New approaches to U-Th-Pb zircon dating using the Cameca ims-1270

NATURAL ENVIRONMENT RESEARCH COUNCIL

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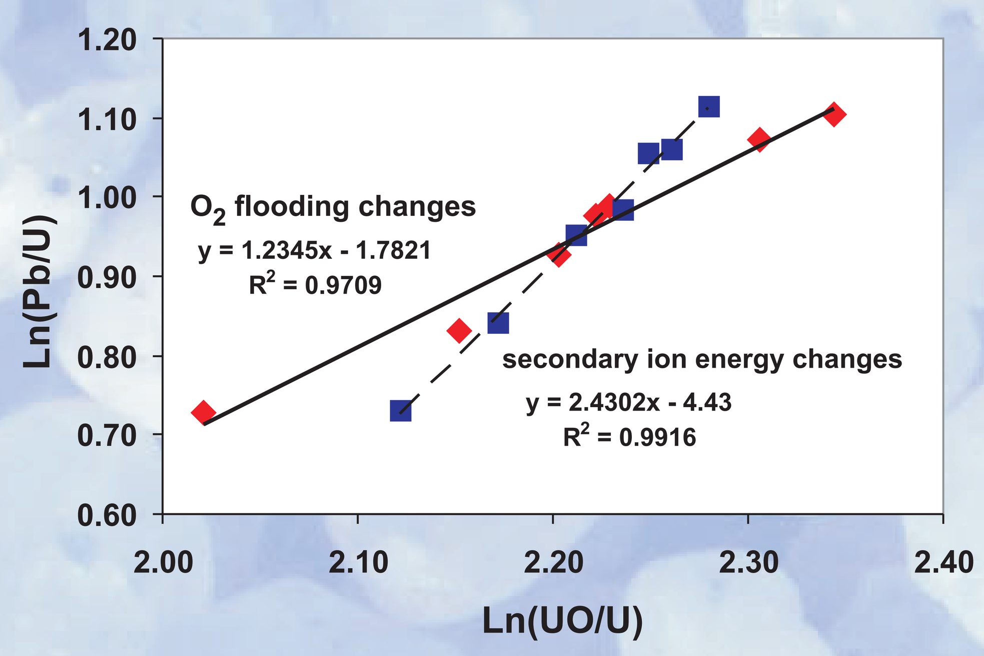
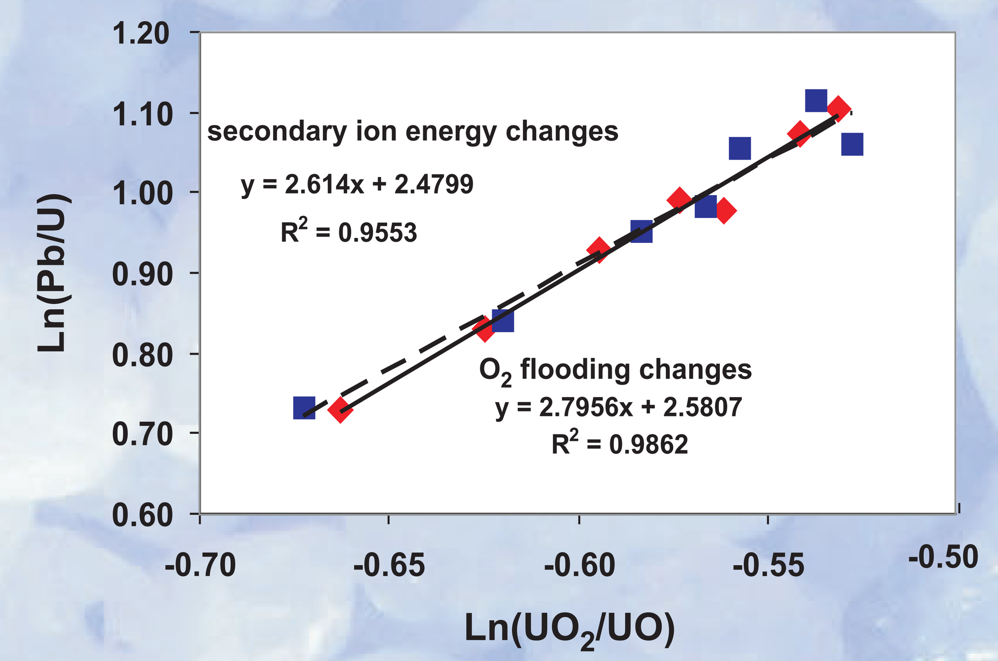
Introduction

New methods have been established for the Cameca ims-1270 to optimise Pb yield and provide precise and accurate Pb/U measurement. With the Cameca ims-1270 instrument we have attempted to find the analytical conditions, and correction procedures, which show the least sensitivity to changes in instrumental conditions. We have also attempted to use standards mounted separately from unknowns. Conventionally instrumental changes in measured Pb/U ratios are corrected using observed correlations between the Pb/U and UO/U ratios. Following, work by Schuhmacher et al. (1994), Whitehouse et al (1997), Stern and Amelin (2003) and Compston (2004) we have found that including UO_2 improves correction procedures and suggest a new correction procedure based on U/Pb vs UO_2/UO:

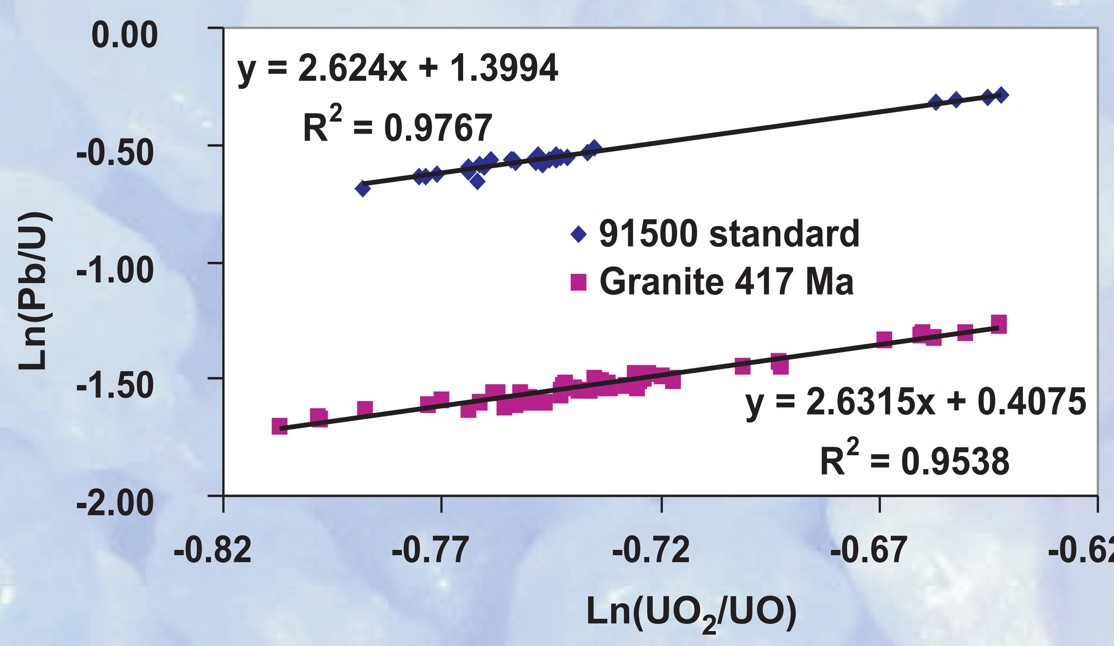
$$\ln\ Pb/U_{corrected} = \ln\ Pb/U_{measured} - ((\ln\ UO_2/UO_{measured} - Constant) \times 2.6)$$



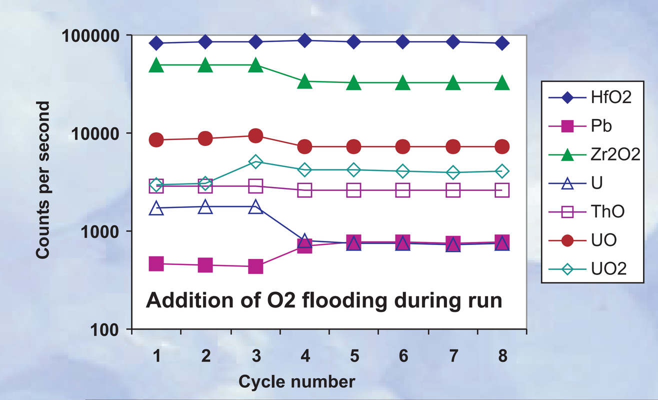
Changes in the amount of oxygen flooding on the surface or the energy of the secondary ions create linear arrays of Pb/U vs UO/U ratios. However, the arrays for energy and flooding have different slopes and cannot be corrected by a constant factor.



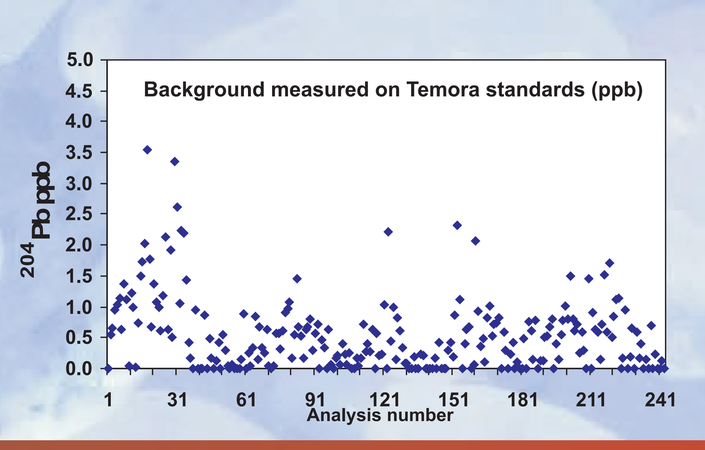
Changes in amount of oxygen flooding on the surface or the energy of the secondary ions create linear arrays of Pb/U vs UO_2/UO ratios which lie on similar arrays. Corrections for more than one instrument effect can be made using a constant factor.



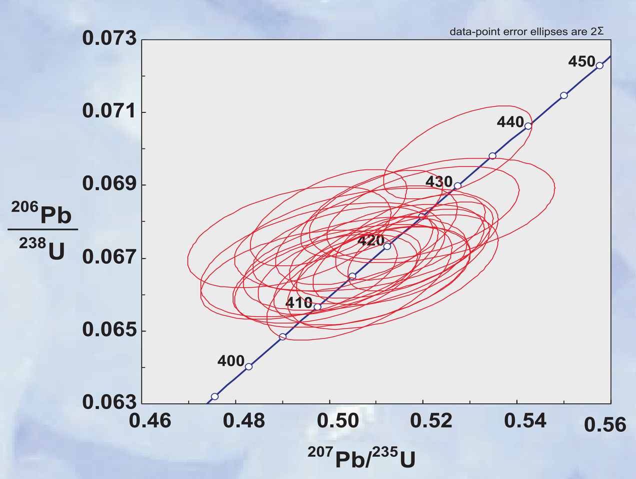
Observed changes in Pb/U vs UO_2/UO ratios measured on a standard and unknown over a 3 day period. Towards the end of the analysis session a change in source conditions resulted in a decrease in the beam density. However, the slope of the lines defined for the Pb/U vs UO_2/UO ratios are the same as those defined by changes in surface oxygen flooding or secondary in energy.



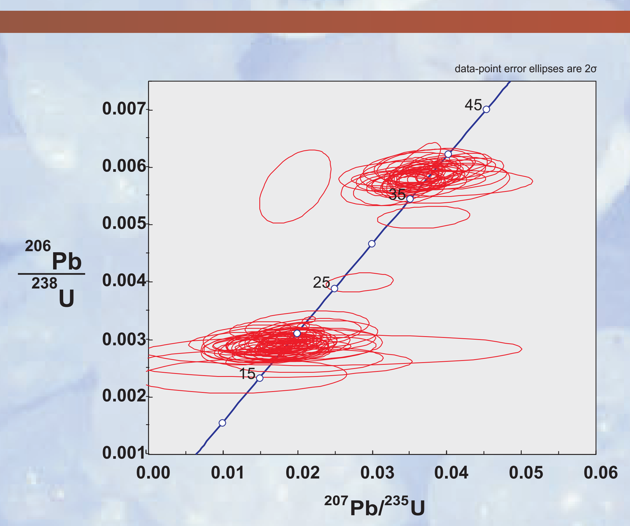
Changes in count rates on zircon created by starting oxygen flooding during an analysis. The Pb ion yield increases by approximately a factor of two. Note the UO_2 increase is similar to Pb and the UO also increases slightly. Although some U counts are lost generally U>>Pb and the precision of the Pb/U ratio is dominated by the Pb counts.



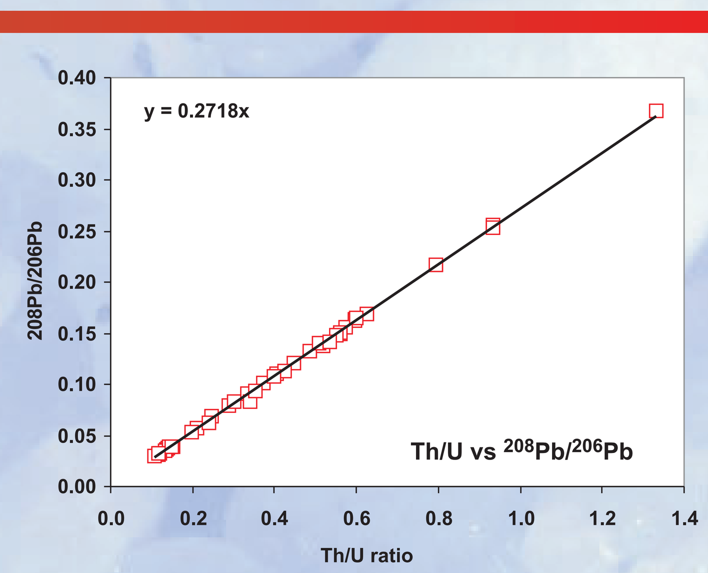
On the ims-1270 a field aperture can be placed at the image plane to restrict ions to the centre of the analysed area. This feature coupled with a sharply defined köhler primary beam and pre-sputtering reduces the effects of surface Pb contamination to a minimum. For 240 repeats of the Temora secondary standard the ^{204}Pb common lead was generally <1.5ppb.

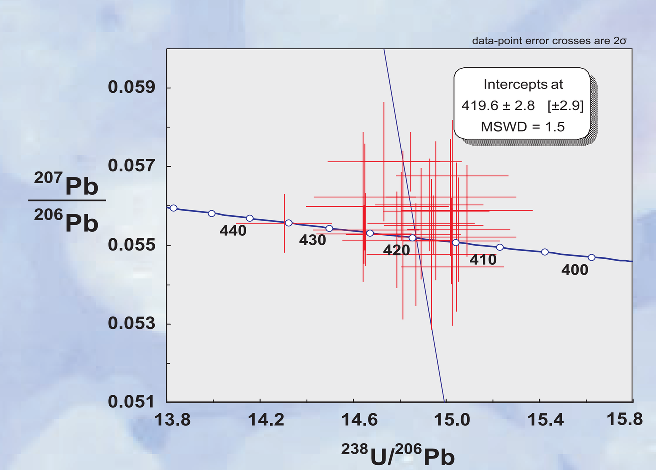


Concordia plot of 420 Ma 'unknowns'. The small common lead correction and high precision of the corrected Pb/U ratio permits good depiction of relatively young samples on the Concordia plot. Error bars/ellipses combine observed variation in both standards and unknowns. In general, observed standard deviations of the Pb/U ratios measured on the standards exceed those purely due to counting statistics by about 0.3%.



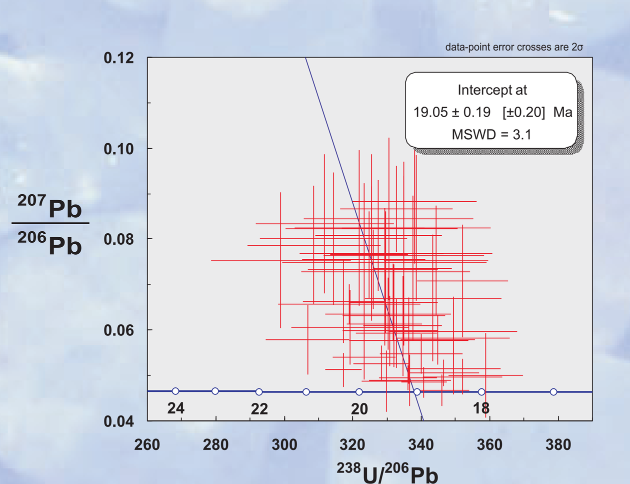
Concordia diagram combining zircons from 7 sedimentary rocks (only the youngest <50 Ma fraction shown, no exclusions). The combined data set demonstrates that even very young zircons can be shown to fall about the Concordia curve and individual source ages accurately defined (data courtesy of A. Kemp, University of Bristol).





Tera-Wasserburg plot of the uncorrected ratios for the 'unknowns' demonstrating low degree of common lead correction. Errors shown includes the variability in the standards measured in the same session.

Plotted using Isoplot (Ludwig 2003).



Tera-Wasserburg plot of the uncorrected ratios for the young fraction (<22Ma) demonstrating the small common lead correction and high precision even for young samples. 5 analyses were excluded for high ^{206}Pb correction (>5%) and 2 for high U content. The majority of the zircons had between 0.2 to 0.7 ppm total lead.

Corrected $^{208}Pb/^{206}Pb$ ratios for a 2850 Ma zircon.

The Th/U ratio is determined by combining the ThO/UO and $^{208}Pb/^{206}Pb$ ratios measured on the standard (and assuming closed system behaviour). Comparisons between the measured $^{208}Pb/^{206}Pb$ ratios and those predicted (from the unknowns Th/U ratio and its approximate age) can be used to detect open system behaviour.

Thermal effects on Pb secondary ionisation

Analysis with Standards and Unknowns on separate mounts

Attempts to make analyses using standards mounted separately from unknowns occasionally gave low Pb/U ratios for approximately an hour after a sample change.

The source of this decrease was somewhat unexpected:

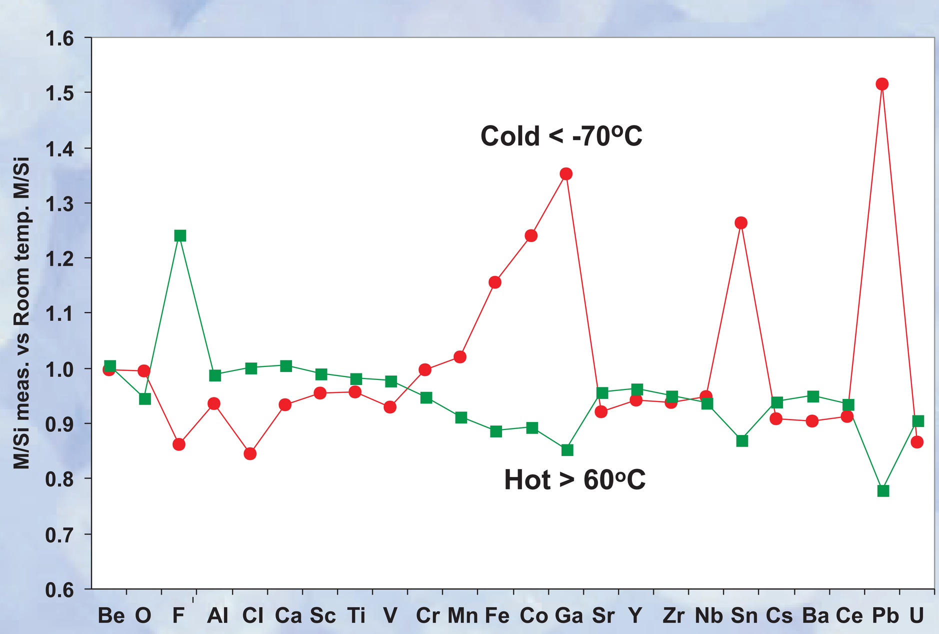
The ionisation efficiency of Pb varies with the sample temperature!

Subsequent experiments by both heating and cooling samples demonstrated that the Pb ion yield decreases with increasing temperature and increases as the temperature is lowered.

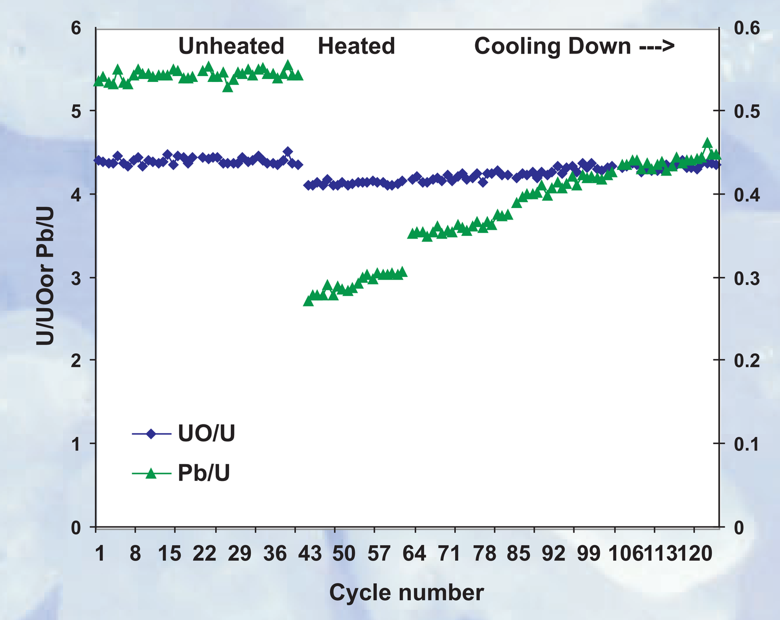
Although Pb is not the only element affected it has been found to be the most sensitive element to this effect.

Conclusion:

Analyses can be made using standards and unknowns mounted separately but care must be taken to avoid differences in temperature between mounts.



Observed changes in the ionisation of elements (relative to Si) for a heated and cooled glass sample relative to yields observed at room temperature. Pb is the most sensitive element to temperature changes followed by Sn and Ga. U is virtually unaffected. Note the ion yield of Pb may increase by up to 70% at very low temperatures. It is estimated that changes as little as 5°C may be sufficient to change the measured Pb/U ratio by 1%.



Observed changes for zircon comparing analyses of an unheated crystal with those of a heated sample which then slowly cools down in the instrument. Note that the U oxides are not significantly affected and no correction procedure appears to adequately compensate for the changes in the Pb/U ratio

References

Schuhmacher M. et al. (1994) In: Secondary ion mass spectrometry SIMS IX (ed A. Benninghoven et al.) pp 919-922. Whitehouse M.J. et al. (1997) Geochim. Cosmochim. Acta 61, 4429-4438. Stern R.A. And Amelin Y. (2003) Chem. Geol. 197, 111-142. Compston W. J. Geol. Soc. Lond. 161, 223-228.

Acknowledgements

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