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Application Note

System Setup and Parameter Optimization for a Desolvating Nebulizer Unit with Multicollector Inductively Coupled Plasma Mass Spectrometry for Stable and Radiogenic Isotope Ratio Measurements

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Introduction

Multicollector ICP-MS instruments are widely used in geochemistry for high precision isotope ratio measurements. Signal enhancement and/or interference reduction (ex. oxides and hydrides) is often necessary for useful measurement of low abundant isotopes and mass-limited samples.

This work will describe the setup and optimization of a new desolvating nebulizer accessory for multicollector ICP-MS. Important accessory benefits include inert wetted components for HF-containing samples, heated inert spray chamber and membrane desolvator for optimum sample transport efficiency, and mass flow controllers with computer software for Ar sweep and N_2 addition gases.

The nebulizer accessory is applied with multicollector ICP-MS for uranium-thorium dating, as is commonly used for cave and coral calcite and aragonite samples.

Instrumentation

The multicollector (MC) ICP-MS instrument used was a ThermoFisher Scientific Neptune, equipped with the Jet sampler and X interface skimmer cones but not with the high performance interface vacuum pump. The desolvating nebulizer system coupled to the Neptune was the Aridus3 (Teledyne CETAC Technologies); a CFlow-100 PFA nebulizer was connected to the PFA spray chamber of the Aridus3 and samples were self-aspirated in a manual mode.

A front view of the Aridus3 is depicted in Figure 1. The CFlow-100 PFA nebulizer and PFA spray chamber are on the left side of the Aridus3, behind a transparent door. Argon gas supply to the PFA nebulizer is from the host Neptune MC-ICP-MS and is under computer control from the Neptune software.

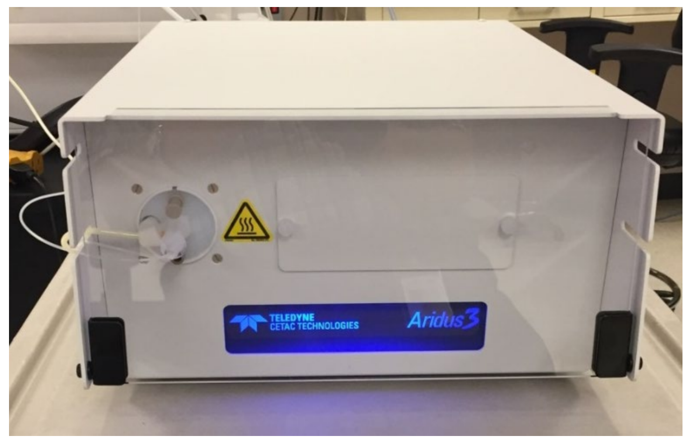

Figure 1. Teledyne CETAC Aridus3 Desolvating Nebulizer System – Front View

The Aridus3 was placed on the sample introduction bench of the Neptune MC-ICP-MS, as shown in Figure 2. A sample introduction line was connected between the outlet of the Aridus3 and the MC-ICP-MS torch.

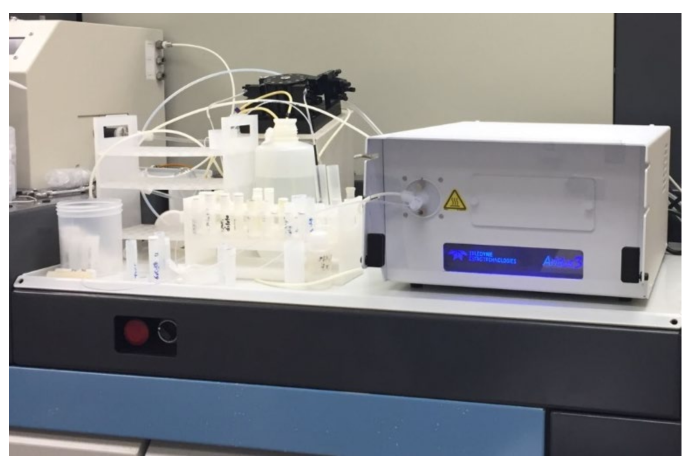

Figure 2. ThermoFisher Neptune MC-ICP-MS & Teledyne CETAC Aridus3 Desolvating Nebulizer System

An argon sweep gas supply and a nitrogen addition gas supply were connected to the back panel of the Aridus3, and both gas flows were set using mass flow controllers built into the Aridus3. An Aridus3 computer software program enabled setting of these gas flows as well as the temperature settings of the PFA spray chamber and the membrane desolvator oven module. This module can be removed for ease of cleaning, as shown in Figure 3.

Figure 3. Aridus3 Removable Membrane Oven Module

Neptune MC-ICP-MS and Aridus3 operating conditions were optimized using a 1 µg/L uranium standard; MC-ICP-MS operating conditions are listed in Table 1. A computer screen picture (Figure 4) of the Aridus3 software (AridusLink) shows argon and nitrogen gas flows and system temperature settings. The uptake rate of the CFlow-100 PFA nebulizer was measured gravimetrically at 99 µL/min using deionized water.

Table 1. Thermo Neptune MC-ICP-MS Conditions:

Parameter	Value
ICP Power	1200 W
Coolant Gas	15.00 L/min
Auxiliary Gas	1.30 L/min
Sample Gas	0.75 L/min
Interface	Jet Type*
Extraction	-2000 V
Focus	-642.0 V
X-Defl.	7.61 V
Y-Defl.	0.02 V
Shape	193.00 V
Rot Quad 1	0.01 V
Source Offset	-10.00 V

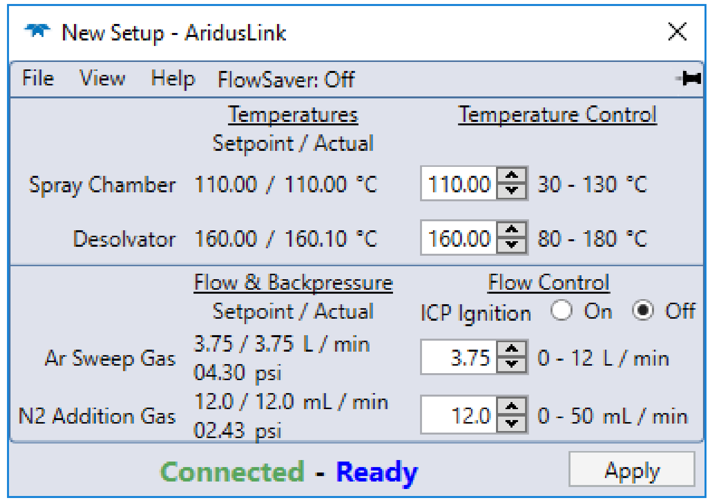

Figure 4. AridusLink Software Control Screen

Sample Type and Preparation

Sample type (BC9-88) was a sub-sample of calcite milled 88 mm from the top of stalagmite BC9 (sample obtained by V. Polyak with US federal permit granted by Carlsbad Cavern National Park, New Mexico USA). The sub-sample was collected to screen this sample for its age. 130 mg of the calcite powder was dissolved, spiked with a mixed solution of ^{233}U-^{236}U-^{229}Th, and the uranium and thorium were cleaned and separated with an anion resin column chemistry. Uranium and thorium were analyzed separately. A picture of the sample is given in Figure 5.

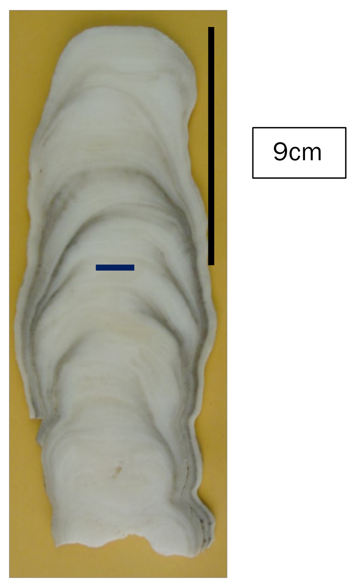

Figure 5. Stalagmite BC9, Carlsbad Cavern, New Mexico USA

Using the Neptune MC-ICP-MS with the Aridus3, the results are given in Table 2. Note that 8 cycles were run per block with an integration time of 20 seconds per cycle.

Table 2. Results for Stalagmite Sample BC9-88

Parameter	Value
^{238}U concentration	0.5205 ± 0.0005 ppm
^{232}Th concentration	0.00320 ± 0.00004 ppm
$^{230}Th/^{232}Th$	18.6 ± 0.3*
$^{230}Th/^{238}U$	0.0374 ± 0.0003*
$\delta\ ^{234}U_{measured}$	1554 ± 3 ‰
$\delta\ ^{234}U_{initial}$	1561 ± 3 ‰
Age uncorrected	1608 ± 14 B2k
Age corrected	1210 ± 120 yr B2k

* = activity ratio, Yr BP = years before 2000CE

Summary

Sample BC9-88mm was dated with a Thermo Neptune multicollector ICP-MS. A CETAC Aridus3 desolvating nebulizer system was used to enhance the signal by 4x above that signal produced from a quartz spray chamber having an equivalent nebulizer feeding into the spray chamber. This enhancement of signal allows for the use of smaller sample sizes and spike/sample ratios, and also reduces run time.

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