



February 20, 2012

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SUBJECT: Results for the determination of $^{234}\text{U}/^{238}\text{U}$ Activity ratios and U concentrations in powdered soil/rock/sediment samples by ICPMS

I am pleased to provide results for the determination of $^{234}\text{U}/^{238}\text{U}$ activity ratios and U concentrations in 31 powdered soil/rock/sediment samples. These samples were received at NAU on November 4, 2011.

Analytical Procedures: Samples were dissolved for the analysis as follows: a nominal 0.2 g aliquot of powdered sample was weighed into a small borosilicate vial, and the material was dry-ashed at 600°C for three hours to remove organic matter. The residue was transferred to a 50 mL polypropylene centrifuge tube; 2.5 mL of 16 M HNO_3 and 1.5 mL of 29 M HF were added; the mixture was heated at 80°C for 16 hours in a convection oven. Following heating, 10 mL of water and 1.2 g of solid H_3BO_3 were added; the mixtures were diluted to 50 mL and heated briefly in a microwave oven to facilitate dissolution of the boric acid and its reaction with the excess HF.

For determination of U concentrations, a 0.500 mL aliquot of the digest was diluted to 50 mL in a solution containing 10 $\mu\text{g}/\text{L}$ of Ir as an internal standard; the diluted sample solutions were analyzed and compared to matrix-matched standards of U and Ir. For determination of AR_{234238} , a 10 mL aliquot of the sample digest was mixed with 1 mL of 16 M nitric acid, and U was isolated using a laboratory-prepared 30 mg TRU resin column.

U concentrations and U activity ratios were both determined by quadrupole ICPMS. The ICPMS was used with a self-aspirating concentric Teflon nebulizer and a Peltier-cooled glass spray chamber. For determination of U concentrations, the ions ^{193}Ir , ^{235}U and ^{238}U were measured in a peak-jump mode with a 10 ms dwell time and 300 sweeps per integration with 3 integrations per sample. Most samples were determined using the ^{238}U calibration curve, though a few of the high concentration samples were determined using a calibration curve prepared using the ^{235}U intensity.

For determination of AR_{234238} , the ions ^{234}U and ^{235}U were measured in a peak-jump mode with a 10 ms dwell time and 2000 sweeps per integration with 3 or more integrations per sample. The measured $^{234}\text{U}/^{235}\text{U}$ atom ratio was corrected for mass discrimination and then converted to AR_{234238} using the known $^{235}\text{U}/^{238}\text{U}$ atom ratio (137.88 ± 0.03) and the known $^{234}\text{U}/^{238}\text{U}$ atom

ratio at secular equilibrium of $(0.00005472 \pm 0.000011)$. Mass discrimination was evaluated and corrected using the results obtained for a modern coral solution ($AR_{234238} = 1.148 \pm 0.002$).

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. Ketterer, Ph.D.
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My Lab ID	USGS ID	AR 234/238	AR sd	U conc, ug/g	U conc sd
1	C-355192	1.954	0.029	2.31	0.02
2	C-355212	1.028	0.011	12.6	0.1
3	C-355210	1.050	0.002	68.1	0.3
4	C-355191	1.148	0.011	8.20	0.01
5	C-355195	0.953	0.003	454	2
6	C-355183	0.848	0.015	5.91	0.03
7	C-355190	0.976	0.023	2.84	0.00
8	C-355184	0.988	0.012	2.17	0.02
9	C-355196	0.733	0.006	57.4	0.1
10	C-355213	0.949	0.022	2.62	0.02
11	C-355202	0.817	0.011	9.18	0.04
12	C-355198	1.185	0.015	6.45	0.02
13	C-355189	1.085	0.009	3.88	0.04
32	C-355189 dup	1.104	0.003	4.13	0.03
14	C-355199	1.257	0.017	1.43	0.01
15	C-355185	0.960	0.023	2.02	0.05
16	C-355205	2.892	0.026	0.87	0.01
17	C-355197	1.010	0.011	2.15	0.01
18	C-355204	0.946	0.006	18.1	0.1
19	C-355186	1.693	0.012	4.02	0.03
20	C-355194	1.067	0.001	3.78	0.01
21	C-355206	1.583	0.014	7.95	0.04
33	C-355206 dup	1.616	0.004	8.30	0.06
22	C-355201	2.133	0.007	15.9	0.1
23	C-355203	1.131	0.010	3.17	0.02
24	C-355187	0.766	0.001	36.1	0.3
25	C-355188	0.769	0.003	80.0	0.4
26	C-355211	1.010	0.008	5.28	0.05
27	C-355207	1.186	0.006	1.97	0.01
28	C-355208	1.054	0.001	822	2
29	C-355209	1.058	0.001	7809	27
34	C-355209 dup	1.057	0.004	7445	32
30	C-355193	1.197	0.024	1.18	0.04
31	C-355200	0.800	0.003	76.3	0.3

Uncertainties are +/- one standard deviation of three sequential measurements (no fill).

Uncertainties are +/- one standard deviation of two or three sequential blocks, each with 3 sequential measurements (blue fill).

A duplicate of the entire analytical procedure was performed for these samples (pink fill).