技术及应用

适用于微分析质控的标准物质的探索

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摘要 本工作选择一种水系沉积物的天然基体进行研究,经研磨、筛选、粒径分布测定后,进行了样品分装和灭菌。结合NAA-PIXE-SRXRF 3种核分析技术,在跨越9个量级,即亚g到亚ng的取样量范围内,对这种基体进行了多元素取样行为研究。对准确可称的取样量(几百mg至1 mg),利用INAA测定了30余种元素,其中,16种元素的取样不确定度小于1%;利用常规PIXE和微束SRXRF结合的方法,对不可准确称量的更小取样量(<1mg)下的多元素取样行为进行研究。根据入射粒子和待测元素特征X射线在基体中的吸收系数,对实验条件下不同元素的有效取样量进行了估算。初步结果表明:在有效取样量几百 μ g的条件下,取样不确定度好于1%的有7种元素,在亚ng到几十 π g的取样量下,Fe、Cu、Rb的取样不确定度均小于10%,其中,Rb的取样不确定度小于5%。本工作将为研制适用于固体取样的微分析质控天然基体的标准物质提供一个可能的途径。

关键词 <u>微分析质控</u> <u>标准物质</u> <u>核分析方法</u> <u>多元素</u> <u>取样行为</u> 分类号

Preliminary Study on Reference Material for Microanalysis Quality Control

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Abstract

DOI

Sampling behavior of multielements in a stream sediment matrix was studied with sample sizes ran ging from several hundred mg down to sub-ng by using a combination of INAA-PIXE-SRXR F. For accurately weighable sample sizes (>1 mg), INAA was used. On the condition of samplin g sizes unable to be accurately weighed (<1 mg), PIXE and SRXRF were used with the effective sample sizes being estimated according to the ranges of incident particles and absorption coefficients of characteristic X-rays. Preliminary results show that the sampling uncertainties for 16 elements are better than 1% on the sample sizes above 1 mg by using INAA, those for 7 elements are better than 1% on the sample sizes of sub-mg by using PIXE, and for 3 elements Cu, Fe, and R b, they are better than 10% on the sample sizes equal to or larger than 7.5, 9.6, 42.4 ng, respectively. This study may pave the way to preliminary establish a new kind of CRMs suitable for quality control of solid sampling microanalysis.

Key words microanalysis quality control reference material nuclear analytical t echniques multielements sampling behavior

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