DOI: 10.1007/s00269-013-0624-2

Phys Chem Minerals (2014) 41:65–73
DOI 10.1007/s00269-013-0624-2

ORIGINAL PAPER

Novel calibration for LA-ICP-MS-based fission-track thermochronology

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Received: 28 May 2013 / Accepted: 5 September 2013 / Published online: 14 September 2013

Abstract We present a novel age-equation calibration for fission-track age determinations by laser ablation inductively coupled plasma mass spectrometry. This new calibration incorporates the efficiency factor of an internal surface, $[\eta q]_{is}$, which is obtained by measuring the projected fission-track length, allowing the determination of FT ages directly using the recommended spontaneous fission decay constant. Also, the uranium concentrations in apatite samples are determined using a Durango (Dur-2, 7.44 µg/g U) crystal and a Mud Tank (MT-7, 6.88 µg/g U) crystal as uranium reference materials. The use of matrix-matched reference materials allows a reduction in the uncertainty of the uranium measurements to those related to counting statistics, which are ca. 1 % taking into account that no extra source of uncertainty has to be considered. The equations as well as the matrix-matched reference materials are evaluated using well-dated samples from Durango, Fish Canyon Tuff, and Limberg as unknown samples. The results compare well with their respective published ages determined through other dating methods. Additionally, the results agree with traditional fission-track ages using both the zeta approach and the absolute approach, suggesting that the calibration presented in this work can be robustly applied in geological context. Furthermore, considering that fission-track ages can be determined without an age standard sample, the fission-track thermochronology approach presented here is assumed to be a valuable dating tool.

Keywords Fission-track thermochronology · LA-ICP-MS · Uranium reference material · Projected fission-track length

Introduction

Fission-track thermochronology (FTT) is a powerful tool used to solve a multitude of geological and geomorphological questions related to provenance studies, exhumation processes, denudation and erosion rates and orogenic belt genesis (e.g., Carter and Gallagher 2004; Donelick et al. 2005). In traditional FTT, samples are irradiated in a nuclear reactor in order to access induced ^{235}U fissions, which are recorded and counted in a muscovite external detector. Hiess et al. (2012) have recently published for a suite of uranium-bearing minerals a data set with a range in ^{238}U/^{235}U values of about 5 per mil. However, since the uncertainties related to FTT are usually in the order of 5–10 %, we still assume a constant ^{238}U/^{235}U ratio as appropriate, and thus, the measurements of induced fission tracks give information about the ^{238}U concentration, allowing the determination of fission-track (FT) ages.

The drawback of neutron irradiation is the long time required for the decay of induced radioactivity. In an optimal case, the waiting time before handling the irradiated sample is around three months. Other problems in handling the radioactive material are the necessity of a

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special license to receive the material in the laboratory and the social pressure to decrease the use of nuclear reactors and handling of nuclear materials. One way to overcome these difficulties is using laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) to quantify the uranium content in samples to be dated (Hasebe et al. 2004; Donelick et al. 2005; Hadler et al. 2003, 2009; Chew and Donelick 2012; Shen et al. 2012).

One major issue in using the LA-ICP-MS for fission-track dating is the difficulty of determining the efficiency factors (etching and observation) in an internal surface of the mineral. Hasebe et al. (2009) have approximated the ratio of etching and counting efficiencies to be 1. Another important issue with LA-ICP-MS-based FTT is the lack of an ideal matrix-matched reference material for uranium content determination. In previous attempts, the uranium concentration was determined by LA-ICP-MS using synthetic glasses such as NIST SRM 610 or 612 for calibration. Despite similarities in the Ca and U behavior during ablation, they cannot be assumed to be identical. Thus, the matrix-matched reference material seems to be a primary necessity for LA-ICP-MS-based FTT.

This paper builds upon previously published works by Hasebe et al. (2004, 2009), Hadler et al. (2003, 2009); Donelick et al. (2005); Chew and Donelick (2012) and Shen et al. (2012), which aimed to determine apatite FT ages by LA-ICP-MS. The novelty in this work is to present a direct calibration, which allows the determination FT ages directly, independent of age standards, using the physical constants and experimentally determined efficiency factors. The efficiency factor in an internal surface of the mineral (subscript *is*) can be determined by the measurements of projected fission-track lengths. The product of observation (η) and etching (q) efficiencies, i.e., $[\eta q]_{\text{is}}$, can be directly determined (e.g., Jonckheere 2003; Enkelmann and Jonckheere 2003; Soares et al. 2013). Furthermore, two apatite crystals (Durango, Dur-2 with 7.44 µg/g U and Mud Tank, MT-7 with 6.88 µg/g U) were used as matrix-matched reference materials. These apatite crystals were rigorously tested found to present sufficient homogeneity to be used as reference materials for FTT (see below, "LA-ICP-MS" section).

To verify the new calibration, the ages of apatite samples from Durango, Fish Canyon Tuff, and Limberg were determined. The results were compared with reported ages determined independently by other radiometric dating techniques (e.g., ^{40}Ar/^{39}Ar and (U–Th)/He dating).

Fission-track age-equation calibrations

Age calibration with projected fission-track lengths

Fission-track ages can be calculated according to the following equation (Wagner and Van den Haute 1992):

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

where $N_S/(N_{238})$ is the number of spontaneous fission tracks (^{238}U atoms) per unit volume; $\lambda_{\alpha(f)}$ is the alpha (^{238}U spontaneous fission decay) constant; and t is the fission-track age in Ma.

For microscopic counting, only fission tracks that reach the polished surface can be etched and eventually observed. The relationship between planar and volumetric track distributions and etching and observation efficiencies is accounted for in geometric and efficiency parameters (Wagner and Van den Haute 1992). According to Soares et al. (2013), Eq. (1) can be rewritten as follows:

$$t = \frac{1}{\lambda_\alpha} \ln\left[1 + \frac{\lambda_\alpha}{\lambda_f} \frac{1}{g_{4\pi}[\eta q]_{is} r_S} \frac{\rho_S}{N_{238}}\right] \tag{2}$$

where ρ_S is the spontaneous fission-track density in the polished surface, r_S is the mean length of horizontal confined spontaneous fission tracks, $g_{4\pi}$ is the geometry factor for an internal surface, and $[\eta q]_{\text{is}}$ is the product between observation and etching efficiencies, which is obtained by comparison between theoretical and experimental projected fission-track length distributions (e.g., Jonckheere 2003; Enkelmann and Jonckheere 2003; Soares et al. 2013).

The N_{238}-value can be calculated according to Hasebe et al. (2004) using the U concentration determined by LA-ICP-MS:

$$N_{238} = N_A \frac{238_U \cdot 10^{-6} \cdot d}{M} \tag{3}$$

where N_A is the Avogadro constant, ^{238}U is the concentration (µg/g) of the isotope ^{238}U determined by LA-ICP-MS, d is the average density of apatite (3.19 g/cm^3), and M is the isotopic weight of ^{238}U.

However, considering that the uranium concentration through LA-ICP-MS is usually given in µg/g, Eqs. (2) and (3) lead to:

$$t = \frac{1}{\lambda_\alpha} \ln\left[1 + \zeta_{\text{ICPMS}} \frac{1}{[\eta q]_{\text{is}} r_S} \frac{\rho_S}{238_U}\right] \tag{4}$$

where

$$\zeta_{\text{ICPMS}} = \frac{\lambda_\alpha}{\lambda_f} \frac{M}{g_{4\pi} \cdot N_A \cdot 10^{-6} \cdot d} \tag{5}$$

Note that $_{ICPMS}$ is a constant for the material to be dated, in this case, apatite. The other quantities in the brackets of Eq. (4) are determined experimentally.

The U concentration and therefore ^{238}U from Eq. (3) can be determined from LA-ICP-MS measurements by normalizing the background-corrected counts per second of ^{238}U to ^{43}Ca, considering their respective isotope abundances, atomic weight of the respective element, and the Ca concentration of the sample and applying in addition a correction factor (relative sensitivity factor (RSF)) based on the measurement on the matrix-matched reference material (e.g., Jochum et al. (2007). Simplified this can be written as follows:

$$^{238}U = \frac{[C_U]_{\mathrm{UNK}}/[C_{\mathrm{Ca}}]_{\mathrm{UNK}}}{[C_U]_{\mathrm{ref}}/[C_{\mathrm{Ca}}]_{\mathrm{ref}}} U_{\mathrm{ref}} \tag{6}$$

In the equation above, $[C_U]_{\mathrm{ref}}$ and $[C_U]_{\mathrm{UNK}}$ are the counts per second of mass 238 of the reference material and the unknown sample, respectively, and $[C_{Ca}]_{\mathrm{ref}}$ and $[C_{Ca}]_{\mathrm{UNK}}$ are the counts per second of mass 43 measured during the same analyses of the reference material and unknown sample, respectively. U_{ref} is the uranium concentration of the reference material previously determined.

Another possible way of obtaining ^{238}U is to disregard calcium normalization and directly compare uranium counts in both reference material and unknown sample:

$$^{238}U = \frac{[C_U]_{\mathrm{UNK}}}{[C_U]_{\mathrm{ref}}} U_{\mathrm{ref}} \tag{7}$$

As it will be shown below, the approach with normalization to ^{43}Ca (Eq. 6) yields more consistent results.

Experimental procedures

Sample preparation

Apatite samples were mounted in epoxy resin, ground with sandpaper (#800 and 1,200), and polished with diamond suspension (9, 3 and 1 µm). Apatite samples were etched in a 5 % HNO_3 solution at 20 °C, for 55 s following the procedure of Tello et al. (2003, 2005). Horizontal confined fission-track lengths and spontaneous fission-track densities were measured to determine the FT age. For $[\eta q]_{\mathrm{is}}$, projected fission-track lengths were measured. According to Jonckheere and Van den Haute (2002), $[\eta q]_{\mathrm{is}}$ determined in a Durango apatite is the same for both spontaneous and induced projected fission-track length distributions. This is due to the low degree of annealing observed in age standard samples ($\approx$10 %) and other samples with fast cooling as in undisturbed volcanic rocks. As all samples used in this work are from a similar geological context (with low degree of annealing), the $[\eta q]_{\mathrm{is}}$ value was measured on the polished surface of an irradiated sample (Durango), resulting in better counting statistic in the measurements of the projected fission-track lengths and leading to an uncertainty associated with the $[\eta q]_{\mathrm{is}}$ value of $\approx$1 %.

After microscopic measurements, 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 procedure makes it possible to monitor whether the spots analyzed by LA-ICP-MS are located in the same area as that where the spontaneous fission-track density was determined previously.

EMPA

The major element composition was determined at the Institut für Geowissenschaften, Johannes Gutenberg-Universität Mainz, Germany, using a JEOL JXA 8900 RL electron microprobe with five WDS detectors. For calibration, natural and artificial crystals were used. For each analysis, at least five spots were used to calculate the average major element composition. We used the following parameters for the measurements: 15 kV acceleration voltage, beam current 12 nA, 5 µm spot diameter, and counting times of about 3 min. For matrix correction, the phi–rho–z method was applied (Pouchou and Pichoir 1984).

LA-ICP-MS

Uranium concentration measurements were taken at the Institut für Geowissenschaften at the Johannes Gutenberg-Universität Mainz, Germany, using an ESI NWR 193 Excimer laser with an output wavelength of 193 nm coupled to an Agilent 7500ce quadrupole ICP-MS. A spot size of 50 µm was chosen in order to scan most of the area where spontaneous fission-track densities were determined. Despite the possibility of raster analysis (Hasebe et al., 2004), our approach reduces the duration of the LA-ICP-MS measurements.

LA-ICP-MS analyses were carried out on a surface with etched fission tracks. Although Hasebe et al. (2009) have shown that no significant difference is observed between etched and non-etched surfaces, this experimental procedure was repeated in order to check the reproducibility of the LA-ICP-MS used in this work. For that purpose, nine apatite crystals from different geological settings were analyzed before and after etching: GH-5 and Bahia (Brazil), Namaqua (Africa), Mud Tank (Australia), Slyudyanka and Nikolaevskiy (Russia), B-2 and B-3 (Bamble, Norway), and Durango (Mexico). The laser spots before and after etching were carried out as close together as possible, thereby minimizing the effects of potential heterogeneity in the uranium concentration within a crystal.

Ablation was carried out in a He atmosphere, and the sample gas was mixed with Ar before entering the plasma, thereby enhancing sensitivity (Eggins et al. 1998). We used a pulse repetition rate of 10 Hz. The energy density was about 5 J/cm^2. A single-spot analysis consists of 20 s background, 50 s ablation, and 20 s washout. Before, and after about 10 spots on the unknown samples, matrix-matched reference materials (Dur-2 with 7.44 µg/g ± 3 % U (1RDS) and MT-7 with 6.88 µg/g ± 4 % U (1RSD) were analyzed for calibration. Additionally, we analyzed NIST SRM 610, USGS GSD-1G, and STDP5 phosphate glass (Klemme et al. 2008) at the beginning and the end of a sequence, as quality control materials (QCM). The program GLITTER 4.4.1 (http://www.glitter-gemoc.com) was used for data reduction. Measured isotope intensities were normalized to ^{43}Ca, applying the CaO content previously determined by EMPA. The U concentrations measured on the QCM agree within 2.9, 1.2, and 8.1 % with the preferred values for NIST SRM 610 and USGS GSD-1G reported in the GeoReM database (http://georem.mpch-mainz.gwdg.de/) (Jochum et al. 2005, 2011) and the preliminary recommended values of Klemme et al. (2008) for STDP5, respectively. For the QCM, relative standard deviation (1RSD) for the averaged U concentrations of the single-spot measurements was <6 % for NIST SRM 610 ($n = 9$) and USGS GSD-1G ($n = 6$), and <10 % for STDP5 ($n = 6$), respectively. We note that before we adopted Dur-2 and MT-7 as matrix-matched reference materials, a thorough study of homogeneity was carried out on large natural apatite crystals. A central 1-mm-thick slice of about 1 cm^2 was cut from each crystal. The surface was scanned by EMPA along a profile to determine the major element composition. Subsequently, the same surface was scanned (doing five times 5 single spots) by LA-ICP-MS to determine the trace element composition (e.g., U, Th, and Sm). In addition, other slices from several locations within the crystal were also analyzed by LA-ICP-MS. The goal of this test was to verify volumetric homogeneity of the monitored elements in the Dur-2 and MT-7 apatite crystals.

Results and discussion

The results of the homogeneity test of the matrix-matched reference materials are shown in Fig. 1. The variation in the uranium concentration is within 3 % (1RSD). Thus, the Dur-2 and MT-7 apatite crystals have proven to be appropriate reference materials for U by LA-ICP-MS analysis. Small pieces (2–3 mm^3) of both crystals have been prepared in order to share them with the fission-track community to be widely used as matrix-matched reference materials.

Figure 2a shows the uranium concentration before and after etching of the analyzed samples. All samples, besides the Namaqua apatite, show good agreement with both measurements. The Namaqua apatite is very heterogeneous (17 % 1RSD) with respect to its uranium concentration, and therefore, we do not expect the age results of both measurements to be as consistent as those of the other more homogenous samples. Figure 2b shows that there is no systematic variation in the uranium concentration before and after etching. Statistically, the difference between both measurements is around 5 %, which corresponds to the typical reproducibility of LA-ICP-MS analyses. This is in agreement with the result of Hasebe et al. (2009).

The fission-track ages calculated by Eq. (4) and the parameters required for the calculation are given in Table 1. Considering that the samples (Durango, Fish Canyon Tuff, and Limberg) have undergone the same degree of annealing, we calculated $[\eta q]_{\mathrm{is}} = 0.91 \pm 1.0$ %, with the induced projected fission-track distribution (3108 tracks) measured in a Durango apatite in agreement with the value reported by Jonckheere and Van den Haute (2002). The value for r_S is the mean length of horizontal confined spontaneous fission tracks, and the value of $g_{4\pi}$ is, by definition, 1. The values of the constants used are as follows: $\lambda_\alpha = 1.55125 \times 10^{-10}$ a^{-1} (Lederer and Shirley 1978) and $\lambda_f = (8.5 \pm 0.1) \times 10^{-17}$ a^{-1} (Holden and Hoffman 2000). The ${}_{ICPMS}$ calculated with Eq. (5) is $(2.26 \pm 0.05) \times 10^{-10}$ cm^3. Only the uncertainty associated with λ_f was taken into account since the other uncertainties are about one-tenth of one percent and, therefore, are considered to be of no consequence for the overall uncertainty.

The ages obtained for Durango apatite are as follows: 31.4 ± 2.5 and 31.8 ± 2.4 Ma for MT-7 and Dur-2 as matrix-matched reference material, respectively. The ages are in agreement, within 1σ, with those reported by McDowell et al. (2005) (31.44 ± 0.18 Ma, ^{40}Ar/^{39}Ar dating and 31.02 ± 0.18 Ma, (U–Th)/He dating). For Limberg, the ages are as follows: 17.8 ± 1.8 and 18.2 ± 1.8 Ma, considering MT-7 and Dur-2 as reference materials, respectively. Also, these values are in agreement, within 1σ, with reported ages (16.8 ± 1.0 Ma for apatite and 16.5 ± 1.0 Ma for titanite, both determined by (U–Th)/He dating) (e.g., Kraml et al. 2007). Finally, for Fish Canyon Tuff, the ages are as follows: 27.0 ± 2.9 and 27.2 ± 2.9 Ma using MT-7 and Dur-2 as reference materials, respectively. The ages are in agreement, within 1σ, with those reported by, e.g., Lanphere and Baadsgaards (1997) (27.55 ± 0.24 Ma determined by K–^{39}Ar dating of biotite).

To prevent apparent variations in the calculated uranium concentration due to variation in equipment operation conditions (e.g., plasma instabilities), the calculation must be based on the normalization of the average ^{238}U counts per second to the ^{43}Ca counts per second of a single-spot analysis (Eq. 6). As can be evaluated from Figs. 3 and 4, the uranium concentrations determined using the ^{238}U cps

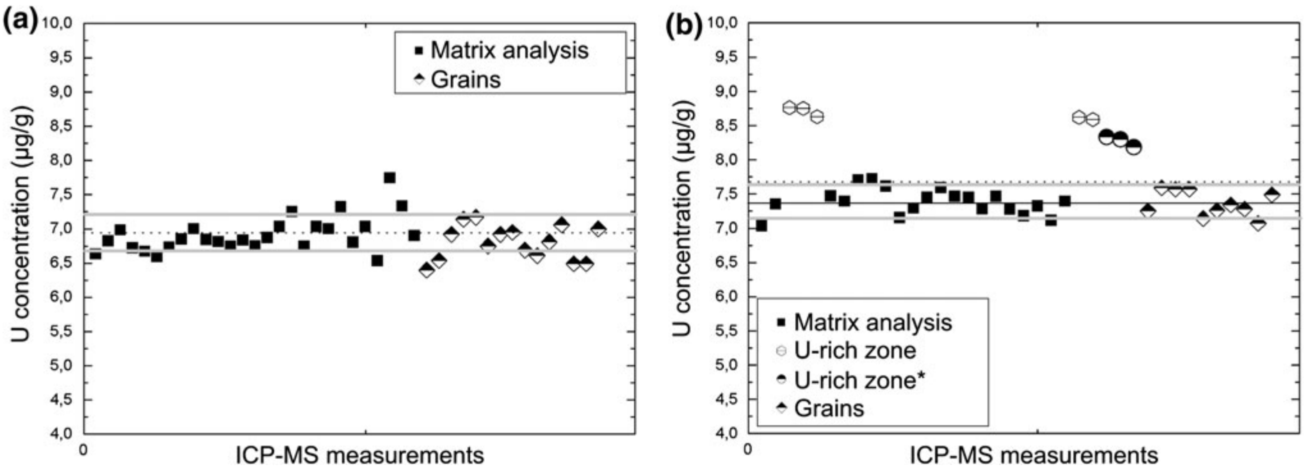

Fig. 1 **a** U concentration of MT-7 and **b** Dur-2 determined by LA-ICP-MS. The *dashed line* represents the average of all analyzed spots. For Dur-2, the *solid line* indicates the average, excluding the spots on a small U-rich zone in the crystal boundary. The *gray lines* represent the standard deviation associated with the average (1SD) considering the recommended value. The U-rich zone on the crystal boundary has been avoided in the calculations of the FTT ages

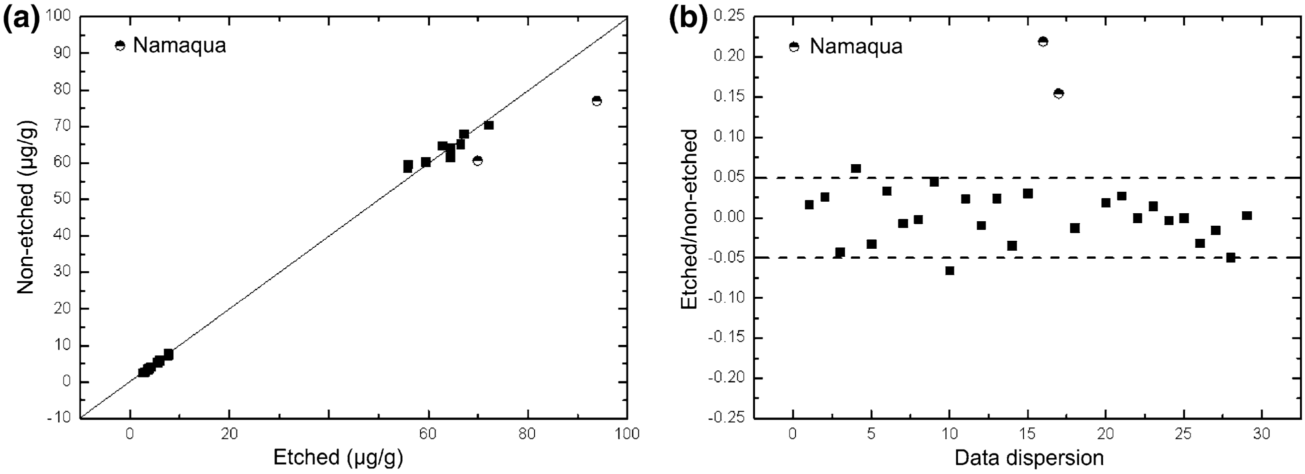

Fig. 2 **a** Uranium concentration (µg/g) determined before and after etching; **b** etched/non-etched ratio of the uranium concentration

Table 1 Fission-track age determination by LA-ICP-MS

Sample	N_g	N_S	ρ_S ($\times 10^5$ cm^2)	r_S ($\times 10^{-4}$ cm)	CaO	U (MT-7)	U (Dur-2)	t (Ma) (MT-7)	t (Ma) (Dur-2)
Dur-5	24	224	3.16 ± 0.2	14.7 ± 0.1	54.6	10.9	10.8	31.4 ± 2.5	31.8 ± 2.4
Lim	23	189	3.70 ± 0.3	15.2 ± 0.1‡	54.5*	22.0	21.4	17.8 ± 1.8	18.2 ± 1.8
FCT	24	207	3.17 ± 0.222	14.5 ± 0.1	51.8*	13.4	13.3	27.0 ± 2.9	27.2 ± 2.9

The values used to calculate the ages using Eq. (4) are as follows: $[\eta q]_{\mathrm{is}} = 0.91 \pm 1$ %; g_4=1; $\lambda_\alpha = 1.55125 \times 10^{-10}$ a^{-1} (Lederer and Shirley 1978) and $\lambda_f = (8.5 \pm 0.1) \times 10^{-17}$ a^{-1} (Holden and Hoffman, 2000); $\zeta_{ICPMS} = (2.26 \pm 0.05) \times 10^{-10}$

Dur Durango apatite, *Lim* Limberg t3 tuff, *FCT* Fish Canyon Tuff, N_g number of grains analyzed, N_S number of spontaneous fission-track counted, ρ_S spontaneous fission-track density, r_S mean length of horizontal spontaneous confined fission tracks, *U* uranium concentration (µg/g) determined by LA-ICP-MS using Dur-2 and MT-7 as matrix-matched reference material, *t* fission-track age in Ma

* values reported by Barbarand et al. (2003). ‡ values reported by Kraml et al. (2007)

without normalization to ^{43}Ca (Eq. 7) are in disagreement with the determinations resulting from Eq. 6 (Figs. 3a,c,e; 4a,c,e), especially for Durango apatite samples. However, if the results of Eq. (6) are compared with U concentrations determined using Glitter 4.4.1 for data reduction, the points fall onto the 1:1 line, since in Glitter, the element concentrations are also calculated by normalizing the measured intensities on, e.g., ^{238}U to an internal standard (here

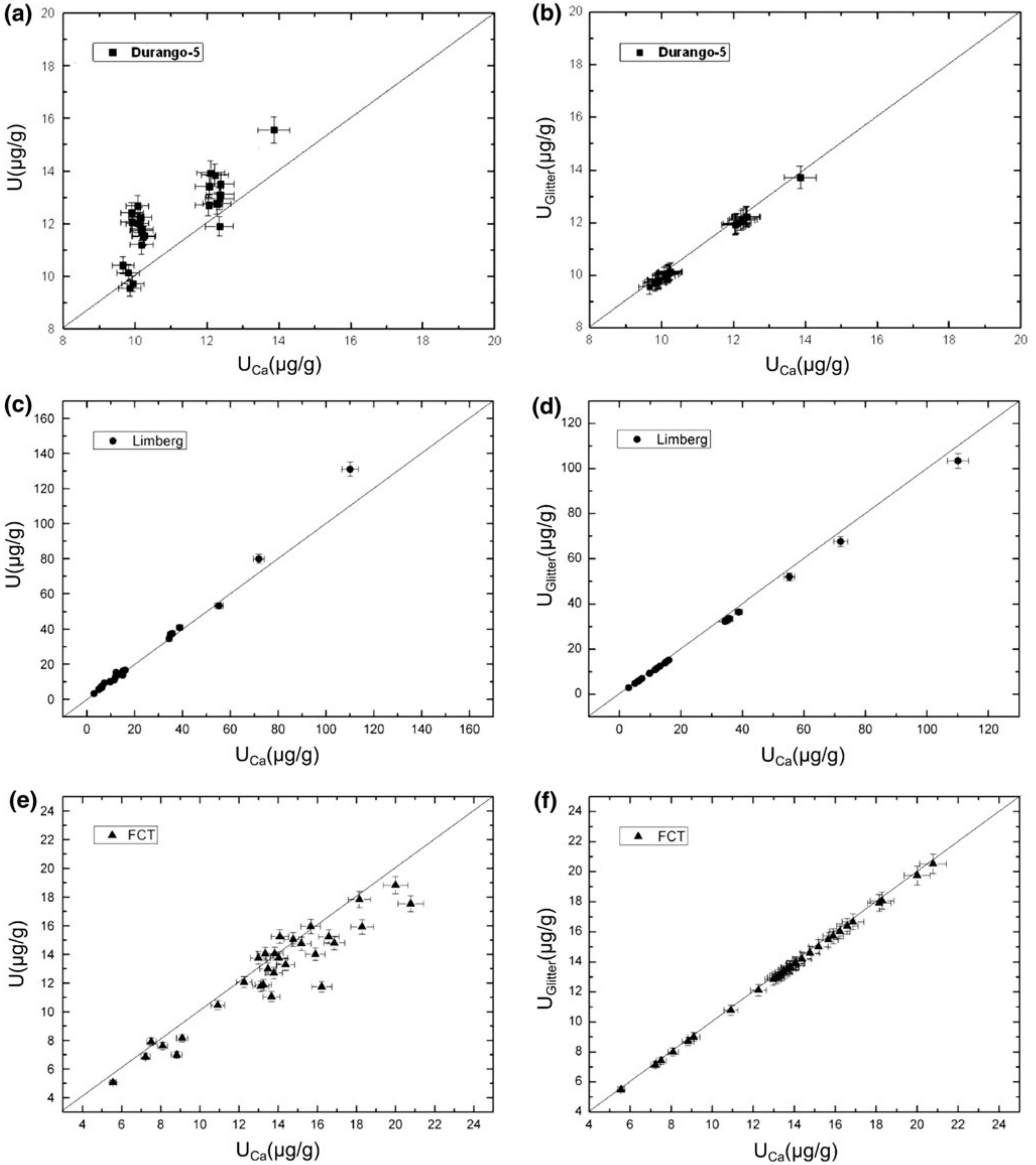

Fig. 3 Comparison of uranium concentrations calculated by using Glitter 4.4.1 ($U_{Glitter}$), Eq. 6 (U_{Ca}), and Eq. 7 (U), using MT-7 as reference material. **a** U_{Ca} versus U for Durango apatite; **b** U_{Ca} versus $U_{Glitter}$ for Durango apatite; **c** U_{Ca} versus U for Limberg apatite; **d** U_{Ca} versus $U_{Glitter}$ for Limberg apatite; **e** U_{Ca} versus U for FCT apatite; **f** U_{Ca} versus $U_{Glitter}$ for FCT apatite

^{43}Ca) (Figs. 3b, d, f; 4b, d, f). In this study, the fission-track ages were calculated using the U concentration resulting from the data reduction using Glitter 4.4.1. Finally, both matrix-matched reference materials present consistent results, and therefore, both can be used for LA-ICP-MS-based FTT.

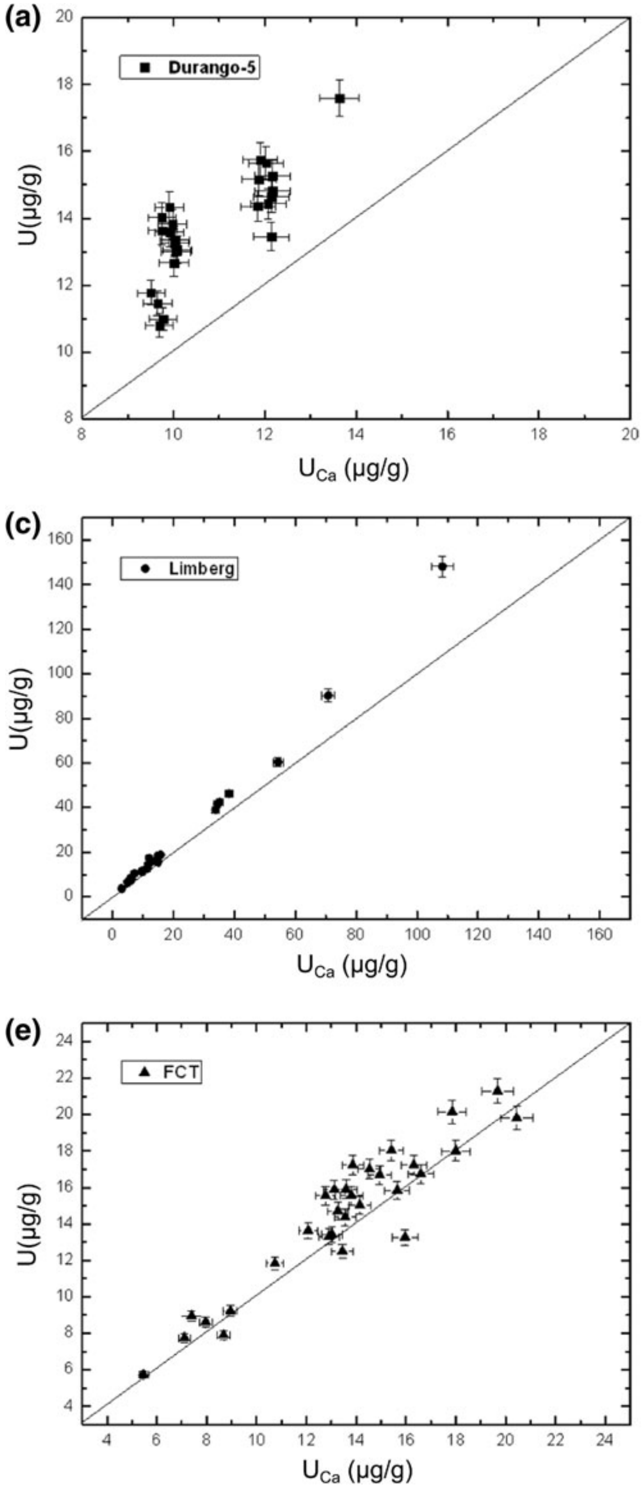

Fig. 4 Comparison of uranium concentrations calculated by using Glitter 4.4.1 (U$_{Glitter}$), Eq. 6 (U$_{Ca}$), and Eq. 7 (U), using Dur-2 as reference material. **a** U$_{Ca}$ versus U for Durango apatite; **b** U$_{Ca}$ versus U$_{Glitter}$ for Durango apatite; **c** U$_{Ca}$ versus U for Limberg apatite; **d** U$_{Ca}$ versus U$_{Glitter}$ for Limberg apatite; **e** U$_{Ca}$ versus U for FCT apatite; **f** U$_{Ca}$ versus U$_{Glitter}$ for FCT apatite

Conclusion

We have shown consistent fission-track ages determined by LA-ICP-MS using the recommended value for the spontaneous fission decay constant and the direct measurements of the efficiency in an internal surface $[\eta q]_{\mathrm{is}}$. For this purpose, two apatite samples were used as matrix-matched reference materials. The results using the direct measurements are in agreement, within 1σ, with ages reported in the literature, dated by other radiometric dating techniques. Furthermore, the ages are also in agreement with those determined by conventional fission-track procedures through absolute approach (metal activation monitor) and traditional ζ-calibration (Enkelmann et al. 2005). In this context, we can state that the method presented in this work is sufficiently consistent to be used for FTT. Furthermore, as the fission-track ages were determined directly without using an age standard sample, the FTT approach presented appears to be an independent dating method.

Acknowledgments This work was founded by Fundação de Amparo à Pesquisa do Estado de São Paulo (FAPESP, Brazil) and Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq, Brazil). The authors thank Dr. Richard Ketcham and an anonymous reviewer for their comments and suggestions which helped to improve this manuscript. We thank Dr. Eleanor Green for helping with language editing. Finally, we thank Dr. Raymond Jonckheere for providing the Limberg sample.

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