

Interactive comment on “Extreme isotopologue disequilibrium in molecular SIMS species during SHRIMP geochronology” by C. W. Magee Jr. et al.

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General Comments

This paper contains some pretty interesting observations. However the presentation style doesn't lead the reader through what is going on. There are a number of abrupt introductions and changes that could be expanded and more descriptive so the reader can follow along. I've noted some of these below.

I'm a bit concerned by some of the statements regarding molecular interferences. Are these species of importance or not? If you think they are U species, then look at the uraninite. There could be a variety of molecules including ^{17}O (which will be at a similar level to UO hydrides for example).

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The paper really needs to lay out the issues at hand and what it is addressing. Is this to improve calibration by using combination of U, UO and UO₂ species? Or is it looking at the issues surrounding using such a scheme?

There are a number of typos through the manuscript.

Figures are good.

References appropriate.

Specific Comments

P1 L10 - It's not clear what this paper is about as you start to read the abstract. It needs a couple of sentences of introductory material concerning the issues to be covered.

L38 "a" quadrupole triplet (change case, cap A)

P2 L4 Compston et al. 1984 should be the prime reference for the foundations.

L18 You could add xenotime in here for alarming matrix effects.

L32 greater than 5 keV (not kV)

L40 is "isotopologuous" a word - just replace with isotopologue.

P3 L18 Not sure this is quite right but it doesn't make a lot of difference. I believe the extraction voltage power supply (2.5 kV) floats on the 10 kV column grnd/sample potential. The offset potential is around say 700 V, so the intermediate extraction sits at 9.3 kV relative to real ground in this example. So there's a 700 V bias from the sample to the IE plate.

The total secondary ion energy is always 10 keV, no matter the setting of the IE.

P4 L17, L32 Remove periods from end of subtitle (and elsewhere)

L23 (Magee et al., 2014).. (double period)

P5 L31 Check for consistency in use of UO/U and UO⁺/U⁺.

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P6 L6 Ratios are then typically multiplied by 100. Why “typically”? Should be consistent. In abstract it is permil. In figures it is left as the absolute ratio.

L13 Maybe put a subtitle giving the analytical configuration. I had to go back and check.

L14 UO/U was about 6.2. Give the range or mean \pm std dev.

L17 “However, U18O18O is very low” to “. . . is a very low. . .”

L19 Scan uraninite over the region of interest to sort out the interferences. If you suspect UO₂H₂ then it should be supported by a higher UO₂H. ²³⁵U species, ¹⁷O species?

L21 Suggest not mentioning the 18O experiments here, I got confused. Put it in as part of Expt B description.

L27 (and a few other places) - cap B on baddeleyite

L28 and elsewhere unusual to have unit expression on both value and uncertainty i.e. 4.9% \pm 4.4 %. More typical to present as 4.9 \pm 4.4 %.

P7 Ickert et al. 2015. The Chamberlain reference would be better here because it is SIMS data. And reference is 2013, not 2014 as listed.

L8 baddeleyite

L10 substantial variation. . . what is the uncertainty on a single measurement?

L17 “Ignoring these. . .” If we’re going to ignore them, why include them? The poor counting stats likely de-weights their significance anyway.

L22 Did you retune down hole? Some of this variability can be recovered by secondary ion retuning. Also there may be some issue concerning different angular emission between the different species. Also, different energy distributions if there is any target charging. Was an energy slit included?

L30 Monazite anomalies - okay so I suspect there are some molecular interferences

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in the monazite spectrum. Some long range scans might have helped identify them. “Unlabeled monazite analyses”; what does this mean?

P8, L1 Seems a bit late to be introducing this terminology.

L14 if they high - typo

L17 Might be useful to have a brief description of the Raman results here.

L25 Abrupt start to Discussion. What is expected from the calibration and the distribution of the isotopologues?

P9 L5 Unclear what this is about. Where did this “Molecular ion escape model” come from? Wouldn’t speciation be a bigger issue (neutral vs ion) or angular/energy distributions? What does it mean “escape without reacting”?

L9 Why should it affect $^{16}\text{O}^{2+}$ preferentially?

L16 Where... should be where.

L31 the tails on UO_2 are not documented. I’ve generally seen the poorest peak shape on U^{+} , which appears to be energy related. The form of these tails could be indicative as to the source.

L34 keV

P10 L 1 “agree” with what? each other? “higher potential valence states of uranium” - where did this come from?

L32 greenschist

L37 only one grain analysed?

P11 L5 where U-Pb geochronology by SIMS has poor accuracy.

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