薄层扫描法测定五灵丸中五味酯甲、丹参酮ⅡA的含量

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摘要 采用薄层扫描法测定了五灵丸中五味酯甲与丹参酮ⅡA的含量，回收率分别为98.34%，CV=2.1%；99.15%，CV=1.13%。本法具有快速、简便、灵敏、重现性好的特点。

关键词 薄层扫描法 五味酯甲 丹参酮ⅡA

五灵丸由五味子、灵芝、丹参等中药组成，具有补肝益脾，改善肝血循环，减轻肝纤维化与脂变，调节免疫的功能。主治急性病毒性肝炎、慢性迁延性肝炎及其他原因所致的转氨酶升高。本文用薄层扫描法测定五灵丸中有效成分五味酯甲、丹参酮ⅡA的含量，方法重现性好，简便灵敏、迅速，为该药内在质量控制提供有价值的参考。

实验部分

1.1 仪器与药品：日本岛津CS-9000型薄层扫描仪；硅胶及硅胶G薄层板，烟台化工科技开发试验厂；微量定量点样管，美国；CQ50超声波清洗器，上海；五味酯甲由陕西省药检所提供；丹参酮ⅡA购自中国药品生物制品检定所，五灵丸由本科自制；试剂均为分析纯。

1.2 实验条件的选择

1.2.1 对照液制备：精密称取五味酯甲3.4mg，置5ml容量瓶中，以甲醇溶解并稀释至刻度，为0.68mg/g。

精密称取丹参酮ⅡA 0.7mg，置2ml容量瓶中以氯仿溶解并稀释至刻度，为0.35mg/ml。

1.2.2 薄层条件：五味酯甲用硅胶GF_{254}板（10×10cm）以环己烷-乙酸乙酯（7∶4）展开，层析结果在紫外灯254nm下观察，为无荧光斑点，见图。丹参酮ⅡA用硅胶G板（10×10cm），以苯-氯仿-丙酮（5∶4∶0.5）展开，自然光下显红色斑点，见图。

1.2.3 扫描波长及有关仪器参数确定：五味酯甲：对照品斑点在紫外区（200～400nm）光谱扫描，最大吸收波长为224nm，最小吸收处在310nm，确定λ_S=224nm，λ_R=310nm，反射法锯齿扫描，S_X=3，最小面积1000。

丹参酮ⅡA：对照品斑点在可见区（400～800nm）光谱扫描，最大吸收峰是485nm，为测定波长。反射法线性扫描，狭缝6.4×10mm，S_X=3，扫描速度20mm/min，灵敏度×1，最小面积。

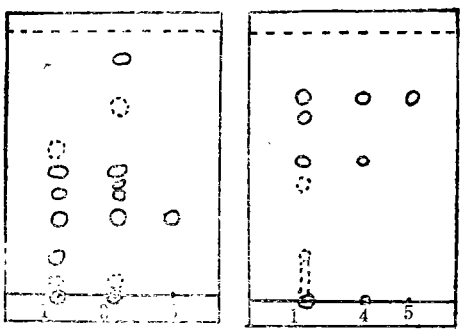

图 五灵丸层析图

1-五灵丸 2-五味子 3-五味酯甲 4-丹参 5-丹参酮ⅡA

1.3 线性范围试验：五味酯甲：准确吸取对照液2、4、6、8、10、12μl，点于同一硅胶GF_{254}板上，按上述层析条件展开，取出，凉干，在荧光灯下观察五味酯甲色点位置，上机扫

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描，以斑点的面积积分值对点样量(μg)作回归分析.得回归方程 Y = 12269.78X + 7815.44，r = 0.9986，线性范围1.36～6.80μg。

丹参酮ⅡA：准确吸取对照液2、4、6、8、10μl点于同一硅胶G薄层板上，按丹参酮ⅡA的层析条件展开，自然光下见丹参酮ⅡA斑点为红色，上机扫描，以斑点面积积分值对点样量（μg）的回归方程为：Y = 2056.0545X + 999.82，相关系数r = 0.9991。

1.4 稳定性试验：精密吸取五味酯甲对照液2μ，6μl，分点于薄层板上，展开后每隔一定时间扫描测定峰面积积分值，结果测定至24h依然稳定。精密吸取丹参酮ⅡA对照液4μl，8μ分点于薄层板上，展开后每隔一定时间扫描，4h内斑点面积积分值稳定。

1.5 回收率试验：采用加样回收试验。五味酯甲6次测定的平均回收率为98.3％，CV = 2.1％。丹参酮ⅡA6次测定的平均回收率为99.15％，CV = 1.13％。

1.6 精密度试验：同一薄板不同斑点扫描测定，峰面积积分值变异系数五味酯甲CV = 1.2％（n = 6），丹参酮ⅡACV = 2.2％（n = 6）。

1.7 样品测定

1.7.1 样品溶液的制备：五味子供试液：取五味子粉（过40目筛）置具塞试管中，加70％乙醇10.00ml，混匀超声20min，振摇，过夜，取上清液点样。

五灵丸供试液（a）：取2丸，剪碎，混均，称取2.00g，置具塞试管中，加3.00ml水泡20min，分散后加7.00ml无水乙醇超声20min，振摇放置过夜，取上清液点样。

丹参供试液：取丹参药材粉末（过40目筛）1.00g，加乙醇10.00ml，超声20min，振摇，放置过夜，取上清液点样。

五灵丸供试液（b）：取2丸剪碎混匀，称取5.00g，置具塞锥形瓶中，加1ml水浸泡10min使其分散，加15ml氯仿，摇匀，超声25min，过滤，药渣继续加12ml氯仿超声20min，过滤，用少量氯仿洗涤药渣、滤纸，合并滤液与洗涤液，挥去氯仿，残渣加少量氯仿转溶于1ml量瓶中定容，供点样用。

1.7.2 测定方法：五味酯甲：采用外标两点法。准确吸取五味子、五灵丸供试液（a）各10μl，点于硅胶GF_{254}板上，并随行点五味酯甲对照液2μl、6μl同标准曲线操作，五味酯甲含量见表。

表 原料及不同批号五灵丸中五味酯甲与丹参酮ⅡA的含量（n=3）

样品	五味酯甲(mg/g)	丹参酮ⅡA(mg/g)
五味子	2.863	
丹参		3.141
五灵丸(4批)	1.03 0.998	0.191 0.217
	0.678 0.861	0.245 0.228

丹参酮ⅡA：采用外标两点法。准确吸取丹参供试液10μl，五灵丸供试液（b）6μl，分点样于硅胶G薄板上，随行点丹参酮ⅡA对照液2、4μl，同标准曲线操作，结果见表。

2 讨论

五味子供试液制备，我们用了60％，70％及95％乙醇、甲醇、氯仿提取，扫描结果无显著差异。五灵丸为大蜜丸，需加少量水使其分散，故选用70％乙醇提取。

对丹参药材和五灵丸用氯仿、乙醇、70％乙醇、甲醇4种溶剂进行了提取率的比较，除70％乙醇提取率偏低外，其余结果无显著差异。因氯仿提取丸药可排除蜜的干扰，浓缩液的提取浓度小，易于点样，所以五灵丸用少量水分散后氯仿提取。

（1993-12-01收稿）

Qian hun (*P.harry-smithii* var.*subglabrum*) and Baihua Qianhu (*P.praeruptorum*) was carried out. Results revealed that the former two Qianhu produced in Gansu are similar to Baihua Qian hu in their main ingredients.Thus the two Qianhu are worthy for further research and develo pm ent.At the same time, it was observed that Baihua Qianhu Produced in Gansu is of inferior quality and the content of EtOH extract of its root is slightly lower than that from elsewhere in China.

(Original article on page 129)

Determination of Schizandrin A and Tanshinone ⅡA in Wulingwan with TLC-Scanner Method

Wang Xiaojuan, Guo Huifang, Wang Jianpo, et al

TLC-scanner method was used to determine the content of schizandrin A and tanshinone ⅡA in Wulingwan. The average recovery of both schizandrin A and tanshinone ⅡA are 98.34% (CV=2.1%) and 99.15% (CV=1.1%) respectively.This method is simple and rapid.Its reproducibility is satisfactory.

(Original aiticle on page 131)

Effect of Extract Zhonghuabie (*Amyda sinensis*) on Syntheses of DNA and Protein in Mice

Huang Tiangui, Tao Zhuliang et al

Extract *Amyda sinensis* raised the levels of Plasma proteins. Plasma albumin was raised from 2.67±0.44 to 3.25±0.34g/dl and the total plasma protein from 5.34±0.88 to 6.74±1.38 /dl. 3H-TdR and.3H-Leucine incorporation techniques were used to measure the syntheses rate of DAN and protein.The rates was accelerated.The specific activities of DNA and protein of liver got up to 3.90±1.41 from 2.42±0.71 dpm/μg, and 21.69±4.84 from 12.81 ±5.83 dpm/μg, respectively.Those of spleen got up to 41.88±18.47 from 19.04±10.54 dpm/μg and 23.12±4.38 from 16.34±7.01 dpm/μg, respectively. Extract *Amyda sinensis* had no effect on DNA synthesis of bone marrow cells and did not raise the hemoglobin level in mice. The results suggest that Extract *Amyda sinensis* has bioactive substance that accelerate synthcses of DNA and protein.

(Origindl article on page 138)

Effects of Sini Decoction on Ischemic (Anoxic) Electrocardiogram

Wu Weikang Jin Wentao, Luo Canhua, et al

Effects of Sini decoction (SD) on ischemic (anoxic) electrocardiogram (ECG) and possible action mechanism of SD were studied.

Results indicate that SD significantly improves the pituitrin induced ischemic ECG of rabbits, significantly prevents S-T segment from desconding and suppresses the elevation of T wave; SD can also lengthen significantly cardioe lectric activity time of anoxic mice. The protective effects of SD on ischemic (anoxic) myocardium may be related to the significant increase of myocardial nutritional blood flow induced by administrating SD.

(Original article on page 141)

Studies on the Pharmacology of Cajanin Preparation

Sun Shaomei, Song Yumei, Liu Jian, et al

Cajanin preparation could significantly reduce the mouse pinna inflammation induced