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GC测定奈拉滨中有机溶剂残留量

Determination of Residual Organic Solvents in Nelarabine by GC

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中文摘要:

目的 建立奈拉滨中有机溶剂残留量的气相色谱分析方法。**方法** 色谱柱为SE-54石英毛细管柱(30 m×0.53 mm, 1.0 μm), 氢火焰离子化检测器(FID), 载气为氮气, 流速40 mL·min⁻¹。进样口温度: 200 °C, 检测器温度: 250 °C, 分流比为50:1, 柱温为程序升温: 初始温度40 °C保持5 min, 以50 °C·min⁻¹升至200 °C保持5 min。外标法进行定量, 并对分离条件进行了研究。**结果** 甲醇、乙腈、二氯甲烷、乙酸乙酯的线性范围分别为14.93~746.4 ng($r=0.999\ 4$), 1.950~97.45 ng($r=0.999\ 8$), 2.980~149.0 ng($r=0.999\ 6$), 25.12~1 256 ng($r=0.999\ 8$); 平均回收率($n=9$)分别为100.6%(RSD=1.87%), 101.0%(RSD=2.27%), 99.7%(RSD=3.41%), 100.4%(RSD=1.52%)。**结论** 该方法快速、准确、灵敏度高、重复性好, 可作为奈拉滨中有机溶剂残留量的测定方法。

英文摘要:

OBJECTIVE To establish a method for the determination of residual organic solvents in nelarabine by gas chromatography. **METHODS** Chromatographic column was capillary column SE-54(30 m×0.53 mm, 1.0 μm), with FID detector and nitrogen as carrier gas, the flow rate was 40 mL·min⁻¹. Injector temperature was 200 °C and the detector temperature was 250 °C, the split ratio was 50:1, column temperature was programmed and initial temperature was 40 °C, maintained for 5 min, raised to 200 °C with a rate of 50 °C·min⁻¹, maintained for 5 min. The external standard method was used for quantitative analysis. Analytical conditions were studied. **RESULTS** The standard curves were linear in the range of 14.93-746.4 ng($r=0.999\ 4$) for methanol, 1.950-97.45 ng($r=0.999\ 8$) for acetonitrile, 2.980-149.0 ng($r=0.999\ 6$) for methylene chloride and 25.12-1 256 ng($r=0.999\ 8$) for ethyl acetate. The average recoveries ($n=9$) were 100.6% (RSD=1.87%), 101.0% (RSD=2.27%), 99.7% (RSD=3.41%), 100.4% (RSD=1.52%). **CONCLUSION** The method is rapid, accurate, sensitive, reproducible and can be used for detecting residual organic solvents in nelarabine.

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