

## **U-Th-Pb ANALYSIS USING THE EDINBURGH CAMECA IMS-1270: ANALYTICAL METHODS**

### **Sample preparation**

Zircons are separated using traditional techniques - rock samples are initially crushed and sieved, with zircon grains then concentrated using heavy liquids and magnetic separation. Hand picked zircon grains are mounted in epoxy along with fragments of the “91500” and “Temora 2” zircon standards. Each grain mount is hand ground and polished on clean laps until grain centres were exposed. The epoxy mounted zircon grains are imaged in detail using transmitted and reflected light microscopes, and the Phillips XL30 Scanning Electron Microscope (SEM) located at the School of GeoSciences, University of Edinburgh. Cathodoluminescence (CL) images (15kV) and Backscattered Electron (BSE) images (20kV) are collected at relatively low magnification to limit burning of the epoxy mount surface surrounding grains. Prior to ion microprobe analysis, mounts are re-polished using felt laps to remove any carbon coating present for SEM analysis then cleaned by placing mounts consecutively in beakers of petroleum spirit and methanol placed in ultrasonic baths. Mounts are bathed in low concentration Nitric acid (2%) and rinsed with deionised water before gold coating.

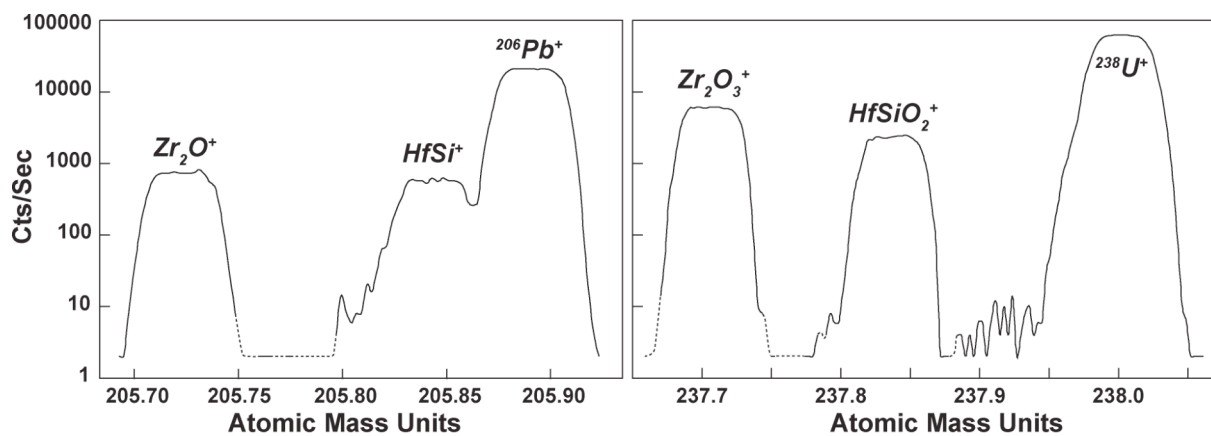
### **Analytical procedure**

The following describes in detail the procedure employed for U/Pb dating of zircons at the Edinburgh Ion Microprobe Facility (EIMF), using the CAMECA ims-1270 ion microprobe located in the Material and Micro-Analysis Centre (EMMAC), School of GeoSciences, University of Edinburgh. Analytical procedures are similar to those described by Schuhmacher et al. (1994) and Whitehouse et al. (1997).

Zircons are analysed using a  $\sim 4\text{nA O}_2^-$  primary ion source with 22.5 keV net impact energy. The beam is focussed using Köhler illumination, which ensures uniform beam density. The

primary beam is mass analysed to ensure a pure beam of  $O_2^-$  ions and to eliminate  $O_2H^-$  from the source. An additional lens immediately after the ion source enables a constant beam density and current to be maintained despite long term changes in the output from the duoplasmatron source. The primary beam alignment gives ellipsoidal analysis pits ( $\sim 25 \mu\text{m}$  max. dimension) with sharp edges and flat bottoms. Clean analysis pits are considered essential to reduce peripheral contamination and allow even *sputtering* over the entire analysed area. Further effects of peripheral contamination are minimised by a field aperture that restricts the secondary ion signal to a  $\sim 15\mu\text{m}$  square at the centre of the analysis pit.

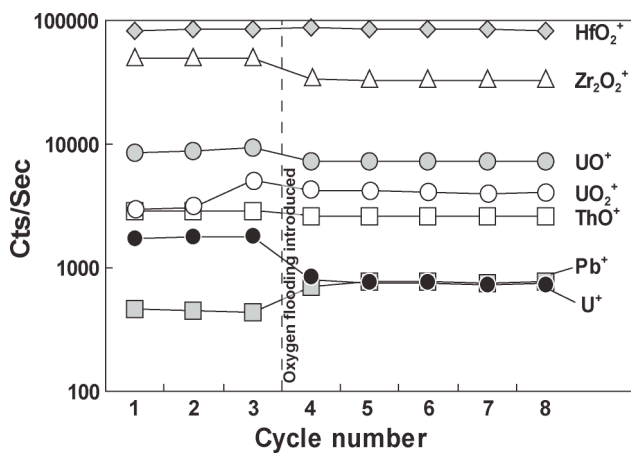
In the secondary ion optics a 60 eV energy window is used together with slit widths (60 $\mu\text{m}$  entry and 190 $\mu\text{m}$  exit) with a measured mass resolution of  $\sim 4000R$  (at 1% peak height) to separate  $Pb^+$  peaks from known molecular interferences (except hydrides). At this mass resolution the tail from the  $HfSi$  peak may potentially overlap the  $Pb$  isotope peaks (Fig. A1).



**Appendix Figure 1:** Mass scan of the regions close to masses 206 and 238, showing resolution of  $^{206}Pb^+$  and  $^{238}U^+$  from molecular interferences ( $Zr_2O^+$  and  $HfSi^+$ ,  $Zr_3O_3^+$  and  $HfSiO_2^+$ ) at a mass-resolution of  $\sim 4000$  (1% peak height).

However, by measuring the tail of intense peaks in the  $Pb$  region it can be shown that the maximum contribution to the total  $Pb$  concentrations during analysis of zircon with  $\sim 2 \text{ wt}\%$   $HfO_2$ , will be the equivalent of  $<0.16 \text{ ppb}$  ( $^{206}Pb$ ) and  $<0.064 \text{ ppb}$  ( $^{204}Pb$ ), and were therefore considered to be a negligible contribution. The instrument is operated in “rectangular mode”

(de Chambost et al., 1996) to maintain optimum conditions for flat-topped peaks (Fig. A1). Sensitivity for Pb in zircon as analysed by ims-1270 varies slightly depending on instrument tuning but was always  $\geq 12$  cps / ppm  $^{206}\text{Pb}^+$  / nA using an  $\text{O}_2^-$  beam). This is slightly lower than that quoted for the ASI SHRIMP ( $>18$  cps / ppm  $^{206}\text{Pb}^+$  / nA using an  $\text{O}_2^-$  beam; data source: <http://www.asi-pl.com/files/pages/shrimpfiles/shrimpOverview.pdf>). However, the addition of oxygen flooding on the surface of the sample (at  $2 \times 10^{-5}$  mbar gas pressure), a process currently unique to the CAMECA instrument, leads to a two-fold increase in the yield of Pb ( $\sim 24$  cps / ppm  $^{206}\text{Pb}^+$  / nA), with a slight reduction in U ion yield. In contrast, the ion yields of UO and  $\text{UO}_2$  increase with oxygen flooding, the  $\text{UO}_2$  signal change closely



**Appendix Figure 2:** Changes to ion yield of selected ions as a result of oxygen flooding during an analysis.

matching that of Pb (Fig. A2).

Secondary ion intensities are measured using an electron multiplier in ion-counting mode; the dead-time is electronically set at 51 nS. Detector

background measurements are made at mass 204.3 and as they are typically so

low (0.01-0.03 cps) they are averaged for

an entire analytical session (normally up to 7 days) and removed from all signals prior to data reduction. Systematic variation or spikes in background intensity are monitored, and in the unusual case of higher than average background counts an assessment is made on an analysis-to-analysis or sample-to-sample basis.

At the start of every analysis point, the secondary ion beam is centred on the field aperture to compensate for any changes in the primary beam position relative to the secondary ion optics.

The position of mass  $^{180}\text{Hf}^{16}\text{O}$  is used to align all other masses. The secondary energy

distribution is scanned automatically at the beginning of each analysis and if necessary a small offset applied (typically <5 eV) to compensate for any minor charging on the sample surface. Prior to measurement, a 15  $\mu\text{m}$  raster is applied on the sample surface for 120 seconds to remove any surface contamination around the point of analysis (total diameter of cleaned area: 40  $\mu\text{m}$ ). Twenty cycles are made through the masses of interest, with the first 5 cycles discarded to further reduce possible near-surface contamination of common Pb.

Masses measured are:  $^{196}\text{(HfO)}$ ,  $^{204}\text{Pb}$ ,  $^{204.3}\text{(background)}$ ,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{208}\text{Pb}$ ,  $^{212}\text{(Zr}_2\text{O}_2\text{)}$ ,  $^{238}\text{U}$ ,  $^{248}\text{(ThO)}$ ,  $^{254}\text{(UO)}$ , and  $^{270}\text{(UO}_2\text{)}$  (see Appendix Table 1 for counting times on individual masses). Total time per analysis is 28 minutes.

### **Calibration of Pb/U ratios**

Calibration of Pb/U ratios follows a variant of procedures employed by SIMS (SHRIMP or Cameca ims-1270) dating facilities elsewhere. Corrections have previously been based on observed relationships between Pb/U or Pb/UO<sub>2</sub> and the ratios of uranium oxide(s) to elemental uranium or each other (e.g. UO/U, UO<sub>2</sub>/U or UO<sub>2</sub>/UO; Compston et al., 1984; Williams and Claesson, 1987; Schuhmacher et al., 1994; Whitehouse et al., 1997; Williams, 1998). It is assumed that the linear arrays observed in such ratio plots are generated by differences in secondary ion energy of these species or by changes in the primary beam density. SHRIMP measurement procedures typically employ the relationship between Pb/U vs UO/U (e.g. Compston et al., 1984; Williams and Claesson, 1987; Claoué-Long et al., 1995), in which the data distribution can be modelled using a quadratic or power law function: using  $\ln(\text{Pb}/\text{U})$  vs  $\ln(\text{UO}/\text{U})$  this defines a near linear array of slope equal to  $\sim 2$ . Whitehouse et al (1997), using an ims-1270, found that better correlations are obtained using Pb/U vs UO<sub>2</sub>/U, rather than Pb/U vs UO/U. They suggested that the higher degree of correlation may be related to close similarity in the shape of the energy spectra for Pb and UO<sub>2</sub>. Because the range of UO<sub>2</sub>/U ratios measured in both the standard and unknowns is small, these authors

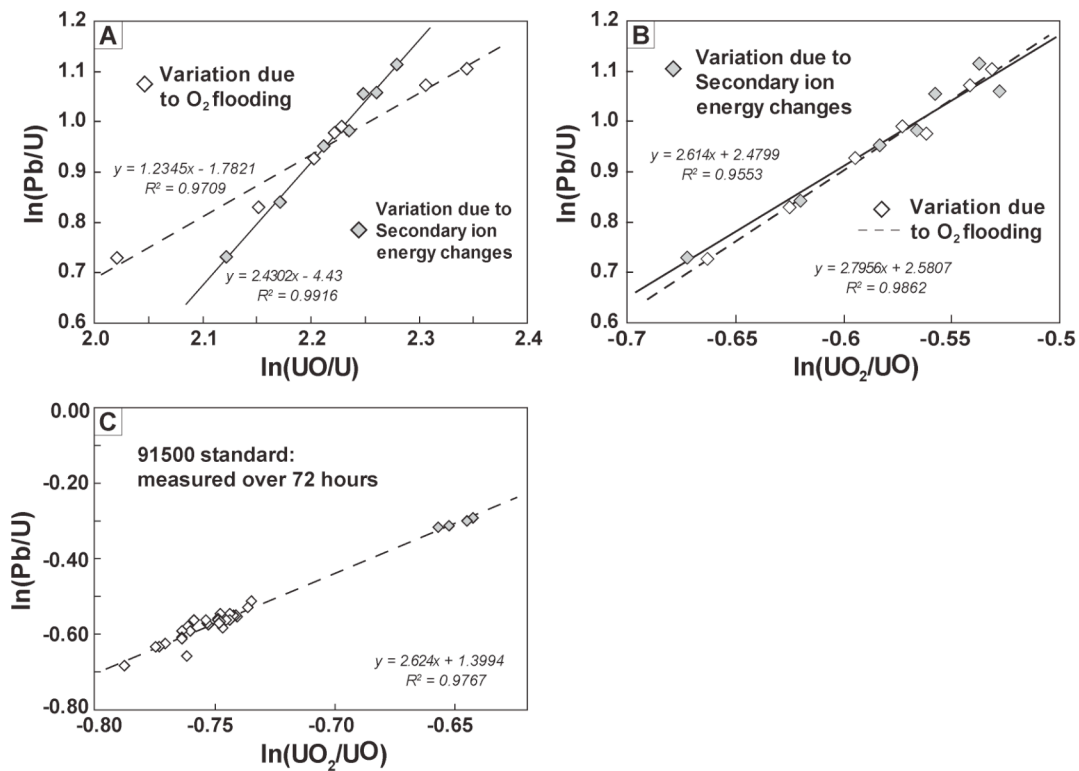
approximated this relationship with a linear function, although have more recently employed a power law relationship (see Whitehouse & Bridgwater, 2001). An alternative that was originally employed by Hollinger (1992), and subsequently by Stern and Amelin (2003) and Compston (2004), is a calibration based on Pb/UO<sub>2</sub> vs UO/U. In addition, Compston (2004) found that correlations between U, UO and UO<sub>2</sub> provide a direct empirical measure of the between-analysis standard deviation of SIMS secondary ion ratios that is independent of the uniformity of Pb/U age within the reference zircon.

In assessing which pairing is most appropriate for the Edinburgh ims1270, a number of comparisons have been made. It has been found that whilst the conventional ln(Pb/U) vs ln(UO/U) method gives linear arrays, the slopes are generated by changing oxygen flooding or the sample voltage (which effectively changes the energy of the secondary ions recorded) are significantly different (Fig. A3a). Interestingly, changes observed on varying the amount of oxygen flooding give a slope closest to the canonical value of 2, suggesting much of the changes in ionisation observed from point to point are related to changes in oxygen presence in the analysed area rather than changes in energy (see also Whitehouse et al., 1997).

It is found that variations in ln(Pb/U) vs ln(UO<sub>2</sub>/UO) caused by changes in oxygen flooding or secondary ion energy not only give good linear arrays but, importantly, the slopes of the arrays are similar (~2.7 and ~2.6, respectively; Fig. A3b). The value of 2.6 (e.g. Fig. A3c) has subsequently been determined to give the lowest errors for variations in either repeats of individual samples or for the comparisons of two standards (e.g. Temora and 91500). The data correction equation employed is therefore:

$$(\text{Pb/U})_{\text{corrected}} = (\text{Pb/U})_{\text{measured}} / [\ln(\text{UO}_2/\text{UO measured})]^{2.6}$$

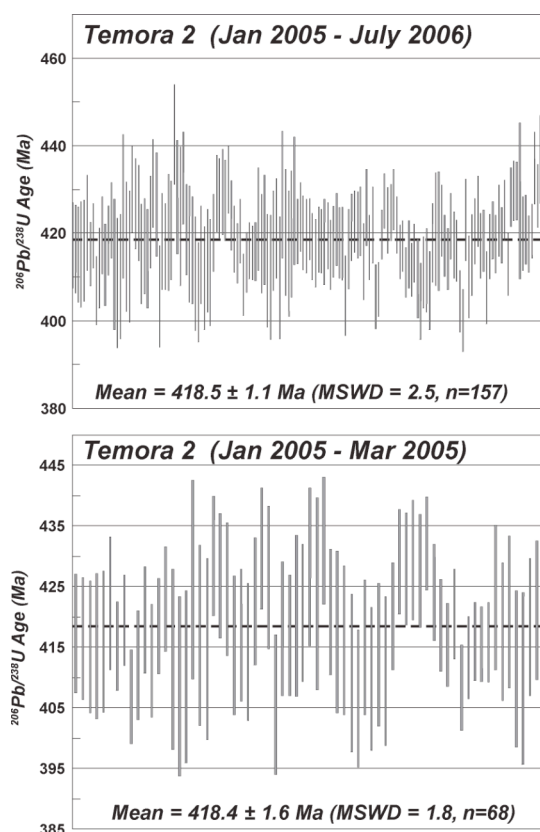
The corrected average Pb/U ratios for a single day or session for the main standard 91500 is then equal to 0.17917 and all other U/Pb ratios are scaled to this value. The use of this relationship in preference to the conventional  $\ln(\text{Pb}/\text{U})$  vs  $\ln(\text{UO}/\text{U})$  method has increased the within-session reproducibility of our own analyses of the standard by approximately a factor of 2.



**Appendix Figure 3:** (a) Difference between variations of Pb/U vs UO/U ratios created by changing the surface oxygen concentrations (the effect of which is similar to changing the primary beam density) and those created by changes in secondary ion energy measured. (b) Variations in Pb/U vs  $\text{UO}_2/\text{UO}$  ratios created by changing the surface oxygen concentrations and secondary ion energy measured. Note that by using  $\text{UO}_2/\text{UO}$  instead of UO/U similar slopes are generated by both processes. (c)  $\ln(\text{Pb}/\text{U})$  vs  $\ln(\text{UO}_2/\text{UO})$  plot showing variations in measurement of the 91500 standard zircon over a 3 day continuous “session”. Towards the end of the session the primary beam source intensity changed, which was compensated by changes in the primary beam lens settings to maintain constant beam current (shaded diamonds). The figure illustrates that although the  $\text{UO}_2/\text{UO}$  ratio changes as a function of the changed instrument conditions, the corrected Pb/U remained constant.

## Standard analysis and measurement of Pb/U ratios

U/Pb ratios are calibrated against measurements of the Geostandards 91500 zircon (Wiedenbeck et al., 1995: ~1062.5 Ma; assumed  $^{206}\text{Pb}/^{238}\text{U}$  ratio = 0.17917), which is measured after 3-4 unknowns. Measurements over a single “session” (a period in which no tuning or changes to the instrument have occurred) give a standard deviation on the  $^{206}\text{Pb}/^{238}\text{U}$  ratio of individual repeats of 91500 of about 1% ( $1\sigma$ ). A single analysis of Temora 2, which is used as a secondary or external reference standard, also was made approximately every 2½-3 hours. Although the age of Temora 1 is given by Black et al., (2003) to be  $416.75 \pm 0.24$  Ma ( $2\sigma$ ), Black et al. (2004) noted that Temora 2, nominally of the same ID-TIMS age ( $416.78 \pm 0.33$  Ma;  $2\sigma$ ), gave an age of  $418.1 \pm 1.6$  Ma ( $2\sigma$ ) when analysed by SIMS and referenced to



**Appendix Figure 4:** Reproducibility of measurements on the Temora 2 standard made during a number sessions between January 2005 and July 2006 (upper) and during analytical sessions related only to the South Harris stud (lower: January to March 2005).

Temora 1. Over the period January 2005 to July 2006 our analyses of Temora 2 give a mean  $^{206}\text{Pb}/^{238}\text{U}$  ratio of  $0.06708 \pm 0.00018$  (MSWD = 2.5; 95% conf.), corresponding to an age of  $418.5 \pm 1.1$  Ma (MSWD = 2.5;  $n=157$ ; Fig. A4a). During the analytical sessions involved the South Harris study, Temora 2 zircon has yielded a mean  $^{206}\text{Pb}/^{238}\text{U}$  ratio of  $0.06706 \pm 0.00026$  (MSWD = 1.8; 95% conf.;  $418.4 \pm 1.6$  Ma;  $n = 68$ ; Fig. A4b). This discrepancy between ages as determined by ID-TIMS and SIMS may reflect matrix effects, or alternatively, be due in this work to the assigned age for the

91500 standard zircon (1062.5 Ma; Wiedenbeck et al., 1995) being too old. Overall the analyses of 91500 and Temora 2 suggest that the observed uncertainty in the Pb/U ratio is larger than that predicted from counting statistics by an additional 0.3%. This additional apparent error is included in all analyses by adding 0.5% in quadrature to the error assigned to the Pb/U ratios. The uncertainty of the  $^{206}\text{Pb}/^{238}\text{U}$  measured on the standard for each session is included in the  $^{206}\text{Pb}/^{238}\text{U}$  error given for the unknowns.

### **Calculation of Th/U ratios and elemental abundances**

The  $^{208}\text{Pb}/^{206}\text{Pb}$  ratio changes only very slowly with geological time (assuming a constant Th/U ratio). Therefore for zircons the  $^{208}\text{Pb}/^{206}\text{Pb}$  and Th/U ratio cannot commonly be measured with sufficient precision to determine the age of the sample. However, for a single age population sample, a high degree of correlation is expected between the Th/U and the measured  $^{208}\text{Pb}/^{206}\text{Pb}$  ratios.

The  $\text{ThO}^+/\text{UO}^+$  ion ratios closely reflect the actual Th/U ratios of the zircon. Although this ratio can be changed by unusually large variations in either the secondary ion energy or oxygen flooding, the ratio can be shown to be largely unaffected by the analytical changes that occur during one analytical session. However, since the  $\text{ThO}^+/\text{UO}^+$  ratio does not give exactly the Th/U ratio expected from the measured  $^{208}\text{Pb}/^{206}\text{Pb}$  ratio for either the standards or the unknowns, a correction must be applied. Although the Th/U ratio is reasonably well known for the bulk 91500 standard, there is sufficient variation within this standard that the bulk value cannot be used directly. The Th/U ratio is corrected assuming that the zircon standard is concordant for both U and Th generated Pb. A small constant correction is made to the measured average Th/U ratio of the standard (measured as  $\text{ThO}^+/\text{UO}^+$ ) to give the Th/U ratio predicted from the average  $^{208}\text{Pb}/^{206}\text{Pb}$  ratio. The correction is generally a few percent and always less than 10%.



U concentrations are determined based on an observed  $\text{UO}_2 / \text{Zr}_2\text{O}_2$  ratio of standard 91500, assuming a U content of 81.2 ppm. All other elemental concentrations (Pb and Th) are calculated relative to this value. The variability of the calculated U concentration for 91500 is ~5%, some of which may be natural variation. Hf concentrations are determined based on the observed  $\text{HfO} / \text{Zr}_2\text{O}_2$  ratio of standard 91500, assuming not matrix effects, a Hf content of 5880 ppm and that  $\text{SiO}_2 + \text{ZrO}_2 + \text{HfO}_2$  sum to 100%.

### **Common Pb correction**

Common Pb contributions to analyses is primarily assumed to be derived from surface contamination of the sample. Therefore non-geological contributions to common Pb are reduced by clean sample preparation, rastering the sample prior to isotope measurement, and by production of flat-bottom analysis pits through carefully tuned beam conditions. However, common Pb from geological sources (i.e. within the zircon itself) may also be present at detectable abundances in some grains.

It has been very occasionally observed that the corrections for common lead gives low values for the average  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios measured for the 91500 standard. This is most easily explained by an overcorrection for common lead. In the worst case the overcorrection of the apparent common lead at mass 204 is calculated to be equivalent to less than 3 additional counts per analysis. The cause of these additional counts are difficult to determine but could include tail of neighbouring molecular peaks from the zircon matrix, contributions from molecular ions generated from the Au coating, incorrect isotopic abundance of the common lead or even  $^{204}\text{Hg}$ . Whilst this excess is small (equivalent to  $<1$  ppb  $^{\text{total}}\text{Pb}$ ), and therefore having an almost negligible effect on old samples (e.g.  $>600$  Ma), if ignored it could have a significant impact on the  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios of young samples. Where excess counts at mass 204 are detected during the measurement of the standard, a correction is applied to both standards and unknowns. In the data presented here only two sessions on the temora

standards had detectable additional counts at mass 204, none of the unknowns are corrected for any additional 204 counts.

Correction for *in-situ* common Pb has been made using measured  $^{204}\text{Pb}$  counts above that of detector background (and in a few cases the additional instrumental background as noted above) and using modern day composition of common Pb. A modern lead correction is made under the assumption that in nearly all cases measured common Pb results from contamination on the sample surface and in exposed cracks. The measured background count rates of  $^{204}\text{Pb}$  are typically in the range 0.03-0.08 cps (0.2 to 1.5 ppb), therefore commonly approaching the background recorded for the electron multiplier. At this level errors on the  $^{206}\text{Pb}$  correction are likely to be <15 ppb. For very young samples (<100 Ma) the corrections based on the  $^{204}\text{Pb}$  lead are assumed to be too inaccurate to correct individual analyses. In these cases the attributable common Pb is predicted using the excesses observed in the  $^{208}\text{Pb}/^{206}\text{Pb}$  ratio above that predicted from the U/Th ratio and the approximate age. This correction is applied if the measured  $^{207}\text{Pb}$  falls below 0.15 ppm. For a general comparison using this method, the KIM and SUE (kimberlitic) zircon 'oxygen' standards have been analysed, giving mean ages of  $91.5 \pm 4.6$  Ma ( $2\sigma$ ,  $n=9$  containing 0.1 ppm Pb) and  $49.9 \pm 1.6$  Ma ( $2\sigma$ ,  $n=7$ , containing 0.05 ppm Pb) respectively. The average age of the KIM zircon obtained in our tests is similar to that quoted by Cavosie et al. (2005) of  $92 \pm 3$  Ma ( $2\sigma$ ,  $^{208}\text{Pb}$  corrected).

At these common Pb levels, and assuming 200 ppm U content, a 1 Ma zircon will contain 30 ppb  $^{206}\text{Pb}$  and the  $^{206}\text{Pb}/^{238}\text{U}$  ratio (after correction) should be measurable with a precision of <30%. At 40 Ma, and assuming 50 ppm U, the common lead correction should permit precisions of <2% in the measurement of the  $^{206}\text{Pb}/^{238}\text{U}$  ratio.

## Data reduction procedure

Data produced by the ims-1270 instrument for each analysis are processed offline using in-house data reduction spreadsheets developed by R.W. Hinton. Within the spreadsheets, raw counting data are initially corrected for dead-time on the electron multiplier (fixed value of 51 nsec). As measurement of  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios on NIST SRM-610 and Equal-atom Pb glass standards using both the ims-4f and ims-1270 instruments indicates that mass fractionation is 2‰ / mass unit. In general, all count rate intensities increase slowly with time during an analysis. A linear correction is applied for the intensity drift on all masses with time, and is based on variations in count rate of  $^{196}\text{(HfO)}$  over the analysis (correction is typically <0.1% on any one ratio). Although slight differences in drift with time are observed between individual species the estimated error created by this effect is less than 0.01% in the  $^{207}\text{Pb}/^{206}\text{Pb}$  ratio. Errors introduced will in general be significantly less than any measurement error of the Pb intensity drift with time, especially for low lead samples. Following exclusion of the first 5 cycles through the masses, and correction for dead time and intensity drift, average ratios are calculated from the remaining 15 cycles. The standard deviation of these ratios are determined from the observed variation over the 15 cycles, with the exception that single cycles are excluded when variations in the measured Pb/U ratio are greater than  $2\sigma$ .

Common Pb corrections are applied based on observed  $^{204}\text{Pb}$  count rates of individual analyses. As noted above an additional background correction may be also applied to the mass 204 count rates. Instrument independent Pb/U ratios are calculated using the  $\ln(\text{Pb}/\text{U})$  vs  $\ln(\text{UO}_2/\text{UO})$  correction, and Th/U ratios and elemental abundances (U, Th, Pb and Hf, in ppm) calculated. Subsequent data reduction follows the grouping of standard and unknown data into “sessions” that reflect a period of constant instrument conditions. Such “sessions” can consist of analyses made over <24 hours or over several days depending on instrument performance. Once the group of standards are established, reference is made to the average

values of  $^{206}\text{Pb}/^{238}\text{U}$ , concentrations and Th/U ratios determined for the standard. Data integrity of individual analyses are evaluated using the concentration of common Pb and the level of  $^{206}\text{Pb}$  correction.

### **Uncertainties**

Uncertainties on the Pb/U ratios includes an error based on observed uncertainty from each measured ratio. This is generally close to that expected from counting statistics. The uncertainty in the U/Pb ratio given for individual points includes the measured uncertainty in the UO/UO<sub>2</sub> ratio (as part of the U/Pb vs UO/UO<sub>2</sub> calibration procedure), uncertainty on the common lead correction and the uncertainty on the standard measured for each session. Observed uncertainties on the U/Pb ratio of the standard zircon is generally some 0.3% in excess of that expected from counting statistics alone. This is assumed to be a random error (see also Ireland and Williams, 2003) and has been propagated (in both standards and unknowns) together with the observed variation in Pb/U ratios measured for each analysis (typically close to the counting errors). For measurement of the 91500 standard uncertainties are typically between 0.7 – 1.0% per analysis. Uncertainty on analyses of the secondary standard (Temora 2) with this additional correction is close to that predicted. The uncertainty introduced in the correction for common lead is included in the uncertainty on corrected  $^{207}\text{Pb}/^{206}\text{Pb}$  values, which are based on observed variations from cycle to cycle during each analysis and commonly approach those expected from counting statistics. Uncertainties on ages quoted in the text and in tables for *individual analyses* (ratios and ages) are at the  $1\sigma$  level. All uncertainties in calculated *group* ages are reported at 95% confidence limits. Plots and age calculations have been made using the computer program ISOPLOT/EX v3 (Ludwig, 2003).

**Table 1: count times for the masses during U-Th-Pb isotopic analysis using the EMIF Cameca ims-1270**

	HfO	<sup>204</sup> Pb	B/gr	<sup>206</sup> Pb	<sup>207</sup> Pb	<sup>208</sup> Pb
<b>Mass</b>	195.941	203.973	204.300	205.974	206.976	207.977
<b>Count time (secs)</b>	2	5	5	5	10	10
<b>Wait time between peaks (secs)</b>	2	1	1	1	1	1

	Zr <sub>2</sub> O <sub>2</sub>	<sup>238</sup> U	<sup>232</sup> ThO	<sup>238</sup> UO	<sup>238</sup> UO <sub>2</sub>
<b>Mass</b>	211.799	238.051	248.033	254.046	270.041
<b>Count time (secs)</b>	5	5	3	2	2
<b>Wait time between peaks (secs)</b>	1	1	1	1	1
<b>Total analysis time ~28 minutes</b>					

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