Answer to Anonymous Referee #1: with respect to the Reviewer's comments, both in the first review and in the answer to our answer, we believe that most of the criticisms raised by the Reviewer lie in the misunderstanding of the aim and the target audience of our paper. Our "defensive" answer does not mean that we are not accepting criticisms or suggestions on how to

- 5 improve the paper (which we would always accept when constructive). It simply means that we think that some of the suggested changes will not bring benefit nor clarifications to the paper. Nonetheless, we have made many of the changes that the Reviewer suggested, and explained why we do not believe that some of the suggested changes are necessary for the goal and readership of our manuscript.
- 10 In the text that follows, we have continued the answer to the Referee's first comments. Since we finally did change some things that the Referee was pointing out in the first comment, we have highlighted the new changes in the first part of the answer in a darker underlined color and we have written them in Italic.
- 15 Summary comments: Thanks for the detailed review. We understand the perspective of Referee #1 with respect to the technicality of the paper and its broader interest for the surface processes community. However we don't agree completely with his/her assertions.

We would like to highlight that in this paper we describe a statistical method that defines a lower threshold useful for the interpretation of low-concentration samples. This method is known in

- 20 analytical chemistry but has never been applied to cosmogenic studies. The method is designed to be used by the final user, after the chemical procedure and the AMS measurements of the samples, and it is not simply an "*exercise done to understand the potential limits of one laboratory*". Rather it is designed to help the cosmogenic nuclide community users define the geomorphic meaning of samples with low nuclide concentration. In no way we suggest that the results which do not lie
- 25 significantly below the blank threshold cannot be used, but rather we use this threshold to determine whether or not a rate or age can be quantified or when we can only interpret the data in terms of a maximum age or minimum erosion rate.

In the following lines we will proceed with commenting the main concerns of Referee #1 (in black ink), especially considering the aim of our work and our targeted audience, which define the

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structure and meaning of our paper. A more detailed answer on the line-by-line comments and changes to the manuscript will follow in the next weeks.

Anonymous Referee #1: Summary: This ms applies what are considered standard approaches in
analytical chemistry to determining detection limits for the analysis of cosmogenic 10Be. The ms
uses a long term set of blanks and sample measurements from one lab but multiple operators to
make calculations and determine several statistical parameters for the detection of 10Be at levels
confidently above the blank.

The manuscript does not break new ground; it is an exercise done to understand the potential limits of one laboratory much more than a significant advance in cosmogenic nuclide science and therefore, not the type or style of paper for publication in a broad readership journal such as Esurf. Overall, this paper presents a data set that most cosmogenic labs have and have likely analyzed internally but the ms does little to advance cosmogenic nuclide science more broadly. As a referee and as a user of this literature, I consider the ms to be much more appropriate for an AMS-specific journal such as NIMs and as such suggest it be shortened and submitted for publication there rather

than eSurf. It just does not fit well nor will be it be of interest to most of the surface process community. It is technical in nature and does not have any significant geomorphic impact.

Significance and Readership: First of all, we would like to highlight that the aim of this paper is to address the issues that one encounters with low 10Be content samples (i.e., $< 10^5$ atoms) and to

- 20 provide a standardized method for the interpretation of the results. The paper is designed for the cosmogenic nuclide final users, including all the people who use cosmogenic nuclides to study earth-surface processes, but who are not necessarily familiar with the technicality of the Be-extraction/measurement procedure. In the last decades, the rapid expansion in the use of this technique has created a growing community of users that apply cosmogenic studies without the
- 25 detailed technical knowledge typical of the people who work in an institute where it is possible to perform Be-extraction and/or AMS measurements (e.g., the LLNL facility) and who may have only a basic knowledge of the technical aspects of these procedures. This paper aims also at this broader community and, as such, it is specifically designed not to be technical, because we want it to be suitable for non-technical users. Some technical information on the chemical procedure for
- 30 quartz extraction, Be-isolation, and the possible sources of blank contamination are, however,

additionally provided in the supplementary material. <u>Considering the importance of boron</u> <u>interference for samples with low 10Be content, we have added a sentence and some reference</u> <u>also in the main text, so that the readers can be aware of the necessary precautions that can be</u> <u>taken when dealing with these kind of samples.</u> Other detailed information on very specific technical aspects, such background AMS beam currents and blank counts, are already central topics of existing and excellent technical papers that the readers can access, if interested (we

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provide some recent summary papers as examples, e.g., Balco, 2011; Granger et al., 2013; and some technical papers that can be of interest for the more curious users, e.g., Schaefer et al., 2009; Rood et al., 2010; Merchel et al., 2013; Portenga et al., 2015; Corbett et al., 2016). Most of this
technical information, indeed, is available for internal usage of specific laboratories (e.g., to define the precision of the AMS machine and/or cleanliness of the laboratory) but are normally not included in the final AMS measurement report that is delivered to the users.

For the above-mentioned reasons, our paper does not deal with technical aspects of the AMS
measurements (for which, we believe, more suitable and technical papers already exist), but rather proposes a statistical procedure (not chemical nor isotopic) based on clear definitions of detection limits, which can be used as standard reference in the interpretation of cosmogenic nuclide measurements. As such, the paper targets a broad audience and it describes a method that can be applied by all cosmogenic nuclide users. Specifically, the method provides a statistical threshold to evaluate whether the nuclide concentration can be used to quantify an exposure age or erosion

- rate versus only limit the age or rate; in the latter case, the data is not rejected (nor there is the risk to *reject data that are likely real*); on the contrary, a limiting value can still be very helpful. This proposed method, additionally, can also be used in cases where low-concentration samples are not expected. In such cases, the user may not be aware of the precautions needed during the chemical
- 25 procedure, but he/she will still need to define how the results should be interpreted. For this goal, the paper describes a procedure that is commonly used in analytical chemistry to define a statistically significant threshold for low-concentration samples and provides a method for its application in the field of cosmogenic nuclide studies. Consequently, it does not describe a "new procedure", but rather we propose to apply this standardized method for cosmogenic nuclide studies, seeing as such a procedure is so far missing in the community. This aspect is very relevant, especially considering that a growing number of researchers will have to deal with low
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concentration samples (see Corbett et al., 2016). We believe that a common and standardized approach that defines a general rule for the interpretation of these data is timely and valuable, and that it can be of interest for a broad audience rather than only for the readers of a technical journal such as NIMs. For these reasons, we strongly believe that the paper will fit well a journal such as ESurf.

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Furthermore, the ms is proscriptive and narrow in its approach, which makes it less likely to be accepted by the community. The manuscript does not set the presented data in context because it does not include a critical evaluation of how previous workers have done blank corrections nor does it demonstrate how different blank corrections would change geomorphic outcomes of extant studies. There are several examples in the recent literature where workers have applied several different approaches to blank correction and tested the sensitivity of results to varying approaches; see for example Corbett et al., GRL (2017) on 10/26 ratios. These are not cited nor are they considered critically. The ms reviewed here is under-referenced, omitting numerous important citations both classical and recent that are germane.

As suggested from the referee, we have added in paragraph 2.3.1 a longer description of how other people have dealt with blank correction. We added the reference to Corbett et al (2017) in several points of the manuscript. This paper is, to our knowledge, the only published paper that discusses different blank corrections. They discussed it, however, only in terms of blank statistics, not

20 highlighting how the choice of a different approach would change the 10Be atoms in the samples or the geomorphic interpretation of a sample (this is also due to the fact that their statistics are very similar, so that differences between the various mentioned approaches would be negligible).

With respect to the blank subtraction methods, we have presented here three different approaches and discussed how their application would change the final interpretation of the results. So we do

- 25 not agree with Referee #1 when he/she said that we did not consider different blank subtraction methods or that we did not "demonstrate how different blank corrections would change geomorphic outcomes of extant studies". Nevertheless, in the revised version of the manuscript, we have added more information on how other people have dealt with this topic (e.g., Corbett et al., as suggested by Referee #1) and discuss their approach. We would also like to highlight that
- 30 very few papers discussed and reported in detail how the blank correction has been performed, and

even fewer papers additionally dealt with low 10Be content samples (i.e., $< 10^{5}$ ¹⁰Be atoms). As such, this information is not so easy to find within the existing literature.

This point raised from Referee #1 demonstrates how a common procedure for these important steps in the calculation of the cosmogenic results is still missing within the community. The way people normally deal with blank corrections is often not reported in the papers, but this could lead

5 people normally deal with blank corrections is often not reported in the papers, but this could lead to slightly different results and to a lack of comparability between different publications. This highlights once more why our paper will represent an important contribution for the cosmogenic nuclide community.

Also, we have already provided, as examples, some of the most recent summary papers (e.g.,
Balco, 2011; Granger et al., 2013) that provide an exhaustive explanation of the cosmogenic nuclide technique and its latest advances. Interested readers can go into the details of the references mentioned within these papers, based on their own specific interests. In the revised version of the manuscript, we will add more references to recent papers (e.g., Corbett et al., 2017), as suggested by Referee #1.

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Critically, the paper does not consider type 1 vs type 2 errors, that is in striving to be certain that 10Be is confidently detected, such as using 99.9% confidence, samples containing real 10Be are almost certainly being rejected. This will lead to errant science and must be considered head on in any revision before publication. It is a critical flaw and must be addressed before publication in any journal.

20 any journal.

For completeness of the paper, we have added the theoretical description of these two types of error in section 2.4.1. However, we would like to highlight that this information would not change the results of the proposed approach. It is true that depending on the chosen confidence level the values of the two errors would change, and is quite obvious that with a lower confidence interval

25 more samples could be used for a quantitative interpretation of ages or rates (i.e., the probability of the type II error would be reduced). However, decreasing the confidence interval would also increase the risk of using a sample for a quantitative interpretation when in reality the 10Be content of that sample may be in large part derived from laboratory contamination (the probability of the type I error would increase). In our opinion, accepting a higher risk of incurring in a higher type I error (thus reducing the confidence interval) is worse than accepting the risk of a higher type II error, and this is one reason for which we propose to use the LOD value as lower threshold for cosmogenic studies. We have better specified this point in the paper.

In this paper we describe an existing approach which uses high confidence intervals, and we

- 5 explain how to apply this approach to cosmogenic studies. We have reported the definitions of LOD and LOQ used in analytical chemistry (respectively representative of 99.9% and 99.9999% confidence intervals) and the users should be aware that changing the confidence interval would alter these definitions. Nonetheless, we have stated in the discussion and in the conclusion that whenever a user want to use a lower confidence interval (e.g., 2σ), the important point is to report
- 10 *it in the paper, together with all the necessary information that would make the results of that study comparable with others.*

Additionally, we would like to highlight that despite the chosen confidence interval, in no way "a sample containing real 10Be" would be rejected, as with this approach all the samples can be used. What changes is only the way with which we can interpret the geomorphic meaning of the

15 <u>samples (e.g., for the quantification of an age or a rate, or as limiting number, a still very helpful</u> <u>and meaningful result).</u>

Lastly, boron, an isobaric interference is neglected. It varies sample to sample and the means by which it is rejected varies between AMS facilities. It can be a very real component of the blank
measurements. At minimum, it needs to be presented and discussed in the context of the ms and the measurements within – better yet would be to consider it broadly across the community. Again, this is narrow, technical information of interest to a small section of the community but critical to the issues here.

We agree that this is a "narrow, technical information of interest to a small section of the community" and for the reasons already mentioned above, we did not go into the detail of this discussion. Additionally, as pointed out by the referee, this is an aspect that changes from AMS to AMS laboratories and its discussion goes far outside the aim of our paper and our targeted audience. Nevertheless, *we have added a sentence to acknowledge the importance of this aspect*

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when dealing with low-concentration samples and reported examples on the possible procedures to use in this case, with the related references for the interested users.

In summary, the ms is not appropriate for Esurf, is too narrowly focused on one approach to blank subtraction, ignores Type 1 vs Type 2 errors, does not consider 10B interference, and has little critical evaluation of the literature in which the current data need be considered in context. As is, it is a formulaic approach to analyzing an isotopic rather than geomorphic data set and not a significant advance of geomorphic science.

We have presented here three types of blank subtraction, an aspect that is not normally discussed
in cosmogenic papers. We have provided additional information using the work of Corbett et al. to include other possible approaches to blank corrections. We have explained why our paper does not want to be too technical (still, technical information can be found in the supplementary material), considering the aim and the target audience for this work, and we have provided two examples of how our proposed approach can be applied to geomorphic studies, discussing its consequences for the geomorphic interpretation of the samples (section 5.4). In the revised version of the paper we will include more examples, as suggested by Referee #1, so that the readers can have a broader idea of the impact of the proposed method on geomorphic studies.

For all these reasons, we believe that the paper is of broad interest and that it presents timely and necessary information for the cosmogenic nuclide community and for a correct interpretation of

20 low-concentration samples. Its publication in a broad journal such as ESurf, rather than in a technical journal, will surely be of benefit for many researchers within and outside the cosmogenic nuclide community.

25 Suggestions for improvement:

I would encourage authors to expand their selection of references in particular citing more studies that make very low level measurements and how these studies have dealt with blanks as well to cite some of the many excellent review papers since 2010 that compile both erosion rate and exposure age data. For example, Carlson and Nelson have recently both published very low concentration measurements from Greenland, one for glacial dating and the other for sediment tracing and neither of these studies are cited.

Thanks for this suggestion. We have added the two papers mentioned by the Referee plus some other study. Two of the "excellent review papers since 2010" (e.g., Balco, 2011; Granger et al., 2013) have already been cited in the manuscript as examples and more detailed literature can be found within these papers.

P2, Ln 25: most AMS measurements in the 10-15 and 10-16 range are not very precise, reword.

10 We have reworded the sentence.

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P2, Ln 27: This would be an appropriate place to cite Corbett et al. (2016) who review in great detail lab and AMS issues affecting detection including blanks and AMS beam currents. They in turn cites others such as the work on precision by Rood at the LLNL facility.

15 *We have added the reference; a direct citation to Rood et al. is already provided on line 28.*

P2, Ln 30-35: It is not clear why a standardized approach is needed or an improvement on the current approach; there is so much buried in blanks including which AMS, operator changes, real contamination. The paper would likely be better accepted by the community if it were to provide a means or variety of means by which the blank subtraction could be done. The way this section is written presumes the authors have defined "the" way to do blank correction not "a" way to do blank correction. This will not advance the science of AMS and 10Be.

We believe proposing a standardized approach to blank correction in cosmogenic nuclide studies that follows well established procedures in the field of analytical chemistry is important to have

25 comparable data and to give the users a reference approach that can be used to interpret the geomorphic meaning of low concentration samples. This is the aim of this paper.

The "variety of means by which the blank subtraction could be done" has been added to section 2.3.1 where it better fits within the content.

P3, Ln 10: This sentence is incorrect. 10Be IS naturally present in earth materials.

5 *We have changed the sentence.*

P3, Ln 17: This set of references fails to cite the original 3 references for nuclides in sediments – a critical oversight that needs to be remedied.

We have added the missing references.

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P4, Ln1: This sentence omits an important part of running blanks, determining the dark current or background of the AMS system including cross talk in the source. This needs to be mentioned and cited properly.

This is a very technical comment, and touches on issues that the final user of AMS data is in no
15 way involved with. We are not attempting to identify all sources of uncertainty in blanks, but rather we are suggesting a procedure for how end-users can determine if they may place uncertainty limits on their exposure ages or erosion rates, or if they can simply limit the age or rate.

P4, Ln 5-8: This is at least not correct for our lab and I think not correct for other labs. For us,
every sample including blanks gets the same reagent amount and same open beaker time.
Otherwise, how could we could compare process blanks and samples and do the subtraction in a meaningful way?

In our experience samples with different amount of quartz always require different amounts of acid and different time periods of open backers for the dissolution of the samples. Nonetheless, we have changed the sentence to include different possible procedures.

P4, ln 15-20: This section omits a critical issue in defining blanks on the AMS – boron as an isobaric interference and how that is handled. The process varies between AMS (some such as PRIME use GFM that completely removes 10B, others like LLNL make a very uncertain correction, others use post stripping). For low level samples, this is critical.

5 We have acknowledge the issue in the text.

P4, ln 21-22: This is one way to do it but if the same amount of carrier is added to all samples, then the ratios can be subtracted. This alternative approach needs to be acknowledged and cited.

We have changed the text.

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P4, ln 36: there needs to be more here. How do blanks change over time? Does contamination increase over time in labware?

As we have mentioned in the text, "this is a point that needs to be addressed for each laboratory and perhaps at the individual-user level".

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P5, Ln 7-10: This is far too simplistic. Only considering the upper value of the blank will result is rejecting data that are likely real. The blank subtraction process is a probabilistic one and different for different purposes. The goal of determining whether something is confidently detected is very different than the goal of best estimating the blank for subtraction. The paper would be much stronger if it considered this subtlety.

20 stron

As stated in previous responses, we have tried to clarify that when a sample yields a 10Be content that cannot be distinguished from the blank, the data does not need to be rejected as meaningless. This is an important point, and the referee's confusion on it highlights the need for this point to be clarified to the community. The maximum 10Be content of the blank distribution can still be

25 interpreted in terms of an exposure age or an erosion rate; when a sample's 10Be content falls below this threshold, we can still say that it has a maximum age or a minimum rate equal to that calculated for the upper limit of the blank distribution. P6, Ln 4: Unmentioned here is the fact that blanks are very imprecise measurements by their nature and because of the Poisson distributed counting statistics of AMS. The lowest blanks can contain only a count or two (or even none). Implicit in the discussion above is that the blanks are normally

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distributed (parametric statistics). If these issues were discussed, the paper would be much stronger. Some of this discussion is in section 3 and could be moved up.

Indeed, this is an important aspect of the paper and it is exactly why we discuss the possibility to use a different approach than the one recommended by the IUPAC, which assume a normal distribution. We clearly stated this issue at the beginning of Section 3 (only 4 lines below the

- 10 mentioned point of Referee#1) where we say that "This recommendation [intended as the IUPAC recommendation], however, assumes a normal distribution of values, which is rarely the case when dealing with low concentrations of an analyte" and explain later in section 4 why this is important and how it changes the results of the statistical interpretation. This is a critical aspect of the proposed method that is already discussed through the paper.
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P8, Ln 5: "Although a minimum of 20 values" this has been stated repeatedly. No need to state again.

Thanks for this comment. We have noted that we have repeated this sentence often through the paper. However, this sentence in this position helps us to explain why we use two different sets of

20 *blanks despite having a low number of blanks (n=8). We have removed this repetition from other parts of the text.*

P8, Ln 35: It is not clear how the average blank constrains any temporal variance? Please explain.

The average blank value constrains the temporal variance because the blanks are created in a

25 relatively short period of time. This is explained few lines above this point where we stated that "In the average-blank correction, all the blanks processed in multiple batches by one operator over a limited time frame [...] are used to obtain a representative value of 10Be atoms for the blanks [...]". P9, ln 18: I find this paragraph very hard to follow and not very informative. A table or graphic would convey the same information much more effectively. Some of the information is summarized in tables but not all of it.

5 We have created a new table (new Table 3) and wrote all these values there.

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P11, ln 13: This is a critical mis-understanding of Type 1 and Type 2 errors. Here, the authors suggest that, "In general, the use of the long-term laboratory blanks (being based on many blank measurements) guarantees more reliable values for the statistics of the blank distribution and for the calculation of the determination limits; as such, they may be preferred." The approach the

authors suggest is very likely to introduce errors of rejection for data (samples) that contain actual 10Be above blank levels.

This is a critical aspect of the paper that we have focused on throughout the text, in the figures and tables. It is not a misunderstanding of the two types of statistical error, but rather the

- 15 description of how the choice of the blank distribution to use for the blank correction may change and affect the interpretation of the results. And again, no data will be rejected, only a higher number of samples can only be used as limiting erosion rates or exposure ages, as we show in the manuscript. This is exactly why it is important to choose the correct method for the blank subtraction and for the calculation of the threshold, and is one of the main conclusion of the paper.
- 20 We have demonstrated through the paper that these elements will strongly change the results of the statistical approach and the way people can use and interpret the data.

Also, we have stated in the following line that "Nevertheless, when the long-term blank ensemble shows a large variance, the assumption of unchanging laboratory conditions is unlikely to be valid, and the blank measurements are unlikely to be representative of the variation occurring within a

25 single batch. Under these circumstances, and when there is an acceptable number of blank measurements available (at least 20; Bernal, 2014), a set of blanks obtained from a single operator over a shorter time interval may be favored for the calculation of the threshold". We are discussing here the results of our proposed work, differentiating between the several approaches that we describe through the paper. To report only one sentence and say that we are suggesting something wrong, omitting all the other part of the discussion, would give an overnegative impression of our work and of the detailed analyses we have done.

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Section 5.4: This is not an adequate critical review of what others have done with low activity samples. The data tables for AMS would be much more informative if they included the standard(s) to which the ratios were normalized, more about the boron counts and rejection procedures, comparison of sample beam currents to standard beam currents, the number of gated 10Be counts, and the actual uncertainties of the measured ratios.

We have included into table 2 the standard to which the ratios were normalized and the amount of added carrier. The AMS uncertainties associated with the measured ratios are already included in the table, whereas the other information are not in our possession, since they have not been

included in the AMS reports or were not reported within the publications.

15 Also, we have carefully read some of the papers that the Referee suggested as examples for publications with low-concentration samples. Unfortunately, due to missing information about sample or blank values in those publications, we were not able to use them as examples in our discussion on the "Implication for geomorphic applications".

In particular, the paper of Nelson et al (2014) report the average blank ratio used for the blank

20 correction but not the 9Be amount added in the carrier nor the carrier concentration. Considering that we need to calculate the LOD and LOQ values using the 10Be atoms included in the blanks, without this information, we cannot convert the blank ratio into 10Be atoms and thus we cannot calculate the desired thresholds.

The paper of Corbett et al. (2017) reports data form previous publications. Looking in detail into
these other papers (Corbett et al., 2011 and 2013), we have found that the AMS ratios for the samples have only been reported after the blank correction. The raw AMS results for the samples are missing. Additionally, these authors performed the blank correction using two different averaged blank ratios (one derived from commercial blanks and one derived from beryl blanks). Not knowing which samples have been corrected with which blank value, we cannot re-calculate

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the samples' ratios before the blank correction. So also for that paper we are unable to calculate the 10Be content of the samples.

In the paper of Carlson et al. (2014) all the necessary information for the blanks are reported, but unfortunately all of the AMS data for the samples are missing.

5 As we have already mentioned above, "very few papers discussed and reported in detail how the blank correction has been performed, and even fewer papers additionally dealt with samples with low 10Be content (i.e., < 10⁵ 10Be atoms). As such, this information is not so easy to find within the existing literature. This highlights once more why our paper is worth to be published and why it will be an important contribution for the cosmogenic nuclide community.