

论文

反相高效液相色谱-蒸发光散射检测法同时测定人工牛黄中多组分含量

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摘要:

目的:建立人工牛黄中多组分色谱分离条件,并对除无机盐外各成分同时测定进行方法学考察。方法:用反相高效液相色谱-蒸发光散射检测法,蒸发光散射检测器参数:漂移管温度 105℃,雾化气体(N<sub>2</sub>)流速:205SLPM。

结果:在选定色谱条件下,胆固醇、各种胆汁酸及无机盐在室温下可达很好分离,胆酸与猪去氧胆酸的非衍生分离尚属首次。除无机盐外,各物质色谱峰面积与浓度呈良好线性关系( $r > 0.998$ )。3个浓度水平的回收率测定值为98.3%~102.4%。进样20μL组分最低检出量为0.05~0.106μg。结论:蒸发光散射检测器与高效液相色谱法联用,可使不含生色团物质的分离、分析更为准确、灵敏。可为人工牛黄的质量控制提供更为科学的依据

关键词: 蒸发光散射检测器 高效液相色谱法 人工牛黄

SIMULTANEOUS DETERMINATION OF COMPONENTS IN ARTIFICIAL BEZOAR BY HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY WITH EVAPORATIVE LIGHT-SCATTERING DETECTOR

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Abstract:

AIM: To establish a simple reverse phase high-performance liquid chromatographic method for the simultaneous determination of several components in artificial ox gallstone. METHODS: Chromatography was performed on a 5 μm Nova-Park C<sub>18</sub> steel column (250 mm×4.6 mm ID, Waters) at ambient temperature. The mobile phase composition was methanol — water — glacial acetic acid (80:20:0.01).

The flow rate was 1.0 mL.min<sup>-1</sup>. An evaporative light-scattering detector (ELSD) Model 500 (Alltech, USA) was used as detector, its parameters were set as follows: nitrogen carrier gas flow 2.05 SLPM; drift tube temperature 105℃. RESULTS: Complete resolution of cholesterol, free bile acids [especially cholic acid (CA) and hyodeoxycholic acid (HDCA)] and inorganic salts in artificial ox gallstone was obtained for the first time. The method fulfills all the standard requirements of precision, accuracy and linearity.

The recoveries of components at three concentration levels were: cholic acid (CA) 98.88%~101.2%, hyodeoxycholic acid (HDCA) 99.02%~102.2%, chenodeoxycholic acid (CDCA) 98.56%~102.4%, deoxycholic acid (DCA) 98.32%~101.8%, cholesterol 98.41%~99.18%. The detection limits for 20 μL injected were CA and DCA 0.10 μg, CDCA 0.106 μg, HDCA 0.09 μg, cholesterol 0.054 μg, respectively. The detectivity of this method for bile acids is higher than that reported. CONCLUSION: HPLC, combined with ELSD, can give more accurate and sensitive results for the analyses of those compounds without chromophore. The new method established is more suitable for analyses of artificial ox gallstone.

Keywords: high-performance liquid chromatography artificial ox gallstone evaporative light-scattering detector

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