

研究快报

## 基于杯 [4] 芳烃探头的固相微萃取-毛细管电泳法测定尿样中的兴奋剂普萘洛尔异构体

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**摘要** 采用溶胶-凝胶法制备了一种双缩水甘油基杯 [4] 芳烃羟基硅油探头, 运用自行设计的反萃取装置实现了顶空固相微萃取与毛细管电泳的离线联用, 结合超声反萃取和场放大进样技术成功地测定了尿样中兴奋剂普萘洛尔异构体的含量。实验考察了固相微萃取条件和反萃取条件对测定结果的影响, 比较了杯 [4] 芳烃探头与商品化探头对于尿样中普萘洛尔异构体的萃取性能, 结果表明基于杯 [4] 芳烃探头的固相微萃取过程能够实现满意的净化效应与预富集效应。利用毛细管电泳-二极管阵列检测器对加标尿样中普萘洛尔异构体测定的线性范围为0.05~10 mg/L, 检测限为8~10  $\mu\text{g/L}$ , 相对标准偏差小于6.5% (n=6), 两种异构体的加标回收率为86%~107% (n=5)。该探头的可重复使用 (>150次) 性能良好。

**关键词** [杯\[4\]芳烃](#) [固相微萃取](#) [毛细管电泳](#) [兴奋剂](#) [普萘洛尔异构体](#)

分类号

## Solid-Phase Microextraction Coupled with Capillary Electrophoresis for Doping Analysis of Propranolol Enantiomers in Urine Using a Sol-Gel Derived Calix [4] arene Fiber

### Abstract

A new type fiber coated with diglycidyl ether calix [4] arene/hydroxy-terminated silicone oil (diglycidyl ether-C [4] arene/OH-TSO) made by sol-gel method was prepared for capillary electrophoresis (CE) sample pretreatment. By using headspace solid-phase microextraction (HS-SPME) combined with a novel back-extraction facility coupled off-line to capillary zone electrophoresis (CZE), the determination of propranolol enantiomers in urine was achieved with combination of ultrasonic back-extraction and field amplified sample injection (FASI) technologies. Extraction and back-extraction parameters were optimized. The clean-up effect and preconcentration effect were realized without derivatization during the SPME process in terms of this strongly polar and thermally stable compound. Preconcentration of the sample by calix [4] arene fiber increased the sensitivity, yielding a limit of detection (LOD) of 0.01 mg/L by CZE-diode array detection (DAD). Method repeatability (relative standard deviations (RSD) < 6.5%) and fiber reusability (> 150 extraction procedures) were observed over a wide linear range of propranolol (0.05-10 mg/L) in urine samples. Compared with commercial SPME stationary phases, the new coating showed higher extraction efficiency and this SPME-CZE-DAD procedures could meet the demand of minimum required performance limits (MRPL) set by the World Anti-Doping Agency (WADA) for the detection of propranolol in urine samples.

**Key words** [calix \[4\] arene](#) [solid-phase microextraction](#) [capillary electrophoresis](#) [doping analysis](#) [propranolol enantiomers](#)

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