

Supplementary Materials: Matrix-Matched Iron-Oxide Laser Ablation ICP-MS U-Pb Geochronology Using Mixed Solution Standards

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Supplementary Text File S1. Additional analytical procedures for calculating and certifying the Pb/U ratios.

The mixed solution standard was made from single natural U and Pb solutions. The concentrations of these two solutions were calibrated using Q-ICP-MS at Adelaide Microscopy.

- Q-ICP-MS results: *Pb concentration of the mother bottle* = 9985 ppb; *U concentration of the mother bottle* = 9994 ppb.

The Pb isotope ratios were analyzed by MC-ICP-MS at Nanjing University.

- MC-ICP-MS results: $^{206}\text{Pb}/^{204}\text{Pb} = 17.9895 \pm 0.0005$; $^{207}\text{Pb}/^{204}\text{Pb} = 15.5847 \pm 0.0005$; $^{208}\text{Pb}/^{204}\text{Pb} = 38.0495 \pm 0.0013$; $^{207}\text{Pb}/^{206}\text{Pb} = 0.86632 \pm 0.00004$.

The constant $^{238}\text{U}/^{235}\text{U}$ (137.88), in combination with the Pb isotope ratio results, allows us to calculate $^{206}\text{Pb}/^{238}\text{U}$.

To achieve this, we must calculate the atomic weight of the Pb and U solution standards:

$$X = \text{Proportion of isotopes}; M = \text{Mass of isotope}$$

- Pb atomic weight = $X^{204} \times M^{204} + X^{206} \times M^{206} + X^{207} \times M^{207} + X^{208} \times M^{208}$
- U atomic weight = $X^{235} \times M^{235} + X^{238} \times M^{238}$

To then calculate the ratio of ^{206}Pb and ^{238}U , the correct amount of Pb and U standard solution to create a concentration compatible with the sample to be analyzed must be weighed.

Con = Concentration of element; W = Weight of solution

$$\frac{\text{Pb}_{206}}{\text{U}_{238}} = \frac{\frac{\text{Con}_{\text{pb}} \times W_{\text{Pb-sol}}}{\text{Pb}_{\text{atomic weight}}} \times X_{206}}{\frac{\text{Con}_{\text{U}} \times W_{\text{U}}}{\text{U}_{\text{atomic weight}}} \times X_{238}} \quad (1)$$

$$\frac{\text{Pb}_{207}}{\text{U}_{235}} = \frac{\frac{\text{Con}_{\text{pb}} \times W_{\text{Pb-sol}}}{\text{Pb}_{\text{atomic weight}}} \times X_{207}}{\frac{\text{Con}_{\text{U}} \times W_{\text{U}}}{\text{U}_{\text{atomic weight}}} \times X_{235}} \quad (2)$$

$$^{207}\text{Pb}/^{206}\text{Pb} = 0.86632 \pm 0.00004 \text{ (measured by MC-ICP-MS)}$$

$$^{206}\text{Pb}/^{238}\text{U} = 0.286356$$

The following tables contain the Pb isotope ratios and U-Pb concentrations.

Table S1. Pb isotope ratios of Pb standard solution.

| Name | Pb ^{206/204} | Pb ^{206/204} SE (abs) | Pb ^{207/204} | Pb ^{207/204} SE (abs) | Pb ^{208/204} | Pb ^{208/204} SE (abs) |
|--------------------|-----------------------|--------------------------------|-----------------------|--------------------------------|-----------------------|--------------------------------|
| NIST987-01 | 16.939 | 0.001 | 15.488 | 0.001 | 36.693 | 0.002 |
| NIST987-02 | 16.937 | 0.001 | 15.487 | 0.001 | 36.690 | 0.002 |
| Pb-1 | 17.990 | 0.001 | 15.585 | 0.001 | 38.049 | 0.001 |
| Pb-2 | 17.991 | 0.001 | 15.586 | 0.001 | 38.052 | 0.001 |
| Pb-3 | 17.988 | 0.000 | 15.583 | 0.000 | 38.047 | 0.001 |
| NIST987-03 | 16.938 | 0.001 | 15.488 | 0.001 | 36.692 | 0.002 |
| Average of Samples | 17.990 | 0.001 | 15.585 | 0.001 | 38.050 | 0.001 |

Table S2. U and Pb concentration of standard solutions. Conc. = Concentration.

| Type | Sample Name | ²⁰⁴ Pb | | ²⁰⁶ Pb | | ²⁰⁷ Pb | | ²⁰⁸ Pb | | ²³⁵ U | | ²³⁸ U | | ¹¹⁵ In (ISTD) | | | Average Pb | | Average U | | Pb/U |
|--------|-----------------|-------------------|-----------|-------------------|-----------|-------------------|-----------|-------------------|-----------|------------------|-----------|------------------|-----------|--------------------------|-----------|-----------------|------------|-----------|-----------|-----------|------|
| | | Conc. | Conc. RSD | Conc. | Conc. RSD | Conc. | Conc. RSD | Conc. | Conc. RSD | Conc. | Conc. RSD | Conc. | Conc. RSD | Conc. | Conc. RSD | ISTD Recovery % | Conc. | Conc. RSD | Conc. | Conc. RSD | |
| CalBlk | Blank | 0.00 | N/A | 0.00 | N/A | 0.00 | N/A | 0.00 | N/A | 0.00 | N/A | 0.00 | N/A | - | - | 100 | - | - | - | - | - |
| CalStd | Standard-1 ppb | 0.98 | 8.07 | 0.95 | 2.34 | 0.96 | 4.70 | 0.95 | 2.01 | 0.98 | 17.39 | 1.00 | 1.95 | - | - | 99 | 0.95 | 0.19 | 0.99 | 1.84 | 0.96 |
| CalStd | Standard-2 ppb | 1.97 | 6.05 | 1.97 | 1.74 | 1.96 | 3.03 | 2.00 | 1.88 | 2.04 | 6.45 | 2.02 | 0.84 | - | - | 97 | 1.98 | 1.14 | 2.03 | 0.40 | 0.97 |
| CalStd | Standard-5 ppb | 5.06 | 3.76 | 5.01 | 1.92 | 4.98 | 2.46 | 5.07 | 2.24 | 4.95 | 5.22 | 5.11 | 2.66 | - | - | 100 | 5.02 | 0.86 | 5.03 | 2.31 | 1.00 |
| CalStd | Standard-10 ppb | 9.89 | 4.49 | 9.91 | 3.38 | 9.90 | 3.82 | 10.02 | 3.97 | 9.85 | 4.14 | 9.99 | 3.18 | - | - | 103 | 9.94 | 0.65 | 9.92 | 1.02 | 1.00 |
| CalStd | Standard-20 ppb | 19.42 | 2.66 | 19.63 | 2.47 | 19.58 | 2.62 | 19.98 | 2.17 | 19.34 | 3.52 | 19.97 | 1.80 | - | - | 106 | 19.73 | 1.09 | 19.65 | 2.29 | 1.00 |
| CalStd | Standard-50 ppb | 50.25 | 1.82 | 50.17 | 2.09 | 50.19 | 2.63 | 51.55 | 2.91 | 50.30 | 2.23 | 51.82 | 2.48 | - | - | 104 | 50.64 | 1.56 | 51.06 | 2.11 | 0.99 |
| Sample | Blank | <0.000 | N/A | <0.000 | N/A | <0.000 | N/A | <0.000 | N/A | <0.000 | N/A | <0.000 | N/A | - | - | 103 | - | - | - | - | - |
| Sample | 1 | 1.17 | 12.14 | 1.04 | 5.38 | 1.03 | 4.95 | 1.04 | 5.54 | 1.01 | 14.29 | 0.99 | 5.38 | - | - | 105 | 1.03 | 0.78 | 1.00 | 1.98 | 1.04 |
| Sample | 2 | 4.50 | 4.97 | 4.07 | 2.91 | 3.95 | 2.33 | 4.11 | 2.89 | 3.64 | 4.42 | 3.77 | 1.29 | - | - | 115 | 4.04 | 5.95 | 3.71 | 2.50 | 1.09 |
| Sample | 3 | 11.80 | 2.90 | 11.88 | 2.78 | 11.79 | 1.75 | 12.05 | 2.11 | 11.37 | 4.19 | 11.76 | 2.39 | - | - | 111 | 11.91 | 1.09 | 11.56 | 2.41 | 1.03 |
| Sample | 4 | 43.61 | 1.65 | 44.42 | 2.10 | 44.09 | 2.21 | 44.97 | 2.38 | 38.70 | 3.25 | 40.39 | 2.95 | - | - | 108 | 44.50 | 1.00 | 39.54 | 3.01 | 1.13 |

Table S3. Relation coefficient of standards.

| Tune Step | Mass | Name | Relation Coefficient | Detection Limit |
|-----------|------|------|----------------------|-----------------|
| 2 | 204 | Pb | 0.99990 | 0.10246 |
| 2 | 206 | Pb | 0.99996 | 0.06304 |
| 2 | 207 | Pb | 0.99995 | 0.07154 |
| 2 | 208 | Pb | 0.99999 | 0.06215 |
| 2 | 235 | U | 0.99987 | 0.09124 |
| 2 | 238 | U | 0.99998 | 0.03269 |



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