

Determination of U, Th and K for Quaternary Sediments by Inductively Coupled Plasma Atomic Emission Spectrometry-Mass Spectrometry

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The inductively coupled plasma mass spectrometry (ICP-MS) and optical emission spectrometry (ICP-OES) are common methods for determining potassium, uranium and thorium contents of sediments in luminescence or ESR dating. The quality of the analysis results depend partially on the pretreatment processes of samples. In this paper, eight national standard sediment samples (GSS-1a~8a) from Yichun of Heilongjiang, Baiyunebo of Inner Mongolia, Laizhou of Shandong, Yizhou of Guangxi, Liuyang of Hunan, Yangchun of Guangdong, Xuwen of Guangdong and Luochuan of Shaanxi were pretreated with hydrofluoric acid and nitric acid at different ashing temperatures, and ¹⁸⁷Re was used as the internal standard element in ICP-MS to determine U and Th elements, and ICP-OES to determine K elements. The results showed that the calcination temperatures of 350 °C, 450 °C and 550 °C did not affect the values of U, Th and K for the samples with a loss of less than 10 %. However, for the samples with a loss of more than 10 %, especially GSS-1a and GSS-7a, the ratios of the measured values of U, Th and K to the standard values after pretreatment at 450 °C were less than 0.95, while the ratios at 350 °C were less than 0.90. However, the measured results of the digestion solution after ashing at 550 °C were consistent with the standard values within the error range. Moreover, the precision (RSD<1.3%) and stability of the determination results are better.

At the same time, this three digestion methods were also applied to the comparative of sediment samples with high organic matter collected from peat land of Dajiu Lake. This revealed that the dry ashing-hydrofluoric acid-nitric acid digestion system was more effective, with less acid addition, less interference and stable test results, which was suitable for the detection of a large number of quaternary sediment samples by ICP-MS and ICP-OES.