The Geology and Tectonics of central Bhutan

Supplementary File A: U-Pb dating of samples

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1. Sample locations and descriptions

1.1 Sample locations

Table S1.1 Sample locations

Sample or Locality	Rock type	Location	Region	GPS	Host unit
LG-09-21	Metavolcanic ash bed	Zhemgang	Central Bhutan	N27.28155 E90.62450	TSS
LG-09-7A	Leucogranite (cross-cutting)	Trongsa	Central Bhutan	N27.47178 E90.47928	GHS
LG-10-33	Leucogranite (cross-cutting)	Nikha Chu	North-central Bhutan	N27.71674 E90.28676	TSS
LG-10-87B	Leucogranite (cross-cutting)	Chukha	Southwest Bhutan	N27.04453 E89.57191	TSS

For all other sample localities, please refer to Supplemental File B

1.2 Sample descriptions

Sample descriptions – metavolcanic ash bed

LG-09-21

LG-09-21 is an *in situ* sample from a road cut section ~10 km NW of Zhemgang. It is a pale cream-coloured metavolcanic ash with large (2 mm), randomly-oriented biotite porphyroblasts set in a fine-grained matrix of muscovite and quartz, with accessory titanite, zircon and magnetite. The sample was collected from a 10 cm thick, bedding-parallel band within well-bedded buff quartzites that in places display cross bedding. The ash bed weathered slightly more recessively than the quartzite beds above and below.

Sample descriptions – granitoids

CROSS-CUTTING

LG-09-7A

LG-09-7A is a coarse-grained pegmatite from the mid GHS just west of Trongsa. It intrudes the orthogneiss of the GHS as a dyke, ~1 m wide cross-cutting the ductile fabrics of the host rock. The pegmatite is very coarse with grains up to 7 cm; it is homogeneous and shows no deformation features.

LG-09-7A is a leucocratic, holocrystalline, phaneritic pegmatite. It is composed of 60% potassium feldspar, 20% plagioclase, 10% quartz and 10% muscovite. Potassium feldspar grains are <70 mm in diameter, plagioclase grains are 4 x 20 mm, quartz grains are equant and 3 mm in diameter and muscovite grains are 1 - 70 mm across. Plagioclase exhibits lamella twinning in elongate grains.

LG-10-33

LG-10-33 is a medium-grained granite dyke intruding carbonate rocks of Tethyan sedimentary series in the upper Nikha Chu. The dyke is 2 m wide, strike north-south and cross cuts the intricate D1 recumbent folding of the carbonate. It is straight and undeformed. The granite is homogeneous and does not show any internal fabric.

LG-10-33 is a leucocratic, homogeneous and undeformed granite. It is composed of 78% feldspar, 15% quartz, 5% tourmaline and 2 % muscovite. Feldspar grains are 5 – 6 mm in diameter, quartz grains are 0.1 – 0.5 mm constituting patches of 5 mm diameter, muscovite and tourmaline grains are 0.1 – 2 mm in diameter. Feldspar is constituted of untwinned grains, plagioclase with lamella twinning and microcline with cross-hatch twinning. Grains of feldspar and quartz, 0.01 – 0.3 mm in diameter, are included in selected large feldspar

grains; the inclusions are distributed in concentric circle in one grain and along two sides of a square in another grain. The inclusions are often aligned parallel to the crystal faces. Rare inclusions of muscovite are found accompanying the feldspar and quartz inclusions in the feldspar.

LG-10-87B

LG-10-87B is a medium- to course-grained leucogranite, from a ~1 m wide dyke intruded into calc-silicate sediments that are interpreted as Tethyan sediment in the Chukha region of south-west Bhutan. The dyke is concordant with the fabric of the country rock and with little internal fabric.

LG-10-87B is a somewhat altered, undeformed granite. It is comprised of approximately 55% feldspar, 35% quartz, 7% chlorite, 1% biotite and 2% tourmaline. Feldspar grains are approximately 7 mm in diameter, quartz grains are 0.2 – 2 mm in diameter, biotite grains - mostly altered to chlorite - are 3 x 10 mm and tourmaline grains are 0.5 mm in diameter and 2 – 3 mm in length. Feldspar has been altered to varying degrees and exhibits some lamellar twinning.

2. Features of analysed zircons

2.1 Descriptions of granitoid zircons

Zircon grains in sample LG-09-7A are generally euhedral and broken so that only fragments have been selected and imaged. The grains are 100 – 300 µm wide although due to the fragmented nature of the grains elongation and whole grain size cannot be measured. CL images indicate that none of the grains picked and imaged contain bright xenocrystic cores, though clearly distinct cores are present in many samples. The grains are generally dark in CL although some patches and zones are brighter. Most grain fragments show both oscillatory and convolute zoning with some additional patchy zoning. Some grains are completely composed of convolute and patchy zoning; in contrast to fragments that are entirely oscillatory zoned, spot analyses were taken on oscillatory zoned regions only.

Zircon grains in sample LG-10-33 are generally euhedral to subhedral with an average elongation ratio of ~3. The grains are 20 – 80 µm wide and 70 – 200 µm in length. CL images indicate that approximately a quarter of the grains picked and imaged contain bright xenocrystic cores which are 30 – 40 µm wide and 50 – 70 µm in length. The cores show oscillatory zoning and are rounded and anhedral in form. Rims and grains without xenocrystic cores are generally dark in CL although some narrow zones are brighter. 5 – 100 µm wide rims are present around some grains. All crystals show oscillatory zoning although many exhibit a proportion of convolute zoning; these areas were avoided in spot sites for analyses. The edges of some grains show resorption textures and fractures in the grains which alter the oscillatory zoning pattern within the grain.

Zircon grains in sample LG-10-87B are generally euhedral to subhedral and have an average elongation ratio of ~3. The grains are 20 – 50 µm wide and 70 – 200 µm in length. CL images indicate that approximately a third of grains picked and imaged contain generally bright xenocrystic cores. The cores show inconsistent zoning and are often euhedral. Rims, 5 – 50 µm wide, are generally dark in CL and show clear oscillatory zoning.

3. Methods

3.1 Sample processing prior to geochronological analysis

Zircons were separated from granite samples by crushing and milling followed by separation with a Rogers© table, heavy liquids and a Frantz© magnetic separator.

For both LA-ICP-MS and ID-TIMS analysis, small (<100 µm long), elongate, clean zircon crystals were hand-picked under a binocular microscope, avoiding inherited grains with distinct cores.

For LA-ICP-MS analysis, hand-picked grains were mounted in a polished epoxy disk. One group of grains was polished, while a second group was left unpolished. Polished grains were imaged individually using cathodoluminescence (CL). CL imaging was undertaken using the FEI Company QUANTA 600 Environmental Scanning Electron Microscope (ESEM) with a tungsten tetrode electron gun equipped with a Centaurus cathodoluminescence detector for visible-light range cathodoluminescence (CL) imaging at the British Geological Survey.

3.2 U-Pb geochronology using ID-TIMS

Fragments and single whole zircons from the ash bed were placed in a muffle furnace at ~900°C for ~60 hours in quartz dishes before being photographed. Crystals were then transferred to 300 µl Teflon FEP microcapsules and 120 µl of 29 M HF and ~25 µl of 30% HNO2 were added. Microcapsules were placed in a Parr vessel, and leached at 180°C for 12-14 hours. The fractions were then removed and rinsed in ultrapure H2O, fluxed at ~80°C on a hotplate for an hour in 6 M HCL, ultrasonically cleaned for another hour and then put back on a hot plate for ~30 minutes. HCl solutions were then removed, the zircons were rinsed again with ultrapure acetone and H2O, and spiked with the ET535 tracer solution. Zircons were dissolved using Parr vessels in 120 µl of 29 M HF with ~25 µl of 30% HNO3 at 220°C for 48 hours and were then dried to fluorides on a 120°C hotplate. Salts were re-dissolved in 6 µl of 3.1 M HCl, ready for column chemistry. Zirconium, hafnium, and rare earth element washes were saved for future analyses. U and Pb were loaded onto a single Re filament using a silica-gel/phosphoric acid combination (Gerstenberger and Haase, 1997).

Isotope ratio measurements were subsequently performed at the Natural Environment Research Council Isotope Geology Laboratory (NIGL) on a Thermo-Electron Triton TIMS instrument equipped with a modified MassCom SEM that is effectively stable with a linear response effect, up to 106 counts/second, and which thus allows for measurement of small Pb loads using a low noise amplifier for UO2+ analysis in static mode.

U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007), combined with a 235U/205Pb ratio of 100.18 and 233U/235U double spike ratio of 0.99464 for the ET535 tracer. All common Pb in the analyses was attributed to the blank and subtracted based on the isotopic composition and associated uncertainties analyzed over time. Errors for U-Pb dates are reported in the following format: ±X(Y)[Z], where X is the internal or analytical uncertainty in the absence of systematic errors (tracer calibration and decay constants), Y includes the quadratic addition of tracer calibration error (using a conservative estimate of the standard deviation of 0.1% for the Pb/U ratio in the tracer), and Z includes the quadratic addition of both the tracer calibration error and additional 238U decay constant errors of Jaffey *et al.* (1971). All analytical uncertainties are calculated at the 95% confidence interval. These 238U/206Pb dates are traceable back to SI units via the gravimetric calibration of the EARTHTIME U-Pb tracer and the determination of the 238U decay constant (Jaffey *et al.*, 1971; Condon *et al.*, 2007).

3.3 U-Pb geochronology using Nu Plasma LA-ICP-MS (NIGL, Keyworth, UK)

Zircons from the ash bed and orthogneiss samples were analysed at the NERC Isotope Geosciences Laboratory (NIGL) using a Nu Instruments, Nu Plasma HR multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) equipped with a multi-ion-counting array. Instrumental parameters are shown in Table 1 and methodology is detailed in Thomas *et al.* (2010).

Rock samples were crushed using a jaw crusher and disk mill. Milled material was then sieved and material <355µm collected. The heavy material was separated from the light material using the Roger's table, which was then separated further using a Frantz magnetic separator. Subsequently the non-magnetic fraction was further separated using diodomethane heavy liquid separation. Finally zircons of varying sizes were picked from this heavy-non-magnetic fraction in ethanol. Samples were mounted in 25mm epoxy resin discs, polished to reveal an equatorial section of the mineral grains and imaged using a cathodoluminescence capability on the scanning electron microscope (SEM) Jeol 5000, at the British Geological Survey, Keyworth.

Ablation was conducted using either a UP193FX (193nm) excimer or UP193SS (193nm) Nd:YAG laser ablation system (New Wave Research, UK) and an in-house designed low volume 'zircon ablation cell' (Horstwood *et al.* 2003) based on the design principles of Bleiner and Gunther (2001). Ablated sample material was transported from the laser cell into the MC-ICP-MS using a continuous flow of 0.8l/min of He gas. Operational parameters of the laser are detailed in Table 1.

The laser was fired using a repetition rate of 6Hz and a 25µm static spot size and a laser fluence of 1-2 $J.cm^{-2}$ or 3–4 $J.cm^{-2}$ due to core and rim uranium concentration variations and disparities between detrital grains. Locations of laser spots were guided by examination of the CL images. Typical ablation pit depths for fluence of 2-3 $J.cm^{-2}$ are of the order of 16µm, as measured previously using an SEM. However, because overgrowth rims were typically narrower than the laser spot size, an external surface rim rastering technique (Cottle *et al.* 2008) was employed on unpolished zircon grains. The aim of this technique was to obtain age data exclusively from the thin rims of grains, within a few microns of the top surface, thereby avoiding inherited Palaeozoic and older material in their core. The disadvantage of this technique is that the unpolished grains cannot be imaged before ablation, so CL information on zonation is lacking. Time –series evaluation of the data was, however, used to determine whether any older zone was penetrated during rastering.

Reference materials were analysed at regular intervals throughout the analytical session (typically 2-3 of each between each ablation run of unknowns). The internationally recognised 91500 zircon (Wiedenbeck *et al.* 1995) and GJ-1 (Jackson *et al.* 2004) were used as primary reference materials depending on the instrumental sensitivity and fluence used at the time. Plešovice (Sláma *et al.* 2008), GJ-1 and/or 91500 were variously used as appropriate secondary reference materials in order to validate the data for each day. As the data were collected in 21 separate analytical sessions spanning over a year, it has been possible to calculate a long-term reproducibility of ~2.5% (2SD) for Plešovice and GJ-1 over the whole analytical period (14/03/2012- 28/06/2012) (Figures S3.1 and S3.2).

Pb-U uncertainties in the data tables are propagated for excess variance as indicated by the primary reference material, whilst uncertainties for Pb-Pb are limited at a minimum of 0.25% (1σ) to allow for reproducibility of the multi-ion counting measurement. Both uncertainty types are without propagation for systematic components to allow relative comparison of similarly aged samples determined at the same time. All quoted age uncertainties reflect this and should be propagated in quadrature with the 2.5% (2SD) long term reproducibility defined in this study for comparison with other published ages. All data were processed using an in-house spreadsheet calculation routine. The data are presented as Wetherill plots or $^{206}Pb/^{238}U$ age relative probability plots generated on all data <5% discordant (in paper) and <25% discordant in the supplementary material for comparison with other studies, using Isoplot 4.15 (Ludwig 2003).

Table S3.1 Laboratory & Sample Preparation (as per recommended reporting guidelines of CIRDLES http://cirdles.org)	
Laboratory name	NERC Isotope Geosciences Laboratory (NIGL), United Kingdom
Sample type/mineral	Metamorphosed ash bed, orthogneisses / zircons
Sample preparation	Conventional mineral separation, 1 inch resin mount, 1um polish to finish
Imaging	CL, Jeol 5000, 10nA, 15mm working distance
Laser ablation system	
Make, Model & type	UP193FX (193nm) Excimer / UP193SS (193nm) Nd:YAG laser ablation system (New Wave Research, UK)
Ablation cell & volume	In-house low volume ablation cell, volume c.3cm^3
Laser wavelength (nm)	193nm
Pulse width (ns)	3-4ns
Fluence (J.cm-2)	1-3 J.cm^{-2}
Repetition rate (Hz)	5Hz
Spot size (µm)	25µm
Sampling mode / pattern	Single spots
Carrier gas	100% He, Ar make-up gas combined using a Y-connector 50% along sample line.
Ablation duration (secs)	30secs
Cell carrier gas flow (l/min)	0.8l/min
ICP-MS Instrument	
Make, Model & type	Nu Instruments, Nu Plasma HR, MC-ICP-MS
Sample introduction	Ablation aerosol combined with co-aspiration of desolvated Tl-$_{235}$U tracer
RF power (W)	1300W
Make-up gas flow (l/min)	0.7l/min Ar
Detection system	mixed Faraday-multiple ion counting array
Masses measured	202-207, 235, 238
Integration time per peak (ms)	200ms
Total integration time per data point (secs)	1 sec
Detection Efficiency (%, element)	0.4% U
IC Dead time (ns)	6, 9 & 7ns IC0, IC1 & IC2 resp.
Data Processing	
Gas blank	30 second on-peak zero subtracted
Calibration strategy	91500 or GJ-1 used as primary reference material, Plesovice & GJ1 or 91500 used as secondaries.
Long-term reproducibility	2.5% (2SD) on Plešovice over a one year period
Reference Material info	91500 (Wiedenbeck *et al.* 1995) Plešovice (Sláma *et al.* 2008) GJ1 ^{206}Pb/^{238}U 602.3 ± 1Ma, ^{207}Pb/^{206}Pb 609.2 ± 0.7Ma (in-house TIMS, unpublished)

Data processing package used / Correction for LIEF	Nu Instruments TRA software used to output data using average LIEF correction, In-house spreadsheet data processing used to reduce data and propagate uncertainties using the decay constants of Jaffey *et al.* 1971.
Mass discrimination	$^{207}Pb/^{206}Pb$ and $^{206}Pb/^{238}U$ normalised to reference material
Common-Pb correction, composition and uncertainty	No common-Pb correction applied
Uncertainty level & propagation	Ages are quoted at 2sigma absolute, propagation is by quadratic addition. Reproducibility and age uncertainty of reference material and common-Pb composition uncertainty are propagated.
Quality control / Validation	Plešovice – Weighted average $^{206}Pb/^{238}U$ age = 335 ± 1.5 (2SD, MSWD = 0.96) (Figure S3.1) GJ-1 – Weighted average $^{206}Pb/^{238}U$ age = 603.47 ± 0.94 (95% confidence limit, MSWD = 1.4) (Figure S3.2)

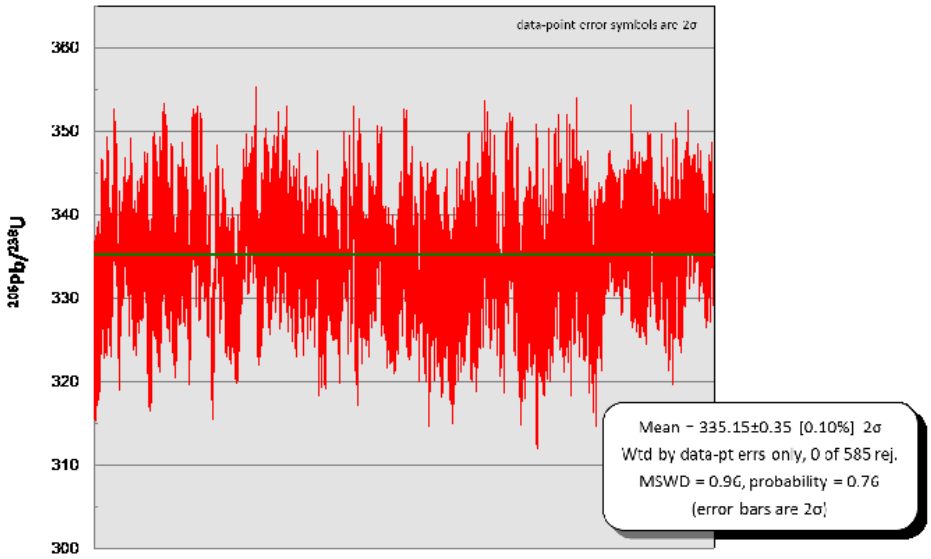

Figure S3.1 2.5% (2SD) long-term reproducibility of Plešovice (Sláma *et al.*, 2008) $^{206}Pb/^{238}U$ age determined as validation over the analytical period 14.03.2011 – 28.06.2012.

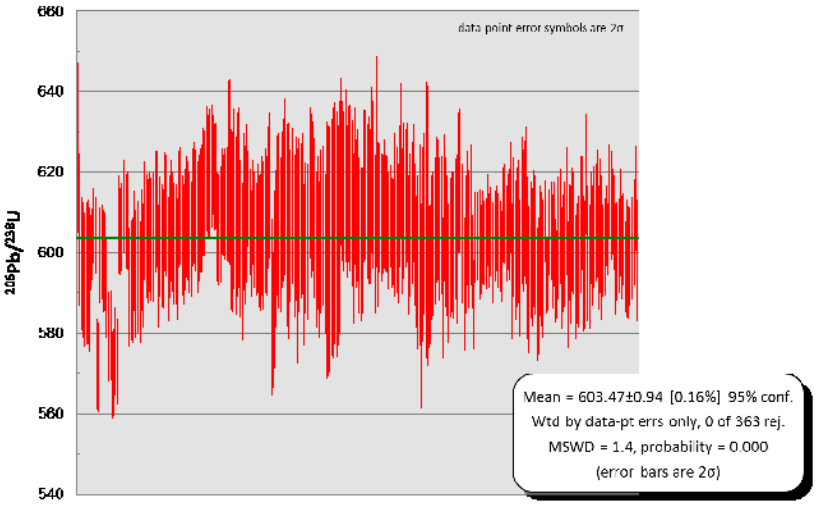

Figure S3.2 Long-term reproducibility of GJ-1 (Jackson *et al.*, 2004) $^{206}Pb/^{238}U$ age determined as validation over the analytical period 14.03.2011 – 28.06.2012.

3.4 U-Pb geochronology using AttoM LA-ICP-MS (NIGL, Keyworth, UK)

Some zircons were analysed for U-Th-Pb geochronology using a Nu Instruments Attom HR single-collector inductively coupled plasma mass spectrometer (HR-ICP-MS). Laser ablation was performed using the equipment stated before. The ablation parameters were a 25µm static spot, a repetition rate of 5Hz, a fluence of 1.5 to 2.5 J/cm2, a 15 second washout period between analyses, and a 30 second ablation time. The instrument was tuned to give ThO of <0.6%, and UO of <0.4%. Data processing used the time-resolved function on the Nu Instuments' software, an in-house Excel spreadsheet for data reduction and uncertainty propagation, and Isoplot for data presentation (Ludwig, 2003). All data are plotted at the 2σ confidence interval. Methodology followed that described in Thomas *et al.* (2013).

3.4.1 Sample preparation

Zircon and monazite minerals were separated from granite samples by crushing and milling followed by separation with a Rogers© table, heavy liquids and a Frantz© magnetic separator. The crystals were hand-picked and mounted in a polished epoxy disk to be imaged and analysed. Individual grains of zircon and monazite were imaged using cathodoluminescence and X-ray maps respectively. Zircons were imaged using the FEI Company QUANTA 600 Environmental Scanning Electron Microscope (ESEM) with a tungsten tetrode electron gun equipped with a Centaurus cathodoluminescence detector for visible-light range cathodoluminescence (CL) imaging at the British Geological Society. Prior to dating monazites were mapped for U, Y, Th and Ce at 1 μm^2 per pixel resolution on the Open University Cameca SX100 EMP in order to identify elemental zoning, assist laser spot location and facilitate age interpretation.

3.4.2 Analytical technique

U-(Th-)Pb geochronology was undertaken using laser ablation on mineral separates. Multi-collector inductively-coupled plasma mass spectrometry (MC-ICP-MS) using the Nu Plasma and single collector sector inductively-coupled mass spectrometry (ICP-MS) using the AttoM at NERC Isotope Geochemistry Laboratories (NIGL), UK were used for analysis.

(i) Laser spot analysis

In general laser spot sizes of 10 – 20 µm were used for analysis. Location of these spots was based on

grain images.

(ii) Rim rastering

In some cases a rim rastering technique (Cottle *et al.* 2008) was used on zircon grains, due to (a) the abundance of inherited zircon containing Palaeozoic and older cores and (b) the overgrowth rims being narrower than the spot size of the laser. The exterior surface of a rastered grain is seen in Figure S3.3. The advantage of rastering over the surface of grains is that the analysis is taken from material which originates from the very top surface, only a few microns deep.

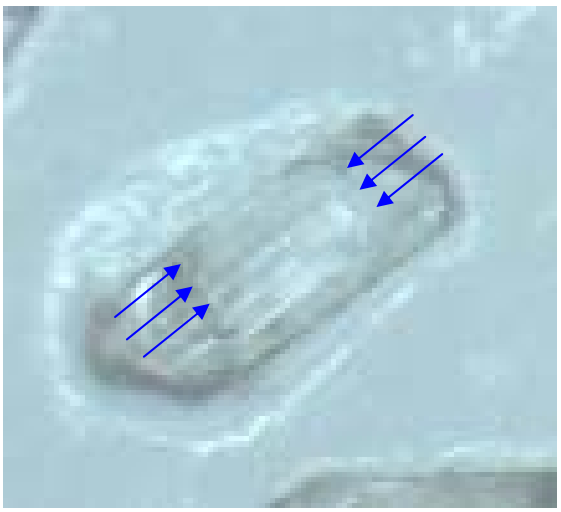

Figure S3.3. Image of the exterior of a zircon grain (approximately 100 µm in length) which has undergone rastering of the surface. Arrows point to ends of raster pit.

The disadvantage of this technique is that the grain cannot be imaged before ablation due to the unpolished nature of the surface and the lack of depth information it would reveal.

3.4.3 Data processing

Data reduction was undertaken using in-house spreadsheets; Concordia plots and Terra-Wasserburg diagrams were plotted using Isoplot version 3.16 September 2008 (Ludwig, 2001).

Data recording <<0.01 mV ^{207}Pb were rejected and data points with >300 cps ^{204}Pb after correction for the isobaric interference of ^{204}Hg were assessed with respect to the contribution of common Pb. The data are plotted uncorrected for common Pb.

U-Pb data for the zircon analysis were normalised to 1065 Ma "91500" (Wiedenbeck *et al.*, 1995) as a primary standard with 608.5 Ma "GJ-1" (Jackson *et al.*, 2004) as a secondary standard. Overall uncertainties achieved on the secondary reference material for each group of samples are detailed below.

U-Th-Pb monazite data were normalized to the 507.3 ± 0.7 Ma "Stern" monazite (NIGL TIMS dated in-house standard) primary reference. The 554 Ma "Manangotry" monazite (Paquette *et al.*, 1994) was used as a secondary reference, analyzed concurrently to monitor data accuracy. Overall uncertainties achieved on the secondary reference material for each group of samples are detailed below. All quoted uncertainties include contributions from the external reproducibility of the standard for ^{206}Pb/^{238}U and ^{208}Pb/^{232}Th ratios.

The excess ^{206}Pb age was calculated by Isoplot using a two-variable (^{206}Pb/^{238}U age versus ^{208}Pb/^{232}Th

age) linear regression fixed through ∞, ∞ (100,000 Ma, 100,000 Ma). A model 1 fit based on York (1969) was used for all samples; model 2 based assigning equal weight to each data point was avoided. The uncertainty values were used in the calculation which was undertaken using Isoplot.

Mean square of weighted derivatives (MSWD) is used as a measure of the scatter in the data and is reported for regressions and calculated ages based on multiple analyses. It provides a measure of the ratio of the observed scatter of the points to the expected scatter. If the MSWD is near unity the scatter is a result only of the assigned or measured errors and would be consistent with all measurements being the same composition with variation arising only from measurement uncertainty. MSWD >>1 indicates either that there is a component of non-analytical (i.e. real age) scatter or that the analytical errors are significantly underestimated. In cases where MSWD <<1 it is implied that either the analytical errors have been significantly overestimated or that there are unrecognised error-correlations.

Table S3.2 Laboratory & Sample Preparation (as per recommended reporting guidelines of CIRDLES http://cirdles.org)	
Laboratory name	NERC Isotope Geosciences Laboratory (NIGL), United Kingdom
Sample type/mineral	Metamorphosed ash bed, leucogranites / zircons
Sample preparation	Conventional mineral separation, 1 inch resin mount, 1/4um polish to finish; mounts for rim raster analysis left unpolished
Imaging	Cathodoluminescence
Laser ablation system	
Make, Model & type	New Wave Research UP193FX
Ablation cell & volume	In-house low volume ablation cell, volume c.3cm^3
Laser wavelength (nm)	193nm
Pulse width (ns)	3-4ns
Fluence (J.cm-2)	1.5-2.5 J.cm^{-2}
Repetition rate (Hz)	5Hz
Ablation duration (secs)	30secs
Ablation pit depth / ablation rate	~15µm pit depth, measured using an optical microscope; a few microns depth only for rim rastering
Spot size (µm)	25µm
Sampling mode / pattern	Static spot ablation; surface rastering
Carrier gas	100% He, Ar make-up gas combined using a Y-connector 50% along sample line.
Cell carrier gas flow (l/min)	0.8l/min
ICP-MS Instrument	
Make, Model & type	Nu Instruments, Attom SC-SF-ICP-MS
Sample introduction	Free air aspiration via desolvator
RF power (W)	1300W
Make-up gas flow (l/min)	0.8l/min Ar
Detection system	Discrete dynode MassCom ion counter
Masses measured	202, 204, 206, 207, 208, 232, 235
Integration time per peak	200µs (202, 204, 206, 208, 232), 500µs (207, 235)
Total integration time per data point (secs)	0.22 seconds
Detection Efficiency (%,	~0.27% for Uranium

element)	
IC Dead time (ns)	15ns
Data Processing	
Gas blank	60 second on-peak zero subtracted
Calibration strategy	91500, GJ-1 & Plešovice (zircon); One used as primary reference material for normalization, and others for validation.
Reference Material info	91500 (1065 Ma; Wiedenbeck *et al.* 1995) Plešovice (337.13 Ma; Sláma *et al.* 2008) GJ1 ($^{206}Pb/^{238}U$ 602.3 ± 1Ma, $^{207}Pb/^{206}Pb$ 609.2 ± 0.7Ma; in-house TIMS, see also Jackson *et al.* 2004
Data processing package used / Correction for LIEF	Nu Instruments TRA acquisition software, in-house spreadsheet data processing
Mass discrimination	$^{207}Pb/^{206}Pb$ and $^{206}Pb/^{238}U$ normalised to reference material
Common-Pb correction, composition and uncertainty	No common-Pb correction applied
Uncertainty level & propagation	Ages in the data table are quoted at 2sigma absolute, propagation is by quadratic addition. Reproducibility of reference material is propagated.
Quality control / Validation	Plešovice – Weighted average $^{206}Pb/^{238}U$ age = 340 ± 2 (2SD, MSWD = 0.36) Figure S3.4 GJ-1 – Weighted average $^{206}Pb/^{238}U$ age = 601.5 ± 3.7 (2SD MSWD = 0.73) Figure S3.5

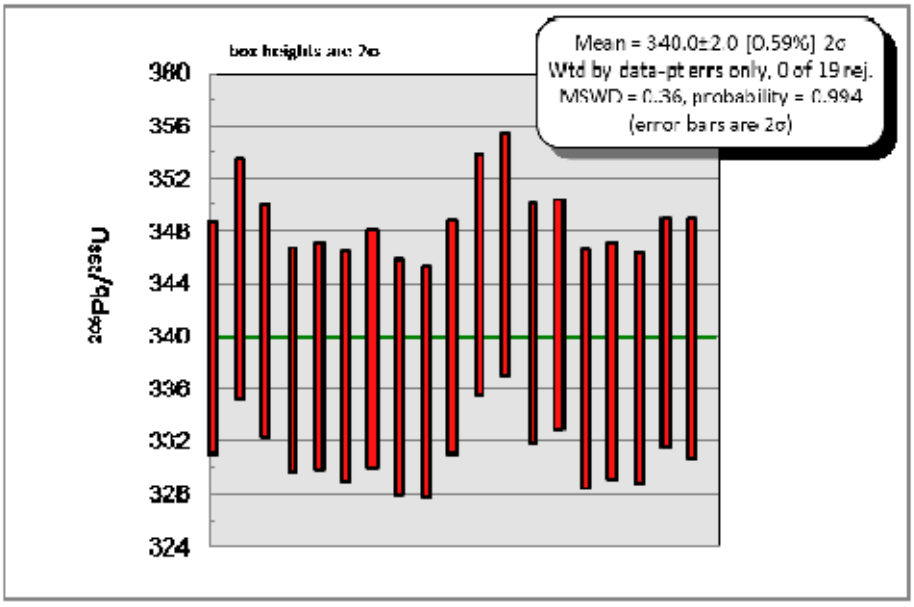

Figure S3.4 Reproducibility of Plešovice (Sláma *et al.*, 2008) $^{206}Pb/^{238}U$ age determined as validation over the analytical period 14.03.2011 – 28.06.2012 on the AttoM ICP-MS.

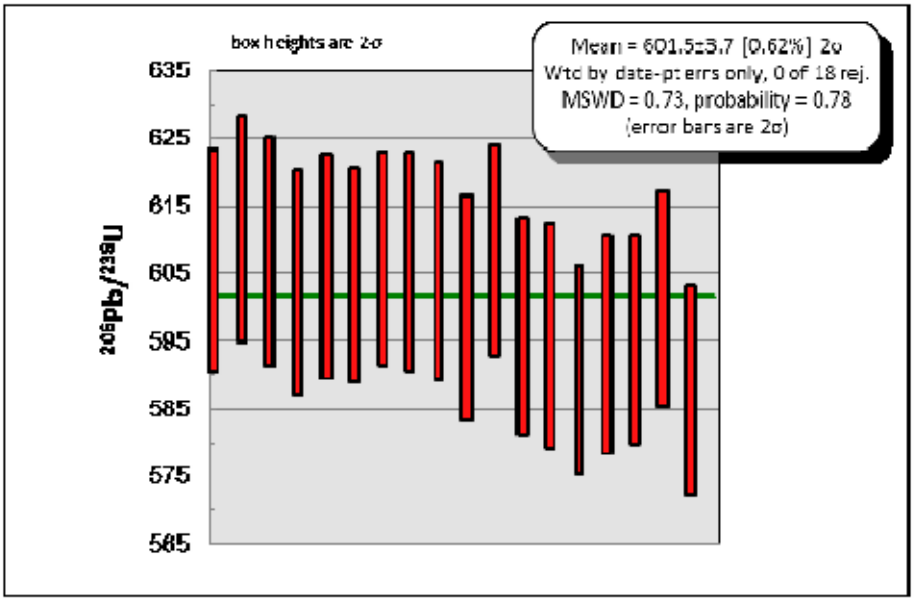

Figure S3.5 Reproducibility of GJ-1 (Jackson *et al.*, 2004) $^{206}Pb/^{238}U$ age determined as validation over the analytical period 14.03.2011 – 28.06.2012 on the AttoM ICP-MS.

4. References

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