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Chinese Journal of Natural Medicines 2010, **8**(6): 0449–0455
doi: 10.3724/SP.J.1009.2010.00449
Chinese Journal of Natural Medicines

Simultaneous Determination of Six Components in the 'Jiang-Zhi' Granule by UPLC-MS Analysis

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Available online 20 Nov. 2010

[ABSTRACT] AIM: A novel and rapid method using an ultra-performance liquid chromatography coupled with mass spectrometry was developed and validated for the simultaneous determination of six constituents in 'Jiang-Zhi' granule. **METHODS:** An acquity UPLC BEH C_{18} column (2.1 mm × 50 mm, 1.7 μm) was utilized. Methanol and 0.1% formic acid were adopted in the elution gradient. The selective ion monitor (SIM) mode was used to detect the target compounds. **RESULTS:** The established method showed a good linearity (R^2 > 0.999 2) over the investigated concentration ranges, good inter-day and intra-day precisions (less than 3%) and good recoveries (from 97.58% to 103.12%) for all six target compounds. **CONCLUSION:** The contents of six main components in 'Jiang-Zhi' granule could be determined using the established method.

[KEY WORDS] Ultra performance liquid chromatography–mass spectrometry; Traditional Chinese medicine; 'Jiang-Zhi' granule

[CLC Number] R917 **[Document code]** A **[Article ID]** 1672-3651(2010)06-0449-07

1 Introduction

Nonalcoholic fatty liver disease (NAFLD) is a fatty liver disease occurring in patients without alcohol consumption [1]. NAFLD has a spectrum ranging from fatty liver alone to steatohepatitis, steatonecrosis, and nonalcoholic steatohepatitis [2-5], and is considered as the most common chronic liver condition in the Western world [6-7]. In recent years, this kind of disease has also received more and more attention in China [8].

'Jiang-Zhi' granule, as a compound prescription of traditional Chinese medicine (TCM), consists of *Radix et Rhizoma Salviae Miltiorrhizae* (Danshen in China), *Folium Nelumbinis* (Heye in China), *Rhizoma Polygoni Cuspidati* (Huzhang in China) and *Herba Artemisiae Scopariae* (Yinchen in China). This prescription has demonstrated significant effects of hypolipidemic and hepatoprotective activity with few side-effects, and it has been used to treat NAFLD in clinical practice in China. Although the main active constituents of the herbal materials of 'Jiang-Zhi' granule were revealed, a quantification method for these active constituents is still deficient and the quality control of this prescription cannot be ensured.

As reported previously, the major components from Danshen are the hydrophilic depside derivatives and the lipophilic diterpenoids [9-12]; pharmacological properties of Heye are mainly attributed to alkaloids [13-15]; Huzhang contains anthraquinones and anthraquinone derivatives [16-19]; and the major constituents in Yinchen are phenolic acids, coumarins, flavonoids and 4-hydroxyacetophenone [20-23].

Based on the reports, six components, including tanshinone II_A, danshensu, salvianolic acid B, nuciferine, emodin and chlorogenic acid, are derived from herb materials of 'Jiang-Zhi' granule and have been considered as the main bioactive components (structures shown in Fig. 1). Both hydrophilic components such as salvianolic acid B and lipophilic components such as tanshinone IIA are included in these compounds. That means a long time may be needed for their

[Received on] 13-Jul.-2010

[Research Funding] This project was supported by the National Nature Science Foundation of China (No. 30873260), the Talents Scheme from the Science and Technology Commission of Shanghai Municipality (No. 09XD1403800), the China Postdoctoral Science Foundation (No. 20090460645), the Supporting Project for Elitists in the New Century from The Ministry of Education (No. NCET-07563) and the Shanghai Leading Academic Discipline Project (Nos. J50305 and E3008).

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These authors have no any conflict of interest to declare.

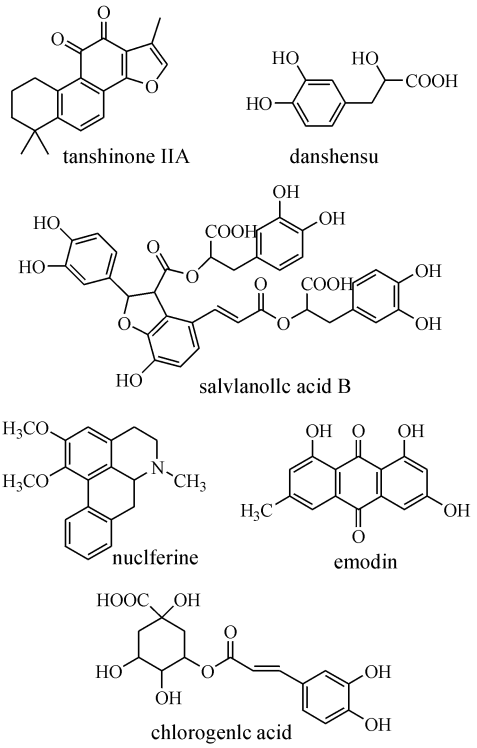


Fig. 1 Chemical structures of the six investigated compounds in 'Jiang-Zhi' granule

separation in high-performance liquid chromatography (HPLC) system. To shorten the analysis time, an ultra-performance liquid chromatography (UPLC) system, a new technology with high column effect and peak capacity [24-25], was adopted in the present study.

The aim of this study was to establish a convenient and effective UPLC-MS method for the quality evaluation of 'Jiang-Zhi' granule through the quantification of six major components. For this purpose, parameters of both UPLC separation and mass spectrometry were optimized in the present study. Results showed that six components were successfully identified and determined. And the analysis time was shortened, which significantly improved the efficiency. And the established method was applied in the commercially available samples.

2 Experimental

2.1 *Chemicals and reagents*

Reference compounds including tanshinone II_A, danshensu, salvianolic acid B, nuciferine, emodin, chlorogenic acid, carbamazepine were purchased from Shanghai Winherb Medical Sci & Tech Development Co. Ltd (Shanghai, China). Methanol and formic acid were of HPLC grade, which were purchased from Merck (Darmstadt, Germany). Water used in the experiment was generated by a Milli-Q water purification system (Millipore, MA, USA). Other reagents were of HPLC grade or the highest grade commercially available. The commercial products of 'Jiang-Zhi' granule were purchased from the market.

2.2 *Preparation of reference compounds solutions*

A stock reference solution containing tanshinone II_A (2.75 μg·mL^{-1}), danshensu (23.25 μg·mL^{-1}), salvianolic acid B (19.63 μg·mL^{-1}), nuciferine (14.50 μg·mL^{-1}), emodin (19.38 μg·mL^{-1}), chlorogenic acid (14.13 μg·mL^{-1}) were prepared in water/methanol (5∶95, *V*/*V*). Subsequently, the stock solutions were diluted to serial work solutions. Carbamazepine (6.78 μg·mL^{-1}) was used as internal standard (I.S.). All solutions were stored at 4 °C away from light until analysis.

2.3 *Preparation of sample solutions*

'Jiang-Zhi' granule was triturated and blended to powder. 20 mg of the powder was extracted with 25 mL water/methanol (5∶95, *V*/*V*) promoted by an ultrasonic bath under the frequency of 42 kHz for 30 min. The lost weight of the extracted solution was compensated prior to filtration. The supernatant was filtered through a membrane filter (0.45 μm) and the filtrate was introduced to UPLC-MS for analysis.

2.4 *Instrumentation and UPLC-MS parameters*

The experiments were performed on a Waters Acquity UPLC system, equipped with a quaternary pump system (Milford, MA, USA). Separations were performed on a 2.1 mm × 50 mm column packed with 1.7 μm particles (Acquity UPLC BEH C_{18} column, Waters) designed to withstand 15 000 psi. Optimum separation was achieved with a binary mobile phase at a flow rate of 0.3 mL·min^{-1}. The column temperature was held at 45 °C. The mobile phase consisted of 0.1% formic acid water (A) and methanol (B). The gradient program was as follows: 0-2 min 10%-40% B; 2-3 min 40%-80% B; 3-3.5 min 80%-90% B; 3.5-5 min 90% B. The sample injection volume was 2 μL.

The detector was a single quadrupole mass spectrometer (Micromass, Waters, MA, USA) with electrospray ionization (ESI) source. The positive ion mode and the selected ion monitoring (SIM) mode were used for quantification with target ions at *m/z* 317.4 for $[M + Na]^+$ ion of tanshinone II_A, *m/z* 221.4 for $[M + H]^+$ ion of danshensu, *m/z* 741.6 for $[M + Na]^+$ ion of salvianolic acid B, *m/z* 296.5 for $[M + H]^+$ ion of nuciferine, *m/z* 271.4 for $[M + H]^+$ ion of emodin, *m/z* 377.4 for $[M + Na]^+$ ion of chlorogenic acid and *m/z* 237.2 for $[M + H]^+$ ion of carbamazepine (I.S.), respectively. The optimal MS parameters obtained were as follows: capillary 4.0 kV, cone voltages 40 V, source temperature 120 °C and desolvation temperature 300 °C. Nitrogen was used as the desolvation gas and cone gas with a flow rate of 550 L·h^{-1} and 50 L·h^{-1}. All data were processed using MassLynx V 4.1 software with a QuanLynx program (Waters).

3 Results and Discussion

3.1 *Optimization of the extraction method*

Various extraction methods and solvents have been

tested to obtain a suitable process for sample preparation. Extraction conditions, such as extraction methods (ultrasonic and refluxing), extraction solvents (65%, 80%, 95% methanol), volume of extraction solvent (20, 25 and 30 mL) and extraction time (15, 30 and 45 min) were investigated one by one to choose the appropriate method. The extraction efficiency was evaluated. Due to its convenience and simple operation, ultrasonic extraction was finally selected. It was found that the target constituents could be efficiently extracted with 95% methanol under ultrasonication and refluxing for 30 min.

3.2 *Optimization of UPLC-MS parameters*

Methanol and formic acid were adopted in the mobile phase to obtain high signal-to-noise ratio, shorten peak tails and to improve the sensitivity of the analytes in the positive ion mode of UPLC-MS.

MS examination of the six standard solutions in positive and negative ionization modes by direct full scan method revealed that the signals obtained from the electrospray ionization source in the positive mode had a good resolution and high intensity to permit quantitative measurement. In order to optimize the MS parameters, different conditions were tested. MS parameters, such as capillary (2, 2.5, 3, 3.5, 4.0 kV), cone voltages (10, 20, 30, 40 V), flow rate of desolvation gas (450, 500, 550 and 600 $L \cdot h^{-1}$), and flow rate of cone gas (30, 40 and 50 $L \cdot h^{-1}$) were investigated one by one for optimization. The result of detection was evaluated. It was found that the optimal MS parameters were as follows: capillary 4.0 kV, cone voltages 40 V, the desolvation gas and cone gas with a flow rate of 550 $L \cdot h^{-1}$ and 50 $L \cdot h^{-1}$. The electrospray ionization of danshensu, nuciferine, emodin, and carbamazepine produced abundant protonated molecular ions ($[M + H]^+$) at *m/z* 221.4, 296.5, 271.4 and 237.2 respectively. In addition, the predominant sodium adduct ions $[M + Na]^+$ at *m/z* 317.4, 741.6 and 377.4 were selected for analyzing tanshinone II_A, salvianolic acid B, and chlorogenic acid respectively. Each mass spectrum of the detective ions is shown in Fig. 2. In this study, cross-talk effect produced using different channels in mass system was not observed.

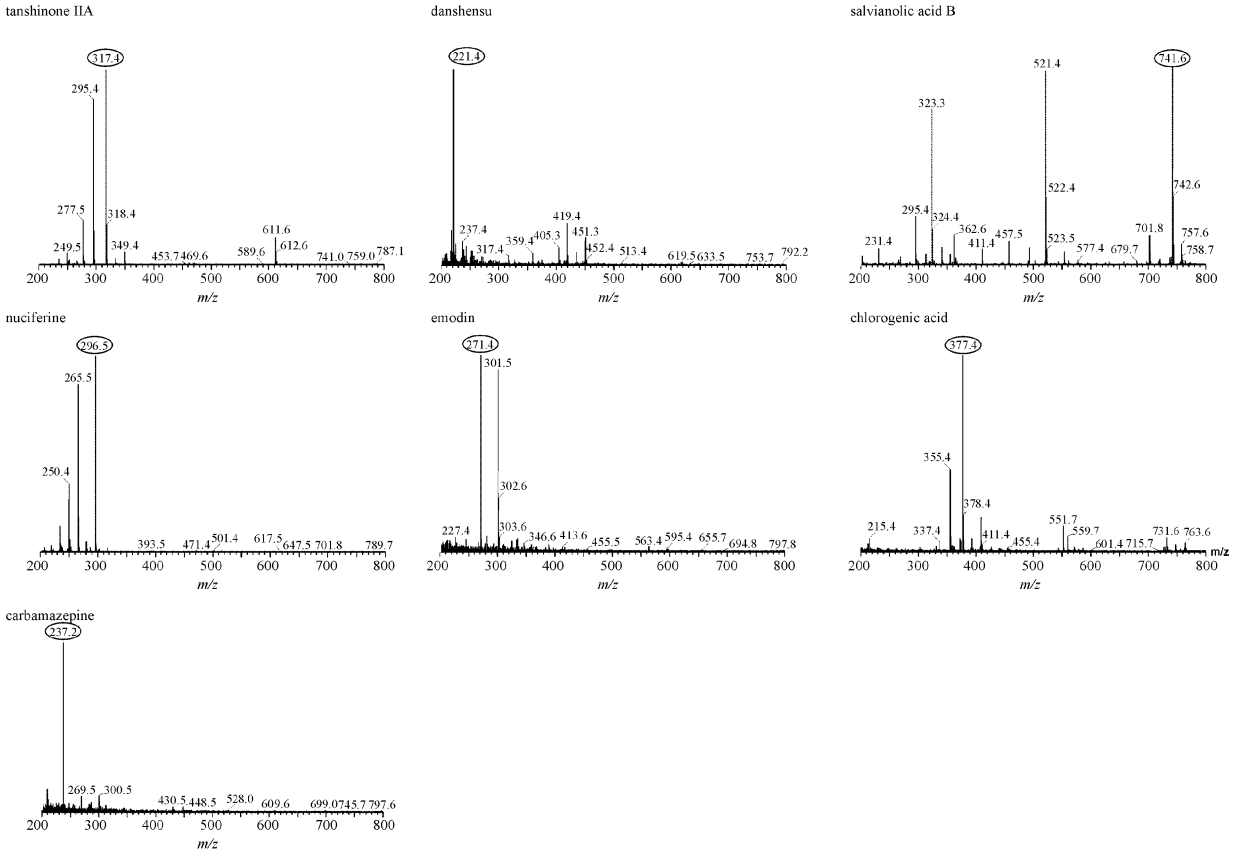


Fig. 2 Ion mass spectra of tanshinone II_A, danshensu, salvianolic acid B, nuciferine, emodin, chlorogenic acid, and carbamazepine (I.S.)

3.3 *Method validation*

The analytical methods had been validated for parameters such as linearity, precision, accuracy, repeatability, and stability. The relative standard deviation (*RSD*, %) was taken as a measure of precision, repeatability and stability.

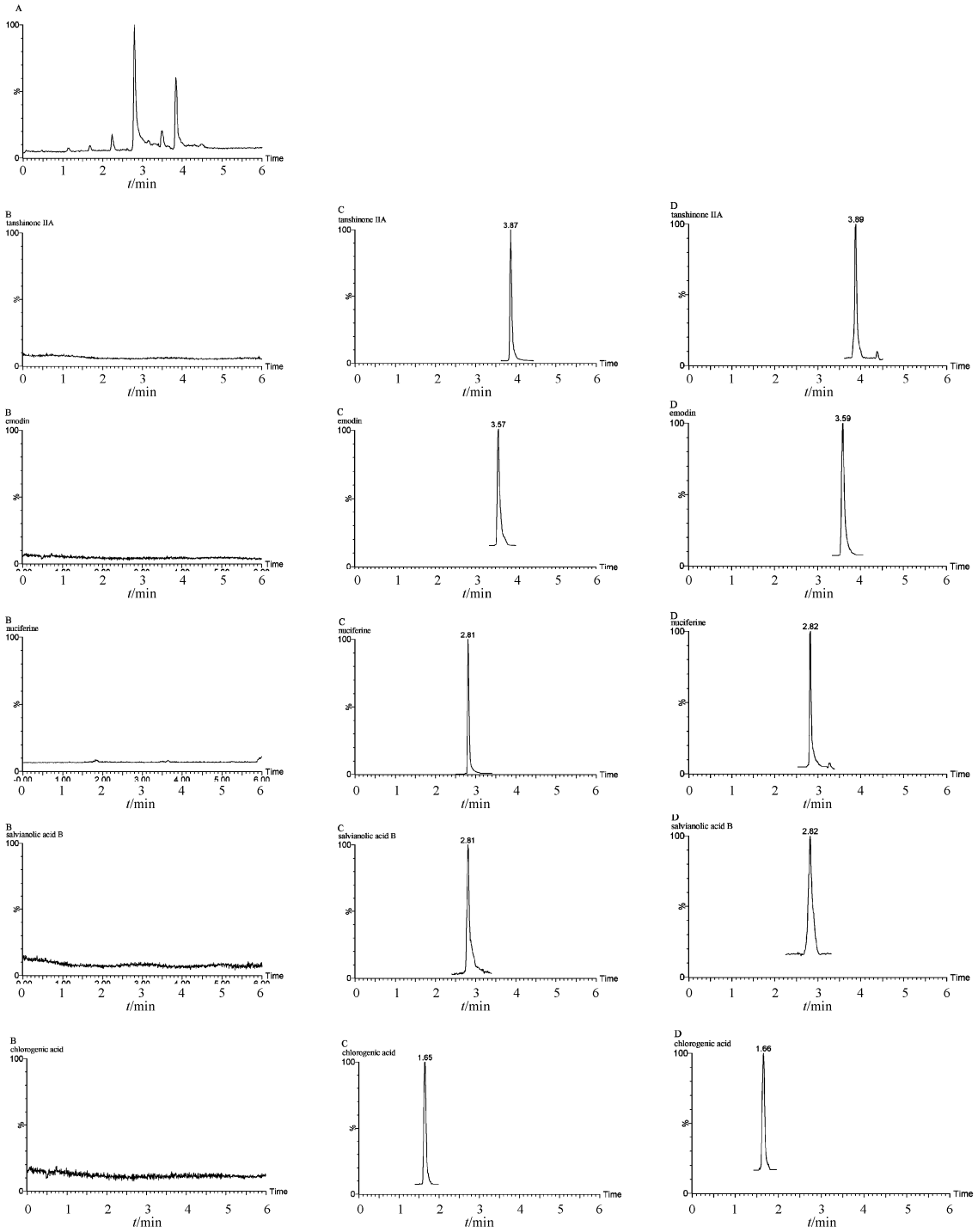
3.4 *Specificity*

The specificity of the method was tested through the analysis of the negative control samples. Based on the prescription of 'Jiang-Zhi' granule, negative control samples were prepared without Danshen, Heye, Huzhang, or Yinchen.

Negative control sample solutions were made according to the preparation of sample solutions. There was no significant chromatographic interference around the retention times of the analytes and I.S. in negative control samples (Fig. 3). The retention times of tanshinone II_A, danshensu, salvianolic acid B, nuciferine, emodin, chlorogenic acid, and carbamazepine were 3.9, 1.1, 2.8, 2.8, 3.6, 1.6 and 2.3 min, respectively.

3.5 *Linearity, range and limits of detection*

The calibration curves were constructed by analyzing at least five different concentrations of standard solutions. As a result, good linearity (R^2 > 0.999 2) of the investigated concentration ranges was observed. The injection concentration, which could be detected at the signal-to-noise ratio of 3, was considered to be the limit of detection (LOD). Limit of quantification (LOQ) was the injection concentration corresponding to the peak heights with a signal-to-noise of 10. All the detailed information of the calibration curves is listed in Table 1. Their regression equations were calculated in the form of $Y = bX + a$, where Y and X were the peak area rate and sample concentration, respectively. The results in Table 1



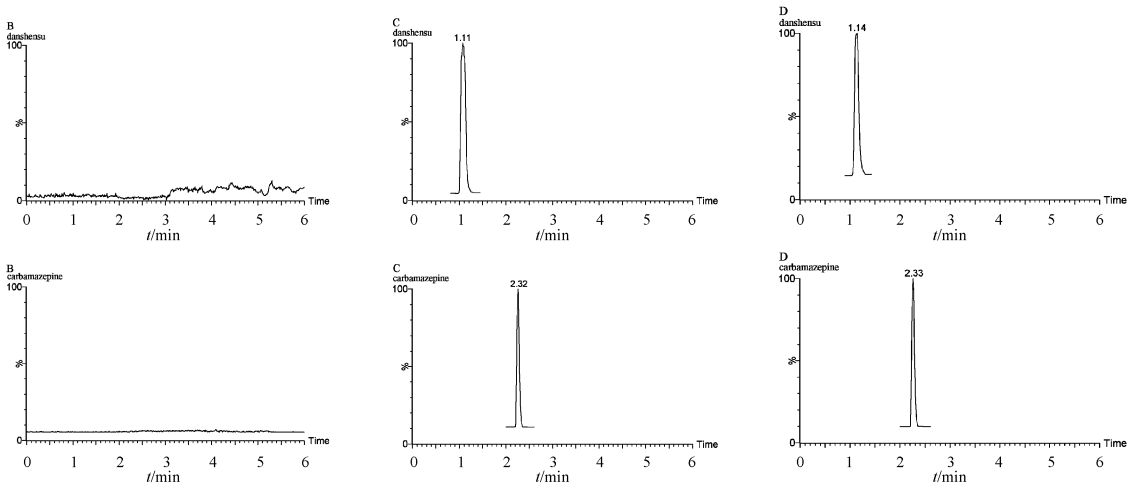


Fig. 3 A Typical chromatograms of TIC, B negative control, C standard, and D sample solutions

Table 1 Regression equation, linear range, detection limits of the developed method, and data of precision, repeatability and stability

Constituents	Regression equation	Correlation coefficient/ R^2	Linearity range/ (μg·mL^{-1})	LOD/ (μg·mL^{-1})	LOQ/ (μg·mL^{-1})	Precision (*RSD*/%)		Repeatability (*RSD*/%)	Stability (*RSD*/%)
						Intra-day	Inter-day		
tanshinone II_A	$Y = 24.75X - 10.03$	0.999 2	0.044-2.75	0.015	0.044	1.46	2.47	2.96	2.16
danshensu	$Y = 0.37X + 0.26$	0.999 5	0.37-23.25	0.12	0.37	0.76	1.11	2.18	1.94
salvianolic acid B	$Y = 0.13X + 0.57$	0.999 7	0.31-19.63	0.11	0.31	1.21	1.06	3.19	2.21
nuciferine	$Y = 70.51X - 31.55$	0.999 5	0.23-14.50	0.077	0.23	1.95	2.23	1.33	1.76
emodin	$Y = 0.55X + 0.83$	0.999 7	0.31-19.38	0.10	0.31	1.37	1.72	2.76	2.97
chlorogenic acid	$Y = 0.55X + 0.16$	0.999 6	0.23-14.13	0.075	0.23	0.98	1.34	1.15	2.53

showed good linear behavior. The investigated concentration ranges were observed with values of R^2 higher than 0.999 2 for all the analytes. The linearity ranges were adequate for the determinations of eight constituents in the samples.

3.6 *Precision tests*

Intra-day precision was examined with the mixture standard solutions during a single day. The inter-day precision was determined twice per day over three consecutive days. The relative standard deviation (*RSD*) values varied from 0.76% to 2.47% for intra- and inter-day assays for all the analytes (results shown in Table 1).

3.7 *Repeatability and stability*

Injection repeatability was examined through the injection of six samples prepared with the same sample preparation procedure. Stability of the sample solution during 24 h at room temperature was tested at the interval of 4 h. For the repeatability and stability test, the *RSD* values varied from 1.15% to 3.19% and 1.76% to 2.97% (results shown in Table 1). *RSD* for the stability of the sample solution indicated that the investigated chemical system was stable enough for the routine analysis within a day at room temperature.

3.8 *Recovery test*

Recovery of the standard from samples is generally utilized to evaluate the accuracy of the newly developed analytical method. In the recovery test, the proposed method was applied to the samples blended with the mixed standard solution at high (initial amount-added amount 1∶1.5), middle (initial amount-added amount 1∶1.0), and low (initial amount-added amount 1:0.5) concentration levels. Each level was performed three times. The mixture was processed by the same extraction procedure as that used in the sample preparation, and analyzed using the same method. The recoveries were between 97.58% and 103.12%. The RSD values of each concentration level were < 3.32%.

3.9 *Sample analysis*

The analytical method was utilized for the determination of six active components in 'Jiang-Zhi' granule, and the results are shown in Table 3. In this article, the commercial samples were obtained from five manufacturers (1-5). The amounts of the six compounds varied substantially among the samples. Variation of the amounts in these samples may arise because of different manufacturing processes, in addition to

Table 2 Recoveries of the major components in 'Jiang-Zhi' granule

Analyte	Initial amount/μg	Added amount/μg	Detected amount/μg	Recovery*/%	*RSD*/%
		4.30	12.86	102.83	1.38
tanshinone IIA	8.44	8.61	17.21	101.81	1.41
		12.91	21.35	99.97	1.36
		85.94	256.54	100.49	1.76
danshensu	170.18	171.88	347.35	103.08	1.65
		257.83	421.85	97.61	1.19
		59.62	175.24	101.77	2.17
salvianolic acid B	114.56	119.23	231.79	98.32	2.67
		178.85	297.60	102.34	2.75
		0.85	2.55	103.12	3.32
nuciferine	1.67	1.70	3.36	99.53	2.71
		2.56	4.17	97.65	2.23
		77.56	232.36	99.59	1.54
emodin	155.12	155.12	314.49	102.74	1.91
		232.68	389.92	100.91	1.70
		57.45	167.69	97.58	2.75
chlorogenic acid	111.63	114.90	229.08	102.22	2.25
		172.35	288.91	102.86	1.89

* Recovery (%) = 100 × (detected amount – initial amount) / added amount; the data presented as average of three determinations

Table 3 Result of the quantification of the major components in 'Jiang-Zhi' granule

Content of analytes ($\bar{x} \pm s$, mg·g^{-1}, n = 5)	Sample No.				
	1	2	3	4	5
tanshinone IIA	0.84 ± 0.011	0.61 ± 0.013	0.73 ± 0.009 6	0.83 ± 0.012	0.44 ± 0.011
danshensu	17.02 ± 0.078	19.76 ± 0.22	17.55 ± 0.13	22.82 ± 0.21	15.16 ± 0.14
salvianolic acid B	11.46 ± 0.18	15.07 ± 0.17	14.10 ± 0.12	16.92 ± 0.19	8.69 ± 0.094
nuciferine	0.17 ± 0.001 6	0.58 ± 0.009 7	0.22 ± 0.002 8	0.35 ± 0.001 8	0.65 ± 0.012
emodin	15.51 ± 0.11	10.89 ± 0.072	12.25 ± 0.11	19.6 ± 0.31	14.42 ± 0.20
chlorogenic acid	11.16 ± 0.18	9.06 ± 0.024	8.67 ± 0.056	8.50 ± 0.055	14.12 ± 0.15

different origins of the raw material. It was believed that the UPLC method would be helpful to improve the quality control of 'Jiang-Zhi' granule.

4 Conclusions

In this study, a UPLC-MS method was established for the determination of major active components in 'Jiang-Zhi' granule. The high resolution obtained within extremely short analysis time makes UPLC a very attractive tool for pharmaceutical analysis. The newly developed method was carefully validated and successfully applied in the quantification of six major components in the commercial products of 'Jiang-Zhi' granule from different manufacturers.

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超高效液相-质谱法测定降脂颗粒中 6 种活性成分的含量

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【摘　要】 **目的:** 利用超高效液相-质谱法建立同时测定降脂颗粒中 6 种活性成分含量的方法。**方法:** 色谱柱为 Acquity UPLC BEH C_{18} 柱(2.1 mm × 50 mm, 1.7 μm), 流动相为甲醇-0.1%甲酸水梯度洗脱, 选择离子监测。**结果:** 在线性范围内相关系数良好, 均大于 0.999 2, 日内、日间精密度良好, *RSD* 值均小于 3%, 加样回收率在 97.58%-103.12%, 符合要求。**结论:** 本方法快速灵敏, 测定结果准确可靠, 可用于降脂颗粒的质量控制。

【关键词】 超高效液相-质谱法; 中药; 降脂颗粒

【基金项目】 国家自然科学基金(No. 30873260), 上海市优秀学科带头人计划(No. 09XD1403800), 中国博士后科学基金(No. 20090460645), 教育部新世纪优秀人才支持计划基金资助项目(No. NCET-07563), 上海市重点学科建设项目(Nos. J50305、E3008)资助