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Standardless LA-ICP-MS fission-track dating in apatite: comparison with uranium thin film and LA-ICP-MS ζ-calibration

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Abstract

Over the past two decades, the fission-track community has sought to calibrate the ages with LA-ICP-MS. This technique has not only made thermochronology faster but also enabled the determination of U-Pb ages and the measurements of trace elements which support geological interpretation. In this study we present an adjusted age equation for absolute LA-ICP-MS approach, herein referred to as the ψ-method, and compare the pooled and grain-by-grain ages with absolute approach based on nuclear reactor (uranium-thin-film based $\emptyset$-method) using one aliquot of Durango apatite. Both methods do not require a standard to obtain fission-track ages. In the absolute approach, every parameter in the age equation must be determined with certain precision. Therefore, an experiment was carried out to determine uranium concentration using matrix-matched reference materials (apatite MT-7) and compare with NIST SRM 610, which enables us to verify if there is the potential matrix interference for uranium determination. After that, a series of apatite ages were then calculated. For this purpose, two data sets were used: 1) standard ages (Durango, FCT and Limberg) as well as Mud Tank, which represent the low annealing samples (~ 10%) and 2) samples with moderate annealing (> 10%). In both cases, track length must be considered, as the reduced track length yields proportional reduced fission-track ages. For the first set, the ψ-method was the only method applied once the ages are well-known, while the ages from second set were compared with LA-ICP-MS zeta calibration (ζ_{ICP}). The results show a consistent age between methods.

1. Introduction

Fission-track thermochronology (FTT) is a method used to determine the age and thermal history of minerals. This is done by counting and measuring the length of fission tracks formed by spontaneous fission of ^{238}U over geological time. The minerals suitable for FTT contain trace amounts of uranium, which substitute for other elements in the crystal structure.

To determine their age, the number of fossil tracks is counted on a polished mineral surface after chemical etching. In the past, the External Detector Method (EDM) was the only method used to determine their uranium content. This involves mounting, polishing, etching spontaneous fission tracks, and then an external detector, usually a nearly uranium-free muscovite is attached to the surface. A set of samples is irradiated with thermal neutrons in a nuclear reactor, and spontaneous fission-tracks are measured in the mineral, and the induced fission-tracks are measured in the equivalent area in the uranium-free muscovite.

The most common method used to determine the fission-track age based on EDM is the ζ-calibration (Hurford, 1990), where age standard samples are irradiated together with the unknown samples. In this case, spontaneous and induced fission-tracks in both standard and unknown samples are used to calculate the ages.

Another two techniques to calculate the fission-track ages are through direct thermal neutron fluence measurements: 1) calibration of the standard glass (CN's and IRMM-540) against calibrated neutron fluence monitors such as uranium thin films (Ø-method), (*e.g.*, Iunes et al., 2002; Soares et al. 2014a) or 2) metallic foils like Au and Co (*e.g.*, Jonckheere, 2003; Enkelmann et al., 2005). In the case of standard glass calibrated against uranium thin films, the thermal neutron fluence is calculated using a uranium-free muscovite attached to a co-irradiated uranium doped standard glass (CN's or IRMM-540). The thermal neutrons induce ^{235}U fission in both mineral and standard glass. Thus, the ^{238}U can be determined considering the ratio $^{238}U/^{235}U = 1/137.88$ (Steiger and Jager, 1977).

The drawback of using EDM involves samples irradiation. The samples become activated during radiation, making it impossible to handle the samples until they reach the safety activity level. To overcome these complications, the fission-track community has been working on other methods for determining uranium concentration, such as the laser ablation inductively coupled plasma mass spectrometer (LA-ICP-MS) (Cox et al., 2000; Svojtka and Kosler, 2002; Kosler and Sylvester, 2003; Kosler, 2007, Hasebe et al. 2004; 2009; 2013; Hadler et al., 2009; Donelick et al. 2005; Chew and Donelick, 2012; Danhara and Iwano, 2013; Soares et al. 2013; 2014b; 2015, Cogné et al., 2019). This method avoids the cooling time of radioactive samples and the need for radiation protection measurements that are demanded by regulatory authorities. Furthermore, the analysis is faster, and the sample preparation is the same used for EDM.

In addition to age determinations, LA-ICP-MS measurements allow for the determination of a series of other isotopes, enabling to collect geochemical signatures and the application of other dating techniques such as U–Pb to the same grains (*e.g.*, Chew and Donelick, 2012; Ansberque et al., 2020; Bilhar et al., 2026). Such additional information is valuable for geological interpretation, especially for basin samples, which contain multiple apatite fission-track populations from various sources.

Establishing a reliable and robust LA-ICP-MS calibration has been a challenge, which is especially complicated when an absolute approach is applied. Some of the challenges are: 1) determination of revelation/observation parameters under an optical microscope to determine the spontaneous fission-track density (efficiency factor); 2) the role of annealing degree in the revelation/observation efficiency factor, once the diminished track length decreases the probability of fission-track fragments reaching the polished/etched surface; and 3) the absolute determination of uranium concentration.

In this paper, a series of experiments was performed to overcome all these described challenges: 1) We suggest an adjustment in the parameter in the absolute age equation of Soares et al. (2014b); 2) the evaluation of uranium and thorium concentrations in a Durango apatite (DR) sample reported by Chew et al. (2016) normalized using matrix matched reference material (MT-7;Soares et al., 2014b; 2015) and the NIST SRM 610, which enables us to detect any potential matrix effect on the determination of these elements, especially uranium used for age calculation; 3) we compare the resulting ages from a single aliquot of Durango apatite dating using two independent absolute dating methods: the uranium thin film (Ø-method) (Iunes t al., 2002; Soares et al., 2014a) and absolute LA-ICP-MS (ψ-method); 4) calculate the

ages of three standards and Mud Tank apatite samples (low annealing ~ 10%) with absolute ψ-method and compare them to reference/published ages and; 5) we compare the ψ-method resulting ages with ζ_{ICP} ages for two groups of samples (Obernsees and Lindau in Germany and Borborema in Brazil) that show moderate annealing (> 10%).

2. Calibration methods

The general age equation for the fission-track method is (Wagner and Van den haute, 1992):

$$t = \frac{1}{\lambda_\alpha} ln \left[1 + \left(\frac{\lambda_\alpha}{\lambda_f} \right) \left(\frac{N_S}{N_{238}} \right) \right]$$

1

,

In Eq. (1), *Ns* represents the number of spontaneous fission tracks present per unit volume in a mineral. N_{238} stands for the concentration of ^{238}U per unit volume in the same mineral. The equation also involves two physical constants, the total decay constant (λ_α), and the spontaneous-fission decay constant (λ_f). Direct volumetric fission tracks cannot be accessed directly. Instead, fission tracks are observed under an optical microscope in a polished surface. Thus, only fission-fragments that reached the surface can be revealed through etching. Thus, parameters such as the geometry (volume) of the mineral and the product *[ηq]* of observation (*η*) and etching (*q*) efficiencies must be considered. The *[ηq]* parameter is obtained by comparing the theoretical and experimental surface-projected fission-track length distributions (e.g., Jonckheere, 2003; Enkelmann and Jonckheere, 2003; Soares et al., 2013; 2014b).

Track observation is dependent on some factors (Wagner and Van den Haute, 1992; Hasebe et al., 2004): 1) the distance between the fissioned nuclei and the observed surface where tracks can be etched and observed; and 2) the range of fission-fragment in which latent tracks are formed. The first factor indicates that the efficiency of etching during the revelation of the tracks must be considered and the second factor must be considered once only part of the tracks are effectively etched. Jonckheere (2003) presents the average of latent track of 21.2 µm. In contrast, the mean confined fission-tracks are 16.25 µm for non-annealed Durango apatite, a deficit of ~ 23%.

The most common adopted approach for etched track range is $R_s = (L_{e-spt})\times 0.5$. However, considering the difference between latent and etched fission tracks described above, we suggest here a correction of this value considering the ratio (L_e/L_f). The average of non-annealed etched track length is (L_e) = 16.25 µm and the average of non-annealed latent track length is (L_f) = 21.2 µm (Jonckheere, 2003). Thus, the $L_e/L_f = 0.766$ and the $R_s = (L_{e-spt})\times(L_e/L_f) = (L_{e-spt})\times 0.766$.

Finally, the geometries involved for fission-track based on LA-ICP-MS is only the one related to the mineral, *i.e.*, $g_{4\pi}$ (= 1). Thus, considering the parameters described above, the fission-track age equation can be re-written as follows:

$$t_{\phi} = \frac{1}{\lambda} ln \left[1 + \left(\frac{\lambda_{\alpha}}{\lambda_{f}} \right) \left(\frac{1}{g_{4\pi} \left[\eta \, q \right]_{S} \left(L_{e} / L_{f} \right) R_{S} \times 10^{-4}} \right) \left(\frac{\rho_{S}}{N_{238}} \right) \right]$$

2

Considering the ^{238}U in the LA-ICP-MS is determined in ppm, we can re-write the N_{238} in the equation as (Hasebe et al., 2004):

$$t_{\psi \, ICP} = \frac{1}{\lambda} ln \left[1 + \left(\frac{\lambda_{\alpha}}{\lambda_{f}} \right) \left(\frac{1}{g_{4\pi} \left[\eta \, q \right]_{S} \left(L_{e} / L_{f} \right) R_{S} \times 10^{-4}} \right) \left(\frac{\rho_{S} \, M}{N_{A} 238_{U_{\mathrm{ppm}}} \times 10^{-6} d} \right) \right]$$

3

,

where N_A is the Avogadro number (6.022×10^{23} mol^{-1}); $^{238}U_{\mathrm{ppm}}$ is the ^{238}U concentration in the mineral determined using LA-ICP-MS (given in ppm), and d is the density of apatite (3.19 g.cm^{-3}). All constants and other measured parameters can be joined into a single parameter, $\boldsymbol{\psi}_{ICP}$. Eq. (3) becomes:

$$t_{\psi \, ICP} = \frac{1}{\lambda} ln \left[1 + \psi_{ICP} \left(\frac{\lambda_{\alpha}}{\lambda_{f}} \right) \left(\frac{\rho_{S}}{238_{U_{ppm}} \times R_{s}} \right) \right]$$

4

,

where:

$$\psi_{ICP} = \left(\frac{M}{N_{A} \times 10^{-6} d} \right) \left(\frac{1}{g_{4\pi} \left[\eta \, q \right]_{S} \left(L_{e} / L_{f} \right) \times 10^{-4}} \right)$$

5

.

It is essential to note that the absolute approach requires an independent determination of all parameters involved. Thus, there is no meaning to use the mean track length of induced confined or the mean track length of spontaneous fission tracks in a standard sample such as Durango apatite to reach absolute ages of an unknown sample. Therefore, to apply an appropriate correction, the R_s in Eq. (4) must be the mean of confined fission-track of the analyzed sample and, thus, this parameter must be explicit in the Eq. (4).

The pooled age associated with Eq. (4) can be obtained as:

$$t_{pooled-\psi\ ICP} = \frac{1}{\lambda} ln \left[1 + \psi\ _{ICP} \left(\frac{\lambda\ _{\alpha}}{\lambda\ _{f}} \right) \left(\frac{\sum\ (N_{Ss})}{\sum\ (A \times\ 238_{U\ ppm})} \right) \left(\frac{1}{R_s} \right) \right]$$

6

,

where N_{Ss} is the spontaneous fission-track measured in an etched surface and *A* is the fission-track count area. For the uncertainty of $\boldsymbol{\psi}_{ICP}$ parameter is given by:

$$\sigma\ _{\psi\ ICP} = \psi\ _{ICP} \times\ \sqrt{\left(\frac{\sigma\ _{[\eta\ q]_S}}{[\eta\ q]_S} \right)^2 + \left(\frac{\sigma\ _{L_e}}{L_e} \right)^2 + \left(\frac{\sigma\ _{L_f}}{L_f} \right)^2}$$

7

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The uncertainty of the ages for equations (4) and (6) is calculated from:

$$\sigma\ _{t\psi\ ICP} = t_{\psi\ ICP} \times\ \sqrt{\left(\frac{1}{N_S} \right) + \left(\frac{\sigma\ _{\psi\ ICP}}{\psi\ _{ICP}} \right)^2 + \left(\frac{\sigma\ _{238_{U\ ppm}}}{238_{U\ ppm}} \right)^2 + \left(\frac{\sigma\ _{R_S}}{R_S} \right)^2 + \left(\frac{\sigma\ _{f}}{f} \right)^2}$$

8

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Ideally, the ^{238}U determination using LA-ICP-MS must be by comparison with a well-characterized uranium matrix-matched reference material. Soares et al. (2015), after testing crystals of Durango, Mud Tank, Bahia, and others, presented two candidates for apatite uranium matrix-matched reference materials using LA-ICP-MS: one piece of Durango (Dur-2 with 7.44 µg. g^{-1} uranium) and one piece of Mud Tank (MT-7 with 6.88 µg. g^{-1} uranium – here preferred uranium matrix-matched reference materials) (with RSD < 1.5% for both apatite crystals). Alternatively, a standard glass (e.g., NIST SRM 610) can be used, which can be calculated as:

$$238_U = \left\{ \left(\frac{\left[\left(238_U / 43_{Ca} \right)_{sample} \right]}{\left[\left(238_U / 43_{Ca} \right)_{NIST} \right]} \right) / \left(\frac{[CaO_{NIST}]}{[CaO_{Sample}]} \right) \right\} \times\ 238_{U_{NIST}}$$

9

Where ^{238}U and ^{43}Ca are is the count per second (CPS) obtained using an LA-ICP-MS in the sample (mineral) and NIST (standard glass). The CaO is the composition wt% of calcium in the sample (mineral)

and NIST (standard glass).

2.1. Absolute age calibration based on U-doped glass calibrated against uranium thin films

Absolute age calibration based on uranium doped glass calibrated against uranium thin film allows determining the fission-track age without the need for an age standard. This method was developed to be used as a neutron dosimeter based on the same nuclear reaction occurring in the mineral, *i.e.*, U(n,f). Ideally, thin films with well-known uranium concentration must be used once the thickness of the film is just a few micrometers in size, and it cannot stop the fission fragments. Consequently, nearly uranium-free muscovite attached to the thin film can register 1.00 ± 0.025 of fission fragments coming from induced tracks of ^{235}U. This efficiency factor was measured by comparison with nuclear emulsion (Hadler, et al., 1996). However, considering the difference between the uranium content in the uranium thin film and in geological samples, besides the common neutron fluence used ~ 10^{14} – 10^{15} cm^{-2}, the direct application of uranium thin film for dating samples is unfeasible. To overcome this, standard glasses were calibrated against the thin film in a beam-hole nuclear reactor position, where the thermal neutron flux is significantly lower, allowing the control of the number of tracks observed on muscovite external detectors attached to both thin films and glasses (Iunes et al., 2002; Soares et al., 2014a). Once the glasses are calibrated, they can be used to determine the thermal neutron fluence received by the samples to be dated.

The data presented in this work uses the re-calibration of Soares et al., (2014a), where the uranium concentration in thin films were re-measured by Curvo et al., (2013) using alpha spectrometry calibration. After re-calibration, uranium-doped glass was tested using the age standards Durango, Limberg, and FCT (Soares et al., 2014a), and applied to alkaline rocks from Alto Paranaíba and Ponta Grossa Arches (Soares et al. 2016). The fission-track age equation, as presented by Iunes et al. (2002) and after Soares et al. (2014a):

$$t = \frac{1}{\lambda} ln \left[1 + g \left(\frac{\lambda_{\alpha}}{\lambda_{f}} \right) \left(\frac{\rho_{S}}{\rho_{i}} \right) \left(\frac{R_U}{C_{238}} \right) \right]$$

10

.

Where *g* is the geometry factor; ρ_S and ρ_i are the spontaneous fission-track density in an internal surface and induced fission-track density in the muscovite external detector, respectively; C_{238} is the isotopic abundance of ^{238}U in natural uranium; R_U is the number of reactions per target nucleus, which for U-doped glasses calibrated against U-thin films can be written as follows (Iunes et al., 2002; Soares et al., 2014a):

$$R_U = \frac{\rho_D}{N_U^V \epsilon_V}$$

11

,

where: ρ_D is the induced fission-track density in the muscovites attached to U-doped glasses, $N_U{}^V$ is the number of uranium atoms per unit volume in the glass dosimeter; ε_V is the detection efficiency of the muscovite external detector attached to it (Bigazzi et al., 1995; Hadler et al., 1996; Iunes et al., 2004, 2005).

The uncertainty for Eqs. (10) and (11) given by Eqs. (12) and (13), respectively.

$$\sigma_t = t \times \sqrt{\left(\frac{1}{N_s}\right) + \left(\frac{1}{N_i}\right) + \left(\frac{\sigma_g}{g}\right)^2 + \left(\frac{\sigma_{\lambda_f}}{\lambda_f}\right)^2 + \left(\frac{\sigma_{R_U}}{R_U}\right)^2}$$

12

$$\sigma_{R_U} = R_U \times \sqrt{\left(\frac{1}{N_D}\right) + \left(\frac{\sigma_{N_U^V \epsilon_V}}{N_U^V \epsilon_V}\right)^2}$$

13

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where N_S, N_i, and N_D are the number of fission tracks counted in mineral, external detector attached to mineral surface, and muscovite attached to uranium doped standard glass dosimeter, respectively.

3. Experimental procedure

Two sets of samples were dated using the LA-ICP-MS absolute $\boldsymbol{\psi}$-method and one set was dated using both $\boldsymbol{\psi}$-method and ζ-calibration based on LA-ICP-MS. The first set is a series of well-known age standards with a low annealing degree, indicated by minor shortening of their fission-track lengths (around 10%). A total of 4 samples were dated using Eq. 4. Durango (Dur; 4 different crystals), Fish Canyon Tuff (FCT; 4 aliquoted), Limberg (Lim; 3 aliquoted) and Mud Tank (MT; 1 crystal). The second group consists of a total of 7 apatite samples, 4 samples from Lindau and Obernsees – Germany, and 3 from the Northeast (NE) Brazil (Borborema Complex). In contrast with standard samples, these 7 samples were chosen because they suffered a moderate annealing degree (> 10%). The grain-by-grain and pooled ages are presented.

All apatite samples were mounted in epoxy resin and polished with 3.0 µm and 0.3 µm oxide powder to expose the internal surfaces. Apatites were then etched to reveal the spontaneous fission tracks using

5.5 N HNO_3, at 21 ^{o}C for 20 s (Donelick et al., 2005). The etching was carried out using an ultra-thermostatic bath, which the temperature variation was ~ 0.1ºC.

Horizontal confined fission-track lengths were measured, and spontaneous fission-track densities were counted using an automated Leica DM6M optical microscope with a nominal magnification of 1000×. During microscope analyses, pictures were taken of each analyzed grain, and a square was drawn on the image defining the area where the spontaneous fission-track density was determined. This makes it possible to monitor whether the spots analyzed by LA-ICP-MS cover the same area as the spontaneous track counts.

The LA-ICP-MS analyses were carried out using a New Wave UP213 laser ablation system coupled to an Agilent 7800ce quadrupole ICP-MS at the ChronusCamp Research – Isotope Geology Laboratory. Itapira-SP, Brazil.

The absolute fission-track ages based on LA-ICP-MS rely on an important parameter which is independently determined absolute ^{238}U concentration. Soares et al. (2014b, 2015) introduced a monocrystal Mud Tank sample (MT-7 6.88 ppm uranium) as candidate for matrix-matched reference material. This sample, along with the standard glass NIST610, was used to standardize two batches of Durango samples (DR – Chew et al, 2016).

For quality control tests for uranium and thorium content were conducted using a specific Durango sample (DR – Chew et al., 2016). The tests were carried out analyzing in two different sessions, both normalized using with MT-7 and NIST SRM 610. The results were compared with each other, and with the values from Chew et al. (2016). The uranium concentrations for all dated samples in this work using LA-ICP-MS were normalized using Mud Tank (MT-7) (Soares et al., 2014b; 2015).

To compare both absolute Ø-method with thermal neutron and ψ-method with LA-ICP-MS, one aliquot of Durango apatite was irradiated at Research Reactor TRIGA Mainz – Germany, with thermal neutron in a carousel position (K). The IRMM540 standard glass calibrated against uranium thin film (Soares et al., 2014a) was used for measuring the neutron fluence. The EDM was used for dating grain-by-grain ages (shards). Then the ^{238}U concentrations were measured using LA-ICP-MS. The spot positions in the Durango were chosen as the equivalent position where the induced fission tracks were counted in the muscovite external detector. Thus, it was possible to compare grain-by-grain, as well as pooled ages for both Ø- and ψ-methods.

For ζ-calibration and ψ-methods, the spot size of 40μm was chosen to scan the maximum area where spontaneous fission-track densities were determined. To avoid chemical zoning, the uranium concentration was measured considering the flat signal normalization of uranium and ^{43}Ca as internal standard (*i.e.*, ^{238}U/^{43}Ca). For laser ablation, the pulse repetition rate was 10 Hz. The energy density was about 5-5.5 J.cm^{-2}. A single-spot analysis consists of 20 s background, 40 s ablation, and 20 s washout.

Before and after about 10 spots on the unknown samples, 2 spots on matrix-matched reference materials (MT-7) were analyzed for calibration.

4. Results

4.1. Absolute LA-ICP-MS uranium and thorium determinations

For the ^{238}U concentration, the Durango batch #1 shows a value of 12.0 ± 1.3 ppm when normalized using MT-7, and 12.0 ± 1 .6 ppm when normalized using the NIST SRM 610. Similarly, Durango batch #2 shows a value of 11.9 ± 0.86 ppm when normalized using MT-7, and 12.0 ± 0.86 ppm when normalized using the NIST SRM 610. Considering both batches, the ^{238}U concentrations are 12.0 ± 1.1 ppm when normalized using MT-7, and 12.0 ± 1.0 ppm when normalized using the NIST SRM 610 (Fig. 1 and supplementary material – S1).

For the ^{232}Th concentration, the Durango batch #1 shows a value of 224.3 ± 24.2 ppm when normalized using MT-7 (Th = 34.5 ± 1.8ppm – an average of 4 pieces analyzed by isotope dilution), and 217.9 ± 23.5 ppm when normalized using NIST SRM 610. Similarly, the Durango batch #2 shows a value of 225.2 ± 15.3 ppm when normalized using MT-7, and 216.5 ± 14.7 ppm when normalized using NIST SRM 610. Considering both batches, the ^{232}Th concentrations are 224.7 ± 17.8ppm when normalized using MT-7, and 217.2 ± 1 7.2ppm when normalized using NIST SRM 610 (Fig. 2 and supplementary material - S-1). The Th/U ratio for both batches is 18.8 ± 0.8 when normalized using MT-7, and 18.1 ± 0.8 when normalized using NIST SRM 610 (Fig. 2 and supplementary material - S-1).

4.2. Absolute U-thin film and absolute LA-ICP-MS

We present absolute grain-by-grain ages and pooled ages in a Durango crystal for both LA-ICP-MS and neutron irradiation EDM (Fig. 3a). For the EDM method, a total of 9992 fission tracks were counted in a muscovite attached to the IRMM-540. The $N_U{}^V\varepsilon_V = 0.52\times10^{14}cm^{-2}$± (3.8%) (Soares et al., 2014a); ρ_D = 6.17×10^5 cm^{-2} (± 1.0%) and the $R_U = 1.19\times10^{-8}$ (± 4.0%). The geometry factor *g* used was the one determined by the same observer using prismatic section of Durango apatite (*g* = 0.60 ± 0.02) (Soares et al., 2013). The pooled age for 39 dated grains was 30.2 ± 4.4 Ma (2σ) (Table 1). For absolute LA-ICP-MS the $\psi_{ICP} = (1.78 \pm 0.064)\times10^{-12}$, and the pooled age was 29.0 ± 3.6 Ma(2σ) (Table 1). Figure 3b presents the distribution of individual LA-Ages/U-thin film ages, LA-Ages/Reference ages and U-thin film ages/Reference ages ratio.

Table 1
Absolute uranium ϕ-thin Film) and Absolute ψ-LA-ICP-MS

NS	NI	ρS/ρi	U(μg.g^{-1})	Age (Ma)ϕ-thin Film	ρS (cm^2)	Age (Ma)ψ-LA-ICP-S
21	77	0.273	12.109	23.0 ± 5.8	2.1E + 05	25.5 ± 5.7
32	68	0.471	12.439	39.7 ± 8.7	3.2E + 05	37.8 ± 7.0
25	68	0.368	11.499	31.0 ± 7.4	2.5E + 05	32.0 ± 6.6
28	62	0.452	12.524	38.1 ± 8.9	2.8E + 05	32.9 ± 6.5
26	72	0.361	11.857	30.5 ± 7.1	2.6E + 05	32.2 ± 6.6
25	68	0.368	11.507	31.0 ± 7.4	2.5E + 05	32.0 ± 6.6
31	62	0.500	11.838	42.2 ± 9.5	3.1E + 05	38.5 ± 7.2
25	63	0.397	11.797	33.5 ± 8.1	2.5E + 05	31.2 ± 6.5
34	64	0.531	11.992	44.8 ± 9.7	3.4E + 05	41.7 ± 7.5
29	66	0.439	13.510	37.1 ± 8.5	2.9E + 05	31.6 ± 6.1
20	71	0.282	11.641	23.8 ± 6.1	2.0E + 05	25.3 ± 5.8
23	68	0.338	13.411	28.6 ± 7.0	2.3E + 05	25.2 ± 5.4
22	71	0.310	11.952	26.2 ± 6.5	2.2E + 05	27.1 ± 6.0
26	61	0.426	12.394	36.0 ± 8.6	2.6E + 05	30.9 ± 6.3
29	70	0.414	12.012	35.0 ± 7.9	2.9E + 05	35.5 ± 6.9
23	65	0.354	12.938	29.9 ± 7.4	2.3E + 05	26.2 ± 5.6
23	52	0.442	13.401	37.3 ± 9.5	2.3E + 05	25.3 ± 5.4
20	70	0.286	11.958	24.1 ± 6.2	2.0E + 05	24.6 ± 5.7
22	70	0.314	12.866	26.5 ± 6.6	2.2E + 05	25.2 ± 5.5
21	69	0.304	12.183	25.7 ± 6.5	2.1E + 05	25.4 ± 5.7
24	81	0.296	12.611	25.0 ± 5.9	2.4E + 05	28.0 ± 5.9
22	68	0.324	12.122	27.3 ± 6.8	2.2E + 05	26.7 ± 5.9
21	79	0.266	11.928	22.5 ± 5.6	2.1E + 05	25.9 ± 5.8
23	77	0.299	11.846	25.2 ± 6.1	2.3E + 05	28.6 ± 6.2

The ψ-LA-ICP-MS is 1.78x10^{-12}±2.7%; The area to determine the ρ_S value is 1×10^{-4}cm2. Fission-track age errors for individual grain-by grain ages are in 1σ. Pooled age ages are in 2σ

NS	NI	ρS/ρi	U(μg.g^{-1})	Age (Ma)ϕ-thin Film	ρS (cm^2)	Age (Ma)ψ-LA-ICP-S
22	90	0.244	11.957	20.7 ± 5.0	2.2E + 05	27.1 ± 6.0
26	60	0.433	12.265	36.6 ± 8.8	2.6E + 05	31.2 ± 6.3
22	73	0.301	12.482	25.5 ± 6.3	2.2E + 05	26.0 ± 5.7
31	75	0.413	13.575	34.9 ± 7.6	3.1E + 05	33.6 ± 6.3
19	60	0.317	12.356	26.7 ± 7.2	1.9E + 05	22.6 ± 5.3
28	56	0.500	12.565	42.2 ± 10.0	2.8E + 05	32.8 ± 6.4
25	67	0.373	13.659	31.5 ± 7.5	2.5E + 05	27.0 ± 5.6
22	64	0.344	13.429	29.0 ± 7.3	2.2E + 05	24.1 ± 5.3
20	64	0.313	12.210	26.4 ± 6.9	2.0E + 05	24.1 ± 5.5
25	73	0.342	12.376	28.9 ± 6.8	2.5E + 05	30.3 ± 6.2
25	69	0.362	12.599	30.6 ± 7.3	2.5E + 05	29.2 ± 6.1
22	68	0.324	12.180	27.3 ± 6.8	2.2E + 05	26.6 ± 5.8
23	66	0.348	12.924	29.4 ± 7.3	2.3E + 05	26.2 ± 5.6
23	78	0.295	13.238	24.9 ± 6.0	2.3E + 05	25.6 ± 5.5
24	70	0.343	12.298	29.0 ± 7.0	2.4E + 05	28.7 ± 6.1
29	66	0.439	13.270	37.1 ± 8.5	2.9E + 05	32.1 ± 6.2
Pooled Age				**30.2 ± 4.4**		**29.0 ± 1.8**

The ψ-LA-ICP-MS is 1.78x10^{-12}±2.7%; The area to determine the ρ_S value is 1×10^{-4}cm2. Fission-track age errors for individual grain-by grain ages are in 1σ. Pooled age ages are in 2σ

4.3. Standard samples

As described above, we applied the absolute LA-ICP-MS method to four samples: 4 aliquots of Durango (different crystals - shards), 4 aliquots of FCT, 3 aliquots of Limberg and 1 crystal (shards) of Mud Tank. Individual pooled ages for all aliquots are in Table 2. The grain-by-grain ages can be seen in supplementary material (S2). When all aliquots are put together, about 131 grains were analyzed for Durango, yielding the final pooled age of 32.6 ± 4.4 Ma (2σ) with an average of U = 8.62 ppm. For FCT about 124 grains were analyzed giving the final pooled age of 26.1 ± 3.4 Ma with an average of U = 14.53 ppm. For Limberg about 82 grains were analyzed giving the final pooled age of 17.8 ± 2.6 Ma with an average of U = 14.66. Finally, the Mud Tank sample, presents the age of 251.3 ± 39 Ma with average of U = 2.41ppm. All details can be seen in Table 3 and Fig. 4.

Table 2
Pooled age of different aliquots for Durango (Dur), Fish Canyon Tuff (FTC), Limberg (Lim) and Mud Tank (MT).

Sample	N grains	N_S	$\rho_S(\times10^5)\pm1\sigma$	U(ppm)	ψ_{ICP}-method (Ma)
Dur-2	33	158	1.92 ± 0.15	9.08	31.0 ± 6.0
Dur-3	33	167	2.02 ± 0.16	8.97	33.2 ± 6.3
Dur-4	32	153	1.91 ± 0.16	8.32	33.8 ± 6.6
Dur-5	33	148	1.79 ± 0.15	8.09	32.6 ± 6.4
FCT-1	32	196	2.45 ± 0.18	13.11	26.8 ± 4.8
FCT-2	32	203	2.54 ± 0.18	13.17	27.7 ± 4.9
FCT-3	31	177	2.28 ± 0.17	12.86	25.5 ± 4.7
FCT-5	29	187	2.58 ± 0.19	15.23	24.3 ± 4.4
Lim-1	28	119	1.70 ± 0.16	12.96	18.0 ± 3.8
Lim-2	28	116	1.66 ± 0.15	13.32	17.1 ± 3.7
Lim-3	26	154	2.34 ± 0.19	17.94	18.2 ± 3.5
MT	32	322	4.03 ± 0.22	2.41	251.3 ± 39.0
Fission-track ages errors in the table are in 2σ. ψ-parameter is 1.78x10-12 ± 5%					

Table 3
Pooled age considering all aliquots for Durango (Dur), Fish Canyon Tuff (FTC), Limberg (Lim) and Mud Tank (MT).

Sample	N grains	N_S	$\rho_S(\times10^5)\pm1\sigma$	L (μm)	U(ppm)	ψ_{ICP}-method (Ma)
Dur	131	626	1.91 ± 0.08	14.18 ± 0.18	8.62	32.6 ± 4.4
FCT	124	763	2.46 ± 0.09	14.53 ± 0.15	13.56	26.1 ± 3.4
Lim	82	389	1.90 ± 0.10	15.17 ± 0.09*	14.66	17.8 ± 2.6
MT	32	322	4.03 ± 0.22	13.69 ± 0.22**	2.41	251.3 ± 39.0
Fission-track ages errors in the table are in 2σ. ψ-parameter is 1.78x10-12 ± 5%. *Kraml et al., 2006; **Green et al., 2006.						

4.4. Moderately annealed samples

The ages of 4 aliquots of samples from Lindau and Obernsees, and 3 aliquots from Borborema Province, NE Brazil, were dated with absolute LA-ICP-MS and ζ_{ICP} (Donelick et al., 2005) (Table 4, Fig. 5). The samples experienced moderate annealing, resulting in reduced confined fission-track lengths (Table 4).

The mean track lengths for Lindau aliquots are L1 (12.28 μm) and L121 (12.10 μm). For the Obernsees aliquots are HM-3 (12.07 μm) and HM-14 (11.59 μm). The ages obtained (U-Pb in apatite and zircon) for Lindau and Obernsees are between 300–340Ma (Caracciolo et al., 2021).

Table 4
Pooled age of different aliquots for Obernsees, Lindau, and Borborema Province

Obernsees							
Sample	**N grains**	**N_S**	**ρ_S ($\times10^6$)**	**U(ppm)**	**L(μm)**	**ζ-method(Ma)**	**ψ-method(Ma)**
HM-14	40	1176	3.46 ± 0.10	73.5	11.59 ± 0.18	86.5 ± 8.3	85.2 ± 10.1
HM-03	32	902	3.13 ± 0.10	86.3	12.07 ± 0.21	65.8 ± 6.6	61.6 ± 7.6
Lindau							
L-1	39	1093	3.55 ± 0.11	71.3	12.28 ± 0.20	89.7 ± 8.7	83.8 ± 10.0
L121	38	1170	3.55 ± 0.10	54.0	12.10 ± 0.22	118.5 ± 11.3	112.3 ± 13.3
Borborema Province							
NE-14A	39	1567	2.54 ± 0.06	32.7	12.40 ± 0.21	138.2 ± 12.6	128.1 ± 14.7
NE15A	40	946	1.48 ± 0.05	21.2	12.09 ± 0.20	124.8 ± 12.4	119.3 ± 14.3
LLM-55	39	1087	1.74 ± 0.05	26.9	12.09 ± 0.21	116.3 ± 11.3	111.1 ± 13.3

Fission-track ages errors in the table are in 2σ. For Obernsees and Liundau, $\zeta_{ICP} = 1.822\times10^3\pm3.2\%$. U = 9.61 ± 2.2%μg.$g^{-1}$, $\rho_S = 1.66\times10^5\pm2.3\%$ (cm^2). For Borborema complex, $\zeta_{ICP} = 1.810\times10^3\pm3.2\%$. U = 9.51 ± 2.3%μg.$g^{-1}$, $\rho_S = 1.66\times10^5\pm2.3\%$ (cm^2).

For the Borborema Province, NE Brazil, the mean track lengths are NE-14A (12.40 μm) andNE-15A (12.09 μm), both samples are from an orthogneiss and migmatite of the Ediacaran Vila Moderna Suite (590 ± 5Ma; Santos, 2012). Finally, the sample LLM-55 (12.09 μm) was obtained from a metagranite of the Ediacaran Chorrochó Suite. Grain by gran ages can be found in supplementary material (S-3).

5. Discussion

5.1. Absolute LA-ICP-MS uranium and thorium determinations.

A Durango sample (DR – Chew et al, 2016) was used to test the ^{238}U measurements with the LA-ICP-MS normalized using MT-7 as matrix-matched reference material and NIST SRM 610. The ^{238}U results of Batch #1 and Batch #2 (Fig. 1) agree with each other (2σ), based on both MT-7 and NIST SRM 610. The values also agree with Chew et al., 2016 when LA-ICP-MS was applied (2σ). The maximum values for uranium concentration are 16.28 ppm and 16.33 ppm for MT-7 and NIST SRM 610, respectively and minimum value for uranium concentration are 9.87 ppm and 9.89 ppm for MT-7 and NIST SRM 610, respectively (Fig. 1 and supplementary material - S1). For ^{232}Th the results of Batch #1 and Batch #2 (Fig. 1) agree with each other (2σ), based on both MT-7 and NIST SRM 610. The values also agree with Chew et al., 2016 when LA-ICP-MS was applied (2σ). The maximum values for thorium concentration are 292.2 ppm and 283.8 ppm for MT-7 and NIST SRM 610, respectively and minimum value for uranium concentration are 189.2 ppm and 183.8 ppm for MT-7 and NIST SRM 610, respectively (Fig. 1 and supplementary material - S1).

Finally, the Th/U ratio for both batches is 18.8 ± 0.79 when normalized using MT-7, and 18.1 ± 0.76 when normalized using NIST610 (Fig. 2 and supplementary material – S-1), with a maximum value of 20.92 and 20.40 for MT-7 and NIST610, respectively, and with a minimum value of 16.35 and 156.84 for MT-7 and NIST610, respectively. The Th/U ratio reported for this sample is 18.67 ± 0.19 using SQ-ICP-MS (Chew et al., 2016).

5.2. Absolute LA-ICP-MS and Absolute U-thin film age calibration.

One piece of Durango apatite was used to determine individual grain-by-grain and pooled ages using absolute thin film calibration for EDM and LA-ICP-MS. Comparing EDM (30.2 ± 4.4 Ma (2σ)) with LA-ICP-MS (29.0 ± 3.6 Ma(2σ)) pooled age the difference is around 4%, an acceptable difference considering the common error for the fission-track method (Fig. 3a and Table 1). Both ages agree with the reference age (31.44 ± 0.18 Ma, $^{40}Ar/^{39}Ar$ dating, McDowell et al. 2005), indicating the reliability of the experimental procedures applied. Despite the agreement, it is important to point out that both methods yield systematically ages below the reference value. This can be a characteristic of the chosen crystal once the ψ-method was also applied in other four different monocrystals and the ages oscillate between reference values, as can be seen below.

Figure 3b presents the distribution of LA-ages/Reference ages ratio. The average variation, i.e., when all grains are taken into account, the LA-ages/Reference age is 0.92 (solid line - Fig. 3b), and U thin film ages/Reference and LA-age/EDM-age, the average variation are 0.96 (dashed line - Fig. 3b).

5.3. LA-ICP-MS fission-track age discussion

5.3.1 Age standard samples

Individual pooled ages for all aliquots are in Table 2. The ages for Durango oscillate between 1.4 and 7.5% (31.0 ± 6.0 Ma and 33.8 ± 6.6Ma), an acceptable variation considering the errors associated to the

fission-track method. Table 3 shows the results of all aliquots (131 grains) yielding the pooled age of 32.6 ± 4.4 Ma (2σ), a 3.7% difference to the reference value. This result overlaps the reference age in 1σ (31.44 ± 0.18 Ma, $^{40}Ar/^{39}Ar$ dating, McDowell et al. 2005).

The FCT ages oscillate between 7.4% and less than 1% (25.5 ± 4.7 Ma and 27.7 ± 4.9 Ma). About 124 grains were analyzed yielding the pooled age of 26.1 ± 3.4 Ma, a 5.3% difference to the published value. This result agrees in 1σ with published data (27.55 ± 0.24 Ma determined by K–^{39}Ar dating of biotite, *e.g.*, Lanphere and Baadsgaards, 1997).

For Limberg, the oscillation of ages was between 1.8% and 8.3% (17.1 ± 3.7 Ma and 18.2 ± 3.5 Ma). About 82 grains were analyzed yielding the pooled age of 17.8 ± 2.6 Ma, a 6.0% difference to the published value, also overlapping the published ages in 1σ (16.8 ± 1.0 Ma for apatite and 16.5 ± 1.0 Ma for titanite, both determined by (U–Th)/He (e.g., Kraml et al., 2007).

Finally, the Mud Tank sample, only one aliquot was determined. The sample presents the age of 251.3 ± 39 Ma, in agreement with the average of 249.8 ± 27.8 Ma (2σ) calculated based on (U–Th)/He published in Green et al. (2006). To confirm the fission-track age of MT sample, the ζ_{ICP} was also applied in the sample crystal used to determine ψ-method. The obtained age was 248.9 ± 31.0 Ma (2σ), For this age calculation, the ζ_{ICP} value was1.52×10^{3}.

5.3.2 Moderate annealing samples

Samples with moderate annealing degrees were determined using both ζ_{ICP}-calibration (using Durango apatite as age standard) and absolute ψ-method. The same ^{238}U concentration and fission-track density were used for both ζ_{ICP} and ψ-method, ensuring that individual grain-by-grain ages will have consistent dispersion between methods. Table 4 lists the pooled ages. For samples HM-14 (Obernsees), their pooled ages for ψ-method (85.2 ± 10.1 Ma) and ζ_{ICP} (86.5 ± 8.3 Ma) overlapping in 1σ. A similar result was found for HM-3: ψ-method (61.6 ± 7.6 Ma) and ζ_{ICP} (65.8 ± 6.6 Ma). For Lindau, the pooled age for L-1 is ψ-method (83.8 ± 10.0 Ma) and ζ_{ICP} (89.7 ± 8.7 Ma) and for L-121 the pooled age for L-1 is ψ-method (112.3 ± 11.3 Ma) and ζ_{ICP} (118.5 ± 13.3 Ma). Both ψ-method and ζ_{ICP} pooled are also in agreement in 1σ, despite such difference is slightly larger than those presented in Obernsees samples. The smaller difference is in the sample HM-14 between ζ_{ICP} and absolute ψ-method (1.0%), and an average of 6.1% difference for HM-3, L-1 and L-121.

The samples from the Borborema Province exhibit similar dispersion when comparing the methods. The ψ-method (128.1 ± 14.7 Ma) and ζ_{ICP} (138.2 ± 12.6 Ma) for NE-14A, ψ-method (119.3 ± 14.3 Ma) and ζ_{ICP} (124.8 ± 12.4 Ma) for NE15A and ψ-method (111.1 ± 13.3 Ma) and ζ_{ICP} (116.3 ± 11.3 Ma) for LLM55. The dispersion of 7.3% for NE14A, 4.4% for NE15A and 4.5% for LLM55. All these dispersions are consistent with the ones presented by Obernsees and Lindau (Table 4). Neoproterozoic granites dated with EDM (conventional ζ-calibration) method published by Galindo and Oliveira (2006) in the same

region yielded apatite fission-track ages of ca. 124 to 138 Ma and mean confined fission-track lengths of 11.77 to 12.80 µm.

5.4. Advantages and disadvantages of the calibration techniques

As is clear from the comparisons above, the calibration techniques yield internally consistent results. The general agreement between the ages determined in this work and the reference ages is always within 2σ. The additional test of dating moderately annealed samples also resulted in internally consistent ages.

The $\boldsymbol{\psi}$-method calibration does not depend on age standard but there are some issues: 1) It is assumed that the physical constants are well-determined. There was once a debate on the value of the ^{238}U spontaneous fission decay constant (λf). By the 2000s, literature values were split between 7×10^{-17} a^{-1} and 8.5×10^{-17} a^{-1}, with most of the fission-track determinations clustering around the first value. In the last 20 years, however, evidence for the latter value has been accumulated and presently there is no doubt that this is the most accurate value (Holden and Hoffman, 2000) and 2) for the $\boldsymbol{\psi}$-method the calibration of the efficiency factors is laborious and involves additional track measurement.

6. Conclusion

A series of experiments to obtain the absolute fission-track ages based on LA-ICP-MS were carried out. For $\boldsymbol{\psi}$-method was suggested to correct the track length replacing the etched track range is R_s= (L_{e-spt})×0.5 to R_s= (L_{e-spt})×(L_e/L_f) = (L_{e-spt})×0.766. This change shows to be quite acceptable for both samples with low annealing (~ 10%) and samples with moderate annealing (> 10%). Also, absolute Ø-method and absolute $\boldsymbol{\psi}$-method was applied in one aliquot of Durango. The results are consistent with reference ages. This implies that absolute age determination can be used as an alternative method which depends on the age standards.

The determination of uranium concentration using MT-7 and NIST SRM 610 yields a consistent internal agreement and overlaps the published data. This result is important to show that there is no matrix effect for uranium determination when NIST or MT-7 (an apatite) are used. This result is particularly important because for absolute ages this parameter must be the correct number and not be relative as ζ-calibration allows. Also, thorium determination was presented here and presents a consistent result.

Declarations

Conflict of interest:

The authors declare no competing interest

Ethic declaration

Not applicable

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Author Contribution

Dr. Cleber Jose Soares: Experimental procedure and Writing the manuscriptDr. Julio Hadler: Writing and revising the manuscriptDr. Sandro Guedes: Writing and revising the manuscriptDr. Luca Caracciolo: Collection and separation of samples and writing and revision of the manuscript.Ms. Leonardo Lopes: Collection and separation of samples and writing and revision of the manuscript.

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Figures

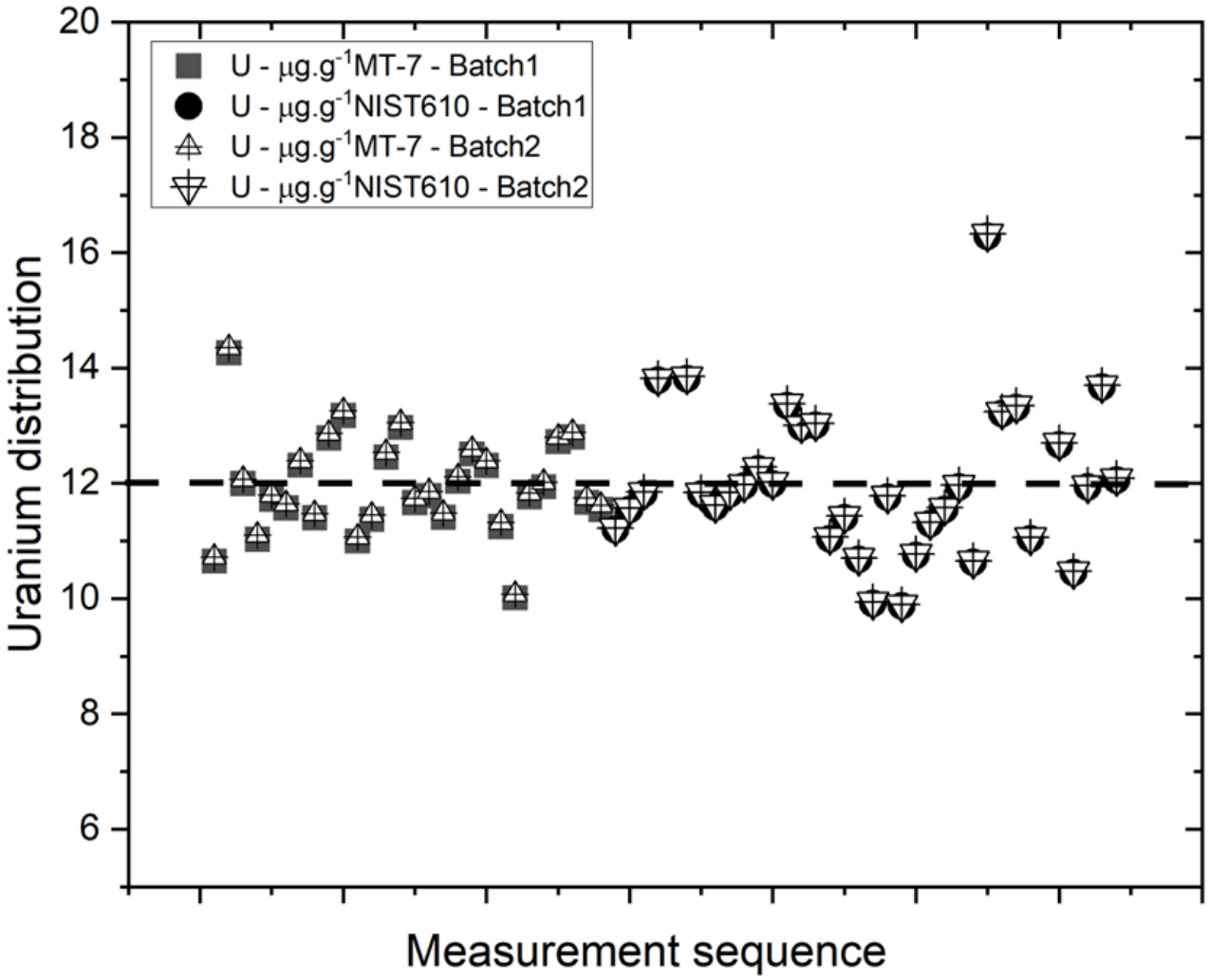


Figure 1

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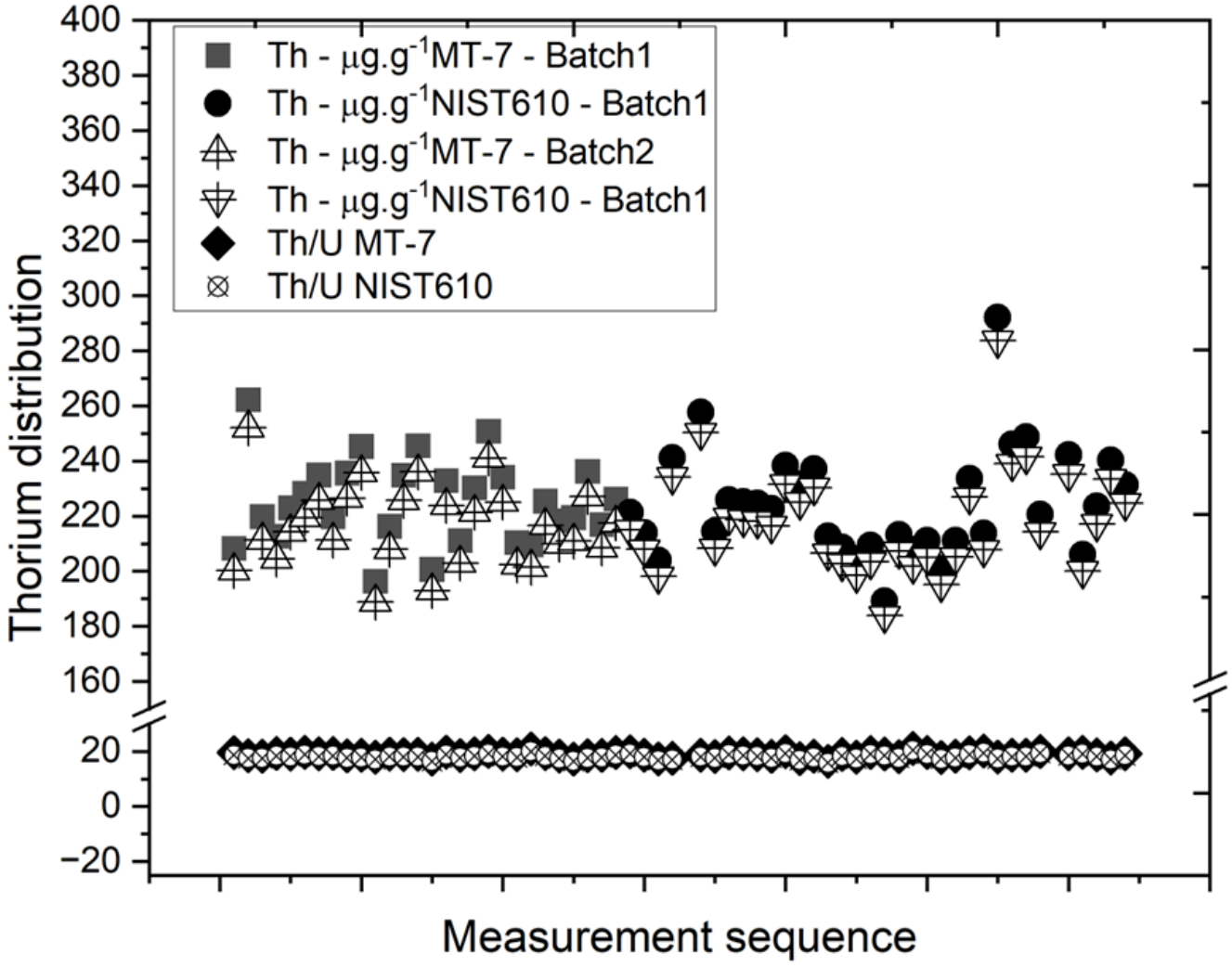


Figure 2

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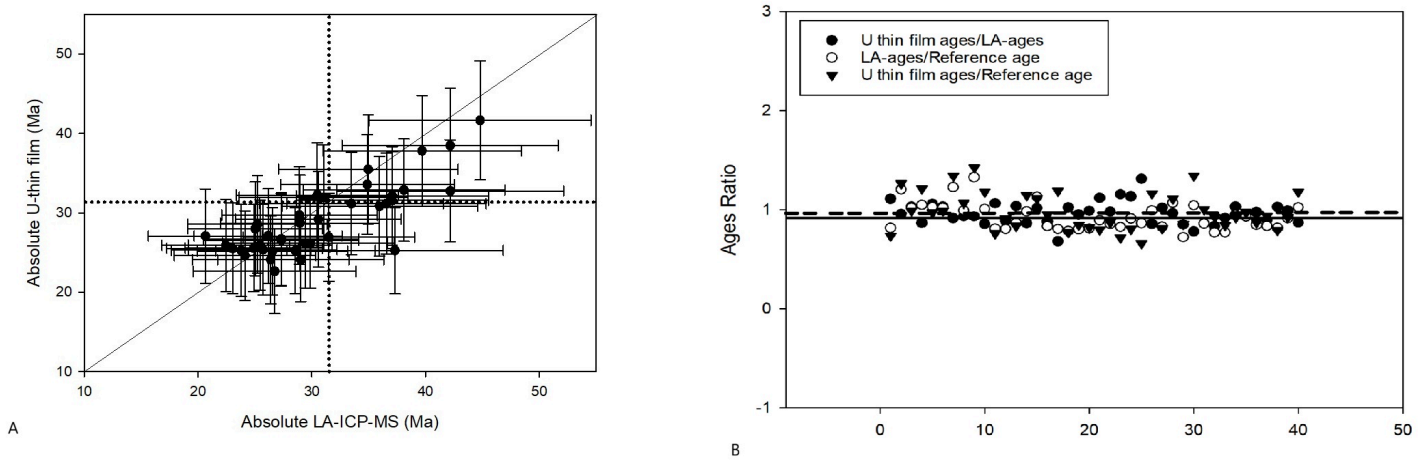


Figure 3

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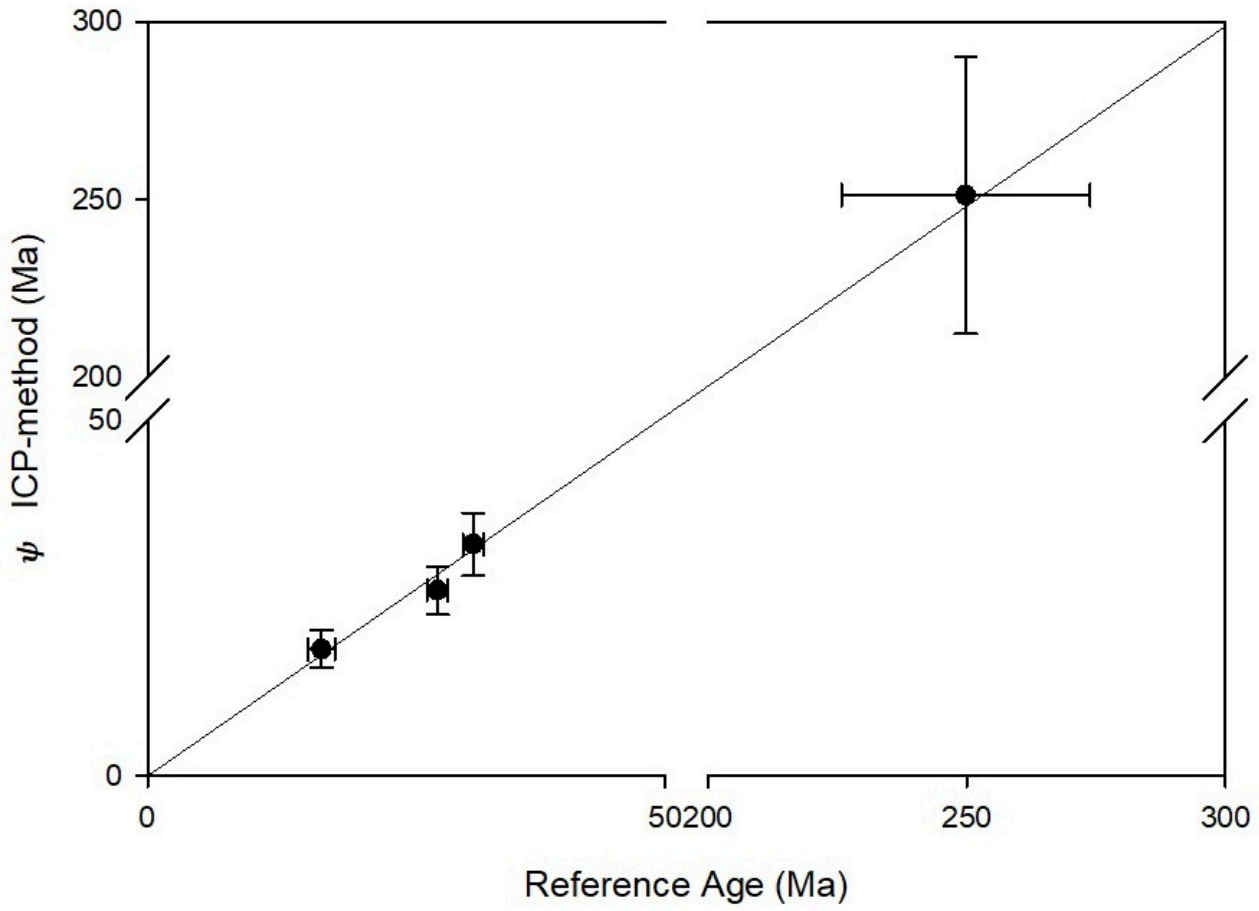


Figure 4

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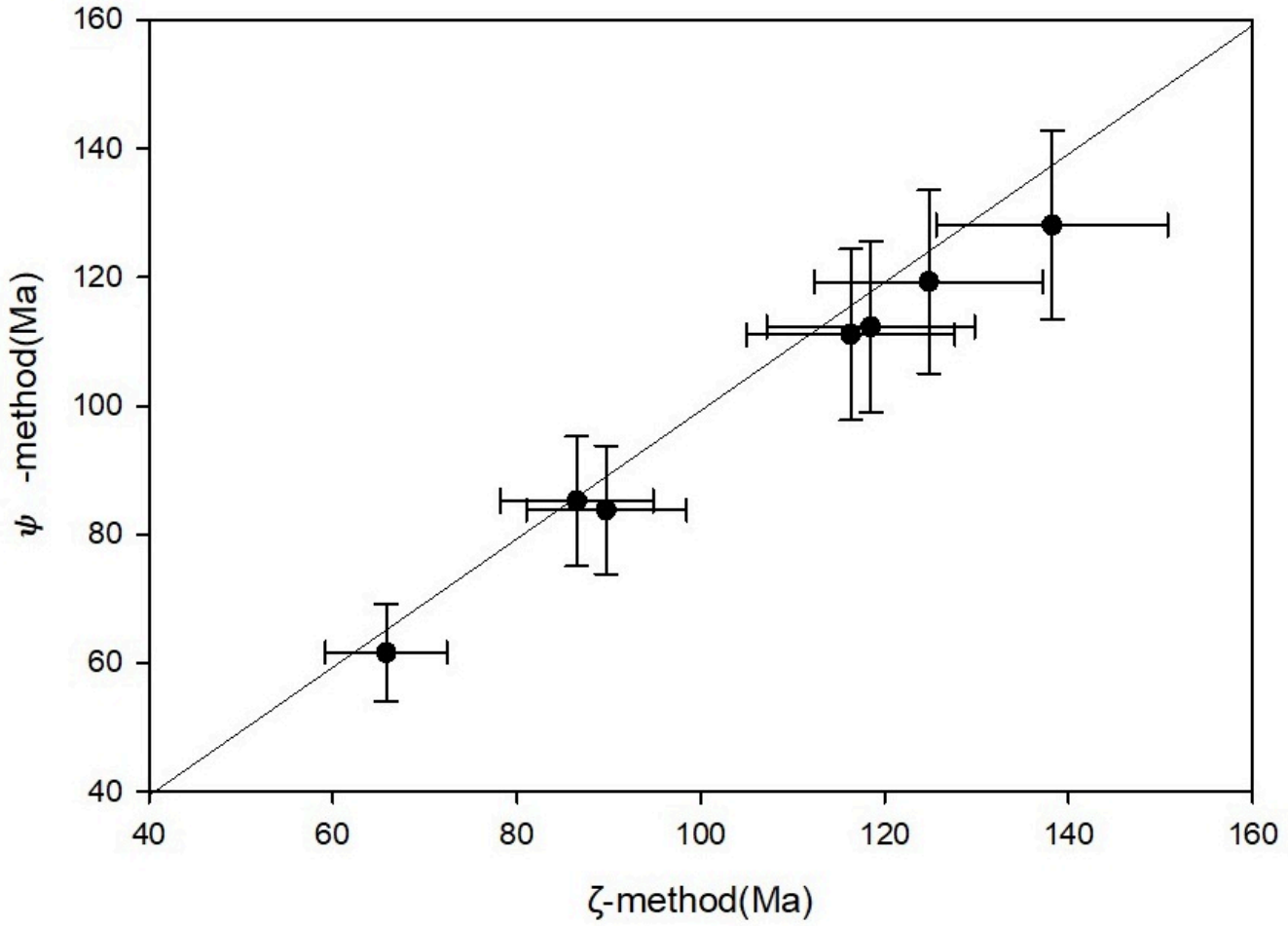


Figure 5

Legend not included with this version.

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- S1UandThconcentrationsupmaterial2026.xlsx
- S2Agestandardssupmaterial2026.xlsx
- S3Moderatesupmaterial2026.xlsx