

Interactive comment on “Alkali element background reduction in laser ICP-MS” by C. W. Magee Jr. and C. A. Norris

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Received and published: 2 March 2015

Dear Editors: This replay is structure as follows: A1: Reviewer #1 comments A2: Reviewer #2 comments B1: Our reply to Reviewer #1 B2: Our reply to reviewer #2

A1: Anonymous Referee #1 Received and published: 27 January 2015 The paper is a well written description of how to improve the accuracy of laser ablation ICP-MS analyses. The authors describe their methods used in calibration measurements where they replace a traditional alkali glass tuning standard with a new synthetic low-alkali-glass reference material. Using this method they demonstrate how they can diminish the alkali contamination significantly without necessary requiring changes to the analytical procedures. The paper is written in a compact but clear manner. The amount of detail

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provided is probably not sufficient to repeat their work. The main measurement result tables 4a and 4b are hard to interpret. It might have been useful (suggestion) to provide some multivariate analysis of the results which would give a relative comparison of performance for different skimmer cone, sampler cone and glass references. It is possible that this would also point at the reasons for the contamination event described in the last paragraph of section “Discussion”. It would also explain the relatively large occasional variation in Na in table 4b. Minor corrections: P4, L22, hrz ! Hz.

A2: Anonymous Referee #2 Received and published: 15 December 2014 Review of Magee & Norris: Alkali element background reduction in laser ICP-MS - Submitted for publication in Geoscientific Instrumentation, Methods and Data Systems General comments: This is an interesting, albeit brief, manuscript that reports on systematic experiments aimed at reducing background levels of the notorious alkali elements in LA-ICPMS. This is highly relevant since the most commonly used standard for instrument tuning and external standardization is a high-Na glass, which potentially contaminates the instrument with easily ionizable alkali-elements, most notably Na, making their low-level analysis near impossible. The authors report on results of standard measures such as segregating cones for different usages, which they admit are rather straightforward. However, their main contribution is the presentation of a custom-made, virtually Na-free (relative to conventional glasses) homogenous glass standard. Its performance relative to the conventional ones forms the main results section. Overall, the paper merits publication, however, several aspects, especially the results/discussion parts, could be improved, as follows. It would be great if towards the end some mention is made where / how these glasses are being made available. They would be of great interest to the community. Specific comments: 1) P4, L9: Insufficient details are provided for the LA-ICPMS section. No information is given on the cell gas, cell type, laser fluence (25% mirror is insufficient), ThO/Th, U/Th, RF power etc. to name but a few. This might be best placed into a short table. 2) P4, L29: In view of the updated Jochum et al (2011) NIST61x dataset, it's surprising to see Pearce et al (1997) to be used in 2014 still. 3) Tab. 4b & P5+6: Not enough is made of the fact that the back-

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ground levels, using the alkali-poor glass, from Aug. 2006 onwards vary remarkably much, not only over time (e.g. for Na almost 200x between 10-Aug and 18-Sep) but also during a single day. Regarding the latter, a nearly 100x fold decrease is seen on 18-Sep, followed by a >30x-fold increase on the same day, assuming these data are in sequence. These large (Na) background variations much be mentioned and explained, e.g. in the context of type of sample material analyzed that day etc. Are such variations seen because NIST61x were used during those days nevertheless given their importance as external standard? Does Na bg become low again with cleaned cones? 4) What is the availability of these glasses? Even if they are not fully standardized, they would still be very valuable as tuning material for the community! 5) Figures: I'd suggest that some figures would benefit from y-axes with breaks in scale, such that the variability is better displayed and not dominated by few high outliers, e.g. Fig. 1b, 1c, 1d. Or a zoomed-in close-up near-zero or indeed logarithmic scaling. Minor corrections (not necessarily comprehensive): P4, L3:kV not keV, as accelerating voltage in EMPA. P4, L22: hrz? Hz. P5, L16-19: There is a wording problem somewhere in this sentence and verbs etc appear to be missing. P1, L3: . . .Sciences; The Australian. . .

B1: Reply to Referee #1 (GID-4-C95-2015) The suggestion of multivariate analysis on the segregated cones is a good one, had it been done at the time the data were taken. The mass spectrometer used in this study has been retired, and is now two generations out of date, so any deeper insight gained by such analysis would be of limited value. More detail has been provided, and is described below in response to the comments from reviewer #2. We feel showing the raw results from our assessment of using the tuning glass is of more use to the readers.

B2: Reply to Referee #2 (GID-4-C254-2014) The methodology description and discussion sections have been expanded to address the comments of the reviewer. Specific comments: 1: More detail is given in the revised manuscript. 2: The reference values used are those popular when the data was taken (2006), so that these data can be compared directly with published studies from that time period.

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Since the assessment of backgrounds is independent of the reference values used for NIST, we do not think this clouds the assessment of their value. Similarly, the few to tens of percent changes in values are negligible to the orders-of-magnitude compositional differences between the tune-1 glass and the NIST glass. 3: More explanation added to the discussion section of the revised manuscript. 4: The authors intend to make the glasses available after publication, and a note has been made in the text of the revised manuscript. 5: Logarithmic scaling is used in figure 1a, and was considered for other figures before being rejected for clarity. We made a point of including all of the data in table 4a and 4b, so that interested parties can plot them. We are happy to make the data available in a machine-readable format as a supplement. The minor corrections noted by the reviewer have been made.

Please also note the supplement to this comment:

<http://www.geosci-instrum-method-data-syst-discuss.net/4/C311/2015/gid-4-C311-2015-supplement.pdf>

Interactive comment on Geosci. Instrum. Method. Data Syst. Discuss., 4, 609, 2014.

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