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TECHNICAL NOTE

U–Th isotopic microanalysis of zircon reference materials and KBSI working standards

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Abstract

Background: The ^{238}U–^{230}Th disequilibrium dating of the mineral zircon ($ZrSiO_4$) provides an efficient tool for investigating the time scales of Quaternary magmatic processes. In situ mass spectrometric U–Th microanalysis of zircon requires careful calibration and correction of the measured isotope data, particularly for the instrumental fractionation of U and Th isotopes.

Findings: For the selection of suitable calibration materials for U–Th isotopic analysis using a laser ablation multiple collector inductively coupled plasma mass spectrometer (LA-MCICPMS), we estimated the homogeneity of four reference zircons (91500, TEMORA 2, FC1, and Plešovice) and two zircon working standards (LKZ-1 and BRZ-1) in terms of their $^{232}Th/^{238}U$ ratios, based on the Pb isotopic compositions measured by a sensitive high-resolution ion microprobe (SHRIMP). The measured LA-MCICPMS $^{232}Th/^{238}U$ ratios of the zircons were calibrated externally using the SHRIMP $^{208}Pb/^{206}Pb$-based average value of the 91500 zircon, $^{232}Th/^{238}U = 0.351 \pm 0.035$ (error corresponds to 1 standard deviation). The molecular interference-corrected $^{230}Th/^{232}Th$ ratios of the zircons were calibrated based on the assumption that the Plešovice zircon is in ^{238}U–^{230}Th secular equilibrium. After the calibration and correction, the activity ratios of $^{230}Th/^{232}Th$ and $^{238}U/^{232}Th$ for all reference zircons and working standards were plotted on the equiline.

Conclusions: This study confirms that the 91500 zircon is relatively homogeneous in terms of U/Th ratios (relative standard deviation = ~ 10%) and does not support a recent claim that the Plešovice zircon is not in ^{238}U–^{230}Th radioactive equilibrium. The working standards LKZ-1 and BRZ-1 can be used to check the reliability of U–Th isotopic analyses for Quaternary zircons.

Keywords: Zircon standard, U–Th isotopes, Uranium-series disequilibrium dating, Laser ablation-ICPMS

Introduction

Geochronological methods using parent–daughter isotope pairs with long half-lives, such as ^{87}Rb–^{87}Sr (half-life = 48.8 Gy), ^{147}Sm–^{143}Nd (106 Gy), ^{238}U–^{206}Pb (4.47 Gy), and ^{40}K–^{39}Ar (1.25 Gy) systems, pose many challenges for the dating of Quaternary rocks and minerals, mainly because the amounts of daughter isotopes accumulated are extremely small. Also, in the case of ^{40}K (or ^{40}Ar)–^{39}Ar dating, problems associated with excess argon cannot always be avoided (e.g., Esser et al. 1997). On the other hand, the widely used radiocarbon dating method is only applicable to the last ca. 50,000 years because of the short half-life of ^{14}C (= 5730 years). The geochronological gaps between the ranges of these dating methods are filled by uranium-series geochronometers using short-lived radionuclides in the decay chains, such as ^{230}Th (half-life = 75,600 years) and ^{231}Pa (32,500 years).

The mineral zircon ($ZrSiO_4$) presents many advantages as a ^{238}U–^{230}Th geochronometer. It has a relatively high uranium content (ca. 100–1000 ppm) and is strongly resistant to physicochemical breakdown. Furthermore, the intracrystalline diffusion of its constituent cations is relatively slow (Cherniak and Watson 2003). Therefore, Quaternary zircons have been a prime target for ^{238}U–^{230}Th disequilibrium dating since the advent of in situ microanalytical

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techniques that employ secondary ion mass spectrometry or laser ablation-assisted inductively coupled plasma mass spectrometry (LA-ICPMS) (Reid et al. 1997; Zou et al. 2010; Ito et al. 2013). These techniques have made it possible to investigate the sub-grain time records in single crystals but require careful calibration and correction of the measured isotope data. The zircon reference material plays a critical role in the calibration of LA-ICPMS data (Guillong et al. 2016).

For the selection of suitable reference materials for calibration of laser ablation data, we estimated the homogeneity of four reference zircons (91500, TEMORA 2, FC1, and Plešovice) and two zircon working standards (LKZ-1 and BRZ-1) in terms of their $^{232}Th/^{238}U$ ratios, based on Pb isotopic compositions measured with a sensitive high-resolution ion microprobe (SHRIMP). Then, we assessed the reliability of the calibration for LA-multiple collector (MC) ICPMS zircon U–Th isotope data using the $^{208}Pb/^{206}Pb$-based average $^{232}Th/^{238}U$ ratio in the most homogeneous zircon reference material (the 91500 zircon), in addition to the ^{238}U–^{230}Th radioactive equilibrium assumed for the Plešovice zircon.

Materials

According to Wiedenbeck et al. (1995), the 91500 zircon specimen stored at the Harvard Mineralogical Museum originally consisted of a single crystal presumably collected from a porphyroblastic syenite gneiss in Ontario, Canada. The authors reported its chemical composition and isotope dilution-thermal ionization mass spectrometric (ID-TIMS) $^{207}Pb/^{206}Pb$ age of 1065.4 ± 0.3 Ma. Its average U concentration and Th/U ratio were estimated to be 81.2 ppm and 0.3444 ± 0.0009, respectively. The sub-grain distributions of trace elements in this reference zircon were investigated by Wiedenbeck et al. (2004). The 91500 zircon is a widely used reference material for O ($\delta^{18}O_{V\text{-}SMOW} = 10.07 \pm 0.03‰$; Valley 2003) and Hf isotopes ($^{176}Hf/^{177}Hf = 0.282308 \pm 0.000006$; Blichert-Toft 2008).

The TEMORA 2 zircon, introduced by Black et al. (2004), was collected from a gabbroic diorite stock in the Lachlan Orogen of Eastern Australia. The ID-TIMS analyses for this zircon yielded a $^{206}Pb/^{238}U$ age of 416.78 ± 0.33 Ma, moderate U concentrations of 320–82 ppm, and Th/U ratios of 0.51–0.33 (Black et al. 2004).

The FC1 zircon was collected from an olivine gabbroic anorthosite in northeastern Minnesota. Paces

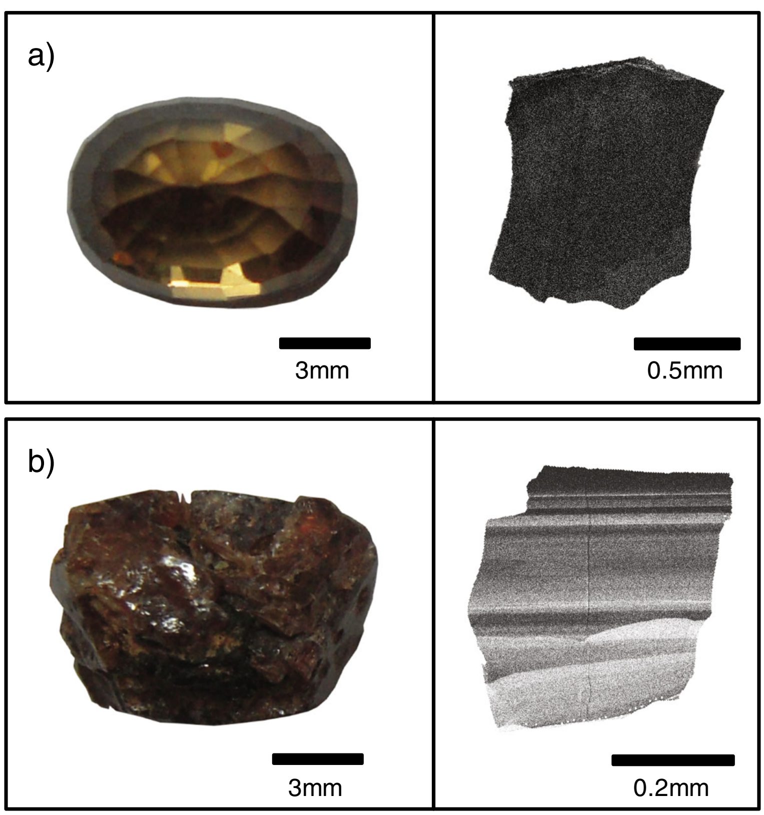

Fig. 1 Photographs of KBSI working standards (**a**) LKZ-1 and (**b**) BRZ-1 and their representative cathodoluminescence images

and Miller Jr (1993) reported its ID-TIMS $^{207}Pb/^{206}Pb$ age as 1099.0 ± 0.6 Ma, with U concentrations ranging from 1510 to 218 ppm and Th/U ratios of 0.604–0.519.

The Plešovice zircon came from a potassic granulite in the southern Bohemian Massif, Czech Republic. The ID-TIMS analyses of this zircon by Sláma et al. (2008) yielded a weighted mean $^{206}Pb/^{238}U$ age of 337.13 ± 0.37 Ma and relatively low Th/U ratios of 0.16–0.04. The U concentrations of the pristine domains in the Plešovice zircon, determined by LA-ICPMS, range from 1106 to 465 ppm (Sláma et al. 2008).

The Korea Basic Science Institute (KBSI) is developing zircon working standards, namely LKZ-1 and BRZ-1. The former, from Sri Lanka, is a single transparent pale yellow crystal (length to width = ~ 1.7:1) with dark cathodoluminescence (CL) emissions, and the latter, from Brazil, consists of translucent dark brown crystals and exhibits a symmetric CL banding (Fig. 1). The SHRIMP analyses by Kim et al. (2015) yielded a weighted mean $^{206}Pb/^{238}U$ age of 591.1 ± 2.7 Ma (n = 29, mean square of weighted deviates [MSWD] = 2.1) for LKZ-1 and 572.2 ± 4.9 Ma (n = 30, MSWD = 4.1) for BRZ-1. The LKZ-1 is fairly high in U content, at 646 ± 45 ppm (hereinafter, errors correspond to 1 standard deviation (SD) unless otherwise stated), whereas the BRZ-1 has a significantly lower U content (90 ± 32 ppm) (Kim et al. 2015).

Methods

For Pb isotopic measurements using the KBSI SHRIMP, the primary O_2^- beam was focused into a ~ 25-μm-diameter spot at an accelerating voltage of 10 kV. The collector slit width was fixed at 100 μm, achieving a mass resolution of ~ 5000 at 1% peak height. The common Pb was removed by ^{204}Pb correction method (Williams 1998) using the model of Stacey and Kramers (1975). Data processing was conducted using the SQUID 2.50 program (Ludwig 2009).

Zircon U–Th isotopic analyses were carried out at the KBSI using a Plasma II MCICPMS (Nu Instruments) equipped with an NWR193-nm ArF excimer laser ablation system (ESI). Typical laser ablation and ICPMS parameters are summarized in Table 1. Signal intensities were measured with Faraday collectors (for ^{238}U and ^{232}Th) and ion counters (for ^{230}Th and 228 mass) simultaneously. The raw data were processed using Iolite 2.5 running within Igor Pro 6.3.5.5 software (Paton et al. 2011). The activity ratios were calculated using the decay constants proposed by Steiger and Jäger (1977) (for ^{238}U and ^{232}Th) and Cheng et al. (2013) (for ^{230}Th).

Table 1 LA-MCICPMS instrument and operational parameters

MCICPMS	
Instrument	Nu Plasma II
RF power	1300 W
Reflected power	< 1 W
Mixed gas and flow rate	Ar, ~ 0.9 L/min
Auxiliary gas and flow rate	Ar, 0.8 L/min
Cool gas and flow rate	Ar, 13 L/min
Sampler cone	Ni (1 mm orifice)
Skimmer cone	Ni (0.7 mm orifice)
Data acquisition mode	Time resolved analysis
Integration times	0.4 s
Collectors	2 Faraday cups and 2 ion counters
Measured isotopes	^{238}U, ^{232}Th, ^{230}Th, 228 mass
Laser ablation system	
Laser	ESI machines 193 nm ArF excimer laser
Ablation mode	Single hole drilling
Cell	Two volume 2 cell (10 × 10 cm)
Sample transport tubing	0.5 m length
Pulse repetition rate and width	10 Hz, < 4 ns
Pulse energy density	~ 6 J/cm^2
Ablation duration	30 s
Carrier gas and flow rate	He, 0.3 L/min
Crater size	30 μm
Drill depth	~ 15 μm

Results and discussion

SHRIMP analysis

Generally, the mass fractionation of Pb in the SHRIMP analysis occurs within the limits of analytical precision (Williams 1998 and references therein), and thus, no correction was applied to the measured $^{208}Pb/^{206}Pb$ ratios. The common Pb-corrected $^{208}Pb/^{206}Pb$ ratios of the zircon reference materials and KBSI working standards are listed in Additional file 1: Table S1. The $^{232}Th/^{238}U$ ratios were calculated by assuming that the U–Pb and Th–Pb isotopic systems in the zircons were concordant (Eq. 1):

$$^{208}Pb/^{206}Pb_{SHRIMP} = {}^{232}Th/^{238}U_{CONCORDANCE}\left[\left(e^{\lambda_{232}\cdot t}-1\right)/\left(e^{\lambda_{238}\cdot t}-1\right)\right] \quad (1)$$

where λ_{232}, λ_{238}, and t are the ^{232}Th decay constant (4.9475×10^{-11} y^{-1}), the ^{238}U decay constant (1.55125×10^{-10} y^{-1}) (Steiger and Jäger 1977), and zircon crystallization age, respectively. The $^{232}Th/^{238}U$ ratios were also calculated by the SQUID program, using the experimental relationship proposed by Williams et al. (1996) (Eq. 2):

$$^{232}Th/^{238}U_{SQUID} = \left[0.03446\left(^{238}UO^{+}/^{238}U^{+}\right) + 0.868\right]\left(^{232}ThO^{+}/^{238}UO^{+}\right) \quad (2)$$

The average $^{232}Th/^{238}U_{CONCORDANCE}$ of the 91500 zircon (0.352 ± 0.035, relative standard deviation [RSD] = 10%, n = 48) is in agreement with the previous ID-TIMS (0.356 ± 0.001; Wiedenbeck et al. 1995) and LA-ICPMS ([Th] = 29.9 ppm (RSD = 7%), [U] = 80.0 ppm (RSD = 10%), $^{232}Th/^{238}U$ = 0.386 ± 0.047; Wiedenbeck et al. 2004) values within the measurement uncertainties. The other reference zircon materials and working standards were less homogeneous in terms of Th/U ratios (Fig. 2). However, the average $^{232}Th/^{238}U_{CONCORDANCE}$ ratios of TEMORA 2 (0.451 ± 0.080, RSD = 18%, n = 47), FC1 (0.577 ± 0.125, RSD = 22%, n = 149), and Plešovice zircons (0.181 ± 0.157, RSD = 87%, n = 31) are consistent with the previous ID-TIMS and LA-ICPMS results (Paces and Miller Jr 1993; Black et al. 2004; Sláma et al. 2008). The Plešovice zircon showed the widest variation in $^{232}Th/^{238}U$ (0.52–0.07). The LKZ-1 and BRZ-1zircons yielded an average $^{232}Th/^{238}U_{CONCORDANCE}$ of 0.139 ± 0.015 (RSD = 11%, n = 6) and 0.381 ± 0.092 (RSD = 24%, n = 23), respectively. As shown in Fig. 3a, the $^{232}Th/^{238}U$ ratios calculated by the two equations (i.e., $^{232}Th/^{238}U_{CONCORDANCE}$ and $^{232}Th/^{238}U_{SQUID}$) agree well with each other. The $^{232}Th/^{238}U_{SQUID}$ ratios with > 1% standard error (SE) are not displayed in this figure. It should also be noted that the $^{208}Pb/^{206}Pb$ ratios with poor internal

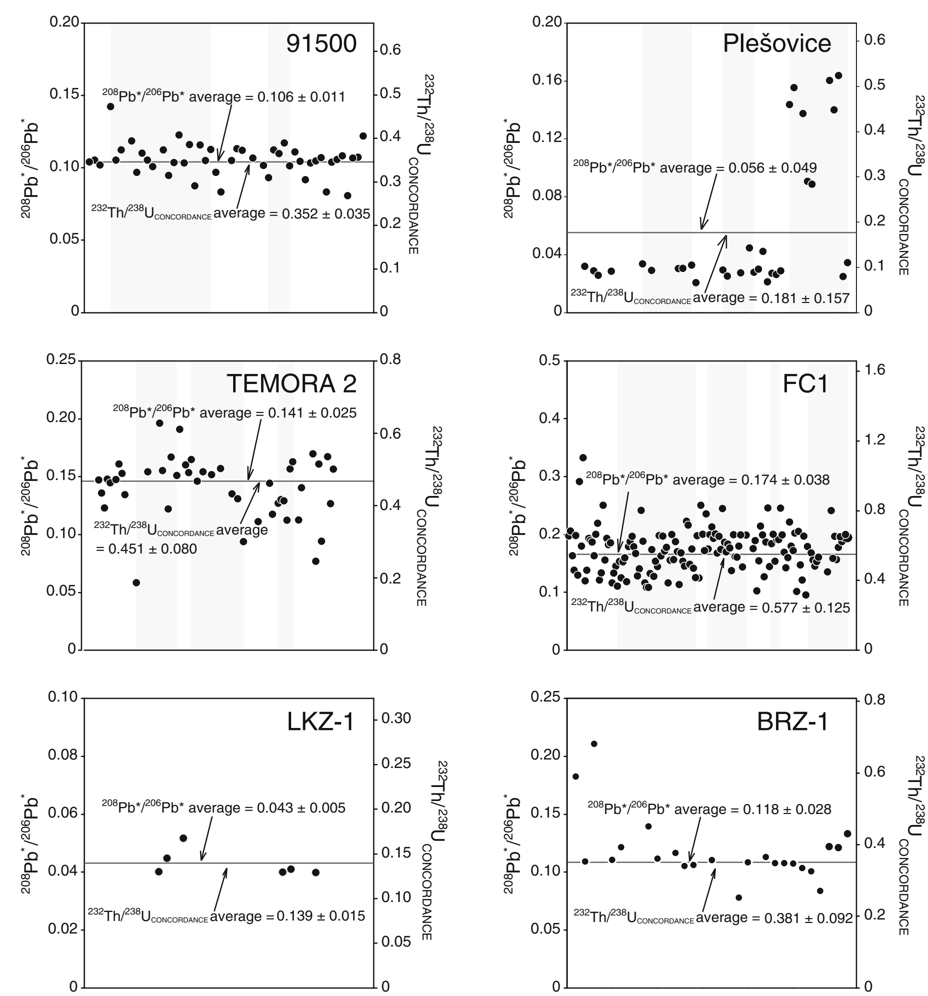

Fig. 2 The SHRIMP $^{208}Pb/^{206}Pb$ and calculated $^{232}Th/^{238}U_{CONCORDANCE}$ for the zircons. Each analytical session is marked in shaded and unshaded areas

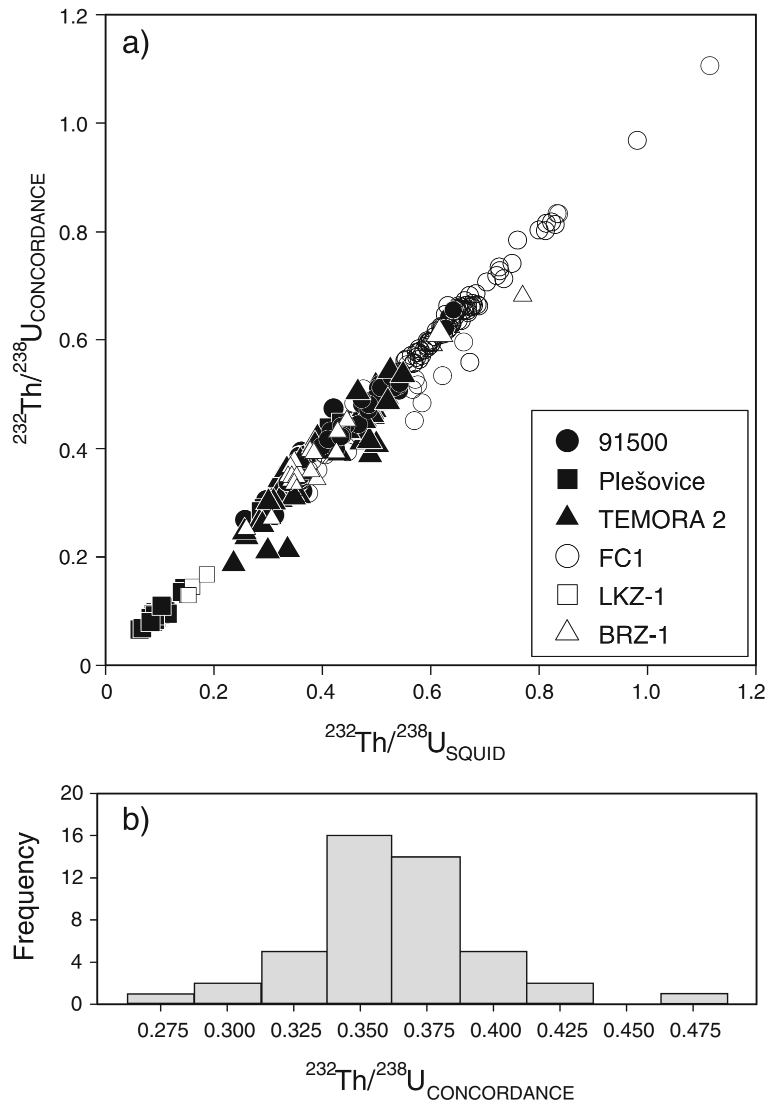

Fig. 3 a Comparison between $^{232}Th/^{238}U$ ratios calculated using SHRIMP $^{208}Pb/^{206}Pb$ data and the assumption that the U–Pb and Th–Pb isotopic systems in the zircons are concordant ($^{232}Th/^{238}U_{CONCORDANCE}$) and those calculated by the SQUID program ($^{232}Th/^{238}U_{SQUID}$). **b** The frequency histogram of $^{232}Th/^{238}U_{CONCORDANCE}$ ratios for the 91500 zircon

precision (defined here as SE > 5%) were not considered in the calculation of $^{232}Th/^{238}U_{CONCORDANCE}$. The $^{232}Th/^{238}U_{CONCORDANCE}$ ratios calculated for the 91500 zircon follow a normal distribution (Fig. 3b).

LA-MCICPMS analysis

Essentially, correction of inter-elemental isotope ratios measured by the LA-ICPMS is unavoidable because the sensitivity varies between elements due to mass bias and differences in the vaporization and ionization efficiency of the aerosol introduced into the plasma. Matrix-matched and homogeneous standard materials are required for the external calibration of inter-elemental LA-ICPMS data. Our SHRIMP analyses confirm that the 91500 zircon is the most homogeneous in terms of the Th/U ratio among the analyzed reference materials. We employed its $^{238}U/^{232}Th_{CONCORDANCE}$ (= 2.85 ± 0.29) for the calibration of LA-MCICPMS $^{238}U/^{232}Th$ data. The relative sensitivity factor (RSF = 2.85 over $^{238}U/^{232}Th_{measured}$) was fairly constant during single analytical tests (RSD < 3%) but varied significantly from run to run (Fig. 4a); this means that the RSF

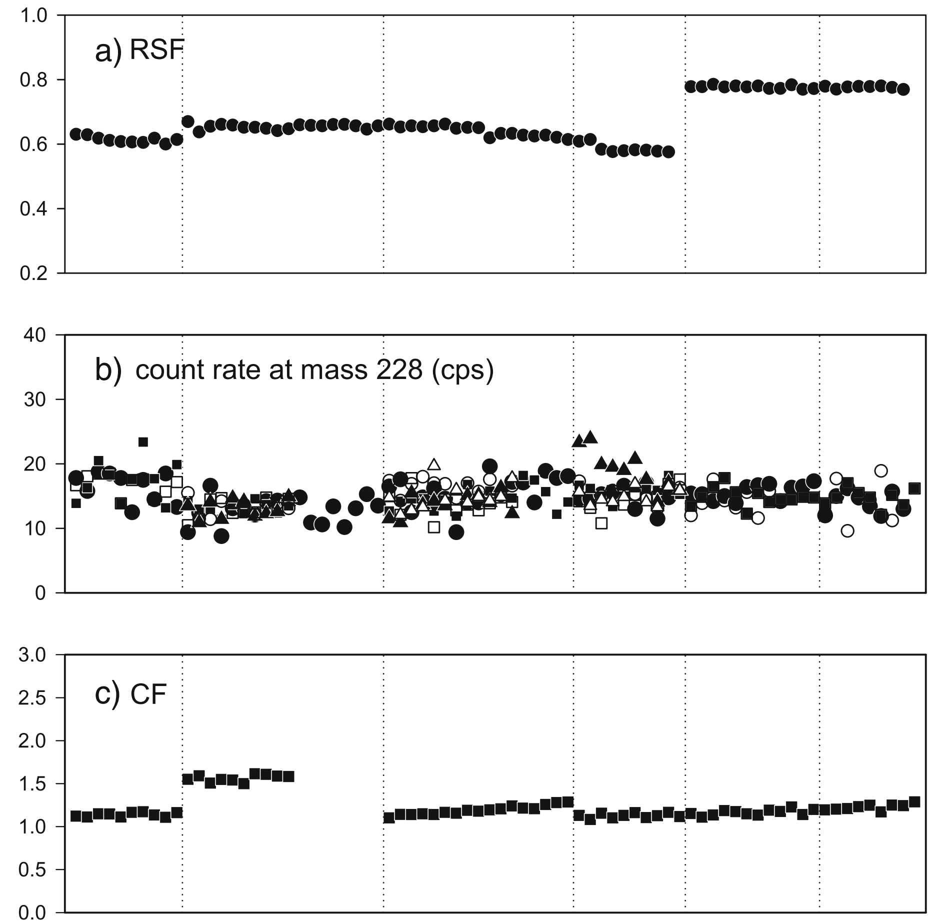

Fig. 4 Variation of **a** the relative sensitivity factor (RSF) measured for the 91500 zircon (= 2.85 over $^{238}U/^{232}Th_{measured}$), **b** the count rate at mass 228, and **c** the correction factor (CF) measured for the Plešovice zircon [= $(^{238}U/^{232}Th)_{corrected}/(^{230}Th/^{232}Th)_{measured}$] in the LA-MCICPMS analysis. Symbols are the same as in Fig. 3

value needs to be checked before and after the sample analysis.

Of all the reference zircons analyzed in this study, the Plešovice zircon showed the widest variation in $^{232}Th/^{238}U$. Ito (2014) argued that the Plešovice zircon is not in secular equilibrium, but this hypothesis may have been skewed by biased corrections for abundance sensitivity and molecular interferences (Guillong et al. 2015). During our LA-MC-ICP-MS analyses, the count rate at mass 228 was relatively constant (Fig. 4b), supporting the interpretation of Guillong et al. (2015) that mass 230 contains contributions from molecular zirconium sesquioxide ions. We removed these contributions using the isotopic abundance ratio proposed for $^{90}Zr_2$ and $(^{90}Zr^{92}Zr + ^{91}Zr_2)$ (= 0.7142; Guillong et al. 2015). Following this, the ^{238}U–^{230}Th radioactive equilibrium assumed for the Plešovice zircon was used for the correction and calibration of the peak tail of ^{232}Th and the Faraday–ion counter efficiency. The correction factor measured for the Plešovice zircon [= $(^{238}U/^{232}Th)_{corrected}/(^{230}Th/^{232}Th)_{measured}$] also varied significantly from run to run (Fig. 4c). After calibration and correction, we confirmed that the activity ratios of $^{230}Th/^{232}Th$ and $^{238}U/^{232}Th$ for all zircons were plotted on the equiline as displayed in Fig. 5 and listed in Additional file 2: Table S2.

Conclusions

This study confirms that the 91500 zircon is relatively homogeneous in terms of U/Th ratios (RSD = ~ 10%). After calibration and correction using the SHRIMP $^{208}Pb/^{206}Pb$-based average $^{232}Th/^{238}U$ ratio for the 91500 zircon (= 0.351 ± 0.035), and the ^{238}U–^{230}Th radioactive equilibrium assumed for the Plešovice

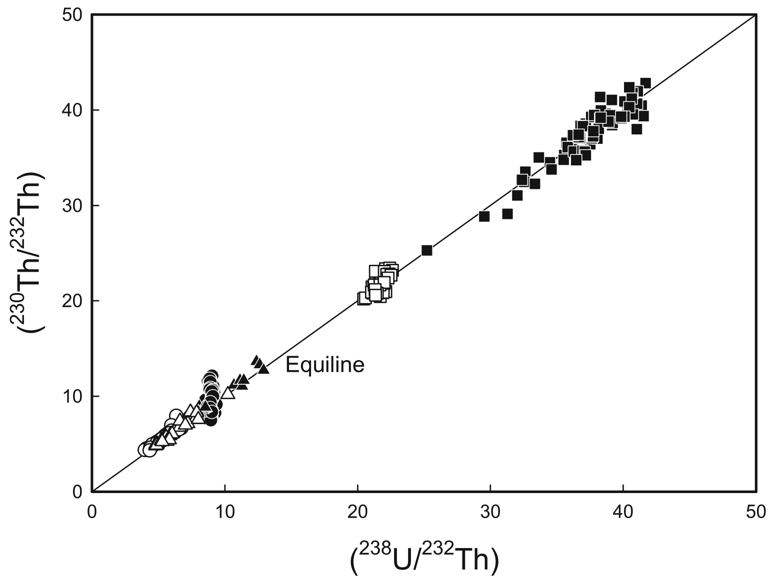

Fig. 5 $^{230}Th/^{232}Th$ vs. $^{238}U/^{232}Th$ activity ratios for the reference zircon materials and KBSI working standards. Note that all zircons demonstrate secular equilibrium and plot along the equiline. Symbols are the same as in Fig. 3

zircon, the activity ratios of $^{230}Th/^{232}Th$ and $^{238}U/^{232}Th$ were plotted on the equiline for all zircon reference materials and KBSI working standards. The LKZ-1 and BRZ-1 employed by the KBSI can be used to check the reliability of U–Th isotopic analyses for Quaternary zircons.

Additional files

Additional file 1: Table S1. SHRIMP Pb isotope data with calculated $^{232}Th/^{238}U$ ratios. (XLSX 50 kb)

Additional file 2: Table S2. U–Th isotopic composition of the zircons. (XLSX 87 kb)

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Authors' contributions
ACSC designed the research and wrote the manuscript. YJJ, SL, SJK, HJJ, and KY analyzed the data. All authors read and approved the final manuscript.

Competing interests
The authors declare that they have no competing interests.

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