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GC测定氧氟沙星和诺氟沙星中N-甲基哌嗪和哌嗪的残留量

Determination of N-Methyl Piperazine and Piperazine in Ofloxacin and Norfloxacin by GC

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中文关键词: [气相色谱法](#) [N-甲基哌嗪](#) [哌嗪](#) [氧氟沙星](#) [诺氟沙星](#)

英文关键词: [GC](#) [N-methyl piperazine](#) [piperazine](#) [ofloxacin](#) [norfloxacin](#)

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中文摘要:

目的 建立气相色谱法测定氧氟沙星和诺氟沙星中N-甲基哌嗪和哌嗪的残留量的方法。方法 采用Agilent HP CAM 毛细管色谱(30.0 m×0.25 mm, 0.25 μm); 进样口温度为230 °C; FID检测器温度为280 °C; 柱温: 程序升温, 70 °C保持3 min, 然后以40 °C min⁻¹的速率升温至180 °C, 保持2 min; 以氮气为载气, 流速为2.0 mL·min⁻¹; 直接进样, 进样量为0.2 μL; 以水为溶解介质, 以吡啶为内标, 测定了氧氟沙星和诺氟沙星中N-甲基哌嗪和哌嗪的残留量。结果 N-甲基哌嗪、哌嗪与内标均能良好分离, 线性范围分别为0.09~201.88 μg·mL⁻¹(*r*=0.999 6), 10.08~201.60 μg·mL⁻¹(*r*=0.999 5)。结论 该方法重复性良好, 回收率符合规定, 适用于氧氟沙星和诺氟沙星中N-甲基哌嗪和哌嗪残留量的测定。

英文摘要:

OBJECTIVE To establish a method for determination of N-methyl piperazine and piperazine in ofloxacin and norfloxacin. METHODS The determination was carried out with Agilent HP CAM capillary column (30.0 m×0.25 mm, 0.25 μm) and FID detector. The inlet temperature was 230 °C and the detector temperature was 280 °C. The column temperature rose by program: the initial temperature was 70 °C, maintained for 3 min, raised to 180 °C with a rate of 40 °C·min⁻¹, maintained for 2 min, The carries gas was nitrogen and the flow rate of carries gas was 2.0 mL·min⁻¹, the direct injection was used and the injection volume was 0.2 μL. The dissolved medium used was water and the internal standard was pyridine. RESULTS N-methyl piperazine and piperazine could be separated from internal standard in chromatogram obtained from system suitability test. The calibration curves were linear in the range of 10.09-201.88 μg·mL⁻¹ (*r*=0.999 6) for N-methyl piperazine, 10.08-201.60 μg·mL⁻¹ (*r*=0.999 5) for piperazine. CONCLUSION The repeatability and recovery met requirements, it can be applied in determination of N-methyl piperazine and piperazine in ofloxacin and norfloxacin.

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