Determination limits for cosmogenic ^{10}Be and their importance for geomorphic applications

S1 – Supplementary Text

Cosmogenic nuclides are widely used for studying and quantifying geomorphic processes. Because of the broadness and complexity of the topic, we do not intend this text to be an exhaustive review of the method, because many detailed papers already exist on this argument (Nishiizumi et al. 1989; Lal, 1991; Brown et al., 1992; Biermann et al., 1996; Gosse and Phillips, 2001; von Blanckenburg, 2005; Dunai 2010; Balco 2011 among others). Instead, we provide a general and simplified description of how the technique can be used in rapidly eroding landscapes, particularly in the case of samples with low ^{10}Be content. Additionally, we provide a description of how to estimate the amount of material to be collected in the field, and suggestions on how to minimize the loss of quartz during the chemical treatment of samples (Portenga et al., 2015; Corbett et al., 2016).

S2 – Challenges arising from samples with low ^{10}Be/^{9}Be ratios

In a sample with low ^{10}Be content, the sample's ^{10}Be/^{9}Be ratio can be very low, and potentially drop below the range that can be precisely measured by an AMS. In cases where a low ^{10}Be content is anticipated, higher ^{10}Be/^{9}Be ratios can be achieved in two ways: 1) by increasing the ^{10}Be content in the sample, and/or 2) by reducing the amount of added ^{9}Be carrier. However, too little carrier added to the samples can result in problematic AMS measurements, so this option may be discarded for samples with low ^{10}Be content. Alternatively, a longer AMS measurement-time or a higher current terminal voltage used during the measurements can help to increase the counts of Be (Schaefer et al., 2009; Balco, 2011; Rugel et al., 2016).

The ^{10}Be content in a sample can be increased by 1) processing more material in the laboratory, and/or 2) minimizing the amount of quartz lost during the chemical procedure (Balco et al., 2011). Indeed, an insufficient amount of quartz can result in a low ^{10}Be content in the sample, even for old surfaces or low denudation rates. As such, sample weight considerations are particularly important when dealing with very young surfaces or rapidly evolving landscapes, whose low ^{10}Be content could require high quantities of the target mineral to increase the ^{10}Be concentration. A general description of how to estimate the necessary amount of material to collect in the field can be found in Sect. S1 and S3 or in the work of Gosse and Phillips (2001).

An alternative or additional strategy to increase the ^{10}Be content in a sample is to minimize the loss of quartz during the chemical procedure. Indeed, samples must undergo physical and chemical processing to concentrate and purify the target mineral and to isolate Be. Depending on the applied method (e.g. Brown et al., 1991; Kohl and Nishiizumi, 1992; von Blanckenburg et al., 2004; Binnie et al., 2015; Corbett et al., 2016), the procedure for quartz purification can result in the loss of a large amount of material (up to 50% when starting with relatively pure quartz, and even more for samples with mixed mineralogy), particularly when samples are treated with concentrated acids. Sample preparation procedures that can be adopted to help minimize the loss of quartz, use, for example, hexafluorosilicic acid (($H_3O)_2SiF_6$) or ortho-phosphoric acid prior to the HF-etching steps to increase the sample's quartz yield (Merchel et al. 2008; Schaefer et al., 2009; Mifsud et al. 2013; see section S4 for more information about chemical treatments). However, this approach

may affect the effectiveness of the removal of meteoric [10]Be and its application may be evaluated case by case.

S3 - Increasing the yield of [10]Be from a sample

Here, we describe a procedure to estimate the amount of material that should be collected in the field to yield sufficient [10]Be for analysis (Gosse and Phillips, 2001) based on (1) the expected concentration of [10]Be in the quartz, and (2) the sample composition and material loss during preparation.

S3.1 - Estimating the concentration of [10]Be in the target mineral

The concentration of [10]Be accumulated in the target mineral (N, atoms g^{-1}) is a function of the local production rate and the surface exposure time or the erosion rate. Hence, from a non-eroding surface, the [10]Be concentration (N) can be estimated as the product of the local production rate at the surface $P(0)$ (atoms g^{-1} yr^{-1}) and the estimated exposure age t (yr) (because we are considering young exposure ages, we can neglect nuclide decay):

$$N = P(0) \cdot t \,. \tag{S1}$$

Alternatively, from an eroding surface, N can be calculated as a function of the local production rate $P(0)$ (atoms g^{-1} yr^{-1}), bedrock density at the sample site ρ (e.g. 2.65 g cm^{-3} for quartz-rich lithologies), attenuation length Λ (ca. 160 g cm^{-2} for spallation reactions; Granger and Smith, 2000), and the estimated erosion rate ε (cm yr^{-1}) (because we are considering rapidly eroding surfaces, we can neglect nuclide decay here, too).

S3.2 - Amount of clean quartz needed for the Be isolation procedure

To calculate the amount of clean quartz that should be processed, W_S (g), we can use the following equation:

$$W_S = \frac{\left[W_{C,S} \cdot \frac{N_A}{M_{^9Be}} \cdot \left(\frac{^{10}Be}{^9Be} \right)_S \right] - \left[W_{C,Blk} \cdot \frac{N_A}{M_{^9Be}} \cdot \left(\frac{^{10}Be}{^9Be} \right)_{Blk} \right]}{N} \tag{S2}$$

where N_A refers to the Avogadro number, defined as the number of atoms contained in 1 mole of a material (6.022 x 10^{23} atoms mole^{-1}), $M_{^9Be}$ describes the molecular mass of [9]Be (9.012 g mole^{-1}), and N (*atoms g^{-1}*) is the [10]Be concentration in the sample calculated as explained in the previous section. For this estimate, the optimal [10]Be/[9]Be ratio for the samples $\left(\frac{^{10}Be}{^9Be} \right)_S$ that can be precisely measured by an AMS must be known; many facilities are optimized to measure a ratio of 10^{-13}. This ratio, however, could be difficult to obtain for samples with very low [10]Be content, in which case chemical treatments may be adjusted to maximize the amount of quartz extractible from the collected material (see following chapter). Many laboratories add a uniform weight of [9]Be carrier

to samples and blanks ($W_{C,S}$ and $W_{C,Blk}$), and a blank $^{10}Be/^9Be$ ratio $\left(\dfrac{^{10}Be}{^9Be}\right)_{Blk}$ can be estimated from long-term blank measurements carried out in the laboratory. With this information, the required amount of clean quartz W_S (g) can be obtained from Eq. (S2).

S3.3 - Estimating the amount of material to collect in the field

To calculate the amount of material to collect in the field to yield a desired mass of quartz for analysis (W_S), three factors need to be taken into account: 1) the relative yield of quartz following the chemical cleaning procedure, Q_{Chem} (%); 2) the percentage of quartz in the rocks/sediment of the study site, Q_{Litho} (%); and 3) the relative yield of the grain size that will be used for sample preparation, gs (%). Even for pure quartz, the loss during cleaning can be up to 50%. The total amount of sample to collect in the field, S (g), can be obtained dividing the estimated amount of clean quartz W_S (g) by the three parameters listed above, as:

$$S = \frac{W_S \cdot 10^6}{Q_{Chem} \cdot Q_{Litho} \cdot gs} .$$

(S3)

For example, considering a local production rate of 20 ^{10}Be atoms g^{-1} yr^{-1} in quartz and an expected exposure age of 1000 years for a non-eroding surface, the concentration of ^{10}Be in quartz would be 20,000 atoms g^{-1}. At the same site, a surface eroding at a rate of 0.05 cm yr^{-1} would yield 24,000 ^{10}Be atoms g^{-1}. In the case of a 1000 year-old surface, for an average $^{10}Be/^9Be$ laboratory blank of 10^{-15} and a ^9Be carrier addition of 0.15 mg, the amount of clean quartz to dissolve during the chemical procedure to achieve a $^{10}Be/^9Be$ ratio of 10^{-13} is 49.6 g. Finally, if 49.6 g of clean quartz is needed for the chemical Be isolation procedure, and assuming that 50% (Q_{Chem}) of the quartz remained after the cleaning procedure, the sampled boulder contains 20% (Q_{Litho}) of quartz, and 15% (gs) of the sample of the desired grain size remains after crushing, the amount of material to collect in the field is ~3.3 kg (the values of Q and gs used as an example here are derived from laboratory observations made during sample preparation at the University of Potsdam, Germany). Other examples and details for different cosmogenic nuclides can be found in Gosse and Phillips (2001).

S4 - Chemical Procedure

Here, we discuss how various alternatives in sample preparation procedures may help minimize the loss of quartz and may be adopted for samples with expected low ^{10}Be content. For detailed descriptions and quantitative information on the chemical procedures, the reader can refer to Hunt et al., (2008), Merchel et al., (2008 and 2013), Binnie et al., (2015), Portenga et al., (2015), and Corbett et al., (2016).

S4.1 - Quartz extraction and purification

The separation of quartz from other minerals is achieved by applying standard techniques including crushing, sieving, magnetic and/or density separation, commonly followed by acid

treatment(s). Magnetic separation of the desired grain size (normally between 250 and 500 µg; Granger et al., 1996; Gosse and Phillips, 2001) can be used to reduce the amount of magnetic minerals in the sample and it is normally followed by a cleaning step with diluted hydrochloric acid (HCl). For samples with low ^{10}Be concentrations, organic material, carbonates, and iron-coatings of minerals can be removed with a mixture of HCl and hydrogen peroxide (H_2O_2) prior to the magnetic separation to avoid the loss of quartz to the magnetic (non-used) fraction. Further reduction of a sample's non-quartz components can be achieved by using Hexafluorosilicic acid (($H_3O)_2SiF_6$) (Merchel et al., 2008) or by boiling the sample in ortho-phosphoric acid (Schaefer et al., 2009; Mifsud et al., 2013). However, the use of these acids may reduce the effective removal of meteoric Be, so that their use may be carefully considered. A final step in quartz purification is achieved through treatments using a mixture of diluted hydrofluoric acid (HF), which dissolves silicate minerals (including quartz), and nitric acid (HNO_3), which dissolves metal compounds (Brown et al., 1991; Kohl and Nishiizumi, 1992) and helps to prevent the precipitation of AlF_3 from silica-oversaturated solutions. At least one treatment with hydrofluoric, nitric, or hydrochloric acid is performed also to remove adsorbed meteoric ^{10}Be from the grain surfaces or from within micro-cracks (Brown et al., 1991). While these final HF etching steps are very effective in producing a very high percentage of pure quartz for the sample, some of the quartz is dissolved in these steps. Hence, maximizing the percentage of quartz in the sample prior to HF etching helps to minimize the loss of quartz by reducing the number of etching steps that are needed.

Naturally occurring ^{9}Be in samples can potentially pose a problem for samples with low ^{10}Be content (Merchel et al., 2013; Corbett et al., 2016), as the addition of ^{9}Be carrier can result in ^{10}Be/^{9}Be ratios that are too low to be precisely measured by an AMS. Although the natural ^{9}Be is usually negligible compared to what is added from the ^{9}Be carrier (Bierman et al., 2002), it can occur naturally in high quantities in pegmatites and alkali rocks, or in soils/sediments derived from these rocks (Portenga et al., 2015). A correction for naturally occurring ^{9}Be can be made based on the Be concentration in an aliquot of HF-etched clean quartz, as naturally occurring ^{9}Be in amounts that require correction will dominate the total Be content of an etched sample. We recommend this procedure to become common practice.

S4.2 - Be isolation

Following HF-etching, many non-BeO components typically remain in the sample, which can reduce or compromise the stable ion beam currents during the AMS measurement (Hunt et al., 2008; Merchel et al., 2008; Corbett et al., 2016). Aluminum and titanium are often the most critical elements to remove from the dissolved quartz samples; this can be done using liquid chromotography, often with the addition of solvent extraction or selective pH precipitation steps.

The different steps performed in this procedure depend on the laboratories in which the samples are processed. The final cleaning usually ensures the removal of any remaining meteoric Be and of other impurities as well as the destruction of fluorides formed during previous HF etching. The addition of ^{9}Be carrier is commonly performed to obtain a sufficient quantity of Be for analysis

and to calculate the ^{10}Be content from the measured ^{10}Be/^{9}Be ratio. After the sample dissolution, all the cations and anions, except Beryllium, are removed mainly through anionic and cationic resin exchange. Subsequently, Be is co-precipitated as Be hydroxide and then calcinated to BeO (e.g., von Blanckenburg et al., 2004, 2016). The last step of target packing follows standard procedures of the laboratories where the AMS measurements are performed.

S5 - Possible contamination sources for the blanks

Apart from ^{10}Be contained in the ^{9}Be carrier (a discussion of which can be found in the main text), other sources of ^{10}Be that would result in blank contamination include: (1) chemicals and disposable labware that come into contact with the sample; (2) re-used labware, beakers, resins etc., (3) poor laboratory practice that results in transfer of sample material during processing; (4) airborne dust, which contains ^{10}Be that has originated from atmospheric production, been recycled from Beryllium sources in the laboratory, or has been brought into the laboratory by users; and (5) the cathode material (e.g. Cu, Al) and conducting matrix (e.g. Nb, Ag) with which the final Be product is mixed prior to target pressing. AMS related effects that could be a source of ^{10}Be counts in the detector, and thus could be included in the final measurement, include (1) memory issues, where ^{10}Be from prior samples is mobilized and reaches the detector, and (2) interference, where a false-positive signal response occurs in the detector despite the absence of any ^{10}Be, such as can happen due to the presence of the ^{10}B isobar in the ^{10}Be region of the detector.

S6 – Blank corrections and error propagation

Our 61 long term blanks processed at the GFZ laboratory show a large scatter between the data. This is also highlighted by the probability density function of the blank values, which results best-fitted by a negative binomial distribution. When performing the blank correction, one has to choose a value that best represents the blank distribution. Here, we select both the mean and the median value, to evaluate the differences resulting from these two different blank correction approaches. The results are shown in Fig. S1 and Table S1.
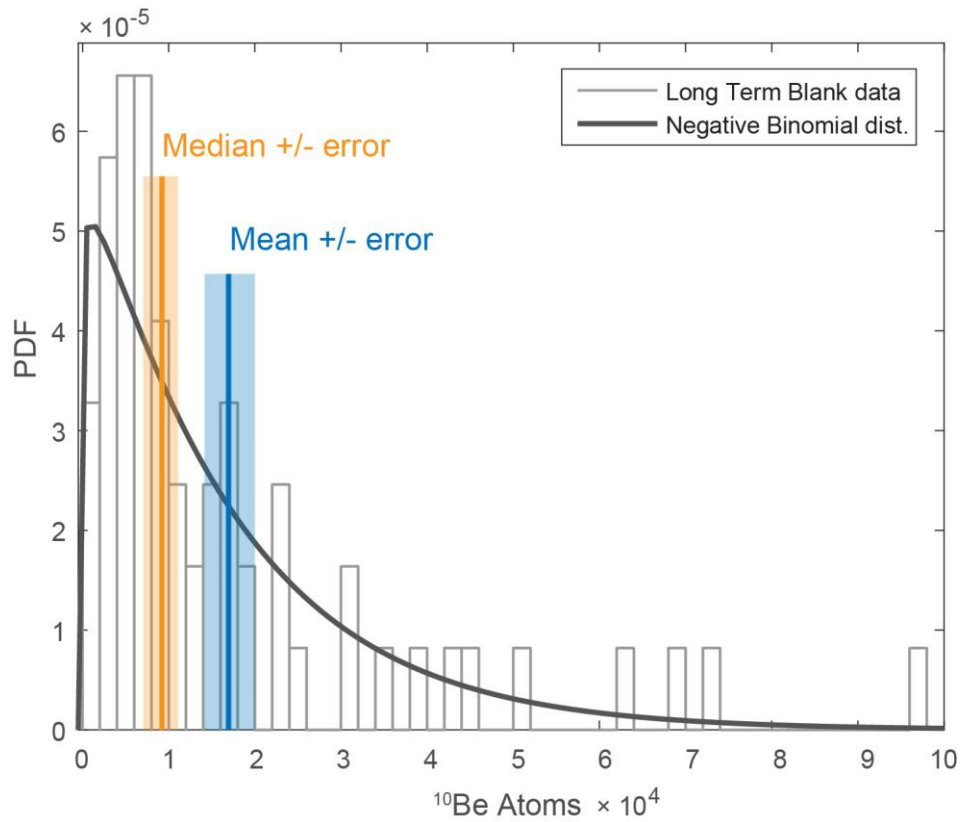
Figure S1. Blank distribution fitted by a negative binomial. Note the differences between the mean and median values of the distribution. For the 61 blank values $\mu = 1.72 \times 10^4$ ^{10}Be atoms, $\varepsilon_\mu = 0.25 \times 10^4$ ^{10}Be atoms, median $= 0.93 \times 10^4$ ^{10}Be atoms, $\varepsilon_m = 0.16 \times 10^4$ ^{10}Be atoms. For the eight blank values (not represented in the figure): $\mu = 0.76 \times 10^4$ ^{10}Be atoms, $\varepsilon_\mu = 0.14 \times 10^4$ ^{10}Be atoms, median $= 0.70 \times 10^4$ ^{10}Be atoms, $\varepsilon_m = 0.09 \times 10^4$ ^{10}Be atoms.

Table S1. Corrected concentrations for the 57 [10]Be samples. Carrier addition is of ca. 0.15 mg of [9]Be, corresponding to ca. 1e[19] [9]Be atoms. For the *average blank correction* and the *long-term blank correction*, blank subtraction was performed using both the mean and the median value of the blank distributions; error propagation was performed using the respective standard errors.

Sample Number	Single-Batch Blank Correction [10Be Atoms]		Average Blank Correction [10Be Atoms]				Long Term Blank Correction [10Be Atoms]			
			median value		mean value		median value		mean value	
	corrected	Err %	corrected	Err %	corrected	Err %	corrected	Err %	corrected	Err %
1	1.36E+04	35.5%	9.15E+03	50.3%	8.50E+03	55.3%	6.66E+03	71.6%	-1.03E+03	-500.6%
2	1.62E+04	37.4%	1.35E+04	41.5%	1.28E+04	44.2%	1.10E+04	52.1%	3.28E+03	184.2%
3	1.77E+04	30.5%	1.32E+04	39.2%	1.26E+04	41.9%	1.07E+04	49.6%	3.03E+03	186.9%
4	**3.23E+04**	**21.7%**	**2.79E+04**	**24.6%**	**2.72E+04**	**25.4%**	2.54E+04	27.5%	1.77E+04	40.9%
5	3.55E+04	19.3%	3.10E+04	21.5%	3.04E+04	22.2%	2.85E+04	23.8%	2.08E+04	34.0%
6	3.96E+04	18.8%	3.51E+04	20.8%	3.45E+04	21.3%	3.26E+04	22.7%	2.49E+04	30.7%
7	4.11E+04	25.3%	4.07E+04	24.3%	4.01E+04	24.8%	3.82E+04	26.1%	3.05E+04	33.3%
8	4.18E+04	21.4%	4.14E+04	20.2%	4.08E+04	20.7%	3.89E+04	21.7%	3.13E+04	27.8%
9	4.57E+04	16.4%	4.12E+04	17.8%	4.05E+04	18.2%	3.87E+04	19.2%	3.10E+04	24.7%
10	4.40E+04	21.4%	4.36E+04	20.3%	4.30E+04	20.7%	4.11E+04	21.8%	3.35E+04	27.4%
11	4.72E+04	16.8%	4.27E+04	18.2%	4.21E+04	18.7%	4.02E+04	19.6%	3.25E+04	25.0%
12	5.03E+04	17.5%	4.58E+04	18.9%	4.52E+04	19.3%	4.34E+04	20.2%	3.57E+04	25.1%
13	6.08E+04	14.3%	5.63E+04	15.2%	5.57E+04	15.5%	5.38E+04	16.1%	4.61E+04	19.3%
14	6.13E+04	20.0%	6.10E+04	19.5%	6.03E+04	19.7%	5.85E+04	20.4%	5.08E+04	23.8%
15	7.17E+04	15.7%	6.90E+04	16.0%	6.83E+04	16.2%	6.65E+04	16.7%	5.88E+04	19.2%
16	7.71E+04	18.1%	7.90E+04	16.8%	7.83E+04	17.0%	7.65E+04	17.4%	6.88E+04	19.6%
17	8.21E+04	14.8%	8.24E+04	14.3%	8.18E+04	14.5%	7.99E+04	14.8%	7.22E+04	16.6%
18	8.28E+04	15.5%	8.68E+04	13.9%	8.62E+04	14.1%	8.43E+04	14.4%	7.66E+04	16.1%
19	9.43E+04	15.5%	9.46E+04	15.1%	9.40E+04	15.3%	9.21E+04	15.6%	8.44E+04	17.2%
20	9.57E+04	14.0%	9.53E+04	13.6%	9.47E+04	13.8%	9.28E+04	14.1%	8.52E+04	15.5%
21	1.06E+05	14.4%	1.06E+05	14.2%	1.05E+05	14.3%	1.03E+05	14.6%	9.54E+04	15.9%
22	1.26E+05	12.4%	1.25E+05	12.2%	1.25E+05	12.3%	**1.23E+05**	**12.5%**	**1.15E+05**	**13.4%**
23	1.59E+05	16.8%	1.56E+05	17.0%	1.55E+05	17.1%	1.54E+05	17.3%	1.46E+05	18.3%
24	1.54E+05	11.6%	1.62E+05	10.6%	1.62E+05	10.6%	1.60E+05	10.8%	1.52E+05	11.4%
25	1.73E+05	11.3%	1.73E+05	11.1%	1.72E+05	11.2%	1.71E+05	11.3%	1.63E+05	11.9%
26	1.77E+05	10.8%	1.77E+05	10.6%	1.77E+05	10.7%	1.75E+05	10.8%	1.67E+05	11.4%

27	1.82E+05	10.6%	1.82E+05	10.4%	1.82E+05	10.5%	1.80E+05	10.6%	1.72E+05	11.1%
28	2.12E+05	11.2%	2.14E+05	10.9%	2.14E+05	10.9%	2.12E+05	11.0%	2.04E+05	11.5%
29	2.45E+05	7.8%	2.38E+05	8.1%	2.38E+05	8.1%	2.36E+05	8.2%	2.28E+05	8.5%
30	4.49E+05	7.4%	4.51E+05	7.3%	4.50E+05	7.3%	4.48E+05	7.4%	4.40E+05	7.5%
31	4.69E+05	6.9%	4.71E+05	6.9%	4.71E+05	6.9%	4.69E+05	6.9%	4.61E+05	7.0%
32	4.94E+05	6.0%	4.94E+05	5.9%	4.94E+05	6.0%	4.92E+05	6.0%	4.84E+05	6.1%
33	5.01E+05	6.8%	5.03E+05	6.7%	5.03E+05	6.7%	5.01E+05	6.8%	4.93E+05	6.9%
34	7.46E+05	5.0%	7.43E+05	5.0%	7.42E+05	5.0%	7.40E+05	5.0%	7.33E+05	5.1%
35	8.40E+05	5.5%	8.40E+05	5.5%	8.39E+05	5.5%	8.37E+05	5.5%	8.29E+05	5.6%
36	1.24E+06	4.9%	1.24E+06	4.9%	1.24E+06	4.9%	1.24E+06	4.9%	1.23E+06	4.9%
37	1.65E+06	4.5%	1.65E+06	4.5%	1.65E+06	4.5%	1.65E+06	4.5%	1.64E+06	4.5%
38	2.27E+06	4.0%	2.27E+06	4.0%	2.27E+06	4.0%	2.27E+06	4.0%	2.26E+06	4.0%
39	2.83E+06	3.9%	2.83E+06	3.9%	2.83E+06	3.9%	2.83E+06	3.9%	2.82E+06	3.9%
40	2.83E+06	3.7%	2.84E+06	3.7%	2.83E+06	3.7%	2.83E+06	3.7%	2.82E+06	3.7%
41	3.22E+06	3.6%	3.22E+06	3.6%	3.22E+06	3.6%	3.21E+06	3.6%	3.21E+06	3.6%
42	4.05E+06	3.5%	4.06E+06	3.5%	4.06E+06	3.5%	4.06E+06	3.5%	4.05E+06	3.5%
43	4.21E+06	3.8%	4.21E+06	3.8%	4.21E+06	3.8%	4.20E+06	3.8%	4.20E+06	3.8%
44	4.30E+06	3.5%	4.30E+06	3.5%	4.30E+06	3.5%	4.30E+06	3.5%	4.29E+06	3.5%
45	4.31E+06	3.6%	4.31E+06	3.5%	4.31E+06	3.5%	4.31E+06	3.5%	4.30E+06	3.6%
46	5.52E+06	3.5%	5.52E+06	3.5%	5.52E+06	3.5%	5.52E+06	3.5%	5.51E+06	3.5%
47	5.85E+06	3.4%	5.85E+06	3.4%	5.85E+06	3.4%	5.85E+06	3.4%	5.84E+06	3.4%
48	5.97E+06	3.4%	5.98E+06	3.4%	5.98E+06	3.4%	5.98E+06	3.4%	5.97E+06	3.4%
49	6.03E+06	3.3%	6.03E+06	3.3%	6.03E+06	3.3%	6.02E+06	3.3%	6.02E+06	3.3%
50	9.03E+06	3.3%	9.03E+06	3.3%	9.03E+06	3.3%	9.03E+06	3.3%	9.02E+06	3.3%
51	9.15E+06	3.3%	9.15E+06	3.3%	9.15E+06	3.3%	9.15E+06	3.3%	9.14E+06	3.3%
52	9.38E+06	3.3%	9.39E+06	3.3%	9.39E+06	3.3%	9.39E+06	3.3%	9.38E+06	3.3%
53	1.19E+07	3.3%	1.19E+07	3.3%	1.19E+07	3.3%	1.19E+07	3.3%	1.19E+07	3.3%
54	2.25E+07	3.1%	2.25E+07	3.1%	2.25E+07	3.1%	2.25E+07	3.1%	2.25E+07	3.1%
55	2.42E+07	3.1%	2.42E+07	3.1%	2.42E+07	3.1%	2.42E+07	3.1%	2.42E+07	3.1%
56	2.98E+07	3.1%	2.98E+07	3.1%	2.98E+07	3.1%	2.98E+07	3.1%	2.98E+07	3.1%
57	4.27E+07	3.1%	4.27E+07	3.1%	4.27E+07	3.1%	4.26E+07	3.1%	4.26E+07	3.1%

In *Italics*: Values below the $LOD_{8, NB}$ (*single-batch blank correction* and *average blank correction*) and below the $LOD_{61, NB}$ (*long-term blank correction*). In **Bold**: Maximum values' difference when the different blank correction methods are applied.

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