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Technical Note

Multicollector ICP-MS with a New Desolvating Nebulizer System for U-Th Series Measurements: an Optimization Procedure

INTRODUCTION

Multicollector ICP-MS instruments are very specialized systems for high precision isotope ratio measurements. For useful measurement of low abundant isotopes and mass-limited samples, signal enhancement is often necessary. In addition, sample preparation and/or sample aerosol desolvation may be needed to reduce or eliminate mass spectral interferences such as oxides and hydrides.

This technical note will review an optimization procedure for uranium-thorium series dating that establishes satisfactory system performance with regards to signal intensity and stability and isotope ratio accuracy and precision. This procedure is a necessary step before analysis as sample types (ex. speleological samples, corals) can be unique and very expensive to acquire. A new desolvating nebulizer system is used as part of this procedure, with benefit of built-in mass flow controllers under computer control for Ar sweep gas and N_2 addition gas flows.

INSTRUMENTATION

Multicollector ICP-MS: ThermoFisher Scientific Neptune with Jet Interface

Desolvating Nebulizer System: Teledyne CETAC Aridus3

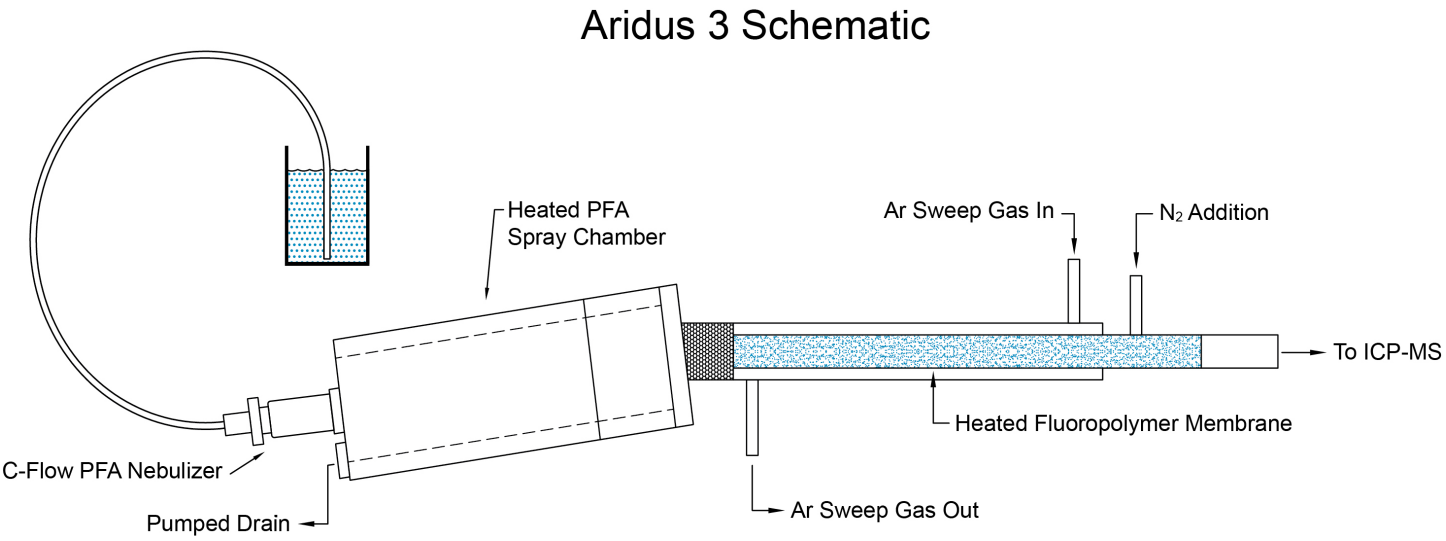

Figure 1. Teledyne CETAC Aridus3 Schematic: Aridus3 core components consist of a PFA nebulizer, a heated PFA spray chamber, and a heated fluoropolymer membrane desolvator; built-in mass flow controllers set the Ar sweep gas and N_2 addition gas flows.

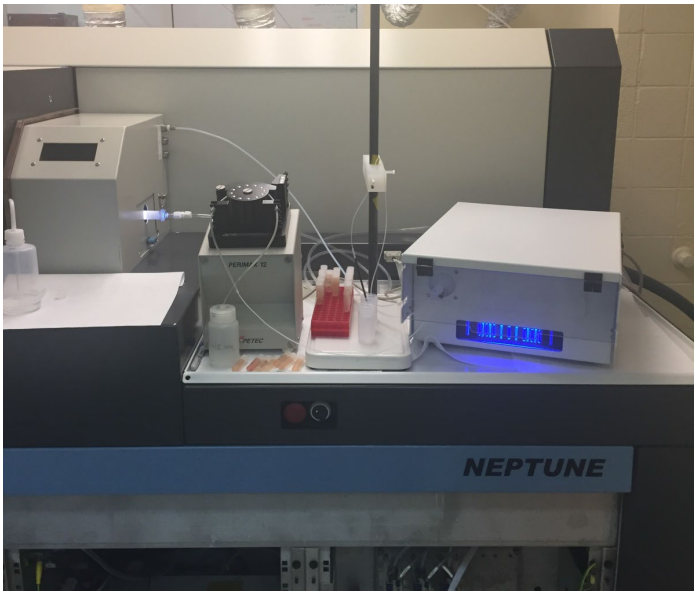

Figure 2. ThermoFisher Scientific Neptune MC-ICP-MS & Teledyne CETAC Aridus3 (beta version, at far right with blue ready status light)

Table 1. Operating Conditions

Thermo Neptune MC-ICP-MS	
RF Power	1100 W
Coolant Gas	15.00 L/min
Aux. Gas	0.70 L/min
Sample Gas	0.90 L/min
X-Pos	-1.680 mm
Y-Pos	0.370 mm
Z-Pos	2.700 mm
Interface	Jet Type
Extraction:	- 2,000 V
Focus:	-698.0 V
X-Defl:	0.85 V
Y-Defl:	-0.58 V
Shape:	220.00 V
Rot Quad1:	-3.16 V
Source Offset:	15.00 V

Teledyne CETAC Aridus3 (beta unit)	
PFA Nebulizer	C-Flow 50
Uptake Rate	56 µL/min
Spray Chamber Temp	110°C
Membrane Oven Temp	140°C
Ar Sweep Gas	3.90 L/min
N_2 Addition Gas	3.0 mL/min

STANDARD REFERENCE SOLUTION

A special standard solution was prepared from CRM 112-A Uranium (normal) Metal Assay and Isotopic Standard (New Brunswick Laboratory, USDOE). CRM-112A contains certified levels of ^{234}U, ^{235}U, and ^{238}U; the resulting solution is then spiked with known levels of ^{233}U and ^{236}U. The ^{233}U and ^{236}U concentrations are ~ 0.0057 pmol/g and that of ^{235}U ~ 0.08 pmol/g

ARIDUS3 (BETA-UNIT) SETUP

The Aridus3 was placed on the sampling stage area of the Thermo Neptune MC-ICP-MS as depicted in Figure 2. A C-Flow 50 PFA nebulizer was connected to the nebulizer port of the Aridus3 PFA spray chamber; sample gas (nebulizer gas) supply to the C-Flow nebulizer was via the Thermo Neptune MC-ICP-MS. A liquid drain line from the front of the PFA spray chamber was attached to the host Neptune peristaltic pump. Argon sweep gas and N_2 addition gas supplies were connected to the back panel of the Aridus3 and the Ar sweep gas out line connected to the trap bottle located next to the Neptune. A sample out line from the Aridus3 to the ICP-MS torch was established via a common 12/5 glass socket adapter.

Aridus Link software was loaded onto the host Neptune MC-ICP-MS computer, and communication established between the computer and the Aridus3 via a USB to RS-232 serial converter cable. The Aridus3 was switched to 220V voltage input and powered on; the pre-set heater temperatures of 110°C (PFA spray chamber) and 140°C (membrane oven) were established in approximately

20 minutes (indicated by blue-color Ready Status bar on front panel of the Aridus3).

A picture of the Aridus Link software control screen is shown in Figure 3. This small screen allows adjustment of all Aridus3 gas flows and temperatures via the Neptune software main page without manual changes of the Aridus3.

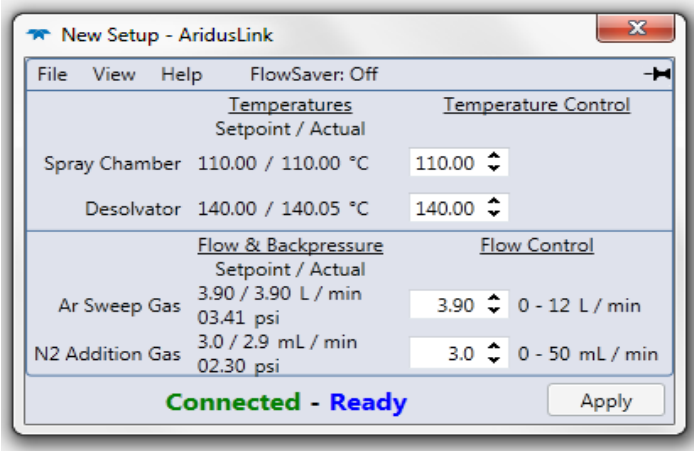

Figure 3. Aridus Link Control Screen

$^{235}U^{+}$ SIGNAL INTENSITY OPTIMIZATION

$^{235}U^{+}$ signal was optimized by adjustments to the Aridus3 Ar sweep gas and N_2 addition gas flows and Neptune parameters such as ion optic voltages, ICP torch position, and sample gas flow to the C-Flow 50 PFA nebulizer; a complete parameter list is given in Table 1. Aridus3 spray chamber and desolvator temperatures were set at 110°C and 140°C, respectively. A screen capture of a ~10 minute $^{235}U^{+}$ signal scan (2 blocks, 500 cycles/block) using the SEM detector is given below in Figure 4. Integration time for $^{235}U^{+}$ was 0.131s with an average signal of 324,904 cps (2.5% RSD).

Integration times for other U isotopes are given in ( ): ^{233}U (0.262s), ^{234}U (1.049s), ^{236}U (0.262s).

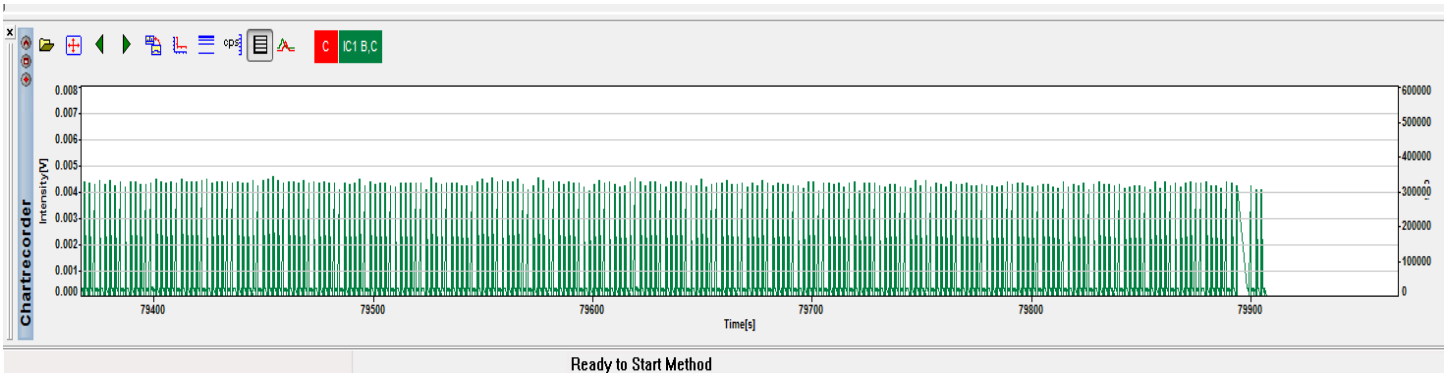

Figure 4. Scan of $^{235}U^{+}$ Signal using Thermo Neptune MC-ICP-MS with Teledyne CETAC Aridus3 (beta-unit)

SUMMARY OF TOTAL SYSTEM CONDITIONS AND PERFORMANCE FOR U-TH SERIES MEASUREMENTS

Tables 2a and 2b are a log summary of Neptune and Aridus3 system conditions prior to sample measurements, including isotope ratio accuracies, precision, and % ionization efficiency. The measured value of $\delta^{234}U$ (activity ratio $^{234}U/^{238}U$) is -38.5 +/- 1.6, in excellent agreement with the CRM 112-A certificate value of -38.7 +/- 1.5; ionization efficiency of 1.3% is above the desired 1.0% efficiency baseline.

Table 2a. System Conditions

% Counting Efficiency $^{235}U/^{235}U$	Abundance Sensitivity, $237/^{238}U$	% Ionization Efficiency
100	5.5×10^{-7}	1.3

Table 2b. Isotope Ratio Measurements

$\delta^{234}U$	$^{235}U/^{233}U$	$^{236}U/^{233}U$
-38.5 +/- 1.6	14.01 +/- 0.02	1.0225 +/- 0.0010

SUMMARY

The Aridus3 enables overall MC-ICP-MS system performance (signal intensity and stability; isotope ratio accuracy and precision) for uranium-thorium series dating measurements. New Aridus3 features such as computer control of gas flows aids system stability and eliminates manual system tuning via gas control knobs. Future work will apply the system to real samples such as corals.

ACKNOWLEDGEMENT

We would like to thank Dr. Pu Zhang and Dr. Larry Edwards of the University of Minnesota Department of Earth Sciences for their assistance in collection of the performance data with the Neptune MC-ICP-MS and the Aridus3 beta unit.