminoides* Ellis的干燥果实,有泻火除烦,清热利尿,凉血解毒的功能。梔子中的有效成分梔子甙(geniposide)具有促进胆汁分泌,缓泻作用。本实验用丙酮-乙酸乙酯-甲酸-水作为展开剂,并用单波长线性扫描方法测定梔子甙含量,方法简单,重现性好,适用于梔子及口服液制剂的含量测定。

1 仪器与试剂

薄层扫描仪(日本岛津CS-930型),SB-乙型薄层色谱仪紫外检测器(天津市分析仪器厂),高效硅胶GF₂₅₄薄层板(10×10cm,青岛海洋化工厂分厂),梔子甙标准品(中国药品生物制品检定所),试剂均为分析纯。

2 实验方法

2.1 标准曲线绘制:精密称取梔子甙标准品,用乙醇制成2mg/ml的标准品溶液,在高效薄层GF₂₅₄板上点样0.5、1、2、3、4、5μl,以丙酮-乙酸乙酯-甲酸-水(5:5:1:1)为展开剂,展开,展距7cm,取出,晾干,置紫外灯下254nm处,观察荧光猝灭斑点的位置,并作标记(图1),在CS-930型扫描仪上进行扫描。扫描条件:波长248nm,狭缝0.2×6mm, S_x = 3,纸速和扫描速度为56mm/min,反射法线性扫描,得线性方程:

$Y = 0.3585X + 0.0243, r = 0.999$,线性范围:1~10μg。

2.2 精密度试验:在同一块薄层板上,精密点7个相同量的梔子甙斑点,依上法展开,测定斑点面积积分值,7个点的面积积分值 $X = 0.486, S_x = 0.0278, CV = 1.72\%$ 。

2.3 稳定性试验:吸取梔子甙标准品的乙醇溶液1、2、3μl点样、展开,在相同条件下,于不同时间,分别测定斑点的积分值,结果表明,斑点在24h内稳定。

2.4 加样回收率试验:精密吸取口服液50μl,加入梔子甙标准品溶液100μl(1.8mg/ml),定容,点样,展开测定,以未加标准品的样品含量为对照,计算回收率,结果为99.39%, CV = 1.48% (n = 5)。

2.5 阴性加标准品回收率试验:按处方及工艺配制不含梔子的阴性样。精密吸取阴性样100μl,加入标准液100μl(2.0mg/ml)定容,点样,展开测定,同时以未加标准品的阴性样为对照,计算回收率,结果为98.70% (n = 5), CV = 4.72%。

2.6 样品的含量测定:精密吸取口服液1ml,用95%乙醇定容到5ml,作为供试品。吸取供试品溶液1μl,标准对照品溶液1、2、3μl,分别点在同一高效硅胶GF₂₅₄薄层板上,以上述

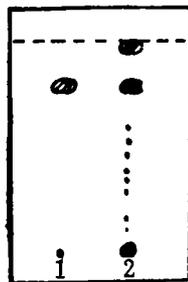


图 薄层层析图

1-标准品 2-样品

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展开剂展开,测定含量,3批的测定结果见表。

表 3批样品中栀子甙含量测定结果

样品批号	含量(g/支,每支按10ml计算)				\bar{x}	SD	CV%
	1	2	3	4			
930729	0.1347	0.1314	0.1362	0.1396	0.1356	0.0034	2.51
930818	0.1530	0.1445	0.1436	0.1419	0.1458	0.0050	3.40
930909	0.1349	0.1330	0.1374	0.1409	0.1366	0.0034	2.50

2.7 栀子原料含量测定:分别采用水煎法、70%乙醇冷渗、索氏提取法(均提取4h),对栀子药材中栀子甙进行含量测定,结果分别为4.23%(栀子甙, g/100g药材)、1.73%和4.49%。

3 讨论

3.1 本实验采用的展开剂,系统稳定,展开效果好,栀子甙斑点集中,没有背景干扰。克服了以往采用氯仿-甲醇-氨水、甲苯-甲醇-冰乙酸等系统所产生的背景干扰和重复性差的缺点。

3.2 栀子甙的结构中有一个“-COO-”基团,它与环烯醚中的双键形成 α, β -不饱和的羰基,在UV中235nm左右应出现最大吸收峰,根据光谱扫描,栀子甙的吸收波长为248nm。

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羊角七外涂治疗9例带状疱疹的疗效观察

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羊角七又名草乌、金牛七、蔓乌头、千锤打、火焰子等。为毛茛科乌头属松潘乌头 *Aconitum sunpanense* Hand.-Mazz.的块根。味辛微苦,性温,有大毒。具有止痛,解痉,麻醉,败毒,去风湿,活血散瘀等功能。近年来,我们采用醋磨浆外涂方法,治疗带状疱疹起到了明显效果,并对其机理作了进一步研究。

1 临床资料

带状疱疹是一种病毒性皮肤病,中医称为缠腰火丹,蛇串疮,俗名蜘蛛疮等名。采用羊角七加食醋磨浆外涂方法,治疗带状疱疹患者,作用明显,安全简便,用药30min左右疼痛即可减轻或消失;轻者当天好转,慢者一周治愈和基本治愈。9例患者均为成年人,其中男4人,女5人。用药1d水疱干涸,红肿及皮疹消退者6例,2d1例,3d2例,均在8d内脱痂痊愈。平均治愈为4.5d,治愈率为100%。

2 治疗方法

取羊角七(生品),粗制碗(最好土制碗),将普通食醋倒入碗内用羊角七磨成糊状,或者把生羊角

七研成细末,过80~100目细筛备用(越细越好)。用时再将羊角七粉同食醋调成糊状,用清洁毛笔或棉签以及鸡、鹅羽毛蘸药糊涂患部,轻者每日2~3次,较重者每日4~5次。涂药后敞露患部,待药糊自行干燥。

3 诊断要点

3.1 沿外围皮神经突然多数绿豆大小水疱,簇集成群疱,膜紧张,基底炎症显著,多数簇集水疱群可排列呈带状,自觉有局部灼痛,淋巴结可肿大。严重者可发热或呈现大疱,血疱或坏疽。

3.2 发病急,有自愈性,病程约1~3周,愈后不复发。但神经痛往往可持续较久。特别是老年患者。

4 疗效标准

4.1 痊愈:水疱干涸吸收消退,自觉症状消失或基本消失。

4.2 减轻:水疱趋向干涸,炎症明显减轻,自觉症状减轻。

4.3 无效:经过1~2周左右治疗,症状仍未明

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ABSTRACTS OF ORIGINAL ARTICLES

Studies on the Chemical Constituents of Korean Monkshood

(*Aconitum coreanum*)

Han Ying, Liu Jinghan, et al

The compounds were isolated from *Aconitum coreanum* (Levl.) Rapaics and identified by TLC, IR, MS and NMR, as β -sitosterol (I), daucosterol (II), Guan-Fu base A (III), Guan-Fu base G (IV), condelphine (V), Guan-Fu base I (VI), hetisinone (VII), isoatisinum chloride (VIII), atisinum chloride (IX), and Guan-Fu base z (X) from the root. Compounds III and IX were also found in the aerial parts of the plant. Compounds II, V, VII and VIII were obtained for the first time from this plant. Most of them exhibited antiarrhythmic activity.

(Original article on page 619)

Studies on Alkaloid and Flavonoids from Roxburgh Wormwood

(*Artemisia roxburghiana*)

Li Yu, Hu Youhua, and Shi Yanping

One alkaloid was isolated for the first time from *Artemisia* genus and four flavonoids also for the first time, from *A. roxburghiana* Wall. They were identified as N-phenyl-2-naphthylamine, penduletin, quercetin, 3,3',4'-trimethyl ether, eupatilin, jaceosidin by their spectroscopic data (MS, ¹HNMR, UV).

(Original article on page 622)

Determination of Geniposide in Cape Jasmine (*Gardenia jasminoides*) and Its Oral Liquid

Zhou Lihong, Zhong Shuhua

Isolation of geniposide in *Gardenia jasminoides* was accomplished by high performance GF₂₅₄ TLC and the content of the said component in the herb and its preparation determined by linear scanning at λ 248nm. The method was simple and accurate.

(Original article on page 627)

Determination of Three Bisbenzylisoquinoline Alkaloids in Fourstamen *Stephania* (*Stephania tetrandra*) by HPLC

He Liyi, Sagaraka Kazuhiko, et al

A simple and precise method using high-performance liquid chromatography was developed for the simultaneous determination of three bisbenzylisoquinoline alkaloids, namely tetrandrine, demethyltetrandrine and berbamine, in *Stephania tetrandra* S. Moore. A reversed-phase system consisting of chemically bonded ODS silica gel column and methanol-acetonitrile-H₂O (3:1:1) containing 0.06% diethylamine as the mobile phase was used. The three alkaloids were completely separated within 10 min. The analytical results for various samples were presented.

(Original article on page 629)