Zircon M127 – a homogeneous reference material for SIMS U–Pb geochronology combined with Hf-, O-, and potentially Li-isotope analysis †

† This article is dedicated to Jan Košler (University of Bergen, Norway), who passed away in 2014 at the age of 49. Jan was involved in the initial characterization of zircon M127.

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Here we document a detailed analytical characterization of zircon M127, a homogeneous 12.7-carat gemstone ofrom Ratnapura, Sri Lanka. Zircon M127 has TIMS-determined mean U–Pb radiogenic isotopic ratios of 0.084743 ± 0.000027 for ^{206}Pb/^{238}U and 0.67676 ± 0.00023 for ^{207}Pb/^{235}U (weighted means; 2*s* uncertainties). Its ^{206}Pb/^{238}U age of 524.36 ± 0.16 Ma (95% confidence uncertainty) is concordant within the uncertainties of decay constants. The δ^{18}O value (determined by laser fluorination) is 8.26 ± 0.06 ‰ VSMOW (2s), and the mean ^{176}Hf/^{177}Hf ratio (determined by solution ICP–MS) is 0.282396 ± 0.000004 (2*s*). The SIMS-determined δ^7Li value is –0.6 ± 0.9 ‰ (2*s*), with a mean concentration of 1.0 ± 0.1 µg/g Li (2*s*). Zircon M127 contains ~923 µg/g U. The moderate degree of radiation damage corresponds well with the time-integrated self-irradiation dose of 1.82×10^{18} alpha events per gram. This observation, and the (U–Th)/He age of 426 ± 7 Ma (2*s*), which is typical of unheated Sri Lankan zircon, enable us to exclude any thermal treatment. Zircon M127 is proposed as reference material for the determination of zircon U–Pb ages by means of SIMS analysis in combination with hafnium and stable-isotope (oxygen and potentially also lithium) analysis.

Introduction

The development of the SIMS (secondary ion mass spectrometry) technique opened the world of high spatial resolution determination of U–Pb ages from polished zircon surfaces (e.g., Andersen and Hinthorne 1972; Compston *et al.* 1984; Williams 1998). The SIMS technique is a comparative method and the determination of accurate isotopic ratios in zircon requires calibration of the unknown results against the results of a well-characterized calibrant material measured under identical conditions during the same analytical session. In principle either a natural or synthetic reference material can be used as such a calibrant; unfortunately well-performing synthetic reference standards for the U–Pb analysis of zircon are not available. This is due to two main reasons. First, no synthesis technique is currently available that allows one to produce homogeneous Pb-bearing zircon crystals, owing to the exceptionally low partition coefficient of Pb in zircon. Second, glass reference materials cannot be used due to their different sputtering behavior under the oxygen-ion beam, compared to that of the zircon crystals to be analysed. Consequently, high-quality natural zircon reference materials must be used, and are therefore increasingly sought.

Any potentially suitable reference zircon needs to be characterized thoroughly in order to provide users with the best currently obtainable values, with regards to precision and accuracy, for the isotope ratios and concentrations of the calibrant material. Also, a well-performing reference zircon needs to be exceptionally homogeneous (Pidgeon 1997, Kennedy 2000), because each individual chip, and micro-areas within chips, of the reference material must have the chemical, physical and isotopic properties of the entire, previously characterized, bulk material. Another important characteristic that determines the quality of a reference material is its reliability during analysis. In U–Pb geochronology, higher count rates – in particular for the radiogenic Pb isotope, which is always lower in abundance than the parent U isotope – lead to better Poisson statistics. This may allow the operator to shorten the analysis time and/or to decrease the spot diameter without loss of precision. A suitable SIMS U–Pb reference should therefore contain sufficiently high concentrations of U and radiogenic Pb (whereas the concentration of non-radiogenic Pb should be negligible). However, higher levels of radiogenic Pb correlate with elevated radiation damage and this leads to an analytical difficulty, as the secondary ion sputtering behaviour under the ion beam may be different for the unknowns and reference if the structural states are sufficiently different. In searching for suitable U–Pb zircon references it is therefore advantageous to find gem-quality specimens that have some, but not too much radiation damage. Zircon M127 fulfils all of these important prerequisites. Zircon M127 contains notably higher levels of U and radiogenic Pb isotopes than most other SIMS references, resulting in a somewhat higher level of radiation damage. The degree of damage is however similar to that of zircon M257, whose damage still does not cause any identifiable matrix effects under the ion beam, which might bias the SIMS results (Nasdala et al. 2008).

The analytical characterization of zircon M127 began in 2006–2008, as this specimen was among a batch of 15 gemstones that were checked for their potential suitability as future SIMS references. After this early round of analyses, zircon M257 was preferentially selected as the first zircon to be characterized as a SIMS U–Pb reference because of its particularly large size (Nasdala *et al.* 2008), whereas zircon M127, in spite of its excellent characteristics, was not fully characterized because it was much smaller than M257. Although small amounts of M127 were used in several research projects (e.g. Mattinson *et al.* 2007; Váczi *et al.* 2009; Mattinson 2010; Guenthner *et al.* 2013; Lenz and Nasdala 2015), a major fraction is still available. It has subsequently been discovered that M127 is exceptionally useful for SIMS U–Pb geochronology, especially for studies that combine U–Pb with Hf and stable-isotope analysis [i.e. SIMS O-isotope and/or LA–ICP–MS (laser ablation inductively coupled plasma mass spectrometry)] using the same sample mounts. In this contribution we therefore summarize both early and recent analytical results characterizing zircon M127.

General characterization of zircon M127

General description and preparation

Zircon M127 was a brownish oval shaped gem (Figure 1a). The cut and faceted stone weighed 2.54 g (12.7 carats) and its longest dimension was 14.5 mm. The specimen came from the collection of the Institut für Edelsteinforschung Idar-Oberstein, Germany. It was originally found in a secondary placer deposit in the Ratnapura area, which belongs to the Highland Complex, Sri Lanka (Munasinghe and Dissanayake 1981; Kröner *et al.* 1994; Zoysa 2014).

Prior to preparation, thorough homogeneity checks of the original gemstone specimen were done by (i) inspection in white-light illumination under a high-resolution optical microscope and in an optical CL (cathodoluminescence) microscope, and by (ii) multiple laser spectroscopy analyses placed on different faces and under different sample orientations. The stone was found to be internally flawless, free of noticeable fractures, and uniform in colour. Neither inclusions nor growth zoning or other heterogeneities were observed. The initial Raman and laser-induced PL (photoluminescence) analyses indicated that zircon M127 has a uniform, moderate degree of radiation damage (Figure 2a) and uniform REE (rare earth element) emission patterns (Figure 2b).

The stone was cut in half along its longest dimension, and halves were cut into ~2.5 mm thick slices (Figure 1b), using a 200 µm, diamond-trimmed tungsten wire. Slices were then crushed in a steel-cylinder apparatus. The central slice pictured in Figure 1b (maximum dimension ~8 mm) was cut again very close to one of the large side planes, i.e. parallel to the initial cut direction. This was done to produce one particularly large, doubly polished thin section for homogeneity tests.

Chemical composition and homogeneity

Contents of major elements were measured by means of EPMA (electron probe micro-analyser) analysis. Multiple spots (n = 63) were measured across two large slices of M127, including the ~8 mm thin section. These wavelength-dispersive X-ray analyses were done using the Jeol 8900 RL EPMA system at Geowissenschaftliches Zentrum, Universität Göttingen, Germany. The accelerating voltage was 20 kV and the beam current was set at 80 nA. The electron beam was focused to a spot area 2 µm in diameter. Analyses of zircon M127 have been done in the same session as those of zircon M257, hence experimental details correspond to the detailed description in Nasdala *et al.* (2008). Counting times varied between 15 s peak and 2 × 5 s backgrounds for main elements (Zr–K_α, Si–K_α) up to 300 s peak and 2 × 150 s backgrounds for actinides (U–M_α and Th–M_α). Synthetic calibrant materials were used, including $ZrSiO_4$ (for Zr and Si), $HfSiO_4$ (for Hf), YAG (yttrium-aluminium garnet, for Y), Yb-glass (for Yb), apatite (for P), wollastonite (for Ca), Al_2O_3 (for Al), hematite (for Fe), $ThSiO_4$ (for Th), and UO_2 (for U). Data were processed using the CITZAF routine in the JEOL software, which is based on the $\phi(\rho Z)$ method (Armstrong 1991, 1995).

Prior to EPMA analysis, detailed imaging was performed using the large, 8 mm thin section as well as seven smaller chips embedded in epoxy sample mounts and polished. First, slices and chips were inspected under a research-grade optical microscope. Neither inclusions nor growth or other zoning were found; uniform interference colours were observed in cross-polarized light. Second, back-scattered electrons (BSE) and panchromatic CL imaging was done using the EPMA. All BSE and CL images revealed uniform greyscale intensity, and neither primary growth zoning nor other internal heterogeneities were observed. In view of the particularly high sensitivity of the CL technique to reveal internal textures (for a summary see Corfu *et al.* 2003), the apparent absence of any heterogeneity in all CL images obtained supports the internal homogeneity of zircon M127.

Trace-element analysis of zircon M127 by means of LA–ICP–MS was done in 2006 by Jan Košler (deceased), University of Bergen, Norway, as part of the analytical characterization of zircon M257. Analyses were done using a ThermoFinnigan Element2 single-collector double-focusing magnetic-sector system coupled to a NewWave/Merchantek UP213 laser. Analytical details and data reduction are described elsewhere (Nasdala *et al.* 2008). In addition, a fairly large amount of M127 (~300 mg) was subjected to a solution ICP–MS analysis at GEUS (Nationale Geologiske Undersøgelser for Danmark og Grønland), Copenhagen, Denmark. This was done in 2008, to determine reliably the U concentration of M127. Zircon M127 was then used to re-calibrate the U data obtained by EPMA for zircon M257 (for discussion and analytical details see Nasdala *et al.* 2008).

The EPMA and LA–ICP–MS results are presented in Table 1. Zircon M127 contains rather moderate levels of non-formula elements; only the hafnium-oxide content (1.50 % m/m) is in the percent range. The uranium concentration was determined by solution ICP–MS at ~923 µg/g U (Nasdala *et al.* 2008). This value is considerably higher than in most other zircon reference materials [with the exception of M257 whose U concentration (~840 µg/g) is at a similar level]. The Th/U weight ratio of zircon M127 is ~0.45. This ratio is not unusual for Sri Lankan zircon (Murakami *et al.* 1991; Nasdala *et al.* 2004); however it should be noted that the value of ~0.45 is much higher than $^1/_9$, which was postulated as typical "Sri Lankan" actinide-concentration ratio by Holland and Gottfried (1955). The homogeneity of the chemical composition, and in particular of the U and Th concentrations, was checked by two line scans across a large polished chip of zircon M127. Results obtained along a ~7.8 mm long traverse are plotted in Figure 3. No lateral chemical variations exceeding the analytical uncertainties were found.

The REE concentrations (Table 1) are typical of igneous zircon (Figure 4; compare Hoskin and Schaltegger 2003). Also, the REE pattern is typical of unaltered zircon from igneous crustal rocks (Hoskin and Schaltegger 2003). First, the HREE (heavy rare-earth elements) are enriched relative to the middle and light REE (Figure 4). Second , a positive Ce anomaly (Ce/Ce* = 15.4) and a negative europium anomaly (Eu/Eu* = 0.13) are observed. The former (i.e. HREE enrichment) is also evident in the PL spectrum (Figure 2b) where strong emissions are observed for the tri-valent Dy and Tm, and to a lesser extent Sm (Gaft *et al.* 2000; Lenz *et al.* 2015).

Finally, it is important to note that elemental species such as Ca, P, and Fe that are typically excluded or low in primary zircon but are often absorbed extensively during alteration events (Smith *et al.* 1991; Geisler *et al.* 2003; Pérez-Soba *et al.* 2007; Nasdala *et al.* 2010), have low concentrations in zircon M127 (Table 1). This observation shows that M127 has not undergone intense weathering or other post-crystallization chemical alteration.

Structural state and radiation damage

Natural zircon may have appreciable structural damage created by alpha-decay of U and Th and their unstable daughter nuclei. The accumulation of this damage leads to systematic changes of physical parameters, which in turn can be used to estimate the degree of radiation damage sustained by an unknown zircon sample. We have therefore applied a number of analytical techniques for characterizing the structural state of M127 (by determining specific gravity, unit-cell parameters, Raman spectral parameters, and sharpness of electron-diffraction maxima). This was done to check the degree to which the observed deviation from the ideal structural state corresponds, within the well-known "Sri Lankan trend", to the calculated self-irradiation. Obvious mismatches, in turn, could unravel structural recovery as for instance caused by post-growth alteration or synthetic thermal gem enhancement.

The self-irradiation causing the radiation damage is quantified commonly by the alpha dose (D_α), i.e. the time-integrated number of alpha-decay events experienced by the zircon since primary growth (more precisely, since the time of closure of the U–Pb system). This parameter is calculated according to (Murakami *et al.* 1991, Nasdala *et al.* 2001)

$$D_\alpha = \frac{6 \cdot c_{Th} \cdot N_A}{10^6 \cdot M_{232}} \cdot \left(e^{\lambda_{232}\, t} - 1\right) + \frac{7 \cdot c_U \cdot 0.0072 \cdot N_A}{10^6 \cdot M_{235}} \cdot \left(e^{\lambda_{235}\, t} - 1\right) + \frac{8 \cdot c_U \cdot 0.9928 \cdot N_A}{10^6 \cdot M_{238}} \cdot \left(e^{\lambda_{238}\, t} - 1\right) \qquad (1)$$

where c_U and c_{Th} = U and Th concentrations in µg/g (Table 1); N_A = Avogadro's Number; M_{238}, M_{235}, and M_{232}, = molecular weights of isotopes; λ_{238}, λ_{235}, and λ_{232} = decay constants of ^{238}U, ^{235}U, and ^{232}Th; and t = zircon U–Pb age (here 524.3 Ma; see below). It should be noted that the common use of the U–Pb age in calculating D_α adds slight uncertainty, as this practice is based on the – often incorrect – assumption that the closure of structural recovery by annealing corresponds exactly with U–Pb system closure. For natural zircon, D_α actually is not a "measure" of the structural damage present in a given natural zircon sample. Rather, D_α quantifies the total self-irradiation since the time of closure of the U–Pb system. How much of the damage has been accumulated or has been thermally annealed, depends on the thermal history of the sample. Nasdala *et al.* (2001; 2004) and Palenik *et al.* (2003) found that the Ratnapura, Sri Lanka, gem-zircon suite is not as radiation-damaged as expected given the U and Th concentrations and ages; only about 55 % of the damage was retained. This is assigned to the particular geological history of the area (involving slow cooling prior to Ordovician uplift, and perhaps a re-heating event; Nasdala *et al.* 2004). In summary, radiation damage cannot be compared among samples of different origin by the simple evaluation of their calculated D_α values. However, internal comparison is straightforward with Sri Lankan zircon gems, because – provided no gem enhancement involving elevated temperatures was applied – they all show uniform trends of parameter changes depending on D_α (Nasdala *et al.* 2004). The calculated value of $D_\alpha = 1.82 \times 10^{18}$ events per gram for zircon M127 is rather moderate. It is well below the first percolation point (for Sri Lanka zircon determined at $\sim 3.5 \times 10^{18}$ α/g; Salje *et al.* 1999) that is characterized by the beginning of three-dimensional inter-connection of amorphous clusters in the crystalline matrix. The nearly complete transformation of Sri Lankan zircon to the amorphous state requires self-irradiation doses of $\sim 10^{19}$ α/g (Salje *et al.* 1999; Nasdala *et al.* 2002). The calculated alpha dose of 1.82×10^{18} events hence suggests a notable but still moderate level of radioactive self-irradiation.

The specific gravity of zircon M127 was determined at 4.625 ± 0.005 g/cm^3. This was done prior to preparation, by repeated weighing of the original stone in air and in distilled water. The gravity of M127 is ~2% lower than that of crystalline zircon (>4.7 g/cm^3) but well above than that of metamict zircon (<4 g/cm^3; e.g. Sahama 1981; Nasdala *et al.* 2002), indicating a notable, but still moderate degree of self-irradiation induced volume expansion. As zircon M127 has a "regular" chemical composition with generally low amounts of non-formula elements, and as it is free of inclusions, any chemical or textural effects on the sample's specific gravity must be very minor. The moderately decreased specific gravity is therefore assigned solely to the moderate degree of radiation damage present.

Unit-cell dimensions of zircon M127 were determined from four 50–100 μm sized chips, by obtaining ten frames each on a Bruker Nonius KappaCCD single-crystal X-ray diffractometer. Analyses were done with Mo–K_α radiation. The distance between chip and detector was 35 mm. The measurement time per frame (width 2°) was 3 seconds. The dimensions of the tetragonal unit-cell were determined at a_0 = 6.6408(4) Å and c_0 = 6.0367(5) Å, resulting in a unit-cell volume of V = 266.22(4) Å^3. Compared to well-crystallized zircon, zircon M127 is characterized by expansion of the unit cell of ~2%. First, this corresponds very well with the ~2% decrease of the specific gravity (cf. above). Second, the ~2% cell expansion characterizes the material as moderately radiation-damaged (Holland and Gottfried 1955; Murakami *et al.* 1991). Note that the observed expansion of parameters a_0 and c_0 is rather "homogeneous" (Holland and Gottfried 1955; compare also Figure 6A in Nasdala *et al.* 2004). Partially annealed zircon, in contrast, is typically characterized by an apparent $a_0 - c_0$ mismatch, caused by preferential recovery of the original expansion along the crystallographic *a* axis (Weber 1990; 1993).

The degree of radiation damage was further estimated from the full width at half maximum (FWHM) of the main Raman band of zircon [$\nu_3(SiO_4)$ vibrational mode; Nasdala *et al.* 1995; 2001]. Spectra were obtained with He–Ne 632.8 nm excitation (8 mW at the sample surface) by means of a Horiba LabRam HR Evolution system (focal length 800 mm). A diffraction grating with 1800 grooves/mm was placed in the optical pathway, resulting in a spectral resolution of ~0.8 cm^{-1}. The system's wavenumber accuracy is better than 0.5 cm^{-1}; wavenumber calibration was done using the Rayleigh line and emission lines of a Kr lamp. Raman spectra were corrected for background and fitted assuming Lorentzian–Gaussian band shapes. Real FWHM values were calculated by correcting measured FWHMs for the instrumental profile function of the spectrometer (Dijkman and van der Maas 1976; Nasdala *et al.* 2001; Váczi 2014). The Raman spectrum of zircon M127 is moderately broadened but bands are still very symmetric (Figure 2a). The FWHM of the $\nu_3(SiO_4)$ band (here observed at 999.8 ± 0.5 cm^{-1}) was determined at 14.0 ± 0.4 cm^{-1}, which indicates significant but still moderate radiation damage. Raman line-scans did not reveal lateral variations of Raman parameters (Figure 3), implying that the moderate radiation damage in zircon M127 is homogeneous.

Electron diffraction was performed by means of a FEI Tecnai G^2 F20 X-Twin TEM (transmission electron microscope) system at Helmholtz-Zentrum Potsdam. The TEM system was operated at 200 kV and 0.5 nA beam current. For TEM analysis, thin foils were cut out of polished mounts containing oriented zircon chips using a FEI FIB200 (for details of the focused ion beam technique see Wirth 2004). An electron diffraction pattern of zircon M127 is shown in Figure 6a. The diffraction pattern of M127 appears somewhat “blurred” when compared with that of crystalline zircon, whereas no “amorphous” diffraction ring (which is typical of highly radiation-damaged zircon; Nasdala *et al.* 2004) is observed. The former observation is visualized in Figure 6b, which shows the diffraction intensity along the line marked in Figure 6a. The intensity profile was extracted using the ImageJ software (Schneider *et al.* 2012). The analogous intensity profile of a nearly undamaged zircon (sample Rata described in Lenz and Nasdala 2015) obtained with the same TEM system under identical experimental conditions shows notably narrower diffraction maxima (Figure 6b).

All of our results indicate that the structure of zircon M127 is moderately damaged due exclusively to radioactive self-irradiation over geologic periods of time. This conclusion is supported by the observations that the specific gravity, the unit-cell volume, and the Raman band broadening correspond well, within the “Sri Lankan” trends of parameter changes, with an alpha dose of 1.82×10^{18} g^{-1}. The degree of damage is significantly higher than most other zircon references, such as Penglai (for sample description see Li *et al.* 2010), 91500 (Wiedenbeck *et al.* 2004), and CZ3 (Nasdala *et al.* 2004; for sample description see Pidgeon 1997). The degree of radiation damage in M127 is however still moderate, very similar to the structural disorder of zircon M257 (Norberg 2007; Nasdala *et al.* 2008). As M257 does not show any significant matrix effects (such as preferred sputtering of Pb from highly radiation-damaged zircon; e.g. McLaren *et al.* 1994; Nasdala *et al.* 2002), such problems are not to be expected for M127 also.

Isotopic composition of zircon M127

U–Pb age determination by TIMS

The U–Pb age of zircon M127 was determined by thermal ionization mass spectrometry (TIMS) analysis in the geochronology laboratories of the University of California at Santa Barbara, USA, and University of Oslo, Norway. Results of chemical abrasion (CA) TIMS analyses of one 1.437 mg chip at the University of California have already been published elsewhere (Mattinson 2010).

At the University of Oslo, one 0.646 mg chip of M127 was subjected to isotope dilution (ID) TIMS analysis. The chip was cleaned with HNO_3, H_2O and acetone, then weighed on a microbalance, and transferred to a Krogh-type bomb (see Krogh 1973), to which a measured amount of ^{202}Pb–^{205}Pb–^{235}U spike was added. The spike was evaporated before adding a mixture of HF and HNO_3 (12:1). The bomb was placed in an oven for 5 days at 190 °C. The solution was then evaporated to dryness and re-dissolved in 3N HCl overnight at 190 °C. The solution was then split with a pipette onto 4 columns with anion exchange resin to separate out Pb and U. The latter were then loaded on zone-refined-Re filaments together with Si-gel and H_3PO_4 and measured in a MAT262 mass spectrometer. The main Pb and U peaks were measured on multiple faraday cups in static mode, and the $^{207}Pb/^{204}Pb$ ratio was measured by peak-jumping on an electron multiplier. Other details of the procedure can be found in Corfu (2004). The spike composition was calibrated against the synthetic ET100 solution (Condon *et al.* 2008) provided by the EARTHTIME initiative (http://www.earth-time.org).

The ^{238}U decay constant of Jaffey *et al.* (1971; 1.55125×10^{-10} a^{-1}) and the re-revised ^{235}U decay constant of Mattinson (2010; 9.8571×10^{-10} a^{-1}), which is marginally higher than the revised value of Schoene *et al.* (2006; 9.8569×10^{-10} a^{-1}), were used for converting isotopic ratios into ages and for plotting the results. Age calculations and the preparation of the Concordia plot were done using the Isoplot program (Ludwig 2003). Note that isotopic ratios are reported as measured (i.e., without Th fractionation correction). The analytical uncertainties reported include quadratically propagated measurement errors, a minimum reproducibility term of ± 0.06 % /amu on the fractionation corrections for Pb and U (based on regular runs of standard solutions), blank corrections of 2 ± 1 pg Pb and 0.1 ± 0.05 pg U, and uncertainties of 2% on the 206Pb/204Pb and 1% on the 207Pb/204Pb compositions of the blank.

The U–Pb results obtained in the University of Oslo laboratory are summarized in Table 2, and a Concordia plot including both these results and the results of Mattinson (2010) is presented in Figure 7. From all 20 TIMS analyses, a weighted mean ^{206}Pb/^{238}U ratio for zircon M127 of 0.084743 $\pm$ 0.000027 (2*s*) was calculated, which corresponds to a ^{206}Pb/^{238}U age of 524.36 $\pm$ 0.16 Ma (uncertainty quoted at the 95 % confidence level). The weighted mean ^{207}Pb/^{235}U isotopic ratio was calculated at 0.67676 $\pm$ 0.00023 (2*s*). The mean Concordia age calculated is 524.37 $\pm$ 0.46 Ma (95 % confidence; errors of decay constants included). The U–Pb system of zircon M127 is well concordant within the uncertainties of the decay constants (for further discussion see Schoene *et al.* 2006; Schmitz and Schoene 2007; Mattinson 2010). We recommend the above mean isotopic ratios and ^{206}Pb/^{238}U age to be used when applying zircon M127 as reference material in SIMS U–Pb dating.

Hafnium-isotope analysis by solution ICP–MS

Hafnium concentrations, and^{176}Hf/^{177}Hf and^{176}Lu/^{177}Hf ratios, were measured by solution isotope-dilution analysis. Three multiple collector (MC) ICP–MS systems at different laboratories were uase: the Radiogenic Isotope and Geochronology Laboratory (RIGL) at Washington State University (WSU), Pullman; the Institute of Geology and Geophysics, Chinese Academy of Sciences (IGGCAS), Beijing; and the joint Köln-Bonn laboratory, Germany. In order to check for the internal homogeneity of Hf isotopes within zircon M127, each of the three laboratories was provided with five small samples (1–3 mg per sample consisting of one, two, or three individual chips). The five samples were recovered from the five main slices of the original gemstone (see Figure 1b; one sample per slice).

In the RIGL facility, each fragment was separately dissolved in a mixture of concentrated HF and HNO_3 in pre-cleaned 0.5 ml Savillex perfluoroalkoxy screw-top vials. The vials were loosely capped and placed in Teflon liner in a large-capacity Parr dissolution bomb to which ~5 ml of concentrated HF were added to provide vapour pressure within the bomb. The sealed bomb was placed in an oven at 250 °C for 48 hours. Following dissolution, the contents of each bomb were transferred to at 7 ml Savillex beaker, dried, and fluxed overnight in a mixture of 2.5M HCl/H_3BO_3 to convert to chlorides and minimize production of fluoride species. Samples were again dried and redissolved in 1ml 1M HCl plus 0.1M HF (precisely weighed). From this solution an aliquot for analysis was removed and added to a clean 7 ml Savillex beaker to which a zircon-specific mixed ^{176}Lu–^{180}Hf tracer solution was added. Further details of sample preparation, column chemistry, and Hf and Lu mass spectrometry are described in Vervoort *et al.* (2004) and Goodge and Vervoort (2006). The purified Hf of samples (and calibrants) were run as 50 ppb solutions and introduced to the RIGL ThermoFinnigan Neptune MC–ICP–MS using an Aridus desolvating nebuliser. Mass fractionation was internally corrected using $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$ and all sample analyses were normalized using the Hf isotope reference material JMC 475 (Patchett and Tatsumoto 1980; with a presently accepted value of $^{176}\text{Hf}/^{177}\text{Hf} = 0.282160$). Five analyses of JMC 475 were interspersed between samples and yielded a mean $^{176}\text{Hf}/^{177}\text{Hf} = 0.282132 \pm 0.000004$ (2*s*; n = 5).

For analyses at the IGGCAS, single chips of zircon M127 were weighed and dissolved in HF-HNO_3 in 3 ml vials placed in high-pressure bombs at 210 °C for one week. Afterwards the samples were dried down and re-dissolved in 3 M HCl. Each sample solution was then split. About 20% was spiked with a mixed ^{176}Lu and ^{180}Hf tracer for determining the Lu and Hf concentrations. The spike solution used was calibrated beforehand against a standard solution made from pure metals (Yang *et al.*, 2010). It was tested on several international calibrants (BCR-2, W-2) and yielded results corresponding to reference values (Münker *et al.* 2001). The remaining ~80% of each initial sample solution was used to determine the Hf isotopic composition. The chemical purification procedure described in Nebel-Jacobsen *et al.* (2005) and Morel *et al.* (2008) was applied. Isotope measurements were performed on a Thermo Scientific Fisher Neptune MC–ICP–MS following the protocol listed in Yang *et al.* (2010). Instrumental mass bias was corrected offline using the exponential law and $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$. For possible interferences of ^{176}Yb and

^{176}Lu on ^{176}Hf, isotopes ^{173}Yb and ^{175}Lu were monitored and corrected for, applying ^{176}Lu/^{175}Lu = 0.02655 and ^{176}Yb/^{173} Yb = 0.79631 (Vervoort *et al.* 2004). The ^{176}Hf/^{177}Hf results for M127 are reported relative to a value of 0.282160 for JMC 475.

The procedure in the Köln-Bonn laboratory was broadly similar. Lutetium and Hf isotope measurements were performed using a Thermo Neptune MC–ICP–MS and a procedure after Nebel Jacobsen *et al.* (2005), employing a mixed ^{176}Lu-^{180}Hf tracer specifically designed for zircon. Results for ^{176}Hf/^{177}Hf are given relative to a value of 0.282160 for the Münster AMEs standard that is isotopically indistinguishable from JMC 475.

All results are presented in Table 3 and Figure 8. They indicate that zircon M127 is fairly uniform in Hf concentration and ^{176}Hf/^{177}Hf ratio. Considering all eighteen individual analyses (but without consideration of re-analyses which are merely reported for completeness), the unweighted mean ^{176}Hf/^{177}Hf ratio is determined at 0.282396 ± 0.000004 (2*s*), which we suggest be used as “working value”. The ^{176}Hf/^{177}Hf ratios determined in Köln may seem to be slightly higher, compared to data produced in the two other laboratories. Differences among the three laboratories are, however, within analytical uncertainties and hence cannot be considered systematic.

Hafnium-isotope analysis by LA–ICP–MS

To check the internal homogeneity of the ^{176}Hf/^{177}Hf ratio, 130 individual LA–ICP–MS analyses were performed using the Neptune MC–ICP–MS system of the Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing. The JMC 475 standard solution with 200 ng/g Hf was used for evaluating the reproducibility and accuracy of the instrument prior to laser ablation analyses. During laser ablation analyses of Hf isotopes, the isobaric interference of ^{176}Lu on ^{176}Hf is relatively small compared to the interference of ^{176}Yb on ^{176}Hf, which must be carefully corrected since the contribution of ^{176}Yb to ^{176}Hf can profoundly affect the accuracy of the measured ^{176}Hf/^{177}Hf ratio (e.g. Fisher *et al.*, 2011). Here, the mean ^{173}Yb/^{171}Yb ratio of all individual spot analyses was used to calculate the fractionation coefficient (β_{Yb}), and then to calculate the contribution of ^{176}Yb to ^{176}Hf using an isotopic ratio of ^{176}Yb/^{172}Yb = 0.5887 (Wu *et al.*, 2006). Reference zircon 91500 was used for external correction. During the analytical sessions, the ^{176}Hf/^{177}Hf value of 91500 was 0.282294 ± 0.000039 (2*s*, n = 436), which was adjusted to 0.282305 (correction of 0.000011), a reference value recommended for 91500 (Wu *et al.*, 2006). During data acquisition, analyses of GJ-1 as an unknown yielded a weighted ^{176}Hf/^{177}Hf ratio of 0.282018 ± 0.000048 (2*s*, n = 116), within error the recommended value (Morel *et al.*, 2008). Data are not presented in detail here; they are available online (Table S1; see link given at the end of the paper). Analyses did not yield any significant variations within and among chips, underlining the homogeneity of zircon M127; the mean ^{176}Hf/^{177}Hf value of all 130 analyses was 0.282392 ± 0.000031 (2*s*).

Hafnium-isotope analysis – Discussion

One puzzling observation related to the solution isotope-dilution results (Table 4) is the diversity of Lu concentrations (and, connected with that, of ^{176}Lu/^{177}Hf ratios). Fairly uniform Lu concentrations were measured in Köln-Bonn whereas the results of the other two laboratories show a comparably large scatter. These differences could point to sample heterogeneity. Recall, however, that all three laboratories received corresponding fragments recovered from the same areas in the five gemstone slices, so samples may be assumed to represent a nearly identical set. This implies that sample heterogeneity is most likely not the cause of scattered Lu concentrations, because otherwise similar scatters would be expected to have been found by all three laboratories. Possible other reasons for Lu-data variations include difficulties relating spiking sub-aliquots some time period after dissolution (as done in the RIGL facility and at the IGGCAS). This practice may have resulted in differing behavior of Hf and Lu during the chemical treatment. For instance, it may be speculated that REE-fluoride complexes might have formed that prevented the spike and sample from equilibrating completely with respect to Lu. We therefore cannot be sure the variations in ^{176}Lu/^{177}Hf observed by the RIGL and IGGCAS laboratories are real. Rather we assess the Köln-Bonn results as reliable, which suggest the true ^{176}Lu/^{177}Hf is homogeneous. Thus we suggest the Köln-Bonn result of ^{176}Lu/^{177}Hf = 0.0006146 ± 0.0000130 (2*s*) be used as “working value” in Hf-Lu studies. It is however clear that the possibility of interlab variations needs to be sorted out in future analytical efforts.

Oxygen-isotope analysis by laser fluorination

Sixteen fragments of zircon M127 (weights in the range 1.83 to 3.98 mg) were analyzed for oxygen-isotope ratios at the University of Wisconsin, Madison. Analyses were done in four separate sessions. The routine procedure at UW is to treat zircons in cold HF for 8 hours before laser fluorination and analysis for oxygen isotope ratios. This procedure removes non-zircon impurities and also dissolves intra-crystalline domains that are severely radiation damaged and thus susceptible to post crystallization alteration of δ^{18}O (Valley et al. 2005, 2015); it has been shown not to affect δ^{18}O of undamaged zircon (Valley 2003). Two chips that were HF-treated for the December 10, 2015, session lost 14 % m/m, which is more than expected for such clean fragments. The fragments were then pre-treated at room temperature in a BrF_5 atmosphere for 12 hours and each was heated in the presence of fresh BrF_5, using an infrared laser (λ = 10.6 µm). The evolved O_2 gas was cryogenically purified, converted to CO_2, and analyzed by dual-inlet gas-source mass spectrometer as described by Valley *et al.* (1995). Garnet reference UWG–2 (Valley *et al.* 1995) was analyzed four to six times in each analysis session of M127. The δ^{18}O values obtained for M127 were normalized to the recommended value of 5.80 ‰ VSMOW (Vienna Standard Mean Ocean Water) for UWG–2 (Valley *et al.* 1995). Nine analyses of untreated fragments yield a value of $\delta^{18}O = 8.26 \pm 0.06$ ‰ VSMOW for zircon M127 (Table 4) Whereas seven analyses of HF-treated fragments yield a value of $\delta^{18}O = 8.49 \pm 0.05$ ‰ VSMOW (2*s*; unweighted means). These data are systematically higher for the HF-treated zircon grains by an average of 0.23 ‰.

The difference obtained by laser fluorination analysis of M127 between untreated and HF-treated zircon is small and comparable to the analytical precision obtained by in situ SIMS analysis. We interpret the HF-treated value as most reliably reflecting the original composition of the M127 zircon. If the HF-treatment has selectively removed zircon domains that are slightly higher in δ^{18}O, then the untreated value for δ^{18}O best represents the bulk composition of the M127 crystal. Any higher δ^{18}O domains will be included in a SIMS analysis and if present, the homogeneity of SIMS analyses suggests that higher δ^{18}O domains are sub-micron and randomly distributed. Thus the untreated value $\delta^{18}O = 8.26 \pm 0.06$ ‰ VSMOW(2*s*) is recommended for calibration of SIMS analysis with the caveat that there are currently no estimates for the effect of radiation damage on the instrument bias (or instrumental mass fractionation). Although, it is usually recommended to use a zircon with less radiation damage for analysis of oxygen isotope ratios, such as KIM-5 (Valley 2003), M127 might be a better reference for samples with comparable amounts of damage.

Oxygen-isotope analysis by SIMS

To check the homogeneity of the oxygen-isotope ratio, SIMS analyses were done at Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, and at the Helmholtz-Zentrum Potsdam (GFZ). In Beijing, a total of 30 point analyses placed in six chips originating from five slices of the original stone were performed using a Cameca IMS 1280. The primary Cs^+ beam was accelerated at 10 kV, with a current of 1.2 nA. The spot size was about 20 µm in diameter (10µm beam diameter plus 10µm raster). Low-energy electron flooding was used to compensate for sample charging during analysis. Secondary ions were extracted with a -10 kV potential. Oxygen isotopes were measured in multi-collection mode with two off-axis Faraday cups. Each analysis consisted of 20 cycles per 4 s counting time. The instrumental mass fractionation factor was corrected using the Penglai zircon reference (with $\delta^{18}O$ = 5.3 ‰ VSMOW; see Li *et al.*, 2010). Analytical repeatability was better than 0.4 ‰ (2*s*) for $\delta^{18}O$. Calibrated against the Penglai reference, the data set yielded a mean of $\delta^{18}O$ = 8.34 ± 0.34 ‰ VSMOW (2*s*).

The Potsdam Cameca IMS 1280–HR performed a total of 41 point analyses on a total of five chips. These five chips were recovered from the five main slices of the original gemstone (see Figure 1b; one chip per slice). These analyses employed a ~2.5 nA, mass filtered $^{133}Cs^+$ beam with a Gaussian density distribution, which was focussed to a ~5 µm diameter on the polished sample surface. The total impact energy of the Cs^+ ions was 20 keV. Each analysis was preceded by 20 µm x 20 µm rastered pre-sputter for 60 s. The total pressure in the sample chamber was 1×10^{-7} Pa at the time of analysis. Charge compensation was achieved with low-energy, normal-incidence electron flooding using a current of ~1 µA. All analyses were conducted in duplicate in sequence with an offset between the two analytical locations of 50 µm, to assess the short-term stability of our technique. In order to suppress within-run drift in the isotope ratio, a 10 µm x 10 µm raster was employed during data collection; thus assuring a crater geometry with a flat bottom.

Secondary ions were accelerated by a -10 kV potential applied to the sample holder. The mass spectrometer was operated in static multi-collection mode, with the $^{16}O^{-}$ being collected in the L2' Faraday cup and the $^{18}O^{-}$ signal being collected in the H2' Faraday cup; the amplifier system employed thermally stabilized 10^{10} Ω and 10^{11} Ω amplifiers, respectively. The mass resolution of the instrument was determined to be $M/\Delta M \approx 1670$ on the ^{16}O mass station and $M/\Delta M \approx 1970$ on the ^{18}O mass station. This is effectively full transmission for the tool and fully suffices to eliminate both the $^{16}O^{1}H_2$ and the $^{16}O^{2}H$ isobaric interferences from the ^{18}O mass station. A single analysis consisted of 20 integrations of 4 s each. Using white light profilometry, we determined the test portion mass for our analyses was equivalent to circa 150 picograms. Data for M127 were corrected for instrumental mass fractionation using the 91500 reference (with $\delta^{18}O = 9.86$ ‰ VSMOW; see Wiedenbeck *et al.*, 2004). Analysis on M127 yielded a mean of $\delta^{18}O = 7.96 \pm 0.20$ ‰ VSMOW (2*s*) for 41 determinations on the five fragments, meaning this data set is consistent with being a homogeneous population .

Data are available online (Table S2; see link given at the end of the paper). No systematic variations or "outliers" were found among the 30 analyses in the Beijing lab. Among the 41 analyses in Potsdam, two measurements in a single area gave results 0.25 ‰ lighter than the remainder of the population, and suspiciously these were the first data acquired during our analytical series. Discarding these two, seemingly discrepant values yields an analytical repeatability of ± 0.16‰ (2*s*) on n = 39 determinations, indicating that M127 is at least as homogeneous as the 91500 zircon, which we presume to be homogeneous in its $\delta^{18}O$ content. We therefore conclude that M127 is well suited for use as a reference in SIMS studies that correlate U–Pb age and $\delta^{18}O$ of unknown zircon samples (see Cavosie *et al.* 2005; Valley *et al.* 2005).

Oxygen-isotope analysis – Discussion

Unlike the homogeneous Sri Lanka zircon references CZ3 (Cavosie *et al.* 2011) and M257 (Nasdala *et al.* 2008), the $\delta^{18}O$ value determined for zircon M127 is not remarkably high and hence does not fall outside the range typical of igneous zircon (Figure 9). Unlike CZ3 and M257, the oxygen-isotope ratio of M127 does not provide a clear indication of the formation environment (whereas the particularly high δ^{18}O values of some Sri Lanka zircon samples are presumed to indicate formation in a marble or Ca-silicate metamorphic rock; Cavosie *et al.* 2011). We can therefore merely speculate that the remarkable homogeneity (i.e. absence of all signs of “igneous” growth zoning) of zircon M127 might be explained by (i) metamorphic formation that however did not result in a very high δ^{18}O value, or (ii) igneous formation that must have resulted in the formation in particularly thick growth zones. As zircon M127 was derived from a gem gravel, with the original host rock being unknown, the formation environment remains uncertain.

Lithium-isotope analysis by SIMS

Lithium-isotope analyses were performed using the Cameca IMS–1280 HR at the Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing. A detailed description of the analytical procedure can be found in Li *et al.* (2011); therefore only a brief description is given here. The primary O^- ion beam was accelerated at -13 kV, with a beam current of 28 nA. The beam was focused to an elliptical spot *ca.* 20–30 μm in size. Positive secondary ions were extracted with a 10 kV potential. A single ion-counting electron multiplier (EM) was used as the detection device. The mass resolution was 1300 (at 10 % peak height). Each measurement consisted of 80 cycles, with a total analytical time of 20 min. The M257 zircon reference (Li = 0.86 μg/g and δ^7Li = 2.1 ± 1.0 ‰; see Li *et al.* 2011) was used as calibrant material.

A total of 55 analyses were performed on 6 fragments of M127 that were embedded in a polished epoxy sample mount. The mean Li concentration of zircon M127 is 1.0 ± 0.1 μg/g (2*s*), and the mean δ^7Li value was determined at –0.6 ± 0.9 ‰ (2*s*). This can initially be used as a working value for M127, though there is an urgent need for further characterization of this system using other, independent analytical methods (i.e., solution ICP–MS). In view of the low δ^7Li variability observed in the present study, M127 may be a useful reference material in SIMS studies that use Li as a petrogenetic tracer (Ushikubo *et al.* 2008; Li *et al.* 2011; Bouvier *et al.* 2012).

(U–Th)/He analysis

The retention of the radiogenic ^{4}He was evaluated by (U–Th)/He analyses at the University of Arizona, Tucson. Our experimental details are described elsewhere (Nasdala *et al.* 2004; Reiners 2005), and our results are presented in Table 5. Analyses on chips M127zA and M127zB were done in 2006, in the course of the analytical characterization of zircon M257; the other chips were analyzed in 2015. All five analyses yielded a weighted mean He age of 426 ± 7 Ma (2*s*) for zircon M127. This value is well within the range of typical helium ages for unheated zircon samples from Sri Lanka (Hurley 1954, Nasdala *et al.* 2004). It also confirms that zircon M127 has never been subjected to heat treatment, as this would have caused near total loss of radiogenic helium (Reiners *et al.* 2005).

Conclusions

Zircon M127 is a suitable reference material for the SIMS U–Pb analysis of unknown zircon samples, especially in studies that subject the same sample mounts to the additional analysis of oxygen (SIMS), lithium (SIMS), or hafnium isotopes (LA–ICP–MS). Zircon M127 is homogeneous in terms of chemical and isotopic composition and has a remarkably concordant U–Pb isotopic system. Therefore M127 is suitable for calibration, quality control, and method validation purposes for analytical techniques working at the low- to sub-nanogram sampling scale, and its broad acceptance by the SIMS community would greatly improve the metrological traceability between facilities. A particularly advantageous property of M127 is the fairly high uranium concentration of ~923 µg/g (and correspondingly high levels of radiogenic Pb), which will result in exceptionally high count rates and good counting statistics for SIMS analyses.

The material will in future be used and distributed by the Beijing SHRIMP Centre, Institute of Geology, Chinese Academy of Geological Sciences (contact: liudunyi@bjshrimp.cn). Zircon M127 is reserved as SIMS reference. In particular, it will not be provided for only U–Pb geochronology in LA–ICP–MS laboratories. Despite this decision, it will of course be possible, and worthwhile, to subject re-polished SIMS mounts containing M127 to subsequent analyses by means of LA–ICP–MS, including Hf-isotope or LASS (laser ablation split stream method for combined U–Pb and Hf analyses; Fisher *et al.* 2014). This decision was made to reduce the consumption of zircon M127 to a minimum and ensure that the material will be available for SIMS work for several more years.

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Supporting information

The following supporting information is available online:

Table S1. Results of a total of 130 Hf-isotope analyses by LA–ICP–MS (six analytical sessions 2009–2013) at the Chinese Academy of Sciences, Beijing

Table S2. Results of O-isotope analyses by SIMS at the Chinese Academy of Sciences, Beijing, and the Helmholtz-Zentrum Potsdam (GFZ)

This material is available as part of the online article from: …

Table 1.

Chemical composition of zircon M127 (EPMA, University of Göttingen; LA–ICP–MS, University of Bergen)

Table 2.

Results of ID–TIMS U–Pb analyses of M127 (University of Oslo)

Table 3.

Results of Hf-isotope analyses by solution ICP–MS

Table 4.

Oxygen isotope analyses by laser fluorination (University of Wisconsin at Madison)

Table 5.

(U–Th)/He age of M127 (University of Arizona at Tucson)

Table 1.

Chemical composition of zircon M127 (EPMA, University of Göttingen; LA–ICP–MS, University of Bergen)

EPMA results (n = 63)*		LA–ICP–MS results (n = 24)					
Oxide	Concentration [% m/m]**	Element	Isotope measured	Concentration [µg/g]**	Element	Isotope measured	Concentration [µg/g]**
SiO_2	32.6 ± 0.2	Y	89	785 ± 55	Dy	163	67.1 ± 2.0
P_2O_5	0.057 ± 0.006	Nb	93	1.61 ± 0.45	Ho	165	25.7 ± 1.6
Y_2O_3	0.109 ± 0.012	La	139	0.35 ± 0.17	Er	166	121 ± 9
ZrO_2	65.9 ± 0.3	Ce	140	16.9 ± 0.8	Tm	169	28.0 ± 2.1
Yb_2O_3	0.028 ± 0.012	Pr	141	0.18 ± 0.07	Yb	172	286 ± 19
HfO_2	1.50 ± 0.02	Nd	146	1.95 ± 0.56	Lu	175	53.7 ± 1.8
ThO_2	0.046 ± 0.006	Sm	147	3.95 ± 0.65	Hf	178	12400 ± 500
UO_2	0.099 ± 0.008	Eu	153	0.39 ± 0.13	Pb	206	78.4 ± 4.2
Total	100.4 ± 0.4	Gd	157	15.6 ± 1.5	Th	232	413 ± 17
		Tb	159	5.55 ± 0.36	U	238	896 ± 36***

* Al_2O_3, CaO, and FeO were not detected or averages were below 0.005 % m/m.

** Quoted uncertainties are 2*s*.

*** The U concentration was determined by solution ICP–MS at ~923 µg/g (Nasdala et al. 2008); which is suggested to be used as "working value".

Table 2.
Results of ID–TIMS U–Pb analyses of M127 (University of Oslo)

Analysis number	Pb [µg/g]	U [µg/g]	Th/U	total common Pb [pg]	^{207}Pb/^{235}U	2*s* (abs.)	^{207}Pb/^{235}U age [Ma]	^{206}Pb/^{238}U	2*s* (abs.)	^{206}Pb/^{238}U age [Ma]	ρ (err. corr.)	^{207}Pb/^{206}Pb	2*s* (abs.)	Discordance [%]
153/53A	72	817	0.45	7.5	0.67763	0.00201	524.88 ± 1.22	0.084856	0.00023	525.04 ± 1.38	0.98	0.057917	0.00004	-0.2
153/53B	72	816	0.45	8.6	0.67689	0.00271	524.44 ± 1.64	0.084798	0.00031	524.70 ± 1.86	0.98	0.057894	0.00005	-0.3
153/53C	72	816	0.45	9.8	0.67838	0.00275	525.34 ± 1.66	0.084956	0.00032	525.64 ± 1.88	0.97	0.057914	0.00006	-0.3
153/53D	72	817	0.46	8.7	0.67785	0.00283	525.02 ± 1.71	0.084876	0.00032	525.16 ± 1.90	0.97	0.057923	0.00006	-0.2
ID–TIMS means:					0.67769			0.084871				0.057912		

mean Concordia age of all four analyses: 525.01 ± 0.92 Ma (95% conf.)

One 646 µg chip was dissolved (split after dissolution).

Table 3.
Results of Hf-isotope analyses by solution ICP–MS

Laboratory	Analysis number	Lu [µg/g]	Hf [µg/g]	^{176}Lu/^{177}Hf	^{176}Hf/^{177}Hf *	2*s* (abs.)	^{176}Hf/^{177}Hf **
Academy of Sciences, Beijing	M127 #1A	46.7	12497	0.00053	0.282393	0.000006	0.282388
	M127 #1A re-analysis				0.282385	0.000007	
	M127 #2D	78.0	12441	0.00089	0.282392	0.000006	0.282383
	M127 #2D re-analysis				0.282385	0.000005	
	M127 #3D	67.9	12239	0.00079	0.282396	0.000004	0.282388
	M127 #3D re-analysis				0.282392	0.000005	
	M127 #4B	81.1	11493	0.00100	0.282388	0.000006	0.282378
	M127 #4B re-analysis				0.282388	0.000006	
	M127 #5B	60.8	12227	0.00071	0.282390	0.000009	0.282383
	M127 #5B re-analysis				0.282400	0.000008	
	M127 #6Y	51.6	12301	0.00060	0.282391	0.000006	0.282385
	M127 #6Y re-analysis				0.282388	0.000007	
Mean observed ^{176}Hf/^{177}Hf ratio of six*** analyses: 0.282392 ± 0.000006 (2*s*)							
Washington State University, Pullman	11-chip 3	64.5	12616	0.00073	0.282397	0.000006	0.282390
	11-chip 3 re-analysis				0.282402	0.000006	
	Chip 12	103.1	12590	0.00116	0.282401	0.000009	0.282389
	Chip 12 re-analysis				0.282395	0.000006	
	Chip 13	48.8	12529	0.00055	0.282394	0.000008	0.282389
	14-chip 1	78.7	12578	0.00089	0.282395	0.000008	0.282386
	14-chip 2	68.7	12604	0.00077	0.282388	0.000006	0.282380
	15-chip 1	69.6	12539	0.00079	0.282385	0.000007	0.282377
	15-chip 2	11.5	12546	0.00013	0.282394	0.000005	0.282393
Mean observed ^{176}Hf/^{177}Hf ratio of seven*** analyses: 0.282393 ± 0.000010 (2*s*)							
Universität zu Köln	M127-1 (# 6)	55.8	12970	0.0006102	0.282406	0.000004	0.282400
	M127-2 (# 7)	56.8	12956	0.0006223	0.282407	0.000005	0.282401
	M127-3 (# 8)	56.1	12847	0.0006198	0.282403	0.000004	0.282397
	M127-4 (# 9)	54.9	12840	0.0006066	0.282401	0.000004	0.282395
	M127-5 (# 10)	54.7	12631	0.0006142	0.282407	0.000004	0.282401
Mean observed ^{176}Hf/^{177}Hf ratio of five analyses: 0.282405 ± 0.000005 (2*s*)							

Results of replicate analyses are quoted but were not considered in the calculation of means.

Note that additional results of Hf-isotope analyses, done by LA–ICP–MS at the Chinese Academy of Sciences, Beijing, are listed in Table S1.

* M127 data are corrected to analyses of JMC 475.

** Age-corrected ^{176}Hf/^{177}Hf ratio (524 Ma).

*** In the calculation of means, results of re-analyses were not considered.

Table 4.

Oxygen isotope analyses by laser fluorination (University of Wisconsin at Madison)

Analysis no.	Sample/reference name	Material analyzed	Weight [mg]	$\delta^{18}O$	
				Raw	[‰ VSMOW]*
Analysis session on January 22, 2014:					
5	UWG-2	Garnet reference	2.00	5.86	
6	UWG-2	Garnet reference	2.09	5.86	
8	UWG-2	Garnet reference	3.11	5.76	
9	UWG-2	Garnet reference	2.81	5.83	
10	M127	Zircon (HF-treated)	3.00	8.50	8.48
11	M127	Zircon (HF-treated)	2.40	8.53	8.51
12	M127	Zircon (HF-treated)	2.52	8.47	8.45
13	M127	Zircon (HF-treated)	3.16	8.54	8.52
14	M127	Zircon (HF-treated)	3.16	8.49	8.47
					M127 average: 8.49 ± 0.06 (2*s*)
15	UWG-2	Garnet reference	2.74	5.83	
16	UWG-2	Garnet reference	2.12	5.77	
Analysis session on May 7, 2014:					
1	UWG-2	Garnet reference	2.92	5.98	
2	UWG-2	Garnet reference	2.26	6.09	
3	UWG-2	Garnet reference	2.37	6.02	
4	UWG-2	Garnet reference	1.86	5.99	
5	UWG-2	Garnet reference	2.90	5.96	
26	M127	Zircon	3.49	8.54	8.32
27	M127	Zircon	2.54	8.47	8.25
28	M127	Zircon	3.98	8.44	8.22
					M127 average: 8.26 ± 0.10 (2*s*)
Analysis session on December 1, 2015:					
2	UWG-2	Garnet reference	2.90	5.80	
3	UWG-2	Garnet reference	2.37	5.77	
4	UWG-2	Garnet reference	2.74	5.78	
5	UWG-2	Garnet reference	2.14	5.85	
6	UWG-2	Garnet reference	1.72	5.79	
7	M127	Zircon	2.62	8.20	8.24
8	M127	Zircon	2.42	8.24	8.28
9	M127	Zircon	2.60	8.19	8.23
10	M127	Zircon	2.45	8.22	8.26
11	M127	Zircon	2.47	8.25	8.29
					M127 average: 8.26 ± 0.05 (2*s*)
12	UWG-2	Garnet reference	2.94	5.65	
13	UWG-2	Garnet reference	2.00	5.74	
14	UWG-2	Garnet reference	2.60	5.73	
15	UWG-2	Garnet reference	2.31	5.76	

(Table 4 continued)

Analysis no.	Sample/reference name	Material analyzed	Weight [mg]	$\delta^{18}O$ Raw	$\delta^{18}O$ [‰ VSMOW]*
Analysis session on December 10, 2015:					
2	UWG-2	Garnet reference	2.76	5.80	
3	UWG-2	Garnet reference	2.22	5.76	
4	UWG-2	Garnet reference	1.75	5.73	
5	UWG-2	Garnet reference	1.51	5.76	
6	M127	Zircon (HF-treated)	1.97	8.44	8.48
7	M127	Zircon	2.09	8.21	8.25
8	M127	Zircon (HF-treated)	1.83	8.45	8.49

Summary:

Zircon M127 (HF-treated; seven individual analyses): Average $\delta^{18}O = 8.49 \pm 0.05$ (2*s*)

Zircon M127 (non HF-treated; nine individual analyses): Average $\delta^{18}O = 8.26 \pm 0.06$ (2*s*)

Note that additional results of O-isotope analyses, done by SIMS at the Chinese Academy of Sciences, Beijing, and the Helmholtz-Zentrum Potsdam (GFZ), are listed in Table S2.

* M127 data are corrected to the respective UWG-2 reference analyses.

Table 5.

(U+Th)/He age of M127 (University of Arizona at Tucson)

Sample name	^{4}He [pmol]	U [ng]	Th [ng]	Age (Ma)
M127zA	11.6 ± 0.2	4.41 ± 0.16	2.04 ± 0.06	425 ± 17
M127zB	24.2 ± 0.6	9.24 ± 0.34	4.26 ± 0.14	423 ± 18
15B346	0.809 ± 0.010	0.303 ± 0.008	0.133 ± 0.004	432 ± 12
15B347	2.05 ± 0.06	0.772 ± 0.024	0.341 ± 0.010	428 ± 17
15B348	0.227 ± 0.006	0.088 ± 0.002	0.041 ± 0.002	417 ± 18
Weighted mean age of five analyses: 426 ± 7 Ma (2*s*)				

All analytical uncertainties are 2*s*.

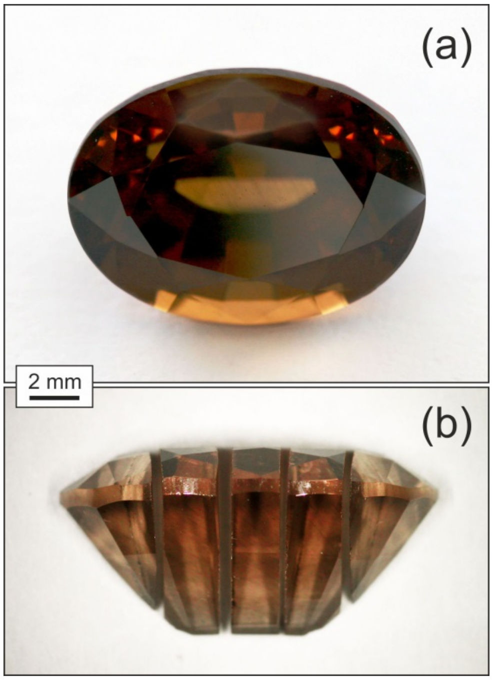

Figure 1. White-light photographs of zircon M127, a clear, dark brownish, cut and faceted gemstone originating from Ratnapura, Sri Lanka. (a) Original stone as purchased. (b) One half after cutting into five slices.

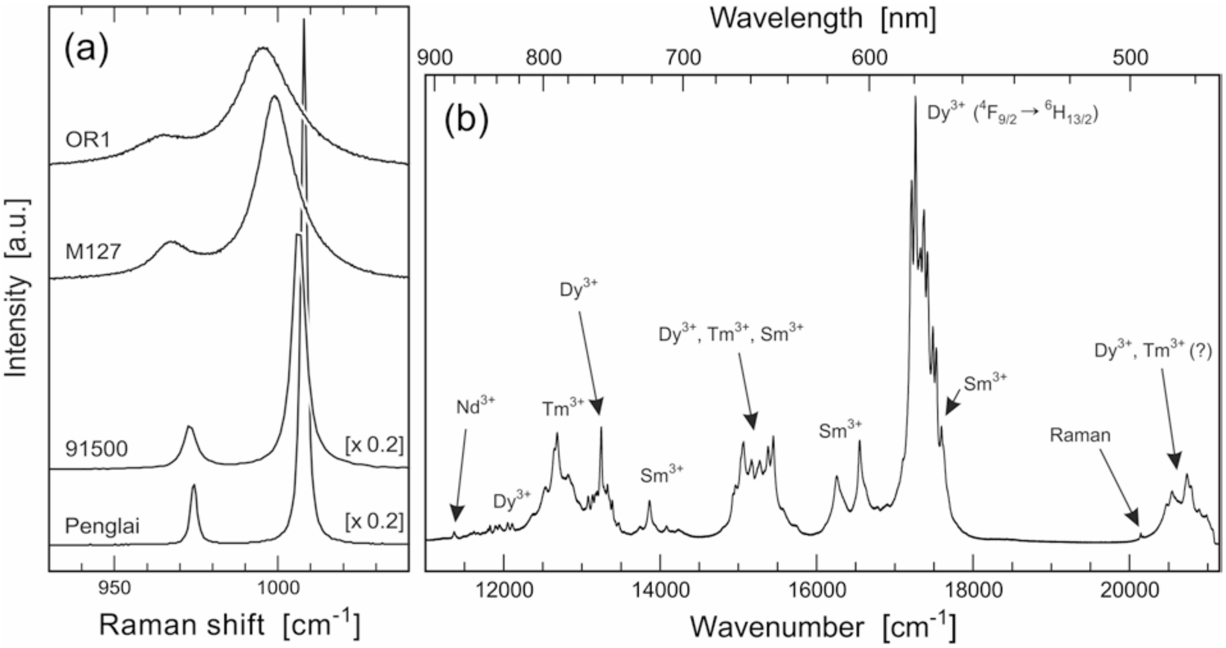

Figure 2. Spectroscopy of zircon M127. (a) Raman spectra in the SiO_4 stretching range, showing M127 in comparison with three zircon references ranging from very well crystallised (Penglai; spectrum this work; for sample description see Li *et al.* 2010), mildly radiation damaged (91500; data from Wiedenbeck *et al.* 2004) to strongly radiation damaged (OR1; data from Nasdala *et al.* 2004). (b) The PL spectrum (473 nm excitation) of M127 is dominated by groups of narrow, REE-related emissions. For assignment see Gaft *et al.* (2000) and Lenz and Nasdala (2015).

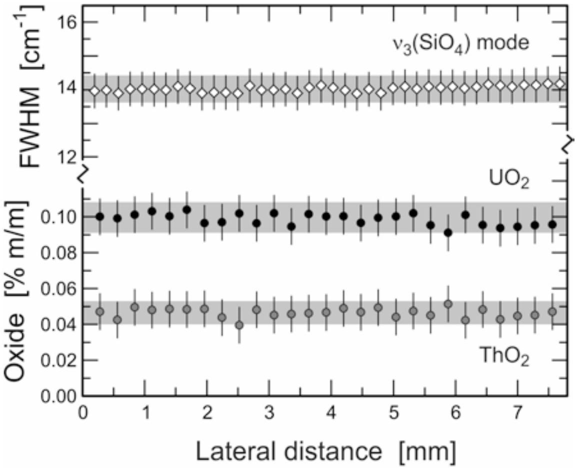

Figure 3. Results of Raman microprobe and EPMA line scans across an ~8mm long traverse. Lateral variations of observed Raman and concentrations parameters are below the analytical uncertainties (visualized by grey bars).

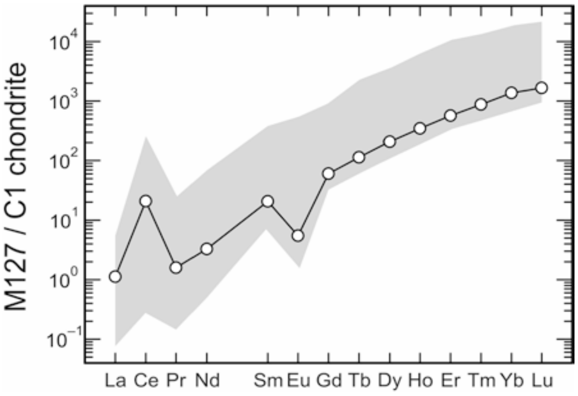

Figure 4. Plot of chondrite-normalised REE concentrations determined by LA–ICP–MS (University of Bergen). The gray area visualises REE concentration ranges obtained for unaltered igneous zircon (graphically extracted from Figures 4 and 5 in Hoskin and Schaltegger 2003).

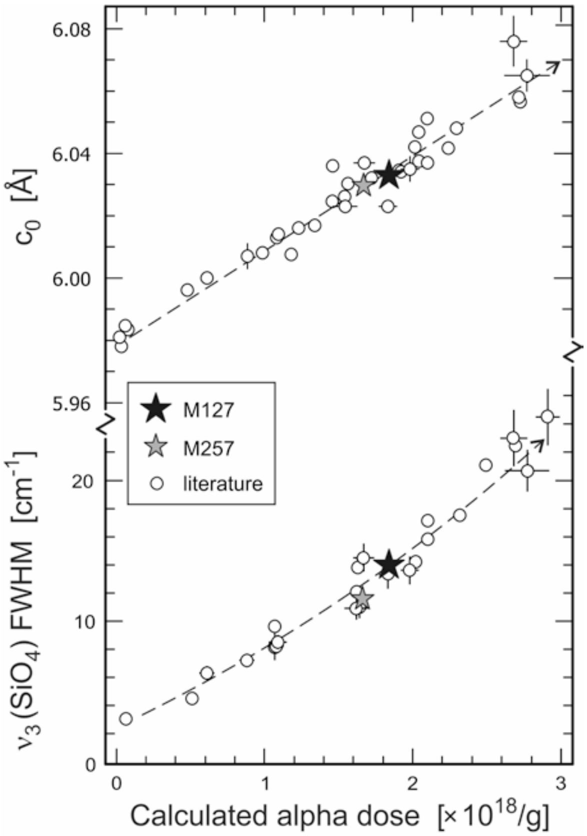

Figure 5. Plots of unit-cell parameter c_0 and the width of the main Raman band at ~1000 cm^{-1} against the time-integrated alpha dose, for zircon samples from Sri Lanka. Literature data for unit-cell constants from Holland (1954), Murakami *et al.* (1991) and Nasdala *et al.* (2004). Literature data for Raman FWHMs from Zhang *et al.* (2000) and Nasdala *et al.* (2001, 2004). Data for zircon M257 from Nasdala *et al.* (2008). Note that parameters of zircon M127 plot well within the "Sri Lankan" trends (visualized by dashed arrows).

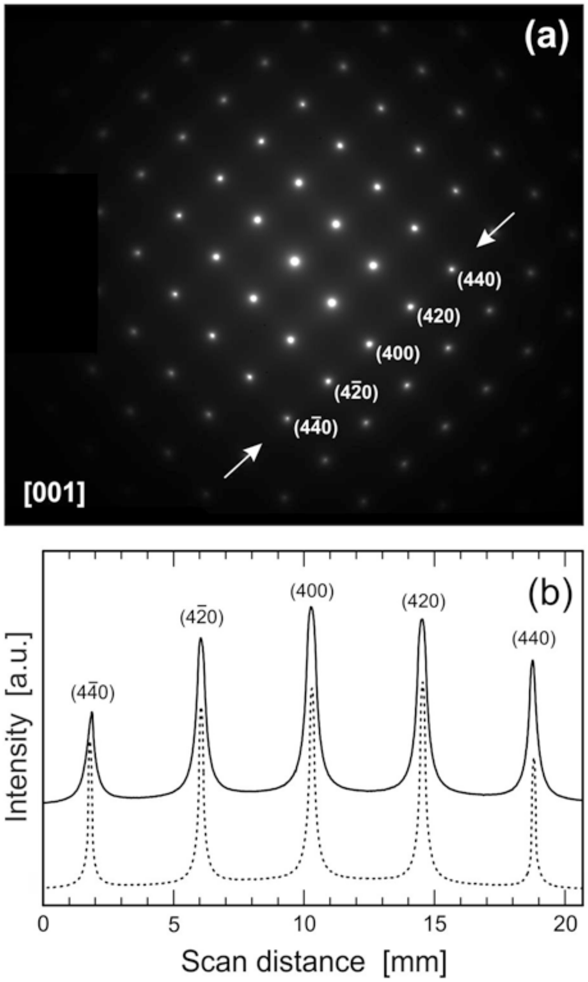

Figure 6. Electron diffraction in the TEM. (a) Photograph of the diffraction pattern of zircon M127 observed along [001]. (b) Plot of the intensity distribution along the line marked in a with arrows (solid graph), in comparison with an analogous intensity distribution pattern obtained from highly crystalline zircon from Ban Lung, Ratanakiri, Cambodia (dotted graph; for sample description see Lenz and Nasdala 2015). Graphs are shown with vertical offset for clarity.

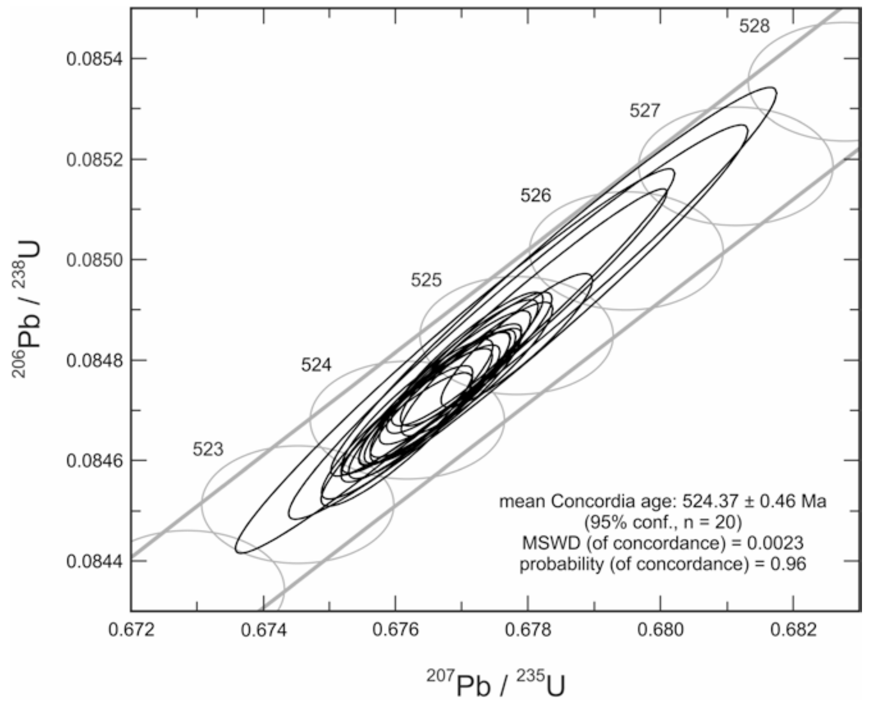

Figure 7. Concordia plot of U–Pb data obtained at the University of California at Santa Barbara (CA–TIMS; 16 plateau steps; data from Mattinson 2010) and at the University of Oslo (ID–TIMS; four individual analyses). All ellipses represent 2*s* uncertainties. The uncertainty of the mean age quoted includes uncertainties of decay-constants.

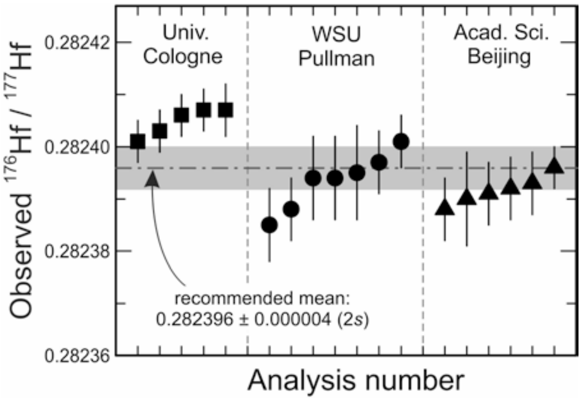

Figure 8. Results of Hf-isotope analyses (solution–ICP–MS) done at University of Cologne (5 individual analyses), Washington State University, Pullmann, WA (7), and the Chinese Academy of Sciences, Beijing (6). Error bars represent 2*s* uncertainties.

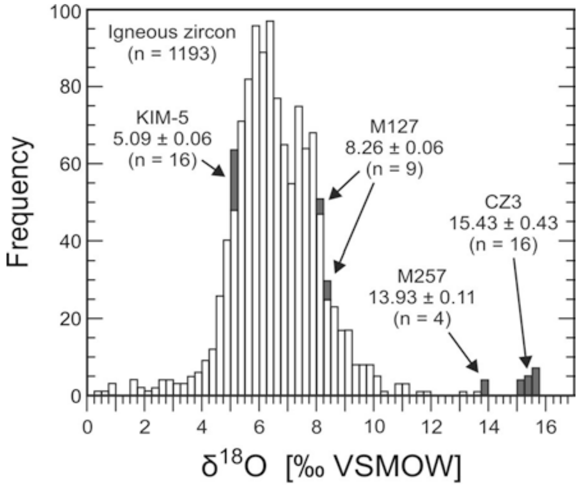

Figure 9. Plot of δ^{18}O data for zircon M127 (laser fluorination; University of Wisconsin, Madison) and data for references KIM–5 (Valley 2003), M257 (Nasdala *et al.* 2008) and CZ3 (Cavosie *et al.* 2011) in comparison with those for igneous zircon from localities worldwide (Valley *et al.*, 2005).

Table S1.

Results of a total of 130 Hf-isotope analyses by LA–ICP–MS (six analytical sessions 2009–2013) at the Chinese Academy of Sciences, Beijing

Analysis name / number	^{176}Yb/^{177}Hf	2*s*	^{176}Lu/^{177}Hf	2*s*	^{176}Hf/^{177}Hf*	2*s*
● *Analyses on November 26, 2009:*						
20091126 M127 40u 4h 01	0.020157	0.000039	0.000659	0.0000005	0.282391	0.000024
20091126 M127 40u 4h 02	0.020244	0.000034	0.000658	0.0000004	0.282415	0.000016
20091126 M127 40u 4h 03	0.020169	0.000040	0.000655	0.0000004	0.282399	0.000018
20091126 M127 40u 4h 04	0.020163	0.000038	0.000657	0.0000004	0.282408	0.000017
20091126 M127 40u 4h 05	0.020114	0.000024	0.000655	0.0000006	0.282366	0.000017
20091126 M127 40u 4h 06	0.020039	0.000039	0.000654	0.0000004	0.282372	0.000016
20091126 M127 40u 4h 07	0.020074	0.000038	0.000660	0.0000004	0.282398	0.000016
20091126 M127 40u 4h 08	0.019962	0.000027	0.000658	0.0000004	0.282355	0.000016
20091126 M127 40u 4h 09	0.019227	0.000019	0.000649	0.0000007	0.282401	0.000013
20091126 M127 40u 4h 10	0.019531	0.000031	0.000648	0.0000003	0.282380	0.000020
20091126 M127 40u 4h 11	0.019715	0.000035	0.000652	0.0000002	0.282411	0.000017
20091126 M127 40u 4h 12	0.019656	0.000034	0.000651	0.0000002	0.282451	0.000017
20091126 M127 40u 4h 13	0.019579	0.000034	0.000650	0.0000003	0.282395	0.000019
20091126 M127 40u 4h 14	0.019705	0.000032	0.000651	0.0000002	0.282397	0.000018
20091126 M127 40u 4h 15	0.019735	0.000030	0.000651	0.0000003	0.282374	0.000016
20091126 M127 40u 4h 16	0.019504	0.000031	0.000644	0.0000002	0.282403	0.000018
20091126 M127 40u 4h 17	0.019483	0.000033	0.000646	0.0000003	0.282359	0.000016
20091126 M127 40u 4h 18	0.019594	0.000033	0.000648	0.0000003	0.282403	0.000018
20091126 M127 40u 4h 19	0.019537	0.000031	0.000648	0.0000002	0.282399	0.000017
20091126 M127 40u 4h 20	0.019541	0.000033	0.000649	0.0000002	0.282410	0.000017
🟩 *Analyses on January 13, 2011:*						
20110113 M127 01	0.015467	0.000007	0.000664	0.0000002	0.282400	0.000014
20110113 M127 02	0.015421	0.000009	0.000663	0.0000003	0.282427	0.000022
20110113 M127 03	0.015539	0.000021	0.000665	0.0000003	0.282381	0.000018
20110113 M127 04	0.015430	0.000006	0.000663	0.0000002	0.282407	0.000017
20110113 M127 05	0.015361	0.000013	0.000661	0.0000002	0.282381	0.000019
20110113 M127 06	0.015360	0.000007	0.000660	0.0000003	0.282386	0.000018
20110113 M127 07	0.015330	0.000008	0.000658	0.0000003	0.282384	0.000018
20110113 M127 08	0.015320	0.000007	0.000659	0.0000002	0.282407	0.000018
20110113 M127 09	0.015382	0.000007	0.000662	0.0000003	0.282406	0.000019
20110113 M127 10	0.015387	0.000006	0.000662	0.0000003	0.282385	0.000017
20110113 M127 11	0.015404	0.000008	0.000661	0.0000002	0.282402	0.000015
20110113 M127 12	0.015434	0.000006	0.000662	0.0000002	0.282389	0.000018
20110113 M127 13	0.015529	0.000008	0.000667	0.0000003	0.282377	0.000017
20110113 M127 14	0.015529	0.000006	0.000667	0.0000002	0.282379	0.000015
20110113 M127 15	0.015403	0.000007	0.000660	0.0000002	0.282367	0.000015
20110113 M127 16	0.015462	0.000008	0.000663	0.0000002	0.282386	0.000016
20110113 M127 17	0.015422	0.000008	0.000662	0.0000003	0.282364	0.000015
20110113 M127 18	0.015432	0.000009	0.000663	0.0000002	0.282391	0.000015
20110113 M127 19	0.015457	0.000007	0.000664	0.0000002	0.282393	0.000017
20110113 M127 20	0.015495	0.000009	0.000664	0.0000002	0.282400	0.000016
◆ *Analyses on June 18, 2013:*						
2013.06.18 M127 01	0.016784	0.000038	0.000666	0.0000015	0.282395	0.000015
2013.06.18 M127 02	0.016662	0.000007	0.000660	0.0000004	0.282377	0.000013
2013.06.18 M127 03	0.016488	0.000008	0.000654	0.0000005	0.282386	0.000013
2013.06.18 M127 04	0.016499	0.000006	0.000655	0.0000003	0.282392	0.000012
2013.06.18 M127 05	0.016647	0.000006	0.000659	0.0000003	0.282375	0.000013
2013.06.18 M127 06	0.016558	0.000007	0.000657	0.0000004	0.282379	0.000013
2013.06.18 M127 07	0.016468	0.000007	0.000655	0.0000004	0.282390	0.000012
2013.06.18 M127 08	0.016521	0.000006	0.000656	0.0000003	0.282382	0.000013
2013.06.18 M127 09	0.016576	0.000009	0.000655	0.0000004	0.282390	0.000012
2013.06.18 M127 10	0.016707	0.000009	0.000654	0.0000003	0.282387	0.000014
2013.06.18 M127 11	0.016535	0.000006	0.000657	0.0000004	0.282377	0.000011

2013.06.18 M127 12	0.016537	0.000008	0.000658	0.0000004	0.282376	0.000013
2013.06.18 M127 13	0.016753	0.000017	0.000657	0.0000003	0.282378	0.000012
2013.06.18 M127 14	0.016713	0.000014	0.000657	0.0000004	0.282391	0.000011
2013.06.18 M127 15	0.016729	0.000013	0.000658	0.0000003	0.282401	0.000012
2013.06.18 M127 16	0.016805	0.000008	0.000667	0.0000004	0.282384	0.000013
2013.06.18 M127 17	0.016835	0.000008	0.000667	0.0000003	0.282405	0.000012
2013.06.18 M127 18	0.016829	0.000012	0.000663	0.0000003	0.282380	0.000012
2013.06.18 M127 19	0.016748	0.000008	0.000661	0.0000003	0.282387	0.000012
2013.06.18 M127 20	0.016490	0.000013	0.000652	0.0000002	0.282357	0.000012
2013.06.18 M127 21	0.016379	0.000014	0.000652	0.0000005	0.282374	0.000012
2013.06.18 M127 22	0.016497	0.000007	0.000656	0.0000003	0.282379	0.000012
2013.06.18 M127 23	0.016583	0.000009	0.000656	0.0000004	0.282364	0.000012
2013.06.18 M127 24	0.016694	0.000010	0.000657	0.0000003	0.282376	0.000013
2013.06.18 M127 25	0.016639	0.000013	0.000657	0.0000004	0.282393	0.000011
● *Analyses on July 27, 2013:*						
20130727 M127 60m 5h 01	0.018043	0.000051	0.000652	0.0000008	0.282388	0.000010
20130727 M127 60m 5h 02	0.017993	0.000059	0.000654	0.0000004	0.282377	0.000010
20130727 M127 60m 5h 03	0.018070	0.000055	0.000653	0.0000004	0.282380	0.000010
20130727 M127 60m 5h 04	0.017941	0.000052	0.000649	0.0000002	0.282385	0.000010
20130727 M127 60m 5h 05	0.017870	0.000050	0.000646	0.0000003	0.282380	0.000011
20130727 M127 60m 5h 06	0.017833	0.000037	0.000648	0.0000005	0.282394	0.000011
20130727 M127 60m 5h 07	0.018109	0.000054	0.000647	0.0000003	0.282411	0.000011
20130727 M127 60m 5h 08	0.018070	0.000049	0.000648	0.0000003	0.282396	0.000010
20130727 M127 60m 5h 09	0.018153	0.000052	0.000644	0.0000002	0.282390	0.000011
20130727 M127 60m 5h 10	0.018183	0.000062	0.000651	0.0000002	0.282402	0.000010
20130727 M127 60m 5h 11	0.018235	0.000066	0.000644	0.0000003	0.282374	0.000011
20130727 M127 60m 5h 12	0.018339	0.000042	0.000651	0.0000002	0.282386	0.000011
20130727 M127 60m 5h 13	0.018644	0.000071	0.000651	0.0000004	0.282375	0.000011
20130727 M127 60m 5h 14	0.018462	0.000051	0.000653	0.0000003	0.282374	0.000010
20130727 M127 60m 5h 15	0.018248	0.000066	0.000646	0.0000004	0.282370	0.000011
20130727 M127 60m 5h 16	0.018273	0.000062	0.000647	0.0000006	0.282396	0.000010
20130727 M127 60m 5h 17	0.018340	0.000057	0.000647	0.0000003	0.282399	0.000010
20130727 M127 60m 5h 18	0.018319	0.000061	0.000647	0.0000003	0.282386	0.000011
20130727 M127 60m 5h 19	0.018223	0.000068	0.000646	0.0000004	0.282387	0.000011
20130727 M127 60m 5h 20	0.018143	0.000059	0.000646	0.0000003	0.282401	0.000010
△ *Analyses on August 19, 2013:*						
20130819 M127 60mm 01	0.017040	0.000014	0.000657	0.0000002	0.282401	0.000014
20130819 M127 60mm 02	0.016935	0.000016	0.000655	0.0000003	0.282387	0.000012
20130819 M127 60mm 03	0.016984	0.000012	0.000658	0.0000002	0.282396	0.000013
20130819 M127 60mm 04	0.016889	0.000013	0.000654	0.0000002	0.282398	0.000013
20130819 M127 60mm 05	0.017053	0.000015	0.000660	0.0000002	0.282401	0.000013
20130819 M127 60mm 06	0.016922	0.000012	0.000654	0.0000002	0.282426	0.000013
20130819 M127 60mm 07	0.017033	0.000015	0.000659	0.0000002	0.282390	0.000013
20130819 M127 60mm 08	0.017013	0.000015	0.000655	0.0000003	0.282403	0.000014
20130819 M127 60mm 09	0.017037	0.000015	0.000657	0.0000002	0.282385	0.000013
20130819 M127 60mm 10	0.017026	0.000021	0.000656	0.0000002	0.282402	0.000014
20130819 M127 60mm 11	0.017040	0.000017	0.000657	0.0000003	0.282392	0.000013
20130819 M127 60mm 12	0.017079	0.000016	0.000657	0.0000002	0.282374	0.000013
20130819 M127 60mm 13	0.017043	0.000018	0.000656	0.0000002	0.282397	0.000012
20130819 M127 60mm 14	0.017104	0.000015	0.000656	0.0000003	0.282378	0.000014
20130819 M127 60mm 15	0.017007	0.000018	0.000655	0.0000002	0.282390	0.000013
▽ *Analyses on September 15, 2013:*						
20130915 M127 01	0.018840	0.000023	0.000645	0.0000002	0.282361	0.000013
20130915 M127 02	0.018825	0.000020	0.000645	0.0000003	0.282392	0.000013
20130915 M127 03	0.018766	0.000023	0.000645	0.0000002	0.282393	0.000014
20130915 M127 04	0.018765	0.000022	0.000645	0.0000003	0.282415	0.000014
20130915 M127 05	0.018648	0.000030	0.000647	0.0000004	0.282390	0.000012
20130915 M127 06	0.018628	0.000029	0.000647	0.0000003	0.282404	0.000013
20130915 M127 07	0.018637	0.000028	0.000647	0.0000004	0.282410	0.000012
20130915 M127 08	0.018631	0.000031	0.000647	0.0000003	0.282412	0.000013
20130915 M127 09	0.018541	0.000024	0.000647	0.0000002	0.282412	0.000013
20130915 M127 10	0.018575	0.000026	0.000647	0.0000002	0.282396	0.000014

20130915 M127 11	0.018559	0.000023	0.000648	0.0000002	0.282390	0.000012
20130915 M127 12	0.018505	0.000023	0.000648	0.0000003	0.282406	0.000012
20130915 M127 13	0.018473	0.000019	0.000647	0.0000003	0.282419	0.000013
20130915 M127 14	0.018536	0.000026	0.000647	0.0000002	0.282409	0.000012
20130915 M127 15	0.018562	0.000026	0.000647	0.0000002	0.282382	0.000012
20130915 M127 16	0.018544	0.000023	0.000646	0.0000003	0.282414	0.000012
20130915 M127 17	0.018457	0.000019	0.000647	0.0000003	0.282408	0.000013
20130915 M127 18	0.018482	0.000028	0.000646	0.0000003	0.282396	0.000012
20130915 M127 19	0.018533	0.000023	0.000646	0.0000003	0.282407	0.000013
20130915 M127 20	0.018527	0.000028	0.000647	0.0000003	0.282420	0.000012
20130915 M127 21	0.018530	0.000023	0.000646	0.0000003	0.282398	0.000012
20130915 M127 22	0.018530	0.000030	0.000648	0.0000003	0.282398	0.000012
20130915 M127 23	0.018581	0.000029	0.000648	0.0000003	0.282389	0.000012
20130915 M127 24	0.018576	0.000024	0.000648	0.0000003	0.282387	0.000012
20130915 M127 25	0.018476	0.000031	0.000644	0.0000002	0.282393	0.000012
20130915 M127 26	0.018544	0.000029	0.000646	0.0000002	0.282408	0.000011
20130915 M127 27	0.018660	0.000027	0.000651	0.0000003	0.282397	0.000012
20130915 M127 28	0.018699	0.000029	0.000651	0.0000004	0.282394	0.000012
Means (2*s*)			0.000654	0.000013	0.282392	0.000031

* Measured ^{176}Hf/^{177}Hf ratios for M127 are corrected to analyses of the 91500 reference zircon, with GJ-1 used as secondary calibrant material.

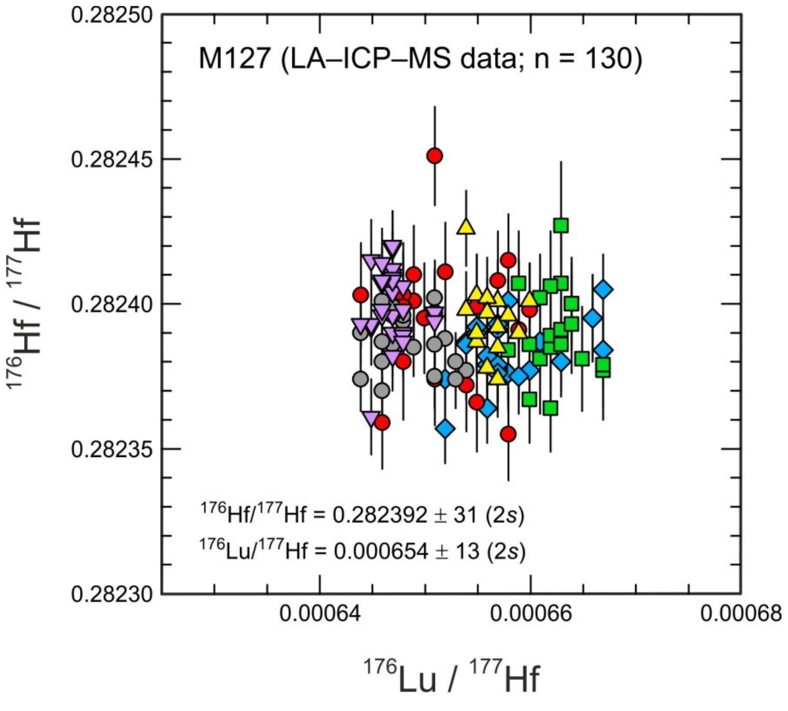

Table S2.

Results of O-isotope analyses by SIMS at the Chinese Academy of Sciences, Beijing, and the Helmholtz-Zentrum Potsdam (GFZ)

Grain and analysis number	Date – time	Primary current [nA]	Intensity ^{16}O [× 10^9 cps]	δ^{18}O [‰ VSMOW]	δ^{18}O means* [‰ VSMOW]
*Analyses at the Chinese Academy of Sciences, Beijing (n = 30)***					
M127–1A #1	May 17, 2014 – 13:53	1.25	1.359	8.07	
M127–1A #2	May 17, 2014 – 13:56	1.24	1.354	8.48	
M127–1A #3	May 17, 2014 – 13:59	1.24	1.351	8.63	
M127–1A #4	May 17, 2014 – 14:08	1.24	1.353	8.16	
M127–1A #5	May 17, 2014 – 14:11	1.23	1.343	8.40	8.35 ± 0.46
M127–2D #1	May 17, 2014 – 14:20	1.23	1.339	8.28	
M127–2D #2	May 17, 2014 – 14:23	1.23	1.341	8.21	
M127–2D #3	May 17, 2014 – 14:29	1.24	1.353	8.56	
M127–2D #4	May 17, 2014 – 14:36	1.23	1.343	8.46	
M127–2D #5	May 17, 2014 – 14:38	1.23	1.338	8.30	8.36 ± 0.29
M127–3D #1	May 17, 2014 – 16:15	1.22	1.337	8.31	
M127–3D #2	May 17, 2014 – 16:21	1.23	1.334	8.33	
M127–3D #3	May 17, 2014 – 16:27	1.22	1.333	8.18	
M127–3D #4	May 17, 2014 – 16:34	1.22	1.328	8.26	
M127–3D #5	May 17, 2014 – 16:37	1.22	1.326	8.53	8.32 ± 0.25
M127–4B1 #1	May 17, 2014 – 14:54	1.21	1.322	8.52	
M127–4B1 #2	May 17, 2014 – 14:57	1.22	1.332	8.67	
M127–4B1 #3	May 17, 2014 – 15:00	1.23	1.334	8.59	
M127–4B1 #4	May 17, 2014 – 15:09	1.23	1.327	8.49	
M127–4B1 #5	May 17, 2014 – 15:12	1.23	1.337	8.35	
M127–4B1 #6	May 17, 2014 – 15:15	1.24	1.334	8.34	8.49 ± 0.29
M127–4B2 #1	May 17, 2014 – 15:24	1.24	1.335	8.17	
M127–4B2 #2	May 17, 2014 – 15:27	1.24	1.340	8.19	
M127–4B2 #3	May 17, 2014 – 15:36	1.23	1.337	8.36	
M127–4B2 #4	May 17, 2014 – 15:39	1.23	1.334	8.19	8.23 ± 0.17
M127–5B #1	May 17, 2014 – 15:45	1.22	1.330	8.03	
M127–5B #2	May 17, 2014 – 15:51	1.23	1.334	8.06	
M127–5B #3	May 17, 2014 – 15:54	1.22	1.339	8.37	
M127–5B #4	May 17, 2014 – 16:03	1.23	1.336	8.33	
M127–5B #5	May 17, 2014 – 16:09	1.24	1.335	8.28	8.21 ± 0.32
			Mean of 30 individual analyses: 8.34 ± 0.34 (2*s*)		
*Analyses at the Helmholtz-Zentrum Potsdam (GFZ) (n = 41)****					
#16: M127–grain1	Nov 15, 2014 – 15:11	1.91	3.57	8.06	
#17: M127–grain1	Nov 15, 2014 – 15:14	1.92	3.56	7.89	
#18: M127–grain1	Nov 15, 2014 – 15:17	1.92	3.56	7.99	
#19: M127–grain1	Nov 15, 2014 – 15:21	1.93	3.56	8.02	
#94: M127–grain1	Nov 15, 2014 – 19:27	1.85	3.49	8.08	
#95: M127–grain1	Nov 15, 2014 – 19:30	1.85	3.48	8.09	
#202: M127–grain1	Nov 16, 2014 – 01:21	1.70	3.66	8.04	
#203: M127–grain1	Nov 16, 2014 – 01:25	1.71	3.65	8.15	
#242: M127–grain1	Nov 16, 2014 – 03:33	1.67	3.60	8.05	
#243: M127–grain1	Nov 16, 2014 – 03:37	1.67	3.60	7.98	8.03 ± 0.14
#74: M127–grain2	Nov 15, 2014 – 18:21	1.86	3.49	8.01	
#75: M127–grain2	Nov 15, 2014 – 18:24	1.86	3.49	7.97	
#112: M127–grain2	Nov 15, 2014 – 20:26	1.82	3.45	7.92	
#113: M127–grain2	Nov 15, 2014 – 20:29	1.83	3.45	7.90	
#200: M127–grain2	Nov 16, 2014 – 01.15	1.71	3.66	8.08	
#201: M127–grain2	Nov 16, 2014 – 01:18	1.71	3.65	7.91	
#214: M127–grain2	Nov 16, 2014 – 02:01	1.69	3.63	7.96	

#215: M127–grain2	Nov 16, 2014 – 02:04	1.70	3.63	8.04	
#256: M127–grain2	Nov 16, 2014 – 04:20	1.66	3.58	7.99	
#257: M127–grain2	Nov 16, 2014 – 04:23	1.67	3.58	7.88	7.97 ± 0.13
#34: M127–grain3	Nov 15, 2014 – 16:10	1.89	3.53	8.08	
#35: M127–grain3	Nov 15, 2014 – 16:13	1.89	3.52	7.88	
#38: M127–grain3	Nov 15, 2014 – 16:23	1.89	3.52	7.86	
#39: M127–grain3	Nov 15, 2014 – 16:26	1.89	3.52	7.98	
#40: M127–grain3	Nov 15, 2014 – 16:29	1.89	3.53	7.97	
#41: M127–grain3	Nov 15, 2014 – 16:33	1.90	3.57	7.95	
#134: M127–grain3	Nov 15, 2014 – 21:38	1.79	3.40	7.90	
#135: M127–grain3	Nov 15, 2014 – 21:41	1.79	3.39	7.95	7.95 ± 0.14
#98: M127–grain4	Nov 15, 2014 – 19:40	1.85	3.47	7.99	
#99: M127–grain4	Nov 15, 2014 – 19:43	1.84	3.46	7.97	
#152: M127–grain4	Nov 15, 2014 – 22:37	1.76	3.36	8.01	
#153: M127–grain4	Nov 15, 2014 – 22:40	1.77	3.36	7.98	
#154: M127–grain4	Nov 15, 2014 – 22:43	1.77	3.36	7.78	
#155: M127–grain4	Nov 15, 2014 – 22:47	1.76	3.35	7.85	7.93 ± 0.19
#5: M127–grain5	Nov 15, 2014 – 13:47	1.94	3.53	7.97	
#6: M127–grain5	Nov 15, 2014 – 14:38	1.92	3.51	7.71	
#7: M127–grain5	Nov 15, 2014 – 14:41	1.93	3.51	7.65	
#176: M127–grain5	Nov 15, 2014 – 23:56	1.74	3.32	7.84	
#177: M127–grain5	Nov 15, 2014 – 23:59	1.74	3.32	7.89	
#232: M127–grain5	Nov 16, 2014 – 03:00	1.69	3.62	7.90	
#233: M127–grain5	Nov 16, 2014 – 03:04	1.69	3.61	8.07	7.86 ± 0.29
			Mean of 41 individual analyses: 7.96 ± 0.20 (2*s*)		

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* All errors reported are 2*s*.

** M127 data obtained in Beijing are corrected to analyses of the Penglai reference zircon.

*** M127 data obtained in Potsdam are corrected to analyses of the 91500 reference zircon.