

研究论文

液相色谱-双通道原子荧光检测联用法同时测定砷和硒的形态

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摘要 建立了一种利用高效液相色谱-双通道原子荧光检测联用同时进行砷和硒形态分析的方法。以10 mmol/L NH₄H₂PO₄溶液(pH 5.6)(添加2.5%(体积分数)的甲醇)为流动相,在12 min内同时分离了三价砷(As(III))、一甲基砷(MMA)、二甲基砷(DMA)、五价砷(As(V))、硒代胱氨酸(SeCys)、硒代蛋氨酸(SeMet)和四价硒[Se(IV)]等化合物。As(III)、DMA、MMA、As(V)、SeCys、SeMet和Se(IV)的检出限分别为1, 3, 2, 3, 4, 18和3 μg/L(进样量为200 μL),5次测定的相对标准偏差为1.9%~6.1%(As 100 μg/L, Se 300 μg/L)。应用该方法对人体尿样及硒酵母片中砷和硒的形态进行了分析,目标物在尿样中的加标回收率为83%~108%,在硒酵母片中的加标回收率为88%~105%。实验结果表明,该方法可用于尿样及药品中砷和硒形态的日常分析。该方法减少了样品的分析时间和试剂用量,降低了工作强度,提高了工作效率。

关键词 [高效液相色谱](#) [双通道原子荧光检测](#) [形态分析](#) [砷](#) [硒](#)

Simultaneous speciation of arsenic and selenium by high performance liquid chromatography-double channel atomic fluorescence spectrometry

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Abstract
A comprehensive method for simultaneously detecting species of arsenic and selenium including arsenite (As(III)), monomethylarsonic acid (MMA), dimethylarsinic acid (DMA), arsenate (As(V)), selenocystine (SeCys), selenomethionine (SeMet) and selenate (Se(IV)) was developed with high performance liquid chromatography-hydride generation-double channel atomic fluorescence spectrometry (HPLC-HG-AFS). An anion-exchange column (PRP-X100) with eluent of 10 mmol/L NH₄H₂PO₄ containing 2.5%(v/v) methanol was employed to separate these species within 12 min. The detection limits of As(III), DMA, MMA, As(V), SeCys, SeMet and Se(IV) were 1, 3, 2, 3, 4, 18 and 3 μg/L (200 μL of injection), respectively. The relative standard deviations in five independent determinations varied from 1.9% to 6.1% for arsenic and selenium species at the concentration levels of 100 and 300 μg/L. The proposed method was applied to analyze the selenium yeast tablet and human urine samples. The recoveries from spiked selenium yeast tablet and urine samples ranged from 88% to 105% and from 83% to 108%, respectively. The results showed that this method can be used for determining arsenic and selenium species in urinary metabolites and drug samples in daily analysis conveniently.

Key words [high performance liquid chromatography \(HPLC\)](#) [double-channel atomic fluorescence spectrometry](#) [speciation](#) [arsenic](#) [selenium](#)

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