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Quality assurance in accelerator mass spectrometry: Results from an international round-robin exercise for ^{10}Be

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ABSTRACT

The first international round-robin exercise for the measurement of the long-lived radionuclide ^{10}Be has been conducted. Ten participating accelerator mass spectrometry (AMS) facilities have each measured three samples at the 10^{-12} to 10^{-14} ^{10}Be/^{9}Be level. All results have been made traceable to the NIST SRM 4325 standard to avoid additional discrepancies that arise when different facilities use different calibration materials. Hence, the data concentrates on pure measurement distinctions. Multivariate statistical investigations have been performed to reveal a bias between facilities, i.e. two distinguished groups could be identified. Maximum discrepancies between two single facilities are in the range of 6–31% depending on the absolute ^{10}Be/^{9}Be value. These findings should be considered when comparing ^{10}Be data produced at one AMS facility with that produced at another facility, which is e.g. often the case for in situ ^{10}Be dating studies. Round-robin exercises are a very helpful tool as part of an overall quality assurance scheme to improve the accuracy, and not only the precision, of AMS data.

1. Introduction

The field of accelerator mass spectrometry (AMS) has recently experienced massive improvements in measurement uncertainties with respect to counting statistics. The AMS community, mainly physicists running AMS facilities and their users, have subsequently started to request and pursue quality assurance schemes to guarantee high-quality data. Scientists investigating the commonly used and commercially important ^{14}C are keenly aware of the need for quality assurance, and have, thus, already introduced periodic round-robin exercises over the past few decades, using common standard reference materials to make results traceable and directly comparable. There have been, however, only a few similar approaches, mostly on a much smaller scale, for other nuclides such as ^{26}Al [1], ^{36}Cl [2], ^{41}Ca [3] and ^{129}I [4,5]. Generally, in all these studies, statistically significant discrepancies between different facilities could be identified. Reasons for these have been partially pinpointed to: (1) the use of non-concordant calibration materials due to different half-life values and ratios; (2) cross-contamination and memory effects while measuring or chemical processing; and (3) data-reduction. Most participating AMS facilities of these round-robins have drawn conclusions and suggested renormalisation of their earlier results, e.g. [3,6,7].

Despite the fact that ^{10}Be is the second most commonly measured radionuclide world-wide, there has yet to be a published ^{10}Be round-robin exercise involving a larger number of AMS facilities. This is astonishing, as international projects such as CRONUS-EU [8] and CRONUS-Earth [9], have been striving to address other problems, which are especially related to ^{10}Be applications in geosciences. Thus far, the following improvements have been made:

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(1) The ratio of a common primary standard issued by a metrology institute (NIST SRM 4325) has been remeasured and yielded a precise $^{10}Be/^{9}Be$ ratio of $(2.79 \pm 0.03) \times 10^{-11}$ [10].
(2) The half-life of ^{10}Be has been determined accurately to (1.387 ± 0.012) Ma [11].
(3) The advantages and disadvantages of the most common scaling models [12–17] have been discussed in public and advices have been given on how to report cosmogenic nuclide data in publications [18].

Taking all this new information into consideration, several scientists have recently suggested different SLHL (sea level, high latitude) ^{10}Be in situ production rates and have also strongly recommended the introduction of the use of local, rather than global average cosmogenic nuclide production rates [19–24]. However, all these studies overlooked the primary need to be able to directly compare AMS results produced at different facilities to allow reconciliation of recently produced data with that published within the past decades.

To overcome this lack, the first international interlaboratory ^{10}Be study has been conducted with ten participants, including most of the world-wide leading AMS facilities as well as some newcomers. All facilities generously declined their right to anonymity in order to optimise consequential conclusions from the results. Special focus was set on $^{10}Be/^{9}Be$ measurements typical for samples used in geoscience applications, thus, $^{10}Be/^{9}Be$ values ranging from 10^{-12} to 10^{-14}.

2. Experimental

2.1. Samples

The original NIST SRM 4325 solution has been diluted step-wise with ^{9}Be-carrier solution (Sigma–Aldrich, Be in HNO_3, $c_{Be} = (1003 \pm 2)$ µg/mL, $\delta = 1.05$ g/mL) to yield small quantities (57–106 mg) of BeO. This powder has been disseminated to each of the ten participating facilities. Sample preparation from this point forward was dependent on the self-determined protocol of each of the AMS labs (such as mixing with metal powder) and was the full responsibility of the individual participants (Table 1).

In order to eliminate discrepancies resulting from the then ongoing debate about the ^{10}Be half-life and the $^{10}Be/^{9}Be$ standard ratios, all facilities were asked to submit results traceable to the standard NIST SRM 4325. Thus, in contrast to "normal" calibration procedures at individual AMS facilities, all ^{10}Be results in this study are measured either directly vs. NIST SRM 4325 or are traceable via a cross-calibrated in-house standard to NIST SRM 4325. Hence, remaining discrepancies are purely based on the measurement itself, including – for those facilities using secondary standards – the overall quality of the cross-calibration, and are not due to the use of different standard materials.

Expected $^{10}Be/^{9}Be$ values for the three samples to be measured at each AMS lab are on the order of $\sim 3 \times 10^{-12}$ (SM-Be-12), $\sim 3 \times 10^{-13}$ (SM-Be-13), and $\sim 3 \times 10^{-14}$ (SM-Be-14), respectively. The calculated values take into account the ^{10}Be contribution from the ^{9}Be carrier for which an unweighted mean $^{10}Be/^{9}Be$ value with one-sigma standard deviation of $(1.13 \pm 0.23) \times 10^{-14}$ has been determined at three AMS facilities (ASTER, Gif-sur-Yvette, and VERA). Thus, the calculated values for the three samples are only given here as comparisons (Figs. 1–3) and do not represent any absolute or "true" values.

2.2. AMS measurements

As the round-robin exercise has not been performed as a blind trial in that each AMS facility was informed that the measurements would be part of an interlaboratory comparison, it has been left to each facility to decide themselves whether the samples were to be treated as "routine" samples or if they were to be measured more often, i.e. as replicates, or for a longer duration than a "normal" sample from a "normal" user, in order to reduce statistical uncertainties. This has been intended to demonstrate the best measurement capabilities of each facility which are, of course, also available to clients upon request.

The most important measurement conditions are summarized for all AMS facilities in Table 1; set-up details can be found in the corresponding references given there. The range of accelerator terminal voltages used by participating AMS facilities (0.5–8.0 MV) is representative for all world-wide machines capable of measuring ^{10}Be.

2.3. Instant data improvement

Following the successful approach of the earlier ^{36}Cl round-robin exercise [2], preliminary ^{10}Be-results have been presented at several international physics and geology meetings and workshops [35–37] in order to enable discussion of preliminary trends and to increase the number of AMS participants adding to the overall value of the outcome. After the initial comparison of individual facility data with data of other participants, all AMS facilities have been given the chance to remeasure samples, improve their measurement procedures and/or method of data evaluation to allow indi-

Table 1
Measurement conditions for round-robin samples at the different AMS facilities. See references for further details. Calibration is directly vs. NIST SRM 4325 or traceable via a cross-calibrated in-house standards to NIST SRM 4325.

AMS facility[a]	Stripping	Terminal voltage (MV)	Ion charge [post-stripping]	Metal binder	Cathode	In-house standard	Reference for set-up
ANU	Gas	8.0	3+	Nb	Cu	None	[25]
ASTER	Gas	4.5	2+ [4+]	Nb	Cu	None	[26,27]
CNA	Gas	1.0	1+ [2+]	Nb	Cu	S555	[28]
ETH	Gas	5.5	3+ [4+]	Cu	Cu	S555	[7]
DREAMS	Gas	4.5	2+ [4+]	Nb	Cu	SMD-Be-12	[29]
LLNL	Foil	7.5	3+	Nb	Stainless steel	07KNSTD3110	[30]
PRIMELab	Foil–gas	7.5	3+	Nb	Stainless steel	Be-01-5-2	[31]
SUERC	Gas	5.0	3+	Nb	Cu	None	[32]
TANDY	Gas	0.5	1+ [2+]	Nb	Cu	S555	[33]
VERA	Gas	3.0	2+ [4+] and 3+ (gas asborber)	Cu	Cu	S555	[34]

[a] ANU = Australian National University, Canberra; ASTER = Aix-Marseille Université, CNRS, CEREGE UM34 CNA = University of Seville-CNA, Seville; ETH = Laboratory of Ion Beam Physics, ETH Zurich, CH-8093 Zurich; DREAMS = DREsden AMS, Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Dresden; LLNL = CAMS, Lawrence Livermore National Laboratory, Livermore, CA 94550; PRIMELab = PRIME Lab, Purdue University, IN 47906; SUERC = Scottish Universities Environmental Research Centre (SUERC), East Kilbride G75 0QF; TANDY = 0.6 MV accelerator facility (TANDY), Laboratory of Ion Beam Physics, ETH Zurich, CH-8093 Zurich; VERA = University of Vienna, Faculty of Physics, VERA laboratory, 1090 Vienna.

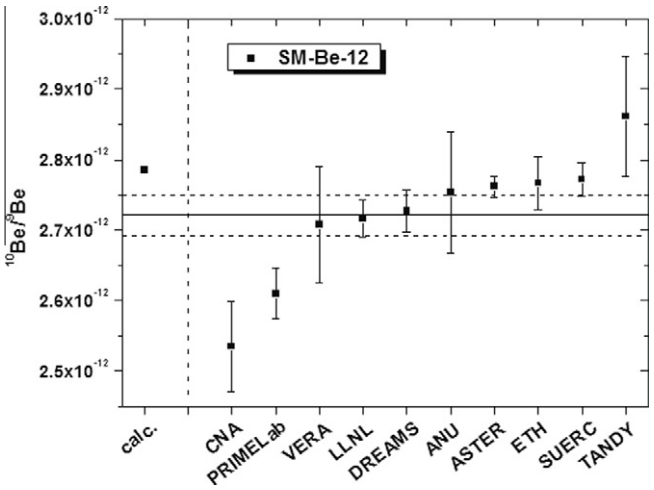

Fig. 1. Single facility results, stated uncertainties, the mean (solid line) and standard deviation of the mean (dashed line) for sample SM-Be-12. The "theoretical" value, calculated by step-wise dilution, is also given for the purpose of information only.

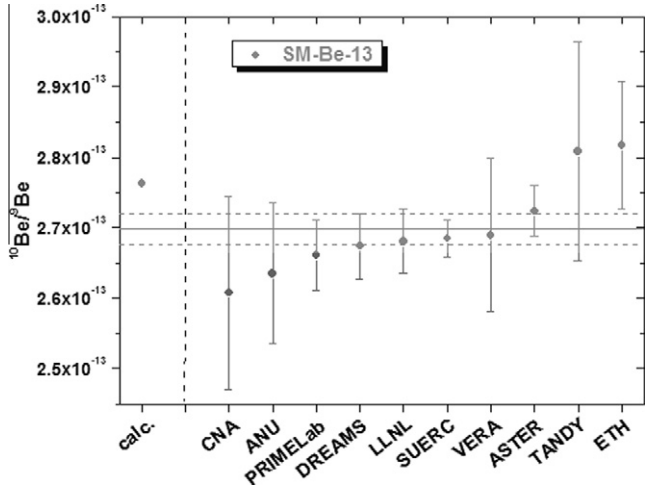

Fig. 2. Single facility results, stated uncertainties, the mean (solid line) and standard deviation of the mean (dashed line) for sample SM-Be-13. The "theoretical" value, calculated by step-wise dilution, is also given for the purpose of information only.

vidual improvement. This opportunity for reassessment and modification of procedures is, of course, not fully consistent with the protocol of a normal proficiency test layout as recommended e.g. by the International Harmonised Protocol for Proficiency Testing of (Chemical) Analytical Laboratories [38]. However, the overarching goal was the improvement of the quality of ^{10}Be data, enabling the statistical analyses of the best measurements possible at each facility and, thus, producing an immediate decrease in the last remaining discrepancies between individual facilities.

One example for this kind of instant improvement is the renormalisation of preliminary results [35] from both ETH and TANDY according to new in-house standard values [7]. Likewise, ANU originally submitted preliminary ^{10}Be data for this exercise based on very quick single measurements [37]. They then later improved their data by performing multiple measurements of the three intercomparison samples over several runs spread out over several days. Additionally, the more recent ANU measurements employed a slightly different method, which involved measuring $^{9}Be^{3+}$ rather than $^{16}O^{5+}$. During this study, CNA increased their final $^{10}Be/^{9}Be$ value for SM-Be-13 by roughly 10% (and decreased the corresponding uncertainty) with respect to the originally submitted value previously presented [37]. Finally, the two facilities ASTER and VERA, which were among the first participants in the round-robin exercise, improved their individual preliminary data by repeated measurements.

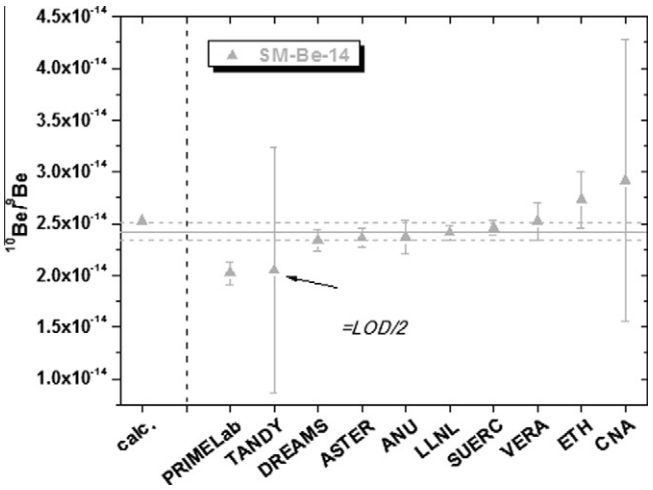

Fig. 3. Single facility results, stated uncertainties, the mean (solid line) and standard deviation of the mean (dashed line) for sample SM-Be-14. The originally submitted TANDY limit of detection (LOD = 4.1 $\times$ 10^{-14}) has been replaced by LOD/2 and adding an uncertainty by (LOD/2)/sqrt(3) to allow incorporation into statistical evaluation. The "theoretical" value, calculated by step-wise dilution, is also given for the purpose of information only.

It should be emphasized that the TANDY results in this round-robin comparison still refer to the status of the TANDY before April 2009 [33], which was limited by a high $^{10}Be/^{9}Be$ background level mainly caused by scattered ^{9}Be. It has been recently shown that the installation of an additional magnetic mass filter to the high energy mass spectrometer effectively suppresses the background to a ratio of <5 $\times$ 10^{-15} [39], but remeasurements of the round-robin samples have not yet been performed.

3. Results and discussion

Single measurement results without replicates and their stated uncertainties of different origin have been reported by the ten participating AMS facilities (Table 2). The uncertainties are assumed to be preferably "Guide to the Expression of Uncertainty in Measurement (GUM)"-oriented uncertainty estimates rather than pure A-type statistical evaluations due to the specific kind of measurement. Thus, all uncertainties have been assumed to be standard uncertainties and are given as such, unless otherwise indicated.

As participants were asked to submit their data and corresponding uncertainties using the same protocol as they would use for any user-submitted sample measured at their facilities, there is no uniformity in how the uncertainty calculations were performed. For example, some facilities do not take into account the uncertainties associated with the used in-house standard or the original calibration standard(s), which is, in this case, NIST SRM 4325 for all facilities. Reported relative uncertainties for $^{10}Be/^{9}Be$ measurements from each facility range from 0.6% to 3.1% for sample SM-Be-12, 1.0% to 5.5% for sample SM-Be-13, and 2.9% to 47% for sample SM-Be-14. All facilities provided values for all three samples with the exception of TANDY, which submitted for the lowest sample SM-Be-14 only a limit of detection (LOD). This indication has been replaced by a value of LOD/2, and an assigned uncertainty estimate of (LOD/2)/sqrt(3) to allow incorporation into the statisti-

Table 2
Measured $^{10}Be/^{9}Be$ ratios as submitted from AMS facilities or rounded to appropriate digits for three BeO samples with mean of facility means and the standard deviations of the means of facility values. Uncertainties are given as submitted. All results are traceable to NIST SRM 4325 with a ratio of (2.79×10^{-11}) [10].

AMS facility	Sample SM-Be-12 (10^{-12})	Sample SM-Be-13 (10^{-13})	Sample SM-Be-14 (10^{-14})
ANU	2.754 ± 0.087	2.64 ± 0.10	2.37 ± 0.16
ASTER	2.762 ± 0.015	2.724 ± 0.036	2.359 ± 0.092
CNA	2.535 ± 0.064	2.61 ± 0.14	2.9 ± 1.4
DREAMS	2.728 ± 0.030	2.674 ± 0.047	2.34 ± 0.10
ETH	2.766 ± 0.037	2.817 ± 0.091	2.74 ± 0.27
LLNL	2.716 ± 0.026	2.681 ± 0.046	2.414 ± 0.071
PRIMELab	2.610 ± 0.036	2.661 ± 0.050	2.02 ± 0.11
SUERC	2.772 ± 0.024	2.685 ± 0.026	2.462 ± 0.071
TANDY	2.861 ± 0.086	2.81 ± 0.16	LOD = 4.1 → 2.1 ± 1.2[a]
VERA	2.708 ± 0.082	2.69 ± 0.11	2.53 ± 0.18
Mean and standard deviation	2.721 ± 0.029	2.698 ± 0.022	2.420 ± 0.086

[a] Replacing submitted limit of detection (LOD = 4.1×10^{-14}) by LOD/2 and adding uncertainty by (LOD/2)/sqrt(3) to allow incorporation into statistical evaluation.

cal evaluation. Table 2 lists all reported results, as well as the mean of facility means and the standard deviations of the means of facility values.

The entire data set has been statistically analysed with respect to the normality of the data sets (Kolmogorov–Smirnov, skewness and kurtosis test), possible Grubbs outliers, and compatibility of the facility value with the mean within the stated uncertainty (application of E_n criterion – see e.g. [40]). Additionally, some mainly multivariate investigations have been performed to reveal a possible bias between facilities, i.e. significant dependence of the $^{10}Be/^{9}Be$ value obtained at the facility at which the samples have been measured. All three "normality" tests proved the data set to be normal, however, SM-Be-12 is slightly skewed to smaller values. No Grubbs outlier has been identified at the significance level of $\alpha = 0.01$. The mean and median are not significantly different for each of the three samples, thus, the mean is a good estimate. Single facility results with stated uncertainties, as well as the mean and standard deviation of the mean are shown individually in Figs. 1–3 for each of the three samples. Values have been separately sorted in ascending order for each sample. For the purpose of information only, $^{10}Be/^{9}Be$ values calculated from the step-wise dilution procedure are also given.

The standard deviation of the mean is proportional to the mean value for samples SM-Be-12 and SM-Be-13, but not for sample SM-Be-14, thus, pointing to a constant minimum absolute uncertainty increasing the relative uncertainty. This might reflect the influence by the relatively high background (up to 10^{-14}) on measurements of SM-Be-14, especially for the lower-energy AMS machines. Thus, a moderate correlation can only be identified between SM-Be-12 and SM-Be-13.

The mutual agreement values E_n between two individual facilities are given by

$$E_n = |x_a - x_b| \Big/ \sqrt{[u^2(x_a) + u^2(x_b)]},$$

where x_i is for the individual facility result, and $u(x_i)$ is the uncertainty of the corresponding quantity as stated by the facility. A $^{10}Be/^{9}Be$ result from facility a can be considered being compatible with the $^{10}Be/^{9}Be$ result of facility b, if E_n does not exceed a value of 2. When the $^{10}Be/^{9}Be$ data for each of the three samples measured at each of the ten AMS facilities are evaluated in this manner, it can be clearly seen that CNA and PRIMELab are causing problems in yielding high E_n-values for sample SM-Be-12, and PRIMELab only for sample SM-Be-14. The latter value is basically due to an underestimation of the uncertainty. Thus, a weighted mean is not applicable.

Hierarchical clustering provides a robust tool for assessing a basic facility bias. It considers all data obtained for all samples, and decides about distant labs, i.e. those who obtained distant data for all samples. A hierarchical clustering (with Euclidean metric and Ward linkage), which uses minimum variance as the linkage criterion (see e.g. [41]), clearly shows that the ten facilities form two distinct groups (Fig. 4). PRIMELab and CNA form a single group, whereas the remaining eight AMS facilities form the second group. More specifically, TANDY falls somewhere "in-between" these two groups, but is a bit closer to ETH than to the PRIMELab/CNA group. If measurement results are normalized to the mean and sorted as ascending normalised means of the samples, a similar grouping of facilities can be shown. However, because differences in measuring sample SM-Be-14, especially by those facilities with a high background, are overinfluencing the mean difference (Fig. 5a), the ordering works better if only samples SM-Be-12 and SM-Be-13 are taken into account (Fig. 5b).

4. Interpretation and further implications

Reasons for the observed partially systematic bias in the ^{10}Be data can be openly discussed here and hopefully beyond the scope of this paper in thanks to the participating AMS facilities who agreed to give up their right to anonymity, which is otherwise usually guaranteed in round-robin exercises. Possible causes such as source memory effects (like those reported for ^{36}Cl [2]), false dead-time, or isobar corrections, seem to be quite unlikely given the advanced level of today's technology. The clustering of AMS facilities into two separate groups cannot be attributed to e.g. wrongly cross-calibrated in-house standards, because PRIMELab does not use the same in-house standard as CNA, whereas three members of the second group, i.e. ETH, TANDY, and VERA, use the same in-house standard S555 like CNA. Besides reasons for these systematic deviations cannot be clearly identified, being for

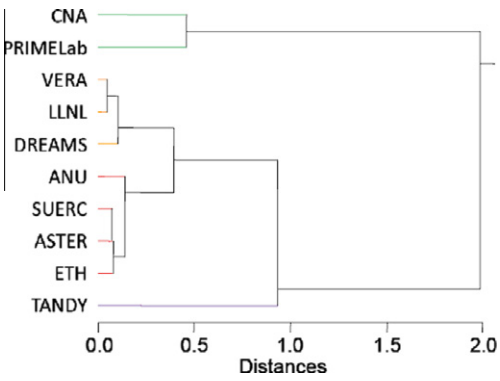

Fig. 4. Hierarchical clustering of all facility results (absolute values) for all samples by Ward linkage, see e.g. [41] for further details.

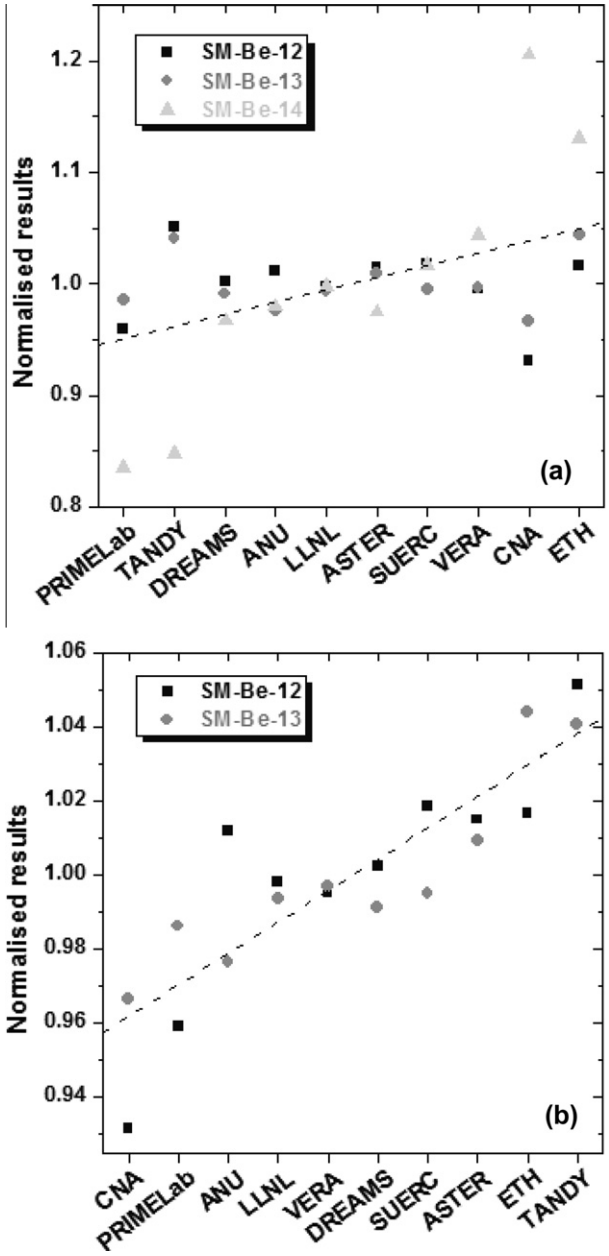

Fig. 5. Regression of normalised measurement results for samples SM-Be-12, SM-Be-13, and SM-Be-14 (a), and SM-Be-12 and SM-Be-13 only (b), respectively, each in the order of ascending average.

all analytes and/or all levels at the low/high end of the scale of the hierarchical clustering should be a reason for those facilities to investigate their method for (hidden) biases when these indications are given.

However, it seems highly advisable for all participants in this study, as well as other AMS facilities that routinely perform ^{10}Be measurements, to regularly check their in-house standards vs. NIST SRM 4325, the only primary standard material, which has yet been issued by a metrology institute, and, thus, the only standard guaranteeing traceable and comparable ^{10}Be data. Unfortunately, the NIST SRM 4325 material is no longer in stock, and thus a replacement standard of the same quality is needed in the very near future.

At the moment - after having already solved some previously existing problems such as those described by Kubik and Christl [7] – the ^{10}Be-"situation" is the same as that for ^{36}Cl [2] if samples are measured in the 10^{-11} to 10^{-13} range (^{36}Cl/$^{35+37}$Cl) as compared to the 10^{-12} to 10^{-14} range (^{10}Be/^{9}Be). Identical BeO powder submitted to two AMS facilities yield ^{10}Be/^{9}Be results that can differ up to maximum values of 12% (CNA vs. TANDY) at the 10^{-12} level. Setting aside CNA and TANDY, which had the most challenging set-ups for measuring ^{10}Be in the 10^{-13} and 10^{-14} range at the time of the round-robin exercise, differences are reduced to 6% (PRIMELab vs. SUERC) at 10^{-12}, 7% (ANU vs. ETH) at 10^{-13}, and 31% (PRIMELab vs. ETH) at 10^{-14}. It seems worthwhile to determine if these effects might help to explain, at least partially, recently observed discrepancies in in situ ^{10}Be production rates studies [19–24] in comparison to earlier ones incorporated into e.g. the CRONUS-Earth online calculator [42].

As lab-to-lab ^{10}Be/^{9}Be values seem to vary by several percent, terrestrial cosmogenic nuclide users should also consider the use of "local" production rates when applying ^{10}Be values to geomorphic settings, not in the sense of "geologically local", but in the sense of "AMS machine local". This could be especially helpful for lowering overall uncertainty and, thus, improving overall in situ data. Or in other words, improvements in accuracy of measurements should follow the recent improvement in precision by avoiding the actual observed experimental lab-to-lab biases when possible. Hence, it might be advisable to incorporate into systems like the CRONUS-Earth online calculator [42], a list with individual production rates and the name of the AMS facility that has produced the production rate to choose from. Thus, if a user has had samples measured at a specific AMS facility (i.e. ANU, ETH, LLNL, etc.), he or she can select all production rates determined at that same "local" AMS facility for his/her calculations. However, this approach is only advantageous if AMS facilities could prove their long-time constancy of their produced data, as most production rates in use nowadays are from measurements some years to decades ago.

Finally, it is of absolute importance to emphasize that data described here is valid only with respect to the ^{10}Be/^{9}Be value stated for NIST SRM 4325, the reference to which all reported values are traceable. Any conclusions taken from this study are, thus fully valid in the sense of inter-comparability between the participating measurement facilities. Additional differences might occur if other non-traceable standards are in use at different AMS facilities.

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