

论文

LC-MS分析乙酰吉他霉素组分及水解产物

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摘要:

目的应用LC-MS鉴定乙酰吉他霉素中组分及其水解产物。方法以0.1 mol·L⁻¹ 乙酸铵溶液-乙腈(35:65)为流动相, 采用Diamonsil C₁₈柱, 单四极杆质谱检测器, 电喷雾正离子扫描, 调节锥孔电压, 对色谱峰进行质谱分析。结果乙酰吉他霉素各组分可基本分离; 根据所得到的有关相对分子质量以及碎片峰的信息, 对乙酰吉他霉素各组分及其水解产物的结构进行了鉴别。发现国产乙酰吉他霉素的主要组分为乙酰吉他霉素A₄, A₅; A₁, A₃和A₆, A₇, 与日本药局方中报道的主要组分相同, 但国产乙酰吉他霉素还含有较多的乙酰吉他霉素A₁₃。乙酰吉他霉素效价测定中的水解产物并非吉他霉素。结论LC-MS方法快速、灵敏, 专属性强, 适合用于多组分抗生素的组分分析。

关键词: 液相色谱-质谱法 乙酰吉他霉素 组分鉴定 水解产物

Identification of the components and products of hydrolysis in acetylleucomycin by LC-MS

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Abstract:

Aim To identify the components of acetylleucomycin and its hydrolytic products by LC-MS. Methods acetylleucomycin was separated on a Diamonsil C₁₈ column with 0.1 mol·L⁻¹ ammonium acetate-acetonitrile (35:65) as mobile phase. The LC-MS was equipped with an electrospray ion source (ESI), which was set at the positive ion mode, and the mass spectra of each component in chromatogram were obtained with difference cone voltage. Results The components of acetylleucomycin and its hydrolytic products can be separated by HPLC. The components were identified according to the molecular weight and its major mass fragment ions. The major components identified in domestic acetylleucomycin were acetylleucomycin A₄, A₅; acetylleucomycin A₁, A₃; acetylleucomycin A₆, A₇, and acetylleucomycin A₁₃. The hydrolytic products of acetylleucomycin were not kitasamycin, but some non-complete hydrolytic product. Conclusion The method is rapid, sensitive and specific. It's suitable to application in the fields of multi-components antibiotics analysis.

Keywords: acetylleucomycin component identification product of hydrolysis LC-MS

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