

Improving precision, accuracy and sample throughput of small sample measurements with TIMS

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The precise and accurate measurement of small sample amounts is required by numerous applications in isotope geochemistry. Technological developments have enabled high precision (i.e. <10 ppm 2 RSD) isotopic measurements with thermal ionisation mass spectrometry (TIMS) using large sample amounts, on the order of several hundreds of nanograms. The precision of isotope ratios from small sample sizes is, however, compromised by the ion yield of the analytical technique and the noise of the detection system. Furthermore, isotope measurements are often hampered by the necessity to be run in manual operating mode requiring high labour costs and reducing sample throughput.

Using the examples of Nd, Sr, Os and U this study investigates different strategies to improve precision, accuracy and sample throughput for sample sizes smaller than 1 ng.

Ion yields could be enhanced by using different sample additives (activators), loading techniques, measuring different element species and optimizing filament heating techniques. Different activators like H₃PO₄, Si-gel or T₂O₅ are known to enhance and stabilize the ion beam and could enhance ion yield by up to a factor of ten.

Higher sample throughput and lower labour costs could be realised by using different analytical methods. The total evaporation (TE) technique with internal mass fractionation correction is able to yield precisions at the limit of counting statistics with acquisition times of less than 15 minutes per sample. The static multicollecion of low intensity ion beams using Thermo Scientific™ 10¹³ Ohm Amplifer Technology™ overcomes the limitations of sequential single collector measurements by enabling 100% duty cycle measurements and could reduce acquisition times by a factor of up to 7. Automatic sample heat-up procedures using a reference pyrometer enable unattended measurements.

Accuracy and precision of isotope ratios of small ion beams could be optimized using different detection systems. Optimum range of use for ion counters, 10¹³ and 10¹¹ Ω amplifiers are discussed together with the design of methods for small sample sizes that includes baseline and acquisition times.