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Abstract: The performance of the Trondheim 1 MV AMS system for 10Be measurements has been investigated. While the initial acceptance tests were done using the 1+1+ charge state, we concluded that the 2+2+ charge state offers superior measurement conditions. With this setting, the source-to-detector efficiency is 5.4±0.2 % and a 10Be/9Be background level of 3.4±0.8·10-15 was achieved. Standard samples (n=14) with a high 10Be content of 10Be/Be=2.7·10-11 could be reproduced to a 0.4 % precision, leaving counting statistics as the limiting uncertainty for most samples. The accuracy of the system was also tested using 5 secondary standards of different 10Be content. The results corroborate the precision achieved with the standard sample and demonstrate the accuracy of the system.

We went through the comments of the reviewers and addressed their points in the revised manuscript. Here we outline the changes that we made to improve the issues pointed out by the reviewers:

Reviewer 1

1. **Why +1+2 got high blank values? Reasons or opinions should be added.**
    - We addressed this point more detailed in the article. The main reason is the more abundant boron in the 2+ state after the degrader foil, which leads to a saturation in the detector.
2. **Sample description and treatment in discussion should be moved to experiments.**
    - The description of the samples has been moved to sections 2 (experiments). The paragraphs were slightly adjusted to fit the narrative.
3. **Three peaks appear in Fig. 3. They are more apparent in Fig. 6. What is the peak besides boron and beryllium? Sum peak? Annotation may be need in the figures.**
    - A deeper discussion about the spectra, especially the not mentioned oxygen peak, as well as the boron and pile-up events, was added to the article. Since this could not be compressed into some small sentences, we did not add this to the figure captions.
4. **By the Fig.7, ESA bias voltage of 44.45 - 44.5 kV seems better than 44.3 kV. Why was 44.3 kV selected as the optimum condition?**
    - While the boron count rate at these voltages are indeed lowest, it also has a significantly reduced ^{10}Be yield. This is not very apparent on the log-plot at first sight, so that a statement in the article was added. We considered using a linear scale for the Be counts in this plot to make this point more visible, but we came to the conclusion that the figure is not well readable using two different scales.

Reviewer 3

- Several language issues that were pointed out by the reviewer, have been fixed.
- A comment about the separation of boron in the 1+2+ charge state was addressed by further explaining that the problem is actually the more abundant boron in comparison to the 1+1+ state.
- A comment about a possible cause for differences in the 1+ transmission among laboratories was incorporated in the discussion.
- Plots have been reworked to provide better visibility of the axes.

First ^{10}Be measurements at Trondheim 1 MV AMS

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Declarations of interest: none

Abstract

The performance of the Trondheim 1 MV AMS system for ^{10}Be measurements has been investigated. While the initial acceptance tests were done using the 1+1+ charge state, we concluded that the 2+2+ charge state offers superior measurement conditions. With this setting, the source-to-detector efficiency is 5.4±0.2 % and a ^{10}Be/^{9}Be background level of $3.4\pm0.8\cdot10^{-15}$ was achieved. Standard samples (n=14) with a high ^{10}Be content of ^{10}Be/Be=$2.7\cdot10^{-11}$ could be reproduced to a 0.4 % precision, leaving counting statistics as the limiting uncertainty for most samples. The accuracy of the system was also tested using 5 secondary standards of different ^{10}Be content. The results corroborate the precision achieved with the standard sample and demonstrate the accuracy of the system.

Keywords: AMS; Low-energy AMS;10Be;Accelerator mass spectrometry

1 Introduction

Downsizing accelerators and spectrometers was an important development in the history of accelerator mass spectrometry (AMS). This trend was led by instruments designed for radiocarbon measurement [1, 2] as it is the largest application of AMS. While these instruments were also used for different isotopes, their initial design is limiting the performance and requires adjustments [3]. Some AMS systems, such as the 1 MV AMS from High Voltage Engineering (HVE), which is in use in Trondheim, are specifically designed for the measurement of different isotopes [4], but have primarily been used for radiocarbon measurements [5, 6]. Extended investigations of other isotopes are therefore rare and often come to different results in the achievable performance [7-9].

We concluded that the performance depends on details either of the instruments or of its tuning. Therefore, we decided to investigate the behavior and performance of our AMS system to identify critical parameters that influence the results.

2 Experiment

The design of the 1 MV AMS system at NTNU is similar to systems in other laboratories, e.g. Centro Nacional de Aceleradores (CNA) in Sevilla. It is built for measuring ^{14}C, ^{10}Be and ^{26}Al. The system would also allow measurements of other isotopes, such as ^{41}Ca and ^{129}I, if additional magnet power supplies were provided. The method suggested for Be measurements is the extraction of BeO from the ion source (SO-110B) and the use of a degrader foil for the separation of boron interferences. The bouncing injector allows fast (≈100 Hz) sequential injection of different ion masses (^{9}BeO, ^{10}BeO) into the accelerator where the molecular break up happens within the Ar stripper gas. The analyzing magnet

separates the ^{9}Be beam for measurement in an off-axis Faraday cup and allows the selection of the desired charge state. The mass 10 beam passes a silicon nitride (SiN) degrader foil where it undergoes an element specific energy loss such that ^{10}B and ^{10}Be can be separated in the electrostatic analyzer (ESA). The ESA has a plate gap of 25 mm and is symmetrically powered by two unipolar 60 kV power supplies. The final particle identification is done in the gas ionization detector (GIC) at the end of the beam line (Fig. 1). A detailed description of the instrument is given in previous publications [4, 5, 7, 8].

The Cs sputtering ion source is used to extract BeO^- ions. We used BeO mixed into a Nb matrix as target material. A Nb matrix has previously been found to provide high output currents, as well as a good ionization yield in comparison to other metals [9, 10]. We found that a Nb/BeO mass mixing ratio of 4 is optimal for our ion source by measuring the ion current for targets with mixing ratios in the range of 0.3 to 6. From this mixture, we extracted a BeO^- ion beam of about 3 µA. The current starts to decrease after a total measurement time of about 1800 s on an individual target.

The 01-5-1 standard sample from K. Nishiizumi et al. [11], with a nominal ^{10}Be/^{9}Be ratio of $2.709 \cdot 10^{-11}$, was used for normalization during the measurements . The other dilutions of the Nishiizumi standard were used as known-ratio. The material was provided by the CNA in Sevilla.

Two different background materials were used. One was the carrier material which is used at the University of Bern in their Be extraction procedure for cosmogenic exposure dating (7 targets). This material has undergone the complete chemical extraction procedure and is therefore a good representation of the blank for the measurement of unknown samples. For each of these samples, 0.5 mg of carrier ^{9}Be were treated following the standard protocol of

the laboratory [12]. The addition of Fe and Ag in the end of the procedure was omitted and the precipitated Be was burnt to BeO at 1000 °C before mixing it with Nb powder to achieve an optimal target matrix. S. Merchel of Helmholtz-Zentrum Dresden-Rossendorf (HZDR) provided the other material (4 targets), which consists of Be extracted from deep minerals (phenakite), which contains a very little ^{10}Be [13]. Its ^{10}Be/^{9}Be ratio was measured at other AMS facilities to be in the 10^{-16} range.

In order to determine the ion transmission through the accelerator, the ion current was measured in a movable on-axis Faraday cup in front of the accelerator. After the accelerator, the ion current can be measured either in an on-axis cup or in the off-axis cup, which is used for ^{9}Be measurement under normal measurement conditions. Both cups on the high-energy side are biased with voltage of -300 V in order to contain secondary electrons and guarantee a correct current measurement. The cup on the low-energy side does not have a bias voltage.

For determining the ion yield in the degrader foil, the measured ratio of ^{10}Be/^{9}Be is normalized to the nominal ratio of the reference sample used. This method does not account for ion optical transmission in the ESA, but simulates actual measurement conditions. However, scanning the beam profile with the adjustable slits indicates that the ion optical losses are negligible. We set the slits to a width of 8 mm for our tests. This width corresponds to the size of the detector entrance window (8x8 mm, 75 nm thickness). Due to the maximal electric field of 4.8 kV/mm and the bending radius of 650 mm of the ESA, not all charge states can be investigated. In particular, charge states lower than the injected ones cannot be bent sufficiently at a 1 MV terminal voltage. The degrader foils are mounted on a moveable arm with four position for different foils. The degrader foils used are made of silicon nitride and the manufacturer recommends a thickness of 150 nm for this application.

To demonstrate the measurement performance for unknown samples, a regular measurement with standard samples of different ratios was also performed.

3 Results

3.1 Transmission

The accelerator transmission was measured at a terminal voltage of 1 MV and charge states 1^+, 2^+ and 3^+ were investigated. The measurement was done in both Faraday cups on the high-energy side, but the results were the same. The 1^+ state showed the highest yield with a transmission of 49.7 %. The 2+ state transmission was at 24.6 % whereas there was no signal in the 3^+ state. The stripper transmission includes charge state yield as well as ion optical losses and represents the best values achieved during the tests. A stripper gas inlet pressure of 0.014 mbar was set during these measurements.

A direct measurement of the ratio without a degrader foil is not possible because the overlapping ^{10}B beam is saturating the detection system. Instead, we investigated the 1+ and 2+ charge states after the degrader foil to identify the best performance. The transmission of the degrader foil also includes ion optical losses in the ESA and at the detector window. These losses are also influenced by the setting of the slits and the ESA voltage, which is crucial for the boron suppression. The results are listed in table I.

3.2 Blank

In addition to the efficiency of the degrading process, the blank level and therefore the separation between beryllium and boron is important for a precise measurement. Closing of the slits in front of the detector obviously reduces the total number of counts in the detector. However, the background to standard ratio is not affected by this. The blank values were normalized to the reference samples at the corresponding settings.

Individual blank values for several targets in the 2+2+ charge state are plotted in Fig. 2. The results were similar for both materials with a mean ratio of $^{10}Be/^{9}Be = 3.4\pm0.8\cdot10^{-15}$. The blank level of the other charge states was also investigated. A summary of the results is given in table I. However, the ratios for these states were only based on a single target each because their overall performance was not good enough to compete with the 2+2+ setting. The 1+1+ settings has a normalized blank value of $1.08\pm0.38\cdot10^{-14}$. The 1+2+ setting has an even higher blank level of $1.36\pm0.38\cdot10^{-13}$ which is mostly due to the boron content of the sample as the peaks in the detector spectrum are almost overlapping (Fig. 3). Another contribution to background in this setting can be ^{9}Be which undergoes charge exchanges and scattering [14].

3.3 Measurement precision

After the initial tests, we decided to continue the investigation with the 2+2+ charge states, i.e. choosing the 2+ charge state after the stripper as well as after the degrader foil. This operation mode has shown the best performance concerning both blank level and measurement efficiency. A total of 14 targets of the 01-5-1 standard have been prepared and were measured in the same batch to check the reproducibility of the measurement. The results are plotted in Fig. 4 and show that the different targets are measured at the same ratio within the single measurement uncertainties derived from counting statistics (χ_v^2=1.037), which is the dominant source of uncertainty. A precision of 0.4 % could be reached before the samples were depleted. Since the $^{10}Be/Be$ ratio of this material is high compared to usual sample material, the achievable precision for other materials is lower.

3.4 Measurement accuracy

In order to test the accuracy of the measurement, the other dilutions of the Nishiizumi standard were treated as unknowns. A total of 22 targets have been measured and

normalized to the Nishiizumi 01-5-1 samples. The results are presented in Fig. 5 as deviation from nominal value according to their nominal ^{10}Be concentration. A more detailed analysis of the variation is given with the results in table II.

We see that the measured ratios are in good agreement with the nominal values throughout the whole range that is covered by these samples. The uncertainties of these samples are larger than the ones of the 01-5-1 samples. This is primarily caused by their lower ^{10}Be content, which leads to a reduced counting statistics. The additional uncertainty due to normalization is small compared to this.

4 Discussion

The high yield of the 1+ charge state in the stripper suggests that it would provide efficient measurements. However, the limited energy of the ions hitting the degrader foil in the 1+ charge state leads to large beam losses due to angular scattering. In addition, the charge state distribution after the degrader foil only provides a low yield in the 1+ state, while the use of the more abundant 2+ state is prohibited by the high background rate. The separation between B and Be, both spatially after the ESA and in the detector spectrum, is rather small for these energies. While the separation is enough to allow the measurements in the 1+1+ state, the more abundant boron in the 2+ state is creating a much larger interference (Fig. 3). In addition, the boron tail is also increased which we assume is coming from the increased number of ions hitting the outer ESA plate, e.g. the 1+ ions. There also seems to be a peak like structure within the tail, which probably originates from scattering at a certain place, but we were not able to identify it. Due to the large number of counts, there is also a large number of pile-up events throughout the spectrum.

Compared to the 1+, the 2+ charge state gives only about half the transmission through the accelerator. However, the ion energy of about 2.3 MeV when hitting the degrader foil leads to a higher transmission there, which actually reduces the total loss. In addition, by carefully optimizing the system, it is possible to achieve a higher separation in the detector spectrum (Fig. 6, bottom left) and a lower blank level compared to the 1+ charge state. We identified two main points that need optimization.

The first is the optimization of the detector pressure. While optimizing the pressure to achieve a good separation is straight forward, a higher detector pressure is also desirable because it stops some of the ions in the first anode and therefore helps to reduce pile-ups in the detector electronics.

The other part is the setting of the ESA before the detector. The ^{10}Be peak is lying on a shoulder of the boron beam (Fig. 7). The boron intensity is exponentially decreasing towards higher settings of the ESA. This means that even a small adjustment can lead to a major change in the boron count rate. Meanwhile, the ^{10}Be peak is relatively wide and therefore allows some flexibility when setting the ESA for the measurement. This allows optimizing the setting to achieve a low background from boron counts with a high ^{10}Be efficiency. The lowest amount of boron is achieved for ESA voltages of about 44.45 kV or higher. Unfortunately, the ^{10}Be detection efficiency at these settings is also reduced by more than 50 %.

Careful optimization of these two elements are required to achieve a low background. The detector spectrum for an “unoptimized” setting shows a large amount of boron events and related pile-ups (Fig. 6, top plots). The background level corresponded to $^{10}Be/^{9}Be=2\cdot10^{-14}$. Optimizing the detector pressure, raising it from 8.0 mbar to 9.1 mbar, and adjusting the ESA

voltage, lead to a background reduction of almost one order of magnitude while only reducing the ^{10}Be counting efficiency by about 4 % (Fig. 6, lower plots). In comparison to the 1+2+ measurement, the spectrum shows an additional peak, which lies above the main boron peak. This peak is coming from oxygen that is part of the injected ions. The ion energy of about 2.6 MeV for the 2+ charge state is in good agreement with the detector signal. While these ions should be well rejected by the analyzing magnet, they can pass the magnet relatively well in charge state 3+, which can be achieved in a collision with the residual gas after the accelerator.

An investigation of a similar AMS system has previously been done at CNA [9]. While their tests investigated the same charge states, their results were slightly different. The stripper transmission in the 1+ charge state was reported to be 58 % by CNA [9]. We were not able to reproduce this and were limited to a transmission of about 50 %, which could be explained by an ion optical loss, which might be caused by small alignment variations between the systems and would affect the 1+ beam the most. However, the results of the 2+ charge state are in very good agreement, indicating that this is a rather minor issue. A difference in the stripper pressure could explain the difference. While we did see a decreased transmission, about 2 % less, for a higher stripper pressure of 0.02 mbar, we observe an increase in transmission for values lower than 0.014 mbar. A badly tuned system would be another explanation for the lower transmission value. However, since we went through all ion optical elements iteratively during optimization, trying to reach the 58 %, we found it unlikely that tuning would be the main reason for the observed difference. For the CNA system a transmission of 55 % has previously been reported as well [7], whereas KIGAM (Korea Institute of Geoscience and Mineral Resources) reports a transmission of 47 % on a similar

system [6]. As we identified better measurement conditions for the 2+ charge state, we did not investigate the different transmission for the 1+ further.

AMS systems with a terminal voltage of 1 MV or lower are usually using the 1+ charge state for Be measurements, commonly resulting in background levels in the order of 10^{-14} [6, 9]. This matches with the acceptance test of the NTNU system [5], which was carried out using the 1+1+ charge state. There has been lower blank ratios, in the 10^{-15} or 10^{-16} range, reported for low-energy AMS systems but these systems are using an extended spectrometer design, e.g. an additional magnet on the high-energy side, for further background reduction [15, 16]. Although somewhat higher than the background levels achieved using extended spectrometer designs, the $3.4 \cdot 10^{-15}$ background measured on our simpler spectrometer is low enough for many applications. The achievable precision of 0.4 % is not limiting for most applications and instead the low counting statistics limits the precision for most of the samples. Variations of ^{10}Be results in cosmogenic analysis are reported to be in order of several percent [17]. In addition, further processing of the results, e.g. to determine exposure ages, add larger uncertainties such that the measurement error is not of major concern [18].

The standards that we treated as unknowns for testing the system had lower ^{10}Be content than the normalization sample so that the precision is slightly lower. However, several targets of the same material were used to demonstrate the accuracy and the mean values are in good agreement with the nominal values. Provided there is enough material, it can be an option to use several targets for a single sample to get a more precise result, which would otherwise be limited by the overall efficiency.

5 Conclusion

The acceptance test of the NTNU AMS system for ^{10}Be measurements were done at the 1+1+ charge state selection, which only allowed a limited performance. Investigating the other charge states led us to use the 2+2+ setting for our measurements instead. This way, we could raise the efficiency by a factor of 2, and lower the background level by a factor of 3. The background level of $3.4 \cdot 10^{-15}$ is the smallest that has been measured in a similar instrument. Combined with an overall efficiency of 5.4 %, our system is ready for measurements of samples from many research fields. The efficiency is limiting the counting statistics, and therefore the achievable precision for ^{10}Be/Be ratios. When the ^{10}Be content is high enough, a precision of 0.4 % could be demonstrated. The accuracy of the system has been demonstrated using 5 standards of different ^{10}Be content. The results are well within the precision of the system.

6 Acknowledgement

We thank S. Merchel for providing ^{10}Be blank material.

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Tables

Table I Transmissions and blank values for different charge states after the stripper and the 150 nm degrader foil.

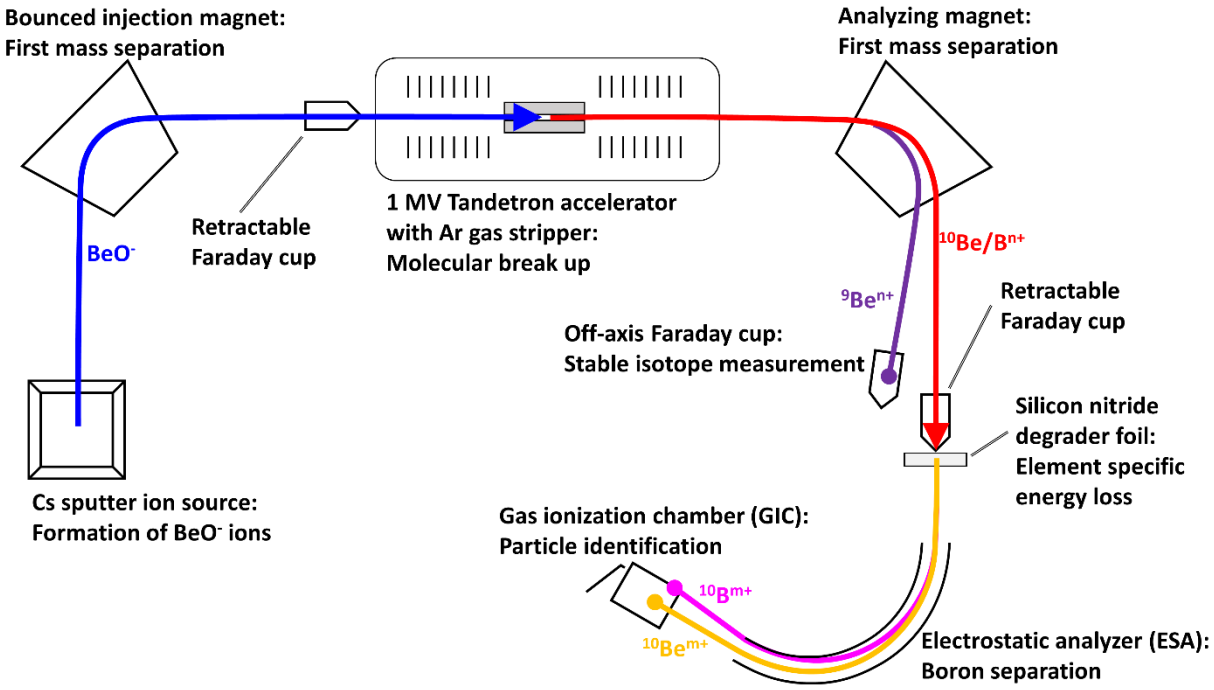
Stripper		Degrader			Overall	
Charge state	Transmission	Energy / MeV	Charge state	Transmission	Transmission	Blank value
1+	49.7±0.5%	1.3	1+	5.7±0.3%	2.8±0.2%	$1.08\pm0.38\cdot10^{-14}$
			2+	15.4±0.6%	7.7±0.3%	$1.36\pm0.38\cdot10^{-13}$
2+	24.6±0.3%	2.3	2+	22.1±0.6%	5.4±0.2%	$3.4\pm0.8\cdot10^{-15}$

Table II Results from the standard samples that were treated as unknowns in the measurement.

	^{10}Be/Be ratio / 10^{-12}			
sample	nominal	measured	1σ	1σ (%)
01-5-2	8.558	8.47	0.10	1.1 %
		8.49	0.10	1.1 %
		8.55	0.06	0.7 %
		8.64	0.05	0.6 %
	mean:	8.566	0.035	0.4 %
		χ_ν^2:	1.05	
01-5-3	6.32	6.30	0.08	1.2 %
		6.32	0.08	1.2 %
		6.28	0.07	1.2 %
		6.45	0.06	1.0 %
		6.41	0.07	1.2 %
	mean:	6.357	0.032	0.5 %
		χ_ν^2:	1.06	
01-5-4	2.851	2.85	0.04	1.3 %
		2.82	0.04	1.4 %
		2.89	0.04	1.3 %
		2.94	0.04	1.4 %
		2.96	0.05	1.5 %
	mean:	2.883	0.018	0.6 %
		χ_ν^2:	1.46	
01-6-1	0.9718	0.987	0.019	1.9 %
		0.963	0.019	2.0 %
		0.949	0.024	2.5 %
		0.970	0.030	3.1 %
	mean:	0.969	0.011	1.1 %
		χ_ν^2:	0.75	
01-6-2	0.5349	0.549	0.015	2.7 %
		0.545	0.014	2.6 %
		0.521	0.015	2.8 %
		0.545	0.012	2.2 %
	mean:	0.540	0.007	1.3 %
		χ_ν^2:	0.87	

Figures

Figure 1 Schematic of the Trondheim 1 MV AMS system. Only the most important components and the ion trajectories for regular Be measurements are shown.



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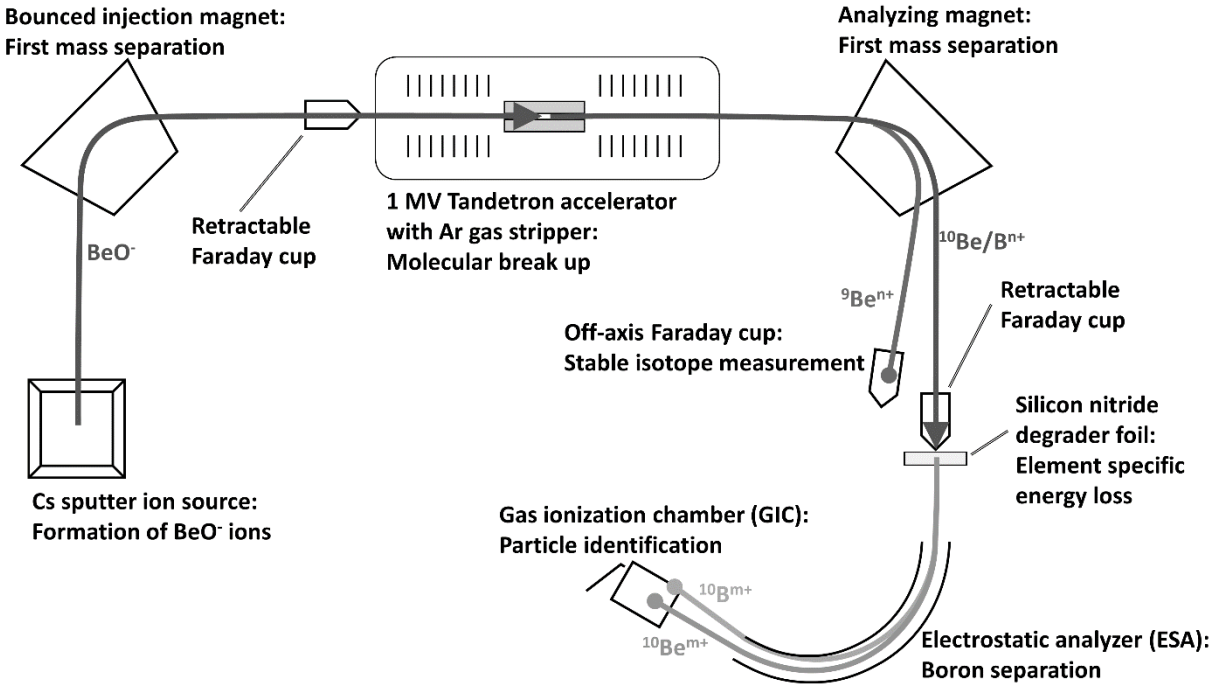


Figure 2 Blank results for both blank materials. There is no statistically relevant difference between the two materials, suggesting that the background level is given by the spectrometer of the AMS instrument and is not coming from the samples.

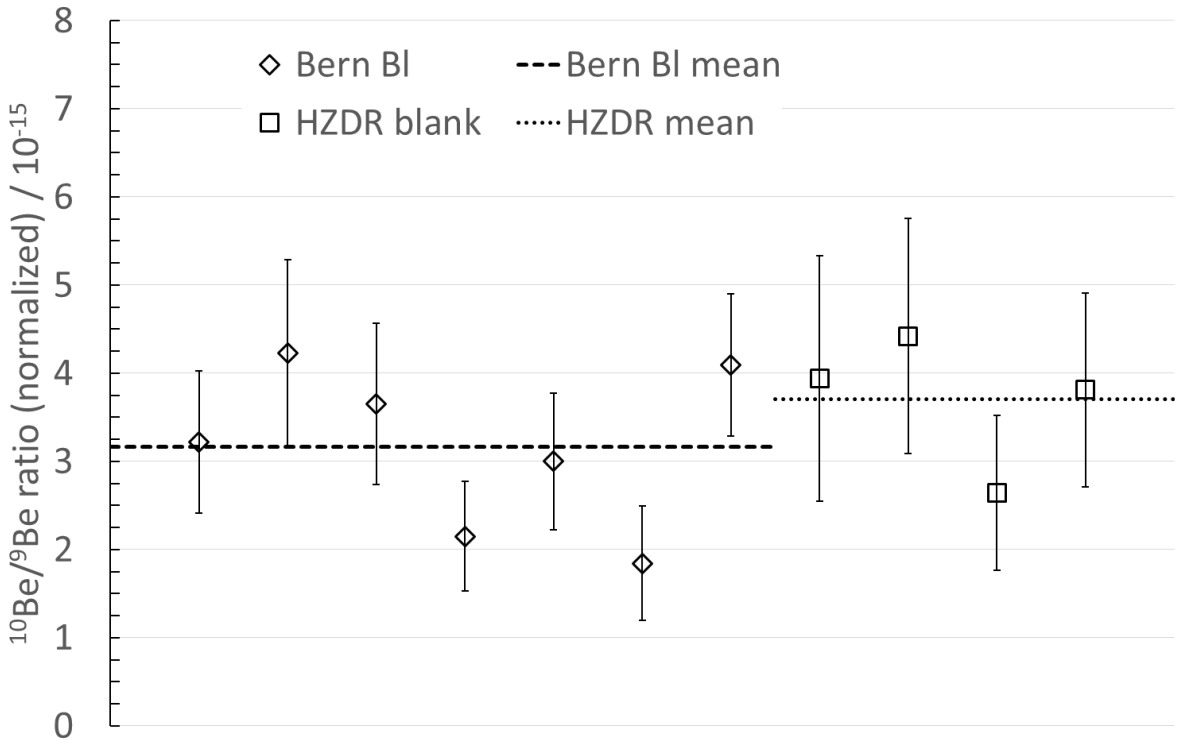
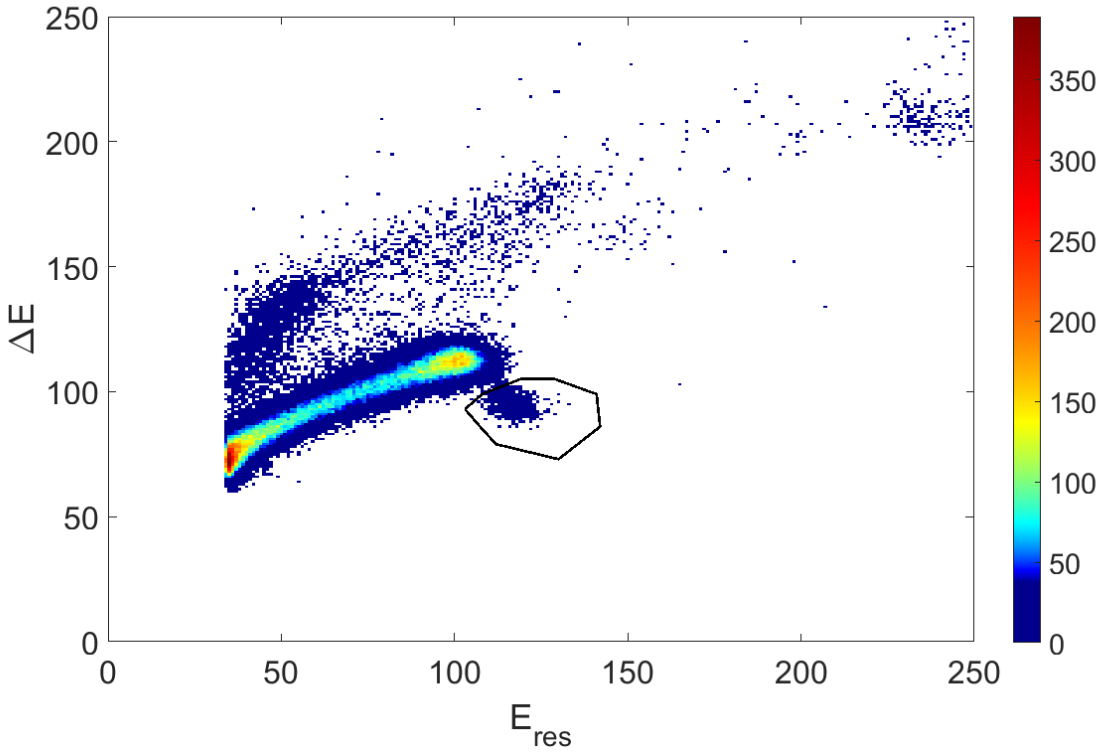


Figure 3 Detector spectrum with a reference sample for the 1+2+ charge state. The ^{10}Be gate is indicated with a black outline. The separation between the boron interference and the ^{10}Be peak is small which leads to a high background level. The recording time for this spectrum was 200 s.



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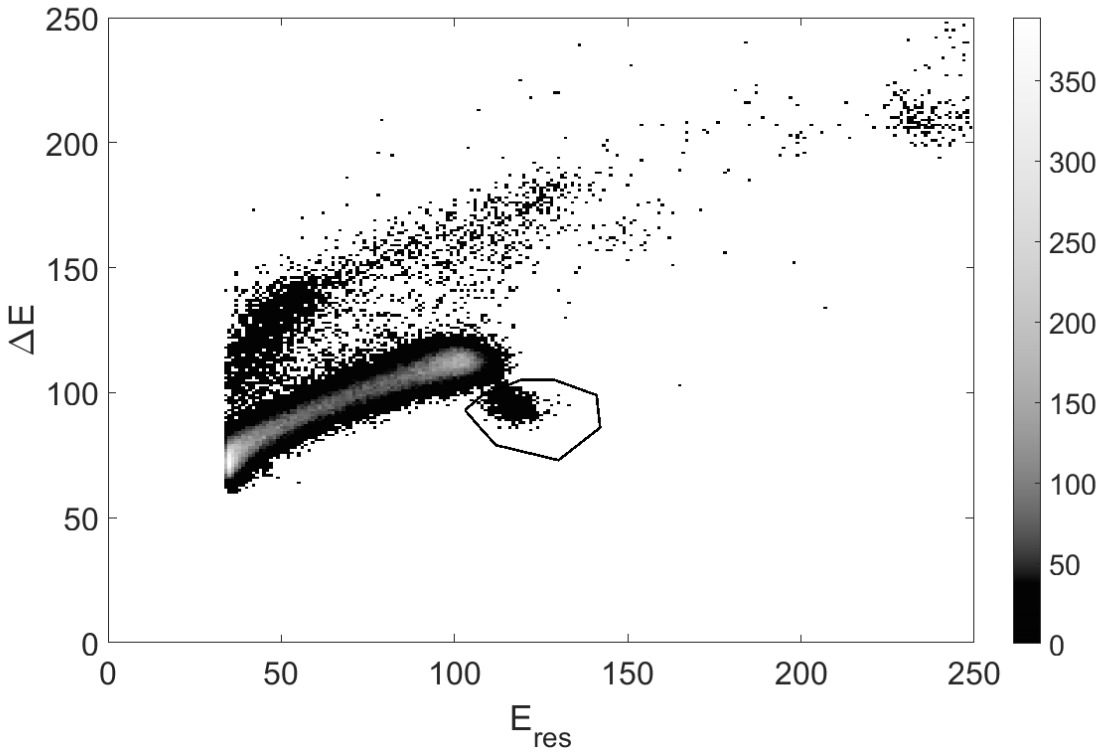

250
200
150
100
50
0
ΔE
0
50
100
150
200
250
E res
350
300
250
200
150
100
50
0

Figure 4 Deviation of the 01-5-1 standard samples, which were used for referencing in the measurement. The 1σ error bars are representing the counting statistics of the individual samples, which is on average 0.42 %. The standard deviation is 0.4 %.

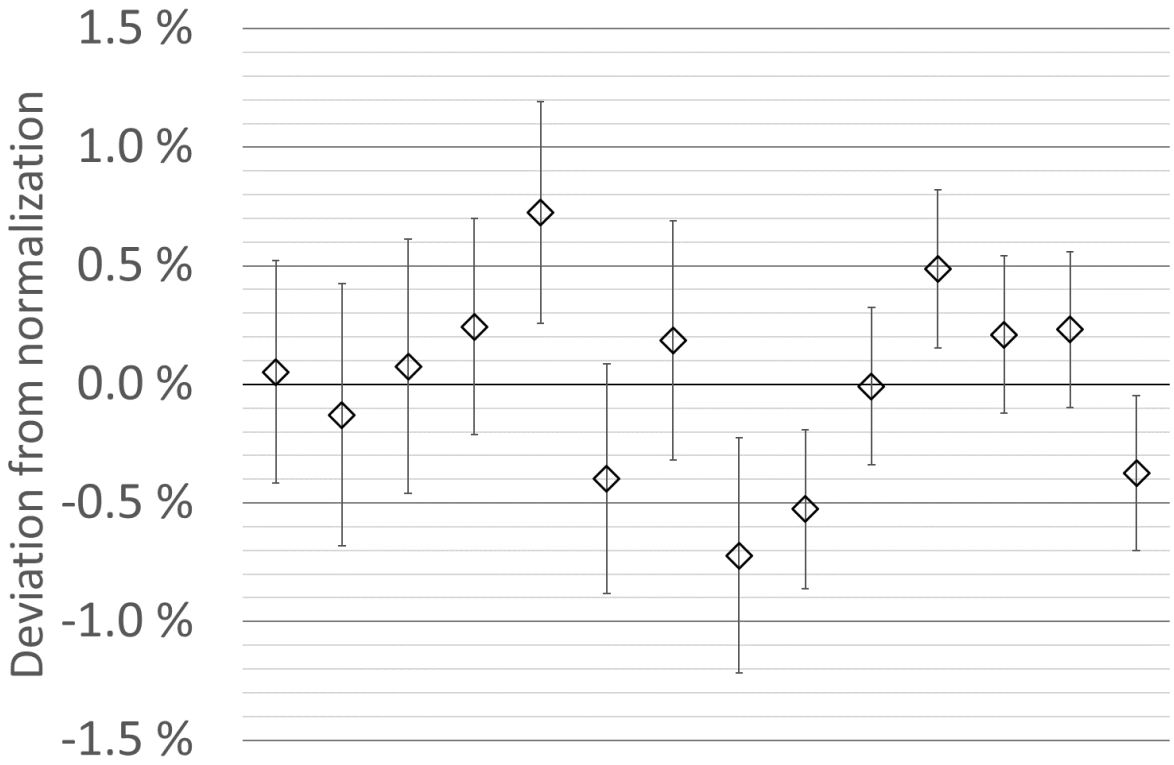


Figure 5 Deviation of the measured values from the literature values for the standard samples. The 1σ error bars include the counting statistics of the single samples as well as the error from normalization.

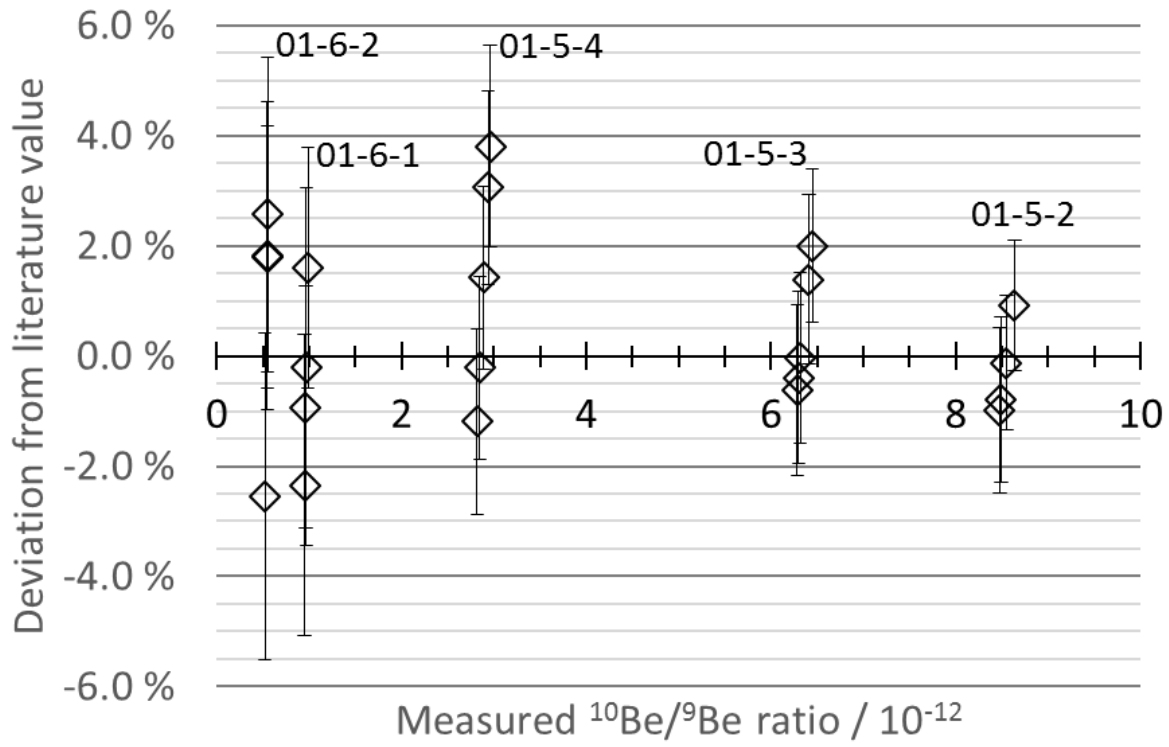
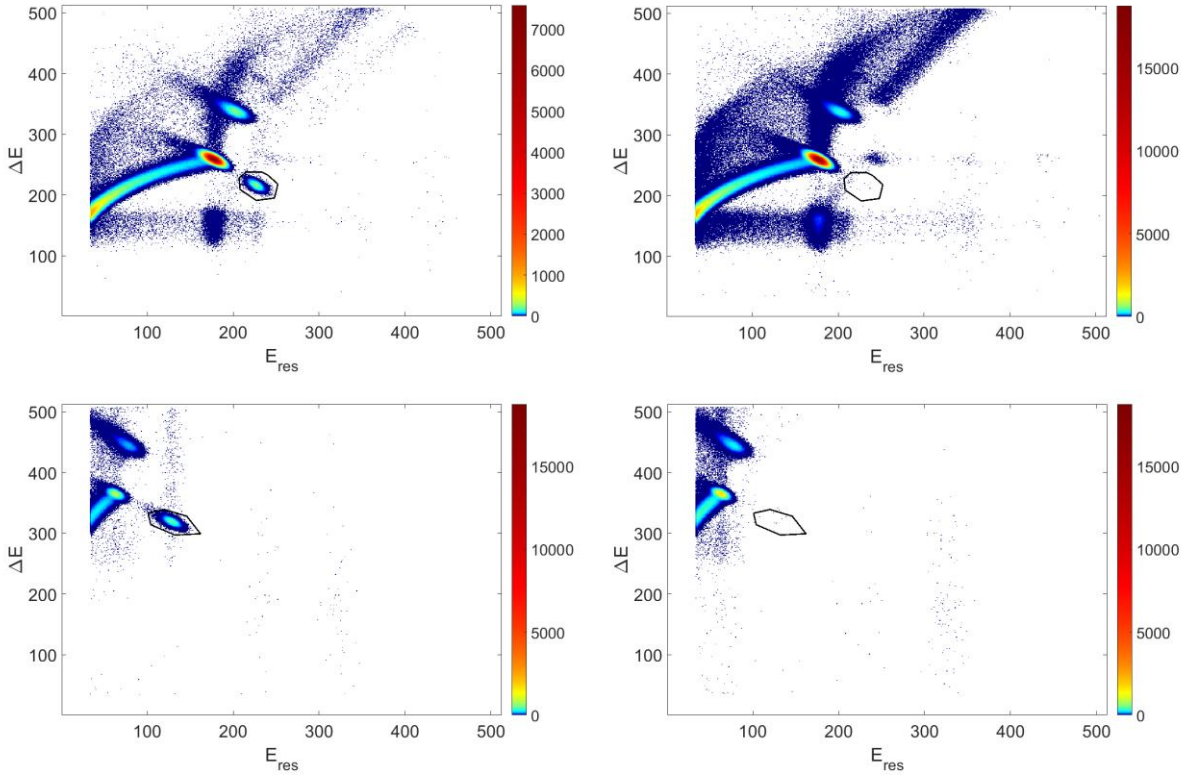


Figure 6 GIC energy spectra for reference samples (left) and blanks (right) for two settings. The top spectra were recorded at an ESA voltage of ±44.244 kV and a detector pressure of 8.0 mbar. The optimized setting (bottom spectra) used ±44.344 kV and 9.1 mbar respectively. The black contour line shows the ^{10}Be gate defined for the measurement.



Uncolored version for printing:

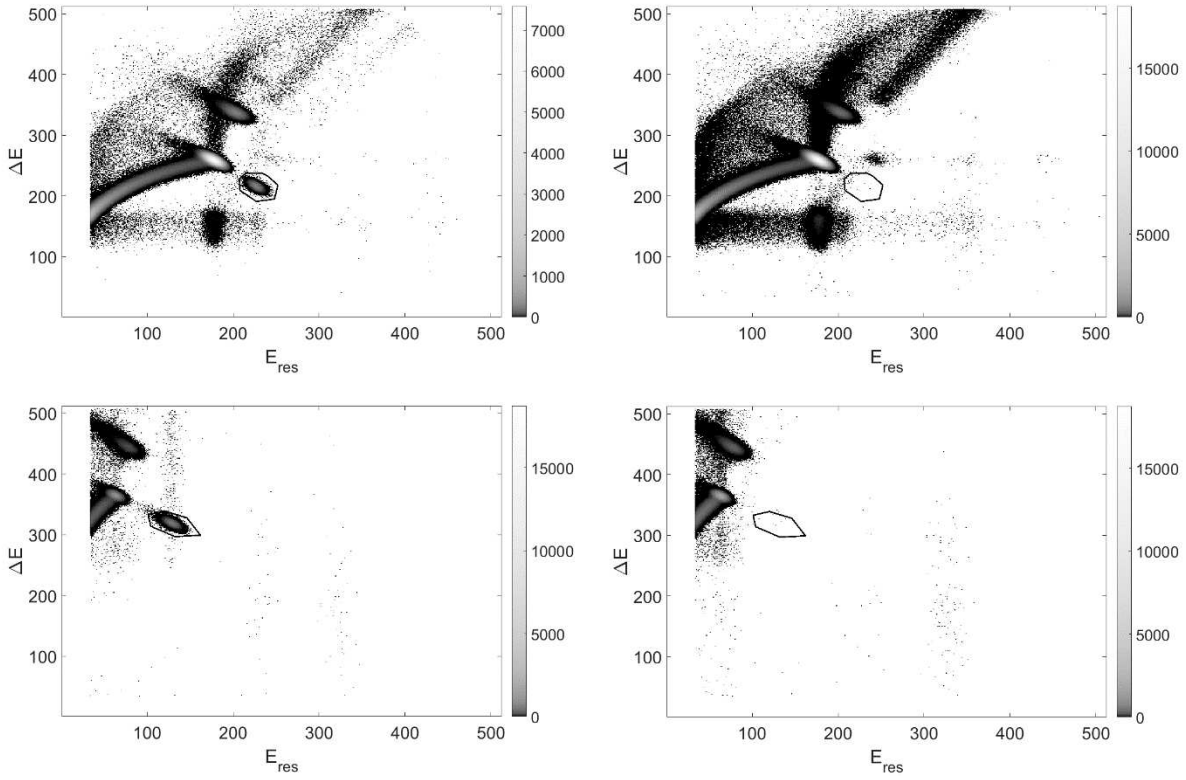

ΔE
E_res
500
400
300
200
100
100
200
300
400
500
7000
6000
5000
4000
3000
2000
1000
0
15000
10000
5000
0

Figure 7 Count rate for ^{10}Be and ^{10}B in the detector vs. ESA voltage. For ESA voltages lower than 44.1 kV, the high boron intensity leads to a saturation of the detector. The ^{10}B peak shows an exponential decrease in this range whereupon the ^{10}Be peak is comparably flat.