

固相萃取-高效阴离子交换色谱-积分脉冲安培法检测人体尿液中的异黄蝶呤

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Determination of isoxanthopterin in human urine by solid phase performance anion-exchange chromatography coupled with integrated amperometric detection

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摘要 建立了固相萃取-高效阴离子交换色谱-积分脉冲安培法(SPE-HPAEC-IPAD)测定人体尿液中异黄蝶呤的分析方法。尿液经ENI型阳离子交换柱串联萃取后,除去了大量干扰物质。采用IonPac AS21分析柱(250 mm×2 mm),以0.025 mol/L NaOH溶液为淋洗液,流速0.40 mL/min,在优化的安培检测波形条件下,异黄蝶呤的质量浓度在0.005~0.200 mg/L范围内与峰面积呈良好的线性关系,相关系数 $r^2=0.9994$,检出限为0.003 mg/L。健康人及癌症病人尿液在2 mg/L和5 mg/L两个添加水平的平均回收率在95.4%~96.8%之间,相对标准偏差在5%以内。此方法环保、快速、准确,可用于健康人与癌症病人尿液中异黄蝶呤的测定。

关键词: 高效阴离子交换色谱 积分脉冲安培法 固相萃取 异黄蝶呤 人体尿液

Abstract: A sensitive, selective and environmental friendly method for the determination of isoxanthopterin in human urine by solid phase extraction (SPE)-high performance anion exchange chromatography (HPAEC) with integrated amperometric detector has been developed. The tandem solid phase extraction was employed to purify isoxanthopterin from human urine. The separation of isoxanthopterin was carried out on an IonPac AS21 anion-exchange column with an eluent of 0.025 mol/L NaOH at the flow rate of 0.40 mL/min. Under the optimized conditions, the detection limit of isoxanthopterin was 0.003 mg/L, and the linear range was 0.005~0.200 mg/L. The spiked recoveries ranging from 95.4% to 96.8% were obtained in the urine samples from healthy persons and cancer patients, and the relative standard deviation (RSD) was less than 5%. The present method was successfully applied to the determination of isoxanthopterin in urine from healthy individuals and cancer patients.

Keywords: high performance anion exchange chromatography (HPAEC) integrated pulsed amperometric detection (IPAD) solid phase extraction (SPE) isoxanthopterin human urine

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