

Protactinium determination in manganese crust VA13/2 by thermal ionization mass spectrometry (TIMS)

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Here we present the first high precision ^{231}Pa measurements in a manganese crust applying thermal ionization mass spectrometry (TIMS) using the double filament technique. The detection limit using TIMS is at least one order of magnitude lower, the statistical uncertainty 6 to 8 times better than for conventional α -spectrometry.

The developed method combines an easy chemical sample preparation (yields of $92 \pm 7\%$) with the possibility to measure down to ten fg of protactinium. The detection is limited to approx. 2 or 3 fg of ^{231}Pa due to instrumental background. Another limiting factor is the total procedural blank. From regularly measured blank samples an amount of $4.9 \pm 0.5\text{fg}$ ^{231}Pa was estimated for the whole procedure of sampling and chemical separation.

While handling extreme low ion beam intensities

(down to 1cps) effects of counting statistics had to be considered.

With the new determination limit of approx. 10fg older sections of manganese crust VA13/2 from the Northern Equatorial Pacific could be measured precisely for their ^{231}Pa concentration. Significant variations of the $^{231}\text{Pa}_{\text{xs}}$ activity during the past 150 ka are corroborated by existing α -spectrometric data. A constant growth rate between 0–450 ka indicates a variable protactinium flux from the water column into manganese encrustations. Thus, $^{231}\text{Pa}_{\text{xs}}$ is not suitable for dating marine Mn/Fe deposits.

This method should be well applicable for the protactinium determination in smallest geological samples and has widespread applications e.g. Pa/U dating of continental and marine carbonates.