NORTHERN ARIZONA UNIVERSITY

August 12, 2011

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**SUBJECT:** Results for the determination of $^{234}U/^{238}U$ Activity ratios and U concentrations in aqueous samples by ICPMS

I am pleased to provide results for the determination of $^{234}U/^{238}U$ activity ratios and U concentrations in 31 water and aqueous solutions. These samples were received at NAU on June 30, 2011.

**Analytical Procedures:** U concentrations and U activity ratios were both determined by sector field ICPMS, using the same sample aliquot and in the same mass spectrometric run with addition of a U-233 spike. 25 mL aliquots of each sample solution were taken, and a U-233 tracer (prepared from IRMM 057) was added. The amount of the U tracer was 0.569 ng of isotopically pure U-233. The sample aliquot was mixed with 2.5 mL of 16 M nitric acid, and U was isolated using a laboratory-prepared 50 mg UTEVA resin column. The ICPMS was a VG Axiom MC equipped with an APEX high efficiency sample introduction system and a glass concentric nebulizer. The instrument was used in the single collector (electron multiplier) measurement mode. Seawater (North Atlantic) was used as a check sample; although this is not a sample with "certified" concentration, it is known that seawater contains approximately 3.3 µg/L U. Three aliquots of seawater resulted in U concentrations found of 3.0-3.1 µg/L, lending credence to the determination of U concentrations in unknown samples. A detection limit of 0.01 µg/L U was obtained for the water samples analyzed in this manner.

Three or more integrations were acquired for each sample; various types of duplicates (measurement only, preparation + measurement, and field duplicates) were also performed. In the mass spectrometric analysis, the mass discrimination was evaluated using runs of IRMM 056 (a U isotopic reference material having known $^{234}U$, $^{235}U$ and $^{238}U$ abundances) interspersed throughout the sample analyses. Mass discrimination was corrected using the results obtained from IRMM 056. The U activity ratio data were evaluated with runs of the seawater samples, compared to the known U AR in seawater (1.148 ± 0.002). The seawater solutions produced results of 1.16-1.18, suggesting some positive bias is present in the unknown sample results. However, this bias is not thought to be of much significance, given the very large variance of the U ratio results, along with the many extreme values that have been found.

Results are as shown on the following pages. I believe that you will find the $AR_{234238}$ results to be of use for your studies, and I would welcome the opportunity to discuss interpretations with you.

Respectfully submitted, Michael E Kett

Michael E. Ketterer, Ph.D.
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| My Lab ID | USGS ID | # Meas | AR 234/238 | AR sd | U conc, ug/L | U conc sd |
|---|---|---|---|---|---|---|
| 1 run 1 | D-631-U | 3 | 6.42 | 0.06 | 2.86 | 0.01 |
| 1 run 2 | D-631-U | 3 | 6.42 | 0.01 | 2.85 | 0.00 |
| 1 run 3 | D-631-U | 3 | 6.41 | 0.03 | 2.86 | 0.00 |
| 2 | B-692-U | 3 | 2.95 | 0.02 | 1.80 | 0.00 |
| 3 | B-690-U | 3 | | | < 0.01 | |
| 4 | D-687-U | 3 | 6.44 | 0.04 | 4.16 | 0.00 |
| 5 | D-685-U | 3 | 6.10 | 0.01 | 5.31 | 0.00 |
| 6 | B-3026-U | 3 | 4.13 | 0.05 | 2.08 | 0.01 |
| 7 | B-680-U | 3 | 3.77 | 0.02 | 6.14 | 0.00 |
| 38 | B-680-U lab dup | 3 | 3.75 | 0.02 | 6.54 | 0.01 |
| 8 | B-684-U | 3 | 4.07 | 0.02 | 24.65 | 0.17 |
| 21 | B-684-U-DUP | 3 | 4.07 | 0.02 | 20.37 | 0.02 |
| 9 | B-51-U | 3 | 45.80 | 5.56 | 0.03 | 0.00 |
| 11 | D-693-U | 3 | 4.08 | 0.24 | 0.01 | 0.00 |
| 12 | B-13-U | 3 | 81.45 | 8.44 | 0.01 | 0.00 |
| 13 | D-681-U | 3 | 5.33 | 0.03 | 8.99 | 0.03 |
| 14 run 1 | B-676-U | 3 | 1.36 | 0.02 | 54.08 | 0.64 |
| 14 run 2 | B-676-U | 3 | 1.36 | 0.01 | 53.80 | 0.11 |
| 14 run 3 | B-676-U | 3 | 1.36 | 0.01 | 53.78 | 0.34 |
| 15 | D-697-U | 3 | 18.93 | 0.38 | 0.08 | 0.00 |
| 16 | B-682-U | 3 | 4.07 | 0.04 | 13.70 | 0.05 |
| 17 | B-698-U | 3 | 1.72 | 0.01 | 49.70 | 0.73 |
| 18 | B-696-U | 3 | 8.78 | 0.05 | 0.54 | 0.01 |
| 20 | D-679-U | 3 | 1.84 | 0.02 | 13.25 | 0.01 |
| 19 | D-679-U-DUP | 3 | 1.82 | 0.01 | 13.30 | 0.04 |
| 22 | B-694-U | 3 | 15.87 | 0.80 | 0.08 | 0.00 |
| 23 | B-689-U | 3 | 6.01 | 0.04 | 2.91 | 0.01 |
| 24 | D-691-U | 5 | 2.67 | 0.76 | 0.02 | 0.00 |
| 25 | B-619-U | 3 | 7.51 | 0.03 | 1.60 | 0.00 |
| 26 | B-688-U | 3 | 15.54 | 1.58 | 0.01 | 0.00 |
| 27 | D-695-U | 3 | 3.33 | 0.04 | 2.82 | 0.01 |
| 10 | D-695-DUP-U | 3 | 3.30 | 0.01 | 2.87 | 0.01 |
| 28 | B-678-U | 3 | 1.49 | 0.01 | 34.40 | 0.20 |
| 29 | B-NBA-U | 3 | 1.88 | 0.02 | 5.32 | 0.03 |
| 40 | B-NBA-U lab dup | 3 | 1.91 | 0.01 | 5.23 | 0.01 |
| 30 | D-705-U | 3 | 4.85 | 0.04 | 7.91 | 0.03 |
| 31 run 1 | D-706-U | 3 | 18.45 | 0.20 | 0.08 | 0.00 |
| 31 run 2 | D-706-U | 3 | 18.35 | 0.43 | 0.08 | 0.00 |
| 31 run 3 | D-706-U | 3 | 18.37 | 0.50 | 0.08 | 0.00 |
| 35 | Seawater | 3 | 1.16 | 0.01 | 3.07 | 0.01 |
| 36 | Seawater | 3 | 1.18 | 0.01 | 3.06 | 0.01 |
| 37 | Seawater | 3 | 1.18 | 0.02 | 3.03 | 0.00 |

Uncertainties are +/- one sd of the indicated number of measurements.

U was not detected in Sample B-690-U at an LOD of 0.01 ug/L.

**Replicates**

Three sequential blocks were measured with the MS for the same sample preparation (mass spec only replicate)

A second aliqout of the same field sample was prepared and analyzed independently.

Field duplicate.